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# The Microgravity Research Experiments (MICREX) Data Base

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# The Microgravity Research Experiments (MICREX) Data Base

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# CHAPTER 12

# FLUID PHYSICS

**Principal Investigator(s):** Petrash, D. A. (1), Nussle, R. C. (2), Otto, E. W. (3)
**Co-Investigator(s):** None
**Affiliation(s):** (1,2,3) During Mercury: National Aeronautics and Space Administration (NASA), Lewis Research Center (LeRC), Cleveland, Ohio, Currently: (1,3) Retired; (2) NASA Lewis Research Center (LeRC), Cleveland, Ohio

**Experiment Origin:** USA
**Mission:** Mercury-Aurora 7 (MA-7)
**Launch Date/Expt. Date:** 1962
**Launched From:** Cape Canaveral Air Force Station, Florida
**Payload Type:** Mercury Capsule
**Processing Facility:** Experimental tank consisting of a 300 milliliter glass sphere with a cylindrical glass surface tension baffle fused at one end to the inside surface of the sphere (see Reference (1)). The tank was mounted slightly above and to the right of the astronaut's head.
**Builder of Processing Facility:** Designed and built by NASA Lewis Research Center, Cleveland, Ohio

**Experiment:**
Effects of Acceleration Disturbances on the Liquid-Vapor Interface in a Baffled Tank

Early drop tower reduced-gravity experiments indicated that when liquids wet container walls (as is the case for most propellants) the location of the special liquid-vapor interface in the low-gravity environment is random. However, research also illustrated that correct placement of specially-designed baffles within the container can result in a predictable positioning of this interface. Earth-bound studies did not illustrate, however, if the surface-tension baffling would (1) maintain control of the liquid-vapor interface during acceleration disturbances or, (2) regain control of the interface after the application of large disturbing forces.

This Mercury-Aurora experiment was designed to study the difficulties associated with storing liquid propellants in a space vehicle. The specific objectives of the investigation were to (1) study the long-term, steady-state configuration of the liquid-vapor interface, (2) determine the ability of the baffle configuration to maintain control of the liquid-vapor interface during spacecraft maneuvers, and (3) determine at what acceleration level (during re-entry) control of the interface is lost.

A specially constructed 3.3 inch diameter, 300 ml glass sphere experimental tank was used during the mission. A cylindrical surface-tension baffle, made of glass, was attached to one end of the inside sphere surface. The baffle, 1.1 inch in diameter, extended 1.9 inches into the tank. Flow between the tank and inside of the baffle was made possible by three holes (each approximately 1/3 of an inch in diameter). The experimental tank was encased and hermetically sealed at ambient conditions. The front of the encasement consisted of a transparent, Lucite hemisphere which allowed visualization of the liquid position. The back section of the encasement consisted of an aluminum hemisphere, painted white for light reflection (see Figure 2, Reference (1)).

The tank assembly was mounted in the Mercury-Aurora 7 capsule at a position just to the right of the astronaut's head (when he was in a sitting position) and was filmed by the pilot observer camera located in the instrument panel. A clock, which was used for correlating fluid behavior with spacecraft maneuvers, was also filmed. No photographic data were obtained during the periods of darkness of the mission.

The experimental tank was partially filled with distilled water. The remainder of the tank volume was filled with air. The water was modified with additives to reduce its surface tension. The additives ensured the compatibility of the space data with data obtained during drop tower experiments (during which ethyl alcohol was employed). (See References (2) and (3) for details of these drop tower experiments.)

Reportedly, salt water contacted the camera during the landing/recovery stage of the mission, and somewhat degraded the quality of the film. However, "...the quality... [of the film was]... satisfactory for analysis of the liquid behavior." (1, p. 4)

The film indicated that the steady-state configuration of the liquid-vapor interface was the same as that observed during drop tower experiments: the baffle being completely filled and the remaining liquid uniformly distributed at the base of the baffle. The fluid level in the tank was lower than that of the baffle.

"The time required for the liquid transition from the thrust configuration to the zero-gravity configuration was of the order of 12 seconds both at initial entry into zero gravity and after retrofire.... This period of time is longer than that observed in the models studied in the drop tower... and is attributed to the fact that the [Mercury] tank was larger and the holes at the base of the surface-tension baffle were comparatively smaller and limited the flow." (1, p. 4)

During orbit, the spacecraft underwent orientation maneuvers and acceleration disturbances to the experiment were realized. "Short-term activation of the 24-pound thrusters in pitch and yaw directions resulted in tangential acceleration disturbances on the experiment of 0.011 and 0.005 g, respectively. The 6-pound roll thruster imparted 0.003 g of acceleration disturbances on the experiment." (1, p. 3) It was reported that these maneuvers did not significantly affect the liquid-vapor interface. During pitch maneuvers (0.011 g), an occasional slosh of the liquid was noticed.

Just prior to re-entry, firing of the retrorockets was performed. This resulted in the liquid leaving the surface-tension baffle, but the interface quickly resumed the steady-state configuration upon re-establishing low-gravity conditions. As the spacecraft entered the Earth's atmosphere and gravity forces became large enough to overcome the surface-tension forces, the liquid left the baffle. Analysis of tracking data indicated that this event occurred at an acceleration level of 0.30 g.

**Key Words:** Fluid Physics, Propellant Storage, Contained Fluids, Liquid Reservoir, Spheres, Partially Filled Containers, Fuel Tanks, Fluid Management, Liquid Transfer, Baffles, Free Surface, Liquid/Vapor Interface, Solid/Liquid Interface, Interface Physics, Acceleration Effects, Sloshing, Liquid Dynamic Response, Surface Tension, Wetting, Wetting of Container, Launch Induced Fluid Motion, Vehicle Re-entry Forces/Vibration, Rocket Motion, Photographic Difficulties

**Number of Samples:** one baffled tank
**Sample Materials:** distilled water with additives to reduce the surface tension (see Reference (1) for a complete listing of additives)
**Container Materials:** glass tank with glass baffle

**Experiment/Material Applications:**
The control of the liquid-vapor interface under low-gravity conditions is required to assure proper delivery of the propellant to the engine. Short-term, drop tower experiments cannot provide the type of acceleration disturbances which may affect the position of the interface.

**References/Applicable Publications:**

(1) Petrash, D. A., Nussle, R. C., and Otto, E. W.: Effect of Acceleration Disturbances Encountered in the MA-7 Spacecraft on the Liquid-Vapor Interface in a Baffled Tank During Weightlessness. NASA TN D-1577, NASA, Washington, DC, 1963, 14 pp.

(2) Petrash, D. A., Zappa, R. F., and Otto, E. W.: Experimental Study of the Effects of Weightlessness on the Configuration of Mercury and Alcohol in Spherical Tanks. NASA TN D-1197, 1962. (pre-flight, drop tower experiments).

(3) Petrash, D. A., Nelson, T. M., and Otto, E. W.: A Study of the Effect of Surface Energy on the Liquid-Vapor Interface Configuration During Weightlessness. NASA TN D-1582, 1962. (pre-flight)

(4) Input received from Principal Investigator R. Nussle, August 1993.

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**Co-Investigator(s):** None
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**Experiment Origin:** USA
**Mission:** Apollo 14
**Launch Date/Expt. Date:** February 1971
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Apollo Command Module Payload, Hand-Operated Test Apparatus
**Processing Facility:** Scale model liquid transfer unit
**Builder of Processing Facility:** NASA Lewis Research Center, Cleveland, Ohio

**Experiment:**
Liquid Transfer Demonstration

During future space operations, the transfer of a liquid from one tank to another will be necessary. A few of the fluid-management challenges involved with the transfer include: (1) gas free outflow from the supply tank, (2) spill free inflow into the receiver tank, (3) control of the liquid vapor interface during acceleration perturbations, and (4) positioning of the fluid at the drain port.

Experimental and analytical investigations (cited by the investigators) of reduced-gravity liquid drainage and liquid inflow indicated that "...liquid-transfer systems with high total delivery efficiency could not be achieved in weightlessness without the addition of internal devices to control the position of the liquid vapor interface." (1, p. 2)

The objective of this Apollo 14 experiment was to demonstrate the effectiveness of two surface tension baffle designs during low-gravity fluid transfer.

Two scale-model, liquid transfer units were employed for the experiment. One unit contained a pair of baffled tanks, the other, a pair of unbaffled tanks. Each tank was a flat cylinder (or disc) 10.16 cm in diameter, 0.635 cm thick (long). These discs, which permitted photographic fluid flow analysis, allowed simulation (in two dimensions) of the three-dimensional flow in a spherical tank.

Each of the two baffled tanks had a different baffle design. The first, called a standpipe-liner baffle design consisted of (1) a standpipe (located over the drain/fill port) and (2) a wall liner (spaced a distance away from the tank wall). "The standpipe has the characteristics of a capillary tube and will retain liquid within the standpipe and externally adjacent to the standpipe. The standpipe tank has been demonstrated to be very effective [in drop tower tests] in positioning the liquid in the tank area about the standpipe base, even when the liquid is initially adversely located and also under limited acceleration perturbations.... The wall liner is a capillary surface which locates a layer of liquid along the wall. The spacing of the liner away from the tank wall and the hole size of the openings in the liner determine the inherent residual liquid remaining after draining. The liner concept has been shown to be effective [in drop tower tests] for expulsion tanks.... The combined effects of these two concepts results in a capillary system that has the characteristics of retaining liquid in a preferential location and permitting liquid flow in or out from the same location. In order to prevent premature vapor ingestion through the standpipe during draining, a solid deflection baffle was placed at the base of the standpipe. In addition, the incoming pressurant is diffused by deflecting the flow 90$^{o}$. The location of this pressurant inlet port is extended within the tank such that it is located at the center of the anticipated ullage space in weightlessness when the tank is 90 percent full of liquid." (1, p. 5)

The second baffle design, called a curved-web design, "...consists of a series of three circular perforated plates nestled around a smaller feeder capillary section.... The circular-shaped plates, or curved-web baffles, are arranged off-center such that the cross sectional area between baffles increases gradually from the feeder capillary section towards the opposite end of the tank. This arrangement tends to locate the liquid vapor interface such that the liquid bulk is adjacent to the feeder capillary. As in the standpipe liner tank, the curved-web baffle system also contains both a small deflection baffle over the feeder capillary section and a pressurant gas diffuser...." (1, p. 6)

Each baffle "...was designed to achieve the following objectives:

"(1) Gas-free outflow from the supply tank with high total delivery efficiency

"(2) Orderly inflow into the receiver tank with no liquid loss through the gas vent

"(3) Ability to achieve a configuration with the gas located at the tank vent and the liquid at the drain/fill port." (1, p. 5)

Each tank was configured with two ports: a drain/fill port and a vent port. "The drain/fill ports on each pair of tanks were connected by a transfer tube that contained a slide-action isolation valve. The vent port on each tank contained an identical slide-action valve." (1, p. 3) The hand operated, screw-driven piston pump provided pressure at one port and created suction at the other.

"The liquid used in the tanks was an inert fluorochemical, perfluorotributylamine, which satisfied the safety requirements for the spacecraft. Furthermore, the liquid had a zero degree static contact angle on the tank surfaces to simulate the contact angle of most propellants on typical spacecraft materials. To improve the quality of the photographic data, a small amount of dye was added to the liquid." (1, p. 3) (Other properties of the liquid were detailed in Reference (3).)

"Each tank section, when connected to the piston pump with the flexible tubing, makes up a closed-loop transfer system. One of the tanks, starting with a predominantly liquid filled condition, represents the supply tank. The other tank in the set, either empty or nearly empty, is the receiver tank. The "vent sides of both tanks were connected to the pressure and suction sides of the pump, respectively, closing the system. The isolation valves on the vent are opened, as well as those on the transfer tube valve interconnecting the "drain/fill" side of the tanks. Operation of the hand-crank on the piston pump in one direction pressurizes the supply tank and simultaneously creates an equivalent amount of suction in the receiver tank causing fluid flow. By rotating the crank uniformly the flow rate remains reasonably constant. Transfer is now continued until (1) liquid is depleted from the supply tank, (2) vapor is beginning to ingest into the outlet of the supply tank, or (3) liquid reaches the vicinity of the vent side of the receiver tank. On completion of a transfer operation liquid transfer can be reversed simply by rotation of the pump in the opposite direction. The receiver tank, which is now relatively full of liquid becomes the supply tank." (1, p. 6)

"During transfer operations, one crew member photographed the tanks either with a motion picture sequence camera or with the onboard video camera, while another operated the piston pump at a prescribed rate." (1, p. 4) Reportedly, "...during operations to familiarize crew members with techniques in the weightless environment, small amounts of the liquid inadvertently became trapped in the piston pump system. As a result, the initial fillings for the tests varied, although this should not detract from the performance demonstration of the system operation. Several transfer operations were performed by the crew. The results of four of these tests have been selected as representative of the system operation. Three of the tests are for the baffled tank system; the fourth for an unbaffled tank test, is included for comparison. (1, p. 6)

The Unbaffled Tank Test

The unbaffled tank test illustrated that at the beginning of the liquid transfer, the interface was, as expected, circular in shape, forming a gas bubble "...located such that a liquid layer covered both the drain and vent sides of the tank. The liquid filling is estimated to be 36 percent of the tank volume, with an additional 10 percent contained in the transfer tube connecting the two tanks. A gas bubble formed directly over the vent or pressurant inlet as the tank pressurized to start the transfer.

"During the transfer... the liquid-vapor interface in the receiver tank was deformed because of the incoming liquid jet; however, the interface appeared stable." (1, p. 7) Calculations of the stability of the interface indicated that stable flow was expected.

"Gas ingestion occurred quickly at the point where the liquid remaining was 24 percent of the tank volume, which for this case resulted in less than 12 percent of the tank volume being delivered.... [L]iquid eventually ingested in the receiver tank vent." (1, p. 7)

It was concluded that "...unbaffled tanks were not suitable for transfer of liquid in weightlessness." (1, p. 10)

The Baffled Tank Tests

"The first of these transfer tests used the curved-web baffled tank as the supply tank and the standpipe liner as the receiver tank. The second test reversed the procedure with the standpipe liner as the supply tank. For these tests, the flow rate was the same order of magnitude as for the transfer with the unbaffled tanks. The third transfer operation was performed at about four times the flow rate with the curved web tank as the supply and liquid flow to the standpipe-liner tank receiver...." (1, p. 8)

It was concluded that "Two surface-tension baffle designs incorporated in separate tanks were shown to be effective both as supply tanks and as receiver tanks. Liquid was transferred to and from either baffled tank in weightlessness to within 2 percent of the nondeliverable liquid fraction (design penalty) without reaching gas ingestion. The liquid interface in the receiver tank was positioned successfully during inflow with the gas at the vent." (1, p. 10)

Further details of each of the transfer operations can be found in Reference (1), pages 8-9.

**Key Words:** Fluid Physics, Propellant Transfer, Refueling in Orbit, Propellant Tanks, Liquid Reservoir, Liquid Transfer, Fluid Management, Contained Fluids, Partially Filled Containers, Baffles, Free Surface, Solid/Liquid Interface, Interface Physics, Liquid/Vapor Interface, Interface Shapes, Interface Stability, Gas Ingestion, Gas-Free Liquid Expulsion, Bubble Formation, Wetting, Contact Angle, Capillary Forces, Capillary Tubes, Capillary Flow, Surface Tension, Sloshing, Piston System, Acceleration Effects

**Number of Samples:** four fluid transfer tanks (two baffled, two non-baffled)
**Sample Materials:** Inert Fluorochemical: Perfluorotributylamine dyed for photographic purposes. (The liquid forms a near zero degree contact angle.)
**Container Materials:** The tanks were made of near-optically-clear plastic.

**Experiment/Material Applications:**
One element of propellant management that will be necessary during future space operations will be the transfer of liquid from a tanker vehicle to a receiver vehicle. Examples of such fluid management include: (1) transfer of propellants from a shuttle-craft to a space station, (2) transfer of propellants to tanks of vehicles designed for lunar or planetary missions, (3) transfer of vital fluids to the space station, and (4) restart of propulsion systems in space.

**References/Applicable Publications:**
(1) Abdalla, K. L., Otto, E. W., Symons, E. P., and Petrash, D. A.: Liquid Transfer Demonstration on Board Apollo 14 During Transearth Coast. NASA Technical Memorandum NASA TM X-2410, November 1971, 30 pp. (post-flight)

(2) Abdalla, K. L. and Berenyi, S. G.: Vapor Ingestion Phenomenon in Weightlessness. NASA TN D-5210, 1969. (related research)

(3) Berenyi, S. G. and Abdalla, K. L.: Liquid-Vapor Interface During Outflow in Weightlessness. NASA TM X-1811, 1969. (related research)

(4) Berenyi, S. G. and Abdalla, K. L.: Vapor Ingestion Phenomenon in Hemispherically Bottomed Tanks in Normal Gravity and in Weightlessness. NASA TN D-5704, 1970. (related research)

(5) Symons, E. P., Nussle, R. P., and Abdalla, K. L.: Liquid Inflow to Initially Empty Hemispherical Ended Cylinders During Weightlessness. NASA TN D-4628, 1968. (related research)

(6) Symons, E. P. and Nussle, R. C.: Observations of Interface Behavior During Inflow to an Elliptical Ended Cylinder in Weightlessness. NASA TM-1719, 1969. (related research)

(7) Symons, E. P.: Interface Stability During Liquid Inflow to Initially Empty Hemispherical Ended Cylinders in Weightlessness. NASA TM X-2003, 1970. (related research)

(8) Symons, E. P.: Liquid Inflow to Partially Full, Hemispherical Ended Cylinders During Weightlessness. NASA TM X-1934, 1969. (related research)

(9) Abdalla, K. L., Flage, R. A., and Jackson, R. G.: Zero-Gravity Performance of Ullage Control Surface with Liquid Hydrogen While Subjected to Unsymmetrical Radiant Heating. NASA TM X-1001, 1964. (related research)

(10) Response received from Principal Investigator, June 1993.

<Note: Additional references to related research can be found on pages 11-15 of publication (1) above.>

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**Co-Investigator(s):** None
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**Experiment Origin:** USA
**Mission:** Apollo 14
**Launch Date/Expt. Date:** February 1971 (The experiment was performed during the Apollo 14 lunar flyback.)
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Apollo Science Demonstration, Apollo Command Module Payload
**Processing Facility:** Carry-on experiment housed in a fold up box, containing cylinders and flat dishes
**Builder of Processing Facility:** Lockheed Missiles and Space Company, Huntsville, Alabama

**Experiment:**
Heat Flow and Convection

This Apollo 14 demonstration was the first in a series of investigations designed by Bannister et al. to study low-gravity heat flow and convection. The experimental apparatus consisted of three independent cells designed to observe thermally-driven convection.

The First Cell
The first cell, called the radial cell, was an enclosed cylinder containing (1) carbon dioxide gas, (2) a tapered axial heater, and (3) a liquid crystal circular patch. During the mission, the gas was warmed by the heater. Temperature changes within the cell were indicated by color changes of the liquid crystal patch and documented via time lapse photography using the Apollo 16mm camera. (The camera was placed at one end of the cylinder looking down its longitudinal axis and focused on the liquid crystal display.) Cell flow patterns were not monitored (the gas contained no tracer particles; only the liquid crystal display provided data for the experiment).

Examination of the space data indicated reduced heat flow had occurred in the low-gravity radial cell. After analyses of the isotherm vs. time curves were calculated for the cell, it was reported that "One of the most striking features of the... curves was their undulatory character or the presence of long period oscillations."(1, p. 4) Experimental time temperature curves were compared to curves calculated based on the assumption that conduction and radiation were the only heat transfer modes. Reportedly, "The Apollo 14 flight curve deviates considerably from the conduction/radiation curve... an indication of convection.... It was concluded that g-jitter of the $10^{-3}$ to $10^{-4}$g level was responsible for the observed Apollo 14 convection." (1, p. 4) (The g-level estimate was made by tracking a trapped, small air bubble in the third cell. A discussion of the third cell is presented below.)

The Second Cell

The second cell, called the flow pattern cell, consisted of a pan with a removable cover. At the start of the experiment, the cover was removed and oil was initiated into the cell. (Fluid positioning in the cell was aided by baffles placed along the pan rim.) Once the free-surface fluid was positioned, the pan was heated from underneath (cover still removed), the geometry of the Bénard problem.

The oil employed for the experiment contained small, flat, aluminum flakes such that the fluid flow pattern could be visualized. This pattern was documented via time lapse photography using the Apollo 16 mm camera. (The camera's focus was directed perpendicular to the liquid free surface.) This cell did not contain liquid crystal patches for temperature change determinations.

The low-gravity flow pattern resulting in this cell was less orderly and symmetrical than Bénard-type hexagonal cell patterns observed during similar ground-based experiments. Although according to the available references, the low-gravity results were hampered by the inability to achieve a flat, free-surface layer of fluid, it was reported that "...[in the low-gravity flow pattern cell] convection cells with three to four sides are clearly visible in the fluid adjacent to the wall. A somewhat less distinct pattern is seen in the fluid layer (estimated to be about 0.2 mm) in the center of the pan. The cells in the thicker wedge appeared about 23 seconds after the cells in the center layer, in general contradiction with the theory which predicts that cellular convection occurs at lower temperature gradients in thick fluid layers than in thinner fluid layers.... The later appearance of the cellular pattern developed during an earlier noncellular flow caused at the pan walls by a radial temperature gradient. Furthermore, an underlying steady flow distorts a regular cellular pattern.... The irregular pattern actually observed, therefore, is in keeping with the postulation of an undercurrent." (2, p. 507)

Reportedly, this experiment was the first low-gravity investigation to conclusively show cellular convection due to surface tension in a space environment.

The Third Cell

The third cell, called the lineal or zone cell, consisted of "...two [equal length] glass cylinders, one containing gas-free distilled water and the other containing a 20 percent (by weight) sucrose solution...." (2, p. 507). (The contents of the two cylinders were isolated from each other throughout the entire experiment by the glass walls.) The cylinders were not configured in the cell end to end, but longitudinally one above another such that a single band heater at the end of the cell heated the contents of each cylinder from the same end. Each cylinder contained a liquid crystal shaft extending lengthwise down the center of the cylinder to allow observation of fluid temperature vs. position. (The cell contained no tracer particles as fluid flow patterns of the cell were not to be documented.)

Reportedly, first and second order convection was observed, although it was small in comparison to that which occurred in the first cell.

Additional information concerning this experiment can be found in Reference (2).

**Key Words:** Fluid Physics, Buoyancy-Driven Convection, Marangoni Convection, Surface Tension-Driven Convection, Thermocapillary Convection, Bénard Convection, Bénard Cells, Thermal Convection, Gaseous Convection, Cellular Convection, Buoyancy Effects Diminished, Thermal Gradient, Thermal Diffusion, Diffusion, Heat Transfer, Heat Conduction, Radiation, Baffles, Fluid Management, Liquid Transfer, Interface Shape, Interfacial Curvature, Surface Tension, Free Surface, Liquid/Gas Interface, Solid/Liquid Interface, Vapor/Solid Interface, Tracer Particles, Acceleration Effects, Liquid Expulsion Through a Small Orifice, Contained Fluids, Wetting of Container, Non-Wetting of Container, Liquid Crystals

**Number of Samples:** Three cells but a total of four samples: one radial cell (one sample), one flow pattern cell (one sample), one zone cell (two samples).

**Sample Materials:** Carbon dioxide (radial cell); Krytox oil (flow pattern cell); water and sucrose solution (zone cell). For photographic purposes the Krytox oil (flow pattern cell) contained a suspension of tiny a flakes.
**Container Materials:** (1) Flow pattern cell: the bottom of the dish was made of aluminum (high wettability); the sides of the dish were made of an insulating plastic (low wettability); (2) lineal cell: two glass cylinders; (3) radial cell: unknown

**Experiment/Material Applications:**
This early science demonstration was performed to graphically illustrate the advantages of low-gravity for suppressing convection in geometries common to processing of materials. It was concluded that buoyancy-driven convection was reduced. However, vivid photographs of surface tension driven convection alerted the science community of the need to consider Marangoni convection effects in space.

**References/Applicable Publications:**
(1) Grodzka, P. G. and Bannister, T. C.: Natural Convection in Low-G Environments. 12th American Institute of Aeronautics and Astronautics Aerospace Sciences Meeting, Washington, D.C., January 30-February 1, 1974, 13 pp., AIAA Paper #74-156 (post-flight)

(2) Grodzka, P. G. and Bannister, T. C.: Heat Flow and Convection Demonstration Experiments Aboard Apollo 14. Science, Vol. 176, May 5, 1972, pp. 506-508. (post-flight)

(3) Grodzka, P. G., Fan, C., and Hedden, R. O.: The Apollo 14 Heat Flow and Convection Demonstration Experiments; Final Results of Data Analysis. LMSF-HREC D22533, Lockheed Missiles and Space Company, Huntsville, Alabama, September 1971. (post-flight)

(4) Bannister, T. C., Grodzka, P. G., Spradley, L. W., Bourgeois, S. V., Hedden, O., and Facemire, B. R.: Apollo 17 Heat Flow and Convection Experiments, Final Data Analyses Results. NASA TM X-64772, July 16, 1973. (post-flight)

(5) Gatewood, E., Morris, M. G., and Holland, R. L.: Acceleration Levels on the Heat Flow and Convection Demonstration- Apollo 14. NASA TM X-64644, February 11, 1972, 19 pp. (post-flight)

(6) Naumann, R. J. and Mason, D.: Heat Flow and Convection Experiment. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 8-9. (post-flight)

(7) Input received from Principal Investigator T. C. Bannister, July 1988 and June 1993.

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**Experiment Origin:** USA
**Mission:** Apollo 17
**Launch Date/Expt. Date:** December 1972
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Apollo Science Demonstration, Apollo Command Module Payload
**Processing Facility:** Carry-on experiment housed in fold-up box containing cylinders and flat dishes
**Builder of Processing Facility:** Lockheed Missiles and Space Company, Huntsville, Alabama

**Experiment:**
Heat Flow and Convection

This Apollo 17 demonstration was the second in a series of investigations designed by Bannister et al. to study low-gravity heat flow and convection (see Bannister, Apollo 14).

Three experiment cells, slightly modified after the earlier Apollo 14 flight, were employed to observe thermally-driven convection.

The First and Third Cells
The first cell, called the radial cell, was an enclosed cylinder containing argon gas. The cell contained a small, cylindrical, axial post heater. The third cell, called the lineal cell, was an enclosed cylinder containing Krytox oil. Magnesium particles were suspended in the oil to permit observation of the fluid flow when the cell was heated from one end. Reportedly, both the radial and lineal cells were configured with a liquid crystal membrane. <Note: Although it was not clear in the available references, it appears that (1) the radial cell had a circular liquid crystal patch configured within the cylinder and (2) the lineal cell had a liquid crystal shaft extending lengthwise down the center of the cylinder.>

During the mission, the contents of both cells were heated. Time lapse photography was used to (1) record temperature changes in both cylinders (as indicated by color changes in the liquid crystals) and (2) document the resulting flow field in the lineal cell. <Note: It is not clear how the camera was positioned with respect to each cell during the experiments.>

Examination of the space data indicated a reduced heat flow in both the radial and lineal cells. The recorded thermal response was compared to analytical predictions. Reportedly, "No significant heat flow over that predicted for the pure conduction cases was observed in the Apollo 17 tests. Nor was any significant movement of the magnesium particles noted. We concluded, therefore, that no significant convection occurred in the Apollo 17 confined fluids tests." (2, p. 166) It was further reported that the spacecraft g-levels of Apollo 17 (which were different from those of Apollo 14) may have been responsible for this reduced convection.

The Second Cell

The second cell, called the flow pattern cell, was a pan with a removable cover. At the start of the experiment, the cover was removed and oil was initiated into the cell. (Aluminum flakes were suspended in the oil such that the fluid flow pattern could be visualized.)

Fluid positioning in the cell was aided by baffles placed along the pan rim. These baffles had been redesigned following the Apollo 14 flight. The improved design "...allowed two somewhat convex layers, approximately 2-mm and 4-mm in depth, to be heated." (2, p. 165) Once the free surface liquid was positioned, the pan was heated from underneath (cover still removed), the geometry of the Bénard problem. The resulting fluid flow pattern was documented via time lapse photography. (The camera's focus was directed perpendicular to the liquid free surface.)

After examination of the space data it was reported that "In both the 2-mm and 4-mm cases a concentric side roll and polygonal cells in the central area were seen to form at just about the same time. After a few minutes, the polygonal cellular convection dominated." (2, p. 166) The convection had a much more regular pattern than that seen in the earlier Apollo 14 flow pattern experiment cell. A critical condition for the onset of surface-tension driven convection was observed.

The results from this cell showed that cellular convection due to surface tension gradients can occur in the space environment.

It was concluded that buoyancy-driven convection was reduced in the low-gravity environment (radial and lineal cell). However, vivid photographs of surface tension-driven convection (in the flow pattern cell) alerted the science community of the need to consider Marangoni convection effects in space.

**Key Words:** Fluid Physics, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Marangoni Convection, Surface Tension-Driven Convection, Bénard Convection, Bénard Cells, Thermal Convection, Gaseous Convection, Cellular Convection, Thermal Diffusion, Diffusion, Heat Conduction, Radiation, Thermal Gradient, Heat Transfer, Surface Tension, Free Surface, Fluid Management, Liquid Transfer, Interface Shape, Liquid/Gas Interface, Solid/Liquid Interface, Vapor/Solid Interface, Tracer Particles, Baffles, Surfactants, Acceleration Effects, Liquid Expulsion Through a Small Orifice, Contained Fluids, Liquid Crystals

**Number of Samples:** three
**Sample Materials:** radial cell: argon gas; flow pattern cell: Krytox 143AZ oil, a perkfluoroalkylpolyether, to which was added tiny flakes of suspended aluminum and a surfactant to aid in the wetting of the aluminum powder; lineal cell: Krytox 143AA oil with a suspension of magnesium particles
**Container Materials:** Unclear; they may have been the same as those cells employed on Apollo 14.

**Experiment/Material Applications:**
See Bannister, Apollo 14.

**References/Applicable Publications:**

(1) Grodzka, P. G. and Bannister, T. C.: Natural Convection in Low-G Environments. 12th American Institute of Aeronautics and Astronautics Aerospace Sciences Meeting, Washington D.C., January 30-February 1, 1974, 13 pp., AIAA Paper #74-156 (post-flight)

(2) Grodzka, P. G. and Bannister, T. C.: Heat Flow and Convection Experiments Aboard Apollo 17. Science, Vol. 187, January 17, 1975, pp. 165-167. (post-flight)

(3) Bannister, T. C., Grodzka, P. G., Spradley, L. W., Bourgeois, S. V., Hedden, R. O., and Facemire, B. R.: Apollo 17 Heat Flow and Convection Demonstration Experiments, Final Data Analyses Results. NASA TM X-64772, July 16, 1973.

(4) Naumann, R. J. and Mason, D.: Heat Flow and Convection Experiment. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 8-9. (post-flight)

(5) Input received from Principal Investigator T. C. Bannister, July 1988 and June 1993.

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**Experiment Origin:** USA
**Mission:** Skylab Program
**Launch Date/Expt. Date:** Not applicable. Although this principal investigator was chosen as a finalist in the student experiment program, the hardware associated with this experiment was never fabricated. The student assisted with another aspect of Skylab.
**Launched From:** Not Applicable
**Payload Type:** Skylab Student Project, High School Student Experiment; Skylab Manned Environment (Planned)
**Processing Facility:** A piston assembly used to force solids through sized openings
**Builder of Processing Facility:** Not Applicable

**Experiment:**
Powder Flow

The objective of this experiment was to study the flow of powdered solids in a low-gravity environment.

Prior to flight, several simple plastic models, implemented with mechanical pistons, were designed to force solid particles through sized openings. Particles of interest included steel marbles, small plastic beads, and a powder similar to talcum. Preliminary ground-based investigations indicated that the particles remained packed together, preventing flow.

Although NASA scientists felt that an experiment of this nature merited investigation, basic research required to remedy the flow difficulties could not be completed within the pre-mission timelines. Thus, the experiment was not implemented into the actual Skylab mission student experiments.

<Note: Co-Investigator H. Politzer reported that (1) Sherhart was told that his experiments would be attempted on planes simulating low-gravity, and (2) Sherhart received reports about the progress of these experiments. (Politzer did not have copies of these reports.) The reports could not be located at this time.>

**Key Words:** Fluid Physics, Powders, Spheres, Powder Flow, Particle Transport, Particle Agglomeration, Fluid Management, Mass Transfer, Contained Fluids, Piston System, Sample Not Processed As Planned

**Number of Samples:** not applicable
**Sample Materials:** steel marbles, plastic beads, and fine powder similar to talcum
**Container Materials:** plastic

**Experiment/Material Applications:**
The experiment was to provide insight into the transfer and manipulation of fluids in space. Control of liquid fuels, coolants, lubricants, etc. continues to be an important aspect of low-gravity fluid management.

**References/Applicable Publications:**
(1) Skylab, Classroom in Space. NASA SP-101, 1977, pp. 92-93. (preflight)

(2) Input received from Co-Investigator H. Politzer, August 1989 and August 1993.

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**Experiment Origin:** USA
**Mission:** Skylab SL-3, Second Skylab Manned Mission
(This experiment was originally scheduled for Skylab SL-4, but because excess crew time was available on SL-3, the experiment was performed on SL-3.)
**Launch Date/Expt. Date:** September 1973 (month experiment was attempted)
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Skylab Student Project, High School Student Experiment, Skylab Manned Environment
**Processing Facility:** Gas/liquid chamber with viewing window (a piston provided an impulse to gas/liquid system)
**Builder of Processing Facility:** NASA Marshall Flight Center, Huntsville, Alabama

**Experiment:**
Liquid Motion (ED78)

When waves are created in a container of liquid on Earth, they take the shape of the container. In space, however, "Adhesion forces may hold the liquid in contact with its container... [or] the liquid... [may] assume a free floating condition." (4, p. 87)

The original objectives of this Skylab experiment were to (1) determine if the free floating condition (mentioned alone) could be achieved in the reduced gravity environment and (2) study the resulting wave motion of the free-floating liquid.

Reportedly, it became apparent that such an experiment would be difficult to design. For instance, the experimental apparatus would have to be constructed such that the water would be truly free-floating and at the same time be in contact with the drive mechanism. Further, it would be difficult to insure that the free-floating water would not break into droplets. Thus, the Skylab experimental objective was redefined. Instead, the response of a liquid/gas interface to a pseudo impulse was to be investigated.

The experimental apparatus was comprised (in part) of a cylindrical chamber containing (1) dyed water (90% of the volume) and (2) an air bubble (10% of the volume). An expandable diaphragm was used to seal the chamber at a pressure of one atmosphere (14.7 psia). A piston (which held the diaphragm in place), was configured with (1) a position retention mechanism, and (2) a piston release mechanism.

During the experiment, the piston was to be released. Since the Skylab atmosphere was at a pressure of 5 psia, "This action [should have] allowed the diaphragm to move, producing a sudden expansion of the gas bubble and creating waves at the surface of the water." (4, p. 88) The subsequent liquid/air motion was to be filmed via a 16 mm movie camera. (Spacecraft maneuvers were to be suspended during the experiment.)

Reportedly, when the piston was released, no impulse was observed. "The crewman commented that the pressure on the gas and liquid appeared to have been previously released....

"The crewman attempted to reset the piston and re-establish the pressure differential without success. He could then observe that the diaphragm was damaged. Fluid loss was not evident nor could a change in size of the gas bubble be detected. The hardware was not returned for analysis. It can be hypothesized that the low internal Skylab pressure and elevated temperature experienced before the first manned period could have caused an excessive differential pressure across the diaphragm resulting in its eventual rupture." (1, p. 12-85)

**Key Words:** Fluid Physics, Contained Fluids, Partially Filled Containers, Liquid/Gas Interface, Solid/Liquid Interface, Wave Propagation, Hydrodynamics, Bubbles, Surface Tension, Free Surface, Wetting of Container, Liquid Dynamic Response, Piston System, Gas Pressure, Payload Survivability, Thermal Environment More Extreme Than Predicted, Hardware Malfunction,

**Number of Samples:** one
**Sample Materials:** Liquid wetting agent (water) with dye. Within this liquid, an air bubble was inserted.
**Container Materials:** unknown

**Experiment/Material Applications:**
This research has applications related to basic flow examinations of gas/liquid interfaces, including wave propagation and surface tension effects.

Although other research applications were not cited in the applicable literature, the results of this liquid motion experiment would be of use when studying low-g fluid manipulation. For example, in the low-gravity environment, the response of a contained fluid to a disturbance is important, especially where propulsion systems are involved. If the fluid does free float, fuel may not be configured at the fuel-line input.

**References/Applicable Publications:**
(1) "Liquid Motion in Zero Gravity (ED78)." In MSFC Skylab Mission Report - Saturn Workshop, Skylab Program Office, George C. Marshall Space Flight Center, Alabama, NASA TM X-64814, October 1974, pp. 12-84 - 12-85. (post-flight)

(2) Chassay R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceeding of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(3) "Experiment ED78 - Liquid Motion in Zero Gravity." In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 6-66 - 6-69. (post-flight)

(4) Skylab, Classroom in Space. Edited by L.B. Summerlin, NASA SP-410, 1977, pp. 86-89. (post-flight)

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**Experiment Origin:** USA
**Mission:** Skylab SL-3, Second Skylab Manned Mission
**Launch Date/Expt. Date:** September 1973 (month experiment was completed)
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration, Skylab Manned Environment
**Processing Facility:** Not applicable
**Builder of Processing Facility:** Not applicable

**Experiment:**
Water Studies, Physics of a Water Globule

This Skylab SL-3 experiment was conducted after the completion of the Skylab Ice Melting Experiment (see Otto, Skylab SL-3 (Chapter 15)). The Ice Melting Experiment had left a globule of water attached to a retaining stick. At the suggestion of Dr. P. Grodzka, the Skylab crew added three substances to the globule of water.

Initially, a drop of soap solution was added to the surface of the water globule via a syringe. After the effects of the soap solution were observed, a small drop of grape juice was added to the surface. A soap solution/air mixture was then injected directly into the globule by a syringe.

It was reported that the surface of the globule vigorously retracted when touched by the soap or grape juice. "Vigorous fluid motion was seen by bubble and grape color movement for some moments after the addition of the surface active solutions. <Note the exact meaning of this sentence is unclear to the editors.> Astronaut Lousma observed that 'the water leaped out after the soap' when he approached the water globule with the soap solution. It was also observed that the globule oscillated or vibrated. Astronaut Lousma attributed the oscillations to small air currents." (1, p. 6)

After injection of the soap solution/air mixture into the globule, small bubbles formed. The astronaut stated that "...it appeared that the globule wanted to contain so many bubbles; i.e., at the end additional injections caused bubbles to come out of the water, it started spitting." (1, p. 6) It was concluded that the cluster of air bubbles in the soapy water had achieved a critical size.

No further information concerning this experiment could be located at this time.

**Key Words:** Fluid Physics, Hydrodynamics, Liquid Mixing, Liquid Injection, Gas Injection, Liquid Expulsion Through a Small Orifice, Droplet Creation Via a Syringe, Drops, Drop Oscillation, Drop Vibration, Droplet Agglomeration, Liquid Dynamic Response, Bubbles, Bubble Formation, Free Surface, Surface Tension, Foams, Liquid/Gas Interface

**Number of Samples:** One water drop, three fluid additions to the drop
**Sample Materials:** water, grape juice, soap
**Container materials:** not applicable

**Experiment/Material Applications:**
No discussion of the material application could be located in the published literature.

**References/Applicable Publications:**
(1) Bannister, T. C.: Skylab III and IV Science Demonstrations Preliminary Report. NASA TM X-64835, March 1974, p. 6. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

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**Experiment Origin:** USA
**Mission:** Skylab, SL-4, Third Skylab Manned Mission
**Launch Date/Expt. Date:** The experiment runs were completed during November 1973.
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration, Skylab Manned Environment
**Processing Facility:** Makeshift Floating Zone Apparatus
**Builder of Processing Facility:** Astronaut Gibson

**Experiment:**
Liquid Floating Zone (SD20 (TV101))

The objective of this Skylab demonstration was to study the dynamic behavior of a column of liquid suspended between two aluminum discs. Such columns of liquid (better known as liquid bridges) are used to model the containerless float zone solidification geometry.

During the third manned Skylab mission, the make-shift liquid bridge apparatus was constructed using onboard equipment. Two socket wrench extension rods were configured in parallel such that a small gap existed between the two rods. At the ends of the rods forming the gap, circular aluminum discs were attached. "The front surfaces of the discs were coated with grey tape which had been previously treated by immersion in acetone so as to reduce the contact angle with water. During the course of the experiments, the outer edges of the discs were coated with Krytox oil to prevent capillary wetting of water in that region." (1, p. 843). The rods were mounted in camera mounts such that the assembly was free to rotate smoothly.

"Rotation of the zones was performed manually by first winding... twine around the socket wrench and then withdrawing it at a uniform rate." (1, p. 843) Liquid bridges were created by (1) deploying a volume of fluid from a syringe on to each aluminum disc, (2) moving the discs close together to create one liquid volume, and (3) separating the discs to suspend the liquid into the desired column length. During 26 separate zone investigations, several water-based bridges were successfully formed, as bridge length, rotational direction, and bridge fluid were varied. Fluid additives such as rope particles aided internal fluid motion observance, while liquid soap was used to vary surface tension and viscosity. Video taping of most of these investigations was successfully achieved. Among the several observations of interest was the creation of a long, static, stable, unduloid bridge which exceeded the theoretical length limit for a right circular cylinder. When the liquid bridge was subjected to "...longitudinal vibrations of the end discs... standing waves occurred when the disc oscillation frequency coincided with a resonant frequency of the surface.

"When standing waves were produced, there was no transfer of liquid from one wave region to another inside the zone." (1, pp. 847-848) Rotation of the zone also resulted in some interesting observations. "For sequences involving the single rotation of only one bounding disc, the zone assumed an axisymmetric bottle shaped deformation possessing stability limits in general agreement with theory. For sequences involving equal rotation rates of both bounding discs, the zone becomes deformed in a non-axisymmetric fashion resembling a turning skipping rope [or C-mode] when the zone lengths exceed some value in the vicinity of... [(2/3)(pi)R]. Short zone lengths do not generally develop the rotating C-mode but do demonstrate capillary wave resonances presumably arising from nonparallel disc alignment." (2, p. 6)

**Key Words:** Fluid Physics, Liquid Columns, Liquid Bridges, Liquid Bridge Stability, Liquid Dynamic Response, Liquid Column Rupture, Hydrodynamics, Liquid Vibration, Axial Oscillations, Oscillation Frequency, Resonant Frequency, Standing Waves, Rotating Fluids, Sample Rotation, Liquid Expulsion Through a Small Orifice, Solid/Liquid Interface, Liquid/Gas Interface, Free Surface, Surface Tension, Viscosity, Wetting, Contact Angle, Capillary Forces, Hydrostatic Pressure, Coated Surfaces, Containerless Processing Applications, Tracer Particles

**Number of Samples:** A total of 26 experimental runs were realized.
**Sample Materials:** Most of the zones were water based. Additives such as rope particles and soap were used for some runs. Soap solution and soap foam liquid bridges were also formed.
**Container Materials:** not applicable

**Experiment/Material Applications:**
These liquid bridge experiments demonstrated some of the fluid dynamic characteristics of reduced gravity bridge formation and rotation. Such bridge formation and rotation characteristics are of interest for floating zone crystal growth. Float zone materials processing is a popular method of growing semiconductor crystals in industry. However, the maximum length of the zone in 1-g is limited by the balance of hydrostatic pressure with the liquid surface tension.

**References/Applicable Publications:**

(1) Carruthers, J. R.: Studies of Liquid Floating Zones in SL-IV, the Third Skylab Mission. In Marshall Space Flight Center Proceedings of the Third Space Processing Symposium on Skylab Results, Vol. 2, June 1974, pp. 837-856. (post-flight)

(2) Carruthers, J. R., Gibson, E. G., Klett, M. G., and Facemire, B.: Studies of Rotating Liquid Floating Zones on Skylab IV. American Institute of Aeronautics and Astronautics, 10th Thermophysics Conference, Denver, Colorado, May 27-29, 1975, AIAA Paper #75-692, 8 pp.

(3) Chassay, R. P. and Schwaniger, A.: Low G Measurements at NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(4) TV101-Liquid Floating Zone. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-4 - 7-10. (post-flight)

(5) Naumann, R. J. and Herring, H. W.: Experiment TV101, Liquid Floating Zone. In Materials and Processing in Space: Early Experiments, NASA SP-443, 1980, pp. 79-81. (post-flight)

(6) Bannister, T. C.: Science Demonstrations on Skylab in the Material Processing Area. In Proceedings of the Third Space Processing Symposium on Skylab Results, April 30-May 1, 1974, Marshall Space Flight Center, Huntsville, Alabama, Vol. 1, June 1974, pp. 491-505. (post-flight)

(7) Bannister, T. C.: Skylab III and IV Science Demonstrations. NASA TM X-64835, March 1974, pp. 6-8. (post-flight)

(8) Naumann, R. J. and Mason, E. D.: Liquid Floating Zone. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 40-41. (post-flight)

(9) Liquid Floating Zone (SD20-TV101). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-89 - 12-90.

(10) Input received from Principal Investigator John Carruthers, June 1993.

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**Experiment Origin:** USA
**Mission:** Skylab, SL-4, Third Skylab Manned Mission
**Launch Date/Expt. Date:** January 1974 (month experiment was performed)
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration
Skylab Manned Environment
**Processing Facility:** Two- and three-dimensional wire shapes which supported liquid films; syringe; water and soap solutions
**Builder of Processing Facility:** Skylab Astronaut, Commander Carr

**Experiment:**
Liquid Films (SD22 (TV103))

On Earth, the rupture of liquid films is primarily dependent on (1) gravity-induced fluid flow drainage and (2) gravity-independent surface tension fluid characteristics. During this Skylab-4 science demonstration, the effect of reduced gravity on the formation and rupture of liquid films was investigated.

During the mission, a Skylab crewman (Commander Carr) fashioned the following two-dimensional and three-dimensional film-confining frames from safety wire: (1) an expandable loop, (2) an expandable rectangle, (3) a 1-inch-diameter hoop, (4) an equilateral tetrahedron, and (5) a three-dimensional cube.

Expandable Loop/Rectangle Experiments

Two different types of films (composed of (1) plain water and (2) soap solution) were formed on the expandable loop and rectangle by (1) delivering a globule of liquid via a syringe onto the frame and (2) expanding the frame until a thin film stretched across the wire boundaries. The following was reported:

(a) One-ml of water placed on the expandable loop was stretched to 7 cm diameter during the Skylab investigation. On Earth, similar water films could not be produced by this method.

(b) One-ml of the soap solution placed on the expandable loop was stretched to approximately 15 cm diameter during the Skylab investigation. On Earth, similar soap expansion diameters were produced.

(c) A 1/8-ml soap solution placed on the expandable rectangle was stretched to a 2 cm by 20 cm rectangle during the Skylab investigation. On Earth, soap films with similar areas as this one could be obtained.

Films on the Other Wire Frames

Prior to forming films with the 1-inch-diameter hoop, equilateral tetrahedron and three-dimensional cube, the wire frames were secured in the weightless environment to a piece of adhesive tape. At the initiation of the experiment, the hoop frame was removed from the tape, immersed into a container holding soap solution, withdrawn from the solution, and restuck to the tape. (Shaking the liquid from the frame into the container resulted in thinner and thinner films.)

During the next step of the experiment, the tetrahedron frame was removed from the tape. This action disturbed the hoop-shaped film which had not yet ruptured. Nevertheless, the reduced gravity hoop film was maintained for 1.5 minutes. (A similar Earth film would probably not have lasted for over a minute.)

The tetrahedron film was formed in the same manner as the hoop (immersing the frame and then drawing the frame from the liquid). The tetrahedron film was described by the crewman "...as having films on all four faces. Although the video tape picture... [was] not clear enough to be certain, it... [was] assumed that this comment was unintentional since it is mathematically impossible." (1, p. 10)

The cube was immersed and withdrawn from the soap solution several different times during the Skylab experiment and various film configurations resulted. Although one lasted approximately 5 seconds, the others were on the order of one minute. (Similar Earth-created films have a lifetime of only a few seconds.) "Of the many "stable" [Skylab] film configurations, the one first formed for the cube was one having a smaller cube in the center held in position by films extending from the wire cube. Having a rather high surface tension, this minimal surface ruptured quickly, taking on one configuration after another until finally all the liquid was concentrated in one film on one face." (6, p. 14)

It was concluded that some of the films created in the low-gravity environment had longer times-to-rupture. Thus, enhanced solidification of films attached to boundaries might be feasible in the space environment.

**Key Words:** Fluid Physics, Liquid Films, Liquid Film Rupture, Thin Films, Surface Tension, Free Surface, Fluid Drainage, Liquid/Gas Interface, Solid/Liquid Interface, Liquid Transfer, Liquid Expulsion Through a Small Orifice, Photographic Difficulties

**Number of Samples:** Five wire frames were fashioned from Skylab safety wire. However, it is not clear how many times each of these frames was employed to produce a film.
**Sample Materials:** (1) plain water and (2) 40 parts water, 1 part Skylab liquid shower soap (Miranol Jem concentrate).
**Container Materials:** not applicable

**Experiment/Material Applications:**
Reportedly, the creation and solidification of stable liquid films in the reduced gravity environment may eventually lead to (1) new semiconductor materials or (2) new foamed materials.

**References/Applicable Publications:**

(1) Darbro, W.: Liquid Film Demonstration Experiment Skylab SL-4. NASA TM-X-64911, January 1975, 28 pp. (post-flight)

(2) Darbro, W.: An Example of Branching in a Variational Problem--Shape of Liquid Suspended From Wire in Zero Gravity. In American Journal of Physics, Vol. 46, November 1978, pp. 1183-1184. (post-flight)

(3) Chassay, R. P. and Schwaniger, A.: Low G Measurements at NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama p. 9-1. (acceleration measurements on Skylab)

(4) Naumann, R. J. and Herring, H. W.: Experiment TV103, Liquid Films. In Materials Processing in Space: Early Experiments, NASA SP-443, 1980, p. 83. (post-flight)

(5) TV103- Liquid Films. In MSFC Skylab Corollary Experiments System Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-14 - 7-17. (post-flight)

(6) Bannister, T. C.: Skylab III and IV Science Demonstrations Preliminary Report. NASA TM X-64835, March 1974, pp. 13-15. (post-flight)

(7) Naumann, R. J. and Mason, E. D.: Liquid Films. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, p. 43. (post-flight)

(8) Liquid Films (SD22-TV103). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-90. (post-flight)

(9) Input received from Principal Investigator W. Darbro, 1988.

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**Experiment Origin:** USA
**Mission:** Skylab, SL-4, Third Skylab Manned Mission
**Launch Date/Expt. Date:** The SL-4 mission took place during November 1973-February 1974. This experiment was not performed because of insufficient crew time.
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration, Skylab Manned Environment
**Processing Facility:** Transparent container with water and seltzer tablet
**Builder of Processing Facility:** Not Applicable

**Experiment:**
Effervescence (SD18 TV113)

The objectives of this Skylab SL-4 experiment were to (1) observe low-gravity bubble formation during an effervescent reaction and (2) determine if the reaction is self-quenching as the bubble grows.

During the mission, a member of the Skylab crew was to (1) fill a transparent container with water, (2) insert an Alka-Seltzer tablet into the water, and (3) observe and film the reaction using a TV camera.

It was reported that the experiment was not performed because of insufficient crew time.

No further information concerning this experiment could be located at this time.

**Key Words:** Fluid Physics, Effervescence, Bubbles, Bubble Formation, Liquid/Liquid Interface, Liquid Transfer, Contained Fluids, Solid/Liquid Interface, Reaction Kinetics, Flammability, Sample Not Processed As Planned

**Number of Samples:** One experiment was planned.
**Sample Materials:** water, Alka-Seltzer tablets
**Container Materials:** transparent container (specific material unspecified)

**Experiment/Material Applications:**
This experiment had research applications in the area of reduced-gravity flammability studies.

**References/Applicable Publications:**

(1) TV113 - Effervescence. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1971, pp. 7-45.

(2) Effervescence. In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-88 - 12-89.

(3) Input received from Principal Investigator A. R. Hibbs, June 1993.

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**Experiment Origin:** USA
**Mission:** Skylab, SL-4, Third Skylab Manned Mission
**Launch Date/Expt. Date:** The SL-4 mission took place during November 1973-February 1974. However, this experiment was not performed because of insufficient crew time.
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration, Skylab Manned Environment
**Processing Facility:** Wire frames to which water, soap solutions or molten soap was to be applied to simulate the formation of lenses.
**Builder of Processing Facility:** Not Applicable. The wire frames were to be fashioned by a crewman on orbit.

**Experiment:**
Lens Formation (TV116) (SD23)

When a lens is created from a liquid in a reduced gravity environment, its shape should be dictated primarily by surface tension forces. The objective of this Skylab SL-4 experiment was to demonstrate the formation of such surface-tension shape-dependent lenses.

The lenses were to be formed on wire loops (of varying sizes) which had been fashioned from on-board safety wire. Although descriptions in References (1) and (2) were not clear, it appears that three different lens fluids were to be used: (1) pure water, (2) soap solutions, and (3) melted bar soap.

A discussion of how the water or soap solution was to be applied to the wire loop was not detailed. However, once the water or soap solution lens had been formed, (1) its shape was to be documented via an edge-view photograph and (2) its optical properties (such as focal length) were to be documented via photographs taken through the lens.

For the bar soap lens, 1 cubic inch of solid bar soap was to be melted in the Skylab food tray. Then, a 1-inch-diameter wire loop was to be dipped into the molten soap. It was expected that the (approximately) 1/2 inch thick soap lens would harden when cooled and could be preserved for post-mission analysis. If time permitted, the crewman was to observe the optical properties of a water prism confined within an appropriate wire frame.

It was reported that none of these lens formation experiments were performed on SL-4 because of insufficient crew time.

**Key Words:** Fluid Physics, Lens Formation, Liquid Films, Thin Films, Fluid Drainage, Surface Tension, Free Surface, Meniscus Shape, Solid/Liquid Interface, Liquid/Gas Interface, Optical Characterization, Sample Not Processed As Planned

**Number of Samples:** The number of expected samples was not detailed.
**Sample Materials:** water, soap solutions, melted bar soap
**Container Materials:** safety wire

**Experiment/Material Applications:**
The specific applications of this low-gravity research were not detailed in the available publications. However, it is suspected that the research may be applicable to superior lens formation endeavors.

**References/Applicable Publications:**
(1) TV116-Lens Formation. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-50.

(2) Lens Formation (SD23-TV116). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, August 1974, pp. 12-90 - 12-91.

(3) Input received from Principal Investigator R. Kroes, May 1991 and August 1993.

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**Co-Investigator(s):** None
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**Experiment Origin:** USA
**Mission:** Skylab, SL-4, Third Skylab Manned Mission
**Launch Date/Expt. Date:** November 1973-February 1974
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration, Skylab Manned Environment
**Processing Facility:** Large hypodermic syringe (The M113 electrolyte dispenser) mounted in a portable vise.
**Builder of Processing Facility:** Not Applicable. The experiment employed onboard materials as makeshift test beds.

**Experiment:**
Cloud Formation (TV118-SD29)

"Terrestrial expansion chambers, for the study of submicron (0.1 to .1$\mu$) nuclei, rely on the assumption that the most active nuclei introduced into the chamber grow at the same rate so that after a finite length of time the spheres of water that are formed can be photographed. However, some nuclei are not as active as others and due to gravitational fallout effects they do not have enough time to grow to spheres of water which can be photographed.

"Since the time of observation is limited in the terrestrial expansion cloud chamber due to the gravitational fallout effects and other effects due to convection (wall effects), the use of an expansion cloud chamber in zero-gravity allows the researcher to examine in more detail the life cycle and dynamics of all the nuclei introduced into the chamber." (7, p. 5)

This Skylab SL-4 cloud formation experiment was designed to study the history and dynamics of an expansion cloud.

A large hypodermic syringe containing a small amount of water was employed for the investigation. During the mission, a sample of cabin air was drawn into the syringe and compressed with the syringe plunger. A few seconds later, (after the gas had reached thermal equilibrium) the syringe was depressurized by releasing the plunger. In this moist environment, water drop creation, motion, coalescence, and lifetime were to be observed.

During similar (typical) terrestrial reference experiments, drops were formed in an expansion chamber on nuclei present in the air after the air sample was depressurized (as discussed above). These water droplets were then detected by a light directed into the chamber at 120 degrees from the observer's position.

During the Skylab experiments, droplets were observed on the Skylab syringe chamber walls, but a cloud was not detected. Reportedly, although data were to be collected using a 16 mm camera, no data were recorded because the cloud was not observed.

During the Skylab mission debriefing, astronauts reported that (1) they attempted to form a cloud by expansion approximately 50 times (but were unsuccessful) and (2) the tests were performed with both a dry and wet syringe chamber.

It was speculated that a cloud was not observed in the chamber because (1) there were too few particles in the cabin air to serve as nuclei for droplets, or (2) insufficient backlighting of the syringe did not allow visibility of the droplets.

Because Skylab also had onboard Experiment T003, the In-Flight Aerosol Analysis Experiment, it was possible to further speculate if the cloud was not observed because there were too few particles in the air acting as nuclei. Reportedly, the objective of Aerosol Analysis Experiment was to measure the Skylab "...aerosol particulate matter concentration and distribution during the... mission as a function of time and location; and return the collected aerosol particles and the logged data for postflight analysis." (1, p. 4-29) <Note: Further details of the Aerosol Analysis Experiment are not contained in this data base as it was not considered a fluids or materials processing experiment.>

In conjunction with this Aerosol Analysis Experiment, the astronauts reported that the Skylab atmospheric environment aerosol particle counts were low each time they took a reading. The T003 Principal Investigator reported that the experiment "...only sampled particles in three size ranges: $1\mu$ to $3\mu$, $3\mu$ to $9\mu$, and $9\mu$ to $100\mu$, and that in the $1\mu$ to $3\mu$ range he recorded only 4,000 particles/$ft^3$--- these being human skin flakes. Since human skin flakes are not hydroscopic, they are not a good nuclei source; however, since there were no instruments to measure the submicron size particles on-board, we cannot say that they were not in the cabin air. Since the Skylab 4 environmental control system relied on a wet wick cold plate unit and other units for humidity and temperature control, it is possible that many particles were caught by these systems. Since data from T003 indicated that the spacecraft environment was classified equivalent to a 10,000 class clean room, it was decided to try the demonstration [on Earth] in a much cleaner environment than the spacecraft (4,000 class clean room operating at 37% RH) to see if a cloud could be formed under these conditions." (7, p. 6)

During the terrestrial demonstration a cloud formed, although it was difficult to see even when a bright light source was employed (a microscope illuminator). (The Skylab experiments did not have such a bright light source.)

Thus, while it was not clear why the cloud was not observed in the chamber, insufficient atmospheric particles as nuclei and/or insufficient backlighting of the chamber were thought to be the source(s) of the difficulty.

**Key Words:** Fluid Physics, Atmospheric Applications, Clouds, Expansion Cloud, Drops, Drop Formation, Nucleation, Drop Coalescence, Droplet Dispersion, Particle Motion, Particle Growth, Convection, Gas Pressure, Contained Fluids, Wall Effect, Thermal Equilibrium, Liquid/Vapor Interface, Piston System, Atmospheric Applications, Illumination Difficulties

**Number of Samples:** Unclear. It appears there was one experimental setup. The experiment was attempted over 50 times.
**Sample Materials:** air and water
**Container Materials:** transparent, glass hypodermic syringe

**Experiment/Material Applications:**
This atmospheric cloud physics experiment was designed to demonstrate the formation of cloud water drops in low gravity. Subsequent motion of the particles due to thermal gradients was also of interest. (See the above experiment summary for more information.)

**References/Applicable Publications:**
(1) TV118-Cloud Formation. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, pp. 7-57 - 7-59 and pp. 4-29 - 4-39, September 1974. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements)

(3) Vaughan, O. H. and Hill, C. K.: Drop Coalescence in Zero-Gravity Environment of Skylab IV. American Meteorological Society, Bulletin, Vol. 55, September 1974, pp. 1127-1130. (post-flight)

(4) Hung, R. J., Vaughan, O. H., and Smith R. E.: A Zero-Gravity Demonstration of the Collision and Coalescence of Water Droplets. In American Institute of Aeronautics and Astronautics and American Geophysical Union Conference on Specific Experiments of Skylab, Huntsville, Alabama, October 30-November 1, 1974, AIAA Paper #74-1256, 8 pp. (post-flight)

(5) Cloud Formation (SD29-TV118). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, pp. 12-91 - 12-92. (post-flight)

(6) Input received from Principal Investigator O. Vaughan, August 1989, 1990, and 1993.

(7) Quick Look Report for Science Demonstrations Performed During the Skylab 4 Mission: (I) TV-107 Fluid Mechanics/Coalescence; (II) TV-118 Cloud Formation. March 22, 1974, 6 pp. (post-flight, supplied from Principal Investigator to C. Winter, 5/93)

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**Experiment Origin:** USA
**Mission:** Skylab, SL-4, Third Skylab Manned Mission
**Launch Date/Expt. Date:** November 1973-February 1974
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration (an extension of Dunlap's Student Experiment Performed on SL-3 (see this chapter)), Skylab Manned Environment
**Processing Facility:** The series of experiments employed (1) onboard medical-type syringes, (2) drinking straws, (3) the teflon coated flat surface of a spider cage, and (4) other associated items.
**Builder of Processing Facility:** Not applicable. Experiments employed onboard equipment as make-shift experimental testbeds.

**Experiment:**
Water Drop and Fluid Mechanic Series (SD9 Part 1,2) (TV 107)
Oscillation, Rotation, Collision, Grazing and Coalescence of Water Droplets

"One of the most interesting phenomena of fluids which can be studied in zero-gravity is the various modes of oscillations, and other phenomena which a fluid will exhibit when perturbed. In spite of research efforts in cloud physics covering a span of 25 years, much remains to be learned of raindrop collision, oscillation, and breakup. Data from field surveys show that beyond a rainfall intensity of about 100 mm/hr the median raindrop size does not increase, but remains constant or even decreases slightly. This curious behaviour has not been explained, but is probably determined by the oscillations leading to drop breakup. Nearly all the work in the behaviour of large drops has been done in vertical wind tunnels and the drops in free-fall have been supported by a stream of air equal to the fall speed of the drop. The use of the zero-gravity environment can give the earth-based researcher an opportunity to study drop dynamics that are controlled only by surface tension and drop inertia effects. The use of zero-gravity also allows the researcher to study the conversion of kinetic energy of impact into the kinetic energy of a capillary wave which will travel over the outer surface of the drop and produce a satellite drop or drops." (12, p. 2)

This Skylab SL-4 experiment consisted of several investigations designed to observe various oscillation modes of water drops.

Free oscillation of a water droplet was achieved by suspending the free-floating drop between two soda straws, and then plucking the straws outward from the water. Oscillation modes (deforming the drop transversely, then longitudinally) were observed. Subsequent decay of the motion caused by internal damping was noted as the droplet returned to its original shape.

Oscillation modes of a water droplet attached to a flat teflon surface were achieved by inserting a drinking straw into the center of the drop and pulling upward. The various modes of oscillation were observed.

Forced rotation of free droplets was studied by first suspending a free floating drop, and then with a string, repeatedly skimming the water surface in a circular motion. The high rotational speed produced unstable oscillations which resulted in "dog bone" or "dumb bell" type shapes that eventually divided into two separate drops.

The collision and coalescence of drops was demonstrated by holding a free drop stationary and ejecting a second drop toward it. The drops (one of which was colored red and the other colored black) experienced some mixing after coalescence. The drops bounced apart rather than coalesced if the ejection velocity was not high enough or if there was a like electrical charge on the drops. One drop ejected toward a second drop with the intent to barely graze the stationary drop, resulted in capture of the moving drop and subsequent mixing.

Many other investigations are described in Reference (3) (listed below).

Reportedly, (1) approximately 172,500 frames of 16 mm film were exposed showing droplet motion during these Skylab investigations and (2) most of the film was of excellent quality. Discussion of the Skylab results as they apply to the theory related to the oscillation of free floating liquid droplets is provided in Reference (5). Discussion of the results as they apply to theory related to the collision and coalescence of water droplets is provided in Reference (9).

Additional information concerning these experiments can be found in several of the references below.

**Key Words:** Fluid Physics, Drops, Drop Dynamics, Hydrodynamics, Drop Oscillation, Drop Rotation, Fluid Oscillation, Oscillation Modes, Rotating Fluids, Liquid Dynamic Response, Fluid Motion Damping, Capillary Waves, Droplet Collision, Surface Tension, Free Surface, Surface Charge, Drop Coalescence, Liquid Mixing, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Coated Surfaces, Atmospheric Applications, Liquid/Gas Interface

**Number of Samples:** The exact number of investigations is unclear.
**Sample Materials:** Water colored with grape drink, strawberry drink, and/or marker pen ink
**Container Materials:** not applicable

**Experiment/Material Applications:**
Drop dynamics investigations are important for many scientific fields including atmospheric microphysics, nuclear physics, astrophysics, fluid mechanics, etc. These investigations lend insight to the study of nuclear deformation energy surfaces, microphysical droplet-droplet interactions, etc. (See the experiment summary above for more information.)

**References/Applicable Publications:**
(1) Vaughan, O. H.: Skylab Fluid Mechanics Simulations: Oscillation, Rotation, Collision, and Coalescence of Water Droplets Under Low-Gravity Environment. In the 8th Conference on Space Simulation, 1975, pp. 563-574 (See N76-11113 02-09). (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements)

(3) TV107-Fluid Mechanic Series. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-28 - 7-33. (post-flight)

(4) Bannister, T. C.: Skylab III and IV Science Demonstrations Preliminary Report. NASA TM X-64835, March 1974, p. 20. (post-flight, short summary)

(5) Vaughan, O. H., Smith, R. E., Hung, R. J., and Wu, S. T.: An Analysis of Oscillations of a Water Droplet Under Low Gravity Conditions. Presented at the JPL Conference "International Colloquium on Drops and Bubbles-Their Science and the Systems They Model," August 28-30, 1974, JPL and California Institute of Technology, Pasadena, California, p. 12. (post-flight)

(6) Vaughan, O. H. and Hill, C. K.: Drop Coalescence in Zero-gravity Environment of Skylab IV. Bulletin American Meteorological Society, Vol. 55, No. 9, September 1974, pp. 1127-1130. (post-flight)

(7) Naumann, R. J. and Mason, E. D.: Fluid Mechanics. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 46-47. (post-flight)

(8) Gibson, E. F.: Skylab Fluid Mechanics Demonstration. International Colloquium on Drops and Bubbles, California Institute of Technology, Pasadena, California, August 28-30, 1974.

(9) Hung, R. J., Vaughan, O. H., and Smith, R. E.: A Zero-Gravity Demonstration of the Collision and Coalescence of Water Droplets. AIAA Paper #74-1256, 1974.

(10) Bannister, T. C. and Bourgeois, S. V.: Liquid Drop Behavior in Weightlessness from Skylab. International Colloquium on Drops and Bubbles, California Institute of Technology, Pasadena, California, August 28-30, 1974.

(11) Input received from Principal Investigator O. Vaughan, August 1989, 1990, and 1993.

(12) Quick Look Report for Science Demonstrations Performed During the Skylab 4 Mission: (1) TV-107 Fluid Mechanics and Coalescence; (II) TV-118 Cloud Formation. March 22, 1974, 6 pp. (post-flight, supplied from Principal Investigator to C. Winter, May 1993)

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**Experiment Origin:** USA
**Mission:** Skylab, SL-4, Third Skylab Manned Mission
**Launch Date/Expt. Date:** Although the SL-4 mission took place during November 1973-February 1974, this experiment was not performed because of insufficient crew time.
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration, Skylab Manned Environment
**Processing Facility:** Taped high frequency signal aimed at reflecting surface
**Builder of Processing Facility:** Not applicable. Experiments employed onboard equipment as makeshift test beds.

**Experiment:**
Acoustic Positioning (SD24(TV114))

This Skylab SL-4 experiment was designed to position and control water drops using sound wave acoustic pressure. The experiment setup consisted of (1) a tape recorder, (2) a speaker, (3) a reflecting surface, and (4) a water-filled hypodermic syringe.

Two tones (2000 and 3000 Hz) were recorded on tape prior to the flight. During the experiment, the tones were to be radiated through the speaker toward the reflecting surface. The combination of the radiated tones and the subsequent reflection was expected to produce an acoustical standing wave about 6 inches from the tape recorder and/or reflector. Water drops were to be placed at this standing wave location using the hypodermic syringe.

It was anticipated that the drops would move to an antinodal point and coalesce into a large globule. Water drop manipulation as a function of sound energy was to be examined. A 16 mm camera was available to film the event.

Reportedly, the experiment was not completed because of insufficient crew time.

**Key Words:** Fluid Physics, Acoustic Positioning, Acoustic Levitation, Standing Waves, Drops, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Drop Coalescence, Drop Stability, Containerless Processing Applications, Liquid/Gas Interface, Sample Not Processed as Planned

**Number of Samples:** not applicable
**Sample Materials:** water drops
**Container Materials:** not applicable

**Experiment/Material Applications:**
These investigations sought to demonstrate that drops could be acoustically controlled or positioned for study. Such study is more complicated to achieve on Earth because high acoustic levels must be employed. In the space environment, a much lower acoustic level is required to manipulate the drop. The experiments also pertained to containerless handling of space fluids (a first step toward crucible-free space processing).

**References/Applicable Publications:**

(1) TV114-Acoustic Positioning. In MSFC Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-46. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements on Skylab)

(3) Acoustic Positioning (SD24-TV114). In MSFC Skylab Mission Report-Saturn Workshop, NASA TM X-64814, October 1974, p. 12-91. (post-flight)

(4) Input received from Principal Investigator O. Vaughan, August 1989, 1990, and 1993.

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**Experiment Origin:** USA
**Mission:** Apollo-Soyuz Test Project (ASTP)
**Launch Date/Expt. Date:** July 1975
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration, ASTP Manned Environment (Command Module)
**Processing Facility:** Transparent cube into which liquid was injected through a syringe.
**Builder of Processing Facility:** Lockheed Missiles and Space Company, Huntsville, Alabama

**Experiment:**
Spreading of Liquids

The objective of this ASTP science demonstration was to study the spreading of fluids over solid and liquid surfaces. In a low-gravity environment, liquid shape change and liquid spreading rate can be examined independent of overwhelming terrestrial gravity effects.

Reportedly, syringes filled with either water (mixed with red dye) or oil (mixed with blue dye) were to be used to inject a small amount of fluid onto the inside, bottom surface of a transparent cube. As the fluid spread across the bottom and up the sides of the cube, more fluid was to be injected from the syringe onto the inside, bottom surface. One of the interior faces of the box had been treated to act as a non-wetting surface; the top of the box had been treated to insure the fluid remained inside the cube. <Note: The anti-wetting material treatment was not specified in the available publications.> A small, anvil-shaped solid was attached to the bottom of the cube to permit the study of fluid-spreading over sharp corners.

<Note: It is not clear (1) how many experiment runs were actually achieved during the mission or (2) what the specific fluid media of each of these runs was to be. Although the Principal Investigator reported that 5-10 runs were anticipated and that all these runs were to be photographed, it appears (Reference (1)) that only one run was actually attempted during the mission.>

Specifically, the Principal Investigator reported that the selected syringe "...never worked properly and was "frozen" when the astronaut first tried it. Therefore the few drops extracted were thrown randomly into the container rather than gently as planned. Thus inertia rather than surface tension determined where fluids went which destroyed [the] purpose of the experiment." (3)

<Note: In addition to the Principal Investigator's summary (above) it was reported (Reference (1)) that "Only one box was photographed during the ASTP mission. The photographs clearly show that the oil placed at the center of the bottom surface quickly spread to the corners and climbed the vertical walls. The oil accumulated in the corners of the cube in a manner that would minimize the surface area. When red water was added to the oil surface, repulsion of oil and water was followed by an equilibrated configuration of water droplets surrounded by an oil film." (1, p. 31-5) Although it is not clear, it appears that this information from Reference (1) describes in more detail what happened during the attempt described by the Principal Investigator (when the drops were thrown randomly into the container).>

No other comments were made in the available documentation which described fluid flow (if any) over the anvil, or the effectiveness of the non-wetting surfaces.

**Key Words:** Fluid Physics, Liquid Spreading, Solid/Liquid Interface, Liquid/Gas Interface, Liquid/Liquid Interface, Wetting of Container, Non-Wetting of Container, Meniscus Shape, Surface Tension, Interfacial Energy, Contact Angle, Free Surface, Coated Surfaces, Liquid Expulsion Through a Small Orifice, Drops, Droplet Creation Via a Syringe, Surface Tension Minimum, Drop Formation, Hardware Malfunction

**Number of Samples:** one container
<Note: It appears that multiple spreading trials were to be performed in the single container.>
**Sample Materials:** oil (mixed with blue dye), water (mixed with red dye)
**Container Materials:** unclear, possibly Lexan$^{TM}$

**Experiment/Material Applications:**
Fluid physics information derived from this science demonstration could be used to understand the effects of gravity on fluid spreading. Fluid spreading and uniformity characteristics are important during coating applications, soldering, brazing, lubrication, lithography painting, etc.

**References/Applicable Publications:**
(1) Snyder, R. S., Clifton, K. S., Facemire, B., Whitaker, A. F., Grodzka, P. G., and Bourgeois, S.: Science Demonstrations. In Apollo-Soyuz Test Project, Marshall Space Flight Center, February 1976, pp. 31-1 - 31-9. (this paper is not the same as the special publication entitled the Apollo Soyuz Test Project)

(2) Naumann, R. J. and Mason, E. D.: Liquid Spreading. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, p. 73. (post-flight)

(3) Input received from Principal Investigator S. V. Bourgeois, April 1988 and July 1993.

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**Experiment Origin:** USA
**Mission:** Apollo-Soyuz Test Project (ASTP)
**Launch Date/Expt Date:** July 1975
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** ASTP Docking Module Payload
**Processing Facility:** Multipurpose Electric Furnace M512 (gradient furnace) located in the ASTP docking module. (The furnace was similar to the Skylab M-518 furnace.)
**Builder of Processing Facility:** Westinghouse, Pittsburgh, Pennsylvania

**Experiment:**
Surface Tension Induced Convection (MA-041)

Fluid mixing in a liquid melt due to density differences created by thermal or compositional gradients is suppressed under low-gravity conditions. However, there are other fluid flow mechanisms which can produce convective flow (see for example early space experiments by Poorman, SL-2 (Chapter 14)). One of these mechanisms, surface-tension-driven convection (the Marangoni effect), may become a major source of convective flows in the space environment.

This Apollo-Soyuz Test Project (ASTP) Experiment was designed to investigate if a step-like compositional variation in a liquid metal would produce convection in wetting and non-wetting ampoules under low-gravity conditions. The specific objective of the experiment was to study surface-tension-driven convective flows present during the diffusion of gold in lead. It was expected that if no convective stirring effects were acting, a normal, diffusion-controlled concentration profile would be detected.

"Surface tension induced convection could be expected if the specimens were not in contact with the ampoule wall. However, if the specimens touch the walls, the interface tension between... [a section of Pb-Au] and pure Pb should not be able to create a convective effect according to the fluid dynamic theories [i.e., no slip boundary conditions] by which zero flow is expected at the walls." (1, p. 368)

Prior to the ASTP mission, six Pb/Pb-0.05 at.% Au diffusion-couple samples were created by pressure bonding the Pb-Au alloy to the Pb material. The samples were placed in three cartridges (two samples per cartridge).

During the mission, the M512 Materials Processing Facility was used to process the materials. Three samples were located in the hot zone of the furnace (923 +/- 10 K) and three in the intermediate zone of the furnace (723 +/- 50 K). This sample configuration allowed the investigation of diffusion at two different temperatures. Two of the three samples in each zone were contained in graphite-lined stainless steel crucibles (not wetted by lead). The graphite crucibles were oriented in opposite directions (with respect to the temperature gradient) to investigate whether melting or solidification would affect the Au distribution. The remaining samples were contained in mild steel crucibles (wetted by lead) and configured with the Pb-Au section aligned so that it melted first.

During the experiment, the samples were heated, melted, and solidified using rates of 2.5 cm/min (923 K temperature zone) and 6.0 cm/min (723 K temperature zone). (Graphs describing the thermal history of the samples are included in Reference (1).) It was reported that "...because of a malfunction in the helium quenching process, the SF [space flight] specimens stayed molten longer than was initially anticipated." (1, p. 382)

The acceleration environment of the samples during the mission was of interest to the investigators because accelerations could affect the diffusive/convective nature of the samples. Reportedly, "the average acceleration of the SF specimens during the experiment was less than 0.0981 $cm/sec^2$. Maximum acceleration, which was caused by firing of the reaction control system thrusters, was less than 5.886 $cm/sec^2$ for time periods of 15 milliseconds." (1, p. 372)

Post-flight analysis of the low-gravity samples illustrated that "... the gold moved about one-half the distance in the low temperature ampoule as in the high temperature ampoule, as would be expected from diffusion theory. Some distortion of the diffusion profile was observed near the [ampoule] walls, and this distortion seems to depend on the sample orientation in the furnace. This suggests that either volume change or segregation effects during solidification may have been partially responsible for this effect. No difference was observed between samples processed in the [mild] steel and graphite[-lined] ampoules. Apparently the melt did not wet the steel as expected. (4, p. 56) <Note: Presumably, the meaning of "No difference" in the above quote means that the diffusion profile near the wall of both the "wetting" and non-wetting ampoules was distorted.>

Ground-based tests were performed to duplicate the low-gravity experiment (see Reference (1) for details). Reportedly, "The ground control samples showed almost complete mixing when processed in the vertical destabilizing orientation (with the heavier gold alloy at the top). In the stable configuration (gold alloy at the bottom), substantial, but not complete, mixing was observed, probably because of the radial gradients.

"The investigators conclude that Marangoni Convection offers the best explanation for the observed distribution [specifically at the walls], and therefore the 'no-slip' boundary condition apparently does not apply to nonwetting materials in low-g [see Reference (1) for detailed discussion]. However, a detailed analysis was not presented to support this assertion.... [T]he bulk migration of Au in the Pb appeared to be almost completely diffusion controlled in the space processed samples. No trace of gold was found away from the diffusion region, indicating negligible mixing resulted from the combination of vehicle accelerations and the various nongravitational effects." (4, p. 56)

**Key Words:** Fluid Physics, Melt and Solidification, Surface Tension-Driven Convection, Marangoni Convection, Binary Systems, Interdiffusion, Diffusion, Diffusive Mass Transfer, Thermal Gradient, Solutal Gradients, Liquid Mixing, Wetting of Container, Non-Wetting of Container, Hydrostatic Pressure, Coated Surfaces, Volume Change, Segregation, Quench Process, Acceleration Effects, Acceleration Measurement, Liquid/Liquid Interface, Solid/Liquid Interface, Hardware Malfunction, Thermal Environment More Extreme Than Predicted, Processing Difficulties

**Number of Samples:** six
**Sample Materials:** Pb/Pb-0.05 at.% Au (Pb-Au alloy pressure bonded to pure Pb creating diffusion couples)
(Pb*Au*)
**Container Materials:** Graphite-lined stainless steel (four ampoules) and mild steel (two ampoules)
(C*)

**Experiment/Material Applications:**
Reportedly, the Pb/Pb-0.05 at.% Au system was selected for this experiment for three major reasons:

(1) "The melting point of Pb (600 K) was sufficiently low to provide a convenient temperature range for liquid diffusion and to ensure that 0.05 at.% Au would be initially soluble in Pb upon solidification. Any subsequent precipitation of $Pb_2Au$ would be in situ and thus would still provide a marker for the original position of the Au." (1, p. 368)

(2) The nuclear properties of Pb and Au make the alloy ideal for neutron activation studies.

(3) It was believed that large variations in surface tension with composition are characteristic of the Pb-Au system.

**References/Applicable Publications:**

(1) Reed, R. E., Uelhoff, W., and Adair, H. L.: Surface-Tension-Induced Convection: Experiment M041. In Apollo-Soyuz Test Project, NASA SP-142, Vol. I, 1977, pp. 367-401. (post-flight)

(2) Boese, A., McHugh, J., and Sidensticker, R.: Multipurpose Electric Furnace. In Apollo-Soyuz Test Project, NASA SP-142, Vol. I, 1977, pp. 353-365. (post-flight)

(3) Reed, R. E., Uelhoff, W., and Adair, H. L.: Surface Tension Induced Convection in Encapsulated Liquid Metals in a Microgravity Environment Experiment MA-041. In Apollo-Soyuz Test Project- Composite of MSFC Final Science Report, NASA TM X-73360, January 1977, Section III, pp. 1-87. (post-flight)

(4) Naumann, R. J. and Mason, E. D.: Surface-Tension-Induced Convection. In Summaries of Early Materials Processing in Space Experiments, NASA TM-78240, August 1979, pp. 56-57. (post-flight)


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**Experiment Origin:** USA
**Mission:** SPAR 1
**Launch Date/Expt. Date:** December 1975
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Directional solidification furnace
**Builder of Processing Facility:** Grumman Aerospace Corporation, Bethpage, New York

**Experiment:**
Thermal Migration of Bubbles and Their Interaction with Solidification Interfaces (74-36)

This SPAR 1 experiment was the first in a series of investigations designed by Papazian et al. to study the interaction of bubbles with a solidification interface. (The experiment also represented one of two investigations performed by Papazian during the SPAR 1 sounding rocket mission (see Papazian, SPAR 1, Experiment 74-37 (Chapter 9)).)

The specific objectives of experiment 74-36 were to (1) directly observe the interaction of a solidification interface with bubbles under low-gravity conditions, (2) obtain information concerning the bubble migration rate under an imposed thermal gradient, (3) evaluate the effects of launch-induced fluid motion on a partially solid/partially liquid sample, and (4) better understand the environmental conditions (thermal and barometric) imposed on the experimental apparatus during a sounding rocket flight.

The experimental apparatus consisted of four parts: (1) a frame to support the working components, (2) an electronics package, (3) a motorized 35 mm Nikon F2 camera (250 exposures), and (4) the specimen holder/furnace assembly. The specimen holder consisted of an aluminum block which held four Pyrex™ sample tubes (10 mm by 100 mm), the lighting system, and the heaters. The temperatures of the top and bottom portions were preset and controlled independently. Fourteen thermistors were bonded to the back of the specimen holder which recorded the specimen temperatures.

Prior to the mission, four samples were configured in the directional solidification furnace. Three of these samples (designated as "B," "C," and "D") were related to this experiment. (The other sample was related to SPAR Experiment 74-15 and will not be discussed further in this summary (see instead Uhlmann, SPAR 1 (Chapter 5)).) Samples B, C, and D each consisted of carbon tetrabromide, $CBr_4$, saturated with a gas: Sample B was saturated with argon, Sample C with hydrogen, and Sample D with nitrogen. (The three gasses have differing molecular weights and solubilities in $CBr_4$.)

Approximately 30 to 45 minutes prior to launch, the furnace heaters were activated, melting the sample material and establishing a thermal gradient along the length of the specimen holder. At liftoff, the power line to the heaters was intentionally severed and the specimen holder began to cool. It was determined that the cooling rate of the flight specimens was slower than that observed during similar, ground-based reference experiments performed in the flight apparatus (see Reference (1) for complete thermal history of samples). The solidification behavior of the samples was recorded by a 35 mm camera. Eighty seconds after launch, photographic recording was initiated at a rate of about 1 frame/sec. A total of 220 frames were exposed during the flight experiment.

Post-flight examination of the photographs revealed that gas evolution occurred at the solidification interface. The evolved gas was trapped in the growing crystal roughly in the form of spheres. Comparison to ground-processed samples indicated that the major effect of the low-gravity conditions was an increase in the number and total volume of the voids. The bubbles formed in the liquid ranged from 0.1 mm to 4 mm in diameter. The large bubbles were due to incomplete filling of the sample tubes prior to the mission. These bubbles were originally located at the top of the samples and were pushed down by rocket-spin induced fluid motion. The smaller bubbles, which were not present prior to launch, were either (1) nucleated by a particular vibrational frequency of the rocket motor, (2) nucleated by the reduction in gravity (hydrostatic head), or (3) released by the solid material during interface erosion by the spinning liquid.

The continuous gas evolution resulted in a growth and coalescence of the bubbles situated at the interface. No pushing of these bubbles could be detected macroscopically (minimum detectable motion was about 0,2 mm). Instead, the bubbles were entrapped by the solidification front. It was expected that bubble motion on the order of a few millimeters per second would have been seen. However, no steady bubble motion could be detected. It was suspected that the lack of steady motion may have been due to surface contamination of the bubbles. The source of this contamination was probably decomposition of $CBr_4$ or original impurities in $CBr_4$. (These impurities were not acutally observed in the solidified samples.)

Significant, unsteady bubble motion was observed in sample C. "...the abrupt coalescence of two bubbles at the solid-liquid interface caused pushing of neighboring small bubbles. These observations [including those from a subsequent SPAR III experiment (see Papazian, SPAR 3, Experiment 74-36 (this chapter))] document an additional source of fluid flow in weightless melts, namely, fluid motion due to bubble coalescence or due to the motion of large bubbles." (2, p. 47)

**Key Words:** Fluid Physics, Melt and Solidification, Directional Solidification, Interface Physics, Ternary Systems, Composites with Gases, Saturation, Gas Formation, Bubbles, Bubble Growth, Bubble Motion, Bubble Migration, Thermomigration, Bubble Coalescence, Nucleation, Bubble Nucleation, Thermal Gradient, Solid/Liquid Interface, Liquid/Gas Interface, Solidification Front Physics, Inclusion and/or Rejection of Particles, Voids, Acceleration Effects, Launch-Induced Fluid Motion, Surfactants, Contamination Source

**Number of Samples:** three
**Sample Materials:** Three carbon tetrabromide ($CBr_4$) samples saturated with argon, hydrogen $H_2$, or nitrogen $N_2$.
(C*Br*Ar*, C*Br*H*, C*Br*N*)
**Container Materials:** Pyrex$^{TM}$ tubes

**Experiment/Material Applications:**
Carbon tetrabromide was chosen as the experimental material because (1) previously published results on the material were available for comparison, (2) its melting point (90 $^{o}C$) was suitable for the experimental goals, and (3) it was a readily available material. The three selected gasses have different solubilities in $CBr_4$.

This study is applicable to (1) material solidification processes where gas bubbles may be formed due to decreased solubility in the solid, (2) schemes for controlling bubble dispersions (e.g., foam materials), and (3) processes causing bubble migration.

**References/Applicable Publications:**

(1) Papazian, J. M. and Wilcox, W. R.: Thermal Migration of Bubbles and Their Interaction with Solidification Interfaces. In Space Processing Applications Rocket Project, SPAR I Final Report, NASA TM X-3458, pp. VII-1 - VII-30, December 1976. (post-flight report)

(2) Papazian, J. M. and Wilcox, W. R.: Thermal Migration of Bubbles and Their Interaction with Solidification Interfaces. In Proceedings from Applications of Space Flight in Materials Science and Technology, NBS SP-520, edited by S. Silverman and E. Passaglia, September 1978, pp. 44-49.

(3) Papazian, J. M. and Wilcox, W. R.: Interaction of Bubbles with Solidification Interfaces. AIAA Journal, Vol. 16, No. 5, May 1978, pp. 447-451.

(4) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications, Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(5) Cole, R., Papazian, J. M., and Wilcox, W. R.: Bubble Departure Radii at Solidification Interfaces. International Journal of Heat and Mass Transfer, Vol. 23, February 1980, pp. 219-224. (SPAR 1 and 3)

(6) Papazian, J. M. and Wilcox, W. R.: Thermal Migration and Bubbles and Their Interaction with Solidification Interfaces. In Applications of Space Flight in Materials Science and Technology, Proceedings of a Conference held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977, issued September 1978, pp. 25-39. (post-flight)

(7) The Interaction of Bubbles with Solidification Interfaces. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, pp. 17-18. (post-flight)

(8) Input received from Principal Investigator J. M. Papazian, December 1987, September 1988, and August 1993.

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**Experiment Origin:** USA
**Mission:** SPAR 3
**Launch Date/Expt. Date:** December 1976
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Directional solidification furnace
**Builder of Processing Facility:** Grumman Aerospace Corporation, Bethpage, New York

**Experiment:**
Thermal Migration of Bubbles and Their Interaction with Solidification Interfaces (74-36)

This SPAR 3 experiment was the second in a series of investigations designed by Papazian et al. to study the interaction of bubbles with a solidification interface (see Papazian, SPAR 1, Experiment 74-36). The specific objectives of this experiment were to (1) repeat the SPAR 1 experiment with a higher thermal gradient and (2) compare the behavior of a commercially pure (CP) $CBr_4$ to a high purity, zone refined (ZR) $CBr_4$.

The experimental apparatus employed during SPAR 1 (see Papazian, SPAR 1) was slightly modified and reused during this SPAR 3 mission. The apparatus still consisted of four major parts: (1) a frame to support the working components, (2) an electronics package, (3) a motorized 35 mm Nikon F2 camera (250 exposures), and (4) the specimen holder/heater block which held four 10 mm by 100 mm sample tubes. However, to achieve a higher thermal gradient than that of the SPAR 1 experiment (and yet maintain the same solidification rate), the SPAR 1 aluminum sample holder/heater block was replaced with a shorter block made of stainless steel. The shorter length and lower thermal conductivity of the stainless steel allowed a thermal gradient of 20 $^{o}C/cm$ (compared to 5 $^{o}C/cm$ for the SPAR 1 experiment).

Four samples were processed during the flight experiment: samples A and B were commercial pure $CBr_4$ and samples C and D were high purity, zone refined $CBr_4$. All four sample materials had been saturated with argon for gas bubble generation. (Argon was selected based on the positive results observed during the SPAR 1 flight.)

The intended experimental sequence included (1) heater on 45 minutes prior to launch (sample melting), (2) heater off 4 minutes prior to launch, (3) launch, $t_o = 0$, (4) camera start at 100 seconds after launch, and (5) camera stop at 330 seconds after launch. "The actual sequence followed this plan with the exception of a 16 min hold which occurred at $t_o$-13 [seconds]. During the hold the heaters were reactivated, the count was reset to $t_o$-15 min, and the normal sequence of operation followed." (1, p. IV-4) (The heaters were turned off prior to launch because ground-based experiments revealed that a stable cooling rate was established only after the heater had been shut off for several minutes.)

Post-flight analysis included (1) examination of the photographs and (2) optical observations and radiographic studies of the solidified samples. It was reported that, generally, the main influence of the reduced gravity environment was an increase (due to a lack of buoyancy) in the number and volume of sample voids (when compared to similarly processed ground control samples). "The optical observations also showed that a void nucleation burst had occurred... at approximately $t_o$+70 s. This time coincides with the establishment of low-gravity conditions in the rocket and, hence, may be indicative of a gravitational effect on nucleation, but there is also the possibility that nucleation was simply caused by impurity build-up and the timing was a coincidence." (11, p. 44)

Bubbles were observed in the liquid sections of all flight samples. The bubbles were uniform in size (0.1 to 0.2 mm in diameter) with the exception of a few larger bubbles (about 4 mm). These larger bubbles were due to incomplete filling of the sample tubes prior to the mission and were pushed down into the sample by fluid motion induced by rocket spin. "The smaller bubbles, none of which were present before launch, were nucleated by a particular vibrational frequency of the rocket motor or by the reduction in gravity (i.e., hydrostatic head) or were released by the solid $CBr_4$ during interface erosion by the spinning liquid. It is unlikely that interface erosion would give rise to the uniform bubble size and spatial distribution observed in [the] SPAR III [experiment]. " (11, p. 46)

In samples A, B, and C a steady state equilibrium between gas evolution, interface motion, and void growth was observed. This resulted in cylindrical voids with constant diameters. In sample D, growth and coalescence of the bubbles at the interface were indicated. These bubbles were trapped by the growing interface and no evidence of macroscopic pushing was observed.

Steady state migration of the bubbles was not observed in any of the samples, although initial estimates predicted bubble velocities on the order of mm/sec. Marangoni numbers (calculated based on measured values of physical constants for $CBr_4$), for bubbles of 0.1 mm, 1 mm, and 4 mm diameters were given as 3, 300, and 5000, respectively. Earlier work (J.R.A. Pearson, J. Fluid Mech., Vol. 4, pp. 489-500, 1958) had indicated that, for planar geometries, significant thermocapillary flow should occur for Marangoni numbers above 80. However, more recent experiments (S. R. Coriell, et al., NBSIR 77-1208 Annual Report, 72, February 1977) have indicated that bubble flow occurs with Marangoni numbers of the order of $10^{-4}$. "The most likely cause for the observed bubble immobility in... [this]... experiment is thought to be contamination of the bubble surface by impurities." (11, p. 47) (The expected sources of this contamination were the same as those detailed for the Papazian's earlier SPAR 1 experiment.)

Significant bubble motion was observed in sample B: "...the lower edge of the large bubble just visible at the top of Specimen B moved downward by 1.3 mm. This motion occurred in a relatively uniform manner over a 60 s time interval. The flight film shows that the motion of the large bubble caused the small bubbles in its vicinity to be pushed along apparent streamlines of fluid flow.... These observations [also, see Papazian, SPAR 1, Experiment 74-36] document an additional source of fluid flow in weightless melts, namely, fluid motion due to bubble coalescence or due to the motion of large bubbles." (11, p. 47)

The results from this experiment, and the similar experiment performed during SPAR 1, were as follows:

(1) The larger number and greater volume of voids in the flight samples can be directly attributed to the lack of buoyancy forces.

(2) The morphology of the voids was dependent on the thermal gradient: for a gradient of 5 °C/cm (SPAR 1) cylindrical voids were present while for 20 °C/cm (SPAR 3) spherical voids were present. Void morphology was dependent upon gravity for the zone refined material only.

(3) Low-gravity conditions favor easier void nucleation and may favor easier bubble nucleation for commercial purity $CBr_4$.

(4) A thermal gradient of 20 °C/cm does not result in (a) detachment of bubbles form the solid/liquid interface or (b) migration toward the hot end in liquid $CBr_4$.

(5) $CBr_4$ solidification interfaces are unable to induce long distance pushing (greater than 0.2 mm) of bubbles.

(6) Significant fluid flow in liquids under low-gravity conditions can be caused by bubble motion or coalescence.

**Key Words:** Fluid Physics, Melt and Solidification, Directional Solidification, Interface Physics, Ternary Systems, Saturation, Composites with Gases, Gas Formation, Bubble Formation, Bubble Migration, Marangoni Movement (Migration) of Bubbles, Bubble Coalescence, Nucleation, Bubble Nucleation, Bubble Dispersion, Particle Size Distribution, Bubble Motion, Bubble Growth, Thermal Gradient, Solidification Front Physics, Solid/Liquid Interface, Liquid/Gas Interface, Inclusion and/or Rejection of Particles, Thermocapillary Convection, Buoyancy Effects Diminished, Surfactants, Voids, Contamination Source, Impurities, Sample Purity, Acceleration Effects, Launch-Induced Fluid Motion

**Number of Samples:** four
**Sample Materials:** Carbon tetrabromide ($CBr_4$) samples; two were of commercial purity and two were high purity, zone refined materials. All samples were saturated with argon.
(C*Br*Ar*)
**Container Materials:** Pyrex™

**Experiment/Material Applications:**
See Papazian, SPAR 1, Experiment 74-36.

**References/Applicable Publications:**

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(2) Papazian, J. M. and Wilcox, W. R.: Bubble Behavior During Solidification in Low Gravity. Grumman Corporate Research Center Report RM-680, February 1979, 21 pp. (post-flight report)

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(9) Cole, R., Papazian, J. M., and Wilcox, W. R.: Bubble Departure Radii at Solidification Interfaces. International Journal of Heat and Mass Transfer, Vol. 23, 1980, pp. 219-224.

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**Experiment Origin:** USA
**Mission:** SPAR 1
**Launch Date/Expt. Date:** December 1975
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Temperature Control Unit (TCU) furnace (heaters, sample cartridges, water quench system, and sample monitoring thermistors)
**Builder of Processing Facility:** NASA Marshall Space Flight Center, Huntsville, Alabama

**Experiment:**
Characterization of Rocket Vibration Environment by Measurement of the Mixing of Two Liquids (74-18)

This SPAR 1 experiment was the first in a series of investigations designed by Schafer et al. to illustrate the nature of the SPAR rocket acceleration environment and its effect on diffusion-controlled fluid systems.

Prior to the SPAR mission, three 0.6 cm cylindrical samples were prepared. Each of these prepared samples consisted of a section of pure indium joined to a similarly-sized section of 80 wt% indium-20 wt% lead. The density differences between the two sections were selected to allow convective mixing to occur if sufficient residual gravity was present when the materials were (later) molten.

Each sample was placed within a separate heater within the experiment Thermal Control Unit (TCU). The three sample/heater assemblies were configured such that each was aligned differently to the rocket longitudinal axis and onboard accelerometer axes when the TCU was mounted in the SPAR rocket.

During the SPAR 1 experiment, the samples were melted and solidified during the low-gravity coasting phase of the mission. Reportedly, the experiment sequence was as follows: (1) 45 seconds after launch, sample heating was initiated, (2) 225 seconds after launch, sample heating was terminated, and (3) 345 seconds after launch, sample quench was initiated.

Inspection of the flight data and/or the returned TCU payload indicated that (1) the experiment was performed as scheduled, (2) the samples were processed with the expected thermal profile, and

(3) the quench system performed as expected.

Post-flight, the three In-(In-Pb) interfaces were examined to determine the degree of mixing that had occurred in each of the samples. Reportedly, the sample "...(oriented parallel to the payload longitudinal axis) experienced interface motion near that... expected from diffusion[,]... the sample (aligned parallel to the accelerometer x-axis) experienced a small amount of interface motion... which might be slightly more than that expected by diffusion[,]... and the sample (aligned parallel to the accelerometer y-axis) experienced flow down one side of the container." (1, p. IV-27)

Both experimental and computational correlation of fluid motion with acceleration environment was performed. It was observed that (1) "...the magnitudes of the flow experienced by the three samples... are consistent with the acceleration arising from the rotation of the payload about its longitudinal axis...." (1, p. IV-36), and (2) the level of this acceleration is of the order of $10^{-5}$ g or less. Thus it was concluded that while the residual accelerations aboard SPAR are very low and the vehicle provides a good platform for experiments requiring up to 5 minutes of low-g time, convective fluid motion can occur at these low-g levels. It was recommended that computations to estimate levels of effective gravity for which convective flows become significant be performed for a given experimental configuration.

**Key Words:** Fluid Physics, Melt and Solidification, Binary Systems, Alloys, Quench Process, Thermosolutal Convection, Density Difference, Liquid Mixing, Liquid/Liquid Interface, Diffusion, Diffusive Mass Transfer, Viscosity, Rotation of Payload, Acceleration Effects, Acceleration Measurement, Launch-Induced Fluid Motion, Rocket Vibration

**Number of Samples:** three
**Sample Materials:** First section: pure indium; second section: 80 wt% In-20 wt% Pb
(In*Pb*)
**Container Materials:** aluminum
(Al*)

**Experiment/Material Applications:**
It is expected that the low gravity environment will reduce convective flow in systems with density gradients. This experiment illustrates the need to characterize the acceleration environment and to consider the effects of possible fluid motions even at low

acceleration levels.

The In/In-Pb system was chosen for several reasons. First, not only could it be used illustrate the mixing behavior of two fluids of differing density, but it was also expected to yield results which could be interpreted as relevant to other experiments involving differing density fluids. Second, its "...kinematic viscosity is in the range of kinematic viscosities of materials of interest to other investigators (e.g. liquid metals). This follows from the fact that for a fixed geometry, the kinematic viscosity of a fluid is a fundamental parameter for governing flow due to accelerations which act through density differences." (1, p. IV-4) "Thirdly, the materials chosen for sample preparation were selected so as to minimize spurious flow effects." (1, p. IV-4)

**References/Applicable Publications:**


(1) Schafer, C. F.: Liquid Mixing Experiment. In Space Processing Applications Rocket Project, SPAR I Final Report, NASA TM X-3458, pp. IV-1 - IV-37, December 1976. (post-flight)

(2) Schafer, C. F. and Fichtl, G. H.: SPAR 1 Liquid Mixing Experiment. 15th AIAA Aerospace Sciences Meeting, Los Angeles, California, January 24-26, 1977, 8 pp. (post-flight)

(3) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements; SPAR 1-4)

(4) Liquid Mixing Experiments. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 11-12. (post-flight)

(5) Input received from Principal Investigator C. F. Schafer, August 1993.


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**Experiment Origin:** USA
**Mission:** SPAR 3
**Launch Date/Expt Date:** December 1976
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Temperature Control Unit (TCU) furnace (heaters, sample cartridges, water quench system, and sample monitoring thermistors)
**Builder of Processing Facility:** NASA Marshall Space Flight Center, Huntsville, Alabama

**Experiment:**
Liquid Mixing (74-18)

This SPAR 3 sounding rocket mission was the second in a series of investigations designed by Schafer et al. to illustrate the nature of the SPAR rocket acceleration environment and its effect on diffusion-controlled fluid systems (see Schafer, SPAR 1). The specific objective the SPAR 3 study was to extend the earlier SPAR research by similarly processing In/In-Pb samples of different aspect ratios.

Prior to the SPAR mission, seven cylindrical samples were prepared. Each of these prepared samples consisted of a section of pure indium joined to a similarly sized section of 80 wt% In-20 wt% Pb. The samples were 2.2 cm long and had a diameter of either 0.6 cm (sample #1, #2, #3, and #4), 0.45 cm (sample #5), or 0.3 cm (sample #6 and #7). Each of the samples was contained in an aluminum cylinder and placed within a separate heater within one of two experiment Thermal Control Units (TCU).

Reference (1), Figure 8 details the placement of the 7 samples within the TCU furnace. Sample #2, #3, and #7 were configured in the TCUs such that forces acting on the samples acted parallel to the original interface between the In and In-Pb. "The fluid mechanics problem presented here is that of a cylinder lying on its side with a constant body force applied vertically." (1, p. III-9) Sample #1, #4, #5 and #6 were configured in the TCUs such that forces acting on the samples were directed radially outward. The fluid dynamics problem presented here is that of a cylinder containing "...a heavier liquid overlaying a lighter liquid in a vertically downward directed force field." (1, p. III-9)

Reportedly, neither fluid problem has an analytical solution, although some modeling of the systems can provide insight. Based on these models it was expected that given a radially directed residual acceleration with a magnitude of between $10^{-6}$ and $10^{-5}$ g, (1) sample #2, #3, #6, and #7 would not exhibit measurable flows and (2) sample #1, #4, and #5 would exhibit measurable flows. <Note: Reference (1) also discusses the modeling of the two fluid problems.>

During the SPAR 3 experiment, the samples were melted and solidified during the low-gravity coasting phase of the mission. The experiment sequence was as follows: (1) 45 seconds after launch, sample heating was initiated, (2) 225 seconds after launch sample heating was terminated (the sample remained molten for approximately 180 seconds), and (3) 345 seconds after launch, sample quench was initiated.

Post-flight examination of the samples included X-ray radiographic inspections. These inspections were used to (1) determine whether fluid flow had occurred in the molten samples and (2) compare the flight results to the theoretical analysis (see Reference (1)). Reportedly, "The experiment performed well in the engineering sense in that all samples melted and were quenched at the proper times. Samples 4 and 5 however, did not have good joints between the In and In-Pb sections so that no flow could have occurred in these samples. Sample 1 experienced flow as expected and by an amount consistent with a radially directed acceleration with a magnitude of $10^{-6}$ g to $10^{-5}$ range (approximately 5 X $10^{-6}$ g). Samples 2, 3, 6 and 7 experience[d] no flow as expected for radial acceleration of that magnitude. In summary, the SPAR III results were predicted by the considerations used to fully explain the SPAR 1 results." (1, p. III-12)

**Key Words:** Fluid Physics, Melt and Solidification, Binary Systems, Alloys, Quench Process, Thermosolutal Convection, Density Difference, Liquid Mixing, Liquid/Liquid Interface, Diffusion, Diffusive Mass Transfer, Aspect Ratio, Rotation of Payload, Acceleration Effects, Acceleration Measurement, Launch-Induced Fluid Motion, Processing Difficulties

**Number of Samples:** seven
**Sample Materials:** First section: pure In; second section: 80 wt% In-20 wt% Pb
(In*, In*Pb*)

**Container Materials:** aluminum
(Al*)

**Experiment/Material Applications:**
See Schafer, TEXUS 1.

**References/Applicable Publications:**

(1) Schafer, C. F. and Fichtl, G. H.: Liquid Mixing. In Space Processing Applications Rocket Project SPAR III-Final Report, NASA TM-78137, pp. III-1 - III-16, January 1978. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications, Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(3) Liquid Mixing Experiments. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 11-12.

(4) Naumann, R.J. and Herring, H. W.: Materials Processing in Space: Early Experiments. NASA SP-443, 1980, pp. 99-100. (post-flight)

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**Experiment Origin:** USA
**Mission:** SPAR 3
**Launch Date/Expt Date:** December 1976
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Single Axis Acoustic Levitator (SAAL)
**Builder of Processing Facility:** Intersonics, Northbruck,Illinois

**Experiment:**
Contact and Coalescence of Viscous Bodies (74-53)

This SPAR 3 sounding rocket experiment was designed to study the viscous coalescence of Newtonian fluid drops under low-gravity conditions. The specific objective of the investigation was to obtain an accurate characterization of the growth of inter-particle necks which form during a viscous sintering process.

During the SPAR 3 mission, it was intended that drops of a viscous fluid (Dow Corning silicone oil, 5000 centistokes) be injected into an acoustic levitation chamber. The drops were to be formed on the tip of a needle and then blown into the chamber by the flow from an air nozzle. The drop size and injection velocity was to be carefully controlled such that contact and coalescence between the drops would result. The entire process was to be filmed and the motion of the drop boundaries determined.

Reportedly, the experiment "...did not succeed because of the failure of drops of the desired size to form at the tip of the injector needle under microgravity conditions. This has since been associated with the fluid being pumped too fast through a small needle. For this reason, a stream of fluid was formed rather than the desired drop at the end of the needle. In addition, the force on the drop from the air nozzle was too large, causing premature detachment of small drops." (1, p. VII-13) (See Reference (1) for details.)

**Key Words:** Fluid Physics, Drops, Acoustic Levitation, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Droplet Injection, Drop Formation, Droplet Size, Drop Velocity, Droplet Collision, Drop Coalescence, Viscosity, Viscoelastic Bodies, Liquid/Gas Interface, Processing Difficulties

**Number of Samples:** One experiment run was planned.
**Sample Materials:** Dow Corning 200 silicone oil with additives
**Container Materials:** not applicable

**Experiment/Material Applications:**
Silicone oil was selected as a model material to evaluate (1) the contact and coalescence process which occurs in some viscoelastic sintering processes as well as (2) the thermal processing of phase separating glasses.

**References/Applicable Publications:**

(1) Uhlman[sic], D. R.: Contact and Coalescence of Viscous Bodies. In Space Processing Applications Rocket Project SPAR III-Final Report, NASA TM-78137, January 1978, pp. VII-1 - V-17. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1987. (acceleration measurements; SPAR 1-4)


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**Experiment Origin:** USA
**Mission:** SPAR 4
**Launch Date/Expt. Date:** June 1977
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Triaxial acoustical levitation resonance chamber
**Builder of Processing Facility:** Unknown

**Experiment:**
Containerless Processing Technology (76-20/1)

This SPAR sounding rocket experiment was the first in a series of experiments designed by Wang et al. to study the stability, manipulation, oscillation, and rotation of a liquid drop held in an acoustic field. The specific objectives of the experiment (as reported in Reference (1)) were to:

(1) Determine the positioning capability of the employed acoustic chamber.
(2) Determine the perturbation of drop shape oscillation due to G-jitter-induced center of mass motion.
(3) Determine the rotation capability of the employed acoustic chamber.
(4) Determine the perturbation of drop rotation induced by G-jitter.
(5) Determine the capability of the employed acoustic position servo system.
(6) Study the natural and resonant frequencies and damping mechanism of drop oscillation.
(7) Study the drop shape change due to rotation.

The experiment was performed in a nearly cubical triaxial acoustical levitation resonance chamber (11.43 X 11.43 X 12.70 cm). Within the chamber, three mutually perpendicular acoustic drivers were designed to excite "...the lowest-order standing wave along the direction that the driver faces. In a resonant mode, the ambient pressure is maximum at the nodes of the velocity wave and minimum at the antinodes. Consequently, there is a tendency for introduced liquids and particles to be driven toward the antinodes, where they collect and remain until excitation ceases."

(1, p. V-5) The experimental setup included a camera and the required hardware to film the drop dynamics from three directions.

A detailed description of the anticipated inflight operation of the acoustic chamber was presented in Reference (1), p. V-6. The procedure was specifically designed to insure that a 1.25 cm radius water droplet (experiencing a residual acceleration level of $10^{-4}$g and an acoustic chamber quality factor (omega/2*(delta omega)) of approximately 25) would rotate and oscillate.

During the mission, a fluid sample was injected into the chamber as planned. <Note: the specific fluid was not identified, although it is suspected that it was water.> However, 130 seconds after liftoff, an unexpected sound power failure occurred, and the suspended drop drifted into the chamber wall. Reportedly, it was suspected that the failure was caused by a circuit design error.

Although drop oscillation and rotation were not realized, "...a thirty second segment of free oscillation, superimposed on a slow drift of the drop in the microgravity environment..." (1, p. V-10) was achieved.

Post-flight, the camera film was developed for analysis. Unexpectedly, the images were foggy. It was suspected that silicone oil (employed during droplet formation experiments on earlier Learjet flight tests which employed the SPAR equipment) vaporized and condensed causing the foggy images. Consequently, analysis (using the film) was hampered. Only one of the three camera views could be processed with some certainty; thus, three-dimensional information of the drops was difficult to resolve. Among other characteristics, drop boundaries (as a function of time) and center of mass motion were analyzed. (See Reference (1), pp. V-10 to V-34 for details of the data analysis.)

It was reported that "The analysis identified the three lowest modes of a freely oscillating drop. However, information concerning line shapes, line widths, and damping factors could not be obtained from the present data...." (1, p. V-34)

The experiment was reflown on SPAR 6 after the problems with the electronics and vaporizing silicone oil had been corrected (see Wang, SPAR 6).

<Note: This experiment appears to be an extension of an earlier experiment by Vaughan and Wang on Skylab SL-4 entitled "Acoustic Positioning" (see Vaughan, SL-4, "Acoustic Positioning" (this chapter)).>

**Key Words:** Fluid Physics, Containerless Processing Applications, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Standing Waves, Hydrodynamics, Drops, Liquid Injection, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Drop Formation, Drop Dynamics, Drop Stability, Liquid Stability, Drop Oscillation, Fluid Oscillation, Drop Vibration, Liquid Vibration, Drop Rotation, Sample Rotation, Rotating Fluids, Fluid Motion Damping, Liquid Dynamic Response, Droplet Collision, Sample Deformation, Free Surface, Surface Tension, Liquid/Gas Interface, Contamination Source, Vaporization, Condensation, Acceleration Effects, Hardware Malfunction, Photographic Difficulties

**Number of Samples:** one
**Sample Materials:** Unclear, appears to have been water.
**Container Materials:** not applicable

**Experiment/Material Applications:**
See Wang, SPAR 6 (this chapter).

**References/Applicable Publications:**

(1) Wang, T. G., Elleman, D. D., Jacobi, N., and Tagg, R.: Containerless Processing Technology Experiment Report, SPAR IV Experiment 76-20. In Space Processing Applications Rocket Project, SPAR IV Engineering Report (Final), NASA TM-78235, pp. Vi-V69, January 1980. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement and Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI Inc., Technical Report 1308, March 1978. (acceleration measurements; SPAR 1-4; post-flight)

(3) Containerless Processing Technology. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 31-32. (post-flight)

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**Experiment Origin:** USA
**Mission:** SPAR 6
**Launch Date/Expt. Date:** October 1979
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Triaxial acoustical levitation resonance chamber (This chamber was the same as that employed during Wang's SPAR 4 experiment but now with minor modifications.)
**Builder of Processing Facility:** Unknown

**Experiment:**
Containerless Processing Technology (76-20/2)

This SPAR 6 sounding rocket experiment was the second in a series of investigations designed by Wang et al. to study the stability, manipulation, oscillation, and rotation of a liquid drop held in an acoustic field (see Wang, SPAR 4). The specific objectives of the experiment (as reported in Reference (1)) were to:

(1) Study the center of mass motion of a drop in an acoustic chamber.
(2) Determine the rotation capability of the chamber.
(3) Study the natural resonant frequencies and damping mechanism of drop oscillation.
(4) Study the drop shape change due to rotation.

The experiment was performed in the triaxial acoustical levitation resonance chamber previously used by Wang during SPAR 4. The chamber had been somewhat modified since SPAR 4, although the acoustic drivers still operated as they did during SPAR 4 (see Wang, SPAR 4 for details of the chamber). <Note: The SPAR 6 chamber modifications were not clearly defined in the available references.> A 16 mm camera, whose line of focus was directed along the z axis of the chamber, documented the motion of the drop in the x and y axes as well, via the use of two mirrors.

During the SPAR 6 flight, a total of 8.6 cc of water was slowly injected into the center of the chamber. During the first 18 seconds of the low-gravity phase of the experiment, the injector tips were withdrawn, exciting free oscillations of the drop surface. These free oscillations eventually damped out, and the drop moved back and forth in the acoustic potential well.

Forced oscillation of the drop was achieved. Planned modulation of the acoustic pressure along the z axis resulted in large, regular drop oscillation. Subsequently, the drop unexpectedly rotated. This rotation was attributed to the modulation. When complimentary modulation of the x and y axes was added, the drop relaxed. During the next 30 seconds, the drop was subjected to a torque generated by "...a phase difference of $90^{\circ}$ between the x and y pressure amplitudes." (1, p. V-5-3) This torque rotated the drop about the z axis. (It was noted that the time allotted to this part of the experiment was probably inadequate for solid body rotation to have been reached.)

Drop boundary data were obtained by digitizing selected frames of the filmed drop motion. A detailed analysis was presented. (Refer to Reference (1), Section 4, beginning on p. V-4-1 for a detailed theoretical background of the SPAR data analysis.)

Speculation concerning the unexpected rotation of the modulated drop was given. "One likely possibility is that an off-center drop in a complicated pressure field... accompanied by larger amplitude motion, would experience an effective torque." (1, p. V-6-1) It was also concluded that when the drop was later rotated by applying the torque, "...the first bifurcation point was almost reached in only thirty seconds of rotation. Since equilibrium... [had] not yet been reached, this... [indicated] that spending more time on the rotation sequence or increasing the torque by using a higher sound pressure might take the drop beyond the first bifurcation point." (1, p. V-6-1)

Additional information describing the experiment and post-flight analysis can be found in several of the publications below.

**Key Words:** Fluid Physics, Containerless Processing Applications, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Standing Waves, Hydrodynamics, Liquid Injection, Drop Formation, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Drops, Droplet Injection, Drop Dynamics, Drop Stability, Liquid Stability, Drop Oscillation, Fluid Oscillation, Drop Vibration, Liquid Vibration, Drop Rotation, Sample Rotation, Rotating Fluids, Fluid Motion Damping, Liquid Dynamic Response, Sample Deformation, Free Surface, Surface Tension, Liquid/Gas Interface, Acceleration Effects

**Number of Samples:** one
**Sample Materials:** distilled water mixed with dye to provide an optimum filming medium; surface tension 71.0 +/- 2.0 dynes/cm
**Container Materials:** not applicable

**Experiment/Material Applications:**
This experiment was designed to aid in the study of containerless processing in a reduced gravity environment. Because containerless processing involves materials which are in the liquid-melt state, these experiments will contribute to such research in the following ways: (1) stability analysis of the drop will allow the investigation of (a) the effect of g-jitter on the sample and (b) the focus requirement of the heat lamp on the sample; (2) oscillation analysis of the drops will allow the investigation of (a) the "...induced mixing currents within the melt..." and (b) the maximum acoustic power required..." (1, p. V-1-1); and (3) rotation analysis will allow the investigation of (a) the feasibility of degassing and shaping the melt, and (b) the feasibility of using only one heat lamp to process the rotating sample.

Thus, this research impacts (1) the thermal insulation requirements on the chamber; (2) the total power, weight, and acoustic insulation requirements of the chamber; (3) the economical feasibility of containerless processing; and (4) the thermal design of the chamber. (1, p. V-1-1)

**References/Applicable Publications:**
(1) Wang, T. G., Elleman, D. D., Jacobi, N., and Croonquist, A. P.: Containerless Processing Technology Experiment Report SPAR VI Experiment 76-20. In Space Processing and Applications Rocket Project SPAR VI Final Report, NASA TM-82433, pp. Vi - V-B-6, October 1981. (post-flight)

(2) Wang, T. G., Elleman, D. D., Jacobi, N., Croonquist, A., and Rayermann, P.: Containerless Processing Technology Experiment Interim Report: SPAR VI Experiment 76-20. October 1980, Jet Propulsion Laboratory, Pasadena, California.

(3) Containerless Processing Technology. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 31-32. (preflight)

(4) Jacobi, N., Croonquist, A. P., Elleman, D. D., and Wang, T. G.: Acoustically Induced Oscillation and Rotation of a Large Drop in Space. <Note: Appears to be SPAR 6. Further information on where this document was published could not be determined at this time.>

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**Experiment Origin:** USA
**Mission:** SPAR 7
**Launch Date/Expt. Date:** May 1980
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Triaxial acoustical levitation resonance chamber (This chamber was modified following the SPAR 6 flight (see detail below).)
**Builder of Processing Facility:** Unknown

**Experiment:**
Containerless Processing Technology: Dynamics of Liquid Drops 76-20/3 (Coalescence and Mixing Experiments)

This SPAR 7 sounding rocket experiment was the third in a series of investigations designed by Wang et al. to study the stability, manipulation, and oscillation of liquid drops held in an acoustic field (see Wang, SPAR 4, SPAR 6). The specific objectives of the experiment (as reported in Reference (1)) were to:

(1) Demonstrate the capability of the acoustic chamber for collision and coalescence of quiescent drops.
(2) Study the mixing effects generated by collision and coalescence of oscillating drops.
(3) Study the shift of natural frequencies of drop oscillation as a function of oscillation amplitude.

The investigation was performed in the triaxial acoustic levitation resonance chamber previously employed by Wang on SPAR 4 and 6. The chamber had been modified for SPAR 7, however, to include a "...multiple-drop injection system and a vacuum system to retrieve drops between experiments." (1, p. IV-5) The nearly cubical chamber (10.59 X 11.42 X 12.70 cm) had slightly different dimensions than the SPAR 4 and 6 chamber "to preclude interference effects." (The mutually perpendicular acoustic drivers still operated as described in Wang, SPAR 4.) A 16 mm camera, whose line of focus was directed along the z axis of the chamber, documented the motion of the drop in the x and y axes as well via the use of two mirrors.

The multiple drop injection system consisted of "two forked syringes." "The water was deployed through... [the] syringes so as to form two drops near the center of the chamber with one arm of the forks acting as a post and the other one, which was hollow, acting as the conduit for the liquid. The posts were necessary so that the drop would remain close to the center of the chamber when the two forks were retracted." (1, p. IV-9)

The droplet liquid consisted of distilled water and a small quantity of dye. The dye was added to produce a contrast between the two drops.

During the mission, the "...4.6 $cm^3$ for each drop were injected over 13 seconds. The acoustic drivers were turned on just after retraction providing positioning forces as the injector tips were withdrawn." (1, p. IV-9)

Four similar sequences were planned during which a red water drop and a green water drop were deployed. Drop collision and coalescence as a result of acoustic forces was to be studied during each run.

Post-flight examination of acceleration levels indicated that acceleration spikes occurred during the first run. "The largest spike was approximately 100 $\mu$g and lasted roughly two seconds.... This motion strongly affected the outcome of the experiment because it occurred at the most critical time just before probe retraction when the drops were full size and attached to the forked probes with no acoustic force to stabilize them." (1, p. IV-13)

This acceleration "...caused the drops to move down the posts (i.e. the non-hollow arms) of the forks... [during] the first sequence. When the probes [forks] were retracted the drops followed the probes to... [an opposite] corner [of the chamber]. In the second and third series the red drop was... [successfully] deployed but the green drops slid down the post as the post had been wetted inadvertently during deployment of previous sequence and were carried to the corner. In the final sequence the two drops were successfully deployed although their volumes were less than the preprogrammed ones because of residual water on the forks.

"Because the drops were not centered in the chamber during deployment, they oscillated within the acoustic potential well. The acoustic force was sufficient to hold the drops and prevented them from contacting the chamber wall and in addition, brought them together to coalesce. The motions caused by the acoustic restoring force are found to be consistent with what has been observed and reported in the SPAR VI report." (1, p. IV-15) (See

Wang, SPAR 6.)

Free oscillation analysis of the red and green drops and the coalesced drop was presented. "Despite the problems engendered by the g-perturbations early in the experiment, the system did deploy a droplet pair which coalesced under the influence of the acoustic fields. In this sequence the drops did behave as planned undergoing large amplitude oscillation." (1, p. IV-28)

Additional information describing this experiment can be located in Reference (1).

**Key Words:** Fluid Physics, Containerless Processing Applications, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Standing Waves, Hydrodynamics, Liquid Injection, Drop Formation, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Droplet Injection, Drops, Drop Dynamics, Drop Stability, Liquid Stability, Drop Oscillation, Fluid Oscillation, Drop Coalescence, Liquid Mixing, Droplet Collision, Collisions, Liquid Dynamic Response, Liquid/Gas Interface, Liquid/Liquid Interface, Free Surface, Surface Tension, Wetting, Vacuum, Acceleration Effects, Processing Difficulties

**Number of Samples:** one setup, four experimental runs
**Sample Materials:** distilled water (dyed red and green to provide a contrast between the two fluids); surface tension (measured prior to flight): 71.0 +/- 2.0 dynes/cm
**Container Materials:** not applicable

**Experiment/Material Applications:**
"Study of the stability and manipulatability of liquid drops at room temperature is a useful and cost effective intermediate step in the development of a better understanding of the fluid physics pertinent to the mixing of two heated materials and the capability to produce alloys and amorphous materials in a zero-G environment. In addition the thermodynamics of chemical reactions can be studied by bringing two reactants together." (1, p. IV-1)

**References/Applicable Publications:**

(1) Croonquist, A. P., Wang, T. G., Elleman, D. D., and Rayermann, P. H.: Containerless Processing Technology Experiment Dynamics of Liquid Drops Report-SPAR VII Experiment 76-20/3. In Space Processing Applications Rocket (SPAR) Project, SPAR VII Final Report, NASA TM-82535, pp. IV-i - IV-32, October 1983. (post-flight)

(2) Containerless Processing Technology. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, Edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 31-32. (preflight)

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**Experiment Origin:** USA
**Mission:** SPAR 7
**Launch Date/Expt. Date:** May 1980
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Triaxial acoustic levitation resonance chamber
**Builder of Processing Facility:** Unknown

**Experiment:**
Containerless Processing Technology Experiment: Dynamics of Liquid Bubbles 77-18/1

This SPAR sounding rocket experiment was the first in a series of investigations designed by Wang et al. to examine the stability and oscillations of liquid shells in the presence of an acoustic field. The investigation was an extension of earlier sounding rocket research initiated by Wang et al. which investigated the stability of liquid drops in an acoustic field (see Wang, SPAR 4, SPAR 6, and SPAR 7 "Dynamics of Liquid Drops 76-20/3" (this chapter)).

The specific objectives of this SPAR 7 experiment (as reported in Reference (1)) were to:

(1) Determine the sphericity of a positioned liquid bubble.
(2) Determine the efficiency of bubble centering by oscillation.
(3) Determine the perturbation on bubble centering by G-jitter.
(4) Study the natural resonant frequencies and damping mechanism of bubble oscillation.
(5) Study the adiabatic expansion of the liquid bubble.

The triaxial acoustic levitation resonance chamber used for this SPAR 7 experiment was similar to the chamber employed during Wang's previous SPAR 6 liquid drop investigation (see Wang, SPAR 6). However, the SPAR 7 chamber had the capability of creating liquid shells by injecting air into the deployed drop. The nearly cubical chamber (10.59 X 11.42 X 12.70 cm) had the same dimensions as the SPAR 6 chamber. Operation of the acoustic drivers was similar to that described in Wang, SPAR 4. A 16 mm camera was used to document the shell motion.

During the flight, 8.0 cc of dyed water was deployed into the center of the chamber by two coaxial syringes. <Note: It is not clear if these "two coaxial syringes" (1) consisted of two syringes which met in the center of the chamber and each supplied half of the 8.0 cc of volume or (2) were in some other unexplained configuration.> After water injection was completed, 0.16 cc of air was injected by concentric tubes located within the water syringes. Next, the syringes retracted from the shell and 30% amplitude modulation of the z-axis began. One hundred and twenty seconds later the modulation was terminated.

Post-flight analysis of the experiment data indicated that the injected air volume was not as large as planned (0.16 cc (actual) vs. 2.4 cc (expected)) "...because the probe tips were not adequately covered by water when the air flowed through the injectors. In addition during deployment, the drop was not centered between the probes. This resulted in an oscillation of the drop in the acoustic potential well. The acoustic force caused the small bubble to move in the drop more than was planned... [complicating] data analysis." (1, pp. V-15-16)

Post-flight examination of acceleration levels of the rocket indicated that the x- and y-axes were affected by several acceleration spikes. The largest acceleration (which occurred 108 seconds into the experiment) caused the drop to move slightly. Reportedly, this motion did not adversely affect the experiment because at that time, the drop was not fully deployed.

Detailed analysis of shell sphericity, free oscillation, forced oscillation, center of mass motion and adiabatic expansion was presented in Reference (1). "Using simple techniques of taking information from the cine film record, several areas were successfully studied: the observation of the natural oscillations of orders 2, 3, and 4; the characterization of the acoustic well in terms of the simple harmonic oscillator potential and observation of the corresponding frequencies, and the study of the expansion of the bubble as the pressure dropped.... The methods used failed to give information about the damping coefficient but allowed a crude analysis of the deviation of the drop boundary from circularity....

"Because of its small size it was impossible to study the natural oscillations of the bubble and the presence of a centering force within the drop was overwhelmed by the drop's motion in the acoustical potential well." (1, p. V-46)

Additional information describing this experiment can be found in Reference (1).

**Key Words:** Fluid Physics, Containerless Processing Applications, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Standing Waves, Hydrodynamics, Drop Formation, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Drops, Shells, Drop Dynamics, Fluid Motion Damping, Bubbles, Bubble Dynamics, Bubble Stability, Bubble Centering, Drop Oscillation, Fluid Oscillation, Bubble Formation, Bubble Motion, Gas Injection, Adiabatic Expansion, Liquid Dynamic Response, Free Surface, Surface Tension, Wetting, Liquid/Gas Interface, Interface Physics, Acceleration Effects, Sphericity, Processing Difficulties

**Number of Samples:** one
**Sample Materials:** distilled water (dyed for photographic purposes); surface tension: 71.0 +/- 2.0 dynes/cm
**Container Materials:** not applicable

**Experiment/Material Applications:**
This experiment was designed to study phenomena associated with the containerless processing of small, spherical shells. Such metal or glass shells have applications as inertial confinement fusion (ICF) targets. Drop sphericity, bubble centering, and adiabatic expansion are all important characteristics in the creation of the precise ICF targets.

**References/Applicable Publications:**
(1) Elleman, D. D., Croonquist, A. P., Jacobi, N., and Wang, T. G.: Containerless Processing Technology Experiment Dynamics of Liquid Bubbles-Report SPAR VII Experiment 77-18 Flight I. In Space Processing Applications Rocket (SPAR) Project, SPAR VII Final Report, NASA TM-82535, pp. V-5 - V-47, October 1983. (post-flight results)

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**Experiment Origin:** USA
**Mission:** SPAR 8
**Launch Date/Expt. Date:** November 1980
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Triaxial acoustic resonance levitation chamber which had flown on SPAR 7
**Builder of Processing Facility:** Unknown

**Experiment:**
Dynamics of Liquid Bubbles (77-18/2)

This experiment was the second in a series of investigations designed by Wang et al. to study the stability and oscillation of liquid shells in the presence of an acoustic field (see Wang, SPAR 7, experiment 77-18/1). The SPAR 8 experiment had the same five goals as the previous SPAR 7 experiment as well as an additional sixth goal: to examine the centering forces due to shell rotation.

The triaxial acoustic resonance levitation chamber used in this experiment was the same as that employed during SPAR 7, experiment 77-18/1. "For this flight the dimensions of the chamber and the signals to the drivers were modified to enable the shell to be rotated." (1, p. III-3). The operation of the acoustical drivers was similar as described in Wang, SPAR 4. A camera was used to document shell motion.

Reportedly, "Experiment 77-18/2 was designed to deploy a shell, maintain it in the center of the chamber, rotate it and stimulate several modes of its natural shape oscillations-all acoustically, and then to decompress the acoustic chamber so the shell would expand." (1, p. III-38)

Deployment of 5.77 cc of dyed water through two liquid syringes, and subsequent injection of 4.21 cc of air into the water, was completed similarly as described in Wang, SPAR 7 (experiment 77-18). During the experiment, "...surface oscillations generated by the retraction of the injectors permitted the study of the natural oscillations of an air-water-air compound drop. Several modes were seen at the frequencies predicted by theory; the n=2/sloshing submode (1.9 Hz), the n=3/sloshing submode (3.7 Hz), the n=2/bubble submode (7.0 Hz) and the n=4/sloshing submode (6.3 Hz) were identified.

"The motion of the shell's center of mass was studied for the entirety of the experiment. From the oscillations in the x and y directions the drag on the shells as it moved through the air were determined....

"...The acoustic torque caused the shell to spin up to a rate of 4.1 rps. An imbalance in the acoustic forces distorted the shell and restricted the analysis to a qualitative comparison with the theory of acoustic torques. The shell rotated sufficiently to center the bubble and continued to rotate for 175 seconds after the torque had been removed....

"The shell responded to the modulated acoustic force during each of... three stimulated oscillation sequences. [For example,] When the modulation was swept through the frequency of the n=2/sloshing submode the shell's response was weakly resonant; when the shell was stimulated at the n=3/sloshing frequency its oscillations were very small and non resonant- as expected....

"The bubble expanded as the pressure in the chamber was decreased-its volume decreased by a factor of 4.5." (1, pp. III-95-96)

For a detailed discussions of results see Reference (1), pp. III-95-97.

**Key Words:** Fluid Physics, Containerless Processing Applications, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Standing Waves, Hydrodyanmics, Drop Formation, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Drops, Shells, Drop Dynamics, Fluid Motion Damping, Bubbles, Bubble Dynamics, Bubble Motion, Bubble Centering, Liquid Stability, Bubble Stability, Drop Oscillation, Fluid Oscillation, Bubble Formation, Gas Injection, Adiabatic Expansion, Bubble Rotation, Sample Rotation, Liquid Dynamic Response, Sloshing, Free Surface, Surface Tension, Liquid/Gas Interface, Interface Physics, Acceleration Effects, Sphericity, Sample Deformation

**Number of Samples:** one

**Sample Materials:** distilled water (dyed red for photographic purposes) with a surface tension of 71.0 +/- 2.0 dynes/cm (measured prior to flight)
**Container Materials:** not applicable

**Experiment/Material Applications:**
See Wang, SPAR 7, experiment 77-18/1 (this chapter).

**References/Applicable Publications:**
(1) Croonquist, A. P., Rhim, W.-K, Elleman, D. D., and Wang, T. G.: Containerless Processing Technology Experiment Dynamics of Liquid Bubbles. In Space Processing Applications Rocket Project, SPAR VIII Final Report, NASA TM-82578, pp. III-i - III-103, June 1984. (final report)

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**Experiment Origin:** USA
**Mission:** SPAR 10
**Launch Date/Expt Date:** June 1983
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Acoustic Levitation Space Processing Rocket Instrument (ALSP)
**Builder of Processing Facility:** Unclear, appears to have been Jet Propulsion Laboratory, Pasadena, California

**Experiment:**
Containerless Processing Technology (76/20-3)

This experiment was the fourth in a series of investigations designed by Wang et al. to study the stability, manipulation, and oscillation of liquid drops held in an acoustic field (see Wang, SPAR 4, SPAR 6, and SPAR 7 (experiment 76-20)). The specific objectives of the experiment (as reported in Reference (1)) were to:

(1) Demonstrate the transport capability of the employed acoustic chamber for collision and coalescence of quiescent drops.
(2) Study the mixing effects generated by collision and coalescence of oscillating drops.
(3) Study the shift in natural frequencies of drop oscillation as a function of oscillation amplitude.

<Note: Very little information describing the experimental setup and expected inflight procedure could be located at this time. The following paragraphs were written based on the brief summary provided in Reference (1).>

The experiment was performed in the Acoustic Levitation Space Processing Rocket Instrument (ALSP). <Note: It is not clear if this apparatus was the same as that used by Wang et al. during earlier SPAR flights. Further information about the apparatus design was not presented.>

Reportedly, "Telemetry data [from the SPAR 10 rocket flight] indicates all program sequences operated as planned, except the on-board camera failed at about T + 107 sec... [(]20 sec after the camera was started[)]. All preflight checks had shown normal camera operation....

"Post flight failure analyses indicated the film take-up spool had malfunctioned and film had jammed in the camera. Flight film development showed all functions normal before the film jammed and was torn at the film advance sprocket." (1, p. 8)

No further information concerning this experiment could be located at this time.

**Key Words:** Fluid Physics, Containerless Processing Applications, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Standing Waves, Hydrodynamics, Drop Formation, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Drops, Drop Dynamics, Drop Stability, Liquid Stability, Drop Oscillation, Fluid Oscillation, Drop Coalescence, Liquid Mixing, Droplet Collision, Liquid Dynamic Response, Oscillation Amplitude, Oscillation Frequency, Liquid/Gas Interface, Free Surface, Surface Tension, Photographic Difficulties, Hardware Malfunction

**Number of Samples:** unclear
**Sample Materials:** unknown, probably distilled water
**Container Materials:** not applicable

**Experiment/Material Applications:**
See Wang, SPAR 7, experiment 76/20-3 (this chapter).

**References/Applicable Publications:**
(1) Space Processing Applications Rocket (SPAR) Project; SPAR X (R-20)-Final Report, Section I, Introduction. In Space Processing Applications Rocket (SPAR) Project, SPAR X Final Report, NASA TM-86548, July 1986, pp. 1-11. (this experiment detailed on pp. 7-8)

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**Experiment Origin:** USA
**Mission:** STS Launch #17, STS-024 (STS 51-B, Spacelab 3: Challenger)
**Launch Date/Expt Date:** April 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility
**Processing Facility:** Drop Dynamics Module (DDM) (three-axis acoustic levitator)
**Builder of Processing Facility:** Unknown

**Experiment:**
Dynamics of Rotating and Oscillating Free Drops (DROPS)

The dynamic behavior of rotating bodies (such as planets, stars, or atomic nuclei) which is dependent on gravitational, electrical, and/or nuclear forces, is difficult to model. Terrestrial experimental verification of existing theoretical models has not been possible because of the overwhelming effects of gravity on the body. Immiscible liquids, suspended in a host media (creating a neutrally buoyant system), have been used in the terrestrial laboratory to lessen the effects of gravity. However, results from these experiments cannot be directly correlated to theoretical predictions because the suspending fluid (host media) exerts an inertial drag on the body (viscous forces). In a low-gravity environment, liquid drops can be suspended with an acoustic positioning device without the interference of a suspending liquid.

This Spacelab 3 experiment was the fifth in a series of investigations designed by Wang et al. to study the stability, manipulation, oscillation, and rotation of a liquid drop held in an acoustic field (see Wang, SPAR 4, SPAR 6, SPAR 7, SPAR 10). The specific objectives of the experiment were to study (1) the large amplitude oscillations of a liquid drop, and (2) the equilibrium shapes of a rotating drop.

The experiment was performed in the Drop Dynamics Module (DDM). The module consisted of a three-axis acoustic positioning chamber. During the Spacelab 3 mission, several liquid drops of water or water/glycerin mixtures were positioned (one at a time) within the chamber and rotated at various rates. (Volumes, viscosities, densities, and surface tensions of the employed drops can be found in the Sample Materials section below).

During a typical experimental sequence, a non-rotating drop was positioned within the chamber using three orthogonal acoustic standing wave generators. (The generators placed steady-state forces (of the order of 1 dyne) on the drop.) The drops were then rotated at various rates and directions by applying an acoustically generated torque to the liquid. The application of the torque caused the drop to spin with increasing velocity. The rotational velocity of the drop was determined via immiscible tracer particles suspended within the liquid. The drop motion and drop shape variation with time was recorded along three orthoganol views using a 16 mm camera.

<Note: It is unclear how many DROPS experiments were performed during the Spacelab 3 mission. However, it was reported that "Failure and subsequent repair (in space) of the [experiment] module took precious time; therefore only the experiment studying the shapes of rotating drops was completed...." (5, p. 50) The nature of the failure and repair procedures were not presented in the available publications.>

Data from one experiment were reported as typical and involved spinups and spindowns of a 1 $cm^3$, 100 cS drop. The initially spherical drop was subjected to a forward torque, resulting in a continual increase in rotation velocity. The drop then began to deform with an increasing aspect ratio, a/b (where a is the equatorial radius and b is the polar radius). When the drop attained a particular rotation velocity, (Omega)II, it assummed a two-lobed configuration. At this bifurcation point, the velocity decreased as a result of the larger deformation. If the acoustic torque was maintained, eventual fission of the drop occurred. However, if the torque was shut off, the deformation decreased and the drop assumed an axisymmetric shape. The rotation velocity at which this reverse transition occurred was very close to (Omega)II. Application of a reverse torque resulted in a faster decay of the rotation velocity than if only drag acted on the drop. The reverse torque caused rotation in the opposite direction, with a repetition of the above described cycle.

Quantified data from this experiment, values of (Omega)II, and drop deformation as a function of rotation velocity, were compared to theoretical predictions. Since the magnitude of the applied torques was kept low (low angular acceleration) and the liquid viscosities were relatively high, the experimental system could be compared to an ideal system of a drop with constant rotation velocity and in gyrostatic equilibrium. Because the rotation velocity changed slowly, it was assumed that the speed remained constant on the time scale of the relaxation time (see Reference (3)).

It was reported that the most obvious difference between theory and experiment was the value for the normalized rotation rate at bifurcation, (Omega)II. For a 3 $cm^3$, 100 cS water/glycerin drop, (Omega)II was experimentally determined to be 0.47 +/- 0.004, while it was calculated as 0.56. "In addition, the experimental rate of change of the relative deformation with rotation rate appears less that that predicted by calculations for the two-lobed shape, while the agreement appears to be quite good for the axisymmetric shape." (3, p. 454)

In addition, higher-lobed shapes were not noted during the Spacelab experiment, while theory predicted multi-lobed shapes. "The fact that no higher-number-lobed configurations were obtained does not necessarily preclude the possibility of the existence of higher-order bifurcation points at larger velocity. It is conceivable that the low rotational acceleration used in this particular investigation or the small translational drop oscillations within the potential well adversely affected the probability of bypassing the first bifurcation point to reach higher order lobed shapes." (3, p. 454)

Fission of the drops occurred experimentally after a monotonic increase in the two-lobed deformation followed by a short decrease in that deformation. The drops symmetrically split, occasionally leaving a very small droplet at the center of mass. Experimental data (water/glycerin drop, 1 $cm^3$, 100 cS) indicated fission occurred at a normalized velocity of about 0.21 +/- 0.04. "There is no simple prediction of a critical velocity at fission, but one could compare the data to the velocity calculated for maximum drop deformation (0.26). Calculations also indicate that the rotational kinetic and surface energies for a single two-lobed drop and for two separate drops match in the region where the normalized velocity is equal to 0.25." (3, p. 454)

It was concluded that (1) good quantitative agreement between theory and experiment for the rotational velocity at fission (two-lobed configuration) was achieved, (2) drop shape may be less stable to rotation velocity fluctuations than predicted, and (3) the observation of no mode higher than the two-lobed configuration supports the diminished stability argument.

**Key Words:** Fluid Physics, Containerless Processing Applications, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Standing Waves, Hydrodynamics, Drop Formation, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Drops, Drop Dynamics, Drop Stability, Liquid Stability, Drop Oscilla-

tion, Fluid Oscillation, Drop Rotation, Sample Rotation, Liquid Dynamic Response, Oscillation Amplitude, Oscillation Frequency, Sample Deformation, Liquid/Gas Interface, Free Surface, Surface Tension, Surface Energy, Tracer Particles, Viscosity, Sphericity, Aspect Ratio, Planet Rotation, Hardware Malfunction, Processing Difficulties

**Number of Samples:** unknown
**Sample Materials:** Drops formed from water or water/glycerine mixtures. Drop characteristics were reported as: (1) volume: 0.5 $cm^3$ to 10 $cm^3$, (2) viscosity: 1, 10, 20, and 100 centistokes (cS), (3) density: 1.0 to 1.22 $g/cm^3$, (4) surface tension: 55 to 65 dyne/cm
**Container Materials:** not applicable

**Experiment/Material Applications:**
Theoretical consideration of surface-tension-controlled shapes of liquid drops in gyrostatic equilibrium is a part of the more general problem of the study of dynamics or rotating masses (planets, stars, atomic nuclei). The modeling of this behavior, which is dominated by gravitational, electrical or nuclear forces, is the major motivation behind solving this problem.

**References/Applicable Publications:**

(1) Trihn, E. H., Wang, T. G., Croonquist, A. P., and Elleman, D. D.: Spacelab 3 Drop Dynamics Experiments. AIAA Aerospace Sciences Meeting 24th Reno, Nevada, January 6-9, 1986, AIAA Paper #86-0196.

(2) Spacelab 3 Early Results. Spaceflight, Vol. 27, 1985, p. 361.

(3) Wang, T. G., Trinh, E. H., Croonquist, A. P., and Elleman, D. D.: Shapes Of Rotating Free Drops - Spacelab Experimental Results. Physical Review Letters, Vol. 56, February 3, 1986, pp. 452-455. (post-flight)

(4) Wang, T. G., Trinh, E. H., Croonquist, A. P., and Elleman, D. D.: Dynamics of Rotating and Oscillating Drops. In Spacelab 3 Mission Science Review, Proceedings of a Symposium held at NASA George C. Marshall Space Flight Center, Alabama, December 4, 1985, NASA CP-2429, pp. 27-30. (see also short summary pp. 5-6). (post-flight)

(5) Wang, T. G.: Equilibrium Shapes of Rotating Spheroids and Drop Shape Oscillations. In Advances in Applied Mechanics, eds. Hutchinson, J. W. and Wu, T. Y., Academic Press, San Diego, California, 1988, pp. 1-62.

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**Experiment Origin:** USA
**Mission:** STS Launch #24, STS-032 (STS 61-C, Columbia)
**Launch Date/Expt Date:** January 1986
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Materials Science Laboratory (MSL-2), Payload Bay
**Processing Facility:** Modified SPAR Three-Axis Acoustic Levitator (3AAL)
**Builder of Processing Facility:** Unknown

**Experiment:**
3 Axis Acoustic Levitator (3AAL)

<Note: It is not clear if this experiment was designed to study the low-gravity acoustic stability and manipulation of (1) a liquid drop, (2) a liquid bubble, or (3) both liquid drops and liquid bubbles. Wang et al. have five previous experiments involving the study of liquid drops (see Wang, SPAR 4, SPAR 6, SPAR 7, SPAR 10, STS-024) and two previous experiments involving the study of the dynamic behavior of liquid shells (see Wang, SPAR 7, SPAR 8). (Also related is a Skylab experiment of Wang's with Vaughan (see Vaughan, SL-4 (this chapter)).)>

No documents published after the STS-032 flight could be located which detailed the objectives, experimental setup, or results of this investigation. A document published prior to the flight reported that "Twelve liquid samples will be suspended in sound pressure waves, rotated and oscillated in a low-gravity, nitrogen atmosphere. Investigators will study the degree of sphericity attainable and small bubble migration similar to that having to do with the refining of glass." (2, p. 14)

**Key Words:** Fluid Physics, Containerless Processing Applications, Acoustic Positioning, Acoustic Levitation, Resonant Frequency, Standing Waves, Hydrodynamics, Drop Formation, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Liquid/Gas Interface, Drops, Drop Dynamics, Drop Stability, Liquid Stability, Drop Oscillation, Fluid Oscillation, Bubbles, Bubble Dynamics, Bubble Stability, Bubble Formation, Bubble Migration, Liquid Dynamic Response, Oscillation Amplitude, Oscillation Frequency, Free Surface, Surface Tension, Surface Energy, Sphericity, Glasses

**Number of Samples:** actual, unknown; possibly twelve
**Sample Materials:** unknown
**Container Materials:** not applicable

**Experiment/Material Applications:**
See Wang, SPAR 7 and 8 or Wang, STS-024

**References/Applicable Publications:**
(1) Materials Processing Experiments in Space: MSL-2 Payload. Document Developed by Teledyne Brown Engineering under the direction of the Applications Payload Project Office, Marshall Space Flight Center, pp. 1-2 and 5-7. (MSL-2; processing facility)

(2) Space Shuttle Mission 61-C, Press Kit, December 1985, p. 14. (pre-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 1
**Launch Date/Expt. Date:** December 1977
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-1: An experiment cell equipped with a shutter contained the employed fluids. This cell had a diameter of 25 mm and was 3 mm deep. A schlieren-optical setup combined with a cine camera allowed observation of interface phenomena.
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Interfacial Convection

Surface tension gradients that are due to concentration or temperature gradients within a flow field cause convective flow at liquid-liquid interfaces.

This TEXUS 1 experiment was the first in a series of investigations designed by Brückner to study convective mass transfer driven by an interfacial tension gradient. The specific objective of the experiment was to study the mass transfer of acetic acid through the interface of two immiscible liquids (amyl alcohol and water).

Prior to the initiation of the investigation, a Teflon™ shutter within the experiment cell separated the two immiscible fluids. A solution of amyl alcohol and acetic acid was contained on one side of the shutter and water (colored with methyl orange) was contained on the other.

During the low-gravity phase of the rocket flight, the shutter was slowly removed, thus bringing the fluids into contact. Fluid flow patterns observed using a schlieren-optical arrangement were recorded on 16 mm movie film (20 frames/sec).

Initial disturbances at the interface were due to the movement of the shutter and the spreading of the amyl alcohol-acetic acid solution (held in the upper part of the chamber) along the lower side of the shutter. "This gave rise to inhomogeneities of the boundary surface and therefore to violent interfacial convection (spreading and swirling effects on both sides of the boundary surface), which was connected with a strong deformation of the boundary surface. At first it seemed that the boundary surface would move totally out of the field of view. However, the strong curvature of the boundary surface into the amyl alcohol phase was reduced after a few seconds to such a degree that it could be seen in the whole field of view. The mean radius of curvature was about 2 cm.... The cause of the curvature is the better wettability (contact angle about zero) of the teflon-... [shutter] by the organic solution than by the water.

"On both sides of the boundary surface the violent interfacial convection seen at first decreased slowly. This interfacial convection in turn caused a violent hydrodynamic flow, which again contributed to and maintained the interfacial convection for 170 seconds. After this time the boundary surface was essentially immobile, this behaviour being interrupted only occasionally by small disturbances. It was not possible to correlate exactly these disturbances with the small accelerations recorded during weightlessness...." (1, p. 512) Interface re-excitation occurred when the rocket re-entered the Earth's gravitational field.

Differences between the rocket $10^{-4}$ g low-gravity mass transfer (when a density instability does not exist at the interface) were compared to the 1-g terrestrial mass transfer (when a density instability does exist at the interface). <Note: Although it was reported in the available references that the experimental procedures of the terrestrial experiment duplicated the sounding rocket experimental procedures, the Principal Investigator noted that the procedures between the two experiments differed. These differences were not further discussed.> It was reported that during the terrestrial investigation (within an experiment time comparable to that of the sounding rocket) "..no end of the interfacial convection was obtained. A considerable and much stronger exchange of acetic acid had taken place during that time, as could be seen from the gradual shift of the boundary surface in the direction of the amyl alcohol." (1, p. 514)

It was concluded that the reduced gravity experiment illustrated the expected results: "...initially disturbances of the boundary surface were due to the movement of the... [shutter] and its wetting, which led to an interfacial convection and showed a self-excitation effect by hydrodynamic coupling. After that a decay of the interfacial convection to complete immobility was observed." (1, p. 514)

**Key Words:** Fluid Physics, Immiscible Fluids, Organic Systems, Surface Tension, Surface Tension-Driven Convection, Marangoni Convection, Convection at the Interface, Mass Transfer, Liquid/Liquid Interface, Solid/Liquid Interface, Interface Shapes, Interface Stability, Interface Phenomena, Interfacial Tension, Interfacial Curvature, Surface Tension Gradients, Concentration Distribution, Density Difference, Solutal Gradients, Wetting of Container, Contact Angle, Coated Surfaces, Contained Fluids, Liquid Reservoir, Hydrodynamics, Liquid Dynamic Response, Acceleration Effects, Vehicle Re-Entry Forces/Vibration, Schlieren Viewing

**Number of Samples:** one experiment cell
**Sample Materials:** acetic acid, amyl alcohol, water
**Container Materials:** Shutter: Teflon™; fluid cell: glass. ("The upper half of the glass windows were siliconiced[sic] in order to obtain a flat interface; because the wettability of pure glass by water is better than by amylalcohol[sic]." (2, p. 269))

**Experiment/Material Applications:**
Such research is applicable to the study of corrosion, liquid mixing, liquid extraction, and liquid transition.

**References/Applicable Publications:**
(1) Brückner, R: Interfacial Convection Observed Under Microgravity Conditions During the TEXUS I Experiment. COSPAR Space Research, Vol. XIX, pp. 511-514, ed. by Rycroft, Pergamon Press, Oxford and New York. (post-flight)

(2) Brückner, R.: Fundamental Experiments on Interfacial Convection; Project TEXUS I and II. In Proceedings of the 3rd European Symposium on Material Sciences in Space, Grenoble, 1980, pp. 267-272. (post-flight)

(3) Brückner, R.: Interfacial Convection Under Reduced Gravity. Spacelab Payloads Materials Science and Processing in Space, 1980, pp. 87-95. (post-flight)

(4) Input received From Principal Investigator R. Brückner, May 1988 and July 1993.

(5) Interfacial Convection. In Summary Review of Sounding Rocket Experiments In Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 36-37. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 2
**Launch Date/Expt. Date:** November 1978
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-1: An experiment cell equipped with a shutter contained the employed fluids. "The chamber for the liquids had a diameter of 25 mm and a width of 3 mm." (2, p. 113) A schlieren-optical setup combined with a cine camera allowed observation of the interface phenomena.
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Interfacial Convection

This TEXUS 2 experiment was the second in a series of investigations designed by Brückner to study convective mass transfer driven by an interfacial tension gradient (see Brückner, TEXUS 1).

During this rocket flight, two experiments were performed which investigated fluid/fluid interface instabilities. The specific objective of the first experiment was to study concentration-induced interfacial convection initiated by injecting a drop of glycerin at the interface between two immiscible liquids. The specific objective of the second experiment was to study thermally induced interfacial convection initiated by local heating of the liquid-liquid interface. The experiments were performed one after another, using the same experimental cell. Fluid flow patterns observed using a schlieren-optical arrangement were recorded on 16 mm movie film (20 frames/sec).

Prior to the initiation of the first experiment, two immiscible fluids (water and amyl alcohol) were separated by a tubular shutter within an experiment cell. (The shutter had a steel tip to prevent total wetting by the amyl alcohol.)

During the low-gravity coasting phase of the rocket, the shutter was withdrawn and the two immiscible fluids were brought into contact with one another. The flat interface was allowed to stabilize for 60 seconds. The first experiment was then initiated: a 40 $mm^3$ drop of glycerin (which is miscible in water, but only slightly so in amyl alcohol) was injected at the interface of the immiscible liquids to create an interfacial tension gradient at the boundary. While the glycerin drop contacted the two-fluid interface, it "...did not coalesce with the water phase, because the orifice of the... [shutter] was wetted by the... [amyl alcohol] phase." (2, p. 114) A planned second injection of a 40 $mm^3$ drop of glycerin at the interface was made, but still no coalescence was observed. During a third planned injection, coalescence of a 120 $mm^3$ drop of glycerin with the water was achieved. Immediately, a clockwise-rotating swirl (related to a positive spreading pressure) was noted on the water side of the interface. "The contrast of the swirl on the side of the... [amyl alcohol] was much weaker because of the very low solubility. The rotation of both swirls was continued as long as the glycerin drop was consumed and an interface was left with a curvature of the upper part of the glycerin drop... according to the total solubility of glycerin in water and nearly no solubility in... [amyl alcohol]." (2, p. 115) "The interface was left curved due to the high solubility of glycerin in water and its low solubility in amyl alcohol." (5, p. 38)

Ninety-five seconds after the third glycerin injection, the second experiment was initiated. A heating element which touched the interface was warmed to 80 $^{o}C$ (in 20 seconds) and then cooled during the next 65 seconds. This heating and cooling procedure was repeated two more times, the third time during the rocket's re-entry into the Earth's atmosphere. All three of the heating/cooling procedures "...showed very clearly the dominating spreading effect connected with some swirl formations, which decayed very rapidly after switching off the heater." (2, p. 115)

Ground-based experiments were performed for comparison. These experiments employed similar fluids in the same apparatus. (To prevent the glycerin drops from sinking through the interface prior to the initiation of interfacial convection, the density of the water was increased by adding $Cs_2CO_3$.)

It was reported "In contrast to the micro-g-experiment the coalescence happened immediately during the first injection of glycerin with water, because... [the density of glycerin is greater than that of amyl alcohol]. At once a similar swirl formation with spreading effect started as in the micro gravity experiment during the third injection. However the following characteristic differences could be observed:

"(a) The rotation speed at the beginning rose to about 1 rps for the swirl and to about 1.4 cm/s for the spreading speed in the micro-g experiment, whereas the values of the g-1 experiment reached about 2 rps and 2.6 cm/s respectively, i.e. about double the amount. The...[easiest] preliminary explanation for that behaviour is the assumption, that the interface tension of water/amylalcohol will be increased by the addition of $Cs_2CO_3$ (38 g/l) in a similar way to the surface tension of the pure water. Therefore the decrease of the interface tension will be much stronger by the mixing of water with glycerin when $Cs_2CO_3$ was added to the water than without any $Cs_2CO_3$ content.

"(b) The second and third injection in the g-1 experiment did not lead to interfacial convection, because the interface had been covered totally with a glycerin-water diffusion layer after the decay of the swirls and of the spreading flow from the first injection....

"(c) As a whole, the acting interfacial-energetic equilibration processes under micro gravital[sic] condition could be observed in a much more undisturbed manner than under gravity condition....

"(d) In the heating experiment under g=1 one could see that the normal thermal convection dominated; however, the spreading flow with some swirls, caused by the local temperature increase in the interface, was observable, too but the effects were much weaker than in the microgravity experiment...." (2, pp. 117-118)

Among the conclusions were (1) the experiments illustrated the expected low gravity fluid behavior, (2) interfacial-energetic equilibration processes can "produce remarkable transport actions, especially under micro-g conditions...." (2, p. 118), and (3) spreading and swirling effects were illustrated more clearly during the rocket flight because they were not influenced by the effect of gravity.

**Key Words:** Fluid Physics, Immiscible Fluids, Organic Systems, Surface Tension, Surface Tension-Driven Convection, Marangoni Convection, Convection at the Interface, Thermocapillary Convection, Buoyancy Effects Diminished, Mass Transfer, Solubility, Interface Shapes, Interface Stability, Liquid/Liquid Interface, Solid/Liquid Interface, Interface Phenomena, Interfacial Energy, Interfacial Tension, Interfacial Curvature, Surface Tension Gradients, Solutal Gradients, Density Difference, Concentration Distribution, Drops, Drop Formation, Droplet Injection, Liquid Expulsion Through a Small Orifice, Wetting, Wetting of Container, Contact Angle, Contained Fluids, Liquid Reservoir, Coated Surfaces, Hydrodynamics, Vehicle Re-Entry Forces/Vibration, Schlieren Viewing

**Number of Samples:** one experiment cell
**Sample Materials:** glycerin, water, amyl alcohol
**Container Materials:** Shutter: Teflon™ ("The top of the... [shutter] was made of... steel... to prevent a total wetting of the... [shutter] by the organic liquids...." (2, p. 114)); experiment cell: glass. ("The upper half of the glass windows were coated with silicon to obtain a flat interface; because the wettability of pure glass by water is better than by amylalcohol[sic]." (2, p. 114))

**Experiment/Material Applications:**
Such research is applicable to the study of corrosion, liquid mixing, liquid extraction, and liquid transition.

**References/Applicable Publications:**

(1) Brückner, R.: Fundamental Experiments on Interfacial Convection; Project TEXUS I and II. In Proceedings of the 3rd European Symposium on Material Sciences in Space, Grenoble, 1980, pp. 267-272. (post-flight, TEXUS 1,2)

(2) Brückner, R.: Interfacial Convection. Shuttle/Spacelab Utilization Final Report, Project TEXUS II, 1978, pp. 109-120. (post-flight)

(3) Brückner, R.: Interfacial Convection Under Reduced Gravity. Spacelab Payload Materials Science and Processing in Space, 1980, pp. 87-95. (TEXUS 1,2)

(4) Input received from Principal Investigator R. Brückner, May 1988 and July 1993.

(5) Interfacial Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 38-39. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3
**Launch Date/Expt. Date:** April 1980
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-1: An experiment cell equipped with a shutter contained the employed fluids. A schlieren-optical setup allowed observation of the interface phenomena.
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
<u>Diffusion-Induced Interfacial Convection</u>

This TEXUS 3 experiment was the third in a series of investigations designed by Brückner to study convective mass transfer driven by an interfacial tension gradient (see Brückner, TEXUS 1; TEXUS 2).

A description of the TEXUS 3 experimental setup and in-flight experiment procedure was not presented.

Reportedly, TEXUS 3 did not achieve the desired low-gravity level due to a rocket despin failure. Documentation detailing any results of this TEXUS 3 experiment could not be located at this time.

The experiment was reflown on TEXUS 3b (see Brückner, TEXUS 3b).

**Key Words:** Fluid Physics, Mass Transfer, Diffusion, Liquid/Liquid Interface, Interface Phenomena, Interfacial Tension, Surface Tension, Surface Tension Gradients, Surface Tension-Driven Convection, Marangoni Convection, Convection at the Interface, Contained Fluids, Liquid Reservoir, Acceleration Effects, Rocket Despin Failure

**Number of Samples:** unknown
**Sample Materials:** unknown
**Container Materials:** unknown

**Experiment/Material Applications:**
See Brückner, TEXUS 1.

**References/Applicable Publications:**

(1) Greger, G: TEXUS and MIKROBA and Their Effectiveness and Experimental Results. Presented at In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP). (identifies rocket failure)

(2) Input received from Principal Investigator R. Brückner, May 1988 and July 1993.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3b
**Launch Date/Expt. Date:** April 1981
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module: TEM 06-1: An experiment cell equipped with a shutter contained the employed fluids. A schlieren-optical setup allowed observation of the interface phenomena.
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Diffusion-Induced Interfacial Convection

This TEXUS 3b experiment was the fourth in a series of investigations designed by Brückner to study convective mass transfer driven by an interfacial tension gradient (see Brückner, TEXUS 1, TEXUS 2, TEXUS 3). The specific objectives of the research were to (1) investigate fluid/fluid interface instabilities caused by interface concentration gradients (during transfer of a solute from one liquid into another) and (2) compare the observed instabilities to the theoretical predictions of Sternling and Scriven.

Prior to the initiation of the experiment, a Teflon™ shutter within the experiment cell separated two immiscible fluids: (1) 68.6 vol% of component C (acetone) dissolved in 31.4 vol% component A (toluene) and (2) an equal volume of component B (water). (The acetone-toluene mixture was expressed symbolically by A(C).)

During the low-gravity coasting phase of the rocket, the shutter was withdrawn and the two immiscible fluids were brought into contact. (The shutter had "...a steel tip to avoid the preferential wetting of the teflon by one of the liquids." (3, p. 40)) Component C then crossed the interface and was dissolved in B (symbolically expressed by B(C)). (Accordingly, the phase boundary moves in the A direction.) Reportedly, "Although not in the centre of the cell, the interface formed was reasonably plane. A strong initial interfacial convection was induced, which lasted 43 s and suddenly stopped. 79 s later, interfacial convection occurred in steps. Periods of intense activity alternated with relatively long periods of almost no activity. The pseudo-periodic transfer of matter was driven by the Marangoni effect.

It led to a concentration of acetone of 24 vol % in water at the end of the microgravity period, and the interface progressed towards the toluene." (3, p. 40)

Reportedly, according to theorists Sternling and Scriven, convective transport is possible only when the following conditions are fulfilled:

$$D_A^C / D_B^C < 1 < v_A^C / v_B^C \quad \text{and} \quad d(\text{gamma}) / dc_C < 0. \quad (1)$$

Where $D_A^C$ and $D_B^C$ are diffusion coefficients of C in A and C in B,

$v_A^C$, $v_B^C$ are kinematic viscosities,

gamma is the interface tension between A(C) and B(C),

and $c_C$ is the concentration of component C.

According to equation (1), the low-gravity created interface should have been stable. It was reported above, however, that interface convection (attributed to the concentration gradient) was observed. Thus, it was concluded that the Sternling-Scriven conditions are not always valid.

"In a similar experiment carried out on Earth, the transfer of acetone after the decay of the perturbations induced by the retraction of the partition was only driven by diffusion since the configuration was stable with respect to the density differences." (3, p. 40)

**Key Words:** Fluid Physics, Immiscible Fluids, Organic Systems, Liquid/Liquid Interface, Interface Stability, Interface Physics, Interfacial Tension, Interface Phenomena, Hydrodynamics, Mass Transfer, Surface Tension, Surface Tension Gradients, Surface Tension-Driven Convection, Marangoni Convection, Convection at the Interface, Diffusion, Diffusion Coefficient, Solutal Gradients, Density Difference, Wetting of Container, Contained Fluids, Liquid Reservoir, Schlieren Viewing

**Number of Samples** one experiment cell
**Sample Materials:** Fluids: 31.4 vol% toluene, 68.6 vol % acetone, water
**Container Materials:** shutter: Teflon™ with steel tip; experiment cell: unknown, possibly glass

**Experiment/Material Applications:**
See Brückner, TEXUS 1.

**References/Applicable Publications:**

(1) Brückner, R.: Nuere Ergebnisse über die Grenzflächen Konvektion unter 1g-und $\mu$g- Beohingungen. In: Spacelab-Nutzung, Status-Seminar 1981 des BMFT; Deutsche Gesellschaft für Luft-und Raumfahrt e.V., S. 57-73, Publ. 1982. (TEXUS 3, 3b, 4)

(2) Input received from Principal Investigator R. Brückner, May 1988 and July 1993.

(3) Diffusion Induced Interfacial Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 40-41. (post-flight)


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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 4
**Launch Date/Expt. Date:** May 1981
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-1: An experiment cell (28 mm high, 30 mm wide and 10 mm deep) equipped with a shutter contained the employed fluids. A He-Ne laser differential interferometer, coupled with a 16 mm camera, allowed observation of the cell.
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Temperature-Gradient Induced Interfacial Convection

This TEXUS 4 experiment was the fifth in a series of investigations designed by Brückner to study convective mass transfer driven by an interfacial tension gradient (see Brückner, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b). The specific objectives of the research were to (1) observe thermally induced Marangoni convection (resulting at the boundary between two immiscible fluids) and (2) compare the resultant convective flow with theoretical predictions.

Prior to the initiation of the experiment, a shutter separated equal volumes of water and n-dodecane within the experiment cell. During the experiment (prior to the removal of the shutter), (1) convection in the fluids generated by the launch and despin of the rocket were allowed to decay (during the initial 180 seconds of low-gravity time), and (2) a linear temperature gradient was imposed on the system (to initiate and sustain the interfacial Marangoni flow). "One lateral wall was heated in order to achieve a temperature difference of 30 $^{o}$C across the cell." (3, p. 42) The shutter separating the two immiscible fluids was then to be removed, bringing the liquids into contact.

It was reported that although an exact linear temperature profile was obtained in each liquid 240 seconds after launch, the shutter separating the two fluids was not removed as planned. Thus, thermally induced convective flow was not observed. However, interferograms, documented using a He-Ne differential interferometer and a 16 mm camera, "...showed that the establishment of a linear thermal gradient required about 100 s whereas numerical simulations indicated that the convection in the liquids was damped about 15 s after the beginning of the microgravity period.... The time needed for the establishment of a linear temperature profile is therefore the determining factor for the timing of such experiments." (3, p. 42)

During similar ground-based experiments (with the shutter successfully removed) a temperature difference of 30 K was established between the hot and cold wall of the cell, and Marangoni flow was observed.

**Key Words:** Fluid Physics, Immiscible Fluids, Liquid/Liquid Interface, Solid/Liquid Interface, Interfacial Tension, Interface Phenomena, Surface Tension, Surface Tension-Driven Convection, Surface Tension Gradients, Marangoni Convection, Thermocapillary Convection, Convection at the Interface, Fluid Motion Damping, Liquid Dynamic Response, Mass Transfer, Diffusion, Thermal Gradient, Contained Fluids, Liquid Reservoir, Interferometric Measurement, Acceleration Effects, Hardware Malfunction

**Number of Samples:** one experiment cell
**Sample Materials:** water, n-dodecane
**Container Materials:** The front and back walls of the experiment cell were made of optical glass. The compositions of the other walls were not detailed.

**Experiment/Material Applications:**
See Brückner, TEXUS 1

**References/Applicable Publications:**

(1) Brückner, R.: Neuere Ergebnisse über die Grenzflächen Konvektion unter 1g- und $\mu$g- Bedhingungen. In: Spacelab-Nutzung, Status-Seminar 1981 des BMFT; Deutsche Gesellschaft für Luft-und Raumfahrt e.V., S. 57-73, Publ. 1982. (TEXUS 3, 3b, 4)

(2) Input received from Principal Investigator R. Brückner, May 1988 and July 1993.

(3) Thermally Induced Interfacial Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 42-43. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 6
**Launch Date/Expt. Date:** May 1982
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-6: An experiment cell equipped with a shutter contained the employed fluids. A differential interferometer, coupled with a 16 mm camera, allowed observation of the experiment cell.
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Thermally-Induced Interface Convection

This TEXUS 6 experiment was the sixth in a series of investigations designed by Brückner et al. to study convective mass transfer driven by an interfacial tension gradient (see Brückner, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4). The specific objective of the experiment was to study thermally induced Marangoni convection (resulting at the boundary between two immiscible fluids).

A rectangular glass cuvette contained the two immiscible fluids: water and n-Dodecane. The fluids within this cuvette were (1) separated prior to the initiation of the experiment by a shutter, and (2) brought into contact as the shutter was removed. When a linear thermal gradient (delta T = 25 $^{o}$C) was applied parallel to the interface during the mission, convective motion was recorded by means of an interferometer and a motion picture camera.

It was reported that very little flow due to the interface temperature gradient could be observed and that the interference patterns (attained via a differential interferometer) were "weak." These weak patterns hampered quantitative analysis supported by computer simulation. It was concluded that a better theoretical treatment combined with additional experiments (applying larger thermal gradients) were needed.

**Key Words:** Fluid Physics, Immiscible Fluids, Liquid/Liquid Interface, Interfacial Tension, Surface Tension, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Convection at the Interface, Mass Transfer, Thermal Gradient, Contained Fluids, Liquid Reservoir, Solid/Liquid Interface, Interferometric Measurement

**Number of Samples:** one experiment cell
**Sample Materials:** water, n-Dodecane
**Container Materials:** Fluid container: glass cuvette; shutter material: unspecified

**Experiment/Material Applications:**
See Brückner, TEXUS 1.

**References/Applicable Publications:**

(1) Brückner, R. and Christ, H.: Final Report TEXUS V/VI, DFVLR-BPT, 1982. <Note: This document was not available to the editors at the time of the experiment summary was written.>

(2) Input received from Principal Investigator R. Brückner, May 1988, July 1993.

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**Co-Investigator(s):** None
**Affiliation(s):** (1,2) Institute for Non-Metallic Materials, Technical University, Berlin, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 8
**Launch Date/Expt. Date:** May 1983
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-1: An experiment cell equipped with a shutter contained the employed fluids. A schlieren-optical setup allowed observation of the interface phenomena.
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Diffusion-Induced Interface Convection

This TEXUS 8 experiment was the seventh in a series of investigations designed by Brückner et al. to study convective mass transfer driven by an interfacial tension gradient (see Brückner, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 6). The specific objective of this experiment was to examine the fluid/fluid interface instabilities caused by interface concentration gradients.

The experiment was directly related to the earlier TEXUS 3b experiment by Brückner. During TEXUS 3b, two immiscible fluids ((1) acetone dissolved in toluene and (2) water)) were brought into contact during the coasting phase of the sounding rocket. Reportedly, acetone crossed the interface and was transported into the water.

This TEXUS 8 experiment was designed to investigate the reverse case of TEXUS 3b. According to theory, such a transport of acetone from a water-acetone mixture into toluol should result in different convective modes at the interface.

Prior to the initiation of the TEXUS 8 experiment, a shutter within the experiment cell separated (1) acetone dissolved in water and (2) toluol. "The same acetone concentration (water-acetone: 1-2 in volume) as in the previous experiment was chosen in order to obtain quantitatively comparable results." (4, p. 44)

During the low-gravity coasting phase of the rocket, the shutter was withdrawn and the two fluids were brought into contact. Fluid flow patterns, observed using a schlieren-optical arrangement, were recorded by a cine camera.

Reportedly, acetone crossed the interface and was transported into the toluol "...about 40 seconds after starting to move the slide (which separated the two immiscible fluids), two eddys[sic] formed on each side of the interface, and they grew continually. After about 40... [seconds]... test time, one eddy on each side of the interface was consumed by the neighboring eddy, so that in each phase there was only one double eddy, which continued to grow at a constant rate, until the entire cell was filled by this double eddy and then, because of the occurring equalization of concentration, it collapsed." (2, translated)

Reportedly, the TEXUS 8 results were qualitatively in agreement with the theory of (1) Sternling and Scriven and (2) Sanfeld et al. <Note: These theories are discussed further under Brückner, TEXUS 11 (this chapter).> Estimates of the magnitude of vortices, based on the theory of Sternling and Scriven, were also in good agreement with experimental values. It was further reported that this was a surprising result because while the theory was derived for low concentrations, large concentrations were used in the TEXUS 8 experiments.

No further information published in English was available.

**Key Words:** Fluid Physics, Immiscible Fluids, Organic Systems, Liquid/Liquid Interface, Interface Stability, Hydrodynamics, Mass Transfer, Surface Tension, Surface Tension Gradients, Interface Phenomena, Interfacial Tension, Surface Tension-Driven Convection, Marangoni Convection, Convection at the Interface, Diffusion, Solutal Gradients, Density Difference, Contained Fluids, Liquid Reservoir, Solid/Liquid Interface, Schlieren Viewing

**Number of Samples:** one experiment cell
**Sample Materials:** acetone dissolved in water, toluol
**Container Materials:** unknown, possibly glass

**Experiment/Material Applications:**
See Brückner, TEXUS 1

**References/Applicable Publications:**

(1) Brückner, R. and Christ, H.: Diffussbedingde Grenzflächen-Konvektion. Final Report, TEXUS VIII, DFVLR-BPT, 1984.

(2) Brückner, R. and Christ, H.: Diffusion-Induced Interface Convection. In TEXUS 11/12 Abschlussbericht 1985, DFVLR. (discusses TEXUS 8 as well as TEXUS 11; in German, post-flight)

(3) Input received from Principal Investigator R. Brückner, May 1988 and July 1993.

(4) Diffusion-Induced Interfacial Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, p. 44. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 11
**Launch Date/Expt. Date:** April 1985
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-1: An experiment cell equipped with a shutter contained the employed fluids. (The experiment module had been modified since TEXUS 3b and 8 to now include larger fluid cells.) A schlieren-optical setup allowed observation of the interface phenomena.
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Diffusion-Induced Interface Convection

This TEXUS 11 experiment was the eighth in a series of investigations designed by Brückner et al. to study convective mass transfer driven by an interfacial tension gradient (see Brückner, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 6, TEXUS 8). The specific objectives of the experiment were to (1) examine fluid/fluid interface instabilities caused by interface concentration gradients, and (2) clarify whether an oscillatory or stationary interface instability exists during the material transition of acetone from water into the toluene phase.

The two earlier TEXUS experiments which examined instabilities caused by interface concentration gradients (TEXUS 3b and 8) had employed a high acetone concentration (66.7% by volume). During this experiment, however, a low acetone concentration was employed (30% by volume) to allow comparison of theoretical instability criteria with experimental results.

Prior to the initiation of the TEXUS 11 experiment, a shutter within a large experiment cell separated (1) acetone and water and (2) toluene. During the low-gravity coasting phase of the rocket, the shutter was withdrawn and the two immiscible fluids were brought into contact. The fluid flow patterns were observed using a schlieren-optical system coupled with a 16 mm camera.

Reportedly, the interface which formed was 34 mm long (which was 2.5 times as long as the interface observed during the TEXUS 3b and TEXUS 8 experiments). "The flight film shows that the interface convection activity in the larger cell and at the much lower concentration difference, proceeds quite differently than was expected from the TEXUS 8 experiment.... [T]he flight film of the TEXUS 11 experiment showed a whole series of... [eddies] along the interface. At the beginning of the test there is a strong pulsating eddy over the entire length of the very smooth interface, which lasts about 30... [seconds].... With increasing test time, the... [eddies] grow without additional, noticeable pulsation." (1, translated) Thus, the fluid system demonstrated a violent, pulsating, interfacial convective instability at the beginning of the experiment which transitioned to a stationary instability. (A further discussion of the analysis of the flight film is presented in Reference (4).)

In the 1-g reference experiment, the material transition, which was directed into the opposite direction (because of the density stability), illustrated more intensive interfacial convection than was seen in the low-gravity experiment.

The comparison of the low-g and 1-g experiments with the instability criteria of (1) Sternling and Scriven and (2) Sanfeld et al. (with the complete data of the system acetone-toluene-water) indicated that qualitative agreement exists only with the low-gravity experiment. Better agreement seems to exist with the theory of Sanfeld, et al. (however, not in a quantitative manner). Specific publications which describe the theory are detailed in References (1) and (2) (as listed below).

**Key Words:** Fluid Physics, Immiscible Fluids, Organic Systems, Liquid/Liquid Interface, Interface Phenomena, Interfacial Tension, Interface Stability, Hydrodynamics, Mass Transfer, Surface Tension, Surface Tension Gradients, Surface Tension-Driven Convection, Marangoni Convection, Convection at the Interface, Diffusion, Solutal Gradients, Density Difference, Contained Fluids, Liquid Reservoir, Solid/Liquid Interface, Schlieren Viewing

**Number of Samples:** one experiment cell
**Sample Materials:** acetone in water, toluene
**Container Materials:** unknown, possibly glass

**Experiment/Material Applications:**
See Brückner, TEXUS 1.

**References/Applicable Publications:**

(1) Brückner, R. and Christ, H.: Diffusion Induced Interface Convection. In TEXUS 11/12 Abschlussbericht 1985, German Publication, 1985. (post-flight, in German)

(2) Christ, H.: Grenzflächen Konvektion unter 1g- und $\mu$g- Bedingungen. Diss. D83, Berlin, 1987. (A Ph.D. thesis discussing all the space experiments and many terrestrial experiments, measurements of material properties and proofing theories)

(3) Input received from Principal Investigator R. Brückner, May 1988 and July 1993.

(4) Diffusion-Induced Interfacial Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 46-47. (post-flight)

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<Note: Chun is currently working part-time at Pohang University, South Korea.>

**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3
**Launch Date/Expt. Date:** April 1980
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-4
**Builder of Processing Facility:** DFVLR, AVA, Göttingen, Germany

**Experiment:**
Marangoni Convection in Float Zones

This TEXUS 3 sounding rocket experiment was the first in a series of investigations designed by Chun et al. to study thermally induced Marangoni convection within a liquid bridge.

Reportedly, due to a rocket despin failure, TEXUS 3 did not achieve the desired low-gravity level. The experiment was reflown on TEXUS 3b (see Chun, TEXUS 3b).

Documentation concerning the results of this TEXUS 3 experiment does not appear to be available.

**Key Words:** Fluid Physics, Liquid Bridges, Free Surface, Surface Tension, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Heat and Mass Transfer, Thermal Gradient, Liquid/Gas Interface, Containerless Processing Applications, Acceleration Effects, Rocket Despin Failure

**Number of Samples:** unknown
**Sample Materials:** unknown
**Container Materials:** unknown

**Experiment/Material Applications:**
See Chun, TEXUS 3b (this chapter).

**References/Applicable Publications:**

(1) Greger, G: TEXUS and MIKROBA and Their Effectiveness and Experimental Results. Presented at In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP). (identifies rocket failure)

(2) Input received from Experiment Investigator, May 1988 and August 1993.


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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3b
**Launch Date/Expt. Date:** April 1981
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-4: Marangoni Chamber (A liquid bridge was formed between two coaxial copper disks.)
**Builder of Processing Facility:** DFVLR-AVA, Göttingen, Germany

**Experiment:**
Marangoni Convection in Float Zones

This TEXUS 3b sounding rocket experiment was the second in a series of investigations designed by Chun et al. to study thermally induced Marangoni convection within a liquid bridge (see Chun, TEXUS 3). Reportedly, visualization of such Marangoni convection had not yet been realized in a reduced-gravity environment.

During the low-gravity phase of the flight, a 10 mm long, 10 mm diameter, silicone oil liquid bridge was formed between two coaxial copper disks. (The oil contained Ti and $TiO_2$ tracer particles to permit visualization of the convective flow pattern.) Fifteen seconds after formation of the bridge, one of the disks was heated, subjecting the column to a constant temperature difference (T1-T2) of 7.82 K. After approximately 150 seconds, a higher temperature difference of 18.9 K was implemented across the bridge for approximately 200 seconds. Reportedly, these two temperature differences within the chosen system corresponded to Marangoni numbers of "$6.10^3$ and $16.10^3$," respectively. The resultant convective flow field was illuminated by a laser beam (0.1 mm thick). The beam illuminated a meridian plane of the bridge which was filmed by a 16 mm camera. Because the frame speed was known, flow velocities could be ascertained from the film by following tracers within the bridge.

Reportedly, the thermal gradient extending across the free surface of the bridge initiated an axisymmetric steady Marangoni flow pattern. <Note: It is not clear at what temperature dif-

ference this Marangoni flow was first observed.> It was further reported that "Although particles had a tendency to stick to the rods, they allowed the visualization of the convective flows in the floating zone. A thin thermal boundary layer could be observed along the gas/liquid interface (Marangoni layer). In the case of the high thermal gradient, two velocity maxima could be measured near the hot and the cold ends of the zone. This was in agreement with theoretical [finite difference solution] predictions. A small corner vortex embedded in the large vortex could also be clearly observed." (9, p. 56)

Reference (1) details the computational efforts.

<Note: Not all the references as listed below were available for review by the editors.>

**Key Words:** Fluid Physics, Liquid Bridges, Free Surface, Surface Tension, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Heat Transfer, Thermal Gradient, Viscosity, Flow Velocity, Tracer Particles, Liquid/Gas Interface, Solid/Liquid Interface, Containerless Processing Applications

**Number of Samples:** One liquid bridge across which was placed two separate temperature differences.
**Sample Materials:** silicone oil (AK 10)
**Container Materials:** Not applicable. (The bridge was formed between two copper coaxial disks.)

**Experiment/Material Applications:**
In the reduced gravity environment, Marangoni convection is often the dominating convective force in free surface experiments. Thermal or solutal gradients along the surface result in interfacial temperature gradients which drive this gravity-independent flow. If low-gravity production of new/or high quality materials by floating zone processes is to be realized, the importance and control of such convective forces must be understood.

**References/Applicable Publications:**

(1) Chun, Ch.-H.: Numerical Study on the Thermal Marangoni Convection and Comparison with Experimental Results from the TEXUS Rocket Program. 33rd International Astronautical Congress, Paris, France, September 27-October 2, 1982, Paper #82-155, 6 pp. (post-flight)

(2) Chun, Ch.-H. and Wuest, W.: A Micro-Gravity Simulation of the Marangoni Convection. 28th International Astronautical Federation, International Astronautical Congress, Prague, Czechoslovakia, September 25-October 1, 1977, 9 pp., IAF Paper #77-203.

(3) Chun, Ch.-H. and Wuest, W.: Experiments on the Transition from Steady to Oscillatory Marangoni Convection in a Floating Zone Under Reduced Gravity Effect. Acta Astronautica, 1073 (1979), 6 pp. (no sounding rocket results)

(4) Chun, Ch.-H. and Wuest, W.: Thermische Marangonikonvektion in einer Schwebezone-$\mu$g Experiment wahrend des Raketenfluges von TEXUS IIIb. Zeitschrift fur Flugwiss. und Weltraumforsch. (ZFW), 1982. <Note: The current publication status of this document is unclear. It was supposed to be published as of 1982.>

(5) Chun, Ch.-H.: Marangoni Convection in a Floating Zone Under Reduced Gravity. Journal of Crystal Growth, 48 (1980), pp. 600-610. (preflight computational analysis)

(6) Input received from Experiment Investigator, May 1988 and August 1993.

(7) Chun, Ch.-H. and Wuest, W.: Flow Phenomena in Rotating Floating Zones with and without Marangoni Convection. Proc. of 3rd European Symposium on Material Sciences in Space, Grenoble, April 24-27, 1979, ESA SP-142 (June 1979). (preflight computational analysis)

(8) Chun, Ch.-H. and Wuest, W.: Correlation of Oscillations of Flow and Temperature in Floating Zone Under Microgravity. Adv. Space Res., Vol. 1, pp. 17-20. (appears to be preflight computational analysis)

(9) Thermal Marangoni Convection in a Floating Zone. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 56-57. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 7
**Launch Date/Expt. Date:** May 1983
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-4. The module contained several items including (1) an experiment chamber to create a liquid bridge between two coaxial copper disks and (2) a light cut illumination unit with He-Ne laser.
**Builder of Processing Facility:** DFVLR-AVA, Göttingen, Germany

**Experiment:**
<u>Oscillatory Marangoni Convection in Float Zones</u>

This TEXUS 7 sounding rocket experiment was the third in a series of experiments designed by Chun et al. to study thermally induced Marangoni convection within a liquid bridge (see Chun, TEXUS 3, TEXUS 3b). While steady, thermal Marangoni convection had been observed on TEXUS 3b, it was expected that such steady convection would have become "...oscillatory-instable[sic] if a certain threshold of the temperature difference... [delta]T, between the top and bottom side of the cylindrical column... [had been] exceeded." (1, p. 271)

Different theories explaining the transition to oscillatory, convective flows are available, one of which is briefly described in Reference (1). Essentially, the theory (which is a model of the physical mechanism of the instability) suggests that the transition is related to the Marangoni number and floating zone aspect ratio.

Thus, the objectives of this experiment were:

(1) To examine liquid bridges whose corresponding Marangoni numbers were far beyond the critical values at which the onset of oscillatory instabilities were expected to occur.

(2) To observe the oscillatory convection in the liquid bridge and determine if the oscillatory state progresses to a turbulent state.

(3) To study the role of the liquid column aspect ratio on (a) the turbulent transition and (b) the column stability.

The TEXUS 7 liquid bridge apparatus consisted of two, co-axial, 10 mm diameter copper disks, between which liquid bridges of methanol could be formed. One of the disks could be heated, imposing the necessary thermal gradient across the bridge to initiate the Marangoni convection. Two thermocouples, attached to the unheated copper disk, extended into the liquid bridge to measure temperature fluctuations. Flow visualization of the bridge's meridian plane was possible using a light-cut illumination unit with a He-Ne laser, an imaging optic and a 16 mm movie camera. Visualization of the fluid flow was made possible by a suspension of titanium metal powder in the methanol.

Prior to the start of the experiment, the copper disks were in contact with each other. During the rocket flight, methanol was delivered through a hole in the middle of one of the disks as it was retracted, and a liquid bridge was formed between the disks. In the first part of the experiment, the disk was retracted 3.3 mm. Approximately 50 seconds later, one of the disks was heated, subjecting the bridge to a constant temperature difference of 7.6 K (Marangoni number = $9.4 \times 10^4$) Such temperature and aspect ratio parameters resulted in a periodic temperature fluctuation of the system. An oscillating flow was demonstrated by "...the up-and downwards oscillating Marangoni vortex near the free surface of [the] liquid column." (1, p. 276) Reportedly, "...a regular oscillatory flow was detected with three distinct frequencies (0.59, 0.86 and 1.16 Hz)." (5, p. 58)

In the second part of the experiment, the disk was further retracted to 10 mm. "While the liquid column... [was] extended the Marangoni vortices penetrate[d] into the middle of the column and... [when the disk had been retracted to 10mm (l/d=1.0),] the whole volume of the column... [was] seized by the flow. In this phase a small bubble of about 1 mm diameter appears in the column...."

"For the enlarged column... [delta] T... [was] kept... [at] the same value of 7.6K.... The only thing changed... [was] the aspect ratio from 0.33 to 1.0. The effect of the aspect ratio change... [was] really striking; the temperature signals $T_{z1}$ and $T_{z2}$... [ceased] to oscillate and exhibit[ed] no periodicity.... [T]he convection... [became] non-periodic in consequence of the large aspect ratio... the visualized flow pattern on [the] meridian plane of the column... [showed] clearly the non-periodic turbulent character of the convection...." (1, pp. 276-277) As the temperature difference was increased from 7.6 K to 10 K (Marangoni number = $1.25 \times 10^5$ "...full turbulent convection with large scale structure fluctuations exhibiting non-periodic three dimensional flow..." was visualized.

**Key Words:** Fluid Physics, Liquid Bridges, Liquid Bridge Stability, Free Surface, Liquid Stability, Fluid Oscillation, Oscillation Modes, Surface Tension-Driven Convection, Marangoni Convection, Oscillatory Marangoni Convection, Thermocapillary Convection, Time Dependent Thermocapillary Flow, Steady to Oscillatory Convective Transition, Heat Transfer, Thermal Gradient, Turbulent Flow, Liquid Transfer, Liquid Expulsion Through a Small Orifice, Bubbles, Bubble Formation, Tracer Particles, Liquid/Gas Interface, Aspect Ratio, Containerless Processing Applications

**Number of Samples:** one
**Sample Materials:** methanol with titanium metal powder
**Container Materials:** The bridge was formed between two copper disks.

**Experiment/Material Applications:**
Methanol was employed as the test liquid in this study because its properties, coupled with the experimental parameters, resulted in the desired oscillatory Marangoni numbers.

Additional applications can be found under Chun, TEXUS 3b.

**References/Applicable Publications:**
(1) Ch.-H. Chun: Verification of Turbulence Developing from the Oscillatory Marangoni Convection in a Liquid Column. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss, Elmau, November 5-7, 1984, ESA SP-222, pp. 271-280.

(2) Chun, Ch.-H. and Wuest, W.: Correlation of Oscillations of Flow and Temperature in Floating Zone Under Microgravity. Adv. Space Res., Vol. 1, pp. 17-20. (preflight computational analysis)

(3) Chun, Ch.-H.: Marangoni Convection in a Floating Zone Under Reduced Gravity. Journal of Crystal Growth, 48 (1980), pp. 600-610. (preflight computational analysis)

(4) Input received from Experiment Investigator, May 1988 and August 1993.

(5) Oscillatory and Turbulent Flow Regimes Due to Marangoni Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 58-59. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3
**Launch Date/Expt. Date:** April 1980
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-1: a single $NaNO_3$ sample was configured between two graphite cylinders. A temperature difference was imposed across the sample.
**Builder of Processing Facility:** TEM 06-1 was built by ERNO, Bremen, Germany; the sample experiment cell was built by Justus-Liebig-Universität Giessen, Germany.

**Experiment:**
Critical Marangoni Number--Temperature Oscillations in Float Zones

Thermal or solutal gradients within a float zone system tend to drive two types of convective flow: (1) gravity-dependent, buoyancy-driven flow and (2) gravity-independent, surface tension-driven flow. On Earth, the gravity-dependent flow often masks the underlying contribution of other fluid dynamic phenomena to the system. In a low-gravity environment, the overwhelming effects of buoyancy-driven convection are reduced, allowing a closer examination of the role of surface tension forces.

The primary surface tension-driven force in a float zone is known as Marangoni convection. Thermal (or solutal) gradients along the free surface of the float zone drive the Marangoni flow. The flow results because fluid elements on the free surface with a low surface tension (usually corresponding to a hotter temperature) tend to flow to fluid areas of higher surface tension (usually corresponding to colder temperatures). In a low-gravity environment, this surface tension-driven convection will be a significant force in the fluid dynamics of the zone. Because the float zone process is one in which a material is melted and then resolidified, axial and longitudinal thermal (and solutal) gradients are sure to exist in the zone acting as drivers of this convective flow. Moreover, the Marangoni convection can become time dependent, often producing oscillatory temperature gradients within the system and thus reducing the compositional homogeneity of the processed crystal.

This TEXUS 3 experiment was the first in a series of investigations designed by Schwabe et al. to examine oscillatory, thermocapillary Marangoni convection in a float zone. The specific objective of the experiment was to determine if oscillatory Marangoni convection would occur in the low-gravity environment.

The experiment chamber housed a single, 6-mm diameter, 4.0-mm long $NaNO_3$ sample. <Note: Reference (1), p. 185, indicates that the zone length was 3.4 mm, while Reference (5) indicates that the length was 4.0 mm. The Principal Investigator verified the 4.0-mm length.> The sample was configured between two graphite cylinders, which could be independently heated and controlled. Two thermocouples measured the temperature distribution in the zone. The first thermocouple was inserted into the zone through one of the graphite cylinders (in an effort to avoid disturbances of the free surface). The second thermocouple appears to have been located at one of the zone ends on the graphite rod heater. A pyrolytic coating was placed on the graphite resulting in a melt/cylinder contact angle of approximately $90^o$, and steps were taken to suppress the convection of the air surrounding the sample.

Because the $NaNO_3$ melt is transparent, direct visualization of the flow is possible by monitoring the motion of small spheres within the fluid. It appears that the ground-based experimental setup was configured with (1) an illumination system which provided a vertical light cut to illuminate the particles and (2) a camera to document the flow field. (Such photographs require correction because the cylindrical zone creates a lens effect.) However, the actual TEXUS 3 hardware did not employ such a visualization/camera system.

During the mission, two critical temperature differences were placed across the graphite heaters (delta T = 40 K and delta T = 90 K). Each temperature difference was applied for approximately 1 minute.

Post-flight it was reported that "Unfortunately TEXUS IIIa failed to despin to the desired low value, and a rest spin of approximately 1... [+/-] 0.1 Hz remained during the ballistic phase. Therefore... [the] floating zone, being located 14 cm off the axis of the rocket, experienced a centrifugal force perpendicular to the zone axis of about 0.4g." (1, p. 185)

Analysis of the flight data from the thermocouple in the molten $NaNO_3$ bridge demonstrated thermal oscillations in the zone and indicated that both temperature differences resulted in oscillatory convection. Similar ground-based experiments of the $NaNO_3$ system at the same temperature differences also demonstrated the oscillatory behavior. Further, the same frequency of oscillation

(0.75 Hz) occurred for both 1-g and low-gravity samples for both temperature differences. It was very interesting to note that although a centrifugal force (described above) was imposed on the system, "...the liquid zone was stable and showed the oscillatory state of... [Marangoni convection] with a frequency different from the spin frequency. We can interpret the result in the sense that temperature oscillations due to oscillatory... [Marangoni convection] are rather independent of the value and orientation of gravity in such a floating zone." (1, p. 185)

This interpretation was further proved by examining the experiment on Earth with the floating zone axis configured perpendicular to the 1-g vector. "The liquid zone was still stable under 1-g and... [Marangoni-driven convection] was the dominating flow.... The typical temperature and flow oscillations were found in this experiment as well but only at [a] higher Marangoni number. The gravity component perpendicular to the zone axis stabilizes... [the Marangoni convection] which, therefore, stays laminar for higher flow speed than in the case with the gravity parallel to the axis." (1, p. 185)

It was concluded that (1) oscillatory convection can occur in the low-gravity rocket environment and (2) the critical Marangoni number (for the transition to time dependence) will be smaller in low-gravity than on Earth.

**Key Words:** Fluid Physics, Float Zones, Liquid Columns, Liquid Bridges, Melt and Solidification, Free Surface Solidification, Transparent Liquids, Free Surface, Surface Tension Gradients, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Fluid Stability, Thermal Gradient, Thermal Oscillations, Oscillatory Marangoni Convection, Oscillation Frequency, Time Dependent Thermocapillary Flow, Steady to Oscillatory Convective Transition, Critical Marangoni Number, Binary Systems, Rotation of Payload, Coated Surfaces, Hydrostatic Pressure, Contact Angle, Wetting, Tracer Particles, Spheres, Liquid/Gas Interface, Solid/Liquid Interface, Containerless Processing Applications, Acceleration Effects, Rocket Despin Failure

**Number of Samples:** One floating zone. (Two temperature differences were placed across the zone during the flight.)
**Sample Materials:** $NaNO_3$ (sodium nitrate)
(Na*N*O*)

**Container Materials:** The free surface zone was maintained between two graphite rods.
(C*)

**Experiment/Material Applications:**
While a reduction of buoyancy-driven convection is achieved in low gravity, detrimental oscillatory Marangoni convection can still occur, resulting in a reduction of compositional homogeneity in the processed system. This research explores the role of the gravity-independent surface tension-driven convection in the reduced gravity environment.

Although the available literature did not detail further research applications, low-gravity floating zone processing has several attractive features other than a reduction of buoyancy-driven flow. For example, the low-gravity contribution results in a reduction of the hydrostatic pressure. Such a reduction in pressure allows longer, more stable zones to be formed. From these longer zones, larger crystals can be formed. Further, the zone is a partially containerless system (contained only at its ends). Such a system eliminates wall effects such as contamination and nucleation at the cylindrical zone wall. In addition, the absence of an ampoule wall allows unconstrained material expansion during freezing, preventing sample breakage and other constrainment effects.

**References/Applicable Publications:**
(1) Schwabe, D., Scharmann, A., and Preisser, F.: Studies of Marangoni Convection in Floating Zones. Acta Astronautica, Vol. 9, No. 3, 1982, pp. 183-186. (flight 3a, post-flight)

(2) Input received from Principal Investigator D. Schwabe, August 1989 and August 1993.

(3) Schwabe, D. and Scharmann, A.: Some Evidence for the Existence and Magnitude of a Critical Marangoni Number for the Onset of Oscillatory Flow in Crystal Growth Melts. Journal of Crystal Growth, 46, 125 (1979). (preliminary analysis; preflight)

(4) Schwabe, D., Scharmann, A., Preisser, F., and Oeder, R.: Experiments on Surface Tension Driven Flow in Floating Zone Melting. Journal of Crystal Growth, 43, 1978, pp. 305-312.

(5) Oscillatory Marangoni Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 48-51. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3b
**Launch Date/Expt. Date:** April 1981
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-2: A single $NaNO_3$ sample was configured between two graphite cylinders. A temperature difference was imposed across the sample. <Note: The Principal Investigator reported that there were no differences between TEM 06-1 (the facility used during Schwabe's earlier TEXUS 3 float zone experiment) and the TEM 06-2 facility employed on this mission.>
**Builder of Processing Facility:** TEM 06-2 was built by ERNO, Bremen, Germany; experiment chamber built by Justus-Liebig-Universität Giessen, Germany

**Experiment:**
Critical Marangoni Number--Temperature Oscillations in Float Zones

This TEXUS 3b experiment was the second in a series of investigations designed by Schwabe et al. to examine oscillatory, thermocapillary Marangoni convection in a float zone (see Schwabe, TEXUS 3).

In an effort to directly compare 1-g and low-g experimental data, the floating zone scenario examined during this TEXUS 3b experiment (1) was stable under 1-g, (2) demonstrated dominating thermocapillary convection under 1-g, and (3) demonstrated oscillatory thermocapillary convection under 1-g. Thus, the objectives of the experiment did not include the investigation of longer zones that are stable only in a reduced gravity environment. (The benefits of processing longer zones can be found under Schwabe, TEXUS 3.)

The experimental setup appears to have been very similar to the TEXUS 3 experiment. The experiment chamber housed a single, 6-mm diameter, 3.4-mm long $NaNO_3$ sample. The sample was configured between two graphite rods, which could be independently heated and controlled. A thermocouple was inserted into the zone through one of the graphite rods and one or more thermocouples was/were positioned 0.5 mm below the front of the graphite rods. A pyrographited coating was applied to insure a proper contact angle ($90^o$). Further, "The mantle outside the zone (and feed through of the thermocouple)... [was] a capillary tube from quartz glass which was pyrographited to avoid capillary outflow of liquid $NaNO_3$ under $\mu g$." (1, p. 267)

Although the $NaNO_3$ melt is transparent and direct visualization of the flow is possible by monitoring the motion of small spheres within the fluid, the experimental setup was not configured with an illumination/camera system to document the fluid motion. Instead, thermocouple measurements of the temperature oscillations were to be used to detect the oscillatory thermocapillary convection.

Prior to the rocket flight, a zone was established in the ground-based laboratory and solidified. The sample was placed in the rocket between the graphite rods and 10 minutes before the rocket launch, the solid sample was preheated below the melting point (the temperature of both heaters was brought to 290 $^{o}C$). The heaters remained at this temperature until 70 seconds after launch. At this time, the temperature of one of the heaters was raised to 360 $^{o}C$ (at a rate of 0.65 $^{o}C$/sec), while the temperature of the other heater was raised to 320 $^{o}C$. The zone was fully molten at 150 seconds, and by this time the low-gravity phase of the rocket had been attained. About 3 minutes into the flight, a temperature difference of 40 K (corresponding to a Marangoni number Ma = 1.59 X $10^4$) was established between the two graphite rods. This difference was maintained until approximately 5 minutes into the flight. During the following 50 seconds, the temperature of the hotter heater was raised to 400 $^{o}C$ and the temperature of the cooler heater was dropped to 310 $^{o}C$ (corresponding to a Marangoni number of 3.57 X $10^4$). This 90 $^{o}C$ temperature difference appears to have been maintained until the end of the low-gravity period. Temperature data of the heaters and temperature data from the zone were sent via telemetry to a magnetic tape recorder on the ground.

Reportedly, because the same $NaNO_3$ sample was used for both the 1-g and low-gravity experiments, the contribution of the sample impurities (in, for example, suppressing Marangoni convection) was similar. Further, both Marangoni numbers examined were higher than the expected critical number. (The critical number being that Marangoni number at which oscillatory, time-dependent, convective flow occurs.)

Post-flight analysis of the thermal data indicated temperature oscillations occurred in both the low-gravity and ground-based experiments. Temperature vs. time graphs of the rocket and laboratory experiments (documented at 240-280 seconds after launch and 200 to 300 seconds after the initiation of the ground-based experiment at a Marangoni number of Ma = 1.6 X $10^4$) illustrated the nature of the temperature oscillations. Interesting results were:

"(1) Temperature oscillations occur under 1-g and $10^{-4}$ g (with the same frequency)... [as shown by Fourier analysis].

"(2) The average temperature is smaller under $10^{-4}$ g (325 °C) than under 1-g (327 °C).

"(3) The amplitude of the oscillations is smaller under $10^{-4}$ (peak to peak 3.3 °C) than under 1 g (peak to peak (9 °C))." (1, p. 267)

Similar time vs. temperature graphs for the Marangoni number at 3.6 X $10^4$ (documented at 380 seconds to 420 seconds after launch and 350 seconds to 450 seconds after the start of the reference experiment) were presented. Interesting results were:

"(1) Temperature oscillations with the same higher frequency and with a more complex spectrum... [as shown by Fourier analysis] occur under 1-g and $10^{-4}$ g.

"(2) The average temperature under $10^{-4}$ g (343-344 °C) is smaller than under 1-g (345 °C). (3) The amplitude of the oscillations is smaller under $10^{-4}$ g (...[approximately] 5K peak to peak) than under 1-g (...[approximately] 15K peak to peak.)" (1, p. 267)

Results (2) and (3) as listed above for both Marangoni numbers were cited as being of minor importance. The temperature oscillations observed under both Marangoni numbers in low-gravity "...are clearly larger by orders of magnitude than any temperature fluctuations from the controllers and are due to flow oscillations, therefore."

"...These oscillations have a characteristic frequency spectrum which is very similar under 1-g and $10^{-4}$g. The lowest main frequency is 0.79 Hz under low-gravity and 0.80 Hz under 1-g for Ma=1.6 X $10^4$ with a squared amplitude approximately three orders of magnitude above noise level. Some harmonics appear as well. For the higher... [Marangoni] number Ma=3.6 X $10^4$, the lowest main frequency is 1.2 Hz under 1-g and $10^{-4}$-g. The shift to higher frequency with higher Ma is expected because of higher flow speed for higher Ma. The frequency spectrum is not as sharp as for Ma=1.6 X $10^4$ in both cases with a squared amplitude of the lowest main frequency only two orders of magnitude above noise level. Higher harmonics are clearly visible in to $10^{-4}$ case, only." (1, p. 267)

Among the conclusions were: "The same characteristic frequency spectrum occurred under 1-g and... [$10^{-4}$g] at two supercritical Marangoni numbers. From this it is concluded that the same oscillatory flow state of the thermocapillary convection developed under low-gravity as under 1-g. Thermocapillary convection in the floating zone becomes... [unstable] at a certain Marangoni number, and this instability is not due to a coupling of thermocapillary and buoyancy forces, but it is one of thermocapillary convection alone. Thermocapillary convection will become... [unstable] in the relevant low-gravity fluid dynamic experiments, if the critical Marangoni number of this transition is exceeded." (1, p. 272)

**Key Words:** Fluid Physics, Float Zones, Liquid Columns, Liquid Bridge Stability, Melt and Solidification, Free Surface Solidification, Transparent Liquids, Free Surface, Surface Tension Gradients, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Fluid Stability, Thermal Gradient, Thermal Oscillations, Oscillatory Marangoni Convection, Oscillation Frequency, Oscillation Amplitude, Time Dependent Thermocapillary Flow, Steady to Oscillatory Convective Transition, Critical Marangoni Number, Buoyancy Effects Diminished, Capillary Flow, Coated Surfaces, Wetting, Contact Angle, Impurities, Hydrostatic Pressure, Binary Systems, Liquid/Gas Interface, Solid/Liquid Interface, Containerless Processing Applications

**Number of Samples:** One floating zone. (Two temperature differences were placed across the zone during the flight.)
**Sample Materials:** $NaNO_3$ (sodium nitrate)
(Na*N*O*)
**Container Materials:** The free surface zone was maintained between two graphite rods.
(C*)

**Experiment/Material Applications:**
See Schwabe, TEXUS 3.

**References/Applicable Publications:**
(1) Schwabe, D., Preisser, F., and Scharmann, A.: Verification of the Oscillatory State of Thermocapillary Convection in a Floating Zone Under Low Gravity. Acta Astronautica, Vol. 9, No. 4, 1982, pp. 265-273. (post-flight)

(2) Schwabe, D., Preisser, F., and Scharmann, A.: Z. Flugwiss. Weltraumforsch, 6 (1982), pp. 309-315.

(3) Scharmann, A. and Schwabe, D.: Final Report "...Oscillatory Marangoni Convection..." for (BMFT) Federal Ministry for Research and Technology, May 1982. (in German)

(4) Preisser, F., Schwabe, D., and Scharmann, A.: Steady and Oscillatory Thermocapillary Convection in Liquid Columns with Free Cylindrical Surface. Journal of Fluid Mechanics, 126 (1983), pp. 545-567. (ground-based studies)

(5) Input received from Principal Investigator D. Schwabe, August 1989 and August 1993.

(6) Schwabe, D. and Scharmann, A.: Oscillatory Marangoni Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 48-51. (post-flight)

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**Co-Investigator(s):** None
**Affiliation(s):** (1,2) Justus-Liebig-Universität Giessen, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 5
**Launch Date/Expt. Date:** April 1982
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-2: A single $NaNO_3$ sample was configured between two graphite cylinders. A temperature difference was imposed across the sample.
**Builder of Processing Facility:** TEM 06-2 was built by ERNO, Bremen, Germany; experiment chamber built by Justus-Liebig-Universität Giessen, Germany.

**Experiment:**
Critical Marangoni Number--Temperature Oscillations in Float Zones

This TEXUS 5 experiment was the third in a series of investigations designed by Schwabe et al. to examine oscillatory, thermocapillary Marangoni convection in a float zone (see Schwabe, TEXUS 3, TEXUS 3b).

During previous float zone experiments on TEXUS 3 and TEXUS 3b (1) a specific temperature difference (corresponding to an expected critical Marangoni number) was placed across a 6-mm diameter, 3.4-mm (or 4.0-mm) long $NaNO_3$ floating zone and (2) time dependent, oscillatory fluid transport behavior was noted. The objective of this TEXUS 5 experiment was to determine (for the given system) at what critical Marangoni number time-dependent fluid transport would occur.

Nondimensional analysis of the governing fluid equations (1-g) and terrestrial experimental results illustrated that thermocapillary forces dominated buoyancy forces in floating zones which are stable on Earth. (Such stable zones had a length of less than 7 mm.) Buoyancy forces became significant, however, in zones longer than 3.5 mm. Further, terrestrial experiments indicated that the longer the zone, the higher the Marangoni number required to produce a transition in the fluid from a non-oscillatory to an oscillatory flow state. The data also indicated that as the zone length increased beyond 3.5 mm (somewhat regardless of radius), a fairly sharp increase occurred in the value of the corresponding required critical Marangoni number.

The TEXUS 5 experimental setup appears to have been very similar to the setup used during the TEXUS 3 and TEXUS 3b experiments. The experiment chamber housed a single, 6-mm diameter, 3.7-mm long $NaNO_3$ sample.

The sample was configured between two graphite cylinders which could be independently heated and controlled. A thermocouple inserted near the center of the zone (l/2, r/2) (through one of the graphite rods) measured the local temperature. As in the earlier TEXUS 3b flight, (1) the graphite cylinders were coated to insure the proper contact angle and (2) the mantle was pyrographited to avoid capillary outflow of the test liquid (see Schwabe, TEXUS 3b). Thermocouple measurements alone (rather than visualization of the flow via an illumination/camera system) were used to determine the oscillatory thermocapillary convection.

Available references provided few specific details concerning (1) the preheating of the sample prior to launch and (2) the time during the flight (or temperature) at which the sample was molten. It appears, however (from a figure in Reference (6)), that both graphite cylinders were preheated to a temperature of 300 $^{o}$C prior to launch. The cylinders remained at this temperature until approximately 80 seconds after launch (at this time the low-gravity phase of the rocket flight had been achieved). Then, (80 seconds after launch), the temperature of one of the rods was raised to approximately 315 $^{o}$C and the temperature of the other rod was raised to approximately 325 $^{o}$C. (The lower temperature heater remained at 315 $^{o}$C for the duration of the low-gravity phase.) At approximately 120 seconds after launch, the 10 K temperature difference was established across the zone, and the temperature of the warmer rod was linearly increased at a rate of 0.1 K/s. This increase in temperature of the warmer rod continued until just prior to the end of the low-gravity phase (approximately 420 seconds after launch).

Analysis of the thermal data resulting from both the rocket and terrestrial experiments indicated that the onset of temperature oscillations occurred at a temperature difference of 23 K. The corresponding critical Marangoni number for the flight was 9.6 X $10^3$, which was identical to the critical Marangoni number in the 1-g reference experiment. However, Reference (6) reported that "The microgravity experiment was slightly disturbed by the vibrations of the cine camera of a neighboring experiment and the exactly identical critical Marangoni number measured on the ground and in microgravity... are interpreted as accidental." (6, p. 52) Further, "The spectrum of oscillation frequencies at 1-g and 0-g are comparable with a main frequency at 0.5 Hz." (6, p. 52) "In the case of the low-gravity experiment there is more low frequency and high frequency noise besides the approximately 0.5 Hz thermocapillary oscillation, reducing the precision of the measurements by 10%." (1, p. 216) The precision of the rocket thermal measurements was also degraded by the insufficient thermal equilibrium of the system during the short rocket flight.

**Key Words:** Fluid Physics, Float Zones, Liquid Columns, Transparent Liquids, Melt and Solidification, Free Surface Solidification, Free Surface, Surface Tension Gradients, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Fluid Stability, Thermal Gradient, Thermal Equilibrium, Thermal Oscillations, Oscillatory Marangoni Convection, Oscillation Frequency, Time Dependent Thermocapillary Flow, Steady to Oscillatory Convective Transition, Critical Marangoni Number, Aspect Ratio, Buoyancy Effects Diminished, Coated Surfaces, Wetting, Contact Angle, Accelerations/Vibrations Produced by Onboard Equipment, Acceleration Effects, Binary Systems, Liquid/Gas Interface, Solid/Liquid Interface, Hydrostatic Pressure, Containerless Processing Applications

**Number of Samples:** one floating zone
**Sample Materials:** $NaNO_3$ (sodium nitrate)
(Na*N*O*)
**Container Materials:** The free surface zone was maintained between two graphite rods.
(C*)

**Experiment/Material Applications:**
See Schwabe, TEXUS 3.

**References/Applicable Publications:**
(1) Schwabe, D. and Scharmann, A.: Measurement of the Critical Marangoni Number in a Floating Zone Under Reduced Gravity. In Proceedings of the 4th European Symposium on Material Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, pp. 213-218. (post-flight)

(2) Schwabe, D. and Scharmann, A.: Microgravity Experiments on the Transition from Laminar to Oscillatory Thermocapillary Convection in Floating Zones. Adv. Space Res., Vol. 4, No. 5, 1984, pp. 43-47. (includes TEXUS 8; post-flight)

(3) Schwabe, D. and Scharmann, A.: Adv. Space Res., Vol. 3, No. 5, pp. 89-92, (1983).

(4) Schwabe, D. and Scharmann, A.: Z. Flugwiss. Weltraumforsch., 9, (1985), pp. 21-28.

(5) Input received from Principal Investigator D. Schwabe, August 1989 and August 1993.

(6) Critical Marangoni Number. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 52-53. (post-flight)

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**Co-Investigator(s):** None
**Affiliation(s):** (1,2) Justus-Liebig-Universität Giessen, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 8
**Launch Date/Expt. Date:** May 1983
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3: A single $NaNO_3$ sample was configured between two graphite cylinders. A temperature difference was imposed across the sample. (The hardware was refurbished after TEXUS 5.)
**Builder of Processing Facility:** TEM 06-3 was built by ERNO, Bremen, Germany; the experiment chamber was built by Justus-Liebig-Universität Giessen, Germany

**Experiment:**
Critical Marangoni Number--Temperature Oscillations in Float Zones

This TEXUS 8 experiment was the fourth in a series of investigations designed by Schwabe et al. to examine oscillatory, thermocapillary Marangoni convection in a float zone (see Schwabe, TEXUS 3, TEXUS 3b, TEXUS 5).

During the earlier TEXUS 5 investigation, a temperature difference, placed across a 6-mm diameter, 3.7-mm long $NaNO_3$ floating zone, was raised linearly at a rate of 0.1 K/s to determine at what point time-dependent, oscillatory fluid transport behavior would occur. Because the TEXUS 5 experiment was unexpectedly disturbed by the operation of motion picture cameras associated with another experiment in the TEXUS 5 module, the experiment was repeated (with slightly different parameters) during this TEXUS 8 mission.

This experiment employed the shortest zone yet examined by the investigators, 3.2 mm. <Note: The reason the zone length was changed from TEXUS 5 was not presented in the available references.> The experimental setup was similar to the setup used during the TEXUS 3, TEXUS 3b, and TEXUS 5 experiments. The single, 6-mm diameter, $NaNO_3$ sample was configured between two graphite cylinders which could be independently heated and controlled. A thermocouple inserted near the center of the zone (l/2, r/2, through one of the graphite rods) measured the local temperature. The graphite cylinders were coated to insure the proper contact angle and the mantle pyrographited to avoid capillary outflow of the test liquid (see Schwabe, TEXUS 3b). Thermocouple measurements alone (rather than visualization of the flow via an illumination/camera system) were used to determine the presence of oscillatory thermocapillary convection.

Available references provided few specific details concerning (1) the preheating (if any) of the sample prior to launch and (2) the specific times at which the temperature difference was applied. It appears that both graphite cylinders may have been preheated to a temperature of approximately 300 $^{o}$C prior to launch. Reference (1) confirmed that 130 seconds after launch, the sample was molten (the sample's melting point was 307 $^{o}$C as stated by Reference (5)).

<Note: If the rocket time schedule for TEXUS 8 was similar to that of the TEXUS 5 flight, the low-gravity phase of the rocket would have been achieved approximately 50 seconds before the sample was molten.> After thermal equilibrium of the sample had been realized, a 15 $^{o}$C temperature difference was placed across the two graphite cylinders. This temperature difference was increased at a rate of 0.85 $^{o}$C/s until a temperature difference of 35 $^{o}$C was attained.

Thermal data obtained during the rocket flight and a similar terrestrial reference experiment were compared. The results indicated that (1) the onset of temperature oscillations in the rocket sample occurred at 270 seconds (corresponding to a critical temperature difference of 22.7 $^{o}$C) and (2) the onset of temperature oscillations in the terrestrial sample occurred at 330 seconds (corresponding to a critical temperature difference of 27.3 $^{o}$C). The corresponding critical Marangoni numbers were 8.5 X $10^3$ (rocket) and 10.2 X $10^3$ (Earth). Fourier analysis of the fully developed oscillatory state as indicated by the temperature vs. time thermocouple data "...show[ed] frequencies $f_1$ = 0.67 Hz and $f_2$ = 1.35 Hz under low-gravity in comparison to $f_1$=0.71 Hz and $f_2$ = 1.4 Hz under 1-g. Higher frequencies under 1-g compared to low-gravity have been found in TEXUS 3... TEXUS 5 and 8...." (1, p. 44)

It was surmised that "Both effects, smaller... [critical Marangoni number] and... [frequency] under low-gravity in comparison to 1-g can be understood considering the stabilizing effect of heating from above in the 1-g experiments: heating from above hinders... [thermocapillary convection] under 1-g to reach the bottom area of the... [float zone]. Thus under 1-g... [thermocapillary convection] dominates in an 'effective zone length' $l_{eff}$ which is smaller than the geometrical zone length l which is operative under low-gravity." (1, p. 44)

**Key Words:** Fluid Physics, Float Zones, Liquid Columns, Transparent Liquids, Melt and Solidification, Free Surface, Free Surface Solidification, Surface Tension Gradients, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Fluid Stability, Thermal Equilibrium, Thermal Gradient, Thermal Oscillations, Oscillatory Marangoni Convection, Oscillation Frequency, Time Dependent Thermocapillary Flow, Critical Marangoni Number, Coated Surfaces, Contact Angle, Binary Systems, Liquid/Gas Interface, Containerless Processing Applications

**Number of Samples:** one floating zone
**Sample Materials:** $NaNO_3$ (sodium nitrate)
(Na*N*O*)
**Container Materials:** The free surface zone was maintained between two graphite rods.
(C*)

**Experiment/Material Applications:**
See Schwabe, TEXUS 3.

**References/Applicable Publications:**


(1) Schwabe D. and Scharmann, A.: Microgravity Experiments on the Transition from Laminar to Oscillatory Thermocapillary Convection in Floating Zones. Adv. Space Res., Vol. 4, No. 5, 1984, pp. 43-47. (post-flight)

(2) Schwabe, D. and Scharmann, A.: "...Messung der kritischen Marangoni-Zahl...," Final Report for the BMFT, May 1984. (in German)

(3) Schwabe, D. and Scharmann, A.: Z. Flugwiss. Weltraumforsch., 9, (1985), pp. 21-28.

(4) Input received from Principal Investigator D. Schwabe, August 1989 and August 1993.

(5) Schwabe, D. and Scharmann, A.: Critical Marangoni Number. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 54-55. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #7, STS-007 (STS 31-C, Challenger)
**Launch Date/Expt. Date:** June 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** West German Get Away Special (GAS) MAUS Canister DG-306 (MAUS: Materialwissenschftliche autonome Experimente unter Schwerelosigkeit); SPAS STS Deployed Satellite
Volume of Canister: 5.0 cubic feet
Location of Canister: The West German Shuttle Pallet Satellite (SPAS-01)
(SPAS was a small experiment carrier initially configured in the STS payload bay but later deployed into orbit by the Canadian Remote Manipulator Arm. The carrier was retrieved prior to the end of the shuttle mission.)
**Primary Developer/Sponsor of DG-306:** Deutsche Forschungs-und Versuchsanstalt für Luft-und Raumfahrt (DFVLR)/Messerchmitt-Boelkow-Blohm (MBB-ERNO), Bremen, Germany
**Processing Facility:** Two Experiment Cells within MAUS DG-306. Each experiment cell contained a single, $NaNO_3$ sample across which an increasing temperature difference was placed.
**Builder of Processing Facility:** MAUS DG-306 was built by MBB/ERNO, Bremen, Germany; the two experiment cells were built by the University of Giessen, Germany

**Experiment:**
Critical Marangoni Number

This STS-007 experiment was the fifth in a series of investigations designed by Schwabe et al. to examine oscillatory, thermocapillary Marangoni convection in a float zone (see Schwabe, TEXUS 3, TEXUS 3b, TEXUS 5, and TEXUS 8).

The specific objective of the experiment was to differentially heat a $NaNO_3$ sample (in a float zone configuration) and determine at what critical Marangoni number there occurs a transition from (1) laminar thermocapillary convection to (2) time-dependent oscillatory thermocapillary convection. This objective was to be realized by increasing the temperature difference across the zone until the laminar thermocapillary flow developed into oscillatory thermocapillary flow. The experiment was also designed to demonstrate a transition from this oscillatory state back into the laminar thermocapillary state (by reducing the temperature difference across the zone).

The payload was configured with two experiment cells. Each cell housed a single, solid, 6-mm diameter $NaNO_3$ sample configured between two graphite cylinders. The length of the sample differed in each of the cells to demonstrate the influence of the zone length on the resulting oscillatory critical Marangoni number. Reportedly (Reference (1)) one of the samples had a length of 4.0 mm and the other had a length of 4.79 mm. <Note: Reference (9) indicates that the lengths of the two zones were 3.86 mm and 4.75 mm.>

During the mission, the experiment was housed in the DG-306 West German MAUS Get Away Special (GAS)-type payload. DG-306 was configured on the German free-flying platform (commercial satellite) SPAS 01. (SPAS 01 was stored in the STS payload bay during launch and deployed by the Canadian Remote Manipulator System (RMS) during the mission. At the end of the STS-007 mission, SPAS 01 was retrieved and stored in the payload bay by the RMS.) The experiment was performed during the SPAS 01 free-flying phase. Reportedly, "The satellite SPAS-01 experienced very low residual gravity (of the order of $10^{-5}$ g) during the time the experiment was active." (1, p. 281)

During the investigation, the rods in both experiment cells were simultaneously heated. Each melted sample formed a floating zone suspended between the rods. A thermocouple inserted into each zone (through one of the graphite rods) measured the local temperature. "After a thermalisation [sic] of 30 minutes the temperature difference... between the two graphite rods was increased 'linearly' in time... to pass the transition point (laminar... [thermocapillary flow] to oscillatory [thermocapillary flow])...." (1, p. 281) ("The rate of heating up was of the order of $+3.6 \times 10^{-3}$ K*s-1.... Actually the temperature changes were in steps: every 100 s the temperature difference was increased by 0.36 K....") (1, p. 282) This heating phase continued for 99.75 minutes.

Once the oscillatory state had been reached, the temperature difference between the two rods was decreased "linearly" at a rate of $-7.2 \times 10^{-3}$ K/s to pass the transition point from oscillatory to laminar flow. ("Actually the temperature changes were in steps: every 100 s the temperature difference was... decreased by .26 K every 50 seconds during cooling down.") (1, p. 282) Thermocouple measurements were used to determine the oscillatory thermocapillary convection.

Reportedly, "Both experiments performed well." (1, p. 283) Post-flight analysis of the thermal data from both experiment cells indicated that

(1) during the time the temperature difference increased, the laminar state existed for approximately 50 minutes, and the oscillatory state existed for approximately 50 minutes, and

(2) during the time the temperature difference decreased, oscillations occurred for approximately 30 minutes, and then no oscillations occurred during the following 20 minutes.

The critical Marangoni numbers and oscillation amplitudes, corresponding to (1) the increase in temperature and (2) the decrease in temperature, were significantly different. These differences were attributed to hysteresis effects which affect the transition point in the fluid system.

Post-flight, the thermal data obtained during the STS flight were compared to thermal data obtained during similar terrestrial reference experiments. The results indicated that the low-gravity critical Marangoni numbers of both zones are of the same order of magnitude as the terrestrial critical numbers. However, the low-gravity numbers were lower than the 1-g numbers for the 4.79-mm zone and higher than the 1-g numbers for the 4.0-mm zone. (The critical Marangoni numbers were reported as follows (Reference (9)): (1) 4.75 mm zone (1983 terrestrial experiments), temperature increasing: 10075, temperature decreasing: 9409; (2) 4.75 mm zone (1984 terrestrial experiments), temperature increasing: 9848, temperature decreasing: 9337; (3) 3.86 mm zone (1984 terrestrial experiments) temperature increasing: 8489, temperature decreasing: 8003; (4) 4.75 mm zone (STS-007), temperature increasing: 9293, temperature decreasing: 8877; (5) 3.86 mm zone (STS-007), temperature increasing: 8679, temperature decreasing: 8115.) <Note: The two critical Marangoni numbers and two STS-007 zone lengths reported directly above (from Reference (9)) differed slightly from the two critical Marangoni numbers and two STS-007 zone lengths reported in Reference (1) (9702 increasing, 8848 decreasing (4.79 mm zone) and 9444 increasing, 8340 decreasing (4.00 mm zone). Reference (1) also lists slightly different values for the terrestrial experiments as well (see Reference (1), p. 286).>

Further, "The temperature oscillations (and flow oscillations under $\mu$-g) are of significant amplitude, comparable to those observed under 1-g.

"A new feature of oscillatory thermocapillary convection has been found under $\mu$-g and 1-g namely frequency jumps and beat frequencies. The beat frequencies are of special interest considering real growth systems like float-zone silicon, because oscillatory flows (and growth conditions) are detected indirectly via dopant striations in the grown crystal." (1, p. 289)

**Key Words:** Fluid Physics, Float Zones, Liquid Columns, Transparent Liquids, Melt and Solidification, Free Surface, Free Surface Solidification, Heat Transfer, Surface Tension Gradients, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Fluid Stability, Thermal Equilibrium, Thermal Gradient, Thermal Oscillations, Oscillatory Marangoni Convection, Time Dependent Thermocapillary Flow, Steady to Oscillatory Convective Transition, Critical Marangoni Number, Oscillation Frequency, Oscillation Amplitude, Binary Systems, Liquid/Gas Interface, Containerless Processing Applications, Acceleration Effects, Acceleration Measurements, Free-Flying Satellite

**Number of Samples:** two experiment cells
**Sample Materials:** $NaNO_3$ (sodium nitrate)
(Na*N*O*)
**Container Materials:** The zones were contained between two graphite rods.
(C*)

**Experiment/Material Applications:**
"In crystal growth[,] time independent growth conditions are favourable because time dependent conditions can give rise to dopant inhomogeneities (compositional striations). In order to avoid such striations in $\mu$-g float zone crystal growth[,] the Marangoni number (given by the growth conditions) must be lower than the critical number $Ma_c$ for transition to oscillatory flow." (1, p. 281)

**References/Applicable Publications:**

(1) Schwabe, D. and Scharmann, A.: Measurements of the Critical Marangoni Number of the Laminar <-> Oscillatory Transition of Thermocapillary Convection in Floating Zones. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 281-289. (post-flight)

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(3) Velten, R., Schwabe, D., and Scharmann, A.: Gravity Dependence of the Instability of Surface-Tension Driven Flow in Floating Zones. In Proceedings of the 7th European Symposium on Material Sciences in Microgravity, Oxford, September 1985, accepted for ESA SP Conference Proceedings.

(4) Schwabe, D., Velten, R., and Scharmann, A.: The Instability of Surface Tension Driven Flow in Models for Floating Zones Under Normal and Reduced Gravity. Submitted to Journal of Crystal Growth, Proceedings of ICCG 7, Sendai, 1989, Japan. <Note: The current publication status of this document is unclear.>

(5) Baum, D., Otto, G. and Vits, P.: MAUS-A Flight Opportunity for Automated Experiments Under Microgravity Conditions. Acta Astronautica, Vol. 11, No. 3-4, pp. 239-245, 1984. (does not discuss experiment results)

(6) Baum, D., Stolze, H., and Vits, P.: First Flight Data From MAUS Payloads on STS 7 and STS 11. 35th Congress of the International Astronautical Federation, October 7-13, 1984, Lausanne, Switzerland, IAF-84-137, 11 pp. (post-flight)

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(8) Input received from Principal Investigator D. Schwabe, August 1989 and August 1993.

(9) Schwabe, D., and Scharmann, A.: Microgravity Experiments on the Transition from Laminar to Oscillatory Thermocapillary Convection in Floating Zones. Adv. Space Res., Vol. 4, 1984, pp. 43-47. (post-flight)

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**Co-Investigator(s):** Lamprecht, R. (2), Scharmann, A. (3)
**Affiliation(s):** (1-3) Justus-Liebig-Universität Giessen, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Process Chamber Double Rack
**Processing Facility:** Rectangular cavity with free surface
**Builder of Processing Facility:** Partly built by the Justus-Liebig-Universität, Giessen, Germany, partly built by Messerschmitt-Boelkow-Blohm (MBB-ERNO), Bremen, Germany

**Experiment:**
Marangoni Convection in an Open Boat-Free Liquid Surface Tension-Driven Flow Thermally Induced in Microgravity (PK-MKB 00)

This STS Spacelab D1 experiment was the sixth in a series of investigations designed by Schwabe et al. to examine oscillatory, thermocapillary Marangoni convection (see Schwabe, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 8, and STS-007).

The D1 experiment was different from all of Schwabe's earlier efforts in that it employed an open cavity (often referred to as an open boat) instead of a floating zone system. An open cavity has one free surface along the top of a container (or cuvette). When the walls of the cavity are differentially heated (thus creating a thermal gradient along the free surface) thermocapillary convection occurs at the surface. "The main advantage of... [the open cavity] configuration compared to a float zone is the visibility of the region near the free surface." (1, p. 92) The specific objective of the experiment was to study low-gravity thermocapillary-driven flow in the cavity.

The experiment hardware consisted (in part) of a cuvette fashioned from quartz glass (20 mm X 20 mm X 20 mm). Two aluminum heaters were placed at opposite ends of the cuvette. Prior to launch, the small cavity between the heaters was filled with liquid paraffin tetracosane ($C_{24}H_{50}$). The liquid, which has a melting point of 50.9 $^{o}$C, was in a solid state during the STS launch and was heated to a liquid state during the low-gravity mission. Thus, the cavity did not have to be filled during the mission (which, reportedly, would have been a difficult task).

"All rims which were in contact with the fluid were made as sharp as possible and coated with NUFLON (trademark of Fluomicron), which is not wetted by the... [paraffin]. The NUFLON acted as antispreading barrier against capillary outflow." (4, p. 122) Further, a double-walled heatshield made from plates of quartz glass, surrounded the fluid sample and guaranteed a temperature gradient mainly parallel to the free surface.

A special visualization system, which could illuminate a central vertical section of the cuvette with a light band, made it possible to document the position of ceramic tracer particles in the fluid with a 16 mm motion picture camera.

During the mission, the paraffin was melted and then the temperatures of both heaters were held at 60 °C for approximately 1 hour. While the right heater remained at 60 °C, the temperature of the left heater was raised in 5 °C steps to 120 °C. Then, the temperature of the right heater was raised to 90 °C, stabilized, and then raised in 10 °C steps to 110 °C.

After each temperature increase, there was a "thermalisation" time of approximately 15 minutes. After this thermalisation, the camera was turned on for 45 seconds and the fluid motion was documented by the camera. "Thus, the experiment was programmed to observe the convection for... [a temperature difference of] 0 °C, 5 °C,... 55 °C, 60 °C with the right heater at 60 °C, and for... [a temperature difference of] 30 °C, 20 °C, and 10 °C with the left heater at 120 °C." (4, p. 123) The Principal Investigator reported the heat transfer was determined via thermocouples.

Post-flight analysis of the film indicated that the sample melted as expected. When both heaters were set at 60 °C, the fluid was clear, without bubbles, and the tracer particles were well illuminated. However, the tracers did not move "...towards the middle of the liquid surface which is the coldest spot when both heaters are at 60 °C. Some tracers... [stuck] to the liquid surface." (4, p. 123)

Further, during the time when the temperature difference between the two heaters was increased (from 5 °C to 55 °C), the expected motion of the tracers from hot to cold along the free surface was not observed. Strong motion in the expected direction occurred only for a temperature difference of 60 °C.

The suppression of the expected Marangoni convection was attributed to an unclean free surface: dirt present on the free surface acted like a solid layer, hindering the thermocapillary flow. At a temperature difference of 60 °C, the dirt layer ruptured, thermocapillary flow was realized "...and the flow compressed the dirt in front of the cold wall (and the dirt mixed with the bulk liquid.)" (4, p. 124)

"Such a stable solid skin on the free surface has never been observed in experiments on [the] ground. On [the] ground there is always some fluid motion due to buoyancy which hinders the formation of a 'solid' and complete skin of dirt." (4, p. 123) In contrast, in the low-gravity environment, the dirt was allowed to diffuse from the bulk liquid to the surface during the hour that both heaters were at 60 $^{o}$C.

Photographs of the low-gravity fluid motion (for a temperature difference of 60 $^{o}$C) further illustrated that the free surface was bent, and that the cuvette was no longer filled to the rim. The anti-wetting barrier failed at the hot block and some of the fluid leaked out at that location. One large convection cell, which occupied almost the whole volume, moved from hot to cold at the free surface driving the flow in the bulk fluid from cold to hot. This was the motion expected for thermocapillary convection (in the absence of strong gravity-driven forces). The convection cell was asymmetric because of the dirt skin compressed at the colder heating block ("...the flow dives down before reaching the cold wall." (4, p. 124))

A 1-g reference experiment coupled with an analytical simulation of the flow fields (which included the bending of the free surface and a dirt-skin in front of the cold heating block) illustrated that "...the flow under normal gravity is much more complex than the flow under microgravity. The fluid volume is occupied by more than one convection roll. In the upper part we have two vortex centres near the free surface.... Both vortex centres are nearer to the free surface than the one under microgravity because it is hot fluid circulating near the free surface which cannot dive down under normal gravity. The fluid circulation near the free surface is mainly driven by surface tension forces. The flow very near the hot wall and very near the cold wall circulating along the bottom of the cuvette is due to buoyancy forces." (4, p. 124)

An analysis of the nondimensional numbers governing the fluid system indicated that thermocapillary forces dominate in the low-gravity experiment. This was confirmed by the low-gravity observations. Further, velocity profiles of an element at the free surface going from hot to cold indicated that "...under microgravity the maximum flow speed in the free surface reaches 20...[mm/s] whereas it is only 10...[mm/s] under normal gravity." (4, p. 126) Other interesting velocity profile results are presented in Reference (4).

**Key Words:** Fluid Physics, Melt and Solidification, Organic Systems, Transparent Liquids, Open Boat, Open Cavity, Free Surface, Surface Tension-Driven Convection, Marangoni Convection, Marangoni Convection Diminished, Thermocapillary Convection, Heat and Mass Transfer, Thermal Equilibrium, Thermal Gradient, Interfacial Tension, Coated Surfaces, Thin Films, Contamination Source, Diffusion, Meniscus Shape, Interfacial Curvature, Wetting of Container, Non-Wetting of Container, Contact Angle, Tracer Particles, Liquid/Gas Interface, Solid/Liquid Interface, Illumination System, Liquid Leakage, Processing Difficulties

**Number of Samples** one
**Sample Materials:** paraffin tetracosane $C_{24}H_{50}$ (Prandtl number: Pr = 50)
(C*H*)
**Container Materials:** quartz glass plates and aluminum walls
(Al*, Si*O*)

**Experiment/Material Applications:**
This experiment was designed to illustrate the significance of thermocapillary convection in crystal growth melts. "The main advantage of the... [open boat] configuration compared to a float zone is the visibility of the region near the free surface." (1, p. 92)

**References/Applicable Publications:**
(1) Schwabe, D., Lamprecht, R., and Scharmann, A.: Marangoni Convection in an Open Boat. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, p. 92. (abstract only; post-flight)

(2) Schwabe, D. and Lamprecht, R.: Marangoni-Konvection im offenen Boot (MKB). Naturwissenschaften, 73.Jahrgang Heft 7, July 1986, pp. 350-351. (abstract in English, text in German; post-flight)

(3) Schwabe, D., Lamprecht, R., and Scharmann, A.: Marangoni Convection in an Open Boat. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 55-56. (preflight)

(4) Schwabe, D., Lamprecht, R., and Scharmann, A.: Marangoni Convection In an Open Boat. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission, D1, Norderney, Germany, August 27-29, 1986, pp. 121-126. (post-flight)

(5) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-56. (post-flight; acceleration measurements on D1)

(6) Schwabe, D., Lamprecht, R., and Scharmann, A.: Experiments on Steady or Oscillatory Thermocapillary Convection in Space with Application to Crystal Growth. In Physicochemical Hydrodynamics-Interfacial Phenomenon, Ed. M. J. Verlade, NATO ASi Series B: Physics, Vol. 174, pp. 291-310, Plenum Press, New York, 1988.

(7) Schwabe, D. and Scharmann, A.: Marangoni and Buoyant Convection in an Open Cavity Under Reduced and Under Normal Gravity. Adv. Space Res., Vol. 8, No. 12, pp. 175-185, 1988.

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(9) Schwabe, D.: Surface Tension Driven Flow in Crystal Growth Melts. In Crystal Growth Properties and Applications, Vol. 11, pp. 75-112, Springer, Berlin. <The date this document was published is unclear at this time.>

(10) Input received from Principal Investigator D. Schwabe, August 1989 and August 1993.

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**Experiment Origin:** USA
**Mission:** SPAR 8
**Launch Date/Expt Date:** November 1980
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Experiment 77-13 Flight Apparatus
**Builder of Processing Facility:** Westinghouse Research and Development Center, Pittsburgh, Pennsylvania

**Experiment:**
<u>Glass Fining Experiment in Low Gravity (77-13)</u>

This SPAR 8 experiment was designed to study the low-gravity behavior of bubbles in a molten glass. The specific objectives of the study were to (1) characterize the thermally induced bubble motion and (2) obtain quantitative data to aid in the verification of theoretical models.

The experiment apparatus consisted of (1) a sample plate with a channel which held the glass material, (2) a tapered platinum-rhodium heater strip located underneath the sample plate, (3) upper and lower heat shields, and (4) a photographic system to record the movement of bubbles within the glass sample. The heater strip was tapered toward the center of the sample material, creating a hot spot at this location. Reportedly, "The [temperature] gradient was oriented along the axis of the [heater] strip pointing horizontally toward the narrow spot where the hot spot occurred and vertically into the strip (because a large vertical gradient also existed)." (1, p. V-18)

The apparatus was designed to (1) hold the sample plate/heater strip in alignment with the camera lens, (2) allow power to be applied to the heater strip to establish a thermal gradient in the glass sample, and (3) act as a sink for the heat generated by the heater strip. (A complete apparatus description is located in Reference (1).)

Prior to the rocket flight, the experiment sample (sodium borate glass with 29.9% $Na_2O$) was melted and resolidified in the silica cell. Because of the different thermal expansion coefficients of the silica cell and glass sample, microcracks were created in both materials. The microcracks in the glass sample acted as bubble generators for the experiment.

It was reported that during the SPAR flight, the experiment apparatus operated flawlessly. Two hundred and forty-six photographs (one per second) of the molten glass were taken to characterize the bubble movement. Time-temperature data were obtained, via telemetry, from the three thermocouples (two within the sample and one welded to the Pt-Rh heater strip).

Post-flight examination of the re-solidified sample indicated a large amount of crazing (cracking), which was attributed to expansion mismatch between the silica and glass sample. A large void around one thermocouple well at the center of the channel had formed during the flight.

The photographed bubbles had formed at either the top or bottom (heater strip side) of the channel. Analysis of the time-temperature data indicated that "...temperatures approached steady state asymptotically. The farther a thermocouple was from the heating area the longer it took to come to equilibrium.... It is clear that any bubble motion studies made roughly during the first 120 seconds will have to consider the changing temperatures and temperature gradients." (1, p. V-41)

Initial analysis of the photographs illustrated the movement of the bubbles toward the hotter areas of the sample. The photographs were transferred to film to utilize the Vanguard Motion Analyzer (see Reference (1) for the analytical procedure). Analysis of the film indicated two distinguishable periods during the experiment. During the first period, melting, and hence clearing (disappearance of microcracks) of the sample occurred and many bubbles moved toward the hot spot. During the second period, only one or two bubbles moved while the rest were stationary. The first period of bubble motion was attributed to thermocapillary motion and mechanical re-equilibration, although the extent of each effect was unknown. "Many of the bubbles initially formed in the glass as it melts are rapidly swept out of the glass by the high temperature gradients that are present while the sample is heating up. Since there is also a strong vertical gradient these bubbles not only migrate along the channel but vertically to the platinum heater strip. Once they contact the strip they become attached to it, and move only slowly at best." (1, p. V-45)

"As the experiment progresses [during the second period] bubbles that were in the cooler areas and/or at the top of the channel are slowly drawn toward the center of the channel. Bubbles touching the top of the channel may tend to stick there." (1, p. V-46)

Preliminary comparison with a Clarkson University model of thermocapillary motion (see Reference (8)) was performed. Reportedly, the observed motions of the bubbles appeared to be consistent with theory. However, only semi-quantitative agreement had been achieved. It was noted that further analysis was required.

No other information concerning this experiment could be located at this time. <Note: Several of the publications listed below were not available to aid in the preparation of this experiment summary.>

**Key Words:** Fluid Physics, Glasses, Glass Melts, Melt and Solidification, Binary Systems, Multiphase Media, Cracks, Bubbles, Bubble Migration, Marangoni Movement (Migration) of Bubbles, Bubble Formation, Bubble Coalescence, Bubble Transport, Thermocapillary Flow, Thermal Equilibrium, Thermal Fining, Thermal Gradient, Surface Tension, Liquid/Gas Interface, Solid/Liquid Interface, Volume Expansion, Buoyancy Effects Diminished, Sample Detachment from Crucible

**Number of Samples:** one
**Sample Materials:** sodium borate glass (29.9% $Na_2O$)
(Na*B*Na*O*)
**Container Materials:** Cell material: fused silica with a platinum-rhodium heater strip
(Si*O*)

**Experiment/Material Applications:**
During the production of glasses, bubbles are always present. On Earth, these bubbles are removed by buoyant (gravity dependent) or chemical (dopant introduction) fining procedures. However, during reduced-gravity containerless production of ultra-pure or other special glass systems, these procedures are not possible. Thermal fining, by the application of a thermal gradient, represents a viable procedure since neither intrusion in the melt nor gravity is required to perform the procedure.

**References/Applicable Publications:**

(1) Wilcox, W. R., Subramanian, R. S., Meyyappan, M., Smith, H. D., Mattox, D. M., and Partlow, D. P.: A Preliminary Analysis of the Data from Experiment 77-13 and Final Report on Glass Fining Experiment in Low Gravity. In Space Processing Applications Rocket Project, SPAR VIII Final Report, NASA TM-82578, pp. V-1 - V-55, June 1984. (post-flight)

(2) Smith, H. D., Mattox, W. R., Subramanian, R. S., and Meyyappan, M.: Experimental Observation of the Thermocapillary Driven Motion of Bubbles in a Molten Glass Under Low Gravity Conditions. In Materials Processing in a Reduced Gravity Environment of Space, Proceedings of the Annual Meeting, Boston, Massachusetts, November 16-18, 1982, New York and Amsterdam, North Holland, G. Rindone, editor, pp. 279-288. (post-flight)

(3) Mattox, D. M., Smith, H. D., Wilcox, W. R., and Subramanian, R. S.: Thermal Gradient Induced Migration of Bubbles in Glass. J. Am. Ceram. Soc., 65, pp. 437-442, (1982). (post-flight)

(4) Meyyappan, M., Subramanian, R. S., Wilcox, W. R., and Smith, H.: Bubble Behavior in Molten Glass in a Temperature Gradient. In Materials Processing in the Reduced Gravity Environment of Space, G. Rindone, ed., pp. 311-314, (1982). (post-flight)

(5) Meyyappan, M., Subramanian, R. S., Wilcox, W. R., and Smith, H.: Bubble Behavior in Molten Glass in a Temperature Gradient. Advances in Ceramics, Vol. V, Materials Processing in Space, American Ceramic Society, pp. 128-131, (1983). (post-flight)

(6) Meyyappan, M., Wilcox, W. R., and Subramanian, R. S.: The Migration of Gas Bubbles in a Thermal Gradient - Interaction Effects. In Proc. 2nd Int. Congress on Chemical Engineering V, pp. 218, (1981) (Canadian Soc. Ch.E., Ottawa, Publishers).

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(8) Meyyappan, M., Wilcox, W. R., and Subramanian, R. S.: The Slow Axisymmetric Motion of Two Bubbles in a Thermal Gradient. J. Colloid Interface Science, 94, pp. 243-257, (1983).

(9) Mattox, D. M., Smith, H. D., Wilcox, W. R., Subramanian, R. S.: Thermal-Gradient-Induced Migration of Bubbles in Molten Glass. Journal of the American Ceramic Society, Vol. 65, No. 9, pp. 437-442 (1982). (ground-based studies)

(10) Input received from Principal Investigator W. R. Wilcox, December 1987, September 1988, August 1989 and August 1993.

(11) Meyyappan, M., Wilcox, W. R., and Subramanian, R. S.: Thermocapillary Migration of a Bubble Normal to a Plane Surface. J. Colloid Interface Sci., 83, p. 199 (1981).

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**Experiment Origin:** USA
**Mission:** STS Launch #4, STS-004 (STS OFT-4, Columbia)
**Launch Date/Expt. Date:** June 1982
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment
NASA Get Away Special (GAS) Canister G-001
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-001:** Utah State University, Logan, Utah/R. Gilbert Moore
**Processing Facility:** Solder-alloy samples processed in a heater assembly
**Builder of Processing Facility:** J. K. Elwell (Principal Investigator)

**Experiment:**
Surface Tension Experiment - Shape of Liquid Meniscus (Experiment Number 10-P)

This experiment was one of ten investigations housed within the G-001 Get Away Special Canister during STS-004. Four other experiments in the canister were applicable to this data base (see Alford, STS-004 (Chapter 18); Dalley, STS-004 (Chapter 5); Laher, STS-004 (Chapter 17); Thomas, T. L., STS-004 Chapter 14)).

The specific objective of this investigation was to observe the shape of a liquid meniscus in the low-gravity environment.

During the mission, nine solder-alloy samples were to be melted and resolidified. It was expected that the molten solder would assume a "meniscus (lens) shape" and that this configuration would be preserved by the solidification process.

Reportedly, experimental goals could not be completed as planned. Data recorder failure, coupled with experiment fuse blowout, disrupted expected results. <Note: It appears that the fuse blowout prevented activation of the experiment during the mission.>

No other information concerning this experiment could be located at this time.

**Key Words:** Fluid Physics, Melt and Solidification, Free Surface, Free Surface Solidification, Surface Tension, Meniscus Shape, Binary Systems, Liquid/Gas Interface, Lens Formation, Hardware Malfunction, Sample Not Processed As Planned

**Number of Samples:** nine
**Sample Materials:** 50/50 solder alloy
**Container Materials:** aluminum
(Al*)

**Experiment/Material Applications:**
None of the available references detailed the specific applications of the research. However, it is speculated that the experimental objectives may be related to the production of sophisticated lenses and mirrors. If the space-created liquid meniscus did assume the shape of a parabola, experimentation employing appropriate materials might result in the production of superior optical devices.

**References/Applicable Publications:**

(1) Yoel, D., Walker, S., Elwell, J., and Moore, G.: The First Getaway Special-How it was Done. Spaceworld, May 1983, pp. 9-16. (post-flight)

(2) STS-4 Fourth Space Shuttle Mission, Press Kit, June 1982, p. 61. (preflight)

(3) Yoel, D. W.: Payload Integration of a Get Away Special Canister. American Institute of Aeronautics and Astronautics, Annual Meeting and Technical Display on Frontiers of Achievement, Long Beach, California, May 12-14, 1981, 5 pp. (preflight)

(4) The STS Getaway Special. NASA Report PB82-10223, May 20, 1982. (preflight)

(5) Cargo Systems Manual: GAS STS-4. May 20, 1982, JSC-17645, pp. 4-1 - 4-4. (preflight; very short description)

(6) Overbye, D.: The Getaway Kids Shuttle Into History. Discover, September 1982. (post-flight)

(7) Yoel, D. W.: Analysis of the First Getaway Special Space Shuttle Payload, Thesis for M.S. in Physics, Utah State University, Logan, Utah, 1984. (post-flight)

(8) Moore, R. G.: Educational Implications of Getaway Special Payload Number One. 32nd International Astronautical Federation Congress, Rome, September 6, 1981, IAF-81-293. (preflight)

(9) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(10) Input received from Principal Investigator J. K. Elwell, August 1989.

(11) Transcripts of press conference at NASA MSFC with G-001 Student Experimenters and Sponsors, NASA, May 20, 1982.

(12) Get Away Special. NASA News, NASA MSFC, June 7, 1982.

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**Experiment Origin:** England
**Mission:** STS Launch #5, STS-005 (STS 31-A, Columbia)
**Launch Date/Expt. Date:** November 1982
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Unknown, possibly a middeck experiment
**Processing Facility:** Unclear. It appears a liquid drop was deployed from a syringe and "tethered" to a solid Teflon™ surface (see **Experiment** summary below).
**Builder of Processing Facility:** Unknown

**Experiment:**
Interfacial Phenomena

This Space Shuttle STS-005 experiment was the first in a series of investigations designed by Haynes to study interfacial phenomena of fluids under low-gravity conditions.

Although discussions of the specific experimental objectives or setup of this investigation were not detailed in available publications, a description of the inflight procedure was presented.

During the STS-005 mission, interfacial phenomena were observed by tethering a liquid drop of di-n-octyl phthalate to a solid Teflon™ surface. Reportedly, the tethering was accomplished "...by use of canthotaxis (= 'anchoring at an edge'), the solid surface being provided with a sharply undercut edge at which the spreading tendency is arrested." (1, p. 260) <Note: This description of the tethering procedure (from Reference (1)) was not clear to the editors. It appears that the working fluid was injected onto the Teflon™ surface via a syringe (although this fact was not specifically stated).> It was reported that this technique worked well with the experimental materials.

Reference (1) (as listed below) discussed only the preliminary observations concerning (1) fluid handling under low-gravity conditions and (2) electrostatic effects. These observations included the following:

(1) Practically, the handling of fluids under low-gravity conditions is somewhat difficult. The working fluid used during the STS-005 mission tended to spread over all surfaces.

(2) Removal of air bubbles from fluids contained in syringes was a problem without the presence of buoyancy forces.

(3) Strong electrostatic forces were encountered during the experimental run. "...a tethered liquid drop of spherical form and about 25 $cm^3$ volume, when presented to a perspex [Teflon™] plate which had been inadvertently charged by stripping off a protective covering, was observed to leap across a distance of several centimeters, presumably as a result of electrostatic attraction." (1, p. 257) Air bubbles apparently gained a charge when they passed through a narrow feed-pipe into a tethered drop. The charged liquid drop then assumed a conical shape with a semi-angle of $25^{\circ}$-$30^{\circ}$. At the tip of the cone, a "pulsatile instability" was observed where small liquid drops were ejected with some regularity.

<Note: Much of the inflight experimental procedures (relayed above in item (3) and written based on Reference (1)), were unclear to the editors.>

No other discussion of the results could be located.

**Key Words:** Fluid Physics, Drops, Drop Formation, Solid/Liquid Interface, Interface Physics, Interface Phenomena, Fluid Management, Liquid Injection, Liquid Spreading, Wetting Kinetics, Surface Tension, Capillary Forces, Contact Angle, Bubbles, Electrostatic Effects, Surface Charge, Liquid Stability, Droplet Creation Via a Syringe, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Bubble Formation, Bubble Removal, Buoyancy Effects Diminished, Liquid/Gas Interface, Free Surface

**Number of Samples:** unknown
**Sample Materials:** Test fluid: di-n-octyl phthalate
**Container Materials:** Teflon™

**Experiment/Material Applications:**
Although no specific research applications were detailed in the available publications, such investigations are important to experiments involving liquid-bridge creation, capillary flow, etc.

**References/Applicable Publications:**

(1) Haynes, J. M.: Interfacial Phenomena and Their Relevance to Microgravity Materials Processing. In Symposium on Industrial Activity in Space, Stressa, Italy, May 2-4, 1984, Proceedings Paris, Eurospace, 1984, pp. 254-261.

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurments on the shuttle)

(3) Martinez, I., Haynes, J. M., and Langbein, D.: Fluid Statics and Capillarity. In Fluids and Material Sciences in Space, Edited by H. U. Walter, Springer-Verlag, 1987, pp. 53-80. (related topics)

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**Experiment Origin:** England
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Material Science Double Rack (MSDR)
**Processing Facility:** Fluid Physics Module (FPM)
**Builder of Processing Facility:** FIAT C. R., Italy

**Experiment:**
Kinetics of Spreading of Liquids on Solids (1ES327)

This Spacelab 1 experiment was the second in a series investigations designed by Haynes to study interfacial phenomena of fluids under low-gravity conditions (see Haynes, STS-005). The specific objective of the experiment was to investigate the dynamic behavior of a tethered liquid drop after contact with a plane solid surface.

The Spacelab-1 Fluid Physics Module (FPM) was used to conduct the experiment. During the planned experimental sequence, two different fluids were to be deployed onto both clean, and prewetted solid steel surfaces. However, the FPM "...suffered catastrophic failure early in the mission because of the inability of the chemical 'anti-spread barrier' to inhibit [excessive] spreading of the silicone oil used as working fluid: tethered drops of the required size simply could not be grown on the feed plate, prior to spreading." (1, p. 44) (A discussion of these failures is given in Reference (2).) Although improvements were made to the FPM, it was still impossible to grow drops of the intended size.

Despite the above mentioned drop-formation difficulties, some liquid spreading experiments were performed. On-orbit changes to the FPM facility were necessary to complete the experiments. A perspex disk (51.5 mm dia.) was attached to the standard FPM feed plate through which a volume (17.25 $cm^3$) of 5 cS silicone oil was injected. The fluid formed a drop of approximately 14.9 mm spherical cap height. A curved fillet of liquid, approximately 2-3 $cm^3$, formed behind the undercut edge of the disk. The drop was slowly contacted with a clean, dry duralumin disc of 10 cm diameter. The subsequent spreading process was recorded with a video camera.

Analysis of the recorded film indicated that initially the silicone oil spread rapidly. After 0.7 seconds, the fluid motion decayed to a slower spreading. Drop volumes and interfacial areas were determined for the first 2.02 seconds of the experiment (see Reference (1) for data). It was reported "...that the area of the liquid/vapour interface increases continuously after spreading. The spreading process therefore does work on the liquid/vapour interface, this being supplied by the spontaneous conversion of solid/vapour to solid/liquid interface at the spreading line. Indeed, in the early stages of spreading the area of the solid/liquid interface grows faster than that of the liquid/vapour interface, and the difference [area of liquid/vapour interface minus area of solid/liquid interface] thus decreases at first, although it reaches a minimum before the end of the rapid process and increases thereafter." (1, p. 45)

It was also reported that before contact with the duralumin plate, the drop exhibited a non-spherical shape. The central axis of the drop was inclined to the normal of the two plates. These results were attributed to electrostatic effects which were also observed during the STS-005 experiment (see Haynes, STS-005).

<Note: It was indicated in Reference (1) that three or four spreading sequences were recorded. However, results on only one of the sequences were located in the applicable publications.>

**Key Words:** Fluid Physics, Drops, Drop Formation, Sphericity, Particle Growth, Solid/Liquid Interface, Liquid/Gas Interface, Liquid/Vapor Interface, Interface Physics, Interface Phenomena, Fluid Management, Meniscus Shape, Liquid Dynamic Response, Liquid Spreading, Wetting Kinetics, Surface Tension, Capillary Forces, Contact Angle, Electrostatic Effects, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Coated Surfaces, Free Surface, Processing Difficulties

**Number of Samples:** unknown
**Sample Materials:** Working fluid: 5 cS silicone oil; surface material: steel
**Container Materials:** not applicable

**Experiment/Material Applications:**
See Haynes, STS-005.

**References/Applicable Publications:**

(1) Haynes, J. M.: Kinetics of Spreading Liquids in Microgravity Experiment 1 ES 327. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 43-46. (post-flight)

(2) Haynes, J. M.: Assessment of SL-1 Results - 1 ES 327: Spreading Kinetics and 1 ES 339 Capillary Hysteresis. Earth Oriented Applications of Space Technology, Vol. 5, No. 1-2, 1985, pp. 67-68. (post-flight)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements on Spacelab 1))

(4) Gonfalone, A.: The Fluid Physics Module-A Technical Description. In ESA 5th European Sympsoium on Materials Sciences Under Microgravity, Results of Spacelab 1, Scloss Elmau, November 5-7, 1984, ESA SP-222, pp. 3-7. (apparatus description)

(5) Haynes, J. M.: Interfacial Phenomena and Their Relevance to Microgravity Materials Processing. In Proceedings of the Symposium on Industrial Activity in Space, Stressa, Italy, May 2-4, 1984, pp. 254-261. (post-flight)

(6) Ceronetti, G.: The Fluid Physics Module Design. XXV Rassegna Internazionale Elettronica Nucleare Ed Aerospaziale, Roma, March 10-19, 1978, pp. 76-83. (prior to Spacelab 1)

(7) Martinez, I., Haynes, J. M., and Langbein, D.: Fluid Physics and Capillarity. In Fluid Sciences and Materials Sciences in Space, Edited by H. U. Walter, Springer-Verlag, 1987, pp. 53-80. (related topics)

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**Experiment Origin:** England
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Material Science Double Rack (MSDR)
**Processing Facility:** Fluid Physics Module (FPM)
**Builder of Processing Facility:** FIAT C. R., Italy

**Experiment:**
Interfacial Instability and Capillary Hysteresis (1ES339)

This Spacelab 1 experiment was the third in a series of investigations designed by Haynes to study interfacial phenomena of fluids under low-gravity conditions (see Haynes, STS-005; Haynes, Spacelab 1, Experiment #1ES 327). Reportedly, the specific objective of the experiment was "...to investigate the stability bounds of alternative capillary configurations of liquid supported in an array of three equal solid spheres, and the hysteresis in the relationship between liquid volume and interfacial curvature which would be caused by irreversible transitions between the accessible configurations at the limits of their stability." (1, p. 67) <Note: additional information describing how this complex objective was to be achieved could not be located at this time. Further, details (or pictures) of the experimental setup were not presented in the available documents.>

The Spacelab-1 Fluid Physics Module was to be used for the experiment. However, the experiment "...could not be operated as planned... because the screw thread on the replacement syringe failed to engage during in-flight assembly of the hardware in the FPM." (1, p. 67) <Note: no further information about the role of the "replacement syringe" could be located at this time.> Instead, another experimental procedure was improvised: the Payload Specialist manually operated the associated equipment outside of the FPM and recorded the results with an on-board cine camera. <Note: further details of the planned and actual experimental procedures could not be located in the available publications.>

At this time, no publications could be located which discussed the results from this experiment. Reference (1) states that the cine film had not been received and thus analysis had not been performed.

**Key Words:** Fluid Physics, Capillary Forces, Solid/Liquid Interface, Surface Tension, Free Surface, Wetting Kinetics, Liquid Spreading, Contact Angle, Spheres, Interface Physics, Interfacial Curvature, Interface Phenomena, Interface Stability, Hardware Malfunction

**Number of Samples:** unknown
**Sample Materials:** unknown
**Container Materials:** unknown

**Experiment/Material Applications:**
See Haynes, STS-005.

**References/Applicable Publications:**

(1) Haynes, J. M.: Assessment of SL 1 Results: 1ES 327: Spreading Kinetics and 1 ES 339: Capillary Hysteresis. Earth Oriented Applications of Space Technology, Vol. 5, No. 1/2, 1985, pp. 67-68. (post-flight)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986 Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements on Spacelab 1)

(3) Gonfalone, A.: The Fluid Physics Module-A Technical Description. In ESA 5th European Symposium on Materials Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 3-7. (apparatus description)

(4) Ceronetti, G.: The Fluid Physics Module Design. XXV Rassegna Internazionale Elettroica Nucleare Ed Aerospaziale, Roma, March 10-19, 1978, pp. 76-83. (prior to Spacelab 1)

(5) Martinez, I., Haynes, J. M., and Langbein, D.: Fluid Statics and Capillarity. In Fluid Sciences and Materials Science in Space, Edited by H. U. Walter, Springer-Verlag, 1987, pp. 53-80. (related topics)

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**Experiment Origin:** USA
**Mission:** STS Launch #5, STS-005 (STS 31-A, Columbia)
**Launch Date/Expt. Date:** November 1982
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** High School Student Experiment, Shuttle Student Involvement Program (SSIP), Middeck Experiment
**Processing Facility:** Seven heater pans were employed. A thermofoil heater was placed under the pans.
**Builder of Processing Facility:** Thiokol Corporation, Brigham City, Utah. Electronic control and data handling designed by James Elwell, Utah State University, Logan, Utah

**Experiment:**
<u>Surface Tension Convection (SE81-9)</u>

This experiment was the first in a series of investigations designed by S. Thomas et al. to study surface-tension-driven convection in the low-gravity environment. The specific objective of the experiment was to investigate the effects of boundary-layer conditions and geometry on the onset and development of Marangoni convection.

During the mission, Krytox oil was injected (via syringes) into a number of 7 cm and 10 cm diameter aluminum-bottomed pans. During most of these injections, the liquid did not spread over the bottom of the pans creating the desired uniform layer of liquid; rather, the oil formed into large drops (many centimeters in diameter) and attached to the pan bottoms. (It was later concluded that lids, placed over the pans for safety reasons, restricted proper injection of the liquid.) When the pans containing these drops were heated from below, radial surface-tension-driven convection, extending from the drop edge to the drop center, occurred. The flow was driven by the thermally induced, radial surface tension gradient along the drop's free surface. In the center of some drops, a single, large convection cell formed, presumably as a result of the Marangoni instability since the radial temperature gradients could not produce such a flow.

One pan was filled to nearly uniform depth. When heated from below, the convection in this pan formed as radially oriented rolls with a single cell in the center. The boundary between the cell and the rolls decreased in diameter with time until the cell disappeared, leaving only radial rolls. This was a result of the interplay between the temperature gradient along the free surface (driving the rolls) and the temperature gradient perpendicular to the surface (driving the cell).

Reportedly, the experiment was scheduled for reflight. <Note: It does not appear that this experiment has been reflown to date, although S. Thomas did have two related experiments on two other Shuttle flights (see Thomas, S. STS-011 and STS-017 (this chapter)).>

**Key Words:** Fluid Physics, Free Surface, Surface Tension, Drops, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Cellular Convection, Bénard Convection, Buoyancy Effects Diminished, Heat and Mass Transfer, Thermal Gradient, Liquid Spreading, Wetting Kinetics, Non-Wetting of Container, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Fluid Management, Droplet Creation Via a Syringe, Liquid/Gas Interface, Tracer Particles

**Number of Samples:** seven
**Sample Materials:** Krytox oil (containing aluminum flakes to allow flow visualization)
**Container Materials:** The heater pans had aluminum bottoms and RTV rubber sidewalls.

**Experiment/Material Applications:**
This experiment was designed to study thermally-induced surface tension convection. Such low-gravity investigations allow a detailed examination of the role of Marangoni forces without the overwhelming gravity-induced forces.

**References/Applicable Publications:**
(1) STS-5 Fifth Space Shuttle Mission, NASA Press Kit, October 1982, pp. 39-40. (preflight)

(2) Spinoff 1983; NASA Publication: Student Experiments, p. 25.

(3) Surface Tension Convection in Microgravity, Summary Report, Prepared by Johnson Engineering Corporation, Boulder, Colorado, 5 pp. (post-flight)

(4) Thiokol Sponsors Student Experiment Aboard Space Shuttle STS-5. Press Release, Thiokol, Wasatch Division, Utah 1982. (preflight)

(5) Input received from Principal Investigator S. Thomas, September 1989.

(6) Surface Tension Convection in Microgravity. In Shuttle Student Involvement Program (SSIP) Final Reports of Experiments Flown, NASA/JSC Internal Note, JSC 24005, October 20, 1989.

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**Experiment Origin:** USA
**Mission:** STS Launch #10, STS-011 (STS 41-B, Challenger)
**Launch Date/Expt. Date:** February 1984
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment
NASA Get Away Special (GAS) Canister G-004
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of this experiment within G-004:** Utah State University, Logan, Utah
**Processing Facility:** Two heated copper cylinders between which a column of paraffin was supended
**Builder of Processing Facility:** Principal Investigator S. Thomas and Utah State University, Logan Utah

**Experiment:**
Thermocapillary Convection (Float Zone)

This experiment was the second in a series of investigations designed by S. Thomas et al. to study surface-tension-driven convection in the low-gravity environment (see Thomas, S., STS-005).

The experiment was one of six investigations housed within Get Away Special Canister G-004 on STS-011. Three other experiments (of the six) were applicable to this data base (see Kitaura, STS-011 (Chapter 2), and Gadsden, STS-011 (two experiments, Chapters 15 and 18)). The objective of this experiment was to examine thermocapillary flow in an experimental setup analogous to a float zone.

During the STS mission, a solid cylinder of paraffin wax, supported by two copper cylinders, was heated differentially from its ends. Surface tension driven flow could be observed by following the motion of aluminum powder suspended within the molten wax. Unexpectedly, a teflon cup, which was employed to partially enclose the paraffin surface for launch, failed to retract on orbit. Reportedly, thermocapillary flow was observed, but no useful data were obtained.

**Key Words:** Fluid Physics, Melt and Solidification, Float Zones, Liquid Bridges, Free Surface, Surface Tension, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Thermal Gradient, Heat Transfer, Tracer Particles, Liquid/Gas Interface, Containerless Processing Applications, Hardware Malfunction, Processing Difficulties

**Number of Samples:** one
**Sample Materials:** paraffin (wax) with aluminum powder
**Container Materials:** sample supported by two copper cylinders (Cu*)

**Experiment/Material Applications:**
Thermocapillary flow and material phase change were studied in a float zone geometry. Such investigations have applications in the area of containerless processing of crystalline materials.

**References/Applicable Publications:**
(1) Cargo Systems Manual: GAS Annex for STS-11, JSC-17645 Annex STS-11, December 2, 1983. (preflight)

(2) Thomas, S.: Thermocapillary Flow and Phase Change in Microgravity: Results from GAS Payload G-004. In Goddard Space Flight Center's 1984 Get Away Special Experimenter's Post Symposium Proceedings (Addendum) Greenbelt, Maryland, 1984, pp. 73-75. (post-flight)

(3) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(4) Getaway Special (GAS) Payloads (STS-11). In Goddard Space Flight Center's Engineering Newsletter, Vol. 2, No. 3, April 1984, published by the Engineering Directorate. (p. 8, short description)

(5) Input received from Principal Investigator S. Thomas, September 1989.

(6) NASA Press Kit, STS-12, p. 23. (preflight)

(7) STS-11 Get Away Special Payload Descriptions. NASA News, NASA/GSFC, 1984.

(8) 1984 Student GAS Program, internal documentation, Utah State University, Logan, Utah.

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**Experiment Origin:** USA
**Mission:** STS Launch #13, STS-017 (STS 41-G, Challenger)
**Launch Date/Expt. Date:** October 1984
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment
NASA Get Away Special (GAS) Canister G-518
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-518:** Utah State University, Logan, Utah
**Processing Facility:** Two heated copper cylinders between which a column of paraffin was suspended (Extensive modifications were made to supporting hardware post STS 41-B (G-004).)
**Builder of Processing Facility:** Principal Investigator S. Thomas and Utah State University, Logan, Utah

**Experiment:**
Thermocapillary Convection (Float Zone)

This experiment was the third in a series of investigations designed by S. Thomas et al. to study surface-tension-driven convection in the low-gravity environment (see Thomas, S., STS-005 and STS-011).

The experiment was one of four investigations housed within the G-518 Get Away Special canister during STS-017. The three other experiments (of the four) were applicable to this data base (see Kitaura, STS-017 (Chapter 2); Walden, STS-017 (Chapter 18); "Solder Flux Separation," STS-017 (Chapter 18, Principal Investigator Unknown)).

During the mission, a solid cylinder of paraffin wax, supported by two copper cylinders, was heated at one end. (The column was suspended in a nitrogen gas environment.) The paraffin melted and thermocapillary flow resulting in the liquid bridge was observed by following the motion of aluminum powder suspended within the molten wax.

Reportedly, "After one-half hour of warmup, two runs were conducted, separated by an hour of cooldown. Early in the runs, when Marangoni numbers were lower, steady laminar thermocapillary flow was established. At Marangoni numbers above 10000, transition to oscillatory flow was expected to begin. This periodic flow was established at a Marangoni number of about 30000 with a period of about 8 sec....(It was fortunate that two runs were made as a camera failure precluded getting any visual data on the first run.)" (6, p. 9) The flow was highly asymmetric along the cylindrical axis, presumably because of asymmetric radial heat loss.

A flow in the nitrogen gas outside the liquid was also observed. When a nylon string (which held a thermistor in place) was accidentally melted and produced smoke, "This smoke serundipitiously acted as trace particles for convection which unexpectedly occurred in the gas to one side of the liquid column." (1, p. 299) This gaseous convection was driven by a strong boundary layer in the liquid just adjacent to the heater. The periodic oscillation in the paraffin thermocapillary flow occurred some time after heating began and after the gaseous convection was well established. A pulse occurred in the gaseous flow coincident with the liquid oscillation, implying the two were strongly coupled. A small deflection in the liquid surface occurred at each pulse and was convected down nearly the entire length of the bridge. It was found that significant convection occurs in the gas bounding a float zone which is undergoing thermocapillary flow. The gaseous convection can significantly affect heat loss and is definitely coupled to the oscillations and instabilities in the liquid.

Unrelated to the flows described above, near the end of the experiment, the liquid bridge underwent a bulk vibration, presumably due to a thruster firing or spacecraft keeping maneuver.

**Key Words:** Fluid Physics, Melt and Solidification, Float Zones, Free Surface, Surface Tension, Transparent Liquids, Liquid Bridges, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Oscillatory Marangoni Convection, Time Dependent Thermocapillary Flow, Steady to Oscillatory Convective Transition, Critical Marangoni Number, Thermal Gradient, Asymmetric Temperature Field, Liquid/Gas Interface, Gaseous Convection, Fluid Stability, Liquid Bridge Stability, Float Zone Stability, Acceleration Effects, Tracer Particles, Containerless Processing Applications, Photographic Difficulties

**Number of Samples:** Two runs were performed using one float zone setup.
**Sample Materials:** paraffin (wax) containing aluminum powder for flow visualization
**Container Materials:** Sample supported by two copper cylinders (Cu*)

**Experiment/Material Applications:**
Thermocapillary flow and gaseous convection were studied in a float zone geometry. Such studies and subsequent findings are of importance to containerless processing of crystalline materials.

**References/Applicable Publications:**

(1) Thomas, S.: Thermocapillary Flow and Gaseous Convection in Microgravity: Results from GAS Payload G-0518. In 1985 Getaway Special Experimenter's Symposium, NASA CP-2401, pp. 293-301. (post-flight Results)

(2) Cargo Systems Manual: GAS Annex for STS 41-G. JSC-17645 41-G, September 4, 1984. (short description; preflight)

(3) Thomas, S.: Thermocapillary Flow and Gaseous Convection in Microgravity. Center for Atmospheric and Space Science Report, Utah State University, Logan, Utah, 1986. (post-flight analysis)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(5) Input received from Principal Investigator S. Thomas, September 1989.

(6) Hill, M. E. and O'Malley, T. F.: A Summary of Existing and Planned Experiment Hardware for Low-Gravity Fluids Research. NASA TM-103706; also, AIAA-91-0777, AIAA 29th Aerospace Sciences Meeting, January 7-10, 1991, Reno, Nevada, 44 pp., specifically p. 9. (post-flight)

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**Co-Investigator(s):** Unknown
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**Experiment Origin:** Belgium
**Mission:** TEXUS 8
**Launch Date/Expt. Date:** May 1983
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-6: experiment cell equipped with electric heaters and viewing ports (a slide door/piston system was used to initiate liquid into the experiment cell).
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Effects of Surface Tension Minimum on Thermocapillary Convection

Surface-tension-driven convection (also known as Marangoni convection) results because fluid elements on the liquid free surface with a low surface tension tend to flow toward fluid areas of higher surface tension. The surface tension of most fluids decreases as the fluid temperature increases. However, some fluids, such as aqueous alcohol solutions, have a surface tension minimum when plotted with temperature: surface tension decreases with increasing temperature down to a minimum temperature (and minimum surface tension) and then begins to experience an increase in surface tension with temperature.

On Earth, both surface-tension-driven convection and buoyancy-driven convection act on the fluid system. In a reduced-gravity environment, the Marangoni convective contributions should be more easily discerned. "Thus, when the motor of the convection is the thermocapillary effect alone, it is expected that at temperatures around the minimum, the convection will slow down and that the onset of oscillatory modes... will appear for higher thermal constraints." (1, p. 301) An imposed temperature difference, (delta T) = $T_1 - T_2$, parallel to a liquid-gas interface, will cause flows in the adjoining phases. It had been observed that, if $T_1$ and $T_2$ are lower than the $T_{min}$ then surface flow is from the hot side to the cold side. The flow is opposite if $T_1$ and $T_2$ are above $T_{min}$.

This TEXUS 8 experiment was the first in a series of investigations designed by Legros et al. to study the effect of a surface tension minimum on thermocapillary convection. The specific objective of the experiment was to observe fluid motion under an

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**Experiment Origin:** Belgium
**Mission:** TEXUS 8
**Launch Date/Expt. Date:** May 1983
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-6: experiment cell equipped with electric heaters and viewing ports (a slide door/piston system was used to initiate liquid into the experiment cell).
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
<u>Effects of Surface Tension Minimum on Thermocapillary Convection</u>

Surface-tension-driven convection (also known as Marangoni convection) results because fluid elements on the liquid free surface with a low surface tension tend to flow toward fluid areas of higher surface tension. The surface tension of most fluids decreases as the fluid temperature increases. However, some fluids, such as aqueous alcohol solutions, have a surface tension minimum when plotted with temperature: surface tension decreases with increasing temperature down to a minimum temperature (and minimum surface tension) and then begins to experience an increase in surface tension with temperature.

On Earth, both surface-tension-driven convection and buoyancy-driven convection act on the fluid system. In a reduced-gravity environment, the Marangoni convective contributions should be more easily discerned. "Thus, when the motor of the convection is the thermocapillary effect alone, it is expected that at temperatures around the minimum, the convection will slow down and that the onset of oscillatory modes... will appear for higher thermal constraints." (1, p. 301) An imposed temperature difference, (delta T) = $T_1 - T_2$, parallel to a liquid-gas interface, will cause flows in the adjoining phases. It had been observed that, if $T_1$ and $T_2$ are lower than the $T_{min}$ then surface flow is from the hot side to the cold side. The flow is opposite if $T_1$ and $T_2$ are above $T_{min}$.

This TEXUS 8 experiment was the first in a series of investigations designed by Legros et al. to study the effect of a surface tension minimum on thermocapillary convection. The specific objective of the experiment was to observe fluid motion under an imposed temperature difference such that $T_1$ and $T_2$ are above $T_{min}$.

The experiment was performed in the TEXUS Experiment Module TEM 06-6. The module contained a rectangular stainless steel cell with two Pyrex™ observation windows. The cell was $3 \times 10^{-2}$ m long by $2 \times 10^{-2}$ m high by $10^{-2}$ m wide. A n-heptanol solution ($6.3 \times 10^{-3}$ molal, $T_{min} = 40$ °C) containing 90-micron diameter Dow latex particles was used as the test fluid.

During the low-gravity phase of the mission, the cell was partially filled by activating a slider between the empty cell and a fluid reservoir. A pneumatic piston slowly pushed the fluid into the cell. "Small... [Teflon™] pieces in the metallic walls and grooves coated with... [Teflon™] in the... [Pyrex™] windows succeeded in stopping the creeping of the fluid. After the filling, the depth of the liquid in the middle of the surface was $6.6 \times 10^{-3}$ m and the radius of curvature of the surface along the meridian plane was $2.95 \times 10^{-2}$ m." (1, p. 305) Opposite sides of the cell were differentially heated imposing a temperature gradient parallel to the liquid-gas interface. Both side temperatures were above the $T_{min}$ temperature of 40 °C (placing them in the range that the surface tension increases with the temperature of the aqueous solution). A 16-mm camera was used to film (1) the filling of the cell and (2) the resultant convective motion (25 frames/sec). <Note: See Reference (1) and/or Reference (2) for further experimental setup details.>

Post-flight examination of the thermal data indicated the temperature of the hot side was a constant 66 °C (plus or minus 0.1 °C) and that of the cold side was 46 °C (plus or minus 0.1 °C).

Reportedly, the initial concentration of the latex particles was not constant. However, this allowed a good visualization of the fluid stream lines because of elongation of the high concentration regions.

Reportedly, the experiment was a success, leading to the following conclusions:

(1) The Teflon™ pieces in the stainless steel walls and Teflon™ coated grooves in the Pyrex™ prevented the fluid from creeping up the walls and resulted in a rather flat gas-liquid interface.

(2) The observed fluid flow was from the cold to the hot side, as expected.

(3) The convective flows were initiated at the interface near the hot side and developed slowly. After about 6 minutes, a single flow cell occupied the entire volume.

(4) At any particular time the velocities at the interface were dependent upon position with respect to the thermal gradient. The velocities were higher nearest the hot wall and decreased at a rate of $-0.0136 \times 10^{-3}$ m/s$^2$ (velocity/time).

(5) The velocities near the interface were higher than those in the bulk liquid.

From these results (see Reference (1) for more details of the velocity fields), it was determined that (within the temperature range of the experiment) "...the surface tension is increasing with temperature, indirectly indicating that a surface tension minimum is present at a lower temperature, and thus is still existing under such non equilibrium convective conditions." (5, p. 135)

Reportedly, the amount of reduced-gravity time that the TEXUS rocket could produce was too short to achieve steady state conditions. Thus, the surface velocities were still increasing at the end of the low-gravity period.

**Key Words:** Fluid Physics, Open Cavity, Partially Filled Containers, Aqueous Solutions, Free Surface, Meniscus Shape, Surface Tension, Surface Tension Gradients, Heat Transfer, Surface Tension Minimum, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Oscillatory Marangoni Convection, Time Dependent Thermocapillary Flow, Cellular Convection, Buoyancy Effects Diminished, Thermal Gradient, Interfacial Curvature, Liquid/Gas Interface, Solid/Liquid Interface, Fluid Stability, Flow Velocity, Liquid Reservoir, Liquid Transfer, Coated Surfaces, Liquid Spreading, Wetting, Piston System, Tracer Particles

**Number of Samples:** one
**Sample Materials:** n-Heptanol aqueous solution ($6.3 \times 10^{-3}$ molal) with 90 micron diameter, Dow latex particles
**Container Materials:** stainless steel cell with Pyrex™ windows

**Experiment/Material Applications:**
Research applications of fluids exhibiting a surface tension minimum were not detailed in the available publications.

The specific reasons why n-heptanol was selected were not detailed in the available publications. It is believed that an n-heptanol aqueous solution was used because of its particular surface tension properties.

**References/Applicable Publications:**


(1) Limbourg-Fontaine, M. C., Pétré, G., and Legros, J. C.: Texus 8 Experiment: Effects of a Surface Tension Minimum on Thermocapillary Convection. PCH PhysicoChemical Hydrodynamics Vol. 6, Number 3, 1985, pp. 301-310. (post-flight)

(2) Legros, J. C., Limbourg-Fontaine, M. C., and Pétré, G.: Influence of Surface Tension Minimum as a Function of Temperature on the Marangoni Convection. Acta Astronautica, Vol. 11, Number 2, 1987, pp. 143-147. (preflight; experimental setup)

(3) Legros, J. C., Pétré, G., Limbourg-Fontaine, M. C., Villers, D., and Platten, J. K.: Thermocapillary Convection Around a Surface Tension Minimum: A Microgravity Experiment (TEXUS 8) and Numerical Simulation. 5th Elgra Meeting Proceedings in Elgra News, 6, 1984, pp. 41-49.

(4) Legros, J. C., Pétré, G., and Limbourg-Fontaine, M. C.: Study of Marangoni Convection Around a Surface Tension Minimum Under Microgravity Conditions. Advances in Space Research, Vol. 4, Number 5, pp. 37-41, 1984. (post-flight)

(5) Legros, J. C., Limbourg-Fontaine, M. C., and Pétré, G.: Surface Tension Induced Convection in Presence of a Surface Tension Minimum. In Scientific Results of the German Spacelab Mission D1, Norderney Symposium, Norderney, Germany, August 27-29, 1986, pp. 131-140. (post-flight)

(6) Influence of a Surface Tension Minimum on the Marangoni Effect. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February, 1991, pp. 60-61. (post-flight)


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**Experiment Origin:** Belgium
**Mission:** TEXUS 9
**Launch Date/Expt. Date:** May 1984
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-8: experiment cell equipped with fluid injection mechanism and electric heaters (same apparatus as used on TEXUS 8 but for TEXUS 9 (1) a bellows system replaced the piston assembly and (2) a differential interferometer was added)
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Effects of Surface Tension Minimum on Thermocapillary Convection

This TEXUS 9 experiment was the second in a series of investigations designed by Legros et al. to study the effect of a surface tension minimum on thermocapillary convection (see Legros, TEXUS 8). The earlier TEXUS 8 experiment in this investigative series subjected a free-surface cell to a temperature difference such that $T_1$ and $T_2$ were above the temperature at which the fluid has a surface tension minimum $T_{min}$. The specific objective of the TEXUS 9 experiment was to subject a similar fluid cell to a temperature difference such that $T_1$ was less than $T_{min}$ and $T_2$ was greater than $T_{min}$. This temperature difference allowed examination of the fluid flows at the surface tension minimum. <Note: See Legros, TEXUS 8 for a detailed discussion of surface tension minimum and imposed temperature differences.>

During the TEXUS 9 sounding rocket mission, the TEXUS Experiment Module TEM 06-8 was used. The apparatus and procedure were the same as those employed during the TEXUS 8 mission (see Legros, TEXUS 8), except for a few hardware improvements. These improvements included (1) a bellows filling device (rather than a piston) for smoother (partial) filling of the observation chamber and (2) a differential interferometer for analysis of the fluid temperature fields. The same (TEXUS 8) n-heptanol solution with 90-micron diameter Dow latex particles was employed as the test fluid. The imposed wall temperatures were 32 $^{o}C$ ($T_1$) and 50 $^{o}C$ ($T_2$).

Reportedly, the post-flight results included:

(1) The partial filling procedure resulted in a flat liquid-gas interface.

(2) Marangoni convection was initiated at the container wall and a convective cell grew.

(3) The fluid flow at the fluid free surface was from the cold side to the hot side in the hot two-thirds of the interface. The cold side remained nearly at rest.

(4) Fewer fringes were seen in the interferograms than observed in ground-based tests. The fringes recorded in the flight experiment were not oscillating.

(5) "The opening of the interface, i.e. motions from the 40°C interface region toward the two opposite lateral walls at 32°C and 50°C was not recorded, as expected from the theoretical arguments and from numerical simulations...." (5, p. 135)

Reportedly, the amount of reduced-gravity time that the TEXUS rocket could produce was too short to achieve steady state conditions. Thus, the surface velocities were still increasing at the end of the low-gravity period.

**Key Words:** Fluid Physics, Open Cavity, Partially Filled Containers, Aqueous Solutions, Free Surface, Meniscus Shape, Surface Tension, Surface Tension Gradients, Surface Tension Minimum, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Oscillatory Marangoni Convection, Cellular Convection, Thermal Gradient, Interfacial Curvature, Liquid/Gas Interface, Solid/Liquid Interface, Flow Velocity, Liquid Reservoir, Liquid Transfer, Liquid Injection, Tracer Particles, Interferometric Measurement

**Number of Samples:** one
**Sample Materials:** n-Heptanol aqueous solution ($6.3 \times 10^{-3}$ molal) with 90-micron diameter Dow latex particles
**Container Materials:** stainless steel cell with Pyrex™ windows

**Experiment/Material Applications:**
See Legros, TEXUS 8.

**References/Applicable Publications:**

(1) Limbourg-Fontaine, M. C., Pétré, G., and Legros, J. C.: Texus 8 Experiment: Effects of a Surface Tension Minimum on Thermocapillary Convection PCH. PhysicoChemical Hydrodynamics Vol. 6, Number 3, 1985, pp. 301-310. (post-flight)

(2) Legros, J. C., Limbourg-Fontaine, M. C., and Pétré, G.: Influence of Surface Tension Minimum as a Function of Temperature on the Marangoni Convection. Acta Astronautica, Vol. 11, Number 2, 1987, pp. 143-147. (preflight; experimental setup)

(3) Legros, J. C., Pétré, G., Limbourg-Fontaine, M. C., Villers, D., and Platten, J. K.: Thermocapillary Convection Around a Surface Tension Minimum: A Microgravity Experiment (TEXUS 8) and Numerical Simulation. 5th Elgra Meeting Proceedings in Elgra News, 6, 1984, pp. 41-49.

(4) Legros, J. C., Pétré, G., and Limbourg-Fontaine, M. C.: Study of Marangoni Convection Around a Surface Tension Minimum Under Microgravity Conditions. Advances in Space Research, Vol. 4, Number 5, pp. 37-41, 1984. (post-flight)

(5) Legros, J. C., Limbourg-Fontaine, M. C., and Pétré, G.: Surface Tension Induced Convection in Presence of a Surface Tension Minimum. In Scientific Results of the German Spacelab Mission D1, Norderney Symposium, Norderney, Germany, August 27-29, 1986, pp. 131-140. (post-flight)

(6) Limbourg, M. C., Legros, J. C., and Pétré, G.: Marangoni Convection Induced in a Fluid Presenting a Surface Tension Minimum as a Function of Temperature. In Proceedings of the 6th European Symposium on Material Sciences under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 245-249. (post-flight)

(7) Influence of a Surface Tension Minimum on the Marangoni Effect. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 62-63. (post-flight)

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**Experiment Origin:** Belgium
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Materials Science Double Rack (MSDR)
**Processing Facility:** Surface Tension Minimum (STEM) cells, attached to Fluid Physics Module (FPM) (same facility as the FPM of Spacelab 1 but with improvements)
**Builder of Processing Facility:** Unknown

**Experiment:**
Surface Tension Induced Convection Around a Surface Tension Minimum (WL-FPM-05)

This Spacelab D1 experiment was the third in a series of investigations designed by Legros et al. to study the effect of a surface tension minimum on thermocapillary convection (see Legros, TEXUS 8, TEXUS 9).

The earlier TEXUS 8 experiment in this investigative series subjected a free surface liquid cell to a temperature difference such that $T_1$ and $T_2$ were above the temperature at which the fluid has a surface tension minimum ($T_{min}$). The earlier TEXUS 9 experiment in this investigative series subjected a free surface liquid cell to a temperature difference such that $T_1$ was less than $T_{min}$ and $T_2$ was greater than $T_{min}$. During both of these earlier experiments, steady state convective flow conditions were not achieved because of the short low-gravity duration of the TEXUS flight (approximately 6 minutes).

The specific objective of this Spacelab D1 experiment was to observe the steady state convective flow of a liquid with a non-isothermal free surface. During two experiments performed on Spacelab D1 $T_1$ was to be greater than $T_{min}$ and $T_2$ was to be less than $T_{min}$. The experiment was designed to answer the following questions:

(1) What is the structure of the convective flow resulting from the imposed temperature difference (in the absence of buoyancy)?

(2) Will the fluid flow at the interface be from the $T_{min}$ zone towards both the hot and cold areas (an "opening" of the interface)?

(3) Will the flow behavior (velocity amplitude and convection pattern) agree with theoretical and numerical predictions?

(4) Will oscillatory Marangoni convection appear?

During the Spacelab D1 mission, two experiment cells attached to the Fluid Physics Module were used for this study (see Reference (2) for complete cell description). The cells were called STEM 1 and STEM 2. The procedure used was similar to that used during the TEXUS missions, except that during Spacelab D1 a Payload Specialist could interact with the experiment.

<Note: Publications released prior to the flight (see Reference (2) and Reference (5)) reported that two aqueous solutions would be examined: (1) n-heptanol (STEM 1) and (2) n-hexanol (STEM 2). However, all publications released after the Spacelab D1 flight discussed only results from the n-heptanol solution. It is unclear if the omission of the n-hexanol results was due to (1) a difficulty during the mission or (2) a pre-flight decision not to use this fluid.>

Under low-gravity conditions, the STEM was attached to the FPM and the observation volume was partially filled with n-heptanol aqueous solution ($6.04 \times 10^{-3}$ molal, $T_{min}$ approximately equal to 40 $^{o}$C). (The solution contained 90.7-micron diameter Latex particles.) Heating of one side of the observation volume was then initiated.

Post-flight examination of the video sequences indicated a thermocapillary-induced convective cell developed 30 seconds after heating began. The fluid moved from the cold to the hot side of the cell. After 15 minutes of heating, a temperature difference of 35 $^{o}$C (plus or minus 1 $^{o}$C) was established and remained constant until the end of heating at 50 minutes. During the first video sequence (3 minutes of heating), $T_1$ = 46 $^{o}$C and $T_2$ = 26 $^{o}$C, one convective cell was observed. The tracer velocity, v, at the surface was 0.5 mm/s with a v/(deltaT) = 0.025 mm/sK.

During the second video sequence (10 minutes of heating), $T_1$ = 60.5 $^{o}$C and $T_2$ = 28.2 $^{o}$C, the convective cell increased in size. The surface velocity was reported to be 1.2 mm/s and v/(deltaT) was 0.04 mm/sK. These values are similar to those recorded during the TEXUS 8 mission (v approximately = 0.8 mm/s and v/(deltaT) approximately = 0.04 mm/sK) with $T_1$ = 66 $^{o}$C and $T_2$ =

46 $^{o}$C.

<Note: Although it was reported that there was a third video sequence, little data from this sequence were presented. It was reported that only one convective cell was present and that "...closed streamlines appeared in the hot part of the cell." (8, p. 137)>

In the fourth video sequence (after about 45 minutes of heating), two convective cells contained within a larger cell were observed. <Note: The exact wall temperatures during this sequence were not reported.> Both cells' convective flows travel in the same direction: from the cold side to the hot side in the upper part of the cell. The surface velocity was 3 mm/s and v/(deltaT) was 0.08 mm/sK. The value of v/(deltaT) was lower than the steady state value, achieved under 1-g conditions, of 0.5 mm/sK.

Reportedly, further analysis of this data and correlation to theoretical and numerical studies was to be published at a later date. However, no documents providing these results could be located at this time.

**Key Words:** Fluid Physics, Open Cavity, Partially Filled Containers, Aqueous Solutions, Free Surface, Surface Tension, Surface Tension Gradients, Heat Transfer, Surface Tension Minimum, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Oscillatory Marangoni Convection, Cellular Convection, Buoyancy Effects Diminished, Thermal Gradient, Liquid/Gas Interface, Liquid Transfer, Liquid Reservoir, Tracer Particles

**Number of Samples:** two
**Sample Materials:** Unclear, although it appears (1) n-Heptanol aqueous solution containing latex tracer particles; (2) n-hexanol aqueous solution containing latex tracer particles (no results from the n-hexanol solution could be located)
**Container Materials:** unknown

**Experiment/Material Applications:**
See Legros, TEXUS 8.

**References/Applicable Publications:**

(1) Limbourg, M. C., Legros, J. C., and Pétré, G.: The Influence of a Surface Tension Minimum on the Convective Motion of a Fluid in Microgravity (D1 Mission Results). Adv. Space Res., Vol 6, No. 5, pp. 35-39, 1986. (post-flight)

(2) Limbourg-Fontaine, M. C., Pétré, G., and Legros, J. C.: Thermocapillary Movements Around a Surface Tension Minimum Under Microgravity Conditions. (Part I. Technical Description of the STEM Experiments. D1 Mission of Spacelab). Acta Astronautica, 13, pp. 197-208. (experiment cell description)

(3) Limbourg-Fonatine, M. C., Pétré, G., and Legros, J. C.: Thermocapillary Movements Under Microgravity at a Minimum of Surface Tension. Natur wissenschaften 73.Jahrgang Heft 7, July 1986, pp. 360-362. (post-flight)

(4) Legros, J. C., Limbourg-Fontaine, M. C., and Pétré, G.: Surface Tension Induced Convection Around a Surface Tension Minimum. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 93-94. (post-flight)

(5) Legros, J. C., Limbourg-Fontaine, M. C., and Pétré, G.: Surface Tension Induced Convection Around a Surface Tension Minimum. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 59-60. (preflight)

(6) Legros, J. C., Limbourg, M. C., Schwabe, D., and Lichtenbelt, J., and Frimout, D.: Some Results from Parabolic Flights. Adv Space Res. Vol. 6, No. 5, 1986 pp. 93-95. (related KC-135 studies)

(7) Limbourg, M. C., Legros, J. C., and Pétré, G.: Marangoni Convection Induced in a Fluid Presenting a Surface Tension Minimum as a Function of the Temperature. In Proceedings of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Brodeaux, France, December 2-5, 1986, ESA SP-256, pp. 245-249. (post-flight)

(8) Legros, J. C., Limbourg-Fontaine, M. C., and Pétré, G.: Surface Tension Induced Convection in Presence of a Surface Tension Minimum. In Proceedings of the Norderney Symposium on Scientific Results of the Germany Spacelab Mission D1. Norderney, Germany, August 27-29, 1986, pp. 131-140. (post-flight)

(9) Gonfalone, A.: The Fluid Physics Module- A Technical Description. In ESA 5th European Symposium on Material Sciences Under Microgravity. Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 3-7. (status post Spacelab 1, prior to D1)

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**Co-Investigator(s):** Limbourg, M. C. (2), Pétré, G. (3)
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**Experiment Origin:** Belgium
**Mission:** TEXUS 13
**Launch Date/Expt. Date:** April 1986
**Launched From:** ESRANGE, Kiruna Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-8: experiment cell equipped with fluid injection mechanism and electric heaters. The module was also equipped with a differential interferometer. (Same apparatus as used for TEXUS 9.)
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Effects of Surface Tension Minimum on Thermal Marangoni Convection in a Floating Zone

This TEXUS 13 experiment was the fourth in a series of investigations designed by Legros et al. to study the effect of a surface tension minimum on thermocapillary convection (see Legros, TEXUS 8, TEXUS 9, Spacelab D1).

The specific TEXUS 13 experiment goals and equipment setup were not detailed in the available publications. Reportedly, the experiment was performed in the TEM 06-8 TEXUS experiment module.

<Note: The TEM 06-8 module had been successfully employed for the TEXUS 9 experiment (see Legros, TEXUS 9 for the module description). However, it is unclear how this module produced a float zone system, (as the TEXUS 13 experiment title indicates) rather than a partially filled experimental cell (as the module did during TEXUS 9). (The Legros TEXUS 8 and Spacelab D1 experiments used a similar module which also created a partially filled cell.) It is unclear if the investigators actually produced a float zone (or liquid bridge) during the TEXUS 13 experiment or instead, used a partially filled cell and attempted to apply the results to a float zone system.>

The experiment procedures of previous investigations in this series are outlined under Legros, TEXUS 8, TEXUS 9, or Spacelab D1. Presumably, during the TEXUS 13 rocket flight, (1) either (a) an experiment cell was to be partially filled or (b) a float zone was to be created; (2) a temperature difference was to be placed across the cell or zone; and (3) the resulting convective patterns were to be observed.

Reportedly, "No results could be gained due to a technical... [failure]. The slider did stick and no fluid was introduced in the cell." (1, p. 43)

No further information concerning this experiment could be located.

**Key Words:** Fluid Physics, Float Zones, Liquid Bridges, Aqueous Solutions, Free Surface, Surface Tension, Surface Tension Minimum, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Thermal Gradient, Liquid/Gas Interface, Liquid Transfer, Liquid Reservoir, Liquid Injection, Containerless Processing Applications, Interferometric Measurement, Hardware Malfunction, Sample Not Processed as Planned

**Number of Samples:** unknown, probably one
**Sample Materials:** N-Heptanol 6.3 x $10^{-3}$ molal aqueous solution. This substance presents a minimum of surface tension as a function of temperature at 40 $^{o}$C. (Reference (1), p. 42)
**Container Materials:** unknown

**Experiment/Material Applications:**
See Legros, TEXUS 8.

**References/Applicable Publications:**

(1) Limbourg, M. C., Pétré, G., and Legros, J. C.: Thermocapillary Motions on a Surface Tension Minimum. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 42-43. (post-flight)

(2) Legros, J. C., Sanfeld, A., and Velarde, M. G.: Fluid Dynamics. In Fluid Sciences and Materials Science in Space. Edited by H. U. Walter, Springer Verlag, 1987, pp. 83-137. (related topics)

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**Experiment Origin:** Belgium
**Mission:** TEXUS 15
**Launch Date/Expt. Date:** May 1987
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-8
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Surface Tension Minimum

This TEXUS 15 experiment was the fifth in a series of investigations designed by Legros et al. to study the effect of a surface tension minimum on thermocapillary convection (see Legros, TEXUS 8, TEXUS 9, Spacelab D1, TEXUS 13).

The specific TEXUS 15 experiment goals and equipment setup were not detailed in the available publications.

Reportedly, shortly after the successful launch of the rocket, data and television transmitters experienced a partial failure. It was discovered that a lateral burnthrough of the second stage of the rocket had occurred, and the stage, in turn, had collided with the prematurely separated payload. An unbraked impact of the lower part of the payload resulted in the destruction of the TEM 06-8 Experiment Module.

Documentation further detailing any in-flight results of this experiment does not appear to be available.

**Key Words:** Fluid Physics, Free Surface, Surface Tension, Surface Tension Minimum, Surface Tension-Driven Convection, Marangoni Convection, Acceleration Effects, Rocket Motion, Payload Survivability, Rocket Failure

**Number of Samples:** unknown, possibly one
**Sample Materials:** unknown

**Container Materials:** unknown

**Experiment/Material Applications:**
See Legros, TEXUS 8.

**References/Applicable Publications:**

(1) Experimentelle Nutzlast und Experimente TEXUS 15. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 107-108. (in German; post-flight)

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**Experiment Origin:** Belgium
**Mission:** TEXUS 16
**Launch Date/Expt. Date:** November 1987
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-8
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Influence of a Surface Tension Minimum on the Marangoni Effect

This TEXUS 16 experiment was the sixth in a series of investigations designed by Legros et al. to study the effect of a surface tension minimum on thermocapillary convection (see Legros, TEXUS 8, TEXUS 9, Spacelab D1, TEXUS 13, TEXUS 15).

The specific TEXUS 16 experiment goals and equipment setup were not detailed in the available publications.

Reportedly, shortly after the successful launch of TEXUS 16, fuel in the second stage of the rocket did not ignite as planned. After the apogee was reached and the rocket began to fall, the yo-yo despin system was deployed as programmed. Due to the unexpected excess rocket mass, however, there was an incomplete reduction of rocket spin. Subsequently, the payload separated from the second stage, but the parachute was not released. An unbraked impact of the payload resulted in the destruction of all experiment modules including TEM 06-8.

Documentation further detailing any in-flight results of this experiment does not appear to be available.

**Key Words:** Fluid Physics, Free Surface, Surface Tension, Surface Tension Minimum, Surface Tension-Driven Convection, Marangoni Convection, Rocket Motion, Acceleration Effects, Payload Survivability, Rocket Failure, Payload Recovery System Failure

**Number of Samples:** unknown, possibly one
**Sample Materials:** unknown
**Container Materials:** unknown

**Experiment/Material Applications:**
See Legros, TEXUS 8.

**References/Applicable Publications:**
(1) Die Kampagne TEXUS 16. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 109-111. (in German; post-flight)

(2) Legros, J. C., Sanfeld, A., and Verlarde, M. G.: Fluid Dynamics. In Fluid Sciences and Materials Science in Space, Edited by H. U. Walter, Springer Verlag, 1987, pp. 83-137. (related topics)

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**Principal Investigator(s):** Perez, R. (1), other students (2)
**Co-Investigator(s):** Snow, J. T. (3)
**Affiliation(s):** (1,2) During STS-007: Purdue University, West Lafayette, Indiana, Currently: Unknown; (3) Purdue University, West Lafayette, Indiana

**Experiment Origin:** USA
**Mission:** STS Launch #7, STS-007 (STS 31-C, Challenger)
**Launch Date/Expt. Date:** June 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment
NASA Get Away Special (GAS) Canister G-009
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-009:** Purdue University, West Lafayette, Indiana
**Processing Facility:** (a) An experiment cell containing a mercury drop suspended within a host fluid, and (b) a 16 mm motion picture camera.
**Builder of Processing Facility:** Unknown, possibly, Purdue University, West Lafayette, Indiana

**Experiment:**
Motion of Mercury Under Low-G

This experiment was one of three investigations housed within the G-009 Get Away Special (GAS) canister on STS-007. Neither of the other two experiments (in the canister) were applicable to this data base. The objective of the experiment was to observe the behavior of a mercury drop immersed in mediflor (a clear, perfluorinated hydrocarbon host liquid). It was anticipated that the following would be observed: (1) drop acceleration, (2) drop breakup into particles, (3) eventual particle coalescence, (4) drop oscillation, and (5) drop oscillation damping.

Prior to the shuttle launch, a 0.55 cc drop of mercury was added to a small experiment cell filled with 10 cc of mediflor. The cell, constructed primarily of stainless steel but configured with a transparent endplate, was mounted at the focal point of an optical assembly. Light emitting diodes embedded in the endplate illuminated the cell, allowing the visualization of the drop motion through the transparent plate. A modified World War II aircraft wing camera, configured with 15 m of 16 mm motion picture film was available to document the drop dynamics for 2 minutes (24 frames/sec).

A document published prior to the flight indicated that "The experiment, once activated by the crew is controlled by an accelerometer and microprocessor. The accelerometer senses [the]

Orbiter motion, such as a roll maneuver, sends the data to the microprocessor, which in turn generates a signal to turn the camera on for a brief period." (1, p. 2-6)

<Note: It should be mentioned that because the information above was taken from a document published prior to the launch of STS-007, and because no document published after the return of STS-007 detailed the accelerometer contribution, it is not certain if an accelerometer/microprocessor system was actually employed during the mission.>

During the mission, the experiment was to be initiated about 5 minutes before the Shuttle performed an orbital maneuver. It was expected that (1) the accelerations generated during the maneuver would cause the mercury to move about the container, (2) the drop would break up, but quickly rejoin to form a single oscillating mass (as indicated during preflight testing), and (3) these oscillations would gradually damp out because of viscous effects.

Reference (4) states that the post-flight evaluation of the payload indicated that the camera failed to operate. "Inspection did not reveal any mechanical problems with the camera mechanism itself, but it is suspected that the film magazine (a sealed unit containing several moving parts) jammed because of vibration during lift-off...." (4, p. 446) However, Reference (6), which was published almost 3 years later, very briefly indicated that the experiment failure, which did occur, was related to "a short in the power system." <Note: Further details of the "power system" were not provided in the Reference.>

<Note: Reference (3) (also published early) detailed the overall canister difficulties. Reportedly, another experiment in the canister (a seed germination experiment) failed when an electric short occurred "...when the payload structure was accidentally placed over a wire from the battery pack." (3, p. 13) Reference (3) further detailed that the failure of the fluid dynamics experiment was independent of the seed germination experiment because the fluids experiment employed "a different battery set," and "Although there is no concrete evidence, a possible source of problems is the film cartridge used by the camera." (3, p. 14)>

"It was also discovered that the mediflor solution in the fluid cell had been lost. The O-ring seal on the cell's transparent cover appeared to be in good condition, though there was some distortion of the... [Plexiglas™] cover itself. Some particles of mercury were found embedded under the O-ring (though no evidence was found of mercury leakage past the O-ring). It was conjectured that, again, the high levels of vibration and acoustic noise at the lift-off were responsible for the distortion of the cover and leakage of the volatile mediflor." (4, p. 446)

**Key Words:** Fluid Physics, Drops, Contained Fluids, Liquid/Liquid Interface, Surface Tension, Hydrodynamics, Liquid Dynamic Response, Acceleration Effects, Drop Dynamics, Drop Oscillation, Drop Coalescence, Viscosity, Fluid Motion Damping, Optics Assembly, Rocket Vibration, Liquid Leakage, Photographic Difficulties, Hardware Malfunction

**Number of Samples:** one

**Sample Materials:** A drop of Mercury (0.55 cc) was immersed in 10 cc of a clear, perfluorinated hydrocarbon host liquid called mediflor.

(Hg*)

**Container Materials:** Primarily stainless steel; transparent endplate: Plexiglas™.

**Experiment/Material Applications:**

The experiment was designed to position a drop of mercury such that a detailed study of the resulting drop dynamics could be performed. Other experiments within the data base have relied on more complex experiment configurations (to produce acoustic fields) to position such drops for observation (see, for example, Wang, SPAR 4 (this chapter)).

**References/Applicable Publications:**

(1) Cargo Systems Manual: Gas Annex for STS-7. JSC-17645 Annex STS-7, April 1, 1983. (short description; preflight)

(2) STS-7 Getaway Specials, NASA News, NASA GSFC, May 1983. (preflight)

(3) Perez, R.: Results of the Purdue University GAS-1 Payload. In Goddard Space Flight Center's 1984 Get Away Special Experimenter's Symposium, August 1-2, 1984, pp. 9-15, NASA CP-2324. (post-flight; no detailed results of this experiment discussed)

(4) Snow, J. T., Crabill, W. H., Kark, D. J., Levin, R., McGinnis, M. E., Perez, R., Stark, J. A., Stwalley, R. M., and Weber, D. A.: A Shuttle Payload Project: PUGAS-1 The First Purdue University Small Self Contained Payload Project. Space Education, Vol. 1, October 1985, pp. 442-447. (post-flight)

(5) Stark, J. A.: Thermal Control System of the Purdue University Space Shuttle Payload, AIAA Journal of Spacecraft and Rockets, May-June 1985.

(6) Olenski, C., Crocker, A. R., Stubbings, B., McCormick, L., and Andersen, P. K.: The Purdue University Get Away Special II. In NASA Goddard Space Flight Center 1988 Get Away Special Experimenter's Symposium, September 27-30, 1988, pp. 109-111. (post-flight G-009; preflight of Purdue's second GAS Canister)

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(8) NASA STS-6 Press Kit, June 1983. (preflight)

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**Experiment Origin:** Italy
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Spacelab Rack, Material Sciences Double Rack (MSDR)
**Processing Facility:** Fluid Physics Module (FPM)
**Builder of Processing Facility:** Centro Ricerche FIAT, Torino, Italy

**Experiment:**
Free Convection in Low Gravity 1ES328

On Earth, thorough study of gravity-independent Marangoni convection is not possible because gravity-dependent forces mask the importance of the Marangoni flow. In a reduced gravity environment, the Marangoni flow may be highly significant in all experiments in which fluid-fluid interfaces of non-uniform properties exist.

This Spacelab 1 experiment was the first in a series of low-gravity investigations designed by Napolitano and/or Monti et al. to study thermal Marangoni convection in a liquid bridge. The bridge configuration was used to model a float zone fluid system.

The first objective of the Spacelab 1 experiment was to verify theoretical calculations which indicated that (1) stable liquid bridges of 10 cm height could be established and (2) Marangoni flow created in the zone would be a of boundary layer type. The second objective of the experiment was to investigate these Marangoni flows under a number of parameters (e.g., stimuli) to determine the local and global properties of the system. (Reportedly, local properties included velocity fields, global properties included overall transport coefficients.)

The experiment was performed in the Spacelab Fluid Physics Module (FPM). Each liquid bridge was created between two coaxial aluminum disks, and then one of the disks was heated. The disks could be (1) rotated, (2) vibrated axially, or (3) positioned according to desired axial distance. In addition, an electric potential difference could be applied to the liquid bridge.

During the mission, several silicone oil bridges (of either 100 cS or 5 cS) were to be created. Reportedly, "...the tracers contained in the more viscous oil did not appear in the liquid column. As a consequence: the 100 cS silicone oil could not be used and all runs had to be made with the less viscous 5 cS oil...." (3, p. 77)

All of the experiment runs (four total) were filmed and a video/voice downlink allowed interaction between the scientist on the ground and the shuttle payload specialist. <Note: Reference (3) contains planned and actual experimental details.>

Several deviations from the nominal performance of the experiment were encountered. "The not ideal quality of the [16 mm] film exposed and a number of problems... [which arose] during the mission made it impossible to reach all the objectives of the experiment and in particular those related to quantitative measurement of the velocity field." (1, p. 25) Some of these reported difficulties are listed below:

(1) "...the 'antispreading barrier' coated on the lateral surface of the disks to prevent the silicone oil from spreading over the edges of the supporting disks did not work properly." (3, p. 77)

(2) "...the FPM had... to be operated outside the MSDR [Material Science Double Rack] in order to provide complete accessibility to all parts of the module...." (3, p. 78)

(3)"...the entrainment of bubbles in the reservoirs and hence in the liquid bridges... although obviously altering the flow field, was nonetheless not entirely negative since we hope to be able to gain some insights on the Marangoni migration of bubbles in a Marangoni flow." (3, p. 78)

Post-flight analysis of the available temperature and photographic data illustrated that liquid bridges of 7 to 8 cm in length with aspect ratios on the order of one were established. The application of an electric potential had no effect on the flows.

Most importantly, it was reported that the established Marangoni flows were of the boundary layer type. This type of flow was predicted by theoretical calculations (see Reference (1) and/or Reference (3) for theoretical details). The thickness of this boundary layer was estimated from the films. The conditional Peclet number and the Marangoni velocity were determined by evaluation of various parameters. These parameters included

"...the velocity at a discrete number of points near the interface and in the recirculating core and... the measured temperature difference between the two disks...." (3, p. 81) It was reported that typical values of (1) the conditional Peclet number were $3 \times 10^5$, (2) maximum velocities were 1 cm/s, and (3) velocities at the core were 0.1 cm/s. These data were in perfect agreement with an accompanying order of magnitude analysis (Reference (1) and Reference (3)). <Note: Reference (1) also includes thermal analysis of the experiment including determination of the Nusselt number expression.>

**Key Words:** Fluid Physics, Liquid Bridges, Transparent Liquids, Free Surface, Surface Tension, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Liquid/Gas Interface, Boundary Layer, Heat and Mass Transfer, Flow Velocity, Liquid Bridge Stability, Hydrodynamics, Liquid Dynamic Response, Aspect Ratio, Rotating Fluids, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Bubbles, Bubble Formation, Marangoni Movement (Migration) of Bubbles, Bubble Migration, Thermal Gradient, Electric Field, Liquid Spreading, Wetting Kinetics, Viscosity, Tracer Particles, Coated Surfaces, Containerless Processing Applications

**Number of Samples:** Four runs were completed.
**Sample Materials:** (1) 100 cS silicone oil (not used due to inability to see tracers) and (2) 5 cS silicone oil
Tracer Material: Echosphere (glass); outer diameter = 50-75 microns
**Container Materials:** Not applicable; disk material: aluminum (Al*)

**Experiment/Material Applications:**
See Martinez, TEXUS 10 or Napolitano, Spacelab D1.

**References/Applicable Publications:**
(1) Napolitano, L. G.: Recent Development of Marangoni Flows Theory and Experimental Results. Advances in Space Research, Vol. 6, Number 5, 1986, pp. 19-34. (post-flight; discusses theoretical background, Spacelab 1 experiment results, and Spacelab D1 experiment results)

(2) Napolitano, L. G., Monti, R., and Russo, G.: Some Results Of the Marangoni Free Convection Experiment. In ESA 5th European Symposium on Materials Sciences Under Microgravity, Results Of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 15-22. (post-flight)

(3) Napolitano, L. G., Monti, R., and Russo, G.: Marangoni Convection in Low Gravity--Experiment 1ES 328. Earth Oriented Applications Of Space Technology, Vol. 5, No. 1-2, 1985, pp. 69-82. (post-flight)

(4) Chassay, R. P. and Schwaniger, A.: Low-g Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements on Spacelab 1)

(5) Monti, R., Napolitano, L. G., and Russo, G.: Experimental Study of Thermal Marangoni Flows in Silicon Oil Floating Zones. In Proceedings of 34th International Astronautical Federation, International Astronautical Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-161. (preflight)

(6) Da Riva, I. and Napolitano, L. G.: Fluid Physics Under Reduced Gravity - An Overview. In Proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, pp. 5-12. (preflight)

(7) Lioy, S., Defilippi, V., and Monti, R.: Microgravity Fluid Science Facilities Operation. In 38th IAF International Astronautical Congress, Brighton, England, October 10-17, 1987, IAF Paper #87-411. (discusses experiment hardware)

(8) Napolitano, L. G., Monti, R., and Russo, G.: Marangoni Convection in One- and Two-Liquid Floating Zones. In Naturwissenschaften 73, pp. 352-355. (post-flight; briefly mentions Spacelab 1 experiment)

(9) Input received from R. Fortezza, August 1993.

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**Experiment Origin:** Italy
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Materials Science Double Rack (MSDR)
**Processing Facility:** Fluid Physics Module (FPM) (The FPM was the same facility as had been used on Spacelab 1 but was improved for Spacelab D1.)
**Builder of Processing Facility:** Centro Ricerche FIAT, Torino, Italy

**Experiment:**
Marangoni Flows-A study of surface-driven convection phenomena in very low gravity (WL-FPM-07)

This Spacelab D1 experiment was the third in a series of low-gravity investigations designed by Napolitano and/or Monti et al. to study thermal Marangoni convection in a liquid bridge (see Napolitano, Spacelab 1 and Monti, TEXUS 9 (both in this chapter)). The liquid bridge configuration was used to model a float zone fluid system.

The specific objectives of this investigation were to (1) quantitatively examine thermal Marangoni flows in a single-liquid system, (2) examine the thermal and/or solutal Marangoni flows in a two-liquid system, and (3) analyze the effects of certain parameters (e.g., geometry of supporting disks, imposed temperature differences) on (1) and (2).

The experiment was performed in the Spacelab Fluid Physics Module (FPM). Two experimental runs were performed. (Reportedly, there were difficulties with the voice/video downlink systems and the camera used to film the process during both experiments. "...it must be noted that the 16-mm film, exposed during [the first experimental run], was completely destroyed because of the failure of the Vinten camera mounted on the FPM...." (1, p. 354) The first run was analyzed utilizing a video tape of the experiment recorded at the German control center during the mission.)

During the first run, a silicone oil liquid bridge was created between two disks. One of the disks was heated to 60 °C and then isorotated. The disk was then heated to 75 °C. Reportedly, "Because of the presence of an overheating condition, the heater of the FD [Feeding Disk] was automatically turned off, bringing rapidly the temperature of the disk down to about 45 °C." (1, p. 353)

Post-flight analysis of the first experimental run indicated that a 6 cm long, cylindrically shaped float zone was created. The first heating of the disk produced non-oscillatory Marangoni flows. Isorotation of the disks (10 rpm, Peclet Number $Pe_{omega} = 2.7 \times 10^4$) demonstrated that the rotational effects were of the same order as the thermal Marangoni effects. It was reported by the Payload Specialist that "...after the breakage [of the fluid bridge] under non-isothermal conditions, the bridge was... reformed [with difficulty], demonstrating the influence of the Marangoni effect on the coalescence...." (1, p. 353) Assuming the physical properties of silicone oil did not change with temperature, analysis of the thermal and photographic data indicated that: (1) the Marangoni velocity equaled 40 cm/s, (2) the conditional Peclet number equaled $4 \times 10^5$, and (3) the dimensionless reference velocity equaled $10^{-2}$. Although the conditional Peclet number was higher than the critical value, no oscillatory flow was observed.

A preliminary analysis of the velocity profiles from the flight experiment indicated a certain agreement with numerical values (based on an integration of the Navier-Stokes equations with a finite difference scheme). Reference (2) contains a graph depicting the dimensionless axial velocity versus the fixed distance from the disk and symmetry axis.

During the second run, a liquid bridge was formed by bringing one drop of 5 cS silicone oil on the cold concave disk and one drop of d-octyl phthalate on a 60 °C heated disk in contact. However, "A number of problems arose during the injection phases [injection of the fluid drops on the disks] due to the malfunction of one of the motors of the Fluid Physics Module...." (1, p. 354) Therefore, only qualitative data were available from the second experimental run. It was reported that once the drops of the two liquids contacted, a liquid bridge was formed immediately. Upon heating, the Payload Specialist reported "...one liquid going into the center of the liquid column, just like a bubble." (1, p. 354)

**Key Words:** Fluid Physics, Liquid Bridges, Liquid Bridge Stability, Liquid Column Rupture, Transparent Liquids, Free Surface, Surface Tension, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Critical Marangoni Number, Thermosolutal Convection, Thermal Gradient, Liquid/Gas Interface, Sample Rotation, Rotating Fluids, Flow Velocity, Drops, Drop Coalescence, Droplet Collision, Liquid/Liquid Interface, Solutal Gradients, Coated Surfaces, Tracer Particles, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Liquid Injection, Containerless Processing Applications, Photographic Difficulties, Hardware Malfunction

**Number of Samples:** Two floating zones were created.
**Sample Materials:** (1) first zone: silicone oil (5 cS), (2) second zone: silicone oil and d-octyl phthalate
tracer material: Echosphere (glass); outer diameter = 50-75 microns
**Container Materials:** Not applicable; disk material: aluminum; anti-spreading coating: 3M FC-721™
(Al*)

**Experiment/Material Applications:**
The liquid bridge configuration models the liquid behavior of a crystal melt during float zone processing.

**References/Applicable Publications:**

(1) Napolitano, L. G., Monti, R., and Russo, G.: Marangoni Convection in One and Two- Liquid Floating Zones. Natur wissenschaften, 73, Jahrgang, Heft 7, July 1986, pp. 352-355. (post-flight)

(2) Napolitano, L. G.: Recent Development of Marangoni Flows Theory and Experimental Results. In Advances in Space Research, Volume 6, Number 5, pp. 19-34, 1986. (post-flight)

(3) Napolitano, L. G., Monti, R., and Russo, G.: The Experiment WL-FPM-07 on Marangoni Flows. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 33-34. (post-flight)

(4) Napolitano, L. G., Monti, R., and Russo, G.: Marangoni Flows. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 56-57. (preflight)

(5) Monti, R.: On the Onset of the Oscillatory Regimes in Marangoni Flows. In Acta Astronautica, Vol. 15, Number 8, 1987, pp. 557-560. (post-flight; discusses conclusions from TEXUS, Spacelab 1, and Spacelab D1 experiments)

(6) Napolitano, L. G. and Monti, R.: Surface Driven Flows: Recent Theoretical and Experimental Results. In Proceedings of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 551-555. (post-flight; not mission specific)

(7) Monti, R., Napolitano, L. G., and Russo, G.: Experimental Study of Thermal Marangoni Flows in Silicon Oil Floating Zones. In 34th IAF International Congress, Budapest, Hungary, October 10-15, 1983, IAF Paper #83-161. (preflight)

(8) Langbein, D.: Fluid Physics. In Scientific Results of the German Spacelab Mission D1, Norderney Symposium, Norderney, Germany, August 17-29, 1986, pp. 93-104.

(9) Input received from R. Fortezza, August 1993.

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<Note: The Principal Investigator listed a new address in Garches, France. However, the name of the present company/university that employs the Investigator was not indicated.>

**Experiment Origin:** France
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility
**Processing Facility:** Fluid Physics Module (FPM)
**Builder of Processing Facility:** Fiat, Torino, Italy

**Experiment:**
Oscillation of Semi-Free Liquid in Space (1ES326)

The objective of this Spacelab 1 experiment was to study the oscillation damping of a liquid sphere in a reduced gravity environment.

During the mission, it appears several silicone oil spheres were injected (one at a time) onto a circular surface of a plate capable of axial vibrations. The Principal Investigator reported that the diameter of these spheres ranged from "2,7 cm to 2 cm for larger spheres and from 2 cm to 1,7 cm for little spheres." (Reference (4)) Oscillations of the liquid were developed and then progressively amortized. A camera documented the movements and the shape of the liquid was recorded as a function of time. The oscillation amplitudes and frequencies were thus obtained as a function of the initial diameter of the liquid sphere and other geometrical parameters. Reportedly, two mathematical models, one by Bisch, Lasek, and Rodot and the other by Strani and Sabetta were compared to the experimental results.

<Note: Reference (2) (below) was not translated prior to preparing this summary. However, the Principal Investigator reported that the reference contains information on (1) employed plate material, (2) the frequencies of the plate axial vibrations, (3) the time duration of the experiment, (4) the resultant oscillation amplitudes and frequencies of the sphere, (5) the nature of the mentioned mathematical models, and (6) the experiment conclusions.>

No other information concerning this experiment (published in English) could be located at this time.

**Key Words:** Fluid Physics, Drops, Transparent Liquids, Liquid Dynamic Response, Free Surface, Surface Tension, Meniscus Shape, Sphericity, Meniscus Vibration, Liquid Vibration, Drop Vibration, Drop Oscillation, Oscillation Amplitude, Oscillation Frequency, Resonant Frequency, Liquid Expulsion Through a Small Orifice, Droplet Creation Via a Syringe, Drop Formation, Solid/Liquid Interface, Liquid/Gas Interface, Containerless Processing Applications

**Number of Samples:** Unspecified by Principal Investigator.
**Sample Materials:** Silicone oil (5 cS)
**Container Materials:** Not directly applicable; plate material and any applied plate coatings: unclear

**Experiment/Material Applications:**
This experiment was performed to investigate the optimum conditions for the levitation of materials during crystal growth. Reportedly, optimal geometrical parameters and vibration frequencies to be avoided were among the conditions investigated.

**References/Applicable Publications:**

(1) Langbein, D.: Liquids and Interfaces. In ESA 5th European Symposium On Material Sciences Under Microgravity, Results Of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 443-446. (post-flight)

(2) Rodot, H. and Bisch, C.: Oscillations of Semi-Free Liquids in Microgravity Spacelab Experiment ES 326. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results Of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 23-29. (in French; post-flight)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

(4) Input received from Experiment Investigator, October 1989 and August 1993.

(5) Rodot, H., Bisch, C., Lasek, A.: Acta Astronautica, 6 (1979), p. 1083.

(6) Rodot, H. and Bisch, C.: Oscillations de Sphères Liquides Semi-Libres en Microgravité- Système de positionnement et d' Amortissement. Document sent by H. Rodot to J. Jones (Wyle), August 1993. (in French)

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**Experiment Origin:** The Netherlands
**Mission:** STS Launch #9, STS-009 (STS 41-A Spacelab 1: Columbia)
**Launch Date/Expt Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Materials Science Double Rack (MSDR)
**Processing Facility:** Fluid Physics Module (FPM)
**Builder of Processing Facility:** Unknown

**Experiment:**
Liquid Motions in Partially Filled Containers (1ES330)

This Spacelab 1 experiment was the first in a series of investigations designed by Vreeburg to study the motion of contained liquids under low-gravity conditions. The specific objectives of the experiment were to (1) study the behavior of a liquid enclosed in a container during spin-up and spin-down of the container, (2) study the response of the contained liquid to forced oscillations (while the container is in a stationary and rotating state), and (3) determine the limitations of a computational model to predict fluid behavior. Reportedly, "Of special interest was the nonlinear growth and decay of the 1/2 sub-harmonic resonance of the free liquid surface." (1, p. 37)

The experiment was one of seven investigations performed in the Fluid Physics Module (FPM) during the mission. The experimental hardware associated with this particular experiment consisted of: (1) five cylindrical containers (6 cm outer diameter, 12 cm high), (2) a Perspex™ viewing lens (to correct for optical distortion), (3) a baseplate for mounting the container on the end of the FPM, and (4) a film camera (16 mm film). Four of the five cylindrical containers had cavities of the following shapes: (1) cylindrical, (2) annular cylindrical, (3) spherical, and (4) rectangular. The fifth container was configured with "...a cylinder and a cylindrical annulus side-by-side and normal to the axis of symmetry of the external cylindrical shape." (1, p. 37) Each container was half-filled with silicone oil of 3 cS viscosity (30 dyne/cm surface tension). The silicone oil contained aluminum flakes for flow observations. (See Reference (1), Figure 1, p. 40.) <Note: Details of the low-gravity experimental sequence were not presented in the available publications.>

<Note: At the time this experiment summary was written, only preliminary results could be located in the published literature. These results are discussed below.>

The data from this experiment "...were to consist of three rolls of film containing images of the liquid in the various containers, plus a print-out of the data, transmitted via the MSDR computer. These data are the actual performance parameters of the FPM, and timing bits to allow correlation of performance and pictorial data. Additional data were expected from the crew's comments during execution of the experiment. However, this highly valued commentary did not materialize, as a consequence of a confounded invention, called 'negative reporting,' which was put in force on Spacelab 1." (2, pp. 83-84) Also, the "Film was generally underexposed and sometimes damaged by the film transport mechanism. Nevertheless quantitative evaluation of pictorial information is possible.... In addition to the nominal data yield, video and 16 mm colour film recordings were produced of various activities with the containers of 1 ES 330 outside the FPM. A fascinating video clip shows Ulf Merbold, assisted by Bob Parker, exchanging the liquid from the cylinder container for water. The water had an almost 90...[degree]... contact angle with the container wall and this allowed some entertaining fluid dynamics demonstrations. Subsequently, a number of experiments with the cylinder container were performed. During some of these it was shown that a hydrostatic configuration in a spinning cylinder, whereby all liquid was centrifuged to the cylindrical wall, could be maintained through a careful spin-down of the cylinder to a stationary state." (2, p. 85) Color film of the annular cylinder free-floating in Spacelab indicated a non-axisymmetric hydrostatic equilibria of the liquid.

"The flight film from the FPM was collected a few days after the landing of STS-9 at Edwards AFB. After processing, the film was taken back to Europe and distributed to the investigators. However, the relevant FPM operational data, that have to be extracted from the total MSDR data records, are not yet available. These quantitative data of the conditions that provoked the liquid behavior recorded on film are necessary for definitive interpretation." (2, p. 88)

Some quantitative information could be obtained from a cylinder which had a grid scratched on the inner surface (grid lines were 24 degrees apart around the cylinder circumference). "When, in the FPM, one of these lines is illuminated by the thin beam (slit illumination) the light is sufficiently scattered that the line becomes a source. The image of this source is recorded by the camera, usually in multiple ways." (2, p. 88) It was determined, from the curvature of the images, that the free liquid surface is either a refracting or reflecting element in the light ray trace.

It was intended that the quantitative data obtained from this experiment would be used to confirm prediction methods. These predictions were obtained utilizing the computer program SAVOF. "SAVOF is a finite difference code of the... Marker-and-Cell (MAC)/Volume-of-Fluid family." (2, p. 83) However, because of the unavailability of quantitative data, this analysis could not be performed.

No further information concerning this experiment could be located at this time.

**Key Words:** Fluid Physics, Contained Fluids, Partially Filled Containers, Propellant Tanks, Spinning Containers, Rotating Fluids, Transparent Liquids, Free Surface, Surface Tension, Meniscus Shape, Contact Angle, Container Shape, Fluid Oscillation, Liquid Dynamic Response, Resonant Frequency, Viscosity, Solid/Liquid Interface, Liquid/Gas Interface, Tracer Particles, Optics Assembly, Illumination System, Photographic Difficulties

**Number of Samples:** Unclear, there were five containers (although multiple runs may have been made for each container).
**Sample Materials:** silicone oil (viscosity = 3 cS, surface tension = 20 dyne/cm) with tracers (flakes)
**Container Materials:** Perspex™

**Experiment/Material Applications:**
Often, spacecraft have fuel tanks of fluid which are partially filled. If a large enough empty tank volume is available the movement of this fluid will exert forces on the spacecraft. It was reported that at the time this experiment was performed, a reliable method to predict these forces did not exist. Therefore, large safety margins had to be accounted for to prevent spacecraft instabilities. It was expected that low-gravity fluid motion experiments such as these would lead to a better understanding of these forces.

**References/Applicable Publications:**

(1) Vreeburg, J.P.B.: Axisymmetric Free Surface Behavior Of Moving Liquid in a Cylinder in Microgravity. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results Of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 37-42. (post-flight)

(2) Vreeburg, J.P.B.: Overview Of Spacelab Experiment 1 ES 330-Coupled Motion of Liquid-Solid Systems in Reduced Gravity. Earth Oriented Applications of Space and Technology, Vol. 5, No. 1-2, 1985, pp. 83-89. (post-flight)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

(4) Vreeburg, J.P.B.: Observations on Behavior of Liquid in Weightlessness. In Proceedings of the 30th Anniversary of the von Karmon Institute for Fluid Dynamics, Rhode-St-Genese, Belgium, June 1986. (post-flight)

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**Experiment Origin:** The Netherlands
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Materials Science Double Rack (MSDR)
**Processing Facility:** Fluid Physics Module (FPM) (the same facility as the FPM of Spacelab 1 but with improvements)
**Builder of Processing Facility:** Unknown

**Experiment:**
Liquid Motions in Partially Filled Containers (WL-FPM-08)

This Spacelab D1 experiment was the second in a series of investigations designed by Vreeburg to study the motion of contained liquids under low gravity conditions (see Vreeburg, Spacelab 1). The specific objectives of this study were to (1) verify the results of a computer model which predicted contained liquid behavior in response to certain excitations and (2) obtain information which would lead to further developments of numerical models of fluid dynamics.

The experiment was conducted in the Fluid Physics Module (FPM). Three experiment containers (made of Perspex™), each with a differently shaped cavity, were to be used during this experiment: (1) spherico-cylinder, (2) double torus, and (3) thin annulus. The cylindrical cavity, designated as "FLIM-1," was partially filled with doubly-distilled water which contained Iriodine (a tracer). Reportedly, the inside surface of the this cylindrical cavity "...bears 32 equispaced meridional scratch lines that show up as luminous lines in the thin parallel beam illumination of the FPM." (5, p. 112) The other two cavities, designated as "FLIM-2" and "FLIM-3," were partially filled with silicone oil.

During the Spacelab D1 mission, the FLIM-1 container was subjected to six sequences of excitation: (1) no rotation, (2) 25 rpm rotation, (3) 33 rpm rotation, (4) spin-down from 35 rpm to 0 rpm, (5) spin-up from 0 rpm to 35 rpm, and (6) no rotation. During the first five sequences the container was also subjected to oscillations of a maximum amplitude of 0.5 mm and a frequency above 1.9 Hz. The container was not oscillated during the sixth sequence.

It is unclear from the available references if data were obtained from FLIM-2 and FLIM-3. Reference (5) briefly stated that "...they [FLIM-2 and FLIM-3] are excluded from consideration in this paper since they have not been operated to yield substantial experimental data." (5, p. 113)

Post-flight analysis of the film of FLIM-1 concentrated on the study of (1) the movement of the contact line between the liquid and the cavity and (2) the oscillation frequency of the liquid. Analysis from sequence 1 (no rotation) indicated that there is some slip of the contact line during oscillation of the liquid surface. Graphing the average contact line position versus time (see Reference (5) for details) for sequence 5 (2.38 Hz oscillation), indicated that "...the contact line moves upward in the course of the adjustment of the liquid to the rotation of the container." (5, p. 115) The graph illustrated certain irregularities which had not been interpreted as of the latest, available publication. However, it was reported that "Stickslip of the contact line does not appear [a likely cause of the irregularities] since the container is oscillated at 2.38 Hz." (5, p. 115)

During the execution of sequences 1 to 3, the Payload Specialist was required to locate the oscillation frequency which resulted in the maximum response amplitude of the liquid. The displacement of a tracer particle with respect to a fixed container point was plotted. Fourier analysis of this data revealed a resonance peak of 2.13 Hz. Within experimental error, this agrees with the peak frequency (2.12 Hz) observed by the Payload Specialist. The same type of agreement occurred when the excitation during sequence 3 was analyzed using a point on a bubble in the liquid. Analysis of the radial location of the bubble (with Fourier decomposition) revealed the correct rotational velocity (33.4 rpm). This analysis also indicated a slosh frequency of 1.67 Hz. These results were compared to various predictions (see Reference (5)).

No other information concerning the results of this experiment could be located at this time.

**Key Words:** Fluid Physics, Transparent Liquids, Contained Fluids, Partially Filled Containers, Spinning Containers, Rotating Fluids, Free Surface, Surface Tension, Meniscus Shape, Contact Angle, Container Shape, Fluid Oscillation, Liquid Dynamic Response, Sloshing, Oscillation Amplitude, Oscillation Frequency, Viscosity, Solid/Liquid Interface, Liquid/Gas Interface, Bubbles, Bubble Formation, Tracer Particles, Illumination System, Processing Difficulties

**Number of Samples:** unclear
**Sample Materials:** (1) in cylindrical container: doubly distilled water with iriodine (a tracer); (2) in double torus and thin annulus containers: silicone oil
**Container Materials:** Perspex$^{TM}$

**Experiment/Material Applications:**
See Vreeburg, Spacelab 1.

**References/Applicable Publications:**


(1) Vreeburg, J.P.B. and Vogels, M.E.S.: Liquid Motion in Partially Filled Containers: Preliminary Results of the D-1 Mission. Adv. Space Res., Vol. 6, No. 5, 1986, pp. 85-92. (post-flight)

(2) Vreeburg, J.P.B.: Forced Liquid Moyions in Microgravity. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1 Symposium, Norderney, Germany, August 27-29, 1986, p. 95. (post-flight)

(3) Vreeburg, J.P.B.: Liquid Motions in Partially Filled Containers. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 45-46. (preflight)

(4) Langbein, D.: Fluid Physics. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norederney, Germany, August 27-29, 1986, pp. 93-94 specifically. (post-flight)

(5) Vreeburg, J.P.B.: Results of the Forced Liquid Motion Experiment "FLIM." In Proceedings of the Norderney Symposium on Scientific Results of the Germany Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 112- 117. (post-flight)

(6) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-56. (post-flight; acceleration measurements on D1)

(7) Vreeburg, J.P.B.: Observations on Behavior of Liquid in Weightlessness. In Proceedings of the 30th Anniversary of the von Karmon Institute for Fluid Dynamics, Rhode-St-Genese, Belgium, June 1986. (post-flight)

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**Experiment Origin:** Italy
**Mission:** TEXUS 9
**Launch Date/Expt. Date:** May 1984
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-4 (the fourth version of the TEM-06 dedicated to fluid physics) The module employed a lightsection optic.
**Builder of Processing Facility:** MBB/ERNO, Bremen, Germany

**Experiment:**
Thermal Marangoni Convection in a Floating Zone

This TEXUS 9 experiment was the second in a series of low-gravity investigations designed by Monti and/or Napolitano et al. to study thermal Marangoni convection in a liquid bridge (see Napolitano, Spacelab 1 (this chapter)). The specific objectives of the investigation were to (1) study the unsteady thermal conditions in the liquid bridge and (2) characterize the different thermal and flow regimes at relatively high Marangoni numbers. Measured characteristics (temperature and flow velocity) were to be compared to theoretical calculations.

The experiment was housed in the TEXUS Experiment Module TEM 06-4. The module was used to create a silicone oil liquid bridge between two disks, one of which was heated. Ten thermocouples were used throughout the system: five located within the bridge, one in each disk, two in the disk support, and one measuring ambient temperature. The silicone oil contained tracers for flow determinations. The module employed a lightsection optic and cine camera to observe fluid motion.

At approximately 40 seconds after rocket lift-off, five thermocouples were inserted into the liquid bridge. After another 90 seconds, the upper disk was heated to 90 $^{o}$C at a rate of 1 K/s. During the low-gravity phase of the experiment, three different flow regimes were established and examined: (1) laminar (Stokes and boundary layer), (2) oscillatory, and (3) non-periodic.

(Reference (1) contains discussions of the data reduction techniques and theoretical calculations.) Post-flight examination of the thermal and photographic data indicated that, for a given Prandtl number, the different flow regimes appeared to occur at a particular temperature difference across the zone. It was reported that the laminar regime occurred sometime between t = 130 and 180 seconds after lift-off. From 180 to 320 seconds after lift-off a turbulent motion appeared. After the turbulent motion decayed, a periodic oscillatory flow was established. "The transition from laminar to oscillatory and from this to non periodic 'turbulent' regime occurs at very high Peclet numbers in the heating phase due to the relatively high heating rates and to the thermal unsteadyness[sic] of the process (its effect is difficult to estimate)." (1, p. 232) <Note: See Reference (1) for presentation of thermal data.>

Numerical calculations of the flow agreed well with the observed velocities during the laminar flow portion of the experiment (Peclet number = $1.8 \times 10^5$). However, for t > 170 seconds there was disagreement between the experimental and numerical velocities. There were several possibilities reported for this discrepancy (see Reference (1)), with the most likely cause attributed to turbulence or microscopic oscillations within the flow field.

It was also reported that "...velocities reach[ed] almost a limit just before the onset of turbulence [which] may be indicative of a laminar to turbulent transition in the boundary layer near the free surface. A possible explanation for the oscillations first appearance is that turbulence was established (when the Reynolds number reaches a critical value) and that the turbulence trigger[ed] the oscillations through the coupling of the temperature field. The surface Reynolds number corresponding to the maximum axial velocity that appear[ed] to prevail just before the oscillations is Rey 10." (1, p. 233) <Note: "Rey" is Reynolds number.>

The thermal data acquisition and heat transfer analysis were difficult. "The failure of the... [thermocouple measuring the temperature of the upper disk] during flight caused many difficulties in the heat transfer data reduction and in the numerical correlation." (1, p. 233)

**Key Words:** Fluid Physics, Transparent Liquids, Liquid Bridges, Free Surface, Surface Tension, Thermal Gradient, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Oscillatory Marangoni Convection, Steady to Oscillatory Convective Transition, Time Dependent Thermocapillary Flow, Critical Marangoni Number, Liquid/Gas Interface, Boundary Layer, Flow Velocity, Turbulent Flow, Stokes Flow, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Tracer Particles, Containerless Processing Applications, Optics Assembly, Hardware Malfunction

**Number of Samples:** one
**Sample Materials:** Silicone oil (viscosity = 5 cS) with tracer particles (1.5 mg/$cm^3$, average diameter = 150 microns)
**Container Materials:** not applicable; disk material: copper (Cu*)

**Experiment/Material Applications:**
See Martinez, TEXUS 10 (Chapter 2) or Napolitano, Spacelab D1 (this chapter).

**References/Applicable Publications:**

(1) Monti, R., Napolitano, L. G., and Mannara, G.: Texus Flight Results on Convective Flows and Heat Transfer in Simulated Floating Zones. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 229-236. (post-flight)

(2) Monti, R. and Mannara, G.: TEXUS Experiment on the Convective Heat Transfer Induced by Marangoni Flows. 25th Congress of the International Astronautical Federation, October 7-13, 1984, Lausanne, Switzerland, IAF-84-144. (post-flight)

(3) Langbein, D.: Fluid Physics. In Scientific Results of the German Spacelab Mission D1, Norderney Symposium, Norderney, Germany, August 27-29, 1986, pp. 93-104. (post-flight; briefly mentions TEXUS 9 experiment)

(4) Thermal Marangoni Convection in a Floating Zone. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 66-67. (post-flight)

(5) Input received from R. Fortezza, August 1993.


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**Experiment Origin:** Italy
**Mission:** TEXUS 14a
**Launch Date/Expt. Date:** May 1986
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-4 (the fourth version of the TEM-06 dedicated to fluid physics): float zone apparatus equipped with light band optics
**Builder of Processing Facility:** MBB/ERNO, Bremen, Germany

**Experiment:**
Thermal Marangoni Convection in a Floating Zone

This TEXUS 14a experiment was the fourth in a series of low-gravity investigations designed by Monti and/or Napolitano et al. to study thermal Marangoni convection in a liquid bridge (see Napolitano, Spacelab 1, Spacelab D1; Monti, TEXUS 9 (all in this chapter)).

Very little information concerning the specific objectives and setup of the TEXUS 14a experiment could be located. The Co-Investigator reported the following:

"The experiment conceived was similar to the one already flown on TEXUS 9 but was interactively controlled from [the] ground. The cinecamera[sic] of TEM-06-04 was replaced by CCD, to receive real-time images from the facility. The thermocouple comb, used to scan the temperature in the liquid bridge, was motorized to change the tips during the flight. The experiment execution (automatic during TEXUS 9) was controlled from the block house... [at the launch site] by the Investigators. <Note: CCD was not defined. Further, it is not clear what "tips" were changed in the thermocouple comb.>

"73 seconds after the lift-off, the two disks,... [(outer diameter= 18 mm)] were separated (l=18 mm) and the liquid was injected to form the liquid bridge between them. Due to an... [unexpected] "wobbling motion" caused by a failure in the rocket control, the residual acceleration on board [the rocket] was much higher than expected, and the silicone oil injected flowed out of the support, without forming the liquid bridge. During the flight the telecontrol capabilities were successfully tested." (3)

The experiment was reflown on TEXUS 14b (see Monti, TEXUS 14b).

**Key Words:** Fluid Physics, Liquid Bridges, Transparent Liquids, Free Surface, Surface Tension, Thermal Gradient, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Liquid Expulsion Through a Small Orifice, Liquid Injection, Liquid Transfer, Liquid/Gas Interface, Acceleration Effects, Rocket Motion, Processing Difficulties

**Number of Samples:** one
**Sample Materials:** Silicone oil (5 cS). <Note: it is not clear if tracer particles were added to the oil.>
**Container Materials:** Not applicable; disk material: copper (Cu*)

**Experiment/Material Applications:**
See Napolitano, Spacelab D1.

**References/Applicable Publications:**
(1) Experimentalmodul TEM 06-4. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, p. 79. (in German; discusses experiment module)

(2) Experimentelle Nutzlast und Experimente TEXUS 14. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 53-55. (in German; post-flight)

(3) Input received from Co-Investigator R. Fortezza, August 1993.

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**Experiment Origin:** Italy
**Mission:** TEXUS 14b
**Launch Date/Expt. Date:** May 1987
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-4 (fourth version of the TEM-04 dedicated to fluid physics): float zone apparatus - modified post TEXUS 14a to include one additional remote command line for control of the temperature gradient.
**Builder of Processing Facility:** MBB/ERNO, Bremen, Germany

**Experiment:**
Thermal Marangoni Convection in a Floating Zone

This TEXUS 14b experiment was the fifth in a series of low-gravity investigations designed by Monti and/or Napolitano et al. to study thermal Marangoni convection in a liquid bridge (see Napolitano, Spacelab 1, Spacelab D1; Monti, TEXUS 9, TEXUS 14a (all in this chapter)). The specific objective of the experiment was to investigate the onset of Marangoni oscillations under different thermal conditions.

The experiment was performed in the TEXUS Experiment Module TEM 06-4. A silicone oil liquid bridge, anchored between two copper disks, could be created in the apparatus. The oil contained glass tracers for flow velocity determination. The system was equipped with a camera which allowed real-time control of the experimental process during the sounding rocket flight. A "comb" of thermocouples, which could be moved during the experiment by operators on the ground was placed within the liquid. A fluxmeter adjacent to each of the copper disks was used to determine heat flux.

"The experiment was fully controlled from Italy, using Telescience approach. Satellite link was used to transmit the video images from... [the launch site]; digital data and commands were transferred using ground leased lines." (5)

At the beginning of the experiment, the "upper" disk was heated with a predetermined temperature ramp. The onset of oscillation occurred 210 seconds after lift-off at a heater ramp of 0.4 $^{\circ}C/s$

and a disk temperature difference of 28.5 $^{o}$C. (The onset of oscillation was determined via real-time video and thermocouple data down-links.) The heater was then switched off 15 seconds after the onset of oscillations and remained off until the oscillations completely decayed (350 seconds after lift-off).

The experimental sequence was then repeated with a different upper disk temperature ramp. During this second sequence, the onset of oscillation occurred after the heater was switched on (t = 350 seconds) at a heater ramp of 0.5 $^{o}$C/s and a disk temperature difference of 33.2 $^{o}$C. Reportedly, the low-gravity phase of the rocket ended before complete decay of the oscillations was observed.

A previous experiment (TEXUS 9) indicated that the onset of oscillation is governed by (1) surface velocity and (2) free surface conditions. Therefore, for this TEXUS 14b experiment, the onset of oscillation was correlated with the dynamic Weber number by defining this in terms of the surface velocity and characteristic capillary velocity. A critical transition parameter was defined as the square root of twice the Weber number which was equated with the ratio of the surface velocity to the capillary velocity (see Reference (4) for further details).

The experimental data were also used to determine the nondimensional value of the heat transfer rate (defined as the ratio of the heat flux through the lower disk to the purely conductive heat transfer through a quiescent liquid). However, "The measure of the experimental heat flux by the upper fluxmeter was affected by an error due to the presence of a copper cylinder (necessary to hold the tracers holding screen) across the insulator (glue+mylar) layer." (2, p. 85) Therefore, the nondimensional heat transfer was evaluated utilizing only the lower fluxmeter (see Reference (2) for analysis).

**Key Words:** Fluid Physics, Liquid Bridges, Transparent Liquids, Free Surface, Surface Tension, Thermal Gradient, Heat Transfer, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Oscillatory Marangoni Convection, Conduction, Liquid/Gas Interface, Flow Velocity, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Liquid Injection, Hydrodynamics, Capillary Forces, Tracer Particles, Containerless Processing Applications

**Number of Samples:** One liquid bridge, two oscillation regimes.
**Sample Materials:** Silicone oil (5 cS) with Echosphere glass tracer particles (outer diameter= 100-125 microns)
**Container Materials:** Not applicable; disk material: copper (Cu*)

**Experiment/Material Applications:**
See Napolitano, Spacelab D1.

**References/Applicable Publications:**

(1) Experimentmodul TEM 06-4. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, p. 79. (in German; discusses experiment module)

(2) Monti, R. and Fortezza, R.: Experimental Results on Microgravitational Fluid Dynamics During TEXUS Program. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 80-88.

(3) Monti, R.: On the Onset of the Oscillatory Regimes in Marangoni Flows. In Acta Astronautica, Vol. 15, Number 8, 1987, pp. 557-560. (post-flight; discusses conclusions from TEXUS, Spacelab 1, and Spacelab D1 experiments)

(4) Experimental Results on Microgravitational Fluid Dynamics. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 68-73. (post-flight)

(5) Input received from Co-Investigator R. Fortezza, August 1993.


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**Experiment Origin:** Japan
**Mission:** STS Launch #13, STS-017 (STS 41-G, Challenger)
**Launch Date/Expt. Date:** October 1984
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** NASA Get Away Special (GAS) Canister G-032
Volume of Canister: 5 cubic feet
Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-032:** Nippon Electric Company (NEC), Yokohama, Japan/Asahi National Broadcasting Company, Tokyo, Japan
**Processing Facility:** A blower used to launch small metal and plastic balls. <Note: Although additional elements of the processing facility were not detailed in the available publications, it is suspected that the payload must have also been equipped with a water ball generator and a water heater.>
**Builder of Processing Facility:** Unknown, possibly Nippon Electric Company, Yokohama, Japan

**Experiment:**
Physics of Solids and Liquids (Water Ball Collision)

This experiment was one of four investigations housed within the G-032 Get Away Special Canister during STS-017. The three other experiments are applicable to this data base and are listed under Asahi, STS-017, Chapter 14, "(1) Indium-Antimony Crystal Production, (2) Lead Zinc Alloy Production, and (3) Glass Composite Production."

The "Physics of Solids and Liquids" experiment was the first in a series of investigations designed by Asahi National Broadcasting Company, Ltd. et al. to study the surface tension of water in a reduced gravity environment.

During the space experiment, "...the collision of a small solid ball projected toward a water ball..." (3, p. 2-2) was to be investigated via video images of the impact.

Reference (4) indicated that the experiment was "unsuccessful" and that the payload had to be repaired before it was reflown on the later STS-023 mission (see Asahi, STS-023, GAS G-035). Based on Reference (5), it appears that the "unsuccessful" nature of the experiment was due to the unexpected freezing of the water to be used in the experiment.

No other publications could be located that further described the G-032 experiment setup or space flight anomalies.

**Key Words:** Fluid Physics, Drops, Solid/Liquid Collision, Drop Formation, Droplet Injection, Liquid Expulsion Through A Small Orifice, Droplet Collision, Impact Velocity, Drop Oscillation, Drop Dynamics, Liquid Dynamic Response, Surface Tension, Free Surface, Solid/Liquid Interface, Thermal Environment More Extreme Than Predicted, Hardware Malfunction, Processing Difficulties, Sample Not Processed As Planned

**Number of Samples:** unknown
**Sample Materials:** solid plastic and metal spheres, water
**Container Materials:** not applicable

**Experiment/Material Applications:**
Reference (2) notes that such collision investigations are of interest to nuclear physicists. The experiments are a macroscopic model of the reaction of atomic nuclei.

**References/Applicable Publications:**
(1) NASA Space Shuttle Mission 51-D Press Kit, April 1985, p. 17. (very brief preflight summary)

(2) Fujimoto, K.: Water Ball Collision. In NASA Goddard Space Flight Center The 1985 Get Away Special Experimenter's Symposium, pp. 81-84. (discusses 51-D results)

(3) STS-41-G Cargo Systems Manual: GAS. JSC-17645 Annex Final Version, NASA JSC, September 4, 1984.

(4) STS-51D Cargo Systems Manual: GAS. JSC-17645 Annex 7 Final Version, NASA JSC, March 20, 1985.

(5) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, the NASA GAS Team, 1989, p. 22. (post-flight; very brief description)

(6) NASA Space Shuttle Mission 41-G Press Kit, October 1984, p. 23. (preflight)

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special Canister mission history)

(8) Shuttle Payload Creates Space Sculpture. Aviation Week and Space Technology, October 15, 1984, p. 21. (post-flight)

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**Co-Investigator(s):** Fujimoto, K. (2), Goans, M. (GSFC Technical Manager) (3)
**Affiliation(s):** (1) Tokyo, Japan; (2) Asahi National Broadcasting Company, Ltd., Tokyo, Japan; (3) National Aeronautics and Space Administration (NASA), Goddard Space Flight Center (GSFC), Greenbelt, Maryland

**Experiment Origin:** Japan
**Mission:** STS Launch #16, STS-023 (STS 51-D, Discovery)
**Launch Date/Expt. Date:** April 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** NASA Get Away Special (GAS) Canister G-035
Volume of Canister: 5 cubic feet
Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-035:** Nippon Electric Company (NEC), Yokohama, Japan/Asahi National Broadcasting Company, Tokyo, Japan
**Processing Facility:** A water ball generator and a solid ball launching facility. Heaters were added to the payload to prevent the water from freezing.
**Builder of Processing Facility:** Unknown, possibly Nippon Electric Company, Yokohama, Japan

**Experiment:**
Physics of Solids and Liquids (Water Ball Collision)

This experiment was one of four investigations housed within the G-035 Get Away Special Canister during STS-023. The three other experiments are applicable to this data base and are listed under Asahi, STS-023, Chapter 14, "(1) Indium-Antimony Crystal Production, (2) Lead-Zinc Alloy Production, and (3) Glass Composite Production."

The "Physics of Solids and Liquids" experiment was the second in a series of investigations designed by Asahi National Broadcasting Company, Ltd. et al. to study the surface tension of water in a reduced gravity environment.

During the low-gravity investigation, 4 mm diameter stainless steel balls were projected toward a 16 mm diameter water ball. Preliminary ground-based experiments had indicated that projection rates varying from 50-1050 mm/second would allow the observation of three different surface-tension related phenomena: (1) the ball passing through the water completely, (2) the ball passing into the water and remaining captured, and (3) the ball failing to break the surface tension of the water. Twenty experiments were completed; fourteen of which gave interesting results.

Collisions and projection rates were recorded by video camera.

Analysis of the video images indicated that at projection rates of 50-300 mm/s, the steel balls appeared to break into the water ball, but bounced back and revolved around the surface of the water ball. At a projection rate of 380 mm/s, the steel ball broke into the water ball, but again revolved around the water ball's surface. "At a rate of 690 mm/second or more, the stainless steel balls passed through the water ball. At the rate of 900 mm/second... [the] water ball, given high energy by the colliding stainless steel ball, shivered vigorously, and when the stainless steel ball left the water ball, a portion of water was separated from the water ball, to form two small water balls. One of the daughter water balls flew away in the direction $90^{\circ}$ to the direction in which the stainless steel ball left." (2, p. 83)

Other observations were detailed in Reference (2).

**Key Words:** Fluid Physics, Drops, Solid/Liquid Collision, Drop Formation, Droplet Injection, Liquid Expulsion Through A Small Orifice, Droplet Collision, Impact Velocity, Drop Oscillation, Drop Dynamics, Liquid Dynamic Response, Free Surface, Surface Tension, Solid/Liquid Interface

**Number of Samples:** twenty
**Sample Material:** stainless steel spheres; water
**Container Materials:** not applicable

**Experiment/Material Applications:**
Please see Asahi, "Physics of Solids and Liquids," STS-017.

**References/Applicable Publications:**
(1) NASA Space Shuttle Mission 51-D Press Kit, April 1985, p. 17. (very brief preflight summary)

(2) Fujimoto, K.: Water Ball Collision. In NASA Goddard Space Flight Center's 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, pp. 81-84, NASA CP-2401. (post-flight)

(3) Cargo Systems Manual: GAS; Annex for STS 41-G. JSC-17645 41-G, NASA JSC, September 4, 1984.

(4) Cargo Systems Manual: GAS; Annex for STS 51-D. JSC-17645 51-D, NASA JSC, March 20, 1985.

(5) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, The NASA GAS Team, 1989, p. 25. (short description; post-flight)

(6) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special Canister mission history)

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**Co-Investigator(s):** Orton, G. (Payload Manager) (2)
**Affiliation(s):** (1) St. Louis, Missouri; (2) McDonnell Douglas Astronautics Company, St. Louis, Missouri

**Experiment Origin:** USA
**Mission:** STS Launch #13, STS-017 (STS 41-G, Challenger)
**Launch Date/Expt. Date:** October 1984
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** NASA Get Away Special (GAS) Canister G-074
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-074:** McDonnell Douglas Astronautics Company, St. Louis, Missouri
**Processing Facility:** Fluid motion was to be observed on stainless steel, mesh-screen baffles inside a Plexiglas™-and-aluminum test tank assembly.
**Builder of Processing Facility:** McDonnell Douglas Astronautics Company, St. Louis, Missouri

**Experiment:**
Zero G Fuel System Test: (A) Surface Tension Propellant Tank Test Test and (B) Inertia-Driven Propellant Transfer Demonstration

Propellant tanks, designed to use the surface tension of the fuel to move or maintain the liquid at an appropriate position within the tank, will be employed on future satellites and spacecraft.

The overall objective of this STS-017 Get Away Special (GAS) experiment was to (1) investigate the use of advanced surface-tension, propellant-acquisition concepts in the design of propellant tanks, and (2) demonstrate the bubble-free delivery of fuel (from a partially filled propellant tank) to the spacecraft engines. More specifically, the experiment was designed to demonstrate "...an off-load capability for a full-tank propellant acquisition system, a capability that does not exist with current full-tank surface tension devices such as the Shuttle RCS propellant acquisition system.... [In the Shuttle RCS system], the gallery legs must be kept full of propellant to ensure gas-free propellant delivery to the engines. This is not possible when the tank is launched with a partial propellant load because, uncovered, the forward gallery leg screens are not able to retain propellant during launch acceleration. The g-0074 experiment... [was] designed to solve this problem by demonstrating passive gallery fill following orbit insertion. In addition,... [the experiment was expected to] demonstrate a passive refillable trap concept that could be used in an orbit maneuver propulsion system to provide multiple engine restart capability." (4, p. 87)

<Note: A figure detailing the Shuttle RCS Propellant Acquisition Device is pictured in Reference (4), p. 88.>

The experimental hardware included (1) a test tank, (2) a movie camera, (3) photographic lighting, and (4) an auxiliary liquid accumulator. The experimental test tank was constructed from a cylindrical section of Plexiglas™. At one end of the cylinder, an aluminum "forward" dome was attached; at the other end of the cylinder, an aluminum "aft" end plate was attached. "The tank is divided into forward and aft compartments by an internal Plexiglas bulkhead. The three gallery legs in the forward compartment... [were made] of Plexiglas to allow visual (movie) evaluation of passive gallery fill. Each gallery leg... [had] a flat, stainless steel screen surface along its outer face (adjacent to the tank wall) and a vent screen inside the forward vent baffle assembly. The internal bulkhead assembly... [contained] a tapered Plexiglas vent stack to provide an exit passage for entrapped gas during passive fill of the aft trap compartment. The vent stack... [was covered] by two perforated plate discs at its forward end." (4, p. 88) The gallery leg dimensions, screen sizes, etc. are presented in Reference (4).

The tank operating sequence was also presented in Reference (4). "The tank will be launched with a partial liquid load (Freon 113 [dyed blue for photographic purposes]) in the forward compartment and a nearly empty aft compartment. During the zero-g interval following main engine cutoff, the three gallery legs will fill by capillary pumping. The baffle assembly at the forward end of the tank keeps the gallery vent screens dry until the gallery fill process is complete....

"After the first Orbital Maneuvering Subsystem (OMS) burn, additional Freon 113 will be injected into the tank forward compartment from an auxiliary positive displacement accumulator in preparation for the trap filling experiment.... The trap filling experiment will be performed during the second OMS engine firing. The transfer valve at the tank outlet will be signaled open to allow liquid flow from the forward to aft compartment. During the burn, gas inside the aft compartment will be expelled through the vent stack perforated plates by the hydrostatic pressure imposed across the entrapped gas bubble, allowing the aft compartment to fill. The aft compartment fill rate is retarded by liquid flow pressure losses through the transfer line and valve and by gas flow pressure losses through the vent stack perforated plates." (4, p. 92)

<Note: The reader is referred to Reference (4) for figures of the Plexiglas™ tank configuration and the pictorial illustration of the expected fluid configuration during the experiment.>

<Note: Although the above experiment summary was written based on information contained within a document published prior to the STS-017 launch (Reference (4)), Co-Investigator G. Orton verified that the information related in this document was correct for the actual payload configuration.>

It appears that the experiment was to be activated when the noise of the shuttle lift-off was recognized by acoustic sensors on the payload. However, while G-074 was being stored (prior to shuttle lift-off) in a building of Kennedy Space Center, a Delta rocket was launched. Reportedly, the noise associated with the Delta launch prematurely activated the experiment. Thus, the experiment was unexpectedly performed on the ground and not during the shuttle launch. Subsequent low-g flight tests onboard the NASA KC-135 aircraft based at Ellington Field, Texas, demonstrated successful operation of the experiment.

**Key Words:** Fluid Physics, Contained Fluids, Partially Filled Containers, Propellant Tanks, Propellant Transfer, Refueling in Orbit, Fluid Management, Liquid Reservoir, Liquid Transfer, Gas-Free Liquid Expulsion, Surface Tension, Hydrostatic Pressure, Capillary Flow, Capillary Forces, Baffles, Liquid/Vapor Interface, Solid/Liquid Interface, Acceleration Effects, Launch-Induced Fluid Motion, Illumination System, Bubbles, Engine Re-Start, Sample Not Processed As Planned

**Number of Samples:** one experiment setup
**Sample Materials:** Freon 113 (dyed blue for photographic purposes)
**Container Materials:** The experimental test tank was constructed of Plexiglas™ with aluminum endcaps.

**Experiment/Material Applications:**
This research is applicable to future propellant tank design and, in particular, to more versatile, lower costing surface tension propellant acquisition approaches.

**References/Applicable Publications:**
(1) Cargo Systems Manual: GAS Annex for STS 41-G, JSC-17645 41-G, September 4, 1984. (preflight; short description)

(2) NASA Press Kit, STS-13, p. 24. (preflight)

(3) Shuttle Payload Creates Space Sculpture, AW&ST, October 15, 1984. (very short description)

(4) Orton, G. F.: Experiment G-074 -- Off-Load Capability for a Full-Tank Propellant Acquisition System Put in Space. In NASA Goddard Space Flight Center's Get Away Special Experiment Symposium, August 1-2, 1984, pp. 87-94, NASA CP-2324. (preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(6) Input received from Co-Investigator G. Orton, August 1993.

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**Co-Investigator(s):** None
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**Experiment Origin:** USA
**Mission:** STS Launch #15, STS-020 (STS 51-C, Discovery)
**Launch Date/Expt Date:** January 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Middeck Experiment, Joint Venture
**Processing Facility:** Storable Fluid Management Demonstration (SFMD)
**Builder of Processing Facility:** Martin Marietta Astronautics, Denver, Colorado

**Experiment:**
Storable Fluid Management Demonstration (SFMD)

Continued operation of spacecraft (e.g., space station) will require periodic, on-orbit re-supply of propellants. Elimination of the need to return spacecraft to Earth for re-supply, followed by spacecraft re-launch, will result in significant cost savings. However, on-orbit re-supply of propellants presents problems which are not encountered on Earth. For example, several gravity-independent fluid phenomena will become dominant in the low-g fluid transfer (e.g., wetting and capillary forces).

The objectives of this STS-020 experiment were to (1) demonstrate the refilling of a tank under low-gravity conditions, (2) demonstrate the expulsion of a tank by a surface tension device, (3) measure the expulsion efficiency, and (4) investigate static and dynamic fluid behavior during orbiter reaction control maneuvers.

A subscale, transparent supply tank and receiver tank were used for the demonstration. The tanks were located in the middeck of the orbiter and mounted to NASA-provided adapter plates. The tanks were configured in a space normally occupied by four mid-deck storage lockers. The receiver tank (12.5 inches in diameter) was located in the upper module of the experiment device. "A [multi-compartmented] surface tension type propellant acquisition device that provided gas-free liquid expulsion and control of liquid motion, was installed in the [receiver] tank. This surface tension device is representative of the devices currently being built for spacecraft that will require on-orbit resupply." (1, p. 1) (Details of the surface tension device, tank construction, pressurization system, and plumbing can be located in Reference (1).) The supply tank, located in the lower module, contained (1) water with various additives and (2) an elastomeric diaphragm for expulsion and refill. A cylinder mounted on the front of the lower module allowed measurement of residual fluid after expulsion of the receiver tank. The amount of fluid was determined by recording the position of a piston located in the cylinder.

"A test began with the receiver tank empty, the supply tank completely full, the cylinder partially filled, and the position of the piston in the cylinder was noted. Following filling and expulsion of the receiver tank, the change in the piston position indicated how much liquid was left in the receiver tank." (1, p. 2) The receiver tank was evacuated to remove all non-condensable gasses. This left a vapor behind which condensed as the pressure increased. One test was performed without evacuation with the tank partially filled.

A total of nine tests were performed over a period of 2 days (8 hours per day). Data collection consisted of (1) a video tape of the receiver tank with comments from the astronaut, (2) still photos at selected times, (3) notes of the test procedures, (4) gage readings, and (5) acceleration measurements using a self-contained data recording system.

Post-flight analysis of the available data indicated that "Successful filling of the receiver tank and the multi-compartmented surface tension device was demonstrated in every test. Following the fill, the surface tension device was always able to resume gas-free expulsion of the liquid. After all tests were completed only a few small gas bubbles could be observed in the supply tank, indicating that expulsion was predominantly gas-free throughout all the tests." (1, p. 4)

Tests concerning the expulsion of the fluid demonstrated that the surface tension device was capable of expelling the fluid from the receiver tank with only small residuals. This was shown over various ranges of flow rate and with both pulsed and steady flow. Gas-free flow was observed in a sight flow indicator in the outlet line. Expulsion efficiency was observed to be 93% to 94% compared to the predicted 92%. The increase in measured performance was attributed to "...some emptying of the internal volume of the channels (all of which was predicted to be residual) and better draining of the liquid surrounding the device." (1, p. 5)

Experiments were performed to measure liquid response to orbiter maneuvers. It was reported that the ambient acceleration was $2 \times 10^{-4}$ g with frequent peaks of $5 \times 10^{-4}$ g. The vernier thrusters produced accelerations of $1 \times 10^{-3}$ g and the primary thrusters produced accelerations of $1 \times 10^{-2}$ g. "Lateral accelerations applied at liquid levels within the compartmented section caused displacement of bubbles inside the compartments, with no liquid flow through the barriers.

The motion was highly damped. Bubbles in the lower dome of the tank became distorted when the larger magnitude acceleration was applied, but then resumed their spherical shape and slowly drifted about. The rotational yaw maneuver caused a slow drifting of the bubbles in the lower dome, with centripetal force positioning the liquid." (1, p. 5)

It was concluded that the Storable Fluid Management Demonstration was successful. The filling tests demonstrated that a tank containing a complex surface tension device can be refilled. The expulsion tests confirmed the modeling methods for the operation of the device and the behavior of the liquid.

The experiment was reflown on STS-53 and STS-57 (under the name FARE 1 and FARE 2) with different tank and surface tension device configurations. <Note: Details of these two later experiments is beyond the scope of this technical memorandum (TM). This information will be reported in later versions of this TM.>

**Key Words:** Fluid Physics, Propellant Transfer, Refueling in Orbit, Orbital Tankers, Liquid Transfer, Fluid Management, Contained Fluids, Partially Filled Containers, Transparent Liquids, Flow Rates, Baffles, Free Surface, Solid/Liquid Interface, Liquid/Vapor Interface, Interface Shapes, Gas Ingestion, Gas-Free Liquid Expulsion, Bubble Removal, Liquid Reservoir, Wetting, Wetting of Container, Capillary Forces, Surface Tension, Bubbles, Acceleration Effects, Acceleration Measurements, Sloshing, Fluid Motion Damping, Liquid Dynamic Response, Condensation, Piston System, Vacuum

**Number of Samples:** one demonstration unit
**Sample Materials:** Water with additives including: (1) Triton X-100 (a wetting agent), (2) Dow Corning FC-10 (an antifoam ingredient), (3) iodide (an antibacteria agent), and (4) blue food coloring (for observation purposes)
**Container Materials:** Tank: acrylic plastic (Plexiglas™); screen: stainless steel

**Experiment/Material Applications:**
Water was selected as the test fluid for safety considerations. Triton X-100 was added to the water as a wetting agent (because water does not wet surfaces well while propellants do). The Dow Corning FG-19 anti-foam agent was added to reduce foaming properties of the wetting agent. Iodine was added to prevent bacterial growth, and blue coloring was added to enhance visibility.

**References/Applicable Publications:**

(1) Tegart, J. and Kirkland, Z.: On Orbit Propellant Resupply Demonstration - Flight Results. AIAA/SAE/ASME/ASEE 21st Joint Propulsion Conference, July 8-10, 1985, Monterey, California, 5pp., AIAA Paper #85-1233. (post-flight)

(2) Kirkland, Z. and Tegart, J.: On-Orbit Propellant Resupply Demonstration. In AIAA/SAE/ASME 20th Joint Propulsion Conference, June 11-13, 1984, Cincinnati, OH, AIAA Paper 84-1342. (preflight)

(3) Tegart, J.: Storable Fluid Management Demonstration-Flight I. Project D-02R, Low G Fluid Behavior and Control, Progress Report, December 1985, Martin Marietta, R85-48602-001. (limited public distribution, available from author)

(4) Input received from Experiment Investigator, September 1988 and August 1993.

(5) Hill, M. E. and O'Malley, T. F.: A Summary of Existing and Planned Experiment Hardware for Low-Gravity Fluids Research. AIAA 29th Aerospace Sciences Meeting, Reno, Nevada, January 7-10, 1991, pp. 14-15, (also NASA TM-103706). (post-flight)

(6) Tegart, J.: Shuttle Middeck Fluid Transfer Experiment-Lessons Learned. In Microgravity Fluid Management Symposium, NASA CP-2465, proceedings of a symposium held at NASA Lewis Research Center, Cleveland, Ohio, September 9-10, 1986, pp. 217-224. (post-flight)


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**Experiment Origin:** USA
**Mission:** STS Launch #17, STS-024 (STS 51-B, Spacelab 3: Challenger)
**Launch Date/Expt. Date:** April 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Spacelab Rack
**Processing Facility:** Geophysical Fluid Flow Cell (GFFC)
**Builder of Processing Facility:** Aerojet, Azusa, California

**Experiment:**
<u>Geophysical Fluid Flow Cell (GFFC)</u>

Large-scale atmospheric motions on rotating planets and stars are influenced by both buoyancy and Coriolis forces. On a spherical surface the Coriolis force varies with latitude, and this variation is thought to be a fundamental influence on a wide range of phenomena of interest to geophysicists and astrophysicists. These phenomena include cloud-band orientation in planetary atmospheres, differential rotation in the solar atmosphere, the formation of jet-streams, and magnetic dynamo action. Except for flows that are homogeneous, so that buoyancy driven motions are absent, it has been impossible to properly model even the simplest global motions in the terrestrial laboratory. When the rotation vector is inclined to Earth's gravity g an oscillatory body force results that has no geophysical analog. Therefore, previous laboratory experiments on global circulations have been confined either to: (1) studies of convection near the poles where the rotation vector and g are nearly parallel and the geometry is effectively flat, or (2) studies of motions close to the equator where the rotation vector and g are nearly perpendicular, with the effects of gravity being simulated by centrifugal buoyancy forces associated with very rapid rotation.

The specific objective of this Spacelab 3 experiment was to study the interaction of rotation and convection similar to that which occurs in the atmosphere of a rotating planet like Earth or Jupiter.

A radially directed electrostatic "gravitational" field, which varied from parallel at the poles to perpendicular at the equator, was made possible in reduced gravity by applying an alternating high voltage across the inner and outer boundaries of a hemispherical shell filled with dielectric silicone oil. A system of computer-controlled heater coils and thermistor sensors maintained radial and latitudinal temperature gradients on the shell. As the shell rotated, these gradients generated thermally-driven circulations. The convective flow fields in the hemisphere were visualized via Schlieren and shadowgraph photography.

Reportedly, "A variety of [thermal] convective structures, or planforms, were observed, depending on the magnitude of the rotation and the nature of the imposed heating distribution." (5, p. 31) For example, when rotation was the dominating parameter, north-south oriented columnar convection was observed. "As the differential heating is increased these rolls interact with the mid-latitude waves, ultimately being destroyed by turbulent, horizontally-isotropic convection that moves down from the pole. When a large equator to pole temperature difference is imposed on the boundaries, spiral waves develop on top of a strong meridional circulation." (1, p. 519)

Experimental results were compared to numerical simulations. Reportedly, "The results are in agreement with numerical simulations that can be conducted at modest parameter values, and suggest possible regimes of motion in rotating planets and stars." (5, p. 31)

A more lengthy discussion of the experimental results and numerical simulations can be found in each of the references listed below.

**Key Words:** Fluid Physics, Contained Fluids, Spinning Containers, Rotating Atmospheres, Planet Rotation, Rotating Fluids, Transparent Liquids, Heat Transfer, Buoyancy-Driven Convection, Turbulent Flow, Coriolis Forces, Thermal Gradient, Thermal Convection, Electric Field, Atmospheric Applications, Shells, Schlieren Viewing

**Number of Samples:** one hemispherical shell
**Sample Materials:** silicone oil (0.65 centistoke viscosity)

**Container Materials:** inner shell fabricated from polished nickel; outer shell fabricated from transparent sapphire (approximately 1 cm thick)

**Experiment/Material Applications:**
Simulation of planetary atmospheres is achieved by producing rotating, spherical convective flows with effectively radial gravity fields induced by dielectric polarization forces.

**References/Applicable Publications:**
(1) Hart, J. E., Glatzmaier, G. A., and Toomre, J.: Space-Laboratory and Numerical Simulations of Thermal Convection in a Rotating Hemispherical Shell with Radial Gravity. Journal of Fluid Mechanics, Vol. 173, 1986, pp. 519-544. (post-flight results)

(2) Hart, J. E., and Toomre, J.: Geophysical Fluid Dynamics with Dielectric Polarization Forces., Rep. 2, NASA Contract NAS-8-31958, 1977, 66 pp.

(3) Hart, J. E., Toomre, J., Deane, A. E., Hurlburt, N. E., Glatzmaier, G. A., Fichtl, G. H., Leslie, F., Fowlis, W. W., and Gilman, P. A.: Laboratory Experiments on Planetary and Stellar Convection Performed on Spacelab 3. Science, Vol. 234, October 1986.

(4) Leslie, F. W.: Geophysical Fluid Flow Experiment. In Research and Technology 1987, Annual Report of the Marshall Space Flight Center, NASA, pp. 62-63. (short description)

(5) Hart, J. E., Toomre, J., Deane, A. E., Hurlburt, N. E., Glatzmaier, G. A., Fichtl, G. H., Leslie, F., Fowlis, W. W., and Gilman, P. A.: Laboratory Model of Planetary and Stellar Convection. Spacelab 3 Mission Science Review. Proceedings of a symposium held at NASA George C. Marshall Space Flight Center, Alabama, December 4, 1985, NASA CP-2429, pp. 31-41. (See also p. 5 for short summary) (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #18, STS-025 (STS 51-G, Discovery)
**Launch Date/Expt. Date:** June 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Commercial version of the West German Get Away Special (GAS) MAUS Canister; NASA Getaway Special (GAS) Canister Designation G-025: Alternate Designation EMTE 1
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay (attached to the Payload Support System (PASS). "PASS is a standardized structure, power supply and data processing unit available commercially to GAS Users." (2, p. 39)
**Primary Developer/Sponsor of G-025:** MBB/ERNO, Bremen, Germany
**Processing Facility:** A hemispherical tank filled with fluid and subjected to acceleration inputs.
**Builder of Processing Facility:** Unknown, possibly: Messerschmitt Boelkow-Blohm (MBB-ERNO), Bremen, Germany

**Experiment:**
Liquid Sloshing: Dynamic Behavior of Liquid Propellants

When spacecraft attitude control motors are fired, both (1) the vehicle and (2) liquid propellant held within the fuel storage tank, accelerate. The dynamic response of the fluid in the tanks coupled with the tank design determines the distribution of the fluid within the container. The storage tanks are often designed to use the surface tension of the stored liquid to move or maintain the liquid at the appropriate position within the tank. Propellant must be adequately positioned within the tank for several reasons including (1) motor fuel intake and (2) vehicle stabilization.

The objectives of this STS Get Away Special (GAS) canister experiment included (1) determining the low-gravity, dynamic behavior of a partially filled hemispherical tank via experimental methods, (2) correlating the experimentally-obtained data to mathematical models designed to describe similar dynamic behavior, and (3) evaluating the outflow of the liquid in the test tank through a tube and solenoid-valve and into an evacuated aluminum chamber, (particularly investigating "...the orientation and stabilization of the fluid (propellant) under the influence of a propellant management device (PMD) especially with respect to outflow cases.") (4, p. 166)

<Note: A specific description of the PMD was not presented in any of the available references.>

"Specific aims [of the experiment] were:

o definition of all significant natural frequencies and damping of the fluid in the low frequency range of 6 Hz;

o determination of the generalized propellant mass, to facilitate the necessary analysis assumptions;

o determination of sloshing forces and the pressure distribution in the fluid and tank;

o observation of the dynamic behavior of the propellant under quasi zero-gravity conditions;

o observation of the fluid orientation stability effect, including the shape of flow during critical operation phases." (4, p. 167-168)

The experimental apparatus was designed to simulate fluid sloshing within a partially-filled satellite liquid propellant tank. A transparent, hemispherical tank contained the test liquid. Details of the tank design were not presented in any document published after the experiment was actually performed. Two documents published prior to the shuttle flight (Reference (2) and Reference (9)) detailed the expected experiment setup. It is clear, however, that the overall experiment design was changed somewhat from the preliminary description provided in the references. For example, the earlier system (1) was to be excited up to 10 Hz (whereas the actual experiment was excited up to 6 Hz) and (2) was to perform four different experiment phases (whereas the actual experiment performed five different phases). Because there is no other source available which describes the setup, the preliminary design concept is summarized here.

"A hemispherical plexiglas tank is suspended in a rocking mechanism such that purely linear, nearly frictionless oscillation can be induced within a limited displacement range. A stepping motor is connected to the tank rocking mechanism by means of a variable stiffness coupling (with a spring). Pressure sensors, force sensors, temperature sensors and accelerometers are located at selected positions on the experiment assembly: certain phases of the experiment will be recorded with a high speed camera.

"The mechanical assembly is locked down during launch, landing and during non-operational periods in orbit. During operation the stepping motor is driven according to a pre-determined program and generates a variety of known excitation profiles comprising shock waves of different forms and sinusoidal sweeps from 0,2 to 10 Hz. The coupling stiffness will be switched in accordance with the experiment run program. In addition, the amount of liquid in the tank can be varied by transferring a portion of it to an evacuated container. The system decay dynamics will be investigated over a range of different natural frequencies." (2, p. 41)

Additional details concerning the drive train, transmission coupling system, and tank support assembly can be reviewed in Reference (2) on pages 42-43.

"Besides the hemispherical test tank, initially 50% filled with fluid, a second evacuated aluminum container is provided, connected to the test tank by a tube and solenoid valve. Outflow of propellant from the test tank will be simulated by opening the valve at specific times." (2, p. 43)

"The major portion of the active operations is assigned to data production by transducers and sensors. The time allowed for filming is very limited, because the high speed used rapidly consumes the film magazine; hence only critical fluid phases are filmed where sensor data cannot provide an adequate picture of the fluids behavior." (2, p. 48)

Reportedly, one of the critical fluid phases to be filmed was "...the influence of the PMD in assuring correct fluid orientation and stability, and the response of the fluid to simulated thruster propellant demand. A source of diffuse light is provided for illumination of the fluid." (2, p. 44)

A discussion of the four expected phases of the experiment was provided: "In phase 1 of the experiment... the mechanical hold-down locks will be released, thus freeing the moving assemblies. The propellant tank receives an impulse to settle (pre-orientate) the liquid propellant. A short time will be allowed for the system to return to test [probably should be rest] and then an additional period allowed for the liquid to stabilize itself. The liquid behavior during the impulse and decay period will be recorded on film....

"Phase 2
In this phase the propellant tank is subjected to a variety of sinusoidal oscillations. Overall, the vibrations will sweep the frequency range 0.2 Hz to 10 Hz in three intervals: 0.2 Hz to 1

Hz in 20 incremental steps; 1 Hz to 5 Hz in 40 steps; 5 Hz to 10 Hz in 50 steps. The amplitude of the oscillations will be small. This procedure will be repeated six times with different amplitude settings and different spring tension settings on the rocker assemblies....

"Phase 3
The experiment run continues with the generation of shock waves (pulses). Following the input of a pulse to the propellant tank, the drive train will be disconnected and the moving mass allowed to decay naturally. A stabilization period enables the liquid in the tank to take up its natural configuration.

"This procedure is carried out nine times with various settings for pulse duration, amplitude and spring stiffness settings, following which all the steps are executed once again. A single pulse input is followed by a decay period of 10 seconds, during which data is generated and a stabilization period of 5 minutes.

"Phase 4
Phase 4 begins with a propellant expulsion operation, in which the tank is emptied to the 30% fill level. The liquid is transferred to an evacuated container, the flow being limited by a solenoid valve. For this operation the camera is switched on to provide visual data of propellant management characteristics under fuel demand conditions. This phase continues with a repetition of the pulsed excitation program carried out in phase 3, following which the propellant tank is emptied completely. This operation will also be filmed. The dynamic characteristics of the empty tank system are then further investigated with a set of shock inputs.... (2, pp. 48-49)

Reference (9) (which was written prior to Reference (2)) presented a more detailed description of the expected design of the test tank. The reader is again reminded that the final experiment configuration may have differed considerably from this description.

The test tank "...is built out of a circular glass cylinder of 10 cm diameter and a length of 10 cm. The glass cylinder is closed on [the] bottom and top sides by aluminum plate...." (9, p. 566) A detailed description of the tank surface coatings and the role of these coatings was also presented in the reference.

It was reported that the test fluid was to be Methylenbromid. Its "good surface properties" were detailed in Reference (9). Please refer to Reference (9) for a detailed description of the expected measurement system.

<Note: This ends the summary of the expected experiment setup. The information which follows describes the actual experiment during the flight and actual post-flight analysis.>

During the low-gravity shuttle mission, the tank was "...subjected to oscillatory disturbances corresponding to inputs used for theoretical simulation and analysis." (4, p. 166)

Each of the five experimental phases had a specific operating mode and objective. It appears (from Reference (4)) that prior to the start of any of these phases the tank had to be in a state of stationary equilibrium. Therefore, time slots (time that the experiment was most likely operationally inactive) were programmed into the experiment sequence to allow for the fluid stabilization. During the over 3 hours the experiment ran, 40% of this time was dedicated to this fluid stabilization. Transducers and sensors documented the majority of the fluid behavior. Critical phases of the fluid dynamics were captured photographically. (The amount of photographic data was limited because "...the high speed employed rapidly used up the film." (4, p. 173)

At the time Reference (4) was written, extensive post-flight analysis of the transducer, sensor, and photographic data was not complete. However, preliminary analysis indicated that the "experiment functioned perfectly." Further, "In principal, the underlying mathematical models developed by MBB/ERNO for the design of surface tension tanks have been supported by the experiment results, and the validity of current product design features has been confirmed. The results will provide additional insight into the effect of fluid sloshing on spacecraft attitude control systems." (4, p. 173)

<Note: Reference (3), which only very briefly mentions this experiment, reported that "This ERNO experiment has been flown on two previous shuttle missions to analyze the behavior of liquid propellants in satellite tanks." However, no further information concerning these other experiments could be located. Further, none of the other publications that described this STS-025 experiment made any mention of the previous experiments. It may be worthy to note that MBB/ERNO was a sponsor/developer of several experiments in GAS or GAS-type payloads prior to this shuttle mission (see G-026, DG-205I, DG-205II, DG-315, DG-206A, DG-306, DG-200A, and DG-318). However, none of these earlier experiments were related to this experiment.>

<Note: Many of the references listed below were not available when this experiment summary was prepared.>

**Key Words:** Fluid Physics, Contained Fluids, Partially Filled Containers, Propellant Tanks, Liquid Reservoir, Propellant Transfer, Liquid Transfer, Fluid Management, Acceleration Effects, Acceleration Measurement, Liquid Dynamic Response, Fluid Motion Damping, Liquid Stability, Sloshing, Meniscus Vibration, Meniscus Stability, Fluid Oscillation, Oscillation Frequency, Oscillation Amplitude, Surface Tension, Free Surface Shape, Coated Surfaces, Solid/Liquid Interface, Liquid/Gas Interface, Engine Re-Start, Illumination System

**Number of Samples:** one experiment tank
**Sample Materials:** The fluid used during the actual STS experiment was not specified. However, a reference published prior to the shuttle launch indicated that Methylenbromid was to be used. The properties of this fluid are listed in Reference (9).
**Container Materials:** Unknown, possibly glass with aluminum endplates.

**Experiment/Material Applications:**
"The results [of this research] will validate and refine mathematical models describing the dynamic characteristics of tank-fluid systems. This will in turn support the development of future spacecraft tanks, in particular the design of propellant management devices for surface tension tanks." (4, p. 165)

**References/Applicable Publications:**
(1) Cargo Systems Manual: GAS Annex for STS 51-G, JSC-17645 51-G, Rev. A, March 20, 1985. (short description, preflight)

(2) Gilbert, C. R., Netter, G., and Vits, P.: Study of Liquid Sloshing Behavior in Microgravity. In Goddard Space Flight Center's 1984 Get Away Special Experimenter's Symposium, Post Symposium Proceedings (Addendum), 1984, pp. 38-54. (preflight)

(3) Kolcum, E. H.: Fuel Contaminant Threatens Delay in Shuttle Launch. AW&ST, June 17, 1985. (preflight).

(4) Gilbert, C. R.: GAS Payload No. G-025: Study of Liquid Sloshing Behavior in Microgravity. In NASA Goddard Space Flight Center's 1985 Get Away Special Experimenter's Symposium, October 8-9, 1985, pp. 165-176, NASA CP-2401. (post-flight)

(5) STS 51-G Press Kit, NASA Press Release 85-83, June 1985, p. 19. (Preflight)

(6) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, The NASA Gas Team, 1989, p. 28. (post-flight, very brief description)

(7) Netter, G. and Beig, H. G.: Preliminary Results From the EMTE Fuel Slosh Flight Experiment-STS-16. In ESA Proceedings of an International Symposium on Fluid Dynamics and Space, pp. 145-157.

(8) Netter, G. and Eckhardt, K.: Fluid Dynamic Experiment in a Surface Tension Tank: Phase 1/Phase 2A. Final Report, June 1981. MBB/ERNO, Report #BMFT-FB-W-83-002. (in German, English summary)

(9) Eckhardt, K. and Netter, G.: Experiment for Investigation of the Dynamic Behavior of Fluid in a Surface Tension Tank Under Microgravity Condition. Acta Astronautica, 1982, pp. 565-571. (preflight)

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**Experiment Origin:** USA
**Mission:** STS Launch #19, STS-026 (STS 51-F, Spacelab 2: Challenger)
**Launch Date/Expt. Date:** July 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Payload Bay
**Processing Facility:** Dewar system of the small helium-cooled Infrared Telescope (IRT)
**Builder of Processing Facility:** NASA Marshall Space Flight Center, Huntsville, Alabama

**Experiment:**
Small Helium-Cooled Infrared Telescope (IRT)

This Spacelab 2 experiment was a multidisciplinary study involving several scientific and technical goals. Several of the scientific objectives involved measurement and mapping of various diffuse and discrete infrared sources from celestial bodies using a cryogenically cooled telescope and its infrared detectors. Other technological objectives included (1) evaluating the space shuttle as a platform for infrared telescopes and (2) evaluating the performance of a newly designed, large superfluid helium dewar system used for cooling the telescope. The following experiment summary reports on the fluids related aspects of the experiment: the performance of the cooling system.

Reportedly, "An infrared telescope must be cooled to keep its own thermal infrared radiation from overwhelming the radiation from celestial sources it seeks to measure. The cryogenic system for cooling this telescope consists of a 250-liter (65-gallon) superfluid helium dewar. A porous plug is used to restrain the liquid helium and allow the flow of gaseous helium to cool the telescope." (11, p. 33)

The Spacelab 2 Infrared Telescope (IRT) was mounted on a pallet in the shuttle cargo bay (see Reference (5) for telescope description). The cooling system was evaluated by performing controlled tests of its behavior during the final day of the

Spacelab 2 mission. Since the mission was extended an extra day, testing of the helium superfluid storage and flow components lasted about 35 hours.

"The measurements of interest were the functioning of the porous plug phase separator and its relationship to the superfluid bath. Porous plugs employ the thermomechanical or fountain effect, a quantum mechanical phenomenon existing only with superfluid helium, to hold liquid helium within a dewar and permit only vapor to vent. The characteristics to be determined were: (1) whether liquid would flow uncontrollably from the dewar if venting was temporarily stopped or when venting was restarted; (2) whether the helium bath could be warmed until it passed from the superfluid phase into the normal phase, and then recooled to superfluidity in a controlled manner when venting was restarted; and (3) whether heat deliberately applied to the vent side of the porous plug could reverse the fountain effect and draw more helium through the plug." (2, p. 6)

Reportedly, "The IRT vent valves were closed a number of times for various periods; no fluid flow through the plug was detected and venting resumed smoothly when the valves were reopened. On three occasions the superfluid bath was allowed to warm above the [lambda]-point into the normal phase (temperature >2.17K); each time when the vent valves were reopened the bath recooled in a controlled manner. Operation of a small heater on the vent side of the porous plug did not enhance fluid flow through the plug, but instead, forced the liquid helium back through the plug and reduced the flow. This latter result is contrary to the laboratory experience of researchers developing fountain effect pumps for the transfer of liquid helium in space, and must be further studied." (2, p. 6)

<Note: Not all of the publications listed below were available at the time this experiment summary was prepared.>

**Key Words:** Fluid Physics, Superfluids, Cryogens, Contained Fluids, Partially Filled Containers, Fountain Effect, Liquid Transfer, Heat Transfer, Thermal Control, Liquid/Vapor Interface, Solid/Liquid Interface, Two-Phase System, Phase Transition, Porous Plug, Flow Rates, Ventilation, Separation of Components, Infrared Telescope

**Number of Samples:** one cooling system
**Sample Materials:** Cooling fluid: superfluid helium
(He*)
**Container Materials:** aluminum
(Al*)

**Experiment/Material Applications:**
See Experiment section (above).

**References/Applicable Publications:**

(1) Spacelab-2 Infrared Telescope (IRT) Ninety Day Science Report. In Spacelab 2 90 Day Post Mission Science Report, Ed., Eugene W. Urban, NASA Marshall Space Flight Center, November 14-15, 1985, 12 pp. (post-flight)

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(3) Fazio, G. G.: A Small Helium Cooled Infrared Telescope. In Spacelab Mission 2 Experiment Descriptions, NASA TM-78198, Edited by K. Stuart Clifton, September 1978, pp. 15-18. (preflight)

(4) Karr, G. R., Hendricks, J. B., Urban, E. W., Katz, L., and Ladner, D.: Cryogenic Sub-System Performance of the Infrared Telescope for Spacelab 2. In Proceedings of the Eighth International Cryogenic Engineering Conference, Geneva, June 3-6, 1980, Edited by C. Rizzuto, pp. 38-42. (preflight)

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(6) Urban, E. W., and Ladner, D. R.: Infrared Telescope (IRT) System Cryogenic Performance. In SPIE Vol. 509, Cryogenic Optical Systems and Instruments, 1984, pp. 216-232. (preflight)

(7) Hendricks, J. B., Karr, G. R., Urban, E. W., and Ladner, D. R.: The Infrared Telescope on Spacelab 2. Space Helium Dewar Conference, Berlin, 1984, 6 pp. (preflight)

(8) Urban, E. W., Katz, L., Hendricks, J. B., and Karr, G. R.: A Cryogenic Helium II System for Spacelab. IPC Business Press, 1978, IPC Science and Technology Press, Ltd., Westbury House, Bury Street, Guildford, Surrey GU2 5AW, England, pp. 113-119. (preflight)

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(12) Urban, E. W. and Ladner, D. R.: Operation of the Cryogenic System of the Infrared Telescope on Spacelab 2. In Proc. 11th International Cryogenic Engineering Conference (ICEC II), p. 341, 1986.

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(14) Input received from Principal Investigator G. G. Fazio, August 1993.

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**Experiment Origin:** USA
**Mission:** STS Launch #19, STS-026 (STS 51-F, Spacelab 2: Challenger)
**Launch Date/Expt. Date:** July 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Payload Bay (on Spacelab pallet)
**Processing Facility:** Super Fluid Helium Experiment Package (instrumented cryostat and support electronics package)
**Builder of Processing Facility:** Ball Aerospace <Note: The location of the builder is unclear.>

**Experiment:**
Super Fluid Helium Experiment (SFHE)

Superfluid helium possesses several characteristics which make it uniquely suited for cooling space-based instruments. Such instruments include the Infrared Astronomical Satellite (IRAS) and the Space Infrared Telescope Facility. Liquid helium 4 (He 4) and its rare isotope, helium 3, remain liquid at absolute zero. At a temperature of 2.17 K and a vapor pressure of 5.1 kiloPascals (38.4 torr), He 4 undergoes a transformation to a superfluid state. In this state, He 4 can transport large amounts of heat at very small temperature differences. Heat is transported by coherent wave motion rather than by diffusion. Its effective thermal conductivity is several orders of magnitude higher than any other material. The high thermal conductivity is maintained in thin films and pores so small that a normal liquid would be immobilized. The advantage of this property is that the thin films, held to walls by van der Waals forces, are superfluid. Therefore, the entire superfluid mass behaves as a single thermal mass with a temperature difference of a few milli-Kelvins.

Under certain conditions, superfluid helium has zero effective viscosity whereas the viscosity for bulk motions is not zero (about 1/100 of water). "In space, the location of the bulk liquid is determined by the weak surface tension forces, and as a result, there will be large motions of the liquid in response to small acceleration forces." (6, p. 5) Therefore, the natural damping of these motions is limited and the resulting forces (caused by the fluid movement) may disturb the instruments cooled by the helium.

Another unique characteristic of superfluid helium is known as the fountain effect. "In small pores or thin films, the application of a small temperature gradient along the pores sets up a pressure differential in the opposite direction tending to push the liquid to the warmer end. This fountain pressure is used in zero gravity to contain the liquid in the cryostat, while allowing the vapor created by heat flow into the bulk helium to evaporate to space. A porous plug is placed in the vent line.... The outer end is cooled by the evaporation of liquid to gas, and the resulting temperature differential generates a pressure which keeps the liquid in the tank.

"It is of practical interest to know whether such a liquid-vapor separator can recover from a condition in which there is liquid on the down stream side and the... temperature gradient is in the wrong direction to maintain the fountain pressure." (6, p. 3)

The behavior of superfluid helium has been described by the two-fluid model of Tisza (see Reference (7)). Here, the fluid is assumed to have two components: (1) superfluid and (2) normal liquid. "The superfluid component is in a single macroscopic quantaum-mechanical ground state and as a result has no viscosity, zero entropy, and cannot transport. The fraction of the liquid in the superfluid state is uniquely determined by the temperature... and approaches 100% at absolute zero. The remainder of the fluid acts like a normal liquid with viscosity, entropy, and the capacity to carry heat.

"Transport mechanisms are pictured as relative motions of the two components. These motions change the local ratio of superfluid to normal fluid and hence are accompanied by temperature changes. Third[-]sound,... is a surface wave on films so thin that the normal component is locked to the substrate by its viscosity. The superfluid component moves as a surface wave, i.e. a change in depth, accompanied by a change in temperature. The restoring force for these waves may be van der waals forces, surface tension and in one g, gravity. In addition, there is a tendency of the liquid to flow from the hotter valleys to the colder peaks. Both the flow and the distillation act as effective restoring forces." (6, p. 3)

This Spacelab 2 experiment was designed to investigate the properties of superfluid helium under low-gravity conditions. There were several technological and scientific objectives of the (SFHE) which was divided into three separate investigations:

(1) The Quantized Surface Wave (QSW) experiment had scientific and technological objectives. Scientifically, the QSW experiment investigated third-sound surface waves, on the order of a micron thick, in films of superfluid helium. Technologically, the ob-

jectives of the QSW experiment were to (a) demonstrate the technology required to observe third-sound waves in films thicker than possible under 1-g conditions and (b) determine the wave velocities and attenuation.

(2) The objective of the Bulk Fluid Dynamics (BFD) experiment was to determine the response (slosh modes and decay time) of superfluid helium to known acceleration levels.

(3) The objective of the Bulk Thermal Dynamics (BTD) experiment was to determine temperature fluctuations and variations (to within 10 microKelvin) associated with slosh modes.

The experimental apparatus, mounted on the aft-most pallet in the cargo bay, consisted of three main parts: (1) a cryostat, (2) a flight electronics assembly, and (3) a vacuum maintenance assembly. The cryostat contained a vacuum insulated tank (volume of 120 liters), which could be operated at temperatures as low as 1.5 Kelvin. All experiments were held in a removable sensor head. The sensor head contained all the electronics for the QSW experiment and for interfacing with the external electronics through a multiplexing circuit. The top mounting plate also contained the seal for the helium vessel. Hermetically sealed, multi-pin connectors allowed signal passage through the plate. (See References (1) and (6) for hardware details.)

Results from the three separate experiments are as follows:

Quantized Surface Wave Experiment: It was reported that "The general features of the waves correspond well; the driven response shows the frequency and amplitude behavior predicted by theory. In particular, the direct path (1/4 turn) wave response is nearly constant in amplitude, while the increase in baseline and peak values at the end of the pulse train correlates with the predicted arrival of the long path (3/4 turn) wave.

"Observations were made at a number of temperatures and frequencies and with pulses of different lengths and amplitudes. Perhaps the most unexpected result, in the light of predictions of very high attenuations and measurements on thinner films in one g, was the presence of waves persisting as long as 60 seconds." (6, p. 5)

Bulk Thermal Dynamics Experiment: Data for this experiment were collected by 12 temperature sensors located in a 3-liter cell within the sensor head. A three-axis accelerometer package was also used. The time profile of one the temperature sensors included for this experiment was reported. However, it was reported that "High time-resolution plots and close correlation with input accelerations will be necessary to interpret the data in terms of disturbed and undisturbed temperature fluctuations." (6, p. 5) (No other results from the BTD experiment were reported.)

Bulk Fluid Dynamics Experiment: Data for this experiment were collected by 132 liquid-vapor sensors placed on a tree-like structure located in the same three liter cell used for the BTD experiment. A major goal was to determine the liquid and ullage bubble location and, therefore, the bulk liquid location and its dynamic response to accelerations. It was reported that "The BFD sensors appeared to work well at temperatures close to the lambda point. Useful data were taken during burns of the Orbital Maneuvering System... and during several hours of thruster firings...." (6, p. 6) (No other results from the BFD experiment were reported.)

(Reference (6) contains further discussions of the cryostat performance, space environment (temperature and accelerations), cryogenic operations, and mission operations.)

**Key Words:** Fluid Physics, Superfluids, Cryogens, Contained Fluids, Partially Filled Containers, Thermal Control, Acceleration Effects, Liquid Dynamic Response, Sloshing, Fluid Management, Oscillation Frequency, Oscillation Amplitude, Heat Transfer, Thermal Gradient, Liquid/Vapor Interface, Solid/Liquid Interface, Thin Films, Fountain Effect, Quantized Surface Waves, Porous Plug, Evaporation, Surface Tension, Viscosity, Van der Waals Forces, Bubbles, Acceleration Measurements, Vacuum

**Number of Samples:** not applicable
**Sample Materials:** superfluid helium
(He*)
**Container Materials:** unknown

**Experiment/Material Applications:**
Liquid helium 4 and its rare isotope, helium 3, are uniquely suited for use as a cryogen under low-gravity conditions (see EXPERIMENT SUMMARY INPUT). They are the coldest liquids known and can, therefore, be used as cooling fluids for instruments which require extremely low temperatures.

**References/Applicable Publications:**

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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Process Chamber Double Rack
**Processing Facility:** Holographic Optical Laboratory (HOLOP) (holographic interferometer)
**Builder of Processing Facility:** Designed and built by the Institute of Space Simulation, DFVLR, Cologne, Federal Republic of Germany

**Experiment:**
Bubble Transport by Chemical Waves (PL-HOL-01)

Chemical waves may result in reacting fluid systems that are far from thermodynamic equilibrium. These waves are produced by a locally varying heat source which results in thermal gradients. The gradients propagate in a wave-like manner through the reacting fluid and cause Marangoni flow. The flow may result in a motion of gaseous or liquid inclusions. Under certain conditions, the inclusions can be caught in the waves and thus move in a 'surfing' manner. On Earth, gravity-induced buoyancy disturbs this motion, disallowing any observation of the phenomenon.

This Spacelab D1 experiment was designed to study low-gravity particle transport by chemical waves. The specific objective of the experiment was to investigate the motions of gaseous inclusions in a reacting liquid.

A specially built reaction chamber was incorporated in the Holographic Optical Laboratory (HOLOP) for the Spacelab D1 mission. The reaction chamber had been (1) cut into a copper block to suppress exterior temperature gradients, and (2) gold-coated to prevent unwanted chemical reaction between the wall material and chemical solution.

During the mission, the reaction was to be initiated by drawing two reacting solutions into the chamber (these two solutions were unspecified). The Zhabotinski reaction was selected for (1) wave generation and (2) experiment ease; the gas inclusions were to be generated from Zn particles. <Note: This process was further explained in Reference (7): The micron-sized particles were contained in the evacuated reaction chamber. When the reacting solutions were drawn into the chamber, the Zn dissolved forming gas. (The composition of the reacting solutions was close to the regular Zhabotinski composition.)> Reportedly, the chemical reaction was optimized by selection of an appropriate species concentration. The concentration was selected to produce a temperature oscillation amplitude of 0.045 $^{o}$C and a wavelength of 14 mm. Holograms were to be taken during the experiment. <Note: the Zhabotinski reaction was discussed further in References (4) and (8).>

It was reported that (1) evacuation and filling of the experiment chamber went according to plan, (2) the employed solutions survived the long storage time; therefore, (3) the wave-generating chemical reaction should have progressed as intended. However, "When the recording of holograms started, a malfunction occurred, which has not yet been resolved. After film development, the holograms exhibited different density values and blurred images. There is no continuity in the frequency of exposures. These malfunctions are the more unexplainable since the two follow-up experiments under identical optical conditions resulted in correct exposures. The interpretation of the experimental results is therefore limited...." (4, p. 142)

Reportedly, it was possible to identify 20 bubbles of 0.2 to 3.0 mm diameter (50-100 smaller bubbles were expected). Of these 20 bubbles, seven could be used for velocity determination. The bubble movement fluctuated with a velocity range of 0.1 to 1.0 mm/minute. It was possible that this movement was caused by chemical waves. From this limited data, the following interpretation was reported: (1) the bubble movement was only for a short period of time as the wave passed, (2) the wave direction remained unchanged, and (3) the wave velocity was 2.5 mm/minute which was greater than the bubbles but of the same order of magnitude. It was thus concluded that the experimental results were in approximate agreement with the expected results.

**Key Words:** Fluid Physics, Chemical Waves, Chemical Oscillations, Thermal Oscillations, Thermal Gradient, Marangoni Convection, Reactant Solutions, Zhabotinski Reaction, Gas Formation, Inclusions, Liquid Transfer, Mass Transfer, Liquid Reservoir, Bubbles, Bubble Formation, Bubble Transport, Particle Transport, Buoyancy Effects, Flow Velocity, Biotechnology, Holography, Liquid/Gas Interface, Solid/Liquid Interface, Coated Surfaces, Contained Fluids, Photographic Difficulties

**Number of Samples:** one experiment chamber
**Sample Materials:** The reacting solutions were unspecified, although reportedly, they contained Zn particles for bubble generation
(Zn*)
**Container Materials:** gold-coated copper
(Au*Cu*)

**Experiment/Material Applications:**
"Remarkable and unexpected phenomena can arise in special chemically reacting systems when they are far from thermodynamic equilibrium. The reaction then abandons its unstructured path and may feature chemical oscillation, spontaneous generation of spatial inhomogeneities, or chemical waves. Such phenomena are of fundamental interest and have an important role in biological systems." (1, p. 97)

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**Experiment Origin:** The Netherlands
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Materials Science Double Rack (MSDR)
**Processing Facility:** Fluid Physics Module (FPM)
**Builder of Processing Facility:** FIAT C. R., Italy

**Experiment:**
Marangoni Convection in Relation to Mass Transfer from the Liquid to the Gas Phase (WL-FPM-01) MACO A and MACO B

Purification processes (e.g., distillation, adsorption, desorption) generally involve mass transfer between two immiscible liquids or a liquid and a gas. Columns often used for these processes are filled with packing material (particles) of a particular shape (e.g., rings, spirals) to create a large contact area between the fluids. Generally, the liquid is distributed over the top of the column and flows continuously downward over the packing material. As the liquid flows downward, the gas percolates upward or downward over the wetted particles.

The design of packed columns depends on (1) the wetting efficiency of the particles and (2) the rate of transport of the components to and from the transferring interfacial area. However, because of the non-linear behavior of the mass-transfer process, packed-column design is often not accurate.

The performance of a packed column is measured by the number of transfer units, $N_{og}$, which is proportional to the product of the mass transfer coefficient, K, and the contact area, A. Earlier research had determined $N_{og}$ may be increased by up to a factor of 3 by surface tension effects.

This Spacelab D1 experiment was the first in a series of investigations designed by Drinkenburg to study the influences of Marangoni convection on low-gravity mass transfer rate. The specific objective of the study was to determine the effect of solutally-driven Marangoni convection (without the masking influences of gravity-induced buoyancy) on the K x A product.

Rather than modeling a packed column or purification process, the experiment was configured to study a more basic form of solutally driven fluid transport.

Two experiments, MACO A and MACO B, were scheduled to be performed during the Spacelab D1 Mission. Prior to the flight, two containers (one for MACO A and one for MACO B) were prepared for the investigations. Because each container was to be partially filled with a weak acetone water solution (MACO A: 3 vol% acetone; MACO B: 2 vol% acetone), the glass inside the containers was coated above the expected liquid interface with polyfluorocarbon. This treatment was done to insure that a flat liquid-gas interface was maintained. To keep the acetone concentration in the gas phase constant, an activated carbon bed was included 2 cm above the interface. The activated carbon was expected to absorb the acetone as it evaporated. Three thermistors, attached to the outside of the container, were available to provide thermal data. A pressure transducer, configured on the system, was to provide the gas phase pressure data. (The thermistor and pressure transducer data allow calculation of the acetone distribution coefficient over both phases.) The two experimental containers (1 cm x 10 cm x 3.5 cm) were housed within the Spacelab Fluid Physics Module (FPM) for activation during the mission. The expected convective flow patterns were recorded with a camera and illumination system. <Note: Although not stated, it appears that particles were included in the acetone solution to enhance visualization.>

During the first experiment (MACO A), the 3 vol% acetone-water solution was introduced into the chamber during 1 minute. Within 7 seconds the fluid movement (induced by the filling process) had damped. A flat interface was created which, to the surprise of the ground observers (viewed via direct images from the shuttle) showed no fluid motion. After about 10 minutes the Payload Specialist removed some fluid from the chamber and a curved interface resulted. Small flow cells developed in the middle of the concave interface. After a period of time, two larger cells formed that had a wavelength matching the chamber length.

It was thought that the stability of the flat interface was due to impurities within the MACO A cell. Therefore, the second experiment (MACO B) was performed, employing the 2 vol% acetone solution, and using the same procedure. However, nearly the same results were obtained. The flat interface was stable with six cells forming with the concave interface. Soon, three of these cells grew into one larger cell.

Since extra time was available, a third experiment was performed with the 2 vol% acetone mixture. The chamber was filled in steps until a flat interface was attained. "In each pause, roll cells could develop and transport the impurities, if present, to the side walls. When completely filled, the flat interface, if not globally stable, would show roll cells." (1, p. 358) Unfortunately, no direct images of this extra experiment were recorded.

Three possible explanations of the stability of the flat interface were presented:

(1) The carbon had been saturated and, therefore, could not absorb more acetone. If this were true, then the evaporation would have been halted after about one second. This explanation was dismissed due to the later roll cell activity which made it unlikely that acetone evaporation had stopped so quickly.

(2) Surface impurities may have stabilized the system. The concave interface would have created a larger interface surface, resulting in the impurities moving to the side walls. In the middle of the interface (clean portion) rolls would have developed. Further analysis of the film and acetone should be able to discern if this was the case.

(3) The side walls stabilized the system. "It is already known that the presence of side walls increases the the critical Marangoni number [value at which surface tension driven flow starts], but it was not expected to such an extent as to stabilize the system completely." (1, p. 359)

<Note: Analysis on the mass transfer characteristics of the system had not yet been performed at the time the available references were published.>

**Key Words:** Fluid Physics, Transparent Liquids, Contained Fluids, Partially Filled Containers, Free Surface, Fluid Motion Damping, Surface Tension, Surface Tension-Driven Convection, Cellular Convection, Marangoni Convection, Critical Marangoni Number, Buoyancy Effects Diminished, Liquid Reservoir, Mass Transfer, Liquid Transfer, Solutal Gradients, Liquid/Gas Interface, Free Surface Shape, Interface Stability, Interface Shapes, Evaporation, Contamination Source, Impurities, Thin Films, Coated Surfaces, Hydrophobic Surfaces, Solid/Liquid Interface, Tracer Particles, Illumination System

**Number of Samples:** two experimental setups, three experiment runs
**Sample Materials:** Water-acetone solutions; for MACO A: 3 vol% acetone solution; for MACO B: 2 vol% acetone solution; tracer materials: unknown
**Container Materials:** glass coated with polyfluorocarbon

**Experiment/Material Applications:**
Marangoni convection (flow by surface tension gradients caused by concentration and/or temperature gradients) leads to enhanced mass transfer. The results from this experiment are related to several areas of research including (1) distillation processes, (2) mass transfer in steel making, and (3) application as liquid withdrawal in thin sheets.

See also Lichtenbelt, MASER 1 and Janssen, MASER 2 (both in this chapter).

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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #22, STS-030 (STS 61-A Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Materials Science Double Rack
**Processing Facility:** Fluid Physics Module, (FPM) (same facility as the FPM of Spacelab 1 but with improvements)
**Builder of Processing Facility:** Centro Ricerche, Torino, Italy

**Experiment:**
<u>Separation of Fluid Phases and Bubble Dynamics in a Temperature Gradient (WL FPM 02)</u>

"Consider a gas bubble or a liquid drop [of medium] A placed in a liquid matrix [of medium] B subjected to a temperature gradient. Due to the temperature dependence of the interfacial tension, tangential stresses arise at the particle interface and generate a liquid flow along the particle contour toward increasing interfacial tension.

"This so-called thermocapillary or Marangoni-convection causes a particle motion in the opposite direction toward decreasing interfacial tension....

"The first theoretical and experimental study on Marangoni bubble motion was published by Young,...[et al.]. Young... derived... [an] equation for the steady, thermocapillary migration velocity V of a fluid particle." (1, p. 179)

On Earth, the examination of such drop or bubble motion is restricted to small radii particles (and thus low Marangoni numbers). The particles must be small enough such that buoyancy forces do not control the motion. Further, terrestrial experiments must be performed in fluid systems with low Reynolds numbers. If the Reynolds number is made too large (by a reduction of the viscosity or an increase in the temperature gradient) gravity-driven cellular convection may occur within the system.

This Spacelab D1 experiment was the first in a series of investigations designed by Nähle et al. to study the thermocapillary convective motion of particles in liquids. The specific objectives of the study were to (1) observe thermocapillary convective particle motion in fluid systems with high Marangoni and Reynolds numbers (numbers larger than those possible on Earth), (2) compare the experimental particle velocity data with theoretical predictions, (3) observe hydrodynamic interactions and particle coalescence, (4) demonstrate the use of Marangoni convection for the low-gravity degassing of liquids, and (5) demonstrate the use of Marangoni convection for the separation of liquid/liquid dispersions.

The experiment apparatus, which was affixed to the front plate of the Spacelab Fluid Physics Module (FPM), consisted (in part) of two test cells (each measuring 30 x 30 x 33 mm). The cells were configured one next to the other, and enclosed by anti-reflective quartz windows. These windows allowed filming of the particle motion. Prior to the flight, both cells were filled with silicone oil. One wall of each cell was configured with a cartridge heater to create a hot wall (with a stabilized temperature of about 60 $^oC$). Six thermistors were available to measure the temperature of the cell. Two reservoirs, one containing air and the other bi-distilled water, were contained in the experimental apparatus. "To prevent a pressure rise due to the high thermal expansion coefficient of silicone oil, pressure compensation systems with membranes were provided for both the cells and the reservoirs." (3, p. 388)

Each cell was configured with a rotor. Air or water could be delivered (from the appropriate reservoir) into the silicone oil via hollow needles extending from the rotors. The rotor/needle assembly was capable of both rotary and translatory motion. Prior to experiment initiation, the rotor needles were capped with glass tips to prevent oil creep in through the needle holes. These glass tips were intentionally broken once the rotors were operated. One cell had been dedicated to observe drop motion and that the other cell was dedicated to observe bubble motion.

During the initial experiment run of the Spacelab D1 mission, "...the bubble/drop generator failed and was activated manually three times repeatedly. Due to the short duration of these sequence[s], quasi-steady state conditions could not be reached and only qualitative evaluations from the film recordings could be performed....

"After reconfiguration and visual inspection via the T/V downlink, the experiment was accomplished... [2 days later] almost nominally.... (4, p. 329) The only mentioned difficulty during this later run was a water drop generator malfunction.

Because of this malfunction, no unambiguous thermocapillary motion of the water droplets was observed.

Post-flight analysis of the available bubble data indicated that the "Marangoni numbers achieved exceed[ed] the limitations for terrestrial observations considerably, Ma... [of the bubble] >0.2 and Ma... [of the oil] >30 were evaluated." (3, p. 389) More specifically, "During the experiment thermocapillary bubble motions with corresponding Marangoni numbers up to Ma... [of the oil approximately equal to] 300 or Ma... [of the bubble approximately equal to] 1 and Reynolds numbers up to Re... [approximately equal to] 0.4 were observed...." (7, p. 212)

The experimentally obtained bubble velocities were compared to the theoretical bubble velocities as predicted by Young's equation. The temperature gradient used in the analysis was calculated using the one-dimensional heat conduction equation and using the hot and cold temperatures as measured during the experiment. The analysis indicated that the theoretical and experimental velocities did not indicate good agreement, except for a particle radius of less than 1.1 mm (see Reference (7)). "For small Marangoni numbers the theory of Young et al. agrees with the experimental data." (4, p. 332) For Marangoni numbers exceeding those of terrestrial experiments by one order of magnitude, agreement between theory and experiment was "satisfactory". However, as the Marangoni number increased beyond this order of magnitude, the limit of the application of Young's theory was exceeded. This excedence was reportedly due to the invalidation of the low heat convection to heat conduction ratio assumption.

It was reported that the applicability of Marangoni effects to degassing of a liquid was demonstrated up to the Marangoni numbers given previously (for the air/silicone oil system).

No bubble coalescence was observed during the experiment. Instead, single bubbles touched and stuck together, or clusters of bubbles decomposed. The single bubbles moved according to the temperature gradient.

"Furthermore, no indication was found that the Marangoni-effect can be utilized for the separation of liquid/liquid dispersions." (1, p. 182)

**Key Words:** Fluid Physics, Transparent Liquids, Multiphase Media, Two-Phase System, Liquid/Gas Interface, Liquid/Liquid Interface, Separation of Components, Hydrodynamics, Thermal Gradient, Heat Transfer, Conduction, Surface Tension, Interfacial Tension, Interface Physics, Marangoni Convection, Thermocapillary Convection, Particle Motion, Bubbles, Gas Injection, Bubble Dynamics, Bubble Formation, Bubble Injection, Bubble Motion, Bubble Migration, Bubble Transport, Marangoni Movement (Migration) of Bubbles, Bubble Removal, Particle Coalescence, Buoyancy Effects Diminished, Liquid Reservoir, Liquid Injection, Liquid Expulsion Through A Small Orifice, Liquid/Liquid Dispersion, Drops, Drop Formation, Drop Velocity, Droplet Injection, Collisions, Contained Fluids, Hardware Malfunction, Processing Difficulties, Sample Not Processed As Planned

**Number of Samples:** two experimental cells
**Sample Materials:** silicone oil (AK 100, Wacker Chemie) with normal air bubbles and/or bi-distilled water bubbles
**Container Materials:** aluminum housing, two quartz windows on opposite sides for illumination and observation/recording
(Al*)

**Experiment/Material Applications:**
The specific reasons why the sample fluids were selected were not reported.

See Neuhaus, STS 61-A and Wozniak, MASER 1 (both in this chapter).

**References/Applicable Publications:**

(1) Siekmann, J., Wozniak, G., Srulijes, J., Nähle, R., and Neuhaus, D.: Experimental Investigation of Thermocapillary Bubble and Drop Motion Under Microgravity. In ESA 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 179-182. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Process Chamber Double Rack
**Processing Facility:** Holographic Optical Laboratory (HOLOP) (Holographic Interferometer)
**Builder of Processing Facility:** Designed and built by the Institute for Space Simulation (Institut für Raumsimulation), DFVLR, Cologne, Germany

**Experiment:**
Bubble Motions Induced by Temperature Gradient (PK-HOL 03)

Even in a low-gravity environment, "...bubbles in a liquid can be moved by a temperature gradient. The temperature dependence of the surface tension, that is a physical property of the thin boundary layer between the liquid and the gas, is responsible for the [gravity-independent] motion ([thermocapillary] Marangoni convection)." (3, p. 52)

"Observation of bubble motions in different chemical systems can provide information about processes at the liquid-gas interface. This is done by correlating chemical and physical properties of the system to deviations in the [predicted] bubble velocity....

"Such measurements can achieve sufficient reliability only in a microgravity environment, where... [buoyant] movement is largely eliminated." (5, p. 118)

This Spacelab D1 experiment was the second in a series of investigations designed by Neuhaus and/or Nähle et al. to study the thermocapillary convective motion of particles in a liquid (see Nähle, STS 61-A (this chapter)).

The Spacelab Holographic Diagnostic System HOLOP was used to examine the behavior of bubbles in three different liquid/gas systems: (1) AK100 silicone oil/air, (2) AS100 silicone oil/air, and (3) AP100 silicone oil/air. The apparatus consisted of three cylindrical, plastic cells, each containing one liquid/gas system. A heater was placed at one end of the liquid/gas system and a heat sink was placed at the other end. This configuration allowed a maximum temperature difference of 3 K between the two ends. During the 45-minute experimental run, 57 holograms were obtained. (The holograms were used for determining bubble velocity determinations.)

Post-flight examination of the reconstructed holograms revealed that the bubble behavior in the three systems was very different. The bubble motion in the AK100 oil followed theoretical predictions. The bubble velocity in the AS100 oil were 40% less than predicted. There was no bubble motion observed in the AP100 oil. Thus, it appeared that the differences between the three oils are not accounted for by the parameters in the theory.

Corresponding experiments with buoyancy driven bubble motion (on Earth) indicated the bubbles AK100 and AS100 move with predicted velocities. However, the bubbles in the AP100 oil, which exhibited no Marangoni-driven motion during the Spacelab D1 experiment, behaved as spheres with a stiff surface.

The above described results "...suggest that the description of the liquid/gas interface using only the surface tension and its temperature dependence... is insufficient. Besides the volume shear viscosity an additional dissipative mechanism at the liquid/gas interface is necessary. The present results suggest dilatational surface viscosity as an additional parameter...." (4, p. 243) <Note: Reference (4) contains a theoretical treatment of the dilatational surface viscosity.>

**Key Words:** Fluid Physics, Transparent Liquids, Two-Phase System, Multiphase Media, Liquid/Gas Interface, Thermal Gradient, Surface Tension, Heat Transfer, Conduction, Marangoni Convection, Thermocapillary Convection, Bubbles, Bubble Motion, Bubble Migration, Bubble Transport, Marangoni Movement (Migration) of Bubbles, Buoyancy Effects Diminished, Interface Physics, Viscosity, Holography, Contained Fluids

**Number of Samples:** three
**Sample Materials:** three types of silicone oil-Wacker Chemie AK100, AS100, AP100; air for gas bubbles
**Container Materials:** plastic

**Experiment/Material Applications:**
The silicone oils employed in this experiment were selected because (1) their low surface tensions reduce their affinity for disturbing impurities, and (2) the chemical differences between the oils are such that "...the ratio of two different radicals of the silicon oil molecule is changed." (4, p. 241)

**References/Applicable Publications:**

(1) Neuhaus, D.: Bubble Motions Induced by a Temperature Gradient. Naturwissenschaften, 73.Jahrgang Heft 7, July 1986, pp. 348-349. <Note: This paper is the same as the entry below for Abstracts of the D1 Symposium.> (post-flight)

(2) Neuhaus, D.: Bubble Motions Induced by a Temperature Gradient. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 98-100. (post-flight)

(3) Neuhaus, D.: Bubble Motions Caused by Temperature Gradients. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 52-53. (preflight)

(4) Neuhaus, D. and Feuerbacher, B.: Bubble Motions Induced by a Temperature Gradient. In 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 241-244. (post-flight results)

(5) Neuhaus, D. and Feuerbacher, B.: Bubble Motions Induced by Temperature Gradient. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 118-120. (post-flight)

(6) Langbein, D.: Fluid Physics. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 93-104. (post-flight)

(7) Wanders, K., Klein, H., Bewersdorff, A., and Neuhaus, D.: Holographic Experiments: Results of Spacelab D1. In Proceedings of the 6th European Symposium on Material Sciences under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, February 1987, pp. 105-107. (post-flight)

(8) Neuhaus, D.: Blasenbewegungen- Hervorgerufen Durch Temperaturgradienten. In Forschung unter Schwerelosigkeit, BMFT-DFVLR Statusseminar, Friedrichshafen, Kongresszentrum, Graf Zeppelin Haus, February 24-26, 1988, pp. 103-108. (in German)

(9) Input received from Principal Investigator D. Neuhaus, July 1993.

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**Co-Investigator(s):** Birman, B. (Program Manager) (3), General Electric Space Division, (Sponsor and Contributor) (4)
**Affiliation(s):** (1,2) During STS 61-C: Pennsylvania State University, University Park, Pennsylvania, Currently: Unknown; (3) General Electric Space Division, Philadelphia, Pennsylvania; (4) Philadelphia, Pennsylvania

**Experiment Origin:** USA
**Mission:** STS Launch #24, STS-032 (STS 61-C, Columbia)
**Launch Date/Expt. Date:** January 1986
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment; NASA Get Away Special (GAS) Canister G-062
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay (on the standard NASA GAS Bridge Carrier System)
**Primary Developer/Sponsor of G-062:** Pennsylvania State University, University Park, Pennsylvania/General Electric Space Division, Philadelphia (Valley Forge), Pennsylvania
**Processing Facility:** The expected experiment configuration was to consist of a sealed pressure vessel containing (1) a liquid droplet injector, (2) a moving piston, and (3) retaining foam.
**Builder of Processing Facility:** Unknown

**Experiment:**
Terminal Velocity Experiment

This experiment was one of three investigations housed within the G-062 Get Away Special Canister during STS-032. The two other experiments (of the three) were also applicable to this data base (see Kedzierski, STS-032 (Chapter 15); Thames, STS-032 (this chapter)). <Note: References (2) and (3) listed a fourth experiment in the canister: "Liquid Droplet Heat Radiator Experiment." However, it appears that this fourth experiment was canceled prior to the shuttle flight and that the canister was configured with only three experiments.>

The terminal velocity of a liquid is defined as the "...highest speed a liquid particle can strike a hard surface without breaking apart." (1, p. 3) This velocity can be used to determine the surface tension of the liquid as the velocity and surface tension are directly proportional.

The objective and expected experimental setup of this investigation was described in a document released prior to the shuttle flight:

"This experiment will determine the terminal velocity and hence the surface tension of two liquids in space by injecting droplets into the path of a moving piston, which will strike the droplets and send them to a special retaining foam.... This procedure will be repeated in... [the] sealed pressure vessel with the piston moving slightly faster each time, until the droplets begin to break up upon impact (i.e. when the terminal velocity has been reached). The impacts will be recorded photographically by a 35 mm Nikon camera. This will yield the piston's speed at [the] time of impact. By comparing the results from a one-G simulation, with all other factors constant, the effect of gravity on surface tension may be determined and hence help quantify surface tension formulae." (1, p. 3)

Reference (3) briefly indicated that either the canister or the G-062 experiments <it is unclear which> "Failed to turn on." The reference further noted that "The batteries tested fine after return to earth. Suspect a relay or controller problem." (3, p. 37)

<Note: It is unclear what batteries tested fine after the return of the shuttle. The only other reference to "batteries" in the available publications was in Reference (1) and is of note. "The electrical power will be supplied by an array of Gates Lead-Acid cells. These cells were chosen due to their consistent safety characteristics in space, a prime concern of NASA. NASA has imposed a three month waiting period prior to launch, which seriously affects the power capacity at launch. Due to these and other factors, low power CMOS circuitry was used throughout the design." (1, p. 5)

It is suspected that because of the above mentioned anomaly (a turn-on failure), no experimental data were obtained.>

No further information describing the experimental setup, suspected experiment timeline, failure analysis, or experimental results could be located at this time.

**Key Words:** Fluid Physics, Terminal Velocity, Surface Tension, Surface Tension Determination, Drops, Liquid Expulsion Through a Small Orifice, Droplet Injection, Solid/Liquid Collision, Droplet Collision, Drop Dynamics, Liquid Dynamic Response, Impact Velocity, Drop Velocity, Solid/Liquid Interface, Piston System, Hardware Malfunction, Sample Not Processed As Planned

**Number of Samples:** The number of drops to be injected: unknown
**Sample Materials:** fluid: unknown
**Container Materials:** retaining foam material: unknown

**Experiment/Material Applications:**
This research was performed to determine the effect of gravity on liquid surface tension and "...hence quantify surface formulae." (1, p. 3)

**References/Applicable Publications:**
(1) Thames, M., Moul, D., and Birman, B.: Penn State Get Away Special G-62. In NASA Goddard Space Flight Center's 1984 Getaway Special Experimenter's Symposium, August 1-2, 1984, NASA CP-2324, pp. 1-8. (preflight)

(2) NASA Space Shuttle Mission 61-C Press Kit, December 1985, p. 16. (preflight; very short description)

(3) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

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**Co-Investigator(s):** Bieber, J. (2) a team of Aerospace Engineers (3), Birman, B. (Program Manager) (4)
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**Experiment Origin:** USA
**Mission:** STS Launch #24, STS-032 (STS 61-C, Columbia)
**Launch Date/Expt. Date:** January 1986
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment
NASA Get Away Special (GAS) Canister G-062
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay (on the standard NASA GAS bridge carrier system)
**Primary Developer/Sponsor of G-062:** Pennsylvania State University, University Park, Pennsylvania/General Electric Space Division, Philadelphia (Valley Forge), Pennsylvania
**Processing Facility:** The expected experiment configuration was to consist of (1) two conispherical tanks and (2) a spin table.
**Builder of Processing Facility:** Students at Pennsylvania State University, University Park, Pennsylvania

**Experiment:**
Liquid Sloshing Experiment

If propellant-tank liquid sloshing should occur in a spin-stabilized satellite, (1) energy of the vehicle would be dissipated and (2) wobbling motion about the spacecraft's spin axis could occur.

This experiment was one of three investigations housed within the G-062 Get Away Special canister during STS-032. The two other experiments (of the three) are also applicable to this data base (see Kedzierski, STS-032 (Chapter 15); Rice, J., STS-032 (this chapter)). <Note: References (2) and (3) listed a fourth experiment in the canister: "Liquid Droplet Heat Radiator Experiment." However, it appears that this fourth experiment was canceled prior to the shuttle flight and that the canister was configured with only the three mentioned experiments.> The specific objective of the research was to measure slosh forces and observe slosh motion in a model satellite propellant tank.

The experimental apparatus was designed to simulate the COMSAT INTELSAT IV spinning communications satellite. (Liquid slosh, previously experienced on the COMSAT, hindered the very precise pointing accuracy of the communication relay.) A document released prior to the shuttle flight described the expected experimental setup:

"The liquid slosh experiment will simulate the INTELSAT IV satellite undergoing an attitude maneuver. Two conispherical tanks, manufactured from clear acrylic and [partially] filled with an aqueous solution, will be mounted on a spin table rotating at about 180 rpm (a spin rate which will provide dynamic similitude between the model and prototype systems). The table will then be impulsively tilted and the resulting slosh motion will [be] analyzed and recorded. Two modified World War II vintage [P-38] gun cameras will visually record the slosh motion at a rate of 16 frames/sec. The lighting will be provided by an array of LED's, which necessitate the use of KODAK's extended red sensitivity Tech-pan 2415 film. This film... has an ESTAR polyester base to survive vacuum exposure and will be a... unique test in itself of photography in space." (1, p. 4)

Slosh forces were to be measured by high resolution piezoelectric force transducers (which had to also withstand the g-forces of the shuttle launch). It was expected that "The simulation will be repeated for three different fill ratios at three different spin rates. This will provide a photographic record of the actual slosh motion and an analog record of the associated forces for nine attitude corrections." (1, p. 4)

Reference (3) (which was published after the return of the shuttle) briefly reported that either the canister or the G-062 experiments <Note: it is unclear which> "Failed to turn on." The reference further noted that "The batteries tested fine after return to earth. Suspect a relay or controller problem." (3, p. 37) <Note: It is unclear what batteries tested fine after the return of the shuttle. The only other reference to "batteries" in the available publications was in Reference (1) and is of interest: "The electrical power will be supplied by an array of Gates Lead-Acid cells. These cells were chosen due to their consistent safety characteristics in space, a prime concern of NASA. NASA has imposed a three month waiting period prior to launch, which seriously affects the power capacity at launch. Due to these and other factors, low power CMOS circuitry was used throughout the design." (1, p. 5)

Due to the above mentioned anomaly (a turn-on failure), no experimental data were obtained.

No further information describing the experimental setup, suspected experiment timeline, or failure analysis could be located at this time.

**Key Words:** Fluid Physics, Aqueous Solutions, Contained Fluids, Partially Filled Containers, Container Shape, Propellant Tanks, Free Surface, Surface Tension, Spin-Stabilized Satellites, Spinning Containers, Rotating Fluids, Sloshing, Fluid Management, Liquid Dynamic Response, Acceleration Effects, Solid/Liquid Interface, Liquid/Gas Interface, Vacuum, Hardware Malfunction, Sample Not Processed As Planned

**Number of Samples:** one setup, three runs
**Sample Materials:** aqueous solution
**Container Materials:** clear acrylic

**Experiment/Material Applications:**
This research is related to liquid propellant sloshing in spin-stablized satellites. More specifically, the research sought to model the liquid slosh created in the COMSAT INTELSAT IV communications satellite as the vehicle underwent altitude maneuvers.

Reportedly, "This experiment will do much to help assess and quantify a very important problem in spacecraft dynamics." (1, p. 4)

**References/Applicable Publications:**
(1) Thames, M., Moul, D., and Birman, B.: Penn State Get Away Special G-62. In NASA Goddard Space Flight Center's 1984 Getaway Special Experimenter's Symposium, August 1-2, 1984, pp. 1-8, NASA CP-2324, pp. 1-8. (preflight)

(2) NASA Space Shuttle Mission 61-C Press Kit, December 1985, p. 16. (preflight; very short description)

(3) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(4) Input received from Principal Investigator M. Thames, July 1993.

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**Co-Investigator(s):** Yoel, D. (Payload Manager and Builder of Hardware) (2), Students from The High School for Engineering (3)
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**Experiment Origin:** USA
**Mission:** STS Launch #24, STS-032 (STS 61-C, Columbia)
**Launch Date/Expt. Date:** January 1986
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** High School Student Experiment
NASA Get Away Special (GAS) Canister G-332
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay (carried on the standard NASA GAS Bridge Carrier System)
**Primary Developer/Sponsor of G-332:** Booker T. Washington High School, Houston, Texas/ The High School for Engineering, Houston, Texas
**Processing Facility:** Heated fluid chamber
**Builder of Processing Facility:** Get Away Special Services, currently located in Moore Park, California

**Experiment:**
Fluid/Air Interaction Experiment

This experiment was one of two investigations housed within the G-332 Get Away Special Canister during STS-032. The other experiment in the canister is not applicable to this data base.

Very little information could be located which described the objectives and setup of this fluid/air interaction experiment. A document published prior to the launch of the STS-032 mission described the expected experimental scenario:

This "fluid physics experiment... will examine the behaviour of fluid when heated in microgravity. As the fluid chamber is heated to cause temperature gradients, readings and photographs will be taken at predetermined intervals." (1, p. 17)

A document published post-flight reported that the experiment was not performed during the mission. "The interface connector for the Orbiter harness was wired incorrectly: pins B and C were connected instead of A and B. The Controller turned on but the batteries were not connected to the experiments." (2, p. 38)

No further information concerning this experiment appears to be available.

**Key Words:** Fluid Physics, Liquid/Gas Interface, Thermal Gradient, Thermocapillary Convection, Partially Filled Containers, Contained Fluids, Sample Not Processed As Planned

**Number of Samples:** unclear, probably one
**Sample Materials:** unknown
**Container Materials:** unknown

**Experiment/Material Applications:**
Unspecified.

**References/Applicable Publications:**

(1) Space Shuttle Mission 61-C, NASA Press Kit, December 1985, p. 17. (preflight)

(2) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

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**Experiment Origin:** The Netherlands
**Mission:** MASER 1
**Launch Date/Expt. Date:** March 1987
**Launched From:** Esrange, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Fluid Science Module (FSM)
**Builder of Processing Facility:** Saab Space, AB, Linköping, Sweden

**Experiment:**
Three-dimensional Marangoni Convection

This MASER 1 experiment was the second in a series of investigations designed by Lichtenbelt and/or Drinkenburg et al. to study concentration-driven Marangoni convection in a reduced gravity environment (see Drinkenburg, STS 61-A (this chapter)). The specific objective of the experiment was "...to visualize Marangoni convection flows coupled to a mass transfer from a liquid to a gas." (9, p. 74) The experiment was one of three investigations performed within the MASER Fluid Science Module (FSM) during the mission (see also Wozniak, MASER 1 (this chapter) and Aalto, MASER 1 (Chapter 15)).

One experiment cell within the FSM was dedicated to this Marangoni convection investigation. The cell, which had a 4-cm inner diameter and was 4-cm deep, contained a dilute (3 vol-%) acetone-water solution and two 4-mm diameter "gaspipes."

Reportedly, just after the low-gravity phase of the mission had been attained, air flow between the two pipes created a single gas bubble of 5-mm diameter in the liquid between the gaspipes. A short time after the bubble was created, the acetone began to evaporate into the bubble. Using the gaspipes and an air-pump/air-filter system in a closed loop, a minimum amount of air flow through the gas bubble was maintained. This airstream ventilated the bubble and maintained a low concentration of acetone within the bubble.

As the evaporation of the acetone occurred, an acetone concentration gradient was created near the acetone-bubble (or gas-liquid) interface. Subsequent Marangoni convection, generated by concentration-induced surface tension differences, was filmed using a television-camera/mirror assembly. It appears that the assembly was configured to obtain a front and a top view of the bubble. Tracer particles within the acetone, coupled with a specialized lighting system, allowed visualization of the flow. Real-time data from the television camera was transmitted to the ground via an S-band video link.

It was reported that the experiment "performed well." Two distinct types of flows were observed during the low-gravity phase of the flight. "Mainly large roll cells were formed [in the bulk solution] with dimensions of up to 15 mm...." (6, p. 367) Four roll cells could be seen in the available two-dimensional picture of the flow pattern (refer to Reference (9) for a drawing of the flow pattern). "Later on, a totally different behaviour was observed around a stable bubble; liquid flow first occurred from the gas outlet toward the gas inlet along the bubble... and then decreased and stopped completely." (9, p. 74)>

It was noted that gas flow velocity highly affected experiment results. If the air flow was small, a high concentration gradient occurred: "...the acetone concentration in the gas increases along the bubble and generates concentration and thus surface tension gradients at the liquid surface (lower surface tension near the outlet pipe.)" (9, p. 74) If the air flow was large, very little concentration gradient resulted: "...the acetone concentration is approximately zero in the bubble and the convection pattern originates from Marangoni instabilities." (9, p. 74)

<Note: The apparatus did not have the capability to actively vary air flow. It appears that the gas flow velocity values fluctuated because of unexpected liquid in the gas circuit. "The flow of the gas through the bubble was not measured during flight. Besides it could not be reconstructed on earth because more than once some liquid was present in the gas circuit disturbing the flow rate. From the time rate of bubble creation... an impression of the flow rate... [could be estimated]. Certainly the flow rate... [was] small enough such that tangential stresses due to the gas flow are small compared to tangential stresses created by the Marangoni effect." (2, p. 186)>

Further preliminary discussions of the results were presented: "The onset of the Marangoni convection due to concentration gradients... [was] fast (<<1 sec) and the velocities... [were] typical[ly] 6 mm/sec for a 3 vol% acetone solution in water. The different lifetimes of the [two types of] air bubbles... [provided] more information than [was] planned. This type of Marangoni convection, with large velocities (about 6 mm/sec), will be responsible for an increase in mass transfer in packed columns for a positive system. This will be due to a strong...

[enhancement] of the mass transfer coefficient by this extra convection compared to diffusion. This conclusion will be especially true for stagnant phases in the packed columns." (1, p. 14)

<Note: It was not clear from available references what "packed columns" referred to but Reference (10) verified that it referred to packed columns typically used for purification processes (see Drinkenburg, STS 61-A (this chapter)).>

**Key Words:** Fluid Physics, Liquid/Gas Interface, Gas Injection, Bubbles, Bubble Formation, Bubble Stability, Surface Tension, Marangoni Convection, Solutal Gradients, Mass Transfer, Evaporation, Flow Velocity, Tracer Particles, Contained Fluids

**Number of Samples:** one experimental setup
**Sample Material:** Dilute acetone-water solution. Small tracer particles in the solution made visualization possible.
**Crucible/Container Material:** Experiment cell: unknown. The gas pipes were coated with Teflon™.

**Experiment/Material Applications:**
"Mass transfers are of great importance in technological processes but the theoretical models used [to describe such transfers] generally assume linear variations of the mass transfer coefficients. In fact, surface tension and density effects induce convection flows in the liquids, usually thin films, which deeply modify these coefficients." (9, p. 74)

See also Drinkenburg, STS 61-A.

**References/Applicable Publications:**

(1) Zaar, J. and Änggard, K.: MASER and It's Effectiveness and Experimental Results. October 13-14, 1987. Japan Space Utilization Promotion Center (JSUP), 32 pp. (post-flight; short description)

(2) Dijkstra, H. A. and Lichtenbelt, J. H.: Mass Transfer Driven Marangoni Convection Under Microgravity. Appl. Microgravity Tech., I (1988), 4, pp. 180-187. (post-flight)

(3) Lichtenbelt, J. H.: Improvement of Flight Hardware and Isothermal Marangoni Convection Under Microgravity Conditions. Adv. Space Res., Vol. 6, No. 5, 1986, pp. 97-100. (related research)

(4) Jönsson, R.: The Microgravity Program in Sweden-Emphasis on the Materials Rocket Maser. In 15th International Symposium on Space Technology and Science, Japan, Tokyo, May 19-23, 1986, pp. 2099-2110. (preflight)

(5) Zaar, J., Björn, L., and Jönsson, R.: Preliminary MASER 1 Results and the Evolution of the MASER Programme. In Proceedings of the 8th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 359-361. (post-flight; very short description)

(6) Grunditz, H.: Flight Results of the ESA Experiment Modules in MASER 1. In Proceedings of the 8th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 363-367. (post-flight)

(7) Grunditz, H.: Experiment Equipment for Metallurgy and Fluid Science Studies Under Microgravity. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

(8) Jönsson, R.: SSC Microgravity Sounding Rocket Program MASER. In 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

(9) Three Dimensional Marangoni Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 74-75. (post-flight)

(10) Input received from Experiment Investigator, August 1993.

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**Co-Investigator(s):** Siekmann, J. (2)
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**Experiment Origin:** Federal Republic of Germany
**Mission:** MASER 1
**Launch Date/Expt. Date:** March 1987
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Fluid Science Module (FSM): The thermocapillary drop motion experiment was one of three experiments performed within the FSM. This experiment employed an experiment cell, droplet injector, heating and cooling facility, and a camera/illumination system.
**Builder of Processing Facility:** Saab Space, AB, Linköping, Sweden

**Experiment:**
Thermocapillary Drop Motion

On Earth, droplet motion caused by thermocapillary forces is largely masked by gravity-driven buoyancy forces.

This MASER 1 experiment was the third in a series of investigations designed by Wozniak and/or Nähle and/or Neuhaus et al. to study the thermocapillary convective motion of particles in liquids (see Nähle (STS 61-A and Neuhaus, STS 61-A (both in this chapter)). The overall goal of this experiment was to demonstrate and study droplet motion induced by thermal variations in interfacial tension at a droplet contour The objectives included (1) the verification of the theory of Young et al. (which described thermocapillary particle motion for droplets at low Reynolds numbers, Re, and low Marangoni numbers, Ma), (2) the procurement of data to test theories for higher Re and Ma numbers (numerical models), and (3) the verification of the MASER experimental setup including a droplet injector, heating and cooling facility, and camera/illumination visualization system.

The experiment was one of three investigations housed in the MASER Fluid Science Module (FSM) during the mission. (The other two experiments are detailed under Lichtenbelt, MASER 1 (this chapter) and Aalto, MASER 1 (Chapter 15)). One (60 x 26 x 12 $mm^3$) test cell, which contained an aqueous ethanol (alcohol and water) solution, was used for the investigation. A droplet injector attached to the test cell was configured to deploy small drops of paraffin oil into the solution. Reportedly, "The water content of the aqueous ethanol solution was matched such that the... [solution] and the droplet liquid exhibited equal densities at a temperature of approximately 22 $^{o}C$." (3, p. 174) ("The use of liquids exhibiting approximately equal densities gives an additional reduction of the influence of buoyancy forces due to residual gravity on the thermocapillary droplet motion." (3, p. 175))

Two minutes prior to rocket launch, the ethanol solution was preheated. During the low-gravity phase of the rocket flight, one wall of the cell was heated by a resistance heater and the opposite wall was passively cooled by conduction (using a phase transition material (paraffin wax)). After approximately 5 of the 7 minutes of low-gravity time available during the flight, a steady, linear thermal gradient had been achieved. (Five thermocouples located near one of the lateral walls measured the thermal gradient in the liquid (approximately 7 $^{o}$C/cm).) Once the gradient was established, small drops of paraffin oil (ranging in size from 1 to 2.5 mm in diameter) were to be injected into the alcohol-water solution "...using an injection needle traversing horizontally through the liquid matrix, releasing the droplets by short acceleration pulses.... The intention was to inject 5 droplets with gradually increasing diameter over the cell length of 6 cm leaving a distance of 1 cm between the particles." (3, p. 174)

The resulting drop motion was documented by a TV camera. (A specialized illumination system associated with the experiment made visualization of the motion possible.) Real-time data taken by the camera was transmitted to the ground via an S-band video link.

Reportedly, only two paraffin drops were deployed. It was suspected that overheating of the FSM, which had occurred during rocket launch count-down, "...caused an over pressure in the experiment chamber. The overheating of the Fluid Science Module was due to larger heat transport from the GEODE module than anticipated. [<Note: More information on GEODE can be found under Redden, MASER 1 (Chapter 9).>] Because of the overpressure, alcohol was pressed into the drop injector. This disturbed the function of the drop injector such that the first three drops probably consisted of alcohol, and the fourth one of paraffin contaminated with alcohol. The fifth drop was pure paraffin and was injected correctly." (1, p. 15)

It was reported that the fact that the fourth drop (2.2 mm diameter) did not move was distinct proof that the residual gravity had no influence on the motion of the fifth (3.1 mm diameter, pure paraffin oil) drop. This fifth drop was driven only by thermocapillarity and moved toward the direction of increasing temperature (hot wall) at a slow, steady speed of (approximately) 0.078 mm/sec. The corresponding Young velocity (for a drop of the same diameter and in a similar temperature gradient) was cited as 0.082 mm/sec. Excerpts (cited in

Reference (1)) from one of Wozniak's papers reported that "the experimentally obtained drop velocity is in satisfactory agreement with the velocity prediction of classical theory [of Young et al.].... This theory is only valid for Mg- and Re-numbers equal to zero. The computation of the velocity by a numerical model which takes finite Re- and Mg-numbers into account, is still in preparation." (1, p. 16)

<Note: It is presumed that Mg refers to Marangoni number although the usual denotation is Ma.>

Encouraging results from this experiment prompted the investigators to perform further investigations on MASER 2 (see Wozniak, MASER 2).

**Key Words:** Fluid Physics, Aqueous Solutions, Liquid/Liquid Interface, Drops, Surface Tension, Interfacial Tension, Buoyancy Effects Diminished, Marangoni Convection, Thermocapillary Convection, Particle Motion, Marangoni Movement of Droplets, Thermal Gradient, Resistance Heating, Passive Cooling, Conduction, Drop Formation, Liquid Injection, Liquid Expulsion Through a Small Orifice, Droplet Size, Droplet Injection, Drop Velocity, Contained Fluids, Acceleration Effects, Illumination System, Contamination Source, Hardware Malfunction

**Number of Samples:** One setup, five drops expected, two attained
**Sample Materials:** Alcohol-water (ethanol) solution (designated as the matrix solution), paraffin oil drops injected into matrix solution.
**Container Materials:** Heating and cooling walls-aluminum; lateral walls: Teflon™; the cell was enclosed by glass of optical quality.

**Experiment/Material Applications:**
This research is applicable to the study of (1) droplet motion and positioning in alloys, two-component melts, etc., and (2) degassing of molten materials (e.g., glass melts during containerless space processing).

**References/Applicable Publications:**

(1) Zaar, J. and Änggard, K.: MASER and Its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP), 32 pp. (post-flight; short description)

(2) Wozniak, G.: Thermocapillary Drop Motion. Uni Essen.

(3) Wozniak, G. and Siekmann, J.: Experimental Investigation of Thermocapillary Droplet Motion in Reduced Gravity-Two Sounding Rocket Experiments. Appl. Microgravity Tech., II (1989), 3, pp. 173-175. (post-flight)

(4) Wozniak, G. and Siekmann, J.: Experimental Investigation of Thermocapillary Droplet Motion in Reduced Gravity Using Sounding Rockets and Simulation Liquids. Israel 30th Conference on Aviation and Astronautics, Tel Aviv and Haifa, pp. 125-130. (postflight; MASER 1 and 2, submitted to Acta Astronautica) <Note: The current publication status of this document is unclear.>

(5) Wozniak, G., Siekmann, J., and Srulijes, J.: Thermocapillary Bubble and Drop Dynamics Under Reduced Gravity-Survey and Prospects. Z. Flugwiss. Weltumforsch. 12 (1988), pp. 137-144. (post-flight)

(6) Jönsson, R.: The Microgravity Program in Sweden-Emphasis on the Materials Rocket Maser. In 15th International Symposium on Space Technology and Science, Tokyo, Japan, May 19-23, 1986, pp. 2099-2110. (preflight)

(7) Zaar, J., Björn, L., and Jönsson, R.: Preliminary Results and the Evolution of the MASER Programme. In Proceedings of the 8th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 359-361. (post-flight; very short description)

(8) Grunditz, H.: Flight Results of the ESA Experiment Modules in MASER 1. In Proceedings of the 8th European Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 363-367. (post-flight)

(9) Grunditz, H.: Experiment Equipment for Metallurgy and Fluid Science Studies Under Microgravity. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

(10) Jönsson, R.: SSC Microgravity Sounding Rocket Program MASER. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

(11) Thermocapillary Drop Motion I. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 78-79. (post-flight)

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**Co-Investigator(s):** Siekmann, J. (2)
**Affiliation(s):** (1,2) Universität-GH-Essen, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** MASER 2
**Launch Date/Expt. Date:** February 1988
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Fluid Science Module (FSM): The thermocapillary drop motion experiment was one of two experiments performed within the FSM. This experiment employed an experiment cell, droplet injector, heating and cooling facility, and a camera/illumination system.
**Builder of Processing Facility:** Saab Space, AB, Linköping, Sweden

**Experiment:**
Thermocapillary Drop Motion

This MASER 2 experiment was the fourth in a series of investigations designed by Wozniak and/or Nähle and/or Neuhaus et al. to study the thermocapillary convective motion of particles in liquid. The overall goal of the experiment was to demonstrate and study droplet motion induced by thermal variations in interfacial tension at a droplet contour (see Nähle, STS 61-A, Neuhaus, STS 61-A, and Wozniak, MASER 1 (all in this chapter)).

The objectives were the same as those listed under Wozniak, MASER 1 and included: (1) the verification of the theory of Young et al. which described thermocapillary particle motion for droplets at low Reynolds numbers, Re, and low Marangoni numbers, Ma, (2) the procurement of data to test theories for higher Re and Ma numbers (numerical models), and (3) the verification of the MASER experimental setup including a droplet injector, heating and cooling facility, and camera/illumination visualization system.

The experiment was one of two investigations housed in the MASER Fluid Science Module (FSM) during the mission. (The other experiment is detailed under Janssen, MASER 2 (this chapter).) One ($60 \times 26 \times 15$ mm$^3$) test cell, which contained an aqueous ethanol (alcohol and water) solution, was used for the investigation. A droplet injector attached to the test cell was configured to deploy small drops of paraffin oil into the solution. Reportedly, "The water content of the aqueous ethanol solution was matched such that the... [solution] and the droplet liquid exhibited equal densities at a temperature of approximately 22 $^{o}$C." (3, p. 174)

Two minutes prior to rocket launch, the ethanol solution was preheated. During this preheating, a nominal temperature gradient resulted in the cell. "The strong accelerations after launch (before the onset of microgravity) resulted in intensive convection flows that were visible by changes in the refractive index of the liquid as...[observed] from the distortion background grid pattern in the cell. During this phase, an unexpected, off-nominal condition developed by the ingestion of gas bubbles at the right lateral wall, apparently through the elastic seal around the filling tube connected to the cell. There were seven gas bubbles, the largest 5.5 mm in diameter, which immediately after entering the cell moved toward the hot wall and remained there." (3, p. 174)

During the low-gravity phase of the rocket flight, one wall of the cell was heated by a resistance heater and the opposite wall was passively cooled by conduction (using a phase transition material (paraffin wax)). After approximately 5 of the 7 minutes of low-gravity time available during the flight, small drops of paraffin oil (ranging in size from 1 to 4.0 mm in diameter) were to be injected into the alcohol-water solution "...using an injection needle traversing horizontally through the liquid matrix, releasing the droplets by short acceleration pulses.... The intention was to inject 5 droplets with gradually increasing diameter over the cell length of 6 cm leaving a distance of 1 cm between the particles." (3, p. 174)

Reportedly, "At the beginning of the injection process, the tip of the injection needle which was initially at rest and situated behind a membrane before droplet motion, carried a small air bubble into the cell. Thus, the injection of the first droplet resulted in an introduction of a small air bubble which moved relatively fast toward the warm wall, where it remained. The next three droplets were injected nominally with sizes between 1.6 mm and 2.4 mm in diameter." (3, p. 174) The fifth (and largest drop) was not injected because (for some unknown reason) the injector needled "stopped".

"Because of the air bubbles attached to the heating wall the thermal profile... [was disturbed near the location of the bubbles.] However, the three paraffin droplets moved parallel to the temperature gradient in the direction toward the hot side. The temperature gradients were fairly linear at both sides of the test cell and exhibited approximately the same values." (3, p. 174)

The resultant thermocapillary-driven drop motion was documented by a TV camera. (A specialized illumination system associated with the experiment made visualization of the motion possible.) Real-time data taken by the camera was transmitted to the ground via an S-band video link.

It was reported that (1) the video-to-ground link failed three times during the mission, resulting in several seconds of real-time data loss and (2) interpretation of the video tape was hampered by the quality of the video image.

"Postflight inspection of the video pictures revealed that the air bubbles resulted from residual air trapped between two cell wall plates. After retrieval and disassembling of the payload the drop injector was examined, but the needle had moved to the stop position, probably when the payload hit the ground. The droplet injection was repeated and worked nominally on [the] ground." (3, p. 174) No explanation as to why the malfunction occurred could be found. <Note: Reference (4), p. 129, indicated that residual air left in the filling tube was responsible for the air bubbles.>

Because the "...three injected droplets moved vertically through the liquid matrix in the direction of the hot wall, as theory predicts... [it was concluded] that the gas bubbles had no influence on the thermocapillary movement of the droplets." (3, p. 175) For all three drops, the experimental droplet velocities were slightly lower than those predicted by Young (when similar drop size and temperature gradients were considered). Because the bubbles moved essentially as predicted, the experiment confirmed the theory and provided further data to check numerical models which simulated the motion.

**Key Words:** Fluid Physics, Aqueous Solutions, Liquid/Liquid Interface, Liquid/Gas Interface, Drops, Surface Tension, Interfacial Tension, Restance Heating, Passive Cooling, Conduction, Marangoni Convection, Thermocapillary Convection, Particle Motion, Marangoni Movement of Droplets, Thermal Gradient, Drop Formation, Liquid Injection, Liquid Expulsion Through a Small Orifice, Droplet Size, Droplet Injection, Drop Velocity, Bubbles, Bubble Formation, Contained Fluids, Illumination System, Acceleration Effects, Launch-Induced Fluid Motion, Hardware Malfunction, Photographic Difficulties

**Number of Samples:** one setup, five drops expected
**Sample Materials:** Alcohol-water (ethanol) solution (designated as the matrix solution; paraffin oil drops were injected into matrix solution.

**Container Materials:** Heating and cooling walls: aluminum; lateral walls: Teflon™. The cell was enclosed by glass of optical quality.

**Experiment/Material Applications:**
See Wozniak, MASER 1.

**References/Applicable Publications:**

(1) Zaar, J., and Änggard, K.: MASER and its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP), 32 pp. (preflight; short description)

(2) Zaar, J. and Dreier, L.: MASER II Final Report, RMLO/1-7, Swedish Space Corporation, Aug. 30, 1988.

(3) Wozniak, G. and Siekmann, J.: Experimental Investigation of Thermocapillary Droplet Motion in Reduced Gravity-Two Sounding Rocket Experiments. Appl. Microgravity Tech., II (1989), 3, pp. 173-175. (post-flight)

(4) Wozniak, G. and Siekmann, J.: Experimental Investigation of Thermocapillary Droplet Motion in Reduced Gravity Using Sounding Rockets and Simulation Liquids. Israel 30th Conference on Aviation and Astronautics, Tel Aviv and Haifa, pp. 125-130. (postflight, MASER 1 and 2) (submitted to Acta Astronautica)

(5) Thermocapillary Drop Motion II. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 80-81. (post-flight)

Papers describing other related work are listed below:

(6) Siekmann, J. and Wozniak, G.: Thermocapillary Bubble Migration at Higher Marangoni Numbers. Israel J. Technol., 23 (1986/1987), pp. 179-182.

(7) Wozniak, G., Siekmann, J., and Srulijes, J.: Thermocapillary Bubble and Drop Dynamics Under Reduced Gravity-Survey and Prospects. Z. Flugwiss. Weltrumforsch. 12 (1988), pp. 137-144.

(8) Siekmann, J., Wozniak, G., Srulijes, J., Nähle, R., and Neuhaus, D.: Experimental Investigation of Thermocapillary Bubble and Drop Motion Under Microgravity Conditions, ESA SP-256, (1987), pp. 179-182.

(9) Szymczyk, J. A., Wozniak, G., and Siekmann, J.: On Marangoni Bubble Motion at Higher Reynolds-and Marangoni Numbers Under Microgravity. Appl. Microgravity Tech., I (1987), pp. 27-29.

(10) Chun, C. H., Ehmann, M., Siekmann, J., and Wozniak, G.: Vibrations of Rotating Menisci. Proceedings of the 6th European Symposium on Material Sciences Under Conditions, ESA SP-256, (1987), pp. 229-234.

(11) Ehmann, M., Wozniak, G., and Siekmann, J.: Numerical Analysis of the Thermocapillary Migration of a Fluid Particle Under Zero-Gravity. Accepted to Archives of Mechanics.

(12) Input received from Principal Investigator G. Wozniak, August 1988 and September 1989.

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**Experiment Origin:** The Netherlands
**Mission:** MASER 2
**Launch Date/Expt. Date:** February 1988
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Fluid Science Module (FSM)
**Builder of Processing Facility:** Saab Space, Linköping, Sweden

**Experiment:**
Three-Dimensional Marangoni Convection

This MASER 2 experiment was the third in a series on investigations designed by Janssen and/or Drinkenburg and/or Lichtenbelt et al. to study concentration-driven Marangoni convection in a reduced gravity environment (see Drinkenburg, STS 61-A, Lichtenbelt, MASER 1 (both in this chapter)). The experiment was one of two investigations performed within the MASER fluid Science Module (FSM) during the mission (see also Wozniak, MASER 2 (this chapter)).

The earlier MASER 1 experiment by Lichtenbelt illustrated Marangoni convective flows at a gas-bubble/fluid interface. In line with this previous research, the specific objective of this MASER 2 experiment was "...to assess the influence of the bubble diameter on convective patterns." (5, p. 76)

Three experiment cells within the FSM were dedicated to this Marangoni convection investigation. Each of these cells (1) had a 4-cm inner diameter and was 4-cm deep, and (2) contained a dilute (3 vol.%) acetone-water solution and two hollow tubes. At the initiation of the experiment (reduced gravity conditions), air-flow between the two pipes in each cell created a single gas bubble in the liquid between the gaspipes. The bubble diameters differed in each cell: (1) 3 mm, (2) 5 mm, and (3) 7 mm. A short time after the bubble was deployed, the acetone began to evaporate into the bubble.

Using the tubes and an air-pump/air-filter system in a closed loop, a minimum amount of air flow through the gas bubble was maintained. This airstream ventilated the bubble and maintained a low concentration of acetone within the bubble.

As the evaporation of the acetone occurred, an acetone concentration gradient was created near the acetone-bubble interface. Subsequent Marangoni convection, generated by concentration-induced, surface-tension differences, was filmed via a video camera/mirror assembly. The assembly was configured to obtain a front view of all three bubbles and a top view of the 3 mm and 7 mm bubbles. Tracer particles within the acetone, coupled with a specialized lighting system, allowed visualization of the flow. Real-time data from the video camera were transmitted to the ground via an S-band video link.

It was reported that (1) these three cells within the FSM operated as expected, (2) the video-to-ground link failed three times during the mission (resulting in several seconds of real-time data loss), and (3) interpretation of the video tape was hampered by the quality of the video image. Characteristics of each of the bubbles were presented in References (4) and (5).

The 7 mm Bubble

Front View:
After the bubble had been created, two large (torus-shaped) convective roll cells developed quickly "at both sides of the bubble." <Note: See Figure 1, Reference (5).> These cells remained throughout the duration of the low-gravity phase of the experiment. Reportedly, "The velocities close to the interface were of the order of 3 mm/sec." (5, p. 76)

Top View:
Random motion appears to have occurred (at least during the initial stages of the experiment). At the time Reference (3) was written, no conclusions had been made concerning the motion.

The 5 mm Bubble:
(Only the Front View was Available)

Two large roll cells quickly appeared as in the 7 mm bubble. (The velocities close to the interface were also of the order 3 cm/s.)

The 3 mm Bubble

Front View:
Video images illustrated that this bubble was unstable and continually changed shape (at times it was nearly cylindrical). "The convective flow pattern...[changed] several times, [and] some interrupted flow...[was] seen at the right hand side of the

bubble." (4, Appendix 5, p. 10) Further, "...convective flows started only slowly around the... bubble.... Occasionally the velocities observed were of the order of 3 mm/s. Frequently strong flows occurred from one point of the interface towards the container wall." (5, p. 76) <Note: a section of the above quote may be referring to what was seen via the top view of the bubble/solution setup (see below).>

Top View:
Although it was reported that the view in this direction was available during the entire low-gravity phase of the experiment, few details about the view were presented. It was mentioned that there appeared to be "flow away from the bubble." <Note: The meaning of "flow away from the bubble" was not clear.>

Preliminary conclusions presented in Reference (4) stated that (1) "Convective flow patterns were detected around all three bubbles...[and] (2) The onset of the convection was fast in all cases." (4, Appendix 5, p. 11)

Further, in Reference (5) it was reported: "[Experimental]... observations show that the convective patterns do depend on the bubble size. The differences to previous observations are interpreted as the result of air flow differences. The mechanical shear stress on the interface increases with an increasing air flow and becomes dominant in the case of the 5 and the 7 mm bubbles in this experiment. In the case of the 5 mm bubble in MASER 1 where the air flow was weaker, the surface tension effects counteracted this shear stress. The single spot Marangoni flows around the 3 mm bubble are not yet clearly understood but are thought to originate in microscale effects." (5, p. 76)

**Key Words:** Fluid Physics, Aqueous Solutions, Liquid/Gas Interface, Gas Injection, Bubbles, Bubble Formation, Bubble Stability, Surface Tension, Surface Tension Gradients, Surface Tension-Driven Convection, Cellular Convection, Marangoni Convection, Solutal Gradients, Concentration Distribution, Mass Transfer, Evaporation, Flow Velocity, Tracer Particles, Contained Fluids, Illumination System, Photographic Difficulties

**Number of Samples:** three
**Sample Materials:** Each cell contained a dilute solution of acetone and water. A single gas bubble (either 3 mm, 5 mm, or 7 mm in diameter) was deployed into each cell.

**Container Materials:** unknown

**Experiment/Material Applications:**
Although applicable publications did not detail the applications of this research, the investigator's other related work (Reference (3)) seems to imply that the results of this research would be useful in experiments pertaining to the creation of small, spherical shells.

See also Lichtenbelt, MASER 1 and Drinkenburg, STS 61-A.

**References/Applicable Publications:**
(1) Zaar, J. and Änggard, K.: MASER and Its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP), 32 pp. (short description; preflight)

(2) Zaar, J. and Dreier, L.: MASER II Final Report/RML01-7, Swedish Space Corporation, August, 30, 1988. (post-flight)

(3) Hoefsloot, H.C.J. and Hoogstraten H. W.: Marangoni Instability in Spherical Shells. Appl. Microgravity Tech., II (1989), 2. (analytical research only)

(4) Marangoni Convection Due to Concentration Gradients. In MASER II Final Report/RML01-7, Swedish Space Corporation, August 30, 1988, Appendix 5, pp. 9-11. (post-flight)

(5) Three Dimensional Marangoni Convection. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 76-77. (post-flight)

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# CHAPTER 13

# LIQUID PHASE SINTERING

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**Experiment Origin:** Italy
**Mission:** TEXUS 8
**Launch Date/Expt Date:** May 1983
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 02-1A (which contained a rocket furnace)
**Builder of Processing Facility:** Unclear, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Liquid Phase Sintering

This TEXUS 8 experiment was the first in a series of investigations designed by Rossitto to study the liquid flow and particle rearrangement which occur during the initial stages of the liquid phase sintering process. The specific objective of the experiment was to evaluate the importance of capillary forces on the rearrangement and densification of metallic powders/spheres.

Prior to the rocket flight, sixteen samples were prepared. Some of these samples were comprised of tungsten spheres (150 or 300 microns diameter) and the rest of the samples were comprised of tungsten technical powders (25 microns average diameter). <Note: It appears (from photographs of the samples in Reference (2)) that each of these samples were cylindrical in shape.> All of the tungsten spheres and powders were coated with either Cu or Ag (15 or 30 volume percent). <Note: It is not clear which sample materials received the Cu coating and which received the Ag coating.> Each sample was inserted into a 3-mm diameter hole drilled in a boron nitride cylinder. <Note: It appears (from photographs in Reference (2)) that the configuration of the cylindrical sample within the hole allowed the sample to be processed as a liquid bridge system (fixed cylindrical ends, free-surface along circumference of the cylinder).> Four such holes were drilled into a single boron nitride cylinder. The cylinders were contained within Ni cartridges.

During the mission, the samples were processed in the isothermal zone of the TEM 02-1A rocket furnace. While under low-gravity conditions, "...the samples were heated above the melting point of copper for 60 s and then rapidly cooled. Similar experiments were carried out on Earth." (2, p. 310)

Post-flight examination of the flight samples included (1) macroscopic investigations (overall dimensions, local shrinkage characteristics, second phase distribution, and densification) and (2) microscopic investigations (spatial arrangement, impurity analysis, correlation of the characteristics with volume fraction, and the shape of the frozen menisci).

It was reported that the low-gravity processed samples "...showed a very good homogeneity in the sphere arrangement and in the second phase distribution, high densification (up to the theoretical density), and isotropy in the shrinkage. The final sphere arrangement was axially homogeneous and radial variations could be correlated with the local amount of the second phase, with transitions from s.c. [simple cubic] to b.c.c. [body-centered cubic] and f.c.c. [face-centered cubic] at the predicted values of the liquid volume fraction." (2, p. 310)

In contrast to the low-gravity results, examination of the ground-processed samples indicated a significant instability in the distribution of second-phase material. This instability was attributed to "...the expulsion of the liquid that induced the coalescence of the liquid bridges with continuous variation of the spatial arrangement along the sample and, accordingly, both radial and axial anisotropy of the shrinkage." (2, p. 310)

No further information concerning the post-flight analysis of the samples could be located at this time.

**Key Words:** Liquid Phase Sintering, Melt and Solidification, Composites with Solid Particles, Coated Particles, Metals and Alloys, Isothermal Processing, Two-Phase System, Binary Systems, Liquid Bridges, Free Surface, Surface Tension, Meniscus Shape, Capillary Forces, Powder Metallurgy, Powders, Spheres, Coated Surfaces, Homogeneous Dispersion, Particle Dispersion, Particle Transport, Particle Distribution, Sample Shrinkage, Solid/Liquid Interface

**Number of Samples:** sixteen
**Sample Materials:** Samples comprised of tungsten spheres and samples comprised of tungsten technical powders. The samples were coated with either Cu or Ag (15 or 30 vol.%) (see <Notes> above).
(W*Cu*, W*Ag*)
**Container Materials:** Crucible: boron nitride; cartridge material: nickel
(B*N*, Ni*)

**Experiment/Material Applications:**

The reasons why tungsten spheres and powders were used in conjunction with a Cu or Ag coating were not presented in the available publications.

**References/Applicable Publications:**

(1) Ossi, P. M., Rossitto, F., Roberti, R., and Silva, G.: An Analysis of the Driving Forces in Liquid Phase Sintering. In Proceedings of the 4th European Symposium on Material Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, p. 343. (preflight, experiment proposal)

(2) Liquid Phase Sintering. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 310-311. (post-flight)

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**Co-Investigator(s):** Unknown, possibly: Eliasson, A. (3)
**Affiliation(s):** (1,3) Department of Casting of Metals, Royal Institute of Technology (RIT), Stockholm, Sweden; (2) During TEXUS 10: Department of Casting of Metals, Royal Institute of Technology (RIT), Currently: National Defense Research Institute, Stockholm, Sweden

**Experiment Origin:** Sweden
**Mission:** TEXUS 10
**Launch Date/Expt. Date:** May 1984
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Swedish TEXUS experiment module containing the High Temperature Mirror Furnace (HTMF)
**Builder of Processing Facility:** Swedish Space Corporation, Solna, Sweden

**Experiment:**
Composites From High Temperature Melting Point Alloys

<Note: References (1), (2), and (5) (written by Ekbom et al.) appear to describe the objectives and results of this TEXUS 10 experiment. However, these references are not in agreement on all facts. These disagreements are highlighted below.>

"On Earth, W-Ni-Fe composites with a W content in the range of 90-97% are produced from powders by liquid phase sintering and consist of almost spherical particles of W in a W-Ni-Fe matrix. To understand the sintering mechanism it is interesting to study composites with a low content of W particles. However, the sedimentation of the W particles in the liquid matrix can only be avoided in microgravity. In such conditions, the influence of surface energy, crystallographic anisotropy and attraction between particles can be studied." (5, p. 312)

This TEXUS 10 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1 (Chapters 14 and 17)). The experiment was also the first in a series of investigations which examined low-gravity liquid phase sintering of a tungsten composite (see Ekbom, TEXUS 16 (this chapter)).

The specific objective of the TEXUS 10 experiment was to investigate sintering phenomena (e.g., particle separation, particle growth, and the influence of surface energies) in materials with a low particle fraction.

Prior to the rocket launch, two samples were powder-metallurgically-produced from a mixture of pure W powder (approximately 2.5 microns in diameter) and a prefabricated, atomized matrix of Ni-Fe-W-Co alloy powder (approximately 50 microns in diameter). Sample 1 contained 25 vol.% W particles, and sample 2 contained 50 vol.% W particles. (A complete discussion of sample preparation procedures is located in References (1) and (2).) After preparation, the samples were placed in plasma sprayed alumina crucibles and configured in the Swedish Module High Temperature Mirror Furnace (HTMF).

References (2) and (5) reported that during the low-gravity phase of the rocket flight, sample 1 was molten for 12 seconds and sample 2 for 60 seconds (the melting point of the matrix alloy is 1460 $^{o}$C). <Note: Reference (1) reports sample 1 was molten for 20 seconds and sample 2 was molten for 50 seconds.> Both samples were solidified prior to the end of the low-gravity portion of the flight. Reference samples were processed on Earth for comparison.

Post-flight examination of the low-gravity processed samples indicated that "Both samples exhibited large shrinkage holes in their central part. However, there was an even distribution of W particles, showing that no surface tension driven segregation occurred. The initially uneven distribution had become even during the short liquid phase sintering period. This indicated that the particles were well wetted by the molten matrix and that the disintegration of the agglomerates was effective. Due to a non-equilibrium reaction, some of the particles dissolved in the molten matrix,... [though] its W content increased to 27%." (5, p. 312) W particle agglomerations separated and grew from 2.5 microns (their pre-processed size) to about 7 or 8 microns in less than 1 minute (References (1) and (5)). <Note: Reference (2) indicated that "The tungsten particles have grown from around 3 $\mu$m to spheres around 10 $\mu$m in diameter in less than a minute".> "In both samples, W particles partly joined by a grain boundary or separated only by a narrow layer of matrix can be observed. This could be the result of a surface tension driven reaction." (5, p. 312) The separation was attributed to strong capillary forces of the molten matrix which effectively force the tungsten particles apart (see Reference (1), p. (12)316). Quantitative evaluation of particle growth was included in Reference (1). Growth and separation in the low-gravity processed samples may also have been facilitated by the low tungsten content (15%) of the matrix.

The number of particles which formed agglomerates was significantly lower in the rocket samples than in the 1-g processed samples. This reduced agglomeration indicated good separation of the tungsten particles due to liquid phase penetration and fragmentation of particle agglomerates. "After melting under microgravity both compositions show an even distribution of tungsten particles...." (2) The particle size distribution for sample 1 was significantly different than that observed in the ground-based sample.

Reportedly, the results indicated that the low-gravity processed samples underwent rapid separation and growth. Ground-based processing resulted in "...particle growth somewhat slower than the cubic law ($t - D^3$). The growth rate increases with increasing particle fraction. The particle separation and disintegration is fast but is combined with sedimentation of the particle." (1, p. (12)318) It was speculated that sedimentation would increase the rate of particle growth since motion would enhance diffusion. "However, the investigation shows the growth to... [occur] faster or at the same rate in the samples sintered under microgravity." (1, p. (12)318)

<Note: References (3) and (4) below, which essentially contain information describing later TEXUS flights by Ekbom et al. also mention the TEXUS 10 results.>

**Key Words:** Liquid Phase Sintering, Melt and Solidification, Ternary Systems, Composites with Solid Particles, Metals and Alloys, Capillary Forces, Surface Energy, Powder Metallurgy, Powders, Spheres, Coated Surfaces, Segregation, Sedimentation, Density Difference, Particle Dispersion, Particle Transport, Particle Agglomeration, Particle Separation, Particle Attraction, Particle Wetting, Particle Growth, Particle Size Distribution, Dissolution, Plasma Spray Coating, Coated Surfaces, Thin Films, High Temperature Materials, Surface Tension-Driven Convection, Solid/Liquid Interface, Grain Boundaries, Sample Shrinkage

**Number of Samples:** two
**Sample Materials:** Sample 1: matrix of 58% Ni-25% Fe-15% W-2% Co with 25 vol% W particles; Sample 2: matrix of 58% Ni-24% Fe-15% W-2% Co with 50 vol.% W particles.
(Ni*Fe*W*Co*)
<Note: It was not indicated in the available publications if the matrix compositions were in volume or weight percents.>
**Crucible Materials:** alumina, $Al_2O_3$
(Al*O*)

**Experiment/Material Applications:**
"Tungsten heavy metals usually containing 90-95% W, 3-7% Ni and 2-4% Fe, [are] produced from powders by liquid phase sintering. The resulting particle composite has good mechanical properties.... The alloy has ideal properties in the study of liquid phase sintering: particles growth, matrix wettability and tungsten solubility in the matrix. Composites containing a low amount of tungsten particles can, depending on the great difference in density between the particles and the molten matrix, only be studied under conditions of microgravity when the tungsten particles can grow freely floating in the molten matrix. The influence of parameters like attraction between particles and surface energy can be studied." (1, p. (12)315)

**References/Applicable Publications:**

(1) Ekbom, L. B. and Eliasson, A.: Liquid Phase Sintering of Tungsten Composites in Space: Results of Tests Performed in TEXUS. In Advanced Space Research, Vol. 8, No. 12, pp. (12)315 - (12)319, 1988. (post-flight)

(2) Ekbom, L. B.: Results from the Melting of Tungsten Composites Under Microgravity in Space (TEXUS 10 Flight). Refractory & Hard Metals, December 1987. (post-flight)

(3) Liquid Phase Sintering of Tungsten Composites Under Microgravity. Effect of Matrix Composition and Particle Growth. Tungsten & Tungsten Alloys, 1992, Metal Powder Industries Federation, Princeton, NJ. (post-flight)

(4) Ekbom, L. B. and Eliasson, A.: Liquid Phase Sintering of Tungsten Composites Under Microgravity. Effect of Matrix Composition. In Proceedings of the VIII European Symposium on Material and Fluid Sciences in Microgravity, Brussels, Belgium, April 12-16, 1992, ESA SP-333 (August 1992), pp. 673-678. (post-flight)

(5) Tungsten Composites. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 312-313. (post-flight)

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**Principal Investigator(s):** Graf, W. (1)
**Co-Investigator(s):** Unknown
**Affiliation(s):** (1) Krupp Forschungsinstitut, Essen, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 12
**Launch Date/Expt. Date:** May 1985
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 01-2: Isothermal Four-Chamber Furnace (the same furnace as flown on TEXUS 11 with slight modifications)
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Liquid Phase Sintering

Liquid-phase sintering of materials composed of tungsten-carbide particles in a Co melt is difficult. During the sintering the sample components separate because of density differences between the WC particles and Co melt. The problem can be avoided by using WC particles in the micron-size range. However, this technique introduces particle Brownian motion, and the WC particles agglomerate. Under low-gravity conditions, the effects of sedimentation and Brownian motion become negligible.

This TEXUS 12 experiment was designed by Graf to study liquid-phase sintering under low-gravity conditions (for related experiments, see Pötschke, TEXUS 11, TEXUS 12 (Chapter 5)). The specific objective of the experiment was to illustrate that under low-gravity conditions, tungsten carbide particles (1) will not agglomerate and (2) will remain dispersed throughout a liquid-phase sintered sample after solidification.

Prior to the mission, three WC-35% Co samples were prepared by hot isostatic pressing. The tungsten-carbide particle size in sample 1 was 10 microns, the WC particle size in sample 2 was 2 microns, and the WC particle size in sample 3 was 0.8 microns. Each sample was encased in its own $Al_2O_3$ crucible. The crucibles were sealed in a gas-tight, welded TZM cartridge. <Note: It is not clear if each of the crucibles was sealed in a separate TZM cartridge or if they all were placed in one cartridge. However, it appears (as discussed below) that the samples were placed in two chambers of the TEXUS furnace, thus, more than one cartridge is suspected to have been employed.>

Prior to the rocket launch, the samples (1) were placed in two chambers of the Isothermal Four-Chamber Furnace and (2) heated to 1200 $^{o}$C. <Note: The available publications did not detail how the samples were configured in the furnace chambers.> Once low-gravity conditions had been achieved, the samples were further heated to 1400 $^{o}$C. The samples were held at this temperature for approximately 5 minutes before they were cooled.

Post-flight examination of the samples revealed that an unexpected foaming reaction (foaming: 30%) occurred during the experiment. Mass spectrometer measurements on a "reference material" indicated that a C + O --> CO reaction occurred. Therefore, the samples could not be used to achieve the original experiment objectives. Instead, the formation of a bubble lattice under low-gravity conditions was evaluated.

"The driving force for the release of CO was the supersaturation of the oxygen dissolved in the molten cobalt which reacted with the dissolved carbon (2.5 mass%). Mass spectrometry measurements indicated a CO production capacity of 50 $\mu$g/g corresponding to 28 $\mu$g/g of oxygen. From the mass action constant of the reaction C + O --> CO in molten cobalt, a CO pressure of 1200 bars could be deduced which represented the driving force of the reaction. Heterogeneous nucleation of bubbles occurred at the interfaces between the molten Co and the WC particles with a critical nucleus radius of $1.6 \times 10^{-8}$ m. Nuclei could have already appeared on melting as the dissolution of 25% of the WC could have led to the formation of cavities with the critical radius. The bubbles stopped growing when an equilibrium pressure of 12 bars was reached in the 80 mm$^3$ free volume of the cartridge (99% of the oxygen was consumed). According to diffusion laws, the maximum time for the bubbles to grow... to an average diameter of 0.15 mm was 2 s. The bubble morphology... indicated that [an] expansion process occurred: coalescence and Ostwald ripening. As the convection was greatly reduced, the coalescence probably stopped at the end of the bubble growth phase. The clear spots which often still contain a bubble in their centre, are a qualitative proof that gas bubbles disappeared by Ostwald ripening. They contain only cobalt and no WC particles on the contrary to their surrounding. A theory of Ostwald ripening of gas bubbles was developed which confirmed semi-quantitatively this assumption." (2, p. 334)

No additional details of this experiment could be located at this time.

**Key Words:** Liquid Phase Sintering, Melt and Solidification, Ternary Systems, Composites with Solid Particles, Composites with Gases, Alloys, Separation of Components, Density Difference, Segregation, Sedimentation, Brownian Motion, Particle Dispersion, Particle Transport, Particle Agglomeration, Solid/Liquid Interface, Interface Physics, Reaction Kinetics, Gas Formation, Bubbles, Bubble Formation, Foams, Bubble Dispersion, Bubble Nucleation, Heterogeneous Nucleation, Bubble Growth, Bubble Coalescence, Ostwald Ripening, Dissolution, Diffusion, Supersaturation, Liquid/Gas Interface, Buoyancy Effects Diminished, Cavity, Material Strength, Hardness

**Number of Samples:** three
**Sample Materials:** WC-35% Co with particle sizes of (1) 10 micron WC particle size, (2) 2 micron WC particle size, and (3) 0.8 micron WC particle size
(Co*W*C*)
**Container Materials:** Samples contained in alumina, $Al_2O_3$ then encased in gas-tight, welded TZM cartridges
(Al*O*)

**Experiment/Material Applications:**
Co alloys (including the WC-Co system) which contain high percentages of hardening elements are of particular interest for various technological applications. These materials exhibit good strength properties with good hardness and wear resistance.

**References/Applicable Publications:**
(1) Graf, W.: Liquid Phase Sintering. In TEXUS 11/12 Abschlussbericht, German Publication, 1985. (in German)

(2) Bubble Formation, Growth and Expansion During the Liquid Phase Sintering of a WC-Co Sample. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 334-335. (post-flight)

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**Co-Investigator(s):** Unknown
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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 15
**Launch Date/Expt. Date:** May 1987
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 01-1
**Builder of Processing Facility:** Unknown, Possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Liquid Phase Sintering

This TEXUS 15 experiment was the first in a series of experiments designed by Rogge to study liquid phase sintering.

Reportedly, shortly after the successful launch of the TEXUS 15 rocket, data and television transmitters experienced a partial failure. It was discovered that a lateral burnthrough of the second stage of the rocket had occurred and the stage, in turn, had collided with the prematurely separated payload. The upper part of the payload including the TEM 01-1 module parachuted to the Earth undamaged.

Documentation further detailing the experimental objectives, setup or results of this investigation does not appear to be available.

<Note: V. Rogge was also involved with the experiments of Pötschke (see Pötschke, TEXUS 11, Spacelab D1 (Chapter 5)).>

**Key Words:** Liquid Phase Sintering, Rocket Motion, Acceleration Effects, Payload Survivability, Rocket Failure

**Number of Samples:** unknown
**Sample Materials:** unknown
**Container Materials:** unknown

**Experiment/Material Applications:**
Unspecified.

**References/Applicable Publications:**

(1) Experimentelle Nutzlast und Experimente TEXUS 15. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 107-108. (in German; post-flight)

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**Co-Investigator(s):** Unknown
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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 16
**Launch Date/Expt. Date:** November 1987
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 01-1
**Builder of Processing Facility:** Unknown, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Liquid Phase Sintering

This TEXUS 16 experiment was the second in a series of experiments designed by Rogge to study liquid phase sintering (see Rogge, TEXUS 15).

Reportedly, shortly after the successful launch of the TEXUS 16 rocket, fuel in the second stage of the rocket did not ignite as planned. After the apogee was reached and the rocket began to fall, the yo-yo despin system was deployed as programmed. Due to the unexpected excess rocket mass, however, there was an incomplete reduction of rocket spin. Subsequently, the payload separated from the second stage, but the parachute was not released. An unbraked impact of the payload resulted in the destruction of all experiment modules including the TEM 01-1 module.

Further information concerning the experimental objectives, setup, and results of this investigation could not be located at this time.

**Key Words:** Liquid Phase Sintering, Rocket Motion, Acceleration Effects, Payload Survivability, Rocket Failure, Payload Recovery System Failure

**Number of Samples:** unknown
**Sample Materials:** unknown
**Container Materials:** unknown

**Experiment/Material Applications:**
Unknown

**References/Applicable Publications:**
(1) Die Kampagne TEXUS 16. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 109-111. (in German; post-flight)

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**Affiliation(s):** (1,2) Department of Casting of Metals, Royal Institute of Technology (RIT), Stockholm, Sweden

**Experiment Origin:** Sweden
**Mission:** TEXUS 16
**Launch Date/Expt. Date:** November 1987
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 01-2
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Liquid Phase Sintering

This TEXUS 16 experiment was the second in a series of investigations designed by Fredriksson and/or Ekbom et al. to study the sintering of composites under low-gravity conditions (see Fredriksson, TEXUS 10 "Composites from High Temperature Melting Point Alloys" (this chapter)).

Very little information which specifically described the experimental setup of this investigation could be located. However, it appears (based on the response from the Principal Investigator) that the experimental setup was similar to that of Fredriksson's TEXUS 10 experiment "Composites from High Temperature Melting Point Alloys."

Reportedly (Reference (4)), "The [two] samples designed for the... [TEXUS] 16 flight contained 10 and 20 vol % tungsten particles and were produced from mixtures of pure powders; tungsten (2.5 $\mu$m), nickel (7 $\mu$m) and iron (6 $\mu$m)." (4, p. 316) While a further discussion of the preflight sample preparation was detailed in Reference (4), the reference did not detail the expected TEXUS 16 experimental procedure.

It was reported (Reference (1)) that shortly after the successful launch of TEXUS 16, fuel in the rocket's second stage did not ignite as planned. After the apogee was reached, the rocket began to fall and the yo-yo despin system was deployed as planned. There was an incomplete reduction of rocket spin, however, because of the unexpected excess rocket mass. Subsequently, the payload separated from the second stage, but the parachute was not released. An unbraked impact of the payload resulted in the destruction of all the experiment modules including the TEM 01-02 module.

The TEXUS 16 experiment was reflown on TEXUS 19 "with good result." (5) <Note: This TEXUS 19 experiment is beyond the publication limits of this document. The results of the TEXUS 19 experiment (and additional (later) TEXUS experiments by Ekbom et al.) will appear in later versions of this Technical Memorandum. Some of the references pertaining to the TEXUS 19 experiment are listed below. Additional note: Reference (3) was not available to aid in the preparation of this experiment summary.>

No further information concerning the specific experiment objectives or setup could be located.

**Key Words:** Liquid Phase Sintering, Melt and Solidification, Ternary Systems, Composites with Solid Particles, Metals and Alloys, Powder Metallurgy, Powders, Spheres, Particle Dispersion, Particle Transport, High Temperature Materials, Solid/Liquid Interface, Rocket Motion, Acceleration Effects, Payload Survivability, Rocket Failure, Payload Recovery System Failure

**Number of Samples:** two
**Sample Materials:** Sample 1 contained 10 vol% tungsten particles and was produced from mixtures of pure powders: tungsten (2.5 μm), nickel (7 μm) and iron (6 μm); Sample 2 contained 20 vol% tungsten particles and was produced from mixtures of pure powders: tungsten (2.5 μm), nickel (7 μm) and iron (6 μm).
(W*Ni*Fe*)
**Container Materials:** alumina crucible
(Al*O*)

**Experiment/Material Applications:**
"Tungsten-nickel-iron composites are...[commercially] fabricated from powders by liquid phase sintering. They consist of almost spherical tungsten particles in a matrix of nickel-iron-tungsten.... [T]he sintering mechanism and the mechanical properties... [can be evaluated (in part) by studying] composites with a low amount of tungsten particles. Depending on the great difference in density between the particles and the matrix, this can only be done under microgravity." (4, p. 315)

See also Fredriksson, TEXUS 10, "Composites from High Temperature Melting Point Alloys."

**References/Applicable Publications:**

(1) Die Kampagne TEXUS 16. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 109-111. (in German; post-flight)

(2) Ekbom, L. B.: Results from the Melting of Tungsten Composites Under Microgravity in Space (TEXUS 10 Flight). Refr. & Hard Metals, Dec. 1987.

(3) Ekbom, L. B., and Eliasson, A.: Liquid Phase Sintering Under Microgravity in Space. In Proceedings P/M 88 Int. Conf. Orlando, USA, Mod. Dev. Powder Met., MPFI, Vol. 19, 1988, p. 63.

(4) Ekbom, L. B., and Eliasson, A.: Liquid Phase Sintering of Tungsten Composites in Space: Results of Tests Performed in TEXUS. Proceedings COSPAR Conf. Helsinki, July 1988; Adv. Space Research, Vol. 8:12, pp. 315-319, 1988. (post-flight, very brief discussion)

(5) Input received from Experiment Investigator, December 1989 and July 1993.

(6) Liquid Phase Sintering of Tungsten Composites Under Microgravity. Effect of Matrix Composition and Particle Growth. Tungsten & Tungsten Alloys, 1992, Metal Powder Industries Federation, Princeton, NJ. (post-flight)

(7) Ekbom, L. B. and Eliasson, A.: Liquid Phase Sintering of Tungsten Composites Under Microgravity. Effect of Matrix Composition. In Proceedings of the VIII European Symposium on Material and Fluid Sciences In Microgravity, Brussels, Belgium, April 12-16, 1992, ESA SP-333 (August 1992), pp. 673-678. (post-flight)

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**Co-Investigator(s):** Unknown
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**Experiment Origin:** USA
**Mission:** Consort 1 (Starfire Rocket)
**Launch Date/Expt. Date:** March 1989
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Powdered metal sintering furnace
**Builder of Processing Facility:** Unknown

**Experiment:**
Metal Sintering Furnace

On Earth, liquid phase sintering experiments are hampered (in part) by sedimentary forces which cause the stratification of differing density particles within the sample. When such stratification occurs, a non-uniform coarsening of the sample microstructure results.

The objective of this Consort 1 research was to study powdered metal compacts processed in a reduced gravity environment. Investigators anticipated that when gravity-dependent sedimentary forces were reduced, a sample might be produced which exhibited (1) "...a smaller grain size, [(2)] a more uniform microstructure (possibly with fewer voids or defects), and [(3)] improved mechanical properties...." (1, p. 33)

The materials chosen for the experiment were selected to allow the study of "...reactive liquid-phase sintering of intermetallic alloys in a solid-phase matrix of nonmelting metal." (2, p. 347) When such materials are heated to a two-phase region, the constituent components react "...forming an alloy liquid that must wet the solid phase." (2, pp. 347-348) Free energy, evaporation, condensation, diffusion, and bulk flow contribute to the rearrangement of particles within the melt.

Prior to flight, nine samples were prepared: three copper-cobalt systems, three copper-iron systems, and three copper-tungsten systems. When these systems were processed, the copper was the liquid phase element, and the tungsten, cobalt, or iron was the solid-phase element. "Each sample used 30 percent (volume) copper. Three different copper particle sizes (149 microns [spherical], 105 microns, and 42 microns) were independently mixed with a majority phase powdered metal (43 microns Co, 6-9 microns Fe or 1 micron W). [Prior to the flight,] Compacts were pressed at 11.32 tons/in$^2$ for one hour and reduced in a reactor under a flowing mixture of 5 percent hydrogen, 95 percent helium. Samples were stacked in a sample holder separated by stainless steel shims. The sample holder was installed in the sintering furnace and the Consort 1 furnace module assembled. A helium atmosphere was maintained in the furnace during operation to minimize oxidation of the samples." (1, p. 33)

Just prior to launch, the sintering furnace was heated to 1040 $^{o}C$. The furnace was held at this temperature during launch and until the low-gravity phase of the rocket had been attained. During the following 287 seconds, the furnace temperature was increased to 1114 $^{o}C$ (at a rate of 16 $^{o}C/min$) and the minority phase was liquefied. During the following 8 minutes, a helium quench system was used to resolidify the samples.

Post-flight, 1-g reference samples (processed with the same operating temperatures and hold times as the rocket specimens) were compared to the Consort 1 samples. Visual inspection of all samples revealed that whereas the 1-g samples generally had a non-uniform shape with an irregular surface, low-g samples were physically symmetric with smooth surfaces.

Usually, the most uniform processing possible in the 1-g environment occurs when the minority phase in the powdered metal compacts is less than 10%. Thus, the 1-g, 30% minority phase samples were highly non-uniform, as expected. For example, the 1-g, Co-Cu samples exhibited extreme deformation. This deformation was generally attributed to the wetting and flow of the liquid phase along the crucible wall. In contrast, the low-g, Co-Cu systems exhibited a high degree of uniformity at this high liquid composition with no indication of flow along the crucible wall. Thus it was concluded that "...it is evident that processing of high liquid fraction Co-Cu compacts in microgravity offer performance and homogeneity not achievable in a 1-g environment." (1, p. 34)

Reference (1) contains a discussion of density and theoretical density determinations of the 1-g and rocket samples (see p. 34). Reportedly, "...the 1-g [W-Cu] samples densify 4 percent more than low-g [W-Cu] samples." (1, p. 34) <Note: Similar density data of the other systems was not presented.> Details of the shrinkage of the samples were given in Reference (1), p. 35.

At the time Reference (1) was published, metallurgical and surface analyses of the low-g samples had been completed using a variety of techniques. However, no discussion of the results of these analyses was presented. It was further reported in Reference (1) that similar metallurgical and surface analyses of the 1-g samples had not yet been completed.

No further information concerning this experiment could be located at this time.

**Key Words:** Liquid Phase Sintering, Melt and Solidification, Binary Systems, Two-Phase System, Composites with Solid Particles, Metals and Alloys, Powder Metallurgy, Intermetallics, Minority Phase, Powders, Spheres, Particle Dispersion, Particle Transport, Particle Distribution, Particle Wetting, Sedimentation, Density Difference, Separation of Components, Free Energy, Evaporation, Condensation, Diffusion, Buoyancy Effects Diminished, Reaction Kinetics, Solid/Liquid Dispersion, Solid/Liquid Interface, Sample Microstructure, Surface Morphology, Coarsening, Grain Size, Sample Shrinkage, Sample Deformation, Quench Process, Oxidation, Wetting, Wetting of Container, Non-Wetting of Container

**Number of Samples:** nine
**Sample Materials:** Three samples of copper-cobalt, three samples of copper-iron, and three samples of copper-tungsten (thirty volume percent copper was used in all samples).
(Cu*Co*, Cu*Fe*, Cu*W*)
**Container Materials:** Stainless steel

**Experiment/Material Applications:**
"Potential uses for liquid-phase sintered products include bearings, magnetic materials, electrical brushes and contact points, advanced cutting tools to cut at higher linear speeds then currently possible, irregular shaped mechanical parts for high stress environments, and possibly new and improved catalysts for chemical production." (1, p. 33)

**References/Applicable Publications:**
(1) Powdered Metal Sintering and Infiltration. In Consortium for Materials Development in Space, University of Alabama in Huntsville, Annual Report, Technical Section, October 1, 1988-September 30, 1989, pp. 33-36. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351.

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Málaga, Spain, IAF #89-439. To be published in Acta Astronautica in 1990. (post-flight) <Note: The current publication status of this document is unclear.>

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# CHAPTER 14

# METALS AND ALLOYS

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**Experiment Origin:** USA
**Mission:** Skylab, SL-2, First Skylab Manned Mission
**Launch Date/Expt. Date:** June 1973 (month experiment was completed)
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Materials Processing Facility (MPF) panels, located forward from the Multiple Docking Apparatus (MDA) area, Skylab manned environment
**Processing Facility:** M512 Materials Processing Facility
**Builder of Processing Facility:** Complete facility: MSFC, Huntsville, Alabama; Electron Beam Gun: Unknown, possibly Westinghouse, Pittsburgh, Pennsylvania

**Experiment:**
Metals Melting (M551)

This Skylab experiment was designed to investigate the melting of metals under low-gravity conditions. The specific objectives of the experiment were to (1) study the low-gravity stability of a molten puddle and its interface with the solidified melt, (2) characterize metals which were melted and solidified in space, and (3) investigate the feasibility of joining and casting metals under low-gravity conditions.

The experiment evolved from an earlier proposal to study welding in space. Studies performed in the 1960's indicated that electron beam (EB) welding would be suitable for assembling structures in space. As a result, a portable EB welder was developed. At about the same time, interest in the processing of materials in space generated a desire to investigate melting and solidification of metals under low-g conditions. It was then proposed that the initial welding experiment be expanded to include studies concerning the melting and solidification of practical metals.

During the SL-2 mission, the M512 Materials Processing Facility was used to process three disc-shaped samples (4.75 inch diameter): (1) type 304 stainless steel, (2) 2219-T87 aluminum alloy, and (3) pure (99.5%) tantalum. The stainless steel and aluminum discs each varied in thickness from 0.025 to 0.250 inch; the tantalum disc varied in thickness from 0.017 to 0.062 inch. These varying dimensions allowed both penetration and non-penetration by the EB.

The basic experimental apparatus consisted of a chamber which contained (1) an electron beam gun (heat source for melting), (2) a 24 volt, 1700 rpm d.c. motor, (3) a gear reduction system (300:1), and (4) a mounting base. Since the EB required a vacuum for operation, the chamber was vented to space (approximately $10^{-4}$ Torr).

During the mission, each disc was mounted to the motor and the chamber was evacuated. The EB was impinged (80 mA and 20 kV) on the thinnest portion of the disc as rotation of the sample was initiated (35 in/min, 2.6 rpm). For the first 45 degrees of rotation, the disc was thin enough to allow complete penetration of the EB. For the next 90 degrees, the disc thickness gradually increased so the beam normally penetrated the material but did not cut through. After another 90 degrees, the thickness of the disc increased (stepped) resulting in only partial penetration of the beam for another 45 degrees. Here, at a 1/4 inch hole, the beam was terminated and the disc rotated another 22 degrees. At this point the beam was defocused and reinitiated for the dwell portion of the experiment. For the stainless steel and aluminum samples, the dwell time was 15 seconds; for the tantalum disc, the dwell time was 45 seconds. The dwell time allowed the formation of a large molten pool for detailed studies of metal solidification.

Post-flight examination of the discs indicated that there was no significant difference in the external appearance of the welds compared to those made under 1-g conditions. The experiment also indicated "...that surface tension in the absence of gravity is sufficient to hold a molten weld puddle in place, and thus demonstrated that welding operations in space are feasible using techniques identical to those employed on Earth." (11, p. 64)

The results from metallographic studies of each sample were reported to be:

2219-T87 Aluminum Alloy: The grains located in the weld nugget (partially penetrated regions) of the flight sample were considerably less elongated than in the 1-g processed material. The heat-affected region in the sample was much more extensive in the Skylab sample.

Type 304 Stainless Steel: As in the aluminum sample, the grain structure was much finer in the Skylab sample. The material welded on Earth revealed large elongated grains perpendicular to the weld puddle/base metal interface. The molten zones were narrower and the interfaces sharper than in the aluminum welds because of the lower thermal conductivity of steel.

Tantalum: Since this material was commercially pure, no complex precipitates were formed during precipitation. Large distinct grains were located in the space-welded sample. Residual vortices, frozen into the grain structure, were left behind after the solidification of the 1-g sample. (This characteristic was attributed to the high melting point and high thermal conductivity of tantalum, which resulted in rapid cooling of the material.) However, in the low-gravity sample, such vortices were not frozen into the sample.

It was concluded that the only significant difference between the 1-g and low-g results was the finer, more equiaxed grain size observed in the Skylab samples. This result was contrary to what was expected: "...the strong convective flows associated with the 1-g sample should be effective in forming the equiaxed zone through dendrite remelting and transport." (11, p. 66)

**Key Words:** Metals and Alloys, Melt and Solidification, Casting, Welding, Electron Beam Welding, Surface Tension, Convection, Buoyancy Effects Diminished, Sample Rotation, Solid/Liquid Interface, Interface Physics, Interface Stability, Sample Microstructure, Surface Morphology, Dendrites, Grain Size, Grain Structure, Vacuum, Space Vacuum, Space Structures

**Number of Samples:** three
**Sample Materials:** (1) 2219-T87 aluminum, (2) tantalum, and (3) 304 stainless steel
(Al*, Ta*)
**Container Materials:** not applicable

**Experiment/Material Applications:**
Welding has been proposed as a method for assembling large structures in space. At the time the experiment was performed, it was not known if electron beam welding would be a suitable method for this task. It was therefore proposed that an experiment be performed to examine the effects of low gravity on this process.

The reasons why the particular experimental materials (aluminum alloy, stainless steel, and tantalum) were chosen were not detailed in the available references.

**References/Applicable Publications:**

(1) McKannan, E. C. and Poorman, R. M.: Skylab M551 Metals Melting Experiment. In Proceedings of the Third Space Processing Symposium, Skylab Results, Vol. I, April 30-May 1, 1974, Marshall Space Flight Center, Alabama, pp. 85-100. (post-flight)

(2) "M551- Metals Melting." In MSFC Corollary Experiments System Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-25 - 5-31. (post-flight)

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**Experiment Origin:** USA
**Mission:** Skylab, SL-3, Second Skylab Manned Mission
**Launch Date/Expt. Date:** September 1973 (month experiment was completed)
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Materials Processing Facility (MPF) (located forward from the Multiple Docking Apparatus (MDA) area in the Skylab manned environment)
**Processing Facility:** Multipurpose Electric Furnace System (MEFS)
**Builder of Processing Facility:** Westinghouse Astronuclear Laboratory, Large, Pennsylvania

**Experiment:**
Copper-Aluminum Eutectic (M566)

This Skylab SL-3 experiment was the first in a series of investigations designed by Hasemeyer et al. to study the solidification of a eutectic alloy under low-gravity conditions.

The objectives, setup and results of this experiment (as detailed in published literature) were combined with a discussion of an experiment of the same name by Hasemeyer performed during the Skylab SL-4 mission. Therefore, the SL-3 and SL-4 copper-aluminum eutectic experiments are reported together. (See Hasemeyer, Skylab SL-4, for the combined summary.)

**Key Words:** Metals and Alloys, Binary Systems, Melt and Solidification, Directional Solidification, Eutectics, Model Materials, Buoyancy-Driven Convection, Thermal Convection, Solid/Liquid Interface, Thermal Gradient, Thermal Soak, Surface Tension, Wetting, Sample Deformation, Sample Detachment from Crucible, Sample Necking, Sample Shrinkage, Lamellar Eutectics, Lamellar Structure, Crystalline Structure, Surface Morphology, Defect Spacing, Defect Density, Electrical Resistivity, Fault Spacing

**Number of Samples:** unknown, possibly three
**Sample Materials:** aluminum-copper eutectic ($CuAl_2$ eutectic), Al-33 wt.% Cu
(Al*Cu*)
**Container Materials:** graphite sheath contained in a stainless steel cartridge
(C*)

**Experiment/Material Applications:**
See Hasemeyer, Skylab SL-4.

**References/Applicable Publications:**

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(4) Data Package for Cartridges for the Multipurpose Electric Furnace System, M518. WANL-TME-2831, Vol. II, November 1972, Westinghouse Astronuclear Laboratory, P.O. Box 10864, Pittsburgh, Pennsylvania 15236 (Contract NAS8-28271). (preflight)

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(9) Otto, G. H. and Lacy, L. L.: The Electrical Properties of Low-G Processed Immiscible Alloys. In Proceedings of the Third Space Processing Symposium, Skylab Results, April 30-May 1, 1974, Marshall Space Flight Center, Alabama, Vol. II, pp. 1031-1044. (post-flight)

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(11) Input received from Co-Investigator, L. L. Lacy, July 1993.

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**Experiment Origin:** USA
**Mission:** Skylab, SL-4, Third Skylab Manned Mission
**Launch Date/Expt. Date:** January 1974 (month experiment was completed)
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Materials Processing Facility (MPF) (located forward from the Multiple Docking Apparatus (MDA) Area, Skylab manned environment)
**Processing Facility:** Multipurpose Electric Furnace System (MEFS)
**Builder of Processing Facility:** Westinghouse Astronuclear Laboratory, Large, Pennsylvania

**Experiment:**
Copper-Aluminum Eutectic (M566)

<Note: Hasemeyer et al. performed two copper-aluminum eutectic experiments during the Skylab program: one on SL-3 and one on SL-4. Documentation describing these experiments combined the results from the SL-3 and SL-4 experiments. Therefore, this summary includes information on both experiments (with distinctions between the two investigations noted).>

Certain eutectic alloys solidify in a lamellar crystal pattern. On Earth, these lamellar platelets often grow irregularly with breaks and discontinuities in the lamellar structure. Such irregularity of the structure is attributed to gravity-dependent, thermal convective flows. In the space environment, such convective flows should be reduced and the processing of crystals with a continuous lamellar structure should be possible.

The Skylab SL-4 experiment was the second in a series of investigations designed by Hasemeyer et al. to study the low-gravity solidification of a eutectic alloy (see Hasemeyer, Skylab SL-3). The specific objective of the SL-3 and SL-4 experiments was to determine if the eutectic lamellae continuity and structure could be improved by reducing gravity-driven convective flow.

During the SL-3 and SL-4 missions, the Multipurpose Electric Furnace System (MEFS) was used to directionally solidify samples of Al-33 wt.% Cu ($CuAl_2$ eutectic). Each sample (6.25 mm in diameter, 12.7 mm long) was contained in a graphite sheath which was sealed in a stainless steel cartridge. The samples were to be (1) heated to 790 °C (furnace temperature of 867 °C), (2) held at this temperature for one hour, and (3) solidified at an average rate of 2.4 °C/min (average thermal gradient of 45 °C/cm). However, "Due to a malfunction of the control thermocouple in the furnace in Skylab 3, the peak temperature did not exceed 844 °C.... Three additional specimens were processed in Skylab 4, at the desired temperature of 867 °C." (1, p. 459) <Note: Although unclear to the editors, it appears three samples were processed during the Skylab SL-3 mission and three during the Skylab SL-4 mission.> Ground-based experiments were also performed using the same thermal history as the flight specimens.

Post-flight examination of the samples included (1) photography, (2) radiography, (3) resistivity measurements (using decay of eddy currents), and (4) quantitative metallography. The photographic and radiographic analysis indicated that the flight specimens from both missions had a reduced diameter (hour glass shape) in the regrowth regions. The Skylab 4 samples had only a slight reduction and the ground-processed material contained no diameter reduction. The shape of the flight specimens was attributed to a failure of the molten material to wet the graphite sheath. It was reported that, in the absence of gravity, surface tension was the dominant force in shaping the melt.

Resistivity measurements indicated little or no difference between the flight and ground specimens. Localized resistivity measurements indicated an increase in resistivity near the surface deformities mentioned above. This increase was due to a disruption of the aligned lamellae near this section of the sample.

Metallographic examination of the samples indicated that the Skylab 4 specimens had 20% fewer defects and deformities than the ground processed materials. The Skylab 3 samples contained a higher fault density than either the Skylab 4 or ground samples. (This was attributed to the more severe surface irregularities of this material.) Quantitative studies of Skylab 4 specimens indicated that the defect spacing (expressed in lamellar width) was 12% better than in samples similarly processed on Earth.

**Key Words:** Metals and Alloys, Binary Systems, Melt and Solidification, Directional Solidification, Eutectics, Model Materials, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Thermal Convection, Solid/Liquid Interface, Thermal Gradient, Thermal Soak, Surface Tension, Wetting, Non-Wetting of Container, Sample Deformation, Sample Detachment from Crucible, Sample Necking, Sample Shrinkage, Lamellar Eutectics, Lamellar Structure, Crystalline Structure, Surface Morphology, Defect Spacing, Defect Density, Electrical Resistivity, Fault Spacing, Semiconductor Applications, Superconductors, Optics Applications, Hardware Malfunction

**Number of Samples:** Three samples were processed during the Skylab SL-4 mission. It appears three samples were processed during the Skylab SL-3 mission.
**Sample Materials:** aluminum-copper eutectic specimens, Al-33 wt.% Cu ($CuAl_2$ eutectic)
(Al*Cu*)
**Container Materials:** Graphite sheath contained in a stainless steel cartridge
(C*)

**Experiment/Material Applications:**
Alloys with continuously perfect lamellar structures can have numerous practical applications as (for example) (1) superconductors, (2) optical materials, (3) capacitors, (4) semiconductors, and (5) directional magnets.

The Al-Cu eutectic system was selected for this study because (1) it is representative of many types of alloys that solidify in a lamellar crystal pattern, (2) it has been studied thoroughly in the past, and (3) it has a moderate eutectic temperature.

**References/Applicable Publications:**
(1) Hasemeyer, E. A., Lovoy, C. V., and Lacy, L. L.: Skylab Experiment M566 Copper-Aluminum Eutectic. In Proceedings of the Third Space Symposium, Skylab Results, April 30-May 1, 1974, Marshall Space Flight Center, Huntsville, Alabama, Vol. 1, pp. 457-467. (post-flight)

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(5) "Experiment M566- Aluminum Copper Eutectic." In MSFC Skylab Corollary Experiment Systems Mission Evaluation. NASA TM X-64820, September 1974, pp. 5-82 - 5-86. (post-flight)

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(7) M518-Multipurpose Electric Furnace System. In MSFC Skylab Corollary Experiment Systems Mission Evaluation, NASA TM X-64820, September 1974, pp. 5-42 - 5-56. (processing facility)

(8) Chassay, R. P. and Schwaniger, A: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements)

(9) Otto, G. H. and Lacy, L. L.: The Electrical Properties of Low-G Processed Immiscible Alloys. In Proceedings of the Third Space Processing Symposium, Skylab Results, April 30-May 1, 1974, Marshall Space Flight Center, Alabama, Vol. II, pp. 1031-1044. (post-flight)

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(11) Input received from Co-Investigator L. L. Lacy, July 1993.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** SPAR 1
**Launch Date/Expt. Date:** December 1975
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Isothermal heating and cooling module within the General Purpose Rocket Furnace (GPRF)
**Builder of Processing Facility:** unknown

**Experiment:**
Preparation of a Special Alloy for Manufacturing of Magnetic Hard Superconductors Under a Zero Gravity Environment (74-63)

"The known second order superconductors are distinguished primarily by their almost undeformable state, e.g., intermetallic compounds such as $Nb_3Sn$. In [the] search... [for] superconductors[,] ductile materials must be found where technical manufacturing will be interesting. By combining those metals, the production of a mixed state is possible and necessary. Within this state a first order superconductor must be combined with a normal conducting material.... The essential supposition is that both phases exist parallel within dimensions at least equal to the mean free length of the path of electrons for cross direction of the electrical flux. This state contains thin fibers or small particles with diameters or thicknesses of approximately 100... [angstroms]....

"Those materials can be divided into two categories. The first can be drawn down to wires and fibers with diameters in the range of 100 to 50... [microns]... and 1000 to 100... [angstroms] respectively, by conventional techniques. For the second, special methods are required because of their brittleness. As a result the first order superconductor changes to a second order superconductor because a magnetic field will penetrate the thin normal conducting areas uniformly and will also enter to some extent into the surrounding material. This field thereby stabilizes the superconducting state magnetically." (1, p. IX-3)

This SPAR 1 experiment was designed to investigate the low-gravity melting and solidification of a PbAgBaO superconducting alloy. The specific objective of the experiment was to produce a second or third order superconductor from a mixture of a first order superconductor (lead) and a normal electrical material (silver).

Prior to the SPAR 1 mission, the flight sample was prepared from a combination of Pb, Ag, and BaO powders by (1) mixing, (2) compression (2000 kN), (3) sintering (2 hours at 220 °C under vacuum), (4) extrusion (13.1 mm diameter), and (5) machining (12.1 mm diameter, 20 mm long). After preparation, the flight sample was placed in a cartridge which was then welded closed.

Two other samples were prepared for ground-based processing: (1) a sample which had been prepared only by mixing, compressing, and sintering and (2) a "thermal test" sample. <Note: It was unclear exactly what processes the thermal test sample underwent (see Reference (1)). Further, it appears that both ground-based samples were also placed in a cartridge and welded closed.>

During the mission, one of three chambers of the General Purpose Rocket Furnace (GPRF) was used to process the sample. Although the actual time-temperature profile of the flight sample was not reported, it appears that the intended melt temperature was about 550 °C. The hold time at this temperature appears to have been approximately 260 seconds.

Post-flight, both the low-gravity and 1-g samples were subjected to a deformation process. "First, the samples... [were]... extruded from [a] 12.1 mm diameter to the final diameters of 5 mm and 4 mm. This reduction by extrusion represents an optimum of deformation. Before the following drawing process the wires... [were]... enclosed in 1 mm or 2 mm thick silver tubes, avoiding a breakdown. The wires... [were]... cold rolled in presses down to 1 mm diameter, then cold drawn by means of dies in small steps to diameters of 900... [microns]..., 180... [microns]..., and 100... [microns]... including the silver sheath. The wire of the flight sample could be drawn to 100... [microns]... in diameter; hence, the true diameter of the alloy is 35... [microns]... and the thickness of the sheath is in the range of 30... [microns].... Only the sintered specimen cannot be reduced to those diameters because a codeformation of the silver particles is not possible." (1, p. IX-11)

After the deformation procedure was completed, metallography and superconductivity analysis was performed on the 1-g and low-g samples. These investigations led to the following results:

1. A deformation of the silver particles within the lead matrix may be obtained by the addition of a small amount of BaO particles. The BaO particles result in a strengthening of the Pb, allowing a codeformation of the Pb and Ag. Deformation of the sintered samples did not result in a codeformation of the silver phase.

"2. The strain hardening of the lead phase increases in such a large range during the deformation (rolling in presses and wire drawing) that the silver particles can be reduced 90 percent in area (length/thickness = 10). At high reductions a softening in strength or recovery of the lead stops further deformation of the silver.

"3. A remarkable effect in the superconducting behavior can be obtained by the flight sample only. A transition of a first order superconductor to a second order superconductor in the form of a mixed state takes place. This effect is expressed by a splitting of the critical magnetic field $H_c$ of the lead phase into a lower field $H_{c1}$ and an upper field $H_{c2}$.

"4. To obtain a better superconducting behavior of this alloy, it is necessary to start at smaller dimensions of the silver particles before the deformation begins. Reaching an optimum process time and/or an eutectic solidification, the silver particles can be precipitated as small fibers. The... wire drawing process can be stopped earlier at large diameters of the wires because the deformed fibers of the silver occur earlier in the dimensions of the mean free path of length of electrons." (1, p. IX-18)

Additional information concerning this experiment is provided in Reference (1).

**Key Words:** Metals and Alloys, Ternary Systems, Melt and Solidification, Powder Metallurgy, Sintering, Powders, Superconductors, Intermetallics, Magnetic Fields, Conductance, Solid/Liquid Interface, Material Strength, Eutectics, Multiphase Media, Dispersion, Dispersion Strengthening, Particle Strengthening, Precipitation, Fibers, Fiber Dispersions, Sample Deformation, Brittleness, Semiconductor Applications

**Number of Samples:** one
**Sample Materials:** PbAgBaO
(Pb*Ag*Ba*O*)
**Container Materials:** unknown

**Experiment/Material Applications:**
The two materials (first order superconductor and normal conductor) selected for a second order superconductor must have equal mechanical strength properties. Lead represented the best first order superconductor and silver was one of the best normal conductors. However, the differences in strength between these two materials would allow only the deformation of lead and not of silver. Therefore, lead must be dispersion hardened by adding a small amount of a large atomic volume element. This addition would increase the strength of lead by the Orowan mechanism. BaO was selected as the additive because it is not soluble in lead or silver and has a large atom volume.

**References/Applicable Publications:**

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**Experiment Origin:** USA
**Mission:** SPAR 1
**Launch Date/Expt. Date:** December 1975
**Launched From:** White Sands, Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Plexiglas$^{TM}$ cuvette with battery-powered thermoelectric cooling units
**Builder of Processing Facility:** NASA, Marshall Space Flight Center, Huntsville, Alabama

**Experiment:**
Direct Observation of Solidification as a Function of Gravity (74-21/1)

Several theories concerning the transition between columnar and equiaxed solidification have been published. Most of these theories have relied on gravity as the driving force for fluid flow and subsequent crystal multiplication. However, a theory by Chalmers (see Reference (10)) which was formulated without gravity terms, suggested that the equiaxed zone forms due to constitutional supercooling ahead of the interface.

This SPAR 1 experiment was the first in a series of investigations designed by Johnston-McCay et al. to study the solidification of materials under low-gravity conditions. The specific objective of this experiment was to determine the influence of gravity-driven flow on the formation of an equiaxed zone. The objective was to be accomplished by observation of dendritic growth in the columnar solidification region.

Before the SPAR mission, a Plexiglas$^{TM}$ cuvette was completely filled with a saturated solution of $NH_4Cl$ (28.4 wt.%). (The complete filling insured that liquid free surfaces would be avoided.) The cuvette was contained in an apparatus which was configured with (1) two battery-powered thermoelectric cooling units, (2) three thermistors, and (3) a mounting bracket. (The bracket also acted as the heat sink for the thermoelectric units.) The assembly was backlighted by a tungsten filament lamp and the solidification process was photographed at one frame per second (240 frames total). Shortly before launch, the material was heated to a temperature above its melting point.

During the low-gravity period of the mission, the $NH_4Cl$ solution was solidified. Ground-based tests, performed using thermal conditions similar to those of the flight sample yielded 1-g reference samples for comparison. (Reference (1) contains plots of the time-temperature data for the flight and ground-based experiments.)

Post-flight examination of the flight photographs revealed that only four nuclei formed during the solidification process. These nuclei grew to form the complete casting. No free floating crystals or dendrite remelting could be detected in the sample. Some necking of the second dendrite arms was observed. However, no fragmentation of the dendrites could be detected. No equiaxed zone was observed in this sample. It was also reported that the growth rate of the solidification interfaces was less than that of individual dendrites.

Examination of the ground-processed samples indicated a large degree of macrosegregation of the material constituents caused by gravity-induced flow. Nucleation started at the cold walls, and then convective flow swept dendrites into the cuvette center. These crystals resulted in an equiaxed zone at the sample center. During the growth process, the secondary dendrite arms were oriented toward the cold wall. The growth rate of the interface fronts was similar to that of the individual dendrites.

**Key Words:** Metals and Alloys, Model Materials, Aqueous Solutions, Binary Systems, Melt and Solidification, Thermal Gradient, Casting, Columnar Solidification, Columnar to Equiaxed Transition, Equiaxial Zone, Saturated Solution, Diffusion, Buoyancy-Driven Convection, Segregation, Macrosegregation, Solid/Liquid Interface, Solidification Front Physics, Growth Rate, Supercooling, Nucleation, Sample Necking, Free Surface Elimination, Dendrites, Dendritic Solidification, Lamps, Illumination System

**Number of Samples:** one
**Sample Materials:** water-ammonium chloride, $H_2O$-28.4 wt.% $NH_4Cl$, (H*O*, N*H*Cl*)
**Container Materials:** Plexiglas$^{TM}$ cuvette

**Experiment/Material Applications:**
The water-$NH_4Cl$ system has been used (1) extensively as a model for the solidification of metallic materials and (2) for the study of grain formation. A 28.4 wt.% $NH_4Cl$ mixture was prepared by saturation at 72 $^{o}F$. This saturation composition was used to avoid the presence of solid $NH_4Cl$ prior to the start of solidification.

**References/Applicable Publications:**

(1) Johnston, M. H. and Griner, C. S.: The Direct Observation of Solidification as a Function of Gravity Levels. In Space Processing and Applications Project, SPAR I Final Report, NASA Technical Memorandum X-3458, pp. V-1 - V-19, December 1976. (post-flight report)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(3) Johnston, M. H. and Griner, C. S.: The Direct Observation of Solidification as a Function of Gravity Level. Met. Trans, 1977, Vol. 8A, pp. 77-82. (post-flight)

(4) Johnston, M. H., Griner, C. S., Parr, R. A., and Robertson, S. J.: The Direct Observation of Unidirectional Solidification as a Function of Gravity Level. Journal of Crystal Growth, 1980, Vol. 50, p. 831.

(5) Dendrite Remelting and Macrosegregation in Castings. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 13-14. (post-flight)

(6) Johnston, M. H., Griner, C. S., and Grodzka, P. G.: Convection Analysis of the Dendrite Remelting Rocket Experiment. AIAA Journal, Vol. 16, 1978, p. 417.

(7) Grodzka, P. G., Pond, J. E., and Spradley, L. W.: Thermal and Convection Analysis of the Dendrite Remelting Rocket Experiment, 74-21. LMSC-HREC TR D496847, Lockheed Huntsville Research and Engineering Center, May 1976.

(8) Johnston, M. H.: Direct Observation of Dendrite Remelting and Macrosegregation in Low-Gravity. In Applications of Space Flight in Materials Science and Technology, Proceedings of a conference held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977, issued September 1978, pp. 40-44. (post-flight; appears to be SPAR 1, may be SPAR 3.)

(9) Input received from Principal Investigator M. H. McCay, September 1988 and August 1993.

(10) Chalmers, B.: Principles of Solidification. John Wiley and Sons, Inc., New York, New York, 1964, p. 265. (solidification theory)

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**Experiment Origin:** USA
**Mission:** SPAR 2
**Launch Date/Expt. Date:** May 1976
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Plexiglas™ cuvettes with thermoelectric cooling units (the cooling units were powered by a SPAR power supply)
**Builder of Processing Facility:** NASA, Marshall Space Flight Center, Huntsville, Alabama

**Experiment:**
Direct Observation of Dendritic Remelting and Macrosegregation in Castings (74-21/2, 74-21/3)

This SPAR 2 experiment was the second in a series of investigations designed by Johnston-McCay et al. to study the solidification of materials under low-gravity conditions (see Johnston-McCay, SPAR 1). The specific objectives of the experiment were to (1) observe the influence of low-gravity on the solidification process, (2) eliminate density-driven flow as an effect on the formation of equiaxed zones in castings, (3) observe dendritic growth in the columnar solidification region, (4) determine the effect of gravity-induced flow on dendritic remelting, (5) determine if macrosegregation is controlled by solidification contractions, (6) explain the formation of equiaxed zones in earlier Skylab welding experiments <Note: This possibly refers to an experiment conducted by R. Poorman (see Poorman, Skylab SL-2 (this chapter)).>, and (7) increase knowledge of casting structures produced on Earth.

During the SPAR 2 sounding rocket mission, two samples of a saturated solution of $NH_4Cl$ (28.4 wt.%) were to be solidified. One sample was to be solidified uni-directionally while the second was to be solidified from three sides. Each sample was contained in its own Plexiglas™ cuvette, which was completely filled to avoid liquid free surfaces. One cuvette (unidirectional) had one thermoelectric cooling unit on the bottom; the other cuvette had three thermoelectric units (one on each side, and one on the bottom) (The cooling units were powered by a SPAR supply). It appears that each cuvette (with cooling unit(s)) was configured in an apparatus which contained (1) three thermistors, and (2) a mounting bracket (which also acted as the heat sink for the thermoelectric units). The assembly was backlighted by a tungsten filament lamp and the samples were photographed at one frame per second (240 frames total). Ground-based tests, performed using thermal conditions similar to those of the flight samples yielded 1-g reference samples for comparison.

Post-flight analysis of the available data revealed that solidification of the SPAR samples did not occur. After a thermal model of the apparatus was created and analyzed it was reported that "The close correlation of the model to the actual flight temperature history indicates that increased Joule heating across the thermoelectrics, due to a loose or cracked junction, was the cause of the anomalous behavior...." (1, p. V-5)

Thus, "The absence of sample solidification during the low-g period of flight precluded achieving the experiment objectives." (1, p. V-5)

**Key Words:** Metals and Alloys, Model Materials, Aqueous Solutions, Binary Systems, Melt and Solidification, Directional Solidification, Thermal Gradient, Casting, Columnar Solidification, Columnar to Equiaxed Transition, Equiaxial Zone, Saturated Solution, Diffusion, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Segregation, Macrosegregation, Solid/Liquid Interface, Solidification Front Physics, Growth Rate, Free Surface Elimination, Dendrites, Dendritic Solidification, Lamps, Illumination System, Hardware Malfunction, Sample Not Processed As Planned

**Number of Samples:** two
**Sample Materials:** water-ammonium chloride, $H_2O$-28.4 wt.% $NH_4Cl$, (H*O*, N*H*Cl*)
**Container Materials:** Plexiglas$^{TM}$ cuvette

**Experiment/Material Applications:**
See Johnston-McCay, SPAR 1.

**References/Applicable Publications:**

(1) Johnston, M. H. and Griner, C. S.: The Direct Observation of Dendrite Remelting and Macrosegregation in Casting. In Space Processing Applications Rocket Project SPAR II-Final Report, NASA TM-78125, pp. V1-V13, November 1977. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(3) Dendrite Remelting and Macrosegregation in Castings. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 13-14. (post-flight)

(4) Johnston, M. H. and Griner, C. S.: The Direction Observation of Solidification as a Function of Gravity Level. Met. Trans., Vol. 8A, 1977, p. 77.

(5) Johnston, M. H., Griner, C. S., and Grodzka, P. G.: Convection Analysis of the Dendrite Remelting Rocket Experiment. AIAA Journal, Vol. 16, 1978, p. 417.

(6) Grodzka, P. G., Pond, J. E., and Spradley, L. W.: Thermal and Convection Analysis of the Dendrite Remelting Rocket Experiment, 74-21. LMSC-HREC TR D496847, Lockheed Huntsville Research and Engineering Center, May 1976.

(7) Input received from Principal Investigator M. H. McCay, September 1988 and August 1993.

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**Experiment Origin:** USA
**Mission:** SPAR 5
**Launch Date/Expt. Date:** September 1978
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Plexiglas™ and quartz cuvettes with thermoelectric cooling units
**Builder of Processing Facility:** NASA Marshall Space Flight Center, Huntsville, Alabama

**Experiment:**
The Direct Observation of Dendritic Remelting and Macro Segregation in Casting (74-21/2), (74-21/3)

This SPAR 5 experiment was the third in a series of investigations designed by Johnston-McCay et al. to study the solidification of materials under low-gravity conditions (see Johnston-McCay, SPAR 1, SPAR 2).

Two experiments were to be performed during this SPAR investigation. During the first (designated as Experiment 74-21/2R), a sample was to be solidified uni-directionally. During the second (designated as Experiment 74-21/3R), a sample was to be solidified from three sides.

The specific objectives of experiment 74-21/2R were to (1) observe dendrite and interface growth during unidirectional solidification and (2) assess the influence of gravity on dendrite remelting and, therefore, on the occurrence of freckling.

During an earlier SPAR experiment (see Johnston-McCay, SPAR 1), low-gravity solidification resulted in the formation of four nuclei which caused a columnar casting. Therefore, the specific objective of experiment 74-21/3R was to further study columnar solidification.

Prior to the SPAR mission, two samples of a saturated solution of $NH_4Cl$ (28.4 wt.%) were prepared. The solution to be used for the unidirectional solidification (experiment 74-21/2R) was contained in a completely filled quartz cuvette, which was sealed with a teflon cork and flat silicon washer. The solution to be used during the 74-21/3R experiment was contained in a Plexiglas™ cuvette, which was sealed by gluing a Plexiglas™ piece over the fill hole. One cuvette (unidirectional) had one thermoelectric cooling unit on the bottom; the other cuvette had three thermoelectric cooling units (presumably one on each side and one on the bottom). (The units were powered by a 28 V dc battery regulated to a constant 4 amperes.) Each cuvette (with cooling unit(s)) was configured in an apparatus which contained four thermistors and a mounting bracket. The assemblies were backlighted by two tungsten filament lamps and the solidification process was photographed at one frame per second (240 frames total, depth of field = 1 mm). Cooling devices were actuated 60 seconds after launch for experiment 74-21/2R and 30 seconds after launch for experiment 74-21/3R. Ground-based tests were conducted using the flight hardware for comparison. A theoretical analysis was performed which simulated heat transfer in both ground-based and flight experiments (see Reference (1)).

Post-flight analysis of the photographs revealed that experiment 74-21/2R (unidirectional solidification) was successfully performed. However, no results from experiment 74-21/3R were reported. "Some of the fluid appears to have leaked out of the cuvette [74-21/3R] prior to flight. The solidification is also severely limited." (1, p. III-15)

Results from the flight experiment 74-21/2R indicated that the interface grew at a slower rate than the dendrites. In the similar 1-g experiment, the interface and individual dendrites grew at approximately the same rate. Generally the dendrites at the interface grew symmetrically under low-gravity conditions and grew asymmetrically in the 1-g sample. These asymmetric dendrites exhibited growth of secondary arms on the side where no flow was apparent. Sometimes, arms located in the flow path were broken off or remelted.

Several crystals were grown ahead of the interface (free floating (FF) crystals) during the low-gravity experiment. In the 1-g experiment, such crystals were carried by visible liquid plumes. However, no such plumes were detected in the low-gravity experiment. Thus, it was suspected that residual accelerations carried the dendrites into the super-saturated fluid. The growth rates of the low-gravity FF crystals were slower than those detected in the ground-based experiment. The total growth in the low-gravity sample was dendritic. No occurrence of freckling was found in the low-gravity sample, although the phenomenon was observed ahead of the interface in the 1-g sample.

**Key Words:** Metals and Alloys, Model Materials, Aqueous Solutions, Binary Systems, Melt and Solidification, Thermal Gradient, Directional Solidification, Casting, Columnar Solidification, Saturated Solution, Diffusion, Buoyancy-Driven Convection, Segregation, Macrosegregation, Solidification Front Physics, Interface Physics, Solid/Liquid Interface, Growth Rate, Dendrites, Dendritic Solidification, Freckling, Free Surface Elimination, Liquid Leakage, Acceleration Effects, Lamps, Illumination System, Processing Difficulties

**Number of Samples:** two
**Sample Materials:** water-ammonium chloride, $H_2O$-28.4 wt.% $NH_4Cl$, (H*O*, N*H*Cl*)
**Container Materials:** Experiment 74-21/2: quartz cuvette; Experiment 74-21/3: Plexiglas™ cuvette
(Si*O*)

**Experiment/Material Applications:**
See Johnston-McCay, SPAR 1.

**References/Applicable Publications:**
(1) Johnston, M. H., Griner, C. S., Parr, R. A., and Robertson, S. J.: The Direct Observation of Uni-Directional Solidification as a Function of Gravity Level. In Space Processing Applications Rocket Project, SPAR V Final Report, NASA TM-78275, pp. III1-III39, August 1980.

(2) Input received from Principal Investigator M. H. McCay, September 1988 and August 1993

(3) Johnston, M. H., Griner, C. S., Parr, R. A., and Robertson, S. J.: The Direct Observation of Unidirectional Solidification as a Function of Gravity Level. Journal of Crystal Growth, Vol. 50 (1980), pp. 831-838.

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**Experiment Origin:** USA
**Mission:** SPAR 7
**Launch Date/Expt. Date:** May 1980
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Casting Furnace
**Builder of Processing Facility:** Lockheed, Huntsville, Alabama

**Experiment:**
Comparative Alloy Solidification (76-36/1)

This SPAR 7 experiment was the fourth in a series of investigations designed by Johnston-McCay et al. to study the solidification of materials under low-gravity conditions (see Johnston-McCay, SPAR 1, SPAR 2, SPAR 5). While the three previous experiments had examined solidification using a metal model system ($H_2O-NH_4Cl$), a major objective of this experiment was to extend the research to an actual metal system. A second major objective of the research was to examine the effect of acceleration (low g, 1-g, and high g) on the solidifying structure.

The low-gravity portion of the experiment was performed during the SPAR 7 sounding rocket mission; the 1-g and high-gravity portions (1.75 g, 3.5 g, and 5 g) were performed during ground-based testing.

Prior to the SPAR 7 mission, one sample of Sn-15 wt.% Pb was prepared. It appears that during this preparation, pure components of the sample were solidified in a cold graphite crucible. The sample material was then placed in a new graphite crucible and configured within the SPAR 7 casting furnace.

Prior to the rocket launch, the specimen was melted and held for a period of time at 220 $^{o}$C. Approximately 1 minute after the start of the low-gravity portion of the mission, cooling was initiated by the application of a helium quench. The sample was solidified during the remainder of the low-gravity period. The temperature, which was measured by six thermocouples located in the crucible, was recorded via telemetric methods.

A centrifuge was used to produce the desired high-gravity levels on similar (terestrially-prepared) samples processed on Earth. The ground-based samples were solidified at each g-level using different cooling rates. (Further details of the high-gravity experiments can be located in Reference (1).)

Post-flight examination of the low-gravity sample revealed that its grain sizes were larger than the similarly processed 1-g sample. Reportedly, the larger grain sizes resulted because gravity-dependent convective flows were reduced and (thus) no fragmentation occurred which would create nuclei for new, small grains.

Analysis of the centrifuged samples indicated that the higher the acceleration level, the smaller the grain size of the solidified sample. It was reported that as the metal was cooled (while subjected to high g levels), "...nucleation of primary dendrites occurred almost simultaneously throughout the melt. The gravity force drove the nuclei towards the bottom of the crucible, producing a greater pileup of tin dendrites and hence a more curved interface with larger accelerations." (1, p. III-8)

It was concluded that acceleration affects the final material structure by driving the fluid flow in the system. Macroscopically, this gravity-driven fluid flow causes (1) dendritic remelting and fragmentation and (2) macrosegregation. The cooling rate also affects the final structure by influencing the dendrite arm spacings and, therefore, the permeability to fluid flow. Microscopically, the fluid flow affects the local temperature and concentration gradients. As acceleration levels decreased, the fluid flow decreased, resulting in a greater driving force for dendritic coarsening. This reduction in fluid flow also resulted in a decrease in local thermal fluctuations, producing a more uniform dendritic arm spacing.

**Key Words:** Metals and Alloys, Binary Systems, Melt and Solidification, Casting, Diffusion, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Segregation, Macrosegregation, Thermosolutal Convection, Solutal Gradients, Thermal Gradient, Thermal Soak, Density Difference, Solidification Front Physics, Interface Shapes, Interfacial Curvature, Solid/Liquid Interface, Growth Rate, Cooling Rate, Surface Morphology, Nucleation, Sample Microstructure, Grain Structure, Grain Size, Dendrites, Dendritic Solidification, Dendritic Arm Spacing, Quench Process, Acceleration Effects, Processing at High Gravity Levels, Centrifuge

**Number of Samples:** one
**Sample Materials:** Sn-15 wt% Pb alloy
(Sn*Pb*)
**Container Materials:** graphite
(C*)

**Experiment/Material Applications:**
The Sn-Pb system was selected for study because (1) there is a large amount of data available on the system, (2) it has a low melting point, and (3) there is a significant density difference between the Sn and Pb components.

**References/Applicable Publications:**

(1) Johnston, M. H. and Parr, R. A.: The Influence of Acceleration Forces on Dendritic Growth and Grain Structure. In Space Processing Applications Rocket (SPAR) Project, SPAR VII Final Report, NASA TM-82535, pp. III-1 - III-18, October 1983. (post-flight)

(2) Johnston, M. H. and Parr, R. A.: The Influence of Acceleration Forces on Dendritic Growth and Grain Structure. Met Trans B, 13B, 1982, pp. 85-90.

(3) Input received from Principal Investigator M. H. McCay, September 1988 and August 1993.


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**Experiment Origin:** USA
**Mission:** SPAR 9
**Launch Date/Expt. Date:** January 1981
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Casting Furnace (CF)
**Builder of Processing Facility:** Lockheed, Huntsville, Alabama

**Experiment:**
Comparative Alloy Solidification (76-36/2)

This SPAR 9 experiment was the fifth in a series of investigations designed by Johnston-McCay et al. to study the solidification of materials under low-gravity conditions (see Johnston-McCay, SPAR 1, SPAR 2, SPAR 5, and SPAR 7). While the four previous experiments had examined either the solidification of the metal model system $H_2O$-$NH_4Cl$ (SPAR 1, 2, and 5) or a Sn-15 wt.% Pb alloy (SPAR 7), a major objective of this experiment was to extend the earlier research and investigate a different metallic system (Sn-Bi). A second major objective of the research was to examine the effect of acceleration (low g, 1-g, and high g) on the solidifying structure.

The low-gravity portion of the experiment was performed during the SPAR 9 sounding rocket mission; the 1-g and high-gravity portions were performed during ground-based testing.

Before the rocket flight, a single sample of Sn-3 wt.% Bi was placed in the SPAR Casting Furnace. Prior to the launch of the rocket, the specimen was melted and held for a period of time at 240 $^{o}$C. <Note: The exact length of time the sample was held at 240 $^{o}$C was not clear. It was held at this temperature for at least an hour.> One hundred and eighty-three seconds after launch, the furnace was switched off and helium quench of the sample was initiated. Reportedly, the cooling rate of the sample (10-12 $^{o}$C/min) was slower than anticipated.

A centrifuge was used to produce the desired high-gravity levels on samples processed on Earth. The ground-based samples were solidified at several gravity levels (1 to 7 g's) using different cooling rates.

Post-flight examination of the low-gravity flight sample revealed "...some degree of centerline porosity...." (1, p. V-2) This condition was found in SPAR 9 and ground-based samples. "Metallographic examination of the flight sample suggests that it began solidification on the 'B' surface with the dendrites proceeding to grow at an angle toward the center and the 'A' face. This is indicated both by the orientation of the dendrites... and by the occurrence of the large cavity on the 'A' surface. It is not known... if unequal acceleration forces drove the molten liquid toward one wall of the crucible, but this seems to be the most probable explanation." (1, p. V-3) <Note: It is unclear exactly what is meant by "A" and "B" surfaces. See Reference (1) for details.>

Data sent by telemetry indicated that solidification was completed a few seconds prior to the end of low gravity. "However, the small grains around the large centerline cavity could be indicative of convection as the sample finished solidifying in a gravity field." (1, p. V-3) Follow-up experiments onboard the KC-135 aircraft (parabolic flight with approximate g-levels between 2-g and $10^{-2}$g) did not reveal a "...macroscopically obvious transition." (1, p. V-3) Another possibility for these grains could be recrystallization, which was observed to be extensive in the ground-based samples. However, in the flight sample, the recrystallization was localized.

Reportedly, the main effect of low-gravity processing was predicted by earlier studies on the metal-model system (see Johnston-McCay, SPAR 1, SPAR 5): "In contrast to the fine-grained earth-processed samples, the low-g sample solidified with relatively few large grains." (1, p. V-4)

<Note: Only preliminary data from the high-gravity, ground-based experiments were presented. It was reported that "Quantitative analysis of the [ground-based, sectioned] sample surfaces will be performed at a later date when the metallographic analysis is complete." (1, p. V-2) See Reference (1) for a table of these preliminary data.>

**Key Words:** Metals and Alloys, Model Materials, Binary Systems, Melt and Solidification, Casting, Diffusion, Buoyancy-Driven Convection, Segregation, Macrosegregation, Solid/Liquid Interface, Growth Rate, Cooling Rate, Surface Morphology, Grain Structure, Grain Size, Dendrites, Dendritic Solidification, Cavity, Porosity, Quench Process, Acceleration Effects, Vehicle Re-Entry Forces/Vibration, Centrifuge, Processing at High Gravity Levels

**Number of Samples:** one
**Sample Materials:** Sn-3 wt.% Bi alloy
(Sn*Bi*)
**Container Materials:** graphite
(C*)

**Experiment/Material Applications:**
This Sn-3 wt.% Bi alloy was used in this experiment because it has an inclination toward dendritic remelting. Rather than having direct technical applications, the Sn-Bi alloy was a model material which (1) had a low-melting point and (2) could be processed in the same furnace as the Sn-Pb sample (SPAR 7).

**References/Applicable Publications:**
(1) Johnston, M. H. and Parr, R. A.: SPAR Experiment 76-36 Comparative Alloy Solidification. Space Processing Applications Rocket (SPAR) Project, SPAR IX Final Report, NASA TM-82549, pp. V1-V24, January 1984. (post-flight)

(2) Johnston, M. H. and Parr, R. A.: Low Gravity Solidification Structures in the Tin - 15wt.% Lead and Tin - 3 wt.% Bismuth Alloys. Materials Processing in the Reduced Gravity Environment of Space, Edited by Guy E. Ridone, pp. 651-656, 1982, Elseveier Science Publishing Company, Inc. (post-flight)

(3) Input received from Principal Investigator M. H. McCay, September 1988 and August 1993.

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**Experiment Origin:** USA
**Mission:** SPAR 10
**Launch Date/Expt. Date:** June 1983
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Casting Furnace
**Builder of Processing Facility:** Lockheed, Huntsville, Alabama

**Experiment:**
Comparative Alloy Solidification (76-36/3)

This SPAR 10 experiment was the sixth in a series of investigations designed by Johnston-McCay et al. to study the solidification of materials under low-gravity conditions (see Johnston-McCay, SPAR 1, SPAR 2, SPAR 5, SPAR 7, and SPAR 9). The five previous experiments had examined solidification using either (1) the metal model system $H_2O$-$NH_4Cl$, (2) a Sn-15 wt.% Pb alloy, or (3) a Sn-3 wt.% Bi alloy. The objectives of this experiment were to extend the research by (1) studying the effect of fluid flow on macrosegregation in an Al-Cu alloy and (2) obtaining quantified, experimental macrosegregation data for comparison with theoretically derived macrosegregation data (calculated using a model developed by General Electric, Inc.).

Before the mission, an Al-4.5 wt.% Cu alloy was prepared by melting pure components in a cold graphite crucible exposed to air. The resulting sample was approximately 60 mm long and 50 mm wide (100 gr total). Prior to the launch of the SPAR 10 rocket, the sample, contained in a new graphite crucible, was remelted and soaked at 720 $^{o}C$ in the SPAR Casting Furnace. During the lift-off portion of the flight, the molten sample was subjected to convective mixing. Approximately 5 seconds after the start of the low-gravity period, the furnace was switched off and quench cooling was initiated. The helium quench gas was directed at two sides (the narrow sides) of the crucible (bi-directional solidification). The sample was solidified during the remainder of the low-gravity portion of the mission. Temperature data were obtained from six thermocouples located in the graphite crucible. A similar sample was processed on Earth for comparison.

It was reported that the cooling rate of (1) the flight sample was 45.4 $^{o}$C/min, and (2) the ground-based ingot was 55.3 $^{o}$C/min. Post-flight examination of the SPAR 10 sample revealed a cavity along one face, which was typical of earlier flight specimens. The ground-processed sample also contained a cavity along one face indicating that it was processed at some angle off the vertical. Porosity was present in both samples, although it appeared to be greater and coarser in the 1-g sample.

The secondary dendritic arm spacing of the flight sample was approximately 23% less than that of the 1-g sample. The solid solution aluminum dendrites of the flight sample contained more copper than the ground sample. The flight sample also contained a smaller volume fraction of eutectic structure (9.3%) compared to the ground-processed material (10.8%). "Copper content in the ground-based sample varied from 5.0%Cu at the upper chill face corner to 4.5%Cu in the center of the ingot, near the shrinkage cavity. The flight sample varied from 5.0%Cu at the lower chill face to 4.3%Cu in the center. There seems to have been more mixing at the center of the ground-based ingot. The flight ingot shows a smooth transition from outside to center." (1, p. 50)

It was reported that "An initial concern in this experiment was the difference in cooling rate between flight and ground-based runs. Properties could not be legitimately compared if the differences were sufficiently large to affect fluid flow. However, temperature parameters in the model show a strong dependence on cooling rate primarily below 30 $^{o}$C/min, and do not differ significantly at the rates used in this experiment." (1, p. 50)

Macrosegregation in the flight sample did not match that predicted by the model, which considered a bi-directionally solidified ingot. The model predicted vertical isoconcentration lines for the flight sample. However, the isoconcentration lines determined from the flight sample were curved. This was attributed to the probability that "...the graphite crucible extracted heat at the top and bottom of the ingot as well as at the chill face." (1, p. 50) The model also predicted that the copper content would sharply drop at about 8 mm from the ingot center. This drop in Cu content would correspond to a porous region caused by shrinkage. "The model assumes that capillary forces, associated with the surface of the interdendritic liquid, are sufficient to prevent vertical drainage through the solid-liquid zone.... However, the porous region is not seen in the experimental ingots. Shrinkage is manifest[ed] as a cavity in the last-to-freeze region in both flight and ground-based samples. Consequently, copper macrosegregation profiles in the model are higher in the outside region of the ingot and lower in the center region than those seen in the experimental ingots." (1, p. 50)

**Key Words:** Metals and Alloys, Binary Systems, Eutectics, Melt and Solidification, Directional Solidification, Thermal Gradient, Solutal Gradients, Thermosolutal Convection, Casting, Diffusion, Buoyancy-Driven Convection, Segregation, Macrosegregation, Concentration Distribution, Density Difference, Thermal Soak, Solid/Liquid Interface, Growth Rate, Cooling Rate, Sample Microstructure, Sample Shrinkage, Surface Morphology, Dendrites, Dendritic Solidification, Dendritic Arm Spacing, Cavity, Porosity, Quench Process, Acceleration Effects, Launch-Induced Fluid Motion

**Number of Samples:** one
**Sample Materials:** aluminum-4.5 wt% copper
(Al*Cu*)
**Container Materials:** graphite
(C*)

**Experiment/Material Applications:**
"Al-4.5wt%Cu was chosen because the density difference between constituents could be expected to lead to measurable macrosegregation under the influence of gravitational acceleration, as well as for its representative status of the commercial 2000 series of aluminum alloys." (1, p. 48)

**References/Applicable Publications:**
(1) Alter, W. S.: SPAR Experiment 76-36, SPAR X Comparative Alloy Solidification. Space Processing Applications Rocket (SPAR) Project, SPAR X Final Report, NASA TM-86548, pp. 47-74, July 1986. (post-flight)

(2) Input received from Principal Investigator M. H. McCay, September 1988 and August 1993.

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**Experiment Origin:** USA
**Mission:** SPAR 1
**Launch Date/Expt. Date:** December 1975
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** General Purpose Rocket Furnace (GPRF)
**Builder of Processing Facility:** National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

**Experiment:**
Solidification of Pb-Sb Eutectic (74-5)

The eutectic structure of a Pb-Sb alloy should be superplastic (able to withstand tensile strains of 100 percent before rupturing). Preparing a eutectic Pb-Sb material on Earth is difficult because if nucleation of either of the primary phases occurs, the density difference between the two phases causes segregation. It was suspected that in the low-gravity environment, this gravity-driven segregation could be eliminated.

With relatively high solidification rates, supercooling of the material occurs. This supercooling results in a shift in the eutectic point. Since solidification rates must be increased during a rocket experiment, it is necessary to determine the extent of the eutectic shift. (According to available phase diagram data, the eutectic composition is 11.2 wt.% Sb.)

This SPAR 1 experiment was the first in a series of investigations designed by Pond et al. to study the low-gravity, eutectic solidification of a Pb-Sb alloy. The specific objective of the experiment was to determine if a complete eutectic structure would result when an alloy composed of 11.2 wt.% Sb was solidified under low-gravity conditions. (During the subsequent SPAR 2 experiment, both hypo- and hypereutectic compositions were investigated to examine the extent of the eutectic shift (see Pond, SPAR 2).)

Prior to the launch of the rocket, the Pb-11.2 wt.% Sb alloy was sealed under vacuum ($1 \times 10^{-4}$ Torr) in a 321 stainless steel cartridge. (Details of sample preparation may be located in

Reference (1).) During the SPAR 1 experiment, the General Purpose Rocket Furnace was used to process the sample. <Note: Details concerning the thermal history of the sample during the SPAR experiment were not provided.>

Ground-based samples were processed for comparison using four different quench rates ranging from 0.5 to 20 $^{o}$C/sec (see Reference (1) for quench methods). Other samples were prepared by quenching in water (either the alloy itself or the crucible containing the molten alloy) which was expected to result in higher quench rates. Reportedly, no thermocouples were included in the water-quenched samples; therefore, the exact quench rates were not determined.

Post-flight examination of the low-gravity sample and 1-g samples included resistivity measurements, tensile testing, and metallography. The resistivity measurements were not conducted until receipt of the SPAR 2 samples (see Pond, SPAR 2). After longitudinally sawing a section for resistivity measurements (0.080 maximum thickness plank), two longitudinal slots, 180 degrees apart, were machined in the samples. Transverse slices, 0.070 inch thick, produced the tensile specimen wafers. The gage length was 0.120 inch with variation of 0.115 to 0.143 in gage width (details concerning preparation and testing of this tensile specimen shape can be found in Reference (1)). Tensile testing was performed at room temperature on a two-screw testing machine with a constant crosshead rate of 0.009 cm/min. A total of fifty tensile tests were performed on all the samples. (Nine of these tests were performed on the low-gravity processed sample.)

The following conclusions were reported from the experiment: <Note: It was reported that these conclusions were tentative until results from SPAR 2 experiment could be obtained.>

(1) No discernible difference between the ground-processed and flight-processed samples could be detected. "...the ground-based specimens have bracketed the SPAR I flight specimen with respect to mechanical and microstructural properties...." (1, p. I-25)

(2) A shift in the eutectic composition, due to supercooling at the solidification rates involved, was detected in both the flight and ground samples. However, this shift was not clearly defined due to the presence of dual primary crystallization products, which, according to the equilibrium diagram, are not possible.

(3) In the ground-processed samples, "...an increase in solidification rate... can cause primary lead dendrites to be replaced by primary antimony dendrites without a change in composition." (1, p. I-28)

**Key Words:** Metals and Alloys, Binary Systems, Eutectics, Eutectic Shift, Melt and Solidification, Multiphase Media, Segregation, Buoyancy Effects Diminished, Density Difference, Nucleation, Solid/Liquid Interface, Solidification Rate, Supercooling, Sample Microstructure, Dendrites, Quench Process, Electrical Resistivity, Tensile Strength, Superplastics, Material Strength, Vacuum

**Number of Samples:** one
**Sample Materials:** Pb-11.2 wt.% Sb (Asarco Grade B-58 (99.99+%)) (Pb*Sb)
**Container Materials:** AISI 321 stainless steel

**Experiment/Material Applications:**
Lead-antimony alloys are considered superplastic materials. Research on superplastic materials was instituted to (1) further understand why these metallic materials can undergo severe tensile strains (of the order of 1000%) prior to rupture and (2) allow operations and manufacturing procedures (e.g., deep drawing of cases and containers) which are impossible with other materials.

A possible application of this type of material is "...in shaped charge liners to enhance the penetrating power of the jet formed when the shaped charge is properly detonated. Besides having use as devices for tapping furnaces and opening up oil sands, the liners are used in warfare." (1, p. I-3)

**References/Applicable Publications:**
(1) Pond, R. B., Sr., Winter, J. W., Jr., Van Doren, S. L., and Shifler, D. A.: Space Solidification of Pb-Sb Eutectic Experiment 74-5. In Space Processing and Applications Rocket Project, SPAR 1 Final Report, NASA TM X-3458, pp. I-1 - I-31, December 1976. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(3) Solidification of Pb-Sb Eutectic. In Descriptions of Space Processing Applications Rocket SPAR Experiments, edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 3-4. (post-flight)

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**Experiment Origin:** USA
**Mission:** SPAR 2
**Launch Date/Expt. Date:** May 1976
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** General Purpose Rocket Furnace (GPRF)
**Builder of Processing Facility:** National Aeronautics and Space Administration (NASA), Marshall Space Flight Center, Huntsville, Alabama

**Experiment:**
Solidification of Pb-Sb Eutectic (74-5)

This SPAR 2 experiment was the second in a series of investigations designed by Pond et al. to study the low-gravity, eutectic solidification of a Pb-Sb alloy (see Pond, SPAR 1 (this chapter)). The overall objective of the investigative series was to determine if a complete eutectic structure would result when an alloy composed of 11.2 wt.% Sb (eutectic composition) was solidified under low-gravity conditions. The specific goal of the SPAR 2 experiment was to examine the extent of the eutectic shift which occurs at high cooling rates.

During the earlier SPAR 1 experiment, a Pb-11.2 wt.% Sb alloy was processed. During SPAR 2, hypo- and hypereutectic compositions were processed to study the extent of the eutectic shift. Examination of these compositions was expected to permit a determination of the eutectic shift as a function of cooling rate.

Prior to the launch of the rocket, two alloys were prepared: (1) Pb-7.0 wt.% Sb (hypoeutectic alloy) and (2) Pb-18.0 wt.% Sb (hypereutectic alloy). Each of the samples was sealed under vacuum (1 x $10^{-4}$ torr) in its own 321 stainless steel cartridge (further details of sample preparation may be located in Reference (1)).

Ground-based (comparison) samples were processed using four different quench rates ranging from 0.5 to 20 $^{o}$C/sec (see Reference (1) for quench methods). Other ground-based samples were quenched in water (either the alloy itself or the crucible containing the molten alloy) to try to produce higher quench rates.

No thermocouples were included in the water-quenched samples; therefore, the exact quench rates were not determined.

Post-flight examination of the samples included resistivity measurements, tensile testing, and metallography. After longitudinally sawing a section for resistivity measurements (0.080 maximum thickness plank), two longitudinal slots, 180 degrees apart, were machined in the samples. Transverse slice, 0.070 in thick, produced the tensile specimen wafers. The gage length was 0.120 inch with variation of 0.115 to 0.143 in gage width (details concerning preparation and testing of this tensile specimen shape can be found in Reference (1)). Tensile testing was performed at room temperature on a two-screw testing machine with a constant crosshead rate of 0.009 cm/min.

Analysis of the SPAR 2 samples revealed "...sharp corners on the vent tube end and extrusion of the alloy for a short distance into the vent tube. Upon sectioning these specimens, it was found that a cavity (or cavities) existed in the approximate center of these specimens on the end nearest the vent tube. An acceptable explanation of these cavities has not been determined. However the presence of the cavities and their location in the specimens severely limited the mechanical test evaluation for these two alloys." (1, p. III-12)

Metallographic examination of the two flight samples revealed a dual primary crystallization product (this feature was also found in the SPAR 1 flight sample): blocky, white Sb crystals either adjacent to or within primary Pb crystals. Reportedly, it appeared that these products were characteristic of the solidification rates employed. However, according to the equilibrium diagram for this alloy, this result was not reasonable. To explain this anomalous solidification behavior, post-flight experiments were performed at high gravity levels in a centrifuge. Alloys of 7, 11.2, and 17 wt.% Sb were rotationally accelerated to 1, 25, 280, and 1000 g for times of 0.1, 1, 10, 100, and 1000 minutes prior to solidification (see Reference (1) for procedures). "It was observed that, although dual primary crystals were observed in every case, the size of the Pb particles increased with time and with an increase in g.... It should be noted that 1 g for 1000 min is not exactly equivalent to 1000 g for 1 min and that the Earth's gravity field effect on the 1 g rotational field is less effective than the Earth's gravity field on 1000 g rotational field." (1, p. III-20)

The following conclusions from the above studies were reported:

(1) The results from the mechanical studies indicated no discernible difference between the low-gravity and ground-based samples. "Due to cavities found in the SPAR II specimens only a minimum number of mechanical tests were possible; however, the microstructural portrayal overrode this deficiency." (1, p. III-25)

(2) A shift in the eutectic composition of the flight and ground-based samples was detected. This shift was attributed to supercooling at the particular solidification rates. However, this shift was not clearly defined due to the presence of dual primary crystallization products.

(3) The dual primary crystallization products were a result of thermal supercooling.

(4) "High gravitational fields tend to sweep the nuclei responsible for primary crystallization from the melt. This sweeping action however results in radical segregation.

(5) "Due to the microgravity field experienced during solidification of the SPAR specimens, the primary crystallization products were homogeneously dispersed. All other gravity fields produced [a] more... [erratic] dispersion of the Pb dendrites." (1, p. III-25)

<It was reported in literature concerning the SPAR 1 experiment (see Pond, SPAR 1) that results from resistivity measurements on the SPAR 1 sample and SPAR 2 samples would be reported together. However, no mention of these examinations was made in the publications available at this time.>

**Key Words:** Metals and Alloys, Binary Systems, Eutectics, Eutectic Shift, Hypoeutectics, Hypereutectics, Melt and Solidification, Multiphase Media, Dispersion, Segregation, Density Difference, Nucleation, Solid/Liquid Interface, Cooling Rate, Sample Homogeneity, Homogeneous Dispersion, Supercooling, Sample Microstructure, Dendrites, Cavity, Quench Process, Sample Deformation, Electrical Resistivity, Tensile Strength, Superplastics, Acceleration Effects, Sample Rotation, Vacuum, Centrifuge, Processing at High Gravity Levels

**Number of Samples:** two
**Sample Materials:** (1) Pb-7.0 wt.% Sb, (2) Pb-18.0 wt.% Sb (both Asarco Grade B-58 (99.99+%))
(Pb*Sb*)

**Container Materials:** AISI 321 stainless steel

**Experiment/Material Applications:**
See Pond, SPAR 1.

**References/Applicable Publications:**


(1) Pond, R. B., Sr., Winter, J. M, Jr., Van Doren, S. L., and Shifler, D. A.: Space Solidification of Pb-Sb Eutectic Experiment 74-5. In Space Processing Applications Rocket Project, SPAR II Final Report, NASA TM-78125, pp. III-1 to III-29, November 1977. (post-flight)

(2) Toth, S. and Frayman, M.: Measurement of Acceleration Forces Experienced by Space Processing Applications. Goddard Space Flight Center, Contract No. NAS5-23438, Mod. 23, ORI, Inc., Technical Report 1308, March 1978. (acceleration measurements, SPAR 1-4)

(3) Solidification of Pb-Sb Eutectic. In Descriptions of Space Processing Applications Rocket (SPAR) Experiments, edited by R. J. Naumann, NASA TM-78217, January 1979, pp. 3-4. (post-flight)

(4) Pond, R. B: Space Processing Rocket Experiment 74-5 - Space Solidification of Pb-Sb Eutectic. In Applications of Space Flight in Materials Science and Technology, proceedings of a conference held at the National Bureau of Standards, Gaithersburg, Maryland, April 20-21, 1977, pp. 25-39. (post-flight)


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**Experiment Origin:** Sweden
**Mission:** TEXUS 1
**Launch Date/Expt. Date:** December 1977
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Swedish TEXUS experiment module containing two gradient furnaces
**Builder of Processing Facility:** Swedish Space Corporation (SSC), Solna, Sweden

**Experiment:**
Dendritic Growth and Segregation Phenomena: Eutectic Al-Cu and Hypereutectic Al-Cu

The TEXUS 1 rocket carried two experiments initiated by Fredriksson to examine dendritic growth and segregation in metallic materials. The experiments were part of a series of investigations designed by the Principal Investigator to study low-gravity solidification phenomena. One of the two TEXUS 1 experiments (reported here) examined the influence of the low-gravity environment on the formation of eutectics and dendrites in two Al-Cu alloys. The other TEXUS 1 experiment (reported under Fredriksson, Dendritic Growth and Segregation Phenomena: Eutectic Sn-Zn (see this chapter)) examined the influence of the low-gravity environment on the formation of plate-like eutectics in the Sn-Zn alloy.

Two gradient furnaces within the TEXUS 1 Swedish Module were used to process two 8-mm diameter, 100-mm long aluminum-copper samples. Prior to the flight, an Al-Cu eutectic alloy was configured in one of the furnaces, and an Al-Cu hypereutectic alloy was configured in the other furnace. The furnaces were designed to melt and directionally solidify samples with melting points below 1000 $^{o}$C. During the flight, the two samples were (1) melted (in approximately 100 seconds) using a ceramic resistive heating element and then (2) directionally solidified (at approximately 25 mm/min) via a phase change heat sink in contact with one end of the sample. Control samples were processed under 1-g conditions for comparison.

Post-flight analysis revealed that the solidification rate of the eutectic low-gravity sample was greater by a factor of two over the 1-g eutectic sample. However, the "solidification acceleration" of the Earth sample was greater than that of the low-g sample. <Note: The meaning of "solidification acceleration" was unclear to the authors of this experiment summary.> Despite these differences in experimental parameters, the structures of the low-gravity and 1-g samples exhibited no significant differences. It was noted, however, that the lamellar spacing was smaller and the fault density was greater in the low-g processed sample. This difference in fault density was probably caused by the higher solidification rate of the space sample. When the fault density was plotted against the lamellar spacing for the 1-g and low-gravity samples, the curves were almost identical. Any differences in the curves were attributed to the large standard deviation in the measured data.

The differences between the solidification rate and the solidification acceleration of the hypereutectic low-gravity and 1-g samples were similar to those observed in the eutectic sample. Further, both the Earth-processed and low-g processed hypereutectic Al-Cu samples exhibited a columnar crystal zone sandwiched between two equiaxed zones. Reportedly, the equiaxed zones found in the low-gravity sample were caused by either (1) the lack of gravity-driven convection in the melt or (2) a difference in the thermal gradient between the two samples. The primary dendrite arm spacing through the columnar zones was constant for both samples, but larger for the space sample. The secondary arm spacing of both 1-g and low-gravity samples first decreased in the first-to-solidify section of the sample, and then increased. Reportedly, these results could not be explained and it was proposed that further experimentation was needed to determine the cause.

**Key Words:** Metals and Alloys, Binary Systems, Eutectics, Hypereutectics, Melt and Solidification, Resistance Heating, Directional Solidification, Thermal Gradient, Columnar Solidification, Equiaxial Zone, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Segregation, Solid/Liquid Interface, Growth Rate, Sample Microstructure, Dendritic Solidification, Dendritic Arm Spacing, Lamellar Structure, Fault Spacing

**Number of Samples:** two
**Sample Materials:** Hypereutectic aluminum copper (15% Cu), and eutectic aluminum copper
(Al*Cu*)
**Container Materials:** boron nitride
(B*N*)

**Experiment/Material Applications:**
The specific reason why the Al-Cu alloys were selected for this experiment was not detailed in the available publications. They may have been chosen because the eutectic alloys are well characterized and have been extensively investigated in the past. They also solidify at a low temperature and thus were suitable for processing in the employed gradient furnace.

**References/Applicable Publications:**
(1) Carlberg, T., Fredriksson, H., Sunnerkranz, P., Grahn, S., and Stenmark, L.: The Swedish TEXUS Experiment-A Technical Description and Some Preliminary Results. In Proceedings of Esrange Symposium, Ajaccio, April 24-29, 1978, ESA SP-135. (post-flight)

(2) Carlberg, T.: Solidification of Some Eutectic Alloys Under Microgravity Conditions and at 1G. In Proceedings of the 3rd European Symposium on Material Sciences in Space, Grenoble, April 24-27, 1979, ESA SP-142, pp. 221-231.

(3) Solidification of Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 212-213. (post-flight)

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**Experiment Origin:** Sweden
**Mission:** TEXUS 1
**Launch Date/Expt. Date:** December 1977
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Swedish TEXUS experiment module containing an ellipsoidal mirror furnace
**Builder of Processing Facility:** Swedish Space Corporation (SSC), Solna Sweden

**Experiment:**
Dendritic Growth and Segregation Phenomena: Eutectic Sn-Zn

Typically, the solidification structure of a Sn-Zn eutectic is that of a fan or plate-like structure (also common in Fe-C, Fe-$Fe_3C$, and Al-Si alloys). This structure "...is formed when fragments of a crystal are broken off, turned a few degrees and then grow to form new plates." (1, p. 336) This solidification mechanism is highly dependent on the temperature fluctuations and convective flow within the liquid. If the fan-like structure cannot form (quiescent melt conditions), then a normal eutectic structure results.

The TEXUS 1 rocket carried two experiments initiated by Fredriksson to examine dendritic growth and segregation in metallic materials. The experiments were part of a series of investigations designed by the Principal Investigator to study low-gravity solidification phenomena. The other of the two TEXUS 1 experiments (reported under Fredriksson, TEXUS 1, Dendritic Growth and Segregation Phenomena: Eutectic Al-Cu and Hypereutectic Al-Cu (see this chapter)) examined the influence of the low-gravity environment on the formation of eutectics and dendrites in two Al-Cu alloys. This TEXUS 1 experiment (reported here) examined the influence of the low-gravity environment on the formation of plate-like eutectics in the Sn-Zn alloy.

During the TEXUS 1 mission, a 1 $cm^3$ sample was isothermally melted and resolidified in one of the ten mirror furnaces which was contained in a section of the TEXUS Swedish experiment module. (The Swedish Module as a whole contained ten ellipsoidal isothermal mirror furnaces and two gradient furnaces.) The mirror furnaces were designed to melt and isothermally resolidify metallic samples with melting points below 1000 $^oC$. In each mirror furnace, radiation from two 150 W halogen lamps was focused by ellipsoidal mirrors on the sample. Heat was extracted from the sample by (1) conduction through the sample holder and (2) radiation to the furnace cavity.

A control sample was also processed under 1-g conditions for comparison. It was reported that 90% of the processed control sample exhibited a fan-like structure and approximately 10% of the sample exhibited a normal eutectic structure. This normal structure was located at the surface area at the center of the sample.

Both the fan-like and eutectic structures were present in the low-gravity sample. Approximately 40% of the structure exhibited the normal type of eutectic interspersed with plate-like Zn crystals. The remaining 60% of the structure exhibited the fan-like eutectic. The normal eutectic was somewhat coarser than that of the control sample. These differences were most likely due to the lack of convection which existed within the space sample.

**Key Words:** Metals and Alloys, Binary Systems, Eutectics, Melt and Solidification, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Segregation, Solid/Liquid Interface, Isothermal Processing, Heat Conduction, Radiative Cooling, Sample Microstructure, Surface Morphology, Dendritic Solidification, Platelike Structure, Fan-Like Structure, Sample Microstructure, Halogen Lamps

**Number of Samples:** one
**Sample Materials:** eutectic Sn-Zn
(Sn*Zn*)
**Container Materials:** unknown

**Experiment/Material Applications:**
The specific reasons why the Sn-Zn alloy was selected for this experiment were not detailed in the available publications.

**References/Applicable Publications:**
(1) Carlberg, T., Fredriksson, H., Sunnerkranz, P., Grahn, S., and Stenmark, L.: The Swedish Texus Experiment--A Technical Description and Some Preliminary Results, In Proceedings of Esrange Symposium, Ajaccio, April 24-29, 1978, ESA SP-135, June 1978, pp. 325-338.

(2) Carlberg, T.: Solidification of Some Eutectic Alloys Under Microgravity Conditions and at 1-g. In Proc. of the 3rd European Symposium on Material Science in Space, Grenoble, April 24-27, 1979, ESA SP-142, June 1979, pp. 221-231.

(3) Solidification of Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 212-213. (post-flight)

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**Experiment Origin:** Sweden
**Mission:** TEXUS 2
**Launch Date/Expt. Date:** November 1978
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Swedish TEXUS experiment module containing one ellipsoidal mirror furnace (MF 7)
**Builder of Processing Facility:** Swedish Space Corporation (SSC), Solna, Sweden

**Experiment:**
Isothermal Solidification of Eutectic Al-Si Alloy

This TEXUS 2 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2). The specific objective of the experiment was to study the solidification of a plate-like eutectic in the absence of gravity-induced convection.

A similar experiment, which examined the expected plate-like eutectic formation of a Sn-Zn eutectic was conducted during the earlier TEXUS 1 mission (see Fredriksson, TEXUS 1, Dendritic Growth and Segregation Phenomena, Eutectic Sn-Zn (this chapter)). It was reported that, under low-gravity conditions, the TEXUS 1 eutectic tended to form a normal, rather than plate-like, structure.

During the TEXUS 2 experiment, an Al-12.5 wt% Si alloy was melted and isothermally resolidified in one of the ellipsoidal mirror furnaces contained in the Swedish TEXUS experiment module. (See Fredriksson, TEXUS 1, Dendritic Growth and Segregation Phenomena, Eutectic Sn-Zn for a detailed discussion of the furnace.) A corresponding reference sample was processed under 1-g conditions for comparison.

Post-flight examination of the low-gravity processed sample revealed that three different structures formed in the sample. Nearly 80% of the sample exhibited the fan-like eutectic (compared to 60% for the Sn-Zn eutectic flown on TEXUS 1). A small percentage of the sample exhibited the normal eutectic, consisting of Si rods within an Al matrix. The remainder of the sample exhibited a "divorced eutectic structure" (1, p. 149) made up of Si particles and platelets distributed randomly throughout the Al matrix. The control (1-g processed) sample exhibited 90%

fan-like structure, 5% divorced eutectic, and 5% normal eutectic structure (located in the center).

Comparison of the thermal histories of the 1-g control sample and the low-g sample indicated that the solidification time of the 1-g sample was shorter (34 seconds) than that of the rocket sample (42 seconds). (A similar trend in the solidification times was also observed for the TEXUS 1 and 1-g Sn-Zn samples.) The increase in solidification time was probably due to the decreased contact (in low gravity) between sample and crucible wall.

Divorced eutectic will form when there exists a large number of Si particles in a localized area. The control sample, which was above the melt temperature longer than the low-gravity sample, probably had a greater opportunity for more complete dissolution of the Si. The increased amount of divorced eutectic in the low-g sample was probably caused by more undisolved Si in the sample, rather than a convective contribution. Reportedly, it could be argued that the dissolution of Si in the melt required more time because of the lack of convection. However, this could not be confirmed.

Since the amount of normal eutectic is the same in both samples, no influence of low-gravity conditions on the formation of the fan-like structure could be discerned.

**Key Words:** Metals and Alloys, Binary Systems, Eutectics, Melt and Solidification, Metallic Matrix, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Segregation, Dispersion, Particle Dispersion, Particle Distribution, Dissolution, Solid/Liquid Interface, Isothermal Processing, Heat Conduction, Radiative Cooling, Solidification Rate, Surface Morphology, Rod Structure, Platelike Structure, Platelet Habit, Fan-Like Structure, Sample Microstructure, Sample Detachment From Crucible, Crucible Effects

**Number of Samples:** one
**Sample Materials:** aluminum-silicon (12.5 wt% Si)
(Al*Si*)
**Container Materials:** unknown

**Experiment/Material Applications:**
The specific reasons why an Al-Si alloy was selected for this experiment were not detailed in the available publications.

**References/Applicable Publications:**

(1) Fredriksson, H.: Solidification Studies. In Shuttle/Spacelab Utilization, Final Report, Project TEXUS II, 1978, pp. 146-157. (post-flight)

(2) Carlberg, T.: Solidification of Some Eutectic Alloys Under Microgravity Conditions and at 1-g. In Proc. of 3rd European Symposium on Material Science in Space, Grenoble, April 24-27, 1979, ESA SP-142, pp. 221-231.

(3) Solidification of Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 216. (post-flight)

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**Experiment Origin:** Sweden
**Mission:** TEXUS 7
**Launch Date/Expt. Date:** May 1983
**Launched From:** ESRANGE, Kiruna Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Unknown
**Builder of Processing Facility:** Unknown

**Experiment:**
Dendritic Growth and Segregation Phenomena

This TEXUS 7 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5 (Chapters 14 and 17)).

No discussion of the objectives, setup, or results from this experiment could be located.

**Key Words:** Metals and Alloys, Melt and Solidification, Segregation, Dendritic Solidification, Dendrites

**Number of Samples:** unknown
**Sample Materials:** unknown
**Container Materials:** unknown

**Experiment/Material Applications:**
unknown

**References/Applicable Publications:**
Applicable publications could not be located.

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**Experiment Origin:** Sweden
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility
**Processing Facility:** Isothermal Heating Facility (IHF) furnace
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Dendritic Growth and Microsegregation (1ES314)

This Spacelab 1 experiment was one in a series of investigations designed by Fredriksson et al. to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7 (Chapters 14 and 17)).

A document published prior to the launch of Spacelab 1 (Reference (1)) reported that a binary Al-Cu alloy was to be investigated. The reference further reported that the objective of the experiment was to investigate the influence of system convection on (1) dendritic formation and (2) microsegregation.

No discussion of the experimental setup or post-flight results from this experiment could be located.

**Key Words:** Metals and Alloys, Binary Systems, Melt and Solidification, Segregation, Microsegregation, Convection, Dendritic Solidification, Dendritic Structure

**Number of Samples:** unknown
**Sample Materials:** Reportedly, binary Al-Cu alloy
(Al*Cu*)
**Container Materials:** unknown

**Experiment/Material Applications:**
unknown

**References/Applicable Publications:**

(1) Spacelab 1 Experiments. NASA/ESA, Marshall Space Flight Center, Document No. 15M983, p. 17. (preflight; brief discussion of experiment purpose)

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

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**Experiment Origin:** Sweden
**Mission:** TEXUS 10
**Launch Date/Expt. Date:** May 1984
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Unknown
**Builder of Processing Facility:** Unknown

**Experiment:**
Unidirectional Solidification of Sn-Al Samples

This TEXUS 10 experiment was one in a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1 (Chapters 14 and 17)).

No discussion of the experimental objectives, equipment setup, or post-flight results of this investigation could be located.

**Key Words:** Metals and Alloys, Binary Systems, Melt and Solidification, Directional Solidification

**Number of Samples:** unknown
**Sample Materials:** tin-aluminum alloy
(Sn*Al*)
**Container Materials:** unknown

**Experiment/Material Applications:**
unknown

**References/Applicable Publications:**
Applicable publications could not be located.

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**Experiment Origin:** Sweden/Canada
**Mission:** MASER 1
**Launch Date/Expt. Date:** March 1987
**Launched From:** Esrange, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Four identical platinum wound gradient furnaces housed within the SSC/Gradient Furnace (SSC/GF) module (This gradient furnace was "...a further development of the ESA/SSC TEXUS Gradient Furnace, featuring higher temperature operation and an improved heat sink control...." (1, p. 14))
**Builder of Processing Facility:** Swedish Space Corporation (SSC), Solna, Sweden

**Experiment:**
<u>Precipitation in Ag-Sn System/Microgravity Growth of Eutectics (Ag-Sn)</u>

<Note: Reference (1) (as listed below) reported that there were two experiments flown on MASER 1 in the Gradient Furnace Module: (1) Precipitation of Ag-Sn System (Principal Investigator, H. Fredriksson), and (2) Microgravity Growth of Eutectics (Ag-Sn) (Principal Investigator, R. W. Smith). In his review of this experiment summary, however, R. W. Smith reported that the two experiments were actually a collaborative experiment between the Principal and Co-Investigators listed above.>

Macrosegregation, "...a large degree of composition variation in an ingot in distances much larger than the dendritic arm spacing..." (2, p. 10) is believed to be related to (among other phenomena) (1) interdendritic fluid flow caused by buoyancy forces, and (2) sedimentation of components of differing densities within the melt. During processing in a low-gravity environment, these gravity-driven phenomena should be somewhat suppressed, thereby reducing the macrosegregation of the solidified system.

This MASER 1 experiment was one in a series of investigations designed by Fredriksson et al. to study low-gravity solidification (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 10, TEXUS 12, TEXUS 14a

(Chapters 13,14 and 17)). The overall objective of the experiment was to investigate the influence of gravity on the macrosegregation of silver-tin hypereutectic alloys. More specifically, the research was to examine sample macrosegregation as a function of Ag content.

Prior to the rocket launch, four alloys, each with a different Ag content (5%, 8%, 12%, and 20%), were prepared from high purity Sn(99.999%) and Ag(99.999%). The 5 mm diameter, 60 mm long samples (which were each housed in a stainless steel tube) were placed in four identical gradient furnaces within the MASER Gradient Furnace Module.

During the low-gravity phase of the flight, the samples were heated to a temperature 50 $^{o}$C above their respective liquidus temperatures. The 5% Ag content sample was heated to 270 $^{o}$C, the 8% sample to 330 $^{o}$C, the 12% sample to 360 $^{o}$C, and the 20% sample to 420 $^{o}$C. (The sample temperature was measured at three points along the specimen surface during the processing.) After the liquidus temperature was reached, heating was terminated. Heat was then extracted through a copper rod (attached at one end of the sample) into a heat sink (consisting of a copper can filled with paraffin wax).

After the rocket flight, the microstructure and composition profiles of the low-g processed samples were compared to ground-processed samples which had been similarly melted and solidified in (1) solid-down positions and (2) solid-up positions. Thermal analysis of the 12% Ag flight sample indicated that a radial heat loss strongly affected the growth. In addition, calculations of the temperature gradient at the eutectic front indicated that the gradient was "...around 15-20 $^{o}$C/cm for the solid-down ground samples and 2-10 $^{o}$C/cm for the flight and solid-up samples." (2, p. 13) Thermal analysis of the other Sn-Ag samples was not detailed.

It was reported that generally, "In [the Sn-Ag system] hypereutectic melts (up to 48 wt.% Ag), solidification starts with precipitation of the... [gamma]-phase ($Ag_3Sn$) and continues with a eutectic reaction at 221 $^{o}$C." (2, p. 10) "Due to [a] relatively low temperature gradient (2-15 $^{o}$C/cm) and noticeable radial heat loss, no directionality of the... [gamma] phase in [the flight] Sn-Ag samples... [could] be observed. The thickness and number of... [gamma particles] in the [ground-based] solid-down samples... [decreased] from bottom to the top while they remained constant in [ground-based] solid-up and flight samples. The structure of flight samples and solid up samples in this work... [were] very similar. The shape and morphology of... [gamma] phase... [did not change] in parallel samples. The experiment and calculations... [indicated] that the macrosegregation observed in this work... [was] caused by the interdendritic fluid flow only, and... can be explained with a mathematical model." (2, p. 15)

The mathematical model, which described the effect of solutal convection on macrosegregation, was based on the interdendritic fluid flow. The model system consisted of "A cylindrical tube with height H and base area A... filled with the melt with initial composition of $C_o$. During cooling... [gamma]-phase precipitates and the melt in the solid-liquid region (mushy zone) becomes enriched with tin with lower density with respect to the bulk. This initiates a flow from the mushy zone to the bulk. A mass balance for the enrichment of the melt due to solutal convection was written..." (2, p. 14) containing $C_a$ (the solute concentration in the interdendritic channels). Details of the analysis is presented on pp. 14-15 of Reference (2).

In summary, the analysis indicated that the macrosegregation depended primarily on the growth velocity and the gravity level. "For increasing growth velocity R, or decreasing B [a term containing gravity], the amount of macrosegregation decreases." (2, p. 15)

**Key Words:** Metals and Alloys, Melt and Solidification, Directional Solidification, Thermal Gradient, Binary Systems, Eutectics, Hypereutectics, Sedimentation, Macrosegregation, Precipitation, Solutal Gradients, Thermosolutal Convection, Buoyancy-Driven Convection, Heat Transfer, Buoyancy Forces, Density Difference, Composition Distribution, Growth Rate, Mushy Zone, Solid/Liquid Interface, Sample Microstructure, Interdendritic Fluid Flow, Dendritic Arm Spacing

**Number of Samples:** four
**Sample Materials:** (1) Sn - 5 wt.% Ag, (2) Sn - 8 wt.% Ag, (3) Sn - 12 wt.% Ag and (4) Sn - 20 Wt.% Ag
(Sn*Ag*)
**Container Materials:** thin walled stainless steel tubes

**Experiment/Material Applications:**
The Sn-Ag system was chosen for this research because "The differences in densities of the hypereutectic and eutectic melts are relatively large, and [a] large degree of macrosegregation is experienced under normal gravity. Furthermore, this system has a low melting point and low reactivity which make it suitable for sounding rocket experiments." (2, p. 10-11)

**References/Applicable Publications:**

(1) Zaar, J. and Änggard, K.: MASER and its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP), 32 pp. (post-flight; short description)

(2) Shahani, H., Fredriksson, H., and Smith, R. W.: The Effect of Gravity on the Macrosegregation in Tin Silver Alloys. Appl. Microgravity Tech. II (1989)1, pp. 10-16. (post-flight)

(3) Jönsson, R.: The Microgravity Program in Sweden- Emphasis on the Materials Rocket MASER. In 15th International Symposium on Space Technology and Science, Tokyo, Japan, May 19-23, 1986, pp. 2099-2110. (preflight)

(4) Zaar, J., Björn, L., and Jönsson, R.: Preliminary MASER 1 Results and the Evolution of the MASER Programme. In Proceedings of the 8th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 359-361. (post-flight; very short summary)

(5) Jönsson, R.: SSC Microgravity Sounding Rocket Program MASER. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

(6) Macrosegregation in Ag-Sn Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 216-217. (post-flight)

(7) Input Received from Principal Investigator R. W. Smith, October 1993.

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**Experiment Origin:** Sweden
**Mission:** MASER 2
**Launch Date/Expt. Date:** February 1988
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Swedish Space Corporation Gradient Furnace (SSC/GF) (The GF furnace was also used by Fredriksson during MASER 1)
**Builder of Processing Facility:** Swedish Space Corporation, Solna, Sweden

**Experiment:**
Primary Precipitated Crystal in Directional Solidification Al-Cu

"The mechanism of the columnar-equiaxed transition process is strongly influenced by the natural convection in the melt. Free crystals can grow ahead of the solidification front when the cooling rate is sufficiently high and the solidification rate sufficiently slow. These crystals can then block the columnar growth by adhering to the front. According to this theory, the columnar zone should be longer under microgravity." (4, p. 218)

This MASER 2 experiment was one of a series of investigations designed by Fredriksson to study low-gravity solidification phenomena (see Fredriksson, TEXUS 1, TEXUS 2, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 7, Spacelab 1, TEXUS 10, TEXUS 12, TEXUS 14a, MASER 1, TEXUS 14b (Chapters 13, 14, and 17)). The specific objective of the experiment was to study the directional solidification of two Al-Cu alloys.

Prior to the rocket launch, two 5-mm diameter, 60-mm long samples were prepared. The first sample consisted of Al-20% Cu and the second sample consisted of Al-43% Cu. <Note Reference (3) indicated that the samples were 5 mm in diameter; Reference (4) indicated that the samples were 4 mm in diameter.>

During the mission, each sample was processed in its own furnace within the MASER Gradient Furnace (GF) Module. (The GF module, which consisted of four furnaces in all, was also used for another MASER 2 experiment (see Fredriksson, MASER 2, "The Gradient Solidification of Immiscible Alloys Zn-Pb" (Chapter 17)).) During the low-gravity phase of the rocket, each sample was heated to 200 $^{o}$C above their liquidus temperature. (The sample temperature was measured during processing at three different locations in the graphite crucible wall.) After 2 minutes, the furnaces were turned off and the directional solidification phase initiated when a copper rod (which touched the bottom of the sample) came into contact with a phase-change heatsink. Heat was extracted through the copper rod and into the heatsink. (The heatsink contained paraffin wax). Reference samples were processed on Earth with the solid down.

Post-flight analysis of the MASER payload indicated that the furnaces operated nominally and that the samples were processed as expected. Metallographic investigations of the flight samples indicated that they exhibited (1) "very good directionality" and (2) seemingly little macrosegregation. More specifically, it was reported that "The Al-43 % Cu flight and ground samples exhibited a well oriented structure up to their top. The Al-20 % Cu flight sample was less oriented. The spacing of the... [theta]-phase in the Al-43% Cu and the secondary arm spacing in the Al-20% Cu were measured in cross-sections ever 1 cm along the samples. The differences between flight and ground processed samples were in the range of the precision of the measurements." (4, p. 218)

Temperature gradient calculations resolved from available flight thermal data illustrated "...that the temperature gradient drops continuously from around 100K/cm to around zero at the end of the sample." (3, Appendix 5, p. 2) Growth velocity calculations indicated that for the Al-20% Cu sample, the "...velocity was constant for almost half the length of the sample at around 400 micrometer per second. This value was increasing to around 2000 micrometer per second at the end of the sample." (3, Appendix 5, p. 2) Similar growth velocity calculations for the Al-43% Cu sample indicated that "...an almost constant growth velocity throughout the sample at around 600 micrometers per second." (3, Appendix 5, p. 3)

The lengths of the samples' columnar zones were presented:

For the 20% Cu sample:
flight: 1.7 cm
ground: 3.4 cm

For the 43% Cu sample
flight 5.4 cm
ground 5.2 cm

<Note: These results somewhat contradicted the theory that columnar zones should be longer in materials produced in a reduced gravity environment. The investigators appeared to explain this discrepancy in Reference (4) although their explanation was not entirely clear to the editors:>

"It is thought that due to the low gradients, the length of the 'mushy' zone was up to several cm and that an extended network was formed ahead of the front. Due to the small size of the samples, convection was hampered on ground and similar structures were obtained." (4, p. 218)

Very little additional information concerning this experiment could be located at this time.

**Key Words:** Metals and Alloys, Melt and Solidification, Directional Solidification, Binary Systems, Columnar to Equiaxed Transition, Macrosegregation, Precipitation, Buoyancy-Driven Convection, Thermal Gradient, Cooling Rate, Solidification Rate, Mushy Zone, Solidification Front Physics, Solid/Liquid Interface, Sample Microstructure, Dendritic Solidification, Dendritic Arm Spacing

**Number of Samples:** two
**Sample Materials:** (1) Al-20% Cu and (2) Al-43% Cu
(Al*Cu*)
**Container Materials:** graphite
(C*)

**Experiment/Material Applications:**
Direct applications were unspecified for the Al-Cu system.

**References/Applicable Publications:**
(1) Zaar, J. and Änggard, K.: MASER and its Effectiveness and Experimental Results. In: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP), 32 pp. (preflight)

(2) Zaar, J. and Dreier, L.: MASER 2 Final Report. Swedish Space Corporation, Solna, Sweden, RMLO/1-7, August 30, 1988. (postflight)

(3) Directional Solidification of Al-Cu Alloys. In MASER 2 Final Report, Swedish Space Corporation, Solna, Sweden RMLO/1-7, August 30, 1988, Appendix 5, pp. 2-3. (post-flight)

(4) Directional Solidification of Al-Cu Alloys. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 218-219. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 2
**Launch Date/Expt. Date:** November 1978
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 02 (large isothermal muffle furnace)
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Alloy Formation

Solution calorimetry is the most accurate method available to determine heats of formation of metallic phases. During the calorimetry (1) a metallic phase is dissolved in a metallic melt, (2) the change in temperature of the melt is measured, and (3) the heat of solution is determined. Similarly, individual elements of the metallic phase under investigation are dissolved in the melt and the heat of solution for the elements is determined. The difference between these two heats of solution is the heat of formation of the phase.

If the metals (for example, chemical elements from the periodic table in (1) group IV, (2) group V, or (3) the Rare-Earth section) react with the crucible, then the heat of solution cannot be determined accurately because it is superimposed upon the heat of reaction. In the low-gravity environment, the calorimetry can take place in a crucible free system, thus eliminating these difficulties.

This TEXUS 2 experiment was the first in a series of investigations designed by Haessner et al. to study the effects of the low-gravity environment on the heats of formation of metallic phases. The specific objectives of the experiment were to determine (1) the time required for a solid metal sample to dissolve in a liquid melt, (2) the influence of the released heat of reaction on the solution, (3) the factors which disturb the measurement process, and (4) the diffusion-inhibitive internal surfaces which affect the process.

The experiment was performed in the TEXUS Experiment Module TEM 02 large muffle furnace. The furnace contained three steel plates (20 mm apart) attached to a central rod. Each plate held three Al samples contained in $Al_2O_3$ rings such that the Al samples protruded above the rings. Six different metals were chosen as alloying samples for the aluminum: copper, nickel, titanium, niobium, tantalum, and molybdenum. An alloying specimen was configured above each Al sample in a specimen holder. (The specimen holders were also attached to the central rod.) The entire apparatus was contained in a quartz tube. Video equipment recorded the experiment.

Prior to the rocket launch, the furnace was preheated to 600 $^{o}$C. After launch, a battery was activated which further heated the furnace to between 796 and 816 $^{o}$C and the Al samples melted forming liquid drops. Once the desired temperature and low-gravity conditions were achieved, the solid, pure metal alloying samples were pushed into the liquid Al drops and held for 190 seconds. Cooling was then initiated (while still under low-gravity conditions) by the introduction of a gas along the outside of the quartz furnace chamber. This external cooling procedure was employed to prevent damage of the specimens by the gas. The measured cooling rates were between 5 and 10 $^{o}$C/sec.

Post-flight examination of the space samples indicated that "[the] space experiment [had] not fulfilled its expectations...." (1, p. 100). A leak in the quartz furnace chamber allowed cooling gas to be introduced into the furnace chamber resulting in two samples being blown from their holders. The remaining samples had been deformed by the force of the gas. The gas had also acted to separate the alloying specimens from the Al drops. Therefore, "[o]ne of the most important requirements of the solution experiment, i.e. solidification of alloys under gravity-free conditions was no longer fulfilled." (1, p. 99)

Metallographic and X-ray micro-analysis of the flight samples illustrated that "...no detectable localized alloying effect took place." (1, p. 98) This was probably due to the fact that "[t]he temperatures registered at the various sensing points did not indicate the actual specimen temperature...." (1, p. 99) It appears that the Al drops were not completely molten and merely deformed when the alloy specimen was inserted. Heat conduction by the sample holders would have prevented any further heating and subsequent melting of the Al drop. Examination by scanning electron microscopy did not contradict this deduction. It was further reported that the presence of a thin oxide layer on the Al samples could have also prevented alloying.

**Key Words:** Metals and Alloys, Binary Systems, Multiphase Media, Melt and Solidification, Solution Calorimetry, Calorimetric Measurements, Heats of Formation, Heat of Solution, Heat of Reaction, Drops, Drop Formation, Dissolution, Diffusion, Liquid/Gas Interface, Solid/Liquid Interface, Cooling Rate, Heat Conduction, Highly Reactive Materials, Containerless Melt, Material Interaction With Containment Facility, Crucible Effects, Oxide Layer, Thin Films, Contamination Source, Gas Injection, Sample Deformation, Processing Difficulties

**Number of Samples:** nine
**Sample Materials:** Pure metals dissolved in liquid aluminum. Dissolving metals: copper, nickel, titanium, niobium, tantalum, and molybdenum.
(Al*Cu*, Al*Ni*, Al*Ti*, Al*Nb*, Al*Ta*, Al*Mo*)
**Container Materials:** alumina ($Al_2O_3$ rings were used to hold the molten aluminum)
(Al*O*)

**Experiment/Material Applications:**
It is difficult to determine the heats of formation of melts which react with their crucible materials using the solution calorimetry method. It was proposed that in the low-gravity environment, the calorimetry method could be performed in a crucible-free system.

The specific reasons why the various metal combinations were chosen for the experiment were not detailed in the available publications.

**References/Applicable Publications:**
(1) Haessner, F. and Hettwer, K. J.: Alloy Formation. In Shuttle/Spacelab Utilization Final Report Project TEXUS II, 1978, pp. 91-108. (post-flight)

(2) Haessner, F. and Hettwer, K. J.: Legierungsbildung unter Schwerelosigkeit. May 1979, 01 QV 168-ZA-SN-SLN 7785/2, DFVLR/BPT, Köln, Porz, Germany. (in German)

(3) Haessner, F., Hemminger, W., and Lukas, H. L.: Solution Caloremetry in Zero Gravity for the Determination of Enthalpies of Formation of Reactive Metals. In AIAA/ASME 1974 Thermophysics and Heat Transfer Conference, Boston, Massachusetts, July 15-17, 1974, AIAA Paper #74-666. (preflight)

(4) Input received from Experiment Investigator, August 1988 and August 1993.

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**Co-Investigator(s):** None
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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 5
**Launch Date/Expt. Date:** April 1982
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 02-1 (large isothermal muffle furnace)
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Alloy Formation

This TEXUS 5 experiment was the second in a series of investigations designed by Haessner et al. to study the effects of the low-gravity environment on the heats of formation of metallic phases (see Haessner, TEXUS 2). The specific objectives of the experiment were to investigate (1) the rate of dissolution of a solid piece of metal in a solvent metal under low-gravity conditions, (2) the influence of surface layers on this dissolubility, (3) the type of microstructure formed under low-gravity conditions, and (4) the heat of reaction influence on the process of dissolution.

The experiment was conducted in the TEXUS Experiment Module TEM 02-1 large isothermal muffle furnace. <Note: No discussion of the experimental apparatus and sample configuration (as specifically configured for TEXUS 5) was provided in the available (English) publications.> During the mission, various metals (see the Sample Materials section below) were dissolved in liquid copper-titanium. Terrestrial experiments were performed under 1-g conditions using the same type of boundary conditions as the flight experiment.

Post-flight examinations of both the flight and terrestrial samples included macroscopic and microscopic inspections of the microstructure as well as SEM and X-ray micro-analysis. The results of the ground and flight experiments were compared and may be located in a table in Reference (4). No discussion of the results or conclusions accompanied the table.

<Note: Further information concerning this experiment (published in English) could not be located at this time. The Principal Investigator reported that additional information (published in German) can be found in Reference (5).>

**Key Words:** Metals and Alloys, Binary Systems, Multiphase Media, Ternary Systems, Melt and Solidification, Solution Calorimetry, Calorimetric Measurements, Heats of Formation, Heat of Solution, Heat of Reaction, Drops, Drop Formation, Dissolution, Diffusion, Solid/Liquid Interface, Cooling Rate, Sample Microstructure, Highly Reactive Materials, Material Interaction With Containment Facility, Crucible Effects

**Number of Samples:** thirty-five
**Sample Materials:** Metals and alloys dissolved in liquid copper-titanium were: cobalt, iron, nickel, silicon, titanium, boron, aluminum-titanium, aluminum-vanadium, cobalt-titanium, cobalt-zirconium, iron-titanium, iron-zirconium, nickel-titanium, nickel-zirconium, silicon-titanium, silicon-zirconium.
(Cu*Ti*Co*, Cu*Ti*Fe*, Cu*Ti*Ni*, Cu*Ti*Si*, Cu*Ti*, Cu*Ti*B*, Cu*Ti*Al*Ti*, Cu*Ti*Al*V*, Cu*Ti*Co*Ti*, Cu*Ti*Fe*Ti*, Cu*Ti* FeZr*, Cu*Ti*Ni*Ti*, Cu*Ti*Ni*Zr*, Cu*Ti*Si*Ti*, Cu*Ti*Si*Zr*)
**Container Materials:** It is not clear if sample containers were employed for this experiment.

**Experiment/Material Applications:**
See Haessner, TEXUS 2.

The specific reasons why the various metal combinations were selected for this experiment were not detailed in the available publications.

**References/Applicable Publications:**

(1) Haessner, F. and Wehr, P.: Legierungsbildung unter Schwerelosigkeit (LEBIUS). December 1982, 01 QV 2098-ZA-SLN-7910, Bundesminister für Forschung und Technologie. (in German) (This report could possibly be ordered from the DFVLR/BPT, Köln-Porz, Germany.)

(2) Haessner, F., Hemminger, W., and Lukas, H. L.: Solution Calorimetry in Zero Gravity for the Determination of Enthalpies of Formation of Reactive Metals. In Proceedings AIAA/ASME 1974 Thermophysics and Heat Transfer Conference, Boston Massachusetts, July 15-17, 1974, AIAA Paper #74-666. (preflight)

(3) Input received from Experiment Investigator, August 1988 and August 1993.

(4) Alloy Formation. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 208-209. (post-flight)

(5) Haessner, F., Hettwer, K. J., and Wehr, P.: Legierungsbildung unter Schwerelosigkeit. In TEXUS 1-10, Zusammenfusseneler Bericht, Deutsche Forschungsanstalt für Luft-und Raumfahrt, im Auftragoles BMFT, 1990, pp. 87-91.

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**Experiment Origin:** Unknown
**Mission:** TEXUS 2
**Launch Date/Expt Date:** November 1978
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** One ellipsoidal Mirror Furnace (MF 4)
**Builder of Processing Facility:** Swedish Space Corporation (SSC), Solna, Sweden

**Experiment:**
<u>Solidification of Al-Ti</u>

<Note: The specific objectives of this experiment could not be identified from the available literature. Further, very little information could be found concerning the equipment setup.>

Reference (1) (as listed below) indicated that a Al-Ti solidification experiment was performed on TEXUS 2 in the Mirror Furnace designated as MF 4. It was reported that the "...furnace did not start and therefore the Al-Ti sample did not melt." (1, p. 215)

No further information concerning this experiment could be located at this time.

**Key Words:** Metals and Alloys, Melt and Solidification, Binary Systems, Furnace Malfunction, Sample Not Processed As Planned

**Number of Samples:** unknown, probably one
**Sample Materials:** aluminum-titanium alloy
(Al*Ti*)
**Container Materials:** unknown

**Experiment/Material Applications:**
unknown

**References/Applicable Publications:**

(1) Grahn, Civ. Ing. S.: Swedish Experiment Module. In Shuttle/Spacelab Utilization, Final Report, Project TEXUS II, 1978, pp. 214-222. (post-flight)

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**Co-Investigator(s):** Unknown
**Affiliation(s):** Unknown

**Experiment Origin:** Unknown
**Mission:** TEXUS 2
**Launch Date/Expt Date:** November 1978
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Gradient Furnace (GF 2)
**Builder of Processing Facility:** Swedish Space Corporation (SSC), Solna, Sweden

**Experiment:**
Solidification

<Note: The specific objectives of this experiment and the materials which were processed could not be identified from available literature. Further, very little information could be found concerning the equipment setup or post-flight results.>

Reference (1) (as listed below) indicated that a solidification experiment was performed on TEXUS 2 in the Gradient Furnace designated as GF 2. However, it was reported that the furnace temperature only reached 400 °C "...while the target temperature was 700 °C. Power was commanded off for some unknown reason at T+26 seconds. The Sn phase of the sample has leaked out of a crack in the crucible but has only caused short circuits for brief moments. The boronnitride spray applied to the heater to avoid a repetition of the corresponding problems during the TEXUS I flight prevented a major short to occur.

"Further analysis of other thermocouple readings are needed before a complete assessment of this furnace can be made. The sample is presently being analyzed, but it is not yet clear if it melted completely." (1, p. 217)

No further information concerning this experiment could be located at this time.

**Key Words:** Metals and Alloys, Melt and Solidification, Liquid Leakage, Furnace Malfunction, Processing Difficulties

**Number of Samples:** unknown, possibly one
**Sample Materials:** unknown, included Sn
(Sn*)
**Container Materials:** unknown

**Experiment/Material Applications:**
unknown

**References/Applicable Publications:**

(1) Grahn, Civ. Ing. S.: Swedish Experiment Module. In Shuttle/Spacelab Utilization, Final Report, Project TEXUS II, 1978, pp. 214-222. (post-flight)

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**Experiment Origin:** Unknown, probably Sweden
**Mission:** TEXUS 2
**Launch Date/Expt Date:** November 1978
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Swedish experiment module containing one ellipsoidal mirror furnace (MF 8)
**Builder of Processing Facility:** Swedish Space Corporation (SSC), Solna, Sweden

**Experiment:**
Isothermal Solidification of Eutectic Al-Si Alloy

This TEXUS 2 experiment was designed to study the low-gravity solidification of eutectic alloys. It appears that the experiment may have been related to an experiment by Fredriksson on TEXUS 2, Isothermal Solidification of Eutectic Al-Si (see Fredriksson, TEXUS 2, Eutectic Al-Si Alloy (this chapter)).

A description of the processing facility can be found under Fredriksson, TEXUS 1, Dendritic Growth and Segregation Phenomena: Eutectic Sn-Zn (this chapter).

It was reported that ellipsoidal mirror furnace MF 8 "...did not start and therefore the Al-Si sample did not melt." (1., p. 216)

No further information concerning this experiment could be located at this time.

**Key Words:** Metals and Alloys, Binary Systems, Eutectics, Melt and Solidification, Isothermal Processing, Solid/Liquid Interface, Furnace Malfunction, Sample Not Processed As Planned

**Number of Samples:** one
**Sample Materials:** aluminum-silicon alloy
(Al*Si*)
**Container Material:** unknown

**Experiment/Material Applications:**
Unknown

**References/Applicable Publications:**

(1) Grahn, Civ. Ing. S.: Swedish Experiment Module. In Shuttle/Spacelab Utilization, Final Report, Project TEXUS II, 1978, pp. 214-222. (post-flight)

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**Experiment Origin:** France
**Mission:** TEXUS 4
**Launch Date/Expt. Date:** May 1981
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 03
**Builder of Processing Facility:** Deutsche Forschungs- und Versuchsanstalt für Luft- und Raumfahrt (DFVLR), Germany <Note: The DFVLR is now called the Deutsche Forschungsanstalt für Luft- und Raumfahrt (DLR).>

**Experiment:**
Directional Solidification of $Al_2$-Cu-Al Eutectics

This TEXUS 4 experiment was the first in a series of investigations designed by Favier et al. to study the directional solidification of eutectic alloys under low-gravity conditions.

<Note: Very little information concerning this TEXUS 4 experiment (published in English) could be located. Reference (4) reported that the experimental results were summarized in Reference (3), Section 3.2.2. However, this section appears to be discussing results from the $Al_2$-Cu-Al system processed during (1) Favier's later TEXUS 6 mission and (2) Favier's later Spacelab mission. (Section 3.2.2, which briefly mentions TEXUS results, does not specifically state which TEXUS mission it is discussing but does list a reference citing TEXUS 6 results.)>

Reference (4) briefly summarized the experimental objectives and results as follows: (1) the objective of the experiment was to study the influence of natural convection on the microstructure of lamellar eutectics and (2) experimental results indicated that there was (a) no effect on lamellar spacing and (b) a small effect on perfection.

No additional discussions of the specific TEXUS 4 experimental setup, or results (published in English) could be located. However, Reference (2) mentions these subjects in conjunction with the subsequent TEXUS 6 experiment.

See Favier, TEXUS 6 (this chapter).

<Note: An English translation of Reference (1) (as listed below) was not available at the time this experiment summary was prepared.>

**Key Words:** Metals and Alloys, Eutectics, Binary Systems, Melt and Solidification, Directional Solidification, Solid/Liquid Interface, Convection, Sample Microstructure, Lamellar Eutectics

**Number of Samples:** unknown
**Sample Materials:** $Al_2$-Cu-Al (33 wt.% Cu)
(Al*Cu*)
**Container Materials:** unknown

**Experiment/Material Applications:**
See Favier, TEXUS 6.

**References/Applicable Publications:**
(1) de Goer, J., Favier, J. J., and Le Maguet, R.: Directional Solidification of the $Al_2$CuAl Eutectic System in a Sounding Rocket; Analysis of the TEXUS IV Experiment; Preparation of the TEXUS V-VI Experiment. DMG Report No. 41/82. (in French)

(2) Favier, J. J. and de Goer, J.: Directional Solidification of $Al_2$-Cu-Al and $Al_3$-Ni-Al Eutectics During TEXUS Rocket Flight. Translation of D.M.G. Report No. 93/83, Centre d'Etudes Nucléaires de Grenoble, Grenoble, France, November 1983, pp. 1-56, NASA TM-77552. (post-flight; mainly discusses TEXUS 6 results)

(3) Favier, J. J. and de Goer, J.: Directional Solidification of Eutectic Alloys. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 127-133. (post-flight results, TEXUS mission unclear)

(4) Input received from Denis Camel (CENG, Grenoble), August 1993.

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**Experiment Origin:** France
**Mission:** TEXUS 6
**Launch Date/Expt. Date:** May 1982
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 03
**Builder of Processing Facility:** Deutsche Forschungs- und Versuchsanstalt für Luft- und Raumfahrt (DFVLR), Germany <Note: The DFVLR is now called the Deutsche Forschungsanstalt für Luft- und Raumfahrt (DLR).>

**Experiment:**
Directional Solidification of (1) $Al_3$-Ni-Al Eutectics and (2) $Al_2$-Cu-Al Eutectics

When a binary alloy of eutectic composition is directionally solidified, an organized, anisotropic structure may result. This structure can take two forms: (1) a lamellar stacking of two phases of defined composition (as in an $Al_2$-Cu-Al alloy) or (2) a fiber cluster of one phase in a matrix of another phase (as in an $Al_3$-Ni-Al alloy). On Earth, the structure (e.g., continuity of eutectic phase) of these eutectic alloys is compromised by gravity-induced convective effects.

This TEXUS 6 experiment was the second in a series of investigations designed by Favier et al. to study the directional solidification of eutectic alloys under low-gravity conditions (see Favier, TEXUS 4). The specific objectives of this experiment were to:

(1) prove the feasibility of a directional solidification study during a sounding rocket flight

(2) determine if the following relation is valid for space processed materials:
$L^2 V = k$
where:
L = inter-lamellar or inter-fiber distance (microns)
V = solidification front velocity (cm/hr)
K = solidification constant

(3) determine if K is the same for 1-g and low-gravity processed materials

(4) determine if the gravity level affects the regularity of either type of eutectic structure

During the mission, the TEXUS Experiment Module TEM 03 was used to melt and resolidify one $Al_2Cu$-Al sample and one $Al_3Ni$-Al sample. It was reported that the TEM 03 furnace was also used during the TEXUS 4 mission. However, slight modifications were instituted for the TEXUS 6 experiment. These included (1) shortening the sample length to 70 mm, (2) including a volume compensation system, and (3) modifying the thermal cycle to provide a 20 to 25 mm seed (unmelted portion).

It was reported that the rocket flight and sample thermal conditions were "virtually nominal" throughout the mission. However, "The actual flight took place on May 8, 1983, but the day before, the initial count down had to be canceled 5 minutes before blast-off, for meteorological reasons." (1, p. 6, translation) It appears that prior to the May 7th mission cancellation, the samples were subjected to thermal processing: melting, thermal soak, controlled solidification, and natural solidification (see Reference (1) for further details of this anomaly).

It appears that during the May 8th flight, the samples were subjected to the same thermal processing. It was reported that cooling of the $Al_3Ni$-Al sample was not according to plan during the actual flight. "...the TS4A instruction (cartridge base 'cold') could not be complied with. Consequently, the cooling was slower and the thermal gradient was smaller than expected." (1, p. 6, translation) <Note: The exact meaning of this quote was not clear to the editors.> (Reference (1) contains details of the thermal performance and analysis.)

Post-flight examination of the two samples included (1) gamma-radiography, (2) light metallography, and (3) laser beam diffraction (Reference (3) contains details of the diffraction procedure). These analysis techniques yielded the following results:

(1) Reportedly, low-gravity processing did not provide more regular lamellar ($Al_2Cu$-Al sample) or fibrous ($Al_3Ni$-Al sample) structures. Rather, the structures were more irregular, "...which [is] certainly linked to the rapid destabilization of the solidification front during the microgravity cycle." (1, p. 28, translation) This result contradicted that from Hasemeyer's Copper-Aluminum Eutectic Skylab experiment (M566) (see Hasemeyer, Skylab SL-3 and Skylab SL-4 (this chapter)). The fiber spacing in the $Al_3Ni$-Al sample did exhibit an increase when compared to the corresponding 1-g samples.

(2) "The constitutional supermelting at the destabilization threshold was evaluated for the Al-Cu sample and Al-Ni sample at (delta)T* = 1.15 and 1.4 K respectively. This supermelting is undoubtedly associated with... [the] presence of noncontrolled impurities in the liquid baths,... [introduced] when the charges were being prepared." (1, p. 28, translation) <Note: The exact definition of T* was not presented in the available publications.>

(3) Information concerning the growth conditions of the samples was derived from (a) thermocouple readings from the cartridge surface and (b) thermal modeling of the cartridge and sample. This information and microstructural studies on the samples led to the following conclusions: (a) the relation between the lamellar or interfiber spacing, L, and the interface velocity, V, was the same for 1-g conditions and low-gravity conditions and (b) the gravity level does not effect the value of the solidification constant, K.

**Key Words:** Metals and Alloys, Eutectics, Binary Systems, Melt and Solidification, Directional Solidification, Solid/Liquid Interface, Solidification Front Physics, Seed Crystals, Buoyancy-Driven Convection, Growth Rate, Thermal Soak, Thermal Gradient, Composition Distribution, Multiphase Media, Impurities, Sample Microstructure, Lamellar Eutectics, Lamellar Structure, Fiber Eutectics, Inter-Fiber Spacing, Volume Compensation

**Number of Samples:** two
**Sample Materials:** $Al_2$-Cu-Al; $Al_3$-Ni-Al
(Al*Cu*, Al*Ni*)
**Container Materials:** unknown

**Experiment/Material Applications:**
One of the objectives of this experiment was to investigate the effect of gravity on the formation of different types of eutectic structures. Therefore, an Al-Cu alloy (lamellar eutectic structure) and an Al-Ni alloy (fibrous eutectic structure) were selected. These materials have been well characterized; a large amount of data exists concerning their solidification behavior.

**References/Applicable Publications:**

(1) Favier, J. J. and de Goer, J.: Directional Solidification of $Al_2$-Cu-Al and $Al_3$-Ni-Al Eutectics During Texus Rocket Flight. NASA TM-77552, 1984, 55 pp.

(2) Favier, J. J. and de Goer, J.: Directional Solidification of Eutectic Alloys. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 127-133. (post-flight results)

(3) Favier, J. J., Morlevat, J. P., and Duvernoy, J.: Method of Analysis of the Regularity of Lamellar Eutectic Structures by Diffraction of a Laser Beam. In Metallurgical Transactions B, Vol. 14B, March 1983, pp. 105-108. (discusses sample characterization technique)

(4) Directional Solidification of Eutectics. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 220-221. (post-flight)

(5) Input received from Denis Camel (CENG, Grenoble), August 1993.

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**Experiment Origin:** France
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Material Science Double Rack (MSDR)
**Processing Facility:** Gradient Heating Facility (GHF) Furnace
**Builder of Processing Facility:** Centre National d'Etudes Spatiales (CNES), Toulouse, France

**Experiment:**
Unidirectional Solidification of Eutectics Alloys (1ES317)

This Spacelab 1 experiment was the third in a series of investigations designed by Favier et al. to study the directional solidification of eutectic alloys under low-gravity conditions (see Favier, TEXUS 4, TEXUS 6). The specific objectives of the research were (1) to examine different types of eutectic structures produced under 1-g and low-gravity conditions and compare their characteristic (a) phase distribution, (b) regularity, and (c) crystallography and (2) to examine the results in terms of eutectic growth theories.

Prior to flight, nine eutectic materials were prepared by vacuum casting: (1) three samples of Ag-17.6 wt.% Ge, (2) three samples of Al-33.053 wt.% Cu, and (3) three samples of Al-6.158 wt.% Ni. (The Al-Cu material was subsequently directionally solidified (on Earth) in a horizontal Bridgman furnace under an Ar atmosphere. This extra processing step produced a monograin sample.) The nine samples (each 5 mm ID, 150 mm long) were configured in three cartridges (three samples per cartridge). Each cartridge contained three thermocouples for temperature and temperature-gradient determination. A volume change compensation system was included in each cartridge.

During the Spacelab 1 mission, the Gradient Heating Facility (GHF) was used to melt and directionally solidify the samples (see Reference (3) for details of apparatus). The thermal profile of the samples included (1) heat-up (approximately 1.75 hours), (2) unidirectional solidification (approximately 3.25 hours), and (3) cool down. Reportedly, no thermal soak time was allowed to prevent "...deposition of one of the two eutectic phases on the initial [solidification] interface." (1, p. 128) (See Reference (1) for further details concerning experimental setup and thermal behavior of the furnace.) Samples were also processed under 1-g conditions for comparison.

Post-flight, metallographic examination (longitudinal cuts and cross-sections) was performed on each sample. The results were reported as follows:

Ag-Ge samples: No significant differences between the 1-g and low-gravity samples were observed. Primary Ge crystals were observed on the surface of the 1-g sample. These crystals were attributed to heterogeneous nucleation at the crucible wall. A silver enriched zone was detected at the initial interface of the low-gravity sample. This was reportedly caused by thermomigration during the period of thermal re-equilibration.

Al-Cu samples: Spherical bubbles (500 micron to 2 mm) were located in the last third of the low-gravity samples. These bubbles were probably formed by the argon absorbed during the sample preparation. No difference in lamellar regularity was observed in the 1-g and low-gravity samples. The value of the constant obtained from the $L^2$ V equation (see Favier, TEXUS 6 for a discussion of this equation) was 1.06 x $10^{-10}$ CGS (+/- 5%). This value agreed with that obtained from the TEXUS 6 experiments (1.08 x $10^{-10}$ CGS). Deviations from this relation were attributed to (1) growth velocity uncertainty at sample ends, (2) large uncertainty at the beginning of regrowth in the relative position of the cross-sections, and (3) the transient regime for the lamellar spacing between seed and steady state. Both 1-g and low-gravity samples demonstrated these transients.

Al-Ni samples: The initial seed material of the 1-g and low-gravity samples was non-oriented, multi-grained. After about 3 cm of the rocket samples had been processed, a limited number of grains remained. These grains exhibited broken lamella and then needles. The 1-g samples contained this transition point at approximately 2 cm. There was no noticeable difference in fiber pattern regularity. However, the low-gravity processed samples contained a 15% larger value of inter-fiber spacing. This result also confirmed the TEXUS 6 findings.

In conclusion, no major differences between the 1-g and low-gravity Ag-Ge samples (irregular eutectic structure) were noted. Since the lamellar structure of the Al-Cu samples were the same for the 1-g and low-gravity samples, "...the convective fluctuations are therefore not the principal... [origin] of fault formation. However for the fiberlike structure ($Al_3Ni$-Al), there is obviously an effect: the structure in space is larger than on the ground." (1, p. 130) When these results were compared to other low-gravity experiments it was concluded that "...only the fiberlike systems are affected by gravity, but that for other systems studied... [see Pirich, Chapter 17, Spar 6 (Mn-Bi); Müller, Chapter 14, Spacelab 1 (InSb-Sb)]... space growth gives rise to finer structures." (1, p. 131)

A simple model was proposed (see Reference (1)) which accounted for an eventual shift of the initial liquid composition. Reportedly, this approach may explain (semi-quantitatively) convective effects on the Jackson-Hunt theory of coupled growth.

**Key Words:** Metals and Alloys, Eutectics, Binary Systems, Melt and Solidification, Directional Solidification, Solid/Liquid Interface, Seed Crystals, Buoyancy-Driven Convection, Nucleation, Heterogeneous Nucleation, Growth Rate, Thermal Gradient, Multiphase Media, Sample Microstructure, Lamellar Eutectics, Lamellar Structure, Fiber Eutectics, Inter-Fiber Spacing, Grain Structure, Needles, Surface Morphology, Fault Spacing, Thermomigration, Crucible Effects, Bubbles, Bubble Formation, Volume Compensation

**Number of Samples:** nine
**Sample Materials:** three each of (1) Ag-17.60 wt.% Ge; (2) Al-33.053 wt.% Cu; (3) Al-6.158 wt.% Ni
(Ag*Ge*, Al*Cu*, Al*Ni*)
**Container Material:** unknown

**Experiment/Material Applications:**
"The eutectic systems are choice cases for investigating the solidification theory in a particular environment since, unvarying in the thermodynamic sense, their growth temperature remains constant during the whole process. Moreover due to its particular two phases structure affected by the growth fluctuations, the local growth conditions can be deduced from the analysis of the structures." (1, p. 127)

These systems were also selected because they each produce different types of eutectic structure. The Ag-Ge sample produces an irregular eutectic structure, the Al-Cu sample produces a lamellar eutectic structure, and the Al-Ni sample produces a fibrous eutectic structure.

**References/Applicable Publications:**

(1) Favier, J. J. and de Goer, J.: Directional Solidification of Eutectic Alloys. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 127-133.

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(3) Cambon, G., Castets, B., Boutemy, O., Giegel, J. P., Aubron, R., Boddaert, M., and Benoit, J.: Gradient Heating Facility Behavior. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 115-119. (post-flight, experimental apparatus)

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(5) Input received from Denis Camel (CENG, Grenoble), August 1993.

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**Experiment Origin:** France
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Material Sciences Double Rack (MSDR)
**Processing Facility:** Gradient Heating Facility (GHF)
**Builder of Processing Facility:** CNES (Centre National d'Etudes Spatiales), Toulouse, France

**Experiment:**
Dendritic Solidification of Aluminum-Copper Alloys (WL GHF-04)

This Spacelab D1 experiment was the fourth in a series of investigations designed by Favier et al. to study the directional solidification of eutectic alloys under low-gravity conditions (see Favier, TEXUS 4, TEXUS 6, Spacelab 1). The specific objectives of the experiment were to (1) check existing theories on dendritic solidification by extending the solidification parameters under which dendritic formation in a purely diffusive regime will occur and (2) perform studies (theoretical and experimental) concerning the effects of convection on dendritic solidification which may provide added control of ground-based processes.

During the Spacelab mission, three Al-Cu samples containing 1 wt.%, 26 wt.%, and 40 wt% copper were melted and directionally solidified using the Gradient Heating Facility (GHF). Each sample was contained within a $ZrO_2$ cartridge. The sample container was arranged to assure (1) minimal radial temperature gradients and (2) reproducible cold-side heat exchanges. Ground-based experiments were performed such that solidification occurred at a low rate in different directions with respect to gravity.

Post-flight examination of all samples (ground- and space-processed) included (1) density measurements, (2) radiography, (3) metallography, (4) quantimetry, (5) Laue patterns, and (6) electron microprobe determinations. The objectives of these analysis techniques were to determine (1) the longitudinal and radial macrosegregation, (2) the microsegregations at different scales (see References (1) and (5)), (3) the primary interdendritic spacing, (4) the crystallographic orientation of the grains during solidification, and (5) the development, direction, and morphology of dendritic arms relative to solidification conditions and crystallographic orientation. The following results were reported:

Ground-based samples: Three different types of segregation were observed which depended on the orientation of the density gradient (in the interdendritic region) relative to the gravity vector:

(1) Vertical solutally stabilizing case: No significant longitudinal segregation but "...radial segregation is so important that the eutectic concentration is reached...." (1, p. 129)

(2) Vertical solutally destabilizing case: Significant longitudinal segregation and no radial segregation.

(3) Horizontal solidification: Longitudinal and radial of segregation were present.

The above results indicated that natural convective effects cannot be eliminated despite changes in solidification direction (relative to the gravity vector). "In all cases, the measured primary interdendritic spacings (lambda), which range between 350 and 450... [microns]..., are much lower than the value of 1400... [microns]... predicted by extrapolating the scaling laws verified at higher solidification rates...." (1, p. 130)

The low-gravity processed samples were significantly different from the ground processed samples:

(1) Longitudinal segregation was below the sensitivity limit of the density measurements.

(2) No radial segregation appeared on the metallographies of the transverse sections.

(3) The primary interdendritic spacing (lambda) was very close to the predicted value (1400 microns). This indicated that the lower values of (lambda) found in the 1-g processed samples were due to convection.

(4) The dendritic morphology was significantly different from the ground samples: "...each dendrite is very well separated from the neighbouring ones, and has well developed secondary and higher order arms as if free dendritic growth conditions were prevailing." (1, p. 131)

(Reference (1) and Reference (5) provided theoretical discussions in comparison to the above results.)

<Note: References (9)-(11) were not available to aid in the preparation of this experiment summary.>

**Key Words:** Metals and Alloys, Eutectics, Hypereutectics, Hypoeutectics, Binary Systems, Melt and Solidification, Directional Solidification, Solid/Liquid Interface, Dendritic Solidification, Dendritic Arm Spacing, Segregation, Macrosegregation, Microsegregation, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Buoyancy-Driven Convection, Thermosolutal Convection, Density Difference, Growth Rate, Solidification Rate, Thermal Gradient, Solutal Gradients, Sample Microstructure, Grain Structure

**Number of Samples:** three
**Sample Materials:** aluminum-copper alloys (1 wt.% Cu, 26 wt.% Cu, and 40 wt.% Cu)
(Al*Cu*)
**Container Materials:** zirconia, $ZrO_2$
(Zr*O*)

**Experiment/Material Applications:**
The specific reasons why the Al-Cu samples were selected for this experiment were not detailed in available publications related to this mission. However, choice of the Al-Cu system is discussed in general under Favier, Spacelab 1.

**References/Applicable Publications:**
(1) Camel, D., Dupouy, D., Favier, J. J., and Le Maguet, R.: Preliminary Results of the D1-WL-GHF-04 Experiment on Dendritic Solidification of Al-Cu Alloys. Adv. Space Res. Vol. 6, No. 5, pp. 127-132, 1986. (post-flight results)

(2) Camel, D., Favier, J. J., and Dupouy, M. D.: Modification of the Dendritic Solidification Structure Under Microgravity Conditions. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1 Symposium, Norderney, Germany, August 27-29, 1986, p. 68.

(3) Camel, D. and Favier, J. J.: Dendritic Solidification of Al-Cu Alloys. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 103-104. (preflight)

(4) Cambon, G., Castets, B., Boutemy, O., Giequel, J. P., Aubron, R., Boddaert, M., and Benoit, J.: Gradient Heating Facility Behavior. In ESA 5th European Symposium on Materials Science Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 115-119. (FPM as configured after Spacelab 1)

(5) Camel, D., Favier, J. J., Dupouy, M. D., and Le Maguet, R.: Microgravity and Low-Rate Dendritic Solidification. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 236-246. (post-flight)

(6) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. Proceedings of the Norderney Symposium on the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (post-flight; acceleration measurements)

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(8) Input received from Denis Camel (CENG, Grenoble), August 1993.

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(10) Dupouy, M. D., Camel, D., and Favier, J. J., Acta Metall. Mater. 40, 1992, pp. 1791-1801.

(11) Dupouy, M. D., Camel, D., and Favier, J. J., J. Crystal Growth 126, 1993, pp. 480-492.

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**Experiment Origin:** The Netherlands
**Mission:** TEXUS 6
**Launch Date/Expt. Date:** May 1982
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Isothermal four-chamber furnace module (TEM 01-2)
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Sulfur Diffusion in Liquid Cast Iron

During the unidirectional solidification of grey cast iron on Earth, it is difficult to distinguish between gravity-induced convection and surface-tension-driven convection within the melt.

This TEXUS 6 experiment was the first in a series of investigations designed by Luyendijk et al. to study the low-gravity solidification of cast iron. The specific objectives of the experiment were to (1) determine the diffusion coefficient of sulphur in liquid cast iron, (2) compare the diffusion coefficient of sulfur in liquid cast iron to the diffusion coefficient of phosphorous in liquid cast iron, (3) assess the capability of skin technology (prior to a future Spacelab experiment) and (4) investigate the origin of convective flow during the unidirectional solidification of cast iron on Earth. (See Sprenger, TEXUS 1 for a discussion of skin technology (Chapter 18).)

Prior to the flight experiment, five cylinders of white cast iron (4.2 to 4.3 wt% carbon, no silicon) were joined at the ends by a diffusion welding process. The cylinders, labeled a through e, also contained either sulphur or phosphorous: (a) 0.093 wt.% S, (b) 0.006 wt.% S, (c) 0.093 wt.% S, (d) 0.006 wt.% S, and (e) 0.1 wt.% P. The stacked cylinders were coated with a plasma-sprayed alumina skin and wrapped in alumina wool. The assembly was then placed in a TMZ cartridge which was configured with three thermocouples placed (1) in the top end, (2) in the bottom end, and (3) on the side surface. (The expected temperature difference in the sample had been determined by prior ground-based investigations.)

Just prior to the rocket launch, the sample was heated to approximately 1000 $^{o}C$. Sample melting and resolidification occurred during the low-gravity period after launch. A diffusion period of 180 seconds and a thermal gradient (between the top and bottom of the sample) of 10 $^{o}C/cm$ were reported. Cooling of the samples was achieved by the application of helium gas to the bottom of the TZM cartridge.

Post-flight examination of the sulphur and phosphorous distribution throughout the cylinders indicated that both elements were transported over tens of millimeters. A distance of less than 2 mm would have been expected if diffusion had been the sole mode of transport. Thus, the results indicated that convective flow was present in the melt. <Note: It appears that the desired diffusion coefficients were not obtained because of the convective flows.> Reportedly, results from subsequent low-gravity experiments indicated that the convection was induced by a thermal gradient related to the surface-active behavior of sulphur. <Note: The subsequent low-gravity experiments were not identified.>

The alumina skin coating the sample contained several cracks. These cracks were attributed to the volume expansion of the cast iron. No reaction between the sample material and alumina skin occurred.

**Key Words:** Metals and Alloys, Skin Technology, Thin Films, Plasma Spray Coating, Melt and Solidification, Directional Solidification, Surface Tension, Diffusion, Diffusion Coefficient, Diffusive Mass Transfer, Buoyancy-Driven Convection, Surface Tension-Driven Convection, Marangoni Convection, Thermal Gradient, Solid/Liquid Interface, Coated Surfaces, Volume Expansion, Quench Process

**Number of Samples:** One long cylinder comprised of five subsamples (cylinders)
**Sample Materials:** Five cylinders (a through e) of white cast iron (4.2 to 4.3 wt.% carbon, no silicon) with either sulphur or phosphorous; (a) 0.093 wt.% S, (b) 0.006 wt.% S, (c) 0.093 wt.% S, (d) 0.006 wt.% S, and (e) 0.1 wt.% P.
(S*, P*, C*, Fe*)
**Container Materials:** Sample plasma-sprayed with alumina, $Al_2O_3$, skin and contained in a TMZ cartridge
(Al*O*)

**Experiment/Material Applications:**
See Luyendijk, Spacelab 1 (this chapter) or Sprenger, Spacelab D1 (Chapter 18).

Sulfur in cast iron was chosen because sulfur is a surface active element in iron alloys.

Such low-gravity experiments should illustrate the origin of convective flow observed during unidirectional solidification of cast iron on Earth.

**References/Applicable Publications:**
(1) Sulfur Diffusion in Liquid Cast Iron. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 136-137. (post-flight)

(2) Input received from Principal Investigator T. Luyendijk, August 1993.

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**Experiment Origin:** The Netherlands
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Spacelab Rack
**Processing Facility:** Isothermal Heating Facility/with Gradient Device (IHF/G)
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Unidirectional Solidification of Cast Iron (1ES325)

Grey and ductile cast iron materials are widely used because they possess good mechanical and technological properties. These properties are determined by the microstructure (e.g., coarseness and distribution of graphite flakes) present after casting. The microstructure is strongly influenced by the sulphur distribution ahead of the solidification interface, which, in turn, is affected by convective currents. Thus it was suspected that a reduction in gravity-dependent convection would strongly affect the microstructure of the sample.

This Spacelab 1 experiment was the second in a series of investigations designed by Luyendijk et al. to study the low-gravity, directional solidification of cast iron (see Luyendijk, TEXUS 6). The specific objectives of the study were to investigate (1) the transport of sulphur during the solidification of eutectic cast iron and (2) the growth of graphite under low-gravity conditions.

Prior to launch, a single, grey cast iron sample was prepared. The 150 mm long, 6.5 mm diameter sample had a composition of 3.9 wt.% C-1.5 wt.% Si-0.025 wt.% S-0.01 wt.% P and consisted of graphite flakes embedded in a matrix of pearlite or ferrite. The specimen was covered by a plasma-sprayed alumina skin to provide a tight cover around the sample. (See Sprenger, TEXUS 1 for a discussion of skin technology (Chapter 18).) The coated sample was placed within a 0.5 mm thick alumina crucible. Four holes drilled into the alumina lid allowed evacuation of the space between the specimen and crucible. Four thermocouples were located at various distances down the length of the sample.

The Spacelab 1 Isothermal Heating Facility "with Gradient Device" (IHF/G) was configured to directionally solidify the sample via (1) furnace translation and (2) a chill block (gradient device). During the mission, the sample was to be (1) heated for a period of time (before translation) to achieve thermal equilibrium and then (2) directionally solidified at four different growth rates: (1) 0.1 mm/min, (2) 0.4 mm/min, (3) 0.7 mm/min, and (4) 1.0 mm/min.

It was reported that "...at the end of the heating-up period of the furnace a failure in the furnace-power supply occurred and the IHF/G broke-down." (1, p. 80) The failure resulted in a large portion of the sample melting and quickly solidifying. After the furnace was repaired on-orbit, a second experimental run proceeded as planned.

It was noted that the fast cooling rate which occurred during the first run may have caused a transformation of the sample from grey cast iron to white cast iron (iron carbide and pearlite). During such a transformation, volume changes occur and shrinkage cavities can result. If present, these cavities may have affected the solidification process during the second experimental run.

Post-flight examination of the sample included X-ray, X-ray fluorescence, and metallographic techniques. Initial X-ray examination revealed that the liquid sample separated into two parts during processing: (1) an 84 mm section (bottom end), 62 mm of which was directionally solidified, and (2) a 25 mm section which was rapidly cooled. It was reported that the sample separation may have been caused by (1) the constriction and break up caused by the volume change of grey cast iron during melting and (2) the development of some gases during the process. (Such sample separation had also been observed in ground experiments.)

The portion of the sample represented by x=0 to x=22 mm (x measured from sample bottom), did not melt. At the interface between the unmelted portion and the directionally solidified portion, one large cavity (approximately 2.5 mm wide) and several smaller cavities (0.02 to 0.2 mm) were observed. These cavities appeared to (1) move down the thermal gradient and pile-up at the interface and (2) affect the heat flux through the specimen.

Metallographic examination revealed different structures corresponding to the different growth rates. At a growth rate of 0.1 mm/min (x = 22 to 31 mm) the sample initially demonstrated a very fine graphite structure and then changed to a coarse graphite structure at x = 31 to 47 mm. At a growth rate of 0.4 mm/min, the sample demonstrated a fine graphite structure (x = 47 to 56 mm). At a growth rate of 0.7 mm/min, graphite flakes were observed between axially aligned, primary dendrites (x = 56 to 69 mm). The remainder of the directionally solidified portion of the sample had a growth rate of 1.0 mm/min. The structure of this last portion was the same as x = 56 to 69 mm. The remainder of the sample (that which had been separated and rapidly cooled) contained primary dendrites which were mostly oriented perpendicular to the crucible wall.

The sulphur distribution through the flight and similarly processed ground-based samples was determined using X-ray fluorescence techniques (see Reference (1) for a description of the technique). It was noted that the sulphur transport in the flight sample may have been influenced by the first experimental run. The sulphur content of the separated (top) portion of the sample was less than the starting material. This difference indicated an evaporation of sulphur from the melt. In the directionally solidified portion of the flight sample, significant convection appears to have occurred in the liquid, although a small diffusion boundary layer was present. "The surface-active element sulphur, which is less soluble in the solid than in the liquid, seems to be responsible... [for] that convection." (1, p. 85) This convection resulted in a gradually increasing sulphur content just ahead of the interface. The refining tendency of the increase in growth rate was counteracted by the coarsening of the graphite by sulphur. "Except the change from 0.1 to 0.4 mm/min, the other transitions in growth rate are not well reflected in the structure due to a higher sulphur content." (1, p. 85)

<Note: Reference (5) was not used in the preparation of this experiment summary.>

**Key Words:** Metals and Alloys, Eutectics, Skin Technology, Thin Films, Plasma Spray Coating, Coated Surfaces, Casting, Melt and Solidification, Directional Solidification, Solutal Gradients, Thermal Gradient, Heat Flux, Diffusion, Diffusion Coefficient, Diffusive Mass Transfer, Diffusion Boundary, Buoyancy-Driven Convection, Surface Tension-Driven Convection, Marangoni Convection, Buoyancy Effects Diminished, Solid/Liquid Interface, Furnace Translation, Growth Rate, Solidification Front Physics, Sample Microstructure, Evaporation, Coarsening, Dendrites, Dendritic Structure, Sample Shrinkage, Gas Formation, Cavity, Volume Change, Vacuum, Furnace Malfunction

**Number of Samples:** one
**Sample Materials:** Grey cast iron: 3.9 wt.% C-1.5 wt.% Si-0.01 wt.% P-0.025 wt.% S (Initially, the sample consisted of graphite flakes embedded in a matrix of pearlite or ferrite.)
(Fe*C*Si*P*S*)
**Container Materials:** Plasma-sprayed alumina contained in alumina crucible, $Al_2O_3$
(Al*O*)

**Experiment/Material Applications:**
See experiment summary (above).

**References/Applicable Publications:**
(1) Nieswaag, H., Luyendijk, T., and Alsen, W.H.M.: Unidirectional Solidification of Cast Iron ES-325. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 79-85. (post-flight)

(2) Luyendijk, T. and Nieswaag, H.: Unidirectional Solidification of Eutectic Iron-Carbon Alloys and the Segregation of Sulphur. In ESA on Material Science in Space, Grenoble, April 24-27, 1979, ESA SP-142 (June 1979), pp. 115-122. (preflight)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986 Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

(4) Whittmann, K.: The Isothermal Heating Facility. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 49-54. (IHF facility)

(5) Luyendijk, T., Nieswaag, H., and Alsen, W.H.M.: Unidirectional Solidification of Cast Iron. Science, Vol. 225, No. 4658, July 1984, pp. 200-202.

(6) Input received from Principal Investigator T. Luyendijk, August 1993.

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**Experiment Origin:** USA
**Mission:** STS Launch #4, STS-004 (STS OFT-4, Columbia)
**Launch Date/Expt. Date:** June 1982
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment
NASA Get Away Special (GAS) Canister G-001
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-001:** Utah State University, Logan, Utah/R. Gilbert Moore
**Processing Facility:** Furnace Heating Facility
**Builder of Processing Facility:** Unknown, possibly Utah State University, Logan, Utah

**Experiment:**
Eutectic Formation (11-M/13-M)

This experiment was one of ten investigations housed within the G-001 Get Away Special canister during STS-004. Four other experiments (of the ten) were applicable to this data base (see Alford, STS-004 (Chapter 18); Dalley, STS-004 (Chapter 5); Elwell, STS-004 (Chapter 12); and Laher, STS-004 (Chapter 17)). The objective of the experiment was to investigate the eutectic alloying of bismuth and tin in a reduced gravity environment.

Reportedly, prior to the shuttle flight, a mixture of bismuth (57%) and tin (43%) metallic powders was prepared. <Note: It was not clear if the percentage of Bi and Sn was in weight or volume percent.> The mixture was sealed in an aluminum chamber. The chamber contained an argon atmosphere.

During the shuttle mission, the mixture was to be heated to 285 $^{o}$C, allowing the formation of the eutectic alloy.

Reportedly, post-flight analysis of the processed samples would (1) determine the metallic bonding of the powders and (2) examine the interaction of the eutectic alloy with the aluminum chamber. However, while the experiment successfully completed its operation cycle, the heater did not reach the temperature necessary to alloy the powders. The tin powder experienced localized melting, creating small nodules interspersed throughout the sample. It was further reported that the sample chamber seal failed allowing severe oxidation of the remaining bismuth subsequent to the opening of the Get Away Special canister. There was no documented post-flight material analysis.

**Key Words:** Metals and Alloys, Melt and Solidification, Eutectics, Binary Systems, Powder Metallurgy, Metallic Bonding, Thermal Gradient, Solid/Liquid Interface, Oxidation, Material Interaction with Containment Facility, Crucible Effects, Processing Difficulties, Hardware Malfunction, Incomplete Sample Processing

**Number of Samples:** one
**Sample Materials:** bismuth-tin alloy
(Bi*Sn*)
**Container Materials:** Aluminum 6061 (no surface preparation or coatings)
(Al*)

**Experiment/Material Applications:**
This experiment was designed to evaluate low-temperature eutectic alloy formation in the low-gravity environment. Comparisons between terrestrial and space-processed alloy systems would distinguish fundamental characteristics for alloy formation in a space environment. These characteristics could be applied to future terrestrial or space materials processing endeavors.

The bismuth-tin alloy was selected because (1) the experiment was subject to finite energy limitations, and (2) the alloy was expected to exhibit enhanced mixing at the imposed temperature difference (melt temperature, 271 $^{o}C$; solidus point, 139 $^{o}C$).

**References/Applicable Publications:**
(1) Yoel, D., Walker, S., Elwell, J., and Moore, G.: The First Getaway Special-How It was Done. Spaceworld, May 1983, pp. 9-16.

(2) STS-4 Fourth Space Shuttle Mission, NASA Press Kit, June 1982, p. 62. (preflight)

(3) Yoel, D. W.: Payload Integration of a Get Away Special Canister. American Institute of Aeronautics and Astronautics, Annual Meeting and Technical Display on Frontiers of Achievement, Long Beach, California, May 12-14, 1981, 5 pp. (preflight)

(4) The STS-4 Getaway Special. NASA Report PB82-10223, May 20, 1982. (no author listed; preflight)

(5) Cargo Systems Manual: GAS STS-4, May 20, 1982, JSC-17645, pp. 4-1 - 4-4. (preflight; very short description)

(6) Overbye, D.: The Getaway Kids Shuttle Into History. Discover, September 1982.

(7) Yoel, D. W.: Analysis of the First Getaway Special Space Shuttle Payload. Thesis for M.S. in Physics, Utah State University, Logan, Utah, 1984. (post-flight)

(8) Moore, R. G.: Educational Implications of Getaway Special Payload Number One. IAF-81-293, 32nd International Astronautical Federation Congress, Rome, September 6, 1981. (preflight)

(9) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(10) Input received from Experiment Investigator, August 1989.

(11) Transcripts of Press Conference at NASA MSFC with G-001 Student Experimenters and Sponsors, NASA, May 20, 1982.

(12) "Get Away Special," NASA News, NASA/MSFC, June 7, 1982.

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**Experiment Origin:** USA
**Mission:** STS Launch #6, STS-006 (STS 31-B, Challenger)
**Launch Date/Expt. Date:** April 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment
NASA Get Away Special (GAS) Canister G-049
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-049:** The United States Air Force Academy, Colorado Springs, Colorado
**Processing Facility:** Sample in sealed tube heated by nichrome wire
**Builder of Processing Facility:** Designed by J. M. Amidon (Principal Investigator), built by a civil servant technician at the Air Force Academy, Colorado Springs, Colorado

**Experiment:**
<u>Metal Alloy Solidification (Sn-Pb)</u>

This solidification experiment was one of six investigations housed within the G-049 Getaway Special Canister on STS-006. Four other experiments (of the six) were applicable to this data base (see Gross, STS-006 (Chapter 18); Neel, STS-006 (Chapter 4); Peter, STS-006 (Chapter 18); Streb, STS-006 (Chapter 14)). The specific objective of this experiment was to investigate if low-gravity solidification of the Sn-Pb system improved the homogeneity of the sample composition. (During ground-based testing of the alloy, macrosegregation of the components was observed as tin floated to the top of the liquid mixture.)

During the space experiment, a Sn-Pb sample, sealed within a glass tube, was "...heated with nichrome wire until liquid was formed (approximately 8 minutes). The mixture was then allowed to cool and produce a uniform distribution of tin in weightlessness." (2, p. 318)

After post-flight analysis of the sample it was reported that "The surprise was not that the mixture changed phases, created more uniform grain structure or did not show gravity-induced orientations, but that spheres 1/4 in. (0.63 cm) in diameter were formed. This unexpected result showed that surface tension tends to dominate in weightlessness." (2, p. 318) <Note: The composition of the spheres was not detailed.>

No further information concerning the experiment could be located at this time.

<Note: Reference (1) (published prior to the launch of STS-006) states that the alloy to be studied was a tin-gallium system. References (2) and (5) state that tin-lead was studied. Since the post-flight publication (Reference (2)) discusses a tin-lead system, it is thought that the sample was originally to be tin-gallium but was changed to tin-lead for the actual space mission.>

**Key Words:** Metals and Alloys, Melt and Solidification, Binary Systems, Sample Homogeneity, Composition Distribution, Macrosegregation, Solid/Liquid Interface, Sample Microstructure, Grain Structure, Spheres, Surface Tension

**Number of Samples:** one
**Sample Materials:** lead-tin
(Pb*Sn*)
**Container Materials:** glass tube

**Experiment/Material Applications:**
It was anticipated that a more homogeneous distribution of sample composition would be achieved in the low-gravity environment.

**References/Applicable Publications:**
(1) Cargo Systems Manual: GAS Annex for STS-6, JSC-17645 Annex STS-6, December 3, 1982. (very short description; preflight)

(2) Swan, P. and Worsowicz, C.: The Eaglets Have Flown. Space Education, Vol. 1(7) May 1984, pp. 317-319. (post-flight)

(3) STS-6 Getaway Specials, NASA News, NASA GSFC, November 24, 1982. (preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special Canister mission history)

(5) NASA STS-6 Sixth Space Shuttle Mission Press Kit, April 1983, pp. 41-43. (preflight)

(6) Input received from Principal Investigator J. M. Amidon, August 1993.

**Contact(s):**

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**Principal Investigator(s):** Streb, J. M. (1)
**Co-Investigator(s):** Hatelid, Major J. E. (2)
**Affiliation(s):** (1) During STS-006: United States Air Force Academy, Colorado Springs, Colorado, Currently: Aerospace Systems Division, Wright Patterson Air Force Base; (2) During STS-006: Assistant Professor of Astronautics, U.S. Air Force Academy, Colorado Springs, Colorado, Currently: Unknown

**Experiment Origin:** USA
**Mission:** STS Launch #6, STS-006 (STS 31-B, Challenger)
**Launch Date/Expt. Date:** April 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment
NASA Get Away Special (GAS) Canister G-049
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-049:** The United States Air Force Academy, Colorado Springs, Colorado
**Processing Facility:** Nichrome heating coil which translated along the length of a sample.
**Builder of Processing Facility:** Unknown

**Experiment:**
Zone Refining Purification

This experiment was one of six experiments housed within the G-049 Getaway Special Canister on STS-006. Four other experiments (of the six) were applicable to this data base (see Amidon, STS-006 (Chapter 14); Gross, STS-006 (Chapter 18); Neel, STS-006 (Chapter 4); Peter, STS-006 (Chapter 18)). The specific objective of the experiment was to determine if a tin sample contaminated with lead could be purified in a low-gravity environment using a zone-refining technique.

During the experiment, the Sn-Pb sample (which appears to have been housed in a quartz tube) was melted by a nichrome heating coil. The coil translated along the 7.5 cm sample length in 80 minutes. During this slow translation, local melting occurred at the location of the heater. As the heater continued to translate past this local melting position, local solidification occurred. It was expected that (1) the lead impurities would be rejected in front of the solidification front throughout the translation and (2) the Pb would "float" to the far end of the sample thus purifying the Sn.

It was reported that the equipment operated (mechanically) during the mission. However, at the time Reference (1) was written, analysis of the sample had not been completed.

No other information concerning this experiment could be located.

**Key Words:** Metals and Alloys, Binary Systems, Melt and Solidification, Zone-Refining, Metal Purification, Sedimentation, Buoyancy-Driven Convection, Impurities, Solid/Liquid Interface, Solidification Front Physics, Inclusion and/or Rejection of Particles, Translation Rate

**Number of Samples:** one
**Sample Materials:** tin-lead
(Sn*Pb*)
**Container Materials:** possibly quartz
(Si*O*)

**Experiment/Material Applications:**
On Earth, gravity-driven forces hamper the purification of metals by the zone-refining process. If this low-gravity refining experiment proved successful, the research could lead to high purity metal production.

**References/Applicable Publications:**

(1) Cargo Systems Manual: GAS Annex for STS-6. JSC-17645 Annex STS-6, December 3, 1982. (very short description; preflight)

(2) Swan, P. and Worsowicz, C.: The Eaglets Have Flown. Space Education, Vol. 1, Number 7, May 1984, pp. 317-319. (postflight)

(3) STS-6 Getaway Specials. NASA News, NASA/GSFC, November 24, 1982. (preflight)

(4) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report # EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(5) NASA STS-6 Sixth Space Shuttle Mission Press Kit, April 1983, pp. 41-43. (preflight)


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**Principal Investigator(s):** Carlberg, T. (1)
**Co-Investigator(s):** None
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**Experiment Origin:** Sweden
**Mission:** TEXUS 7
**Launch Date/Expt. Date:** May 1983
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Mirror Furnace
**Builder of Processing Facility:** Swedish Space Corporation (SSC), Solna, Sweden

**Experiment:**
Examination of Floating Zones in a Mirror Furnace

This TEXUS 7 experiment was the first in a series of investigations designed by Carlberg et al. to study low-gravity float zones. <Note: The remainder of the experiments in this series can be found in Chapter 9, Crystal Growth from the Melt.> The specific objectives of the experiment were to (1) examine the formation of a Al-Cu liquid zone, and (2) study the possible effects of Marangoni convection on zone heat transfer.

Information describing the employed mirror furnace or the TEXUS 7 experimental setup could not be located at this time.

Reportedly, no results are available due to a furnace failure.

**Key Words:** Metals and Alloys, Binary Systems, Eutectics, Melt and Solidification, Float Zones, Surface Tension, Free Surface, Thermocapillary Convection, Marangoni Convection, Surface Tension-Driven Convection, Heat Transfer, Solid/Liquid Interface, Liquid/Gas Interface, Sample Homogeneity, Semiconductor Applications, Furnace Malfunction

**Number of Samples:** unknown
**Sample Materials:** eutectic Al-Cu alloy
(Al*Cu*)
**Container Materials:** unknown

**Experiment/Material Applications:**
Float-zone space processing may lead to an improvement in the homogeneity, purity and defect distribution of semiconductor crystalline materials.

**References/Applicable Publications:**
(1) Input received from Experiment Investigator, May 1988.

**Contact(s):**
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**Co-Investigator(s):** MBB-ERNO (2)
**Affiliation(s):** (1) Deutsche Forschungs-und Versuchsanstalt für Luft-und Raumfahrt (DFVLR), Institute for Space Simulation, Cologne, Germany <Note: The DFVLR is now called the Deutsche Forschungsanstalt für Luft-und Raumfahrt (DLR)>; (2) Bremen, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #7, STS-007 (STS 31-C, Challenger)
**Launch Date/Expt. Date:** June 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** West German Get Away Special (GAS) MAUS Canister DG-315
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay (carried on the Office of Space and Terrestrial Applications (OSTA-2) pallet (a NASA experiment carrier))
**Primary Developer/Sponsor of DG-315:** DFVLR, Cologne, Germany/Messerschmitt-Boelkow-Blohm (MBB-ERNO), Bremen, Germany
**Processing Facility:** Cartridge Heater Furnace
**Builder of Processing Facility:** Unknown

**Experiment:**
Particle Transport Induced by Directional Solidification

Ground-based production of dispersion strengthened materials is strongly influenced by gravity-induced sedimentation and convection. While production under low-gravity conditions significantly reduces these disturbing influences, gravity-independent mechanisms such as boundary forces, percolation, and particle transport can still produce very complex phenomena in the low-g solidified materials.

This STS-007 experiment was designed to study the low-gravity production of solid metallic dispersions from the melt. The specific objective of the investigation was to examine the movement of second phase inclusions during the directional solidification of a model system.

During the STS-007 mission, a transparent model system of CsCl containing air bubbles and Pb drops was melted and directionally solidified using a MAUS Get Away Special (GAS) canister. The air bubbles were included to (1) account for thermal expansion and volume increase during sample melting and (2) act as second phase particles.

The pure nickel furnace used for the investigation contained a test volume of 6 mm x 12 mm x 6 mm. Two sapphire windows allowed filming of the melting and solidification processes (Hasselblad 500 EL/M motor camera, one photograph every 12 seconds). After activation of the experiment by a payload specialist, the experiment ran automatically. (See Reference (3) and/or Reference (8) for complete details of experimental hardware.)

Post-flight examination of the film indicated the solidification front remained planar for the first 5 minutes of low-gravity solidification. Reportedly, the growth rate during the first five minutes of the solidification was 4 microns/sec. However, the planar interface became disturbed by the presence of the air bubbles. "In the early stages of the solidification process formation of gas bubbles at the solidification front was favoured by the discontinuous change of solubility of gas in melt and gas in crystal. Later gas bubbles were formed at the furnace walls also. Gas bubbles at the advancing solidification front were not pushed ahead into the melt but were built into the CsCl crystal as canal bubbles from the statu nascendi...." (3, p. 376) <Note: a definition of the words "statu nascendi" could not be located at this time.> It was also reported that "...bubble growth with advancing solidification proceeded faster than the engulfment of the bubbles by the crystal." (3, p. 376)

By comparing sequential flight photographs taken during the melting and solidification processes, a global pattern of particle motion was revealed. Reportedly, this clock-wise motion was due to gravity-induced convection. The measured residual gravity normal to the thermal gradient (65 K/cm) was between $10^{-5}$ and $10^{-6}$ g. The range of free convection velocities, estimated by the Navier-Stokes equations, was within the range of those velocities observed in the flight experiment.

Displacement of 40 micron Pb drops by the solidification front (moving at 4 microns/sec) was clearly visible. This result agreed favorably with critical velocity determinations from terrestrial experiments employing an organic matrix. (See Reference 8 for details of terrestrial experiments.)

**Key Words:** Metals and Alloys, Model Materials, Transparent Liquids, Melt and Solidification, Directional Solidification, Thermal Gradient, Dispersion, Metallic Dispersion, Dispersion Strengthening, Multiphase Media, Bubbles, Bubble Formation, Bubble Dispersion, Bubble Growth, Drops, Particle Transport, Particle Motion, Particle Dispersion, Bubble Motion, Inclusion and/or Rejection of Particles, Critical Velocity, Marangoni Convection, Thermocapillary Convection, Surface Tension-Driven Convection, Buoyancy-Driven Convection, Sedimentation, Interface Physics, Interfacial Tension, Interface Shapes, Solidification Front Physics, Planar Solidification Interface, Growth Rate, Solid/Liquid Interface, Liquid/Gas Interface, Thermal Expansion, Volume Increase, Acceleration Effects

**Number of Samples:** one
**Sample Materials:** molten cesium-chlorine with lead droplets and gas bubbles
(Cs*Cl*, Pb*)
**Container Materials:** unknown

**Experiment/Material Applications:**
Cesium chloride, CsCl, was chosen as the matrix material because (1) it is transparent and the solidification process can be filmed and (2) its relevant solidification parameters are similar to metallics (e.g., entropy of solidification, melt temperature).

**References/Applicable Publications:**

(1) Klein, H. and Nähle, R.: Particle Transport Induced By Directional Solidification. In ESA 5th European Symposium on Material Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 37-40. (preflight)

(2) Klein, H., Nähle, R., and Wanders, K.: Metal-Like Solidification Of a Multi-Phase Dispersion in Low Gravity During a Space Shuttle Flight. Zeit-schrift fuer Flugwissenschaften und Weltraumforschung, Vol. 9, Jan.-Feb. 1985, pp. 14-20. (post-flight)

(3) Klein, H., Nähle, R., and Wanders, K.: Transport Processes in Directional Solidification During Space Shuttle Flight. In Proceedings of the 5th European Symposium on Materials Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 375-378. (post-flight)

(4) Baum, D., Otto, G., and Vits, P.: MAUS-A Flight Opportunity for Automated Experiments Under Microgravity Conditions. Acta Astronautica, Vol. 11, No. 3-4, pp. 239-245, 1984. (no discussion of experiment results)

(5) Baum, D., Stolze, H., and Vits, P.: First Flight Data From MAUS Payloads on STS 7 and STS 11. 35th Congress of the International Astronautical Federation, October 7-13, 1984, Lausanne, Switzerland, IAF #84-137, 11 pp. (post-flight)

(6) Klein, H., Walter, H. U., Bewersdorff, A., and Pötschke, J.: Direct Observation of Processes in a Solidifying Dispersion. Adv. Space Research, Vol. 4, No. 5, pp. 57-62, 1984. (post-flight)

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(8) Omenyi, S. N., Neumann, A. W., Martin, W. W., Lespinard, G. M., and Smith, R. P., Journal of Applied Physics, Vol. 52, 1981, p. 796. (ground-based experiments used for comparison)

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**Co-Investigator(s):** Unknown
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**Experiment Origin:** Italy
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Material Sciences Double Rack (MSDR)
**Processing Facility:** Ultra-High Vacuum (UHV) Chamber
**Builder of Processing Facility:** Centro Informazioni Studi Esperienze (CISE), Milano, Italy

**Experiment:**
Adhesion of Metals (1ES340)

Although adhesion forces are fundamental to all contact problems involving macroscopic bodies, very little is known about their nature and magnitude. Experimental studies of adhesion phenomena must take into account the contacting surfaces' (1) cleanliness or controlled contamination, (2) surface topography, and (3) deformation properties. Experimental results (such as those related to interface energy determinations) strongly depend on the method of measurement and reportedly have not been satisfactory. "In particular, static experiments cannot give information about the onset of dissipative phenomena which constitutes a controlling parameter of adhesion between solid bodies." (5, p. 347)

In a reduced gravity environment, it should be possible to perform dynamic adhesion experiments such that (1) energy transfers are of the same order of magnitude as interface energies and (2) a minimum disturbance of the surface (e.g., mechanical, chemical) is realized.

This Spacelab 1 experiment was designed to investigate the surface forces and interface energies present during the contact of two metallic surfaces (a sphere and target). A major objective of the investigation included the measurement of the restitution coefficient, the contact time, and the time evolution of the contact force as a function of impact velocity.

During an experimental run, the small (3.17 mm diameter) metallic sphere was released from a holding area toward a force transducer (metal target). Reportedly, "...the sphere rebounds on the target, progressively reducing its energy so as to reach the velocity values required for the investigation of adhesion forces." (1, p. 207) (A percussion subsystem contained within the experimental facility, "...hit[s] the base of the transducer when the sphere was resting on it, making the sphere start a series of rebounds." (1, p. 208) Incoming and outcoming velocities of the sphere at each impact were to be deduced from from the time interval measured between successive rebounds. Data from the transducer enabled determination of the contact force versus time during each sphere impact. ("The transducer output signals were time sampled and recorded, and from these data the time evolution of the contact force was obtained." (1, p. 207))

Both the sphere and target were contained in an ultra high vacuum (UHV) chamber to control possible contamination of the colliding surfaces. The chamber was connected to an ion pump that maintained the the facility at a dynamic vacuum level of $10^{-10}$ Torr. "In order to neglect the actual microgravity aboard SPACELAB and parasitic accelerations, a known... [gravity]... field, of the order of $10^{-3}$g,... [was] produced by means of a magnetic field acting on the sphere...." (1, p. 207)

The 1-hour-long experiment was originally scheduled for the 4th day of the mission. During this hour, data related to approximately 80 impacts were to be collected. "However, since the facility operation required minimum involvement from the payload specialists and minimum power consumption, it was possible to start the experiment several additional times during the Mission." (1, p. 209) (The facility was actually active approximately 4 hours during the mission.)

Reportedly, "In most cases, no effective operation of the facility was obtained: the experiment run was started several times, each time automatically ending to abort after a short period, since no data were available in the UHV chamber buffer memories when the experiment sequence reached the step 'data transfer to CCO'. In other cases, significant data were actually collected, so the experimental runs were performed as expected." (1, p. 209) (Reference (1) includes details of the above mentioned difficulty.)

Analysis of the data indicated the time intervals between successive rebounds (which were to be resolved from the time-of-flight data) could not be resolved. Therefore, single pulse analysis was used to obtain interface energy data (see Reference (1) for details). Reportedly, the surface energy per unit area for the experimental materials could be attained and was 1.15 (+/- 0.02) $J/m^2$. These data fit within the expected range for iron-based materials.

**Key Words:** Metals and Alloys, Adhesion of Metals, Collision of Metals, Impact Velocity, Solid/Solid Interface, Spheres, Interfacial Energy, Surface Energy, Magnetic Fields, Acceleration Effects, Vacuum, Processing Difficulties

**Number of Samples:** unclear
**Sample Materials:** Sphere material: AISI 420 Steel; target material: AISI 325 Steel
**Container Materials:** not applicable

**Experiment/Material Applications:**
See **Experiment** section (above).

**References/Applicable Publications:**
(1) Rossitto, F. and Ghersini, G.: Adhesion of Metals-Experiment 1 ES 340. In ESA 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 207-211.

(2) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

(3) Adorni, N., Ghersini, G., Sona, P. G., Rossitto, F., and Cortellazzi, G.: <Note: Title of article not specified.> In Proc. of the 3rd European Symposium on Materials Sciences in Space, Grenoble, ESA SP-142 (June 1979), pp. 125-132.
(UHV facility)

(4) Adorni, N., Ceserani, A., Cortellazzi, G., Ghersini, C., Grugni, G., Oriani, A., Rossini, T., Rossitto, G., Sona, P. G., Squilloni, A., Torsello, G., and Vaghi, F.: <Note: Title of article not specified.> In Proc. 21st Int. Sci. Meeting on Space, Rome, 1981, pp. 153-162. (UHV facility)

(5) Rossitto, F., Sona, P. G., Grugni, G., and Ghersini, G.: "Adhesion of Metals" Experiment Simulation and Ground-Based Reference Tests Results. In Proceedings of the Fourth European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, pp. 347-351. (preflight, ground-based tests)

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**Co-Investigator(s):** Unknown
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**Experiment Origin:** France
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Spacelab Rack
**Processing Facility:** Isothermal Heating Facility (IHF) Furnace
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Nucleation Behavior of Eutectic Alloys 1ES312

"The eutectics obtained with a metal and a semiconductor show extensive clustering in the liquid just above the melting temperature. The existence of these clusters seems to have a strong influence on nucleation.

"On earth destabilizing currents are induced by thermal convection and sedimentation. In space liquid stability should be greater than on earth and consequently undercooling should be more important." (3, p. 106)

This Spacelab 1 experiment was the first in a series of investigations designed by Malmejac to study the low-gravity nucleation of a crystal in a liquid. (The experiment was also the one of two Spacelab 1 investigations designed by Malmejac to examine the behavior of metallic melts (see Malmejac, Spacelab 1, Thermodiffusion in Tin Alloys (Chapter 11)).)

A document published prior to the Spacelab 1 mission (Reference (2)), indicated that four alloys (Ag-Ge, Al-Si, Al-Ge, and Au-Si) were to be processed in the Isothermal Heating Facility (IHF) during the Spacelab mission.

No technical papers (published after the flight) could be located which discussed the objectives, experimental setup, or results of this investigation.

**Key Words:** Metals and Alloys, Melt and Solidification, Binary Systems, Eutectics, Buoyancy-Driven Convection, Thermal Convection, Sedimentation, Liquid Stability, Solid/Liquid Interface, Undercooling, Nucleation

**Number of Samples:** unknown, possibly four
**Sample Materials:** Unknown. Alloys of Ag-Ge, Al-Si, Al-Ge, and Au-Si were to be processed.
(Ag*Ge*, Al*Si*, Al*Ge*, Au*Si*)
**Container Materials:** unknown

**Experiment/Material Applications:**
A publication published prior to the Spacelab 1 launch (Reference (2)) indicated that the alloys to be studied were selected because of their unique behavior (1) while liquid and (2) during solidification.

**References/Applicable Publications:**
(1) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

(2) Spacelab 1 Experiments. ESA, NASA, Marshall Space Flight Center, Publication Number 15M983, p. 17. (preflight; press release)

(3) Malmejac, Y.: Nucleation of Eutectic Alloys. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, p. 106. (preflight)

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**Experiment Origin:** France
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Materials Science Double Rack (MSDR)
**Processing Facility:** Isothermal Heating Facility (IHF) Furnace
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Nucleation of Eutectic Alloys-Analysis of Crystal Liquid Phase Nucleation in Microgravity (WL-IHF-09)

This Spacelab D1 experiment was the second in a series of investigations designed by Malmejac to study the low-gravity nucleation of a crystal in a liquid (see Malmejac, Spacelab 1, Nucleation Behavior of Eutectic Alloys).

Reference (1) indicated that four alloys (Ag-Ge, Al-Si, Al-Ge, and Au-Si) were to be processed in the Isothermal Heating Facility (IHF) during the mission. The facility was to consist of "...4 graphite blocks each of them containing 4 crucibles filled with the 4 different alloys and one thermocouple." (1, p. 106)

No papers (published after the flight) could be located which discussed the objectives, experimental setup, or results of the investigation.

**Key Words:** Metals and Alloys, Melt and Solidification, Binary Systems, Eutectics, Buoyancy-Driven Convection, Sedimentation, Liquid Stability, Solid/Liquid Interface, Undercooling, Nucleation

**Number of Samples:** unknown, possibly sixteen

**Sample Materials:** Unknown. Alloys of Ag-Ge, Al-Si, Al-Ge, and Au-Si were to be processed.
(Ag*Ge*, Al*Si*, Al*Ge*, Au*Si*)
**Container Materials:** unknown

**Experiment/Material Applications:**
A preflight publication (Reference 1) indicated that the alloys to be studied were selected because of their unique behavior (1) while liquid and (2) during solidification.

**References/Applicable Publications:**
(1) Malmejac, Y.: Nucleation of Eutectic Alloys. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, p. 106. (preflight)

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**Co-Investigator(s):** None
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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Materials Sciences Double Rack (MSDR)
**Processing Facility:** Low-Temperature Gradient Heating Facility (GHF) Furnace
**Builder of Processing Facility:** CNES, Grenoble, France

**Experiment:**
Unidirectional Solidification of Eutectics (InSb-NiSb) (1ES319)

Interest in the technical applications of composite materials has spurred the development of "in-situ" composites. These composite materials are formed by the solidification of a homogeneous melt composed of a eutectic composition. While well-defined composite structures can be created via directional solidification of the material, gravity-driven convection can detrimentally alter the resulting eutectic structure.

This Spacelab 1 experiment was the first in a series of investigations designed by G. Müller et al. to study the low-gravity directional solidification of an InSb-NiSb eutectic. The specific objective of the investigation was to examine the influences of gravity on the solidification of a model eutectic system.

Prior to the shuttle flight, three samples of an InSb-NiSb alloy (eutectic composition) were prepared. Each sample was configured in a sealed quartz ampoule filled with an inert gas. Two graphite plugs at each end of the sample maintained the heat flux, and a quartz spring enclosed in each ampoule compensated for volume increase (13.6% for InSb-NiSb) upon melting. Two thermocouples (enclosed in quartz tubes) were included in each sample.

During the low-gravity mission, the three samples were simultaneously processed in the Gradient Heating Facility (GHF). The samples were heated and melted (approximately 1 hour) and then directionally solidified using a "power down" technique (approximately 2 hours). This technique produced three directionally solidified samples of equal lengths (32.7 +/- 0.8 mm).

Post-flight examination of the samples was performed using metallographic techniques. Each sample was cut longitudinally and mechanically polished. Samples cut perpendicular to the growth direction were also examined. After counting the number of eutectic needles per unit area, it was determined that the space-grown samples contained approximately 30% more needles than corresponding Earth-processed samples. As in the Earth-processed sample, the number of needles was proportional to the growth rate, but with a 30% higher proportionality constant. It was also reported that no dendrites were observed in the samples.

<Note: Reference (1) includes a discussion of a theory, according to Jackson and Hunt, which relates growth rate and eutectic spacing.>

**Key Words:** Metals and Alloys, Melt and Solidification, Directional Solidification, Eutectics, Binary Systems, Model Materials, Buoyancy-Driven Convection, Thermal Gradient, Sample Homogeneity, Heat Flux, Solid/Liquid Interface, Growth Rate, Sample Microstructure, Composition Distribution, Needles, Volume Change, Volume Compensation, Semiconductors, Semiconductor Applications

**Number of Samples:** three
**Sample Materials:** InSb-NiSb; Indium-Antimonide - Nickel-Antimonide
(In*Sb*Ni*Sb*)
**Container Materials:** quartz covered with graphite foil
(Si*O*, C*)

**Experiment/Material Applications:**
The semiconductor material InSb-NiSb was selected for study as a model system because:

(1) The eutectic structure is highly sensitive to solidification parameters
(2) The eutectic has been extensively investigated and has been well characterized
(3) The eutectic melting point is very low (520 $^{o}C$).

Further, the material has been employed as a galvanomagnetic (field-plate) device.

**References/Applicable Publications:**

(1) Müller, G. and Kyr, P.: Directional Solidification of InSb-NiSb Eutectic. In ESA 5th European Symposium on Materials Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 141-146. (post-flight)

(2) Chassay, R. P. and Schwaniger, A: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements; post-flight)

(3) Cambon, G., Castets, B., Boutemy, O., Gicquel, J. P., Aubron, R., Boddaert, M., and Benoit, J.: Gradient Heating Facility Behavior. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 115-119. (post-flight; discusses experimental apparatus)

(4) Input received from Principal Investigator G. Müller, August 1993.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 10
**Launch Date/Expt. Date:** May 1984
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 02-3
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Directional Solidification of InSb-NiSb Eutectic

This TEXUS 10 experiment was the second in a series of investigations designed by G. Müller et al. to study the low-gravity directional solidification of an InSb-NiSb eutectic (see G. Müller, Spacelab 1). The specific objectives of the experiment were to (1) determine the influence of convection on the growth and structure of fiber-like eutectic alloys, (2) test a mirror furnace to be later employed on the Spacelab D1 mission, and (3) test a current pulse technique used for solidification rate determination.

Prior to the flight, one InSb-2.3 wt.% NiSb eutectic sample was prepared. The sample was contained in a sealed quartz ampoule filled with an inert gas. Two graphite plugs at each end of the sample maintained the heat flux and a quartz spring enclosed in the ampoule compensated for volume increase (13.6% for InSb-NiSb) upon melting. The sample contained at least one thermocouple and current wires for interface demarcation by the Peltier pulse technique. (Interface demarcation allowed accurate determination of the growth rate).

The sample was contained in the TEXUS Experiment Module TEM 02-3 mirror furnace. Just prior to launch, approximately 11 mm of the sample was directionally solidified. Solidification was accomplished by translating the sample at growth rates of about 1 mm/min. During the low-gravity phase of the mission, approximately 4 mm of the sample was directionally solidified. Demarcation of the solid/liquid interface was achieved during the whole growth period.

Post-flight examination of the sample revealed that the number-density of eutectic fibers, N, increased with solidification rate, V. It was also reported that for a given solidification rate, N was greater in the low-gravity processed samples when compared to Earth-processed samples.

"The analysis of the microstructures along a longitudinal section of the sample showed that the initial interface was practically plane. 11 mm of sample were resolidified before launch which induced a growth discontinuity. At that time, the interface was then slightly convex.... [The low-gravity] microstructure confirmed the results of the previous space experiment. The average spacing between the NiSb needles decreased by 20% during the flight." (2, p. 222)

No further information concerning this experiment could be located at this time.

**Key Words:** Metals and Alloys, Melt and Solidification, Directional Solidification, Eutectics, Fiber Eutectics, Binary Systems, Model Materials, Buoyancy-Driven Convection, Thermal Gradient, Homogeneity, Heat Flux, Solid/Liquid Interface, Interface Demarcation, Peltier Pulsing, Growth Rate, Solidification Rate, Translation Rate, Interface Shapes, Planar Solidification Interface, Interfacial Curvature, Sample Microstructure, Inter-Fiber Spacing, Composition Distribution, Fibers, Needles, Volume Change, Volume Compensation, Semiconductors, Semiconductor Applications

**Number of Samples:** one
**Sample Materials:** Indium-antimony - nickel-antimony, InSb-2.3 wt.% NiSb
(In*Sb*Ni*Sb*)
**Container Materials:** quartz
(Si*O*)

**Experiment/Material Applications:**
See G. Müller, Spacelab 1.

**References/Applicable Publications:**
(1) Müller, G. and Kyr, P.: Directional Solidification of the InSb-NiSb Eutectic. In Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 246-259. (post-flight; discusses results from TEXUS 10, Spacelab 1, and Spacelab D1 experiments)

(2) Directional Solidification of Eutectic InSb-NiSb. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, February 1991, ESA SP-1132, pp. 222-223. (post-flight)

(3) Input received from Principal Investigator G. Müller, August 1993.

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**Co-Investigator(s):** None
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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, M E D E A Double Rack
**Processing Facility:** Monoellipsoidal Mirror Furnace (ELLI)
**Builder of Processing Facility:** Dornier DS, Friedrichshafen, Germany

**Experiment:**
<u>Directional Solidification of InSb-NiSb Eutectic - Determination of Gravity-Induced Convection on Fiber Spacing (MD-ELI 04/05)</u>

This Spacelab D1 experiment was the third in a series of investigations designed by G. Müller et al. to study the low-gravity directional solidification of an InSb-NiSb eutectic (see G. Müller, Spacelab 1; TEXUS 10). The specific objective of the investigation was to examine the influences of gravity on the solidification of a model eutectic system.

Prior to the D1 mission, two InSb-NiSb eutectic samples were prepared. Each sample was contained in a sealed quartz ampoule filled with an inert gas. Two graphite plugs at each end of the sample maintained the heat flux and a quartz spring enclosed in each ampoule compensated for volume increases (13.6% for InSb-NiSb) upon melting. Each sample contained at least one thermocouple and current wires for interface demarcation by the Peltier pulse technique. (Interface demarcation allowed accurate determination of the growth rate.)

During the mission, the samples were directionally solidified in the Monoellipsoid Mirror Heating Furnace.

To thoroughly examine the effects of gravity on the eutectic spacing, several directional solidification experiments were performed on Earth. These experiments included processing InSb-NiSb eutectics (1) at normal (1-g) gravity levels and (2) at higher centrifuge-induced g-levels. The centrifuge subjected the samples to gravity levels of 50 to 30 g. The results from these ground-based studies were compared to those of the Spacelab D1 mission as well as other low-gravity experiments (see Reference (3)).

Post-flight examination of the D1 samples indicated a larger number of eutectic fibers per unit area, N, than in those samples processed (at the same growth rate) under 1-g or high-g conditions. All samples illustrated a proportionality between N and the solidification rate, V, but with different proportionality constants. It was reported that, by employing the Jackson and Hunt theory (see Reference (3) for detailed discussion), the variation between fiber spacing and growth rate could not be explained assuming an exact eutectic composition. By assuming an off-eutectic composition, the model could quantitatively describe the results from the InSb-NiSb eutectics. This was also true when the theory was applied to results from other eutectic systems (see Reference (3)). It was reported that a possible explanation for the off-eutectic behavior of these experiments was an effect from Soret diffusion.

**Key Words:** Metals and Alloys, Melt and Solidification, Directional Solidification, Eutectics, Fiber Eutectics, Binary Systems, Model Materials, Buoyancy-Driven Convection, Diffusion, Soret Diffusion, Thermal Gradient, Homogeneity, Heat Flux, Solid/Liquid Interface, Solidification Rate, Interface Demarcation, Peltier Pulsing, Growth Rate, Sample Microstructure, Inter-Fiber Spacing, Composition Distribution, Fibers, Volume Change, Volume Compensation, Centrifuge, Acceleration Effects, Processing at High Gravity Levels, Semiconductors, Semiconductor Applications

**Number of Samples:** two
**Sample Materials:** InSb-NiSb, Indium-Antimony - Nickel-Antimony, eutectic composition
(In*Sb*Ni*Sb*)
**Container Materials:** quartz
(Si*O*)

**Experiment/Material Applications:**
See G. Müller, Spacelab 1.

**References/Applicable Publications:**


(1) Müller, G. and Kyr, P.: Directional Solidification of the InSb-NiSb Eutectic. In BMFT/DFVLR Scientific Results of Spacelab Mission D1, Abstracts of the D1 Symposium, Norderney, Germany, August 27-29, 1986, p. 67. (abstract only; post-flight)

(2) Müller, G. and Kyr, P.: Directional Solidification of InSb-NiSb Eutectic. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 105-106. (preflight)

(3) Müller, G. and Kyr, P.: Directional Solidification of the InSb-NiSb Eutectic. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 246-259. (post-flight)

(4) Müller, G., Schmidt, E., and Kyr, P.: Investigation of Convection in Melts and Crystal Growth Under Large Internal Accelerations. In Journal of Crystal Growth, Vol. 49, 1980, pp. 387-395. (Preflight; results from high-g experiments)

(5) Müller, G. and Neumann, G.: Suppression of Doping Striations in Zone Melting of InSb by Enhanced Convection on a Centrifuge. In Journal of Crystal Growth, Vol. 59, 1982, pp. 548-556. (preflight; results from high-g experiments)

(6) Input received from Principal Investigator G. Müller, August 1993.


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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 10
**Launch Date/Expt. Date:** May 1984
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-6 (the facility included a total field interferometer)
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Boundary Layer in Solidifying Transparent Melts

<Note: The following summary was derived from information provided by the Principal Investigator, A. Ecker, and brief discussions provided in References (3) and (10). It was reported (Reference (8)) that the experimental objectives and setup of this experiment were published in Reference (9). However, Reference (9) was not available at the time this experiment summary was prepared.>

This TEXUS 10 experiment was the first in a series of investigations designed by Ecker et al. to study the low-gravity solidification of a transparent material using holographic techniques. The specific objective of the experiment was to study the inter-dependencies of temperature, concentration, and fluid flow in the melt adjacent to the solid-liquid interface.

During the mission, the TEXUS Experiment Module TEM 06-6 was used to melt and solidify a hypomonotectic succinonitrile-ethanol mixture. A total field interferometer included in the module produced interferograms of the sample during the rocket flight. These interferograms were recorded by a cine camera. From these interferograms, the concentration fields of the sample were determined. Thermocouples in the melt provided temperature data of the experiment.

Reportedly, the research demonstrated significant differences in low-gravity and terrestrial temperature and concentration profiles. These differences were attributed to the "...rising droplets of the $L_2$-phase (ethanol rich) in the gravity environment.... Separation processes in the melt below the consulate temperature and the different wetting characteristics at the [solid/liquid] interface between liquid ($L_2$)-solid ($S_1$) liquid ($L_1$) causes fluid flow in the vicinity of the solidification front. Density differences between the liquid ($L_1$) and the liquid ($L_2$) augment or diminish the thermocapillary driven motion of the droplets toward the higher temperature, resulting from thermal and solutal differences in the interface. The droplets are resolved after reaching the critical mixing... [temperature] $T_c$. The resulting concentration distribution is not described by any known theory. In contrast the concentration and temperature profiles measured during the TEXUS 10 flight are compatible to the textbook behavior of heat and mass transport due to pure diffusion. Experiments employing a hypermonotectic composition cannot be used because of the large number of nucleating droplets in the undercooled liquid. The principle behavior was observed using hypomonotectic compositions. There droplets will be created only by the monotectic solidification.... The small droplets rise owing to... [thermocapillary] augmented by... [buoyancy] and decompose at the critical isotherm...." (3, p. 213)

**Key Words:** Metals and Alloys, Melt and Solidification, Monotectic Compositions, Hypermonotectic Compositions, Transparent Liquids, Model Materials, Solid/Liquid Interface, Boundary Layer, Heat and Mass Transfer, Solutal Gradients, Thermal Gradient, Density Difference, Concentration Distribution, Thermosolutal Convection, Buoyancy-Driven Convection, Buoyancy Effects, Diffusion, Undercooling, Drops, Liquid/Liquid Interface, Wetting, Drop Migration, Marangoni Movement of Droplets, Thermocapillary Convection, Nucleation, Interferometric Measurement, Holography

**Number of Samples:** one
**Sample Materials:** succinonitrile-15 wt.% ethanol
**Container Materials:** stainless steel, optical glass windows; side walls: PVC (polyacetal)

**Experiment/Material Applications:**
"The microstructure of cast metallic materials is largely determined by the primary dendrite solidification phenomena which are influenced by the nature of the fluid flow in the melt. Because transparent model systems [e.g., succinonitrile-ethanol, succinonitrile-water] solidify in the same manner as metals, they

are useful for fundamental investigations and allow in situ observation and interferometric measurement of the environment close to the solid-liquid interface." (1, p. 309)

**References/Applicable Publications:**

(1) Ecker, A., Alexander, J.I.D., and Frazier, D. O.: Simultaneous Temperature and Concentration Measurement in Front of Solidifying Monotectic Systems Using the Two Wavelength Holographic Technique. In Proc. of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, February 1987, pp. 309-311. (post-flight)

(2) Ecker, A.: A Two Wavelength Holographic Technique for Simultaneous Measurement of Temperature and Concentration During the Solidification of Two Component Systems. AIAA 25th Aerospace Sciences Meeting, January 12-15, 1987, Reno, Nevada, AIAA #87-0579, 6 pp.

(3) Ecker, A., Schmitz, G. J., and Sahm, P. R.: Boundary Layers of Solidifying Transparent Melts. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 212-216. (post-flight)

(4) Input received from Principal Investigator A. Ecker, July 1988 and July 1993.

(5) Ecker, A.: Two-Wavelength Holographic Measurement of Temperature and Concentration During Alloy Solidification. Journal of Thermophysics and Heat Transfer, Volume 2, Number 3, July 1988, pp. 193-196. (related research)

(6) Ecker, A., Frazier, D. O. and Alexander, J.I.D.: Fluid Flow in Solidifying Monotectic Alloys. Metallurgical Transactions A, Volume 20A, November 1989, pp. 2517-2527. (related research)

(7) Ecker, A., Frazier, D. O., and Alexander, J.I.D.: Classification of Fluid Flow in Front of Solidifying Monotectic Alloys. In Proceedings VIIth European Symposium on Materials and Fluid Sciences in Microgravity, Oxford, UK, September 10-15, 1989, ESA SP-295, January 1990, pp. 115-120. (related ground experiments and KC-135 experiments)

(8) Input received from S. Rex (ACCESS e. V. (Aachen Center for Solidification in Space)), August 1993.

(9) Ecker, A.: Estarrungs front dynamik in Durchsichtigen Modellsystemen. Ph.D. Thesis, RWTH-Aachen, 1985.

(10) Boundary Layers in Transparent Melts. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 224-225. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Process Chamber Rack (Spacelab Double Rack)
**Processing Facility:** Holographic Optical Laboratory (HOLOP) (including a holographic interferometer)
**Builder of Processing Facility:** (1) Institute for Space Simulation, Deutsche Forschungs-und Versuchsanstalt für Luft-und Raumfahrt (DFVLR), Köln, Germany and (2) Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany <Note: The DFVLR is now called the Deutsche Forschungsanstalt für Luft-und Raumfahrt (DLR).>

**Experiment:**
<u>GETS - Boundary Layers in Solidifying Transparent Melts: Holographic Observation of Solidification Processes (PK HOL 04)</u>

"Transparent melts allow, by utilizing appropriate optical methods, the in-situ observation of solidification processes. The formation of microstructures depends on heat and mass transport in close vicinity to the solidification front. This means, in effect, the interplay of temperature, concentration, and natural convection interface layers....

"In the earth's laboratory diffusion and convection both function as transport mechanisms. While the first is always independent of gravity, the latter may both be gravity <u>in</u>dependent (e.g., in the form of [M]arangoni convection) as well as gravity <u>de</u>pendent (as natural convection). Alloys systems, in particular those that contain less dense components than the alloy base may, despite stable temperature layering, show natural convection while solidifying in the earth laboratory.... Obviously, this would influence diffusion layers close to the solidification front. To be able to separate the individual mechanisms a reference experiment under microgravity could be particularly valuable. In those cases, distinctions between various convection-causing phenomena could be more easily established." (2, p. 93)

This Spacelab D1 experiment was the second in a series of investigations designed by Ecker et al. to study the low-gravity solidification of a transparent material using holographic techniques (see Ecker, TEXUS 10). The specific objective of the experiment was to investigate the inter-dependencies of temperature, concentration, and fluid flow in the melt adjacent to the solid-liquid interface.

The experimental cell (called GETS) was contained within the Spacelab holographic optics laboratory, HOLOP. The cell was configured with (1) two, 60 mm x 10 mm x 15 mm cuvettes (which contained the sample materials), (2) six thermistors, (3) heating foils and Peltier elements (for heating and cooling of the samples), (4) an electronic circuit (for experiment control), and (5) various interfaces to the HOLOP.

The HOLOP module was designed to produce holograms which allowed the "...determination of phase shifts in the object beam evoked by transparent objects placed into the beam." (4, p. 214) Double exposure holograms produced by equipment within the module can provide an interference fringe pattern which illustrates optical density differences in the material system at two different exposure times. Because the interference fringe density is proportional to both the temperature and concentration changes between the two exposures, thermistors are included in the experiment cell to independently provide temperature data; thus, the concentration data can be resolved.

During the mission, two succinonitrile-ethanol sample mixtures (5 wt.% ethanol and 12 wt.% ethanol), contained within the cuvettes, were directionally solidified during the experiment using a power down process (fixed voltage for heaters, fixed currency for Peltier). <Note: The inflight procedural parameters of the investigation (heating and cooling temperatures, heating and cooling times, etc.) were not detailed in the available publications.>

It was reported that no results from this experiment were obtained. "Due to a malfunction of the film transportation system of the Hasselblad camera during the Mission, only 25 of the 85 planned holograms were taken. The evaluation of these holograms after the Mission showed that even these 25 had not been exposed." (4, p. 215) (See Reference (4) for a discussion of the sources of this failure.)

The original objectives of the experiment could not be achieved by evaluating the remaining documented experimental data (thermistor voltage, Peltier element currents, heating strip currents, and video exposures). However, it was reported that "...fluid flow was insignificant as expected and previously suggested by the TEXUS 10 experiment." (4, p. 215)

Some additional information concerning the experiment can be found in the references below.

**Key Words:** Metals and Alloys, Transparent Liquids, Melt and Solidification, Directional Solidification, Monotectic Compositions, Model Materials, Solidification Front Physics, Interface Phenomena, Interface Demarcation, Peltier Pulsing, Solid/Liquid Interface, Boundary Layer, Heat and Mass Transfer, Solutal Gradients, Thermal Gradient, Thermosolutal Convection, Diffusion, Interferometric Measurement, Holography, Photographic Difficulties

**Number of Samples:** two
**Sample Materials:** Two succinonitrile-ethanol mixtures: (1) 5 wt.% ethanol and (2) 12 wt.% ethanol.
**Container Materials:** glass, stainless steel, polyacetal

**Experiment/Material Applications:**
See Ecker, TEXUS 10.

The succinonitrile-ethanol mixtures (5 wt.% ethanol and 12 wt.% ethanol) were selected because "...preparatory tests showed that no relevant decomposition occurred at concentrations of less than 5 wt.% ethanol...." (4, p. 215)

**References/Applicable Publications:**
(1) Ecker, A., Schmitz, G., and Sahm, P. R.: Experiment: Grenzschichten in Erstarrenden Transparenten Schmelzen, (GETS). In BMFT/DFVLR Scientific Results of the German Spacelab Mission: D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, pp. 102-105. (abstract only)

(2) Ecker, S. and Sahm, P. R.: Boundary Layers in Solidifying Transparent Melts. In Scientific Goals of the German Spacelab Mission D1, WPF, 1985, pp. 93-95. (preflight)

(3) Ecker, A. and Sahm, P. R.: Solidification Front Dynamics in Model Systems. In Proceedings of the 4th European Symposium on Materials Sciences Under Microgravity, Madrid, Spain, April 5-8, 1983, ESA SP-191, June 1983, pp. 331-336. (related research and experiment setup)

(4) Ecker, A., Schmitz, G. J., and Sahm, P. R.: Boundary Layers of Solidifying Transparent Melts. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 212-216. (post-flight)

(5) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986. (post-flight; acceleration measurements)

(6) Input received from Principal Investigator A. Ecker, July 1988 and July 1993.

(7) Ecker, A., Frazier, D. O., and Alexander, J.I.D.: Fluid Flow in Solidifying Monotectic Alloys. In Metallurgical Transactions A, Vol. 20A, November, 1989, pp. 2517-2527. (Post-flight; ground-based results)

(8) Ecker, A.: Two-Wavelength Holographic Measurement of Temperature and Concentration During Alloy Solidification. Journal of Thermophysics and Heat Transfer, Volume 2, Number 3, July 1988, pp. 193-196. (related research)

(9) Ecker, A., Frazier, D. O., and Alexander, J.I.D.: Classification of Fluid Flow in Front of Solidifying Monotectic Alloys. In Proceedings VIIth European Symposium on Materials and Fluid Sciences in Microgravity, Oxford, UK, September 10-15, 1989, ESA SP-295, January 1990, pp. 115-120. (related ground experiments and KC-135 experiments)

(10) Input received from S. Rex (ACCESS e. V. (Aachen Center for Solidification in Space)), August 1993.

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**Experiment Origin:** Japan
**Mission:** STS Launch #13, STS-017 (STS 41-G, Challenger)
**Launch Date/Expt. Date:** October 1984
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** NASA Get Away Special (GAS) Canister G-032
Volume of Canister: 5 cubic feet
Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-032:** Nippon Electric Company (NEC), Yokohama, Japan/Asahi National Broadcasting Company, Tokyo, Japan
**Processing Facility:** Five small electrical furnaces
**Builder of Processing Facility:** Unknown, possibly Nippon Electric Company, Yokohama, Japan

**Experiment:**
(1) Indium Antimony Crystal Production, (2) Lead Zinc Alloy Production, and (3) Glass Composite Production

These three materials processing experiments were housed within the G-032 Get Away Special Canister during STS-017. (A fourth (fluids) experiment was also contained within G-032 during the mission and is applicable to this data base (see Asahi, STS-017, "Physics of Solids and Liquids" (Chapter 12)).) The materials experiments were the first in a series of investigations designed by Asahi National Broadcasting Company, Ltd. to study the low-gravity processing of indium antimony crystals, lead-zinc alloys, and glass composites.

Few details of these STS-017 materials processing experiments could be located. The STS-017 press kit, which was released prior to the launch of the STS-017 mission, briefly described the experiment objectives, setup, and expected results. Reportedly, the objective of the experiments was to "...produce five kinds of new materials simultaneously in space. The formation of crystals of three metal alloys and two glass composites in 5 small electrical furnaces in weightlessness will be observed." (1, p. 23) Reference (4) indicates that the new materials to be produced were "...a lead zinc alloy, a glass composite (or ceramic) and crystals of an indium-antimony mixture." (4, p. 22).

Reportedly, the materials science experiments went "smoothly" (4, p. 25) No other publications could be found that further discussed specific experiment objectives, setup, or results.

<Note: References (1),(3), and (4) indicate that five small furnaces were used. Reference (2) indicates that three small furnaces were used.>

**Key Words:** Metals and Alloys, Glasses, Binary Systems, Melt and Solidification, Ceramics, Solid/Liquid Interface, Sample Microstructure

**Number of Samples:** five
**Sample Materials:** Indium-antimony, lead-zinc, and a glass composite (or ceramic).
(In*Sb*, Pb*Zn*)
**Container Materials:** unknown

**Experiment/Material Applications:**
It was anticipated that low-gravity processing of the selected samples would result in materials with unique properties.

**References/Applicable Publications:**
(1) NASA Space Shuttle Mission 41-G Press Kit, October 1984, p. 23. (preflight)

(2) Cargo Systems Manual: GAS Annex for STS 41-G, JSC-17645 41-G, NASA JSC, 1984, September 4. (preflight)

(3) Shuttle Payload Creates Space Sculpture. Aviation Week and Space Technology, October 15, 1984, p. 21. (post-flight, very short description)

(4) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, the NASA GAS Team, 1989, p. 22. (post-flight; very brief description)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report, EAC-TR-RWR87-11, October 2, 1987. (Get Away Special Canister mission history)

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**Co-Investigator(s):** Fujimoto, K. (2), Goans, M. (GSFC Technical Manager) (3)
**Affiliation(s):** (1) Tokyo, Japan; (2) Asahi National Broadcasting Company, Ltd., Tokyo, Japan; (3) National Aeronautics and Space Administration (NASA), Goddard Space Flight Center (GSFC), Greenbelt, Maryland

**Experiment Origin:** Japan
**Mission:** STS Launch #16, STS-023 (STS 51-D, Discovery)
**Launch Date/Expt. Date:** April 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** NASA Get Away Special (GAS) Canister G-035
Volume of Canister: 5 cubic feet
Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-035:** Nippon Electric Company (NEC), Yokohama, Japan/Asahi National Broadcasting Company, Tokyo, Japan
**Processing Facility:** Five small electrical furnaces
**Builder of Processing Facility:** Unknown, possibly Nippon Electric Company, Yokohama, Japan

**Experiment:**
(1) Indium Antimony Crystal Production, (2) Lead-Zinc Alloy Production, and (3) Glass Composite Production

These three materials processing experiments were housed within the G-035 Get Away Special Canister during STS-023. (A fourth (fluids) experiment was also contained within G-035 during the mission and is applicable to this data base (see Asahi, STS-023, "Physics of Solids and Liquids" (Chapter 12)).) The materials experiments were the second in a series of investigations designed by Asahi National Broadcasting Company, Ltd. to study the low-gravity processing of indium-antimony crystals, lead zinc alloys, and glass composites (see Asahi, STS-017). Specifically, the investigations were designed to "...produce five kinds of new materials simultaneously in space." (1, p. 17)

The goals of this experiment were similar to that of the previous experiment which flew on STS-017. The "...formation of crystals of three metal alloys and two glass composites in five small electrical furnaces will be observed." (1, p. 17) The new materials to be produced were "...a lead-zinc alloy, a glass composite, and an indium-antimony mixture." (3, p. 2-2)

No other publications could be found that further discussed the specific experiment objectives, equipment setup, or post-flight results.

<Note: Reference (1) indicates that five small furnaces were used. Reference (3) indicates that three small furnaces were used.>

**Key Words:** Metals and Alloys, Glasses, Binary Systems, Melt and Solidification, Ceramics, Solid/Liquid Interface, Sample Microstructure

**Number of Samples:** five
**Sample Materials:** Indium-antimony, lead-zinc, and a glass composite
(In*Sb*, Pb*Zn*)
**Container Materials:** unknown

**Experiment/Material Applications:**
See Asahi (in this chapter), STS 41-B, "(1) Indium Antimony Crystal Production, (2) Lead-Zinc Alloy Production, and (3) Glass Composite Production."

**References/Applicable Publications:**

(1) NASA Space Shuttle Mission 51-D Press Kit, April 1985, p. 17. (very brief preflight summary)

(2) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report EAC-TR-RWR87-11, October 2, 1987. (Get Away Special Canister mission history)

(3) Cargo Systems Manual: GAS Annex for STS 51-D, JSC17645 51-D, NASA JSC, 1985, March 20. (preflight)


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**Co-Investigator(s):** The Alabama Space and Rocket Center (2) <Note: The Alabama Space and Rocket Center is now called The U.S. Space and Rocket Center>, Buckbee, E. O. (Customer) (3), Alabama A&M University (4)
**Affiliation(s):** (1) At Proposal Stage (1978): Johnson High School, Huntsville, Alabama, Interim: Alabama A&M University, Huntsville, Alabama and the Alabama-Mississippi Section of the American Institute of Aeronautics and Astronautics (AIAA), At Time of Launch (STS-017) and Currently: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama; (2) Huntsville, Alabama; (3) The Alabama Space and Rocket Center, Huntsville, Alabama; (4) Normal, Alabama

**Experiment Origin:** USA
**Mission:** STS Launch #13, STS-017 (STS 41-G, Challenger)
**Launch Date/Expt. Date:** October 1984
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** When Originally Proposed: High School Student Experiment
NASA Get Away Special (GAS) Canister G-007
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-007:** The Alabama Space and Rocket Center, Huntsville, Alabama
**Processing Facility:** Two miniature furnaces (lava cores wrapped with nichrome wire)
**Builder of Processing Facility:** NASA Marshall Space Flight Center, Huntsville, Alabama; Incon, Huntsville, Alabama; University of Alabama at Huntsville

**Experiment:**
Solidification of Lead-Antimony and Aluminum-Copper Alloys

This STS-017 experiment was the first in a series of investigations designed by Henderson et al. to study the low-gravity solidification of two hypereutectic alloys. (The experiment was one of four investigations housed within the G-007 Get Away Special canister during STS-017. One other experiment (of the four) is applicable to this data base (see Lee, STS-017 (Chapter 8)).) The specific objective of the experiment was to determine if the low-gravity processed samples exhibited any differences in alloy structure and strength when compared to similarly processed terrestrial samples.

Prior to the shuttle launch, one sample of lead-antimony and one sample of aluminum cooper were prepared. It appears that each sample was then placed in a separate furnace. Reference (1) (written prior to the launch of the STS-017 mission) detailed the expected furnace setup:

The setup "...will house two small cylinders that will encompass two miniature furnaces 4 1/2" H and 3/4" [d]iameter.... The wall thickness of the cylinders are 1/16" thick. The melting furnaces are made of lava cores and are wrapped with Nichrome wire (spring coils). The alloy samples are centrally located in the middle of each furnace core and can be heated to temperatures of up to 700 $^{o}$C...." (1, p. 113) It appears both of these cylinder/heater/sample assemblies were configured inside one internally insulated aluminum cylinder.

The 8-hour experiment was to be performed when the shuttle exhibited a "very low" gravity level (e.g., a sleeping period of the crew). It was expected that (1) the samples would be heated to 700 $^{o}$C (using a 28 V dc supply), and (2) a data acquisition and control unit associated with the experiment would measure operating temperatures, control the two furnaces, and perform other related tasks.

It was reported "that because of an operational error... [G-007] was not activated as scheduled. Consequently, no experimental data were acquired. A careful postflight analysis revealed no evidence of anomalies within the canister and plans were developed for a reflight on a subsequent shuttle mission." (3, p. 225) (See Henderson, 61-C for details of the subsequent shuttle experiment.)

**Key Words:** Metals and Alloys, Binary Systems, Hypereutectics, Melt and Solidification, Solid/Liquid Interface, Sample Microstructure, Material Strength, Acceleration Effects, Sample Not Processed As Planned

**Number of Samples:** two
**Sample Materials:** (1) lead-antimony and (2) aluminum-copper (Pb*Sb*, Al*Cu*)
**Container Materials:** Unclear, appears to have been aluminum. (Al*)

**Experiment/Material Applications:**
See Henderson, STS-032.

**References/Applicable Publications:**

(1) Henderson, A. J., Jr.: Project Explorer: Get Away Special #007. In Getaway Special Experimenter Symposium, NASA Goddard Space Flight Center, August 1-2, 1984, pp. 111-117, NASA CP-2324. (preflight)

(2) Henderson, A. J., Jr.: Project Explorer's Unique Experiments: Get Away Special #007. In Goddard Space Flight Center's Getaway Special Experimenter's Symposium, October 8-9, 1985, pp. 125-131, NASA CP-2401. (post STS-41-G, pre 61-C)

(3) Kitchens, P. H.: GAS-007: First Step in a Series of Explorer Payloads. In NASA Goddard Space Flight Center's 1986 Getaway Special Experimenter's Symposium, October 7-8, 1986, pp. 223-232, NASA CP-2438. (post-flight)

(4) Cargo Systems Manual: GAS Annex for STS 41-G, JSC-17645 41-G, September 4, 1984. (short description; preflight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(6) NASA STS 41-G Press Kit, October 1984. (preflight)

(7) Input received from K. K. Dannenberg (U.S. Space and Rocket Center), July 1993.

(8) Input received from Principal Investigator A. J. Henderson, Jr., August 1993.

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**Co-Investigator(s):** The Alabama Space and Rocket Center (2) <Note: The Alabama Space and Rocket Center is now called The U.S. Space and Rocket Center>, Buckbee, E. O. (Customer) (3), Alabama A&M University (4)
**Affiliation(s):** (1) At Proposal Stage (1978): Johnson High School, Huntsville, Alabama, Interim: Alabama A&M University, Huntsville, Alabama and the Alabama-Mississippi Section of the American Institute of Aeronautics and Astronautics (AIAA), At Time of Launch (STS-032) and Currently: National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), Huntsville, Alabama; (2) Huntsville, Alabama; (3) The Alabama Space and Rocket Center, Huntsville, Alabama; (4) Normal, Alabama

**Experiment Origin:** USA
**Mission:** STS Launch #24, STS-032 (STS 61-C, Challenger)
**Launch Date/Expt. Date:** January 1986 <Note: The experiment was originally scheduled (and ready) to fly on an earlier shuttle mission in June 1985 on the orbiter, Discovery.>
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** When Originally Proposed: High School Student Experiment
NASA Get Away Special (GAS) Canister G-007R
  Volume of Canister: 5.0 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-007R:** The Alabama Space and Rocket Center, Huntsville, Alabama
**Processing Facility:** Two miniature furnaces (lava cores wrapped with nichrome wire/spring coils)
**Builder of Processing Facility:** NASA Marshall Space Flight Center, Huntsville, Alabama; Incon, Huntsville, Alabama; University of Alabama at Huntsville

**Experiment:**
<u>Study of Solidification of Alloys in Zero-G</u>

This experiment was one of four investigations housed within the G-007 Getaway Special Canister during STS-032. (One other experiment (of the four) is applicable to this data base (see Lee, STS-032 (Chapter 8)).)

The experiment was the second in a series of investigations designed by Henderson et al. to study the low-gravity solidification effects of two hypereutectic alloys (see Henderson, STS-017). The major objective of the experiment was to determine if the low-gravity processed samples exhibited any differences in alloy structure and properties when compared to similarly processed terrestrial samples. Secondary objectives included determining "(1) whether any nitrides might be formed by leakage of gas into the samples and (2) the special surface effects applicable during solidification." (3, p. 226)

Prior to the shuttle launch, one sample of lead-antimony and one sample of aluminum-copper were prepared. <Note: The Principal Investigator reported that the aluminum copper sample "accidentally turned out to 'actually be' lead-antimony".> Each sample was then placed in its own furnace (oven #1 and oven #2) within a sealed, insulated, aluminum cylinder. These two miniature cylinders were housed inside a larger aluminum canister measuring 15" high and 6 1/2" in diameter.

During the mission, the two small samples were (1) heated from ambient temperature to a temperature above their respective eutectic temperatures, (2) "temperature controlled briefly," and (3) allowed to solidify. A data acquisition and control unit (DAQ2-K) associated with the experiment reported time, measured operating temperatures, controlled the furnaces, and performed other related tasks.

The Principal Investigator provided a more detailed discussion of the experimental setup and inflight procedures:

"The furnaces were made of lava cores 4 1/2"...[high and 3/4" in diameter] wrapped with Nichrome wire (spring coils). The lava cores could be heated-up to 800 degrees Celsius. Temperatures above 800 degrees caused a furnace melt-down and an eventual heater short. The power was supplied to the heaters via a 28 V DC battery supply." (12)

"This experiment had no apparent mechanical failures during the flight. At approximately 5:45 PM, Monday, January 13, 1986, oven #2 began.... [O]ven #2 ([containing the] proposed aluminum-copper sample) [had a] furnace set point... [of] 706 degrees Celsius. The temperature of the sample reached a maximum of 676 degrees Celsius, before dropping to a 15 minute regulated temperature of 636 degrees Celsius as programmed. Run #1 ran for 2 hours [and] 30 minutes total- before shut-down, recording the final sample temperature (after cool-down) at 176 degrees Celsius. The oven #2 surface temperature reached a maximum of 33 degrees Celsius. Target peak temperatures... [were] achieved.

"At approximately 4:32 PM, Tuesday, January 14, 1986, oven #1 began. [The] Oven #1 (lead-antimony sample) furnace set-point was at 501 degrees Celsius. The temperature of the sample reached a maximum of 464 degrees Celsius, before dropping to a 20 minute regulated temperature of 441 degrees Celsius, as programmed. Run #2 ran for 2 hours, [and] 15 minutes total- before experiment shut-down, recording a final sample temperature (after cool-down) at 106 degrees Celsius. The oven #1 surface temperature reached a maximum of 17 degrees Celsius. Again, target peak temperatures... [were] achieved." (12)

It was reported that "Several primary payloads [not including this one] on this Shuttle mission failed, so the mission was terminated early to avoid a potential weather problem at landing. This GAS can was commanded off after 3 days into the mission during deorbit preparations, and thus some experiment objectives were impacted. The nominal plan was 5 days of operation. As it turns out, the orbiter remained in orbit another 2 days because of weather anyway." (6, p. 36)

Preliminary analysis of the experimental payload indicated that both samples "...cooled, from the end of the temperature-regulation period to the phase-transition point (solid-liquid equilibrium line), at a rate of approximately 11-12 degrees C per minute." (3, p. 227)

Reportedly, at the time Reference (3) was published, the data were still being analyzed. The Principal Investigator provided the following summary of the results:

"Postflight results for this experiment uncovered several beneficial unknowns. Apparently during preflight preparations, a mistake was made with the aluminum-copper sample. The aluminum copper sample was not flown, as planned. Two lead-antimony samples were flown instead. This mistake had to occur due to a last minute rush.

"Oven #2 lead-antimony sample exhibited the best results overall, thanks to a little serendipity. During solidification, due to the higher temperature reached, a truly unique homogeneous nucleation phase was achieved. [The sample]... produced an evenly dispersed antimony material throughout the lead-based matrix. Moreover, nitrides appeared to be formed around the surface of the sample where it was originally attached or came into contact with the alumina crucible walls. The density differences did not caused a separation due to gravity, as predicted.

"Oven #1 lead antimony sample also demonstrated a unique homogeneous nucleation phase. However, it was not as pronounced as the Oven #2 sample.

"Therefore in conclusion and in comparison, the lead-antimony samples conducted in a 1-g environment did not demonstrate that a homogeneous nucleation phase could occur. The gravity-based studies exhibited the opposite results. Antimony tended to rise up around the top of the crucible walls. Yet the lead-based matrix tended to settle around the bottom of the crucible due to gravity and density differences.

"Also, observed in the ground-based studies, samples exhibited air pockets and large areas of porosity within the melts, whereas, no cavities of the sort were noticed in the microgravity samples.

"GAS #0007R experiment ran successfully during [the] STS... 61-C flight. The best available microgravity conditions were sufficient to compare low-gravity processed samples to earth-gravity processed samples.

"Furthermore, Henderson still advocates the need to refly his experiment to validate his initial results [and] to prove that the same results can be repeated, as well as, proposing another flight to evaluate the aluminum-copper species." (12)

**Key Words:** Metals and Alloys, Binary Systems, Eutectics, Hypereutectics, Melt and Solidification, Density Differences, Separation of Components, Buoyancy Effects Diminished, Solid/Liquid Interface, Growth Rate, Homogeneous Nucleation, Nucleation, Dispersion, Dispersion Alloys, Sample Microstructure, Surface Morphology, Porosity, Cavity, Crucible Effects, Material Interaction With Containment Facility, Gas Leakage, Material Strength, Superplastics, Space Structures, Processing Time Not As Long As Planned

**Number of Samples:** two
**Sample Materials:** lead-antimony
(Pb*Sb*)
**Container Materials:** alumina
(Al*O*)

**Experiment/Material Applications:**
Lead-antimony material in its eutectic structures should possess superplastic properties which makes it amenable for various defense related motor case structures and liners.

Aluminum-copper material in its eutectic structures should have improved structural properties which lends itself to more space related buss structures, as well as, numerous other commercial and government related high-strength light weight applications. (Reference (12))

**References/Applicable Publications:**


(1) Henderson, A. J., Jr.: Project Explorer: Get Away Special #007. In Getaway Special Experimenter Symposium, NASA Goddard Space Flight Center, August 1-2, 1984, pp. 111-117, NASA CP-2324. (preflight)

(2) Henderson, A. J., Jr.: Project Explorer's Unique Experiments: Get Away Special #007. In Goddard Space Flight Center's Getaway Special Experimenter's Symposium, October 8-9, 1985, pp. 125-131, NASA CP-2401. (post STS-41-G, pre 61-C)

(3) Kitchens, P. H.: GAS-007: First Step in a Series of Explorer Payloads. In NASA Goddard Space Flight Center's 1986 Getaway Special Experimenter's Symposium, October 7-8, 1986, pp. 223-232, NASA CP-2438. (post-flight)

(4) Cargo Systems Manual: GAS Annex for STS 41-G, JSC-17645 41-G, September 4, 1984. (short description; preflight)

(5) Get Away Special... the first ten years. Published by Goddard Space Flight Center, Special Payloads Division, The NASA GAS Team, 1989, p. 34. (post-flight; very short description)

(6) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987, p. 36. (Get Away Special canister mission history)

(7) NASA Press Kit, STS 61-C, December 1985. (preflight)

(8) Stluka, E.: STS-61 C, Columbia Flight Final Report. In NASA Goddard Space Flight Center's 1986 GAS Experimenter's Symposium.

(9) The National Technical Association's 55th Annual Convection, July 25-30, 1983, A Study of a Solidification of Alloys in Zero-G.

(10) Space Shuttle Get Away Special Experiment Performance Summary for Missions Through 1991, October 20, 1992.

(11) Input received from K. K. Dannenberg (The U.S. Space and Rocket Center), July 1993.

(12) Input received from Principal Investigator A. J. Henderson, Jr., August 1993.

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**Co-Investigator(s):** None
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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, M E D E A Double Rack
**Processing Facility:** Gradient Furnace with Quenching (GFQ)
**Builder of Processing Facility:** Dornier Friedrichshafen, Germany

**Experiment:**
Solidification Front Convection (MD-GFQ 02)

Thermosolutal convection is caused by constitutional buoyancy of a lighter phase in conjunction with the concentration profile immediately ahead of the solid/liquid interface. It was expected that this convection would not occur under low-gravity conditions if the correct processing parameters were selected.

This Spacelab D1 experiment was designed to study low-gravity, thermosolutal convection near the solid/liquid boundary of a directionally solidifying Al-Mg alloy. The specific objective of the investigation was to determine the effect of the solutal convection on the segregation behavior of the alloy. The selection of the employed processing parameters was based on the work of S. R. Coriell (see Reference (5)).

Prior to the shuttle flight, two Al-0.3 wt.% Mg rods were prepared. The materials were cast in an inert atmosphere and underwent subsequent mechanical operations. Each sample was then plasma-spray coated with a 100 micron thick $Al_2O_3$-MgO ceramic film and packed into a MgO powder "soft crucible" (0.3 mm thick). The crucible/sample was contained within a hermetically sealed, He filled steel cartridge. A boron-nitride cone was placed at the top of each sample to compensate for the expected volume increase. Four thermocouples, placed in a ceramic tube, were located in a central bore of each sample.

During the mission, each sample was partially melted and directionally solidified in the Gradient Furnace "with Quenching unit" (GFQ). Approximately 27% of the total sample length was directionally solidified under steady state conditions. A growth velocity of 0.37 to 0.40 mm/min and a thermal gradient of 12.95

to 13.38 K/mm were reported. The remainder of the remelted sample was then solidified by the application of a water spray, resulting in a quench rate of approximately 40 K/mm. This quenching process allowed preservation of the concentration profile ahead of the solid/liquid interface.

It was reported that one of the cartridges was damaged when removed from the furnace. "The analysis of the sample show[ed] that, due to sintering of the powder crucible the liquid aluminum alloy contacted the cartridge walls, resulting in lowering its melting temperature. Thus the cartridge was destroyed at the position of contact between sample and [Boron-Nitride]-cone...." (5, p. 225)

Post-flight analysis of the undamaged sample was performed using X-ray, metallographic, and microprobe techniques. <Note: The available publications did not detail the results of the broken sample.> X-ray analysis revealed no porosity throughout the sample length. However, there was a 7 mm diameter shrinkage hole at the top. No detaching of the sample from the ceramic skin was detected.

Metallographic examination revealed a planar solidification front with no cellular growth of the crystal. It was, therefore, concluded that "...the temperature gradient achieved in the experiment in front of the growing crystal appeared to fulfill the G/v-criterion [gradient/velocity criterion determined by stability diagram of S. R. Coriell]." (5, p. 226) (See Reference (5) for the stability diagram and complete discussion.) Microprobe determination of the longitudinal concentration profile and comparison of this data with similar 1-g processed samples indicated that purely diffusive controlled growth conditions occurred in the low-gravity processed samples.

**Key Words:** Metals and Alloys, Binary Systems, Melt and Solidification, Directional Solidification, Thermal Gradient, Solutal Gradients, Plasma Spray Coating, Thin Films, Coated Surfaces, Ceramics, Thermosolutal Convection, Marangoni Convection, Buoyancy Effects, Buoyancy Effects Diminished, Segregation, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Heat and Mass Transfer, Concentration Distribution, Sintering, Solid/Liquid Interface, Planar Solidification Interface, Steady-State Solidification, Quench Process, Growth Rate, Interface Shapes, Solidification Front Physics, Cellular Morphology, Porosity, Sample Microstructure, Crucible Effects, Material Interaction with Containment Facility, Wetting of Container, Volume

Compensation, Volume Change, Sample Shrinkage, Deterioration of Samples After Low-G Flight

**Number of Samples:** two
**Sample Materials:** Al-0.3 wt.% Mg
(Al*Mg*)
**Container Materials:** Each sample was plasma-spray coated with a 100 micron thick $Al_2O_3$-MgO ceramic film and packed into a MgO powder "soft crucible" (0.3 mm thick). The crucible/sample was contained within a hermetically sealed, He filled steel cartridge. A boron-nitride cone was placed at the top of each sample to compensate for the expected volume increase.
(Al*O*Mg*O*)

**Experiment/Material Applications:**
The final properties of a solidified melt are dependent on the heat and mass transport mechanisms active during crystallization. In order to control the final microstructure, these mechanisms must be distinguished. On Earth, thermal fluctuations, Marangoni convection, and constitutional buoyancy (of the lighter segregating phase) result in convective mixing. Solidification under low-gravity conditions allows the separation and examination of these effects.

An Al-Mg alloy was selected for this experiment because directional solidification experiments (and the corresponding concentration profile determination) had been performed prior to this study. Thus, comparison data already existed. The Mg content of the alloy was selected according to morphological and convective stability criteria determined by S. R. Coriell (see Reference (5)).

**References/Applicable Publications:**
(1) Rex, S. and Sahm, P. R.: Experiment Estarrungskonvection. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney, Germany, August 27-29, 1986, p. 73. (abstract only; post-flight)

(2) Rex, S. and Sahm, P. R.: Solidification Front Convection. In Scientific Goals if the German Spacelab Mission D1, WPF, 1985, pp. 100-101. (preflight)

(3) Sahm, P. R. and Rittich, M.: The Solidification Point Volume Increment in Solidification Front Dynamics. Adv. Space Res., Vol. 3, No. 5, 1983, p. 103. (related research; preflight)

(4) Sahm, P. R. and Tensi, H. M.: Adv. Space Research, 1, 1984, p. 97. (preflight)

(5) Rex, S. and Sahm, P. R.: Planar Front Solidification of Al-Mg Alloy-Crystallization Front Convection. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 222-230. (post-flight)

(6) Rex, S. and Sahm, P. R.: Thermosolutal Convection and Macro-segregation in an Alloy Directionally Solidified Under Reduced Gravity. In Proc. of the 3rd Int. Conference "Solidification Processing 1987," September 21-24, 1987, Sheffield, UK, The Inst. of Metals, London 1988, Book 421, pp. 102-103. (post-flight)

(7) Input received from Principal Investigator, S. Rex, August 1989 and August 1993

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**Affiliation(s):** (1) Grumman Aerospace Corporation, Bethpage, New York

**Experiment Origin:** USA
**Mission:** STS Launch #24, STS-032 (STS 61-C, Columbia)
**Launch Date/Expt. Date:** January 1986
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Payload Bay/Materials Science Laboratory (MSL-2) (The MSL-2 was attached to the Mission Peculiar Equipment Support Structure (MPESS))
**Processing Facility:** Automated Directional Solidification Furnace (ADSF-2 high temperature version). Each of the four furnaces within the ADSF had its own translation mechanism, platinum-rhodium heating element, adiabatic zone and quench block.
**Builder of Processing Facility:** Originally built by General Electric (GE) Corporation for the SPAR sounding rocket program; refurbished by GE, NASA Marshall Space Flight Center, Huntsville, Alabama, and Teledyne Brown Engineering, Huntsville, Alabama

**Experiment:**
Directional Solidification of Magnetically Aligned CoSm Composites (ADSF-2)

This investigation was the third in a series of experiments designed by Larson to study the directional solidification of magnetic composites in the space environment (see Larson, ASTP, STS-025). <Note: Larson's other solidification experiments can be found in Chapter 17 "Systems Exhibiting a Miscibility Gap".>

During the mission, the high temperature version of the Automated Directional Solidification Furnace (ADSF-2) was employed to process Co-Sm off-eutectic and peritectic magnetic composites. Directional solidification of the Co-Sm system was to complement earlier Bi-Mn studies which examined the role of convection in the resultant microstructure.

Post-flight analysis of the furnace ampoules indicated that only "...one of the four Co-Sm samples reached temperature and a portion of it was solidified directionally. The other samples were unmelted and, as a result will give no scientific return." (2, p. v)

Analysis efforts centered on the 21.7 wt.% Sm sample which was partially solidified. Reportedly, the sample typically lost 3.5 to 4.0 wt.% Sm during the processing "...due to the vaporization and reaction with the ampoule walls and residual atmosphere." (2, p. 29) "The telemetry record for the partially processed sample shows that it was equilibrated for 45 min at 1500 °C, that directional solidification was then initiated and continued for 2.05 hr, that the sample was then held stationary approximately 2.5 hr, and that it was then furnace cooled. As a consequence, the sample was partially solidified directionally and then solidified by gradient freeze and subsequent furnace (radial) cooling. The microstructure reflected the sequential nature of the solidification process; however, the solute redistribution (macrosegregation) appeared continuous. In addition, the morphologies seemed to indicate substantially greater interfacial undercooling than was documented in the ground-based studies....

"The undercooled sequence consisted of primary dendrite nucleation of the hypereutectic Co phase, rather than the equilibrium $Co_{17}Sm_2$ phase, secondary nucleation of $Co_{17}Sm_2$ dendrites and rapid growth of an undercooled rod eutectic followed, with a course dendritic structure in the last regions to solidify and in the regions where ripening has occurred. The relative amounts of the phases changed with bulk composition, but the structures were otherwise similar. The amount of undercooling was surprising in light of the fact that a proeutectic single crystal of $Co_{17}Sm_2$ was in direct contact with the melt.

"The sample was found to be free of porosity and exhibited a fully developed meniscus on the "hot" end, rather than the flattened meniscus typical of ground processing. The meniscus region provided an extraordinary opportunity to assess surface morphology, solidification mechanism, and solute redistribution. An intermediate dendritic case was documented for the first time, having both nonfaceted and faceted attributes." (2, p. v-vi) A more detailed discussion of this assessment of the surface morphology can be found in Reference (2), p. 38.

**Key Words:** Metals and Alloys, Binary Systems, Magnetic Composites, Eutectics, Hypereutectics, Faceted Eutectics, Peritectic Reaction, Melt and Solidification, Directional Solidification, Bridgman Technique, Translation Rate, Thermal Gradient, Thermosolutal Convection, Macrosegregation, Solutal Gradients, Undercooling, Solid/Liquid Interface, Interface Shapes, Meniscus Shape, Solidification Front Physics, Sample Microstructure, Surface Morphology, Nucleation, Dendrites, Dendritic Solidification, Dendritic Structure, Rod Structure, Platelet Habit, Single Crystals, Porosity, Vaporization, Crucible Effects, Material Interaction with Containment Facility, Sample Not Processed As Planned

**Number of Samples:** four
**Sample Materials:** Four cobalt-samarium composites: two of off-eutectic composition (Co-21.7 wt.% Sm) and two of peritectic composition (Co-26.5 wt% Sm)
(Co*Sm*)
**Container Materials:** Each cartridge consisted of an outer $Al_2O_3$ ampoule and an inner pyrolized boron nitride sample ampoule
(Al*O*, B*N*)

**Experiment/Material Applications:**
The study of Co-Sm eutectic, off-eutectic and peritectic magnetic composites complimented earlier Bi-Mn studies by Larson because
"o Each is a high-performance binary magnetic composite
o Each is a faceted/nonfaceted rod eutectic
o Each rod eutectic undergoes a morphological transition to a platelet morphology.

"The systems are different in that:
o The Co-Sm system distributes a soft magnetic phase in a hard magnetic phase rather than vice versa
o The Co-Sm system disperses a nonfaceted phase in a faceted matrix, the opposite of the Bi-Mn eutectic
o The $Co/Co_{17}Sm_2$ is a high volume, rather than low-volume, fraction eutectic
o The $Co/Co_{17}Sm_2$ morphological transition is initiated chemically rather than kinetically
o Both Co-Sm phases contract on solidification whereas Bi expands and MnBi contracts." (2, p. 23)

See also Larson, STS-025.

**References/Applicable Publications:**
(1) Space Shuttle Mission 61-C, NASA Press Kit, December 1985, p. 14. (short description; preflight)

(2) Larson, D. J., Jr. and Dressler, B. S.: Shuttle Mission 61-C Experiment MPS77F075 Final Flight Sample Characterization Report. Report RE-756, October 1988, Grumman Corporate Research Center, 45 pp. (post-flight)

(3) Materials Processing Experiments in Space: MSL-2 Payload. Document developed by Teledyne Brown Engineering under the Direction of the Application Payload Projects Office, Marshall Space Flight Center, pp. 1-2, and 7-8. (MSL-2 and processing facility)

(4) Space Shuttle Mission 51-G. NASA Press Kit, June 1985, p. 22. (used to determine applications of experiment)

(5) Larson, D. J., Jr. and Thompson, B. S.: Co-Sm Solidification. Research Report RE- Volume 3, Solid State Physics Research Directorate, Grumman Corporate Research Center, Bethpage, New York, Final Report on Experiment MPS77F075, 65 pp. (post-flight)

(6) Input received from Principal Investigator D. Larson, August 1993.

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**Co-Investigator(s):** Morril, V. (Teacher/Advisor) (2), McDonnell Douglas (Sponsor) (3), Chong, D. (Experiment Advisor) (4)
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**Experiment Origin:** USA
**Mission:** STS Launch #26, STS-26 (Discovery)
**Launch Date/Expt. Date:** September 1988
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** High School Student Experiment, Shuttle Student Involvement Program (SSIP), Middeck Experiment
**Processing Facility:** A titanium alloy wire, contained within a partially evacuated tube, was melted by resistance heating (passing an electric current through the wire).
**Builder of Processing Facility:** McDonnell Douglas, St. Louis, Missouri

**Experiment:**
Effects of Weightlessness on Grain Formation and Strength in Metals (SE82-4)

This STS-26 experiment was designed to determine the effects of low-gravity processing on the crystal grain reorganization of a titanium-aluminum-vanadium alloy. It was expected that space-processed samples would exhibit larger crystalline grains and increased alloy strength.

Four, 20-millimeter wire samples of a "Ti-6Al-4V (Ti-64)" alloy were chosen for the experiment. Two of the samples were 5 inches long, the other two were 2.5 inches long. During the experiment, the wires were resistance heated "...to either just above the melting temperature (1660 $^{o}$C) (the shorter wires) or just above the beta transus (1000 $^{o}$C) of the material (the longer wires)." (3, p. 4) (Reportedly, "At a temperature of 882 degrees C, the titanium-aluminum alloy crystal lattice network undergoes a metamorphosis from closely packed hexagonal crystals (alpha phase), to body-centered cubic crystals (beta phase)." (2, p. 36))

The space samples were compared with similar ground-based solidification efforts. Both space and ground-based shorter wires that melted illustrated heavy oxidation. "All these wires [space and ground] had broken during the performance of the experiment. The cross-sectional shapes changed from a round shape to... [a]... flattened shape.... The wire expanded during heating, and because it was constrained... it twisted and deformed in the process. The heavily oxidized material broke brittlely before melting...." (3, p. 13) Both space and ground-based longer wires had little or no oxidation. "The lower temperature allowed the formation of an alpha case, but there was no embrittlement of the wire through the cross section." (3, p. 19).

Reportedly, "The results of the experiment indicate that there is no difference between processing the titanium wire in space or on earth. The following observations were also made. There is an inadequate vacuum in the double... [Pyrex™] tubes in which the wires were supposed to melt. This lead to an oxidation of the very high temperature wires. Large grains were seen in the Ti-64 wire specimens as a result of the time that the wire was held in the beta region. Mechanical properties could not be determined because of insufficient material. All of these results are specific for the titanium wire and are not indicative of what would happen to other forms of titanium." (3, p. 1)

More details of the sample analysis can be found in Reference (3).

**Key Words:** Metals and Alloys, Ternary Systems, Melt and Solidification, Resistance Heating, Solid/Liquid Interface, Grain Structure, Grain Size, Sample Microstructure, Homogeneity, Oxidation, Coated Surfaces, Volume Expansion, Sample Deformation, Mechanical Strength, Material Strength, Vacuum, Processing Difficulties

**Number of Samples:** four
**Sample Materials:** filament (wire) samples of a titanium-aluminum-vanadium alloy
(Ti*Al*V*)
**Container Materials:** Pyrex™ tubes

**Experiment/Material Applications:**
If crystalline reorganization of the alloy results in larger grain size, a stronger, lightweight metal may be produced. Titanium alloy Ti-6Al-4V (Ti-64) "...was chosen for the experiment because of its wide-spread applications in the aerospace industry. Ti-64 is important because of its relatively low density and elevated melting temperature, Ti-64 was also chosen because

of its dimorphic nature, i.e., at 885 $^{o}C$... titanium undergoes an allotropic transformation from a hexagonal close-packed structure (alpha phase) to a body-centered cubic (beta phase)." (3, p.3)

**References/Applicable Publications:**

(1) Report to Educators. NASA Publication, Volume 16, No. 2, Vol. 16, Number 3, Summer/Fall 1988, p. 5. (preflight)

(2) NASA Marshall Space Flight Center Space Shuttle Mission STS-26 Press Kit, September 1988, pp. 36-37. (preflight)

(3) Shuttle Student Involvement Project: The Effects of Weightlessness on Grain Formation and Strength in a Metal. McDonnell Douglas Missile Systems Company, St. Louis, Missouri, MDC E3449. (post-flight)

(4) Input received from Experiment Investigator, August 1989 and July 1993.

(5) The Effect of Microgravity on Grain Formation and Strength in Metals. In Shuttle Student Involvement Program (SSIP) Final Reports on Experiments Flown, NASA/JSC Internal Note, JSC 24005, October 20, 1989. (post-flight)

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# CHAPTER 15

# PHYSICAL CHEMISTRY

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**Experiment Origin:** USA
**Mission:** Skylab Program
**Launch Date/Expt. Date:** Not applicable. Although this investigation was originally chosen as a Skylab student experiment, the low-gravity research was never performed on the mission because "Skylab... [was] unable to to provide the absolutely stable operating site for the time periods up to 1 month that the experiment... [required]."(1, p. 12-86)
**Launched From:** Not applicable
**Payload Type:** Skylab Student Project, High School Student Experiment, Skylab Manned Environment
**Processing Facility:** Copper sulfate crystal immersed in a constant temperature liquid
**Builder of Processing Facility:** Not applicable

**Experiment:**
Brownian Motion (ED75)

Colloidal particles, suspended (for example) in a liquid medium, often vibrate erratically. This vibratory, erratic motion, known as Brownian motion, occurs because the colloidal particles are subjected to unbalanced molecular impacts by the liquid molecules.

The objective of this Skylab student experiment was to study Brownian motion under low-gravity conditions. It was proposed that a crystal of colored salt (copper sulphate) be held in place at the bottom of a graduated cylinder filled with a constant temperature liquid. As the crystal dissolved, periodic photographs would be made. If undisturbed, the color would migrate throughout the cylinder by Brownian motion.

Although the experiment was initially selected as a candidate investigation for the Skylab mission, it was determined prior to the flight that "Skylab... did not provide a perfectly stable platform for such an experiment. Maneuvers to maintain or change the orientation of the vehicle due to pumps, fans, and other machinery all produced small accelerations that would have precluded the satisfactory performance of... [the]... experiment." (2, p. 92) Thus, the experiment was not performed.

<Note: See McGee, Skylab (Chapter 1) for a more detailed description of colloidal suspensions.>

**Key Words:** Physical Chemistry, Brownian Motion, Collisions, Solid/Liquid Collision, Colloidal Chemistry, Particle Motion, Particle Dispersion, Particle Mobility, Suspension of Particles, Dissolution, Diffusion, Salt Crystals, Solvent, Mass Transfer, Solid/Liquid Interface, Accelerations/Vibrations Produced by Onboard Equipment, Acceleration Effects, Sample Not Processed As Planned

**Number of Samples:** not applicable
**Sample Materials:** copper-sulfate
(Cu*S*)
**Container Materials:** not applicable

**Experiment/Material Applications:**
"Such an experiment is a classical high school demonstration of the kinetic theory of matter...." (2, p. 91)

**References/Applicable Publications:**

(1) "Brownian Motion (ED75)." In MSFC Skylab Mission Report-Saturn Workshop, Skylab Program Office, George C. Marshall Space Flight Center, Alabama, NASA TM X-64814, October 1974, pp. 12-86.

(2) Skylab, Classroom in Space, edited by Summerlin, L. B., NASA SP-401, 1977, pp. 89-92.

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**Experiment Origin:** USA/Federal Republic of Germany
**Mission:** Skylab, SL-3, Second Manned Mission
**Launch Date/Expt. Date:** September 1973 (month experiment was completed)
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Science Demonstration, Skylab Manned Environment
**Processing Facility:** Food storage refrigerator, scopedex pill dispenser
**Builder of Processing Facility:** Not Applicable

**Experiment:**
Ice Melting (SD16- TV111)

This Skylab science demonstration was designed to simulate low-gravity, containerless, materials-melting. The specific objective of the experiment was to visibly study a melting process not governed by gravity-driven convective flows.

During the Skylab SL-3 mission, a plastic, cylindrical pill container (30 mm diameter by 75 mm long) was filled with drinking water. An astronaut "...shook the water to the point where it did not have any bubbles to speak of in the solid mass." (1, p. 1) The water was frozen using the onboard food freezer. (A wooden cotton swab was included in the container to provide support for the ice.) After freezing, the cylinder of ice was removed from the pill container and the ice was weighed. Reportedly, there were 39.7 +/- 0.2 grams of ice available for melting. The ice, which was mounted so the cylinder could melt freely, was placed beside a ruler, thermometer and timer (all of which was in the field of view of a 16 mm camera). The camera was used to photograph the melting process approximately every 10 minutes (22 photographs total).

The low-gravity experiment was duplicated on Earth using the same experimental conditions (75% $O_2$, 25% $N_2$) and temperature (78 $^{o}$F, 25.5 $^{o}$C). During the ground-based experiment, the melted water was collected in a beaker and the volume measurements (+/- 0.1 $cm^3$) were made every 5 minutes. This allowed an accurate determination of the accumulative heat input to the material.

Initial post-flight examination of the returned photographs indicated:

(1) The total melting time of the ice cylinder was 190 +/- 5 minutes. (The total melting time of the identical Earth-based cylinder was 130 +/- 2 minutes.)
(2) In space, the cylindrical ends melted first while the diameter of the cylinder remained approximately constant.
(3) The water from these melting ends was driven onto the cylindrical surfaces by surface tension.
(4) The overall shape progressed from cylindrical to spherical with an intermediate, nearly ellipsoidal form. The spherical shape became evident at about half the melting time when the water totally surrounded the remaining ice.

Dimensional analysis of the ice was performed using the returned photographs. It was reported by the investigator that the photographs were slightly out of focus. Therefore the determination of the solid/liquid interface was not very accurate. The accuracy of the data was estimated to be about 10%. The volumes of the ice and water, as a function of time, were determined. "It should be mentioned that the change in volume of ice or water with time is a smooth non-linear curve for the ground experiment, whereas it is a linear function of time for the skylab experiment (t > 70 min)." (1, p. 3) These curves were used to calculate the total heat flow into the ice (using the heat of fusion of 79.8 cal/gram and the known mass and volume of the ice). The individual heat inputs to the ice from radiation, conduction, and convection were theoretically calculated for both ground-based and low-gravity experiments (see Reference (1) for details of these calculations). The sum of these values was then compared to (1) the calculated values from the photographs of the flight experiment and (2) the volume measurements from the ground-based experiment.

It was reported that, for the ground-based experiment, the theoretical and experimental values of total heat flow into the ice agree to within 6% over the duration of melting. "Therefore, since both values agree, it can be concluded from these calculations that, for the ground-based melting, the total heat flow is achieved by 55% convection, 38% radiation, 7% conduction." (1, p. 6)

For the Skylab experiment, "An accurate record of the quantitative analysis for the first 10 min of melting... [was]... not available [no photographs were taken during this time]. However, very good data of volumes... [were]... obtained for t > 50 min up to the total melting time." (1, p. 6) Comparison of the theoretical calculation of heat flow to those determined from the experiment (t > 60 min) indicated no contribution from convection. Therefore, it was concluded that in the low-gravity experiment the latent heat of melting was supplied by 81% radiation and 19% conduction.

Two major conclusions were reported:

(1) As illustrated in the Skylab demonstration, surface tension effects are important during low-gravity containerless melting.

(2) On Earth, convection is the dominant heat transfer mechanism (for supply of latent heat of melting); in space, radiation is the dominant heat transfer mechanism.

**Key Words:** Physical Chemistry, Phase Transition, Ice, Freezing, Melting (Without Resolidification), Containerless Melt, Containerless Processing Applications, Transparent Liquids, Model Materials, Surface Tension, Surface Energy, Free Surface, Sphericity, Heat Transfer, Heat of Fusion, Radiation, Conduction, Convection, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Bubbles, Bubble Removal, Solid/Liquid Interface, Liquid/Gas Interface, Photographic Difficulties

**Number of Samples:** one
**Sample Materials:** water from Skylab drinking water dispenser; wooden cotton swab
**Container Materials:** not applicable

**Experiment/Material Applications:**
"The ice/water system was selected because of the availability of the necessary materials onboard, the negligible impact on the mission, the ready availability of all significant data, and the good visibility of the solid/liquid interface which represented a transparent furnace." (1, p. 1)

Information from this experiment provided knowledge of containerless melting and heat transfer in the absence of convective flow.

**References/Applicable Publications:**

(1) Otto, G. H. and Lacy, L. L.: Observations of the Liquid/Solid Interface in Low-Gravity Melting. In American Institute of Aeronautics and Astronautics and American Geophysical Union, Conference on Scientific Experiments of Skylab, Huntsville, Alabama, October 30-November 1, 1974, AIAA, 8 pp. (NASA supported research, post-flight)

(2) Otto, G. H. and Lacy, L. L.: Observation of the Liquid Solid Interface in Low Gravity Melting. In Material Sciences in Space with Applications to Space Processing, AIAA Progress Series in Astronautics and Aeronautics, Edited by Leo Steg, Vol. 52, p. 483, 1977. (post-flight)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of the Measurement and Characterization of the Acceleration Environment on Board the Space Station, August 11-14, 1986, Guntersville, Alabama, p. 9-1. (acceleration measurements)

(4) Naumann, R. J. and Herring, H. W.: Ice Melting Demonstration. In Materials Processing in Space: Early Experiments, NASA SP-443, p. 87. (post-flight)

(5) TV111-Ice Melting. In MSFC Skylab Corollary Experiment Systems, Mission Evaluation, NASA TM X-64820, September 1974, pp. 7-39 - 7-43. (post-flight)

(6) Bannister, T. C.: Skylab III and IV Science Demonstrations Preliminary Report, NASA TM X-64835, March 1974, pp. 4-6. (post-flight)

(7) Otto, G. H. and Lacy, L. L.: The Liquid/Solid Interface in Low Gravity Melting. In Scientific Investigations on the Skylab Satellite, AIAA Progress Series in Astronautics and Aeronautics, Vol. 48, p. 455, 1976. (post-flight, short description)

(8) Naumann, R. J. and Mason, E. D.: Ice Melting. In Summaries of Early Materials Processing in Space Experiments, NASA Technical Memorandum NASA TM-78240, August 1979, p. 38. (post-flight)

(9) Ice Melting. In MSFC Skylab Mission Report- Saturn Workshop, NASA TM X-64814, October 1984, pp. 12-88.

(10) Input received from Principal Investigator G. H. Otto, July 1989 and August 1993.

(11) MSFC Science Demonstrations Performed by Pilot J. Lousma on Skylab 3. Quick Look Report, November 5, 1973, NASA Marshall Space Flight Center, Space Sciences Laboratory.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 1
**Launch Date/Expt. Date:** December 1977
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-2: electrolytic cell
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Dispersion Electrolysis

During the electrodeposition of a dispersion, the powdered, non-metallic phase (the phase to be dispersely embedded) must be suspended in an electrolyte. On Earth, processing difficulties may occur (1) due to the large (gravity-induced) density differences between the particles and electrolyte or (2) when the cathode, which is to receive the dispersion layer, has a geometrically complicated shape (resulting in an uneven embedment of particles). Low-gravity electrodeposition experiments (1) will allow investigation of deposition mechanisms without gravitational influence and (2) may permit plating of surfaces with complicated shapes. <Note: See Reference (5) for a detailed discussion of the electrodeposition process; see also Riley, Consort 1 (in this chapter) for related experiments.>

This TEXUS 1 experiment was the first in a series of investigations designed by Ehrhardt et al. to study the low-gravity electrodeposition of a dispersed material.

The experiment was performed in an electrolytic cell configured in the TEXUS Experiment Module TEM 06-2. The cell contained (1) a $Ni(NH_2SO_3)_2,4H_2O$-based electrolyte, (2) a suspended phase of (approximately 0.3 micron diameter) $Al_2O_3$ alumina powder, (3) a Ni anode and (4) a "profiled" Au cathode.

During the ascent phase of the rocket flight, the electrolyte was to be stirred to prevent settling of the alumina particles. Once low-gravity conditions were achieved, the electrolysis process was initiated without stirring.

During the low-gravity experiment, both the metal ions (from the electrolyte) and the dispersed non metallic particles moved towards the cathode and were embedded into the depositing layer.

Reportedly, a 5 micron thick Ni-$Al_2O_3$ layer was electro-co-deposited on the Au cathode. (2) This deposited layer contained several cracks, the extent of which increased with decreasing current density. Concentration of the $Al_2O_3$ particles in the deposited layer was lower than anticipated. This result was attributed to a fault in the stirring unit (which occurred during the ascent phase). Further, the electric current efficiency was 10% less than on Earth (more hydrogen was formed) but the throwing power of the electrolyte was higher." (2)

"The corrosion resistance of the layer deposited under microgravity conditions was much higher. It was thus demonstrated that it is possible to perform electro-deposition experiments under microgravity conditions and to investigate the incorporation of particles in the absence of buoyancy-driven convection and sedimentation." (6, p. 90)

**Key Words:** Physical Chemistry, Electrodeposition, Codeposition, Electrocodeposition, Dispersion Layers, Thin Films, Coated Surfaces, Plating, Substrates, Electric Field, Cathode, Anode, Electrodes, Electrolyte Solution, Ionic Solution, Electrolysis, Powders, Particle Dispersion, Homogeneous Dispersion, Particle Mobility, Particle Motion, Particle Distribution, Suspension of Particles, Stirring of Components, Shear Forces, Density Difference, Concentration Distribution, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Sedimentation, Hydrogen Formation, Bubbles, Bubble Formation, Cracks, Corrosion, Surface Morphology, Metallic Matrix, Solid/Liquid Interface, Superconductivity, Contained Fluids, Launch-Induced Fluid Motion, Hardware Malfunction

**Number of Samples:** one
**Sample Materials:** nickel anode, gold cathode, $Ni(NH_2SO_3)_2{,}4H_2O$ (Ni-sulfamate) electrolyte, alumina particles ($Al_2O_3$)
(Ni*, Au*, Ni*N*H*S*O*, Al*O*)
**Container Materials:** stainless steel coated with PTFE (polytetrafluoroethylene)

**Experiment/Material Applications:**
Non-metallic dispersions within a metallic matrix can improve the properties of the parent metal. Depending on the nature of the metallic matrix and dispersion materials, electro-deposited composite materials can have significantly improved (1) high-temperature stability, (2) corrosion resistance, (3) wear resis-

tance, (4) sliding capacity (lubrication), and/or (5) superconductivity.

**References/Applicable Publications:**

(1) Ehrhardt, J.: Dispersion Electrolysis Under Zero Gravity Under Spacelab Rocket Program Texus 1--A Spacelab Experiment Feasibility Study. BMFT-FB-W-79-30. (in German)

(2) Ehrhardt, J.: Dispersionselektrolyse unter Schwerelosigkeit; Forschungsbericht W 79-90 des BMFT. (in German)

(3) Ehrhardt, J.: Versuche zur elekrolytischen Metallabscheidung unter Schwerelosigkeit; Galvanotechnik, 72 (1981) 1, pp. 13-18. (in German)

(4) Input received from Experiment Investigator, July 1988 and July 1993.

(5) Ehrhardt, J.: Investigations into the Electrodeposition of Dispersion Layers Under Zero-G-Conditions. In Translation of BMFT Report BMFT-FB-W-77-11, ESA Document ESA-TT-574, November 1979. (discussion of electrodeposition, does not discuss TEXUS experiments)

(6) Dispersion Layers by Electrodeposition. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 90-91. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 4
**Launch Date/Expt. Date:** May 1981
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-5: electrolytic cell
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Dispersion Electrolysis

This TEXUS 4 experiment was the second in a series of investigations designed by Ehrhardt et al. to study the low-gravity electrodeposition of a dispersed material (see Ehrhardt, TEXUS 1). The specific objectives of the experiment were to (1) determine if under low-gravity conditions the dispersed particles (a) coagulate or (b) drift separately toward the cathode (as they do on Earth) and (2) understand fully those electrodeposition mechanisms which would permit improvement of ground-based processes. <Note: See Riley, Consort 1 (in this chapter) for a related experiment.>

The experiment was performed in an electrolytic cell configured in the TEXUS Experiment Module TEM 06-5. The cell contained (1) a Ni-sulfamate electrolyte, (2) a suspended phase of (approximately 0.3 micron diameter) $Al_2O_3$ particles, (3) a Ni anode, and (4) an Au cathode. (These materials were the same as those used during the TEXUS 1 Mission.) The data recorded during the flight included (1) the electric current through the cell, (2) the gas pressure variations, and (3) the temperature variations. A new stirring device (improved after TEXUS 1) was employed.

During the electrolytic process, (1) hydrogen bubbles formed at the cathode and (2) a 7 micron thick layer of Ni-$Al_2O_3$ was deposited at the cathode under reduced gravity conditions. Reportedly, The layer contained a more homogeneous distribution of particles than was achieved during ground-based studies. Results from a later experiment (see Ehrhardt, TEXUS 7) led to the conclusion that the formation of bubbles during the TEXUS 4 experiment helped to prevent coagulation of the alumina particles. "...the single particles which were co-deposited with the Ni layer were detached from loosely coagulated particles by motions in the electrolyte induced by the formation of hydrogen bubbles." (3, p. 94)

The low-gravity, deposited layer "...also had a higher density than the 7-9... [micron]... thick layer obtained on Earth, which had a surface density of... 4000[particles]/$cm^2$. In addition, the layer deposited in space was almost amorphous and was much less prone to corrosion by artificial sea water than the layer deposited on Earth. In the latter, the Ni grains were large enough to exhibit crystalline behavior." (3, p. 92) Due to contamination of the gold plated cathode surface (prior to launch), good adhesion between the substrate and deposited material was not achieved.

Reportedly, the low-gravity current efficiency of the deposition process was reduced by about 10% from the 1-g current efficiency. <Note: It appeared that this reduction in current efficiency was caused by the hydrogen bubble formation. It also appeared that the particles drifted separately toward the cathode (without prior coagulation) during the rocket experiment.>

**Key Words:** Physical Chemistry, Electrodeposition, Codeposition, Electrocodeposition, Dispersion Layers, Thin Films, Coated Surfaces, Plating, Substrates, Electric Field, Cathode, Anode, Electrodes, Electrolyte Solution, Ionic Solution, Electrolysis, Powders, Particle Dispersion, Homogeneous Dispersion, Particle Mobility, Particle Motion, Particle Distribution, Suspension of Particles, Coagulation, Stirring of Components, Shear Forces, Density Difference, Buoyancy-Driven Convection, Sedimentation, Hydrogen Formation, Bubbles, Bubble Formation, Corrosion, Surface Morphology, Amorphous Materials, Grain Size, Contamination Source, Metallic Matrix, Solid/Liquid Interface, Superconductivity, Contained Fluids

**Number of Samples:** one
**Sample Materials:** nickel anode, gold cathode, nickel-sulfamate electrolyte, alumina particles, $Al_2O_3$
(Ni*, Au*, Al*O*, Ni*N*H*S*O*)
**Container Materials:** stainless steel coated with PTFE (polytetrafluoroethylene)

**Experiment/Material Applications:**
See Ehrhardt, TEXUS 1.

**References/Applicable Publications:**
(1) Ehrhardt, J.: Dispersionselectrolyse unter Schwerelosigkeit im Spacelab-Vorprogramm TEXUS 4. Final Report 01 QV 219-AK-SN/A-SLN 7910-5, DFVLR/BPT, April 1982. (in German)

(2) Input received from Experiment Investigator, July 1988 and July 1993.

(3) Dispersion Electrolysis. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 92-95. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 7
**Launch Date/Expt. Date:** May 1983
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-5: electrolytic cell
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Dispersion Electrolysis

This TEXUS 7 experiment was the third in a series of investigations designed by Ehrhardt et al. to study the low-gravity electrodeposition of a dispersed material (see Ehrhardt, TEXUS 1, TEXUS 4). The specific objective of the experiment was to determine whether dispersed phase particle transport is dominated by electrophoresis or diffusion. <Note: See Riley, Consort 1 (in this chapter) for a related experiment.>

The experiment was performed in an electrolytic cell configured in the TEXUS Experiment Module TEM 06-5. The cell contained (1) a Ni-sulfate electrolyte, (2) a suspended phase of $Al_2O_3$ particles, (3) a Ni anode, and (4) a gilt brass cathode. (Except for the brass cathode, these materials were the same as those employed during the TEXUS 1 and TEXUS 4 experiments.)

A discussion of the TEXUS 7 experimental procedure is reported in Reference (1). <Note: A translated version of this Reference was not available to aid in the preparation of this experiment summary.>

Post-flight, it was reported that an 8 micron thick layer (primarily composed of Ni) was deposited on the cathode (1) without codeposition of $H_2$ bubbles and (2) without any contamination. A current efficiency of 98% was achieved. Unexpectedly, almost no $Al_2O_3$ particles (except for a few largely built agglomerates) were embedded in the well-bonded, crack-free layer. (Such agglomerates are observed during Earth-processing.)

It was also reported that, since the cathode surface in this experiment was free from contaminations and therefore free from $H_2$ bubbles, the formation of bubbles during the earlier TEXUS 4 experiment helped prevent coagulation of the alumina particles. "...in the previous experiment [TEXUS 4], the single particles which were co-deposited with the Ni layer were detached from loosely coagulated particles by motions in the electrolyte induced by the formation of hydrogen bubbles." (3, p. 94). (See Ehrhardt, TEXUS 4 for further discussion of the hydrogen bubbles.)

It was concluded that "...electrophoresis is not the dominating force of the [particle] transport..." since (1) the time available for the experiment was too short (340 seconds) to allow the diffusion of the agglomerates and (2) only a few agglomerates were embedded in the Ni layer.

**Key Words:** Physical Chemistry, Electrodeposition, Codeposition, Electrocodeposition, Dispersion Layers, Thin Films, Coated Surfaces, Plating, Substrates, Electric Field, Cathode, Anode, Electrodes, Electrolyte Solution, Ionic Solution, Electrolysis, Powders, Particle Dispersion, Particle Mobility, Particle Motion, Particle Distribution, Suspension of Particles, Particle Agglomeration, Adhesion of Metals, Electrophoresis, Coagulation, Diffusion, Density Difference, Buoyancy-Driven Convection, Sedimentation, Corrosion, Surface Morphology, Metallic Matrix, Solid/Liquid Interface, Superconductivity, Contained Fluids

**Number of Samples:** one
**Sample Materials:** nickel anode, gold-cathode, nickel sulfate electrolyte, alumina particles, $Al_2O_3$
(Ni*, Ag*, Al*O*, Ni*S*O*)
**Container Materials:** stainless steel coated with PTFE (polytetrafluoroethylene)

**Experiment/Material Applications:**
See Ehrhardt, TEXUS 1.

**References/Applicable Publications:**
(1) Ehrhardt, J.: Dispersionselectrolyse unter Schwerelosigkeit im Spacelab vorprogramm TEXUS 7; Final Report 01 QV 219-AK-SN/A-SLN 7910-5, DFVLR/BPT, November 1983. (in German)

(2) Input received from Experiment Investigator, July 1988 and July 1993.

(3) Dispersion Electrolysis. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 94-95. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 9
**Launch Date/Expt. Date:** May 1984
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-5: electrolytic cell
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Dispersion Electrolysis

This TEXUS 9 experiment was the fourth in a series of investigations designed by Ehrhardt et al. to study the low-gravity electrodeposition of a dispersed material (see Ehrhardt, TEXUS 1, TEXUS 4, TEXUS 7).

Previous low-gravity experiments in this investigative series indicated that (1) coagulation of particles had prevented their co-deposition in an electrolytically deposited metallic layer and (2) particle separation could be achieved by application of a weak shear stress (stirring). Therefore, the objective of this experiment was to verify that particle separation could be achieved by the application of weak shear stress. <Note: see Riley, Consort 1 (in this chapter) for a related experiment.>

The experiment was performed in an electrolytic cell configured in the TEXUS Experiment Module TEM 06-5. The cell contained (1) a Ni-sulfate electrolyte, (2) a suspended phase of $Al_2O_3$ particles (60 g/l, 0.3 micron), (3) a 30 $cm^2$ Ni anode, and (4) a 10 $cm^2$ disk-shaped gilt brass cathode.

The TEXUS 9 experiment was performed in two phases. First, electrodeposition took place without stirring of the electrolyte; second, electrodeposition took place while the electrolyte was gently stirred.

It was reported that during the first phase of the experiment the particles coagulated and no single particles were available for co-deposition. However, during the second stirred phase, single particles were incorporated in the deposited Ni layer.

It was concluded that under low-gravity conditions, "...a weak shearing force is sufficient to release single particles from non-compact coagulates for galvanic co-deposition." (3, p. 96)

<Note: At the time this experiment summary was prepared, a publication (written in English) which described the quality of the deposited layers resulting from either phase of the experiment, could not be located.>

**Key Words:** Physical Chemistry, Electrodeposition, Codeposition, Electrocodeposition, Dispersion Layers, Thin Films, Coated Surfaces, Plating, Substrates, Electric Field, Cathode, Anode, Electrodes, Electrolyte Solution, Ionic Solution, Electrolysis, Powders, Particle Dispersion, Particle Mobility, Particle Motion, Particle Distribution, Suspension of Particles, Stirring of Components, Shear Forces, Coagulation, Density Difference, Buoyancy-Driven Convection, Sedimentation, Corrosion, Surface Morphology, Metallic Matrix, Solid/Liquid Interface, Superconductivity, Contained Fluids

**Number of Samples:** one
**Sample Materials:** nickel anode, gilt brass cathode, Ni-sulfate electrolyte, alumina particles, $Al_2O_3$
(Ni*, Au*, Ni*S*O*, Al*O*)
**Container Materials:** stainless steel coated with PTFE (polytetrafluoroethylene)

**Experiment/Material Applications:**
See Ehrhardt, TEXUS 1.

**References/Applicable Publications:**
(1) Ehrhardt, J.: Dispersionselectrolyse unter Schwerelosigkeit im Rahmen von TEXUS 9. Final Report 01 QV 014-AK/SN DFVLR/BPT, November 1984. (in German)

(2) Input received from Experiment Investigator, July 1988 and July 1993.

(3) Dispersion Electrolysis. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 96-97. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 1
**Launch Date/Expt Date:** December 1977
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-2 (experiment cell containing (1) a half gold-plated Pt cathode, (2) a copper anode, and (3) a $Na_2SO_4$ water solution).
**Builder of Processing Facility:** Unknown

**Experiment:**
Cathodic Hydrogen Formation

This TEXUS 1 experiment was the first in a series of investigations designed by Raub to study low-gravity hydrogen bubble generation on a cathode surface. It was anticipated that in the absence of the gravity-induced buoyancy forces, hydrogen bubbles formed on the surface of a cathode would remain on the surface. If this phenomenon occurred, a film of hydrogen would be created which would completely isolate the electrode.

The investigation was performed in an experimental cell housed within the TEXUS Experiment Module TEM 06-2. The cell contained (1) a half gold-plated Pt cathode, (2) a copper anode, and (3) a $Na_2SO_4$ water solution. During the mission, the hydrogen generation on the cathode was observed with a cine camera as a potential was imposed across the anode and cathode.

It was reported that while under low-gravity conditions (1) the bubbles which formed on the gold and platinum surfaces during the TEXUS mission were smaller than those formed under 1-g conditions, (2) the generated hydrogen bubbles remained in front of the cathode surface but did not isolate the electrode, (3) hydrogen, copper, and copper oxide were deposited on the cathode surface, (4) the potential drop through the cell (at a constant current) was constant during the study indicating the electrolyte films formed between the bubbles were still conducting, and (5) a small amount of motion of the bubble agglomerates was attributted to either (a) surface tension driven convection induced by local heating caused by the illumination system or (b) residual accelerations of the rocket.

**Key Words:** Physical Chemistry, Electrodeposition, Thin Films, Coated Surfaces, Electric Field, Conduction, Cathode, Anode, Electrolysis, Electrodes, Electrolyte Solution, Transparent Liquids, Aqueous Solutions, Hydrogen Formation, Bubbles, Bubble Formation, Particle Agglomeration, Buoyancy Effects, Surface Tension-Driven Convection, Marangoni Convection, Thermocapillary Convection, Solid/Liquid Interface, Illumination System, Contained Fluids, Thermal Environment More Extreme Than Predicted, Acceleration Effects

**Number of Samples:** one
**Sample Materials:** (1) a half gold-plated Pt cathode, (2) a copper anode, and (3) a $Na_2SO_4$ water electrolyte solution
(Pt*, Cu*, Na*S*O*)
**Container Materials:** unknown

**Experiment/Material Applications:**
Available English publications did not detail (1) the reasons why these materials were chosen for the experiment or (2) the specific research applications of the experiment.

**References/Applicable Publications:**

(1) Wospernov, W. and Raub, C. J.: Electrolytical Hydrogen Generation Under Zero Gravity. Hydrogen Bubble Experiment in the TEXUS 1 Project. BMFT-Fb-W-79-31, 36 pp. (in German; post-flight)

(2) Wospernov, W. and Raub, C. J.: Electrolytic Hydrogen Evolution in the Absence of Gravity. Zeitschrift fuer Flugwissenschaften und Weltraumforschung, Vol. 2, September-October 1978, pp. 341-345. (in German; presumably discusses TEXUS 1 results)

(3) Electrolytic Hydrogen Formation. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 84-85. (post-flight)


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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 6
**Launch Date/Expt Date:** May 1982
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-7 (experiment cell containing (1) a half gold-plated Pt cathode, (2) a copper anode, and (3) a $Na_2SO_4$ water solution).
**Builder of Processing Facility:** Unknown

**Experiment:**
Cathodic Hydrogen Formation/Bubble Electrolysis

This TEXUS 6 experiment was the second in a series of investigations designed by Raub to study low-gravity hydrogen bubble generation on a cathode surface (see Raub, TEXUS 1). The specific objectives of the investigation were to observe (1) the nucleation, growth, and coalescence of bubbles on a cathode and (2) the passivation of the electrode (the condition in which the reaction is impeded by an adsorbed surface film on the electrode).

The investigation was performed in an experimental cell housed within the TEXUS Experiment Module TEM 06-7. The cell contained (1) a half gold-plated Pt cathode, (2) a copper anode, and (3) a $Na_2SO_4$ water solution. During the mission, the hydrogen generation on the cathode was observed with a cine camera.

It was reported that bubble formation was observed on both the Au and Pt portions of the electrode. Growth of the bubbles was largest during the initial stages of the experiment because of supersaturation during and just after nucleation. Bubble size was attributed to cathode material. <Note: This dependence of bubble size on cathode material was not further discussed.> After a bubble separated from the electrode, its bubble size decreased due to dissolution of hydrogen in the electrolyte. Neither a potential drop across the cell nor passivation of the electrode was observed indicating that the electrolytic process under low-gravity conditions may be viable.

**Key Words:** Physical Chemistry, Electrodeposition, Thin Films, Coated Surfaces, Electric Field, Conduction, Cathode, Anode, Electrolysis, Electrodes, Transparent Liquids, Aqueous Solutions, Electrolyte Solution, Hydrogen Formation, Bubbles, Bubble Formation, Bubble Growth, Bubble Nucleation, Bubble Coalescence, Dissolution, Supersaturation, Solid/Liquid Interface, Buoyancy Effects, Contained Fluids

**Number of Samples:** one
**Sample Materials:** (1) a half gold-plated Pt cathode, (2) a copper anode, and (3) a $Na_2SO_4$-water electrolyte solution
(Pt*, Cu*, Na*S*O*)
**Container Materials:** unknown

**Experiment/Material Applications:**
See Raub, TEXUS 1.

**References/Applicable Publications:**

(1) Wospersnov, W. and Raub, C. J.: Electrolytic Hydrogen Evolution in the Absence of Gravity. In Zeitschrift fuer Flugwissenschaften und Weltraumforschung, Vol. 2, September-October 1978, pp. 341-345. (in German; presumably discusses TEXUS 1 experiment)

(2) Electrolytically Generated Hydrogen Bubbles. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 86. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 2
**Launch Date/Expt. Date:** November 1978
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-2
**Builder of Processing Facility:** TEM 06-2: ERNO, Bremen, Germany
<Note: The Principal Investigator reported that the "Master Form" was built by Institut für Werkstofftechnologie der Fachhochschule Kiel (F. H. Kiel), Kiel, Germany. However, it was not clear (to the editors of this document) to what "Master Form" refers.>

**Experiment:**
Electrochemical Corrosion

The corrosion of a material within an electrolytic solution is influenced significantly by the transport of substances through the electrolyte. Because this transportation is influenced by the amount of convection within the electrolyte, the measurement of the electrochemical process is also effected. Under low-gravity conditions, it was expected that convection would be reduced and the effect on the corrosion of the electrodes could be discerned.

This TEXUS 2 experiment was designed to investigate the influence of convection on electrochemical corrosion. The specific objectives of the experiment were to study

(1) the influence of gravity on the marginal current and the resulting effects on (a) the thickness of the adherent boundary layer and (b) the convection behavior.

(2) "...comparative experiments with viscosity-increasing additives to the electrolyte.

(3) "...the fundamental usefulness of reference electrodes for 0-g experiments. (1, p. 133)

The experiment was performed in the TEXUS Experiment Module TEM 06-2. The module was configured with two electrolytic cells (150 ml each). Each cell contained (1) two electrolytic copper electrodes (12.82 $cm^2$, corroded for several days in a KCl solution) and (2) 1-m KCl solution (5.6 mg $O_2$/liter) exposed to oxygen saturated air. Tylose was added to cell 2 to increase the viscosity of the electrolyte. Reference voltage was measured by an Ag/AgCl electrode. (The maximum current that could be measured with this system was 500 microamperes.)

Fifteen days prior to launch, the cells were maintained while the electrodes were intermittently polarized with 100 mV. During the 20 seconds just prior to launch, the cells were polarized, providing the "1-g comparison data." A total of six polarization cycles were then applied during the launch, low-gravity, and re-entry periods of the flight. During these cycles, the current and time were recorded.

Post-flight analysis of the collected data (see Reference (1) for complete details) revealed that the lengthy pre-launch, 15-day stand-time of the experiment reduced the low-gravity effects. The copper was "...oxidized by the oxygen given off in the electrolyte especially in the presence of chloride ions. Since the electrolytic vessel was tightly sealed, the consumed oxygen was not replaced. The decrease of oxygen content during the standing time was determined by means of comparative experiments.... According to this, the electrolyte at the time of the experiment contained only residual oxygen." (1, p. 143).

It was reported, however, that the TEXUS experiment illustrated the effects of the low-gravity conditions. The marginal current measured under low-gravity conditions was 40% less than that of the "1-g comparison data" and comparative ground experiments. This difference was attributed to the wider adherent boundary layer formed in the absence of convection.

The tylose added to cell 2 "...[suppressed] convection to such an extent that in cell 2 during the 0-g phase the existence of convection can practically be ruled out." (1, p. 144) (It was reported that the tylose additive also suppressed convection in 1-g experiments.) Reportedly, direct comparison between the behavior of cell 1 and cell 2 was not possible. The 15-day pre-launch stand time resulted in differences between the electrodes and electrolytes of the two cells. Post-flight examination of the two electrodes in cell 1 revealed a smooth, evenly browned surface. Those of cell 2 were nearly copper colored and spotty. These differing surface conditions indicated that the electrolytes also changed to different extents. Therefore, "...in cell 1 lesser currents were measured, including during convection, than in cell 2." (1, p. 145)

The Ag/AgCl reference electrode performed well. "It [was] simple, robust and insensitive. A change in the reference potential by the particular marginal conditions of the 0-g experiment were not observed." (1, p. 145)

**Key Words:** Physical Chemistry, Electrochemical Corrosion, Corrosion, Thin Films, Coated Surfaces, Electric Field, Conduction, Electrodes, Electrolyte Solution, Solid/Liquid Interface, Oxide Layer, Oxidation, Buoyancy-Driven Convection, Buoyancy Effects Diminished, Mass Transfer, Viscosity, Polarization, Contained Fluids, Deterioration of Samples Prior to Launch

**Number of Samples:** two electrolytic cells
**Sample Materials:** Electrodes: electrolytic copper corroded for several days in a potassium chloride solution; Cell 1 Electrolyte: 1-m KCl solution (5.6 mg $O_2$/l) exposed to air which was saturated with oxygen, $O_2$; Cell 2 Electrolyte: Same as Cell 1 with a Tylose additive. Reference electrode: silver/silver chloride.
(Cu*, K*Cl*, O*, Ag*, Ag*Cl*)
**Container Materials:** Plexiglas$^{TM}$

**Experiment/Material Applications:**
The measurement of electrochemical effects is significantly influenced by the degree of convection within the electrolyte. Under low-gravity conditions, the effects of convection can either be eliminated or reduced, resulting in more accurate studies of the electrochemical corrosion process.

The specific reasons why the electrode/electrolyte materials were selected were not detailed in the available publications.

**References/Applicable Publications:**
(1) Mix, G.: Intermittent Polarization. In Shuttle/Spacelab Utilization Final Report Project TEXUS II, 1978, pp. 132-145. (post-flight)

(2) Intermittent Polarization. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 88-89. (post-flight)

(3) Input received from Principal Investigator G. Mix, July 1993.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3
**Launch Date/Expt. Date:** April 1980
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Nucleate Pool Boiling

This TEXUS 3 experiment was the first in a series of investigations designed by Straub et al. to study nucleate pool boiling under low-gravity conditions.

No publications detailing the experimental objectives or equipment setup could be located.

Reference (2) reported that due to a rocket despin failure, TEXUS 3 did not achieve the desired low-gravity level.

Further information concerning this TEXUS 3 investigation does not appear to be available. The experiment was reflown on TEXUS 3b (see Straub, TEXUS 3b).

**Key Words:** Physical Chemistry, Nucleate Pool Boiling, Boiling, Heat and Mass Transfer, Bubbles, Bubble Formation, Bubble Growth, Liquid/Gas Interface, Rocket Motion, Acceleration Effects, Rocket Despin Failure

**Number of Samples:** one
**Sample Materials:** Freon 113
**Container Materials:** experiment cell: unknown

**Experiment/Material Applications:**
See Straub, TEXUS 3b.

**References/Applicable Publications:**

(1) Weinzierl, A., and Straub, J.: Nucleate Pool Boiling in Microgravity Environment. In Heat Transfer 1982; Proc. of the 7th International Conference, Munich, West Germany, September 6-10, 1982, pp. 21-27. (appears to be TEXUS 3b; mission is unspecified; post-flight)

(2) Greger, G.: TEXUS and MIKROBA and Their Effectiveness and Experiment Results. Presented at: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center, (JSUP). (identifies rocket failure)

(3) Input received from B. Vogel (Technische Universität, Munich), August 1993.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3b
**Launch Date/Expt Date:** April 1981
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Bubble Formation: Nucleate Pool Boiling

The boiling process and its associated heat transfer are extremely important in industry (e.g., spray cooling of hot metal ingots). However, despite extensive investigations of the phenomena, the process is not fully understood. Mathematical equations which model the process provide controversial results, especially in terms of gravitational influences. When the contribution of gravity is significant, two heat transfer mechanisms become active: (1) free convection and (2) enthalpy transfer by buoyancy-induced detachment and flotation of bubbles.

In the low-gravity environment, gravity-related mechanisms are decoupled from the boiling process and investigations can be conducted concerning the three active heat and mass transfer mechanisms: (1) evaporation/condensation, (2) interface convection, and (3) heat conduction.

This TEXUS 3b experiment was the second in a series of investigations designed by Straub et al. to study nucleate pool boiling under low-gravity conditions (see Straub, TEXUS 3). The specific objectives of the investigation were to (1) determine the relative contributions of the two gravity dependent, active heat transfer mechanisms and (2) investigate (a) the behavior of the bubbles during boiling and (b) the related heat transfer.

The TEXUS Experiment Module TEM 06-3 was employed for the investigation. The module contained a 20-mm diameter cylindrical cell filled with Freon 113. A platinum wire (0.2 mm diameter, 25 mm long) spanned the cell's diameter. During heating and boiling of the Freon, (1) a bellows system associated with the cell allowed for volume expansion and (2) a cine camera recorded the nucleation and growth of bubbles.

During the rocket flight, three constant voltages were applied to the wire which corresponded to three heat fluxes: (1) 1.7 $W/cm^2$, (2) 4.0 $W/cm^2$, and (3) 7.7 $W/cm^2$. Wire temperatures were determined from the electric currents. Two thermocouples immersed in the liquid near the Pt wire measured the liquid temperature. Pressure measurements were also recorded.

Post-flight examination of the recorded flight data indicated that the temperature at the onset of nucleate boiling was not influenced by gravity for the three heating rates. Steady nucleate boiling was established within 0.5 seconds and was accompanied by a pressure increase of approximately 5000 Pa. The lifetime of the bubbles was a few tenths of a second as they moved along the wire. Mass transport by evaporation/condensation was also observed.

Reportedly, (1) the heat transfer coefficient appeared to be approximately equal to or slightly larger than seen on Earth and (2) "These results proved the dominating role of Marangoni convection in the boiling heat transport in microgravity." (4, p. 98) <Note: The bubble behavior at each of the three heat fluxes was not detailed.>

No further information (published in English) concerning this experiment could be located at this time. <Note: References (6)-(9), which were not available to aid in the preparation of this experiment summary, may contain more information about this investigation.>

**Key Words:** Physical Chemistry, Transparent Liquids, Phase Transition, Nucleate Pool Boiling, Boiling, Heat Transfer, Heat and Mass Transfer, Bubbles, Bubble Formation, Bubble Growth, Bubble Nucleation, Flotation of Bubbles, Evaporation, Condensation, Conduction, Convection at the Interface, Marangoni Convection, Thermocapillary Convection, Surface Tension-Driven Convection, Solid/Liquid Interface, Liquid/Gas Interface, Electric Field, Volume Compensation, Volume Expansion, Buoyancy Effects, Contained Fluids

**Number of Samples:** one (three different heating rates)
**Sample Materials:** Freon 113
**Container Materials:** experiment cell: unknown; heater surface material: platinum wire

**Experiment/Material Applications:**
Compared to natural convection or film boiling, nucleate pool boiling results in high heat transfer rates. The pool boiling process is very complicated with several mechanisms (gravity dependent and gravity independent) contributing to heat transportation. The study of such boiling under low-gravity conditions (1) allows closer study of gravity-dependent effects and (2) provides a distinction between the relative contributions of the gravity-dependent and gravity-independent mechanisms. Understanding these mechanisms will lead to a greater knowledge of heat transfer on Earth and under low-gravity conditions.

**References/Applicable Publications:**

(1) Weinzierl, A. and Straub, J.: Heat Transfer with Bubble Formation-Results of the TEXUS 3b Experiment. Zeitschrift für Flugwissenschaften und Weltraumforschung, Vol. 6, September 1982, pp. 339-343. (in German; post-flight)

(2) Weinzierl, A. and Straub, J.: Nucleate Pool Boiling in Microgravity Environment. In Heat Transfer 1982, Proc. of the 7th International Conference, Munich, West Germany, September 6-10, 1982, pp. 21-27. (TEXUS 3b; post-flight)

(3) Zell, M. and Straub, J.: Microgravity Pool Boiling- TEXUS and Parabolic Flight Experiments. In ESA 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 155-160. (does not specify TEXUS flight number; post-flight)

(4) Nucleate Pool Boiling. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 98-99. (post-flight)

(5) Input received from B. Vogel (Technische Universität, Munich), August 1993.

(6) Straub, J. and Weinzierl, A.: Untersuchung der Blasenbildung bei Phasenänderung unter Schwerelosigkeit-Werkstofforschung und Verfahrenstechnik im Weltraum; BMFT-Status Seminar "Spacelab-Nutzung," Meersburg, Germany, 1977, DGLR-Bericht 78/02, Cologne, DGLR. (in German)

(7) Straub, J. and Weinzierl, A.: Optische Voruntersuchungen sum Wärmeübergang bei Siedenvorgängen unter reduzierter Schwerkraft-Werkstofforschung und Verfahrenstechnik im Weltraum; BMFT-Status Seminar "Spacelab-Nutzung," Meersburg, Germany, 1978, DGLR-Bericht 79/01, Cologne, DGLR, pp. 177-184. (in German)

(8) Straub, J. and Weinzierl, A.: Untersuchung der Blasenbildung bei Phasenänderung unter Schwerelosigkeit-Werkstofforschung und Verfahrenstechnik im Weltraum; BMFT-Status Seminar "Spacelab-Nutzung," Tübingen, Germany, 1978, DGLR-Bericht 79/01, Cologne, DGLR, pp. 33-43. (in German)

(9) Blasenbildung und Wärmeübergang unter 1-g-und Micro-g-Bedingungen-Werkstofforschung und Verfahrenstechnik im Weltraum; BMFT-Status Seminar "Spacelab-Nutzung", Würtzburg, Germany, 1980, DGLR-Bericht 80/02, Cologne, DGLR, pp. 95-101. (in German)

(10) Weinzierl, A. and Straub, J.: Wärmeübergang und Blasenbildung- Ergebnisse des TEXUS III-b Experimetns; Z. f. Flugwissenschaft und Weltraumforschung, Bd. 6, pp. 339-343, 1982. (in German)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 5
**Launch Date/Expt Date:** April 1982
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Bubble Formation

This TEXUS 5 experiment was the third in a series of investigations designed by Straub et al. to study nucleate pool boiling under low-gravity conditions (see Straub, TEXUS 3, TEXUS 3b). The specific objective of the experiment was to investigate "...several heat fluxes into a subcooled liquid in order to observe the onset of boiling, and condensation effects at the bubble surface." (3, p. 100)

The TEXUS Experiment Module TEM 06-3 was employed for the investigation. The cylindrical test cell and associated hardware were the same as those used during the TEXUS 3b investigation (see Straub, TEXUS 3b for a complete description).

During the low-gravity phase of the TEXUS 5 mission, nine different heat fluxes from a platinum wire into subcooled Freon 113 (bulk temperature 25 $^{o}$C) were investigated. (The heat flux was increased incrementally up to 50 W/cm$^2$.) In order to achieve steady-state conditions, each power level was maintained for 13 seconds.

Post-flight, it was reported that "The onset of boiling started by the formation of a a 'vapour hose' around the wire... and at a superheating 25% higher than on Earth where steady state is reached long before, owing to natural convection." (3, p. 100) It was also observed that, after removal of the initially formed "bubble", a steady-state boiling occurred. <Note: It appears from photographs in Reference (3) that the "vapour hose" transformed into this "bubble."> Reportedly, this boiling was significantly stabilized by Marangoni convection at the surface of the bubbles. The heat transfer during steady-state boiling appeared to be independent of gravity level. <Note: It is not clear if this same vapour hose/bubble/boiling process occurred at each incremental heat flux step. The bubble behavior and condensation effects at each of the nine heat fluxes were not

detailed.>

No further information (published in English) concerning this experiment could be located at this time.

**Key Words:** Physical Chemistry, Transparent Liquids, Phase Transition, Nucleate Pool Boiling, Boiling, Heat Transfer, Heat and Mass Transfer, Heat Flux, Superheating, Vaporization, Bubbles, Bubble Formation, Bubble Growth, Condensation, Conduction, Convection at the Interface, Marangoni Convection, Thermocapillary Convection, Surface Tension-Driven Convection, Solid/Liquid Interface, Liquid/Gas Interface, Electric Field, Volume Compensation, Volume Expansion, Buoyancy Effects, Contained Fluids

**Number of Samples:** one (nine different heating rates)
**Sample Materials:** test fluid: Freon 113 - triclorotrifluroethane
**Container Materials:** experiment cell: unknown; heater surface material: platinum wire
(Pt*)

**Experiment/Material Applications:**
See Straub, TEXUS 3b.

**References/Applicable Publications:**

(1) Zell, M., Straub, J., and Weinzierl, A.: Nucleate Pool Boiling in Subcooled Liquid Under Microgravity-Results of Texus Experimental Investigations. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 327-333. (post-flight; does not specify TEXUS flight number)

(2) Zell, M. and Straub, J.: Microgravity Pool Boiling-TEXUS and Parabolic Flight Experiments. In ESA 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 155-160. (post-flight; does not specify TEXUS flight number)

(3) Bubble Formation. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 100-101. (post-flight)

(4) Input received from B. Vogel (Technische Universität, Munich), August 1993.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 8
**Launch Date/Expt Date:** May 1983
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Bubble Formation

This TEXUS 8 experiment was the fourth in a series of investigations designed by Straub et al. to study nucleate pool boiling under low-gravity conditions (see Straub, TEXUS 3, TEXUS 3b, TEXUS 5).

No documents (published in English), describing the experimental objectives, setup, or results from this investigation could be located.

**Key Words:** Physical Chemistry, Phase Transition, Nucleate Pool Boiling, Boiling, Heat and Mass Transfer, Bubbles, Bubble Formation, Bubble Growth, Liquid/Gas Interface

**Number of Samples:** one
**Sample Materials:** Freon 113
**Container Materials:** experiment cell: unknown

**Experiment/Material Applications:**
See Straub, TEXUS 3b.

**References/Applicable Publications:**

(1) Weinzierl, A. and Straub, J.: Nucleate Pool Boiling in Microgravity Environment. In Heat Transfer 1982; Proc. of the 7th International Heat Transfer Conference, Munich, West Germany, September 6-10, 1982, pp. 21-27. (specifies no mission number; preflight)

(2) Zell, M. and Straub, J.: Microgravity Pool Boiling-TEXUS and Parabolic Flight Experiments. In ESA 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 155-160. (specifies no mission number; post-flight)

(3) Experimentelle Untersuchung des Wärmeübergangs und seiner Transportmechanismen bei Siedevorgängen unter reduzuierter Schwerkraft; Jahrbuch der Technischen Universität München, 1985, pp. 163-178.

(4) Input received from B. Vogel (Technische Universität, Munich), August 1993.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 10
**Launch Date/Expt Date:** May 1984
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Bubble Formation

This TEXUS 10 experiment was the fifth in a series of investigations designed by Straub et al. to study nucleate pool boiling under low-gravity conditions (see Straub, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 8).

Previous TEXUS sounding rocket experiments in this investigative series indicated that steady-state, boiling heat transfer from a heated platinum wire was not significantly different than that on Earth. Therefore, the objective of this experiment was to determine whether low-gravity boiling heat transfer was influenced by the geometry of the heater. This was accomplished by replacing the heated wire configuration (used during the earlier missions) with a heated flat plate. The following phenomena were to be investigated: (1) bubble nucleation, (2) bubble dynamics, (3) phase change, and (4) Marangoni transport with respect to the stability of boiling heat transfer.

As in the earlier low-gravity experiments, the TEXUS Experiment Module TEM 06-3 was employed for the investigation. However, certain changes were incorporated to accommodate the flat plate geometry. It appears that the TEM 06-3 module still retained (1) a cylindrical cell filled with Freon 113 (and the associated bellows system to allow for volume expansion) and (2) a cine camera to record nucleation and growth of bubbles.

The flat plate (20 mm by 40 mm) contained within the test cell consisted of a thin gold film on a cerodur glass substrate. The plate was used simultaneously as a heater and resistance thermometer (range of 20 to 200 $^{o}$C with +/- 1 K accuracy). The plate could produce eight discrete heat flux levels up to 8 W/cm$^2$. Five thermocouples were located in the working fluid (Freon 113) and four thermocouples were located in the glass substrate for calculation of the deficit heat flux.

During the low-gravity phase of the rocket, the plate was heated at different rates, and the resulting behavior of the fluid was recorded.

Post-flight examination of the recorded data indicated that, under low-gravity conditions, "...the onset of boiling in the subcooled liquid (Tsat - Tbulk = 25 $^{o}$C) occurred at a superheating higher than on Earth but the activation of the first nucleation site required only 45% of the heat flux. Also the spreading of nucleate boiling was 20 times faster than on Earth (<0.1 s...). However, the duration of the thermal transition to steady state increased up to several seconds, due to the long time required for the condensation of the initial 'vapor explosion'. When the heat input to the plate was increased above 5 W/cm$^2$, the limits of the heat transport mechanisms in microgravity (dissipation of the condensation enthalpy from the top of the bubbles towards the subcooled bulk by thermocapillary flows and bubble dynamics) were exceeded and a film boiling regime was set up.... The Experiment ended with the burnout of the heater, as it was isolated by a layer of vapour." (3, p. 102) <Note: It is not clear how many heating rates were examined prior to heater burnout. Further, the boiling heat transfer characteristics were not detailed at each of the heating rates.>

No further information concerning this experiment could be located at this time.

**Key Words:** Physical Chemistry, Transparent Liquids, Phase Transition, Nucleate Pool Boiling, Boiling, Heat Transfer, Heat and Mass Transfer, Heat Flux, Superheating, Vaporization, Contained Fluids, Bubbles, Bubble Formation, Bubble Growth, Bubble Nucleation, Bubble Dynamics, Condensation, Convection at the Interface, Marangoni Convection, Thermocapillary Convection, Marangoni Movement (Migration) of Bubbles, Surface Tension-Driven Convection, Solid/Liquid Interface, Liquid/Gas Interface, Substrates, Thin Films, Coated Surfaces, Volume Compensation, Volume Expansion, Buoyancy Effects, Heat Exchangers

**Number of Samples:** one (eight different heating rates were possible)
**Sample Materials:** Freon 113 - triclorotrifluroethane.
**Container Materials:** experiment cell: unknown; heater material: thin gold film on cerodur glass substrate.

**Experiment/Material Applications:**
See Straub, TEXUS 3b.

**References/Applicable Publications:**

(1) Zell, M., Straub, J., and Weinzierl, A.: Nucleate Pool Boiling in Subcooled Liquid Under Microgravity-Results of Texus Experimental Investigations. In Proceedings of the 5th European Symposium on Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, p. 327. (post-flight)

(2) Zell, M. and Straub, J.: Microgravity Pool Boiling- TEXUS and Parabolic Flight Experiments. In ESA 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 155-160. (does not specify TEXUS flight number; post-flight)

(3) Bubble Formation II. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 102-103. (post-flight)

(4) Input received from B. Vogel (Technische Universität, Munich), August 1993.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 11
**Launch Date/Expt Date:** April 1985
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Bubble Formation

This TEXUS 11 experiment was the sixth in a series of investigations designed by Straub et al. to study nucleate pool boiling under low-gravity conditions (see Straub, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 8, TEXUS 10). The specific objectives of the experiment were to investigate the boiling process (1) in an saturated liquid, (2) during the transition from a saturated to subcooled liquid, and (3) in a subcooled liquid.

The TEXUS 11 experiment was performed in the TEM 06-3 Experimental Module. Reportedly, the experimental hardware was the same as that used during Straub's earlier TEXUS 10 investigation. The module contained (1) a cylindrical cell filled with Freon 113 (and configured with a 20-mm by 40-mm flat heater plate) and (2) a cine camera to record nucleation and growth of bubbles.

During the TEXUS 11 mission, three distinct experimental steps (corresponding to the three experimental objectives (mentioned above)) were performed. <Note: A detailed discussion of each of these steps and the subsequent results were not provided in the publications that were available to the editors at the time this summary was prepared (References (1-3)). The summary below represents nearly the entire available information related to the experimental steps, subsequent analysis and reported results.> The low-g data from each of the steps were compared to data obtained during terrestrial reference experiments performed in the TEM 06-3 apparatus.

Step 1: Study of the Boiling Process in a Saturated Liquid

It appears that this part of the TEXUS 11 experiment was initiated 75 seconds after the rocket was launched (t = 75 seconds) and continued until t = 230 seconds. During this time, the heater power was increased and subsequently decreased resulting in a heat flux, q, range of 3-7 W/cm$^2$. <Note: It is not clear what the baseline pressure or temperature of the liquid was when the experiment was initiated (based on the references available at the time this experiment summary was prepared). Reference (4) indicated that this information is available, but the reference did not identify in which publication(s) this information could be found.>

Reportedly, the onset and evolution of boiling in the saturated liquid were observed. Based on these observations it was concluded that:

"- at low system pressure and in the absence of gravity, the energy exchange between the heating surface and the fluid deteriorates shortly after boiling starts because there is no enthalpy transport by buoyancy driven detachment and flotation of vapour bubbles. This results in film boiling;
- the detachment of the bubbles in not due to impulse forces but exclusively to buoyancy forces (i.e. occurs only on earth)." (3, p. 104)

Step 2: Study of the Boiling Process During the Transition from Saturated Boiling to Subcooled Boiling

This part of the experiment was initiated at t = 230 seconds and continued until t = 300 seconds. During this time, a transition from saturated boiling to subcooled boiling was achieved by increasing the pressure in the cell at a constant heating power (q = 3 W/cm$^2$. <Note: It is not clear what the baseline pressure or temperature of the liquid was when the experiment was initiated (based on the references available at the time this experiment summary was prepared). (See note concerning Reference (4) above.)>

Reportedly, "...the increased pressure from the saturation line to a subcooling of... [Tsat - Tbulk] = 25 K, causes a sudden condensation of the vapor front...." (1, p. 33, translation) "After about 2 s, the heat transfer became stationary with a heat transfer coefficient identical to the one measured on Earth. (3, p. 104)

Step 3: Study of the Boiling Process in a Subcooled Liquid

This part of the experiment was initiated at t = 300 seconds and continued until t = 500 seconds. <Note: It is not clear what the baseline pressure and temperature of the liquid was when the experiment was initiated although at one point during this step

Tsat-Tbulk reportedly equaled 48 K. (See note concerning Reference (4) above.)> During this time, the heater power was gradually increased and subsequently decreased (heat flux q = 3-7 $W/cm^2$, similar to Step 1).

Reportedly, "Boiling started with an explosive formation of bubbles accompanied by a decrease of the heater temperature.... After a stabilization time of 2.5 s, which is significantly longer than on Earth, the temperature field stayed stationary and the heat transfer coefficient was again identical to the one measured under 1-g. In microgravity, the absence of buoyancy convection was compensated by an augmented... [vaporization]/ condensation process resulting from a bigger bubble surface area (diameter 10 to 20 times larger than on Earth.)" (3, p. 104)

It was concluded that

"-Adhesion forces at the heating surface dominate the impulse forces for bubble generation and growth" (1, p. 35, translation)

"-during subcooled boiling in microgravity, the condensation at the top of the bubbles guarantees their stability [stationary boiling] but with a reduced critical heat flux" (3, p. 105)

"-The influence of the Marangoni convection on the heat transfer appears to be negligible for the used heater geometry (smooth heating surface)" (1, p. 35, translation)

"-the current boiling process models which are generally based on gravity dependent mechanisms, should be reconsidered specially regarding the real influences of gravity." (3, p. 106)

Additional information concerning this experiment may be contained in References (5-11).

**Key Words:** Physical Chemistry, Transparent Liquids, Phase Transition, Nucleate Pool Boiling, Boiling, Heat Transfer, Heat and Mass Transfer, Heat Flux, Vaporization, Bubbles, Bubble Formation, Bubble Growth, Bubble Nucleation, Saturated Solution, Evaporation, Condensation, Marangoni Convection, Thermocapillary Convection, Surface Tension-Driven Convection, Solid/Liquid Interface, Liquid/Gas Interface, Volume Compensation, Volume Expansion, Buoyancy Effects, Buoyancy Forces, Coated Surfaces, Surface Roughness, Thin Films, Heat Exchangers, Contained Fluids

**Number of Samples:** one (three different heating/cooling steps)
**Sample Materials:** test fluid: Freon
**Container Materials:** experiment cell: unknown; heater material: vapor-coated gold on glass surface

**Experiment/Material Applications:**
See Straub, TEXUS 3b.

**References/Applicable Publications:**

(1) Straub, J. and Zell, M.: Bubble Formation II. In TEXUS 11/12 Abschlussbericht 1985, German Publication, 1985. (in German)

(2) Zell, M. and Straub, J.: Microgravity Pool Boiling-TEXUS and Parabolic Flight Experiments. In ESA 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 155-160. (does not specify TEXUS flight number; post-flight)

(3) Bubble Formation. Microgravity Measurements of the Boiling Heat Transfer from Flat Heated Surfaces. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 104-106. (post-flight)

(4) Input received from B. Vogel (Technische Universität, Munich), August 1993.

<Note: Although B. Vogel (Reference (4)) provided an extensive list of applicable publications related to "Boiling Under Microgravity" (several of which had been previously identified and already contained within the data base), it was not possible to obtain several documents (and translate those in German) prior to the publication of this Technical Memorandum. References (5-11) are applicable publications from Vogel's list that could not be obtained (and that were not previously listed under other TEXUS experiments).>

(5) Weinzierl, A., Zell, M., and Straub, J.: Influence of Acceleration on Subcooled and Saturated Pool Boiling-Results of Parabolic Flight Investigations. ESA Microgravity Division, EWP-1457, 1986, pp. 3-14.

(6) Weinzierl, A., Zell, M., and Straub, J.: Spacelabnutzung- Untersuchung des Wärmeübergangs und seiner Transportmechanismen bei Siedevorgängen unter Schwerelosigkeit; BMFT-FB-W 86-021, 1986, 148 pp. (in German)

(7) Zell, M., Straub, J., and Vogel, B.: Pool Boiling Under Microgravity. J. of Physico-Chemical Hydrodynamics, 11, 5/6, 1989, pp. 813-823.

(8) Straub, J., Weinzierl, A., and Zell, M.: Thermokapillare Grenzflächenkonvektion an Gasblasen in einem Temperaturgradientenfeld; Wärme- und Stoffübertragung, Bd., 25, 1990, pp. 281-288.

(9) Straub, J., Zell, M., and Vogel, B.: Der Einfluss der Schwerkraft auf den Wärmeübergang beim Sieden; Chem. Ing. Tech., Bd. 62, 1990, p. 128.

(10) Straub, J., Zell, M., and Vogel, B.: Pool Boiling in a Reduced Gravity Field. Proc. 9th International Heat Transfer Conference, Jerusalem, Israel, August 19-24, 1990, Hetsrony, G. (ed.), New York: Hemisphere, Vol. 1, 1990, pp. 91-112.

(11) Straub, J., Zell, M., and Vogel, B.: Boiling Under Microgravity Conditions. Proc. First European Symposium Fluids in Space, Ajaccio, France, November 18-22, 1991, ESA SP-353, pp. 269-297.

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 13
**Launch Date/Expt Date:** April 1986
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-6: experiment cell filled with Freon and configured with a surface (spot) heater (temperature recording at 10 locations). Bubble formation and resultant density and temperature gradients made visible by a differential interferometer.
**Builder of Processing Facility:** ERNO, Bremen, Germany

**Experiment:**
Single Bubble

This TEXUS 13 experiment was the seventh in a series of investigations designed by Straub et al. to study nucleate pool boiling under low-gravity conditions (see Straub, TEXUS 3, TEXUS 3b, TEXUS 5, TEXUS 8, TEXUS 10, TEXUS 11). The overall objective of the experiment was to investigate the thermodynamic and fluid dynamic processes involved in the formation of boiling bubbles on a heated surface. More specifically, the objective was to obtain optical and thermal data associated with an individual vapor bubble and analyze in detail the gravity-independent mechanisms affecting the bubble. (Details concerning thermodynamic and fluid dynamic behavior along the bubble surface may be important to heat transfer phenomena.)

The experiment was performed in the TEM 06-6 Experimental Module. The module contained a test cell filled with Freon R113. A spot heater (comprised of gold thin film on a glass substrate) was configured within the cell. "The experimental setup was designed to allow observation of single vapour bubbles under different conditions in the liquid (saturated, subcooled)." (2, p. 108) Temperature in the liquid could be recorded at ten different locations and a bellows system allowed pressure adjustment of the test liquid during heating.

During the mission, bubbles of Freon R113, generated on the spot heater, grew under controlled thermal gradients. A differential interferometer was employed to determine the density and temperature fields around the bubble (without contact and thermal inertia). The interferometer images were recorded with a motion picture camera. Also recorded were (1) the spot heater temperature, (2) the heat flux, (3) the fluid temperature, and (4) the cell pressure. <Note: Additional details concerning the pressures recorded during the experiment were not detailed.>

Reportedly, "Technical difficulties during the performance of the experiment under microgravitation severely restricted the ability to analyze the information obtained from the interferometer. By means of uncontrolled bubble formation and deficient optical recording quality, it was impossible to obtain the goal of the experiment, in other words, a detailed analysis of boundary surface phenomena, so that heat transfer measurements [could] be derived only from the electrical data." (1, p. 38, translation)

"A quantitative analysis of the optical record was only possible during two short sequences where a single bubble could be generated in microgravity. It could be seen clearly... that with increasing subcooling, the convection at the bubble interface became the dominating and thermally stabilising(sic) liquid transport mechanism. The phase change mechanisms involved in the boiling process (evaporation/condensation) can reach a steady state equilibrium only if the energy released at the top of the bubble condensation is evacuated by hydrodynamical mechanisms. From a thermodynamic point of view, the temperature differences in pure liquids and thus the surface tension gradients along the bubble surface are negligible. Practically, the driving force of the interfacial convection resides in heat transfer resistances at the bubble surface which originate in impurities enrichment (e.g. traces of components with higher boiling points or dissolved gases). With increasing subcooling and thus smaller bubbles, this generated strong gravity independent convection flows perpendicular to the heating surface. The heat transfer was then comprable[sic] with the one measured on Earth.

"With a low subcooling (...[delta]T = 22$^{o}$C) in microgravity, the bubbles grew fast and the intensity of the interfacial convection varied periodically (f...[approximately equal to] 2 Hz). This is reflected by the temperature/time profile of the heating surface... which is inversely proportional to the heat transfer coefficient. The minima which occurred periodically correspond to the ceasing of the interfacial convection.

"The observation of the dynamical behaviour of the surface of vapour bubbles during boiling requires improved experimentation. The analysis of the resulting data should allow the various numerical models of bubbles during boiling... to be tested." (2, p. 108)

No further information concerning the results from this experiment could be located in the available publications. It is suspected that such data may not be available due to difficulties with the optical record.

**Key Words:** Physical Chemistry, Transparent Liquids, Phase Transition, Nucleate Pool Boiling, Boiling, Heat Transfer, Heat Transfer Coefficient, Heat and Mass Transfer, Heat Flux, Thermal Gradient, Vaporization, Bubbles, Bubble Formation, Bubble Growth, Bubble Nucleation, Bubble Dynamics, Saturated Solution, Evaporation, Condensation, Marangoni Convection, Thermocapillary Convection, Surface Tension-Driven Convection, Solid/Liquid Interface, Liquid/Gas Interface, Convection at the Interface, Impurities, Volume Compensation, Volume Expansion, Buoyancy Effects, Buoyancy Forces, Substrates, Coated Surfaces, Thin Films, Interferometric Measurement, Contained Fluids, Heat Exchangers, Processing Difficulties, Photographic Difficulties

**Number of Samples:** one test setup (the number of single bubbles generated is unclear)
**Sample Materials:** test fluid: Freon R113
**Container Materials:** experiment cell: unknown; heater surface material: "...a two-layer glass plate upon which gold [had] been steamed." (1, p. 36, translation)

**Experiment/Material Applications:**
See Straub, TEXUS 3b.

**References/Applicable Publications:**

(1) Straub, J. and Zell, M.: Einzelblase. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 36-40. (post-flight; in German)

(2) Single Bubble. In Summary of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 108-109. (post-flight)

(3) Input received from B. Vogel (Technische Universität, Munich), August 1993.

<Note: See the "References/Applicable Publications" section under Straub, TEXUS 11 for additional publications which may be applicable to this experiment.>

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**Co-Investigator(s):** Kayser-Threde GmbH/Klett, R. (Contributor/Customer) (2)
**Affiliation(s):** (1) When experiment was proposed: High School Student, During STS-007: Technical University of Munich, Federal Republic of Germany, Currently: Unknown, Possibly Technical University of Munich; (2) Munich, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #7, STS-007 (STS 31-C, Challenger)
**Launch Date/Expt. Date:** June 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** High School Student Experiment
NASA Get Away Special (GAS) Canister G-002
Volume of Canister: 5.0 cubic feet
Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of G-002:** German Youth Fair/Kayser-Threde, Munich, Germany
**Processing Facility:** Small isothermal furnace facility housing four specimen cartridges
**Builder of Processing Facility:** Kayser-Threde GmbH, Munich, Germany

**Experiment:**
<u>Nickel Catalyst</u>

"Nickel formiate is decomposed to elementary nickel, when it is heated to temperatures... [over] 206 $^{o}$C. This reaction is used to prepare a catalyst, able to perform hydrogenations of C=C double bonds and some other chemical species." (4, p. 359)

On Earth, the resultant surface of the nickel is a function of many parameters, including (1) the composition and purity of the solvent and (2) the diffusion and convection of the reaction partners. In space, the convection contribution should be reduced, possibly producing catalysts with improved properties.

This experiment was one of five investigations housed within the G-002 Get Away Special canister on STS-007. One other experiment (of the five) was applicable to this data base (see Pascherat, STS-007 (Chapter 8)).

The objective the research was to compare the physical and chemical properties of a nickel catalyst prepared (1) during the low-gravity shuttle mission and (2) during ground-based testing.

Prior to the shuttle launch, four nickel cartridges were configured within a single 55 W isothermal furnace. Reference (4) indicated that each cartridge contained "...potassium hydroxide and one of... two kinds of nickel formiate." (4, p. 359) The potassium hydroxide was used to (1) conserve the suspension, (2) absorb gases resulting from the decomposition, and (3) (thus) inhibit the formation of bubbles (which are disturbing to the transport of the reaction material). <Note: In contrast to Reference (4), Reference (2) indicated that in an effort to avoid moisture, each cartridge contained a mixture of (1) either potassium hydroxide/nickel formiate or sodium hydroxide/nickel formiate and (2) dry nitrogen. G. Schmitt (of Kayser-Threde) reported that Reference (4) (not Reference (2)) had correctly identified the chosen materials.>

References (2) and (4) also differ slightly on the reported processing temperatures. It appears that during the first hour of the shuttle experiment, all four cartridges were slowly heated from approximately 10 °C through the melting point (160 °C), and then heated to the decomposition point of nickel formiate (206 °C). (The inner samples appear to have been heated even more (215 °C) because of the available heat capacity of this section of the furnace.) After this initial hour of heating was completed, the furnace was turned off and the cartridges cooled passively. It appears that the samples reached a temperature of 160 °C in about 45 minutes. At this temperature, the samples resolidified, preserving the suspension which had occurred during the low-gravity experiment.

Analysis of the GAS payload indicated that the furnace performed as predicted and the experiment "worked perfectly." Space-processed samples were compared to Earth-processed samples using a number of methods including the measurement of (1) hydrogen peroxide decomposition and (2) stereoselective hydrogenation with hydrazine. These analyses indicated that an "abnormal thermal behavior" existed in the low-gravity processed catalyst. (Further details on this analysis can be found in Reference (4), pp. 360-363.) An X-ray scattering diagram of the finely divided form of the low-gravity catalyst illustrated three peaks. "Two of them belong to the well-known crystal structure of nickel, the third one is unknown." (4, p. 361) A similar diagram of the terrestrial sample illustrated only two main peaks.

Raster electronmicroscopic photos of the space and terrestrial catalysts indicated that, in general, the "...shape of the nickel catalysts... [was] plate like, with hexagonal structure. The plates or cubes... [were] built up in layers, like a geological sediment." (4, p. 363) From this visual inspection, significant differences between the space-processed and Earth-processed samples could not be found. However, when "...etching a probe of the catalyst with argon ions, 45 minutes, for example in a Balzers high vacuum ion etching machine, and monitoring afterwards, the fine divided form reveals a characteristic structure on its surface." (4, p. 363) Although it is unclear from the available reference (Reference (4)) it appears that the low-gravity catalyst exhibits a structure which is "...centered in the grains and follows disturbances of the margin. Lines in a concentric arrow are microscopically often frontiers of concentric terraces, piled up over each other. The center of the arrangement is a thick 'mountain'....

"The step height of the terrace is very small and it is possible, that these terraces are not real. They can be otherwise induced fluctuations in the back scattered secondary electrons, originated by regions of other structure." (4, p. 363) In contrast, the etched terrestrial sample did not exhibit a concentric structure.

Reference (2) (very briefly) reported that the "space processed catalysts experience higher efficiency". (2, p. 139) <Note: Efficiency was not defined in the reference.>

It was concluded that the microgravity-processed catalyst illustrated an abnormal thermal behavior and abnormal kinetic data.

**Key Words:** Physical Chemistry, Melt and Solidification, Catalysts, Suspension, Decomposition, Solvent, Bubble Formation, Buoyancy-Driven Convection, Diffusion, Isothermal Processing, Passive Cooling, Sample Purity, Crystalline Structure, Platelike Structure, Surface Morphology, Grain Structure, Composition Distribution

**Number of Samples:** four
**Sample Materials:** potassium hydroxide and one of two kinds of nickel formiate
**Container Materials:** nickel
(Ni*)

**Experiment/Material Applications:**
Low-gravity manufacturing of nickel catalysts was expected to produce higher quality catalysts with improved properties.

The reasons why potassium hydroxide was used are detailed above.

**References/Applicable Publications:**

(1) Cargo Systems Manual: GAS Annex for STS-7. JSC-17645 Annex STS-7, April 1, 1983. (short description; preflight)

(2) Schmitt, G.: The G-002 JUFO-1 Payload, Its Objectives and Results. In NASA Goddard Space Flight Center's Get Away Special Experimenter's Symposium, August 1-2, 1984, pp. 135-142, NASA CP-2324. (post-flight)

(3) STS-7 Seventh Space Shuttle Mission, NASA Press Kit, June 1983, p. 55. (short description; preflight)

(4) Riepl, H.: Physical and Chemical Properties of a Nickel Catalyst Processed Under Microgravity. In Proceedings of the 5th Symposium on Material Sciences Under Microgravity, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 359-363. (post-flight)

(5) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

(6) Input received from K. Kemmerle, December 1989.

(7) "STS-7 Getaway Specials," NASA News, NASA/GSFC, May 1983.

(8) Input received from G. Schmitt (Kayser-Threde), July 1993.

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**Co-Investigator(s):** Megill, L. R. (Payload Manager and Contributor) (2), Busboso, E. (NASA Technical Manager) (3)
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**Experiment Origin:** Scotland
**Mission:** STS Launch #10, STS-011 (STS 41-B Challenger)
**Launch Date/Expt. Date:** February 1984
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** NASA Get Away Special (GAS) Canister G-004
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of this Experiment Within G-004:** University of Aberdeen, Scotland
**Processing Facility:** Evacuated aluminum chamber containing small glass cylinders
**Builder of Processing Facility:** University of Aberdeen Workshop, Aberdeen, Scotland

**Experiment:**
<u>Three Dimensional Brownian Motion</u>

This experiment was one of six investigations housed within the G-004 Get Away Special canister during STS-011. Three other experiments (of the six) were applicable to this data base (see Kitaura, T., STS-011 (Chapter 2); Thomas, S., STS-011 (Chapter 12); Gadsden, M. "Stability of Materials-Residual Acceleration Measurements," STS-011 (Chapter 18)).

Reportedly, the objective of the experiment was to "...measure the mean rotational speed of small glass cylinders suspended in a low pressure gas" [i.e., three-dimensional Brownian motion]. (3, p. 4)

On Earth, such an experiment would be difficult to perform because (1) the cylinders would have to be large enough to be visible and (2) the gas pressure would have to "...be low enough for the mean free path of the gas molecules to be greater than the length of the cylinders." (3, p. 4) In such a terrestrial experiment, the cylinders would fall essentially unhindered to the bottom of the experimental container.

The experimental apparatus consisted of "A small evacuated chamber containing a number of glass cylinders 1 mm long and 0.02 mm diameter.... A computer-controlled flashlight was arranged to give 60 flashes, equally spaced over a period of 30 seconds.

[During the mission] A camera (without a shutter) recorded the movement of the suspended cylinders by the multiflash technique familiar to photographers.

"Images of several cylinders were...[obtained] and all the photographic arrangements worked well, with some fogging of the film over the nine months between loading the film in the summer of 1983, and the Shuttle flight with subsequent development of the film in spring of 1984..." (3, p. 4)

Analysis of the film indicated the cylinders turned more slowly than the rate predicted by theoretical analysis. It was determined that a higher than expected pressure in the chamber contributed to this slow rate of rotation. This higher pressure appears to have been caused by a tiny leak in the vacuum tight joints. "What was of interest was to learn that the vibration and acceleration of the Shuttle during the 30-second operating period is quite undetectable in this experiment." (3, p. 4)

No further information concerning this experiment could be located at this time.

**Key Words:** Physical Chemistry, Brownian Motion, Suspension of Particles, Particle Motion, Acceleration Effects, Accelerations/Vibrations Produced by Onboard Equipment, Vacuum, Photographic Difficulties

**Number of Samples:** One evacuated chamber; a number of glass cylinders
**Sample Materials:** glass cylinders
**Container Materials:** aluminum
(Al*)

**Experiment/Material Applications:**
Unspecified.

**References/Applicable Publications:**
(1) Cargo Systems Manual: GAS Annex for STS-11, JSC-17645 Annex STS-11, December 2, 1983. (mention of experiment only; preflight)

(2) Student Gas Program Internal Document, Utah State University, Logan Utah, 1984.

(3) Letter From Michael Gadsden (Dept. of Natural Philosophy, Aberdeen University, Aberdeen, Scotland) to L. Rex Megill (Utah State University Student GAS Program Manager, Logan, Utah) which included a short report by Thomson, D. A. entitled: Getaway Special Undergraduate Project-Brownian Motion (Rotation) (Preliminary Results of University of Aberdeen's G-004 Experiments). (post-flight)

(4) Getaway Special (GAS) Payloads. In Goddard Space Flight Center's Engineering Newsletter, Vol. 2, No. 3, April 1984, Published by the Engineering Directorate, p. 8. (very short summary)

(5) STS-11 Getaway Special Payload Descriptions, NASA News, NASA GSFC 1984.

(6) NASA Press Kit, Mission 41-B, p. 27. (preflight)

(7) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(8) Input received from Experiment Investigator, July 1993.

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**Co-Investigator(s):** Birman, B. (Program Manager) (2), General Electric Space Division (Sponsor and Contributor) (3)
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**Experiment Origin:** USA
**Mission:** STS Launch #24, STS-032 (STS 61-C, Columbia)
**Launch Date/Expt. Date:** January 1986
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** College Student Experiment
NASA Get Away Special (GAS) Canister G-062
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay (on the standard NASA GAS bridge carrier system)
**Primary Developer/Sponsor of G-062:** Pennsylvania State University, University Park, Pennsylvania/GE Space Division, Philadelphia (Valley Forge), Pennsylvania
**Processing Facility:** The expected experiment configuration was to consist of three concentric cylinders (the inner cylinder containing a resistive heat source)
**Builder of Processing Facility:** Principal Investigator M. Kedzierski, Pennsylvania State University, University Park, Philadelphia, Pennsylvania

**Experiment:**
Convection Experiment

Radiation, conduction, and convection simultaneously contribute to the overall heat transfer of fluids and materials systems. It therefore becomes difficult to separate the individual heat transfer contributions of each mode in a terrestrial-based system. In a space environment, however, the convective, gravity-driven mode is greatly reduced, and heat transfer should be primarily governed by the conductive and radiative contributions.

The objective of this research was to determine the role of gravity-driven convection in the overall heat transfer in Earth-based systems. The experiment was one of three investigations housed within the G-062 Get Away Special canister during STS-032. Two other experiments (of the three) were also applicable to this data base (see Rice, J., STS-032 Chapter 12); Thames, M., STS-032 (Chapter 12)). <Note: References (2) and (3) listed a fourth experiment in the canister: "Liquid Droplet Heat Radiator Experiment." However, it appears that this fourth experiment was canceled prior to the shuttle flight and that the canister was configured with only the three mentioned experiments.>

A description of the actual inflight experimental setup and procedure could not be located at this time. A document released prior to the shuttle flight described the expected experimental setup:

"The experiment consists of three concentric cylinders. The inner cylinder contains a resistive heat source that will heat oil between the inner and middle cylinders. There will exist a partial vacuum between the middle and outer cylinders. The temperature measurements will be spatially recorded by 16 high sensitivity thermobeads. Heat transfer will be measured, and after comparison to one-G results, the effect of convection in heat transfer will be determined." (1, p. 3)

Reference (3) (a document published after STS 61-C returned to Earth) briefly noted that either the canister or the G-062 experiments <it is unclear which> "Failed to turn on." The reference further noted that "The batteries tested fine after return to Earth. Suspect a relay or controller problem." (3, p. 37)

<Note: It is unclear what batteries tested fine after the return of the shuttle. The only other reference to "batteries" in the available publications was in Reference (1) and is of note. "The electrical power will be supplied by an array of Gates Lead-Acid cells. These cells were chosen due to their consistent safety characteristics in space, a prime concern of NASA. NASA has imposed a three month waiting period prior to launch, which seriously affects the power capacity prior to launch. Due to these and other factors, low power CMOS circuitry was used throughout the design." (1, p. 5)>

<Note: It is suspected that due to the above mentioned anomaly (a turn-on failure), no experimental data were obtained.>

No further information describing the experimental setup, suspected experimental timeline, failure analysis, or experimental results could be located at this time.

**Key Words:** Physical Chemistry, Buoyancy-Driven Convection, Resistance Heating, Conduction, Radiation, Heat Transfer, Vacuum, Contained Fluids, Hardware Malfunction, Sample Not Processed As Planned

**Number of Samples:** unclear, appears one
**Sample Materials:** oil
**Container Materials:** unknown

**Experiment/Material Applications:**
No specific application of this research was cited. However, understanding the nature of convective flow and/or heat transfer is a basic goal of most space experiments. During terrestrial experiments, convective flow occurs in many fluid systems (e.g., crystal growth melts, metallic melts, etc.).

**References/Applicable Publications:**
(1) Thames, M., Moul, D., and Birman, B.: Penn State Get Away Special G-62. In NASA Goddard Space Flight Center's 1984 Get Away Special Experimenter's Symposium, August 1-2, 1984, pp. 1-8, NASA CP-2324. (preflight)

(2) NASA Space Shuttle Mission 61-C Press Kit, December 1985, p. 16. (preflight; very short description)

(3) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR 87-11, October 2, 1987. (Get Away Special canister mission history)

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**Co-Investigator(s):** None
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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 14A
**Launch Date/Expt. Date:** May 1986
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-7: Ultrasound Absorption Cell (The cell included an isothermal furnace for sample melting, a stationary wave generation system to produce ultrasonic waves, and a laser to permit sound absorption measurements.)
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Ultrasonic Absorption in Molten Salts

When ultrasound waves are absorbed in liquids, periodic pressure and temperature changes in the fluid result in molecular processes within the system. One of these processes is a structural transformation of the liquid. Sound attenuation is related, (in part), to internal friction and heat conduction. It follows that both the friction and heat are linked to the volume viscosity. Experimental determination of this viscosity is possible only by sound absorption measurements. On Earth, convective flows in the melt hamper measurements of the ultrasound absorption coefficients.

This TEXUS 14a experiment was the first in a series of investigations designed by Richter et al. to study the ultrasonic absorption of molten salts. The specific objective of the experiment was to use an improved Debye and Sears sound lattice method to determine ultrasound absorption coefficients of pure molten silver nitrate. (This ion melt has a more simple stature than watery solutions and thus, makes structural investigations more easy to clarify.)

During the experiment, a periodic sound wave of 25 MHz generated periodic density fluctuations in the 275 $^{o}$C melt. Such fluctuations represented a phase grid for a laser directed perpendicular to the soundwave and through the grid. If convection had been absent from the fluid, the bending of the laser beam would have been proportional to the intensity of the sound, and an absorption measurement could have been made.

Although the experiment proceeded as planned, an atypical wobbling motion of the sounding rocket produced a higher than expected low-gravity environment. This environment produced a high degree of convection in the system. Reportedly, however, the successful functioning of the module was proved.

**Key Words:** Physical Chemistry, Molten Salts, Melt and Solidification, Ultrasonic Waves, Ultrasonic Absorption, Sound Absorption Measurements, Sound Attenuation, Sound Lattice Method, Sound Grid Method, Debye-Sears-Effect, Structural Transformation, Buoyancy-Driven Convection, Density Distribution, Heat Conduction, Viscosity, Laser Light Scattering, Laser Beam Diffraction, Rocket Motion, Acceleration Effects

**Number of Samples:** one
**Sample Materials:** pure silver nitrate melt
(Ag*N*O*)
**Container Materials:** unknown

**Experiment/Material Applications:**
On Earth, measurements of molten-salt ultrasound absorption coefficients are difficult to perform using the sound grid method. Stray laser light, (which seriously impairs an accurate measurement of light intensity) is produced by the convective currents in the melt. The low-gravity environment allows investigation of such coefficients under reduced convective conditions. In addition the investigations provide reference data for ground-based experiments.

**References/Applicable Publications:**
(1) Experimentmodul TEM 06-7. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, p. 103. (in German; experiment module)

(2) Kirschbaum, E. and Richter, J.: Bestimmung der Schallabsorption von Salzschmelzen durch Beobachtung des DEBYE-SEARS Effektes. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 104-106. (in German; post-flight)

(3) Richter, J., Kirschbaum, E., Merkens, W., and Vogt, M.: Microgravity Effects on Ultrasonic Absorption in Molten Salts. In ESA SP-295, October 1989, p. 481.

(4) Fuchs, B., Fuchs, W., Kirschbaum, E., and Richter, J.: Messung der Volumenviskosität von binären Salzschmelzen unter Schwerelosigkeit. Final Report BMFT (1986).

(5) Kirschbaum, E. and Richter, J.: Bestimmung der Schallabsorption von Salzschmelzen durch optische Beobachtung des Debye-Sears-Effecktes. Final Report TEXUS 13/14. <Note: The year this document was published is unclear at this time.>

(6) Kirschbaum, E.: Bestimmung der Ultaschallabsorption in Salzschmelzen unter verminderter Schwerkraft. Thesis, RWTH Aachen (1988).

(7) Input received from Experiment Investigator, August 1989.

(8) Input received from K. M. Schefold (CPC-$\mu$g, RWTH Aachen), August 1993.

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**Co-Investigator(s):** None
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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 14B
**Launch Date/Expt. Date:** May 1987
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-7: Ultrasound Absorption Cell (The cell included an isothermal furnace for sample melting, a stationary wave generation system to produce ultrasonic waves, and a laser to permit absorption measurements. The module was modified post TEXUS 14a to monitor melt characteristics during rocket countdown.)
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Ultrasonic Absorption in Molten Salts

"An acoustic wave in a medium is absorbed and dissipated by inner friction, heat conduction, thermal radiation, relaxation processes, and chemical reactions. However, the classical theory only considers shear viscosity and heat conduction. The other effects are usually summarized under the phenomenological term 'volume viscosity.' The experimental determination of the volume viscosity can thus give insights into the process of relaxation and chemical reactions, provided that the frequency of the relaxation is higher than the frequency of the transmitted sound wave.

"Ultrasound absorption measurements can be performed using the Debye Sears optical method.... The intensity of the first order diffraction of the laser beam is proportional to the sound intensity in the melt. With the relation: I(x) = Io exp (-2[alpha]x) where I(x) is the sound intensity in the melt at a distance x from a source of intensity Io, one can deduce the absorption coefficient [alpha]. Also, the sound velocity can be deduced from the distance between the +1. and -1. orders. To avoid wall effects, this method requires a large volume of melt where, on Earth, convective flows cannot be avoided. These flows cause additional and unstable diffraction effects which impede a proper... [focusing] of the beam on the detection diode, which then yields lower intensity measurements and thus higher absorption values. The longer the distance from the source, the higher the cumulated disturbance effect, i.e. that even the slope of the curve Ln(I) versus x, and thus [alpha], cannot be reliably determined." (6, p. 110)

This TEXUS 14B experiment was the second in a series of investigations designed by Richter et al. to study the ultrasonic absorption of molten salts (see Richter, TEXUS 14A). The specific objective of the experiment was to use the improved Debye and Sears sound lattice method to determine the ultrasonic absorption of the binary salt melt rubidium nitrate + silver nitrate (33.3% by mol of $AgNO_3$ and 66.6% by mol of $RbNO_3$).

During the mission, a periodic sound wave of 25 MHz generated periodic density fluctuations in the 230 $^{o}C$ melt. An absorption measurement was made by examining the laser intensity through the fluid and relating it to the intensity of the sound.

Reportedly, although a lower sound absorption under microgravity was expected, a 20% higher sound absorption was measured. "This difference was well beyond the accuracy of the method. This was contrary to expectations, since the absence of convection should have led to higher intensities and thus lower absorption values." (6, p. 110)

No further information concerning this experiment could be located at this time. <Note: References (3) and (4) (as listed below) could not be located at this time.>

**Key Words:** Physical Chemistry, Melt and Solidification, Molten Salts, Binary Systems, Ultrasonic Waves, Ultrasonic Absorption, Sound Absorption Measurements, Sound Attenuation, Relaxation Processes, Sound Lattice Method, Debye-Sears-Effect, Structural Transformation, Buoyancy-Driven Convection, Density Distribution, Conduction, Heat Conduction, Radiation, Viscosity, Laser Light Scattering, Laser Beam Diffraction, Wall Effect

**Number of Samples:** one
**Sample Materials:** binary salt melt from 33.3% by mol of silver nitrate and 66.6% by mol of rubidium nitrate
(Rb*N*O*Ag*N*O*)
**Container Materials:** unknown

**Experiment/Material Applications:**
See Richter, TEXUS 14A.

**References/Applicable Publications:**

(1) Experimentmodul TEM 06-7. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, p. 103. (in German; experiment module)

(2) Kirschbaum, E. and Richter, J.: Bestimmung der Schall-absorption von Salzschmelzen durch Beobachtung des DEBYE-SEARS-Effektes. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht 1988, pp. 104-106. (in German; post-flight)

(3) Kirschbaum, E. and Richter, J.: Bestimmung der Schallabsorption von Salzschmelzen durch optische Beobachtung des Debye-Sears-Effektes. Final Report TEXUS 13/14. (in German) <Note: The date this paper was published is unclear at this time.>

(4) Kirschbaum, E.: Bestimmung der Ultraschallabsorption in Salzschmelzen unter verminderter Schwerkraft. Thesis, RWTH Aachen (1988). (in German)

(5) Input received from Experiment Investigator, August 1989.

(6) Ultrasound Absorption in Molten Salts. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 110-11. (post-flight)

(7) Input received from K. M. Schefold (CPC-μg, RWTH Aachen), August 1993.

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**Co-Investigator(s):** None
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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 17
**Launch Date/Expt. Date:** May 1988
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-7: Ultrasound Absorption Cell
**Builder of Processing Facility:** Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Ultrasonic Absorption in Molten Salts

This TEXUS 17 experiment was the third in a series of investigations designed by Richter et al. to study the ultrasonic absorption of molten salts (see Richter, TEXUS 14A, TEXUS 14B). The specific objective of the experiment was to use the improved Debye and Sears sound lattice method to determine the ultrasonic absorption of the binary salt melt rubidium nitrate + silver nitrate (50% by mol of $RbNO_3$ and 50% by mol of $AgNO_3$).

During the mission, a periodic sound wave of 25 MHz generated periodic density fluctuations in the 230 $^{o}$C melt. An absorption measurement was made by examining the laser intensity through the fluid and relating it to the intensity of the sound.

Reportedly, "When compared with ground results, the flight results showed again that a higher absorption of ultrasound (25 MHz) occurred in microgravity. The absorption coefficient deduced from the flight measurements was 14% higher than the one obtained on Earth, which was in agreement with the previous space results.

"These values cannot be explained as the result of the absence of convection in microgravity. In addition, the ground results were confirmed by using two different methods: the Debye Sears method and the Pulse-Transmission method and by performing measurements with the laboratory equipment (both methods) and in the flight module on the ground (Debye-Sears method). It is therefore believed that the higher absorption was strictly a microgravity effect whose further study may allow some insights into the structure of liquids." (3, p. 112)

<Note: Reference (4) was not available to aid in the preparation of this experiment summary. Efforts are being made to procure the document at this time.>

**Key Words:** Physical Chemistry, Melt and Solidification, Molten Salts, Binary Systems, Ultrasonic Waves, Ultrasonic Absorption, Sound Absorption Measurements, Sound Attenuation, Sound Lattice Method, Debye-Sears-Effect, Structural Transformation, Buoyancy-Driven Convection, Density Distribution, Conduction, Heat Conduction, Radiation, Viscosity, Laser Light Scattering, Laser Beam Diffraction, Wall Effect

**Number of Samples:** one
**Sample Materials:** rubidium nitrate + silver nitrate melt from 50% by mol of $RbNO_3$ and 50 by mol of $AgNO_3$
(Rb*N*O*, Ag*N*O*)
**Container Materials:** unknown

**Experiment/Material Applications:**
See Richter, TEXUS 14A.

**References/Applicable Publications:**

(1) Richter, J. and Behret, H.: Physical Chemistry-Overview and Selected Experiments. In Fluid Science and Materials Science in Space, edited by Walter, H. U., Springer Verlag, 1987, pp. 141-156. (related topics only)

(2) Input received from Experiment Investigator, August 1989.

(3) Ultrasound Absorption in Molten Salts. In Summary Review of Sounding Rocket Experiments In Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 112-113. (postflight)

(4) Kirschbaum et al.: Microgravity Effects on Ultrasonic absorption in Molten Salts. In the 7th European Symposium on Material and Fluid Sciences in Microgravity, ESA SP-295, October 1989, pp. 481-484.

(5) Input received from K. M. Schefold (CPC-$\mu$g, RWTH Aachen), August 1993.

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**Experiment Origin:** Sweden
**Mission:** MASER 1
**Launch Date/Expt. Date:** March 1987
**Launched From:** Esrange, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment; College Student Experiment
**Processing Facility:** Fluid Science Module (FSM)
**Builder of Processing Facility:** Saab Space, AB, Linköping, Sweden

**Experiment:**
Thermal Conductivity of Electrically Non-conducting Liquids

Accurate thermal conductivity measurements (due to diffusion alone) are difficult to obtain in terrestrial fluids systems. On Earth, the overall heat transfer of the system is dependent on both gravity-independent diffusive forces and gravity-dependent convective forces. In a low-gravity environment, superior thermal conductivity characterization is expected because heated liquid systems should exhibit purely diffusive heat transfer.

This MASER 1 experiment was designed to measure the thermal conductivity of an electrically non-conducting liquid using a transient hot wire technique.

Two fluid cells within the MASER Fluid Science Module (FSM) were dedicated to the experiment. <Note: It is unclear if these cells were cylindrical, three-dimensional rectangular, or two-dimensional rectangular, although they appear to have been cylindrical.> Prior to the flight, each cell was filled with pure ethanol and configured with a 50-micron diameter platinum (hot) wire. The wire stretched down the center, vertical (long) axis of the cell.

<Note: Although it was reported that one cell was "preheated" to 30 $^{\circ}$C and the other to 50 $^{\circ}$C, it is unclear if this heating was initiated prior to the start of the low-gravity phase or after the low-gravity phase had been obtained. (It appears that the liquid in each cell was warmed by an outer, concentric cylindrical heater (the same length as the cell)).>

During the experiment, a constant current was passed through the wires. Thermal conductivity measurements were derived from voltage drops which occurred across the 100-mm measuring length of the wire. The temperature in the liquid was measured by two thermocouples located near the wire.

Although pure conduction was expected, data from the "30 $^{o}$C cell" indicated that an abrupt change in wire temperature occurred "after 120 seconds." <Note: It appears "after 120 seconds" means 120 seconds after the initial preheating of the cell.> The wire temperature later oscillated.

These results were further detailed in Reference (7):
"The data obtained with the 30 $^{o}$C cell were normalized to dimensionless data with a value of heat conductivity taking into account the aging of the liquid which had been carefully evaluated. These data were compared to those one should obtain in the ideal case....

"The difference between the data at the very beginning originated in the heating of the wire itself. Then, up to 13 s, the data were in good agreement, thereby yielding the value of the heat conductivity of ethanol: 0.179... [+/-] 0.0045 W/Km at 28 $^{o}$C. After 13 s, there was a slight change in slope and after 120 s, the wire temperature decreased drastically. This was interpreted as the result of the onset of a convective regime probably induced by the accumulation of the effects of the radial component of the residual accelerations of the rocket." (7, p. 114)

In the "50 $^{o}$C cell," a similar deviation from pure conduction was observed as well as the wire temperature oscillation. Reportedly, because this cell had a slight leak, this data was thought to be "less relevant."

Because similar (preflight) terrestrial experiments had illustrated a high degree of data measurement reproducibility, it was suspected that no malfunction of the cell/electronic system had taken place. It was reported (Reference (4)) that the deviation in wire temperature was to be further investigated.

No other information concerning the results of this experiment could be located at this time.

**Key Words:** Physical Chemistry, Diffusion, Diffusive Mass Transfer, Thermal Conductivity Measurements, Transparent Liquids, Buoyancy-Driven Convection, Hot-Wire Technique, Electric Field, Thermal Oscillations, Contained Fluids, Liquid Leakage, Acceleration Effects, Rocket Motion

**Number of Samples:** two
**Sample Materials:** pure ethanol
**Container Materials:** plastic

**Experiment/Material Applications:**
No direct application of this research was presented. However, accurate thermal conductivity measurements are required for a number of fluids and materials science applications.

**References/Applicable Publications:**

(1) Zaar, J. and Änggard, K.: Maser and Its Effectiveness and Experimental Results. In: In Space '87, Japan Space Utilization Promotion Center (JSUP), October 13-14, 1987. (post-flight; short description)

(2) Jönsson, R.: The Microgravity Program in Sweden - Emphasis on the Materials Rocket Maser. In 15th International Symposium on Space Technology and Science, Tokyo, Japan, May 19-23, 1986, Vol. 2, pp. 2099-2110. (preflight)

(3) Zaar, J., Björn, L., and Jönsson, R.: Preliminary MASER 1 Results and the Evolution of the MASER Programme. In Proceedings of the 8th ESA Symposium on European Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 359-361. (post-flight; very short description)

(4) Grunditz, H.: Flight Results of the ESA Experiment Modules in MASER 1. In Proceedings of the 8th ESA Symposium on Rocket and Balloon Programmes and Related Research, Sunne, Sweden, May 17-23, 1987, ESA SP-270, pp. 363-367. (post-flight)

(5) Grunditz, H.: Experiment Equipment for Metallurgy and Fluid Science Studies Under Microgravity. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

(6) Jönsson, R.: SSC Microgravity Sounding Rocket Program MASER. 37th Congress of the International Astronautical Federation, Innsbruck, Austria, October 4-11, 1986. (preflight)

(7) Thermal Conductivity of Liquids. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, ESA SP-1132, February 1991, pp. 114-115. (post-flight)

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**Experiment Origin:** The Netherlands
**Mission:** TEXUS 15
**Launch Date/Expt Date:** May 1987
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-12
**Builder of Processing Facility:** ERNO, Germany

**Experiment:**
Colloidal Chemistry

This TEXUS 15 experiment was the first in a series of investigations designed by Stein to study colloid chemistry under low-gravity conditions.

Information describing the specific experimental objectives and equipment setup of this investigation could not be located at this time.

Reportedly, shortly after the successful launch of the TEXUS rocket, data and television transmitters experienced a partial failure. It was discovered that a lateral burnthrough of the second stage of the rocket had occurred, and the stage, in turn, had collided with the prematurely separated payload. An unbraked impact of the lower part of the payload resulted in the complete destruction of the TEM 06-12 experiment module.

Documentation further detailing any results of this experiment does not appear to be available.

**Key Words:** Physical Chemistry, Colloidal Chemistry, Coagulation, Particle Motion, Particle Dispersion, Suspension of Particles, Sedimentation, Solid/Liquid Interface, Payload Survivability, Acceleration Effects, Rocket Failure

**Number of Samples:** nine

**Sample Materials:** silica; quartz <Note: The employed solution (if indeed one was employed) was not detailed. (A solution was employed on the later TEXUS 17 mission.)>
(Si*O*)
**Container Materials:** unknown

**Experiment/Material Applications:**
See Stein, TEXUS 17.

**References/Applicable Publications:**

(1) Experimentelle Nutzlast und Experimente TEXUS 15. In BMFT/DFVLR TEXUS 13-16 Abschlussbericht, 1988, pp. 107-108. (in German; post-flight)

(2) Input received from Experiment Investigator, August 1993.

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**Co-Investigator(s):** Krutser, L.L.M. (2)
**Affiliation(s):** (1) Lab. Colloid Chemistry, Technische Hogeschool Eindhoven, Eindhoven, The Netherlands; (2) Unclear, possibly: Technische Hogeschool Eindhoven, Eindhoven, The Netherlands

**Experiment Origin:** The Netherlands
**Mission:** TEXUS 17
**Launch Date/Expt Date:** May 1988
**Launched From:** ESRANGE, Kiruna, Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-12
**Builder of Processing Facility:** ERNO, Germany

**Experiment:**
Colloidal Chemistry

The rate of ortho-kinetic coagulation of particles greater than 1 micron in diameter can be described by the modified van Smoluchowski equation: $dn/dt = (-16/3)\ [\mathrm{Alpha}]_o\ n^2\ a^3\ [\mathrm{Gamma}]$, where n is the number of particles per unit volume, t is time, a is the particle radius, Gamma is the shear rate, and $\mathrm{Alpha}_o$ is the capture efficiency.

To determine the capture efficiency, the following must be accounted for: (1) particle attraction and repulsion forces, (2) particle deviation from a spherical shape, and (3) particle inertial forces. Earth-based experimental investigations which examine the influence of the last two factors at low shear rates are hampered by gravity-induced sedimentation of the particles.

This TEXUS 17 experiment was the second in a series of investigations designed by Stein et al. to study colloid chemistry under low-gravity conditions (see Stein, TEXUS 15). The specific objective of the experiment was to determine the capture efficiency of particles in a colloidal system without the interference of gravity-induced sedimentation.

During the mission, the TEXUS Experiment Module TEM 06-12 was used to investigate the coagulation of quartz particles (0.015 vol.%, 1.25 micron diameter) in a 0.5 M NaCl solution. Samples of the experimental material were contained in nine separate cells. After homogenization of the solutions (by ultrasonic mixing), the liquid in several of the cells was stirred with rotating magnets. Reportedly, (1) two of the cells had a shear rate of 0 rpm, (2) two of the cells had a shear rate of 50 rpm, (3) two of the cells had a shear rate of 100 rpm, and (4) two of the cells had a shear rate of 220 rpm. <Note: The 220 rpm shear rate was confirmed by the Experiment Investigator. (Reference (1)

indicated that a rate of 220 rpm was employed.)> A laser beam was transmitted through each of these eight cells. (The particle density evolution with time was determined via beam transmission measurements.) The ninth cell, which was equipped with a cine camera, was used to record the fluid flow patterns (within that cell) produced by applying different shear rates. <Note: These shear rates were not detailed.> Similar experiments were performed on Earth for comparison.

<Note: Very little information concerning the post-flight analysis of the data could be located. The following short summary of the experiment results was presented in Reference (1) and is reproduced here nearly in its entirety. It should be noted that the contents of this summary (as written) were not clear to the editors.>

"Whereas the flight and ground results were comparable at 220 rpm, the light transmission measurements increased further in microgravity (increased coagulation) than on Earth, at lower shear rate.... Also, the shape of the [light transmission vs. time] curves was different. That the Brownian motions are more important in microgravity was not a satisfactory explanation of these results and nothing indicated that the damping of the residual flows should have been slower in the flight cells than in the same cells on the ground." (1, p. 116)

<Note: Reference (2) was not available to aid in the preparation of this experiment summary.>

**Key Words:** Physical Chemistry, Colloidal Chemistry, Coagulation, Particle Motion, Particle Dispersion, Particle Mobility, Particle Attraction, Particle Repulsion, Suspension of Particles, Inclusion and/or Rejection of Particles, Sphericity, Solid/Liquid Interface, Sedimentation, Shear Rate, Ultrasonic Mixing, Liquid Mixing, Stirring of Components, Fluid Motion Damping, Liquid Dynamic Response, Brownian Motion, Laser Light Scattering, Contained Fluids

**Number of Samples:** nine
**Sample Materials:** 0.015 vol.% quartz particles (1.25 microns in diameter) in a 0.5 M NaCl solution
(Si*O*, Na*Cl*)
**Container Materials:** unknown

**Experiment/Material Applications:**
See **Experiment** summary (above).

The reasons why the quartz particles and NaCl solution were selected for this experiment were not presented in the available publications.

**References/Applicable Publications:**

(1) Colloid Chemistry. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 116-117. (post-flight)

(2) Krutser, L.L.M.: The Influence of Flow Type, Particle Type and Gravity on Orthokinetic Coagulation. Ph.D. thesis. <Note: Additional access information unavailable.>

(3) Input received from Experiment Investigator, August 1993.

(4) van Diemen, A.J.G. and Stein, H. N.: Coagulation of S/L Dispersions Under Microgravity Conditions: Status Report 1.11.1986. Proc. of the 6th European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 173-177. (preflight)

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**Co-Investigator(s):** None
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**Experiment Origin:** USA
**Mission:** Consort 1 (Starfire Rocket)
**Launch Date/Expt. Date:** March 1989
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Eight electrodeposition cells and two electrocodeposition cells
**Builder of Processing Facility:** University of Alabama in Huntsville and McDonnell Douglas Space System Company, Huntsville, Alabama

**Experiment:**
Electrodeposition of Cells/Electrocodeposition of Cells

During electrodeposition experiments performed on Earth, gravity-induced convection currents in the electrolytic solution (1) drive heavy ion-rich solutions (near the anode) to the bottom of the experiment cell and (2) drive lightweight, ion-depleted solutions (near the cathode) to the top of the experiment cell.

Similarly, during electrocodeposition experiments performed on Earth (electrodeposition of metal together with an inert constituent), fluid motion in the electrolytic solution is produced by (1) gravity-induced convective currents (mentioned above) and (2) gravity-induced sedimentary forces (which drive the typically micron-sized particles (cermets) to the bottom of the experiment cell).

Convective and sedimentary forces in these two types of experiments contribute to overall crystalline microstructure of the deposited material. In an effort to reduce these gravity-induced flow phenomena and possibly produce improved surface coatings and metal catalysts, electrodeposition and electrocodeposition experiments were performed on the Consort 1 flight. The specific experiment objectives included (1) examining the role of convection and sedimentation in the overall electrodeposition process, (2) understanding how the low-gravity electrodeposition rate affects the deposited crystalline structure, (3) determining if the cermets can be suspended, dispersed, and electrocodeposited, and (4) verifying the findings of Ehrhardt who, during his TEXUS sounding rocket experiments, produced amorphous nickel when an electrodeposition current of 80 $ma/cm^2$ was applied (see Ehrhardt, TEXUS 7; TEXUS 9 (this chapter)).

The experimental payload consisted of 10 small electrodeposition cells: seven to deposit nickel, one to deposit pure cobalt, one to codeposit nickel and diamond dust, and one to codeposit a cobalt/$Cr_3C_2$ composite.

Electrodeposition Experiments:

The rate of electrodepositioning of nickel was studied by passing a variety of current densities across the seven nickel experiment cells. During the mission, the applied voltage in four identical cells was potentiostatically controlled in "...1.5 volt increments from 4.5 to 9.0 volts producing current fluxes that correspondingly varied between approximately 35 and 85 milliamps (ma/cm$^2$)...." (3, p. 8) (These values bracketed the values Ehrhardt employed in his TEXUS experiments). The remaining three cells (which contained nickel sulfamate) were "...potentiostatically controlled at 3 V, but the distance between electrodes was shortened to 1/4 that of the other cells to reduce resistance. Also, in two of these shortened cells the cathode to anode area ratio was decreased to 1/2 and 1/4 of that of the... [other cell of this type] to give different [electrodeposition] rates." (4, p. 17)

Reportedly, "The pure cobalt was deposited at 1.5 V and a pH of 4.2." (4, p. 17)

Post-flight analysis of the electrodeposition products in the shortened cells indicated that the anticipated rate extremes had not been achieved. It was found that the resulting surfaces produced in the short cells were identical to those found in 1-g face centered cubic nickel.

Post-flight analysis of the four cells whose deposition rates had been altered by changing the applied voltage indicated that these experiments worked well and produced the results hoped for. "The X-ray diffraction spectra of the cathode surfaces at the four different rates... [was presented]. The lowest rate (4.5 volts and... [approximately] 35 ma/cm$^2$)... [produced] the usual Face Centered Cubic (FCC) nickel structure. As the rate progressively... [increased] the nickel... [became] more X-ray transparent. Scanning electron microscopy... [indicated] a definite change in the granularity of the nickel surface going from relatively large granularity to small with increasing rate. Either the nickel... [had] deposited amorphously or in a microcrystalline state too small for X-ray analysis. Unfortunately, the expected nearest neighbor position of glassy (amorphous) nickel would be masked by the AU 200 diffraction." (3, p. 8)

No discussion of the electrodeposition of pure cobalt was presented because no difference between low-g and 1-g was apparent.

Electrocodeposition Experiments:

After the rocket had been launched and the low-gravity phase of the mission had been attained, the materials in each codeposition cell were mixed by a stirring motor for 15 seconds. "The fluid motion in the cells was allowed 10 seconds to damp itself before 400 seconds of electrocodeposition was initiated." (Reference (9)) The two experiment cells were photographed before stirring, during damping, and then approximately every 15 seconds during codeposition.

Post-flight analysis of the electrocodeposition photographs indicated that (1) "...the cermets clumped in a large mass immediately after stirring." (3, p. 9) and (2) both types of micron-sized particles (diamond dust and chromium-carbide) "...had been moved but were clumped all around the sides of the cell cavities.... [Although a] good suspension/dispersion [was not maintained,] codeposition... [was] evident. Clumping of upwards of tens of particles... [was] noted in the $Cr_3C_2$ with cobalt while only small groupings of several particles... [could] be seen in the diamond with nickel." (4, p. 19)

The results indicated "...that either a surfactant and stirring, a surfactant and continued stirring throughout the flight or just continued stirring may be necessary to carry out codeposition in low gravity." (3, p. 9)

<Note: References (7) and (8) were not available to aid in the preparation of this experiment summary.>

**Key Words:** Physical Chemistry, Electrodeposition, Electrocodeposition, Codeposition, Dispersion Layers, Thin Films, Coated Surfaces, Plating, Electric Field, Cathode, Anode, Electrodes, Electrolyte Solution, Ionic Solution, Particle Dispersion, Particle Mobility, Particle Motion, Particle Distribution, Suspension of Particles, Sedimentation, Stirring of Components, Shear Forces, Fluid Motion Damping, Liquid Dynamic

Response, Coagulation, Buoyancy-Driven Convection, Crystalline Structure, Sample Microstructure, Surface Morphology, Metallic Matrix, Solid/Liquid Interface, Amorphous Materials, Catalysts, Hardness, Surfactants, Contained Fluids

**Number of Samples:** ten experiment cells
**Sample Materials:** seven cells: electrodeposition of nickel; one cell: electrodeposition of pure cobalt; one cell: codeposition of nickel and diamond dust; one cell: codeposition of a cobalt/$Cr_3C_2$ composite
**Container Materials:** Plexiglas™

**Experiment/Material Applications:**
"The purpose of these experiments... [was] to improve the understanding of the plating process, to determine if pure amorphous metals may be formed by electrodeposition, to determine any differences in catalytic activity between surfaces prepared in unit g and low g and to determine if there are improved characteristics of codeposited surfaces in low g." (2, p. 341)

"Codeposition processes are used to produce surface coatings with special abrasive qualities and extra hardness. These processes normally require some means of continuous mixing to keep inert products from settling out of the electrolytic solution.... [In principle it should be easier to maintain homogeneous suspensions of particles in low-g than 1-g.] Codeposition will be studied... with the objective of producing richer, more homogeneous surface coatings." (7, p. 217)

**References/Applicable Publications:**
(1) Riley, C., Abi-Akar, H., and Benson, B.: Electrodeposition of Metals and Metal/Cermet Composites on a Sub-Orbital Rocket. AIAA 27th Aerospace Sciences Meeting, January 9-12, 1989, Reno, Nevada, AIAA #89-0308. (preflight; concentrates on KC-135 results)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439, Acta Astronautica, Vol. 21, No. 9, 1980, 11 pp. (post-flight)

(4) Surface Coatings and Catalyst Production by Electrodeposition and Composite Coatings. In Consortium for Materials Development in Space, The University of Alabama in Huntsville, Huntsville, Alabama, Annual Report, Technical Section, October 1, 1988-September 30, 1989, pp. 17-21. (post-flight)

(5) Riley, C., Coble, H. D., Loo, B., Benson, B., and Abi-Akar, H.: Electrodeposition and Codeposition Under Low Gravity/Nonconvecting Conditions. Polymer Preprints, Vol. 28, No. 2, 1987, pp. 470-471. (KC-135 work only)

(6) May, C. B., Riley, C., Coble, H. D., and Loo, B. H.: Surface Coatings and Catalyst Production by Electrodeposition. NASA CR-181508, Annual Report, The University of Alabama in Huntsville, Consortium for Materials Development in Space, September 15, 1986 - September 14, 1987, pp. 32- 48. (KC-135 work only)

(7) Riley, C., Abi-Akar, H., Benson, B., and Maybee, G.: Low-gravity Electrodeposition of Metals and Metal Cermet Composites. Journal of Spacecraft and Rockets, Vol. 27, No. 4, pp. 386-390, 1990. (preflight)

(8) Abi-Akar, H. M.: Electrodeposition in Low-Gravity. Ph.D dissertation, University of Alabama in Huntsville, 1992. (preflight/post-flight)

(9) Input received from Experiment Investigator, August 1993.

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# CHAPTER 16

# PROTEIN CRYSTAL GROWTH

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**Co-Investigator(s):** Unknown
**Affiliation(s):** (1) Chemisches Laboratorium der Universität Freiburg, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3
**Launch Date/Expt. Date:** April 1980
**Launched From:** ESRANGE, Kiruna Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3
**Builder of Processing Facility:** Unknown, Possibly, ERNO, Bremen, Germany

**Experiment:**
Diffusion and Growing of Protein Beta Galactosidase Crystals

This TEXUS 3 sounding rocket experiment was the first in a series of investigations designed by Littke to study the low-gravity growth of protein crystals.

The specific TEXUS 3 experiment goals and equipment setup were not detailed in the available publications.

Reportedly, due to a rocket despin failure, TEXUS 3 did not achieve the desired low-gravity level. The experiment was reflown on TEXUS 3b (see Littke, TEXUS 3b).

No further documentation of this TEXUS 3 experiment appears to be available.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Organic Crystals, Reactant Solutions, Three-Dimensional Crystalline Structure, Buoyancy-Driven Convection, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Liquid/Liquid Interface, Solid/Liquid Interface, Crystal Morphology, Crystalline Structure, Acceleration Effects, Rocket Despin Failure, Biotechnology

**Number of Samples:** unknown
**Sample Materials:** protein, Beta-galactosidase
**Container Materials:** unknown

**Experiment/Material Applications:**
See Littke, TEXUS 3b

**References/Applicable Publications:**

(1) Greger, G.: TEXUS and MIKROBA and Their Effectiveness and Experiment Results. Presented at: In Space '87, October 13-14, 1987, Japan Space Utilization Promotion Center (JSUP). (identifies rocket failure)

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**Principal Investigator(s):** Littke, W. (1)
**Co-Investigator(s):** Unknown
**Affiliation(s):** (1) Chemisches Laboratorium der Universität Freiburg, Germany

**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 3b
**Launch Date/Expt. Date:** April 1981
**Launched From:** ESRANGE, Kiruna Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3
**Builder of Processing Facility:** Unclear, possibly ERNO, Bremen, Germany

**Experiment:**
Diffusion and Growing of Protein Beta Galactosidase Crystals

Understanding the behavior of organic, macromolecular compounds involves the determination of their three-dimensional crystalline structure. Structure determination by X-ray analysis requires large, highly perfect single crystals. On Earth, gravity-driven convection inhibits the solution growth of such high-quality crystals by promoting multiseed formation. Attempts to reduce convective flows by processing organic components in a gel medium fail because the fragile crystals break when they come in contact with the tight gel network. It was thus proposed that crystal growth during a reduced-gravity sounding rocket flight may produce larger, more perfect organic crystals.

This experiment was the second in a series of investigations designed by Littke to study the low-gravity growth of protein crystals (see Littke, TEXUS 3). The specific objectives of the experiment were to (1) evaluate the performance of the processing facility and (2) determine if diffusion-controlled conditions were achieved during the sounding rocket flight. Reportedly, the experiment was performed to help prepare for a future Spacelab 1 investigation (see Littke, Spacelab 1 (this chapter)).

Prior to the mission, a protein solution (high molecular weight Beta-galactosidase), a salt solution (ammoniumsulfate), and a buffer solution (M/30 phosphatebuffer, pH=6.4) were placed in separate chambers within the experimental apparatus. The solutions were bubble free and each container was closed by a flexible membrane for pressure compensation and equalization.

During the low-gravity portion of the mission, the three solutions were brought into contact when a sliding device separating the chambers was moved. The reactants (protein and salt solutions) diffused toward the buffer chamber where the reaction occurred. During the reaction (which took place at 20 $^{o}$C) the slowly diffusing salt ions allowed the protein molecules to crystallize. The process was observed using Schilieren optics and a cine camera. Reference experiments were performed on Earth for comparison.

It was reported that under low-gravity conditions, a strictly laminar diffusion process was observed and crystallites (approximately 1 micron in size) were formed. In contrast, turbulent convection was observed during the 1-g experiment and only amorphous protein material was obtained. Because of the short low-gravity period available (approximately 6 minutes), the quality of the flight crystals was inferior, but comparable, to that of the Earth crystals (which were obtained after several days of growth). Reportedly, these results were caused by "...a near-range order close to the order in crystallized materials, which is established in the solution in the absence of convection." (2, p. 200)

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Organic Crystals, Reactant Solutions, Salt Solution, Membranes, Three-Dimensional Crystalline Structure, Buoyancy-Driven Convection, Turbulent Flow, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Double Diffusion, Liquid/Liquid Interface, Solid/Liquid Interface, Crystal Morphology, Crystalline Structure, Pressure Compensation, Bubbles, Bubble Removal, Reaction Kinetics, Schlieren Viewing, Contained Fluids, Liquid Reservoir, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design

**Number of Samples:** one
**Sample Materials:** Protein solution: high molecular weight Beta-galactosidase; salt solution: ammoniumsulfate; buffer solution: M/30 phosphatebuffer
**Container Materials:** unknown

**Experiment/Material Applications:**
The specific reasons why Beta-galactosidase, ammoiumsulfate, and M/30 phosphatebuffer were selected for the experiment was not detailed in available publications.

**References/Applicable Publications:**

(1) Littke, W. and John, C.: Protein Single Crystal Growth Under Microgravity. In Proceedings of a joint ESA-DFVLR Workshop held at Chemisches Laboratorium der Universität, Freiburg, Germany, March 19-20, 1984, pp. 55-64. (post-flight; discusses results from all TEXUS and Spacelab experiments without distinguishing between particular missions.)

(2) Diffusion-Controlled Growth of Protein Single Crystals: B-galactosidase. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 200-201. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 4
**Launch Date/Expt. Date:** May 1981
**Launched From:** ESRANGE, Kiruna Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-5
**Builder of Processing Facility:** Unclear, possibly Messerschmitt-Boelkow-Blohm (MBB/ERNO), Bremen, Germany

**Experiment:**
Diffusion and Growing of Protein Beta Galactosidase Crystals

This experiment was the third in a series of investigations designed by Littke to study the low-gravity growth of protein crystals (see Littke, TEXUS 3, TEXUS 3b). The specific objective of the experiment was to assess the temperature range of low-gravity protein crystallization.

It was reported that the experimental apparatus, protein solution, salt solution, buffer solution, and procedure were the same as those used during the TEXUS 3b experiment with two exceptions: (1) a reaction temperature of -8 $^{o}C$ was used for the experiment (instead of TEXUS 3b temperature of + 20 $^{o}C$) and (2) the melting points of all the solutions were lowered by the addition of glycol.

<Note: It appears the temperature range of low-gravity protein crystallization was investigated by comparing the TEXUS 4 experiment to the TEXUS 3b experiment. The specific reason why the employed temperatures were selected for the TEXUS 3b and TEXUS 4 experiments was not detailed in the available publications.>

It was reported that, as in the TEXUS 3b experiment, a strictly laminar diffusion process was observed. "But the tendency for protein to crystallize at -8 deg C was reduced and nothing was obtained but a precipitate of amorphous protein material." (2, p. 202)

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Organic Crystals, Reactant Solutions, Salt Solution, Membranes, Three-Dimensional Crystalline Structure, Precipitation, Buoyancy-Driven Convection, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Double Diffusion, Liquid/Liquid Interface, Solid/Liquid Interface, Reaction Kinetics, Amorphous Materials, Crystal Morphology, Crystalline Structure, Pressure Compensation, Schlieren Viewing, Contained Fluids, Liquid Reservoir, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design

**Number of Samples:** one
**Sample Materials:** Protein solution: high molecular weight Beta-galactosidase; salt solution: ammoniumsulfate; buffer solution: M/30 phosphatebuffer
**Container Materials:** unknown

**Experiment/Material Applications:**
See Littke, TEXUS 3b.

**References/Applicable Publications:**
(1) Littke, W. and John, C.: Protein Single Crystal Growth under Microgravity. In Proceedings of a joint ESA-DFVLR Workshop held at Chemisches Laboratorium der Universität, Freiburg, Germany, March 19-20, 1984, pp. 55-64. (post-flight; discusses results from all TEXUS and Spacelab experiments without distinguishing between particular missions.)

(2) Diffusion-Controlled Growth of Protein Single Crystals: Investigation of B-galactosidase at Low Temperature. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, p. 202. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** TEXUS 5
**Launch Date/Expt. Date:** April 1982
**Launched From:** ESRANGE, Kiruna Northern Sweden
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** TEXUS Experiment Module TEM 06-3
**Builder of Processing Facility:** Unclear, possibly ERNO, Bremen, Germany

**Experiment:**
Diffusion and Growing of Protein Beta Galactosidase Crystals

This experiment was the fourth in a series of investigations designed by Littke to study the low-gravity growth of protein crystals (see Littke, TEXUS 3, TEXUS 3b, TEXUS 4). The specific objective of the experiment was to determine the reproducibility of results obtained during the earlier TEXUS 3b experiment.

Reportedly, "The same experimental conditions as in TEXUS 3[b] were reproduced and yielded the same promising results. Some rod-shaped single crystals of the enzyme B-galactosidase, molecular weight 465 000 D, embedded in amorphous protein material were obtained within the 6 minutes of the coasting phase of the rocket. Their linear dimensions are about three times larger than those of crystals grown on Earth over a period of 60 hours." (2, p. 204)

No further information concerning this experiment could be located at this time.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Organic Crystals, Single Crystals, Reactant Solutions, Salt Solution, Membranes, Three-Dimensional Crystalline Structure, Buoyancy-Driven Convection, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Double Diffusion, Liquid/Liquid Interface, Solid/Liquid Interface, Reaction Kinetics, Amorphous Material, Crystal Morphology, Rod Structure, Crystalline Structure, Pressure Compensation, Schlieren Viewing, Contained Fluids, Liquid Reservoir, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design

**Number of Samples:** one
**Sample Materials:** Protein solution: high molecular weight Beta-galactosidase; salt solution: ammoniumsulfate; buffer solution: M/30 Phosphatebuffer
**Container Materials:** unknown

**Experiment/Material Applications:**
See Littke, TEXUS 3b

**References/Applicable Publications:**

(1) Littke, W. and John, C.: Protein Single Crystal Growth Under Microgravity. In Proceedings of a joint ESA-DFVLR Workshop held at Chemisches Laboratorium der Universität, Freiburg, Germany, March 19-20, 1984, pp. 55-64. (post-flight; discusses results from all TEXUS and Spacelab experiments without distinguishing between particular missions.)

(2) Diffusion-Controlled Growth of Single Crystals of Protein. In Summary Review of Sounding Rocket Experiments in Fluid Science and Materials Sciences, TEXUS 1 to 20, MASER 1 and 2, ESA SP-1132, February 1991, pp. 204-205. (post-flight)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #9, STS-009 (STS 41-A, Spacelab 1: Columbia)
**Launch Date/Expt. Date:** November 1983
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Spacelab Rack
**Processing Facility:** Protein Crystal Growth Facility; two Plexiglas™ blocks each with four samples
**Builder of Processing Facility:** Unknown

**Experiment:**
Crystal Growth of Proteins 1ES334

This Spacelab 1 experiment was the fifth in a series of investigations designed by Littke to study the low-gravity growth of protein crystals (see Littke, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5). The specific objectives of this experiment were to investigate (1) crystal growth at a constant temperature (20 °C), (2) crystal growth in an increasing thermal gradient (-4 to 20 °C), (3) the effect of different protein concentrations on crystal growth, and (4) the influence of wide and narrow diffusion fronts on crystal growth.

The Spacelab Protein Crystal Growth Facility was used for the experiment. This apparatus consisted of two Plexiglas™ blocks which contained several protein, buffer, and salt solution chambers. (See Reference (5) for complete description of experimental facility.) The experimental procedure was similar to that of the TEXUS experiments (see for example, Littke, TEXUS 3b). Four experiments involving Beta-galactosidase protein solutions were performed under an imposed thermal gradient. In addition, two experiments involving Beta-galactosidase and two involving lysozyme were performed at a constant temperature (20 °C). To prevent unwanted protease activities, the blocks were maintained at 0 °C until they could be placed in the apparatus under low-gravity conditions.

Post-flight examination of the protein crystals led to the following conclusions:

(1) Prior to experiment initiation, crystallization occurred within the protein solution chamber because of the slow diffusion rate of the protein molecules.

(2) The space-processed Beta-galactosidase crystals were 27 times larger (volume) than their Earth-processed counterparts. The space-processed lysozyme crystals were 1000 times larger than their Earth-processed counterparts.

(3) The crystals were well shaped and of good quality (polarized-light examination).

(4) The formation of large crystals was improved by wide diffusion fronts.

(5) The crystallization of Beta-galactosidase was enhanced by increasing thermal gradients.

(6) Crystals not touching the walls (free floating) indicated perfect habit.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Organic Crystals, Single Crystals, Reactant Solutions, Reaction Kinetics, Salt Solution, Three-Dimensional Crystalline Structure, Buoyancy-Driven Convection, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Double Diffusion, Liquid/Liquid Interface, Isothermal Processing, Thermal Gradient, Solid/Liquid Interface, Crystal Morphology, Crystalline Structure, Free Floating Crystals, Crucible Effects, Contained Fluids, Liquid Reservoir, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design

**Number of Samples:** eight
**Sample Materials:** Proteins: (1) Beta-galactosidase (Beta-D-galactoside galactohydrolase, EC 3.2.1.23, molecular weight = 465000 Daltons) and (2) lysozyme (mucopeptide N-acetylmuramoylhydrolase, EC 3.2.1.17, molecular weight = 14307 Daltons); buffer solution: unknown; salt solution: unknown
**Container Materials:** Plexiglas™

**Experiment/Material Applications:**
The specific reasons why the solutions were selected for the experiment were not detailed in available publications.

**References/Applicable Publications:**

(1) Littke, W. and John, C.: Protein Single Crystal Growth Under Microgravity. In ESA 5th European Symposium On Material Sciences Under Microgravity, Results of Spacelab 1, Schloss Elmau, November 5-7, 1984, ESA SP-222, pp. 185-188. (post-flight; includes TEXUS results)

(2) Littke, W. and John, C.: Protein Single Crystal Growth Under Microgravity. Earth Oriented Applications of Space Technology, Vol. 5, No. 1/2, pp. 63-66, 1985. (post-flight)

(3) Chassay, R. P. and Schwaniger, A.: Low-G Measurements by NASA. In Workshop Proceedings of Measurement and Characterization of the Acceleration Environment On Board the Space Station, August 11-14, 1986, Guntersville, Alabama, pp. 9-1 - 9-48. (acceleration measurements)

(4) Littke, W.: The Growth of Single Crystals from Proteins in Gravity-Free Space. 17th Aerospace Sciences Meeting, New Orleans, January 15-17, 1979. (preflight; experimental apparatus)

(5) Littke, W. and John, C.: Protein Single Crystal Growth Under Microgravity. Journal of Crystal Growth, Vol. 76, 1986, pp. 663-672. (post-flight; appears to be discussing Spacelab 1 results)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt. Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Spacelab Facility, Materials Science Double Rack (MSDR)
**Processing Facility:** Unknown
**Builder of Processing Facility:** Unknown

**Experiment:**
<u>Protein Single Crystal Growth under Zero Gravity (WL-CRY-00)</u>

This Spacelab D1 experiment was the sixth in a series of investigations designed by Littke to study the low-gravity growth of protein crystals (see Littke, TEXUS 3, TEXUS 3b, TEXUS 4, TEXUS 5, Spacelab 1). The specific objective of the experiment was to crystallize a Beta-galactosidase inhibitor complex.

Prior to the shuttle flight, an organic compound containing mercury was synthesized, which acted as a competitive inhibitor for hydrolases. The inhibitor binds itself to the active site of the corresponding enzymes. "In case single crystals of such a... [Beta]-galactosidase inhibitor complex will be obtained, the heavy mercury atoms and thereby the active sites can be localized by x-radiation and their special arrangement determined." (2, p. 127)

The planned, low-gravity experimental procedure included mixing (by infusion) ammonium sulphate and Beta-galactosidase solutions across a narrow buffer zone.

Reportedly, the fully automatic apparatus allowed the experiment to be performed at a constant temperature (20 °C) as well as under an imposed thermal gradient (-4 to 20 °C). However, "...human and technical failure... prevented... [the carrying out of] the scientifically well prepared experiments." (4, p. 294)

No other information concerning this experiment could be located in the published literature.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Organic Crystals, Single Crystals, Reactant Solutions, Reaction Kinetics, Salt Solution, Three-Dimensional Crystalline Structure, Buoyancy-Driven Convection, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Double Diffusion, Concentration Distribution, Liquid Mixing, Liquid/Liquid Interface, Isothermal Processing, Thermal Gradient, Solid/Liquid Interface, Crystal Morphology, Crystalline Structure, Contained Fluids, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design, Processing Difficulties, Hardware Malfunction, Sample Not Processed As Planned

**Number of Samples:** unknown
**Sample Materials:** Protein: Beta-galactosidase and organic compound containing mercury; buffer solution: unknown; salt solution: ammonium sulphate
**Container Materials:** unknown

**Experiment/Material Applications:**
See **Experiment** section (above) and Littke, TEXUS 3b.

**References/Applicable Publications:**


(1) Littke, W. and John, Chr.: Protein-Single Crystal Growth Under Microgravity. In BMFT/DFVLR Scientific Results of the German Spacelab Mission D1, Abstracts of the D1-Symposium, Norderney Germany, August 27-29, 1986, p. 63. (abstract only; post-flight)

(2) Littke, W.: Protein Crystal Growth Under Zero Gravity. In Scientific Goals of the German Spacelab Mission, D1, WPF, 1985, p. 127. (preflight)

(3) Littke, W.: An Experimental Set-Up for Single-Crystal Growth from Solution Under Terrestrial and Microgravity Conditions. In Sixth European Symposium on Material Sciences Under Microgravity Conditions, Bordeaux, France, December 2-5, 1986, ESA SP-256, pp. 95-101. (appears to be an extension of research goals; does not discuss this experiment directly)

(4) Littke, W. and John, Chr.: Protein-Single Crystal Growth Under Microgravity. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 292-294. (post-flight)

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**Experiment Origin:** USA
**Mission:** STS Launch #10, STS-011 (STS 41-B, Challenger)
**Launch Date/Expt. Date:** February 1984
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** NASA Get Away Special (GAS) Canister G-008
  Volume of Canister: 2.5 cubic feet
  Location of Canister: STS Payload Bay
**Primary Developer/Sponsor of this experiment within G-008:** University of Utah, Salt Lake City, Utah
**Processing Facility:** Crystallization chamber (temperature control achieved via the use of two containers of eutectic salts)
**Builder of Processing Facility:** (1) Experiment enclosure: Megill, R. et al., Utah State University, Logan, Utah
Experiment apparatus: Gerpheide, G. et al., University of Utah, Salt Lake City, Utah

**Experiment:**
Crystallization of Proteins

This experiment was one of four investigations housed within the G-008 Get Away Special canister during STS-011. Two other experiments (of the four) were applicable to this data base (see Alford, STS-011 (Chapter 18); Walden, STS-011 (Chapter 18)). The specific objective of this experiment was to determine the effect of low-gravity on the crystallization of proteins from aqueous solutions.

Three proteins were chosen for the experiment: (1) one which successfully crystallizes under normal (Earth) gravity conditions, (2) one which crystallizes poorly under normal gravity conditions, and (3) one which does not crystallize under normal gravity conditions (as of the experiment date).

<Note: Very little information could be located which described the experimental setup and inflight procedure. The following short summary represents nearly all of the experiment information available at this time.>

The reduced gravity protein crystal growth was to be achieved in a crystallization chamber. Two containers of eutectic salt solutions were configured in the payload to insure that crystallization took place in a constant temperature environment. (It was expected that the significant thermal mass of the salt solutions would maintain the desired protein temperature range of the crystallization chamber and prevent decomposition or freezing of the aqueous protein solutions.) <Note: The desired temperature range of the chamber was not detailed.>

During the STS mission, crystallization was to be initiated on orbit via a pump-driven fluid exchange. <Note: Further details describing this "pump driven fluid exchange" could not be located.>

It was reported that the experiment was not activated. "A battery pack latching relay stuck closed..., resulting in battery drain before launch." (6, p. 29) Thus, the canister was not turned on in space and no results were obtained.

No further information could be located which described (1) the faulty latching relay, (2) the experimental setup, or (3) the expected inflight procedure.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Aqueous Solutions, Thermal Control, Isothermal Processing, Diffusion, Diffusive Mass Transfer, Solid/Liquid Interface, Liquid/Liquid Interface, Liquid/Liquid Diffusion, Liquid Transfer, Decomposition, Freezing, Crystalline Structure, Crystal Morphology, Salt Solution, Contained Fluids, Liquid Reservoir, Biotechnology, Biological Structures, Pharmaceutical Applications, Sample Not Processed As Planned, Battery Drain

**Number of Samples:** three
**Sample Materials:** Three different proteins in aqueous solution(s) were employed. <Note: The specific proteins were not identified.> One of the proteins successfully crystallizes Earth, one of the proteins poorly crystallizes on Earth, and one of the proteins does not crystallize on Earth.
**Container Materials:** glass

**Experiment/Material Applications:**
It was expected that this experiment would demonstrate improved crystallization (over Earth crystallization) in the low-gravity environment. The complex protein molecular structures of these superior crystals could then be better analyzed using X-ray crystallography techniques.

**References/Applicable Publications:**


(1) Cargo Systems Manual: GAS Annex for STS-11, JSC-17645 Annex STS-11, December 2, 1983. (very short description; preflight)

(2) Input received from Principal Investigator, G. Gerpheide, August 1988.

(3) Getaway Special Payloads (STS-11). In Goddard Space Flight Center's Engineering Newsletter, Vol. 2, No. 3, April 1984, published by the Engineering Directorate, pp. 8-9. (very short description)

(4) STS-11 Getaway Special Payload Descriptions, NASA News, NASA GSFC, 1984. (post-flight)

(5) STS 41-B Tenth Space Shuttle Mission, NASA Press Kit, February 1984, p. 28. (brief mention of experiment; preflight)

(6) Ridenoure, R.: GAS Mission Summary and Technical Reference Data Base. Ecliptic Astronautics Co., Technical Report #EAC-TR-RWR87-11, October 2, 1987. (Get Away Special canister mission history)

(7) STS-11 GAS Payloads. NASA Goddard Space Flight Center Engineering Newsletter, April 1984.


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**Experiment Origin:** USA
**Mission:** STS Launch #12, STS-014 (STS 41-D, Discovery)
**Launch Date/Expt. Date:** August 1984
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Middeck Experiment
**Processing Facility:** Liquid-liquid diffusion apparatus
**Builder of Processing Facility:** McDonnell Douglas Astronautics Company, St. Louis Missouri

**Experiment:**
Protein Liquid Diffusion Experiment

This STS-014 experiment, which was performed by McDonnell Douglas Astronautics Company and supported by the Scripps Institute of LaJolla, California, was designed to better understand techniques of protein crystal growth in low gravity.

"The experiment was conducted in three diffusion chambers. Each of these chambers was configured with two 12cc syringes (one partially-filled with the precipitating agent, the other with the lysozyme protein) separated by a three-way valve. A fourth single syringe chamber contained lysozyme crystals grown on Earth, sealed and carried as a control. On the second day of [the STS] flight the two chambers in each pair were opened to each other. Mixing was induced by manually infusing the 5cc contents of one syringe into the other thus inducing precipitation and crystallization of the protein. The syringes were then isolated from one another again by the valve.

"During the manual infusion step, it was found that the syringe pistons were very balky in action. This was thought to be due to a film of solid precipitate between the baffles and the pistons and possibly within the valves as well. The consequent jerky action induced the formation of many small bubbles then carried into the crystallization chamber with the infused solution. They remained there throughout the experiment.

"Upon visual examination of the syringes in orbit near the end of the mission, no crystals could definitively be seen by the unaided eye. Although along with the bubbles in each of the experimental chambers the solution appeared to be slightly opaque. Ground analysis of the contents by the Scripps Institute revealed that only myriad small crystals had formed, on the order of 0.5 mm. It was hypothesized that the bubbles provided numerous nucleation sites thus increasing the number of crystals forming simultaneously and resulting in the small size of each.

"The experiment was deemed to be unsuccessful due to the mechanical problem that prevented the slow mixing desired and induced excessive bubble nucleation sites." (2)

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Reaction Kinetics, Precipitation, Diffusion, Diffusive Mass Transfer, Liquid/Liquid Diffusion, Liquid/Liquid Interface, Solid/Liquid Interface, Liquid Mixing, Liquid Transfer, Liquid Expulsion Through a Small Orifice, Bubbles, Bubble Formation, Nucleation, Nucleation Sites, Crystalline Structure, Biotechnology, Piston System, Baffles, Contained Fluids, Liquid Reservoir, Three-Dimensional Crystalline Structure, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design, Hardware Malfunction

**Number of Samples:** four
**Sample Materials:** Proteins and precipitating agent solutions, specifically: lysozyme, monosodium aspartate, and sodium nitrate solutions.
**Container Materials:** glass and latex

**Experiment/Material Applications:**
Crystallization of proteins in the low-gravity environment could allow the production of larger and higher quality crystals. If such crystals could be produced, resolution of the proteins' three-dimensional atomic structure might be possible using high resolution X-ray diffraction. "Protein crystallography is the only technique available for elucidating the atomic arrangements within complicated biological molecules." (1, p. 1)

**References/Applicable Publications:**

(1) Bugg, C. E. and Clifford, D. W.: Protein Crystal Growth in Space. AIAA 25th Aerospace Sciences Meeting, January 13-15, 1986, Reno, Nevada, McDonnell Douglas Astronautics Company, St. Louis Division, 10 pp. (post-flight)

(2) Input received from Principal Investigator C. Walker, August 1993.

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**Experiment Origin:** USA
**Mission:** STS Launch #16, STS-023 (STS 51-D, Discovery)
**Launch Date/Expt. Date:** April 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Middeck Experiment
**Processing Facility:** (1) Two handheld Vapor Diffusion Apparatuses (VDAs). Each VDA housed 17 reaction chambers into which precipitant solutions were extruded from hand-operated syringes. The VDAs were each fashioned from an aluminum block; Lexan™ windows allowed visualization of the chambers. (2) One Dialysis Apparatus (DA): two-chambered Lucite™ block
**Builder of Processing Facility:** VDAs and DA: Campbell Engineering, Huntsville, Alabama

**Experiment:**
<u>Human Protein Crystal Growth (PCG) Experiment HPCG1 (Vapor Diffusion and Dialysis Techniques for Space Experiments)</u>

"One of the most widely used methods of crystallizing proteins involves the slow precipitation of protein from droplets of solution by vapor pressure equilibration against a solution containing a higher concentration of the precipitating agent. The "hanging-drop method" is a common version of this general technique. Most protein crystallography laboratories have extensive experience with vapor-diffusion crystallization methods, and a large percentage of the protein crystals described in recent publications have been obtained by these methods." (3, p. 683)

"A second micro-crystallization technique that is widely used in protein crystallography laboratories is dialysis, by use of microdialysis cells. These cells contain small chambers with volumes typically in the 5-50 μl range, which hold the protein solutions.... [T]he chambers are covered with semi-permeable membranes that retain macromolecules, but allow passage of salts and other low molecular weight substances. These dialysis cells can be used to grow protein crystals by placing them into a solution that contains a low molecular weight precipitating agent." (3, p. 683)

This space shuttle experiment was the first in a series of investigations designed by Bugg et al. to study the growth of protein crystals. The overall objectives of the investigations were to (1) evaluate the effects of reduced gravity on several protein crystalline systems and (2) develop methods and hardware for optimizing crystal growth in the space environment.

While terrestrial grown protein crystals are flawed and often too small for sufficient structural analysis, similar crystals grown in space are expected to be (1) larger and of higher quality than terrestrial grown crystals and (2) of suitable size and quality for three-dimensional structural determination.

During the STS-023 mission, two methods for growing protein crystals were examined: dialysis and vapor diffusion.

## Dialysis Experiments

The dialysis experiments were performed in a single, two-chambered Lucite™ block. It appears that each chamber contained (1) a protein solution, (2) a semi-permeable membrane, (3) a glass vial (filled with the precipitating solution) and (4) a metal striker. <Note: The spatial arrangement of each of these four items within the chambers was unclear to the editors and was not detailed in the available publications. Further, the solutions employed were not detailed in the available publications.>

It appears (Reference (8)) that the experiment was activated prior to launch when the hand-driven striker broke the glass vial within each of the chambers. <Note: It is not clear if this prelaunch activation was intentional.> This action released the precipitate toward the membrane, thus initiating the dialysis. <Note: The experiment duration time was not detailed in the available publications.>

Post-flight analysis of the dialysis chambers revealed that amorphous precipitates, rather than the expected crystals, were produced. <Note: No further information concerning these dialysis experiments could be located at this time.>

## Vapor Diffusion Experiments

The "hanging drop method" vapor-diffusion experiments were performed in two Vapor Diffusion Apparatuses (VDAs). Each of these VDAs contained 17 similar crystal growth chambers. Configured just above each growth chamber was a polypropylene syringe, the tip of which extended into the growth chamber. Configured at the bottom of each chamber was a cotton wick (spaced opposite the syringe tip).

Prior to the initiation of the experiments, each syringe was filled with the appropriate protein sample, and each wick was saturated with the appropriate precipitating agent (reservoir solution). (A listing of the employed proteins can be found in the **Sample Materials** section below.)

During the mission, a protein drop was extruded from each syringe into the growth chamber using a hand-deployed Teflon™ piston. The resulting drop (some with sizes up to 80 μl) (1) was maintained on either a flared or unflared syringe tip and (2) was left to equilibrate with the reservoir solution. (The drops did not touch the wick.) Droplet stability and crystal growth were monitored through plastic windows enclosing the chambers.

Drop stability on the syringe tips was noted. Reportedly, (1) many drops were lost during the mission (especially on non-flared syringe tips) during drop deployment, equipment storage, etc. and (2) all surviving drops were maintained on flared syringe tips.

<Note: It appears that when the experiment time was exhausted (typically 3-4 days), newly grown crystals floating in the drop and the surrounding protein solutions were withdrawn into the syringes. It was noted (Reference (8)) that the syringes were not then stoppered with syringe tip plugs to insure the liquid remained in the syringe. (Such tip plugs were employed during subsequent experiments by Bugg et al. (e.g., STS-026).)>

Post-flight analysis of the contents of the VDAs indicated that (1) nearly all of the liquid from the syringes was lost during the shuttle landing and (2) protein crystals had formed during the mission in some of the deployed drops. Specifically, it was determined that one large lysozyme crystal (measuring 1.6 X 1.6 X 1.22 mm) had grown from an 80 μl droplet. "The quality of this crystal was assessed via X-ray methods and was found to be equal to the best observed for earth-grown crystals of lysozyme. We have been unable to grow a crystal this large on earth under identical conditions. Furthermore it is interesting that all of the control experiments performed on earth have produced several lysozyme crystals within each protein drop as opposed to the single crystal that was obtained in the microgravity environment." (6, p. 67) In addition, small crystals of bacterial PNP, green fluorescent protein toxin V, 13 MER DNA, 133 MER DNA and Neuramindase-FAB complex were also formed. <Note: Further description of these crystals was not provided in the available publications.>

Additional information concerning the results found in each of the 34 crystal growth chambers could not be located at this time.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Single Crystals, Drops, Drop Formation, Droplet Creation Via a Syringe, Drop Stability, Hanging Drop Method, Droplet Size, Piston System, Vapor Diffusion Method, Dialysis, Semipermeable Membranes, Membranes, Precipitation, Liquid/Liquid Diffusion, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Solutal Gradients, Convection, Sedimentation, Liquid/Liquid Interface, Solid/Liquid Interface, Liquid/Vapor Interface, Free Floating Crystals, Contained Fluids, Liquid Reservoir, Containerless Processing, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Antibodies, Structure Based Drug Design, Three-Dimensional Crystalline Structure, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Liquid Leakage, Acceleration Effects, Vehicle Re-Entry Forces/Vibration, Deterioration of Samples After Low-G Flight

**Number of Samples:** Dialysis: two; vapor diffusion: thirty-four
**Sample Materials:** Proteins, Human Purine Nucleoside Phosphorylase-PNP, Bacterial Purine Nucleoside Phosphorylase-PNP, alpha2-Interferon, Human C-Reactive Protein-CRP, Fab Monoclonal Antibody Component, Lysozyme, Scorpion Toxin, Sea Anemone Toxin, Wisteria Lectin, Monoclonal Antibody Complexes of Neuramindase from Influenza Virus, Green Fluorescent Protein, Human Serum Transferrin, Synthetic DNA Fragment, Human Nuclear t-RNA Gene
**Container Materials:** DA: Lucite™ block, glass vials; VDA-syringes: polypropylene. The VDA reaction chambers appear to have been comprised of aluminum with Lexan™ windows.

**Experiment/Material Applications:**
High quality protein crystals are needed for (1) basic studies of biological structures, (2) protein crystallography pharmaceutical agent design, and (3) protein engineering efforts. It is expected that low-gravity protein crystal processing will (1)

result in the elimination of crystal sedimentation, (2) lead to the reduction of density-dependent flows, and (3) permit containerless, high quality crystal production.

"In order to evaluate the possible effects of microgravity on protein crystal growth, it will be necessary to investigate a range of different types of proteins that have been well-characterized in ground-based studies. Many of the proteins that are being extensively studied in crystallography laboratories around the world are only available in milligram quantities. Vapor diffusion techniques are particularly suitable for crystallization experiments involving small quantities of protein and can be used for most types of biological materials. Therefore, we focused our initial experiments on development of a space version of the popular hanging-drop method." (6, p. 683)

The specific reasons why each of the chosen proteins were selected were not detailed in any of the publications specifically describing this experiment. However, some of the later experiments by Bugg et al. did detail the uses of several of these proteins (see for example, Bugg, STS-32 (this chapter)).

**References/Applicable Publications:**

(1) Presentation by Blair Herren, NASA Marshall Space Flight Center, Protein Crystal Growth (PCG) Office of Commercial Programs, March 1987. (view graphs only, post-flight)

(2) Bugg, C. E.: The Future of Protein Crystal Growth. Journal of Crystal Growth 76(1986), pp. 535-544.

(3) DeLucas, L. J., Suddath, F. L., Snyder, R., Naumann, R., Broom, B., Pusey, M., Yost, V., Herren, B., Carter, D., Nelson, B., Meehan, E. J., McPherson, A., and Bugg, C. E.: Preliminary Investigations of Protein Crystal Growth Using the Space Shuttle. Journal of Crystal Growth, 76(1986), pp. 681-693. (post-flight)

(4) Bugg, C. E. and Clifford, D. W.: Protein Crystal Growth in Space. AIAA 25th Aerospace Sciences Meeting, January 13-15, 1986, Reno, Nevada, 10 pp. (post-flight)

(5) Hermann, F. T.: Advanced Protein Crystal Growth Flight Hardware for the Space Station. AIAA Aerospace Sciences Meeting, January 11-14, 1988, Reno, Nevada, AIAA #88-0345.

(6) DeLucas, L. J., Suddath, F. L., Snyder, R., Naumann, R., Broom, M. B., Pusey, M., Yost, V., Herren, B., Carter, D., Nelson, B., Meehan, E. J., McPherson A., Bugg, C. E.: Protein Crystal Growth in Microgravity. Space: Biomedicine and Biotechnology Workshop, Toronto, Canada, May 1986, pp. 65-71. (post-flight; little 51-D information)

(7) Personal communication with Mark Pusey, NASA Marshall Space Flight Center, 1992.

(8) Information describing mission by mission PCG results. On display at NASA Marshall Space Flight Center, Huntsville, Alabama, 1991.

(9) Bugg, C.: Protein Crystal Growth Results from Shuttle Flight 51-F. Final Report, The University of Alabama Comprehensive Cancer Center, NASA CR-178540, October 1985. (post-flight; comparisons are made to 51-D results)

(10) Naumann, R. J.: Microgravity Science and Applications. In: In Space '87, Japan Space Utilization Promotion Center (JSUP), pp. 19-21. (post-flight)

(11) Input received from Principal Investigator C. E. Bugg, September 1988.

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**Experiment Origin:** USA
**Mission:** STS Launch #19, STS-026 (STS 51-F, Spacelab 2: Challenger)
**Launch Date/Expt. Date:** July 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Middeck Experiment
**Processing Facility:** (1) Two handheld 17-chamber Vapor Diffusion Apparatuses (VDAs) modified from STS 51-D to include syringe tip plugs and stainless steel pistons
(2) One Dialysis Apparatus (DA) modified for activation in orbit using a metal striker
**Builder of Processing Facility:** VDAs and DA: Campbell Engineering, Huntsville, Alabama

**Experiment:**
Human Protein Crystal Growth Experiment HPCG2 (Vapor Diffusion and Dialysis Techniques for Space Experiments)

This space shuttle experiment was the second in a series of investigations designed by Bugg et al. to study the growth of protein crystals (see Bugg, STS-023). The overall objectives of the investigations were to (1) evaluate the effects of reduced gravity on several protein crystalline systems and (2) develop hardware for optimizing crystal growth in the space environment.

Two methods for growing protein crystals were examined during the mission: dialysis and vapor diffusion.

Dialysis Experiments

The dialysis experiments were performed in a single, two-chambered Lucite™ block. Each chamber contained (1) three wells (or dialysis cells) filled with lysozyme, (2) a glass vial filled with the precipitating solution (sodium chloride), and (3) a metal striker. <Note: The spatial arrangement of each of these

three items within the chambers was unclear to the editors and was not detailed in any of the available publications.> Semi-permeable membranes (or dialysis buttons) were placed over the six lysozyme-filled wells.

One hour and 25 minutes into the STS-023 mission, the metal striker within each chamber was used to break the glass vials releasing the precipitate (and thus initiating the dialysis). <Note: The total dialysis experiment time was not detailed in the available publications.>

Post-flight analysis of the growth chambers indicated that three of the six dialysis cell membranes had ruptured, apparently during the activation sequence. "The three surviving cells contained large tetragonal crystals of Lysozyme with average dimensions of 1.3 X 0.65 X 0.65 mm." (1, p. 6) It was reported that these crystals were significantly larger than those obtained during ground-based testing performed in the space hardware.

It was concluded that alternative activation methods should be sought for the dialysis experiments.

Vapor Diffusion Experiments

The "hanging drop method" vapor diffusion experiments were performed in two Vapor Diffusion Apparatuses (VDAs). Each of these VDAs contained 17 similar growth chambers. (The VDAs had been modified significantly since the earlier 51-D flight to increase drop stability, address leakage difficulties, etc.)

Configured just above each growth chamber was a glass syringe, the tip of which extended into the growth chamber. These syringes had either a blunt glass tip or a bell-shaped glass tip. Teflon™ tape was placed around several of the tips (producing a hydrophobic barrier). Syringe tip plugs were employed to contain protein solutions within the syringes prior to experiment initiation. <Note: A listing of the employed proteins can be found in the **Sample Materials** section below.>

Configured at the bottom of each chamber was a cotton wick (spaced opposite the syringe tip). Contrary to the earlier 51-D experiments, "...the reservoir solutions [precipitating agents] were added to the wicks prior to launch and were separated from the protein syringes by a metal grid." (1, p. 4)

During the flight experiment, the syringe plugs were retracted to a position opposite the syringe tip and a protein drop was extruded from the syringe into the growth chamber using a hand-deployed Teflon™ or stainless steel piston. (O-ring seals were integrated with the steel pistons to prevent leakage.) Reportedly, drops were maintained either (1) on the blunt glass tips, (2) in the bell-shaped tips, or (3) tethered to the syringe tip plugs. The drop was left to equilibrate with the reservoir solution during the remainder of the experiment. (Reportedly, in each VDA, two drops (of the 17) were tethered to the oppositely retracted syringe plugs and consequently stabilized.)

Hand-operated drop deployment in the first VDA took place early in the mission (when the VDA was not securely mounted) to assess the effects of the OMS rocket burns on drop stability. Four droplets were lost during deployment. It was noted that none of these droplets had been lost from syringe tips that had been treated with Teflon™ tape. (The tape had been applied to the tips in an effort to enhance drop stability.) Later, three drops were lost during the shuttle flight in this VDA, although it was not known if they were lost because of an OMS burn.

Hand-operated drop deployment of the second VDA took place later during the mission when the VDA was securely mounted. Several drops were successfully deployed. After deployment, only one drop was lost during flight. It was noted that in the second VDA, all the drops were still intact after a major OMS burn.

Near the end of the mission, most of the drops and crystals were retracted into the syringes and sealed with the syringe tip plugs. Some syringes were not plugged because crystalline structures remained outside the syringe even after the drops had been withdrawn.

Post-flight analysis indicated that none of the crystals (plugged or unplugged) had been lost during landing. It was surmised, however, that the crystals underwent a large temperature change (post-flight) because of extreme heat on the shuttle runway. Reportedly, this high temperature environment may have caused some crystal dissolution.

A total of eight crystals were formed in the two VDAs, all of which were small and of poor quality. <Note: Further details of these eight crystals were not presented.> It was concluded that drop stability had increased over Bugg's earlier 51-D experiments, and drop tethering was seen as highly successful.

Additional details of the inflight experimental procedure, post-flight conclusions, etc. can be found in Reference (6).

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Salt Solution, Single Crystals, Drops, Drop Formation, Droplet Creation Via a Syringe, Drop Stability, Hanging Drop Method, Piston System, Vapor Diffusion Method, Dialysis, Semipermeable Membranes, Membranes, Precipitation, Solutal Gradients, Liquid/Liquid Diffusion, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Convection, Sedimentation, Liquid/Liquid Interface, Solid/Liquid Interface, Liquid/Vapor Interface, Contained Fluids, Liquid Reservoir, Biotechnology, Biological Materials Processing, Containerless Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design, Three-Dimensional Crystalline Structure, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Coated Surfaces, Hydrophobic Surfaces, Liquid Leakage, Acceleration Effects, Thermal Environment More Extreme Than Predicted, Dissolution, Deterioration of Samples After Low-G Flight

**Number of Samples:** Dialysis: six; vapor diffusion: thirty-four
**Sample Materials:** Proteins, in the VDA units: lysozyme crystallized from sodium-chloride solutions, bacterial purine nucleoside phosphorylase-PNP crystallized from ammonium sulfate salt solutions, alpha-2 interferon crystallized from polyethylene glycol, and human C-reactive protein-CRP crystallized from methylpentanediol. DA: lysozyme, sodium-chloride.
**Container Materials:** VDA syringes: glass; dialysis apparatus-Lucite™ block; glass vial

**Experiment/Material Applications:**
See Bugg, STS-023.

The above proteins were selected because (1) the Principal Investigator and other investigators at the University of Alabama in Birmingham had considerable experience crystallizing the proteins and (2) "They permitted us to assess drop stability for solutions that contained different types of precipitating agents. Bacterial PNP is crystallized from ammonium sulfate salt solutions; C-reactive protein is crystallized from methylpentanediol; lysozyme is crystallized from NaCl solutions; and a2 interferon is crystallized from polyethylene glycol. Lysozyme usually crystallizes easily, so it serves as a useful control. The other three proteins generally crystallize within two days, but crystals of these proteins obtained in our laboratory at U.A.B. are small, and diffract only to moderate resolutions." (1, p. 3)

**References/Applicable Publications:**

(1) Bugg, C.: Protein Crystal Growth Results from Shuttle Flight 51-F. Final Report, The University of Alabama at Birmingham Comprehensive Cancer Center, NASA CR-178540, October, 1985. (post-flight)

(2) Bugg, C. E.: The Future of Protein Crystal Growth. Journal of Crystal Growth, 76 (1986), pp. 535-544.

(3) DeLucas, L. J., Suddath, F. L., Snyder, R., Naumann, R., Broom, B., Pusey, M., Yost, V., Herren, B., Carter, D., Nelson, B., Meehan, E. J., McPherson, A., and Bugg, C. E.: Preliminary Investigations of Protein Crystal Growth Using the Space Shuttle. Journal of Crystal Growth, 76 (1986), pp. 681-693.

(4) Bugg, C. E. and Clifford, D. W.: Protein Crystal Growth in Space. AIAA 25th Aerospace Sciences Meeting, January 13-15, 1986, Reno, Nevada, 10 pp. (post-flight)

(5) Hermann, F. T.: Advanced Protein Crystal Growth Flight Hardware for the Space Station. AIAA Aerospace Sciences Meeting, January 11-14, 1988, Reno, Nevada, AIAA #88-0345.

(6) Protein Crystal Growth Results from Space Shuttle Flight 51-F. Memorandum from Charlie Bugg (University of Alabama, Birmingham) to Mr. Vaughn Yost (George C. Marshall Space Flight Center, Huntsville, Alabama) September 11, 1985, 7 pp. (post-flight)

(7) Information describing mission by mission PCG results. On display at NASA Marshall Space Flight Center, Huntsville, Alabama.

(8) Presentation by Blair Herren, NASA Marshall Space Flight Center, Protein Crystal Growth, Office of Commercial Programs, March 1987. (viewgraphs only; post-flight)

(9) Naumann, R. J.: Microgravity Science and Applications, In: In Space '87, Japan Space Utilization Promotion Center (JSUP), pp. 19-21. (post-flight)

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**Experiment Origin:** USA
**Mission:** STS Launch #23, STS-031 (STS 61-B, Atlantis)
**Launch Date/Expt. Date:** November 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Middeck Experiment
**Processing Facility:** (1) Two handheld Vapor Diffusion Apparatuses (VDAs) each modified (post STS-026) to include 24 growth chambers, a new reservoir wicking system, etc.
(2) One Dialysis Apparatus (DA) containing two chambers. A metal striker was employed to break a vial containing the precipitating agent.
**Builder of Processing Facility:** The VDAs and DA: Campbell Engineering, Huntsville, Alabama

**Experiment:**
Human Protein Crystal Growth Experiment HPCG3 (Vapor Diffusion and Dialysis Techniques for Space Experiments)

This space shuttle experiment was the third in a series of investigations designed by Bugg et al. to study the growth of protein crystals (see Bugg, STS-023, STS-026). The overall objectives of the investigation were to (1) evaluate the effects of reduced gravity on several protein crystalline systems and (2) develop hardware for optimizing crystal growth in the space environment.

Two methods for growing protein crystals were examined during this mission: dialysis and vapor diffusion.

Dialysis Experiments

The dialysis experiments were performed in a single, two-chambered Lucite™ block. One of the chambers contained three 50 microliter dialysis cells filled with alpha2-interferon; the other chamber contained (1) two dialysis cells filled with canavalin and (2) one cell filled with concanavalin B. Each chamber also contained (1) a glass vial containing an "acidic solution," and (2) a metal striker. <Note: The spatial arrangement of the items within the chambers was unclear to the editors and was not detailed in the available publications.>

During the mission, the Lucite™ block was shaken such that the metal striker in each chamber would break the vial containing the precipitating agent (thus initiating the dialysis).

Reportedly, during the flight, "Efforts were made to activate the dialysis unit, but only one of the dialysis vials [that vial associated with the alpha2-interferon dialysis cells] was successfully broken." (1, p. 2) Analysis of the contents of alpha2-interferon cells indicated that only a light precipitate resulted.

It was concluded that a more reliable technique for initiating the dialysis should be conceived.

Vapor Diffusion Experiments

The "hanging drop method" vapor diffusion experiments were performed in two Vapor Diffusion Apparatuses (VDAs). Each of these VDAs contained 24 similar growth chambers. (The VDAs had been slightly redesigned after Bugg's STS-026 experiment to enhance experimental success.)

Configured just above each growth chamber was either a small or large glass syringe, the tip of which extended into the growth chamber. A Teflon™ coating was added to some of the tips in an effort to reduce glass tip nucleation sites. <Note: The shape of syringe tip was not detailed.> Syringe tip plugs were employed to contain protein solutions within the syringes prior to experiment initiation. (A listing of the employed proteins can be found in the Sample Materials section below.)

Configured at the bottom of each chamber was a cellulose wick (spaced opposite the syringe tip) saturated with an appropriate precipitating agent. <Note: It is not clear at what point the precipitating agent was added to the wick (prior to, or after launch).>

Approximately 9 hours into the mission, the experiments were initiated. The syringe tip plugs were retracted to a position opposite the syringe tip and a protein drop was extruded from the syringe into the growth chamber using either a stainless steel piston or a Micromedics Teflon™ piston. "One of the... [VDAs] contained 30 $\mu l$ droplets and the other apparatus contained 60 $\mu l$ droplets (a few exceptions were made in cases where limited quantities of protein solutions were available for the experiments)." (1, p. 1) Reportedly, drops were maintained (1) on the glass tips or (2) tethered to the the syringe tip plugs. Reportedly, "...three seeding experiments were attempted [in each of the VDAs]. These involved introducing seed crystals into the protein solutions after they were extruded from the syringes. Two of the protein droplets in the seeding experiments were tethered, using the opposing plungers in the vapor diffusion apparatuses. The third seeding sample was an untethered droplet. The seed crystals were introduced through screw holes.... The remaining 21 vapor diffusion experiments in each of the... [VDAs]... were of the standard type, where a droplet of protein was extruded from the syringe into the vapor diffusion chamber." (1, p. 1) All of the deployed drops were left to equilibrate with the reservoir solutions (precipitating agents). (Inflight observations of each of the 48 chambers are described in Reference (1).)

Droplet equilibrium and stability during the mission was examined. It was reported that (1) the 60 $\mu l$ droplets did not completely equilibrate, and (2) stability greatly improved (over Bugg's previous STS experiments) as only four of the drops were lost during the experiments (none of which were on Teflon™-coated tips).

Approximately 6 days after experiment initiation, the VDAs were deactivated by withdrawing the liquid/crystals into the syringes and sealing the tips with the syringe plugs.

Post-flight analysis of each of the 48 chambers is briefly described in Reference (1). Overall it was noted that the experiments were conducted at an elevated temperature, probably too hot to result in optimum crystals. "However, good crystals, which are suitable for X-ray diffraction studies, were obtained of C-reactive protein, bacterial purine nucleoside phosphorylase, and lysozyme. Diffraction analyses of C-reactive protein and of bacterial purine nucleoside phosphorylase indicated that the crystals diffracted as well as the best earth-grown crystals." (1, p. 12) It was also noted that usable crystals were grown from 30 microliter seeded droplets.

Additional information concerning this experiment can be found in Reference (1).

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Drops, Drop Formation, Droplet Creation Via a Syringe, Droplet Size, Piston System, Drop Stability, Hanging Drop Method, Vapor Diffusion Method, Dialysis, Precipitation, Solutal Gradients, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Convection, Sedimentation, Seed Crystals, Liquid/Liquid Interface, Liquid/Liquid Diffusion, Solid/Liquid Interface, Liquid/Vapor Interface, Nucleation, Nucleation Sites, Biotechnology, Containerless Processing, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design, Three-Dimensional Crystalline Structure, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Coated Surfaces, Contained Fluids, Liquid Reservoir, Thermal Environment More Extreme Than Predicted, Processing Difficulties

**Number of Samples:** Dialysis: six; vapor diffusion: forty-eight
**Sample Materials:** In the VDA units: human C-reactive protein (6 seeded, 8 non-seeded samples), human purine nucleoside phosphorylase as a 6-idopurine complex (10 samples), lysozyme (4 samples), alpha2-interferon (10 samples), canavalin (6 samples), concanavalin B (4 samples). Dialysis: alpha2-interferon, canavalin, concanavalin B. Both chambers were to be crystallized by adding an acidic solution.
**Container Materials:** VDA syringes: glass; Diffusion apparatus: Lucite™ block/glass vials

**Experiment/Material Applications:**
See Bugg, STS-023 and Bugg, STS-026.

The specific reasons why each of these proteins were selected was not detailed in the available publications.

**References/Applicable Publications:**
(1) Postflight Analysis of Protein Crystal Growth Experiments Performed on STS-61-B. Memorandum from Charlie Bugg (Univ. of Alabama at Birmingham) to Mr. Vaughn Yost (Marshall Space Flight Center, Huntsville, Alabama), January 17, 1986, 14 pp. (post-flight)

(2) Bugg, C. E.: The Future of Protein Crystal Growth. Journal of Crystal Growth, 76 (1986), pp. 535-544.

(3) DeLucas, L. J., Suddath, F. L., Snyder, R., Naumann, R., Broom, B., Pusey, M., Yost, V., Herren, B., Carter, D., Nelson, B., Meehan, E. J., McPherson, A., and Bugg, C. E.: Preliminary Investigations of Protein Crystal Growth Using the Space Shuttle. Journal of Crystal Growth, 76 (1986), pp. 681-693. (post-flight)

(4) Bugg, C. E. and Clifford, D. W.: Protein Crystal Growth in Space. AIAA 25th Aerospace Sciences Meeting, January 13-15, 1986, Reno, Nevada, 10 pp. (post-flight)

(5) Hermann, F. T.: Advanced Protein Crystal Growth Flight Hardware for the Space Station. AIAA Aerospace Sciences Meeting, January 11-14, 1988, Reno, Nevada, AIAA 88-0345.

(6) Information describing mission by mission PCG Results. On display at NASA Marshall Space Flight Center, Huntsville, Alabama. (post-flight)

(7) Presentation by Blair Herren, NASA Marshall Space Flight Center, Protein Crystal Growth, Office of Commercial Programs, March 1987. (viewgraphs only; post-flight)

(8) Naumann, R. J.: Microgravity Science and Applications. In: In Space '87, Japan Space Utilization Promotion Center (JSUP), pp. 19-21. (post-fLight)

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**Experiment Origin:** USA
**Mission:** STS Launch #24, STS-032 (STS 61-C, Columbia)
**Launch Date/Expt. Date:** January 1986
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Middeck Experiment, Performed by Congressman Bill Nelson
**Processing Facility:** (1) Two modified 24-chambered, handheld Vapor Diffusion Apparatuses (VDA-1,2)
(2) Two Dialysis Apparatuses (DAs) identically prepared, employing a new thin-walled vial for the precipitating agent. A metal striker broke the vial.
**Builder of Processing Facility:** VDAs and DAs: Campbell Engineering, Huntsville, Alabama

**Experiment:**
<u>Human Protein CrystalGrowth Experiment HPCG4 (Vapor Diffusion and Dialysis Techniques for Space Experiments)</u>

This space shuttle experiment was the fourth in a series of investigations designed by Bugg et al. to study the growth of protein crystals (see Bugg, STS-023, STS-026, STS-031). The overall objectives of the investigations were to (1) evaluate the effects of reduced gravity on several protein crystalline systems and (2) develop hardware for optimizing growth in the space environment.

Two methods for growing protein crystals were examined during this mission: dialysis and vapor diffusion.

Dialysis Experiments

The dialysis experiments were performed in two, double-chambered Dialysis Apparatuses (DAs). The two DAs were similarly prepared: (1) In the first chamber, three dialysis cells were filled with canavalin; (2) In the second chamber, two dialysis cells were filled with concanavalin B. Semi-permeable membranes were placed over all of the cells. Each chamber also contained (1) a newly designed vial containing the precipitating solution, and (2) a metal striker. (The end of the vial was specially designed to break when it came in contact with the striker). <Note: The spatial arrangement of these items within the chambers is unclear to the editors and was not detailed in any of the available publications.>

The dialysis experiments were initiated approximately 3 hours into the flight. <Note: The experiment duration time was not detailed in the available publications although it was reported that this shuttle mission was prematurely shortened and that protein crystallization times were shorter than originally expected.>

Reportedly, it was difficult to tell if the vials had indeed broken upon activation. Post-flight examination indicated that in each DA, two out of three of the Canavalin dialysis cell membranes were torn, apparently by glass from the broken vial. In the two other Canavalin cells, a number of large crystals and/or microcrystals were grown. The largest Canavalin crystal was 1.05 X 0.5 X 0.25 mm. Most of these Canavalin crystals were of poor quality. Reportedly, it was interesting that the majority of the crystals did not grow on the dialysis cell wall or membrane.

Concanavalin B dialysis cells in one of the DAs indicated a heavy precipitate, but no crystals were found. In the other DA, needle-like crystals were found in both dialysis cells. "Cell 1 had approximately 500 needle-like crystals. Typical length was 1.0mm with the longest 2.5mm. The needles were extremely thin when compared to ground-based crystals." (1, p. 13) The second cell had fewer and smaller needle-like crystals.

It was concluded that the "...present dialysis technique is not adequate. It is unreliable and occupies too much space per number of experiments." (1, p. 16).

Vapor Diffusion Experiments

The "hanging drop method" vapor diffusion experiments were performed in two Vapor Diffusion Apparatuses (VDAs). Each of these VDAs contained 24 similar growth chambers. (The VDAs had been slightly modified after Bugg's STS-031 experiment to enhance experimental success.)

Configured just above each growth chamber was either a 4.0 mm or 5.0 mm (outer diameter) glass syringe, the tip of which extended into the growth chamber. A Teflon™ coating was added to some of the syringe tips to reduce glass tip nucleation sites. <Note: The shape of the tips was not specified.> Syringe tip plugs were employed to contain protein solutions within the syringes prior to the experiment initiation. (A listing of the employed proteins can be found in the **Sample Materials** section below.)

Configured at the bottom of each chamber was a cellulose wick saturated with an appropriate precipitating agent. <Note: It is not clear at what point the precipitating agent was added to the wick (prior to, or after launch).>

During the experiment, the syringe tip plugs were retracted to a position opposite the syringe tip and a protein drop was extruded from the syringe into the chamber using a piston. Reportedly, drops of either 30 $\mu$l (VDA-1) or 40 $\mu$l (VDA-2) were (1) maintained at the glass tips or (2) tethered to the tip plugs. Some seeding of the drops took place. The drops were left to equilibrate with the reservoir solutions (precipitating agents). <Note: Because results from Bugg's earlier STS-031 flight indicated that 60 $\mu$l drops were somewhat unstable and may not equilibrate totally in the time allotted for this mission, 30 and 40 $\mu$l drops were deployed for these experiments. As stated above, it was reported that this shuttle mission was prematurely shortened and that protein crystallization times were shorter than originally expected. Even so, it was reported that droplets equilibrated sufficiently during the shortenned mission.>

Three days after initiation, the experiment was terminated. (Reference (1) indicates that not all syringes were restoppered prior to the shuttle landing.)

Inflight and post-flight analyses of each of the 48 chambers are briefly reported in Reference (1). Overall it was concluded that despite apparent temperature fluctuations aboard the shuttle, "...good crystals suitable for X-ray diffraction studies were obtained of canavalin, concanavalin B, bacterial purine nucleoside phosphorylase, human serum, and C-reactive protein. The canavalin and human serum albumin crystals appear to diffract to higher resolution than earth crystals." (1, p. 15)

"It appears that the elimination of density-driven sedimentation can affect crystal morphology. The best example of this is canavalin, which grew crystals that were dispersed through the droplets in space.... Nearly all the space-grown crystals appear to have formed from separate nucleation sites, resulting in uniform morphologies." (6, p. 689) Similarly, Earth-grown Canavalin crystals "...generally form as fused aggregates at the bottom of the droplets." (6, p. 689)

See Reference (1) for additional information.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Drops, Drop Formation, Droplet Creation Via a Syringe, Piston System, Droplet Size, Drop Stability, Hanging Drop Method, Vapor Diffusion Method, Dialysis, Precipitation, Solutal Gradients, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Convection, Sedimentation, Seed Crystals, Liquid/Liquid Interface, Liquid/Liquid Diffusion, Solid/Liquid Interface, Liquid/Vapor Interface, Semipermeable Membranes, Nucleation, Nucleation Sites, Needles, Contained Fluids, Liquid Reservoir, Containerless Processing, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design, Three-Dimensional Crystalline Structure, Crystalline Structure, Crystal Morphology, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Coated Surfaces, Thermal Environment More Extreme Than Predicted, Processing Difficulties, Processing Time Not As Long As Planned

**Number of Samples:** Dialysis: ten; Vapor diffusion: forty-eight
**Sample Materials:** Proteins, in the VDA units: hen egg white lysozyme, human serum albumin (HSA), 3 different preparations of canavalin, designated as canavalin I, canavalin II, and canavalin III, concanavalin-B, human C-reactive protein (CRP) and bacterial purine nucleoside phosphorylase hypoxantine complex (B-PNP); in the dialysis units: canavalin, concanavalin B
**Container Materials:** VDA syringes: glass; DAs: unclear

**Experiment/Material Applications:**
The proteins were chosen for several reasons:

"(A) The University of Alabama at Birmingham were experienced in the crystallization of the proteins.

"(B) Canavalin crystals grow rather large in less than 3 days. However, earth-grown crystals have visible surface defects and only diffract to moderate resolution.

"(C) CRP crystals grow quickly, but are weakly diffracting.

"(D) B-PNP grows thin needle-like crystals in 24 hours. Improvement of the size and resolution of these crystals would greatly aid in the structural determination.

"(E) HSA crystals diffract poorly and the crystal morphology appears to be affected by the orientation of growing crystals with respect to gravity.

"(F) Concanavalin B crystals grow as thin plates, which is not an ideal morphology for diffraction studies.

"(G) Lysozyme is a standard protein that has been included on all flights." (1, p. 2)

Applications of such research can be found under Bugg, STS-023.

**References/Applicable Publications:**

(1) Protein Crystal Growth Results from Space Shuttle Flight 61-C. Memorandum from Charlie Bugg/Larry Delucas (University of Alabama in Birmingham) to Mr. Vaughn Yost (NASA MSFC), June 16, 1989, 17 pp. (post-flight)

(2) Bugg, C. E.: The Future of Protein Crystal Growth. Journal of Crystal Growth, 76 (1986), pp. 535-544.

(3) DeLucas, L. J., Suddath, F. L., Snyder, R., Naumann, R., Broom, B., Pusey, M., Yost, V., Herren, B., Carter, D., Nelson, B., Meehan, E. J., McPherson, A., and Bugg, C. E.: Preliminary Investigations of Protein Crystal Growth Using the Space Shuttle. Journal of Crystal Growth, 76 (1986), pp. 681-693. (post-flight 61-C)

(4) Bugg, C. E. and Clifford, D. W.: Protein Crystal Growth in Space. AIAA 25th Aerospace Sciences Meeting, January 13-15, 1986, Reno, Nevada, 10 pp. (post-flight)

(5) Hermann, F. T.: Advanced Protein Crystal Growth Flight Hardware for the Space Station. AIAA Aerospace Sciences Meeting, January 11-14, 1988, Reno, Nevada, AIAA #88-0345.

(6) DeLucas, L. J., Suddath, F. L., Snyder, R., Naumann, R., Broom, M. B., Pusey, M., Yost, V., Herren, B., Carter, D., Nelson, B., Meehan, E. J., McPherson, A., and Bugg, C.: Preliminary Investigations of Protein Crystal Growth Using the Space Shuttle. Journal of Crystal Growth, 76 (1986), pp. 681-693. (post-flight)

(7) Information describing mission by mission PCG results. On display at NASA Marshall Space Flight Center, Huntsville, Alabama. (post-flight)

(8) Presentation by Blair Herren, NASA Marshall Space Flight Center, Protein Crystal Growth, Office of Commercial Programs, March 1987. (viewgraphs only; post-flight)

(9) Naumann, R. J.: Microgravity Science and Applications. In: In Space '87, Japan Space Utilization Promotion Center (JSUP), pp. 19-21. (post-flight)

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**Experiment Origin:** USA
**Mission:** STS Launch #26, STS-26 (Discovery)
**Launch Date/Expt. Date:** September 1988
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Middeck Experiment
**Processing Facility:** New Protein Crystal Growth (PCG) apparatus differing from previous flights by now offering temperature control (including refrigeration) and some automation. Three 20-chambered trays (Vapor Diffusion Apparatuses (VDAs)) were used. Double barreled syringes were employed to allow separation of precipitate and protein for later mixing in flight.
**Builder of Processing Facility:** Growth chamber trays: Teledyne Brown Engineering, Huntsville, Alabama; refrigerators: McDonnell Douglas, St. Louis, Missouri. (Refrigerator upgrades including flammability precautions, improved temperature sensing, etc., were completed at NASA Marshall Space Flight Center, Huntsville, Alabama.)

**Experiment:**
Protein Crystal Growth (PCG) (PCG-II-01)

This space shuttle experiment was the fifth in a series of investigations designed by Bugg et al. to study the growth of protein crystals (see Bugg, STS-023, STS-026, STS-031, and STS-032). The overall objectives of the investigations were to (1) evaluate the effects of reduced gravity on several protein crystalline systems and (2) develop hardware for optimizing crystal growth in the space environment. The specific objective of the experiment was to grow protein crystals by a technique closely related to the vapor diffusion "hanging drop method." <Note: Although growth of

crystals by the dialysis method had been examined during earlier experiments in this investigative series, these experiments were discontinued on this flight.>

During earlier STS experiments by Bugg, environmental temperature fluctuations occurred both inflight (during crystal growth) and post-flight (on the shuttle runway). Thus, for this mission, a temperature control unit was added to vapor diffusion experimental hardware to maintain the PCG growth chambers at the appropriate temperature. This unit, called a Refrigeration Incubator Module (RIM), was capable of maintaining any set temperature between 2 and 35 +/- 0.8 $^{o}$C.

The Vapor Diffusion Apparatuses (VDAs) employed during Bugg's previous STS experiments were redesigned for this mission to enhance experimental success. The new vapor crystal growth hardware consisted of three "PCG growth trays." Each of these trays contained 20 similar growth chambers.

Configured just above each 5.3 cm$^3$ growth chamber was a double barreled syringe, the tip of which extended into the growth chamber. <Note: Several characteristics of the syringes were not detailed in the available publications including (1) the syringe tip shape and (2) the syringe material.> The chamber also contained an absorbent material which could be saturated with a solution of the precipitating agent.

Approximately 24 hours prior to launch, the syringes in each of the chambers were filled: (1) one syringe barrel was filled with a protein solution, (2) the second syringe barrel was filled with the appropriate precipitating agent, and (3) the absorbent material was saturated with the precipitating agent. <Note: A listing of the employed proteins can be found in the **Sample Materials** section below.> Syringe tip plugs were employed to contain the fluids within the syringe. The growth trays were placed within the RIM (approx. temperature 22 $^{o}$C) until experiment activation in orbit.

During the mission, the trays were removed from the RIM and the experiment was initiated. Hand-operated deployment of each drop (during Bugg's earlier experiments) was alleviated by using a ganging mechanism which activated all the drops in a vapor diffusion apparatus simultaneously. Upon activation, mixing of the protein and precipitating agent was realized by repeatedly extruding and retracting a drop from the syringe. After mixing (and seeding of some solutions), 40 microliter drops were left attached to the syringe tips. The trays were then returned to the RIM (22 $^{o}$C) where, during the next few days, the drops were left to equilibrate with the precipitating agent (contained within the saturated material). The protein drops were photographed after activation and at regular (24-hour) intervals. After 3 days, the resulting crystals and surrounding solution were retracted into the syringes and stoppered with the tip plugs.

Post-flight analysis indicated that five out of the eleven proteins investigated during the mission resulted in crystals suitable for X-ray diffraction evaluation. The best results were obtained from the crystallization of gamma-interferon, porcine elastase, and isocitrate lyase.

The gamma-interferon crystals were grown from a solution of 49% ammonium sulfate, 0.05 M Na acetate, pH = 5.9. Reportedly, two gamma-interferon crystals "...were obtained that were larger than the best that have ever been produced in any ground experiments; one was... [approximately]... 50% larger than the largest crystal that had been obtained previously. The overall morphology was similar to the Earth-grown crystals." (5, p. 652)

It was reported that three dimensional intensity data of one of the space-produced gamma-interferon crystals (0.7 X 0.5 X 0.4 mm) (1) displayed "...significant increase in measurable data at resolutions where the earth-grown crystals display no significant diffraction," (6, p. 306) and (2) "...is clearly superior to any data obtained previously." (6, p. 306) "In principle, this improvement could reflect enhanced counting statistics resulting from the larger crystal volume." (6, p. 306) <Note: This thought was further discussed in Reference (6).>

Porcine elastase crystals were grown on the mission by adding small seed crystals (approx. 50 $\mu$l in the maximum dimension) into the syringe containing the precipitating agent (1.5 M $NaSO_4$, 0.1 M sodium acetate, pH = 5.0) In one of the growth chambers, the seed crystal could be seen suspended in the deployed drop at experiment initiation, and then attached to the surface of the droplet 1 day later. The crystal continued to grow into the solution during the remainder of the experiment attaining a length of 2.05 mm. Reportedly, this length was "...considerably larger than any elastase crystals obtained in ground control studies." (6, p. 307)

"A number of well-formed [porcine] elastase crystals in the range of 0.5 to 2.1 mm were obtained from six crystallization experiments on STS-26. Comparison of 3-D intensity data for a space-grown crystal with dimensions comparable to Earth-grown crystals studied earlier... revealed that the space-grown crystal yielded significantly more data at all resolution ranges, with enhancement in the ultimate resolution at which measurable data can be obtained." (5, p. 652)

Crystals of isocitrate lyase were grown from a solution of 1.7 M sodium citrate, 0.1 M Tris-HCl, pH = 8.0. It was reported that "An improved habit for isocitrate lyase was observed.... Although some dendritic growth was found in the space samples, a number of well-formed prisms... were obtained. These prisms belong to the same space group as the Earth-grown dendrites, but they yielded better intensity data throughout the density range." (5, p. 653).

Other observations were discussed in References (5) and (6).

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Drops, Drop Formation, Droplet Creation Via a Syringe, Hanging Drop Method, Vapor Diffusion Method, Precipitation, Solutal Gradients, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Convection, Sedimentation, Seed Crystals, Solid/Liquid Interface, Liquid/Vapor Interface, Nucleation, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design, Three-Dimensional Crystalline Structure, Crystalline Structure, Dendritic Structure, Crystal Morphology, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Liquid Mixing, Containerless Processing, Refrigeration

**Number of Samples:** sixty
**Sample Materials:** Proteins: (1) canavalin, (2) human serum albumin, (3) gamma-interferon, (4) human C reactive protein, (5) porcine elastase, (6) phosphorylase, (7) renin, (8) synthetic peptide (alpha helicin), (9) isocitrate lyase, (10) human purine nucleoside, (11) reverse transcriptase
**Container Materials:** syringes: unknown

**Experiment/Material Applications:**
High quality protein crystals are of interest to several areas of research, especially in the field of crystalline structural analysis. Structural determination of these complex molecules "...provides the key to understanding their biological function and could lead to methods of altering or controlling the function in ways that may result in new drugs." (2, p. 21)

The proteins as numbered above (in the Sample Materials section) have the following applications (as detailed in Reference (4)): (1) protein plant engineering for nutrition, (2) binds and transports drugs and other materials in serum, (3) antiviral agent-stimulates the immune system, (4) plays a central role in the human immune system, (5) involved in lung disease - structure used for drug design, (6) protein that interacts with phospholipids, (7) involved in controlling high blood pressure - structure will be used in drug design, (8) structural determination needed - human cell regulation of electrolytes-potassium, sodium, calcium, (9) crop disease control, (10) degrades anticancer drugs -- target for drug design, and (11) enzyme essential to the AIDS virus to complete its life cycle.

**References/Applicable Publications:**


(1) Investigators Examining Every Particle of Data from STS-26 Experiments. In NASA Marshall Star Newspaper, Vol. 29, Number 16, November 16, 1988, NASA Marshall Space Flight Center, Huntsville, Alabama, p. 1. (post-flight)

(2) NASA Press Kit: Space Shuttle Mission STS-26, September 1988, pp. 21-23. (preflight)

(3) Dumoulin, J. M.: STS-26 Experiment Protein Crystal Growth. NASA Fact Sheet, George C. Marshall Space Flight Center, June 1988. (preflight)

(4) Rose, J.: Post Launch Mission Report. Letter from C/Assistant Administrator for Commercial Programs to A/Administrator, Subject: Protein Crystal Growth (PCG) Experiment Post Launch Mission Operation. (post-flight)

(5) DeLucas, L.: Protein Crystal Growth in Microgravity. Science, Vol. 246, November 1989, pp. 651-654. (post-flight)

(6) DeLucas, L. J., et al.: Protein Crystal Growth Results For Shuttle Flights STS-26 and STS-29. Journal of Crystal Growth, 110 (1991), pp. 302-311. (post-flight)


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**Experiment Origin:** USA
**Mission:** STS Launch #28, STS-29 (Discovery)
**Launch Date/Expt. Date:** March 1989
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Middeck Experiment
**Processing Facility:** Protein Crystal Growth (PCG) apparatus used on STS-26 which (1) employed three 20-chambered trays (Vapor Diffusion Apparatuses (VDAs)), (2) offered temperature control (using a Refrigerator-Incubator Module (RIM)), and (3) provided some automation of drop deployment.
**Builder of Processing Facility:** Growth chamber trays: Teledyne Brown Engineering, Huntsville, Alabama; refrigerators: McDonnell Douglas, St. Louis, Missouri. (Refrigerator upgrades including flammability precautions, improved temperature sensing, etc. were completed at NASA Marshall Space Flight Center, Huntsville, Alabama.)

**Experiment:**
Protein Crystal Growth (PCG) (Vapor Diffusion) (PCG-III-01)

This space shuttle experiment was the sixth in a series of investigations designed by Bugg et al. to study the growth of protein crystals (see Bugg, STS-023, STS-026, STS-031, STS-032, STS-26). The overall objectives of the investigations were to (1) evaluate the effect of reduced gravity on several protein crystalline systems and (2) develop hardware for optimizing crystal growth in the space environment. The specific objective of this experiment was to grow protein crystals by a technique closely related to the vapor diffusion "hanging drop method."

The vapor diffusion experiments were performed in three 20-chamber crystal growth trays (see Bugg, STS-26, for additional details of the growth trays). Each chamber was configured with (1) a double-barreled syringe (one barrel contained the protein solution, the other the precipitating solution) and (2) a reservoir saturated with the precipitating solution. <Note: Several characteristics of the syringes were not detailed in the available publications including (1) the syringe tip shape and (2) the syringe material.> A listing of the employed proteins can be found in the **Sample Materials** section below. (The syringes in each of the chambers were reportedly filled approximately 24 hours prior to shuttle launch, and maintained at a temperature of approximately 22 $^{o}C$ in a Refrigerator-Incubator Module (RIM).) Tip plugs stoppered the syringes prior to experiment activation.

Once in orbit, the trays were removed from their temperature-controlled environment. The removal of the syringe tip plugs and subsequent repeated extrusion and retraction of drops from the double-barreled syringes was performed semi-automatically via a hand-operated wheel. After mixing of the solutions, 40 $\mu l$ drops were deployed attached to the syringe tips. The trays were then returned to the temperature-controlled units where, during the next several days, the drops were left to equilibrate with the reservoir solution. The protein drops were photographed after activation and at regular intervals. Upon deactivation, the resultant crystals and the surrounding solution were retracted into the syringes and stoppered with the syringe tip plugs.

<Note: Detailed information of the post-flight results could not be located at this time. The following brief summary was reported in Reference (6):>

"Fifteen different proteins were flow on STS-29. Unfortunately, showers of small crystals were produced in every chamber flown for all fifteen of these proteins. It is not clear what caused this. In spite of this, one crystal of the protein... [alpha] interferon grew large enough to collect 3D data. Although the crystal was 0.2 mm smaller in each dimension that the four earth-grown crystals used for comparison of the STS-26 data, the STS-29 space-grown crystal still produced data that were measurably better throughout the resolution range and extended further in resolution than the best ground-based crystals.... In addition, three large crystals ([...approximately] 0.4 X 0.3 X 0.5 mm) of the protein Lathyrus ochrus lectin I were also obtained. Data were collected to high resolution from one of these crystals and the structure was determined by molecular replacement methods.

crystals grown on earth by similar methods are smaller and display a different habit. A detailed comparison of the space and ground data will be published elsewhere...." (6, p. 310) <Note: This publication could not be procured at this time.>

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Drops, Drop Formation, Droplet Creation Via a Syringe, Hanging Drop Method, Vapor Diffusion Method, Precipitation, Solutal Gradients, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Convection, Sedimentation, Solid/Liquid Interface, Liquid/Vapor Interface, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design, Three-Dimensional Crystalline Structure, Crystalline Structure, Dendritic Structure, Crystal Morphology, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Liquid Mixing, Containerless Processing, Refrigeration, Crystal Showers, Processing Difficulties

**Number of Samples:** sixty
**Sample Materials:** <Note: Reference (3), which was published prior to the launch of the STS-29 mission, indicated that 19 different proteins were to be examined during this mission. They are listed in this section. However, Reference (6), which was published after the mission was completed, reported that only 15 different proteins were examined, but did not list them individually.> (1) diacetinase, (2) human growth hormone (human somototropin), (3) isocitrate lyase, (4) porcine elastase, (5) purine nucleoside phosphorylase, (6) renin, (7) AK&F 104662, (8) aridicin aglycone, (9) interferon, (10) neuraminidase, (11) alcohol oxidase, (12) green fluorescent protein, (13) LAC repressor, (14) lectin, (15) bovine pancreatic procarboxypeptidase, (16) ribsome, (17) tobacco mosaic virus, (18) urease, (19) canavalin
**Container Materials:** syringes: unknown

**Experiment/Material Applications:**
During this investigation, the growth of high-quality protein crystals was sought. If such crystals could be produced, high resolution X-ray diffraction studies might indicate the protein's three-dimensional structure. Once the structure is known, "...it may be possible to design drugs that will either block or enhance the protein's normal function within the body." (3, p.1)

The proteins as numbered above (see the Sample Materials section) have the following applications as reported in References (3) and (5).

(1) Enzyme which catalyzes the hydrolysis of glycerol esters and is specific for alkyl esters where the alkyl group has from 1 to 4 carbon atoms.

(2) Protein marketed for the treatment of children who are unusually small because their pituitary glands produce too little growth hormone.

(3) Target enzyme for crop disease control.

(4) Enzyme associated with the degradation of lung tissue.

(5) Target protein for the design of immunosuppressive and anticancer drugs.

(6) Enzyme produced by the kidneys which plays a major role in blood pressure control.

(7) Molecule representative of a class of antibiotics in which there is much current industrial interest.

(8) Glycopeptide antibiotic used to treat infection due to gram-positive organisms.

(9) Enzyme stimulates body's immune system and is used to treat cancer.

(10) Protein is one of two glycosylated surface proteins from the influenza virus.

(11) Enzyme which can be used to test for alcohol levels in body fluids.

(12) Protein is an acidic, globular, energy transfer protein.

(13) Regulates the expression of lactose operon in E.coli.

(14) Protein that binds glycames of glycoproteins localized at the surface cell.

(15) Enzyme that breaks down protein, similar to human proteins.

(16) Plays a major role in protein processing of cells.

(17) Protein food source in jack beans.

(18) Urea is a major solid nitrogen source in world agriculture. The development of a urease inhibitor could have a large impact on the pattern of world agriculture.

(19) Protein of interest because of its potential as a nutritional substance.

See also Bugg, STS-023.

**References/Applicable Publications:**

(1) "STS-29 Secondary Payloads," NASA Activities, March 1989, Vol. 20, No. 3, pp. 9-10. (preflight; very brief summary)

(2) "Discovery Lands at Edwards Ending Successful Mission," Marshall Star, Vol. 29, March 22, 1989, Number 31. (very brief summary)

(3) Rose, J.: Prelaunch Mission Operation Report No. C-420-89-29-01. From C/Assistant Administrator for Commercial Programs to A/Administrator, Subject: Protein Crystal Growth (PCG) Experiment Mission Operation Report-STS-29. (preflight)

(4) NASA Space Shuttle Mission STS-29 Press Kit, March 1989, pp. 15-16. (preflight)

(5) Dumoulin, J. M.: STS-29 Experiment-Protein Crystal Growth. NASA Fact Sheet, January 1989, NASA Marshall Space Flight Center, Huntsville, Alabama, 3 pp. (post-flight)

(6) DeLucas, L. J.: Protein Crystal Growth Results for Shuttle Flights STS-26 and STS-29. Journal of Crystal Growth, 110 (1991), pp. 302-311. (post-flight)

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**Experiment Origin:** USA
**Mission:** STS Launch #30, STS-32 (Columbia)
**Launch Date/Expt. Date:** January 1990
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** STS Middeck Experiment (three lockers were used)
**Processing Facility:** Two Refrigerator-Incubator Modules (RIMs) were employed. One of the RIMs one was to be maintained at 22 $^{\circ}$C, the other at 4 $^{\circ}$C. Each RIM held three Vapor Diffusion Apparatus (VDA) trays. Each VDA had 20 experiment chambers. A seventh VDA tray (not contained within a RIM) was used to evaluate drop stability.
**Builder of Processing Facility:** Not specifically stated, but it is assumed that
(1) the growth chamber trays were built by Teledyne Brown Engineering, Huntsville, Alabama and (2) the refrigerators were built by McDonnell Douglas, St. Louis, Missouri

**Experiment:**
Protein Crystal Growth (PCG) (Vapor Diffusion) (PCG-III-02)

This space shuttle experiment was the seventh in a series of investigations designed by Bugg et al. to study the growth of protein crystals (see Bugg, STS-023, STS-026, STS-031, STS-032, STS-26, STS-29). The overall objectives of the investigations were to (1) evaluate the effect of reduced gravity on several protein crystalline systems and (2) develop hardware for optimizing crystal growth in the space environment. The specific objective of the experiment was to grow protein crystals by a technique closely related to the vapor diffusion "hanging drop method."

The vapor diffusion experiments were performed in seven, 20-chamber trays. Each chamber was configured with (1) a double-barreled syringe (one barrel containing the protein solution, the other the precipitating solution) and (2) a porous reservoir saturated with the precipitating solution. (See Bugg, STS-26 for additional details of the growth trays.) <Note: A listing of the employed proteins can be found in the **Sample Materials** section below.> Tip plugs stoppered the syringes prior to experiment activation.

During the mission, six of the seven growth trays were housed within Refrigerator-Incubator Modules (RIMs); the seventh tray was housed in a stowage locker without temperature control. The RIMs were to be maintained at two different temperatures (22 $^{o}$C and 4 $^{o}$C) "...making it possible to crystallize a wider selection of proteins." (2, p. 2) During activation of the experiments, a hand-operated wheel provided semi-automation of (1) the removal of the syringe tips and (2) subsequent extrusion of drops from the double-barreled syringes. (Trays within the RIMs were removed for this activation procedure.) Mixing of the protein and precipitating agent was realized by repeatedly extruding and retracting the drop from, and back into, the syringe. After mixing, six of the trays were returned to the temperature-controlled units. (The seventh tray was used to study droplet stability and crystal formation as a function of accelerations. Droplets were photographed during orbiter maneuvers and crew activities.)

During the next several days, the drops were left to equilibrate with the reservoir solution. Reportedly, "Because the water content is greater in the droplet than in the reservoir, water vapor will move from the droplet to the reservoir, thus increasing the concentrations of both the protein and the precipitant in the droplet. When these concentrations reach a certain level, the protein molecules begin to align, forming crystals suspended in the droplet." (1, p. 6)

In-flight deactivation of all the experiments occurred several days after experiment initiation. At deactivation, the resultant crystals and surrounding solution were retracted into the syringes and stoppered with the tip plugs.

No further information concerning this experiment (including post-flight sample analysis) could be located at this time.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Drops, Drop Formation, Drop Stability, Droplet Creation Via a Syringe, Hanging Drop Method, Vapor Diffusion Method, Precipitation, Solutal Gradients, Diffusion, Diffusion Mass Transfer, Diffusion-Controlled Growth, Convection, Sedimentation, Solid/Liquid Interface, Liquid/Vapor Interface, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Structure Based Drug Design, Three-Dimensional Crystalline Structure, Crystalline Structure, Crystal Morphology, Liquid Expulsion Through a Small Orifice, Liquid Transfer, Liquid Mixing, Containerless Processing, Refrigeration, Acceleration Effects

**Number of Samples:** 140
**Sample Materials:** Proteins: (1) human growth hormone, (2) isocitrate lyase, (3) gamma-interferon, (4) porcine elastase, (5) cyanobacterium photosystem 1 complex, (6) neuraminidase, (7) aldose reductase, (8) aridicin aglycone, (9) phospholipase A-2, (10) cyclosporin A-cyclophilin complex, (11) catalase, (12) canavalin, (13) 12 base Pair DNA, (14) diacetinase, (15) reverse transcriptase, (16) lysozyme, (17) lectin, (18) porcine pancreatic phospholipase $A_2$, (19) TRP repressor/operator complex, (20) LAC repressor/operator, (21) satellite tobacco mosaic virus, (22) ribosome, (23) human serum albumin, (24) neuraminidase
**Container Materials:** syringes: unknown

**Experiment/Material Applications:**
High-quality protein crystals are of interest to several areas of research, especially in the field of crystalline structural analysis. If investigators can process proteins "...in such a way that protein molecules organize symmetrically into crystals large enough to study..." (1, p. 1) then high resolution X-ray diffraction will reveal the three-dimensional structure of the protein. "Determining a protein's molecular structure is an es-

sential step in several phases of medical research. Knowing the three-dimensional structure allows for a more systematic approach to designing compounds which alter the activities of these proteins." (1, p. 1)

Experiments performed during this mission "...could improve food production and lead to innovative new drugs to combat cancer, AIDS, high blood pressure, organ transplant rejection, rheumatoid arthritis, and many other diseases." (2, p. 1)

The specific reasons why proteins 1, 2, 4, 6, 8, 11, 12, 14, 17, 20, 21, 22, and 24 (as numbered above in the Sample Materials section) were selected can be found under Bugg, STS-29 (this chapter). The specific reasons why proteins 3, 15, and 23 we selected can be found under Bugg, STS-26. The rest of the listed proteins have these applications (as detailed in Reference (1)):

(Sample Material #5) Knowledge of the structure of this material will provide a clearer understanding of photosynthetic mechanisms.

(Sample Material #7) Enzyme that has been implicated in a number of diabetic complications.

(Sample Material #9) Enzyme performs functions associated with cell membranes-better understanding of it could lead to improved medications for pain and inflammation.

(Sample Material #10) Principal drug in current use to suppress immune rejection during organ transplants in humans.

(Sample Material #13) A major mammalian detoxifying enzyme responsible for clearing potentially harmful peroxide radicals from rapidly metabolizing tissues.

(Sample Material #16) Enzyme involved in degradation of bacteria cell membranes. (Flown on this flight only to assess impact of acceleration environment.)

(Sample Material #18) Enzyme associated with many human disease states, including rheumatoid arthritis and septic shock.

(Sample Material #19) Protein used to study the structural bases for the specific affinity of a genetic regulatory protein and its target.

**References/Applicable Publications:**

(1) Mission Operation Report. Office of Commercial Programs, Report No. C-600-32-89-01, NASA, Protein Crystal Growth, 18 pp. (preflight)

(2) Dumoulin, J. M.: STS-32 Experiment-Protein Crystal Growth. NASA Fact Sheet, NASA Marshall Space Flight Center, Huntsville, Alabama, November 1989, 2 pp. (preflight)

(3) NASA Space Shuttle Mission STS-32 Press Kit, December 1989, pp. 19-21. (preflight)

(4) Bugg, C. E. and DeLucas, L. J.: Microgravity Protein Crystal Growth. AIAA 29th Aerospace Sciences Meeting, January 7-10, 1991, Reno, Nevada, AIAA #91-0111. (short overview of results from all of the missions)

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**Experiment Origin:** Federal Republic of Germany
**Mission:** STS Launch #22, STS-030 (STS 61-A, Spacelab D1: Challenger)
**Launch Date/Expt Date:** October 1985
**Launched From:** NASA Kennedy Space Center, Florida
**Payload Type:** Spacelab Facility, Materials Science Double Rack (MSDR)
**Processing Facility:** D1 Cryostat
**Builder of Processing Facility:** Unknown

**Experiment:**
Bacteriorhodopsin - A Membrane Protein to Convert Light Energy (WL-CRY 00)

In order to understand "...the molecular mechanism of vectorial transport in light [solar radiation], and therefore to uncover the thermodynamic secret of photoenergetics, detailed knowledge of the tertiary structure of the... [bacteriorhodopsin] molecule is necessary. Spatial resolution, to trace the peptide chain and to position the amino acids involved in the [proton] pumping process, is ensured so far only by crystallography. This technique demands large and well ordered crystals." (1, p. 295)

Bacteriorhodopsin single crystals of sufficient size and order have not been produced in the terrestrial laboratory. In the space laboratory, however (where gravity-induced convection and sedimentation are reduced), slow, undisturbed diffusion-dominated growth of the crystals may be possible.

It appears (from Reference (1)) that a free interface diffusion technique was used during the D1 experiment to initiate crystal growth. "A protein solution is positioned on top of a concentrated salt solution, separated by a thin layer of buffer solution. Thus, slow and undisturbed diffusion is ensured, a major precondition for limited seed formation and growth of large and well-ordered crystals." (1, p. 295) Figure 2 in Reference (1) indicates that the three solutions were placed in a test tube capped with a parafilm seal. No additional information concerning the Spacelab experiment configuration could be located.

A discussion of the experimental results could not be located.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Single Crystals, Salt Solution, Three-Dimensional Crystalline Structure, Buoyancy-Driven Convection, Diffusion, Diffusive Mass Transfer, Diffusion-Controlled Growth, Liquid/Liquid Diffusion, Double Diffusion, Sedimentation, Concentration Distribution, Liquid/Liquid Interface, Solid/Liquid Interface, Crystal Morphology, Crystalline Structure, Contained Fluids, Biotechnology, Biological Materials Processing, Biological Structures, Membranes, Photoenergetics

**Number of Samples:** unknown
**Sample Materials:** The exact materials were unspecified: (1) a protein solution (presumably containing bacteriorhodopsin), (2) a buffer solution, and (3) a salt solution
**Container Materials:** unknown, possibly glass test tubes

**Experiment/Material Applications:**
See above experiment summary and Reference (1).

**References/Applicable Publications:**

(1) Altmüller, D., Grolig, F., Lindhardt, R., and Wagner, G.: Bacteriorhodopsin - A Membrane Protein to Convert Light Energy. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 294-296. (post-flight)

(2) Hamacher, H., Merbold, U., and Jilg, R.: Analysis of Microgravity Measurements Performed During D1. In Proceedings of the Norderney Symposium on Scientific Results of the German Spacelab Mission D1, Norderney, Germany, August 27-29, 1986, pp. 48-56. (post-flight; acceleration measurements)

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**Experiment Origin:** USA
**Mission:** Consort 1 (Starfire Rocket)
**Launch Date/Expt. Date:** March 1989
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Two diffusion chambers within the Materials Dispersion Apparatus (MDA) minilab
**Builder of Processing Facility:** Instrumentation Technology Associates, Inc., Exton, Pennsylvania

**Experiment:**
Protein Diffusion

The overall, long-term objective of this protein diffusion research was to examine the process of free-fluid separation of bone marrow cells and sub-elements. Specifically, processing techniques were sought which would eliminate selected subgroups of T-lymphocytes that react with major histocompatible (MHC) antigens and cause immune rejection of human bone marrow transplants.

This Consort 1 experiment was designed to (1) obtain model-cell diffusion measurements which were not masked by strong convective forces and (2) use these diffusion measurements to determine cell mobility information.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Cassanto, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2, and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams (including this one) used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (eleven experiments), the apparatus was also used to (1) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (2) determine the effect of re-entry loads on terrestrial-grown crystals (one experiment).

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together. Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (1) aligned or (2) misaligned wells on the top with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place. Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively by Cassanto et al. for a protein crystal stability experiment which investigated the effect of re-entry loads on terrestrial-grown crystals (see Cassanto, Consort 1). The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1). This experiment was allotted two "Type 2" test wells. Discussion of this specific well-type is detailed here.

Each "Type 2" test well provided the investigator with one experimental opportunity. The well-type used two sample wells, one on the top block and one on the bottom block. Prior to the rocket flight, one of the wells in each of the "Type 2" test wells was filled with a solution of urokinase (molecular weight of 53,000 daltons), while the other well in each of the "Type 2" test wells was filled with a solution of monoclonal antibody (MAb) to T-helper and T-suppressor lymphocytes. <Note: It is unclear which fluid was placed in the wells in the top block and which was placed in wells in the bottom block.> The blocks were joined together such that the well in the upper block was purposely misaligned with the well on the lower block. Once the rocket had been launched and the low-gravity phase achieved, a motor and cam mechanism moved the upper block to the right, aligning the wells on the upper and lower blocks. Once the wells were in contact, material diffusion was realized across the liquid-liquid interface. Just prior to the termination of the low-gravity rocket phase, the upper block again moved right misaligning the upper and lower wells. This misalignment prevented further material diffusion between the liquids.

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected allowing diffusion to take place in the "Type 2" test wells. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination.

Post-flight examination of the samples allotted to this experiment indicated that diffusion between the wells (in both "Type 2" test wells) had occurred as expected. Reportedly, the amount of diffused molecules was measured and used to calculate the effective diffusion constants for both proteins.

No further information discussing the results could be located at this time.

**Key Words:** Protein Crystal Growth, Crystal Growth From Solution, Convection, Diffusion, Diffusive Mass Transfer, Liquid/Liquid Diffusion, Double Diffusion, Particle Mobility, Buoyancy Effects Diminished, Separation of Components, Liquid/Liquid Interface, Interface Physics, Liquid Leakage, Contamination Source, Biotechnology, Biological Materials Processing, Biological Structures, Pharmaceutical Applications, Medical Applications, Immune System, Antibodies, Human Bone Marrow Transplants, Fibrin Structures, Blood Clotting

**Number of Samples:** two
**Sample Materials:** Concentrated solutions of urokinase (molecular weight 53000 daltons) diffused into concentrated solutions of Anti Leu-1 monoclonal antibody (MAb) to T-helper and T-suppressor lymphocytes.
**Container Material:** inert material

**Experiment/Material Applications:**
Separation and purification of biological materials such as cells, cell components, cell surface antigens, proteins and hormones may be enhanced in the low-gravity environment if buoyancy-driven convective effects are sufficiently reduced. Experiments during this sounding rocket flight were performed to explore model T-lymphocyte cell mobility. Urokinase (an enzyme which dissolves fibrin blood clots after they are formed) was diffused

into a monoclonal antibody (MAb) to T-helper and T-suppressor lymphocytes (a material to suppress blood clots).

The experiment was the first in a series of investigations designed to better understand how arthritis cripples joints. Ultimately shuttle investigations "...may lead to pharmaceuticals that prevent the disease from distorting hands and other body joints." (5, p. 1)

**References/Applicable Publications:**


(1) Memo from W. Vardaman (RANTEK, Inc.) to C. Winter (NASA, Marshall Space Flight Center, Huntsville, Alabama), March 9, 1990. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight, brief description of MDA)

(3) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort I Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(4) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989; Màlaga, Spain, IAF #89-439. (post-flight)

(5) Arthritis Experiment Scheduled for Space Flights. Information prepared by RANTEK, Inc., October 28, 1988, 3 pp. (provided by ITA to C. A. Winter on 1/91)

(6) Project Plan for Conducting Biotechnology Experiments on Joust 1. September 5, 1990. (post-flight; provided by ITA to C. A. Winter on 1/91.)

(7) Input received from Experiment Investigator, May 1991 and July 1993.

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**Experiment Origin:** USA
**Mission:** Consort 1 (Starfire Rocket)
**Launch Date/Expt. Date:** March 1989
**Launched From:** White Sands Missile Range, New Mexico
**Payload Type:** Sounding Rocket Experiment
**Processing Facility:** Materials Dispersion Apparatus (MDA) Minilab
**Builder of Processing Facility:** Instrumentation Technology Associates, Inc., Exton, Pennsylvania

**Experiment:**
Protein Crystal Stability

Fragile protein crystals produced on orbiting spacecraft are subjected to rigorous, high-level g-loads during vehicle re-entry. Because the survivability of these crystals is a matter of concern, the objective of this short-duration, Consort 1 experiment was to determine the effects of sounding rocket re-entry g-loads on terrestrial-grown crystals.

The experiment was one of 17 investigations simultaneously performed in the Materials Dispersion Apparatus (MDA) on Consort 1 (see also Burgess, Consort 1 (Chapter 16); Fiske, Consort 1 (Chapter 11); Hammerstedt, Consort 1 (Chapter 1); Luttges, Consort 1 (Chapter 1); Pellegrino, Consort 1 (three experiments, Chapter 1); Rodriguez, Consort 1 (Chapter 8); Schoonen, Consort 1 (Chapter 8); Stodieck, Consort 1 (Chapter 1); Todd, Consort 1 (four experiments, Chapters 1, 2, and 11); Vera, Consort 1 (two experiments, Chapter 1)). While most of the investigative teams used the MDA to conduct a liquid-liquid or liquid-solid diffusion study (eleven experiments), the apparatus was also used to (1) determine the role of fluid physics on the formation of films and the casting of membranes (five experiments) and (2) perform this protein crystal stability experiment.

The MDA was fashioned (in part) from two blocks of inert material. Each block had over 100 sample wells strategically drilled into its surface. A well was capable of holding 100-500 ml of fluid. After all of the wells had been filled with the appropriate sample materials (depending on each investigator's specific objectives), the blocks were joined together.

Twice during the mission, the upper block moved relative to the bottom block. Each movement of the block either (1) aligned or

(2) misaligned wells on the top block with wells on the bottom. When the wells were aligned, material transfer from one well to the other could take place. When the wells were not aligned, material transfer from one well to the other could not take place.

Appropriate positioning of the wells on the top block to those on the bottom resulted in what was called "Type 1," "Type 2," "Type 3," or "Type 4" test wells. The "Type 1" test wells were used exclusively for this protein crystal stability experiment. The "Type 2" and "Type 3" test wells were used for either the liquid-liquid or liquid-solid diffusion experiments. The "Type 4" test wells were used exclusively for the film formation and membrane casting experiments (see Pellegrino, Consort 1; Vera, Consort 1). This experiment was allotted thirteen "Type 1" test wells. Discussion on this specific well-type is detailed here.

A "Type 1" test well provided the investigator with one experimental opportunity. Each of the 13 "Type 1" well-types used only one well during the entire mission. This single well was located in the lower block of the MDA. Each of these 13 wells contained a terrestrial-grown protein crystal and a liquid solution. Whenever the upper block moved to the right during the mission (as previously detailed), the single well in the lower block contacted only a flat surface of the upper block. Thus, a "Type 1" well was never aligned to another well during the flight. (Because the MDA performed 17 different experiments at once, the upper block moved to align/misalign wells as required in other experiments. However, this experiment required only one well in which a protein crystal and liquid could be contained).

Specific crystalline and liquid types were not detailed in any of the available publications. However, information released prior to the Consort 1 flight indicated that "Earth grown lysozyme crystals, still in their mother liquor will be exposed to the acceleration levels associated with the launch and re-entry of the sounding rocket." (5, viewgraphs)

Post-flight evaluation of the operation of the MDA indicated that the upper block moved as expected. Although it was reported that minor fluid leakage in the top and bottom blocks of the MDA resulted in contamination of some of the test wells allotted to the 17 investigations, it was not clear if this particular experiment was affected by the contamination. A brief note in Reference (1) (viewgraphs) indicated that crystal shattering occurred. <Note: It is not clear if the crystals could have shattered during vehicle takeoff as high g-loads during this time would also be prevalent.>

No further information discussing the nature of the shattering or other specific results could be located at this time.

**Key Words:** Protein Crystal Growth, Payload Survivability, Acceleration Effects, Rocket Vibration, Vehicle Re-Entry Forces/Vibration, Crystal Shattering, Liquid Leakage, Contained Fluids, Contamination Source

**Number of Samples:** thirteen
**Sample Materials:** Unknown, a document published prior to the flight reported that "Earth grown lysozyme crystals, still in their mother liquor...." (5, viewgraphs)
**Container Materials:** inert material

**Experiment/Material Applications:**
"Protein crystal destruction could be a problem on unmanned [low-gravity] carriers, owing to high re-entry 'g-loads'. It is necessary to determine the magnitude of this risk and to develop countermeasures if necessary." (5, viewgraphs)

**References/Applicable Publications:**
(1) Cassanto, J. M.: Overview/Summary of Results from the Material Dispersion Apparatus (MDA) on the Consort 1 Rocket Flight. Presentation to the Spring CMDS Scientific and Technical Project Review, Guntersville, Alabama, April 18-20, 1989. (post-flight)

(2) Wessling, F. C. and Maybee, G. W.: Consort 1 Sounding Rocket Flight. Journal of Spacecraft and Rockets, Vol. 26, No. 5, September-October 1989, pp. 343-351. (preflight; brief description of MDA)

(3) Wessling, F. C., Lundquist, C. A., and Maybee, G. W.: Consort 1 Flight Results-A Synopsis. Presented at the IAF 40th International Astronautical Congress, October 7-13, 1989, Màlaga, Spain, IAF #89-439. (post-flight)

(4) Cassanto, J. M. and Korszun, R.: The Materials Dispersion Apparatus (MDA) for MPS Space Flight Application. 38th International Astronautical Congress IAF, Brighton, England, October 10-17, 1987, 28 pp. (preflight)

(5) MDA Experiment No. 7 on Consort 1 and 2 Rocket Flights: Protein Crystal Stability. (Information provided by ITA to C. A Winter on 1/91, preflight)

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**12b. DISTRIBUTION CODE**

**13. ABSTRACT** *(Maximum 200 words)*

An electronic data base identifying over 800 fluids and materials processing experiments performed in a low-gravity environment has been created at NASA Marshall Space Flight Center. The compilation, called MICREX (MICrogravity Research Experiments) was designed to document all such experimental efforts performed (1) on U.S. manned spac vehicles, (2) on payloads deployed from U.S. manned space vehicles, and (3) on all domestic and international sounding rockets (excluding those of China and the former U.S.S.R.). Data available on most experiments include (1) principal and co-investigator (2) low-gravity mission, (3) processing facility, (4) experimental objectives and results, (5) identifying key words, (6) sample materials, (7) applications of the processed materials/research area, (8) experiment descriptive publications, and (9) contacts for more information concerning the experiment. This technical memorandum (1) summarizes the historical interest in reduced-gravity fluid dynamics, (2) describes the importance of a low-gravity fluids and materials processing data base, (4) describes the MICREX data base format and computational World Wide Web access procedures, and (5) documents (in hard-copy form) the descriptions of the first 600 fluids and materials processing experiments entered into MICREX.

**14. SUBJECT TERMS** data base, low-gravity materials processing, fluid dynamic experiments, low-g facilities, early rocket design, aircraft low-g test-beds, drop tubes, drop towers, sounding rockets, Apollo program, Skylab, space shuttle, low-g experiment history

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