Application of a Modified Gas Chromatograph to Analyze Space Experiment Combustion Gases on Space Shuttle Mission STS–94

William K. Coho and Karen J. Weiland
Lewis Research Center, Cleveland, Ohio

David M. VanZandt
Aerospace Design and Fabrication, Inc., Brook Park, Ohio

Prepared for the
Pittcon '98
sponsored by The Pittsburgh Conference
New Orleans, Louisiana, March 1–6, 1998
Acknowledgments

The authors wish to thank Kevin Magee and Terry Wright for their assistance in making the gas chromatography measurements. The authors wish to thank Russ Steinbach, Sheryl Sandridge, and Tom Hudach for their assistance in performing the modifications to the instrumentation.

Trade names or manufacturers' names are used in this report for identification only. This usage does not constitute an official endorsement, either expressed or implied, by the National Aeronautics and Space Administration.

Available from

NASA Center for Aerospace Information
800 Elkridge Landing Road
Linthicum Heights, MD 21090-2934
Price Code: A03

National Technical Information Service
5287 Port Royal Road
Springfield, VA 22100
Price Code: A03
APPLICATION OF A MODIFIED GAS CHROMATOGRAPH TO ANALYZE SPACE EXPERIMENT COMBUSTION GASES ON SPACE SHUTTLE MISSION STS-94

William K. Coho, Karen J. Weiland
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

and

David M. VanZandt
Aerospace Design and Fabrication, Inc.
Brook Park, Ohio 44142

ABSTRACT

A space experiment designed to study the behavior of combustion without the gravitational effects of buoyancy was launched aboard the Space Shuttle Columbia on July 1, 1997. The space experiment, designated as Combustion Module-1 (CM-1), was one of several manifested on the Microgravity Sciences Laboratory-1 (MSL-1) mission. The launch, designated STS-94, had the Spacelab Module as the payload, in which the MSL-1 experiments were conducted by the Shuttle crewmembers.

CM-1 was designed to accommodate two different combustion experiments during MSL-1. One experiment, the Structure of Flame Balls at Low Lewis-number experiment (SOFBALL), required gas chromatography analysis to verify the composition of the known, premixed gases prior to combustion, and to determine the remaining reactant and the products resulting from the combustion process in microgravity.

A commercial, off-the-shelf, dual-channel micro gas chromatograph (gc) was procured and modified to interface with the CM-1 Fluids Supply Package and the CM-1 Combustion Chamber, to accommodate two different carrier gases, each flowing through its own independent column module, to withstand the launch environment of the Space Shuttle, to accept Spacelab electrical power, and to meet the Spacelab flight requirements for electromagnetic interference (EMI) and offgassing.

The gc data was downlinked to the Marshall Space Flight Center for near-real time analysis, and stored on-orbit for post-flight analysis.

The gas chromatograph operated successfully during the entire SOFBALL experiment and collected 309 runs. Because of the constraints imposed upon the gas chromatograph by the CM-1 hardware, system and operations, it was unable to measure the gases to the required accuracy. Future improvements to the system for a reflight of the SOFBALL experiment are expected to enable the gas chromatograph to meet all the requirements.

INTRODUCTION

The Combustion Module-1 (CM-1) is a state-of-the-art, space laboratory designed to study the behavior of combustion phenomena in the absence of gravity. The facility first flew on the STS-83 and STS-94 missions in April and July 1997, aboard the space shuttle Columbia as part of the Microgravity Science Laboratory-1 payload. The facility was designed to conduct multiple combustion experiments studying gaseous flames using advanced diagnostics and was operated by the astronauts. Among the measurements conducted on-orbit in support of the experiment known as the Structure Of Flame Balls At Low Lewis-number (SOFBALL) was gas sampling and analysis by a specially modified gas chromatograph. The gas chromatograph was intended to verify the gases in the chamber prior to combustion so that the initial composition would be known and could be used as input to numerical models. It was also intended to measure the composition of the products resulting from combustion and any remaining reactants, which would reveal the extent of combustion prior to extinction.
The SOFBALL experiment studied the flammability, propagation, and extinction of weakly burning flames (refs. 1 and 2). Despite many years of study, the behavior of premixed gas flames near extinction or stability limits is not well known. Both the chemical reaction rates at near-limit conditions and the interaction of chemistry with transport processes such as convection, diffusion, and flame radiation are not fully understood. Understanding combustion under lean conditions is, however, critical to the design of efficient, clean-burning combustion engines. Also, knowledge of near-limit behavior of flames is necessary for the assessment of fire and explosion hazards in mine shafts, oil refineries, and chemical plants.

It has been known for some time that many near-limit combustion phenomena are influenced by buoyant convection. In the absence of buoyant convection, other transport mechanisms are emphasized. These include the unequal rates of diffusion of thermal energy relative to diffusion of molecular reactants (the Lewis number effect) as well as the spectral radiation emitted from the gaseous combustion products. This has motivated a number of recent experiments on near-limit flame propagation in a microgravity environment. Perhaps the most unusual of newly-observed flame phenomena are flame balls. A flame ball is a flame structure that consists of one or more steady, stationary, spherical flames, not supported by any sources of reactants or sink of products at its center, that evolve from an ignition source in a chamber filled with an initially quiescent combustible gas. Observed flame ball radii range from 0.1 to 1 cm. In a flame ball structure, fuel and oxygen diffuse from the surrounding gases inward to the reaction zone while combustion products and heat diffuse outward. The flame balls also lose some of their heat as emitted radiation.

Flame balls have a number of interesting properties. Since they are one-dimensional, they are the simplest possible type of premixed flame structure and therefore provide a useful testbed for theoretical and numerical models of the interaction between chemical and transport processes in flames near extinction. Some of these interactions are not predicted well even by the best currently available models. Flame balls may be relevant to turbulent combustion, as structures reminiscent of flame balls could be the prevalent ones in near-limit turbulent combustion of lean hydrogen-air mixtures. The drop tower and aircraft microgravity experiments indicated that a long duration and high quality of microgravity, available only in a space experiment, was necessary to observe the steady-state behavior of flame balls because they are predicted to evolve on the order of hundreds of seconds.

In the space experiments on STS-83 and STS-94, mixtures of hydrogen, oxygen, and a third component, either nitrogen, carbon dioxide, or sulfur hexafluoride, were ignited with a spark at the center of a combustion chamber and produced one to nine spherical, steady, stationary flame balls that lasted for up to 500 sec, when the experiment was ended. In addition to the gas sampling and analysis, measurements of flame shape, motion, radiant emission, temperature, and pressure, and the acceleration environment were made.

In order to use the gas chromatograph on-orbit, a commercial unit was modified to enable it to survive the environmental qualification tests and the safety requirements mandated for equipment used in human spaceflight. A commercial off-the-shelf gas chromatograph was procured and modified to interface with the CM-1 Fluids Supply Package and Combustion Chamber, accommodate two different carrier gases flowing through independent column modules, withstand the launch environment, accept Spacelab electrical power, and meet the requirements for electromagnetic interference and offgassing. This paper contains a summary of the flight qualification process and the on-orbit results. In addition, it contains a summary of additional gas chromatographic work conducted on the ground that was required to support the SOFBALL experiment.

COMBUSTION MODULE-1 INTERFACES TO GC

The CM-1 experiment consisted of a Spacelab single rack and a Spacelab double rack (fig. 1). The single rack contained the Fluid Supply Package that supplied the SOFBALL test mixtures and the gc carrier gases. The double rack contained the Experiment Package and the computers. The Experiment Package included the combustion chamber, the gas chromatograph, and the gas sampling subsystem. The gas chromatograph interfaced with the fluid supply system (fig. 2) for its carrier gases, the gas sampling subsystem (fig. 3) for calibration gas and gas sample inlet, and the combustion chamber, for gas sampling of the chamber contents. The location for the gas chromatograph was selected to be the extreme left of the double rack, next to the combustion chamber, in order to keep the length of the fluid lines to a minimum.

The gas chromatograph had an RS-232 serial interface data link with the Dedicated Experiment Processor Package (DEPP). The DEPP was the computer that controlled the CM-1 experiments and transferred data to and from the gas chromatograph (fig. 4).
The gas chromatograph received electrical power from the CM-1 Power Distribution Package (PDP). This assembly converted Spacelab electrical power to +12 VDC to a DC-DC Converter.

The SOFBALL Experiment was mounted in an Experiment Mounting Structure (EMS). The EMS was slid into the Combustion Chamber by the crew member and electrical connections were made manually to the EMS (fig. 5).

GAS CHROMATOGRAPH REQUIREMENTS

The science requirements for the gas chromatograph were to measure H₂, H₂O, CO, CO₂, O₂, N₂ and SF₆, from 0.1 to 100 percent with an accuracy of 2 percent of reading. One sample was to be extracted before the combustion test, after the gas filling process was completed. A second sample was to be extracted after the combustion test, after the combustion products had reached thermal and mechanical equilibrium.

The Engineering Requirements for the gas chromatograph were the following:

1. Maximum Physical Volume: 0.025 m³.
4. Vibration Requirements: Must function properly after being subjected, unpowered, to the vibration levels shown in figures 6(a) to (c). These vibration profiles represent the launch loads for flight qualification.
5. Thermal Requirements: Must function properly after being subjected to an unpowered thermal soak at +5 °C, a +50 °C hot soak and two +10 °C cold soaks involving a sequence of cold start power up tests, and a powered-up test subjecting the GC to nine (9) thermal cycles between +10 °C and +45 °C (fig. 7(a)).
6. Electromagnetic Interference (EMI) Requirements: Must not conduct or radiate electromagnetic interference to other electrical and electronic equipment on board the Space Shuttle, nor be susceptible to emi generated by other electrical and electronic equipment aboard the Space Shuttle.
7. Offgassing Requirements: Must comply with toxic offgassing requirements for missions aboard a manned space flight environment where crewmembers are present.

SPECIFICATIONS FOR A COMMERCIAL GAS CHROMATOGRAPH

In order to begin to meet the above requirements, a Micro Gas Chromatograph was procured that met the specifications listed in Appendix A. The MTI P200D Micro Gas Chromatograph was the gc selected. Among the most significant features were:

1. Physical Size: 15 cm (6 in.) H × 36 cm (14 in.) W × 36 cm (14 in.) D
2. Weight: 10.4 kg (23 lb)
3. Dual, Independent Channels
4. Injection Volume: Selectable 0.5 to 15 microliters
5. Detection Range: 1 ppm to 100% for most gases
6. Linear Dynamic Range: 10⁵ ±5%; 10⁶ ±10%
7. Separation Time: 140 sec or less
8. Repeatability: 2% RSD at constant temperature and pressure

MODIFICATION OF MICRO GAS CHROMATOGRAPH FOR FLIGHT QUALIFICATION

The Micro Gas Chromatograph was made flightworthy by having the following modifications made:

Remove and/or relocate the following items: (Refer to fig. 8)
1. Top Cover
2. Carrier Gas Bottle
3. Battery Pack

NASA/TM—1998-206965 3
4. Carrying Handle  
5. Printed Wiring Board (PWB)  
6. Rear Panel  
7. Support Bar  
8. Gas Knob  
9. Side Panels

Fabricate and install the following items: (Refer to fig. 9)

10. New Top  
11. New Bottom  
12. Support Bars for Side Panels  
13. PWB Mounting Bracket  
14. Modifications to Bezel  
15. EMI Gaskets  
   New Front Panel  
   New Rear Panel

Make the following additional modifications: (Refer to figs. 10 and 11)

16. Add four Rubber Feet  
17. Modify Manifolds  
18. Leak Test Internal Pump; Rework or Replace if Necessary  
19. Install EMI Gaskets  
20. Install Circuit Breaker  
21. Install Circular Connectors  
22. Install new Cable Assemblies  
23. Apply Scotchweld on PWB Connectors  
24. Install Ty-Wrap around internal Cables

The following checkout tests were performed after modification of the gas chromatograph was completed:

- Electrical Tests  
- Proof Pressure Tests  
- GC Functional Tests

SPACE FLIGHT QUALIFICATION TESTING

Four (4) Commercial Micro Gas Chromatographs were procured for the CM-1 Project. The first gc to be modified became the Prototype, or QUAL, Unit. The second gc became the Primary Flight Unit and the third gc became the Backup Flight Unit. The fourth gc was used for replacement parts when needed.

The Prototype Unit was subjected to the following Flight Qualification Environmental Tests:

1. Vibration Testing: A flight qualification vibration test was performed on the gas chromatograph to demonstrate that the unit would operate properly after being subjected to vibrations occurring during launch of the Space Shuttle. The gas chromatograph was installed on a vibration table and subjected to the vibration profiles shown in figures 6(a) to (c). The X-axis vibration is from top to bottom, the Y-axis vibration is from front to back, and the Z-axis vibration is from side to side. Since the gas chromatograph is unpowered during launch of the Space Shuttle, the test was performed with the gas chromatograph unpowered. Satisfactory performance of the gas chromatograph after the vibration test was demonstrated by having the gas chromatograph take an air sample and provide data to generate a chromatograph.
Results of the test are shown in figure 12 and demonstrated that, after a minor modification of applying Loctite to lock nuts, the Prototype gas chromatograph would survive the Space Shuttle launch environment.

2. Thermal Testing: In order to demonstrate that the Prototype gas chromatograph would perform properly during temperature extremes of the Spacelab environment, the gc was subjected to a thermal test following the thermal profile shown in figure 7(a). Thermocouples were placed on the gc as shown in figures 7(b) and (c).

Results of the test showed proper performance of the gas chromatograph during the functional tests performed, as shown on the thermal profile. Responses of the thermocouples are shown in figure 13.

3. Electromagnetic Interference (EMI) Testing: In order to demonstrate that the gas chromatograph does not generate electrical interference to other electrical and electronic equipment aboard the space shuttle, the Prototype gas chromatograph was subjected to several emi tests. Requirements for emi compliance are found in a specification generated by NASA Marshall Space Flight Center.

A Bulk Current Test (BC01) was conducted on the gas chromatograph to observe electrical peaks on the interface lines and to aid in correcting any problem areas during the other emi tests. There is no specification for the Bulk Current test, as it is a diagnostic tool used by the EMI Laboratory personnel to indicate which interface lines may be acting as antennas.

For conducted emissions, DC Power Bus Ripple (Test CE01), the narrowband conducted emissions on the DC power lines must not exceed 130 dBuA from 30 Hz to 2 kHz, decreasing to 80 dBuA at 20 kHz.

For conducted emissions, DC Power Bus RF (Test CE03), the narrowband conducted RF emissions on the DC power lines must not exceed the profile 80 dBuA from 20 kHz, decreasing to 30 dBuA at 50 MHz.

For radiated emissions, Electric Field (Test RE02), the narrowband radiated emissions must not exceed the profile 45 dBuV/m at 14 kHz, decreasing to 30 dBuV/m at 25 MHz, then increasing to 70 dBuV/m at 10 GHz. The broadband radiated emissions must not exceed the profile of 110 dBuV/m/MHz at 14 kHz, decreasing to 65 dBuV/m/MHz at 200 MHz, then increasing to 100 dBuV/m/MHz at 10 GHz.

For radiated emissions, AC Magnetic Field (RE04), at a distance of 1 m away from the gas chromatograph, the magnetic field strength must not exceed 130 dB above 1 picotesla from 50 Hz to 1 kHz, decreasing at 40 dB/decade to 63 dB above 1 picotesla at 50 kHz.

The above tests were performed on the Prototype gas chromatograph and the initial results showed that an in-line emi filter circuit and ferrite beads needed to be installed in the power lines.

Results of the tests after the modifications were made are shown in figures 14(a) to (i).

An emi susceptibility test was also performed to show that other electrical and electronic equipment within the CM-1 Spacelab racks would not affect the performance of the gas chromatograph. Results of the susceptibility test showed that other equipment would not interfere with correct performance of the gas chromatograph during the mission.

4. Offgassing: Hardware flown on a manned mission where crewmembers are present must be tested for offgassing. Offgassing is the evolution of gaseous products from a solid material or assembled article. Flight hardware flown in a manned environment must be free from offgassed noxious or toxic materials. An offgassing test was conducted on the CM-1 Experiment Package, the Flight Gas Chromatograph, and the Gas Sampling System to measure the level of offgassed materials and to verify that the concentration of offgassed materials were below the maximum allowable concentration values.

Criteria for an offgassing test is that the total Toxic Hazard Index (T) values for all volatile offgassed products must be less than 0.5. The T-Value is calculated by determining the ratio of the measured concentration of each offgassed product to the Spacecraft Maximum Allowable Concentration (SMAC) value for that product and summing the rations for all offgassed products.

The result of the offgassing test for the CM-1 Experiment Package, the Flight gas chromatograph, and the gas sampling system was a T-Value of 6x10⁻⁷.

GROUND GAS CHROMATOGRAPHY

A separate gas chromatograph was used on the ground to verify the gas mixtures prepared on the ground and loaded into the Fluid Supply Package bottles and to measure the presence and quantity of hazardous byproducts during reduced-gravity testing aboard the NASA aircraft. In addition, gas samples were taken after the space flight
from the Fluid Supply Package bottles for comparison to those measured prior to the flight. Finally, a gas chromatograph was used for a ground test to measure the selective leakage of hydrogen from the Fluid Supply Package bottles.

The gas mixtures for SOFBALL were made on the ground at the NASA Kennedy Space Center by the CM-1 team to exact specifications, because the flammability and the number of flame balls produced is extremely sensitive to the amount of fuel and oxygen present. The amount of each component was required to be precise to within ±2 percent of its target amount. The gas chromatograph served as a check of the composition after gravimetric preparation of the gas mixtures. The gas chromatograph used was an MTI Model P200D with two columns, a 10 m Molecular Sieve 5A PLOT and an 8 m PoraPLOT Q, with the carrier gases argon and helium, respectively.

The gases measured were hydrogen, oxygen, and either nitrogen, carbon dioxide, or sulfur hexafluoride as the diluent. Table A contains data from each of the mixture families for the test points with the most and least fuel. The table includes the target composition for the test point, the composition that was obtained during the gravimetric preparation of the gas mixture, the gas chromatography results for the source bottle and the gas chromatography results from the flight bottles, both pre- and post-flight.

The gravimetric results show, that with time and careful handling, these precise mixtures could be obtained. The gas mixing process will not be detailed here, except to say that initially a bottle containing a roughly 50:50 binary mix by weight of fuel and diluent was prepared and rolled for 60 hr. This was done so that the error from weighing a small amount of fuel in the presence of a large amount of oxygen and diluent would be reduced. The binary was then used to prepare the three component mix that was also rolled for 20 hr prior to gas sampling.

The pre-flight gas chromatography results confirm the gravimetric results, albeit with higher errors, with only one exception. The errors are within ±4 percent except for one of the carbon dioxide test points where it is believed that liquefaction occurring during the filling of the flight bottle led to higher fuel and oxygen amounts in the mixture. Data from the post-flight samples have higher errors because of contamination from an air leak in the gas chromatograph inlet line. We believe that the pre-flight errors could be reduced further with a more extensive set of calibration gases for each test mixture family because of the different responses for hydrogen and oxygen in the diluents. The sets used for these results were hydrogen in nitrogen and oxygen in nitrogen, oxygen in carbon dioxide, and oxygen in sulfur hexafluoride. Using calibration mixes with hydrogen in carbon dioxide and hydrogen in sulfur hexafluoride would allow a more complete set of calibration curves.

Some of the gas mixtures were expected to produce small amounts of hazardous byproducts, such as carbon monoxide, sulfur dioxide, and other acid gases. During testing aboard the NASA reduced-gravity aircraft, each gas mixture was ignited several times during consecutive parabolas. Then a sample of the gas was taken in a sample container and analyzed on the ground using a gas chromatograph. The byproducts detected were carbon monoxide, hydrogen sulfide, sulfur dioxide, and sulfonyl fluoride. The byproduct levels as measured by the gas chromatograph were used as supporting evidence during the stringent safety review process. The gas mixtures were allowed to be flown with the appropriate precautions taken, such as an on-orbit leak check prior to combustion of the hazardous points and a scrubbing system to remove acid gases prior to venting to space or access of the chamber by the crew.

Finally, a gas chromatograph was used to examine the long-term leakage of the gases from the Fluid Supply Package bottles. Several bottles were filled with test mixtures and samples were periodically taken from the bottles over a six month period. The gas chromatograph detected no preferential leakage of hydrogen from the bottles.

FLIGHT GAS CHROMATOGRAPHY

During the shortened STS-83 mission in April 1997, insufficient time was available to operate the gas chromatograph on-orbit. The STS-94 mission in July 1997 ran its entire 16 days during which 27 SOFBALL combustion events were attempted and 24 combustion events were ignited. A total of 309 runs were analyzed by the gas chromatograph. The operating conditions are listed in Table II. During mission operations, the sample data files were stored on hard disk cartridges and also downlinked to the ground for near-real-time analysis.

The pre-test gases were sampled in the chamber to ensure that the amount of each component was known prior to combustion. A total of seven runs, four at low detector sensitivity and three at medium sensitivity, were taken for
each test point. Although the gases were mixed and measured precisely on the ground, the filling of the flight bottles was performed from 2 to 11 months prior to the flight, depending on the individual bottle. The number and size of flame balls are extremely sensitive to the composition. The gas composition is also an input to the theoretical model used to predict the flame ball behavior.

Typical chromatographs obtained from the flight gas chromatograph early in the mission for pre-test mixtures are shown in figures 15 to 18. Figure 15 shows the species detected in a nitrogen test point on column A; figure 16 shows the species detected in a sulfur hexafluoride test point on column A; figures 17 and 18 show the species in a carbon dioxide test point on column A and B. The amplitude scale in figure 18 is such that the major species peaks are off-scale. Note also that the hydrogen, oxygen, and nitrogen fully overlap on this column. From these chromatographs, it is evident that even in these initial samples, the hydrogen and oxygen peaks are not fully resolved on column A. There is also contamination from test point to test point. Figure 16 shows nitrogen remaining from the test point 4 runs previous, and figures 17 and 18 shows sulfur hexafluoride remaining from the test point three and five runs previous. The flight gas chromatograph sample inlet ran for 255 sec on the first two runs and 10 sec for the runs thereafter. It is apparent that these sample times were insufficient to purge the previous test point from the inlet line.

After the combustion extinguished, gas sampling and analysis for the major reactant and product species was performed in order to determine what affected the duration of the flame balls lifetime. The objective was to determine whether flame balls could exist until a significant portion of the reactants in the chamber is depleted or do the flame balls extinguish spontaneously before fuel depletion becomes significant because of another influence. The composition indicates the extent of the reaction. If extinguishment occurs before the experiment timeout of 500 sec when the flames are forcibly extinguished, the gas composition measurement enables a determination of how much depletion of reaction occurs before the mixture becomes nonflammable, a property that can be compared to detailed computations. A total of seven runs as above was taken for each air and sulfur hexafluoride test points; each carbon dioxide test point had an additional three runs at high detector sensitivity to allow the detection of any carbon monoxide. The gas composition measurement was also desired for determining the initial composition of reburn mixtures. Occasionally, the gas mixture was sparked a second time and burned again. After the second burn, the gas was sampled the same as after the first burn. Table III contains the sample history for the first four test points described above and below.

Post-test (post-reburn) gas chromatographs are shown in figures 19 and 20 for a carbon dioxide test point. The hydrogen and oxygen peaks are still unresolved on column A, but the amount of sulfur hexafluoride detected is significantly reduced from the pre-test chromatograph seen in figures 17 and 18. By this time, an additional 1140 sec of sampling has occurred, thereby cleaning out the sample inlet line of sulfur hexafluoride.

After the STS-94 mission was concluded, the columns of the flight gas chromatograph were baked out on the ground. Gas chromatographs of the calibration gases were obtained and are shown in figures 21 to 23. The peaks are significantly sharper and could be clearly resolved.

From these results, we conclude that the gas chromatograph operated successfully during the entire mission and can be considered an engineering success. The unit twice survived the launch into space and conducted all its operations as requested. It is because of the constraints imposed upon it by the CM-1 hardware, systems, and operations that the gas chromatograph was unable to measure the pre- and post-combustion gases to the required accuracy. Under the proper conditions, it is expected that the flight gas chromatograph would perform as well on-orbit as on the ground, and as well as the ground gas chromatograph used in the gas mixing and filling.

FUTURE OUTLOOK

It is anticipated that the SOFBALL experiment will be flown again within the next several years. Based on their experience, the Combustion Module project is planning several improvements. These would be to increase the column length to 10 m, add the capability for an on-orbit bake-out, modify the sample inlet system to have a vacuum vent, and have a more complete calibration. With these improvements, the gas chromatograph performance would be greatly enhanced.
APPENDIX A—SPECIFICATIONS FOR A COMMERCIAL MICRO GAS CHROMATOGRAPH

1. The Micro Gas Chromatograph being procured shall be:
   (a) a portable, self-contained dual gas chromatograph with two (2) independent modules containing the auto-
       injectors, analytical and reference columns, column heaters, and solid-state detectors.
   (b) capable of supplying its own electrical power for eight (8) hours or more, and supplying its own carrier gas
       for forty (40) hours or more.

   Features must include:

   • Two (2) independent column heater zones
   • Independent control of auto-injectors, column heaters, column head pressure, and detector sensitivity
   • Built-in autosampling of gases with internal pump
   • Silicon micromachined injector system, volume selectable from 20 to 1000 nL, independently controlled for
     each column
   • Built-in carrier gas cylinder; D.O.T. certified to 1800 psig.

2. The Micro Gas Chromatograph System shall be capable of performing analysis of C1 to C8 n-hydrocarbons, air
   and carbon dioxide in eighty (80) seconds or less.
3. Maximum Weight: 11 kg (23 lbs)
4. Maximum Volume: 0.025 m³
5. The Micro Gas Chromatograph System shall contain two (2) gas-chromatographic subsystems with independent
   injectors, detectors, and column ovens for each GC subsystem. The internal GC subsystems shall allow column,
   or column module, interchange, for both GC subsystems, by the user, to allow analysis of different gas mix-
   tures. The system shall allow independent control of auto-injectors, run times, and each column oven tempera-
   ture range.
6. Injection System: Built-in manual, or automatic, injection; using computer-controlled sampling pump. User-
   selectable injection volumes from 20 to 1000 nL. The injector system shall allow either continuous flow of the
   sampled gas stream through the sampling valve, or shall be able to draw a discrete sample from a vessel using
   the internal sampling pump.
7. Detection range: Shall be able to detect most gases from 1 ppm (part per million) to 100 percent, with the appro-
   priate column module.
8. Linear Dynamic Range: $10^5 \pm 5$ percent; $10^6 \pm 10$ percent
9. Column Heater Range: Independently controllable temperatures for each column with control range of 30 to
   180 °C in isothermal operation, or better.
10. Carrier Gas: Argon (Ar) for Column A; Helium (He) for Column B. Internal Carrier gas Bottle shall be config-
    ured for Column B (Helium).
11. Power: built-in rechargeable battery pack; providing up to eight (8) hours of continuous operation before requir-
    ing recharge.

   Voltage: +12 VDC at 30 W, maximum.

12. Environmental: Shall be moisture and dust resistant. Shall be able to operate from 0 to 50 °C. Shall have rugged
    packaging.
13. The Micro GC System shall have a column module capable of providing separation of N₂, O₂, H₂, CH₄, and
    CO, using a 4 m, 5 Å Molecular Sieve Packed Column (Column A) and Argon (Ar) carrier gas. The Micro GC
    System shall have a second column module capable of separating C1-C5 Hydrocarbons, CO, Water vapor, SF₆,
    H₂S, and SO₂, using an 8 m PoraPlot Q Packed Column (Column B), and Helium (He) carrier gas. Separation
    shall be achieved within 140 sec or less. Modules shall be capable of being replaced in the field by the user.
14. The Sample Inlet shall be installed on the Rear Panel of the Gas Chromatograph System.
15. Repeatability: 2 percent RSD at Constant Temperature and Pressure, or better.


REFERENCES


<table>
<thead>
<tr>
<th>TABLE I.— SUMMARY OF GAS MIXING AND FILLING RESULTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percent</td>
</tr>
<tr>
<td>hydrogen</td>
</tr>
<tr>
<td>4.00</td>
</tr>
<tr>
<td>4.00</td>
</tr>
<tr>
<td>3.91</td>
</tr>
<tr>
<td>3.88</td>
</tr>
<tr>
<td>3.45</td>
</tr>
<tr>
<td>3.38</td>
</tr>
<tr>
<td>3.35</td>
</tr>
<tr>
<td>3.36</td>
</tr>
</tbody>
</table>

| Target | 5.20 | 10.40 | 84.40 CO \(_2\) |
| Gravimetric | 5.20 | 10.42 | 84.38 |
| GC Source | 5.34 | 10.37 | 84.29 |
| GC FSP pre | 5.40 | 10.55 | 84.05 |
| GC FSP post | 5.16 | 9.96 | 84.88 |

| Target | 4.60 | 9.20 | 86.20 CO \(_2\) |
| Gravimetric | 4.62 | 9.19 | 86.19 |
| GC Source | 4.74 | 9.21 | 86.05 |
| GC FSP pre* | 4.94 | 9.57 | 85.49 |
| GC FSP post | 4.64 | 8.87 | 86.49 |

| Target | 7.00 | 14.00 | 79.00 SF \(_6\) |
| Gravimetric | 7.00 | 14.03 | 78.97 |
| GC Source | 7.18 | 13.98 | 78.84 |
| GC FSP pre | 7.12 | 13.95 | 78.93 |
| GC FSP post | no sample | no sample | no sample |

| Target | 6.50 | 13.00 | 80.50 SF \(_6\) |
| Gravimetric | 6.51 | 12.99 | 80.50 |
| GC Source | 6.66 | 12.81 | 80.53 |
| GC FSP pre | 6.61 | 12.77 | 80.62 |
| GC FSP post | 6.38 | 12.25 | 81.37 |

* Slight liquefaction of diluent is suspected during the filling that would lead to higher fuel and oxygen amounts in the gas mixture.
TABLE II.—OPERATING CONDITIONS DURING STS-94 FOR THE GAS CHROMATOGRAPH.

<table>
<thead>
<tr>
<th></th>
<th>Column A</th>
<th>Column B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Column type</td>
<td>Mole Sieve 5A, 4 m</td>
<td>Pore Plot U, 4 m</td>
</tr>
<tr>
<td>Carrier gas</td>
<td>argon</td>
<td>helium</td>
</tr>
<tr>
<td>Column head pressure</td>
<td>20.5 psig</td>
<td>26.2 psig</td>
</tr>
<tr>
<td>Column temperature</td>
<td>49 °C</td>
<td>69 °C</td>
</tr>
<tr>
<td>Instrument gain</td>
<td>low, medium, high</td>
<td>low, medium, high</td>
</tr>
<tr>
<td>Sample time</td>
<td>10, 255 s</td>
<td>10, 255 s</td>
</tr>
<tr>
<td>Inject time</td>
<td>50 ms</td>
<td>50 ms</td>
</tr>
<tr>
<td>Run time</td>
<td>160 s</td>
<td>160 s</td>
</tr>
</tbody>
</table>

TABLE III.—SAMPLE HISTORY OF THE GAS CHROMATOGRAPH FOR THE FIRST FOUR TEST POINTS ON STS-94

<table>
<thead>
<tr>
<th>Run number</th>
<th>Test point</th>
<th>Diluent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-5</td>
<td>Calibration Gas</td>
<td>Nitrogen</td>
</tr>
<tr>
<td>6-12</td>
<td>03A Pre-test</td>
<td>Nitrogen</td>
</tr>
<tr>
<td>13-19</td>
<td>03A Post-test</td>
<td>Nitrogen</td>
</tr>
<tr>
<td>20-26</td>
<td>14A Pre-test</td>
<td>Sulfur hexafluoride</td>
</tr>
<tr>
<td>27-33</td>
<td>14A Post-burn</td>
<td>Sulfur hexafluoride</td>
</tr>
<tr>
<td>34-40</td>
<td>14A Post-test</td>
<td>Sulfur hexafluoride</td>
</tr>
<tr>
<td>41-47</td>
<td>13A Pre-test</td>
<td>Sulfur hexafluoride</td>
</tr>
<tr>
<td>48-54</td>
<td>13A Post-burn</td>
<td>Sulfur hexafluoride</td>
</tr>
<tr>
<td>55-61</td>
<td>13A Post-test</td>
<td>Sulfur hexafluoride</td>
</tr>
<tr>
<td>62-68</td>
<td>10A Pre-test</td>
<td>Carbon dioxide</td>
</tr>
<tr>
<td>69-78</td>
<td>10A Post-burn</td>
<td>Carbon dioxide</td>
</tr>
<tr>
<td>79-88</td>
<td>10A Post-test</td>
<td>Carbon dioxide</td>
</tr>
</tbody>
</table>

Figure 1.—CM-1 experiment in spacetab racks.
Figure 2.—Gas chromatograph fluids interfaces.
Figure 3.—GC gas sample supply system.

Figure 4.—CM-1 data flow from gas chromatograph to downlink.
Figure 5.—CM-1 experiment. (a) SOFBALL experiment mounting structure (EMS). (b) Crewmember installing EMS into CM-1 combustion chamber.
Figure 6.—GCU random vibration qualification test spectrum. Spacelab Payload Accommodations Handbook requirements +3dB and workmanship axis spectral density plot.
Figure 7.—CM-1 gas chromatograph prototype thermal test profile.

Figure 8.—Commercial microgas chromatograph with one top cover removed.
Figure 9.—CM-1 gas chromatograph system flight unit fabricated parts.

Figure 10.—CM-1 gas chromatograph system flight unit. Top view (cover and PWB omitted for clarity).
Scotchweld applied to all connectors

Figure 11.—CM-1 gas chromatograph system flight unit. Top view (cover omitted for clarity).

Figure 12.—Results of the CM-1 flight vibration test. Note: this table presents the sequence in which test runs were conducted. Run #’s are designations for test run identification on data plots.
Figure 13.—Results of the CM-1 flight qualification thermal test. Chamber air (TC1), temperature vs time.
(a) Module A (TC2). (b) Module B (TC3). (c) Pump motor (TC4). (d) Analog case (TC5).
Figure 13.—Continued. (e) Intel chip (TC8). (f) Voltage regulator (TC7). (g) Bottom enclosure (TC8). (h) Enclosure top (TC9). (i) Upper support (TC10). (j) Lower support (TC11).
Figure 14(a-i).—EMI test results conducted emissions, DC power bus. Results of radiated emission field (computer on, GC on, pump on).
Figure 14(j).—Test setups for EMI tests. (a) S (RE02). (b) BC01. (c) RE04. (d) CE01 & CE03.

Figure 14(k).—Additional EMI tests setups. (a) Overal test setup. (b) Breadboard filter on connector.
2.5
SB03A gravimetric
Hydrogen-3.52%
Oxygen-19.81%
Nitrogen-75.49%

2.0
Hydrogen-3.52%
Oxygen-19.81%
Nitrogen-75.49%

1.5

O2-19.81%

N2-75.49%

1.0

0.5

0.0

-0.5

-1.0

Time, sec

Figure 15.—Column A gas chromatograph of hydrogen-oxygen-nitrogen test point prior to combustion.

3.52
SB14A gravimetric
Hydrogen-6.51%
Oxygen-12.99%
Sulfur hexafluoride-80.50%

3.0
H2-4.51%
O2-unresolved

2.5

SF6-82.46%

N2-not measured

2.0

1.5

1.0

0.5

0.0

-0.5

-1.0

Time, sec

Figure 16.—Column A gas chromatograph of hydrogen-oxygen-sulfur hexafluoride test point prior to combustion.

2.25
SB10A gravimetric
Hydrogen-4.62%
Oxygen-9.19%
Carbon dioxide-86.19%

2.0

1.5

1.0

0.5

0.0

-0.5

-1.0

Time, sec

Figure 17.—Column A gas chromatograph of hydrogen-oxygen-carbon dioxide test point prior to combustion.

1.25
SB10A gravimetric
Hydrogen-4.62%
Oxygen-9.19%
Carbon dioxide-86.19%

1.00

0.75

0.50

0.25

0.00

-0.25

-0.50

-0.75

Time, sec

Figure 18.—Column B gas chromatograph of hydrogen-oxygen-carbon dioxide test point prior to combustion. (The amplitude is off scale for the composite peak of hydrogen, oxygen, and nitrogen, and the peak for carbon dioxide.)
Figure 19.—Column A gas chromatograph of hydrogen-oxygen-carbon dioxide test point after a reburn (second burn for the same mixture).

Figure 20.—Column B gas chromatograph of hydrogen-oxygen-carbon dioxide test point after a reburn (second burn for the same mixture).

Figure 21.—Column A gas chromatograph of hydrogen-nitrogen and oxygen-nitrogen calibration gases taken post-flight after bakeout.

Figure 22.—Column A gas chromatograph of oxygen-sulfur hexafluoride calibration gases taken post-flight after bakeout. The amplitude is off-scale for the peak for sulfur hexafluoride.

Figure 23.—Column B gas chromatograph of oxygen-carbon dioxide calibration gases taken post-flight after bakeout.
**Title:** Application of a Modified Gas Chromatograph to Analyze Space Experiment Combustion Gases on Space Shuttle Mission STS-94

**Authors:** William K. Coho, Karen J. Weiland, and David M. VanZandt

**Performing Organization:**
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135-3191

**Sponsoring/Monitoring Agency:**
National Aeronautics and Space Administration
Washington, DC 20546-0001

**Abstract:**
A space experiment designed to study the behavior of combustion without the gravitational effects of buoyancy was launched aboard the Space Shuttle Columbia on July 1, 1997. The experiment, designated as Combustion Module - 1 (CM-1), was one of several manifested on the Microgravity Sciences Laboratory - 1 (MSL-1) mission. The launch, designated STS-94, had the Spacelab Module as the payload, in which the MSL-1 experiments were conducted by the Shuttle crewmembers. CM-1 was designed to accommodate two different combustion experiments during MSL-1. One experiment, the Structure of Flame Balls at Low Lewis-number experiment (SOFBALL), required gas chromatography analysis to verify the composition of the known, premixed gases prior to combustion, and to determine the remaining reactant and the products resulting from the combustion process in microgravity. A commercial, off-the-shelf, dual-channel micro gas chromatograph was procured and modified to interface with the CM-1 Fluids Supply Package and the CM-1 Combustion Chamber, to accommodate two different carrier gases, each flowing through its own independent column module, to withstand the launch environment of the Space Shuttle, to accept Spacelab electrical power, and to meet the Spacelab flight requirements for electromagnetic interference (EMI) and offgassing. The GC data was downlinked to the Marshall Space Flight Center for near-real time analysis, and stored on-orbit for post-flight analysis. The gas chromatograph operated successfully during the entire SOFBALL experiment and collected 309 runs. Because of the constraints imposed upon the gas chromatograph by the CM-1 hardware, system and operations, it was unable to measure the gases to the required accuracy. Future improvements to the system for a reflight of the SOFBALL experiment are expected to enable the gas chromatograph to meet all the requirements.

**Keywords:** Chromatography; GC; Fuels; Instrument development

**Security Classification:** Unclassified

This publication is available from the NASA Center for AeroSpace Information, (301) 621-0390.