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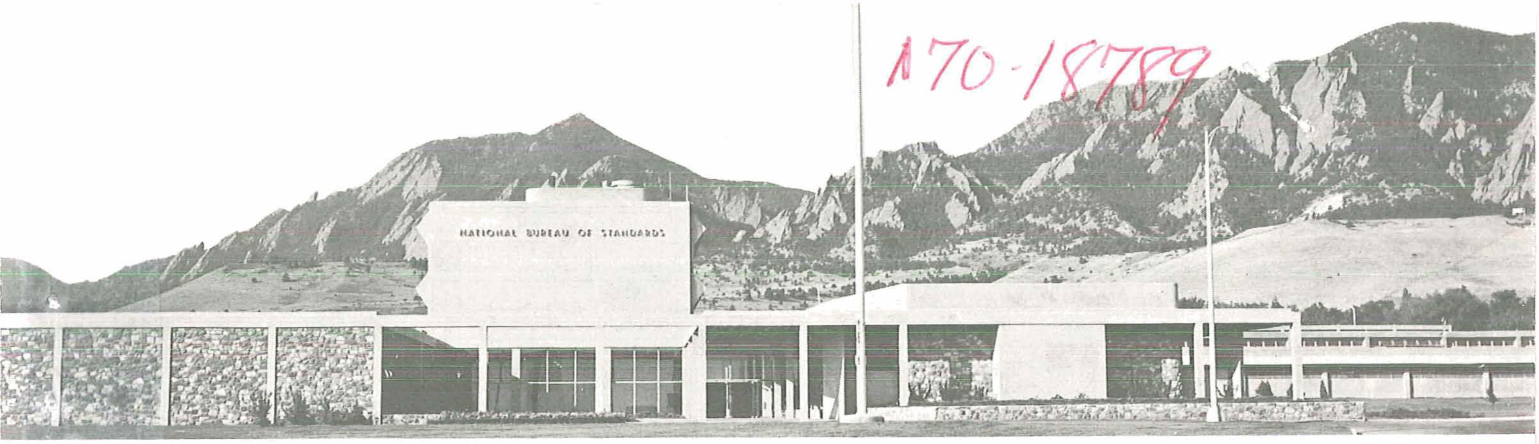

NBS REPORT

9738

R-06-006-007

SLUSH AND SUBCOOLED METHANE CHARACTERIZATION

CASE FILE
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C. F. Sindt, P. R. Ludtke, H. M. Roder, and N. A. Olien

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NATIONAL BUREAU OF STANDARDS
Institute for Basic Standards
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SLUSH AND SUBCOOLED METHANE CHARACTERIZATION

C. F. Sindt, P. R. Ludtke, H. M. Roder, and N. A. Olien

Cryogenics Division
Institute for Basic Standards
National Bureau of Standards
Boulder, Colorado 80302

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ABSTRACT

Methane gas of 99 percent purity was condensed to liquid methane, and slush was subsequently produced using the freeze-thaw method. An apparatus was assembled which allows solid particles to be photographed in triple-point liquid with a reference grid in the background. Fresh and aged solid particles were photographed. Photos of the particles indicate a definite aging effect between fresh and 4-hour-old particles. Particles of two different size ranges appeared during the production process. Bibliographies on methane and methane mixtures have been compiled. A tabulation of the areas where thermodynamic properties for methane are available was prepared from the bibliography and is presented. The table is supplemented with a skeletal temperature-entropy diagram showing the regions where insufficient property data are available.

Key Words: Cryogenic fluid; liquefied natural gas; liquid-solid mixtures; methane; natural gas; slush; thermodynamic properties.

SLUSH AND SUBCOOLED METHANE CHARACTERIZATION*

C. F. Sindt, P. R. Ludtke, H. M. Roder, and N. A. Olien

1. Introduction

The Cryogenics Division of NBS-Institute for Basic Standards is currently involved in an experimental study to characterize liquid-solid mixtures (slush) of methane and natural gas and to compile thermodynamic properties of methane and other natural gas constituents. The program is entitled "Slush and Subcooled Methane Characterization Study." The general scope of the program is to determine production techniques for slush methane and natural gas, to study aging effects on solids, to determine transfer and pumping characteristics, to develop required instrumentation, and to compile the thermodynamic properties of methane and its mixtures with other natural gas components.

Since the beginning of the program, a bibliography on methane has been updated and another bibliography on methane mixtures has been prepared. These bibliographies were used in compiling data on the solid, liquid, and gaseous states from triple point (90.7 K, 11.6 kN/m^2 pressure [Din, 1961]) to 203 kN/m^2 pressure. An experimental apparatus has been assembled to produce and observe subcooled liquid and slush methane, and several experiments have been conducted in this apparatus with 99 percent pure methane. Also, available data on constituents of natural gas wells and pipe lines have been compiled [Mann and Hall, 1969].

2. Experimental Apparatus

A schematic and a photograph of the experimental apparatus is shown in figures 1 and 2. The apparatus consists basically of two concentric glass dewars suspended from top plates. The inner experimental dewar is 16 cm inside diameter.

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*This work was carried out at the National Bureau of Standards under the sponsorship of NASA-Headquarters, Washington, D. C.

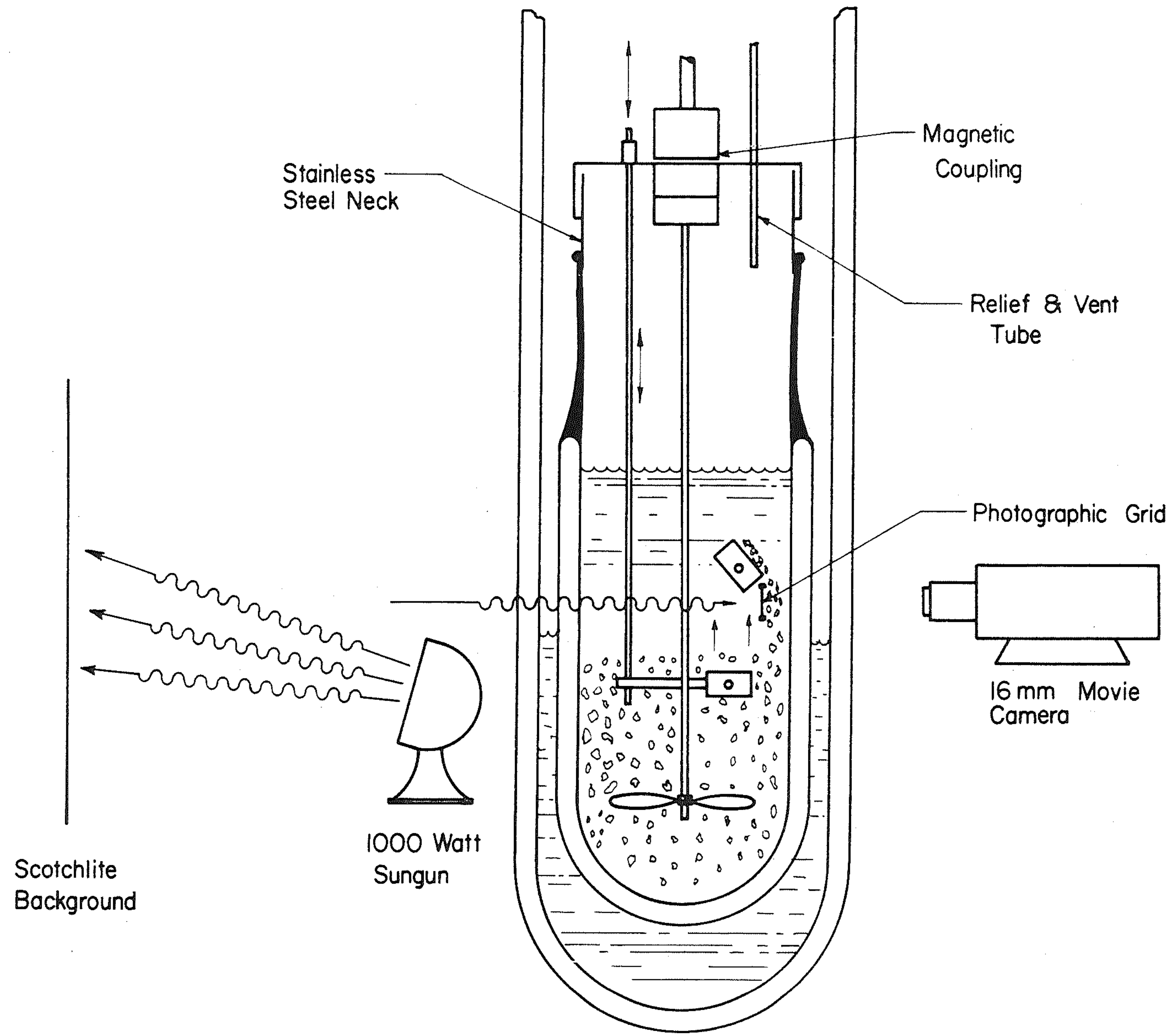

Figure 1. Experimental Apparatus

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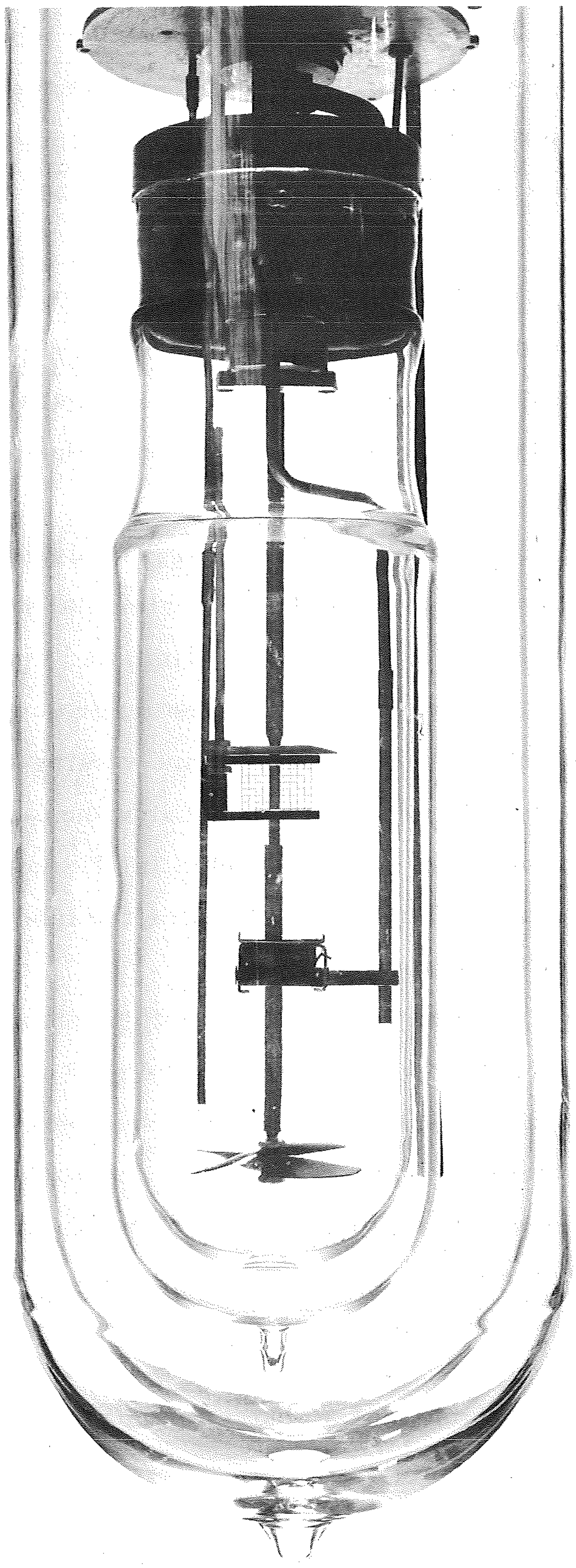

Figure 2. Experimental Dewar

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The shielding dewar has a glass flange at the top which is clamped to a top plate and sealed with a Teflon® gasket. The top plate contains a tube for filling the shielding dewar with liquid nitrogen, a vent or pressure relief line, and a tube for pressurizing the dewar with any desired gas.

The lower part of the inner experimental dewar is vacuum insulated. The top of the dewar is uninsulated Pyrex® glass which is bonded to a 1-mm-thick stainless steel neck as shown in figure 1. The stainless steel neck is soldered to a brass plate which seals the inner dewar from the outer shielding dewar.

The brass plate has a magnetic coupling mounted in the center which provides a rotary drive inside the experimental dewar without using a dynamic rotary seal. Two tubes of 1mm and 5 mm inside diameter also pass through the brass plate into the inner dewar. These tubes are used for introducing gas into the dewar and venting and pumping the dewar. A 3-mm-diameter rod is installed inside the larger tube to provide a means for obtaining vertical movement inside the experimental dewar. There is a dynamic O-ring seal between the tube and the rod in an ambient temperature location.

The experimental dewar contains a photographic grid, an elevator, and a stirring propeller. The elevator and grid are used for examining solid particles in the slush. Figure 3 shows the grid with the elevator in a lowered position. The glass grid is approximately 1.8 cm by 2.8 cm by 1 mm thick and has 2.3-mm squares photographically etched on the surface. It is rigidly mounted to a small tube and sets approximately 5 mm from the inside surface of the dewar. The top of the grid

® Pyrex-Corning Glass Works, Scotchlite-3M Co., Teflon-E. I. du Pont de Nemours & Co., Trademarks. Precise specifications in this paper of the material employed has been necessary to make the results of the work sufficiently meaningful. Identification of the material or its manufacturer by the National Bureau of Standards in no way implies a recommendation or an endorsement by the Bureau.

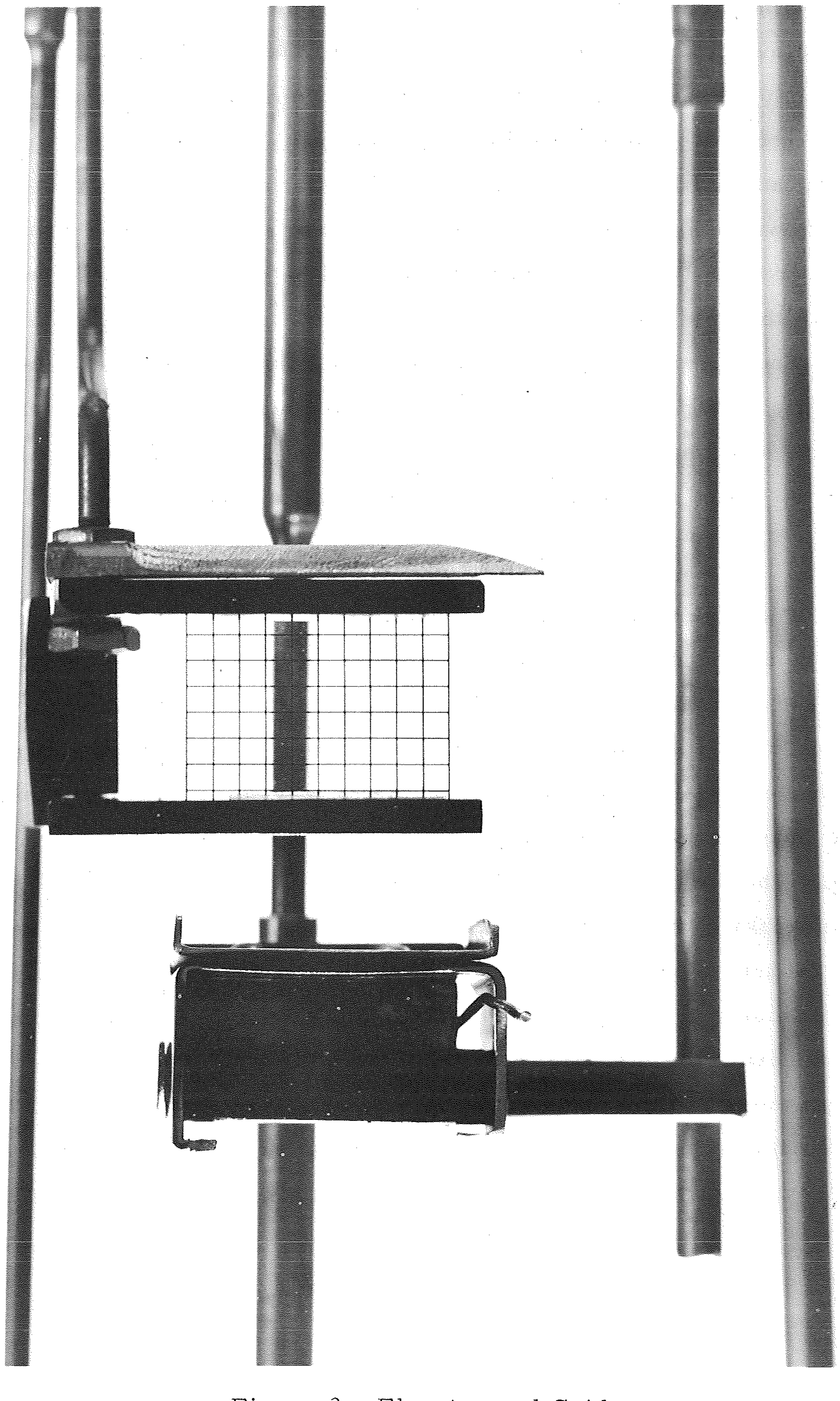

Figure 3. Elevator and Grid

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mount is beveled to direct the particles in front of the grid as they slide off the elevator. The elevator is attached to the 3-mm-diameter rod so that it may be moved up and down. This movement allows an operator to lower the elevator into the solid, bringing solid to the top of the grid where it automatically tips, dumping solid particles in front of the grid as is shown in figure 4. The solid particles are photographed as they pass in front of the grid so particle size can be determined by comparison with the grid size.

The stirring propeller is powered with an externally mounted pneumatic motor. The drive linkage goes through hermetically sealed magnetic couplings at the top of the experimental dewar and the shield dewar top plate, eliminating the need for rotating seals.

3. Experimental Procedure

The experimental dewar is evacuated and purged with nitrogen gas to remove the air, followed by evacuation and purging with methane gas. The outer dewar is evacuated and purged twice with nitrogen gas and then filled with liquid nitrogen. During the methane condensing phase the nitrogen level is maintained near the stainless steel-to-glass joint on the inner dewar in order to condense methane inside the experimental dewar. The nitrogen in the shielding dewar is vented at 21 kN/m^2 gauge (3 psig) making the temperature of the shielding bath approximately 76 K. Since the triple-point temperature of methane is 90.7 K [Din, 1961], the condensing procedure must be closely controlled. It is very important to prevent the methane inlet tubes from freezing shut, which could occur if the brass top plate of the experimental dewar were cooled below the methane triple-point temperature. Moreover, some surface of the inner dewar has to be at or below the boiling temperature in order to condense the methane gas, which requires that the 76 K shielding liquid be near or above the vacuum insulated portion of the experimental dewar.

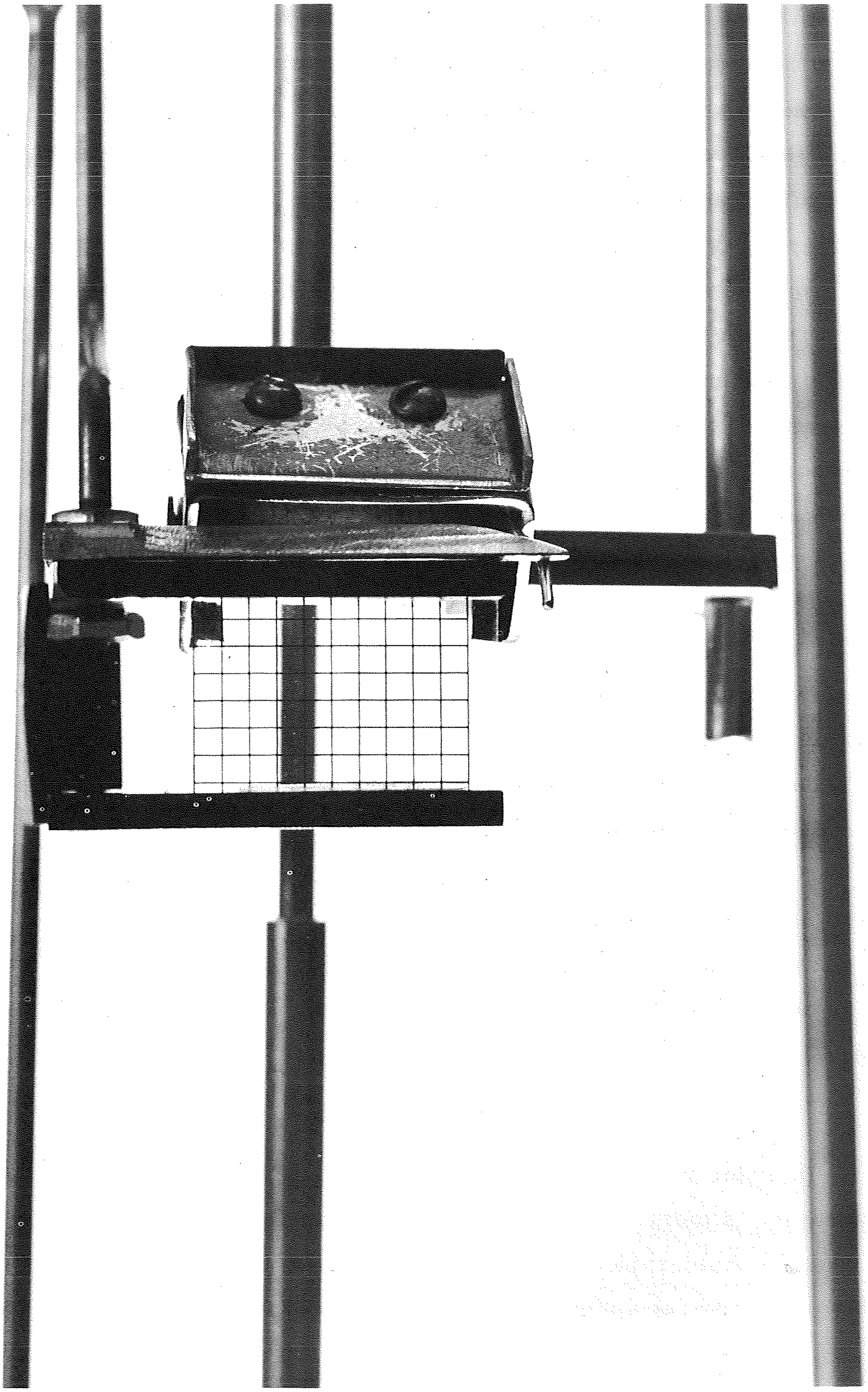

Figure 4. Elevator in Dumping Position

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In the experiment, it was found that the methane gas does condense at an acceptable rate ($\approx$ 1 dm^3/10 minutes) when the liquid nitrogen is maintained at the prescribed level. After condensing methane to the desired liquid level in the experimental dewar, the methane gas supply is shut off. The nitrogen liquid level is lowered to a point just below the grid where it is maintained for the remainder of the experiment, during which time the nitrogen is used only as a shield for the methane liquid.

The surface above the methane is then pumped with a 7.08 dm^3/s (15 CFM) vacuum pump until solid begins to form on top of the liquid. Usually very little pumping is required since the liquid is already near triple-point temperature (90.7 K). More pumping would be required if the methane were not precooled with nitrogen. Methane slush is then made by the freeze-thaw process in the following manner: (1) The surface of the liquid is pumped to the triple-point pressure, 11.6 kN/m^2 (87.7 mm Hg) [Din, 1961]; (2) Pumping is continued until a solid layer forms on the top of the liquid; and (3) The pumping is then stopped and soon the solid layer melts at the dewar walls and settles into the liquid. A slight rise in ullage pressure may occur after the solid is immersed in the liquid. Stirring at this time helps to break the solid and encourages settling. The cycle is then repeated. The slush becomes more dense and the solid particles no longer tend to settle out of the liquid. The dewar is then closed and the slush is aged. Very little melting occurs because of the thermal shielding of the nitrogen bath in the outer dewar.

4. Particle Observation

In order to observe and measure the individual particles or groups of particles, a method previously developed to photograph solid hydrogen was used to photograph the methane particles in triple-point liquid. A sketch of the photographic arrangement is included in figure 1. A 16-mm movie camera is focused immediately in front of the grid. A 1000-watt sungun is directed on Scotchlite® and the reflected light provides backlighting for the grid. In order to take pictures, the slush level must be below the grid, as the solid particles diffuse the reflected light. The particles are photographed in the following manner. The elevator is lowered into the solid to pick up the solid particles. It is then raised above the slush level, through the triple-point liquid to a point at which it trips on the top of the grid as shown in figure 4, and dumps the solid particles in front of the grid. The camera is turned on immediately before the particles fall in front of the grid. The depth of field of the camera is approximately 3.3 mm so the particles near the grid are the only ones in focus.

5. Experimental Results and Conclusions

Methane gas of 99 percent purity was condensed to provide liquid methane for the two tests conducted to date. Immediately after condensing the desired amount of liquid, the methane was pumped to the triple point. It was observed that the triple-point pressure was very close to 11.6 kN/m^2 (87.7 mmHg). The temperature of the liquid was not measured.

When producing slush, it was observed that the solid layer which formed on top of the liquid was more friable than either nitrogen or hydrogen. The methane solid seemed to have very little strength and quickly settled into the liquid.

From the few tests made to date, it appears that the solid particles fall into two different size ranges. One group of particles appears to be in the range of 2 mm and larger, and when stirred, these particles settle within a short time (15 seconds) to the bottom of the dewar. Another much smaller group of particles, probably 0.2-mm diameters and smaller are present immediately after forming solids. These particles are still in suspension after all the larger particles have settled to the bottom of the dewar, and tend to remain in suspension for 2 to 3 minutes before settling to the bottom with the larger particles. It should be pointed out that the particle sizes mentioned above are estimates. No attempt has been made to date to take a representative sample and measure particle sizes.

At this point there is speculation that the smaller particles may be due to the 1 percent impurity in the methane gas. Similar small particles have been observed with a gas mixture of 4.4 mole percent oxygen in nitrogen [Sindt, et al., 1969]. Future tests with 99.97 percent purity gas should indicate if the small particles are methane or carbon dioxide, one of the most prevalent impurities in the methane gas.

A definite aging effect was observed between the fresh and aged methane particles. A photo of the fresh particles falling in front of the grid is shown in figure 5, and a similar photo of particles aged four hours is shown in figure 6. The fresh particles are agglomerates of an intricate network of very small crystals loosely attached. The much smaller particles that have a significantly lower settling rate, as previously mentioned, are not clearly visible in the figure because of their small size. After aging the slush for four hours within a nitrogen shield, the slush was stirred with the propeller. The slush stirred very easily and there appeared to be no tendency for the solid particles to freeze together and form a large clump in the bottom of the dewar. However, the heat leak to the particles with nitrogen shielding is extremely low. In the aged solids it appears that some of the crystals grow at the expense of others and the particle becomes an agglomerate of spherical-shaped crystals. The very small particles that could be observed in the fresh slush during manufacture and after stirring were not apparent in

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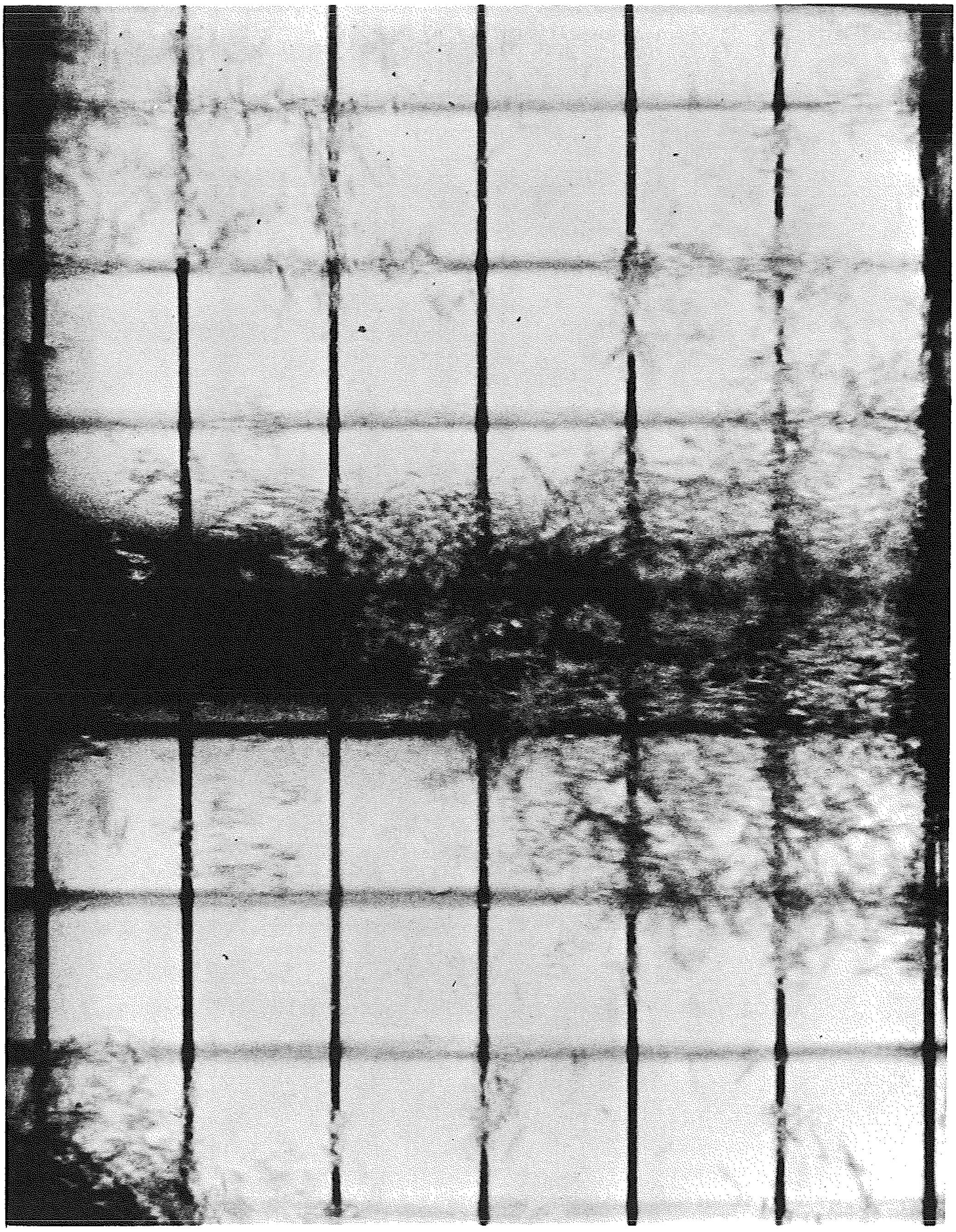

Figure 5. Slush Methane--Age 15 Minutes

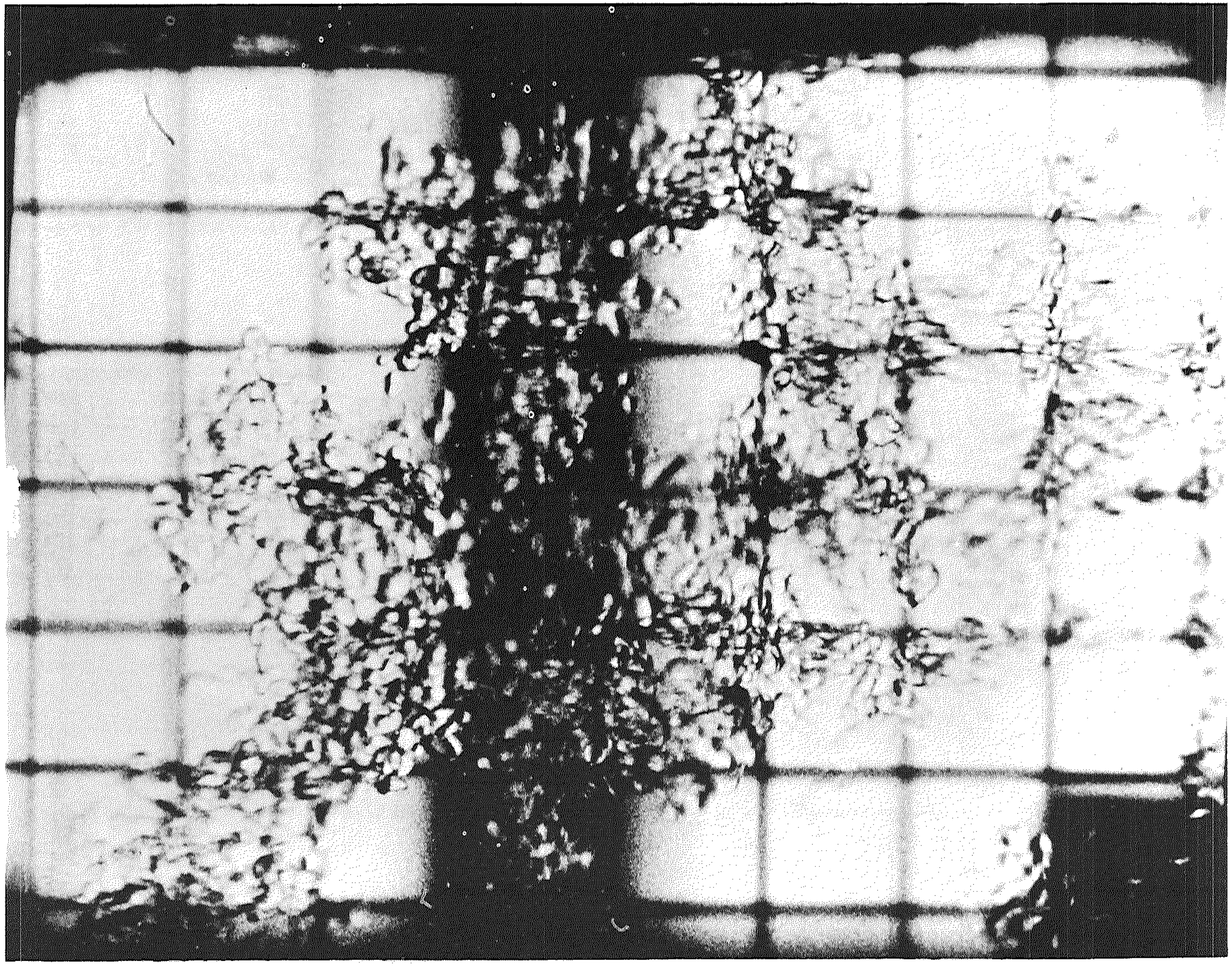

Figure 6. Slush Methane--Age 4 Hours

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the aged slush. They are either trapped in the growing solid crystals or are melted in the aging process. More aging tests of longer duration will be conducted to further characterize this aging effect.

Another observation made was that after saturating the liquid methane with nitrogen gas, there was a definite tendency for the liquid to foam at the surface when the surface was pumped. However, if no nitrogen gas had been introduced into the dewar there appeared to be no tendency for the condensed liquid methane to foam while pumping to the triple point or while producing slush. Indications from the few tests to date are that nitrogen may be responsible for a foaming tendency. More investigation will be conducted to determine the effect of nitrogen and other impurities in methane.

6. Methane Thermofunctions

6.1 Objective

The objective of this portion of the project is to select from the world's literature the best physical and thermodynamic data for methane and to evaluate it and use it to compile tables and charts as required for this slush methane studies program. Data on the solid, liquid, and gaseous states, from the triple point to 203 kN/m^2 (2 atm.) pressure, are to be compiled.

6.2 General Approach

The comprehensive bibliography on the properties of methane prepared by the Cryogenic Data Center's bibliographic service has been updated with current literature that has appeared recently. All pertinent data will be extracted from the literature and fitted to suitable thermodynamic functions to be used in generating the required data. Since there is a dearth of experimental data in some of the regions to be covered, the best methods of estimating the probable values of the properties in such areas will be used.

6.3 Bibliographies

For the past ten years the Documentation Group of the Cryogenic Data Center has been maintaining an awareness of the published and report literature on the properties of fluids and solids of interest in low temperature applications. This literature is regularly procured, cataloged and coded into the Data Center's mechanized storage and retrieval system. As a result, comprehensive bibliographies can be produced quickly and at modest cost. Since the articles have been coded in depth by material, property, and conditions, extensive property indexing of a search is also possible.

A formal annotated bibliography on the thermophysical properties of methane was issued under a prior NASA contract (R-06-006-046). This task was based on a search of the storage and retrieval system and yielded bibliography B-179, which listed 940 references of which 660 were selected for publication [Hall, 1968]. Two bibliographies* were prepared as the first step in this program: 1) Bibliography B-446, "The Thermophysical Properties of Methane in the Solid, Liquid, and Gaseous Phases;" and 2) Bibliography B-449, "The Thermophysical Properties of Methane Mixtures." Bibliography B-446 is an updating of B-179, which lists 990 references, and is believed to be a reasonably complete coverage of the world literature in this specific subject area.

6.4 Properties and Thermodynamic Functions

Industry has considerable interest right now in the properties of methane and its mixtures, particularly as these apply to liquefied natural

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*Copies may be ordered from the Cryogenic Data Center, National Bureau of Standards, Boulder, Colorado 80302.
Bibliography B-446, "The Thermophysical Properties of Methane in the Solid, Liquid and Gaseous Phases," 215 pp (May 1969) price $7.00.
Bibliography B-499, "The Thermophysical Properties of Methane Mixtures," 109 pp (May 1969) price $5.00.

gas (LNG). As an example, the Cryogenics Division was asked to present a paper on LNG at the recent conference of the American Gas Association [Mann and Roder, 1969] . A few of the conclusions presented in this paper are of interest here.

1. The analysis of the available densities of saturated liquid methane is inadequate.
2. Experimental data for the dielectric constant of saturated liquid methane are scarce, those for ethane nonexistent.
3. Compressed liquid densities are required for a slush methane temperature-entropy chart, but are not available. A recent inquiry requested liquid densities for ethane near the normal boiling point of methane. Values are apparently not available [Hiza] .

The properties required and those available for a methane temperature-entropy chart in the slush region are given in table 1. Considering the correlations presently in print, Din [1961] has done the best evaluation of the data available. Using table 1 and the values given by Din, a skeleton diagram shown in figure 7 was constructed. The dashed lines and dotted areas show graphically the areas of missing data.

The problem can be divided into two parts; 1) estimating values in the compressed liquid region (region I in figure 7) up to and including the liquid-solid boundary, and 2) estimating values in the two-phase liquid-solid region (region II in figure 7) up to and including the compressed solid boundary. To get values of derived properties such as entropy we need a pressure, volume, temperature (PVT) surface. The extrapolation of a rudimentary PVT surface was attempted first. The assumption was made that the lines of constant volume are straight, thus they have an intersection temperature, T_o, and a slope, α, in a P-T plane. Values for T_o and α as given by Van Itterbeek, et al. [1963] were extrapolated graphically. The volume obtained in this manner for the pressure and temperature of the triple point is 2.224 cm^3/g, whereas,

Table 1

Properties Required For A Slush Methane T-S Chart

Vapor Pressure to Triple Point	Available	
Triple Point to Critical Point	Available	
Triple-Point Temperature	Available	
Boiling Point Temperature	Available	
Melting Pressures	Available	
Sat. Liquid Density	Available	
Sat. Vapor Density		?
Below Triple Point		?
Freezing Liquid Density (Liquid/Solid)		Not Available
Sat. Solid Density (100% Solid)		Not Available
Compressed Liquid Density		Not Available
Sat. Solid Density (Vapor/Solid)	Available	
Volume Change on Fusion (Triple Point)	Available	
At Elevated Pressures		Not Available
Heat Capacity of Solid	Available	
Heat of Fusion (Triple Point)	Available	
At Elevated Pressures		Not Available
Heat Capacity of Sat. Liquid	Available	
Heat of Vaporization		
(At a Single Pressure)	Available	
At Other Pressures		Not Available
Ideal Gas Properties	Available	

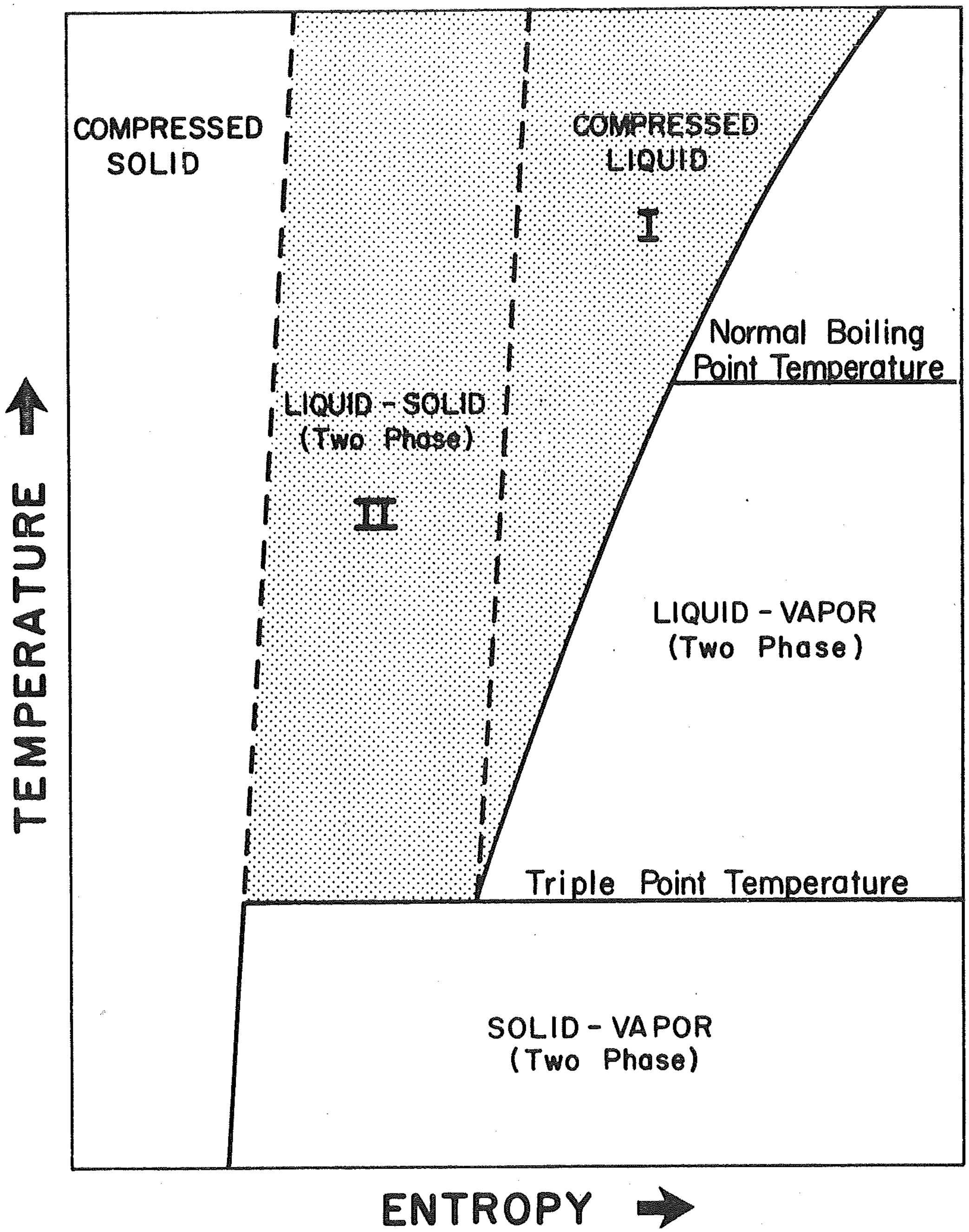

Figure 7. Skeleton Temperature-Entropy Diagram for Methane

the actual value according to Din [1961] is 2.216 cm^3/g. The difference of 1 part in 350 is not significant in terms of volume or density, however, the equivalent error in pressure is quite large. We estimate that at a pressure of approximately 23.3 MN/m^2 (230 atm) along the melting line, the error equivalent to ± 1/350 in density is ± 7.1 MN/m^2 (70 atm). Furthermore, if an entropy increment is calculated from this rudimentary equation of state at a temperature of 95 K, then the slope of the liquid-solid boundary is negative, i.e., physically not realistic. This extrapolation is obviously not satisfactory. An extrapolation of entropy increments as given in Van Itterbeek [1963] will be tried next. After that the plan is to look at the extrapolation of an equation of state, such as that used by Weber [1968] for the compressed liquid states. It may turn out that a publication by Vennix [1966], presently in the process of being printed, has already solved the first part of the problem. The second part of the problem will have to be solved by scaling from the knowledge of other gases, such as nitrogen [Din, 1961] and hydrogen [Bartholome, 1936; Dwyer, et al., 1965; and Sindt and Mann, 1966].

7. Future Work

During the next reporting period continued effort will be made to use existing data and techniques to arrive at a better definition of the properties of liquid methane especially along the freezing line. Experimental effort will be directed toward effects of long duration aging of liquid-solid mixtures. The effects of small amounts of impurities on solid particle formation and size will be determined by using condensed 99.97 percent pure methane gas as the basis of comparison. Characteristics related to foaming of boiling liquid will be investigated using the high purity methane as a reference.

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