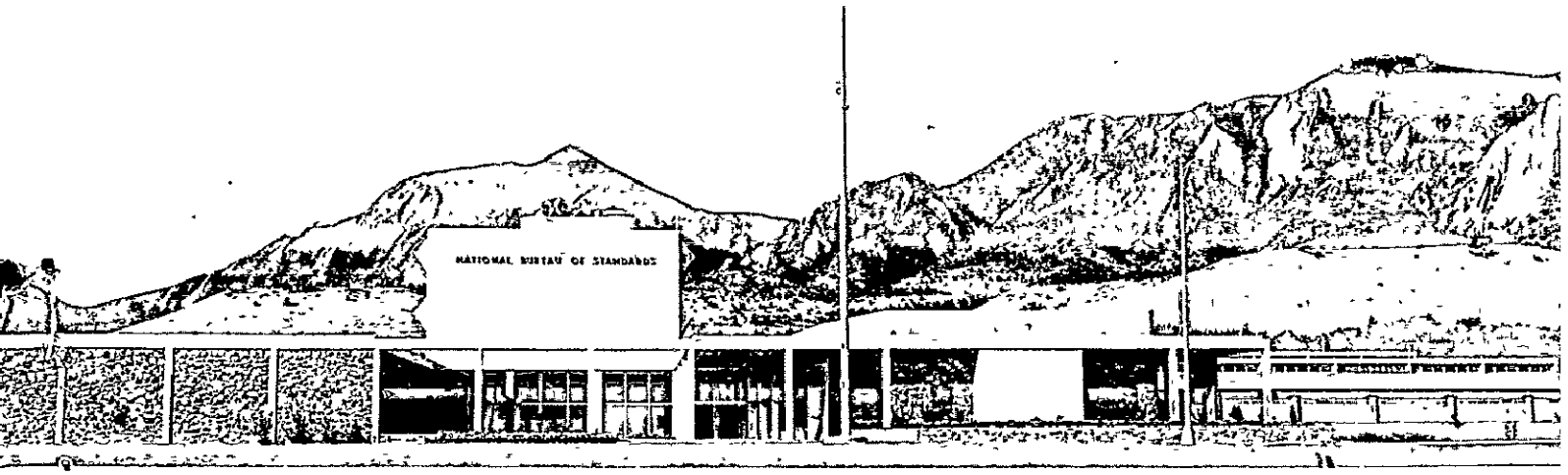

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NBS REPORT

9745

SLUSH HYDROGEN FLUID CHARACTERIZATION AND INSTRUMENTATION

C. F. Sindt and P. R. Ludtke

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
Institute for Basic Standards
Boulder, Colorado 80302

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ABSTRACT

Areas of slush hydrogen fluid characterization and instrumentation covered are transfer, storage, and density measurement.

A technique of increasing solid content of slush hydrogen in a storage vessel was devised and proven. Retaining solids with a 30-mesh screen while drawing off triple-point liquid was the method used. Solid fraction was upgraded to 0.54 in a vessel by this method, using slush less than 2 hours old. This slush remained fluid enough to mix and transfer. Solid fraction as high as 0.64 was achieved in slush that had been upgraded and then aged 7 hours.

Two types of nuclear radiation attenuation densitometers were used to determine the density of the upgraded slush. Two beta ray point sensors which were submerged in the fluid were tested. Data from these sensors are compared with data from an externally mounted gamma ray densitometer.

Evaluation was made of two types of mixers in the upgraded slush. Two propeller mixers were tested. A single propeller either did not stir high solid fraction slush or did not give adequate mixing in the 234 cm (92 in.) deep vessel. Three equally spaced propellers on a single shaft provided good mixing.

A ducted propeller design with suitably located discharge ports in the duct gave good homogeneous mixing in slush of solid fractions in excess of 0.5.

Temperature stratification in the liquid hydrogen over settled solids was measured in the upgrading vessel and the data are presented.

Key Words: cryogenics; densitometer; liquid solid mixtures; mixing; nuclear radiation; slush hydrogen; temperature stratification.

SLUSH HYDROGEN FLUID CHARACTERIZATION AND INSTRUMENTATION*

C. F. Sindt and P. R. Ludtke

1. Introduction

The Cryogenics Division of the NBS Institute for Basic Standards is currently involved in an analytical and experimental study to characterize liquid-solid mixtures of hydrogen. This program is entitled "Slush Hydrogen Fluid Characterization and Instrumentation Analysis." The general scope of the program is to develop knowledge and understanding of production techniques, storage, and transfer of slush hydrogen.

Previous to work reported here, equipment and techniques have been developed to produce, transfer, pump and store slush. Instrumentation to measure solid particle size, to determine density, to measure flow losses in transfer lines and restrictions, and to determine friction angles of slush on metal surfaces has been developed and utilized. Also, slush hydrogen has been gelled.

Increased density and reduced fuel loss from evaporation are the principal advantages of liquid-solid mixtures of hydrogen over normal boiling liquid. For these advantages to be fully realized the solid content of slush must be at the maximum that is usable. To achieve maximum concentrations of solids in space program applications, the mass solid fraction will need to be continuously or periodically increased in ground or orbital storage as well as in flight vessels. Also, certain applications

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*This work was carried out at the National Bureau of Standards under the sponsorship of the NASA-Marshall Space Flight Center, Huntsville, Alabama.

may require that the solids be retained in the vessel while the liquid is used, thus taking full advantage of the increased heat capacity of the slush.

Current studies involve the investigation of a technique for increasing the mass solid fraction in a storage vessel, referred to as upgrading, and of methods to stir the resulting liquid-solid mixture to maintain a homogeneous mixture. Combined with these experiments are experiments to evaluate new density measuring devices and to measure thermal stratification in the liquid over the settled solids.

2. Solid Fraction Upgrading Facility

The test facility constructed for upgrading and mixing experiments was an addition to the existing slush hydrogen flow facility described by Sindt, et al. [1969]. A schematic of the system is shown in figure 1. The addition included the upgrading dewar and its associated liquid and vent lines.

The upgrading dewar was an existing vacuum insulated vessel. It has a capacity of about 1 m^3 (35 ft^3), has 0.89 mm (0.035 in.) stainless steel walls and is 76 cm (30 in.) in diameter. Total depth is 234 cm (92 in.). Attached to the vessel at approximately 101 cm (40 in.) from the bottom is a copper thermal radiation shield 0.8 mm (0.032 in.) thick. This shield completely encloses the inner vessel below the attaching point. The top plate is flange mounted and has 7.6 cm (3 in.) of polystyrene foam insulation attached to the bottom side. Figure 2 is a cross section showing the assembly. Located in the top plate are two 10-cm (4-in.) glass windows for visual and photographic access. These windows are of double pane construction with a vacuum space between. This type of construction is used primarily for safety purposes. In the event that the cold window fails, while the dewar is at a pressure below atmospheric, the warm window provides the seal against air contaminating the hydrogen. Also located in the top plate is an access port 23 cm (9 in.) in diameter.

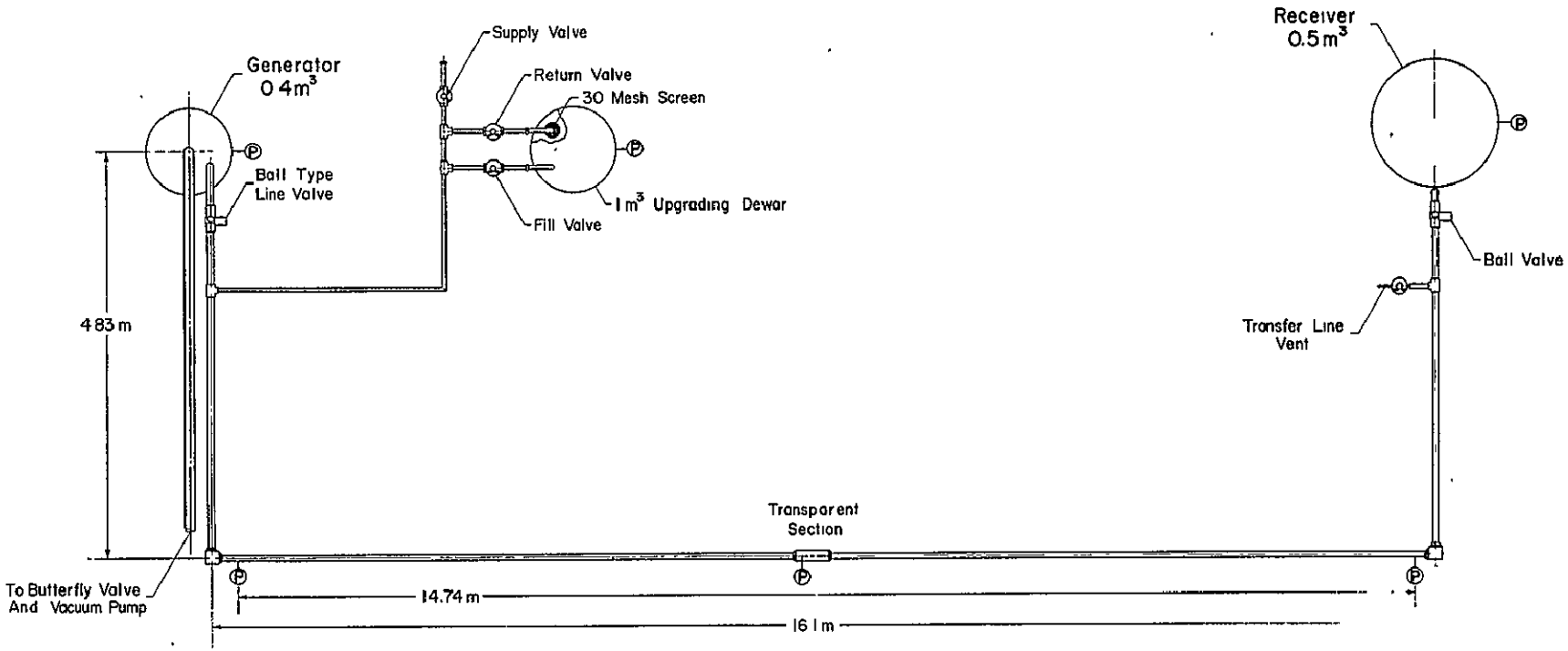

Figure 1. Schematic of Upgrading Facility

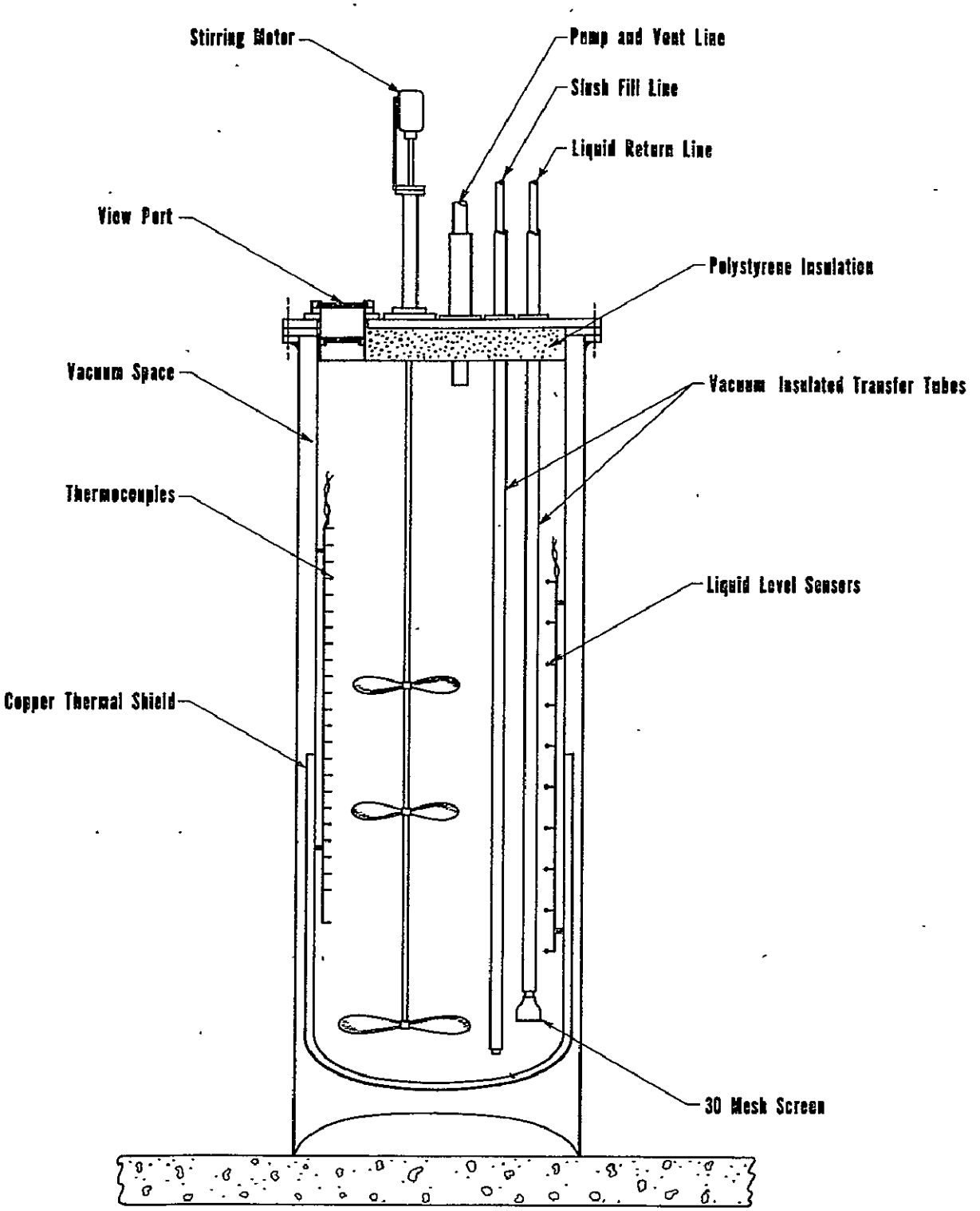

Figure 2. Cross Section of Upgrading Dewar

This port is used for installation and mounting of various mixing and instrumentation devices. The slush fill and liquid return lines also enter through the top plate. A one and one-half inch vent line is located in the center of the top plate. Venting may occur through this line via a 10 psi relief valve or by a remotely operated vent valve followed with a check valve. The vent is also connected to the vacuum pumps used for slush generation. A manual valve is used in this line for flow control. This system can be used to pump the hydrogen in the upgrading dewar to triple-point pressure if desired.

The transfer lines, shown in figure 1, connecting the generator, the upgrading dewar, and the liquid hydrogen supply dewar are 1-inch copper or stainless steel tubing and are all vacuum insulated. The line in the generator enters at a bottom sump. A ball valve controls flow in this section. Two lines connect to the line from the generator and both lines enter the upgrading dewar through the top plate and extend to near the bottom of the dewar. These lines have vacuum insulated valves with operators remotely controlled. The line for slush filling is open on the end. The bottom of the liquid return line flares to 8.9 cm (3-1/2 in.) in diameter and is covered with a 30-mesh screen with openings of 0.6 mm (0.023 in.). Figure 3 shows the screen assembly.

The transfer line continues beyond the slush fill and liquid return line to the liquid supply valve and to the supply dewar. With this arrangement liquid from the supply dewar can be used to precool the transfer line from the fill and return valves to the ball valve prior to slush flow. The cooling fluid is deposited in the receiver dewar.

Slush for the experiments is made in the generator, which has a capacity of approximately 0.4 m^3 (14 ft^3), by the freeze-thaw production method reported by Mann, et al. [1966]. Batches of fresh slush with solid fraction of 0.45, in quantities of 0.35 m^3 (12.3 ft^3) can be made in about

Figure 3. Slush Screen

30 minutes. The solid fraction of the slush can be ascertained using a nuclear radiation attenuation densitometer as described by Sindt, et al. [1969].

Upgrading the solid fraction in the system is accomplished by retaining solids in the dewar with the screen while drawing off liquid. The upgrading technique consists of four steps: 1. Slush is produced in the generator to the desired solid fraction. 2. The transfer line is precooled with liquid from the supply dewar. 3. The slush is transferred into the upgrading dewar through the fill line. 4. The triple-point liquid is drawn off through the return line.

3. Instrumentation

3.1 Gamma Ray Densitometer

A density measuring instrument was needed for the upgrading dewar tests. A nuclear radiation attenuation (NRA) densitometer was supplied by the program sponsor. This unit was originally designed to attach to a 27.9 cm (11 in.) diameter, vacuum insulated liquid hydrogen line. It uses a 17 curie cesium^{137} gamma source mounted on a shutter control assembly. This unit, which is shown in figure 4, is referred to as the fast response (F.R.) densitometer. The source holder, which appears as a ball shape in the figure, attaches to the shutter mount with pins and is, therefore, removable for safe storage.

The sensing unit consists of two ion chambers and an electrometer. This unit is shown in figure 5. The signal from the electrometer is carried to a signal conditioning unit via 91 m (300 ft) of coaxial cable. The control panel for the densitometer is shown in figure 6. Power requirements are 28 volt direct current as well as 120 volt alternating current. The 28 volt power supply is shown in figure 6 as well. The signal conditioning unit has two output channels. One channel is to drive a recorder, the other is to drive a digital voltmeter. The output signal has

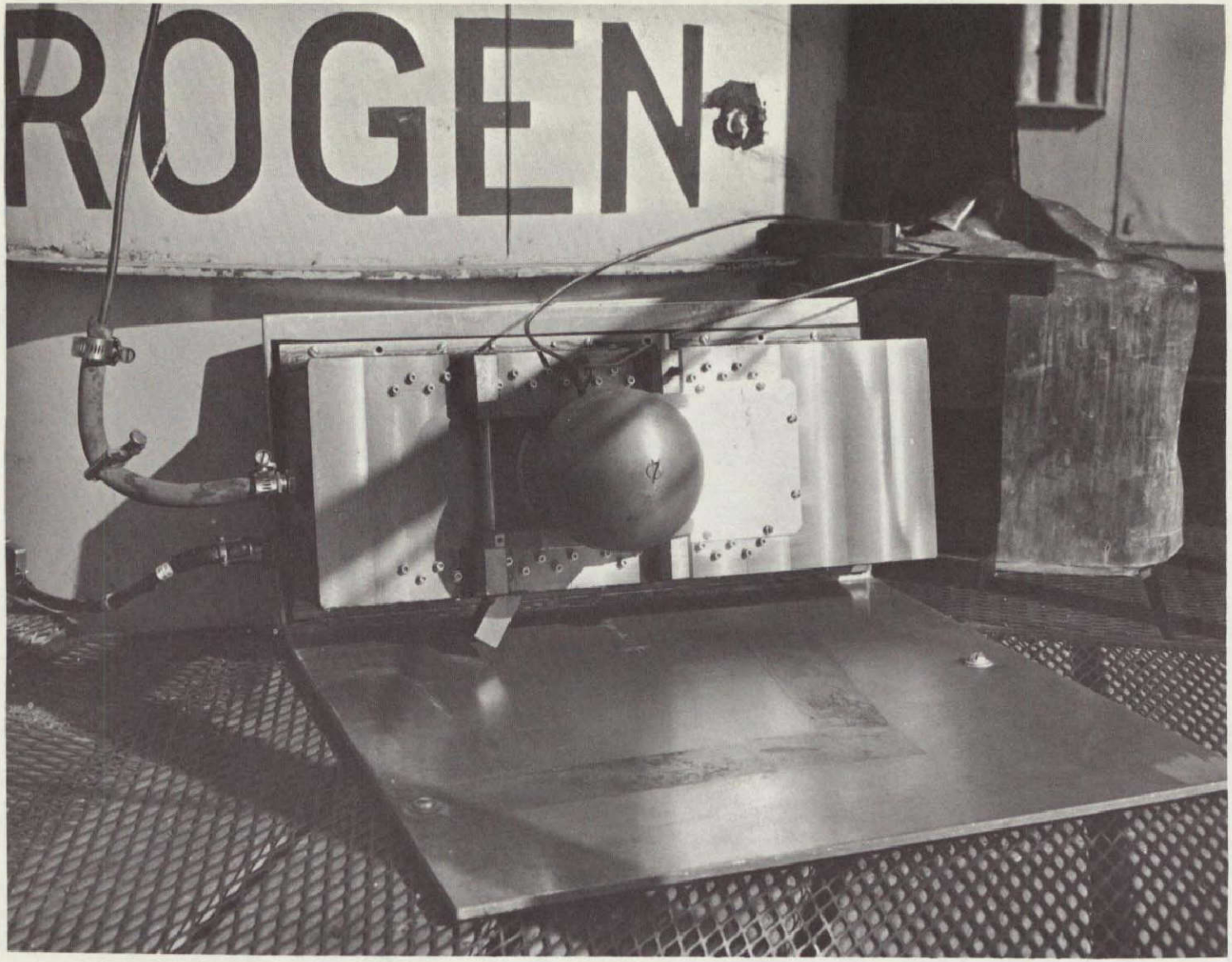

Figure 4. Densitometer Source Assembly

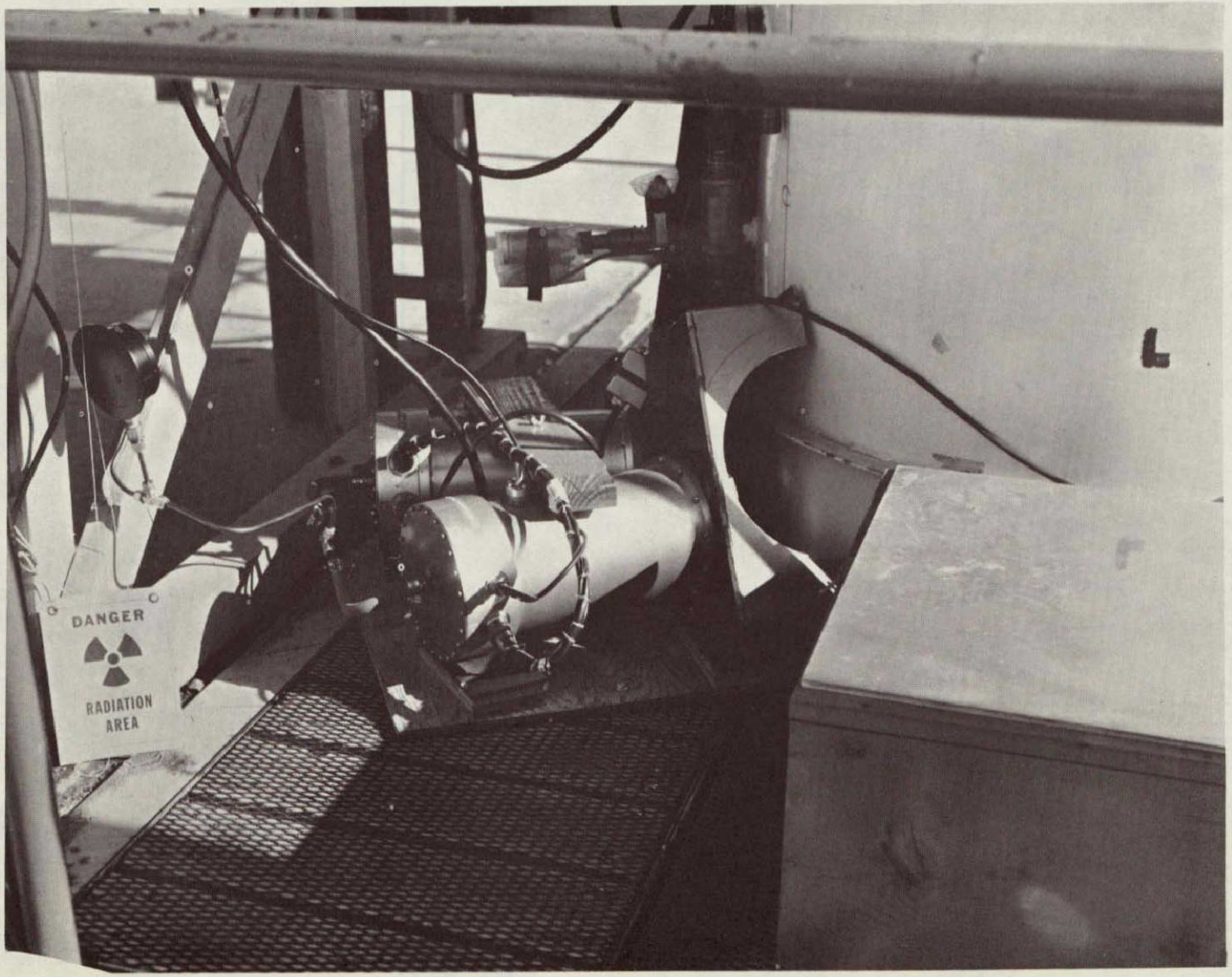

Figure 5. Densitometer Sensor Assembly

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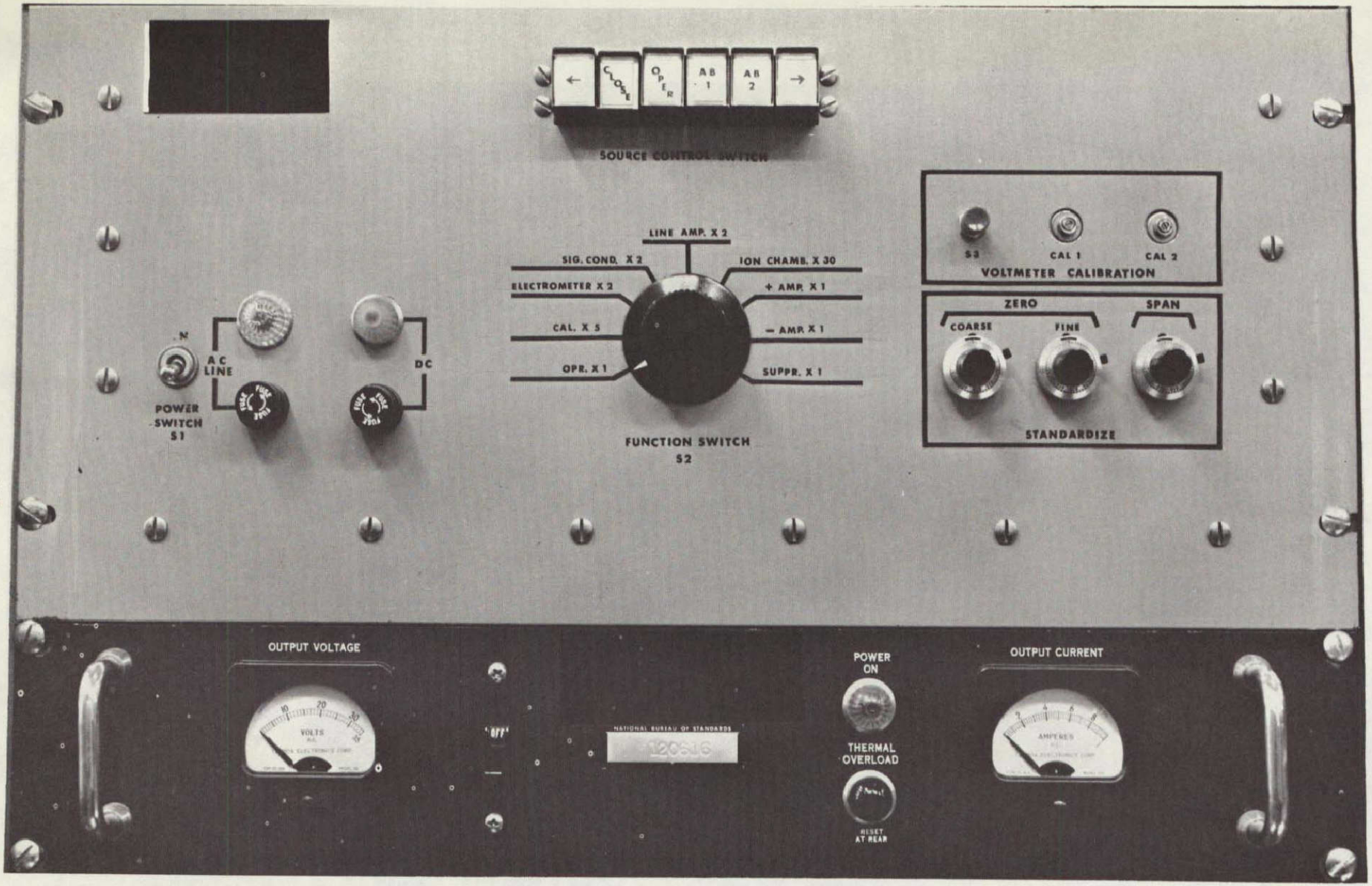

Figure 6. Densitometer Control Panel

a range from minus 10 volts to plus 10 volts. The design specifications for this unit call for an accuracy of ± 2% of the span and 0.1 second response time for a 95% of span change. These specifications are for a 25.4 cm (10 in.) diameter liquid hydrogen transfer line using liquid with a density range of 65 kg/m^3 (4.06 lb_m/ft^3) to 71 kg/m^3 (4.43 lb_m/ft^3).

3.1.1 Densitometer Modifications

Since the densitometer was designed to be used on a transfer line, modifications were required to use it on the upgrading dewar and the generator. Part of the mounting assembly was modified so that it could be mounted on the platform surrounding the generator as is shown in figures 4 and 5. Since the distance between source and sensor was greater than the original design intended, only one opening in the shutter was used. The radiation from this opening covered both ion chambers. Because the attenuation in the generator was much greater than would be experienced in the transfer line the resistor in the electrometer was changed from the existing 30 MΩ to 513 MΩ. This resistance was higher than the optimum, but was dictated by the availability of a good quality resistor. Because the resistance was too great, an absorber was used to reduce the output signal.

3.1.2 Tests of the Gamma Ray Densitometer

The program requirements for a densitometer for the upgrading dewar were essentially the same as those for the densitometer on the generator. For this reason a comparison test between a 17 curie source densitometer and a 4 curie source densitometer described by Weitzel, et al. [1968] was made on the generator. The uncertainty of the 4 curie source unit has been estimated to be ± 0.4% [Sindt, et al., 1969]. No attempt was made to test the new unit's response time or its precision other than to make the comparison test. Figure 7 is the data taken with

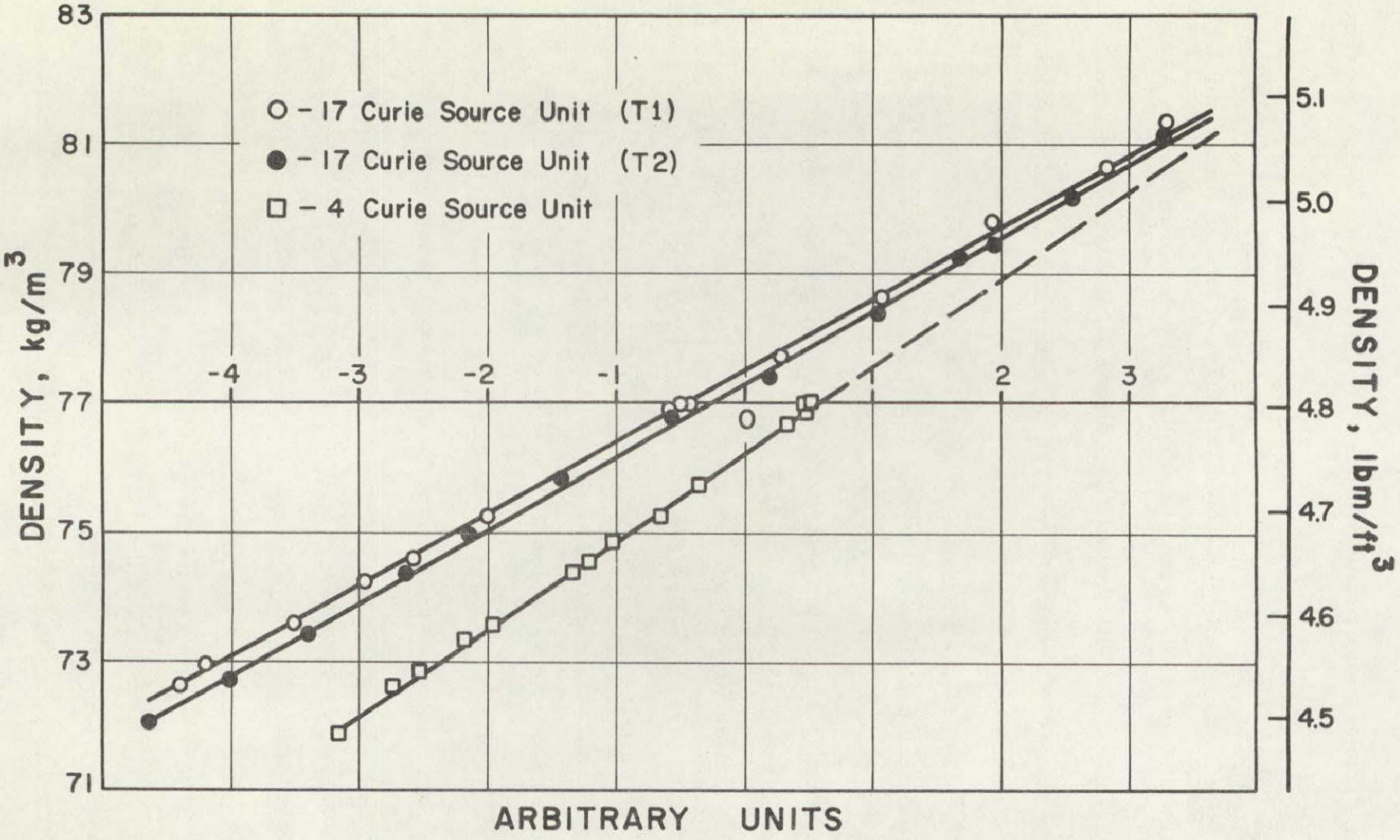

Figure 7. Densitometer Calibration Data

both units. In the liquid phase from normal boiling point to triple point, the vapor pressure over thoroughly agitated liquid was measured. Density was then determined using the properties of saturated liquid from Roder, et al. [1965]. In the slush region the density measured by the 4 curie source unit was used to plot the points for the F.R. densitometer. Slush density determined from the 4 curie unit was based on the fact that the output versus density was linear as reported by Weitzel, et al. [1968]. A significant drift occurred in the F.R. densitometer signal between the two calibration periods. This drift was later found to be due to changes in electrometer temperature and is discussed in more detail in section 3.1.4.

3.1.3 Densitometer Installation on the Upgrading Dewar

Platforms were attached to the upgrading dewar for mounting the densitometer. The source assembly was bolted to a metal platform at an angle to direct the beam from one shutter opening along a diameter through the dewar. The sensing unit was clamped to a similar platform opposite the source. The installation is shown in figure 8. Figure 9 is a schematic of the installation. The beam was at 63 cm (25 in.) from the bottom of the dewar. A box was built to cover the sensor unit to protect it from adverse weather. It was later determined that the ovens for temperature control in the sensor were not maintaining constant temperature so the box was insulated with fiberglas and a strip heater was installed with a control to maintain a more stable environment.

A new, good quality, low temperature sensitivity, 500 MΩ, resistor was installed when the densitometer was mounted on the upgrading dewar because the total attenuation for the upgrading dewar was different than for the generator. Also, it was desirable to remove the auxiliary attenuators.

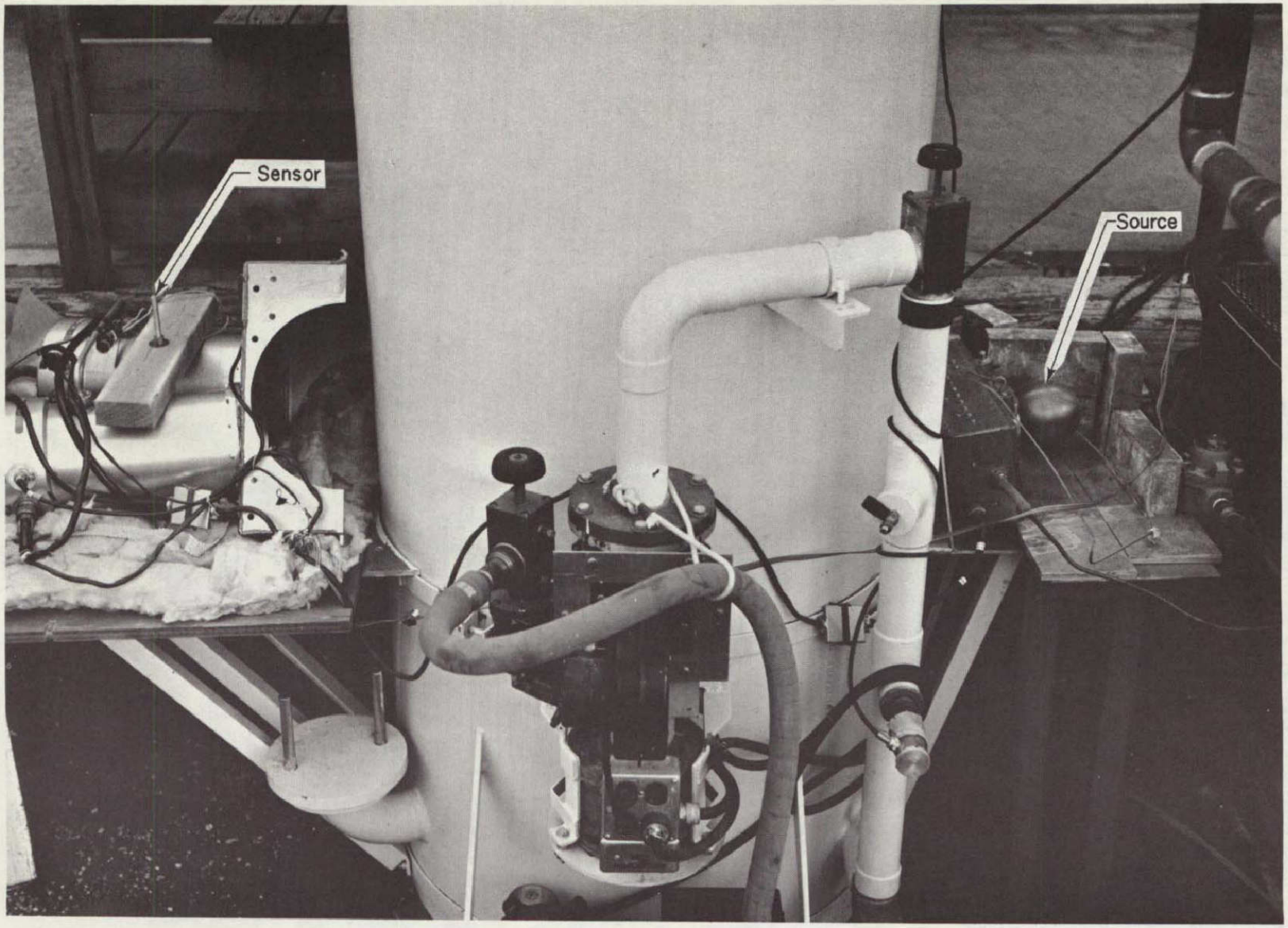

Figure 8. Densitometer on Upgrading Dewar

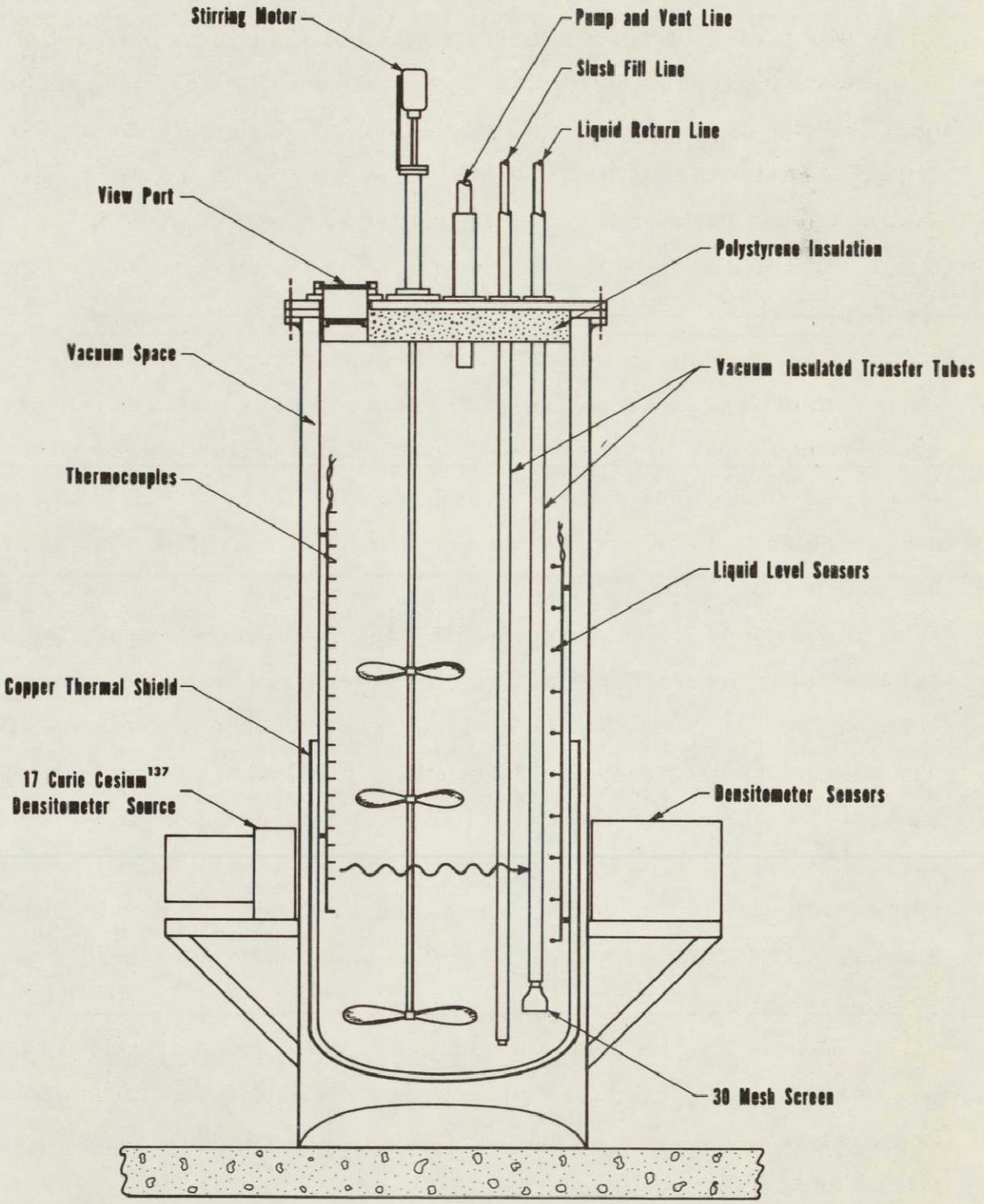

Figure 9. Densitometer and Upgrading Dewar Cross Section

3.1.4 Problems with the Densitometer

When the densitometer was first mounted on the generator a check was made to determine long-term stability using a fixed absorber to simulate hydrogen. A significant drift occurred. However, the meg-ohm resistor used for the first test was not of good quality or high stability. A better quality 513 MΩ resistor was installed and the stability improved, but some drift was still present as was mentioned in section 3.1.2. The comparison tests, shown in figure 7, were made with this configuration.

After the unit was installed on the upgrading dewar further difficulty with drift occurred. The unit had thermistors installed for monitoring temperature in the critical areas. These critical areas all have ovens with controllers. The controlled units and temperatures are the electrometer at 337 K and the ion chambers at 325 K. The electrometer has both a coarse and a fine temperature oven while the ion chambers have single ovens. Monitoring the temperature of the electrometer during a stability test revealed that the drifts were due to temperature changes and that the electrometer ovens were not functioning properly. The coarse oven heater was disconnected. Insulation was added to the cover and a tape heater and controller were placed inside the box to reduce the sensor environmental changes to a minimum. This configuration worked very well. When data were taken with the densitometer, electrometer temperature data were also taken and a correction was made if required.

Late in the test program, the oven for the number two ion chamber became erratic and this oven heater was also disconnected. The temperature in the cover remained stable enough to prevent any adverse effects and testing continued.

On several occasions during the testing program, mixing units were moved and mixer configurations were changed. This changing of

the mixing unit changed the densitometer zero point as the mixer was in the radiation beam. When the mixing shaft or propellers were near the beam center, the densitometer output became sensitive to mixer operation, which necessitated a small rotary adjustment of the source and sensor to avoid mixer interference.

3.1.5 Conclusions from the Desitometer Test

From the test using the fast response densitometer, three conclusions are made:

1. For short duration, six hours or less, the densitometer is as precise as the 4 curie source unit described by Weitzel, et al. [1968].
2. The random scatter in the F.R. densitometer data, as determined in the subcooled liquid region, is less than for the 4 curie source unit, indicating this densitometer to be potentially a more precise unit.
3. The oven systems are adequate when functioning but the oven temperature controllers have a high failure probability.

3.2 Beta Ray Densitometers

3.2.1 Description of Beta Ray Gauges

Two nuclear radiation attenuation densitometers using beta sources were tested in the upgrading dewar. These units are submerged in the fluid and are essentially point sensors. Figure 10 shows the probe assembly. The sensor is a solid state unit and is enclosed in a stainless steel housing which is connected to a 1/4 inch stainless steel tube. The signal is carried in a single wire from the sensor through the tube to a fitting at the tube end. This fitting also has the necessary valves and seals so that the tube and sensor enclosure can be evacuated and purged with helium gas. A thin stainless steel window is welded into the side of the sensor housing facing the source for minimum attenuation of the beta rays.

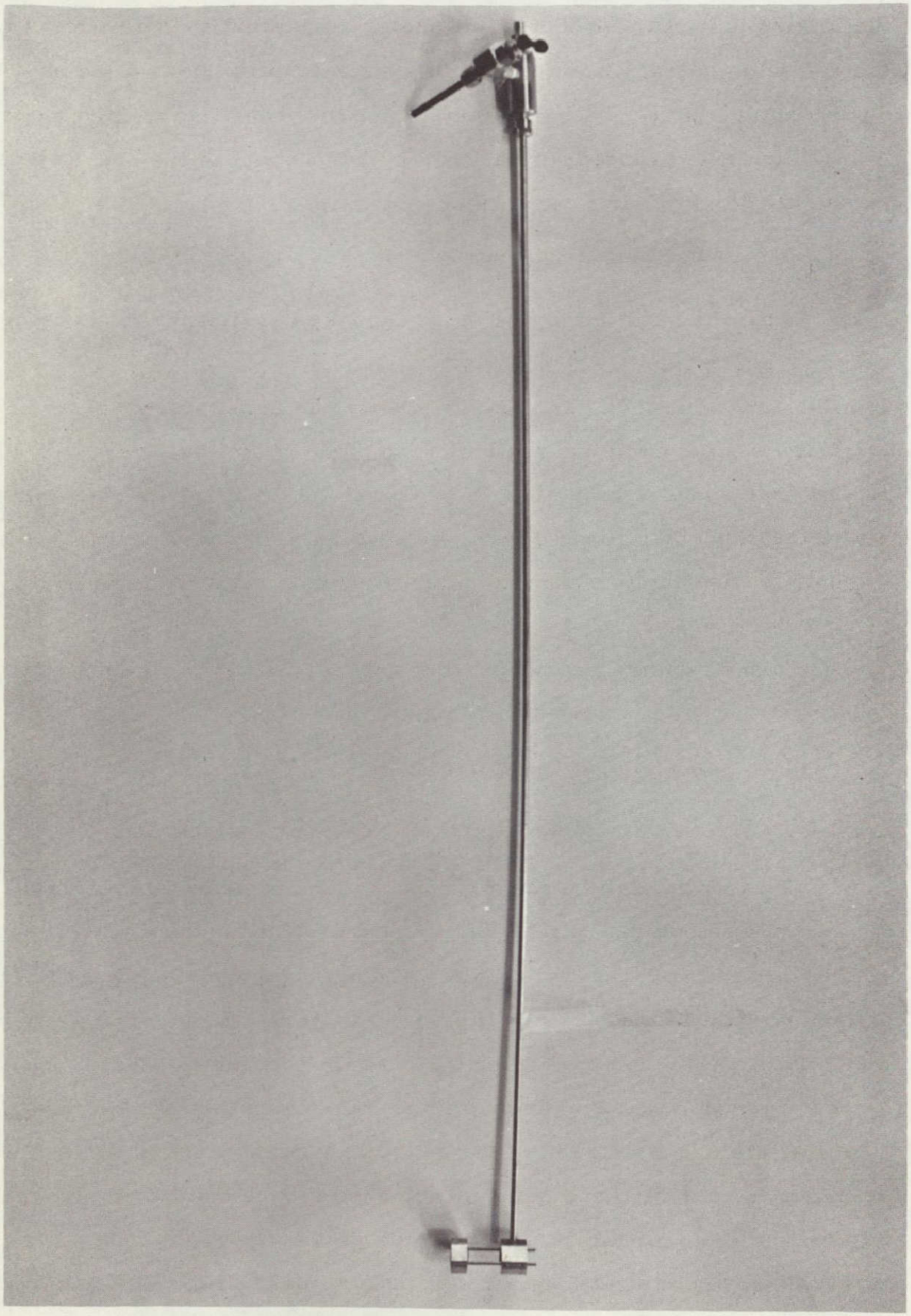

Figure 10. Beta Gauge Densitometer Probe

The source, which is strontium^{90}, is sealed with double enclosures in a stainless steel housing. These enclosures are thin windows to pass beta radiation. The housing assembly attaches to the sensor with two small rods which slide through holes in the sensor housing. The source to sensor spacing is thus variable. Spacing used for hydrogen was 4 cm (1.57 in.) which was the optimum as determined by previous calibrations performed in the Density Reference System described by Weitzel, et al. [1968]. The source and sensor are shown in figure 11.

The signal conditioning and detecting units for the beta source gauges consist of preamplifiers, amplifiers, discriminators, counters, and a voltage source for applying bias voltage. Figure 12 shows the panel mounted signal conditioning and readout units. The system has three channels and is capable of measuring three samples simultaneously.

The probes are connected to the preamplifiers with a single conductor lead 12.2 m (40 ft) long. In the tests reported here the preamplifiers were located at about 10.7 m (35 ft) from the dewar. The remainder of the electronics were approximately 22.9 m (75 ft) beyond the preamplifiers and were connected with 30.5 m (100 ft) leads. In figure 12 the preamplifiers are shown at the top of the picture. The analog ratemeters shown in the center panel were not used for numerical data but were used to drive a recorder. Numerical count-rate data were determined with an electronic counter-timer and were automatically recorded on magnetic tape.

3.2.2 Installation of Beta Ray Gauges in the Upgrading Dewar

The beta gauges were installed so that the source and sensor were submerged in the fluid to be measured and the end of the 1/4 inch tube, where electrical and helium fittings were made, extended outside the vessel. A seal around the 1/4 inch tube was required at the top plate. Installation in the upgrading dewar required extension of the

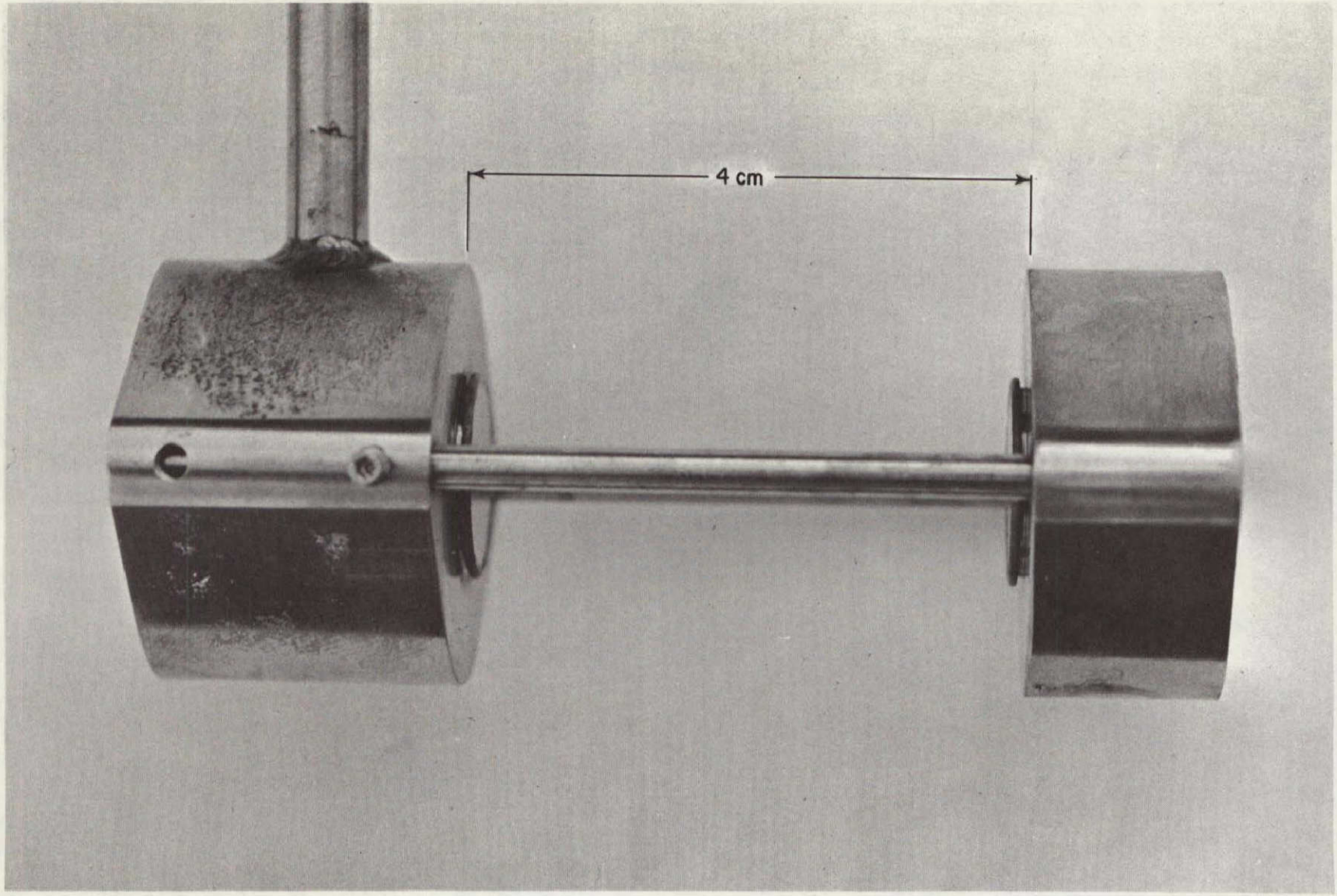

Figure 11. Beta Gauge Densitometer Source and Sensor

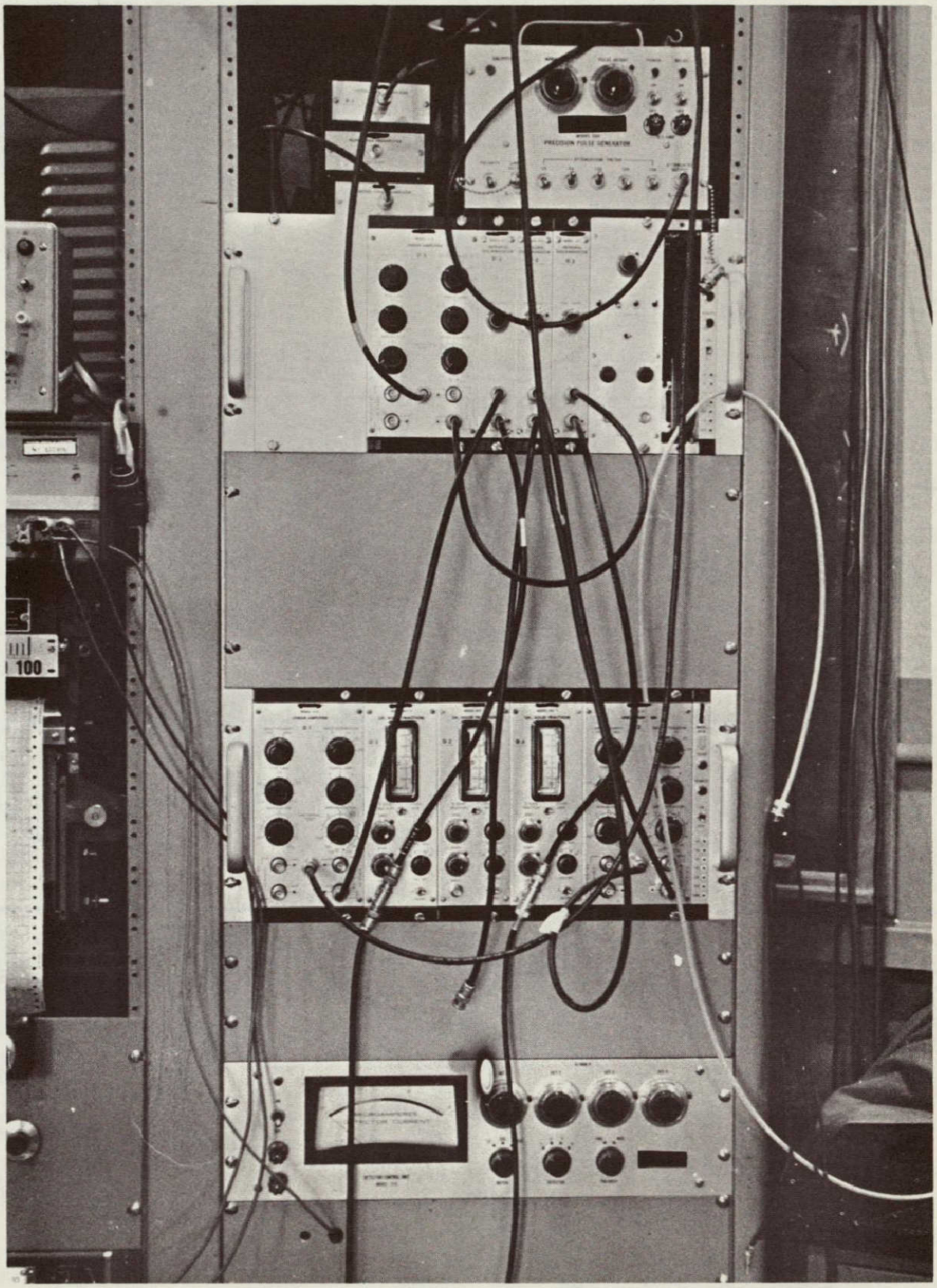

Figure 12. Beta Gauge Densitometer Signal Conditioning Unit

1/4 inch tube and the signal conducting wire to reach the desired sampling levels. The longer probe was 35 cm (13-3/4 in.) from the dewar bottom and was about 7.6 cm (3 in.) from the dewar wall. The shorter probe was mounted 84 cm (33 in.) above the dewar bottom, and the same distance from the wall.

3.2.3 Test Results from Beta Densitometers

During the test program the beta source densitometers were calibrated in the subcooled liquid region using vapor pressure as described in section 3.1.2. A composite of these calibrations for both gauges is shown in figures 13 and 14. As can be seen from figure 13 the short probe retained its zero and slope consistently throughout the testing, which covered a period of 23 days. The line drawn through the data is the best least squares mathematical straight-line fit to the data. The long probe changed zero and slope from day to day. The source for this probe experienced a window failure in test 12, at the end of the test, and the zero drift and slope change may be related to the window failure. Because of the changing zero no attempt was made to fit a single straight line to the data.

In the program, tests were made where the output counts from the beta gauge signal conditioning unit were recorded and the density of well stirred slush was determined by the F.R. densitometer described in section 3.1. The data from these tests are shown in figures 15 through 17 with the density from the F.R. densitometer plotted versus the counts from the beta source densitometers. The data taken in the subcooled liquid region are also shown in the figures to provide a reference calibration for the particular test. The curves drawn on the upper probe data are from the mathematical fit to all of the calibrations. Curves drawn on the lower probe data are the best estimates based on each day's calibration data.

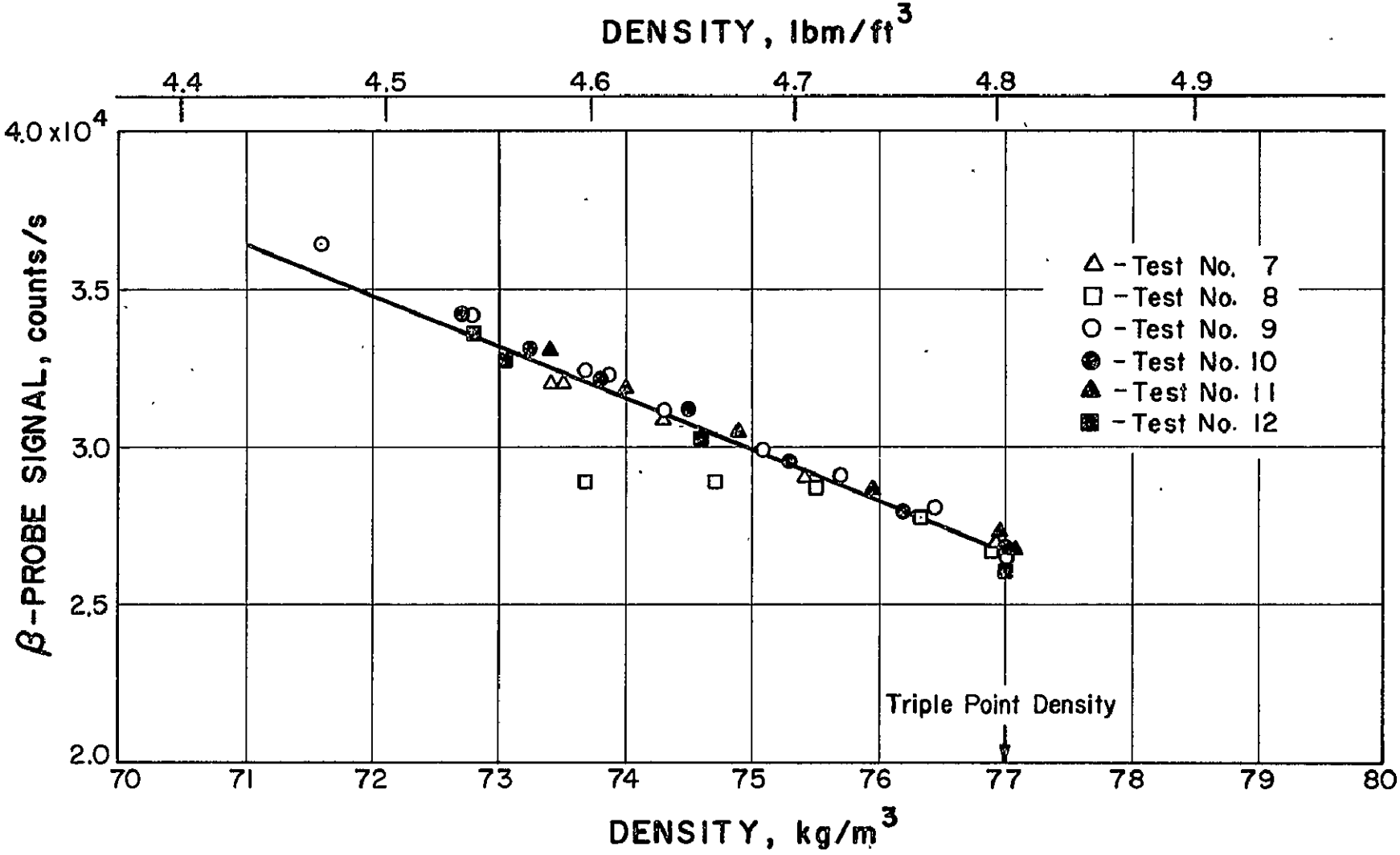

Figure 13. Upper Beta Gauge Calibration

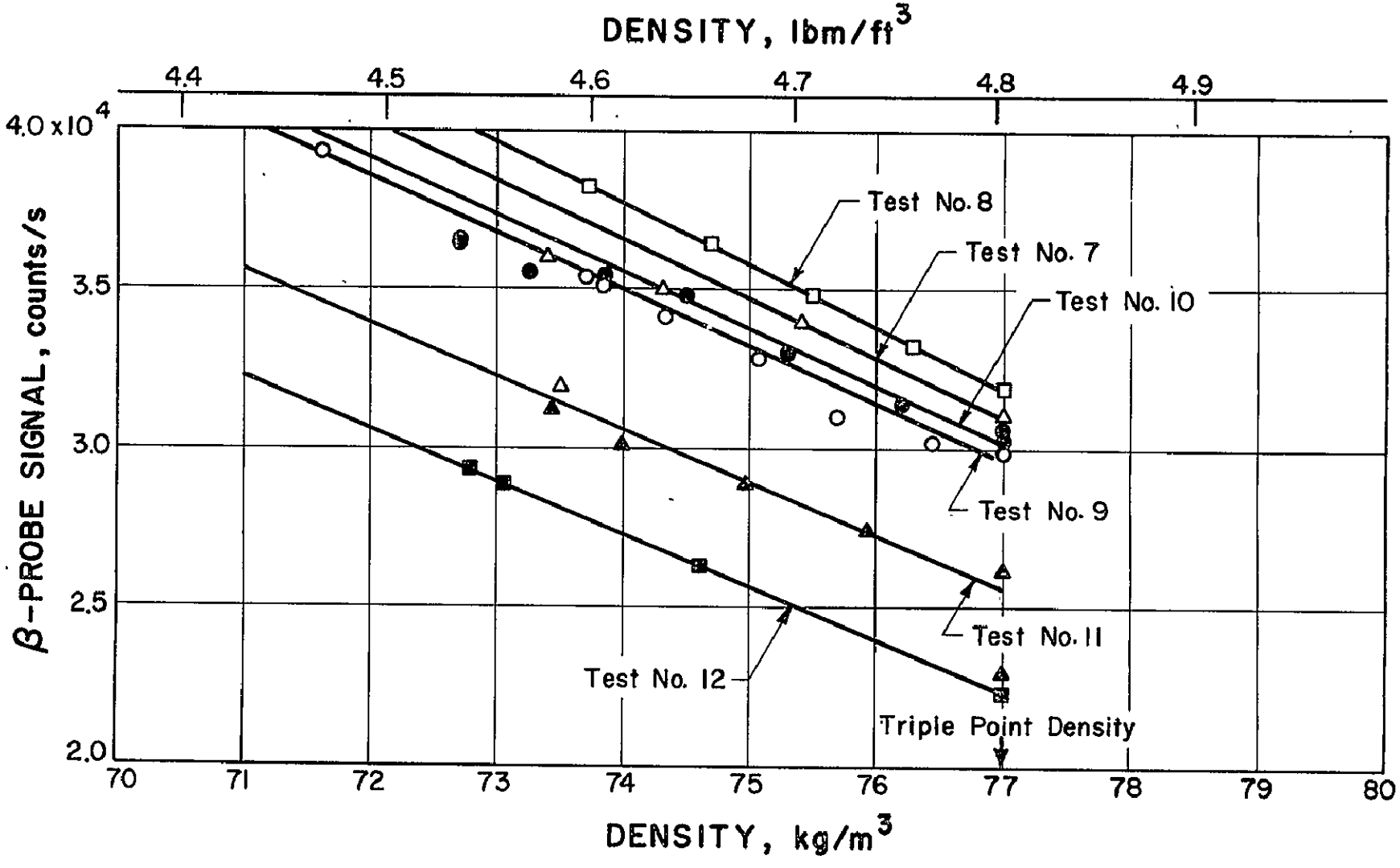

Figure 14. Lower Beta Gauge Calibration

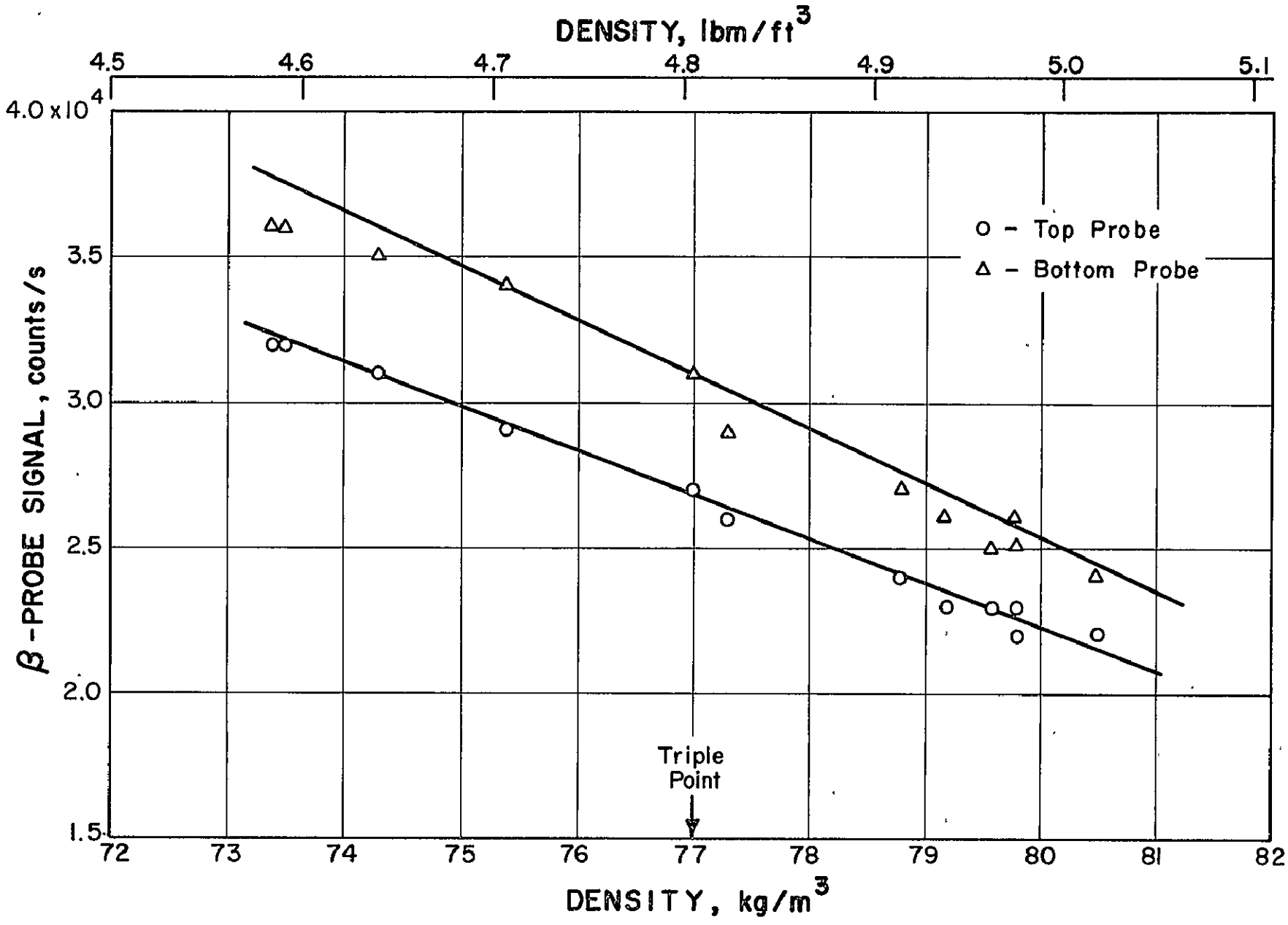

Figure 15. Beta Gauge Data in Mixed Slush--Test 7

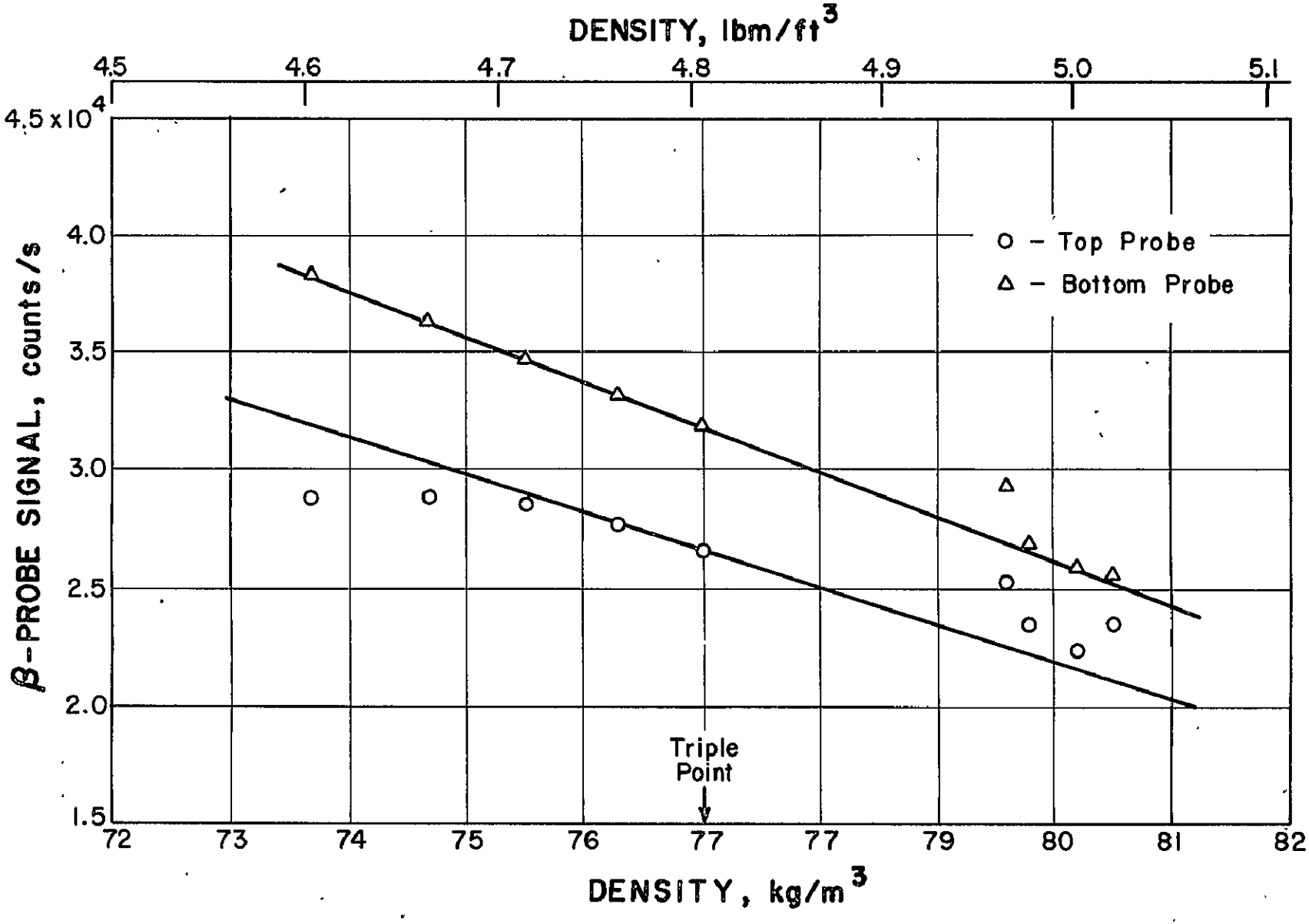

Figure 16. Beta Gauge Data in Mixed Slush--Test 8

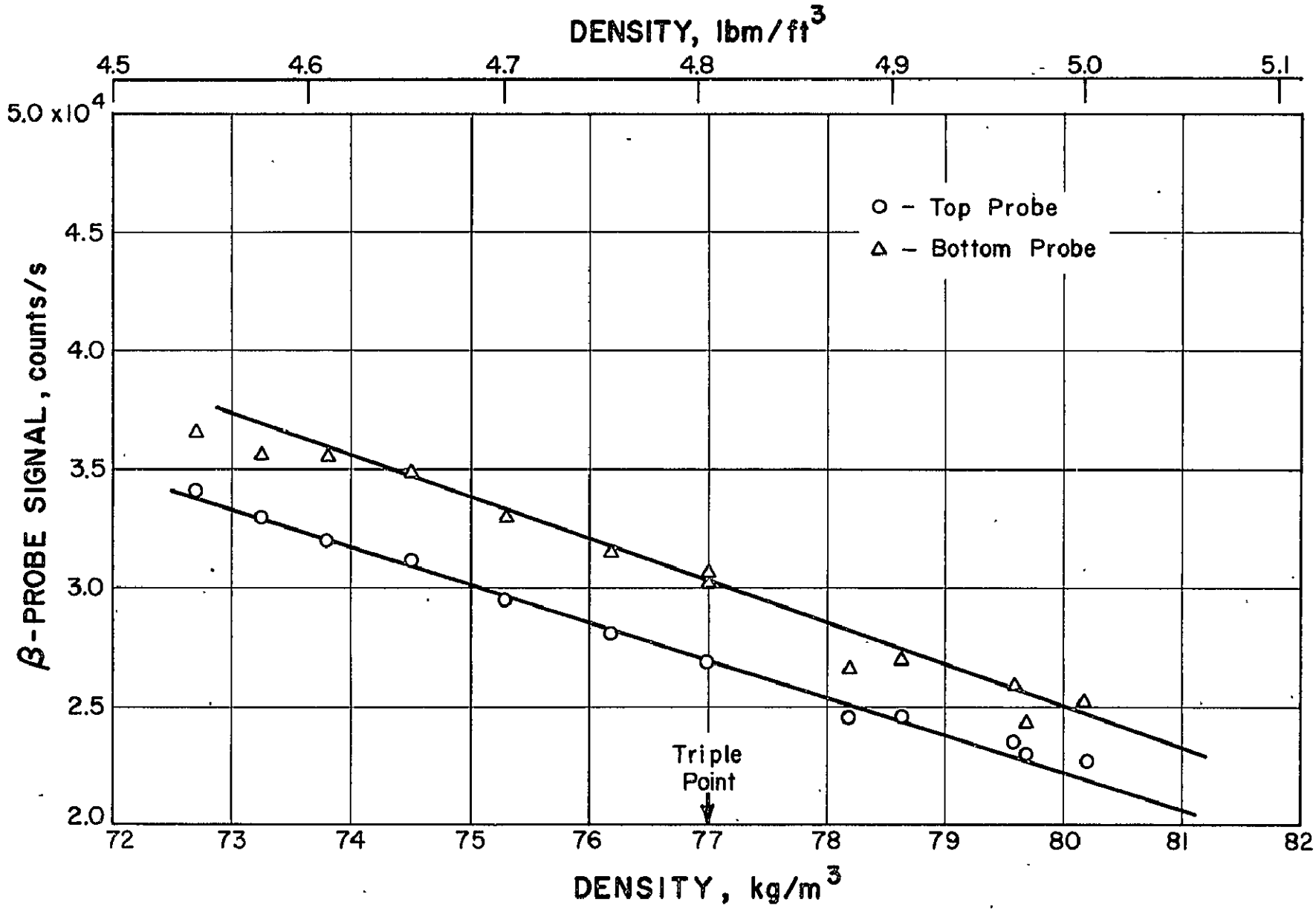

Figure 17. Beta Gauge Data in Mixed Slush--Test 10

Difficulty in mixing was encountered with the mixer used in test 7 and the apparent higher density indicated by the lower beta gauge may be a result of mixing problems. The mixer was changed for the remainder of the test so no mixing variables should be present in those tests. The data scatter is believed to be primarily attributable to the beta gauge units.

A number of tests were made where the solids were allowed to settle and the density in the settled slush was measured using the F. R. densitometer and the beta gauges. Figures 18 through 22 show these data. The calibrations are shown for comparison. From these data it is apparent that the beta gauges tend to indicate a low density reading above 81 kg/m^3 (5.06 lb_m/ft^3) density. The data trend for the upper probe in test 11 is explained by the fact that the settled slush level dropped below the probe level. The lower probe data in test 11 and 12 are obviously inconsistent. This is very likely related to the source window failure which occurred during test 12.

3.2.4 Discussion and Conclusions

Several beta gauge failures occurred during the test program. The first short probe which was installed failed during the first test. This gauge was checked in liquid nitrogen several days prior to the hydrogen test and was found to be stable. After the first cooling to triple-point hydrogen temperature the signal became very erratic and the gauge was assumed inoperative. Later checking in liquid nitrogen with different sources proved the gauge to be erratic.

The second failure, which occured in test 12, was previously mentioned. The outer window on the source ruptured and blew outward, remaining attached at one location so that the window material protruded out perpendicular to the window opening. No source material escaped as the inner window remained sealed.

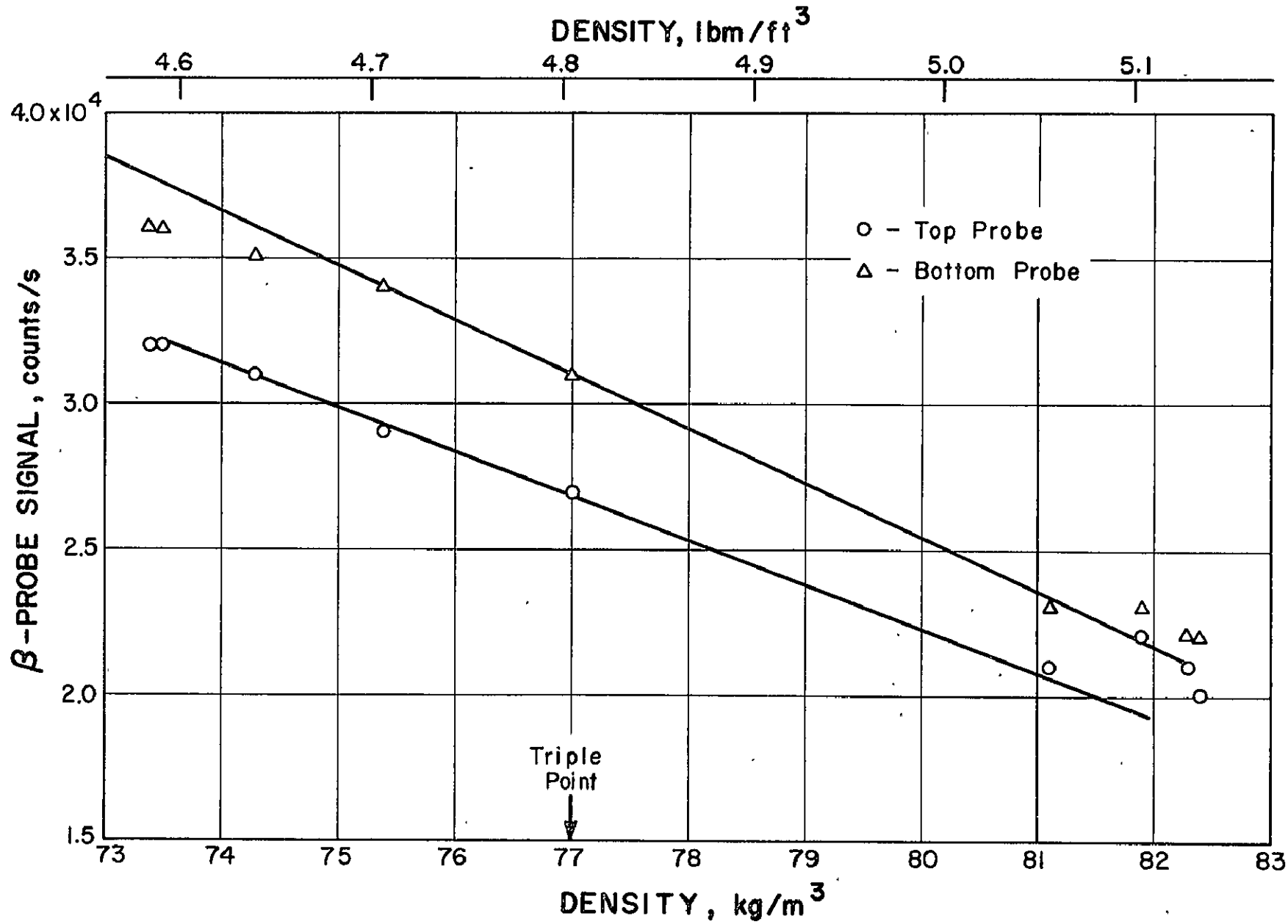

Figure 18. Beta Gauge Data in Settled Slush--Test 7

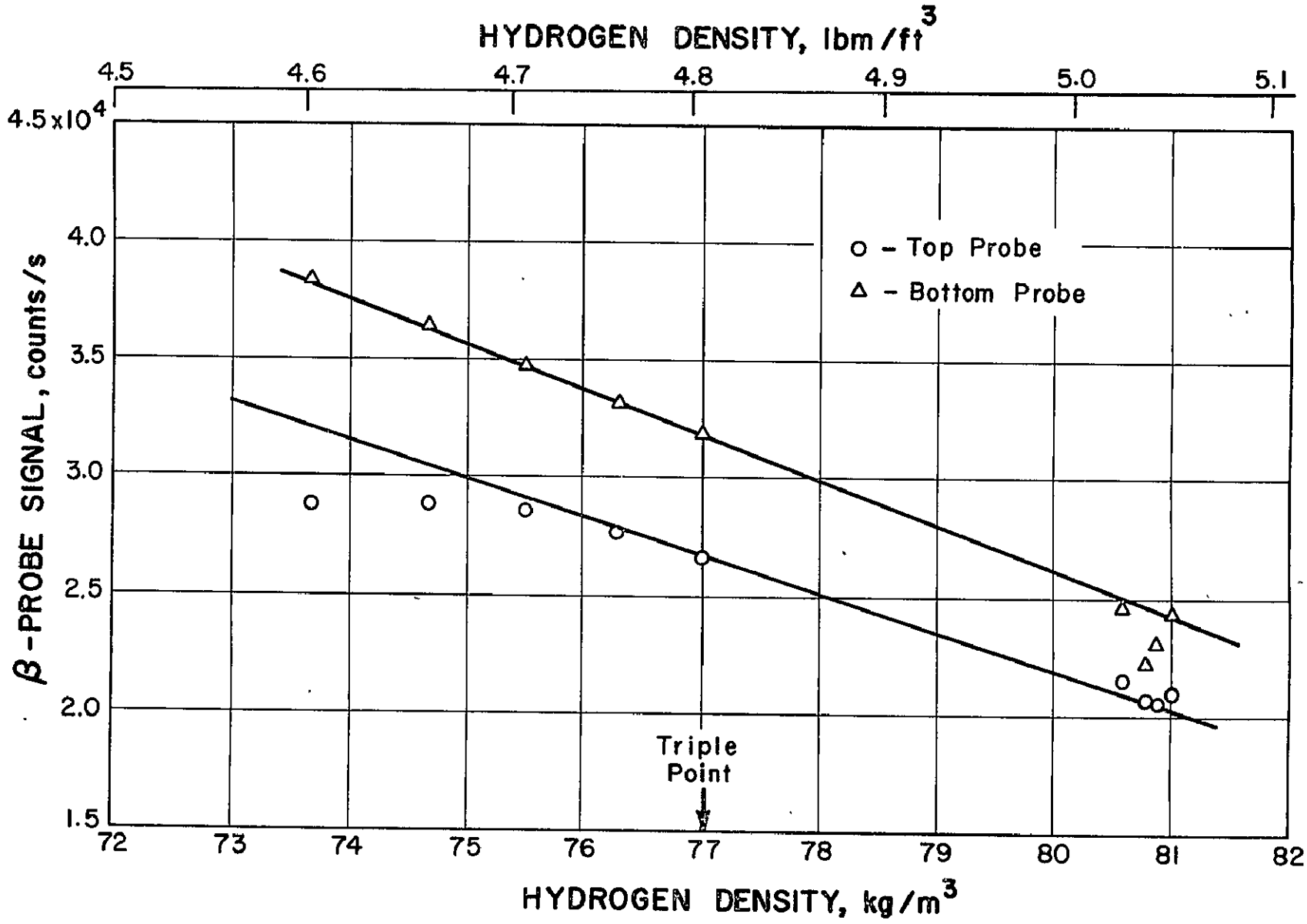

Figure 19. Beta Gauge Data in Settled Slush--Test 8

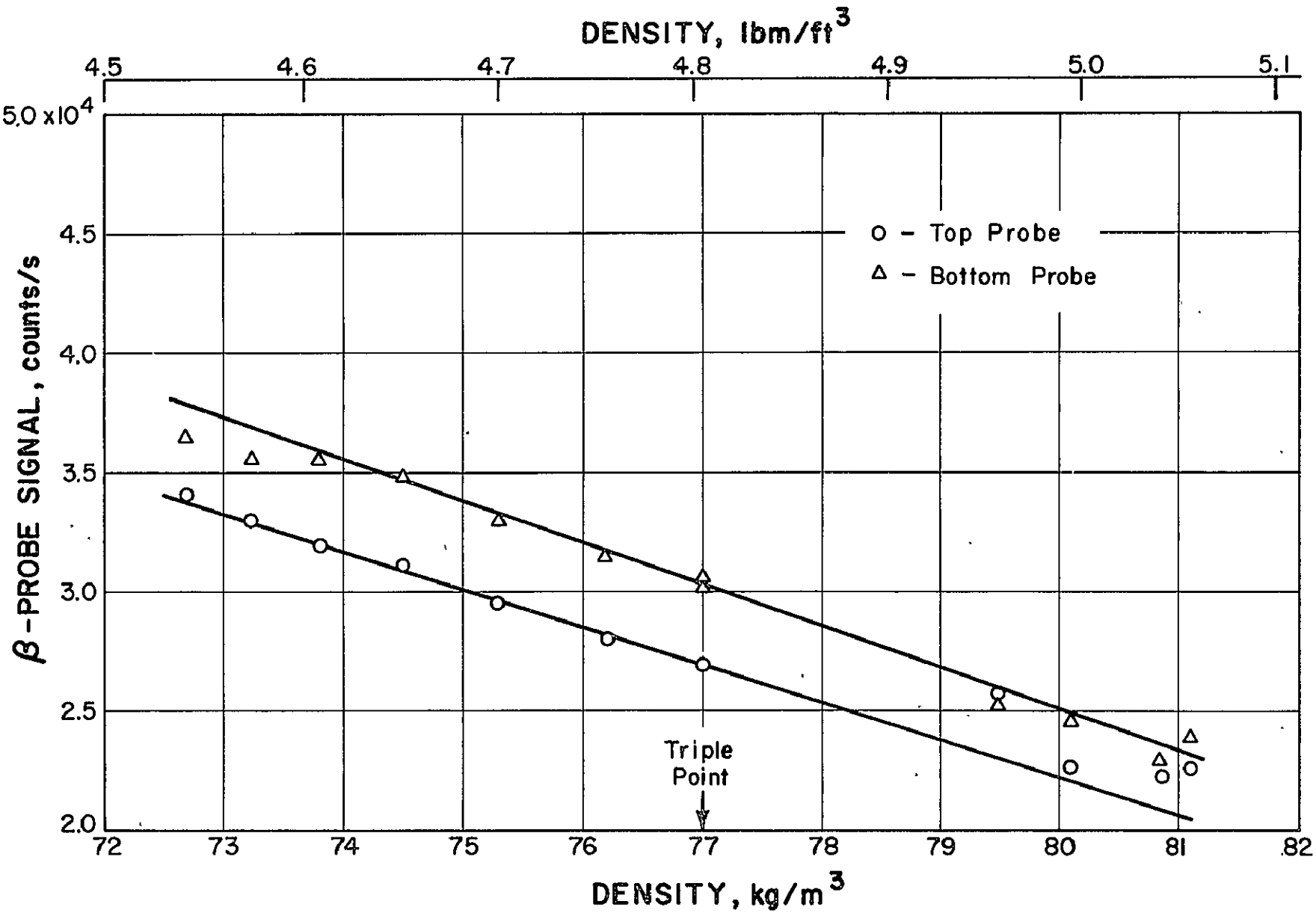

Figure 20. Beta Gauge Data in Settled Slush--Test 10

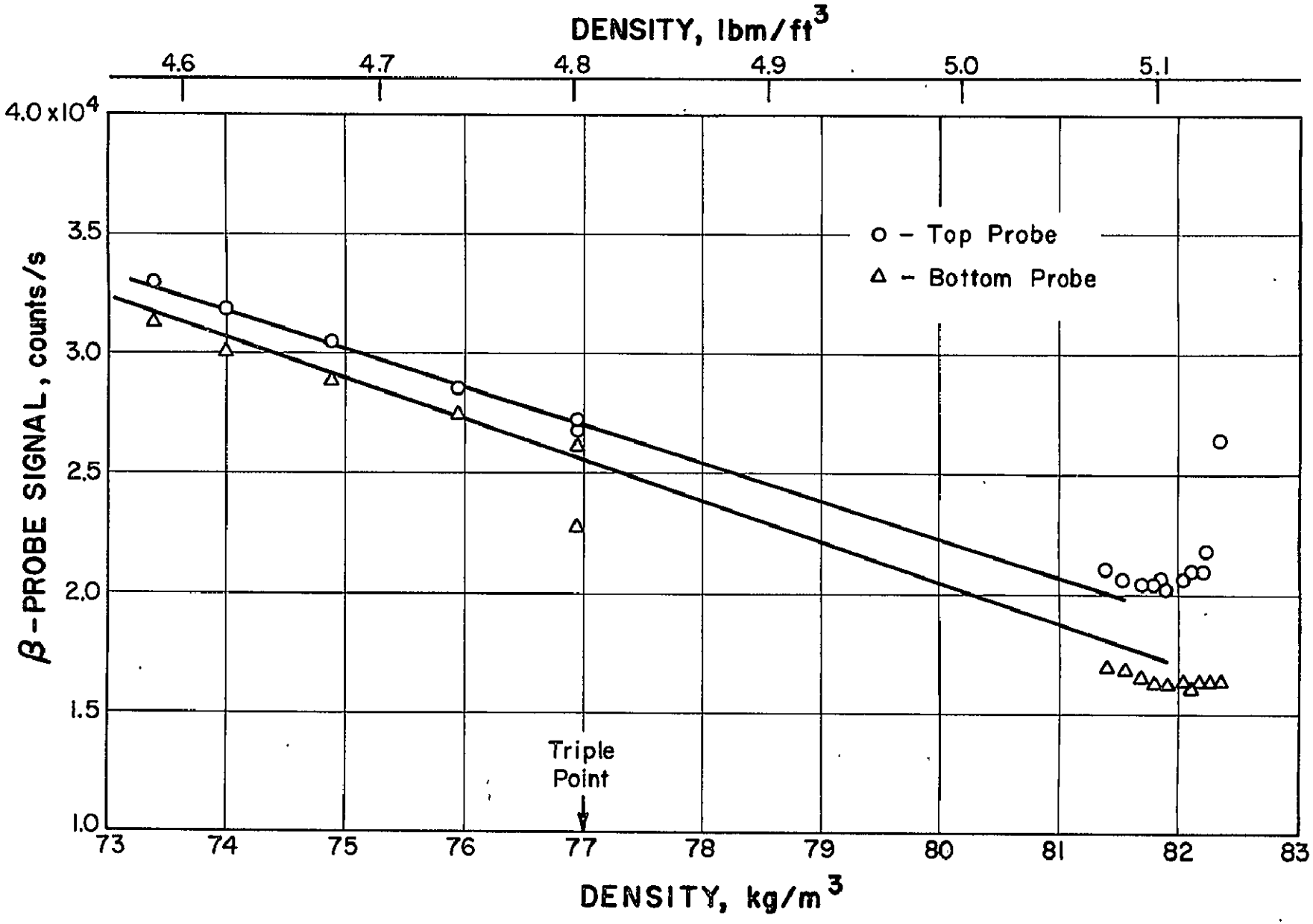

Figure 21. Beta Gauge Data in Settled Slush--Test 11

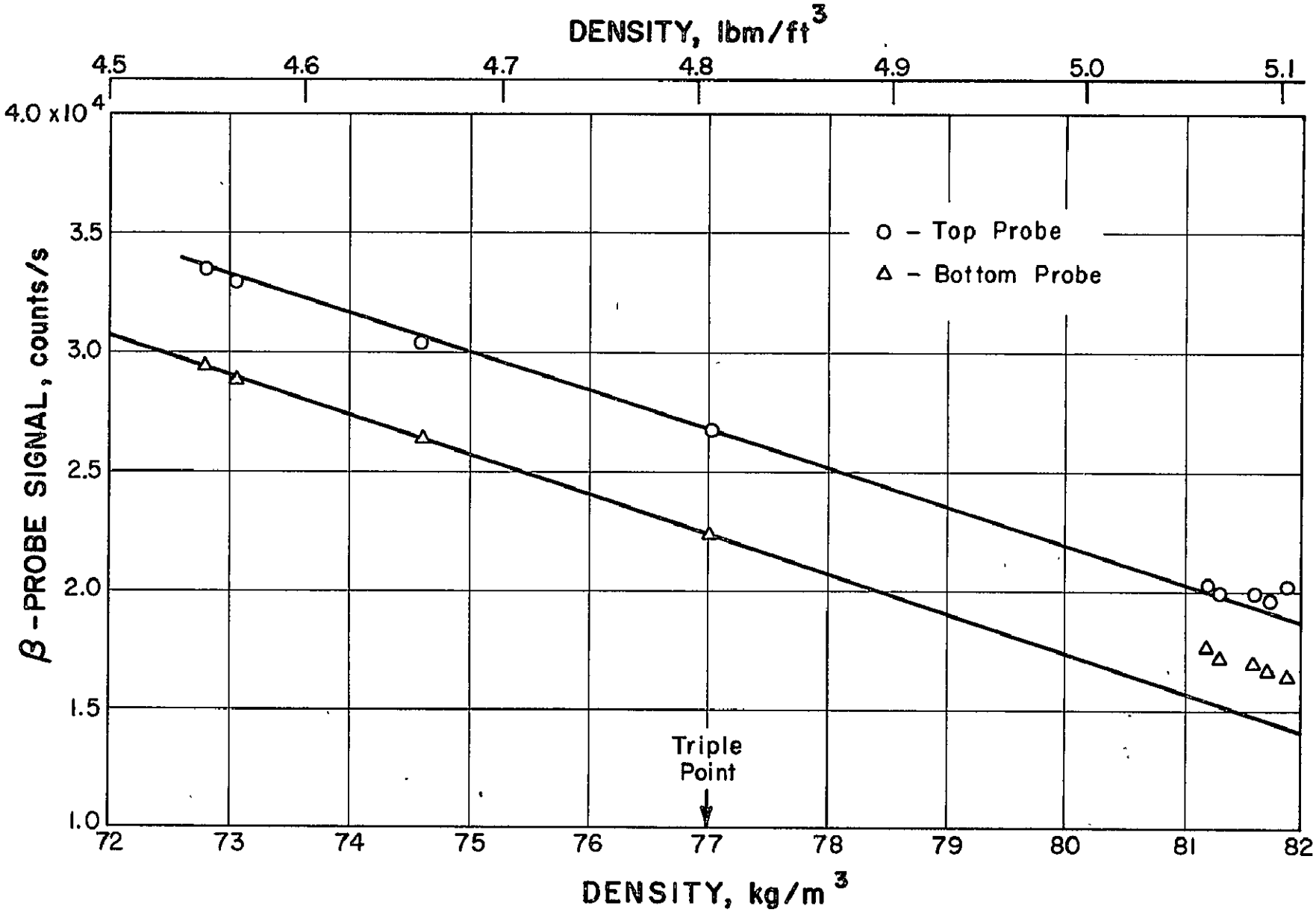

Figure 22. Beta Gauge Data in Settled Slush--Test 12

From the tests performed two conclusions can be made:

1. The beta source densitometer can be used as a density measuring device in homogeneous mixtures of slush.
2. At densities greater than 81 kg/m^3 (5.06 lb m/ft^3), in settled slush, the beta gauges apparently have a sampling problem.

Low density indication above 81 kg/m^3 (5.06 lb_m/ft^3) may be caused by some bridging of solids across the sensor to source spacing which prevents complete settling of solid from occurring, especially at the source and sensor windows, or it may be caused by the heat leak down the probe to the sensor window melting solids.

3.3 Microwave Densitometer

The technique of density measurement by microwaves uses the principle of interferometry. Microwaves from a source are divided into two branches. One branch is used for reference and the other branch passes through the fluid to be sampled. The phase shift between branches is then determined and this phase shift is used as the measurement signal. In the system used, the microwave frequency was held constant at 9.81 gHz, giving a signal wavelength of about 3 cm. A sending and a receiving horn were submersed in the fluid and their positions were fixed. The fluid sample path length was, therefore, maintained constant. If the path length is L, the relation between path length and phase angle is

$$L = \frac{\varphi\lambda_o}{n} = \frac{\varphi\lambda_o}{\sqrt{K}} \qquad (1)$$

where φ = phase angle in cyles

λ_o = free space wavelength

n = index of refraction

K = relative dielectric constant.

From (1) the expression for phase angle is

$$\varphi = \frac{360\ L\sqrt{K}}{\lambda_o} \text{ degrees.} \qquad (2)$$

If K_o is the relative dielectric constant for triple-point liquid, then the observed phase shift for subcooled liquid and slush will be

$$\Delta\varphi = \frac{360\ L}{\lambda_o}\left(\sqrt{K} - \sqrt{K_o}\right) \qquad (3)$$

where K is the dielectric constant for the unknown fluid density. From the Clausius-Mossotti equation the relationship between dielectric constant and density is

$$K = \frac{1 + 2\ p\rho}{1 - p\rho} \qquad (4)$$

where ρ is the density and p is a constant for a given substance [Kittel, 1961]. Equations (3) and (4) can be combined to form an expression for the change in phase angle as a function of wavelength, path length, and the change between triple-point liquid density and the unknown fluid density.

3.3.1 Microwave Installation

The technique for measuring density using microwaves was tried in the Density Reference System in liquid and slush nitrogen. The results were promising, indicating that the technique would work on hydrogen. The microwave system was then transferred to the upgrading dewar for hydrogen slush experiments. The general area was better suited to separate the electronics from the experimental area, thus reducing the fire hazard in case of a hydrogen leak. Also, the upgrading dewar provided more room for the installation of the horn assemblies.

The system used for the experiments was assembled from existing microwave equipment which was not designed to operate in a potential explosive environment or in a cryogenic environment. The sending and receiving waveguide entered the dewar through the access port and curved to the dewar wall to miss the mixing blades. The centers of the horns were 10 cm (4 in.) from the dewar wall. The sending horn was approximately 135 cm (53 in.) from the dewar bottom and sending and receiving horns were 76 cm (30 in.) apart. The horn and waveguide assembly is shown in figure 23. A thin plastic window was installed in both waveguides. The waveguides were vented on the dewar side of the plastic windows to assure that they would fill with liquid. The waveguides were then pressurized with helium gas above the plastic windows. The lower horn had a plastic screen cover to prevent solid from entering the horn and being trapped there.

3.3.2 Results and Discussion

The microwave phase change was measured in the subcooled liquid and the slush region. In the subcooled liquid region the density was determined from vapor pressure as explained in section 3.1.2. In the slush region the density was taken from the measurements of the F.R. densitometer. Figure 24 shows the data for the microwave unit. The slope change at the triple point is due to the path length in slush being less than in the liquid. The path length in slush is shorter because the screen over the receiving horn excludes slush from the horn and waveguide but does not exclude liquid, and liquid fills the waveguides to the plastic windows. At this time it is not known why the data deviates from the theoretical curves.

Although the data are inconclusive with respect to precision and accuracy they do prove the concept of slush hydrogen density measurement with microwaves. A smaller pair of horns are proposed for further

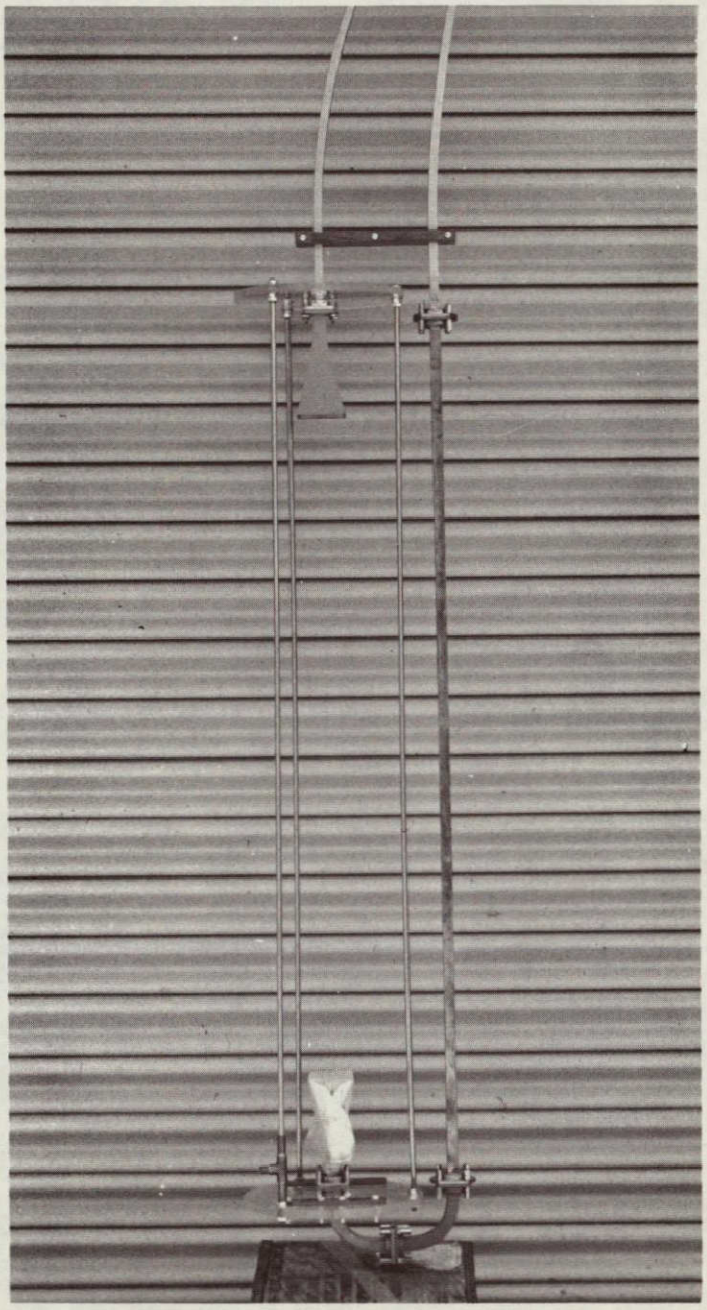

Figure 23. Microwave Horn Assembly

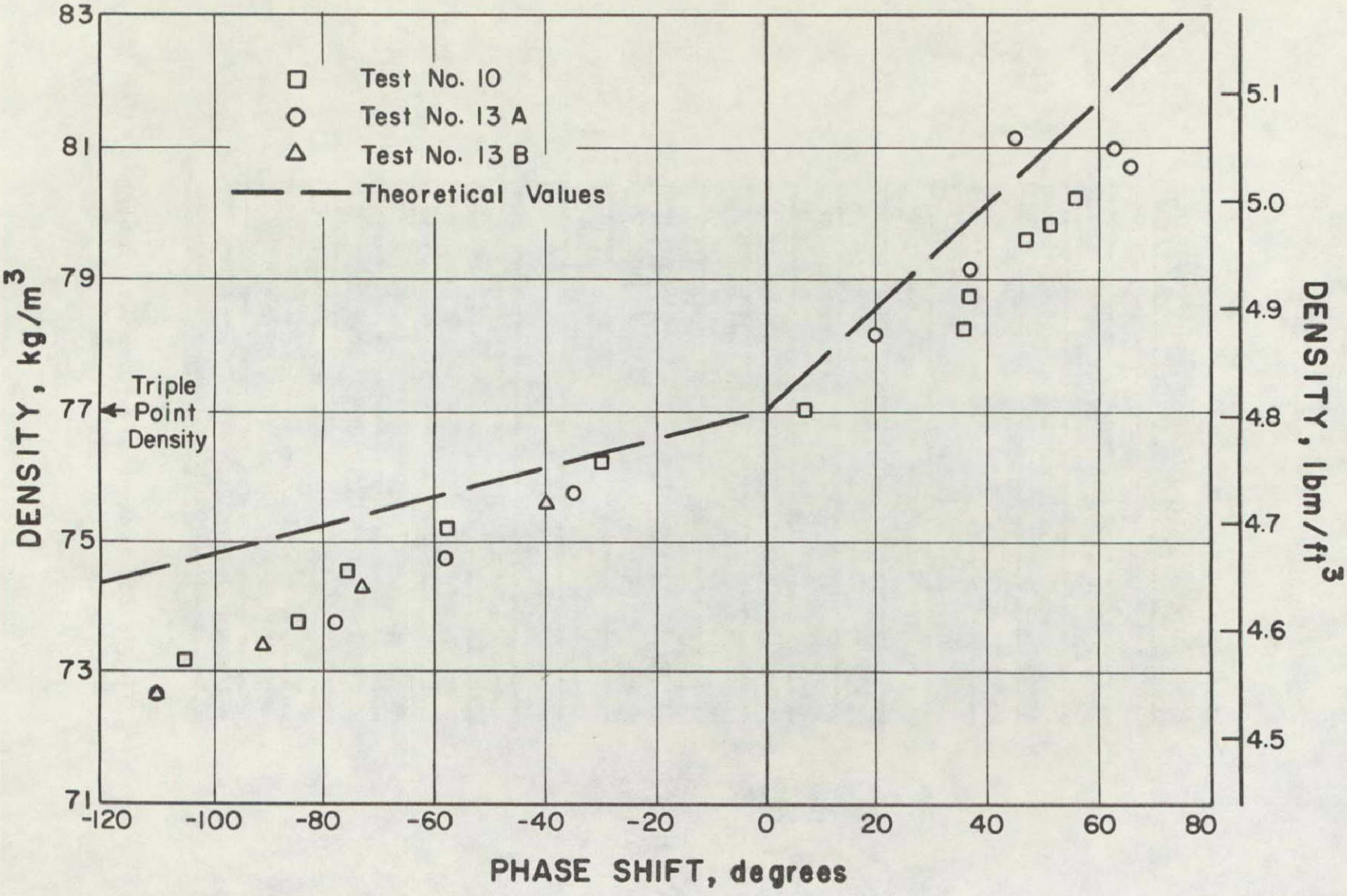

Figure 24. Microwave Densitometer Calibration

testing with other refinements. This new system will be installed in the Density Reference System for future experimental evaluation.

3.4 Temperature Stratification Instrumentation

In many applications the temperature stratification profile over the slush is important. The surface temperature becomes important to determine pressurization requirements in undisturbed slush and the profile development is important to estimate the rate at which solids may be melted.

A thermocouple probe was installed in the upgrading dewar to measure the temperature profile while aging slush hydrogen. There were 23 copper vs. gold-cobalt thermocouples equally spaced 5.1 cm (2 in.) apart and one reference couple. The reference couple which was always submerged in slush when data were taken, was 38 cm (15 in.) above the bottom of the dewar. The lowest of the 23 couples was 48 cm (19 in.) above the dewar bottom. The readout for the thermocouples was a 5-digit digital voltmeter capable of a precision of $\pm$ 1 μV. Data taken with this unit are presented in section 4, with the associated upgrading tests.

4. Upgrading Test

The primary purpose of the upgrading tests was to prove the feasibility of increasing solid fraction in a vessel by transferring in slush and removing liquid. The upgrading facility described in section 2 makes provision for the manufacture of slush, the transfer of the mixture to the upgrading dewar, and for the removal of liquid while retaining the solid with a 30-mesh screen.

The maximum solid fraction of liquid-solid mixtures attainable by upgrading is of great importance to the potential user as the heat sink available is then known. Since a significant difference in solid particle

character is evident between fresh slush (1 or 2 hours old) and aged slush (6 hours and older) [Mann, et al., 1966], the maximum solid fraction attainable in both fresh and aged slush is of importance. Besides proving the feasibility of upgrading, the resulting solid fraction of the mixture was determined using the F.R. densitometer and the beta gauges described in section 3.

The heat leak plays an important role in most experiments conducted in a dewar, therefore a measurement was made on the upgrading dewar using boiling liquid nitrogen. The boil-off gas was measured with gas meters and the heat leak was calculated from the mass of nitrogen boiled away. The heat leak under these conditions was found to be about 100 watts (341 BTU/hr) which should be very nearly the same with triple-point hydrogen.

4.1 Upgrading to Maximum Solid Fractions

During the first tests, the upgrading dewar was partially filled with triple-point liquid prior to transferring in slush. Three to four batches of slush were required to fill the upgrading dewar to the desired level under these conditions. Four batches of slush required about 2 hours to produce, so the fresh slush in the upgrading dewar was a mixture of fresh to two-hour-old solids. Starting with triple-point liquid in the upgrading dewar, the technique of flowing slush, then drawing off liquid while retaining solid, was found to be an effective way to upgrade solid fraction. With this technique it is possible to remove all of the liquid, leaving the solids essentially dry. However, dry solids are not transferable. The maximum solid fraction of slush made in 2 hours that remained fluid enough to mix and transfer was 0.54. In a slush mixture that was fresh to 2 hours old, the 0.54 solid fraction was the maximum that could be achieved without leaving solids exposed at the surface.

Five tests were made where the upgraded slush was aged for at least 4-1/2 hours. In three of the tests there was no stirring during any part of the tests. The maximum solid fractions attained in these tests, in the settled slush as determined by the F. R. densitometer, was 0.62, 0.64, and 0.59. Fresh slush was upgraded to the maximum solid fraction obtainable without draining liquid below the solid level. The dewar was then closed until the ullage pressure reached 14 kN/m^2 gauge (2 psig), the vent check valve operating pressure. The dewar was allowed to vent for the remainder of the test. A significant amount of helium gas was present in the ullage at the time the dewar was closed as helium was used for transfer during upgrading and some gas always flowed into the dewar at the end of the transfer. During two of these tests, temperature stratification data were taken after the upgrading was completed. These data are shown in figures 25 and 26.

One result that is immediately apparent from the 13.8 K isotherm is the slope change that occurs at the point where the shield attaches. This is explained by the fact that the heat transfer rate above the shield is much greater than below the shield. A significant portion of the radiation and conduction energy that is transferred from the outer wall in the area of the shield is absorbed by the shield and conducted to the point of contact. The fluid at the contact point thus absorbs most of the heat flux for the entire area below the attaching point. In all of the aging tests where temperature stratification was measured, visual observation of the solid level revealed that it was the same as the 13.8 K isotherm as near as could be observed.

Data were taken on the beta gauge densitometer probes during one of these tests. This is the data shown on figure 21. As was noted in section 3.2.3 the upper probe showed a decrease in counts, that is an increase in density, to a density of 81.9 kg/m^3 (5.11 lb_m/ft^3), then

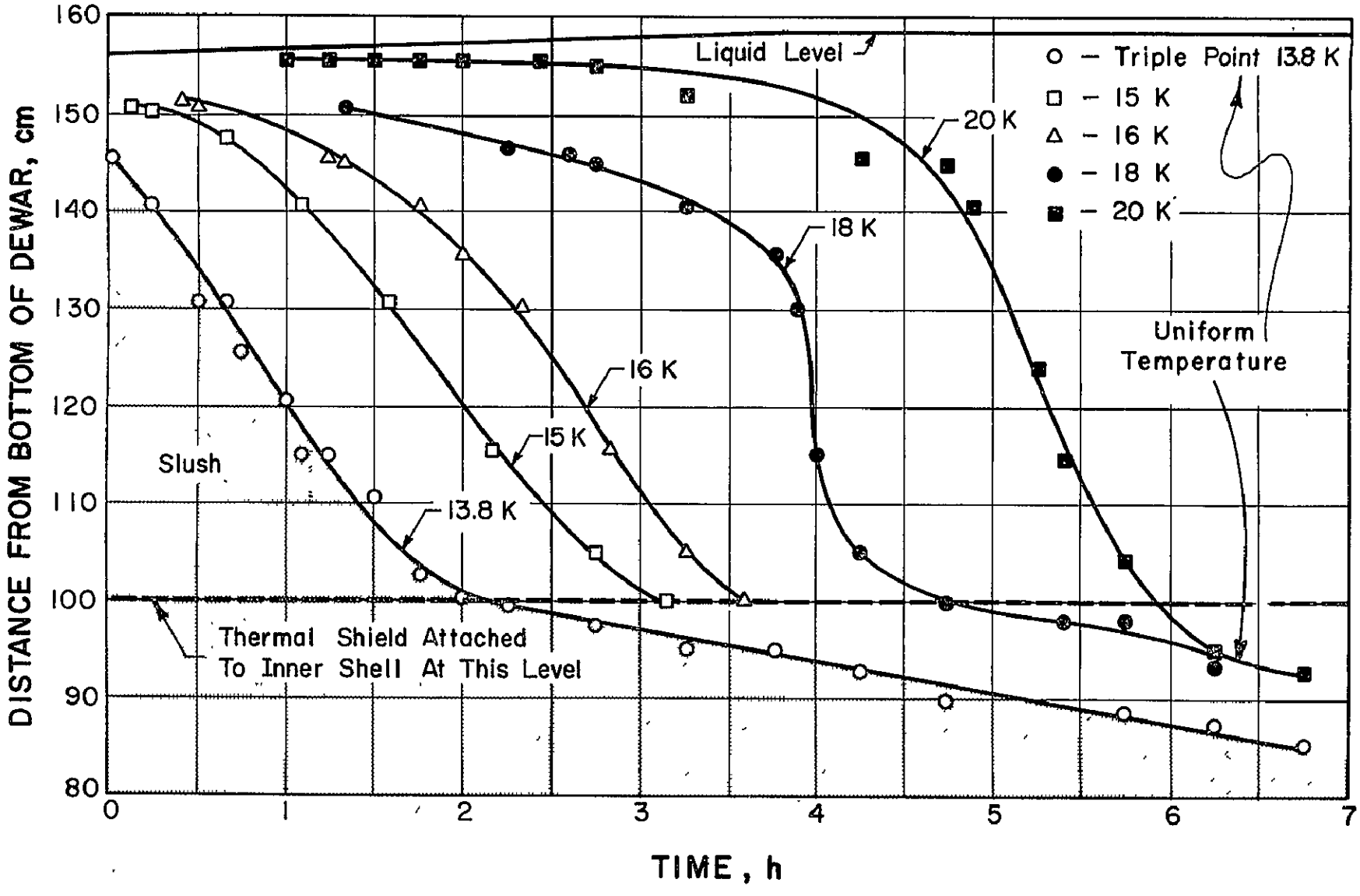

Figure 25. Temperature Stratification--Test 6

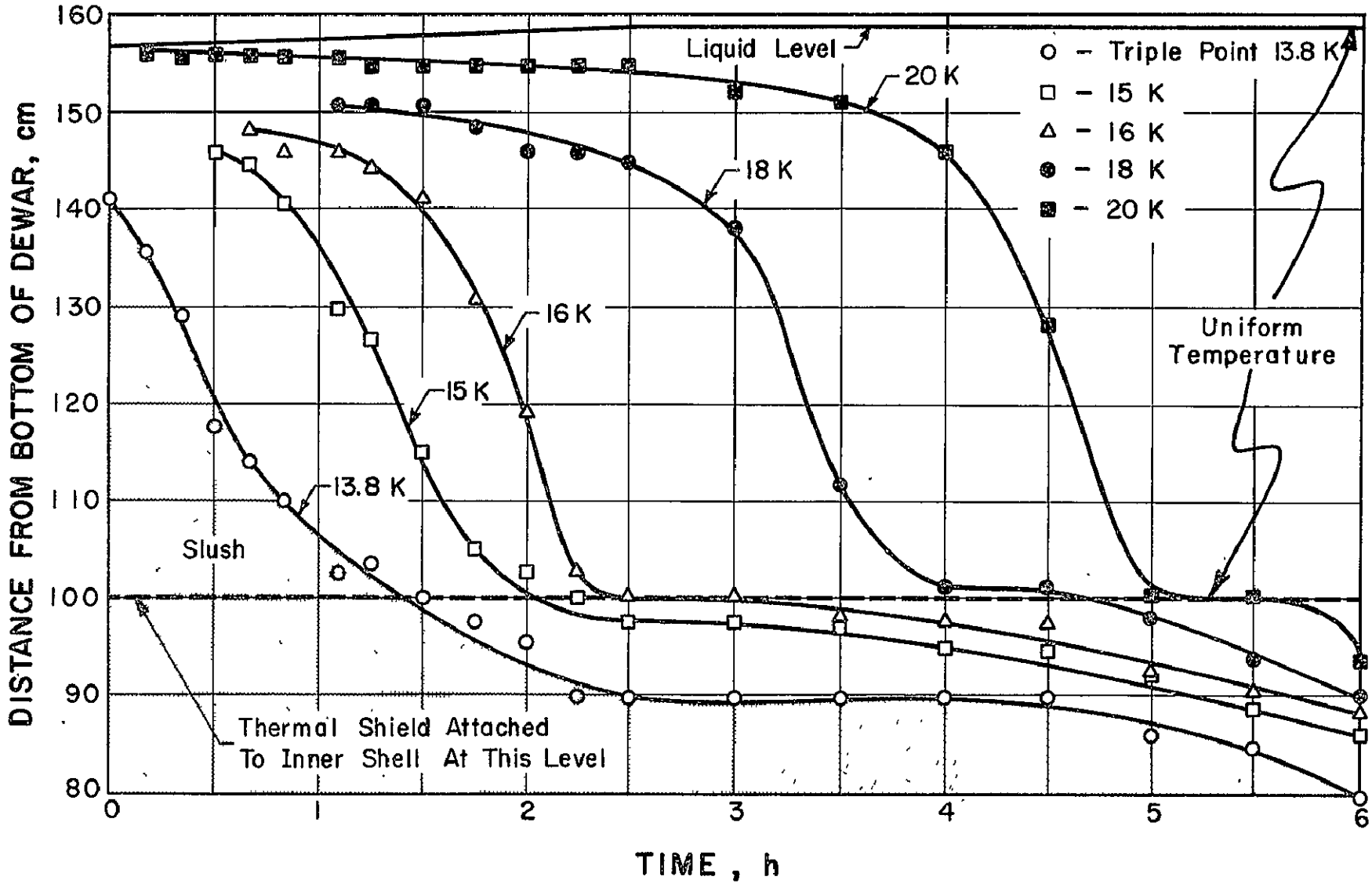

Figure 26. Temperature Stratification--Test 11

the count rate increased indicating a density decrease. This occurred because the top surface of the settled solid reached the upper probe and finally dropped below the probe level of 84 cm (33 in.).

The data for the lower probe, which was always at least 46 cm (18 in.) below the solid surface, indicates a leveling off of density. However, the F.R. densitometer, which samples at a level above the lower probe, indicates a continuing increase of density to the end of the test. As is explained in section 3.2, the low density indication of the beta probe is very likely due to bridging in the sample area at the high settled density.

The hope was that the beta probe could be used to determine if a density stratification occurred in the settled slush. The density measured by the upper probe before the slush level settled across it does appear to be slightly less than that measured by the F.R. densitometer, however, this density is very near the density at which the probes appear to have a sampling problem. The lower probe data are so questionable because of the shift in triple-point count rate that no conclusion can be drawn from them.

Two tests were conducted where slush was stirred while being upgraded. One objective of these tests was to determine the effect of stirring on aging characteristics. In these tests the maximum solid fraction attained was 0.59 and 0.54. The aging times were 6 hours and 4-1/2 hours. The undisturbed aging time in these tests was about 2 hours so the comparison of maximum solid fraction attained while aging must be made during the 2-hour period. The solid fraction in settled slush that has been stirred during upgrading does appear to be less than in slush that has not been stirred, if the comparison is made immediately after upgrading. A typical example is shown in figure 27 where the unstirred slush starts at a density of 81.4 kg/m^3 (5.08 lb_m/ft^3) while the settled density of slush that has been stirred is

81.2 kg/m^3 (5.07 lb_m/ft^3). As the slush ages the densities in the two tests appear to converge but are still slightly different at 2 hours.

Temperature stratification data were taken during one of the tests in which slush was stirred during upgrading. These data are shown in figure 28. There is no significant difference in the shape of the isotherms for the slush that has been stirred when compared to unstirred slush except during the first 45 minutes when an inadvertent drop of about 5.1 cm (2 in.) in liquid level occurred due to malfunction of a valve.

Data were taken with the beta gauge densitometers during one of these tests. These are the data shown on figure 22. The data for the upper probe are consistent, with a slight tendency to measure low density above 81 kg/m^3 (5.06 lb_m/ft^3) as previously mentioned. The lower probe data are consistent in the slush region but indicate a very low density based on the liquid region calibration; however, this probe failed on the day these data were taken. The data discrepancy between liquid and slush is very likely related to the probe failure.

4.2 Miscellaneous Testing in the Upgrading Dewar

Several auxiliary tests were made during the upgrading and aging studies. One such test consisted of adding slush during the aging to maintain a fixed solid fraction in the upgrading dewar. In this test batches of fresh slush were added as required at about 1-1/2 hour intervals. During the 1-1/2 hour intervals the dewar was closed until the ullage pressure built to the vent pressure of 14 kN/m^2 gauge (2 psig), then the vent was opened. Prior to making a new batch of slush, liquid was drawn from the upgrading dewar through the return line. This resulted in the warm liquid being drawn through slush and melting some solids. The test was conducted in this way to simulate a continuous upgrading system with mixing in which the slush would be absorbing all of

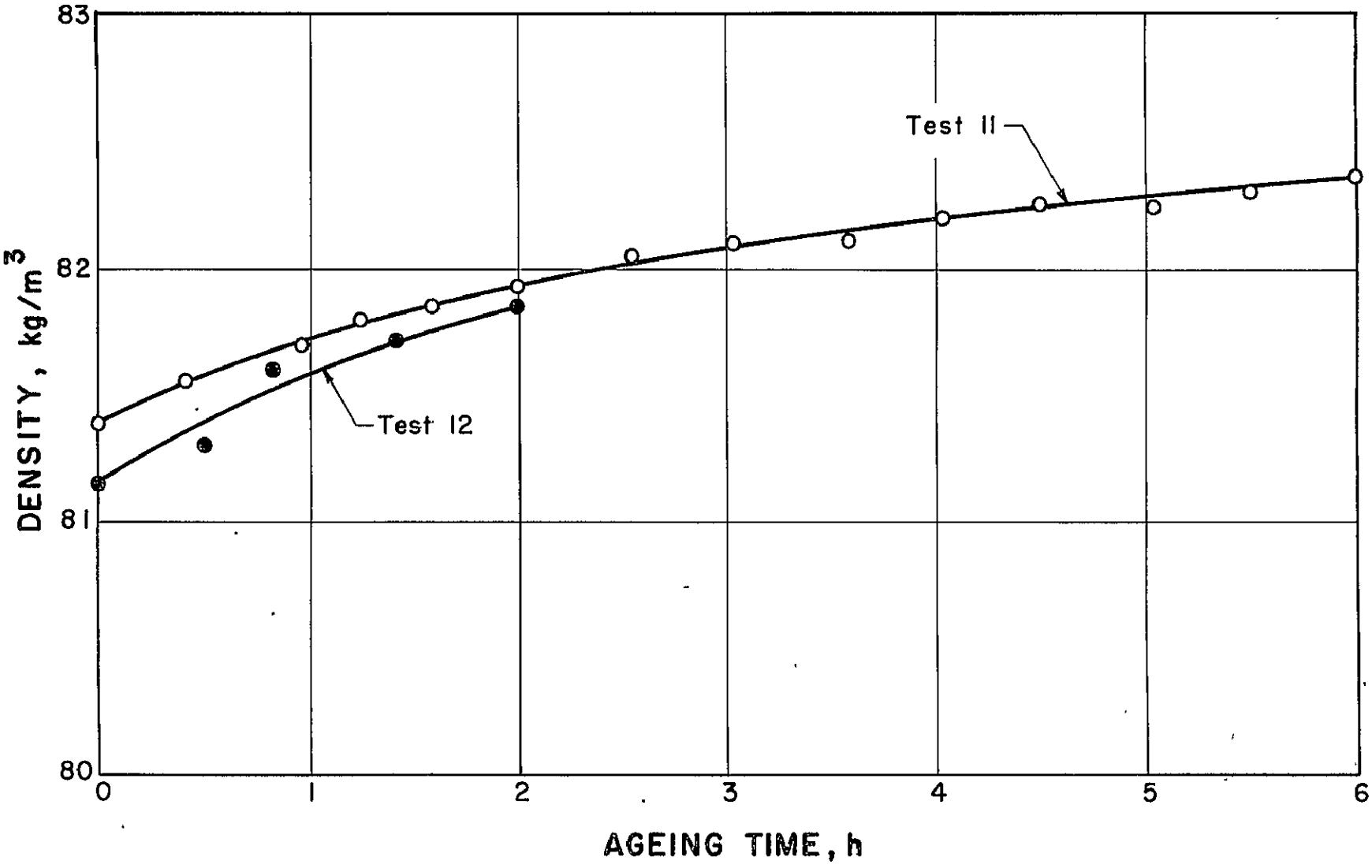

Figure 27. Settled Density vs. Time

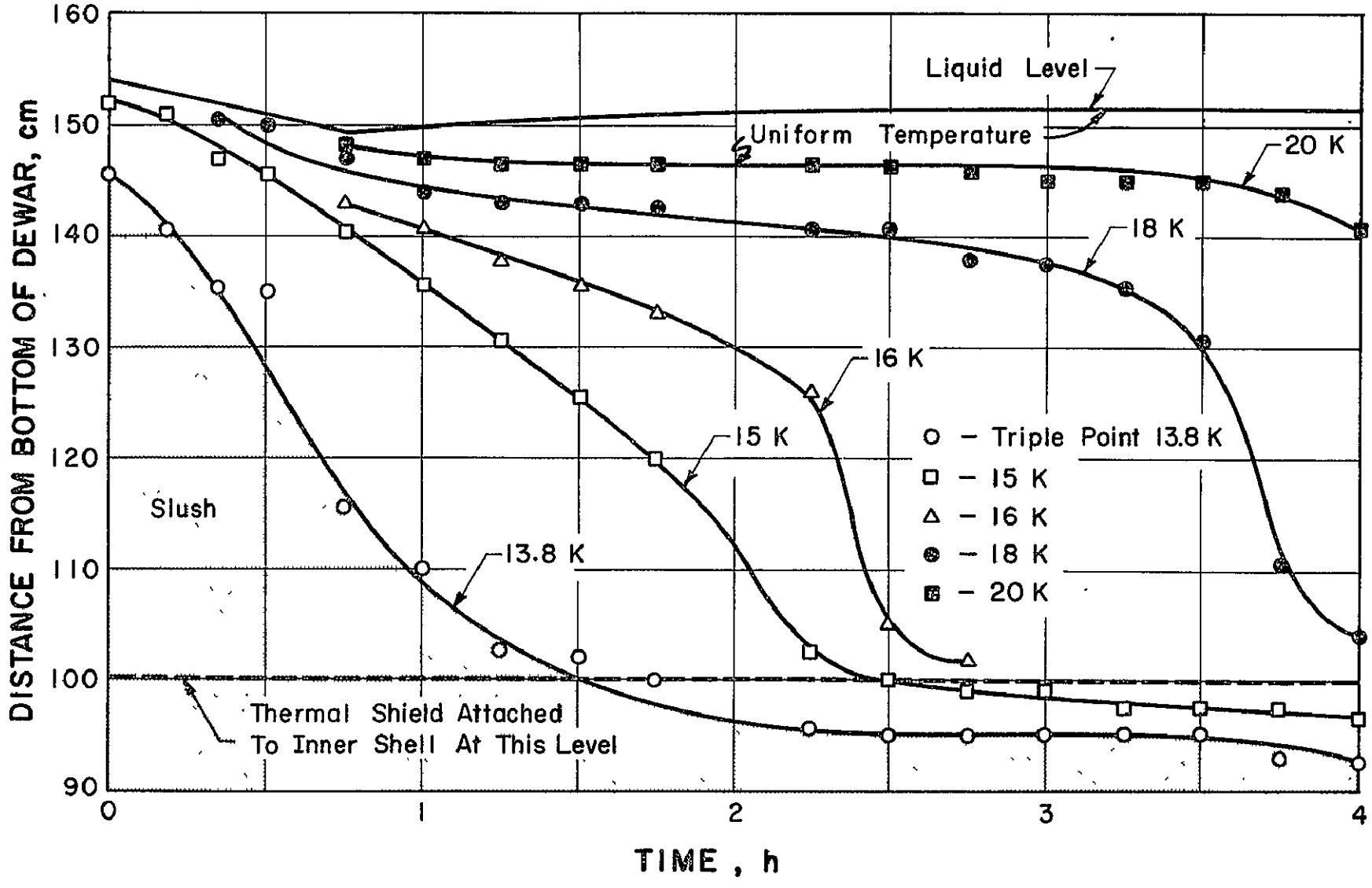

Figure 28. Temperature Stratification--Test 12

the dewar heat leak. A mass and energy balance was calculated and the upgrading was equivalent to supplying 138 watts (471 BTU/hr) of refrigeration. This value is greater than the 100 watts (341 BTU/hr) heat leak determined from the nitrogen boil-off tests but included in this value are the transfer losses, part of which is the cool-down of the line between the generator and the ball valve. This section could not be precooled with liquid hydrogen from the supply dewar.

A second test was made to determine the amount of slush required to accumulate solids in the dewar when it initially contained normal boiling liquid. Upgrading, starting with normal boiling liquid, is probably the technique that will be used in filling storage vessels. In this test the upgrading dewar contained 0.44 m^3 (15.5 ft^3) of normal boiling liquid. After three batches of slush were transferred into the dewar the resulting solid fraction was 0.19. An accounting of the mass and energy transfer indicated a final solid fraction of 0.35, not accounting for the system heat leak. A heat leak of 125 watts (426 BTU/hr) would result in the 0.19 solid fraction measured. This value is slightly higher than the 100 watts (341 BTU/hr) determined by nitrogen boil off but does include transfer losses for the three batches of solid. As stated in the previous test, a section of the transfer line cannot be precooled so the transfer losses would be significant. The test does prove that upgrading can start with normal boiling liquid and the results can be predicted by calculation.

To evaluate the effects on temperature stratification of a high heat leak system, a series of tests were conducted with a good dewar vacuum and poor dewar vacuum. Tests were run in which slush was mixed at frequent intervals to prevent stratification and the resulting solid melting rate was used to determine heat leak. The dewar vacuums were 66 $\mu N/m^2$ (5×10^{-7} torr) and 0.53 N/m^2 (4×10^{-3} torr). Helium gas was used to produce the poor vacuum. The resulting solid melting

rates are shown on figure 29 with the calculated heat leaks. The heat leak with good vacuum determined by this method is higher than that given in section 4.0 or as stated above and is attributable to the effects of stirring. When the slush is vigorously stirred, the ullage gas is cooled as it exchanges heat with the solid and subcooled liquid at the surface and the gas is partially condensed. This condensing of ullage gas as well as some vent line gas results in a higher heat loss than occurs with no stirring.

During the stratification tests there was no stirring, so the heat leaks determined with stirring are not the same as those during the stratification tests. However, since both heat leak tests were made with vigorous mixing, the difference in heat flux should be nearly the same as the difference in heat flux in the stratification tests. After making the two tests to approximate the change in heat flux, a temperature stratification test was made in the dewar with the high heat flux. These data are shown in figure 30. The effect of the shield is very great in this test as no significant temperature gradient occurs above the shield level. Once all of the liquid above the shield reaches its boiling point, the total temperature gradient occurs over a 10 cm (4 in.) interval and progresses deeper into the dewar as solids are melted. The tests are not indicative of what would occur in a vessel with no shield but they do indicate that a very high heat flux does not introduce unexpected results such as fusing together of solids. No attempt is made to calculate the change in heat flux because of the complications introduced by the shield.

After slush had been aged several hours, attempts were made to flow it out of the dewar without prior mixing to see if aged, unmixed slush would flow as well as fresh, unmixed slush. In the first experiment, the ullage pressure in the dewar was pumped to triple-point pressure to precool the warm liquid that had accumulated during aging as is

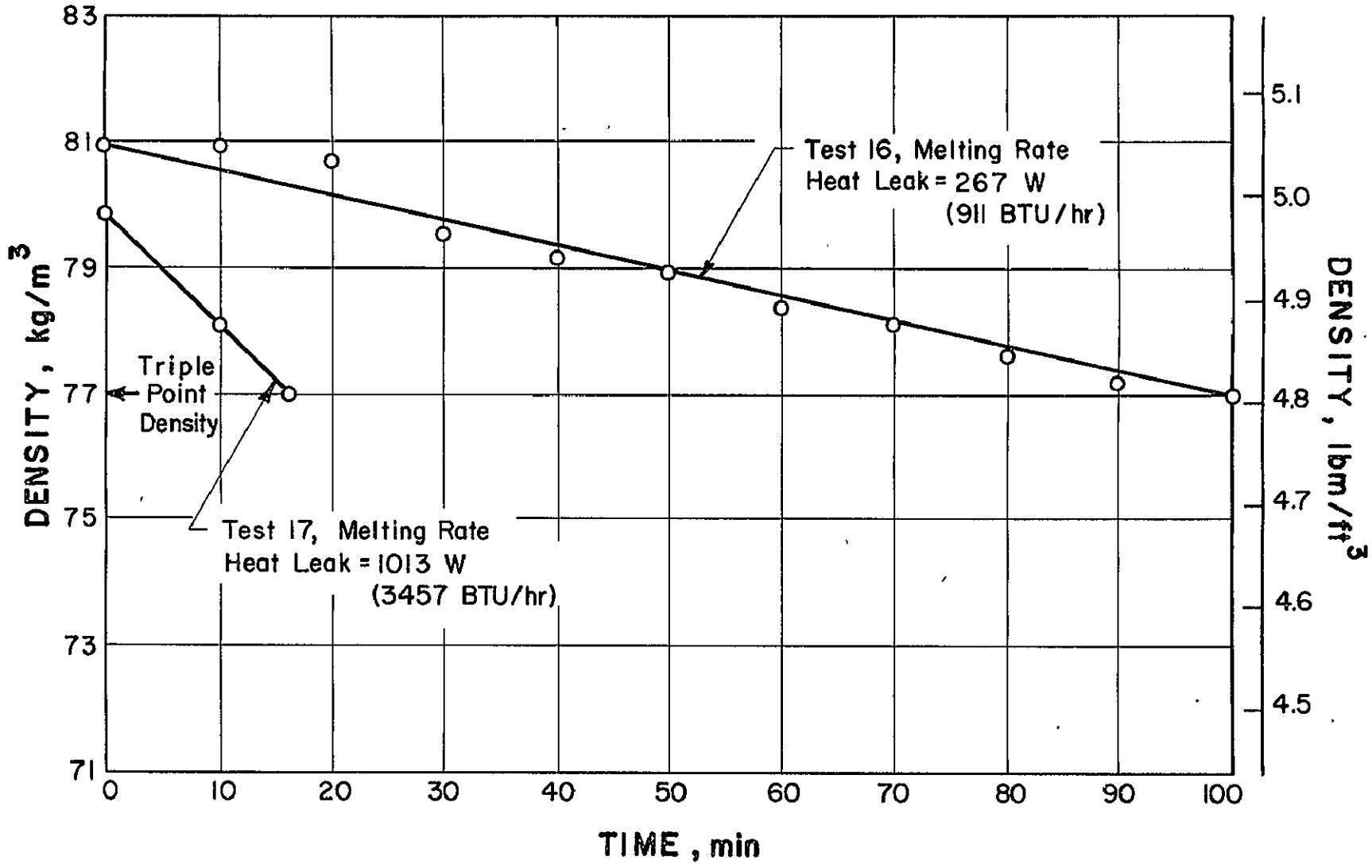

Figure 29. Heat Leak from Solid Melting Rate

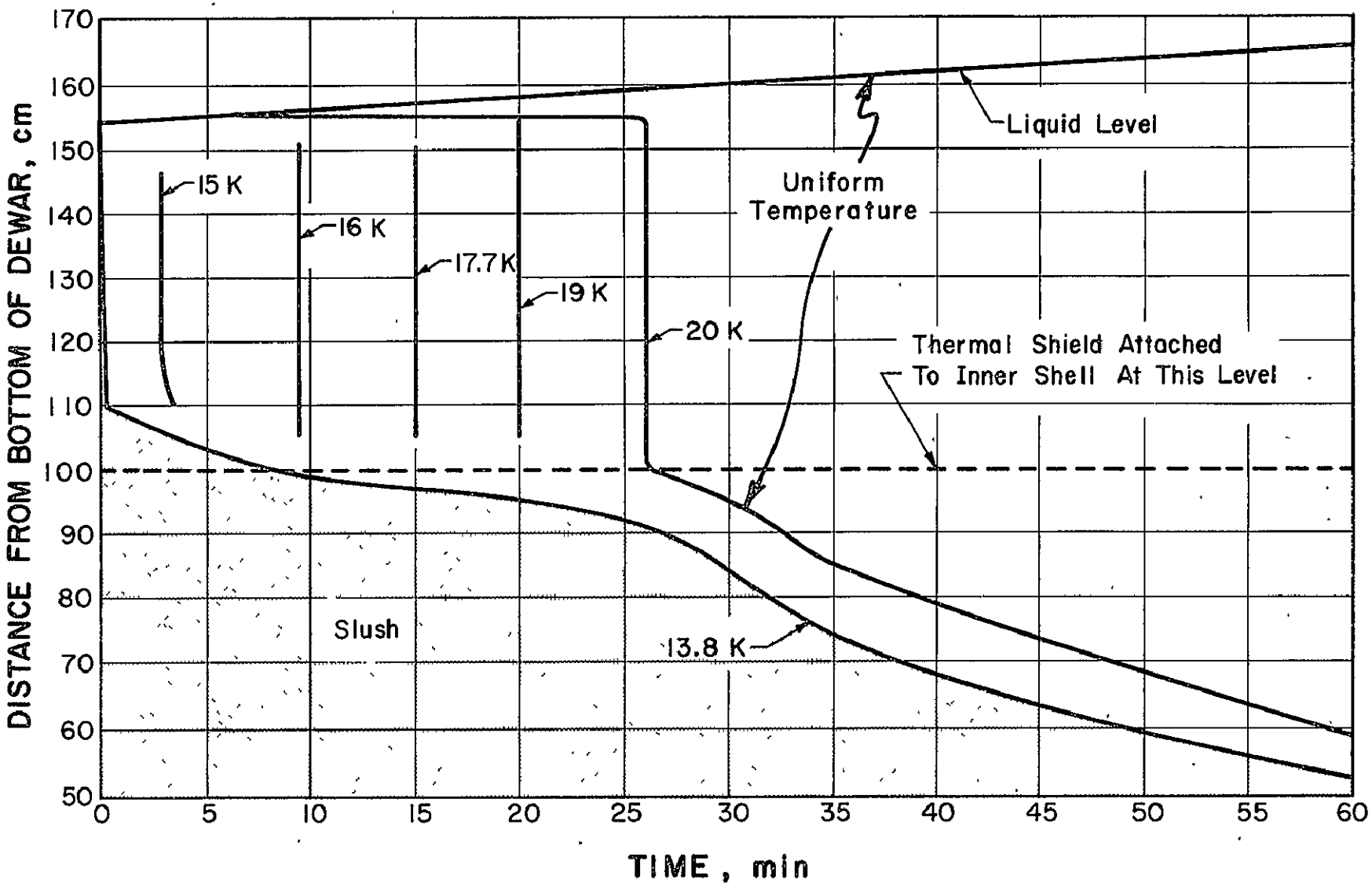

Figure 30. Temperature Stratification--Test 17

shown by stratification data. After the liquid was completely cooled to triple point, the fill line valve was opened and the transfer attempted. Solids around the transfer line broke down and were carried out with the liquid. However, this only occurred over a diameter of about 0.46 m (1.5 ft). The remainder of the solid tended to stand, maintaining a perpendicular wall even to the point of exposing solid to the ullage gas. On occasion some solids would break off and fall into the open area around the transfer line. The total height of solid was about 79 cm (31 in.). Stirring during the later part of the transfer caused all of the solid to be mixed and the remaining slush was transferred out of the dewar. The aged slush does not flow out of the vessel as readily as fresh slush. Fresh solids do not support a vertical edge to nearly the height of the aged solids.

Another test was made in which the stratified liquid was not precooled before outflow. In this case considerable melting of solid occurred during the outflow as would be expected. Other than melting of solid, the transfer did not appear to be significantly different from the previously described one. The solids did not fuse into an unmixable lump due to the high heat flux from the liquid as mixing of the remaining solids was performed to complete the transfer.

The ullage pressure in a vessel during filling with slush and subcooled liquid is of great interest because of the possibility of collapsing a thin-walled vessel such as a rocket tank. Structural collapses have been encountered even when transferring normal boiling liquid hydrogen into such a vessel. Several tests were conducted in the upgrading dewar to determine the ullage pressure while flowing slush into an empty vessel. In the first test, the warm vessel, near ambient temperature, was evacuated and filled with hydrogen gas. Approximately 0.3 m^3 (10.6 ft^3) of 0.45 solid fraction slush was made in the generator. This slush was then transferred into the upgrading dewar at a pressure of 69 kN/m^2

gauge (10 psig). The upgrading dewar was vented through the 1-1/2 inch vent check valve which opened at 14 kN/m^2 gauge (2 psig). The ullage pressure built to 41 kN/m^2 gauge (6 psig) then dropped back to the vent check valve pressure of 14 kN/m^2 gauge (2 psig) where it remained until the end of the transfer.

A second test was conducted in which the upgrading dewar was prechilled with liquid hydrogen. In this test the dewar pressure dropped below atmospheric, to approximately 28 kN/m^2 absolute (4 psia). Although these test results may be geometry dependent, they indicate that pressures below atmospheric can develop in thin-walled rocket tanks.

4.3 Thermally Induced Pressure Oscillations

During the slush hydrogen program a number of problems have occurred because of thermally induced pressure oscillations [Daney, et al., 1967]. A related problem occurred in the transfer line of the upgrading system. In the section of line between the supply valve and the upgrading dewar fill and return line, the transfer line has a vertical rise of about 1.5 m (5 ft) to the level of the return valve. If this section of line was not precooled with liquid from the supply dewar prior to transfer of slush, violent pressure surges occurred in the transfer line. It was concluded that the pressure surges were caused by the alternate forming and collapsing of a gas pocket in the vertical section. Initially, this section fills with warm gas, then, as slush reaches the top of the section and flows by gravity down into it, the solid and subcooled liquid condenses the gas. Because the lower portion of the line and the valve body are warm, liquid is immediately flashed to vapor blowing the slush out of the vertical section. The cycle repeats causing an oscillation of pressure which continues until the vertical line and valve are cooled to below boiling temperature. Designers of slush transfer lines should be advised that thermal induced pressure oscillations can be a problem in

well insulated sections during cooldown if slush or subcooled liquid is used as the cooling fluid.

4.4 Conclusions

From the upgrading and aging experiments a number of conclusions can be drawn.

1. Slush hydrogen solid fraction can be increased in a vessel by transferring out liquid hydrogen and retaining solids with a screen having openings of 0.6 mm (0.023 in.).
2. In slush 2 hours or less in age, the maximum solid fraction that remains fluid enough to mix and transfer is approximately 0.54.
3. In slush 2 hours or less in age, the maximum solid fraction that can be attained and still maintain sufficient liquid to cover the solid is approximately 0.54.
4. Solid fraction in settled slush as high as 0.64 can be achieved by upgrading without mixing and by aging to approximately 6 hours or more.
5. Mixing during upgrading or aging of slush reduces the solid fraction attainable in a given time interval.
6. It is possible to upgrade to solid fractions in excess of 0.5 starting with the upgrading vessel filled with normal boiling liquid.
7. If the heat leak of the vessel is known and the transfer losses are known, it is possible to calculate the solid fraction attainable by the upgrading techniques described here.
8. It is possible and even probable that the ullage pressure in a vessel will collapse when slush is transferred into the vessel especially if no helium gas is used as pressurant and if the vessel is precooled to any extent.

6

5. Mixing of Slush

During the fuel replenishing phase of a rocket launch, slush of uniform solid fraction will be transferred into the flight vessel to maintain a constant solid fraction of a fixed volume of fuel. To meet this requirement, slush of a uniform solid fraction will be needed for the transfer, therefore, a homogeneous mixture will be required from the slush system. To provide homogeneous mixtures of the fresh and aged slush, total mixing will be a necessity. This mixing requirement, as well as possible flight mixing requirements, were the impetus for investigating techniques for mixing slush in the upgrading dewar.

The tests were conducted with stirring devices of two types. The first experiments were with mixers, similar to those commercially available, that would be suitable for storage vessels. These are basically propellers of different numbers and configurations. The second type mixer tested was one that could be applied to flight hardware. The unit was a ducted propeller with transfer ducting to distribute the discharge to create local mixing.

5.1 Propeller Mixers

The first propeller used had three blades and was 20.3 cm (8 in.) in diameter, with a 15.2 cm (6 in.) pitch, that is, the blades were designed to give 15.2 cm (6 in.) of lift per revolution. This unit, which was a commercial mixing propeller, is shown in figure 31. The drive for the mixer was an air motor mounted on the 23 cm (9 in.) access port in the top plate. The maximum angular velocity of the motor was 47.1 rad/s (450 rpm). The driving shaft from the motor passed through a sealing box which contained a double O-ring rotating seal. The volume between the O-rings was purged with helium gas under pressure to prevent air from entering the dewar when pressures were below atmospheric. With this arrangement helium instead of air would be drawn into the

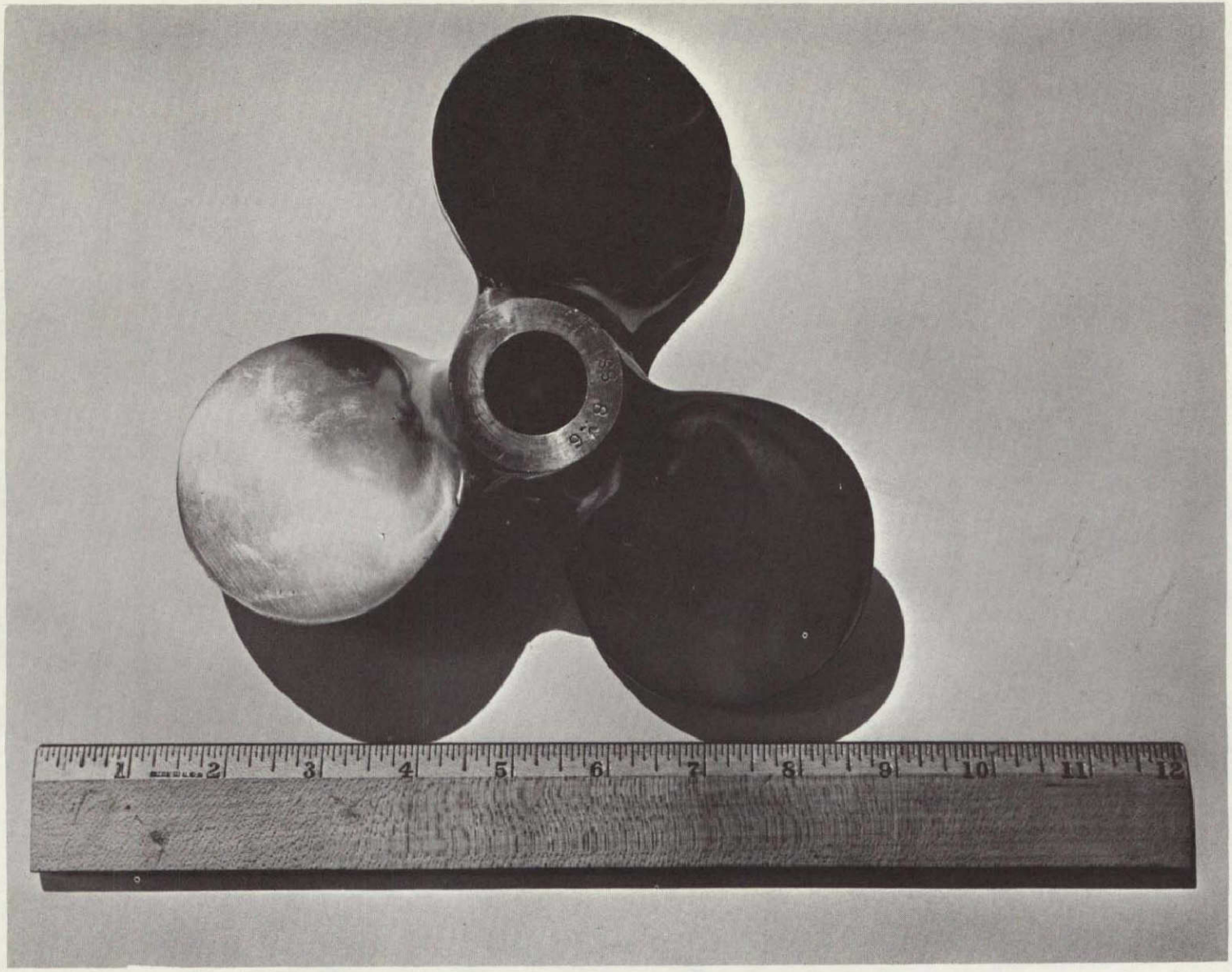

Figure 31. Three-Bladed Mixing Propeller

dewar if a leak occurred at the rotating seal. The mixing unit was mounted 17.8 cm (7 in.) from the center of the dewar and 25.4 cm (10 in.) from the bottom of the dewar. The off-center location was selected to reduce swirl and centrifuging of solids. Liquid was easily mixed with the unit and slush to solid fractions of about 0.25 mixed well, but when solid fractions reach 0.45 no mixing was visible.

Since the three-bladed unit did not mix, a new mixer was built. This unit consisted of four 40.6 cm (16 in.) two-bladed propellers. One of these propellers is shown in figure 32. They were mounted 11.4 cm (4-1/2 in.) from the dewar center and equally spaced at 49.5 cm (19.5 in.) intervals with the lowest one at 25.4 cm (10 in.) from the dewar bottom. The same air motor was used to drive the mixer. This unit stirred slush of a solid fraction in excess of 0.5. To further evaluate the mixing using the large propeller, one was removed and the spacing was changed to 61.6 cm (24-1/4 in.) with the lowest propeller remaining at the 25.4 cm (10 in.) level. During the next test, slush was aged and reached a solid fraction of 0.62. At the end of the aging period the settled solids covered two of the three propellers and liquid covered all three. The unit stirred the settled aged slush quite easily.

In the next mixing experiment only one 40.6 cm (16 in.) propeller was used at the 25.4 cm (10 in.) level. In this test the slush was unmixed and aged for 7 hours to a solid fraction of 0.64. At the end of the aging period the single propeller would not mix the settled solids immediately. After the mixer had run at maximum angular velocity, which was about 23.6 rad/s (225 rpm), for about 5 minutes, the settled bed of solids finally lifted and total mixing occurred. The angular velocity of the mixer immediately increased to approximately 42 rad/s (400 rpm).

For the next experiment two propellers were used. The lowest one was moved down to 13.3 cm (5-1/4 in.) from the bottom and the upper one was at 133.3 cm (52-1/2 in.). The center of the mixer was

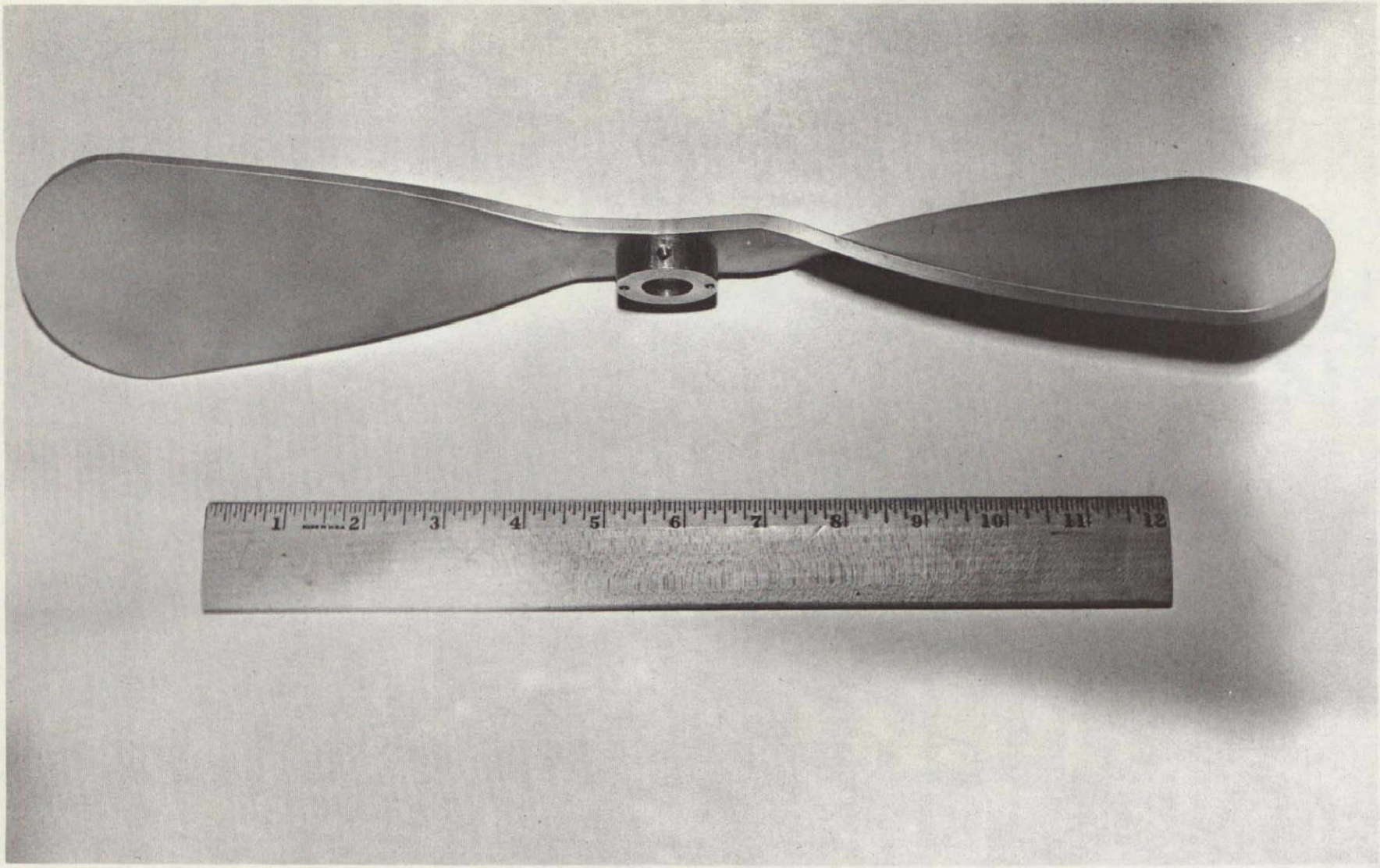

Figure 32. Two-Bladed Mixing Propeller

moved to 10 cm (4 in.) from the dewar center line. This position of the mixer was selected because of interference problems with the microwave densitometer described in section 3. This unit was also driven by the air motor. The unit mixed, but vortexing occurred and solids tended to centrifuge to the dewar walls, apparently because the center of the mixer was too near the dewar center. Mixing was not considered satisfactory.

The next configuration was selected to accommodate the microwave unit and still provide propellers in a more desirable location. The mixing center was moved to 12.7 cm (5 in.) from the dewar center and three propellers were used. The lowest one was 17.8 cm (7 in.) from the bottom and was one of the 40.6 cm (16 in.) propellers. Two shorter ones were used at 84 cm (33 in.) and 126 cm (49-1/2 in.) from the bottom. These propellers were 33 cm (13 in.) in diameter and were made by cutting the tips off the 40.6 cm (16 in.) units. This configuration mixed aged settled slush to 0.6 solid fraction.

5.2 Ducted Propeller Mixer

The principal reason for testing a ducted propeller unit was to determine if high solid fraction slush could be mixed by high velocity streams of fluid. If this were the case, a high volume, low head pump could conceivably be used to pump slush to suitably located discharge nozzles in a flight vessel and thereby mix slush with a relatively simple installation.

The three-bladed propeller described in section 5.1 was mounted in a 21 cm (8-1/4 in.) diameter duct as shown in figures 33 and 34. The bottom opening of the duct was 15 cm (6 in.) from the dewar bottom. The propeller was 12.7 cm (5 in.) from the bottom of the duct which extended to the top plate. Three pairs of 8.3 cm (3-1/4 in.) diameter holes were cut in the 21 cm (8-1/4 in.) diameter duct at 66 cm (26 in.), 96.5 cm

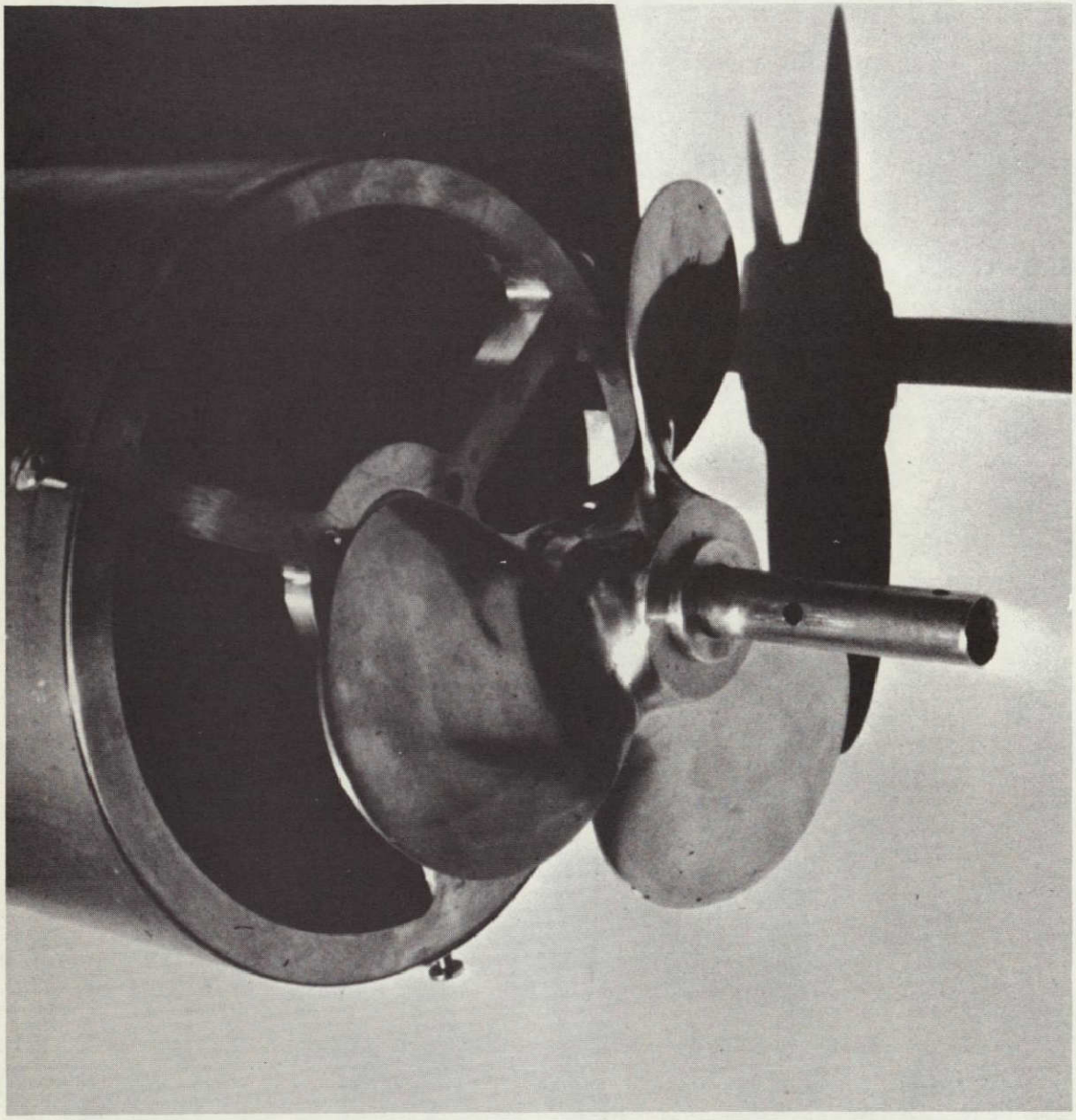

Figure 33. Propeller Assembly for Duct Mixer

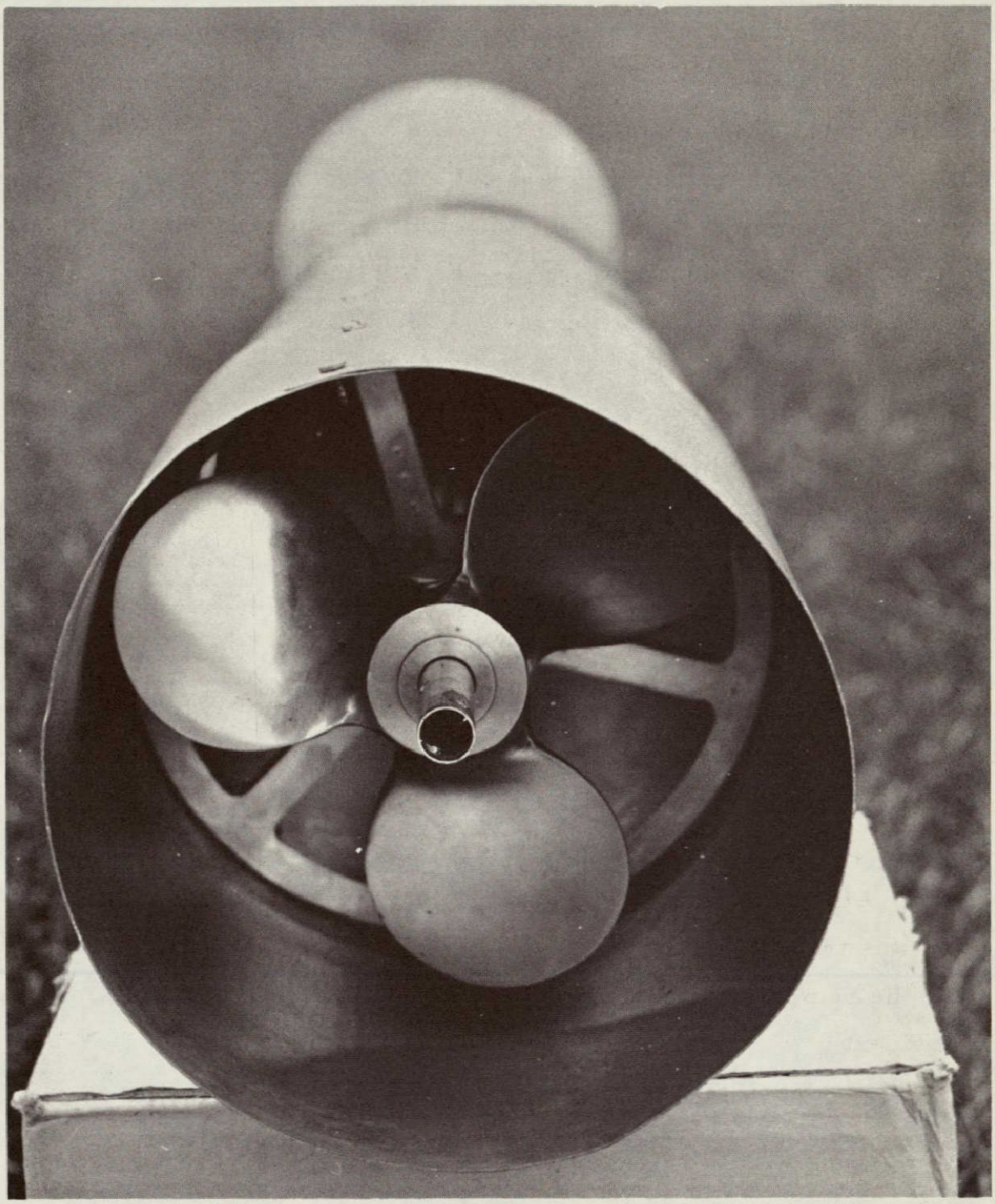

Figure 34. Propeller in Duct Mixer

(38 in.), and 127 cm (50 in.) from the bottom. The holes were 2.1 rad (120°) apart. The center of the duct was mounted at 17.8 cm (7 in.) from the center of the dewar. A top baffle was placed in the duct at 2.2 m (87 in.) and another hole was cut above the baffle to prevent slush from impinging on the cover plate and chilling the cover seal. The assembly was mounted on the access cover with the driving motor externally mounted using the rotating seal box described in section 5.1. This unit is shown in figure 35.

In the first test the air motor was used as the drive. Mixing was good with liquid and low solid fraction slush. When the solid fraction reached 0.45 the mixing stopped. No fluid was being pumped by the propeller and no discharge was visible from the ports. The pneumatic motor was replaced with a 178 rad/s (1700 rpm) electric motor. With this unit, slush to a solid fraction of 0.55 could be mixed very well.

In one experiment the liquid was drawn off approximately 15 cm (6 in.) below the solid level. After the liquid level was drawn this low the mixer would not pump the slush out of the ports. A very small amount oozed out of the lowest visible port when the mixer was first started. After that no pumping occurred and, therefore, no mixing. Liquid was transferred back into the dewar to well above the solid level before the pumping action of the mixer again started. Total mixing was then evident.

From the visual observation of the mixing that occurred when the ducted propeller pumped fluid, it appeared that mixing was more than adequate. This pumping rate is equivalent to about one-twelfth of the fluid volume per second, based on the assumption that the propeller is 50% effective in moving liquid. Since the actual effectiveness of the propeller is not known and the minimum pumping rate as well as minimum number of discharge nozzles were not determined, the limiting conditions are not predictable with available information.

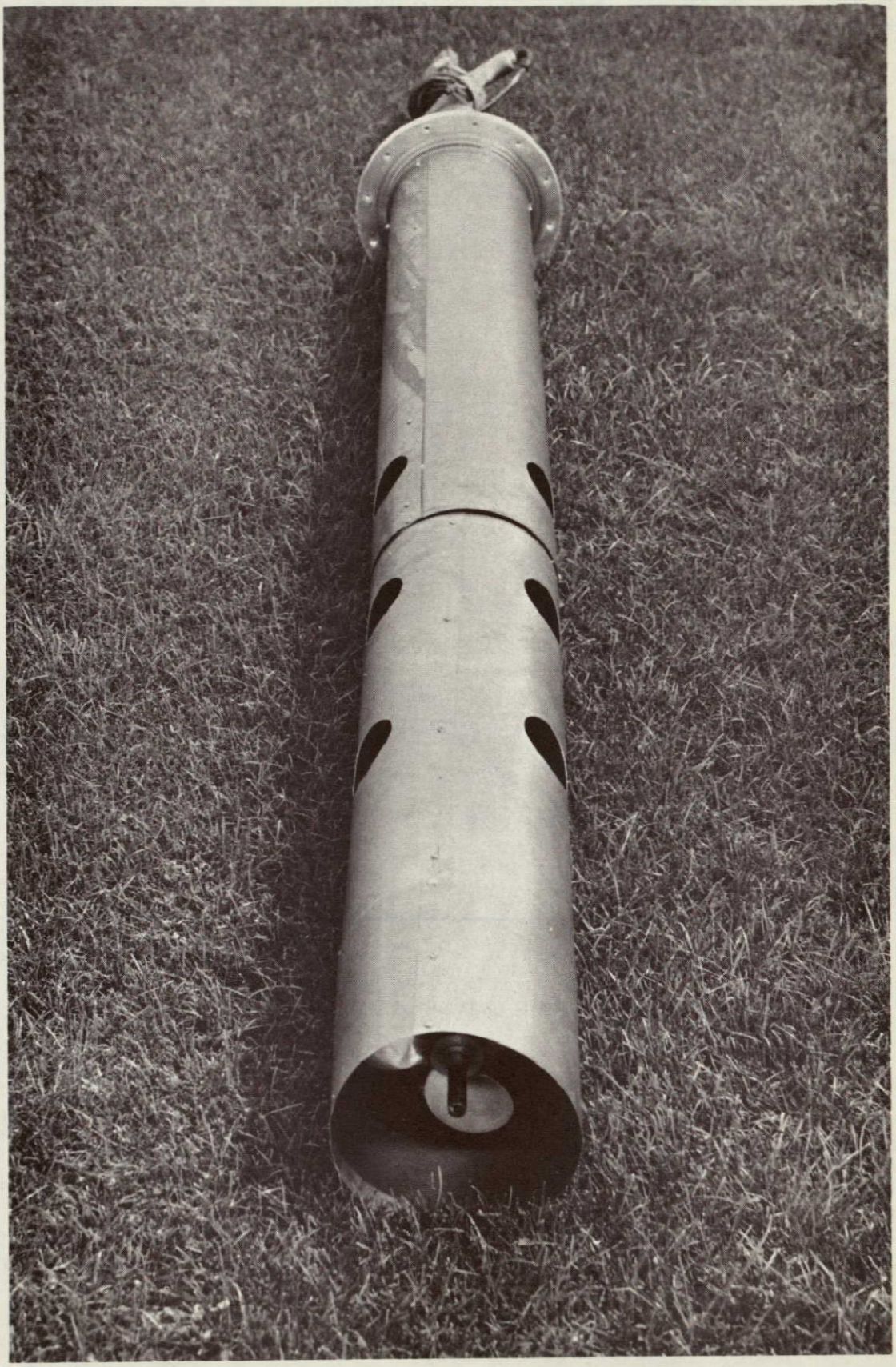

Figure 35. Duct Mixer

5.3 Conclusions

Four conclusions are made from the mixing studies.

1. Aged slush hydrogen to 0.64 solid fraction can be mixed if the solids have never been exposed to the ullage gas.
2. The amount of stirring required for homogeneous mixing is significantly greater for mixtures of a solid fraction of 0.45 and higher than that required for liquid hydrogen.
3. Homogeneous mixing of slush hydrogen to solid fractions of 0.55 can be attained by fluid discharge from nozzles properly placed.
4. Mixing of slush of solid fraction in excess of 0.5 can be accomplished with open-blade propellers if tip velocity is high and if the blades are properly spaced. The mixing center should be off the vessel center at least 1/6 of the diameter to prevent vortexing and centrifuging of solids.

6. References

Daney, D. E., P. R. Ludtke, C. F. Sindt, and D. B. Chelton (1967), Slush Hydrogen Fluid Characterization and Instrumentation Analysis, NBS Report 9701.

Kittel, Charles (1961) Introduction to Solid State Physics, Second Edition, (John Wiley & Sons, Inc., New York, N.Y.).

Mann, D. B., P.R. Ludtke, C. F. Sindt, and D. B. Chelton (1966), Liquid-Solid Mixtures of Hydrogen Near the Triple Point, Advances in Cryogenic Engineering 11, pp. 207-217, K. D. Timmerhaus, Ed. (Plenum Press, New York, N.Y.).

Roder, H. M., L. A. Weber, and R. D. Goodwin (1965), Thermodynamic and Related Properties of Parahydrogen from the Triple Point to 100 K at Pressures to 340 Atmospheres, NBS Monograph 94.

Sindt, C. F., P. R. Ludtke, and D. E. Daney (Feb. 1969), Slush Hydrogen Fluid Characterization and Instrumentation, NBS Technical Note 377.

Weitzel, D. H., C. F. Sindt, and D. E. Daney (1968), Hydrogen Slush Density Reference System, Advances in Cryogenic Engineering 13, pp. 523-533, K. D. Timmerhaus, Ed. (Plenum Press, New York, N. Y.).

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