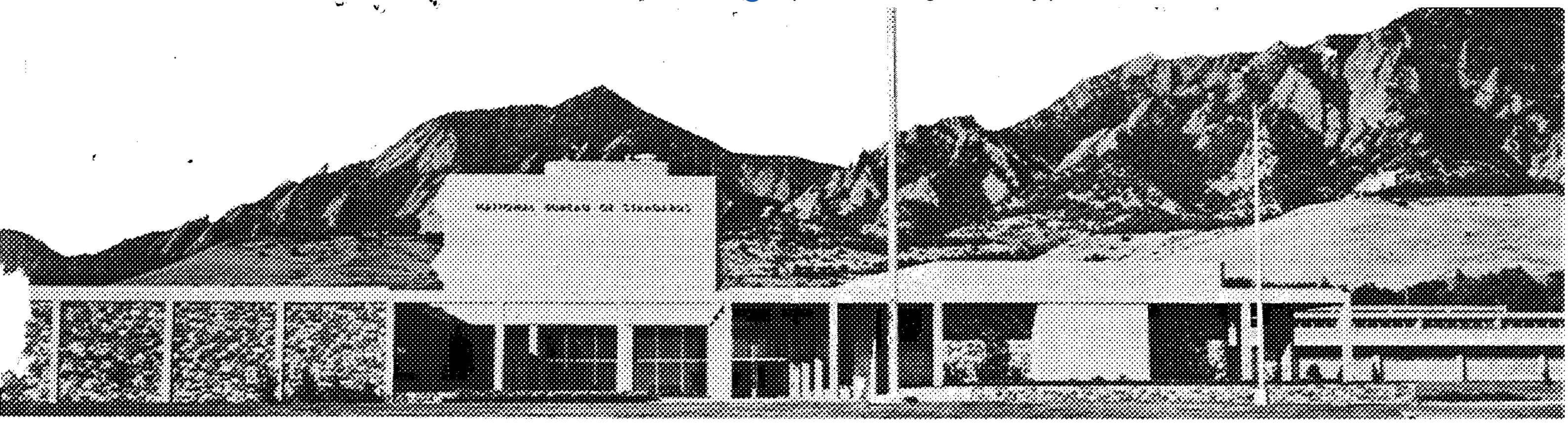


NBS REPORT

9701

SLUSH HYDROGEN FLUID CHARACTERIZATION AND INSTRUMENTATION ANALYSIS

by

D. E. Daney, P. R. Ludtke, C. F. Sindt, and D. B. Chelton

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
BOULDER LABORATORIES
Boulder, Colorado

THE NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards[1] provides measurement and technical information services essential to the efficiency and effectiveness of the work of the Nation's scientists and engineers. The Bureau serves also as a focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. To accomplish this mission, the Bureau is organized into three institutes covering broad program areas of research and services:

THE INSTITUTE FOR BASIC STANDARDS . . . provides the central basis within the United States for a complete and consistent system of physical measurements, coordinates that system with the measurement systems of other nations, and furnishes essential services leading to accurate and uniform physical measurements throughout the Nation's scientific community, industry, and commerce. This Institute comprises a series of divisions, each serving a classical subject matter area:

—Applied Mathematics—Electricity—Metrology—Mechanics—Heat—Atomic Physics—Physical Chemistry—Radiation Physics—Laboratory Astrophysics[2]—Radio Standards Laboratory,[2] which includes Radio Standards Physics and Radio Standards Engineering—Office of Standard Reference Data.

THE INSTITUTE FOR MATERIALS RESEARCH . . . conducts materials research and provides associated materials services including mainly reference materials and data on the properties of materials. Beyond its direct interest to the Nation's scientists and engineers, this Institute yields services which are essential to the advancement of technology in industry and commerce. This Institute is organized primarily by technical fields:

—Analytical Chemistry—Metallurgy—Reactor Radiations—Polymers—Inorganic Materials—Cryogenics[2]—Office of Standard Reference Materials.

THE INSTITUTE FOR APPLIED TECHNOLOGY . . . provides technical services to promote the use of available technology and to facilitate technological innovation in industry and government. The principal elements of this Institute are:

—Building Research—Electronic Instrumentation—Technical Analysis—Center for Computer Sciences and Technology—Textile and Apparel Technology Center—Office of Weights and Measures —Office of Engineering Standards Services—Office of Invention and Innovation—Office of Vehicle Systems Research—Clearinghouse for Federal Scientific and Technical Information[3]—Materials Evaluation Laboratory—NBS/GSA Testing Laboratory.

---

[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D. C., 20234.

[2] Located at Boulder, Colorado, 80302.

[3] Located at 5285 Port Royal Road, Springfield, Virginia 22151.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT		NBS REPORT
31505-3150452	December 29, 1967	9701

SLUSH HYDROGEN FLUID CHARACTERIZATION AND INSTRUMENTATION ANALYSIS

by

D. E. Daney, P. R. Ludtke, C. F. Sindt, and D. B. Chelton

Summary Report to
George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama

Cryogenics Division
Institute for Materials Research
National Bureau of Standards
Boulder, Colorado

NBS

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

TABLE OF CONTENTS

LIST OF FIGURES

ABSTRACT

The pumping characteristics of slush hydrogen were investigated with a Saturn S-IV B liquid hydrogen chilldown pump. As would be predicted from theory, pump performance and cavitation for slush and liquid hydrogen are the same when the difference in fluid density is considered. No pump wear or damage can be attributed to slush.

A nuclear radiation attenuation densitometer was installed on the 450-liter generator. The precision of the densitometer is comparable to the accuracy of the liquid and slush density determination methods to which it was compared.

Flow data were taken in the flow loop for triple-point liquid and slush hydrogen. A comparison of the data to the pressure drop prediction of simple slurry models was made. The pressure drop of freshly prepared slush is less than that of triple-point liquid at velocities above 12 feet per second.

Slush was transferred through several typical cryogenic globe valves and bayonet fittings to evaluate suspected problem areas. Thermally induced pressure oscillations occurred where clearances were excessive.

A new apparatus was constructed to perform slush hydrogen particle studies in more detail. Several aging experiments, up to 100 hours, were completed in this apparatus. Slush solid fractions greater than 0.60 were obtained.

Key Words: Cavitation, cryogenic pump, densitometer, hydrogen pump, slurry flow, slush hydrogen, solid hydrogen, thermal oscillations.

SLUSH HYDROGEN FLUID CHARACTERIZATION AND INSTRUMENTATION ANALYSIS

D. E. Daney, P. R. Ludtke, C. F. Sindt, and D. B. Chelton

INTRODUCTION

An analytical and experimental study to characterize liquid-solid mixtures of hydrogen is presently in progress. The following is a summary report for work completed during the period December 1, 1966 to December 29, 1967, covering work performed under NASA Contract H-25490A entitled, "Slush Hydrogen Fluid Characterization and Instrumentation Analysis." The general scope of the program is to develop knowledge and understanding of production techniques, storage, transfer, and pumping of slush hydrogen.

Work prior to the above reporting period has included the investigation and development of slush production techniques. The size distribution and terminal velocity of the solid hydrogen particles were determined. Aging effects on the particles were investigated. A flow loop was designed and constructed. The flow loop consists of a 450-liter slush generator with three observation windows, a vacuum insulated transfer line with a transparent section, and a receiver dewar for storage and stratification studies. The flow loop is instrumented with pressure taps, liquid level sensors and a thermocouple probe. Slush stratification profiles of stored slush were determined, and photographic techniques were developed to photograph slush production, flow, and individual solid hydrogen particles.

1. Slush Hydrogen Pump Characteristics

The pumping characteristics of slush hydrogen are of considerable interest since its use as a rocket propellant will require that it be pumped on both ground installations and space vehicles. Theoretical considerations, together with slush flow data, indicate that the pumping characteristics of liquid and slush hydrogen should be the same when the difference in density is considered. In order to experimentally verify this conclusion, a pump program was initiated with the following objectives:

1. To determine if slush can be pumped with a conventional type liquid hydrogen pump.
2. To investigate the effects of slush on inducers, impellers, propellant cooled bearings, and labyrinth and dynamic face seals.
3. To determine pump cavitation characteristics.
4. To check pump operating performance with varying qualities of slush.

1.1 Pump Description

The pump used in the tests was a commercially available centrifugal type chilldown pump designed for the Saturn S-IV B. Figures 1, 2, and 3 show the pump assembled and disassembled. The pump has been modified by the addition of a helium gas driven turbine and by the separation of the discharge volute from the motor housing.* The latter modification was made so that the pump could be inserted into the slush generator through the 6-inch diameter windows.

With the induction motor drive supplied with 400 Hz, ,42 volt, a-c power, the pump runs at a nearly constant speed of 11, 000 rpm and has a nominal flow rate of 135 gpm at 7 psi developed pressure. With the

---

* modification by NASA-MSFC.

B- 69474

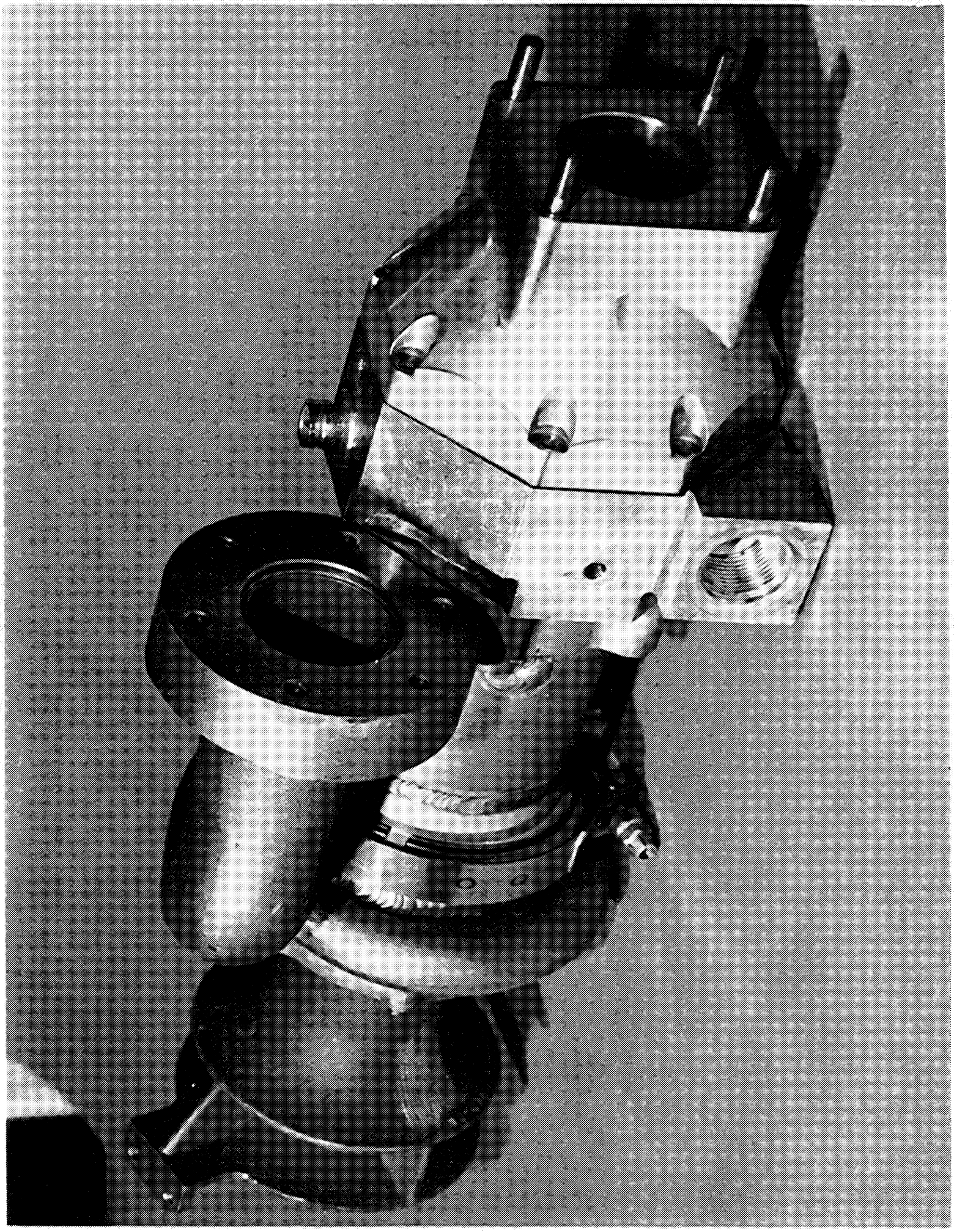


Figure 1. Pump Assembly

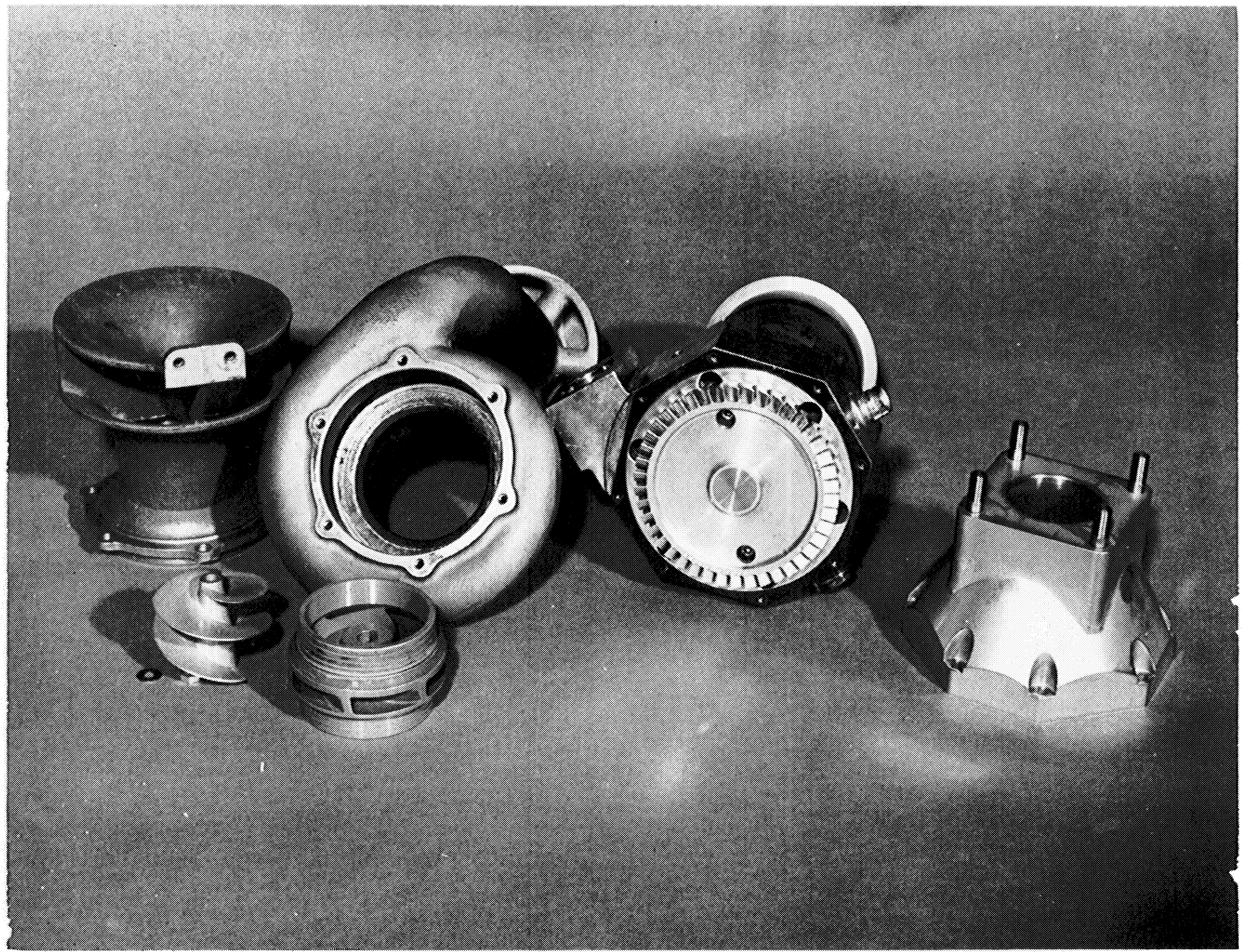


Figure 2. Pump Disassembly

B- 69477

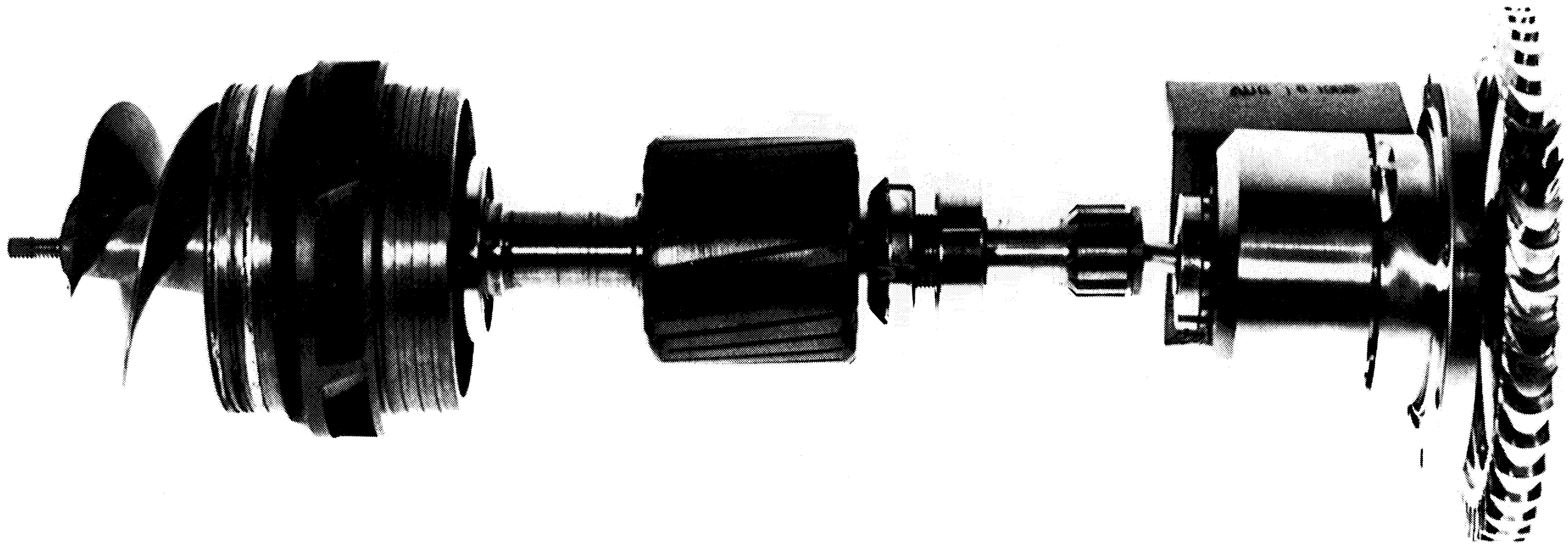


Figure 3. Pump Shaft Assembly

B-70866

helium turbine drive, the pump has a variable speed of 6,000 - 19,000 rpm, flow rates to 350 gpm, and developed pressures to 26 psi. The specific speed range of the pump is 1600 to 3100.

The turbine and pump bearings are stainless steel ball bearings with phenolic bearing cages and are cooled by liquid flow. The impeller diameter is 3-1/2 inches and the minimum dimension of the impeller passage is 1/4-inch.

1.2 Pump Test Facility

Because of the desirability of using the existing slush hydrogen facility, the pump was mounted inside the 450-liter slush generator as shown in figure 4. The generator has three 6-inch diameter, equally spaced observation windows through which the pump was inserted. In order to insert the pump, each component was separately placed through a 6-inch window. The components were then reassembled inside the dewar. A 2-inch discharge tube and smaller helium inlet and discharge lines bolted to the dewar window flange, support the pump.

Figure 5 shows the pump flow system. It consists basically of a slush generating dewar containing the pump and a 2-inch discharge line which passes through the window to a 500-gallon receiving dewar. Slush may be pumped to the receiver or alternately through the return loop back into the generator. The globe valves provided in the transfer line allow control of the flow and developed pressure of the pump.

The turbine is driven by helium gas, pre-cooled with liquid nitrogen. A square wave inverter requiring a 54 volt d-c input, supplied the 400 Hz., 42 volt, a-c power for the motor drive.

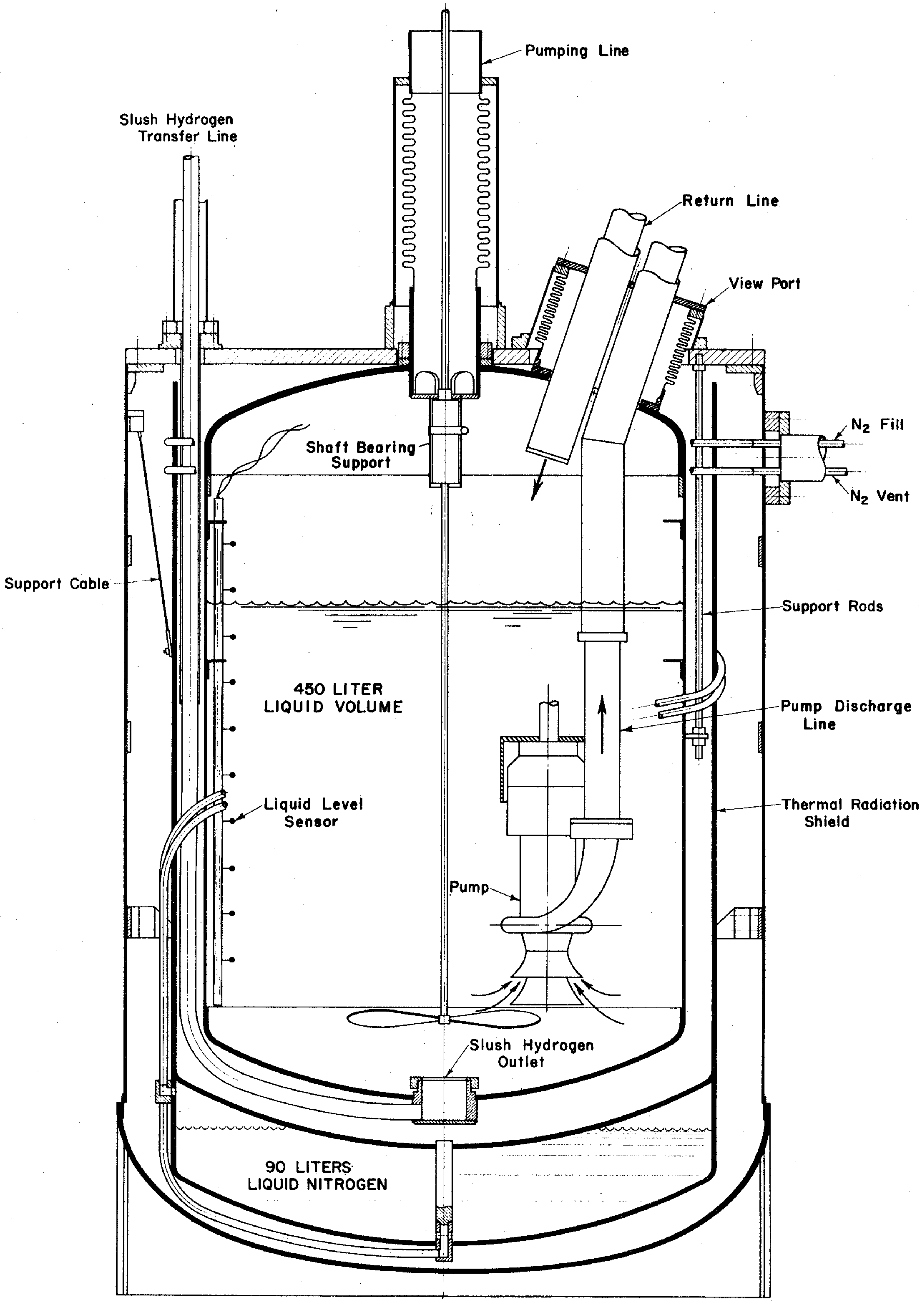
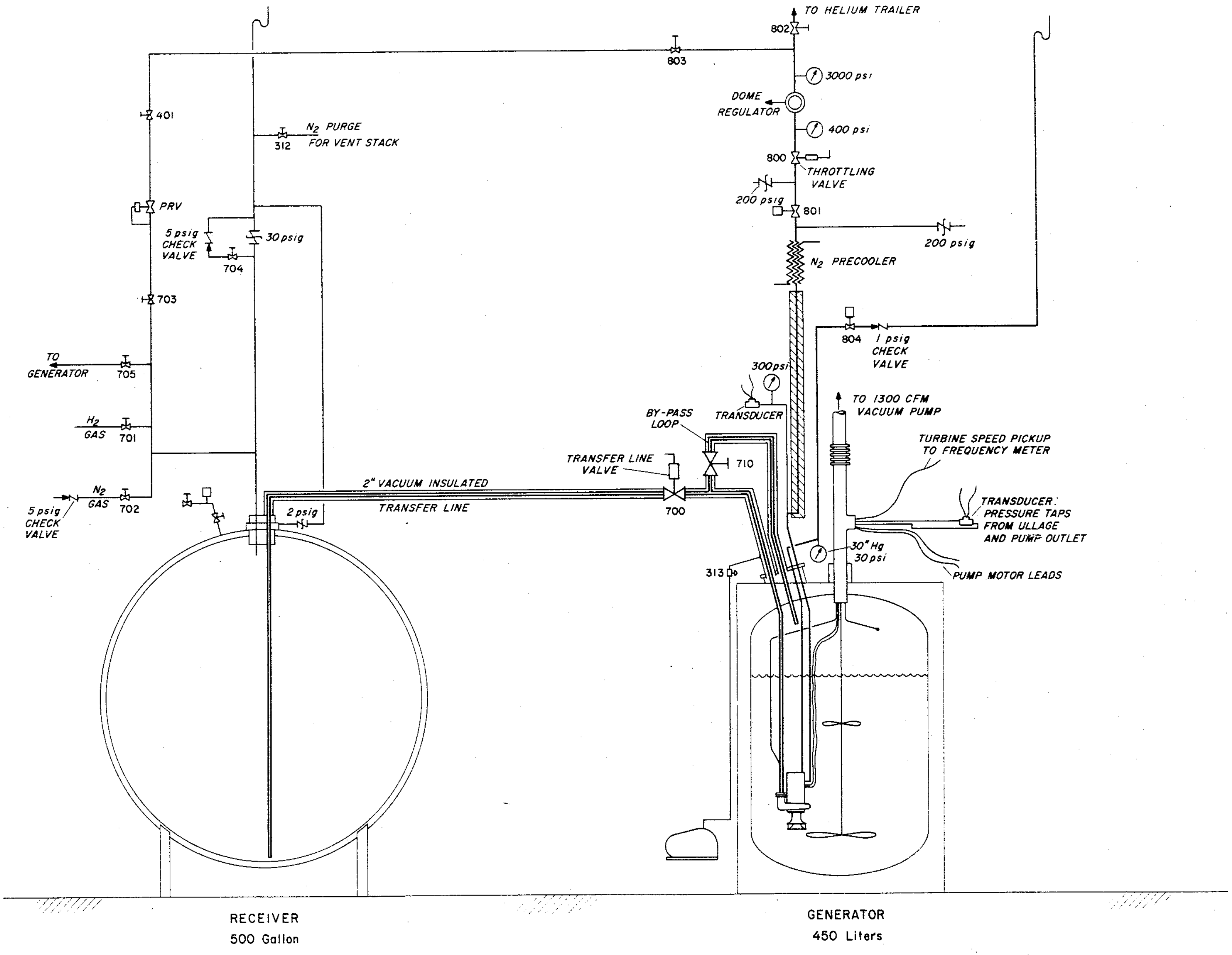


Figure 4. Pump Installation.

B- 73156



B-72343

Figure 5. Pump Flow Schematic

1.3 Instrumentation and Data Acquisition

The data, with the exception of the d-c power to the inverter and the slush density, were recorded at 0.2 second intervals on magnetic tape by a data acquisition system. The pressures were measured with transducers mounted external to the generator. A magnetic pickup located at the splines in the pump shaft sensed the pump speed. Liquid or slush volume was determined at fixed intervals by the 10 carbon resistor liquid level sensors described in section 3. A nuclear radiation attenuation densitometer described in section 2 was used to determine the solid fraction of the hydrogen slush in the generator.

1.4 Test Procedures

1.4.1 Performance Tests

Following the production of slush or triple-point liquid, the generator was pressurized to approximately one atmosphere with helium gas. The transfer line valve was opened to give the desired flow rate and the pump started. The receiver was vented to the atmosphere through a one psi check valve, and the helium gas was admitted to the generator to maintain the pressure at one atmosphere. During the electric motor runs the turbine was purged with helium gas to maintain the pumped fluid below the turbine blades. In general, the drop in liquid level spanned several liquid level sensors, corresponding to a volume of approximately 225 liters (60 gal.). Only a portion of each test was considered for the performance calculations, the first portion of the test being dominated by transient conditions.

1.4.2 Cavitation Tests

The cavitation tests were performed in a manner similar to the performance tests with the following exceptions. Instead of pressurizing the generator and receiver to one atmosphere, they were pressurized to about 0.2 atmospheres and the receiver ullage was connected to the generator ullage. Immediately after the pump was started, evacuation of the ullage began. The test was terminated either when severe cavitation occurred or when a low liquid level sensor was reached. Because of the flow resistance of the 1-inch line through which the receiver ullage was pumped, the receiver pressure lagged that of the generator. This effect, coupled with the transients mentioned previously, sometimes resulted in non-steady operating conditions before establishing cavitation.

1.5 Test Results

Table 1 summarizes the pump tests. The solid fraction of the slush hydrogen varied from 0.19 to 0.55. The average solid fraction was 0.33. Although in most cases the slush was freshly made, two runs were made with slush aged to 6 hours. No difficulty was experienced in pumping any of the mixtures regardless of the age or concentration.

1.5.1 Pump Performance Characteristics

The experimentally determined characteristic curves, figure 6, are presented in the form of developed head in feet of fluid pumped versus pump capacity in gpm. Because of variations in the pump speed, both within tests and between tests, the pump affinity laws were used to obtain speed. Analysis of the data indicates that at the 95 percent confidence level there is no statistical difference between the developed head in feet of fluid pumped for liquid or slush hydrogen.

In the time available, the pump efficiency was not determined in an absolute sense because of the complexity involved in measuring the 6-lead, 3-phase, square wave, 400 Hz., a-c power supplied to the motor by the inverter. However, the d-c power to the inverter was measured, and no significant difference (over that due to the density difference)

Table 1

Pump Test Summary

Accumulated Test Times	
Electric Drive	
Liquid	17.5 minutes
Slush	14.5 minutes
Turbine Drive	
Liquid	27.7 minutes
Slush	19.3 minutes
Total Accumulated Times	79.0 minutes
Number of Tests	
Electric Drive	
Liquid	30
Slush	28
Turbine Drive	
Liquid	47
Slush	36
Total Number of Tests	141

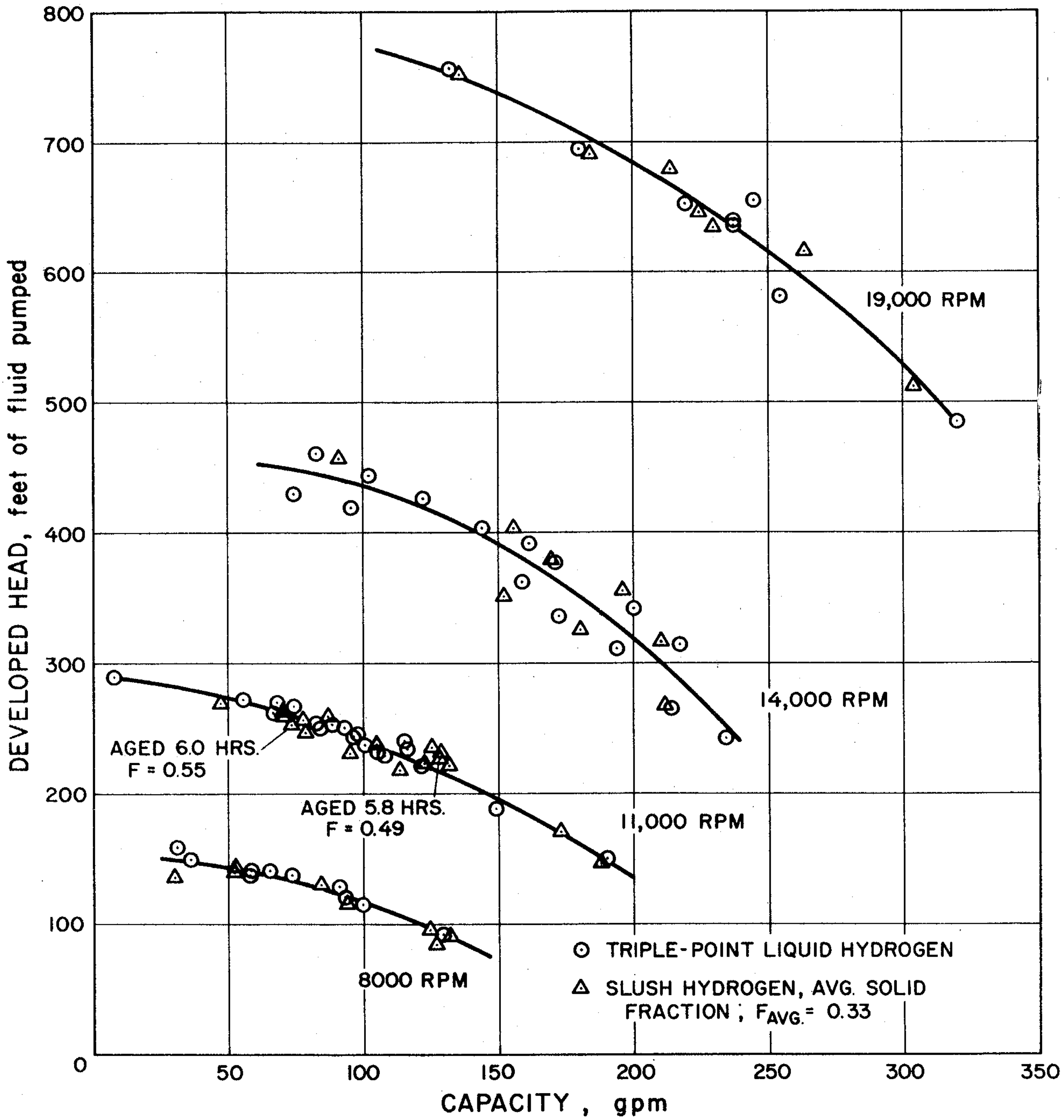


Figure 6. Pump Performance Data

between triple-point liquid and slush existed. Stepanoff (1963) states the following relationship between the head, H, and the pump efficiency, e, for slurry flow:

$$\frac{H_m}{H_\ell} = \frac{e_m}{e_\ell},$$

where the subscript m refers to the mixture and the subscript ℓ, to the liquid. It is thus consistent with the performance data that there should be no change in the efficiency when pumping hydrogen slush.

1.5.2 Cavitation Characteristics

Since in most applications it will be desirable to pressurize hydrogen slush with helium gas, the cavitation tests were performed with the mixture saturated with helium. The results of these runs are summarized in figure 7 which presents the cavitation constant, $\sigma = NPSH/H$, for a 15 percent reduction in head versus the specific speed,

$$n_s = \frac{N\sqrt{Q}}{H^{3/4}}.$$

Figures 8 and 9 summarize the smoothed experimental curves of NPSH versus developed head, H, at 11,000 and 14,000 rpm respectively. The cavitation constant and NPSH requirements are the same for liquid and slush.

1.5.3 Pump Wear

Of the total running time (79 minutes), slush hydrogen was pumped for a total of 33.8 minutes. The pump (which was still in operating condition at the conclusion of the tests) was disassembled; all pertinent components including the inducer, impeller, labyrinth and dynamic face seals, and bearings were inspected. No sign of wear or damage due to pumping slush hydrogen was observed.

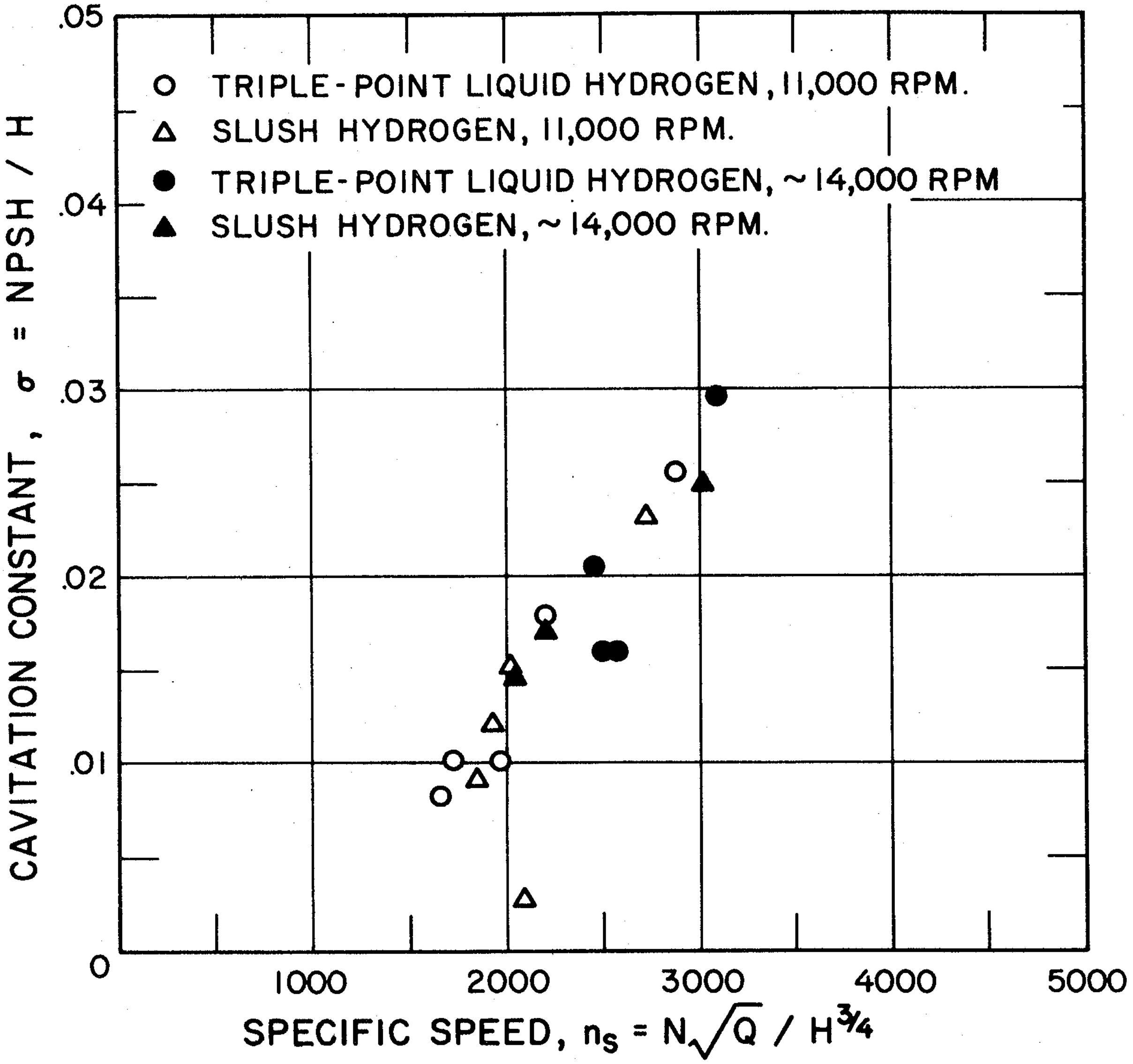


Figure 7. Pump Cavitation Constant For A 15 Percent Reduction In Developed Head

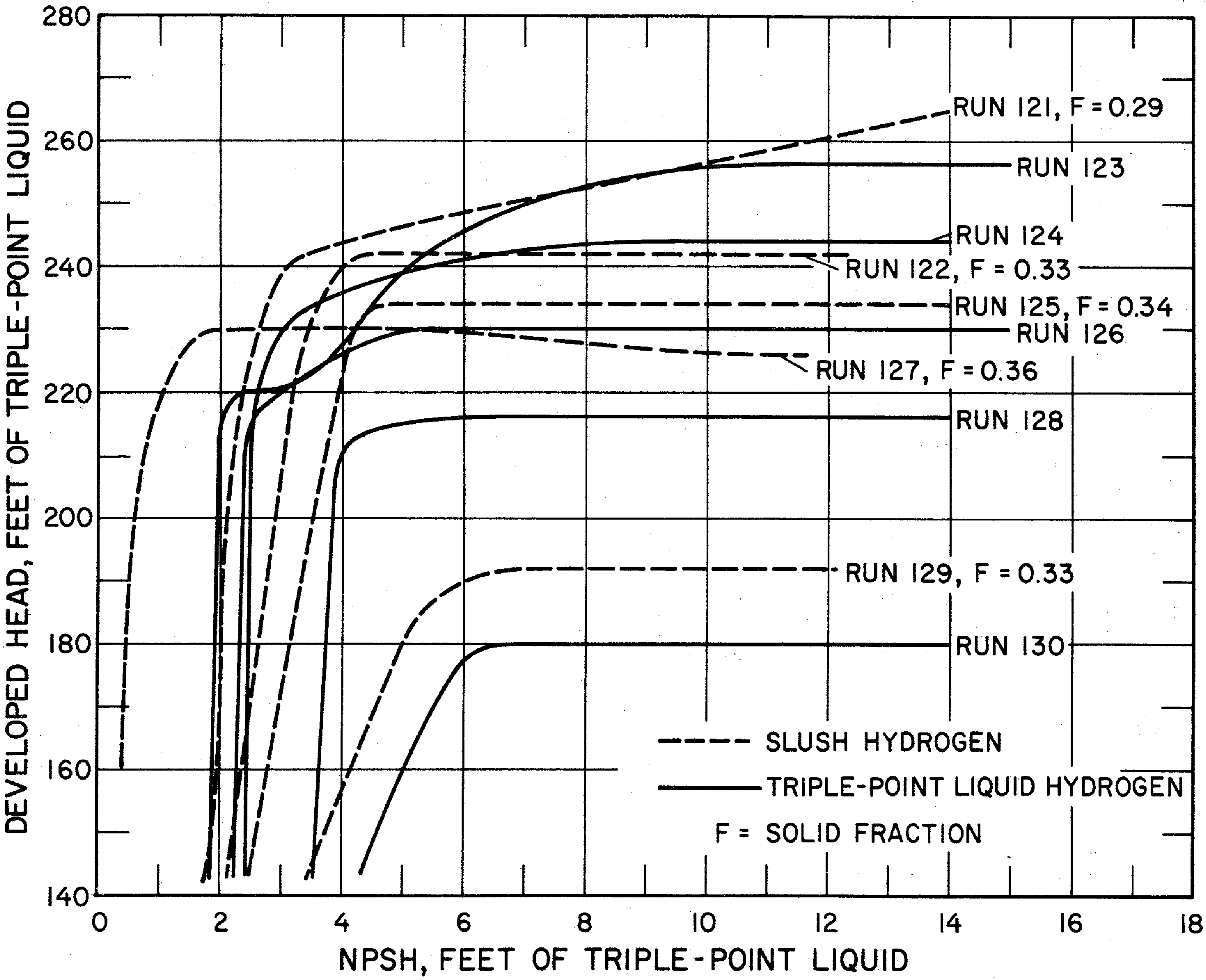


Figure 8. Pump Cavitation Tests - 11, 000 RPM

B- 72373

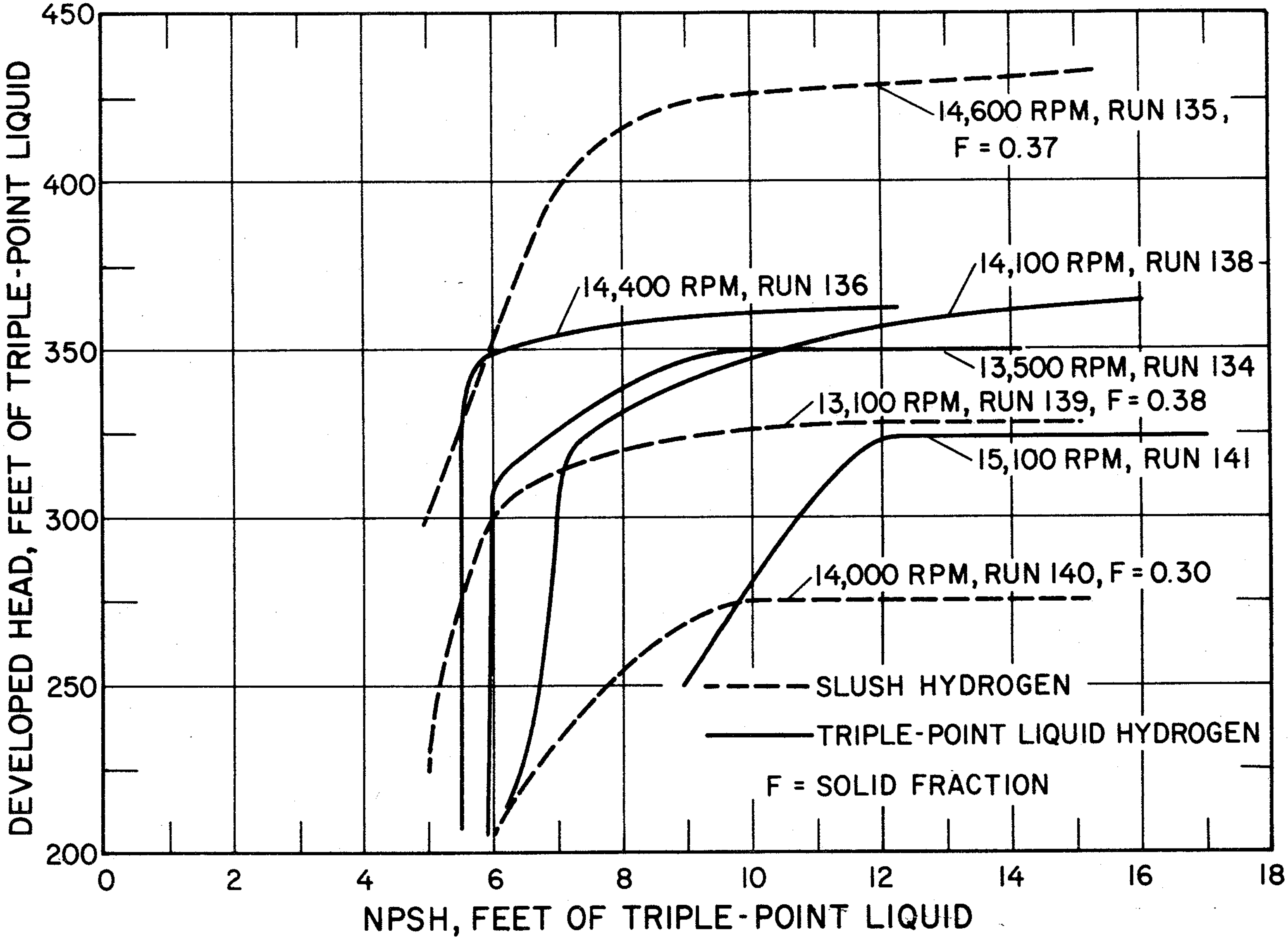


Figure 9. Pump Cavitation Tests - Approximately 14,000 RPM

B- 72372

1.6 Conclusions

As a result of the pump tests with liquid and slush hydrogen the following can be concluded:

1. Both aged and fresh slush hydrogen prepared by the freeze-thaw process can be pumped by a properly selected conventional liquid hydrogen pump.

2. The developed head expressed in feet of fluid pumped is the same for liquid and slush hydrogen.

3. The pump efficiency is unchanged when pumping slush hydrogen.

4. The NPSH requirements and the cavitation constant, σ, for helium pressurized hydrogen slush and triple-point liquid are the same.

5. Slush hydrogen causes no additional wear to pump components over that caused by running the pump in liquid. A total time of 33.8 minutes was accumulated with liquid-solid mixtures.

2. Slush Density Determination by Nuclear Radiation Attenuation

A number of methods for determining slush hydrogen density have been tested or proposed. For some systems employing special design features (i.e., the Hydrogen Slush Density Reference System, Weitzel et al., 1968), a weighing system can be used to determine slush density to within 0.2 percent. For most systems, however, the tare weight of the weighing container becomes so large that accurate density determination of slush hydrogen becomes impossible.

The mass-energy accounting method discussed by Daney and Mann [1967] is accurate to 0.5 percent in density for systems in which the heat leak and mass fraction of gas removed to produce the slush are well known. This method has been applied to a 450-liter generator for slush density determination, but errors in density as large as 1.5 percent have resulted. These errors result from poor accuracy in the measurement of the heat leak and of the gas pumped off during slush production.

A capacitance probe has been used for slush hydrogen density measurement with limited success [Alspach, et al., 1966]. Two disadvantages of this method are that the probe may not be filled with slush of the same density as that in the remainder of the system, and that it is a point sampling technique.

Measurement of the volume change on melting or the heat required to melt slush are additional density determination techniques. The heat measurement or calorimetric method can give accuracies to 0.3 percent in density [Daney and Mann, 1967], but both of these methods require destruction of the slush and hence are of only limited usefulness.

The nuclear radiation attenuation (NRA) principle is the most promising method of large-scale bulk density determination tested to date. A commercially available density measuring system has been calibrated in the 450 liter slush generator with encouraging results. This densitometer is being used to measure solid fraction in the flow and pump tests.

2.1 Description of the Apparatus

The densitometer consists of a 4 Curie, 0.663 MEV, Cesium 137 source; an ion chamber detector; an impedance matching unit and an instrument console. The ionization chamber current passes through a 10^{10} ohm hi-meg resistor giving the instrument a response time of one second. The signal from the densitometer is measured with a digital voltmeter and recorded on a strip recorder. Figures 10 and 11 show the position of the source and the detector with respect to the slush generator.

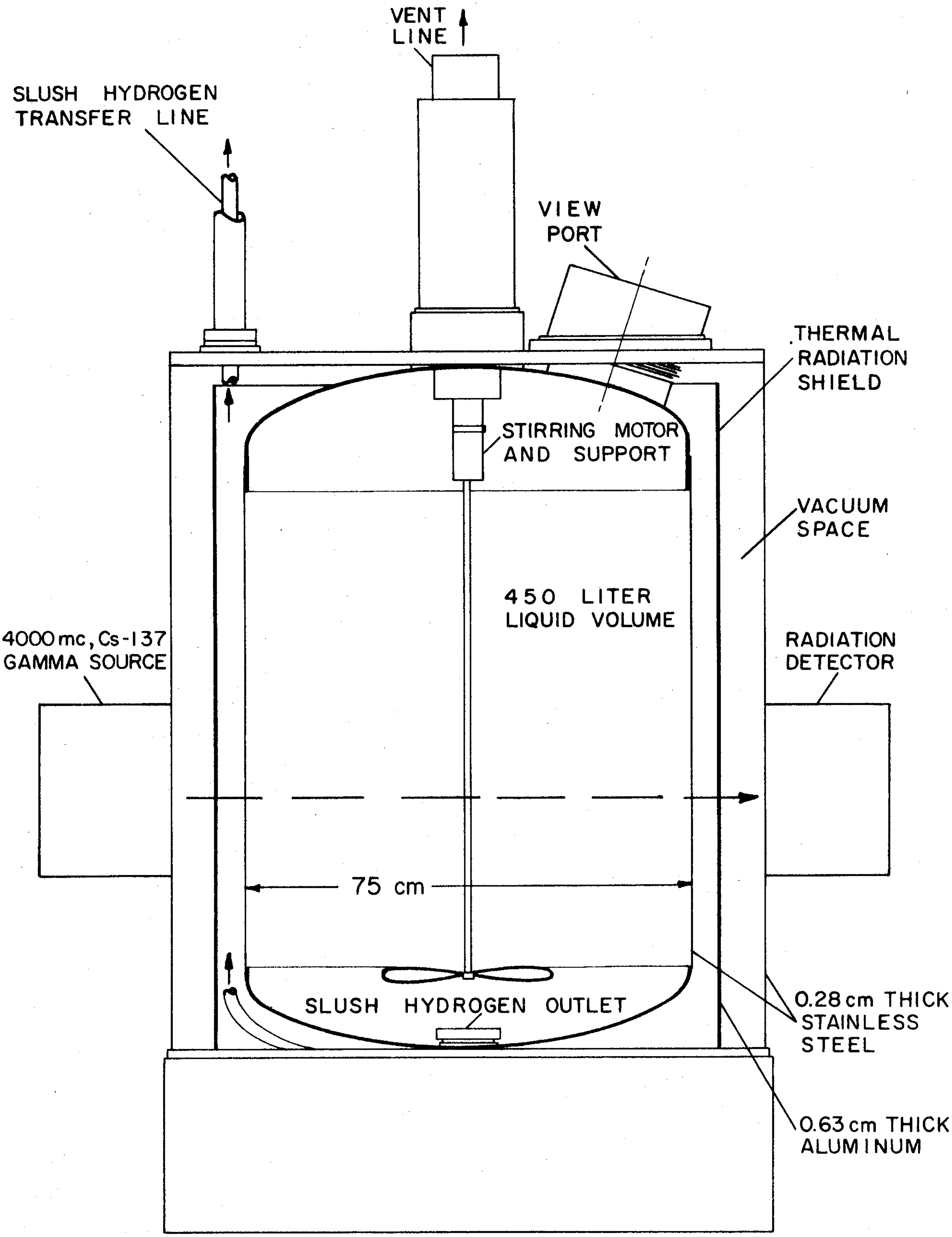


Figure 10. NRA Installation - Schematic

B-70038

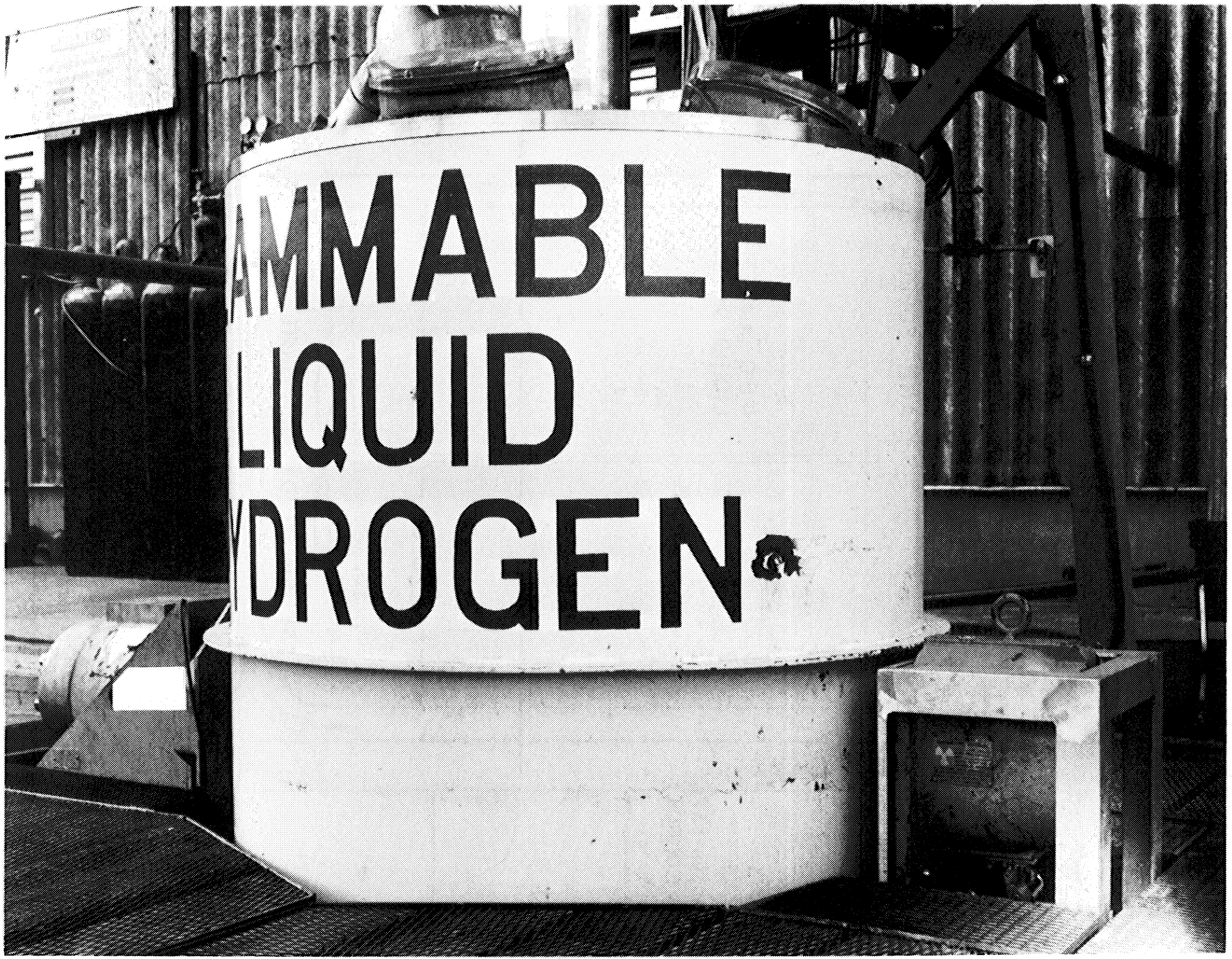


Figure 11. Densitometer Installation - Photo

B- 67289

The beam must penetrate 1.12 cm (0.44 inches) of stainless steel, 1.27 cm (0.5 inches) of aluminum and 76.2 cm (30 inches) of liquid or slush hydrogen. About one-half of the attenuation of the beam takes place in the hydrogen.

2.2 Experimental Results and Techniques

Figure 12 is the densitometer calibration curve. The output of the densitometer, which was adjusted to give zero emf with triple-point liquid, is plotted versus the independently determined density. The millivolt output of the densitometer is, in each case, the mean value of at least 10 digital voltmeter readings. The density was determined by three different methods, depending on the state of the hydrogen.

In the liquid region at pressures above the triple point, the density was determined by measurement of the vapor pressure over the liquid. The triple-point liquid condition was determined by making a small amount of solid in the slush generator, mixing the solid particles thoroughly by stirring. The densitometer reading was observed as the last of the solid particles settled past the region penetrated by the beam.

The density in the liquid-solid region was determined calorimetrically. After a densitometer reading was taken on stirred slush formed by the freeze-thaw production process [Mann, et al., 1966a], a heater in the bottom of the generator was energized until the triple-point liquid condition was achieved. The solid fraction, and hence the density, was then determined from the sum of the heater energy and heat leak. The densities used in the liquid region are those reported by Roder et al., [1965] and the triple-point solid density is given by Dwyer et al., [1965].

Figure 13 is a typical trace of the densitometer output as recorded on the strip chart. Two millivolts represents a solid fraction of 0.28. Similar traces were obtained with the generator empty, using lead to

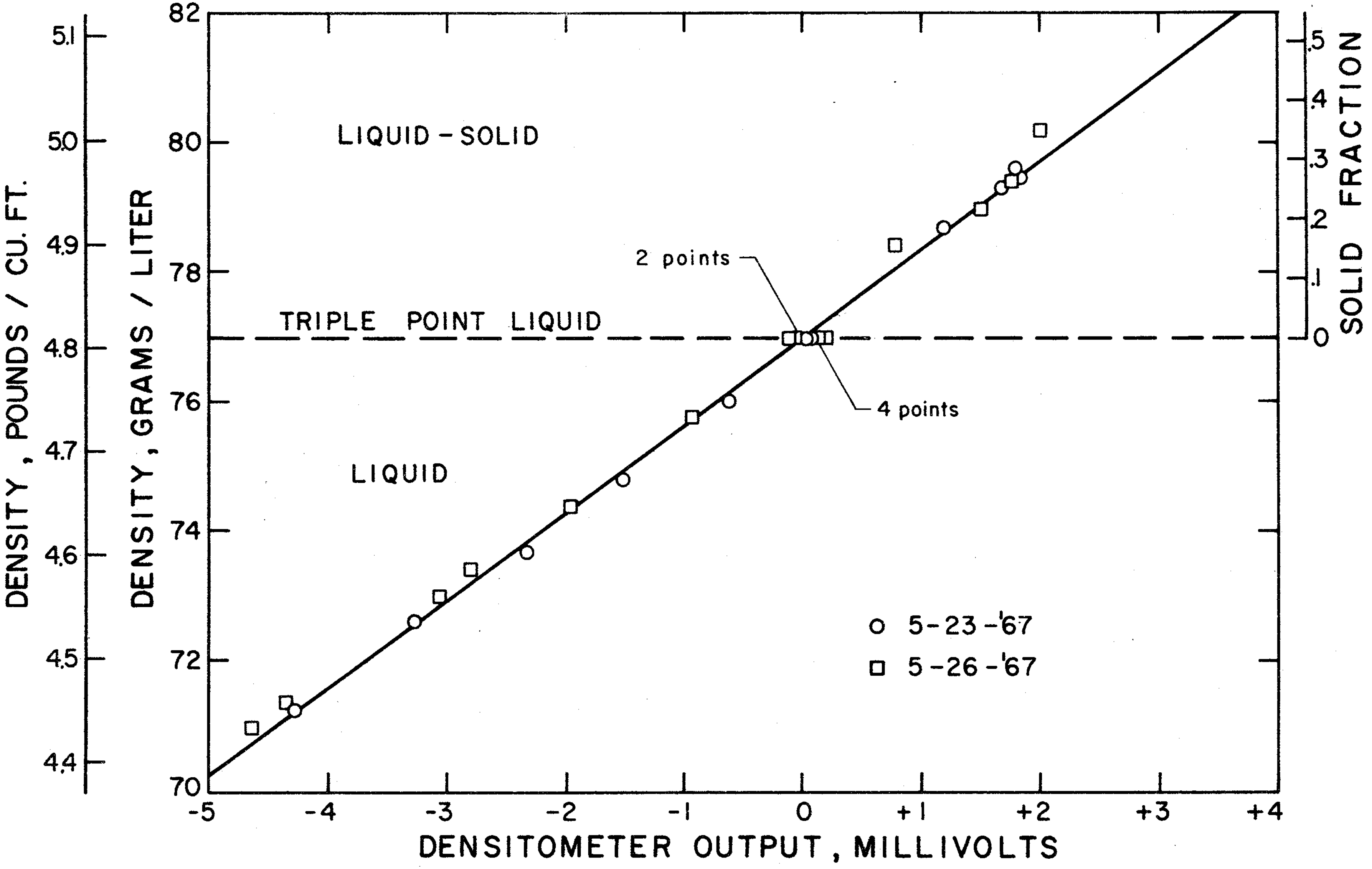


Figure 12. Densitometer Calibration Curve

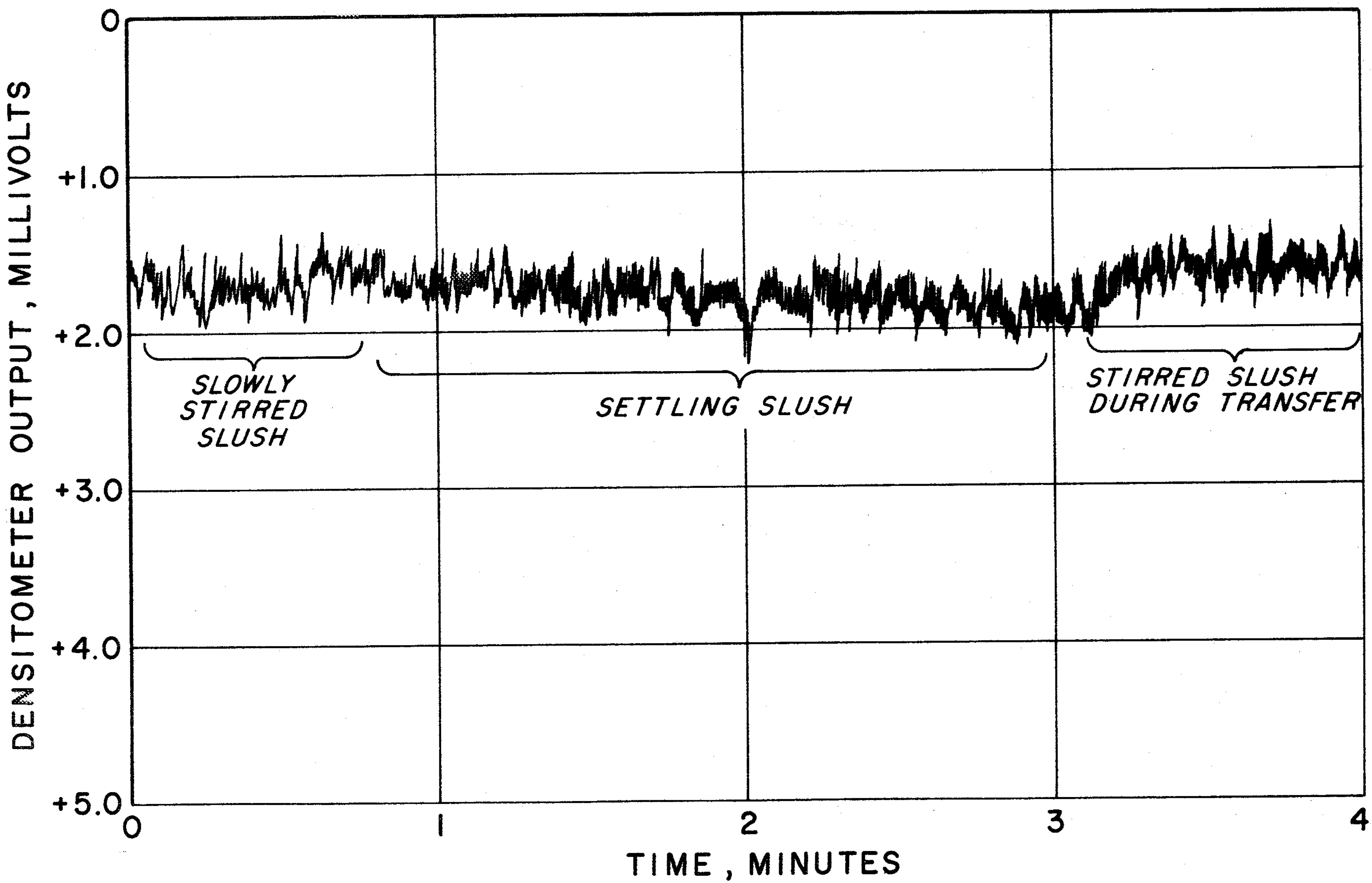


Figure 13. Densitometer Strip Chart

simulate the liquid. Thus it is concluded that the fluctuations in the signal are characteristic of the densitometer and are not due to density variations in the hydrogen.

2.3 Discussion of Results

The standard deviation of the measured densities about the least squares straight line in figure 12 is 0.17 grams per liter. The slope of the curve does not change in passing from the liquid to the liquid-solid region, thus it is possible to predict densities in the liquid-solid region by linear extrapolation of a liquid region calibration.

The uncertainty of the measured density values is estimated as three parts in 1000. Since this uncertainty is of the same order as the scatter in the results, further evaluation of the NRA densitometer awaits completion of the density reference system now being developed.

3. Slush Flow System

The slush flow system consists of two vacuum insulated dewars connected by a vacuum insulated transfer line. This system is described in detail by Mann et al., [1966b]. Figure 14 is a schematic of the system.

The procedure for performing flow experiments in the system is as follows:

1. Slush hydrogen is produced to the desired solid fraction in the generator.
2. The generator is pressurized with helium gas to the desired transfer differential pressure.
3. The receiver is vented to atmospheric pressure.
4. The ball valves are opened for the flow experiment.

The pressure differential is maintained by a regulator in the helium gas

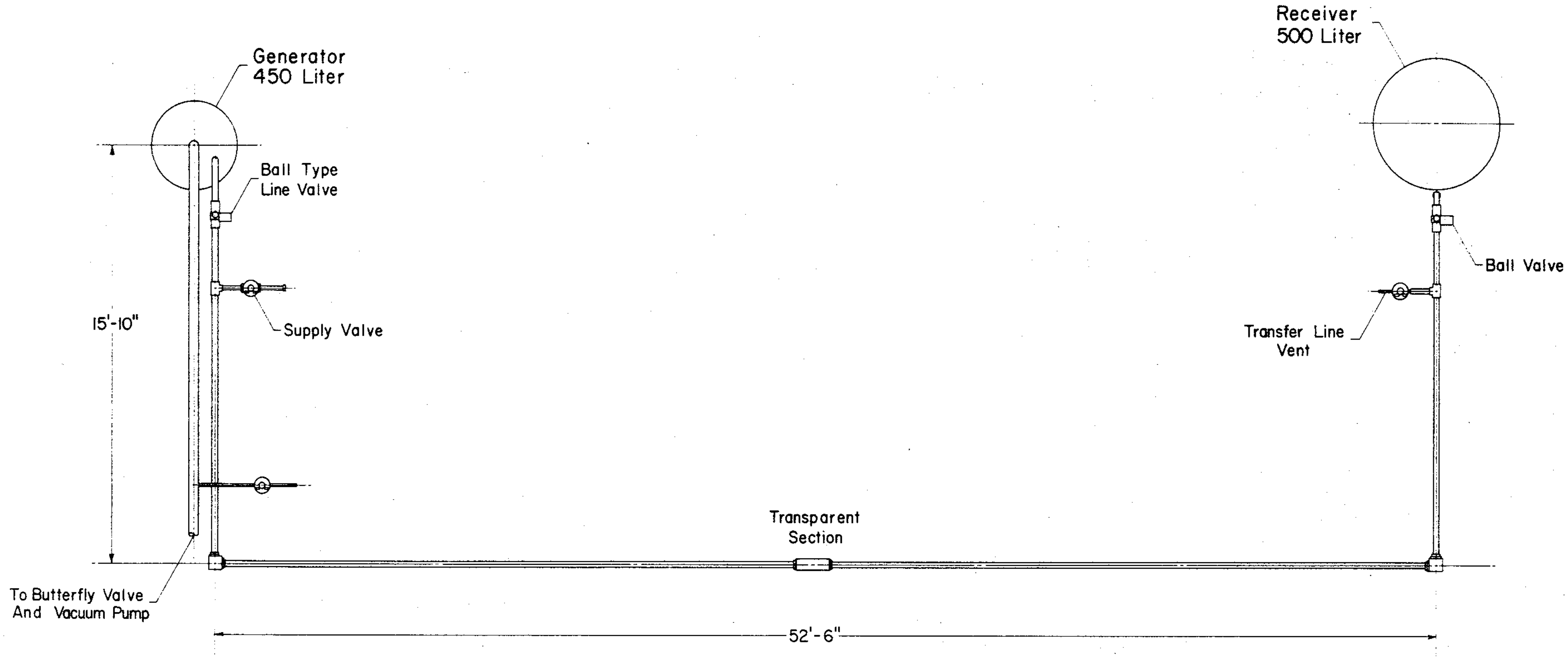


Figure 14. Flow Loop Schematic

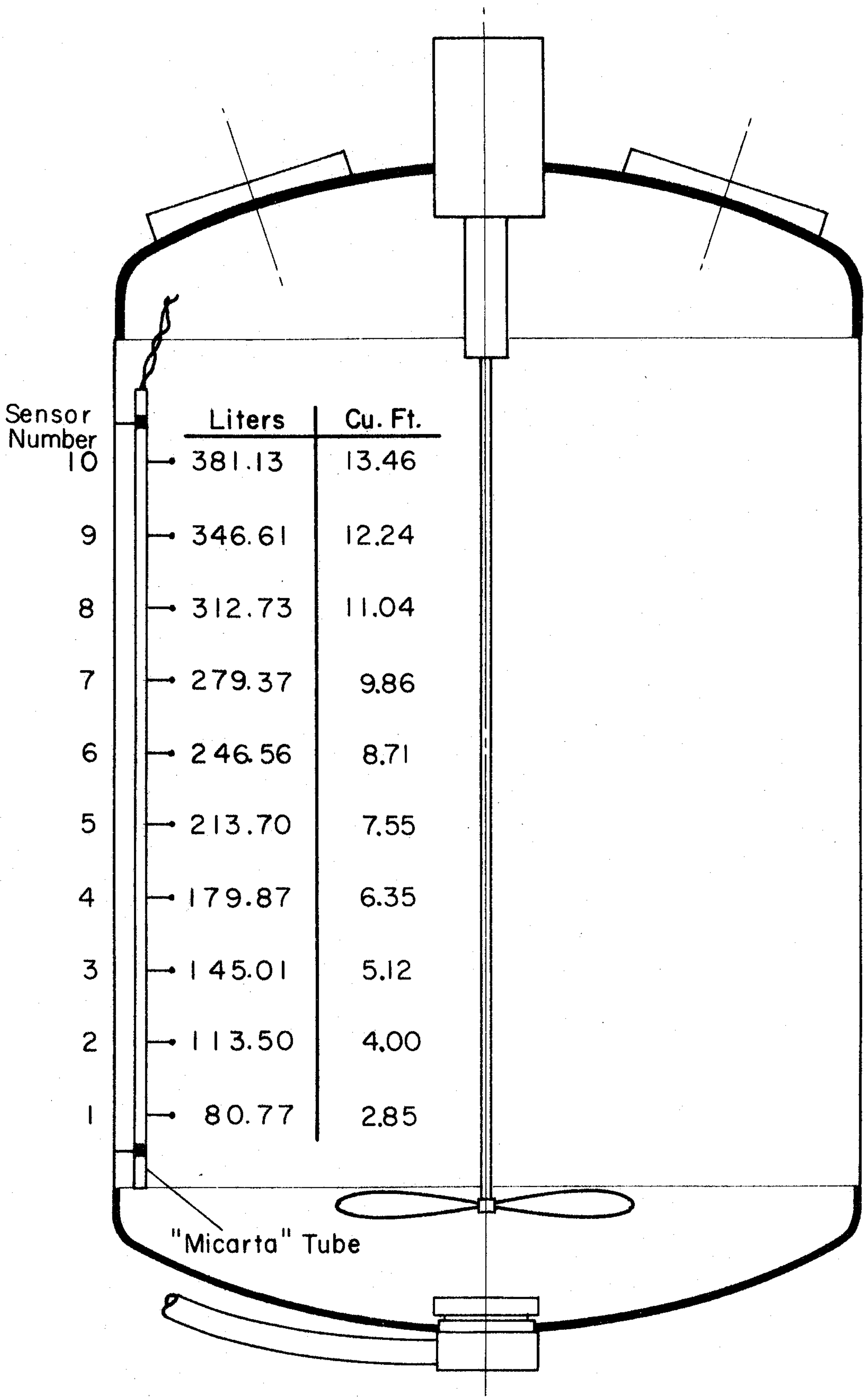
B-72247

supply to the generator and by venting the receiver through feather valves to the atmosphere.

The slush generator is used as the supply vessel in the flow system. Incorporated in the generator is a ten point liquid level sensor probe. These level sensors are used to determine the volume flow rate during slush transfer. The sensors are 200 ohm, 1/4-watt carbon resistors mounted on a 5/8-inch micarta tube. The calibration of the level sensors was performed using liquid nitrogen as the fluid. Electric heaters were used to provide energy to vaporize the liquid nitrogen and wet test meters were used to measure the volume of the boil-off gas. The volume below each resistor, as shown in figure 15, was determined from the volume of boil-off gas.

An error analysis of the volume flow system has been performed. The random error between two adjacent level sensors was two percent. The random error between two level sensors located 15 inches apart was approximately one percent. These errors assume liquid under quiescent conditions. Unless care is taken, surface disturbances and solid accumulation on sensors may cause errors as great as 5 percent. Experimental flow rates were determined using two level sensors 15 inches apart.

Pressure drop in the flow system is measured with variable reluctance type pressure transducers. Pressure transducer taps for the test section are located in the straight section of the transfer line. The pressure taps are located 30 diameters from the elbows to assure fully developed flow profiles. Figure 16 shows the transfer line pressure tap installation. A resonator tube was connected to each pressure tap to dampen the thermal oscillations present with slush transfers. The actual distance between the pressure taps is 48 feet, 4 inches. The pressure transducers are calibrated before and after each



Sensor Number	Liters	Cu. Ft.
10	381.13	13.46
9	346.61	12.24
8	312.73	11.04
7	279.37	9.86
6	246.56	8.71
5	213.70	7.55
4	179.87	6.35
3	145.01	5.12
2	113.50	4.00
1	80.77	2.85

Figure 15. Generator Volume Calibration

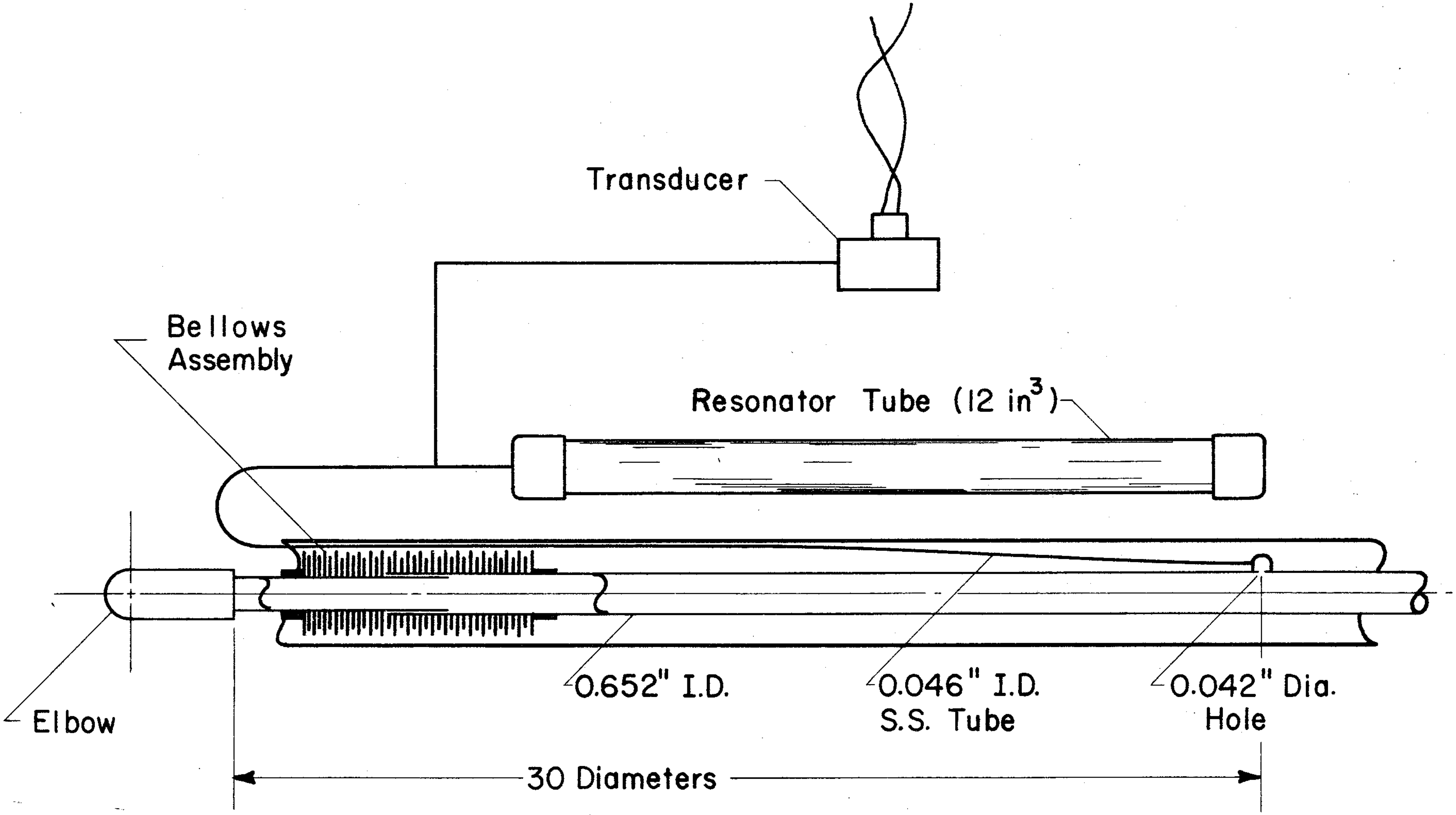


Figure 16. Transfer Line Pressure Tap

series of flow experiments. An error analysis of the pressure measurement system determined the error band to be plus or minus 0.05 psi with a 95 percent confidence level.

During the initial flow experiments, slush solid fraction was determined by the mass fraction of vapor pumped off as described by Daney and Mann[1967]. The parameters required to determine solid fraction with this technique are initial mass, mass of gas pumped off, and heat leak of the generating vessel. The initial mass was determined by filling the generator to a liquid level sensor with triple-point liquid. The pump used for slush generation was calibrated for flow rate versus inlet pressure for determining the mass of gas pumped off. The heat leak of the generator versus liquid level was determined by measuring boil-off gas. The data retrieval system interrogated the pump inlet pressure and liquid level at intervals of 0.2 second. A numerical integration was performed in a computer program to sum the mass fraction of gas pumped off and to calculate the solid fraction.

In the later flow experiments the NRA densitometer described in section 2 was used for determining solid fraction. During some of the tests discrepancies between the two measurements occurred. The discrepancies were attributed to the following reasons:

1. Heat leak during the slush manufacture being significantly different than that measured by the boil-off technique.

2. Errors in the measurement of the mass of gas pumped off. Careful examination and comparison of the two measurement methods indicate that the mass-energy method can result in errors in density as large as 1.7 percent, while the error in the solid fraction measurements determined with the densitometer are estimated to be within 0.5 percent.

The NRA densitometer has the advantage of measuring density just prior to and during actual slush transfer. Another added advantage of the densitometer is the ability to make slush to a predetermined solid fraction. Some degradation of solid fraction was noted during transfer due to the inflow of the relatively warm pressurant gas. To avoid slush degradation, the pressurant gas was precooled with liquid nitrogen in later tests.

A total of 80 data points of either triple-point liquid or slush have been taken using the flow system. The triple-point liquid data are shown in figure 17. The line on the figure was generated using the Darcy-Weisback [Binder, 1949] pressure drop versus flow rate equation for smooth pipes, using the Moody friction factor. The excellent agreement with this equation gave added confidence in the instrumentation. The slush data are shown in figure 18. The solid fraction is noted for each point. Solid fractions determined with the NRA densitometer are noted by solid symbols.

3.1 Discussion

The triple-point liquid data fit the prediction of pressure drop versus flow to within plus or minus two percent over the upper 80 percent of the flow range investigated. At the lower flow rates the percent deviation is greater but is within the range of the precision of the pressure transducers and the flow measurement. More triple-point liquid data are needed at higher and lower flow rates to include the range covered by slush data.

The slush data cover the flow velocity ranges from 3 feet per second to 30 feet per second. At flow velocities below 12 feet per second, the pressure drop with slush is greater than with triple-point liquid. The percent

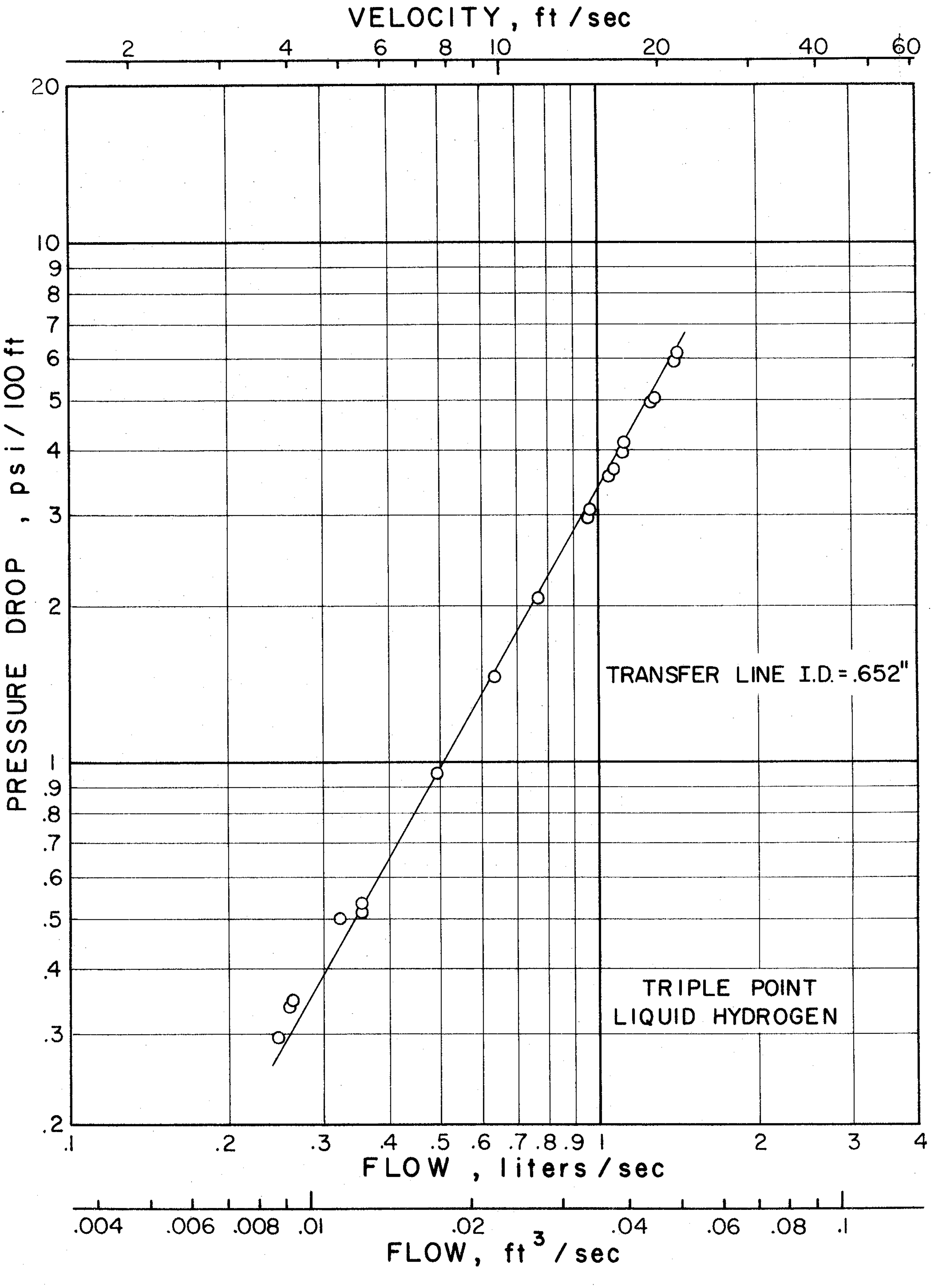


Figure 17. Triple-Point Liquid Flow Data

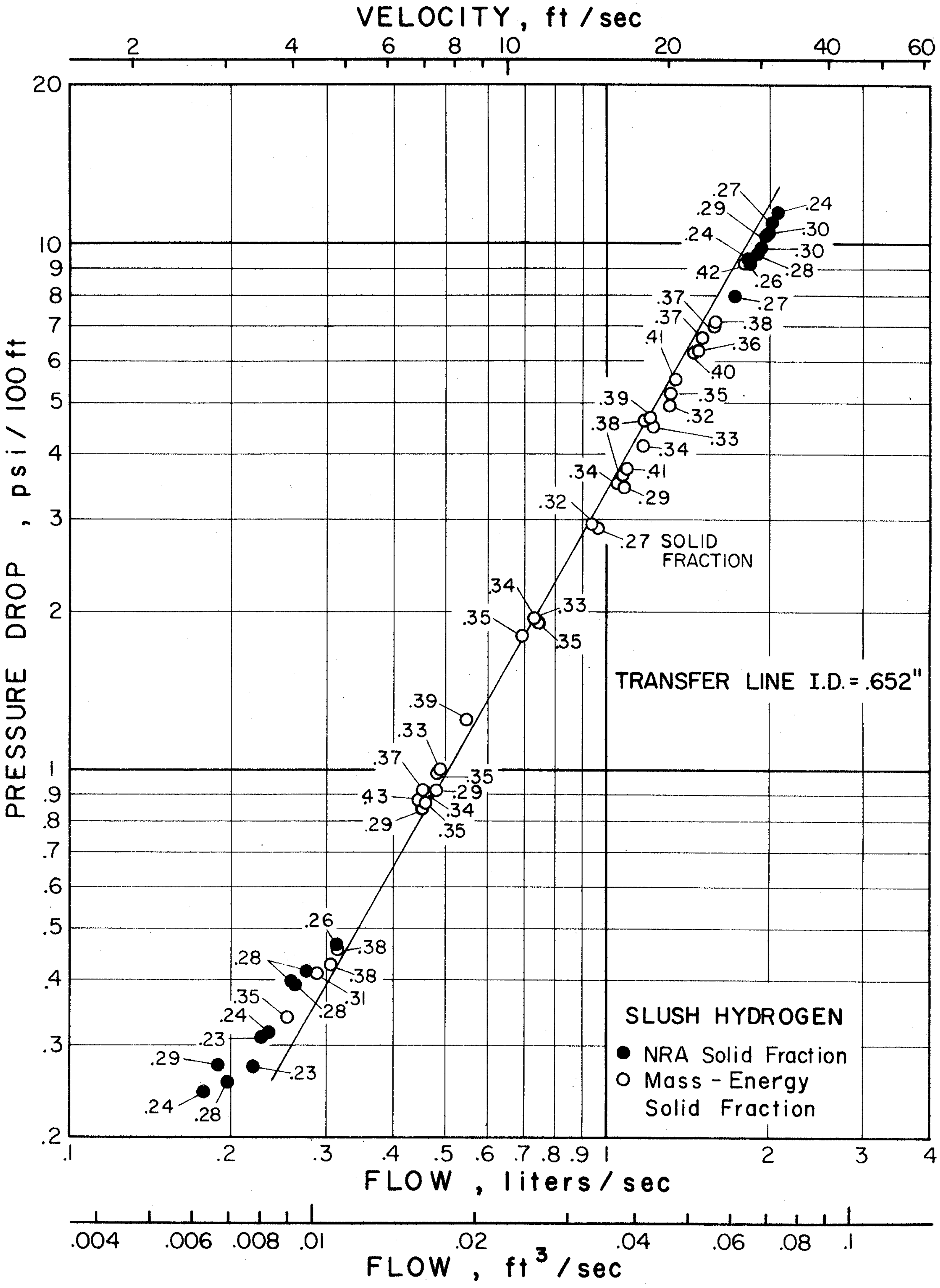


Figure 18. Slush Hydrogen Flow Data

deviation from triple-point liquid increases as the flow rate decreases. This would be expected from empirical slurry flow predictions of Durand and Condolios [1952]. The deviation is somewhat greater than that predicted for the particle size distribution and the terminal velocity measurements of slush particles. At flow velocities above 12 feet per second the slush data show a trend toward lower pressure drops than for triple-point liquid flowing at the same rates. At flow rates of 30 feet per second the pressure drop is 10 percent below that of triple-point liquid. The data for slush flow and triple-point liquid are consistent and repeatable in this region. Also, the deviation from triple-point liquid data is much greater than can be expected from the error predicted by the error analysis. Therefore, the conclusions are that the data are accurate and the trend is valid.

More data are needed at the higher flow rates with varying solid fraction to determine the dependence of pressure drop on solid fraction. Higher flow rates will be attained by the addition of a pressure controller on the receiver that will maintain pressure below atmospheric.

In the low flow region, improvements in the accuracy of pressure and flow measurement are needed to determine the dependence of pressure drop on solid fraction. Some improvement in pressure drop measurement will be made by increasing pressure transducer sensitivity. Improvement in the control of flow rate will be made by precooling the helium pressurant and by more sensitive pressure regulation of the generator pressure.

Future work will also include the addition of orifices and line restrictions to the transfer line in order to investigate pressure and flow characteristics due to their presence in slush hydrogen flow.

4. Thermal Oscillations

Thermally induced oscillations have been a recurring problem in the slush program. Oscillations may exist with normal liquid hydrogen but are more prevalent and present a more severe limitation with slush hydrogen. They have been observed in the 500-liter receiver dewar pressure tap line, in the slush particle study apparatus, in the warmup tube and outlet line of the generator, in the 3/4-inch transfer line vent tube, and in three pressure taps. In each of the above locations the oscillations have been either stopped or damped to a negligible amplitude by various schemes. Check valves were placed in the warmup tube of the generator and in the 3/4-inch transfer line vent. A sealed tube, instead of an open bottom tube, eliminated the thermal oscillations in the slush particle apparatus. A ballast volume connected to the pressure tap stopped the receiver oscillations; and the combination of a bare copper wire inside the pressure line and an external resonator tube damped the oscillations in the transfer line pressure taps.

There was speculation that thermal oscillations might be a problem when transferring slush through bayonet type couplings in a portable transfer line and also through valves other than ball valves. Previous work has not required the transfer of slush through these components. To further investigate slush behavior, an experiment was devised to flow slush through the components in question. In this experiment slush was transferred from the generator through two bayonet couplings and two globe valves into a supply dewar. The bayonet couplings were horizontal and facing opposite directions. One globe valve was a commercial, vacuum insulated valve specially designed for cryogenic use. The other was an extended stem, vacuum insulated globe valve on the supply dewar shown in figure 19. This valve was fabricated at NBS.

When slush was transferred through the experimental section, the plugs in both globe valves rattled. The transfer line valve did not become cold at the top of the bonnet but the dewar valve displayed excessive heat leak and frosted as shown in figure 19. There was no unusual frosting or cooling on either of the bayonet fittings.

The experimental section also had a small (1/8-inch) stainless steel pressure and relief tap extending through the vacuum space to the inner line. Thermal oscillations have never occurred in this tube while flowing normal boiling liquid through the transfer line, but when flowing slush the thermal oscillations were very prevalent. The line frosted as shown in figure 20 and the attached pressure gauge vibrated so fast the needle could not be seen. This tap is an ideal arrangement for thermal oscillations and would have to be corrected by one of the aforementioned methods if slush were normally transferred through the line.

After observing the globe valves, it is clear that the annular clearance to the bonnet gas space between the body and stem should be kept to a minimum if slush is to be transferred.

No definite conclusions can be made concerning the flow of slush through bayonet fittings. The annular clearance between the bayonet nose and the surrounding line was 0.017 inches for each of the couplings. It is believed that thermal oscillations would be present in a bayonet coupling if the clearance were much larger.

5. Slush Hydrogen Particle Study

The general objectives of this part of the program are to characterize more fully the structure and properties of hydrogen slush and to establish what variations in the slush particle structure and size may occur due to variations in slush production, treatment processes, and aging effects that occur during slush storage. A more complete knowledge and

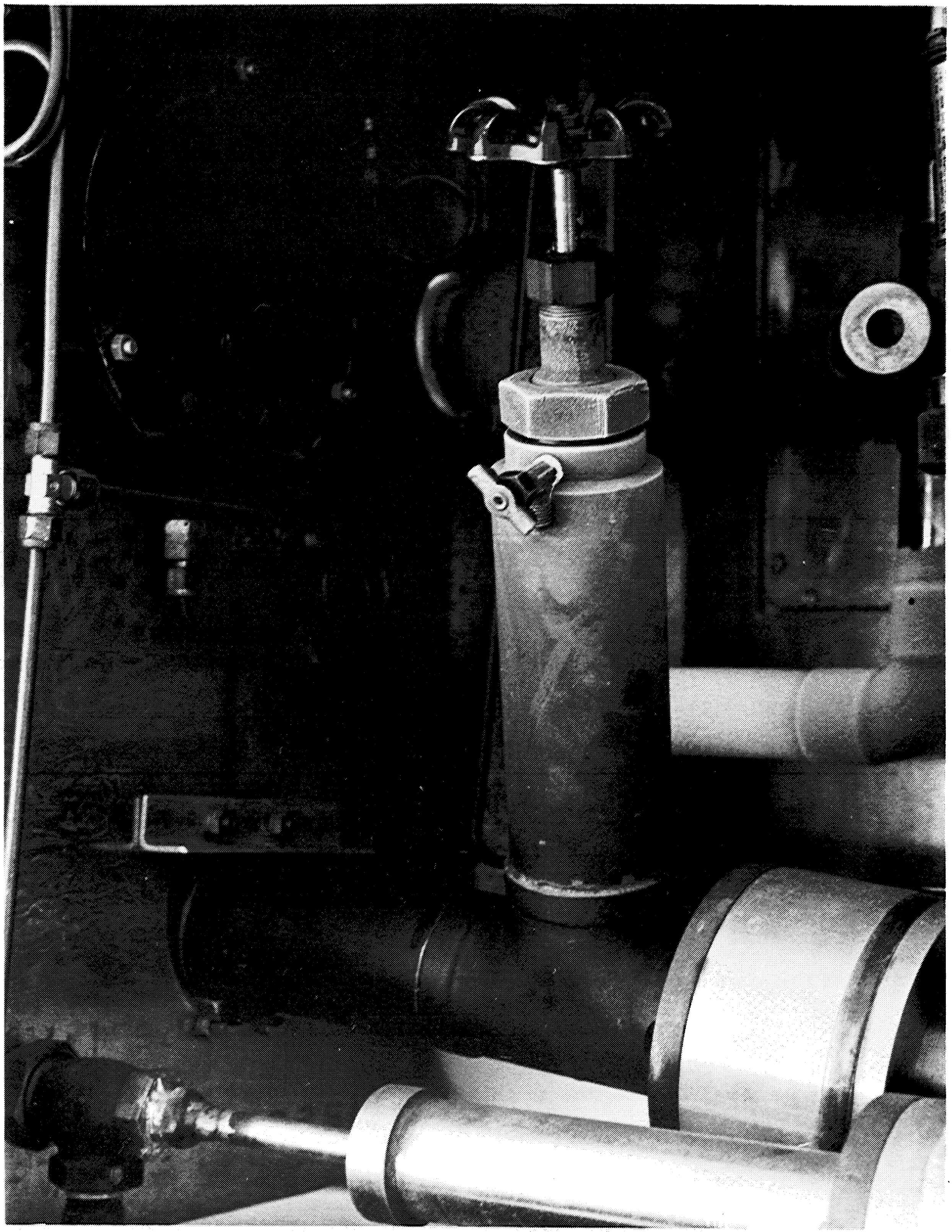


Figure 19. Supply Dewar Valve

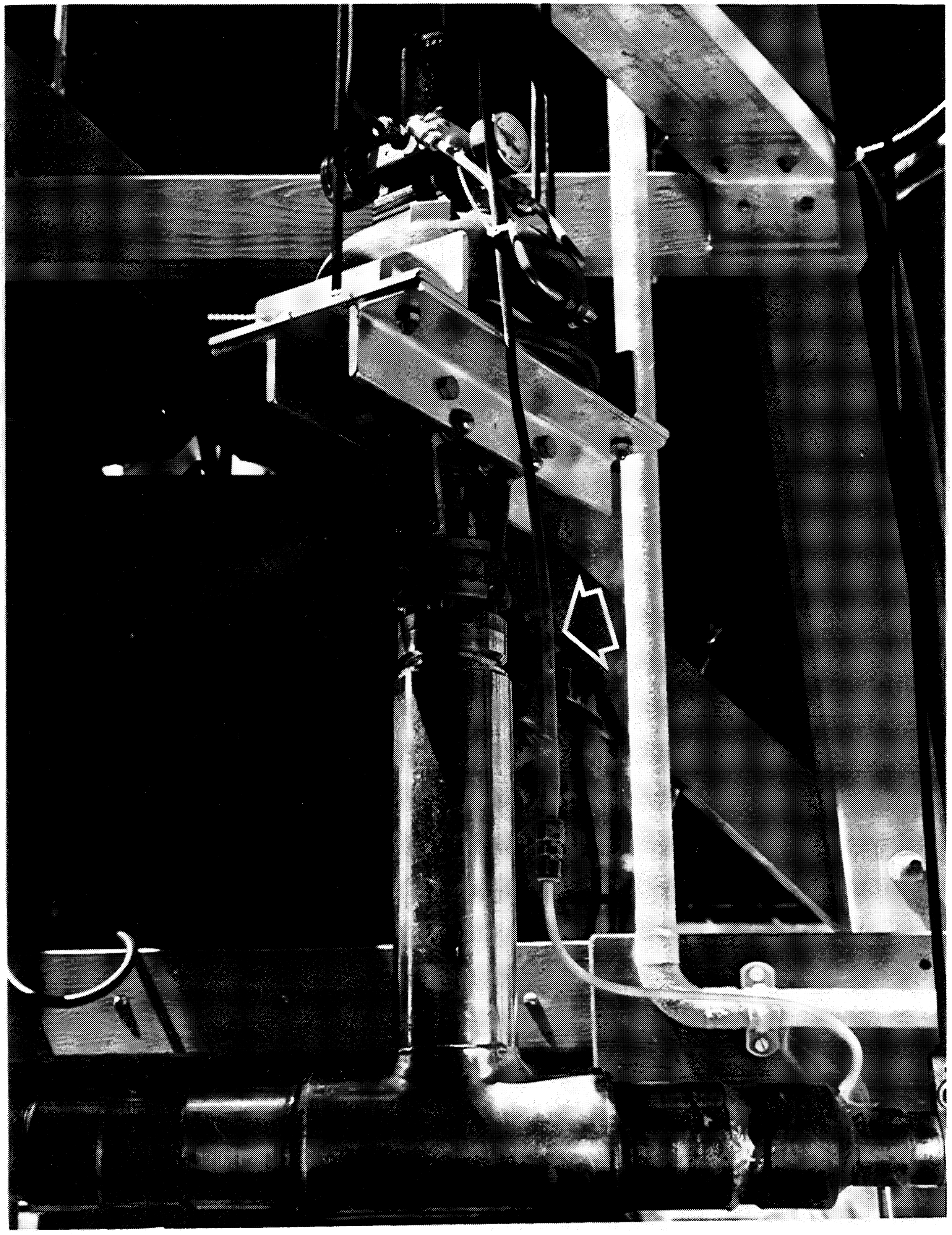


Figure 20. Pressure Tap Oscillations

understanding of these effects should help determine if production techniques may be varied to produce hydrogen slush with a higher density and more desirable transport and storage characteristics. The detailed objectives of the task are:

1. Measure the solid fraction of aged slush making use of the bulk quality measurement method discussed by Daney and Mann [1967].

2. Study the effects of slush aging and compaction on the mechanical properties of the bulk slush. The main point of this part of the program will be to observe if fusion of the individual particles into larger groups occurs during long storage times.

3. Measure the angle of repose of slush hydrogen (the maximum angle with the horizontal that the surface of the solid particles can sustain without sliding) as well as the coefficient of friction with simulated tank surfaces as a function of particle age.

4. Investigate the possibility of slush particle size reduction by means of high shear mixing and other techniques.

5. Measure the hardness and other appropriate mechanical properties of solid hydrogen particles.

5.1 Description of the Apparatus

In order to study aging effects in slush with some assurance that the effects observed are indeed due to aging rather than melting, very low heat leak storage is required. Observation of these effects requires visual access. A cross section of the apparatus which permits visual observation while maintaining the heat leak at a low value is shown in figure 21. Figure 22 is an overall view of the apparatus.

The innermost dewar (figure 23), which holds the hydrogen slush, is successively surrounded by a copper radiation shield with sliding doors (figure 24), a liquid hydrogen shielding bath pumped to just above the

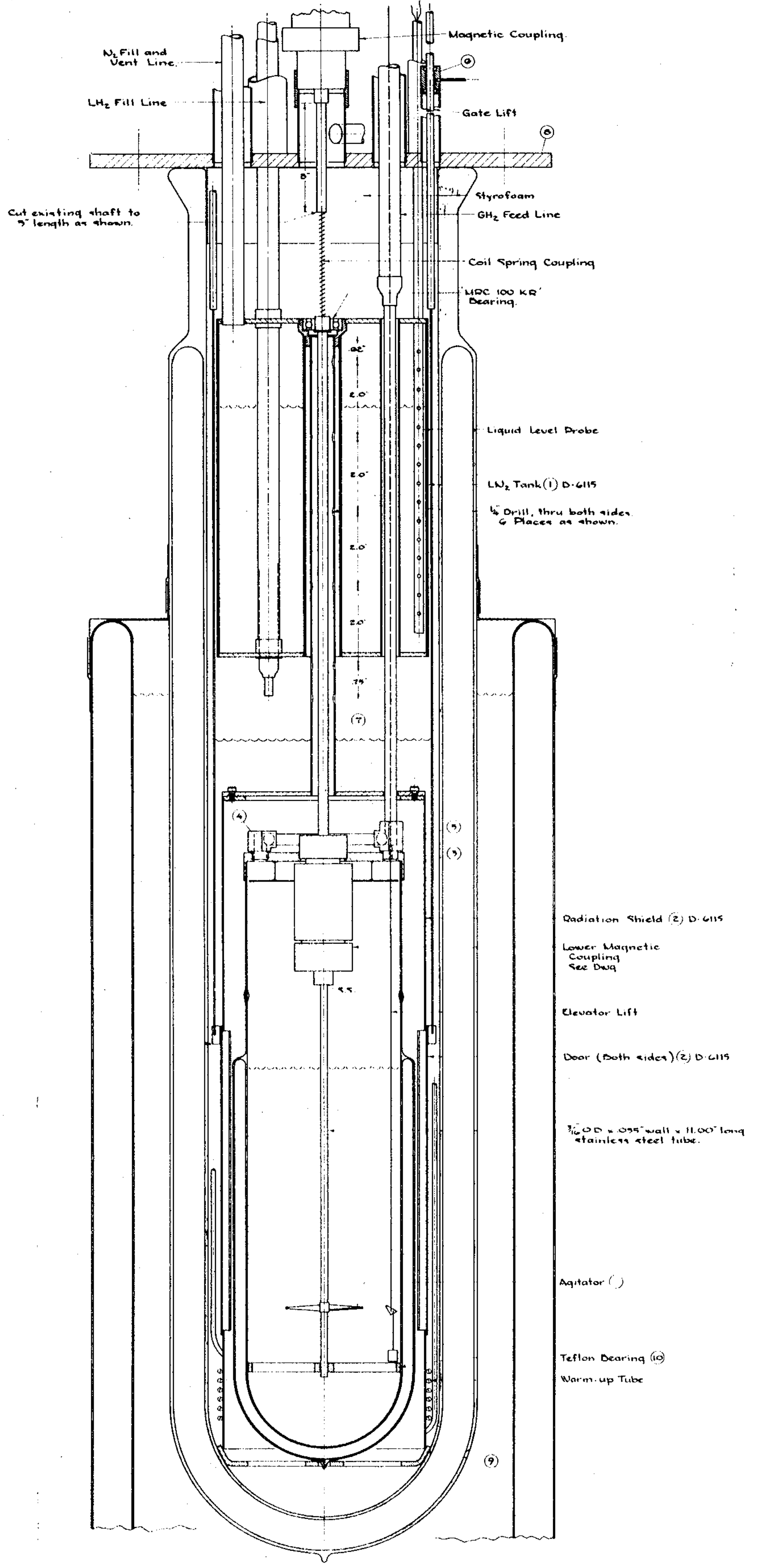


Figure 21. Slush Particle Apparatus - Cross Section

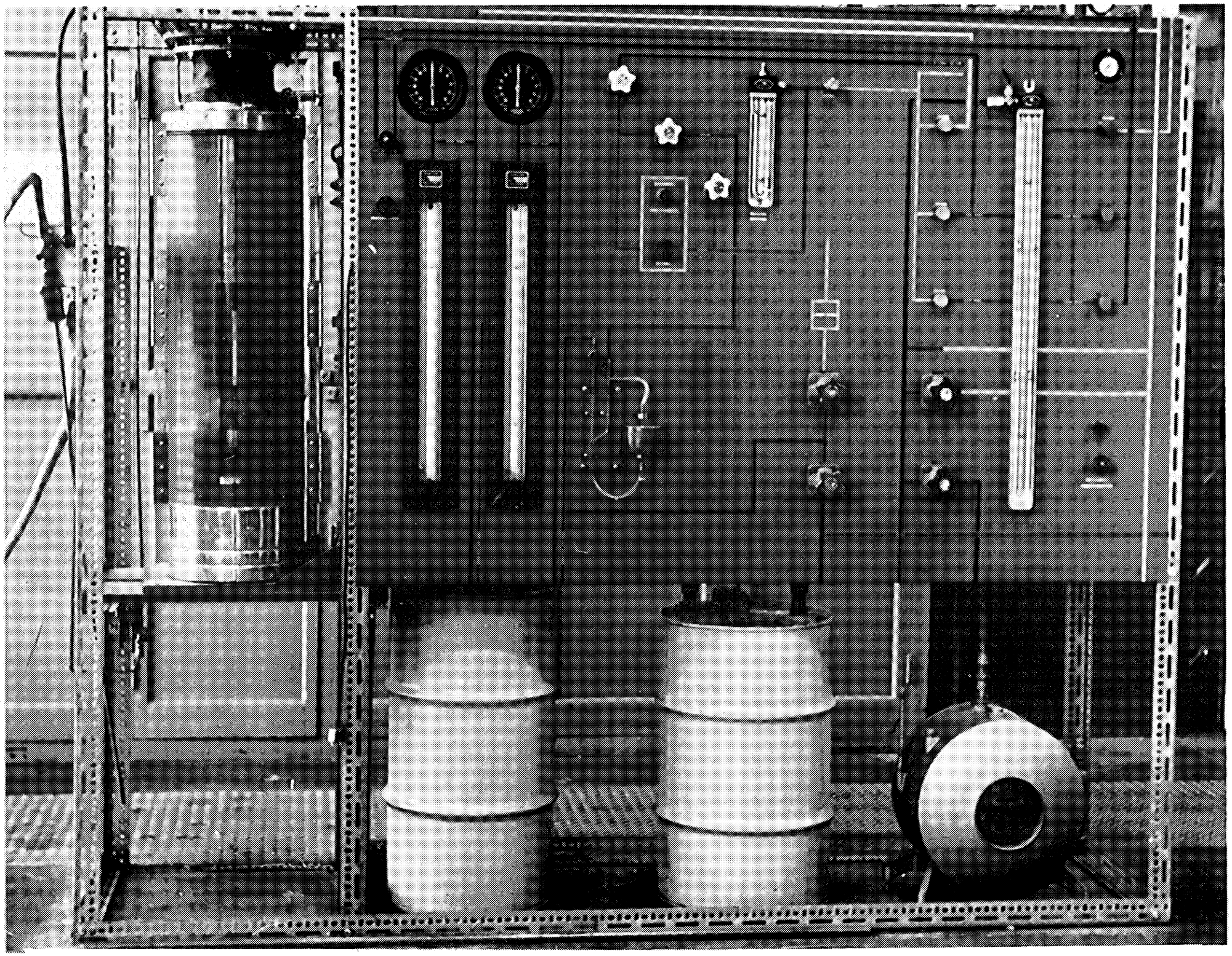


Figure 22. Slush Particle Apparatus - Photo

B- 73776

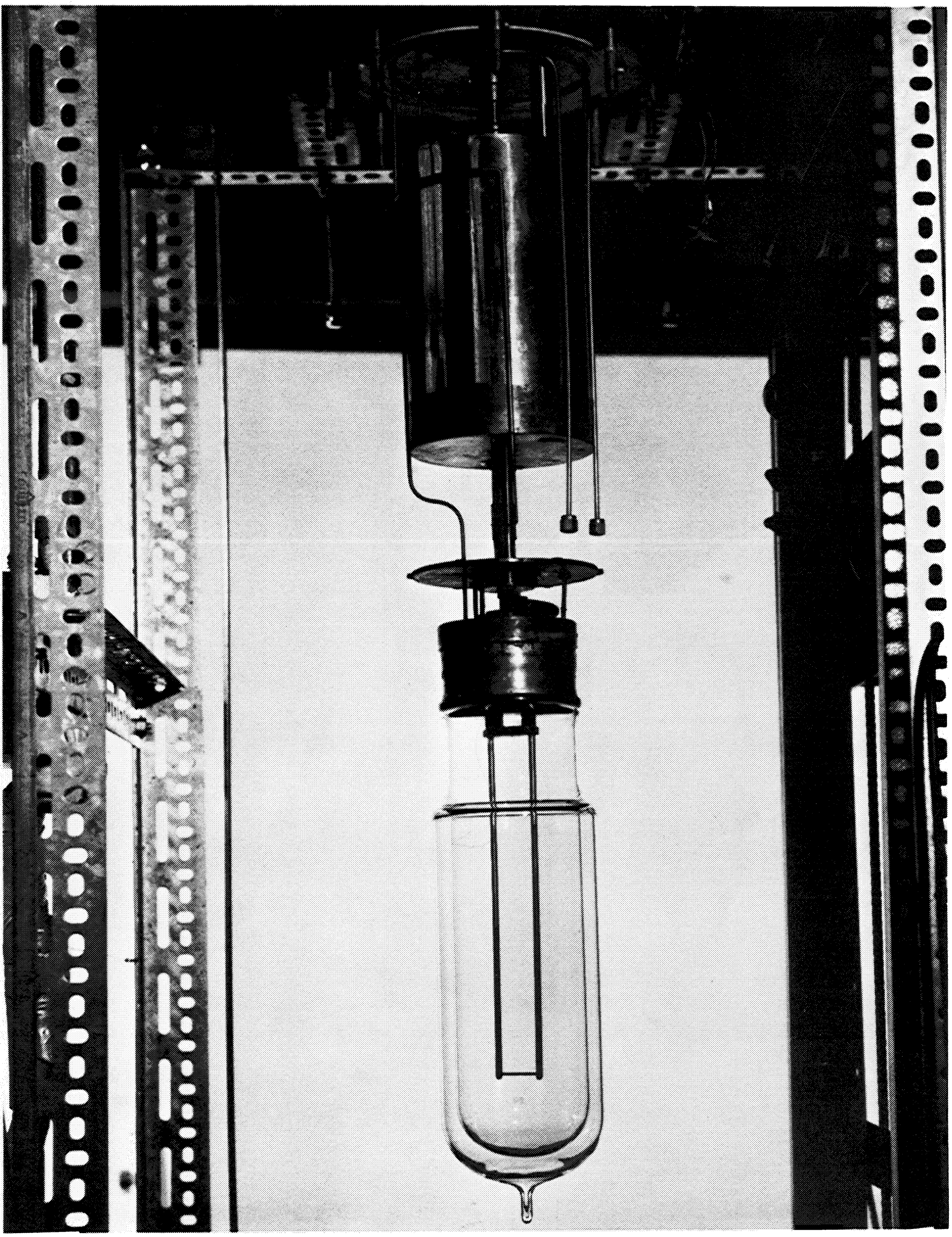


Figure 23. Experimental Dewar

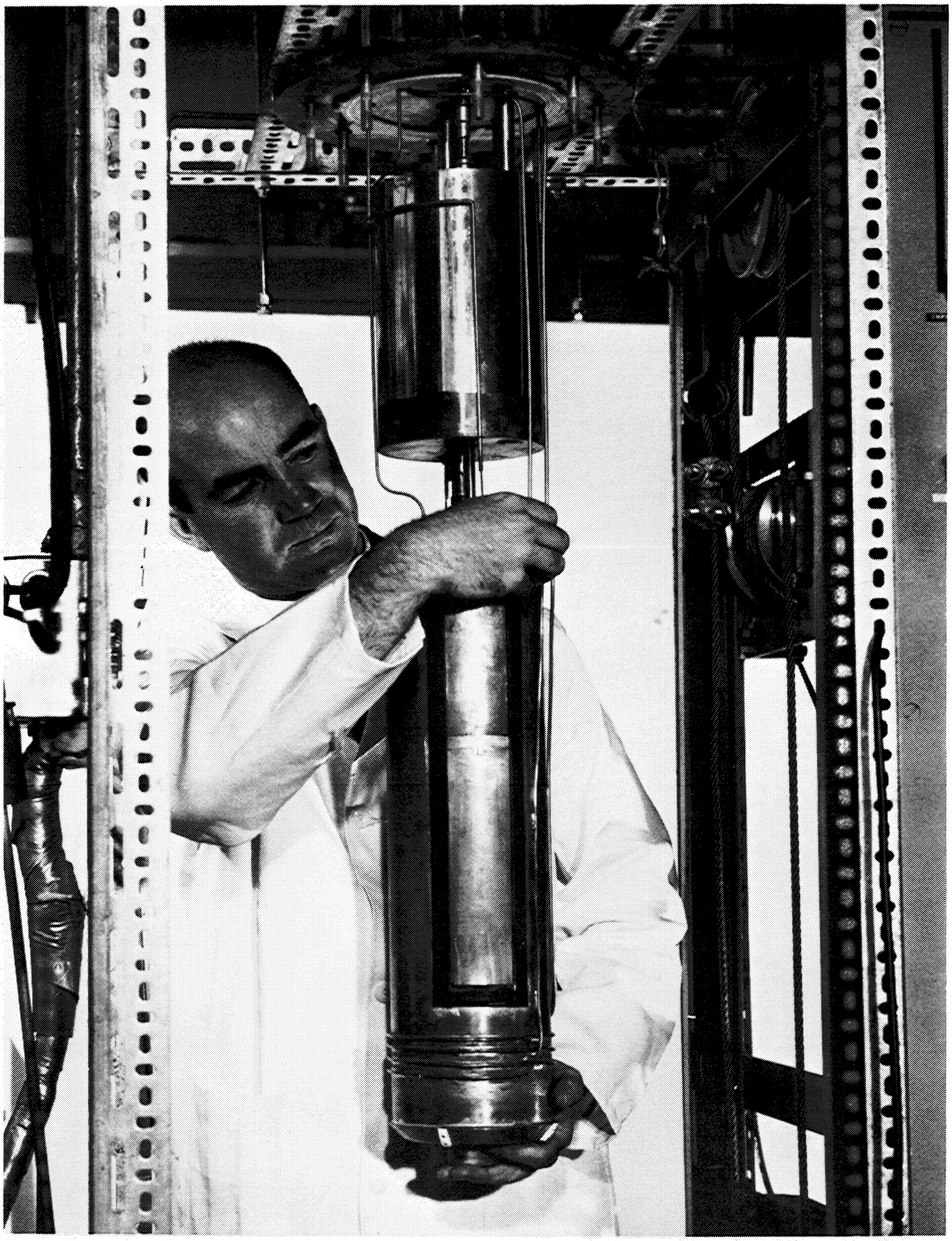


Figure 24. Thermal Radiation Shield

B- 69463

triple point, and a liquid nitrogen bath. This arrangement permits periodic visual or photographic observation of the slush particles by raising the shutters on the radiation shield, while holding the heat leak into the slush to a very low value. Stirring of the slush may be accomplished with the agitator shown. It is connected to a pneumatic motor via two magnetic couplings in series which provide hermetic shaft seals.

Care has been taken to reduce to a minimum the amount of operator attention required by the apparatus. A manostat regulates the pressure over the hydrogen bath to within a fraction of a torr. Automatic liquid level controllers maintain the desired levels in the liquid nitrogen cooled radiation shield and the liquid nitrogen shielding dewar. The operation of the camera and the radiation shield doors is controlled by a timer. Intervals of 10, 30, or 60 minutes between sequences may be selected. A second timer permits slush production according to a predetermined freeze-thaw cycle.

The apparatus is easily disassembled, permitting a variety of experiments to be performed by modification within the inner dewar.

5.2 Solid Fraction of Aged Hydrogen Slush

Experiments were carried out in the slush hydrogen particle study apparatus to measure the solid fraction of hydrogen slush as a function of its age.

The solid fraction of aged slush is defined as the solid fraction by mass of the material in the settled portion of the slush,

$$F_s = \frac{m_s}{m_\ell + m_s},$$

where m_ℓ is the mass of the interstitial liquid and m_s is the mass of the solid. Any clear liquid above the liquid-slush interface is not included in m_ℓ.

The solid fraction is determined by measuring the mass of the vapor pumped from the production dewar together with the heat leak, as described by Daney and Mann [1967]. The expression used to calculate the settled solid fraction is

$$F_s = \frac{7.61\, m_v - \dfrac{Q_{HL}}{L_f}}{m - m_v - m_{\ell c}} .$$

Here m_v is the mass of gas pumped off during the freeze-thaw production process measured by a wet test meter connected to the vacuum pump exhaust. The initial mass, m, is determined from a liquid level measurement, the dewar having been volume calibrated. The mass of the clear liquid above the settled slush $m_{\ell c}$ is determined from the difference between the measurements of the liquid level and the slush level. The remaining terms are Q_{HL}, the heat leak into the dewar, and L_f, the latent heat of fusion. The constant 7.61 is the best experimental value for the ratio of the refrigeration produced during the freeze-thaw process to the latent heat of fusion of parahydrogen.

5.3 Experimental Results

Five hydrogen tests have been made with the slush hydrogen particle study apparatus. Liquid-solid mixtures of hydrogen have been stored for periods of 24, 38, 51, 59, and 100 hours while measurements of the solid fraction of the aged slush and the heat leak were made. Figure 25 shows the experimental values of the solid fraction of aged slush for two of these tests as a function of the aging time. Solid fractions greater than 0.60 have been achieved.

When these aged quantities of slush were stirred, they settled back to approximately their undisturbed density in less than a minute. No

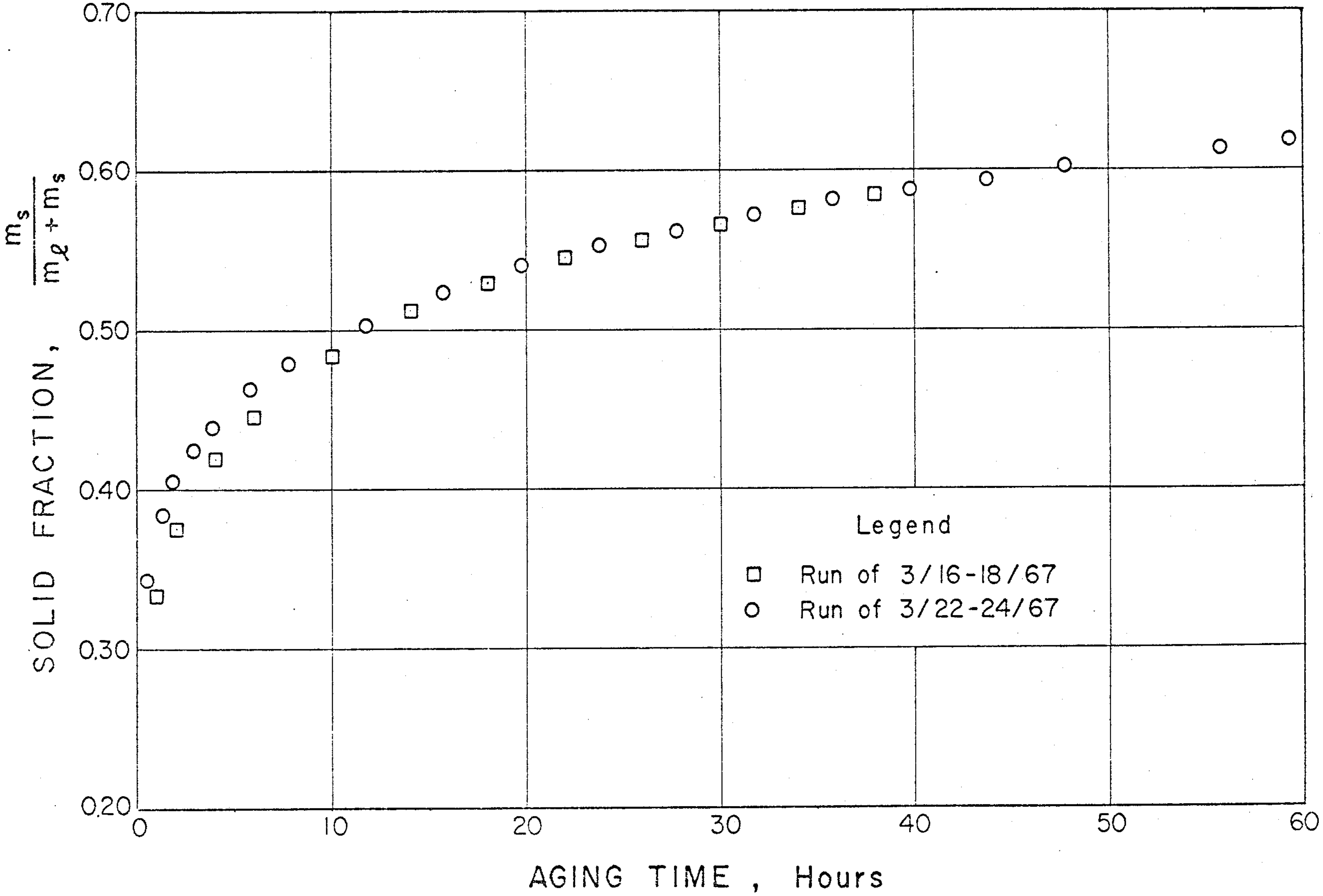


Figure 25. Solid Fraction of Aging Slush Hydrogen

tendency for the solid particles to fuse together has been observed. In fact, several large multigrain pieces of solid, formed during two of the runs, were observed to disintegrate into their individual grains while aging.

5.4 Heat Leak

The heat leak into the stored slush is determined from the volume change due to melting as observed from the liquid level change. The average heat leak during four of the tests was 1×10^{-4} watts with a standard deviation of 1×10^{-4} watts. The small change in the liquid level which occurs makes it difficult to determine the heat leak with much greater precision. However, this rate of heat leak amounts to a solid loss of only 0.3 percent per day. A liquid storage dewar with the same heat leak would lose 0.04 percent of its liquid per day. Thus the heat leak into the slush hydrogen is well below that which would be experienced in the field.

5.5 Conclusions

Some preliminary conclusions can be made as a result of the work completed so far. Solid mass fractions of at least 0.60 may be obtained by allowing the slush to age for two days or more. The aged slush will immediately settle back to a solid fraction within a few percent of the solid fraction observed before stirring. There is no tendency for individual solid particles to fuse together. Instead, there is a tendency for larger, multigrain structures to break up as they age.

Finally, it should be pointed out that aging is not a special treatment given to the mixture; it is a naturally occurring rearrangement of the particle structure that occurs inevitably.

6. References

Alspach, W. J., T. M. Flynn, and R. J. Richards (1966), Slush Hydrogen Instrumentation Study, NBS Report 8879.

Binder, R. C. (1949), Fluid Mechanics, Prentice Hall, (New York, N. Y.)

Daney, D. E. and D. B. Mann (1967), Quality Determination of Liquid-Solid Hydrogen Mixtures, Cryogenics, Vol. 7, p. 280, 1967.

Durand, R. and E. Condolios (1952), Proceedings of a Colloquium on Hydraulic Transport of Coal, London (November 5-6, 1952).

Dwyer, R. F., G. A. Cook, O. E. Berwaldt, and H. E. Nevins (1965), Molar Volume of Solid Parahydrogen Along the Melting Line. J. Chem. Phys. Vol. 43, p. 801.

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

Mann, D. B., C. F. Sindt, P. R. Ludtke, and D. B. Chelton (1966b), Slush Hydrogen Fluid Characteristics, Proceedings of the Conference on Long-Term Cryo-Propellant Storage in Space. At NASA, George C. Marshall Space Flight Center, Huntsville, Alabama (October 12-13, 1966).

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.

Stepanoff, A. J. (1963), Pumping Solid-Liquid Mixtures, Paper No. 63-WA-102. ASME Winter Annual Meeting, Philadelphia, Pa., November 17-22, 1963.

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