NBS TECHNICAL NOTE

377

Slush Hydrogen Fluid Characterization and Instrumentation

C.F. SINDT, P.R. LUDTKE, AND D.E. DANEY

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards

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ABSTRACT

Areas of the slush hydrogen fluid characterization program covered are production, transfer, and storage of liquid-solid mixtures.

An experiment has been performed in which a second component was added to liquid hydrogen to determine the effect on the size of the solid particles produced by the freeze-thaw method. Also, an experiment performed with 4.4% oxygen in nitrogen resulted in particles an order of magnitude smaller in size than those normally observed in freeze-thaw production of pure nitrogen slush.

The friction angle of settled solids of slush hydrogen on several metals was determined for surface finishes ranging from 2 to 110 microinches rms. The angle at which solids slide from the surface was found to be a function of the surface finish.

Data were taken in the flow loop over a range of velocities from 1.5 to 40 ft/sec (0.46 to 12.2 m/s). The friction losses determined from the flow data are compared to losses predicted for Newtonian fluids. Losses with slush of solid fractions from 0.1 to 0.4 were found to be lower than with triple-point liquid hydrogen at the higher Reynolds numbers.

Liquid-solid mixtures of hydrogen with solid fraction of 0.5 were transferred through three types of flow restrictions. The restrictions were a globe valve, an orifice, and a venturi. Losses through the restrictions were essentially the same with the high solid fraction slush as with triple-point liquid.

A brief review of the pumping characteristics of slush hydrogen is presented. A comparison of developed head, efficiency, cavitation constant, and net positive suction head is discussed.

Gelling of slush hydrogen with pyrogenic silica was accomplished with solid fractions from 0 to 0.2. The amount of gelling agent required to gel slush was found to be proportional to the liquid volume of the slush.

Key Words: Friction angle, cryogenic, flow restrictions, gel, liquid-solid mixtures, particle size, plug flow, slurry flow, slush hydrogen.

SLUSH HYDROGEN FLUID CHARACTERIZATION AND INSTRUMENTATION*

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

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. The 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 the work reported here, equipment and techniques were developed to produce, observe, and transfer liquid-solid mixtures of hydrogen through a flow loop with generator and receiver dewar capacities of approximately 14 cubic feet (0.4 cubic meters) each. The flow loop was designed for visual and photographic access through view ports. Pumping slush hydrogen had been demonstrated using a centrifugal pump, slush density had been measured with a nuclear radiation attenuation densitometer, and slush of a solid fraction greater than 0.6 had been obtained.

2. Fine Particle Production with Two-Component Mixtures

Production of solid hydrogen particles, finer than the 0.5 to 7 mm size produced by the current freeze-thaw technique [Mann et al., 1966], would offer two potential advantages. First, finer particles would enhance flow and mixing characteristics because they are more easily

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

kept in suspension. Second, and more important, the solid fraction of a mixture of fine and coarse particles would be considerably greater than for a single size because the finer particles could fill the voids between the larger particles.

The settling velocity of particles in still liquid is a good indication of the flow and mixing characteristics of the particles. The lower the settling velocity, the better the flow and mixing characteristics should be for a given liquid-solid mixture. In the laminar regime the settling velocity, V_s, is given by

$$V_s \approx r^2$$

where r is the particle radius. In turbulent settling experienced with most freeze-thaw particles, the radius dependence is not as strong, but it is still significant. The expression is

$$V_s \approx r^{1/2}.$$

If a significant size reduction were obtained it might be expected that laminar settling would occur. Since the flow properties of freeze-thaw hydrogen slush are excellent, little improvement in this area would be expected. The improvement in the mixing characteristics would be more marked and important because of the lower mixing velocities required to keep the finer particles in suspension.

Far more significant, however, would be the increased densities that might be achieved by mixing two or more groups of different sized particles. Using the freeze-thaw process, solid fractions in the range of 0.40 to 0.45 can be achieved with freshly prepared slush, and solid fractions in the range of 0.60 to 0.70 can be achieved by aging the slush up to 100 hours. By comparison, a volume solid fraction of 0.60 is

typical of randomly packed uniform spheres [Herdan, 1960], and 0.80 for log normally distributed spheres [Wise, 1952]. The corresponding mass solid fractions for hydrogen spheres would be 0.63 and 0.82, respectively. By using a mixture of several spheres with the properly selected size ratios, solid fractions as high as 0.95 are possible [Herdan, 1960]. Thus, a very significant increase in solid fraction or density could be obtained by producing very fine solid hydrogen particles to mix with the particles currently produced by the freeze-thaw process.

In the course of the NBS slush work, freeze-thaw slush production was carried out over a wide range of operating conditions. The pumping rate was varied by two orders of magnitude and a variety of different stirring arrangements were tried. In spite of this wide variation in conditions, no significant change in the particle size was observed. Thus, little hope is held for particle size reduction due to these variations in the freeze-thaw process.

During experiments with slush natural gas, it was observed that very fine particles were produced (at least an order of magnitude smaller than freeze-thaw nitrogen or hydrogen particles). Because natural gas contains a number of components in addition to methane, this experiment suggested that addition of a second soluble component to hydrogen might accomplish similar results with slush hydrogen.

Two additional experiments were conducted to test this hypothesis. In the first experiment, deuterium was chosen as the second component with concentrations ranging from 0.2 to 0.8 percent D_2 in H_2. No significant reduction in the particle size during freeze-thaw slush production was observed. A second experiment, to determine if the concept held for any mixtures other than methane mixtures, was made using a mixture of 4.4 percent oxygen in nitrogen. In this case, particles an

order of magnitude smaller than those normally observed during freeze-thaw slush nitrogen production were observed.

The detailed mechanism of this phenomenon is not completely understood. In general, however, it appears that the presence of a second soluble component in some cases drastically increases the number of nucleation sites in the liquid. The difficulty in producing this phenomenon in hydrogen results because only a limited number of substances (viz, helium, neon, and deuterium) are soluble to a significant extent in liquid hydrogen. In addition to the deuterium experiment mentioned above, low concentrations of helium have been tried since it is a standard procedure to use helium as the pressurizing gas in the slush work. Neon and orthohydrogen appear to be the next most likely candidates.

Because of the large potential benefits which would accrue from the production of fine solid hydrogen particles, a continuing effort in this area will be made.

3. Friction Angle of Slush Hydrogen on Metal Surfaces

During transfer of unstirred slush hydrogen from a tank, residual solids could remain in the tank unless the slope of the bottom exceeds the friction angle (the angle from the horizontal at which sliding of the particles on a metal surface begins). Tests have indicated that solids will be retained in vessels with flat bottoms. As an aid to the design of slush hydrogen tank bottoms, measurement of the friction angle of slush hydrogen on metal surfaces was made.

The slush hydrogen particle study apparatus, described by Daney, et al. [1967], was modified for these measurements. Figure 1 views the inner dewar of the apparatus with the shielding dewars removed. The flat plate is pivoted on its trunnions by a thin wire attached to one

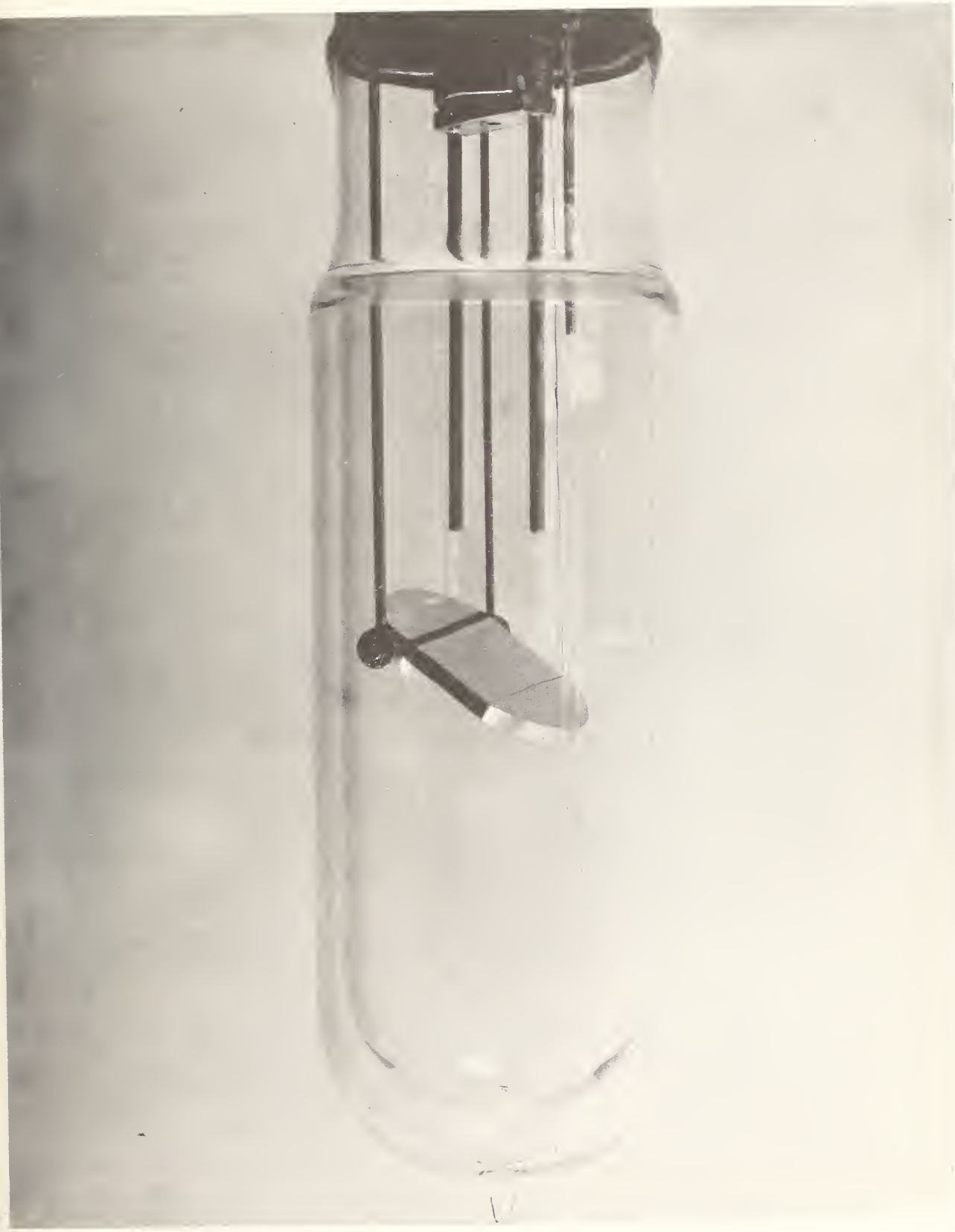

Figure 1. Friction Angle Test Apparatus

end. Slush can be deposited onto the plate by stirring with the two stirring blades in the top of the dewar.

The sample plates were 3-inch (7.6 cm) diameter circles modified by cutting 1/4-inch (0.64 cm) parallel segments from two sides. The surface finishes and plate materials are summarized below.

Surface Finish (micro-inches rms)	Surface Finish Method	Plate Material
2	polished	aluminum
10	cold rolled	stainless steel
57	vertical end mill	aluminum
80	# 36 grit abrasive	stainless steel
110	coarse grit abrasive	aluminum

A profilometer was used for the surface finish measurements.

The experimental procedure begins with freeze-thaw slush production in the 4-inch (10.2 cm) dewar. The slush is accumulated on the sample plate either by letting it settle from the liquid surface during slush production, or by stirring particles from the bottom of the dewar onto the plate. With the liquid in the dewar quiescent, the plate is rotated until the solid hydrogen particles begin to slide off. If only partial clearing of the plate occurs, the plate angle is increased until about 95 percent of the surface is free of particles. The angle of the plate is measured on a photograph of the plate silhouette. Well defined vertical shadows are used as a reference angle.

Figure 2 presents the experimental friction angle (the angle from the horizontal at which the particles slide off the plate) versus the surface finish, for both freshly prepared stirred slush and slush aged an average of 4 hours. Each symbol plotted as friction angle is the average

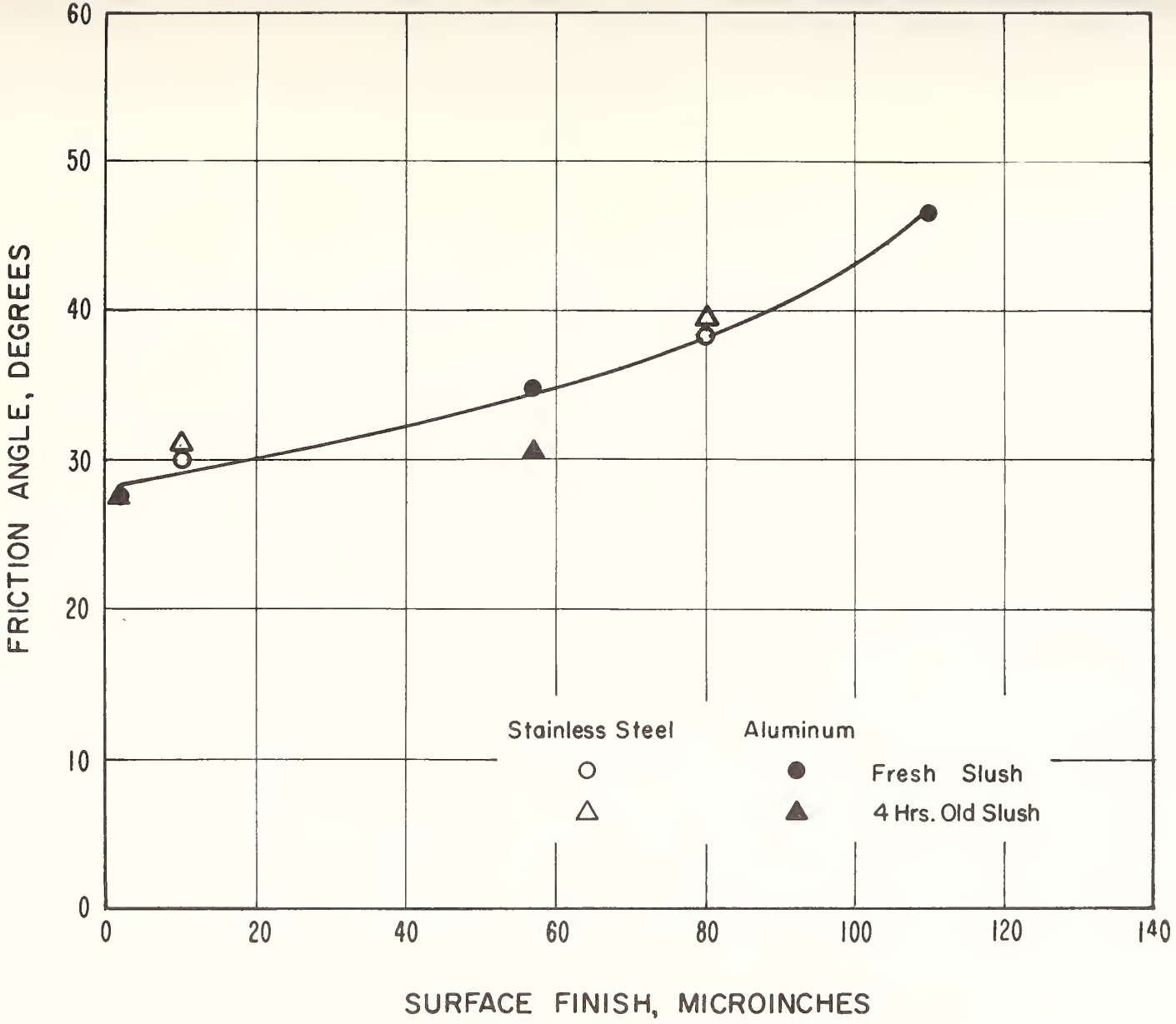

Figure 2. Friction Angle Data

of 5 measured values. No significant difference between the freshly prepared and aged slush data is apparent.

The random error of the friction angle, expressed as the estimated standard deviation of the average of a series of 5 measurements, is 1.6 degrees. The systematic error is believed to be less than 2 degrees.

Because the curve in figure 2 defines the minimum slope at which unstirred slush hydrogen slides, tank bottom slopes should be greater than these by at least 5 degrees. Thus, slush tank bottom slopes should generally never be less than 35 degrees and need exceed 45 degrees only when a very rough surface finish is used. However, design of tank bottom exits is a special problem which requires more than friction angle considerations. Even with the correct tank bottom slope, bridging of solids at the tank exit may occur at low transfer rates.

4. Slush Hydrogen Flow through Smooth Tubing

Characterization of slush hydrogen flowing in insulated transfer lines is required for the evaluation of existing facilities, and the design of new systems. Pressure losses in transfer lines must be predicted for the transfer of slush in ground support equipment and the loading and upgrading of flight vessels. Hydrogen facilities also include many flow restrictions such as orifices, valves, and nozzles which will be required to pass slush. To assist in the design of such systems, experiments to determine the pressure losses in transfer lines and flow restrictions have been made in the NBS flow facility.

4.1 Flow Facility

The flow facility used to measure the pressure drop of slush hydrogen flowing in a vacuum insulated transfer line is shown in figure 3. The apparatus consists basically of a generator dewar, where slush is

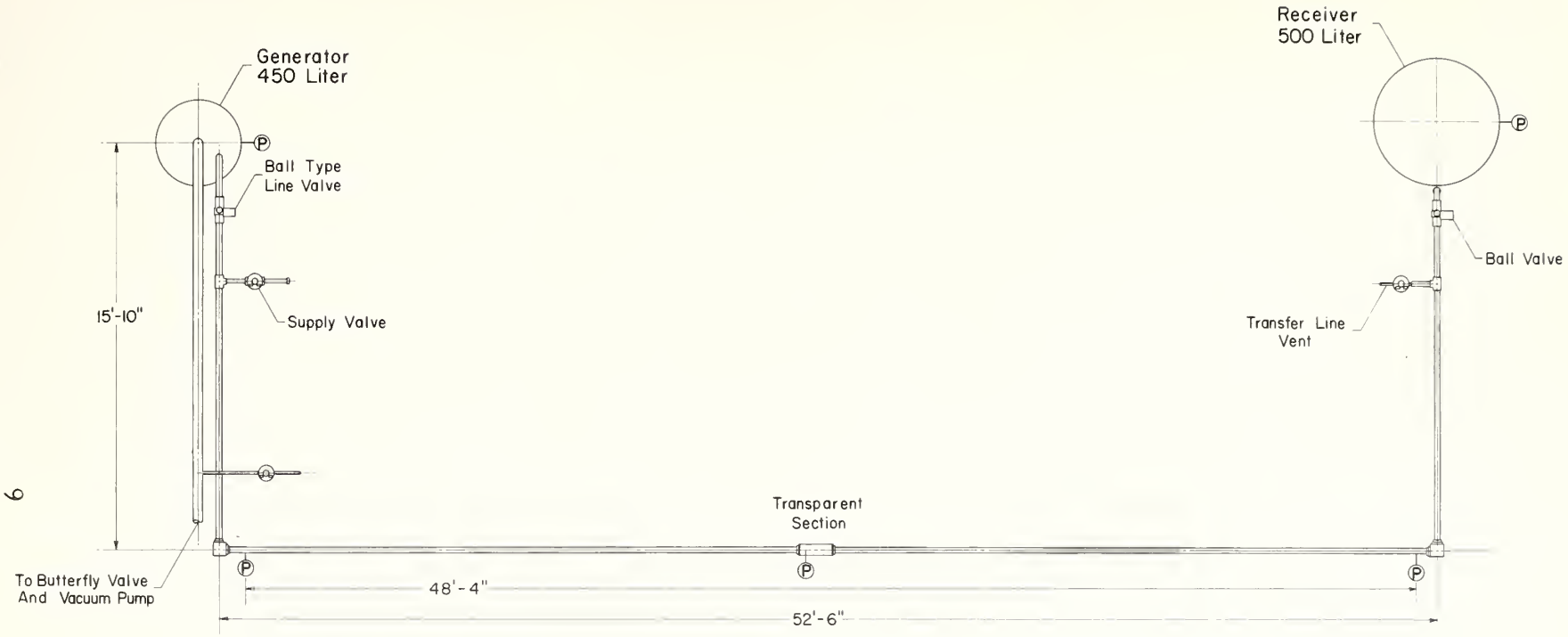

Figure 3. Flow Loop Schematic

produced, approximately 80 feet (24.4 m) of vacuum insulated transfer line with a transparent section near the middle, and a receiver dewar to collect the slush.

A cross section of the generator is shown in figure 4. The dewar has a 4-inch (10.2 cm) pumping line at the top which is connected to three vacuum pumps with a hydrogen pumping capacity of 1300 cfm for producing slush by the freeze-thaw method. A stirring shaft connected to an external air motor extends through the vertical 4-inch (10.2 cm) line to the dewar, where agitators are attached for stirring the slush. Carbon resistor liquid level sensors spaced 3 inches (7.6 cm) apart are attached to a vertical micarta rod located near the side of the dewar.

A nuclear radiation attenuation (NRA) densitometer is used to determine slush mass-solid fraction in the generator. The densitometer consists of a 4 curie, 0.663 MeV, Cesium^{137} gamma source that emits a radiation beam through the center of the dewar to a detector 180° from the source. The center of the beam is one inch below liquid level sensor number four. The beam penetrates 30 inches (76.2 cm) of slush, 0.44 inches (1.12 cm) of stainless steel, and 0.5 inches (1.27 cm) of aluminum. Approximately one-half of the attenuation of the beam takes place in the slush.

Three 6-inch (15.2 cm) diameter windows for observation are located 120° apart on top of the dewar. The generator has a bottom outlet into the vacuum insulated transfer line.

The receiver is a vacuum insulated, nitrogen shielded dewar which also has a bottom outlet.

The transfer line connecting the two dewars is a conventional vacuum insulated line. The inner line is 0.652 inches (1.66 cm) inside diameter and has bellows assemblies at each corner and adjoining the

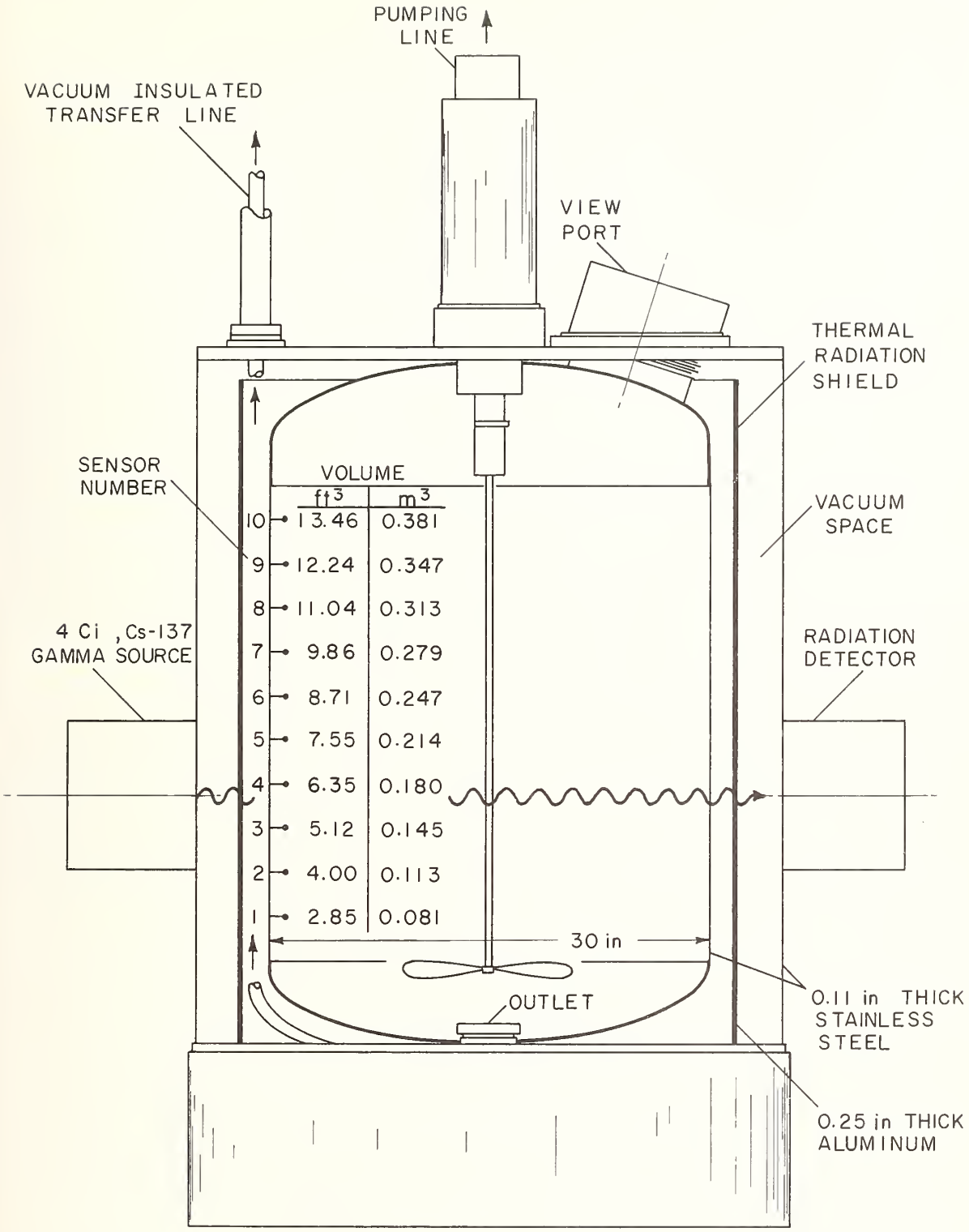

Figure 4. Slush Generator

transparent section, to allow for differential contraction. The bellows have internal telescoping tubes to provide a smooth uninterrupted flow path. The transparent section is formed by a large box around the inner line with clear plastic side plates. This affords a convenient location to observe slush flow through a Pyrex tube in the inner line and provides easy access to the inner line for installing restrictions.

Ball valves are located near each of the dewars to control flow and precooling operations. Pressure taps are located at the points indicated, one on each dewar and three on the straight section of the transfer line. The pressure taps on the dewars are small lines into the ullage space. The transfer line taps are more involved because of thermally induced pressure oscillations which occur in lines exposed to slush on the open end and to a warm environment on the closed end [Daney et al., 1967].

A schematic of a transfer line corner pressure tap is shown in figure 5. The tap consists of a 0.042-inch (0.11 cm) diameter hole in the top of the inner line. The hole is located approximately 30 diameters from the elbow to assure a fully developed flow profile. A small copper cap covers the hole and a 0.046-inch (0.12 cm) inside diameter stainless steel tube is soldered into the side of the cap and extends to the corner where it passes through a seal in the corner box to the outside. The line is then connected to a variable reluctance type pressure transducer and a resonator tube. The resonator tube was found to be necessary in order to dampen the thermal oscillations present with slush transfers [Daney, et al., 1967]. The transducers are differential pressure measuring type and are connected to a common reference pressure.

Transducer diaphragms of 0.5, 2, and 5 psid (0.35, 1.38, and 3.45 N/cm^2) ranges were used to measure pressure drop in the transfer

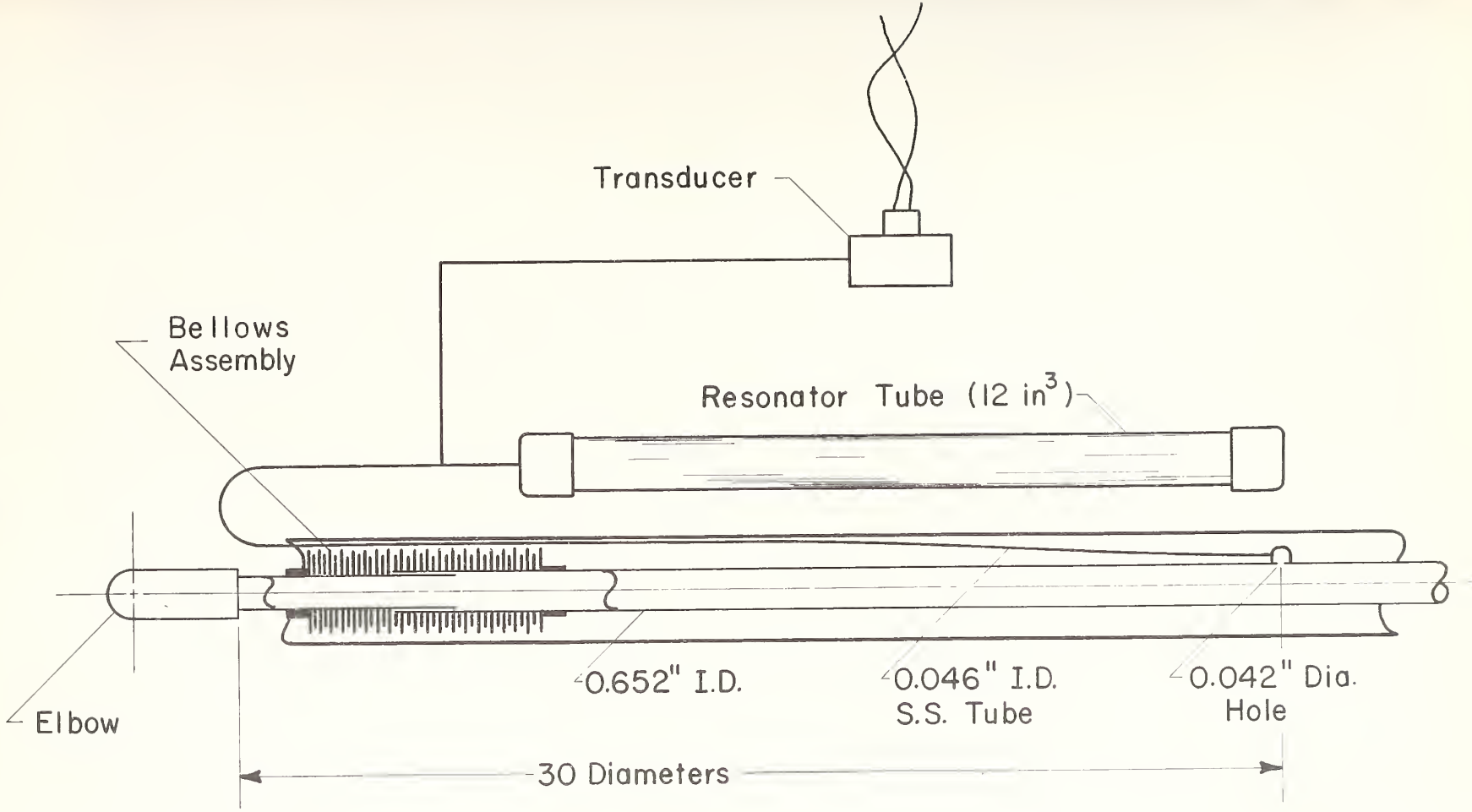

Figure 5. Transfer Line Pressure Tap

line. Pressure drop was measured only in the straight section of the line between the two corner pressure taps, located 48 feet, 4 inches (14.74 m) apart, as shown in figure 3.

All the experimental data were recorded in an adjacent room at the data acquisition console shown in figure 6. The console contains the readout for the densitometer, a recording chart for recording densitometer output, and the liquid level sensor signal conditioning unit which has lights for visual observation that indicate which resistors are immersed in liquid. This unit also puts out a d-c voltage from -1 to +1 volts in 0.2 volt increments for resistors in liquid. The power supply and signal conditioning unit for the pressure transducers are also located in the console. The unit generates a d-c voltage from -1 to +1 volts proportional to pressure. All pertinent data for a particular test may be recorded on a 16-channel data acquisition unit capable of recording 400 characters per second on magnetic tape. The data are then taken from the magnetic tape and reduced to printed form using a high speed digital computer.

Other types of density measurement instrumentation have been, or are being evaluated in addition to the NRA 4 curie source densitometer described. A 17 curie, cesium^{137} source NRA densitometer has been used in the slush generator and its performance compared to the 4 curie source unit. It is at least as good as the 4 curie unit and very likely better. It will be further evaluated in the density reference system described by Weitzel, et al. [1968]. A submersible point densitometer is being evaluated in the density reference system. This unit uses a strontium 90 beta ray source. The unit is designed for use in large vessels and will be used in the slush generator when evaluation in the density reference system is complete. The 4 curie source densitometer was the only one used in the flow tests.

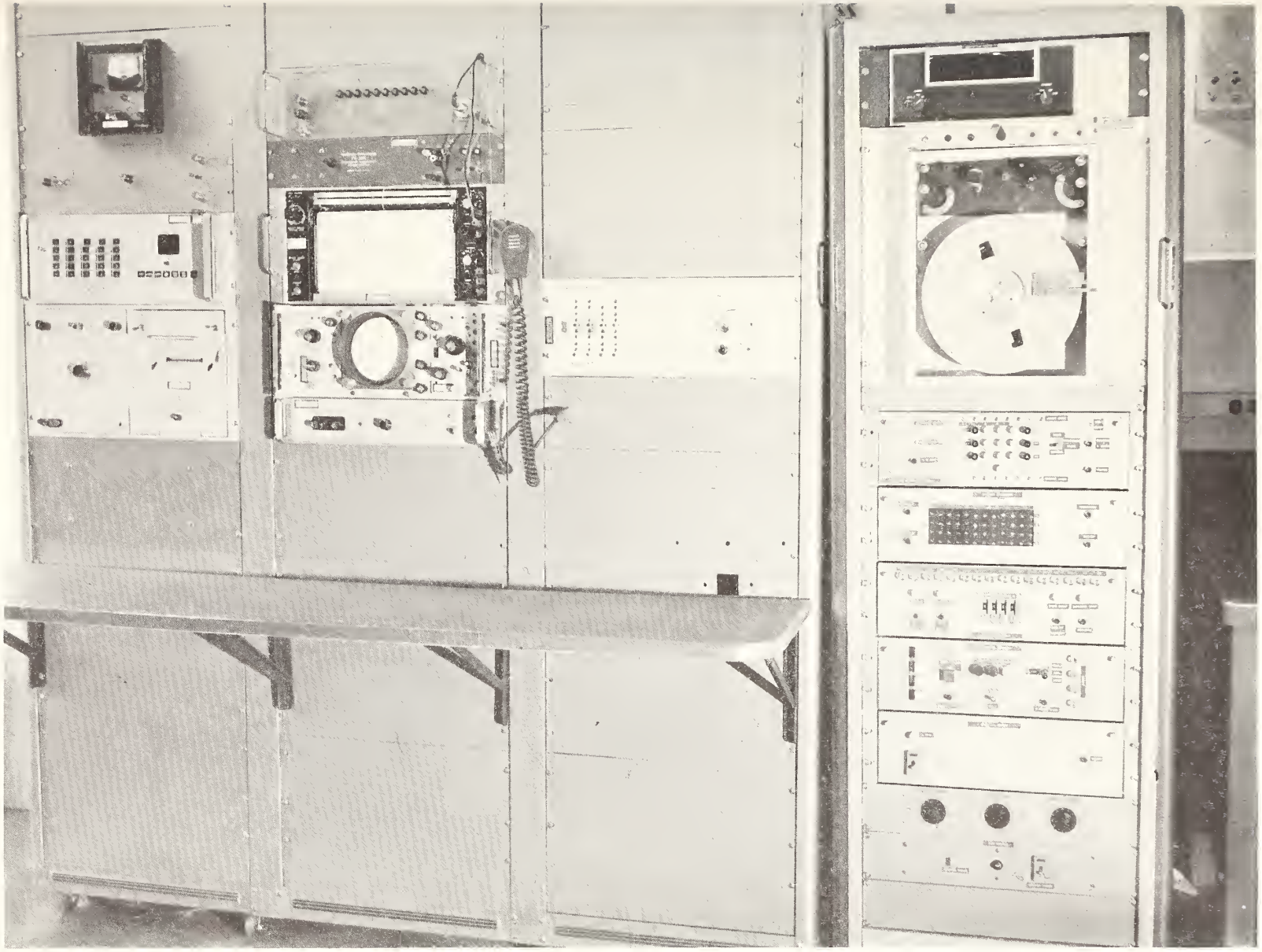

Figure 6. Data Acquisition Console

4.2 Experimental Procedure

4.2.1 Fresh Slush Flow Tests

Slush is produced in the generator by the usual freeze-thaw method [Mann et al., 1966]. The slush density is determined using the NRA densitometer. The transfer line is precooled with liquid from the supply dewar if necessary. The generator ullage is then pressurized with liquid-nitrogen-cooled helium gas. A precision regulator is opened at a positive pressure to maintain constant ullage pressure during transfer. The receiver dewar is either vented through large feather valves or pumped to triple-point pressure and automatically controlled at 1.02 psia (0.703 N/cm^2). Slush flow is started by opening the ball valves. During transfer, the slush is stirred to maintain a homogeneous mixture. Pressure and density measurements are taken between liquid level sensors number 8 and number 3. The data acquisition unit is turned on before approaching sensor number 8. Pressure measurements are recorded every second and liquid level recorded every 0.2 seconds. Stirring is stopped when the liquid level approaches liquid level sensor number 8 because a quiet surface is necessary for precise flow measurements. The stirring is resumed immediately after passing sensor number 8 and the densitometer measurement is also taken immediately after passing sensor number 8. Thus, the density measurement is actually taken at the same time the pressure drop is being measured. Slush may be observed flowing in the inner line at the transparent section during the test. The stirring is stopped again as the liquid level approaches sensor number 3. After passing sensor number 3, flow is stopped and the slush in the receiver is returned to the generator for another test.

4.2.2 Aged Slush Flow Tests

An aged slush flow test is started by making slush in the normal freeze-thaw manner to approximately 0.45 solid fraction as measured with the densitometer. The control for the butterfly valve between the pump and the generator is set to operate automatically, opening the valve when the ullage pressure reaches 1.15 psia (0.793 N/cm^2). Each time the valve opens, a thin layer of solid is made on top of the slush. This continuous upgrading keeps replacing solid melted by the heat leak, and maintains a constant solid fraction. This procedure continues for about 8 hours, during which time the liquid level sensors are shut off to reduce heat leak and approximately 2.4 cubic feet (0.068 m^3) of liquid are stored in the receiver dewar and held at triple-point pressure to maintain that dewar temperature near 14 K. After an 8-hour aging period, the transfer line is cooled with liquid from the supply dewar, the liquid level sensors are turned on, and the aged slush is flowed in the previously described manner.

One noticeable difference between the aged and fresh slush flow tests is the manner in which the solid settles to the bottom of the generator when stirring is stopped. The aged solid settles to the bottom much faster than the unaged solid. This is obviously due to the aged particle having higher apparent density and a more spherical shape. This change in particle character, as reported by Mann, et al. [1966], is nearly complete at 6 hours aging time. To assure that as much of the solid as possible had aged at least 6 hours, the 8-hour aging period was used.

4.2.3 Restriction Flow Tests

The restriction tests are very similar to the normal flow tests. A restriction is soldered into the inner line at the transparent section instead of the usual nonrestricted Pryex tube, and additional pressure taps are placed on each side of the restriction to measure the pressure drop across the restriction.

4.3 Flow Data in a Smooth Tube

4.3.1 Pressure Drop of Fresh Slush

A plot of pressure drop vs. flow and velocity is shown in figure 7. The pressure drop was actually measured across 48 feet, 4 inches (14.74 m) of straight transfer line, and then calculated for 100 feet (30.49 m). The flow was measured with the liquid level sensors in the generator. Seventy-eight data points are shown, each representing a flow test. The mass solid fraction for all of these tests was measured with the densitometer.

The data points are grouped as to solid fraction instead of indicating the solid fraction of each point. The legend gives the symbols for each group. For example, a data point with a solid fraction of 0.43 would be indicated by a solid triangle.

The lines of constant solid fraction were generated by applying a curve fit to these data points. It should be pointed out that the actual solid fraction for each data point, to two significant figures, was used in generating the curves. The solid line represents triple-point liquid and was generated by the same curve fit.

The most important results of the flow tests shown in this plot are that the slush of solid fractions from 0.1 to 0.4 flows with less pressure loss than triple-point liquid. This reduced pressure loss occurs over the velocity range from 12 ft/sec (3.7 m/s) to 45 ft/sec (13.7 m/s). This phenomenon is shown more clearly in figure 8 which is a plot of the pressure loss deviation from triple-point liquid versus flow and velocity, again with lines of constant solid fraction. The maximum deviation from triple-point liquid is approximately 8 percent and occurs with a solid fraction between 0.2 and 0.3.

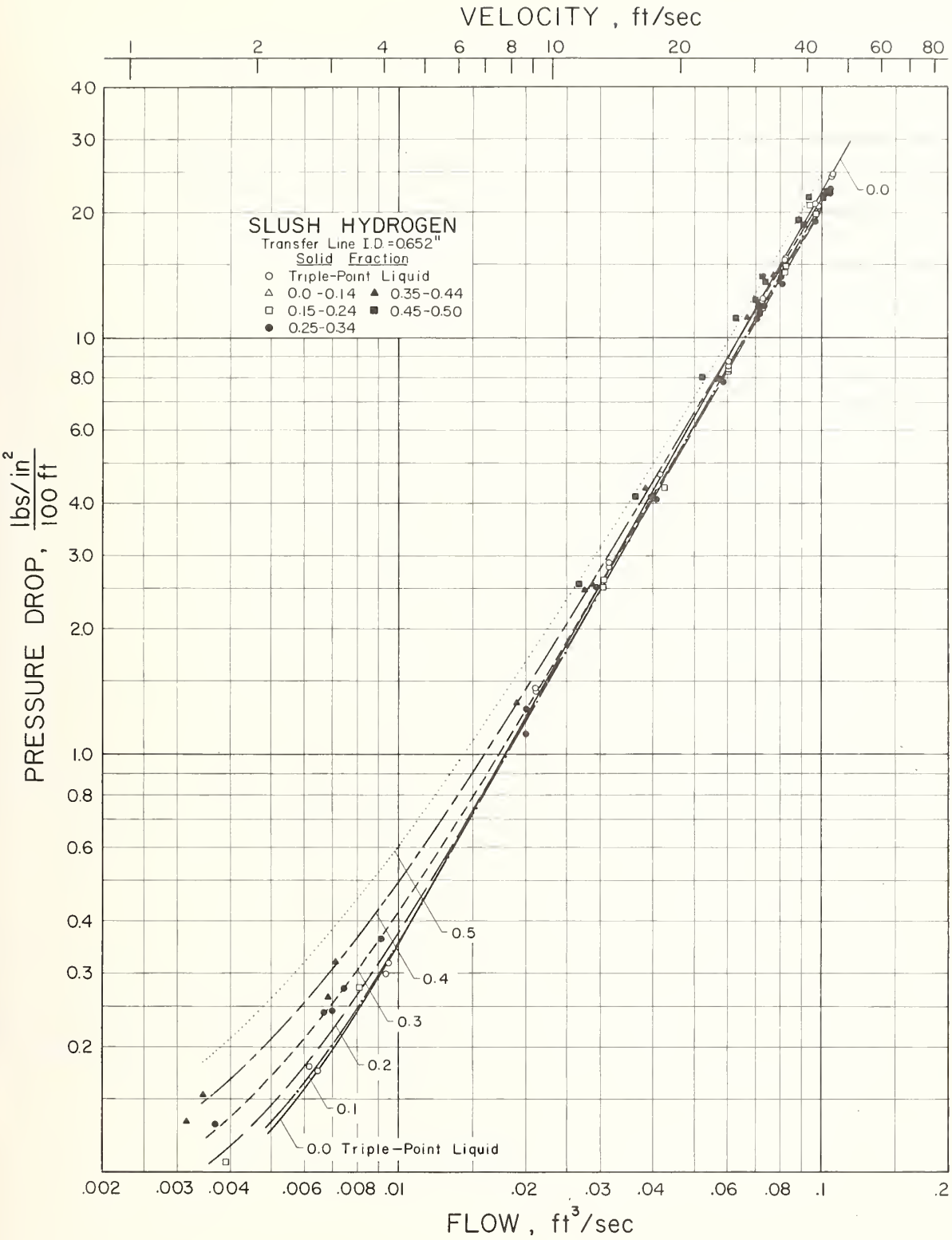

Figure 7. Fresh Slush Flow Data

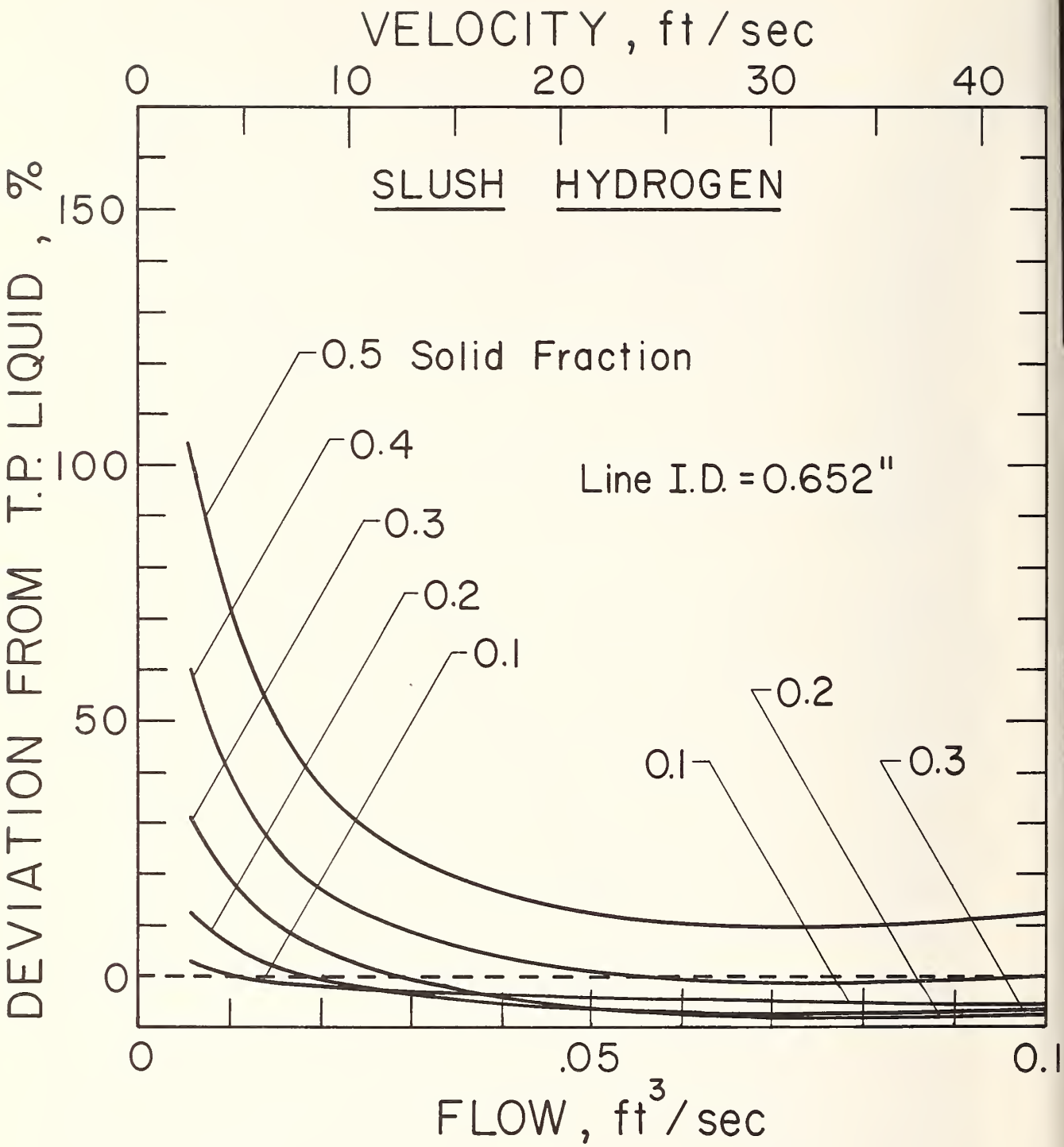

Figure 8. Slush Pressure Drop Deviation Plot

4.3.2 Pressure Drop of Aged Slush

A separate plot showing the data for the aged liquid-solid mixtures is given in figure 9. It should be emphasized that the data points on this figure represent aged slush flow tests, while the curves are from the 78 unaged slush points. The actual solid fraction of each point is indicated in the figure. A detailed comparison of the data for aged and for fresh mixtures indicates that the aged slush has a pressure drop 4 to 10 percent higher than similar solid fraction unaged slush in the flow regime covered. Each data point represents a test which included at least 8 hours aging time.

4.3.3 Friction Factor and Reynolds Number

A plot of friction factor versus Reynolds number for the pipe flow data with fresh slush is shown in figure 10. No aged mixture data are included in this plot. The solid line is an analytic expression generated from considerable experimental flow work on incompressible fluids in smooth pipes by Kármán, Nikuradse, and Prandtl, [Binder, 1949]. The lines of solid fraction were calculated from the curve fit data as indicated below. The friction factor was calculated using the Darcy-Weisbach equation [Binder, 1949] for pressure drop,

$$\Delta P = \rho f \frac{L}{D} \frac{V^2}{2g_c}$$

or

$$f = \frac{2g_c D \Delta P}{L \rho V^2} .$$

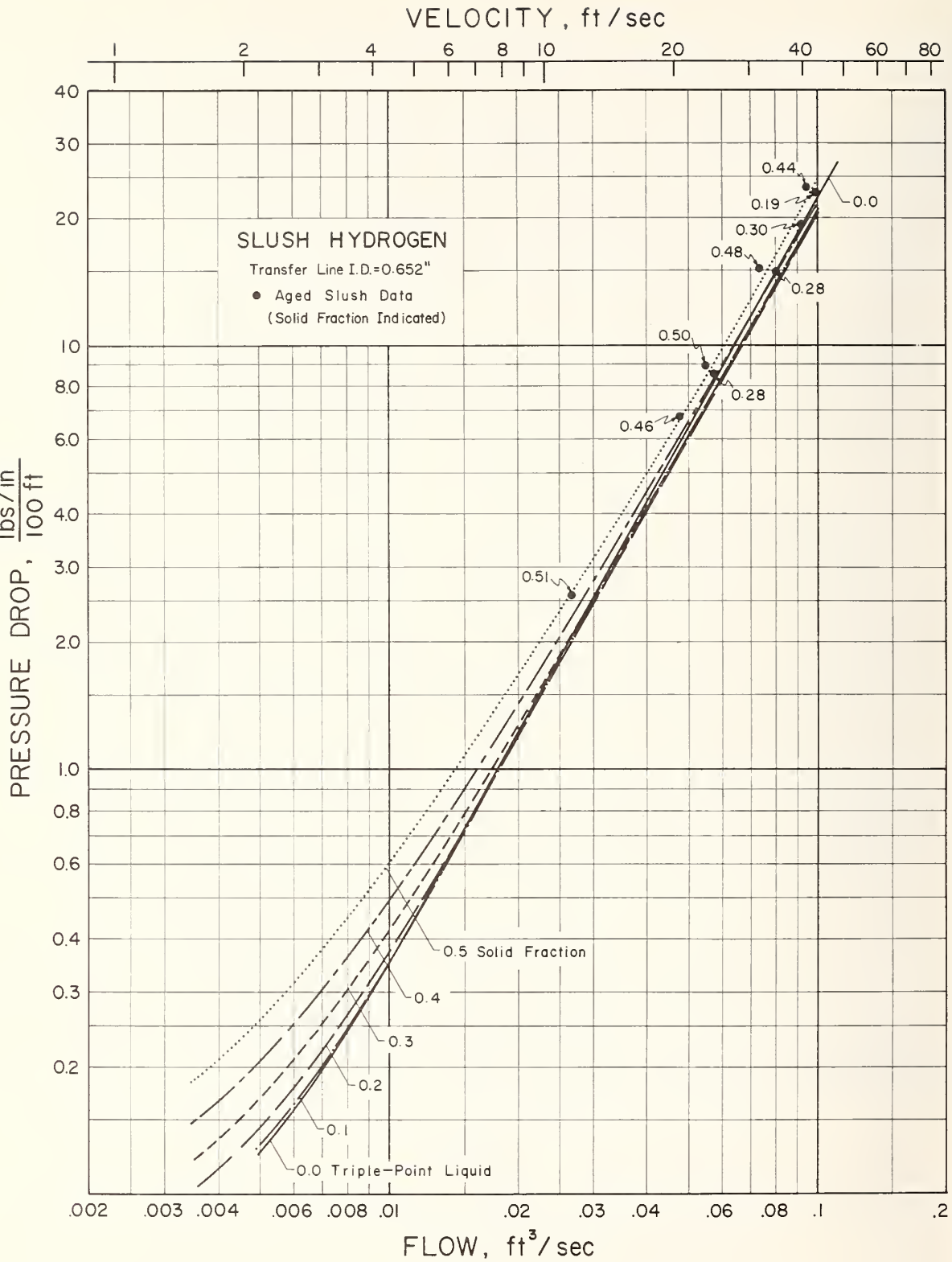

Figure 9. Aged Slush Flow Data

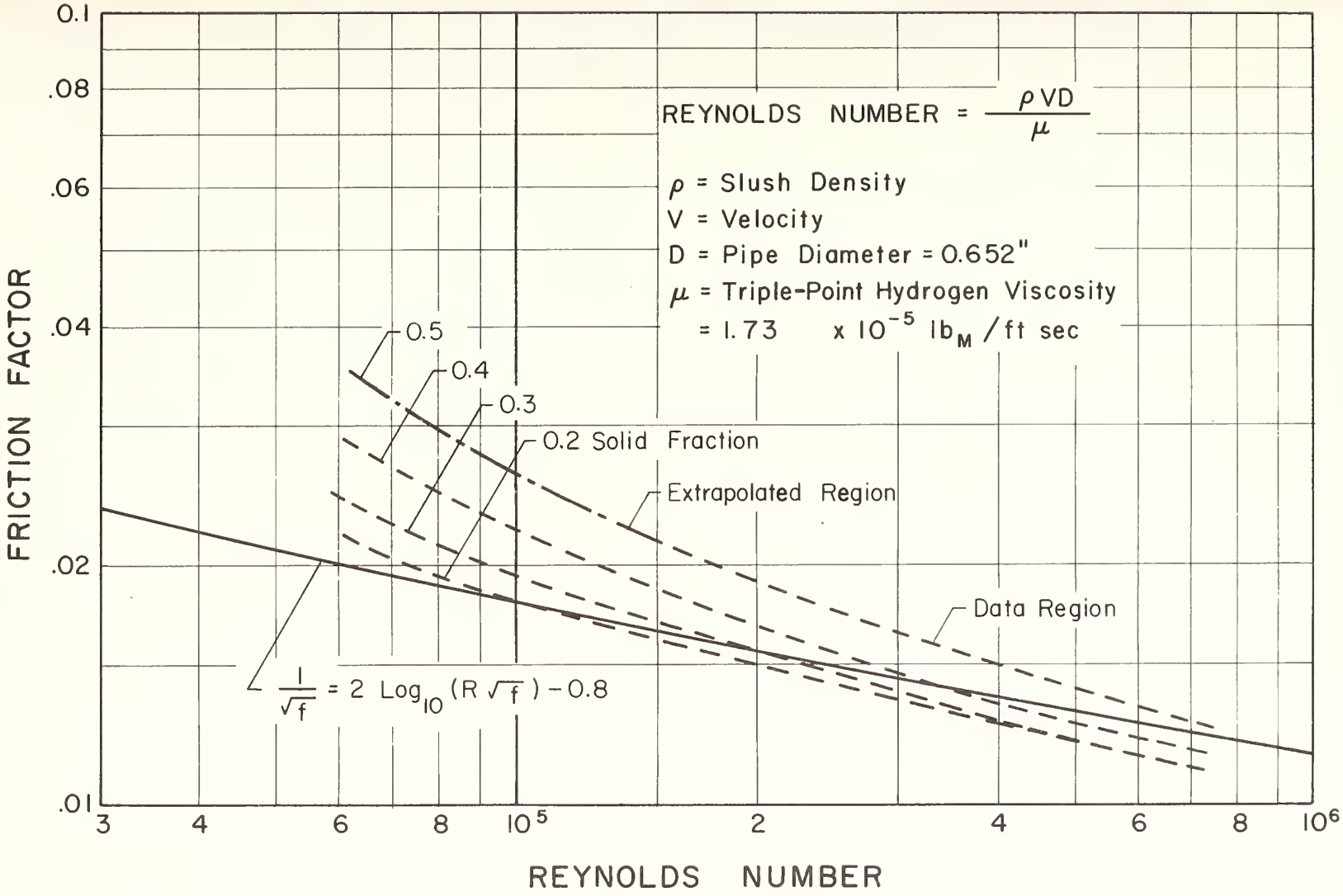

Figure 10. Friction Factor vs. Reynolds No. for Slush Hydrogen

In the equation the independent experimental variables are pressure drop, ΔP; velocity, V; and density, ρ. The constants for this test are D the pipe diameter, L the pipe length, and g_c the gravitational constant. The Reynolds number was calculated using the experimental density of the fluid, experimental velocity, the pipe diameter, and the viscosity of triple-point liquid [Diller, 1965]. It should be emphasized that the viscosity of triple-point liquid was used to calculate the Reynolds number for each solid fraction line. Using the viscosity of triple-point liquid was necessary because the viscosity of slush hydrogen is unknown and indeed may be a meaningless parameter. The data points for triple-point liquid have a standard deviation of 3.3 percent about the curve of Kármán's analytic expression.

The reduced friction losses of slush compared to triple-point liquid are more evident in the dimensionless plot of friction factor versus Reynolds number than in the pressure drop curves. The effect of the higher slush densities on pressure loss is eliminated in the dimensionless plot. It is thus apparent that the friction losses with slush are less than triple-point liquid at higher velocities or Reynolds numbers and continue to decrease as the Reynolds number increases. The friction factor for slush of 0.4 solid fraction is less than for triple-point liquid at Reynolds numbers as low as 3.5×10^5, which is equivalent to a flow of 0.053 ft^3/sec (1500 cm^3/s) in the NBS flow loop. From the slope of the 0.5 solid fraction line, it appears that at higher Reynolds numbers the friction factor for 0.5 solid fraction slush would also be less than for the friction factor of triple-point liquid.

4.3.4 Discussion of Data

In the data analysis for flow versus pressure drop some of the earlier data points were discarded in which the solid fraction was determined by the mass-energy method [Daney and Mann, 1967]. Determining

solid fraction by the mass-energy method was not acceptable in the larger flow system because of inaccuracies in measuring the mass removed and the heat leak to the generator.

The reduced pressure loss of slush compared to triple-point liquid at the higher velocities is not readily explained by classical hydrodynamics or rheology. The decreased pressure drop is most likely due to a reduction in friction factor as suggested by figure 10.

Other experimentalists have observed reduced friction losses. Some refer to viscosity changes instead of friction losses and assign apparent viscosities to the mixtures. However, since solid particles settle out of slush hydrogen unless turbulence is maintained, viscosity of the mixture is not readily obtainable nor very meaningful. Thomas [1962] has done work on transport characteristics of non-Newtonian suspensions in which turbulent flow friction factors were determined for flocculated suspensions flowing in tubes 1/8 inch (0.3 cm) to 1 inch (2.5 cm) in diameter. These suspensions were of the Bingham plastic type in that they support a given stress before yielding or flowing. Thomas found that these turbulent friction factors were always less than those of Newtonian fluids and that, depending on the yield values, the friction factors diverged from or converged with the Newtonian values as Reynolds number increases. For high yield values of the fluids the friction factor diverged from the Newtonian values. He states that the friction factor behavior "is believed to be due to a damping of the turbulent fluctuations by the flocculated solids, with the effect increasing both with volume fraction solids and with the ratio of the attractive force between particles to the disruptive force of turbulent fluctuations." Thomas recommends studies to identify the basic mechanism responsible for the observed flow phenomenon.

Durand [1953] observed a reduction of the friction loss in the range of laminar plug flow when flowing mixtures of coarse sand mixed with clay. He found that "for high concentrations, the mixtures obtained are homogeneous, and the head losses are considerably reduced."

Stepanoff [1964] cites several investigators who have found reduced friction loss when flowing liquid-solid mixtures. He refers to a series of recent tests (1962) run for the Bureau of Mines by the Montana School of Mines in which slurries of sandstone (specific gravity 2.62), mine tailings (2.9), and barite (4.24) in consistencies up to 70 to 80 percent by weight were flowed in 2, 3, and 4-inch (5, 7.6, 10.2 cm) pipes. They found that slurries in consistencies from 40 to 80 percent have shown the pipe friction loss, in feet of mixtures, to be lower than that of clear water.

Stepanoff also refers to a pipeline between Cadiz and Cleveland, Ohio, in which it has been found by long experimentation that plug flow of coal slurry is the most economical and practical mode of flow to transport coal by pipeline.

Stepanoff indicates that one of the economical advantages of operating a pipeline carrying a liquid-solid mixture in the plug-flow region is that "for a given pipe velocity, the friction loss is lower with plug flow than with laminar or turbulent flow." Stepanoff also states "the discovery that solid-water mixtures with solids content up to 70 percent by weight cause less pipe friction loss expressed in feet of mixture, than clear water" is, in his opinion, one of the most important discoveries in hydraulics in the past 100 years.

The work performed with slush has several significant differences from that of other experimentalists. The most important is that the density of the solid in the slush mixture is only 11 percent greater than

the liquid. Data available are usually on liquid-solid mixtures with the solid two to four times more dense than the liquid. Stepanoff [1964] does refer to one case of flowing paper pulp in water where friction losses are less than with clear water losses in the turbulent region. Liquid-solid mixtures of hydrogen may be similar to pulps or flocculated slurries when the particles are fresh as fresh particles have the appearance of an intricate network of very small crystals loosely attached [Mann, et al., 1966]. This may account for the apparent increase in friction losses in the aged slush which has particles similar in appearance to spherically shaped crystals. Another area of significant difference in slush hydrogen from most liquid-solid mixtures is that the solids are of the carrier liquid and any contact of solids with the tube wall will likely result in melting to always form a liquid film at the contact point.

In the flow work referred to above, the reduced friction loss appears to be a function of flow velocity and plug flow and not pipe size. Therefore, it is possible that the friction factors determined in the turbulent region, especially at the higher Reynolds numbers, would apply to larger pipe sizes. Further analysis and testing in other pipe sizes would be required for a better understanding of the reduced friction loss phenomenon and to assure application to other sizes of tubes.

4.3.5 Error Analysis

There are three independent variables subject to error in the flow system; 1) the flow or velocity, 2) the pressure measurement, and 3) the fluid density.

The flow is measured with the generator liquid level sensors and a clock on the data acquisition unit. An error analysis of the flow measuring instrumentation has been made which indicates that a random error of 2 percent can be expected when measuring a level change of 3

inches (7.6 cm), (one sensor), and this error decreases to 1 percent if the measurement is taken over 15 inches (38.1 cm). Most of the experimental data points were taken with flow being measured over 12 inches (30.5 cm).

Another flow error may be introduced if the liquid surface is not quiescent as the measuring sensor is uncovered. During the tests, the slush was stirred constantly to keep the fluid homogeneous, except when the surface passed by the measuring sensors. Although an effort was made to maintain the surface quiescent at this time, this was another possible source of error which can only be estimated. Thus, considering the human and other random errors, it is estimated that the error in flow measurement could be as high as 2 percent.

The pressure drop across the straight section of transfer line was measured using three different sets of transducer diaphragms of a range of 0.5, 2, and 5 psid (0.35, 1.38, 3.45 N/cm^2). The standard deviation from the least squares straight line curve fit to the calibration data for each of these diaphragms was calculated. Since these calculations were made with the transducers installed in the system, the standard deviation of the calibration should be a good estimate of the random error under test conditions. The standard deviations of the calibrations were 3.7 and 6.8 percent for each of the 0.5 psid (0.35 N/cm^2) diaphragms, 1.6 and 1.8 percent for each of the 2 psid (1.38 N/cm^2) diaphragms, and 2.8 and 1.8 percent for the 5 psid (3.45 N/cm^2) diaphragm Since there are two transducers involved in determining the pressure drop, the standard deviation for the pressure measurement using both transducers is expressed as

$$\sigma_P = \sqrt{\sigma_1^2 + \sigma_2^2}.$$

Combining the deviations of both transducers for each range results in a standard deviation for the pressure drop of 7.4 percent in the pressure drop range from 0.01 to 0.03 psi/100 ft (0.007 to 0.02 N/cm^2 per 30.5 m), a standard deviation of 2.4 percent in the range from 0.03 to 12 psi/100 ft (0.02 to 8.3 N/cm^2 per 30.5 m), and a standard deviation of 3.3 percent in the range from 12 to 25 psi/100 ft (8.3 to 17.2 N/cm^2 per 30.5 m).

The density or solid fraction measurement was taken with the NRA densitometer. There are three possible sources of error to consider for the density measurement: an error due to sampling, an error due to solid degradation (melting) between the generator and transfer line test section, and the accuracy of the densitometer calibration.

There is the possibility of a density gradient in the dewar when stirring the slush. However, observation of the stirring action in the dewar does not indicate this is the case. The slush is stirred in a circular pattern and it also rolls over from bottom to top due to the baffles and high pitch propellers. Since the radiation beam penetrates the center of the mixture and it appears to be homogeneous, the error due to sampling should be negligible.

There is also the possibility of significant solid melting, due to heat leak between the generator (where the density is measured) and the actual test section of the transfer line. A heat loss calculation was made to determine the extent of the degradation at various flow rates. A plot of solid fraction loss versus flow rate is given in figure 11. The loss of 0.05 solid fraction at 0.004 ft^3/sec (113 cm^3/s) flow rate is significant. Four data points were taken at this flow rate. The next eleven data points were taken at a flow rate of 0.008 ft^3/sec (227 cm^3/s) where the solid fraction loss is 0.025. The next set of data points were taken at a flow rate of 0.02 ft^3/sec (566 cm^3/s) where the loss is only 0.01

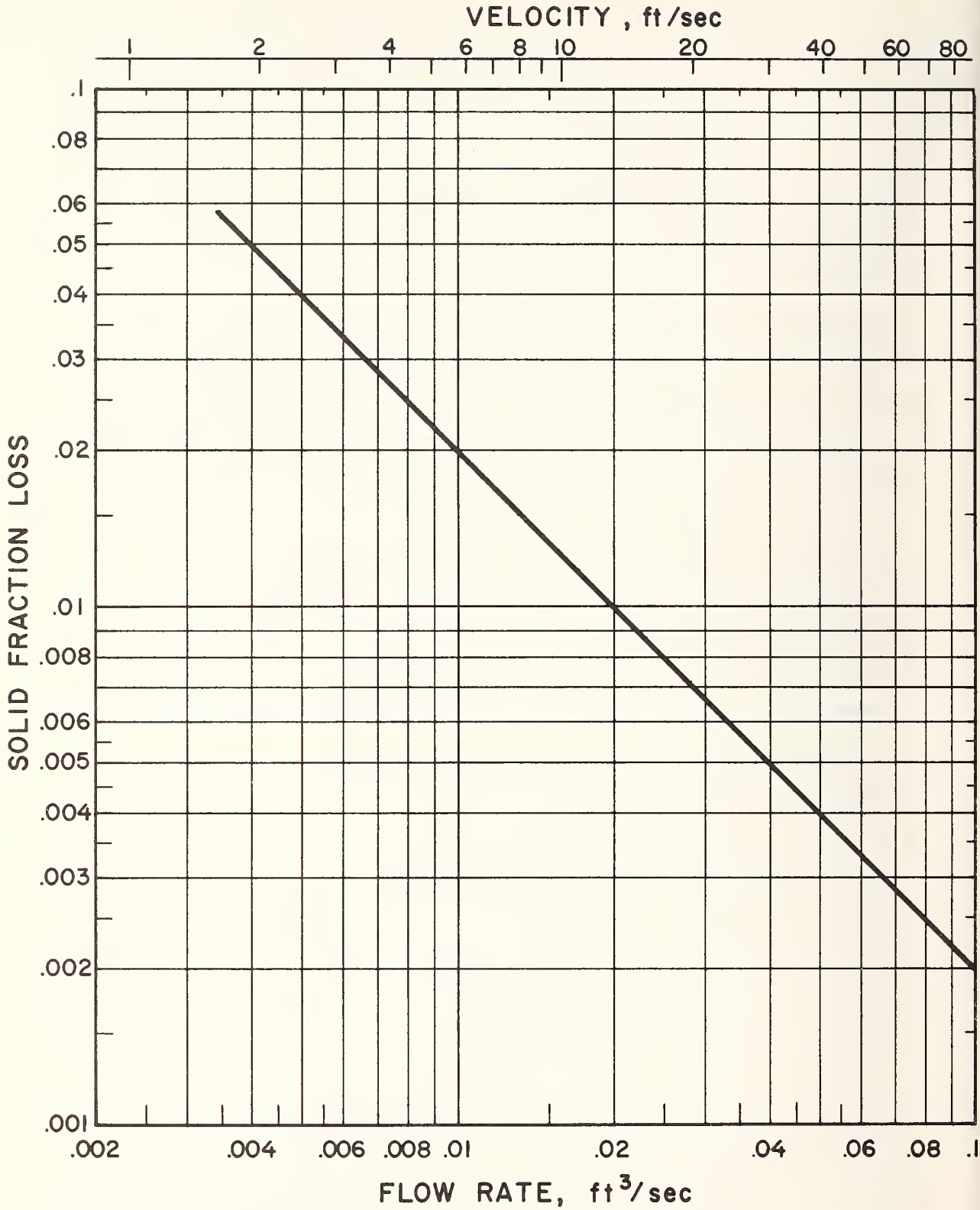

Figure 11. Solid Fraction Degradation from Heat Leak

solid fraction and at flow rates higher than this, the solid fraction loss is insignificant. The net effect of the heat leak would be increased pressure drop for the lines of constant solid fraction in figure 7 at the very low flow rates. This effect is not included in the curves of figures 7 and 9.

Another possible source of density error is the calibration of the densitometer. A previous error analysis of the calibration procedure has shown the uncertainty of the measured density values to be 0.3 percent at 0.5 solid fraction [Weitzel et al., 1968]. However, a zero-shift between morning and evening calibrations has become apparent, which was not previously considered. This shift was first noticed during the aging tests and is most likely due to a change in temperature of the ionization chamber or other associated electronics between a morning and evening calibration. An arithmetic average of the shift for six calibrations is equivalent to a densitometer output of 0.07 millivolts or about 0.01 solid fraction. Combining both of these errors, it is estimated that the error in density measurement due to the densitometer calibration could be as high as ±0.4 percent.

Considering all three sources of error; the sampling technique, the solid degradation, and the densitometer calibration, it is estimated that the error in measuring the density of the slush is ±0.5 percent for all of the data points at flow rates greater than 0.02 ft^3/sec (566 cm^3/s). Below this value the error is greater depending on the flow rate.

The error analysis of each independently measured variable has been given to supply some estimate of the accuracy of the instrumentation. Because the variables are not linear functions of one another, the combination of these errors is not attempted. The random error in the pressure determination due to all sources, including the effects of the random error in each of the measured parameters of flow, density, and pressure is more easily found by the scatter of the data about the three

dimensional curve fit of figure 7. These data have a standard deviation of 4 percent over the entire range. The random error in the position of the curves is only a fraction of this value.

To estimate the systematic error, the triple-point liquid data are compared to the curve predicted by the Darcy-Weisbach equation and the experimental friction factor of Kármán. These data have a 3.3 percent standard deviation about the predicted curve. It is estimated that less than one percent of the uncertainty in the triple-point liquid data is due to the systematic error. The systematic error in the slush data region includes an error in density or solid fraction measurements. This error in the density determination has been previously estimated to be 0.5 percent [Daney, et al., 1968] which is equivalent to approximately ±0.05 in solid fraction. The effect on pressure of the systematic error in solid fraction is not linear and varies considerably depending on the solid fraction and flow rate. At a solid fraction of 0.5 and a flow of 0.1 ft^3/sec (2832 cm^3/s), the estimated systematic error in density results in an uncertainty of 7 percent in pressure. However, at the same flow rate and a solid fraction of 0.3 the error in density results in a pressure uncertainty of only 2 percent. The worst case is at low flow rates where a change in solid fraction results in large pressure changes. At a flow rate of 0.005 ft^3/sec (141.6 cm^3/s) the density uncertainty of 0.5 percent results in pressure uncertainties as high as 20 percent. The total uncertainty thus varies greatly depending on the area of data. At the higher flow rates, and solid fractions of 0.2 to 0.4 the total uncertainty in the curves for pressure is estimated to be as low as 4 percent. At the lowest flow rates, and a solid fraction of 0.5 the total uncertainty in pressure may be as much as 30 percent.

4.3.6 Conclusions

Slush of mass solid fraction of approximately 0.3 flows with less pressure drop than triple-point liquid at velocities greater than 12 ft/sec (3.7 m/s). Higher solid fractions flow with greater pressure losses in this velocity range, as much as 20 percent greater for 0.5 solid fraction. At low velocities, slush flows with a pressure drop as much as 100 percent greater than triple-point liquid.

The triple-point liquid data agree very well with predicted performance. The slush data are very consistent as is evident by a standard deviation of 4 percent for all data including triple-point liquid. Since the triple-point liquid data agree so well with predicted performance, and the slush data are so consistent, and since both sets of data were taken with the same instrumentation in the same flow system, there is considerable confidence in the slush flow data.

Slush hydrogen flows at very low velocities. Flow at velocities as low as 0.5 ft/sec (0.15 m/s) has been observed in motion pictures. At this velocity the solid settles to form a vertical density gradient with the top portion of the line flowing clear liquid. At the bottom of the line, the solid moves in a sliding-bed flow regime at a reduced velocity of approximately 0.3 ft/sec (0.09 m/s). In the center of the tube the flow is in the saltation regime with particles tumbling over the sliding bed. At flows of 1.5 ft/sec (0.46 m/s) the line is filled with flowing solids, but a significant vertical velocity and solid concentration gradient is still present and the bottom solids are still near a sliding-bed flow. At velocities above 1.5 ft/sec (0.46 m/s) the moving bed type flow changes to a uniform flow in which the solids concentration appears to be uniform. From these observations, it appears that the velocity at which solids tend to settle, referred to by some authors as the standard or critical velocity, is about 1.5 ft/sec (0.46 m/s).

5. Slush Hydrogen Flow through Restrictions

Restrictions of three different types were installed in the transfer line in the center transparent section. This positioned the restrictions at a point halfway between the pressure taps used for the tube flow measurements. Additional pressure taps were installed to measure the pressure drop across the restriction. The procedure for data acquisition was the same as that explained in section 4 for obtaining the flow data in a smooth tube. The pressure drop across the restriction was measured with an additional pressure transducer at the same interval as the other pressures. The diaphragm in this transducer was selected to cover the expected pressure drop for each restriction.

The three different types of restrictions used were a globe valve, an orifice, and a venturi. The orifices and the venturi were made of glass and were fabricated in a glass tube to allow visual and photographic observation. A glass tube was installed downstream of the valve for the same purpose.

The orifices and the venturi were not instrumented to evaluate their performance as flowmeters, because of the difficulty of providing pressure taps built to specifications in glass apparatus. Also, the use of glass prevented fabrication to the tolerances required by flowmeters.

5.1 Description of the Restrictions

The globe valve was a nominal 3/4-inch (1.9 cm) copper valve with solder-joint end fittings. To eliminate packing leaks, the valve stem was soft soldered in position. Two positions were evaluated, one with the valve open 1/4 inch (0.64 cm), the other with the valve full open. The valve plug gasket was originally a composition material for a water valve and this was replaced with Teflon. Figure 12 is a drawing of the valve. The valve installation in the transfer line transparent section is shown in figure 13.

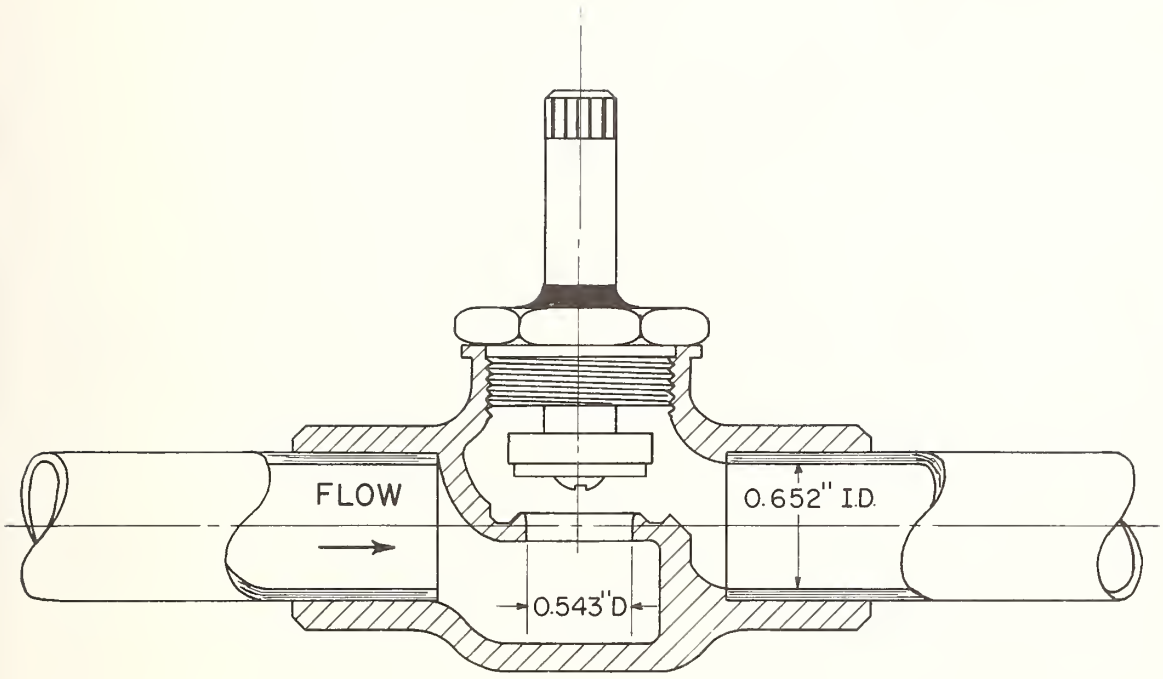

Figure 12. Globe Valve Restriction

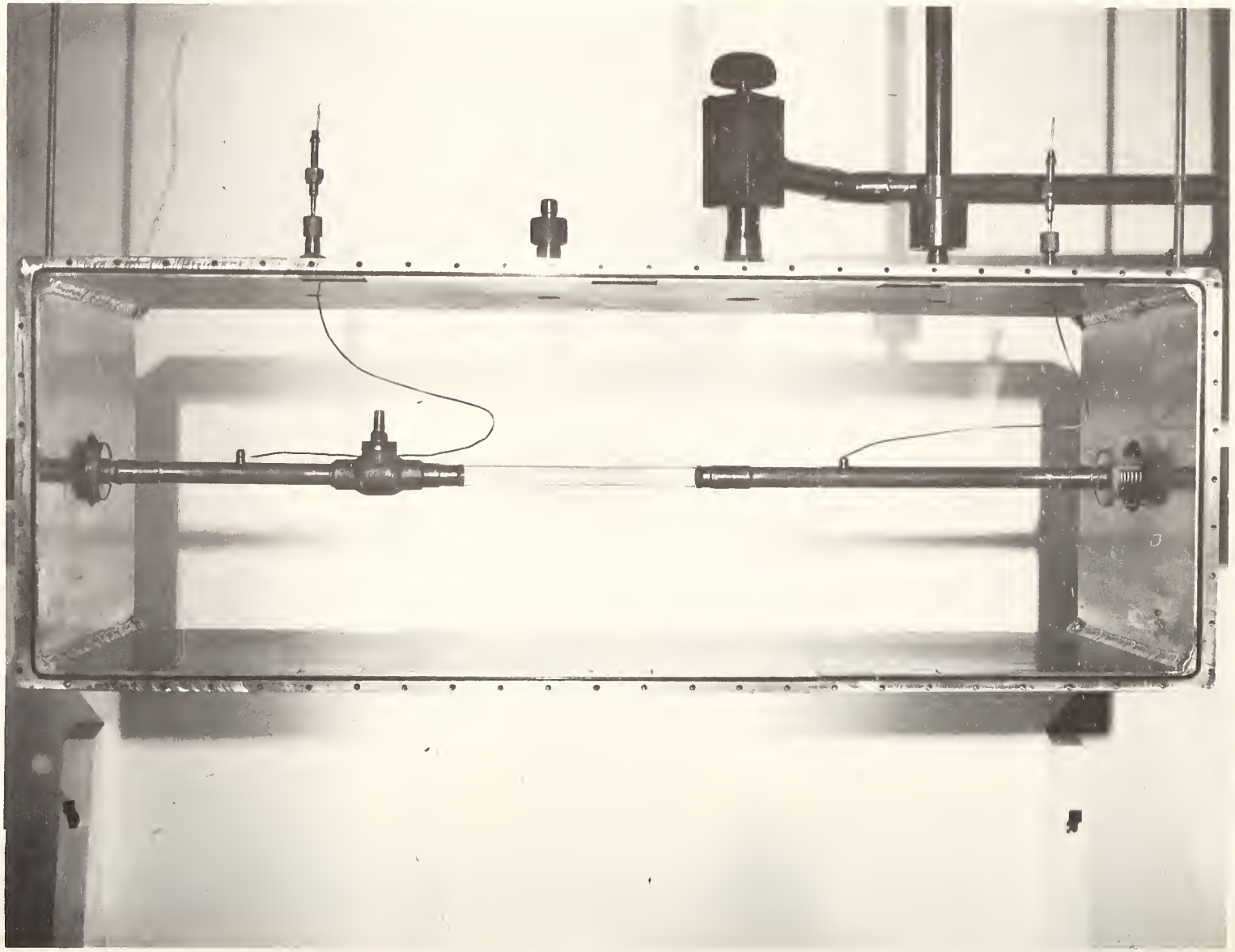

Figure 13. Valve in Transparent Section

Two orifice sizes were evaluated. They were 1/4-inch (0.64 cm) and 3/8-inch (0.95 cm) diameter. Both orifice plates were 0.094-inches (0.239 cm) thick and both were fabricated of glass and mounted in a glass tube of 0.635-inch (1.61 cm) inside diameter. Figure 14 is a line drawing of the orifice, figure 15 is a photograph showing both the 1/4-inch (0.64 cm) orifice assembly and the venturi assembly. The glass to metal transitions on both assemblies were of the glass to stainless steel type joint.

The venturi design was of the Herschel type as this was better suited to glass fabrication. Figure 16 is a drawing showing the dimensions of the venturi. Figure 17 is a photograph of the venturi installed in the transparent section of the transfer line.

The orifices and the venturi were fabricated similar to the specifications of the ASME Power Test Code [ASME, 1959a].

5.2 Test Data and Results

The data for all of the restrictions were taken in the same manner, using the same procedure as that used for the tube flow data with the additional measurement of the differential pressure across the restriction. The pressure drop across the restriction was then determined by two methods, by direct measurement and by the difference of the total transfer line pressure drop with and without the restriction. During several of the tests the differential pressure measurement across the restriction was inoperative because of leakage. Comparison of the data for the remainder of the test showed that the direct difference measurement was 5 to 10 percent higher than the value obtained by the total line pressure drop. The values determined by the total line pressure drop were more consistent and were available for all tests, therefore, these data were used.

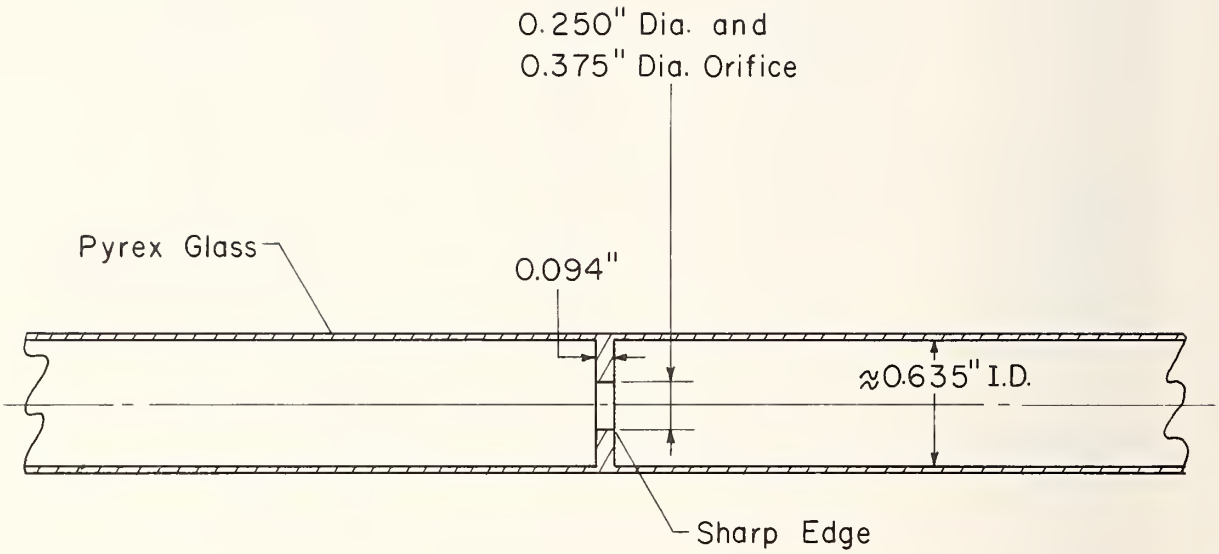

Figure 14. Sharp Edged Orifice

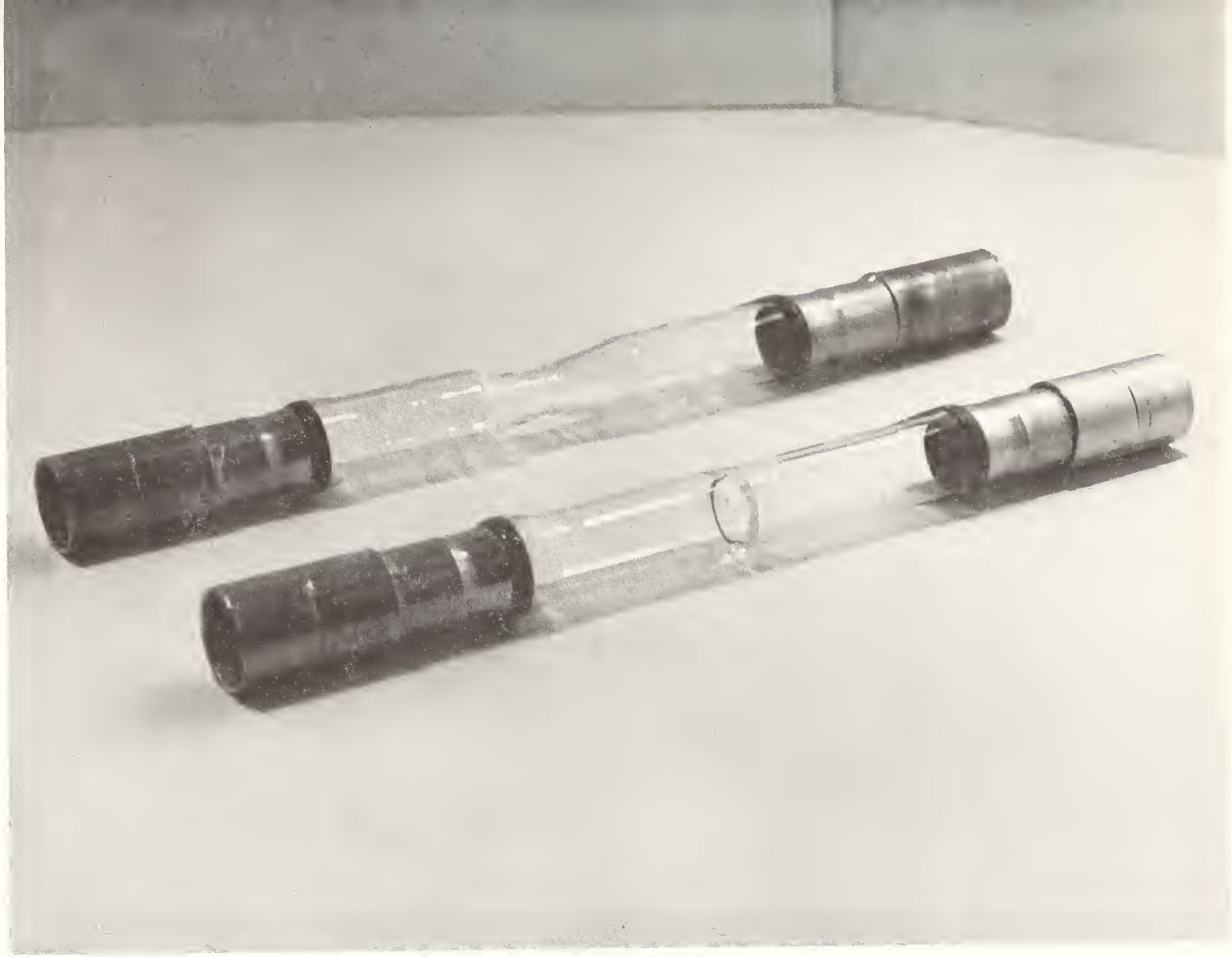

Figure 15. 1/4" Orifice and Venturi Tube

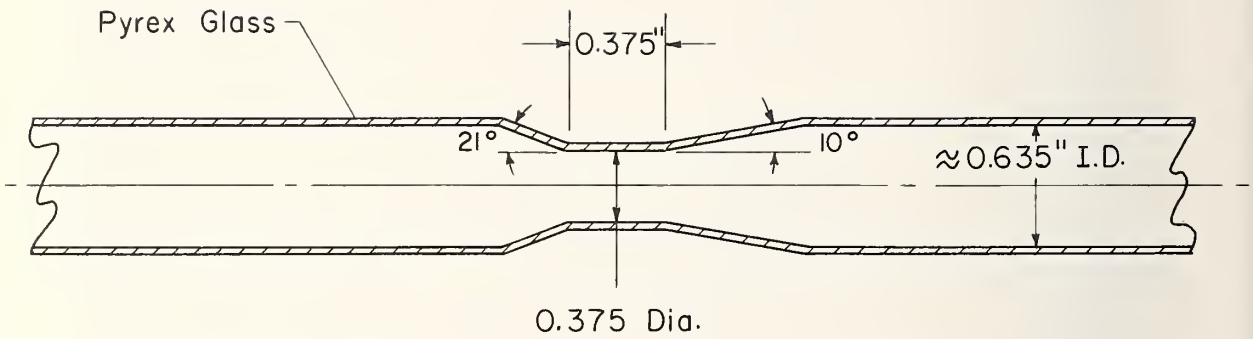

Figure 16. Herschel Type Venturi Tube

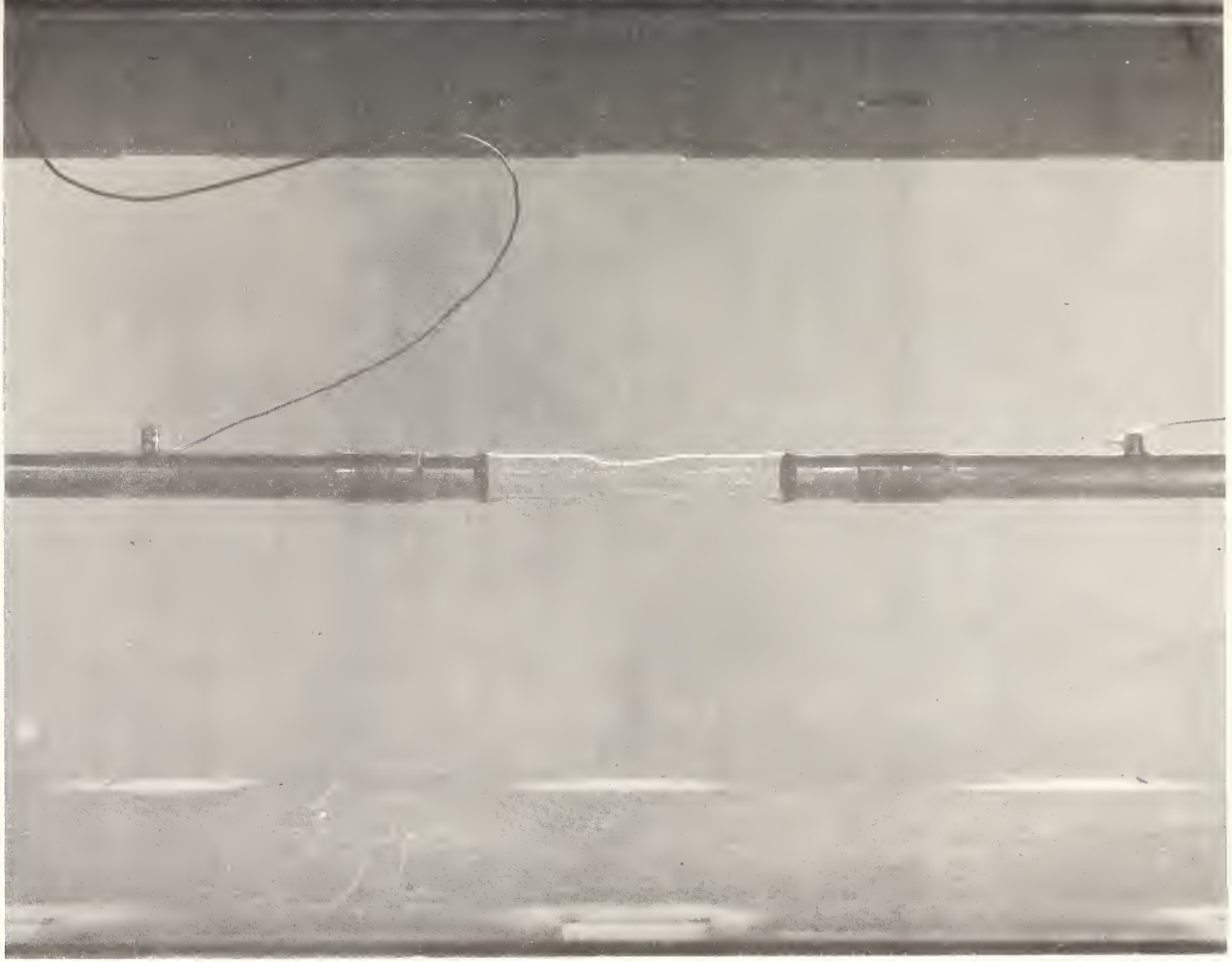

Figure 17. Venturi Tube in Transparent Section

The data for the globe valve were reduced using the conventional resistance coefficient. This coefficient is defined as follows:

$$K = \frac{2 \cdot g_c \cdot \Delta P}{\rho V^2}$$

where ΔP is the differential pressure, g_c is the gravitation constant, ρ is the fluid density, and V is the average fluid velocity in the upstream pipe. The values determined for K with the valve 1/4 inch (0.64 cm) open are shown in figure 18. The values for K with the valve full open are shown in figure 19. The K values for the 1/4 inch (0.64 cm) open valve appear to drop with increased solid fraction. This trend is reversed in the full open valve. The resistance coefficient for the fully opened valve is lower than that predicted by Crane Company [1960], which is approximately 9 for a valve with an inside diameter of 0.6 inch (1.52 cm).

The data for the orifices are plotted directly showing overall pressure drop versus mass flow rate. For comparison purposes the predicted values of pressure drop versus flow rate based on the ASME Power Test Code [1959a] are shown for triple-point liquid and for a fluid having the density of 0.5 solid fraction slush. The equation used for the predicted curves is

$$\omega = \frac{K\pi d^2}{4} \sqrt{2 g_c \rho \Delta P},$$

where ω is the mass flow rate, d is the orifice diameter, g_c is the gravitational constant, ρ is the fluid density, ΔP is the differential pressure between the point upstream of the plate to the vena contracta of the fluid, and K is the orifice coefficient including the velocity of approach factor for vena contracta pressure taps. The pressure loss

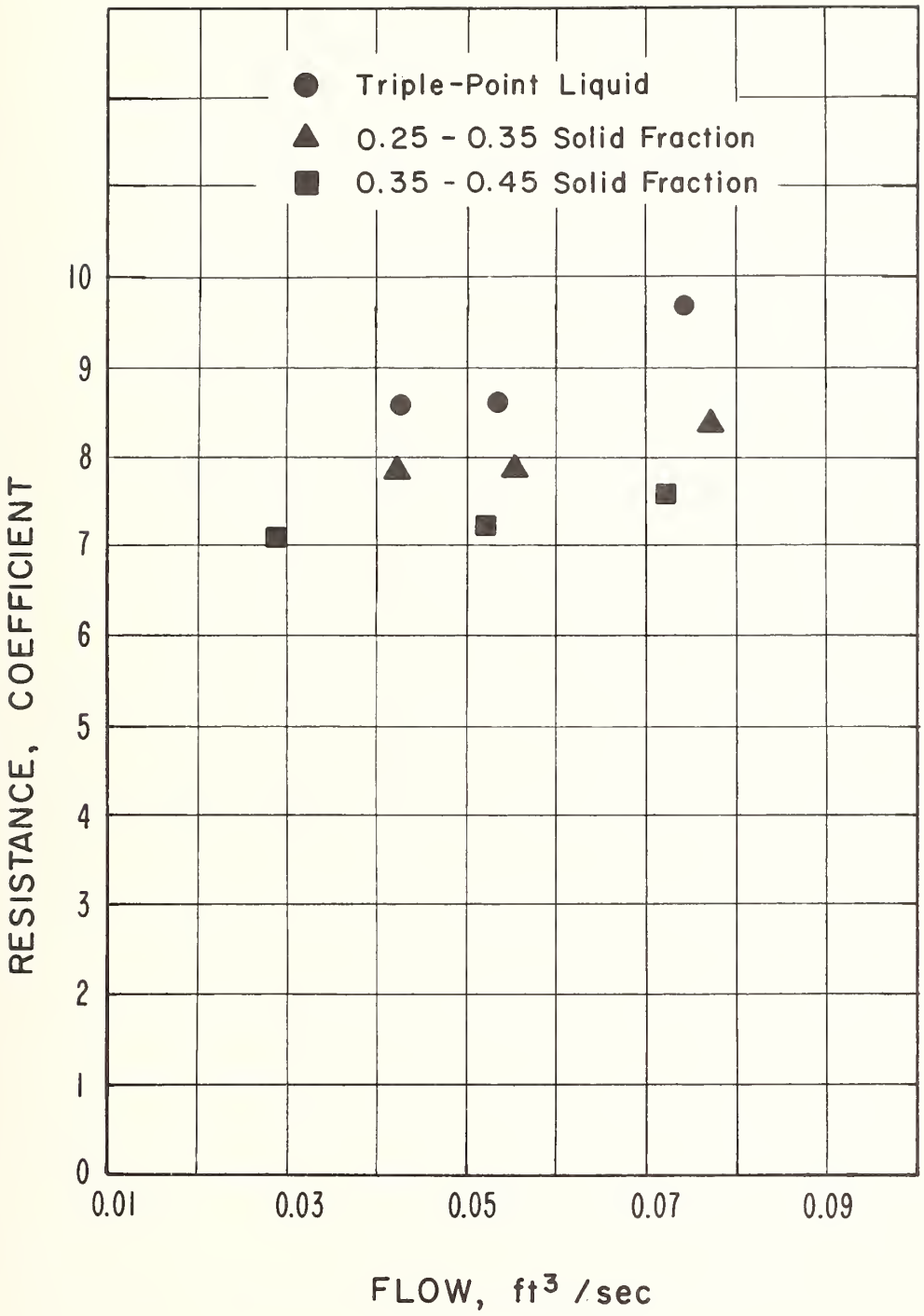

Figure 18. Slush Hydrogen Flow thru a Globe Valve 1/4" Open

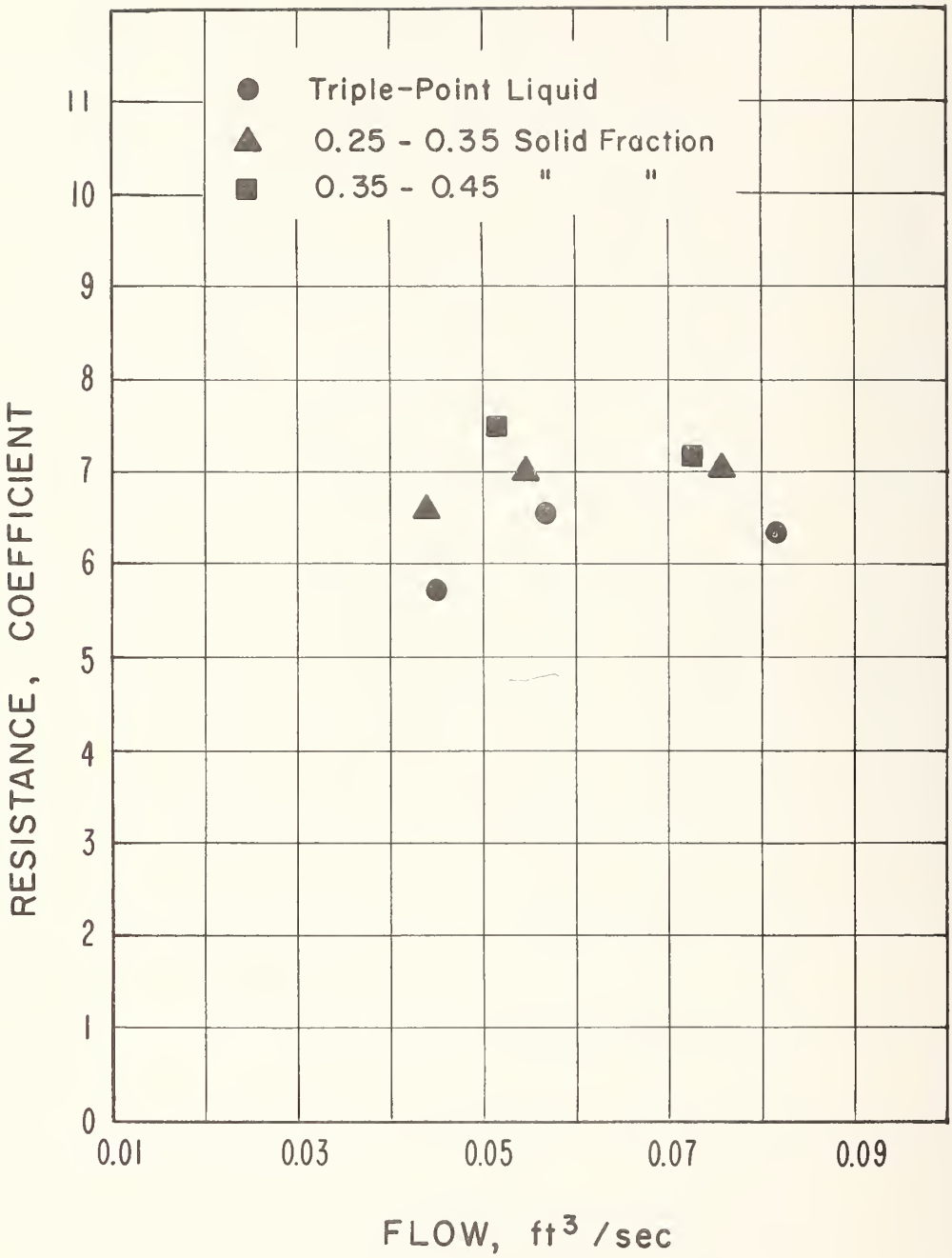

Figure 19. Slush Hydrogen Flow thru a Globe Valve Full Open

across the orifice was calculated from the maximum differential pressure determined from the above equation and the curve of pressure loss for orifice plates given in the publication, Fluid Meters [ASME, 1959b].

In some of the tests, the minimum pressure in the orifice reached the triple-point pressure and resulted in cavitation downstream of the orifice. From the orifice equation, the point of cavitation can be predicted for a given upstream pressure. If thermal equilibrium is assumed, the flow rate should remain constant after this predicted point is reached. Points of maximum predicted flow rates were determined for the values of upstream pressure used in the experiment, 13.6 psia (9.38 N/cm^2) and 15.6 psia (10.76 N/cm^2). These predicted pressures and flow rates are shown in figure 20 for the 1/4-inch (0.64 cm) diameter orifice along with the actual data points. The overall orifice pressure drop was less than predicted by using the K for vena contracta taps and the total pressure loss curve given in Fluid Meters [ASME, 1959b]. However, the cavitating flow for the upstream pressure used does display good correspondence with the predicted values, indicating that the data are in agreement with the orifice coefficient but not with the overall loss predicted.

A line has been drawn through the noncavitating data points demonstrating that the curve is parallel to the predicted curve, thus indicating that the orifice flow does behave as an orifice flowing liquid but experiences a lower pressure loss than predicted. A single aged slush data point is also shown. The curve in Fluid Meters [ASME, 1959b] predicts an overall pressure loss of approximately 80 percent for the 1/4-inch (0.64 cm) orifice and the data show a loss of approximately 50 percent.

The data for the 3/8-inch (0.95 cm) diameter orifice were treated in the same manner as the 1/4-inch (0.64 cm) diameter orifice data.

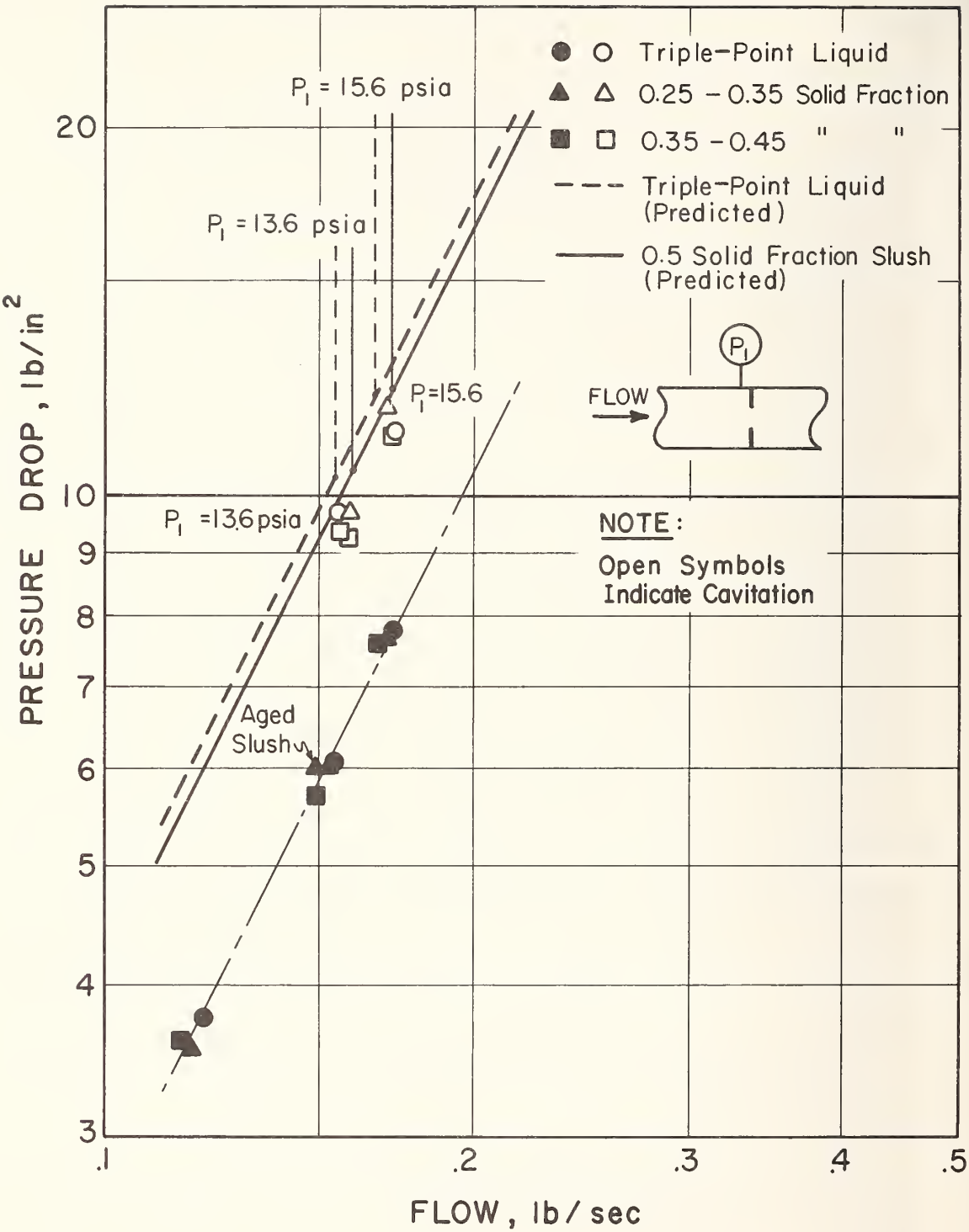

Figure 20. Slush Hydrogen Flow thru a 1/4" Diameter Orifice

These data are shown in figure 21. Figure 22 shows slush flowing through the 3/8-inch (0.95 cm) orifice. The inside diameter of the 3/8-inch (0.95 cm) hole is visible as two very faint light lines in the dark orifice area. The very center of the orifice is transparent as solid particles can be seen passing through the orifice. The flow velocity upstream of the orifice was approximately 1.5 ft/sec (0.46 m/s) and the aforementioned solid gradient is visible. Turbulence on the downstream side disperses the solids uniformly over the diameter of the tube.

Fewer data were taken with this orifice and just one set of data was taken during cavitation. This was with an upstream pressure of 10.6 psia (7.31 N/cm^2). As is shown in the figure, the data for overall pressure loss and the cavitating flow are not in agreement with the predicted values. The orifice coefficient would need to be approximately 0.75 instead of 0.65, the predicted value, to agree with the cavitating data and the overall pressure loss would then need to be 33 percent of the maximum pressure drop instead of the 66 percent given in Fluid Meters [ASME, 1959b] to agree with the noncavitating pressure loss.

The venturi data are also plotted directly showing overall pressure loss versus mass flow rate. These data are shown in figure 23. Also shown on the curve is the predicted pressure drop versus flow rate which was determined from the general hydraulic equation and the total pressure loss curve. The hydraulic equation is the same as that used for the orifice except that the flow coefficient for a Herschel venturi was used instead of the orifice coefficient. The flow coefficient and the total pressure loss for the Herschel venturi are both taken from Fluid Meters [ASME 1959b]. The data in general fit the predicted values. No cavitation data were taken; however, at the high flow rates the pressure in the venturi throat was very near triple-point pressure.

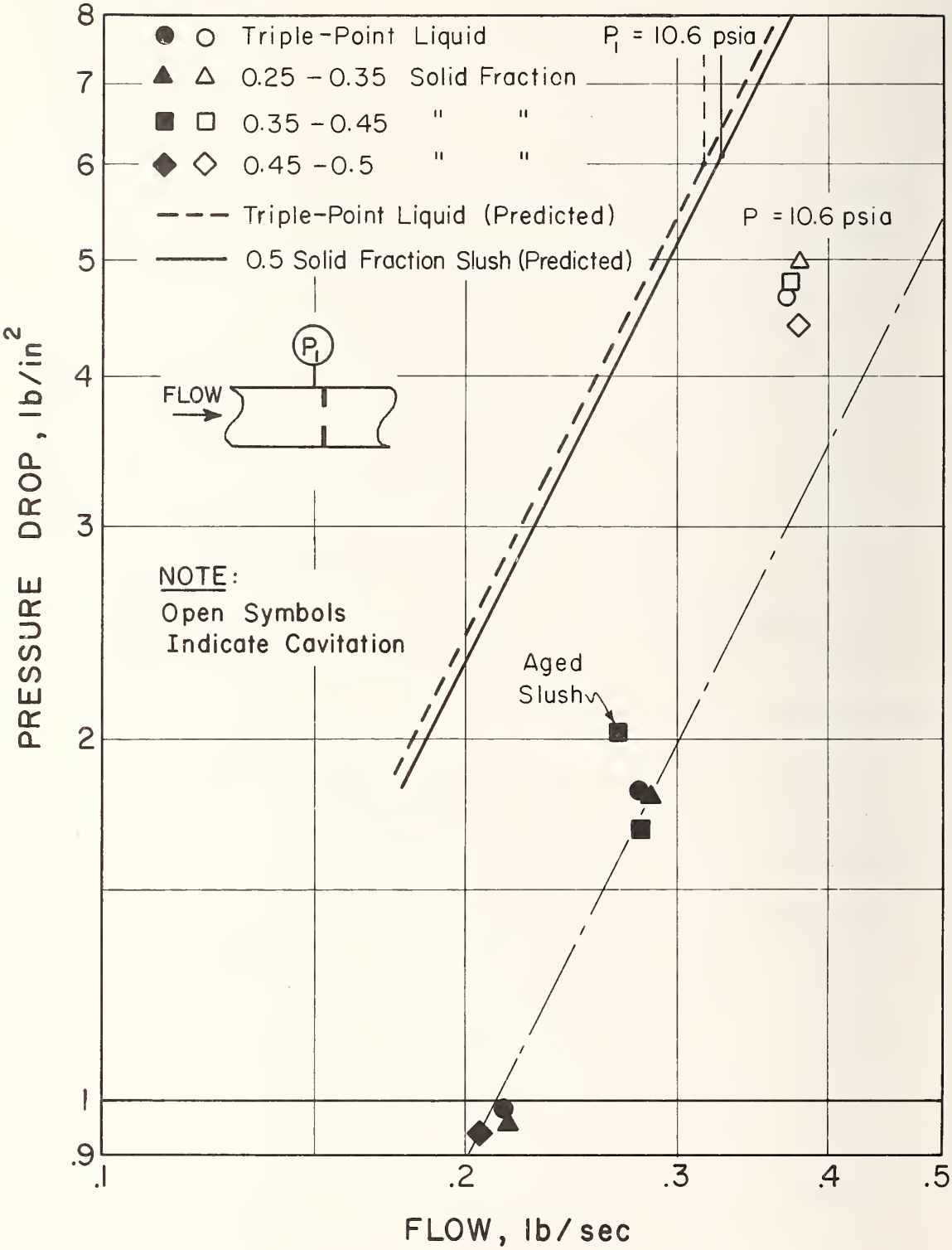

Figure 21. Slush Hydrogen Flow thru a 3/8" Diameter Orifice

Figure 22. Slush Hydrogen Flowing thru a 3/8" Diameter Orifice

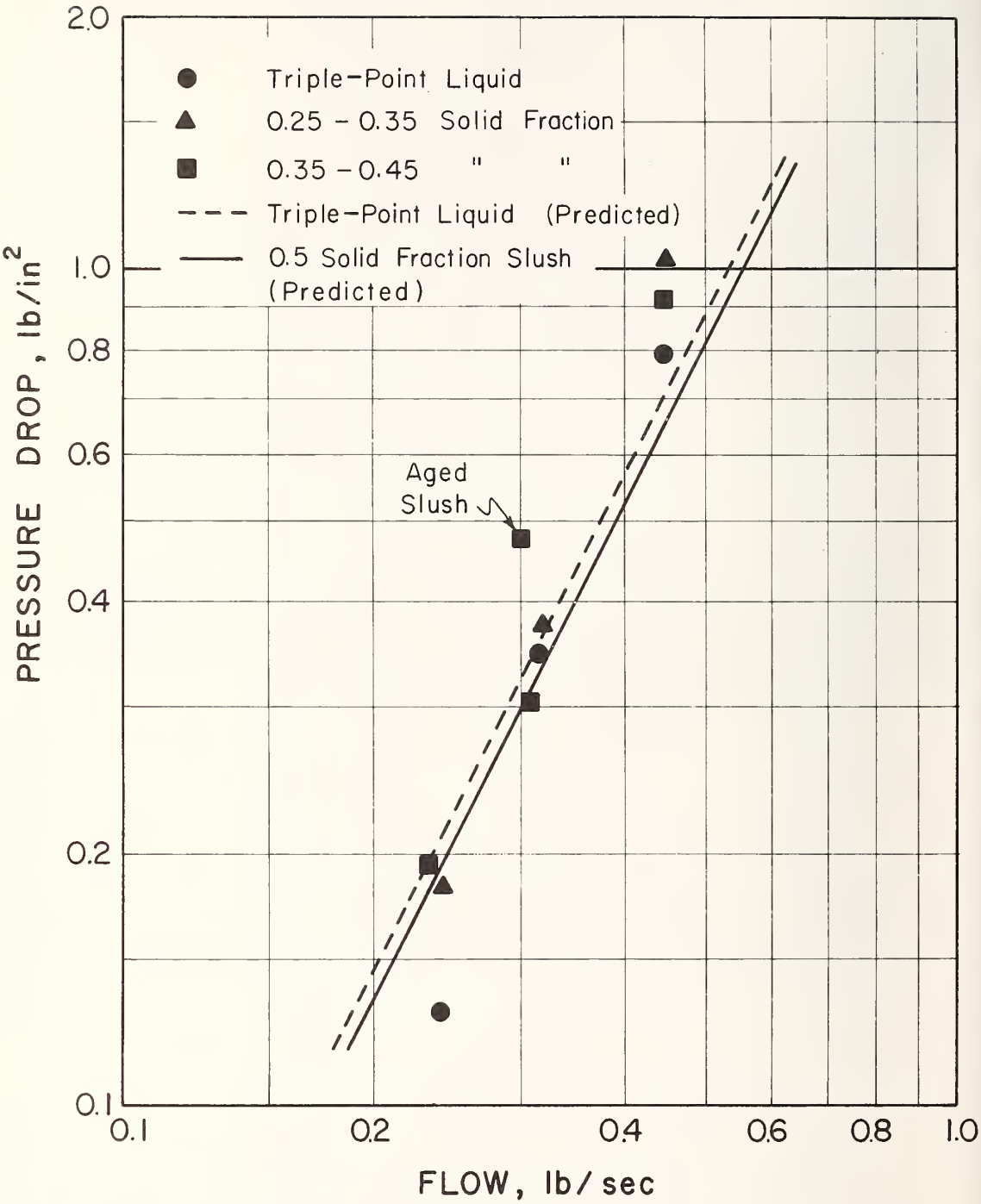

Figure 23. Slush Hydrogen Flow thru a Venturi

5.3 Discussion and Conclusions

The resistance coefficients for the valve compare favorably with that given by the Crane Company [1960]. The data values are slightly lower. Internal geometry of valves differ with different brands and variation in the coefficient is not unexpected. Also, the uncertainty in the pressure loss data with and without restrictions may account for part of the difference in the valve resistance coefficients as well as the orifice and venturi coefficients. The indication that slush flows with less resistance than triple-point liquid through the valve when 1/4 inch (0.64 cm) open is consistent with the data for smooth pipe flow; however, with the valve full open this trend was reversed. The trends indicated may be due to data scatter. No indication of plugging of the valve was observed.

Both the 3/8-inch (0.95 cm) and the 1/4-inch (0.64 cm) diameter orifices flowed slush as freely as triple-point liquid. The overall pressure losses of the orifices were the same with slush of solid fractions to 0.45 as with triple-point liquid. The fact that neither orifice completely followed the predicted coefficients and pressure losses may be attributed to the geometry of the glass tube upstream of the orifice plate and to the roughness in the orifice edge due to small chips. The data are consistent, indicating that orifices could be used as volume flowmeters in slush if they were calibrated.

The data for the Herschel venturi appears to have significant scatter, but the overall pressure loss in the venturi is small. The data scatter is no greater than would be expected with the pressure measureing system used. The greater deviation of data from the predicted curve at the highest flow rates may be due to some localized cavitation in the venturi throat.

The conclusions reached from the results of the restriction work reported here are:

(1) slush of mass solid fractions to 0.5 will flow through restrictions with openings as small as 1/4 inch (0.64 cm) as readily as triple-point liquid,

(2) pressure losses in restrictions flowing slush solid fractions to 0.5 are essentially the same as with triple-point liquid, and

(3) there are no unexpected behavior patterns in flowing slush through restrictions such as valves, orifices, and venturi tubes.

6. Slush Hydrogen Pumping Characteristics

The pumping characteristics of slush hydrogen were investigated using a centrifugal type liquid hydrogen pump. NBS Technical Note 364 [Daney, et al., 1968] describes these tests and the results in detail; therefore, only a brief summary of each is given here.

The pump was a modified* Saturn S IV-B centrifugal type chill-down pump with a design specific speed of approximately 2,200. Impeller diameter was 2.8 inches (7.1 cm). At 1100 rpm it had a nominal flow rate of 135 gpm (8520 cm^3/s) at 7 psi (48 K N/m^2) developed head. Performance tests from 8,000 to 19,000 rpm and cavitation tests at 11,000 and 14,000 rpm were made. The slush solid fraction varied from 0.19 to 0.55. As predicted by theory, the developed head for liquid and slush hydrogen is the same when the difference in density is considered. The pump efficiency, cavitation constant, and net positive suction head requirements are also the same for slush hydrogen and triple-point

* Modification by NASA-George C. Marshall Space Flight Center.

liquid. The operating time with slush hydrogen was 34 minutes out of a total running time of 79 minutes with both liquid and slush. The pump components showed no wear over that expected from operation in liquid hydrogen.

7. Slush Hydrogen Gel

The desirable properties of both slush hydrogen and gelled liquid hydrogen are well known. The increased density and heat capacity of slush hydrogen make it a desirable potential rocket fuel because more hydrogen can be stored in fuel tanks for far longer periods with greatly reduced venting losses. The finite yield strength of gelled liquid hydrogen minimizes tank sloshing; and in some cases, evaporation losses from gelled hydrogen are reported to be reduced by a factor of 2 to 3 [Kartluke, et al., 1964].

By combining slush and gelation production techniques, a unique product, gelled slush hydrogen, results. This mixture, which combines the desirable properties of both slush and gelled hydrogen, has a high density and heat capacity combined with the semi-solid structure of a thixotropic gel.

Other experimentalists have worked with gelling liquid hydrogen and other propellants [McKinney and Tarpley, 1966; Aerojet-General, 1967a, 1967b], but no previous work on gelled slush hydrogen has been reported.

A study of gelled slush hydrogen was made by Ajit Singh Rapial of the Indian Bhabha Atomic Energy Research Center while at the NBS Cryogenics Division as a guest worker. The project was sponsored by NASA-MSFC as part of Contract H-25490A and personal support was provided by the International Atomic Energy Agency Fellowship Program, and the Chemical Engineering Department, University of Colorado.

The following is a summary of this work which was accepted as the thesis for an M. S. Chemical Engineering degree at the University of Colorado.

7. 1 Theory

In discussing a model for gelled slush hydrogen, it is first important to discuss the properties of gelled liquid hydrogen. Unlike water gelatines which are characterized by long-chain molecules partially soluble in water, hydrogen must be gelled by surface interaction with a large number of finely divided particles. The low associative property of hydrogen, its low ionization state, and its nonsolvent property with respect to most substances, exclude gelling by formation of a macromolecular structure. Since hydrogen gelation is a surface interaction phenomenon, almost any finely divided powder is a suitable gelling agent. As a first approximation, the number of particles per unit volume of gel is a constant, so that the finer the particles, the lower the weight of gelling agent required.

In gelling slush, the solid hydrogen particles are formed before the addition of the gelling agent, so only the liquid portion must be gelled. Thus, a major advantage of gelled slush over gelled liquid becomes apparent, viz., the weight of gelling agent is reduced proportionately as the liquid fraction is reduced. These two principles, 1) that the number of particles per unit volume is a constant, and 2) that only the liquid portion is gelled, are expressed by Rapial [1969] as

$$Y_{S\ell} = \frac{Y_l(1 - X)}{\frac{\rho_{\ell t}}{\rho_{\ell b}}(1 - Y_l) + Y_l(1 - X)} \qquad (1)$$

where $Y_{S\ell}$ is the weight fraction of gelling agent in the gelled slush, X is the weight fraction of solid hydrogen in the slush, Y_l is the weight

fraction of gelling agent in a liquid gel for a given gel consistency and a saturated liquid density $\rho_{\ell b}$, and $\rho_{\ell t}$ is the density of the triple-point liquid.

Ideally, the gelling agent should have a high fuel value if the gel is to be used as a rocket fuel. However, in these tests Cab-O-Sil® (a commercially available pyrogenic silica with a 0.005 to 0.01 μm particle size) was used because it is readily available and has been used by previous investigators.

7.2 Apparatus

Figure 24 schematically shows the apparatus used in the experiments. The outer 6-inch (15.2 cm) shielding dewar provides a triple-point thermal environment for the two 4-inch (10.2 cm) I.D. inner vessels. The lower 4-inch (10.2 cm) I.D. vessel is charged with the gelling agent prior to assembly. Slush hydrogen or nitrogen is produced in the upper slush generator by the freeze-thaw method. When the gelling agent in the lower dewar has cooled to the triple-point temperature, the valve separating the two chambers is opened; the slush is transferred into the gelling agent, and the mixture is stirred until homogeneous. Measurement of the consistency or yield strength of the gel is made by lowering, in succession, the mercury filled plumb bobs onto the gel surfaces. The gel is then characterized by which plumb bob sinks and which does not sink. As the test proceeds, solid hydrogen melts and weakens the gel so that successively lighter plumb bobs penetrate the surface. Similar

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® Cabot Corporation trademark. Precise specifications in this paper of the pyrogenic silica employed has been necessary to make the results of the work sufficiently meaningful. Identification of the silica or its manufacturer by the National Bureau of Standards in no way implies a recommendation or an endorsement by the Bureau.

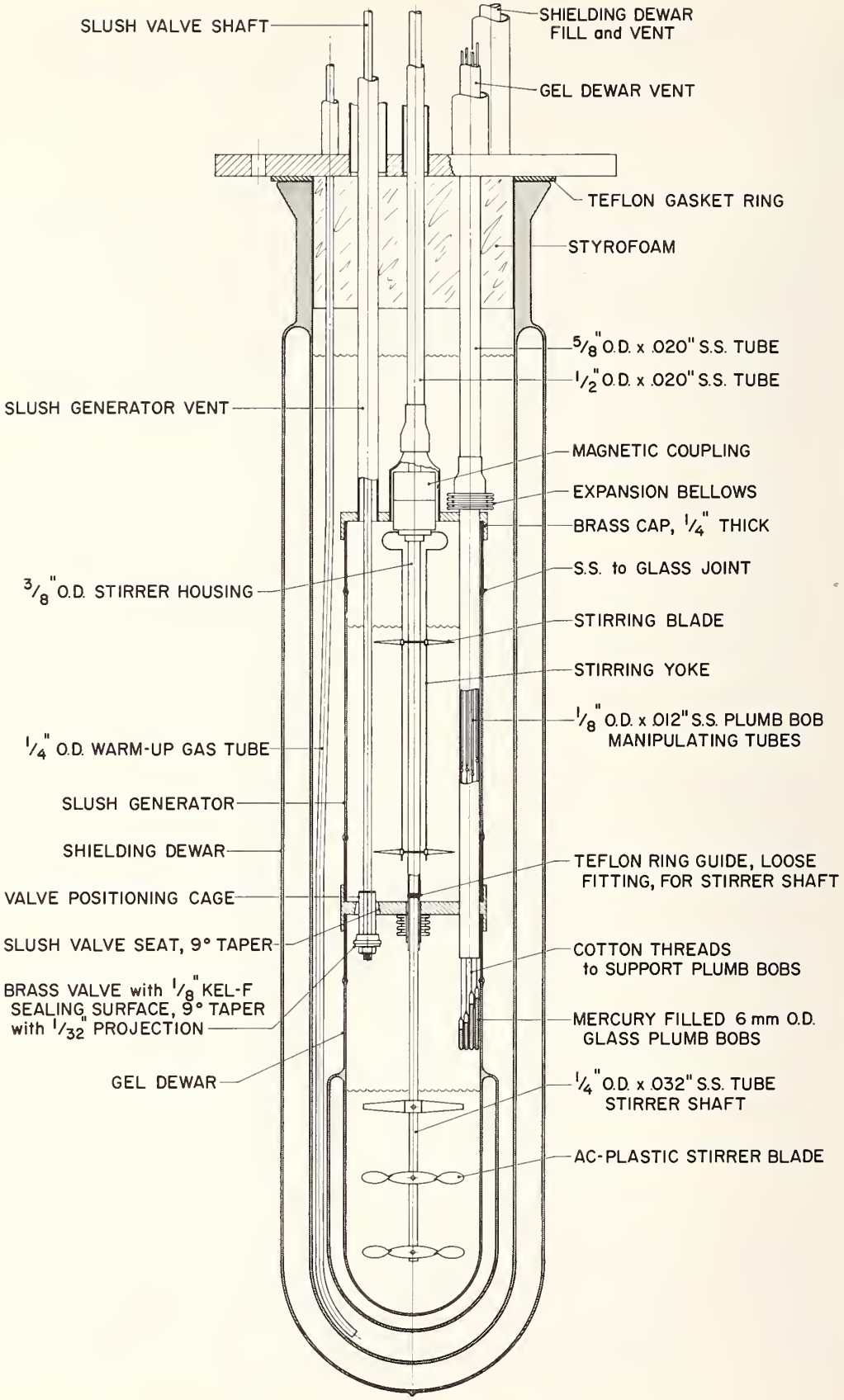

Figure 24. Gelled Slush Hydrogen Apparatus

plumb bobs have been used by Mc Kinney [1968] and the relationship between plumb bob weight and gel yield strength is given below.

Yield Strength as a Function of Plumb Bob Weight

Plumb bob weight supported by gel grams	Plumb bob weight not supported by gel grams	Has yield strength of N/m^2
--	2.3	<20.0
2.3	5	20.0 - 34.5
5	7.5	34.5 - 47.8
7.5	10	47.8 - 61.1
10	--	>61.1

7.3 Experimental Results

Both nitrogen and hydrogen were gelled in the liquid and slush states. Cabo-O-Sil was used as the gelling agent in all cases. Figure 25 is a plot of the liquid nitrogen data in the form of weight-bearing capacity of the gel versus weight percent of Cab-O-Sil in the gel. Each data point is the average of 4 experiments. Figure 26 is a plot of the nitrogen slush data in the same form. The boiling-point liquid line is taken from figure 25 and the remaining lines are calculated using equation (1) and the boiling-point line.

The results of the liquid hydrogen experiments are given in figure 27, and the slush hydrogen experiments in figure 28. Again, the liquid curve (fig. 27) and equation (1) are used to predict the triple-point liquid and slush consistencies in figure 28.

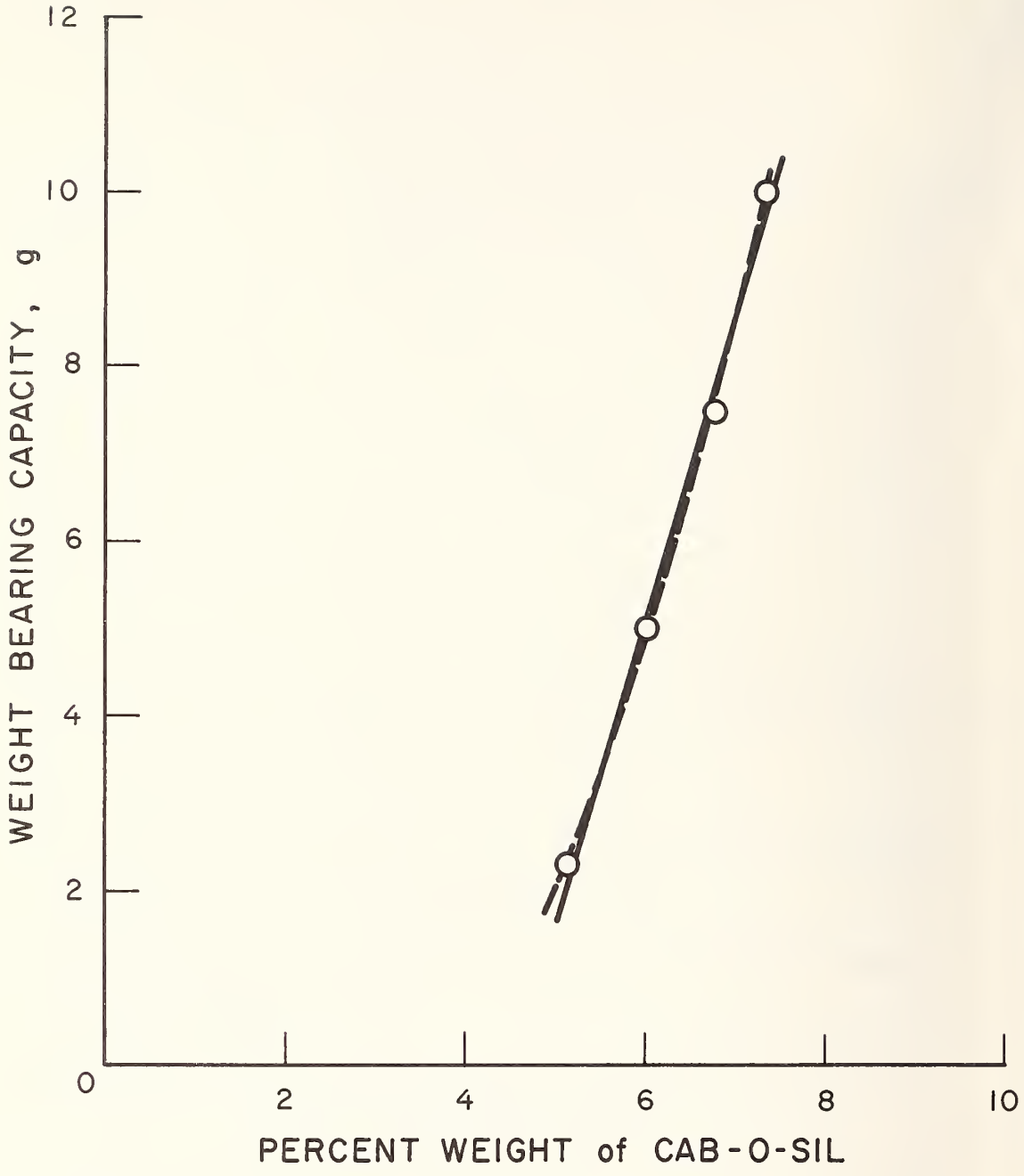

Figure 25. Weight-bearing Capacity of Liquid Nitrogen Gel

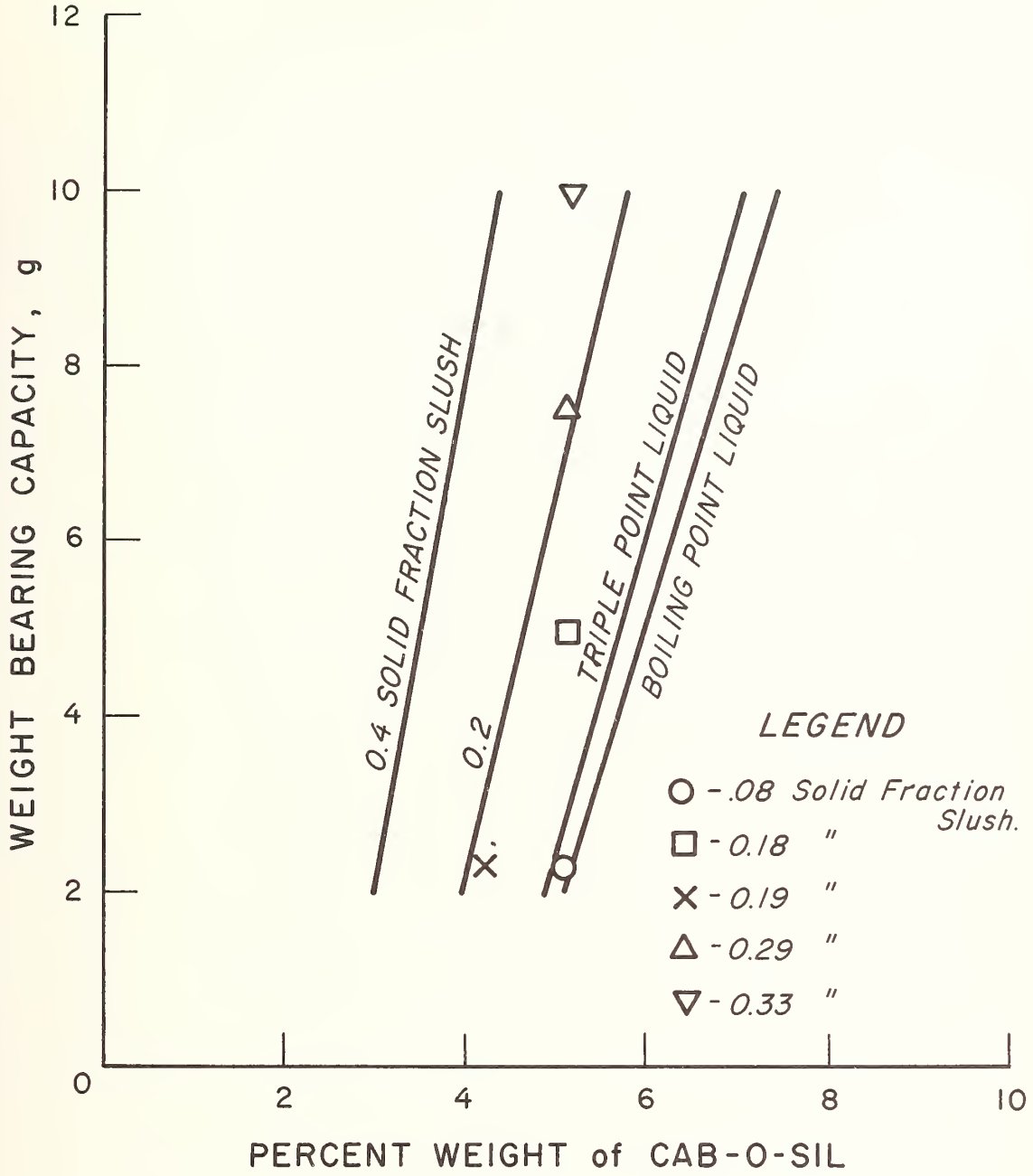

Figure 26. Slush Nitrogen Gel Characterization

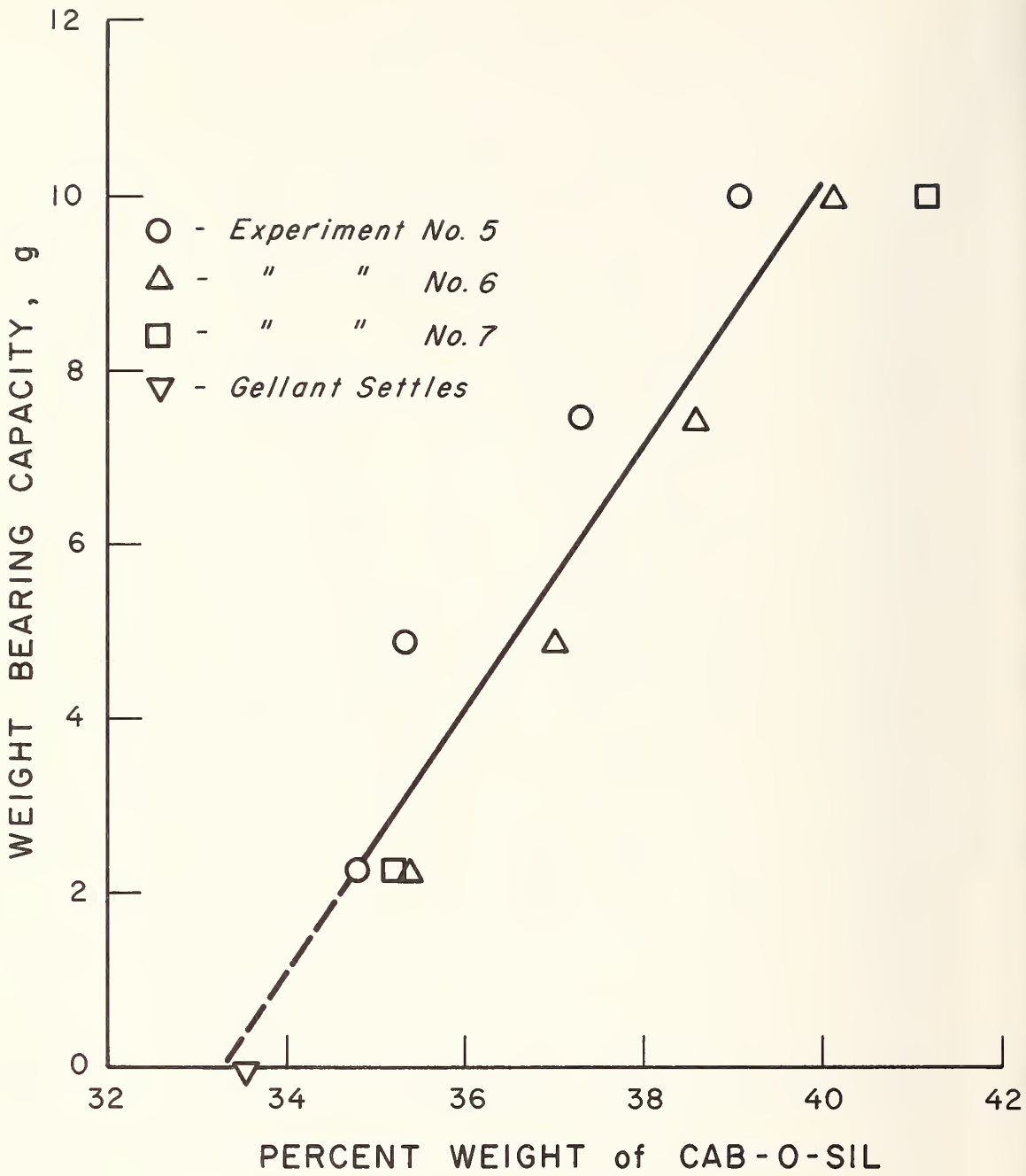

Figure 27. Weight-bearing Capacity of Liquid Hydrogen Gel

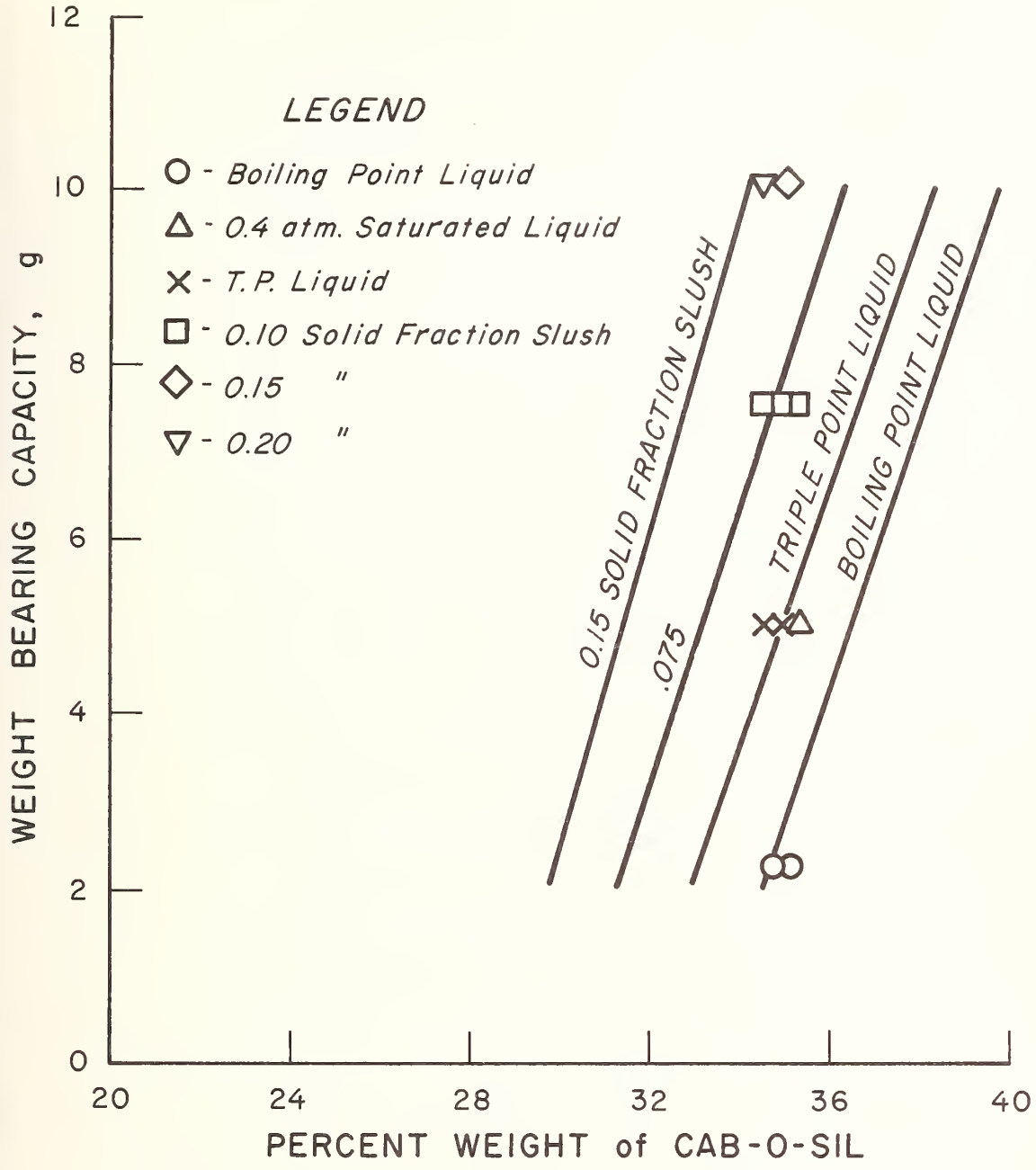

Figure 28. Slush Hydrogen Gel Characterization

7.4 Conclusions

These experiments have demonstrated that slush hydrogen can be gelled, and that within the limited accuracy of the measurements, equation (1) adequately describes the relationship between the weight percent of gelling agent and slush solid fraction. Thus, the amount of gelling agent required to produce a specific yield stress is proportional to the liquid volume fraction of the slush hydrogen. Although Cab-O-Sil was used as the gelling agent in this work, the reduction in quantity of gelling agent expressed by equation (1) should apply to any gelling agent For gelled rocket fuels, this reduction in the amount of gelling agent required by slush hydrogen would result in less fuel degradation due to the presence of gelling agents with lower specific impulses than hydrogen.

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