RESEARCH OF PRODUCTION TECHNIQUES FOR OBTAINING OVER 50% SOLID IN SLUSH HYDROGEN

L. R. Niendorf
O. J. Noichl

Linde Division
Union Carbide Corporation

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Air Force Aero Propulsion Laboratory
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FOREWORD

This is the final report of research performed by Union Carbide Corporation, Linde Division under Contract No. AF 33(615)-1357. This contract was initiated under Project No. 8169, Task No. 816901. The contract work was performed under the direction of the Air Force Aero Propulsion Laboratory, Research and Technology Division. Mr. C. W. Elrod, Jr. was the Project Engineer for RTD.

The major contract activities were performed during the period from February 17, 1964 to December 17, 1964 under the supervision of Mr. L. R. Niendorf. He was assisted by Messrs. O. J. Noichl and R. L. Webb. Additional assistance and contributions were rendered by Dr. H. M. Long and other Linde Division consultants. Mr. L. R. Niendorf was also the Contract Administrator for this project.

ABSTRACT

Liquid-solid mixtures of hydrogen (slush hydrogen) were produced by vacuum pumping, gaseous helium injection with vacuum pumping, and by cooling with liquid helium in a low heat leak apparatus that permitted visual observation of the experiments through a periscopic device. The slush hydrogen produced by these methods was compressed to a maximum pressure of 1.25 psi and the resulting solid content (slush quality) was determined by calculation methods. For the compressed slush hydrogen produced by vacuum pumping, the quality was generally in the 65 to 85 per cent range, while the compressed quality produced by the helium injection with the vacuum pumping method ranged from 55 to 85 per cent depending upon solid formation procedure. The compressed quality for slush hydrogen produced by cooling with liquid helium generally ranged from 65 tc 95 per cent depending on the method of freezing and the time elapsed during the compression. A number of characteristics were noted which varied with the production techniques employed.

This technical report has been reviewed and is approved.

B. C. Dunnam
Branch Chief
Fuels and Lubricants
A.F. Aero Propulsion Laboratory
Research and Technology Division

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SECTION 1

INTRODUCTION

The work described in this report on the research of production techniques for obtaining over 50 per cent solid in slush hydrogen results from recommendations made during previous Linde Division work on Contract No. 33(657)-10248, Theoretical, Experimental, and Analytical Examination of Subcooled and Solid Hydrogen. During this previous contract, it was determined that liquid-solid hydrogen mixtures could be produced by vacuum pumping methods having solid contents ranging up to approximately 55 weight per cent. In order to further increase the density and heat capacity of slush hydrogen, it was the object of this contract to investigate production methods and techniques to produce higher quality slush hydrogen.

Under the terms of the contract with RTD, the following objectives were established:

1. To produce slush hydrogen by the following methods and determine the resulting slush hydrogen quality.

    a. Vacuum pumping followed by mechanical compression.

    b. Helium gas injection followed by vacuum pumping.

    c. Liquid helium freezing of hydrogen followed by mechanical compression.

2. To learn as much as possible about the characteristics of liquid-solid hydrogen mixtures consistent with the visual observation capability, which is limited due to apparatus heat leak considerations.

3. To gain more feeling of how a proposed large-scale production system employing one or more of the experimentally examined production methods would operate.

4. To determine problem areas associated with slush hydrogen production requiring work, which is beyond the scope of this contract because of time and funding considerations.

The contract work performed in pursuit of these objectives and the results of this work are recorded in this document.

Manuscript released by authors 17 February 1965 for publication as an RTD Technical Report.

SECTION 2

SUMMARY

Under the provisions of this contract, methods for producing slush hydrogen with a solid content (slush quality) in excess of 50 per cent were experimentally investigated. Qualitative observations of the slush hydrogen were made in addition to the determination of the slush quality produced.

The basic experimental apparatus was available from Contract AF 33(657)-10248. This apparatus was modified to include a metal test reservoir with a liquid helium heat exchanger coil. Heater wires were provided to allow a variation of the heat leak during the production runs. For system reliability and safety only the periscope guard dewar was fabricated from glass. Observation of the slush production was accomplished with a periscope-type viewing device.

The experimental apparatus can initially be filled with 40 liters of liquid hydrogen. Vacuum pumping rates up to 5 cfm (NTP) could be provided, and the heat leak could be varied from 0.252 to 2.8 cfm (NTP) of vaporized triple point liquid hydrogen.

Slush hydrogen was produced by three basic experimental methods which included: vacuum pumping followed by compression, helium gas injection followed by vacuum pumping and compression, and liquid helium freezing followed by compression.

The compressed slush quality produced by the vacuum pumping method generally ranged from 65 to 85 per cent. A correlation between the slush quality produced and the heat leak or pumping rate was not found. Solid hydrogen particles could not be distinguished in either the settled or compressed slush. However, while breaking the crust, both needle- and flake-shaped solid particles were observed settling through the triple point liquid.

Solid tubes were formed in triple point liquid hydrogen and in settled slush with the injection of either warm or cold helium gas. The tubes were grown into solid columns by continued vacuum pumping. The compressed slush quality resulting from compressing the solids of the columns ranged from 55 to 70 per cent. Compressed slush qualities ranging from 55 to 85 per cent were produced by compressing columns formed in settled slush.

Slush hydrogen was produced by direct and indirect heat exchange with liquid helium. Direct liquid helium injection produced a bulb-shaped solid formation on the end of the helium transfer line. Solid tubes extended from this formation to a solid crust that was formed on the liquid surface. Indirect liquid helium heat exchange produced a solid layer on the test reservoir wall and solid crust wafers bridging the diameter of the test

reservoir above the receding triple point liquid level. Breaking the solids formed by the indirect heat exchange method produced transparent plate-shaped solid particles. The compression of these particles resulted in compressed slush qualities ranging from 65 to 95 per cent. The settled slush produced by breaking the solid forming during direct heat exchange included some large chunks of solid. The compressed slush qualities produced by this method were in the same range as for the indirect heat exchange method.

Since the accuracy of the slush hydrogen quality produced is not of high integrity because of inherent measurement problems which are discussed in this report, the data presented should be considered as a range in which to proceed with more quantitative investigations when an accurate slush hydrogen quality measurement device has been developed.

Photographs could not be taken through the periscope-type viewing device used for visual observations of the slush hydrogen production process. Artists conceptions of the production process are therefore used in some of the figures in this report. Quite naturally, there may be some variation between that which was actually observed and that which is presented in the artist's conception of the observation; however, it is felt that a good representation of the actual observations in presented in this report.

SECTION 3

EXPERIMENTAL EXAMINATION OF

SLUSH HYDROGEN PRODUCTION METHODS

The experimental work under this contract was performed to evaluate three slush hydrogen production methods. The methods experimentally investigated included: vacuum pumping followed by mechanical compression; helium gas injection followed by vacuum pumping; and liquid helium freezing followed by compression. The object of this experimental work was to determine the slush hydrogen quality which can be produced by each of the production methods and also to qualitatively determine the characteristics of the production method and the produced slush hydrogen.

3.1 SCOPE OF THE EXPERIMENTAL PROGRAM

To acquaint the reader with the various slush hydrogen production methods which were experimentally tested under this contract, a brief description of each is presented in the following discussions.

3.1.1 Vacuum Pumping Production Followed by Compression

In the production system which utilizes vacuum pumping followed by mechanical compression, the test reservoir is initially filled with saturated liquid hydrogen. Vacuum pumping is then performed on this hydrogen until slush is produced. The refrigeration for this production method is supplied by evaporating some of the hydrogen to cool the remaining portion. Either the freezing technique, which requires that the crust be broken into particles during vacuum pumping; or the melting technique, which allows the crust to grow downward until all solid is produced, may be employed. When settled slush has been produced (the condition in which the liquid level and solid level coincide), the solids in the mixture are compressed by a porous compactor which allows the triple point liquid to flow through while packing the solids. The realization of high quality in this compressed solid-liquid slush hydrogen mixture is the object of the experimental work.

3.1.2 Helium Gas Injection Followed by Vacuum Pumping and Compression

In the production system using helium gas injection followed by vacuum pumping, helium gas (either warm or cold) is injected directly into triple point liquid hydrogen which has been produced by vacuum pumping. This injection, by producing evaporative cooling, causes solid hydrogen tubes to form in the liquid following the path of the helium bubbles. After a number of solid tubes are formed, the helium injection is stopped and gentle vacuum pumping is started. Under certain conditions the solid tubes will grow into a solid column-like mass; also, the crust on the liquid

surface forms and grows as in the vacuum pumping production. At the conclusion of vacuum pumping, the solids are broken into settled slush which is compressed with a porous compactor as in the vacuum pumping production method.

3.1.3 Liquid Helium Freezing of Hydrogen Followed by Compression

In this production method, the refrigeration for producing solids is provided by the liquid helium, and therefore no hydrogen is lost from the system. This production method can be operated by two methods. One method consists of introducing liquid helium directly into the liquid hydrogen, thus causing vaporization of the helium which cools the hydrogen and produces solids. As the solubility of helium in hydrogen at the triple point is very low, this method should not adversely affect the resulting slush hydrogen product. The other method of using liquid helium is to employ a heat exchanger so that the liquid helium does not come in contact with the hydrogen. In this case solid hydrogen is produced on the heat exchanger and mixed with the triple point liquid hydrogen to produce slush hydrogen. Compression can then be applied as in the previous production methods to produce high quality slush hydrogen.

3.2 APPARATUS AND INSTRUMENTATION

3.2.1 Description of Test Apparatus

The experimental test apparatus used during the conduct of this test program is composed of a guard chamber dewar with insertable test components which are utilized for the various slush hydrogen production methods. The basic guard chamber dewar, which has been modified for this work, was fabricated and utilized during Contract No. AF 33(657)-10248. This dewar is composed of a stainless steel annular liquid hydrogen guard chamber vessel (16 in. I.D. x 18 in. O.D.) suspended inside a vertical cylindrical vacuum casing. Super Insulation was used to allow the liquid hydrogen guard chamber to be installed without the customary liquid nitrogen shielding.

3.2.1.1 Guard Chamber Dewar Modifications

As a result of a critical review of the experimental apparatus utilized during the previous contract work, it was determined that an experimental test system could be designed incorporating additional features offering better reliability and more chance of success. The following features were incorporated in the guard chamber dewar modifications. See Figure 1, which is a schematic of the modified guard chamber dewar.

1. The guard chamber insulation vacuum system is separate and independent of the vacuum utilized for the slush processes. This vacuum space is enclosed by an all-metal system which is welded to insure that the vacuum is maintained at all times. During work on the previous

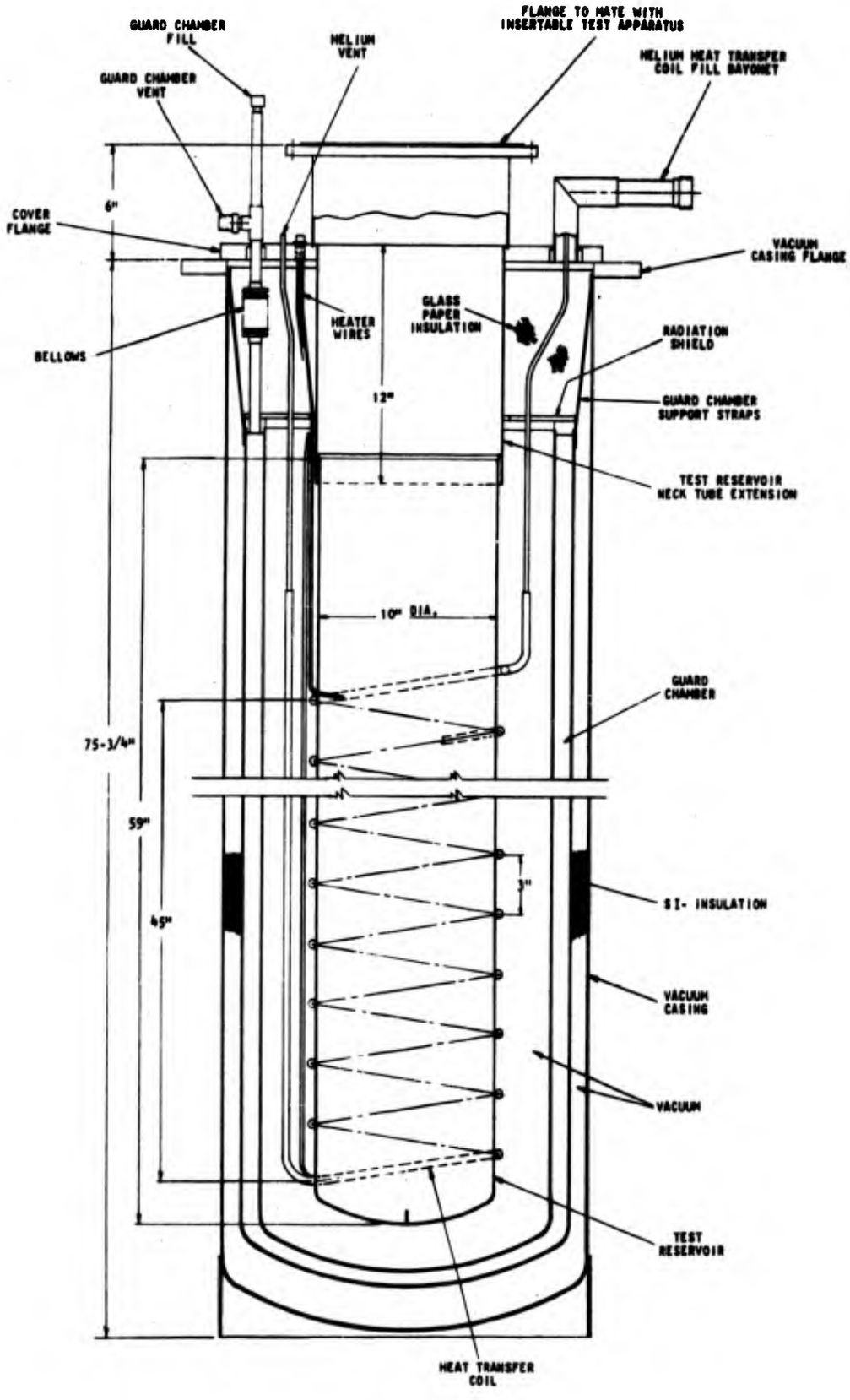

Figure 1. Schematic of Modified Guard Chamber Dewar

contract, the guard chamber insulation vacuum was sealed with elastomers which proved to be not entirely satisfactory as, at times, they became chilled due to rapid production of slush and leaked, causing the experiment to be terminated.

2. The guard chamber fill and vent tube is permanently sealed by welding where it exits from the insulation vacuum space. This tube previously was also sealed with an elastomer which leaked at times during the filling of the guard chamber with liquid hydrogen. An expansion bellows is provided in this tube to accommodate thermal contractions.

3. A metal liquid hydrogen reservoir for slush production is permanently installed in the guard chamber dewar to eliminate the need for glass liquid hydrogen reservoirs. The use of a metal liquid hydrogen reservoir also eliminates the need for any glass-to-metal joints that are subject to reduced temperature. Only one glass-to-metal joint is now required, that being the joint used to seal the glass periscope guard chamber in the production system. This joint, which is in the production system rather than the guard chamber vacuum system, operates at room temperature at all times.

4. Electrical resistance heaters are permanently installed on the metal liquid hydrogen reservoir to provide a variable heat leak to the production system. This feature was incorporated because it was suggested in previous work that slush qualities may vary with different heat leaks and production times for the vacuum pumping production process.

5. A heat exchanger tube for cooling the hydrogen with either liquid or gaseous helium is permanently attached to the metal liquid reservoir within the guard chamber dewar. This type of heat exchanger will allow the entire inner wall of the liquid reservoir to be cooled at one time to produce solids. This heat exchanger can also be used in reverse (by passing warm helium gas through it), in conjunction with the electrical resistance heater, to quickly melt the slush at the conclusion of a production run.

6. A room temperature operation "O" ring flange is provided at the top of the modified guard chamber dewar so that the various production test apparatus associated with each production method can be quickly and easily installed.

Figure 2 shows the assembled components for the dewar modification before the neck tube insulation was applied. The lower portion of the figure shows the copper test reservoir upon which is soldered the copper heat transfer coil; this coil, which is 3/8-inch O.D. x 0.032-in. wall, is coiled around the copper test reservoir and thermally connected to the test reservoir by means of spot brazing and continuous solder fill. To provide for reasonable spacing between the coils and to eliminate cold spots adjacent to the coil on the inside of the wall, it was necessary to specify that

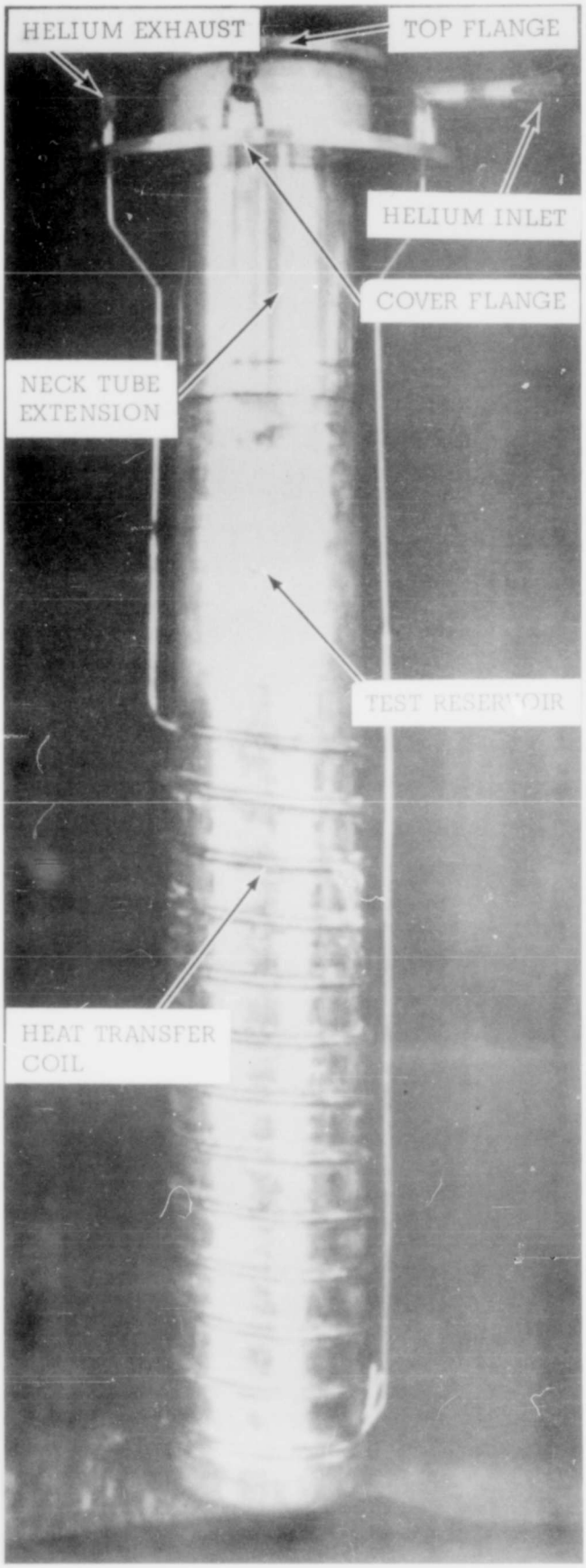

Figure 2. Assembled Components for Dewar Modifications

the test reservoir be fabricated from 14-gauge copper. The upper portion of the figure shows the cover flange which seals the opening of the existing dewar. The test reservoir is supported by a thin-wall stainless steel neck tube extension welded directly to the cover flange. The function of this thin-wall neck tube is to support the reservoir while minimizing the heat leak from the cover flange to the liquid hydrogen in the test reservoir. The helium inlet and exhaust lines, shown on each side of the test reservoir, are thin-wall stainless steel and are brazed to the copper heat transfer coil approximately midway between the neck tube extension and the upper coil.

The inlet to the heat transfer coil is provided by a standard Linde 1/2-inch female bayonet coupling which is mounted by means of extended tubes directly to the cover flange. This coupling accepts the standard Linde vacuum-insulated transfer line which is used to transfer the liquid helium from the storage reservoir to the heat transfer coil.

Figure 3 is a close-up of the lower portion of the test reservoir showing the heat transfer coil and the heater wires. Two independent heater wires were wound on the test reservoir and secured with epoxy. One heater wire was positioned near the upper side of the heat transfer coil, while the other wire was positioned near the lower side as shown in the figure. Each heater wire has the capacity to supply up to 85 watts to the hydrogen in the test reservoir. The heater wires enter the vacuum system through a commercial feedthrough connection which is soldered to the cover flange.

Figure 4 shows the insulation of the neck tube extension. Approximately two inches of glass paper-type insulation was wrapped around the neck tube extension to insulate this portion of the apparatus from the warm unguarded vacuum shell. The upper portion of the heater wires and the transition joint between the stainless neck tube and the copper test reservoir are shown in more detail.

Figure 5 shows the bellows assembly installed on the guard chamber fill and vent line. The bellows assembly was provided to accommodate the contraction caused when the tube becomes cold during the guard chamber venting. The modified radiation shield is shown installed on the top edge of the guard chamber. The cut-out slot on the inside diameter is to allow passage of the heat transfer coil inlet line. Approximately 3/4-inch thickness of Super Insulation disks was applied on top of the radiation shield prior to installation of the insulated test reservoir assembly.

The upper portion of the assembled modified guard chamber dewar is shown in Figure 6. The fill and vent tube is brazed to the cover flange, where it exits, to insure vacuum integrity during fill and normal venting. A tee connection is brazed to the bellows extension tube, the right angle of the tee being used for the venting gas while the straight portion is used for the insertion of a vacuum-insulated transfer line through a quick-connect coupling for filling the guard chamber. A ring flange with an "O"

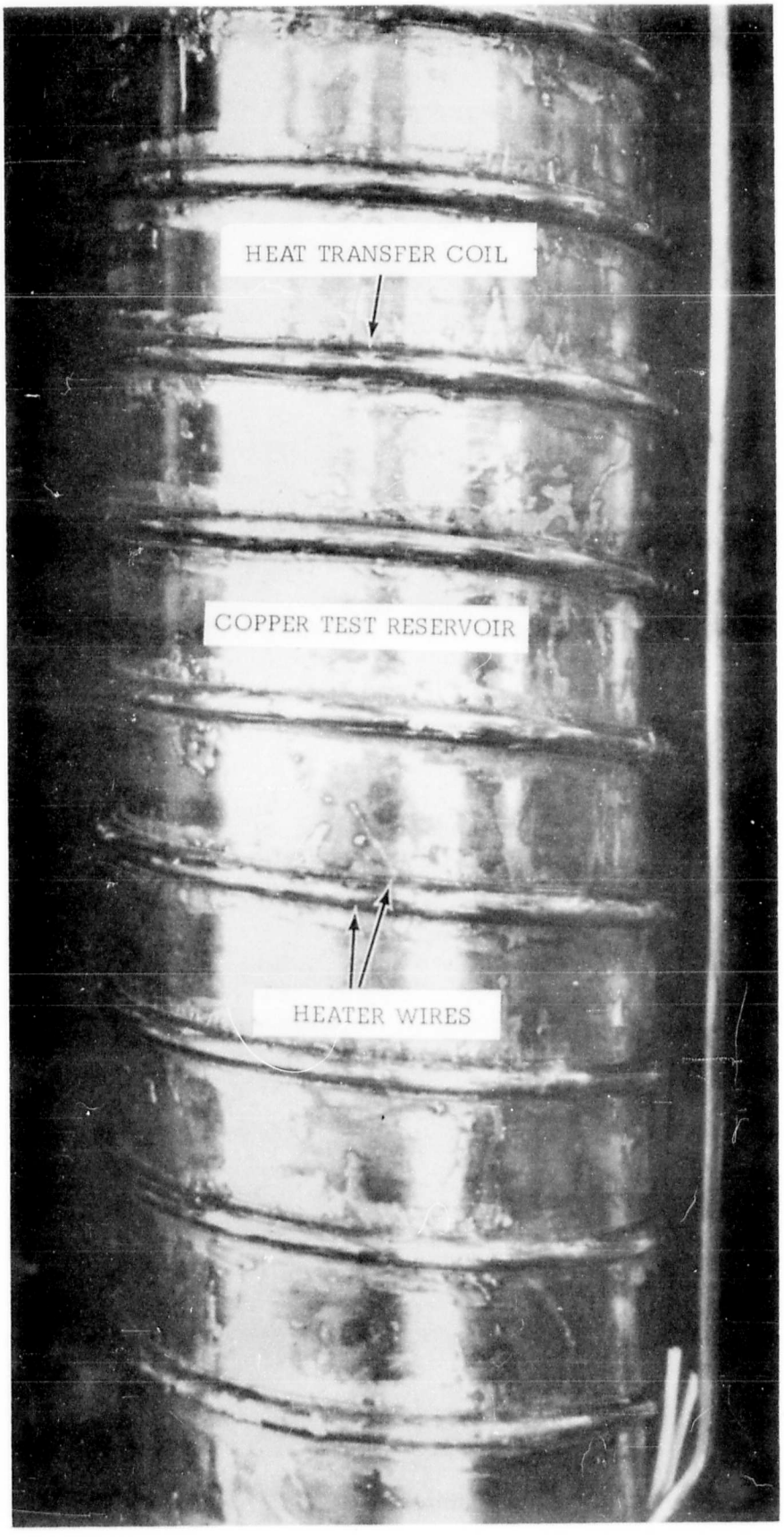

Figure 3. Test Reservoir Showing Heat Transfer Coil

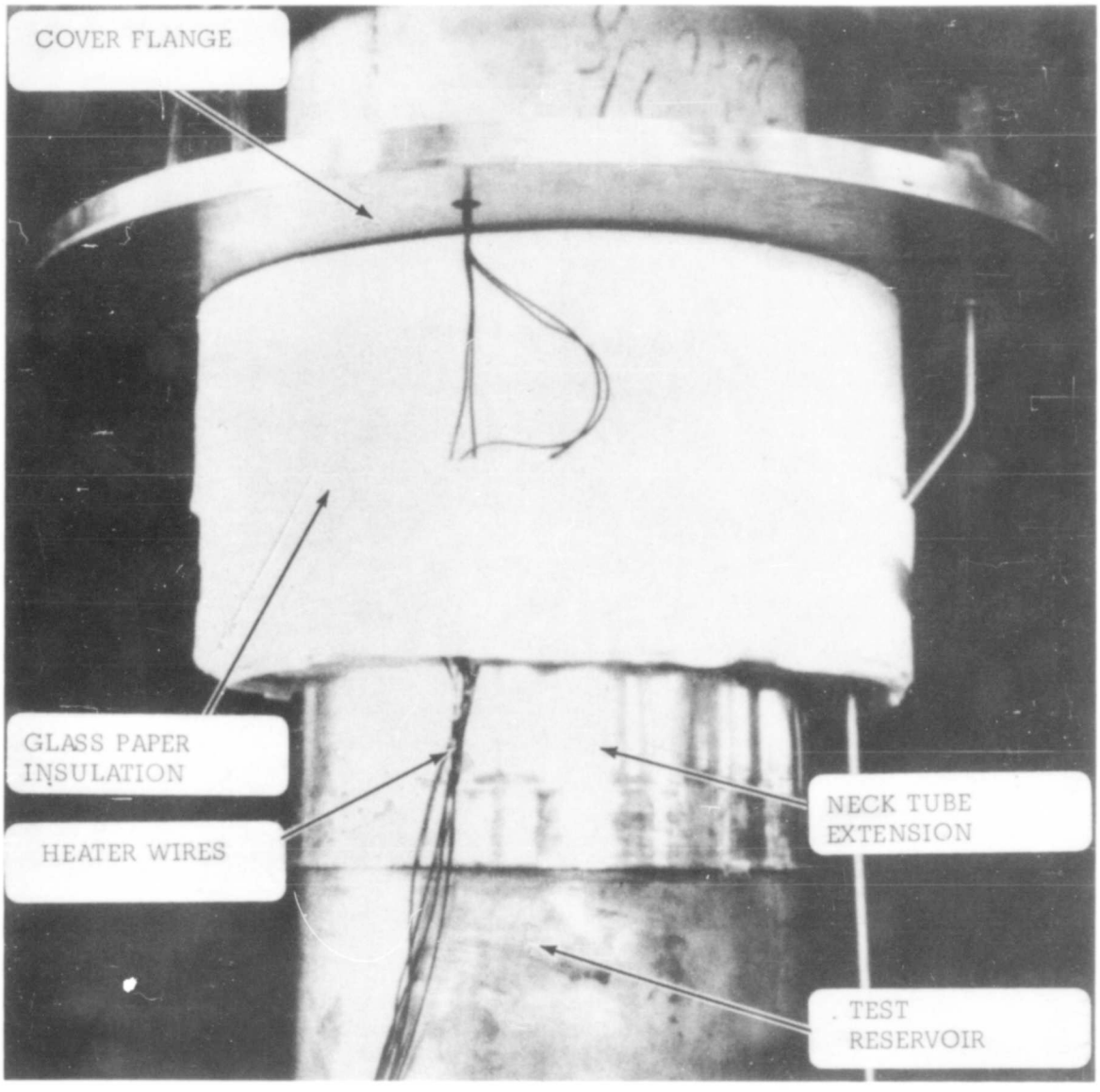

Figure 4. Insulation Detail for Dewar Modification

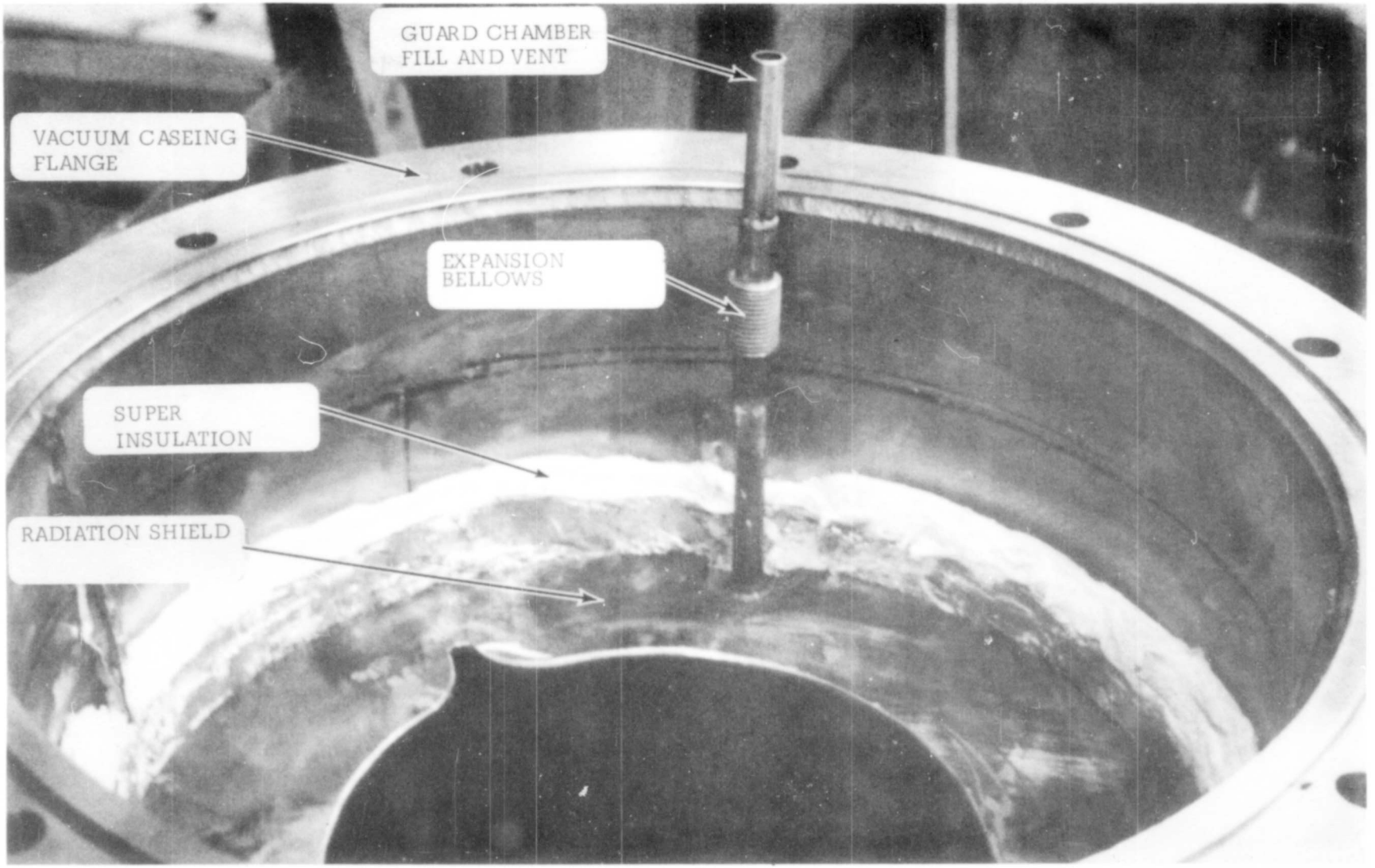

Figure 5. Guard Chamber Dewar Prior to Test Reservoir Assembly Installation

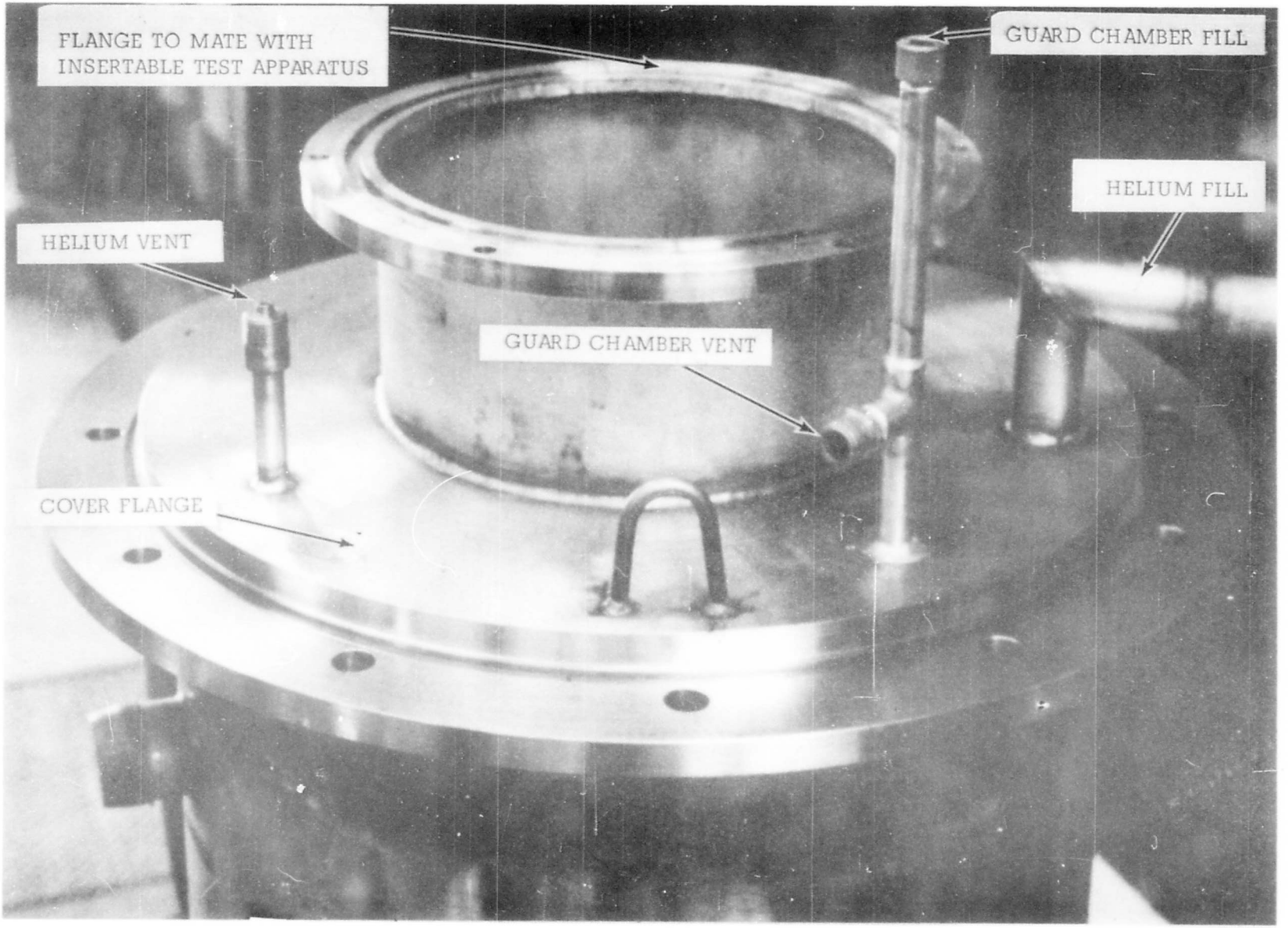

Figure 6. Assembled Modified Guard Chamber Dewar

ring groove to mate with the insertable test apparatus base flange, described in the next section, is welded to a stainless steel cylinder which, in turn, is welded directly to the cover flange.

A helium leak check of all welds and brazed joints was performed during the assembly to insure vacuum integrity. After the modified dewar was completed, the test reservoir, heat transfer coil, and guard chamber bellows assembly were cold shocked with liquid nitrogen, and a final helium leak check showed no detectable leakage.

3.2.1.2 Insertable Test Components

To provide a quick transition of the experimental test system from one slush hydrogen production method to another, insertable test components have been designed for use with each particular production method. The following paragraphs describe the design of the insertable test apparatus as it applies to each of the production techniques.

3.2.1.2.1 Vacuum Pumping Production

Figure 7 shows a side section of the insertable test apparatus for this production method. The base flange mates with the upper flange of the modified guard chamber dewar and is made vacuum-tight by means of an "O" ring seal. This flange will operate at ambient temperatures at all times so that the "O" ring will not become cold and therefore leak.

A double-walled evacuated glass dewar which extends to the bottom of the metal hydrogen test reservoir is vacuum sealed to the cover flange by a set of double "O" ring seals. A slight helium gas pressure will be maintained between the "O" rings to prevent condensables from entering the system in the event of any leakage. The glass dewar is graduated on the outside surface with platinum markings (minimum division 0.2 mm) for determination of slush quality by hydrogen level measurement.

The periscope used for observing the hydrogen during the experiment is inserted into the evacuated double-wall glass dewar and is sealed where it enters the glass dewar with a flexible plastic bag which is sealed to the upper portion of the periscope. A positive helium gas pressure is maintained in the bag and in the periscope guard chamber to preclude the entry of condensables to the inner walls of the glass dewar which will operate near liquid hydrogen temperature.

For this production system, it is necessary to provide both a crust burster and a porous filter with which to compact the solid particles while allowing the triple point liquid to pass. The filter is located above the crust burster and is used to compact the solid particles after slush is produced. Force for the compression is transmitted from outside the apparatus through three drive rods fabricated from thin-wall

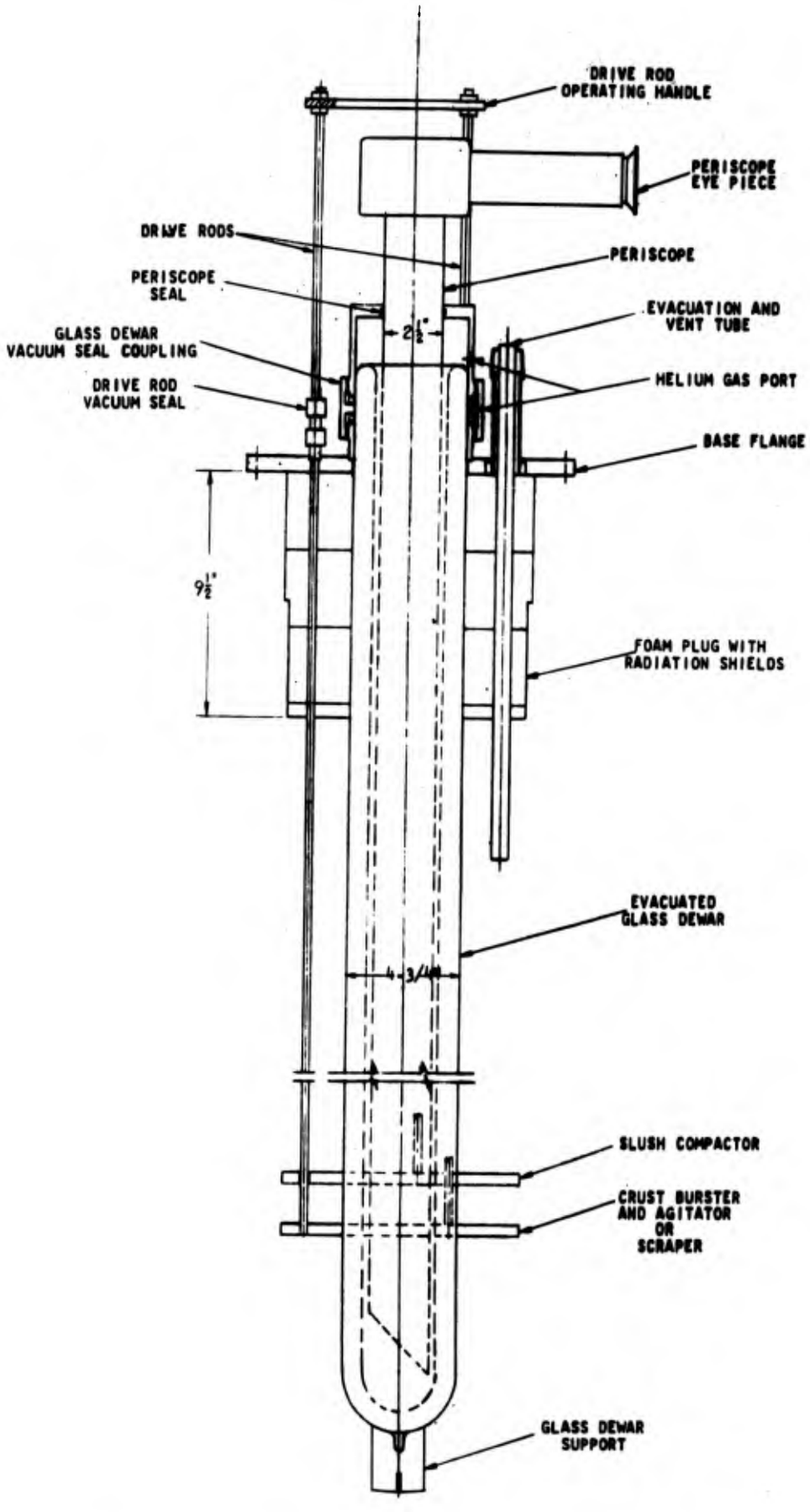

Figure 7. Schematic of Insertable Test Apparatus

stainless tube. The drive rods are vacuum sealed at the base flange by a double "O" ring seal provided with a slight helium gas pressure between the seals so that a condensable gas cannot leak into the test apparatus. The crust burster is located below the filter and is also operated by means of three drive rods with sliding vacuum seals.

The slush compactor which is shown in Figure 8 was fabricated from two brass disks with a center hole cut out to accommodate the glass periscope guard chamber. A number of smaller holes have been cut in the solid portion of the disks to allow passage of fluid. A 150-mesh bronze screen is located between the disks and covers the smaller holes. The purpose of this screen is to allow triple-point liquid to pass through while compressing the solid particles in the hydrogen slush. To reduce leakage and friction where the compactor contacts the test reservoir and the glass periscope guard chamber, a cut-out Teflon washer was also placed between the brass disks and has an outside diameter greater than the brass disks and an inside diameter less than the brass disks. The two brass disks with the screen and Teflon washer between are held together with four bolts. Three additional holes are provided in the compactor for the passage of the crust burster drive rods. Three 3/8-inch x 0.010-inch wall drive rods, which extend to the outside of the test system, are brazed to the upper brass disk and transmit the driving force necessary for the compression.

Figure 9 shows the crust burster and agitator. The crust burster, which is similar to the slush compactor, was fabricated from two stainless steel disks. A center cut-out is provided for the glass periscope guard chamber, and a large number of smaller holes are cut in the disk to provide as small a surface area as possible consistent with good crust bursting. The surface area should be a minimum so that, with a constant force applied to the drive rods, the pressure produced for breaking the hydrogen crust will be a maximum. Also, the smaller holes are provided for the passage of the hydrogen slush while traversing the crust burster during periods of agitation. The crust burster also utilizes a Teflon washer between the stainless steel disks which is smooth where it contacts the glass periscope guard chamber and toothed where it contacts the copper test reservoir wall. Three 3/8-inch x 0.010-inch wall stainless steel drive rods are brazed to the upper stainless steel disk, and the Teflon washer is held firmly in place between the two disks with four bolts.

A cylindrical foam plug with aluminum radiation shields is placed directly below the cover flange and extends down nearly to the level of the top of the liquid hydrogen guard chamber. This foam plug is coated with a low temperature epoxy to prevent hydrogen gas saturation in the foam and also to prevent foam particles from dropping into the liquid hydrogen in the test reservoir. The purpose of this plug is to reduce the radiative heat load from the upper portion of the apparatus and to maintain the base flange at ambient temperature. Holes are provided in the plug for the entry of the periscope guard chamber, driving rods, evacuation tube and fill line.

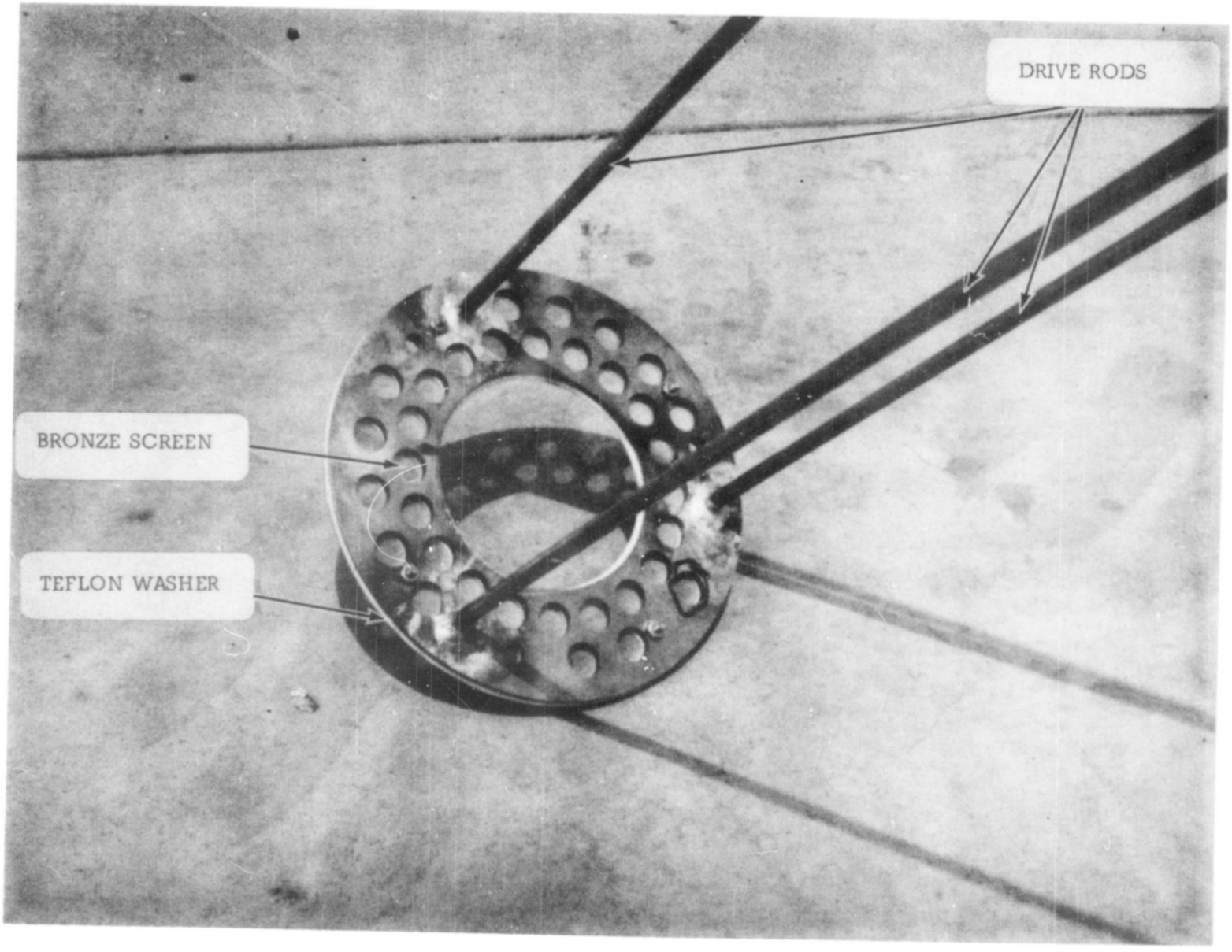

Figure 8. Slush Compactor

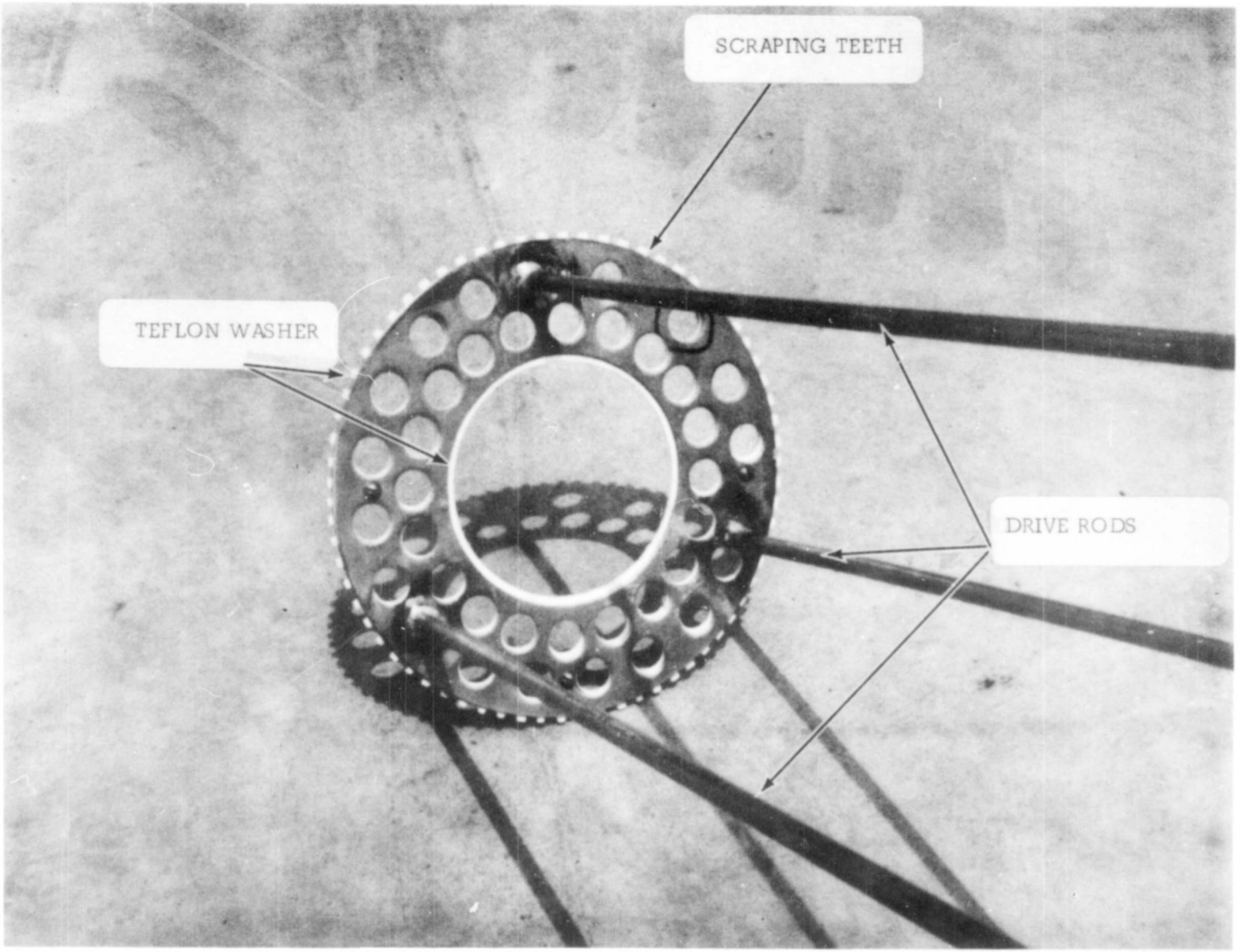

Figure 9. Crust Burster and Agitator

The system evacuation and vent tube passes through the cover flange and extends to the bottom edge of the foam plug. To prevent the cover flange from becoming cold during vacuum pumping and venting, an extended double-wall vacuum-insulated tube is utilized for connection to the base flange.

The liquid hydrogen fill port consists of a quick-connect coupling brazed directly to the base flange (not shown in Figure 7). Through this coupling the vacuum-insulated transfer line can be inserted for transferring the liquid hydrogen into the test reservoir. The gas generated by vaporization during the fill is discharged through the evacuation and vent tube directly to the hydrogen vent stack.

3.2.1.2.2 Helium Injection Production

The insertable test apparatus for this production method is essentially the same as for the vacuum pumping followed by mechanical compression method. The helium gas sparger for injecting cold helium gas into the hydrogen consists of a small diameter metal bellows with holes drilled in the convolutions. Two helium injection lances were built, one for use with warm gas and the other for cold gas resulting from the vaporization of liquid helium. The lance for the warm gas injection (see Figures 10 and 11 lower lance) consists of a long 3/8-inch diameter stainless steel tube of 0.010-inch wall thickness, having a one-foot-long bronze flexible tube of 5/32-inch inside diameter brazed to it. The end of the flexible tube was sealed and six holes of 1/32-inch diameter were drilled into the flexible tube near the lower end. The holes were spaced one inch apart. The cold gas lance (see Figures 10 and 11 upper lance) has a 1/4-inch stainless steel tube of 0.010-inch wall thickness brazed to the end of a vacuum-insulated transfer line. Attached to the lower end of the stainless steel tube is a one-foot length of bronze flexible tubing of 1/8-inch inside diameter. Again six holes were drilled into the flexible tubing.

The injection lance will be inserted through the liquid hydrogen fill port. Upon insertion the bellows will deflect and snake across the bottom head of the hydrogen test reservoir and thus provide good distribution of the helium gas throughout the test reservoir. To insert the lance through the compactor, it was necessary to punch a hole in the porous screen covering one of the flow passages.

3.2.1.2.3 Liquid Helium Freezing Production

Essentially the same apparatus as described above is used for this production method. For indirect cooling, the heat exchanger coil which is attached to the test reservoir is utilized by passing liquid helium through this coil to provide refrigeration for cooling the hydrogen. For scraping the solid off the inside walls of the test reservoir three devices were used. First, the crust burster (Figure 9) with the toothed

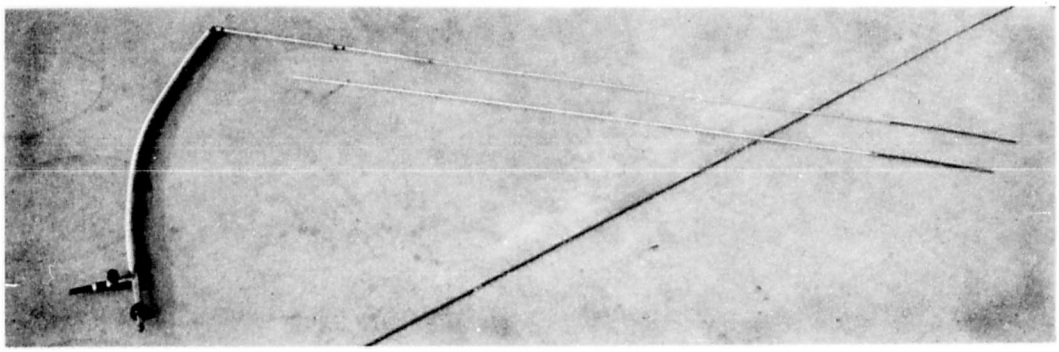

Figure 10. Gaseous Helium Injection Lines

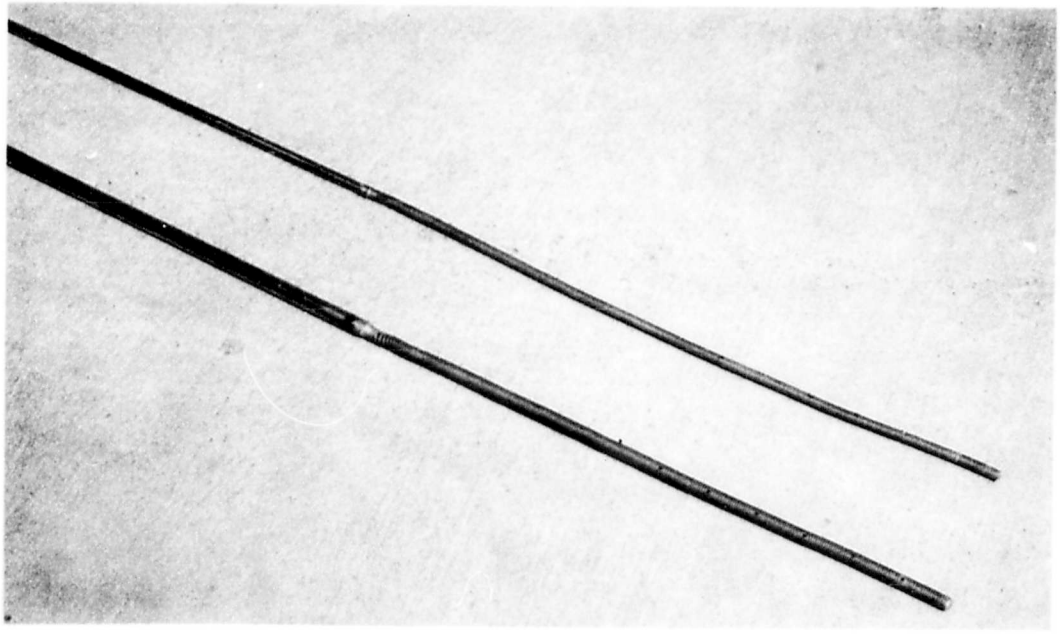

Figure 11. Flexible Ends of Gaseous Helium Injection Lines

edge was used with only partial success; and secondly, a knife-edge scraper was used with moderate success until failure. Figure 12 shows the knife-edge scraper used to remove the solids from the inner wall of the test reservoir. This scraper is fabricated from stainless steel sheet. The outermost band is the slicing edge, while the inner band provides additional stiffness to the unit and aids in mixing the solids in the liquid hydrogen. The operating rods and their locations in the test reservoir are the same as for the crust burster since this scraper replaces the crust burster. Figure 13 shows the second knife-edge scraper which was fabricated upon failure of the scraper described above. This scraper is fabricated from heavier gauge stainless than the first scraper.

For direct heat exchange, the liquid helium is transferred into the hydrogen through the vacuum-insulated transfer line which is used for filling the test reservoir with liquid hydrogen.

3.2.1.3 Assembly of the Test System

The assembly of the insertable apparatus in the modified guard chamber dewar is shown in Figure 14. Both the crust burster and the slush compressor have been inserted in the test reservoir as evidenced by the drive rods extending from the apparatus. The foam plug with aluminum radiation shields is inserted into the test reservoir neck tube with the drive rods projecting through the holes provided. The glass periscope guard chamber which extends to the bottom of the test reservoir also projects through the foam plug.

Figure 15 shows the completely assembled test system located in a specially excavated pit in the hydrogen laboratory. By placing the test system in the five-foot-deep pit, it is possible to operate the drive rods and observe through the periscope while standing on the floor most of the time. Shown in Figure 15, of particular interest, is the upper portion of the periscope which was used during the experimental test program. The periscope is a modified and extended war surplus periscope that has the capability to rotate within the glass guard dewar without rotating the eye-piece extension. Also, the periscope can be moved in and out of the glass guard dewar with a slight force since the periscope is counterbalanced. The line of sight of the periscope can be rotated through 90 degrees with an adjustment knob. With this feature it is possible to look down on the surface of the hydrogen as well as to look parallel to the surface of the hydrogen while determining the level. Lighting to observe the hydrogen is provided by two miniature 6-8 volt bulbs which are operated through a Variac. Either one or both bulbs may be lighted as the liquid requirements demand. As the experimental program proceeded, a larger 25-watt light was installed on the periscope to improve the observations.

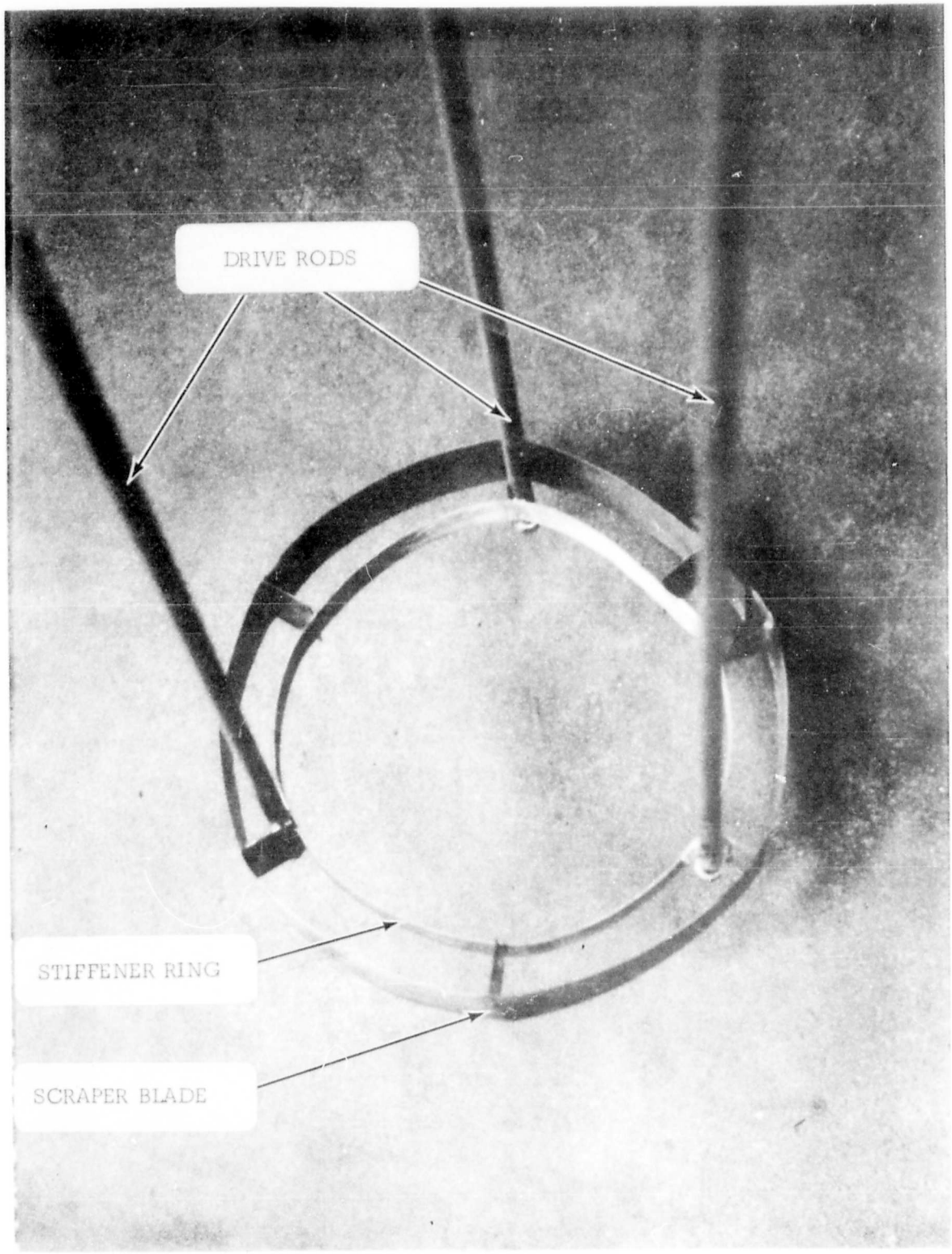

Figure 12. Knife Edge Scraper Number 1

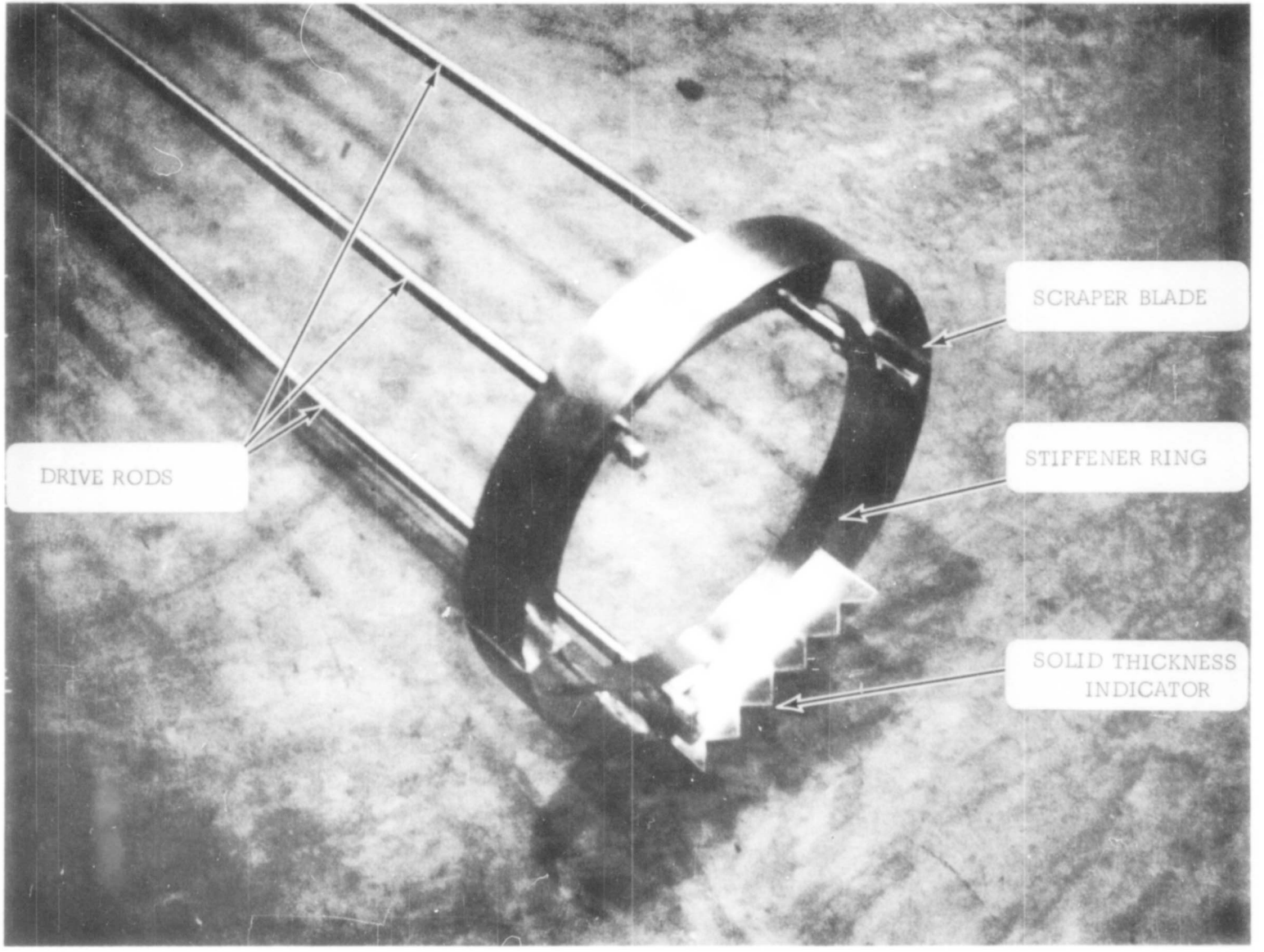

Figure 13. Knife Edge Scraper Number 2

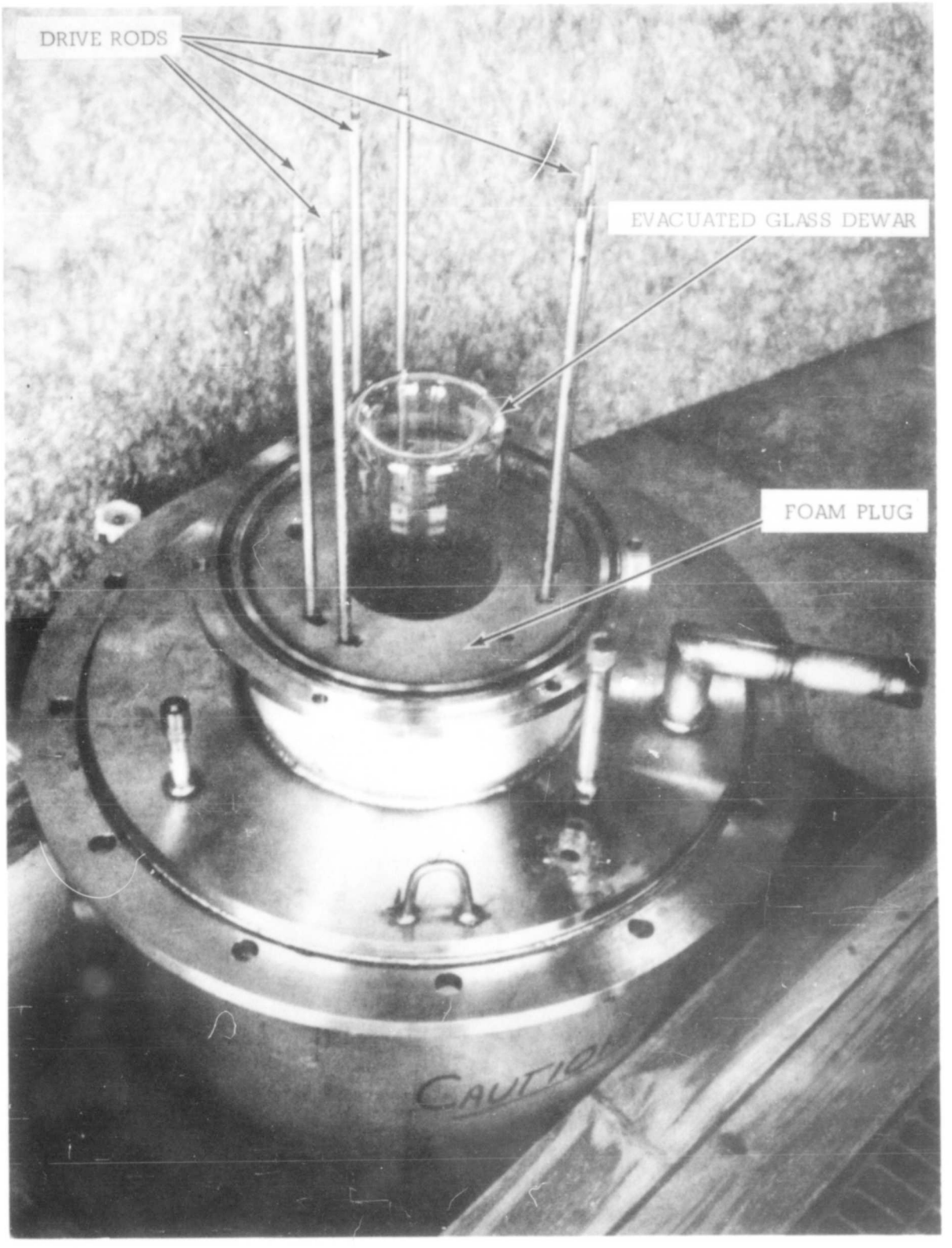

Figure 14. Assembly of Insertable Test Apparatus Components

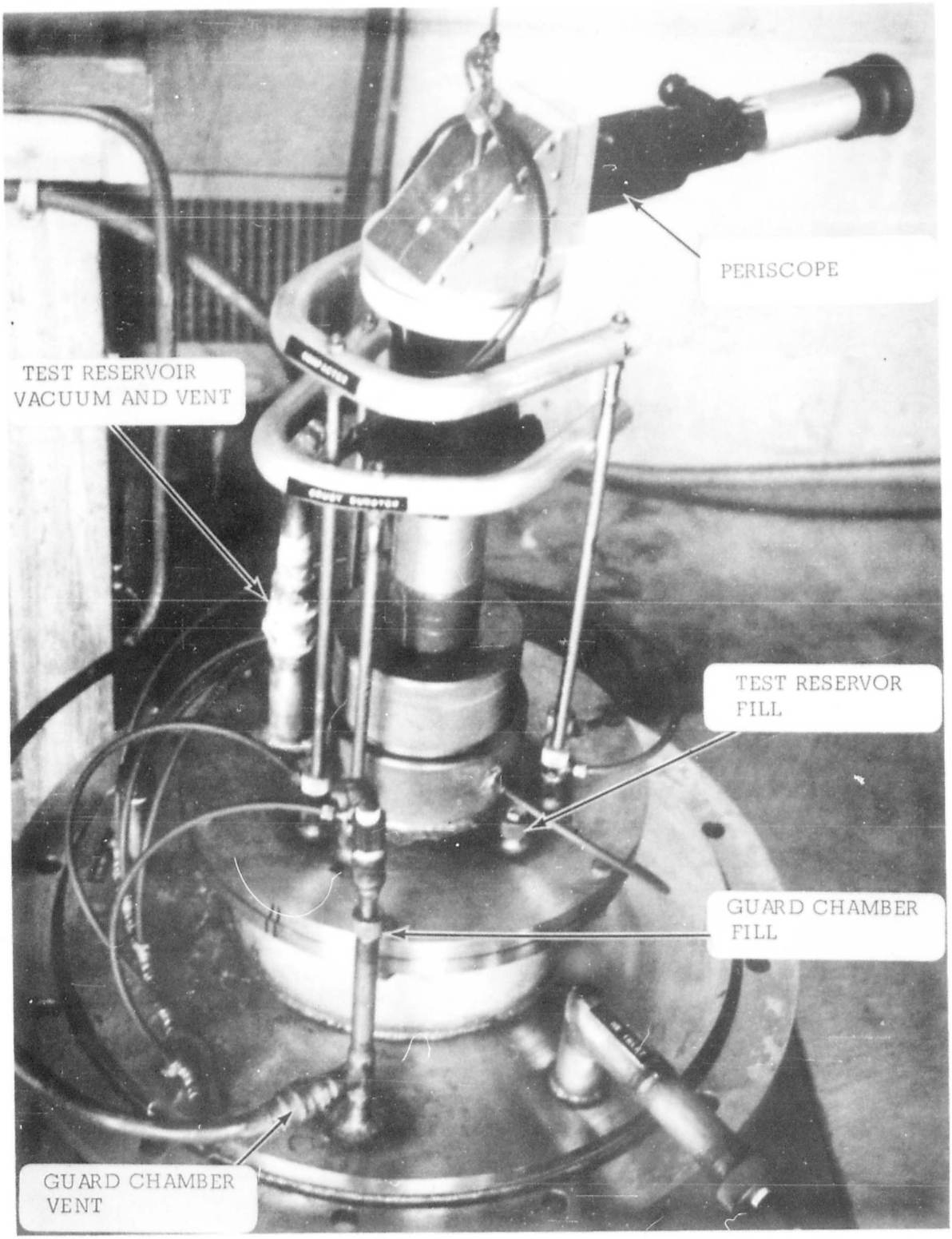

Figure 15. Assembled Test System

The drive rod handles shown in the upper portion of Figure 15 are U-shaped so that the periscope can be translated in the test system regardless of the position of the slush compactor and crust burster. Figure 16 is a schematic drawing of the experimental test system for hydrogen slush studies. The piping layout used during the experimental testing can be divided into two major systems--the test reservoir vacuum pumping and vent system, and the guard chamber vent system.

The test reservoir vacuum pumping and vent system is composed of two branches, only one of which will be in use at a given time. The vacuum pumping rate on the test reservoir is controlled by valve V_1 and throttle valve V_2 while the system pressure is read on mercury manometer P_1. A 140 cfm mechanical vacuum pump located outside the laboratory provides the vacuum for producing hydrogen slush. The gas flow from the exhaust of the vacuum pump is filtered through an oil filter, saturated in a water bubbler and passed through wet drum meter F_1I before being exhausted to the stack.

While filling the test reservoir with liquid hydrogen, valves V_1, V_2 and V_9 are closed, and valve V_7 is opened to exhaust the vaporized gas directly to the stack. At the conclusion of the fill and during normal evaporation of the test reservoir, valve V_7 will be closed and valve V_2 opened, allowing the hydrogen gas to be metered in wet drum meter F_2I prior to discharge to the stack.

While filling the guard chamber with liquid hydrogen, valve V_3 is closed and valve V_4 is opened to allow the gas caused by flashing and cooldown to be rapidly vented. During normal venting valve V_4 is closed, and valve V_3 is opened to allow the guard chamber boil-off to be metered in wet drum meter F_3I and then vented to the stack.

All safety precautions normally employed in the design of liquid hydrogen systems were incorporated in the design of the test apparatus. The gaseous hydrogen from the wet drum meters and the safety relief valves were piped to a vent stack which exhausted high above the laboratory. The safety relief valves were employed wherever the possibility of trapping cold fluid existed.

Figure 17 is a photograph showing the arrangement of the test system components as located in the hydrogen laboratory. Positioned on the tabletop from left to right in Figure 17 are wet drum meters F_1I, F_3I, and F_2I. The system control valves V_1 through V_9 are attached to the front of the table for easy manipulation. On the floor beneath the table, two of the three water bubblers can be seen. The third water bubbler is located outside the laboratory adjacent to the main vacuum pump. Also, beneath the table are located a liquid nitrogen dewar and Molecular Sieve cold traps for cleaning the gaseous helium supplied from K cylinders. The test apparatus, which can be seen located in the right-hand side of the figure, is positioned in an especially prepared pit in order to locate the top portion of the apparatus at a convenient working level.

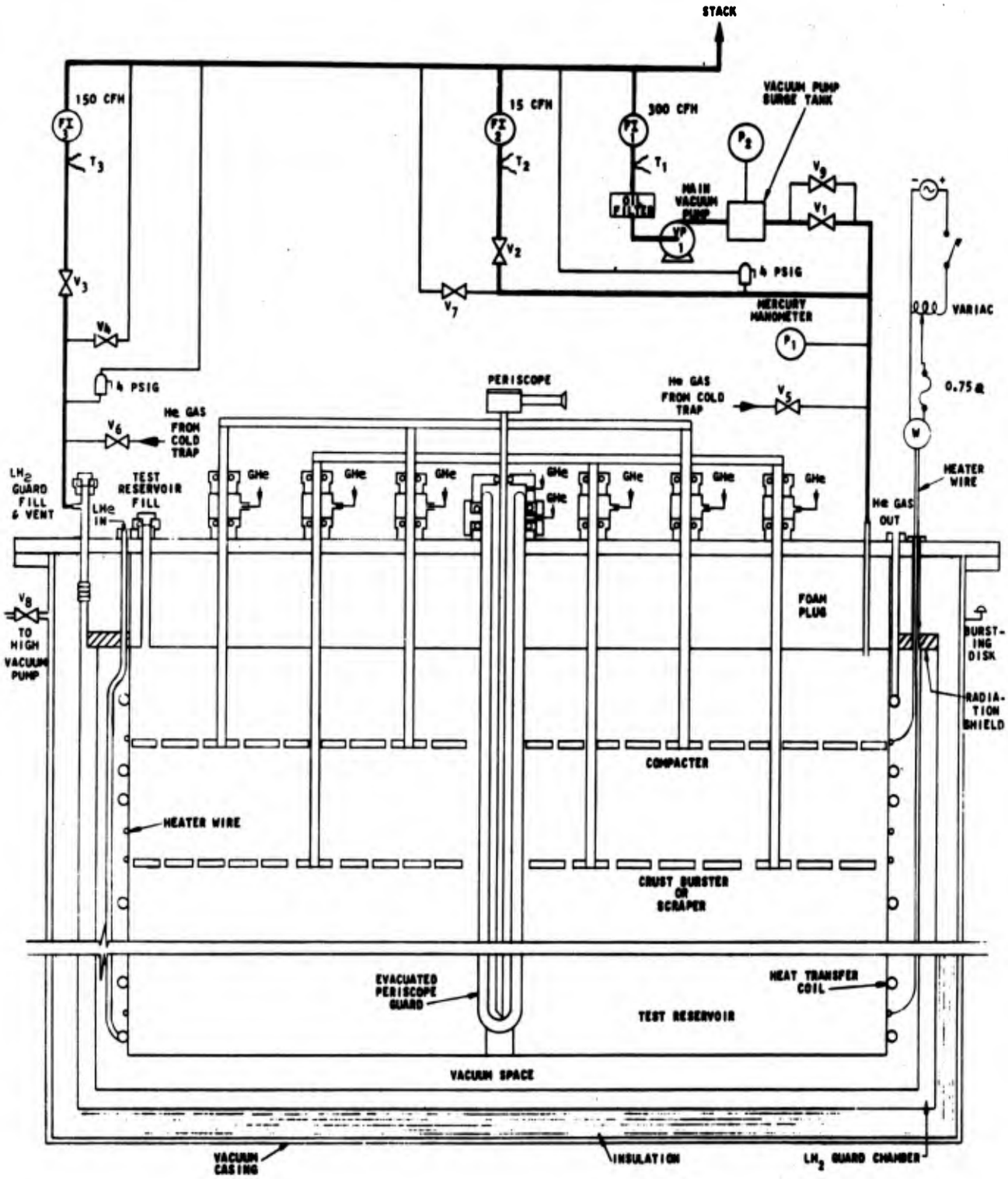

Figure 16. Experimental Test System for Hydrogen Slush Studies

Figure 17. Arrangement of Experimental Test System Components

3.2.2. Instrumentation Used to Obtain Data

Sufficient instrumentation was installed with the experimental apparatus to obtain the data necessary for calculating the quality of the produced slush hydrogen. The parameters that required measurement were time, total gas volumes, liquid levels, pressures, and temperatures. Visual observation of the vessel contents provided important qualitative parameters.

3.2.2.1 Time

The time intervals for various operations were determined by using a large face, synchronous electric clock with a sweep second hand.

3.2.2.2 Total Gas Volumes

The total gas volumes were measured by standard laboratory wet drum meters FI, FI_2 and FI_3. The meters were calibrated over a range of flow rates using a 5 cubic foot gas prover. All hydrogen gas being measured was passed through water bubbles to saturate the gas prior to entering the meters. Before entering the water bubbler for meter FI, the main vacuum pump exhaust gas was passed through an oil filter to prevent oil contamination of the meter. A check of system integrity was accomplished by blanking off the vacuum pump suction and observing meter FI for indications of gas flow. As mentioned earlier in this section, safety was insured by venting all the meters directly to the stack.

3.2.2.3 Liquid Levels

Liquid levels were measured by using the periscope viewing device that had been modified for cryogenic service. Attached to the periscope were the lights which illuminated the liquid level to be observed. The low voltage applied to the lights resulted in a low illumination level that made photographing the vessel contents impossible.

To measure a liquid or slush level, the periscope was inserted into the periscope guard chamber dewar until the level was in view. The observation angle is adjusted until the view is parallel to the level. The level is then read directly from the graduations which are applied to outside surface of the periscope guard chamber dewar.

To determine the relationship between the level and the contained volume, a calibration was performed prior to the final apparatus assembly. The copper test reservoir which is permanently installed in the guard chamber dewar was graduated on the inside surface at one-centimeter intervals of height. Every fifth centimeter was scribed with the corresponding number starting with zero at the bottom. The glass periscope guard chamber was also graduated in centimeters and numbered at each centimeter with submarkings at each 0.2 centimeter. The glass periscope guard chamber was inserted into the

test reservoir and, with the use of the modified war surplus periscope, the volume-height relationship of test reservoir was determined using water as the test fluid. For the graduations on the glass periscope guard chamber, the volume of the test reservoir in liters is equal to 3.240 + 0.3767 x (glass graduations in centimeters), and for the graduations on the wall of the copper test reservoir, the volume of the test reservoir in liters is equal to 3.235 + 0.3804 x (copper graduations in centimeters). The volumes thus determined by reading the graduated scales with the periscope will be used to determine the slush quality by the volume change method upon melting.

The deviation of the second terms of the above two equations is most likely due to the copper graduations being slightly inaccurate as they were hand-scribed inside the copper test reservoir. The copper graduations extend to 125 centimeters, while the glass graduations extend to 120 centimeters. The test reservoir can accommodate approximately 48 liters before the liquid level rises above the graduations.

3.2.2.4 Pressures

The pressure in the test reservoir was measured by pressure gauge P_1 (glass mercury U-tube manometer). One leg of the manometer was open to the atmosphere while the other was connected to the test reservoir through the manifold piping (see Figure 16). During pumping, the absolute pressures indicated on the manometer were lower than the true pressure in the test reservoir by the value of the pressure drop in the manifold piping. To obtain true pressures, all vacuum pumping was stopped momentarily. Atmospheric pressure was determined before and after a run by taking a pressure reading of the surge tank with the test apparatus valved off. Under these conditions the surge tank is normally maintained at approximately 7 microns; therefore, the pressure read on surge tank manometer (P_2) is atmospheric pressure within the accuracy of the millimeter scale used.

The vacuum in the guard insulation space was measured with a tilting-type McLeod gauge that had a measurement range of 0.01 to 5000 microns. This gauge was selected primarily because it is non-electric and would not cause any sparking in the laboratory. However, readings had to be taken manually since the gauge did not provide a continuous reading.

3.2.2.5 Temperatures

The temperatures at the total gas meters FI, FI_2 and FI_3 were measured with mercury-well-type thermometers T_1, T_2 and T_3, specially designed for use with wet drum meters. The temperature at this point is required for computing the wet drum meter correction.

3.3 APPARATUS CHECK OUT

After completion of the test system piping and installation of the instrumentation and pumps, the system was checked out to insure integrity and operation of the following components: guard chamber insulation vacuum system, insulation system vacuum pump, main test reservoir vacuum pump, control valves, wet drum gas flow meters, crust burster, slush compactor, glass periscope guard dewar, periscope, and the electric heater.

To check out the test apparatus, the guard chamber and the test reservoir were both purged with nitrogen gas to remove all remaining moisture and air, and then the test reservoir was evacuated with the main vacuum pump and back-filled with helium gas twice to insure the removal of all contaminants. The inside of the glass periscope guard chamber was also purged with gaseous helium to preclude the presence of air.

The guard chamber was filled with liquid nitrogen, and the operation of vent valves V_3, V_4 and wet drum meter FI_3 was checked. The test reservoir was filled with approximately 12 liters of liquid nitrogen, and the insulation vacuum was measured at 0.1 micron with a non-electric tilting-type McLeod gauge, indicating that the guard chamber vacuum system was tight. The periscope was inserted into the glass guard chamber and observation of the liquid levels was very good. Vacuum pumping on the test reservoir with the main vacuum pump was started and continued until approximately three centimeters of solid crust were formed. Operation of the crust burster and slush compactor was attempted; however, it was found that these components had contracted quite tightly around the glass periscope guard chamber and could not be moved. The heater was turned on at 50 watts and rapid melting of the solid was observed. The test reservoir was then back-filled with helium gas to atmospheric pressure and the liquid nitrogen removed from both the test reservoir and the guard chamber.

The test apparatus was warmed to permit removal of the crust burster and slush compactor by passing hot nitrogen gas through the liquid helium heat transfer coil, which is soldered to the test reservoir, and also by purging the guard chamber with hot nitrogen gas. It was later determined that a faster warm-up could be accomplished by breaking the guard chamber insulation vacuum with gaseous helium.

The crust burster and slush compactor were removed, and a portion of the Teflon washer on the inside diameter, where contact is made with the glass periscope guard dewar, was filed away. After reinstallation of the burster and compactor, the test reservoir was evacuated and pumped overnight. The total gas flow measured by FI_1 was less than 0.1 cubic foot, indicating the system to be leak-tight as a great majority of this gas measured no doubt came from the foam plug and residual water vapor.

Again the guard chamber was filled with liquid nitrogen and 17 liters of liquid nitrogen were transferred to the test reservoir. Operation of the crust burster and slush compactor was found to be satisfactory. The heater wire resistance was found to be 125 ohms when at liquid nitrogen temperature and 135 ohms at ambient temperature. Vacuum pumping on the test reservoir was started and continued until nitrogen solids formed on the liquid nitrogen surface in needle-shaped particles. Vacuum pumping was stopped and the solid crust settled to the bottom of the test reservoir. The crust burster was then pushed down to the bottom, and some particles could be seen floating up through the holes in the burster. With the crust burster remaining in the bottom of the test reservoir, vacuum pumping was started and a solid crust began to form on the surface of the liquid nitrogen. The burster was raised and the crust was carried into the vapor space above the liquid. When the burster was lowered into the liquid, the crust broke up into wafer-shaped particles. More crust was allowed to form with the burster below the surface. The pumping was then stopped and the slush compactor lowered, forcing the crust into the liquid until the crust was filling the gap between the burster and compactor. Moving the burster and compactor closer together caused needle-shaped solid nitrogen particles to be forced through the crust burster and to settle to the bottom of the test reservoir. Additional solid formation and manipulation of the crust burster and slush compactor indicated that the test apparatus was in operating condition.

With the liquid nitrogen check-out satisfactorily completed, all the liquid nitrogen was removed from the test reservoir and guard chamber. The apparatus was completely purged with helium gas and the guard chamber was filled with liquid hydrogen. After the cooldown was complete, as determined by a constant guard chamber evaporation rate, 22 liters of liquid hydrogen were transferred to the test reservoir. The operation of the crust burster and slush compressor was determined to be satisfactory; however, the observation through the periscope was found to be unsatisfactory as condensables had formed on the inside of the glass periscope guard dewar. It was judged that this condensation was caused by a poor seal where the periscope enters the test apparatus. The liquid hydrogen was removed from the test reservoir, and the inside of the glass periscope guard chamber was cleaned by alternating purges with warm helium gas, followed by evacuation. After cleaning, the periscope was reinstalled utilizing a polyethylene bag which surrounded the periscope and was sealed to the upper portion of the periscope and also to the periscope feedthrough coupling on the test apparatus. To preclude entry of condensables, the bag was continuously purged with a slight flow of helium gas.

The test reservoir was filled with approximately 18 liters of liquid hydrogen, and observation through the periscope was found to be clear. Vacuum pumping was started on the liquid hydrogen and continued until solids formed on the liquid surface appearing as a crust. The crust burster was moved down to contact the crust, and three centimeters of crust

were broken. The broken particles appeared as solid chunks when settling to the bottom of the test reservoir. More solid crust was formed and the vacuum pumping was stopped. Upon partial melting the crust appeared to settle to the bottom of the test reservoir, retaining the initial crust shape.

Again problems were encountered with the periscope observation due to condensables which had formed inside the glass periscope guard chamber. This time the cause was found to be leakage through the periscope where the electrical wires enter and also where the two sections of the periscope are joined with a coupling. The ployethylene bag was extended on the upper portion of the periscope to cover the above-mentioned leaks. Figure 17 shows the polyethylene bag as it was finally installed. A manometer and relief device were also installed in the bag purge system so that positive helium pressure could be kept in the bag at all times. When the periscope is raised, the displaced volume in the glass periscope guard chamber must be filled with helium gas or else a partial vacuum will be created in the bag, causing condensables to enter through any small leak which may develop and not be detected.

During cleanup of the glass periscope guard chamber, the outside glass wall shattered almost to a powder. No explanation could be found for the glass breakage unless the rubber "O" ring seal had been tightened excessively, causing an excessive stress. The glass was cleaned out of the test reservoir and the previously fabricated standby glass periscope guard chamber was installed. No further glass breakage has been experienced since that time. Upon refilling the test reservoir with liquid hydrogen, the calibration of the replacement glass periscope guard chamber was determined with reference to the graduations which were etched in the copper test reservoir. The volume of the test reservoir in liters is equal to $3.290 + [0.3767 \times (\text{glass graduations in centimeters})]$.

With the check-out of the experimental apparatus considered acceptable, heat leak determinations were started.

3.3.1 Heat Leak Determination

Determination of the experimental test apparatus heat leak is necessary so that the quality of the slush hydrogen produced may be calculated by the vapor-volume method described in Section 3.4.2. Also, knowledge of the heat leak during a slush production run may allow a correlation to be found between the heat leak and the resulting slush hydrogen quality. The quality of slush hydrogen produced during an experimental run may be determined, however, without use of the test apparatus heat leak by using the volume change method of calculation described in Section 3.4.1.

The heat leak of the test apparatus may be determined by three separate methods with varying degrees of accuracy. The most accurate method, and the method employed, is to measure the boil-off of the liquid hydrogen in the test reservoir with a wet drum meter while the test reservoir is maintained at atmospheric pressure. Any small leakage in the test system piping will have a negligible effect on the measured flow, as there would be essentially no pressure drop across the leak. The heat leak thus determined at atmospheric pressure can be corrected to a triple-point heat leak by multiplication with the heats of vaporization ratio.

The second most accurate method of determining the test system heat leak is to vacuum pump on the liquid hydrogen in the test reservoir maintaining a constant, reduced pressure. Under this condition, the evacuation rate, as measured on a wet drum meter connected to the vacuum pump exhaust, is equal to the heat leak rate. With this method any small leak in the system piping or across any of the valve seats will have a considerable pressure differential and, therefore, a small leak will have a very noticeable effect on the measured heat leak. Also with this method it is quite difficult to properly adjust the flow to maintain constant pressure.

The third, and no doubt least accurate, method of heat leak determination is to measure the change of volume in the test reservoir at constant pressure over a given period of time by use of the periscope. This change in liquid volume, or lost liquid volume, can be converted by appropriate constants and a division by time to yield a heat leak in the same units as the first two methods. The inaccuracy in this method evolves from the minimum graduation increment in the test reservoir for determining the volume. In the test apparatus the minimum graduation is two millimeters. The accuracy of the above method is improved if long test times and consequently relatively large volume changes are employed.

The heat leak corrected to triple-point conditions measured with the liquid hydrogen at atmospheric pressure as described in the first method above was found to be 0.252 cu. ft./min. NTP with the periscope below the liquid level, the two lights operating at 7.8 V. and with both the crust burster and the slush compactor in the liquid hydrogen. Removal of the crust burster and the slush compactor from the liquid hydrogen had little or no effect on the measured heat leak as the heat path through these components was not eliminated as surface contact was still present with the copper test reservoir wall.

The heat leak to the guard chamber with the insulation vacuum maintained at 0.06 micron was determined to be 0.174 cu. ft./min. NTP. Heat leak tests conducted while using the electric heater wire showed no increase in the guard chamber heat leak. This indicates that all the heat supplied by the heater wire is being transferred to the liquid hydrogen in the test reservoir. The heat leak in cu. ft./min. NTP at triple-point conditions being supplied by the heater to the test reservoir is the product of the input watts multiplied by 0.05625.

3.4 DETERMINATION OF SLUSH HYDROGEN QUALITY

For the analysis of the slush hydrogen produced during this experimental work, two calculation methods were used to calculate the compressed slush quality. The volume-change method utilizes the change in density and corresponding volume change when slush hydrogen is melted to determine the slush hydrogen quality. The vapor-volume method utilizes the knowledge of the refrigeration provided (vaporization to produce solid) and the volume occupied by the slush hydrogen in order to determine the quality. A discussion of the above quality determination methods follows.

3.4.1 Volume-Change Method

This slush hydrogen quality determination method as indicated above requires a knowledge of the volume occupied by the compressed slush and also the volume occupied by the triple point liquid resulting from the melting of the solids in the slush. As indicated, the use of this method for calculation of slush quality requires that the slush be destroyed; however, no prior knowledge of the slush production, such as the amount of vacuum pumping performed or the heat leak during production, is necessary to determine the required information for the calculation. Reference 1 gives the equation which is used to determine quality by the volume-change method. The equation is shown below for reference as Equation (1).

$$\text{Slush hydrogen quality} = \frac{1 - \frac{V_1}{V_2}}{0.1109} \times 100 \qquad (1)$$

where V_1 is the volume occupied by the compressed slush. For our apparatus V_1 (liters) = 3.29 + [0.3767 x (glass graduations in centimeters)]. V_2 is the volume of triple point liquid which results from the complete melting of the compressed slush. The constant 0.1109 results from the relationship $[1 - \frac{\rho_{TP}}{\rho_S}]$ where ρ_{TP} is the liquid density at triple point and ρ_S in the solid density.

3.4.1.1 Accuracy of Volume-Change Method

Errors in the calculated quality when using the volume-change method can result from the following considerations: accuracy in reading the glass graduations, accurate determination of the level when all the solids are melted, and the possibility of solids remaining in the vapor space after compression.

Reference 1: R. R. Carney, et al., Theoretical, Experimental, and Analytical Examination of Subcooled and Solid Hydrogen, APL-TDR-64-22. Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, April 1964.

The minimum division of the graduations on the glass periscope guard dewar is 0.2 centimeter. The accuracy in reading the levels with this minimum division is 0.1 centimeter. With this reading accuracy, it is interesting to show how much the quality can be in error at various melted slush levels. Equation (1) can be written in terms of the levels X_1 and X_2 as:

$$\text{Quality} = \frac{1 - \dfrac{X_1\ (0.3767) + 3.29}{X_2\ (0.3767) + 3.29}}{0.1109} \times 100 \qquad (2)$$

where X_1 is the compressed slush level and X_2 is the level resulting from melting the compressed slush. Equation (2) can be further manipulated to the following:

$$\text{Quality} = \frac{X_2 - X_1}{0.1109\ X_2 + 0.9686} \times 100 \qquad (3)$$

In measuring the quality of compressed slush, there is always some triple point liquid above the compressor which must be subtracted from the final melted level in order to determine X_2, the level resulting from melting the compressed slush. X_2 can therefore be expressed:

X_2 = Final melted level - [Level of triple point liquid above compressed slush at compression - Compressed slush level (X_1)]

Inspection of the above shows that X_2 could possibly be in error by $\pm$ 0.3 cm. because of the three levels necessary to determine X_2; however, the difference ($X_2 - X_1$) can only be in error by $\pm$ 0.2 cm. To determine the effect of the above possible errors on the quality, a sample calculation follows. Assuming the true melted slush level was 20 cm., then using Equation (3) the possible error in quality points (not percentage of calculated quality) can be calculated as follows:

$$\text{Maximum quality points error} = \frac{-0.2}{(20.0 - 0.3)\ 0.1109 + 0.9686} = -6.34$$

$$\text{Maximum quality points error} = \frac{+0.2}{(20.0 + 0.3)\ 0.1109 + 0.9686} = +6.21$$

As the melted level increases, the maximum error which may be present from reading the calibrations on the glass is reduced. Figure 18 is a plot of the maximum error which can be caused by measurement versus the melted slush level. Because the error can be quite appreciable at low levels, the test reservoir in the experimental apparatus is long, thus providing working depths where the calculations should be fairly accurate.

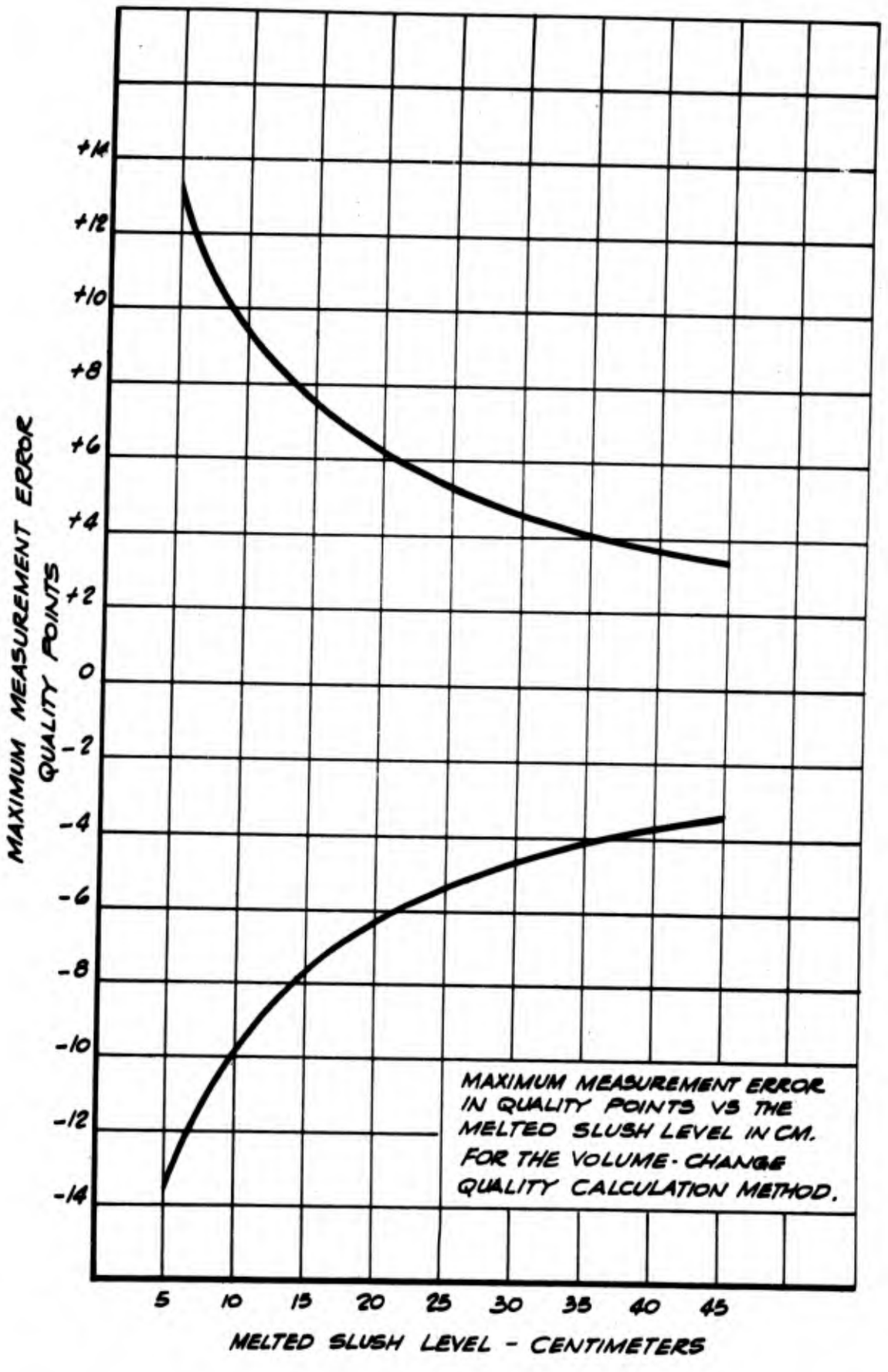

Figure 18. Maximum Measurement Error for Volume-Change Quality Calculation

A second possible cause for error in the calculated quality is the difficulty in determining the exact time and therefore level when all the solids in the compressed slush are melted. This difficulty arises from the fact that solids in the very bottom of the test reservoir cannot be seen. To determine the presence or absence of solids, it is necessary to agitate the liquid near the bottom of the test reservoir with the crust burster, causing the remaining solid particles to be moved into view. The magnitude of an error of the above type is difficult to estimate; however, it is felt that with very careful and exacting observation when the solids are melting this type of error can be nearly eliminated.

A third possible cause for quality calculation error is the presence of some solid hydrogen which may be sticking to the walls in the vapor space or in the triple point liquid above the compactor. This condition always causes the calculated quality to be higher than the actual quality because upon melting these solids will cause the melted slush volume to be greater than it would have been if only the compressed slush had melted. With examination of Equation (1), it can be seen that V_2 will be greater due to the extra solids, causing the numerator of the equation and therefore the quality to be higher. It is difficult to determine the presence of solids in the vapor space or in the triple point liquid above the compressor in our apparatus because only a small area of interest can be observed at one time, and generally this area includes determining the correct liquid and solid levels during the compression. When a quality, however, is calculated to be well in excess of 100 per cent, it is obvious that there must have been solids in the vapor space in order to cause such a large calculation error.

3.4.2 Vapor-Volume Method

This quality determination method does not require that the compressed slush be melted; however, the history of the slush production is necessary to make the calculation. This method is based on the principle that refrigeration produced by vacuum pumping after the triple point is reached is used directly to produce solids and intercept the system heat leak. The refrigeration produced is determined by measuring the vacuum pump exhaust gas with a wet drum meter. The system heat leak is determined by methods described in Section 3.3.1, or it can be determined by a system volume balance if the compressed slush is allowed to melt as it was during our experimental runs. To make this volume balance, the corrected total volume pumped (from the start of the first solids until the vacuum pump is stopped) is equated to the unknown heat leak times the time expired between the point where all the solids are melted and the point of first solids plus any additional known heat input such as the electric heater heat leak; the compressed slush level is also required to perform the quality calculation.

The equation used to determine the slush hydrogen quality can be developed as follows. From the basic definition of slush hydrogen quality, the quality is equal to the mass of solid in the slush divided by the mass of solid plus the mass of triple point liquid in the slush.

$$\text{Quality} = \frac{M_s}{M_s + M_l} \tag{4}$$

where M_s is the mass of solid in the hydrogen slush and M_l is the mass of triple point liquid in the hydrogen slush. Also, the volume occupied by the triple point liquid plus the volume occupied by the solid is equal to the volume of the slush hydrogen.

$$V_l + V_s = V_{ls} \tag{5}$$

where V_l is the volume of the triple point liquid, V_s is the volume of the solid, and V_{ls} is the volume of the slush in liters and is given by the relationship $V_{ls} = 3.29 + [0.3767 \times (\text{glass graduations in centimeters})]$. Equation (5) may also be written as:

$$\frac{M_l}{\rho_l} + \frac{M_s}{\rho_s} = V_{ls} \tag{6}$$

where ρ_l is the triple point liquid density and ρ_s is the solid density. Rearranging Equation (6) in terms of the mass of the liquid yields:

$$M_l = \left[V_{ls} - \frac{M_s}{\rho_s}\right] \rho_l \tag{7}$$

Equation (7), substituted into Equation (4), yields the basic equation used to determine the slush hydrogen quality by the vapor-volume method.

$$\text{Quality} = \frac{M_s}{M_s + \left(V_{ls} - \frac{M_s}{\rho_s}\right) \rho_l} \times 100 \tag{8}$$

The mass of solid present at the slush compression is determined from the volume of the gas pumped from the triple point conditions to the termination of pumping minus the heat leak rate times the time elapsed between the triple point conditions and the time of the compression. Other heat sources such as the heater or the large light must be added to the heat leak if present during the time interval considered.

$$M_s = \frac{\left(\begin{array}{l}\text{Volume pumped from triple point conditions to pump stop, ft}^3\text{ (NTP)}\end{array} - \begin{array}{l}\text{Heat leak rate (ft}^3\text{/min.) times time between triple point condition and compression}\end{array}\right) \times \frac{193.63}{25.03}}{192\ \text{ft}^3/\text{lb.}} \quad (9)$$

where 192 ft^3/lb. converts NTP hydrogen gas to an equivalent mass, 193.63 Btu/lb. is the heat of vaporization of triple point liquid, and 25.03 Btu/lb. is the heat of fusion.

3.4.2.1 Accuracy of Vapor-Volume Method

Errors in the slush hydrogen quality calculation by vapor-volume method may be caused by the following: (1) the measurement error in determining the compressed slush level used in the calculation of V_{ls}, (2) the presence of solids in the vapor space or in triple point liquid above the compressor which by virtue of Equation (8) are included in the compressed slush, (3) the exact determination of the heat leak during the entire period from the start of solid formation to the compression, (4) the presence of system leakage which will affect the determination of M_s as the volume pumped will include this leakage.

Errors caused by the inaccuracy of reading the compressed slush level by $\pm$ 0.1 cm. will have a very minor effect of less than one quality point error in the calculated slush quality. This possible error should therefore not be considered to basically affect the compressed slush quality.

The magnitude of the error which may be caused by solids in the vapor space or in the triple point liquid above the compressed slush cannot be estimated with any accuracy. As stated previously, the detection of solids in these areas is extremely difficult with the periscope which does not allow depth perception.

The determination of the system heat leak should be quite accurate by the methods presented in Section 3.3.1; however, in some runs discrepancies exist between the heat leak as determined by conventional methods and the heat leak determined by a volume balance performed as discussed above. If this error is, however, large and a number of compressions are performed on the same run as in the last runs 16-27 of the vacuum pumping production method, the calculated quality will tend to sharply increase or decrease as the individual compressions proceed.

The presence of system leakage during the pumping to produce hydrogen can cause an error in the determination of the mass of solids produced with use of Equation (9). This system leakage, if not determined and subtracted from the total pumped volume, will yield a mass produced which is larger than the actual mass produced. This larger solid mass when used in Equation (8) will yield a slush quality which is higher than that actually present.

In some of our first few vacuum pumping production runs, it was later determined that some system leakage was present, being caused by leakage through a vent valve connecting the system to the stack vent manifold. This caused the entry of some hydrogen gas from the manifold to enter the system during the vacuum pumping. The amount of leakage could be computed in some cases where the system pumpdown data was complete, allowing the slush quality to be calculated by the vapor-volume method.

3.5 VACUUM PUMPING SLUSH HYDROGEN PRODUCTION

3.5.1 Experimental Production Procedure

For the runs performed, two production process variations were employed--namely, production by the freezing method and production by the melting method. Experimental production runs utilizing both the melting and freezing production methods were performed, because it was determined in work presented in Technical Documentary Report APL-TDR-64-22 that the average settled slush quality produced by the freezing method was 45 per cent, while the average settled slush quality produced by the melting method was 57 per cent. It is desirable to determine if these different production methods will yield different compressed slush qualities.

3.5.1.1 Freezing Method

A typical experimental production run by the freezing method was conducted in the following manner:

The guard chamber would first be filled with liquid hydrogen to insure a constant heat leak to the test reservoir. The test reservoir would then be filled to a level of 60 to 70 centimeters with liquid hydrogen. The importance of a high starting level for accuracy in computing slush quality by the volume-change method was discussed in Section 3.4. With the test reservoir filled, the crust burster and slush compactor would be raised out of the liquid, and the periscope would be adjusted to observe the liquid level. Vacuum pumping would then be started on the test reservoir, cooling the liquid hydrogen in the test reservoir until a solid crust would start to form. The pumping rate for the cooldown to triple-point liquid condition was generally 4-5 cfm, which is near the limit of the wet drum meter capacity. Also during the pumpdown, it is desirable to hold the test reservoir pressure constant for a few minutes by adjusting the flow control valve V_1 (see Figure 16) in order to determine the actual test apparatus heat leak just prior to slush hydrogen production. When a small amount of solid crust is produced, the vacuum pumping is stopped and the desired heat leak for the experimental run is supplied through the electric heater wire. When the solid crust has melted, vacuum pumping is again started and the desired pumping rate is adjusted with valve V_1. As the crust again begins to form, it is allowed to grow to a thickness of 1-2 centimeters and then the crust burster is pushed down, breaking the crust into solid particles which settle

to the bottom of the test reservoir. This process of growing and breaking the crust is continued until the level of solid particle buildup approaches the liquid level where the crust is being formed. At this time vacuum pumping is terminated and the electric heater turned off. The slush compactor is then forced into the settled slush compressing the solid particles while allowing the triple point liquid to pass through. Force is maintained on the slush compactor during the solid particle melting, causing the slush compactor to slowly move downward until all the solid particles have melted and the compactor has bottomed in the test reservoir. By periodically taking the compressed slush level and liquid level above the compressed slush during the solid melting, it is possible to secure many data points for the calculation of the compressed slush quality by the volume-change method.

3.5.1.2 Melting Method

A typical experimental production run by the melting method would be conducted in a manner very similar to that presented above for the freezing method, except for the following deviation: Vacuum pumping would not be stopped when the settled slush level approaches the liquid level, but would continue until all solid was present in the test reservoir as determined by a reduction in pressure from the triple point pressure. In some runs, the crust was allowed to grow without breaking until all solids were present. At this time the vacuum pumping would be stopped and the slush compressor forced down on the solid hydrogen. Continuous force would be applied while the solid partially melted, creating a triple-point liquid level above the slush compressor. With triple-point liquid above the slush compactor, the melting run would be continued exactly in the same manner as the freezing run described above.

In summary, the only difference between the two production methods is that in the freezing method only enough solid hydrogen is produced to make settled slush, while in the melting method all the hydrogen is solidified and slush hydrogen is produced by melting the solidified hydrogen.

3.5.2 Quality Results of the Experimental Runs

A total of 27 experimental production and slush hydrogen compression runs were performed by both the freezing and melting production methods. The heat leak and pumping rates were varied for each run to determine if a correlation exists between the heat leak and compressed slush quality or between the pumping rate and compressed slush quality. The production runs were performed as described in Section 3.5, except variations, where present, are noted in the discussion of the runs. The compressions were performed manually and exerted a pressure on the compressed slush of not more than 1.25 psi.

Table I presents the results of the experimental production runs. Shown are the pump rate, electric heater input, total heat leak rate during production, method of production (freezing or melting), pumping rate minus the heat leak rate, compressed slush quality calculated by the volume-change method, and the melted slush levels for each quality calculation.

Many of the compressed slush qualities by the vapor-volume method are not shown because they could not be calculated with any accuracy. The inaccuracies involved arise from the fact that some system leakage through a vent valve was found to be present up to Run 18, at which time it was corrected. Also, in some runs the absolute heat leak could not be accurately determined. In the runs where qualities are shown by the vapor-volume method, it was possible to use the system pumpdown data to compute the system leakage which was subtracted from the total pumped volume for the vapor-volume quality calculation.

In Runs 16-27, more than one compression data point was taken during the melting of the compressed slush. The calculated qualities are given in the order of the compressions reading down. Also, the average of all the calculated qualities for each individual run is given.

TABLE 1

SUMMARY OF VACUUM PUMPING SLUSH PRODUCTION

Run	Pump Rate $ft^3/min.$	Heater watts	Heat Leak $ft^3/min.$	Method Freezing-Melting	Net Pumping (Pump Rate-Heat Leak) $ft^3/min.$	Compressed Slush Quality-Per Cent Vapor-Volume Method	Compressed Slush Quality-Per Cent Volume-Change Method	Melted Slush Level cm.
1	1.39	no	.252	F	1.138	93.3	132.7	36.1
2	1.03	5	.533	F	.497	77.2	73.9	24.2
3	1.07	10	.815	F	.255	93.7	87.5	20.1
4	.926	7	.646	F	.280	64.2	58.7	5.1
5	3.51	no	.252	F	3.258	---	115	9.3
6	1.38	no	.252	F	1.128	---	75.0	6.9
7	3.65	30	1.94	F	1.71	---	73.2	19.6
8	3.99	45	2.78	F	1.21	---	74.2	10.7
9	2.92	15	1.096	F	1.824	77.3	66.7	18.3
10	3.89	no	.252	F	3.638	---	73.8	19.4
11	1.41	no	.252	M	1.158	---	79.4	27.6
12	1.50	no	.252	M	---	---	---	---
13	1.32	no	.252	M	1.068	---	69.7	4.2
14	1.50	no	.252	M	---	---	---	---
15	1.44	no	.252	M	1.188	---	60.0*	10.8
16	3.72	no	.252	M	3.468	---	83.9 75.1 80.4 Av. 79.8	36.4 23.7 36.1
17	80 min at 1.5 33 min at 3.0 17 min at 4.0	no	.252	M	---	---	102.0 103.9 99.6 Av. 101.8	33.7 28.6 32.9
18	---	---	---	---	---	---	---	---
19	1.40	no	.401	F	.999	---	115 117.4 117.6 110.3 113.5 Av. 114.8	18.70 9.70 18.1 6.80 13.5
20	1.77	no	.902	M	.868	---	66.2 73.9 75.7 74.4 73.6 72.1 72.9 73.7 76.3 Av. 73.1	24.4 12.0 23.4 8.85 21.9 5.65 18.5 3.5 14.9
21	1.72	no	.94	F	.780	70.5 73.1 73.0 69.0 Av. 71.4	75.4 74.6 73.1 54.9 Av. 74.4	15.2 9.4 11.0 7.7
22	---	---	---	---	---	---	---	---
23	3.34	no	.916	M	2.424	82.8 77.3 83.5 73.7 80.3 Av. 79.5	69.3 65.4 71.7 58.1 68.3 Av. 66.7	39.2 20.65 34.1 14.30 26.6
24	3.51	30	2.27	M	1.24	---	56.9 55.6 56.9 50.5 57.6 41.0 Av. 53.1	33.8 21.8 30.6 16.0 27.0 10.8
25	1.50	10	1.153	F	.347	66.5 74.5 74.1 76.5 Av. 72.9	65.4 65.7 66.1 64.8 Av. 65.5	17.2 10.2 12.8 7.7
26	3.59	no	.801	F	2.789	---	82.3 88.9 83.0 86.2 Av. 85.1	21.7 12.3 17.1 6.7
27	3.77	30	2.48	F	1.29	55.9 85.8 81.0 75.7 82.7 Av. 77.2	58.6 92.8 82.5 86.9 88.7 Av. 81.9	35.6 11.4 20.5 7.6 15.4

System leakage through vent valve present until after run 18.
Glass periscope guard chamber replaced after run 22.

*Only 2/3 of slush compressed.

Notes for Table I

Run 1 There was difficulty breaking the crust during production when it grew 2.8 to 3.4 cm. thick. The slush was compacted with the crust burster, with the compactor being one centimeter above the crust bruster. Possibly some solids were present in the vapor space causing the high calculated quality.

Run 2 The crust was broken with the compactor, while the crust burster remained down in the hydrogen. Compression was performed with the compactor.

Run 3 This was a long-term production run (171 minutes) because of low pump rate and high heat leak. The crust was broken with the crust burster and the slush was also compressed with the crust burster.

Run 4 The crust was broken every two minutes with the crust burster, and the slush was also compressed with the crust burster. A very low melted level of 5.1 cm. allows for a large possible measurement error to be present.

Run 5 The crust was broken almost continuously with the crust burster as solid formed very rapidly. The compression was performed with the compactor; however, there was poor visibility during this run. Some solids were observed above the compactor, causing the calculated quality to be too high.

Runs 6-10 The crust was broken with crust burster and the resulting settled slush compressed with the compactor.

Run 11 The crust was allowed to grow with no breaking until all solid was produced. The slush was compressed with the crust burster when the melted liquid level was above solids level.

Run 12 The crust was allowed to grow with no breaking. The compression was performed too late, with not enough solids present; therefore, compressed slush quality could not be calculated.

Run 13 The crust was allowed to grow without breaking, and the compression after some melting was performed with the crust burster. Calculated settled slush quality was 54.6 per cent before compression.

Run 14 This was a handling run. Observations are discussed under characteristics of compressed slush hydrogen.

Run 15 The crust was allowed to grow with no breaking until all solids were produced. Only two-thirds of the slush volume was compressed with the crust burster. The quality calculation is based on the slush level above crust burster.

Run 16 The crust was allowed to grow with no breaking until all solids were produced. After some melting the slush was compressed with the crust burster. A few mounds of solid were noted projecting slightly through the holes in the crust burster.

Run 17 The crust was broken every minute until all settled slush was produced. Pumping was then continued without breaking until all solids were produced. The pumping rate was varied during the production. After partial melting the compressions were performed with the compactor.

Run 18 This run was aborted because the periscope guard dewar became cloudy during the production, making it impossible to observe the levels in the apparatus. After this run the source of system leakage was detected and corrected.

Run 19 The crust was broken during production until all settled slush was produced. All compressions were performed with the compactor. High calculated qualities indicate there may have been solids above the compactor.

Run 20 The crust was broken every minute during production until all settled slush was produced. After partial melting the first compression was made with the crust burster. Subsequent compressions were made with the compactor.

Run 21 The crust was broken during production until all settled slush was produced. Before compression the settled slush quality was determined to be 32.7 per cent by the volume-change method and 36 per cent by the vapor-volume method. All compressions were performed with the compactor.

Run 22 This run was aborted because the O-ring seal between the glass periscope guard chamber and the upper flange was lost, causing gaseous helium from the bag surrounding the periscope to be pulled into the apparatus. The glass periscope dewar was replaced with a standby unit and the new relationship between level and volume was found to be: Volume in liters = 3.39 + [(0.3767) (glass graduations in centimeters)].

Runs 23-24 The crust was broken every minute with the crust burster during the production of settled slush. Vacuum pumping was continued until all solids were produced. After partial melting the slush was compressed for all data points with the compactor.

Run 25 The slow forming crust was broken every two minutes with the crust burster during the production of settled slush. The settled slush was compressed with the compactor. Some solids had accumulated in the vapor space during production as evidenced by a sudden increase in the liquid level when the compactor was pushed into the liquid to compress the settled slush.

Run 26 The crust was broken with the crust burster during the production. The compactor was used for all compressions; however, only approximately 3/4 of the volume of slush was compressed. Cloudy liquid was observed on top of the compactor. The cloudy liquid level was used in determining the slush quality.

Run 27 The crust was broken with the crust burster during production, and the compactor was used to compress the settled slush. The liquid above the compactor was slightly cloudy, indicating some solids present; however, the copper graduations could be seen all the way down to the compactor.

Figure 19 is a graph showing the calculated compressed slush quality as determined by the volume-change method plotted against the heat leak. Runs 1, 5, 17 and 19 were not plotted because the calculated quality was in excess of 100 per cent, thus indicating an error. Also, runs 15 and 26 were not plotted because the entire volume of slush was not compressed. When more than one quality was determined for a production run, the average of the qualities was used in the figure. From the scattering of points on the figure, apparently a correlation between heat leak and quality does not exist for compressed slush. It is interesting to note, however, that out of the 17 points plotted, 9 of the points fall between 70 and 80 per cent quality, while 4 points are between 60 and 70 per cent quality, 2 points are between 50 and 60 per cent quality, and 2 points are above 80 per cent quality.

Figure 20 is a graph showing the calculated compressed slush quality as determined by the volume-change method plotted against the net pumping rate (pump rate - heat leak rate). Again, as in the case of the heat leak, it appears that a correlation between the compressed slush quality and the net pumping rate does not exist, at least within the accuracy of calculated compressed slush qualities.

From the qualities calculated for Table 1 by both the vapor-volume method and the volume-change method for both freezing and melting type runs, it is apparent that the compressed slush quality resulting from either of the above production methods falls within the range of 65 to 85 per cent quality. If all the freezing type runs as calculated by the volume-change method are averaged discounting runs 1, 5, 19 and 26, the resulting compressed slush quality is 73.2 per cent. For the freezing type runs calculated by the vapor-volume method, discounting run 1, the average compressed slush quality is 76.3 per cent. The average quality of all the melting runs discounting runs 15 and 17, determined by the volume-change method, is 70.3 per cent, while the quality as determined by the vapor-volume method is 79.5 per cent.

Shown in the Appendix are sample calculations illustrating the methods used to calculate the slush hydrogen quality presented in Table 1.

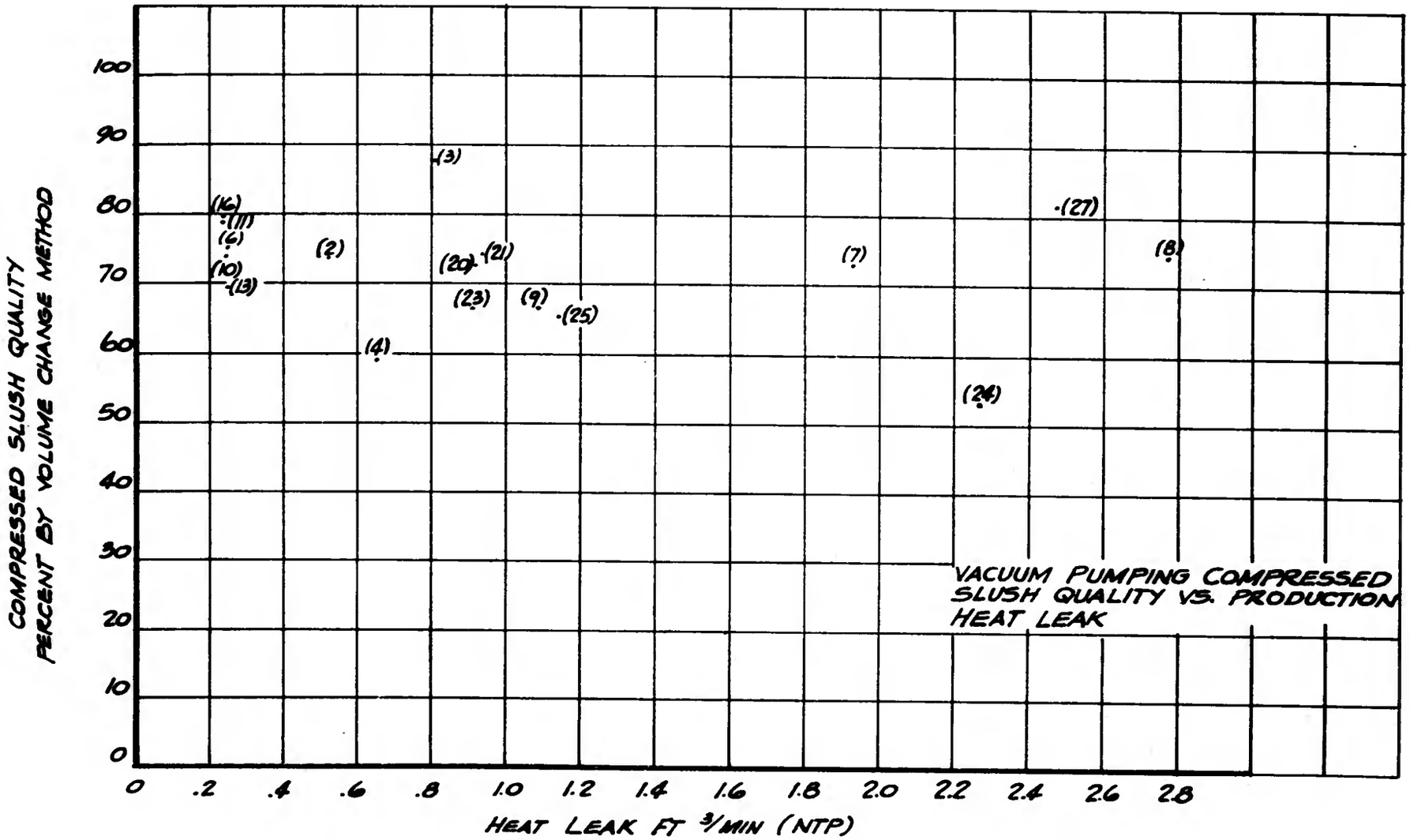

Figure 19. Compressed Slush Quality Versus the Heat Leak

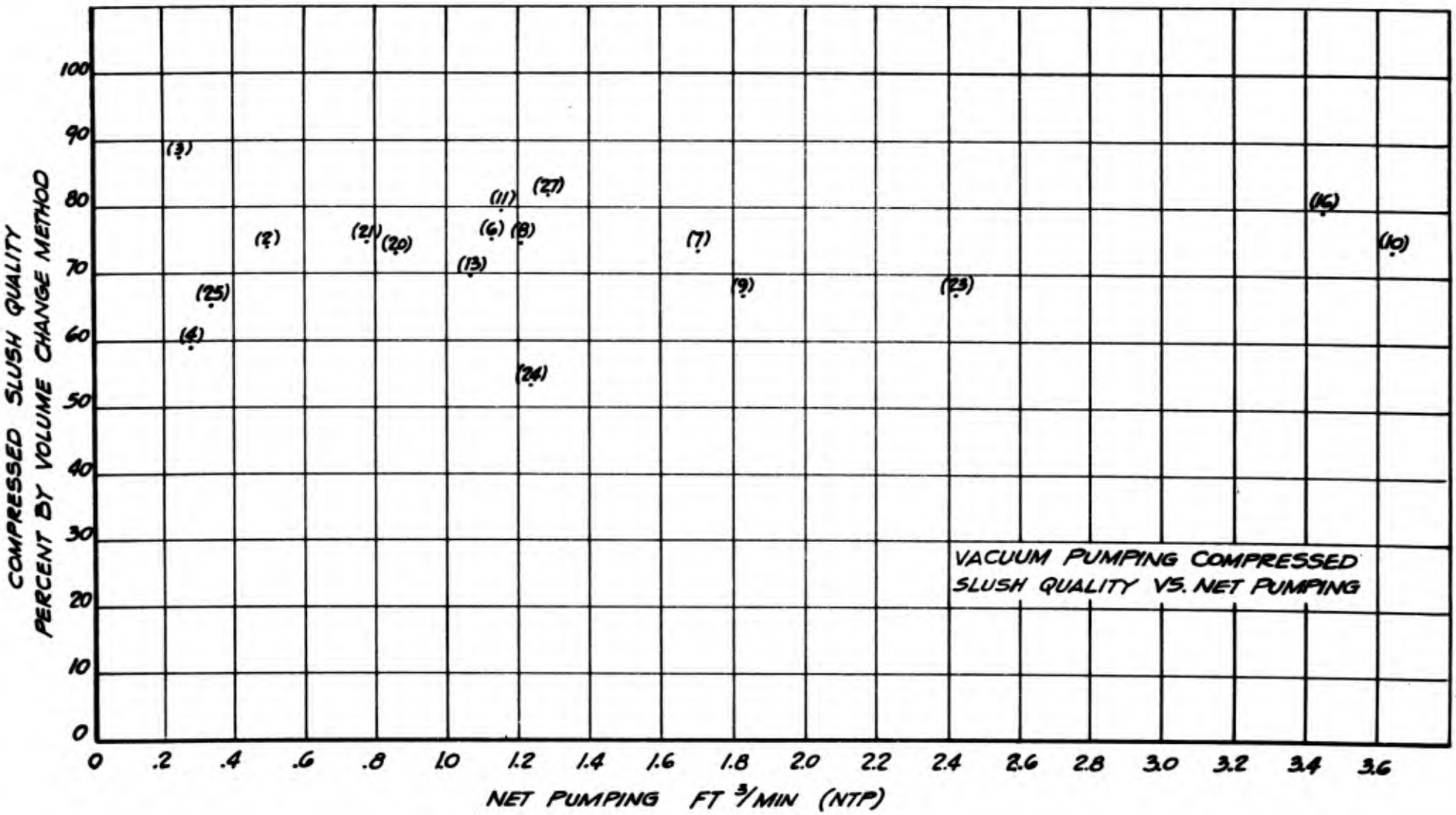

Figure 20. Compressed Slush Quality Versus Net Pumping

3.5.3 Characteristics of Compressed Slush Hydrogen

Compressed slush hydrogen as observed through the periscope appears as a homogeneous, chalky-colored substance as differentiated from triple point liquid hydrogen, which is clear. The graduations on the copper test reservoir wall can be clearly seen through the triple point liquid but cannot be seen through settled or compressed slush. Individual solid particles cannot be detected in the settled or compressed slush; however, solid particles can be observed settling through the triple point liquid when the crust is broken. Also, solid particles can be observed at the conclusion of a run when all the slush is nearly melted and is being agitated with the crust burster.

In the early part of run 5, the crust burster was placed in the triple point liquid and the crust as it formed was broken with the compactor. Solid particles could be seen settling through the triple point liquid and accumulating on the top of the crust burster. Some solid particles passed through the holes in the crust burster accumulating in the bottom of the test reservoir. After an accumulation had occurred on the crust burster, the crust was allowed to grow to a thickness of approximately 5 cm. This crust could not be broken with the compactor. The vacuum pumping was stopped and the crust burster was raised, carrying the accumulated solids and the crust out of the triple point liquid into the vapor space. The crust burster position was held constant and the compactor was forced down on the solids, forming a compressed solid plug 11 cm. thick. No extrusion of solids through the crust burster holes was noted. As melting occurred, liquid could be seen dripping off the crust burster. The crust burster was then lowered back into the triple point liquid, and the compressed solid plug followed as a unit.

In run 14 further observations of the solid were made. For this run the crust burster was located near the bottom of the test reservoir in the triple point liquid hydrogen, while the compactor was located above the liquid level in the vapor space. A crust was formed and allowed to grow downward without breaking until all solids were present in the test reservoir, at which time the vacuum pumping was stopped. An attempt was made to force the crust burster farther down, but only 0.2 cm. travel could be accomplished before it would no longer move. The crust burster was then lifted up approximately 3 cm. before it stopped with only slight resistance, carrying the solids up with it. A gas space was thus formed below the crust burster where the solids had been displaced upwards. Liquid hydrogen could be seen dripping from the crust burster, resulting from the melting of the solids above. The crust burster was then moved down into the liquid, and it was noted that solids were still present in the bottom of the test reservoir. The crust burster was again moved upwards while the compactor was forced down on the solids in an attempt to extrude the solids through the holes in the crust burster. No solids were extruded. The plug of solids was held between the crust burster and the compactor and repeatedly pushed in and out of the triple point liquid with the result that the solids did not break up and pass

through the holes in the crust burster. The solid plug was then moved up into the vapor space, and some voids could be observed in the plug adjacent to the periscope guard dewar. The crust burster was then pushed to the bottom of the test reservoir, and the solid plug followed along with the burster. The triple point liquid above the solids was then agitated with the compactor, causing the liquid to become somewhat cloudy, indicating that solid particles must have broken loose from the top portion of the solid plug. The crust burster was again raised out of the liquid carrying the solids with it into the vapor space. When the crust burster was lowered into the liquid, the solids followed; however, the liquid became cloudy but shortly became clear again, indicating settling of the solids.

In run 15 the same production procedure was employed as in run 14 (all solids produced). During this observation a 16 cm. thick plug of solid crust was raised into the vapor space on the top of the crust burster. The compactor was then forced down while maintaining the crust breaker position. The solid plug was compressed to a thickness of 6.2 cm., indicating the solid crust as formed is rather porous. No extrusion of solids through the holes in the crust burster was noted.

Additional handling of the slush was performed at the conclusion of the 27 numbered production runs. For this work a probe was fabricated from 3/8-inch O.D. thin wall stainless tube with a pointed brass tip (Figure 21). The probe was inserted into the test apparatus through the liquid hydrogen fill quick-connect coupling and had the capability of being translated and rotated in the test reservoir.

The first experiment with the probe was to determine the relative hardness of the crust and the slush. The experiment was started with the probe and compactor located in the vapor space above the triple point liquid and the crust burster located in the triple point liquid. A thin crust was formed by vacuum pumping, and the probe was pushed down through the crust with no noticeable resistance. The probe was raised and a 5 cm. thickness of crust was formed; again, when the probe was pushed through the crust, there was no noticeable resistance. A twenty-centimeter-thick crust was then produced and the probe passed easily through this thickness, also with no noticeable resistance. The crust was grown to a thickness of 31 cm. and was then compressed between the crust burster and the compactor and pushed down into the triple point liquid. The probe was lowered and pushed through the compressed slush with no noticeable resistance. Vacuum pumping was then continued until all solids were produced. Again the probe was pushed through the solids to the bottom of the test reservoir. There was no noticeable resistance while going through the newly formed solids on top of the compressed slush; however, there was a slight resistance and some sticking while going through the solidified compressed slush in the lower portion of the test reservoir.

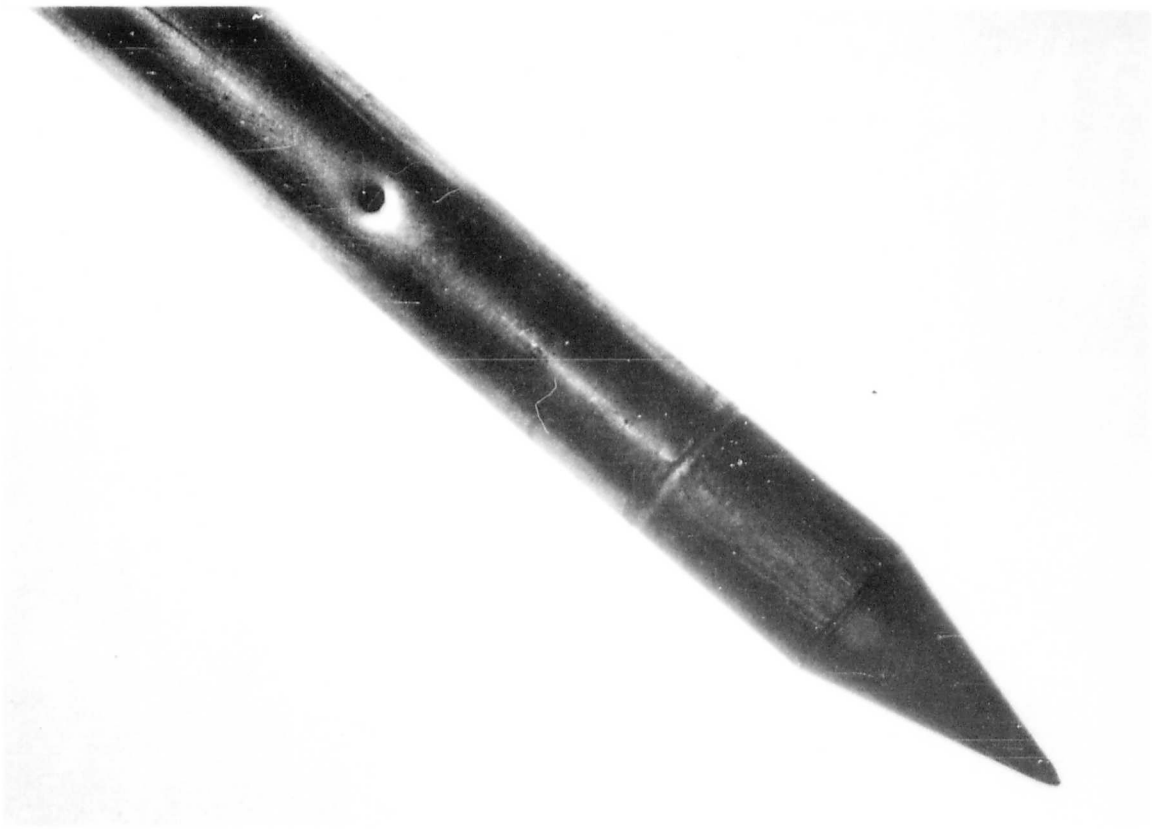

Figure 21. Construction of Pointed Probe

In another run utilizing the probe, the probe was pushed through a thin solid crust which had formed on the liquid surface. A small crystal of solid adhered to the tip of the probe. On the side of the probe, a thin plate of transparent solid started to grow from this crystal (Figure 22). As the crust grew downward, the probe was continuously lowered to be just below the crust. When the probe was raised into the vapor space through the crust, some solids from the crust stuck to the probe. These solids then melted, and the liquid dripped off even though vacuum pumping was still being performed.

When the relatively warm probe was pushed through the crust while vacuum pumping and some solids from the crust would stick to the probe, streams of gas bubbles would originate from this solid and form tubes of solid hydrogen. The solid walls of the tubes were clear and the tube diameter was approximately 1/16 inch (Figure 23). If the tip of the probe was lowered more than approximately three inches below the bottom of the crust, the tubes would not grow; and, if the crust was thicker than approximately 5 cm., the tubes would not grow. When the top of the grown tube reached the bottom of the crust, the tube would fuse to the crust. Also, if there was not solid from the crust stuck to the probe, there would be no bubbles or tubes.

In other cases when the same procedure as above was followed, there would be no bubbles or tubes; however, if a small amount of crust solid was on the tip of the probe, a single radial solid plate or fin would grow. If a considerable amount of crust solid was stuck to the probe, a number of radial solid plates or fins would form. The solid forming the plates or fins was clear with the largest fins or plates being 1/4 to 3/8 inch wide and approximately one inch high. Again as above, no solid would form if the probe was more than three inches below the crust or if the crust was more than 5 cm. thick (Figure 22).

It is not known why tubes will form in some runs, while in others tubes will not form but rather solid plates or fins will form.

3.5.4 Density of Porous Hydrogen Crust Formed by Vacuum Pumping

During vacuum pumping production Runs 11 through 16, the hydrogen crust was allowed to grow without breaking until all the hydrogen in the test reservoir was in the form of a porous crust. By utilizing the data which was recorded during the conduct of the production and compression of these runs, it is a simple matter to compute the as-formed density of the porous hydrogen crust. To make the calculation it is only necessary to know the volume occupied by the crust (computed from the crust level) and the mass of hydrogen present (computed from the all-melted triple point liquid level).

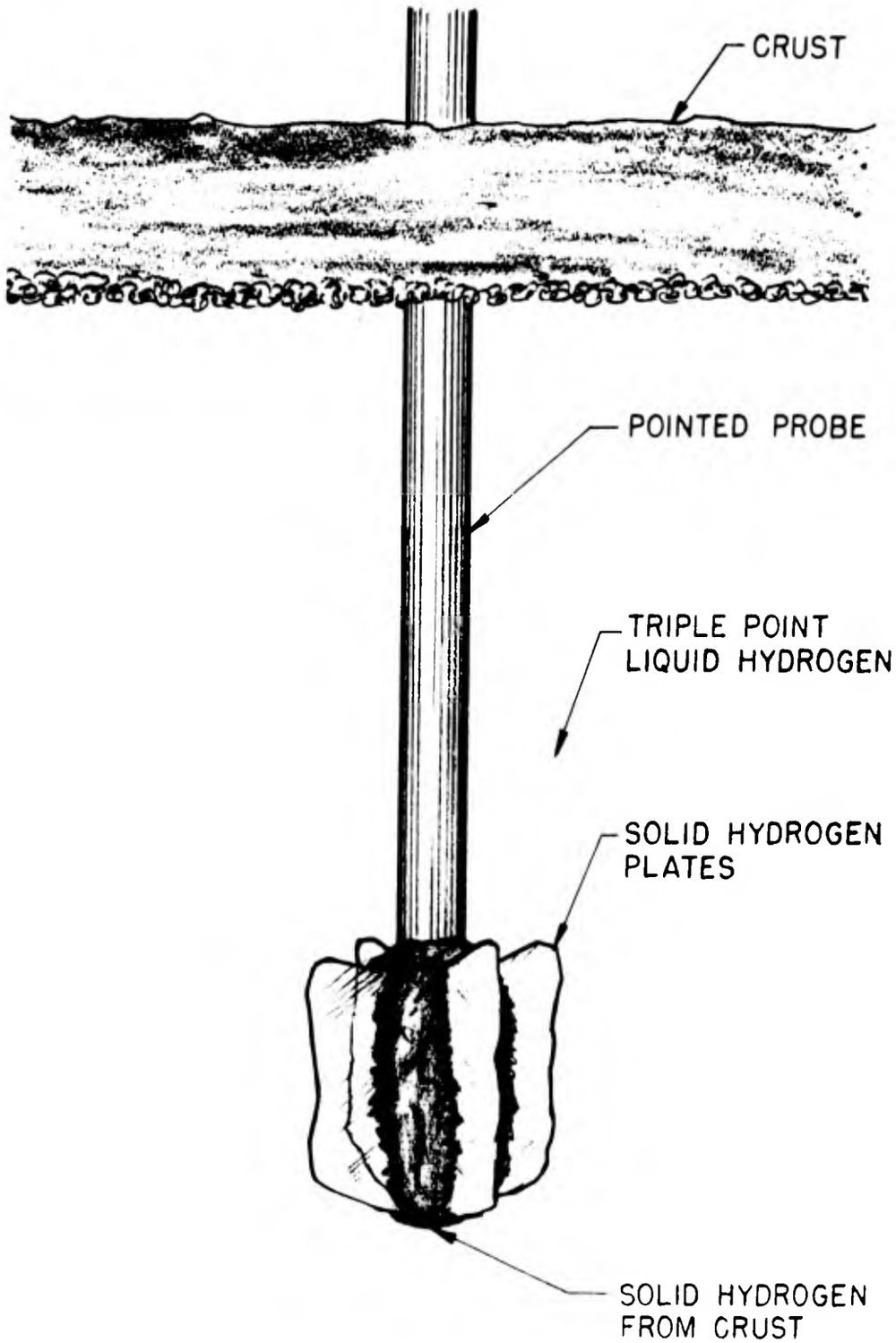

Figure 22. Solid Plates Growing on Pointed Probe

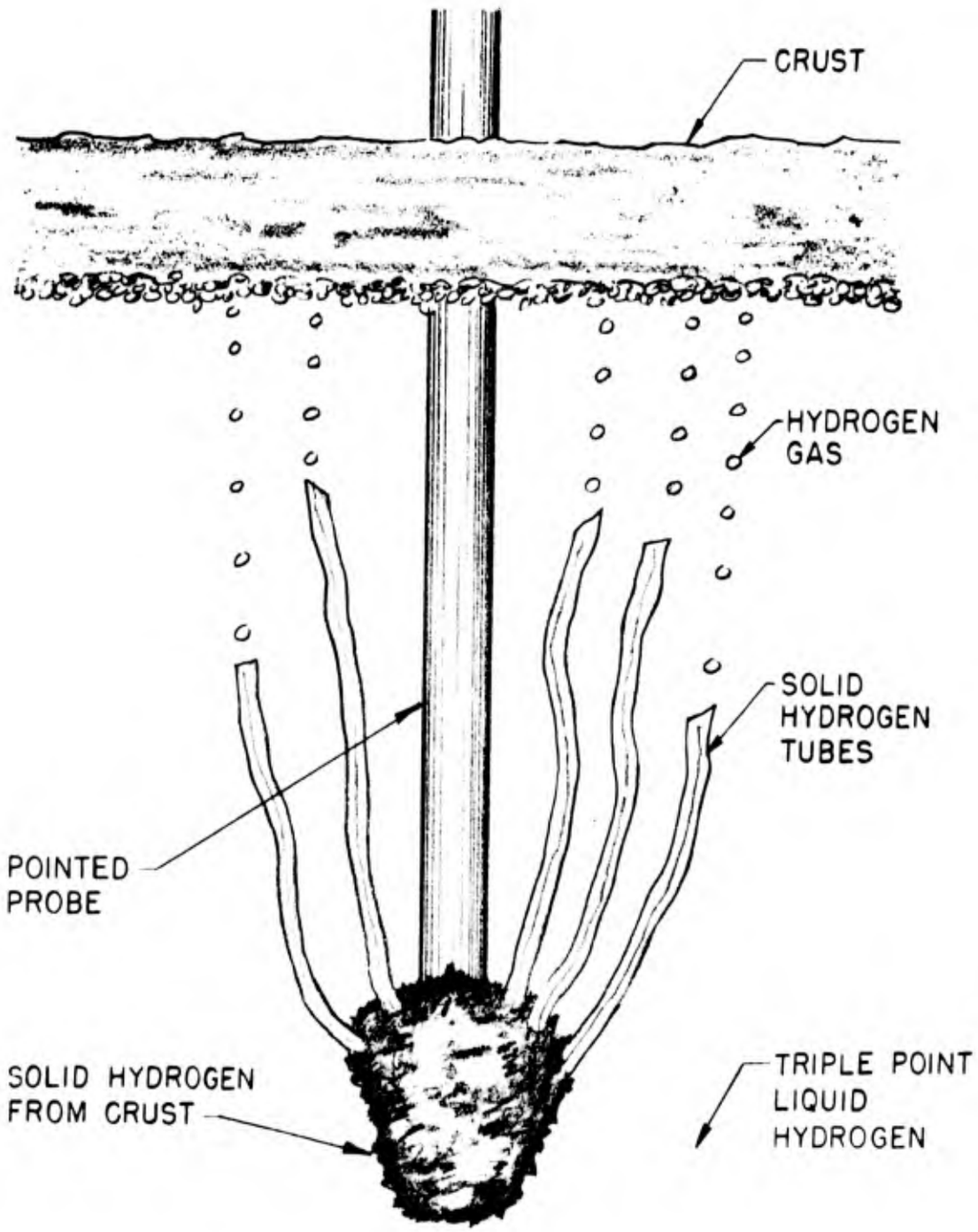

Figure 23. Solid Tubes Growing From Pointed Probe

Table 2 presents the test data extracted from runs 11-16 which were used to calculate the hydrogen crust density. The calculated crust density for each run is also presented and is calculated by dividing the mass of hydrogen present by the volume occupied by the hydrogen crust.

TABLE 2

DETERMINATION OF HYDROGEN CRUST DENSITY

Run	Hydrogen Crust Level (cm.)	Hydrogen Crust Volume (ft^3)	Melted Crust Liquid Level (cm.)	Triple Point Liquid Volume (ft^3)	Mass of Hydrogen Crust (lb.)	Density of Hydrogen Crust (lb/ft^3)
11	54.0	0.8345	33.1	0.5565	2.6756	3.2062
12	38.6	0.6296	23.0	0.4221	2.0294	3.2233
13	40.0	0.6482	27.1	0.4766	2.2916	3.5353
14	39.4	0.6403	28.4	0.4939	2.3746	3.7068
15	40.5	0.6549	29.0	0.5019	2.4131	3.6847
16	59.0 to 53.0 46.8 to bottom	0.8185	40.8	0.6589	3.1679	3.8704

Average Hydrogen Crust Density = 3.5378

The average hydrogen crust density produced by vacuum pumping in runs 11-16 was determined to be 3.5378 lb/cu. ft. The density of solid hydrogen at the triple point is 5.412 lb/cu. ft. The hydrogen crust produced during this experimental work is therefore 65.4 per cent solid and 34.6 per cent voids.

It is perhaps interesting to compute the slush hydrogen quality that would be produced if the void volume of the hydrogen crust could be filled with triple point liquid hydrogen without causing any melting of the solids. The total mass of a one-cubic-foot volume of this slush would consist of the mass of the solid crust which would be 3.5378 pounds plus the mass of the triple point liquid hydrogen required to fill the voids, which would be ($0.346\ ft^3 \times 4.8079\ lb/ft^3$) = 1.6635 pounds. The total mass would therefore be 5.2013 pounds. Utilizing Equation 4 the resulting slush hydrogen quality would be:

$$\text{Quality} = \frac{M_g}{M_g + M_l} \qquad (4)$$

$$\text{Quality} = \frac{3.5378}{5.2013} \times 100 = 68\%$$

3.6 SLUSH HYDROGEN PRODUCTION BY HELIUM GAS INJECTION FOLLOWED BY VACUUM PUMPING

During prior slush hydrogen experimental work, it was observed that, when helium gas is injected into triple point liquid hydrogen, hollow tubes of solid hydrogen are formed. Also, if vacuum pumping was continued on the triple point liquid, the tubes would grow in diameter as solids would form on the outside surfaces.

For this phase of the slush hydrogen production experiments, the conditions leading to the growth of tubes were to be determined. To be determined in particular were the following: if continued vacuum pumping is required to cause tubes to grow, if these tubes could be broken and settled slush would be produced, or if the tubes would form in settled slush and continue to grow without vacuum pumping.

In our apparatus it must be concluded that vacuum pumping is required to cause tubes to continue to grow after the initial formation. It has been determined that only a small initial flow of helium gas is required to form tubes. The solid of the tubes is rather soft since the tubes tend to bend under a load. If tubes are grown into columns by vacuum pumping, the solid depositing on the outside of the tubes looks similar to that of the crust, which is formed during straight vacuum pumping. The rate of growth of the columns and the crust while vacuum pumping appears to be about equal on a volume produced basis. Compressed slush produced from the columns has a quality slightly lower than compressed slush made by breaking the crust. When the columns were grown in settled slush, in an attempt to upgrade the quality, the resulting compressed slush has a quality from 60 to 85 per cent, the same quality range as obtained from the freezing or melting vacuum pumping production process.

3.6.1 Experimental Production Procedure

The experimental apparatus components utilized for this production method are discussed in Section 3.2.1.2.2. The procedure for the formation of solid tubes with cold helium gas was as follows:

After both the guard chamber and the test reservoir had been filled with liquid hydrogen, the injection lance was inserted into the test reservoir through the liquid hydrogen quick-connect filling coupling. During insertion, the lance (Figures 10 and 11) was purged with a small flow of helium gas and care was taken to get the lance through the proper holes in the compactor and crust burster by observing with the periscope. The lance was lowered until about six inches of the flexible tube were resting on the bottom of the test reservoir. Sharp bends in the flexible tube were avoided by using the compactor and crust burster as guides. The lance was then connected to the helium source with the valve closed.

The cold helium gas was obtained by vaporizing liquid helium stored in a standard LHe-101 container.* The vent valve of the test reservoir was then closed, and vacuum pumping started. Pumping was continued until the liquid hydrogen was cooled to the triple point when solid hydrogen crystals would begin to form, as seen through the periscope. At this time, the valve on the helium source was opened, allowing helium gas to flow through the lance. By the time the helium reached the holes in the flexible tube, any liquid helium present in the flow was vaporized to cold gas. Even when the diameter of the holes was increased to 1/8 inch, no liquid helium reached the flexible tube. The helium flow rate was regulated with the liquid helium withdrawal valve. To obtain helium flow rates, weight loss of the LHe-101 cylinder and time were measured. However, the weight loss is very small, making the flow rate measurements highly inaccurate.

The procedure for producing solid tubes by injection with warm helium gas is essentially the same as stated above except that the source of the helium gas was a "K" - type cylinder. With the warm gas more accurate flow rates were possible. A small rotameter, located upstream of a throttle valve, was utilized to measure the flow rate.

3.6.2 Solid Hydrogen Tube Formation

Solid hydrogen tubes were formed in the triple point liquid hydrogen by the initial helium flow. As soon as tubes had been formed, the helium bubbles disappeared, since all the helium gas flowed up inside the hollow tubes. If vacuum pumping was continued after the triple point pressure had been reached, a crust of solid hydrogen would also form on the surface of the liquid hydrogen (Figures 24 and 25).

With cold gas injection and a hole diameter of 1/64 inch, tubes of about 1/16-inch diameter would form. Surging in the helium lance caused vibrations, which would break the thin tubes. However, the tubes would quickly form again. The end of a tube which was growing constantly upward emitted bubbles. Evaporation of liquid hydrogen by these helium bubbles caused more solid hydrogen to form at the end of the tube. Thus, the tube would grow until it reached the crust at the surface of the liquid hydrogen. The upper end of the tube would attach itself to the crust, making the tube less sensitive to vibration. In order to retain a tube for a reasonable time, it had to have a larger diameter. This was accomplished by increasing the hole diameter in the flexible tube on the lance. With a 1/16-inch hole diameter, some tubes would reach a diameter of 1/8 inch. With the 1/8-inch hole, most tubes would be 1/8 inch in diameter, occasionally reaching 1/4-inch diameter.

With warm helium gas injection, tubes again formed with the initial helium flow. However, since no surging occurred, about ten tubes were grown with warm gas compared to about four with cold gas. Whether the hole diameter was 1/32 inch or 1/16 inch, most tubes would range between 1/16-inch and 1/8-inch diameter.

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* Linde Division 100-liter liquid helium container.

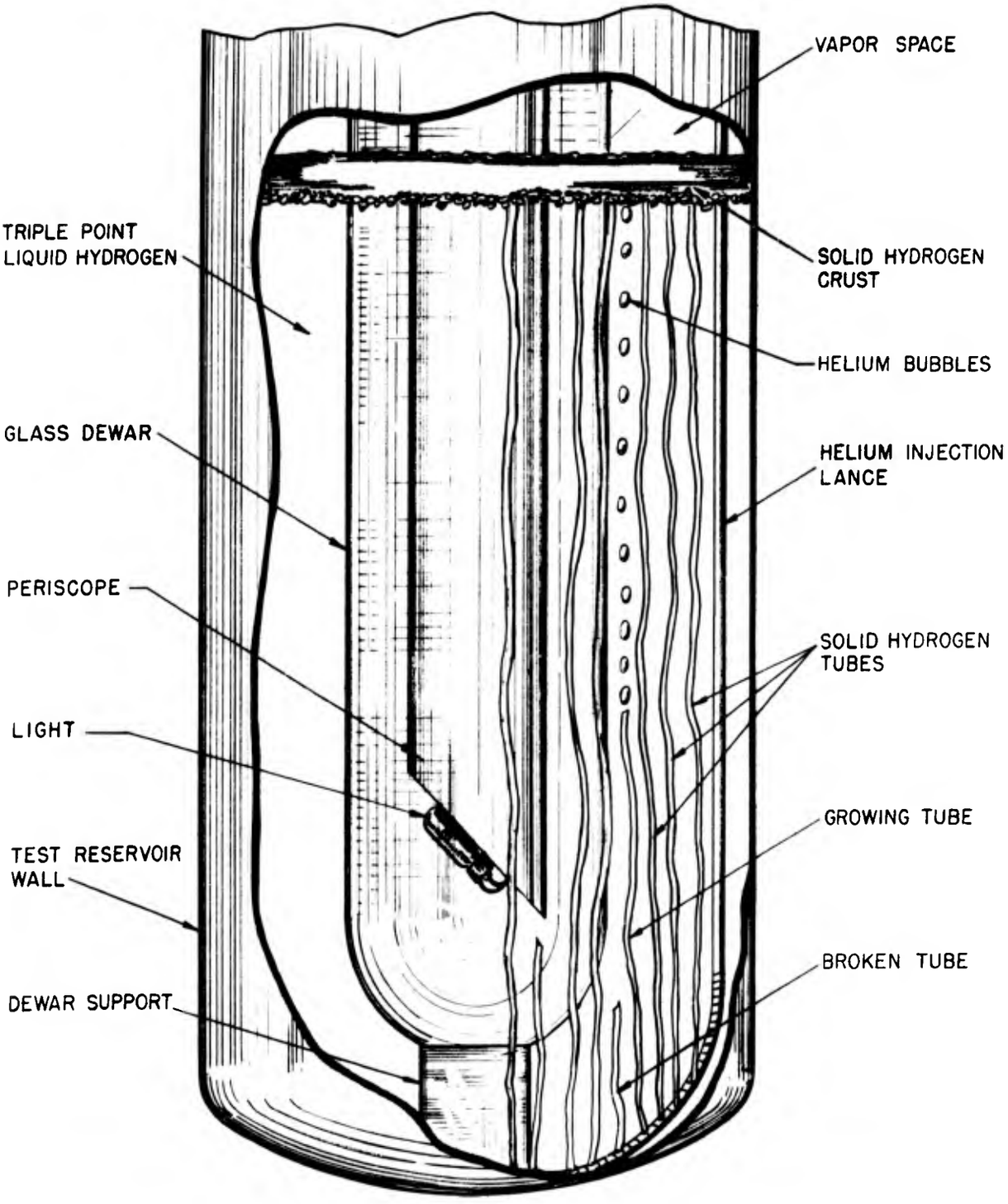

Figure 24. Solid Hydrogen Tubes Formed by Gaseous Helium Injection

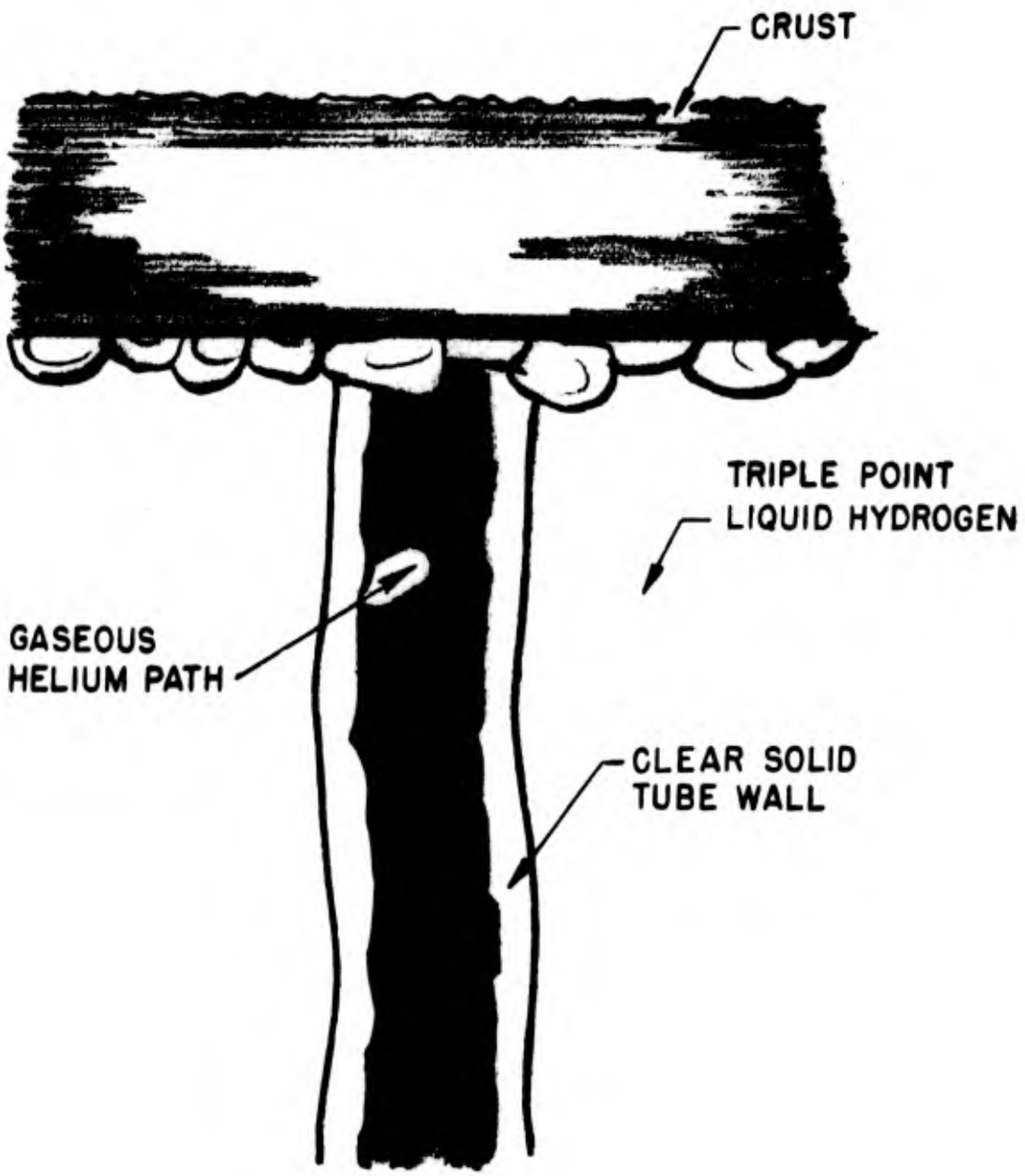

Figure 25. Enlarged View of Solid Hydrogen Tubes

The solid hydrogen comprising the tube was transparent. The outside surface of the tube wall visually appeared to be smooth. The inside wall had a fuzzy, white appearance, caused by the deposition of small plates of solid hydrogen. For the initial tubes the wall thickness was quite thin. The tubes, though vertical, were not straight or of uniform diameter throughout their length. Some tubes would grow along the glass wall of the periscope dewar, forming half a cylinder of solid hydrogen.

When a tube broke off, it immediately reformed if helium gas was flowing up through the tube. Once a tube was formed, continuation of helium flow was not necessary to retain the tube. However, should the tube break for any reason and no helium gas was flowing through it, liquid hydrogen would fill the tube. The fuzziness of the inside wall disappeared for filled tubes, clearly showing small solid plates which had become transparent. The broken tube could remain in this position, or it might melt or break up into small pieces of solid.

3.6.3 Breaking of Solid Hydrogen Tubes

When tubes had been formed in the liquid and the vacuum pumping rate and helium flow rate adjusted so as to maintain triple point vacuum, attempts were made to break the tubes in order to produce settled slush. Under these conditions, if the tubes would break, they would immediately be reformed by the continuing helium flow. However, when the crust burster was forced down, the tubes did not break but rather bent. As the burster was moved down, a plug of solid would be formed just below the burster, consisting of fused tubes. Only when the burster was moved very fast did the top section of the tubes break off. Figure 26 shows solid tubes being compressed after the gaseous helium flow has been terminated.

Since the crust burster has holes, it could be left in the liquid, allowing the helium bubbles, forming tubes, to go through the holes. When the burster was moved upward, the tubes were severed just below the burster. If the crust at the surface was thin, tubes and crust would be lifted into the gas space. If the crust was thicker, it would not move and the tubes would bend to finally form a plug of fused tubes which had been pressed against the crust.

By rotating the injection lance, tubes could be broken at the holes in the end of the lance. The turbulence created by the helium escaping from the holes reduced the tubes to small pieces of solid. At times, tube sections of a maximum length of 5 cm. could be observed as they settled down through the liquid. However, the longest tube sections which were seen in the resulting slush were, at most, 2 cm. long. The slush consisted mostly of small plates and needle-shaped particles, generally of smaller size than obtained by breaking the crust. As stated before, new tubes were quickly formed again. But the period of turbulence lasted long enough to destroy the tubes previously formed.

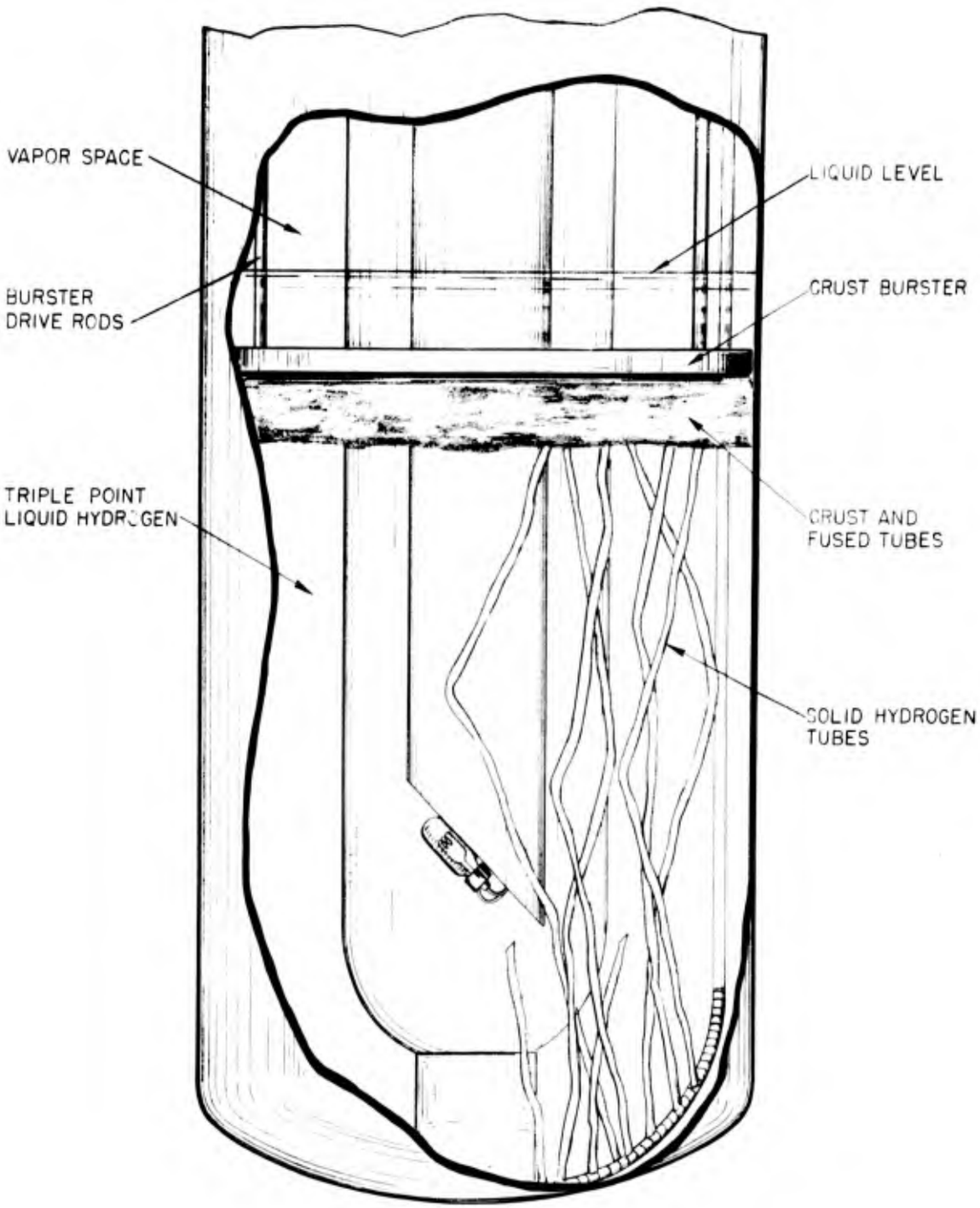

Figure 26. Compression of Solid Hydrogen Tubes

3.6.4 Growing Solid Tubes into Columns

Once the liquid hydrogen was at the triple point and some tubes had been formed by gaseous helium injection, three different growth techniques could be followed:

1. Vacuum pumping could be stopped, but the helium flow continued.
2. Pumping could be continued, but helium injection stopped.
3. Both vacuum pumping and helium injection could be continued.

The objective now was to increase the wall thickness of the tubes, so that eventually all the liquid hydrogen would be solidified during the lateral growth of the tube walls.

When helium injection was continued and vacuum pumping stopped, the vacuum which was initially at triple point pressure would drop until one atmosphere was reached and the vent valve was opened. The tubes, which had formed with the initial helium flow, would melt as the vacuum decreased. By the time a vacuum of 20 in. mercury was reached, all tubes had melted. This applies to tubes formed by cold or warm helium injection. When cold gas injection at high flow was used, the vibrations of the lance and the rapidly dropping vacuum prevented the formation of any tubes. The vacuum in the test reservoir could decrease from the triple point, generally somewhat above 27 in. of mercury, to about 25 in. of mercury without causing any melting of the tubes.

If helium injection was stopped once the tubes had formed and the vacuum pumping was continued, solid hydrogen would deposit on the outside wall of the tubes. This new solid had a white color and seemed to consist of small flake- and needle-shaped particles. Tubes would grow into thick columns which would, in time, replace all the liquid hydrogen (Figure 27). The initial tubes could be closely spaced to form a bundle, or they could be far apart. On continued pumping, a bundle of tubes would soon merge to form a thick white column. The column would keep on growing, perhaps merging with other columns, until all solid hydrogen had been produced. However, as the column was growing in diameter, the crust of solid hydrogen at the surface would also grow in a downward direction. Both the crust and the column appeared to grow at about the same volume rate. The higher the vacuum pumping rate, the faster both crust and column would grow. Pumping rates from about 1.5 to 4 cfm were used. The solid hydrogen of the crust and the column had the same appearance.

A single tube could form a growing column as described above. Often, however, the tube would melt and break at some point, or it would grow for only a part of its length. The tube might grow along the upper half of its length, just as if it were forming a column. But at some point along the tube, growth would cease and the tube would eventually melt at this point, leaving a bulge extending downward from the crust and the stub of a tube in the liquid.

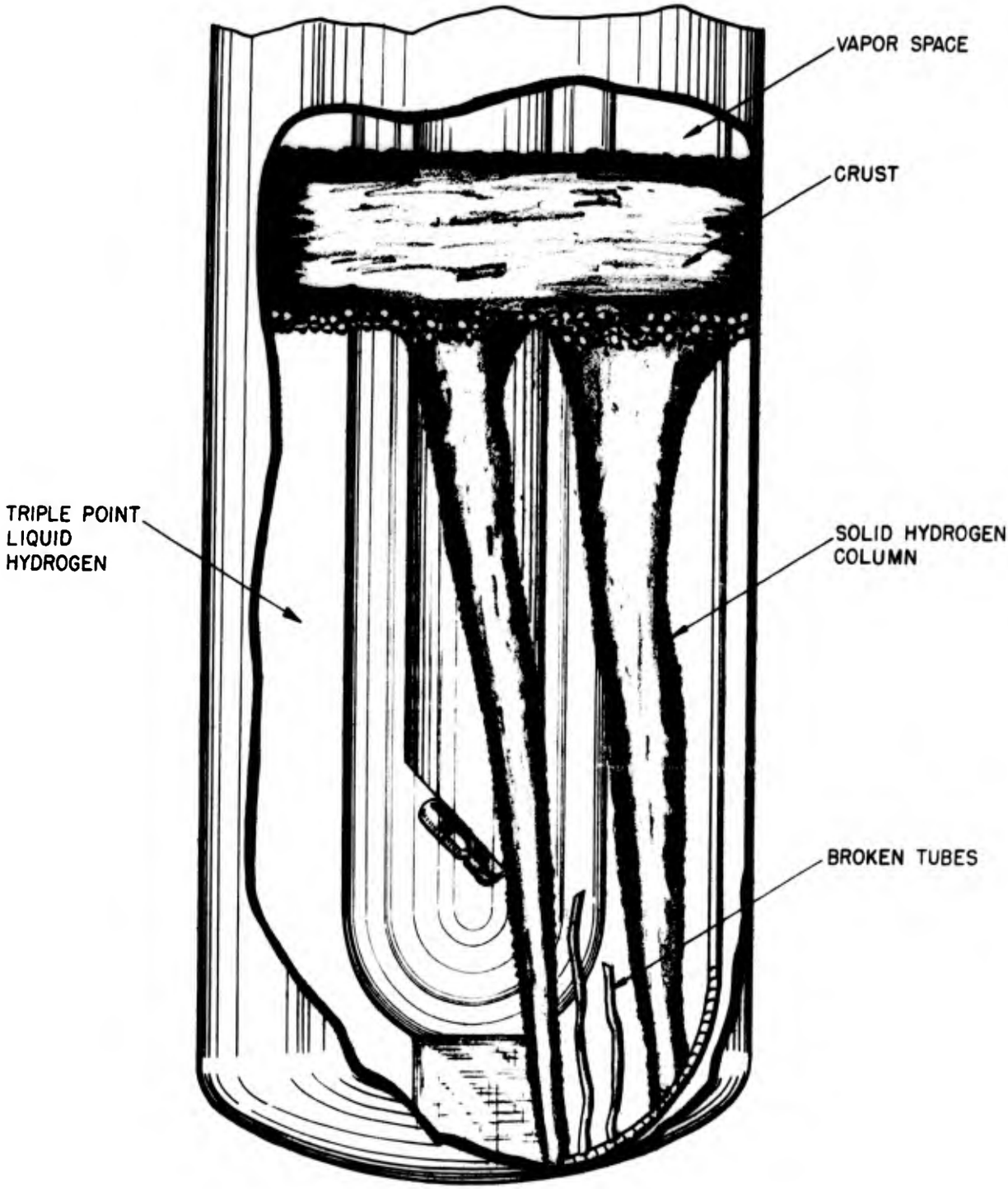

Figure 27. Growth of Solid Hydrogen Tubes into Columns

Several quality determinations were performed on slush resulting from solid hydrogen obtained from growing tubes. When all the liquid hydrogen had been converted to solid, vacuum pumping was stopped and the crust burster and compactor were used to compress the solid. Heat leak caused some of the solid to melt, and as soon as the liquid level was above that of the compressed solid, the hydrogen below the level of the compactor could be regarded as compressed slush. For pumping rates of 1.5 to 4 cfm and a heat leak up to 20 watts at 4 cfm, qualities ranged generally from 55 to 70 per cent. Table 3 presents the results of the individual production runs.

The last method for growing tubes comprised vacuum pumping with continued helium flow. The helium flow and vacuum pumping rate were adjusted to keep the pressure at the triple point. The helium flow rate was limited by the vacuum pump exhaust wet drum meter capacity (5 cfm). Since the helium flow had to be closely controlled, warm helium was used for most runs. Tubes would not grow into columns except when the pumping rate was high, 4 cfm, and helium flow rate low, 22.7 cfh. When the ratio of pumping rate to helium flow was lower, the tubes remained separated in the liquid. In this condition, the tubes, usually of 1/16-inch to 1/8-inch diameter, would get thicker, perhaps to 3/8-inch diameter, with most of the gain resulting from an increase in wall thickness. At this point, the wall thickness was about equal to the inside diameter of the tube. With continued helium injection, the outside diameter of the tube would remain the same, but the inside diameter would increase at the expense of wall thickness. In the end, the wall thickness would be as it was at the start, but the tube would have a larger diameter. Although tubes would get somewhat thicker when cold helium gas was used, larger diameters were obtained with warm helium gas. For this type of experiment, triple point pressure had to be maintained. At a pumping speed of 1.5 cfm, a helium injection rate of 22.7 cfh could be used; at a pumping speed of 4 cfm, the injection rate could be increased to 68.0 cfh, while maintaining triple point pressure.

3.6.5 Helium Injection into Settled Slush

Gaseous helium was also injected into settled slush in the hope of increasing the quality of the slush. As before, the injection lance was inserted at the beginning of the run and vacuum pumping was started. The crust which was formed at the triple point, as pumping was maintained, was continually broken to produce settled slush. A layer of liquid was kept above the settled slush so that the tubes could be seen. Helium injection was then started and, as before, tubes were formed with the initial flow and could be seen emerging from the settled slush. In order to grow the tubes into columns and convert the remaining liquid to solid, vacuum pumping was continued and helium injection stopped. When all the liquid had been solidified by vacuum pumping, the solid was compressed and allowed to partially melt to form compressed slush. The quality resulting from this production method generally ranged from 55 to 85 per cent and is presented in Table 3.

3.6.6 Compressed Slush Hydrogen Quality

Table 3 presents the results of the experimental runs which involved a slush hydrogen compression and subsequent quality determination. Only the volume-change slush hydrogen quality determination method could be used since the vacuum pump was pumping helium gas as well as hydrogen gas. Within the scope of this work, it was not possible to determine the relative volumes of each gas as they were pumped by the vacuum pump, which would be necessary to employ the vapor-volume quality determination method.

TABLE 3

SUMMARY OF HELIUM INJECTION SLUSH PRODUCTION

Run	Pump Rate ft^3/min (NTP)	Heater watts	Heat Leak ft^3/min (NTP)	Type of Run	Compressed Slush Quality-Per Cent Volume-Change Method	Melted Slush Level cm.
1	---	NO	0.443	T	36.0	17.35
2	1.48	NO	0.443	S	69.2 78.9 76.9 79.5 Av. 76.1	27.5 20.15 25.0 14.8
3	2.94	NO	0.443	S	55.3 56.4 61.2 Av. 57.6	18.7 11.8 16.0
4	3.70* 2.80	NO	0.443	X	72.8 76.9 75.5 75.9 78.4 80.1 Av. 76.6	46.8 28.5 42.4 21.9 34.7 13.5
5	3.58 2.74	20	1.569	X	58.8 62.4 62.7 61.9 Av. 61.5	29.3 22.05 25.5 14.3
6	1.94 2.03	NO	0.443	X	78.0 86.4 80.1 93.1 83.3 Av. 84.2	41.9 29.6 39.4 23.9 33.2
7	2.07 2.01	5	0.725	X	83.9 79.5 82.7 Av. 82.0	20.0 13.7 16.1

Run	Pump Rate ft^3/min (NTP)	Heater watts	Heat Leak ft^3/min (NTP)	Type of Run	Compressed Slush Quality-Per Cent Volume-Change Method		Melted Slush Level cm.	
8	---	NO	0.443	T	64.1	65.0	28.25	14.6
					68.7	60.1	24.45	10.5
					68.1		18.8	
					Av. 65.2			
9	1.99	10	1.006	X	66.0	72.2	34.7	22.85
	1.93				70.9	74.6	31.7	18.8
					72.0	74.3	28.6	15.3
					71.8		24.9	
					Av. 71.7			
10	3.77	NO	0.443	S	53.5	67.0	43.3	23.3
					60.7	68.5	38.5	19.9
					63.9	68.2	33.3	14.8
					66.2		27.8	
					Av. 64.0			
11	3.72	20	1.569	S	44.8	59.2	43.3	27.6
					56.4	57.0	40.6	24.2
					57.5	58.3	38.0	19.55
					57.5		34.9	
					Av. 55.8			
12	3.66	NO	0.443	S	37.1	59.0	47.0	39.9
					50.1	66.6	44.9	34.3
					57.0		43.2	
					Av. 54.0			

T - Compressed tubes.

S - Tubes, which have been grown by vacuum pumping to form all solid.

X - Tubes, which have been grown in settled slush until the remaining liquid had been converted to solid.

* - First number denotes pumping rate while making slush; second number denotes pumping rate while growing columns.

Notes for Table 3

Cold helium gas from a LHe-101 container was used for all runs except run 12, for which warm helium gas was injected from a "K" cylinder.

Run 1 Cold helium gas was injected slowly while the pressure was kept at triple point level by continued vacuum pumping. Many tubes of 1/16 to 1/8 inch diameter were formed; also a thin crust formed at the liquid level.

As the crust burster was moved down in order to break the tubes, the tubes bent but did not break and a layer of solid, consisting of compacted tubes, formed ahead of the crust burster. Tubes would also form above the burster if it is left below the liquid level. The tubes were periodically compressed, and the quality of the resulting slush was calculated. The low melted slush level may be responsible for the low quality which was obtained, since only a few compressions were made.

Run 2 Cold helium gas was injected slowly. As soon as tubes had formed, the helium injection was stopped and vacuum pumping was continued. Closely spaced tubes merged to form a white-colored solid column. The column grew horizontally while the crust grew downward until all the liquid was converted to solid. At this time the vacuum pumping was stopped and, as the solid hydrogen partially melted, the compression was performed. When the liquid level rose above the solid level, the quality of the compressed slush was determined.

Run 3 Same as Run 2 except a high vacuum pumping rate was used.

Run 4 Settled slush was produced by vacuum pumping and breaking the resulting crust. A layer of triple point liquid was left above the settled slush to observe the tubes. Cold helium gas was injected into the settled slush and tubes were grown. The tubes could be seen emerging from the slush into the triple point liquid. When tubes had been formed, helium injection was stopped. Vacuum pumping was then continued until the tubes and crust had converted the remaining liquid in and above the slush to solid. When this solid partially melted, it was compressed and, as the liquid level rose above the solid, the quality of the resulting slush was determined.

Run 5 Same as Run 4 except additional heat at a rate of 20 watts was supplied by the heater wire.

Run 6 Same as Run 4 except lower vacuum pumping rates were used during and after slush formation.

Run 7 Same as Run 6 except that additional heat was supplied at the rate of 5 watts by the heater wire.

Run 8 Same as Run 7; however, high melted slush levels were obtained, making this run perhaps more accurate than the first run.

Run 9 Same as Run 6 except that an additional heat input of 10 watts was supplied by the heater wire.

Run 10 Same as Run 2 except that a higher vacuum pumping rate was used.

Run 11 Same as Run 10 except that additional heat leak of 20 watts was supplied by the heater wire.

Run 12 Same as Run 10; however, warm helium gas was injected to produce the initial tubes.

3.7 SLUSH HYDROGEN PRODUCTION BY LIQUID HELIUM FREEZING

3.7.1 Experimental Production Procedure

Slush hydrogen production by liquid helium freezing was experimentally performed utilizing two production method variations, namely, cooling with indirect heat exchange and cooling with direct heat exchange. The indirect heat exchange method employs the flow of liquid helium through the heat transfer coil (Figure 3) which is permanently attached to the test reservoir. With this cooling method, the liquid helium does not come in contact with the liquid hydrogen. The direct heat exchange method employs the flow of liquid helium directly into the liquid hydrogen, thus achieving direct contact between the liquid helium and the liquid hydrogen.

For both production methods, the test reservoir is initially filled with liquid hydrogen to a minimum level of 70 cm. with levels up to 100 cm. being used on occasion. The importance of a high slush level on the accuracy of the slush hydrogen quality calculation is discussed in Section 3.4.1.1. After the initial fill, the liquid hydrogen was vacuum pumped to produce triple point liquid as evidenced by the formation of solids on the liquid surface. Starting the slush production with triple point liquid hydrogen conserves much liquid helium.

3.7.1.1 Indirect Heat Exchange

For the indirect heat exchange method, an LHe-101 liquid helium storage container, which was on a scale, was connected to the heat transfer coil on the test reservoir by means of a standard vacuum-insulated transfer line. The flow of liquid helium through the heat transfer coil was controlled with a globe valve on the gaseous discharge line while pressure for the liquid transfer was maintained between 2 and 3 psig with gaseous helium from a "K" type cylinder. Liquid helium flow rates, which generally ranged from 0.2 to 0.3 lb/min., were determined by measuring the weight loss of the storage container during the liquid transfer. When the heat exchanger coil was not in use, it was kept under gaseous helium pressure to prevent air leakage and subsequent plugging.

With the liquid hydrogen in the test reservoir at the triple point, the flow of helium through the heat exchanger coil was started. For the first few minutes of the flow, while the transfer line was being cooled, the hydrogen in the test reservoir would be heated slightly, causing the pressure to rise. To bring the hydrogen back to triple point conditions, vacuum pumping was performed. Continued flow of liquid helium would also cool the hydrogen; however, the vacuum pumping allowed some liquid helium to be conserved.

As the flow of liquid helium was continued, hydrogen gas would start to condense on the wall of the test reservoir above the liquid level, and soon a thin film of solid hydrogen formed along the wall just above the liquid level. Next a crust of solid hydrogen would grow across the surface of the liquid from the test reservoir wall to the glass periscope guard chamber dewar and simultaneously solid hydrogen formed on the test reservoir wall below the liquid level. At this point the procedure was varied for the different runs. For some runs, breaking and scraping was performed for as long as possible during the solid production, and for other runs the solid was formed without any breaking or scraping. At the conclusion of the liquid helium flow, the slush was compressed with the compactor. Compression was maintained during melting to get as many data points for the calculation of the compressed slush quality as possible. At the conclusion of the compression, the remaining solids were melted and the final melted level determined.

3.7.1.2 Direct Heat Exchange

For the direct heat exchange method, liquid helium was injected into the liquid hydrogen through the vacuum-insulated transfer line used normally to fill the test reservoir with liquid hydrogen. This transfer line, which is inserted through the fill coupling in the top flange, was extended to reach nearly to the bottom of the test reservoir by brazing a 1/4-inch O.D. stainless steel tube to the outlet.

With the hydrogen in the test reservoir at the triple point, the vacuum-insulated valve on the liquid helium storage container was opened, starting the flow of helium into the hydrogen. The pressure in the test reservoir rapidly increased to atmospheric and the vent valve to the stack was opened. Further cooling of the hydrogen occurred at one atmosphere helium pressure, and it was found that low helium flow rates in the order of 0.3 lb/min. delivered refrigeration at about the same rate as high helium flow rates. The first helium flow into the test reservoir caused violent bubbling while the transfer line was being cooled. As soon as cooldown was achieved, solid hydrogen started to form at the end of the transfer line. Within a short time, a bulb of solid had formed with a number of solid tubes extending from the bulb to a crust which had formed on the liquid surface. With continued helium flow, the growth rate of solid bulb decreases as most of the cold helium gas traveled up through the tubes into the vapor space. At the conclusion of the helium flow, the test reservoir pressure was reduced by vacuum pumping and the knife edge scraper was used to break up the solid formation into settled slush upon partial melting. Data points were taken to determine the settled slush quality, and then the settled slush was compressed with the compactor. At the conclusion of the compression, the remaining solids were melted and the final melted level determined.

3.7.2 Quality Results of the Experimental Runs

3.7.2.1 Indirect Heat Exchange Runs

A total of 13 quality determination runs were performed by the indirect heat exchange method. Table 4 is a summary of the individual runs that presents the liquid helium flow rate, mass of hydrogen present, compressed slush quality, and the melted slush level. The procedure and observations of each run are discussed briefly in the notes following Table 4.

TABLE 4

SUMMARY OF INDIRECT LIQUID HELIUM FREEZING SLUSH PRODUCTION

Run	Liquid Helium Flow Rate (lb/min)	Mass of Hydrogen Present (lb)	Compressed Slush Quality-Per Cent Volume-Change Method	Melted Slush Level (in.)
1	0.26	5.09	76.4 71.6 77.2 76.1 76.1 74.9 74.2 Av. 75.2	68.9 52.7 63.4 44.3 59.7 39.75 56.05
2	0.18	3.90	54.8 68.4 61.2 63.1 68.2 Av. 63.1	48.5 20.0 40.3 15.3 28.6
3	0.26	4.87	89.4 83.0 89.0 80.8 87.9 77.7 86.0 Av. 84.8	61.1 37.7 54.8 33.9 50.5 29.8 45.5
4	0.28	4.00	83.7 74.3 81.8 67.6 Av. 76.8	47.0 27.4 32.9 23.0
5	0.32	3.70	72.9 85.3 88.5 88.5 Av. 83.8	43.0 25.9 30.7 17.5
6	0.25	4.82	96.1 92.2 96.7 90.4 96.1 87.8 92.9 Av. 93.2	60.85 42.8 53.5 38.35 51.05 34.1 47.3

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2	0.18	3.90	54.8 68.4 61.2 63.1 68.2 Av. 63.1	48.5 20.0 40.3 15.3 28.6
3	0.26	4.87	89.4 83.0 89.0 80.8 87.9 77.7 86.0 Av. 84.8	61.1 37.7 54.8 33.9 50.5 29.8 45.5
4	0.28	4.00	83.7 74.3 81.8 67.6 Av. 76.8	47.0 27.4 32.9 23.0
5	0.32	3.70	72.9 85.3 88.5 88.5 Av. 83.8	43.0 25.9 30.7 17.5
6	0.25	4.82	96.1 92.2 96.7 90.4 96.1 87.8 92.9 Av. 93.2	60.85 42.8 53.5 38.35 51.05 34.1 47.3

Run	Liquid Helium Flow Rate (lb/min)	Mass of Hydrogen Present (lb)	Compressed Slush Quality-Per Cent Volume-Change Method	Melted Slush Level (cm)
7	0.17	4.07	91.5 60.4 87.1 55.4 81.2 Av. 75.1	26.95 16.4 24.65 11.35 21.0
8	0.18 and 0.11	4.31	80.0 72.2 74.7 64.6 Av. 72.9	33.03 23.83 28.18 19.33
9	0.04 and 0.40	5.12	67.0 71.2 74.2 67.6 Av. 70.0	35.5 25.2 31.1 19.0
10	0.26 and 0.33	4.24	82.2 79.0 80.7 Av. 80.6	27.75 21.25 23.95
11	0.32	4.25	86.7 73.8 Av. 80.3	20.1 17.9
12	0.36 and 0.50	5.33	96.7 84.3 92.5 77.4 91.3 68.1 86.8 Av. 85.3	36.81 25.86 34.01 21.41 31.06 16.96 28.51
13	0.29	3.60	88.1 91.1 90.7 87.3 Av. 89.3	32.56 24.26 28.36 18.46

Notes for Table 4

Prior to starting the liquid helium flow for all the runs, the liquid hydrogen in the test reservoir was vacuum pumped to produce triple point liquid hydrogen. Starting the experimental runs with triple point liquid hydrogen considerably reduces the quantity of liquid helium necessary to produce slush.

During all runs except Run 8, the pressure in the test reservoir was maintained higher than the triple point pressure by the addition of gaseous helium to the test reservoir.

Run 1 The liquid helium flow was started through the heat transfer coil and the crust burster was used to scrape the walls and break the crust during the first few minutes of cooling; however, it soon became stuck. The helium flow was continued, producing additional solids, and then the flow was terminated. The solids produced were then compressed with the crust burster. When the triple point liquid level was above the compressed slush level, data was recorded for the seven compressions performed.

Run 2 Liquid helium flow was provided through the heat transfer coil and solids were produced without any breaking with the crust burster. After the liquid helium flow was stopped, the solids were compressed with the crust burster.

Run 3 Liquid helium flow was provided through the heat transfer coil producing solids in the test reservoir. The knife edge scraper number 1 (Figure 12) was operated at first every minute and later during the production every two minutes. It was difficult to move the scraper near the bottom of the travel in the test reservoir. The liquid helium flow was terminated and the slush was compressed with the compactor.

Run 4 Scraping was provided with the knife edge scraper number 1 every two minutes during the first portion of the helium flow. The scraping was then stopped and the liquid helium flow continued until approximately 16 cm. of solid were above the liquid level. Compression was performed with the compactor. Compressed qualities were determined after the liquid level was above the compressed solids.

Run 5 Scraping was performed every three minutes during the production with knife edge scraper number 1 until the settled slush built up to the liquid level. Scraping was then terminated and the liquid helium flow continued until all solids were produced. The solids were then compressed with the compactor. Quality of the compressed slush was determined when the liquid level, caused by partial melting, was above the compressed slush level.

Run 6 Scraping with knife edge scraper number 1 was performed during the production. The slush produced was compressed with the compactor.

Run 7 Scraping with knife edge scraper number 2 (Figure 13) was performed every half minute producing settled slush. The settled slush was compressed with the compactor. Some observation problems were present during this run.

Run 8 Gaseous helium was not added to the test reservoir during this run. A crust like that produced during straight vacuum pumping was formed on the liquid hydrogen surface. The scraper could not break through the crust during production. The liquid helium flow was stopped and the crust was pushed to the bottom of the test reservoir and broken up with knife edge scraper number 2. The liquid helium flow was resumed and more crust was produced. At the conclusion of the liquid helium flow, the slush was compressed with the compactor.

Run 9 The production of solid was started with a slow liquid helium flow rate, scraping and mixing the solid as it formed. The helium flow rate was then increased to produce more solids. At the conclusion of the helium flow, the solids were scraped and mixed with scraper number 2 and then compressed with the compactor. Prior to the compression, one of the scraper drive rods bent and developed a small crack. The crack was temporarily patched; however, the triple point liquid above the compressed slush was cloudy.

Run 10 Two liquid helium flow periods were provided with scraping and mixing performed after the liquid helium flow was terminated. The resulting slush was compressed with the compactor.

Run 11 Solids were produced without scraping during the liquid helium flow period. After the helium flow was terminated, the solids were scraped and mixed with the knife edge scraper number 2. The following settled slush qualities were determined by mixing before each settling: 81.4%, 78.5%, 75.2%, 80.9%, 77.4%, 77.2%, 79.4%, and 75.3%. These qualities are no doubt high because of the presence of a large chunk of solid in the bottom of the test reservoir which could not be broken. The remaining settled slush was then compressed with the compactor and two compressed slush qualities determined. After the compression, the slush was again mixed with the scraper and allowed to settle. The quality of this settled slush was calculated to be 52.2%.

Run 12 Two liquid helium flow periods were provided. The solids were scraped and mixed after each period with the knife edge scraper number 2. After the first flow period, the settled slush qualities were calculated to be: 80.7%, 86.1%, and 78.2%. After the second flow period, the settled slush qualities were calculated to be: 88.2%, 97.4%, and 84.8%. These qualities are high as explained in Run 11. The settled slush was compressed with the compactor after the second flow period. After the remaining solids were melted, vacuum pumping to produce triple point liquid was performed so that the final melted level could be accurately calculated.

Run 13 Solids were produced without scraping during the liquid helium flow period. After the liquid helium flow was terminated, the solids were scraped and mixed into the triple point liquid. The resulting slush was then compressed with the compactor. Vacuum pumping was performed after melting to calculate true melted level of triple point liquid.

During this series of runs, a problem was encountered in determining the true melted slush level which is necessary to determine the quality of the slush by the volume-change method. Ideally, the liquid hydrogen in the test reservoir would remain at the triple point temperature and pressure until all the solid had melted. Actually, during this series of runs, while the remaining solid was melting, the pressure in the test reservoir increased considerably, indicating that the liquid above the melting slush is

not at a uniform temperature. With this warmer temperature and lower density stratified liquid hydrogen, the levels observed are higher than those which would be present if all the liquid were at the triple point. Consequently, the melted level was too high, causing the computed slush quality to be also too high. Fortunately, during this indirect heat exchange method of slush production, no hydrogen was lost from the system; therefore, the melted level must equal the triple point liquid level that was present prior to the helium cooling. Knowing that these two levels must be equal, a technique was developed for determining the final melted triple point liquid which would also be applicable to production methods where some hydrogen is lost from the system as in the direct injection liquid helium cooling. This technique involves vacuum pumping on the liquid hydrogen, after all the solids in the slush have melted, until triple point liquid is again produced as evidenced by the start of a crust formation. The hydrogen gas pumped, as measured on a wet drum meter, was converted to an equivalent triple point liquid volume. This volume, when added to volume which remained after the vacuum pumping, comprised the true melted volume of triple point liquid. The accuracy of this technique for determining the true triple point melted level was confirmed during Runs 12 and 13 where it was found that the calculated melted slush level determined by this technique was indeed very close to the initial triple point liquid level.

It should be noted in many of the runs presented in Table 4 that the calculated compressed slush quality decreased as the run progressed. It is felt that this quality decrease results partially from the stratification of the liquid above the slush during the compression and partially from the changing solid particle size during melting.

The melted compressed slush level is determined by subtracting the liquid above the compressed slush at the time of the compression from the final melted level. The final melted level can be determined in terms of triple point liquid conditions by the technique described above; however, the liquid above the compressed slush at the time of compression may be stratified and occupy a volume greater than if it were at triple point conditions. This increased volume when subtracted from the final melted volume yields a volume for the melted slush which is less than would be indicated if stratification were not present. This reduced melted slush volume causes a reduction in the slush quality calculated by Equation 1. As the number of compressions, and consequently the compression time, increases for a particular run, it is felt that the liquid above the compressed slush becomes more stratified, therefore indicating a continued decrease in the calculated compressed slush quality.

Another possible cause for the decreasing compressed slush quality during an individual run may result from the solid particles in the compressed slush being of varied sizes, initially resulting in good packing, thus producing high quality. As the compression time increases, perhaps the smaller solid particles melt first, causing the remaining particles to be more uniform in size, which would result in a lower slush quality as the spaces between the

uniform particles would be filled with triple point liquid rather than small solid particles. Actual observation of this phenomena could not be performed as it was not possible to see the individual solid particles in the compressed slush; however, in the scraping and breaking prior to the compression, it could be observed that the solid particles varied greatly in size.

3.7.2.2 Direct Heat Exchange Runs

Table 5 presents a summary of the six individual runs performed by the direct heat exchange method. Included in the table are the liquid helium flow rate, mass of hydrogen present, settled slush quality, compressed slush quality, and the melted slush levels. A brief description of the procedure in each of the runs is presented in the notes following the table.

TABLE 5

SUMMARY OF DIRECT LIQUID HELIUM FREEZING SLUSH PRODUCTION

Run	LHe Flow Rate lb/min.	Mass of Hydrogen Present (lb)	Settled Slush Quality-Per Cent by Volume-Change Method	Melted Settled Slush Level (cm)	Compressed Slush Quality-Per Cent by Volume-Change Method	Melted Compressed Slush Level (cm)
1	0.25	3.91	67.8	48.31	74.2	22.71
			68.0	45.51	72.4	21.01
			66.6	38.51	67.1	17.36
			Av. 67.5		61.0	14.06
					Av. 68.7	
2	0.15	4.33	62.0	46.84	68.9	23.64
			62.6	39.14	65.4	21.19
			66.7	30.09	59.8	18.74
			60.4	28.94	Av. 64.7	
			Av. 62.9			
3	0.25	4.14	97.5	50.2	90.7	25.8
			89.0	35.6	91.1	20.7
			90.4	28.9	92.6	16.3
			Av. 92.3		90.5	13.9
					Av. 91.2	
4	0.31 and 0.32	4.41	58.1	48.5	77.6	32.8
			57.1	46.3	77.0	27.3
			60.3	41.8	74.4	23.7
			52.9	40.4	72.5	19.6
			Av. 57.1		Av. 75.4	
			78.9	52.15		
			73.6	47.4		
			72.9	44.1		
			69.2	41.2		
			Av. 73.6			

Run	LHe Flow Rate lb/min.	Mass of Hydrogen Present (lb)	Settled Slush Quality-Per Cent by Volume-Change Method	Melted Settled Slush Level (cm)	Compressed Slush Quality-Per Cent by Volume-Change Method	Melted Compressed Slush Level (cm)
5	0.34	3.63	87.6	40.4	95.9	23.9
			82.0	37.2	97.8	20.95
			84.2	36.0	94.7	16.7
			82.8	31.85		
			Av. 84.1		Av. 96.1	
6	0.33	4.01	85.0	49.3	94.3	32.1
			84.5	42.2	94.5	28.2
			82.8	38.65	92.1	24.3
			Av. 84.1		90.4	20.9
					88.6	16.95
					Av. 92.0	

Notes for Table 5

For all the runs, the liquid hydrogen in the test reservoir was vacuum pumped until triple point liquid was produced prior to starting the run. Starting the runs with triple point liquid hydrogen enabled the performance of the run while conserving liquid helium. The probable reason for the high calculated slush quality for runs 3, 5 and 6 is discussed below for each of the individual runs.

Run 1 The liquid helium injection was started and solids began to form on the end of the liquid helium injection line; also, solid tubes extended to the liquid surface where a solid crust formed. With a 4.2 cm. build-up of crust, the scraper would not break through the crust. The liquid helium flow was then stopped, and the test reservoir pressure was reduced by vacuum pumping. During the vacuum pumping, the crust could be pushed down slightly but still could not be broken. The vacuum pumping was then stopped and the crust was pushed below the liquid level with the scraper. The scraper was then operated to chop and mix the solids in the lower portion of the test reservoir. The slush was allowed to settle, and three data points for computing the settled slush quality were recorded. The settled slush was then compressed with the compactor to determine the compressed slush quality. At the conclusion of the four compressions, the remaining solids were melted to determine the melted level. Vacuum pumping was then started and continued until all the liquid was again at the triple point. Vacuum pumping at the end of a run allows the calculation of the true melted liquid level and eliminates the use of inaccurate levels which may be caused by stratification of the liquid.

Run 2 The liquid helium injection was started and the solids began to form. The scraper was operated during the injection, but it stuck about half way down in the test reservoir and could not be moved. The solids produced were white in color. The liquid helium flow was continued in order to produce more solids and was then terminated. The white solids were then broken and mixed in the triple point liquid hydrogen with the scraper. Upon settling, four settled slush data points were recorded. The settled slush was then compressed with the compactor and four compressed slush quality data points were recorded. After the compression, the remaining solids were melted and the liquid was vacuum pumped to produce triple point liquid, and thus a true melted liquid level could be determined.

Run 3 The liquid helium injection was started, and the first solids produced were noted in the area of the injector tip. Wafers of solid crust were formed approximately 1 to 2 cm. apart as the liquid surface dropped. Solid particles were noted settling through the liquid at this time. The solid growing from the injection lance to the crust appears to have a number of holes in it. As the liquid helium flow continues, apparently much of the helium is channeling through the existing tubes as solid particles were not seen settling in the liquid. The liquid helium flow was stopped, and after some melting the scraper was worked to the bottom of the test reservoir, breaking and mixing the solids during the process. The calculated settled slush qualities are high because a large piece of solid hydrogen was attached to the injector lance above the settled slush level. The settled slush was then compressed with the compactor and four compressed slush qualities were determined. Vacuum pumping was started when the solids were melted in order to accurately determine the true melted level. The high resulting calculated qualities may be caused by an inaccurate final melted level as the viewing became cloudy at the end of the run.

Run 4 Liquid helium injection was provided producing solids without the use of the scraper. The helium flow was stopped, and after partial melting the solids were broken and mixed with the scraper. The first four settled slush qualities were then determined. The liquid helium injection was resumed and additional solids were produced without breaking. The liquid helium flow was stopped, and again the solids were broken and mixed with the scraper and the last four settled slush qualities were determined. The settled slush was then compressed with the compactor, and four compressed slush qualities were determined. After the remaining solids had melted, vacuum pumping was performed to determine the true triple point melted level.

Run 5 The liquid helium injection was performed producing solids. Eight solid wafers approximately 1 cm. apart were formed below the crust. After the helium flow was terminated, the scraper was used to break the crust and mix the solids into the triple point liquid. Four settled slush qualities were then determined. The settled slush was then compressed with the compactor and three compressed slush qualities were determined. The remaining solids were melted, and vacuum pumping was performed to determine the true melted level; however, the accuracy of this run may not be good, since

it was suspected that a vent valve was leaking slightly during the vacuum pumping. This leakage would cause the calculated melted level to be high, thus indicating high slush qualities.

Run 6 This run was performed in the same manner as Run 5. It is felt that the leakage that was suspected during the final pumpdown for Run 5 was also present during this run, causing both the settled and compressed slush qualities to be too high.

3.7.3 Characteristics of the Solids Produced

3.7.3.1 Indirect Heat Exchange

When employing the indirect heat exchange cooling method without the addition of helium gas to the vapor space to maintain the total pressure above the triple point pressure, a thin crust of solid hydrogen would be first formed on the surface of the liquid hydrogen. With continued helium flow through the heat exchanger, the crust would continue to increase in thickness with the solids appearing to be very similar to the solid produced by vacuum pumping. Actually, the test reservoir wall in the vapor space which is being cooled with liquid helium acts as a vacuum pump by solidifying the hydrogen vapor on the wall. This removal of hydrogen vapor from the vapor space causes some of the liquid hydrogen to vaporize as in conventional vacuum pumping. Simultaneously with the crust formation, solid hydrogen began forming on the test reservoir wall below the crust. This solid, which was transparent, would grow inward towards the glass periscope guard dewar. When all the liquid in the test reservoir had been converted to solid, the solid was mostly transparent at the bottom and predominantly white in the top portion.

To avoid the vacuum pumping effect described above, gaseous helium was bled into the test reservoir during production. If desired, the test reservoir could be brought to atmospheric pressure with helium; however, a pressure of about two inches of mercury above the triple point was generally used. The use of a large volume of helium gas containing impurities would lead to the deposition of these impurities as solid patches on the outside of the glass periscope dewar. The change in helium gas pressure from above the triple point to atmospheric pressure did not affect the formation of solid hydrogen, either on the walls of the test reservoir or on the surface of the liquid hydrogen.

With gaseous helium in the vapor space and liquid helium being transferred through the heat exchanger, a thin, approximately 1/4-inch-thick, transparent flat crust would form at the liquid level. This crust, when observed from above, appeared to have many hairline cracks (Figure 28). While the solid crust was forming, a transparent solid was also forming on the test reservoir wall below the liquid level. An increase in thickness of the solid film on the wall would cause the liquid level to drop away from the bottom of the crust. Another thinner crust would then form at the new liquid level. This process of forming crust wafers (Figure 29) may repeat itself about ten

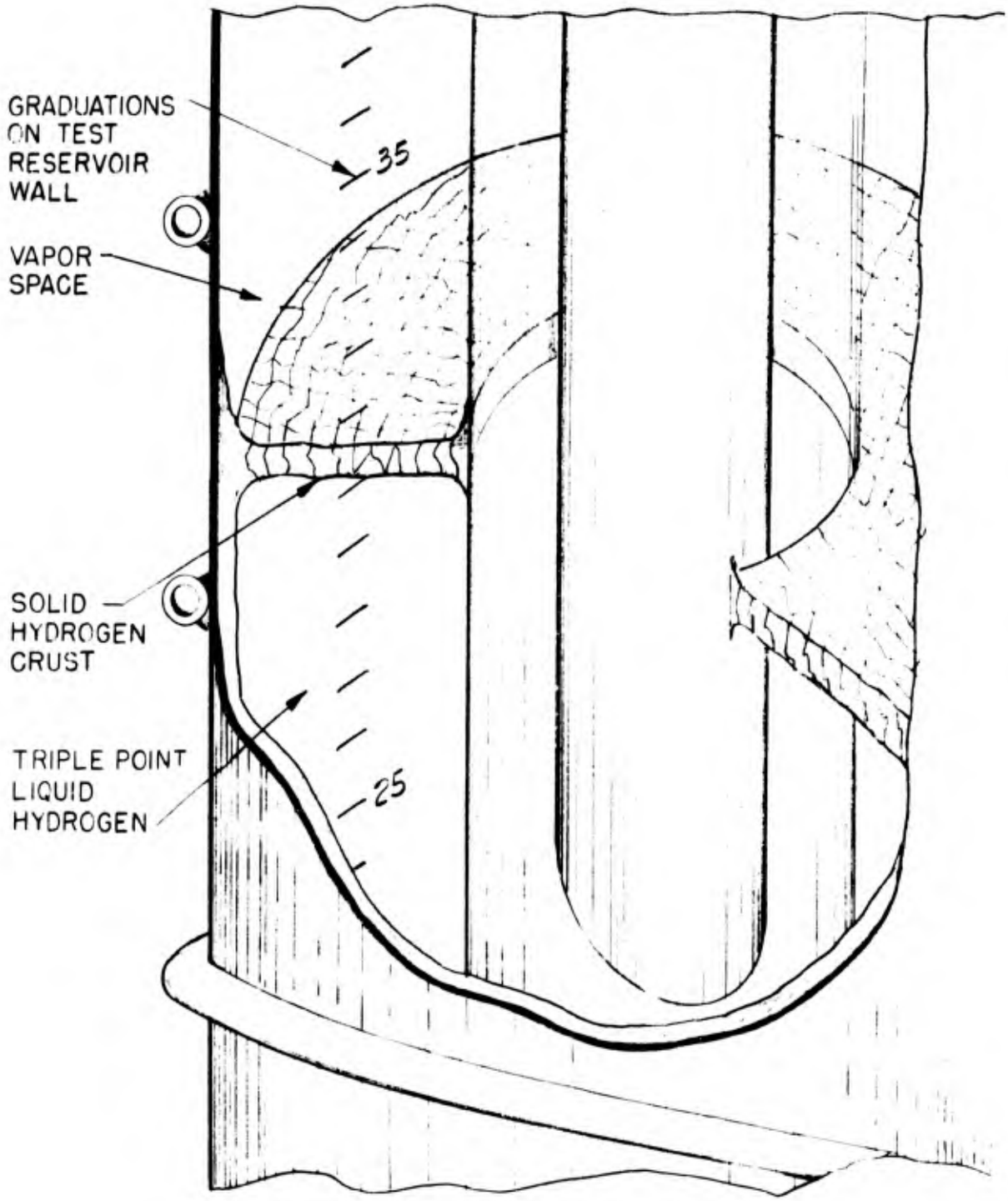

Figure 28. Solid Crust Formed by Indirect Heat Exchange Helium Freezing

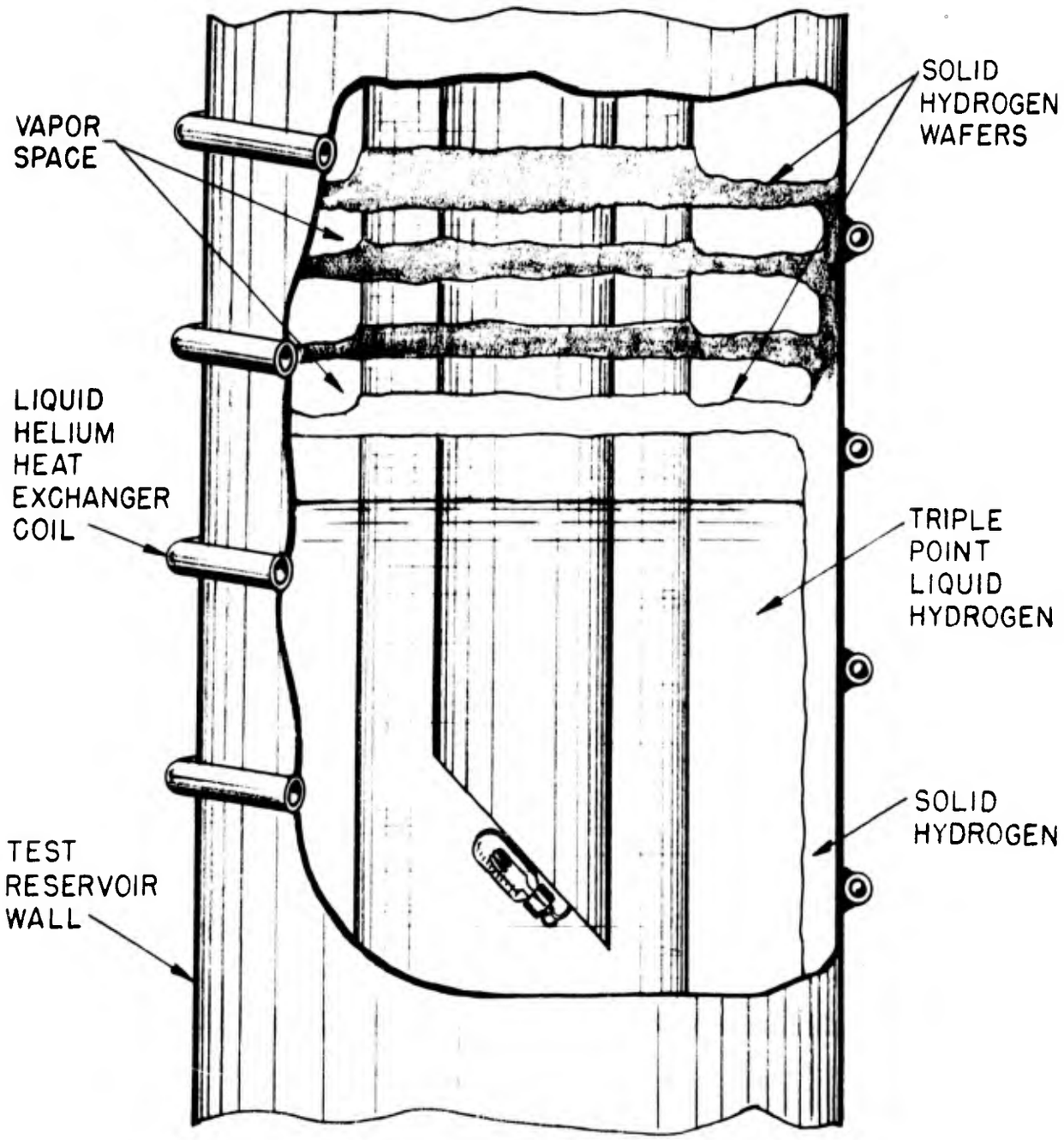

Figure 29. Solid Wafers Formed by Indirect Heat Exchange Helium Freezing

times if all the liquid is converted to solid. The solid crust wafers which span from the test reservoir wall to the glass periscope dewar were spaced approximately 1/2 to 1 cm. apart. To convert all the triple point liquid hydrogen initially at a level of approximately 55 cm. to all solid in this manner required approximately 10 pounds of liquid helium.

In attempts to break the crust and scrape the wall with the crust burster (Figure 9), it was determined that once the crust thickness had reached 1/4 inch it could not be broken. In other runs the crust burster was located below the liquid level before the crust was formed so that the test reservoir wall could be scraped. Under these conditions the crust burster could only be operated for about five minutes before it could no longer be moved. As the solid formed on the wall, the tooth marks of the crust burster in the solid hydrogen layer could be observed. Apparently a film thickness of about 1/16 inch was sufficient to prevent movement of the crust burster. The solid hydrogen, which was scraped off, is carried along on the crust burster to accumulate at the end of the stroke in a ridge of soft, sticky solid, which apparently hardens if further refrigeration is provided.

To provide for better scraping of the walls and breaking of the crust, the crust burster was replaced with knife edge scraper number 1 (Figure 12). With this scraper the crust could be broken and the solids sliced off the test reservoir wall. This scraper, which was of lightweight construction, was soon deformed; although it still removed solid from the test reservoir wall, the solid was sliced at a greater distance from the wall. Several runs were made using this scraper to produce settled slush by breaking the crust and scraping the walls. During a preliminary run employing direct liquid helium injection, the scraper got entangled with the injection lance and was badly damaged (Figure 30).

The badly bent knife edge scraper number 1 was removed and replaced by a more sturdy knife edge scraper number 2 (Figure 13) which was fabricated from thicker material. This new scraper did not perform as well as the previous scraper because the cutting edge was maintained close to the test reservoir wall at all times and would not deform. The maximum solid thickness that could be scraped was between 1/16 and 1/8 inch; however, with continued liquid helium flow this scraper would become stuck. As the edge of the scraper blade is forced into the solid, cracks form in the solid hydrogen where the load is applied. To determine the thickness that could be scraped, a small toothed blade was attached to the scraper and the distances from the test reservoir wall to the tips of the teeth were measured. During production with scraping, as the teeth hit the solid, cracks were observed; thus it was possible to determine the thickness being scraped. In most of the runs performed with this scraper, the solids were formed without breaking or scraping and then the helium flow was terminated. As the clear solid on the wall began to melt, a network of lines, probably cracks, appeared to form (Figure 31). As more and more lines formed, the clarity of the solid diminished rapidly and

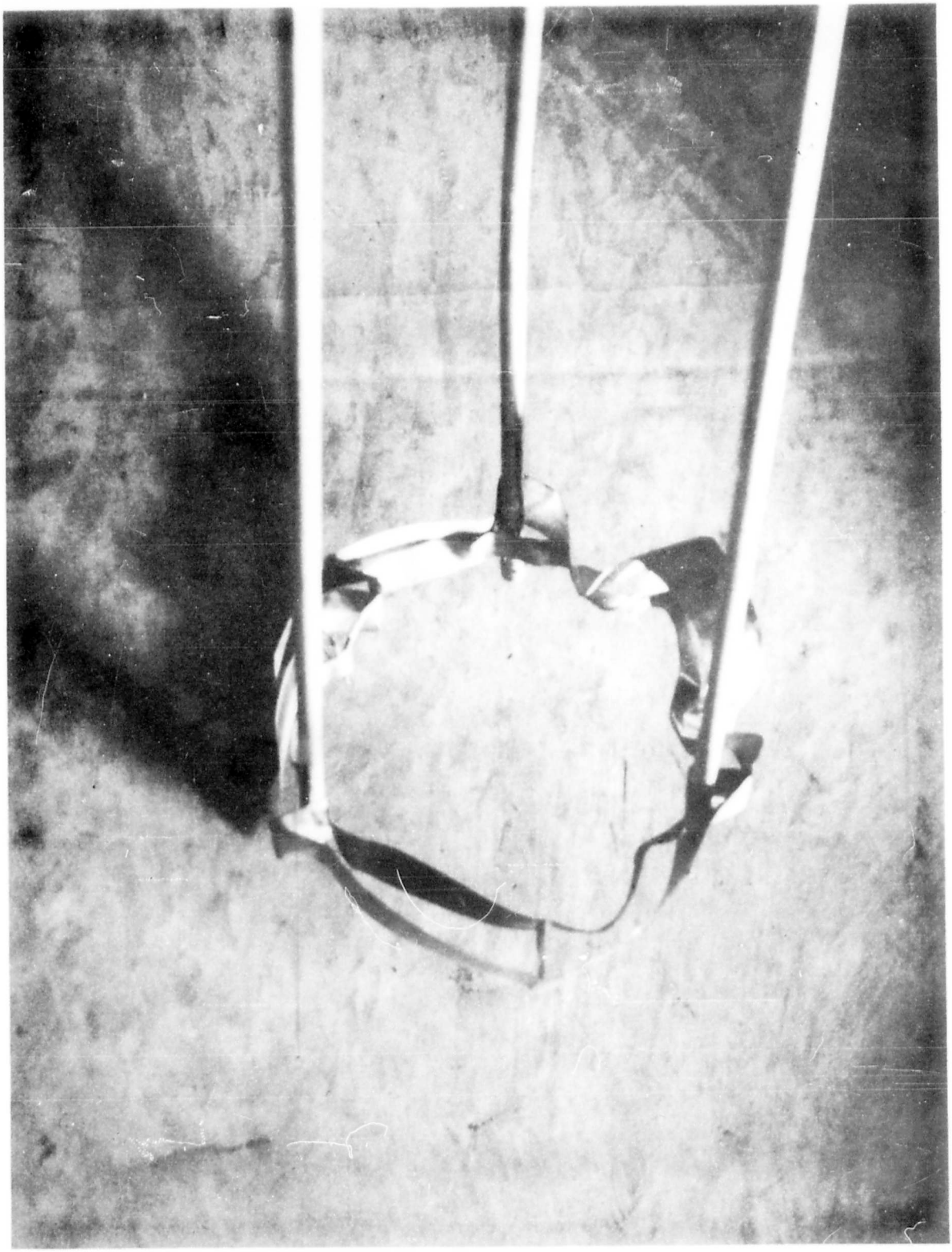

Figure 30. Damaged Knife Edge Scraper Number 1

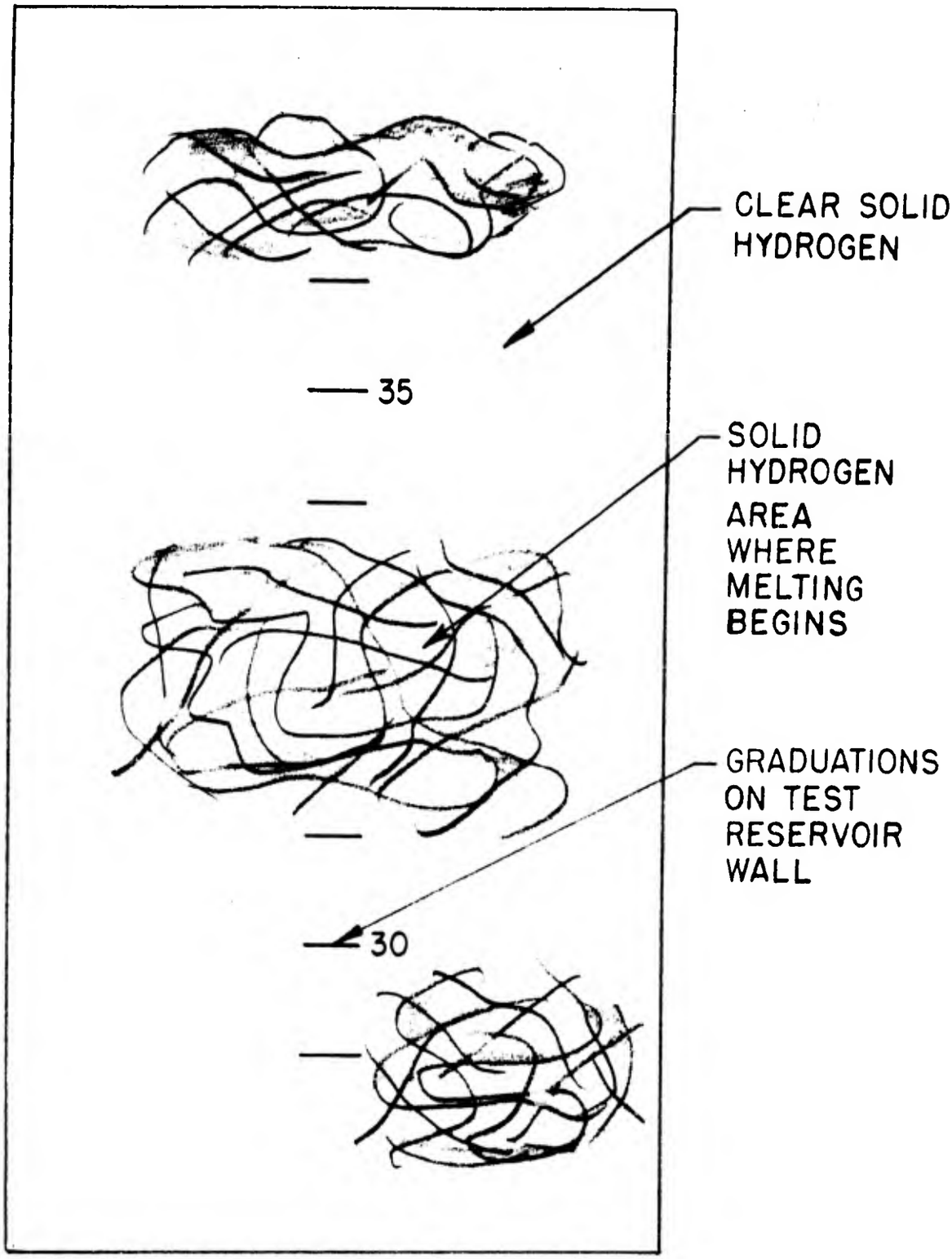

Figure 31. Melting Solid Produced by Indirect Heat Exchange Helium Freezing

finally only the color of the copper wall could be perceived. After this partial melting, the scraper could be driven though the solids with a chopping action, eventually producing a settled slush Solid thicknesses up to 3/4-inch could be scraped during partial melting. After the chopping some solid particles were clearly observed against the background of the scraper blade. The particles had the form of thin plates which were about 1/2 cm. in length and width. These particles were transparent and the perimenter consisted of straight lines joined by rounded intersections (Figure 32). Because of the geometry of the scraper, the large chunk of solid which formed in the bottom of the test reservoir could not be broken and mixed.

At various times the pointed probe (Figure 21) was pushed into the solid and the slush. The clear solid could not be completely penetrated with the probe. After a partial penetration of the solid, it was very difficult, if not impossible, to withdraw the probe. It should be noted that this high resistance of the solid endured only as long as refrigeration was supplied by the liquid helium. When the liquid helium flow was stopped and melting began, only moderate resistance was encountered and the probe could be pushed all the way through the solid. When the probe was pushed through settled slush or compressed slush, no noticeable resistance was felt. The white solid produced when helium gas was not added offered only slight resistance.

To determine if the solid hydrogen would stick to Teflon, a 3/8-inch diameter Teflon rod approximately 6 inches long was attached to the end of a thin wall stainless steel tube which was inserted into the test reservoir through the fill coupling. The Teflon probe was positioned so that it projected through the liquid hydrogen surface. Vacuum pumping was performed and a solid crust was formed around the Teflon rod. When the rod was pulled out of the crust, no solid adhered. Also, when the rod was pushed through the crust, solids did not cling to the sides of the rod. A column of crust was pushed out of the crust ahead of the probe; this column, which did not adhere to the probe, settled in the triple point liquid. Crusts were formed several times with different vacuum pumping rates with the results as above; however, a few times a small solid crystal would stick to a rough spot on the Teflon and a small tube would grow with continued vacuum pumping. These tubes, if formed, would drop off after about one minute.

During indirect heat exchange with liquid helium, the Teflon rod was placed so that the wafers of solid hydrogen would form around the rod. Since the wafers are generally spaced about 1/2 to 1 cm. apart, it was possible to form approximately 10 solid wafers along the length of the rod. After the solid wafers were formed around the rod, the force exerted by two men was required to pull the Teflon rod out of the solid. Also, while in the solid, the Teflon rod could not be rotated. When the Teflon rod was pulled free of the solid wafers, no solids were attached to the rod and pulled up with the rod.

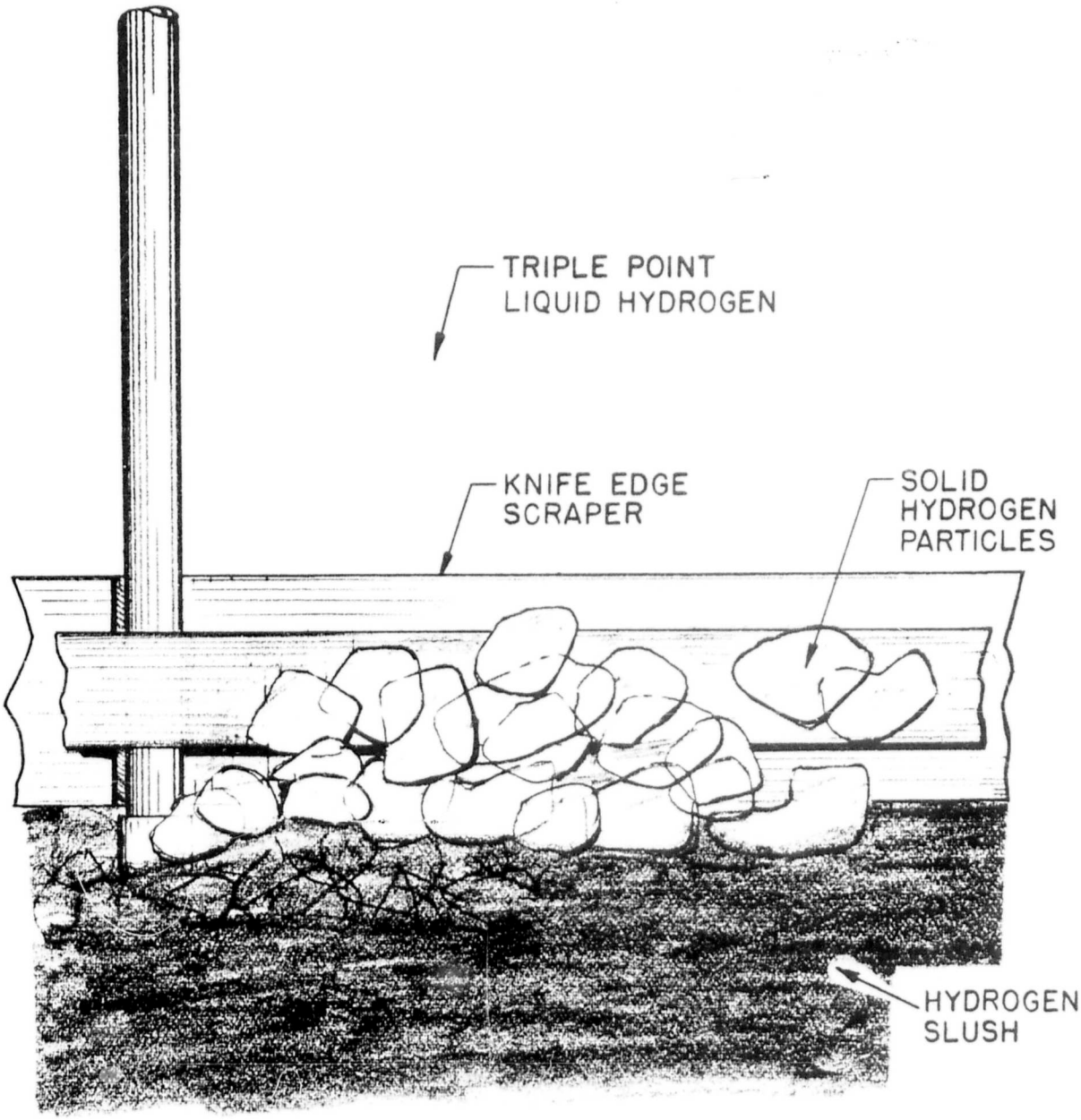

Figure 32. Solid Particles Observed Against the Scraper Background

3.7.3.2 Direct Heat Exchange

Within a short time after the transfer line was cooled, a bulb-shaped solid formation would be formed around the lower portion of the transfer line. The top of this solid formation consisted of many solid tubes which extended to a crust formation which would form on the liquid surface. The solid bulb-shaped formation would rapidly grow to occupy approximately 1/4 of the test reservoir volume (Figure 33). On continued helium flow apparently most of the helium travels upwards through the tubes, slowing the solid formation. The solid hydrogen in the formation had a white color and was not transparent as was the solid which formed on the wall during indirect heat exchange. While observing the solid formation through the periscope, it was noted that there were bubble-shaped void spaces in the solid.

The crust which formed on the liquid surface could not be broken with the crust burster. Upon partial melting as force was exerted on the crust burster, the solid broke loose from the wall and was pushed down in one piece without being broken. Further work on this direct heat exchange method was performed after knife edge scraper number 2 had been installed. This scraper also could not break the solid as long as the liquid helium flow was continued. With the termination of the helium flow and partial melting, the solids softened and could be chopped up with the scraper. Large chunks of solid were produced in addition to the smaller flake-shaped particles. Figure 34 is an artist's sketch of the solid produced by chopping with the scraper.

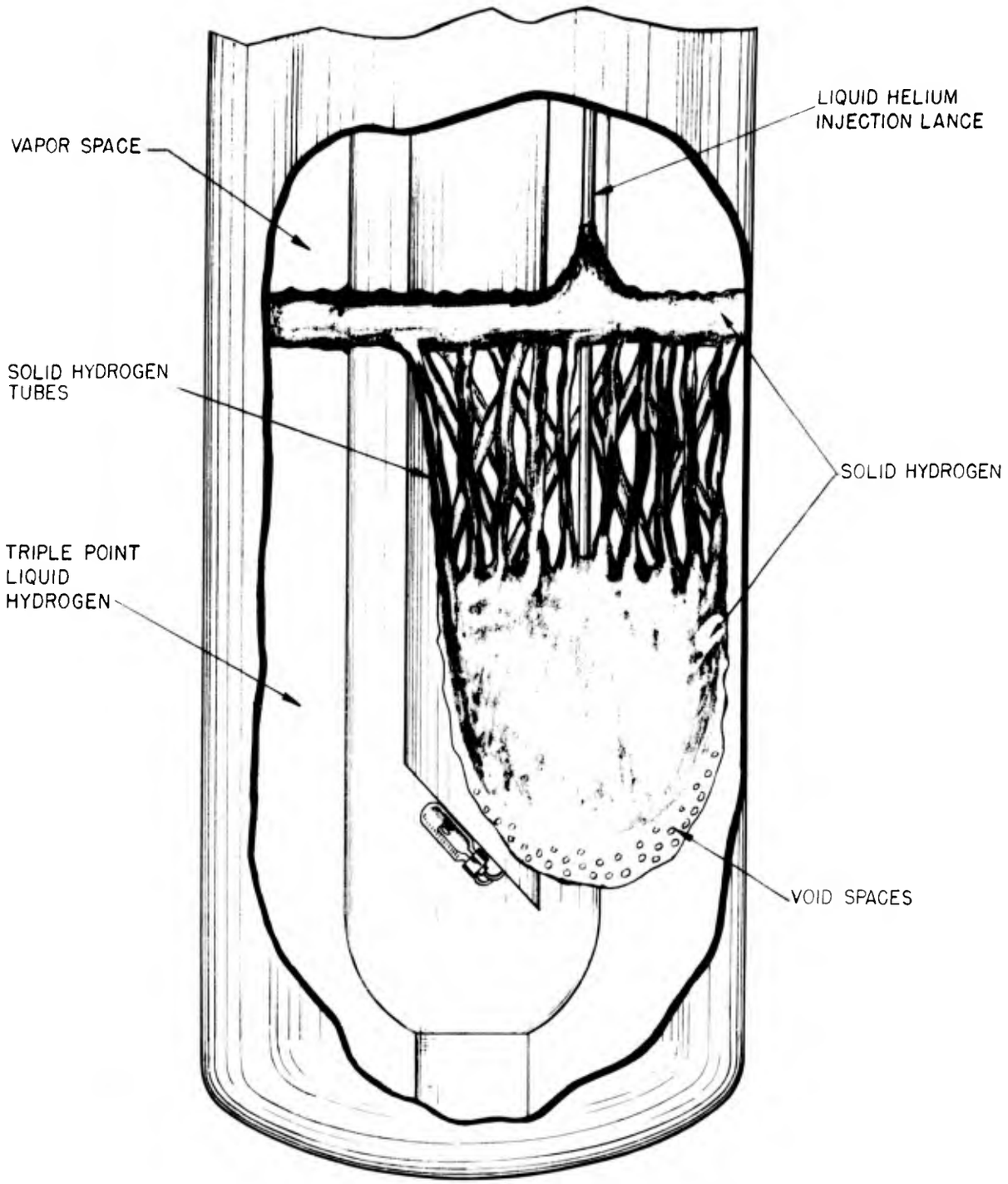

Figure 33. Solid Formation by Direct Heat Exchange Helium Freezing

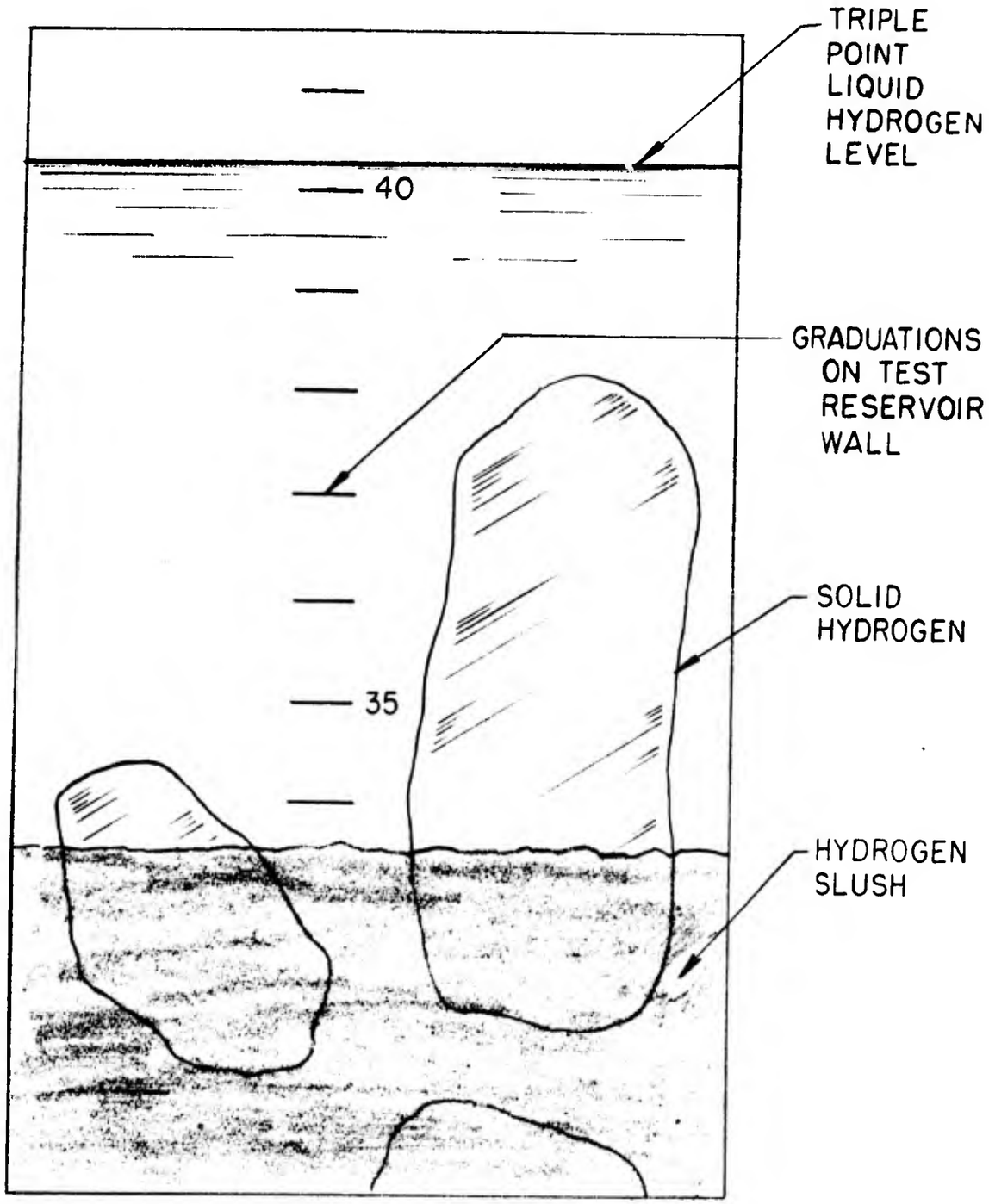

Figure 34. Chopped Solids Formed by Direct Heat Exchange Helium Freezing

SECTION 4

CONCLUSIONS AND RECOMMENDATIONS

4.1 CONCLUSIONS

Neither of the two methods developed for determining the solid content (slush quality) have been found to be entirely satisfactory. A direct reading instrument is desirable, if not essential, to gain meaningful results from any future work. The vapor-volume quality determination method is workable only when the slush is being produced or stored under carefully controlled conditions and depends on accurate knowledge of the heat leak, volume pumped, and slush volume produced. The volume-change quality determination method requires that the produced slush be melted; therefore, this method is unacceptable for a production process.

The periscope utilized for the experimental work offered only a limited field of vision while in any one position. The periscope could, however, be adjusted to observe the entire test reservoir, except the extreme bottom by appropriate translation and rotation. The requirement for a low heat leak apparatus precluded the use of high power lights on the periscope. It was not possible to photograph the slush hydrogen through the periscope because of the low power lights that were used and because of focus difficulties which resulted when the camera was attached to the periscope.

The conclusions, which result from the individual production methods, are presented in the following sections.

4.1.1 Vacuum Pumping Slush Hydrogen Production

1. A solid hydrogen crust formation, which was thicker than 4 cm., could not be broken up with the crust burster. Thicker crusts could, however, be dislodged from the walls of the test reservoir and glass dewar, with the crust being pushed downward as one piece.

2. A solid hydrogen crust, while forming, would cling to the copper test reservoir wall and the glass guard chamber wall, but not to a Teflon rod, which was located in the growing crust.

3. The solid hydrogen crust was found to consist of approximately 65% solid and 35% voids.

4. The solid particles, which were observed settling through the triple point liquid after breaking of the crust, consisted of transparent needles and flakes. The largest flakes were approximately 1 cm. in length and width. Solid particles could not be distinguished in the settled or compressed slush.

5. Solid crust, settled slush, and compressed slush could easily be pierced with a pointed probe.

6. The solids in the crust formation are white in color, while the settled slush appears opaque. The compressed slush exhibits a dirty white color.

7. Settled slush, once compressed, tends to remain compressed unless a long melting period is provided with no compression of the slush. When agitated, the triple point liquid above the compressed slush becomes cloudy, indicating that very fine particles break loose from the compressed slush.

8. Compressed slush could not be extruded through the holes in the crust burster when approximately 1.25 psi was applied to the slush with the compactor.

9. With both the freezing and melting production methods, the compressed slush quality was generally in the 65 to 85 per cent range.

10. A relationship between the compressed slush quality and heat leak, or between the compressed slush quality and the net pumping rate, was not found.

4.1.2 Slush Hydrogen Production by Helium Gas Injection Followed by Vacuum Pumping

4.1.2.1 Formation of Tubes

1. Solid hydrogen tubes form in triple point liquid hydrogen along the gaseous helium bubble path. Either warm or cold helium gas will form tubes.

2. The liquid in the test reservoir had to be maintained at or very close to triple point conditions in order to form tubes of solid hydrogen. Continued vacuum pumping was therefore required during the formation of tubes.

3. The solid hydrogen tube diameters generally ranged from 1/16 to 1/4 inch, although with warm helium gas injection tubes of approximately 3/8-inch diameter have been observed.

4. The solid hydrogen of the tube wall was transparent and smooth on the outside wall, while the inner wall surface was rough and white in color. The white color disappears if liquid hydrogen enters the tube.

5. Solid tubes, which had broken off, did not continue to grow unless helium gas was still flowing through the stub of the broken tube.

6. Solid tubes can be formed in settled slush as in triple point liquid hydrogen.

4.1.2.2 Manipulation of Tubes

1. The solid tubes could be continuously broken by rotating the injection lance. The tubes initially break near the holes in the lance and are then fractured into smaller pieces by the rising helium gas bubbles. The largest broken tube section observed was at most 2 cm. long.

2. When the crust burster was pushed down in an attempt to break the tubes, the tubes would bend rather than break. With further downward movement of the crust burster, a layer of solid accumulated on the lower surface of the burster. This layer of solid consisted of a thin crust and fused tubes.

3. If the crust burster is located below the liquid surface during tube formation, the tubes will grow through the holes in the burster to the liquid surface. An upward movement of the burster will cause the tubes to break just below the burster. With continued helium flow, the tubes will reform.

4.1.2.3 Formation of Columns from Tubes

1. The growth of solid hydrogen columns from tubes required that the gaseous helium flow through the tube be terminated and vacuum pumping continued. Several closely spaced tubes will merge to form a single column while vacuum pumping. A single tube may grow into a column for only a portion of its length, with the portion of the tube below the column growth eventually melting.

2. As the tubes were grown into columns by vacuum pumping, the crust also continued to grow. The growth rate of both the columns and the crust appeared approximately equal on a volume-produced basis. Both the columns and the crust were white in color. The surface of the growing columns was very rough.

3. Columns were grown from tubes formed in settled slush. The white solid of the column could be distinguished from the settled slush.

4. The quality of compressed slush produced by compressing columns and crust generally ranged from 55 to 70 per cent. The quality of compressed slush produced by growing columns in settled slush generally ranged from 60 to 85 per cent. It appears that higher compressed qualities can be produced by growing columns in settled slush formed by vacuum pumping.

4.1.3 Slush Hydrogen Production by Liquid Helium Freezing

4.1.3.1 Indirect Heat Exchange

1. The liquid helium heat exchanger coil performed as designed, producing a uniform film of solid hydrogen on the test reservoir wall.

2. Solid formation could be accomplished under atmospheric helium pressure.

3. If gaseous helium was not bled into the system to maintain the system pressure above the triple point pressure, a white crust, similar to that formed by vacuum pumping, would be formed on the surface of the liquid. If the system pressure was maintained above the triple point pressure, several transparent crust wafers, separated by vapor spaces, would form as the liquid volume decreased, because of solid formation.

4. As long as the liquid helium flow was provided, the transparent solid hydrogen could not be completely penetrated with the pointed probe. The probe could penetrate some distance into the solid but then could not be pulled free. As soon as the liquid helium flow was terminated, partial melting of the solids would begin and the solid would soften. The pointed probe could then be easily pushed through the solid.

5. A knife edge scraper, which was somewhat flexible, was operated to break the crust and scrape the solids off the wall during production. A more rigid scraper of the same geometry did not perform satisfactorily, since it would become stuck during scraping when solid was produced. When the helium flow was terminated, the solids could be scraped and chopped with the rigid scraper.

6. The solid particles comprising the settled slush were observed to be in the shape of thin transparent plates, which were approximately 1/2 cm. in length and width.

7. The quality of the compressed slush produced by indirect helium cooling ranged from 65 to 95 per cent.

4.1.3.2 Direct Heat Exchange

1. Direct liquid helium injection into liquid hydrogen produced a white-colored, bulb-shaped, solid formation around the end of the transfer line. Numerous solid tubes extended from the solid formation to a crust which was formed on the liquid surface.

2. The growth rate of the solid formation decreased as the liquid helium flow continued; however, all solid could eventually be produced in the test apparatus if the helium flow was continued.

3. Upon termination of the liquid helium flow, the solid produced was chopped and broken with the scraper. The settled slush contained some large chunks of solid which showed no tendency to break up into thin plates.

4. The compressed slush quality produced by the direct heat exchange production method ranged from 65 to 95 per cent. This is in the same quality range as the compressed slush produced by the indirect heat exchange method.

4.2 RECOMMENDATIONS

During the performance of experimental work detailed in this final report on Contract AF 33(615)-1357, the requirement for further examination of slush hydrogen became apparent. A review of these requirements, taking into consideration the experience gained to date, has resulted in the recommendations presented here in both summary and detailed forms. The recommendations which follow were selected because they represent immediate problems in the overall slush hydrogen program. There are also a number of other areas, not discussed, which may well merit further investigation in the future.

4.2.1 Summary

1. A direct reading slush hydrogen quality meter should be developed for accurately determining the solid percentage in slush hydrogen. This meter should be applicable to production, transfer and storage systems.

2. An experimental investigation should be undertaken to determine the relationship between the slush hydrogen quality produced and the applied compressive pressure.

3. Improved methods for breaking the solids during production and mixing these solids into slush should be developed and experimentally tested.

4. Improved methods and devices for observing and photographing the slush hydrogen production should be developed and experimentally tested.

4.2.2 Discussion of Recommendations

4.2.2.1 Slush Hydrogen Quality Meter

Present methods used to determine the quality of slush hydrogen require either a prior knowledge of the production and storage of the slush or require the destruction of the slush by melting. Also, present methods require the observation of the slush in order to determine the volume occupied by the slush. It is apparent from these facts that a slush hydrogen quality meter must be developed and tested before the overall slush hydrogen program can progress very far. The quality meter developed should be useful both in a static slush system such as a storage reservoir, or in a dynamic slush system application as would be encountered in a production system or a transfer system.

Various slush hydrogen quality measuring methods, which may include beta-particle attenuation, capacitance, viscosity or photoelectric effect, should first be theoretically examined for practicality. The most promising device should then be fabricated and evaluated first in a small static slush hydrogen system and then in a dynamic system.

The importance of developing an accurate and reliable slush hydrogen quality meter cannot be overemphasized, since this tool would significantly improve the accuracy of all future slush hydrogen development work.

4.2.2.2 Compressive Pressure - Slush Quality Relationship

Relatively high slush hydrogen qualities were produced during this experimental work with the application of only low compressive pressures. It would be desirable, from the standpoint of producing a given quality slush, to determine the slush quality that can be produced by employing both higher and lower compressive pressures. With the aid of an accurate slush hydrogen quality meter, it would be possible to determine the relationship between the slush hydrogen quality produced and the applied compressive pressure. It may be found that with the use of only moderate compressive pressures very high quality slush hydrogen can be produced; conversely, it may be found that high compressive pressures will only yield a small increase in slush quality over moderate compressive pressures.

During the work on this contract, it was not possible to produce high compressive pressures because the slush compressor was operated manually from outside the test apparatus. A pressure of approximately 1.25 psi was generally applied in all the compression runs. This pressure was computed from the maximum driving force and the area upon which this force was exerted. For future work, the pressure should be applied by mechanical means so that higher pressures can be employed. Also, pressure measurement transducers should be located in the compressed slush to determine the true compressive pressure throughout the slush.

4.2.2.3 Breaking and Stirring Devices

Both vacuum pumping and liquid helium cooling slush production methods produce a solid formation which must be broken up to produce slush hydrogen. The breaking and mixing of the solids is an area that certainly requires investigation. With vacuum pumping the solids form on the surface of the liquid in a crust formation. During formation the crust will adhere to metals and glass, making it difficult to break up the crust into small solid particles. It has been found during this work that the crust while forming will not adhere to Teflon. From the experience gained with slush hydrogen to date, it is apparent that a rotating and translating type breaker is more efficient than a breaker that only translates.

With the indirect heat exchange liquid helium production method, the solid produced on the heat exchange wall can be scraped with a knife-edge-type scraper. This scraper appears to work best when the scraping is performed some distance away from the wall where the solids are somewhat softer. Other scraper configurations should be considered that may perform a more efficient scraping and mixing function, including the use of a rotating scraper.

Experimental work should be performed to determine the optimum breaker, scraper and stirrer configurations for use in slush hydrogen production equipment.

4.2.2.4 Production Observation Devices

To provide more insight into future system problems while still performing experimental production work, it would be desirable to investigate better methods for observing the slush production. Even when an accurate slush hydrogen quality meter is developed so that the quality determination is not dependent on the observed levels, much can be learned by observing the slush production. As an example, when evaluating breaking and stirring devices, it is important that the action of the breaker be noted and the size of the solid particles be observed in order to correlate the solid particle size with the produced slush quality. During production with compression, good observation will determine if any solids are passed through the compactor and therefore not being compressed. By determining which compactors will not pass solid particles, a good compactor design can be achieved.

The periscope-type device used for the experimental work described in this report performed satisfactorily in general; however, there were some areas where improvement is necessary to provide better observation. One shortcoming was that only a small field of vision was provided with the periscope in any one position. The position of the periscope could be changed by rotation and translation in the system; however, only a small area could be observed at any one time. It would be desirable to observe the entire production system at one time, but this appears only possible with an all-glass system which has inherent disadvantages. Also with the periscope-type device, it was not possible to take photographs of the production process because of inadequate lighting and camera focus problems. It is extremely desirable to have the capability to photographically record the production process because different observers in some instances may render a different account of that which was actually observed. With the addition of a wide angle lens, a high intensity flash-type light, and a properly matched camera, a periscope-type device could be developed into an excellent observation and recording tool.

Another method which offers promise for good observation is the use of glass windows in a metal slush production tank. With this system the prime requirement is that an effective seal be provided and maintained between the glass and the metal tank. If the seal is lost with hydrogen in the tank, the vacuum insulation system would become ineffective, resulting in rapid evaporation of the tank contents. Other development areas with this system include the ability to reduce the heat leak through a window when the window is not in use, methods to eliminate condensation and frost from the outside and inside window should it occur, methods to provide sufficient lighting to the tank contents for photography, and methods to provide a wide viewing angle when the windows are separated by a vacuum space.

The use of television or movie cameras should also be investigated as possible methods to record the production process. It may be possible with modifications to use these cameras in a guard chamber such as is used for a periscope-type device, or in a vacuum space in conjunction with a window in the production tank.

APPENDIX

SAMPLE CALCULATION FOR DETERMINING SLUSH HYDROGEN QUALITY

A.1 CALCULATIONS

To illustrate how the quality calculations were performed for the experimental runs as shown in Table 1, a sample calculation using that data of Run 2 follows. Run 2 was specifically chosen because it involves the use of the heater, and also some system leakage was present during this run.

The required experimental data extracted from the data book, which is necessary for the calculations, includes the following:

TIME	PUMPED GAS WET DRUM METER (cu. ft.)	LIQUID HYDROGEN LEVEL (cm.)	REMARKS
1255	1615.08	45.8	Start vacuum pumping atmospheric pressure liquid hydrogen. Crust burster in liquid, compactor in vapor space.
1356	1737.02	36.0	First solid produced. Crust burster in liquid, compactor in vapor space.
1520	1870.29		Stop pumping - settled slush. Crust burster in settled slush, compactor in vapor space.
1522			Compressed slush to 22.0 cm. with 1.0 cm. of liquid above compressed slush after removal of compactor. Crust burster in compressed slush.
1541		25.2	All solids melted, crust burster and compactor out of liquid.

Barometric pressure during run 751.08 torr.
Average meter temperature during pumping to first solids 84.6°F.
Average meter temperature during production of settled slush 86.0°F.

A.1.1 Vapor-Volume Method

The vapor-volume method of quality calculation will be performed first, using the above data. First the system leak rate is calculated by subtracting the actual volume removed from the system, as determined from the start liquid level and the first solid liquid level, from the volume pumped as determined from the wet test meter. The volume pumped as determined by the wet test meter is:

$$\text{Volume pumped} = 1737.02\ \text{ft}^3 - 1615.08\ \text{ft}^3 = 121.94\ \text{ft}^3$$

This volume must be corrected for atmospheric pressure, water vapor pressure, temperature, and meter constant. The corrected pumped volume is:

$$\text{Corrected pumped volume} = 121.94\ \text{ft}^3 \left(\frac{751.08 - 30.2}{760}\right)\left(\frac{530}{544.6}\right)(1.00)$$

$$= 112.56\ \text{ft}^3\ \text{(NTP)}$$

where 751.08 is the atmospheric pressure, 30.2 is the water vapor pressure at 84.6°F, and 1.00 is the meter constant.

The system leakage is determined by subtracting the gas volume removed during the pumpdown as determined by the change in liquid levels from the corrected pumped volume.

$$\text{System leakage} = 112.56\ \text{ft}^3 - 192\left[\left[\frac{(45.8 - .5)(.3767) + 3.29}{28.32}\right] 4.4182 - \left[\frac{(36.0 - .5)(.3767) + 3.29}{28.32}\right] 4.8079\right]$$

where 192 converts pounds of hydrogen to cubic feet (NTP) of hydrogen, 28.32 converts liquid liters to liquid cubic feet, 4.4182 is the density of atmospheric liquid hydrogen, and 4.8079 is the density of triple point liquid hydrogen. The subtraction of 0.5 cm. from the liquid levels is performed because the crust burster which was in the liquid in both cases displaces 0.5 cm. of liquid hydrogen.

The system leakage for this run during the pumpdown is therefore:

$$\text{System leakage} = 112.56\ \text{ft}^3\ \text{(NTP)} - 66.57\ \text{ft}^3\ \text{(NTP)}$$

$$= 45.99\ \text{ft}^3\ \text{(NTP)}$$

The system leakage rate during the pumpdown is:

$$\text{Leakage rate} = \frac{45.99\ \text{ft}^3\ \text{(NTP)}}{61\ \text{minutes}} = 0.754\ \text{ft}^3/\text{min. (NTP)}$$

The pumping which was performed from the time the first solids were produced until the pump was stopped is composed of the following system requirements which were satisfied: (1) pumping to produce solids, (2) pumping to provide for heat leak, (3) pumping to overcome system leakage. In order to determine the slush quality, it is necessary to determine the amount of the total pumping which was utilized to produce solids. The corrected total pumping is therefore calculated:

$$\text{Total pumping between first solids and stop pump} = (1870.29-1737.02)\left(\frac{751.08-31.8}{760}\right)\left(\frac{530}{546}\right)(1.00)$$

$$= 149.74 \text{ ft}^3 \text{ (NTP)}$$

The average pumping rate during production is the total pumping rate minus the system leakage.

$$\text{Average pumping rate during production} = \frac{149.74}{84}\frac{\text{ft}^3\text{(NTP)}}{\text{min.}} - 0.754\frac{\text{ft}^3\text{(NTP)}}{\text{min.}}$$

$$= 1.03\frac{\text{ft}^3\text{(NTP)}}{\text{min.}}$$

The mass of solids present at the time of the compression can be computed from the total pumping minus pumping for heat leak and pumping to overcome the system leak. The system heat leak is composed of the heat provided by the five-watt heater (0.281 ft^3/min.) and the normal system heat leak (0.252 ft^3/min.). The total heat leak during this production was was therefore 0.533 ft^3/min. The mass of solid present when the slush was compressed is:

$$M_s = \left[\frac{149.74 - (0.533 \times 86) - (0.754 \times 84)}{192}\right] \times \frac{193.63}{25.03} = 1.6345 \text{ lb.}$$

where 192 converts cubic feet of hydrogen to mass of hydrogen, 193.63 is the heat of vaporization of triple point hydrogen, and 25.03 is the heat of fusion for solid hydrogen.

The required volume to hold this mass of solid is computed by dividing the solid mass M_s by the solid density ρ_s:

$$\frac{M_s}{\rho_s} = \frac{1.6345}{5.412} = 0.3020 \text{ (ft}^3\text{)}$$

The volume occupied by the compressed slush (V_{ls}) is calculated from the compressed slush level.

$$V_{ls} = \frac{(22.0 - 0.5)(0.3767) + 3.29}{28.32} = 0.4022 \text{ ft}^3$$

Utilizing Equation 8, the compressed slush quality is calculated.

$$\text{Slush quality} = \frac{M_s}{M_s + \left[V_{ls} - \frac{M_s}{\rho_s}\right] \rho_l} \times 100 \qquad (8)$$

$$\text{Slush quality} = \frac{1.6345}{1.6345 + (0.4022 - 0.3020)\ 4.8079} \times 100$$

$$\text{Slush quality} = 77.2\%$$

A.1.2 Volume-Change Method

The calculation of the compressed slush quality by the volume-change method is much simpler and only depends on the compressed slush level and the melted level resulting from the compressed slush. It is not necessary to know the history of the slush production which, as shown in Section A.1.1, includes volumes pumped, heat leak rates, and system leakage if present. Equation 2 is used to determine the quality by the volume-change method:

$$\text{Quality} = \frac{1 - \frac{X_1\ (0.3767) + 3.29}{X_2\ (0.3767) + 3.29}}{0.1109} \times 100 \qquad (2)$$

In the case of this calculation X_1 = 22.0 cm. - 0.5 cm. = 21.5 cm. where the 0.5 cm. accounts for the slush displaced by the crust burster. X_2 = 25.2 cm. - 1.0 cm. = 24.2 cm. where the 1.0 cm. subtraction accounts for the triple point liquid above the compressed slush at the time of the compression. The quality calculation by the volume-change method is:

$$\text{Quality} = \frac{1 - \frac{21.5\ (0.3767) + 3.29}{24.2\ (0.3767) + 3.29}}{0.1109} \times 100$$

$$\text{Quality} = 73.9\%$$

In runs where more than one compression was performed, the same procedure as above was used to calculate the quality for each compression point. For the slush hydrogen production by helium injection and the slush hydrogen production by liquid helium freezing, the above method was used to calculate the slush hydrogen quality.