SLUSH HYDROGEN PRODUCTION, STORAGE, AND DISTRIBUTION STUDY PROGRAM

Final Report

for

Space Nuclear Propulsion Office
National Aeronautics and Space Administration
Cleveland, Ohio

May 13, 1966

Prepared under Contract No. SNPC-41 by

UNION CARBIDE CORPORATION, Linde Division

Tonawanda, New York

FOREWORD

This is the final report of a study performed by Union Carbide Corporation, Linde Division under Contract No. SNPC-41. The contract work was performed under the technical direction of Mr. Paul M. Ordin, Space Nuclear Propulsion Office Cleveland Extension.

The study was conducted during the period from November 4, 1965 to April 29, 1966 by Mr. C. R. Baker, Mr. J. D. Brunt, Mr. C. F. Fails, who also served as Project Manager, and Mr. R. A. Pike. Dr. G. A. Cook and Dr. L. C. Matsch served as special consultants and Mr. L. C. Kun contributed the appendix on hydrogen compression equipment.

TABLE OF CONTENTS

APPENDICES

ILLUSTRATIONS

SECTION 1

INTRODUCTION

The Space Nuclear Propulsion Office of the United States Atomic Energy Commission and the National Aeronautics and Space Administration plans the construction of a nuclear rocket propulsion module test complex at the Nuclear Rocket Development Station, Jackass Flats, Nevada for the ground testing of large NERVA engine systems. Tests at this complex, to be known as Engine/Stage Test Stand 2-3, will use large quantities of hydrogen as conventional liquid, as subcooled liquid or as a mixture of solid and liquid, the so-called "slush hydrogen".

Under Contract SNPC-41 Union Carbide Corporation, Linde Division, has completed a study of the technological, economic, and logistic factors relating to the manufacture and distribution of slush hydrogen in order to recommend a system to satisfy the requirements of the Nuclear Rocket Test Program proposed for the above complex.

Under the terms of this contract, certain objectives were established. They can be summarized as follows:

1. To review the findings of previous work on slush hydrogen, updating these to incorporate the present state-of-the-art and any novel approaches.

2. To determine if slush production is limited to "on-board" tankage or if distribution of slush from a facility, either adjacent to the test site or at a remote liquid hydrogen plant, is feasible.

3. To determine the most attractive cycle and to define the size, location and operating requirements of a plant to satisfy the slush hydrogen requirements of the Nuclear Rocket Test Program.

4. To produce a conceptual design of this plant, including layouts, flowsheets, project schedules, and cost estimates.

This report records the work done by the Linde Division in pursuit of these objectives.

The system recommended in this report is an 18 ton per day plant using the straight vacuum pumping process and located within the Jackass Flats Complex. It must be recognized that this is a recommended solution to the very specific problem posed by the contract statement of work.

If NASA's slush hydrogen requirements expand beyond the program discussed in this report, it is likely that production at commercial liquid hydrogen plants with flexible distribution systems will be required. This will require more sophisticated processes and techniques than those used in the present study and will require considerable development effort.

The final section of this report contains recommendations for development programs to raise the state-of-the-art in slush production and distribution above the level necessary to solve the immediate problem. Successful completion of these programs would permit commercial production and distribution of slush on a scale comparable to liquid hydrogen.

SECTION 2

SUMMARY

2.1 REVIEW OF PROPOSED PROCESSES

Through a review of the available literature, the number of proposed processes for the production of slush hydrogen was reduced to four. Other proposed processes were found to be theoretically unsound or economically quite unattractive. The four processes which merited further consideration are:

1. Straight vacuum pumping
2. Cascade vacuum pumping
3. External helium refrigeration
4. Helium-hydrogen vapor recirculation

These four processes are reviewed in Section 3.

2.2 THE NUCLEAR ROCKET TEST PROGRAM

On August 25, 1965, the Space Nuclear Propulsion Office issued a document, "Criteria for Budgetary Study for Engine/Stage Test Stand 2-3". Using this as a guide, certain criteria were established to define the slush hydrogen requirements of the present test program. These criteria, given in Section 4, defined the present problem and the remainder of this report is a solution to this specific problem only. It must be emphasized that any significant change in these criteria or an improvement in the present state-of-the-art might lead to a radically different solution.

2.3 APPLICATION OF PROPOSED PROCESSES

Using the established criteria, material and heat balances were made for the four proposed processes. These indicated that only straight vacuum pumping and the external helium refrigeration processes merited further study. Sections 5 and 6 contain a preliminary economic evaluation of these processes with an evaluation of the logistic requirements and problems. There were indications in the literature that slush containing more than 50% solids would not have acceptable transport characteristics. Thus, 50% quality was adopted as a standard.

2.4 SELECTION OF THE SYSTEM

The economic and technical evaluations mentioned above were used to define a system considered most likely to meet the requirements of the current test program criteria. The recommended system (see Section 7) can be summarized as follows:

"A plant to produce 660,000 lbs. per month of 50% slush hydrogen from liquid para-hydrogen feed, using the straight vacuum pumping process and located in the E/STS 2-3 complex adjacent to the bulk liquid hydrogen storage".

Several special techniques within the straight vacuum pumping process are discussed in Section 8. These include cryogenic versus ambient vacuum pumps, mechanical crust breakers and the "freeze-thaw" technique.

2.5 CONCEPTUAL DESIGNS

Sections 9, 10 and 11 of this report are devoted to conceptual designs of two systems. Layouts, flowsheets, and plot plans are included for plants using both cold and warm vacuum pumps and provision is made for both mechanical crust-breaking and the "freeze-thaw" technique.

Section 12 contains a preliminary concept of the slush storage requirements of the test facility. Operating features are discussed in Section 13.

2.6 COST AND SCHEDULE ESTIMATES

Estimates of the capital and operating costs for these two plants are presented in Section 14, together with a construction schedule.

It is estimated that the system using the cold vacuum pump would cost $767,000 and require 20 months to design and construct. For the warm vacuum pump, the comparable figures are $884,000 and 20 months.

These costs and schedules are for plants designed, fabricated, and installed to commercial standards. It is assumed that the necessary development and pilot plant work will be completed before the design of the plant begins.

2.7 PROBLEM AREAS AND RECOMMENDATIONS

During this study, a number of areas were found in which design efforts were running ahead of the state-of-the-art. Section 15 discusses the problem areas and contains recommendations for Research and Development programs to remedy these defects.

SECTION 3

REVIEW OF PROPOSED SLUSH HYDROGEN PRODUCTION PROCESSES

A review of the literature revealed that virtually all the proposed processes for converting liquid hydrogen into hydrogen slush are contained in U. S. Air Force Technical Documentary Report APL-TDR-64-22. This work was reviewed and the various proposals were reduced to five which seemed to warrant further study.

All the proposed processes require the addition of refrigeration to liquid hydrogen, using either hydrogen, helium or a mixture of both to provide the refrigeration. The processes of interest employ vacuum pumping (both straight and cascaded), external helium refrigeration, recirculation of a helium-hydrogen mixture and isentropic expansion of liquid hydrogen.

As an introduction to the body of this report, the five processes are summarized below. These brief summaries are not directly related to the immediate problem but constitute only a review of past work.

3.1 STRAIGHT VACUUM PUMPING

This is a batch process in which a container of liquid parahydrogen is vacuum pumped until the refrigeration, produced by evaporation of the vacuum-pumped hydrogen, cools the liquid to its triple point and partially solidifies it. The residual material in the container is the product slush hydrogen. The process is illustrated in Figure 1.

Process calculations were conducted in the manner illustrated in Technical Documentary Report APL-TDR-64-22. The process was assumed to consist of a series of small but finite isentropic expansions between the normal atmospheric boiling point and the triple point, followed by another isentropic expansion at the triple-point temperature and pressure as the slush is formed. The specific mass requirement (SMR) was defined as the mass required to produce a unit quantity of final material and values are listed in Table 1 of the cited report for producing liquid hydrogen at its triple point and solid hydrogen at its triple point. These values are as follows:

Liquid at triple point: $SMR_{ot} = 1.12269$

Solid at triple point: $SMR_{oq} = 1.26778$

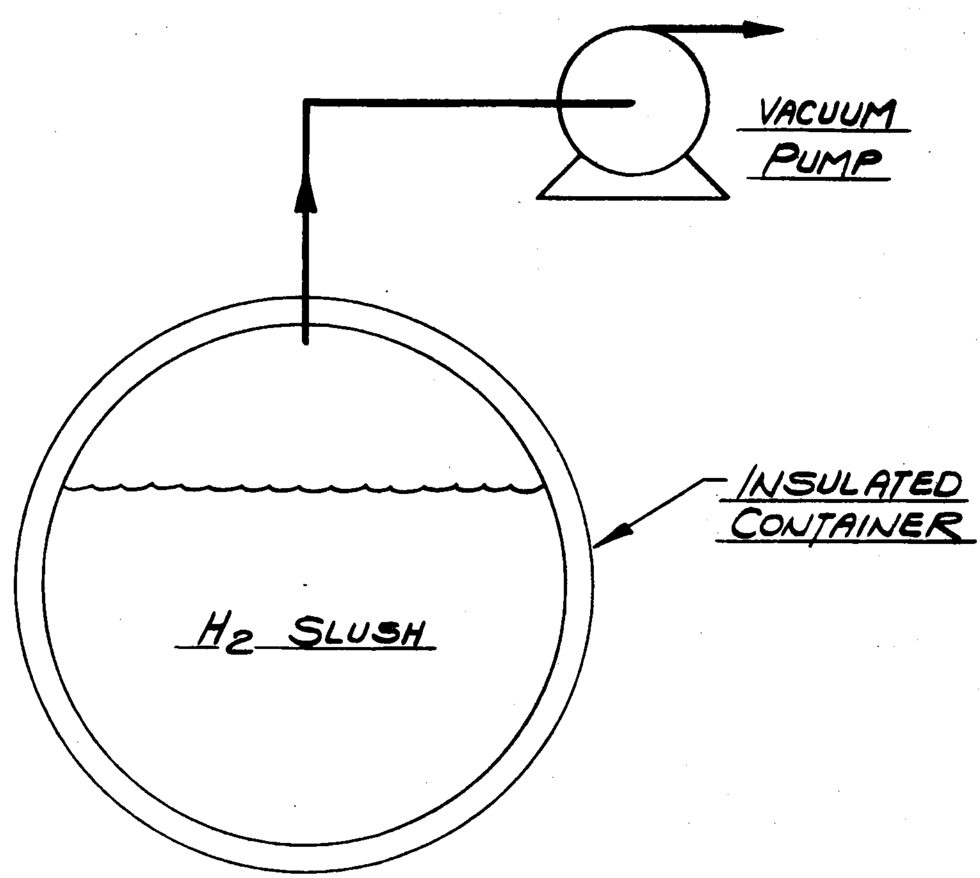


FIGURE 1

STRAIGHT VACUUM PUMPING PROCESS EQUIPMENT

The SMR for complete solidification of hydrogen from triple point liquid is:

$$SMR_{tq} = 1.12923$$

and for production of 50% slush from triple point liquid,

$$SMR_{tq} \ (q = 50\%) = 1 + \frac{50}{100} \ (1.12923-1) = 1.06462.$$

To produce 50% slush hydrogen from liquid hydrogen at its atmospheric boiling point thus requires:

$$SMR_{oq} \ (q = 50\%) = (1.12269) \ (1.06462) = 1.19523.$$

3.2 CASCADED VACUUM PUMPING

The cascaded vacuum pumping method is a continuous process in which liquid para-hydrogen is fed successively to several containers, each container being maintained at a lower pressure level than the preceding container by means of a series of vacuum pumps. The final container constitutes the slush-producing stage and the product slush hydrogen is discharged continuously from it. This process is shown in Figure 2.

The straight vacuum pumping batch process has an inherently higher thermodynamic efficiency than the continuous cascaded vacuum pumping process because the pressure above the liquid hydrogen is reduced gradually and continuously and, therefore, in a more reversible manner. Pressure reduction in the cascaded vacuum pumping process occurs in a series of discrete irreversible steps and can approach the efficiency of the straight vacuum pumping process only by the use of a very large number of stages.

3.3 EXTERNAL HELIUM REFRIGERATION SYSTEM

Manufacture of slush by refrigeration produced in an "external" cycle using helium as the working fluid is shown in Figure 3. In this cycle, helium is compressed to an appropriate superatmospheric pressure and precooled to a temperature close to that of hydrogen boiling point by means of liquid nitrogen and a countercurrent stream of cold low-pressure helium. The precooled helium is work-expanded to a temperature below the triple point of hydrogen and heat exchanged with the liquid triple-point hydrogen to produce a slush. The helium is then used to subcool the liquid hydrogen between the feed temperature and the triple point, followed by its return to ambient temperature through the aforementioned heat exchange with the compressed helium. Return of the helium to the inlet of the compressor completes the cycle.

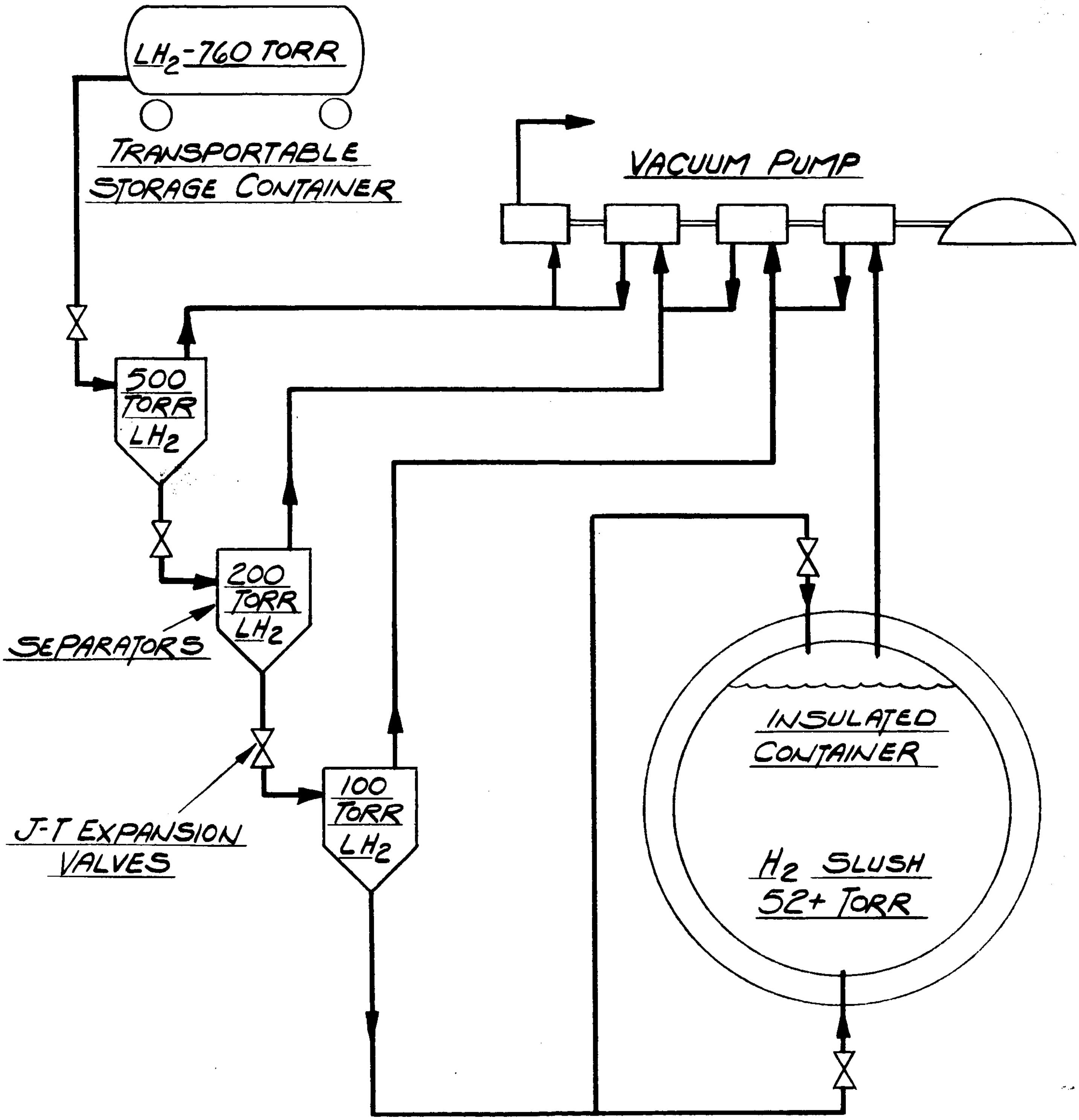


FIGURE 2

CASCADE VACUUM PUMPING PROCESS EQUIPMENT

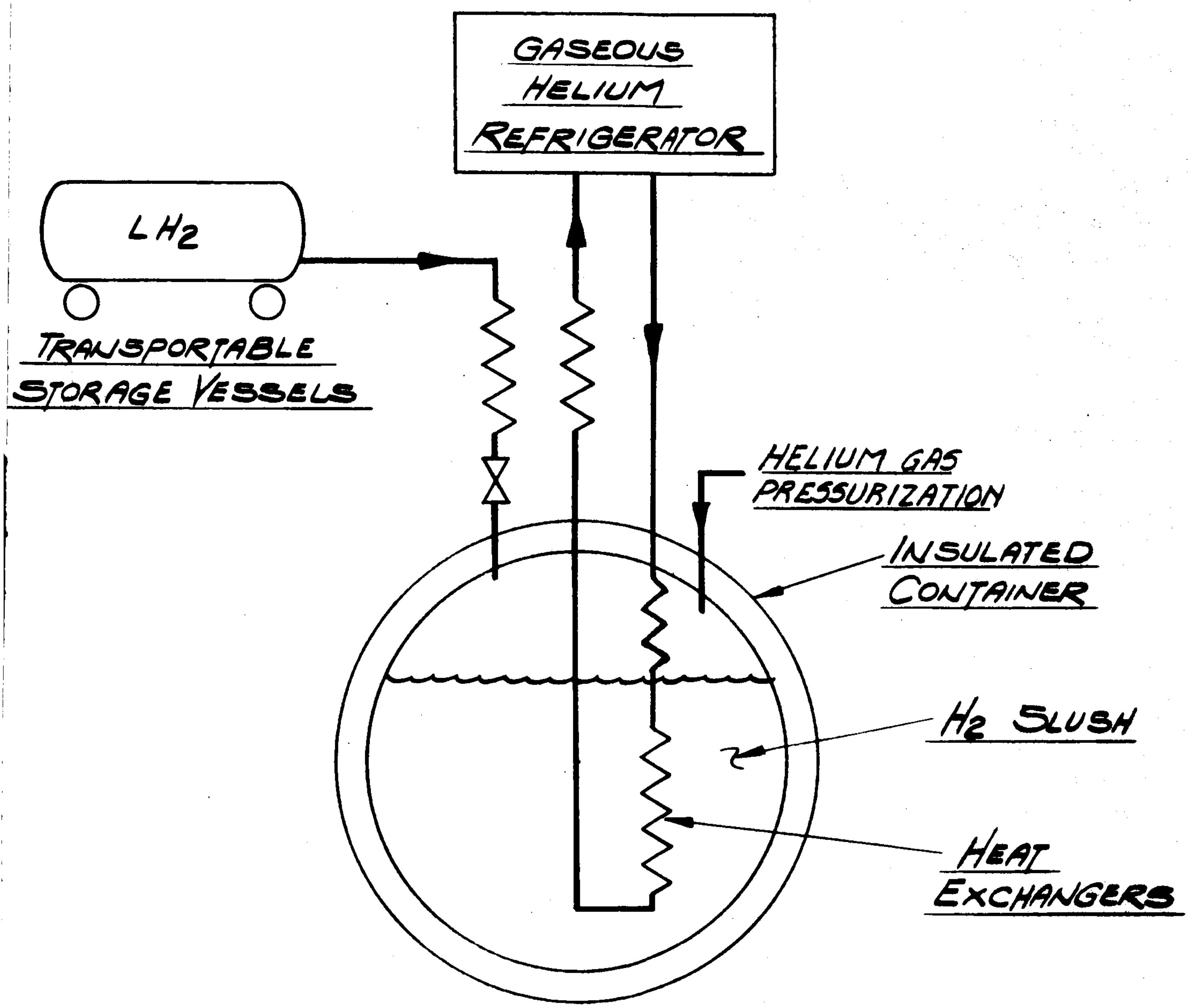


FIGURE 3

HELIUM GAS REFRIGERATOR COOLING, PROCESS EQUIPMENT

3.4 HELIUM-HYDROGEN VAPOR MIXTURE RECIRCULATION

This technique, shown in Figure 4, produces slush by means of refrigeration obtained in the Joule-Thomson expansion of a mixture of helium and hydrogen. An appropriate mixture of helium and hydrogen is compressed to a superatmospheric pressure and precooled to about the liquid hydrogen boiling point, the final stage of precooling being accomplished by heat exchange with boiling liquid hydrogen. The precooled mixture is further cooled by heat exchange with low-pressure gas returning from the slush chamber and is isenthalpically throttled into the slush chamber to produce a hydrogen slush which is continuously withdrawn. The equilibrium gas mixture is warmed to ambient temperature in heat exchange with the compressed feed and returned to the compressor. Make-up is obtained from the hydrogen evaporated in the precooler, which is fed to the compressor after warming in heat exchange with the compressed feed.

3.5 ISENTROPIC EXPANSION OF LIQUID HYDROGEN

APL-TDR-64-22 also proposed a cycle in which liquid hydrogen would have been compressed, the heat of compression and cycle losses removed by helium refrigeration, and the subcooled hydrogen liquid isentropically expanded to produce a mixture of liquid and solid hydrogen. Part of the product would have been used as the slush of suitable composition, while part of the liquid would have been recirculated.

Although there is a general dearth of information regarding liquid expansion machines, and also the reported efficiencies are extremely low, the cycle, at least in principle, is feasible with certain liquids (e.g. CO_2).

Unfortunately, as it turned out, the thermodynamic behavior of the liquid/solid hydrogen made this cycle unfeasible for hydrogen slush production.

Based on data resulting from an earlier study of the thermodynamic properties of liquid and solid hydrogen performed at Linde Division, and on NBS Report No. 7987, a Mollier diagram and a temperature-entropy diagram were constructed for the triple point region including the solid, solid-liquid, liquid region.

According to these diagrams (Figures 5 and 6), the cycle is not feasible. On both diagrams, the freezing curve, which separates the single-phase liquid region from the two-phase solid-liquid region, has a positive slope. This fact does not permit an isentropic pressure reduction to start in the liquid phase and end in the slush region. Therefore, an expansion engine could not accept liquid hydrogen and discharge slush hydrogen.

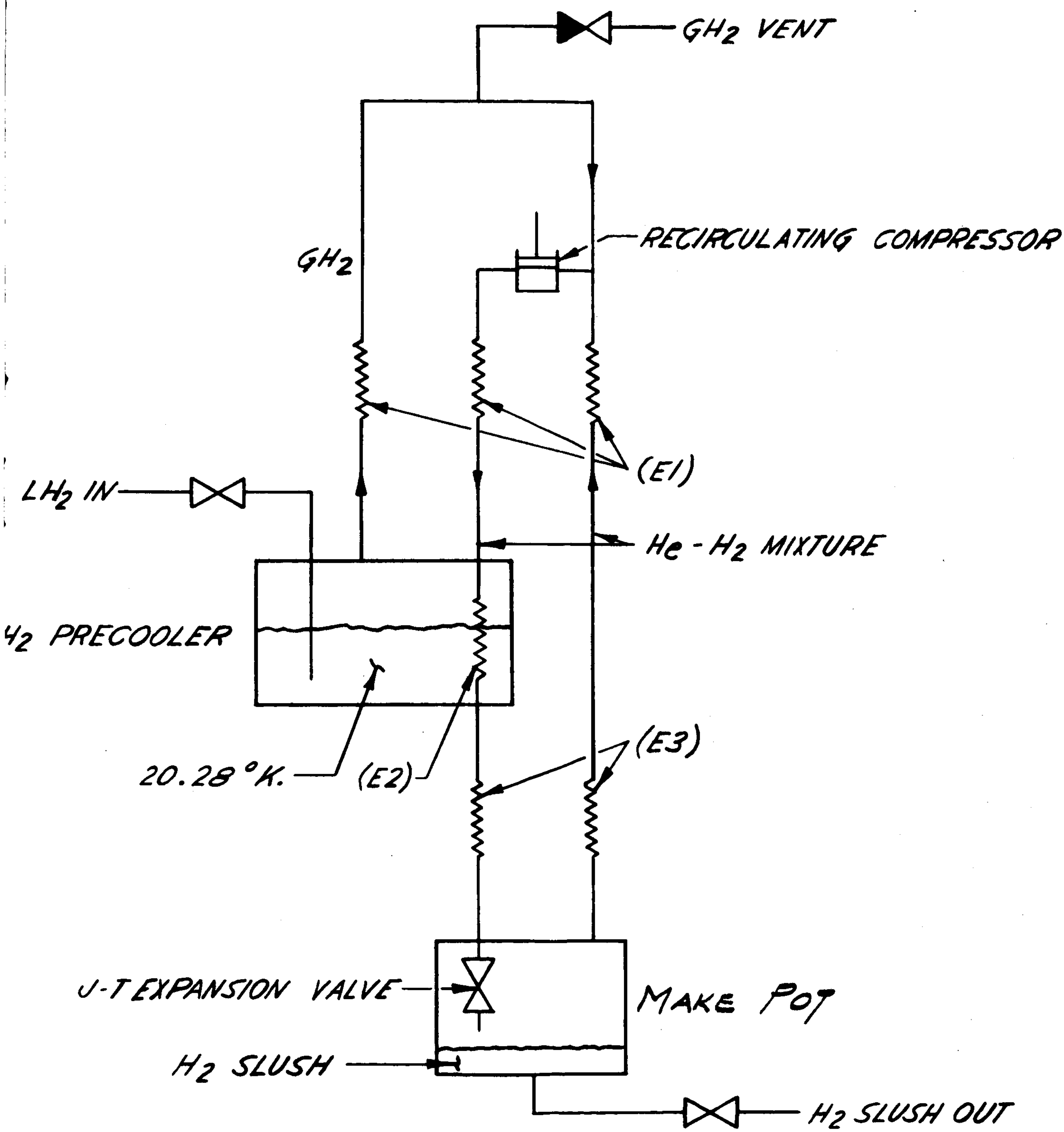
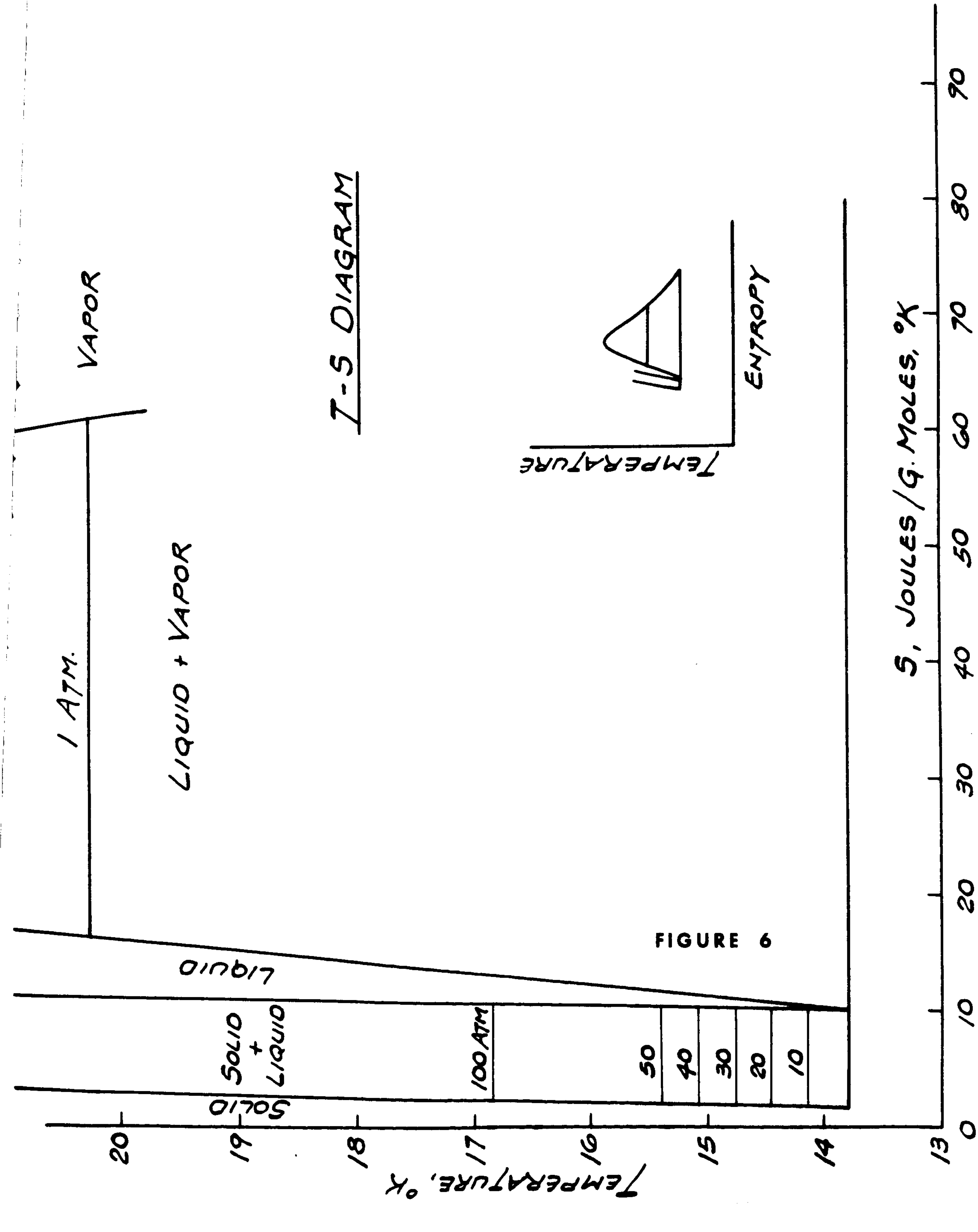


FIGURE 4

JOULE-THOMSON COOLING OF A HELIUM-HYDROGEN MIXTURE


T-S DIAGRAM
VAPOR
1 ATM.
LIQUID + VAPOR
LIQUID
SOLID + LIQUID
SOLID
100 ATM
50
40
30
20
10
TEMPERATURE
ENTROPY
S, JOULES/G. MOLES, °K
0
10
20
30
40
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60
70
80
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TEMPERATURE, °K
13
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17
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19
20

FIGURE 6

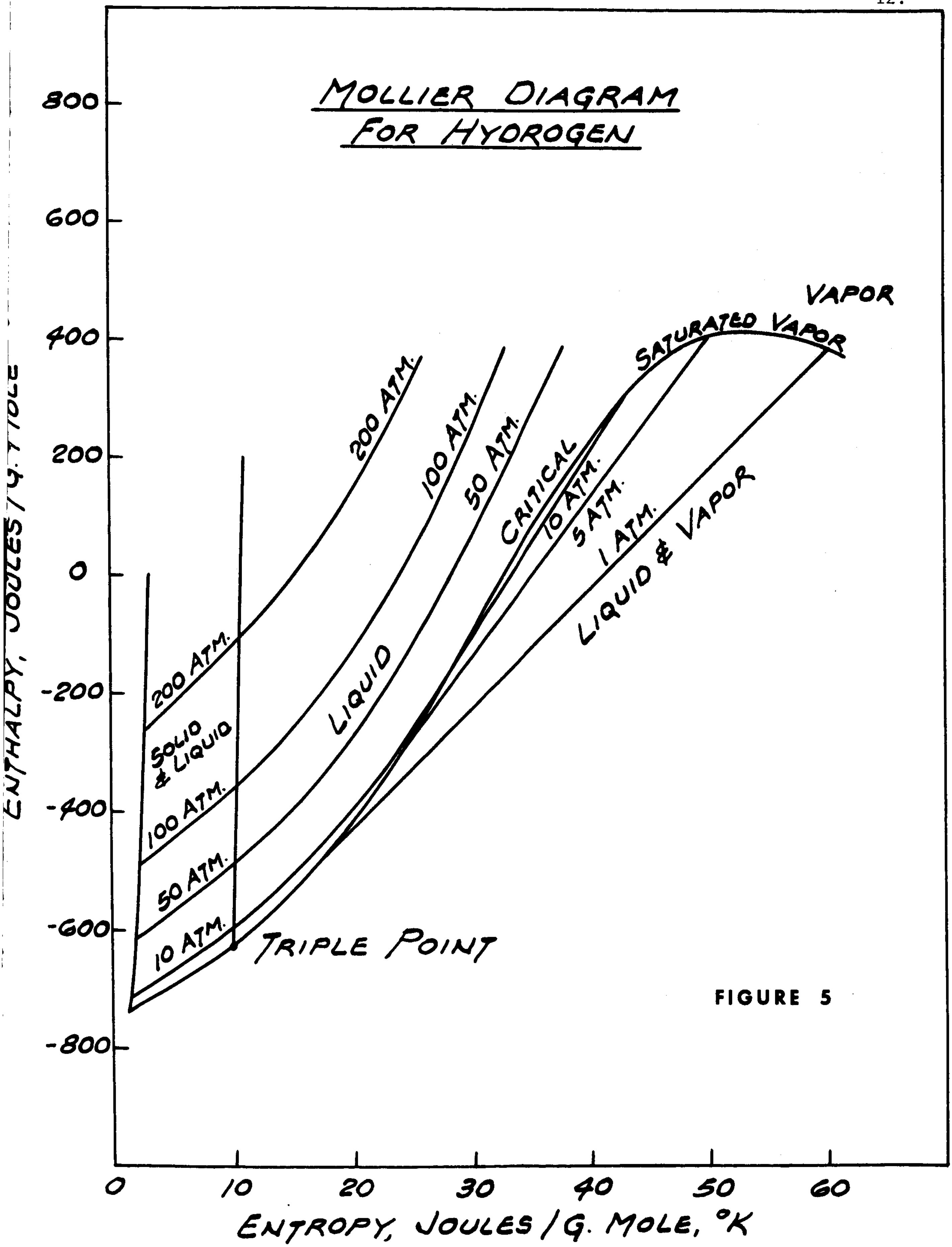

MOLLIER DIAGRAM
FOR HYDROGEN
800
600
400
200
0
-200
-400
-600
-800
ENTHALPY, JOULES / G. MOLE
0
10
20
30
40
50
60
ENTROPY, JOULES / G. MOLE, °K
VAPOR
SATURATED VAPOR
200 ATM.
100 ATM.
50 ATM.
CRITICAL
10 ATM.
5 ATM.
1 ATM.
LIQUID & VAPOR
LIQUID
200 ATM.
SOLID & LIQUID
100 ATM.
50 ATM.
10 ATM.
TRIPLE POINT

FIGURE 5

SECTION 4

SLUSH HYDROGEN REQUIREMENTS OF THE NUCLEAR ROCKET TEST PROGRAM

The liquid hydrogen requirements of the test program at Jackass Flats are defined in some detail in "Criteria for Budgetary Study for Engine/Stage Test Stand 2-3" prepared by the Space Nuclear Propulsion Office and issued August 25, 1965. This document does not consider the use of slush hydrogen in the program but sufficient information is given to enable some reasonable assumptions to be made.

4.1 CRITERIA

Early in the study several criteria were adopted in order to define the slush requirements of the test program. These criteria, which are listed below, have been maintained throughout.

4.1.1 Slush Quality

The quality of the slush is defined as the per cent solid by weight in the fluid.

4.1.2 Test Duration

An engine run of 30 minutes at 343 lbs. per second of 50% slush is considered as a normal test. An additional 10,000 lbs. of slush will be required for engine start-up.

4.1.3 Test Frequency

One normal test every 30 days is assumed.

4.1.4 Slush Storage Capacity

Sufficient tankage is assumed to store 105 per cent of the total slush necessary for one start-up and normal test.

4.1.5 Liquid Hydrogen Storage

It is assumed that conventional liquid hydrogen will be used for all support functions such as cooldown of cryogenic components, shutdown of the reactor, provision of purge and pressurization gas, etc.

4.1.6 Tank Module

Design of the tank module to hold 266,000 lbs. of slush (i.e., same volume as 230,000 lbs. of conventional liquid) is assumed.

4.1.7 Production System Capacity

A production system capacity of 1500 lbs. per hour of 50% slush has been adopted for the determination of equipment sizes and production costs.

4.2 STATEMENT OF THE PROBLEM

Using these criteria, the essential problem presented by this contract was reduced to a single sentence - "Make a conceptual design for a system most likely to accomplish the accumulation of 660,000 lbs. of 50% slush hydrogen in a 30 day period in storage tanks at Jackass Flats, Nevada."

In general, where alternatives were considered, the technically simplest and most developed solution was adopted, unless the economics were most unfavorable. Very little was known of the proposed duration of the test program (i.e., the total amount of slush to be produced) but a reasonably short program was assumed. With this in mind, low capital investment was generally favored over low operating cost in making economic choices.

SECTION 5

APPLICATION OF PROPOSED PRODUCTION PROCESSES TO THE PRESENT PROGRAM

It can be seen from the discussion in Section 3 that four proposed processes merited further consideration in the present study:

1. Straight vacuum pumping

2. Cascaded vacuum pumping

3. External helium refrigeration

4. Helium-hydrogen vapor recirculation

These processes were evaluated using technical feasibility, economics and current state-of-the-art as principal factor governing the selection of the optimum system. Results of the evaluation narrowed the choice to straight vacuum pumping and external helium refrigeration. This does not imply that all areas of uncertainty have been removed, and a considerable amount of development will be required, either before constructing the equipment or after constructing but before operating it as a production unit. Areas of uncertainty that do exist and for which additional information must be obtained will be cited in the appropriate sections of this report.

5.1 PRODUCTION SYSTEM CAPACITY

Using the criteria established earlier, a production capacity of 1500 lbs. per hour of 50% slush was used in comparing the four processes. This would provide the required 660,000 lbs. of slush (i.e., one test firing) in a 30 day period, operating 24 hours per day and 18.3 days in the 30 day period. An additional 3 operating days per month will be required to make allowance for deterioration in the slush quality during storage.

5.2 STRAIGHT VACUUM PUMPING

The straight vacuum pumping process is illustrated in Figure 7. Liquid hydrogen is held in storage until such time as it is withdrawn and delivered to one of the slush production tanks. The transfer between the liquid hydrogen storage and the slush production tank can be effected by either exerting pressure in the gas space of the storage tank or by a liquid hydrogen pump in the transfer pipe line. Liquid hydrogen arrives in the slush production tank saturated at 1 atm. pressure. The transfer line valve is closed and the vacuum pump started. Reduction in pressure above the liquid causes some of the liquid to evaporate, thereby cooling the remainder of the liquid. The process is continued until the triple

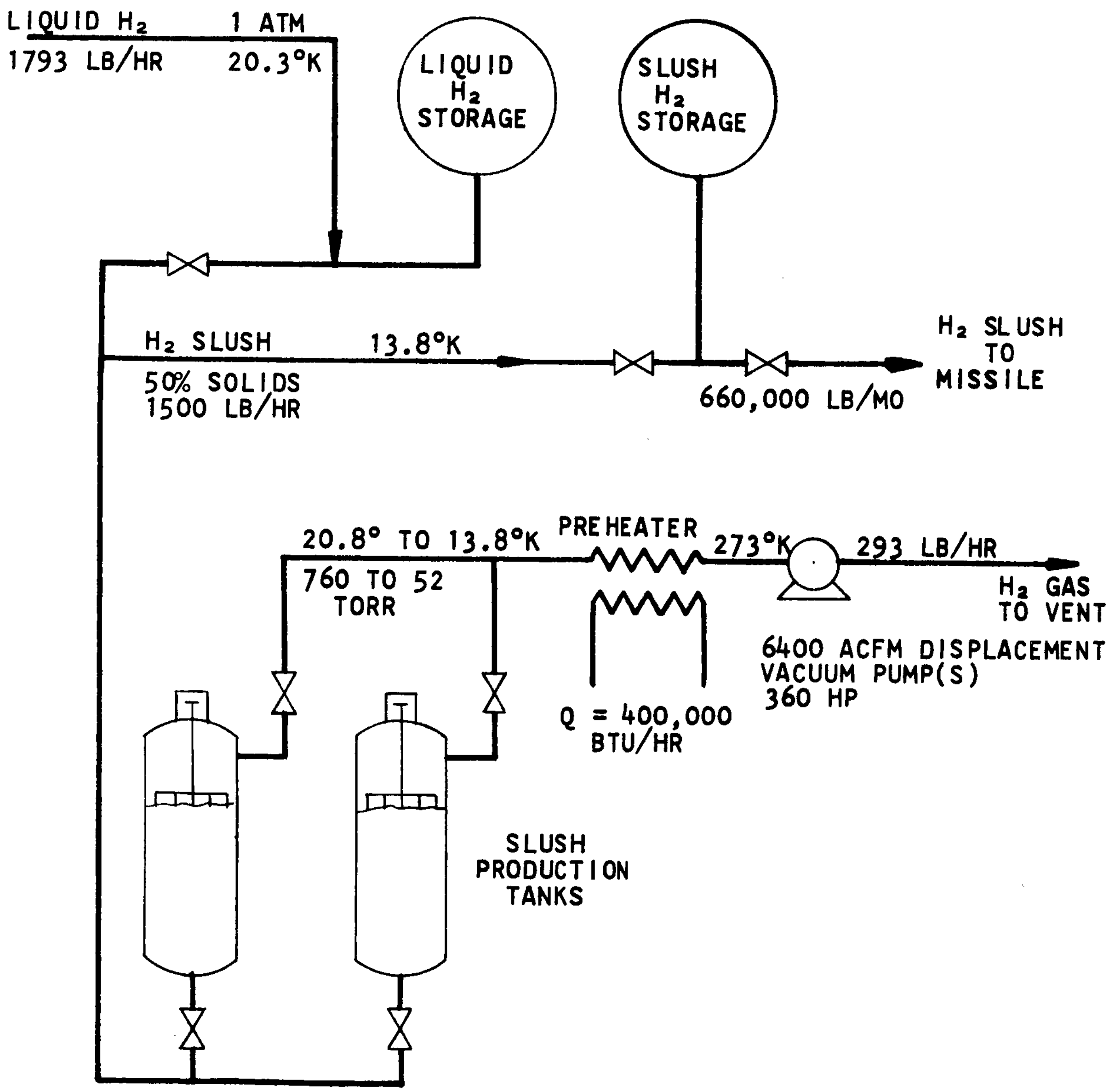


FIGURE 7

FLOW DIAGRAM-STRAIGHT VACUUM PUMPING PROCESS point of the hydrogen is reached, after which continued pumping causes solidification of part of the hydrogen. The vacuum pumped vapor which evolves from the liquid in the slush production tanks is warmed by suitable means to the required suction temperature of the vacuum pump. The hydrogen gas which exhausts from the vacuum pump is vented from the process to the atmosphere, flared, or recovered.

Upon reaching the desired slush quality, the vacuum pumping is transferred to the alternate production tank which, in the meanwhile, has been filled with liquid hydrogen. During vacuum pumping on the second slush tank, the slush in the first tank is aging. After the proper aging period, the slush is transferred to storage and the first production tank again filled with liquid, thereby completing the cycle.

From the process calculations of paragraph 3.1, a production rate of 1500 lb. per hour of 50% slush requires a feed rate of:

$$1500 \times 1.19523 = 1793 \text{ lb. per hour.}$$

The difference of 293 lb. per hour is the mass of hydrogen which must be vaporized to produce the required slush.

5.3 CASCADED VACUUM PUMPING

For large production capacities, continuous processes are usually preferred to batch processes. The straight vacuum pumping process is inherently a batch process while the cascaded vacuum pumping method is a continuous or at least a semi-batch process. Therefore, the cascaded vacuum pumping process was studied to determine whether its adaptability to continuous processing would produce any process advantages.

This process is illustrated in Figure 8. Liquid hydrogen is brought to the production system by any suitable means such as transport tankage or the product delivery line of a hydrogen liquefier. Suitable storage is provided from which liquid hydrogen, saturated at 1 atm. of pressure, is continuously withdrawn. The liquid is then throttled through a valve and fed to the first of a series of vacuum phase separators. The gas phase is withdrawn, preheated to a suitable temperature, and vacuum pumped; the liquid is throttled and passed to the next separator. Each separator is in turn at a successively lower pressure and the final stage is the slush production tank, maintained at the hydrogen triple point. Slush produced in the production tank is continuously discharged to storage while the vapor is preheated and fed to the suction side of the appropriate vacuum pump. Hydrogen gas exhausting from all vacuum pumps is collected, discharged to the atmosphere, or flared.

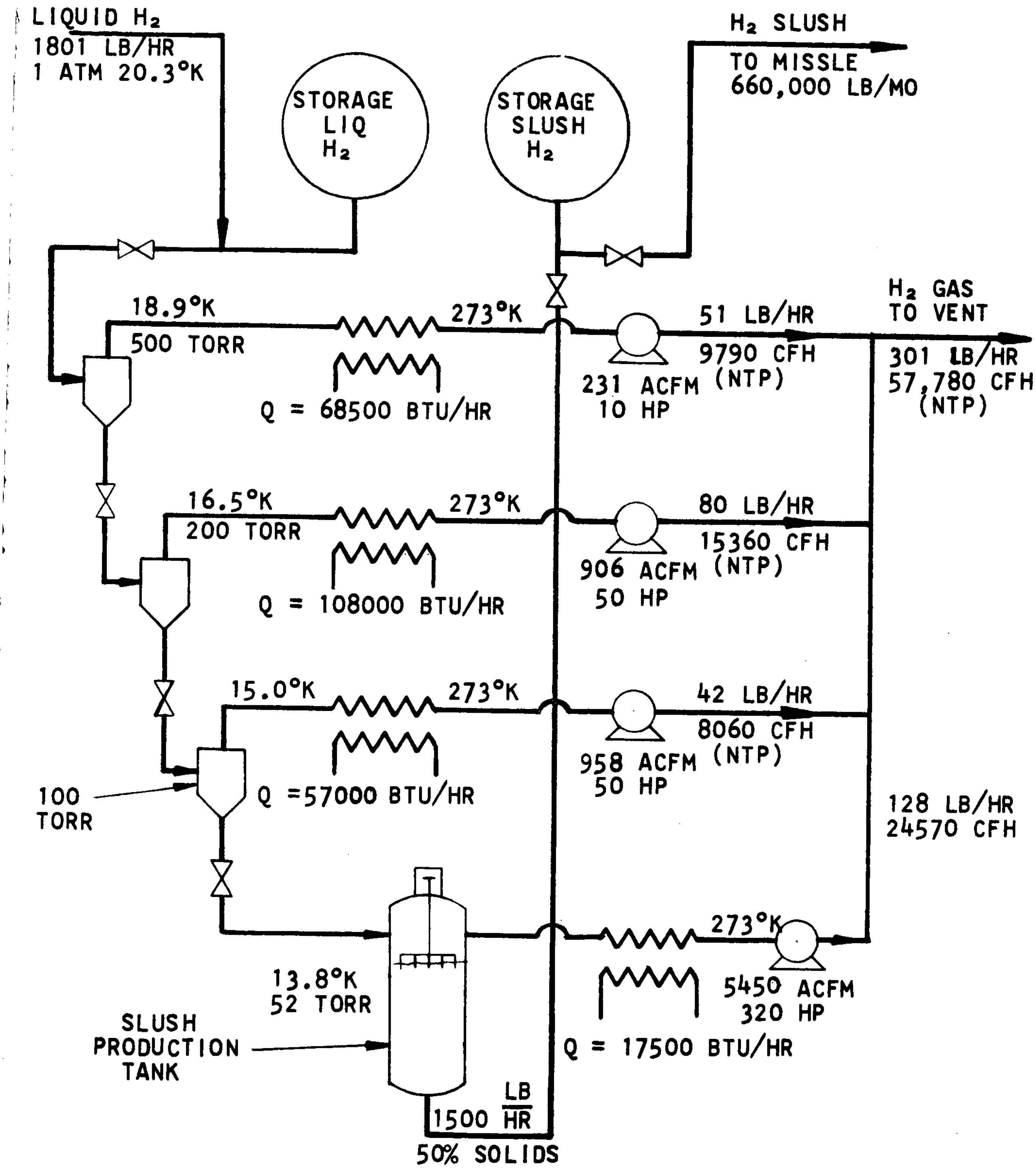


FIGURE 8

FLOW DIAGRAM-CASCADE VACUUM PUMPING PROCESS

Process requirements can be expected to be greater for the cascaded vacuum pumping process than for the straight vacuum pumping process because the former consists of several isenthalpic throttling steps which are thermodynamically less efficient than the isentropic steps of the straight vacuum pumping process. The difference is revealed by comparison of the process requirements which are listed in Table 1. In each case, warming of the exhaust gas to ambient before vacuum pumping has been assumed.

TABLE 1

COMPARISON OF PROCESS REQUIREMENTS

CASCADED VS. STRAIGHT VACUUM PUMPING

	Cascaded	Straight
Feed rate, lb/hr.	1,801	1,793
Vacuum pumped hydrogen, lb/hr.	301	293
Vacuum pumped hydrogen, ACFM	7,545	6,400
Pump Horsepower	430	360

Whether the cascaded vacuum pumping process would be continuous or a semi-batch would depend greatly upon certain slush transport characteristics which currently fall in the area of uncertainty. First, the effect of slush aging upon transport characteristics is not well known. If slush must be aged prior to transporting it, then a semi-batch process would be favored. Second, a continuous process requires that a pump be used to transfer slush from the production to the storage tank. If a pressure transfer must be used, then the process must again be semi-batch. The semi-batch concept would apply only to the slush production tanks, which would be provided in duplicate for alternate operation. The rest of the equipment would be single-train and the production tanks would be switched on and off the line in accordance with a predetermined schedule.

5.4 EXTERNAL HELIUM REFRIGERATION SYSTEM

This process produces slush by transferring refrigeration, which is generated in an external process, to the liquid hydrogen which is fed to the process. Helium is used in the refrigeration cycle because it is the only material which remains fluid below the hydrogen triple point temperature. Figure 9 illustrates this process. Considering first the refrigeration loop, helium is compressed to a suitable pressure (285 psig in this example), aftercooled, and fed to the first stage heat exchanger of the refrigeration cycle. The helium emerges at approximately 80 - 81^{o}K, is further cooled against boiling liquid nitrogen and then passes through a silica gel adsorption bed to remove any materials which would freeze at the lower temperature level. The helium is then additionally precooled before being admitted to the work expander (expansion engine) from which it emerges at the lowest temperature in the cycle. Passage of the helium through the coil in the slush production tank removes heat from the liquid hydrogen in the tank in sufficient amount to produce 50% slush. The helium, which has been warmed to nearly the hydrogen triple point temperature, leaves the slush production tank and passes through the liquid hydrogen subcooler, cooling the liquid hydrogen feed to its triple point. The helium is then warmed to ambient temperature in countercurrent heat exchange with the incoming compressed helium and returned to the suction side of the compressor, completing the cycle. Liquid nitrogen is also fed to the process to forecool the compressed helium. The sensible refrigeration in the evaporated nitrogen is recovered by passing it in countercurrent heat exchange with the helium feed in the first stage three-pass heat exchanger.

The flowsheet shown represents one of three refrigeration cycles which were evaluated to determine the effect of head pressure. Several conditions were maintained constant. These were:

1. A temperature approach of 1.5^{o}C at the helium effluent of the nitrogen forecooler.

2. A temperature approach of 2.6^{o}C at the hydrogen inlet of the liquid hydrogen precooler.

3. An expander exhaust pressure of 1.5 atmospheres.

4. Liquid hydrogen feed saturated at 10 psig (21.3^{o}K).

5. A liquid nitrogen feed saturated at 1 psig (78^{o}K).

6. An efficiency of 70% on compression and expansion machinery.

Head pressures of 20, 40 and 60 atmospheres were selected and process requirements were determined for each head pressure. These requirements are presented in Table 2.

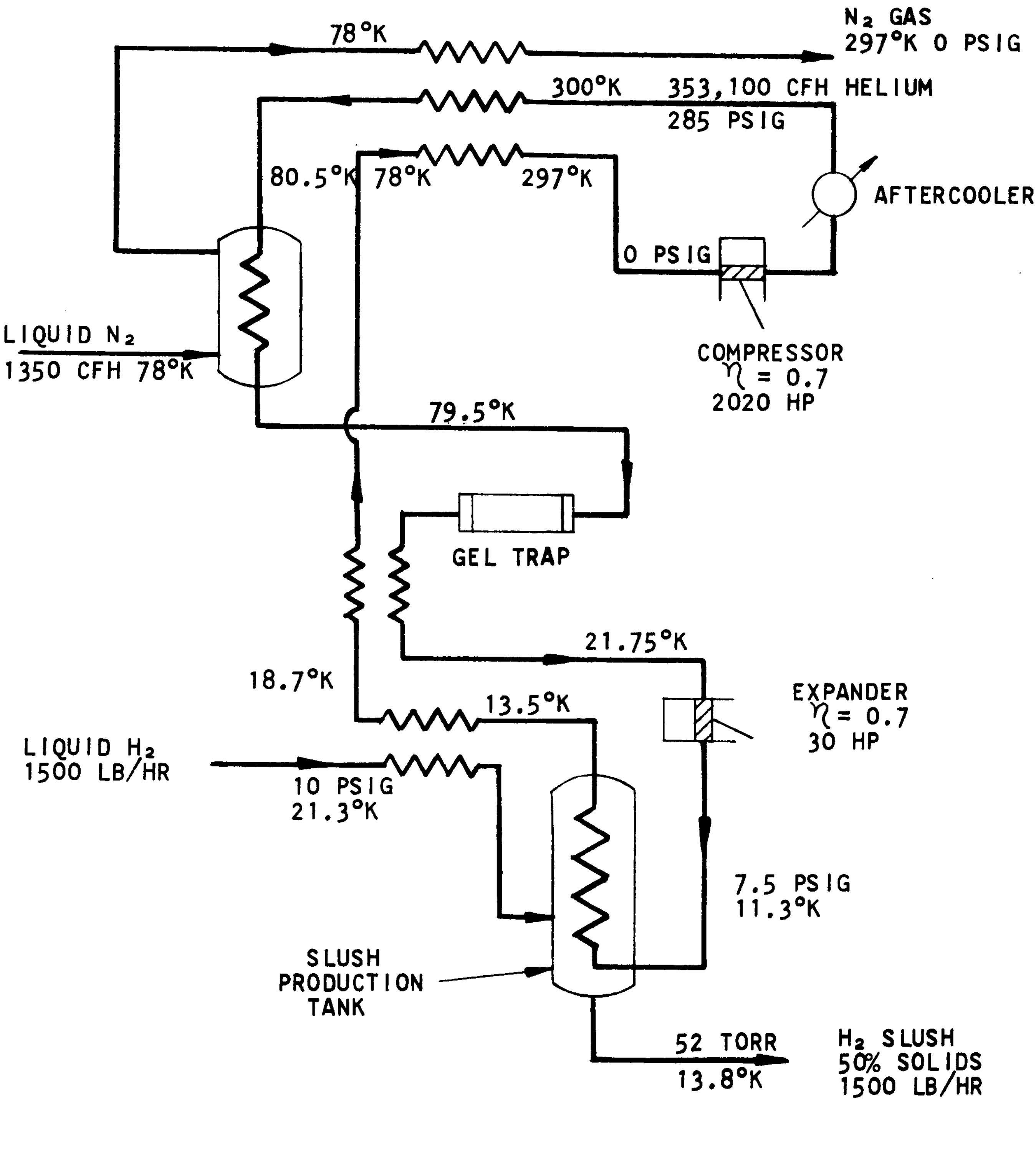


FIGURE 9

FLOW DIAGRAM-EXTERNAL HELIUM REFRIGERATION PROCESS

TABLE 2

EFFECT OF HEAD PRESSURE ON

HELIUM REFRIGERATOR PROCESS REQUIREMENTS

Head Pressure, Atm.	20	40	60
Helium flow, cfh (NTP)	353,100	305,300	296,600
Compressor power, HP	2,020	2,110	2,320
Expander output, HP	30	31	33
Liquid nitrogen, cfh (NTP)	1,350	1,765	2,060

Both power and liquid nitrogen consumption are at a minimum for the 20 atm. head pressure. Further reduction in head pressure was not attempted because it was apparent that the helium recirculation rate was increasing rapidly and that the increase in equipment size would overcome any gain achieved through reduced process requirements.

5.5 HELIUM-HYDROGEN VAPOR MIXTURE RECIRCULATION SYSTEM

In this process a suitable mixture of precooled helium and hydrogen is subjected to a Joule-Thomson throttling in such a manner that slush hydrogen of the desired quality emerges from the throttling valve. The process is shown in Figure 10. A helium-hydrogen mixture containing 20.7% hydrogen is compressed to 25 atm. This pressure was chosen because it produces the maximum Joule-Thomson cooling at the final throttling valve. The mixture is aftercooled to ambient temperature, followed by cooling in countercurrent heat exchange with the returning low pressure streams to within a few degrees of liquid hydrogen temperature.

It is now forecooled against boiling liquid hydrogen at atmospheric pressure, further precooled by means of the cold expanded helium-hydrogen and throttled into the slush production tank. Slush is withdrawn from the production tank and the gas phase is warmed and recycled to the compressor.

Most of the hydrogen which is fed to the precooler eventually leaves the process as slush product; the excess is provided for refrigeration purposes. Sensible refrigeration in this hydrogen vapor is recovered by heat exchanging it with the incoming feed stream. The excess hydrogen is vented from the system.

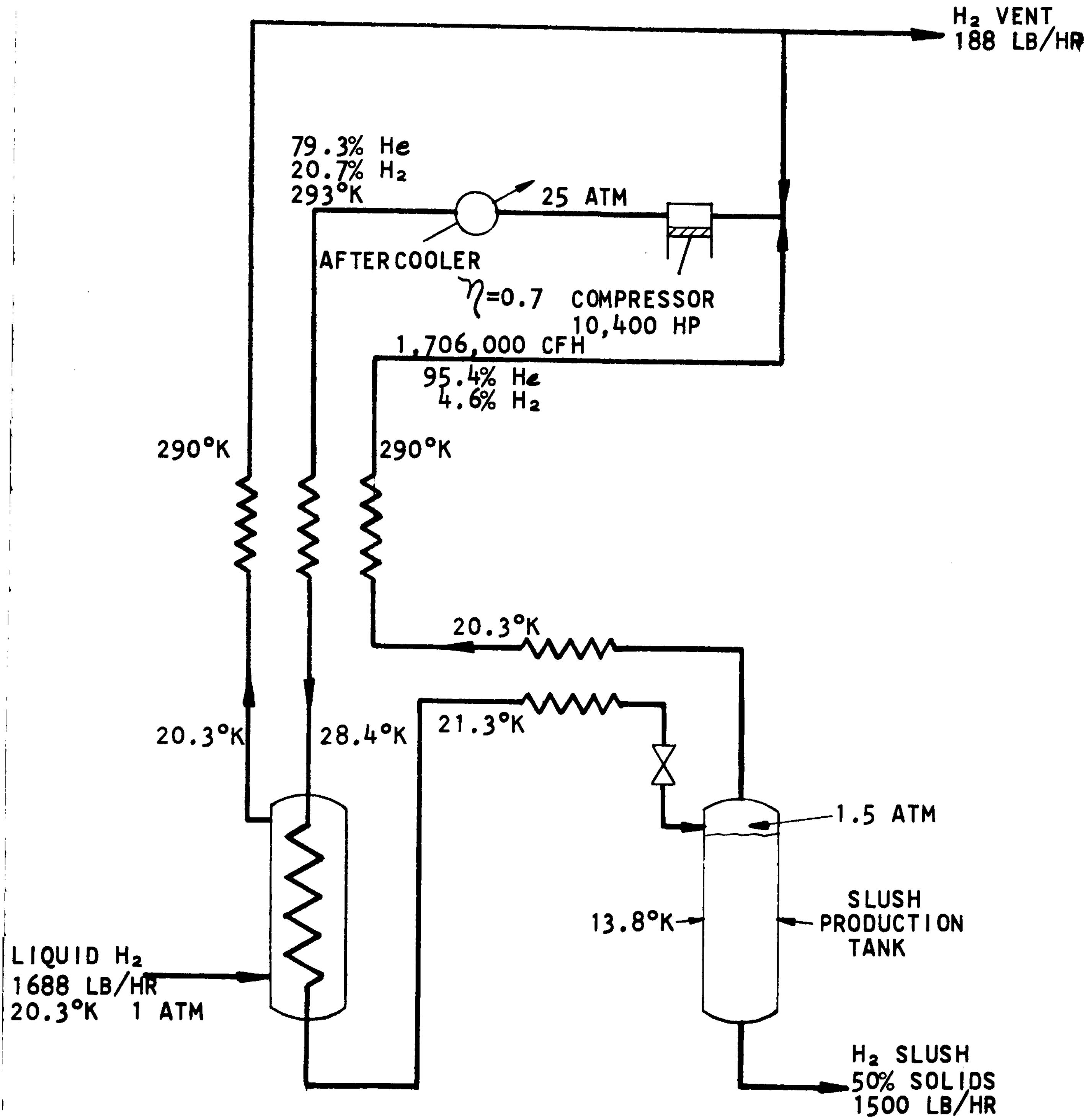


FIGURE 10

FLOW DIAGRAM

HELIUM/HYDROGEN MIXTURE REFRIGERATION PROCESS

The compression requirement of 10,400 HP exceeds that of the external helium refrigeration system by more than a factor of five. This results from the low efficiency of the Joule-Thomson throttling process as applied to helium-hydrogen mixtures. In this temperature region, the hydrogen liquid undergoes a temperature rise upon throttling so that the helium must not only provide the Joule-Thomson refrigeration to sustain the process but also it must provide additional refrigeration to overcome the adverse behavior of the hydrogen. This unfavorable situation is further aggravated by the low Joule-Thomson coefficient for helium.

One of the principal attractions of this system is that it permits production of slush with the tank maintained at a pressure equal to or greater than the surrounding atmosphere. Vacuum pumps or vacuum producing equipment are, therefore, not needed, which may enhance the safety of operation. Pressurization of the production tank permits continuous transfer of the product slush to a storage vessel maintained at atmospheric pressure, with the assumption, of course, that unaged slush is, indeed, transferrable in this manner.

The production tank in the process is pressurized at 1.5 atmospheres for this reason. It is, of course, possible to maintain the pressure in the slush production tank closer to one atmosphere, with tank pressurization effected only when a transfer is to be made. This would convert the process to a semi-batch type operation for which duplicate production tanks would be used. Reduction of the tank pressure to approximately atmospheric pressure would increase the pressure difference across the throttling valve and increase the refrigeration. This would result in a lower helium recycle rate and a reduction in compression requirements. Although some reduction in process requirements can be achieved by this reduction in tank pressure, the reduction cannot be expected to be great enough to make this process competitive with the external helium refrigeration process.

5.5.1 Thermodynamic Data for Mixtures

In determining energy balances for the helium-hydrogen vapor mixture recirculation process, only the simplest concepts were employed in the calculation of the thermodynamic properties of the mixtures involved. Although it is recognized that, at the low temperatures encountered in the process, significant quantum effects exist, both these effects and the effects of interactions between the molecular species have been ignored. The enthalpy of a mixture was thus assumed to be a mean value of the enthalpies of the pure components, weighted in accordance with the mixture composition. This procedure was considered adequate for this process, which was shown to have process requirements greatly in excess of the other processes. Had this process been more nearly competitive, more rigorous thermodynamic calculations would have been employed.

5.6 PROCESS SELECTION

In making a technological evaluation of the various processes, the guiding principal which was maintained was that the selected process must have characteristics which would permit equipment to be designed, built, and operated with the greatest assurance of success and require a minimum of development work in support of the design. It is recognized that many areas of ignorance and uncertainty exist and these areas must be minimized in selecting the process and either eliminated or circumvented in designing the equipment.

By inspection, the choice of processes can be narrowed to two:

1. Straight vacuum pumping

2. External helium refrigeration

The helium-hydrogen vapor mixture recirculation process may be discarded on the basis of the extremely high power requirement and the high recycle rate which results in high equipment costs. The cascaded vacuum pumping process may be discarded on the basis that, if vacuum pumping is to be employed, the straight vacuum pumping process is the simpler and has lower hydrogen feed, power, and equipment requirements.

SECTION 6

DISTRIBUTION OF SLUSH HYDROGEN

From the criteria established in paragraph 4.1, it was apparent that continuous topping-off of the tank module will be required during a test run. Thus it was seen that "on-board" production of slush would not meet the requirements of the test program and that transfer of slush between vessels was an absolute necessity.

6.1 TRANSFER CHARACTERISTICS OF SLUSH HYDROGEN

After discussions with those within Linde's Engineering and Research Departments who were most familiar with slush hydrogen, it appeared reasonable to assume that slush of 50 to 55% quality could have acceptable flow characteristics. This assumption was supported by the publication of National Bureau of Standards Report No. 8881 which reports the results of some simple transfer tests. At the same time, it was recognized that the subcooling of liquid hydrogen could, under some circumstances, result in large "lumps" which could not be considered as slush.

It was then decided that, for the balance of this study, it was necessary to assume that 50% slush could be made to flow through fairly simple, well-designed transfer systems. This does not, of course, mean that the problem of slush transfer can be minimized. Continued investigation of flow characteristics is still of paramount importance.

6.2 SLUSH PLANT LOCATION

Three alternatives were considered:

1. Location of the slush facility at the test site.

2. Location of the primary slush facility at a commercial liquid hydrogen plant with a secondary facility at the test site to reconstitute the slush.

3. Location of the facility at a commercial liquid hydrogen plant, making a high quality slush with no reconstituting at the test site.

Alternate (3) appeared to be economically attractive and was given some consideration. However, it was found that slush qualities of 60 to 90% would be required at the plant if slush of 50% quality was to be available at the site. Since it could not be predicted that a slush of over 50% quality would flow readily, this approach was technically unattractive.

A modification was considered in which the transport vehicle (trailer or tank car) was used as the slush producing facility, producing a high quality slush which would deteriorate to 50% quality in-transit. This approach assumed the formation of a flowable material from the high quality slush but the distinct possibility exists that a large "lump" may be formed in the triple-point liquid. Since convincing evidence on this point could not be found, this approach, which may be economically the most attractive, was shelved.

Transportation of slush (Alternate 2) was recognized as a long-range objective which would be attractive to NASA but would add greatly to the flow and transfer problem. However, in spite of this disadvantage this alternative was retained for economic evaluation along with the most obvious solution (Alternate 1).

Once the transfer problem has been solved, several modifications to Alternate 2 can be considered, including the return of excess triple-point liquid from the site to the production plant. These variations are discussed in paragraph 8.5

SECTION 7

SELECTION OF THE RECOMMENDED SYSTEM

From the analysis of the various production cycles described in Section 5 and from the distribution systems study described in Section 6, it was seen that final definition of the system could be reached by a more detailed study of four cases.

7.1 STUDY CASES

The operations involved in the four cases selected for economic and technical evaluation are described briefly as follows:

7.1.1 Case 1

a. Make 50% slush at liquid hydrogen plant by straight vacuum pumping.

b. Transport slush to test site by road or rail.

c. Upgrade to 50% at site by vacuum pumping and store.

7.1.2 Case 2

a. Transport liquid hydrogen to site by road or rail.

b. Make 50% slush at site by straight vacuum pumping and store.

7.1.3 Case 3

a. Make 50% slush at liquid hydrogen plant by external helium refrigeration.

b. Transport to site by road or rail.

c. Upgrade to 50% at site by vacuum pumping and store.

7.1.4 Case 4

a. Transport liquid hydrogen to site by road or rail.

b. Make 50% slush at site by external helium refrigeration and store.

7.2 CAPITAL INVESTMENT

In order to evaluate these four cases, preliminary estimates of the capital investment required in each case were made. It must be emphasized that these are rough preliminary estimates only. A detailed cost estimate of the selected system will be presented later in this report.

In Cases 1 and 3, the size of the upgrading plant required at the test site is influenced by the heat leak into the slush during shipment from the hydrogen plant and, therefore, the distance of the producing plant from the site is a factor. Estimates have been made based on plants at 320 miles and 1800 miles from the Nevada test facility, representing the extremes of the existing large commercial hydrogen plants.

Using the criteria established in Section 4, it is assumed that 660,000 lbs. of slush will be required every 30 days. The estimates in Table 3 are based upon a plant to produce this quantity in 18 working days, i.e., about 18 tons per day capacity.

TABLE 3

CAPITAL INVESTMENT

PRELIMINARY ESTIMATES

Distance, liquid plant to test site	320 Miles	1800 Miles
Case 1		
At liquid plant	$ 637,000	$ 637,000
At test site	340,000	473,000
Total	$ 977,000	$1,110,000
Case 2		
At liquid plant	$ 0	$ 0
At test site	696,000	696,000
Total	$ 696,000	$ 696,000
Case 3		
At liquid plant	$2,780,000	$2,780,000
At test site	340,000	474,000
Total	$3,120,000	$3,254,000
Case 4		
At liquid plant	$ 0	$ 0
At test site	2,780,000	2,780,000
Total	$2,780,000	$2,780,000

7.3 OPERATING COSTS

Preliminary estimates of the operating costs on a "per month" basis for the four cases are given in Table 4. In Case 1, credit is given for the return of the gaseous hydrogen from the vacuum pumps to the liquid plant at fuel value. No credit is given for gaseous hydrogen at the test site as it is assumed that the vacuum pump discharge will be vented. The cost of the liquid hydrogen has not been included.

TABLE 4

OPERATING COSTS PER MONTH

PRELIMINARY ESTIMATES

Distance, liquid plant to test site	320 Miles	1800 Miles
Case 1		
Labor	$ 7,950	$ 7,950
Utilities	1,540	1,885
	$ 9,490	$ 9,835
Credit, GH_2 returned	-2,640	-2,720
Total	$ 6,850	$ 7,115
Case 2		
Labor	$ 4,425	$ 4,425
Utilities	1,850	1,850
Total	$ 6,275	$ 6,275
Case 3		
Labor	$ 8,820	$ 8,820
Utilities	6,380	6,800
Total	$15,200	$15,620
Case 4		
Labor	$ 5,545	$ 5,545
Utilities	9,175	9,175
Total	$14,720	$14,720

7.4 LIQUID REQUIREMENTS

Making due allowance for transfer losses etc., the estimated monthly requirements of liquid hydrogen produced at the plant and transported to the site (either as liquid or as slush) are presented in Table 5.

TABLE 5

LIQUID HYDROGEN REQUIREMENTS, PER MONTH

(1 Month = 660,000 lb. 50% Slush)

Distance, liquid plant to test site	320 Miles	1800 Miles
Case 1		
Total LH_2 produced at plant	829,000 lb.	845,000 lb.
Total hydrogen transported (as slush)	687,000 lb.	700,000 lb.
Case 2		
Total LH_2 produced at plant	827,000 lb.	844,000 lb.
Total hydrogen transported (as liquid)	819,000 lb.	836,000 lb.
Case 3		
Total LH_2 produced at plant	701,000 lb.	714,000 lb.
Total hydrogen transport (as slush)	687,000 lb.	700,000 lb.
Case 4		
Total LH_2 produced at plant	687,000 lb.	700,000 lb.
Total hydrogen transported (as liquid)	680,000 lb.	693,000 lb.

7.5 ECONOMIC EVALUATION

Since the actual value which NASA will place upon liquid hydrogen delivered to the test site is not known at this time, no attempt has been made to estimate an actual cost for slush at the site. However, the information presented in the preceding three tables has been used to make estimates of the ratio of delivered cost of slush to the delivered cost of conventional liquid. These ratios are plotted versus the total number of tests to be run with slush in Figure 11.

These curves are somewhat sensitive to changes in the cost of liquid, the cost of delivery and the distance between the plant and the site. They are intended only for use in comparing the four cases presented here and should be used only with great discretion in estimating the actual cost of slush.

It can be seen that, if the capital investment is to amortized in less than 50 to 60 tests, Cases 1 and 2 (the vacuum pumping process) show the lowest costs. There is probably some overlap in the ranges of ratios between Cases 1 and 2, but Case 2 shows a cost advantage for a program of less than about 20 tests.

7.6 TECHNICAL EVALUATION

The transformation of liquid hydrogen into small particles of solid, dispersed through the liquid phase as slush, has been most amply demonstrated by means of vacuum pumping on a container of liquid hydrogen. This is the technique employed by Linde Division in their experimental investigation of slush hydrogen as reported in Technical Documentary Report APL-TDR-64-22. The Cryogenics Division, National Bureau of Standards, Boulder, Colorado, in their experimental work, as reported in N.B.S. Report No. 8881, also employed vacuum pumping as did the Research and Technology Division at Wright-Patterson Air Force Base.

This process is the simplest and most straightforward approach to slush production and is undoubtedly at a higher state of development than other proposed processes. The vacuum pump, which represents the heart of this process, was the subject of considerable study during this contract. It was concluded that suitable pumps operating at near ambient temperatures were commercially available but some development would be necessary to obtain a suitable cryogenic pump. A detailed discussion of this study follows later in this report.

The external helium refrigeration system employs a heat transfer surface immersed in liquid hydrogen, upon which solid hydrogen is formed. There is no background of experience for this technique and it is, therefore, not recommended. Anticipated difficulties and areas of uncertainty include:

PRELIMINARY ESTIMATE
SLUSH HYDROGEN COSTS

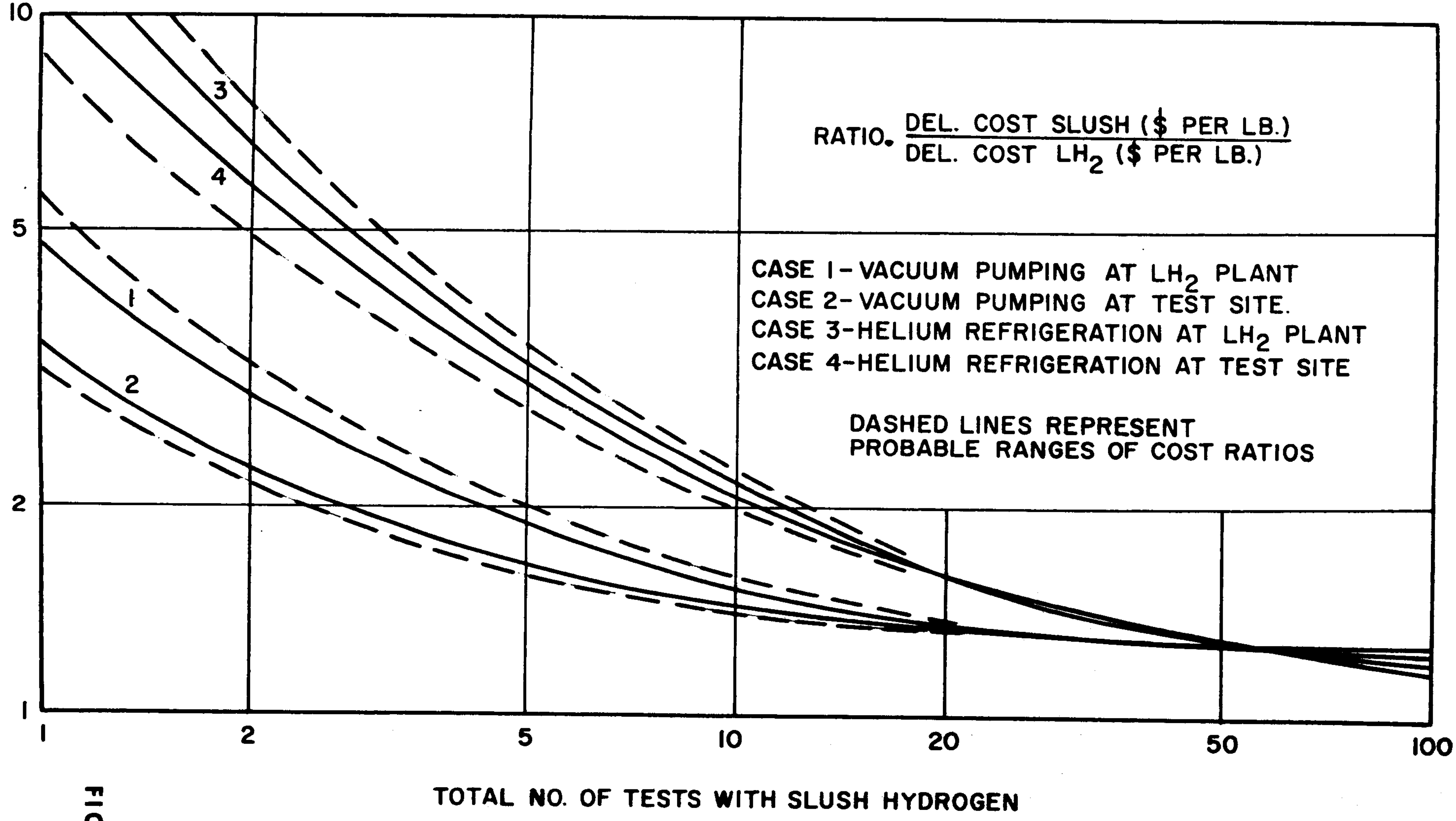


FIGURE 11

1. Removal of solid hydrogen from the heat transfer surface may be difficult.

2. The physical nature of solid hydrogen formed on a heat transfer surface is largely unknown.

3. Heat transfer characteristics from the refrigeration surface to solid hydrogen are unknown.

Formation of solid hydrogen by throttling precooled liquid through an orifice into an evacuated space has been demonstrated by N.B.S.'s Cryogenics Division. This is quite similar to the final process step in the helium-hydrogen vapor mixture recirculation system although, in the latter case, the presence of helium may result in some divergence from the demonstrated performance of the former case. This technique, therefore, remains to be proven.

7.7 RECOMMENDED SYSTEM

The apparent low state-of-the-art in the helium refrigeration process, together with the economic information presented in the preceding paragraphs seemed to justify the elimination of study cases 3 and 4 from further consideration.

Location of the slush plant at the liquid plant (Case 1) offers a number of attractive features (trained labor, cheap utilities, flexible distribution systems, etc.), but has one major disadvantage. Case 1 requires that the slush be transferred into transport vehicles, moved to the site and transferred to storage, involving flow problems much more complex than those encountered in Case 2. Since the latter may offer some economic advantage in a short test program, it was felt that Case 1 could be eliminated and Case 2 selected as the most likely system to achieve the effectiveness criteria.

It must again be emphasized that the selection of a system using the straight vacuum pumping process and located at the rocket test site is a recommended solution to a specific problem posed by the contract statement of work. A change in NASA slush hydrogen requirements (e.g., long-term use of slush in several different programs) or a break-through in the state-of-the-art in handling slush (e.g., a convincing demonstration that slush can be transported by road or rail) might lead to an entirely different recommendation.

7.7.1 Summary of the Selected System

The system for which a conceptual design was prepared is summarized in Table 6.

TABLE 6

SUMMARY OF SELECTED SYSTEM

PROCESS	Straight vacuum pumping batch process.
LOCATION	Nevada test site, adjacent to main liquid hydrogen storage.
PRODUCTION RATE	18 tons of 50% slush per day.
LIQUID HYDROGEN CONSUMPTION	21½ tons per day.
ANTICIPATED DUTY	24 hours per day, 18.3 days per month plus approximately 3 days for upgrading of stored slush.

SECTION 8

SPECIAL TECHNIQUES IN SLUSH HYDROGEN PRODUCTION

Having selected the process and the plant site it was necessary to consider several proposed variations or techniques within the basic process before the conceptual design criteria could be fully established.

8.1 CRUST BREAKING TECHNIQUES

In vacuum pumped processes, solid hydrogen first appears at the walls of the vessel and gradually bridges over the liquid surface until a solid crust is formed, covering the entire cross section of the container. This crust is soft in the center and sufficiently porous to permit the passage of hydrogen gas through it. To obtain slush, this crust must be broken into particles of only a few millimeters in length or width.

Two successful crust breaking techniques have been demonstrated. The first of these employed a stirrer which, by its mechanical operation, caused breakage to occur. In the Linde Division experiments, this stirrer was manipulated manually, and it was found that crust thicknesses of up to 8 cm. could be broken by this means. This same technique, as employed at Wright-Patterson AFB, used a rake-like stirrer rotating at a very low speed. The prongs of the rake were moved through the newly-formed crust, breaking it into particles which settled through the liquid. This work is reported in Technical Report AFAPL-TR-65-63.

The second technique was developed by the N.B.S. at Boulder and consists of a pressure modulation on the slush tank to cause crust breakage. This has been given the name "freeze-thaw" method and ample description has appeared in N.B.S. Report No. 8881.

It was convincingly demonstrated at Boulder that the "freeze-thaw" technique would produce slush with apparently good flow characteristics in a small glass dewar. From this work it appeared that the rate of heat leak into the periphery of the crust and also the ratio of pumping speed to exposed surface area may be critical parameters. Since the scale-up of these factors from a diameter of 6 inches to a diameter of 10 feet could not be predicted, "freeze-thaw" was not adopted as the primary crust breaking technique. However, an annulus was included in the process vessels at the liquid-vapor interface. This annulus, with external connections, can be used as a controlled heat source if required for the operation of either crust-breaking technique.

The conclusions reached in AFAPL-TR-65-63 indicate that an optimum design for the rake-like stirrer will be reached shortly but it has not been demonstrated that the slush made by this method has flow characteristics equal to that made by "freeze-thaw". Nevertheless, a mechanical rake modeled after the latest design described in that report has been included in this process vessel.

8.2 SLUSH AGING REQUIREMENTS

The appearance of newly formed slush has been shown to differ from that which has aged for several hours. New slush particles are very porous and irregular in both size and shape but, after standing, they become less porous, and more dense, more uniform in size and more regular in shape. Work done by N.B.S. has shown that nearly all the change occurs in the first four hours of aging and very little after that.

To be able to predict the flow characteristics of a solid-liquid mixture, it is necessary to have rather complete data on particle size and distribution. A large number of measurements have been made at N.B.S. on slush particles produced by the freeze-thaw method as a function of the age of the slush for the purpose of determining the effect of particle properties on flow characteristics. Although considerable data has been accumulated, its application to classical methods for determining pressure drop of solid-liquid mixtures in pipes remains unverified.

For lack of any information to the contrary, it is assumed that to successfully and reproducibly transfer slush from one vessel to another, the slush must be suitably aged and that this aging period amounts to four hours. This aging time, therefore, governs the minimum batch size in all batch production methods.

8.3 COLD VS. WARM VACUUM PUMPING

Vacuum pumping requirements are determined by the density of the hydrogen gas existing at the suction of the vacuum pump. If the vapor is cold, the volume of a particular mass of gas will be smaller than if the vapor is warm and a smaller vacuum pump can be used. In the former situation, the vacuum pump would have to operate at low temperatures. This would pose certain problems not encountered when operating warm. With the possibility that a vacuum pump operating at cryogenic temperatures might be procured, the actual volume of gas was determined for operation at both cryogenic and at near-ambient temperatures. Suction conditions and gas volumes are listed in Table 7.

TABLE 7

GAS VOLUMES AT VACUUM PUMP SUCTION

STRAIGHT VACUUM PUMPING

	Cryogenic	Ambient
Suction temperature, ^{o}K	20.3 - 13.8	273.2
Suction pressure, torr	760 - 52.8	760 - 52.8
Slush quality, %	50	50
Actual gas volume, cu.ft./lb. slush	13.539	259.99
Actual gas volume for 1500 lb/hr. slush, cfm.	338.48	6400

Suction temperature and pressure both decrease for the cryogenic pump as vacuum pumping proceeds, with the gas always at saturation conditions. For ambient-temperature operation, the gas is assumed to be preheated to a constant temperature of 273.2^{o}K at the pump inlet. In either case, no allowance has been made in this comparison for pressure drop between the slush production tank and the vacuum pump. The effect of this is to slightly underestimate the required vacuum pump capacity. The comparison shows that the pump operated at cryogenic suction conditions would require a capacity only 5.2% of one operating at ambient temperature.

A rather brief study of hydrogen vacuum-pumping methods and equipment was made to define the present state-of-the-art and to identify favorable areas for development effort. The results of this study, which are thought to be of general interest to the overall slush hydrogen program, are included as Appendix I of this report.

Discussions with several manufacturers of hydrogen compression equipment at the early stages of this contract yielded very little information or encouragement towards a cryogenic pump of the required capacity. Owing to the pressure of time, it was reluctantly agreed that the conceptual plant design must be based on an ambient pump. When design of this system was well advanced, a reply favoring a cryogenic pump was received from one major manufacturer and the situation was re-evaluated. As a result of discussions with this manufacturer, the conclusion was reached that a reciprocating compressor of fairly standard design could be made to operate at suction temperatures down to the triple point, with only a small development program.

With this new information, the decision was reached to re-design the system using this cryogenic pump but, since the design of the ambient system was well advanced, this work was also completed. Therefore, this report now offers two conceptual designs, the first using the cryogenic pump and the alternate warming the hydrogen to near ambient before entering the pump. It is felt that these two designs will form a useful basis on which to estimate the value of a future development program in the area of hydrogen compression equipment.

8.4 STEAM EJECTOR PUMPS

Instead of a mechanical compressor, a vacuum of about 50 torr can easily be obtained using a commercially available steam ejector.

A steam ejector has the advantage of simplicity of operation plus low maintenance and investment, but to produce 1500 lb. per hour of slush would require a two-stage non-condensing ejector and a steam consumption rate of approximately 25,000 lb. per hour. This is the thermal equivalent of 9,800 hp. compared with the 360 maximum hp. required by the vacuum pumps. Evaluated at $1 per 1000 lb. of steam and 1.00 cent per kw-hr. of electricity, the cost of steam exceeds that of electricity by $10,000 for each test firing.

It is conceivable that the low maintenance and investment might be attractive if large volumes of waste steam were available, but this is clearly not the case in the present situation. Consequently, no further effort was made in this direction.

8.5 UPGRADING OF SLUSH

Heat leak into storage vessels will result in the degradation of the quality of slush in storage. It is estimated that in a 280,000 gallon spherical tank, 24.3 lb. per hour of solid hydrogen will melt to triple point liquid because of heat inleakage. Upgrading capacity capable of producing, for each 280,000 gallon storage tank, 48.6 lb. per hour of 50% slush from triple point liquid must be provided, either as a separate upgrading facility or as increased capacity of the main slush producing facility.

Of the several means considered for slush upgrading, the one which seems by far superior consists of merely draining the excess liquid through a suitable strainer to prevent passage of the solid crystals, and returning this triple point liquid to the slush production tank for reconversion to slush. The quality of the residual slush in storage is established by the amount of liquid removed, and qualities greater than the maximum level fixed by transfer considerations are conceivable.

This concept has perhaps its greatest value when applied to "on-board" upgrading. Here it may be possible to achieve upgrading in the rocket tankage with a minimum of cost and added equipment.

If the transportation problem described earlier is solved and the slush production plant located at a point remote from the storage area (i.e., a commercial liquid hydrogen plant), the excess triple point liquid could be utilized in several ways.

One possibility, that of upgrading in a small auxiliary plant, has been discussed as Case 1 in paragraph 7.1.1. As a second possibility, the excess could be drained back into the transport vehicle and returned to the slush plant for reuse. This would help to keep the vehicle at triple point temperature and reduce losses on a subsequent delivery. Thirdly, the excess could merely be transferred to the conventional liquid storage tank at the test site, using the extra refrigeration to reduce boil-off losses in this tank.

SECTION 9

PRELIMINARY DESIGN CONCEPTS

For the purposes of this study the following assumptions were made:

1. A suitably graded and prepared site, approximately 180 ft. by 160 ft., will be made available, as close to the bulk liquid hydrogen storage as safety provisions will allow.

2. The battery limits of the plant will be established by a fence which will form a part of the plant.

3. All utilities, fluids, access roads etc. will be brought to the fence-line by others. Storage facilities for cryogenic fluids, hydrogen slush, gases etc. will not form a part of the plant.

4. A suitable building, housing a control room, primary maintenance area, office space and personnel facilities, will form a part of the plant.

9.1 DESIGN PHILOSOPHY

The design requirements of the plant appeared to be fairly simple with no real control problems, so that manual control of the operation seemed reasonable. However, in view of the environmental conditions, remote operation was considered desirable. Accordingly, it was decided that all primary controls should be remotely operated from inside a climate-controlled building but with no automatic sequencing.

All electrical equipment in the plant, except inside the building, would be Class I, Division I, Group B (or Group D with nitrogen purge where Group B was not readily available). The fence line would be established at least 50 ft. from the nearest hydrogen equipment.

Exposing the compression equipment to the extreme environmental conditions described in the "Criteria for Budgetary Study of Engine/Stage Test Stand 2-3" caused some concern but, after discussions with manufacturers, it was concluded that this was permissible. An all-outdoor plant was then adopted.

9.2 PLANT DESIGN CRITERIA

Using the information already developed, the system criteria presented in paragraph 7.7 were extended to produce some design criteria. These are given in the following table.

TABLE 8

PLANT DESIGN CRITERIA

PROCESS	Straight vacuum pumping batch process.
LOCATION	Nevada test site, adjacent to main liquid hydrogen storage.
PRODUCTION RATE	18 tons of 50% slush per day
LIQUID HYDROGEN CONSUMPTION	21-1/2 tons per day
ANTICIPATED DUTY	24 hours per day, 18.3 days per month plus approximately 3 days for upgrading of stored slush.
BATCH TIME	4 hours.
CRUST BREAKING	Mechanical rake with provision for "freeze-thaw".
VACUUM PUMPS	Either cryogenic (preferred) or ambient.
PROCESS CONTROL	Manual remote control from plant control room.
PROCESS VESSELS	(2) Approximately 11,000 gallon capacity (One on stream, one aging)

SECTION 10

CONCEPTUAL DESIGN OF PLANT WITH CRYOGENIC VACUUM PUMP

This section presents a summary of the conceptual design for the slush hydrogen plant utilizing a vacuum pump operating at the saturation temperature of liquid hydrogen.

10.1 PLANT OPERATION

Figure 12 is an operational flow sheet for this plant.

10.1.1 Slush Production

Liquid hydrogen, saturated at approximately one atmosphere, will be transferred into one of the duplicate processing vessels to the level of the upper level sensor, while the excess gas is vented from the vessel. After filling, the fill and vent valves will be closed, the vacuum valve opened and the vapor from the boiling liquid withdrawn through the vacuum pump.

Initially, the pump will withdraw approximately 4000 lb. per hour of hydrogen but, as the vessel pressure falls, the flow will gradually decrease. Meanwhile the liquid level will be maintained by gradual addition of liquid hydrogen.

The small stirrer will be used intermittently to prevent stratification in the vessel until the vapor space pressure reaches the triple point (52.8 torr). This will occur approximately 60 minutes into the cycle.

The liquid level controller will be turned off and the mechanical rake started as the formation of solid begins. The pumping rate for the next three hours will be constant at about 130 lb. per hour. As the solid is formed, it will be broken by the rake and will settle into the liquid. At intervals, the slush will be stirred and the percent solids read on the quality meter. About four hours into the cycle, at 50% solids, the vacuum pump will be switched to the alternate vessel and a new cycle begun.

The newly-made slush will remain in the vessel for about 3 hours of aging before being pressure transferred out of the plant to storage. The vessel will be re-filled with liquid for the next cycle.

10.1.2 Gas Disposal

The gas, in passing through vacuum pump, will undergo an adiabatic temperature rise but the discharge temperature will remain at less than 80^{o}R.

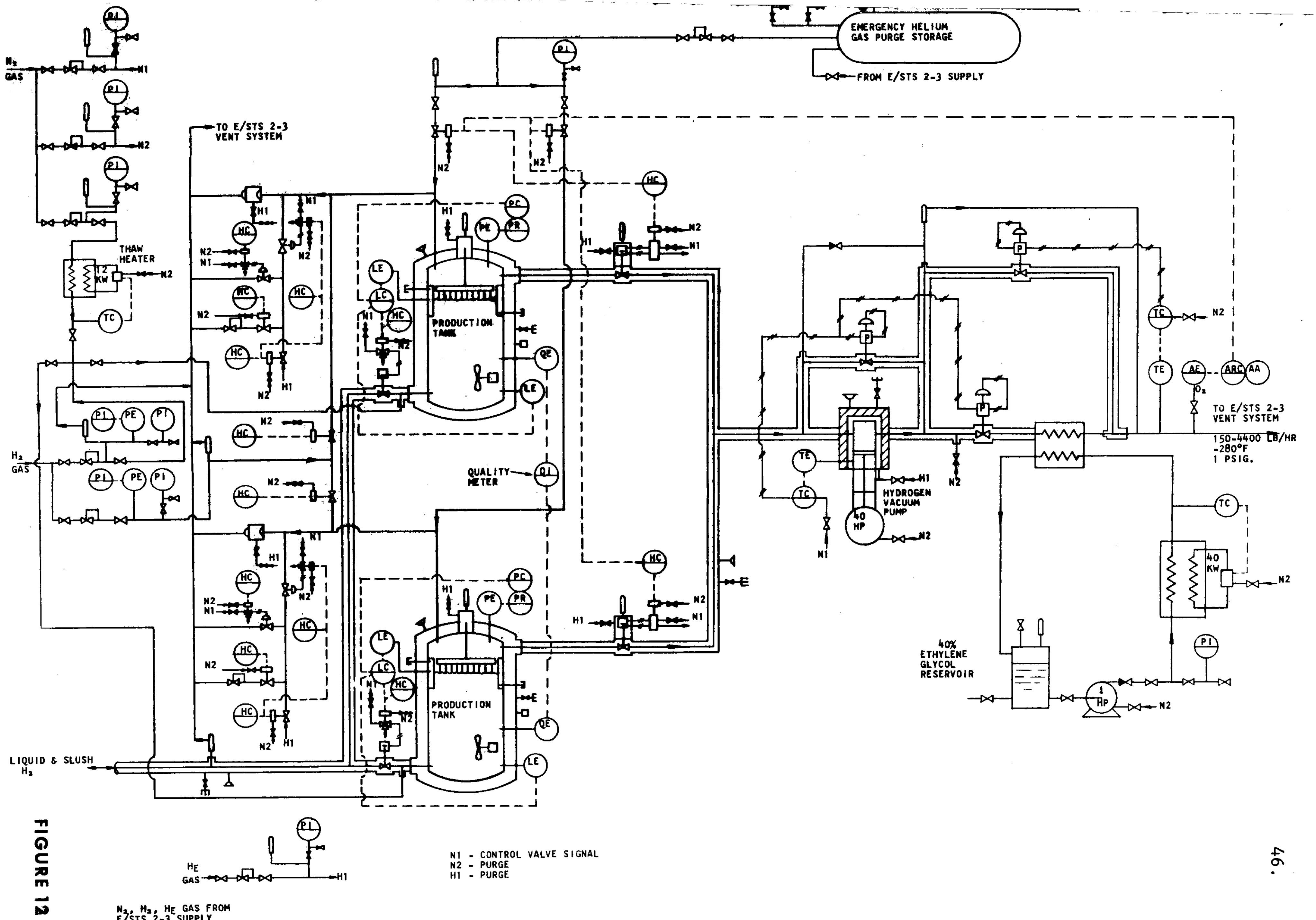


FIGURE 12

OPERATIONAL FLOW SHEET - PLANT WITH CRYOGENIC VACUUM PUMP

At this temperature and at one atmosphere pressure, hydrogen is more dense than ambient air and venting is hazardous; also liquid air will form on any uninsulated lines. To reduce these hazards the pump discharge will be warmed against recirculated ethylene glycol. The discharge, now at 180°R, will be taken from the plant to the main facility hydrogen disposal system.

10.2 PLANT LAYOUT

A plot plan is shown in Figure 13 and a layout of the equipment in Figure 14. The plant has two main sections, the processing vessels and the vacuum pump, which will be discussed separately.

10.2.1 Processing Vessels

As discussed earlier, there appears to be some doubt whether freshly made slush can be transferred as readily and reproducibly as slush which has been aged for some hours. In order to make provision for aging, a reasonably long batch processing time is required with duplicate vessels, one on stream and one aging.

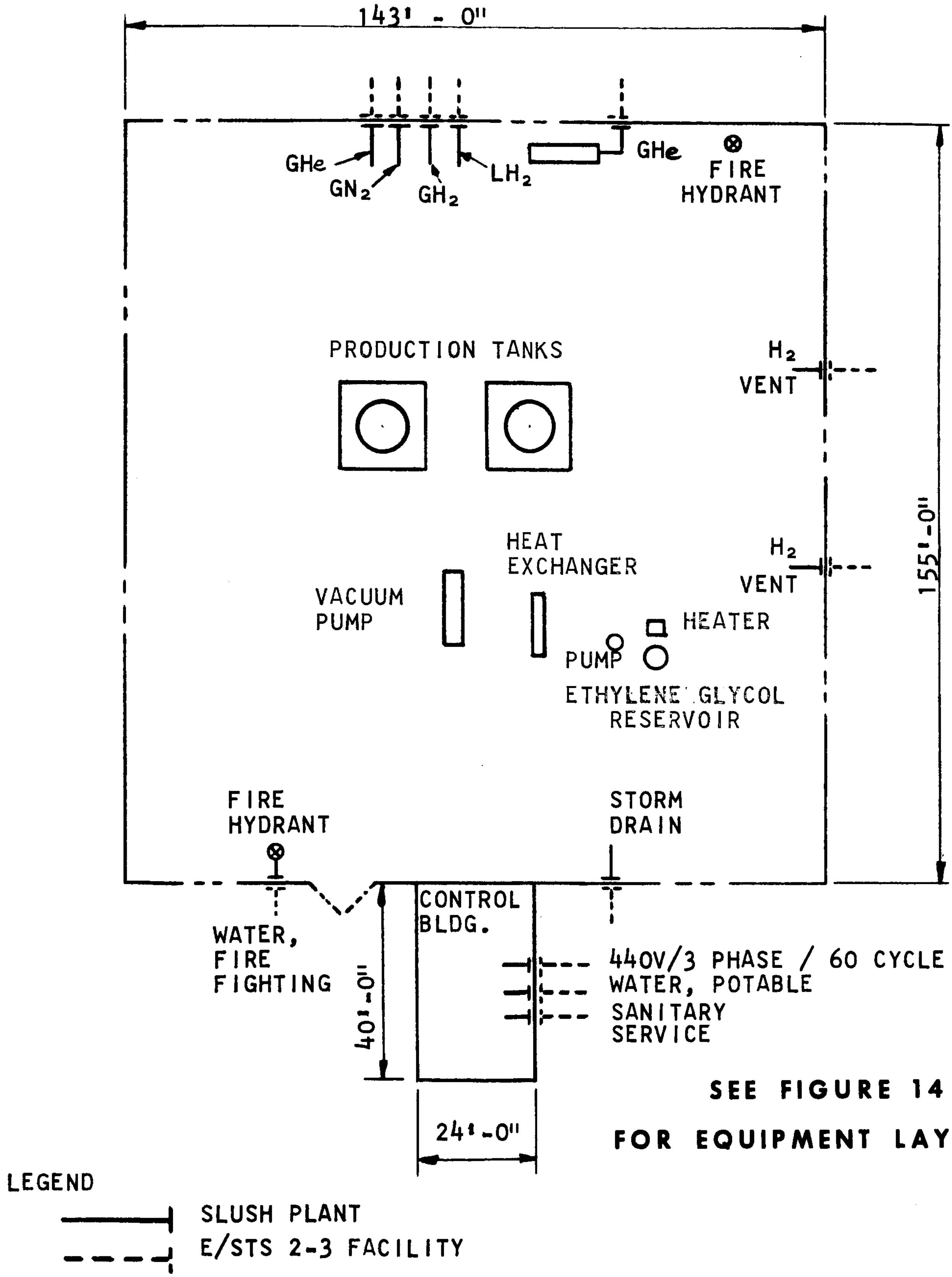
To give a reasonable aging time with a vessel size which could be shop fabricated, a batch time of four hours was selected, i.e., the process will produce a 6,000 lb. batch of slush every four hours. The following table lists the main design features of these vessels.

TABLE 9

PROCESSING VESSEL

PRELIMINARY SPECIFICATIONS

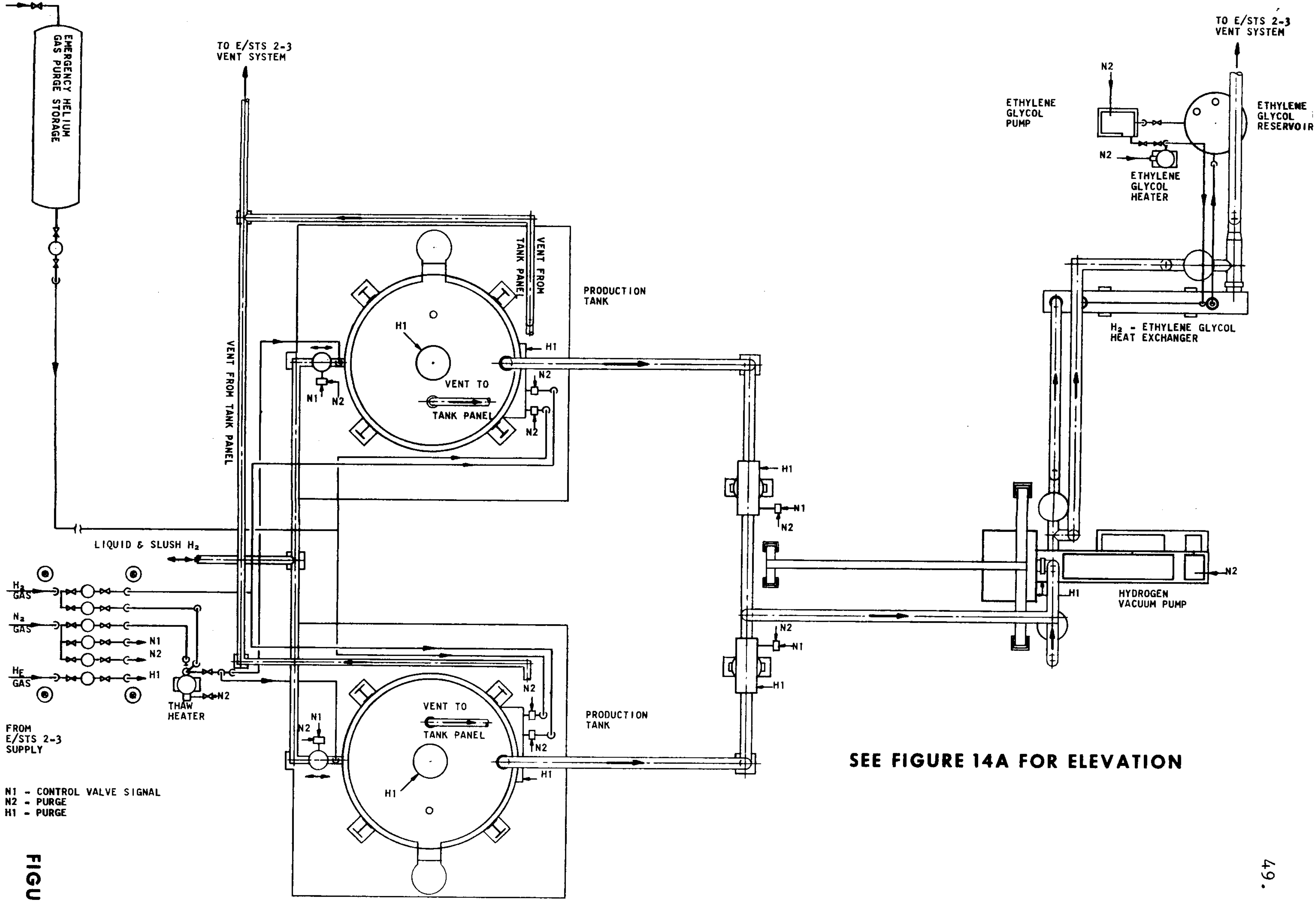
General Configuration	Vertical cylinder, vacuum insulated
Inner Vessel:	
Material	Stainless Steel
Dimensions	10 ft. diameter x 20 ft. 8 in. high
Working pressure	20 psig to full vacuum
Casing:	
Material	Carbon steel
Dimensions	11 ft. diameter x 29 ft. high



SEE FIGURE 14
FOR EQUIPMENT LAYOUT

FIGURE 13

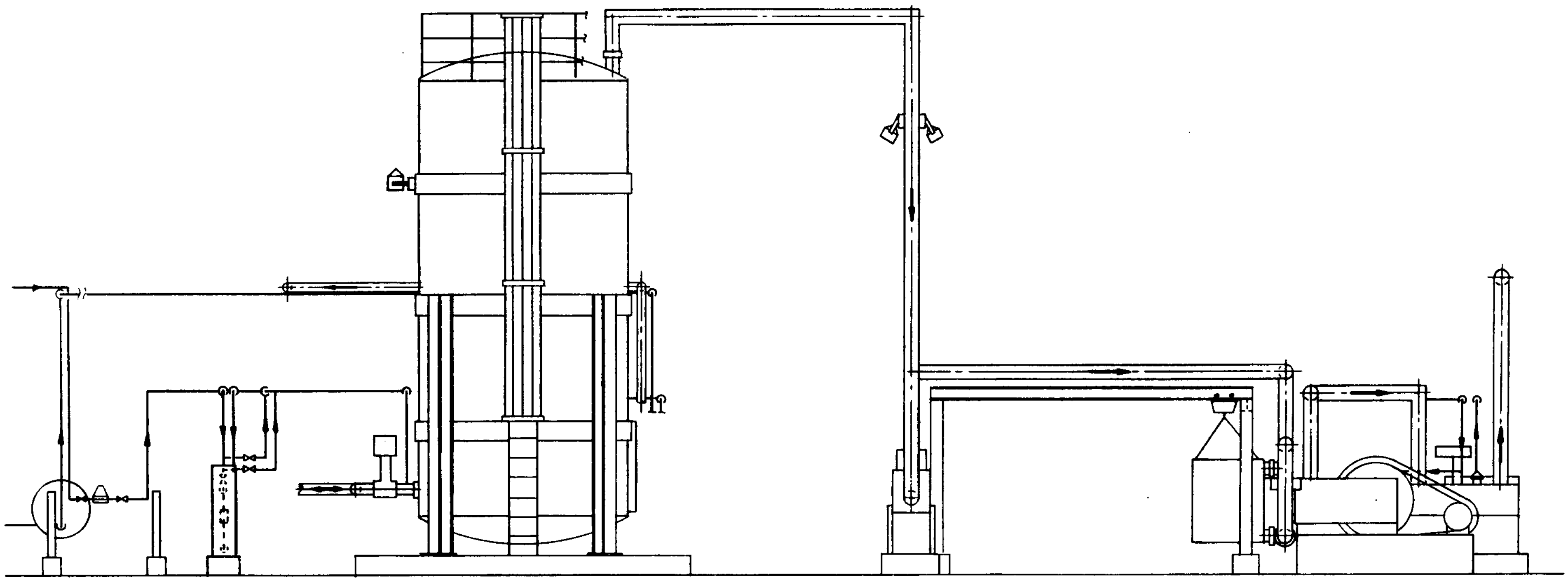
PLOT PLAN-PLANT WITH CRYOGENIC VACUUM PUMP



SEE FIGURE 14A FOR ELEVATION

FIGURE 14

EQUIPMENT LAYOUT - PLANT WITH CRYOGENIC VACUUM PUMP



EQUIPMENT LAYOUT - PLANT WITH CRYOGENIC VACUUM PUMP

FIGURE 14A

Heat Leak	Less than 350 BTU per hour
Fill-Drain Line	3 in. pipe, vacuum insulated
Vacuum line	6 in. pipe, vacuum insulated
Vent line	4 in. pipe
Safety Devices	(2) 4 in. burst disks with helium purge. Blowdown valve with helium purge. Emergency helium purge line.
Stirrer	Propeller with submerged 1/2 hp motor
Crust Breaker	Rake, driven by external 0 - 80 rpm motor drive, 100 in. lbs., helium purged.
Slush Quality Indicator	(4) Ikor quality sensors giving average or individual readings
Liquid Level Control	(2) Point sensors, capacitance type
Estimated Weights:	
Empty	37,500 lb.
Full	44,000 lb.

The liquid phase vacuum insulated line is connected via a control valve to the facility liquid and slush hydrogen storage tanks and also to nitrogen and hydrogen gaseous purge lines for pre-startup purging.

The vent line is connected to double bursting disks (in place of a safety valve), double blow-down valves and a back pressure valve. These are manifolded together and connected to the facility hydrogen disposal system. A helium purge is provided between the bursting disks and between the valves to reduce the risk of air in-leakage.

A separate purge system is provided to flood the vessel and piping with helium in an emergency.

The crust breaking rake is driven by a small motor-drive mounted in a helium purged housing on top of the vessel, with a small positive pressure of helium maintained around the shaft seals. In addition to the mechanical crust-breaker, an annular ring is provided inside the inner container at the slush/vapor interface, with entry and exist connections through the outer casing. These connections are for future use in the "freeze-thaw" technique.

The main vacuum line from the vapor phase of the vessel is vacuum jacketed to prevent air in-leakage and to prevent the formation of liquid air on the outside. Each vessel can be isolated by a control valve in the main line.

10.2.2 Vacuum Pump

Some brief process requirements were prepared in order to discuss the cryogenic pumps with various manufacturers. These are listed in Table 10.

TABLE 10

CRYOGENIC VACUUM PUMP

PROCESS REQUIREMENTS

Fluid Pumped	Cold gaseous hydrogen
Duty cycle:	240 minute cycle in 2 phases
Phase I	50 to 100 minutes
Phase II	Balance of 240 minutes
Suction Pressure:	
Phase I	Falling from 760 mm to 45 mm Hg
Phase II	Constant at 45 mm Hg.
Suction Temperature:	
Phase I	Falling from 36.5°R to 24.8°R
Phase II	Constant at 24.8°R
Required Throughput:	
Phase I	820 lb. total (in 50 to 100 minutes)
Phase II	380 lb. total (in 140 to 190 minutes)
Operation	Continuous, minimum of 30 four hour cycles between shut-downs.

It was soon recognized that experience in compression of gases below 140°R was restricted to a few expansion engines in liquid helium and hydrogen plants and one vacuum pump built for NASA in 1963. Several major manufacturers were contacted with discouraging results, and this approach was temporarily shelved in favor of a pump operating at ambient temperature. However, rather late in the study, one major manufacturer submitted an offer to design and build a suitable pump.

After reviewing this proposal, it was decided that the cryogenic concept should be re-activated and made the primary design. Table 11 is a summary of the principle features of this compressor. It should not be assumed that this machine has already been designed, but successful design and manufacture now seems entirely feasible.

TABLE 11

CRYOGENIC VACUUM PUMP

PRELIMINARY DESIGN

Type	Single-stage, horizontal non-lubricated reciprocating.
Size	22 in. bore x 9 in. stroke.
Construction:	
Cylinder, piston heads	Ductile iron
Piston rings	Teflon, carbon filled
Distance piece	Ductile iron, sealed, helium purged.
Motor	40 HP explosion proof, nitrogen purged.
Cooling water	None required
Insulation	Vacuum jacket enclosing pump cylinder
Operation:	
Speed	250 rpm
Time to complete Phase I	58 min.
Max. throughput	Approximately 65 lb. per min. (at 760 mm Hg suction)

Throughput in Phase II	Approximately 2.2 lb. per min. (at 45 mm Hg suction)
Discharge Pressure	760 mm Hg
Discharge Temperature	45° to 77°R.
Estimated Weight	6,000 lb.

To avoid excessive thermal stresses, a slow, controlled cool-down will be necessary. After purging with nitrogen and ambient hydrogen, the pump will be started with a by-pass control valve open. Thermocouples inside the pump cylinder will indicate the internal temperature and the bypass valve will be closed gradually to increase the mass flow through the pump.

The discharge from the pump will be divided, part of the flow going through a control valve and the tubes of a heat exchanger, and the balance going through a second control valve and by-passing the heat exchanger. The split will be controlled to maintain a temperature of 180°R downstream. The re-combined flow at 180°R and slightly above atmospheric pressure will be taken from the plant to the facility disposal system.

The shell side of the exchanger will be connected to a closed ethylene glycol loop containing a small pump, a 40 kw electric heater and a 350 gallon storage tank. As noted in Table 11, the flow through the vacuum pump will decrease from 65 lb. per min. to 2.2 lb. per min. in the first hour of each cycle and the heat load on the exchanger will undergo a similar 30:1 turndown. The glycol reservoir will serve as a heat sink to even out the demand on the electric heater over the four-hour cycle. This will permit the use of a small heater operating continuously rather than a very large heater operating intermittently.

SECTION 11

CONCEPTUAL DESIGN OF PLANT WITH AMBIENT VACUUM PUMP

As mentioned in paragraph 10.2.2, the cryogenic vacuum pump was, for a time, shelved. During this period, design work was concentrated on a plant in which the gas withdrawn from the process vessels would be heated to near ambient temperature before entering the pump. When the cold pump was re-instated, this "warm" design was well advanced and it was decided that it would be completed as an alternative.

The conceptual design presented in this section will serve as a "back-up" to the design in the preceding section in the event that a cryogenic pump is not forthcoming. It also gives a useful comparison of the equipment requirements and costs of the two concepts.

11.1 PLANT OPERATION

The operational flowsheet for this plant appears in Figure 15.

The slush-producing operation will be identical with that described in paragraph 10.1.1 up to the gas withdrawal from the process vessels. In the case now under consideration, the suction temperature at the pump is limited to a little below ambient (nominally $0^{\circ}F$) and a heat exchange section must be added between the vessels and the pump. This added section will be discussed in the next paragraph. The discharge from the pump, now heated above ambient, can be taken directly to the facility disposal system.

11.2 PLANT LAYOUT

Figure 16 shows a plot plan for this plant and Figure 17, the equipment layout. It may be seen that this plant requires an area some 20% larger than the previous design.

11.2.1 Process Vessels

These are identical with the vessels described in paragraph 10.2.1.

11.2.2 Heat Exchange Section

The hydrogen gas withdrawn from the process vessels will vary in pressure from 760 to 52 mm Hg, in temperature from 36.5 to $24.8^{\circ}R$ and in mass flow rate from 60 to 2.2 lb. per min. This flow must be warmed to the minimum acceptable temperature at the entrance to the vacuum pump. This was fixed for design purposes at $460^{\circ}R$, although $420^{\circ}R$ could probably be tolerated by most machines.

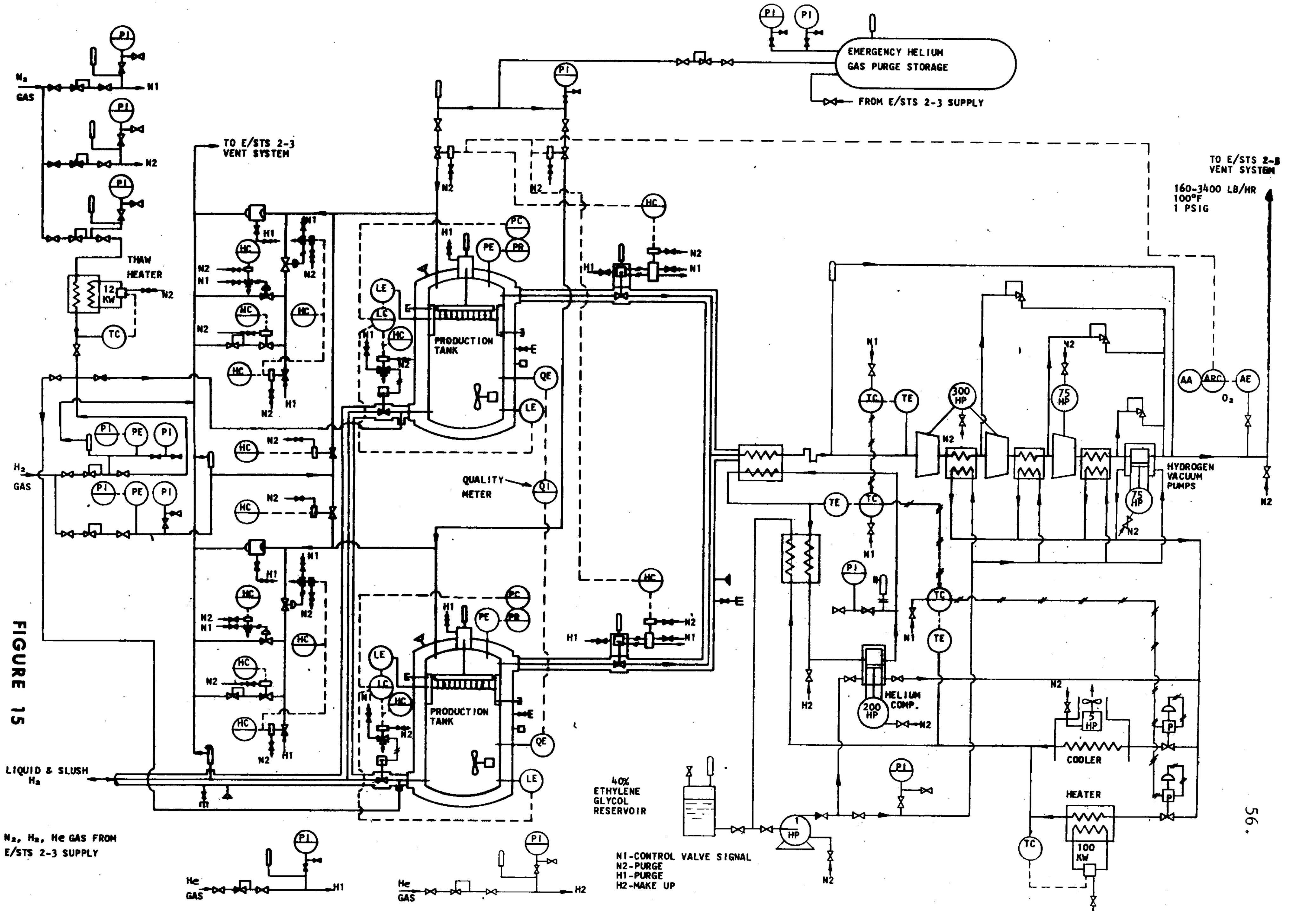


FIGURE 15

OPERATIONAL FLOW SHEET - PLANT WITH AMBIENT VACUUM PUMP

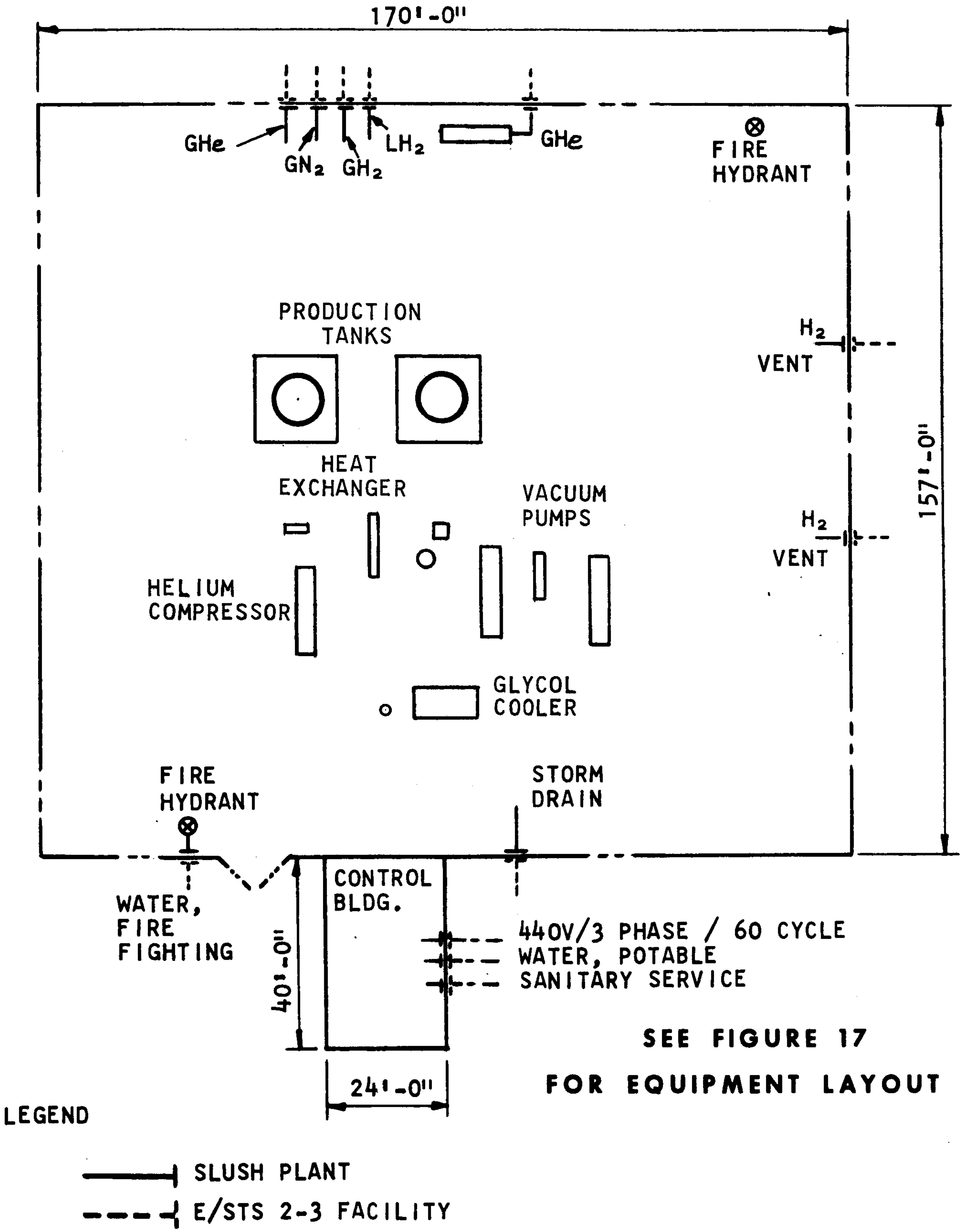


FIGURE 16

PLOT PLAN-PLANT WITH AMBIENT VACUUM PUMP

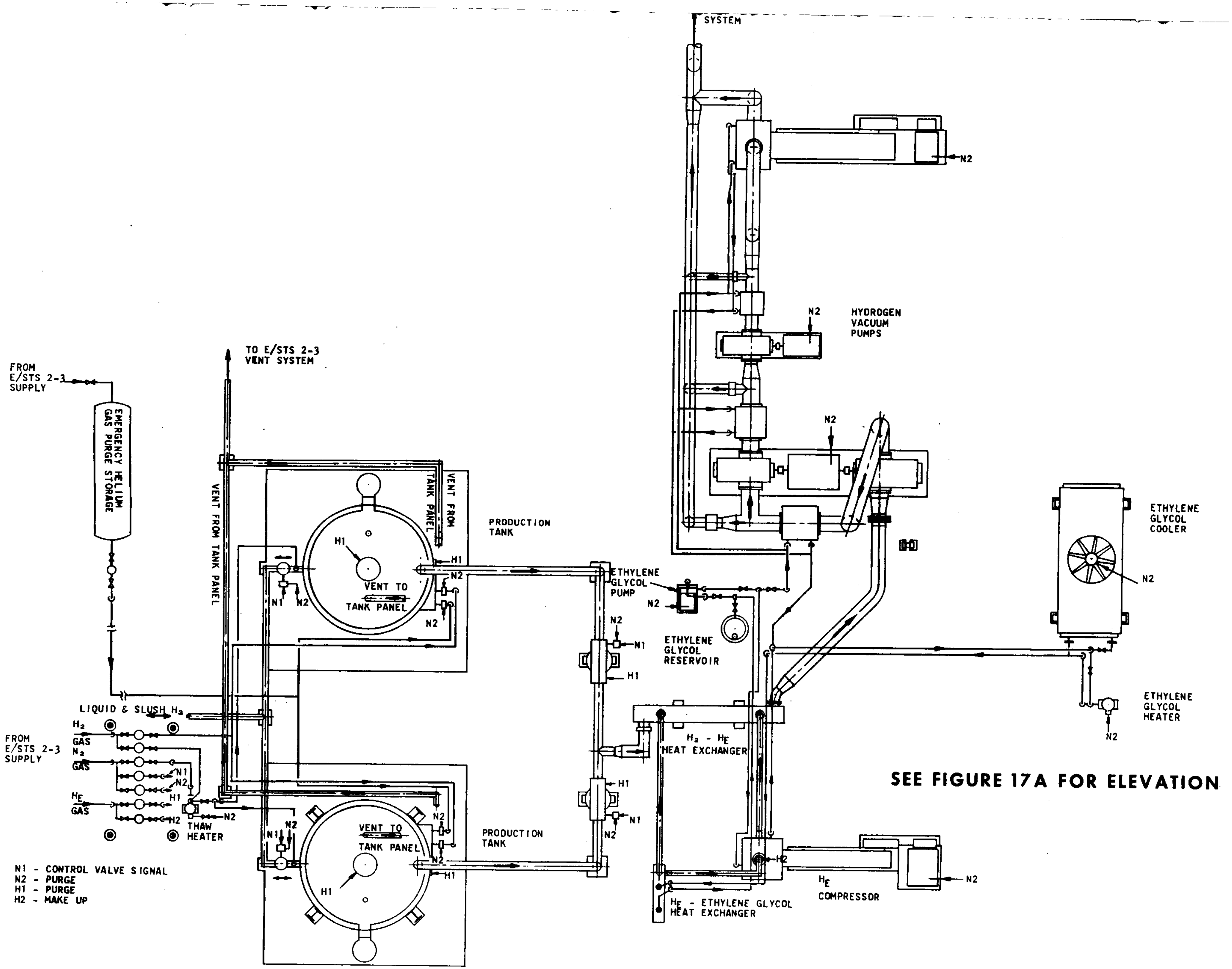
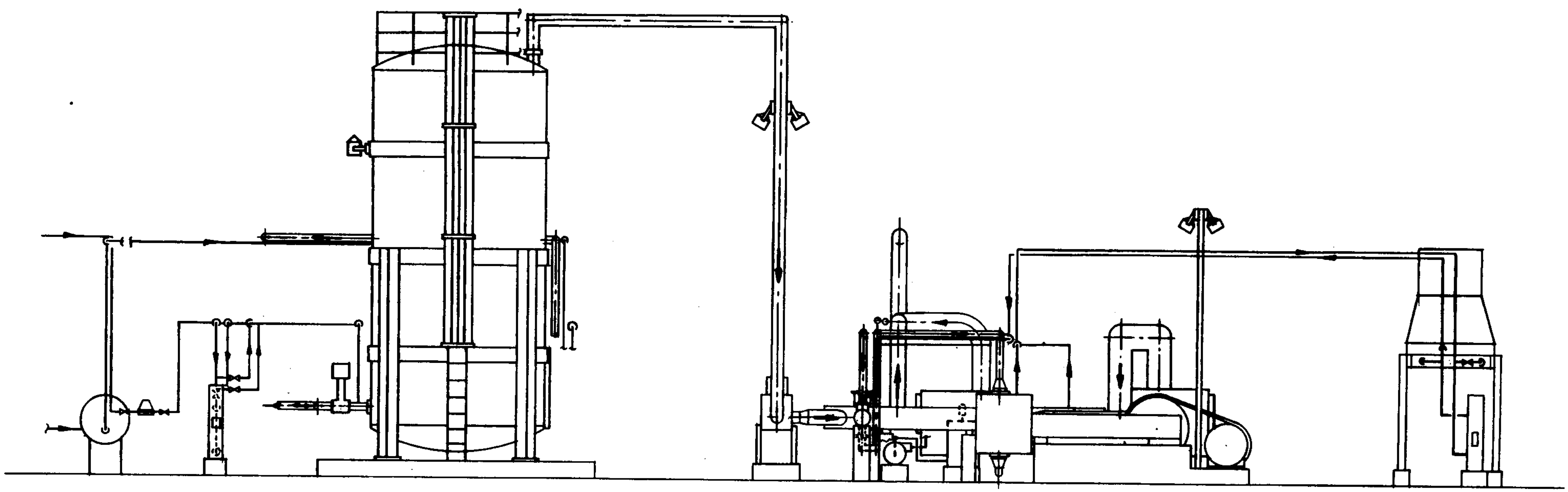


FIGURE 17

EQUIPMENT LAYOUT - PLANT WITH AMBIENT VACUUM PUMP



EQUIPMENT LAYOUT - PLANT WITH AMBIENT VACUUM PUMP

FIGURE 17A

The hazards and problems surrounding the heat exchanger for this service are self-evident. Any fluid leaking into the sub-atmospheric cold hydrogen stream (except helium or hydrogen) would freeze, causing at least partial plugging of the heat exchanger or worse if air was involved. It was decided that the conventional heat exchange media (water, air, ethylene glycol, etc.) could not be recommended for this service and a shell and tube exchanger with helium in the shell was adopted. All sections of the plant containing cold sub-atmospheric hydrogen would then be jacketed, either with vacuum or with helium. It was recognized that this approach might add 5 percent to the total cost of the plant but it seemed to be justified on the grounds of safety.

Over the four hour cycle, an average of 420,000 BTU per hour or about 125 kw, must be added to the hydrogen stream. A brief look at the vacuum pumps showed that, owing to the inefficiency of these machines, all this heat would be available in the pump coolant and could be used to heat the helium stream. A closed helium loop, transferring heat from the pump coolant to the hydrogen stream via two heat exchangers and a compressor was included in the design. Owing to the varying hydrogen flow there will be a deficiency of heat at the start of the cycle but an overall surplus. For this, a small electric heater and an air cooler were included in the pump coolant loop.

11.2.3 Vacuum Pump

The process requirements for this "warm" pump are the same as those listed in Table 10 for the cold pump except that the suction temperature will be constant at $0^{o}F$.

Some past experiments in hydrogen slush production have used oil-sealed rotary piston vacuum pumps. These are readily available and were satisfactory in laboratory - scale work. However, for the present larger application, they have two apparent disadvantages. First, a battery of nine of the largest commercial units in parallel would be required, and second, there is a problem of oil back-streaming from the pumps into the heat exchanger with consequent fouling or plugging of the unit. A partial solution to this could be a two-stage unit, a single dry lobe-type booster backed by five oil-sealed pumps in parallel. Another possibility was two ethylene glycol sealed lobe-type blowers in series. This, however, presented an even worse back-streaming problem and was not thought to be fully developed. All these alternatives required 325 to 360 HP and ranged in cost from \$62,500 to \$125,000 (installed).

It was decided that backstreaming could not be tolerated, even at the cost of a more complex compression system, and attention was focused on dry-sealed blowers. The system finally adopted will use four stages in series, three dry lobe-type blowers followed by one lubricated reciprocating compressor. Intercoolers will be provided between each stage with blow-off check valves after the first three stages. It is felt that the three dry stages, all operating continuously, will effectively prevent any oil back-streaming from the fourth stage. . This system will require one 300 HP motor and two 75 HP motors but the maximum total demand at any time will be approximately 350 HP. This pumping system is estimated to cost $105,000 installed. The estimated weight is 35,000 lb.

The discharge from this system, somewhat above ambient temperature and pressure, will be conducted directly to the main facility disposal area.

SECTION 12

SLUSH HYDROGEN STORAGE

In the preceding sections, it has been assumed that the slush produced in this plant will be stored in bulk storage tanks which will form a part of the main E/STS 2-3 facility. No attempt has been made under this contract to design these storage tanks, but in this section some of the features of slush storage will be outlined.

12.1 EFFECT OF HEAT LEAK

It should be noted that, as long as all three phases are present in a storage tank, any heat added to the tank will convert solid to triple point liquid at a rate of one pound of solid lost for every 25 BTU added. As an example, slush stored in a 500,000 gallon tank with a normal evaporation rate (NER) in liquid service of 0.06% per day (heat leak = 1500 BTU per hour) would deteriorate at 0.5% per day if the tank was full or 2% per day if the tank was only 25% full. Thus, there may be greater incentive to minimize heat leak in designing for slush storage than for conventional liquid hydrogen.

This same problem will, incidentally, arise in any future study of the transportation of slush, for example slush transported in a vehicle having a liquid NER of 0.5% per day will deteriorate at 4% per day.

12.2 UPGRADING OF SLUSH

Since some of the slush produced in this plant will be in storage for up to 30 days, some system of upgrading or reconstituting the stored slush will obviously be required. The first approach to this was to connect the gas phase of the storage tank to the plant vacuum pump and upgrade directly. This approach, however, is thought to be unnecessarily hazardous and is not recommended.

An alternate method which appears quite feasible would separate some of the triple point liquid from the solid in the tank and return it to the slush plant for re-processing. A small line with a screened entrance would be provided at the bottom of the storage tank to separate and return the liquid.

12.3 STORAGE AND TRANSFER

Storage of slush under hydrogen vapor at the triple point is considered unacceptable for safety reasons. N.B.S. Report 8881 discusses storage under either helium or hydrogen at a small positive pressure and concludes that either gas may be used. However, to fill a million-gallon storage tank with helium at 25°R and 1 atm. before starting slush production, would require approximately 3 million NTP cubic feet of gas. Although this helium could be recovered for re-use as the tank is filled, there is an obvious incentive to use hydrogen.

It appears that, once a stratified layer is formed at the interface, storage under hydrogen would be feasible. In fact, it can be theorized that, owing to hydrogen's lower thermal conductivity at low temperatures, it would be superior to helium as a pressurization gas.

As far as it is known, hydrogen slush has not yet been transferred by pumping and, until suitable pumps are available, only pressure transfer can be recommended. Hydrogen gas, either warm or cold, should be suitable for short-duration transfer of slush between tanks.

12.4 STORAGE AND RUN CONCEPT

The first tests at E/STS 2-3 will probably use conventional liquid hydrogen with slush being used in a later phase. Since it will obviously be desirable to use the liquid storage tank for slush storage, the peculiar requirements of slush must be recognized early in the design of the facility.

This was the subject of a brief study, based on the limited information now available. The concept presented in Appendix II is preliminary in scope and has not been optimized, but does present some of the requirements which should be met by the liquid storage design if slush is to be added later in the program.

SECTION 13

OPERATIONAL FEATURES

13.1 SAFETY CONSIDERATIONS

The slush hydrogen plant, as presently conceived, involves all the well-known hazards inherent in the handling and processing of liquid and gaseous hydrogen plus the additional problem of handling cold hydrogen gas, liquid and solid, at sub-atmospheric pressure.

Plant safety was, of course, considered continuously throughout the entire study. It is proposed here to highlight some of the main safety considerations in the conceptual design.

13.1.1 Plant Layout

To minimize the slush transfer problem the plant will be located as close as possible to the bulk hydrogen storage, the actual distance being governed by the quantity-distance criteria for the storage tanks. However, it was felt that the plant should be enclosed in a fenced area, and the boundary was rather arbitrarily set at 50 feet from the nearest hydrogen equipment within the plant.

It was assumed that the entire area, except the interior of the plant control building, will be classified Class I, Division 1, Group B under the National Electric Code. It must be recognized that some pieces of equipment, notably large motors, are not available in Group B. In these cases a nitrogen purge has been indicated on the operational flowsheet.

13.1.2 Hydrogen Under Vacuum

During operation, the processing vessels and main plant piping will contain hydrogen at sub-atmospheric pressure. There is an obvious risk that any leakage in the system will produce contamination of the hydrogen stream and, if the stream temperature at the point of leakage is below the freezing point of the foreign material, a buildup of solid contaminant may occur. In the case of most contaminants, this buildup may be no more than a nuisance, but if oxygen is involved, (e.g., in air) the hazard is real.

To reduce this risk all these sections of the plant are jacketed either with a higher vacuum or with helium so that the hydrogen stream is not exposed to the air. Relief valves are not used on the process vessels and a helium purge between double burst disks has been added. Valves are enclosed in helium pressurized jackets to prevent in-leakage of air through the packing where there may be a vacuum on both sides of the valve. In other locations a positive pressure of hydrogen is maintained above the valve seat or, in the case of the vessel blow-down line, double valves with helium purging is used.

Gaseous nitrogen and hydrogen purge connections are provided in the liquid hydrogen fill line to permit purging of the entire plant from the fill line through to the vent line before start-up.

13.1.3 Disposal of Waste Hydrogen

The flow rate of the hydrogen discharged from the vacuum pump will vary from about 3,600 to 130 lb. per hour. Consideration was given to either venting or flaring this gas within the slush plant but this was abandoned. Venting of the large flow was thought to be at best marginal in safety and flaring of the widely varying flow would be quite difficult. Consequently, the discharge will be connected directly into the main E/STS 2-3 disposal system.

In the event of a failure of the vacuum pump there is a possibility of back-streaming of air from the disposal system into the plant. An oxygen analyzer will be located in the discharge piping and connected to an alarm and an emergency purge system. Should this analyzer detect a hazardous concentration of oxygen, the process vessels, piping and compressor will automatically be purged with helium from a special emergency gas storage.

13.2 PLANT CONTROL

The vacuum pumping process is simple and no control problems are anticipated. It is estimated that either plant will require two operators on each shift plus one plant supervisor.

The only critical instrumentation is the slush quality meter. Development of a suitable instrument will be described in Section 15.

13.3 PLANT RELIABILITY

The use of rotating machinery inside the processing vessels presents some obvious problems. However, it is felt that careful design effort can result in reliable mechanical crust breakers and stirrers. The other major items of equipment, vacuum pump, compressor, etc. are of essentially standard design and should have commercial reliability. Approximately $10,000 has been allowed for spare parts in the cost estimates.

SECTION 14

COST AND SCHEDULE ESTIMATES

Three estimates have been prepared for each of the two conceptual designs, capital cost, construction schedule, and operating cost. These are presented below.

14.1 CAPITAL COST ESTIMATE

Before discussing the "capital cost" of this plant it is essential that the term be defined. The figures presented here are estimates of the price which NASA should expect to pay in order to have the plant designed, fabricated, installed and checked out ready for start-up, using existing commercial standards for liquid hydrogen plants.

Since the plant is to be at a NASA facility, there may be standards applied which would exceed normal commercial standards but additional cost for these has not been estimated. Some additional cost could, most likely, be encountered in meeting rigorous cleaning specifications.

Numerous problem areas have been indicated from time to time in this report. The capital cost estimates do not include any development money for solving the basic problems such as optimum design of the crust-breaker, scale-up of "freeze-thaw" technique, etc. It is assumed that the state-of-the-art will have advanced to the point where only normal engineering design effort will be required.

14.1.1 Capital Cost - Plant with Cryogenic Pump

The following table lists the cost of the plant described in Section 10.

TABLE 12

CAPITAL COST - PLANT with CRYOGENIC PUMP

Yardwork, buildings and foundations		$ 47,600
Installed equipment:		
Production Vessels	$226,000	
Vacuum Pump	48,300	
Instrumentation	62,500	
Other	89,400	
		426,200
Installed piping:		
Vacuum insulated	33,100	
Other	38,100	
		71,200
Electrical equipment and wiring		33,100
Spare parts		10,600
Engineering		141,000
Field supervision and job set-up		37,000
		$766,700

14.1.2 Capital Cost - Plant with Ambient Pump

Table 13 gives the estimated cost of the plant described in Section 11.

TABLE 13

CAPITAL COST - PLANT with AMBIENT PUMP

Yardwork, buildings and foundations		$ 51,100
Installed equipment:		
Production vessels	$226,000	
Vacuum pump	105,000	
Instrumentation	62,500	
Other	119,800	
		513,300
Installed piping:		
Vacuum insulated	21,800	
Other	53,200	
		75,000
Electrical equipment and wiring		56,300
Spare parts		10,600
Engineering		141,000
Field supervision and job set-up		37,000
		$884,300

14.2 CONSTRUCTION SCHEDULE

It is estimated that, after the necessary development work is completed, either of the two alternate plants could be designed, fabricated, installed and checked-out, ready for start-up in 20 months from the award of a contract. Again, this estimate is based on existing commercial standards; application of more stringent specifications might well extend the schedule. A list of the major milestones in the schedule follows:

TABLE 14

CONSTRUCTION SCHEDULE MILESTONES

Project authorized	D + 0 months
Process design completed	D + 2 months
Equipment purchase orders written	D + 5½ months
Equipment delivered to site	D + 16 months
Installation complete	D + 18 months
System checkout complete	D + 20 months

14.3 OPERATING COST ESTIMATES

The following tables contain estimates of the direct operating costs of the two plants on a monthly basis.

TABLE 15

OPERATING COST ESTIMATE

PLANT with CRYOGENIC PUMP

Labor, 2 men per shift	$2,520
Supervision	800
	3,320
Overhead, 55%	1,830
	5,150
Nitrogen and helium	1,000
Power, 40,000 Kw hr.	400
	$6,550

TABLE 16

OPERATING COST ESTIMATE

PLANT with AMBIENT PUMP

Labor, 2 men per shift	$2,520
Supervision	800
	3,320
Overhead, 55%	1,830
	5,150
Nitrogen and helium	1,200
Power, 260,000 Kw hr.	2,600
	$8,950

The total productive life of this plant is not known, but is assumed to be relatively short; hence no estimate of maintenance cost has been made. Also, the unit cost of liquid hydrogen is not known and has been omitted. The estimated consumption of liquid is 800,000 lb. per month to produce 660,000 lb. of slush.

SECTION 15

REVIEW OF PROBLEM AREAS AND DEVELOPMENT PROGRAMS

It could readily be seen throughout this study that the requirements of the conceptual design were running ahead of the present state-of-the-art in slush hydrogen production and handling. In this concluding section the areas of doubt will be reviewed, with some recommendations for development programs to close the obvious technological gaps which now exist.

The development work which is in process at NBS and Wright-Patterson A.F. Base is yielding much valuable information from laboratory-scale equipment. However, it is unlikely that a confident scale-up from the laboratory to tonnage production will be possible without experience at an intermediate size. It is therefore recommended that a pilot plant be built and operated before the tonnage facility is designed. This plant would be used to provide information on production techniques and would also provide the product slush for other test and development programs. A capacity of about 5,000 lb. of slush per day is suggested for this pilot plant. This would produce a road trailer of slush in one to two days and a tank car in three to four days and would be of adequate size to provide scale-up data for the larger plant.

The following paragraphs outline some of the short-range and long-range programs porposed for this pilot plant.

15.1 IMMEDIATE DEVELOPMENT PROGRAMS

The following is a summary of the development programs which will be required before confident design can be made of a system to satisfy the immediate needs of the Nuclear Rocket Test Program. Programs to extend the use of slush beyond the immediate objectives will be described in a later paragraph.

15.1.1 Slush Transport Characteristics

The future use of slush on any reasonably large scale depends almost entirely on the successful development of methods of transferring slush through pipelines. The only controlled experiments on any practical scale which have been reported to date are those at N.B.S. The success of these preliminary tests has caused some justified optimism but definitive answers are urgently needed.

The objectives of this program are four in number:

1. Determine the maximum quality slush which can be reliably and reproducibly transferred between storage tanks in a simple pipeline system.

2. Determine the design parameters for such a system, using hydrogen gas pressurization for the transfers. This work will require the collection of data on the viscosity of slush of varying quality.

3. Develop techniques for the long-term bulk storage of slush at atmospheric pressure.

4. Develop methods for upgrading or reconstituting slush in storage.

15.1.2 Vacuum Process Scale-Up

The true hydrogen slush, sometimes called "fluid hydrogen slush", appears to be a fairly homogeneous mixture of triple-point liquid and more-or-less uniform solid particles, about 3 mm in diameter. There have been reports of successful production of this material in laboratory work and also some unpublished reports of failures in larger equipment. From this it can be deduced that the use of wrong scale-up factors can produce liquid-solid mixtures which are not true slush.

The pilot plant would be used to evaluate the various production techniques discussed in Section 8 at a reasonably large scale.

15.1.3 Cryogenic Vacuum Pump

As discussed in paragraph 10.2.2, a reciprocating pump operating at liquid hydrogen temperature now seems feasible but this machine has not been designed. The pilot plant mentioned above would, of course, provide an opportunity to develop a reliable cryogenic pump, with significant benefits to the cryogenic industry.

15.1.4 Instrumentation

Optimum operation of the batch process requires that the cycle be terminated when the slush quality is just below the maximum dictated by transport characteristics. Thus, an indication of average percent solid in the vessel is almost essential.

In response to Linde's request, Ikor, Inc. of Burlington, Massachusetts submitted a technical proposal for a densitometer for this service, with a budgetary cost estimate of $24,000 for 8 sensors (4 in each vessel) and one converter and display. The proposed instrument uses a matrix type of capacitance transducer whose output is said to be independent of the geometry of phase distribution. Although it was felt that use of this instrument in liquid-solid mixtures is not yet fully proven, it was included in the conceptual design and cost estimate.

There appears to be an immediate need for improved instrumentation for slush production and transfer. The order of priority for a development program should be:

1. Quality measurement in static vessels.
2. Remote pressure indication in the 50 torr range.
3. Rate of flow of slush in pipelines.

15.2 LONG RANGE DEVELOPMENT PROGRAMS

Most of the development efforts mentioned so far in this section would be directed toward a prompt and sure solution to the immediate problem at E/STS 2-3. However, if slush is to reach a state of development and use comparable with liquid hydrogen, some more fundamental work will be necessary.

The programs discussed below go beyond the scope of the present program and would lead to the production and distribution of slush on a wide scale.

15.2.1 Distribution of Slush

The program outlined in paragraph 15.1.1 is directed only toward the minimum objective of transferring slush between vessels by pressurization. Obviously, if slush is to be used in a widespread program of testing and actual use in nuclear and chemical rocket engines, a second development program of much broader scope must be undertaken when slush is available in adequate quantities.

The objectives of this program would be:

1. Develop suitable pumps for the transfer of slush in the range of flows and pressures now covered by liquid hydrogen transfer pumps.

2. Develop transport vehicles (road trailers and/or rail tank cars) for the long-distance transportation of slush. The possibility exists, of course, that existing vehicles can be used unchanged or with modifications but this must be demonstrated by actual test.

15.2.2 "On-board" Use of Slush

Very little is known of the behavior of slush when subjected to flight conditions, particularly the effect of high and low g-fields. There is an obvious need for exploratory studies in this area early in the overall slush development program.

15.2.3 Commercial Production of Slush

The batch vacuum process, while recommended for the test program for which this study was made, has obvious limitations for long-term commercial operations. As indicated in Section 7, the helium refrigeration process may have definite advantages if the initial investment is to be amortized over a reasonably long period, but there has been virtually no practical experience with this process. The most obvious problem will be the lack of information on the production of cryogenic solids on a heat transfer surface. If suitable equipment can be developed it is at least theoretically possible to operate this process entirely in the liquid-solid phase, without the disadvantages of operation at sub-atmospheric pressure.

It is felt that the final objective of a slush development program would be centrally located plant or plants, operating on a continuous process, and supplying slush by road or rail to remote use points.

APPENDIX I

HYDROGEN COMPRESSION EQUIPMENT

The present study of hydrogen slush production techniques proved the validity of an earlier finding (Reference 1) that the vacuum pumping method offered technically and economically the most straightforward approach for earth based slush productions in the quantities as specified by the present contract.

Compared to the other examined methods, the vacuum pumping scheme is proven and is characterized perhaps by the simplest cycle of all, resulting in low investment cost and highest reliability.

The reliability which is of great importance will be mostly influenced by the only equipment which may contain moving parts (with the exception of valves) i.e., the compression equipment. It is of importance, therefore, to review the presently available compression (vacuum-pumping) methods and equipment in order to select the best candidate for immediate use and to identify problem areas where development work may effect a favorable return.

A. Positive Displacement Compressors

The most straightforward approach is, of course, to employ commercially available, positive displacement vacuum pumps compressing the hydrogen gas from triple point pressure to ambient, discharging the hydrogen -- after reasonable safety precautions are taken (for example, burning or sufficient dilution) -- to the atmosphere. Commercially available positive displacement compressors, however, could not accept the hydrogen at triple point temperature for several reasons, and therefore a suitable heat source and heat exchanger equipment would be needed to warm up the hydrogen gas to an acceptable inlet temperature level.

This, of course, is undesirable from the point of view of energy consumption. The horsepower needed to compress w lb/sec. ideal gas isotropically can be expressed by the following formula:

$$HP = \frac{w}{550} \frac{k}{k-1} \frac{1545}{M} T_{inlet} \left[\left(\frac{P_{out}}{P_{in}} \right)^{\frac{k-1}{1}} - 1 \right]$$

It can be seen that the power requirement is directly proportional to the inlet temperature in case of a single stage machine. This consideration is important, not only because of the operating cost but also because the investment cost of motor-driven reciprocating compressors and vacuum pumps is (nearly) proportional to the horsepower input. Therefore, there is a definite incentive to utilize compressors which are capable of compressing low temperature gas, preferably without any warming up. It is believed that suitably designed reciprocating machines could fulfill this condition. After all, helium reciprocating engines were successfully developed working in the 10-20^{o}K temperature range. The required development work is believed to be modest. Material selection, lubrication, thermal insulation, and suitable valve design would be the development targets.

B. Ejectors

High reliability is the strongest claim which favors the ejector due to the lack of moving parts. Unfortunately, this device is known to have a notoriously low efficiency. Indeed, the cost penalty is so high using ejectors with (nearly ambient) hydrogen inlet temperature that, unless special consideration favors them, these devices should not be contemplated. Special consideration may include availability of medium pressure steam (approximately 100 psia) and infrequent operation.

The situation would be considerably improved if an ejector could be developed which would be able to work with triple point temperature hydrogen gas. Investigation of literature of basic (References 2 and 3) and of applied nature (Reference 4) revealed no basic obstacle to developing such a device. The required development work is believed to be relatively small. It would be mostly analytical with few key experiments performed.

C. Dynamic Compressors

Centrifugal compressors are favored by high reliability, high efficiency, and, above a certain horsepower range, by lower prices in comparison with the positive displacement devices.

It was therefore examined to determine whether the contemplated flow rate (270 lb/hr.* hydrogen gas) in the required compression ratio would result in an acceptable number of stages, impeller diameter and operating speeds.

After some exploratory calculations, it was established that the number of stages would be four. The next step was to calculate the required operating speed and impeller diameter resulting in reasonable efficiency. It is known that the efficiency of turbo compressors is (mainly) a function of the so-called specific speed:

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* NOTE: These calculations were made before the 1,500 lb. per hour production rate was established.

$$N_S = \frac{N\sqrt{v_{inlet}}}{H_{ad}^{3/4}}$$

where N = speed rpm

v_{inlet} = ft. inlet flow - $ft^3/sec.$

H_{ad} = adiabatic head $\frac{ft\ lb_f}{lb_m}$

and specific diameter

$$D_S = \frac{D\ H_{ad}^{1/4}}{\sqrt{v_{inlet}}}$$

where D = impeller diameter - ft.

For a given set of operating conditions, that is, volumetric inlet flow, adiabatic head, there is a unique value of specific speed and specific diameter where the efficiency is the highest.

It is customary to divide the total compression ratio equally between stages for an application where intercooling is possible. As can be seen in the following, this method is unattractive for the slush hydrogen production application due to the fact that the successive stages would require higher and higher operating speeds and smaller and smaller impel diameters. In the examined case, a specific speed of 60 was selected in order to keep the rotational speed low. The reduced diameter is 2.4, which will assure an efficiency of about 70 per cent with a suitable aerodynamic design. (See Reference 5).

The situation is similar if the compression ratio is progressively reduced at higher stages. The following tabulation shows the result of the described approaches.

CENTRIFUGAL COMPRESSOR

STAGE	COMPR.RATIO	HEAD $\frac{ft\ lb_f}{lb_m}$	T_{inlet} ^{o}R	INLET VOLUME $ft^3/sec.$	SPEED RPM	IMP. DIA. inch	N_s	D_s
Equal compression ratio. Constant N_s and D_s flow rate 270 lb/hr.								
1	1.948	1.4×10^4	24.8	9.75	24,700	8.75	60	2.4
2	1.948	1.88×10^4	32.8	6.5	37,700	6.27	60	2.4
3	1.948	2.35×10^4	41.8	4.37	54,400	4.86	60	2.4
4	1.948	3.06×10^4	54.5	2.92	66,500	4.55	60	2.4
Progressively reducing compression ratio. Constant N_s and D_s flow rate 270 lb/hr.								
1	2.55	2.46×10^4	24.8	9.75	37,800	7.16	60	2.4
2	2.02	2.26×10^4	37.9	5.89	45,500	5.7	60	2.4
3	1.76	2.34×10^4	49.8	3.83	57,800	4.56	60	2.4
4	1.6	2.4×10^4	62.2	2.72	70,000	3.82	60	2.4

The inlet temperature was calculated according to the following formula.

$$T_{n1} = T_{(n-1)1} \frac{P_2/P_1^{\frac{k-1}{k}} - 1}{\eta(n-1)}$$

where T_{n1} = Stage inlet temperature - ^oR

$T_{(n-1)1}$ = Inlet temperature of the preceding stage - ^oR

(P_2/P_1) = Compression ratio

$\eta(n-1)$ = Efficiency of the preceding stage

After examining several possible compression distributions, it was found that the most practical case is where the compression ratio and the operating speed are constant. In that case, the specific speed and the specific diameter will be changing from stage to stage. The following tabulation shows the unoptimized result of this approach. The flow rate of the compressor was increased to 500 lb/hr. in order to bring the operating speed to a commercially accepted level.

STAGE	COMPR. RATIO	HEAD $\frac{ft\text{-}lb_f}{lb_m}$	T_{inlet} ^oR	INLET VOLUME $ft^3/sec.$	SPEED RPM	IMP.DIA. inch	N_s	D_s
Equal compression ratio. Constant RPM. Flow Rate 500 lb/hr.								
1	1.948	1.4×10^4	24.8	17.8	35,000	6.75	114	1.45
2	1.948	1.88×10^4	37.9	11.9	35,000	6.95	75	2
3	1.948	2.35×10^4	49.8	8	35,000	6.26	52	2.5
4	1.948	3.06×10^4	62.2	5.35	35,000	7.5	35	4

It is believed that with some further work an optimum design could be arrived at having constant operating speed and impeller diameter.

This compressor could be developed with a relatively small effort. Most of the work would have to be concentrated on mechanical development. The spinoff of this development to industry might be significant.

Since the efficiency of the cold compression equipment is of secondary importance, the relative merit of the so-called regenerative or peripheral compressor was also examined. This type of compression equipment is known to have low speed requirements. The following tabulation, which is based on equal compression ratio, was worked out by using design data and charts as per References 6 and 7.

REGENERATIVE COMPRESSOR

STAGE	COMPR. RATIO	HEAD $\frac{ft\text{-}lb_f}{lb_m}$	T_{inlet} ^oR	INLET VOLUME $ft^3/sec.$	SPEED RPM	IMP.DIA. inch	N_s	D_s
Equal compression ratio. Constant RPM. Flow rate 270 lb/hr.								
1	1.948	1.4×10^4	24.8	9.75	13,000	9.8	30	2.8
2	1.948	2.24×10^4	38.4	7.75	13,000	9.8	18.8	3.6
3	1.948	3.58×10^4	60	6.28	13,000	11	12.3	5
4	1.948	5.38×10^4	96	5.16	13,000	10.7	8.35	6

D. Summary

The findings of the compression equipment study are summarized in the following tabulation.

TYPE OF COMPRESSOR	HP (Normalized)	No. of Stages	Does It Require Development Work	Remarks
Warm, positive displacement	1	1 or 2	No	
Warm, ejector	18.75	2 or 3	No	
Cold, positive displacement	0.047	1	Yes	Small amount of development work only.
Cold, ejector η = .15 ounce	0.236	1 or 2	Yes	Promising. Basic analytical and experimental work is indicated. Disadvantages: Fluid is contaminated. Effluent is diluted.
Cold, centrifugal	0.0885	4	Yes	For flow quantities larger than 500 lb/hr H_2 pumped. Fallout for industry is expected. Medium size effort mostly on the field of mechanical development.
Cold, regenerative	0.183	3 or 4	Yes	For flow quantities larger than 100 lb/hr

Positive displacement compressors are readily available for the specified operating conditions. Disadvantage is a large horsepower requirement and the additional heat exchange equipment to warm up the gas from triple point temperature to (nearly) ambient temperature.

A small amount of development work will be required only to design a cold, positive displacement compressor. It is believed that cooperation between a commercial reciprocating compressor manufacturer and a cryogenic firm would bring the best and quickest results. These two possibilities should be considered for the present application.

Should the slush requirement be considerably higher, investigation and development of other vacuum pumping schemes should be made. Candidates in this category are the cold ejector, cold centrifugal, and cold regenerative compressor. While the cold ejector might be the most practical answer for quantities somewhat larger than the present, for very large-scale slush hydrogen production the cold centrifugal and regenerative compressor could undoubtedly assure higher efficiencies.

It should also be noted that, while the ejector is basically an open cycle device, the centrifugal and regenerative compressors could work in a closed cycle; that is, the boiloff hydrogen gas would not be wasted but it could be recycled. Although this may be far in the future, these devices could be utilized for extraterrestrial-based hydrogen slush production. In this case, an overall system weight reduction would also result with dynamic compressor.

REFERENCES - APPENDIX I

The following references apply to Appendix I:

1. R. R. Carney, R. F. Dwyer, H. M. Long and L. R. Niendorf, Theoretical, Experimental and Analytical Examination of Subcooled and Solid Hydrogen. Technical Documentary Report APL-TDR-64-22. AF RTD Wright-Patterson Air Force Base, Ohio, Project No. 8169, Task No. 816901.

2. J. H. Keenan and E. P. Naumann, A Single Air Ejector, Journal of Applied Mechanics, June 1942.

3. J. H. Keenan, E. P. Neumann and F. Lusterwerk, An Investigation of Ejector Design by Analysis and Experiments, Journal of Applied Mechanics, September 1950.

4. R. Frumerman, Steam Jet Ejector, Chemical Engineering, June 1956.

5. O. E. Baljè, A Study on Design Criteria and Matching of Turbomachines, Journal of Engineering for Power, January 1962.

6. P. S. Cates, Performance Characteristics of a Peripheral Compressor Rotating Machinery for Gas-Cooled Reactor Application, AEC TID-7690, November 1963.

7. Liquid Helium, Closed-Cycle Cryogenic Refrigerator Development, First Interim Report, General Electric Company, Contract No. AF(615)-1048.

$$HP = \frac{w}{550} \quad \frac{k}{k-1} \quad \frac{1545}{M} \quad T_{inlet} \left[\left(\frac{P_{out}}{P_{in}} \right)^{\frac{k-1}{1}} -1 \right] \qquad \text{Eq. 1}$$

$$N_s = \frac{N\sqrt{v_{inlet}}}{H_{ad}^{\;3/4}}$$

Eq. 2

where N = speed RPM

v_{inlet} = inlet flow ft^3/sec

H_{ad} = adiabatic heat $\frac{ft\ lb_f}{lb_m}$

$$D_s = \frac{D\ H_{ad}^{\;1/4}}{\sqrt{v_{inlet}}} \qquad \text{Eq. 3}$$

where D = impeller diameter ft.

$$T_{n\,1} = T_{(n-1)\;1} \left[\frac{{P_2/P_1}^{\frac{k-1}{k}} -1}{\eta\ (n-1)} + 1 \right]$$

Eq. 4

where T_{n1} = Stage inlet temperature. ^oR

$T_{(n-1)\;1}$ = Inlet temperature of the preceding stage ^oR

(P_{2/P_1}) = Compression ratio

$\eta(n-1)$ = Efficiency of the preceding stage

APPENDIX II

SLUSH STORAGE CONCEPT

The following is a preliminary concept of the facilities required for storing slush hydrogen at E/STS 2-3, Jackass Flats, Nevada.

Reference - "Criteria for Budgetary Study for Engine/Stage Test Stand 2-3"

A. Summary of Fluid Requirements

Basis - One 30 minute test run (See criteria, Fig. III-14, Fig. III-17, Table III-3)

	OPERATION	METHOD	FLUID CONSUMED LH_2	FLUID CONSUMED SLUSH
1.	Cooldown module tank.	Pressure transfer LH_2 from storage, boil in module and vent.	31,000 lb.	-
2.	Fill module tank.	Pressure transfer slush from storage.	6,000 lb.	266,000 lb.
3.	Start-up and run.	Pressure transfer slush from storage.	13,500 lb.	361,400 lb.
4.	Shut down.	Pressure transfer LH_2 from storage.	42,000 lb.	-
			92,500 lb.	627,400 lb.
	LH_2 reserve 20%		18,500 lb.	-
	Slush reserve 5%		-	32,600 lb.
	Total fluid in storage		111,000 lb.	660,000 lb.

B. Storage Concept

The liquid hydrogen will be stored in one 200,000 gallon tank (No. 1 in Figure 18). This tank will have a working pressure slightly above the maximum anticipated pressure in the module tank and will be provided with a pump and vaporizer to provide cold H_2 gas for the facility. In addition to the operations listed above, this tank will supply LH_2 and cold gas to the slush production plant.

The 440,000 gallon tank (No. 2) will supply only the slush required to fill the modular tank (Operation No. 2). It may have a low working pressure, only enough to give an acceptable transfer rate during filling.

Tank No. 3 of 600,000 gallon capacity will be connected in parallel with the module during the test run. The working pressure will be slightly above the maximum anticipated, pressure in the module tank.

An additional line with a screened entrance will be provided in the slush tanks (2 and 3) to permit drainage of triple point liquid back to the slush plant.

Tanks 2 and 3 can of course be used for storage of conventional liquid hydrogen, triple point liquid or slush as required.

C. Calculations

Operation No. 1 Assume pressure transfer of LH_2 at 3 psig using cold H_2 gas.

Gas density = 0.101 lb/ft.^{3}

$$71{,}000 \text{ lb. Aluminum} \times \frac{75 \text{ BTU/lb.Al}}{193 \text{ BTU/lb.}LH_2} = 27{,}600 \text{ lb. } LH_2$$

Add 10% for piping, etc. $\quad$ 2,760

$\qquad$ 30,360

$$30{,}360 \times \frac{0.101}{4.42} = 690$$

$\qquad$ 31,050 lb. LH_2

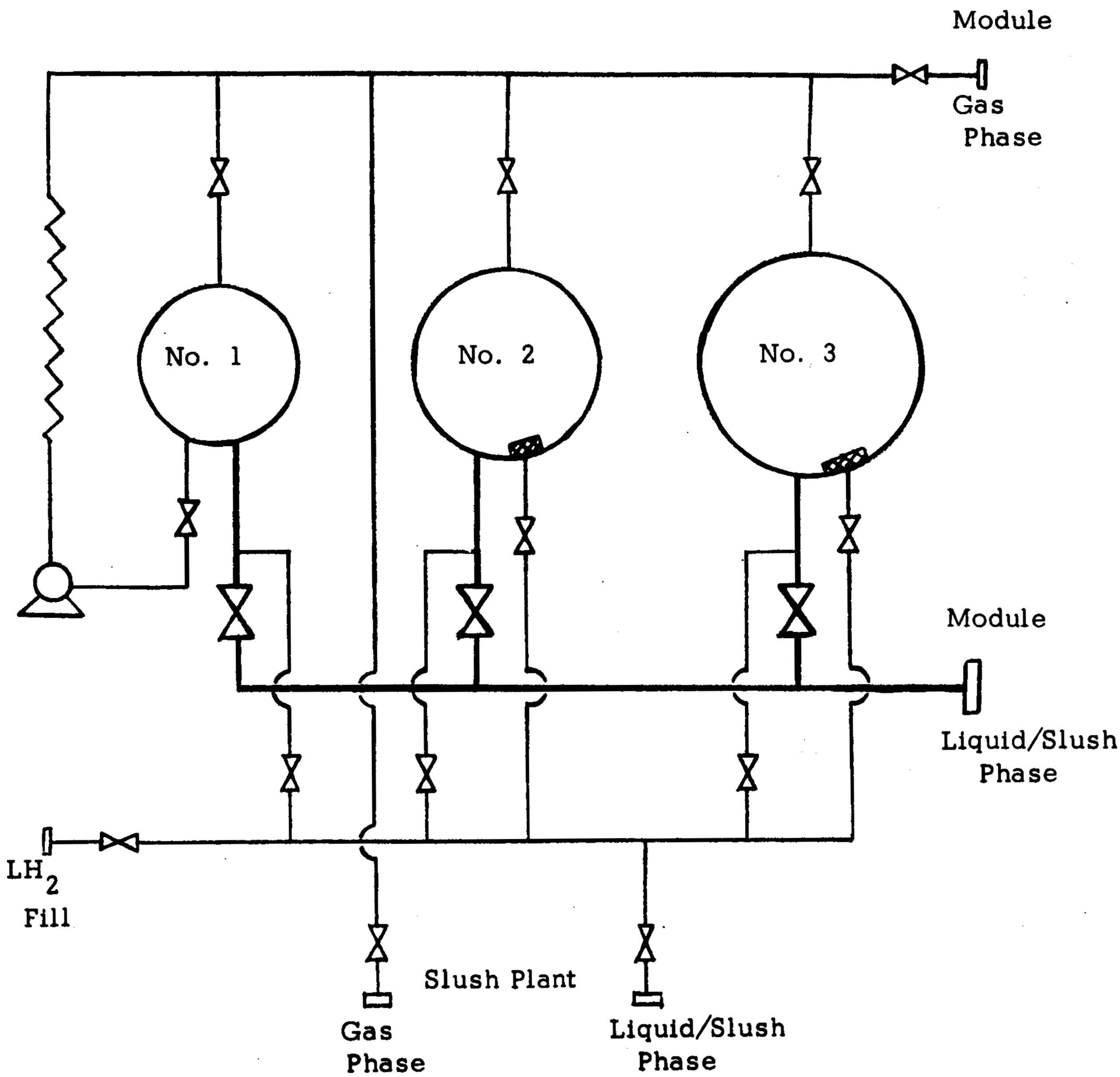

FIGURE 18

FLOW DIAGRAM-LIQUID AND SLUSH HYDROGEN STORAGE

C. Calculations - contd.

Operation No. 2

$$230{,}000 \text{ lbs. } LH_2 \times \frac{5.13}{4.42} = \underline{\underline{266{,}000 \text{ lb. slush}}}$$

Assume pressure transfer at 3 psig using cold H_2 gas.

$$266{,}000 \times \frac{0.101}{5.13} = 5{,}250 \text{ lb. } LH_2$$

$$5{,}250 \times \frac{0.101}{4.42} = 120$$

Add 10% for losses, etc. 537

$\underline{\underline{5{,}907 \text{ lb. } LH_2}}$

Operation No. 3

Run 343 lb./sec x 1,800 sec	=	617,400	lb. slush
Startup		10,000	
		627,400	
Less slush in module tank	-	266,000	
		361,400	lb. slush

Assume pressure transfer at 15 psig using cold H_2 gas.

Gas density = 0.168 lb./ft.^{3}

$$361{,}400 \times \frac{0.168}{5.13} = 11{,}800 \text{ lb. } LH_2$$

$$11{,}800 \times \frac{0.168}{4.42} = 450$$

Add 10% for losses, etc. 1,225

$\underline{\underline{13{,}475 \text{ lb. } LH_2}}$

C. Calculations - contd.

Operation No. 4

Assume pressure transfer at 15 psig using cold H_2 gas.

From "Criteria" page III-20		37,000 lb. LH_2
$37{,}000 \times \frac{0.168}{4.42}$	=	1,400
Add 10% for losses, etc.		3,830
		42,230 lb. LH_2

APPENDIX III

BIBLIOGRAPHY

1. Finger, H. B., Criteria for Budgetary Study for Engine/Stage Test Stand 2-3, August 25, 1965. Space Nuclear Propulsion Office, U. S. Atomic Energy Commission, Washington, D. C.

2. Dwyer, R. F. et al., Laboratory Production of Fluid Hydrogen Slush, I and EC Product Research and Development, Vol. 3, Page 316, December 1964.

3. Mann, D. B., et al., Characteristics of Liquid-Solid Mixtures of Hydrogen at the Triple Point, NBS Report No. 8881, October 1, 1965 National Bureau of Standards, Boulder Laboratories, Boulder, Colorado.

4. Carney, R. R., et al., Theoretical, Experimental, and Analytical Examination of Subcooled and Solid Hydrogen, Technical Documentary Report APL-TDR-64-22, April, 1964. U. S. Air Force, Wright-Patterson Air Force Base, Dayton, Ohio.

5. Cook, G. A. and Dwyer, R. F., Research on Rheologic and Thermodynamic Properties of Solid and Slush Hydrogen, Technical Report APL-TDR-65-82, October, 1965. U. S. Air Force, Wright-Patterson Air Force Base, Dayton, Ohio.

6. Elrod, C. W., Large-Scale Slush Hydrogen Production Experiments, Technical Report AFAPL-TR-65-63, July, 1965. U. S. Air Force, Wright-Patterson Air Force Base, Dayton, Ohio.

7. Paris, Leonard, Technical Proposal for Slush Hydrogen Densitometer, Document No. P-468, February, 1966. Ikor, Inc., Northwest Industrial Park, Burlington, Massachusetts.