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QUALITY DETERMINATION OF LIQUID-SOLID HYDROGEN MIXTURES

by

D. E. Daney and D. B. Mann

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to
Space Nuclear Propulsion Office
and
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Cryogenics Division
Institute for Materials Research
National Bureau of Standards
Boulder, Colorado

NBS

U.S. DEPARTMENT OF COMMERCE
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Abstract

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A theoretical and experimental investigation of the feasibility of predicting the quality of liquid-solid hydrogen mixtures from the mass fraction of vapor pumped off in the freeze-thaw process has been completed. Three independent methods of experimental quality determinations were used to check the correctness of the qualities predicted from the measured mass fraction pumped off in forming liquid-solid mixtures. In all cases only freshly made mixtures were used.

It is suggested that an independent means of determining the edge of the triple point region, such as measurement of the vapor pressure, be used. With this modification, measurement of the mass fraction pumped off during the freeze-thaw process provides a simple, non-destructive and accurate method of bulk quality determination. The method is not appropriate for cases in which a partial transfer from the dewar is made and it requires accurate knowledge of the heat leak if long storage times are to be used. Finally, since the method is only as accurate as the accuracy of the flow and liquid volume measurements, possible low accuracy in large volume gas flowmeters places a restriction on the method.

Key Words: Freeze-thaw process, liquid-solid hydrogen mixtures, quality determination, rocket propellent, slush hydrogen

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1. Introduction

Current interest in liquid-solid mixtures of parahydrogen as a potential rocket propellent (see Bibliography) has lead to this theoretical and experimental investigation of one method of determining the liquid-solid qualities. A previous investigation [Carney et al., 1964] was concerned with the general problem of quality determination while this study concentrates on one specific method and demonstrates that the measurement of one critical parameter (mass fraction removed during production pumping) is adequate for the determination of liquid-solid quality. The production method under consideration, the "freeze-thaw" process [Mann et al., 1966], is different than that used by Carney and co-workers [1964] but this difference does not affect determination of quality.

In the freeze-thaw method of forming a triple-point mixture of hydrogen, a quantity of liquid is partially evaporated under the reduced pressure maintained by a vacuum pump. A refrigeration effect, which is approximately equal to the latent heat, is experienced by the remaining liquid or liquid-solid mixture. By specifying the initial state and the process or path followed, it is possible to predict the end state, i. e., the liquid-solid quality as a function of the mass of the vapor removed.

A thermodynamic analysis of the freeze-thaw process was presented in NBS Report 8881, Section 10 [Mann et al., 1965], and is included in the Appendix. Since the irreversibilities in the actual process might result in a substantial reduction in the actual quality from that predicted by consideration of an idealized process, an experimental determination of quality versus the mass of vapor pumped off was made.

Accurate knowledge of the quality of a liquid-solid mixture is necessary in order to:

1. Determine the mass in a given volume
2. Determine the slush storage time possible
3. Determine the transport properties

2. Symbols and Properties

The symbols used in the text are given below. The values of the thermodynamic properties of parahydrogen were taken from the tables of Roder et al. [1965] except as noted.

CF	flowmeter correction factor
C_p	specific heat at constant pressure
E	heater potential in volts
F	quality, i.e. the mass fraction of the solid
F_c	quality measured by the calorimetric method
F_T	quality predicted from m_g/m
F_{v1}	quality measured from the change in volume on freezing
F_{v2}	quality measured from the change in volume on melting
F'	"adjusted" quality $F' = F\left(1 - \frac{m_g}{m}\right) + \frac{Q'_{HL}}{mT\,(s_\ell - s_s)}$
F'_c	"adjusted" quality by the calorimetric method
F'_T	"adjusted" quality predicted from m_g/m
F'_{v1}	"adjusted" quality from the change in volume on freezing
F'_{v2}	"adjusted" quality from the change in volume on melting
h_v	specific enthalpy of the vapor
I	heater current in amperes
m or M	initial mass before pumping down to the triple point, or the initial mass before solid formation
m_g or M_g	mass of the vapor pumped from the liquid as measured by the flowmeter

Δm_{liq}	change in mass of the liquid
$\mathring{Q}$	heat leak rate from the environment
Q	the total heat leak during the pump down to the triple point
Q_1	heat leak during the pump down to the triple point which is due to the heat capacity of the dewar
Q_2	the heat leak during the pump down to the triple point which comes from the environment
Q_{Heater}	the heat input from the heater
Q_{HL}	the heat input during the heating period which is due to the heat leak from the environment
Q'_{HL}	the heat input during the solid formation period which is due to the heat leak from the environment
s_ℓ	specific entropy of the liquid
s_s	specific entropy of the solid
s_v	specific entropy of the vapor
u_ℓ	specific internal energy of the liquid
u_v	specific internal energy of the vapor
v_ℓ	specific volume of the liquid
v_s	specific volume of the solid
v_v	specific volume of the vapor
ΔV_g	change in the flowmeter reading
$\Delta V_{freezing}$	change in volume of the liquid-solid mixture during solid formation
$\Delta V_{melting}$	change in volume of the liquid-solid mixture during solid melting

$(s_\ell - s_s)$	the specific entropy change on melting; the value at the triple point is taken as 8.50 joules/g-mole [Woolley et al., 1948]
$(v_\ell - v_s)$	the specific volume change on melting; the value at the triple point is taken as 2.85 cc/gm-mole [Woolley et al., 1948]

3. Description of the Apparatus

3.1. General experimental arrangement

The experimental apparatus provides for three independent determinations of the liquid-solid quality. The quality is determined from 1) the heat input required to melt a batch of slush after it has been formed, 2) the volume change during solid formation, and 3) the volume change during melting. Figure 1 is a schematic of the entire system; figure 2 shows the experimental dewar in detail. Figures3 through 5 are photographs of the apparatus.

The arrangement shown in figure 1 allows for either pumping on the dewar (V-1 open and V-2 closed) or by-passing the pump (V-1 closed and V-2 open). Valves V-3, V-4 and V-9 permit selection of either flowmeter, or the by-passing of both meters. Because of the wide variation in the flow between the boil-off measurements and the pumping measurements, two flowmeters with different capacities were used. The dewar may be filled with either hydrogen or nitrogen gas through V-10 and V-8 respectively. The inside of the heater can either be evacuated through V-5, or filled with helium from the helium supply bottle.

Figures 2 and 5 show the experimental space in greater detail. The experimental container is a 6-inch I. D. x 35-inch Pyrex dewar. It is shielded by a second Pyrex dewar filled with liquid nitrogen. Each dewar has a pair of unsilvered vertical strips oriented 180° to each other which permit visual observation of the liquid hydrogen. The stirring shaft is supported by ball bearings situated at the heater and at the radiation shield. A magnetic coupling transmits the motion from the pneumatic stirring motor to the stirring shaft inside the dewar. This coupling provides a hermetic shaft seal, allowing the dewar to be operated at low pressures without danger of air leakage.

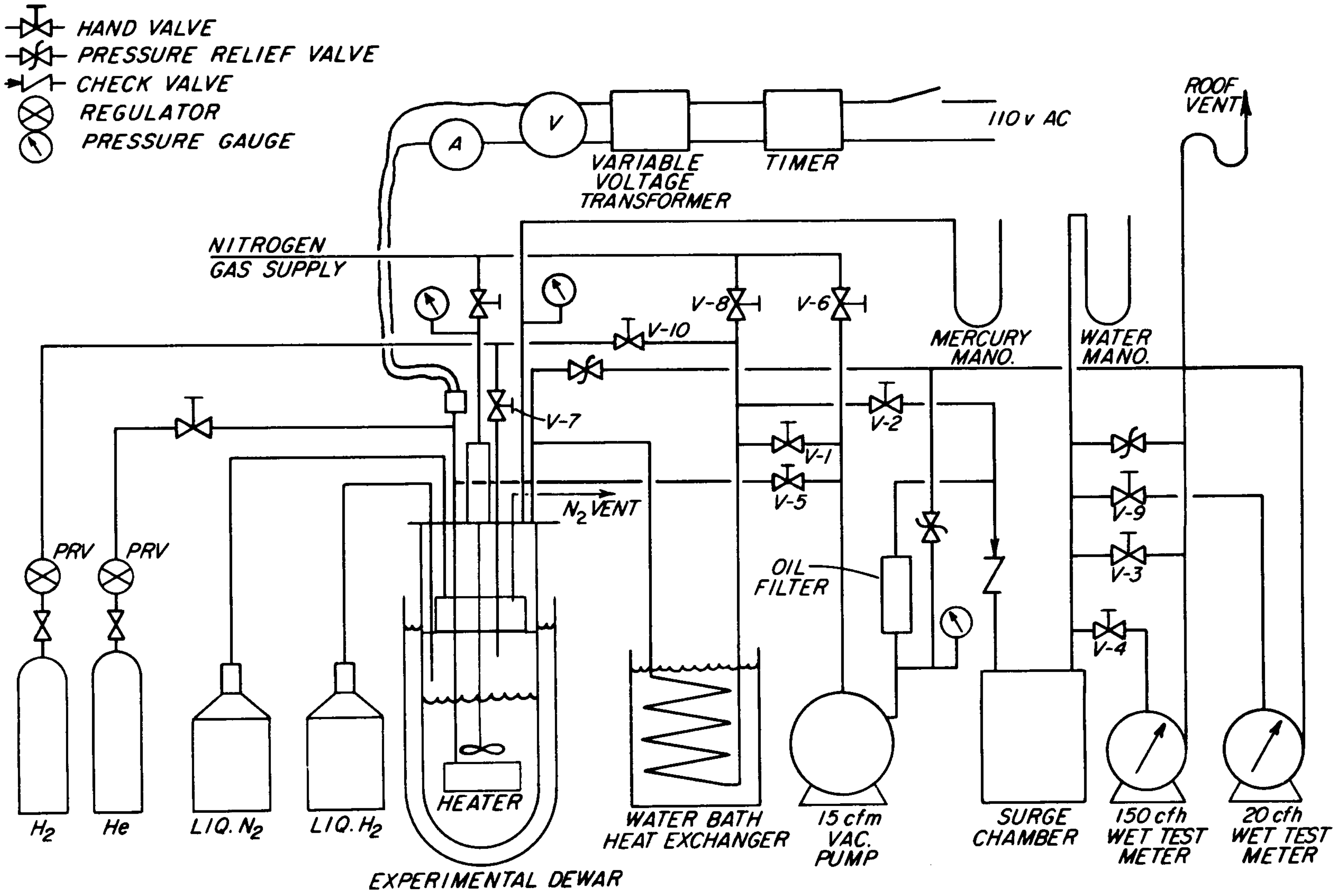

Figure 1. Flow Schematic

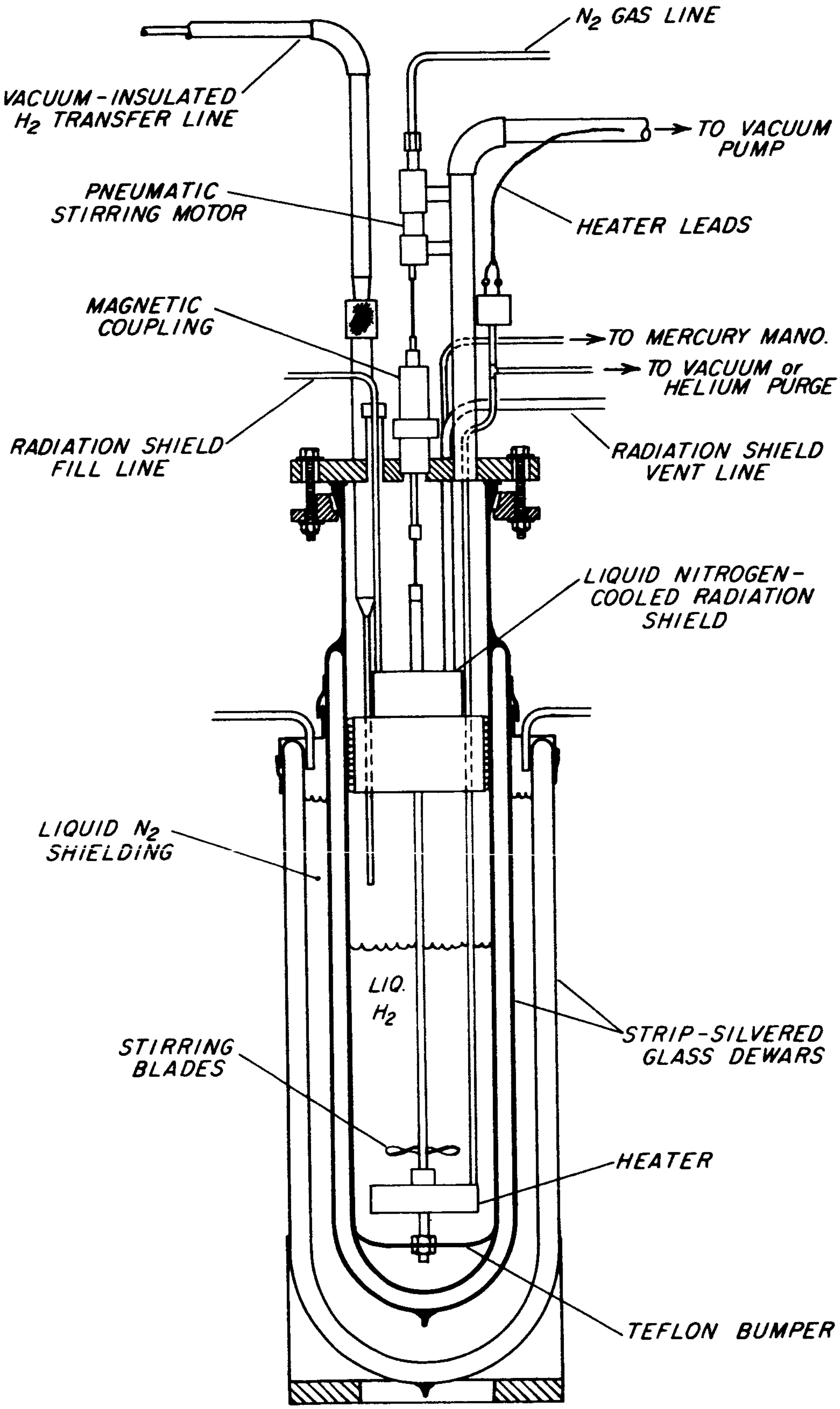

Figure 2. Dewar Cross Section

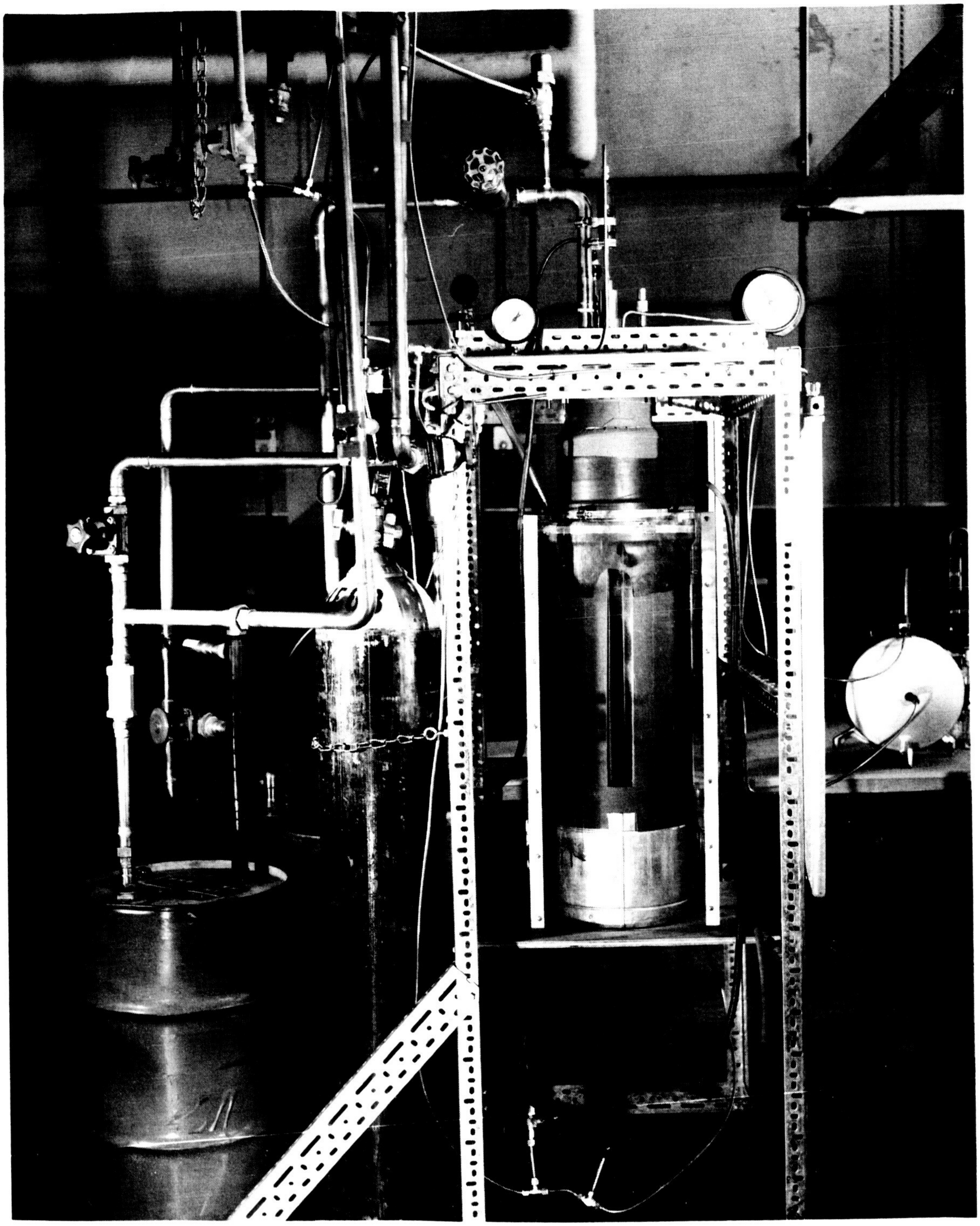

Figure 3. Photograph of the Apparatus

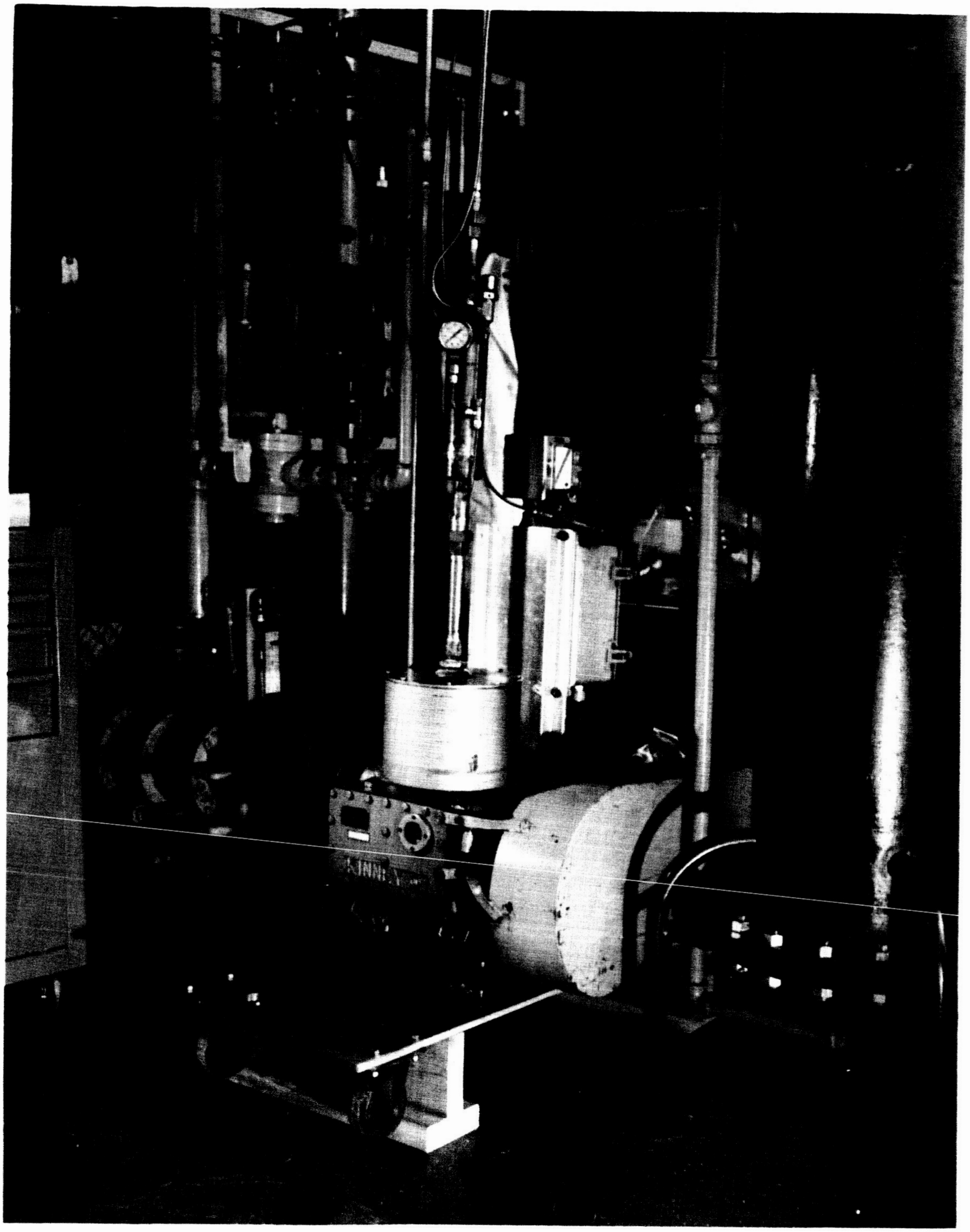

Figure 4. Photograph of the Vacuum Pump

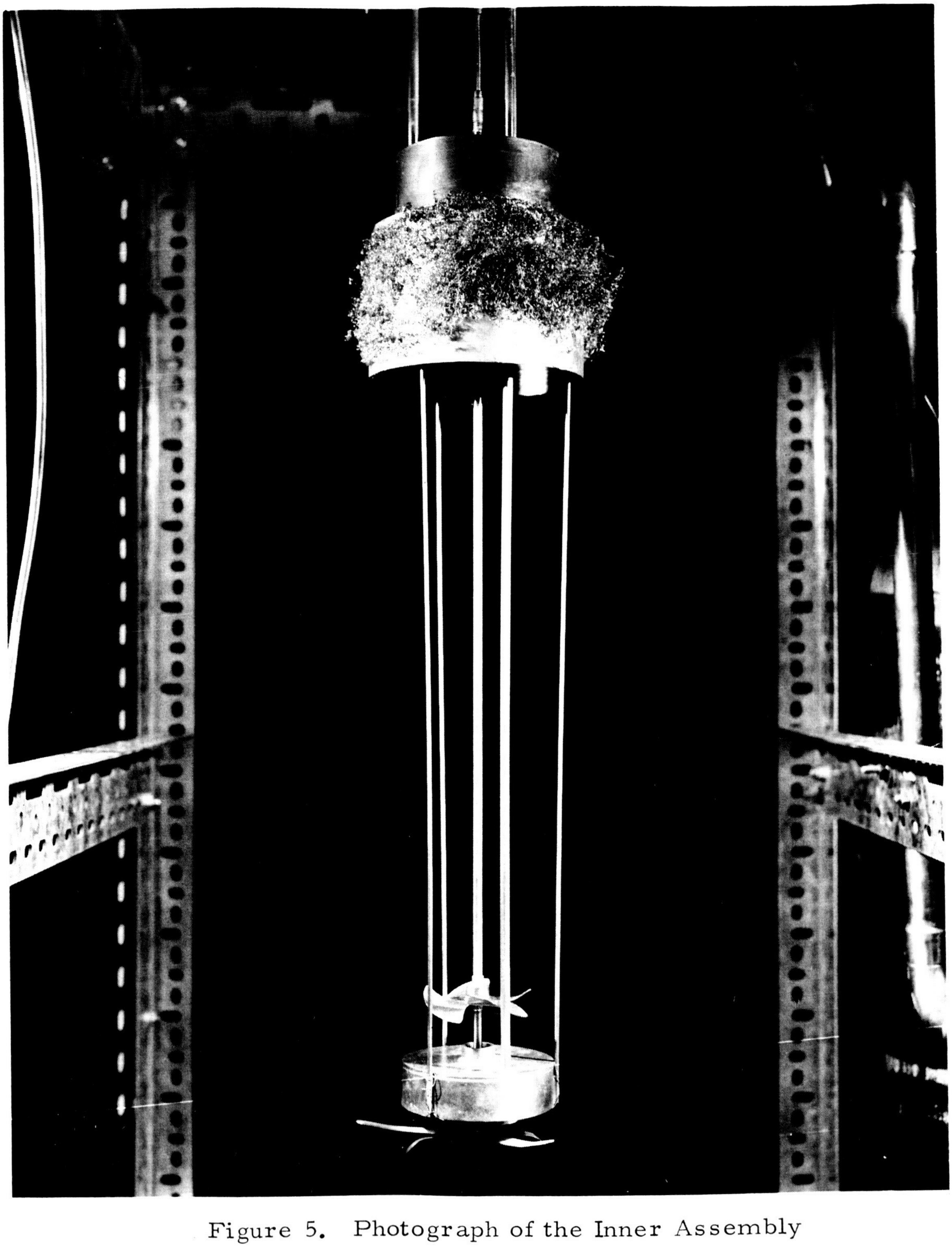

Figure 5. Photograph of the Inner Assembly

The heater is a diffusion pump heater encapsuled in a 4-inch O.D. by 1-inch copper cylinder. It is supported by a 1/4-inch stainless steel tube, which contains the heater leads, and three 1/8-inch stainless steel rods.

3.2. Instrumentation

3.2.1. Electrical energy measurement

The electrical energy introduced into the heater was measured by the arrangement shown in figure 1. The specifications for the instruments used are as follows:

Volt meter

Sensitive Research Instrument Corporation
A. C. Volt Meter
Model DEW
Scale, 0 to 75 volts
Sensitivity 0.1 volts

Ammeter

Sensitive Research Instrument Corporation
A. C. Ammeter
Model MIEW
Scale, 0 to 1.5 amperes
Sensitivity 0.001 amperes

Timer

Dimco-Gray Company
CRA-LAB Universal Timer
Type 171
Sensitivity 0.5 seconds

From calibration of the meters, correction factors of 0.984 and 0.995 were applied to the readings of the volt meter and ammeter respectively.

The heater power level is controlled by adjusting the heater voltage with a variable voltage transformer. The timer is controlled by the heater switch. The resistance of the heater leads is 0.2 ohms compared to a heater resistance of 71 Ω. Thus 0.997 of the electrical energy measured by the instruments is introduced into the experimental dewar. The electrical energy measurements were adjusted accordingly. For the power levels and heating times used in the experiment, a precision of about 0.8 percent is estimated for the measurement of the heater energy input.

3.2.2. Liquid volume measurement

The volume of the liquid or liquid-solid mixture was determined with the aid of a Wild cathetometer. The dewar was volume calibrated by filling it with water in one liter increments from a graduated flask. The distance of the liquid level from the dewar top plate was measured with the cathetometer. A table of liquid volume versus distance from the top plate (Table II) was then constructed in 1 liter increments. Linear interpolation between the 1 liter increments was used in calculating the volume from a cathetometer reading. The beam of a 1-kW sun gun, reflected off a Scotchlite screen was used to illuminate the liquid level so that it could be more easily sighted with the cathetometer. The pneumatic motor driving the stirrer was shut off during the liquid level measurements in order to obtain a relatively quiescent surface. Even so, some difficulty was experienced in measuring the liquid level due to occasional sloshing of the liquid and difficulty in picking out the same spot in the liquid meniscus each time. It is estimated that this error in the liquid level measurement could be as high as 0.2 mm. The dewar calibration indicates a variance in the dewar cross section so that some additional error in the calculated liquid volume may result from interpolating between the calibration points. The overall accuracy of the liquid volume measurement is estimated at ± 0.5 percent for measurements of the total volume. Measurements of changes in volume may contain an additional error as high as ± 0.4 mm in the liquid level measurement or 0.008 liters, although the indicated error is somewhat less than this.

3.2.3. Gas flow measurement

The volume of gas pumped through the vacuum pump was measured by wet test gas meters. An American Meter Co. No. AL-20 with a rated capacity of 150 cfh was used during high flow rates and a Precision Scientific Co. meter with rated capacity of 20 cfh was used during low flow rates.

At the rates of flow (up to 120 cfh) which were achieved when pumping on the liquid, it was found that complete saturation of the gas could not be achieved with the water bubbler which was used initially. It was decided to run the dry gas from the pump directly into the meter, and calibrate the meters for this dry gas condition using the facilities of Public Service Co. of Colorado in Denver, Colorado. A volume prover with a capacity of 10 ft^3 and a claimed accuracy of better than 1 part in 1000 was used. The agreement between a series of flow calibration points was generally within 1 part in 1000 for a given flow rate. The calibrations were made using air at 74°F with a relative humidity of 10 to 15 percent. The calibration curve for the 150 cfh meter is given in figure 6. The overall accuracy of the flow measurement is estimated at ± 0.5 percent (see Section 5.3).

3.2.4. Experimental agreement

The measurement of the change in the liquid level and the flowmeter provide two independent means of determining the mass of hydrogen pumped from the experimental dewar. These two methods should give consistent results within the limits of the experimental error. The ratio of the change in the mass measurement by the flowmeter to that

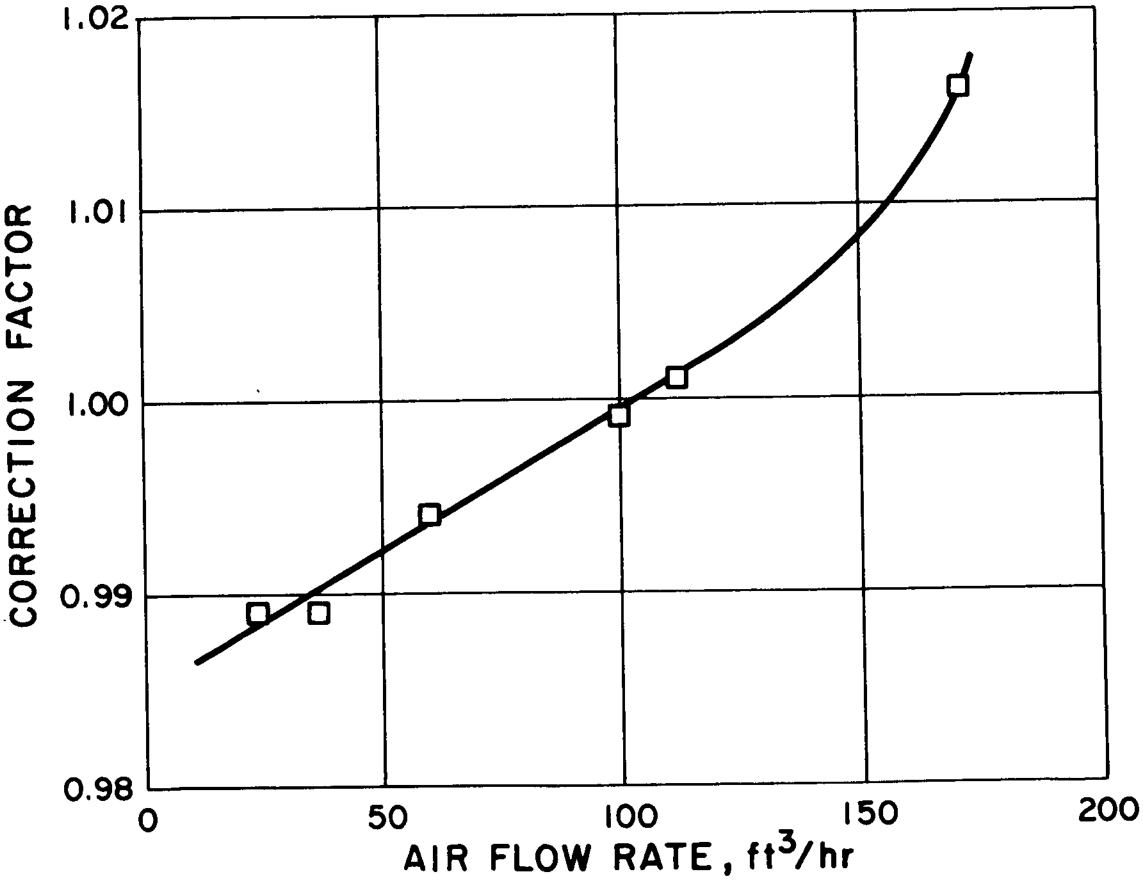

Figure 6. Flowmeter Calibration

given by the liquid level change is given in Table I.

Table I

Comparison of Mass Measurements

Date	$m_g/\Delta m_{liq}$	Liquid Level Change (mm)
1/4/66	0.991	57.01
1/19/66	0.995	71.24
1/27/66	0.996	60.11
2/21/66-A	0.992	33.67
2/21/66-B	0.985	26.77
2/23/66	0.993	96.33

These values are within the limits of the experimental error and indicate that the mass of the vapor pumped off is probably known to within the 0.5 percent estimated.

4. Experimental Procedure

The following experimental procedure was used in making the quality determinations.

Initial Preparation

1. The apparatus was tested for leaks and attention was given to liquid hydrogen safety precautions such as putting on grounding straps, lab coats, turning on ventilating fans etc.

2. The system was purged with nitrogen gas, then hydrogen gas, and the experimental dewar was filled with liquid hydrogen.

Heat Leak Measurement

1. The flow rate of the dewar boil-off gas was measured after the system came to equilibrium. This test was made with the liquid at approximately 1 atm. The stirring motor was run at an inlet pressure of 5 psig for all the tests made to maintain a reasonably uniform input of stirring work.

2. A liquid-solid mixture of low quality was formed and the time required for the heat leak to melt the solid was measured.

Pump-down to Triple Point

The liquid was pumped down to the edge of the triple-point region by opening V-1 (figure 1) with all valves other than V-4 closed. The following measurements were recorded:

1. Flowmeter at start of recording period
2. Flowmeter at finish of recording period
3. Liquid level at start of recording period
4. Liquid level at finish of recording period
5. Flowmeter inlet pressure
6. Flowmeter temperature
7. Elapsed time

8. Initial dewar pressure.

Some difficulty was experienced in determining the exact edge of the triple-point region. The criterion chosen for deciding when the triple-point boundary was reached was that there should be a few solid particles present and that they should melt only at a rate considered consistent with the heat leak. Finding the edge of the triple-point region at the end of the melting process presented the greatest difficulty since there was a tendency to overheat and melt all the particles. The maximum uncertainty in terms of F, in the edge of the triple-point boundary is estimated to be about 0.004.

Solid Formation

Once the edge of the triple point was established, solid was formed by alternately opening and closing V-1 (figure 1) i.e., by the freeze-thaw process. When the desired amount of solid had been formed, the following measurements were made:

1. Flowmeter
2. Liquid level
3. Temperature of the flowmeter
4. Elapsed time
5. The fraction of the liquid volume that contained settled slush.

Melting

The heater was then energized and the solid melted until the edge of the triple-point boundary was again achieved. After measurements were made, a new batch of slush would be made unless refilling of the dewar were required or shut-down desired. The specific measurements were:

1. Volt meter
2. Ammeter

3. Heater Time
4. Elapsed time
5. Liquid level

5. Description of the Calculations

The measurements made permit three independent evaluations of the liquid-solid quality together with the mass fraction required, both at the triple-point, and in pumping down from 1 atm.

5.1. Pump down to triple point

The heat leak term for the pump down is composed of two parts:

1. The refrigeration required to cool down the dewar walls and the heater which is given by

$$Q_1 = \left[\left(mc_p \right)_{\text{dewar}} + \left(mc_p \right)_{\text{heater}} \right] \Delta T, \text{ and}$$

2. The heat leak from the environment which is given bv

$$Q_2 = \overset{\circ}{Q} \text{ x time of the pump down.}$$

For the run of 2/23/66 these components were evaluated as $Q_1 = 136$ joules and $Q_2 = 960$ joules; the sum being 1096 joules.

Since the theoretical values of m_g/m calculated from equations (A8), (A10) and (A11) of the Appendix were evaluated for $p_o = 1$ atm with $Q/m = 0$ and $Q/m = 3.08$ j/g-mole, it was necessary to adjust these calculations to the experimental conditions. This was done using the expression

$$\frac{m_g}{m_1} = \left(\frac{m_g}{m_o} \right)_{1-2} \left(\frac{1}{1 - \left(\frac{m_g}{m_o} \right)_{0-1}} \right), \tag{1}$$

where m_o is the mass of the liquid at 1 atm and m_1 the mass at pressure p_1.

The subscripts denote the following pressures:

0 - one atm
1 - initial pressure for the experiment, and
2 - the triple-point pressure.

The quantity $\frac{m_g}{m_1}$ is calculated for the $Q/m = 0$ and $Q/m =$ 3.08 joules/g-mole using linear interpolation to find $(m_g/m_o)_{0-1}$ and $(m_g/m_o)_{1-2}$. Linear interpolation is then used between the values of (m_g/m_1) for $Q/m = 0$ and $Q/m = 3.08$ joules/g-mole for the experimental value of the heat leak. The values used for (m_g/m_o) are as follows:

For $Q = 0$

(m_g/m_o) 1 atm to 0.9 atm	0.00709
(m_g/m_o) 1 atm to the triple point	0.1057

For $Q/m = 3.08$ j/g-mole

(m_g/m_o) 1 atm to 0.9 atm	0.00727
(m_g/m_o) 1 atm to the triple point	0.1088

Since the amount of vapor which is removed in evacuating the space above the liquid was not included in the theoretical calculation of m_g/m, the flowmeter reading must be reduced by this amount. Calculations which assume a linear temperature distribution in the gas space above the liquid indicate that 22 liters should be subtracted from the flowmeter reading. However, this results in a change in mass which is about 2 percent less than that indicated by the change in the liquid level. A correction of 10 liters, which gives a mass agreement that is about that obtained while pumping at the triple point, was used in the calculations. For the pump-down to the triple point it is then estimated that m_g is known to about 1 percent instead of the 0.5 percent claimed for the other parts of the experiment.

5.2. Quality calculations

<u>Calorimetric Method</u>

$$F_c = \frac{Q_T}{(m - m_g)\ T\ (s_\ell - s_s)} , \tag{2}$$

where

$Q_T = Q_{\text{Heater}} + Q_{\text{heat leak}}$,

$Q_{\text{Heater}} = E \times I \times \text{time} \times 0.997$, and

$Q_{\text{heat leak}} = \overset{\circ}{Q}_{\text{heat leak}} \times$ heating period time.

$\overset{\circ}{Q}$ is taken from figure 7. The factor 0.997 gives the fraction of the electrical energy measured by the meters which reaches the heater.

<u>Volumetric Method - Freezing</u>

$$F_{v1} = \frac{\Delta V_{\text{freezing}} - v_\ell\, m_g}{(m - m_g)\,(v_\ell - v_s)} , \tag{3}$$

where $\Delta V_{\text{freezing}}$ is the decrease in volume during solid formation.

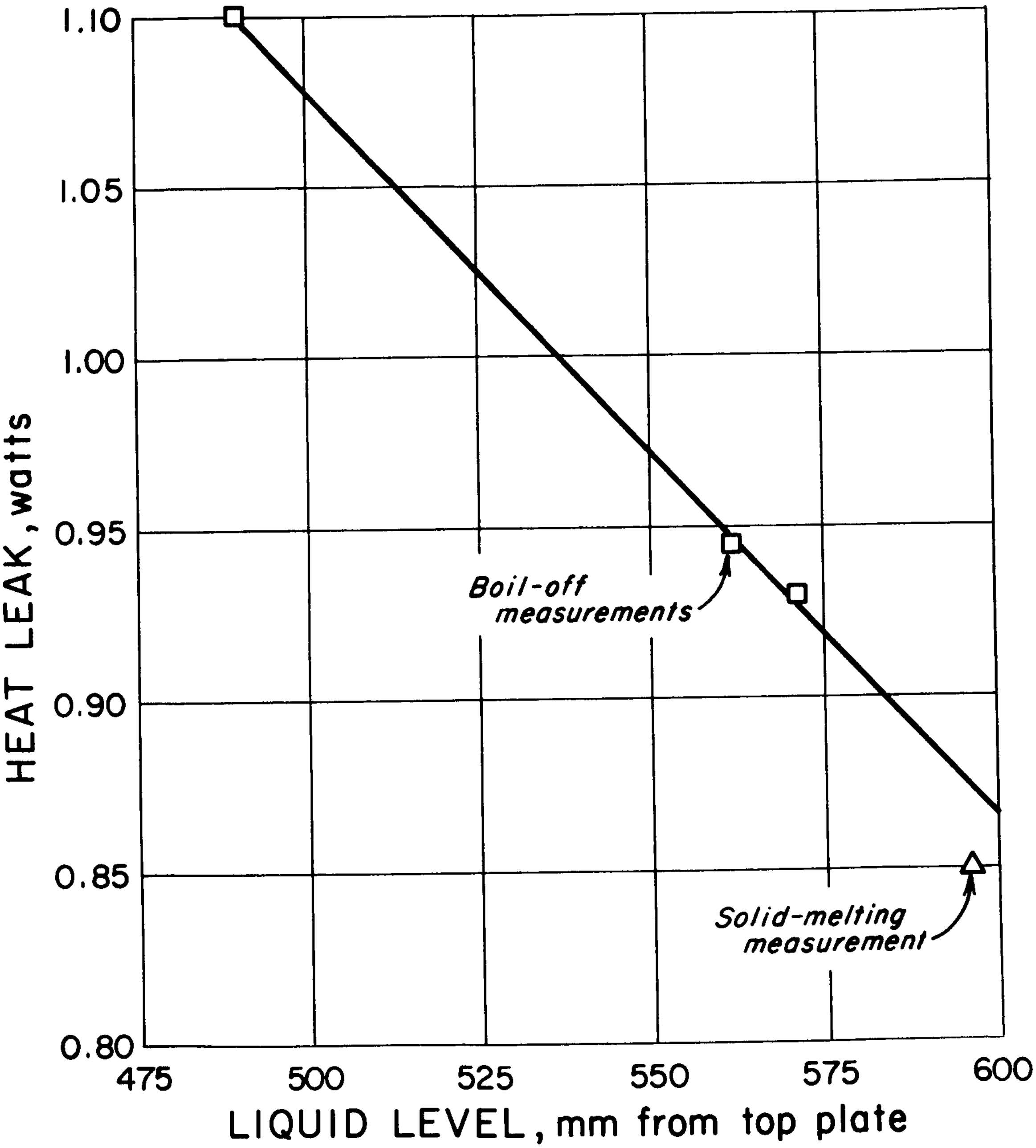

Figure 7. Dewar Heat Leak

Volumetric Method - Melting

$$F_{v2} = \frac{\Delta V_{melting}}{(m - m_g)(v_\ell - v_s)} \quad , \tag{4}$$

where $\Delta V_{melting}$ is the decrease in volume during solid formation. In all cases, m is the mass at the beginning of the solid formation, so that $m - m_g$ is the mass of the mixture when a mixture of quality F is reached.

5.3. Mass of the vapor

The mass of vapor removed by the pump was calculated from the flowmeter measurement since it gives greater precision than the liquid level measurement for small mass changes. It was calculated from the expression

$$m_g = \frac{\Delta V_g \times CF_T}{v_v} \quad . \tag{5}$$

The total flowmeter correction factor, CF_T , is calculated from

$$CF_T = CF_1 \times CF_2 \times CF_3 \quad .$$

The correction factor CF_1 is obtained from the flowmeter calibration curve, figure 6, correcting from the hydrogen flow rate to the air flow rate by the expression

$$\text{Air Rate} = \text{Hydrogen Rate} \times \left(\frac{\text{Mol. Wt. H2}}{\text{Mol. Wt. Air}}\right)^{1/2} = 0.26 \ .$$

The correction factor CF_2 corrects for the difference between the partial pressure of the water in the flowmeter at the test temperature and the calibration temperature of 74°. It is given by

$$CF_2 = 1 + \frac{0.4\,(P_c - P_t)}{P} ,$$

where

P_c = vapor pressure of water at 74°F,

P_t = vapor pressure of water at the test temperature and

P = ambient pressure.

The factor 0.4 results from the fact that the measured relative humidity of the gas leaving the flowmeter was about 40 percent.

The correction factor CF_3 corrects for the vapor mass that occupies the space of the liquid evaporated from the dewar. It is given by

$$CF_3 = 1 + \frac{v_\ell}{v_v} .$$

Typical values for the correction factor are:

CF_1 = 0.988

CF_2 = 1.001

CF_3 = 1.002

CF_T = 0.991.

It is concluded that the experimental uncertainty in the gas measurement is about ± 0.5 percent.

5.4. Mass of the liquid

The volume of the liquid or liquid-solid mixture in the dewar was taken from the dewar calibration, Table II, using linear interpolation. The mass was then determined by

$$m = \frac{V}{v_{\ell}} \quad .$$

Table II

Dewar Calibrations

Distance From Top Plate (mm)	Total Volume (liters)	Δ L For One Liter (mm)
737.86	3.000	
		52.78
685.08	4.000	
		51.87
633.21	5.000	
		51.70
581.51	6.000	
		51.88
529.63	7.000	
		50.38
479.25	8.000	
		51.42
427.83	9.000	
		49.69
378.14	10.000	
		49.04
329.10	11.000	
		51.57
277.53	12.000	

5.5. Heat leak

From the measurements of the dewar boil-off rate the heat leak was calculated by the expression

$$\overset{\circ}{Q} = \frac{\Delta m}{\text{time}} \times \left[h_v - \left(\frac{1}{1 - v_\ell / v_v} \right) \left(u_\ell - u_v \frac{v_\ell}{v_v} \right) \right] . \tag{6}$$

Measurement of the time required to melt a quantity of slush without the aid of the heater gives a second means of heat leak determination, the expression being

$$\overset{\circ}{Q} = \frac{F_T \ (m - m_g) \ T \ (s_\ell - s_s)}{\text{time}} . \tag{7}$$

The rate of heat leak versus the liquid level is plotted in figure 7.

5.6. The adjusted quality F'

The three experimentally determined values for the quality F may be compared directly to the quality predicted from the measurement values of m_g/m and Q'_{HL}/mT. A plot of F versus m_g/m would be meaningless, however, unless all the experimental F's had the same value of Q'_{HL}/mT. Equation (A16) of the Appendix suggests a means of avoiding this difficulty. Solving equation (A16) for F, the following expression is obtained.

$$F = \frac{\left(s_v - s_\ell \right) \frac{m_g}{m} - \frac{Q'_{HL}}{mT}}{\left(s_\ell - s_s \right) \left(1 - \frac{m_g}{m} \right)}$$

Further rearrangement yields the expression

$$F\left(1 - \frac{m_g}{m}\right) + \frac{Q'_{HL}}{mT\,(s_\ell - s_s)} = \left(\frac{s_v - s_\ell}{s_\ell - s_s}\right) \frac{m_g}{m} \quad . \tag{8}$$

A plot of the left-hand side of (8) versus m_g/m yields a straight line passing through the origin with a slope of $(s_v - s_\ell)/(s_\ell - s_s)$ equals 1/0.130. Plotting the experimental points in this manner has the advantage that, regardless of the value of the heat leak Q'_{HL}, the experimental values of F are compared to a single straight line passing through the origin.

The heat leak Q'_{HL} in equation (8) is the heat leak during the period of solid formation. It is calculated by picking the heat leak rate $\overset{\circ}{Q}$ corresponding to the liquid level in the dewar from figure 7.

$$Q'_{HL} = \overset{\circ}{Q} \text{ x solid formation time.}$$

6. Experimental Results

The measured values of the mass fraction of the initial liquid which is removed in pumping down to the triple point are given in Table III. The values of $(m_g/m)_{\text{predicted}}$ are calculated as described in Section 5.1.

Table IV and figures 8, 9 and 10 give the experimental values of the mass fraction of triple-point liquid pumped off to form a mixture of quality F. The adjusted quality

$$F' = F\left(1 - \frac{m_g}{m}\right) + \frac{Q'_{HL}}{mT\,(s_\ell - s_s)}$$

is plotted in the figures (see Section 5.6.). The line giving the predicted value of F' is obtained from equation (8), Section 5.6.

Table III

Mass Fraction–Pump Down To Triple Point

Run No.	Initial Pressure (mm Hg)	Heat Leak-Q/m (joules/g-mole)	m_g/m Predicted	m_g/m Experimental
2/21/66-A	735.9	3.67	0.1073	0.1079
2/21/66-B	727.4	3.22	0.1061	0.1027
2/23/66	730.9	3.38	0.1067	0.1005

Table IV

Mass Fraction For Quality F

RUN NO.	M_g/M	F_C	F_C'	F_T'	$F_C' - F_T'$	F_{V1}	F_{V1}'	$F_{V1}' - F_T'$	F_{V2}	F_{V2}'	$F_{V2}' - F_T'$	$\frac{Q_{HL}'}{MT(S_\ell - S_V)}$
2/21/66-A												
1	0.0264	0.178	0.194	0.204	-.010	0.199	0.215	+.011	0.193	0.209	+.005	0.021
2	0.0208	0.144	0.159	0.161	-.002	0.143	0.158	-.003	0.134	0.149	-.012	0.018
3	0.0274	0.184	0.201	0.212	-.011	0.199	0.216	+.004	0.202	0.218	+.006	0.022
2/21/66-B												
1	0.0226	0.157	0.175	0.175	0	0.163	0.181	+.006	0.167	0.185	+.010	0.022
2	0.0209	0.141	0.159	0.162	-.003	0.153	0.171	+.009	0.145	0.163	+.001	0.021
3	0.0201	0.140	0.156	0.155	-.001	0.139	0.155	0	0.139	0.155	0	0.019
4	0.0296	0.206	0.222	0.229	-.005	0.210	0.226	-.003	0.205	0.221	-.008	0.022
2/23/66												
1	0.0379	0.249	0.279	0.293	-.014	0.273	0.302	+.009	0.258	0.287	-.006	0.039
2	0.0552	0.376	0.409	0.427	-.018	0.416	0.447	+.020	0.389	0.422	-.005	0.054
3	0.0557	0.378	0.405	0.430	-.025	0.369	0.396	-.034	0.393	0.419	-.011	0.048
4	0.0335	0.240	0.274	0.259	+.015	0.259	0.292	+.033	0.260	0.293	+.034	0.042
5	0.0213	0.161	0.176	0.165	+.011	0.158	0.173	+.008	0.157	0.172	+.007	0.018
6	0.0138			0.107		0.102	0.113	+.006	0.108	0.119	+.012	0.012
7	0.0145	0.098	0.111	0.112	-.001	0.096	0.109	-.003	0.090	0.103	-.009	0.014
8	0.0117	0.080	0.090	0.090	0	0.071	0.081	-.009	0.085	0.095	+.005	0.011
9	0.0117	0.082	0.091	0.090	+.001	0.093	0.102	+.012	0.087	0.098	+.008	0.010
10	0.0180	0.122	0.132	0.139	-.007	0.136	0.146	+.007	0.120	0.130	-.009	0.012

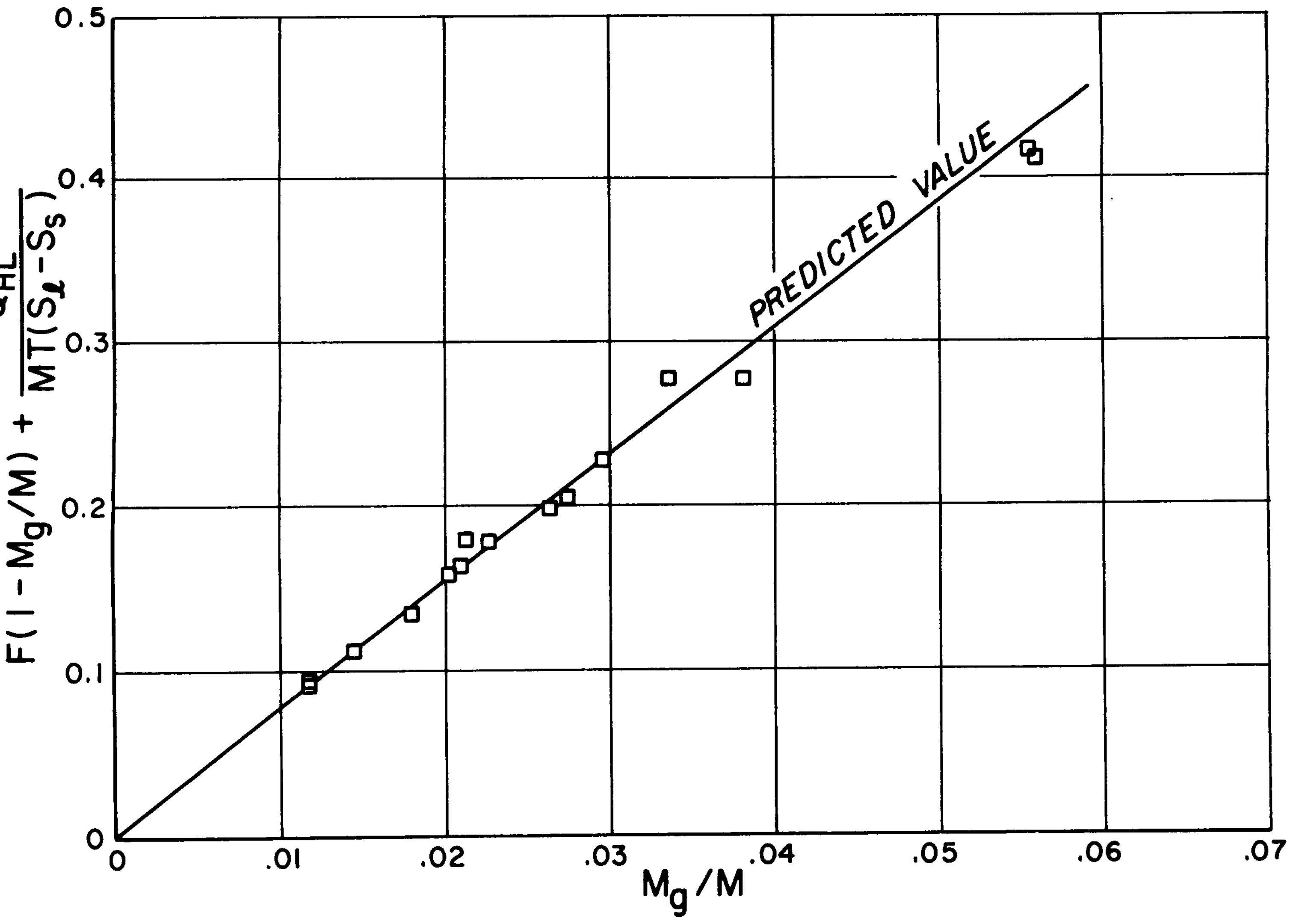

Figure 8. Experimental Quality - Calorimetric Method

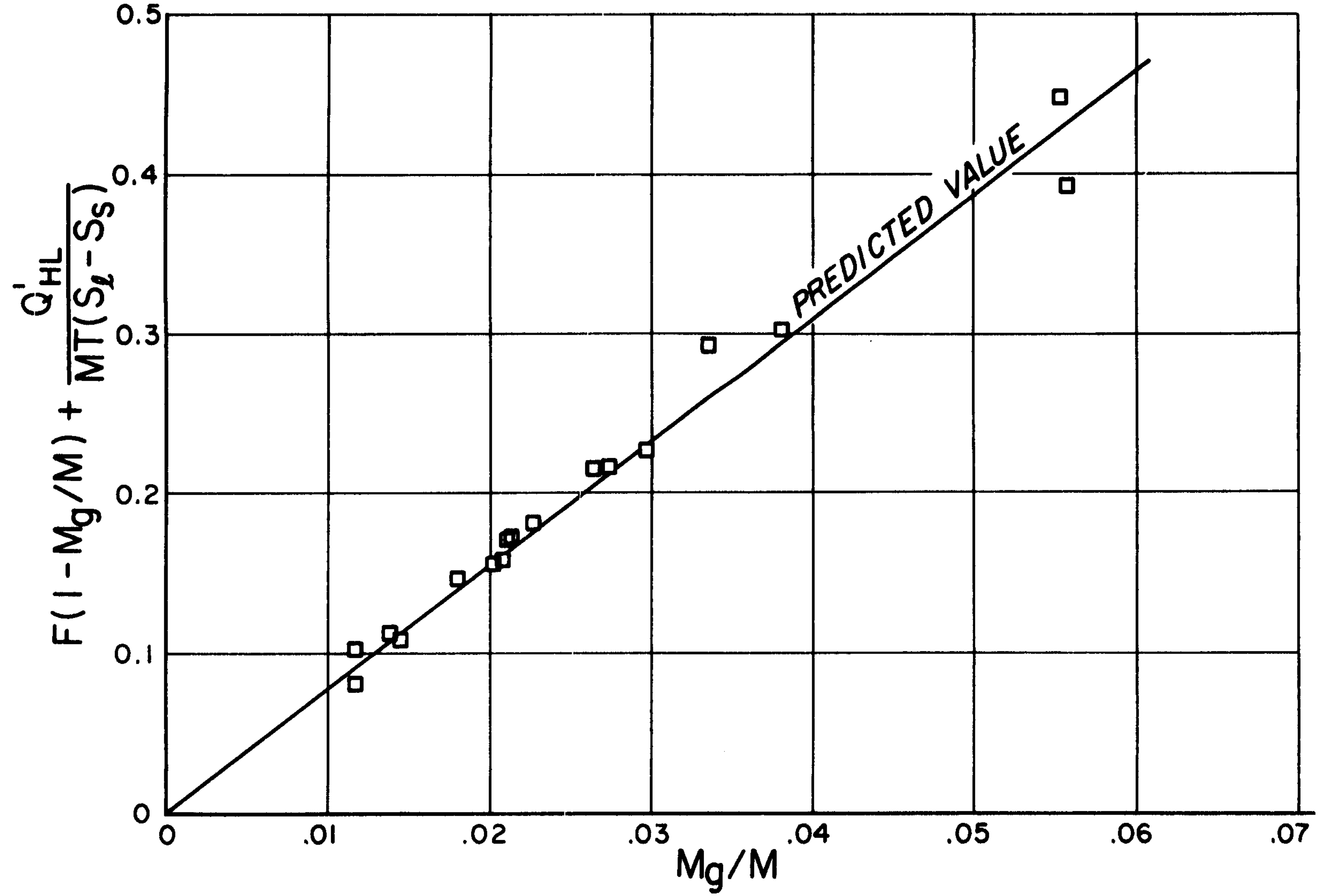

Figure 9. Experimental Quality - Volumetric Method - Freezing

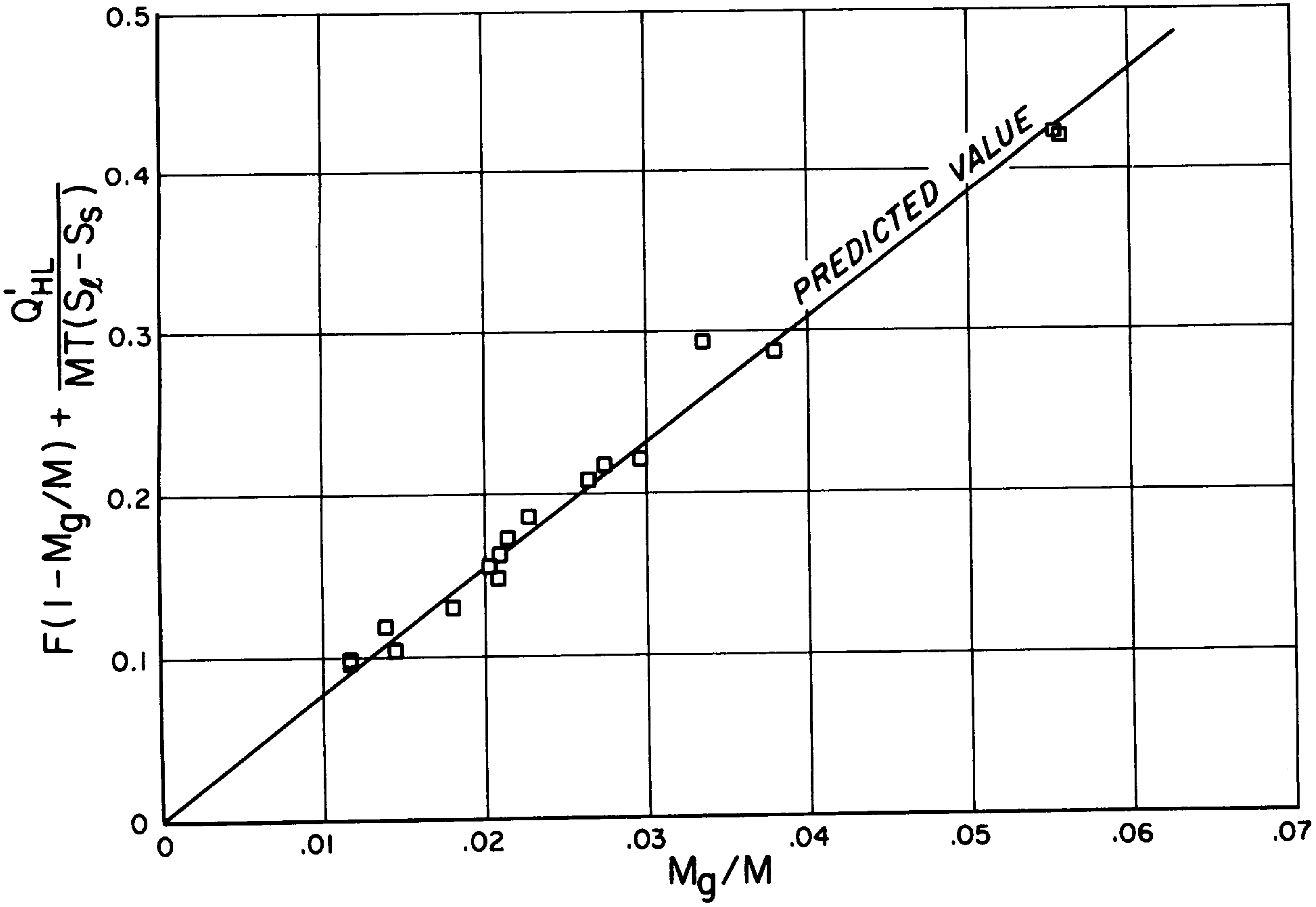

Figure 10. Experimental Quality - Volumetric Method - Melting

7. Discussion of the Results

7.1. Pump down to the triple point

The standard deviation of the three points (adjusted to the same value of $(m_g/m)_{predicted}$) from their mean value is 2.6 percent of m_g/m. The standard deviation of the experimental values of m_g/m from the predicted values of m_g/m is 4.0 percent. The mean value of $(m_g/m)_{experimental}$ is 2.8 percent below $(m_g/m)_{theoretical}$. The small number of experimental points may be one source of the disagreement in the results since an error in one of the measurements would have a significant effect on the standard deviation. Other possible sources of the disagreement between the experimental and predicted results are as follows:

1. The uncertainty in the theoretical calculation could be as high as 1.6 percent. The uncertainty in the value of the change in the saturated liquid entropy between one atmosphere and the triple point is ± 0.048 joules/g-mole [Roder et al., 1965]. The uncertainty in the calculated value of m_g/m, resulting from the uncertainties in the entropy values used is 1.1 percent. There is an additional uncertainty, which is estimated at 0.5 percent, due to the approximate nature of the calculation procedure for m_g/m.

2. The uncertainty in the determination of m_g/m, as given previously, is about ± 1.5 percent for this part of the experiment.

3. The uncertainty in determining the edge of the triple-point region is estimated to cause an uncertainty ± 0.3 percent in m_g/m.

4. Some uncertainty exists in the heat leak rate during this portion of the experiment since the conditions are different from those under which the heat leak was measured. The high rate of vapor flow cools the dewar wall, while the recession of the liquid level by 70 to 80 mm results in a

dewar wall section above the liquid with a smaller temperature gradient. Since the primary source of heat leak is by solid conduction down the dewar walls, the total heat leak into the dewar should be reduced. An attempt to measure this reduction in the heat leak was made by comparing the heat input from the heater to the mass of vapor given off at a flow rate comparable to that experienced during the pump down. The results of the tests were inconclusive since the difference was beyond the experimental accuracy. However, as an estimate of the possible magnitude of this effect, a 50 percent reduction in the conduction into the liquid would cause a 1.3 percent reduction in the theoretical value of m_g/m.

To summarize, the possible sources of error are as follows:

1.	Theoretical calculation	± 1.6%
2.	m_g/m_{exp}	± 1.5%
3.	Triple-point boundary	± 0.3%
4.	Heat leak	± 1.3%
	Total possible disagreement	± 4.7%

The 4 percent standard deviation of the experimental m_g/m from the predicted m_g/m is within the limits of the experimental error. If it is desired to predict the quality from the mass fraction which includes the pump down to the triple point, greater accuracy is desired. The effect of this error is discussed in section 8.

7.2. Quality measurements

The standard deviations and the maximum deviations of the experimental qualities from the predicted qualities in terms of the adjusted quality F', are as follows:

	$\Delta F'$
1. Calorimetric Method	
Standard deviation	0.011
Maximum deviation	0.025

2.	Volumetric Method - freezing	$\Delta F'$
	Standard deviation	0.014
	Maximum deviation	0.034
3.	Volumetric Method - melting	$\Delta F'$
	Standard deviation	0.011
	Maximum deviation	0.034

Some of the sources of experimental uncertainties have been discussed previously. They are summarized as follows:

Possible Experimental Errors

Gas measurement	± 0.5%
Total liquid volume	± 0.5%
Changes in liquid volume	± 0.008 liters
Heater energy	± 0.8%
Triple-point boundary	± 0.004 in terms of the quality F
Heat leak rate	± 0.1 watt

The uncertainties in the values used for $(s_v - s_\ell)$ [Roder et al., 1965], $(s_\ell - s_s)$ and $(v_\ell - v_s)$ [Woolley et al., 1948] are ± 0.2 percent, ± 0.5 percent and ± 3 percent respectively.

The standard deviations of the experimentally determined F's from the predicted values lie well within the limits of the experimental error. The maximum deviations are about the same as estimated maximum experimental error.

Reference to Table IV shows that these maximum deviations occur in runs two and three of 2/23/66 with opposite sign. This suggests that an error in reading the flowmeter, missing the triple-point boundary, or a combination of errors may have occurred in the experiment at this point.

Any deviations of the true value of F' from the predicted value of F' should be due either to

1) irreversibilities in the freeze-thaw process
2) errors in the thermodynamic properties used, or
3) use of an incorrect model for the process.

Only 1), would cause scatter in the data, and this scatter should be small compared to the total magnitude of the irreversibility.

If each series of solid formations are considered as a single run, the error due to missing the edge of the triple-point region and incorrect meter reading will be reduced, and a better idea gained of the true deviation of F' experimental from F' predicted. Table V presents the data in this manner.

In some of the runs, pieces of solid were frozen to the dewar wall and the stirring shaft above the liquid level when the measurement of the liquid level was made at the end of the solid formation. This accounts for at least some of the difference between the values of F'_{v1} and F'_{v2}.

Table V

Consideration Of A Series Of Runs As A Single Run

Run	$\frac{\Sigma (F' - F'_T)}{\Sigma F'_T}$	$\Sigma F'_T$
Calorimetric Method		
1/21/66-A	-.040	0.577
1/21/66-B	-.008	0.932
1/23/66	-.019	2.002
All Runs	-.019	3.511
Volumetric Method - freezing		
All Runs	+.020	3.511
Volumetric Method - melting		
All Runs	-.005	3.511

It is concluded, then, that

1) no significant irreversibilities occur in the freeze-thaw process,

2) the quality may be predicted to at least 0.011 from the mass fraction removed, and

3) the error in predicting the quality by this method is less than 2 percent for a quality of 0.50.

7.3. Observations

The 15 cfm pump used in the experiment seemed to be marginal for the freeze-thaw process. It was difficult to consistently get the violent action at the surface of the liquid which is required. When the apparatus was used with liquid nitrogen (with a mass removal rate about 1.7 times that of H_2) in preliminary tests, the surface action seemed satisfactory. The pumping rate to surface area ratio for the apparatus is 77 cfm/ft^2.

The maximum liquid-solid quality attained during the experiments was 0.39. To achieve this concentration it was necessary to stop the stirring motor for most of the last half of the solid formation period in order to allow the slush to settle. When the measurements were made at the end of the slush formation period, about 5 percent of the volume below the liquid-vapor interface was clear liquid. Attempts to predict the maximum quality are difficult for short settling times.

It is recommended that the insertion of tubes into the liquid be avoided since large heat leaks may result from the oscillation of the liquid inside the tube. Heat leaks as large as 8 watts can occur down a 1/4-inch tube.

8. Conclusions

The most accurate determination of the quality requires an independent determination of the triple-point boundary. Since the mass fraction of the initial liquid which is removed in pumping down to the triple point is almost twice that required to form a mixture of 0. 5 quality, once at the triple point, errors in determining the flow will be magnified in the determination of the quality. In addition, there is a 3 percent difference between the average experimental and theoretical mass requirement for the pump down.

An independent determination of the boundary of the triple point region, for instance by measuring the pressure in the production dewar, largely eliminates this difficulty. A 1-mm Hg error in determining the triple point by the vapor pressure results in an error in the quality of only 0. 003 or about 1/20 the error that might easily occur when starting the vapor measurement from 1 atm.

Prediction of the quality by measuring the mass fraction of the gas pumped off once at the triple point appears to be an accurate and practical method of quality measurement. The experimentally measured qualities agree with the predicted qualities to within 2 percent. No significant irreversibilities appear to occur in the freeze-thaw process. The primary sources of error in this method are the errors in the flow measurement and the liquid volume measurement.

For large scale applications flow and volume meters will be less accurate than those used in this laboratory experiment. As an example, for a flowmeter accurate to ± 2 percent and a liquid volume measurement accurate to ± 2 percent, the maximum uncertainty in the quality of a 0. 50 solid mixture would be 0. 030, 0. 010 of this being the maximum uncertainty if the flow and volume are known exactly. This 0. 030 uncertainty in the quality contributes an uncertainty of 0.36 percent in the determination of the total mass held in a container. For a container holding 10,000 gallons, a maximum error in the estimated weight of 24 pounds would result.

In conclusion, measurement of the mass fraction pumped off during the freeze-thaw process provides a simple, non-destructive and accurate method of quality determination. It offers the following advantages:

1. It requires the measurement of only
    a) the mass of the vapor pumped off,
    b) the liquid mass either before or after pumping, and
    c) the heat leak into the dewar.
2. It is non-destructive, i.e., it does not require melting the slush.
3. It requires no viewports or apparatus inside the dewar, with the exception of a liquid level indicator.
4. It does not require a homogeneous distribution of the solid.

Since this method measures the bulk quality, it is not good for cases in which a partial transfer from the dewar is made. If the slush is stored for long periods, the uncertainty in the quality will be increased due to uncertainties in the heat leak into the dewar. Finally, since the method is only as accurate as the accuracy of the flow and liquid volume measurement, possible low accuracy in large volume gas flowmeters places a restriction on the method.

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11. Appendix

11.1. Quality determinations

In the freeze-thaw method of forming a triple-point mixture of hydrogen, a quantity of liquid hydrogen is partially evaporated under the reduced pressure maintained by a vacuum pump. A refrigeration effect, which is approximately equal to the latent heat of vaporization, is experienced by the remaining liquid or liquid-solid mixture. By specifying the initial state and the process or path followed, it is possible to predict the end state, i.e., the liquid-solid quality as a function of the mass of the vapor removed.

Figure 11 shows the path of the processes, on a temperature-entropy diagram, when the material in a constant volume container is considered. Saturated liquid at 1 atmosphere is cooled to the triple point, process 1 -2, and then solid is formed, process 2 - 3''.

11.1.1. Expansion to the triple point

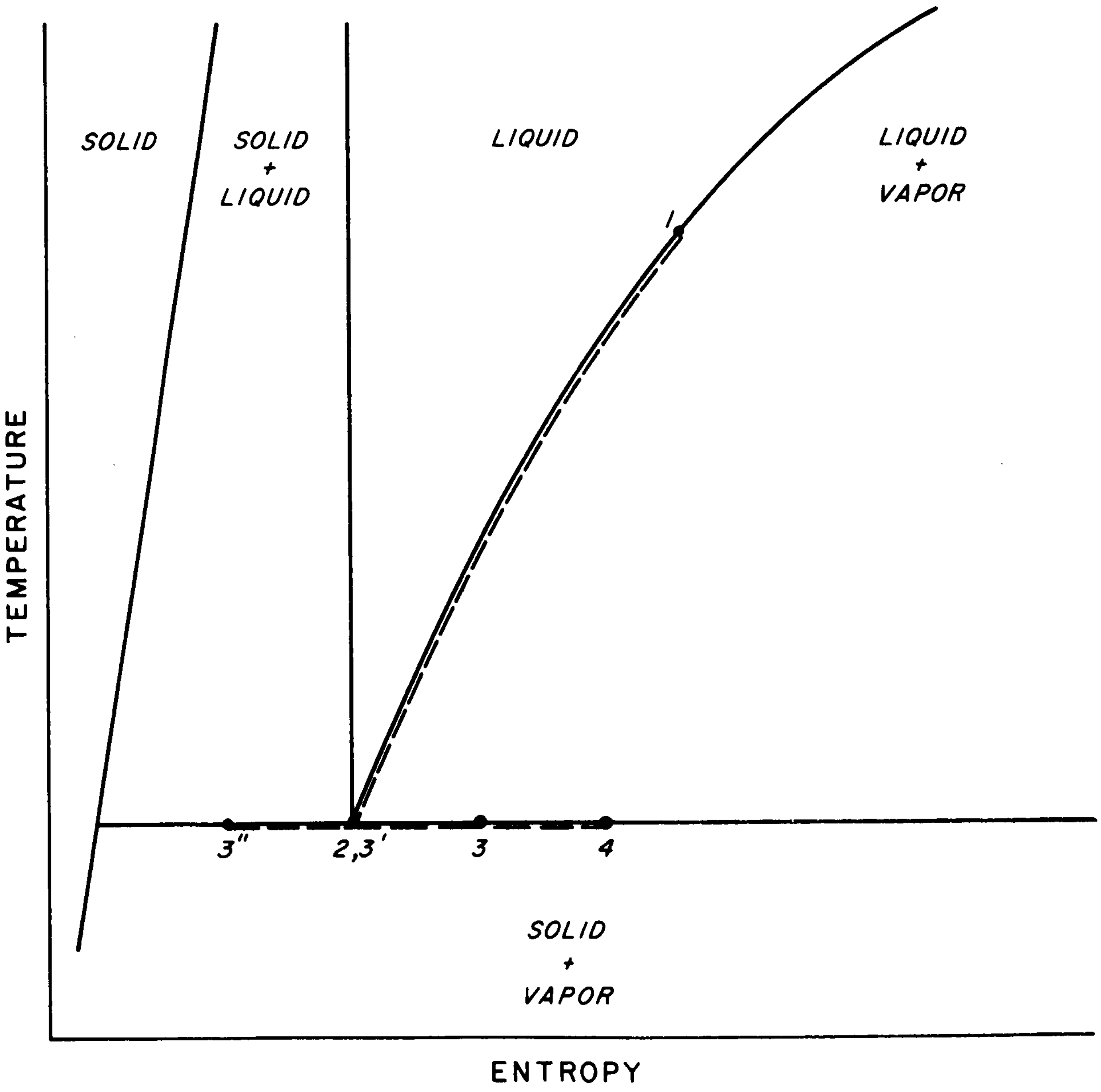
Process 1 -2 may be analyzed by considering a control volume around a container initially filled with liquid hydrogen at one atmosphere. For a reversible process with heat transfer dq and mass leaving dm_i, the expression for the entropy in the control volume is

$$S_1 + \int_1^2 \frac{dq}{T} = S_2 + \int_1^2 s_{vi}\, dm_i . \tag{A1}$$

Since the evaluation of

$$\int_1^2 s_{vi}\, dm_i$$

requires solution of the problem at hand, it is necessary to consider a series of incremental expansions and replace the mass average entropy of the gas leaving by the arithmetic mean entropy. For such an incremental step, equation (A1) may be rewritten as:



PROCESSES IN HYDROGEN SLUSH PRODUCTION AND QUALITY MEASUREMENT

Figure 11. T-S Diagram for Solid Production

$$\frac{dq}{T} + m_{\ell 1} s_{\ell 1} + m_{v1} s_{v1} = m_{\ell 2} s_{\ell 2} + m_{v2} s_{v2} + \Delta m_{1-2} s_{avg}, \qquad (A2)$$

where

$$s_{avg} = \frac{s_{v1} + s_{v2}}{2} \quad .$$

With the specification of the conservation of mass,

$$m_{\ell 1} + m_{v1} = m_{\ell 2} + m_{v2} + \Delta m_{1-2} \quad , \qquad (A3)$$

and the requirement of constant volume

$$v_{\ell 1} m_{\ell 1} + v_{v1} m_{v1} = m_o v_o \quad , \text{ and} \qquad (A4)$$

$$m_{\ell 2} v_{\ell 2} + m_{v2} v_{v2} = m_o v_o \quad , \qquad (A5)$$

the expansion process is defined and equations (A2) through (A5) may be solved for $\frac{m_{\ell 2}}{m_{\ell 1}}$ and $\frac{\Delta m_{1-2}}{m_{\ell 1}}$. The subscripts 1 and 2 refer to the initial and final states for any incremental step, not the initial and final steps of the complete expansion. The subscript o refers to the initial conditions at 1 atm., state 1 on figure 11. The resulting expressions are

$$\frac{m_{\ell 2}}{m_{\ell 1}} = \frac{\left(s_{v1} + s_{v2} - 2s_{\ell 1} - 2 \frac{dq}{m_{\ell 1} T}\right) + R_1}{\left(s_{v1} + s_{v2} - 2s_{\ell 2}\right) + \frac{v_{\ell 2}}{v_{v2}} \left(s_{v2} - s_{v1}\right)} \quad , \qquad (A6)$$

where

$$R_1 = \left(s_{v2} - s_{v1} \right) \left[\frac{m_o}{m_{\ell 1}} \; \frac{v_o}{v_{v2}} \left(1 + \frac{v_{v2}}{v_{v1}} \right) - \frac{v_{\ell 1}}{v_{v1}} \right] , \qquad \text{(A7)}$$

and

$$\frac{\Delta m_{1-2}}{m_{\ell 1}} = 2 \; \frac{\left(s_{\ell 1} - s_{\ell 2} \right) + \left(1 - \frac{v_{\ell 2}}{v_{v2}} \right) dq/m_{\ell 1} T + R_2}{\left(s_{v1} + s_{v2} - 2 s_{\ell 2} \right) + \frac{v_{\ell 2}}{v_{v2}} \left(s_{v2} - s_{v1} \right)} , \qquad \text{(A8)}$$

where

$$R_2 = \frac{m_o \, v_o}{m_{\ell 1} \, v_{v2}} \left[\left(1 - \frac{v_{v2}}{v_{v1}} \right) s_{\ell 2} - \left(1 - \frac{v_{\ell 2}}{v_{v1}} \right) s_{v2} + \left(\frac{v_{v2} - v_{\ell 2}}{v_{v1}} \right) s_{v1} \right]$$

$$+ \frac{v_{\ell 2}}{v_{v2}} \left[\left(1 - \frac{v_{\ell 1}}{v_{v1}} \right) s_{v2} - s_{\ell 1} \right]$$

$$+ \frac{v_{\ell 1}}{v_{v2}} \left[\frac{v_{v2}}{v_{v1}} \, s_{\ell 2} - s_{v1} \left(\frac{v_{v2} - v_{\ell 2}}{v_{v1}} \right) \right] . \qquad \text{(A9)}$$

If only an approximate solution is desired, equations (A6) and (A9) may be simplified to

$$\frac{m_{\ell 2}}{m_{\ell 1}} = \frac{\left(s_{v1} + s_{v2} - 2 \, s_{\ell 1} - 2 \, dq/m_{\ell 1} T \right)}{\left(s_{v1} + s_{v2} - 2 s_{\ell 2} \right)} \qquad \text{(A10)}$$

and

$$R_2 = 2 \, \frac{v_{\ell o}}{v_{v2}} \left(s_{\ell 2} - s_{\ell 1} \right) . \qquad \text{(A11)}$$

Equations (A8), (A10), and (A11) give a value of 0.106 for $\frac{\Delta m_{0-1}}{m_o}$, the fraction of the initial mass vaporized in going from 1 atm to the triple point, for the case of zero heat leak. The error resulting from the simplification of the expressions is estimated at less than one percent. The parahydrogen properties used are those reported by Roder, Weber, and Goodwin [1963] and Roder [1964].

The specification of the heat leak dq could pose a problem if dq were sufficiently large. For the low rates of heat leak that are expected to be encountered, however, the assumption that Q is distributed evenly over the temperature range, i. e.,

$$dq_{i \rightarrow (i+1)} = Q \frac{T_i - T_{i+1}}{T_o - T_1} \qquad \text{(A12)}$$

will give sufficiently accurate results. The minimum change in entropy would occur if all the heat were added at temperature T_1 and the maximum entropy change would occur if it were added at temperature T_2. If process 1 - 2 were to require one hour in a vessel having a heat leak equivalent to a loss of one percent of the liquid per day, Q/m would be 0.38 joules/gram mole and the maximum possible error introduced in the determination of m_v by using equation (A12) would be 0.05 percent for hydrogen. The actual error would be considerably smaller. Larger rates of heat leak or larger times for the process would result in proportionately larger errors.

11.1.2. Formation of solid at the triple point

Since the formation of solid at the triple-point, process 2 - 3'', takes place at constant temperature and pressure, the process may be analyzed by considering a system contained by a piston and cylinder. If no heat is transferred to the system and the process takes place reversibly, then as the piston is drawn out, the hydrogen expands with no change in entropy. For this closed system, the end of the expansion with some quality F is denoted by state 3' at the same entropy as state 2. If there is heat added to the system, the expansion takes place with an increase in entropy to state 3. The change in the specific entropy of the system, due to the addition of heat, is

$$\Delta s = \frac{Q}{mT} .$$

At state 3, the entropy of the individual phases is equal to the total entropy of the system and the total mass equal to the sum of the masses of the individual phases, so that

$$s_{\ell t} m_{\ell 3} + s_{vt} m_{v3} + s_{st} m_{s3} = m s_2 + \frac{Q}{T} , \qquad \text{(A13)}$$

$$m_{\ell 3} + m_{v3} + m_{s3} = m , \qquad \text{(A14)}$$

and the quality F is defined as

$$\frac{m_s}{m_s + m_\ell} = F . \qquad \text{(A15)}$$

In order to separate the calculations for process 2 - 3, from those for process 1 - 2, assume state 2 is pure liquid at the triple point. Then s_2 becomes $s_{\ell t}$ and

$$\frac{m_{v3}}{m} = \frac{F\left(s_{\ell t} - s_{st}\right) + \frac{Q}{mT}}{\left(1 - F\right)\left(s_{vt} - s_{\ell t}\right) + F\left(s_{vt} - s_{st}\right)} . \qquad \text{(A16)}$$

Figure 12 gives the mass of vapor m_v/m vs. the quality F.

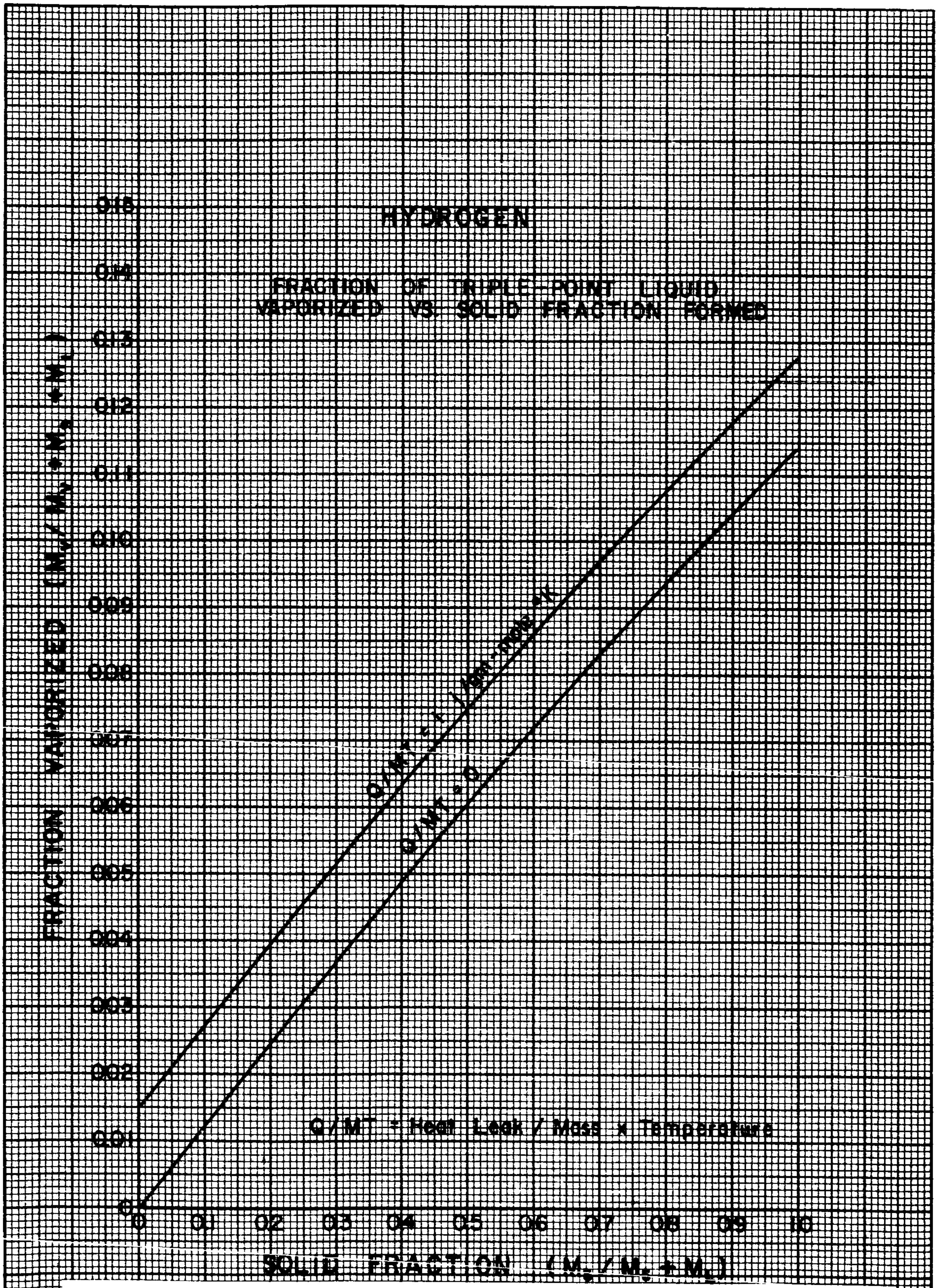


Figure 12. Fraction of Triple-Point Liquid Vaporized vs. Quality

The quantity of vapor calculated by equation (A16) is the total amount of vapor formed, not the amount that would be removed from a container such as a dewar. A volume of vapor equal to the decrease in the volume of the liquid and solid phases remains in the container. The error resulting from the use of equation (A16) is approximately equal to ratio of the specific volumes of the liquid to vapor phase $\frac{v_{\ell t}}{v_{vt}}$. For hydrogen, $\frac{v_{\ell t}}{v_{vt}}$ is 1/608. Unless accuracies better than 1/2 percent are desired, equation (A16) may be used without correction.

11.1.3. Experimental determination of the liquid-solid quality

Two methods will be used to experimentally determine the quality of a mixture of liquid and solid hydrogen. Both methods can be carried out as successive operations in the same apparatus. Figure 13 is a schematic diagram of the experimental arrangement.

11.1.3.1. Volumetric method

When only the liquid and solid phases of a triple-point mixture are considered, the density of the two phases depends only on the quality. This provides a means for determining the quality which depends only on mass and volume measurements. The measurements required are as follows:

1. The mass of the liquid hydrogen at zero quality is determined by measuring the level of the liquid with a cathetometer after the dewar has been volume calibrated.
2. The decrease in the mass of the liquid-solid mixture is determined by measuring the volume of gas leaving the dewar with a flowmeter.

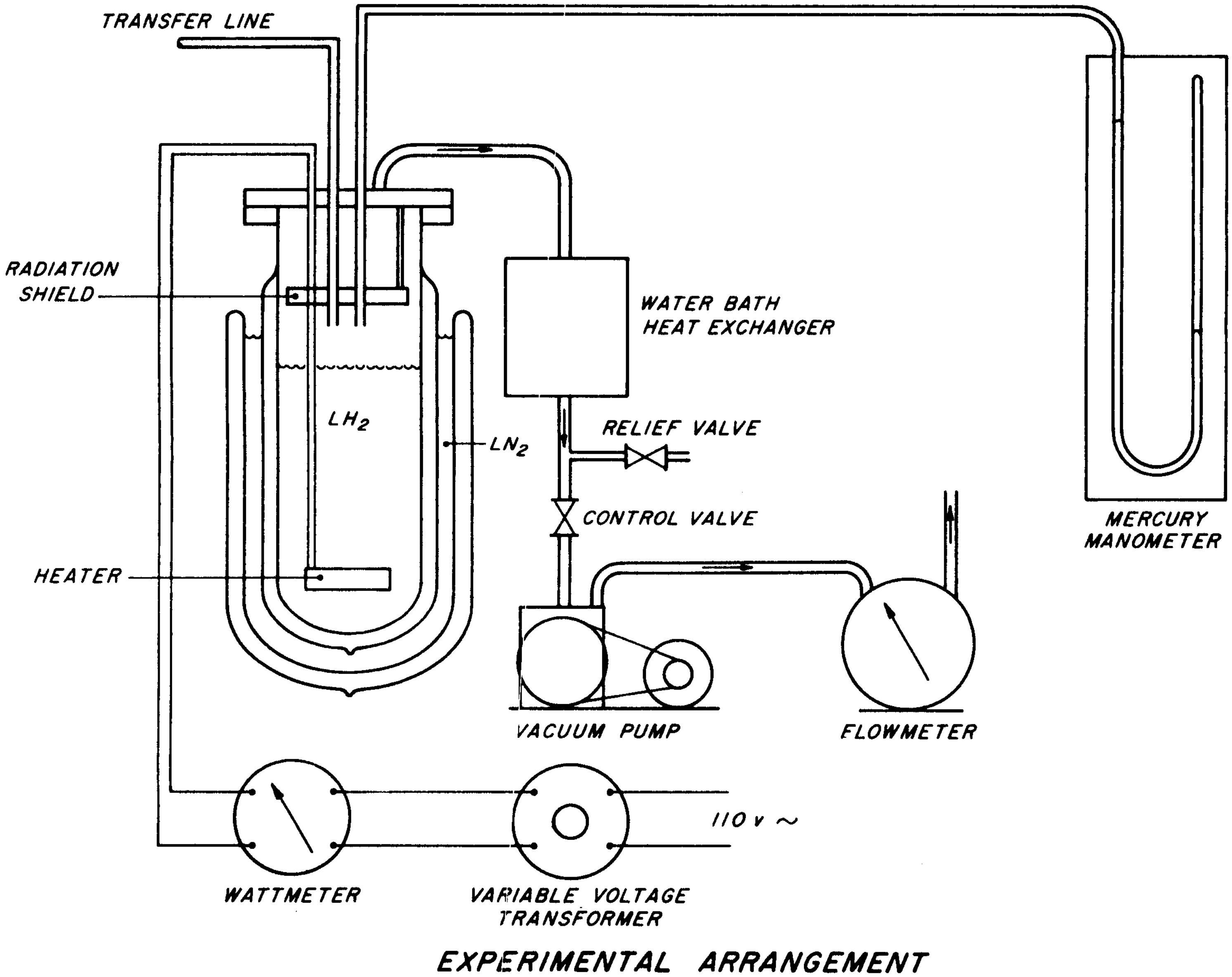


Figure 13. Quality Determination Apparatus

3. The change in volume of the mixture in going from zero quality to the quality F is determined by measuring the change in the level in the dewar with a cathetometer.

The quality F is then determined from the expression

$$F = \frac{\Delta V + v_{\ell} m_v}{\left(v_s - v_{\ell}\right)\left(m - m_v\right)} , \tag{A17}$$

where m is the initial mass, m_v the mass of vapor removed, and ΔV the change in volume of the mixture.

11.1.3.2. Calorimetric method

After the measurement of the quality by the volumetric method, the heater will be turned on until the solid just disappears, the heat being measured by a wattmeter. The process follows the path 3 - 4 on figure 11. The quality of the liquid-solid mixture at the state 3 can then be determined from the expression

$$F = \frac{\dfrac{Q}{mT} - \dfrac{m_{v4}}{m}\left(s_{vt} - s_{\ell t}\right)}{s_{\ell t} - s_{st}} , \tag{A18}$$

where m_{v4} is the mass of the vapor pumped off during the heating, m is the mass at state 3 and Q is the heat introduced by the heater plus the heat leak. It should not be necessary to pump on the dewar during the process, so m_{v4} would be zero and m would remain constant from 3 to 4.

Equation (A16) evaluated for F = 0 gives

$$\frac{Q}{m_{v3} T} = s_{vt} - s_{\ell t} . \tag{A19}$$

If Q is taken as the total heat introduced since the beginning of the solid formation, and m_{v3} is the total amount of vapor given off, then the equation (A19) should hold. If values of $s_{vt} - s_{\ell t}$ obtained from equation (A19) differ consistently from the accepted values, then the values obtained from equation (A19) will probably give the best estimates of F when used in equation (A16).

It is possible to estimate the liquid-solid quality by measuring the total mass of vapor removed from the system in going from state 1 to state 3 through the use of equations (A8), (A 11), and (A16). However, if the arrival at state 2, the edge of the triple-point region, is established by measuring the temperature of the liquid, and/or visual observation for the presence of solid, then the uncertainties in the estimation of the quality F should be reduced. By measuring the vapor pressure in the dewar with a mercury manometer, the temperature may be determined from established values of vapor pressure versus temperature at saturation.

11.2 Experimental data

2/21/66-Run A

Barometer	632.9 mm Hg
Level of dewar top plate	731.40 mm

Heat leak test

Time for 10 liters of gas to boil off	5 min. 44 sec.

Pump down to the triple point

Flowmeter (start)	0000.0
Liquid level (start)	239.14 mm
Dewar pressure (start) - gauge	103 mm Hg
Flowmeter temperature	73°F
Flowmeter pressure (gauge)	10 mm water
Flowmeter (finish)	844.5 liters
Liquid level (finish)	168.14 mm
Elapsed time	15 min.

Solid formation	Run #1	Run #2	Run #3
Flowmeter - finish (liters)	1032.9	1177.5	1364.9
Flowmeter temperature (°F)	73	73.5	74
Flowmeter pressure (mm water)	2	2	2
Liquid level (finish) (mm)	152.48	147.60	137.04
Elasped time (minutes)	26	43	61
Fraction of settled slush	3/4	2/3	7/8
Solid Melting			
Volts	32.8	31.1	28.5
Amps	0.462	0.438	0,405
Heater time (total) (minutes:seconds)	5:5	9:31	15:53
Liquid level	159.21	152.20	143.74
Elapsed time (minutes)	34	50	72

2/21/66-Run B

Barometer	632.4 mm Hg
Level of dewar top plate	731.40 mm

Pump down to the triple point

Flowmeter (start)	000.0
Liquid level (start)	289.05 mm
Dewar pressure	95 mm Hg
Flowmeter temperature	74.5°F
Flowmeter pressure (gauge)	10 mm water
Flowmeter (finish)	940.3 liters
Liquid level (finish)	211.89
Elapsed time	14 min.

Solid formation	Run #1	Run #2	Run #3	Run #4
Flowmeter - finish (liters)	1124.2	1290.5	1447.0	1673.7
Flowmeter temp. (°F)	74.5	74.5	74.5	74.5
Flowmeter pres. (mm H_2O)	2	2	2	2
Liquid level (finish) (mm)	197.28	190.28	183.52	170.66
Elapsed time (minutes)	24	44	62	81
Fraction of settled slush	3/4	2/3	2/3	7/8
Solid melting				
Volts	29.2	29.3	29.2	29.5
Amps	0.413	0.413	0.413	0.418
Heater time (min:sec)	6:15	11:43	17:3	24:36
Liquid level (mm)	203.82	195.95	188.80	178.22
Elapsed time (minutes)	33	52	70	91

2/23/66

Barometer	629.9 mm Hg
Level of dewar top plate	731.40

Pump down to the triple point

Flowmeter (start)	000.0
Liquid level (start)	315.55
Dewar pressure (start) - gauge	101 mm Hg
Flowmeter temperature	69.5
Flowmeter pressure - gauge	10 mm water
Flowmeter (finish)	965.6
Liquid level	234.95
Elapsed time	14.5 min.

Solid formation	Run #1	Run #2	Run #3	Run #4
Flowmeter - finish (liters)	1219.1	1751.1	2190.2	2439.4
Flowmeter temp. (°F)	70.5	73.0	73.6	73.5
Flowmeter pres. (mm H_2O)	2	2	2	2
Liquid level (finish) (mm)	209.32	182.84	164.32	158.20
Elapsed time (minutes)	35	77	120	150
Fraction of settled slush	3/4	19/20	19/20	3/4
Solid melting				
Volts	29.2	29.3	29.5	29.5
Amps	0.413	0.414	0.416	0.416
Heater time (min:sec)	10:28	25:32	39:40	48:18
Liquid level (mm)	219.75	198.14	178.88	167.65
Elapsed time (minutes)	48	94	137	161

Solid formation	Run #5	Run #6	Run #7	Run #8
Flowmeter - finish (liters)	2592.5	2689.6	2790.7	2870.9
Flowmeter temp. (°F)	74.2	74.2	74.1	74
Flowmeter pres. (mm H_2O)	2	2	2	2
Liquid level (finish) (mm)	155.10	152.72	148.58	145.60
Elapsed time (minutes)	170	183	195	206
Fraction of settled slush	2/3	1/2	1/2	1/2
Solid melting				
Volts	29.2		29.4	29.5
Amps	0.413		0.415	0.416
Heater time (min:sec)	53:52		3:17	5:52
Liquid level (mm)	160.75	156.48	151.65	148.45
Elapsed time (minutes)	177	188	200.5	211

Solid formation	Run #9	Run #10
Flowmeter - finish (liters)	2950.2	3070.6
Flowmeter temp. (°F)	74.5	74.5
Flowmeter pres. (mm H_2O)	2	2
Liquid level (finish) (mm)	141.73	134.71
Elapsed time (minutes)	216	228
Fraction of settled slush	1/2	1/2
Solid melting		
Volts	29.3	29.5
Amps	0.413	0.414
Heater time (min:sec)	8:27	12:22
Liquid level (mm)	144.60	138.62
Elapsed time (minutes)	222	235

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