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An Experimental Study Concerning the Pressurization and Stratification of Liquid Hydrogen [1, 2]

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(December 19, 1960)

A 625 gallon powder-insulated Dewar has been built and appropriately instrumented to provide information concerning pressurization gas consumption, ullage pressure, liquid level (ullage volume, indirectly), horizontal and vertical temperature surveys throughout the test fluid, and lapsed time. Pressurization levels of 25, 75, 125, and 180 psig have been examined individually. Two techniques—one based on the case of one-dimensional heat flow in a semi-infinite solid and the other founded on an electrical network analog of the thermal system—are given for making reasonable predictions concerning liquid-phase hydrogen temperature distribution in well-insulated, rapidly-pressurized storage vessels. A thermal analysis is presented which indicates the mechanics of heat transmission and adsorption in the fluid.

1. Introduction

The so-called temperature stratification phenomenon, which has been observed in containers of cryogenic fluids under certain conditions, is of sufficient interest in the field of cryogenic engineering to warrant special investigation. Areas in which the results of such research may be applied profitably include those concerning the transfer of liquefied gases by pressurization, the transfer of liquefied gases by pumping, and the sealed storage of liquefied gases. In the first case, the stratification of contents in a vessel used to supply transfer fluid permits the utilization of subcooled liquid, the properties of which are desirable from the aspect of single-phase flow characteristics. In the second case, stratification provides a means to attain, and sustain, Net Positive Suction Head for transfer by pumping, the absolute magnitude of NPSH required here depending upon both fluid and pump characteristics and the geometry of the pumping situation. For the final case, the effect of stratification is undesirable, generally, due to the accompanying promotion of an excessive rate of pressure rise in the storage vessel ullage space; the undesirability is pronounced for sealed "no-loss" transport containers in which the maximum time permissible in transit is a direct function of the container design pressure.

2. Experimental Equipment

An elevation assembly drawing of the 625 gallon Dewar used in this study is presented in figure 1. The inner vessel is supported within the outer shell by means of a 15 in. diam stainless steel cylinder which is fastened between central portions of the two top dished heads as shown in the drawing. The diameter of this cylinder was chosen to provide a manway into the test container for relatively easy access to the internal instrumentation. Evacuated perlite is the insulating medium, and filtered vacuum pump connections are located on both the top and the bottom dished heads; flanges attached directly to the base of each evacuation port permit filling and draining of the insulation space. A static Dewar vacuum of 10 to 15 μ is achieved in the cold, hydrogen-filled Dewar.

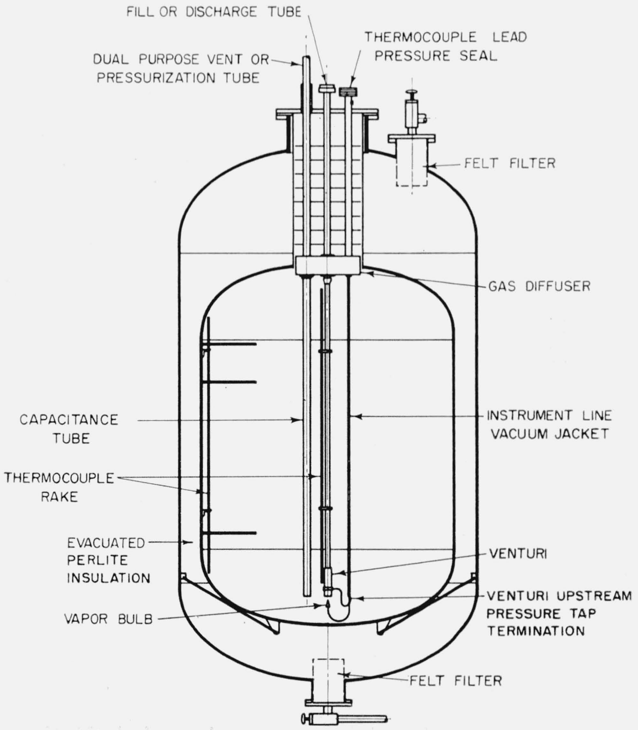

FIGURE 1. *625 gallon test Dewar.*

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[1] This work was supported by the U.S. Atomic Energy Commission.
[2] This paper is an expanded version of a paper presented at the 1959 Cryogenic Engineering Conference, Berkeley, Calif., September 4, 1959.
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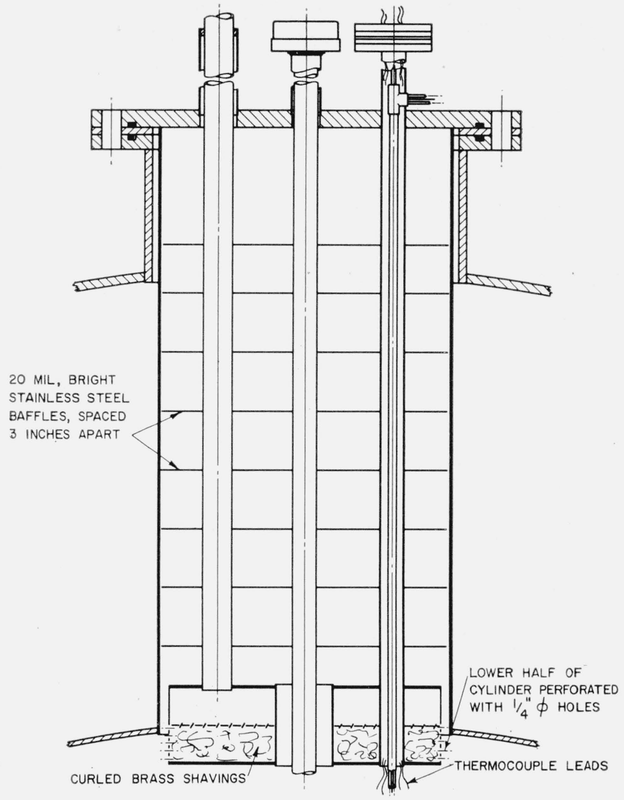

FIGURE 2. *625 gallon test Dewar-manway section*

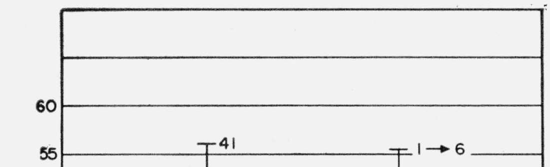

FIGURE 3. *Position of thermocouples in vertical direction 625 gallon Dewar*

Essentially all critical test components are attached to the manway cover, thereby providing an orderly means for placing, removing, and examining internal equipment, when necessary, with a minimum of difficulty (fig. 2). Horizontal, bright stainless steel baffles are soldered to the fill, vent, and instrument lead tubes at regular intervals within the access cylinder to minimize hydrogen gas circulation and thermal radiation in this region. One common method of insulating a zone such as this, which may be exposed to temperatures approximating 300 °K on one side and something less than 100 °K on the other, is to build an evacuated-powder or high-vacuum insulated plug with a cylindrical stainless steel wall. However, in the case of a moderate or high-pressure rated container, the plug must be built to withstand vessel design pressure—implying relatively heavy plug walls. As a consequence of the thermal conduction characteristics of various materials involved, the hydrogen gas-baffle arrangement provides a better heat barrier across the manway than would a plug of adequate pressure rating. For this reason, and for the sake of simplicity and economy, the baffle construction was used. It may be of interest to note that the thermal conductivity of hydrogen gas is approximately one hundred times that of evacuated powder and one-hundredth that of stainless steel.

Located immediately beneath the bottom opening of the access cylinder is a gas diffuser which diverts the incoming pressurization gas 90° through curled brass shavings and introduces the stream tangentially to the underside of the inner shell top dished head. The diffuser is fed by a 1½ in. tube which also serves as a gas vent when the test vessel is being filled or depressurized.

A capacitance probe, hanging from the bottom of the diffuser, is used to provide the liquid contents (level) determination—the signal of the probe being furnished to a direct-reading recorder. Auxiliary level information may be obtained by observing the Dewar ullage pressure, by then noting the saturation temperature corresponding to this pressure on a hydrogen vapor pressure-temperature diagram, and finally by locating this temperature in data provided by the vertical thermocouple rake. Knowledge of the liquid surface location derived by the latter method has proved to be quite precise in the zone of closely-grouped thermocouples; in other regions, the precision is understandably lacking.

Further examination of figure 1 reveals a Venturi tube which is attached to the inlet of the fluid discharge tube to permit evaluation of such a device in cryogenic media. Results of this work are presented in another paper.[4]

In the experimental program, two thermocouple rakes are utilized—one installed vertically in the center of the vessel, and the other fastened to the side wall, capable of sensing horizontal temperature profiles from zero to 11 in. from the wall at three distinct levels (approximately 17, 49, and 56 in. from

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[4] J. R. Purcell, A. F. Schmidt, R. B. Jacobs, The Venturi tube as a liquefied gas flow measuring device, Advances in Cryogenic Engineering, **5**, pp. 282 to 288 (Plenum Press, New York, N.Y., 1960).

the tank bottom, respectively). Figure 3 illustrates the placement of each thermocouple in the vertical direction.

The system of differential thermocouples—59 in all—is composed of one 36 gage gold-cobalt lead and an appropriate number of 36 gage copper leads, each of which passes through a rubber compression-type seal at the top of the tank. A reference junction for the entire series of 'couples is attached to a small parahydrogen-filled vapor-pressure bulb located below the inlet of the Venturi; reference temperature information, therefore, is derived from bulb pressure data and a parahydrogen vapor pressure-temperature curve. Temperatures, corresponding to the vapor bulb pressure readings, are accurate to ±0.05 °K.

Hydrogen pressurization gas is stored at high pressure in a standard tube trailer of 260 ft^3 free volume, from which it passes through a preset pressure-recorder-actuated solenoid valve and on into the Dewar during pressurization.

3. Test Procedure and Results

In an effort to present the data accumulated from each pressurization run most descriptively, tests A–2, A–3, A–4, and B–1 have been analyzed graphically through temperature-time and temperature-height curves (fig. 4 to 9). Test identification symbols listed above refer to the conditions of table 1.

Tests A–1 through A–4 were conducted in the following manner: The test vessel liquid inlet connection was sealed off, and the container was maintained at ¾ psig for a minimum of 16 hr by venting into a hydrogen gasholder. At the end of this period (actually the start of the test day), during which time the fluid contents had reached an equilibrium state, a complete set of thermocouple readings was taken and recorded as the initial condition. The vent-to-gasholder valve was then closed, a pressure-recorder-actuated solenoid valve between the hydrogen gas tube trailer and the test container gas diffuser was opened and pressurization of the Dewar to a preselected level was effected within 30 sec. Additional small quantities of gas were generally required for 15 min after the initial pressurization in order to maintain the desired pressure level, after which time the liquid evaporation rate became more than sufficient to furnish the needed gas. Rather than disturb the temperature gradients being established in the test space by continuously releasing

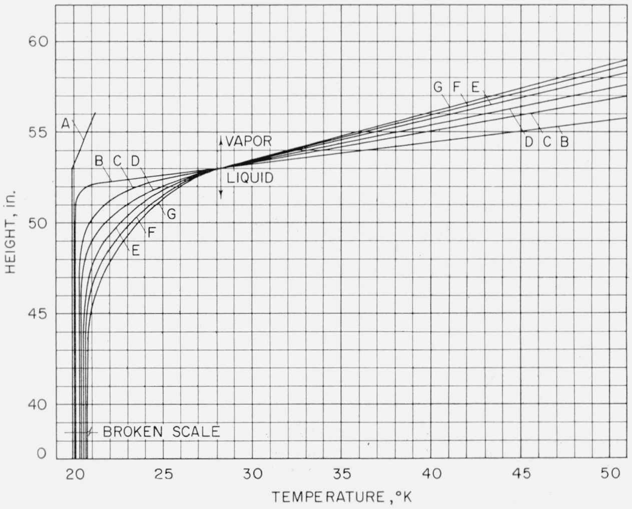

FIGURE 4. *Temperature versus height, hydrogen test A-2, 75 psig.*

A, O+ time
B, O+¼ hr
C, O+1¾ hr
D, O+3¼ hr
E, O+4¾ hr
F, O+6¼ hr
G, O+7¾ hr

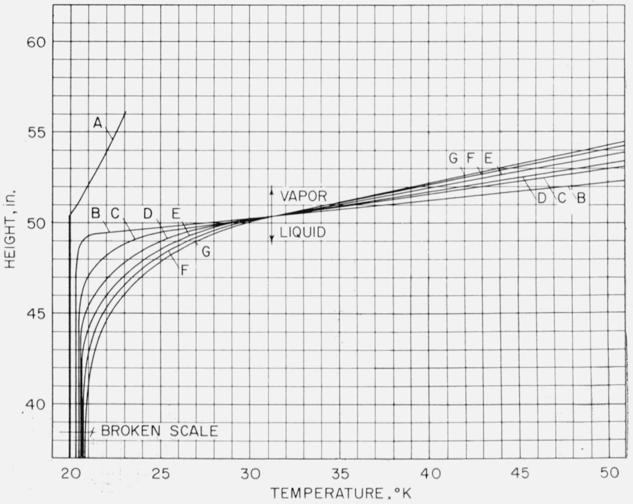

FIGURE 5. *Temperature versus height, hydrogen test A–3, 125 psig.*

A, O+ time
B, O+¼ hr
C, O+1¾ hr
D, O+3¼ hr
E, O+4¾ hr
F, O+6¼ hr
G, O+7¾ hr

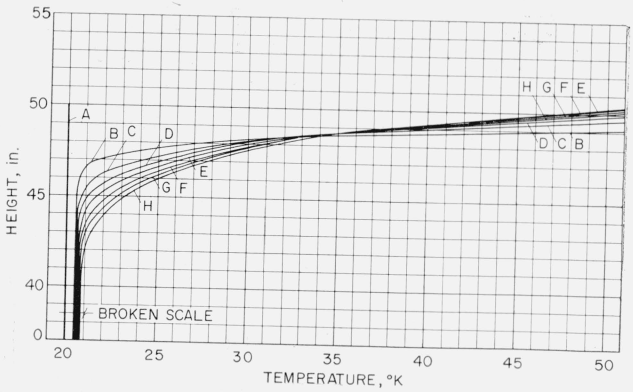

FIGURE 6. *Temperature versus height, hydrogen test A–4, 180 psig.*

A, O+ time
B, O+¼ hr
C, O+¾ hr
D, O+1¼ hr
E, O+1¾ hr
F, O+2¼ hr
G, O+2¾ hr
H, O+3¼ hr

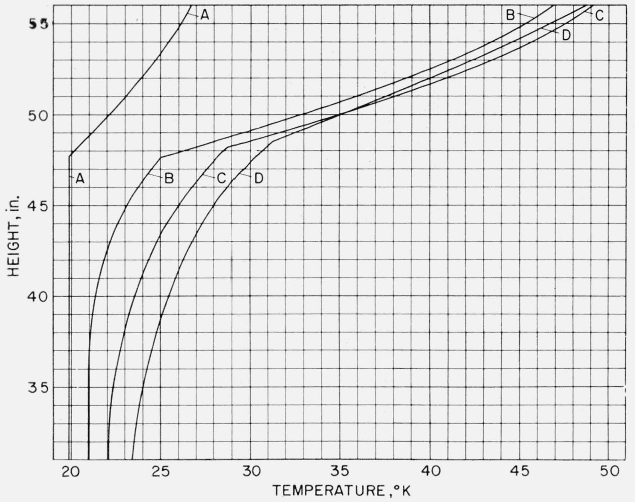

FIGURE 7. *Temperature versus height, hydrogen test B–1, pressure build-up due to normal heat leak.*

A, O+ time
B, O+24 hr
C, O+48 hr
D, O+72 hr

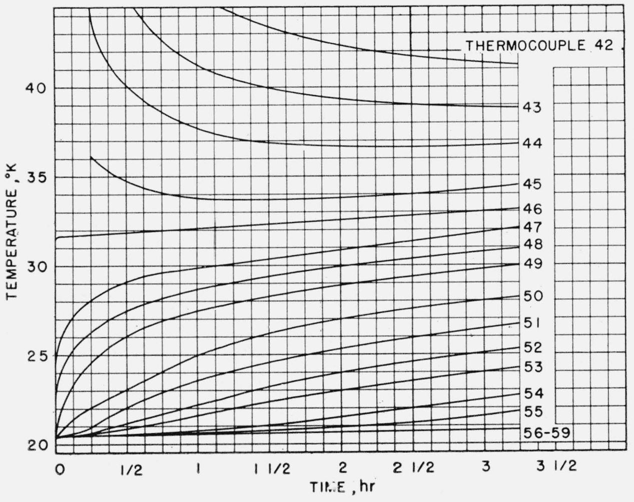

FIGURE 8. *Temperature versus time, hydrogen test A–4, 180 psig.*

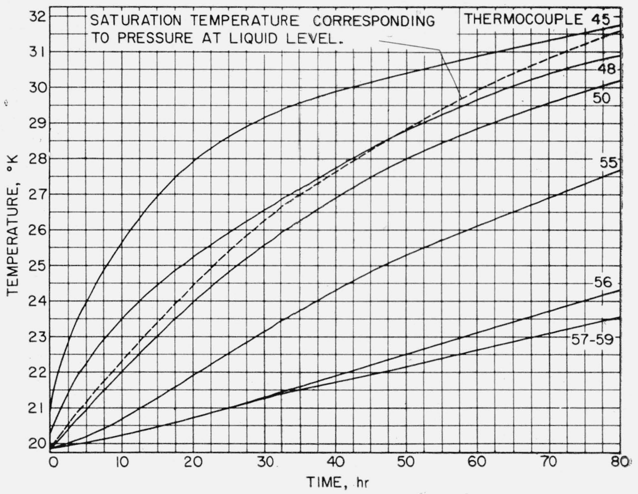

FIGURE 9. *Temperature versus time, hydrogen test B–1, pressure build-up due to normal heat leak.*

the gas overpressure, the vent was kept closed and the pressure was permitted to rise naturally throughout the test period. Thermocouple and vapor pressure data were recorded every ½ hr for 8 hr. Termination of the test after 8 hr was accomplished simply by deactivating the recorder-actuated solenoid valve and depressurizing the tank to gasholder conditions for overnight standby. Gas consumed in each of the tests listed above was measured by opening enough tubes in the gas trailer manifold to exceed, by a small margin, a precalculated demand; the temperature and pressure of this gas were then recorded, pressurization of the test Dewar occurred, and the final trailer manifold temperature and pressure were noted. Knowledge of the fluid capacity of each tube in the trailer, coupled with the pressure-temperature data recorded above, then provided information concerning the amount of pressurizing gas required in each test.

Pressure build-up test B–1 was effected by completely sealing off the experimental chamber and recording the necessary data three times a day over an 80 hr test run.

TABLE 1.

Test No.	Initial pressure (zero time)	Test pressure [a] (zero time plus ½ min)	Final pressure [b] (zero time plus 7¾ hr)	Pressurization gas consumption	Liquid volume	Ullage volume (including manway volume)
	psig	*psig*	*psig*	*scf*	*cf*	*cf*
A–1	¾	25	46	110	67.2	20.3
A–2	¾	75	102	310	63.2	24.3
A–3	¾	125	160	465	59.7	27.8
A–4	¾	180	(195) [c]	700	57.3	30.2
B–1	¾	¾	10	----	56.3	31.2

[a] Test pressure refers to level achieved with tube trailer pressurization gas.

[b] Final pressure refers to level achieved at completion of test (pressure resulting from combination of tube trailer gas and gas evolved during test period).

[c] Final pressure after 3¼ hr.

4. Discussion

Data obtained from the program described here were studied and used in several different ways:

(1) Pressurization gas consumption data were recorded and several attempts were made to correlate this information; an insufficient number of vapor-phase thermocouples plus the additional complication of irregular vessel geometry due to the manway volume discouraged extensive work in this area;

(2) Considering system geometry and certain properties of the test fluid, metal containing wall and evacuated-powder insulation, a thermal analysis was made to determine the mechanics of heat transmission and absorption in the fluid;

(3) A method useful in predicting the change of temperature with time at any given point in a solid was applied to the experimental quantity of liquid hydrogen—the theoretical results were compared with experimental information;

(4) An electrical network analog of the thermal system was constructed and a comparison with the theoretical method (3) was made.

4.1. Pressurization Gas Consumption

Information pertaining to the consumption of pressurization gas, pressure level and ullage volume in this series of tests is presented in table 1. One interesting observation can be made from these data. For a perfect gas—using Boyle's Law ($pV=p'V'$)—the amount of gas required to elevate the pressure p in a known volume V' to a specified level p' may be calculated (assuming no condensation, evaporation, etc.). Results of the four tests reported here indicate that the pressurization gas consumed in any test can be predicted to an accuracy of 10 percent by first using the perfect gas relationship to compute a required volume V of gas at standard conditions, then doubling the answer. To restate, the amount of gas needed to pressurize a vessel of this particular geometry is approximately twice that computed from Boyle's Law. Gas temperature, pressurization time and diffuser configuration should also be considered in the final analysis. Although the correlation is crude, it may be of some value in estimating the requirements of subsequent work of this nature.

4.2. Thermal Analysis

Heat transmission into the liquid hydrogen was considered to occur in the following manner:

(1) The uniform (bulk) temperature portion of the liquid is warmed by heat transferred from the containing surface and thence by convection;

(2) The stratified liquid layer is warmed by heat conducted from vapor above the interface and by condensation at the interface;

(3) Vaporization occurs at the outer periphery of the interface where liquid contacts the warmer metal.

Validity of these assumptions may be tested by heat balances using internal energy and apparent heat transfer information. Taking experimental data from the last hour and a half of test A–3 (fig. 5) as an example, calculations indicate a total internal energy change rate of 53.8 w:

Internal Energy Change Rate

In uniform temperature portion of liquid (below 38″)	17. 5 w
In stratified portion of liquid (above 38″ to 50.3″)	24. 0 w
To raise mass of liquid to be vaporized to saturation temperature	6. 5 w
To raise mass of liquid to be vaporized from saturated liquid to final vapor state	22. 3 w
	70. 3 w
To cool initial mass of vapor to final state	−16. 5 w
	53. 8 w

In determining apparent heat transfer through the evacuated-powder tank insulation and down the manway, a value of insulation conductivity ($k=40$ μw/cm °K) was chosen such that the heat transmission computed from Dewar geometry (below) equalled the computed rate of change in internal energy (above). The reason for choosing a value of k in the manner described relates back to some uncertainty existing in the absolute value of this property, even with a fair knowledge of material substance, size and interstitial pressure. The value computed is within the range of values considered reasonable for the material.

Heat Transfer Rate

Through bottom head of test vessel	10.7 w
Through cylindrical wall of uniform temperature portion of liquid (below 38″)	14.2 w
Through cylindrical wall of stratified portion of liquid (above 38″ to 50.3″)	7.3 w
Through vessel wall surrounding vapor phase (above 50.3″)	8.1 w
Through manway (hydrogen gas and metal neck)	13.5 w
	53.8 w

Checking premise (1), *The uniform (bulk) temperature portion of the liquid is warmed by heat transferred from the containing surface, and thence by convection:*

$$\text{Internal energy change rate} \approx \text{Heat transfer rate}$$
$$17.5\ \text{w} \approx 24.9\ \text{w}$$

Then with respect to the stratified layer, an assumption is made that heat transferred through the wall surrounding this portion of fluid provides the energy required to raise the temperature of the liquid, which is to be vaporized, from the bulk temperature to a saturated condition.

$$\text{Internal energy change rate} \approx \text{Heat transfer rate}$$
$$6.5\ \text{w} \approx 7.3\ \text{w}$$

An apparent surplus of transferred heat ($7.4+0.8=8.2$ w), from the two zones (or regions) considered, may be assumed to contribute to vaporization at the periphery interface.

From premise (2), *The stratified liquid layer is warmed by heat conducted from vapor above the interface and by condensation at the interface:*

$$\underset{\text{(interface)}}{\text{Condensation rate}} \approx \underset{\text{(stratified layer)}}{\text{Internal energy change rate}} - \underset{\text{(vapor to liquid)}}{\text{Heat conduction rate}}$$

$$\approx 24.0\text{ w} \qquad -5.5\text{ w}$$

$$\approx 18.5\text{ w}$$

where the heat conduction rate is computed from the gas phase portion of curves F and G (test A–3) near the interface.

To complete the account of heat distribution in the Dewar, from interface up, gas phase temperature and pressure information over the chosen hour and one-half test interval is used to calculate a system mass (gas) increase. This Δm is then multiplied by the latent heat of vaporization of hydrogen, resulting in a net vaporization energy value of 8.5 w. Total vaporization rate, therefore, is equal to the condensation rate computed previously plus the net vaporization rate computed above, or

$$\text{Total vaporization rate} = 18.5 + 8.5 = 27.0\text{ w}$$

This rate, plus the 5.5 w conducted through the vapor to the interface, must be supplied from above the interface and from the apparent surplus beneath the interface.

4.3. Semi-Infinite Solid

The experimental volume of liquid hydrogen has been treated as a semi-infinite solid with the liquid-vapor interface representing a constant temperature bounding face. Since equilibrium temperatures are observed throughout the test fluid prior to pressurization, and since the plane surface temperature immediately after pressurization is also known (saturation temperature corresponding to interfacial pressure), the uniform temperature of the liquid and the suddenly-changed surface temperature are known. With this information, and certain physical fluid properties, the case of one-dimensional heat flow in a semi-infinite solid may be calculated.

A solution for the temperature history $T(x, \theta)$ must satisfy the partial-differential equation of Fourier,

$$\frac{\partial T}{\partial \theta} = \alpha \frac{\partial^2 T}{\partial x^2}.$$

By imposing appropriate initial and boundary conditions and proceeding through the necessary mathematical sequence, the desired relationship is found to be

$$\frac{t - t_1}{t_i - t_1} = \frac{2}{\sqrt{\pi}} \int_0^{x/2\sqrt{\alpha\theta}} e^{-\lambda^2} d\lambda$$

where

t = temperature at distance x below liquid-vapor interface,

t_1 = saturation temperature corresponding to pressure at interface,

t_i = initial fluid equilibrium temperature,

x = distance below interface at temperature t,

θ = time,

α = thermal diffusivity, $= k/\rho C_p$

k = thermal conductivity,

ρ = density,

C_p = specific heat at constant pressure,

$\frac{2}{\sqrt{\pi}} \int_0^{x/2\sqrt{\alpha\theta}} e^{-\lambda^2} d\lambda$ = error function, or probability integral.

A primary decision one faces in working out the mechanics of this problem concerns a somewhat arbitrary choice in the value of thermal diffusivity. Clearly, it must be evaluated at some temperature between t_i and t_1. For the case of liquid hydrogen, the variation in α is not considerable—the value at 20 °K being approximately one-third higher than that at 31 °K; further, it is $\sqrt{\alpha}$ that is used in this computation. With this knowledge then, and the additional stipulation that C_p be used as heat capacity in the diffusivity determination, one may choose essentially any value of α between t_i and t_1, run through the simple mathematics of the problem and arrive at an excellent approximation of fluid temperature distribution. In most cases, the accuracy associated with such a method of choosing α is more than sufficient. If, however, it is desired to refine the calculation, the initial approximation (above) may be used to provide basic temperature versus distance curves, from which any value, or values, of α can be selected for the final computation.

This method of theoretically simulating the actual physical conditions described previously is not without limitation when applied to liquefied gases of small latent heat of vaporization and low temperature. An efficient thermal insulation was used to envelop the test container in this work, thereby permitting relatively little heat to flow into the fluid. Should the liquid be submitted to a progressively higher heat flux, degradation of the established temperature gradients would be noted at some increased value of this flux due to greater boiling, and hence agitation, of the liquid. A poorly insulated volume of liquid, fluid containers utilizing high-thermal conductivity materials of construction in shell walls, liners, baffles, etc., and Dewars in transit, may not be adapted to this type of analysis.

As implied in preceding paragraphs, the representation of a quantity of liquefied gas by a semi-infinite solid is interesting and apparently feasible. The method has been applied to the reported experimental data with excellent results. Test data were not completely analogous in that the bulk fluid temperature shifted slightly with time, due to the combined effects of heat transmitted into the liquid from the containing wall and compression of the liquid, and also because of the fact that the interface temperature shifted somewhat during the test period as a result of the pressure increase in the nonvented Dewar.

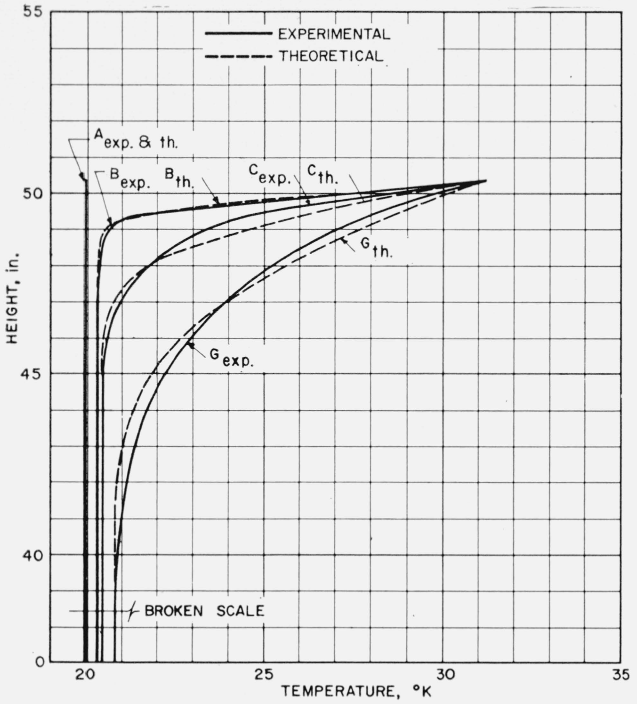

Figure 10. *Temperature versus height, hydrogen test A–3, 125 psig.*

A, 0+ time
B, 0+¼ hr
C, 0+1¾ hr
G, 0+7¾ hr

However, these deviations had only a secondary effect on the problem. A comparison of theoretical and experimental values for the 125 psig test (A–3) reveals the following information (see fig. 10): Using an interface temperature of 30.8 °K and a liquid bulk temperature of 20.3 °K, a temperature difference of 10.5 °K has been established. For distances greater than 3 in. below the surface of the liquid, and for any time between zero and 8 hr, the *maximum* deviation between theoretical and experimental work is 0.3 °K, or 3 percent of the temperature span, while the average deviation is less than 0.1 °K. The first 3 in. beneath the surface are affected more by an increase in interface (saturation) temperature with time and by the slight increase in volume of the liquid, so that a direct comparison here is likely to show the greatest error. Even so, the maximum error is seen to be less than 1.5 °K.

4.4. Electrical Network Analog

In order to evaluate more directly the effect of bulk and surface temperature shift previously mentioned, some time was devoted to the use of an electrical analog as a means of most conveniently examining this shift. Also, the ability to physically observe experimental phenomena requiring 8 hr being simulated electrically in a few seconds is of considerable interest.

The electrical analog is based on the equation $\frac{\partial V}{\partial \theta}=\frac{1}{RC}\frac{\partial^2 V}{\partial x^2}$ (compare with the Fourier equation $\frac{\partial T}{\partial \theta}=a\frac{\partial^2 T}{\partial x^2}$ previously mentioned), if distributed constants are used in the circuitry. Using lumped constants—that is, conventional resistors and capacitors—the error is slight if reasonable consideration is given to circuit construction. It is seen from the above equations that voltage is analogous to temperature in the thermal circuit and that the electrical diffusivity 1/RC corresponds to the thermal diffusivity α. In the overall circuit, it is not necessary to define resistance and capacitance individually—only the product which determines the time constant and thus the ratio α/1/RC, or αRC. This ratio is generally quite large (approximately 20,000 in the present case) due to practical limitations of the circuit. However, the advantage of such a circuit is understandable in that the curves are plotted very rapidly.

Results obtained by this technique were exactly comparable with those achieved by the method of the semi-infinite solid. Unfortunately, circumstances did not permit further refinement of the circuit to provide for the gradual shift of bulk and surface temperatures with time; it is hoped that this may be accomplished in the near future.

5. Conclusions

It is evident from the preceding paragraphs that reasonable predictions can be made concerning liquid-phase hydrogen temperature distribution in well-insulated, rapidly-pressurized storage vessels.

By treating the liquid as a semi-infinite solid, one may foretell the temperature history, temperature gradient and heating rate of the stratified fluid portion beneath the liquid-vapor interface; where numerous succeeding initial and boundary conditions are to be imposed in the manner of trial and error, where these conditions are to vary with time for any one given situation, or where a variation of thermal properties must be taken into account, the electrical analog method may be modified to permit more rapid solutions.

Additionally, if heat flux through the containing vessel wall is known, bulk liquid temperature rise may be predicted in a very rough manner.

Finally, it will be noted by the curves presented here, and by theory, that an increase in ullage pressure—hence an increase of interfacial temperature—results in the transfer of more heat to the stratified layer, the quantity of which is calculable using the method of the semi-infinite solid. Knowledge of this may serve as one criterion in the optimization of pressure level within a liquefied gas storage or transfer system.

(Paper 65C2–58)