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Initial Warmup of 500,000-Gallon
Liquid Hydrogen Dewar

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INITIAL WARMUP OF 500,000-GALLON LIQUID HYDROGEN DEWAR

by

D. H. Liebenberg and E. Murley

ABSTRACT

Two 500,000-gal liquid hydrogen dewars were installed as a part of the Test Cell C modification at NRDS. Included in the Los Alamos Scientific Laboratory design was a four-section, 32.3-kW electric heater.

Since no dewar of this size has been warmed by electric heaters before, this report has been prepared as a guide for future warmups. It will hopefully be of general interest to designers of large liquid hydrogen dewars concerned with their periodic warmup.

The dewar warmup was started on December 16, 1966. The electric heaters were turned on on December 19, and the warmup was completed on December 22. The internal temperature at completion was a minimum of 493°R, and the dewar could be entered without fear of water vapor contamination from the winter air at NRDS.

INTRODUCTION

The two 500,000-gal liquid hydrogen dewars at Test Cell C were installed by the Chicago Bridge and Iron Company and include instrumentation designed by Los Alamos Scientific Laboratory. An electric heater, previously tested in smaller dewars, was installed to provide rapid warmup of the dewars, in keeping with the developmental nature of the reactor testing planned at this test cell.

Each 500,000-gal dewar has an inner shell of 304 stainless steel, the plates averaging 0.74-in. thick. This shell has a warm diameter of 52.05 ft, a cold diameter of 51.84 ft, and a total volume of 546,000 gal at liquid hydrogen temperatures. The inner shell is suspended on 20 strain-hardened

stainless rods each 1.5 in. diam and 8.33 ft long. Twenty additional sway rods, each 1.125 in. diam and 12.83 ft long, prevent rotation of the inner shell. Several pipes communicate with the inner shell through the annular space between it and the outer shell. These pipes are listed in Table I.

The dewar annulus is filled with perlite insulation of an estimated density of 9 lb/ft³. Evacuation of this perlite-filled annulus is accomplished through a vacuum manifold. Characteristically, a vacuum of 30 μ Hg is maintained without pumping during the storage of liquid hydrogen. The outer shell is carbon steel plate approximately $\frac{1}{2}$ in. thick. A series of stiffening girders support this plate against the 13-psi pressure difference.

TABLE I

PIPING AND ITS CALCULATED HEAT LEAK INTO THE
INNER SHELL OF THE 500,000-GAL DEWAR

Pipe Diam (in.)	Function	Location	Heat Leak (Btu/hr) ^a
16	Main propellant line	Bottom	10.0
3	LH ₂ fill line	Bottom	5.5
10	Vent and pressure line	Top	22.1
24	Manway	Top	113.2
8	Instrumentation cable	Top	16.5

^aCalculated by Chicago Bridge and Iron Company.

Instrumentation is provided in the dewar to monitor its operations during reactor tests, storage and fill conditions, and cooldown and warmup operations. In the inner shell of Dewar 1, the southernmost of the two dewars, an axial main rake structure contains Rosemount Type 146AA platinum resistance probes for temperature measurement, Allen-Bradley 0.1-W, 27 Ω carbon resistors for liquid level measurement, a time domain reflectometer¹ for continuous liquid level measurement, and a dewar heater to aid in warmup. There is also an off-axis instrumentation rake for measurement of liquid level with carbon resistors and temperature with resistance probes.

In the annular space, several types of instrumentation are available to monitor temperatures and to study the perlite compaction problem.² Sixteen copper-constantan thermocouples are located along two meridians at 30° intervals. These probes are attached to the outside of the inner shell and permit the shell temperature to be monitored when a reference junction and potentiometer are connected outside the dewar. A series of five thermocouples along a rake that extends radially between the inner and outer shells allows perlite temperature gradients to be measured.

Evacuated perlite has been shown to be capable of maintaining the sizeable compressive loads that a temperature cycle can introduce. If perlite fills in the space left by the contracting inner shell during cooldown, a force will be produced on this perlite during the warmup of the inner shell and

loads of several psig can be developed. Strain gauges on the support rod structure, displacement gauges between the inner and outer shells, and a special perlite pressure gauge on the outer shell permit external measurement of these effects during a temperature cycle.

The electric heater installed along the central instrumentation rake was developed at LASL by J. C. Bronson. A 0.040-in.-diam, No. 18 Nichrome wire is coiled on a 1/4 in. mandrel. The resulting coils are mounted on a radiation shield with cured Lavite insulators. Some 33 ft of coiled wire is mounted in three lengths side by side on an 11.5-ft platen. Each 33-ft length has a resistance of approximately 36 Ω . On each platen are three 33-ft lengths wired as a delta (see Fig. 1). Four platens are installed end to end along the rake structure as shown in Fig. 2.

An isolation transformer is installed as additional safety between the 480-V, 3-phase utility power and ground. Each platen is separately wired to a switch³ to permit individual control of the vertically mounted sections. Experience gained with a similar heater installed on Dewar C, Test Cell A, had indicated that the top instrumentation could overheat while the bottom of the dewar was still cool. The total power supplied to these heaters is 32.3 kW at the ambient wire resistance.

The length of heater wire is chosen to keep the surface temperature of the wire less than 580°C, the lowest ignition temperature of a hydrogen-air mixture.⁴ In one atmosphere of air, such low surface

Fig. 1. Section of Nichrome wire heater as installed in 500,000-gal liquid hydrogen dewar.

Fig. 2. Heater sections installed along the main vertical instrumentation rake.

temperatures are achieved and an incipient red heat is barely detectable.

CALCULATIONS

The energy required to warm a liquid hydrogen dewar inner shell is easily calculated from the known metal density and specific heat. For the dimensions given and the metal density, $\rho = 489 \text{ lb/ft}^3$, the total mass of the inner shell is

$$M = 4\pi R^2 \Delta R \rho, \quad (1)$$

$$M = 2.55 \times 10^5 \text{ lb}.$$

The integrated specific heat between 36° and 540°R is

$$E = \int_{36}^{540} C_p dT = 38.17 \text{ Btu/lb}. \quad (2)$$

The energy, ME , needed to warm the inner shell is $9.73 \times 10^6 \text{ Btu}$. The time that a 32.3-kW heater needs to remain on can be calculated to be 3.7 days.

The above is a minimum energy calculation in

the sense that it neglects the energy required to warm the perlite insulation. If the usual approximations are made for a cold dewar, then a linear temperature gradient is expected throughout the perlite in the annular space. (More recent observations cast doubt on these theoretical models. See Reference 5.) From the specific heat average, $\bar{C}_p = 0.18 \text{ Btu/lb } ^\circ\text{R}$, over the same temperature region, the energy required to warm the perlite is $1.25 \times 10^7 \text{ Btu}$. This is greater than that required to heat the metal. From recent observations with this dewar, a reduction of this energy to about $5.4 \times 10^6 \text{ Btu}$ is indicated.

The total expected energy required to warm the dewar is thus about $1.5 \times 10^7 \text{ Btu}$ which would require 5.7 days to supply entirely by the electric heater.

Over this period some heat leak to the inner shell would occur. The piping heat leaks are given in Table I, but they are small compared to the calculated leak of 918 Btu/hr through the perlite. Even with the full temperature difference, the total heat leak is only $3.1 \times 10^4 \text{ Btu/day}$, so its effect can be neglected.

As will be discussed, several variances from the outlined calculations were required to ensure safe operation and as complete perlite decompaction as possible, but these calculations should serve as a guide.

RESULTS

The dewar warmup procedure was determined by a desire to speed the warmup process and to take no risks with the new system. Thus, the first step was to empty the liquid from the dewar about one week before the warmup began. The initial temperature distribution of on- and off-axis internal temperature probes is shown in Table II. The probes some 15 ft distant at the same altitude gave the same temperature, within probe accuracy, as those on the axis. Hence, a temperature stratification in horizontal layers is observed. In fact throughout the dewar warmup these off-axis probe readings differed but little from those of the corresponding on-axis probes, and only the on-axis information is presented. This temperature agreement is of more than passing interest since it shows that the radiation shielding of the probes from the heater was quite

TABLE II

INITIAL INTERNAL TEMPERATURES ON DECEMBER 16
AFTER THE DEWAR HAD BEEN NOMINALLY EMPTY OF
LIQUID HYDROGEN FOR ONE WEEK

On-Axis		Off-Axis	
kgal level	°R	kgal level	°R
500	224	500	224
400	192	400	192
300	---	300	170
200	152	200	153
100	136	100	137
5	124		

satisfactory and that the instrumentation platens did not warm faster than the dewar.

After the initial set of inner shell readings was taken, some 390,000 std ft³ of gaseous hydrogen was passed through the dewar. This gas was surplus to the test cell, and, hence, it was decided to use the stored energy available. If the maximum average temperature change of the hydrogen is taken as 250°R, then some 2×10^6 Btu could be available. It was clear that nowhere near this amount of energy could be extracted. Indeed after the blowdown a temperature rise of 70° to 130°R was observed in the gas space, but even this is optimistic as will be seen.

A technique successfully employed to promote dewar warmup on a number of other liquid hydrogen dewars in use at LASL is to admit a quantity of gaseous nitrogen such as would produce a pressure rise to 1 to 10 mm Hg in the annular space at ambient temperature. Initially this gas is mostly condensed out on the cold surface, a process that costs the heat of vaporization from the cold surface and the change of enthalpy to vaporization temperature from the perlite. The residual pressure is generally about 1000 μ Hg, and, thus, the heat transfer is increased. Finally the gas pressure acts as a lubricant in the perlite flow process, and thus the temperature-induced forces dissipate in small perlite displacements - viscosity.

Two problems that could generate a hazard were considered during the warmup. A dewar that has been cold for a time and has had an air leak from the outside, accumulates liquid or solid air on the outer surface of the inner shell. Upon rapid warming of

the inner shell, the pressure rise might endanger the vessel (in our case protection was provided by a 6-in.-diam burst disc on the annular space) or the liquid air might flow down through the perlite and puddle on the bottom of the outer shell. This shell could rupture since carbon steel becomes very brittle at these temperatures. Other facilities have reported such difficulties. During the warmup the pressure was monitored with a vacuum thermocouple gauge (5 to 500 μ Hg), a Wallace and Tiernan differential pressure gauge (1 to 760 mm Hg), and a 0- to 21-psia gauge. During critical times the temperature of the bottom of the outer shell of the dewar was observed. Pressures are reported in the operational summary of the Appendix.

The second problem was to establish a nonexplosive atmosphere in the dewar inner shell before turning on the heaters. There was no need to depend on the low surface temperature to prevent an explosion. Small pressure taps were available at the top of the dewar for taking gas samples at the top and bottom of the dewar. The gas samples were analyzed by standard techniques.⁶ Gaseous nitrogen was introduced, starting on December 16, to inert the inner shell. The accumulation of air impurities that may have been introduced during the transfer or storage of liquid hydrogen is not easily calculable. The gas sample analysis, sensitive to about 2 ppm oxygen provided adequate knowledge to avert a problem. The observations over the period of interest are shown in Table III. It should be

TABLE III
VARIATION OF OXYGEN CONTENT MEASURED
WHILE INNER SHELL WARMED ABOVE LIQUID
OXYGEN VAPORIZATION TEMPERATURE

Gas	After GN ₂ Pressure to 25 psig Dec. 16	1020 hr Dec. 19	After Further GN ₂ Purge 1345 hr Dec. 19
	Top of Dewar 1		
H ₂	40%	7%	4%
O ₂	106 ppm	93 ppm	123 ppm
N ₂	60%	93%	96%
Gas	Bottom of Dewar 1		
H ₂	40%	7%	5.6%
O ₂	67 ppm	93 ppm	137 ppm
N ₂	60%	93%	94.4%

noted that the purchased nitrogen vaporized on site for gas use contained about 40 to 100 ppm oxygen. As may be seen, there was no oxygen content increase over the weekend when the temperature rose above the oxygen vaporization temperature throughout the dewar.

Following the weekend warmup using the increased heat leak through the annular space and some energy from gas blowdown and purge, the inner shell hydrogen concentration was decreased to about 5%, and the dewar heaters were turned on. Full heater power was used day and night from 1518 hr on December 19 until 1935 hr on December 21. During this time the average temperature in the dewar rose from 356° to 501°R as shown in Fig. 3.

The lower sections of the heater were left on until 0915 hr on December 22 when the lowest temperature of the inner space was above the water freezing point. The greatest temperature was 530°R, well within the tolerance of installed equipment. Thus the warmup was completed in about six days.

DISCUSSION OF RESULTS

Two aspects of the warmup are of particular interest. Over the time that the electric heaters were turned on we can form an energy balance. The annular space thermocouple data during the warmup can be compared with the data obtained from the inner shell gas space.

The period of full power heater operation was 52.25 hr. Thus 1.69×10^3 kW hr was supplied to the dewar. From the average specific heat in this temperature region, the total energy absorbed by the metal shell was equivalent to 1.2×10^3 kW hr. From observations of the increase in perlite temperature using only a very crude number for specific heat, that is the average number used earlier, energy absorption by the perlite is equivalent to 0.53×10^3 kW hr. Since the perlite warming calculation is based on participation of only the inner 1-ft thickness and does not take into account the external heat leak, this agreement is within present accuracy of computation. As suspected, the amount of energy to warm the perlite is much less than that predicted by present theory.

The progress of the warmup of the annular space perlite is shown in Fig. 4. The first gradient

shown was taken after the weekend when the annular space was filled with a small pressure of gaseous nitrogen. The gradient is constant, indicating that conduction in the annular space is the dominant heat transfer mechanism. During the warming of the inner shell some energy is deposited into the perlite in addition to the small atmospheric heating at the outer shell.

The inner shell temperatures have been compared with the main axis gas temperatures. At the start of the electric heating, a 7°R difference at the dewar midpoint was observed. Well after constant heating rate was established, a 14°R difference was found. Some variation occurred later, but the final temperature was about 18°R lower in the wall than in the gas space. In any case these differences are sufficiently small that dewars could be safely warmed without these additional temperature measurements.

The strain gauge and displacement gauge measurements during the warmup showed only slight unloading of the inner shell due to perlite compaction forces.⁷ From initial to final measurements during the warmup, an unloading equivalent to ~1 psi perlite pressure occurred. Some slight reduction was observed when additional gas was put in the annular space. This supports the idea that the gas allows the perlite to redistribute by decreasing its apparent viscosity, though these measurements are still preliminary. The displacement gauge showed the direct return of the inner-outer shell warm spacing. Thus, this first temperature cycle was completed without developing large measurable perlite compaction forces.

CONCLUSIONS

A 500,000-gal liquid hydrogen dewar has been warmed to ambient temperature by 1) breaking the annular space with a small quantity of gaseous nitrogen and 2) using an installed electric heater of 32.3-kW capability. Some previously reported hazards were not observed using this procedure, and sufficient instrumentation was available to observe the details of the warming process. Energy balances both precalculated and calculated from observed data have shown that the energy required to warm the perlite insulation is less than that calculated from present theory.

Fig. 3. Time history of selected probes at 5, 100, 200, 300, and 500 kgal levels. Significant operations are listed. No work was done over this weekend December 17 and 18, 1966.

Fig. 4. Annular space thermocouple temperature measurements versus location in the annular space.

APPENDIX

Condensed Time History of the First Warmup of Dewar 1.

Date	Time		
12-16	1015	Annular space pressure 30 μ Hg.	1055 Pressurize inner shell to 26 psig.
	1030	Blowdown 390,000 std ft ³ of hydrogen from top to bottom outlet.	1400 Vent inner shell to 0 psig.
	1200	Annular space pressure 50 μ Hg. Start GN ₂ inerting.	1425 Pressurize inner shell to 11 psig.
	1340	Pressurize to 26 psig.	1518 Turn on dewar heater sections 1, 2, 3, and 4. Monitor temperatures hourly day and night.
	1400	Annular space pressure 80 μ Hg.	12-20 1055 Vent inner shell to 0 psig.
	1405	Vent inner shell to 0 psig.	1100 Pressurize inner shell to 26 psig.
	1535	Pressurize inner shell to 14 psig GN ₂ .	1145 Vent inner shell to 12 psig.
	1630	Introduce approximately 120 std ft ³ GN ₂ into annular space. Annular space pressure not measurable.	12-21 0820 Vent inner shell to 0 psig.
	1730	Introduce an additional 150 std ft ³ GN ₂ into annular space. Pressure approximately 7 mm Hg.	0945 Pressurize inner shell to 12 psig. Gas sample shows H ₂ $\approx$ 1% (other gas samples are not included in this summary.)
12-19	0830	Annular space pressure 10 mm Hg. Inner shell pressure 14 psig.	1935 Turn off top section of heater, No. 1.
	1020	Pressurize inner shell to 26 psig.	12-22 0220 Vent inner shell to 2 psig.
	1040	Vent inner shell to 0 psig.	0300 Turn on heater section 1.
			0330 Turn off heater section 1.
			0405 Turn off second heater section from the top, No. 2.
			0810 Vent inner shell to 0 psig. Purge helium from outlet valve block system into dewar.
			0915 Turn off heater sections 3 and 4 and obscure all heaters.

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REFERENCES

1. W. Willis and J. Taylor, private communication.
2. F. J. Edeskuty and J. K. Novak, private communication.
3. The wiring diagram is on Drawing 56Y117142D, available from CFSTI.
4. "Liquid Hydrogen: A Guide for the Safe Handling and Storage of Liquid Hydrogen at LASL Facilities," The Liquid Hydrogen Safety Committee, Los Alamos Scientific Laboratory, University of California (15 June, 1962).
5. D. H. Liebenberg, R. W. Stokes, and F. J. Edeskuty, "Chilldown and Storage Losses of Large Liquid Hydrogen Storage Dewars," *Advances in Cryogenic Engineering*, Vol. 11, K. D. Timmerhaus, Ed., Plenum Press, New York, 1966, p. 554.
6. D. H. Liebenberg and F. J. Edeskuty, "Use and Calibration of a Gas Chromatograph for Gas Analysis at the Project Rover Test Facility," *Ibid.*, Vol. 9, p. 430.
7. J. K. Novak, private communication.