NBSIR 73-308

Insulation of Liquid Oxygen Dewars

C. F. Sindt

Cryogenics Division
Institute for Basic Standards
National Bureau of Standards
Boulder, Colorado 80302

April 1973

Final Report

Prepared for
Naval Air Engineering Center
Philadelphia, Pennsylvania 19112

NBSIR 73-308

INSULATION OF LIQUID OXYGEN DEWARS

C. F. Sindt

Cryogenics Division
Institute for Basic Standards
National Bureau of Standards
Boulder, Colorado 80302

April 1973
Final Report

Prepared for
Naval Air Engineering Center
Philadelphia, Pennsylvania 19112

U.S. DEPARTMENT OF COMMERCE, Frederick B. Dent, Secretary
NATIONAL BUREAU OF STANDARDS, Richard W. Roberts, Director

CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABSTRACT

The Navy has experienced failure of vacuum insulation in dewars used for storage and handling of liquefied breathing oxygen for aircraft pilots. Because of the vacuum insulation failures, a search was made for a more rugged insulation that has thermal performance similar to the currently used vacuum with multilayer or powder. No system was found that compared in thermal performance and did not require a vacuum. Two systems were experimentally evaluated that did not require vacuum. One was polyurethane foam with an intermediate fiber glass shell and the other was glass bubbles in argon gas at one atmosphere pressure. The polyurethane foam system was successful in that no cracks penetrated to the outside surface; however, the average thermal conductivity was 160 μ W/cm-K which is about 15 times greater than vacuum and powder. The glass bubbles in argon gas was also successful since the argon gas pressure always remained high enough to prevent air and moisture from entering the insulation through small leaks in the outer shell. The thermal performance was poorer than the polyurethane foam. The average thermal conductivity was 212 μ W/cm-K or about 20 times greater than for the same glass bubbles in a vacuum.

Key words: Cryogenic insulation; insulation; LOX dewars; microspheres; polyurethane foam.

INSULATION OF LIQUID OXYGEN DEWARS

Charles F. Sindt

1.0 Introduction

Breathing oxygen for the crews of military aircraft is supplied by vaporizing liquid which is stored on board the aircraft in a cryogenic container. These cryogenic liquid oxygen containers (converters) are filled prior to each flight by either removing the oxygen supply unit for filling, or in the earlier models, by filling the unit in place. The aircraft containers are filled from portable dewars ranging in size from 0.19 m^3 (50 gallon) to 1.9 m^3 (500 gallon) capacity. The portable dewars are used to transport the liquid oxygen from a larger storage dewar to the flight deck or flight line to service a number of aircraft. The large storage dewars are usually about 15 m^3 (4000 gallon) capacity and are serviced from a transport truck or ground based liquefier or from a shipboard liquefier on the aircraft carriers.

All of the current liquid oxygen containers require vacuum in the insulation spaces. The airborne containers are insulated with vacuum and some also include alternate layers of good reflectors separated by poor conductors commonly referred to as multilayer (MLI) or super (SI) insulation. Either system requires a vacuum of 1 mN/m^2[*] for best performance. The portable vessels are also vacuum insulated, some with MLI and some with powder in the insulation space. The powder is usually perlite. Powder and vacuum insulation is always used in the large storage dewars. Powder systems also require a good vacuum, about 0.15 N/m^2 or less.

---

[*] 1 mm Hg = 133.32 N/m^2; 1 atm = 101 325 N/m^2 $\approx$ 101 kN/m^2.

In the past, the Navy has experienced a significant failure rate in the vacuum insulation in all dewar sizes. Vacuum failures usually require extensive repair by highly skilled craftsmen using specialized equipment. Therefore, since vacuum failure usually renders the cryogenic vessel unusable and vacuum failures normally require repairs that cannot be made in the field or on shipboard, a failure may result in loss of the equipment.

Because of the number of failures occurring in the vacuum-jacketed vessels, the Naval Air Engineering Center has requested a review of insulation systems to find a system that is more reliable and less fragile and, if feasible, a method of repairing existing dewars.

This report covers the experimental program to evaluate several candidate insulation systems and resulting recommendations.

2.0 Experimental Apparatus and Procedure

The experimental apparatus for measuring the average thermal conductivity and the temperature profiles in the evaluated insulation systems is shown schematically in figure 1. The gas vent from the experimental vessel was connected to a barostat which maintained the vessel pressure constant to ± 260 N/m^2. The discharge of the barostat flowed through a dry gas meter which measured the volume of gas vented.

Instrumentation for the experiment consisted of temperature measurements in the insulation, in the liquid, and in the venting gas at the dry gas meter. Temperatures were measured with type T thermocouples with the reference thermocouple in the bottom of the experimental vessel. Pressure measurements were taken at the dewar vent and at the inlet to the dry gas meter. Pressures were measured with variable reluctance type pressure transducers. The gas flow measurement was made using a dry gas meter which gave an electrical pulse

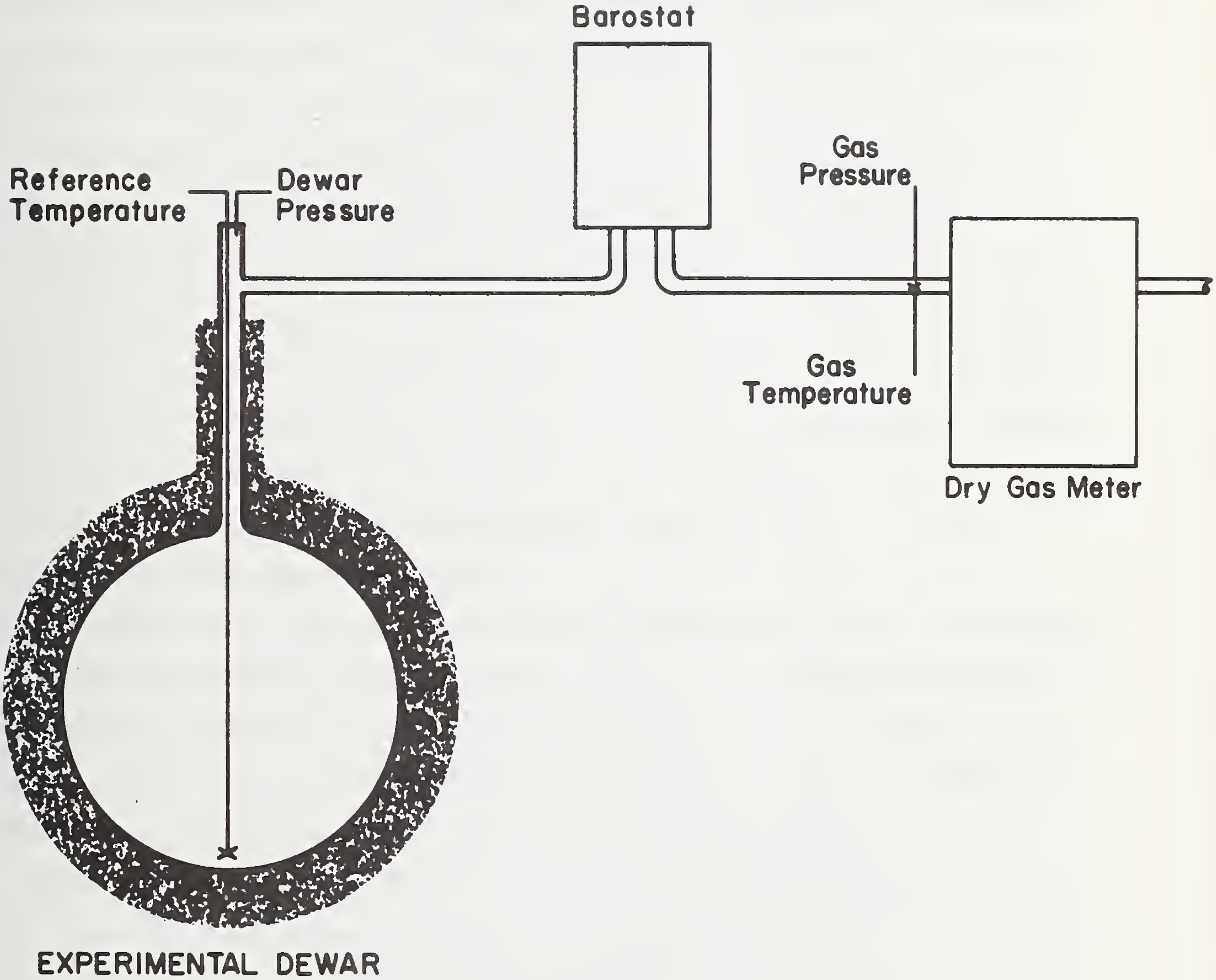

Figure 1. Schematic of experimental apparatus.

for each 2.83×10^{-3} m^3 of gas flow. The signals from the thermocouples, the pressure transducers, and the dry gas meter were automatically interrogated and recorded with a computerized data acquisition system. Data reduction was performed on-line by the computer, and temperatures, pressures, and total heat leak to the experimental vessel were printed out at the desired time intervals. The overall accuracy of the heat leak measurement is estimated to be within ± 4%.

The experimental procedure started by filling the experimental vessel with liquid nitrogen and connecting the vent to the barostat. The barostat was set to maintain the desired pressure. The computer was then started and data were taken at 1 or 2 hour intervals. When the heat leak was nearly constant for several hours, thermal stability was assumed and the test was terminated. For the vessels tested, the time to reach stability varied from 12 to 36 hours.

3.0 Experimental Dewars

A search of the literature for insulation schemes that would be applicable for liquid oxygen did not reveal any system that was as good as vacuum insulation systems. No systems were discovered that were rugged and that approached the performance of the currently used vacuum and MLI or vacuum and powder. One article was published during the latter stage of our work that described a system that was comparable in performance to some of the evacuated powders and appeared to be more rugged as well as compatible with the environment on aircraft carriers [Haynes, 1972]. The system consisted of polyurethane foam with fiber glass reinforcement. The only information given on the thermal insulation performance was that the nominal thermal conductivity between 110 K and ambient temperature was 14 μW/cm-K. This system had not been put into the intended liquefied natural gas service at the time the article was printed.

A second application using urethane foam as an insulation at cryogenic temperature was reported by F. Mac and M. Smith [1972]. This application with liquid hydrogen and that reported with liquefied natural gas indicated that the urethane foam may be a good candidate for a rugged insulation that would not be susceptible to failure because of small shallow punctures. Also, the urethane foam would be easily repaired in the field or onboard ship.

For our tests, a urethane foam was selected that was very similar to that used in the liquid hydrogen application; two significant differences were that the selected foam was a pour-type foam instead of a spray-on type foam and the thickness of the foam was 9.5 cm instead of the 1.9 cm used in the liquid hydrogen application. The pour-type foam was selected for two reasons. First, special spray equipment was not required for application and second, if successful, the foam would be especially attractive to use for repairing existing dewars as well as repairing new foam insulated dewars.

The second insulation method selected for evaluation was hollow glass microspheres (bubbles) similar to those described by Cunnington [1972]. The glass bubbles selected were not aluminum coated as the current cost and supply of coated bubbles was not well established and availability for experimental use was on a long term delivery. The glass bubbles were selected as they did represent a new insulation material and, also, reported performance was good. Since the reported performance was in a vacuum environment and was similar to perlite in a vacuum, the decision was made to measure performance of the glass bubbles in gases at one atmosphere pressure. The reasoning was that under one atmosphere pressure, no significant insulation degradation due to vacuum leaks would occur while the vessel sat idle and warm. The gases selected for the insulation environment were nitrogen, argon, and carbon dioxide.

The glass bubbles selected contain a gas that condenses and freezes at liquid oxygen temperatures. Therefore, the thermal conductivity of the colder glass bubbles and the gas surrounding them near the inner dewar wall should be nearly the same as the thermal conductivity of the surrounding gas since the glass bubbles have thin walls and point contact. The wall thickness of the glass bubbles is estimated to be 1 μm from the manufacturer's data which specify diameters from 60 to 120 μm, bulk density of 0.076 g/cm^3, and true density of 0.155 g/cm^3.

The nitrogen, argon, and carbon dioxide gases were selected because they are all readily available, and they are inexpensive. Argon was selected because the thermal conductivity of argon gas in the temperature range of 90 K to 300 K is lower than any other fluid that remains gaseous in this temperature range and at one atmosphere pressure.

Carbon dioxide was selected because it was a gas that was readily available and inexpensive, and because it would freeze out on the dewar walls at liquid oxygen temperatures. The liquid oxygen temperature is 105 K at the maximum dewar operating pressure of 374 kN/m^2. Carbon dioxide has a vapor pressure of less than 0.13 N/m^2 at 105 K and the pressure drops rapidly with lower temperatures [Honig and Hook, 1960]. This pressure is compatible with that required for powder or glass bubbles to give maximum insulation.

A second reason for selecting the glass bubbles was that handling may be easier than perlite or other fine particle powders simply because of the spherical shape.

Performance tests were conducted on each of the dewars. Test data consisted of temperatures at radial distances 0.65 cm apart in the insulation, dewar pressure, gas meter pressure and temperature, and the heat leak of the dewar. The heat leak for all of the tests was calculated from the volume of boil-off gas, from the gas temperature and pressure at the gas meter inlet, and from the calculated heat of vaporization of the liquid nitrogen. The nitrogen in the dewar was assumed to be at constant temperature and in thermal equilibrium with the ullage pressure. The heat leak of the dewar was measured after a minimum of 12 hours thermal stabilization time and the inner vessel was made of copper; therefore, the assumption of equilibrium conditions in the dewar is substantiated.

The thermocouple used as a reference for all temperature measurements was located at the bottom of the experimental dewar. With thermal equilibrium in the dewar, this temperature must be within 0.3 K of the liquid saturation temperature as calculated from the measured dewar pressure.

3.1 Foam Insulated Dewars

Four dewars were made using foam insulation. A total of nine tests were conducted on these dewars. All of the foam dewars were made using 30.5 cm diameter copper hemispheres for the cold vessel. These hemispheres were soldered together and a 1.27 cm diameter tube, 45 cm long, was soldered to a center hole in the upper hemisphere. This tube was used for the liquid fill and gas vent line. Three of the four foam insulated dewars had an intermediate fiber glass shell in the foam insulation. The other dewar insulation was all foam. The intermediate fiber glass shells were all 40 cm in diameter and the outside diameter of the outer foam layer was 49.5 cm.

Dewar A was made by pouring the premixed foam into the fiber glass shells. On dewar A, the external surface of the copper inner vessel was cleaned with trichloroethane and wiped dry with clean paper towels prior to pouring the foam. The foam was poured into the lower fiber glass hemisphere first. Then the foam was trimmed to the top of the fiber glass shell. The upper fiber glass hemisphere was attached to the lower one with fiber glass tape and resin. Next the foam was poured into the upper hemisphere. Construction of this vessel was our first attempt at using the selected pour-type foam and the quality of the foam was very poor. The poor quality was caused by inadequate mixing of the two liquid components. These two components must be thoroughly mixed in less than 30 seconds as the foam starts to rise at approximately 30 seconds. After the rise has started, the mixture cannot be poured from a container. The mixing problem was resolved after consulting with the manufacturer and other users of the foam.

The poor quality of the foam was evident by color streaks, surface voids, and sub-surface voids which could be detected by pressing on the foam surface. Even though the first foam layer was of poor quality, the second layer was poured. This second layer was poured similar to the first except that mixing was improved and 49.5 cm diameter steel hemispheres were used for the outer molds. The inner surfaces of these shells were coated with silicone grease prior to pouring the foam. The grease was a releasing agent and the steel shells were removed after the foam had cured. The upper steel hemisphere had a centrally located hole, 10 cm in diameter. A 10 cm diameter cylinder was attached at the hole and was used as a mold to form a 40 cm long cylinder of foam around the fill and vent line. This foam was formed when the upper hemisphere filled with foam and the foam continued to rise. A schematic of the foam dewars is shown in figure 2.

Performance tests were made on dewar A even though the foam quality was poor. However, the insulation quality of this foam was

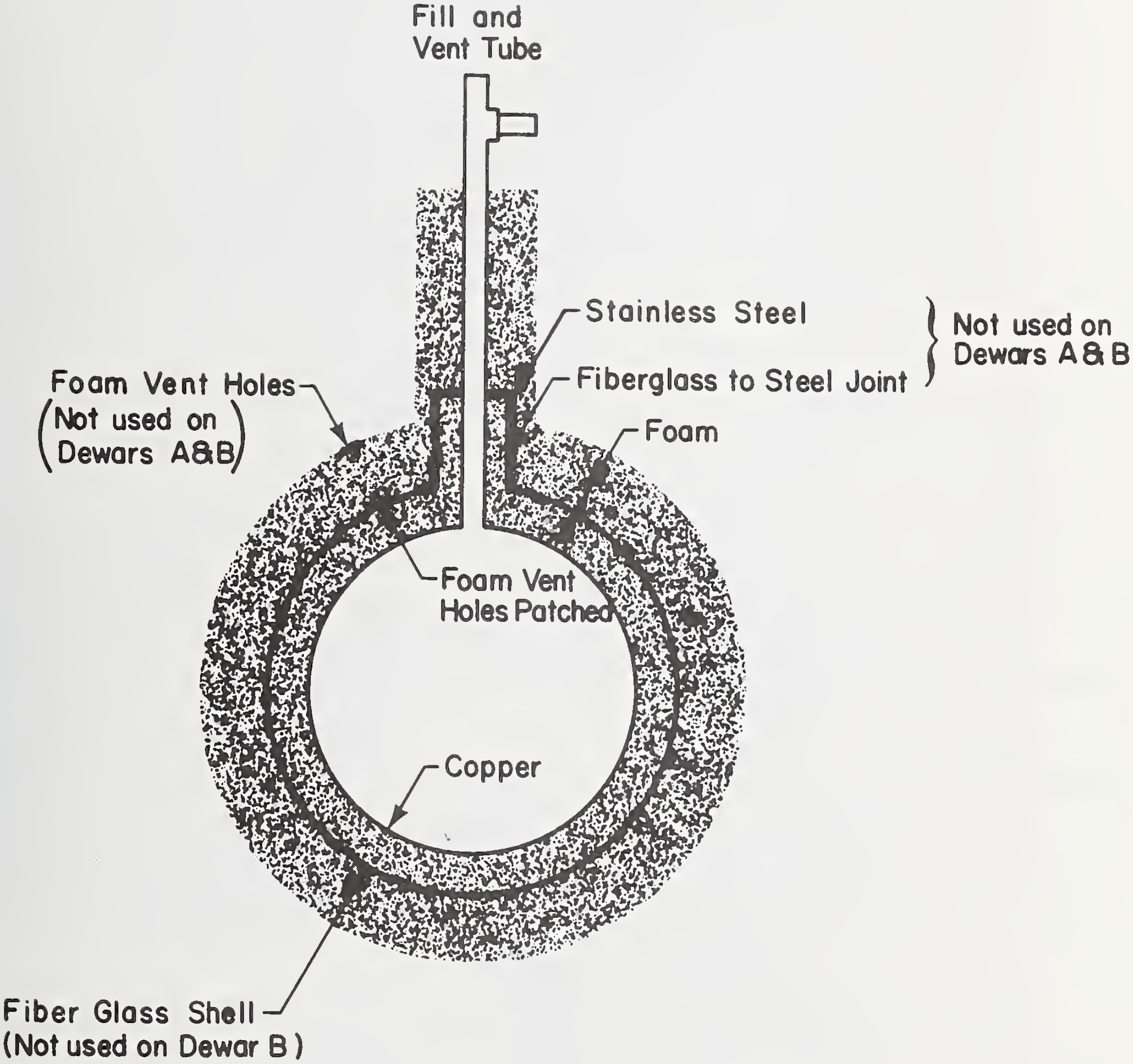

Figure 2. Schematic of dewars insulated with foam.

not much different than the other dewars. The thermocouple data for temperatures within the foam insulation were not valid for this test as many were located in common voids in the insulation.

Insulation on dewar B was all foam with no fiber glass shell. The copper inner vessels of dewars B, C, and D were primed with a polyurethane primer prior to pouring the foam. The primer was used to improve bonding between the foam and the cold wall. The improved bonding was considered best because the stress in the foam due to differential thermal contraction between the copper shell and the foam would then be evenly distributed in the foam. Sectioning after the test indicated the bond between the primer and the metal and the primer and the foam was excellent. No bond failure was visible and removal of the primer and foam from the copper shell required buffing with a wire brush.

The 49.5 cm diameter steel shells were used as the mold for dewar B and the foam was made in two pours, one for each hemisphere. The foam quality appeared good from the outer surface. Sections of the foam taken after tests showed that the quality was good everywhere except at the transition from the sphere to the cylindrical neck. In this area, gas bubbles were trapped during the foam rise and voids occurred. These voids and the poor quality foam surrounding them are shown in figure 3.

The data for foam insulated dewar B appeared to be good. Even though some voids in the foam occurred around the neck, the effect of these voids on the overall dewar performance appeared small since data indicated that the temperature of the gas in the dewar neck was within 2 K of the temperature of the liquid at the bottom of the dewar.

The performance of dewar B deteriorated with each test as the total heat leak for tests one, two, and three was 16.9, 17.7, and 18.4

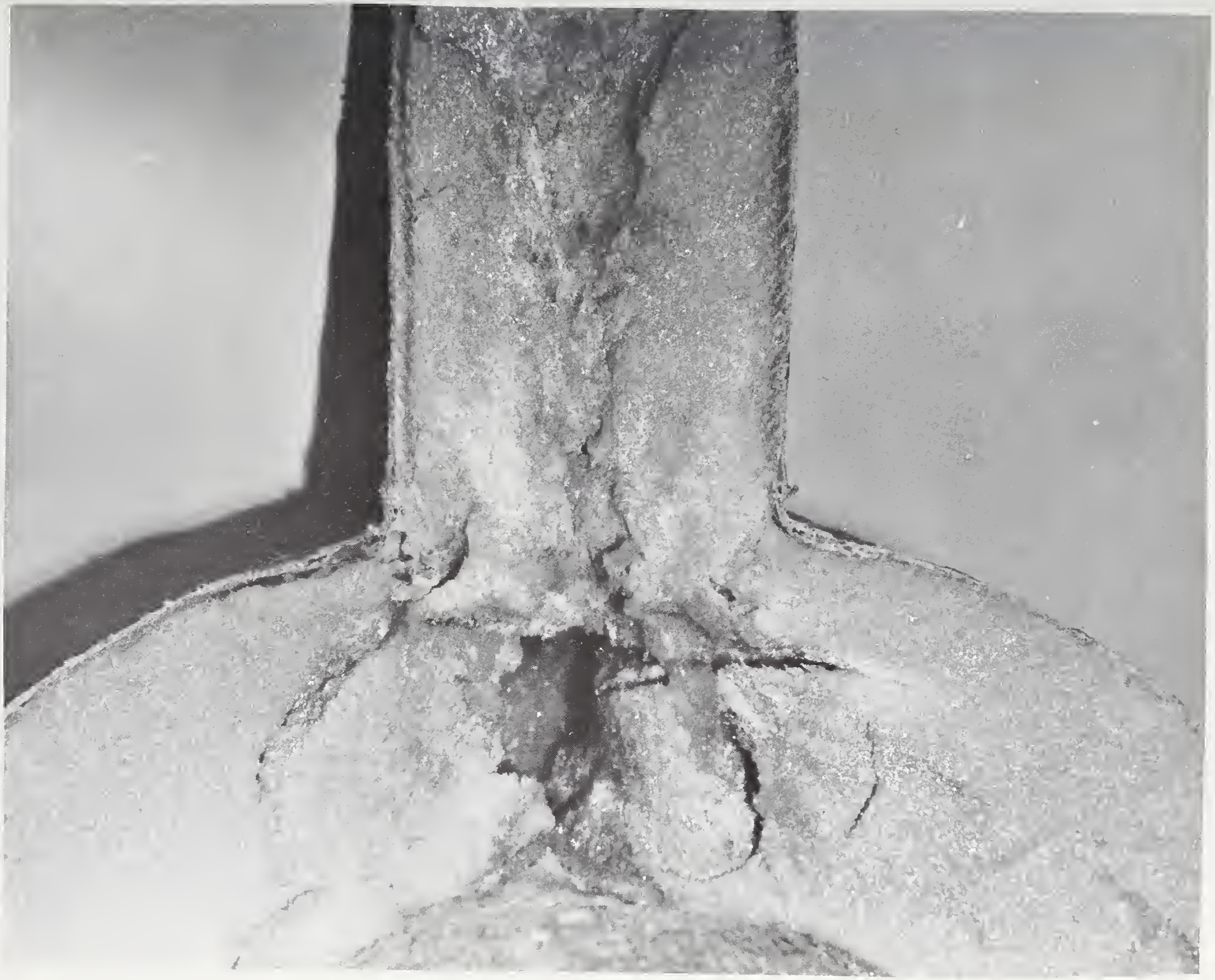

Figure 3. Voids in foam insulation at the dewar neck.

watts, respectively. This deterioration of performance was probably not due to general deterioration of the foam insulation but rather to specific failures of the foam. The failure during test two was around the neck and frost formed at this failure. After test two, the dewar was covered with fiber glass and resin on the outside of the foam. On test three, the foam failed in the lower hemisphere. Figure 4 shows the frost formation on the outer fiber glass shell. The frost formed at each end of a large crack. This crack is shown in figure 5 which is a photograph of a section of the foam taken after the test.

Data taken from test one for dewar B should be quite accurate for determining the thermal conductivity versus the temperature for the foam insulation. The results of this test are shown in figure 6.

Dewars C and D were both made with the intermediate fiber glass shell using a procedure similar to that used for dewar A. The fiber glass shell was used because the dewar without the intermediate fiber glass shell failed. The reasoning was, if the inner layer of foam cracked, the cracking would stop at the fiber glass shell, and since the temperature would be much higher at the fiber glass than at the inner wall, the probability of this crack propagating through the fiber glass was very small.

In addition to the fiber glass shell, a second feature was added to these two dewars. The feature was to seal off the inner foam by closing the fiber glass around the fill and vent line. This was accomplished by soldering a 2.5 cm diameter stainless steel transition tube 10 cm long to the fill and vent line. The steel tube was then joined to the fiber glass shell with a fiber glass joint. Figure 2 shows a schematic of this construction.

To help alleviate the problem of trapped gas at the sphere-to-neck transition during foam rise, holes were placed in the upper hemispheres.

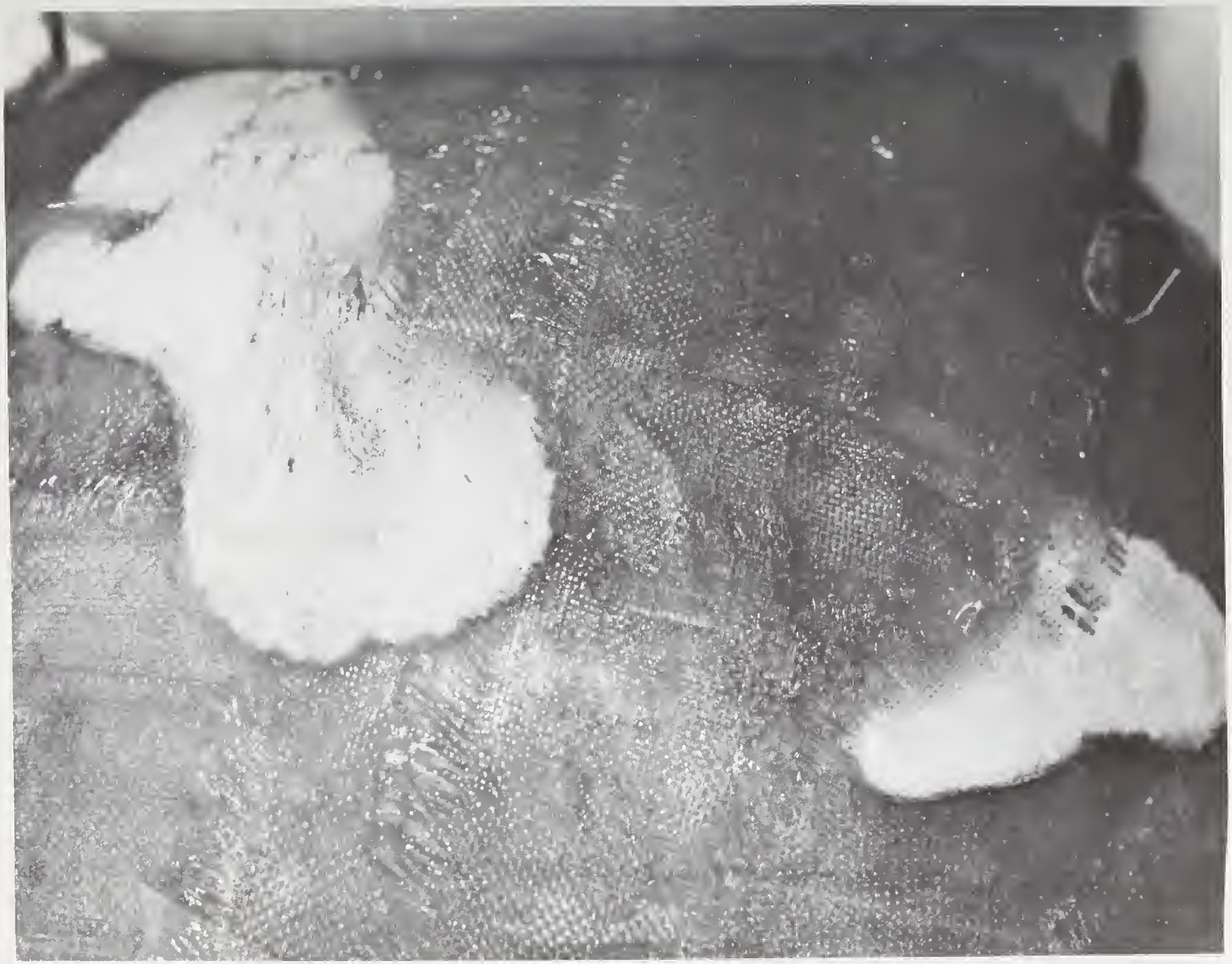

Figure 4. Frost formation on the foamed dewar failure.

Figure 5. Crack in foam insulation.

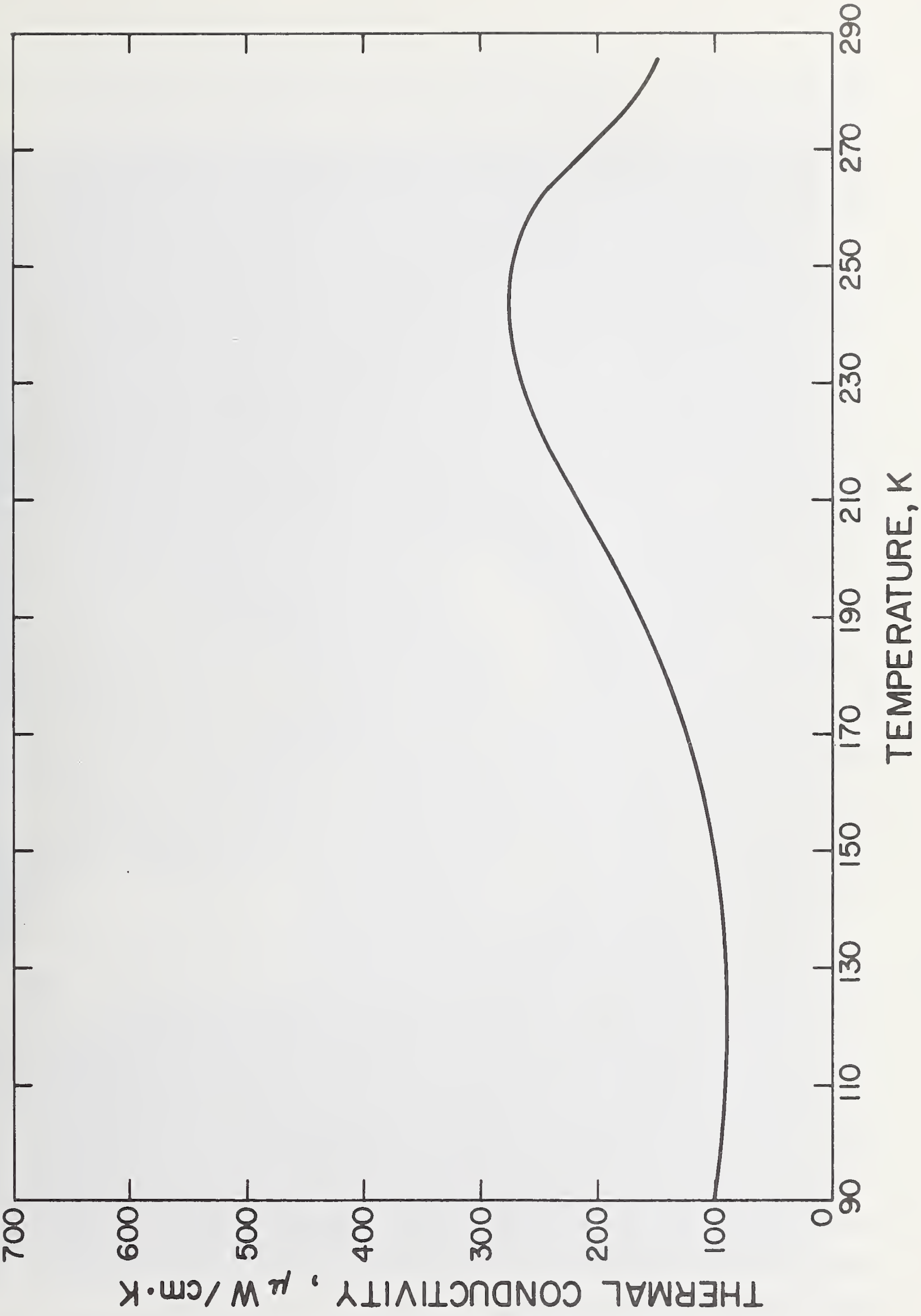

Figure 6. Thermal conductivity versus temperature for polyurethane foam.

Six holes about 1.5 cm in diameter were evenly spaced about 8 cm away from the neck hole in the fiber glass and in the steel shell molds for the outer foam layer. The holes in the fiber glass were patched before the outer foam layer was added. These holes are shown in figure 2.

Although the holes helped reduce the undesirable condition shown in figure 3, some voids still occurred in the outer foam layer. These voids caused subsequent failure at the neck-transition on the first cooling of both dewars. This failure is shown in figure 7. However, no frost appeared at the failure, so the fiber glass shell with the seal around the fill and vent line must have provided sufficient insulation to prevent frost formation. The failure in dewar D was repaired by removing all of the foam that contained voids and then refoaming the neck transition. This dewar is shown in figure 8.

Dewars C and D were slightly different in that the inner wall of the fiber glass shell on dewar D was coated with silicone grease prior to pouring the inner foam layer. The silicone grease was used to allow release of the inner foam layer from the fiber glass shell. This difference in fabrication did not produce any significant differences in dewars other than a small difference in the performance on the first cooldown.

The tests conducted on foam dewars C and D did not include temperatures in the insulation. Tests were made for heat leak after the first cooldown and after the 17th cooldown. Both dewars were cooled thirty times from ambient to 77 K. No surface failure occurred except as previously noted, which was at the neck-to-sphere transition. Dewar D, which was repaired, did not show any surface failures after repair. The average thermal conductivity of the foam on the two vessels was different during the first tests. The average thermal conductivity for the dewar with the foam bonded to all surfaces, dewar C, was greater than for the dewar with the silicone grease on the inner wall of the fiber glass shell.

Figure 7. Failure of foam at the dewar neck.

Figure 8. Foam insulated dewar with a repair of the neck.

The average thermal conductivity after the 17th cooldown was larger for both dewars but there was practically no difference between the dewars. Table 1 shows the thermal conductivity of the foam dewars. The subscript on $\bar{k}$ is the number of the cooldown when $\bar{k}$ was measured.

Some of the properties of foam samples taken from dewars A and B are given in table 2. The samples from dewar A are from the outside layer of foam. As is indicated in the table, the samples from dewar A and dewar B are of different density. This density difference was probably caused by a difference in temperature of the foam components when poured, or a difference in temperature of the metal molds at pouring time. The molds were heated but the temperature was not closely controlled.

The large difference in thermal contraction between samples is related to the cell orientation in the foam. Foam cells were in general elongated in the direction of foam rise and the elongation was as much as two to one for many of the cells. Cell elongation was also affected by the geometry of the annulus being foamed. If the annulus was increasing in cross section normal to rise the cells are less elongated.

Elasticity of foam is also affected by cell structure [Reed, 1972]. Elasticity of urethane foams may be several times greater normal to the cell elongation, especially at cryogenic temperatures. The combination of elasticity and thermal contraction will determine if the foam will fail at the bond to the cold wall or in the bulk insulation due to the large thermal gradient. Since cell geometry affects thermal contraction, and elasticity and cell geometry is a function of confinement during the foam rise, spray foam may be less susceptible to cracking from differential thermal contraction.

Experiments in making and testing the four foam dewars has shown that cryogenic dewars can be made of foam. Special techniques are required to prevent cracks that may form in the insulation at the cold wall from progressing through to the outside during thermal cycling and to assure that the foam is of nearly uniform density.

Table 1. Average thermal conductivity of foam insulated dewars

Dewar	Construction	$\bar{k}$ (77 to 292 K) μW/cm-K	Remark
A	Foam - Fiber glass - Foam	$\bar{k}_1 = 185$	Inner foam layer of poor quality
B	Primer - Foam	$\bar{k}_1 = 153$	Foam cracked at neck
	Primer - Foam - Fiber glass	$\bar{k}_3 = 165$	Foam failed in lower hemisphere
C	Primer - Foam - Fiber glass - Foam	$\bar{k}_1 = 157$	Failure at neck on 1st cooldown
		$\bar{k}_{17} = 167$	
D	Primer - Foam - Silicone grease - Fiber glass - Foam	$\bar{k}_1 = 150$	Failure at neck on 1st cooldown
		$\bar{k}_{17} = 166$	Repaired neck failure

Table 2. Properties of foam insulation

Dewar	Sample No.	Density g/cm^3	Thermal Expansion $\frac{L_{293} - L_{77}}{L_{293}}$	Cells orientation to expansion measurement
A	1	0.0276	0.029	Perpendicular
	2	0.0283	0.009	Parallel
	3	0.0274	0.025	Perpendicular
B	1	0.0252	0.010	Parallel
	2	0.0256	0.020	30° to Parallel
	3	0.0253	0.018	15° to Parallel

As has been previously discussed, voids may form in poured foam. The voids may be eliminated by venting and control of the geometry of the pour molds. To assure that the correct and consistent density foam results in the dewar insulation, each vessel configuration should be experimentally evaluated. Temperatures during foaming, foam density and insulation quality should be measured from dewar to dewar to determine the best foaming technique.

For the reasons previously stated, spray-type foam is probably superior. Spray foam has been used in an experiment to evaluate the possibility of replacing vacuum and powder insulation on storage dewars [R. W. Arnett, 1972]. The experiment was made on an existing 7.5 m^3 (2000 gallon) liquid oxygen vessel. Spray foam has the disadvantage of requiring good access to all surfaces to be foamed but has the advantage of consistency in application. Three other features are apparently needed to assure dependable foam insulation. The first is that at least one intermediate shell of fiber glass and resin is required to stop crack propagation in the cold foam. Second, this fiber glass shell should be gas tight to prevent air and moisture from entering at insulation penetrations such as fill and vent tubes. Third, the cold metal surface should primed to assure good uniform bonding of the foam and the metal.

The requirements for foam insulation, especially spray foam, should be investigated more thoroughly to determine if the primer is really the best system or if the cold surface should be treated for total release. In conjunction with total release, some form of seal would need to be made around the foam and to all penetrations of the foam to keep air and moisture from the cold surfaces. The seal could be the fiber glass shell with metal-to-fiber glass transitions for cold surfaces. Further investigation of spray foams should also include the effects of the cell orientation on the resistance to cracking. This investigation should include determination of basic foam properties, such as thermal contraction, elasticity, density and thermal conductivity of the selected spray foam.

The conclusions from the foam insulation experiments are 1) that foam systems such as those used in dewars C and D will work for cryogenic applications but more development of foaming technique for each specific vessel will be required, 2) spray foam is more desirable because voids caused by gas entrapment can be eliminated, and 3) foam with fiber glass reinforcing layers and a fiber glass cover is a potentially rugged insulation system.

3.2 Glass Bubble Insulated Dewars

The dewar for the glass bubbles used the same size inner vessel made of the 30.5 cm diameter copper hemispheres. The outer vessel was made of two 56 cm diameter steel hemispheres. The fill and vent tube was 1.27 cm in diameter and 45 cm long. The insulation for the fill and vent line was provided by joining to the steel hemispheres a 11.5 cm diameter cylinder 30 cm long. The pumping line and valve and an instrumentation vacuum feed-through were installed in the neck. A schematic of the dewar is shown in figure 9. The glass bubbles were poured into the insulation space through the vacuum valve using a funnel and a 1.3 cm diameter tube that extended through the valve opening. The insulation space was filled to the level of the vacuum valve which was 19.5 cm up from the outer hemisphere. After filling the insulation space with glass bubbles, it was pumped to 0.13 N/m^2 pressure.

Seven tests were conducted on the dewar with glass bubbles as insulation. Two tests were made with a total pressure in the insulation of 0.13 N/m^2; one test was made with nitrogen gas at 101 kN/m^2, two with 80% argon and 20% nitrogen gas mixture at 101 kN/m^2, and two with carbon dioxide at a pressure of 101 kN/m^2 (with the dewar warm). The tests were all conducted as described in section 2. The test data included temperatures in the insulation, temperature and pressure at the gas meter, pressure in the dewar, and heat leak to the dewar.

The first test with glass bubbles, which was conducted with a vacuum in the insulation space, was made after the insulation space was pumped for the first time.

After completing test one, the insulation space was filled to an absolute pressure of one atmosphere with dry nitrogen gas. Test two was conducted with this configuration.

For tests three and four, the insulation space was again pumped to 0.13 N/m^2 pressure, then filled to one standard atmosphere with a mixture of 80 mole percent argon and 20 mole percent nitrogen gas. Tests three and four were then conducted using liquid nitrogen at 223 kN/m^2 pressure. At this pressure, the equilibrium temperature

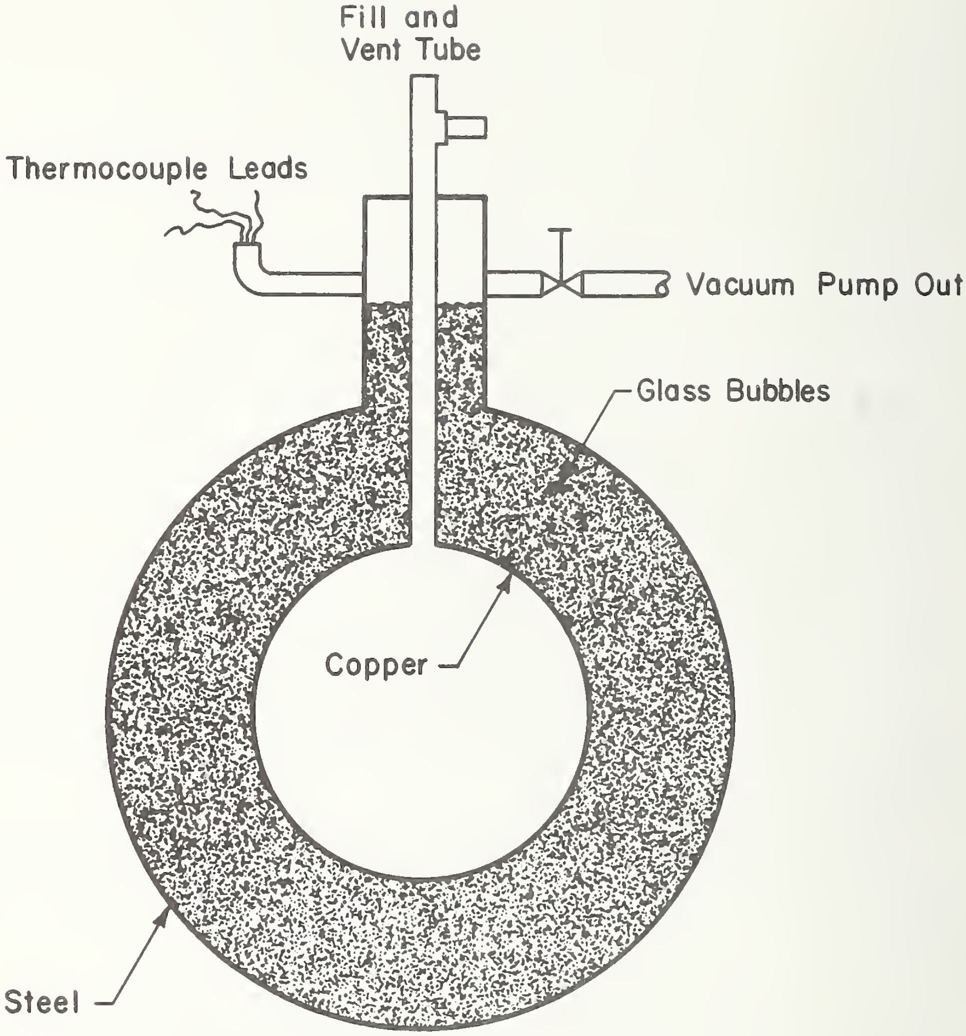

Figure 9. Schematic of dewars insulated with glass bubbles.

of liquid nitrogen is slightly over 84 K. This is near the condensation temperature of argon at 0.8 atmosphere pressure which was the partial pressure of argon in the insulation space. The pressure in the insulation space dropped 10 kN/m^2 after the dewar was at the controlled pressure and boil-off had stabilized for several hours. The argon-nitrogen gas mixture was used instead of pure argon gas because liquid argon was not available to use as the cryogenic fluid; therefore, liquid nitrogen was used. The system was pressure limited to 223 kN/m^2, so the test temperature was limited to slightly less than 85 K. Pure argon gas at one atmosphere pressure would have condensed and run down onto the warm surface of the outer shell causing refluxing and high heat transfer. The 0.8 atmosphere partial pressure of argon prevented the refluxing.

The argon-nitrogen gas mixture data were extrapolated to get estimated data for pure argon. The extrapolation was made assuming conductivity follows a linear relationship between pure nitrogen and pure argon. This extrapolation is believed to be as precise as the experimental data.

Tests five and seven were conducted using carbon dioxide in the insulation space. The insulation space was pumped to 0.4 N/m^2 pressure before the carbon dioxide gas was added. The carbon dioxide gas pressure was one standard atmosphere with the vessel at ambient temperature. Between tests five and seven, the insulation space was pumped to 0.1 N/m^2 and a second test was run with vacuum. The insulation space was then refilled with carbon dioxide gas and test seven was run. A difference in dewar performance was apparent between test five and seven. Curves showing thermal conductivity versus temperature for the tests are given in figure 10. The data presented are for thermal equilibrium conditions.

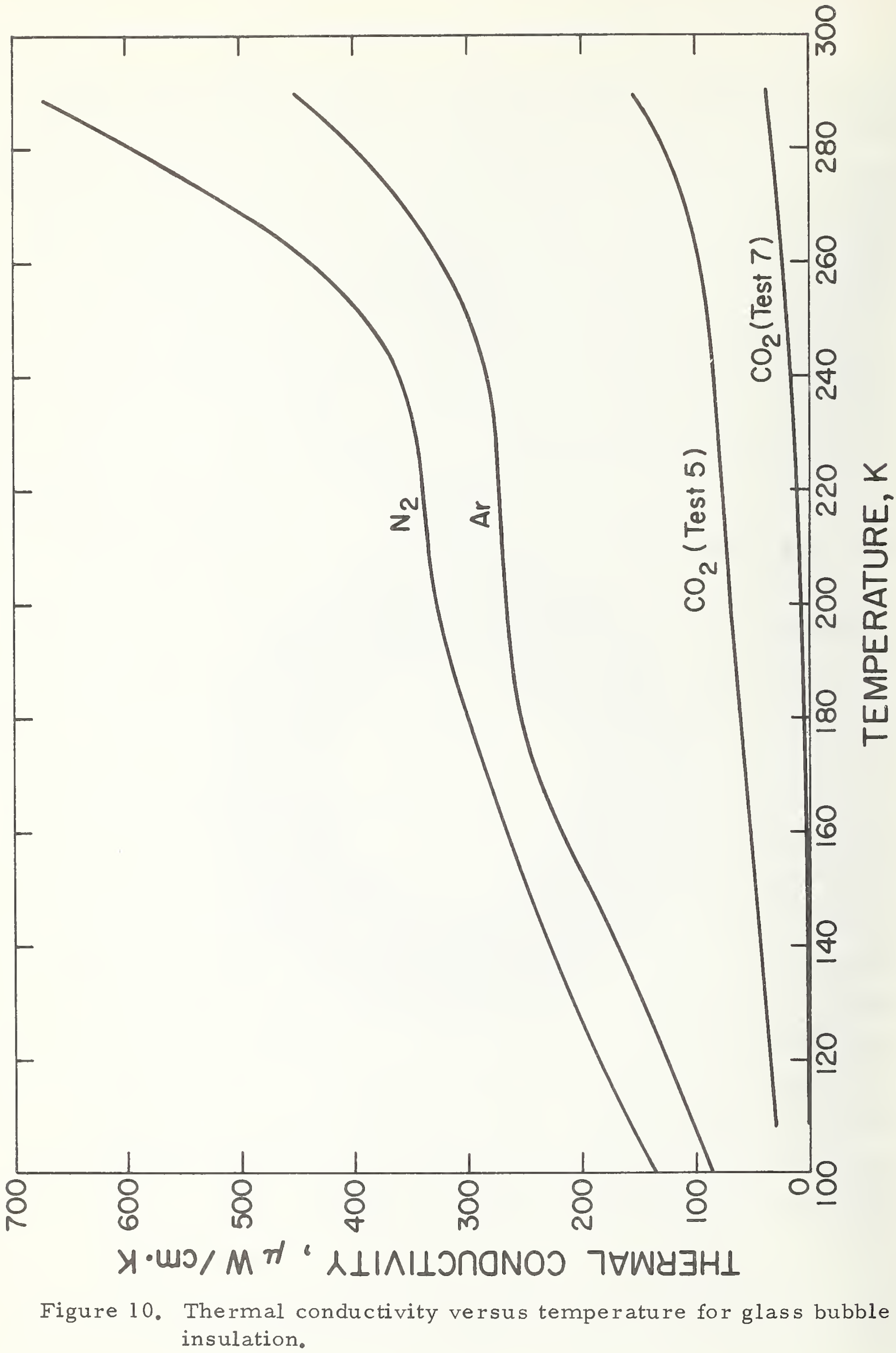

Figure 10. Thermal conductivity versus temperature for glass bubble insulation.

The thermal conductivity for test five, the first test with carbon dioxide, is obviously in disagreement with the data from test seven. This difference in conductivity is assumed to have been caused by the residual argon and nitrogen gas. This assumption cannot be confirmed because the pressure in the insulation space was not measured when the dewar was cold. The insulation was pumped to 0.13 N/m^2 prior to test six which was the second test with vacuum and glass bubbles. The carbon dioxide was again added to a total pressure of 101 kN/m^2 and test seven was run. A vacuum gauge was installed and for this test the dewar insulation space dropped to 0.15 kN/m^2 after the dewar had been cold for about 2 hours. The performance was nearly the same as with a vacuum this time. The average thermal conductivities for the seven tests and the estimated value for pure argon are given in table 3.

Oxygen compatibility tests were made on the glass bubbles used in the seven thermal performance tests and on two other batches of glass bubbles. The first of the other two batches of glass bubbles were identical to those used for thermal tests except that they were not treated with a hydrocarbon washing compound. The second batch were half aluminum coated glass bubbles. The oxygen compatibility tests were impact tests and were performed* by NASA, Marshall Space Flight Center, Huntsville, Alabama. The uncoated glass bubbles were tested in liquid oxygen at one atmosphere pressure. The aluminized bubbles were tested at 1 atmosphere but they were also tested at pressures up to 10.3 MN/m^2 in liquid and gaseous oxygen. No reactions occurred in any of the tests.

The conclusions from the seven tests with glass bubbles as insulation are that: 1) the performance of the glass bubbles in a vacuum is very similar to -80 mesh perlite in a similar vacuum. 2) Carbon dioxide can be used in the insulation space with the glass bubbles without affecting performance and the carbon dioxide will prevent air and moisture

---

* Personal communication with John G. Austin

from entering the insulation while the dewar is warm. 3) Argon gas used with the glass bubbles for insulation of liquid oxygen vessels will degrade performance (about 20 times worse than with a vacuum).
4) Glass bubbles aluminum coated and plain are compatible with oxygen.

Table 3. Average thermal conductivity of glass bubble insulation.

Test	Gas	Insulation Pressure Warm	 Cold	$\bar{K}$ (77 to 295 K)
1	vacuum	0.13 N/m^2	0.13 N/m^2	11 μW/cm-K
2	N_2	101 kN/m^2	87 kN/m^2	259 μW/cm-K
3	80% Ar, 20% N_2	101 kN/m^2	91 kN/m^2	221 μW/cm-K
4	80% Ar, 20% N_2	101 kN/m^2	91 kN/m^2	220 μW/cm-K
	100% Ar		(estimated)	212 μW/cm-K
5	CO_2	101 kN/m^2	~	63 μW/cm-K
6	vacuum	0.13 N/m^2	0.13 N/m^2	11 μW/cm-K
7	CO_2	101 kN/m^2	0.15 N/m^2	13 μW/cm-K

4.0 Proposed Techniques for Extending Service Life of Dewars

4.1 Techniques for Existing Liquid Oxygen Dewars

Dewars that are vacuum insulated or vacuum-and-powder insulated and have developed a vacuum leak can be treated to extend their service life and reduce the necessity of vacuum pumping in the field. The treatment is to be used if the vacuum leak is small, ≤ 0.1 N/m^2 pressure rise per 30 days and if the dewar is used less than 10 percent of the time. The technique consists of pumping the vacuum jacket to as low a pressure as possible, ≤ 0.13 N/m^2 is desirable; then, the vacuum jacket is filled to approximately 14 kN/m^2 gauge pressure with carbon dioxide gas while the dewar is warm. Caution. Do not overpressure the insulation space as the inner vessel may be collapsed. Continued leakage of air and water into the insulation is prevented while the dewar is not in use, but the carbon dioxide will freeze out on the cold walls (cryopump) when the dewar is cooled and result in a low pressure insulation that performs as well as a vacuum. Of course, this salvage is temporary and not acceptable for dewars in continual use. It can be used to keep a dewar in service for an extended period of time while awaiting repair.

If the leak in the vacuum jacket of a powder insulated dewar is greater than 0.1 N/m^2 pressure rise per 30 days or the dewar is in continual use, a temporary fix can also be made but the dewar performance will be degraded. The temporary fix is to pump the vacuum jacket to as low a pressure as possible, at least 1 N/m^2, and to fill the vacuum jacket with argon gas to a maximum of 14 kN/m^2 gauge pressure. This will degrade the insulation but will allow continued use of dewars that are needed and cannot be repaired in the field. The pressure in the insulation will need to be monitored as the argon gas will leak out. When the pressure in the insulation drops below 5 kN/m^2 gauge pressure, argon gas should be added to bring the pressure back to 14 kN/m^2 gauge. Comparative dewar performance for spherical and cylindrical vessels, using argon gas, is shown in figures 11 and 12. The comparative performance presented is calculated from the experimentally measured thermal conductivity and does not include losses due to penetration of the insulation such as fill and vent line or inner vessel supports.

Vacuum insulated liquid oxygen dewars that have small vacuum jacket leaks that exceed the 0.1 N/m^2 pressure rise in 30 days can also be salvaged for temporary use. The method will degrade the performance but in some cases it may be useful to salvage a container and keep it in service for an indefinite time. The procedure for

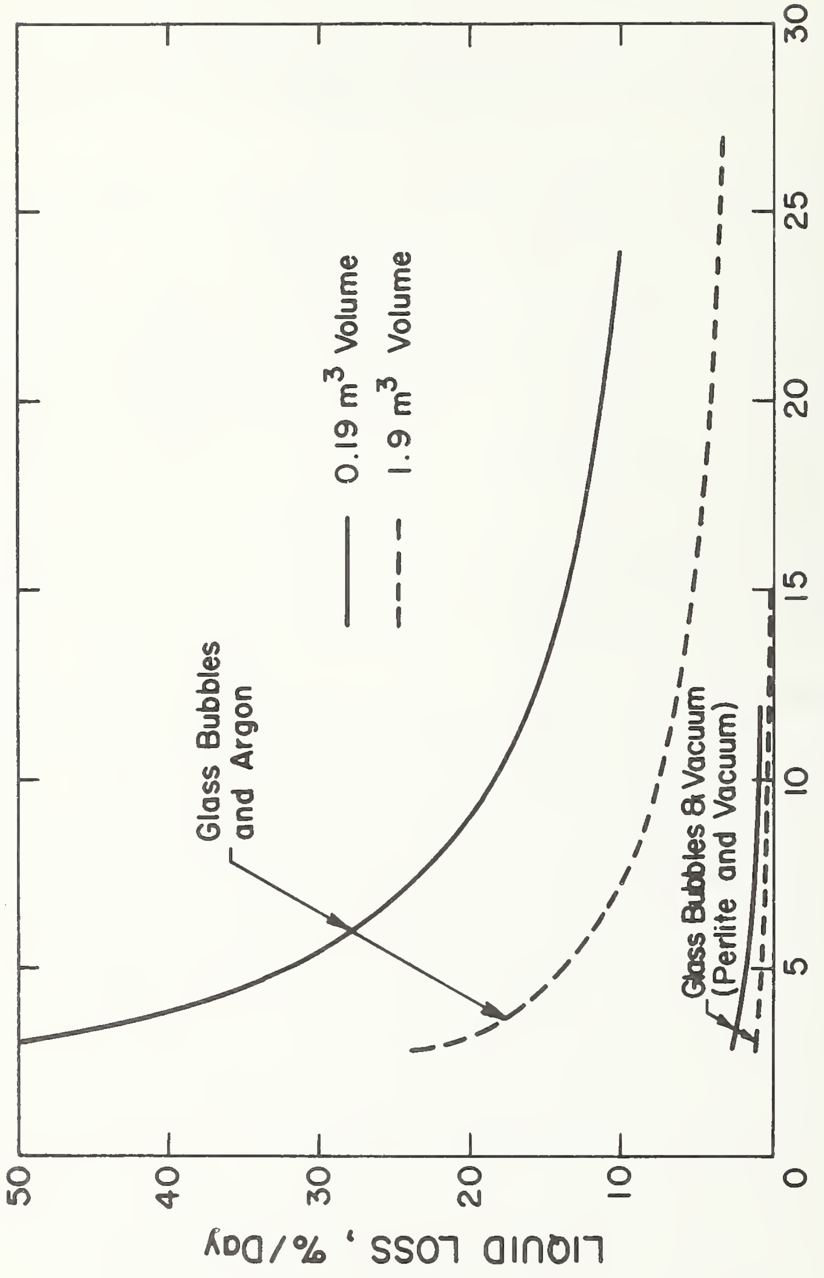

Figure 11. Performance of spherical dewars insulated with glass bubbles.

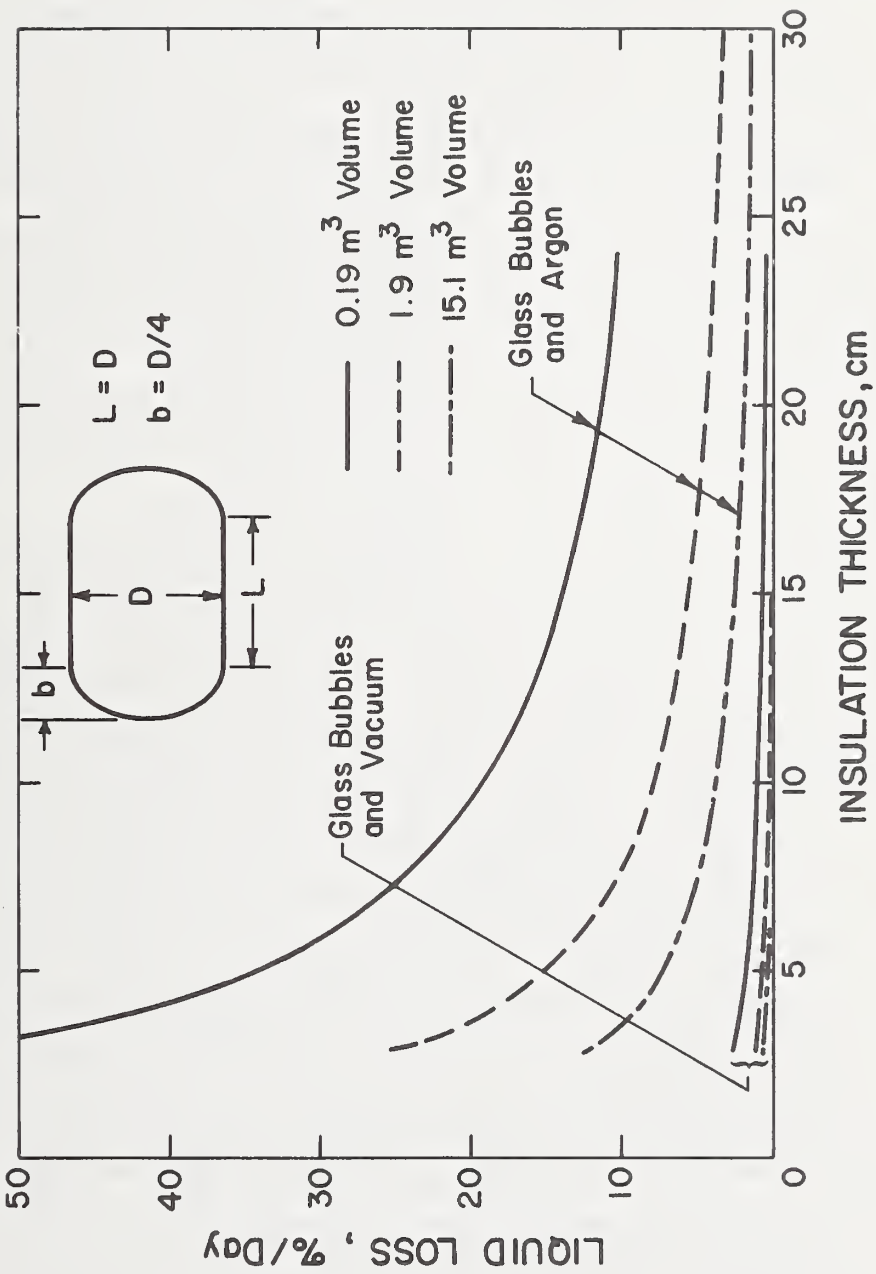

Figure 12. Performance of cylindrical dewars insulated with glass bubbles.

salvaging such dewars is to fill the available insulation space with powder such as perlite or with glass bubbles similar to those described in section 3. Then, the insulation space is pumped to a suitably low vacuum, 1 N/m^2 or less. After vacuum pumping, the insulation space is filled with argon gas to 14 kN/m^2 gauge pressure. The dewar performance will be degraded similar to the comparisons shown in figures 11 and 12. The argon gas pressure will need to be monitored and replenished as in the previous 'fix.' MLI insulated liquid oxygen dewars that have small vacuum jacket leaks can be salvaged for temporary use as well. The thermal performance will also be degraded. The method of salvage is to pump the insulation space to the lowest vacuum possible, $\leq 1\ N/m^2$, then to fill it with argon gas to 14 kN/m^2. Again the performance will be similar to that shown in figures 11 and 12.

A method of repairing dewars with bad vacuum jackets is to remove the vacuum jacket and any associated attaching devices and to cover the vessel with a polyurethane foam using a spray application. Performance of foam insulation is better than glass bubbles with argon gas and has the additional advantage of requiring no vacuum equipment; foam can be applied to any desired thickness, thereby more nearly approaching the original overall dewar performance. Comparative performance of foamed dewars is given in figures 13 and 14. The curves represent calculated performance based on thermal conductivity measured experimentally and do not include losses due to penetrations such as fill and vent lines. The disadvantages of foam are that foam is not oxygen compatible, the application requires removal of the vacuum jacket, special treatment of surfaces to be insulated is necessary, and an intermediate fiber glass shell must be applied with special adaptors for each penetration of the insulation. An exterior cover of fiber glass or metal is required to protect the foam. A complete description of a suitable foam insulation system is given in section 3 except that spray application is recommended rather than pouring.

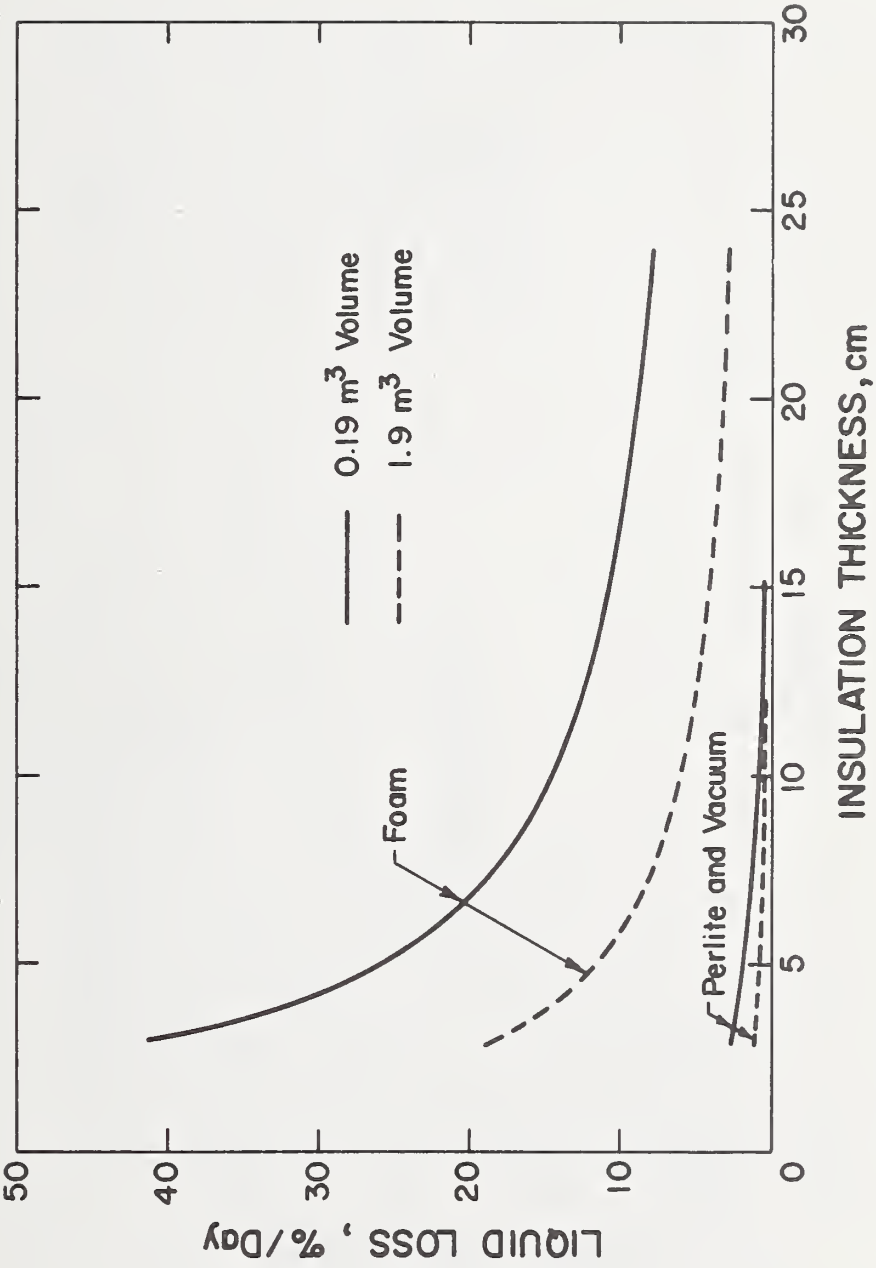

Figure 13. Performance of spherical dewars insulated with polyurethane foam.

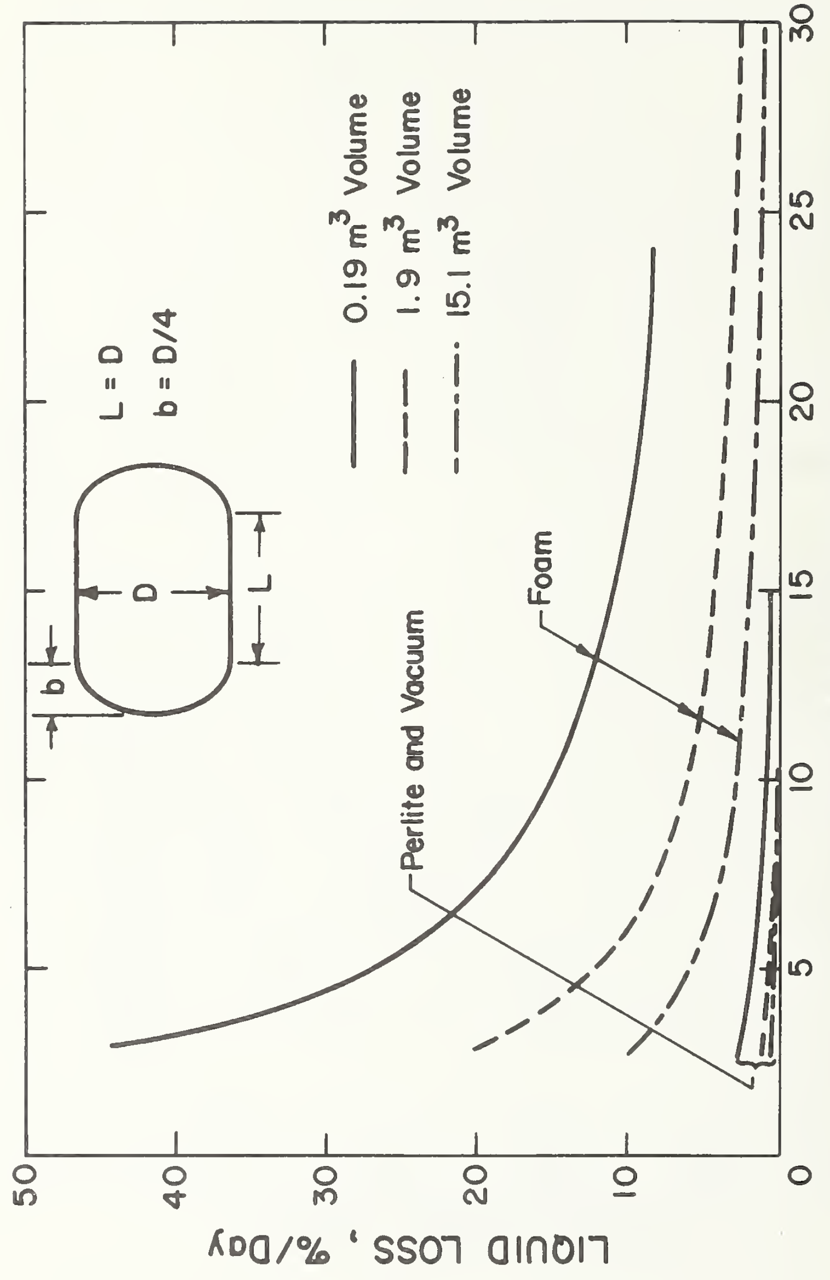

Figure 14. Performance of cylindrical dewars insulated with polyurethane foam.

Foams that can be poured in place may be used in some instances, thus eliminating the requirement for removal of the vacuum jacket; however, the results of tests on foam dewars A and B described in section 3 indicate that considerable development of pouring techniques may be required before a pour-in-place foam could be relied upon for insulation.

4.2 Techniques for New Liquid Oxygen Dewars

Because no new insulation system was conceived that rivals the performance of the vacuum systems, continued use of the vacuum systems is recommended in new vessels. If the vacuum jacket of the system subsequently fails in service, the previously described methods of salvage may be implemented as required. To accommodate these methods, the recommendation is to enlarge the vacuum space in new vessels, where possible, to make the salvage method a more effective insulation.

Foam insulation with an intermediate fiber glass shell and an external protective shell can be considered if the performance is acceptable. The poorer performance of foam may be acceptable for vessels that are used intermittently and for short durations. Also, if the dewar is in continual use (always cold) a thick layer of foam can be applied, if space limitations permit, and the performance is improved. In this case the performance will more nearly approach that of powder-and-vacuum. In either case, the degradation in performance must be balanced with the advantages of a foam insulation system. These advantages are greater durability because of no vacuum requirements, lighter weight, no support structure in the insulation space, and field repairability. Field repairs can be made with a pour-in-place foam and a fiber glass and resin patch.

A cost comparison was made on a foam insulated liquid oxygen system versus a currently used MLI system to see if the foam insulated system had a cost advantage. The cost analysis was provided by a firm producing liquid oxygen dewars for the military. This firm also has some experience with foam insulated dewars. The cost comparison was made on liquid oxygen systems similar to the currently produced Type TMU 70/m which is Navy part number FSN-RX 3655-158-0657-5X-7X. Both systems considered were identical except for the insulation system. The cost reduction for a foam insulated system was estimated to be 25 to 30% or $1000 to $1200 less than the $4000 current production contract costs. These costs are based on an order of 200 items.

A significant disadvantage of the urethane foams is that they generally are not compatible with oxygen--posing a potential safety hazard .

5.0 Recommendations for Further Investigation of Insulation for Liquid Oxygen Dewars

The tests performed have proven that foam can be used as insulation for cryogenic dewars. Since pour foams result in problems in getting uniform density throughout the insulation, spray foams are recommended. A spray foam insulation system similar to the pour foam system used should be tested for thermal performance, for ruggedness, and for aging and thermal cycling effects. Combined with this prototype test should be tests of foam compressive strength, elasticity, thermal contraction, and oxygen compatibility to assure that the spray foam selected is not significantly worse in thermal and mechanical properties than the pour foam we tested.

The use of glass bubbles to replace perlite should be considered because of greater ease in handling and a small gain in performance when in a one atmosphere pressure environment. Glass bubbles that are half aluminum coated are reported to improve insulation performance [Cunnington, 1972] and may be desirable over the complexities of fabrication of MLI even though performance may not be so good as with MLI. Current cost of half aluminum coated glass bubbles is prohibitive; however, the process is in the development stage and price of mass produced bubbles are expected to be competitive with MLI and possibly competitive with powders.

6.0 References

Arnett, R. W., Unpublished report, Substituting polyurethane foam for evacuated perlite insulation in a 2000 gallon liquid oxygen trailer, prepared for Naval Air Engineering Center by the Cryogenics Division, Institute for Basic Standards, NBS, Boulder, Colorado (April 1972).

Cunnington, G. R., and Tien, C. L., Heat transfer in microsphere cryogenic insulation, Paper presented at the Cryogenic Engineering Conference, Boulder, Colorado, August 10, 1972.

Hayes, R. G., Cellular plastics for cryogenic insulation, Cryogenic Technology (July/August 1972).

Honig, R. E., and Hook, H. O., Vapor pressure data for some common gases, R. C. A. Review, Vol. XXI, No. 3, September 1960, David Sarnoff Research Center, Princeton, New Jersey.

Mac, F., and Smith, M., High-Performance spray-foam insulation for application on Saturn S-II stage, Book, Advances in Cryogenic Engineering 16, Ed K. Timmerhaus, pp. 118, 127 (Plenum Press, New York, N. Y., 1970).

Reed, R., Arvidson, J., and Durcholz, R., Mechanical properties of polyurethane and polystyrene foams from 76 to 300 K, Paper presented at the Cryogenic Engineering Conference, Boulder, Colorado, August 10, 1972.

FORM NBS-114A (1-71)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET	1. PUBLICATION OR REPORT NO. NBSIR 73-308	2. Gov't Accession No.	3. Recipient's Accession No.

4. TITLE AND SUBTITLE

INSULATION OF LIQUID OXYGEN DEWARS

5. Publication Date

6. Performing Organization Code

7. AUTHOR(S) Charles F. Sindt

8. Performing Organization NBSIR 73-308

9. PERFORMING ORGANIZATION NAME AND ADDRESS

NATIONAL BUREAU OF STANDARDS, Boulder Labs
DEPARTMENT OF COMMERCE
Boulder, Colorado 80302

10. Project/Task/Work Unit No. 2750554

11. Contract/Grant No. PO 2-8061

12. Sponsoring Organization Name and Address

Naval Air Engineering Center
Philadelphia, Pa. 19112

13. Type of Report & Period Covered Final

14. Sponsoring Agency Code

15. SUPPLEMENTARY NOTES

16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)

The Navy has experienced failure of vacuum insulation in dewars used for storage and handling of liquefied breathing oxygen for aircraft pilots. Because of the vacuum insulation failures, a search was made for a more rugged insulation that has thermal performance similar to the currently used vacuum with multilayer or powder. No system was found that compared in thermal performance and did not require a vacuum. Two systems were experimentally evaluated that did not require vacuum. One was polyurethane foam with an intermediate fiber glass shell and the other was glass bubbles in argon gas at one atmosphere pressure. The polyurethane foam system was successful in that no cracks penetrated to the outside surface; however, the average thermal conductivity was 160 μW/cm-K which is about 15 times greater than vacuum and powder. The glass bubbles in argon gas was also successful since the argon gas pressure always remained high enough to prevent air and moisture from entering the insulation through small leaks in the outer shell. The thermal performance was poorer than the polyurethane foam. The average thermal conductivity was 212 μW/cm-K or about 20 times greater than for the same glass bubbles in a vacuum.

17. KEY WORDS (Alphabetical order, separated by semicolons)

Cryogenic insulation; insulation; LOX dewars; microspheres; polyurethane foam.

18. AVAILABILITY STATEMENT ☒ UNLIMITED. ☐ FOR OFFICIAL DISTRIBUTION. DO NOT RELEASE TO NTIS.	19. SECURITY CLASS (THIS REPORT) UNCLASSIFIED	21. NO. OF PAGES
	20. SECURITY CLASS (THIS PAGE) UNCLASSIFIED	22. Price

USCOMM-DC 66244-P71

USCOMM - ERL