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SUMMARY

Twenty-one leak specimens were fabricated in the ends of stainless-steel and aluminum tubes. Eighteen of these tubes were coated with a copolymer material to seal the leak. The other three specimens were left uncoated and served as control specimens. All 21 tubes were cold shocked in liquid helium 50 times and then the leak rate was measured while the tubes were submerged in superfluid helium at 1.7 K. During the cold shocks two of the coated specimens were mechanically damaged and eliminated from the test program. Of the remaining 16 coated specimens one suffered a total coating failure and resulting high leak rate. Another three of the coated specimens suffered partial coating failures. The leak rates of the uncoated specimens were also measured and reported.

The significance of various leak rates is discussed in view of the Infrared Astronomical Satellite (IRAS) dewar performance.

INTRODUCTION

Objective

This study was undertaken to measure the effectiveness of a copolymer of trifluorochloroethylene and vinyl chloride to seal small leaks in stainless-steel and aluminum pressure vessels filled with superfluid helium at a temperature of approximately 1.7 K. The motivation behind the study was that this material was used to seal a small leak in the Infrared Astronomical Satellite (IRAS) main cryogen tank and a measure of its durability was desired.

Scope

Twenty-one leaks were fabricated in the ends of 0.3175-cm-diameter (0.125-in.) tubing. Eleven of these were from 5052 aluminum alloy and 10 from 304 stainless steel. The specimens were first fabricated, cold shocked in liquid nitrogen 5 times, and then tested at room temperature for their leak rate. After the initial testing, 18 of the specimens were coated with a solution of xylene and a copolymer of trifluorochloroethylene and vinyl chloride while being evacuated internally. After the room-temperature leak rate was remeasured, the specimens were thermally shocked between 4.2 K and room temperature 50 times. The room-temperature leak rate was again measured and the specimens installed in a bath of superfluid helium. The superfluid helium leak rate was measured with the specimens at a temperature of 1.7 K. The results were statistically evaluated and comparisons were made between the coated and uncoated specimens.

APPARATUS

Leak Specimens

Eleven 0.3175-cm-diameter (0.125-in.) by 0.071-cm-wall-thickness (0.028-in.) 5052-0 aluminum alloy tubes were cut to approximately 1.524-m (5-ft) lengths. Two different techniques were used to fabricate very small leaks in one end of these tubes. The first (and unsuccessful) technique was to cold press the tube between a hardened steel block and a hardened steel 0.953-cm (0.375-in.) hex bar while measuring the resulting leak on a helium leak detector. This technique produced leaks of the desired magnitude (1×10^{-7} SCC/sec), but the resulting tube end was pressed to a thickness of approximately 0.0127 cm (0.005 in.) and was very fragile. Only one of the tubes was tested by this technique (AL 3), and it was damaged later during handling and was eliminated from the test program.

The second leak-fabricating technique was successful. In this technique approximately 1.9 cm (0.75 in.) of the tube was flattened in a sheet metal vise. The flattened portion was folded over itself and repressed in the vise. The helium leak rate was measured and then the tube was repressed if necessary to produce a leak in the desired range. Ten leaks in the 0.3175-cm (0.125 in.) aluminum tubes were produced by this second technique.

Ten leaks were fabricated in the ends of 0.3175-cm-diameter (0.125-in.) by 0.051-cm-wall-thickness (0.020-in.) 304 stainless-steel tubes. The technique for fabrication was identical to the second technique used on the aluminum tubes with one exception. It was discovered that the stainless-steel work hardened considerably during the flattening process and a considerable amount of "spring back" was observed. This resulted in higher leak-rate values than desired. To overcome this difficulty, the flattened tubes were heated with an oxygen-acetylene torch to a dull red (approximately 1073 K (800° C)) and repressed in the sheet metal vise while still hot. This technique produced leaks in the desired range. All specimens were identified by filing notches on the tube near the open end.

The aluminum leak specimens are shown in figure 1. The stainless-steel specimens are shown in figure 2.

Coating Material

The coating material used was a solution of 30% by weight of a copolymer of trifluorochloroethylene and vinyl chloride powder (also known as FPC 461) and 70% by weight of xylene (USP). Occasional hand agitation and mild heat (323 K (50° C)) over a 2-hr period was required to dissolve the copolymer powder. The resulting solution is basically clear with a slightly yellowish cast and has a viscosity approximately the same as honey. More information about the material can be found in reference 1.

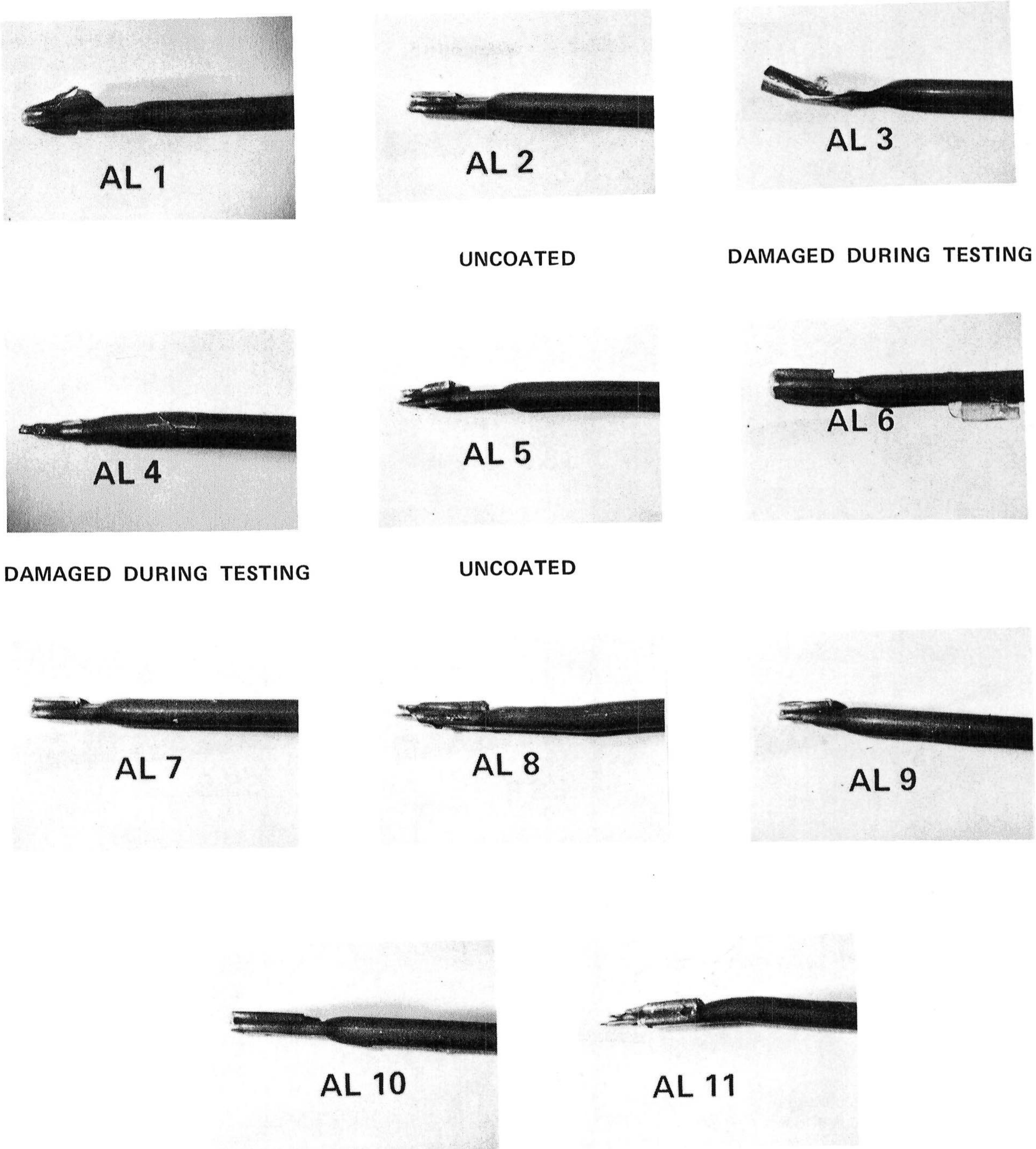

Figure 1.– Aluminum specimens.

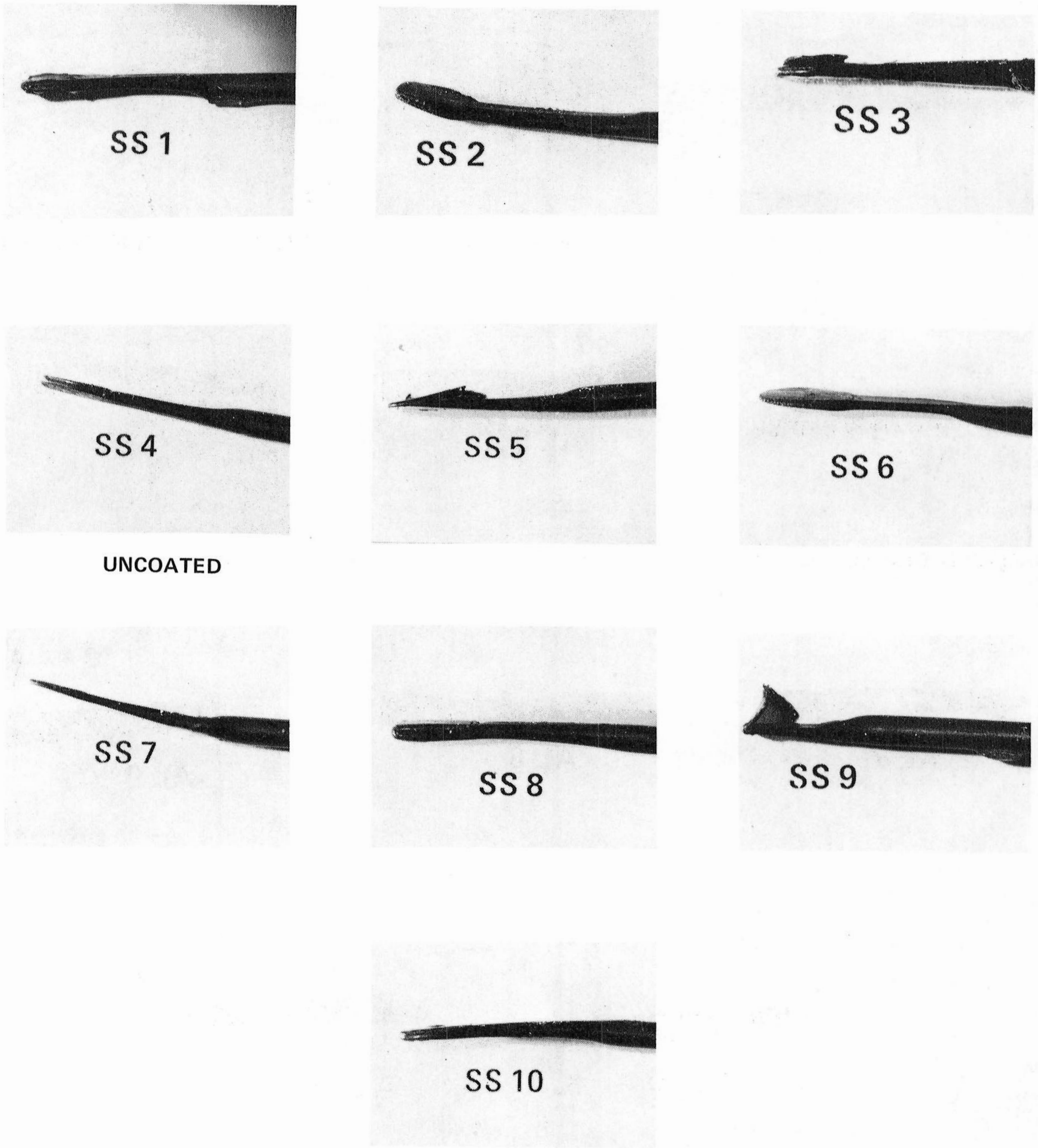

Figure 2.– Stainless-steel specimens.

Leak Detector

The leak detector used was a mass spectrometer type purchased in 1977. It was calibrated daily with a calibrated standard leak. The leak detector output was compared with two other calibrated leaks and all values were well within the ±10% accuracy stated for the calibrated leak. The leak detector has a range from a maximum of 7.9×10^{-6} SCC/sec to the minimum background available, which varied from a high of 2.5×10^{-10} SCC/sec to a low of 1.3×10^{-10} SCC/sec. The background varied because the tests were performed in a cryogenic laboratory in which helium dewars were venting and liquid helium transfers were occasionally occurring. On these occasions the helium background increased considerably because of backstreaming of the helium through the leak-detector vacuum pumps.

Flowmeters

For the higher value leaks two helium flowmeters were used. One had a usable range of 0.05 SCC/min to 10.0 SCC/min or 8.3×10^{-4} SCC/sec to 1.6×10^{-1} SCC/sec. The other flowmeter had a usable range of 0.5 SCC/min to 50 SCC/min or 8.3×10^{-3} SCC/sec to 8.3×10^{-1} SCC/sec. Both flowmeters were factory calibrated for use with helium. These calibrations are shown in table 1.

Pressure Gauge

The primary pressure gauge (a 0–50-mm gauge) was calibrated as shown in table 2.

Flow Network

As mentioned previously, the leak detector measured leaks from 1.3×10^{-11} SCC/sec to 7.9×10^{-6} SCC/sec and the flowmeters measured flows between 8.3×10^{-4} SCC/sec to 8.3×10^{-1} SCC/sec. Therefore, there was no *direct* measure of leaks in the range 7.9×10^{-6} to 8.3×10^{-4}. Measurements in this range were accomplished by splitting the flow between the small vacuum pump VP2 and the vacuum pumps in the leak detector with the flow network shown in figure 3. The needle valves V1N and V2N were adjusted to split the flow. The ratio of the flow split was calibrated by opening valve V4 to the standard leak and recording the response of the leak detector.

TABLE 1.– HASTING-RAYDIST FLOWMETER

Meter reading, SCC/min	Standard mass flow, SCC/min	% Error of reading	% Full-scale error
(a) Mod: NALL log; S/N: 5182; Range: 0-10 SCC/min; Temp: 21° C (294 K); Atmospheric pressure: 30.3 INHG; Gas used: Helium			
0.57	0.572	-0.35	0.02
1.03	1.25	-17.6	-2.20
2.00	2.17	7.8	-1.70
3.06	3.24	-5.56	-1.80
4.05	4.26	-4.93	-2.10
5.01	5.23	-4.21	-2.20
6.04	6.30	-4.13	-2.60
6.99	7.23	-3.32	-2.40
8.05	8.32	-3.25	-2.70
9.05	9.28	-2.48	-2.30
9.97	10.31	-3.40	-3.40
(b) Mod: NALL 50G; S/N: 5183; Range 0-50 SCC/min; Temp: 20° C (293 K); Atmospheric pressure: 30.3 INHG; Gas used: Helium			
4.8	4.65	3.23	0.3
10.2	9.43	8.17	1.54
15.1	14.55	3.78	1.10
20.1	19.56	2.76	1.08
25.1	24.80	1.21	.60
29.8	29.45	1.19	.70
34.9	34.44	1.34	.92
39.8	39.23	1.45	1.14
44.7	44.18	1.18	1.04
50.0	49.38	1.24	1.24

TABLE 2.– PRESSURE CALIBRATION
[Wallace & Tiernan, S/N AC12165; NASA no. 59868;
Range: 0-50 mmHg]

Point no.	Absolute pressure	Standard pressure	Error, mmHg	% Full scale error
1	0.0	0.000	0.000	0.00
2	5.0	5.231	.231	.46
3	10.0	10.144	.144	.29
4	15.0	15.166	.166	.33
5	20.0	20.246	.246	.49
6	25.0	25.363	.363	.73
7	30.0	30.301	.301	.60
8	35.0	35.444	.444	.89
9	40.0	40.602	.602	1.20
10	45.0	45.549	.549	1.10
11	50.0	50.719	.719	1.44
12	45.0	45.572	.572	1.14
13	40.0	40.508	.508	1.02
14	35.0	35.358	.358	.72
15	30.0	30.156	.156	.31
16	25.0	25.224	.224	.45
17	20.0	20.217	.217	.43
18	15.0	15.070	.070	.14
19	10.0	10.195	.195	.39
20	5.0	5.165	.165	.33
21	.0	.000	.000	.00

Note: Indicator needle less than zero at 0.0 mmHg.

Vacuum Pump and Filter

The small vacuum pump (VP2) identified in figure 3 was a 0.9 cfm pump with a blank-off pressure of 30 μm of Hg. The pump discharged into a water/air filter filled with 2.37 lbm of molecular sieve, and then into one of the two flowmeters or directly into the atmosphere. The filter was an oil and water filter. Quoting from the manufacturer's catalog, "the [filter] dehydrates to dew-points less than –105° F and simultaneously removes hydrocarbons to less than 1 PPM/w (hexane equivalent) and oil vapor and most noxious gases to less than 1 PPM/w." The pump-filter-flowmeter combination worked quite well except when large amounts of air or helium went through the vacuum pump. On those occasions, the heat of compression would slightly heat the molecular sieve in the filter. When the flow dropped to the small flows associated with the specimens, the temperature of the sieve would cool, causing the flowmeter to read negative as air was sucked back into the filter to equalize the pressure. The same effect was noted when there was a local drop in room temperature as a result of filling the liquid nitrogen trap in the leak detector or filling the liquid nitrogen tank in the helium test dewar. In general, the flow would take 20 min to 1 hr to stabilize after one of these molecular sieve temperature excursions.

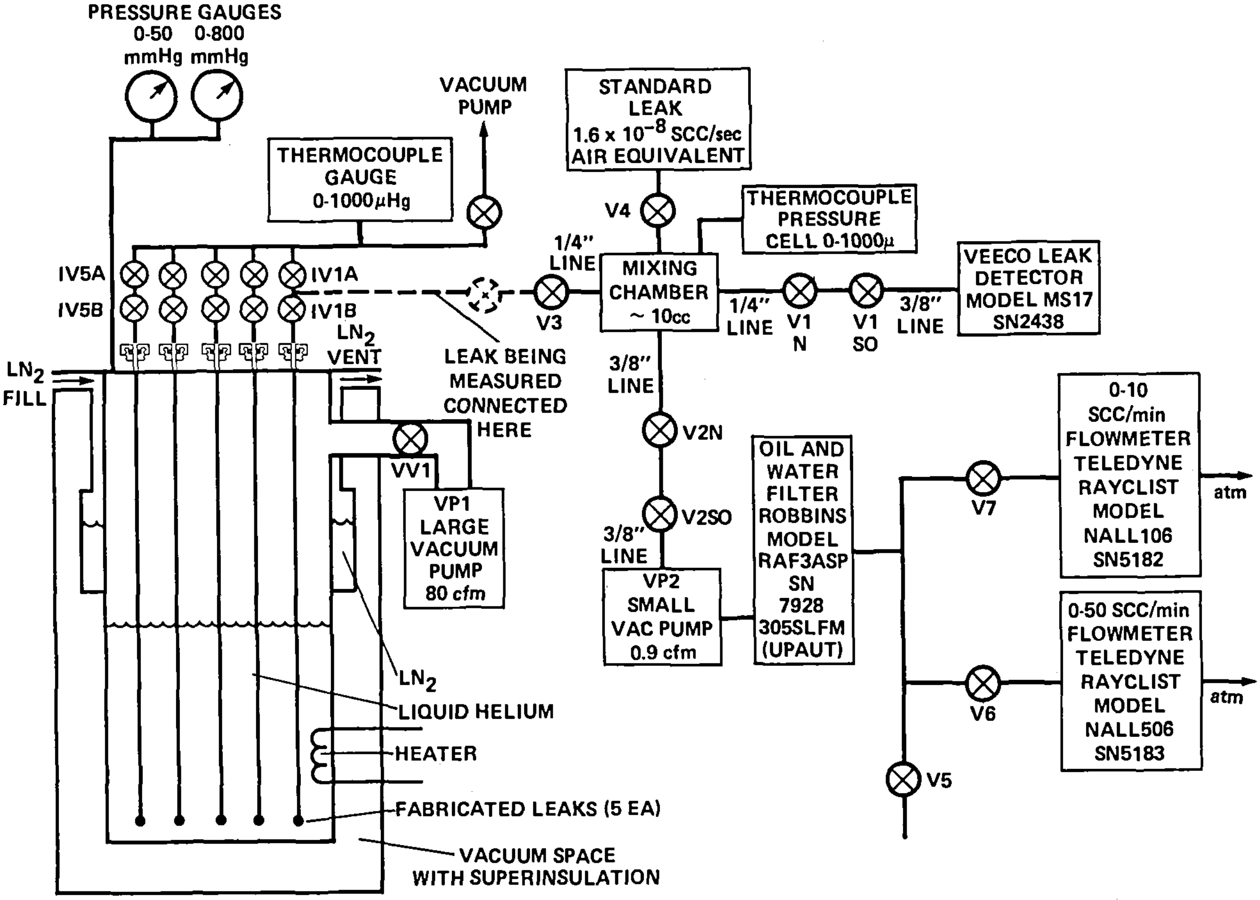

Figure 3.– Flow network.

Test Dewar and Test Fixture

The test dewar used for this investigation was an open mouth dewar with a 15.24-cm (6-in.) inside diameter. The liquid helium capacity was 13 liters and the liquid nitrogen capacity was 8 liters. The hold time with five stainless-steel specimens was approximately 70 hr. The hold time with five aluminum specimens was somewhat less, approximately 60 hr. The test fixture with five test specimens is shown in figure 4. The test fixture consisted of a brass flange with "O" ring connectors through which the tube could be inserted for testing. Four radiation shields were suspended from the brass flange with thin woven fiberglass tubing.

Cold-Shock Apparatus and Procedure

A 50-liter cryogenic supply dewar with a 1.905-cm-diameter (0.75-in.) neck opening was used in cold shocking the tubes. The tubes were bundled together in groups of four or five and lowered through the dewar neck until they contacted the bottom. They went from room temperature to 4.2 K in approximately 30 sec. After the helium boiloff dropped back to a low value the specimens were removed and quickly placed in an insulated cardboard mailing tube. Dry nitrogen at a

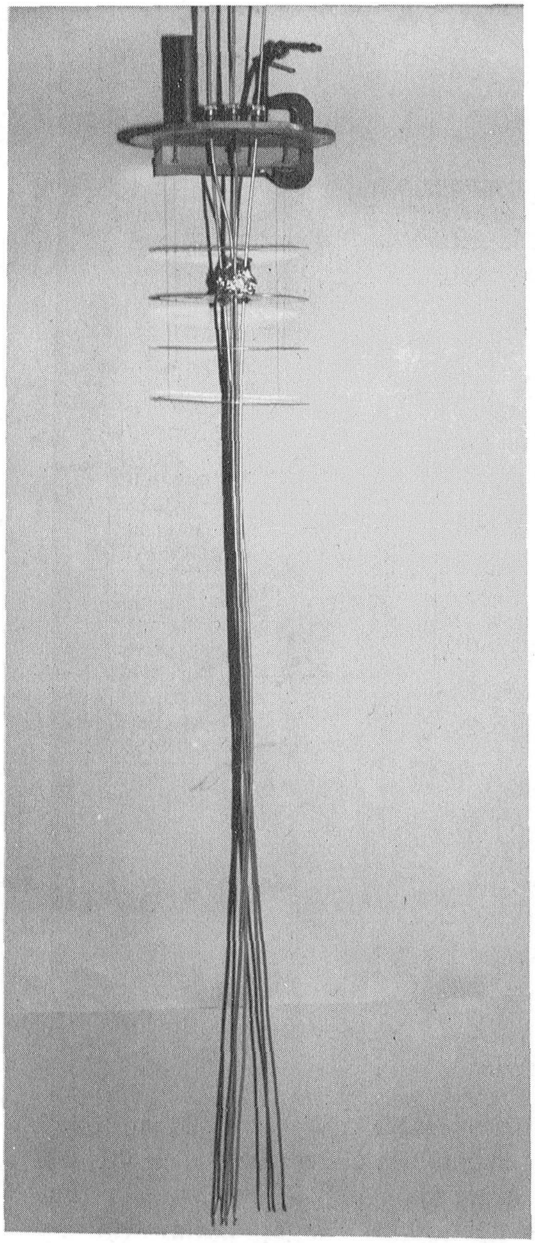

Figure 4.– Test fixture.

temperature of approximately 323 K (50° C) was injected into the bottom of the tube to keep out water vapor and speed the warmup process. Approximately 40 min were required to bring the tubes back to room temperature. The open ends of the tubes were covered with surgical tubing to prevent moisture and frozen air from collecting on the insides of the tubes. During these cold shocks two of the aluminum leak specimens were damaged. The tube made of AL 3, which was the only one tested by the first technique, was badly bent when it contacted the bottom of the 50-liter cold shock dewar. It was straightened but not included in the test program. Another of the aluminum tubes, AL 4, was also eliminated from the test program when it snagged and broke on the dewar neck as it was being removed from the 50-liter cold shock dewar.

TEST PROCEDURE

Coating Procedure

After fabricating the leaks, each tube was individually cleaned. The cleaning procedure consisted of a vigorous 20-sec hand agitation in methel ethyl ketone (MEK) and then a vigorous 20-sec hand agitation in a solution of 95% ethyl alcohol and 5% distilled water. The tubes were then allowed to air dry for several hours prior to coating. To coat the leaks, the open ends of the tubes were connected to a vacuum pump and were evacuated to a pressure of less than 10^{-4} Torr. The leak ends of the tubes were then immersed approximately 2.54 cm (1 in.) in the copolymer/xylene solution and held there for exactly 30 sec. They were then removed and placed on a drying rack for 64 hr at room temperature. The specimens were not precoated as recommended by the coating manufacturer because an attempt was made to match the procedure used on the IRAS tank leak.

Room-Temperature Leak-Measurement Procedure

The leak measurements at room temperature were made by individually connecting the open ends of the tubes directly to the leak detector. The fittings on the leak detector were then sprayed with helium to detect leaks and tightened if necessary. The leak ends of the tubes were inserted about 30 cm (11.8 in.) into a 0.95-cm-diam (0.375-in.) flexible plastic tube through which approximately 1 SCC/sec of helium was flowing. The leak detector typically responded within seconds and when the indicated value did not change for a 2-min time period the value was recorded and the procedure repeated for the next specimen.

Superfluid Leak-Measurement Procedure

The measurement of the superfluid leak rate was very difficult and time consuming. Several different techniques and procedures were tried before the following was selected. After the leak specimens were assembled in the test fixture and inserted in the test dewar both the dewar itself and the specimens were evacuated to a pressure of less than 100 μm of Hg. The test dewar was then backfilled to atmospheric pressure with dry, oil-free helium and then evacuated a second time. It was then backfilled to a pressure of 700 Torr Hg with helium. The liquid nitrogen jacket on the dewar was then filled and the entire assembly allowed to cool until the pressure dropped to 500 Torr. This cool-down to liquid nitrogen temperature took approximately 2 hr.

After the cool-down to liquid nitrogen temperature the contents of a 25-liter liquid helium storage dewar (containing approximately 20 liters) were transferred into the test dewar. The liquid in the test dewar was typically 25 cm (9.84 in.) deep at the end of this transfer. After the liquid helium transfer the test dewar was pumped down slowly to 10 Torr over a 2- to 3-hr time period. The leak specimens were connected one at a time to the flow network shown in figure 3. Initially, valves V3, V2N, V2SO, and V7 were opened and all others closed. When the flowmeter output dropped to less than 0.1 SCC/min the valve identified on figure 1 as IV1B (or whichever leak specimen was being measured) was opened. At this time one of several things would happen. If the leak specimen had a fairly large leak, the flowmeter output increased to very high values (20 to 30 SCC/m) as the accumulated helium in the tube was pumped out. If this occurred, it generally required a long pumping period (10 to 16 hr) before a leak measurement could be taken. If the leak specimens had only a small leak, the flow rate dropped to less than 0.01 SCC/min in a few minutes and stayed low. Several hours of pumping on the leak specimen were generally required prior to a measurement to confirm the absence of air or water ice inside the tube. For specimens with larger leaks (such as SS 4) the leak rate could be measured directly with the flowmeter. Generally, however, the flowmeter output dropped to a value of 0.01 ±0.01 SCC/m, which was approximately the drift rate of the instrument.

In cases where the leak rate was below the range of the flowmeter, the flow was diverted directly to the leak detector by closing valve V2SO and opening valves V1N and V1SO. If the specimen leak rate was less than 7.93×10^{-6} SCC/sec of helium, the leak could be measured directly with the leak detector. If the specimen leak rate was greater than 7.9×10^{-6} SCC/sec but below the minimum range of the flowmeters (8.3×10^{-4} SCC/sec), the flow was split between the small vacuum pump (VP2) and the vacuum pumps in the leak detector. The procedure for this split-flow technique was first to open valve V2N (needle valve) approximately two full turns and fully open valve V2SO (shutoff valve). After the pressure stabilized in the mixing chamber, valve V1SO was fully opened and valve V1N was opened until the leak detector read significantly, above the background. When the leak-detector output stabilized, a process which often took 2 to 3 hr, the output was recorded and then the flow split calibrated.

To calibrate and measure the flow split between VP2 and the leak-detector vacuum pumps, valve V3 was closed to shut off the flow from the leak specimen. When the leak-detector output was stable the output was recorded as the tare value, and then valve V1SO was closed and valve V4 to the calibrated standard leak was opened. Because of a finite volume between V4 and the actual calibrated leak, a substantial amount of helium was ingested into the mixing chamber and then pumped out by VP2. After the pressure again stabilized in the mixing chamber, valve V1SO again was fully opened, thus theoretically splitting the flow from the standard leak in the same way it was split from the leak specimen. The detector output was then recorded as the gross value. The actual flow split was calculated by using the formula:

$$\text{flow split} = X{:}1$$

$$X = \frac{(\text{calibrated leak output } 4.23\times 10^{-6}\ \text{SCC/sec})}{\begin{bmatrix}\text{leak detector out-}\\ \text{put}\\ \text{(gross value)}\end{bmatrix} - \begin{bmatrix}\text{leak detector out-}\\ \text{put}\\ \text{(tare value)}\end{bmatrix}}$$

The flow-split ratios varied from 3.2:1 to 80:1.

The accuracy of the measurements made by the direct-flowmeter technique was approximately ±5% of the absolute value. That for measurements made by the leak detector was approximately ±20% of the absolute value. The accuracy of the split-flow technique was much lower; it is estimated at ±100% of the absolute value.

Of the 19 leak specimens, 4 were measured by the direct-flowmeter technique, 9 were measured directly on the leak detector, and the remaining 6 required the flow-splitting technique.

RESULTS AND DISCUSSION

Coated Specimens

Table 3 shows the measured leak rates of the 16 coated specimens before being coated, after being coated, and after 50 liquid-helium cold shocks. Also given are the superfluid helium leak rates at 1.74 K. All the leak rates, except the superfluid leak rate, were measured at room temperature.

Because no two of the coated tubes behaved the same, the results can be viewed from a statistical viewpoint or from a percentage viewpoint. The author prefers the statistical viewpoint.

The 16 tubes originally had a median leak rate of 1.35×10^{-7} SCC/sec. The coating process reduced this value considerably by a factor of 34 to a median rate of 4.0×10^{-9} SCC/sec.

The 50 thermal shocks from room temperature to 4.2 K had only a slightly negative effect, increasing the median value by a factor of 1.05 to 4.2×10^{-9} SCC/sec. At this point in the testing the distribution of the leak valves became very erratic because of the performance of two individual specimens. The sealant material on the specimens identified as SS 1 and SS 2 obviously failed because the leak rates for these two specimens were higher after the thermal shocks than they were prior to coating. Specifically, the leak rate of specimen SS 1 increased by a factor of 150 to a value slightly higher than its precoating value. The leak rate of specimen SS 2 increased by a factor of 6.5

TABLE 3.– COATED SPECIMEN RESULTS

Tube no.		Leak rate before coating, SCC/sec	Leak rate after coating, SCC/sec	Leak rate after 50 cold shocks, SCC/sec	Superfluid leak rate at 1.74 K, SCC/sec	Standard deviations from mean
Aluminum	AL 1	3.7×10^{-6}	4.7×10^{-9}	3.9×10^{-9}	1.3×10^{-8}	-0.26
tubes	AL 6	1.1×10^{-6}	4.2×10^{-9}	4.4×10^{-9}	$<1.6\times10^{-9}$	-.26
	AL 7	3.9×10^{-6}	3.7×10^{-9}	3.1×10^{-9}	4.2×10^{-7}	-.26
	AL 8	2.0×10^{-7}	$<2.1\times10^{-9}$	$<1.6\times10^{-9}$	2.9×10^{-8}	-.26
	AL 9	6.1×10^{-9}	$<1.7\times10^{-9}$	$<1.3\times10^{-9}$	6.3×10^{-8}	-.26
	AL 10	1.3×10^{-7}	$<1.7\times10^{-9}$	$<1.6\times10^{-9}$	4.7×10^{-7}	-.26
	AL 11	2.1×10^{-8}	$<1.6\times10^{-9}$	$<1.5\times10^{-9}$	1.3×10^{-7}	-.26
Stainless-	SS 1	1.5×10^{-6}	1.2×10^{-8}	1.8×10^{-6}	3.9×10^{-2}	3.8
steel	SS 2	1.1×10^{-6}	7.6×10^{-7}	5.0×10^{-6}	2.5×10^{-5}	-.25
tubes	SS 3	2.3×10^{-6}	3.8×10^{-7}	6.6×10^{-7}	4.4×10^{-4}	-.21
	SS 5	$<1.3\times10^{-9}$	$<1.7\times10^{-9}$	5.5×10^{-8}	3.1×10^{-9}	-.26
	SS 6	7.4×10^{-8}	2.3×10^{-8}	8.4×10^{-8}	7.1×10^{-6}	-.26
	SS 7	1.4×10^{-7}	4.2×10^{-9}	8.2×10^{-9}	5.2×10^{-9}	-.26
	SS 8	1.1×10^{-8}	$<2.1\times10^{-9}$	$<1.3\times10^{-9}$	4.2×10^{-6}	-.26
	SS 9	$<1.3\times10^{-9}$	$<2.0\times10^{-9}$	2.5×10^{-9}	1.3×10^{-5}	-.26
	SS 10	7.1×10^{-8}	2.6×10^{-8}	8.9×10^{-8}	4.7×10^{-9}	-.26
Mean		8.9×10^{-7}	7.7×10^{-8}	4.8×10^{-7}	2.4×10^{-3}	
Standard deviation		1.3×10^{-6}	2.0×10^{-7}	1.2×10^{-7}	9.4×10^{-3}	
Median		1.35×10^{-7}	4.0×10^{-9}	4.2×10^{-9}	2.8×10^{-7}	

to a value about 5 times higher than its precoating leak rate. One reason for the failure of the sealant material to survive the cold-shock tests is the thermal contraction mismatch between the coating material and the tube material. Although there is no specific data on the thermal contraction of the coating material, similar materials (as KEL-F) show a larger contraction at cryogenic temperatures than either the stainless steel or the aluminum. This mismatch in the contraction at cryogenic temperatures plus the poor adhesion to the metal surface caused the coating to flake off parts of the specimens, as can be seen in figures 1 and 2. It is assumed that this mismatch and poor adhesion, as observed on a large scale in the photograph, likely occurred on a small scale in the leak passageway itself.

As was expected, the mean leak rate of the 16 specimens increased significantly when they were exposed to superfluid helium. The mean leak rate increased by a factor of 500 to a value of 2.4×10^{-3} SCC/sec. The median value, which is probably a more realistic measure of the group performance, increased by a factor of 66 to 2.8×10^{-7} SCC/sec. Again, as with the previous cold-shock results, the results were significantly skewed by the performance of a few individual specimens. Specimen SS 1 showed an increase 2.2×10^{4} in its leak rate under superfluid conditions. Such an increase might be considered an incorrect measurement except when viewed against the results for the uncoated tubes discussed in the next section. The uncoated tubes showed very similar huge increases when exposed to superfluid helium.

When viewed on a percentage basis, it can be said of the original specimens when measured at room temperature:

19% had leak rates less than 1×10^{-8} SCC/sec
44% had leak rates less than 1×10^{-7} SCC/sec
63% had leak rates less than 1×10^{-6} SCC/sec
100% had leak rates less than 1×10^{-5} SCC/sec

The effect of the coating reduced the leak rate so that when they were measured at room temperature:

69% had leak rates less than 1×10^{-8} SCC/sec
81% had leak rates less than 1×10^{-7} SCC/sec
100% had leak rates less than 1×10^{-6} SCC/sec

The cold shocks generally increased the specimen leak rate so that when they were measured at room temperature:

63% had leak rates less than 1×10^{-8} SCC/sec
81% had leak rates less than 1×10^{-7} SCC/sec
87% had leak rates less than 1×10^{-6} SCC/sec
100% had leak rates less than 1×10^{-5} SCC/sec

Finally, the superfluid helium exposure significantly increased the specimen leak rate to the point that when measured at 1.7 K:

25% had leak rates less than 1×10^{-8} SCC/sec
43% had leak rates less than 1×10^{-7} SCC/sec
63% had leak rates less than 1×10^{-6} SCC/sec
75% had leak rates less than 1×10^{-5} SCC/sec
88% had leak rates less than 1×10^{-4} SCC/sec
93% had leak rates less than 1×10^{-3} SCC/sec
93% had leak rates less than 1×10^{-2} SCC/sec
100% had leak rates less than 1×10^{-1} SCC/sec

This percentage viewpoint is shown graphically in figure 5.

Uncoated Specimens

The three uncoated tubes were included in the test as control samples. They were tested in the same manner as the coated specimens. For the superfluid leak tests the uncoated tubes were tested in the same dewar lot as the coated specimens.

Table 4 shows the measured leak rates of the three uncoated specimens before being cold shocked, after being cold shocked to 4.2 K 50 times, and during exposure to superfluid helium. All the leak rates were measured at room temperature except the superfluid helium leak rates, which were measured at 1.74 K.

Statistically, the original mean and median values of the leak rates of the uncoated specimens were 8.5×10^{-7} SCC/sec and 1.1×10^{-6} SCC/sec, respectively. The 50 cold shocks to 4.2 K had a slightly negative effect and increased the mean and median values to 2.3×10^{-6} SCC/sec. The after-cold-shock leak rate of tube AL 5 was not accurately measured because the split-flow technique had not been developed at that point in the investigation.

The mean and median leak rates of the three specimens when exposed to superfluid helium at 1.7 K were 7.9×10^{-2} SCC/sec and 7.8×10^{-2} SCC/sec, respectively. It should be noted that this superfluid-leak-rate mean value for the uncoated tubes is quite close to the value for the coated tube SS 1, which demonstrated a coating/sealant failure.

The uncoated specimens showed a substantial change in leak rate for various superfluid pressures, as one would expect from a classical two-fluid superfluid viewpoint. This change with pressure was measured for tube SS 4 over a wide range of superfluid helium pressures and is shown in figure 6.

The data taken at this slower pressure-change rate show a more dramatic change in leak rate and reflect a more accurate measure of the "true" leak as a function of pressure because of the time constant of the measuring system.

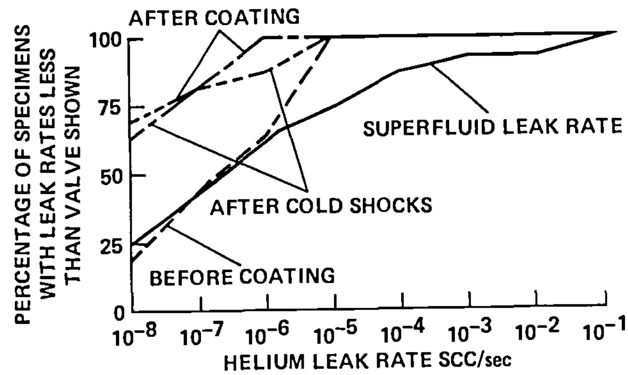

Figure 5.– Coated specimen leak rate integrated distribution.

TABLE 4.– UNCOATED LEAK SPECIMEN RESULTS

Tube no.	Leak rate, SSC/sec		
	Before cold shocks	After 50 cold shocks	Superfluid helium leak rate
AL 2	1.1×10^{-6}	1.9×10^{-6}	8.7×10^{-3}
AL 5	1.6×10^{-7}	$<7.9 \times 10^{-6}$ [a]	1.5×10^{-1}
SS 4	1.3×10^{-6}	2.6×10^{-6}	7.8×10^{-2}
Mean	8.6×10^{-7}	2.3×10^{-6}	7.9×10^{-2}
Standard deviation	4.9×10^{-7}	3.5×10^{-7}	5.7×10^{-2}
Median	1.1×10^{-6}	2.3×10^{-6}	7.8×10^{-2}

[a]Not used in considering mean, standard deviation, or median.

INVESTIGATION RESULTS COMPARED WITH IRAS TANK LEAK

Brief History

On May 31, 1979, a leak of 4.8×10^{-8} SCC/sec was discovered in the IRAS main cryogen tank when it was filled with superfluid helium at a pressure of 12 mmHg. The leak location could not be pinpointed at the time. Suspected areas were painted with the coating material used in this study (a copolymer of trifluorochloroethylene and vinyl chloride dissolved in xylene), and the suspected areas were thereby narrowed down to eight welds (four of which were in the nonflight test fixture). When the coating material was removed from the welds one at a time, the leak did not reappear.

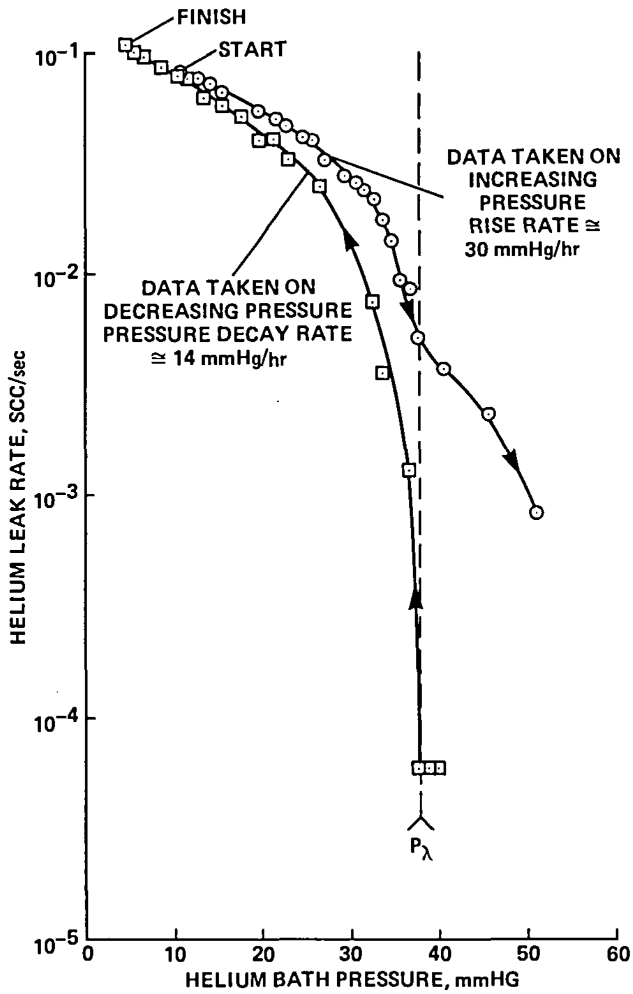

Figure 6.– Leak rate as a function of pressure for tube SS 4 – uncoated.

The weld areas were then recoated and the IRAS dewar construction continued. This investigation was undertaken to identify the subsequent probability of recurrence of the leak.

The problem was also attacked from another angle by installing approximately 50 g of activated charcoal in two screen-covered cups on the main cryogen tank near the leak area.

When the IRAS tank was coated, the leak reportedly dropped below the measurement threshold of 5×10^{-9} SCC/sec. It did not increase above that threshold when the tank was exposed to superfluid helium at 1.7 K. That particular behavior was demonstrated by only 4 of the 16 coated specimens in this investigation. Perhaps a significant difference between the IRAS tank and the leak specimens investigated here was that the test specimens underwent 50 cold shocks prior to the superfluid helium leak-rate measurement while the IRAS tank underwent only one cold shock. However, it is expected that the IRAS tank will be cold shocked up to 20 to 30 times before it is launched.

The exact geometry of neither the leaks described herein nor the IRAS leak is known. The IRAS leak (hole) has been estimated as being a hole approximately 1 μm (3.9×10^{-5} in.) in diameter. The leak specimens described herein have leak rates both greater than and less than the IRAS leak rate and, therefore, must have hole diameters (or multiple leak paths) both larger and smaller than 1 μm.

Several differences exist between the test specimens described herein and the current configuration of the IRAS leak region. First, the leaks described in this report were formed by forging, and it is postulated that the IRAS leak is in a welded seam. The differences are probably insignificant for the aluminum specimens but may be significant for the stainless-steel specimens. The stainless-steel specimens were heated and then forged; this process oxidized the surface more than would be expected in a welded seam. Second, the suspected leak region on the IRAS tank was cleaned, precoated with the recommended primer, and overcoated with a 15% copolymer/xylene solution. This overcoating would likely reduce the leak.

Comparison

The results of this investigation indicate that a much reduced IRAS leak still exists at a level below the threshold of the leak measuring system used. If we let the median value of the after-coating test leak rate represent the IRAS leak rate, then the IRAS leak rate is approximately 4.0×10^{-9} SCC/sec and under superfluid conditions should increase to approximately 2.8×10^{-7} SCC/sec after the IRAS tank undergoes a series of vibrations and cold shocks. The process of overcoating will likely reduce the leak.

The activated charcoal should be able to adsorb the leaking helium with little degradation of the dewar guard vacuum. Data by Wiederman and Smolic (ref. 2) indicate that charcoal will adsorb approximately 8×10^{-2} grams of helium per gram of charcoal at 4.2 K and 10^{-6} Torr. If we assume a 2-wk period with no pumping prior to launch and a 2-wk period in orbit prior to cover ejection, the 50 g of charcoal in the IRAS getter should (with 100% efficiency) be expected to adsorb a leak as calculated by the following equation:

$$\left.\begin{aligned} \dot{M} &= \left(\frac{8 \times 10^{-2} \text{ g of He}}{\text{grams of charcoal}}\right)\left(\frac{50 \text{ g of charcoal}}{2.4 \times 10^{6} \text{ sec}}\right)\left(\frac{\text{SCC}}{1.625 \times 10^{-4} \text{ g}}\right) \\ \dot{M} &= 1.025 \times 10^{-2} \text{ SCC/sec} \end{aligned}\right\} \qquad (1)$$

Manes and Grant (ref. 3) have a theoretical extrapolation of experimental data which shows that a coconut-based charcoal identified as PCB adsorbs 220 SSC per gram of charcoal at a temperature of 4.2 K and a pressure of 1×10^{-6} Torr. This adsorption would allow a leak as follows:

$$\left.\begin{aligned} \dot{M} &= \left(\frac{220 \text{ SCC}}{\text{grams of charcoal}}\right)\left(\frac{50 \text{ g of charcoal}}{2.4 \times 10^{6} \text{ sec}}\right) \\ \dot{M} &= 4.6 \times 10^{-3} \text{ SCC/sec} \end{aligned}\right\} \qquad (2)$$

Although the leaks calculated in references 2 and 3 differ by a factor of 2.2 it is felt that the difference is not significant. In both references the trend is toward higher adsorption at colder temperatures. The IRAS getter will operate at approximately 2 K rather than 4.2 K and, therefore, it should adsorb slightly more than the values shown in equations (1) and (2).

CONCLUDING REMARKS

A copolymer of trifluorochloroethylene and vinyl chloride was found to substantially reduce, but not eliminate, the helium leak rate in both stainless-steel and aluminum lean specimens. The median value of the leak specimens increased from a room temperature value of 4.0×10^{-9} SCC/sec to 4.2×10^{-9} SCC/sec after 50 cold shocks to 4.2 K. The median leak value increased substantially to 2.8×10^{-7} SCC/sec when exposed to superfluid helium at a temperature of 1.74 K. Two of the stainless-steel leak specimens showed evidence of coating failure after the 4.2 K exposure. These leak rates were compared to the observed leak in the IRAS main cryogen tank. Calculations were also presented showing that the activated charcoal adsorber in the IRAS dewar will likely be sufficient to maintain the dewar guard vacuum over the expected life of the mission for leak rates characteristic of these leak specimens.

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Moffett Field, Calif. 94035, May 27, 1980

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16. Abstract

Twenty-one leak specimens were fabricated in the ends of stainless-steel and aluminum tubes. Eighteen of these tubes were coated with a copolymer material to seal the leak. The other three specimens were left uncoated and served as control specimens. All 21 tubes were cold shocked in liquid helium 50 times and then the leak rate was measured while the tubes were submerged in superfluid helium at 1.7 K. During the cold shocks two of the coated specimens were mechanically damaged and eliminated from the test program. Of the remaining 16 coated specimens one suffered a total coating failure and resulting high leak rate. Another three of the coated specimens suffered partial coating failures. The leak rates of the uncoated specimens were also measured and reported.

The significance of various leak rates is discussed in view of the Infrared Astronomical Satellite (IRAS) dewar performance.

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