FINAL REPORT

Cryopumping-Cryosorption Array System

For a 3' x 3' Vacuum Facility

Contract No. NAS 5-5881

for

Goddard Space Flight Center Greenbelt, Maryland

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FINAL REPORT

1.0 INTRODUCTION

This report summarizes the results of tests performed on the cryopumping - cryosorption array system supplied under contract NAS 5-5881.

The report is specifically concerned with the test results and their analysis as related to system performance. Details of system operation as well as background information on theoretical aspects of cryogenic pumping are included only where necessary for logical presentation. The reader is directed to the system Operating Instructions or the Acceptance Test Procedure for supplemental information.

2.0 SUMMARY

- 1. The overall design and performance of the supplied cryo-pumping cryosorption system was proven to be satisfactory and within the requirements of NASA specification GSFC P-322-06-64.
- 2. Ultimate system pressures in the high 10^{-14} to low 10^{-13} Torr range were attained during the testing period. These pressures were attained after a relatively mild system bakeout of 12 hours at 200 230°C.
- 3. Pumping speeds for hydrogen gas exceeded the specified array value based on a 0.16 sticking coefficient for the adsorbent panels. This sticking coefficient is consistent with the indicated panel temperatures of $18 21^{\circ}K$.
- 4. Satisfactory thermal performance was demonstrated while operating a 50 watt heat load inside the test volume.
- 5. The vacuum integrity of the system with its associated flanges and interface connections was satisfactorily demonstrated by bagged helium mass spectrometer leak tests.
- 6. The overall cleanliness of the system and the integrity of the bonded sieve panels was successfully demonstrated when no traceable contaminants were found.

3.0 DISCUSSION

3.1 General Configuration

The supplied array concept and configuration consists of two vertically oriented cryogenically cooled concentric cylinders surrounding a shielded cryosorption pumping system. The supplied hardware was installed in an existing 3 ft. by 3 ft. vacuum facility located in Building 10 at Goddard Space Flight Center, Greenbelt, Maryland. The outer cylinder is cooled with liquid nitrogen and is supplied with a removable, flanged head

located at the top end of the cylinder. The cylindrical shroud is flanged to mate with the baseplate on the GSFC facility. The primary functions of this element are to serve as an integral vacuum vessel, thus minimizing inleakage to the internal vacuum spaces, and to act as a heat sink for absorption of radiant energy from the room temperature chamber enclosure. In addition, the liquid cooled walls act as cryopumping surfaces for higher boiling species, i.e., $H_{\rm p}O$, $CO_{\rm p}$, etc.

The inner cylinder is cooled with helium gas and serves the same qualitative functions as the liquid nitrogen shroud only at a lower temperature level. The shielded internal cryosorption pumping array is attached to a removable plate which forms the top end of the helium shroud. The plate-array assembly is attached to and removed with the liquid nitrogen shroud head during disassembly. The lower end of the helium shroud contains an open port (8 inch diameter). The area extending on both sides of the port to a diameter of 10 inches is machined to a flat surface to mate with the GSFC supplied vacuum valve plate seal.

The cryosorption pumping array consists of an upper and lower adsorbent panel-baffle assembly. The total effective projected array area is a minimum of 8.0 square feet. Four adsorbent panels are provided as two sandwich assemblies, located at the top and bottom of the helium shroud. The panels facing the work zone are shielded with optically tight chevron baffles. The panel facing the top plate of the shroud is unshielded since all molecules approaching this surface must first make at least one collision with the helium shroud. The panel facing the lower pumping port is also shielded with optically tight chevron baffles.

All electrical and cryogenic feedthrough penetrations servicing the supplied nitrogen and helium elements enter the system through an extension collar which is flange mounted between the 3 ft. x 3 ft. chamber and head units. Separate liquid nitrogen refrigeration circuits are supplied for the liquid nitrogen shroud and head. In addition, three individual helium circuits are routed to 1) cryosorption panels, 2) chevrons and top plate of helium shroud and, 3) helium shroud cylinder.

As indicated, the cryosorption pumping array (both upper and lower elements) is removed from the system as a single unit. A test object support stand is located between the upper and lower array elements and allows a free working volume 20 inches diameter by 20 inches deep.

3.2 In-House Leak Test

An in-house leak check of the system was conducted at the Linde Factory before shipping the system to NASA. The test procedure involved hot and cold thermal cycles on the system as described in Paragraph 2.11 of the Acceptance Test Procedure.

A leak developed in the helium array circuit during the first hot cycle test. This leak was repaired and with the helium mass spectrometer at maximum senstivity, no further leaks were detected in any of the tested circuits through the remainder of the in-house test cycle. It should be noted that the liquid nitrogen vessel itself was not leak checked during the in-house test program because suitable test equipment was not available.

3.3 Field Leak Test

Leak tests were performed on the complete system during and after final installation on the NASA baseplate. This procedure generally followed that outlined in Paragraph 2.12 of the Acceptance Test Procedure.

During installation testing, a relatively large leak was discovered in a weld seam area of the liquid nitrogen vacuum vessel. In addition a small leak was indicated in the No. 31 Pin on the D-2 power feed-through. The system was disassembled and the weld seam leak successfully repaired. The suspect feedthrough pin was removed and a plug welded in its place.

After reassembly the complete system was leak checked. The inner vacuum vessel and all the cryogenic tubing showed no leaks at maximum sensitivity of the mass spectrometer. The outer guard vacuum vessel showed no leaks at the maximum attainable sensitivity of 1×10^{-7} standard cc/sec. The system was then baked for 12 hours at an average temperature of 200°C. Subsequent leak checks at ambient temperature still showed no detectable leaks.

Because of equipment delays etc., the cryogenic leak test was not conducted until after the system had been opened once and baked for the second time. The cryogenic leak test showed no detectable leaks in any of the cryogenic circuits; however, a 1×10^{-6} Torr-liters/sec. leak was indicated between the guard vacuum and the inner volume. Later when the guard vacuum vessel was opened, small leaks were suspected in the G. E. cold cathode gauge, a flange joint on the gas in-bleed line, the liquid nitrogen vessel upper flange and the liquid nitrogen vessel to baseplate flange.

The G. E. guage was replaced and all the suspect joints except the baseplate flange were retorqued. A repeat leak check still showed positive; however, it was decided to proceed with the test program since the leak was protected by the guard vacuum and would not effect the overall system performance.

Upon completion of the acceptance test program, the system was disassembled and inspected. A misalignment of the aluminum wire gasket on the liquid nitrogen vessel flange was discovered in the vicinity of the G. E. gauge. It was decided that correct placement of the gasket plus retorquing of the baseplate bolts would resolve the problem.

3.4 Performance Data

During the course of the acceptance test, the system was baked out and cold cycled a total of three different times. The first bakeout was in conjunction with the leak test and the first cooldown was a shakedown of the cryogenic refrigerant system. A third complete cycle was necessary because the second had to be aborted after difficulties were encountered with the inleak measuring equipment.

Performance data from the second and third test cycles are given in Tables 2 through 7. The data is identified as Run No. 1 or Run No. 2.

Table 2 lists the vacuum pressure versus time for the system. The guard vacuum pressure was measured by a hot filament, Bayard-Alpert type ionization gauge. A nude, hot filament, Bayard-Alpert ionization gauge was mounted inside the work zone. The intermediate vacuum space was monitored by a hot filament, Bayard-Alpert type ionization gauge that was mounted on the liquid nitrogen vessel head. Finally, a General Electric cold cathode, trigger discharge gauge was mounted on the liquid nitrogen vessel head with the tubulation extending into the work zone.

From the test data it is observed that the system pressure as read by the G. E. guage repeatedly reached the low 10^{-13} Torr range. No effort has been made to adjust or correct these readings for gauge sensitivity since a partial pressure analyzer was not used during the acceptance tests.

The bakeout data given in Table 3 shows that the hot gas heater system is capable of achieving bakeout temperatures in the desired 200°C. range.

Tables 5, 6, and 7 show the temperature profile of the gaseous helium refrigerated system. From the observed temperature readings, it is concluded that the Collins Cryostat used for the tests was marginal in the operation of the system. Temperatures average near 20°K whereas a cryosorption system of this type would be more effective if the panels could operate at temperatures below 15°K.

Overall, the system performed very well during both bakeout and cold cycle operation. The relatively high system pressures noted after the bakeout cycles can be attributed to the outgassing load imposed by great amounts of instrumentation wiring inside the vacuum areas. Once cryogenic cooling began, the system pressures dropped predictably into the base pressure range.

Output from the liquid nitrogen copper-constantan thermocouples was recorded directly on a Brown, self compensating, strip chart recorder. The gaseous helium copper-constantan thermocouple output was readout on a

liquid nitrogen referenced Dymec Digital Voltmeter. Standard NBS conversion tables were used to record temperature in $^{\circ}K$. Reading accuracy at the 20 $^{\circ}K$ level was assumed to be + 1 $^{\circ}K$.

3.5 Thermal Performance Test

The thermal performance test was conducted to insure an overall temperature uniformity while operating a 50 watt test load in the work volume.

A quartz iodine lamp was suspended in the work zone to provide the 50 watt load. The power to the lamp was regulated by measuring the voltage and current input.

The test was conducted during the first cooldown cycle after the system had stabilized. Readout data during the test is given on data sheet - Table 5. From this data it is concluded that thermal performance is within the specification requirements.

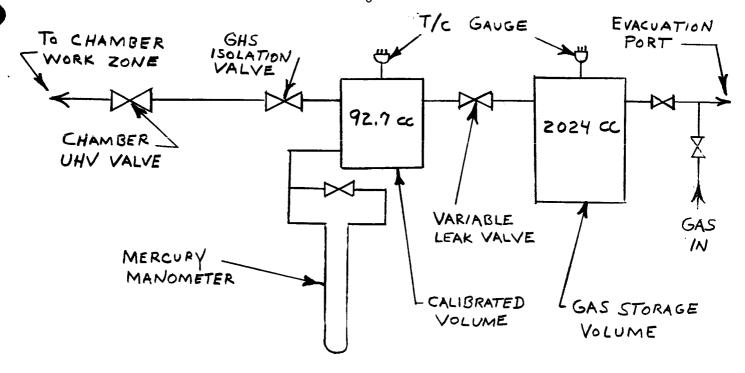
3.6 Pumping Speed Test

Hydrogen pumping speed tests were conducted to verify the predicted adsorbent panel performance. The test method involved measurement of system pressure as a function of time for a specific predetermined gas admission rate. For a more detailed description of the test, the reader is referred to the Acceptance Test Procedure. The specification requires that at 20°K, the adsorbent panels possess a pumping speed of 7,000 liter/sec. per sq. ft. for hydrogen. With 8 sq. ft. of panels and associated baffles, the total effective pumping speed in the work zone is estimated to be 33,000 liter/sec.

The NASA gas handling system was used for controlling and measuring the hydrogen inleak rate. It was originally intended to use a Linde supplied system; however, some difficulties were encountered with the variable leak valve and it was decided to use the NASA system.

The NASA gas handling system measures the change in pressure in a known volume (initially under vacuum) as atmospheric hydrogen gas is inleaked across a low conductance variable leak valve. The change in pressure is observed with a mercury manometer or with a calibrated thermocouple gauge.

A description of the NASA system is as follows:



The sequence of operation included the following steps:

- 1. The GHS (gas handling system) to the chamber UHV valve was evacuated with an independent pumping system to 10^{-5} Torr and charged to one atmosphere with hydrogen gas. This evacuating flushing process was repeated twice.
- 2. The GHS was again evacuated to 10^{-5} Torr, and with the variable leak valve closed, the 2024 cc storage volume was charged with one atmosphere of hydrogen gas.
- 3. With the GHS still isolated from the chamber, the variable leak valve was opened and a leak rate was established to the small evacuated volume. Pressure change was monitored using the mercury manometer.

Time Minutes	∆h mm Hg	V-L <u>Setting</u>
0	0	63
55	34	
65	40	
85	52	
Q _{300°K} =	$V = \frac{\Delta h}{t} = Gas inleak rate$	
V = 92:7	cc	
Q _{300°K} =	$9.5 \times 10^{-4} \frac{\text{T-L}}{\text{Sec.}}$	

4. The chamber UHV isolation was opened and the line between the valve and the GHS was allowed to equilibrate with the vacuum system. Then the GHS ioslation valve was opened and the leak rate admitted to the chamber. Pressure time data was recorded using the G. E. cold cathode gauge and the nude hot cathode gauge. Pressure time data is given in Table I. The curve is shown in Figure 1.

From the data shown in Figure 1 it is observed that the system pressure increased whenever the nude gauge was turned on. The nude gauge was not degassed because the 90 watt load would have been too great for the refrigeration system. In addition, the higher recorded pressures with the nude gauge could be due to a beaming effect because of its proximity to the inleak gas tube.

Pumping speed calculations are based on the pressures as measured by the G. E. gauge with corrections for gauge temperature and sensitivity for hydrogen gas.

$$S_{300} \circ K = \frac{Q_{300} \circ K}{k_{H_2} (P_{ss} - P_o) (\frac{300}{T_g})} 1/2$$

$$Q = 9.5 \times 10^{-4} \frac{T - L}{Sec}.$$

$$P_{ss} = 4.5 \times 10^{-9} \text{ Torr}$$

$$P_o = 7.3 \times 10^{-11} \text{ Torr}$$

$$T_g = 83 \circ K$$

$$k_{H_2} = 2.1$$

$$S_{300} \circ K = 53,000 \text{ liters/sec}.$$

3.7 Adsorbent Integrity

A clean, polished stainless steel plate was installed in the work volume of the chamber prior to starting the acceptance test. This plate was mounted horizontally parallel to and beneath the top adsorbent panel so as to collect any molecular sieve particles that become dislodged during the test program.

Upon completion of the test program, the plate was removed and analyzed by NASA personnel. No trace of sieve particles were found.

3.8 Equipment Status

In general, the vacuum equipment as supplied under the contract was proven to be suitable and practical for the application. Actual installation proceeded without major difficulty and with only a few equipment modifications.

The NASA supplied valve plate which is used to isolate the 10 inch diffusion pump system from the test volume, worked very well.

The cap screw bolts used on the liquid nitrogen vessel top and bottom sealing flanges were replaced with hex-headed bolts to facilitate torquing. Also a high temperature lubricant was used on the threads of all the stainless steel bolts in the guard vacuum to eliminate a seizing problem.

After initial alignment, the cryogenic feedthrough flanges were found to seal satisfactorily to the extension collar. The copper gaskets supplied per drawing A-611541 were found to seat better if the O.D. was reduced .030 inches.

The therocouple and power feedthrough except A-1 were wired as shown in the supplied drawings. This feedthrough was removed to accommodate the inleak equipment and at NASA's request was not re-installed.

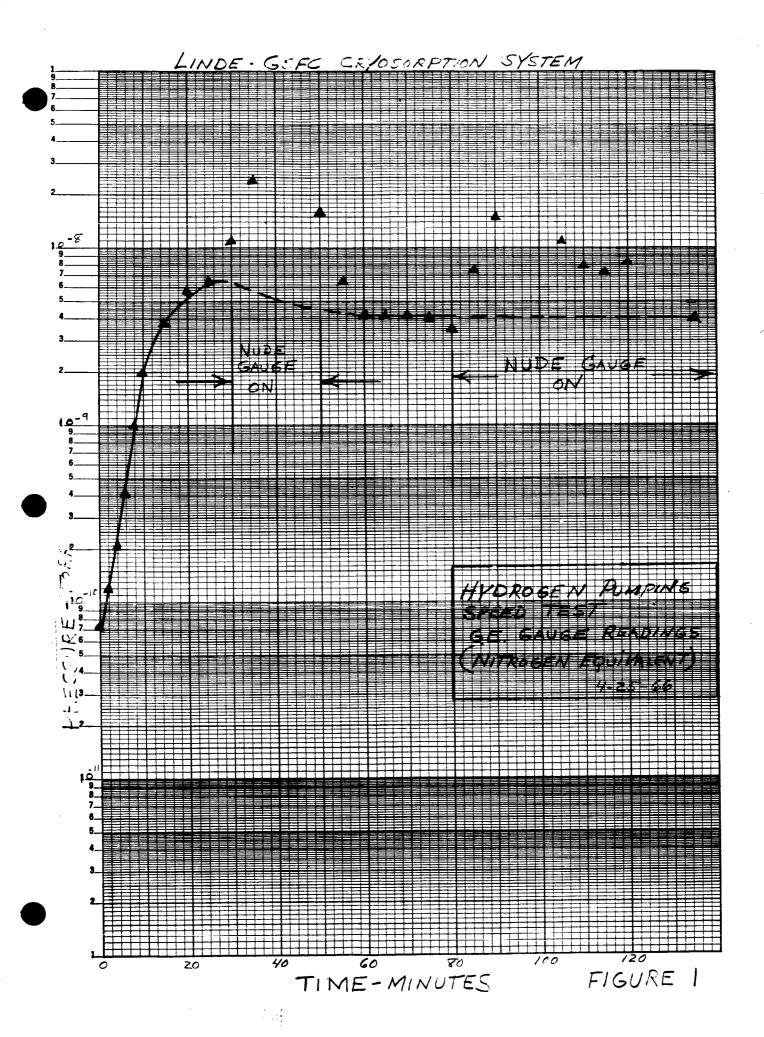


TABLE I

Time Minutes	Pressure Torr (G. E.)	Pressure Torr (Nude)	<u>Remarks</u>
0	3×10^{-12}		Opened UHV
0	7.3×10^{-11}		Admitt leak
1	9 x 10 ⁻¹¹		Moura C. Tonn
2	1.2×10^{-10}		
4	2.2×10^{-10}		
6	4.3×10^{-10}		
8	1×10^{-9}		
10	2×10^{-9}		
15	3.8×10^{-9}		
20	5.8 x 110 ⁻⁹		
25	6.5×10^{-9}		
30	1.1×10^{-8}	1.1×10^{-7}	Nude gauge ON
35	2.4×10^{-8}	1×10^{-7}	,
50	1.6×10^{-8}	1×10^{-7}	Nude gauge OFF
55	6.4×10^{-9}		
60	4.1×10^{-9}		
65	4.2×10^{-9}		
70	4.5×10^{-9}		
75	4×10^{-9}		
80	3×10^{-9}	. <u></u> .	
85	7.6×10^{-9}	8×10^{-8} *	Nude gauge ON
90	1.5×10^{-8}	9.3×10^{-8}	
105	1.1×10^{-8}	3×10^{-8}	
110	8 x 10 ⁻⁹	7.1×10^{-8}	
115	7.4×10^{-9}	7×10^{-8}	
120	8.4×10^{-9}	6.5×10^{-8}	
135	4×10^{-9}	3×10^{-8}	Close UHV

Hydrogen Leak Test Completed

^{*} Nude gauge not degassed.

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