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CRYOSORPTION PUMPING OF He BY CHARCOAL AND A COMPOUND CRYOPUMP DESIGN FOR TSTA*

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Summary

This paper presents the studies of cryosorption pumping of He by charcoal at 4.2°K and between 10 and 20°K. We conclude that coconut charcoal at 4.2°K is suitable for evacuating He from fusion reactors. A compound cryopump design, which utilizes cryocondensation for hydrogen isotopes and cryosorption for helium is also presented.

I. Introduction

In fusion reactors, reliable high-speed vacuum pump system will be required to evacuate the reactor chamber during and between burns. The plasma exhaust gases are expected to be largely mixtures of hydrogen and helium isotopes which can be pumped by cryopumping. The advantage of cryopumping lies in its ability to achieve very high pumping speeds at low equilibrium pressure and to confine the pumped species onto cryosurface. The hydrogen isotopes can be pumped efficiently by either cryocondensation or cryosorption at liquid helium temperature. Helium, however, can only be pumped by cryosorption at low temperature. The two most commonly used adsorbents for cryosorption of helium are activated charcoal and molecular sieves. Cryosorption pumping of helium on charcoal has been reported in previous publications,[1,2] however, neither the extent nor the characteristics of these studies has covered the needs for fusion devices.

This investigation was initiated to study the cryosorption behavior of helium on activated charcoal at low temperature under conditions similar to those anticipated on future fusion devices. We have obtained the following information during the course of this study: (a) The sorption capacity of He between 10 and 20°K and at 4.2°K. (b) The pumping speeds of He at different surface coverage at the same temperature ranges. (c) The equilibrium pressures at different surface coverages at these temperatures. (d) The behavior of He cryosorption pumping in the presence of various amounts of hydrogen isotopes. (e) Regeneration characteristics from charcoal at higher temperatures.

Based on the information obtained on cryosorption of He on charcoal in this study and on cryocondensation of hydrogen isotopes on polished surface at 4.2°K,[3-5] the design of a compound cryopump is presented which utilizes cryosorption for helium and cryocondensation of hydrogen isotopes. This pump is under fabrication and will be delivered to Tritium System Test Assembly (TSTA) at Los Alamos Scientific Laboratory for testing.

II. Experimental Setup

The system used in this study is shown schematically in Fig. 1. An Air Product Displex Refrigerator (Fig. 1(a)) was used to cool down the cryosorption panel to desired temperature between 10 and 20°K, while a modified LHe cooled Excalibur Cryopump (Fig. 1(b)) was used for measurements at 4.2°K. The cryosorption surface sits inside the main vacuum chamber which was initially pumped out by turbomolecular pump TP. The main chamber is separated from the gas inlet chambers by calibrated orifices C_1 and C_1'. These orifices have conductances of 7 ℓ/s for He and 10 ℓ/s for H_2. The

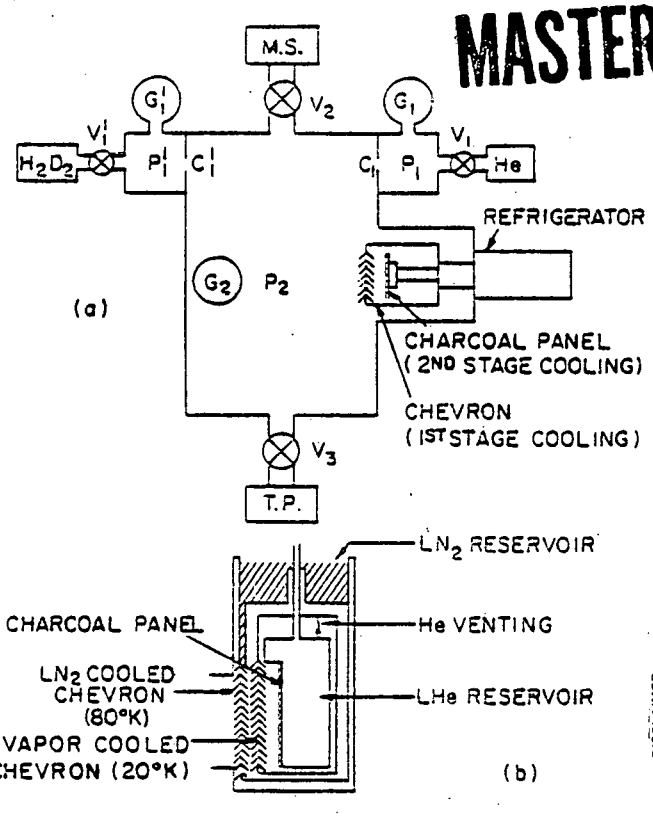

Fig. 1. Experimental Setup (a). Vacuum System with Refrigerator; (b). 4.2°K Cryosorption Pump.

desired gas flow rates were obtained by using Granville-Phillips servodriven bleed valves V_1 or V_1' and monitoring pressures P_1 or P_1' with Schulz-Phelaps type gauges G_1 or G_1'. The partial pressure of gases in equalibrium with cryosorption surface was determined by measured total pressure P_2 at Bayard-Alpert gague G_2 and by analyizing mass spectrum at MS. The gas flow rate Q (in Torr-liter/sec) is calculated from $Q = C(P_1 - P_2)$ and the pumping speed S (in liter/sec) from $S = Q/P_2$.

When Air Product Refrigerator, which has a zero heat-load 2nd stage temperature 10°K was used, a black chevron baffle was attached to 1st stage cooling panel (~ 70°K) to cool down the incoming gas and to supply radiation shielding. This 6" diameter baffle has a conductance of 1500 ℓ/s for He. Charcoal was epoxy bonded to a copper plate before being attached to the 2nd stage cold finger. Calibrated silicone diode type sensors which have uncertainty less than 0.1°K were used to monitor the temperatures at both chevron and charcoal plate. The modified Excalibur Cryopump (Fig. 1(b)) has two chevron baffles, one is cooled by LN_2, the other by cold He vapor (~ 20°K) from LHe reservoir. These two baffles together give a conductance of ~ 1500 ℓ/sec for He. Charcoal was epoxy-bonded on the chevron side of the LHe reservoir and its temperature was also monitored with silicone diode type sensor.

Although most measurements were done with epoxy-bonded coconut charcoal, a tin-silver alloy (3.5% Ag)

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which has an eutectic point 221°C has been successfully tested as bonding material. Charcoal was mechanically embedded in melted alloy which was in turn manufactured into ~1/8" thick plate. Metalized bonding has several advantages over epoxy bonding: (a) better thermal conductivity, (b) less temperature gradient, (c) higher resistance to neutron radiation damage under fusion environment, and (d) nonblocking of the porous path on the surface of charcoal.

III. MEASUREMENTS AND RESULTS

Prior to each measurement, the entire system was baked out to ~ 100°C for several hours and evacuated simultaneously by turbomolecular pump TP (Fig. 1(a)). The valve V_3 was closed after the residual pressure dropped below 1×10^{-6} Torr (N_2 equivalent) and the cryosorption surface was cooled down to the desired temperature by either refrigerator or liquid helium. The blank-off pressure immediately before the gas influx was between 1×10^{-8} and 1×10^{-9} Torr which varied with cryosurface temperature and the duration of bakeout. H_2 outgassed from the chamber wall which was at ambient temperature was the predominant residue gas.

A. Adsorption Capacity and Pumping Speed

Desired amount of He (5×10^{-3} T · ℓ/s to .5T · ℓ/s depending on temperature of cryosurface) at ambient temperature was introduced into the main chamber through valve V_1 and orifice C_1. Two different modes were employed during the measurements; (a) by continuous feeding of He at constant flow rate until charcoal panel reached its capacity which was indicated by rapid increase in chamber pressure (monitored by BA gauge G_2) followed by convection and warmup of cryosorption panel; (b) by intermittent feeding of He at constant flow rate for predetermined duration (typically 5 to 30 minutes), after that valve V_1 was closed for sufficient time (1 to 2 hours) to allow the system to reach equilibrium, then the cycles were repeated until charcoal panel reached its capacity. The measured total adsorption capacities at different temperatures are plotted in Fig. 2. No appreciable sorbing capacity was observed for temperature higher than 20°K. The capacities in the intermittent flow mode (b) were found 20% to 30% higher than those in the continuous flow mode (a) at same temperature.

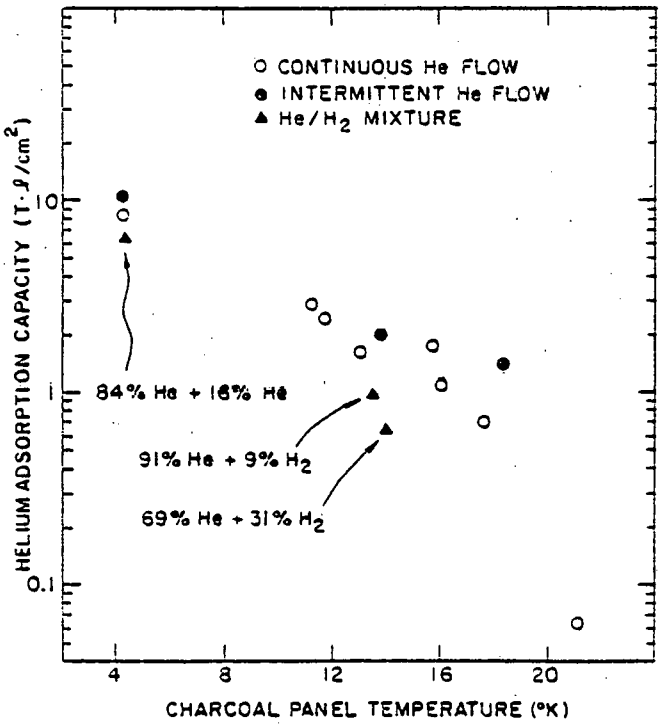

Fig. 2. Helium Adsorption Capacity at Different Cryopanel temperature.

Fig. 3 shows the pumping speed as a function of surface coverage. The initial pumping speed differs little at different temperature, however, at higher temperature it drops off more rapidly with increasing surface coverage. In the intermittent runs, the pumping speeds were found to be increasing at the beginning of each cycle (see Fig. 4.) and were higher

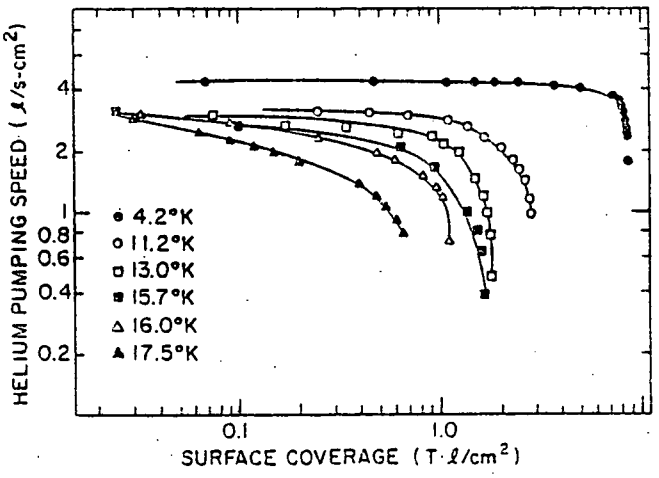

Fig. 3. Pumping Speed of Helium by Charcoal at Difference Cryopanel Temperatures

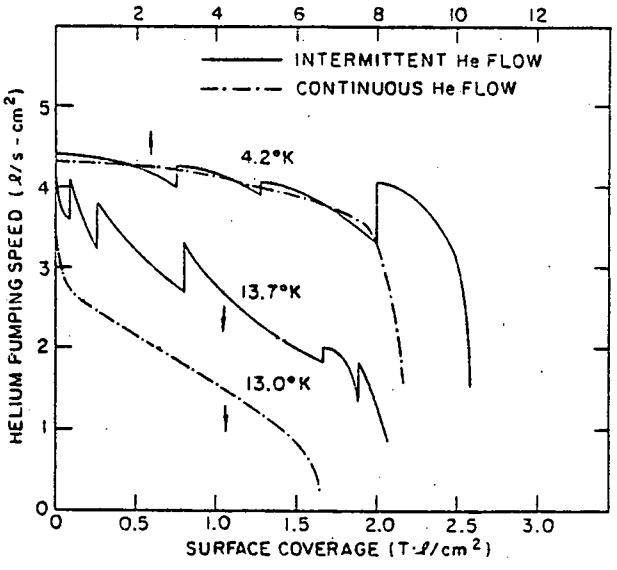

Fig. 4. Pumping Speed of Helium by Charcoal with Continuous and Intermittent He Flow. Flow Rate: 2.5×10^{-4} T · l/s-cm² (13°K); 6.7×10^{-4} T · l/s-cm² (4.2°K).

than those in the continuous run under the same surface coverage and temperature. This difference became smaller at lower temperature i.e. 4.2°K. Less than 30% decrease in pumping speed was observed at 4.2°K when gas flow rate was increased by forty fold (from 1.7×10^{-5} T · ℓ/s-cm^2 to 7×10^{-4} T · ℓ/s-cm^2).

B. Equalibrium Pressure and Heat of Adsorption

The residual helium pressure in vacuum chamber at different surface coverages were measured after the influx of helium was stopped as mentioned in A in the intermittent runs. Durations between 1 and 2 hours were needed for the system to reach equilibrium which was indicated by little pressure change for, say, 15 to 20 minutes. Fig. 5 shows the residual pressure (N_2 equivalent) versus surface coverage 2 minutes, 15 minutes, and 1 hour after helium flow was stopped when the cryosorption panel was at 4.2°K. Fig. 6 shows the

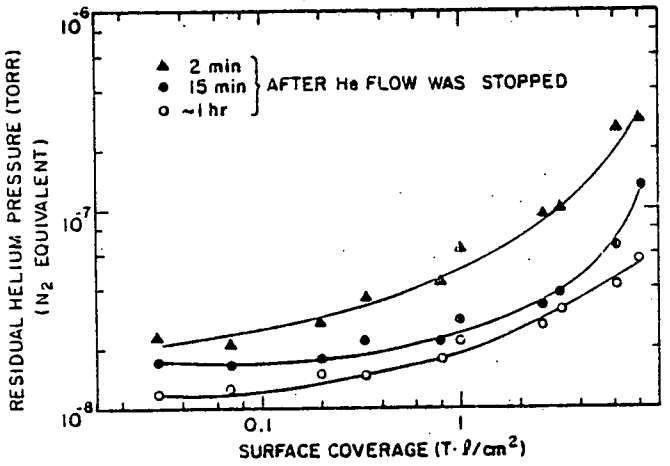

Fig. 5. Residual Helium Pressure at 4.2°K.

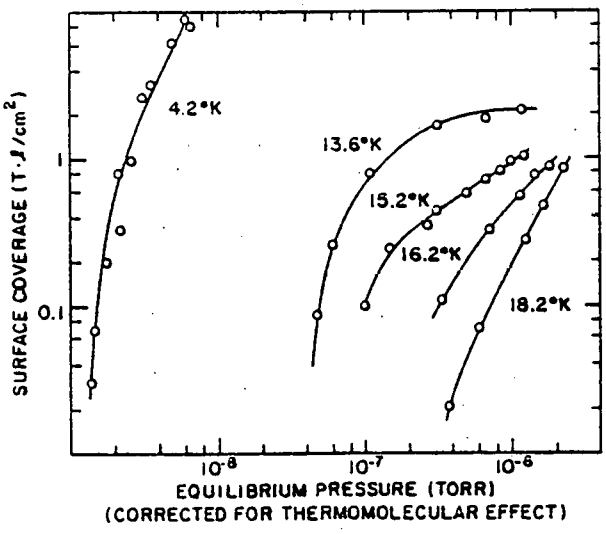

Fig. 6. Adsorption Isotherm of Helium on Coconut Charcoal.

adsorption isotherms measured at several different temperatures. The equilibrum pressures have been corrected for thermomolecular effect.

In order to estimate the heat load from cryosorption, it is necessary to determine the magnitude of heat of adsorption. The heat of adsorption of helium on charcoal was determined by measuring adsorption isosters between 12 and 19°K after desired amount of gas was adsorbed and by using Clausius Clayperon expression $\Delta H = 4.575\ \Delta \log P / \Delta \frac{1}{T}$ cal/mole. The values obtained decreased slightly with increasing surface coverage from 940 cal/mole at 1.7×10^{-2} T · ℓ/cm^2 to 760 cal/mole at .5 Tℓ/cm^2. The low heat values indicate that the adsorption was purely physical in nature and the heat load to cryopanel is neglibible comparing with those from other sources like radiation and conduction.

C. The Adsorption of He/H_2 Mixture

Due to the presence of large quantity of hydrogen isotopes in a fusion reactor, their influence on the adsorption behavior of helium will be crucial to the design of a compound cryopump. We have studied the changes in pumping speed and capacity of helium adsorption at 4.2°K and 13.5°K in the presence of various amounts of hydrogen. The desired amounts of He and H_2 were admitted simultaneously into the system through valves V_1 and V_1' respectively. The gas flow rate ratio Q_{H_2}/Q_{He} was determined by pressure gauges G_1 and G_1' and conductance C. The partial pressure of individual gas in equilibrium with charcoal panel was determined by BA gauge G_2 and mass spectrometer MS. The resultant capacities and relative pumping speeds were shown in Fig. 2 (filled triangles) and Table I

TABLE I. Relative Pumping Speeds of He in H_2/He Mixture

T = 13.5°K (a) C_{He}=.15 C_{H_2} = .03		T = 4.2°K C_{He}=.67 C_{H_2}=.10		T = 4.2°K C_{He}=2.8 C_{H_2}=.47	
(b) Q_{H_2}/Q_{He}	(c) S_{He}	(b) Q_{H_2}/Q_{He}	(c) S_{He}	(b) Q_{H_2}/Q_{He}	(c) S_{He}
.04	.96	.02	1.00	.02	.91
.07	.91	.04	.95	.04	.93
.15	.78	.10	.95	.10	.87
.37	.56	.19	.96	.19	.88
.91	.46	.38	.97	.38	.89
1.83	.39	.76	1.00	.76	.88
3.65	.33	1.90	1.19	1.52	.83
		3.80	1.15	3.80	.83
				7.60	.75

(a) C: Surface coverage expressed in T · ℓ/cm^2.
(b) Q_{H_2} not corrected for hydrogen cryocondensation.
(c) S_{He} relative to pure helium pumping speed at same temperature.

respectively. No significant change in He pumping speed was observed when Q_{H_2}/Q_{He} or He surface coverage was small, however, He Pumping speed drops at higher Q_{H_2}/Q_{He} value and the percentage of decrease is more severe at 13.5°K (Table I). The combined adsorption capacity of He and H_2 (Fig. 2) is very close to the capacity of pure helium, this suggests that H_2 is competing with He for active site on charcoal surface but not blocking the adsorption of He.

D. Desorption of He from Charcoal

Controlled regeneration of He from charcoal without disturbing the cryocondensed hydrogen isotopes will be required in the design of compound cryopump. The study of desorption of He from charcoal was carried out by warming up the cryopanel slowly after known amounts of He was adsorbed on charcoal. The turbomolecular pump TP with the known conductance valve V_3 (26 ℓ/s for He) was used to evacuate the desorbed He gas. Both vacuum chamber pressure and charcoal panel temperature were monitored during the warmup process. Our preliminary data indicate that mass desorption of He from charcoal occurs at $T \gtrsim 20^{\circ}K$ depending on the amount of adsorbed helium and is 99+% complete before a temperature of 35°K is reached. However due to high mobility of He in charcoal at this temperature range, either high speed pump or good temperature control will be required to maintain low pressure during regeneration and to avoid convection.

IV. CONCLUSION

Based on the results obtained so far, we have reached the following conclusions pertinent to the design of a cryosorption pump for He using charcoal as adsorbent.

(a) Adsorption Capacity ~ 8 T ℓ and pumping speed ~ 4 ℓ/s per cm^2 adsorbent (~ .1 gm coconut charcoal per cm^2 area) can be achieved at LHe cooled panel. The pumping speed is limited by the transmissivity of chevrons and corresponds to a sticking coefficient ~.8.

(b) Pumping speed is virtually unchanged for surface coverage up to 75% of capacity with a gas flow rate 7×10^{-4} T · ℓ/s-cm^2 at 4.2°K.

(c) Equilibrium pressure of less than 1×10^{-8} Torr (N_2 equivalent) is obtained at small surface coverage, however, it slowly increases to ~ 2×10^{-8} Torr at 80% capacity.

(d) Helium can be desorbed by warming up charcoal slowly. More than 99% adsorbed helium is regenerated before cryopanel temperature reaches 35°K.

(e) The presence of a small amount of hydrogen will not disturb the cryosorption of He.

V. TSTA COMPOUND CRYOPUMP

The TSTA vacuum facility at LASL is intended to demonstrate realistic fuel supply and clean up operations similar to those anticipated on fusion devices. For cryopumps which are to be evaluated at TSTA, design should meet the following major requirements: (a) pumping capacities of 34,000 T · ℓ for DT and 5,300 T · ℓ for He, (b) pumping speeds of 10,000-15,000 ℓ/s for DT and pumping speed of He greater than 20% of those of DT, (c) ability to start pumping at an initial postburn torus pressure as high as 7.5×10^{-3} Torr with initial flow rate of 52 T · ℓ/s and being able to achieve a blank-off pressure of ~ 10^{-8} Torr at near full capacity, (d) ability to regenerate He and DT separately.

A compound cryopump was designed by us to meet these requirements. A schematic cross-sectional drawing of this pump is shown in Fig. 7. This pump consists of (a) LN_2 cooled chevrons to cool down the incoming gases, to condense impurity gasses and to serve as radiation shieldings, (b) LHe reservoir and LHe cooled chevron for cryocondensation of hydrogen isotopes, (c) LHe cooled cryosorption panel for He pumping with metallically bonded coconut charcoal as adsorbent. With the exception of chevron and cryosorption panel, the entire unit is constructed of 304 stainless steel. Some important specifications of this pump are listed in Table II and compared with TSTA requirements. This compound cryopump is currently under fabrication by Janis Research Corporation. Upon completion, it will be tested at BNL prior to delivery to LASL for evaluation.

TABLE II. BNL Compound Cryopump Specifications

	BNL Compound Cryopump	TSTA Requirements
1. Physical Dimensions		
Input Port	16" Isopneurop flange	Same
Regeneration Port	6" Conflat	Same
Weight	~ 600 lb	
Size	35" O.D., 32" tall	
2. Estimated Pumping Speed		
S_{D_2} (ℓ/s)	13,000	10,000-15,000
S_{T_2} (ℓ/s)	10,400	10,000-15,000
S_{He} (ℓ/s)	3,200	≥ 20% of S_{D_2}
S_{He}/S_{D_2} (%)	25	≥ 20
3. Estimated Capacity		
DT (T·ℓ/s)	> 34,000	34,000
He (T·ℓ/s)	~ 8,000	5,300
He/DT (%)	~ 20	≥ 14
4. Cryogen Consumption		
LN_2 (ℓ/hr)	3 } 10 hr. operation before refill.	
LHe (ℓ/hr)	2	

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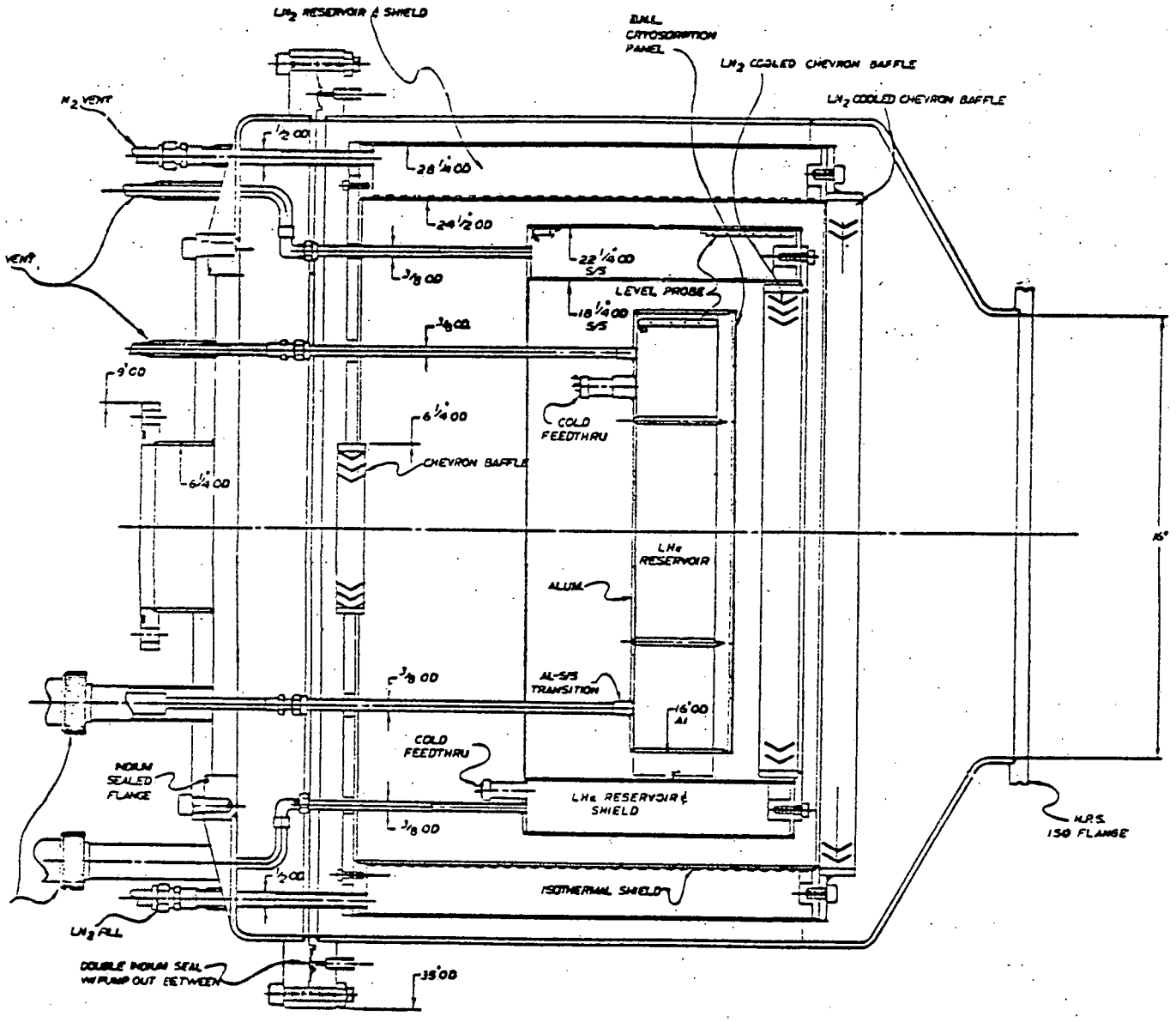

Fig. 7. TSTA Compound Cryopump.