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CRYOSORPTION PUMPING OF DEUTERIUM BY MS-5A AT TEMPERATURES ABOVE 4.2 K FOR FUSION REACTOR APPLICATIONS*

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CRYOSORPTION PUMPING OF DEUTERIUM BY MS-5A AT TEMPERATURES ABOVE 4.2 K FOR FUSION REACTOR APPLICATIONS*

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ABSTRACT

An Excalibur CVR-1106 cryosorption pump was fitted with a special cooling system to permit measurement of deuterium pumping speeds at temperatures between 6 and 20 K. Pumping speeds were found to be a function of feed rate, loading prior to each run, loading during runs, and thermal treatment between runs. At feed rates < 3 x 10^{-4} Torr-ℓ s^{-1} cm^{-2}, speeds were near 1 ℓ s^{-1} cm^{-2} initially and declined monotonically with loading. At high feed rates, speeds reached a higher maximum (∿ 3 ℓ s^{-1} cm^{-2}) but also generally declined with loading; however, after 50 to 100 Torr ℓ had accumulated, the pump underwent a spontaneous transition which effected a return to the original (high) pumping speed. This transition was accompanied by pressure spikes in the test chamber and temperature spikes in the sieve panel. Initial speeds for each consecutive run equaled the final speed for the preceding run if the pump was maintained at operating temperature; however, if it was warmed to 77 K and recooled, a restoration to the maximum speed was observed at the beginning of the next run.

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INTRODUCTION

Several fusion reactor vacuum systems will be required to recover only hydrogen isotopes (no helium). These systems include neutral beam injection systems, pellet fueling systems, and some embodiments of blanket recovery systems. Requirements of these systems can be fulfilled by either cryocondensation or cryosorption pumps. Neutral beam injection systems are presently being operated with cryocondensation pumps, but cryosorption pumps have possible advantages which would make them attractive for future systems. These advantages include the ability to operate at higher temperatures; the ability to operate in more hostile environments where heat fluxes would preclude the use of liquid helium as a coolant; more operating flexibility because equilibrium adsorption is less sensitive to temperature fluctuations than equilibrium vapor pressure; and easier control of pump regeneration.

Response of the Excalibur CVR 1106 pump to deuterium and hydrogen pumping at 4.2 K has been reported previously.[1] The pumping speed of deuterium was found to be constant at 1000 $\ell\ s^{-1}$ from 10^{-8} to 10^{-4} Torr; however, it increased from 1000 $\ell\ s^{-1}$ at 10^{-4} Torr to 1800 $\ell\ s^{-1}$ at 3×10^{-3} Torr. It was possible to pump at least ten times the molecular sieve (MS-5A) adsorption capacity without any decrease in pumping speed. This indicates that the principal pumping mechanism (for deuterium at this temperature) is cryocondensation, not cryosorption. The initial pumping speed for hydrogen was 1300 $\ell\ s^{-1}$ at a pressure in the range of 10^{-8} to 10^{-4} Torr. This speed began to increase to 1800 $\ell\ s^{-1}$ at 3×10^{-3} Torr. However, after a short period of pumping in the range from 4×10^{-6} to

4×10^{-4} Torr, the speed was reduced by the buildup of solid hydrogen. The minimum steady-state speed was found to be 600 ℓ s^{-1} (less than one-half the initial speed) at 10^{-5} Torr. Speeds for helium[2] fell into two regimes, depending on whether the feed rate was above or below 3×10^{-3} Torr-ℓ s^{-1}. Below this point, the speed decreased monotonically with loading from 600 to 200 ℓ s^{-1}. Above this point, the speed varied periodically with loading, and the pump went through a transition at the end of each cycle which restored the initial high speed.

Several studies of hydrogen cryosorption on MS-5A in the range of 10 to 20 K have been reported. Southerlan[3] reported initial pumping speeds (with no previously accumulated hydrogen) that are equivalent to between 1300 and 1400 ℓ s^{-1} on the CVR 1106 pump. (Corrections were made for 300 K pressure measurement, conductance of 1600 ℓ s^{-1} between the gauge and the cryopanel, and 320-cm^2 cryosorption surface area.) Measured unbaffled pumping speeds were inversely proportional to feed rate; the data followed this relationship even when the feed rate was not increased monotonically from run to run. Stern, Hemstreet, and Ruttenbur[4] reported pumping speeds for three runs in the 10^{-8} to 10^{-7} Torr range at 20 K to 0.2% of the saturation capacity. The speeds are all approximately equal, and equivalent to 950 ℓ s^{-1} on the CVR 1106. Gareis and Pitlor[5] measured speeds near 5×10^{-8} Torr with 20 K panels. Initial speeds were equivalent to 600 ℓ s^{-1} with a 77 K chevron and 1000 ℓ s^{-1} with a 20 K chevron on the CVR 1106.

Cryosorption pumping of deuterium, hydrogen, and helium at 4.2 K has shown that speeds are dependent on the location of the vapor pressure with respect to the operating pressure. If the vapor pressure is much lower than the operating pressure (as for deuterium), the speed is constant; if the vapor pressure is near the operating pressure (as for hydrogen), the speed is significantly reduced by condensation on the external surface of the adsorbent; and if the vapor pressure is much greater than the operating pressure (as for helium), the speed is a complicated function of feed rate and loading. Diffusion rates into the micropores of the sieve can affect speeds. Cryosorption pumps for deuterium operating at 10^{-4} Torr (probable fusion reactor conditions) are, therefore, expected to enter regions where behavior is hampered by condensation at temperatures > 6.6 K. Data for hydrogen at elevated temperatures (where the vapor pressure is much greater than the operating pressure) show the complexity of the problem. Two distinct types of behavior have been reported: high initial speeds which decrease with increasing feed rate and loading, and low initial speeds which increase with increasing feed rate and pressure. By extrapolating these results to 10^{-4} Torr, one could conclude either that this type of pump will operate adequately or it will not operate at all for fusion reactor applications. An understanding of the physics of cryosorption pumping is not sufficient for predicting behavior in regions of interest; therefore, experimental programs have been initiated at Oak Ridge National Laboratory (ORNL) to obtain the necessary data. This paper describes the initial phase of this work in which deuterium pumping speeds were measured at temperatures > 6.6 K.

EXPERIMENTAL APPARATUS

The Excalibur CVR 1106 cryosorption pump is not well suited for operation at elevated temperatures. In its normal operational mode, it is cooled by pool boiling of liquid helium. To obtain the data in the desired temperature range, the pump reservoir was fed with a gas at a controlled temperature. Figure 1 shows a schematic diagram of the apparatus. The system has two basic components: a liquid helium dewar fitted with a heater to supply 4.2 K gas, and a feed gas heater which controlled the temperature of the gas as it entered the pump reservoir. Liquid helium was supplied in a standard Linde LSHe-100 dewar. A nichrome resistance heater delivering $\sim$ 6 W was submerged in the liquid helium to produce $\sim$ 25 ℓ min^{-1} of cooling gas (at room temperature). The feed gas heater consisted of a vacuum-insulated and liquid-nitrogen-shielded double-pass heater. The temperature of the exit gas was measured with a germanium resistance thermometer (GRT) and controlled with a Scientific Instruments model 3610 controller driving a 35-Ω nichrome heater mounted on a vertical tube. Gas was routed down through the center of the heated tube and then up around the outside of the tube. Gas at the controlled temperature was delivered to the pump and discharged across the back of the cryosorption panel. Panel temperature was determined by a calibrated GRT mounted in a nipple at the bottom of the reservoir. Most experimental runs were made with the inner chevron cooled to 77 K by backflushing with liquid-nitrogen-cooled helium gas flowing at a rate of 1 ℓ min^{-1}. One run was made with helium vent gas cooling the chevron. Pump inlet pressures were measured by nude Varian Millitorr and UHV gauges located

ORNL DWG. 77-1251

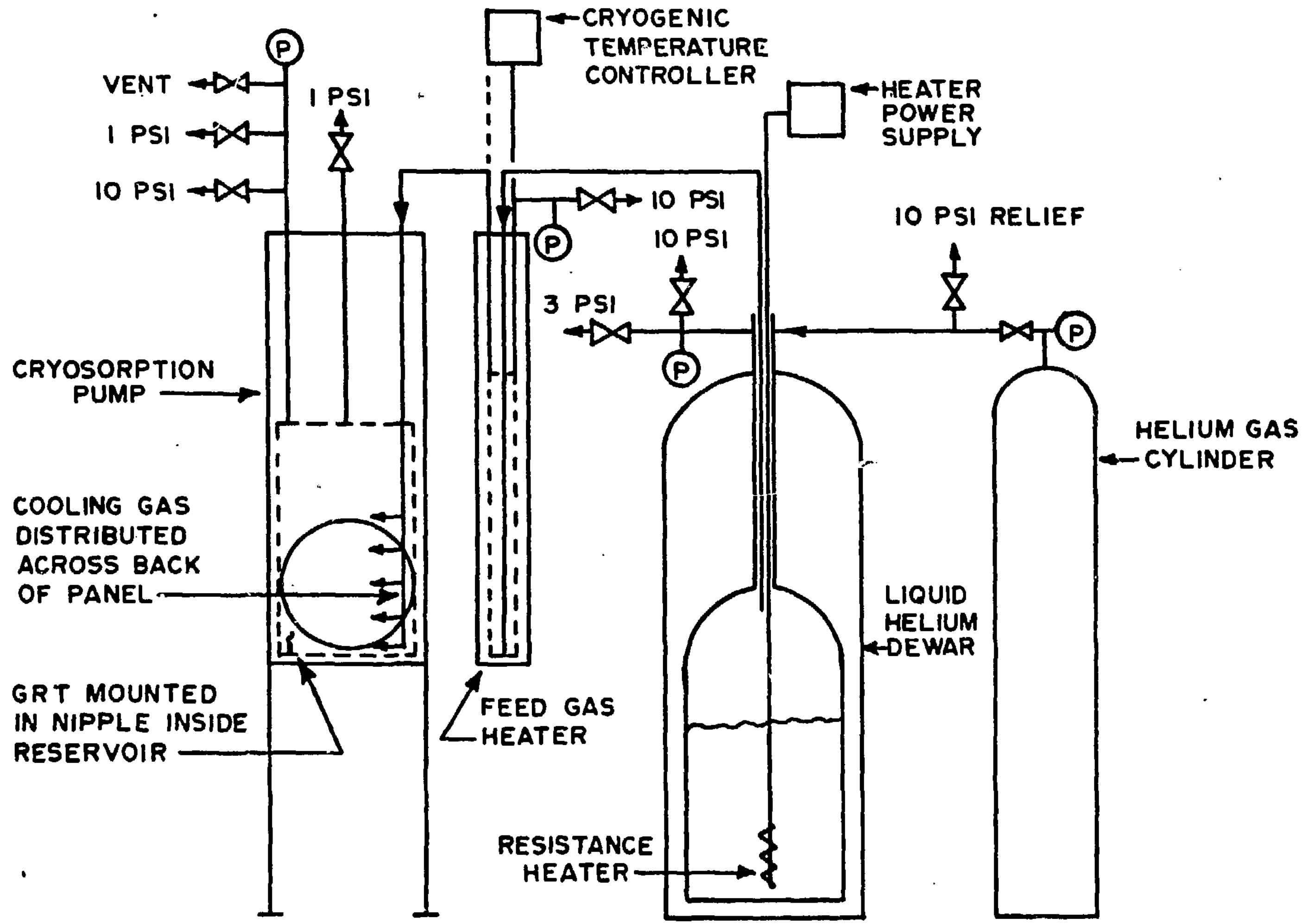

FIG. 1

25.4 cm in front of the outer chevron of the pump. The calculated conductance for deuterium between the ion gauges and cryopanel[6] is 1600 ℓ s^{-1}; pumping speeds have not been corrected for this restriction. Specific speeds were based on the entire area of the cryosorption panel, which is 320 cm^2.

RESULTS AND DISCUSSION

Twenty-eight separate runs were made at temperatures between 7 and 20 K. Initial speeds for the first run after regeneration ranged from 400 to 900 ℓ s^{-1}; no speeds < 400 ℓ s^{-1} (1.2 ℓ s^{-1} cm^{-2}) were measured until ∿ 100 Torr ℓ (∿ 2% of equilibrium saturation capacity) of deuterium had accumulated on the panel. Speeds up to 1000 ℓ s^{-1} (3.1 ℓ s^{-1} cm^{-2}) were obtained with the panel containing up to 200 Torr ℓ. It was possible to attain speeds near 400 ℓ s^{-1} with an accumulation of 1000 Torr ℓ on the panel. Maximum pumping speeds decreased with the total panel loading; that is, the speed went from 1000 ℓ s^{-1} at 200 Torr ℓ to zero at 5000 Torr ℓ.

Figure 2 shows the pumping speed as a function of feed rate. Maximum speeds go through a maximum of 1000 ℓ s^{-1} at ∿ 10^{-1} Torr-ℓ s^{-1}. This corresponds to a pressure of 10^{-4} Torr. Below 10^{-2} Torr-ℓ s^{-1}, maximum speeds are always < 400 ℓ s^{-1}. Similar behavior was observed for helium at 4.2 K in which pumping speed for a given loading was increased at high feed rates[2] (> 3 x 10^{-3} Torr-ℓ s^{-1}). Both these gases appear to have a higher sticking coefficient on a surface which contains some preadsorbed material. At low feed rates, the diffusion rate of material from the

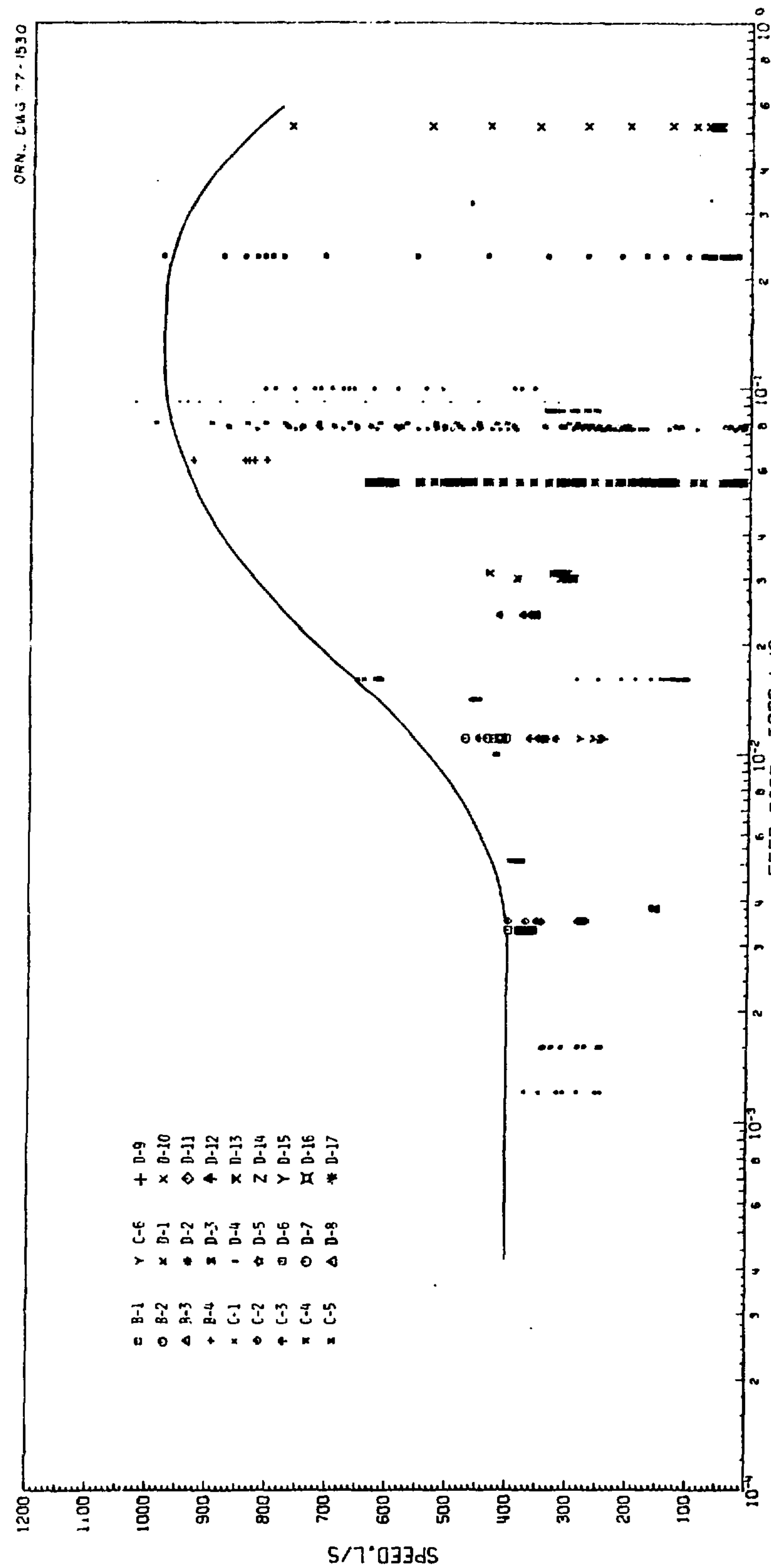
ORNL DWG 77-1530
SPEED, L/S
FEED RATE, TORR-L/S
1200
1100
1000
900
800
700
600
500
400
300
200
100
B-1
B-2
B-3
B-4
C-1
C-2
C-3
C-4
C-5
C-6
D-1
D-2
D-3
D-4
D-5
D-6
D-7
D-8
D-9
D-10
D-11
D-12
D-13
D-14
D-15
D-16
D-17

FIG. 2

surface into the bulk is faster than the sticking rate, so that no surface accumulation occurs; but at high feed rates, diffusion into the bulk is slower than the sticking rate and accumulation occurs.

Figure 3 shows a series of four consecutive runs, all with a feed rate near 0.1 Torr-ℓ s^{-1} and a panel temperature of ∿ 7 K. Run C3 shows speed decreasing monotonically with loading as expected. Runs C3 and C4 were separated by an 8-min interruption in the feed, but the panel was maintained at ∿ 7 K. The initial speed for run C4 was essentially the same as the final speed in run C3. This indicates that little or no material diffused from the surface micropores to the bulk adsorbent. Between runs C4 and C5 the cryopanel was put through an artificial temperature excursion by cutting the coolant gas flow to the reservoir. The panel was then recooled to ∿ 7 K, and run C5 was initiated. No gas was removed from the system; hence, all gas adsorbed on the panel at the end of run C4 was readsorbed at the beginning of run C5. An initial speed of 1000 ℓ s^{-1} with a subsequent decrease in speed with loading (comparable to run C3) was observed. This recovery indicates that the surface had been cleared during this interruption. A 37-min interruption in flow, during which the surface temperature was maintained at ∿ 7 K, was made between runs C5 and C6 to determine whether the time interval had any effect on pumping speed; it did not. The speed in run C6 started where run C5 ended. Run C6 went through two spontaneous recoveries before it was terminated. In each one, the pressure rose to ∿ 10^{-2} Torr, thus causing the panel temperature to rise to ∿ 12 K (the surface temperature may have been much higher). The temperature excursions were followed by

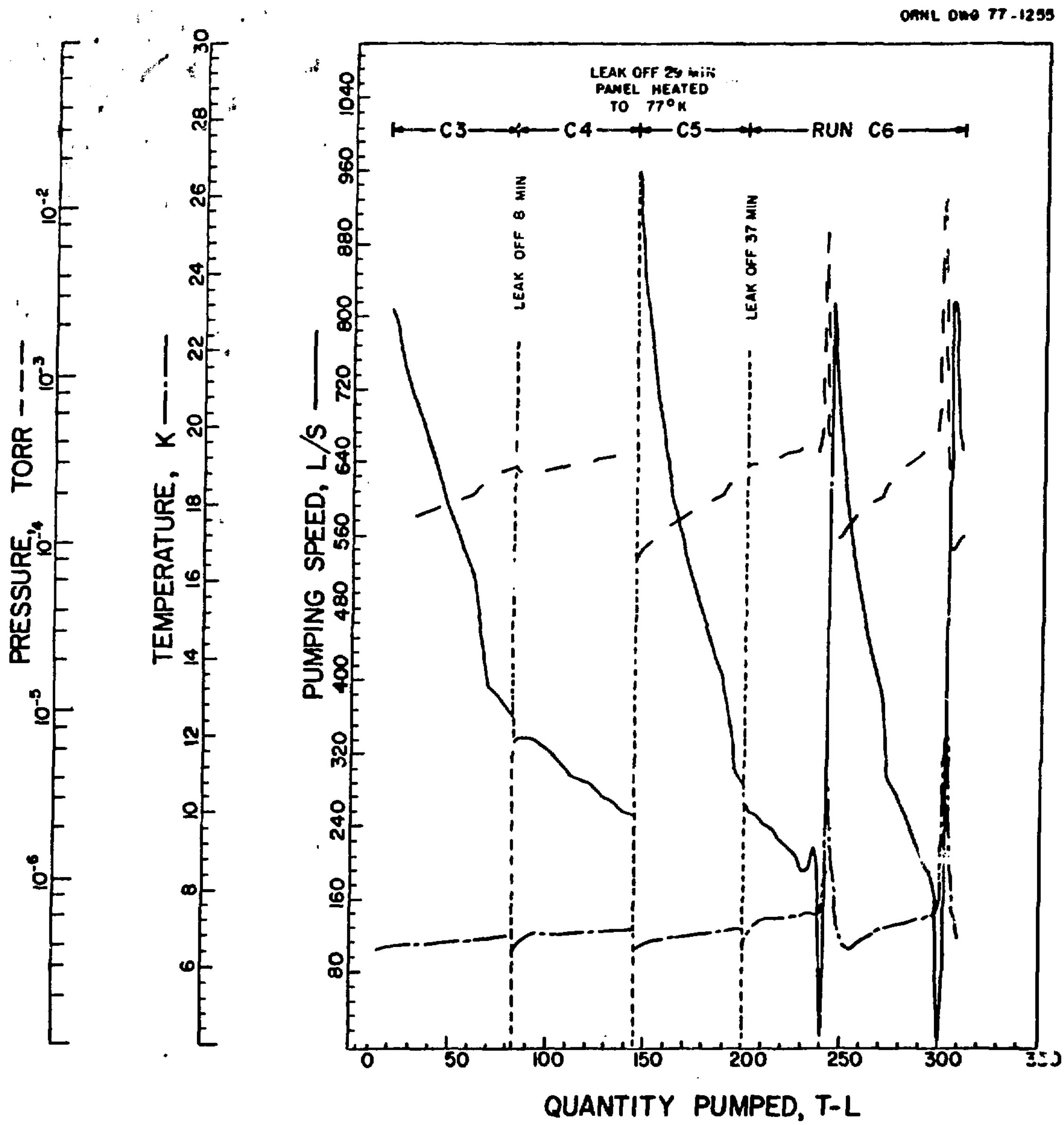
ORNL DWG 77-1255
PRESSURE, TORR
TEMPERATURE, K
PUMPING SPEED, L/S
LEAK OFF 29 MIN PANEL HEATED TO 77°K
C3
C4
C5
RUN C6
LEAK OFF 8 MIN
LEAK OFF 37 MIN
QUANTITY PUMPED, T-L

FIG. 3

recovery to high pumping speeds. The characteristics of these cycles were the same as those observed for helium pumping at 4.2 K.[2]

Figure 4 shows two consecutive runs made near 15 K. The decrease in speed observed in the first run was similar to that shown in Fig. 3. After the 21-min interruption in flow (during which time the panel temperature was maintained at 15 K), a high initial pumping speed was observed, indicating that the surface had partially cleared at this higher temperature. The speed, however, fell very rapidly and eventually caused a pressure spike. The peak pressure was not sufficiently high to cause a significant increase in the panel temperature, and no recovery in speed was observed. Subsequently, a second more intense pressure peak (near 10^{-2} Torr) occurred after ∿ 100 Torr ℓ was collected on the panel. This caused the panel temperature to reach ∿ 24 K; the temperature peak was closely followed by a recovery in pumping speed. Clearly, high temperature excursions, whether naturally occurring or artificially produced, result in improved pumping speed.

CONCLUSIONS

Deuterium pumping at higher temperatures exhibits many qualitative aspects of helium pumping at 4.2 K. This would be expected because the vapor pressure of deuterium is much higher than the desired operating pressure. Maximum speeds were observed near 10^{-4} Torr. This is favorable because 10^{-4} Torr is approximately the desired operating pressure for fusion reactors. Speeds are, however, a strong function of pump operating history (panel temperature and loading), and speeds far below maximum

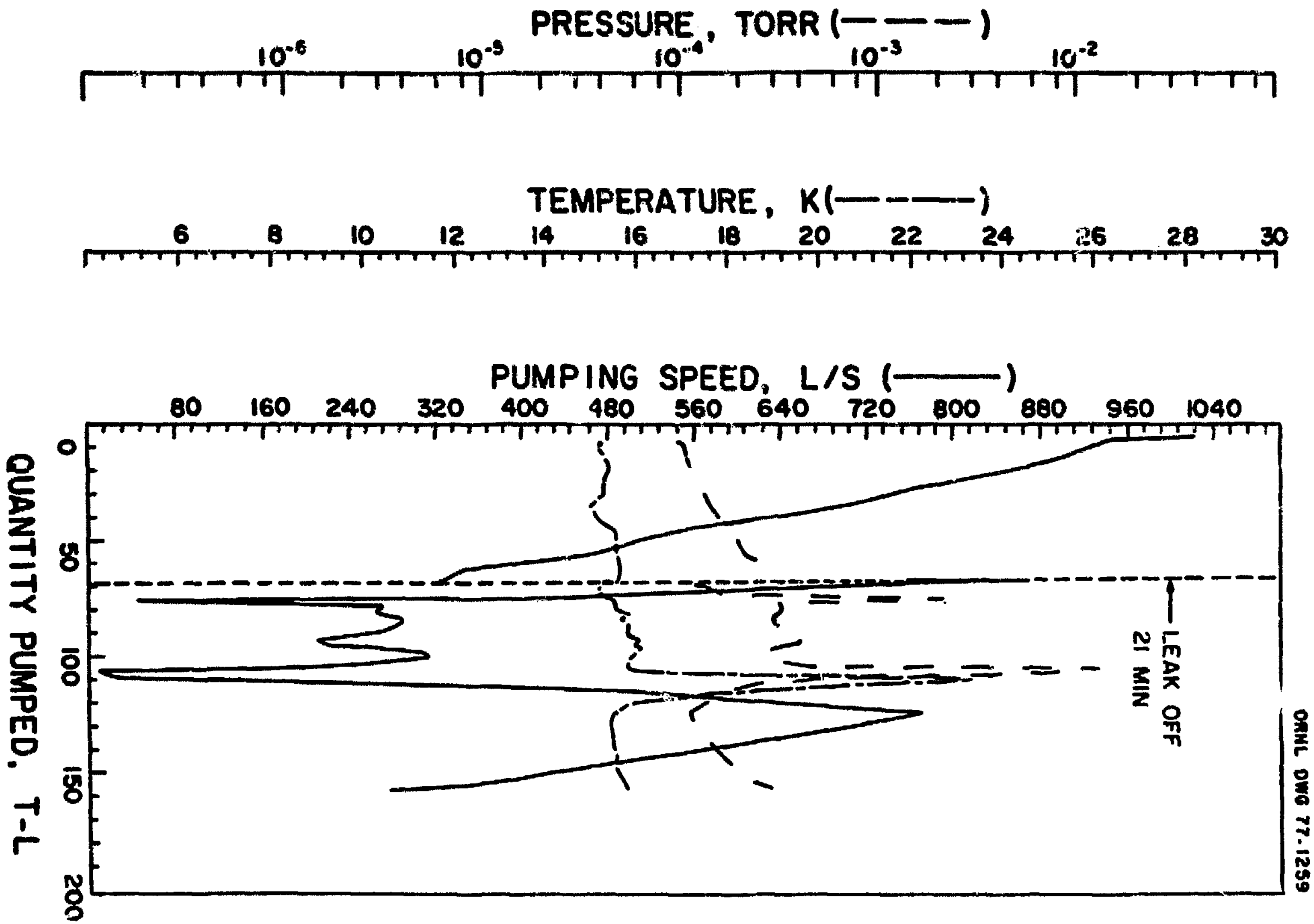
PRESSURE, TORR (— — —)
10^{-6}
10^{-5}
10^{-4}
10^{-3}
10^{-2}
TEMPERATURE, K(— - — - —)
6 8 10 12 14 16 18 20 22 24 26 28 30
PUMPING SPEED, L/S (———)
80 160 240 320 400 480 560 640 720 800 880 960 1040
0 50 100 150 200
QUANTITY PUMPED, T-L
LEAK OFF
21 MIN
ORNL DWG 77-1259

FIG. 4

were obtained in many instances. High pumping speeds can be restored by warming the cryosorption panel between runs. Because diffusion hinders performance, improved results can be expected from adsorbents with higher diffusivities.

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FIGURE CAPTIONS

ORNL DWG NO.	
77-1251	FIG. 1. Schematic of the experimental apparatus.
77-1530	FIG. 2. Dependence of deuterium pumping speed on gas feed rate.
77-1255	FIG. 3. Deuterium pumping for four consecutive runs with various indicated treatments between runs.
77-1259	FIG. 4. Deuterium pumping for two consecutive runs (D4 and D5) in which the pump temperature was maintained between runs.