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DEUTERIUM PUMPING SPEED MEASUREMENTS ON 77 K CRYOPANELS AND
IMPLICATIONS FOR D-T RETENTION IN NEUTRAL BEAM SYSTEMS

By

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Abstract

An upper limit for the pumping speed of deuterium on 77 K surfaces has been determined by in-situ pressure measurements in a TFTR neutral beam line pumped by 423 m² of LN₂-cooled cryopanel. The measurement has importance for estimating the tritium retention in the beam line following operation of the ion sources with tritium. No D₂ pumping was observed. An upper limit for D₂ pumping on 77 K surfaces of $\leq 2.4 \times 10^{-7}$ Vs cm² was determined, corresponding to a D₂ sticking coefficient of $\leq 1.5 \times 10^{-8}$. Based on the upper limit a D-T retention factor, equal to the ratio of retained D-T to D-T input, has been determined to be $\leq 5 \times 10^{-3}$. This upper limit for D-T retention bounds the tritium inventory within the beam line to a small fraction of the tritium throughput. Comparably small upper limits for hydrogenic sticking coefficients, of the order of 10^{-6} - 10^{-10} , have been determined from a review of H₂O cryotrapping measurements at 77 K and from the physical adsorption studies of H₂ on H₂O at 4 K.

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MASTER

1.0 Introduction

For the D-T phase of plasma experiments in the Tokamak Fusion Test Reactor (TFTR), the plasma will be fueled with four 120 keV neutral beam injection systems that deliver approximately 30 MW to the tokamak target plasma.¹ Two of the neutral beam injectors will be fueled with deuterium and two fueled with tritium to ensure a 50/50 fuel mix and to capitalize on the enhanced confinement discharge mode discovered in TFTR which requires beam fueling.^{2,3} An important technical consideration is the tritium inventory in the neutral beam injection systems because of the large surface areas of the beam line vacuum vessels which will be exposed to tritium.⁴⁻⁶ A tritium input of 0.23 g per discharge is the estimated tritium input requirement for the full power D-T experiments. Only a small fraction (~5%) of this 0.23 g tritium-beam input is accelerated to high energy and injected as neutrals in the tokamak. The majority of the tritium input ends up adsorbed onto the LHe cryopanel in the beam system. When the LHe cryopanel is regenerated, the D-T loading from a planned operational sequence (~40,000 torr-liters) will desorb, exposing the surface area of the large LN₂ cryopanel (423 m²) and the remaining internal surface area of the beam line vacuum vessel (1218 m²) to a hydrogenic pressure in the range of ~1 torr for the ~10³ second desorption period.⁴ The adsorption or pumping speed of hydrogen isotopes on 77 K surfaces typically is assumed to be negligible. However, considering the large surface area of the neutral beam LN₂ cryopanel, a quantitative upper limit for hydrogenic pumping on 77 K surfaces is required in order to bound the tritium inventory. In Sec. 2.0, we describe in-situ measurements of the deuterium pumping speed on the LN₂ cryopanel of an operating TFTR neutral beam line. No pumping was observed. The upper limit for D₂ pumping estimated from the in-situ measurements is

compared with related observations from the vacuum technology (Sec. 3.0) and surface physics (Sec. 4.0) literature.

LN₂-cooled cryopanel are typically fabricated with metallic (aluminum or Cu) cryosurfaces and chevrons that are often coated with a black paint to increase the emissivity to near unity. Since the dominant residual gas in most vacuum systems is H₂O, the real surface of an LN₂ cryopanel exposed to subsequent gas adsorption is an amorphous form of H₂O-ice. Depending on the H₂O deposition rate and thermal history of the cryogenic surface, the ice layer that forms is a clathrate structure that exposes a high surface area ($\leq 600 \text{ m}^2/\text{g}$) to the vacuum environment.⁷⁻⁹ For any gas that has a reasonable sticking probability on such surfaces the pumping capacity can be significantly enhanced over adsorption on bare surfaces without prior or concurrent H₂O adsorption. Such "cryotrapping" phenomena have been observed for N₂ and Ar pumping on 77 K surfaces,^{7,10} where both the adsorption rate and adsorption capacity are dependent on the co-adsorbed quantity of H₂O. These studies^{7,10} have not detected H₂ pumping on H₂O at 77 K (see Sec. 3.0); however, an upper limit has been estimated to compare with the data in this paper.

In addition to the relevance to the problems of tritium retention in neutral beam systems and the vacuum technology of LN₂ cryopanel, an upper limit for hydrogenic pumping on 77 K surfaces is also important for a number of surface physics problems with astrophysical implications.^{8,9} Amorphous ice is believed to be a major component of interstellar dust,¹¹ comets,^{12,13} and the satellites of the outer planets.¹⁴ Hydrogenic adsorption and recombination on interstellar dust grains have been subjects of intense study in order to quantify the role of such

particles in the formation of interstellar molecules.^{15,16} Hydrogenic adsorption studies¹⁷ related to this problem show negligible binding of H₂ on H₂O above 20 K (see Sec. 4.0), thus supporting the conventional assumption that there is no significant hydrogenic pumping on 77 K surfaces.

2.0 In-Situ Measurements of D₂ Pumping in a TFTR Neutral Beam Line

In this section we describe measurements of the D₂ pumping on the large area LN₂ cryopanel that are contained within a TFTR neutral beam line. A pictorial diagram of one of the TFTR neutral beam lines is shown in Fig. 1. The primary components of the beam line are housed in a 46 m³ stainless steel vacuum vessel. A simplified schematic of the vacuum pumping and cryogenic systems for one of the four TFTR neutral beam lines is shown in Fig. 2. Detailed discussions of the design of these systems are given in Refs. 18-20. The primary pumping of hydrogenic species is provided by the 29.5 m² of LHe-cooled cryopanel. The sources of hydrogenic species include cold neutral gas effluent from the ion sources and neutralizers, reflected neutrals from the ion dumps, and the neutralized plasma exhaust which streams down the beam lines from the torus following cessation of a plasma discharge. A much larger set of cryopanel (423 m²) cooled with LN₂ thermally shields the LHe panels and provides additional pumping for molecules which condense at or above 77 K (primarily H₂O, CO₂, and CH₄ for the TFTR residual gas composition). A third pumping component, a nominal 3500 l/s turbo pump is installed on the beam line to provide He pumping during beam line operation, and to pump down the beam line during atmospheric exhaust and regeneration operations.

The D₂ pumping measurements were made during a LHe cryopanel

regeneration cycle of one of the four neutral beam injection systems installed on TFTR. This procedure is done routinely whenever the total hydrogenic loading of the LHe cryopanel approaches an equivalent pressure of approximately 1 torr when confined to the beam line volume (46,000 l). This criterion ensures that the hydrogenic pressure during a possible loss-of-vacuum accident is well below the explosive limit for hydrogen-air mixtures (≥ 3 torr partial pressure of H_2 at atmospheric pressure). The LHe cryopanel is regenerated by shutting off the flow of LHe to allow the panels to warm up to ~ 25 K by briefly thermally shorting the system by raising the beam line pressure to ~ 5 mtorr. The LN_2 panels are maintained at 77 K during this procedure and are normally regenerated only prior to atmospheric venting of the beam line. Normally during a LHe cryopanel regeneration cycle, the beam line turbo pump is connected to the beam line volume enabling continuous pumping of the hydrogenic effluent as the LHe panels warm up. The thermal time constants of the LHe system are such that the regeneration scheme takes approximately 15 minutes for the entire LHe panel area to rise above hydrogenic desorption temperatures.

Following a successful regeneration of the LHe cryopanel, LHe was not re-introduced into the LHe cryopanel circuits and the beam line turbo pump was valved off. For the purposes of the measurements to be described, this left the isolated volume of the beam line pumped only by the LN_2 panels. Deuterium gas was then introduced into the beam line volume until a pressure of ~ 0.1 torr was obtained as determined by a Pirani gauge calibrated for D_2 (noted as P1 in Fig. 2). The pressure in the beam line was recorded for a period of eight minutes. Figure 3 shows the first three minutes of pressure recording after the D_2 pressure was raised to 0.11 torr at

$t = 0$. No change in the pressure reading was observed over the eight minute period within the measurement resolution of $\pm 1\%$.

The observed constancy of the D_2 pressure can be used to calculate an upper limit for the pumping speed, S , of D_2 on the LN_2 surfaces. Since the final pressure p_f , after eight minutes of observation time Δt , differed by less than 1% from the initial pressure, p_i ,

$$p_f/p_i = \exp(-\Delta t/\tau) \leq 0.99 \quad . \quad (\text{Eq. 1})$$

The pumping time constant, $\tau = V/S$, is at least a factor of 10^2 larger than the observation time

$$V/S = \tau > 10^2 \Delta t \quad . \quad (\text{Eq. 2})$$

Substituting the beam line volume $V = 46,000$ l into the above relation yields an upper limit for D_2 pumping speed: $S < 1$ l/s for the entire beam line. With the approximation that the entire surface area of the LN_2 cryopanel (423 m^2) is equally accessible to the D_2 within the beam line volume, an upper limit for the pumping speed per unit area, S' , is simply $S/423 \text{ m}^2$ yielding $S' \leq 2.4 \times 10^{-7} \text{ l/s} \cdot \text{cm}^2$. A sticking coefficient, σ , can be defined as the ratio:

$$\sigma = S'/3.64 (T/M)^{1/2} \quad , \quad (\text{Eq. 3})$$

where the denominator is ideal volume flow rate or "black hole" pumping speed for a gas of mass M (amu) at temperature T (K). For D_2 at 77 K, the measured upper limit

for S' yields a sticking coefficient limit of:

$$\sigma < 1.5 \times 10^{-8}$$

The above upper limit for D_2 pumping speed can be used to calculate an upper limit for the deuterium retained on the LN_2 panels during the pressure excursion which occurs during a LHe cryopanel regeneration cycle. The proposed D-T operational cycle on TFTR⁴ involves one day of plasma operations with two D- and two T-fueled neutral beam lines, and generates approximately 150,000 torr-liters of D-T on the LHe cryopanel of the four beam lines, or approximately 37,500 t-l per beam line. Upon regeneration an approximate average pressure; $p = 37,500/2$, $V = 0.4$ torr is produced with a typical desorption time of $t_d \sim 10^3$ sec.

We can define a hydrogenic retention factor, RF , for the LN_2 panels as the ratio of the retained (or pumped) deuterium to the deuterium input. For the above regeneration cycle, the retained deuterium is the product of the average local deuterium pressure, p' , above the LN_2 panels, the deuterium pumping speed, and the exposure time, t_d . The deuterium temperature in the vicinity of the LN_2 panels is assumed to be 77 K, therefore, the local pressure is related to the pressure, p_g , as measured on a system gauge calibrated at room temperature (such as P1 in Fig. 2) by the usual thermomolecular relation

$$p' = (77/300)^{1/2} p_g \quad (Eq. 4)$$

Therefore, the estimated upper limit for a hydrogenic retention factor is:

$$RF \sim [0.5(p_g t_d)S/37,500] \leq 5 \times 10^{-3} \quad (\text{Eq. 5})$$

3.0 Sticking Coefficient Estimates from Cryotrapping Studies

Cryotrapping normally is a term applied to the pumping of a gas species which is normally not condensible at a pumping surface but is trapped by the prior or concurrent condensation of a condensable species.^{7,10} H₂O condensed onto 77 K surfaces has been studied as a cryotrapping agent for a number of species which would have little or no significant pumping on bare 77 K surfaces (see Table 1). Schmidlin et al.⁷ have measured cryotrapping rates for N₂ on H₂O (77 K) which shows a trapping rate, $w = 8 \times 10^{-3}$ N₂ molecules per H₂O molecule (Fig. 4a). The observed N₂ partial pressure dependence (Fig. 4b) of the cryotrapping rate, ρ , to H₂O condensation rate, R , fits a Langmuir type isotherm²¹:

$$\rho/R = \beta'p/[1 + (\beta'p/w)] \quad (\text{Eq. 6})$$

where $\beta' = w/p_2'$, where p_2' is the Langmuir saturation pressure.

From the N₂ isotherm, Schmidlin et al.⁷ obtain a value of $\beta' = 6.6 \text{ torr}^{-1}$. No detectable cryotrapping of H₂ on H₂O (77 K) was seen and the estimated upper limit for β' was $<3 \times 10^{-3}$. We can use this upper limit for an H₂/H₂O cryotrapping rate at 77 K, and an estimate of the worse-case H₂O partial pressures in the beam line, to generate an upper limit for H₂ cryotrapping on the beam line LN₂ cryopanel.

The worse case partial pressure of H₂O in a beam line is of the order $\sigma: 1 \times 10^{-7}$ torr. This partial pressure causes the maximum permissible level of H₂O within the TFTR torus ($\sim 2 \times 10^{-8}$ torr), and can be the result of an H₂O leak from internal hardware in the beam line of magnitude $Q_1 \sim 10^{-1}$ torr-l/s, or a similar H₂O outgassing load during the initial stages of pump down of the $\sim 10^3 \text{m}^2$ beam line vacuum vessel. At a partial pressure of 1×10^{-7} torr, the condensation rate of H₂O onto the LN₂ panels is $\sim 5 \times 10^{13} \text{cm}^{-2} \text{s}^{-1}$. From the Schmidlin et al.⁷ data shown in Fig. 4, a sticking coefficient (or "trapped" fraction) for N₂ on H₂O (77 K) is 5×10^{-7} for low ($< 10^{-3}$ torr) N₂ pressures. The upper limit for the H₂ sticking coefficient under these conditions is estimated to be lower by the ratio of $\beta'_{\text{N}_2}/\beta'_{\text{H}_2} > 2 \cdot 10^3$. Therefore,

$$\sigma \leq 3 \times 10^{-10} \quad (\text{low pressure})$$

At high pressures the adsorption of N₂ follows a Langmuir isotherm (Fig. 4b). For the pressures characteristic of the measurements in Fig. 3 and regeneration cycles ($\sim 10^{-1}$ torr), the N₂/H₂O sticking coefficient is $\sim 10^{-2}$; Therefore, the estimated upper limit for the H₂/H₂O sticking coefficient is

$$\sigma \leq 5 \times 10^{-6} \quad (\text{high pressure})$$

assuming the possible H₂ trapping sites are saturable according to a similar Langmuir type isotherm. The high and low pressure estimates of the H₂/H₂O (77 K)

sticking coefficients are small, and it is interesting to note that the upper limit inferred from the Fig. 3 data falls between these two estimates. Cryotrapping of H₂ by concurrent deposition of H₂O vapor if it occurs at all at 77 K is an insignificant process.

4.0 Measurements from the Astrophysical Literature

The adsorption of H₂ onto H₂O surfaces at low temperatures has received considerable attention in the astrophysical literature because of the relevance of the phenomena to molecule formation on interstellar dust grains,¹⁵⁻¹⁶ and the presence of amorphous ice in comets^{12,13} and the satellites of the outer planets.¹⁴ Much of this literature has examined adsorption-desorption phenomena at surface temperatures <20 K where the coverage of both H₂ and H is demonstrably high.¹⁷ Several of these studies support the contention that the retention of H₂ on H₂O surfaces at 77 K should be negligible. However, the porous structure which amorphous ice develops under vacuum deposition at low deposition rates (which is the primary condensate on LN₂ cryopanel) is an interesting clathrate-type structure with high porosity^{7,8} that can significantly affect gas-surface interactions.

Mayer and Pletzer^{8,9} have examined the N₂ adsorption properties of H₂O vacuum deposited onto 77 K substrates at relatively low deposition rates (~10¹⁵ cm⁻² s⁻¹). They obtain an adsorption isotherm which saturates at a ten times higher coverage (6 millimole N₂ per gram of H₂O) than the data of Schmidlin et al.⁷ The difference may be the result of the slower desorption rate of 10¹⁵ cm⁻² s⁻¹ in comparison to the higher rates (10¹⁶ - 10¹⁸ cm⁻² s⁻¹) used in the Schmidlin et al.⁷ studies. The lower rates may result in a more porous structure; however, the

adsorbate surface areas inferred from both measurements are similarly high, in the range of 350 - 420 m² g⁻¹ for the Mayer and Pletzer^{8,9} study and ~600 m² g⁻¹ for Schmidlin et al.⁷ study. Despite the discrepancies between the two studies, it is clear that the amorphous ice structure which forms at 77 K has significant pumping capacity for molecules such as N₂ which will bind at these temperatures.

The surface binding energy, E_b, of H₂ onto amorphous ice has been measured by Lee¹⁷ to be E_b/k = 860 K. This low value, which is characteristic of weak physisorption can be used to show that negligible H₂ adsorption should occur at 77 K. Assuming first-order kinetics for the H₂ desorption process, the surface residence time, t_R, can be calculated with the usual formulation²¹:

$$t_R = \nu^{-1} \exp (E_b/k = kT) \quad . \quad (Eq. 7)$$

Substituting 10¹³ s⁻¹ for the frequency factor ν , yields a residence time of only 7 × 10⁻⁹ s at 77 K.

We can estimate the instantaneous D₂ coverage which would be present on the LN₂ cryopanel during the regeneration cycle described in Sec. 2.0. Since p(D₂) ~0.2 torr, the D₂ molecular incidence, Γ_D , rate of T = 77 K is:

$$\Gamma_D = 6 \times 10^{20} \text{ cm}^{-2} \text{ s}^{-1} \quad .$$

The instantaneous D_2 coverage is the product $t\Gamma_D = 4 \times 10^{12} D_2$ molecules cm^{-2} , which is a small fraction ($\sim 10^{-3}$) of monolayer coverage. Therefore, even during the time (10^3 s) of high pressure D_2 exposure, the LN_2 panels are retaining a negligible fraction, $\leq 0.48 t/37,500 t-l = 1.3 \times 10^{-5}$ of the gas phase deuterium.

5.0 Conclusions and Implications for Tritium Retention in Neutral Beam Systems

From the data and discussions presented in Secs. 2.0 - 4.0, the adsorption of deuterium onto 77 K surfaces is a negligible process. Using the in-situ measurement for the deuterium retention reactor, tritium retention on the TFTR LN_2 cryopanel can be estimated, assuming there are not important isotopic or radiolytic effects. For the proposed D-T operation scenario,⁴ the tritium-fueled neutral beam will be regenerated after 2688 torr-liters of tritium is input. Calculations⁴ of the injection efficiency based on scaling of deuterium injection indicate that only ~5% of the tritium input is injected into the torus; the majority will be absorbed on the LHe cryopanel. Upon regeneration, the tritium retention on the 77 K surfaces is estimated to be ≤ 13.5 torr-liters (T_2) = 5 mg per beam line using the retention factor upper limit measured in Sec. 2. For the entire D-T phase of experimental operations on TFTR, an estimated maximum of 75 regeneration cycles would be required. To estimate the total retention after 75 cycles, the kinetics of the adsorption process is required. To be consistent with the N_2 cryotrapping data (Sec. 3.0), a Langmuir type (Eq. 6) relation for the retention vs. exposure would be appropriate. However, since no hydrogenic retention has been measured in any of the experiments discussed in this paper, the Langmuir saturation parameter, p_2' for D_2 , cannot be estimated.

Independent estimates of the tritium retention in TFTR are described in a companion paper⁶ that are based on D-T adsorption measurements on small samples of beam line materials, including the LN₂ cryopanel material. These measurements were performed at ambient temperatures, therefore, the relevant adsorption on 77 K H₂O is not being measured. However, these D-T adsorption measurements were performed over a reasonably large range of exposure and show retention varying as $(p \cdot t)^{1/2}$. If the 5 mg retention estimate from the in-situ measurements is scaled by the square root of the exposure for 75 cycles, the total retention estimate is:

$$\leq 0.04 \text{ g per beam line}$$

This result compares favorably with the ambient temperature retention estimates,^{4,6} even though there is no good reason to expect agreement.

The tritium retention estimate based on the upper limit for deuterium pumping on 77 K surfaces assumes there are no radiolytic effects with tritium adsorption which may increase the adsorption. Several possible effects remain to be quantified. Radiation damage from transiently adsorbed tritium on the H₂O substrate may lead to increased D-T trapping, or beta emission may promote the formation of tritiated hydrocarbons and H₂O which would be strongly adsorbed on the LN₂ cryopanel. Some indications of radiolytic enhancement of tritium retention have been observed in the ambient temperature adsorption measurements, where differences in the total D-T retention were found to be dependent on the D/T ratio during the exposure.^{4,6}

6.0 Conclusions

No pumping of D₂ was observed on a large area (423 m²), LN₂-cooled cryopanel. An upper limit for D₂ pumping of $\leq 2.4 \times 10^{-7}$ l/s · cm² was determined, corresponding to a sticking coefficient of $\leq 1.5 \times 10^{-8}$. An upper limit for D-T retention on the LN₂ cryopanel was determined to be $\leq 5 \times 10^{-3}$ of the D-T input typical of the regeneration gas load in a TFTR neutral beam line. Comparably small sticking coefficients (10^{-6} - 10^{-10}) have been determined from a review of H₂O cryotrapping studies at 77 K and physical adsorption studies of H₂ on H₂O at 4 K. Using the 5×10^{-3} retention factor, the calculated tritium inventory in one TFTR neutral beam line is ≤ 5 mg per regeneration cycle which exposes the beam line to a tritium throughput of 0.9 g.

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TABLE 1: H₂O Cryotrapping Results (After Hopson, Ref. 10)

<u>Non-Condensable</u> <u>Molecule</u>	<u>Substrate</u> <u>Temperature</u> <u>(K)</u>	<u>Minimum Ratio</u> <u>of H₂O Molecules</u> <u>to Non-Condensables</u>	<u>Reference</u>
H ₂	~20		22
O ₂	60 - 90	50	22
H	4 - 10	--	22
N	77	50	22
N ₂	77	125	7
Ar	77	77	7
H ₂	77	$\geq 3 \times 10^5$	7
He	77	$\geq 10^5$	7

Figure Captions

Fig. 1. TFTR 120 keV neutral beam line.

Fig. 2. Simplified vacuum and cryogenic schematic for one TFTR neutral beam line.

Fig. 3. D_2 pressure in an isolated TFTR neutral beam line that is being pumped only by the 423 m^2 of LN_2 -cooled cryopanel. The D_2 gas input was shut off at time $t = 0$.

Fig. 4. (a) Pumping speed of N_2 vs. condensation rate (R) of H_2O on 77 K surfaces.
(b) Trapped N_2 per condensed H_2O on a 77 K surface vs. N_2 partial pressure (From Schmidlin, Heflinger, and Garwin, Ref. 7).

Fig. 5. The physical adsorption binding energy, E/k , of H_2 on H_2O ice at 4 K vs. H_2 coverage (From Lee, Ref. 17).

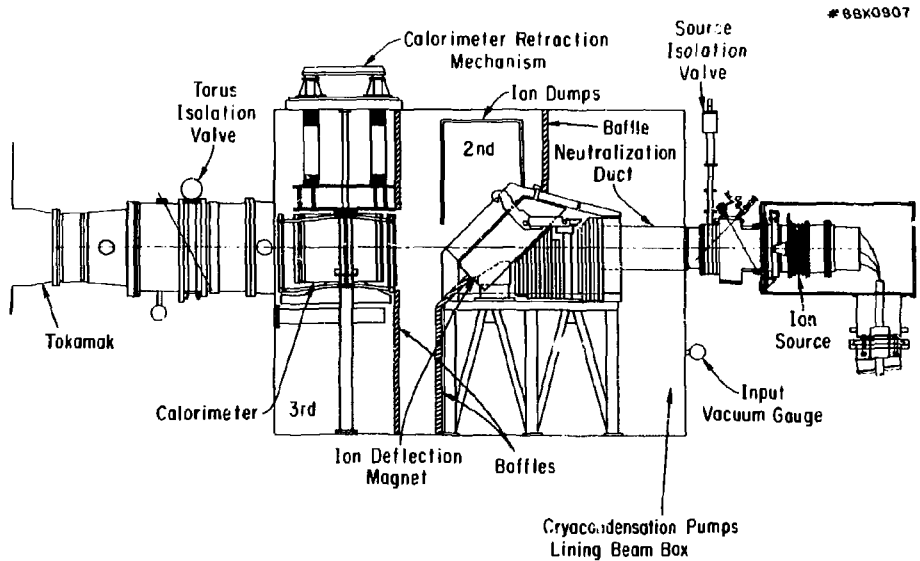


Fig. 1

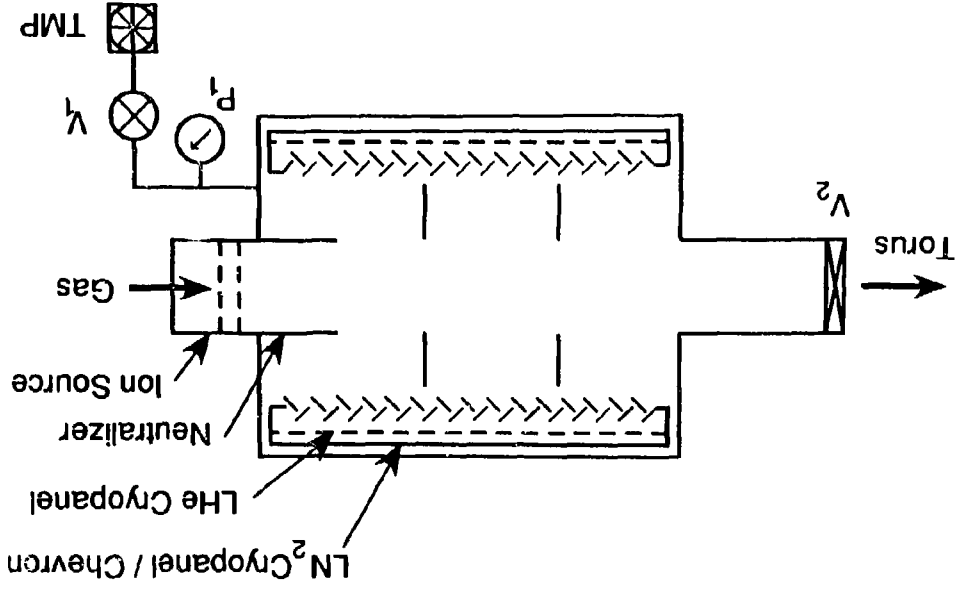
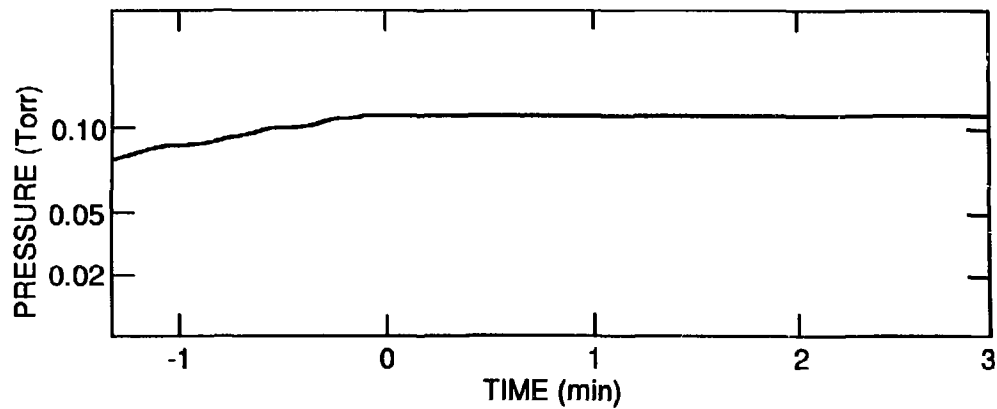


Fig. 2

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Fig. 3

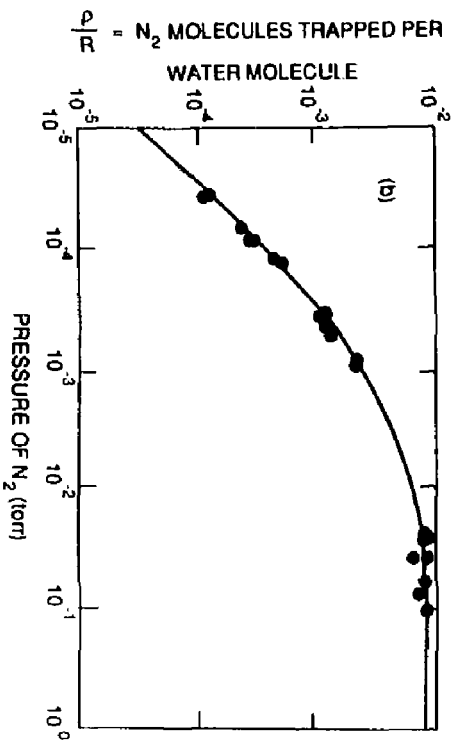
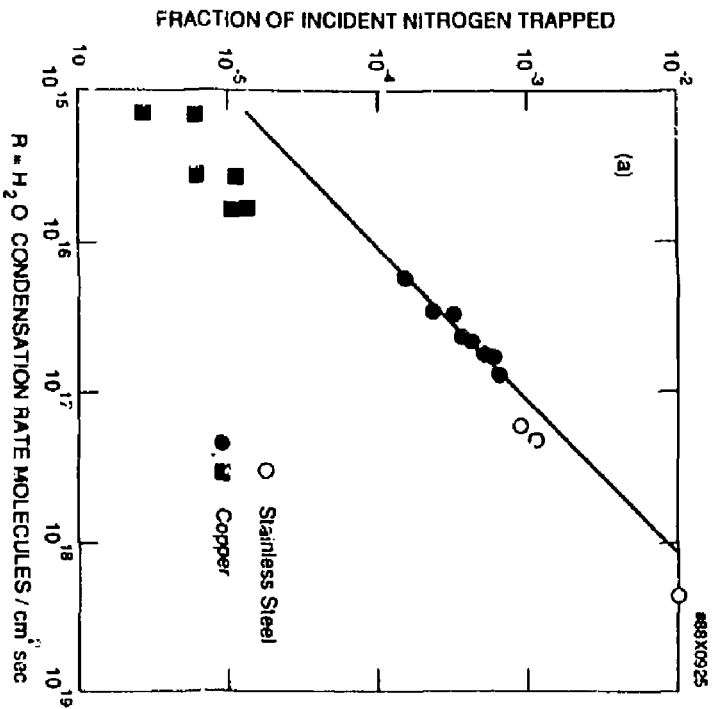


Fig. 4

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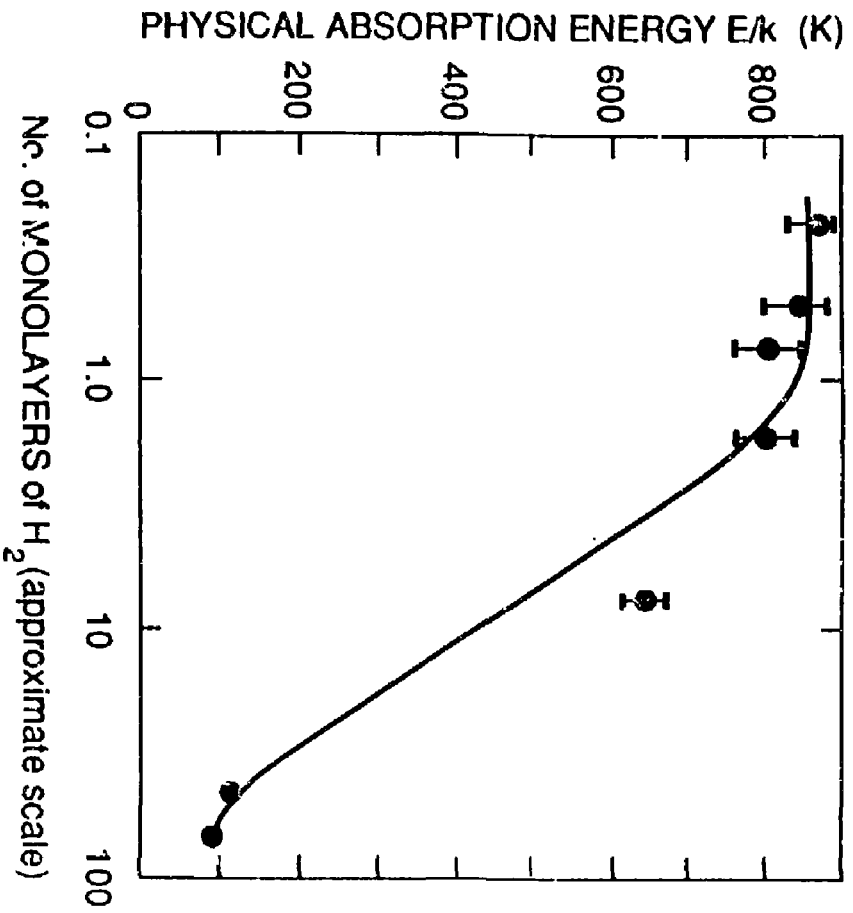


Fig. 5

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