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Grant No. NGR. 31-003-043, Supplement No. 1

On: Experimental Investigations of Desorption
Cooling Methods at Cryogenic Temperatures

Covering Period: May 1968 - June 1970

Submitted by:

John G. Daunt

Dr. John G. Daunt
Cryogenic Center

Date: May 1971

FINAL REPORT ON NASA GRANT NO. NGR-31-003-043, SUPPLEMENT NO. 1

ON

EXPERIMENTAL INVESTIGATIONS OF DESORPTION COOLING METHODS
AT CRYOGENIC TEMPERATURES

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SUMMARY

This is the Final Report on Grant No. NGR-31-003-043, Supplement No. 1, covering the period May 1968 to June 1970.

The persons associated with the work are listed, Dr. John G. Daunt being the Principal Investigator and Dr. Carol Z. Rosen being Research Associate.

The results of the work are presented in the form of a pre-print for a paper to be submitted to "Cryogenics." The results give the performance of two low temperature desorption refrigeration systems which were investigated experimentally. Using He⁴ as the working gas and synthetic zeolite as the working substance, it was found that temperatures down to 1.8K could be readily obtained. Measurements were made of the rate of warm-up under various power loadings (powers up to 12.8 milliwatts were employed) at the final low temperatures. With the larger cryostat containing 130 g. of synthetic zeolite refrigeration was maintained for periods longer than 25 hours. An assessment was made on the limitations of the cooling process. The data are presented in tabular form for the expected duration of refrigeration and total refrigerative capacity at various temperature levels from 3K to 6K for one kilogram of synthetic zeolite; as an example, this would permit refrigeration to be maintained between 4K and 5K for 130 hours for a 1 milliwatt power loading.

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EXPERIMENTAL INVESTIGATIONS OF DESORPTION COOLING METHODS
AT CRYOGENIC TEMPERATURES

1. Period Covered

This report covers work carried out in the period May 1968 to June 1970 in the Cryogenics Center, Stevens Institute of Technology, under the direction of Dr. J. G. Daunt, which work was assisted by Grant No. NGR-31-003-043, Supplement No. 1, from NASA.

2. Personnel

The following persons were associated with the research program during the period covered by this report.

Dr. John G. Daunt	Principal Investigator
Dr. Carol Z. Rosen	Research Associate
J. Baer	Technician
J. Clavin	Technical Assistant
J. Hughes	Technical Assistant

3. Summary of the Results of the Research Program

Two cryostats were built for desorption cooling in the temperature range 20K to 2K, using desorption of ^4He gas from synthetic zeolite. The desorption cooling experiments carried out with these cryostats are described below in Section 4. The initial temperatures chosen were 12K, 10K, 8.0K and 6.0K and initial pressures between 0.25 atmos and 1.00 atmos. Final temperatures reached ranged down to 1.83K. Measurements were made of the rate of warm-up under various power loadings (powers up to 12.8 milliwatts were employed) at the final low temperatures and the results, as a function of the average temperature, are presented below in Section 4. With the larger cryostat containing 130 g. of synthetic zeolite refrigeration was maintained for periods longer than 25 hours. An assessment has been made (Section 4) on the limitations of the cooling process based on our results. Data are presented in tabular form (Section 4) for the expected duration of refrigeration and total refrigerative capacity at various temperature levels from 3K to 6K for one kilogram of synthetic zeolite; as an example, this would permit refrigeration to be maintained between 4K and 5K for 130 hours for a 1 milliwatt power loading.

Section 4, below, gives the results of the work in detail and is in the form of a preprint of a paper to be submitted to "Cryogenics" for publication.

4. Desorption Cooling below 12K.

I. INTRODUCTION

The experimental investigations reported herewith were undertaken to study the potential application of desorption cooling as a second stage for further lowering of temperature in miniaturized refrigeration systems. Desorption cooling offers some advantages, especially in simplicity, for maintaining temperatures in the range 2K to 10K starting from temperatures readily produced by mechanical refrigerators¹⁾ or solid hydrogen packs.²⁾

The method is well-known having being devised many years ago by Simon³⁾ and, for example, used for helium liquefaction by Mendelssohn⁴⁾ as early as 1931. Moreover, some experimental studies of the performance of desorption cooling systems in the temperature range below 20°K using activated charcoal as the adsorbent were made some time ago by van Itterbeek and van Dingenen⁵⁾ and by Kanda⁶⁾. More recently Bewilogua and Reichel⁷⁾ have reported similar experimental studies at higher temperatures (40°K to 70°K) using silica gel as the adsorbent. Some of our preliminary results on desorption cooling using various adsorbents, namely: charcoal, silica gel, alumina and synthetic zeolite, have already been reported⁸⁾⁹⁾ by one of us (JGD). These results indicated that synthetic zeolite (Linde Molecular Sieve, type 13X) was a more favorable material for desorption cooling, using He⁴ as the working gas, in the temperature range 2K to 10K than many others. A more detailed experimental study, therefore, of desorption of He⁴ from synthetic zeolite seemed justified and the results are presented below. In the meantime we have published¹⁰⁾¹¹⁾ detailed measurements of the adsorption isotherms of He³ and He⁴

on synthetic zeolite 13X in the temperature range 4.2K to 20K, together with the equilibrium thermodynamic data deducible therefrom. The latter are of value in interpretation of the present desorption experiments.

A description of the synthetic zeolite adsorbent together with details of its preparation is given in Section II. Section III describes the experimental arrangements and procedures. The latter covered not only measurement of the final temperatures (T_f) and final pressures (p_f) reached from a variety of initial temperatures (T_i) and pressures (p_i) but also covered extended measurements of the maintenance of the low temperatures for periods up to 25 hours. During these extensive runs, heat was applied electrically in various measured amounts up to about 13 milliwatts to the desorbed sample cell and the resulting warming rates recorded. Data from many of these runs are reported in Section IV together with other pertinent experimental results that were obtained.

Section V discusses the results in detail, presents an interpretation of the physical processes determining the desorption coolings and the refrigeration capacities, and provides in tabular form engineering data for future design.

II. DETAILS OF THE ADSORBENT AND ITS PREPARATION

The synthetic zeolite which was used in our experiments as adsorbent was obtained from the Linde Division of Union Carbide Corporation and is referred to as "Molecular Sieve 13X". The chemical composition of a hydrated unit cell is given by:

$\text{Na}_{86} \left\{ (\text{AlO}_2)_{86} (\text{SiO}_2)_{106} \right\} \cdot 276\text{H}_2\text{O}$. It consists of SiO_4 and AlO_4 tetrahedra which build up structural units of truncated octahedra, called β -cages^{12) 13) 14)} of interior diameter 6.6\AA . The β -cages are tetrahedrally arranged and there are 8 β -cages in a unit cell. The tetrahedral framework of the β -cages enclose larger polyhedral cavities called α -cages^{13) 15)} having a free diameter of about 11.8\AA . There are 8 α -cages per unit cell, which is a cube of side 24.95\AA . The 8 α -cages and 8 β -cages per unit cell provide an intracrystal void volume of $8,080\text{\AA}^3$, or about 50 percent of the total volume of the crystal.

Pellets of zeolite 13X were used in the experiments reported here. They were specified to contain 20% by weight of an "inert clay binder". They were approximately 1/4 in. long and 1/16 in. in diameter. The partially dehydrated pellets used contained about 2% by weight of water and had a density of about 1.0 gm/cm^3 ; this water is necessary to prevent structural distortion¹⁶⁾. The different volumes occupied by the crystals and the binder¹⁶⁾ are noted in Table I, taking one pellet as the basis for 100% of the volume.

In both the cryostats used in our experiments, each desorption cell was first filled with pellets to its maximum capacity. In doing this, an electric vibrator was used to maximize the filling. Then this filling of zeolite was removed and dried in a vacuum oven at 110°C for over 24 hours. It was then weighed in a covered weighing bottle at room temperature. An identical heating and weighing procedure was repeated several times until there was

no further weight change. The final weight of the filling for the first (small) cryostat (system A) was 3.53 g, which corresponded to a pellet density of about 1.08 g/cm^3 . This treated and measured quantity of zeolite was then replaced in the desorption cell. The weight of the pellets for the second cryostat (System B) was measured after an identical procedure and was 129.6 g.

After the above procedure of filling each desorption cell, the following preparatory treatment was carried out before experiments began: (1) high-vacuum pumping at 60°C for several hours; (2) high-vacuum pumping at room temperature for more than 48 hours; (3) purging several times with dry He^4 gas at room temperature and (4) high vacuum pumping at room temperature for more than 48 hours. It is noted that purging with the adsorbate is recommended by Brunauer¹⁵⁾ to be one of the most effective methods of purifying the surface.

Furthermore, between each desorption run we adopted a routine of high vacuum pumping of the adsorption cell at room temperature for a 48 hours in order to assure reproducibility of our data.

III. EXPERIMENTAL ARRANGEMENTS AND PROCEDURES

Two desorption apparatus were used in the experiments, a small scale one (System A) containing 3.53 g. of 13X adsorbent and a larger one (System B) containing 129.6 g. of 13X adsorbent.

System A is described first. In it the adsorbent was contained in a desorption cell, V_1 , comprising of a copper vessel of internal volume 5.70 cm^3 and mass 24.2 g to which was attached a

calibrated germanium resistance thermometer (Solitron, type No. 2401), a 100 ohm heater comprised of a bifilarly wound awg No. 32. Evanohm wire and a 2.8 mm. I.D. thin walled stainless steel filling and exhaust tube which led to the room temperature part of the cryostat. The vessel V_1 was located within an inner vacuum vessel, V_2 , which in turn was located within an outer vacuum vessel V_3 . The outer vessel V_3 was immersed in a liquid He^4 bath at 4.2K and could be evacuated to high vacuum.

The inner vacuum vessel, V_2 , was equipped with two calibrated germanium resistance thermometers, (same type as on V_1), a 200 ohm heater (similar to that on V_1). One resistance thermometer and the heater were connected to an electronic temperature controller by means of which V_2 could be maintained at any desired preset temperature between 4.2K and 80K with a long term precision of $\pm 0.003\text{K}$. By having initially exchange gas in V_2 , the desorption cell V_1 could be filled with ^4He gas at any desired initial temperature in the above mentioned temperature range. Before starting a desorption run, V_2 would be highly evacuated in order to isolate V_1 thermally from its surroundings.

System B, with the larger amount of adsorbent, incorporated a larger diameter tube for pumping on the desorption cell and a separate tube for measurement of the pressure in the cell. It incorporated otherwise similar general qualitative arrangements as System A and it is sketched in Fig. 1. The significant differences were as follows: The desorption cell, V_1 , was a cylinder of copper with internal dimensions 10.2 cm long and

5.1 cm I.D. and weighing 386 g. The pumping tube connected to V_1 was 0.90 cm I.D. and 5.0 cm long between V_1 and V_2 (see Fig. 1); 1.85 cm. I.D. and 48.0 cm between V_2 and the 60K level, where it enlarged to 3.68 cm I.D. to the top flange of the cryostat. It contained two radiation baffles, as shown in Fig. 1. Above the top flange it enlarged to 5.1 cm I.D. and led through a liquid nitrogen cooled trap to a NRC B-2 oil booster pump backed by a 1/3 h.p. rotary mechanical pump (Welch DuoSeal 1400).

The tube leading to the desorption cell, V_1 , for measurement of the pressure was 2.7 mm. I.D. and 2.5 cm long between V_1 and V_2 and 5.8 mm. I.D. and 81.3 cms. long between V_2 and the top of the cryostat. The pressure in the desorption cell was measured by various gauges located at room temperature, including, for the final pressures, p_f , reported below, helium calibrated Veeco thermocouple gauges, types DV3M (0-1000 microns) and DV5M (1-100 microns).

After each cryostat was cooled to 4.2°K, an experimental desorption run proceeded first by adjusting V_2 to a desired initial temperature, T_i , with exchange gas within it so that V_1 attained the same temperature. Then dried He^4 gas was introduced into V_1 to a predetermined initial pressure, p_i . When equilibrium was attained, V_1 was thermally isolated. This was achieved by high-vacuum pumping of V_2 . The desorption was initiated immediately after p_i and T_i were recorded.

Desorptions were carried out using the 1400 Welch mechanical pump for pumping on the adsorption cells and, in the case of System B, using in addition the NRC B-2 Booster Pump. The final pressures, p_f obtained in the desorption cell were: (1) without the Booster Pump about 50 to 70 microns for pumping with the mechanical pump alone and (2) about 10 to 13 microns for pumping with the Booster pump. These pressure values are corrected ones, corrections having been made in the case of System B for thermomolecular pressure effects¹⁸⁾ and in the case of System A for the pressure drop due to the gas flow up the tube between the desorption cell and the gauge, as well as for the thermomolecular pressure effects.

In each desorption run the temperature of the adsorption cell and its pressure were observed throughout the duration of the run. Moreover measured heating powers were applied in varying amounts, when desired, to the desorption cell and the resulting temperature and pressure changes observed.

IV. THE EXPERIMENTAL RESULTS

The initial data of interest are the minimum final temperatures, $T_{f,min}$ and their associated pressures, p_f , attained by desorption from various initial temperatures, T_i , and pressures, p_i . The observed results, obtained using both cryostats (System A and System B) are presented in Tables II and III, which also include sample data on the time, τ_{min} , taken to reach $T_{f,min}$ from the initial conditions and evaluations of the ratio r , where

$r = T_i/T_{f,\min}$. Table II gives the results for desorption using the mechanical pump (Welsh model 1400) only for both System A and System B, whereas Table III presents the results obtained for desorption using the Booster Pump as well as the mechanical pump on System B only.

Fig. 2 shows a plot of the observed r values as a function of initial pressure, p_i , for initial temperatures, T_i , of 6K, 8K, 10K and 12K for desorption to pressures (p_f) of 50 to 70 microns (mechanical pumping only). Fig. 3 shows a plot of the observed r values as a function of initial pressure, p_i , for an initial temperature $T_i = 8K$ for desorption to 10 to 13 microns (Oil diffusion Booster Pump). It will be seen that the ratio, r , for a given pumping arrangement increases monotonically with increasing p_i and, at constant p_i , r decreases with increasing T_i . It is noted that the r values obtained with both System A and System B are in accord with one another. Also the increase in the ratio, r , for desorption using the Booster pump with System B is clearly shown for the $T_i = 8K$ data. For example, starting at $T_i = 8K$ and $p_i = 0.5$ atmos, use of the Booster Pump decreases the value of $T_{f,\min}$ by about 15% below the value obtained with the mechanical Pump alone.

Fig. 4 presents the desorption data, plotting the ratio, r , versus the initial temperature, T_i , for two different initial pressures, p_i , of about 0.5 and 1.0 atmos. and for desorption to p_f values between 50 and 70 microns. It is evident that the r values increase markedly as the initial temperature is decreased.

The temperature-time history of a typical desorption run is shown in Fig. 5 which is for a run starting with $T_i = 8K$ and $p_i = 0.5$ atmos. In this figure it will be seen that the desorption was first made using the mechanical pump alone, with which the final temperature was reduced to 3.75K in about 40 minutes, and thereafter the temperature remained essentially constant until minute 98. At this time the Booster Pump was activated and further desorption cooling occurred and in about 20 minutes the temperature fell to a steady value of 3.20K. It remained at 3.20K for approximately 20 minutes with a very small warm-up rate, after which time heat was put into the sample by means of the electrical heater in amount 5.0 mw. This heating was maintained until minute 250 and the desorption cell settled down to a reasonably steady rate of warming during this period. At minute 250 the heating was increased to 12.8 mw and was maintained at this value until minute 516. The rate of warming during this period is shown on the graph of Fig. 5. At minute 516 the heating was cut off and at the same time the Booster Pump was deactivated, so that the pumping from this time on was by the mechanical pump alone. By minute 1304, when the run was terminated, the temperature of the adsorption cell had fallen to 4.35K.

Many desorption runs similar to that shown in Fig. 5 was made and from them the steady heating rates, dT/dt , for various input heating powers P , were determined. Typical data obtained in this way using System B are shown in Fig. 6, which plots two curves of the steady warming rate (dT/dt) versus the heating power, P , for warmings at about $3.5K \pm 0.1K$ and at about $5.5K \pm 0.3K$. It is

evident that (dT/dt) increases approximately linearly with P and grows larger as the average temperature is reduced at constant P . It is further evident that the zero power rate of temperature rise is quite small, $(dT/dt) \leq 5$ microKelvin/sec. By extrapolation of the curves backwards, one estimates that the heat leak to the absorption cell (System B) in the absence of a heater power ($P=0$) lies between 0.2 and 0.5 mw.

By plotting all the data for (dT/dt) we had for $P=5$ mW, using System B, as a function of the average temperature, T , we concluded that the rapid rise of (dT/dt) with decreasing T could be described by the equation:

$$\log_{10} (dT/dt) = 4.36/T + 0.977 \quad (1)$$

where (dT/dt) is in microKelvin/sec.

V. DISCUSSION

It is of interest to consider in connection with our results the variables which affect the low temperature reached, and in particular T_{\min} , in the desorption process. For a given adsorbent material and working gas, the possible variables are:

- (1) The initial temperature, T_i .
- (2) The initial pressure, p_i .
- (3) The heat capacity of the desorption cell and its addenda.
- (4) The residual heat leak into the desorption cell and, if applied, the power dissipated in the cell.
- (5) The throughput and pressure capability of the pump.
- (6) The flow resistance of the pumping line.
- (7) The flow resistance within the pores of the absorbent material.

Considering first item (3), the heat capacity of the desorption cell and its addenda, we note that for System A the mass of copper in the desorption cell was 24.2g. and that for System B 286 g. If we consider, as an example, a cooling from 8K to 4K, the enthalpy difference, ΔH , for the copper between these two temperatures is about 0.03 joules for System A and 0.3 joules for System B. This is to be compared with the heat of desorption in each system. Taking, as previously reported by us¹⁰⁾, the heat of desorption at 2 layer coverage to be about 500 j/mole, the surface area of the synthetic zeolite 13X to be 527 m²/g and a "layer" to be about 0.3 cm³(STP)/m², then the total heat of desorption per layer (at about 2 layers coverage) is about 25 joules for System A and 900 joules for System B. It appears therefore that in the desorption process, the cooling of the desorption cell, its addenda and even the body of the adsorbent material itself, imposes a negligible load on the coolant process at these low temperatures. At higher temperatures, for example liquid nitrogen temperatures, however this favorable situation is no longer valid, as has been substantiated by experiment. For initial temperatures therefore below about 10K to 20K, it is possible to neglect in first approximation the effect of item (3), the heat capacity of the desorption cell and its addenda and contents, on the cooling process.

One may therefor consider the cooling process, for sufficiently low initial temperatures, as described above to be as indicated in Fig.7 which sketches a family of typical equilibrium adsorption isotherms of the mass adsorbed versus vapor pressure. Note that $T_1 > T_2 > T_3 > T_4$. If the initial mass adsorbed is m_i and the initial pressure, p_i , is represented by the point A, then a desorption

would be represented by the path A→B, where at B the mass adsorbed is m_f ($m_f < m_i$) and the pressure p_f , ($p_f < p_i$). For adequately low values of T_i , as discussed above, the difference between m_f and m_i would be small, as sketched in Fig. 7. The final temperature reached (in Fig. 7, this is T_3) therefore is determined by p_f . Each adsorbent will have its own characteristic family of isotherms similar to that sketched in Fig. 7 and hence for a given p_f the value of T_f will be characteristic of the material used.*

Now p_f , in practical situations is determined by dynamical considerations that are largely independent of the adsorbent material but that are determined by the apparatus used. In practice there is always some heat leak into the adsorbent material and there may be an external power applied to it, if some refrigerative load is applied. To offset this heat input power, P , there must be a continuous desorption, in amount \dot{n} moles/sec., P given by $\dot{n}Q=P$, where Q is the heat of desorption at the point (m_f, p_f) . In any given apparatus, the gas flow \dot{n} from adsorbent to pump will determine p_f , the variables (5), (6), & (7) being those which are appropriate in this determination.

For the apparatus used in our experiments, both for System A and System B, we know in detail the values of (4), (5) and (6). The residual heat leak into desorption cell of System A was about $25 \mu W$, whereas as that into the cell of System B was (as

* There is also some contribution to the cooling process due to isentropic expansion of the gas in the void volume of the adsorption cell (Simon expansion¹⁹). At these initial pressures of one atmosphere or less, however, this contribution is small.

stated earlier) from 200 μ W to 500 μ W. Moreover, as reported in Section IV, many input powers were applied to the desorption cell of System B in amounts up to 12.8 mW. The approximate values of the gas flows, \dot{n} , from adsorbent to pump are known for every experimental situation encountered, since the values of the heat of adsorption (desorption) are known for He⁴ on synthetic zeolite from our previous work¹⁰⁾.

Item (5), the characteristics of the pump are known and item (6) the flow resistance of the pumping tubes was estimated in a manner similar to that previously employed by one of us (JGD)²⁰⁾. In the situation here, account was taken of the change from Poiseuille flow to Knudsen flow as one went up the pumping tube towards room temperature. The evaluations of items (4), (5) and (6) at our disposal in this way lead to the following conclusions:

- (a) The primary limitation of p_f and hence on T_f for System A and System B, when used with the Booster Pump, was the flow resistance of the pumping tubes. Larger tubes with lower resistance would result in lower T_f until limitations due to items (5) or (7) were encountered.
- (b) The primary limitation on p_f and hence on T_f for System B using the mechanical pump alone was the pump itself, i.e. item (5).
- (c) No evidence was apparent for any limitation on p_f due to pressure drops within the pores of the adsorbent. This may appear at first somewhat surprising in view of the finding in our earlier work²⁰⁾ that this process was a limiting factor in adsorption

pumping using cocoanut charcoal grains as adsorbent. It is not in fact contradictory however when one considers the much higher pressures that obtain in the present experiments. It should be noted however that this factor may become of significant influence if desorptions to much lower temperatures than those reported here were to be carried out.

Some remarks may be made concerning the observed steady warm-up rates when significant powers were applied to the desorption cell. For a given power, P , the gas flow \dot{n} will remain approximately constant provided there is not too much depletion of the mass adsorbed (A discussion of the effect of depletion is given immediately below). For a steady pumping regime therefore, for a give power P there will be a dynamically equilibrium value of p_f . In first approximation therefore, the state of the system will follow a constant pressure line, as indicated by the line $B \rightarrow C$ in Fig. 7. This line is one of warming and the rate of warming is determined by the value of $(\partial m / \partial T)_p$ in the region $B \rightarrow C$, since we have:

$$P = Q \left| \frac{dm}{dt} \right| = Q \left| \frac{dm}{dT} \right| \cdot \frac{dT}{dt} \quad (2)$$

i.e.
$$P = Q \Sigma \left| \frac{d(m/\Sigma)}{dT} \right| \cdot \frac{dT}{dt} \quad (3)$$

where Σ is the total surface area of the adsorbent.

In confirmation of this relationship(3) the experimental data of Section IV showed that the rate of warming (dT/dt) under various input powers was approximately proportional to P at a given average temperature level, which is in accord with these considerations. They showed moreover that the warm-up rate, (dT/dt) at a given power level increased with decreasing average

temperature level. This would mean that the absolute value of $(d(m/\Sigma)/dT)$ must increase as T increases, since Q is approximately independent of T . An indicator that this is so, at least at somewhat higher pressures than those employed here, is given in Fig. 8, which plots an experimental evaluation of an isobar of a quantity proportional to m/Σ (the volume V in cm^3 (STP) of gas adsorbed per m^2 of surface) versus T for He^4 on synthetic zeolite 13X deduced from observations reported by us earlier¹⁰⁾. This curve shows that $|d(m/\Sigma)/dT|$ increases with increasing T ; however the accuracy of the data is not sufficient to check whether the rate of increase of $|d(m/\Sigma)/dT|$ would yield the experimental function of equation (1) for the variation of (dT/dt) with temperature. It would appear that a more detailed study of the isotherms of He^4 on 13X at low pressures (1 to 100 microns) would be of value in assessing these results in more detail.

Now a comment regarding lengthy periods of refrigeration with the desorption system, as for example was carried out from minute 250 to minute 516 in the desorption run using System B which is shown in fig. 5. It will be seen in the figure that (dT/dt) diminished gradually over this period. This can be attributed to significant depletion of the mass adsorbed. As has been shown earlier by us¹⁰⁾ for He^4 on 13X, the heat of desorption, Q , increases as m is decreased. This increase in Q , which would take place gradually, will result in a diminution of (dT/dt) for constant power P , as is evident from equation (2).

Finally it may be of interest to tabulate some derived data for a desorption system similar in general features to our System B, but modified for and normalized to a charge of synthetic zeolite 13X of 1 kilogram, which data is directly computed from the results given

the previous Section. Table IV shows this data for the elapsed time, Δt , and the total energy absorbed, E , for a kilogram system for various power inputs (1 mW, 5 mW and 10 mW) for temperature excursions between (a) 3K to 4K; (b) 4K to 5K and (c) 5K to 6K. It will be noted, for example, that for a 1 mW refrigerative load a 1 kilogram system would last 130 hours in the temperature range 4K to 5K.

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Table I

Data on content by volume of zeolite 13X pellets having 2% by weight of water. (Frm Mr. F. Harris,¹⁶⁾ Private Communication)

<u>Volume</u>	<u>Percent of Pellet Volume</u>
Intracrystal Void	28.1
Solid Portion of Crystal	29.3
Solid Portion of Binder	9.3
Intercrystal Void	33.4

Table II

DESORPTION EXPERIMENTS DATA TAKEN WITHOUT USE OF BOOSTER PUMP
(FINAL PRESSURE 50 to 70 MICRONS)

T_i (K)	p_i (Atmos)	$T_{f,min.}$ (K)	$r = T_i/T_{f,min}$	τ (min)
6.0	0.95	1.83	3.28	15
8.0*	0.25	4.80	1.67	10
8.0	0.48	3.70	2.16	35
8.0*	0.50	3.64	2.20	10
8.0	0.50	3.72	2.15	35
8.0	0.50	3.75	2.13	40
8.0	0.83	3.31	2.42	20
8.0*	1.00	3.07	2.61	10
8.0	1.06	3.25	2.46	30
10.0*	0.25	7.04	1.42	
10.0*	0.50	6.10	1.64	
10.0	1.00	4.85	2.06	
12.0*	0.25	9.45	1.27	
12.0*	0.50	8.51	1.41	
12.0*	1.00	7.27	1.65	

The data marked with an asterisk were taken using System A

Table III

DESORPTION EXPERIMENTS. DATA TAKEN WITH USE OF BOOSTER PUMP (FINAL PRESSURE 10 to 13 MICRONS)

T_i (K)	P_i (Atmos)	$T_{f,min.}$ (K)	$r=T_i/T_{f,min.}$
8.0	0.50	3.20	2.50
8.0	0.50	3.10	2.58
8.0	1.06	2.72	2.94

Table IV

Warm-up times, Δt , and total energy absorbed, E , by a desorption system with synthetic zeolite (13X) of mass 1 kilogram in various temperature ranges for three refrigerative power loadings of 1 mW, 5 mW and 10 mW.

Temp Power	From 3K to 4K	From 4K to 5K	From 5K to 6K
1 mW	$\Delta t = 64$ hours $E = 230$ j	$\Delta t = 130$ hours $E = 460$ j	$\Delta t = 200$ hours $E = 730$ j
5 mW	$\Delta t = 12.8$ hours $E = 230$ j	$\Delta t = 26$ hours $E = 460$ j	$\Delta t = 40$ hours $E = 730$ j
10 mW	$\Delta t = 6.4$ hours $E = 230$ j	$\Delta t = 13$ hours $E = 460$ j	$\Delta t = 20$ hours $E = 730$ j

Legends for Figures

- Fig. 1. Scale drawing of the second desorption cryostat (System B) in section.
- Fig. 2. Plot of the observed r values ($r = T_i/T_f$) versus initial pressure, P_i , for several values of the initial temperature, T_i . The data were taken using the mechanical pump only.
- Fig. 3. Plot of the observed r values ($r = T_i/T_f$) versus initial pressure, P_i , for an initial temperature, T_i , of 8.0K. The data were taken using the Booster and the mechanical pump.
- Fig. 4. Plot of the observed r values ($r = T_i/T_f$) versus initial temperature, T_i , for two initial pressures $P_i = 0.5$ atmos. approx. and $P_i = 1$ atmos. approx. The data were taken using the mechanical pump only.
- Fig. 5. Plot of temperature versus time for a typical run, with $T_i = 8.0K$ and $P_i = 0.5$ atmos. For detailed commentary, see text.
- Fig. 6. Plot of observed warm-up rates, (dT/dt) in microKelvin per sec. versus heater power, P , in milliwatts. The two curves are for average temperatures $3.5K \pm 0.1K$ and $5.5K \pm 0.3K$.
- Fig. 7. Sketch of a typical family of adsorption isotherms plotting the mass adsorbed, m , versus pressure p . Four typical isotherms are shown, where $T_1 > T_2 > T_3 > T_4$. The path A \rightarrow B \rightarrow C represents a desorption run. For detailed commentary, see text.
- Fig. 8. Plot of quantity of He⁴ adsorbed on synthetic zeolite (13X) measured in, V , cm³ (STP) per m² of adsorbing

surface area, as a function of temperature for
constant pressure from data taken previously by us.¹⁰⁾















