

Design of cryogenic gas adsorption units extends the potential range of ultrahigh vacuum pumping

Cryosorption

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Cryosorption, the most recent innovation in ultrahigh vacuum pumping, makes practical industrial attainment of hard vacuum as low as 10^{-13} Torr. Physical capture and retention of gaseous molecules on a refrigerated adsorption medium distinguish the new technique from traditional cryopumping methods which are based on cryogenic condensation alone. Because molecules are captured by adsorption rather than condensation, the need for cooling the cryogenic panel below the boiling point of gaseous components to be evacuated is no longer required. Thus, hard vacuums are attainable with economic energy, and with the advantage—characteristic of cryogenic pumping—of producing an ultraclean oil-free vacuum. Among the expected applications of the newly attained hard vacuum is utilization in vacuum metallurgy, in high-energy research, and in surface chemistry.

Of particular importance to this extension of cryogenic technology has been the development of low-temperature refrigeration units, which implemented the scale-up of cryopumping from laboratory to an effective industrial ultrahigh vacuum pumping technique for the so-called condensable gases, or specifically atmospheric constituents other than hydrogen, helium, and neon. Recently, effort (2, 5, 7-11, 13) has been directed toward development of cryogenic pumping techniques to evacuate the noncondensable species, with particular interest expressed in the mechanisms of cryotrapping

and cryosorption. Results with the latter approach have been most promising (2, 5, 8).

Theory

Dushman (3) points out that vacuum production may be achieved by utilization of one of two basic mechanisms. Pumps may operate as momentum transferring machines which remove mass from the system and exhaust it to the surroundings, or as units which capture and retain mass within the system with associated decrease in momentum by physical and/or chemical combinations. Examples of the two types of equipment and the ultimate pressure normally attained are presented in Table I.

Since the latter of the two types capture and ultimately become saturated with mass, pump capacity or practical life is an important design parameter.

The differential equation which defines the speed of a vacuum pump is expressed by:

$$V \frac{dP}{dt} = -S(P - P_u) \quad (1)$$

Equation 1 is sufficient to describe the pressure behavior of systems in the rough vacuum regions, or about 10^{-2} Torr, since the primary gas load consists of

TABLE I. TWO TYPES OF VACUUM PUMPS AND THEIR ATTAINABLE VACUUMS

MOMENTUM TRANSFERRING MACHINES P_u (TORR)	
Mechanical rough pumps	10^{-3}
Oil-diffusion pumps	10^{-7}
Oil-diffusion pumps with cryogenic trapping	10^{-11}
Steam ejectors	10^{-2}
TRAPPING DEVICES	
Cryopumps	10^{-11}
Ion gettering	10^{-10}
Cryosorption	10^{-13}

the bulk chamber atmosphere. At lower pressure levels, however, the major gas load assuming no detectable external system leakage results from virtual leaks. These leaks arise from faulty fabrication techniques, such as trapped air volumes in welded joints and/or outgassing of absorbed and adsorbed species from material surfaces. The former gas load can be minimized by proper ultrahigh vacuum construction practices, and the latter rate can be controlled by operational procedures.

The outgassing rate of materials is an exponential function of temperature and increases with increasing temperature. The rate also decays with time at constant temperature (6) such that:

$$Q_o = Q_{oi}e^{-at} \quad (2)$$

As a consequence of this outgassing phenomenon, Equation 1 is modified for the ultrahigh vacuum range such that for chamber materials at a relatively constant temperature level:

$$V \frac{dP}{dt} = Q_o - S(P - P_u) \quad (3)$$

Spinks (14) refers to the "Ohms Law" of an ultrahigh vacuum circuit as being expressed by:

$$P = \frac{Q}{S} \quad (4)$$

Equation 4, though simple in form, is most significant to the vacuum system designer. Specifically, it infers that the attainment of a specific pressure level requires a minimization of system load by proper design and operation procedures and existence of adequate pump-speed capability to handle this load. The definition of a required net pumping speed involves consideration of resistance to flow within the system. Any pipe or duct imposes resistance to gas flow whatever the nature of the flow, be it turbulent, viscous, or molecular. This resistance may be defined as:

$$R = \frac{\Delta P}{Q} \quad (5)$$

In ultrahigh vacuum work, where molecular flow conditions prevail, it is convenient to speak in terms of conductance, or the reciprocal of resistance. In the pressure range where free molecular flow takes place, the probability of intermolecular collisions is small relative to probability of collision with chamber walls. Thus, molecules will behave independently of each other and the conductance of gases can be predicted by the kinetic theory relationship:

$$C_g = \beta K(T/M)^{1/2} \quad (6)$$

$$\text{and } C = 1/R = Q/\Delta P \quad (7)$$

The net pumping speed therefore at any point in a vacuum circuit will be defined by:

$$\frac{1}{S_n} = \frac{1}{S_p} + \frac{1}{C} \quad (8)$$

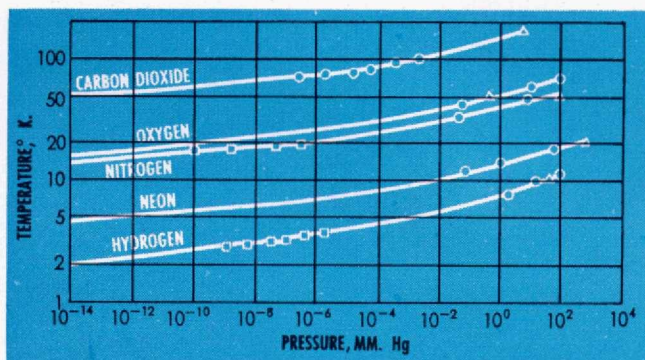


Figure 1. Vapor pressure of gases at low temperatures; circles denote typical published data; squares denote data from Russian Zh. Tekhn. Fiz. 30, 539-595 (1960); triangles show the triple points

Hence the vacuum designer must select a pump and system which will provide the necessary net pumping speed at the work zone to handle the system load and effect the specified working pressure. As already mentioned, in the ultrahigh vacuum region, Q is primarily an outgassing load and its magnitude can be controlled by temperature regulation, for example, by high temperature bakeout and/or cryogenic cooling. Speed requirements, of course, govern selection of pump size and type. In this respect further discussion is required relative to the pressure dependence of the pumping speed.

Equation 1 assumes a pressure-independent pumping speed. In practice, ultrahigh vacuum pumps exhibit a minimum feasible ultimate pressure for any given system, which is an inherent function of the pumping fluid and the gas load associated with the pump components, such as the housing seals. Dushman (4) defines a useful quantity, E , which is a measure of the net exhaust rate of the pump such that:

$$E = S(1 - P_u/P) \quad (9)$$

If P_u is small relative to P , $E \simeq S$, but as P approaches P_u , E will approach zero. In subsequent sections it will be shown that the mechanisms of cryopumping and cryosorption allow attainment of low ultimate pressures so that maximum net pumping speeds are maintained for extended ranges of pressure.

Assuming that free molecular flow conditions prevail, the volumetric pumping speed of any ultrahigh vacuum pump (net exhaust rate) may be derived from kinetic theory considerations of the quantity of gas approaching the pumping port per unit time and the capture probability of the pump. This relation is expressed by:

$$E = \theta S_t A (1 - P_u/P) \quad (10)$$

$$\frac{E}{A} = \theta (3.64 \sqrt{T/M}) (1 - P_u/P) \frac{1}{\text{sec.-sq. cm.}} \quad (11)$$

The physical significance of capture probability, θ , and minimum attainable vacuum pressure, P_u , for the cryogenic pumping mechanisms, cryopumping and cryosorption, becomes evident in the following discussion.

Cryopumping, the capture and retention of mass on a

refrigerated surface, is basically a phase equilibrium phenomenon. Below its triple point each constituent exerts a characteristically low vapor pressure suitable for ultrahigh vacuum service. The temperature corresponding to this pressure level for each gas therefore dictates refrigeration requirements for effective cryopumping and the overall effectiveness of a cryopumping mechanism depends not only on panel temperature but also on gas composition. As a consequence, even small traces of hydrogen and helium could result in relatively high chamber pressures even at very low temperatures, unless auxiliary pumping schemes are available to handle these species. As an example of this situation consider Figure 1, which lists vapor pressure data for common gases found in vacuum chamber atmospheres. It is observed that at 20° K. the vapor pressure of oxygen and nitrogen is about 10^{-11} Torr and this temperature corresponds to the normal boiling point of hydrogen. Even at 4.2° K., the normal boiling point of liquid helium, the vapor pressure of hydrogen is relatively high, 10^{-6} to 10^{-7} Torr. Thus, other techniques must be employed to effect removal of the noncondensables to achieve ultrahigh vacuum levels.

In Equation 11, P_u is effectively replaced by P_c , the vapor pressure of the condensate, for the cryopumping mechanism. In addition, θ is a function of panel geometry and the sticking coefficient, α . This latter parameter is defined as the ratio of molecules which are cryopumped on the first collision with the panel to those approaching the panel. The sticking coefficient is a function of panel and gas temperature, and by proper refrigeration design can approximate unity. The effect of panel geometry requires further discussion.

Ideally, the cryogenic pumping surface should be unobstructed from impinging molecular flow; in other words, conductance limitations should be minimized. Cryopump design usually facilitates this condition when liquid-nitrogen refrigerated surfaces are employed for cryopumping water vapor, carbon dioxide, and other high boiling species. However, lower temperature levels such as the 20° K. surfaces usually utilized for cryopumping oxygen and nitrogen are shielded by liquid-nitrogen cooled, optically tight radiation baffles because refrigeration cost at this temperature level is quite expensive. This combination of baffles and cryopanel is termed an array, and many such configurations have been proposed and utilized. One consequence of this attempt to reduce refrigeration cost is an associated reduction in net pumping speed since the baffles represent a resistance to molecular flow. Efficient array design, therefore, requires an optimization of flow con-

ductance to transmitted radiant heat load. As a consequence of these factors, from consideration of Oatley's (12) treatment of conductance through baffles in series, the parameter θ for cryopumping can be defined by:

$$\frac{1}{\theta} = \left[\frac{1}{D_1} + \frac{1}{D_2} + \dots \frac{1}{D_n} \right] + \frac{1}{\alpha} - n \quad (12)$$

As mentioned above, auxiliary pumping techniques are necessary to supplement cryopumping for ultrahigh vacuum service and diffusion and/or ion pumps have been successfully used for this function. Recent work (2, 5, 8) with cryosorption pumping of hydrogen and helium has demonstrated the practicability of this mechanism as an alternate to diffusion or ion pumping for ultrahigh vacuum service.

Cryosorption pumping, as mentioned earlier, refers to the capture and retention of gaseous molecules on a refrigerated solid adsorbent and differs primarily from cryopumping in that the mechanism of capture is not condensation but rather physical adsorption. The significance of this mechanism is that species may be more effectively cryosorbed with resulting low pressures at temperatures near the normal boiling point of the gas being adsorbed. For example, at 20° K. the vapor pressure of hydrogen is one atmosphere, hence it cannot be effectively cryopumped. However hydrogen can be successfully cryosorbed on selected adsorbents at 20° K. to maintain ultrahigh vacuum levels. In addition, helium can be effectively cryosorbed at temperatures near 4° K. The utilization of physical adsorption at cryogenic temperatures to reduce mass concentration in a closed system has been studied by many investigators for the past 50 years. However, most attention has been directed only at equilibrium aspects of the mechanism, or more specifically, at a cooled adsorbent in communication with an isolated gas volume containing a known mass of the gas to be adsorbed. At some time, t , the system reaches an equilibrium pressure value and these data are used to define a point on an equilibrium adsorption isotherm plot. This concept is applied for rough pumping vacuum chambers with adsorbent beds cooled to liquid nitrogen temperature levels, and rough vacuums of 10^{-2} to 10^{-8} Torr can be obtained in this manner starting from atmospheric pressure. Recent studies (7-3, 8) have been concerned with the phenomenon of dynamic cryosorption pumping, or the utilization of a refrigerated adsorbent to act as a constant speed pump when gas loadings are applied. It has been demonstrated in these investigations that this technique is not only a feasible ultrahigh vacuum tool but also a practical one for a well designed cryogenic pumping system.

These studies have led to the development of cryosorption pumping panels consisting of Linde Molecular Sieve adsorbent bonded directly to metallic substrates. These panels are more attractive than adsorbent beds since thermal performance is thus greatly improved, and panel configuration allows attachment in the chamber enclosure in any position without net pumping speed reduction associated with external transition pieces.

Experimental results with unbaffled panels indicate

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near theoretical pumping speeds for hydrogen and helium when refrigerated at 20° and 5° K., respectively. Hence α for the cryosorption pumping mechanism approximates unity. In addition, the ultimate pressure, P_u , can be reduced to very low levels by proper sizing of the adsorbent mass, since P_u is defined by the equilibrium loading of the panel. For dynamic cryosorption pumping, the effective equilibrium loading will be less than the capacity determined from an isotherm since diffusion through the panel bed is rate limiting (5).

The combination of cryopumping and cryosorption pumping mechanisms has led to the design and fabrication of useful pumping units capable of exhausting chamber volumes from atmospheric pressure to the ultrahigh vacuum range and maintaining these pressures under dynamic gas loads. Design parameters for these pumps as well as a description of available equipment are presented in the following paragraphs.

Cryogenic Pumping Design Parameters

The design parameters associated with liquid-nitrogen refrigerated cryosorption rough pumps have been discussed in detail elsewhere (7) and are thus only briefly summarized in this paper. Since, as previously described, the rough cryosorption pump is basically an equilibrium application of physical adsorption, adequate sizing of the adsorbent bed to the chamber volume based on isotherm data is an important consideration. In addition, however, thermal design ensuring proper adsorbent bed cooling is most critical since capacity is a strong exponential function of adsorbent temperature. Furthermore, since pressures approaching the free molecular range can be approximated with properly designed cryosorption rough pumps, conductance limitations to pumping performance must be considered.

An interesting effect (7) observed with utilization of two or more rough pumps, termed staging, is that pressures of the order of 10^{-5} to 10^{-6} Torr can be obtained with these liquid nitrogen-cooled adsorbent beds when properly sequenced. From consideration of the noncondensable (hydrogen, helium, neon) content of air and the equilibrium adsorption data for liquid nitrogen-cooled molecular sieve, it would appear that the lower pressure of these units was in the low 10^{-3} Torr range. However, by utilization of two (or more) rough pumps such that one unit is used to reduce chamber pressure to a level of about 1 Torr and then isolated and the second is then opened to the system, pressure typically two to three decades lower can be effected. The mechanism of this process is not completely understood but is believed to involve a trapping and isolation of the majority of the noncondensable load during the initial rapid exhaust. Hence efficient design of a rough pumping system utilizing two or more rough pumps involves consideration of staging effects. It is pointed out that the success of staging is enhanced if inherent system gas load is minimized since the pumping speed of these units decreases rapidly at lower pressures, 10^{-5} Torr range.

The combination of cryopumping with cryosorption in a single unit to effect an operable, independent, cryogenic, ultrahigh vacuum pump involves many considerations. Basically, however, the pump design involves integration of a refrigerated cryosorption panel with a conventional cryopumping array such that refrigeration requirements are minimized and pumping speed for all gases including the so-called noncondensables are maximized. The capacity factor of the adsorbent panel dictates to a large extent allowable array configuration. Since the panels are intended to pump only the lower boiling gases and hence prolong effective life, higher boiling species, air, for example, should be intercepted and cryopumped at intermediate stages. The criteria for cryopumping arrays mentioned earlier are also applicable, hence design bases can be summarized such that radiant heat and mass loads should be handled at the highest temperature levels consistent with overall pump performance. A diagram of a cryogenic, ultrahigh vacuum array fulfilling these criteria is illustrated in Figure 2. The function of each of the elements follows.

The liquid nitrogen shield serves to pump high boiling species such as H_2O and CO_2 and shield internal helium-cooled elements from chamber wall radiation loads. The surfaces of the shield are maintained at a low emissivity to minimize nitrogen consumption.

The high-emissivity coated liquid nitrogen chevrons serve the functions listed for the nitrogen shield and serve to absorb the bulk of radiation impinging on the array, thus minimizing the heat load reflected through the chevrons to the helium-cooled surfaces.

The helium reservoir serves as an internal storage Dewar for supply of refrigeration to the low temperature pumping surfaces. Steady-state boil-off of fluid is controlled by careful thermal design and minimization of heat loads approaching the cryosorption panel.

The cryosorption pumping panel consisting of Linde molecular sieve bonded to a metallic substrate is in intimate contact with the liquid helium reservoir. Surface temperature of the adsorbent is maintained quite close to that of the fluid by utilization of the latent heat refrigeration. The primary function of the panel is to serve as a pumping surface for hydrogen, helium, and neon gas loads.

The chevron cryopanel is refrigerated by utilizing sensible heating from the vaporized helium leaving the reservoir. Array design allows for cryopanel temperature to be maintained below 20° K. under steady-state operation. These optically tight chevron baffles have the primary purpose of cryopumping all species except hydrogen, helium, and neon which pass through the liquid nitrogen-cooled chevrons, particularly oxygen and nitrogen.

The gaseous helium shield is cooled by cold helium gas leaving the cryopanel refrigerant tubes, routed through attached passages. It minimizes direct radiation heat loading on the panel and reservoir from liquid

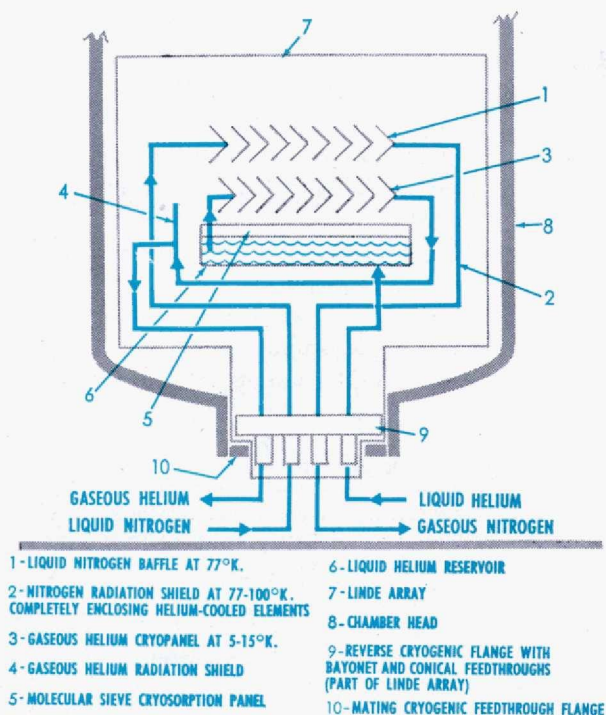


Figure 2. Schematic diagram of cryogenic vacuum array

Figure 3. Linde SN-2 cryosorption pump typifies the units available for rough pumping vacuum systems

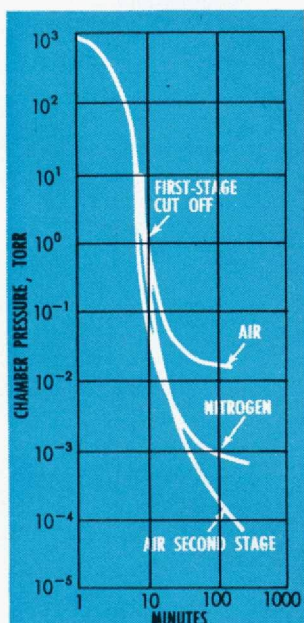
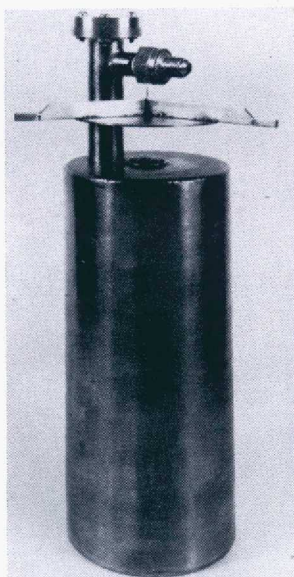


Figure 4. Performance data for one- and two-stage operation of cryosorption pumps show improvement when initial chamber volume is purged with liquid nitrogen

nitrogen-cooled surfaces. This arrangement allows efficient refrigeration recovery of the helium vapor before leaving the array.

Existing units embodying these design elements are described in the following section with associated performance data. It is observed that the suggested mode of refrigeration is a batch-type operation utilizing an integral, internal helium reservoir. This mode of transfer may, of course, be replaced by continuous open or closed cycle refrigeration depending upon the availability of required equipment.

Because the cryogenic array operates on a mass retention principle, periodic regeneration of the adsorbent is required to restore capacity. This operation is achieved by routing preheated gas through the helium reservoir, the design of which should include suitable internal baffles to promote even distribution of heat over the adsorbent surface. This procedure may be effected during system roughing and/or chamber bakeout.

The inherent nature of this mode of vacuum pumping offers distinct advantages with regard to net available pumping speed, ultimate attainable pressure, and system cleanliness. In addition, well designed systems are economically competitive with conventional pumping equipment and offer more pumping speed for extended ranges per dollar invested.

The cleanliness factor has received considerable attention in recent years as applications of low pressures have been extended to space simulation, vacuum metallurgy, studies of the physics and chemistry of surfaces, and thin film deposition. Conventional pumping system design has not neglected this factor but often it has been compromised in favor of higher net speeds and lower ultimate pressures. Cryogenic pumping however, reflects an ultimate in state-of-the-art techniques in this regard since no hydrocarbon fluid or impurity is associated with the pumping elements. Hence no design trade-off need be made relative to net pumping speed and ultimate pressure. Specific performance data for existing units are described in the following section.

Cryogenic Pumping Equipment

Some available cryogenic vacuum pumping units manufactured by Linde Division of Union Carbide Corp. are described in subsequent paragraphs. The SN-2 cryosorption rough pump pictured in Figure 3 is typical of a line of units available for rough pumping chamber systems. This particular pump contains two pounds of Linde Molecular Sieve and when cooled with liquid nitrogen can be used to exhaust chambers as large as 2.5 cu. ft. to the lower pressure levels in the low micron range. Figure 4 illustrates some typical performance data for one- and two-stage operation of these pumps in combination with a 1.1-cu.-ft. chamber. It will be noted that better performance is obtained with single pumps if the initial chamber volume is purged with nitrogen. In general, a useful rule of thumb for approximate sizing of a pump of this type is that each pound of adsorbent can exhaust 1.5 cu. ft. of chamber volume to the micron pres-

sure range. At present Linde has built units containing as much as 150 pounds of adsorbent.

The array designs available as integral ultrahigh vacuum units are divided into two categories: external pumps and internal units. Both of these designs utilize the basic array concepts discussed previously and illustrated in Figure 2.

The attainment of suitable operating temperature levels is achieved by refrigeration with helium, either as a liquid or a cold gas, and with liquid nitrogen. Nitrogen may be added from a pressurized storage Dewar whereas helium refrigeration can be achieved in one of three ways depending on user preference, equipment availability, and test-cycle time:

1. Batch Filling. The array design includes an internal liquid helium reservoir which can be periodically filled. Low steady-state boil-off is then controlled by efficient thermal design much in the same manner as that of a conventional cryogenic storage container.

2. Continuous Addition. Helium can be added continuously from a storage Dewar wherein temperature

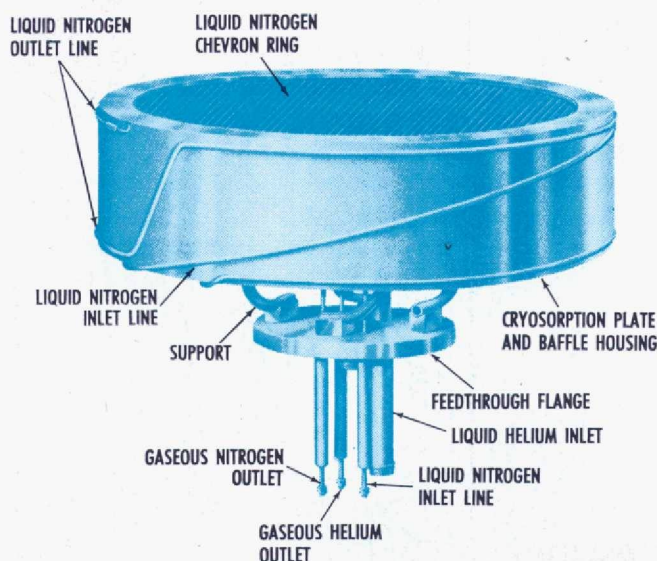


Figure 5. Configuration of Linde S-14 and S-25 cryosorption pumps includes inside flange compatible with inside chamber to facilitate internal use of the array

indication of the cryopumping surface is utilized to adjust flow.

3. Closed Cycle Refrigerators. If available, a small closed cycle refrigerator can be easily integrated with array interface connections to effect an operable system.

As an example, the Linde Model SHe-8 external pump can be manifolded to a rough pump system consisting of two SN-2 units shown in Figure 3. In operation the pump can be externally flange mounted to a chamber. The over-all unit size is decreased by immersing it in an external liquid nitrogen Dewar and cooling internal nitrogen refrigerated elements by conduction. Net pumping speeds of approximately 900 l./sec. can be obtained for hydrogen. Helium pumping speed is about 500 l./sec. Steady-state helium consumption of 1 l./day or less has been obtained with this design.

Internal cryogenic array designs have been developed

for two standard units, Linde Models S-14 and S-25. The configuration of these pumps is illustrated in Figure 5. The design includes an inside flange which can be mated to a chamber penetration so that the full net pumping speed of the array is available inside the chamber adjacent to the work zone. These internal array units possess approximately, 5000 l./sec. sq. ft. and 3000 l./sec. sq. ft. of projected array area for hydrogen and helium, respectively. The S-14 model has 1 sq. ft. of projected area and the S-25 pump has 3 sq. ft. of effective pumping surface. Steady-state helium consumption approximates 2-8 l./day depending upon array size.

NOMENCLATURE

V	= system volume to be evacuated, l.
dP/dt	= rate of change of pressure in a vacuum system, Torr/sec.
S	= pump speed assumed independent of pressure, l./sec.
P_u	= lowest attainable pressure in a vacuum system, Torr
Q_o	= system outgassing rate, Torr-l./sec.
Q_{oi}	= initial outgassing rate, Torr-l./sec.
a	= characteristic constant related to shape of outgassing curve, l./sec.
R	= resistance of a vacuum system or any component thereof to gas flow, sec./l.
C	= conductance of a vacuum system or any component thereof to gas flow, l./sec.
P	= pressure in a vacuum system at any point, Torr
Q	= vacuum system throughput or gas load assumed to be constant when the system is in equilibrium when molecular current is constant, Torr-l./sec.
S_n	= net pumping speed at any point in a vacuum system, l./sec.
Δp	= pressure drop across a vacuum system or part thereof, Torr
C_g	= conductance of a given geometric configuration for a particular gas of molecular weight M , and temperature T , l./sec.
K	= conductance characteristic of a given geometric configuration, dimensionless
β	= kinetic theory constant
T	= absolute temperature, ° K.
S_p	= known pumping speed at a reference point, l./sec.
θ	= capture probability of a pump, dimensionless
S_t	= theoretical pumping speed, l./sec.
A	= projected area of pumping surface, sq. cm.
E	= measure of net exhaust rate of pump, l./sec.
P_u	= ultimate pressure of pump, Torr
D_i	= mass transmission coefficient for the i th baffle of a cryosorption pumping system, dimensionless
α	= sticking coefficient (ratio of molecules captured on the first collision to total molecules striking cryosorption panel), dimensionless
n	= number of baffles

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