PUMPING SPEED OF GETTER-ION PUMPS AT LOW PRESSURES

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Abstract

Pumping speeds of commercial diode and triode type getter-ion pumps were measured for the gases H_2, He, and N_2 over the pressure range from about 10^{-11} Torr to 10^{-6} Torr. At 10^{-10} Torr, the pumping speed was 15-25% of the speed at 10^{-6} Torr where the pumps attained the rated speed. Pumping speed measurements as a function of time showed that the pumping speed decreased generally only slightly over a period of one day. Strong saturation was found only for He at pressures above 10^{-7} Torr. No significant difference between diode and triode in the pumping speed vs. time or vs. pressure curves was observed.

In the diode, pump currents were measured. The quantity $P \cdot S/I$ (P = pressure, S = pumping speed, and I = current) was found to be pressure independent for short pumping times. The numbers (in molecules or atoms pumped per charge) are: 0.5 (H_2), 0.6 (He), and 0.2 (N_2).

1. Introduction

Getter-ion pumps have been used for about ten years to obtain low pressures. It is well known that the pumping speed of these pumps is fairly constant over the pressure range from 10^{-4} to about 10^{-8} Torr. For small pumps, no data have been published on their pumping speed below 10^{-8} Torr. Rutherford[1] measured discharge intensities I/P (I = pump current, P = pressure) down to 10^{-11} Torr. From the assumption that the number of molecules or atoms pumped per electric charge (PS/I) is a basic property of the pump and independent of pressure, he calculates the pumping speed, S, from the discharge intensity I/P. Klopfer[2] and Davis[3] estimated S from the pressure rise after turning off the pump at very low pressures.

Saturation in getter-ion pumps has been observed by several authors[4,5]. We know of no work in which pumping speeds at constant pressure have been measured as a function of time. Under nonequilibrium conditions, e.g. pumpdown of a system, it is difficult to separate the influence of the rest of the system from that of the pump.

The first goal of this work was to measure S and I/P for a small getter-ion pump down to very low pressures for different gases. The second goal was to study saturation effects by measuring the pumping speed as a function of time at constant pressures for a diode and a triode type pump.

2. Method and Apparatus

The pumping speed was measured with the two-gauge method[6] (pressure drop along a known conductance). Figure 1 shows the apparatus

schematically. The formulas are:

$$S_M = G_1 \cdot \frac{P_1 - P_2}{P_2 - P_o} \cong G_1 \cdot \left(\frac{P_1}{P_2} - 1 \right) \quad \text{if } P_2 \gg P_o \tag{1}$$

$$\frac{1}{S} = \frac{1}{S_M} - \frac{1}{G_2} \tag{2}$$

S_M = measured pumping speed at Gauge Two (P_2)

S = pumping speed of getter-ion pump taking into consideration conductance G_2

G_1 = conductance between Gauge One and Gauge Two

G_2 = conductance between Gauge Two and pump

P_1, P_2 = respectively, pressures in Gauge One and Gauge Two

P_o = ultimate pressure of pump as measured by Gauge Two.

The pumps used in this experiment were commercial diode and triode getter-ion pumps. The diode, which was rated at 15 ℓ/sec for nitrogen, was operated at 7.2 kV in a magnetic field of 1400 Gauss. The triode was rated at 8 ℓ/sec. The voltage for the triode was 5 kV, the magnetic field 1350 Gauss.

The vacuum system (Fig. 1) was made from Pyrex glass (Corning 7740) and had a volume of about 2 liters. A 2-stage fractionating oil-diffusion pump (CVC GF-20), filled with Monsanto OS-124 oil, pumped the system to very low pressures before the getter-ion pump was started. An optically dense zeolite trap filled with Linde 13 X molecular sieve at liquid nitrogen temperature prevented backstreaming oil from getting

into the system. A one-half inch Granville-Phillips valve separated the trap and diffusion pump from the rest of the system during the pumping speed measurements. This valve was also necessary for proper system processing (see below). With this valve closed, it was possible to open the rest of the system up to air with the diffusion pump running. Total pressure measurements were made with Bayard-Alpert gauges (WL-5966) and, in some cases, with Schuemann photocurrent suppressor gauges[7]. Partial pressure measurements were made with a 90^{o} magnetic deflection type mass spectrometer[8]. Gas was admitted from one liter Linde flasks through a one-half inch Granville-Phillips valve and a leak valve. The latter was operated by a Granville-Phillips automatic pressure controller which kept the pressure constant within a few percent.

System processing followed the procedure described by Singleton and Lange[9]. After any glassblowing, the part of the system which was exposed to air was first roughed with a forepump. When the system was at $\sim 10^{-3}$ Torr, the pump was sealed off and the valve to the diffusion pump was opened for a few hours. Then the valve was closed again and the trap baked for about four hours at 350^{o}C. The valve and the glass tubing between valve and trap were kept at 150^{o}C. After bakeout, the trap was immersed in liquid nitrogen, the valve opened, and the system baked for half a day at 350^{o}C. The gauges were then outgassed at 50 watts grid bombardment power for about six hours. Again the valve was closed, the trap baked as before and then cooled again. The pressure dropped to the low 10^{-11} Torr range within an hour after opening the valve.

The getter-ion pump was finally started and the valve to the diffusion pump closed.

Pumping speeds at constant pressure were recorded over a period of one day. After every measurement, the system was processed. The whole pressure range was investigated by changing the pressure in steps of a factor of three or four.

In general, no attempt was made to regenerate the pump before a pumping speed measurement except to bake it. When pumping helium for one day at pressures of 10^{-7} Torr or higher, however, strong saturation was found. After the pumping speed measurement with helium, the pump was bombarded with nitrogen for one hour at a pressure such that 50 watts power was dissipated. During this bombardment the valve to the diffusion pump was kept open. After this discharge cleaning followed by bakeout, the re-emission of helium was seen to be very low even during pumping of another gas. The cleanup process and the behavior of the pump afterward was followed with the mass spectrometer.

Current measurements for the diode pump were made on the ground return side. A shielded box with batteries provided 7.2 kV. The cable to the pump was double-shielded. Under low humidity conditions, the leakage current with the pump electrically connected was below 1×10^{-12} A. For currents larger than a few microamperes, a regular pump power supply was used.

Field emission currents in the triode pump reached values of a few hundred microamperes. It was impossible to subtract these currents because they changes rapidly. The diode showed field emission up to a

few microamperes, but only when pumping nitrogen, and even in this case only occasionally. A straight line on a Fowler-Nordheim plot was considered a clear indication of field emission. In the diode pump the whiskers responsible for field emission could be flattened by application of an overvoltage of 20 to 25 kV. To get still lower field emission currents, nitrogen was admitted at 10^{-5} Torr with the overvoltage applied. Short starting times in the presence of field emission were observed at low pressures due to the abundance of electrons.

The two-gauge method requires only pressure ratios to be measured. No absolute pressure calibration was therefore made. Gauge Two was our standard, and the sensitivity of Gauge One compared to Gauge Two was determined with helium over a wide pressure range. The difference in sensitivity never exceeded 10% and was taken into account for the pumping speed calculations. The conductance to the gauges was increased to about 15 ℓ/sec for nitrogen by attaching a one inch tubulation to them. Ion currents in the gauges were measured with Keithley micromicroammeters which were accurate within a few percent as compared with a constant current source. The difficulties of measuring hydrogen pressures with hot filament ionization gauges are enumerated in several papers by Hickmott[10]. An enhanced pumping rate for hydrogen was observed in the presence of a hot ($T > 1100^{o}K$) filament due to dissociation of hydrogen. The pumping speed of glass or metal walls for atomic hydrogen is very large. To avoid dissociation, low temperature filaments have to be used in gauges. Unfortunately, the ion gauges used for these measurements had only regular tungsten filaments. The emission current in the gauges was held at 1 mA

for the hydrogen measurements compared to 10 mA for nitrogen and helium. Pumping speed measurements for the clean system with the pump current off showed values of about one ℓ/sec for hydrogen and .15 ℓ/sec for nitrogen for times up to one day and at different pressures. These values were subtracted from the results obtained with the pump current on. The system was allowed to reach an equilibrium before measurements were made.

The conductance G_1 was .5 ℓ/sec, and G_2 about 15 ℓ/sec for nitrogen. The error in determining these values is less than 10% for G_1, less than 20% for G_2. As can be seen from formula (2), errors in G_2 have an influence on S if S_M is of the same order of magnitude as G_2. This is the case only for nitrogen. The overall error in the determination of S is estimated to be less than 30%.

3. Results

The values for pressure, P, and pumping speed, S, in this paragraph always mean the values inside the pump.* Pressures are the actual pressures for the different gases (not nitrogen equivalent).

In Fig. 2, I vs. P curves for the diode pump are given for the gases hydrogen, helium, and nitrogen. The slope is practically the same for the different gases at the same pump current. Above $\simeq 10^{-7}$ Torr for nitrogen, $I \propto P$. Between 10^{-9} and 10^{-8} Torr, there is a transition region. No bistable operation was found, i.e., the pump current at a

*Pumping speeds are generally given as speeds at the pump flange. In this paper, however, P and S are correlated to I. This should be done at the same point, i.e., inside the pump.

given pressure was always the same whether one was increasing or decreasing the pressure. Below the transition region, $I \propto P^{1.2}$. The pump was found to go out regularly when the pump current dropped below 2×10^{-11} A. This current corresponds to a nitrogen pressure of $\simeq 1 \times 10^{-12}$ Torr. To reach this condition, the valve to the diffusion pump had to be opened and the gauges shut off.

For short pumping times, the number of molecules or atoms pumped per electric charge was found to be independent of pressure within the accuracy of our measurements. The values for hydrogen, helium, and nitrogen are, respectively, 0.5, 0.6, and 0.2. Over a period of one day, these values decreased significantly at pressures above 10^{-8} Torr for all gases investigated, the decrease being especially high for helium. One exception is the case of hydrogen in the triode pump above 10^{-7} Torr. Here, the pumping speed increased with time. The same effect has been observed in the diode pump at pressures of $\sim 10^{-5}$ Torr after pumping for two days[11]. It is believed that this results from a cracking of the titanium cathode after prolonged hydrogen pumping which increases the permeability of the metal for the gas.

Fig. 3 shows pumping speed, S, and discharge intensity, I/P, plotted versus pressure in the diode pump for different gases and pumping times T = 0 and T = 1 day. The dependence of I/P on pressure for nitrogen is practically the same as reported by Rutherford[1] for the same magnetic field and geometry. Our absolute values of I/P for T = 0 are larger by a factor of 2.7 due to the larger voltage (7.2 kV instead of 3 kV). Fig. 4 shows the pumping speeds for the triode for similar conditions.

4. Discussion

In getter-ion pumps, the discharge intensity, I/P, is proportional to pumping speed, S, for different gases and pressures below 10^{-6} Torr for short pumping times. In other words, the number of molecules or atoms pumped per electric charge is pressure independent.

Saturation occurs after pumping times of the order of a day at pressures above 10^{-7} Torr. This saturation is serious only in the case of noble gases. All other gases are pumped for a very long time with a pumping speed which is a large fraction of the initial speed. In the case of hydrogen in the triode, the pumping speed increased with time for many days at pressures above 10^{-7} Torr.

No significant difference in pumping speed or saturation time was found between diode and triode.

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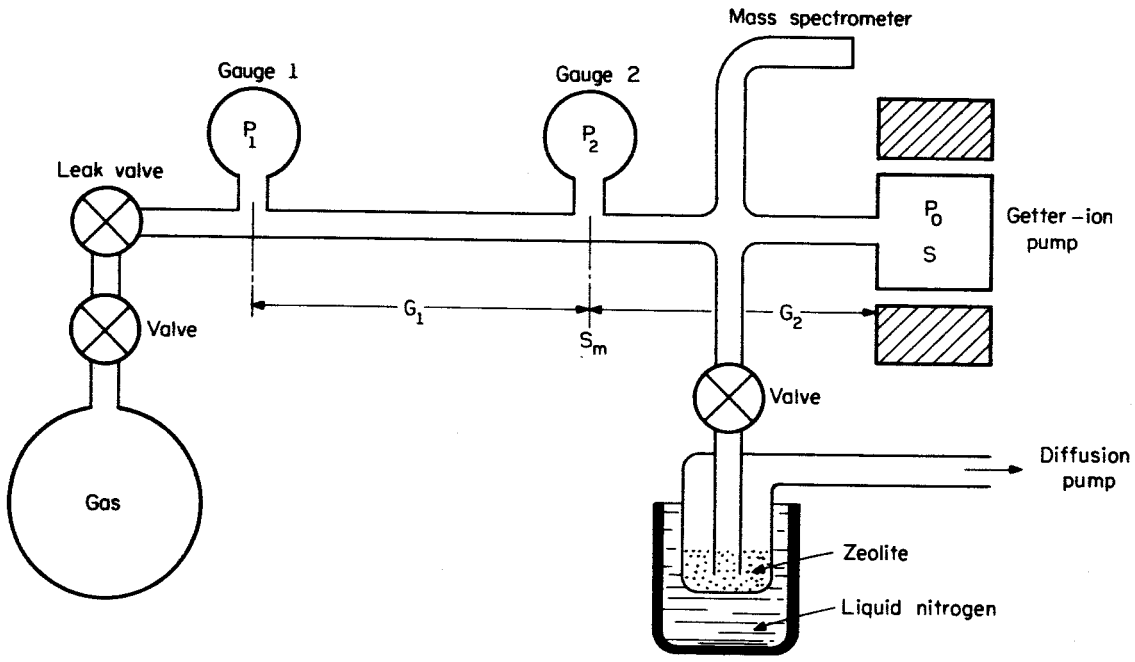

Fig. 1. A schematic representation of the vacuum system used for pumping speed measurements.

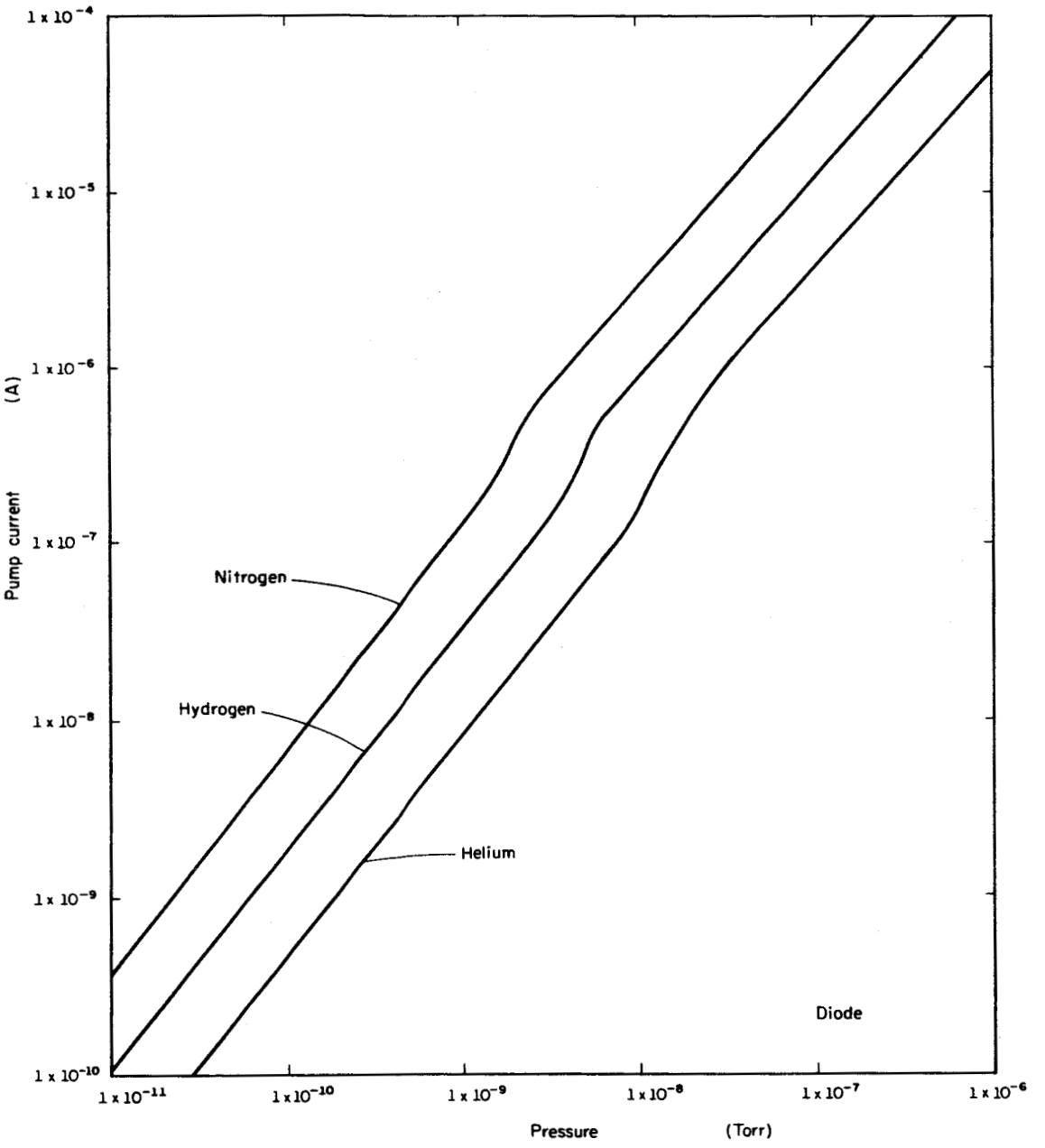

Fig. 2. I vs. P in the diode pump for the gases hydrogen, helium, and nitrogen.

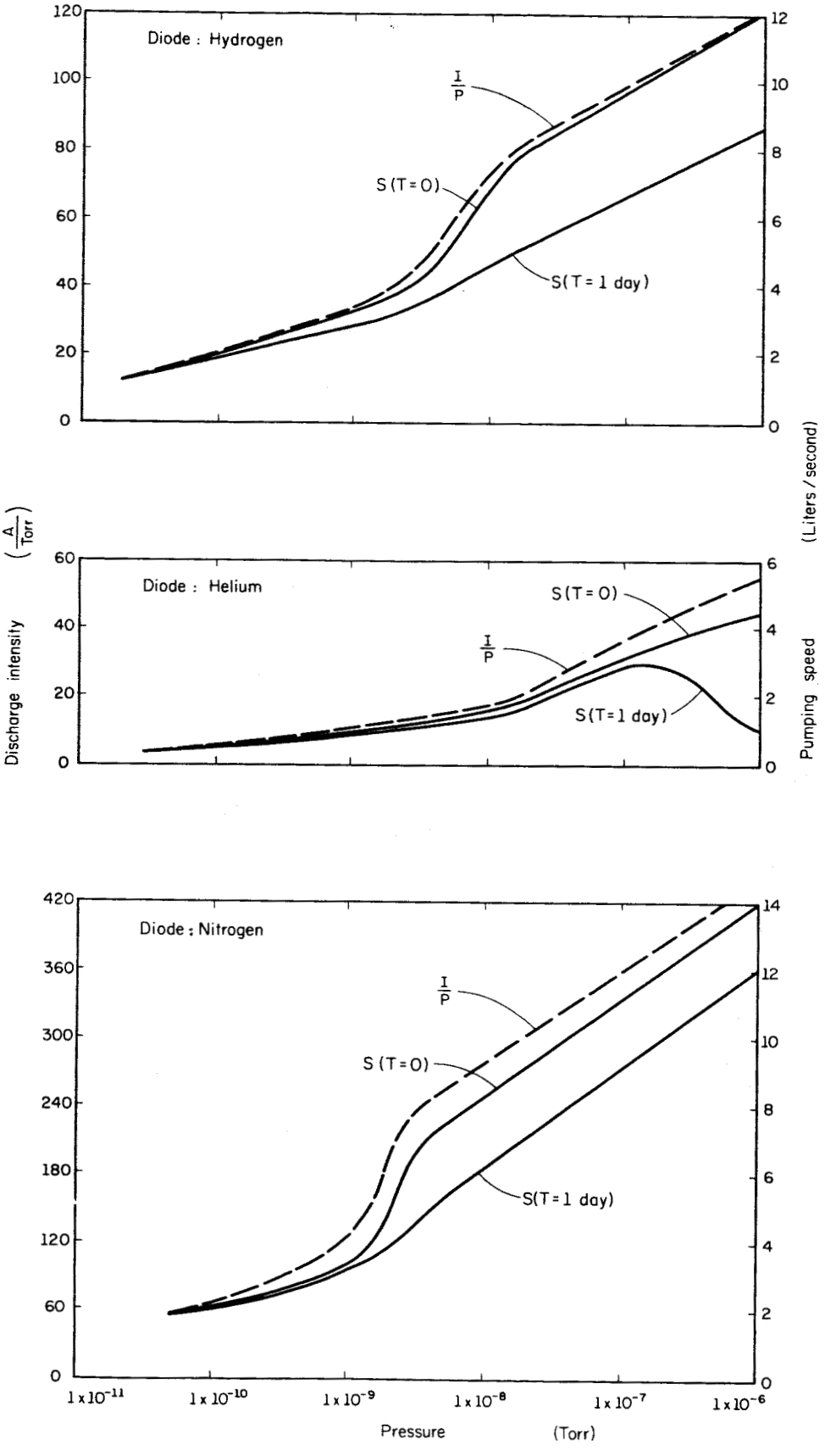

Fig. 3. Pumping speed S and discharge intensity I/P vs. pressure in the diode pump for a) hydrogen, b) helium, and c) nitrogen.

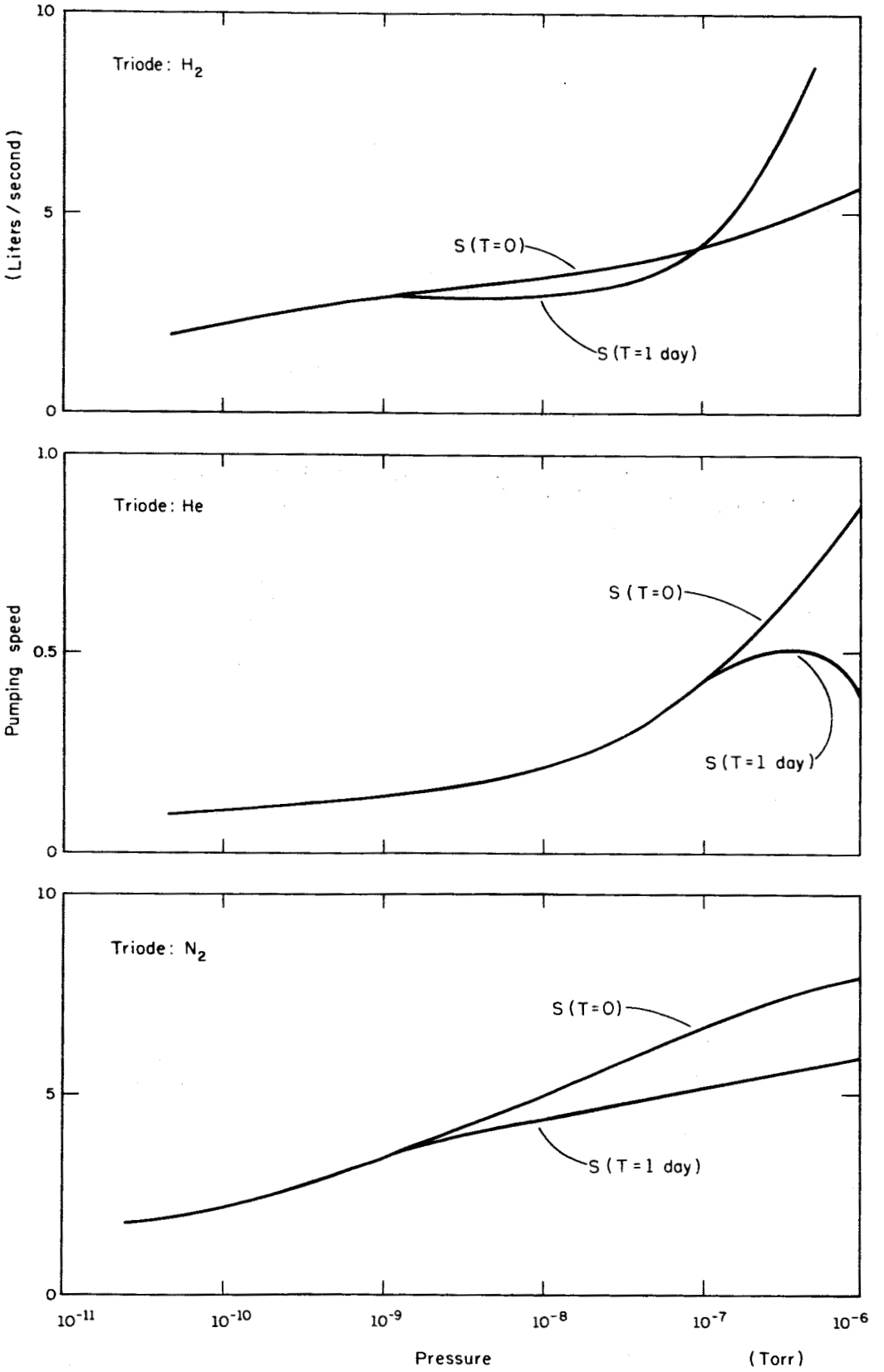

Fig. 4. Pumping speed S vs. pressure in the triode pump for a) hydrogen, b) helium, and c) nitrogen.

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PUMPING SPEED OF GETTER-ION PUMPS AT LOW PRESSURES

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13. ABSTRACT

Pumping speeds of commercial diode and triode type getter-ion pumps were measured for the gases H_2, He, and N_2 over the pressure range from about 10^{-11} Torr to 10^{-6} Torr. At 10^{-10} Torr, the pumping speed was 15-25% of the speed at 10^{-6} Torr where the pumps attained the rated speed. Pumping speed measurements as a function of time showed that the pumping speed decreased generally only slightly over a period of one day. Strong saturation was found only for He at pressures above 10^{-7} Torr. No significant difference between diode and triode in the pumping speed vs. time or vs. pressure curves was observed.

In the diode, pump currents were measured. The quantity $P \cdot S/I$ (P = pressure, S = pumping speed, and I = current) was found to be pressure-independent for short pumping times. The numbers (in molecules or atoms pumped per charge) are: 0.5 (H_2), 0.6 (He), and 0.2 (N_2).

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Security Classification