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Author
Milleron, Norman.

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Norman Miles

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Norman Milleron

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EVALUATING ULTRA VACUUM PUMPING AND OUTGASSING*
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Norman Milleron

My central topic, how to achieve an ultra vacuum environment quickly and to know when you have one is approached along lines perviously taken.\textsuperscript{1,2,3} Briefly these notions are: the central dogma, "Vacuum is an antifluid that flows and can be pumped," can be profitably replaced by an old heresy, "Vacuum is an environment in which to work". Why philosophize like this? This different emphasis is offered to further practical ends by focusing attention on the shortcomings of conventional attitudes and to offer more fruitful paths as follows.

In our everyday habits of speech we talk about vacuum in terms of residual air at such and such a pressure, tacitly assuming that vacuum is like a fluid (antifluid) that flows and can be pumped, and that quoting base pressure before we turn on our process or experiment yields a reasonable prediction that the vacuum will be adequate later on. These speech habits may serve well enough if their implications are not taken too literally. For example, it is more fruitful to make at least the mental reservation that vacuum is an environmental quality, specified by a set of parameters arbitrarily chosen to suit the needs of a particular application. Parameters often of importance are: sorption and desorption energies and probabilities, molecular and atomic concentrations, in the gas phase and harbored by materials - all as a function of chemical type, atomic number, temperature and bombardment. Because the pumping system performance is a vital part of the vacuum specification, figures of merit can be established by predicting the gas flux to, out through, and back from a pumping system. The degree of vacuum then, required for any purpose such as space simulation, plasma processes and experiments,
thin film production, etc. is divorced from a gas pressure specification simply because gas pressure, (defined as the time rate of transfer of momentum in a given direction across unit area normal to that direction) is rarely of any consequence when operating in the free molecular regime.

Having a molecular counter and a method of identifying what is counted, allows determination of the above mentioned three gas fluxes to, through and back from a pump. As yet we have had time to apply this instrument to a roughing system and LN trap with combination valve only (see below). A brief description of what can be achieved with this instrument follows. Discussing the strengths and weaknesses of ion counting and the details of the electric circuits and mechanics involved is far beyond the scope of this paper. In a nutshell, this arrangement for counting ions gives a digital readout, and using multiple scans can provide a signal-to-noise ratio of $> 10^6$, can yield $< 1$ noise count/min, can record the total number of ions collected at each m/e position as a function of time, permits observing adjacent m/e peaks that differ in total counts by $10^6$, and permits scanning mass spectra at 50 a.m.u./0.12 sec. (Note that signals may be followed far below the ambient level.) Based upon a year's experience ironing out troubles, the major drawback so far is that the signal-to-noise ratio from the multiplier decreases with time.

A brief sketch of essentials follows: Gas-phase molecules are ionized by electron bombardment, and the resulting ions are filtered by the quadrupole electric lens into groups according to their mass-to-charge ratio. If the numbers of ions per group is kept sufficiently small, each ion produces a discrete pulse when the ion hits the first stage of the 20-stage electron multiplier. The maximum rate at which single ions can be observed is probably $\leq 10^8$/sec and is determined by the integrating effect of the capacitance of
the anode lead from the electron multiplier. Most noise pulses are smaller in amplitude than ion pulses, and a discriminator lets only pulses above a fixed threshold come through. Ultimately, the pulses are stored in the memory of a 400 channel pulse height analyzer operating in the multiscaling mode or in a computer. The counts stored in this memory are displayed on an oscilloscope screen as a dot pattern corresponding to the mass spectrum. Contents of this memory can be printed or punched on paper tape or recorded on magnet tape. Limitations on accuracy, scan rate and sensitivity may be understood as follows: The pulse height analyzer used accepts a maximum of $3 \times 10^5$ counts/sec and has 400 channels. If 57 mass peaks are to be recorded, $400 / 57$ channels are then available/mass peak. Since statistical accuracy varies as $1 / \sqrt{N}$, where: $N$ is the number of counts/peak, 200 counts/peak is about the minimum acceptable. Thus a single scan of 57 peaks was done in a minimum 0.12 sec. (Note: A factor of ~300 shorter scan time using commercial equipment appears possible.

Figure 2 is a schematic cross section of the essential features of the outgassing and backstreaming testor. The ion counting quadrupole is within side arm (1). Samples of vacuum plumbing (3) are welded on and cut off routinely with a special can opener and welding tool. The system is let up to atmospheric pressure by sucking in liquid nitrogen through a bronze filter (not shown) to keep out ice crystals. Roughing is done by oil-sealed mechanical pump operating through a 'U' trap (6) with heavy copper walls whose temperature is insensitive to change in LN level. This stainless steel roughing line has all metal valves between the system and trap and is always kept at 200°C.

The first tests made examined the roughing system and D.P. system as sources of contamination. The measuring dome (Fig. 2) has an internal tubular
ring (not shown) that may be heated or cooled and an all stainless steel
poppet valve (4) carrying two small orifice holes. Thus by shutting this
poppet valve, either one or the other, or both or none, of the holes can be
open. Three known flow rates can be thus obtained in addition to isolation
between the diffusion pump being tested and diagnostic gear. By heating and
then cooling the tubular ring to 520°K and < 80°K, thus providing an accumu-
lator for a known time, and opening and closing the poppet valve, contamination
already in the dome can be distinguished from contamination steadily enter-
ing the dome. Approximately $10^8$ molecules/sec of m/e > 44 came from the roughing
system (This amount from roughing was << than the amount already in the sample
tube initially.) under free molecular flow. No input flux of m/e > 44 could
be detected coming from the D.P. at an average sensitivity of $10^5$ molecules/sec.

An in situ calibration procedure permitting a quick check of output
signal linearity, sensitivity, resolution, signal-to-noise ratio and ion
counts in terms of numbers of molecules is followed. The central idea is to
compare apparent pumping speeds in steady state to absolute pumping speeds
during free gas expansion. Two small orifices are used, each in turn to
obtain three known speeds. Leak-up rates proportional to outgassing into the
dome are determined by closing valve (5, Fig. 2) and recording ion counts as
a function of time. Some of the peaks thus obtained are composites that can be
identified by searching a library of known spectra with a regression \(^4\) computer
code. Next, apparent pumping speeds in steady state are taken using the
leak-up rates. True pumping speeds are then obtained by closing (5, Fig. 2)
accumulating gas and then opening (5) quickly to achieve a free-gass expansion
through the orifices. By keeping the walls of the system at > 200°C (except
for 150°C around the electron multiplier) and because the geometry is
appropriate, well known simple formulas can be applied. In future, more exact expressions will be tried (for example Hobson and Earnshaw's⁵). Dividing the apparent speeds by the corresponding true speeds gives the calibration constants.

Six commercial pumps have been given at least preliminary tests. Five of these pumps, three sputter ion type, one orbitron type and one LN-trapper D.P., were operated on reasonably identical 4-in diam x 17-in long stainless steel pipes (see discussion of plumbing) capped by dual tungsten dilament nude ionization gauges made by Varian. Cross checks on the plumbing were made showing that the variations in pump down performance were due to outgassing phenomena originating within the pumps themselves. The sixth pump was turbo-molecular type tested on a calibration dome (Fig. 2). The performance of a homemade cryopump, variable between 1.5⁰ and 5⁰K, measured on a separate system.

Welch Turbo-molecular Pump. A single peak of m/3 ~ 60 characteristic of the turbine oil lubricating the turbo-molecular bearings was monitored by a Veeco mass spectrometer. Using the measuring dome (Fig. 2) results show that, with the pump case at room temperature, > 10¹⁰ turbine oil molecules issue from the mouth of the turbo-molecular pump per second. With the pump body at 90⁰C this number increased to > 10¹¹ per second. Apparently the turbine oil can diffuse along surfaces by-passing the rotor. A short pulse of air was admitted to the foreline of the turbo-molecular pump, raising the foreline pressure to slightly more than 200µ. Air as well as bearing lubricant passed into the measuring dome requiring approximately a thousand seconds after the pulse for the dome to recover to the former steady state conditions. This bearing effluent could be eliminated by putting a well-designed liquid nitrogen trap
over the turbo-molecular pump however, one might then consider a liquid nitrogen-trapped oil diffusion pump for reasons of cost and increased speed for light gases (see below).

**LN-trapped D.P.** A straight-through, nominal 6 in trap and valve with liquid fill and vent lines on the bottom, made by Davis and Wilder, Inc., was mounted on an N.R.C. oil diffusion pump. Liquid nitrogen was supplied automatically from a 160 liter dewar by a controller made by Harvey Control Company. In these tests the same measuring dome (Fig. 2) was used, again having the two orifices and accumulating ring. For these tests the quadrupole mass filter made by Electronics Associates, Inc. and modified by us. Using the sensitivity of this instrument, together with the same accumulation and flash-off method, (Fig. 2) no peaks of m/e > 44 could be detected after baking the trap neck and dome at 300°C for 24 hours. This 300°C bakeout was necessary to eliminate the contamination from two years of testing wherein quantities of hydro and fluorocarbon had been admitted to the dome. Also the dome was not (and cannot be) cleaned in the Diversey DS9 process (see discussion below). The point may be raised here about how to keep oil out of plumbing if an accident should occur (see headings, Porous Trap and Valve and Discussion of Pump Test Results, below). In principle, < 10^5 molecules/s average was the upper limit of sensitivity due to background noise. Pulsing the foreline pressure of the diffusion pump well above 200μ also yielded no detectable contamination of m/e > 44. With the dome at 150°C, the same nude ionization gauge read 1.4 x 10^{-10} torr. The ionization gauge and mass filter were calibrated at will by means of the interposable orifices (Fig. 2) either by utilizing residual background gas or leaks of He or N₂. At 150°C, the m/e 18-peak height was the only peak not proportional to the orifice area.
R.C.A. Sputter-Ion Pump. Figure 1 gives some results obtained from pumping down our standard 4-in diam by 17-in long stainless steel assemblies (see discussion below). Curve number 1 shows the response of the BAG as a function of time for the RCA 500 l/s pump shown in Fig. 1. The pump was first roughed down to $< 5 \times 10^{-6}$ torr through the LN-trapped D.P. system shown in Fig. 2, turned on and then pinched off. Upon turning on, the pump immediately outgassed so heavily that its maximum discharge current was exceeded and 15 min were required for the pump to recover its voltage. Referring to Fig. 2, one can see that RCA pump is very much larger than the standard stainless steel tube assembly. Not only does the pump have a much larger internal surface area than the other pumps loaned by other manufacturers, numerous fingerprints were visible inside the pump body. Thus the rated speed of the pump (available at higher pressures) became operationally zero at $4 \times 10^{-8}$ torr.

Ultek Sputter-Ion Pump. Figure 1, curve number 2 represents performance by a 100 l/s Ultek differential ion pump shown in Fig. 1. After three weeks the BAG response became constant at $1.5 \times 10^{-9}$ torr.

Varian Sputter-Ion Pump. Figure 1, curve number 3 renders the performance of a Varian 140 l/s diode pump shown in Fig. 1. This pump evolved much less gas upon starting than the other sputter ion pumps. After one month of continuous operation the BAG response became constant at $2.0 \times 10^{-10}$ torr while the ion current in the pump became 0.06 μA.

N.R.C. Orbitron-Type Pump. Figure 1, curve number 5 gives the performance of the 400 l/s Orb-ion pump made by the National Research Corp. shown in Fig. 1. It should be made clear that curve number 5 was obtained under different procedures and conditions than curves 2 and 3 (discussion below). After operation for one week, the Orb-ion pumpdown curve became constant at a reading of $2.5 \times 10^{-10}$ torr on the BAG. Both pump filaments
burned out after two months at 0.04 A and 8000V. The filament life may be governed by interaction with Titanium. Although the Pump started very quickly at $10^{-6}$ torr, a later test showed the pump very hard to start at $1 \times 10^{-3}$ torr. Probably due to the geometry of the test, $< 10^{-12}$ A was drawn between BAG and Orb-ion pump.

Discussion of Pumpdown Results

In Fig. 1, curves 1 and 5 only, the vacuum furnace treatment of the stainless assemblies had to be omitted for reasons beyond our control. The Orbitron-type pump and the R.C.A. pump were thus operated on stainless steel assemblies that had been Diversey cleaned and then welded only. Because of this unplanned variation in the procedure, water cooling was not applied to the pump body of the Orb-ion until the pump wall temperature had run at 100°C for an hour. The pump barrel water cooling was then turned on, the BAG response recorded, and the copper pumpout pinched off. Each of the pieces of stainless pipe used in obtaining curves 1-6, Fig. 1, had been welded after careful Diversey cleaning; but only tubes yielding curves 3 and 4 were then fired by electron bombardment from an axial filament to a temperature of $800^\circ <$ in a vacuum furnace for 30 minutes. Each time the furnace was let up to dry air.

Considering the above pump tests one can ask, "Were the variations in BAG response due to pumps themselves or to the stainless steel tubing assembly?" After being run on the Ultek pump, the 17-in standard assembly used was hooked up to the measuring dome shown in Fig. 2. The measured speed applied to the end of the 17-in pipe was 50 l/s. Curve number 4, Fig. 1, gives the pumpdown history. This information, together with the BAG response when the sputter ion power supplies were turned on and off, shows that variation in pumpdown performance was due to gas load coming from the pumps themselves. This conject-
ure was fortified by curve number 5, Fig. 1. Referring to Fig. 1 again, curve 6 shows the repumping of the same stainless steel tube assembly by the same 50 liter diffusion pump system that generated curve 4. In curve 4, the stainless steel assembly had been covered by aluminum foil but exposed to air during rainy weather for a month. After pumping for several days a BAG reading of $5 \times 10^{-10}$ torr had been obtained. At this point an all metal valve was closed to the D.P. system and the stainless steel tube assembly was let up to dry helium gas having a principle impurity one part in ten thousand of nitrogen gas. Repumping the tube then gave curve 6. Unfortunately, the welded bellows in the valve used to separate the test assembly from D.P. system probably was major source of outgassing.
REFERENCES

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FIGURE CAPTIONS

Fig. 1 Plots of pumpdown performance for pumps operating on 4-in diam x 17-in long s.s. tubing.

Fig. 2 Outgassing and Backstreaming Testor.
   1. Quadrupole gas analyzer
   2. Ion gauge
   3. Outgassing sample
   4. Measuring orifice
   5. Fast poppet valve
   6. Liquid nitrogen trap
   7. Water cooled baffle
   8. Oil diffusion pump
   9. Liquid nitrogen trap roughing line
Fig. 1
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