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High-Vacuum Pumps in Mass Spectrometers

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The last regular installment of this column covered basic vacuum concepts relevant to mass spectrometry. We reviewed basic gas parameters, such as particle density and mean free path, and the details of the curve that results from the application of Paschen's law to describe the maintenance of electric potentials under vacuum. We divided the realms of vacuum pressures into various ranges and described how the proper operation of a mass spectrometer required the correct vacuum in the right place at the right time.

In this final get-your-fingernails-dirty technical installment, we describe the operation of the diffusion and turbomolecular pumps (1, 2) often used to achieve the high vacuum necessary for operating many mass spectrometers. These pumps are used with a rough pump (or forepump) to move gas molecules from inside a vacuum chamber (a mass spectrometer) to outside the system. Although the pressures achieved by these two pumping systems are similar, the cost of equipment, the operating costs, and the procedures and speed of pumpdown and vent cycles are quite different.

In the past, a customer purchasing a large mass spectrometer could specify one pumping system option or the other, choosing either diffusion pumps or turbomolecular pumps. Modern instruments are more thoroughly integrated, and customer choices in vacuum systems are usually no longer possible. However, in assembling new custom-built systems and in designing new instruments, differences in the two major pumping systems used to achieve high vacuum should be understood and appreciated.

At any pressure, gas molecules move in random directions, changing directions of motion only through collision. The *mean free path* — the average distance between collisions — of gas molecules can be calculated, and this parameter serves to define the conditions of viscous and molecular flow. When the mean free path of the gas molecules exceeds the dimensions of the vacuum container, the system is under molecular flow conditions. Under such conditions, the residual gas molecules move without colliding with other gas molecules, instead ultimately colliding with the surfaces and devices within the vacuum chamber. Pumping is accomplished when a net (nonrandom) direction to the movement of residual gas molecules in the vacuum chamber is attained.

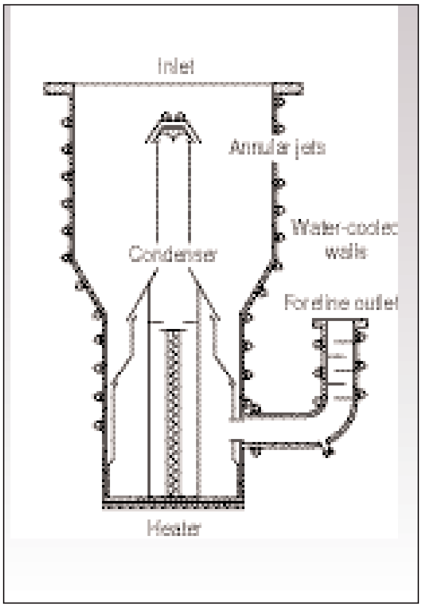

Figure 1. Components of a diffusion pump.

Diffusion pumps and turbomolecular pumps accomplish this same goal differently. In a diffusion pump, a net direction is achieved by collision of the residual gas molecules with a directed and confined stream of gas-phase molecules of the pump's working fluid. In a turbomolecular pump, the residual gas molecules collide with the angled spinning rotors on a turbine shaft. In both cases, the net direction imparted to the residual gas molecules is into a region of higher pressure and toward the exhaust of the high-vacuum pump. The exhaust of the high-vacuum pump is connected through the foreline to a rough pump that accomplishes the transport of the residual gas molecules to the final exhaust at atmospheric pressure.

The diffusion pump is a relatively simple device that has been used in vacuum pumping systems for many years following its initial description by Langmuir in 1916 (3, 4). Figure 1 illustrates the basic components of the diffusion pump. The diffusion pump contains a working fluid that is evaporated by the boiler at the bottom of the pump. (Early literature about diffusion pumps touted the advantage of electric heaters versus gas heaters for the boiler; no earlier references to shoveling coal or stacking firewood could be found.) The working fluid vapor rises through the tower (or chimney) and exits from the tower through a series of annular jets that direct the flow downward and toward the walls of the pump, which are air- or water-cooled. The diffusion pump starts to exhibit a significant pumping speed at a pressure (about 50 mTorr) at which the mean free path of the working fluid molecules is large enough that they reach the cooler pump walls. There, the fluid condenses and returns under gravity flow downward into the boiler. Local pressure in the vicinity of the annular jets is high, and collisions of the working fluid molecules with the residual gas molecules from the system impart a net direction of movement to the residual gas mol-

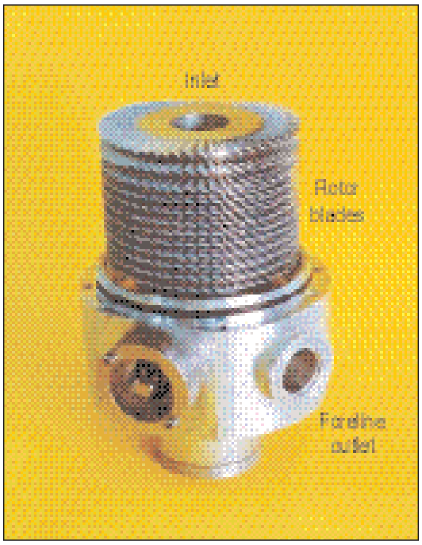

Figure 2. Components of a turbomolecular pump.

ecules. Repeated collisions move the residual gas molecules toward the region of higher pressure at the bottom of the diffusion pump. At the ejector jet, the pressure (300–500 mTorr) is such that the rough pump attached to the foreline can capture and exhaust the residual gas molecules to the atmosphere.

The working fluid used in diffusion pumps is a low-vapor-pressure hydrocarbon fluid such as Octoil, Convoil, or Santovac 5 (all proprietary product names), or a silicone oil fluid such as DC 702, DC 704, or DC 705 (also proprietary). The requisite characteristics of the working fluid are an appropriate vapor pressure and chemical inertness. The ultimate vacuum pressure attainable with a diffusion pump is directly related to the vapor pressure of the working fluid; the lower the vapor pressure, the lower the ultimate vacuum. Of course, a lower vapor pressure means that a higher boiler temperature is needed to evaporate the fluid and achieve a pumping action.

The chemical inertness is important on general principles, but also especially during the vent and pumpdown cycle sequences, and especially if the proper sequences are not followed (5). Hydrocarbon-based oils will crack and oxidize to tar if exposed to air when hot. The resultant tar coating the boiler of a diffusion pump and clogging the annular jets is difficult to remove. An abused silicone-based oil will crystallize in the boiler; the crystals are striking in appearance, but the residue is often impossible to remove, and the entire pump may need to be replaced.

Backstreaming is a term that refers to the migration of working fluid out of the diffusion pump into the main vacuum chamber. Backstreaming occurs when the operating pressure of the diffusion pump is too high, and the net downward motion of the working fluid molecules cannot be retained. The gas molecule motions become randomized, and the working fluid molecules diffuse back into the main vacuum chamber, coating the relatively cool metal surfaces that they eventually encounter. A vacuum system disassembled for inspection will often exhibit a thin, greasy-looking film on interior metal surfaces; this is the result of backstreaming of a hydrocarbon-based working fluid. Backstreaming occurs to a small degree even in normal operation but is minimized by the use of cold caps or cold traps.

The aftermath of a vacuum accident — the unintentional or too-rapid venting of a "hot" system — will be a thorough dispersal of working fluid throughout the mass spectrometer, and therefore the need for a lengthy, involved, and complete cleaning and parts replacement, in addition to a rejuvenation of the diffusion pump itself. The proper vent cycle includes a cooldown for the diffusion pump oil before venting (30–60 min). A warm-up time is also required during the pumpdown cycle for the system to reach its full pumping speed. The ability of turbomolecular-pumped systems to cycle from atmosphere to working vacuum in a shorter time is an advantage in some situations, but the initial cost of a diffusion-pumped system is lower. Costs of electricity and cooling water required for diffusion pump operation also have to be figured into a cost-based decision between the two approaches to achieving high vacuum.

The turbomolecular pump must also impose a net directional motion to the residual gas molecules in the vacuum system. The turbomolecular pump does so by repeated collisions of the residual gas molecules with rotating blades of a turbine rotor (Figure 2). The blades of the rotor spin at speeds of 20,000–50,000 rpm; the edge speed of the rotors approaches the velocities of the residual gas molecules themselves. When a collision occurs, a direction is imparted to the motion of the residual gas molecules toward a region of higher pressure and toward the pump exhaust (again, a rough pump operating on the foreline).

There are several stages of spinning rotors, with the angles of the rotor blades changing along the profile of the pump from inlet to exhaust, as shown in Figure 2. Residual gas-phase molecules are moved toward the exhaust and then are removed from the foreline by the rough pump. Higher molecular mass gas molecules are pumped more efficiently by the turbomolecular pump than lower molecular mass molecules; therefore, there is very little backstreaming of lubrication oil molecules into the main vacuum chamber. The turbomolecular pump has the characteristic of being a clean pumping system with no backstreaming.

Clearly there is no heater to warm up or cool down in a turbomolecular pump. A system so pumped can be vented to atmosphere and then pumped down more quickly than can a diffusion-pumped system (minus the use of isolation gate valves in the latter, which decrease the effective pumping speed). Diffusion pumps must be mounted at the bottom of a vacuum chamber, and in a vertical orientation so that gravity can return the condensed working fluid to the boiler. A turbomolecular pump can be mounted in any orientation relative to the vacuum chamber. There is residual vibration from a turbomolecular pump, so the mounting system often includes an integral vibration isolation device. Finally, although the pumping speed is not as high as with diffusion pumps, the smaller size of the turbomolecular pump is often an advantage in today's smaller mass spectrometers, and there is no need for a cooling water connection.

Mass spectrometrists seem to pay less attention to vacuum-related issues today than in the past. High vacuum has become easier to attain, pumps are more reliable, and pumping systems are more automated. In vacuum, as with so much else, the modern mass spectrometrist exhibits the habit of deferring or ignoring maintenance. And, as with much else, there is an ultimate cost to be paid for so doing. In a mass spectrometer, the cost is decreased performance in terms of lower sensitivity or lower resolution; mass spectra that deviate from those measured and archived under more ideal conditions; and shorter lifetimes for replaceable items such as electron multipliers, ionization gauges, and source filaments. The rough pump has the final task of exhausting the gas molecules to atmosphere, and maintenance of the rough pump is probably most important.

Rough pump maintenance involves changing the rough-pump oil on schedule, checking and replacing the seals when necessary, managing the heat generated by these pumps, and regularly inspecting vacuum hoses and clamps. But high-vacuum diffusion and turbomolecular pumps also require maintenance. Internal maintenance requirements are described in the operators' manuals and instructions; these requirements may involve checking the pump oil properties in a diffusion pump, or seals and rotor maintenance procedures in a turbomolecular pump. Diffusion pumps generate a great deal of heat; the pump may be air cooled, in which case the fan and ductwork must be kept clean. If the diffusion pump is water- or fluid cooled, there is more to be done. A closed-loop chiller will engender its own specific maintenance requirements, including regular checks of the thermal trips that shut down the system when there is a loss of chilling capacity. A tap water–cooled diffusion pump system will include an in-line filter that requires regular replacement. If you drink tap water, don't look too closely at the filter or think too deeply about why monthly filter replacement is needed. Check the integrity of the water connections on a regular basis. A flooded laboratory is not a pleasant way to begin one's morning, mop in hand, with abject apologies to colleagues on the floor beneath yet to be made.

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Kenneth L. Busch *is associate dean of Sponsored Programs and a professor of chemistry at Kennesaw State University (Kennesaw, Georgia). He can be reached by e-mail at kbusch@kennesaw.edu. He has spent a few mornings in the laboratory with mop in hand, reflecting on why water connections on diffusion pumps always seem to fail at 3:00 A.M. local time, regardless of the actual geophysical location of the lab. Retentive in nature, he has kept a 25-year observation log of diffusion-pump cooling-water connections, noting that approximately 15% of home-built systems are plumbed in reverse. The coldest water directly from the tap should enter at the high-vacuum top of the diffusion pump. Late at night, he wonders who first came up with the term "guppy pump" and whether the world would be a better place if mass spectrometrists held all positions of power and influence.* ◆