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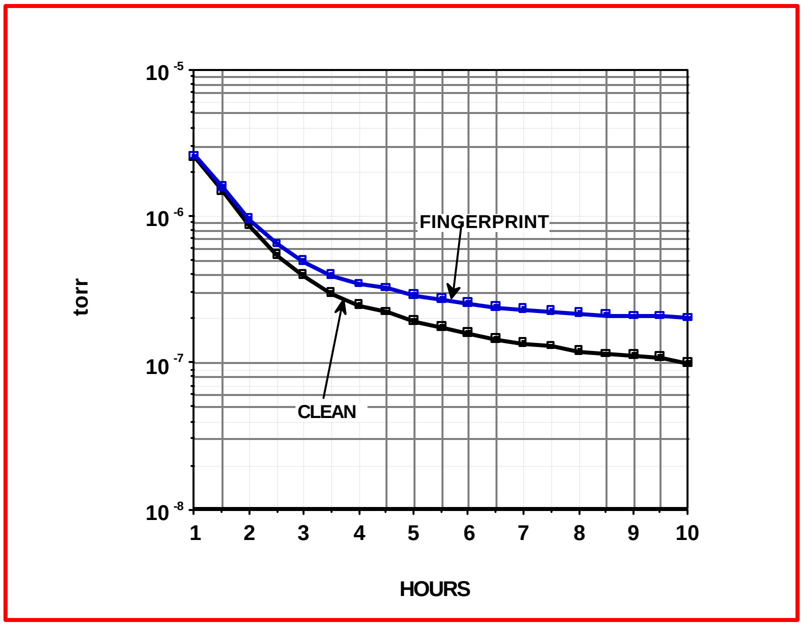
Contamination in Vacuum Systems: Sources and Remedies

Contamination in vacuum systems is not a specific topic within vacuum technology. It's vacuum technology itself. If you consider that anything within a vacuum system that hurts or inhibits what you're trying to do with vacuum technology is a contaminant, then contamination becomes the entire subject. If, for example, a system won't pump down to the specified pressure that's required to carry out a process, the residual gases within the chamber are a contaminant. Contaminants of this sort are usually covered in any book or article on practical vacuum technology, but the more insidious sorts are often side-stepped or given short shrift. We include here contaminants introduced into the system during normal operation and contaminants arising from the system itself. In general, these contaminants can be broken down into two overall categories. The first group is made up of contaminants that enter the process chamber as gases and are capable of being pumped away. These are called contamination resulting in additional partial pressure (CRAPP). The second group is composed of contaminants that enter the chamber and are not pumped away. These are called contamination resulting in undesirable deposits (CRUD.)

Viton O-rings present a good example to help sort out the two types. A fresh and vacuum unbaked o-ring will contain contaminants trapped within its bulk. Water, in large quantities, is chemically formed within the material during the manufacturing process. Additionally, solvents and plasticizers with a fairly high vapor pressure will be also trapped within the bulk. When the O-ring is used as a seal on a system, these materials will slowly diffuse to the surface, desorb, and then are pumped away. As they enter the chamber, they provide CRAPP in that they exist within the chamber as partial pressures of gases that can poison a process or limit its pumpdown capability. Although they will be pumped away in time, the time period to remove them from the O-ring might be weeks or months. O-rings also often contain unreacted monomers and relatively low vapor pressure plasticizers that are left following the manufacturing process. If the O-ring is baked or heated by the process, these materials can be released from the O-ring's bulk and then re-condense within the chamber. The resulting condensates can easily inhibit pumpdown or become impurities that pass into the process. Under these conditions, the O-ring has also provided CRUD. Both of these types of O-ring generated contamination can be avoided by pre-baking them under vacuum before installation.

A major source of contamination is often encountered by merely using the system for its intended purpose. Work loads that are placed into the system during each product run usually contain some sort of potential contaminant. For example, a substrate to be coated could easily contain water vapor, solvents, or other materials either within its bulk or on its surface. When they are placed within the process chamber, they will provide CRAPP for some time before the outgassed contaminants are pumped away. This extension in pumpdown time can obviously result in extended process times which will, in turn, limit the product throughput per shift. Simple procedures to avoid the addition of contaminants can be vacuum pre-baking, air baking with an infrared lamp, room temperature vacuum treating, or merely storing in a desiccator prior to use. The basic key to avoiding such problems is to look into the prior history of the material to be placed within the process system to ascertain any steps in manufacture, storage, or preparation that could introduce contaminants. In many practical processes, the materials cannot be pre-treated, and it is necessary to accept and live with the fact that they will outgas for a long period. Still, it is useful to be aware of their potential behavior by adjusting the process parameters accordingly.



A single fingerprint can have a large effect on a system's pumpdown.

The act of loading the system can also introduce problem contaminants. A single fingerprint exposed to the vacuum has a gas load of about 10^{-5} torr liters/sec. This translates to 1.3×10^{-5} atm. cc/sec., which would be an intolerably high leak rate for most high-vacuum processes. A gas load of this magnitude would require a pumping speed of 100 liters/sec. just to maintain a partial pressure of 1×10^{-7} torr of the gas emanating from the fingerprint. Depending upon the cleanliness and dryness of the offending finger, it could take between 24 to 36 hours just to pump away the CRAPP. Additionally, CRUD will be left behind. In the late 1960s, some researchers thought they'd discovered a form of polymeric water left in capillary tubes placed in a vacuum chamber. They called it polywater, but it turned out to be the residue of perspiration from handling the tubes prior to their being placed in the vacuum chamber. The obvious solution is to handle materials and system parts with clean nylon or plastic gloves, or clean tools such as forceps or pliers.

Cleanliness is, of course, highly important to all surfaces exposed to the vacuum. The stringency of this requirement has been so ingrained into the minds of most vacuum practitioners that any and all surfaces are routinely wiped down with solvent-soaked lint-free tissues prior to installation or pumpdown. As a routine technique, this can lead to a fool's paradise scenario. Most solvents will leave some degree of residue behind when they evaporate in air. Whatever impurities are in the solvent will remain behind. This might be impurities already in the solvent as it came from the supplier or anything dissolved within it during handling. Elastomer O-rings present a particularly vexing problem. O-rings that are about to be installed are often solvent-wipedin the belief that they will be clean as they are installed. The elastomer material will absorb enough solvent that it will cause swelling of the material. This non-visible swelling causes the polymeric chains to move and open up sufficient to greatly increase the O-ring's permeability to air. Thus, in effect, building a higher than intended leak rate into the system that results in a higher total gas load than would be encountered with no cleaning at all. The increased permeability will remain until all the solvent-induced CRAPP is pumped away over perhaps months of pumping. In the case of Viton, the higher rate of permeability often remains permanently. Vacuum baked O-rings should be used whenever possible, and neither cleaned nor greased unless they are re-baked. Pre-baked o-rings are commercially available.

Vacuum pumps can provide one of the most commonly encountered sources of both CRAPP and CRUD. The most prevalent source is from oil-sealed mechanical pumps although other pumps can provide contamination as well.

Any pump containing oil as a sealant for the pump mechanism as in rotary pumps or as a shaft sealant as in some blowers will tend to allow oil vapor to backstream from the pump into the chamber. When, for example, a rotary vane oil-sealed pump is used for roughing/backing, little transport of oil vapor occurs when the chamber and pumping line are under viscous flow conditions. The number of molecules flowing from the chamber to the pump cause enough molecule-to-molecule collisions to bar most of the oil vapor from going "upstream." Once the pressure drops to molecular flow conditions, though, the oil vapor can freely flow through the pumping line and into the chamber. Some of the oil vapor will condense on the cooler surfaces of the pumping line, but some will reach the chamber. A monolayer or so of oil on the chamber's inner surfaces probably can't be detected visually, but a water drop test will either bead or not to tell the story. The usual response to this problem is to interpose a foreline trap of one of the common types between the pump and the chamber. This can be an effective solution, but only if the system is handled properly and a mistake is never made.

Transport of oil into a chamber is not only by vapor vector. Since oil will readily wet surfaces, it can, and will, creep along the inside walls of the pumping line until it reaches the chamber. A good foreline trap will usually stop the oil vapor, but many commercial traps have warm walls with no creep barrier to stop the surface transport through the trap. The surface creep problem requires a certain amount of vigilance to make sure that the trap is cleaned with solvents in a regular maintenance schedule to stop the oil's motion before it can reach past the trap. Additionally, any of the three commercially available trap types, cryogenic, adsorption, and absorption, have trapping media that will become saturated with oil at some point and require maintenance. In most cases, the maintenance should be accomplished with the trap removed from the system. Any time that trapped oil is released from any trap that's in place, some of the oil will go to the pump side and some oil will go to the chamber side. Any oil going to the chamber side is too much.

The most obvious and most effective way to avoid oil contamination is to use an oil-free pump. These pumps are sometimes called "dry pumps". Assuming that a pump called a dry pump cannot provide a source of oil contamination is a dangerous assumption. The term has come, erroneously, to refer to any pump other than an oil-sealed rotary vane or rotary piston pump. Many of these pumps contain lubricants in bearings or shaft seals that can emanate hydrocarbon vapors that will transport into the chamber. Before assuming that any of them are intrinsically oil-vapor or hydrocarbon free, it is necessary to look carefully into each and every design in terms of both type and manufacturer.

Both CRAPP and CRUD contamination can enter any vacuum system that is not carefully guarded in terms of barring contamination. In fact, sporadic or "one time" contaminants have been known to act as a catalyst or facilitator to make a process work once and never again. A repeatable process will only work if the conditions are repeatable run after run, and contamination can turn a routine process into a nightmare. A small dose of paranoia can be a healthy addition to vacuum technology.