

## How Low Can You Go? Part 1

### Improving Base Pressure in HV and UHV Chambers

So, you'd like a lower base pressure in your chamber, eh? Wouldn't we all! If you've been paying attention while reading earlier *Lesker Techs* you know there are two choices for lowering the base pressure:

**Increase Effective Pumping Speed**

or

**Decrease gas load**

In principle, increasing EPS is simple: shorten tubes; increase diameters; minimize number of bends; increase number of pumps or replace with higher pumping speeds; and double up on, then differentially pump, all o-ring seals. As is obvious, this is 'big bucks' country. So, apart from some simple add-ons to increase pumping speed, (to be identified in a later article) we are done with EPS. Except, I can't resist a parting shot, "How come you designed this system so poorly in the first place?"

To decrease gas load takes time, may cost major money, and on rare occasions, is just not possible. There are many gas load reducing techniques available. Each has its own quirks, suggesting that before you jump on one particular fix, perhaps you should understand the general characteristics of gas load. In this, Part 1 of a trilogy, I'll tell about normal gas load sources and why they exist.

### "Given" & "Observed"

To start somewhere, let's assume your chamber:

- \* Passes a He leak check and is, therefore, leak and permeation free
- \* Has no virtual leaks
- \* Was never exposed to high vapor pressure elements (Hg, Cd, etc)
- \* Has no conductance restriction between the pressure gauge's measuring volume and the chamber

That is, in this 'first pass' the inadequate base pressure (**IBP**) seems to be caused by some gas or vapor desorbing from the chamber walls and internal fixtures.

But we can refine this with a 'second pass' asking ourselves, how did this **IBP** arise?

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Did the base pressure change:

1. Suddenly?
2. Slowly over days/weeks?
3. Only when you fill the LN2 trap?
4. Never has changed but isn't low enough?

One glance at the system's log book will immediately give you the answers. But what's that I hear? Did you say, "What log book?" Incredible! Go to jail! Go directly to jail! Do not pass... For those with system log books, throughout this series I'll try to drop clues indicating what your log book might be telling you about the conditions you're experiencing. But you'll have to do the 'Dick Tracy' bit.

## Gas Load Components

The surprising thing about gas load is, the components and sources are often predictable. For example:

- \* Pumping a chamber from atmosphere—when it's between  $10^{-2}$  torr and  $10^{-7}$  torr, the gas load is ~80% water vapor.
- \* If a chamber remains between  $10^{-2}$  torr and  $10^{-6}$  torr when pumped for many days, the major component is often 'hydrocarbons'.
- \* In a stainless chamber between  $10^{-9}$  torr and  $10^{-12}$  torr, the gas load is ~80% hydrogen.
- \* If the base pressure rises when you fill the LN2 trap, the gas load is ~80% nitrogen.
- \* If the base pressure jumps up quickly, and there is no detectable leak, the gas load is either water vapor or gas from an attached gas cylinder.

No doubt enquiring minds are already asking "How do you know this?" And the **short answer** is: too much time spent playing with residual gas analyzers (RGAs) attached to vacuum chambers.

You too can flirt with a Sherlock act by adding an RGA to your system. Although some vacuum purists decry the quadrupole RGA as semi-quantitative at best, who cares? It's the qualitative aspects that tell you what gases are present. If you know how to interpret a mass spectrum qualitatively, having semi-quantitative results is a bonus. The RGA is the best available diagnostic tool for vacuum problems—at least the equal of Holmes' magnifying glass.

## The long answers are...

### Water Vapor

The unresolved atomic forces on any clean solid surface are reduced by the adsorption of molecules from the surrounding air. Initially the gas adsorbed may be  $N_2$  and  $O_2$ . But quickly, they are replaced by water molecules which form a multi-molecular layer. And why is water such a problem?

If the binding energy (BE) between a surface and adsorbed gas is  $<15$  kcal/mole (probably Ar and  $N_2$  are in this group) then, when the surface is under vacuum, all molecules desorb at room temperature and there is no gas load problem. If the BE is  $>25$  kcal/mole then, under typical vacuum conditions, gas molecules desorb too slowly to cause a gas load problem. Unfortunately, water's BE to most surfaces is  $\sim 20$  kcal/mole.

### 'Hydrocarbons'

To a chemist, a hydrocarbon contains only carbon and hydrogen. The sources of vacuum 'hydrocarbons' may include: pump oils, fingerprints, skin flakes, hair, plasticisers from wire insulation, machining oils, dust mites, glycol coolant, even the occasional cheese sandwich. While all these materials are organic, few are strict hydrocarbons. However, we'll continue with the common vernacular and lump such contaminants as hydrocarbons.

If the base pressure is still high after pumping for many days, hydrocarbons may predominate because:

- \* The chamber's initial cleaning was inadequate.
- \* It has major fingerprint contamination
- \* Pump oil vapor is backstreaming
- \* Bad operating procedure dumped oil in the chamber (and you don't know it happened since there's no log book and the perp's keeping *stumm*).
- \* The chamber has a slew of PVC insulated wires, a chunk of Plexiglass, or that cheese sandwich.

### Hydrogen

Stainless steels are made, wrought, and rolled in air. (Didn't Elvis sing something about 'wrought & roll'?) At high temperature, iron and water vapor react reversibly:



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with the equilibrium position determined by temperature. A type 300 austenitic stainless steel at room temperature may contain as much as 0.1 atom % H in the bulk metal.

Perhaps fortunately, hydrogen atoms diffuse slowly through austenitic stainless and at pressures between  $10^{-5}$  torr and  $10^{-8}$  torr,  $H_2$  is a minor gas load component. However, to reach UHV pressures, we first remove the surface-bound water and hydrocarbons. This leaves the hydrogen and, unfortunately, its combined rates of: diffusion as atoms to the surface; re-combination to molecular form; and desorption from the surface as molecules are just enough to stymie efforts to get below  $10^{-9}$  torr without some serious clean-up. No, Nellie, getting to UHV is no walk in the park.

## Nitrogen

This is an 'odd-man-out'—a really weird leak. In a normal vacuum chamber, making a surface LN2 temperature reduces the base pressure, by Charles' Law if for no other reason. If, when you fill a (commercial) LN2 trap the base pressure **rises**, yet there is no detectable leak, you should immediately suspect a bad weld in the trap. Since the leak opens only when the trap is cold, locating it is tough. Fixing it requires someone skilled at leak detection and a good welder. Typically, replacement is the cheapest option.

And what was the 'commercial' crack about? Well, if your trap was designed and built in-house, it's possible the LN2 is freezing an o-ring seal. You should, however, find that as a detectable leak.

## Water or Cylinder Gas

How can I be sure that a base pressure jump is caused by either water or the gas from a cylinder? I can't, but I'm playing with good odds.

Any system with internal water cooling must have a heater (or why bother cooling?). A heater causes expansion and differential expansion is a mechanical joint killer. For example, a copper tube brazed to a heated stainless device can cycle through a temperature range that causes a leak to develop. The water evaporates into the chamber which promptly goes into **IBP**-land. Trouble is, leak testing the outside vacuum shell tells you nothing.

To check this, you first blow water out of the cooling coil and then flush helium through it. Unfortunately, even this is hardly a guaranteed test. The residual water in the 'hole' may be liquid now, but when the chamber is evacuated (to allow the leak checker to work) it turns to ice and

acts as a good, if massively outgassing, seal. My suggestion is: put the chamber under vacuum; pass warm (hot?) nitrogen gas through the coil for many hours. Then try helium leak checking.

What's this cylinder gas deal, then? OK, everyone knows that needle valves do not shut off gas flow, right?

Why is there dead silence? Hello, out there! You do all remember that, don't you? Good!

So, no one has ever connected the output from a cylinder regulator through a needle valve directly to the chamber. [And unless I hear a collective shout of "Dam' right, Skippy!" you're all confined to barracks to read Nupro needle valve literature for a week.]

Since we are 'as pure as the driven snow', we know the gas must be shut off at the chamber with a real shut-off valve, typically a right-angle HV/UHV valve. Unfortunately, since the needle valve leaks, when the right-angle valve is closed, pressure builds behind it until the volume reaches the regulator's set pressure. And this causes two levels of embarrassment:

1. When the angle valve is opened, gas trapped in the volume does its Boyle's Law thing. The results can vary from a mild pressure spike to a full-blown pumping stack melt-down.
2. But, of particular interest for this article, if the angle valve's seat develops a leak, the **BP** goes to **IBP**. Again, external leak testing shows you zilch. You need to attach a leak checker to the chamber, disconnect the needle valve, and flood the angle valve's seat with helium.

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## Conclusions

Although deliberately excluded here, leaks and permeation are a commonly observed reasons for **IBPs**. However, leaks rarely happen spontaneously while the vacuum system is just rolling along at base pressure. They are caused!

Since your log book now notes **everything** with dated and timed observations including: pressures at all measurement points; heater temperatures; added and removed components; gasket and flange changes; tightened bolts and clamps; sample introductions; actuation of mechanical feedthroughs; ventings; pumpdown start times; pressures at various times during pumpdown; and even the time your cheese sandwich disappeared; you should be all over leak and permeation **IBP** problems like crop circles over spring wheat.

For non-leak and non-permeation **IBP** questions, with a little cogitation, we can often predict the cause and what contaminants that suggests. If one of those undetectable leaks is suspected, check the three most likely places: cooling coils; gas connections; and LN2 traps. If the system has no such devices to confuse us but still has an **IBP**, we can consider clean-up options... which we'll do in the next issue of Lesker Tech.

If you have comments, complaints, or want expansion on certain topics, please contact: [techinfo@lesker.com](mailto:techinfo@lesker.com) but remember, I'll ask for the relevant entries in your log book.

**If you don't understand terms like Charles' Law or Boyle's Law, go to [www.google.com](http://www.google.com) and search for them. They're out there somewhere.**

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