

We appreciate the response to this publication feature and welcome all contributions. Contributions may be sent to Phil Oshel, our Technical Editor, at his e-mail address: peoshel@facstaff.wisc.edu. Phil can also be reached at 1-(608) 833-2885

A Note on Iodine and Vacuum Pumps

Since iodine is a reactive gas, an activated carbon trap is required between the mechanical pump and the turbopump. When I was at the University of Alabama at Birmingham, students on two occasions left the iodine gun valves open. They destroyed the mechanical pump springs and rendered it useless. I found out that we were not the only ones that this happened to. GATAN fixed a bunch of mechanical pumps under warranty and came up with a fix: a carbon trap with a sight glass on the mechanical pump side. Inside the tube where it could be seen, there was a silver strip that would change to black when the trap got saturated. The trap was regenerated by replacing the charge of activated carbon, otherwise iodine would get into the mechanical pump. I would worry that copper pipe used for an exhaust line might react similarly to iodine as the silver. The iodine does come out the exhaust of the mechanical pump.

I thought that the fix was rather expensive at the time, but it did work. A cheaper way to do this would be to get a zeolite trap and replace the zeolite with activated carbon from a pet store that sells aquarium supplies.

I should add that even though the iodine guns were left open twice, the turbo pump was in pristine condition afterward. It was

the mechanical pump on the inside, mostly at the exhaust that was saturated with the iodine, and where the mess was.

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A Simple Way to Eliminate Frost Build-up on Cryo-SEM Samples

In the event of frost contamination of a sample, here is a cheap trick that I use to sublimate it off the surface once it is in the SEM: Just hook up a bottle of dry nitrogen gas to the air input of the SEM via a regulator set to about 3 to 5 psi. Vent to atmosphere, do not open chamber door, and then immediately pump back down. All the while the cryostage is kept at usual maximum cold temperature, about -120°C in my case. The gas imparts just enough thermal energy to the surface frost to sublimate it off, but not enough nor long enough to sublimate the sample itself. It may not even be necessary to vent the SEM all the way to room pressure before pumping back down—experiment and see what works.

The big advantage is this only takes 4 minutes for a complete defrost cycle. A disadvantage is that the beam must be turned off, so the process can't be watched, but this is not a problem. I prefer not to defrost by heating up the stage, the sample stub, and the entire mass of the sample just to get some heat to the surface frost, as it takes so long to do that, plus cool down again, and it runs the risk of overheating and drying out or etching the surface layers of the sample. Also, with my model of cryo-system (Emitech K-1150, little sibling to the K-1250) if I should need to

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