

## Making a Dispersion of Silica Particle for Microscopic Analysis:

Silica particles don't know that they are supposed to separate and stay away from each other when dispersed in a liquid followed by a droplet of this liquid suspension being placed on a solid surface. They tend to agglomerate very quickly leading to a difficult-to-analyze situation, especially using automated means of analysis. The size range expected could be on the order of 3 to 6 nm.

This is the ideal application for the camphor/naphthalene method which I described several years ago. Credit for the technique, or at least the one who taught it to me was an innovative microscopist then working at the DuPont Experimental Station in Wilmington, DE by the name of Robert P. Schatz, in about 1968, now deceased:

Take a 60% camphor/40% naphthalene mixture and heat it to 20 or so degrees above room temperature on a hot plate in a small beaker or flask, the two organics are miscible in each other and this is the eutectic composition.

Once a clear liquid, add a small amount of the silica (not more than 0.1%), which disperses quite readily. Then, using a pipette, take out some liquid and put a drop onto a carbon coated glass slide, at which time the drop is instantly frozen solid (it is at room temperature). Put the slide into your vacuum evaporator to pump out all night and the "magic" is that the solid eutectic sublimates at room temperature at such a rate that by morning it is completely gone, leaving the silica particles uniformly dispersed on the carbon film!

The rest is obvious. You can pick this up on a grid as is, or in order to bring out more contrast, Pt/C shadow, probably using an angle not more than 30 degrees. You can float the "replica" off of the slide directly onto a grid and viola! You have particles completely dispersed, virtually no doublets or triplets, and a field quite amenable for automated image analysis (as a bonus).

One important further suggestion: Some times these silica particles tend to fuse together as little "chains". If you suspect this is happening, be sure to take the micrographs as stereo pairs because you can in fact capture this three dimensional spatial information.

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*Disclaimer: We do not sell either the camphor or naphthalene so have no vested interest in whether people use this method or not. It is just a really neat method for the preparation of fine particle samples in this size range. We are, however, obviously set up to use this method as a service for others.*

## OIL DROPS ON AN EDS DETECTOR:

### A) Diagnosing and Preventing Contamination:

Oil drops on an EDS detector in an instrument with a vacuum system based on an oil diffusion pump could be from either the diffusion pump, or the rotary backing pump, or both. I don't know any easy way to tell which: however, if a few drops of it can be collected, an infra-red spectrum could be run, and that should give a definitive identification.

In the past, we have had trouble with oil collecting on the EDS detector in one of our SEMs and an FTIR spectral analysis showed it to be mainly from the rotary vane backing pump. As discussed on p. 144 of my book, "Vacuum Methods in Electron Microscopy", the molecules of the roughing pump oil get broken down into volatile low molecular weight fragments by the mechanical rubbing of the parts inside the pump, and these fragments readily migrate into the backing line and hence through the diffusion pump and into the vacuum system. Most diffusion pump oils are quite resistant to thermal and oxidative degradation, and so are unlikely to be a major contributor unless the vacuum system on your instrument is poorly designed or your diffusion pump has been subjected to one of the improper operating situations described on p. 214-223

of VM in EM.

Although there is not much that can be done to reduce contamination from the diffusion pump once an instrument is built (except to avoid the operating hazards just mentioned), there are two fairly effective ways of dealing with contamination problems arising from the roughing pump:

- 1) Give the instrument a good cleaning and then install a good foreline trap, which must then be faithfully maintained to preserve its effectiveness (VM/EM p.147-149).
- 2) Use an inert gas purging system, which is brought into operation when the instrument can be put into the standby mode, to attempt to sweep the oil molecules back out of the vacuum system - VM/FM p. 145

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### B) Cleaning EDS Detector Windows - I:

In any event, the contaminating oil must be removed from the EDS detector window, otherwise it will begin to alter the detector sensitivity. This can be a very tricky process, and should be done with a great deal of care. It is best to check with the manufacturer of your detector for their recommendations before you try anything. However, we have cleaned the standard beryllium detector windows several times in the past by using squeeze bottle or a dropper to run a bit of petroleum ether over the face of the window. You must, of course, be very careful not to puncture the window, but then the whole operation is one of high risk to begin with. We have even been able to do this without removing the detector from the SEM by using a long dropper and collecting the petroleum ether on a tissue as it runs off the detector.

Petroleum ether is basically a low-boiling hydrocarbon fraction that is a reasonably good general solvent, especially for the hydrocarbon-based rough pump oils, but which does not attack epoxies and which also evaporates quite readily and cleanly. Do NOT use a solvent such as acetone, methyl-ethyl ketone, or an alcohol, which may attack the epoxy cement normally used to hold the window in place.

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### C) Cleaning EDS Detector Windows - II

The above method is fairly similar to ours. Silicone pump oil is little used in EMs, but if it is, and kerosene is used to clean the detector, a second solvent would be needed to remove the kerosene residues.

Reagent grade methanol has been suggested (by an EDS manufacturer) as a suitable solvent for hydrocarbon pump oils. Presumably this was recommended because it did not affect the Be window's glue and it is a good solvent of vacuum oils and fluids. Petroleum ether is likely a better choice.

We too could leave the detector in place, and I collected the used solvent in a Petri dish and tissue.

The significant difference in our methods was the application. Because the window is recessed and our detector had some tilt, it seemed that little solvent would reach the window if the methanol or petroleum ether was just dripped from the top. This is the method we adopted:

Use a polyethylene Pasteur type pipette with a fairly large bulb. Pull the tip into a fine capillary after brief (5-10 seconds) heating in a low flame. (Tip: move and rotate pipette while heating: pull vertically, this keeps the tip straight while the plastic sets). Cut the tip so that about 100 mm of capillary tip and a small bore, perhaps 0.2 mm, is available.

FILL the bulb with methanol; releasing air once or twice by up-turning and squeezing the bulb. Filling is slow but no trouble.

Slide the stand until the pipette tip is within 20 to 40 mm of the

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