TECHNICAL NOTE: MAKING MOLYBDENUM TRIOXIDE SPECIMENS

SPI Supplies
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Using SPI-Chem™ Ammonium Molybdate to make TEM Specimens

There are several methods that are used for the making of the Molybdenum Trioxide test specimens.

Ammonium molybdate direct method:

Operating strictly in a well ventilated fume hood, heat a small quantity of SPI #02551-AB ammonium molybdate crystals in the bottom of a small beaker on a hot plate until white smoke starts to be evolved. At this point, quickly expose a carbon coated grids to this white smoke. Crystals should form on the grids.

Ammonium molybdate crystal dispersion method:

A variation of this approach is to put a small quantity of the ammonium molybdate into a laboratory crucible, preferably new and other wise unused, which is then heated using a laboratory Bunsen burner. Heating should be done slowly and at some point, the crystals will become red hot. At this point it will start to sublime. Because the top of the crucible is a bit cooler, the vapor will condense and crystallize near the top lip of the crucible, and after the crucible is cooled down to room temperature, the crystals can be removed and dispersed in water or methanol. A single drop of this dispersion is then placed on a high quality carbon coated grid and you are ready to do your image rotation calibration.

As with the first method, the entire procedure must be done inside of a well ventilated fume hood.

Molybdenum metal decomposition method:

Using an SPI # 01808-AB molybdenum boat in a vacuum evaporator, apply voltage to the boat, in air, and not under vacuum. Slow turn up the voltage so that as the boat starts to become hotter and hotter, there will come a time when white smoke will start to be evolved. And as with the first method, at this point, expose carbon coated grids to the smoke.

Of course, in the end, you can always purchase our own SPI produced molybdenum trioxide rotation calibration specimens, ready for immediate use!

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