Tricks of the Trade: Particle Mounting Methods

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Those of us who have particle identification problems sometimes have very small samples, say, only a few nanograms - even less sometimes. We also like to make up permanent Aroclor 5442 preparations of these tiny samples for safe keeping and future reference. If we mount such small samples in a normal drop of Aroclor filling an 18 mm diameter coverslip, we may spend a few frustrating minutes looking for the sample in a few acres of Aroclor.

My solution is to use a very tiny Aroclor droplet under a small coverslip. Ten millimeter or smaller round coverslips are available from Erie Scientific in Portsmouth, New Hampshire. To obtain a suitable Aroclor droplet (Figure 1), I keep my Aroclor-filled canada balsam bottle nearly empty (2-3 mm deep) on my hotplate at 98° C and with a fine-tipped glass rod permanently therein. I place my clean microscope slide with a centrally Sharpie pen-marked "q" on the under surface on the hotplate. Ten seconds later, I remove the glass rod and tick the tip wet with Aroclor to the circle of "p" on the other side of the slide. I then place a clean 10 cm round coverslip on this drop (no sample), heat it to 100° C, and press on the coverslip to thin the Aroclor. It then cools to room temperature as I walk to the refrigerator with the prep and a razor blade. After 10 seconds for the slide on the cold freezer compartment surface, I remove the slide and flick off the coverslip with the razor under one edge. Now, back to the stereo microscope where I use a No. 2 or 3 tungsten needle to place the sample at the center of the Aroclor circle and replace the coverslip (Figure 2). Now, another 10 seconds on the hotplate followed by removal to a heat insulating Post-It pad and quick use of a coverslip gripper-particle crusher and disperser (i.e., a pencil eraser). I disperse the sample into single particles in a small area at the center of the Aroclor preparation. After circling its expanse on top of the coverslip (Figure 3) I am ready to find and examine the particle(s) by PLM.

Once in a great while, I then find I cannot identify one of the important components of this Aroclor preparation by size, shape, color, refractive indices, and birefringence. I must then get one of the biggest of those particles out, cleaned off and remounted in a different liquid or for SEM/EDS or FTIR/ microscopy. This I do, starting with circling the chosen particle location on top of the coverslip. I then invert the slide and trace the particle circle on the bottom of the slide. Then back to the freezer to flick off the coverslip.

A fine-tipped tungsten needle, then draws a square through the Aroclor and around the particle. With the same needle working under a small drop of water over the particle on top of the Aroclor, I spear the square of Aroclor (and particle) and bring it out through the water drop surface to a clean slide. The Aroclor is removed using a drawn-out polyethylene pipette filled with amyl acetate leaving a clean particle. A small droplet placed near the particle is drawn by a nearly flat No. 2 needle tip over the particle, hesitating a few seconds for the Aroclor to dissolve and then continuing on to move the amhyl acetate droplet plus Aroclor away from the particle. Several airborne trips back to the particle and drawing more droplets away usually leaves a clean particle in less than 30 seconds total time. Needless to say, this operation takes place under the stereo at times 40-50.

The dry particle is then mounted in a different liquid for PLM, for SEM/EDS on a beryllium stub or in a diamond cell for FTIR/microscopy. ■

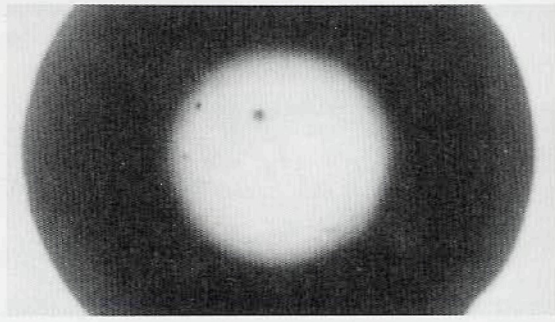

Figure 1: Droplet of <2 mm Aroclor 5442 on the slide, 25X.

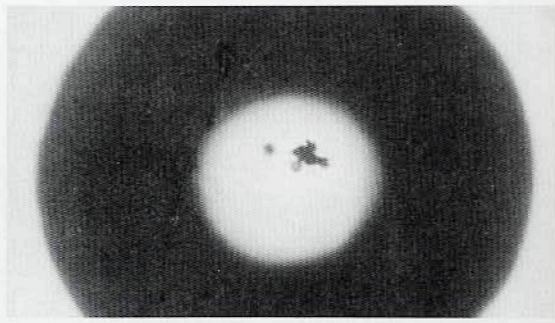

Figure 2: Aroclor droplet plus sample.

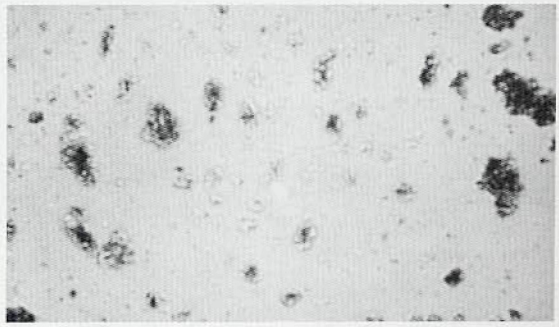

Figure 3: Dispersed particles of the sample in a 4 mm² area.

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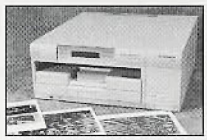

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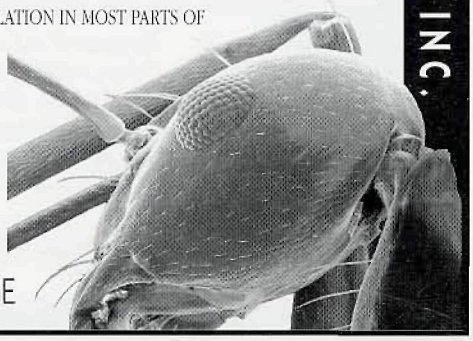

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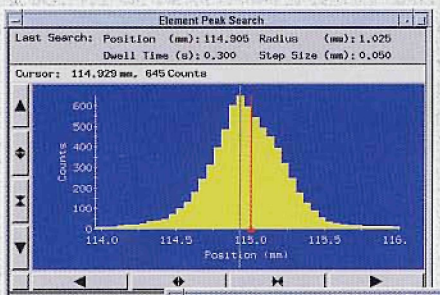

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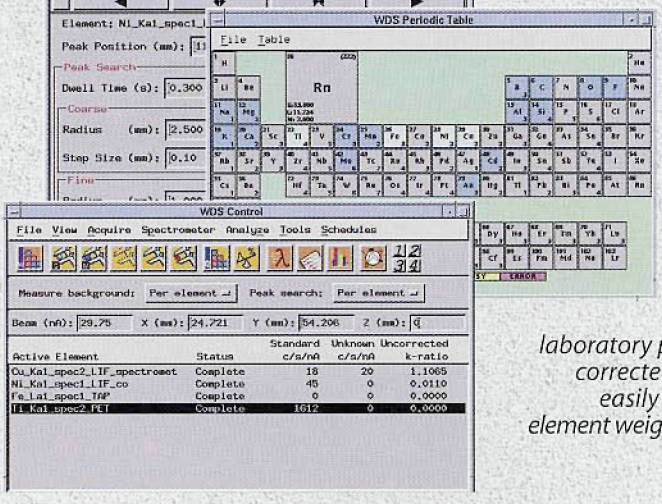

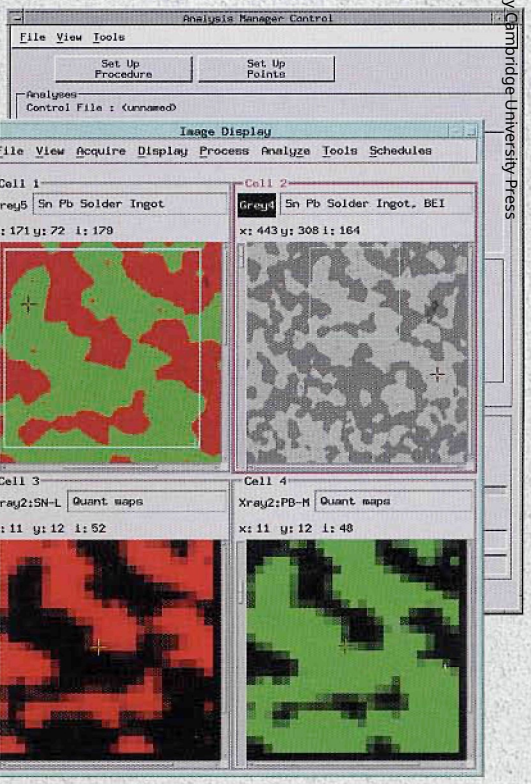

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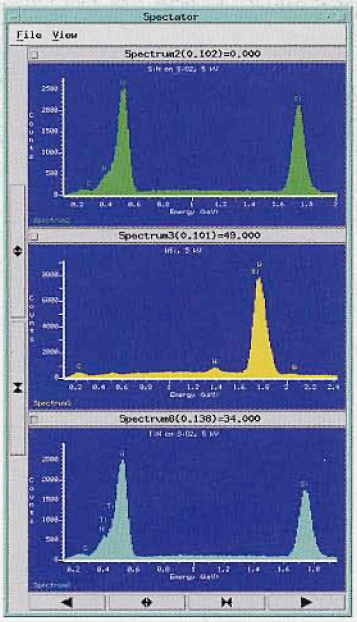

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