

Handling and Mounting Small Crystals

Handling and mounting very small (below 100 μm) samples is challenging. Small crystals seem to swim away from a mounting tool; if you do manage to mount them, they end up on the neck instead of in the aperture; and by the time you're ready to flash cool, the crystal and/or your drop have dried out.

Mounts seem to push small crystals away, as if the mounts were too hydrophobic. What you see is a simple demonstration of laminar flow and Stoke's Law. When you move the mount through a crystal-containing drop, the liquid flows laminarly around it. If the crystal's density matched that of the liquid, it, too, would just flow around the mount, and it would be nearly impossible to snag it.

If the crystal's density is larger than that of the liquid, the crystal will sediment under the influence gravity toward the mount below it. The crystal's sedimentation speed is determined by the balance between the gravitational force pulling down and the viscous drag force given by Stoke's law that opposes motion. This sedimentation speed varies as the square of the crystal diameter. Thus, the time you have to wait for a crystal to sediment onto the mount increases rapidly as the crystal gets smaller. A 10 μm crystal sediments at less than 10 $\mu\text{m}/\text{s}$, 100 times more slowly than a 100 μm crystal.

Fortunately, the presence of the mount's aperture makes things a bit easier. As you move the mount through the liquid, liquid flows through the aperture. The crystal can flow along with this liquid and be sieved out. However, as the aperture gets smaller, the flow speed through it gets much smaller. If you move the mount too quickly, the liquid won't have time to flow through the aperture. Your crystal may just flow around the aperture and end up on the neck of the mount.

The key to mounting very tiny crystals, then, is to **move the mount very slowly** toward the crystal, allowing enough time for the crystal to sediment through the liquid toward the mount, and for liquid to flow through the aperture. For the smallest crystals (<30 μm), consider using some kind of micromanipulator unless you have very steady hands, or else using a MicroMesh™.

Dehydration is a serious risk when working with very small crystals. Crystals smaller than 50 μm can dry out in seconds, degrading crystal diffraction. If you see diffraction spots that look like they come from salt, you know you've got a problem.

To minimize dehydration, we recommend:

- Using Mitegen's MicroMounts™, MicroLoops™ or MicroMeshes™
- Transferring your crystal to a drop of LV CryoOil before mounting
- Working in a humidified environment (a small steam vaporizer inside a plastic enclosure works nicely) or in a cold room

Mitegen's mounts allow you to easily and quickly mount and freeze small samples, minimizing the time for dehydration. Oils block evaporation during and after mounting. Evaporation rates are roughly proportional to the quantity $\Delta r.h. = 100\% - r.h.$, so that increasing the humidity from a typical laboratory value of 50% to 90% can reduce the evaporation rate by a factor of five. Evaporation rates plummet with decreasing temperature: the saturated vapor pressure of water at 4°C is 1/4 that at 25°C. If you've ever compared how quickly puddles evaporate in the summer and in the winter (on sunny days on equal side of the solstices), you know that evaporation rates vary strongly with temperature.

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