Minimizing Sample Motion in Cold Gas Streams

Sample motion in the cold gas stream can be problematic. Sample motion can broaden the apparent mosaicity and corrupt diffraction spot intensities so as to make indexing and refinement difficult.

How do you know that you have sample motion?

You may sometimes see your sample move on your video screen. Does this mean your sample is moving? Probably not. **Most apparent sample motion has the same origin as the twinkling of stars.**

As light rays from the star pass through the atmosphere, they are bent by refractive index variations caused by temperature and density variations along the light's path. The star then appears to be at a different position in the sky than it actually is. If the temperature and density fluctuate in time, so will the apparent position of the star, producing twinkling.

If the gas flow around your sample is perfectly laminar and steady, you'll see the sample at a fixed position. But if there is any turbulence or other nonsteadiness to the flow, the resulting temperature and density fluctuations will cause your sample's apparent position to fluctuate.

Note that if you turn off the gas stream or block it, you'll eliminate the nonsteady flow, the nonsteady refraction of light, and the apparent sample motion - **just like what you'd expect if the sample were actually moving in the gas stream!**

The safest and most reliable way to detect sample motion is via diffraction, which doesn't care about what the gas around the crystal is doing. Typical "flutter" motions occur on timescales of roughly 1 second. To detect them, collect several still frames (i.e., no rotation) in succession, using as short an exposure time as is feasible. Then compare intensities of the brighter spots from frame to frame. If the intensities change significantly from frame to frame, you have sample motion.

What causes sample motion? A partial list:

- Improper adjustment of the cold gas stream and the surrounding shield flow. With modern gas stream equipment, following the manufacturer's instructions generally gives the best results.
- Partial loss of vacuum (and thus thermal insulation) in the gas stream's flexible hose, or ice accumulation inside the flow tubes. These can cause fluctuating gas flows and gas temperatures, and may require larger flows to achieve adequate cooling.
- Improper centering of the gas stream on the sample, and improper distance between the gas stream outlet and the sample.
- For off-axis gas streams, orienting the sample so that the gas flows perpendicular to (rather than parallel to) the plane of the sample mount.
- Excessive drag around large samples encased in large amounts of liquid.
- Inadequate mount rigidity. Nylon loop mounts are prone to motion, especially if the twisted nylon necks are not encased in frozen water.

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Perhaps 90% of sample motions are due to turbulence associated with the first three items in this list. (But beware: the same turbulence will produce apparent (not actual) sample motion! Check your diffraction.) Aside from making sure that your gas stream hardware is functioning properly, is properly aligned, and has the correct flow speeds, what can you do if sample motion is a problem? Some suggestions:

- Reduce the amount of excess liquid around your sample, to reduce mass and drag.
- Mount a smaller sample.
- Mount your sample with an orientation that minimizes data collection when the gas stream is nearly perpendicular to the plane of the mount.
- Use Mitegen products, which have the greatest rigidity and smallest sample motion of commercially available mounts: Use a thicker sample mount, such as Dual-Thickness MicroMounts.
- Use a mount that creates less drag, such as Dual-Thickness MicroLoops LD.
- Leave a thin layer of liquid (mother liquor or oil) on the mount, including the neck region. When it freezes, it will increase the mount's rigidity.

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