Technical Note





Minimizing Ice Accumulation

Frosting - the formation of ice crystals near and on the sample - can be a nuisance during data collection. Frost generates ice rings in diffraction patterns. It can also perturb cryostream flows and increase turbulence, leading to more frosting and to sample motion.

Frosting is generally due to a combination of unsteady / turbulent gas flow near the sample and high ambient humidity. If gas flows are laminar and steady in the vicinity of the sample, you should get very little icing. But any turbulence creates eddies that can entrain moist air from the surroundings and condense it on your sample.

How can you reduce frosting/icing?

Adjust the cold gas stream and the surrounding shield flow to minimize turbulence. With modern gas stream equipment, following the manufacturer's instructions generally gives the best results. Older systems can be a bit tricky and can require some trial and error experimentation.

Make sure that the gas stream hose feels warm to the touch. Partial loss of vacuum (and thus thermal insulation) in the gas stream's flexible hose, or ice accumulation inside the flow tubes, can cause fluctuating gas flows and gas temperatures. They can also increase gas flows required to achieve a given stream temperature. Both will increase turbulence around the sample.

Make sure that the gas stream is properly centered on the sample, and that the distance from the gas stream tip to the sample is as recommended by the cold stream manufacturer. Generally, closer is better, but too close and the gas stream hits the goniometer base too forcefully and generates too much turbulence.

All of the ice that deposits on your sample is due to water vapor entrained from the surrounding air by turbulent vortices. In the summer when humidities are high, this can be a particular problem. Possible solutions:

- Run a dehumidifier or air conditioner in your experimental area.
- Keep the door to the hutch or experimental area closed.
- Turn off or block any fans or air flows that may disturb the air near your sample.
- Partially enclose the region near the sample to trap the nitrogen gas from the cryostream and keep out humid room air.
- Direct a very slow, large-diameter flow of dry nitrogen gas or air near your sample to ensure that any entrained air is dry. The flow should not perturb the cryostream flow.

Please contact <u>xtals@jenabioscience.com</u> with comments or suggestions.