Figure 10.1.4.3
Schematic drawing of a single-stream setup with the stream parallel to the diffractometer φ axis. The cold stream is represented by a grey region. The nozzle is heated above the dew point with a heating coil. The goniometer head is protected by a shield.

Figure 10.1.4.4
Schematic drawing of a single-stream setup with the stream angled relative to the diffractometer φ axis. The cold stream is represented by a grey region. The crystal mounting pin protrudes 1–2 mm into the cold stream. This prevents frost from forming on the mounting fibre. The nozzle is heated above the dew point with a heating coil. The cold stream misses the goniometer head, so no shield is required. In general, the simplest operation is attained with a setup similar to that shown here.

enough to prevent frost or condensation. The head-on direction results in reliable frost protection for the crystal, but requires that the goniometer head is equipped with a heated stream deflector.

Fig. 10.1.4.4 shows the functional equivalent of Fig. 10.1.4.2. This arrangement leads to the simplest design, although some precautions are needed. The mounting pin itself supplies the heat needed to prevent ice formation on the pin. The tip of the pin should be smooth in order to prevent turbulence. About 1–2 mm (but not more) of the tip must protrude into the cold stream. If too much of the pin is in the cold stream, the rest of the pin can become too cold and ice up. If the pin is too far out of the stream frost prevention will also fail, because glass or other insulating mounting materials will invariably collect ice at the cold/warm interface. As frost prevention depends on heat conducted from the rest of the pin, it must be made from copper (a requirement not strictly necessary for the dual-stream design). This design has been extensively tested and has been used for many years for the collection of a large number of data sets. Results are uniformly good, with simple operation and reliable frost prevention even in high humidity.

Fig. 10.1.4.5 is a photograph of a laboratory implementation of the setup of Fig. 10.1.4.4, with helium as the coolant. Operation is as simple as with nitrogen cooling.

Omission of the warm stream results in significant design simplification. The entire apparatus for production of the outer stream is left out, resulting in real savings in manufacture, operation and maintenance. There is no obvious disadvantage, as ice protection is as good as with the dual stream. The main cost to the user is in the requirement that the mounting system be constructed within somewhat narrower limits, including the requirement of a copper-shafted mounting pin. The fact that operator errors tend to become apparent through frost formation can actually be an advantage. With a dual-stream device, an improperly positioned cold stream or an improperly prepared crystal mount may not produce overt signs, even though the crystal temperature is ill-defined.

In all configurations shown, correct positioning of the cold stream is essential. The centre of the stream should not miss the centre of the diffractometer (and hence the crystal) by more than 0.5 mm.

With helium, the single-stream technique represents the best solution and reliable frost prevention at temperatures down to around 5 K is easily attained. Crystal mounting is simple and the sample is always visible, simplifying centring. In the authors' experience, liquid-helium cooling is as simple as liquid-nitrogen cooling.

10.1.4.3. Temperature calibration

Measurement of the temperature at the crystal site with sensing devices that require attached leads is very difficult, mainly because of heat conduction along the leads. It is usually necessary to loop the leads into the delivery nozzle.

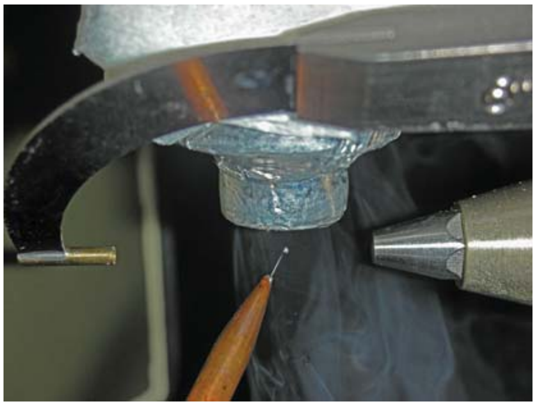

Figure 10.1.4.5
A crystal in a helium stream at 8 K in a setup corresponding to Fig. 10.1.4.4. A thin layer of fog forms at the helium–air interface. The cold stream breaks up well below the crystal position. Rising cold gas (visible as fog to the right of the cold stream) has been mixed with air to prevent cooling of diffractometer parts. The nozzle temperature is 295 K. The crystal is attached to a tapered glass fibre. The light-coloured region at top is not ice; it is part of the insulation. This photograph was taken after the end of data collection.