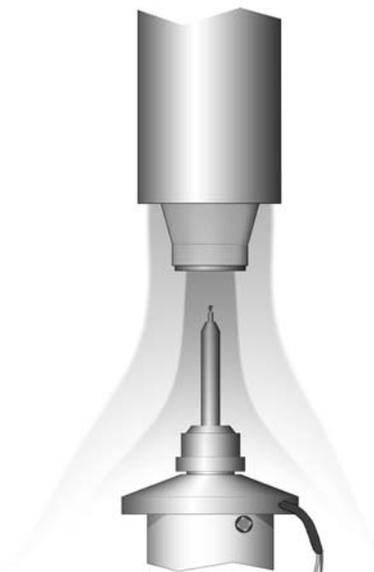


10.1. INTRODUCTION TO CRYOCRYSTALLOGRAPHY

**Figure 10.1.4.1**

Schematic drawing of a dual-stream setup with the streams parallel to the diffractometer φ axis. The top part represents the outlet end of the stream delivery device. The outer stream (lighter grey) is dry, warm air. The goniometer head is protected by a shield.

For a given setting of the flow control valve, the flow rate depends on the tank pressure. For a constant temperature, a constant pressure is required. The pressure is controlled with a pressure regulator that can reduce the tank pressure by releasing helium gas or raise it by adding helium gas from an external helium supply source. A typical delivery tank pressure is around 20 kPa. It is very important that the pressure is constant. The precise value attained is less important.

At atmospheric pressure, helium at around 40 K has the same density as room-temperature air. This means that very cold helium will rise in air, and has the capacity to seriously cool instrument parts in its way. Goniometer heads, beam stops and beam collimators are particularly vulnerable. A simple remedy is to use a small fan to mix the cold helium with room air.

10.1.3.3. Frost prevention

Three areas must be kept frost-free: the crystal, the crystal mount and the delivery end of the transfer tube. The first successful solution to this problem was the dual-stream design of Post *et al.* (1951). It provides for a cold stream surrounded by a concentric warm stream. If the warm stream is sufficiently dry, this will prevent frost around the outlet. The crystal will remain frost-free only if mixing of the two flows occurs downstream from the crystal. For a stream aligned with the axis of the goniometer head, an additional shield is needed to keep the goniometer head frost-free.

10.1.4. Operational considerations**10.1.4.1. Dual-stream instruments**

Fig. 10.1.4.1 shows a schematic drawing of the region around the crystal in a traditional dual-stream apparatus, first described by Post *et al.* (1951). The device provides for a cold stream surrounded by a concentric warm stream. The diameter of the cold stream is typically around 7 mm with a shield stream of 2–3 mm. The two streams flow parallel to the axis of the crystal mount. In a properly functioning apparatus, the warm stream

**Figure 10.1.4.2**

Schematic drawing of a dual-stream setup with the streams angled relative to the diffractometer φ axis. Stream representations are the same as in Fig. 10.1.4.1. The cold stream misses the goniometer head, so no shield is required.

supplies enough heat to keep the tip of the cold-stream tube above the dew point. It is important that the streams do not mix, or the crystal temperature will not be stable. This is achieved by careful balancing of flow rates to minimize turbulence. (Absence of turbulence can be judged by the shape of the shadow of the cold stream in a parallel beam of bright light.) In a laminar cold stream, the crystal is well protected and no special precautions are needed. The region of constant, minimum temperature will typically have a diameter of about 3 mm. Turbulent flow will result in the absence of any constant-temperature region, so it is vitally important to verify the stream quality.

The cold stream has sufficient heat capacity to cool down the goniometer head, and sometimes other adjacent equipment parts as well. A simple solution consists of an aluminium cone equipped with a heating coil on the back. A shield that functions well has been described by Bellamy *et al.* (1994).

Fig. 10.1.4.2 illustrates a situation where the stream direction deviates substantially from the head-on direction in Fig. 10.1.4.1. An angle of 35–55° will give good results. An advantage of an angled delivery is that the cold stream will not touch the goniometer head, and therefore the heated stream deflector is not needed, resulting in simplified installation and operation.

Analysis of the dual-stream apparatus reveals a twofold function of the outer stream: it keeps the nozzle frost-free and it supplies heat to the mounting pin. Protection of the crystal is, in reality, already provided by the laminar cold stream. The nozzle can be kept frost-free simply with an electric heater. Ice formation on the crystal mount can be easily suppressed by appropriate design of the mounting pin and mounting fibre, and attention to their interaction with the cold stream. A successful solution is sketched in Figs. 10.1.4.3 and 10.1.4.4.

10.1.4.2. Electrically heated nozzle

Fig. 10.1.4.3 shows the functional equivalent of Fig. 10.1.4.1. Instead of the warm stream, an electrical heating element is used to keep the tip of the delivery tube ice free. An actual construction will usually consist of a nozzle that can be attached to the delivery tube. The heating element is made from standard resistance wire (*e.g.* Nichrome). About 5 W will usually be