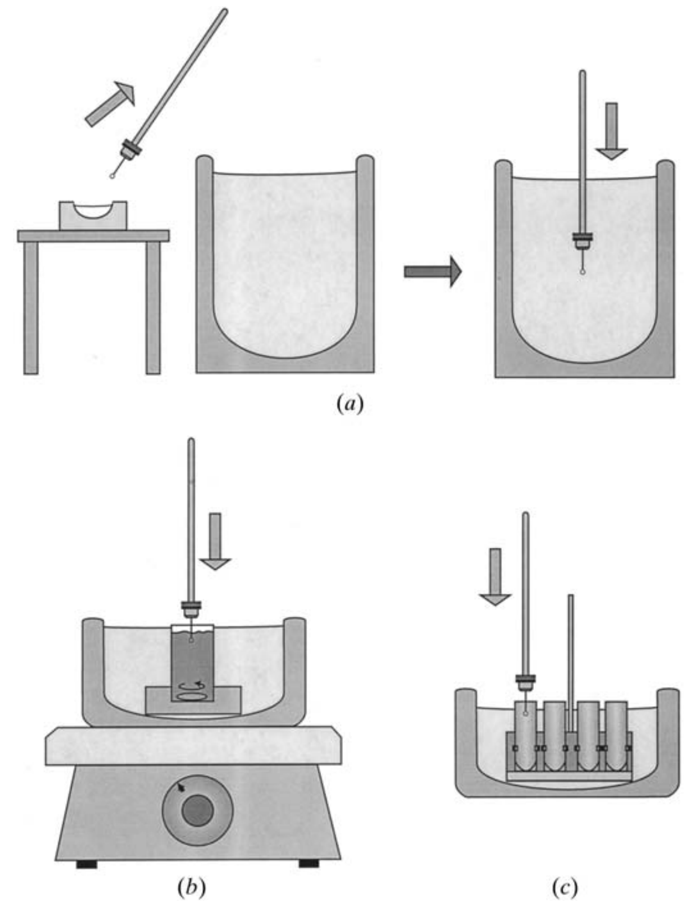

Figure 10.2.4.2
Flash cooling in a liquid cryogen. (*a*) Cooling in liquid nitrogen. The loop assembly is attached, *via* a magnet mount, to a short rod and the crystal is captured. It is then quickly plunged into a nearby Dewar of liquid nitrogen. (*b*) One method of flash cooling in a liquid cryogen such as propane. The cryogen is placed in a weighted container, which itself stands in a Dewar of liquid nitrogen. The Dewar rests on a stir plate, which mixes the liquid cryogen to ensure a uniform temperature. When the temperature of the cryogen is just above its melting point, the loop assembly is plunged into the liquid. (*c*) A variation on cooling in propane or a similar cryogen. The cryogen is placed into small plastic vials designed for cryogenic storage. Just before the cryogen freezes, the loop assembly is plunged directly into a vial. A holder for the vials allows multiple samples to be prepared sequentially.

gas around the crystal. To minimize drying of the sample during transfer, the crystal container and the Dewar are located as close as possible. If necessary, drying can be further reduced by using a portable humidifier to add moisture in the area.

Other cryogens, such as propane, can be tested if results with liquid nitrogen are not satisfactory. Two methods for flash cooling in these other cryogens are illustrated in Figs. 10.2.4.2(*b*) and (*c*). In the first (Fig. 10.2.4.2*b*), the liquid cryogen is held in a small container with a weighted base, which is placed in a Dewar of liquid nitrogen to cool the cryogen. The cryogenic liquid is mixed using a magnetic stir bar to ensure a uniform temperature throughout the sample. Since the boiling points of these cryogens are well above their melting points, it is possible in the absence of stirring to have relatively warm, and therefore less effective, cryogen near the top of the container. When a temperature probe indicates that the cryogen is just above its melting point, the crystal is mounted and plunged quickly into the liquid. A variant of this technique (Fig. 10.2.4.2*c*) calls for plunging the loop assembly directly into cryogen-filled plastic vials, which are used for low-temperature transfer and storage of the crystals (see Section 10.2.5). The cryogen is then allowed to solidify around the crystal before it is placed on the X-ray camera or stored for later use. With this technique, it is more difficult to ensure that the temperature of the cryogen is uniform throughout the container. Other mechanisms for flash cooling in liquid cryogens have been described (Hope *et al.*, 1989; Abdel-Meguid *et al.*, 1996), and devices for combining xenon derivatization with flash cooling (Soltis *et al.*, 1997) are available commercially.

10.2.5. Transfer and storage

Crystals flash cooled in a liquid cryogen must be placed for data collection in the cold gas stream of a cryostat without any substantial warming. One common transfer method (Rodgers, 1994, 1997) is shown in Fig. 10.2.5.1. Once the loop assembly has been plunged into the Dewar of liquid nitrogen, it is inserted into a small plastic vial of the type normally used for cryogenic storage, ensuring that the sample remains below the liquid surface during the operation. There is then sufficient liquid nitrogen in the vial to keep the sample cold as it is transferred to the cryostat gas stream. Again, the magnetic mounting system is used to reduce the time required for transfer. For X-ray cameras with vertical spindles, as shown in Fig. 10.2.5.1, some means of pointing the magnetic mount downward is required to prevent the nitrogen from spilling out of the vial. The goniometer illustrated has a detachable arc extension (Engel *et al.*, 1996; Litt *et al.*, 1998) that provides this capability. When the loop assembly is attached to the magnet, the vial is quickly withdrawn, exposing the crystal to the gas stream. The arc slide can then be returned to the normal position and the arc extension removed.

Another device (Mancia *et al.*, 1995) for achieving the correct transfer geometry is shown in Fig. 10.2.5.2. This 'flipper' mechanism can be extended to permit transfer of the crystal. The device is then rotated about the hinge to reorient the loop assembly for data collection. The

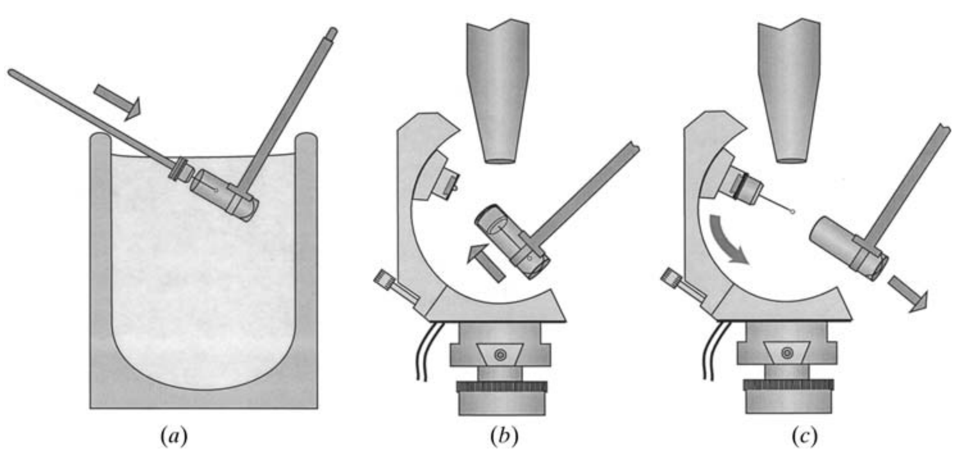

Figure 10.2.5.1
Transfer of the flash-cooled crystal and loop assembly to a goniometer using a cryovial. (*a*) The loop assembly with a flash-frozen crystal is placed in the vial, which is held by a rod-shaped tool. The operation is carried out beneath the surface of the liquid nitrogen in a Dewar. (*b*) The loop assembly is transferred to the goniometer using the magnetic mounting system. (*c*) The vial is withdrawn, exposing the crystal to the cold gas stream. With this arrangement of goniometer and cryosystem nozzle, it is necessary to use a device that allows the magnetic mount to point downward. Here, a detachable arc extension provides this ability. After crystal transfer, the arc slide can be returned to the normal position and the extension removed.

references