Table 3.4.1.1. *Single-crystal and powder mounting, capillary tubes and other containers*

Material	Temperature range (K)	Comments
(*A*) Capillary tubes		
Glass Lindemann glass Vitreous silica	< 773 < 773 < 1373	Lindemann glass scatters less, but is moisture sensitive Thinner walled tubes that are less sensitive to atmospheric influences can be obtained using other types of glass
Collodion Polyvinyl methylal resin (*e.g.* Formvar) Cellulose acetate	93 to 343 < 323 < 373	These capillaries can be made by coating a copper wire with a solution of the polymer in an appropriate organic solvent. When dry, the metal core may be removed by stretching, to reduce its diameter
Polyethylene	< 373	Tubes may be drawn from the molten polymer using a glass tube and a slow stream of air. The polymer gives a distinct diffraction pattern
(B) Other containers		
Gelatin capsules	< 303	Vessels with very thin, 20 μm, windows can be made
Methyl methacrylate resin (*e.g.* Perspex)	< 338	
Mica	< 1073	Mica windows useful in vessels for small-angle scattering, but the wall size is generally thicker, ∼0.3 mm, and there are discrete lines at 10.00, 3.34 and 2.60 Å in the diffraction pattern
Regenerated cellulose film (*e.g.* cellophane)	Ambient	

For optimum results, tube diameters should be between 0.3 and 0.5 mm with wall thicknesses of 0.02 to 0.05 mm. The materials listed above, except where stated, give diffuse diffraction patterns. If necessary, control diffraction patterns, recorded only from the capillary or other container, should be taken.

(1993) have developed a mirror furnace working at up to 2300 K and suitable for polycrystalline or single-crystal samples.

A comprehensive account of cryogenic studies pertinent to both polycrystalline and single-crystal samples is given by Rudman (1976). Nieman, Evans, Heal & Powell (1984) have described a device for the preparation of low-temperature samples of noxious materials. The device is enclosed in a vanadium can and is therefore only suitable for neutron diffraction studies. Ihringer & Küster (1993) have described a cryostat for powder diffraction, temperature range 8–300 K, for use on a synchrotron-radiation beam line at HASYLAB, Germany (Arnold *et al.*, 1989).

3.4.1.3. *Single crystals (small molecules)*

3.4.1.3.1. *General*

Small single crystals of inorganic and organic materials, suitable for intensity data collection, are normally glued to the end of a glass or vitreous silica fibre, or capillary (Denne, 1971*b*; Stout & Jensen, 1968). A simple device that fits onto a conventional microscope stage to facilitate the procedure of cementing a single crystal to a glass fibre has been constructed by Bretherton & Kennard (1976). The support is in turn fixed to a metal pin that fits onto a goniometer head. For preliminary studies, plasticine or wax are useful fixatives, since it is then relatively easy to alter the orientation of the support, and hence the crystal, as required. For data-collection purposes, the support should be firmly fixed or glued to the goniometer head pin. The fibre should be sufficiently thin to minimize absorption effects but thick enough to form a rigid support. The length of the fibre is usually about 10 mm. Kennard (1994) has described a macroscope that allows specimens to be observed remotely during data collection and can also be used for measurement of crystal faces for absorption correction. Large specimens can be directly mounted onto a camera or onto a specially designed goniometer (Denne, 1971*a*; Shaham, 1982). A method using high-temperature diffusion to bond ductile single crystals to a metal backing, for strain-free mounting, has been described by Black, Burdette & Early (1986).

Prior to crystal mounting, it is always prudent to determine the nature of any spatial constraints that are applicable for the proposed experiment. Some diffractometers have relatively little translational flexibility, and the length of the fibre mount or capillary is critical. For some low-temperature devices where the cooling gas stream is coaxial with the specimen mount, the

references