Laboratory Notes


Preparation of capillary mounts in a dry box

Lange & Haendler (1972) have recently described an apparatus to facilitate the handling of fragile glass capillary tubes, in a dry box or glove bag, while they are being loaded for X-ray diffraction analysis. An alternative method makes use of the simple accessory shown in Fig. 1. By this method, the sample mounting is completed under a controlled atmosphere in that the sample is sealed in the spindle and ready for X-ray analysis before it is removed from the dry box.

The capillary tubes containing samples to be analysed by the Debye–Scherrer method are held in brass tubes (spindles) 10 to 12 mm long, 1 mm in ID, and 3 mm in OD. To prepare a spindle to receive the capillary tube it is aligned with a longer piece of similar brass tubing and held in place by a short length of plastic tubing: flame-seal insulation stripped from solid 12-gauge copper wire is suitable for this purpose. The combined length of the two pieces of brass tubing is such that while the capillary is supported therein, its sealed end projects about 4 mm from the end of the spindle.

A drop of cement is applied around the capillary and is shaken down to the sealed end by flicking the top of the spindle. With the brass tubing held in one hand, the spindle is grasped with pliers at its lower end and snapped out of the plastic tubing with a sharp wrist movement to break the capillary even with the top of the spindle. Finally, the open end of the spindle is sealed with a drop of cement.

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Reference


A simple two-circle goniostat for use with a high-power microscope

A simple goniostat has been constructed which is useful in making measurements of the faces of small polyhedral crystals under a microscope for X-ray absorption corrections. The required accuracy can be achieved if each face is carefully measured in turn with a high-power microscope and a graduated eyepiece; the faces may then be 'assembled' mathematically to produce the best-fit polyhedron, from which can be derived the indices of the faces, crystal volume, etc. (Denne, 1973). This procedure requires a crystal-orienting device which is rather small, as the space around a high-power microscope objective lens is very restricted, and which is effectively eucentric, as the working distance to the microscope objective lens is small and the crystal is otherwise liable to be dislodged during adjustments of its orientation.

A simple design of two-circle goniostat which satisfies these requirements has been developed for crystals mounted for the Nonius conical goniometer head (Denne, 1971). The device is illustrated in Fig. 1. If the standard microscope stage is removed, plate 1 can be bolted horizontally to the bracket on which the microscope stage was supported. The L-shaped plate 2, is bolted to plate 1 with a screw which has a countersunk head. This provides a simple bearing with a smooth action and very little play (less than 5 pm in the present unit). Small rotations of plate 2 effectively provide a translational adjustment of the crystal to centre it under the microscope. Plate 3 is bolted to plate 2 in a similar fashion and this provides a translational adjustment at right angles to the first. The tightness of the countersunk screws may be varied so that the desired amount of friction is provided. The screws may be locked with locknuts.

Angular adjustment is achieved by rotation of the component labelled 4 and of the mounting rod, 6, itself. The component, 4, consists essentially of a block with a V-groove and an \( \frac{3}{4} \) inch rod, 5, screwed in at right angles to it. The groove is designed to take the mounting rod which may be slid up or down till the crystal, which lies close to the mounting-rod axis, also lies on the axis of the \( \frac{3}{4} \) inch rod perpendicular to it. The device is then eucentric to a good approximation. The mounting rod can be rotated with the tommy bar normally used for conical goniometer-head adjustment and the member 4 can be rotated about its brass rod which locates in a V-groove cut in plate 3. Phosphor bronze shim spring clips hold both rods in their respective V-grooves and provide sufficient friction to clamp the adjustments when not in use. While this device is not intended for measurement of the goniometer settings, some numerical assessment could be made from helical scales such as that engraved on the conical goniometer-head mounting rod.

The device has been in use for some time and as yet no crystals have been lost even though the working distance of the microscope is of the order of 1 mm. The five adjustments (3 linear and 2 angular) have been found quite adequate for work at \( \times 200 \) magnification using a standard Vickers polarizing mic-