Laboratory Notes

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Rapid and precise method for preserving orientation in crystal transfer

The crystal to be studied is mounted on a goniometer head with modelling clay and aligned by one of the various X-ray techniques. For crystals less than 1 cm on edge the precession method is used, while back-reflection methods are needed for larger specimens. Once the sample is satisfactorily aligned the goniometer head is removed from the X-ray camera and screwed on a mounting with a $\frac{3}{8}$ inch diameter shaft (detail *A* in Fig. 1). The mounting together with the goniometer head and crystal, is inserted into the chuck of a drill press.

The crystal is now placed within the cavity of a cylindrical receiver (detail *B* in Fig. 1). This is a solid metal cylinder of brass, steel, *etc.* with a conical depression in the center of one end. The dimensions are not critical, but are typically about 1 inch in diameter and 1 inch high with a conical depression of $\frac{1}{4}$ inch deep with a semivertex angle of 45°. The depression is filled with molten wax; glycol phthalate, which softens at about 140°C, is suitable. The cylinder is placed on the drill-press table directly under the crystal. The crystal is lowered into the hot wax and locked down. The wax hardens in a matter of minutes as the cylinder is a good heat sink. The drill press is then raised, separating the crystal from the goniometer head. The crystal can now be cut or polished to give a face perpendicular to the desired direction. X-ray methods reveal that such faces are within about 1° of the desired orientation.

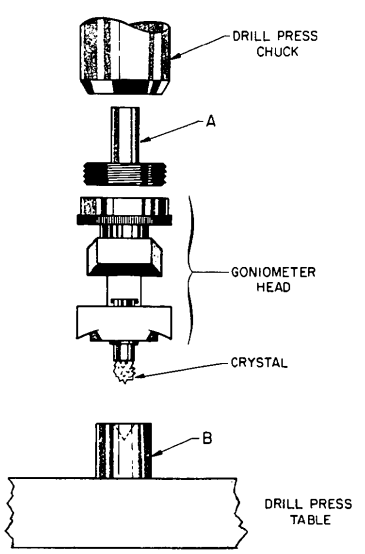

Fig. 1. Crystal-transfer apparatus.

The method is routinely employed to cut parallel faces in crystals for measurement of pyroelectric and piezoelectric coefficients. Crystals as small as 2 mm on edge have been successfully handled by this technique.

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A method for preventing crystal slippage in macromolecular crystallography

A new method for preventing crystal slippage in macromolecular single crystal X-ray diffraction is described which involves enclosing the crystal and its associated mother liquor with a very thin plastic film.

Crystal slippage during data collection has occasionally been an enigma in macromolecular single-crystal X-ray diffraction. In studies of southern bean mosaic virus type II rhombohedral crystals (Akimoto, Wagner, Johnson & Rossmann, 1975) slippage was a major problem in collecting the SBMV 3·5 Å resolution data until the procedure described here was adopted. The problem was accentuated as it was necessary to leave the crystals very wet in order to preserve diffraction to high resolution. In the case of satellite tobacco necrosis virus this problem was solved by growing the crystals in thin-walled capillaries until they were completely wedged between the walls (Åkervall & Strandberg, 1971).

Our solution to the slippage problem consists of enclosing the crystal and its associated mother liquor with a very thin plastic film after mounting. The plastic film was prepared *in situ* with a 0·2% solution of Poly(Vinyl Formal) 15/95 powder (Polyscience Inc., Warrington, Pa. 18976, USA) in 1,2-dichloroethane. This mixture, which is immiscible with water, will spread over an aqueous surface and leave a very thin plastic film when the solvent carrier evaporates.

The SBMV crystals were grown in microdialysis cells and mounted in the conventional manner by drawing the crystal into a thin-walled quartz capillary. The excess mother liquor was removed with either a thin glass rod or a narrow piece of filter paper. The crystal and its accompanying mother liquor were then coated with a thin film of plastic by drawing a short column (~1 mm) of the Poly(Vinyl Formal) solution over the crystal and subsequently allowing the solvent to dry for a few minutes. A small column of mother liquor was then drawn into the capillary which was sealed in the normal manner.

Crystals mounted by this procedure could be used immediately without observing any crystal slippage, whereas previously the crystals had to stand for at least 24 h before they could be used. There was also no detectable alteration in the background level or the diffraction pattern observed on the photograph.

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Crystallographers

This section is intended to be a series of short paragraphs dealing with the activities of crystallographers, such as their changes of position, promotions, assumption of significant new duties, honours, etc. Items for inclusion, subject to the approval of the Editorial Board, should be sent to the Executive Secretary of the International Union of Crystallography (J. N. King, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England).

Walter L. Bond, long a member of the American Crystallographic Association, died on 30 March 1977, after a second heart operation. He was widely known for his ingenuity and skill in designing and personally constructing a variety of in-