The X-ray photography of volatile or deliquescent single crystals.

By F. J. Llewellyn, *Department of Chemistry, Auckland University College, Auckland, New Zealand*

In the crystal-structure determination of ammonium trinitrate by Duke & Llewellyn (1950) considerable difficulty was experienced in handling and preserving a single crystal. This substance is extremely hygroscopic and even when kept in dry air decomposes by loss of nitric acid vapour. Single crystals can, however, be preserved for long periods in closed capsules containing a desiccating agent together with a quantity of ammonium trinitrate which ensures an equilibrium nitric acid vapour pressure.

In the capsule prepared by Duke & Llewellyn (1950) polythene was heated in an atmosphere of nitrogen and cast on to a cylindrical mandril which had previously been turned between centres on a precision lathe. When cold the mandril was reset in the lathe and the polythene coating turned down to wall thickness of 0·3 mm. Ends suitable for handling were left on this sheath and these were stopped with machined perspex plugs; one carried the crystal mounted on a fibre, the desiccant and the ammonium trinitrate, and the other was fitted with a glass window. Polythene was the only plastic available which was not attacked by nitric acid vapour, and, since it is not transparent, special arrangements had to be made for viewing and setting the crystal.

More recently, in examining a number of aliphatic nitro bodies, similar precautions have become necessary because of volatility of the specimen. Capsules of perspex, with wall thickness 0·15 mm., have been made and these have proved entirely satisfactory. A brass mandril is turned accurately parallel between centres; a perspex rod of greater diameter, bored and reamed to 0·02 mm. less than the mandril diameter, is then pushed on to the cooled mandril. After turning the perspex sheath to the desired wall thickness the mandril is removed by solution in dilute nitric acid.

Sheaths of 0·15 mm. wall thickness are robust and are not easily damaged by normal handling. The absorption coefficient for Cu $K\alpha$ X-radiation is extremely small $(I/I_0 \doteqdot 0·90)$ and, moreover, optical goniometry of a single crystal mounted inside is quite practicable. Suitable ends are left on the sheath to facilitate the fixing of machined plugs and the whole is made air-tight by covering the plug–sheath joints with Scotch tape. When care is taken in mounting the crystal, so that the deviation of the axis of the sheath from the axis of rotation of the crystal does not exceed 3°, absorption in the sheath can be calculated and appropriate corrections made to the observed intensities of the diffracted beams.

The following is a specification of a sheath which has proved satisfactory in Weissenberg photography:

Length	40 mm.
Internal diameter ...	5 mm.
Wall thickness ...	0·15 mm.
Outside diameter of ends	10 mm.

Reference

Duke, J. R. C. & Llewellyn, F. J. (1950). *Acta Cryst.* **3**, 305.

A simple method of applying the rotation factor correction in equi-inclination Weissenberg photographs.

By F. H. Herbstein, *Department of X-ray Crystallography, The Weizmann Institute of Science, Rehovoth, Israel*

We should like to draw attention to the simple form which the expression for the rotation factor for the Weissenberg equi-inclination method takes when expressed in cylindrical co-ordinates ξ, ζ and ρ.

Tunell (1939) gives the following expression for this factor D when $\mu = -\nu$:

$$D = \frac{(\cos^2 \mu - \cos^2 \theta)^{\frac{1}{2}}}{\sin \theta}. \tag{1}$$

Now $\quad \sin \mu = \frac{1}{2}\zeta$, whence $\cos^2 \mu = 1 - \frac{1}{4}\zeta^2$.

Also $\quad \sin \theta = \frac{1}{2}(\xi^2 + \zeta^2)^{\frac{1}{2}}$, whence $\cos^2 \theta = 1 - \frac{1}{4}(\xi^2 + \zeta^2)$.

On substituting these values in the expression above we obtain $D = \xi/\rho$.

The device described by Goldschmidt & Pitt (1948) provides a convenient means of determining the rotation factor at the same time as the other correction factors. In the instrument described (see Fig. 1, where the case of rotation about the *b* axis of a monoclinic crystal is illustrated), ξ is the distance from the origin to P, while $\rho = OP$. Now $\xi/\rho = \sin \phi$, and so D may be read on a suitably graduated scale placed at O. For the case of rotation about a non-orthogonal axis, it is necessary to allow for the shift of origin of the *n*-levels of the reciprocal lattice but equation (1) still holds.
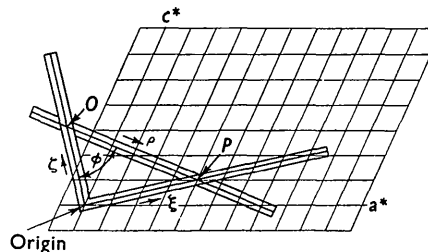

Fig. 1.

References

Goldschmidt, G. H. & Pitt, G. J. (1948). *J. Sci. Instrum.* **25**, 397.

Tunell, G. (1939). *Amer. Min.* **24**, 448.