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Powder diffraction patterns from microsamples.

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The conventional powder-diffraction techniques usually require about 1 mg. of sample for satisfactory diffraction patterns. Although microcameras have been described (Kratky, 1930 *a*, *b*; Fankuchen & Mark, 1943; Kreger, 1946 *a*, *b*) which can be employed for samples available only in microgram amounts, the mounting of such powder specimens on the axis of a 0·01–0·02 mm. pinhole collimator becomes difficult and almost precludes the rotation of the sample in the X-ray beam in order to reduce the spottiness of the micropattern. Glass fibers and thin-walled pyrex capillaries have been used successfully by Zachariasen (1949) to obtain satisfactory X-ray diffraction patterns with a few micrograms of heavy-metal compounds. However, the presence of a relatively large amount of glass (as compared with the weight of the microsample) has two disadvantages: (1) for soft radiation such as Cu $K\alpha$, or Cr $K\alpha$ the scattering from the glass raises materially the background 'fogging', and (2) the spreading of the sample over the inside wall of the capillary reduces the potential sharpness of the powder reflections and can produce 'absorption doublets' for low values of the Bragg angle.

A simple and effective mounting has been developed which is capable of handling powder samples weighing 0·001 mg. This technique consists in coating the 2 mm. knife edge of a microwedge of lead with a thin film of mineral oil which permits one to pick up very tiny specks and have them bathed in a very narrow beam of X-rays. Fig. 1 depicts the design of the lead microwedge and holder. A 20° oscillation of the wedge provides random orientation to the tiny crystallites and thus reduces the spottiness of the powder pattern. The sharp diffraction lines from the lead of the wedge are useful for an accurate calibration of the effective camera radius and cause no serious complication in the phase identification of unknown mixtures. Any corrosion-resistant material with a high absorption coefficient may be substituted for lead; e.g. tantalum, platinum, gold, lead glass, etc. To reduce the air scatter for prolonged exposures it has been found expedient to displace the air in the camera and slit system with helium. Fig. 2 reproduces two diffraction patterns of $NH_4H_2PO_4$ taken with an equatorial shutter (Frevel, 1935), and illustrates the reduction of background scatter for a helium atmosphere.

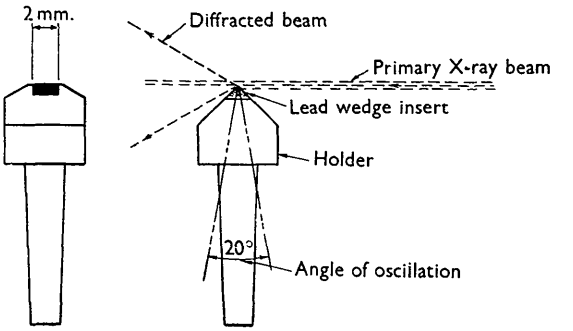

Fig. 1.

References

FANKUCHEN, I. & MARK, H. (1943). *Record Chem. Progress*, **4**, 54.
FREVEL, L. K. (1935). *Rev. Sci. Instrum.* **6**, 214.
KRATKY, O. (1930*a*). *Z. Krystallogr.* **76**, 261.
KRATKY, O. (1930*b*). *Z. Krystallogr.* **76**, 517.
KREGER, D. (1946*a*). *Proc. Acad. Sci. Amst.* **48**, 336.
KREGER, D. (1946*b*). *Nature, Lond.*, **158**, 199.
ZACHARIASEN, W. H. (1949). *Record Chem. Progress*, **10**, 47.

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A circular slide rule for structure-factor calculations.

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We wish to describe a simple mechanical device for the rapid computation of the function cos (sin) $2\pi(hx+ky+lz)$ directly from a given set of x, y, z for positive or negative values of h, k, l.

The device is basically a circular slide rule with two concentric scales A and B: A is the scale of x (y, z), i.e. 2π radians divided into 1000 intervals; B is a cosine (sine) scale graduated over 2π in intervals of 0·05, except in the region of 0·95–1·00 where the intervals are in units of 0·01. The two scales have a common origin at the points 0·000 and 1·00 respectively.

Above the fixed plate carrying these two scales, and mounted concentrically, runs a cursor consisting of a plate of 'perspex', which has engraved on it two mutually perpendicular diameters. It is thus possible to read off, at one setting of the cursor, $\pm$cos (sin) $2\pi\theta$ for a given angle θ, from the appropriate one of the four radius vectors.

The innovation of the slide rule consists in the movement of the cursor: the radius vector can be advanced repetitively through a given angle x. Once this angle x has been set, the function cos (sin) $2\pi hx$ can be read off without the necessity of first computing hx.

In practice, this angle x is set on the movable stop X which runs along another scale C, also graduated in millicycles. Scale C can be turned about the centre of the slide rule until the angle stop X hits the fixed zero stop O, which is attached to the base of the instrument and does not move with C. A ratchet movement takes the perspex cursor along through the angle x on the forward journey of scale C, but disengages on the return of C to O. A second motion of C through x now advances the cursor

Sample: $NH_4H_2PO_4$, $0{\cdot}010 \pm 0{\cdot}01$ mg.
Radiation: filtered Cu $K\alpha$.
Camera radius: 71·7 mm.
Pinhole system: 0·76 mm. diam. × 70 mm.
Composite pattern taken with equatorial shutter.

Sample: $NH_4H_2PO_4$. 0·001 mg.
Radiation: filtered Cu $K\alpha$ (20 mm. 30 kV. 12 hr.).
Camera radius: 71·7 mm.
Pinhole system: 0·76 mm. diam. × 70 mm.
Marked lines refer to lead pattern.

Fig. 2.

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