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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.

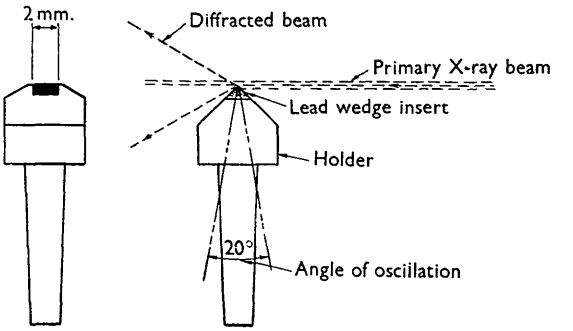

Fig. 1.

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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.

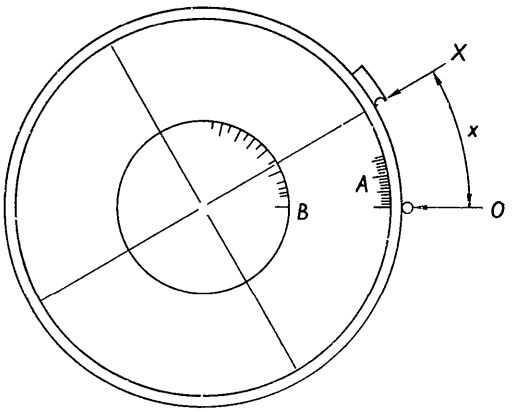
The device is basically a circular slide rule with two concentric scales A and B: A is the scale of x (y, z), i.e. 2π radians divided into 1000 intervals; B is a cosine (sine) scale graduated over 2π in intervals of 0·05, except in the region of 0·95–1·00 where the intervals are in units of 0·01. The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

to $2x$, and so on. A simple clutch enables the operator to reverse the direction of motion of the cursor so that cos $2\pi(-h)\,x$ can be obtained from the same angle setting.



The ratchet mechanism for moving the cursor consists of a hardened polished C-shaped steel double pawl pivoted in a recess in the operating and stop-carrying ring, and is sprung with two leaf springs. Either of the directionally opposed pawl faces can be brought into position to engage with the ratchet teeth of the cursor, to transport it in either direction, by shifting the pressure of the leaf springs on the pawl by means of two knurled knobs which control the spring positions.

The contribution of an atom i to the geometrical structure factor of the form cos $2\pi(hx_i \pm lz_i)$ is computed along lines in the reciprocal lattice, say for constant l, by setting the angle stop to x, advancing the cursor to lz, and successively determining cos $2\pi(hx_i+lz_i)$ from 0 to h, and from 0 to $-h$.

An obvious improvement is the inclusion of a second and third angle stop to hold the values of y and z. If such stops Y and Z are to be added, then X, Y and Z must be capable of being swung out so as not to interfere with one another.

In its present form the device has been found fully accurate and reliable in the summation of $(hx+lz)$. It has been most useful in the (three-dimensional) analysis of phenazine and the (two-dimensional) analysis of tetrabenznaphthalene (7 and 26 atoms in the asymmetric unit respectively), which are now being carried out in this laboratory. Two hundred F's of the form cos $2\pi(hx_i+lz_i)$ of 26 atoms have been computed in 16 working hours.

It is a pleasure to thank the chief instrument maker of the Weizmann Institute, Mr B. Feldmann, for designing and constructing this instrument.

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An orthorhombic variety of chrysotile.

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The structure of chrysotile asbestos has been studied by Warren & Bragg (1930), Gruner (1937) and Aruja (1943). Although the detailed structural conclusions of these workers have differed, they have all agreed that the structure is based on a monoclinic cell having the approximate parameters

$$a=14{\cdot}6,\quad b=9{\cdot}2,\quad c=5{\cdot}32\text{ A.},\quad \beta=93^\circ\,12',$$

or simple multiples or submultiples of these a and b axial lengths.

It appears from the literature that these results were all obtained using specimens from Thetford, Quebec. More recently Padurow (1950) has claimed that the structure is only pseudo-monoclinic and is really triclinic with cell parameters

$$a=7{\cdot}36,\quad b=9{\cdot}26,\quad c=5{\cdot}33\text{ A.},$$
$$\alpha=92^\circ\,50',\quad \beta=93^\circ\,11',\quad \gamma=89^\circ\,50'.$$

The chrysotile used in this work was also from Quebec.

As a result of a survey of specimens from a variety of sources in Canada, Rhodesia, Swaziland, India and Australia the author has found that the diffraction patterns obtained differ quite extensively. Certain of these differences have already been reported (Whittaker, 1949), but at the time the nature of the corresponding structural differences had not been ascertained. It has now been found that the photographs may be explained on the assumption that the specimens consist of mixtures, in different proportions, of the normal monoclinic variety (or pseudo-monoclinic according to Padurow) and a new orthorhombic variety with substantially identical unit-cell dimensions. Chrysotile from Canadian sources does not appear to contain any of the orthorhombic variety, which accounts for this not having been observed by previous workers. The other sources mentioned yield material of varying ortho content both as between sources and as between different specimens from the same source. This content varies from zero up to a value which is still uncertain, but is probably more than 50%. No evidence has been found of fluctuation in this proportion when a specimen is repeatedly halved in cross-section down to a diameter of about 0·05 mm., so that the two varieties are very finely dispersed, quite probably as individual fibrils.

The diffraction phenomena given by ortho-chrysotile closely resemble those given by the clino variety. Similar restrictions exist on the indices of the observed reflexions, and similar diffuse streaks are obtained. Owing to the restrictions on the indices of the observed reflexions, the relationship between the unit-cell dimensions, and the diffuse scattering on the odd-order layer lines, the zero- and odd-order layer lines of the two varieties are practically identical; but the difference is seen clearly on the even-order layer lines, owing to the different positions of the $h0l$ reflexions of the two varieties. There are also notable differences between the varieties in other respects. Whereas in clino-chrysotile $h0l$ reflexions are very weak for h odd, in ortho-chrysotile there is no restriction on h for strong reflexions with l even. Also, the proportion of