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LATTICE CONSTANTS FROM WEISSENBERG PATTERNS

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In a recent article Christ (1956) has called attention to the possibility of correcting for film shrinkage and related errors in the measurements from Weissenberg patterns by recording the powder pattern of a standard substance on the same film. This provides a scale of standard angular distances on the film. Christ discussed the procedures for calibration at length and claimed "that a precision of about 2 to 4 parts in 10,000 may be easily obtained." To record the powder pattern over but a limited strip of the film a "slotted brass cylinder" was added to the usual rotation shield. Judging from the appearance of the powder pattern shown at the left of Christ's Fig. 1, the width of the slot must have been just over one centimeter. Christ (p. 571) stated that "whenever possible spots measured on the Weissenberg part of the film were taken close to the calibration pattern."

A few years ago the writer (Pabst, 1951, footnote on p. 557) mentioned the use of a c-axis zero layer for calibration. Since then it has been regular practice in single crystal work in the Geology Department at Berkeley to use zero-layer strips of quartz c-axis rotation patterns recorded at each edge of the zero-layer Weissenberg patterns for calibration. Figure 1 shows a Weissenberg pattern calibrated in this manner. The calibration strips are recorded at each edge by insertion of the Weissenberg screen, whose slot width is 0.2 cm., and proper placing of the cassette. Ordinarily an exposure of one hour for each strip will suffice with copper radiation. The general procedure to be followed is that described by Christ and it yields about the precision that he claims.

For the method here reported one must have a suitable reference crystal perfectly oriented on the goniometer head before being placed on the Weissenberg apparatus so that no x-ray adjustment is needed. The writer has used very slim needles of quartz. That used for the pattern shown in Fig. 1 was just under 0.04 mm. in thickness and had been oriented on a Stoe two-circle goniometer fitted with adapter to accommodate the goniom

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eter head of the Weissenberg apparatus. It is convenient, if the operation is to be carried out at frequent intervals and a goniometer head can be spared for the purpose, to keep a standard crystal mounted and adjusted ready for use. The crystal under investigation in the film shown on Fig. 1, a mesolite from Kings Valley quarry, Benton County, Oregon, was



FIG. 1. One half of Weissenberg pattern calibrated with quartz *c*-axis layer strips at each side. Original overexposed to afford better reproduction.

also a slim needle. The α_1 and α_2 lines are well resolved for both the quartz and the mesolite in the outer part of the film.

Though a powder pattern offers a greater number of lines for calibration than does the zero layer of the same substance it turns out that the quartz *c*-axis zero layer furnishes a sufficient number of suitably spaced spots with most of the wave-lengths of *x*-rays commonly used. For copper radiation there are thirteen $hk \cdot 0$ lines from $10 \cdot 0$ at $2\theta = 20.876^{\circ}$ ($\alpha_{unres.}$) to $24 \cdot 0$ at $2\theta = 147.602^{\circ}$ (α_2) at 18° C. If β lines and the resolved α_1 and α_2 are considered, the calibration points are more numerous.

The method of calibration here reported has a number of advantages:

- 1. No modification of the standard Weissenberg apparatus is required.
- 2. No part of the Weissenberg pattern need be sacrificed to make room for the calibration pattern.
- 3. Any departure of the film from a cylindrical shape during exposure

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is readily detected when the calibration strips are aligned on the usual measuring device. Since the cylindrical position of the film is supposed to be fixed when the film is fitted in the cassette in the dark room it will be subject to any small errors in trimming of film, folding of the protective black paper sheath or securing of the metal parts of the cassette.

4. If the two calibration strips have been found to indicate a satisfactory approach of the film to perfect cylindrical shape during exposure, measurements may be made with equal confidence on any part of the film and need not be confined to the region "close to the calibration pattern."

A limiting factor in any calibration method that involves the use of a standard substance is the constancy of the d values of that substance. The range of variation of cell dimensions cited for quartz by Frondel and Hurlbut (1955) is only of the order of 1/10,000. This is well within the limits of precision assigned to the method of calibrated Weissenberg patterns by Christ. Hence the arbitrary use of Wilson and Lipson's (1941) cell dimensions as a basis of 2θ values of quartz for calibration seems justified.

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SERPENTINES WITH 6-LAYER ORTHO-HEXAGONAL CELLS*

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A new variety of serpentine mineral from Unst, Shetland Isles, was described by Brindley and v. Knorring (1954). The most prominent lines of its powder pattern were indexed on the basis of an ortho-hexagonal

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