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# NOMOGRAMS FOR DETERMINING $2\theta$ FROM PRECESSION PHOTOGRAPHS[1]

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## Abstract

Two nomograms are presented which permit $2\theta$ to be evaluated from diffraction spot locations on precession photographs. By means of the first nomogram, $2\theta$ for the wavelength used to produce the precession photograph is obtained. The second permits $2\theta$ (CuK$\alpha$) to be determined from precession spots produced by molybdenum (K$\alpha$) radiation. The accuracy is usually sufficient to allow, in conjunction with observed intensities, unequivocal indexing of the more intense powder diffraction lines of crystalline materials.


## Introduction

The writers have occasionally used indexed precession photographs of single crystals to index in turn the more intense lines of the corresponding powder diffraction pattern. In the course of this work, two nomograms were developed to permit evaluation of $2\theta$ ($\pm 0.3°$) for any reflecting plane whose diffraction spot is recorded on a precession photograph. Since they may similarly save others time in indexing powder diffraction patterns, these nomograms and their use are herein described.

## Development of Nomograms

Figure 1 illustrates the relationship between dimensions in reciprocal lattice space (CO, OP, etc.) and those (CO′, O′P′, etc.) in what may be called "precession lattice" space. By similar triangles

$$\frac{\mathrm{PO}}{\mathrm{CO}} = \frac{\mathrm{P'O'}}{\mathrm{CO'}}. \qquad \text{Eq. (1)}$$

However, CO equals $1/\lambda$, the radius of the sphere of reflection; PO equals $1/d_{hkl}$ for the diffracting plane; and CO′ equals F, the crystal-to-film distance (6 cm.). Substituting these values, Eq. (1) becomes

$$\frac{\lambda}{d_{hkl}} = \frac{\mathrm{P'O'}}{6\ \mathrm{cm}}. \qquad \text{Eq. (2)}$$

From the Bragg equation for reflection, however,

$$\frac{\lambda}{d_{hkl}} = 2\sin\theta.$$

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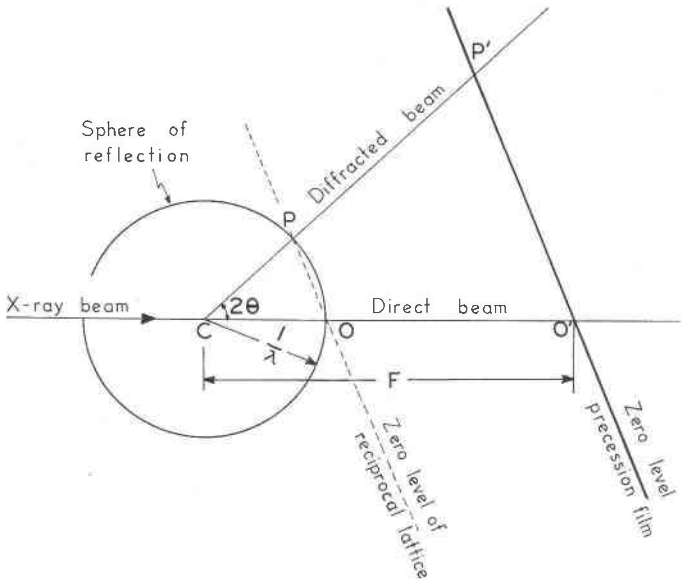


FIG. 1. Relationship between distances in the reciprocal lattice (origin at O) and precession film lattice (origin at O′) for a crystal located at C. CO′ is the crystal-to-film-distance, and P is the reciprocal lattice projection of a crystal plane satisfying the Bragg equation.

Consequently Eq. (2) may be rewritten

$$\sin\theta = \frac{P'O'}{12\text{ cm}} \qquad \text{Eq. (3)}$$

where P′O′ represents the distance of the precession spot (in cm.) from the center of the film.

For *n*-level precession photographs similar reasoning may be applied to develop the relation

$$\sin\theta = \frac{\sqrt{(Fd^*)^2 + (P'O')^2}}{12\text{ cm}} \qquad \text{Eq. (4)}$$

where P′O′ now represents the distance of the precession spot (in cm.) from the center of the *n*-level photograph and Fd* is the film displacement used in making this photograph.

The nomogram permits rapid solution of Eq. (3) or Eq. (4). On the scale reproduced here, Fig. 2 converts 2P′O′, where P′O′ represents the distance of a precession spot from the film center, into the value of $2\theta$ for the reflecting plane responsible for the spot. Its use is best described by an example.[4]

[4] The original Figs. 2 and 3 were exactly 14 cm. and 9 cm. wide, respectively. Glossy prints of the originals are available by request from the U. S. Bureau of Mines, Electrotechnical Experiment Station, Norris, Tenn.

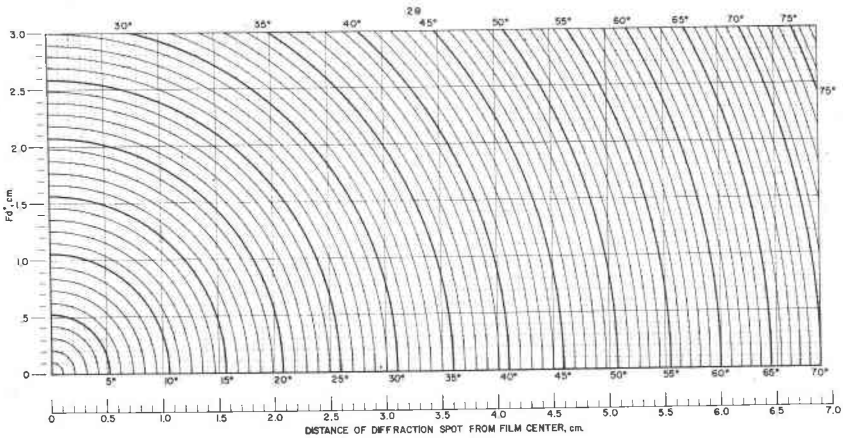


FIG. 2. Nomogram for converting precession spot distances to the corresponding $2\theta$ values.

## USE OF NOMOGRAMS

The first three columns of Table 1 represent data taken, with one exception, from two precession photographs, (*hk*0) and (*hk*1), of synthetic proto-amphibole, an orthorhombic amphibole recently described by Gibbs, Bloss, and Shell.[5] From indexed photographs, twice the distance from the film center (i.e. 2 O′P′) was measured for each precession spot of moderate intensity or greater. This measurement, as well as the precession spot's index and estimated intensity, was then recorded in Table 1 (but not necessarily in the present order). The value of 2 O′P′ was, for each point, translated into its $2\theta$ equivalent by means of Fig. 2, about 15 minutes being required for the entire conversion. Column 5, Table 1, contains $2\theta$ values from powder diffraction data. These angles have an accuracy of $\pm 0.01$. Comparison of $2\theta$ values in Columns 4 and 5 shows that the angles agree closely.

The conversion of the distances 2 O′P′ into $2\theta$ by means of Fig. 2 is particularly simple for the precession spots of a O-level photograph. The Friedel law indicates that, except in the neighborhood of a wave crystal resonance level, a precession spot (*hk*0) will be accompanied by a companion spot ($\bar{h}\bar{k}0$) collinear with it and the film center. Both are at an equal distance from the film center, and, therefore, the distance between the centers of these two spots represents 2 O′P′ for either.

In actual practice the distance 2 O′P′ need not be measured. Prefer-

[5] *Am. Mineral.*, **45**, 974–989, 1960.

TABLE 1. SINGLE CRYSTAL AND POWDER DATA FOR PROTO-AMPHIBOLE

| Precession data (CuK $\alpha$) | | | | Powder diffraction data | |
|---|---|---|---|---|---|
| Index | 2 O′P′ (cm.)[1] | I est. | $2\theta$ from nomogram | $2\theta_{(obs)}$[2] | $I/I_1$ |
| 1) | (2) | (3) | (4) | (5) | (6) |
| 020 | 2.07 | S | 10.0° | 9.85($\alpha$) | 5 |
| 110 | 2.24 | S | 10.9 | 10.69($\alpha$) | 67 |
| 130 | 3.79 | M | 18.25 | 17.67($\alpha$) | 4 |
| 200 | 3.98 | S | 19.3 | 19.02($\alpha$) | 14 |
| 040 | 4.14 | VS | 20.1 | 19.87($\alpha$) | 28 |
| 131 | 4.65[3] | S | 24.5 | 24.428($\alpha$) | 16 |
| 150 | 5.56 | M | 26.7 | 26.697($\alpha$) | 6 |
| | | | | 27.36($\alpha$) | 13 |
| 240 | 5.74 | VS | 27.7 | 27.63 | 33 |
| 310 | 6.07 | VS | 29.3 | 29.13 | 100 |
| 231 | 5.07 | S | 29.7 | 29.58($\alpha$) | 7 |
| 151 | 5.55 | VS | 31.7 | 31.71($\alpha$) | 22 |
| 330 | 6.72 | VS | 32.7 | 32.42 | 25 |
| | | | | 35.25 | |
| 161 | 6.53 | M | 36.1 | 35.90 | 21 |
| 251 | 6.53 | S | 36.1 | | |
| 170 | 7.52 | M | 36.7 | | |
| 350 | 7.90 | M | 38.5 | 38.33($\alpha$) | 4 |
| 400 | 7.96 | S | 38.7 | 38.58($\alpha$) | 4 |
| 261 | 7.37 | M | 39.7 | 39.71 | 7 |
| 080 | 8.30 | M | 40.3 | | |
| 171 | 7.52 | S | 40.5 | 40.36 | 8 |
| 421 | 8.20 | M | 43.8 | 43.54 | 4 |
| 280 | 9.21 | M | 45.0 | | |
| 361 | 8.59 | S | 45.5 | 45.48 | 7 |
| 370 | 9.39 | S | 46.0 | | |
| 190 | 9.55 | M | 47.0 | | |
| 510 | 9.98 | VS | 49.3 | 49.04 | 8 |
| 460 | 10.10 | M | 49.9 | | |
| 451 | 9.48 | W | 50.0 | | |
| 191 | 9.52 | M | 50.1 | | |
| | | | | 51.26 | 4 |
| 461 | 10.07 | M | 52.9 | 52.73 | 8 |
| | | | | 57.05 | 3 |
| | | | | 57.56 | 5 |
| 561 | 11.69 | S | 61.2° | 61.10 | 14 |
| | | | | 62.30 | 10 |
| | | | | 70.55 | 11 |
| | | | | 73.68 | 6 |

[1] Except where indicated, all precession spots were measured from either an (*hk*0) or an (*hk*1) precession photograph. Fd* for (*hk*1) equaled 1.75 cm.

[2] For CuK$\alpha_1$ except where noted as CuK$\alpha$.

[3] From (1*kl*) film; Fd*=0.99 cm.

ably it may be "picked off" the photograph with a pair of dividers and then set off along the abscissa of Fig. 2. The position of the divider point with respect to the circular $2\theta$ scale then permits $2\theta$ to be estimated to the nearest tenth of a degree. The accuracy, generally within $\pm 0.2°$, increases as the size of the precession spots decreases.

For an *n*-level photograph, the process is precisely similar to that above except that the scale used to convert 2 O′P′ to $2\theta$ is not the abscissa line of Fig. 2. Instead, it is a line parallel to the abscissa but above it by the amount Fd*, the film displacement utilized in photographing this particular *n*-level. In the example of Table 1, the value of Fd* for the (*hk*1) photograph was 1.75 cm. A transparent straight edge was therefore so placed over Fig. 2 that it (1) passed through this point on the Fd* scale and (2) was parallel to the abscissa. The precession spot distances for (*hk*1) planes were then set off along the line marked by the straight-edge to obtain $2\theta$ from the circular scales as before.

In this manner $2\theta$ was estimated to the nearest tenth of a degree for all the indexed spots of moderate intensity (or stronger) on the (*hk*0) and (*hk*1) precession photographs. These spots were listed in order of increasing $2\theta$ value so as to facilitate comparison with the more accurately determined $2\theta$ peaks and intensities of a powder pattern of proto-amphibole (Table 1, cols. 5 and 6). As may be seen, the precession indexing may now be carried over to index unequivocally over 50 per cent of the powder peaks. Index (171) rather than index (080) was assigned to the 40.36° powder peak, partly on the basis of its being represented by a more intense precession spot and partly from the observation that, in general, $2\theta$ determined from the nomogram usually exceeded the $2\theta$ value of the powder peak even in the unambiguous cases. Using the $2\theta$ values of the newly indexed powder peaks, the lattice parameters could now be calculated for proto-amphibole with the accuracy characteristic of the powder method.

The method works particularly well for all O-level photographs as well as for *n*-level photographs of orthogonal crystals and monoclinic crystals in the first setting. For *n*-level photographs of non-orthogonal crystals, it is necessary to (a) locate the film center and (b) measure the distance O′P′ of points from this center (rather than 2 O′P′ as was done heretofore to reduce the measuring error). In this case, since Fig. 2 represents a scale twice that of the normal precession photograph (in which F=6 cm.), it is necessary to set off twice the distance O′P′ along the appropriate abscissa-parallel line in Fig. 2 to obtain $2\theta$.

The method developed around Fig. 2 applies to precession photographs made with any wavelengths. Precession spot distances are converted into $2\theta$ values for the wavelength used in making the precession photograph.

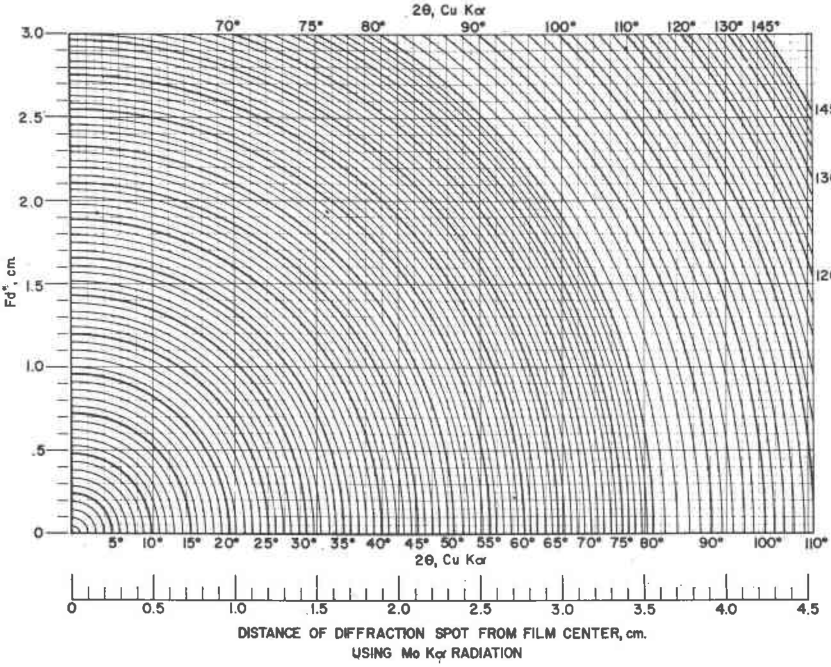


FIG. 3. Nomogram for converting precession spot distances on a precession film made with MoKα into 2θ CuKα values.

Thus, for proto-amphibole CuKα precession spot distances were converted to 2θ CuKα. If FeKα had been used to produce the precession photograph, 2θ FeKα values would have resulted.

Most powder diffraction measurements are carried out with copper radiation, whereas certain advantages accrue in using MoKα in the photography of the reciprocal lattice. Consequently a second nomogram has been developed (Fig. 3) which is used precisely like Fig. 2. Its use is confined, however, to precession photographs made with MoKα. By means of it, precession spot distances for MoKα may be converted to 2θ (CuKα) values.

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