the name for the Ag-bearing mineral which inspired his investigation. It is hoped that the powder data of Table 1 and the contact print of the powder pattern (Fig. 1) will result in locating sufficient material to permit a more complete description of this rare sulpho-salt.

A NOTE ON CONE AXIS AND UPPER LEVEL PRECESSION PHOTOGRAPHS

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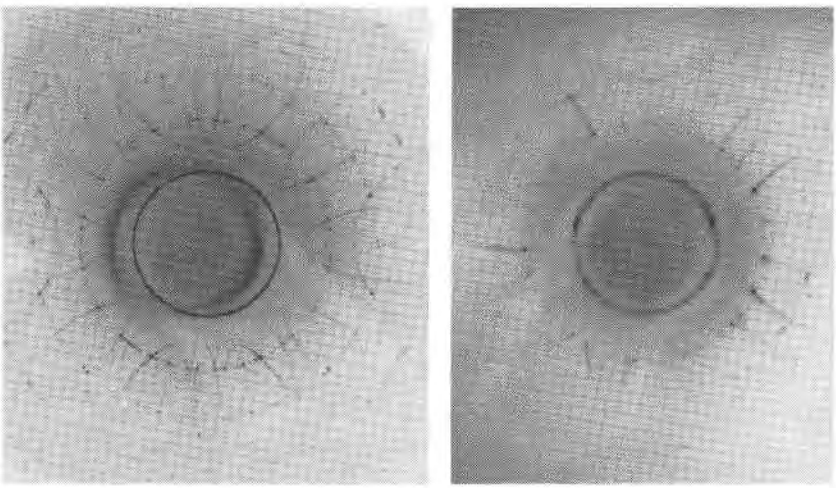
I. *Accuracy of Parameters Obtained from Cone Axis Photographs*

In the determination of the unit cell constants of triclinic crystals with the Buerger precession camera, it is useful to be able to measure the d^* spacing normal to a particular zero-level plane with a reasonable degree of accuracy. Together with the shear obtained from the displacement of an upper-level net, this gives a complete description of a possible unit cell (*cf.* Buerger, *The Photography of the Reciprocal Lattice*, 1944; Fisher, *Am. Mineral.*, **37**, 1007–1054, 1952), which can then be transformed as



a b

FIG. 1. Cone-axis photographs ($\bar{\mu}=20°00'$) of (a) pyrobelonite, (b) melanovanadite. (unfiltered Mo radiation)

desired. The direct determination of d^* from measurements of the diameters of the rings on a cone-axis photograph is not very accurate because (*a*) the crystal-to-film distance usually is known only approxi-

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mately, and (*b*) the film is rarely held perfectly flat in the layer-line screen holder.

If the usual beam trap is used, the zero level is recorded on the film and serves as a calibration standard for the effective crystal-to-film distance, thus,

$$S_e = r_0/\tan \bar{\mu} \qquad (1)$$

where $\bar{\mu}$ is the precession angle , and r_0 is the radius of the zero-level circle on the cone-axis photograph. This value of S_e can then be used to calculate $\bar{\nu}$ for the other circles recorded on the film. In this way an estimate of d^* accurate to 1% or better can be obtained. It is preferable to use unfiltered radiation to ensure that the zero-level ring will appear as a continuous circle, thus increasing the accuracy with which its radius can be measured.

Fig. 1 shows cone-axis photographs of (*a*) pyrobelonite (orthorhombic) and (*b*) melanovanadite (triclinic) (Barnes & Quarashi, *Am. Mineral.*, **37,** 407–422, 1952). Two diameters for each circle were measured in the usual precession film measuring device; this is necessary because the traces of the levels frequently are markedly elliptical. The data for pyrobelonite are given in Table 1. It will be seen that the effective crystal-to-film distance (S_c) is about 5% greater than the nominal value of 30 mm. for which the layer-line screen, carrying the film, was set.

TABLE 1. CONE-AXIS DATA FOR PYROBELONITE ($\bar{\mu}=20°00'$)

h	0	1	2	3	4
mean r_h (mm.)	11.48	19.82	27.40	35.48	45.28
	$S_e = r_0/\tan \bar{\mu} = 31.54$ mm.				
$\bar{\nu}$	20°00′	32°09′	40°59′	48°22′	55°08′
$\cos \bar{\nu}$	0.93969	0.84666	0.75490	0.66436	0.57167
hd_a^*	0	0.09303	0.18479	0.27533	0.36802
d_a^*	—	0.0930_3	0.0924_0	0.0917_8	0.0920_0
	mean $d_a^* = 0.0923 \pm 0.0003$				

The values of d_a^* are all within 1% of that (0.0927_6) obtained directly from the a^*b^* zero-level precession photograph. There is, however, some evidence of a systematic variation which probably is due to bulging of the film in the centre. Errors in d_a^* due to this cause will vary approximately as r_h^2 so that a more accurate value can be obtained by plotting d_a^* against r_h^2 and extrapolating linearly to r_0^2, for which S_e is known accurately.

Graphs of this type are shown in Fig. 2 for the two minerals. In the case of pyrobelonite, the extrapolated value of d_a^* is within 0.25% of

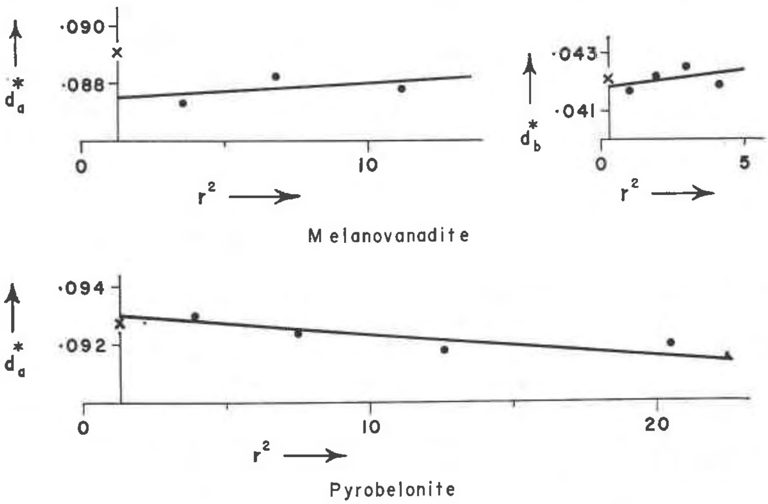


FIG. 2. Graphs of d^* against r^2 for cone-axis photographs of pyrobelonite and melanovanadite; crosses at $r^2 = r_0^2$ indicate values of d^* obtained from 0-level precession films.

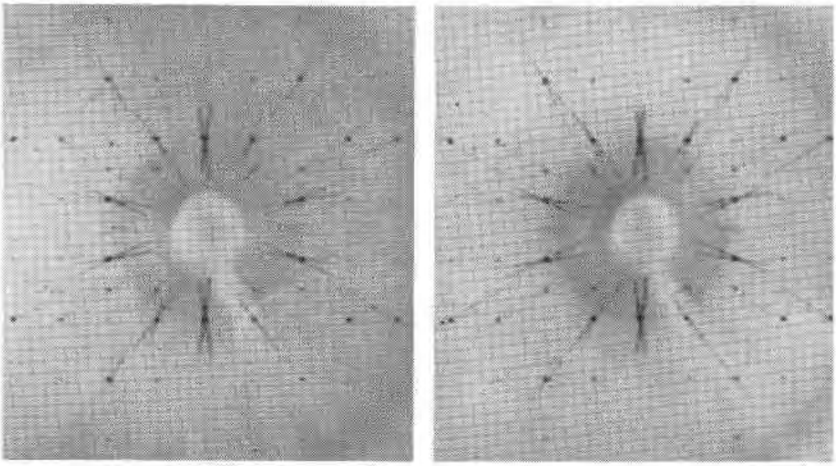
that obtained from the appropriate zero-level precession photograph and is most satisfactory. In the case of melanovanadite, however, the agreement between reciprocal spacings obtained from cone-axis and from precession photographs is not as good; 0.7% for d_b^* and 1.8% for d_a^*. The latter figure, however, represents the largest discrepancy observed among a number of such tests and is given to show that the method is useful even with crystals of poor quality. Those of melanovanadite usually occur as long needles with a helical twist about the needle axis.

In the foregoing discussion, the value of $\bar{\mu}$ is assumed to be exact. It is easily shown that the fractional error $\Delta d^*/d^*$ caused by an error $\Delta\bar{\mu}$ is less than $2\Delta\bar{\mu}/\bar{\mu}$. With the large vernier of the precession instrument, $\bar{\mu}$ is set readily to better than 0.05° so that this error will be well below 0.5% if $\bar{\mu} > 20°$. Film shrinkage errors, of course, are eliminated by the calibration and extrapolation procedure. Under favourable conditions, therefore, this method will yield values of d^* that are accurate to 0.5% or better. In fact, if an accuracy of only 2% or 3% is required, the diameters of the circles can be measured with a centimeter rule.

II. *Accurate Setting of the Cassette for Upper Level Photographs*

The increasing use of upper level precession photographs for intensity estimations is evident from the publication of charts for the appropriate L.p factors (Burbank, *Rev. Sci. Instr.*, **23,** 321–327, 1952; Grenville-Wells

& Abrahams, *Rev. Sci. Instr.*, **23,** 328–331, 1952). For such work it is desirable to obtain spots of a uniformly circular shape, which necessitates making Fd^* settings of the film cassette to better than 0.1 mm. Some methods of estimating and correcting errors in Fd^* are described by Buerger (*loc. cit.*).



a b

FIG. 3. First-level precession photographs of conichalcite, unfiltered Mo radiation, (*a*) setting error, $\Delta(Fd^*) = -0.3$ mm., (b) setting error, $\Delta(Fd^*) = +0.3$ mm.

For some time we have been using a somewhat different method which is both simple and effective. The basic principle is brought out in the photographs of Fig. 3, which were taken with unfiltered MoK radiation and for which Fd^* was intentionally misset by ± 0.3 mm. It will be seen that the position of the cross-over of the two streaks of white radiation corresponding to each reflection is a function of the setting error; for perfect setting it should coincide with the position of the K_α reflection.

If the distance f between the K_α and K_β spots on either of the two streaks for a given reflection is measured, it can be used as a calibration standard, *viz.*, $(Fd^*)_\alpha - (Fd^*)_\beta \propto f$. Also, the error $\Delta(Fd^*) \propto f_1$, where f_1 is the distance of the crossover from either of the K_α spots that would coincide if there was no setting error. It follows that

$$f/f_1 = ((Fd^*)_\alpha - (Fd^*)_\beta)/\Delta(Fd^*) = (Fd^*)_\alpha(1 - \lambda_\beta/\lambda_\alpha)/\Delta(Fd^*)$$

whence,

$$\Delta(Fd^*) = (Fd^*)_\alpha(1 - \lambda_\beta/\lambda_\alpha)(f_1/f) \tag{2}$$

The direction of the required correction in Fd^* is evident from inspection of the photographs, *e.g.*, if the cross-over is between the K_α and K_β spots, the (Fd^*) setting is too small and must be increased.

Since $(1-\lambda_\beta/\lambda_\alpha)$ is approximately 0.1, even a rough estimate of (f_1/f) gives an accurate value of $\Delta(Fd^*)$. The accuracy can be estimated theoretically as follows.

From the geometry of upper-level precession recording and by an extension of Buerger's equation 29 (*loc. cit.*, p. 30),

$$_1 = \Delta(Fd^*) \tan \bar{\nu} + \Delta(Fd^*)\xi/d^*$$

so that,

$$f_1 = \Delta(Fd^*) (\tan \bar{\nu} + \xi/d^*) \tag{3}$$

An estimate of the probable value of ξ is required to evaluate this expression. Now, $\xi_{\min.} = (\sin \bar{\nu} - \sin \bar{\mu})$, and $d^* = (\cos \bar{\mu} - \cos \bar{\nu})$, from which

$$F\xi_{\min.} = Fd^* \cot ((\bar{\mu} + \bar{\nu})/2) \text{ cm.} \tag{4}$$

(if F is given in cm.). In view of the rapid decrease in intensity with increasing ξ, a reasonable average value for $F\xi$ is $(F\xi_{\min.} + 2)$cm. Thus, by substitution in (3),

$$f_1/\Delta(Fd^*) \sim \tan \bar{\nu} + \cot ((\bar{\mu} + \bar{\nu})/2) + 2/Fd^* \tag{5}$$

and, therefore,

$$f_1/\Delta(Fd^*) \simeq 2.5 + 2/Fd^* \tag{6}$$

which is of the order of 4 or more for practical values of Fd^*. Consequently, if f_1 is measured to 0.5 mm., $\Delta(Fd^*)$ is known to about 0.1 mm. or better.[1] For comparison with Buerger's eq. 29,

$$f_B = \Delta(Fd^*) \tan \bar{\nu} \tag{7}$$

we obtain, from (3) and (7),

$$f_1/f_B = (1 + (\xi/d^*) \cot \bar{\nu}) \tag{8}$$

or, from (5) and (7),

$$f_1/f_B \sim (1 + (\cot ((\bar{\mu} + \bar{\nu})/2) \cot \bar{\nu} + (2/Fd^*) \cot \bar{\nu}) \sim 5 \text{ (or more} \tag{9}$$

The present method, therefore, is much more sensitive to ΔFd^* than one based solely on the linear displacement of the K_α spot.

[1] For extreme accuracy, the white radiation streaks can be drawn on tracing paper by suitable manipulation of the precession film measuring device; f_1 and f can then be measured to within 0.1 mm.