

The new analysis shows that the volborthite from Woskressenskoi, Perm, U.S.S.R., is similar in V_2O_5 and CuO content to volborthite from other localities, and that it is not unique by reason of its low V_2O_5 or high H_2O content, as indicated by Genth's adjusted analysis.

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THE AMERICAN MINERALOGIST, VOL. 50, MAY-JUNE, 1965

PITFALL IN DETERMINING 2V IN MICAS

F. DONALD BLOSS,¹ *U. S. Bureau of Mines, Norris, Tennessee.*

The value of the optic axial angle 2V in flakes of synthetic mica varies greatly according to whether they are truly single crystals or instead are interlayerings of twinned individuals such as described by Bloss *et al.* (1963). For such interlayerings the value for 2V is likely to be significantly lower than the true value obtainable from a single crystal, a possibility that was recognized early by Reusch (1869). Present day investigators perhaps lose sight of this possibility when they report variations of 2V for a given mica in a thin section without investigating the reason. In such cases the question arises as to whether, for the mica flakes examined, this variation in 2V is the result of variation in (a) composition, (b) degree of interlayering of twinned components, or (c) polytypism.

The various micas synthesized at the Norris Metallurgy Research Laboratory of the Bureau of Mines were from melts of closely controlled chemical compositions. Thus far, the variations in 2V observed for flakes from the same batch appear to be attributable to differences in degree of interlayering. For synthetic phlogopite flakes which are solid solutional variations from the ordinary $K_2Mg_6Al_2Si_6O_{20}F_4$ composition, interlayering seems more the rule than the exception. It is thus understandable why low values for 2V have generally been reported for these, especially be-

¹ Present address: Dept. of Geology, Southern Illinois University, Carbondale, Ill.

Material	Measured 2V	
	This paper	Reported value
Fluorphlogopite $K_2Mg_6Al_2Si_6O_{20}F_4$	$14.0^1 \pm .3^\circ$	9° (ca) Van Valkenburg and Pike, (1952, p. 367) 14.0 (Emerson, 1955, p. 22) 14.0 (Kohn and Hatch, 1955, p. 15)
Fluormica intermediate $K_{1.3}Mg_{4.8}Li_{1.25}Si_{7.96}O_{20.03}F_{3.97}$	$10.4^1 \pm 1^\circ$	2–5° (Miller and Johnson, 1962)
Germanium fluorphlogopite $K_2Mg_6Al_2Ge_6O_{20}F_4$	$16.7^1 \pm 1^\circ$	2° (Miller <i>et al.</i> , 1964)
Barium disilicic fluormica $Ba_{2.03}Mg_{5.73}Al_{4.06}Si_{4.06}O_{20.10}F_{3.90}$	$25^2 \pm 2^\circ$	4–6° (Miller <i>et al.</i> , 1963)

¹ Average of 40 measurements. The value after the decimal point has been retained but is not considered a significant figure.

² Average of 24 measurements.

cause the investigators were aware of neither the problem nor its remedy.

The degree of underestimation of the value for 2V in synthetic fluormicas is at times surprising. The writer has had occasion to recheck the value of 2V for four of the synthetic micas previously studied at this laboratory. Results are as follows:

Use of examples largely from this laboratory does not signify that the problem itself is so confined, but merely that suitable material was close at hand to check previous optical work personally. The writer suspects that Van Valkenburg and Pike's (1952) report of 2V as "about 9°" for potassium fluorphlogopite, $K_2Mg_6Al_2Si_6O_{20}F_4$, synthesized in their laboratory represents a measurement on a twinned flake. The basis for this suspicion is their determined 2V's lack of agreement with the value of 14.0° obtained by other investigators on material from Norris (but not necessarily from the same batch).

Although the problem may be less acute for natural micas, particularly those of non-volcanic origin, the writer suspects that it exists. D. Cummings (personal communication) cites micas in petrographic thin sections which possessed interference figures resembling the anomalous types described by Bloss *et al.* (1963, Fig. 1, p. 538) as typical of twinned interlayers. Whenever the petrographer observes a range in the value of 2V he should be suspicious of the possibility of interlayering and should study the interference figure for a more positive clue.

A twinned interlamination acts like a compensator on the remainder of the flake except that, because of its location, the degree of compensation increases for the more oblique rays emerging nearer the edge of the field of view in the interference figure. Consequently, when measuring $2V$ for a mica flake, the investigator is well advised to examine first the higher order isochromes in the interference figure. If offsets occur in these isochromes as they pass across the isogyres at 45° off extinction—in other words, if the interference figure is of the anomalous type (Bloss *et al.*, 1963, Fig. 1) associable with interlaminations of twins—an incorrectly small value of $2V$ should be expected. If such is the case, $2V$ should be reported as “greater than” the value actually determined. More reliable values for $2V$ will be obtained from mica flakes which show sharp extinction rather than undulatory extinction and which do not exhibit either (a) areas of slightly different birefringence or (b) offsets of the isochromes in their interference figures.

The writer's measurements of $2V$ for the four phlogopites here reported were made on flakes which conformed, as closely as possible, to the above requirements. Only very thin flakes of the barium disilicic mica so conformed. As a result the isogyres were rather broad, and the accuracy of determining $2V$ decreased somewhat.

Each flake was examined conoscopically with a Zeiss polarizing microscope equipped with a universal stage and a sodium light source. After optic direction Y was brought to coincidence with the A_4 -axis of the U-stage, the positions of both optic axes were measured three to five times, as the A_4 -axis was at 45° , 135° , 225° , and 315° to its initial position. The crystal was then rotated 180° on the A_1 -axis, and the measurements were repeated. Thus, the values reported by the writer represent the average of from 24 to 40 measurements. Where the mica flakes were sufficiently large in area, the Zeiss objective U.D. 16/0.17 yielded good interference figures without insertion of the substage condensing lens. The glass hemisphere of the U-stage evidently acted as a condensing lens. Because of its smaller angular aperture a relatively low power objective produced a greater separation of the melatopes in the field of view. The greater separation of the melatopes should yield increased accuracy. For flakes of smaller area, higher power objectives must be used, in order to visualize the interference figure.

SUMMARY

Accurate determinations have shown that, at least for the synthetic fluormicas, the reported values for the optic axial angle $2V$ were seriously in error and low. A single crystal must be used,—not one interlayered or twinned. The higher order isochromes in the interference figure should

be examined for offsets. Single crystals are characterized by the absence of offsets occurring as the isochromes pass across the isogyres at 45° off extinction. The postulation is made that some values of 2V for natural micas may also be erroneous.

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THE AMERICAN MINERALOGIST, VOL. 50, MAY-JUNE, 1965

NOTE ON "BIOTITE MICA EFFECT IN X-RAY SPECTROGRAPHIC ANALYSIS OF PRESSED ROCK POWDERS" BY A. VOLBORTH

A. K. BAIRD, D. A. COPELAND, D. B. MCINTYRE AND E. E. WELDAY,
Department of Geology, Pomona College, Claremont, California.

In Professor Volborth's recent paper in this journal, preliminary chemical values for "Q. Monzonite 84" (Volborth, 1964, Table 3, col. 6) as determined by us, are quoted. These values were obtained using W-1 and G-1 only for calibration. Because samples of this rock (now termed Standard #5, Pomona College) have been widely distributed to spectrographers it is important that newer, more precise determinations based upon multiple standards be given to avoid misuse of the analytical values in x-ray calibration procedures. Furthermore, the comparison of these newer values with Volborth's values adds emphasis to his conclusions concerning the chemical biases introduced by mica in rocks when using unfused samples for analysis by x-ray spectrography.

Revised values by oxide for this standard are shown in Table 1 where they may be compared with those of Volborth (1964, Table 3, col. 7). With the direct determination of oxygen in silicates now possible (Baird