NOTES ON PRECISION AND ACCURACY OF OPTIC ANGLE DETERMINATION WITH THE UNIVERSAL STAGE

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Abstract

Precision of determination of 2V varies greatly with the method used, with maximum precision being shown by the so-called direct method. Optimum conditions demand double axis measurement and settings at both 45° and 135° positions of the microscope stage. Precision increases roughly with birefringence and also with section thickness. Based on quintuplicate measurements, precision varies between .02° and .6°. Accuracy was tested by using an optic angle goniometer as control instrument. A satisfactory calibration (within a few tenths of a degree) was obtained for five of the six U-stages used for the test.

As an example of the need for information on precision and accuracy, careful work on several high-temperature, unzoned plagioclase grains in a rhyolite shows a range in optic angle of about 6°. With more casual examination this range of angle could easily be ascribed to errors of various kinds. Since refractive index study (even by the double variation method) cannot be substituted for 2V determinations in cases as critical as this, the advantage of mastery of the Universal stage technique is obvious.

Introduction

The increased use of the U-stage in investigations of the optic character and properties of anisotropic crystals is encouraging evidence of its acceptance as standard equipment for quantitative work. Published U-stage measurements however show in many cases a lack of concern about precision and accuracy, both of which can be checked with little extra work and which would seem to be essential in forming conclusions about critical mineralogic and petrologic problems. Fairbairn and Sheppard (1945) dealt with some aspects of precision from the standpoint of single measurements. Although similar studies have undoubtedly been made in other laboratories, the dearth of published data on the subject is somewhat disturbing. Wright (1907) gives data for topaz; more recently Hess (1949) published replicate measurements on monoclinic pyroxene. It is the purpose of this paper to demonstrate the relative precision which may be expected under certain situations and to present also some data regarding the accuracy of U-stage determinations. We are not primarily concerned here with variations *between* crystals in a rock section; that is a purely petrologic problem which can properly be considered only after the precision of single crystal measurements has been evaluated.

Precision of Optic Angle Determinations

The precision, or reproducibility, of a measurement is a matter which vitally concerns every U-stage operator. A fundamental knowledge of proper stage and lamp adjustment, in order to obtain correct centering and approximately parallel illumination, is, of course, a prerequisite. With these details attended to and standardized the degree of precision attainable is controlled by birefringence, thickness, orientation, etc., all of which must be evaluated. A number of methods of determining 2V with the U-stage are known, not all of equal importance for one reason or another. They are discussed in the following sections.

Extinction Angle Procedures

These methods, developed for the U-stage by Federow and Berek, are all based on the fundamental Biot-Fresnel law relating extinction directions to optic axes. The construction of "optic curves" for precise location of an optic axis is an illustration of this law. It is an elegant method, but is too time-consuming for the present generation of petrographers and requires exceedingly careful work with the stereographic net. Federow himself recognized this handicap of the method a half-century ago. Wright (1911) and Johannsen (1918) have described the procedure fully.

The principal use of extinction angle methods at present is as an aid in identification of indicatrix directions and for approximating 2V. Berek (1924), followed by Dodge (in Emmons, 1943), developed this application of the Biot-Fresnel law. Single determinations are rapid, but as a precision method for 2V it leaves much to be desired, since a considerable number of trials are needed before a satisfactory mean value is reached. Thus in a section of anhydrite having Bx_o vertical, 9 replicate extinction angle measurements were needed to obtain the mean of $2V = 43.3°$ found by direct measurement (see Table 1). Moreover the error of this mean is 1.6° (based on 9 trials) compared with 0.3° by direct measurement (based on only 5 trials). Undoubtedly for certain orientations the Berek-Dodge method is capable of greater precision than this, but as already mentioned its principal usefulness is as a guide for orientation and optic angle measurement (Emmons 1943, p. 30).

Relative Retardation Method

Long before quantitative work with compensators was possible, it was known that 2V could be approximately determined from the Michel-Lévy interference color chart, based on the relation $\sin^2 V = (\beta - \alpha)/(\gamma - \alpha) = (\Gamma_z/\Gamma_y)$. Until the Berek and other graduated compensators became available, however, the quality of the results obtained was not commensurate with the time expended. The combination of the U-stage (for exact orientation) and a graduated compensator (for exact retardation) now make possible the indirect determination of 2V with moderate precision. Best results are likely to be attained if (1) all necessary retardations can be obtained from one grain (this excludes possible errors due to variable section thickness, or to compositional differences), (2) grains are used having symmetry planes as nearly parallel as possible with the microscope axis. Both these goals are obviously impossible to achieve simultaneously and one must compromise. A determination of 2V with the Berek compensator for barite is typical of the results to be expected by this procedure. A grain was found which was adequate for the entire computation (Table 1). A mean orientation position of its symmetry

TABLE 1. PRECISION OF EXTINCTION ANGLE AND RELATIVE RETARDATION METHODS OF 2V DETERMINATION

Mineral	Extinction Angle Method (based on 9 measurements)	Relative Retardation Method (based on 5 measurements)	Comparison with Double Axis Method (based on 5 measurements)
Anhydrite	43.3 (1.6)		43.3 (.3)
Barite		39.6 (2.1)	40.3 (.2)

Error of each mean is shown in parentheses.
In this and succeeding tables error of the mean is the standard error

$$s_{\bar{x}} = \sqrt{\frac{\Sigma(\bar{x} - x)^2}{n(n-1)}}$$

where $\bar{x}$ = arithmetic mean
x = single measurement
n = number of measurements.

planes was obtained, based on five individual trials, and the standard error of the angle (normal to the section $\wedge$ microscope axis) was computed. This turned out to be .5°. Five sets of readings of the compensator were made,[1] an average computed and also the error of this mean (.02). From this the retardations can be found and, knowing the error in the U-stage settings and in the compensator readings, the total error in each retardation can be obtained. These are of the order of magnitude of 10 mμ or less. Computation of 2V (or careful reading of the 2V nomogram) gave 39.6°; the error comes out to 2.1°. Direct measurement of optic angle on this same grain gave 40.3° with an error (for 5 measurements) of 0.2°. As with the extinction angle procedure, the theory of the retardation method is superior to the practice, and it is not a satisfactory substitute

[1] For best results the method of using extinction bands several wavelengths higher than the compensation position, as recommended by Mosebach (1949), should be followed.

for the direct measurement of 2V. As a by-product of a birefringence determination, which requires a graduated compensator, 2V can often be determined from the same compensator readings without additional measurement. Since many grains are unsuitably oriented for direct determination of 2V, the method, although not of great precision, is nevertheless useful.

Direct Method

This method is so superior to the previous ones that comparisons can scarcely be made. It requires, for highest precision, orientation of both optic axes, but reasonably good work can also be accomplished with one axis only. It is the method in common use today with the U-stage. Two operations are involved, (*a*) the orientation of the optic plane parallel to the microscope axis, (*b*) the orientation of the optic axes parallel to the microscope axis. As a result of a considerable number of tests, the writers recommend the following procedural details for double axis orientation.

(1) The optic plane is oriented in a vertical and north-south position (for either the 4- or 5-axis stage). No angles need be recorded for this step. The acute bisectrix in the unoriented section must be approximately vertical in order that *both* optic axes can be oriented.

(2) The optic axes positions are read on the outer east-west axis (single readings) for two settings of the microscope axis, approximately 90° apart. The two readings for each axis are averaged; the difference between the two averages is the observed value of 2V.

(3) Steps (1) and (2) are repeated four times, using the same grain. The five values of 2V are averaged and the standard error of this mean is computed.

The duplicate settings of each optic axis for two orientations of the microscope axis increase precision considerably and are well worth the few extra minutes required. The procedure tends to compensate for inaccuracies in the optic plane setting. Data have been obtained (Table 2) for a group of selected minerals to illustrate the error in their means for various birefringences and section thicknesses. Each mean is obtained from measurements of the same grain and, particularly in the example of plagioclase, from the same part of the same grain.* The study is therefore not concerned with variation in optic angle from grain to grain in a rock section, but only with the experimental error of replicate measurements of the same grain. These two goals should not be confused.

Except for barite, each mineral was investigated by two operators,

* Except for plagioclase (2) in Table 2, where the mean is the result of measurements at different points in the grain. Since the error of this mean is comparable with those for the other plagioclases, the essential homogeneity of the crystals is reasonably well established.

TABLE 2. PRECISION OF DOUBLE AXIS DETERMINATION OF 2V

Mineral	Operator	Mean 2V, based on 5 settings		Error of mean		Thickness of section t	Birefringence B	Angle of tilt of optic plane
		4-axis stage	5-axis stage*	4-axis stage	5-axis stage*			
Aragonite	H.W.F. T.P.	18.05 18.26	18.38	.05 .02	.06	.105	.155	None
Anhydrite	H.W.F. T.P.	42.55 42.41	42.95 43.13	.25 .30	.13 .20	.012	.044	~10°
Muscovite	H.W.F. T.P.	42.80	 42.67	.16	 .09	~.07	~.04	None
Barite	H.W.F.		40.3		.2	~.012	.012	~35°
Andalusite	H.W.F. T.P.	85.2	84.6	.1	.1	~.09	~.01	None
Topaz	H.W.F. T.P.	65.46 65.40	65.03 65.04	.13 .06	.2 .2	~.06	.010	None
Plagioclase (1)	H.W.F. T.P.	68.2 69.2	68.9 67.7	.4 .6	.3 .1	~.02	~.008	~15°
Plagioclase (2)	H.W.F. W.F.B†	61.7 61.5		.3 .3		~.02	~.008	~10°
Plagioclase (3)	H.W.F. L.F.H.†	 64.0	64.3	 .3	.3	~.02	~.008	~30°
Plagioclase (4)	H.W.F. W.H.†	63.2 63.2		.2 .25		~.02	~.008	~25°

* Stage No. 5 (See Table 5).

† The writers are indebted to W. F. Brace, L. F. Herzog, and W. Holyk for their co-operation in making these plagioclase determinations

each without knowledge of the other's results. Some were measured with the 4-axis, others with the 5-axis stage, as noted. All readings of the optic axes were made on the east-west axis. The following points are obvious from the table: (1) any two operators obtain errors of comparable order of magnitude for a given mineral; (2) the expected direct relation between precision and birefringence holds throughout, modified to some degree by variations in thickness (precision varies in general with thickness); (3) the effect of angle of tilt of the optic plane is not clear. High angles are expected to lower precision, but the 35° maximum in Table 2 does not seem to be high enough to achieve this; (4) the average error obtained with the 5-axis stage (.17) is essentially the same as with the 4-axis stage (.23). More extensive measurements would be needed, however, to establish this as a general condition. Mechanically the 5-axis instrument is at a disadvantage, but the scope of Table 2 is too limited to indicate this; (5)

the maximum difference between means for any given mineral is 1° (plagioclase); the minimum is .13° (muscovite). This supports the general thesis that increased precision is to be expected where birefringence and thickness are relatively high.[2]

Although offering somewhat lower precision, and a tendency to a bias in the means, the single axis procedure may in certain cases be the only source of 2V data by direct measurement. Table 3 gives information based on the same grains and same general procedure as used for the double axis method. Precision is reduced about one-half and the means vary between 0.6° and 2.8° from the double axis results. The large variations are confined to plagioclase; for all the other minerals the differences are slightly less than 1°. Even with optimum conditions for measurements, therefore (high birefringence and relatively thick sections), the single axis is not a satisfactory substitute for the double axis method and if from necessity it is used, the results must be interpreted with considerable care.

TABLE 3. COMPARISON OF SINGLE AND DOUBLE AXIS DETERMINATIONS OF 2V

Mineral	Single axis mean* (5 settings)	Double axis mean (from Table 1)	Diff. in 2V	Thickness of section t	Birefringence B	Error of single axis mean	Error of double axis mean
Plagioclase (1)	70.8	68.5	2.3	~.020	~.007	.4	.2–.3
Plagioclase (2)	63.0	61.6	1.4	~.020	~.007	.5	.2–.3
Plagioclase (3)	67.0	64.2	2.8	~.020	~.007	.5	.2–.3
Aragonite	18.8	18.2	0.6	~.105	.155	.1	.05
Andalusite	84.2	84.9	0.7	~.090	~.01	.4	.1
Anhydrite	43.8	43.0	0.8	~.012	.044	.3	.2
Muscovite	41.9	42.7	0.8	~.07	~.04	.2	.1
Barite	40.9	40.3	0.6	~.012	.012	.3	.2

* Measurements on same grains as for double axis mean.

ACCURACY OF OPTIC ANGLE DETERMINATIONS

Evaluation of precision in physical measurements is a relatively simple matter; the same however cannot be said for accuracy, since the bias of

[2] Orientation on the U-stage by the interference figure method was not possible with our equipment for all the minerals. The writers agree with Hallimond (1950) that this method gives higher precision in symmetry plane settings than is to be expected with conventional extinction position methods, particularly for crystals of low birefringence. An interesting extension of the present study would be a comparison of precision by the interference figure procedure for a range of minerals such as in Table 1.

the mean of any set of measurements from the true value is unknown. One must therefore be satisfied with an approach to the true value and select an independent and presumably superior method for control. In the case of optic angle measurements the best control instrument for the U-stage is the optic angle goniometer. With this instrument 2V can be determined indirectly with an oriented thin section by measurement of 2E; or directly, using a sphere cut from the selected mineral. Both procedures were carried out, with results as shown in Tables 4 and 5.

TABLE 4. ACCURACY OF U-STAGE MEASUREMENTS OF 2V
(Based on 2E determinations)

Mineral	2E (Optic angle goniometer)	Error in 2E	β	Error in β	2V (calc.)	Error in calc. 2V	2V (measured with U-stage)	Error in measured 2V
Aragonite (H.W.F.)	30.90 (mean of 5 sets of readings)	.03	1.6816 (from Rosenbusch Wülfing Mügge)	.0002 (Assumed value)	18.23	~.03	18.22 (see Table 1)	.05
(T.P.)	30.97	.06		.0002	18.27	~.05	18.26	.02
Aragonite (Data from Rosenbusch Wülfing Mügge)	30.90	Not stated	1.6816	Not stated	18.23	Not stated	No data	
Aragonite (Data from Winchell)	30.87	Not stated	1.681 (?)	Not stated	18.18	Not stated	No data	
Topaz (Wright, (1907)	126.2	Unknown	Not stated but probably determined by minimum deviation method	Unknown	66.7	Unknown	66.6	Unknown

An oriented section of aragonite was used for the indirect computation of 2V. In Table 4 the authors' results may be compared with published data for 2E. It is apparent that, whether instrumental or compositional (or both), variations in 2E are satisfactorily small. The intermediate index β must also be relatively constant; even if an uncertainty of $\pm.0002$ in the published value is assumed, the error of the calculated 2V is still quite small. As it was not possible to determine β in our section of aragonite within limits smaller than $\pm.001$, the published value has been accepted. The computed value of 2V is practically identical with the values obtained by U-stage measurement. Even if the uncertainty in β were

±.001, the resultant error in 2V would be less than 0.1° (computed from the differentiated form of sin E/sin V $=\beta$). This test of the accuracy of the U-stage may therefore be considered satisfactory.

For the direct method a sphere about $\frac{7}{8}$-inch in diameter was cut from a large, clear, single-crystal fragment of topaz. From the same material an oriented thin section was prepared. The sphere, properly centered on the goniometer, passes light without deviation across the air-topaz boundary surface and gives values directly comparable with those

TABLE 5. ACCURACY OF U-STAGE MEASUREMENTS OF 2V
(Direct method, using topaz sphere)

Operator	Illumination	Optic angle goniometer	4-axis stages				5-axis stages	
			1	2	3	4	5	6
H.W.F.	Sodium lamp	65.17 (.01)*	65.03 (.15)	65.86 (.08)	65.48 (.09)	65.31 (.17)	65.03 (.2)	66.60 (.09)
	Tungsten lamp	—			65.63 (.16)			66.28 (.04)
T.P.	Sodium lamp	65.18 (.02)			65.15 (.07)		65.04 (.2)	66.54 (.2)
	Tungsten lamp	—			65.65 (.05)			

Mean for all U-stages (excluding #6) 65.35. No. 1 is a Fuess stage; all others are Leitz.
* Numbers in parentheses are standard errors for each mean.

obtained on the U-stage with the thin section. These measurements are given in Table 5. Six U-stages were tested; only one failed to yield 2V values within a few tenths of a degree of the goniometer value. This stage was known however to have a slightly defective bearing and the high values, obtained by both of us, are ascribed to this cause. Two stages gave consistently lower, three gave consistently higher values than the goniometer. The mean for all five is less than 0.2° from the goniometer result. Although in the aragonite measurements the two U-stages tested were closer to the goniometer value than this, we believe that the topaz calibration is preferable,[3] since no assumption regarding refractive index

[3] A check on the order of magnitude of errors in 2V which would result if the sphere were slightly elliptical was carried out by a graphical method. The sphere, in the circumference parallel to the optic plane, was known to vary in diameter by .0001 inch. Assuming the least favorable orientation of the optic axes relative to this slightly elliptical optic plane section, the error in 2V turns out to be less than .05°. This is of the same order of magnitude as the errors computed for the means in Table 5 and is therefore relatively insignificant.

need be made. In either case a check on the magnitude of the bias in measurements made with a given U-stage can be obtained.[4]

As far as the writers are aware the results of only one previous calibration of a U-stage have been published (Wright, 1907). These figures are included in Table 4 and indicate that even the small, early models of the U-stage were apparently well constructed. As Wright gives no data on precision, further comparison with our work is not possible.

General Discussion

The optic angle of a crystal, if properly determined and evaluated for error, is a significant and highly diagnostic property of a crystal. If carelessly done, without regard for matters of either precision or accuracy, one's time is largely wasted, for a reasonable estimate of 2V can be made from an interference figure without need for a U-stage at all. Furthermore, incorrect petrologic conclusions may be drawn in some instances if optic angle work is poorly done. As an illustration we cite the data for the plagioclase crystals in Table 2. These are phenocrysts from a thin section of a rhyolite from Nevada known to contain high temperature plagioclase.[5] Detailed measurements on four unzoned phenocrysts, using the double-axis procedure, gave four different means* for 2V, ranging from 61.5 to 68.5. Since the error of the means is only 0.3 (approximately) no question can arise regarding the validity of the separate 2V values. But if the single-axis procedure had been used (see Table 3), or some other of the approximate methods, it might have been concluded that the plagioclase was uniform throughout the rock and that discrepancies shown by 2V represented instrumental and operator error. Furthermore, if optical study of these phenocrysts had been limited to the refractive index immersion procedure, the variations indicated by the optic angle investigation would not be detected. If, for example, indices are determined to $\pm.001$ one can easily show that, for the values determined ($\alpha=1.530$, $\beta=1.536$, $\gamma=1.540$), computed values of 2V can vary between 52° and 83°. If the uncertainty is $\pm.0001$, as might be the case with the double variation technique, computed values of 2V will still have a range of 5°. This sensitivity of 2V to extremely small changes in refractive index is of course well known and is a characteristic of the optic indicatrix

[4] Since cleavage fragments of topaz are normal to the acute bisectrix they provide naturally oriented sections and facilitate determination of 2V with the U-stage. The large single-crystal cleavage fragment from which the sphere was cut for this investigation will provide several hundred small cleavage plates suitable for mounting on glass slides. The authors will be glad to supply such cleavage plates without cost to those interested in U-stage standardization.

[5] Specimen loaned to the authors by O. F. Tuttle.

* Hemisphere corrections were made as standard procedure.

which, although it demands careful treatment, should be exploited fully rather than avoided. On the other hand, even the most painstaking refractive index investigation may not reveal any but gross inhomogeneities in a group of supposedly uniform crystals. Since the example cited here for plagioclase may have its counterpart in other minerals and in other rocks, no effort should be spared to reveal valid inhomogeneities by carefully controlled optic angle investigation. According to Tuttle (personal communication) one explanation of the range in 2V in these plagioclase phenocrysts might be sluggish inversion from the high to the low form which has progressed further in some grains than in others. Where *x*-ray spectrometric analysis can be combined with precise optic angle investigation it is probable that many petrologic riddles may be solved. Tuttle and Bowen (1950) have already given some indication of this in their recent work on feldspar. Success is possible however only if meticulous attention to operational details is maintained. It is the hope of the authors that this paper will arouse some long overdue interest in matters pertaining to precision and accuracy in optic angle investigations.

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