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# PHOTOMETRIC MEASUREMENT OF ROTATION PROPERTIES

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## ABSTRACT

Photometric measurement of rotation properties is described and illustrated by measurements for breithauptite. Photometric measurements of $A_r$ can have 3 to 5 times the precision of visual measurements, and photometric measurements of $2\vartheta$ can be 5 to 10 times as precise as visual measurements. Photometric measurements of maximum precision are more time-consuming than visual methods and require special adaptation of the analyzer, but they have possible applications in measurement of standard values of rotation properties and in studies of crystal orientation. The photometric method may also be used with ordinary microscope equipment as a substitute for the visual method. Measurements then require no more time than visual methods and are equally precise.

## INTRODUCTION

Measurement of the rotation properties of an anisotropic mineral grain involves finding the settings of a rotating analyzer, or both the analyzer and a rotating mica compensator, at which illumination of the mineral is at a minimum. This paper describes a photometric method of finding these settings. The precision of the method is far greater than the precision of the visual methods described by Berek (1937), Hallimond (1951) and Cameron (1957, 1961). The method has potential applications to studies of crystal orientation in ores and metallurgical products.

## EQUIPMENT EMPLOYED IN PHOTOMETRIC MEASUREMENT

The equipment used consists basically of a Leitz Dialux-Pol microscope with trinocular tube, a Photovolt 520-M photometer, a Cooke-Wright slotted ocular, and a stabilizing circuit for the lamp of the vertical illuminator. The photomultiplier tube of the photometer is housed 12 inches above the ocular on an extension tube. The Cooke-Wright ocular, with analyzer, is mounted over the vertical tube of the trinocular. The circular scale of the ocular is modified by attachment of a graduated arc having a radius of 6 in., compared to the 1.5 in. radius of the unmodified ocular. The arc can be read to 0.01° against a special vernier.

The stabilization circuit for the light bulb, designed by W. F. Berry and J. H. Moses, of Bituminous Coal Research, Inc., is a combination of a Sola 6V, 10A constant voltage transformer and a capacitor. Bausch and Lomb 2nd order interference filters with auxiliary glass filters are used to provide essentially monochromatic light. The filter pass bands have halfwidths of 8 to 10 mμ. They are inserted in the extension tube; the observer therefore sees the specimen in white light.

For maximum sensitivity, a Leeds-Northrup Speedomax G 10mv. recorder is placed in series with the photometer galvanometer, and intensities are read in terms of deflections registered on the recorder chart.

## GENERAL CONSIDERATIONS

If a polished section of an anisotropic mineral is rotated on the stage to a 45° position, *i.e.*, to a position midway between two adjacent extinction positions, the incident polarized vibration is resolved into two components vibrating parallel to two mutually perpendicular vibration directions of the mineral. These directions now lie at 45° on either side of the polarizer vibration direction. If the mineral is transparent, the reflected portions of the two components differ only in amplitude, and the resultant reflected vibration is linear. If the mineral is absorbing, the general case for ore minerals, the components differ in both amplitude and phase. The reflected resultant is an elliptical vibration (Fig. 1).

The vibration ellipse is defined by the azimuth ($A_r$) of its semi-major axis a referred to the vibration direction of the polarizer and by b/a, the ratio of the length of the semi-minor axis to the length of the semi-major axis. The azimuth is determined as the angle $A_r$. The ellipticity is $\tan^{-1} b/a = \vartheta$, $2\vartheta$ being the phase difference for the two components of the ellipse at 45° to its axes. The purpose of photometric measurements is to determine, for a given mineral, the values of $A_r$ and $2\vartheta$. For a fuller discussion of the theory involved, reference may be made to Berek (1937) or Cameron (1961).

## PHOTOMETRIC MEASUREMENT

*General remarks.* Steps involved in photometric measurement of rotation properties correspond to those described by Cameron (1961, p. 120–136) for the visual method. First, the 45° positions of the mineral grain under investigation are determined. Second, the angle through which the analyzer must be rotated, from its zero position, to produce minimum illumination at each 45° position is measured. In Fig. 1, this angular rotation is $A_0OA_1 = A_r$. Third, the mica compensator is inserted, and the analyzer and mica compensator are rotated to find their respective positions of compensation ($A_1'$ and $G_1'$). At compensation, the mica compensator reduces the elliptical vibration to a linear vibration (Op) that is extinguished in the analyzer. The illumination of the mineral grain is now at a minimum.

*Determination of 45° positions.* The first step in finding the 45° positions

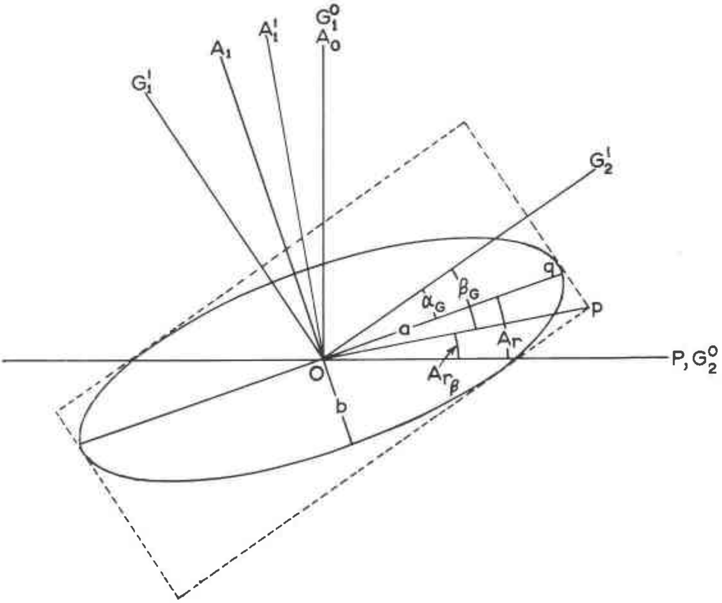

FIG. 1. Diagram showing an elliptical vibration produced by an anisotropic mineral at a 45° position, and the angular relations involved at compensation. Oq = a—semi-major axis of vibration ellipse; b—semi-minor axis; $A_0$—zero position of analyzer (vibration, direction $A_0O$ perpendicular to OP, vibration direction of polarizer). $OG_1^\circ$ and $OG_2^\circ$, slow and fast ray vibration directions, respectively, of mica compensator when compensator is at zero position. $A_1O$—vibration direction of analyzer perpendicular to Oq; $A_1OA_0 = A_r$; with mica compensator withdrawn from the system, the grain shows minimum illumination. $A_1'O$, $G_1'O$, and $G_2'O$, vibration directions of analyzer and slow and fast ray vibration directions of mica compensator, respectively, at compensation (minimum illumination with mica compensator in the system).

of a mineral grain is to locate its extinction positions as the stage is rotated. For this, we use the relation between intensity of illumination and the stage setting (Fig. 2) when an anisotropic mineral is rotated on the stage with the analyzer and polarizer exactly crossed, the polarizer vibration direction being parallel to the axis of rotation of the reflecting plate. Experiments indicate that the intensity curve is symmetrical about each position of maximum or minimum illumination within the error of measurement. Because the rate of change of intensity is small in the vicinity of the positions of maximum and minimum illumination, however, these positions cannot be measured directly with a high degree of accuracy. To determine extinction positions, pairs of points of equal intensity on the curve, such as y and y′, or x and x′, are therefore used. One extinction position must now be midway between point y, at a stage setting of 94°, and point y′, at a stage setting of 136°; the extinction position is therefore at 115°. Use of points x and x′ (90° and 140°, respectively) gives the same result. In the same manner, the other three extinction positions are determined.

In theory, extinction positions should be exactly 90° apart, but in practice this is not always the case, because of strain in the objective or elsewhere in the optical system, or because the analyzer is not quite perpendicular to the polarizer. An error in the setting of the analyzer (or polarizer) of a fraction of a degree is enough to cause the intervals between extinction positions to depart fom 90°. These departures pose no problem, because alternate extinction positions, carefully measured, are found to be 180° apart. If this condition is satisfied, then as Berek (1937) pointed out, the 45° positions are the bisectrices of adjacent pairs of extinction positions. For example, if the stage settings at four extinction positions are 0°, 86°, 180°, and 266°, the corresponding 45° positions are 43°, 133°, 223° and 313°.

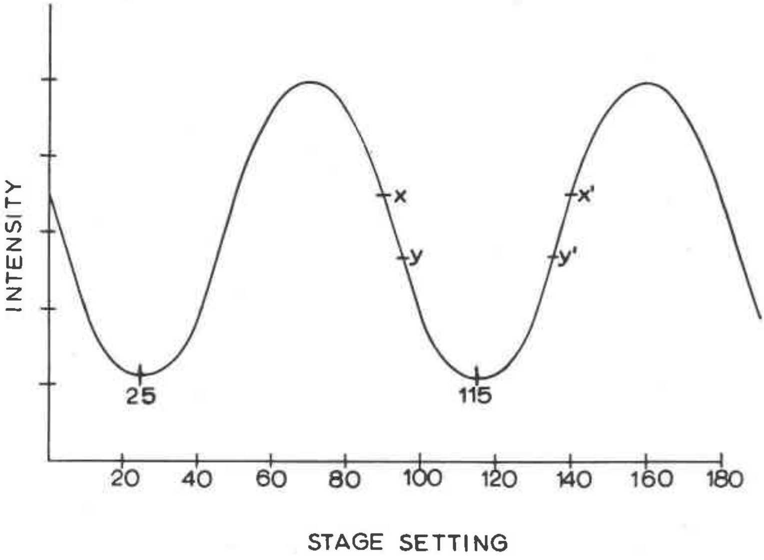

FIG. 2. Diagram illustrating the change in intensity as an anisotropic mineral is rotated on the microscope stage under crossed polars. Extinction positions of grain are midway between points of equal intensity, x and x′, and y and y′.

*Determination of* $A_r$. At any 45° position, with crossed polars, the intensity of illumination is a function of the azimuth of the semi-major axis of the reflected ellipse of vibration, or $A_r$. At two 45° positions 180° apart,

$A_r$ is clockwise from the polarizer direction, and a clockwise rotation of the analyzer, from its crossed position, equal to $A_r$, is necessary to produce minimum illumination. At the other two, also 180° apart but at 90° to the first two, $A_r$ lies counterclockwise from the polarizer vibration direction, and the position of minimum illumination is found by rotating the analyzer also counterclockwise. In either case, the angular rotation of the analyzer from its crossed position ($A_0$ in Fig. 1) to the position of minimum illumination gives the value of $A_r$.

The relationship between the rotation of the analyzer and illumination in the four 45° positions is given in Fig. 3. In the figure, $A_0$ is the analyzer setting perpendicular to the polarizer direction at 0°. $A_1$ and $A_3$ are the analyzer settings at minimum illumination for one pair of 45° positions. $A_2$ and $A_4$ are the analyzer settings at minimum illumination for the second pair of 45° positions. $A_1$ and $A_3$ are midway between x and x′, and $A_2$ and $A_4$ are midway between y and y′. The point of minimum illumination cannot be measured directly with sufficient accuracy because the change in intensity per degree of rotation of the analyzer is small near this position. In measurement, therefore, the points of minimum illumination are taken to be midway between points of equal illumination on the curves, such as x, x′ and y, y′. From the analyzer settings $A_r$ may be determined from the equation:

$$\frac{\frac{A_1 + A_3}{2} - \frac{A_2 + A_4}{2}}{2} = A_r \text{ (uncorrected)}$$

The observed value of $A_r$ must be corrected for the effect of the reflecting plate. When the polarizer vibration plane is parallel to the reflecting plate, the correction factor is less than 1.0 because the plate produces a rotation additional to that produced by the mineral. However, when the polarizer vibration plane is perpendicular to the reflecting plate, the correction factor is greater than one. The former case is more desirable as any error in setting the analyzer is reduced when the correction factor is applied. A correction factor is not necessary if a reflecting prism is employed, but it is extremely important that the prism be properly aligned. The procedure employed for determination of the correction factor is given by Cameron (1961). For the Leitz microscope used in this study, the correction factor is .79 at 589 $m\mu$.

The precision of measurement of $A_r$ by the photometric method is found to be ±0.02° to ±0.03°, whereas the precision of measurement by the visual method is never better than ±0.1° and in some cases is ±0.2° or more.

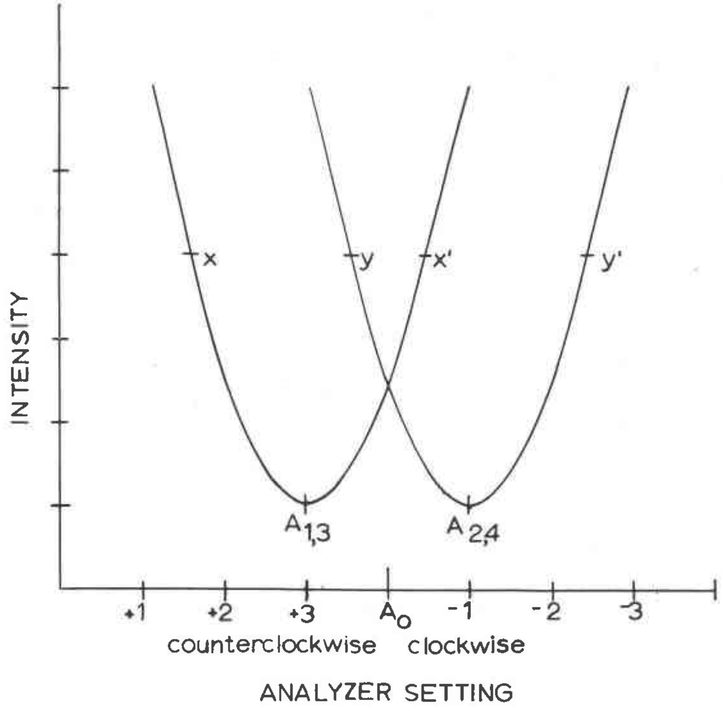

FIG. 3. Diagram illustrating change in intensity as analyzer is rotated over a mineral in each of the four 45° positions. Points $A_{1,3}$ and $A_{2,4}$ represent minimum illumination for 45° positions separated by 180° and are midway between points of equal intensity, x and x′, and y and y′.

*Determination of $2\vartheta$.* For visual determination of $2\vartheta$ the polarization figure as observed in monochromatic light is used. The mineral is brought to a 45° position. The analyzer and mica plate are inserted at their zero positions ($A_0$ and $G_1^0$, Fig. 1) and then rotated simultaneously until maximum blackness (minimum illumination) is produced at the center of the field. The figure is now a black cross. Viewed orthoscopically, the mineral grain is dark. The angular rotations of analyzer and mica plate from their zero positions to the positions of compensation are respectively $A_0OA_1'$ $(=A_{r_\beta})$ and $G_1'OG_1^0$. From the angular relationships between $A_r$, $G_1'OG_1^0$, and $A_{r_\beta}$, the angles $\alpha_G$ and $\beta_G$ (Fig. 1) are calculated as follows:

$$A_r \pm G_1'OG_1^0 = \alpha_G \tag{1}$$

$$A_{r_\beta} \pm G_1'OG_1^0 = \beta_G \tag{2}$$

The values of $\alpha_G$ and $\beta_G$ thus obtained must be corrected for the effect of the reflecting plate, using the factor employed in correcting the observed $A_r$. The value of $2\vartheta$ is now calculated from either

$$\tan 2\vartheta = \tan \Delta_G \sin 2\alpha_G, \quad \text{or} \tag{3}$$

$$\sin 2\vartheta = \sin \Delta_G \sin 2\beta_G, \tag{4}$$

in which $\Delta_G$ is the phase difference of the mica plate for the wave length of light employed. The mineral is now rotated 90° to a second 45° position, and the procedure is repeated.

In the same manner the value of $2\vartheta$ is calculated from settings at each of the other three 45° positions, and the final value is obtained as the average of the values for all four positions.

In applying formulas (1) and (2), depending on the mineral under observation, any of four possible cases may arise. One of these is shown in Figure 1. It is illustrated by breithauptite, for which calculations are given below. Calculations involved in the other cases are described by Cameron (1961, pp. 122–131).

The weakness of the visual method for determining $2\vartheta$ is that the eye is not very sensitive to different degrees of darkness, and experience has shown that reproducibility of results in general is poor. Values for $2\vartheta$ may have a precision as low as $\pm 0.5°$ or even $\pm 1.0°$, depending on the mineral.

The photometer is more sensitive than the human eye, but the rate of change of intensity is very small in the region of minimum illumination. However, this difficulty is avoided in photometric measurement because positions of minimum illumination are determined, as in the measurement of $A_r$, in terms of symmetrically disposed positions of equal intensity of illumination.

In photometric measurement, the mineral is brought to a 45° position. The analyzer is set at $A_1$, $A_2$, $A_3$, or $A_4$, whichever corresponds to $A_r$ for the particular 45° position of the mineral. The mica compensator is then inserted into the accessory slot. A curve similar to that of Fig. 3 represents the relationship between illumination and rotation of the mica compensator. The mica compensator is set midway between points of equal intensity on the steepest part of the curve. The midway point corresponds to a point of minimum illumination. Next, the analyzer is rotated while the mica compensator is fixed and a point of minimum illumination is defined for the analyzer. The mica plate and analyzer are employed alternately in this manner until the settings cease to change, indicating that compensation is obtained. The same procedure is followed for each of the other 45° positions. The settings of the analyzer and mica plate at each 45° position are now used to calculate $A_{r_\beta}$ and $G_1'OG_1^0$, and from these the values of $A_r$, $\alpha_G$ and $\beta_G$ are obtained from equations (1) and (2). The values of $2\vartheta$ calculated from the four sets of values of $\alpha_G$ and $\beta_G$ are averaged to give the final value of $2\vartheta$.

Experience indicates that values of $2\vartheta$ measured photometrically with the apparatus here employed have a precision of ±0.1°.

## Measurement of the Rotation Properties of Breithauptite

The results of measurements of breithauptite are presented as an example of the precision that can be expected from the photometric method. Comparable results have been obtained with other minerals. Measurements were made on a large grain of breithauptite, all parts of which showed extinction simultaneously. A Leitz 45× objective (.85 n.a.) was used in the experiment. The mica compensator employed has a phase difference of 16.2° at 589 mμ. The area of the grain exposed to the photomultiplier tube was approximately 20 microns. The area of measurement was free of visible imperfections.

The 45° positions are accurate to ±1.0°. The corresponding error in the determination of $A_r$ is ±0.01. Two independent sets of determinations of $A_r$ on a perfectly leveled polished section gave an uncorrected value of 3.55°±0.02° (589 mμ.), corresponding to a corrected value of 2.80° ±0.02°. Values for $2\vartheta$ were calculated from the angular relationships of the analyzer and mica compensator, shown diagrammatically in Fig. 1. For breithauptite, using corrected values of $A_r$, $A_{r_\beta}$, and $G_1'OG_1^0$,

$$A_r - G_1'OG_1^0 = 2.80° \pm 0.02° - 5.72° \pm 0.1° = 2.92° \pm 0.1° = \alpha_G$$

$$A_{r_\beta} - G_1'OG_1^0 = 2.71 \pm 0.02° - 5.72° \pm 0.1° = 3.02° \pm 0.1° = \beta_G$$

Using these values for $\alpha_G$ and $\beta_G$, and $\Delta_G$ for the mica plate in equations (1) and (2) gives $2\vartheta = 1.70° \pm 0.1°$.

## Requirements for Accurate Measurement

If measurements of the precision indicated above are to be obtained, the photometric system must meet the following requirements:

1. The intensity curves obtained by rotation of the stage (Fig. 2), rotation of the analyzer (Fig. 3), and rotation of the mica compensator must be symmetrical about points of minimum intensity. In the experiments made to date, this requirement is satisfied within the limits of error when the system is properly aligned.
2. The grain area measured; *i.e.*, the area from which light is received by the photomultiplier, must have its center at the center of the field of view.
3. The objective must be carefully centered.

4. The polished section must be carefully leveled.
5. The grain area measured must be free from pits, scratches, or other defects visible at the magnification employed and must show no internal reflections.
6. The photometer should be used only at wave lengths at which it has adequate sensitivity.
7. Both lamp and photometer circuits must have a high degree of stability.
8. All optical elements should be kept as dust-free as possible.

## Evaluation of the Photometric Method and Conclusions

Photometric measurement of rotation properties is at least 3 to 5 times more precise than visual measurement. Photometric measurement is free from the subjectivity of the visual method. Except for the specially adapted Wright slotted ocular, the apparatus used for measurement of rotation properties is the same as that employed in measurement of uniradial reflectivities. The chief disadvantage of precise photometric measurement is that it is time-consuming compared to visual measurement. The latter requires only a few minutes each for measurement of $A_r$ and $2\vartheta$, whereas precise photometric measurement of $A_r$ for a mineral grain may require as much as 15 minutes, of $2\vartheta$ as much as 30 minutes. The technique has value, however, for determining standard reference values of the rotation properties of anisotropic minerals or synthetic materials. In addition, it has potential applications in studies of crystal orientation in ores and metallurgical products, since $A_r$ is a function of crystal orientation. Finally, greater precision in measurement of rotation properties means that use of these properties in calculating indices of refraction can now be explored. We are currently investigating this matter.

For routine investigation of ore minerals, the photometric method has additional importance in that it can be employed, without a special ocular, as a substitute for the visual method. In this case, it is only necessary that the analyzer be equipped with a vernier so that its setting can be read to 0.1°, and that a rotating mica compensator be available. With this simple equipment, the precision of measurement of $A_r$ will be at least that of the visual method, and $2\vartheta$ can be determined with somewhat better precision than by the visual method. The time of measurement of $A_r$ is now reduced to a few minutes. The procedure of measurement is essentially the same as that described above.

## Acknowledgments

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