

USE OF THE SPINDLE STAGE FOR DETERMINATION OF
PRINCIPAL INDICES OF REFRACTION
OF CRYSTAL FRAGMENTS*

RAY E. WILCOX, *U. S. Geological Survey, Federal Center, Denver, Colorado*

ABSTRACT

A small crystal or crystal fragment mounted on the tip of a spindle in a suitable holder on the polarizing microscope stage may be oriented quickly and accurately for measurement of all its principal indices of refraction by the immersion method. Independent measurements or estimations of other properties, such as optic angle, optic sign, dispersion, pleochroism, and the relation of the indicatrix to cleavage or crystal faces may be made on the same fragment. Besides its usefulness in determinative mineralogy, this inexpensive device has much to recommend it as a teaching aid in optical crystallography, since, with its help, the student rapidly acquires facility and confidence in the application of interference figures and extinction angles to mineralogic and petrographic problems. The device used here is based on that of Rosenfeld (1950) but is of somewhat more rugged construction and provides for reading of angular rotations about the spindle axis. For mounting the fragment a mixture of ordinary carpenter's glue and molasses is recommended. Either orthoscopic or conosopic illumination may be used to orient the fragment for measurement of its principal indices. The orthoscopic procedure can give results to the limit of accuracy of the immersion method employed, while the conosopic procedure is usually faster and gives results sufficiently accurate for all but the most exacting work, besides providing additional optical information.

INTRODUCTION

The device described, for which the general name "spindle stage" is used here, is designed to rotate a crystal fragment in a matching liquid whose upper and lower plane surfaces remain perpendicular to the microscope axis. This, it should be noted, is different from the universal stage, which rotates not only the crystal, but also the plane surfaces of the liquid. Because of this and other mechanical features, the spindle stage has several advantages over the universal stage for study of individual crystal fragments by the immersion method: (1) The crystal may be viewed in *any* direction at right angles to the spindle, and *any* desired line in the crystal may be rotated into the plane of the microscope stage. Thus all three principal indices may be determined on the same fragment. (2) There are no corrections to be made to the angular rotations, and no appreciable disturbing effects due to high birefringence. (3) Either orthoscopic or wide-angle conosopic illumination may be used as desired. (4) It is simple and inexpensive.

The conosopic method suggested by Rosenfeld (1950) for direct determination of all principal indices of refraction of anisotropic crystal grains using a spindle is so effective and so instructive in itself that the

* Publication authorized by the Director, U. S. Geological Survey.

following extension, together with a brief review of other possible applications, is provided as a recommendation for its wider use in determinative and descriptive mineralogy as well as for teaching optical crystallography.

Briefly the procedure is as follows: The crystal fragment is attached with glue to the tip of a spindle (Fig. 1), which is turn is clamped over a glass window in a flat metal plate so that the grain lies in immersion liquid in a small cell formed by window, coverglass, and supporting

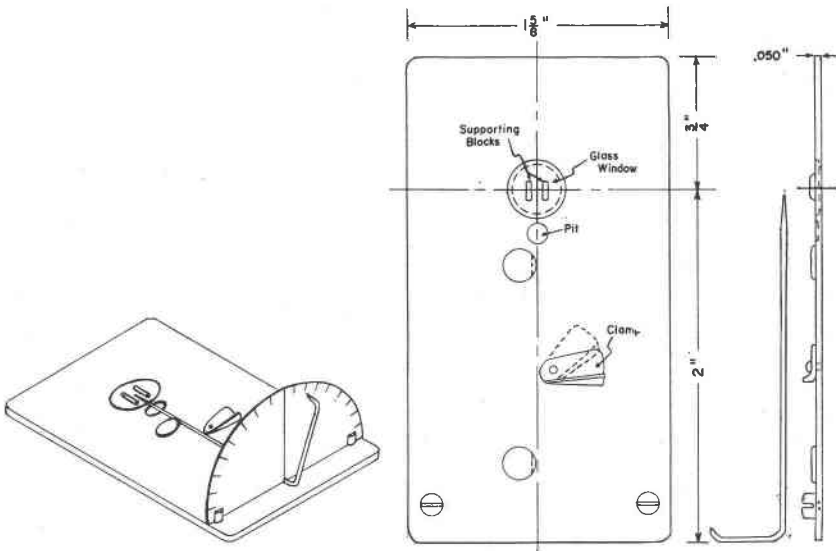


FIG. 1. Sketch of spindle stage, showing detachable plastic scale for reading angular rotations about the spindle axis. Cover glass is not shown.

blocks. To compare a principal index of refraction of the crystal with the immersion liquid it is only necessary to rotate the appropriate symmetry axis of the indicatrix into the plane of the microscope stage and the vibration plane of the lower nicol.

The method is suited for determining optical properties of homogeneous transparent anisotropic crystals or fragments of appropriate size, say between 0.03 and 0.3 mm. diameter for the form of the device illustrated here; no doubt somewhat larger or smaller grains could be handled by special design. In a uniaxial crystal both principal indices, and in a biaxial crystal all three principal indices, may be measured on the same grain. In case such complete data are not required for identification of an unknown or for locating a mineral accurately in its isomor-

phous series, the grain may simply be oriented for a desired principal index. In the common clinopyroxenes for instance it is sufficient to measure the beta index and the optic angle (which in many cases also may be measured on the same grain). In orthopyroxenes and olivine it is usually sufficient to measure α or γ . In micaceous minerals α , difficult to measure by ordinary methods, becomes tractable with this device, as do the principal indices of refraction of minerals with prominent oblique cleavages, such as the carbonates and amphiboles. Difficulties arise with minerals of strong selective absorption, such as the very dark micas and hornblendes, due to their abnormal interference figures, yet even some of these can be handled conoscopically by taking into account observable characteristics of the pleochroism (Schumann, 1948), or orthoscopically by working on thin wedge-shaped edges.

In case only a small amount of material is available, as sometimes happens in work with heavy mineral concentrates or products of microchemical tests, the complete set of optical properties may be measured on a single grain and, if desired, this grain retained attached to the spindle for future reference. Where repeated measurements are to be made on the same grain, the grain may be attached to and detached from the spindle at will. A simple modification of the spindle should permit relating x -ray and optical data of the same grain. The device is especially effective in the not uncommon situation in which the "unknown" is actually a mixture of two or more substances of similar optical properties, for identifications may be made from complete optical properties of each grain of a series.

The potential of the instrument under conoscopic observation is appreciated when one notes that the interference figure is a "directions-image," that is, each point in the field shows the behavior of light traversing the crystal between crossed nicols in a specific direction, and the position of the point is nearly the orthogonal projection of that direction. The cone of observation *in the crystal* using an objective of N. A. 0.85 is commonly 60° to 70° wide, depending on the refractive index of the crystal. By rotating the grain about the axis of the spindle, the number of directions that may be studied is greatly increased, and a wide choice of favorable orientations is made available for observation of crystallographic elements and for special optical tests. The directions-image cone of a 0.65 N. A. objective, although less broad than that of a 0.85 N. A. objective, is quite adequate for most work, and objectives of even smaller numerical aperture may furnish useable interference figures. Under orthoscopic illumination, on the other hand, the field is a projected image of the object itself, and as used here shows the behavior of light vibrating essentially perpendicular to the microscope axis. Hence, although a smaller number of directions is available in which the behavior of light in the crystal may be studied during rotation of the spindle, a more precise, if less direct, means of determining optical properties is furnished.

CONSTRUCTION OF THE SPINDLE STAGE

Possibly the first of the many rotating devices of this type was that of von Ebner in 1874 (Johannsen, 1918, p. 301). Others have been described by Vedeneeva and Kolotushkin (1934), Rawlins and Hawksley (1934), Wood and Ayliffe (1935), Bernal and Carlisle (1947), Rosenfeld (1950), Hartshorne and Stuart (1950, p. 215), Tatarskii (1951) and Hartshorne and Swift (1955). In addition Steinbach and Gibb (1957) provide for tilting the spindle axis as much as 30° from horizontal. Many more variants of this simple apparatus are possible for general use and for special problems. In working with several forms of the device I have been led to the one described below, essentially a modification of Rosenfeld's (1950) device, as a practical combination of ruggedness, ease of construction and effectiveness in problems of determinative mineralogy. The disadvantages of most previous devices inherent in the use of a drum or round ball for rotating the spindle have been avoided by bending the spindle into an L, the arm of which may be moved to rotate the crystal about the spindle axis. The resulting limit of 180° for angular rotation is no hindrance, for further rotation would merely reciprocate orientations already attained; rather it has the advantages of preventing inadvertent duplication of settings and of providing a pointer for reading values of angular rotation about the spindle axis. While such angular readings are not specifically required in the procedures suggested here for determination of indices of refraction, they do aid in returning quickly to previous settings. Angular readings are required for plotting relations of crystallographic and optical elements stereographically and for determination of optic angle by extinction characteristics.

As shown in Fig. 1, a stainless steel plate about 0.040 or 0.050 inch thick is bored to receive and seat a slightly thinner glass window so that the top surface of the glass, when cemented into its seat, is essentially flush with that of the plate. Two steel slugs are slightly undercut and soldered to the plate along one side of the center line of the spindle, and an undercut clamp is riveted to swing in against the other side of the spindle and hold it with the desired firmness. Two short lengths of the spindle stock are cemented to the glass window on either side of the center line of the spindle about a sixteenth of an inch apart to support the cover glass and form a cell of thickness equal to the diameter of the spindle.* A pit bored into the upper surface of the steel plate along the

* To accommodate the short working distance of the 0.85 N. A. objectives of American Optical and of Bausch and Lomb, material about 0.20 inch thick may be used to support the cover glass instead of the 1/32-inch material of the spindle stock. This of course decreases somewhat the maximum permissible size of crystal fragment and freedom of movement within the cell. "Cementyte A" of Schaar & Company serves to cement the supporting blocks to the window and to seal the glass window in its seat. A satisfactory expedient is the mixture of carpenter's glue and molasses described below for mounting the grain.

axis of the spindle just behind the window helps to break the capillary between spindle and plate and decreases creeping of immersion liquid along the shank of the needle. (A drop of water in the pit during operation further prevents the escape of immersion liquid.)

The spindle is made of a straight $2\frac{1}{2}$ -inch length of piano wire $1/32$ inch in diameter, tapered for about a quarter of an inch at the end but with a definite flat blunting of the extreme tip. The other end of the spindle is bent into a right angle just at the heel of the holder plate, as shown in Fig. 1, taking care not to bend the shank of the spindle, especially near the sharpened tip. It is convenient to have a half dozen or more of these spindles on hand. The small protractor scale is cut out of the 6-inch transparent plastic ruler no. W-5 of the C-thru Ruler Company of Hartford, Conn., and fits into the slots of two stainless steel machine screws in the heel of the base plate. (Care should be taken when using methylene iodide-sulfur immersion liquids of index greater than 1.7 as they soften this plastic after prolonged contact.)

MOUNTING THE GRAIN

For many beginners the task of fastening the grain to the tip of the spindle is the most difficult part of the procedure, but dexterity and confidence are usually quickly acquired with practice. The choice of adhesive is of primary importance, for it must remain tacky until contact has been made with the grain, yet should set rapidly thereafter and be insoluble in the index liquids and whatever cleaning fluid is used. Dental cement is suggested by Rawlins and Hawksley (1934) and dental "sticky wax" by Wood and Ayliffe (1935). Vedeneeva (1937) suggested the use of water glass, Kolotushkin (1940) suggested "sindetikon" "koloditikon" or "plain office glue," and Rosenfeld (1950) suggested beeswax, subsequently coated with waterglass to prevent solution in the index oils; Tatarskiĭ (1951) reports success with freshly melted Rochelle salts, and also states that melted sugar moistened with water may be used.

I have found that several common mucilages (Lepage, Sanford, Ross) are satisfactory for mounts at room temperature. Most dependable, however, has been a mixture of about 4 parts of common water-soluble carpenter's glue (such as Lepage's "Liquid Glue") and one part of crude ("black-strap") molasses. This mixture has the desired tackiness and setting time, will hold a grain firmly and indefinitely in the immersion oils commonly used in mineralogical laboratories, and is affected by acetone only after prolonged contact. Liquids of the methylene iodide-sulfur series above 1.7 appear to decompose somewhat more rapidly than usual in contact with this adhesive, and it is therefore advisable to test the Becke effect immediately after their introduction into the cell, as of course it is

also advisable to do in any immersion mount of these liquids. For immersion liquids made up of water solutions (Bryant, 1932), this adhesive would be unsuitable, and an appropriate one may be sought among the many water-insoluble adhesives now available.

In detail the mounting procedure is as follows: A drop of the adhesive is placed on a flat surface and the tip of the spindle is just touched to it in order to bring up the minimal quantity that will hold the crystal fragment. (To ensure tackiness, Kolotushkin (1940) recommends touching the glue-tipped spindle to water before bringing it to the desired grain.) The spindle is brought down to touch the desired grain so that it will become attached as nearly as possible in line with the spindle axis. For platy or elongate grains a choice may be made for orientation of the grain in respect to the spindle axis, although this is not usually necessary because all appropriate positions for measuring the principal indices may be reached regardless of the relation of spindle axis to grain elongation or optic indicatrix. For substances of strong birefringence, dispersion, or pleochroism a smaller grain is generally easier to work with optically than a larger one. For mounting very small grains, or for picking a particular type of grain from a mixture, it is helpful to use a binocular microscope or hand lens. In some cases spreading the grains on a soft electrostatically charged surface, such as rubber or plastic, may facilitate securing the grain in a desired position on the spindle point. Limited adjustment of the position of the grain may be made by nudging the grain with another spindle under the binocular before the adhesive sets. The glue of course should not mantle the grain, and, in case the grain is to be removed for remounting, it is essential to rinse off the glue completely, as a film of glue will prevent index comparisons.

After allowing the glue to harden, the spindle is clamped in position and the protractor scale inserted. A small coverglass or fragment of coverglass is placed across the supporting blocks on the window and a drop or two of a chosen immersion liquid introduced into the open-ended cell so formed, where it is held by surface tension. The device may be fastened to the microscope with the stage clips or directly in the jaws of a mechanical stage. The grain may be centered in the usual manner, or if desired, under a high power objective with the Bertrand lens inserted and the tube raised.

ORIENTATION AND INDEX DETERMINATION

Conoscopic Procedure

As pointed out by Rosenfeld (1950), a symmetry axis of the indicatrix lies horizontal NS when an isogyre lies EW across the center of the cono-

scopic field,* and there are three such settings of spindle axis and microscope stage for a biaxial crystal, corresponding to the three principal indices of refraction. With a high power objective (say 0.65 or 0.85 N. A.), crossed nicols and conoscopic illumination, the crystal is rotated on the spindle and microscope stage until an isogyre has been maneuvered into a position symmetrically along the EW crosshair, that is, so that the EW crosshair divides the isogyre brush into two mirror-image halves. Examples of such interference figures as seen with a 0.65 N. A. objective and Bertrand lens are sketched in Fig. 2. Upon reaching the position at which the isogyre lies symmetrically along the EW crosshair, the readings of spindle and stage scales may be noted for convenience in returning later to this setting. Then, under orthoscopic illumination with the analyzer removed, the relation between index of crystal and of liquid is noted. Restoring crossed nicols and conoscopic illumination, the same procedure is repeated to reach a second and third position at which an isogyre lies symmetrically along the EW crosshair, taking care not to duplicate a position on the microscope stage at 180° from one already found.

With this preliminary information, often also a knowledge of optic sign, approximate optic angle, dispersion, and pleochroism observed at strategic points in the orientation procedure, one is ready to determine specifically the indices of refraction. The initial liquid may be drawn off conveniently with a fragment of filter paper or blotting paper placed against the spindle at the edge of the cell, after which the cell is rinsed with acetone or with a drop or two of the appropriate new liquid (being careful to avoid contamination of the liquid in the bottle) and again drawing off with the absorbent paper. This may be repeated once or twice to completely flush out the old liquid. Then with the new liquid in the cell the indices of liquid and crystal are again compared and noted at the respective oriented positions. The procedure is then repeated with other chosen liquids until all principal indices have been determined.

Accuracy of orientation is aided by attention to the following details: For interference figures, the substage assembly should be centered, the Bertrand lens adjusted symmetrically to the crosshairs, and of course the nicols adjusted parallel to the crosshairs. It will be noted on many microscopes that the readability of an interference figure with a Bertrand lens is much improved by slightly raising the tube of the microscope with the fine-focus drum until the interference figure fills the whole field and becomes smooth. The same technique may be used in confirming the optic orientation of the very edge of the grain on which the Becke line

* Throughout the lower nicol is assumed to be NS, as in most microscopes of U. S. manufacture.

effect is to be observed. In a microscope without a useable Bertrand lens, the isogyre is seen with the pinhole ocular (thus without crosshairs) and may be brought by eye sufficiently close to a symmetrical EW position for much determinative work. If the grain migrates due to excentric mounting it must be recentered by slight shifting of the device on the microscope stage. If angular readings of the microscope stage are to be used in plotting, as in the optic angle method mentioned below, the alignment of the needle axis must be preserved during such shifts, and this may be guaranteed by placing the device in a mechanical stage. Comparison of index between liquid and crystal using the central illumination effect is best made with a low power objective, say a 10X or 20X, rather than the high power objective used for the interference figure.

The basis of the centered EW isogyre as a criterion of correct orienta-

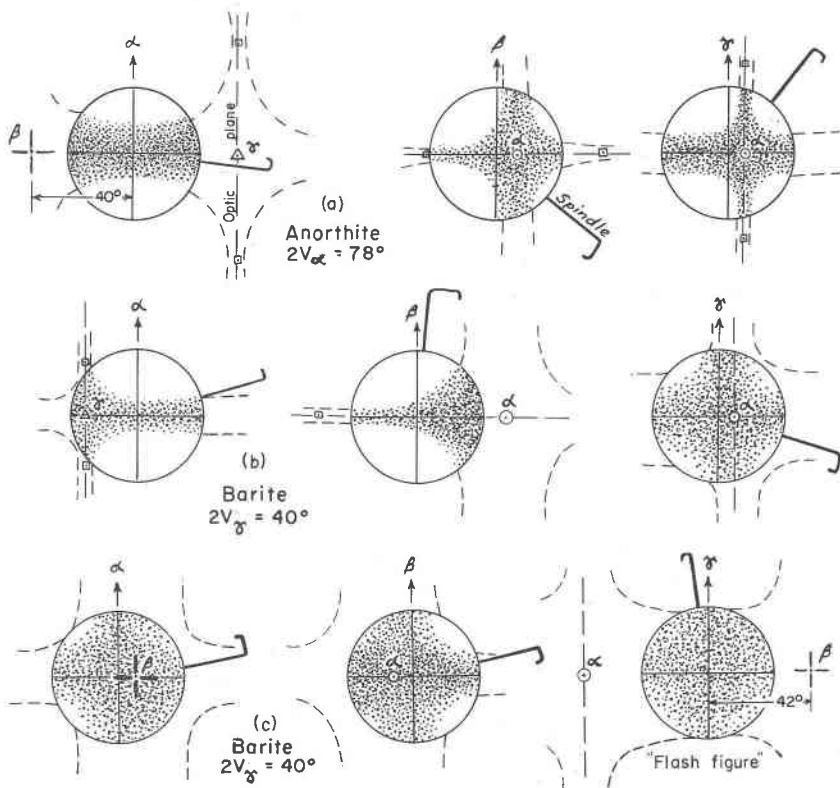


FIG. 2. Conoscopic fields of view (in circles), using .65 N. A. objective and Bertrand lens, with crystals in positions for measuring principal indices of refraction. (a) Anorthite, $2V_{\alpha} = 78^{\circ}$. (b) and (c) Barite, $2V_{\gamma} = 40^{\circ}$.

tion may be demonstrated by skiodrome diagrams (Becke, 1905a; Johannsen, 1918, p. 424, 429 ff) or the alternative diagrams proposed by Kamb (1958). The centered EW isogyre indicates that an optical symmetry plane is vertical and EW and that therefore the normal to this plane, a principal axis of the indicatrix, is horizontal and NS. This is an extinction position under orthoscopic observation, and, with the analyzer removed, the light observed vibrates parallel to this symmetry axis and has the corresponding principal index of refraction. Generally at the other extinction position on the microscope stage, a wave is passed having a nonprincipal index of refraction, α' or γ' . It may be shown that centered interference figures represent special cases as follows: a centered optic axis figure furnishes β in all positions of the microscope stage; a truly centered bisectrix figure furnishes β at one extinction position and either α or γ at the other; a truly centered optic normal figure (flash figure) furnishes α and γ at respective extinction positions. In working with apparent obtuse bisectrix or optic normal figures, however, it should be kept in mind that the bisectrix or optic normal may actually diverge appreciably from the microscope axis, as implied by Becke (1905a) and as discussed below for crystals of small optic angle, and in this case only one of the extinction positions furnishes a principal index.

Uniaxial crystals could be oriented in the same manner as biaxial, but, except for checking that the crystal is indeed uniaxial, it is only necessary to orient to obtain the flash figure, for in that position the indices of both the ordinary and principal extraordinary waves may be measured at their respective extinction positions on the microscope stage. In this orientation also the optic sign may be determined by use of the accessory plates and Lommel's Rule, which states that the optic axis lies in the quadrants from which the brushes of the flash figure leave the field upon slight rotation of the microscope stage.

An often overlooked characteristic of biaxial crystals of small to moderate optic angle is that *when the acute bisectrix is horizontal*, regardless of the position of the optic plane otherwise, *the interference figure behaves much like the flash figure of a uniaxial crystal*. Thus one is unable to distinguish whether a particular figure at hand is that of an optic normal section, obtuse bisectrix section or *any intermediate section*. In orienting such crystals, two general situations may be considered here: (1) that in which the acute bisectrix is at a large angle to the spindle axis, and (2) that in which the acute bisectrix is at a small angle to the spindle axis. These situations are illustrated in Figs. 2b and 2c, respectively, for different mounts of barite of $2V_\gamma = 40^\circ$.

(1) If the crystal fragment happens to be mounted so that the acute bisectrix is at a large angle to the spindle axis, as in Fig. 2b, the orienta-

tion may be carried out much in the usual manner. During rotation about the spindle axis the acute bisectrix or an optic axis may become visible in the conoscopic field and at once reveal the biaxial character. If both optic axes lie outside the conoscopic field the biaxial character may often be recognized by a difference in behavior of the isogyres near the respective oriented positions. Mounted thus, there are only two settings of the spindle arm, in addition to the setting for the "flash figure," in which an isogyre may be placed strictly symmetrically along the EW crosshair. With a similarly mounted uniaxial crystal, on the other hand there are any number of such settings of the spindle arm before the setting for the flash figure is reached. This distinction becomes more difficult of course as the optic angle approaches zero. Having reached the spindle setting for the "flash figure," one may locate the acute bisectrix by analogy to Lommel's Rule for uniaxial crystals and place it in the NS position for measuring its index of refraction.

(2) If the acute bisectrix of the crystal with small optic angle lies at a small angle to the spindle axis, the "flash figure" is again obtained when the acute bisectrix is horizontal, and generally broad and diffuse isogyres are obtained when the other two symmetry axes are horizontal. A fragment of barite mounted with γ at 22° to the spindle axis serves as an example, and Fig. 2c represents the conoscopic fields observed for NS positions of α , β , and γ . Repeated trials with this mount under conoscopic illumination gave a range of spindle arm settings about 5° on either side of the settings for α and β that were indicated by stereographic plotting according to the "extinction curve" method of Joel and Garaycochea (1957). It is important to note here that, in spite of the $\pm 5^\circ$ range of uncertainty of the settings for α and β , only small errors in their principal indices could result (see for instance the discussion in the later section on "precision"). Although the "flash figure" of the setting for gamma of this mount of barite behaved quite normally—that is, the dark field separated into two isogyres that swept rapidly out of the field from the quadrants into which the acute bisectrix had been moved by slight rotation of the stage—the stereographic plot indicated that in reality the optic normal was 42° from parallelism with the microscope axis. Thus, with the acute bisectrix NS, γ may be measured accurately, but at the other extinction position only a non-critical index is measured. If the situation justifies it, more exact settings of α and β than obtained above may perhaps be obtained by remounting the grain or another grain with the hope or design that the acute bisectrix will be at a large enough angle to the spindle axis to enable orientation in the manner outlined for case (1), as well as direct observation of the size of the optic angle. Or, if a sufficiently precise immersion index facility is available, such as single- or

double-variation, the principal indices of this barite or other crystals of low birefringence, mounted as in Fig. 2c, could be determined with greater accuracy using the simplified orthoscopic procedure described below.

Orthoscopic Procedure

Separate orthoscopic procedures for bringing the crystal into correct positions for measuring its principal indices of refraction have been outlined by Vedeneeva and Kolotushkin (1934; see also Vedeneeva, 1937; Kolotushkin, 1940) and by Joel (1950; see also Joel and Garaycochea, 1957). Both require stereographic constructions, and that of Vedeneeva and Kolotushkin is subject to error in the setting for β . A simplified orthoscopic procedure, which removes the tedium of graphical plotting and furthermore is accurate for all three principal indices, is as follows:

Rotate the microscope stage to make the spindle axis EW and rotate *about the spindle axis* to extinction. Then by incremental rotations about the spindle axis and subsequent slight corrections of extinction by rotation of the microscope stage and observation of the Becke effect (changing liquids as necessary), determine both the maximum index and minimum index encountered during the full 180° traverse about the spindle axis. One of these extreme values is β and the other is an extreme principal index, α or γ . Rotate now on the microscope stage to make the spindle axis NS and rotate about the spindle axis to extinction. Observing the refractive index, there are two possibilities: (1) If the index is lower in this new position than before, the extreme principal index determined before was γ , and α will be found as the minimum in a new series of incremental rotations about the spindle axis from this new extinction position. (The maximum index in this series will be a nonprincipal index, α' , and need not be determined.) (2) If the index is higher in the second extinction position, the extreme principal index determined before was α , and γ may be determined as the maximum index of this new series of extinction positions. In this procedure it is not necessary to read the microscope stage or spindle arm settings except as reference positions for convenience in returning to them later. It is good practice of course to confirm the correctness of setting for each principal index by observing the interference figure in the oriented position, as described above for the conoscopic method.

The basis for this procedure rests on the Biot-Fresnel law and on the relations between indicatrix (fixed on the spindle axis), microscope stage axis, and plane of the polarizer. As the circular sections of the indicatrix are perpendicular to the optic axes, the Biot-Fresnel law may be stated in the manner used by Joel and Garaycochea (1957) quite simply as fol-

lows: For a given position of the crystal the vibration directions of light bisect the traces of the circular sections of the indicatrix in the plane of the microscope stage. As shown in Fig. 3, these vibration directions are 90° apart, and extinction results when, by suitable rotation of the microscope stage, they are made parallel to the directions of light vibration in the polarizer and analyzer. A small rotation of the crystal about the spindle axis to a new setting will result in a new pair of vibration directions, which again may be rotated on the stage into parallelism with the nicols to restore extinction.

These extinction positions, plotted stereographically, as in Fig. 4, trace out two curves whose corresponding points at a given setting of the spindle arm are 90° apart. Each curve is confined to its respective pair of dihedral angles formed by the circular sections. Duparc and Pearce (1907) plotted such extinction curves for representative crystallographic zone axes—analogue here to the spindle axis—and these showed that the curve of extinction positions passing through the zone (spindle) axis also passes through one of the bisectrices, α or γ . The curve of extinction positions passing through a point of the great circle of the zone (spindle) axis also passes through the other bisectrix and the optic normal, β . These curves were studied in detail by Joel and Garaycochea (1957), who named them the “polar” and “equatorial” curves, respectively. Referring to Figs. 3 and 4, it will be seen that by rotating on the microscope stage to make the spindle axis EW, then rotating about the spindle axis, one will reach the extinction position E in Fig. 4. This is on the equatorial series of extinction positions, and following this series by small rotations on the microscope and spindle axes, one must pass through the positions in which β and one of the bisectrices, here γ , become horizontal.

Recognition of the principal indices is made possible by the fact that they must be either the maximum or minimum values of indices encountered on their respective series of extinction positions. The extreme principal index α is of course the smallest value of index in the extinction series of lower indices, while γ is the largest value encountered in the extinction series of higher values. Again with reference to Figs. 3 and 4, and starting with the horizontal NS position of γ , the indices at successive extinction positions decrease but never reach β until the optic normal itself is horizontal NS at which position β may be measured. This is the minimum index on this extinction series, for with further rotation about the spindle axis the extinction position passes into the opposite dihedral angle and the index at successive extinction positions becomes progressively greater, until it again passes through the maximum, γ (assuming the spindle may be rotated that far). Had α been the extreme principal index in the equatorial series, similar reasoning could be applied to dem-

onstrate that β would be the maximum of index encountered upon 180° of rotation about the spindle axis.

A special situation, illustrated in Fig. 4*b*, occurs when the biaxial crystal fragment happens to be mounted with one of its optic axes nearly or exactly perpendicular to the spindle axis. As before, two sets of extinction positions at 90° from each other may be followed, but as the optic axis approaches parallelism with the microscope axis the definition of the two

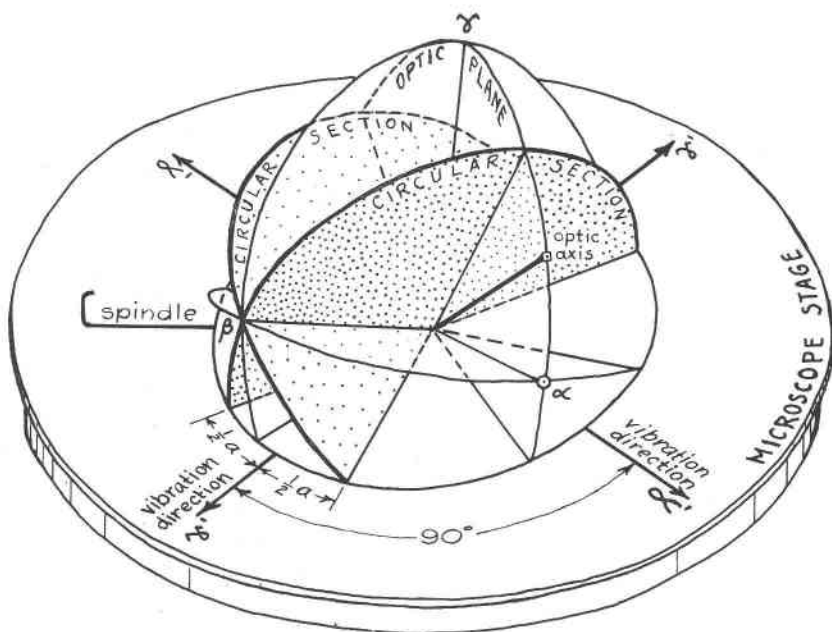


FIG. 3. Vibration directions in a biaxial crystal with the symmetry axes of the indicatrix inclined to the microscope stage and spindle axis.

extinction positions fades, and the crystal remains equally dark on complete rotation of the microscope stage. As the optic axis is advanced beyond parallelism with the axis of the microscope the extinction positions once more become definite, apparently a continuation of the same trends (although in reality, as seen in Fig. 4*b*, the curves bend sharply and follow their prescribed course close along the circular section). Fortunately no practical problem arises, for the one apparent series of extinction positions includes α as its minimum and γ as its maximum index, and β may be measured at that setting in which the distinction between extinction positions is lost, that is, when the optic axis coincides with

the microscope axis. Here also a nearly centered optic axis figure may be observed under conoscopic illumination, and if desired the true course of the extinction curves may be followed by the interference figure. With crystals having marked dispersion a disturbing behavior similar to that just described may occur if an optic axis is merely in the neighborhood of perpendicularity to the spindle axis, and therefore only becomes roughly coincident with the microscope axis. It becomes ad-

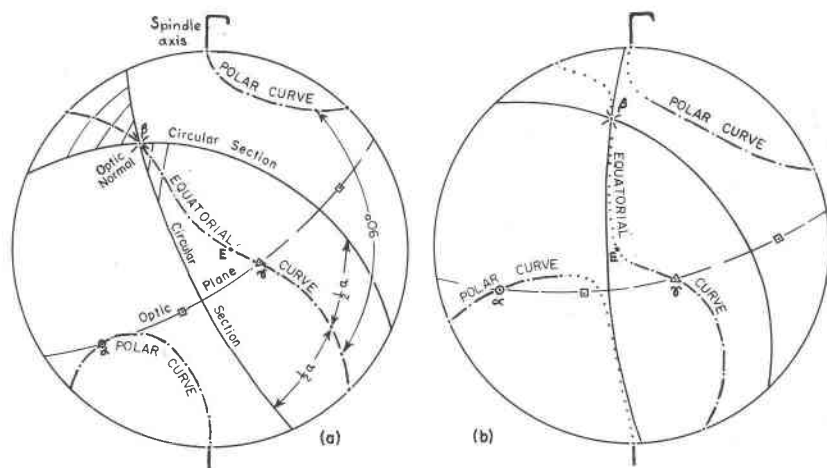


FIG. 4. Locus of extinction positions of anorthite, $2V_{\alpha} = 78^{\circ}$, plotted stereographically in relation to the spindle axis. (a) Spindle axis at 23° from a circular section of the indicatrix. (b) Spindle axis at 2° from a circular section.

vantageous in this case to follow the two series of extinction positions with monochromatic illumination.

Other special situations arise when a symmetry axis of the indicatrix happens to coincide with the spindle axis, recognized orthoscopically by the fact that no adjustment is necessary on the microscope stage to restore extinction as the grain is rotated about the spindle axis. Again no particular problem is presented, for when either of the bisectrices coincides with the spindle axis, measurements of the maximum and minimum indices encountered in the equatorial series and the one index of the polar series as before furnish the three desired principal indices. When the optic normal coincides with the spindle axis, both optic axes may be seen to pass through the field during rotation about the spindle axis. In the case of optically uniaxial crystals, the values of indices encountered in the equatorial series of extinction positions do not vary. If the crystal is biaxial with low optic angle and low birefringence, the range of index

through the equatorial series of extinction positions might be so small as to go unnoticed in ordinary immersion procedures, and interference figures would be necessary to demonstrate its biaxial character.

PRECISION

Errors in index of refraction of an anisotropic crystal determined by the immersion method arise mainly from two sources, one the inherent limitations in the sensitivity of the Becke line or its allied effects, and the other the misorientation of the optical indicatrix. With sodium light, small intervals of spacing of index liquids, and close attention to temperature corrections, the error from the first source may probably be held to $\pm .001$. With double variation techniques under favorable conditions it may be held perhaps to $\pm .0002$ (Emmons, 1943) or with additional special techniques perhaps to $\pm .0001$ (Saylor, 1935, Micheelsen, 1957).

In the orthoscopic procedure outlined above it seems clear that the chances of errors in principal index due to misorientation are small, since in following it carefully one cannot avoid passing through each of the principal indices as maximum or minimum values of index in one or the other series of extinction positions. The errors of determination of indices therefore reduce mainly to those of the particular immersion method employed. (It is worth noting that this technique may also be used on the universal stage for confirming or refining the accuracy of the principal indices of refraction obtained at oriented settings.) The orthoscopic procedure of Joel and Garaycochea (1957) for finding the positions of α , β and γ likewise appears to be very precise, provided errors do not enter during the plotting and reading off of the proper settings. The orthoscopic procedure of Vedeneva (1937) and Kolotushkin (1940) may be made to furnish precise values for α and β , but the setting for β and thence the value of the index β is subject to some uncertainty because it is derived from the orientations of α and γ determined at very flat minima and maxima of index.

In the conoscopic procedure the chances of error due to misorientation would appear greater than in the orthoscopic procedure described above, since it depends on the positioning of sometimes broad and diffuse isogyres. On the basis of tests described below, however, it is concluded that the actual error of index of refraction due to misorientation in most cases is smaller than that of all but the most precise immersion methods, so that it usually is not a matter of great concern. This may be illustrated by the case in which the optic plane of the crystal is perpendicular to the spindle axis. The index then varies with angular distance from α or γ according to the equation of the ellipse as written out in Fig. 5.

To enable general application, it is convenient to represent error in

index due to misorientation as a percentage of the birefringence, and on this basis the two curves of Fig. 5 are plotted against the angular divergences from the correct setting. It is seen that the percentage error is very slight for the first degree or so of misorientation and increases at an increasing rate for succeeding increments. Taking as an example a crystal of extreme birefringence, say .100, and placing the optic plane in the NS position with γ at 2° from its correct horizontal position, a very exact measurement of the index of refraction would give a result differing from true γ by 0.13 per cent of the birefringence of that NS section, or an index of refraction that is .00013 too low. With γ placed 4° off, the index measured would be .00054 too low. Similarly a divergence of 4° from the correct setting for α would lead to an index about .00044 too high. It should be noted that, since in this example the crystal has been mounted with the optic plane perpendicular to the spindle, these represent the maximum errors that might be encountered due to these amounts of angular misorientation of this crystal. The error in index for

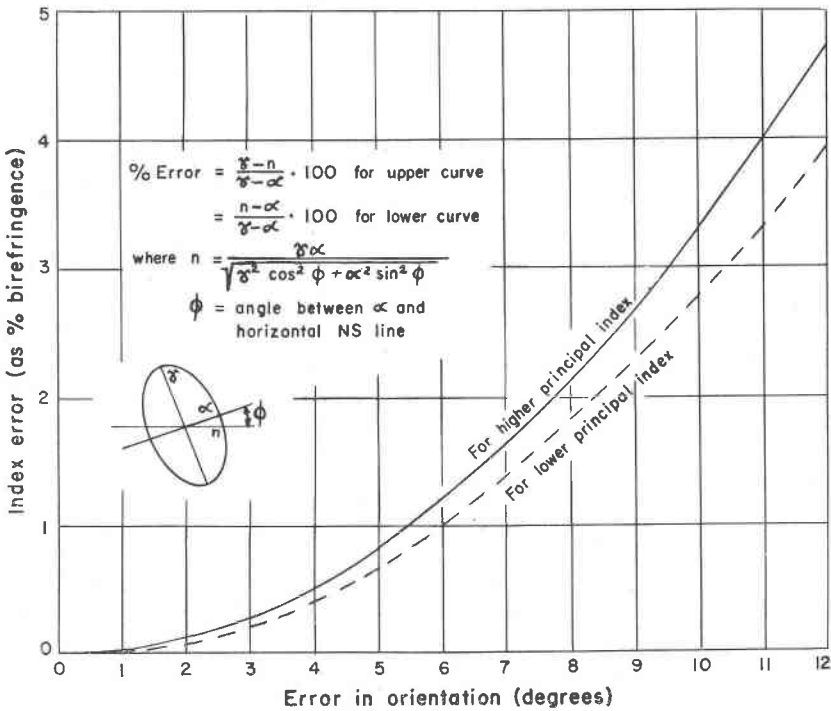


FIG. 5. Error in principal index of refraction due to misorientation in the optic plane, expressed in terms of percent of total birefringence.

the same amount of misorientation in any other cross section of this crystal would be less, usually much less, since the erroneous settings follow courses of smaller variation of index than that of the optic plane. Likewise for other crystals of smaller birefringence the analogous errors in index would be proportionately less.

The question of what amount of angular error may be expected in orienting a grain by the conoscopic method cannot be answered precisely, but it is instructive to compare such orientations with those obtained on the universal stage. For this a special miniature model of the spindle stage similar to that described at another place (Wilcox, 1959) was made as follows: A hole was bored in a thin 1×2-inch brass plate and a slot cut to accommodate a spindle made of a small sewing needle of diameter equal to the thickness of the plate, and a plate-size coverglass was cemented to the bottom of the plate. After mounting the crystal grain on the spindle it was placed in the slot with a few drops of immersion liquid, and a large coverglass was slid over the cavity, taking care not to trap bubbles. The device was then clipped to the microscope stage and the grain oriented conoscopically, as described earlier, to a position in which an isogyre lay symmetrically along the EW crosshair. Thereupon the spindle was secured with a small drop of glue at the end of the slot, and, after the glue had hardened, the plate-and-spindle assembly was transferred with due care to a 5-axis universal stage on another microscope. There the orientation of the grain was carried out independently by rotations about the axes of the universal stage without disturbing the spindle. A measure of discrepancy between the results of the two methods was given by the reading on the scale of the appropriate horizontal axis of the universal stage (here the Inner East-West axis). In all cases the reading of the vertical axes—the microscope stage for the device alone and the Inner Vertical axis for the universal stage—turned out to be within a degree of each other, about the precision with which the transfer could be made.

The results of such tests on six crystalline substances of moderate to large optic angle are summarized in Table 1. Of the eighteen determinations, the largest discrepancy was 4°, for α of a strongly colored hedenbergite of optic angle (+)2V=58°. A discrepancy of 3° was found in the setting for γ of sucrose of optic angle (-)2V=47°. For all remaining settings the divergence was 2° or less. No minerals of optic angle smaller than 40° were tested, since for these, accurate distinction between the obtuse bisectrix and optic normal becomes increasingly difficult with either the spindle stage or universal stage. Since it is not known what the true orientations should be in the cases tested, we cannot tell whether those of the universal stage are any closer than those of the spindle. The

striking feature, however, is that they do correspond so closely, and to me this suggests that both instruments are capable of about the same precision, and that it is fairly good.

If it be assumed for comparative purposes, however, that the universal

TABLE 1. ANGULAR DIFFERENCES BETWEEN ORIENTATIONS OF OPTICAL SYMMETRY AXES USING SPINDLE STAGE AND USING UNIVERSAL STAGE

Crystal, birefringence, and 2V	Symmetry axis horizontal NS	Appearance of isogyre on EW crosshair	Difference (universal stage reading on IEW axis)
Olivine .037 -84°	α	broad	1°S
	β	narrow, optic axis at west edge of field	0°
	γ	broad, Bx_α east of center	2°S
Montecellite .020 -73°	α	} Bx_0 centered	2°S
	β		1°S
	γ	broad	0°
Hedenbergite .025 +58°	α	broad	4°N
	β	broad, optic axis beyond west edge of field	2°S
	γ	broad, Bx_0 east of center	0°
Sucrose .032 -47°	α	"flash figure" east of center	1°S
	β	narrow, optic axis just west of center	1°N
	γ	broad	3°N
Wollastonite .014 -41°	α	"flash figure"	1°S
	β	narrow, optic axis just beyond west edge of field	1°N
	γ	moderately narrow isogyre	1°S
Barite .012 +40°	α	broad	1°S
	β	Bx_0 east of center	0°
	γ	"flash figure"	1°S

stage orientation was correct, the error of index may be estimated. Had the presumed 4° error of orientation of α of the hedenbergite been in the optic plane, the index α as determined on the spindle device would have been about .0001 too high. Since it was not in this plane, but in some non-planar figure of much less severe variation, the error would amount to something appreciably less than .0001. Similarly, the presumed 3° error in the setting for γ of the sucrose implies that the error of index would be something less than .0001. Those of the other tested crystals would be even less. Such amounts of error are dwarfed by the inherent

errors in routine work using the Becke effects and hence may be considered negligible. For crystals of higher birefringence, the generally narrower isogyres permit more precise orientation and hence should keep the error of index correspondingly low.

From this it appears that the precision of determination of a principal index at a setting established conoscopically on the spindle would not be much different than that on the universal stage, and, although the absolute angular accuracy of neither method has been determined, it is probably very good for practical purposes. Should extreme accuracy be required, the results may be refined to the limit of accuracy of the particular immersion method by use of the orthoscopic procedure. Nevertheless, one should not fail at the same time to take advantage of the very direct information concerning the other optical properties of the crystal available from interference figures.

ADDITIONAL APPLICATIONS

The spindle device can be used to determine other properties and may be of help in any problem requiring viewing the fragment in different positions. Both indicatrix and crystallographic elements, for instance, may be plotted stereographically in relation to the spindle axis and microscope axis using readings of the spindle scale and microscope stage taken at the horizontal NS position of each *linear* element. Thus a symmetry axis, α , β , or γ is horizontal NS when an isogyre is placed symmetrically along the EW crosshair, and the pole of a crystallographic plane (cleavage or crystal face) is horizontal NS when the plane is rotated into a vertical EW position.

The optic angle may be determined on the same grain as the indices of refraction if the indicatrix axes are oblique to the spindle axis. (Only exceptionally will the optic plane be perpendicular to the spindle axis, thus in position to measure the optic angle directly in the manner of Wood and Ayliffe, 1935). One first plots the extinction curves and positions of the indicatrix axes by the method of Joel and Garaycochea (1957), then chooses plausible optic angles and tests them by Fresnel constructions. This leads quickly to the correct optic angle, whose constructed extinction positions fall on the observed extinction curve. Although based on the same principle as the Berek-Dodge indirect method with the universal stage (Emmons, 1943, p. 58), this spindle stage procedure does not require the rotational corrections which make the Berek-Dodge method and that of Joel and Muir (1958*b*) difficult and sometimes fruitless for crystals of high birefringence. The method furthermore appears to have greatest sensitivity with optic angles near 90° , just the ones most difficult to measure directly on the universal stage. A more precisely con-

structed spindle scale than used here, together with refinements in establishing exact extinction positions, would seem necessary to achieve the full accuracy of this method. The spindle stage may of course also be used to obtain favorable orientations for determination of optic angle by the conoscopic methods described by Becke (1905*b*), Tertsch (1940), Johannsen (1918, p. 418, 467) and recently by Kamb (1958), and precisely determined principal indices furnish a calculated value of optic angle (Wright, 1951).

I have used the double-variation technique to a limited extent with a temperature-control cell of the Vigfusson (1940) type, modified to hold a spindle.* The results of this work were highly satisfactory and, together with considerations of accuracy discussed above, lead me to conclude that the double-variation technique is quite feasible for use with the spindle stage. Further, a miniature of the spindle device may be constructed very simply for use on the universal stage (Wilcox, 1959) and enables rotation of the grain into positions for direct measurements of all three principal indices of refraction as well as increasing the possible cone of observation of the grain for determination of other optical and crystallographic properties.

Dispersion of the principal indices may be found using the spindle stage by precise determinations of each principal index of refraction for different wavelengths. Dispersion of the optic axes and bisectrices may be estimated in the usual manner from the appearance of the interference figures in white light, or the dispersion can be explored quantitatively with variable monochromatic light by interference figures and by tracing the extinction positions at different wavelengths.

An inexpensive microrefractometer is provided by mounting a known crystal with its optic plane perpendicular to the spindle axis (Wood and Ayliffe, 1935). The index may be calculated from the equation of the ellipse, using the angle of rotation from α or γ to the position of match with the unknown liquid. Actually with proper calibration, it should not be necessary to make the optic plane perpendicular to the spindle axis, and a uniaxial would serve as well as a biaxial crystal.

As a teaching aid in optical crystallography the spindle stage with a few judiciously mounted crystals enables the student to familiarize himself with a wide range of interference figures, to see their interrelations, and thence to put to good use the variety of off-center figures he will encounter in working with thin sections and random immersion mounts. The optical behavior of a crystal under conoscopic illumination may be

* This cell was constructed by Lyman Nichols, Fort Collins, Colo., according to specifications.

compared directly to that under orthoscopic illumination, and the operation of the Biot-Fresnel law, which is so fundamental in applied crystal optics, may be convincingly demonstrated. The simplicity of the spindle device makes it within the reach of many teaching laboratories that could not afford more elaborate rotating devices, and within the capability of the student to construct himself if needed later in his career.

ACKNOWLEDGMENTS

I am indebted to colleagues in the U. S. Geological Survey for helpful suggestions and discussions based on experience with various models of this device, to I. J. Mittin for translations of the Russian literature, to Elmer Wehrly for construction of spindle stages as illustrated in Fig. 1. and to B. F. Leonard. Constructive criticisms of an early draft of the manuscript were received from Prof. R. C. Emmons and Prof. R. M. Gates of the University of Wisconsin.

REFERENCES

- BECKE, F., 1905a, Die Skiodromen. *Tschermaks Min. Petr. Mitt.*, **24**, 1-34.
 ———, 1905b, Achsenwinkelmessung aus der Hyperbelkrümmung. *Tschermaks Min. Petr. Mitt.*, **24**, 35-45.
- BERNAL, J. D., AND CARLISLE, C. H., 1947, A simple stage goniometer for use in connexion with X-ray crystal analysis: *Jour. Sci. Instruments, London*, **24**, 107.
- BRYANT, W. M. D., 1932, Optical properties of some derivatives of lower aliphatic alcohols and aldehydes: *Am. Chem. Soc., Jour.*, **64**, 3758-65.
- DUPARC, F., AND PEARCE, F., 1907, Ueber die Auslöschungswinkel der Flächen einer zone: *Zeit. f. Krist.*, **42**, 34-46.
- EMMONS, R. C., 1943, The universal stage: *Geol. Soc. America, Mem.* **8**, 1-205.
- HARTSHORNE, N. H., AND STUART, A., 1950, Crystals and the polarizing microscope: 2d edition, Arnold, London, p. 1-473.
- HARTSHORNE, N. H., AND SWIFT, P. M., 1955, An improved single-axis rotation apparatus for the study of crystals under the polarizing microscope: *Royal Microscope Soc., Jour.*, **75**, 129.
- JOEL, N., 1950, A method to determine the indicatrix of small crystals: *Mineral. Mag.*, **29** (1950-52), 206-214.
- JOEL, N., AND GARAYCOCHEA, ISABEL, 1957, The "extinction curve" in the investigation of the optical indicatrix: *Acta Cryst.*, **10**, 399-405.
- JOEL, N., AND MUIR, I. D., 1958, New techniques for the universal stage. II. The determination of 2V when only one optic axis is accessible: *Mineral. Mag.*, **31**, 878-882.
- JOHANNSEN, ALBERT, 1918, Manual of petrographic methods: McGraw-Hill, New York, 1-649.
- KAMB, W. B., 1958, Isogyres in interference figures: *Am. Mineral.*, **43**, 1029-1067.
- KOLOTUSHKIN, A. G., 1940, Ismereniê glavnykh pokazatelei prelomleniâ mikrokrystallov metodom vrashchiûuschcheisîa igly [Measurement of the principal indices of refraction of microscopic crystals by the rotating needle method]: *Trudy Vses. Nauchno-Issled. Inst. Mineral. Syr'ia*, fasc. **165**, 56-66.
- MICHEELSEN, HARRY, 1957, An immersion method for exact determinations of refractive indices: *Meddel. Dansk Geol. Foren.*, **13**, 177-191.

- RAWLINS, F. I. G., AND HAWKSLEY, C. W., 1934, A cell for refractivity measurements on minute crystals: *Jour. Sci. Instruments, London*, **11**, 282-284.
- ROSENFELD, J. L., 1950, Determination of all principal indices of refraction on difficultly oriented minerals by direct measurement: *Am. Mineral.* **35**, 902-905.
- SAYLOR, C. P., 1935, Accuracy of microscopical methods for determining refractive index by immersion: *Nat. Bur. Standards, Jour. of Research*, **15**, 277-94.
- SCHUMANN, HILMAR, 1948, Über den Einfluss relativ starker selektiver Absorption auf konoskopischer Bilder: *Neues Jahrb. Min., Monatshefte*, Abt. A, für 1945-48, 100-6.
- STEINBACH, BETTY J., AND GIBB, T. R. P., JR., 1957, Device for orienting small crystals under the microscope: *Analytical Chemistry*, **29**, 860.
- TATARSKII, V. B., 1951, K praktike metoda vrashchaŭschcheisia igly [Application of the rotating needle method]: *Zapiski Vses. Mineral. Obshchestva*, ser. 2, pt. 80, p. 293-296.
- TERTSCH, HERMANN, 1940, Becke's Achsenwinkel Bestimmung des Hyperbelkrümmung unter Verwendung des Schraubenmikrometer-Okulars: *Zentrbl. f. Min., Geol. u. Pal. Abt. A*, Jahrg. **1940**, 166-174.
- VEDENEVA, N. E., 1937, Laboratornoe rukovodstvo po immersionnomu metodu. [Laboratory manual for the immersion method]: *Trudy Vses. Nauch.-Issled. Inst. Mineral. syr'ia*, fasc. no. **124**, 1-68.
- VEDENEVA, N. E., AND KOLOTUSHKIN, A., 1934, Determination of the main refractive indices of crystal grain by means of a rotating needle: *Trudy Inst. Prikladnoi Mineralogii, Moscow*, no. **61**, 19-20 (English summary p. 26-27).
- VIGFUSSON, V. A., 1940, Equipment for the double variation method of refractive index determination; 1, An improved cell; 2, Variable temperature control apparatus: *Am. Mineral.* **25**, 763-766.
- WILCOX, RAY E., 1959, Universal stage accessory for direct determination of the three principal indices of refraction: *Am. Mineral.*, **44**, 1064-1067.
- WOOD, R. G., AND AYLIFFE, S. H., 1935, An instrument for measuring the optical constants of small crystals: *Jour. Sci. Instruments, London*, **12**, 194-196.
- WRIGHT, F. E., 1951, Computation of the optic angle from the three principal refractive indices: *Am. Mineral.*, **36**, 543-556.

Manuscript received March 31, 1959.