

Results from a McCrone Spindle Stage Short Course, a New Version of EXCALIBR, and Building a Spindle Stage

Mickey E. Gunter^{*1}, Robert Weaver², Bryan R. Bandli³, F. Donald Bloss⁴, Stanley H. Evans⁵,
and Shu-Chun Su⁶

¹Department of Geological Sciences, University of Idaho; ²McCrone Research Institute; ³MVA, Inc.; ⁴Department of Geological Sciences, Virginia Tech; ⁵Tuscon, AZ; ⁶Hercules Inc., Research Center

KEYWORDS

Spindle stage, EXCALIBR, 2V, dispersion, polarized light microscopy, crystallography

ABSTRACT

This paper discusses the results of a 3-day short course in spindle stage techniques recently offered at the McCrone Research Institute. The 14 students enrolled had levels of experience with spindle stage and optical crystallography which varied from novice to expert, but all were soon able to collect research quality crystallographic data on crystals less than 100 μm using a simple, home-made poster-board spindle stage and the newest version of EXCALIBR, EXCALIBRW. EXCALIBRW is Windows compatible, free, and has three major improvements over the previous version: 1) easier data entry, 2) a refinement method for the calculation of the reference azimuth, and 3) a more robust numerical solution to the Joel equations for determining the orientation of the optical indicatrix. Advantage 3) now allows the solution of extinction data for crystals in less favorable orientations. Also, during the course, the students built their own spindle stages from poster-board.

INTRODUCTION

The spindle stage is, mechanically, a very simple accessory for the polarized light microscope (PLM) which is used to identify and characterize single crystals. The spindle stage is a one-axis rotation device, onto which a single crystal is mounted. The rotation axis is parallel to the plane of the microscope's circular, rotating stage, thus greatly increasing the user's

ability to locate and orient a crystal's crystallographic directions. The spindle stage first appeared as the "Klein's rotating apparatus" before 1900 (1). However, in 1959 Wilcox (2) simplified the design to include only a spindle, a metal plate, and a removable protractor. This simplification and Wilcox's exposition of the use of the spindle stage kindled a renewed interest in spindle stage methods as the device was more readily available than ever before. Figure 1 depicts several examples of the newer spindle stage models.

The spindle stage provides the ability to determine the orientation of a crystal's indicatrix so that its principle refractive indices (i.e., α , β , γ , or ϵ , ω) can be measured by the immersion method. Measurement is performed after the spindle axis and microscope stage have been properly rotated to place a principle refractive index parallel to the plane of the microscope stage and parallel to the polarizer's privileged direction. Traditionally the orientation of the crystal was found by observations of interference figures, in a manner similar to that used to find correctly oriented crystals in a grain mount. This technique became known as the "conoscopic method" (See Bloss (3, 4)). The "orthoscopic method" was soon developed which only required measurement of extinction positions at different spindle axis settings. These extinction positions could be measured in either conoscopic or orthoscopic view. The extinction positions were then plotted stereographically and graphical methods were used to locate the three (biaxial) or two (uniaxial) principal refractive index directions (3, 4). The main advantage of the orthoscopic method during the mid 1960's, was it could be used on grains too small to yield an interference figure.

Although data collection by the orthoscopic method is fairly simple, the stereographic method for plotting the data and determining the principal re-

* Moscow, Idaho 83844

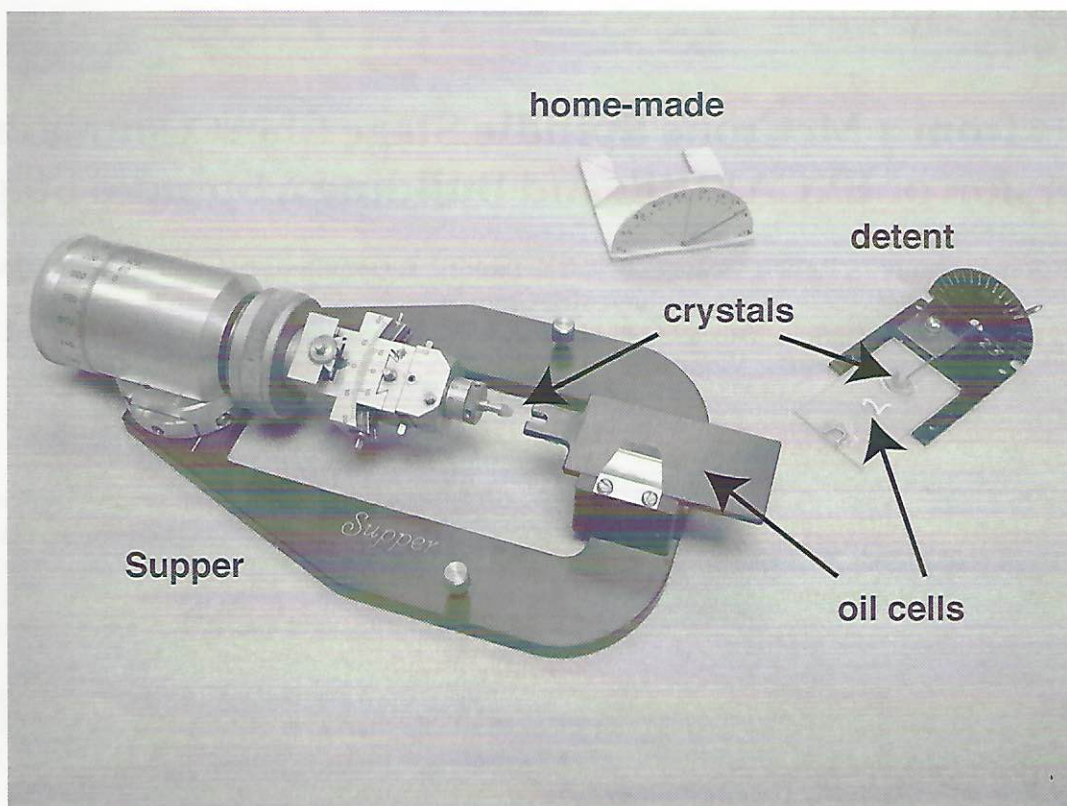


Figure 1. Photograph of three different types of spindle stages and their associated oil cells. The Supper model uses an X-ray goniometer head / brass pin combination to hold the crystal in place. The detent and home-made models can use a needle of some type (e.g., tungsten needle, sewing needle or straight pin) to mount the crystal.

fractive indices were tedious, time consuming, and contained an uncertainty of at least $\pm 1.0^\circ$ due to manual plotting error. In the early 1980's, Bloss and coworkers (3) refined an existing mathematical algorithm that related extinction directions to the optic axes of the biaxial indicatrix. These efforts resulted in the computer program, EXCALIBR, which took as its input extinction positions measured on the microscope stage obtained at different angular settings of the spindle axis. The program then calculated 2V (see Table I for definitions of acronyms and symbols) and the microscope stage and spindle stage settings required to orient the crystal's three principal refractive index directions in the plane of the microscope stage and parallel to the lower polarizer.

Like many mathematical routines, the algorithm in EXCALIBR produces better results given certain boundary conditions of the input data, which in this case relates to the orientation of the crystal's indicatrix (i.e., how the crystal was mounted on the spindle's tip). Good orientations produce a stable mathematical model and low uncertainty, while less favorable ori-

entations produce low quality results, or no solution. With the advent of EXCALIBR extinction data sets could be collected and processed in a fraction of the time required for the stereographic method and with increased accuracy and data statistics.

In the past 30 years, Bloss and coworkers have performed many optical studies of minerals using the spindle stage methods (See Bloss (4), Preface). Recently, Gunter (5) reviewed past optical studies that used the spindle stage and described ongoing research including the utilization of the spindle stage with other analytical methods (e.g., the orientational dependence of X-ray absorption spectra as measured with synchrotron radiation). Also, refinements have been made to EXCALIBR, first by Gunter et al. (6) and next by Bartelmehs et al. (7). These refinements were mainly focused on easier data entry and more robust algorithms to increase the accuracy of the results when the crystal is in a less favorable orientation.

One goal of this paper is to describe the most recent update of EXCALIBR, termed herein as EXCALIBRW because it is Windows compatible, and

Table I. Definitions for Symbols and Acronyms Used in this Paper.

E or Es	the angle between an extinction direction and the spindle stage the angle a vibration direction makes with the spindle axis
Ms	the microscope stage reading
Mr	the reference azimuth
S	the spindle stage reading
AB	the acute bisectrix (AB), as the bisector of the acute angle formed by the two optic axes,
OB	the obtuse bisectrix (OB), as the bisector of the obtuse angle formed by the two optic axes
ON	the optic normal (ON), is the normal (the cross product) of the two optic axes
N	the large refractive index
n	the small refractive index
2V	the angle between (the dot product of) the two optic axes

to provide a general overview of collecting and analyzing spindle stage data. Additional discussion is devoted to determining if a crystal is in a favorable orientation. This paper also describes how to build a simple spindle stage. Many of the results presented herein originate from student data sets collected during a 3-day short course taught in July 2003 at McCrone Research Institute in Chicago, Illinois. The authors were the instructors with the exception of Evans who debugged and provided EXCALIBRW for use in the course.

COLLECTING AND PLOTTING EXTINCTION DATA

The spindle stage uses a polar coordinate system represented by S and E angles. The S angle is read directly from the spindle stage dial (Figures 1 and 2) and is the coordinate for the plane within the crystal that is currently parallel to the microscope stage (i.e., perpendicular to the incident light). E is the angle between the spindle axis and any vector with its origin at the end of the spindle. Physically, E is the angle between a vibration direction (or any direction within the crystal) and the spindle axis (Figure 2). An appropriate rotation on S will place a principal vibration direction parallel to the plane of the microscope stage, and then the appropriate rotation of the microscope stage (i.e., the Ms angle), will rotate that direction parallel to the lower polarizer. An extinction data set for a crystal typically includes the recording of an extinction position (i.e., Ms values) for every 10° of S, from 0°

to 180°. Although the user is not required to understand the mathematical calculations involved in an EXCALIBR run, it is beneficial for the expert to understand how the Ms and S coordinates for the crystal's principal vibration directions (X, Y, Z) are calculated. The newest version of EXCALIBR outputs both numerical and graphical results. To understand the graphical results, which can be useful for refining crystal orientation, a basic understanding of stereographic projections is required.

Figure 2 illustrates both the polar (S, E) and Cartesian coordinate (x, y, z) systems used by EXCALIBR. The program performs all the calculations in the Cartesian system and outputs results in the polar system. The Cartesian system places +x in the plane of the microscope stage and horizontal (i.e., E-W), +y is also in the plane of the microscope stage, but vertical (i.e., N-S), and +z is perpendicular to the stage, coinciding with the light path of the microscope. S angles represent rotations about the spindle axis and are read from the spindle stage's dial. Ms values represent rotations of the microscope stage and are mathematically related to the E angles, as discussed below.

Before this relationship can be developed, the reference azimuth, Mr, must be defined. Mr is the microscope stage setting that places the spindle axis to coincide with the +x direction with its tip pointing to the west. This is the orientation shown in Figure 2. Also in Figure 2, Mr = 0°. If Mr = 0°, the relationship is simplified. However, the value of Mr depends on how the spindle stage was mounted on the microscope stage. A precision of 0.1 to 0.2° is required for accurate work,

and it is very difficult to mount the spindle stage that precisely. Also, there might be mechanical limitations for mounting the spindle stage. For these reasons, Mr must be determined once the spindle stage is mounted on the microscope by use of extinction measurements as follows:

$$Mr = (Ms0 + Ms180) / 2 \quad (\text{Equation 1})$$

where Mr is the reference azimuth, Ms0 is the extinction position (Ms) obtained at $S = 0^\circ$, Ms180 is the extinction position obtained at $S = 180^\circ$, but is measured by rotation in the opposite direction from Ms0.

Step 1) Mr is first estimated visually by reading the vernier scale on the microscope stage when the spindle is oriented parallel to the E-W microscope direction.

Step 2) The spindle stage is set to the visually estimated Mr0.

Step 3) Ms0 is determined.

Step 4) The microscope stage is reset to the estimated Mr, and Ms180 is found.

The Mr value calculated in Equation 1 should be a refined value of the estimated Mr. Care must be taken that the refined Mr is not 90° , or a multiple of 90° , from the estimated Mr, which is very possible. If this occurs, add or subtract 90° increments so the estimated and refined values agree.

Before equations can be given to relate Ms to E, one must consider the type of microscope stage being used. Because microscope stage graduations may increase in a clockwise (cw) or counterclockwise (ccw) direction, depending on the manufacturer and vintage, it is necessary to have two equations which relate Ms to E, one for each stage type.

Stage graduations increase in counterclockwise direction (most modern stages):

$$E_s = Ms - Mr \quad (\text{Equation 2a})$$

Stage graduations increase in a clockwise (cw) direction:

$$E_s = Mr - Ms \quad (\text{Equation 2b})$$

where E is the angle between an extinction direction and the spindle stage, Ms is the microscope stage reading causing extinction, and Mr is the reference azimuth. Note that, if Mr is 0° , then $E = Ms$ for the ccw stage. For a cw stage, when $Mr = 0^\circ$, $E = -Ms$. Thus a calculation is still required to convert Ms to E angles to produce stereographic plots.

The stereographic plotting technique for the extinction angle data is similar to other plotting systems used in the geosciences, except the stereonet is oriented slightly differently and when plotting there is no need to rotate the overlying tracing paper (if plotted by hand). In Figure 2 (modified from Gunter and Twamley (8)), an equal angular stereonet (i.e., a Wulff net) is shown in the appropriate orientation for plotting S and E values. Recall that an S angle is the number of degrees required to rotate any plane within the crystal parallel to the microscope stage. Thus, $S = 0^\circ$ is the microscope stage; correspondingly increasing S values are printed on the great circles of the stereonet. The E angles are plotted on the small circles of the stereonet and represent the angle a vibration direction makes with the spindle axis. These are printed on the circumference of the stereonet. For example, two perpendicular vibration directions, N (the slow ray) and n (the fast ray), are shown on the tip of the spindle stage. The plane in which they are located has been rotated 40° (i.e., they are in the $S = 40^\circ$ plane), and they are placed in the plane of the page (i.e., microscope stage). N then makes an angle of 20° with the spindle axis ($E = 20^\circ$ for N) and n must make an angle of 110° ($90^\circ + 20^\circ$) with the spindle axis ($E = 110^\circ$ for n). To plot N on the stereonet, locate the $S = 40^\circ$ great circle (shaded grey in Figure 2) and the $E = 20^\circ$ small circle (shaded grey in Figure 2); N plots at their intersection, and n plots 90° from N on the $S = 40^\circ$ plane at $E = 110^\circ$. Next,

Figure 2. Schematic showing the conventional directions for the axes of the Cartesian and spherical coordinate systems (modified from Gunter and Twamley (8)). The directions for the two coordinate systems are provided: 1) a Cartesian system (x, y, z) and 2) a polar system (S, E). The right-handed Cartesian system has the $+x$ direction parallel to the spindle axis direction. The $+y$ direction is perpendicular to x . Both x and y are within the plane of the microscope stage and the $+z$ direction is perpendicular to the projection/microscope stage, plotting in the middle. The S-angle is measured on the spindle stage dial as shown on the right. The S-angles are represented by great circles in the stereographic projection. The line representing the $S = 40^\circ$ plane is shown in bold. The S-angle defines a plane within the crystal that is parallel to the microscope stage. The E-angle is measured on the microscope stage, and is represented by small circles on the stereographic projection. The line representing the $E = 20^\circ$ small circle is shown in bold. The E-angle is the angle between the spindle axis and a vibration direction in the crystal.

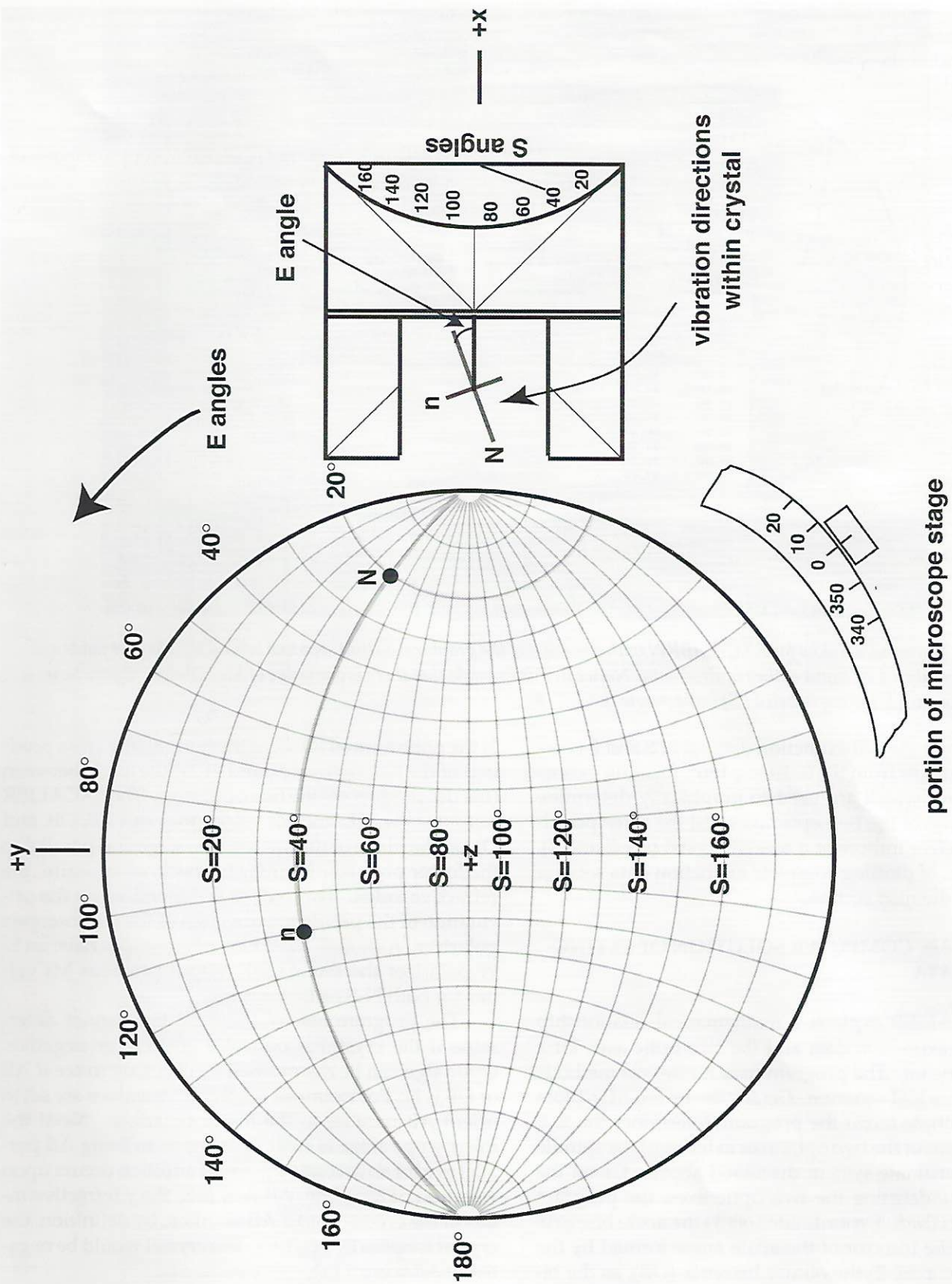


Figure 2. See caption on facing page.

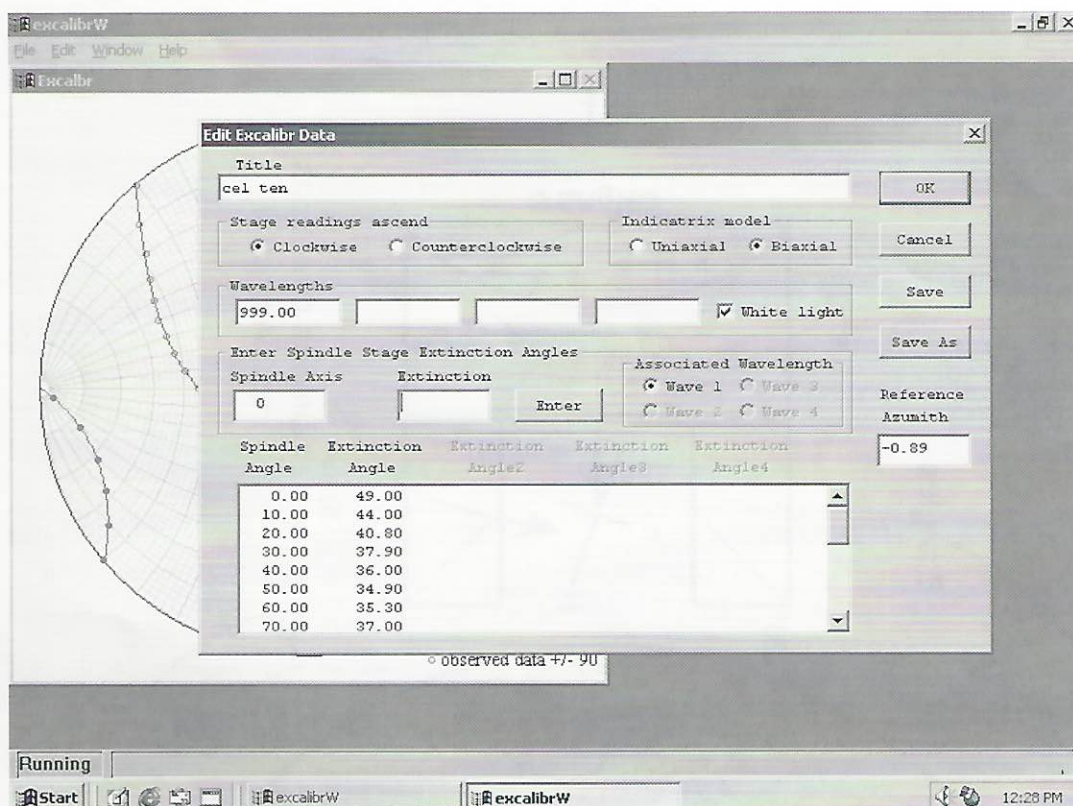


Figure 3. The input window for EXCALIBRW with a portion of the graphical output window behind it. The text fields and buttons are labeled for input of the required data. Notice in this example that there is one value of M_s (labeled "Extinction Angle") for each S setting (labeled "Spindle Angle").

one can plot the full extinction data set of S and E coordinates values from $S = 0, 10, \dots, 180^\circ$, forming extinction curves which are used to graphically determine the location of the two optic axes and the three principal refractive indices of a biaxial crystal (see 3 and 4). Examples of plotting complete extinction data sets are given in the next section.

EXCALIBR: COMPUTER SOLUTION OF EXTINCTION DATA

EXCALIBR exploits a mathematical relationship between extinction data and the two optic axes for a biaxial crystal. The program uses numerical methods to solve the Joel equation. Details can be found in Bloss (3). In simple terms the program determines the S , E coordinates of the two optic axes in terms of the spindle stage coordinate system discussed above. Given the directions defining the two optic axes, the program calculates the S , E coordinates of: 1) the acute bisectrix (AB), as the bisector of the acute angle formed by the two optic axes, 2) the obtuse bisectrix (OB), as the bisector of the obtuse angle formed by the two optic axes,

3) the optic normal (ON), as the normal (the cross product) of the two optic axes, and 4) $2V$, the angle between (the dot product of) the two optic axes. The EXCALIBR output is the S , E , and M_s values to orient AB, OB, and ON in the plane of the microscope stage and parallel to the lower polarizer in order to check, or measure, the refractive index. This output is dependent on the orientation of the privileged direction of the microscope's polarizer. Although most new microscopes have an E-W polarizer the EXCALIBR output provides M_s values for both N-S or E-W setting.

The program also outputs $2V$, but cannot determine if the crystal is optically positive or negative. Optic sign can be determined by checking to see if AB or OB is N. For example, the S and M_s values are set to orient AB parallel to the lower polarizer. Next, the microscope stage is rotated 45° so as to bring AB parallel to N of an accessory plate. If addition occurs upon insertion of the plate, AB is N (i.e., the γ refractive index of the crystal) and if AB is γ , then, by definition, the crystal is optically positive. The crystal would be negative if AB were $n(\alpha)$.

Using EXCALIBR:

In many ways using EXCALIBR, especially the new Windows based version, is the easiest aspect of using the spindle stage. After opening EXCALIBRW, click the "file" menu and select "New *.dat". Figure 3 shows the input window (with a data set). There are input fields for data entry and radio buttons for specific input selections. The title field accepts any text and is typically used to identify the sample. Next, the microscope stage type and mathematical models (i.e. uniaxial or biaxial) are selected. In general "biaxial model" should be selected unless you are sure the crystal is uniaxial. There will be more discussion on the model selection in the "Crystal Selection and Orientation" section of this paper. The fields for "wavelength" are used to specify the wavelength(s) of the light source(s). If white light (i.e., polychromatic) is used, then "999" should be entered. EXCALIBRW can accept up to four wavelengths. When more than one wavelength of light is used, EXCALIBRW calculates if movement of the optical directions occurred (i.e., determines the dispersion of the crystal). The recognition of dispersion may help determine the crystal system of a biaxial crystal. Dispersion is discussed more fully for the labradorite data presented in the "Results" section. The program will calculate a refined reference azimuth, Mr, based on the input data, but an approximate reference azimuth should be input. This input Mr value prevents the program from calculating a Mr value which may be 90° in error, or some multiple of 90°, as explained above. Lastly, the "Extinction" field is used to enter the Ms values for a given "spindle axis" (i.e. S) value. The S values automatically start at "0" and increase by 10° after each Ms value is entered. One must click the "enter" button to accept the typed Ms value. The S and Ms pairs will accumulate in the window below. If a mistake is made in entering an Ms value, double click on that line in the lower window and edit it in the input "extinction" window. Once all of the data are input, it is a good idea to click "Save" or "Save As". When "OK" is selected, the input "edit EXCALIBR data" window vanishes and a stereographic plot of the data and its graphical solution are shown. To view the numerical results, go to the "Edit" menu and select "Output." Selecting "Data" from the "Edit" menu will return you to the input window.

Figure 4 shows the numerical results from EXCALIBRW and Figure 5A shows the corresponding graphical results. At the top of the numerical results are the title and refined Mr value. Note in this case the refined Mr value is -0.89°; this would correspond to a microscope stage setting of 359.11°. The next section

MEG10

Experimental Treatment ID number = 999.0

Refined Reference Azimuth, Mr = -0.89

Clockwise Stage

Number of iterations(100 max.) = 13

R-squared = 0.99920

S	Ms	Es	CAL(Es)	Es-CAL(Es)
0.00	49.00	130.11	130.19	-0.09
10.00	44.00	135.11	134.92	0.18
20.00	40.80	138.31	138.64	-0.33
30.00	37.90	141.21	141.33	-0.13
40.00	36.00	143.11	143.05	0.05
50.00	34.90	144.21	143.83	0.38
60.00	35.30	143.81	143.62	0.19
70.00	37.00	142.11	142.28	-0.17
80.00	39.80	139.31	139.46	-0.16
90.00	44.50	134.61	134.48	0.13
100.00	53.40	125.71	126.17	-0.47
110.00	65.30	113.81	113.63	0.17
120.00	79.70	99.41	99.01	0.40
130.00	92.80	86.31	86.46	-0.15
140.00	102.00	77.11	76.80	0.31
150.00	110.70	68.41	68.88	-0.47
160.00	117.20	61.91	61.91	0.06
170.00	124.00	55.11	55.46	-0.35
180.00	129.00	50.11	49.81	0.30

Optic Axial Angle, 2V (ese) = 49.359 (0.577)

Spindle Stage Coordinates to measure refractive indices.

	S (ese)	Es (ese)	Ms	
OA1	76.03 (0.45)	44.02 (0.33)		
OA2	23.32 (0.33)	68.94 (0.28)	(e-w pol)	(n-s pol)
AB	45.53 (0.14)	53.60 (0.09)	125.51	215.51
OB	156.13 (0.56)	64.48 (0.51)	114.62	204.62
ON	92.40 (0.77)	132.84 (0.45)	46.27	136.27

Figure 4. An example of the numerical output from EXCALIBRW based on the input data given in Figure 3 for the MEG10 sample in Table II. The program outputs the Ms and calculates Es for each of the spindle stage settings in the first three columns. The fourth column, "CAL(Es)," is the Es value calculated for each S value. The last column, "Es-CAL(Es)," is the difference between the observed and calculated values of Es. Next, the program gives 2V and its estimated standard error (ese). Lastly, the program lists the S and Es values for the five optical directions and the Ms values for the three principal refractive index directions. The S and Ms values provide the coordinates for measuring the principal refractive indices of the crystal.

gives a value for R-squared, which is an indicator of the overall quality of the fit of the calculated data to the input data. The center portion of the output repeats the input S and Ms values followed by the Es value (from Equation 2). Because Es would be negative in this case (a cw stage was used), 180° is added to the Es values. In the next column are the calculated Es values "CAL(Es)" determined after EXCALIBRW solved the extinction data set. The last column, "Es-CAL(Es)", is the observed Es values minus the calculated ones; errors greater than 2 to 3° usually result from a misread or mistyped extinction position. The lower portion of Figure 4 provides the 2V value, s, Es, and Ms coordinates for orienting the crystal. First, 2V and its estimated standard error "ese" are given and then the S, Es, and Ms (for both E-W and N-S polarizers) are provided along with their estimated standard errors. The output in this data set shows that all of the optical directions are located to better than 1° and 2V is determined to within 0.6° . This high degree of precision would not be possible using manual graphical methods. However, the graphical results shown in Figure 5A do provide a useful means to visualize the input data (shown as dots) and the output data which are the calculated extinction curves and the locations

of OA1, OA2, AB, OB, and ON. The graphical output is also useful to see if any Ms values were misread by observing any departures from the smooth curved pattern of the calculated extinction curves. Misread extinction values would also be seen in the numerical analysis in the Es-CAL(Es) column.

CRYSTAL SELECTION AND ORIENTATION

Probably the most difficult aspect of spindle stage use is mounting the crystals onto either a glass fiber, which is used for the Supper spindle stage, or a metal needle (e.g., a sewing needle) used in the detent or home-made spindle stages (Figure 1). While it is true any direction within a biaxial indicatrix can be made parallel to the lower polarizer, it is not always the case that graphical or numerical methods will provide a solution from an extinction data set. In the case where these methods do not work, one can always resort to using interference figures to orient the crystal, though with less precision. To apply the numerical methods used by EXCALIBRW, the crystal must be in a "favorable" orientation. There are several definitions of what "favorable" means. Methods have been developed, and will now be discussed, to check if a crystal has been

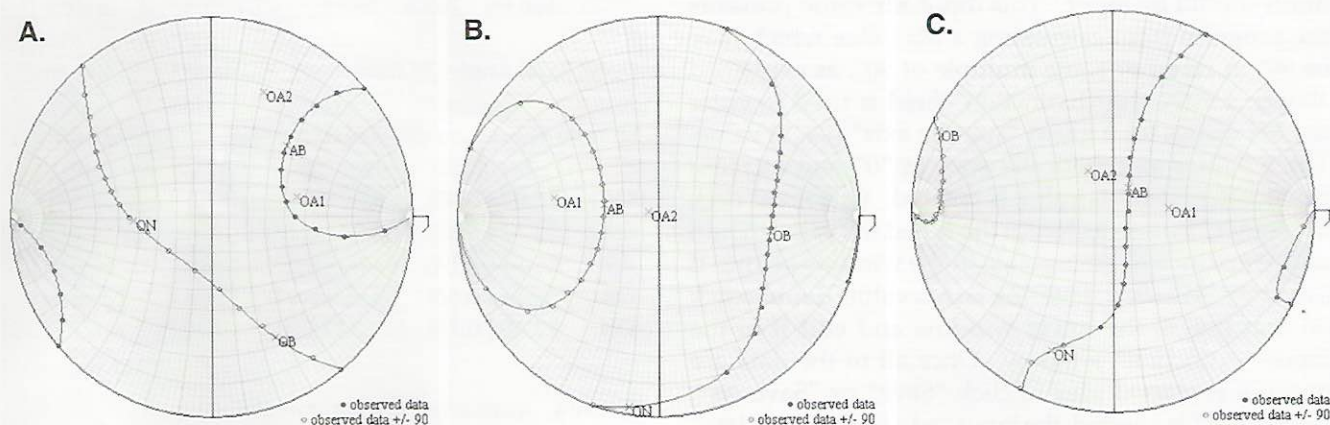


Figure 5. Stereographic plots produced by EXCALIBRW. These graphical representations of the data show: 1) input Es values for each S setting, 2) the calculated extinction curves (and how well they fit the observed data), and 3) the location of the five optical directions. A.) A stereographic projection of the input data and results for MEG10, Table II. This sample passes the 40° -test with 4 intersections (see text for details) of the extinction curves with the $E = 40^\circ$ and $E = 140^\circ$ curves, and is thus in a good orientation for EXCALIBRW. B.) A stereographic projection of the input data and results for MEG18, Table II. Like MEG10, this sample passes the 40° -test with 4 intersections; however, OA2 is nearly perpendicular to the spindle axis, so when OA2 is brought near parallel to +z, (i.e., the light path of the microscope) a circular section is nearly parallel to the microscope stage. In this orientation the retardation is very low, near zero, and precise extinction measurements are difficult to obtain. C.) A stereographic projection of the input data and results for MEG12, Table II. This sample fails the 40° -test and has an optic axis nearly perpendicular to the spindle axis. Despite this unfortunate orientation, EXCALIBRW still found a solution.

mounted in a "favorable" orientation on the spindle axis. As a first approximation, the indicatrix should be in a random orientation to the spindle axis. This means none of the optical directions (i.e., OA1, OA2, AB, OB, ON) should be parallel or perpendicular to the spindle axis.

The 40° test revisited

The 40° test is a simple observation made before collecting an extinction data set to make sure the crystal is likely to produce a solution using EXCALIBRW. The 40° test was historically of great value because plotting the extinction data on a stereonet took longer than mounting the crystal. However, today with a modern PC and the more robust code of EXCALIBRW, it is quicker to just see if EXCALIBRW can obtain a solution even if the 40° test suggests the crystal is in a less than "favorable" position. To perform the test, the microscope stage is set at $M_r + 40^\circ$ (thus the test's name), the S axis is rotated from 0° to 180° , and the number of extinctions is counted. Next, the stage is set to $M_r + 140^\circ$, the S axis is rotated again from 0° to 180° , and the number of extinctions is again counted. If the sum of extinctions is 4, this is the highest number and the crystal is in a most favorable orientation. If there are 3 extinctions, the crystal is less favorable, but still likely to provide a solution. With 2, 1 or zero extinctions, it is recommended that the crystal be repositioned slightly or remounted. In effect, this test shows the number of times an extinction curve intersects the small circles at $E = 40^\circ$ and $E = 140^\circ$. The greater the number of intersections, the more "randomly" oriented the principal vibrations are relative to the spindle axis.

Figure 5A shows an example where the 40° test results in four extinctions with both the 40° and 140° small circles intersected twice (marked by an asterisk). In Figure 5B, the 40° test yields three extinctions, one for the $E = 40^\circ$ small circle and two for the $E = 140^\circ$ small circle. In Figure 5C, the 40° test yields zero extinctions. One may use $E = 30^\circ$ or 20° . As the E angles for the tests decrease, the grain is in a less favorable orientation (i.e., its principal vibration directions are becoming less random). For Figure 5C, we could apply a 30° test (i.e., the intersections of the extinction curves with the $E = 30^\circ$ and $E = 150^\circ$ small circles), which still would yield no extinctions, but two extinctions occur for a 20° test ($E = 20^\circ$ and $E = 160^\circ$ small circles). The samples in Figure 5A and 5B would be expected to produce acceptable results. These samples correspond to MEG10 and MEG18 in Table II. The sample shown in Figure 5C failed the 40° test and would thus not be in a suitable orientation to solve by extinction data. How-

ever, this sample corresponds to sample MEG12 in Table II and was solved by EXCALIBRW.

Recall that graphical methods were developed before numerical methods. The graphical methods require plotting the data on a stereonet by hand, which takes almost as much time as collecting the data. Moreover, the graphical solutions explained in (4) might require even more time to solve than that devoted to both data collection and plotting. Thus it was prudent to develop a test to quickly determine if a mounted crystal were in a favorable orientation.

Optic axis is near perpendicular to the spindle axis

In the seldom case that a crystal is mounted with an optic axis near perpendicular to the spindle axis, a circular section will be nearly parallel to the microscope stage at one S setting. In this orientation, the retardation is very low and it becomes very difficult to measure the precise extinction positions (i.e., Ms values). However, of greater concern is that the EXCALIBRW algorithm becomes unstable. However, EXCALIBRW helps minimize this problem. Notice in Figure 5B that an optic axis (OA2) plots within only a few degrees of the $E = 90^\circ$ small circle (note, this "small circle" plots as a vertical line). To obtain the highest precision from EXCALIBRW, it is best if samples not only pass the 40° test, but also have their optic axes no closer than about $5\text{--}10^\circ$ from perpendicular to the spindle axis. Under these conditions, the retardation of the crystal will show a greater range of intensity and thus a more precise extinction position (Ms) can be measured. If an extinction position cannot be accurately measured, then it is best to omit it by entering a Ms value of "361". Although this may allow a solution, it would be best to reorient the crystal if precise results (e.g., to obtain dispersion information) are needed. In fact, EXCALIBRW will not yield a result if the crystal is oriented with one of the principle optical directions nearly parallel to the microscope stage. This condition results from the direction cosines of the particular direction being very small and EXCALIBRW being unable to provide a solution. If this occurs, remount a grain and try again.

Biaxial vs. Uniaxial model

EXCALIBRW can model uniaxial crystals as well as biaxial ones. However, the uniaxial model is more sensitive to grain orientation. This is a result of the uniaxial model using two rather than four fit parameters. As with biaxials, if a failure occurs when using the uniaxial model, reorient a grain and try again. This strategy is preferable to using the graphical methods

Table II: EXCALIBR Results for 21 Samples of Celestite Measured by Gunter (MEG) Prior to the Course and 11 Samples Measured During the Course by the Students (STU). The Columns "40," "30," and "20" Represent the Number of Extinctions the Crystal Exhibits for the 40-, 30-, and 20- Test as Discussed in the Text.

sample	R 2	2V°	2V°ese	40	30	20
MEG1	0.9963	55.5	2.0	2	2	2
MEG2	0.9995	52.2	0.8	2	2	2
MEG3	0.9993	52.0	0.7	2	2	2
MEG4	0.9973	49.7	1.0	3	2	2
MEG5	0.9721	50.7	0.9	4	2	2
MEG6	0.9617	45.6	1.8	4	2	2
MEG7	0.9989	46.8	1.4	0	1	2
MEG8	0.9991	48.6	1.2	0	2	2
MEG9	0.9620	49.3	0.6	4	4	4
MEG10	0.9992	49.4	0.6	4	2	2
MEG11	0.9968	54.0	1.2	2	2	2
MEG12	0.9321	48.9	1.2	0	0	2
MEG13	0.9980	53.9	1.8	2	2	2
MEG14	0.9984	51.8	1.0	3	2	2
MEG15	0.9989	53.2	1.8	0	2	2
MEG16	0.9975	54.0	1.1	4	2	2
MEG17	0.9214	51.4	1.2	2	2	2
MEG18	0.6473	49.4	0.5	4	3	3
MEG19	0.9806	54.8	1.0	3	2	2
MEG20	0.9911	50.2	2.6	0	2	2
MEG21	0.9371	50.3	0.5	4	2	2
STU1	0.9414	53.7	1.0	4	3	3
STU2	0.9997	45.9	0.9	0	2	2
STU3	0.7711	51.3	0.7	4	4	4
STU4	0.9575	49.7	1.4	0	0	2
STU5	0.9976	52.3	1.6	3	2	2
STU6	0.8966	55.9	1.6	4	2	2
STU7	0.9852	48.2	2.6	1	2	2
STU8	0.9865	49.3	1.3	4	4	4
STU9	0.9959	52.3	0.7	4	4	4
STU10	0.9937	51.3	3.3	4	4	4
STU11	0.9809	50.4	1.0	3	2	2
average	0.9622	51.0	1.3	2.5	2.2	2.4
MEG	0.9659	51.0	1.2	2.3	2.0	2.1
STU	0.9551	50.9	1.5	2.8	2.6	2.8

mentioned above because EXCALIBRW provides the result so quickly. If a crystal is suspected of being uniaxial, but does not yield a solution using EXCALIBRW, a stereographic plot will allow the user to orient a grain and determine its optical properties rather than using EXCALIBRW. Graphical methods may be used to quickly identify a uniaxial crystal because one of the extinction curves will plot as a great circle. This extinction curve corresponds to the circular section of the uniaxial indicatrix, and its normal is the optic axis. However, in certain unfavorable orientations, a biaxial crystal may also have one of its extinction curves nearly parallel to a great circle. In this case, it is recommended that one first run the program with the uniaxial model selected to refine the value of M_r . Then, if the refined M_r value is used with the biaxial model, a solution may be found.

RESULTS

Prior to the MCRI short course, EXCALIBRW was tested and debugged by collecting data sets for the minerals celestite and labradorite using a Supper spindle stage (Figure 1). To simulate use of the detent, or home-made spindle stage that the students would be using, the data were collected only from $S = 0$ to 180° . The crystals were mounted on straight pins instead of glass fibers, which allowed them to be reused by the students.

Celestite

Table II summarizes the results of the 21 celestite samples measured prior to the course and 11 samples measured by the students. The table gives R^2 , $2V$, ese for $2V$, and the number of extinctions for the 40° , 30° and 20° tests. The descriptive statistics given at the bottom of Table II for the instructor (i.e., MEG) and the students (i.e., STU) show very good agreement. This shows the ease with which spindle stage data may be collected. However, the students spent more time obtaining crystals that performed better with the 40° test. MEG was more interested in determining how EXCALIBRW would handle crystals in less favorable orientations. For example, the sample MEG12 (Table II and Figure 5C) was in a very poor orientation, yet EXCALIBRW found a solution, although the $2V$ is below the average of the entire data set. A student who became frustrated after mounting several crystals that failed the 40° test also had success with a very poor orientation (STU4, Table II). In general, better orientations yield more precise and accurate results, so if one is interested in precise results, say for mineral charac-

terization, then effort should be spent to obtain a good orientation. However, if one is only interested in identification of a single crystal, or to confirm its identity by checking its refractive index or $2V$, then a poorly oriented crystal may be sufficient.

Dispersion in labradorite

The first goal for the students in the class was to obtain an extinction data set and use EXCALIBRW to solve that data to obtain $2V$ and the sample's orientation. These first measurements were in polychromatic light. It was hoped that some of the students could advance to collecting extinction data sets in monochromatic light at several different wavelengths. The data collected in monochromatic light are not only more precise, but when multiple wavelengths are used, EXCALIBRW provides the orientation of the five optical directions (i.e., OA1, OA2, AB, OB, and ON) and determines if these directions move as a function of wavelength (i.e., if dispersion occurs). For biaxial crystals (orthorhombic, monoclinic, triclinic), $2V$ often changes as a function of wavelength. For an orthorhombic crystal, the three mutually perpendicular crystallographic axes coincide with the three directions in the biaxial indicatrix; thus, no movement of these directions can occur as a function of wavelength. For monoclinic crystals, only one principal vibration direction is symmetry-constrained along the b crystallographic axis. For triclinic crystals, there are no symmetry constraints on the indicatrix. Thus dispersion behavior can identify the crystal system of a biaxial crystal. However, just because a crystal can exhibit dispersion is no guarantee that it will. Also, different samples of the same mineral may show different degrees of dispersion.

For example, Bloss (3) has shown that Tiburon albite exhibits horizontal dispersion (i.e., AB and ON moved, but OB did not) (also see, 9). To further investigate the dispersion behavior of plagioclase we collected similar dispersion data on a triclinic labradorite (i.e. plagioclase, $\sim\text{An}70$) (10). This An content placed the sample at the species boundary between labradorite and andesine. We used the name labradorite here. The sample was chosen because of its gem character and it had been well characterized in an earlier study (10); however, dispersion had not been determined.

To obtain reliable dispersion data, the crystal must be mounted in a favorable orientation. This is because the magnitude of the dispersion may be small compared to the measurement uncertainty. Only a few students had enough time to calculate an extinction

data set at wavelengths of 430 nm, 589 nm and 680 nm, using their detent stages. However, greater speed can be expected with the Supper stage because its goniometer arcs allow refinement of the crystal's orientation.

Figure 6 shows the input window for EXCALIBRW with extinction data (i.e., S-values) for each of the three different wavelengths. Figure 7 shows the corresponding extinction curves and optical directions for the three wavelengths. It appears all the optical directions, except for AB, have moved. However, one must ask if these movements are statistically significant or from experimental error. Figure 8 shows a portion of the EXCALIBRW output which provides data statistics. The "Treatments" represent the three different wavelengths. Treatments are compared on a paired basis and each optical direction is listed with three numbers: 1) the amount it moved between the two treatments, 2) the estimated standard error of that movement, and 3) a p-value, which is the probability that the movement was real and not from experimen-

tal error. For instance, comparing the 430 and 680 treatment, OB moved 2.203° with an estimated standard error of 0.729, which gives a p-value of 0.005. Usually p-values below 0.10 imply that movement (i.e., dispersion) occurred. From a common sense perspective, if the error is similar to the movement (e.g., AB in 430 to 589), then the results are not precise enough to determine if movement really did occur. Thus, the higher the precision of the input data, the more likely one can determine if dispersion occurred. For the output in Figure 8, it appears that OB and ON have moved. This would indicate horizontal and inclined dispersion occurred. Winchell and Winchell (9) state andesine shows horizontal and inclined dispersion and labradorite shows crossed and inclined dispersion. Thus, our An70 sample (which is between andesine and labradorite) shows similar results. The bottom of Figure 8 shows there is good agreement for the dispersion of the 2V obtained by two students. Both found $r > v$, which is also in agreement with Winchell and Winchell (9).

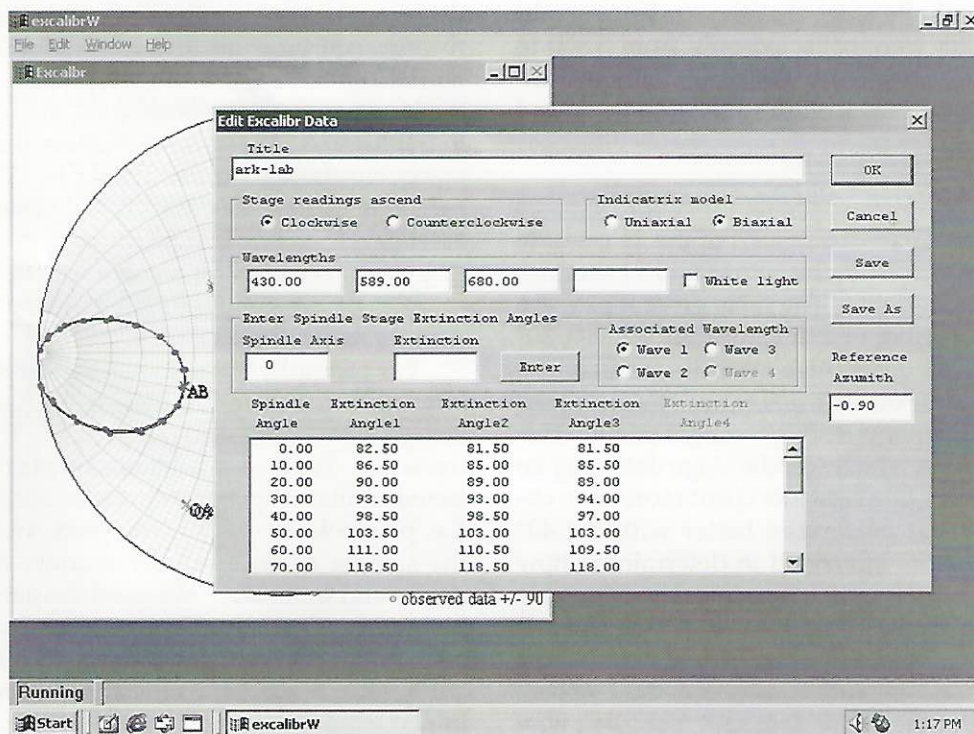
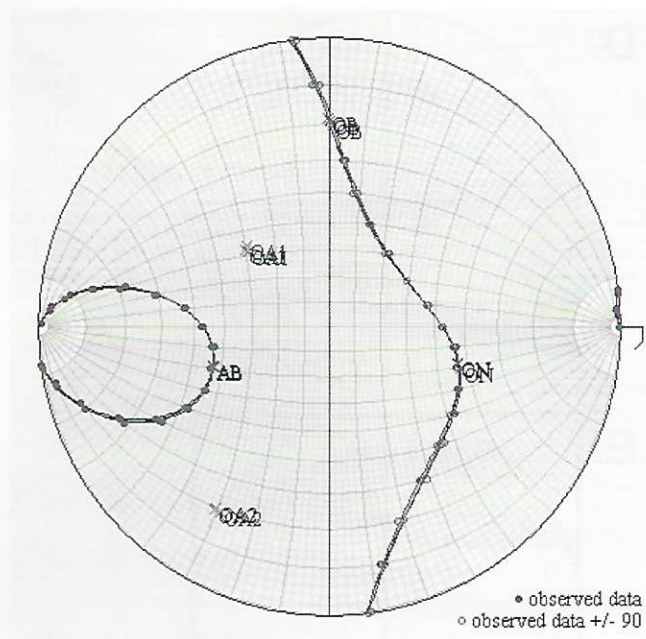


Figure 6. Example input window for multiple wavelength measurements. In this case, extinction data were collected at three wavelengths: 430, 589, and 680 nm. From this input, EXCALIBRW will determine if dispersion (i.e., movement) of the optical directions (AB, OB, ON, or OA's) occurred.



7. Graphical output from EXCALIBRW for a labradorite sample showing extinction curves for data collected at three different wavelengths. (Partial input data are shown in Figure 6.) Visually it appears that AB's position is the same for all wavelength sets while both ON and OB move. Also it appears that both of the optic axes, OA1 and OA2, moved as a function of wavelength.

HOW TO BUILD A SPINDLE STAGE AND FLUID CELL

The original prototype for the Wilcox spindle stage was built with a piece of used floor-tile (Dan Kile, personal communication), proving the stage can be easily constructed. In the mid-1990's Thomas Armbruster of Bern University, Switzerland fabricated a spindle stage out of poster board. His design was later modified for a "Teaching Mineralogy" Workshop at Smith College (Gunter, 11). Further improvements were made prior to the McCrone short course. These improvements allowed students to build their own poster board spindle stage in about 15 minutes using readily obtainable materials. The experience of building their own spindle stage was beneficial in that it allowed the students to realize how simple the device is and because there is currently no supplier for a simple, inexpensive spindle stage.

Dispersion Analysis

For a given pair of optic vectors:

1. Angle (ese) in degrees between vectors determined at two treatments.
2. P-value computed from a t-test assuming the null hypothesis of non-dispersion.

Treatment		OA1	OA2	AB	OB	ON
ID#	ID#					
430.0	589.0	1.256(0.568)	0.628(0.587)	0.438(0.343)	1.386(0.775)	1.452(0.737)
p-value		0.034	0.292	0.209	0.082	0.057
ID#	ID#	OA1	OA2	AB	OB	ON
430.0	680.0	2.288(0.569)	1.506(0.535)	0.465(0.286)	2.203(0.729)	2.192(0.700)
p-value		0.000	0.008	0.112	0.005	0.004
ID#	ID#	OA1	OA2	AB	OB	ON
589.0	680.0	1.639(0.625)	1.159(0.598)	0.342(0.296)	0.848(0.815)	0.803(0.780)
p-value		0.013	0.061	0.256	0.306	0.311

The p-value represents the probability of observing a t-value greater than the computed t-value ($t = \text{ang}/\text{ese}$). P-values less than 0.10 imply rejection of the null hypothesis of non-dispersion. In other words, P-values less than 0.10 imply dispersion.

2V430 = 84.8(7), 2V589 = 84.6(8), 2V680 = 87.2(7) (2Vs for above)

2V430 = 83.2(1.5), 2V589 = 84.2(5), 2V680 = 86.3(8) (2Vs from different student)

Figure 8. Numerical results for dispersion data of labradorite from Figure 7. This figure shows a section of EXCALIBRW's output used to determine if dispersion of the optic directions occurred. The center row of numbers shows the amount of angular movement of the optical directions between 430 nm and 630 nm (e.g., OA2 moved by 1.5°). The values in parentheses are the ese for each measurement (e.g., the ese for OA2 is 0.535). Below these numbers are the p-values used to ascertain if the movement of the vector is statistically significant. P-values less than 0.10 imply that dispersion (i.e., movement) of an optical direction occurred. Lastly, 2V values have been added to the figure to show how 2V changed as a function of wavelength for this dataset and that of another student's.

Figure 9 shows the individual parts required to build a spindle stage and how they are cut. Gunter (11) used poster board as the building material, mainly because it is easy for students to work with, but other materials (e.g., plastic, wood, metal, glass, etc.) could be used. However, it is important that the thickness of the material be similar to that of the glass slide (Figure 10), which will be used to build the fluid cell. If the thicknesses differ slightly, either the spindle stage or the slide can be shimmed by adding layers of Scotch™ tape to their under surfaces.

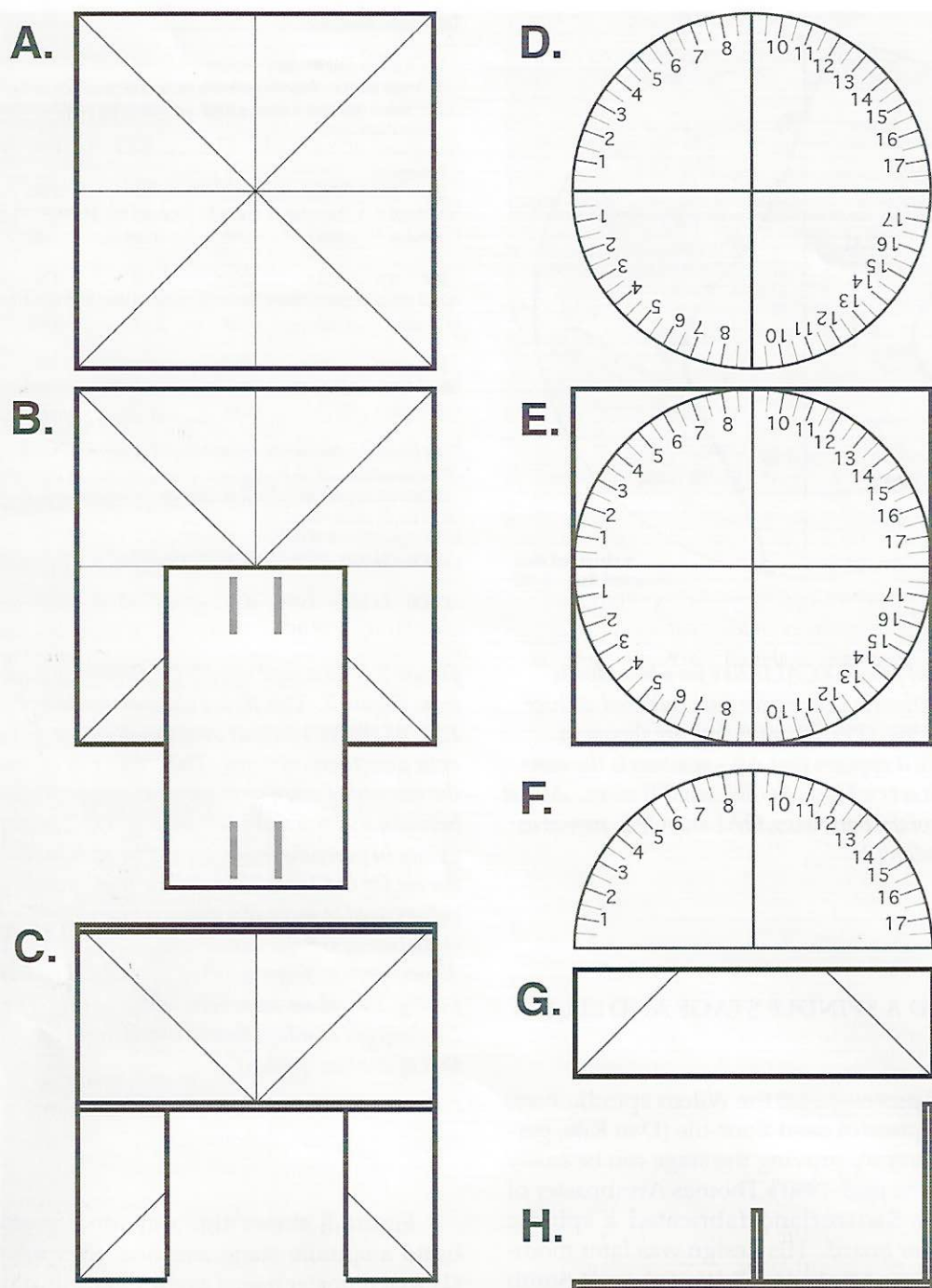


Figure 9. "Blueprint" for each part of a poster board spindle stage. (A detailed description is given in the text on how to assemble it, and a brief description of each part is given here.) A.) This is the base of the stage and is 50 x 50 mm. Also shown on the base are scribed lines to locate its center. B.) The base with a fluid cell (see Figure 10 for a photograph) placed over it. Next, lines are marked on the base (parallel to the fluid cell) and cut out so the fluid cell can be inserted. C.) Sketch of the base with the fluid cell dock area removed. D.) A template for the S-angles; reproduce this (i.e., photocopy or scan it). E.) Another 50 x 50 mm square with the S-angle template attached. F.) The template / square cut in half, trimmed to a semi-circle, and now ready to mount onto the rear of the base. G.) A 15 x 50 mm piece, with scribed edges to be removed. This piece is mounted in the middle of the stage. H.) A side view of the base with the 15 x 50 mm piece mounted in the middle and the S-angle dial on the end. The image in C. is a top view of the assembled parts. Perspective photos are also in Figure 11 and Figure 12.

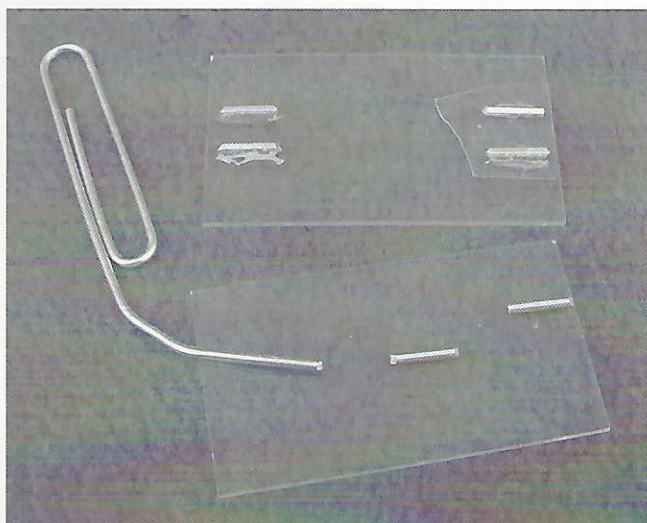


Figure 10. A photograph of the fluid cell. To make the fluid cell, cut two 8 to 10 mm pieces off of a large paper clip and epoxy them 5-8 mm apart on a glass slide being careful to center the space between them. A small cover slip (in this case broken from a larger one) is shown placed on top of the fluid cell. Lastly, a refractive index liquid can be placed in the void created by the cover slip and paper clip pieces.

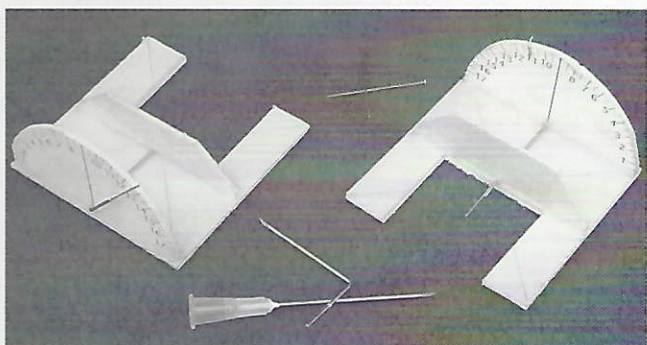


Figure 11. Front and back perspective views of two poster board spindle stages. Also, shown in the foreground is a 2.5" 20 gauge needle. The plastic tip can be cut or pulled off of the needle and the needle bent to make the spindle axis tube. Also, shown in the background are two straight pins, which can be inserted into both ends of the spindle axis tube to facilitate the reading of the S-angle from either side of the protractor scale. The other pin can be used to mount single crystals and then slid into the spindle axis end that extends into the fluid dock area.

Building the base

Once a material has been selected, the first step is cut out two 50 x 50 mm squares. One of these will be the base of the spindle stage and the other will form the protractor scale. For the base, scribe lines as shown in Figure 9A to locate the center. Next, take a glass slide (shown in Figure 10) and place it as shown in Figure 9B. Then trace the outline of the slide on the poster board and cut along these lines. The opening formed will be the bay for the fluid cell. Figure 9C shows the result.

Building the dial

Make a 1:1 copy of the circle protractor scale in Figure 9D (either with a copy machine, or scan the image into a computer). The circle has a diameter of 50 mm. Glue it onto the second 50 x 50 mm square, see Figure 9E. Trim the edges, and cut the circle in half to complete the spindle dial (Figure 9F). If desired, you can glue another circle protractor onto the back of the 50 x 50 mm block before trimming and cutting it, thus providing a dial from either side. If a scale is placed on the back, make sure to place it so the S angles correspond front to back (see Figure 11).

Assembling the stage

Cut a 15 x 50 mm piece and trim the two corners along the diagonal lines (Figure 9G). Glue this piece vertically in the center of the base. Figure 9H shows a side view, Figure 9C a top view, and Figure 11 a photograph. Then glue the protractor dial on the end of the base. Again refer to Figures 9H, 9C, and 11. Now all that remains is to insert a hollow metal tube, which serves two functions: 1) a needle is inserted in one end to hold the crystal and 2) the other end is used to read the S-angle. Take, for example, a 20 gauge 2-1/2" hypodermic needle, and use the needle to make a hole in the center of the assembled stage, that passes through the center of the protractor base and the 15 x 50 mm mid piece. Next, insert a piece of tubing approximately 2 1/2" long through this hole and allow it to extend a few millimeters into the bay for the cell. Finally, put a 90° bend in the tubing where it passes through the back side of the protractor scale. Refer to Figure 11 to see the location of the spindle tube. If the tube is not long enough on the protractor scale, it can be extended by inserting a straight pin in its hollow end, as shown in Figure 11. If the pin fits too loosely, bend it slightly before inserting.

Building the fluid cell

A standard petrographic glass slide, or any glass slide, can be used for the fluid cell. However, choose the one that most closely matches the thickness of the poster board or other material. Cut two 8-10 mm pieces off the end of a large paper clip. Epoxy them onto one end of glass slide as shown in Figure 9B and Figure 10. Make sure they form a cavity that is centered on the slide with an approximate width of 5-8 mm. Refractive index liquid is placed between the paper clip pieces and a mini cover slip placed on top. If desired, one can place fluid cells at both ends of the slide.

Mounting the spindle stage on a microscope stage

Figure 12 shows a completed home-made spindle stage and the fluid cell mounted on the stage of a polarizing light microscope using Scotch™ tape. The tape is first placed in front of the protractor dial. Then the spindle stage, with an affixed crystal, is placed on the microscope stage and the crystal positioned at the center of the crosshairs viewed at low magnification. The tape is then firmly pressed to secure the spindle stage to the microscope stage. Additional strips of tape should be placed on the ends of the spindle stage (next to the fluid cell). Before mounting, the microscope stage should be at zero so that when the spindle stage is mounted E-W, the Mr will be near 0° (i.e., the spindle is pointed to the W, see Figure 2). In Figure 12, it can be seen that a straight pin has been bent into the form of a "U" so the S-angles can be read on the back, as well as the front, of the protractor. If the "U" shaped needle is removed, one can translate the crystal in the "X" direction to center it in the field of view.

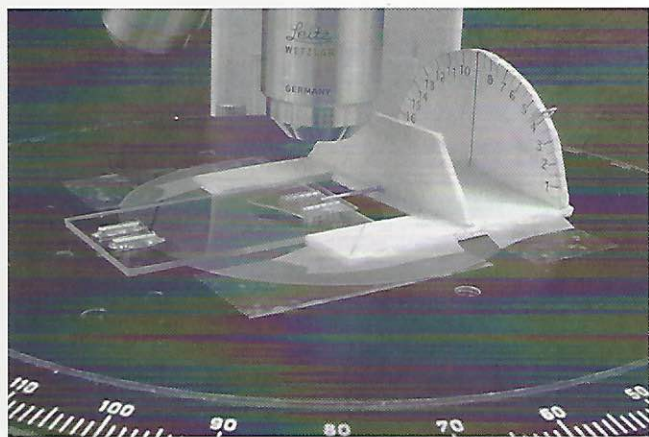


Figure 12. A close-up view of the home-made spindle stage / oil cell taped onto the stage PLM and ready for use.

The resulting spindle stage, although very simple in design, is highly functional and inexpensive. It does, however, lack certain features of value. For instance a translation in the Y direction, or detents for the S setting. Individuals are encouraged to improve upon and share with others their designs.

CONCLUSIONS

The students taking this three-day short course obtained research quality results using the detent or poster board spindle stages and the new version of EXCALIBRW. The 2V results for celestite were nearly identical in precision and accuracy to that obtained by an experienced spindle stage user using the Supper spindle stage. Some of the students were even able to collect data of high enough accuracy to determine the dispersion of a plagioclase crystal. The students' work demonstrated that EXCALIBRW is easier to use than earlier versions and is more robust in its numerical methods. It is the hope of the authors that the availability of a home-made spindle stage and a more robust Windows version of EXCALIBRW will increase spindle stage use.

NOTE: EXCALIBRW is maintained by S. H. Evans. He may be contacted via the internet at clanevans@msn.com. Any issues with the program should be communicated to him. Evans would also like to build up a user database so that updates can be efficiently communicated.

ACKNOWLEDGEMENTS

The authors (who were also the instructors of the course, except for Stan Evans, who debugged and refined the Windows version of EXCALIBRW) would like to thank Don Halterman for providing the idea to conduct this short course. We would also like to thank the students who took the course; we enjoyed their interest and enthusiasm. The McCrone Research Institute provided excellent facilities for the course; it could not have been taught without the infrastructure of that organization. Either direct or indirect financial support was provided by the National Science Foundation (NSF-CCLI 0127191) (Gunter), MVA, Inc. (Bandli), and Hercules, Inc. (Su).

REFERENCES

1. Kile, D.E. "The petrographic microscope: Evolution of a mineralogical research instrument"; *Mineralogical Record* (in press).
2. Wilcox, R.E. "Use of the spindle stage for determination of principal indices of refraction of crystal fragments"; *American Mineralogist* **1959**, 44, 1272-1293.
3. Bloss, F.D. *The Spindle Stage: Principles and Practice*; Cambridge University Press: Cambridge, England, 1981.
4. Bloss, F.D. *Optical Crystallography*; Mineralogical Society of America: Washington, D.C., 1999.
5. Gunter, M.E. "The future of polarized light microscopy: Bright, dim, or extinct?"; *The Microscope* **2003**, 51, 53-63.
6. Gunter, M.E., Bloss, F.D., and Su, S.C. "Computer programs for the spindle stage and double-variation method"; *The Microscope* **1989**, 37, 167-171.
7. Bartelmebs, K.L., Bloss, F.D., Downs, R.T., and Birch, J.B. "EXCALIBUR II"; *Zeitschrift fuer Kristallographie* **1992**, 199, 185-196.
8. Gunter, M.E. and Twamley, B. "A new method to determine the optical orientation of biaxial minerals: A mathematical approach"; *Canadian Mineralogist* **2001**, 39, 1701-1711.
9. Winchell, A.N. and Winchell, H. *Elements of optical mineralogy, Part II: Descriptions of minerals*; Wiley: New York, 1951.
10. Johnston, C.L., Gunter, M.E., and Knowles, C.R. "Sunstone labradorite from the Ponderosa Mine, Oregon"; *Gems and Gemology* **1991**, 27, 220-233.
11. Gunter, M.E. "Laboratory exercises and demonstrations with the spindle stage"; In *Teaching Mineralogy*; Mineralogical Society of America, 1997, pp 309-318.