Orienting and Measuring the Optical Properties of Crystals

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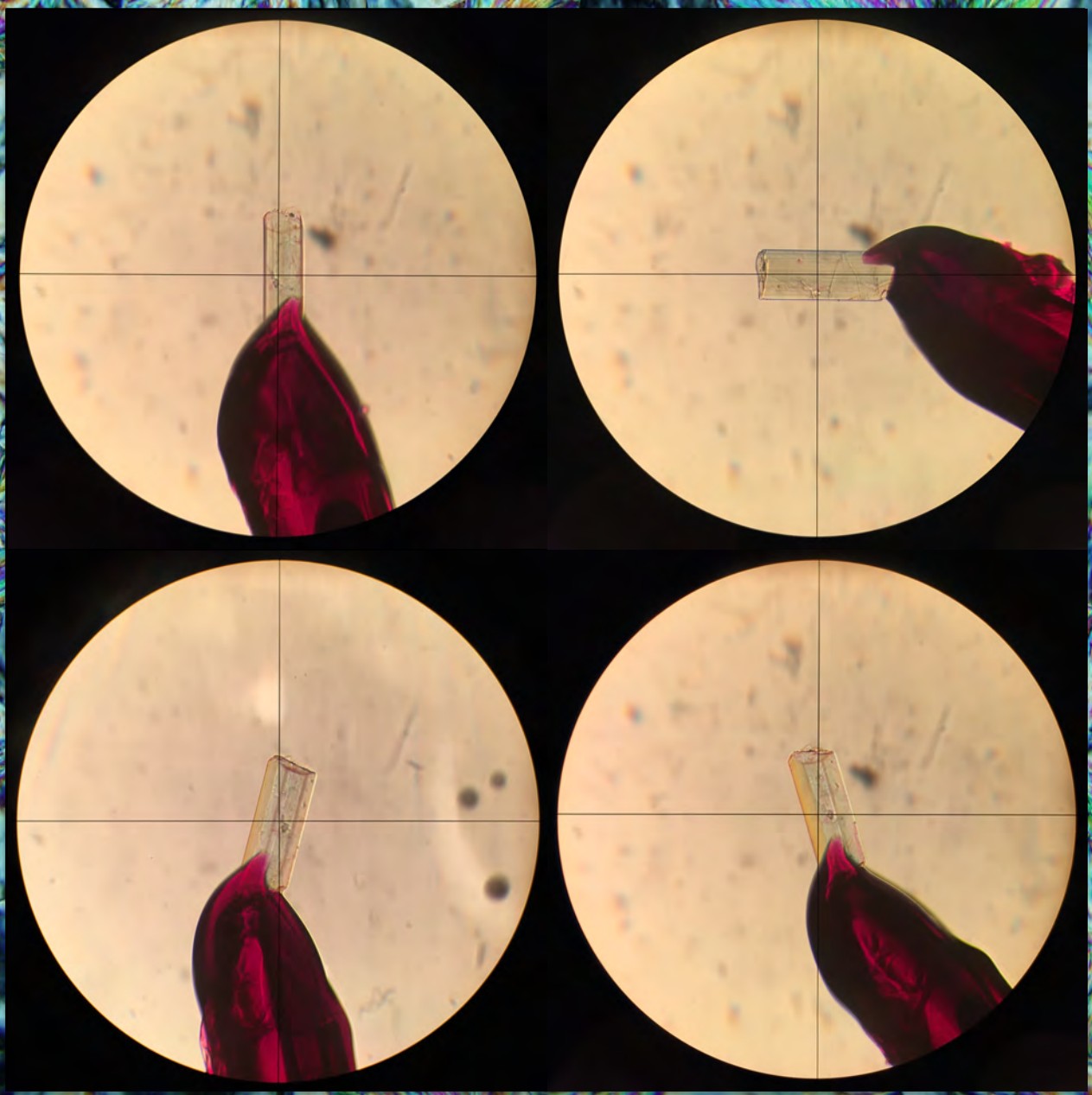

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Preface

The purpose of this guide is to supplement the process of orienting crystal optically and measuring their principle refractive indices. This guide demonstrates how to use the Excel spreadsheet EXCELIBR with a few example methods, while also demonstrating practical skills in optical crystallography. EXCELIBR contains algorithms similar to the program EXCALIBR, a program first developed in the early 1970s and continuously revised until its last iteration in 2004 with the program EXCALIBRW (Bloss & Reiss, 1973; Bartelmehs et. al., 1992; Gunter et. al., 2004). EXCELIBR's orientation solutions are demonstrably close to the solutions of the last version of EXCALIBR, EXCALIBRW. Along with orientation solution tabs, EXCELIBR also has a series of other optical calculations, like that of Opt_cal (Gunter & Schares, 1991). For more detailed instruction on the use of the spindle stage methods, see (Bloss, 1981). For an overview on the theory of the algorithms used in the spreadsheet see (Joel, 1965) (Bloss & Reiss, 1973) or (Steven & Gunter, 2018)

1 Tab Overview

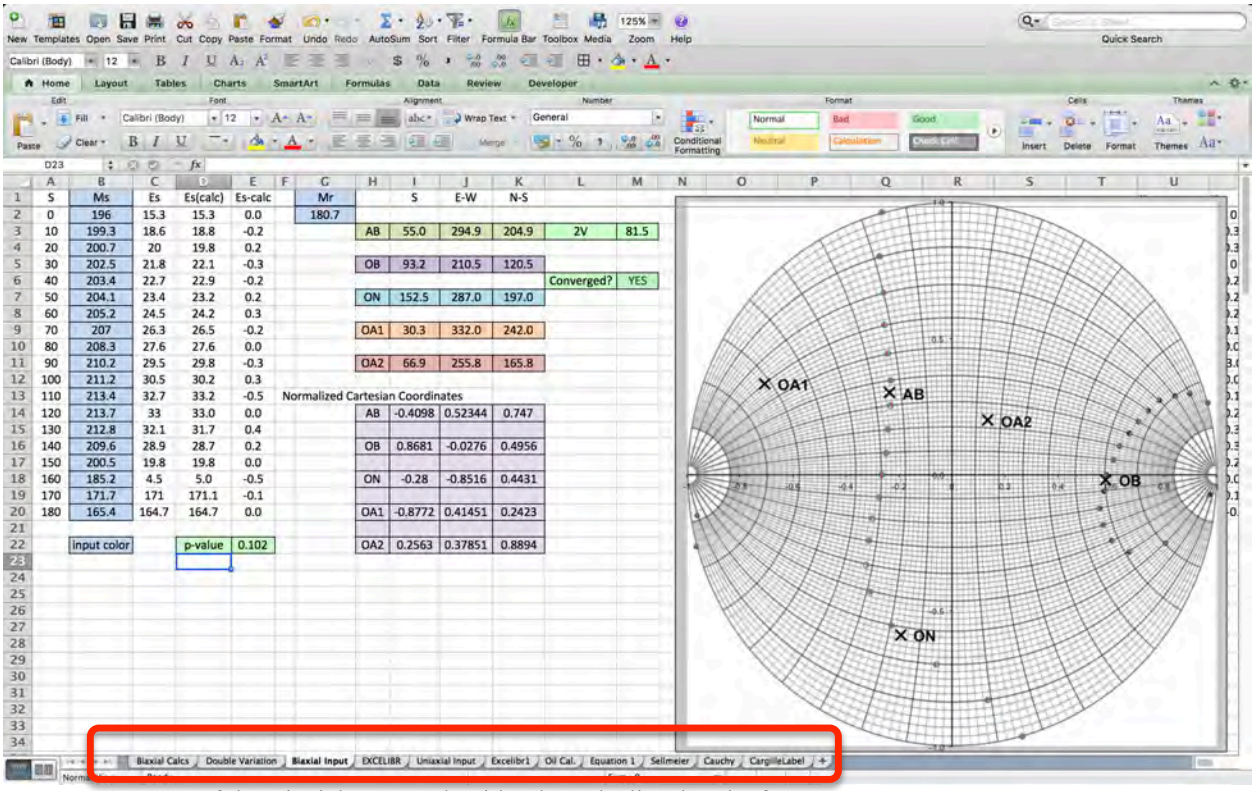


Figure 1.1 Image of the Biaxial Inputs tab with other tabs listed at the footer.

Biaxial Calcs

Contains calculations for alpha, beta, gamma, and 2V of biaxial crystals. Given any of the three, the fourth can be calculated. Also contains birefringence calculations and comparison of calculated versus observed refractive indices and 2V.

Double Variation

Contains temperature and wavelength corrected values for a refractive index liquid for measuring refractive index of crystals. Given a series of refractive index measurements on a crystal at a variety of wavelengths, a refractive index value of match is interpolated for 589.3 nm. Setup is designed for a slide monochromator (graded interference filter) but can easily be modified to use interference plates for monochromatic light.

Biaxial Input

Input and output tab for orienting biaxial crystals. Contains coordinate outputs for optical vectors, 2V, and a stereographic plot for where the optical vectors exist in 3D.

EXCELIBR

Tab containing the algorithm and majority of the calculations for orienting biaxial minerals. The 'EXCELIBR' tab is referenced by the 'Biaxial Input' tab to provide the solution for the orientation of the optic axes. The extinction data entered in the 'Biaxial Input' tab is referenced by the 'EXCELIBR'. It is not necessary to use this tab for anything unless a user wants to modify the programming of EXCELIBR (increase the number of iterations or revise the algorithm).

Uniaxial Input

Contains the input and output cells for solving for the optical orientation of uniaxial crystals.

Excelibr1

Contains the calculations and algorithm for solving for the optical orientation of uniaxial crystals.

Oil Cal.

Contains precise temperature and wavelength corrected values of refractive index for liquids of unknown, or crudely known refractive index. This tab is used for modeling dispersion of a refractive index liquid for precise and accurate calibration of liquid using a refractometer. This tab is designed to use a *Bellingham and Stanley Abbe 60 Refractometer* though it can be modified to use any refractometer that is wavelength corrected.

Equation 1

Contains the dispersion model for a refractive index liquid using an equation introduced by Gunter (1989). This tab references the 'Oil Cal.' tab to model dispersion of a refractive index liquid.

Sellmeier

Contains the dispersion model for a refractive index liquid using the Sellmeier equation. This tab references the 'Oil Cal.' tab to model dispersion of a refractive index liquid.

Cauchy

Contains the dispersion model for a refractive index liquid using the Cauchy equation. This tab references the 'Oil Cal.' tab to model dispersion of a refractive index liquid.

CargilleLabel

Contains a dispersion model for refractive index given information listed on a Cargille refractive index liquid. Tab calculates the Cauchy constants for a Cargille liquid. The information listed on a Cargille liquid is valid if it is relatively new or well taken care of.

2 Equipment Overview

For Orienting Crystals:

Polarized Light Microscope

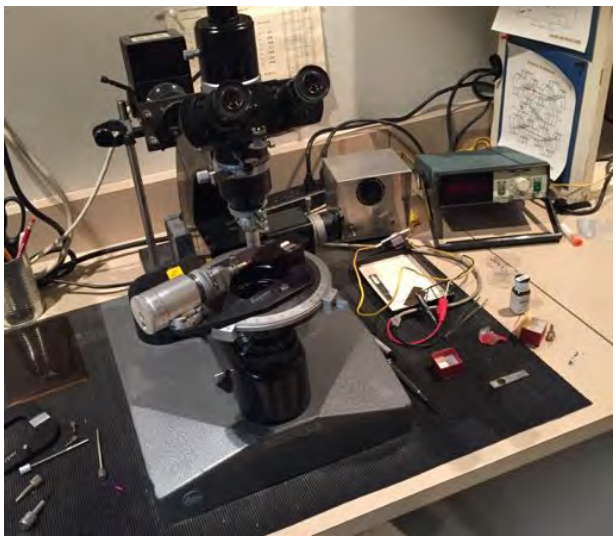


Figure 2.1 1960's *Lietz Ortholux Pol* equipped with a *Supper* spindle stage. A heating cell and power source sit to the right of the microscope, along with a digital thermometer. This microscope, along with other models of its generation, are great for mounting *Supper* and *Olaf* stages due to it's adjustable working distance of both the stage (focus and stage z position) and the tube position. Any microscope designed to mount a universal stage can mount a *Supper* stage. The preferred objective in this setup is a 32x long working distance lens.

Stereoscope

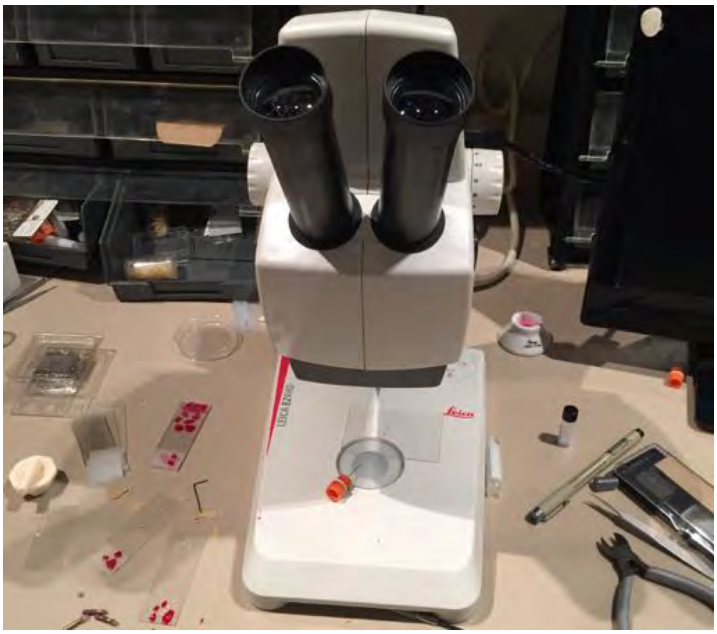


Figure 2.2 Leica stereoscope used for mounting crystals. Crystals are sorted on glass slides and adhered to a specimen pin with nail polish.

Spindle Stage

Figure 2.3 Micro-refractometer spindle stage made by Olaf Medenbach. When used with a dispersion-staining lens, this spindle stage can be used to plot dispersion curves without the use of a monochromator.

Figure 2.4 Homemade (left) and detent or "Wilcox"(right) spindle stage. Research quality orientation solution may be collected using these stages as well (Gunter, 2004).

For Measuring Refractive Indices:

Cargille liquids

Figure 2.5 Boxes of *Cargille* refractive index liquids. With relatively new refractive index liquids, the values on the label are very accurate and can be used without calibration. These are essential to measuring refractive indices of minerals, and are helpful for seeing extinction positions in XPL.

Refractometer

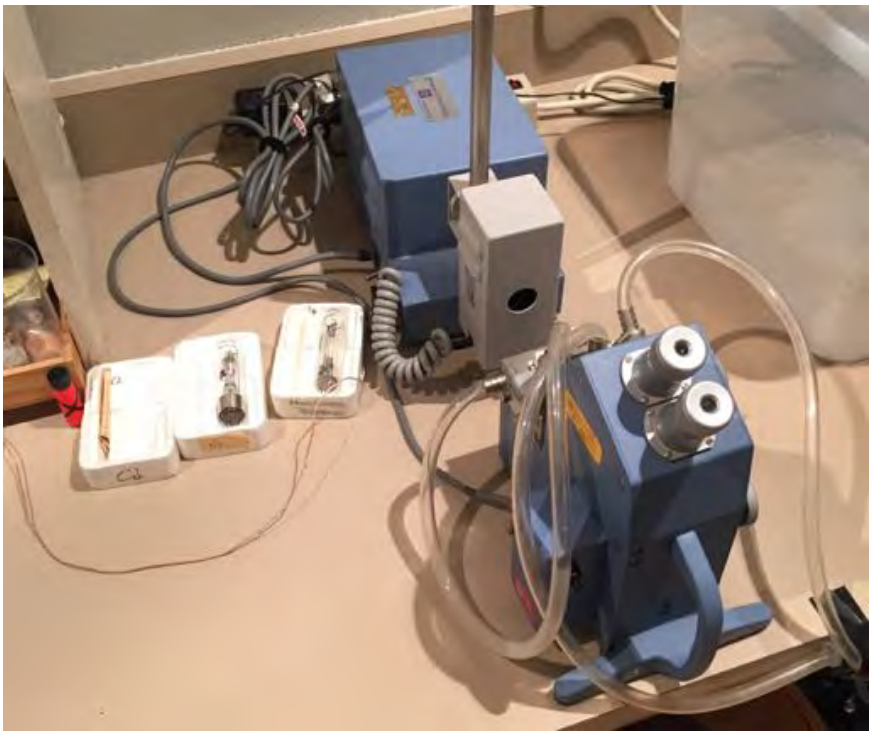


Figure 2.6 Bellingham and Stanley Abbe 60 refractometer. This is a high precision refractometer that can be used for refractive index liquid calibration. The word 'calibration' is used in this context to describe accurately measuring refractive index of a *Cargille* liquid at multiple wavelengths to model its dispersion.

3 Mounting Crystals

Materials: glass fibers, brass pins (*Supper* or other crystallographic specimen pin), adhesive (nail polish, epoxy, or super glue), glass slides, stereoscope, acetone, plasticine or beeswax, sewing needles, sieves (optional)

Figure 3.1 A, B, C, D, E Example materials which include (A) 60 and 150 mesh dry sieves, (B) a mounting adhesive (nail polish works well, superglue dissolves in RI liquids, epoxy is permanent) along with a dulled sewing needle, (C) acetone for dissolving the nail polish for remounting, (D) glass slides as a substrate for mounting crystals, and brass pins beeswax, plasticine, and (E) glass fibers for assembling the specimen pin.

3.1 Assembling Specimen Pin

3.1.1 Pulling Glass Fibers

With a detent or homemade stage, crystals will be mounted on the end of a small metal pin with the head clipped off (Gunter, 2004).

With spindle stages that use goniometers, you will need to assemble a specimen pin.

Glass fibers make an excellent spindle for mounting crystals since they have a small diameter, they are straight, and they don't interfere much with x-rays if a user wants to X-ray a specimen.

To make glass fibers you will need a glass starting material such as a stir stick or capillary tube, a Bunsen burner or blowtorch, and a knife (**Figure 3.2**).

Figure 3.2 Cutting tool and glass starting material for pulling fibers.

Start the flame source with the glass starting material in hand. Begin holding the center of the glass starting material over the flame while continuously twisting the glass (**Figure 3.3**).

Figure 3.3 Under the torch, the glass starting material will burn orange and a glass dropper tube like this takes about 30 seconds to become pliable.

Once the glass begins to droop, it's about ready to be pulled (**Figure 3.4**).

Figure 3.4 Somewhere between the point where the glass becomes pliable, and the point where it is totally molten is when the glass is ready to be pulled. Finding this point takes practice but there should be some resistance in the glass, otherwise it will fall apart.

In one motion, take the glass off the flame and begin pulling at a relatively quick and steady pace (**Figure 3.5**).

Figure 3.5 Pulling the fiber too slow will result in thick fibers and pulling too fast results in pulling the glass apart. The key is to have the right pliability and constant speed of pull.

Once pulled, the glass rapidly cools so it can be set down and sectioned (**Figure 3.6**). The faster you pull, the thinner the fiber, but if the glass is pulled too fast and is too hot, it won't stay together. This process takes practice to get optimal fiber sizes.

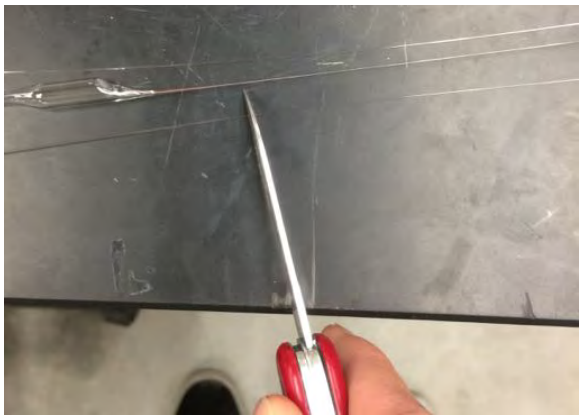


Figure 3.6 With long thin strands of glass, fibers can be sectioned with a cutting tool

After fibers have been sectioned, sort the fibers by diameter. Many of the fibers may be too large or too small to be useable, however, it still helps to have a spectrum of sizes for dealing with different crystal sizes.

3.1.2 Assembling Brass Specimen Pin

Now that you have glass fibers, the specimen pin is ready to be assembled. The main components of the specimen pin include: a brass pin (acquired from *Charles Supper Co.*), glass fiber, and beeswax (**Figure 3.9**).

Figure 3.9 Glass fiber, beeswax, and Supper specimen pin fit for optical and/or x-ray goniometers.

Begin by softening the beeswax using the heat of your hand. Once the beeswax is malleable, shape it around the end of the brass pin (**Figure 3.10**).

Figure 3.10 Specimen pin with beeswax. Plasticine may also be used in place of beeswax.

Now puncture a hole through the center of the brass pin and beeswax using a sewing needle (**Figure 3.11**).

Figure 3.11 Punctured beeswax ready for threading fiber through.

Thread the glass fiber through the hole ensuring that the length of the fiber sticking out is either short enough or long enough for your purposes (**Figure 3.12**). Generally, a fiber that sticks out of the pin by about a centimeter is good enough.

Figure 3.12 Unsecured specimen pin.

Gently squeeze the wax with pliers to secure the glass fiber (**Figure 3.13**)

Figure 3.13 Securing the glass fiber.

To ensure that the fiber doesn't move, coat the wax and fiber with nail polish and let it dry (**Figure 3.14**).

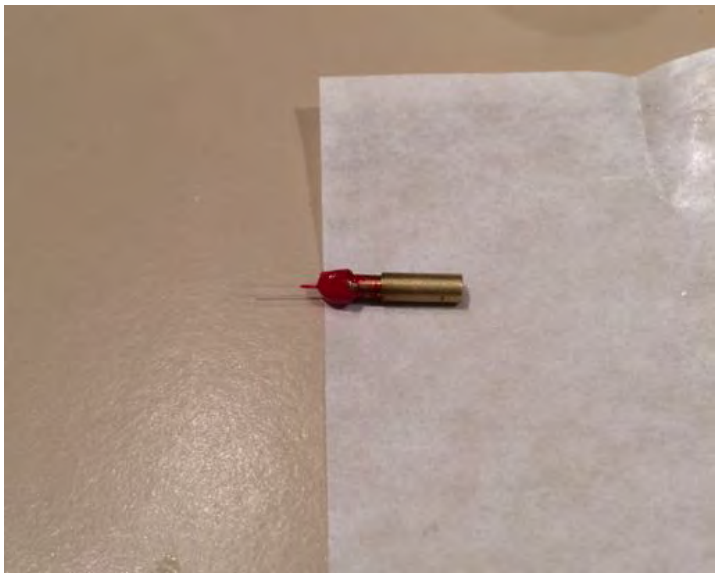


Figure 3.14 Specimen pin secured with nail polish.

3.2 Choosing crystals

The first step is selecting an optically uniform crystal. Ideally, the crystal should transmit light and be free of alteration, twinning, zoning, inclusions, and exsolution. Additionally, the best crystals to measure are generally 50 to 250 microns in diameter, therefore, running grains through 60 and 150 mesh (106 to 250 micron) sieves provides optimal grain sizes. Once you have an assortment of grains, pour the grains onto a glass slide for selecting crystals viewing under a stereoscope. In Figure 3.2, the crystal on the far right is the best crystal and has a diameter of ~120 microns.

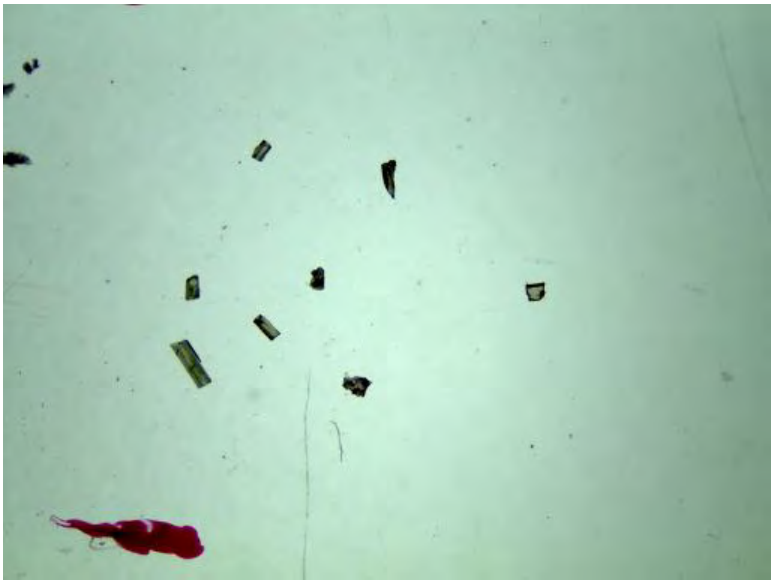


Figure 3.15 Group of crystals (in air) for selection. Though crystals are easiest to discriminate under oil and in XPL, they are difficult to adhere.

3.3 Crystal Mounting

There are a myriad of ways of mounting crystals for x-ray purposes but the primary consideration in optical crystallography is having a crystal mounted at the end of a specimen pin that is not coated by any mounting medium (nail polish, glue etc).

When mounting a crystal onto a glass fiber, a crystal that has a similar diameter to the glass fiber is easiest to mount (**Figure 3.3**).

Figure 3.16 Comparison of the glass fiber diameter to the diameter of the crystal of interest.

Prior to mounting a crystal, place a drop of nail polish onto the slide containing your grains and dip the tip of the glass fiber in the nail polish (**Figure 3.4**).

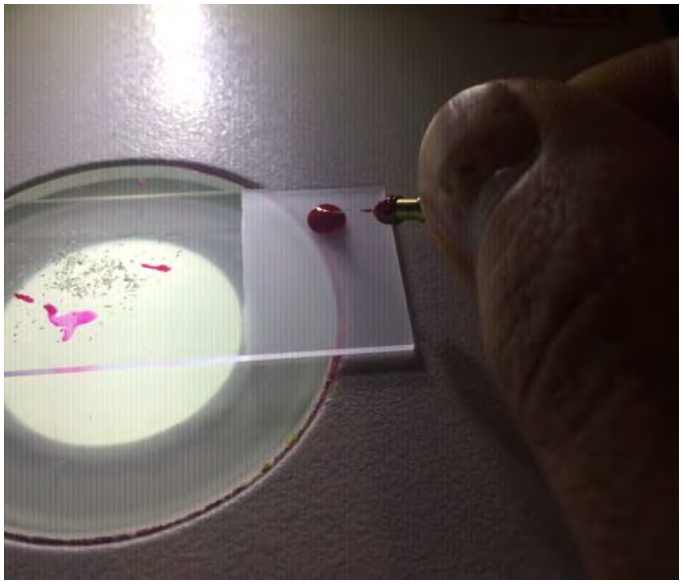
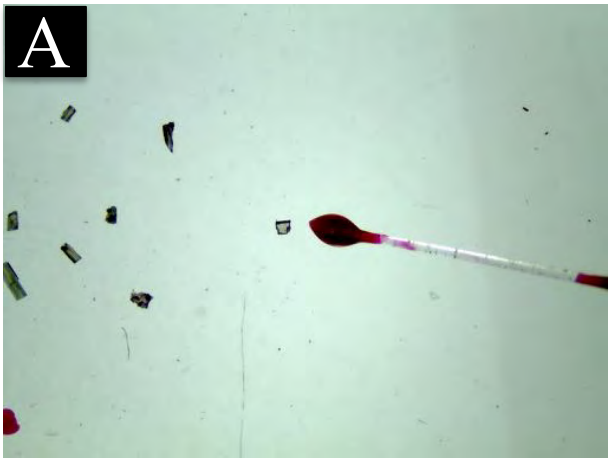
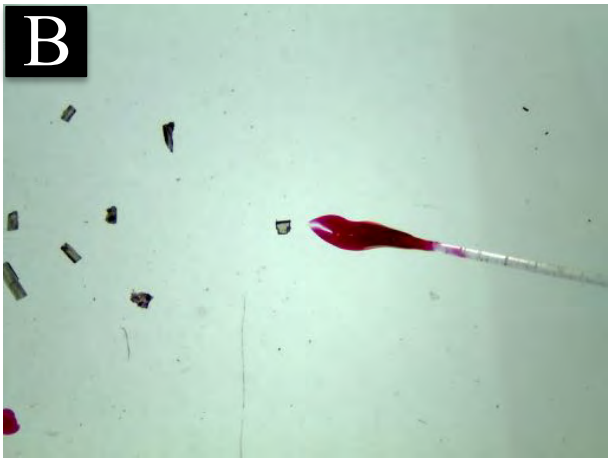
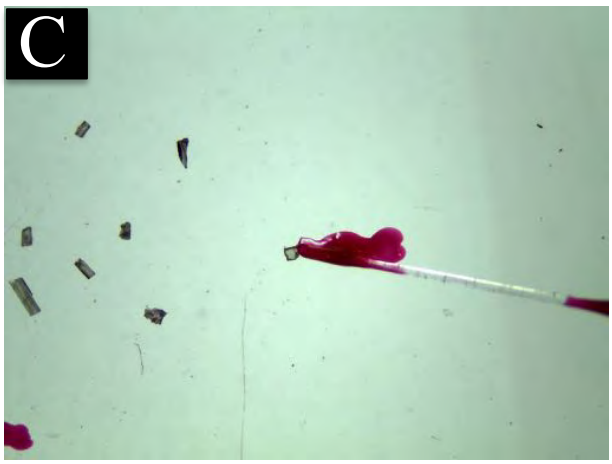


Figure 3.17 Dipping the end of the specimen pin in nail polish.

Figure 3.5 (A-D) outlines the motion of adhering a grain to the end of the glass fiber. (A) The nail polish at the end of the fiber is slathered onto the slide just behind the grain of interest. (B) The glass fiber is pulled back so that the tip of the fiber can be coated. (C) The glass fiber is driven through the nail polish an into the grain. (D) Once the grain and fiber is away from the nail polish, lift the fiber up off the slide. Steps (A-D) is done in one, relatively swift motion.







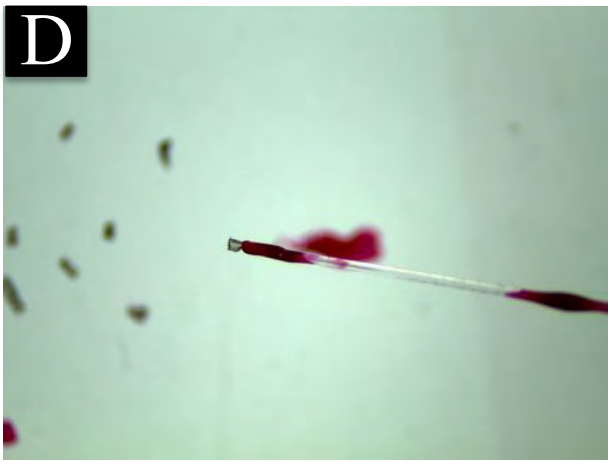
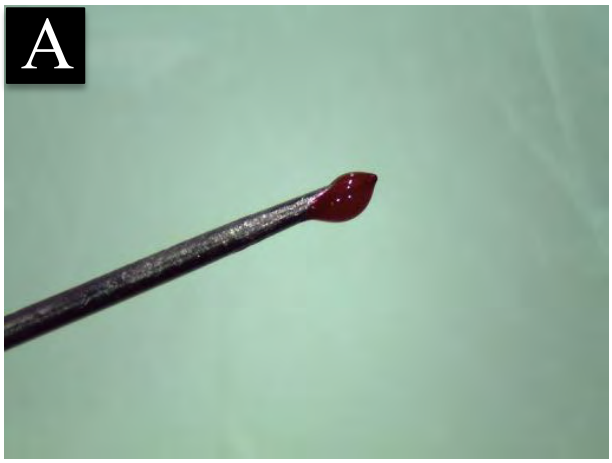
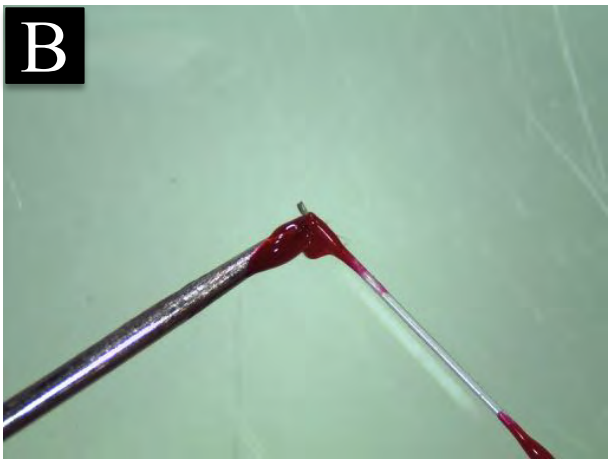


Figure 3.18 A-D (A) Placing nail polish behind the crystal, (B) pulling back on the fiber to immerse the tip in nail polish, (C) driving the fiber through the nail polish and into the crystal, and (D) pulling the fiber and crystal straight up off of the slide.

An optional but recommended step is to reinforce the base of the grain onto the fiber (**Figure 3.6 A-C**). (A) Take the tip of a sewing needle and dip it into some fresh nail polish. (B) Bring the fiber and grain to the coated sewing needle and wrap it around the base of the crystal. This is easiest when the nail polish that has adhered the grain to the fiber is already dried. (C) Once reinforced, clean the sewing needle with acetone and straighten or angle the grain against the needle as needed. Step (C) is helpful if the grain is drooping to far to the side, or if you find that the program takes issue with the orientation with which it was mounted.





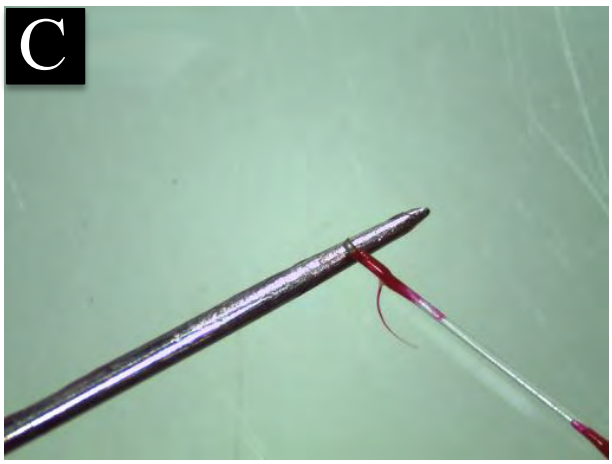


Figure 3.19 A-C (A) Dulled sewing needle with a drop of nail polish. (B) Wrapping the base of the crystal in nail polish. (C) Adjusting the crystal to be crooked for a good orientation solution later on. Sometimes globs of nail polish off the side of the fiber get in the way of the glass coverslip later on but can be removed by dabbing it with the end of an acetone-soaked KimWipe.

4 Centering

4.1 Centering Procedure with a *Supper* Stage

The centering procedure is done prior to inserting an oil cell. Before centering a crystal on a *Supper* spindle stage, make sure that your objective lens is well centered to the axis of rotation of the stage (see Bloss, 1999). Also, it helps to lock the stage in the zero position for the spindle stage to point East or West; in this case, the stage is locked at the 180 degree setting to point East (**Figure 4.1**).

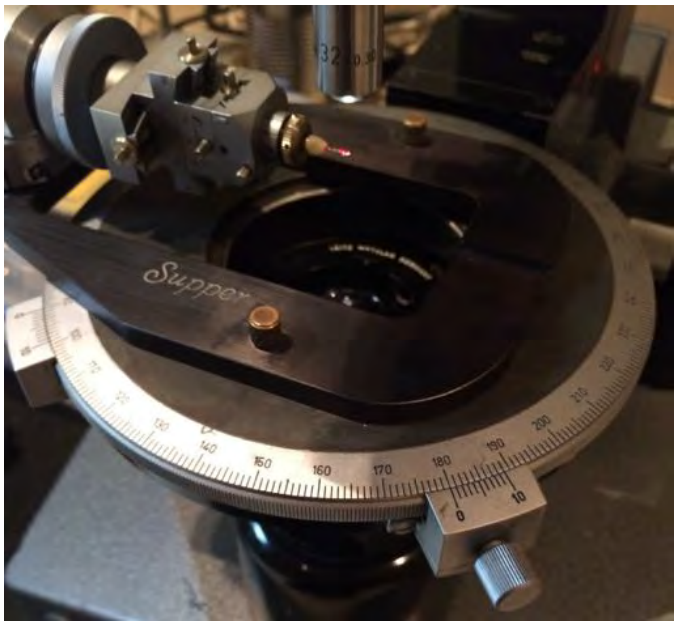


Figure 4.1 Microscope stage locked in the position where the spindle axis faces East.

The next step is to adjust the tension on the X-Y translational screws on the spindle stage. The tension should be enough so that the dovetail doesn't wobble significantly, but loose enough so that the screw won't strip when you adjust it (**Figure 4.2**).

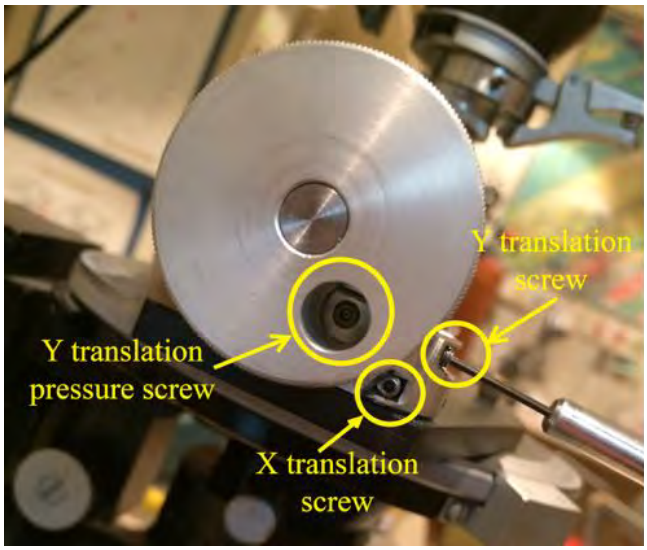


Figure 4.2 Outline of the back of a *Supper* stage showing the translation and pressure screws.

4.1.1 Centering

The centering procedure on a spindle stage is to account for the way that a specimen mount can be, and likely is, off-center. This is generally because of the way a glass fiber is mounted in the brass pin. If everything is perfectly centered on the specimen pin, then the zero settings on the goniometer would be adequate, and the crystal would only need to be translated into the center of the crosshairs under the microscope.

The goniometer is mounted onto the spindle stage, which means that the X-Y component of the spindle stage affects the goniometer. This is why the first step is to center the spindle stage. Conceptually, this is the most difficult to center because it involves **centering an axis of rotation**, not the crystal. Once a user understands this, this procedure becomes relatively easy. After centering the axis of rotation, the crystal is then centered to this axis using the goniometer adjustments. The following outlines the procedure for centering a crystal where the Y translation, which influences the axis of rotation, is grossly off-center.

Before mounting the goniometer, make sure that all of the readings on your goniometer are at the zero position.

With the goniometer on the stage, you can achieve a rough center by eyeballing the position of the crystal. In **Figure 4.3**, the crystal is not far enough out (in the X direction) to be viewed under the objective.

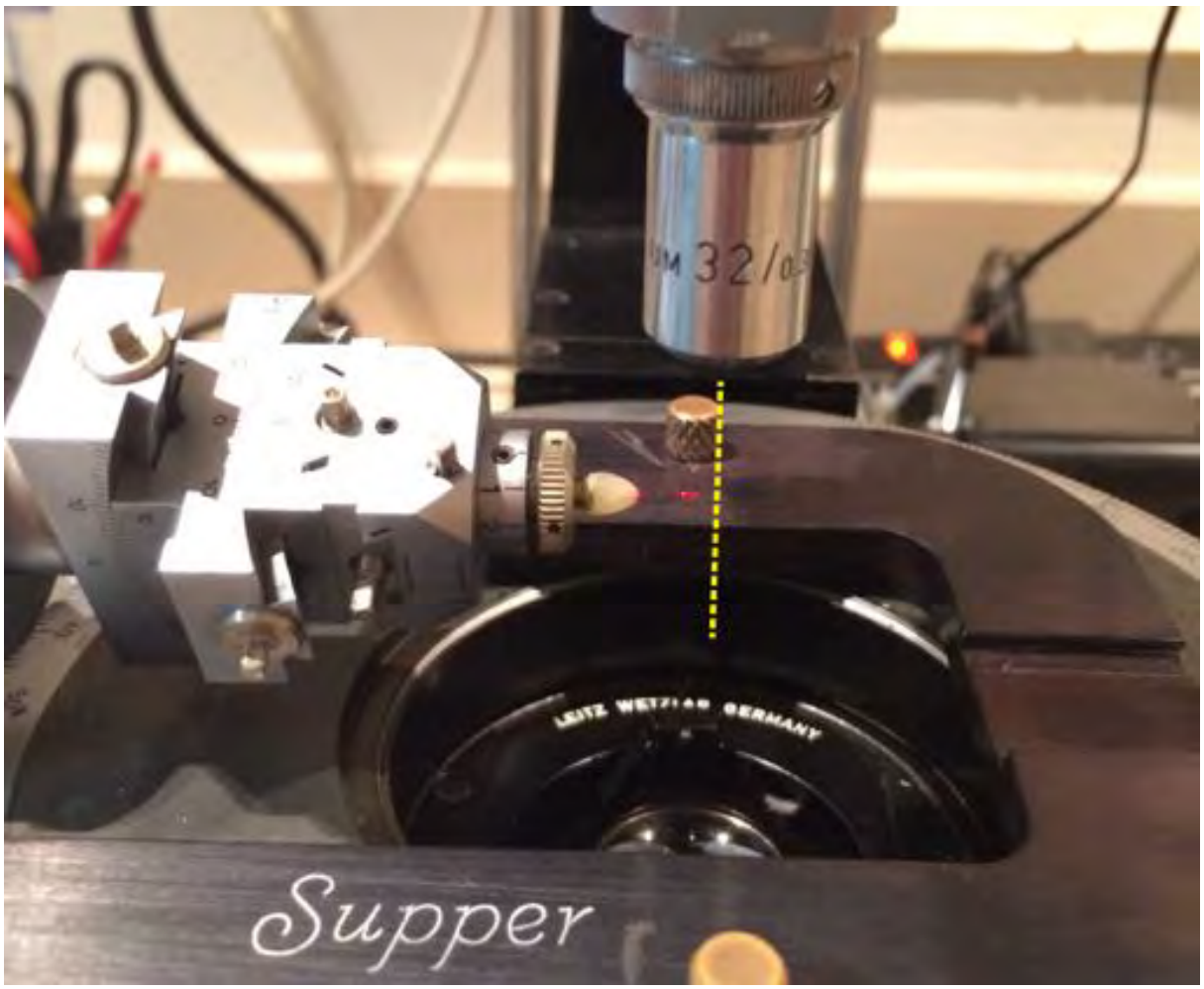


Figure 4.3 Image showing the approximate line of view of the objective. The crystal does not intercept this line here.

Adjust the "X" translation screw to bring the crystal far enough to be under the center of the objective lens (**Figure 4.4**). It's better to overshoot this than to not be far enough.

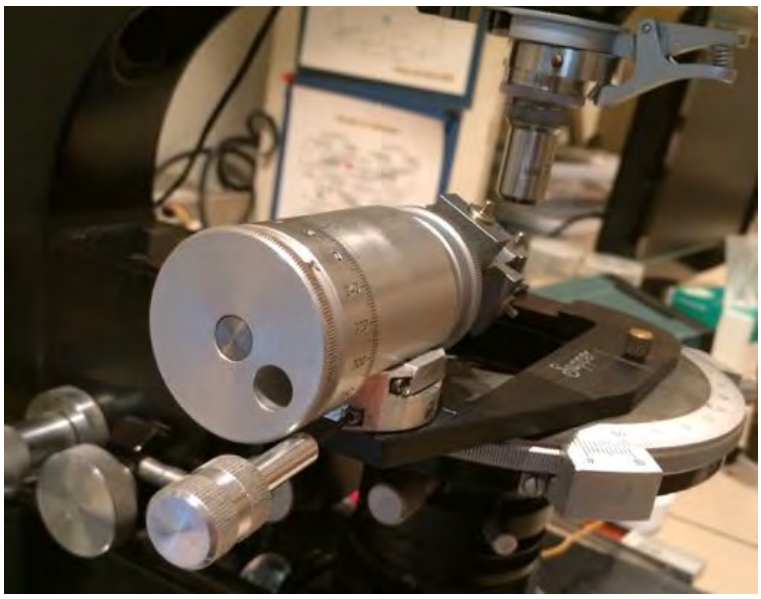


Figure 4.4 The Y-translation screw is used to translate the crystal into the line of view of the objective. Goniometer usually also have a Y-translation screw.

Here, the crystal is at least far enough under the objective (**Figure 4.5**).

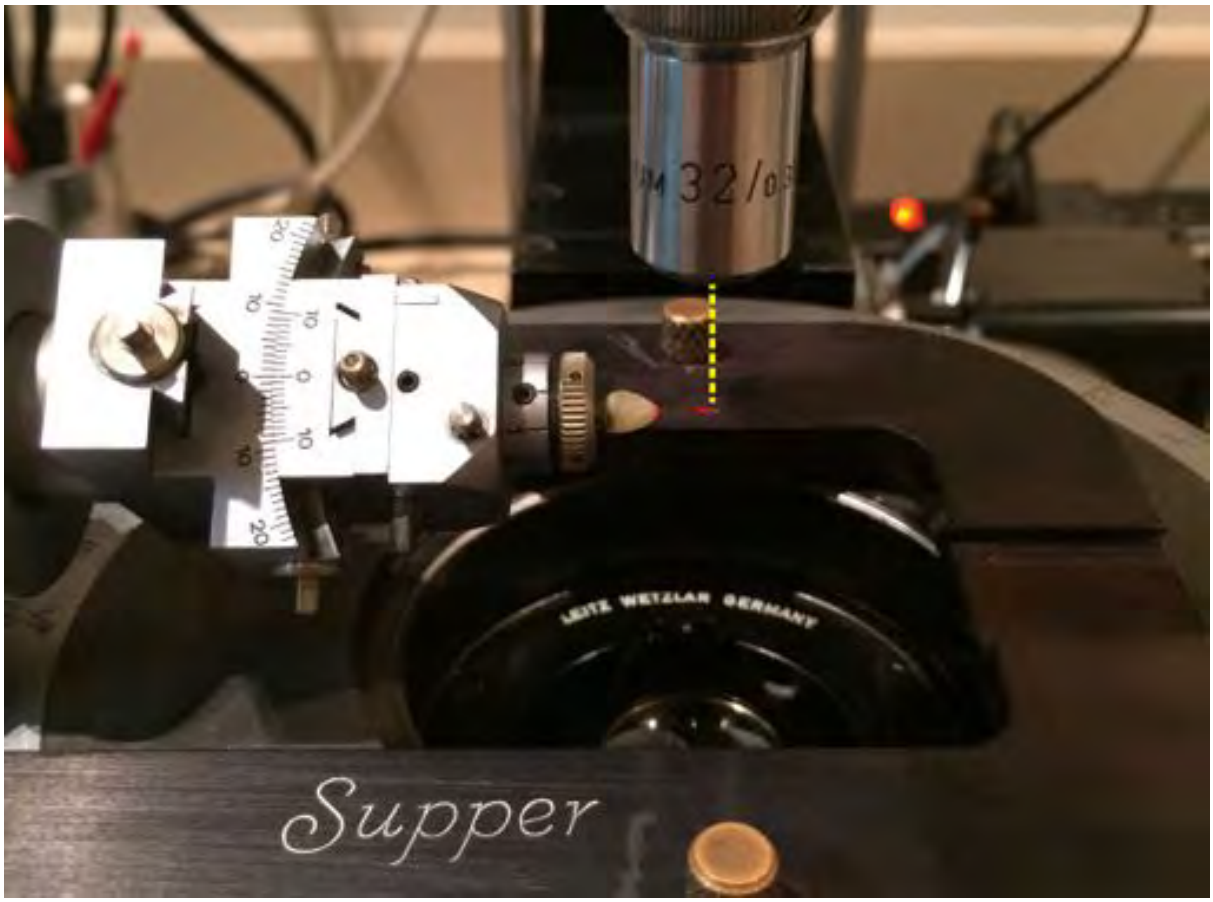


Figure 4.5 Here the crystal is brought to the line of view of the objective.

The “Y” direction can also be eyeballed by viewing down the axis of the spindle stage (**Figure 4.6**) and adjusting the “Y” translational screw (**Figure 4.7**).

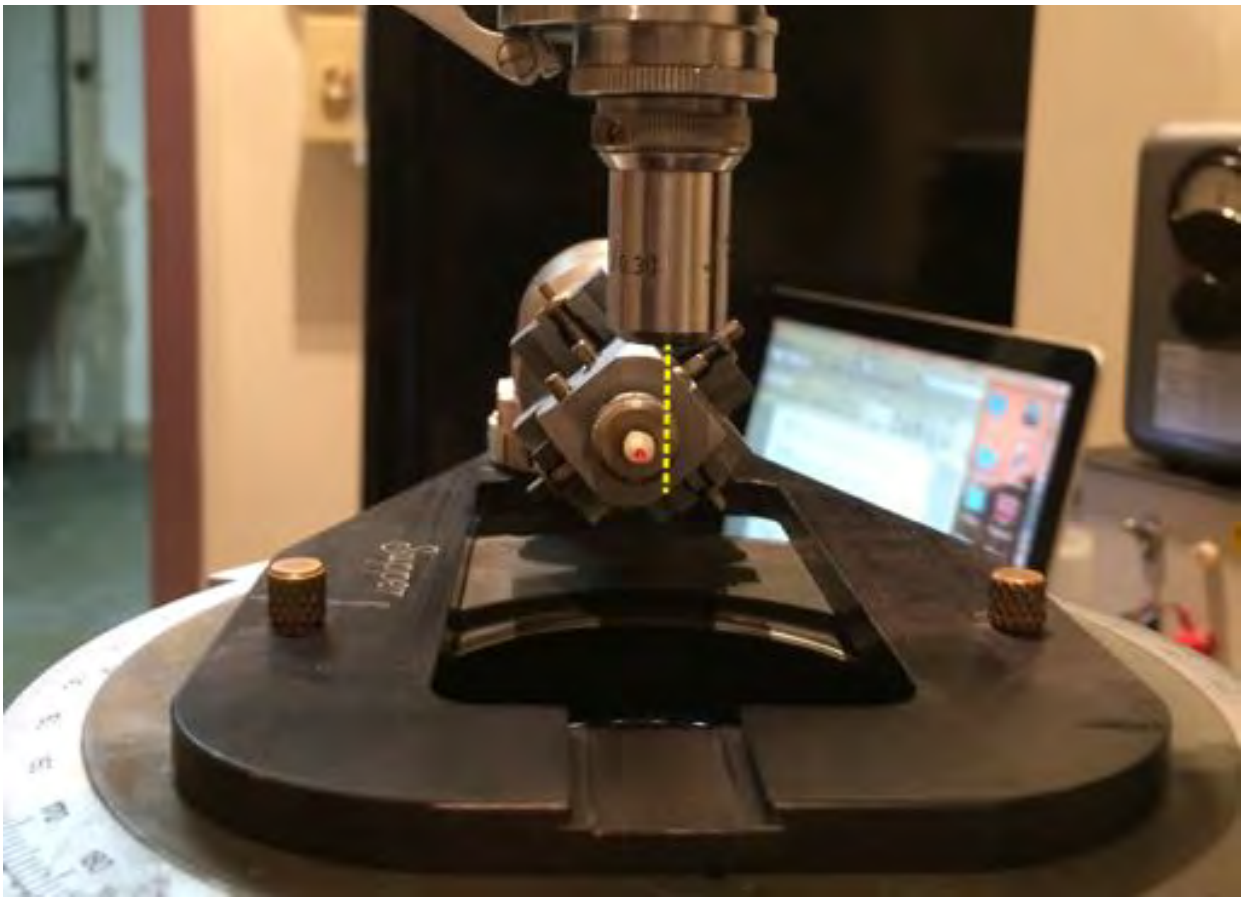


Figure 4.6 Viewing down the axis of the spindle stage, the crystal may be off-axis in the X direction like here. Translating the spindle stage moves the axis of rotation, whereas translating the goniometer screws translates the crystal to or away from the axis of rotation of the spindle.

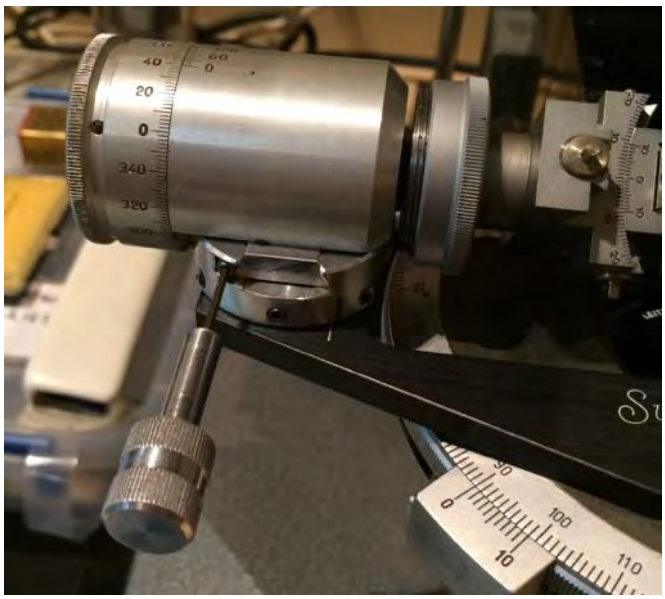


Figure 4.7 Adjusting the translation screw to move the axis of rotation. If the goniometer readout is zero for all axes, the crystal should be roughly near the axis of rotation.

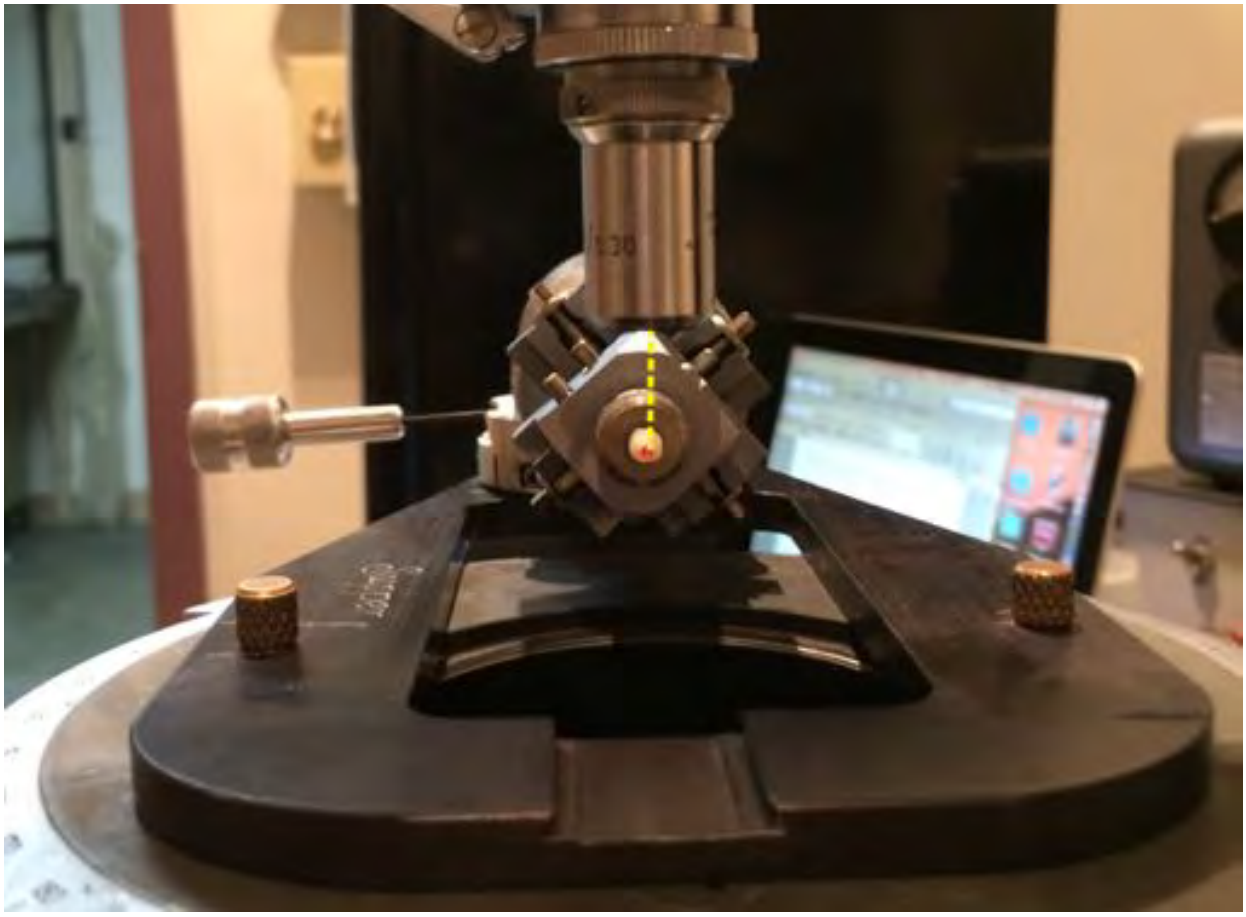


Figure 4.8 Since the crystal is roughly near the axis of rotation, translate the spindle stage X-axis to the line of view of the objective.

To this point, the crystal and/or glass fiber should be seen under the microscope, if not, rotate the spindle until they are in the field of view (**Figure 4.9**).

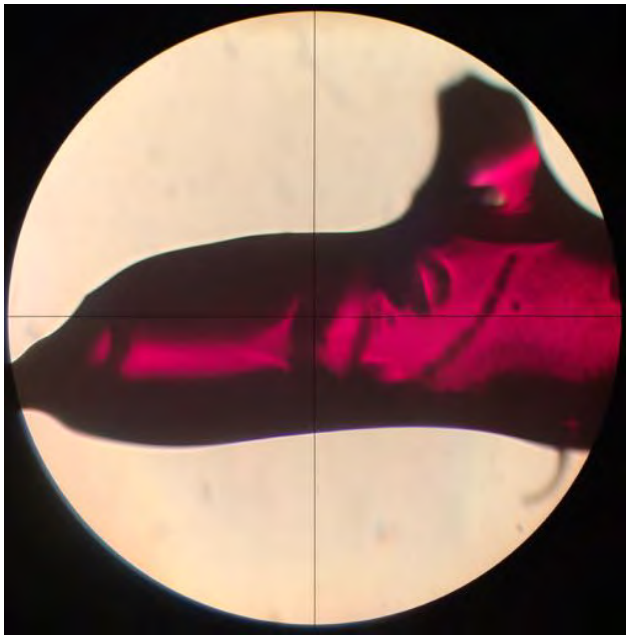


Figure 4.9 While rotating to spindle stage, the fiber coated in nail polish comes into view.

If the crystal is too far or not far enough out still, adjust the "X" translation screw on the goniometer or the spindle stage (**Figure 4.10 A-B**).

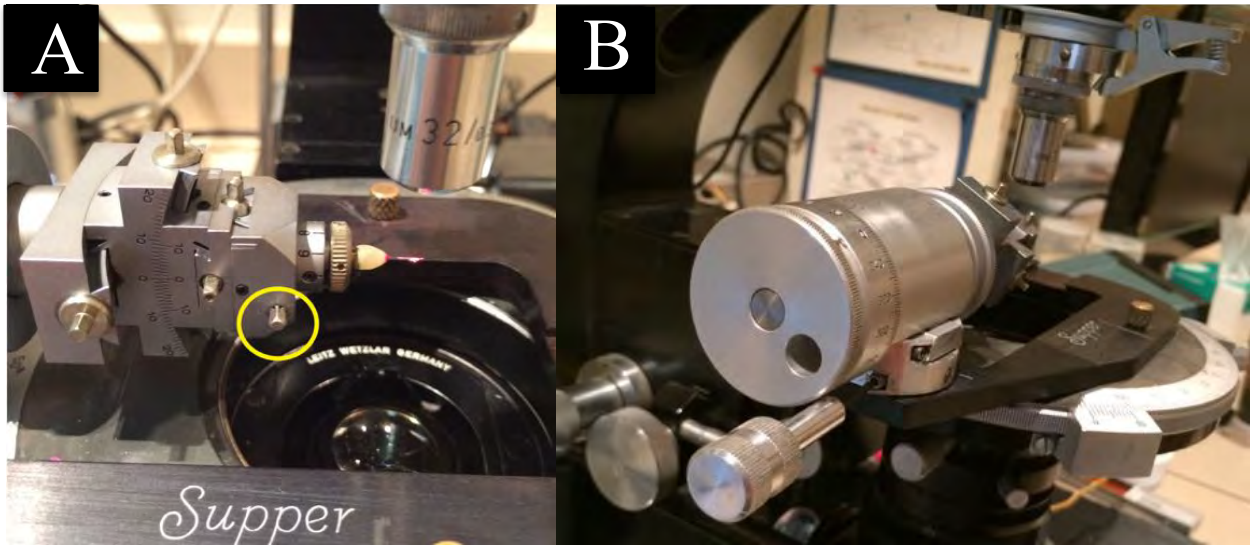


Figure 4.10 (A-B) Translate the crystal slightly back with either (A) the goniometer Y-translation screw (faces you), or (B) the spindle stage Y-translation screw (B).

Now that the "X" translation has brought the crystal in line with the North-South crosshair, the next step is to adjust the "Y" translation of spindle stage and goniometer. Normally, the "Y" translation of the spindle stage doesn't have to be adjusted much, but this will be an extreme example.

Set the spindle to 0˚ and look down the microscope (**Figure 4.11 A-B**).

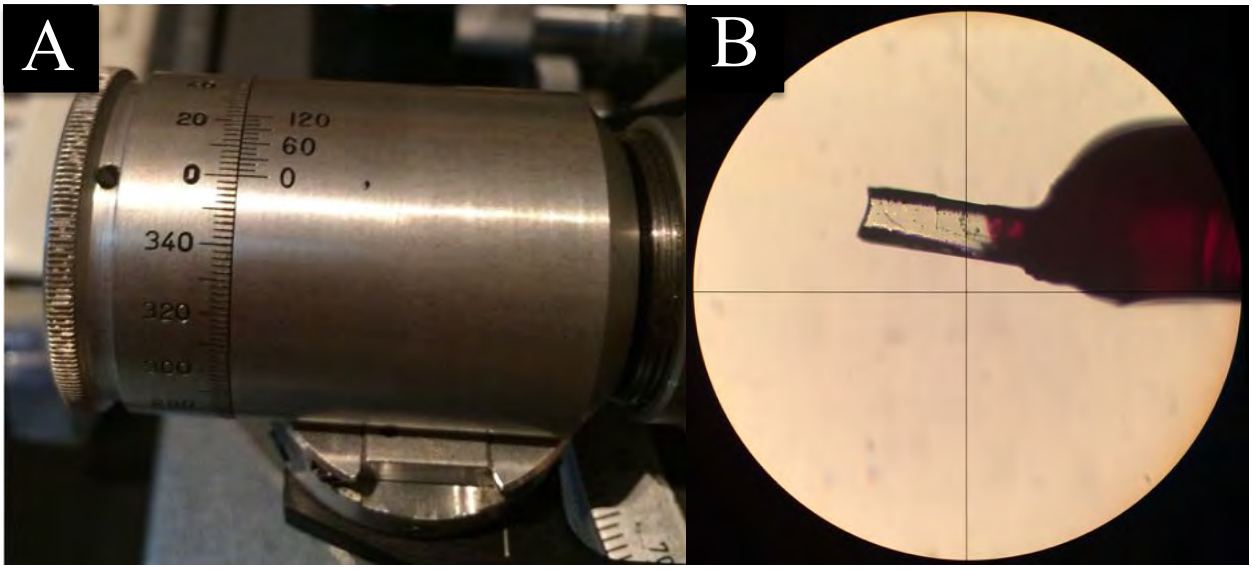


Figure 4.11 (A-B) Since the spindle stage is notched on a 90˚ interval to be fitted with the goniometer, centering adjustment are done on the goniometer screw that faces you at 90˚ intervals. Here is an example of the position of the crystal a 0˚

Even if the crystal is outside the field of view, you can adjust the goniometer's translation screw that faces you until the crystal is in the middle of the crosshair (**Figure 4.12 A-B**).

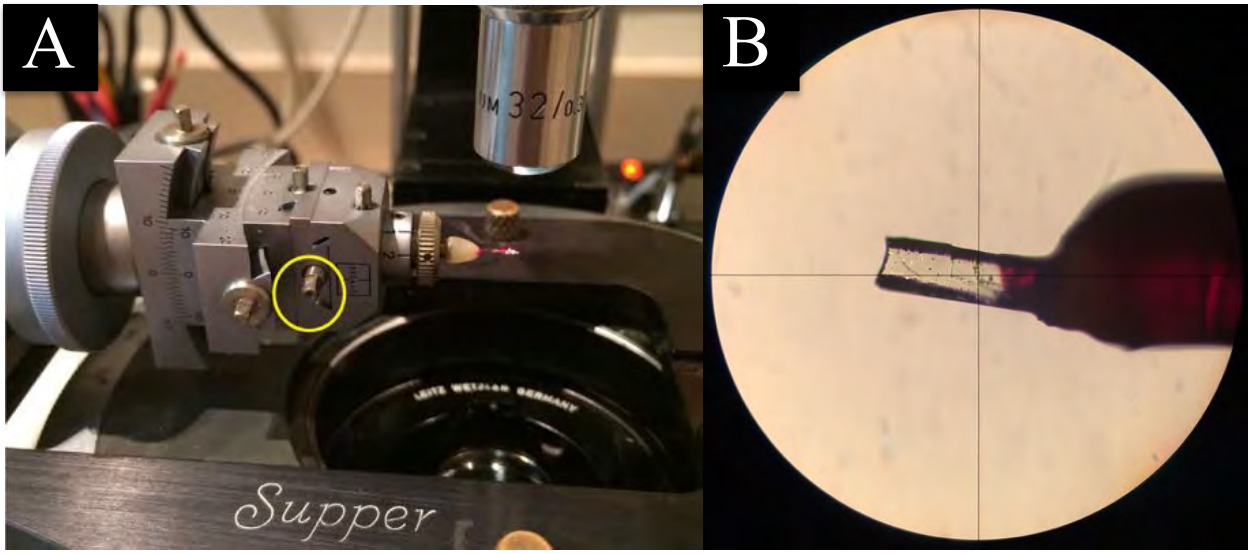


Figure 4.12 (A-B) (A) The location of the goniometer translation screw and (B) its influence on aligning the crystal. This goniometer also has lower arc screws for tilting axes.

Next, rotate the spindle to the 90˚ setting, and again, adjust the goniometer screw that faces you until the crystal is in the center (**Figure 4.13 A-C**).

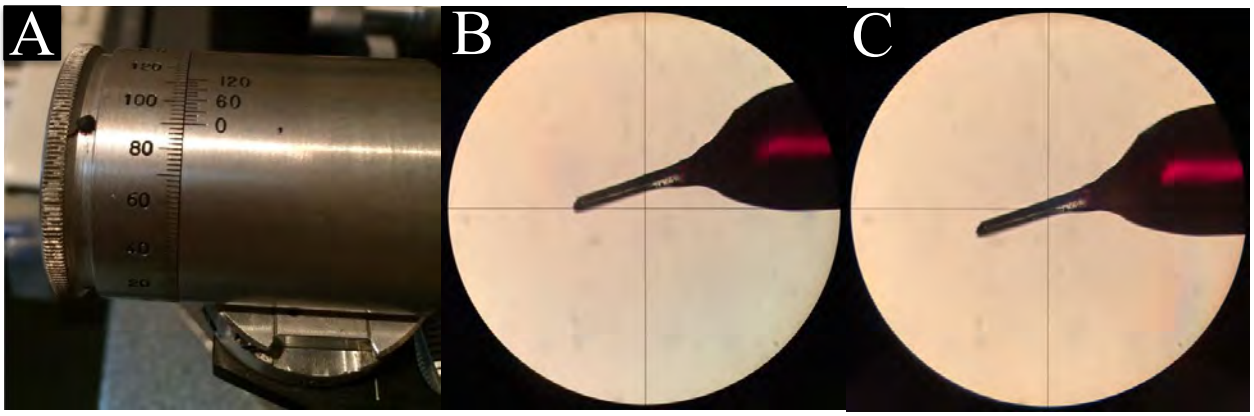


Figure 4.13 (A-C) Adjustments made 90˚ from the previous setting.

Now rotate to the 180˚ spindle setting. Notice that the crystal in this case is outside the field of view (**Figure 4.14 B**). To this point, if your crystal moves out of the field of view or out of center as you rotate, it means that the axis of the spindle stage is not centered. From here, we need to find the axis of rotation, center the axis, and bring the crystal to the axis.

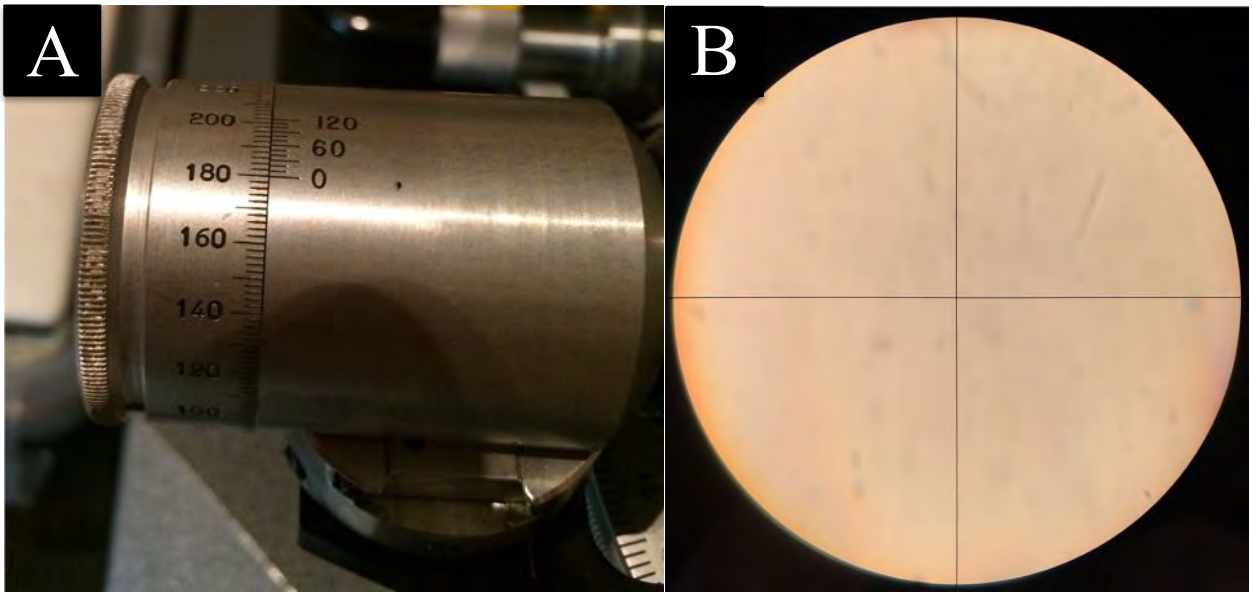


Figure 4.14 (A-B) 90˚ from the last setting, the crystal rotates out of the field of view. The reason for this is the axis of rotation is far enough off from the goniometers original zero position. Moving back to the previous setting while viewing under the microscope will give an indication of where the 'imaginary' axis of rotation is. Once this axis if found once, only very subtle adjustments will ever need to be made on the X-translation screw of the spindle stage, even with different goniometers/ specimen pins.

Go back to the 90˚ spindle setting. To determine where the axis of rotation is, rotate the spindle as you look down the eyepiece. The side (top or bottom) where the crystal swings wider is the side where the rotation axis is. In this case, while rotating the spindle, the crystal rotates slightly up then swings very wide to the bottom side and out of the field of view. This indicates that the rotation axis is somewhere below the East-West crosshair.

Rotate back to the 90˚ spindle setting. Since the rotation axis is somewhere below the East-West crosshair we want to adjust the spindle stage "Y" translation, such that the crystal and the rotation axis move up when looking down the eyepiece (Figure 4.15 A-D).

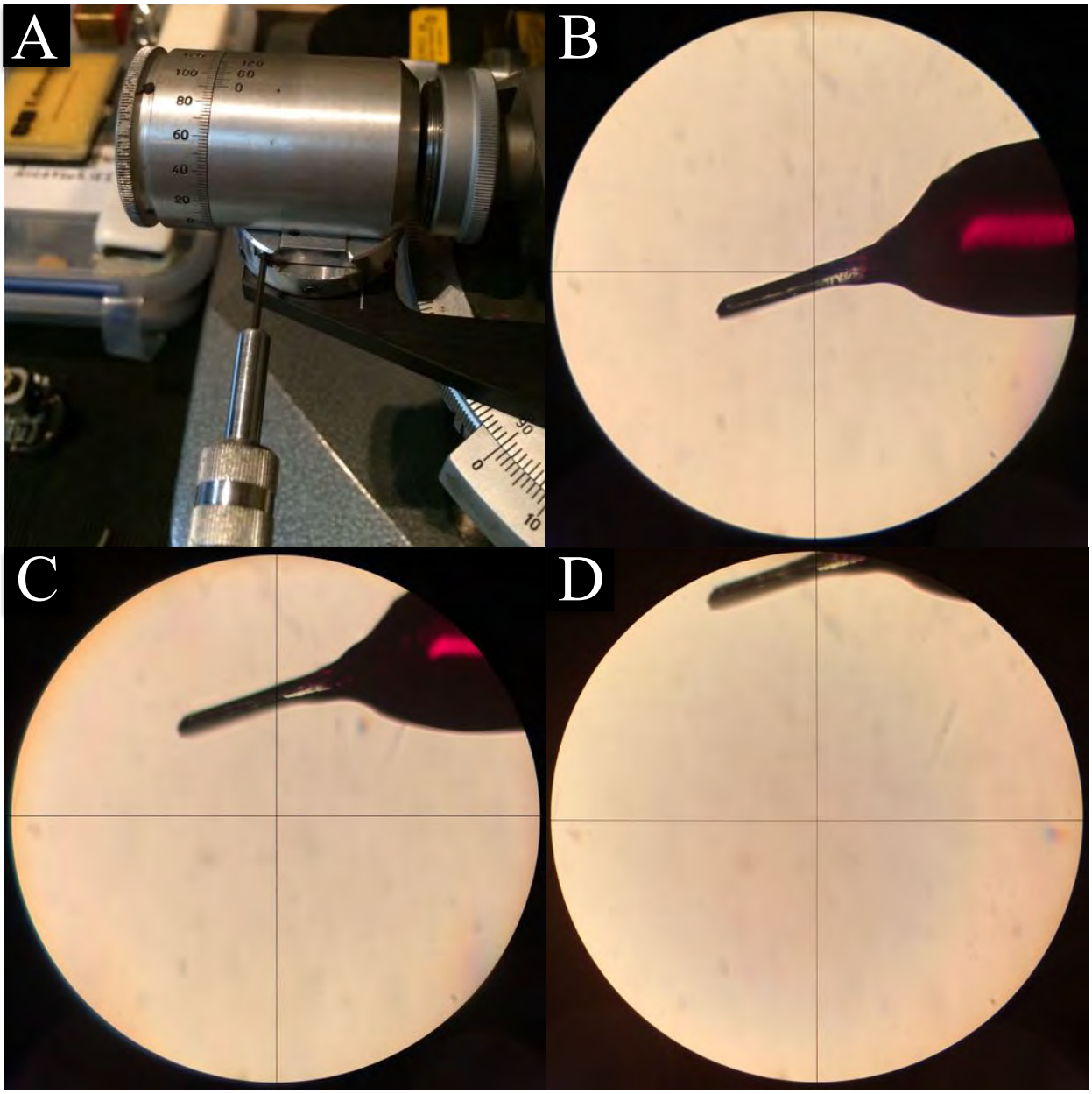


Figure 4.15 (A-D) (A) Moving the axis of the spindle stage from (B) the crystal's original position 'up' towards (C-D) the edge of the field of view.

With the crystal near the edge of the field of view in **Figure 4.14 D**, bring the crystal closer to the axis by adjusting the goniometer screw until the crystal is in the center of the crosshair (**Figure 4.16 A-B**).

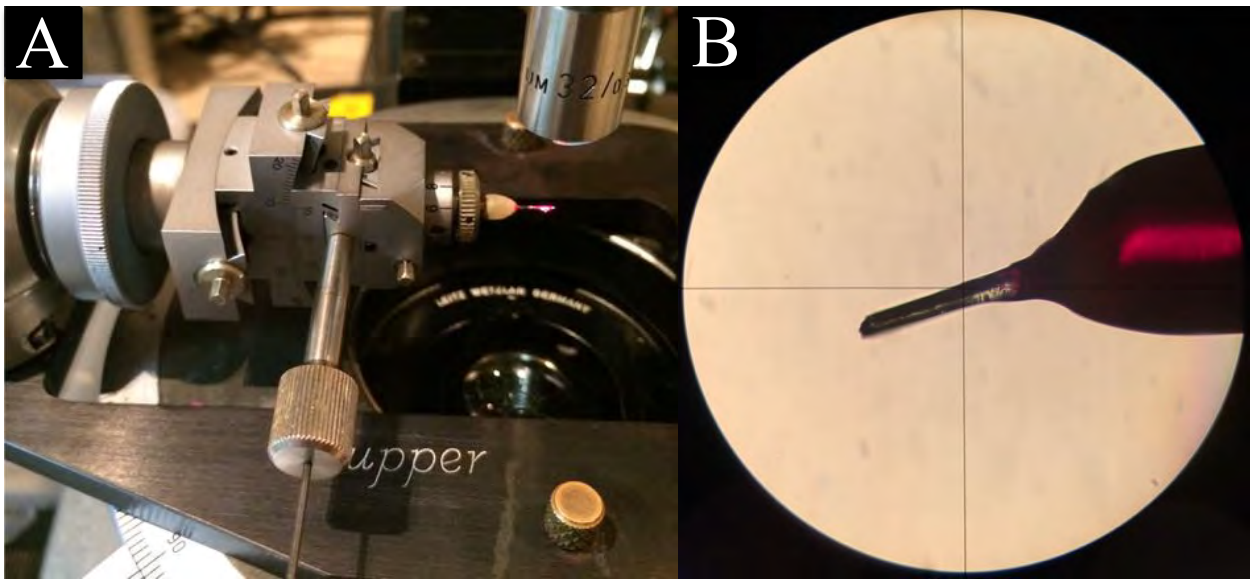


Figure 4.16 (A-B) Translating the crystal towards the axis of rotation using the goniometer translation screw in the 90˚ spindle stage position.

Next, rotate to the 0˚ spindle setting. The crystal is near the edge of the field of view (**Figure 4.17 B**).

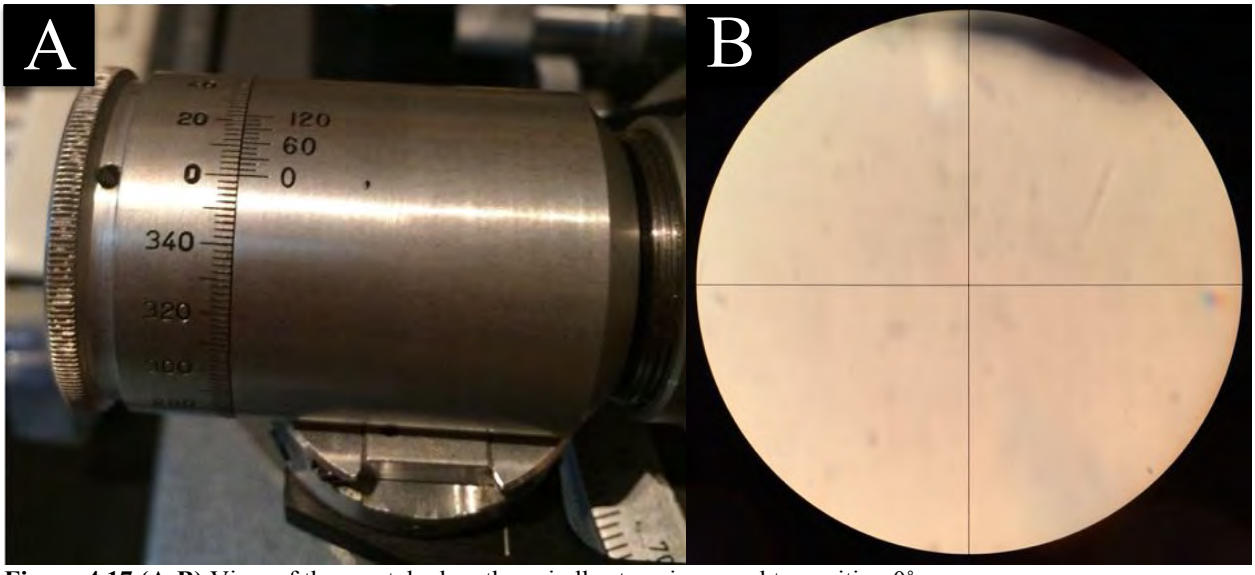


Figure 4.17 (A-B) View of the crystal when the spindle stage is moved to position 0˚.

Adjust the goniometer to bring the crystal to the center of the crosshair (**Figure 4.18 A-B**).

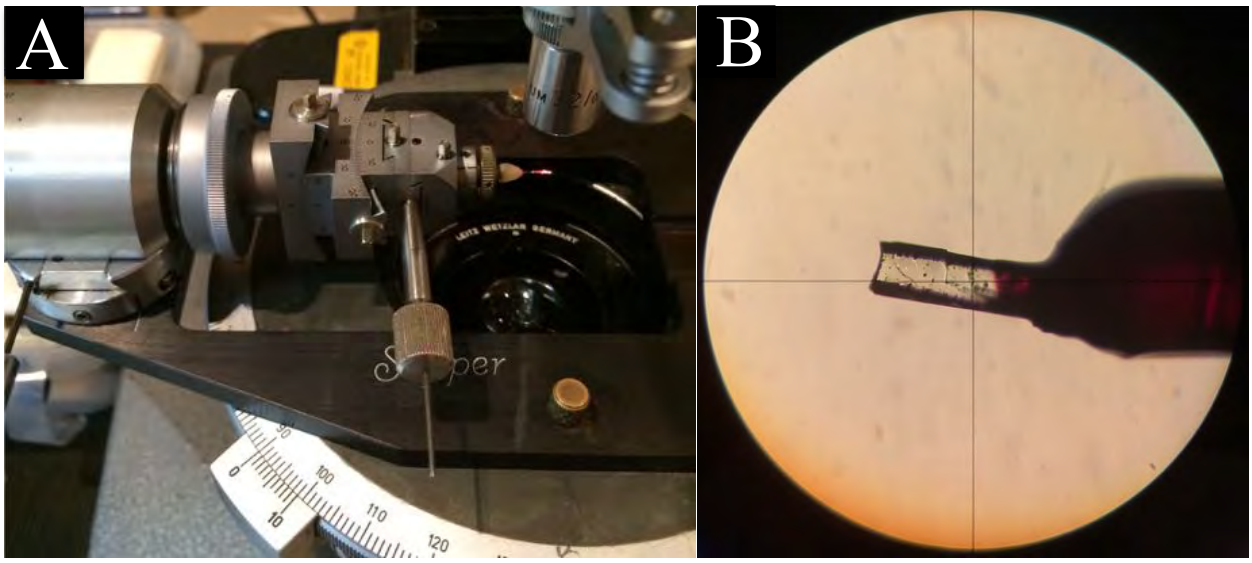


Figure 4.18 (A-B) Translating the crystal to the center crosshair using the goniometer screw in the 0˚ spindle position.

Now rotate the spindle and notice that the crystal is rotating around an axis that is in the field of view (**Figure 4.19 A-B**).

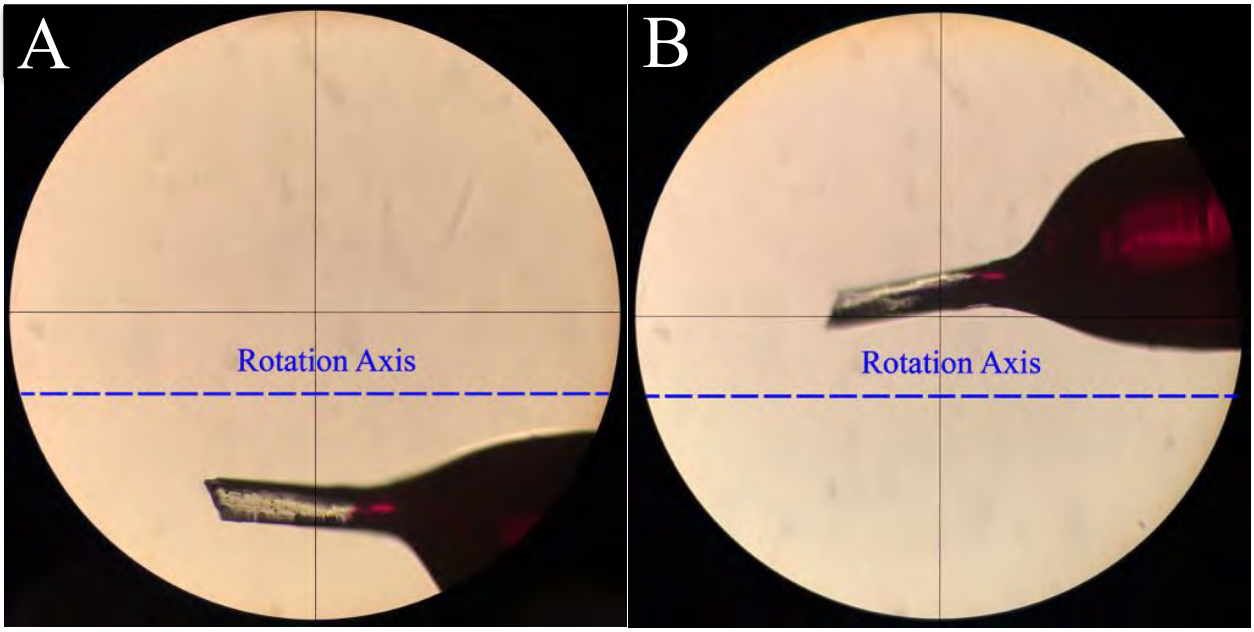


Figure 4.19 (A-B) Illustration of where the rotation axis is as the spindle is turned continuously.

Now the rotation axis can be brought to the East-West crosshair. For visual aid, you can rotate the crystal until it is halfway between the far "South" and "North" swing of the crystal in **Figure 4.19 A-B**.

After rotating the crystal to the rotation axis bring the crystal to center by adjusting the "Y" translation screw of the spindle stage (**Figure 4.20 A-C**).

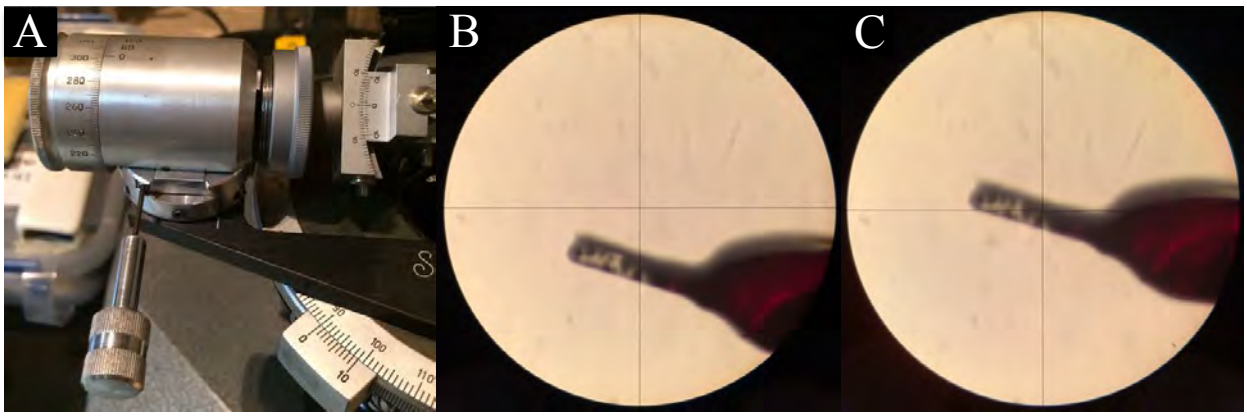


Figure 4.20 (A-C) (A) Adjustment of the X-translation screw of the spindle stage from (B) the crystal positioned near the axis of rotation to (C) it's final position.

Now that the crystal rotates around the East-West crosshair, the final adjustments on the goniometer for spindle setting 0˚, 90˚, 180˚, 270˚ will bring the crystal to center (**Figure 4.21 A-B**). You can also do fine adjustment of the spindle axis and goniometer from here from this point.

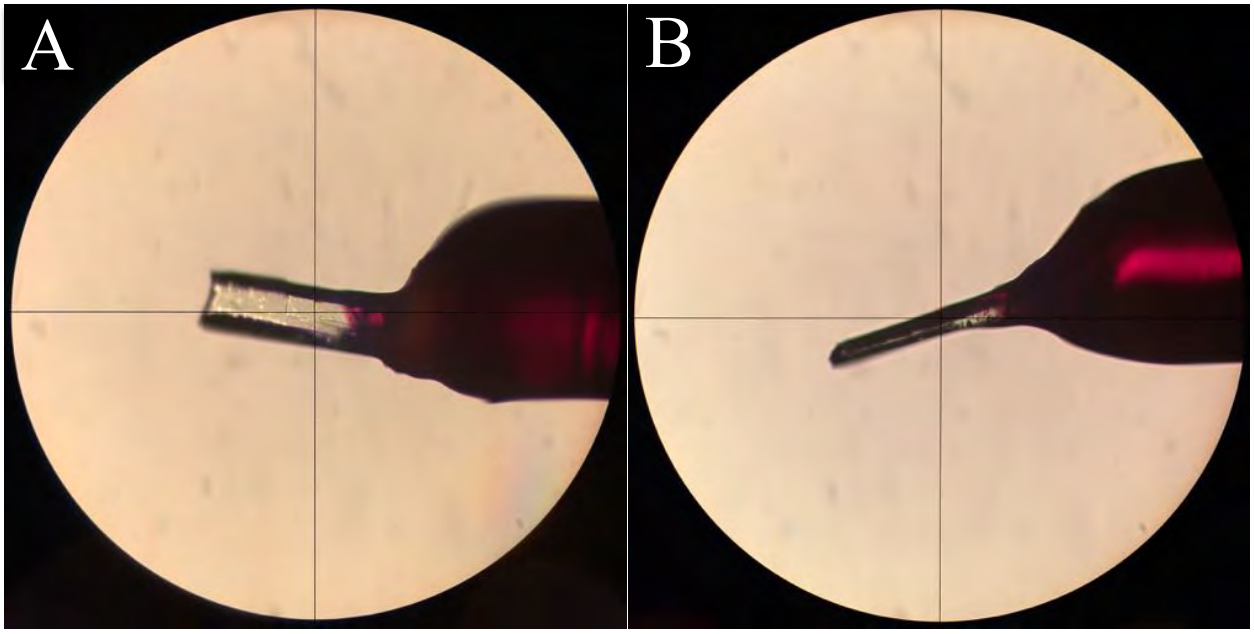


Figure 4.21 (A-B) Example of a centered crystal at spindle positions (A) 0˚ and (B) 90˚ after adjusting the goniometer screws.

4.2 Centering Procedure with Detent Stage

The goal with centering a crystal on a detent stage is keeping the crystal in the field of view, since it's unlikely that the crystal will stay in the center of the crosshairs as you rotate the spindle.

As with the procedure with a *Supper* Stage, make sure your objective is centered to the rotation of the microscope stage. The centering procedure with a detent stage is much like centering the *Supper* Stage but without a goniometer, which means you can center an axis rotation and bring the crystal to in line with the North-South crosshair.

Begin by locking the stage and tape one leg of the detent stage onto the microscope stage. The goal is to have the spindle pointing East with the crystal in the field of view.

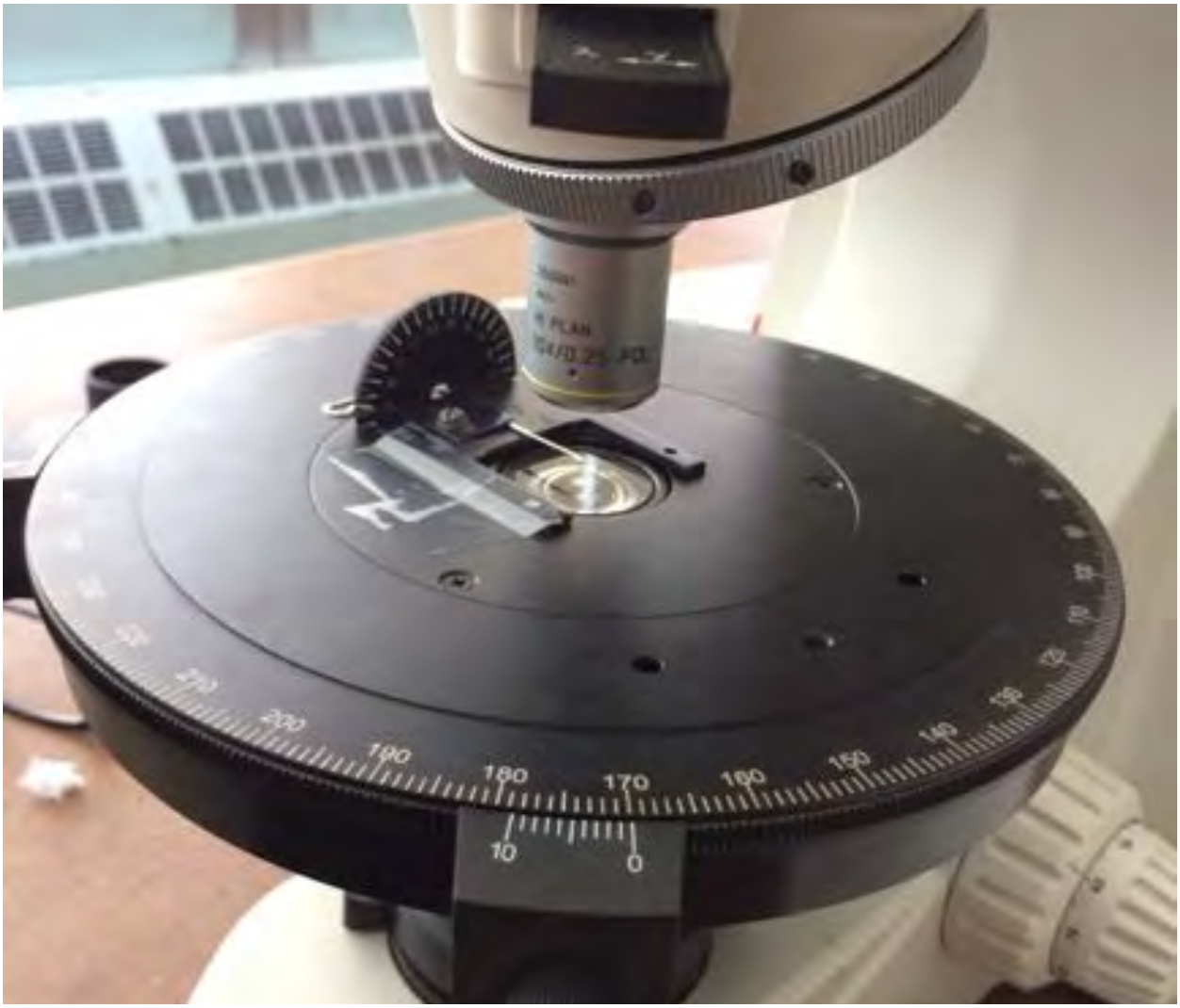


Figure 4.22 Leica student microscope with a *Wilcox* stage mounted with one piece of tape initially.

Move the stage such that the crystal is in line with the North-South crosshair. When you rotate the spindle, the crystal will move along the North-South crosshair.

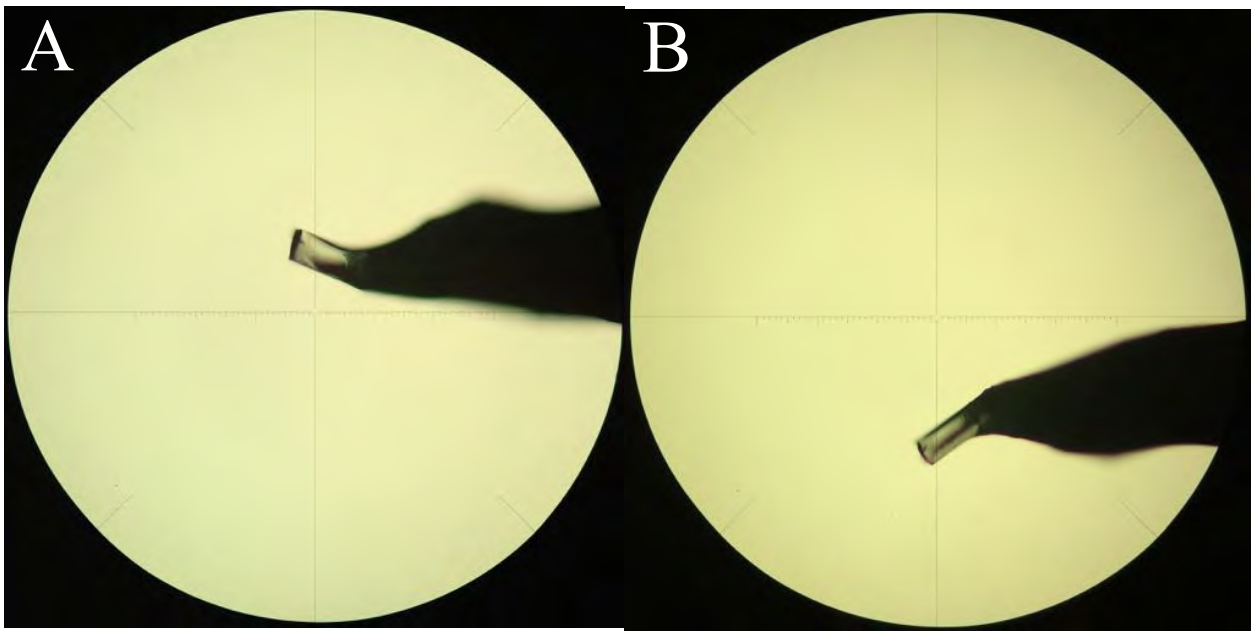


Figure 4.23 (A-B) The (A) northernmost and (B) southernmost swing of the crystal as the spindle stage is rotated. The rotation axis is halfway between these positions.

The axis of rotation is halfway between the furthest down, and furthest up that the crystal goes to as you rotate the spindle stage. Rotate the crystal to this halfway position.

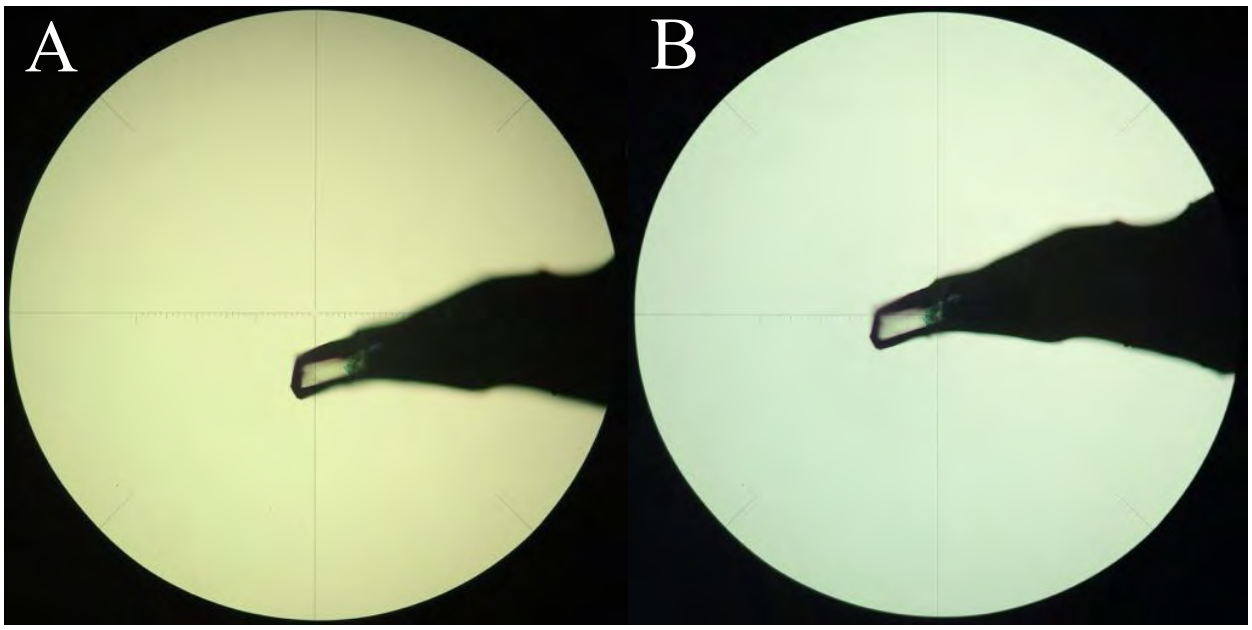


Figure 4.24 (A-B) The crystal position (A) near the axis of rotation and (B) at the crosshair after carefully moving the spindle stage.

Once the crystal is in position, move the detent stage so that the crystal is in the center of the crosshair and tape the other leg of the detent stage down. Keep the spindle in this position as you add an oil cell.

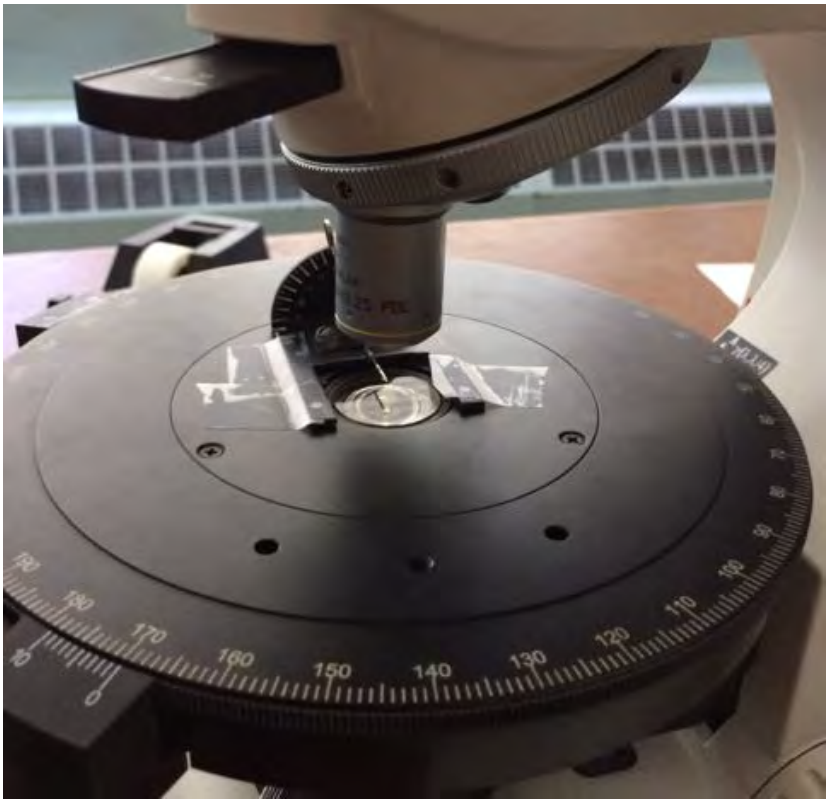


Figure 4.25 Spindle stage now with the other leg taped down.

Now the setup is ready for an oil cell that is the proper height.

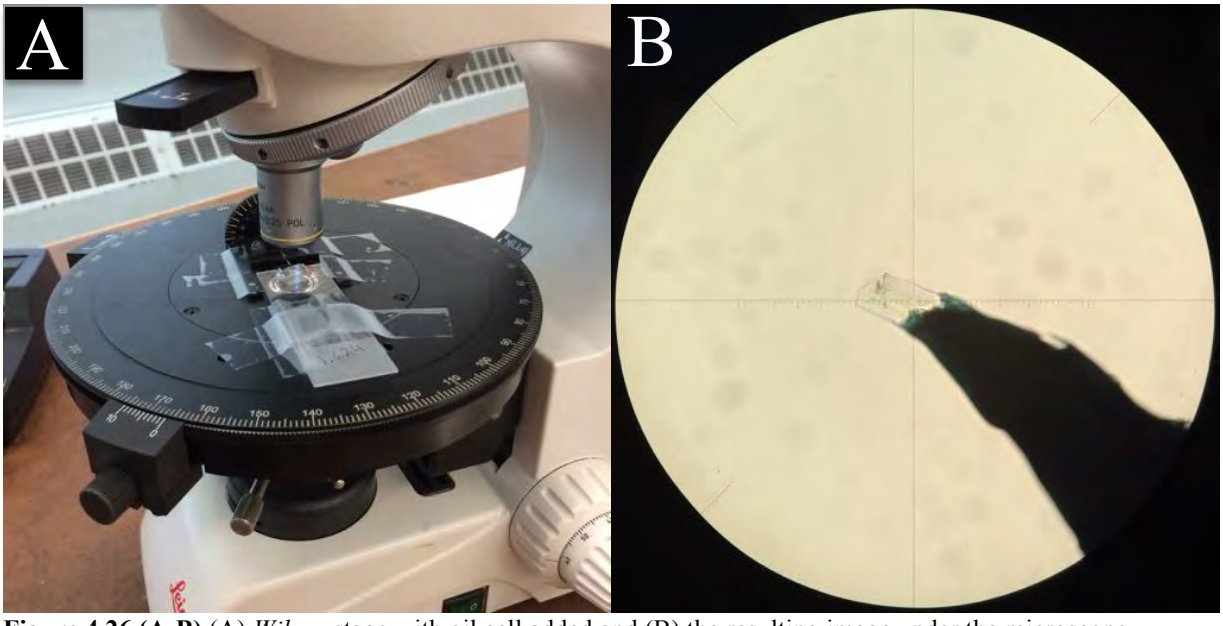


Figure 4.26 (A-B) (A) *Wilcox* stage with oil cell added and (B) the resulting image under the microscope.

5 Crystal Orienting

5.1 40 Degree Test

Now that the crystal is centered, an oil cell is used to reduce contrast from refraction and the 40 degree test may be executed. The procedure described here is from chapter 1 of *The Spindle Stage: Principles and Practice* (Bloss,1981). The 40 degree test is a quick test to see if the crystal is mounted in a good orientation for a program to solve its optical orientation. If the crystal passes the 40 degree test, it is in an ideal orientation for solving its orientation. The 40 degree test is performed by rotating the stage 40 degrees from its reference azimuth (see Section 5.2) and rotating the spindle until the crystal goes extinct (or not).

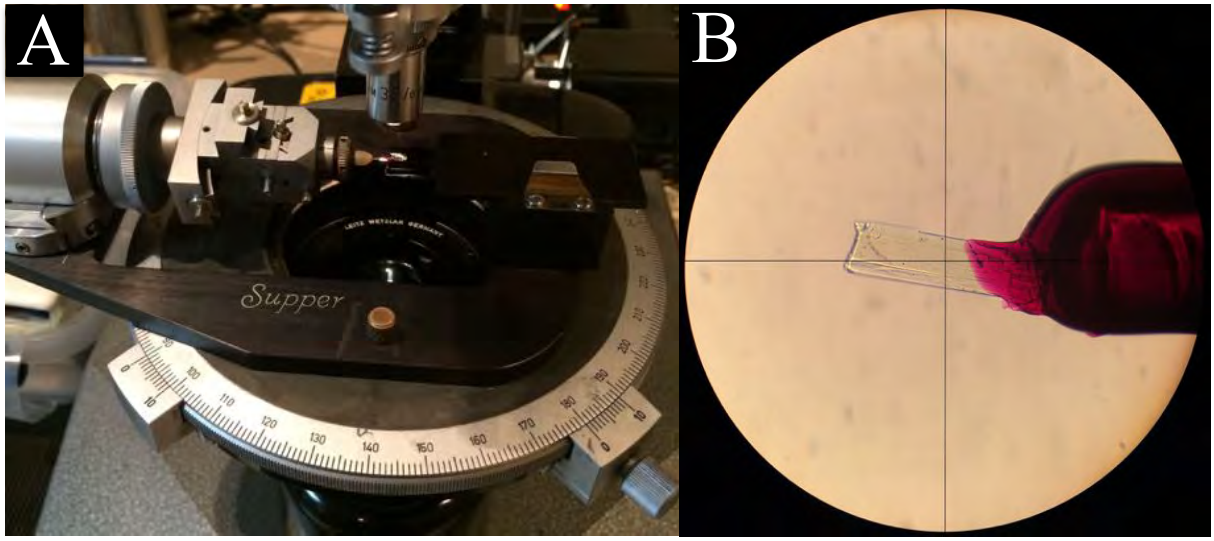


Figure 5.1 (A-B) Spindle stage with the addition of an oil cell to see extinction more easily.

If the crystal does not go extinct at any point when rotating the spindle, it does not pass the 40 degree test. This does not mean that the crystal is in a bad orientation though, it just means that it isn't in the absolute best orientation.

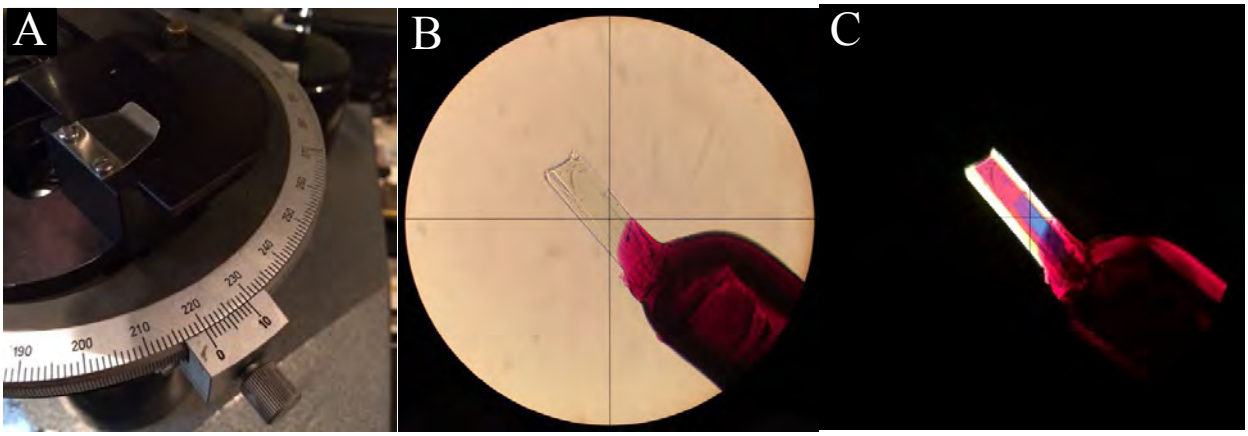


Figure 5.2 (A-C) (A) Positioned 40˚ from the approximate reference azimuth, (C) the crystal remains illuminated in XPL as it's rotated and does not pass the 40 degree test.

When rotated 35 degrees away from the reference azimuth, the crystal passes the 35 degree test, so the crystal is still in a very good orientation for solving for its optical orientation.

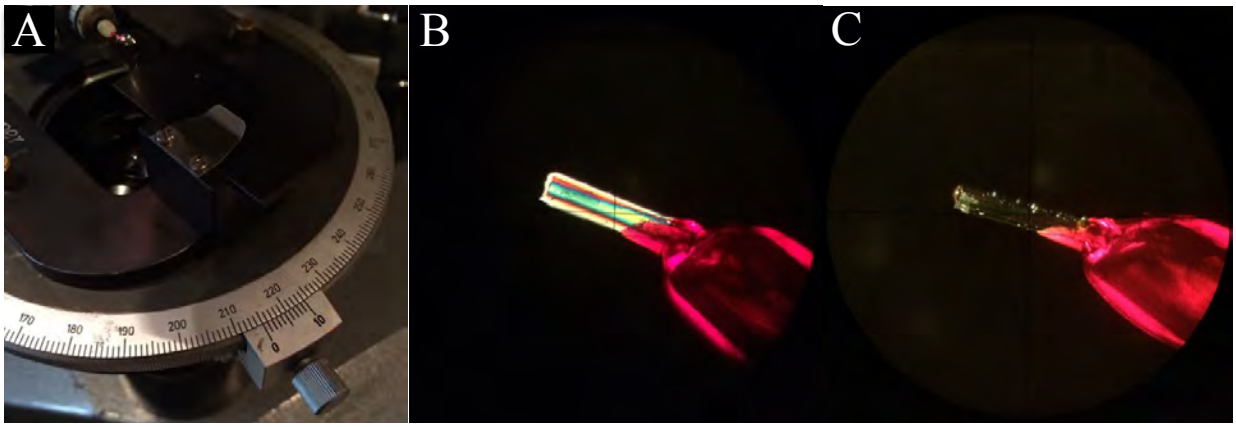


Figure 5.3 (A-C) Positioned 35˚ from the approximate reference azimuth, as rotated, (C) the crystal goes extinct and passes the 35 degree test.

5.2 Reference Azimuth

The reference azimuth is the microscope stage reading where the spindle points directly East (this example) or West. It is a zero position for which all microscope stage readings a relative to for the programming of EXCALIBR or EXCELIBR. Determining the reference azimuth also ensures that extinction positions account for error in the alignment of your polarizers. The reference azimuth can also be used as a reference point from which to perform the 40 degree test.

5.2.1 Vernier Scale

If you do not know how to read a vernier scale, now is the time to learn, as this can improve the data and output. A vernier scale is a 0˚ to 10˚ scale on the outer edge of the stage. 10˚ on a vernier scale only spans 90% of the distance that 10˚ spans on the stage, which allows a user to read decimal degrees.

To read a vernier scale, read where the 0 tic mark aligns on the stage. The decimal is equal to whichever tic mark on the vernier scale aligns with a tic mark on the stage. In the example in **Figure 5.4**, this stage reading is 195˚ plus the decimal tic mark on the vernier scale that aligns with another tic mark. In this case, the 200˚ tic mark aligns with a tic mark on the vernier scale, which is the 0.5˚, so this stage reads 195.5˚.

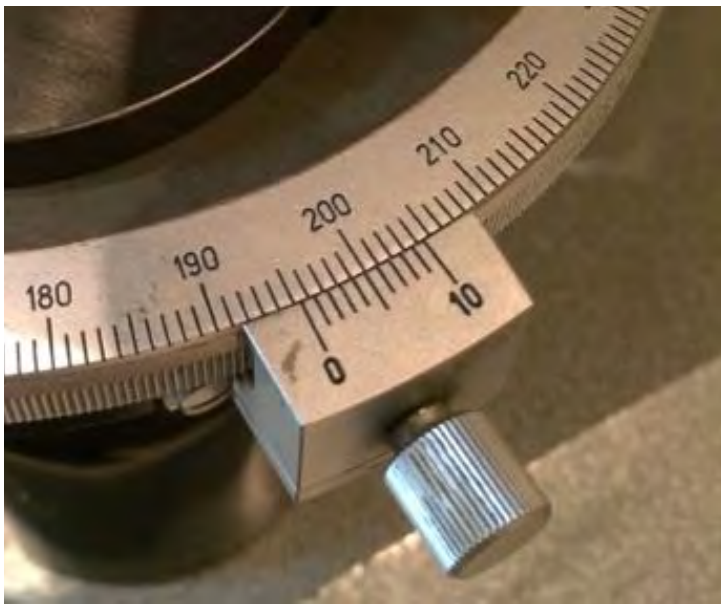


Figure 5.4 Vernier scale readout of 195.5

5.2.2 Finding the Reference Azimuth

To find the reference azimuth, first point the spindle so that it is facing East. If you have a *Supper* Stage which screws into the microscope stage, it should be reading close to 0˚ or 180˚.

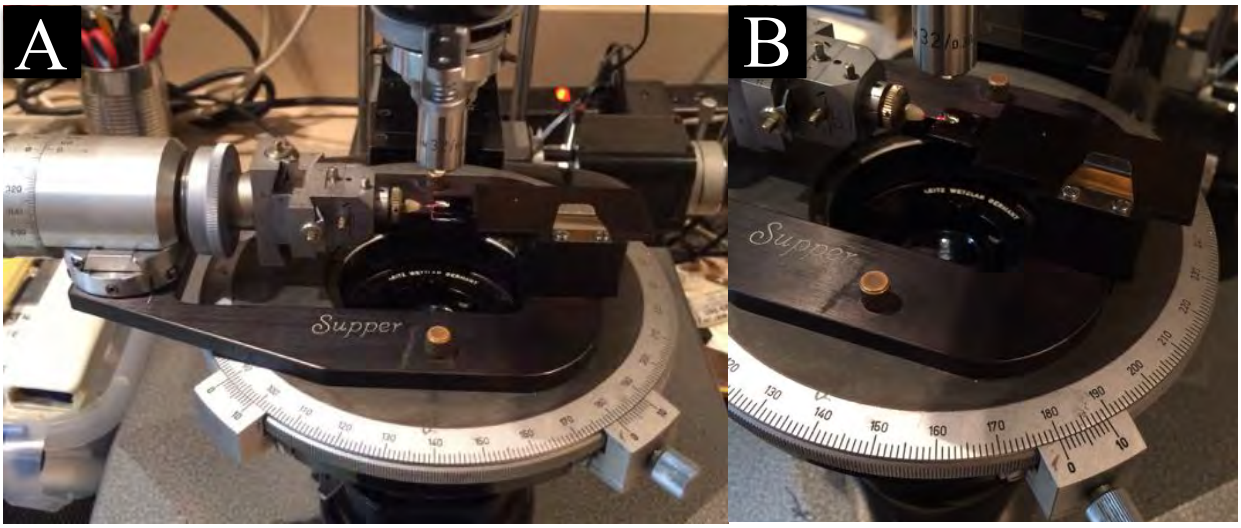


Figure 5.5 (A-B) Spindle stage aligned along an approximate reference azimuth position at 180˚. In this case, by design, 180˚ is a good reference azimuth to use as long as the polarizers are aligned correctly.

From this position, set the spindle to read 0˚.

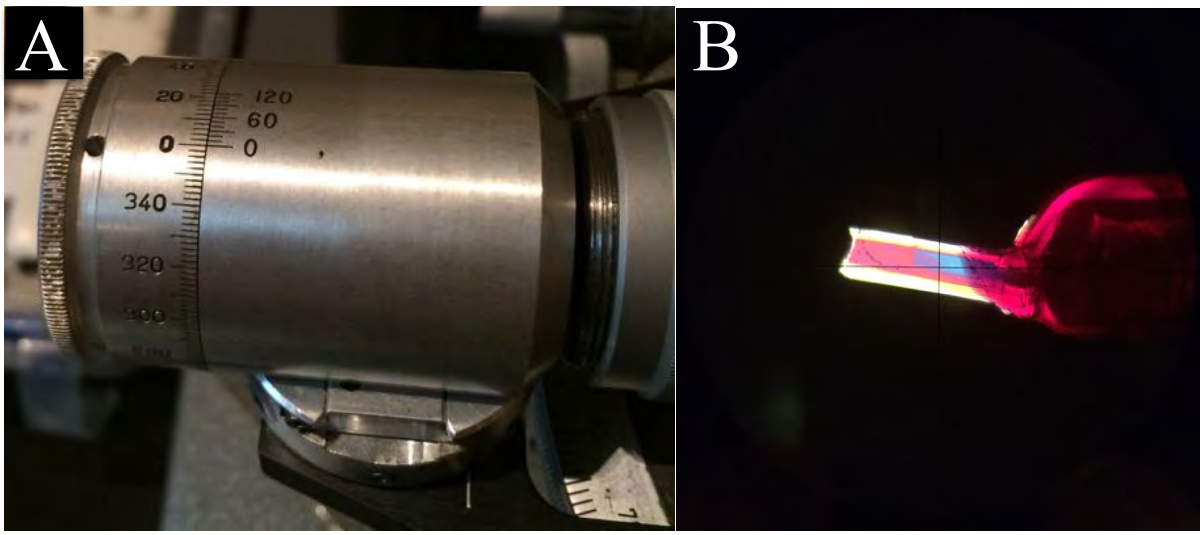


Figure 5.6 (A-B) (A) Spindle aligned roughly East-West in the 0˚ spindle position. (B) The crystal should be visible in this position if it's mounted in a good orientation.

Now rotate the stage to the nearest extinction position. In this case, the stage needed to be rotated 15.5˚ clockwise (stage reading is 195.5˚). Record this stage position.

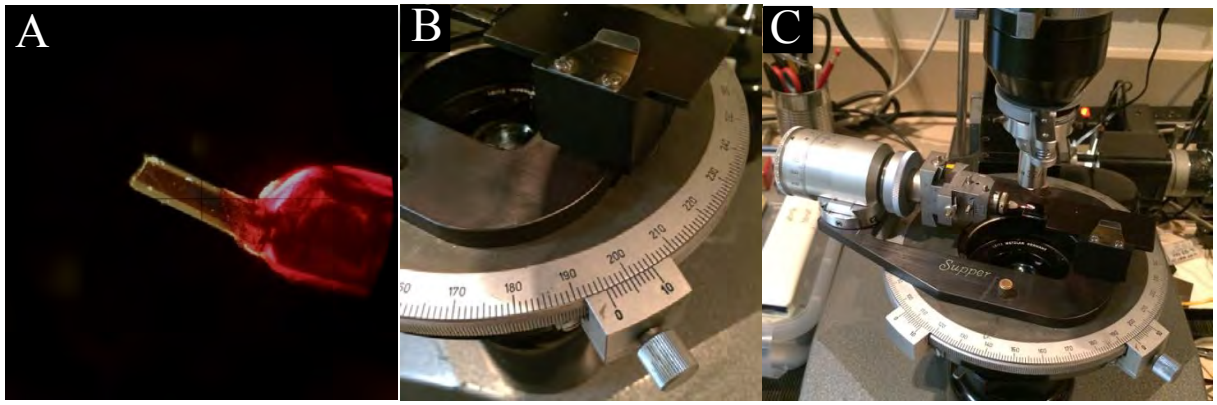


Figure 5.7 (A-C) Crystal at extinction for one of the values used to calculate reference azimuth.

Now set the spindle to the 180˚ setting and rotate the opposite way to an extinction position. Record this stage reading.

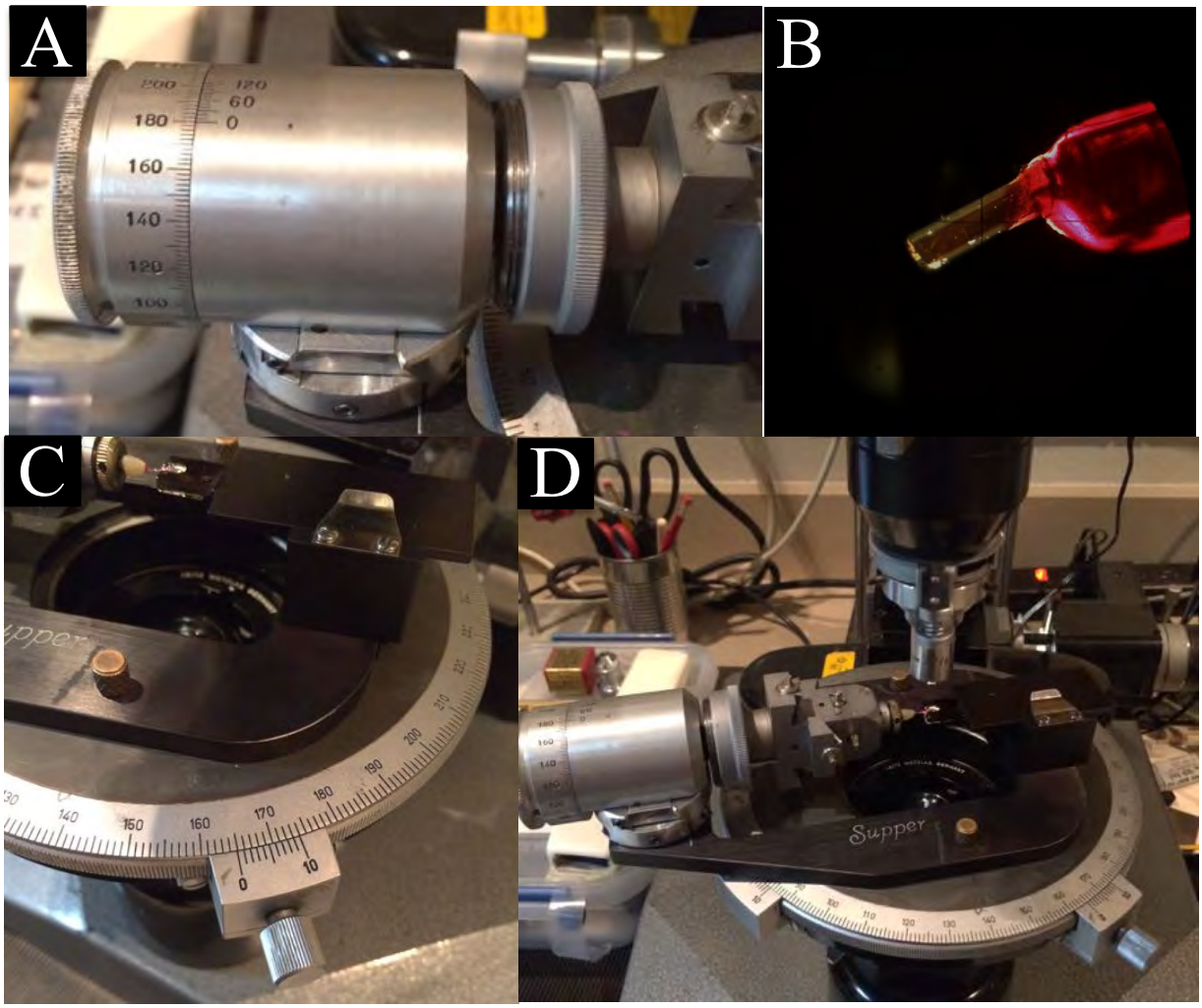


Figure 5.8 (A-D) The second value used for calculating reference azimuth.

In this example, the first stage reading is 195.5˚ and the second stage reading is 164.9˚. The reference azimuth is the microscope reading between these two readings: (195.5+164.9)/2 = 180.2

If one of your readings is on one side of zero, and another reading is on the other side of zero, this equation will not work. For example, if the spindle stage is mounted so that the direction pointing East is somewhere near zero, say that one of your stage readings is 15˚ and the other is 343˚. (15+343)/2 would not equal the reference azimuth because one value is on the other side of zero. If this happens, the values below zero should be treated as "negative" degrees away from zero. 343˚ becomes -17˚ and the equation (15+(-17))/2 is now valid. The resulting reference azimuth is -1, which is the same as 359˚.

An optional final step is to refine the reference azimuth, which is an operation similar to the refined reference azimuth operation of ExcalibrW (Gunter, 2004). To do this, select Data > What-If > Goal Seek under the Ribbon tab. For the 'Set cell:' entry, use the p-value cell (E22). For the 'To value:' entry, use zero. For the 'By changing cell:' entry, use the reference azimuth cell (F2). What this will do is initiate an iterative algorithm that will attempt to minimize the

overall difference between calculated and observed data points. This can also be used for extinction angles that are near an optic axis, where the error is typically greater.

5.3 Locating Principle Vibration Directions

Now that you have a crystal that is mounted in a good orientation and you have a reference azimuth you can now begin entering extinction data. Remember to enter the reference azimuth in the "Mr" box and the extinction angles under "Ms" in the 'Biaxial Input' tab.

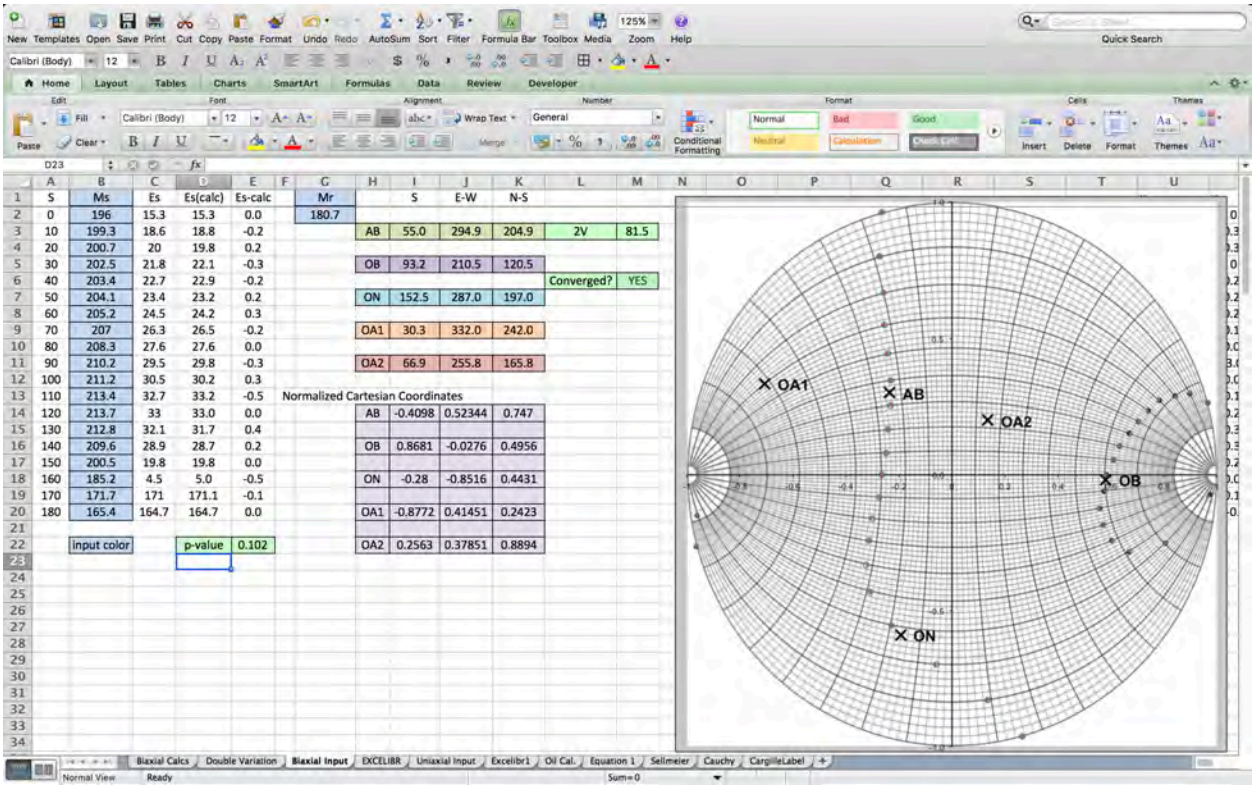


Figure 5.9 Example of the Biaxial Input tab, where extinction data is entered in the Ms column for the listed spindle setting S. Also entered is a reference azimuth Mr.

5.3.1 Optic Sign

After entering all 19 extinction positions, coordinates for the principle vibration directions are given in the middle part of the tab (**Figure 5.10**). The output lists the coordinates of the principle vibration directions in terms of their geometry, since there isn't a way for the program to figure out which refractive index direction is which.

S stands for spindle, and it is the spindle angle setting for bringing a vector into the plane of the microscope stage. E-W and N-S stand for East-West and North-South respectively, and they denote the microscope stage coordinates for aligning an optical vector with the lower polarizer for refractive index measurement. If you have an East-West lower polarizer, use the E-W coordinate.

AB stands for acute bisectrix, and it is the vector that bisects the acute angle between optic axes. OB stands for obtuse bisectrix, and it is the vector that bisects the obtuse angle between optic axes. ON stands for optic normal, and it is the vector normal to the intersection of the optic axes.

	S	E-W	N-S		
AB	55.0	294.9	204.9	2V	81.5
OB	93.2	210.5	120.5		
				Converged?	YES
ON	152.5	287.0	197.0		
OA1	30.3	332.0	242.0		
OA2	66.9	255.8	165.8		

Figure 5.10 Output section of the Biaxial Input tab, which gives 2V angle, whether or not the algorithm converged, and the coordinates for aligning an optical vector either East-West (E-W) or North-South (N-S) depending on the orientation of the privileged direction of the lower polarizer.

In biaxial positive crystals, AB is gamma and OB is alpha. In biaxial negative crystals, OB is gamma and AB is alpha. In both cases the optic normal is always beta (**Figure 5.11 A-B**).

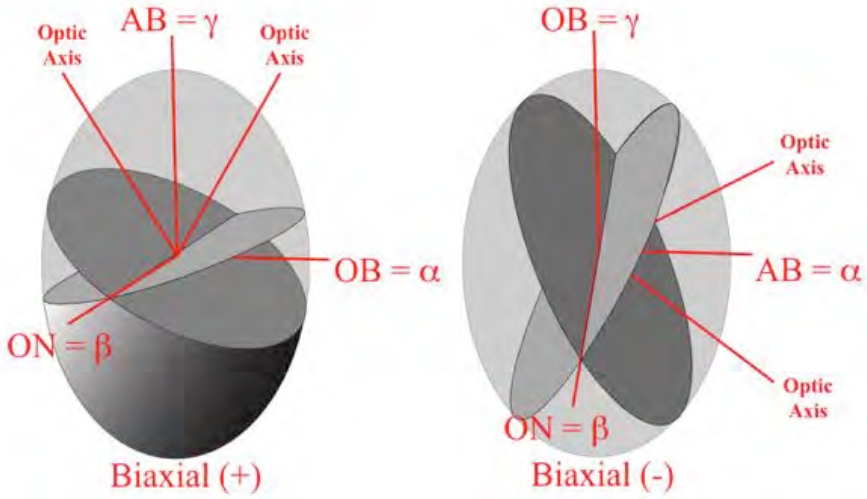


Figure 5.11 Indicatrix models for biaxial (+) and biaxial (-) crystals, showing the relationship

between optical geometry and principle refractive index directions. From The Microscope (Steven & Gunter, 2018).

To determine the optic sign of a crystal, begin by aligning the acute bisectrix line with the lower polarizer. The microscope in this example uses an East-West lower polarizer. Insert the upper polarizer to verify that the crystal is at extinction (**Figure 5.12 B**).

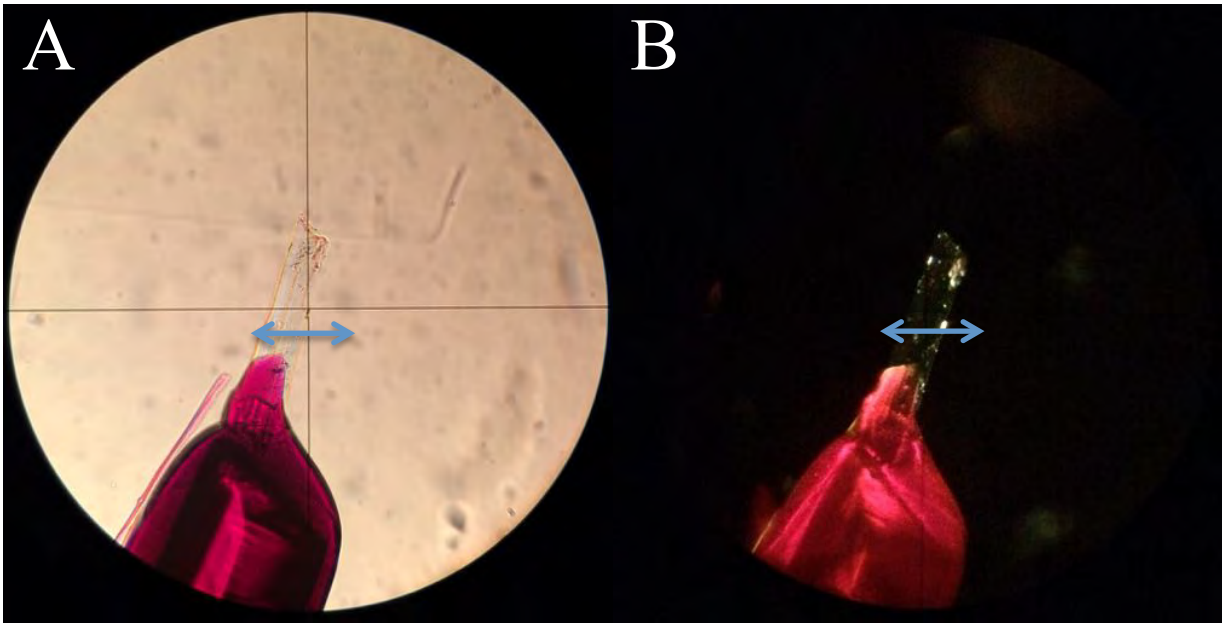


Figure 5.12 (A-B) (A) Example of the acute bisectrix vector "AB" aligned in the East-West direction. (B) If a vibration direction is indeed aligned with a polarizer, the crystal should be at extinction.

Now rotate the microscope counterclockwise 45˚ (clockwise for a North-South lower polarizer). This is to align the AB vector line with the N vibration direction (or gamma) of the waveplate. Insert the waveplate (**Figure 5.13 B**). Here the retardation colors go from 4th order colors to 3rd order colors, indicating subtraction. If AB subtracts then AB must be alpha and the crystal is biaxial (-). If AB adds, then AB is gamma and the crystal is biaxial (+).

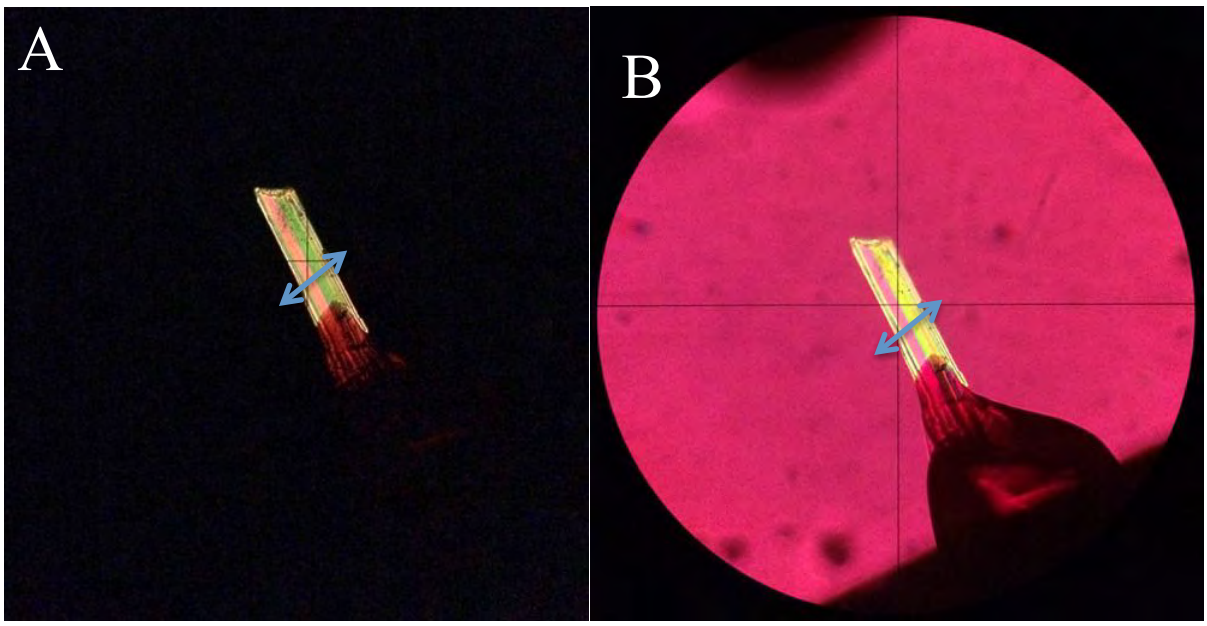


Figure 5.13 (A-B) (A) Acute bisectrix vectoreinclined 45˚ to the lower polarizer to show interference and (B) in parallel with the gamma vibration direction of the waveplate. The acute bisectrix vector subtracted and is the alpha refractive index, and the crystal is biaxial (-).

5.4 Locating Crystallographic Axes

Determining where crystallographic axes lie relative to optical vectors is another great way for identifying minerals. Even if there is overlap in optical properties, sometimes the angles between crystallographic and optical vectors differ significantly between mineral groups. For example, augite and most hornblende species have similar refractive index ranges, 2V, and they share the same crystal system. Despite this, the angle between the Z optical vector and the c crystallographic axis is very different. In augite, the angle between Z and c is greater than 35˚ and with hornblende and other amphiboles, the angle between Z and c is less than 34˚ but is almost always near 15˚, as with tremolite, actinolite, magnesiohornblende etc.

5.4.1 X-ray method

The X-ray method for finding crystallographic axes is the best way for definitively finding the positions of the a, b, and c axes. The X-ray method involves a coordinate transformation of the geometry of a single crystal X-ray diffractometer, to the geometry of a microscope (Gunter, 2001).

To find the coordinates of a, b, and c, after X-raying a crystal to determine its unit cell, the orientation matrix is found in a txt document stored in the same folder that X-ray frames are stored. The orientation matrix lists the coordinates of the reciprocal lattice vectors. Normalize and convert the orientation matrix to direct space using the methods outlined by (Gunter, 2001).

5.4.2 Visual method

Another way to determine the orientation of crystallographic axes is visually. As described in chapter 7 of (Bloss, 1981), planes that are normal to crystallographic axes can be used to locate crystal axes by aligning the plane perpendicular to the lower polarizer and recording that position. The difficulty with the spindle stage is that it is hard to accurately determine if an axis (not bounded by planes normal to the axis) is in the plane of the stage and not angled up or down.

Proposed here is the solution to this problem when dealing with crystals that are elongate along an axis. Knowing two planes that contain the same crystallographic axis, the intersection of these two planes is the orientation of the crystallographic axis. Planes can be mathematically defined by their normal vectors, and taking the cross product of two normal vectors that intersect is the same as finding the intersection of two planes.

In this example, the length of the crystal is the c axis, therefore, aligning the apparent length of the crystal with the North-South crosshair will in turn align the vector normal to a plane containing c with the East-West lower polarizer (this example). Doing this with two different spindle settings (preferably about 90˚ apart) will give two planes that intersect along the c axis. In this example, I used the coordinates of the optic normal and a random plane containing c because c lies in the optic axial plane for this mineral.

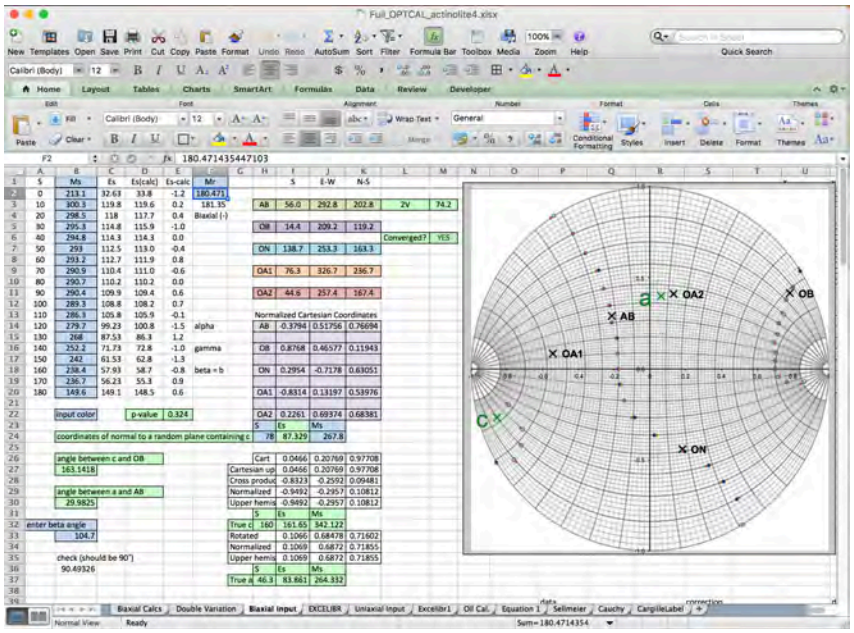


Figure 5.14 Modified Biaxial Input tab for plotting crystal axes of monoclinic minerals.

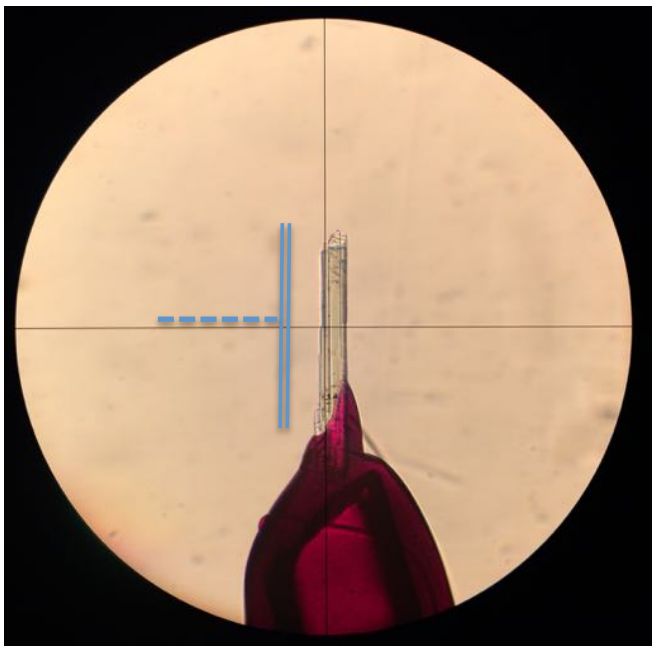


Figure 5.15 Illustration of the pole (dashed line) to the plane that the long axis of the crystal lies in.

Transform the microscope coordinates to Cartesian and take the cross product of the two vectors to arrive at the true orientation of c. Once two crystallographic axes are known, the third can be located by using a rotation matrix (if the angle between axes is known). With amphiboles, for example, the beta angle is always ~104.7˚, however, the rotation matrix must be applied in the correct direction using known crystal faces or cleavage planes as references.

6 Immersion Method

6.1 Modeling Dispersion of Refractive Index Liquids

6.1.1 Cargille Liquid Label

With relatively new Cargille liquids, the refractive index of a given liquid, as well as its variation with temperature and wavelength, is extremely close to what is listed on a liquid's vial. As a result, refractive index and its variation with wavelength can be modeled using a dispersion equation.

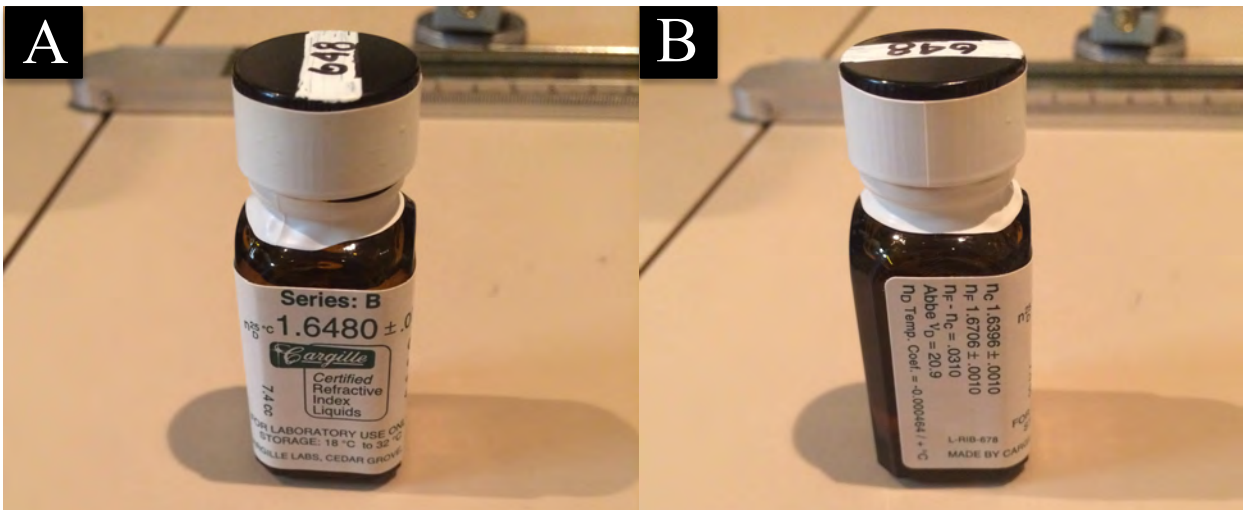


Figure 6.1 (A-B) (A) Front label of a *Cargille* liquid with a value of refractive index at 589.3 nm at 25˚ C. (B) Side view of a Cargille liquid with dn/dt, and refractive index values at 656.3 nm (n_C) and 486.1 nm (n_F) at 25˚ C.

The tab named "CargilleLabel" is the tab for modeling a Cargille liquid's dispersion. Simply enter the refractive index for each wavelength on the label (**Figure 6.1 A-B**) and the spreadsheet will list the liquid's Cauchy constants (**Figure 6.2**). These constants can be used later for single or double variation.

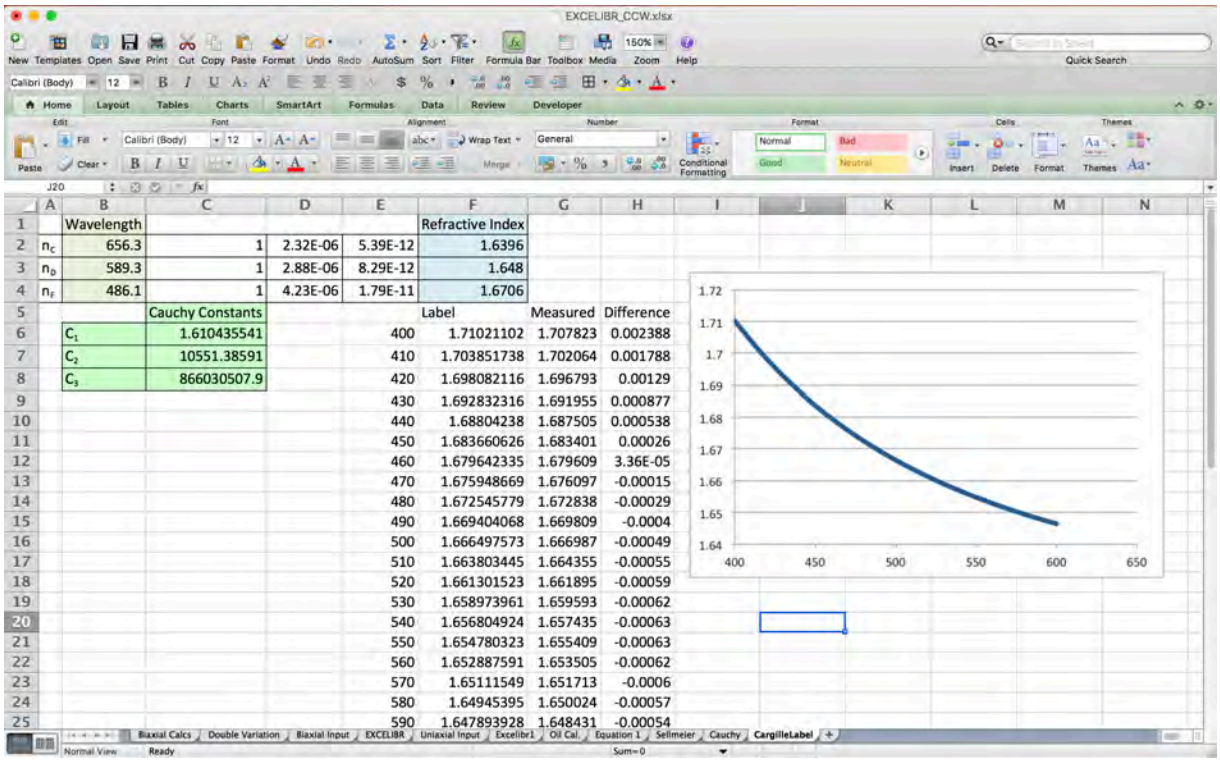


Figure 6.2 Model of dispersion of a refractive index liquid using the values given on a *Cargille* label.

6.1.2 Direct Measurement

Direct measurement is by far the best way of knowing the exact refractive index of a liquid, which is the preferred method for research. With a good refractometer, a liquid's refractive index can be determined with 4th decimal accuracy. Additionally, some refractometers can measure refractive index at multiple wavelengths such as the *Bellingham and Stanley Abbe 60 Refractometer*, so dispersion can be modeled as well (**Figure 4.3**). The "Oil Cal." tab is meant for the aforementioned refractometer but the principle is the same with any high precision refractometer.

Begin by figuring out what wavelength your refractometer measures for. Most measure a single, standard wavelength (589.3 nm). In **Figure 6.3**, a sodium gas discharge bulb is used for measuring at 589.3 nm.

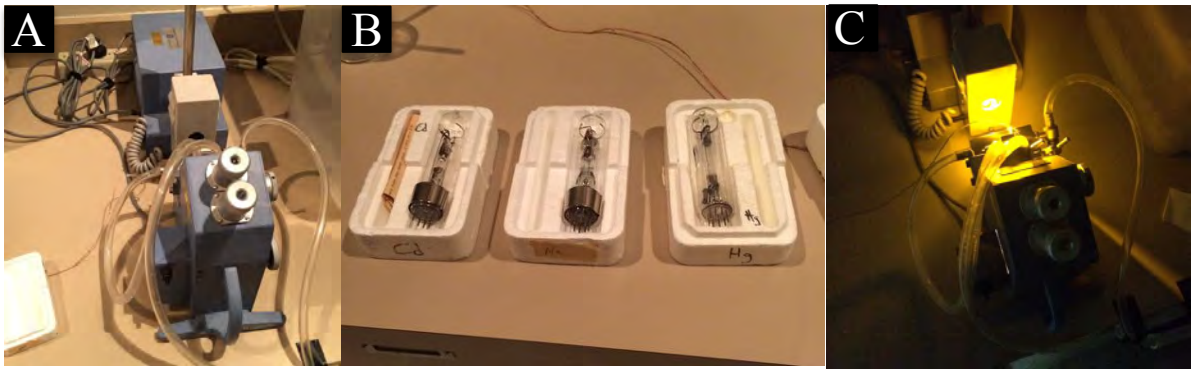


Figure 6.3 (A-C) (A) Refractometer, (B) gas discharge bulbs, (C) and light source of the refractometer.

A refractometer that measures at multiple wavelengths measures critical angle, since refractive index varies with wavelength. Not only does the refractive index of what you are measuring vary with wavelength, but also the prism in the refractometer itself, so this must be accounted for. Below is an example of a readout for refractive index at 589.3 nm (**Figure 6.4**). This measurement is also made at a known temperature by placing a thermocouple probe where the liquid is measured.

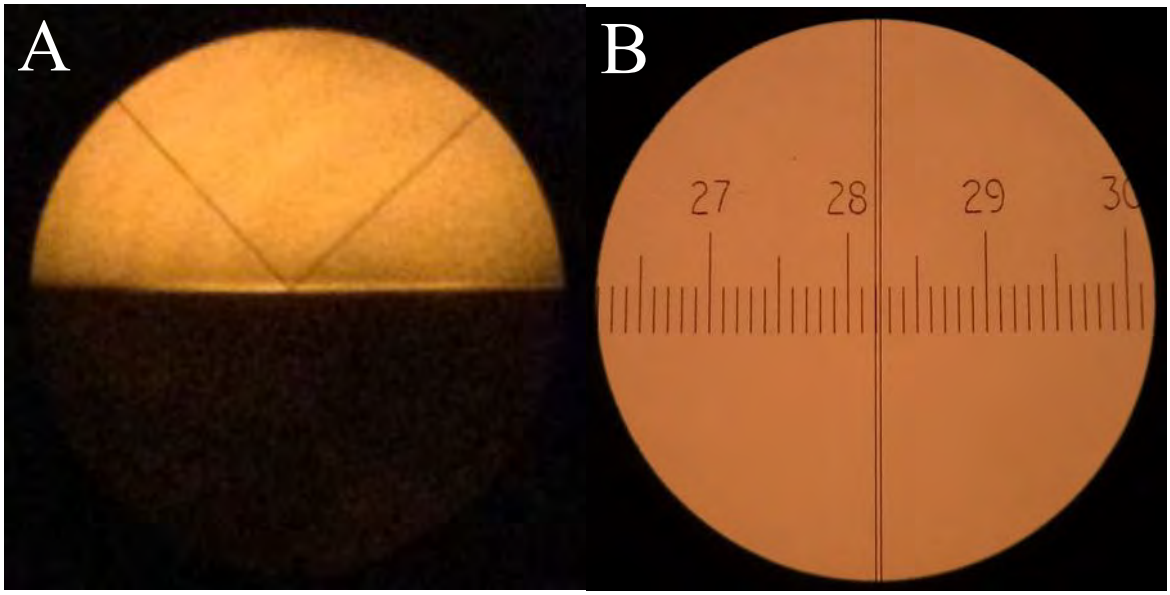


Figure 6.4 (A-B) (A) 589.3 nm readout and coinciding (B) critical angle (28.2137˚).

The critical angle readout in **Figure 6.4** is 28.2137, which corresponds with a refractive index of 1.650028148 at 21.6˚ C. The value to enter in the dispersion matrix needs to be at a standard temperature though (25˚ C), so the correct entry is 1.648450548. **Figure 6.5** has this entry, as well as the entries for other wavelengths filled out. Once all the desired wavelengths are entered, the Cauchy constants are given (**Figure 6.5**). The tabs named "Equation 1" and "Sellmeier" are other mathematical models of dispersion that reference the "Oil Cal." tab.

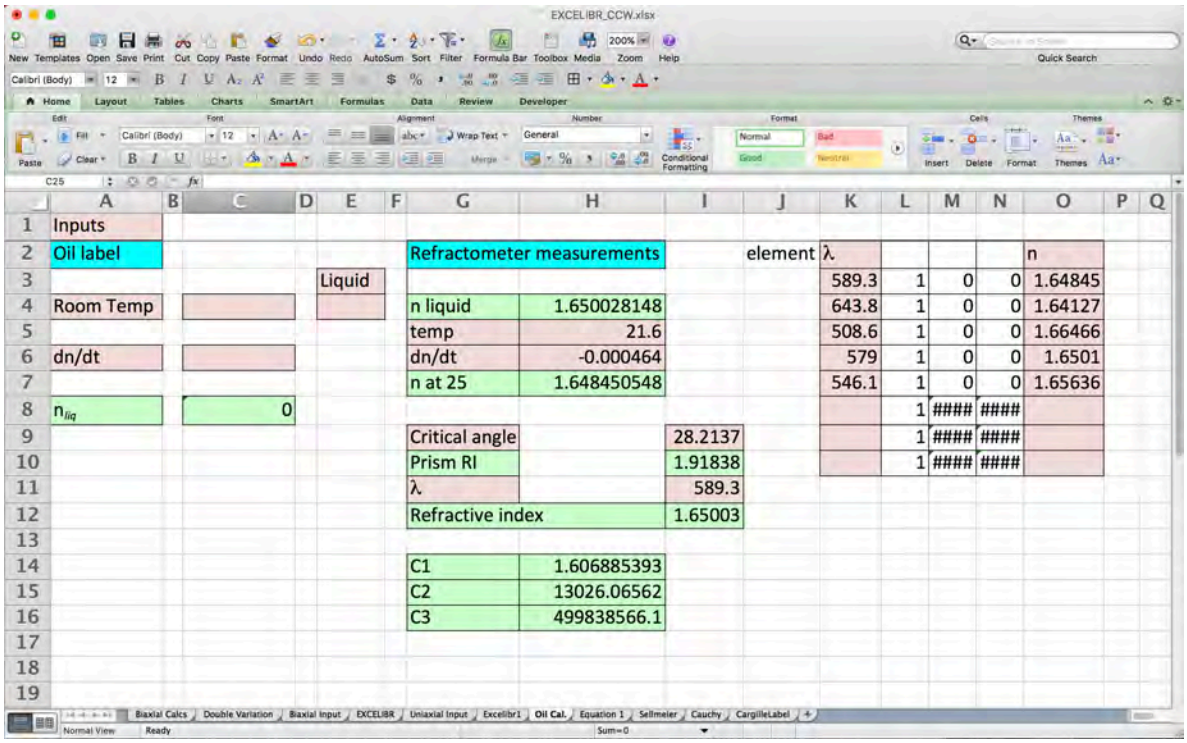


Figure 6.5 Oil Cal tab used for modeling dispersion of refractive index liquids by measurement.

Note that the mathematical models for dispersion depend on the amount of entries, so modify the equation to use the entries for your use (**Figure 6.6**).

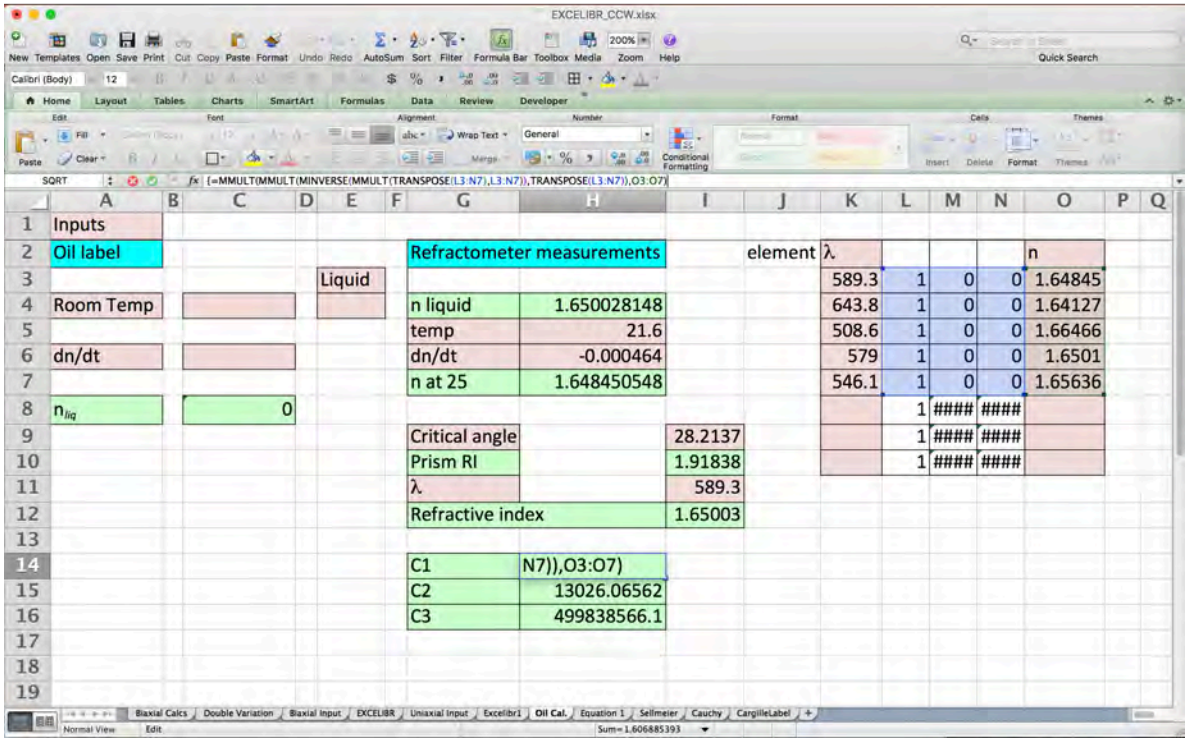


Figure 6.6 Selection of the data entries to calculate Cauchy constants. Enter by pressing command (or ctrl) + shift + enter after selecting the data.

6.2 Single Variation

6.2.1 Measuring alpha, beta, and gamma

The single variation method of refractive index measurement generally refers to measuring refractive index at a given wavelength by heating a refractive index liquid to match a crystal. This is accomplished by using the "Double Variation" tab and entering the wavelength you are measuring at under the lambda symbol (usually 589.3 nm interference plate). The next entries are the Cauchy constants of the liquid you are matching with (see section 6.1) and the dn/dt of the liquid. From here, the refractive index output will be a temperature corrected refractive index value (**Figure 6.7**).

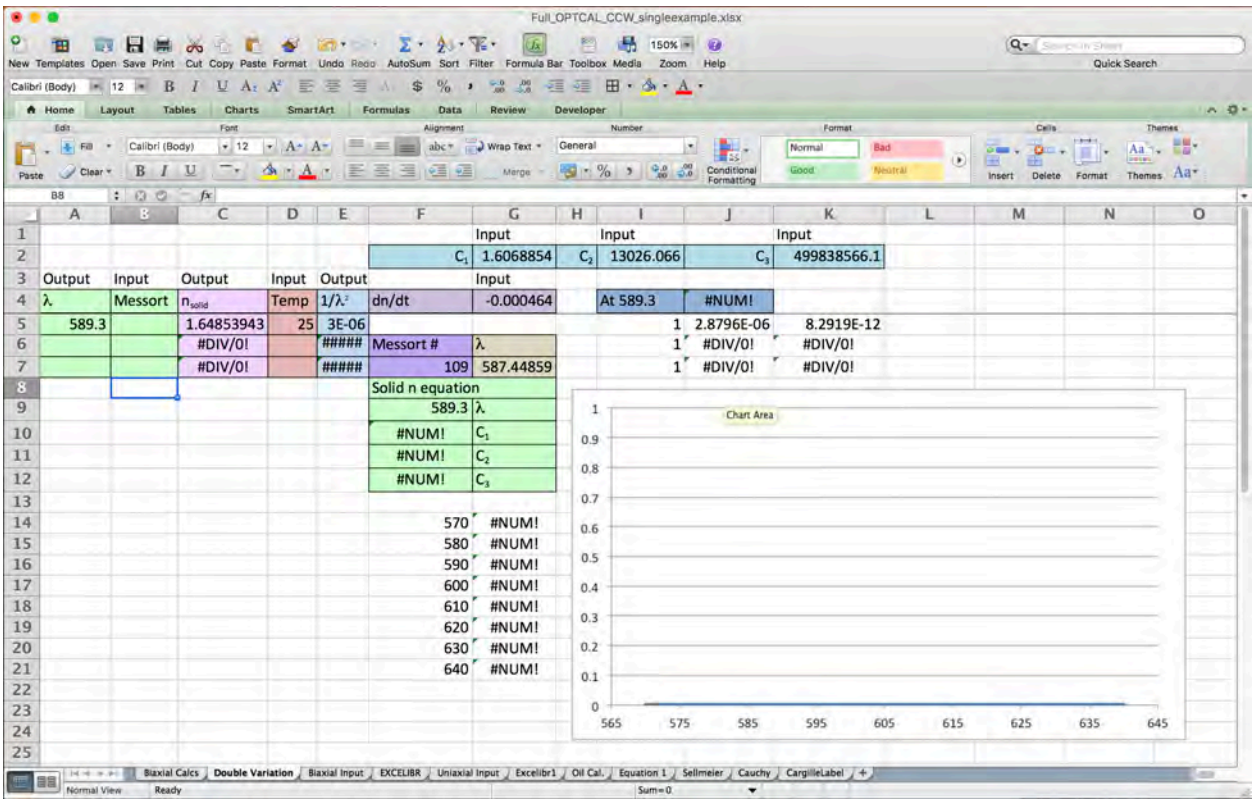


Figure 6.7 Double Variation tab for keeping track of refractive index (as a function of temperature) at a given wavelength. This tab uses Cauchy constants as a preset to interpolate dispersion curves of crystals.

6.3 Double Variation

The double variation method is a great way of interpolating refractive index across multiple wavelengths. The double variation method is used for determining an accurate refractive index match and can also be used to model dispersion of solids.

6.3.1 Measuring alpha, beta, and gamma

To begin, the double variation tab is designed for using a heating cell, and a graded interference filter, which has a gradient of wavelengths that it can filter. The graded interference filter is where the Messort # comes from. If you do not have one of these, you can use interference plates. The wavelength of the plates should be entered under the lambda symbol in place of the equation for the Messort #.

Since there are 3 variables in a standard Cauchy equation, you will need at least 3 inputs to model the dispersion curve of a solid (**Figure 6.8**). As with the single variation method, you must enter the dn/dt of the liquid you are using, in addition to its Cauchy constants (see section 6.1). The only other entry is a temperature for which the liquid matches the crystal and the ouput is given under n_{solid}. I recommend saving a separate spreadsheet for each orientation you measure (alpha, beta, gamma or epsilon and omega).

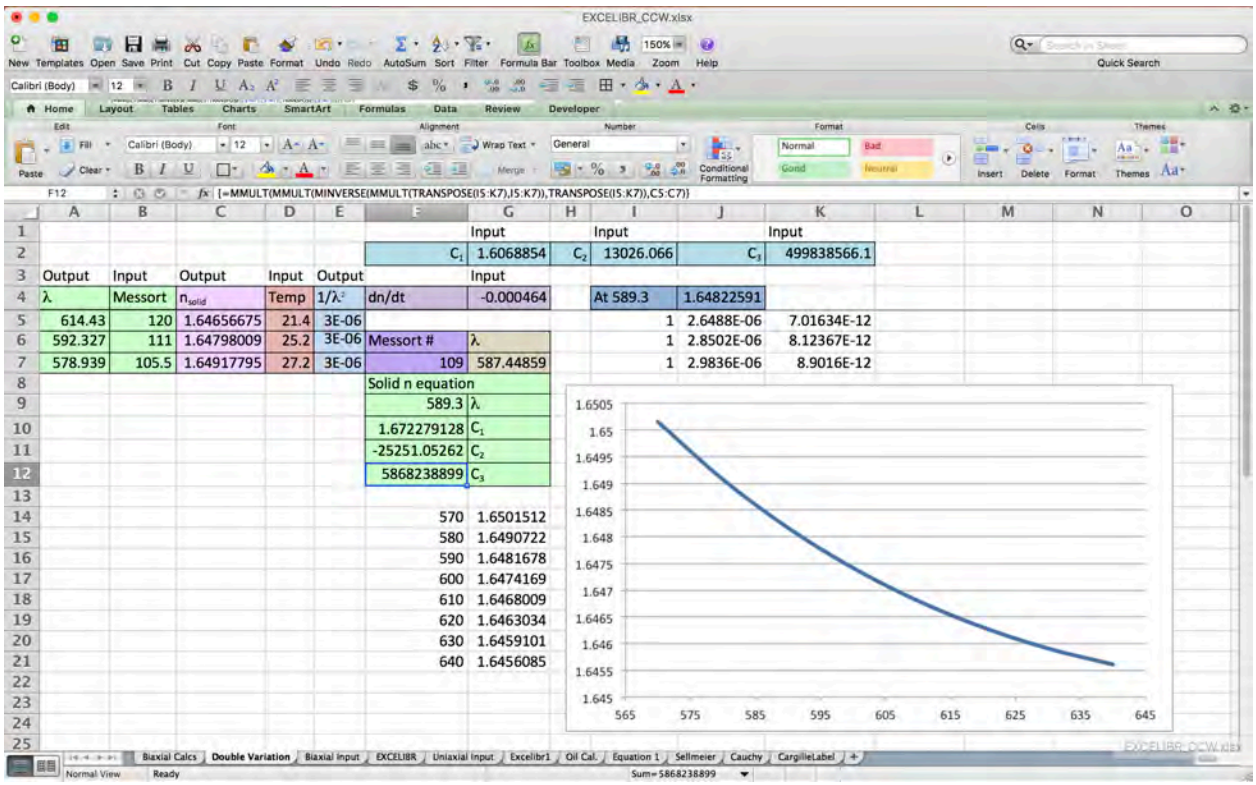
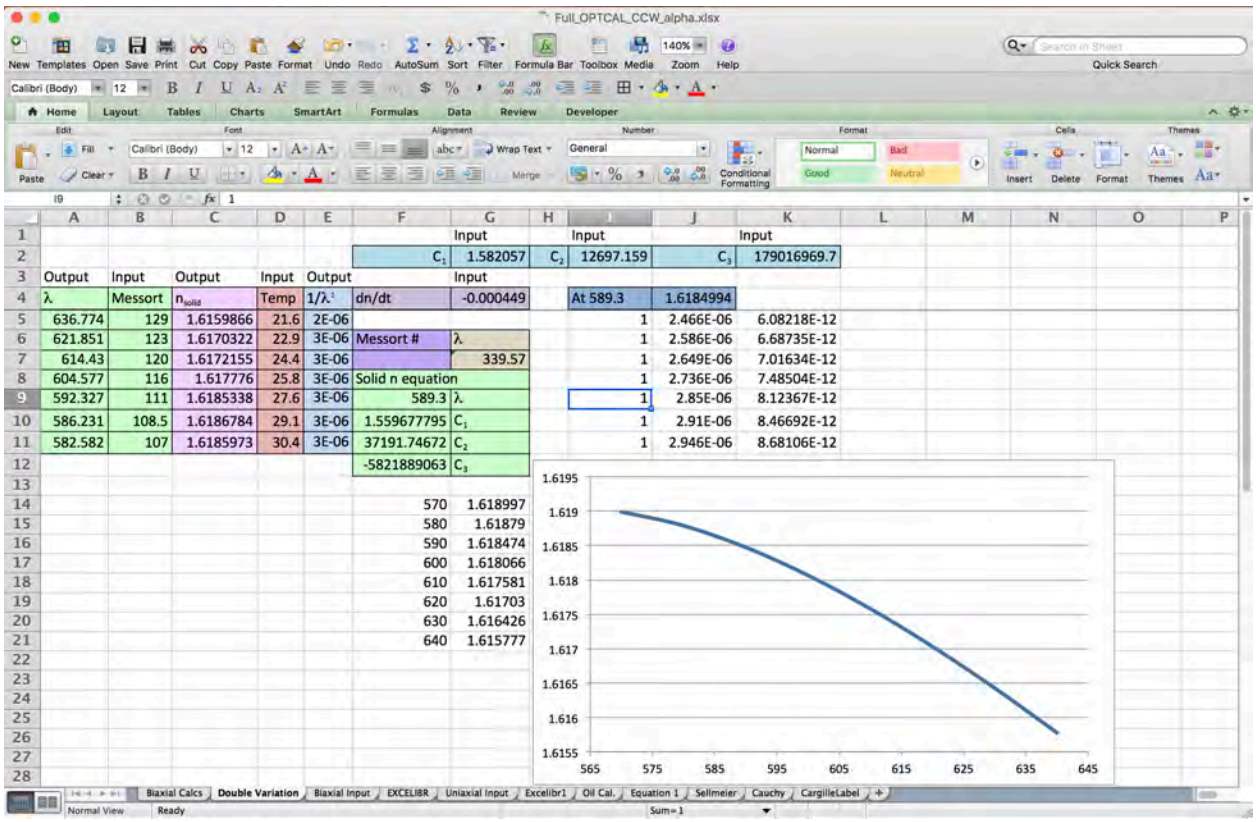


Figure 6.8 Filled in entries for plotting the dispersion curve of a principle refractive index.

For entering more than 3 entries modify the dispersion matrix by dragging down the equations to accommodate the number of data entries. Also drag down the refractive index and wavelength outputs on the left. Finally, select the Cauchy constant equations and modify them to select the full dispersion matrix and refractive index output and enter Command+Shift+Enter (**Figure 6.9**).



	A	B	C	D	E	F	G	H	I	J	K
1							Input		Input		Input
2						C_1	1.582057	C_2	12697.159	C_3	179016969.7
3	Output	Input	Output	Input	Output		Input				
4	λ	Messort	n_{solid}	Temp	$1/\lambda^2$	dn/dt	-0.000449		At 589.3	1.6184994	
5	636.774	129	1.6159866	21.6	2E-06				1	2.466E-06	6.08218E-12
6	621.851	123	1.6170322	22.9	3E-06	Messort #	λ		1	2.586E-06	6.68735E-12
7	614.43	120	1.6172155	24.4	3E-06		339.57		1	2.649E-06	7.01634E-12
8	604.577	116	1.617776	25.8	3E-06	Solid n equation			1	2.736E-06	7.48504E-12
9	592.327	111	1.6185338	27.6	3E-06	589.3	λ		1	2.85E-06	8.12367E-12
10	586.231	108.5	1.6186784	29.1	3E-06	1.559677795	C_1		1	2.91E-06	8.46692E-12
11	582.582	107	1.6185973	30.4	3E-06	37191.74672	C_2		1	2.946E-06	8.68106E-12
12						-5821889063	C_3				
13											
14						570	1.618997				
15						580	1.61879				
16						590	1.618474				
17						600	1.618066				
18						610	1.617581				
19						620	1.61703				
20						630	1.616426				
21						640	1.615777				
22											
23											
24											
25											
26											
27											
28											

Figure 6.9 Additional entries made with a coinciding matrix dragged down formulas and selecting all entries to solve for Cauchy constants and a dispersion curve.

For each principle vibration direction, enter the refractive index along with 2V in the 'Biaxial Calcs' to interpolate and compare other principle refractive indices (**Figure 6.10**).

Calculate	Inputs	Calc Output	Obs-Calc		Input	Calc Output
$2V_x$	81.5	#DIV/0!	#DIV/0!	$2V_z$		#DIV/0!
α	1.6185	0	1.6185			
β		1.63535721	-1.63536			
γ	1.64822	#DIV/0!	#DIV/0!			
Birefringence						
β−α	1.6185					
γ−β	1.64822					
γ−α	0.02972					

Figure 6.10 Biaxial calcs tab to predict relatively which refractive index liquid to use after determining any 3 of the four values listed.

7 Evaluating Data

One critical step in measuring principle refractive indices and orienting minerals is making sure that the solution is correct. Several factors may be responsible for bad data (poor crystal quality, bad orientations, incorrect reference azimuth) but it's important to be able to discern a bad dataset from a good one. Most of the time, bad data can be avoided by following the steps outlined in this guide.

7.1 Sources of error

7.1.1 The crystal is mounted very near a principle vibration direction

When crystals are mounted very near a principle vibration direction they will not pass the 40˚ test, which is why it is important to perform this test prior to attempting to solve its orientation, but again, passing the 40˚ test is for optimal solution, but passing anywhere from the 30˚ to 40˚ test is typically sufficient. In this case, principle vibration directions may still be located, though the 2V angle output can be significantly off. If a crystal is mounted this way, it will stay extinct

as it is rotated in XPL when the spindle axis is oriented N-S or E-W. Generally, this is avoided by mounting the crystal randomly or by tweaking a crystal's orientation after it fails the 40˚ test. For example, for orthoamphiboles or orthopyroxenes, a crystallite will usually have a long axis 'c' which, because they are orthorhombic, will coincide with a vibration direction, and therefore, mounting an elongate crystallite as crooked as possible will result in an optimal orientation solution.

7.1.2 The reference azimuth is incorrect/ left blank

In this case, Microsoft Excel will treat a blank entry as 0˚ so be sure to enter the correct reference azimuth.

7.1.3 The crystal is twinned, exsolved, fibrous etc.

When dealing with a crystallite that has more than one crystal in two or more orientations, it will not go perfectly extinct in XPL and the orientation solution will not be accurate.

However, do not mistake this for extinction positions that are viewing down an optic axis or near one. The dispersive properties of a mineral effect how extinction (or lack thereof) appears viewing down optic axes, and though there is more error with this extinction entry, the overall crystal is fine.

7.1.4 One or more extinction entries is incorrect

This case is easy to deal with, particularly when the rest of the data looks good. To check if any data entries are 'bad' check the column Es-calc to see what the degree difference is between the predicted calculated extinction value, and the observed. If there is large error (greater than about 3˚) check that extinction position again. If the extinction position doesn't appear to be different, leaving one entry probably won't affect the solution significantly.

7.1.5 The 2V angle of the mineral is near 90˚ or near 0˚

In both cases, the solution to this problem is to again, make sure the crystal passes the 40˚ test, but in these cases, it is absolutely imperative to the solution. With crystals where the 2V is at or near 90˚ in particular, the AB and OB orientations become ambiguous and the optic sign may be inaccurately (or invalid anyways) determined because these are assumed to be correct. However, assigning gamma and alpha to these axes is still easily accomplished by the immersion method.

7.2 Identifying Problems

Convergence Parameter

Once you have entered your extinction data, the first indicator that the program found a solution is the convergence parameter. The convergence parameter indicates whether or not the program found a solution that does not change significantly in the final iterations. The convergence parameter is based on a solution not changing by more than 1×10^{-14} in the final iterations. The

convergence parameter does **not** indicate that the solution is correct, it just indicates that a solution has been determined.

Calculated Extinction Data

Perhaps the best indicator of a good dataset is the calculated extinction data. The calculated extinction data is based on the solution of the optic axes. If the solution for the optic axes is correct, it will result in the calculated extinction dataset for a given spindle angle. A calculated extinction data point is a rewritten version of the Joel equation that is in the form of a quadratic equation.

The closer an observed dataset corresponds with the calculated dataset the better. An observed versus calculated column is provided to the right of the data entries. Below the observed versus calculated extinction data is a p-value correlation for the overall dataset. The closer the p-value is to zero the better, and in general, p = 0-0.3 are typically excellent datasets, p = 0.3-0.5 are good datasets, p = 0.5-0.7 are moderately poor datasets, and p > 0.7 are likely throw-away datasets.

Refractive indices

After measuring the principle refractive indices, measured 2V and the principle refractive indices can be compared to the calculated ones in the 'Biaxial Calcs' tab. Though this is a way of checking data, 2V measured from extinction data is inevitably going to deviate from the calculated 2V from refractive index values but should not be significantly different. Both calculated and measured 2V should be provided for an optical study.

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