LABORATORY EXERCISES AND DEMONSTRATIONS WITH THE SPINDLE STAGE

Mickey E. Gunter
Department of Geology
University of Idaho
Moscow ID 83844-3022 U.S.A.
mgunter@uidaho.edu

Introduction and background

The goal of this lab session is to introduce you to the spindle stage and its possible uses in an undergraduate mineralogy lab. A spindle stage is a one-axis rotation device that mounts on a polarizing microscope and is used to aid in the measurement of optical properties of single crystals. At the undergraduate level, it can be used to identify minerals and to demonstrate the relationships among grain shape, retardation, and interference figures. A natural extension of these uses is undergraduate research on the optical properties of minerals. These notes and other references for the spindle stage are posted at the web site: www.uidaho.edu/~mgunter/opt_min/ss/ss.html.

This lab session would not be possible without the work of Professor F. Donald Bloss, and those fortunate ones who have worked with him over the past two decades to develop, refine, and integrate the methods presented here. Bloss and Light (1973) developed a student spindle stage, Bloss and Reiss (1973) developed a computer program to calculate a biaxial mineral's 2V and indicatrix orientation based upon extinction data, and Louisnathan et al. (1978) refined the double variation method for precise (+/-0.0001) refractive index determination of minerals. These works culminated in Bloss' MSA Presidential Address (Bloss, 1978) and a book devoted entirely to the spindle stage (Bloss, 1981). Several computer programs were also developed to aid in reducing data collected with the spindle stage, especially EXCALIBR (Bloss and Reiss, 1973; Bloss, 1979; Bloss, 1981). With the evolution of mainframes to microcomputers, EXCALIBR has been modified to work on both PC's and Mac's (Gunter et al., 1988). Other programs have been developed to aid in refractive index calculation based upon the double variation method (Su et al., 1987) and for routine optical mineralogy calculations (e.g., the relationship between 2V and the principal refractive indices for biaxial minerals) (Gunter and Schares, 1991). Most recently, Barthelmehs et al. (1992) rewrote EXCALIBR, making it much easier to use. All of these programs are available from the web site: www.uidaho.edu/~mgunter/programs/programs.html.

The spindle stage has helped research in optical mineralogy from A (i.e., andalusites, see Gunter and Bloss, 1982) to Z (i.e., zeolites, see Gunter and Ribbe, 1992), with many other optical secrets of minerals unraveled in the middle (e.g., corderites (Armbruster and Bloss, 1982) and feldspars (Su et al., 1986)). These research projects did not use the student spindle stages but the more advanced, and expensive, Supper spindle stage that uses an x-ray goniometer head to mount and hold the crystal. However, student model spindle stages can provide almost the accuracy and precision of the Supper spindle stage. Much more research could be done, especially at the undergraduate level, using the spindle stage. Every department has the necessary equipment (polarizing microscopes) for undergraduates to conduct this type of research. It is hoped this lab session will encourage faculty to use the spindle stage in teaching optical mineralogy, or at least to use it to demonstrate the relationships among grain shape, retardation, and interference figures.

Lab session

Listed below are the necessary steps to implement the spindle stage in an undergraduate lab. Details are provided on how to build a poster board spindle stage (PBSS). Thomas Armbruster, University of Bern, Switzerland, is credited with the idea and original design of the PBSS, which has been slightly modified here. Student spindle stages are commercially available - for example, the detent spindle stage of Bloss and Light (1973) (from McCrone Accessories and Components, 800-622-8122, price = $50) for those who do not wish to build them. The detent spindle stage is well made and worth the investment if funds allow. If you purchase it, skip step 1 below.

Regardless of the type of spindle stage used, an oil cell must be built in which to view the crystal and to determine its refractive index by the immersion method. To build an oil cell, mount a bent paper clip (or some other type of wire of the correct thickness) to a petrographic slide. Place a drop or two of immersion liquid in the u-shaped paper clip, and place a glass cover slip on top.

Glue a single crystal of a mineral onto the end of a needle, and insert the other end of the needle into the spindle stage. Next, attach the spindle stage and the needle combination to the stage of a polarizing light microscope. Carefully slide the oil cell into the docking port of the spindle stage until the crystal is immersed into the liquid. At this point, you are ready to make optical measurements.

For a complete description of all aspects of the spindle stage refer, to Bloss (1981). Also, Nesse (1991) and Stoiber and Morse (1994) provide brief descriptions of applied spindle stage use. For those rusty in optical mineralogy, refer to Gunter (1992) for a short review, or Bloss (1961), Nesse (1991), or Stoiber and Morse (1994) for thorough treatments.

Please note: As stated above, my intent is to introduce you to the spindle stage, give some idea of what it can do, some "hands-on" experience, and, mainly, provide the resources for you to continue to use the spindle stage in the future. Remember, there is an entire book devoted to the subject (Bloss, 1981), and Bloss taught a semester-long course on its use. And you **will** knock a crystal off the end of needle occasionally, but that's part of the fun!

Lab procedure

The following is a seven-part, step-by-step procedure. The first line in each step (marked with a "*****" and in bold) is the action to be taken. The following text in that step are pitfalls, words of wisdom, and hints.

1. Build a poster board spindle stage (PBSS)

<u>materials</u>: poster board, 20-gauge hypodermic needle (Fisher Scientific), petrographic slide, white glue, straight edge, compass, small protractor

Follow the instructions in Appendix A.

2. Build oil cells

materials: petrographic slide, cover slip, large paper clip, epoxy

Follow the instructions in Appendix B.

3. Mount crystals
materials: sewing needle (size #12), fingernail polish, acetone, transparent crystals (0.05 to 0.5 mm), binocular microscope, glass slide, patience
Mounting crystals is a skill that comes with time. At first, it seems very hard, but it gets much easier after you have mounted a few thousand !

Follow the instructions in Appendix C.

4. Align the PBSS and oil cell
materials: PBSS, oil cell, sewing needle

* **Place a sewing needle (point first) into the PBSS tubing.**

* **Move the oil cell (using the oil port farther back on the slide) into the docking section of the PBSS and make sure the needle does not hit the slide or the cover slip.** If it does hit, it will need to be adjusted up or down by removing the tubing and changing the height of the hole (in the tubing holder, Figure 1.4). I usually number the oil cells and PBSS's to know which "fit" well together.

* **Also, check that the needle does not go too far and hit the back of the oil mount (i.e., the epoxied paper clip), or off goes the crystal!**

* **If the alignment is a problem, then use the oil port mounted at the slide's edge.** This paper clip is a bit thicker than the other, and it will be harder to view an interference figure with this oil port.

5. Attach PBSS to microscope and view a sample
materials: PBSS, polarizing microscope, sewing needle, Scotch tape (about 20 mm wide), patience

* **Insert the needle without a sample into the PBSS until its end lines up with the pencil marks on the arms of the PBSS.** The needle should be sticking out about 13 mm (see Figure 1.4). The needle will appear a bit loose in the hypodermic needle. This is intentional. The loose fit will help keep you from knocking off the crystal. Later, a tighter fit will be important. This can be accomplished by slightly bending the sewing needle before inserting it, putting a bit of fingernail polish where the needle slides into the tubing, or carefully crimping the end of the tubing. The tubing ends can be exchanged (i.e., the handle taken out and reversed), so one end can be tight and the other loose fitting.

* **Obtain one piece of tape about 100 mm long and two pieces about 30 mm long.**

* **Place the long piece of tape across the PBSS between the tubing holder and the protractor.** This separation was designed for the tape to fit into.

* **Place the PBSS on the microscope stage.**

* **Using low power, unpolarized light, center the end of the needle in the middle of the cross-hairs.**

* **Press the tape onto the microscope stage.** Also, add the two shorter pieces of tape to the PBSS arms, being careful to keep them out of the way of the oil cell.

* **Insert the oil cell and make sure the needle does not hit the slide or the cover slip.**

* **Rotate the microscope stage and the needle around for awhile to make sure everything is near-centered.**

* **Remove the PBSS from the stage by first removing the oil cell and carefully lifting up the tape.** It is not necessary to remove the PBSS to change samples. You can use a small pair of needle nose pliers or tweezers to remove the needle and insert a new one - this is when you get good at it. For now, it is easier just to remove the entire PBSS to change samples.

Later, we will repeat the process with a sample. This was intended to give you some experience without worrying about knocking the crystal off the needle.

6. Sample exercises

A. determine the refractive index of a mineral - keep the same crystal and change the liquids
B. find the indicatrix orientation and 2V of a biaxial mineral from extinction data

Please note: I think this is the section I would concentrate on in a mineralogy lab. After you do the next section and demonstrate it to the students, the students should be motivated to build and use the spindle stage to confirm a mineral's identity by observing its optical properties. Also, you will develop the skills needed to assist your students by performing the next section.

7. View grain shape, retardation, and interference figures
materials: samples A & B provided. Sample A is a uniaxial mineral with its c-axis perpendicular to the spindle stage axis. Sample B is a biaxial mineral with its optic normal mounted parallel to the spindle axis.

* **Obtain the sample marked "A" from your glass vial.** You will use the PBSS to observe this grain.

* **Fill the oil cell with 1.510 or 1.512 or 1.514 liquid.** Use the oil port farther back if possible.

* **Repeat the process from "Section 5" above to get the grain, PBSS, and oil cell aligned and affixed on the microscope.**

* **Using low power and plane-polarized light, rotate the spindle axis and observe the shape of the crystal.**

*** What is the crystal's shape?**

*** Switch to cross-polarized light and rotate the spindle axis to obtain a minimum retardation.**

*** Check for minimum retardation while rotating the microscope stage.**

*** Repeat the adjustments on the spindle stage and microscope stage until you find minimum retardation** (i.e., the grain shows 1st order gray or lower retardation as the scope stage is rotated).

*** Check the refractive index of the grain against that of the oil. Is it higher or lower?**

*** What index are you measuring if the crystal is uniaxial? Biaxial?**

*** Rotate the spindle axis 90 degrees and watch what happens to the retardation.**
*** Obtain an interference figure for the sample.** This may be tricky. Sometimes the highest power lens (i.e., the lens with the largest NA) may not be able to get close enough to mineral without hitting the cover slip. These high NA lenses are usually spring loaded, so nothing bad will happen. Two hints are: 1) use the next highest power lens. It will image less of a cone of rays and thus show a smaller portion of the interference figure, but in many cases, especially if the figure is near-centered, one can determine the type of interference figure, and 2) lower the stage so the end of the high power lens is 10-20 mm from the oil cell. Switch to a conoscopic illumination with the high power lens; raise the stage slowly and watch the interference figure form and fill the field. Because of a possible collision between the lens and oil cell's cover slip, I prefer to glue the cover slip down. It will not come off the oil cell and make a mess on the microscope.

*** What is the optic sign?**

*** Rotate the spindle axis 90 degrees and watch what happens to the interference figure.**

*** Replace sample "A" with sample "B"**.

*** Obtain an interference figure.** Use the same cautions as above.

*** What is the approximate 2V and the optic sign?**

Please note: I would not recommend having students do this exercise. Instead, I recommend this as a demonstration for you to do. The main problem with having students do this is obtaining and mounting crystals in a preferred orientation, and the inevitable crisis of knocking the crystal off. It takes 5 to 6 minutes to mount and check each sample, so I think this works best as a demonstration. My students seem to enjoy this, and it convinces them that the same mineral will look much different depending upon its orientation. These oriented crystals can also replace the very expensive oriented thin sections, which are also hard to obtain.

For uniaxial minerals, one needs crystals with perfect (001) or (hk0) cleavage. I have used eudialyte and scapolite - this is a good choice for sample "A". (If anyone knows of any other minerals please tell me, especially if they are common.) Given these morphological conditions, the spindle axis can be made perpendicular to c, the optic axis. Another method is to use crystals with no cleavage (e.g., quartz) and view crushed quartz crystals with a binocular microscope set up with cross-polarized light. The big quartz crystals that exhibit low retardation more nearly lie on a circular section. They can then be mounted with their optic axis perpendicular to the needle, and, in turn, the spindle axis.

For biaxial minerals, one needs a cleavage direction that is perpendicular to the optic normal. Gypsum is almost perfect. It has perfect (010) cleavage with b=Y (i.e., the optic normal is perpendicular to the nice flat (010) plane). The problem with gypsum is that you need to mount several crystals (at least I have had to) to find one with minimal "deformation" to show good interference figures. The feldspars, especially K feldspars, are also good candidates. They all have perfect (010) cleavage. For high sanidine, b=Y, so it should be perfect, but I have never tried it. Low sanidine has Y perpendicular to another prominent cleavage, (001). These low sanidines can be mounted, with the aid of cross-polarized light, with the needle perpendicular (001) while they are lying on (010) - this is a good choice for sample "B". Both orthoclase and microcline share this same optical orientation and should work as well as low sanidine, but I have not tried them yet. If anyone knows of biaxial minerals that fit this condition, please let me know.

In case we cannot get the interference figures to work, you can view them on my web site: www.uidaho.edu/~mgunter/opt_min/ss/ss.html. I placed short quicktime movies on the web site demonstrating this exercise. There is a uniaxial mineral, eudialyte, mounted with its c axis perpendicular to the needle; thus, one can rotate from a centered optic axis figure to a centered flash figure. There is also a biaxial mineral, gypsum, mounted with its optic normal parallel to the needle so one can rotate from a centered acute bisectrix, to a centered optic axis, to a centered obtuse bisectrix. There are three movies for each mineral. One movie is made in unpolarized light to show the grain shape as it is rotated. This is good for viewing cleavage, grain thickness, pleochroism, etc. The second movie is made in orthoscopic illumination with polarized light. In this setup, changes in retardation can be observed; anytime a circular section is parallel to the microscope stage (synonymous with an optic axis perpendicular to the stage), retardation is near zero. Retardation is increased to a maximum when, for the uniaxial mineral, the single optic axis is in the microscope stage. For the biaxial case, maximum retardation occurs when the obtuse bisectrix is perpendicular to the stage. The third movie shows how the interference figures change as each sample is rotated about the spindle. It can be instructive to place all three movies for one crystal on the screen at the same time and "rotate" each image the same amount to show how they correlate.

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Appendix A: Building a poster board spindle stage

materials: poster board, 20-gauge hypodermic needle (Fisher Scientific, these come in 6 and 12 inch lengths, #14-82516E and 14-825-15AB), petrographic slide, white glue, straight edge, compass, small protractor

* **Cut two 50 x 50 mm squares and one 10 x 50 mm rectangle from poster board as shown below.** Part A will be the PBSS base, part B will be the protractor scale, and part C will help hold the tubing in place.

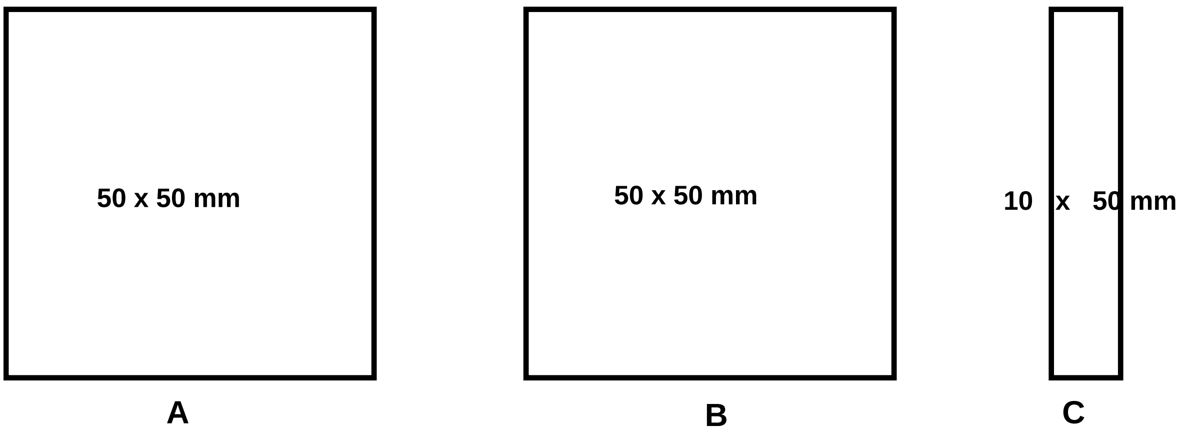

Figure 1.1: Starting material sizes for base (A), protractor (B), and tubing holder (C).

* **Mark all three pieces as below (Figure 1.2), and cut pieces B and C as shown to produce the round protractor as shown in the Figure 1.3.** Part B should first be cut into a circle and then cut in half along the horizontal line.

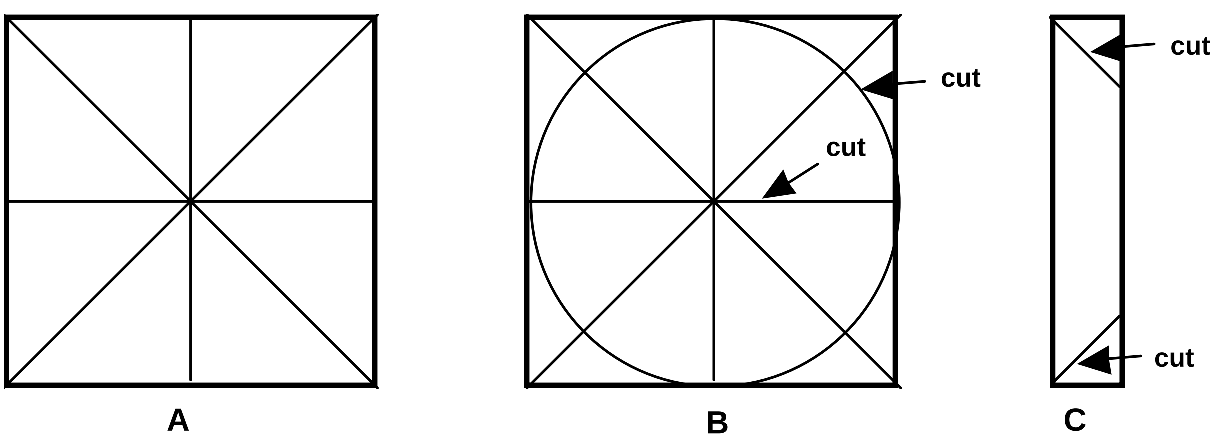

Figure 1.2: Marked-up base (A), protractor (B), and tubing holder (C). B should first be cut into a circle and then cut horizontally. C should just have the edges trimmed.

* **Overlay a petrographic slide onto the base as shown in A below (Figure 1.3).** Use the slide to mark the base and then cut out the marked area. This is the dock for the oil cell to fit into.

* **After making the cuts on B above (Figure 1.2), write numbers on it like B below.** This is the template (i.e., protractor) to measure the S angles on the spindle stage.

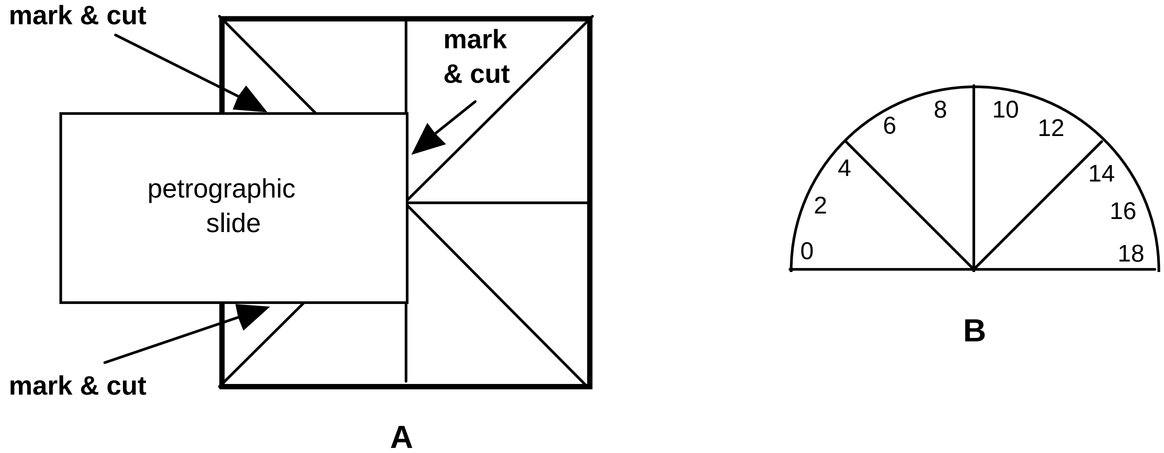

Figure 1.3 : The base (A) with slide overlain for marking the oil cell dock. The protractor (B) cut in a half-circle and numbered.

* **Glue the protractor and tubing holder onto the base as shown below (Figure 1.4).**

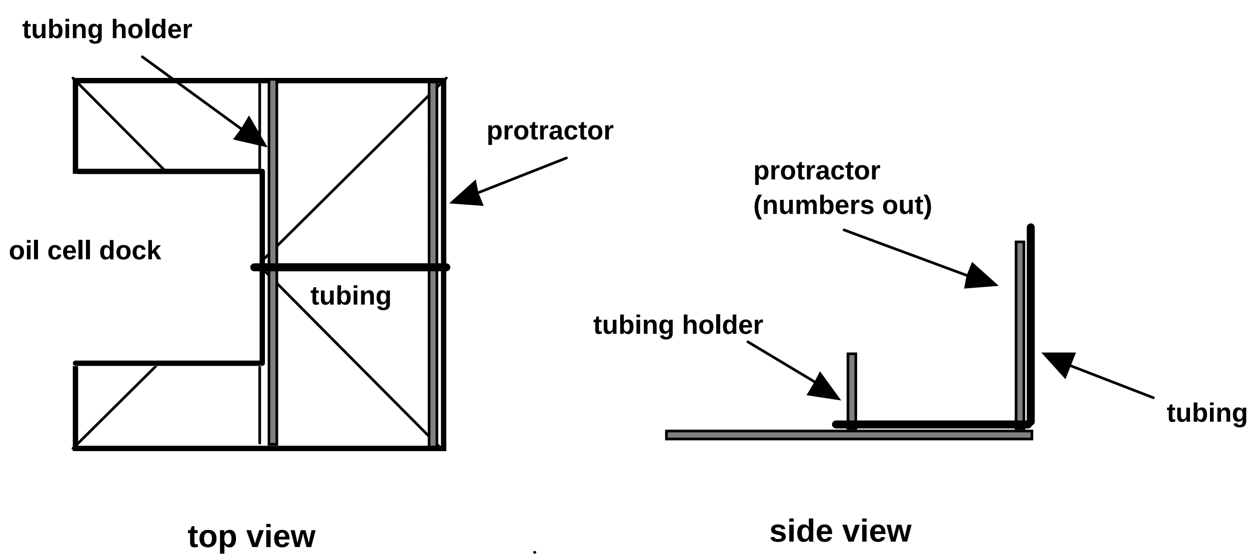

Figure 1.4: Finished PBSS. Left is the top view showing all the assembled parts; right is a side view.

* **Add the hollow tubing.** Cut a piece of 20-gauge hypodermic needle 55 mm long and make a 90 degree bend in the middle. (Hypodermic needles can be cut with a triangular file which

does not collapse the needle.) The hole for the tubing in the protractor's and tubing holder's centers can be made with a needle of similar diameter. The two holes should cause the tubing to be parallel to the base so a needle will project from it and be parallel to the oil cell slide. Adjustments may need to be made as described in section 4.

Appendix B: Building oil cells

<u>materials</u>: petrographic slide, cover slip, large paper clip, epoxy

* **Bend one side of a large paper clip into a small "U" about 5 mm long and epoxy it to the center of a petrographic slide about 13 mm from the end in the slide's center (Figure 2.1 below).** The "U" can be flattened a bit by placing it on a hard surface and hitting it with a hammer. The thicker the "U," the easier the alignment of the oil cell - needle - spindle stage combination. However, the thinner the "U," the better chance one has to see interference figures. These "U"s are the oil ports on the oil cell. We have also used staples and wire to make oil ports.

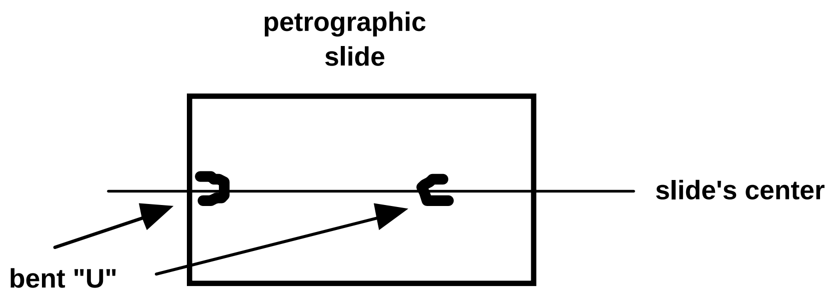

Figure 2.1: A petrographic slide with two bent "U"s epoxied on the slide's center.

* **Make a second 5 mm "U" and epoxy it on the end of the cell (Figure 2.1).** Less care needs to be taken for alignment with this thicker "U" at the slide's edge.

* **Cover slips will be placed on top of the "U" to hold in the immersion liquid.** They can be epoxied onto the "U," but surface tension will hold them on. If they are epoxied, they stay on better, but it is harder to clean out the immersion liquid when you need to change it. When the "U" is very thin and the cover slip is epoxied on,. air bubbles can form in the oil port. Bubbles can be cleared by holding the oil cell vertically and allowing the bubble to rise. If this does not work, stick a small needle into the bubble while holding the cell vertically. Oil can be removed from the oil port by sticking a small piece of rolled tissue paper between the cover slip and slide.

Appendix C: Mount crystals

materials: sewing needle (size #12), fingernail polish, acetone, transparent crystals (0.05 to 0.5 mm), binocular microscope, glass slide, patience

* **Obtain several crushed mineral grains of interest (0.05 to 0.5 mm).** They can be sieved if you want to remove the fine and the course fractions.

* **Place them on a glass side under a low power binocular microscope.**

* **Locate a good single crystal with the microscope.** Good means not twinned, homogenous, etc. You may not be able to tell if you like the crystal until you see it with the polarizing light microscope.

* **Get a needle and dip its tip into a drop of fingernail polish.** Super glue, Duco cement, or many other glues can be used; fingernail polish has the advantage of being slow to set, allowing for repositioning of the crystal, and the crystal can be removed with acetone.

* **Bring the needle next to the crystal and gently touch the crystal with the needle end.** The crystal should stick. If not, add more fingernail polish and try again.

* **Observe the crystal on the end of the needle.** It should be near-centered at the needle's end. You might want to move it around a little to get it more centered or in a particular orientation. The crystal can be moved by gently pushing it with another small needle.

* **Reinforce the fingernail polish around the crystal/needle contact.** This can be done by dipping another needle in fingernail polish and working it carefully around the contact; avoid covering the entire crystal with fingernail polish (mineralogists do not care to measure the refractive index of fingernail polish).