McBurney Geoarchaeology Laboratory, Division of Archaeology, University of Cambridge

A step-by-step guide to the making of soil thin sections

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Introduction

Large thin sections of soils and sediments and archaeological materials are made in the basement lab B5 of the West Building (or WB B5) of the Division of Archaeology, Downing Site, University of Cambridge, under the supervision of Tonko Rajkovaca (Geoarchaeology Chief Research Technician). Risk assessments, protocols, emergency procedures, and procedures for the importing, storing and handling of soils from non-EU countries are posted on the door of the WB B5 lab, and will be given to each person who is to undertake thin section production by the Chief Research Technician responsible prior to any lab work taking place. No lab work will be allowed out of normal business hours, nor unaccompanied by the Chief Research Technician.

Samples are collected from the field, and brought to the laboratory for drying by air, or acetone replacement (if waterlogged samples). The samples are then impregnated with crystic polyester resin, and stored to cold-cure in a ventilated cabinet for up to one month. When the resin has set, the samples are moved to an oven to be hard-cured with low heat. After the blocks have cooled, they are then sawed and cut to a desired size for the next stage of thin-section production using the Brot multi-plate grinder. The following illustrated, step-by-step guide sets out how to make a thin section of a soil/sediment block sample.

The WB B5 lab is equipped with one large ventilated cabinet, one large standing fume-cupboard, one fan-assisted standing oven, one vacuum chamber/oven, one Perspex-surround sawing unit, two multi-plate Brot grinding machines and Buehler bench-top thin-section equipment.

All products used in the production of thin-sections are solvent-based and flammable. It is lab policy that all chemical and resin preparation must be confined within a fume-cupboard. Under the guidance of the University Fire Officer, there are spillage kits, a fire-bin for flammable lab waste, one fire blanket, and the appropriate fire extinguishers in the laboratory. A First Aid Kit and Eyewash supplies are available within WB B5.

Collection and Drying of Samples

Protective Measures: All Persons working with soil, sediment, or archaeological materials should wear a lab coat in the laboratory, and vinyl or nitrile gloves if necessary.

1. Soil, sediment, and/or archaeological samples are taken from the field and brought into the lab.

Figure 1: Samples are collected from the field as intact blocks, labelled with an indication of direction and site context details, and wrapped in cling film and parcel tape to seal and transport

2. Samples are set in open trays or plastic boxes with appropriate field markings, and cut open with scissors or a razor blade to reveal enough surface to allow air drying. Special care should be taken not to disturb the original composition and structure.

3. Site designations and section orientation/profile are checked on the blocks, and written on the outside of the containers. Arrows can be added as an indication of the top of the profile, and where the block should be cut for thin-sectioning.

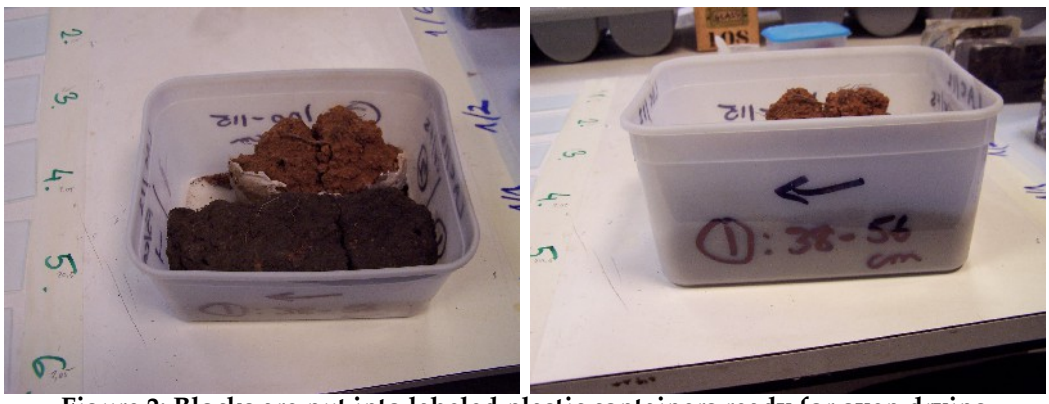


Figure 2: Blocks are put into labeled plastic containers ready for oven drying

4. The samples are then placed on the shelves in the lab for a period of up to a month to remove moisture before impregnation. If samples arrive from the

field wet, water will have to be removed by acetone replacement. See separate protocol.

Figure 3: Samples are placed to dry in labelled trays on the shelves at the back of the lab

5. After 4-6 weeks time, the samples are taken from the shelves and placed in the oven for a final drying period, at about 35 degrees C.

Figure 4: Placing samples in the oven for final drying

6. The next step is impregnation.

Impregnation

Protective Measures: It is mandatory for all persons impregnating with resin to wear a lab coat and nitrile gloves, and work within the fume-cupboard. Safety glasses, and face-masks must be worn as additional personal protection equipment when transferring samples to the vacuum chamber, and the ventilated curing storage cupboard.

Before you start the impregnation process, **make sure that you record the sample contextual details, drying times and impregnation recipe in the blue impregnation A4 folder.**

1. Impregnation mixes may vary dependant on the soil, sediment, and/or archaeological material type and structure. The standard mix for impregnation is 1800ml of resin, poured into a graduated plastic decanter. The colour of the resin is a clear light blue, and special attention should be given to the expiry date. All resin must be used within the shelf-life of the product, or the impregnation and cure will be poor.

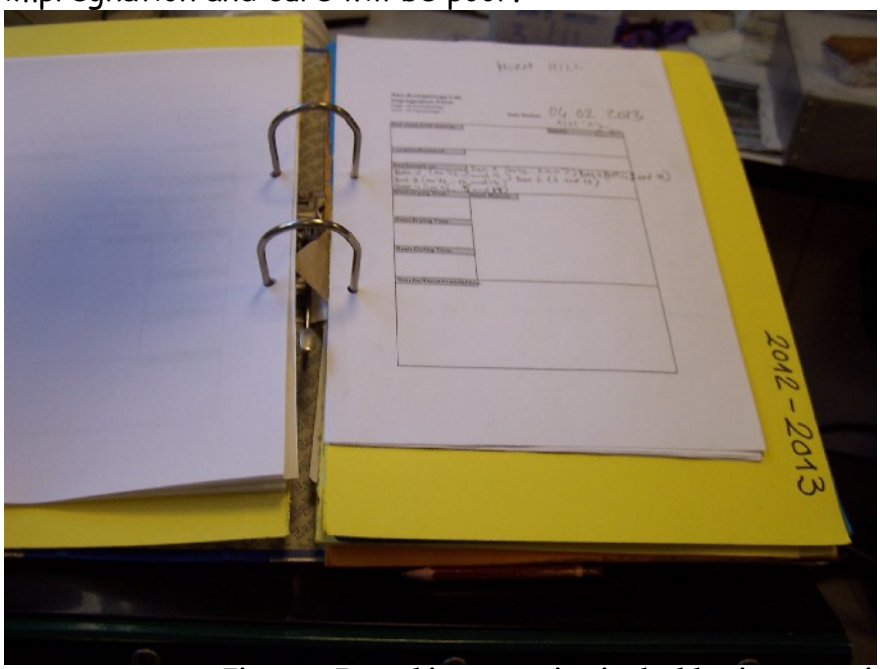
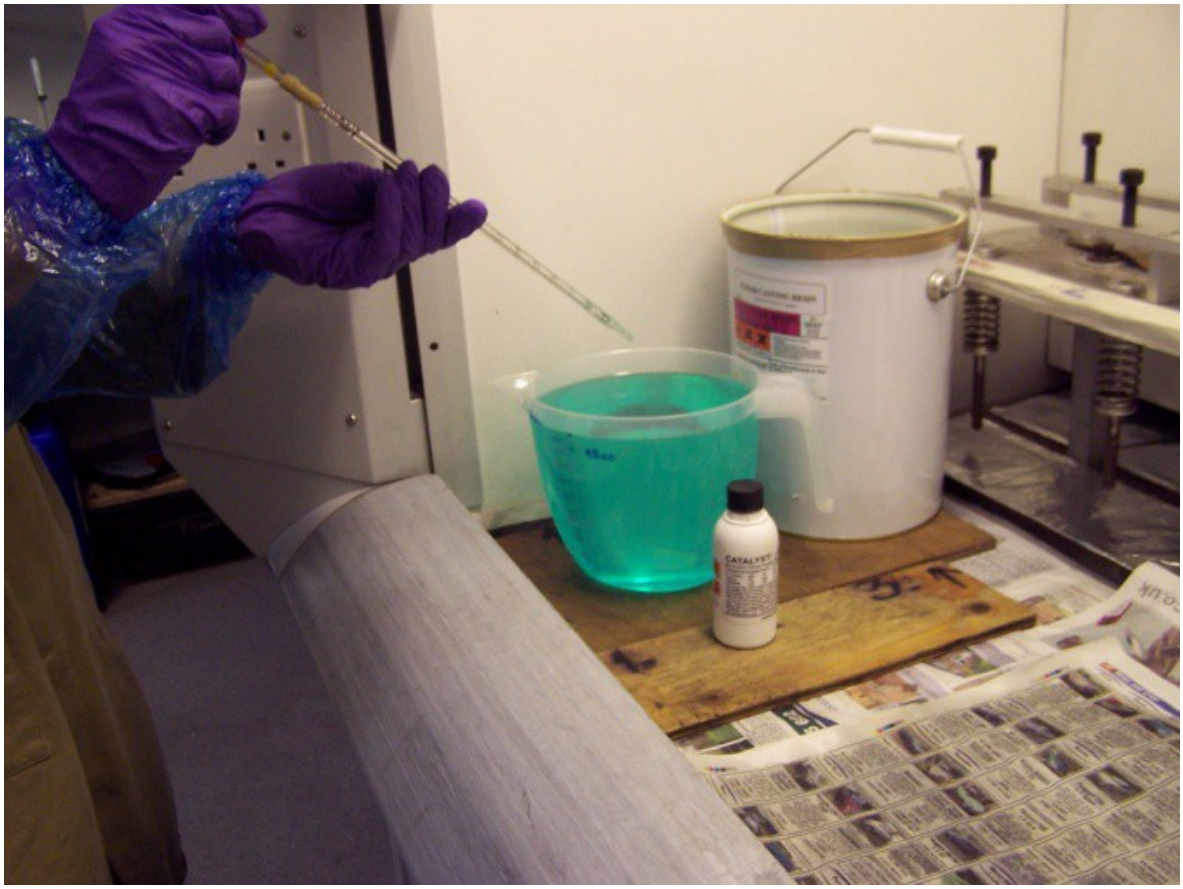


Figure 5: Record impregnation in the blue impregnation folder

Figure 6: Materials needed

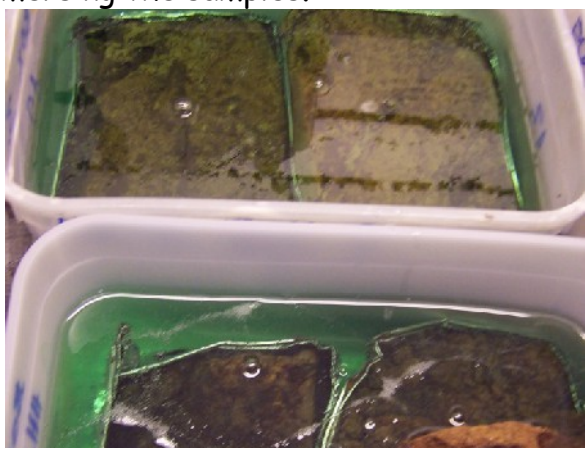
2. Add 200ml of acetone to the resin to increase the viscosity. The amount of acetone needed is dependant on the density, structure, and composition of the material. The acetone must be slowly folded and stirred into the resin until it disappears and amalgamates with the resin.

3. Add 1.0ml of Methyl Ethyl Ketone (MEKP), the catalyst for hardening the resin, by pipette and stirred until thoroughly mixed with the resin-acetone mix. This should take 3 to 5 minutes, and an immediate colour change from blue to green should take place. Any gloves that become contaminated with MEKP must be discarded into a sealed plastic bag, and new gloves put on to protect against potential chemical burn.



4. The samples previously placed in the oven overnight (at *c.* 35C) for drying, are taken to the fume cupboard for impregnation while still warm. No more than eight plastic containers should be used in each impregnation session.

5. Check that all the air bubbles in the resin from mixing have settled out, and then slowly pour around and down the inside of the container to prevent disturbance, fully immersing the samples.



6. The samples are left to infiltrate with resin by capillary rise for up to an hour within the fume cupboard. The resin level should be monitored, and further topped up if it should drop below the original immersion mark. Place the date of impregnation on the outside of the container. This will act as an aide in monitoring the length of the curing time.

7. A facemask and goggles must be worn when transferring samples to the vacuum chamber. After capillary rise, the samples are placed within the vacuum chamber, and slowly brought to 12 to 28 mercury vacuum, or until bubbles can be seen to gently evacuate from the samples. The samples are left under pressure for an initial 24-hour period.

Figure 7: Increasing pressure in the vacuum chamber (l-r) close door, engage pressure by pulling lever down, turn on vacuum pump, wait for pressure to reach required, turn off pump

8. After 24 hours, a second, top-up, resin mixture is prepared. After releasing the vacuum, the samples are taken from the chamber and placed in the fume cupboard. They are topped-up, and completely re-immersed in the resin, and then placed back into the vacuum chamber for a further 24 hour period, using the same 12 to 28 mercury vacuum pressure as before.

9. After the final vacuum, the samples are taken from the chamber and placed in the ventilated curing storage cupboard. Curing takes place over at least a month to six weeks, or until the blocks are completely hardened. Periodic

checks should be made on the resin level in the containers, and topping-up carried out if needed.

10. The resin and MEKP used for impregnation must be stored in the ventilated curing storage cupboard after use. Beakers and associated impregnation equipment are wiped clean with acetone and tissue. The MEKP graduated cylinder must be rinsed with at least 30ml of acetone and all consequent liquid stored in the Toxic Waste Winchester. Any tissue or contaminated gloves used in the cleaning of MEKP must be disposed of separately, and placed in sealed plastic bags. All lab waste from the impregnation process is disposed of in the blue/black fire-bin. This is collected monthly for appropriate incineration.

Figure 8: Curing cabinet with samples in storage

Figure 9: Processing waste bin

11. After the samples have hardened, they are subjected to a final curing in the oven at 50 degree C for a 24-48 hour period.

12. The next step is sawing the blocks for thin-sectioning.

Figure 10: An impregnated block ready for sawing

Sawing of Samples

Protective Measures: All persons using the large saw must wear a lab coat, plastic apron, rubber gloves, sleeve protectors, and ear protectors. As additional protection eye goggles or glasses, and cotton masks are available. No researcher is allowed to use the large saw unless monitored by the Chief Research Technician or laboratory director.

1. The main switches for the saw and extractor fan are turned on, and the silt-box is topped up with fresh water. Extra care is taken to hose out the drainage tray, so that all wastewater from sawing will run away smoothly to the silt-box. Any adjustments to the water spray feeding the saw blade must be made before beginning to saw the samples. **No person should ever attempt to saw samples without the saw blade being fed with water.**

Figure 11: The saw

2. The hardened resin blocks are brought to the sawing sled, and the plastic container is cut to separate the samples. Slow, even pressure is used to push the sawing sled with the container to the blade. The saw should be allowed to pull the block through at an even pace; extra force will cause friction problems.

3. Before making an actual cut to the block, note which face of the block is wanted for thin-sectioning. Decide the best way to cut down the block to obtain the needed sample slice, and proceed to cut the block with a plan.

Figure 12: Sawing correctly

4. Final sample slices should be at least 4-5mm. thick. It is better to cut a thicker slice, than to cut a slice too thin to run on the thin-section machine. Ideally, two sample slices should be cut from every block for backup in the thin-sectioning process.

5. A small notch should be cut on the top of each sample slice to indicate which side of the slice is up in section.

6. The cut block, with samples slices are laid upright on newspaper, within the fume cupboard to dry. When completely dry, the sample slice needed for thin-sectioning is selected, and the face crossed and labeled with a permanent marker. The block is re-marked if needed, and put in a sealed bag with site designations.

Figure 13: Cut blocks with a notch to indicate top of sample

7. When sawing is finished, care should be taken to hose out residual soil and resin. Wash off and wipe down the sawing sled and Perspex guards. Turn off the main switches for the saw.

8. Clean off all protective clothing worn whilst sawing. If the rubber gloves and plastic sleeves used are in good condition, they can be rinsed with water and hung to dry for the next use. Cotton masks and plastic aprons should be thrown away, and renewed when sawing again. Ear guards, and eye goggles or glasses for sawing must be safely stored away for next use in the laboratory.

Thin-Section Grinding of Soil Samples

Protective Measures: All students, staff, and researchers undertaking the preparation and grinding of soil samples must wear a lab coat, and nitrile gloves. Safety glasses, plastic aprons, cotton masks, and respirators are available if needed.

1. Sample slices are selected in groups of three (there are three slots on the mounting head of the thin-section machine), usually of the same size and width.

2. The slices are fixed; face outwards, to prepared coarse ground glass slides with super-glue gel. A small drop of super-glue in each corner is sufficient to hold the sample through the temporary grinding/polishing of the face.

3. The machine is turned on at the wall and set to manual. On this setting, the mounting head should be rotated and wiped clean. Each slot should be liberally squirted with clean oil to remove dust and fine particles. The glued slices/slides are then placed on the mounting head, with the aid of oil squirted on the back of the glass slides. Each slice/slide should be pressed firmly into the slot, and pushed up and down to check that it is held fast to the plate by the capillary vacuum of the oil.

Figure 14: Turn on the extractor fan

Figure 15: Turn on the Brot

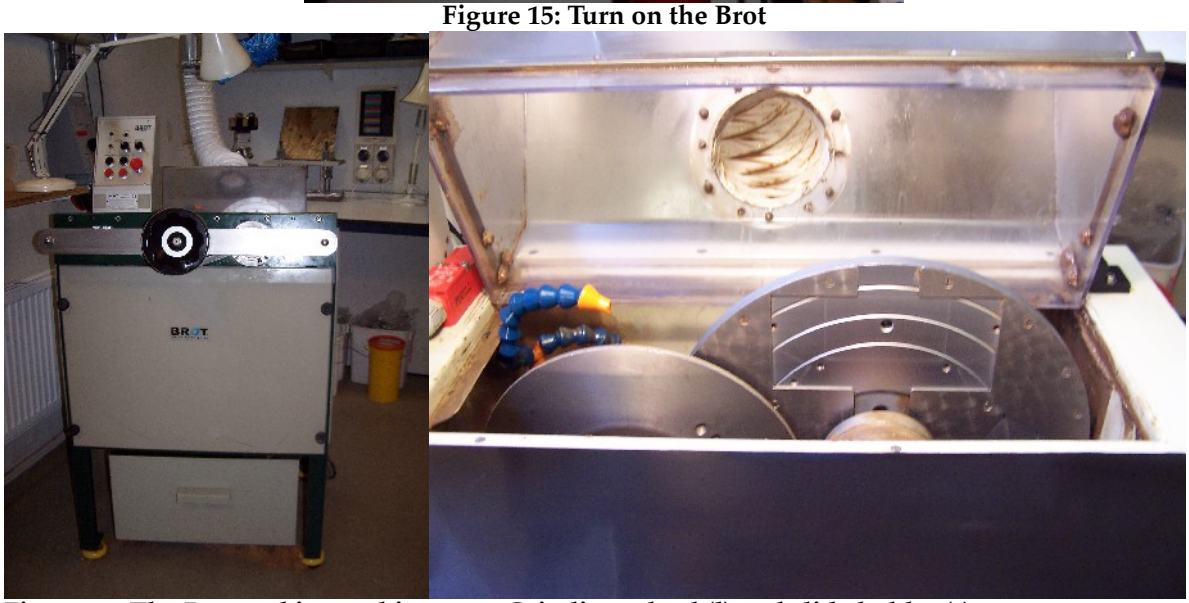


Figure 16: The Brot and its working area. Grinding wheel (l) and slide holder (r)

4. The machine is set to automatic, and goes through a coarse wheel grinding process. As soon as all outward markings have been ground away on the slice faces, the machine is stopped. The grinding wheel is changed to a finer grade, and the machine is set to proceed grinding until a fine finish/polish is accomplished on all the slices.

(a)

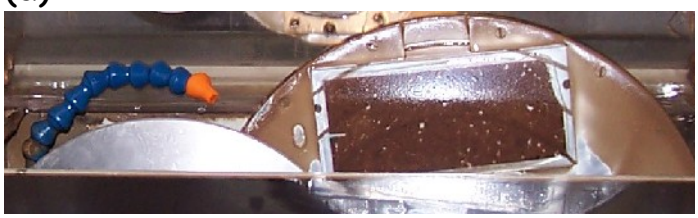


Figure 17: Sequence of beginning grinding after loading slides. 1: turn the machine from hand to auto. 2: turn on lubrication, and adjust nozzle accordingly (a). Wind grinding wheel two turns away from the slides (fig. 15), against the arrow direction. Set appropriate measurement to grind (fig. 15, 2) and set the gear (15.3). Close lid of machine. 3: start grinding wheel. 4: start sample holder, and flick gear on (15.4).

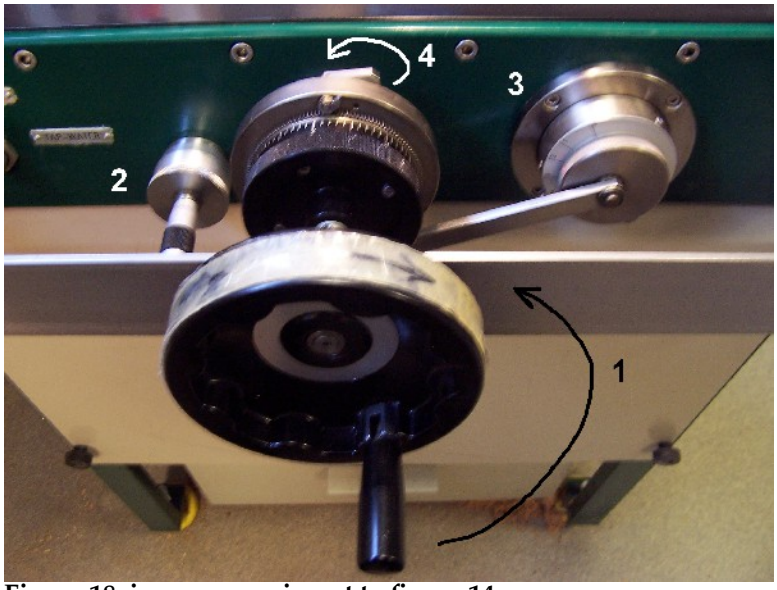


Figure 18: in accompaniment to figure 14.

Figure 19: Changing the grinding wheel

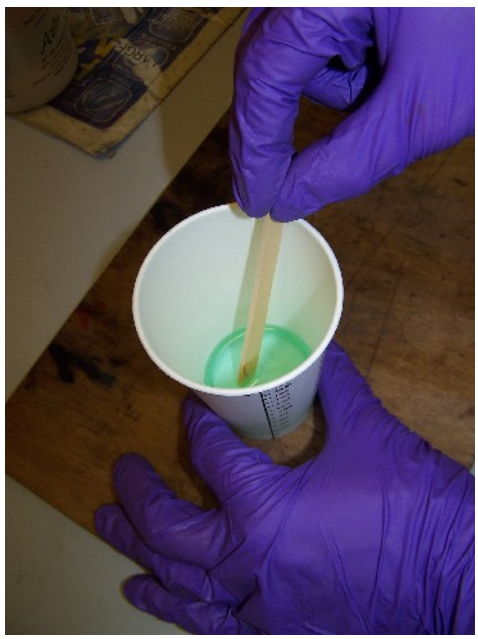
5. The machine is set back to manual, and the slices/slides are taken off the machine, and set face down on a tissue to dry. They are then removed from the glass slide with a palette knife or Stanley blade, and acetone if necessary to soften the super-glue.

6. All oil from machining is wiped from the slices with blue toweling, and they are re-labelled on the rough face. They are then placed on a drying rack, and a cool/warm hair-dryer is used to blow remnant oil out of the slices. Periodic cleaning of the slices with acetone helps lift the oil during the drying-out process.

7. As soon as most oil has been removed from the slices, they can be permanently mounted to prepared, finely polished glass slides. The mounting mixture is 20ml of Polyester Crystic Resin, with .07ml of MEKP. Special care must be taken to wipe the thin-section face clean with acetone, in order to remove any remnant oil or dust before applying the resin. Resin is poured in small amounts onto the slice face, and spread thinly and evenly with a wooden mixing stick. A clean glass slide is then placed, polished face to sample, on top of the resin slice laid on the press. Pressure is applied on the press, and the slices are allowed to set and cure on the glass slides over a 24-hour period.

Figure 20: Pour 20ml of resin into a cup

Figure 21: Add 0.7ml MEKP and stir until resin has changed from green to blue



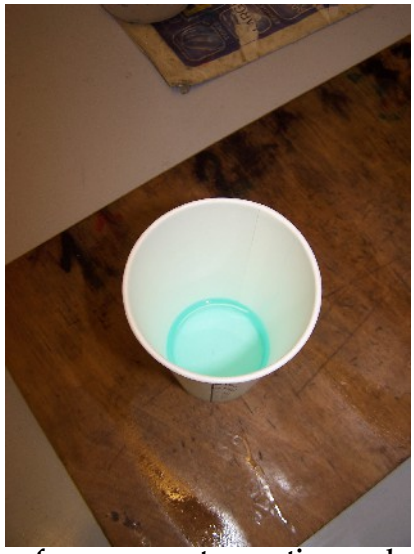


Figure 22: Making resin mixture for permanent mounting, and cover-slipping. Note the colour change in final resin mixture

8. The slices/slides are then taken from the press, and remnant resin cleaned off the glasses back and sides with a Stanley blade, acetone and toweling.

9. The permanently mounted slices/slides are put in their original order back onto the sectioning machine, and the same process of coarse and fine grinding takes place to achieve finished micron-width thin-sections.

Figure 23: Watching slides grind

10. The thin-sections are taken from the machine, and all oil is wiped away. Hand-finishing may be needed to achieve the right over-all micron thickness for microscope analysis. Silicon Carbon sandpaper's, of assorted grades, can be used with some oil to obtain a finished section.

Figure 24: Check thickness of finished slides under the polarizer. Hand finish if required.

Figure 25: Hand finishing of slides on the light block using sand paper and oil.

11. The finished thin-sections are thoroughly cleaned with acetone, and a glass cover slip is applied with the same resin mix used for permanent mounting. A spray cover is also available to seal the finished thin-sections. (Note that if you intend to do micro-probe work on the thin section, the slide should not be cover-slipped at this stage.)

12. Special care should be taken to store thin-sections properly, either in sealed plastic containers, or in a laboratory reference drawer in foam slots. Periodically, the slides should be maintained and cleaned with acetone.