Epoxy impregnation of lake sediment slabs for thin sections
Standard Operating Procedure

LacCore, National Lacustrine Core Facility *1/2015*

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Purpose

Sediment thin-sections allow study of virtually undisturbed sedimentary structures, fabrics, and particles. This technique is used primarily in high-resolution studies of laminated sediments where individual sets of years can be identified. Sediment components studied include charcoal, precipitated components, diatoms, pollen, insects, zooplankton, and in-washed materials. IN the case of hand samples, friable/highly altered igneous/metaphoric rocks are easier to handle and analize.

Carefully prepared blocks of sediment core are embedded in epoxy resin. The blocks are then hardened under heat lamps. The hardened blocks can then be treated as "rocks" for thin-sectioning. This includes cutting, mounting to a slide, making a second cut, and finally grinding and polishing the slide to the desired thickness (usually 30 to 50 µm).

Note: this procedure covers only embedding of freeze-dried slabs with Spurr's-type epoxy. Other techniques, such as acetone-exchange with Spurr's epoxy, and PEG-80 embedding of wet sediments, are not routinely used in the Core Facility.

Equipment

Some of the specialized materials and equipment listed below are pictured throughout this procedure.

Materials

- Slabbing boxes, ends snipped and folded in
- Aluminum trays
- Liquid nitrogen
- Small pieces of paper
- Plastic craft mesh
- Lay flat plastic tubing (only to the dimensions of the metal tray)
- Pink insulation (if only embedding two or less sections)
- Disposable glass pipets and bulb
- Disposable beakers (500 mL or larger for full batch of resin)
- Stir sticks
- Epoxy chemicals (see end of document for recipe)

Equipment

- Slab cutter and/or fishing line, guitar string, wire, etc
- Scoopula
- Engraver
- Freeze dryer
- Dewar (for liquid Nitrogen transport)
- Styrofoam cooler
- Long tongs
- Insulated gloves
- Desiccator with port attached to (house) vacuum
- Vacuum tubing/hose
- Two way valve (that fits into the vacuum tubing)
- Balance weighing to 0.1g (in hood), covered with plastic wrap or foil
- Butyl gloves
- Lab coat, goggles
- Heat Lamp (or two) attached to a ring stand
- Thermometer

Safety

All chemical components of Spurr's type resin are hazardous – carcinogenic, blistering, respiratory hazards, etc. Every step involving resin (mixing, pouring, degassing, etc.) must be done in a fume hood while wearing butyl gloves, lab coat, and goggles. Keep the workplace clean and wipe up all spills. Towels, wipes and other materials should be hazardous-wasted in a baggie kept in the fume hood. At the end of the procedure this baggie can be disposed of using proper hazardous waste disposal procedures. Leftover prepared resin can be cured in its beaker in the hood under the heat lamps along with slabs and discarded in the trash when hard.

Notes

- Humidity can cause problems because one of the chemicals (NSA) is anhydrous, so a climate-controlled environment (such as 6 floors underground) is preferable.
- Slab samples must be completely dry when you begin the impregnation. For clay-rich samples, this can take three days in the freeze dryer, depending on the number of slabs and whether the freeze dryer is defrosted daily.
- The embedding process cannot be interrupted once it is started.
- Impregnation is done by introducing resin from beneath the slabs: resin is poured in beside the slabs and then drawn up into the sample by capillary forces. If the resin is poured on top of the samples, it can destroy the fragile sediments and there is a higher likelihood of air bubbles. Rely on capillary forces.
- Resin has a pot life of 1-4 days (depending on the recipe) if kept cool and airtight.

Procedure

1) Label everything:
   a. Engrave the sample depth and an UP arrow on each slabbing box.
   b. Engrave the name of each sample on the outside bottom of the aluminum trays in approximately the position where the samples will be placed. (3-4 samples can fit in each tray, depending on width of sample boxes used.)
   c. On ~2x10 cm pieces of paper, write a label for each sample with the sample name and an UP arrow in PENCIL (pen will be dissolved by solvents in the resin chemicals). Place label face down inside the aluminum tray in the position where the sample will go.
2) Cut plastic mesh to fit in the bottom of the aluminum tray and place on top of paper labels. Mesh elevates the sample boxes in the aluminum trays while allowing the resin to flow underneath it.
3) Insert a labeled slabbing box into the core and cut under it using the slab cutter or fishing line. Lift out, clean exterior of sampling box and set aside. Repeat for additional samples, overlapping 1-2 cm to provide continuity between slabs.
4) Pour liquid nitrogen into the Styrofoam cooler to a depth of ~10 cm. Lower the slab into the NO2 using tongs. If your sediments are extra watery, you may wish to use a slush of dry ice plus alcohol or acetone for a potentially less violent freeze. Freezing in a regular freezer will allow ice crystals to grow slowly and get large, potentially disrupting the sedimentary structures. Once the samples are fully frozen (bubbling will slow or stop; 1-10 minutes) move them to a freeze dryer tray and either store in a freezer temporarily (more than a few hours and the ice may recrystallize) or directly to the freeze dryer.

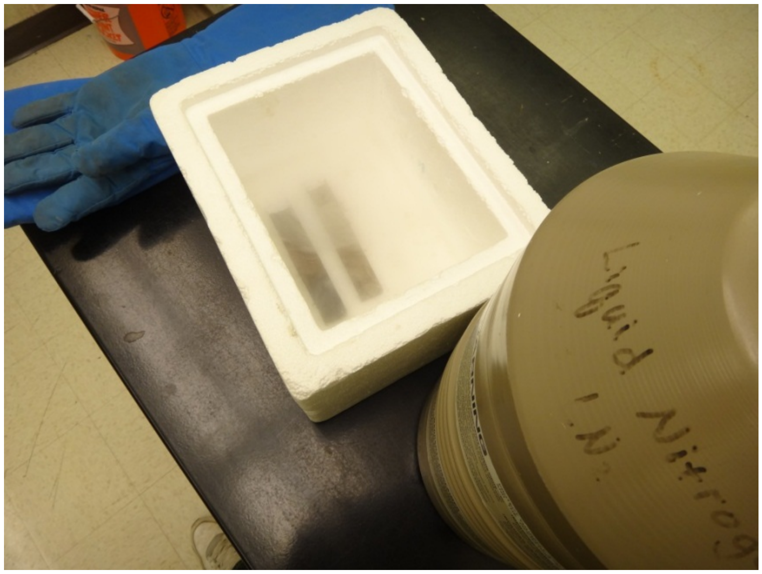

Figure 1. Two slab samples in a Styrofoam cooler, submerged in ~10 cm liquid nitrogen. Dewar is to the right.

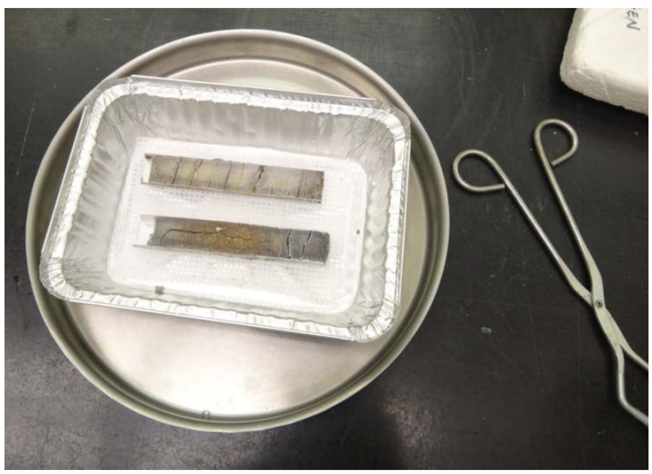

Figure 2. The two slab samples are completely frozen and placed in a metal freeze dryer tray for immediate freeze dry or storage in a freezer.

5) Freeze dry the samples (in the aluminum trays) overnight or for a couple days, depending on the water content of the samples. Drying will progress more quickly if freeze dryer is defrosted and restarted each day, but the process of bringing the chamber to room pressure and moving slabs out and in may disturb samples. Check temperature of the slabs (by hand) for completeness of drying; a cold slabbing box indicates remnant ice in the sample. Samples that are completely or partially dry, especially if they started out with high water content, are very fragile and should be handled as little as possible.
6) Take the samples out of the freeze dryer and check to be sure that the labels are still in place, and match the corresponding slabbing boxes. They can be pushed back into place with a dental tool.

7) If there are less than two sediment slabs being impregnated, fill up the empty space between and around the slabs with pink vacuum insulation. This will make it easier, as you will not have to cut the slabs apart after the epoxy is cured. Be sure to secure the pink insulation to the bottom and/or sides of the tray so it doesn't more around. Regardless of how many slabs are being impregnated, cover the entire tray with lay flat plastic wrap, which allows the thin sections to be removed easily from the sides of the metal tray. Lay small pieces of plastic craft mesh over the plastic tubing that covers the spaces in the metal tray where the thin sections will be placed (See photo, below). If you are impregnating more than two slabs, cut the craft mesh to the size of the tray. Make sure the craft mesh lies flat.

Figure 3. Metal tray set up for two thin sections: Pink insulation in the middle, covered by lay flat plastic tubing. Pieces of plastic craft mesh are cut to the appropriate size to cover the spaces where the thin sections will be placed.

8) Mixing of Spurr resin:

Tables at the end give proportions of different chemicals for various recipes and for a full aluminum tray of samples.

DMAE should not be added unless the ERL 4221, DER and NSA are mixed together. ERL 4221 and DMAE may undergo an exothermic reaction if not properly mixed.

a. Place a tray on the balance to protect the balance from spills, and then place a plastic mixing jar in the tray.
b. Tare the scale with the tray and mixing jar on it. Add the required amount of ERL 4221, tare the scale, add the required amount of DER, tare the scale and add the required amount of NSA.

   c. After adding the ERL 4221, DER, and NSA, stir with a paint stirrer or stir stick (depending on required volume of epoxy) for at least one minute, until the epoxy is well blended. You can see that it is well-mixed when the epoxy is no longer stringy-looking, but even and translucent.
   d. Then, using a disposable glass pipet, add the required amount of DMAE. The DMAE should always be added last, after the first three components have been well mixed.
   e. After adding the DMAE, stir for at least one more minute, until the color is even and orange.
   f. To add blue dye, start with a small volume (pencil eraser sized or smaller). Mix well, stirring at least 1 minute. Check color by removing stir stick and checking film remaining on stir stick. Add more if desired. Adding too much dye can result in opaque epoxy.

9) Once mixed, place the plastic beaker with the resin in the desiccator under vacuum for two minutes to degas.
10) Place aluminum tray in desiccator on a stiff piece of cardboard slightly larger than the tray itself (you may have to bend the aluminum tray slightly to fit it into the desiccator). Place the paper labels under the custom cut craft mesh in the aluminum tray. Then place the samples into the tray, making sure their orientation matches that of the paper labels.
11) If resin beaker is more than about half full, transfer some resin to another container to allow better pouring control. A small funnel or pouring stick can also be a useful tool. Pour the resin into the aluminum trays, to the side of the samples, NOT ON TOP. Begin with a small amount of resin (~0.5 cm depth in tray). As the resin rises through the sediment and displaces the air in void spaces, the air must be able to exit easily out the top. Resin on top of the slabs leads to constriction of escape passages and to bubbles, which can move whole chunks of sediment and disrupt structures.

Figure 4. Desiccator setup: the metal tray is bent to fit into the desiccator, a piece of cardboard is placed under the tray to protect desiccator from spills, samples are carefully placed in their slots, and the tray is labeled T and B (according to orientation)

Figure 5. Adding resin. This can also be done while trays are in desiccator, and should be done under a hood. (Figure courtesy of Pierre Francus)

12) Close dessicator and put under vacuum for 10 minutes to one hour to aid sediment in wicking up the resin. Clay-rich samples (with smaller void spaces) will take longer to take up the resin. If there is bubbling, reduce the vacuum.

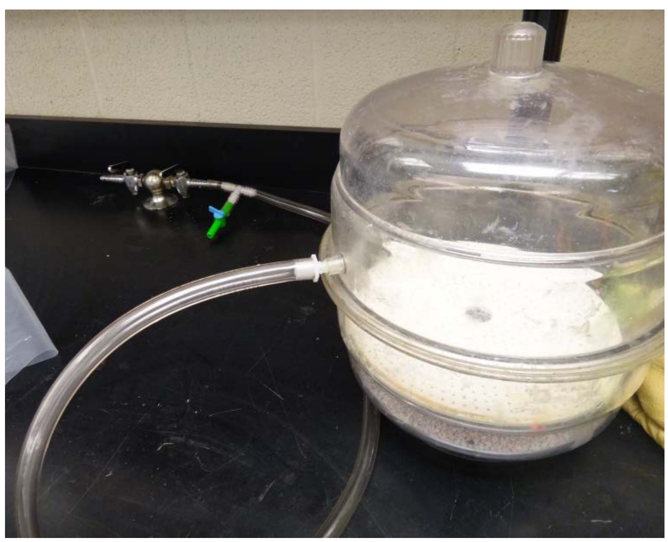

Figure 6. Vacuum set up (note the desiccator should be in the fume hood when epoxy is being poured)

Figure 7. Vacuum set up (2). Note the use of the green T-switch, which allows for the vacuum to be broken without disconnecting the tubing.

13) When first round of resin has been sucked up by samples, turn off the vacuum and release slowly using the T-switch. It is crucial to release the vacuum slowly so the samples don't get disturbed.
14) Add a second, third, and possibly fourth round of resin alongside the samples (making sure not to cover samples with resin), placing trays under vacuum after each addition. The number of times you have to add the resin depends on how fast the slabs absorb the epoxy. In general (and if you have the time), adding the resin slowly is preferable because this will help minimize pockets of air in the slabs. The process is complete when resin has visibly saturated the upper surface of slabs throughout.
15) Once the sediments have finished taking up the resin, only then cover them with enough resin to make a flat surface on top of the slabs.
16) Leave in the desiccator under vacuum for two hours. After two hours, check to be sure no bubbles are evident on slab surfaces and that all slabs are still fully flooded with resin.
17) Remove tray CAREFULLY from the desiccator by lifting the cardboard piece it is sitting on and set in the hood. At this point the trays are heavy and unstable, and any bending can seriously compromise the samples. Leave the tray approximately 45 cm below a heat lamp at ~50°C for 5-7 days to cure; curing is complete when resin is hard and the surface is not sticky at all. A thermometer placed somewhere on the metal tray (not in the epoxy) can give a rough estimate of the curing temperature. If the temperature becomes higher than 80°C, the risk of the epoxy boiling is high, which will damage samples. Cure any leftover mixed resin at the same time.

Figure 8. Epoxy cores curing via heat lamp in a fume hood. Note the heat lamp is approximately 45 cm above the cores so as not risk boiling the epoxy.

18) When curing is complete, “break” the epoxied samples out of the aluminum tray. BE CAREFUL. The easiest way is to peel/cut the tray and plastic off of the epoxied samples, rather than use the samples as leverage (you may break them). Throw the tray and any other cured leftover epoxy in the trash. The epoxy is not hazardous once cured. You can use your fingernale to check to see if the epoxy is cured. Cured epoxy will not be dented with your fingernail.
19) If you embedded more than two samples (and thus a whole tray), use a table saw to carefully to cut the slabs into discrete sections. If the pieces of label paper shifted around, be sure to re label the sections before you cut.

Figure 9. (Left) Finished product (top) Figure 10. (Right) finished product (bottom). Note the paper indicating which side of the sample is at the top (T) and bottom (B) of the core.

Epoxy recipe

Table 1. Chemicals and amounts used in epoxy preparation, per aluminum tray (about 3-4 slabs for lacustrine cores or 8-10 chips for hard rock embedding)

Chemical	Percent of mixture (%)	Amount (g)
ERL 4221	35.7	178.2
DER 736	12.5	60.4
Nonenyl succinic anhydride (NSA)	51.4	225.1
2-diethylaminoethanol	0.4	1.9
Totals	**100.00**	**465.6**

Document History/References

This SOP is based on a long line of sequentially modified versions by A. Myrbo, J. Heck, V. Stanley, N. Schachtman. Technique and initial write up by J. Black, P. Francus.