PREPARATION OF TEPHRA SAMPLES FOR ELECTRON MICROPROBE ANALYSIS

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Pre-requisite

The tephra sample should have previously been processed to remove all residual organic material i.e. it should consist only of tephra +/- mineral grains.

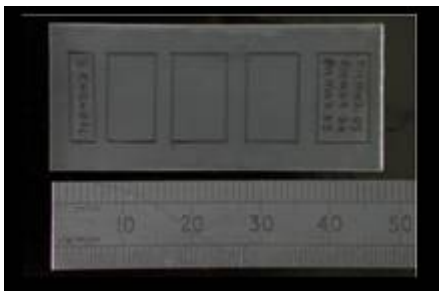
Equipment required

- Glass slides (46mm x 22mm or wider to suit instrument to be used, but **not** longer than 47mm)
- Struers SpeciFix-20 resin (7g) and hardener (1g).
- Top-loading balance, weighing boat, hotplate.
- Sharp pencil, razor blade, tweezers, lollypop and cocktail sticks.
- Pipettes (at least one of each per tephra sample).
- 600 grit loose carborundum powder. Silicon carbide grinding papers with a range of grades (400, 800, 1200, 2500, 4000 grit) flat plate (glass) to support paper.
- 6 µm 3 µm 1 µm diamond laps, compounds and polishing lubricant.
- Decon90, acetone, petroleum ether, beakers and an ultrasonic bath for cleaning.
- Reflected light microscope.

Slide Preparation

Take a clean glass slide of the appropriate size to fit the electron microprobe sample holder and the polishing chucks. Gently grind one face with the 600 carborundum powder on a flat glass plate to produce a frosted surface. This will allow the resin to bond properly to the slide. Ultrasonically clean the slide for a few minutes in water and detergent to remove any residual coarse carborundum. Degrease the slide by washing with acetone.

Using a sharp pencil, mark out areas on the slide to scatter the tephra. Add the sample names. A suggested layout for 3 or 4 samples is shown below in Figure 1. Six samples can be mounted with care by making the 'boxes' smaller. Be sure to leave a 4mm gap between the ends of the slide and the sample/your name areas.



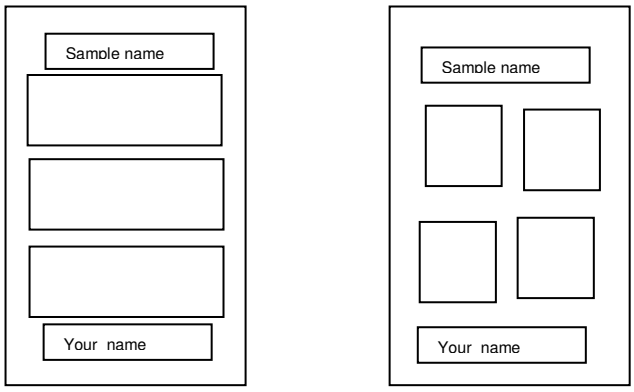


Figure 1.

Resin Mixing

Mix up a batch of resin in a polystyrene weighing boat. Each individual sample will require ~4-6 drops of the mixed resin. The resin should be mixed using a lollypop in the *proportion* 7:1 **by weight** resin to hardener (e.g. 14g resin to 2g hardener). Do not introduce bubbles while mixing, but ensure the two components are thoroughly mixed.

Sample mounting

Spread a thin film of resin over the sample labels and the sample areas. Then sprinkle the tephra shards onto resin film. Allow to dry until tacky, then with the pipette add more drops of resin on top. This method will stop shards from 'floating' to surface and help keep the shards at the same relative height (depth) for ease of polishing.

Repeat for other samples until the slide is full. Try to leave a gap between the blocks to prevent cross contamination. Also, be sure to leave a gap of ~4 mm at the top and bottom of the slide - this is important for correct (level) mounting in the e-probe sample holder

Leave the slide at room temperature for 1-2 hours then transfer the slide to the hot plate for 60 minutes to allow the resin to fully cure. Remove from hotplate and allow to cool. The slide can also be left to cure overnight with no need for heating on the hotplate.

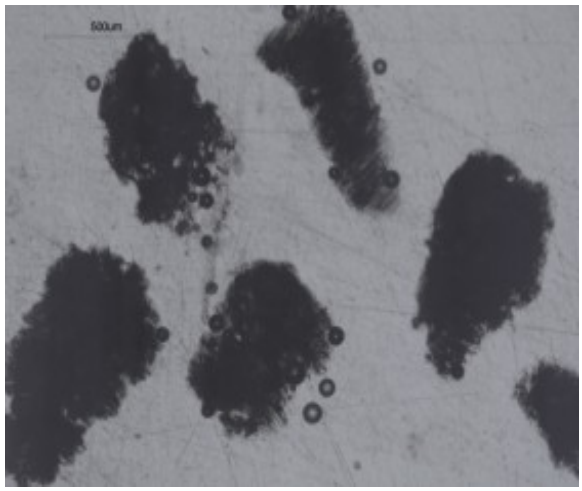
Grinding

The sample must be ground to expose the tephra shards on a flat surface prior to polishing. This should be done progressively and carefully to produce a uniform, flat surface, with minimal loss of material.

Grinding should be done sequentially through the following grades: 400, 800, 1200 and finally 2500: The coarser grades cut, the finer grades polish. Ensure the papers are wet. When the paper is new, a small amount of washing up liquid can be added to the water to help the slide move easily over the grinding paper. Spend enough time on the 400 grade to expose tephra shards on the surface of the resin (but do not grind to a *sample thickness* of less than 0.1mm). Exposed shards will be reflective relative to the resin when viewed

under the reflected light microscope. Move the sample with a "figure of eight" motion, also rotating the sample periodically to ensure even grinding. Then follow the procedure with the other grades spending 2-3 minutes on each grade. The micrometer should be used frequently to monitor sample thicknesses and 'bias' pressure during polishing to maintain relative equal sample thicknesses over section – it is important that the sample is flat!

Example of what slide should look like after 2500 silicon carbide paper



Following grinding put the slides in a slide holder in a beaker of pet-ether and place in the ultrasonic bath for 5 – 10 minutes to remove grinding media and any loose but trapped sample cuttings.

Final Polishing

Samples should be polished using the 6 μm diamond polishing lap and then the 1 μm polishing lap for up to15 minutes each – *BUT* check the surface under the optical microscope after approximately 2 minutes. Ultrasonically clean the slides in pet-ether for 5-10 minutes between the 6 and 1 μm polishing grades and after the 1 μm polishing step. Check that the tephra shards are well polished using the reflected light microscope before carbon coating the samples prior to analysis.

Example of what slide should look like after 1um polishing