

## School of the Environment Environmental Geochemistry Laboratory (EGL) procedures

### Major Element Determination by Lithium Metaborate Fusion and ICP- OES finish (University of Queensland)

Major element geochemistry is determined using lithium metaborate flux followed by digestion of the fused disc using 5% nitric acid (HNO<sub>3</sub>) and analysed using a Perkin Elmer Optima 8300 DV inductively-coupled plasma optical emission spectroscope (ICP-OES).

100 mg of the sample is weighed into a platinum crucible together with 400 mg of lithium metaborate flux and fused in a Katanax automatic fluxer. The crucibles are heated to a temperature of 920 °C, kept there for three minutes and lightly agitated to ensure good mixing. Then the temperature is raised to 1000 °C for two minutes to ensure total dissolution before cooling. The slightly cooled glass bead is decanted into a Teflon vessel containing 100 mL of 5% nitric acid and agitated by magnetic stirrer until totally dissolved. The solution is then analysed by ICP-OES after addition of Lu, Sc and Y for quality check and drift correction.

The elements analysed include Al, Ca, Fe, K, Mg, Mn, Na, P, Si and Ti, and results are expressed as percentages of oxides with detection limits typically ranging from 0.001 to 0.003%. Oxides are calculated as a percentage of the total sample composition.

Water and volatile contents are determined by loss on ignition. Approximately 2 g of sample material is placed in a porcelain crucible and accurately weighed. The crucibles are placed in an oven and heated to 105 °C for two hours, cooled and weighed to determine moisture content. The crucibles are then placed in a muffle furnace at 1000 °C for two hours, cooled, and weighed again. The difference in weight is calculated as a percentage weight loss of volatiles.

The percentage oxide values are added to the percentage weight loss of moisture and volatiles. A margin of 100% + 0.5% is deemed as an acceptable result.

### Trace Elements Determination by Open Beaker Digestion using Multiple Acid Digestions (HF, HCl, HNO<sub>3</sub>) and

### ICP-MS finish (University of Queensland).

Multi-acid open beaker digestion uses a combination of HNO<sub>3</sub> (nitric acid), HF (hydrofluoric acid) and HCl (hydrochloric acid) to dissolve silicate minerals that produces a 'near-total digestion' of sample material.

100 mg of the samples are digested in closed Teflon beakers on a hot plate set at 140 °C overnight to ensure total dissolution. The first step is total digestion by 2mL of concentrated nitric acid (HNO<sub>3</sub>) at 140 °C overnight. The digest is then dried. The next step is addition of 3 mL of concentrated HF + 1 mL concentrated HNO<sub>3</sub>, heating overnight again, followed by drying down as previously. The dried samples are then treated with 2 mL of 6M HCl, heated and dried as previously. Chlorides are removed by another treatment with 1 mL of concentrated HNO<sub>3</sub>, and dried. The final step takes up the dry, digested sample in 2 mL of 6M HNO<sub>3</sub> and heated for 2 hours at 140 °C. This solution is then transferred to an auto-sampler tube and made up to 10 mL with milli-Q water. This is the stock solution from which a further dilution is made for analysis by inductively-coupled plasma mass spectrometry (ICP-MS) after the addition of a range of internal standards covering all masses (<sup>6</sup>Li, <sup>61</sup>Ni, <sup>103</sup>Rh, <sup>115</sup>In, <sup>187</sup>Re and <sup>235</sup>U).

The combination of acids is effective for most multi-element dissolution at trace levels concentrations, however in some cases and depending on the original nature of the sample some volatile elements (B, As, Pb, Ge and Sb) may be lost and refractory minerals containing Zr, Hf, Sn, W, Nb and Ta may not be completely digested. This possibility can be verified by cross-checking ICP-MS results for Zr and Nb with ICP-OES results (ICP-OES analyses are performed on fused, and therefore totally dissolved, samples), and ICP-OES results are used where appropriate. This correction cannot be applied to Sn, W, Ta and Hf because their concentrations are typically below the limit of detection of ICP-OES.

An Agilent 7900 quadrupole ICP-MS is used to analyse trace concentrations of metals. The entire range from 7Li to 238U may be determined, although non-metals and noble gases are not optimally analysed by this technique. Elements most commonly analysed by our laboratory using this technique are the transition elements, the heavy metals, rare earth elements, Pb, Th and U.