

Analytical Conditions for the Electron Microprobe (EMP) Analysis

EMP analysis was undertaken to determine the concentrations of the major elements in the individual glasses and to check for homogeneity. It was thought that due to the glass-making process the final glasses may not be homogenous, and as different batches were made at different times by different people there may also be variations in major element concentrations.

Randomly selected chips of each REE glass were mounted in epoxy resin (Buehler, Epo-thin) ground and polished (6µm and 1µm diamond) to a flat surface. The sample was then cleaned and coated with thin layer of C.

The analysis was carried out using a CAMECA SX100 5WDS instrument (at the University of Edinburgh) using 20kV, 20nAmp beam current (measured in a Faraday Cup) and a 5 micron spot. The crystal configuration used was PET-LLIF-LLIF-TAP-LPET. Y, La, Ce, Pr and Nd were acquired on the LPET. The other Rare Earth Elements (REEs) were acquired on the LLIF crystals. Calcium was measured on the PET and the Si and Al was analysed using the TAP crystal.

Peak and background counting times were as follows: the peak count times for the REEs were 20s with the background time optimised to around 4s using the following formula:

$$\frac{Time_{peak}}{Time_{background}} = \sqrt{\frac{Peak_{cps}}{Background_{cps}}}$$

The backgrounds were measured at standard offsets of +/- 500 sinθ. The Ca, Si and Al peak and background counting times were 20s. The individual analytical time for each analysis was in the order of 4 minutes.

The Lα x-ray lines were measured for the REEs and the Kα lines for Ca, Si and Al with the flow counters operated in differential PHA mode to remove the effects of high order REE x-ray lines. In addition 1st order REE M-series line overlaps on Al and Si were corrected online. Data were processed using the PAP matrix correction algorithm.

The calibration standards used were the Smithsonian R.E.E. phosphates (Jarosewich, E and Boatner, L.A 1991) with adjustment made for their Pb concentrations for the R.E.E, wollastonite for Ca and Si and a synthetic spinel for Al.

The analytical sequence was arranged in such a way that different batches of the same element glass were analysed consecutively over the shortest possible time interval to avoid any possible instrument drift.

It was assumed that there was no mutual contamination or interferences from other R.E.E. during the analysis.

References

Jarosewich, E and Boatner, L.A. (1991): Rare-earth element reference samples for electron microprobe analysis. *Geostandards Newsletter*, 15: 397-399.