Analytical Conditions for the Atomic Absorption (AA) Spectroscopy and UV Assay (UV)

AA Method

AA analysis was undertaken to determine the concentration REEs in the individual glasses. It is proposed that the theoretical and actual concentrations may vary.

Samples were ground to a fine powder (to aid dissolution) and weighed accurately into crucibles (platinum or Teflon).1ml of Perchloric and 5ml of Hydrofluoric acid were then added. The crucible was placed on a sand bath and the contents evaporated to fumes of Perchloric. A further 1ml of Perchloric was added and the crucible was put back in the sand bath and re-evaporated to fumes of Perchloric.

To the residue 1ml of Perchloric was added and the crucible was warmed gently on a hotplate until the residue had dissolved. The solution was the transferred quantitatively to a 10ml volumetric flask and diluted to the mark with water.

For preparation of the calibration standards the following method was used:

5ml of the appropriate standard solution was pipetted into either a Teflon or platinum crucible and to this 4ml of Perchloric. The crucible was placed on a sand bath and the contents evaporated to fumes of Perchloric. A further 1ml of Perchloric was added and the crucible was put back in the sand bath and re-evaporated to fumes of Perchloric.

To the residue 1ml of Perchloric and 2ml of water were added and the crucible was warmed gently on a hotplate until the residue had dissolved. The solution was the transferred quantitatively to a 10ml volumetric flask and diluted to the mark with water. Standard solutions were made by pipetting known volumes of this solution into 50ml volumetric flasks and diluting to the mark with 1M Perchloric, these volumes were also weighed as an extra check.

Standard solutions containing the appropriate amounts of Al and Ca were also made to check for inferences. No such interferences were found.

UV Method

UV analysis was undertaken to determine the concentration REEs in the individual glasses. It is proposed that the theoretical and actual concentrations may vary.

Samples were ground to a fine powder (to aid dissolution) and weighed accurately into Teflon crucibles.4ml Nitric acid, 2ml of Perchloric and 12ml of Hydrofluoric acid were then added. The crucible was placed on a sand bath and the contents evaporated to fumes of Perchloric. Additional Perchloric was added as needed followed by 1ml water warmed gently on a sand bath until the residue had dissolved. If the residue did not dissolve then the solution was re-evaporated more Perchloric was added, re-evaporated as necessary until a clear solution was achieved. The solution was the transferred quantitatively to a volumetric flask and diluted to the mark with water.

For preparation of the calibration standards the following method was used:

Standards were made from the solution stock originally used to make the gels. 6 different concentrations were made by weighing out increasing amounts of solution stock. 4ml Nitric acid, 2ml of Perchloric and 12ml of Hydrofluoric acid were then added. The crucible was placed on a sand bath and the contents evaporated to fumes of Perchloric. Additional Perchloric was added as needed followed by 1ml water warmed gently on a sand bath until the residue had dissolved. The solution was the transferred quantitatively to a volumetric flask and diluted to the mark with water.

Analysis

The samples were analysed spectrophotometrically (Perkin Elmer) for AA.
The following wavelengths were chosen for each of the glasses
Ce- **446.021nm**
Nd- **792.2nm AA**
Sm**- 401.6nm UV**
Dy- **421.2nm**
Ho- **537.00nm UV**
For UV the cell size chosen was the optimum for the glass in question. In AA the slit size was set in a similar manner. Repeats were carried out varying number of times depending on the amount of material available.