

## **APPENDIX C—**

### **XRF Analysis of Major and Minor Elements Methodology**

#### Sample preparation

Coarse rock fragments were crushed in a Bico Corporation alumina-plate equipped mullite grinder and a Spex shatter box equipped with a ceramic grinding vessel. All rock powder was ground to <80 mesh. Lithium tetraborate in the amount of  $3.6000 \pm 0.0002$  grams was placed in a clean glass bottle with  $0.4000 \pm 0.0001$  grams of the rock powder and mixed for 10 minutes in a Spex Mixer Mill. The homogenous powder was transferred into a lidded, 25-cc platinum (95%)-gold (5%) crucible with three drops of 2% Lil solution (to reduce viscosity) and heated, with the lid on, over a Meeker burner. The molten sample was poured and cast into the crucible lid, forming a 29-mm diameter disk.

It normally took 10 minutes to melt the rock powder. The powder was vigorously stirred during heating. Near the end of heating, the lid of the crucible was removed and heated over a Meeker burner unit it was red-hot. Then the crucible's contents were poured onto the hot lid.

Immediately upon completing pouring, the still hot crucible was placed into a beaker containing enough 4N HCl to cover the crucible.

On the other hand, the lid was placed flat on a surface to cool. The molten rock would freeze to a glass. In 3 to 5 minutes glass disk was cool enough to label the curved side with a magic marker. The resulting disk can be stored indefinitely in a desiccator. The disk was analyzed for the major elements and Sr, Zr, Cr, and V.

A slightly different procedure was done for preparing sample for trace element analysis. Whole rock powder in the amount of  $7.0000 \pm 0.0001$  grams was mixed for 10 minutes with  $1.4000 \pm 0.0002$  grams of high purity microcrystalline cellulose. The mixture was then pressed into a briquette. If the SiO<sub>2</sub> content of the whole rock was greater than 55%, copolywax powder was substituted for the cellulose. The elements measured from the briquette included Rb, Sr, Y, Zr, Nb, Ni, Ga, Cu, Zn, U, Th, Co, Pb, Sc, Cr, and V. La, Ce, and Ba were determined by using an L X-ray line and a mass absorption correction.

#### XRF Methodology

Whole-rock analyses for major and trace elements were performed using a Phillips 2404 X-ray fluorescence vacuum spectrometer equipped with a 102-position sample changer. Working curves for each element of interest were determined by analyzing 55 geochemical rock standards. Accepted chemical data for each of these rock standards has been synthesized by Abbey (1983) and Govindaraju (1994). Between 30 and 55 data points were gathered for each working curve, and various elemental interferences were taken into account. The Rh Compton peak was utilized for a mass absorption correction. Slope and intercept values, together with correction factors for the various wavelength interferences, were calculated and stored on computer.

Loss on Ignition (LOI) was determined by heating pre-weighed rock powder to 950° C for one hour, and then re-weighing the sample to determine the relative percentage of weight loss.

The amount of ferrous Fe was titrated using a modified Reichen and Fahey (1962) method.