X-RAY FLUORESCENCE ANALYTICAL PROCEDURE
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INSTRUMENTS

PHILIPS PW2440 4kW automated XRF spectrometer system with a Rhodium 60kV end window x-ray tube, five x-ray detectors, four primary beam filters, eight analysing crystals, two fixed channels for simultaneous measurement of **Na** and **F,** and **PW2540** 168 sample X-Y autochanger.

AFT 6000/C automated fusion preparation system. **HERZOG HTP 40** Pelletizing Press

SAMPLE PREPARATION

The Major Elements Si Ti Al Fe Mn Mg Ca Na K P and the trace elements **Ba Ce Co Cr Cu Ni Sc V Zn** are analysed using **32mm** diameter fused beads prepared from a 1:5 sample: Lithium Tetraborate mixture.

Other trace elements, from **Fluorine** to **Uranium**, are determined by analysing **40mm** diameter pressed pellets prepared at a pressure of 20 tons from a mixture of 10g sample powder with 2g **Hoechst Wax C Micropowder**.

CALIBRATION

Calibration lines are prepared using between 15 to 40 International Standard Reference Materials (**Geostandards Newsletter**, XVIII, Special Issue, July 1994).

MASS ABSORPTION CORRECTIONS

All concentration values are corrected for mass absorption effects by using a combination of alpha coefficients and/or Compton scatter (in cases where the concentration is less than 1000ppm, etc.).

ACCURACY

The accuracy for Silica is within 0.5%. For the other major elements it is within 1%. For trace elements the accuracy is within 5%. The limiting factor for accuracy is the degree of scatter of analyses from which the consensus values are determined (**Geostandards Newsletter**, XVIII,2, 1994, p 256, see values for **Nb** as an example).

PRECISION

Instrument precision: within 0.3% relative, generally within 0.23% relative

This is the percent relative variation obtained when the same disc is analysed repeatedly for the same element.

Overall precision for beads and pressed pellets: within 0.5% relative

This is determined by repeatedly analysing two beads or pressed pellets prepared from two different aliquots removed from the same sample powder vial during the same day and used to prepare a fused bead or pressed pellet by an experienced operator using the routine procedure.

Nota bene: The above accuracy and precision values apply only when the concentration for a particular element exceeds its **quantitation limit** (ACS Committee on Environmental Improvement, 1980), which is defined as the 10 sigma value of the background for it.

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