**X-ray Laboratory**

The X-ray lab contains two major research instruments. The first is a PANalytical 2404 X-ray fluorescence vacuum spectrometer equipped with a PW2540 X-Y sample handler capable of dealing with 168 individual samples and a 4kW Rh super sharp X-ray tube. The lab also has a PANalytical X’Pert PRO X-ray diffractometer equipped with a 15-position sample changer and a ceramic Cu X-ray tube. Both instruments are available for contract work. Please contact Dr. Stan Mertzman for details concerning cost per analysis, turnaround time, etc.

The personnel who handle day-to-day operation of the X-ray instrumentation are Dr. Stan Mertzman, the Earl D. Stage and Mary E. Stage Professor of Geosciences, Steve Sylvester, Research Specialist, and Karen Mertzman, senior lab technician.

**XRD and XRF in Brief - More detailed instructions are available if needed.**

**X-Ray Diffraction**

Students and faculty in the Departments of Earth and Environment, Chemistry and Physics use the PANalytical X’Pert Pro x-ray diffractometer. Examples of student and faculty XRD research include identification of minerals in rocks, soils and lake sediment, characterizing nanoparticles, and the properties of synthetic double chain salts.

**X-Ray Fluorescence**

**Sample preparation for major element analysis**

Crushed rock powder (0.4000 +/- 0.0001 grams) is mixed with lithium tetraborate (3.6000 +/- 0.0002 grams), placed in a platinum crucible and heated with a meeker burner until molten. The molten material is transferred to a platinum casting dish and quenched. This procedure produces a glass disk that is used for XRF analysis of SiO\textsubscript{2}, Al\textsubscript{2}O\textsubscript{3}, CaO, K\textsubscript{2}O, P\textsubscript{2}O\textsubscript{5}, TiO\textsubscript{2}, total iron reported as Fe\textsubscript{2}O\textsubscript{3}, MnO, Na\textsubscript{2}O and MgO.

Working curves for each element are determined by analyzing geochemical rock standards (See Abbey (1983) and Govindaraju (1994) for chemical analyses of the rock standards). Between 30 and 50 data points are gathered for each working curve; various element interferences are also taken into account. Results are calculated and presented as percent oxide.
**Ferrous iron titration and Loss on Ignition**

The amount of ferrous Fe is determined by the titration using a modified Reichen and Fahey (1962) method. XRF determines total iron as Fe$_2$O$_3$. Loss on ignition is determined by heating an exact aliquot of the sample at 950°C for one hour.

**Preparation for XRF trace element analysis**

Trace element analysis is accomplished by weighing out 7.0000 +/- 0.0004 grams of whole rock powder and adding 1.4000 +/- 0.0002 of high purity copolywax powder, mixing for 10 minutes, and pressing the powder into a briquette. Data are reported as parts per million (ppm) for Rb, Sr, Y, Zr, Nb, Ni, Ga, Cu, Zn, U, Th, Co, Pb, Sc, Cr, V, La, Ce, and Ba. Working curves for each element are determined by analyzing geochemical rock standards, data for which has been synthesized in Abbey (1983) and Govindaraju (1994). Between 30 and 50 data points are gathered for each working curve; various elemental interferences are also taken into account. The Rh Compton peak is utilized for a mass absorption correction for region one elements.

**Important**

Always keep in mind that the original rock or mineral powder must be crushed so the ALL of the sample passes through a clean 80-mesh sieve screen. Do NOT use Tungsten Carbide grinding vessels if at all possible.

**References**


