

**GEOCHEMICAL ROCK ANALYSIS AND THE
THIRTYNINE MILE VOLCANIC FIELD:
A WORKSHOP**

convened by

Stanley A. Mertzman
Department of Geosciences
Franklin and Marshall College
Lancaster, Pennsylvania 17604

Beginning officially on Friday morning, January 4, 1991 and continuing through Wednesday afternoon, January 9, 1991, Lisa Dameron, Chris Roessler, George Booth, and Andrew Tittler accomplished all the analytical chemistry necessary for the successful completion of their independent research projects concerning the Thirtynine Mile volcanic field located in central Colorado. Bill Burris, who was not able to join the group during this time interval, had previously completed his analytical work by spending Thanksgiving weekend and two days between Christmas and New Years in the laboratory. Four separate analytical techniques were utilized by workshop participants. In each case homogeneous finely crushed whole rock powder was utilized as a starting material. Bill and Lisa's samples were crushed at F&M while the remainder were crushed at their home institutions. In this latter case all the powders had to be crushed further because they were quite heterogeneous with regard to grain size. The entire sample powder must be fine enough to pass a 80 mesh sieve screen.

A description of each of the four techniques follows:

LOI (Loss on Ignition)

A loss on ignition procedure was carried out on each sample to determine the percent of volatile components. Approximately 1 g of rock sample powder was weighed into a porcelain crucible. The samples were weighed to the nearest 0.0001 g. The samples were heated to 950°C for 50 minutes. After heating, the samples were immediately transferred into a dessicator for cooling. The samples were re-weighed when they reached room temperature.

FeO Titration

An iron titration was performed on each rock sample in order to determine the percentage of iron present as FeO. A sample weighing $0.5000 \text{ g} \pm 0.0001 \text{ g}$ was weighed into a large alumina crucible. To this sample was added exactly 5 ml of disilled water, 5 ml of concentrated H_2SO_4 , and 5 ml of HF. The crucible was then heated until boiling over a Meeker burner and transferred to a hot plate. The crucible was warmed on the hot plate for 15 minutes, and then transferred to a plastic beaker containing 400 ml of a boric acid solution and several drops of 0.3 M sodium diphenylamine sulfonate which is an indicator.. The concentrated acid solution was transferred into the boric acid solution with minimal contact with the air. The solution was titrated immediately with a standard solution of 9.280×10^{-3} molar $\text{K}_2\text{Cr}_2\text{O}_7$. The amount of titrant was recorded when the solution reached the purple endpoint.

X-ray Fluorescence Analysis (XRF)

In order to perform major element, rock sample powders weighing $0.4000 \text{ g} \pm 0.0001 \text{ g}$ were added to $3.6000 \text{ g} \pm 0.0002 \text{ g}$ of lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$). The samples were placed in clean glass bottles and mixed for ten minutes in a mixer mill to insure homogeneity. The powder was transferred to a 25 ml 95% Pt-5%Au crucible. Three drops of a one percent lithium iodide solution were added to the powder to reduce the viscosity of the sample upon heating. The sample was heated with vigorous stirring over a Meeker burner at full power for approximately 8 minutes. The molten sample was then poured into the hot lid of the platinum crucible. The molten sample cooled into a glass disk suitable for X-ray analysis.

Samples for trace element analysis were prepared by weighing $1.0000 \text{ g} \pm 0.0001 \text{ g}$ of rock powder and $0.5000 \text{ g} \pm 0.0002 \text{ g}$ of high purity microcrystalline cellulose into a plastic vial. The samples were mixed in a mixer mill for 10 minutes. The mixture was then placed on top of a coarser grade of cellulose and pressed into a sample briquette under $50,000 \text{ lbs/in}^2$ of pressure. All X-ray samples were stored in a dessicator until they could be analyzed.

X-ray analysis was carried out on a Diano 8300 X-ray fluorescence

vacuum spectrometer. The instrument was previously calibrated by collecting data for 51 geochemical rock standards and establishing concentration vs. intensity curves for each element of interest. Elemental interferences were taken into account such as high Ti on Al, high Ca on Mg, high Mg on Fe, and SrK γ on Zr, RbK γ on Y. Slope and intercept values are calculated and stored on the IBM hard drive. Trace and major elements were run in similar fashion, except that major elements were reported as weight percent constituent oxide, and trace elements were reported in ppm. Some trace samples required multiplication by a mass absorption coefficient in order to determine the correct values of concentration. The mass absorption coefficients were calculated by a computer program that utilized the major element content of each sample.

Inductively Coupled Plasma Analysis (ICP)

All ICP work was done at Franklin & Marshall College using a Thermo Jarrell Ash ICAP 61. Inductively coupled plasma spectrometry was used for the analysis of trace and selected rare earth elements. Samples for ICP analysis were prepared by weighing 0.7500 ± 0.0001 LiBo₂ and 0.2500 ± 0.0001 g. rock sample powder into a graphite crucible. These samples were then heated to 950°C for 20 minutes, after which they were poured, while still molten, into 50 ml of 6% HNO₃ containing 2 ppm of Cd as an internal standard. The sample bottles were shaken for at least thirty minutes, or until all of the solid had dissolved. All samples were prepared in plastic sample bottles. Samples were run within 72 hours of sample preparation. Aliquots of the solutions were transferred to 8 ml plastic test tubes. A series of samples were aspirated into the plasma by an ISC-240 autosampler. The ICP was interfaced with an IBM PS2 and used ThermoSPEC software to control the experiment. Both standard rock samples and standard solutions of salts were analyzed. Three measurements were taken for each element and an average was reported in ppm.

Lastly, we had a chance as a group to sit down and talk about which chemical diagrams (Harker, oxide-oxide, element-element) might be appropriate to depict their data and why. In addition discussion of the K-Ar dating I've been doing and its impact on each of their respective field areas also occurred. Finally, these students are to be congratulated because of their ready willingness to work into the wee hours of each morning they were here to get the work done.

Workshop on Computer Applications in Geology

Faculty

Robert Burger, Smith
Walter Coppinger, Trinity
William Fox, Williams
Glenn Kroeger, Trinity
Richard Stenstrom, Beloit
Robert Sternberg, Franklin and Marshall

Students

Laura Banfield, Amherst
Andrew Brill, Williams
Karen Christensen, Whitman
Robert Cooper, Amherst
Kathyrn Desmarais, Beloit
Alison Harper, Trinity
Susan Jennings, Trinity
Christy Johnson, Williams
Gia Khazaradze, Tbilisi/Williams
Ian King, Amherst
Joe Klinger, Franklin and Marshall
Ryan Lapidus, Amherst
Nick Loizeaux, Williams
Bob Pohl, Beloit
Ann Puffal, Smith