# FROM THE INDOOR LAB TO THE OUTDOOR LAB: USING XRF DATA IN AID OF INTERPRETING FIELD RELATIONSHIPS

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## Abstract

Many geology departments at N. American universities contain a plethora of sophisticated instruments for the analysis of rocks, minerals, waters, and other kinds of earth materials. The questions arise as to how to train students effectively in the use of such equipment, and how to demonstrate that their analytical results can be used to address real geological problems. One approach developed in the XRF lab at WashU involves a semester-project in a graduate course taught by RFD, ?Chemical Petrology.? St. Louis is situated close to the St. Francois Mountains, the structural culmination of the Ozark Dome, where the only significant exposures of Precambrian crystalline rocks in the mid-continent region are found. The bedrock consists almost entirely of ~1.4 Ga granites and rhyolites, exposed in a series of nested caldera complexes. Students are each assigned 4-6 closely related samples to investigate, collected previously by the instructor. They are given thin sections of each sample, accompanied by a sawn rock slab. The students learn how to process the slabs for analysis (i.e., by using crushers and a shatterbox to make powders). They are then shown by the lab manager (RAC) how to prepare fused-glass discs and pressed-powder pellets for major- and trace-element XRF analyses, respectively. (The students also prepare a pellet and disc from an in-house working standard, which allows for later comparison among results obtained by each person.) Once the analyses are completed, the students are invited to characterize and interpret their data using all of the theoretical and practical approaches developed in the lecture part of the course. Each student then makes a short presentation on their sample suite to the class, and the class then considers the results in their entirety. The most interesting part of this exercise comes later when the class visits the localities from which the samples were collected. Each student leads a discussion on the outcrop, wherein they consider the petrogenesis of ?their rocks? in light of petrographic observations and evaluation of their chemical analyses. The net result is that everyone, including the instructor, is humbled by the rocks! Nevertheless, this exercise enhances the learning experience for the students because it establishes a close connection between real data and real rocks.

# Indoor Lab

SELECT YOUR SAMPLE. Sample of fine-grained leucogranite that will be processed for chemical analysis.

STEP #1 - SAWING A SLAB. The first step is to cut a representative slab from the sample, using a rock saw. Be sure to trim off exterior portions.

STEP #3 - CRUSHING. A few pieces of the cleaned and dried slab are placed into the steel mortar and pestle, and the entire assembly is then seated into the hydraulic press. The 20-ton ram descends, crushing the rock into small fragments (a wailing sound indicates that it's time to stop). The crushed material (all of it) is then removed from the mortar and pestle, and poured into the ¼" sieve. Repeat this procedure until everything passes through the sieve.

STEP #2 - GRINDING. The next step is to use a diamond grinding wheel to remove any saw marks and any remaining exterior. The sample should be washed thoroughly to remove gunk (note the use of scientific terms), and then cleaned in an ultrasonic cleaner for ~10 minutes. The sample is then dried overnight in a simple lab oven at 110 °C.

STEP #4 - SHATTER-BOXING. This step involves the use of a SPEX shatter-box, equipped with an alumina-ceramic vessel. Although not as efficient as the heavier tungsten-carbide vessel, many years ago we discovered that the use of WC introduces substantial contamination by Co and Ta (elements that we analyze routinely by INA methods). Samples are manipulated using small paintbrushes and tongue depressors.

We begin by "self-contaminating" the vessel, which already has been cleaned, washed, and dried. We put about a half-scoop of sample into the vessel, and then run it in the shatter-box for ~3 min. The resultant powder is discarded, and any adhering dust is removed with a blast of compressed air (under the hood).

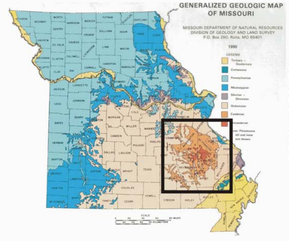
We then add two scoops of rock fragments to the vessel, and these are run for ~10 minutes. We have found that this length of time - for this amount of sample - works very well for virtually any rock (except carbonates, which cake up after about 5 minutes and require special handling).

About 0.4 g of pre-ignited sample powder are weighed into a Pt crucible, to which enough $Li_2B_4O_7$/flux is added to bring the total mass to 4.0 g (actually the most important issue is the 9:1 flux:sample ratio, not the mass per se).

The flux+sample mixture is blended by stirring with a curved spatula. Then we add ~100 ml of LiBr, which acts as a non-wetting agent, especially useful for low-viscosity (viz, high Fe) melt compositions.

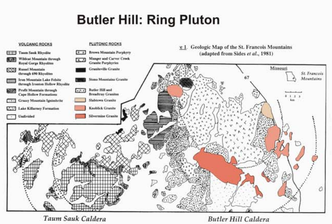
The crucible (covered) is then placed into a cage above a Fisher "Blast Burner," which rotates during fusion to mix the melt. After about 10 min, the melt is poured into a pre-heated Pt mold, and allowed to cool. The result - nearly 100% of the time - is a clear, strain-free glass disc ready for analysis!

# Outdoor Lab




The St. Francois Mountains (SFM), located in southeast Missouri, represent the structural culmination of the Ozark Dome. Of particular interest is the presense there of extensive outcroppings of 1.3-1.4 Ga rhyolite and granite. As such, the rhyolite-granite outcrops in the SFM represent the largest exposures of Precambrian bedrock in the mid-continent area.

## Butler Hill: Ring Pluton




Extensive mapping and laboratory studies by M.E. Bickford and colleagues have revealed the presence of (at least) two caldera complexes in SFM, the largest of which is highlighted in the above map. This so-called "Butler Hill" caldera is characterized by central plutons of fine- to coarse-grained high-silica granite, by medium-grained ring plutons of intermediate silica content, and by voluminous occurrences of rhyolitic ignimbrites. The ring plutons are colored on the above map, and one of these - the Silvermines Pluton - was the main focus of this project.

At Hughes Mountain, a rhyolitic ignimbrite with a well-developed subhorizotal fabric consisting of flattened pumice fragments is characterized by beautiful columnar jointing


The Knoblick Granite, another of the ring plutons, is a shallow-level intrusion that was emplaced into its own volcanic cover.

The Silvermines Granite is locally cut by basaltic dikes, one of which is shown here.

At "Tiemanns Shut-ins," a locality near the center of the Silvermines Granite, typical medium-grained, pale pinkish-gray granite is intruded by a finer-grained diorite, and by later fine-grained aplites. The diorite has commingled with the granite, as manifested by cuspate contacts and widespread globules of diorite within granite.

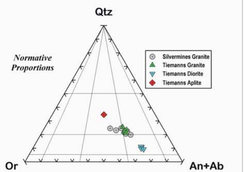
Irregular contact between aplite at the top diorite (left) and granite (right)

Granite (left center) invaded by diorite (lower right), both of which are cut by the aplite (top)

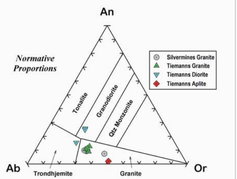
Rounded bodies of diorite within granite.

# Results

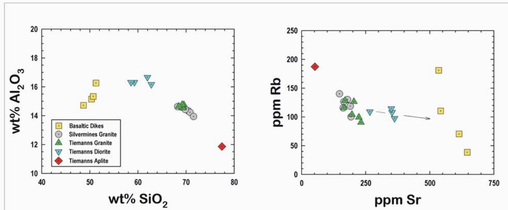
Although granites and rhyolites were analyzed by the class from several localities within the SFM, we focused on the striking features found at Tiemanns Shut-ins. In particular, we wanted to test the hypothesis that the diorites represent the product of mixing between granite and basalt, as proposed for similar-looking mafic enclaves in other settings. So, samples of Silvermines Granites, from Tiemanns and elsewhere, samples of diorite and an aplite from Tiemanns, and basaltic dikes were analyzed for major and trace elements by XRF methods. The resultant data are illustrated in this panel.

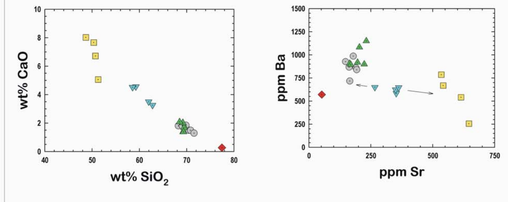


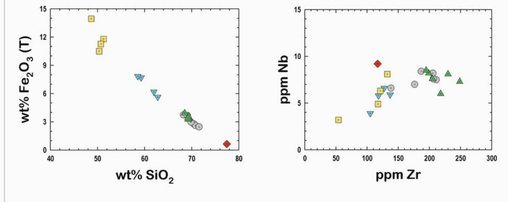

In the Qtz-Or-An+Ab, the analyzed granites and diorites form a linear array, approximately radial to the plagioclase corner. The "granites" lie in the granodiorite field, whereas the "diorites" lie in the quartz-monzodiorite field. Of particular note is the fact that the "Tiemanns granite" are close in composition to "Silvermines granite" from other localities in the pluton.




In the An-Ab-Or ternary, all of the "granites" and the "aplite" lie in the granite field, whereas the "diorites" analyze as granodiorite and trondhjemite.

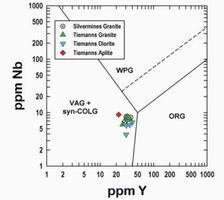


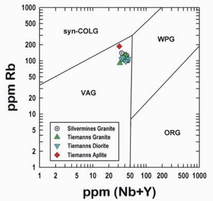



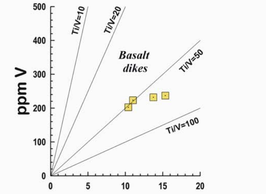

On the Harker variation diagrams for major elements, the diorites lie between the fields for the granites and the basalts, which permits a "mixed" origin for the diorites. The composition of the aplite is consistent with it being a late differentiate of the granites.

The distribution of data points on the Rb-Sr and Ba-Sr plot is consistent with a "mixed" origin for the diorites. However, on the Zr-Nb plot, the diorite analyses overlap with those of the basalts, which is problematic for the mixing hypothesis.




The granites and rhyolites of the SFM are widely regarded by some investigators as examples of A-type or anorogenic granites. Hence, we might expect their compositions to fall within the proscribed fields on various discriminant plots. However, on the Nb - Y and Nb+Y - Rb plots of Pearce et al., the SFM granites (and diorites) lie in the field of "volcanic arc granites" (VAG) rather than the field of "within plate granites" (WPG).




On the Ti - V discriminant plot of Shervais (1982), the basaltic dikes fall in the field of alkalic basalts and continental flood basalts, which is consistent with their setting and overall chemical compositions.