

## **TRACE ELEMENT BACKGROUND CORRECTION AND SMALL SPOT ANALYSIS FOR GEOLOGICAL SAMPLES WITH A NEW 4.2 KW XRF SPECTROMETER**

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Geological samples present difficulties in trace analysis because of the wide range of potential matrices. Common rocks can be rich in Si, Fe, Mg, Ca, Al, or P. Background correction for trace analysis can be challenging across this compositional spectrum, because natural materials are often mixtures rich in several elements. The most common correction applied in trace analysis is use of the tube Compton scattered intensity, but this usage fails unless accompanied by detailed background correction that takes into account changes in often element dependent background slopes. This problem is acute for measured K and L lines with energies below that of the Fe absorption edge, as has been known for some time. In addition to correction for element dependent background slopes, trace (and even major) backgrounds require correction for Compton tailing from large major and minor (and even trace) sample peaks. For elements with K and L line energies above that of the Fe absorption edge these problems are simplified, but the sole usage of a Compton scatter intensity correction for trace backgrounds leads to much larger errors than if a nearby background intensity is measured. For example, in the case of Pb and Th L line measurement, model estimation of background would need to be better than ~0.2% relative to be as effective as actual measurement of background.

Investigation of optimum tube voltage and amperage settings with the new spectrometer yields some surprises. Background count rates for some elements can remain low and unchanged across a wide range in voltage (30-50 kv), rising slightly when voltage reaches 60 kv, and increasing dramatically for 70 kv. For Sc, a difficult trace element to measure due to an overlapping Ca satellite, the optimal voltage appears to be 60 kv, the higher background is compensated by reduction of the Ca interference.

Small spot (0.5 mm) analysis of geological samples can attain precision approaching that of larger samples, but only if enough time is taken to collect the spectra. Experience with analysis at 8 and 15 mm diameter, and some experiments at 5 mm, indicates that a wide range of sample diameters may be analyzed with high precision. There does not appear to be any minimum spot diameter that would not yield useful data. Diameters approaching those used for laser ablation sampling (10-200 microns) are desirable, if the engineering allows. Major and trace element analysis of minerals and natural or fusion glasses would be very useful at these lowest possible diameters, allowing non-destructive analysis for a wide range of earth and material science problems.