# Large Area Standards-based Quantitative EDS Mapping of a Monzogabbro Sample

Stephen M. Seddio[1*]

[1.] Thermo Fisher Scientific, Surface Analysis and Microanalysis, Madison, Wisconsin, United States.
*Corresponding author: stephen.seddio@thermofisher.com

Modern EDS (energy-dispersive spectroscopy) X-ray microanalysis systems enable the acquisition of EDS hyperspectral datasets over entire samples. From these datasets, various maps can be extracted including, qualitative and quantitative elemental distribution maps and phase maps. Here, I explore current possibilities and challenges for large area, standards-based quantitative EDS mapping.

A sample of monzogabbro rock was mounted in epoxy in a 1-inch mount, polished with a final polish of 0.25 μm diamond, and coated with ~1 nm of Ir. The sample was analyzed in an FE-SEM with a 15 kV accelerating voltage and a beam current of approximately 2 nA. The beam current was measured by Faraday cup with the acquisition of each standard EDS spectrum and at the beginning of the automated EDS hyperspectral imaging acquisition. A 100 mm$^2$ active area windowless Thermo Scientific UltraDry EDS detector was used to detect X-rays. The Thermo Scientific Pathfinder X-ray microanalysis system and software were used to acquire and process EDS spectra and hyperspectral images and extract quantitative elemental distribution maps. Dynamic templates for the acquisition of EDS hyperspectral data was used to automatically avoid the acquisition of EDS data from pixels with a backscattered electron image contract that was specified as being epoxy or fracture space. A 40×40 grid of EDS hyperspectral images was acquired, each with 256×192 pixels and a spatial resolution of 1.05 μm/pixel. The EDS spectrum at each pixel was acquired using a 1,600 ns pulse processing time constant, 25% deadtime, and a storage rate of 60,200 counts/s yielding an average counts/pixel of 370.

Standard spectra, used for their X-ray intensities, were acquired from mineral samples an SPI Mineral mount. Albite was used to standardize the Na K-lines. Diopside was used to standardize the Mg and Ca K-lines.  Sanidine was used to standardize the Al and K K-lines. Quartz was used to standardize the Si K-lines. Rutile was used to standardize the Ti K-lines. Cr-Diopside was used to standardize the Fe K-lines. When possible, peak shapes were also used from the intensity standard spectra; however, the factory (standardless) peak shapes for Al and Fe K-lines were used. F, P, S, Cl, Cr, Cu, Y, Zr, and many of the rare earth elements are present at EDS-detectable concentrations in this sample, but appropriate standards were not available for those elements. Therefore, those elements were quantified using standardless EDS analysis. O was calculated by stoichiometry. EDS spectral backgrounds were removed using a top hat filter. Interfering peaks were deconvolved using linear least-squares fitting. Elemental concentrations were determined by the correction of k-ratios using PROZA [1].

The resulting quantitative maps consist of 78,643,200 quantitative analyses and are 10.7×8.0 mm$^2$. In this sample, mapping such a large area at a fine pixel size (1.05 μm/pixel) reveals the abundance of major (pyroxene, plagioclase; Fig. 1), minor (e.g., K-feldspar, silica, magnetite, titanomagnetite, amphibole, apatite), and trace minerals (e.g., zirconolite, Fig. 2; baddeleyite, Fig 2.; chalcopyrite) as well as zoning (pyroxene, plagioclase) and exsolution (plagioclase) within these minerals.

Each map is a TIF file that is 0.23 GB in file size. Displaying multiple maps requires 64-bit software to hold such large files in memory. Typical mineralogical analysis would also include consideration of the

compositions in the mapped area by plotting elemental concentrations on binary or ternary plots. However, with over 78,000,000 analyses, such plotting is prohibited by common spreadsheet software. Additional means of plotting elemental concentrations will be investigated in the future.
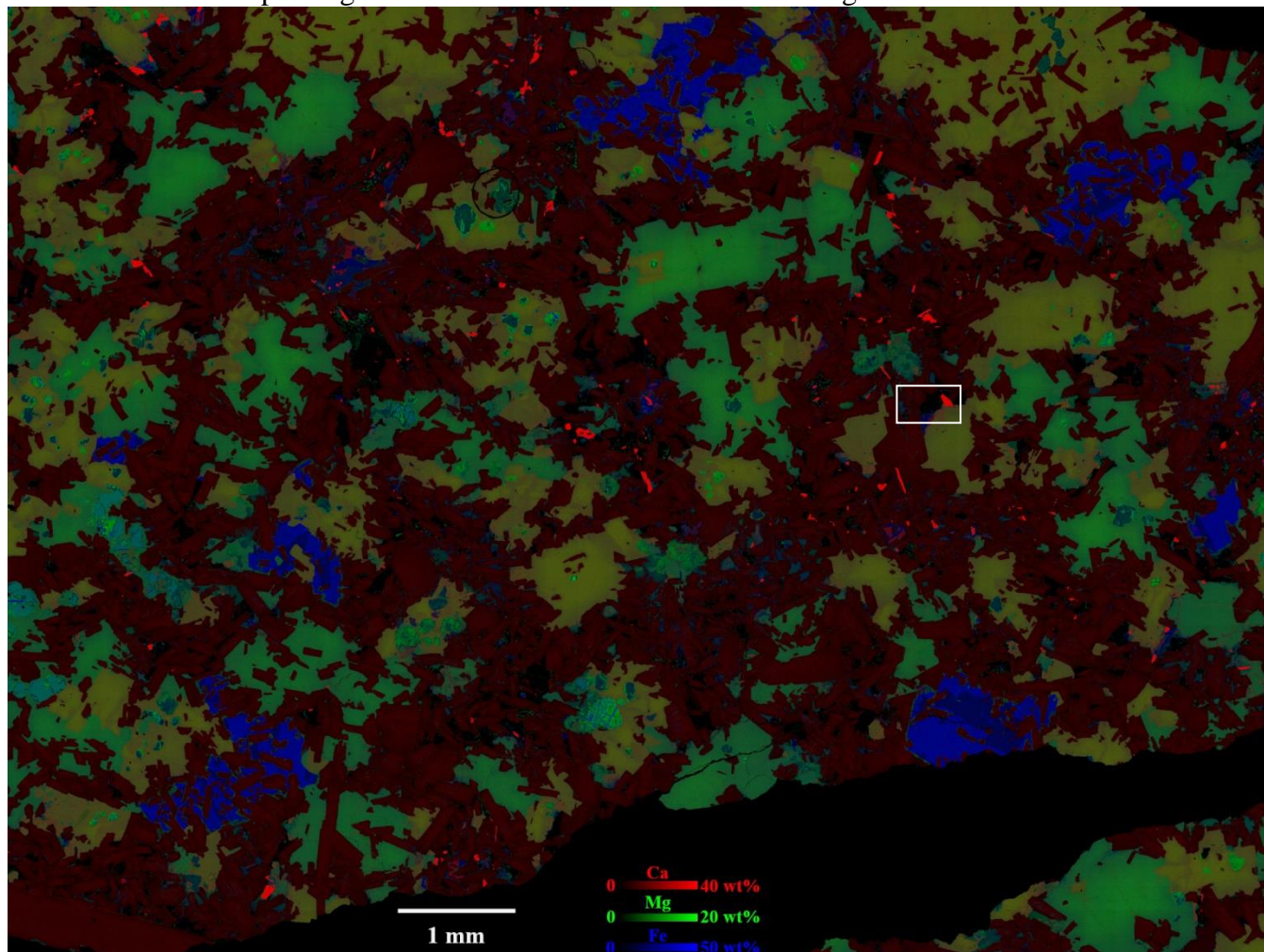


**Figure 1.** Standards-based quantitative EDS maps of Ca (Red), Mg (Green), and Fe (Blue).
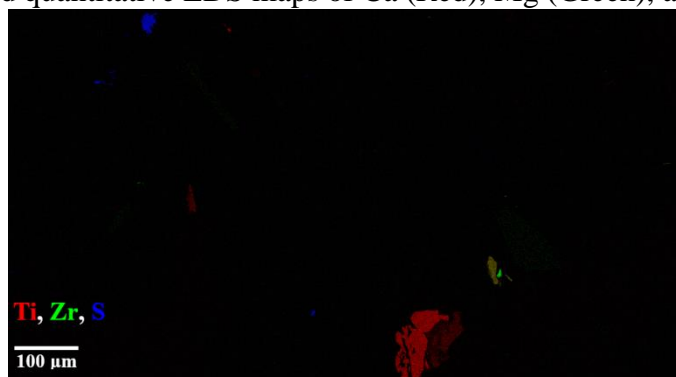


**Figure 2.** Full scale view of the white rectangle in Fig.1. Maps are Ti (Red), Zr (Green), and S (Blue).

Reference:

[1] GF Bastin and HJM Heijligers, Scanning **13** (1991), p. 325. doi:10.1002/sca.4950130502