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Smithsonian Microbeam Standards: Not Just Our Father's Microprobe Standards

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Thanks to the classical gravimetric analytical work of Eugene Jarosewich and his coworkers in the Department of Mineral Sciences of the Smithsonian Institution, laboratories all over the world are using natural and synthetic minerals and glasses for the calibration of electron microprobes. Many of the Smithsonian microbeam standards (SMS), being natural materials, are impure. Gene cautioned us about impurities in the original materials and gave examples of the few that were known at the time [1,2]. Currently, impurities in the SMS are being characterized in composition, size and abundance in order to improve the value of these materials as analytical standards. In an effort to increase the usefulness of these widely distributed materials, cathodoluminescence (CL) spectra have been collected from ten of these. In addition, the SMS are being used as the core materials for a library of energy dispersive x-ray spectra of minerals and glasses.

A polished mount containing grains of twenty-nine of the SMS has been closely examined using a field emission scanning electron microscope. Of these, impurities have been found in twelve. Most of the impurities can be easily observed in backscattered electron images. Compositions of these have been determined by energy dispersive x-ray spectrometry (EDS) and their abundances have been visually estimated. Rockport fayalite (USNM 85276) is particularly problematic as ~10% of the grains are of a higher silica phase, possibly the amphibole grunerite, a known accessory phase in the Rockport fayalite assemblage (see figure 1). The accessory phase also occurs as inclusions in the fayalite. Table 1 lists impurities identified in the standard materials in this preliminary investigation. This listing serves as the beginning of a catalog of these impurities.

Many microprobe and SEM users are familiar with the strong blue CL of the SMS benitoite (USNM 86539, see figure 2A). CL can be a powerful tool in the characterization of geologic materials. Preliminary work has found CL in ten of the SMS mineral standards. Spectral features range from sharp to broad peaks from ultraviolet to infrared wavelengths. The CL spectrum of corundum (USNM 6578) is shown in figure 2B. Table 2 lists selected minerals and preliminary observations of their CL spectral features. Refinement of precise peak positions through improved resolution as well as deconvolution of complex overlapping peaks is underway. It is hoped that the identification of these peaks will become useful as inter-laboratory calibration standards similar to the way these materials have been used as compositional standards for microprobe analysis.

A library of energy dispersive x-ray spectra of minerals and glasses is being created using the minerals in the SMS. Spectra of additional minerals, largely from the Smithsonian collections, are being added to the EDS library. It is intended that this library will continue to grow.

The CL and EDS spectra, as well as the catalog of inclusions, are available on the Department of Mineral Sciences website. These data will be updated as new information about these materials becomes known. Contributions to the catalog of impurities in the standards are welcome. The above information about the SMS, including reference literature and how to request them can be found at: http://mineralsciences.si.edu/

[1] E. Jarosewich et al., *Geostandards Newsletter*, vol. 4 (1980) 43-47.
[2] E. Jarosewich, Journal of Research of the NIST, vol. 107 (2002) 681-685.

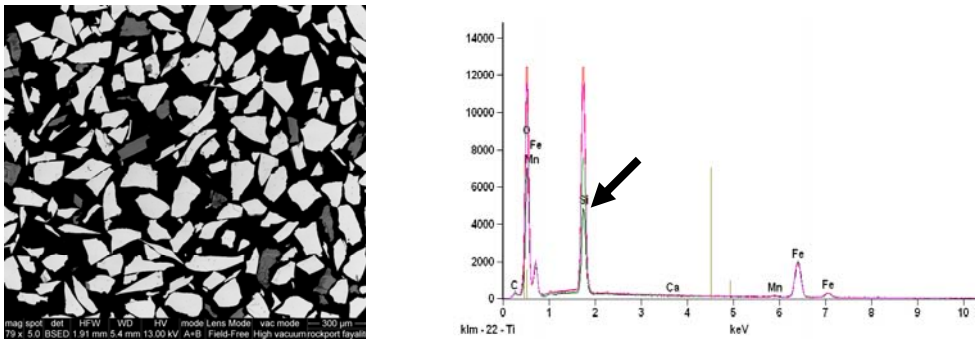

Figure1. BSE image showing obvious population of darker grains in fayalite standard. EDS spectra of fayalite and other phases of higher silica content. Arrow points to fayalite silicon peak.

Table 1. SMS materials found to contain impurities.

Standard material	impurities
augite (USNM 1221420	rare grains with lower Na, Al and higher Fe,Mg, Ca
diopside (USNM 117733)	common calcite/barite inclusions, rare apatite and pyrite inclusions
fayalite (USNM 85276)	10% grains are higher silica phase possibly grunerite, also intergrown
glass VG-2 (USNM 111240)	common tiny olivine crystals, large plagioclase crystal on one grain
glass A-99 (USNM 113498)	common tiny plagioclase, rare tiny clinopyroxene phenocrysts
glass 568 (USNM 72854)	common 5 micron Fe oxide inclusions
hornblende (USNM 143965)	abundant tiny FeTi oxide crystals, rare larger ones
hypersthene (USNM 746)	common chromite veins and included crystals
ilmenite (USNM 96189)	abundant tiny different NbFeTi oxide, rare Nb and Zn phses
magnetite (USNM 114887)	rare ilmenite
microcline (USNM 143966)	rare albite crystals included
omphacite (USNM 110607)	different CaNaMgAl oxide enclosed in jadeite

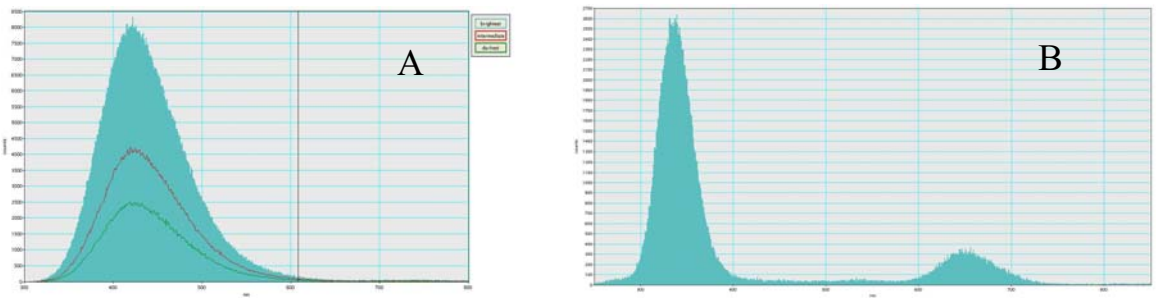

Figure 2: A. 420nm peaks show differing CL intensity of subregions in a single crystal of benitoite. B. CL spectrum of corundum show strong 330nm and weak 680nm peak.

Table 2. CL spectral features of selected SMS. Peaks are in nanometers.

SMS material	approximate peak positions
apatite (USNM 104021)	370, 410, 450, 480, 580, 610, 650
calcite (USNM 136321)	370, 615
anorthite (USNM 137041)	425, 550, 710, 855
strontianite (USNM R10065)	320, 380, 520, 550, 600, 640, 850