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Calibrating Standardless Quantitative EDS Results with Limited Standards

John Konopka[1]

[1]Thermo Fisher Scientific, Madison, WI

Quantitative analysis by EDS using standards is recognized as providing the best accuracy when quantifying elements by EDS. However, standardless quantification is used most frequently due to fewer requirements: no need to measure beam current, no need for a steady electron beam, no need for precise standards. If standardless techniques could provide results with quality approaching that of quantitation with standards it would provide a useful complement to results obtained with standards or with the use of a microprobe. This work will test methods for improving standardless quantitative results by EDS and document their usefulness.

A major challenge with standardless calculations is knowing the response of a given system to soft x-rays. This work will show that the response of the system to one known sample containing some light elements can be applied to a broader suite of samples. This does not require measuring beam current nor is a standard for every element required.

For this work two kinds of samples were analyzed both with and without standards; magnesium silicates and garnets. These were chosen to test the response of the calibration to a range of sample types. All samples were mounted in epoxy, polished and coated with carbon.

The samples were analyzed with a Thermo Scientific NS7 EDS analyze equipped with a 10mm^2 UltraDry SDD EDS detector. This was mounted on a JEOL JSM-7001F thermal assisted FESEM.

The accelerating voltage was set to 15kV and the beam current was about 7.5 nA. Spectra were acquired for 40s live time. This ensured that the major peaks would contain at least 200k counts ensuring maximum precision.

Each unknown was measured seven times in cluster of distinct areas in one field of view. The scan area for each analysis was a circle with a diameter of about 10 microns. The values for oxygen were calculated directly from the peak and not by stoichiometry.

The system response was calibrated by using the spectra from a forsterite standard (Mg_2SiO_4). The thickness of an x-ray absorber was varied till the standardless results agreed with the expected values.

Figure 1 shows the results. Results are presented for each sample calculated with and without standards. Each result is the average of seven individual results. The standard deviations for each set of seven results was mostly less than 0.1 percent and not more than 0.17 percent.

As expected, results reveal that the precision obtained is virtually the same whether the results are calculated with or without standards. In this regard providing a flat, polished, conductive surface on a homogeneous sample and collecting many counts are the most important factors in obtaining a precise result regardless of the correction method applied..

The results by both methods agree quite well for most samples and elements. The samples most similar to forsterite (enstatite, hypersthene, peridot, olivine and serpentine) showed the best agreement. The demantoid garnet results showed the largest variance. As expected, results for oxygen showed the largest variance of any element. This is the lowest energy line measured and likely to be most affected by any part of the measurement process.

This shows that calibration of the EDS system is a simple and valuable action that can significantly improve the accuracy and confidence one has in the results.

Further work will be performed to test a second variety of calibration factor that will improve the results when working with different families of samples. The goal is to further improve the confidence that can be had in standardless results making them a reliable complement to more rigorous forms of analysis.

Atomic Percents	O	Mg	Al	Si	Ca	Ti	V	Mn	Fe	(Mg+Fe)/Si
demantoid garnet stds	59.91	6.50	17.53	12.91	2.92	0.08	0.14		0.01	
demantoid garnet (stds)	56.95	8.09	16.79	15.88	2.19	0.04	0.06		0.01	
almandine garnet stds	60.58	2.63	11.24	13.67	2.80			0.08	8.99	
almandine garnet (stds)	60.77	3.02	9.67	14.95	1.86			0.06	9.66	
enstatite stds	61.22	17.50	0.00	19.45	0.30			0.06	1.47	0.98
enstatite (stds)	59.87	18.35	0.00	20.04	0.19			0.05	1.49	0.99
hypersthene stds	62.12	12.04	1.56	18.08	0.55			0.14	5.50	0.97
hypersthene (stds)	61.27	12.76	1.25	18.70	0.35			0.11	5.56	0.98
peridot stds	58.59	24.41	0.00	13.65	0.03			0.05	3.27	2.03
peridot (stds)	57.36	25.37	0.00	13.95	0.02			0.04	3.26	2.05
Olivine stds	58.32	24.07	0.00	13.68	0.11			0.06	3.75	2.03
Olivine (stds)	57.08	25.06	0.00	14.00	0.07			0.04	3.74	2.06
serpentine stds	66.11	17.94	0.00	14.20	0.03			0.02	1.70	1.38
serpentine (stds)	64.90	18.77	0.00	14.59	0.02			0.01	1.71	1.40

Figure 1. Results. stds indicates use of standards. (stds) indicates standardless results. Results are calculated as atomic percents. Each is the average of seven measurements.

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