doi:10.1017/S1431927615008946 Paper No. 0815 Microsc. Microanal. 21 (Suppl 3), 2015

Quantitative Analysis of Heterogenous Samples by SEM/EDS

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It is well established that samples for quantitative analysis by SEM/EDS must be flat, conductive and homogeneous [1]. However, many analysts are often forced to estimate the composition of a complex sample and their only tool is the standardless quantitative analysis button. Also, one hears that a spectrum from a large field of view produces an average result. The point of this work is to document this problem and offer some suggestions and guidance. The algorithms for quantitative corrections assume a sample with a uniform composition. If the element distribution is not uniform then the wrong correction factors are calculated and applied resulting in a wrong result.

Sampling errors arise as there may be subsurface objects not seen at the analyzed surface. Also, the distribution of materials may change depending on the area viewed causing the result to change depending on the field of view. There is no generic way to account for unseen or unanalyzed portions of the sample short of slicing the sample and fully analyzing each slice or milling the sample and pressing a pellet for analysis.

A suggestion is to analyze a number of areas and then examine the variance of the results. If the results are not consistent then clearly this is a problem sample. A second suggestion is to parse the sample view into regions of homogenous composition and then quantify each area. This result is legitimate as each analysis of a uniform area adheres to the assumptions of the algorithms. Area fractions do not easily extend to bulk composition but at least this result is honest.

Non-homogeneous samples were analyzed with a JEOL JSM-7001F FESEM and a Thermo Scientific NS7 EDS analyzer equipped with an UltraDry silicon drift detector. All measurements were conducted at 15 kV accelerating voltage. Corrections were by standardless Phi Rho Z and results are reported as weight percents. A steel standard was analyzed at seven different locations. The results are shown in Table 1. The results did not agree closely with the known standard composition. A meteorite sample was mapped with Spectral Imaging and the results were parsed with COMPASS^2 and XPhase. The resulting nine phases were quantified by standardless analysis and the overall spectrum was quantified. The results are shown in Table 2.
If forced to characterize a complex sample by SEM/EDS one should, at a minimum, analyze multiple areas to check for consistency. Second, parse the sample and report

the constituents as area fractions. When forced to tackle a challenging sample be honest and report the manner in which the results were acquired. Do not report a result from a single field of view as an "average" result.

[1] Beaman, D. R., Isasi, J. A., ASTM Special Tech. Publ. 506, American Society for Testing and Materials, Philadelphia, Pa., 1972, p. 51.
[2] Advanced Materials and Processes, September 2002, p. 17.

	Si	S	Cr	Mn	Fe	Ni	Cu	Mo
min	0.9	0.2	13.9	0.0	79.7	0.3	0.1	0.2
max	1.0	1.9	14.8	1.8	83.7	0.4	0.1	0.4
avg	1.0	0.7	14.2	0.8	82.5	0.4	0.1	0.3
sd	0.01	0.55	0.31	0.55	1.40	0.03	0.01	0.05
std val	0.84	0.33	13.28	0.41	84.1	0.42	0.10	0.20

Table 1. Summation of analyses of Brammer standard SS 416 at seven locations. Due to the variation in the density of inclusions from one view to the next the variance in the results is quite high. Note that the Mn result ranges from 0.0 to over four times the expected result. Results are weight percents.

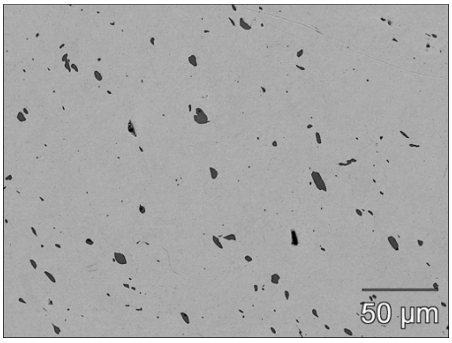

Figure 1. Image of SS 416 steel sample

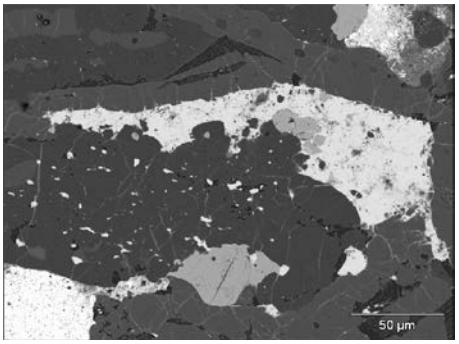

Figure 2. Image of meteorite sample.

	O K	Na K	Mg K	Al K	Si K	S K	Ca K	Ti K	Cr K	Mn K	Fe K	Ni K	Area
Phase 1	41.9	0.0	15.9	0.0	27.0	0.0	0.6	0.0	0.3	0.4	14.0	0.0	0.38
Phase 2	8.8	0.0	1.4	0.1	1.8	31.6	0.0	0.0	0.0	0.0	55.2	1.0	0.16
Phase 3	27.0	0.0	1.7	2.5	0.9	0.8	0.0	1.6	39.0	0.0	26.5	0.0	0.04
Phase 4	2.6	0.0	0.6	0.2	0.4	0.0	0.7	0.0	0.0	0.0	79.0	16.4	0.03
Phase 5	44.0	5.4	4.4	7.1	28.3	0.0	2.6	0.0	0.0	0.0	7.6	0.7	0.05
Phase 6	35.7	0.0	4.6	0.5	7.8	5.5	0.5	0.0	0.0	0.0	30.1	15.3	0.02
Phase 7	39.7	0.0	20.7	0.1	17.4	0.0	0.0	0.0	0.3	0.3	21.4	0.0	0.29
Phase 8	4.4	0.0	0.8	0.0	1.0	5.6	0.0	0.0	0.0	0.0	51.2	37.2	0.01
Phase 9	45.9	0.0	10.1	1.1	23.9	0.0	10.4	0.0	0.0	0.0	8.6	0.0	0.02
Field of View	22.1	0.0	15.2	0.4	20.4	7.2	0.6	0.0	2.3	0.4	29.2	2.2	

Table 2. Weight percent results. Individual phases vs. one spectrum covering the field of view.

https://doi.org/10.1017/S1431927615008946 Published online by Cambridge University Press