doi:10.1017/S1431927615004730 Paper No. 0394 Microsc. Microanal. 21 (Suppl 3), 2015
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Homogeneity and Sample Preparation from Grams to Microns using NAA, µXRF, and SEM-EDS

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When a sample is described as homogeneous, we mean that there is a lack of variation in a measured property over the volume of the sample. The notion of homogeneity is somewhat ill defined because all samples are inhomogeneous at sufficiently small length scales. So to be precise, one must say that a sample is homogeneous to a certain level of variation over a certain length scale. Whether a sample can be considered homogeneous depends upon the application and the length scale, mm or µm.

During Instrumental Neutron Activation Analysis for the certification of NIST SRM 2711a, a soil reference material, excess variability was found in the Al concentration above that expected from counting statistics. Analyses were done by µXRF as an additional test of homogeneity. To investigate further at shorter length scales, we made additional measurements using µXRF and SEM-EDS x-ray spectrum imaging.

For two µXRF preparations, one of which was used for spectrum mapping, a small amount of the sample was embedded in epoxy. For one preparation, the bottom of the mount was examined without polishing. For the second mount, the epoxy was polished. The samples were imaged in an Eagle III µXRF system, which has a nominal spot size of approximately 50 µm. The amount of material probed by the µXRF was significantly smaller than that done by INAA and larger than that done by the SEM (see below).

The polished mount was also investigated by x-ray spectrum imaging on a TESCAN MIRA3 Schottky field emission SEM with 4 Pulsetor silicon drift detectors at 15 keV, 1 nA and 51.2 ms/displayed pixel. The point spectra were quantified against standards using NIST DTSA-II [xx] (Figure 2). We also found that there were some differences between the two µXRF preparations. The mount that was examined from the bottom of the epoxy was much higher in fines than the other mount where the mount that had been polished, and thus was sampled from a more representative location was less homogeneous. The x-ray spectrum mapping showed a wide variety of compositions and almost no homogeneity at all.

The take-away message is that homogeneity like beauty is in the eye of the beholder. A sample that is homogeneous on one length scale may be well suited for its intended purpose but totally unsuited to other purposes at other length scales.

https://doi.org/10.1017/S1431927615004730 Published online by Cambridge University Press

	ppm Al	±			ppm Al	±
srm2710a.1a	59258	492		srm2711a.1a	67710	408
srm2710a.139a	60141	396		srm2711a.368a	67698	421
srm2710a.483a	59906	649		srm2711a.484a	67981	429
srm2710a.756a	58928	532		srm2711a.733a	67943	416
srm2710a.936a	59701	524		srm2711a.1094a	65774	413
srm2710a.1118a	59252	472		srm2711a.1342a	66388	447
srm2710a.1255a	58832	479		srm2711a.1563a	67129	408
srm2710a.1459a	59725	496		srm2711a.1688a	67209	409
srm2710a.1973a	60322	447		srm2711a.1212a	65675	411
srm2710a.1973c	60136	502		srm2711a.1950a	66569	401
srm2710a.1716a	59179	401		srm2711a.2290a	66507	399
srm2710a.1871a	59917	476		srm2711a.last.a	66458	404
srm2710a.2061a	59680	512				
Wtd mean	59621				66912	
Chisq/df	1.06				3.77	

Figure 1. The Al concentration after INAA of SRM 2711a and a comparable material SRM 2710a.

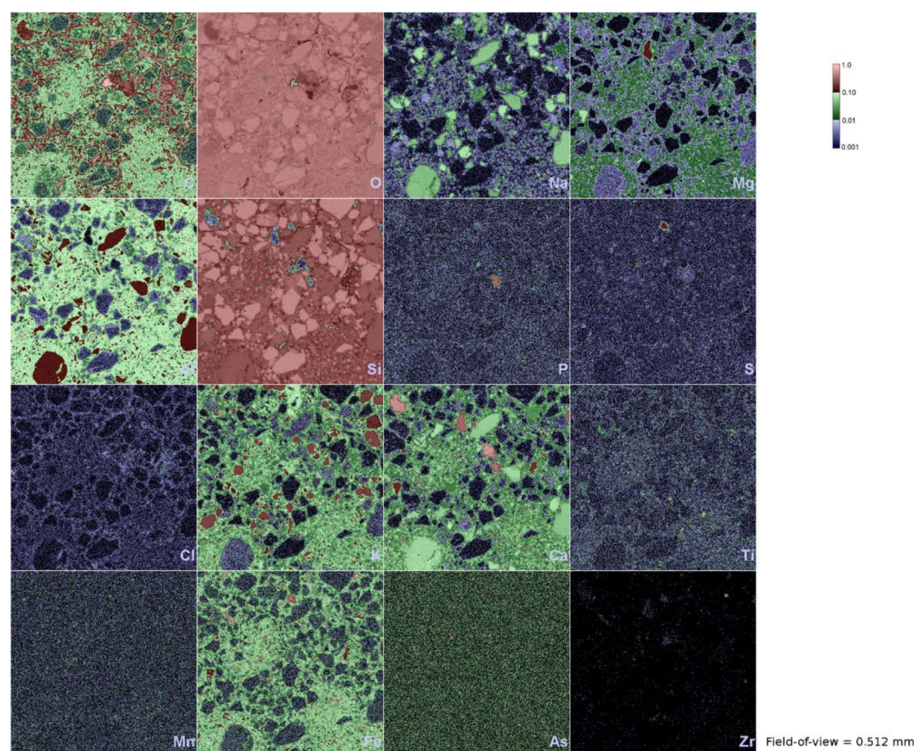

Figure 2. Elemental maps of a region on the polished mount collected on the TESCAN MIRA3 at 15 keV and 51.2 ms per displayed pixel (FOV = 512 µm).

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