

How does homogeneity affect micro-XRF on certified reference materials?

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The homogeneity of standards has always been an issue for standard-based micro-XRF quantification, as well as for any validation routines for respective analytical instruments. Especially for non-technological reference materials, such as geological samples, the question of a representative value arises. Rock samples are composed of individual mineral phases and, hence, show a highly inhomogeneous elemental distribution. Therefore, they are sieved and ground to particle sizes of around 100 μm , in order to be used for XRF elemental analysis. These particle sizes are well above the spot size of nowadays micro-XRF spectrometers.

Even for large spot XRF the grain size effect is a well-known phenomenon. For micro-XRF the additional problem arises that individual particles can be hit which would yield completely misleading results. The only solution is to measure the sample on multiple points or to perform an area scan over a large portion of the sample. Neither is a good solution, as the number of points that will give the “true” average is undefined, and the sum spectrum of an area is not the same as the spectrum of a homogeneous sample.

The best approach is to make the particles smaller. Here we present initial results of a measurement campaign on 10 geological CRMs ground to particles sizes of around 200 nm and subsequently pressed to pellets. The instrument used was Bruker’s M4 Tornado micro-XRF instrument with a Rh-anode X-ray tube and a polycapillary optic with a spot size of less than 20 μm (for Mo-K α).

The results reveal several analytical advantages compared to the untreated CRMs: The sub-micrometer particles are highly adhesive and can be pressed to stable, flat pellets without the use for binders. The restricted size of individual grains suppress diffraction, which makes quantification of trace elements much more stable.

Above all, with the particle size going below both, the spot size of the instrument and the information depth of the analyzed elements in their respective matrices, all matrix effects are inherently taken into account.

With all these benefits combined any single point of the sample gives a representative value for the whole material.