Homogeneity test on a candidate microanalytical Mn-nodule reference material using micro x-ray fluourescence spectroscopy (µXRF)

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Towards improved quantification for µXRF

X-ray fluorescence spectroscopy (XRF) is an established analytical technique. In recent years, the development of improved capillary optics and silicon drift detectors (SDD) has brought forth instruments capable of focusing the incident x-ray beam to a spot size of 10-15 µm enabling analyses with high-spatial resolution at increased speed. These µXRF-instruments acquire an energy dispersive spectum and quantify the data using fundamental parameter (FP) calculations. This quantification is, presently, the best option for µXRF since sufficiently homogenous and matrix-matched reference materials (RM) are scarse. For improved quantification matrix-matched RMs are preferable [1].

Generally, a large variety of powdered geological RMs is available. However, their particle size ranges from 20-150 µm, which is larger than the focused x-ray spot. Therefore, no stoichiometric sampling is achieved and a larger area of the sample would need to be mapped and averaged for meaningful calibration. Using a novel technique, we are able to reduce the particle-size of most rocks and minerals to a size-distribution of D_{50} 200 nm. This enables the production of binder-free pressed pellets which are sufficiently homogneous at the µm-scale and fit for purpose of serving as microanalytcal RMs. Here we present the results of a homogeneity-test conducted on 22 pressed pellets. The original material used in this study were NOD-A1 and NOD-P1, manganese nodules processed by the United States Geological Survey (USGS). These powders were milled in a planetary ball mill and pressed into 10 pellets per material and analysed in a randomised order. One pellet of each respective original material was also analysed as a reference.

[1] Flude et al. (2017) *Mineralogical Magazine* **9,** 923-948.