pXRF and ICP-MS Methodology for REE Quantification in Sedimentary Systems

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Abstract

Assessing the precision of different geochemical methods is crucial for quantifying rare earth elements (REEs) and critical minerals (CMs) in coal-bearing sedimentary strata, with application to broader sedimentary systems. This study analyzed 230 samples via portable X-ray fluorescence (pXRF) and subsequently high-graded them for inductively coupled plasma mass-spectrometry (ICP-MS) analysis. Lithologies include coal, siltstone, shale, sandstone, and igneous dikes. REE quantification relies heavily on pXRF and ICP-MS datasets, but the precision between these two methods is unconstrained. Systematic differences inherently lead to variations in results, encouraging the investigation into the controlling variables associated with each analytical technique. With precision as the primary objective, several homogeneity and spatial experiments were conducted to compare pXRF and ICP-MS geochemical datasets. Results indicate that both datasets exhibit the greatest similarity when analyzing semi-equivalent areas and volumes of samples. Across all lithologies, powdered samples consistently exhibit greater precision between pXRF and ICP-MS datasets than whole rock samples, as seen in improved linear regression r2 values. This finding supports a proposed spatial approach to pXRF that quantifies the homogeneity of a three-dimensional sample. Four whole rock samples spatially analyzed via pXRF show REE-enrichment values statistically similar to that of ICP-MS analysis. Similarly, 64.7% of all samples show no statistically significant difference between repeated and averaged pXRF analyses and correlating ICP-MS data, despite slight variations between individual pXRF analyses of the same sample. Ultimately, coupled high-resolution analyses using complimentary pXRF and ICP-MS methods provide the greatest understanding of REE enrichment with the objective of precision.