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Three natural minerals – sanidine, pyrite and columbite - as potential geologic reference materials. Characterization of chemical homogeneity at a micrometer scale.

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A supply risk assessment of strategic high-technology metals requires a thorough quality assurance of their concentration in ores. Because such metals are usually unevenly distributed at a micrometer scale within natural ore-minerals matrices, spatially-resolved methods must be employed in geometallurgic investigations. Unfortunately, obtaining reliable data with non-absolute micro-analytical methods requires the use of reference materials (RMs) that fulfill the matrix-match criterion. While using natural minerals with optimum chemical composition as reference materials seems to satisfy the matrix criterion, assuring their chemical homogeneity at the sub-μg/g sampling masses usually fails. A solution is to produce synthetic minerals, doped with high-technology metals at trace concentrations, evenly distributed in homogenous chemically-optimal matrices.
Prior to the synthesis, three natural minerals - sanidine, pyrite and columbite - have been tested for lateral chemical and structural homogeneity. The assessment involved both microscopic (optical) and spectroscopic methods. The samples have been visually examined with a reflected light microscope and electron microscope (Back Scattered Emission imaging) exhibiting no optical heterogeneities. The chemical composition has been analyzed with three methods based on X-ray detection: EPMA (Electron Probe Micro Analysis), PIXE (Particle Induced X-ray Emission) and Sy-μXRF (Synchrotron radiation-induced X-ray Fluorescence). EPMA analyses were carried out at the TU Bergakademie Freiberg using a wavelength dispersive spectrometer (WDS) and an accelerating voltage of 20 keV and a beam size of 2 μm2. PIXE data were obtained using a 3 MeV proton beam of about 5x5 μm2 from a 3 MV tandem accelerator at the HZDR in Dresden. Sy-μXRF measurements were performed at the hard X-ray beamline “BAMline” at the BESSY synchrotron facility in Berlin. Samples were exposed in atmosphere to monochromatic X-rays of 20 keV focused with a compound refractive lens to 3x3 μm2.
Petrographically-sensible homogeneity testing procedure has been implemented into statistical analysis of the results accounting for both random and systematic heterogeneity patterns such as nugget and island type as well as periodic wave-type heterogeneities.
Quantitative (EPMA, PIXE) and qualitative (Sy-μXRF) elemental spatial distribution maps have been obtained for major, minor and trace elements for each scan. Several trace elements were detected in each of the matrices: Ga, Ge, Rb, Sr, Ba in sanidine; Ni, Cu, As in pyrite and Zr, Sc, Y, W in columbite. All of them showed irregular distribution patterns, proving that selected mineral specimens are not suitable candidates for reference materials.
The proposed sequence of testes including microscopic and spectroscopic microanalytical techniques and standardized statistical procedures turn out to be adequate in quality assurance of minerals and will be used as a template in examining our synthetic material.