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DAAS Deutscher Arbeitskreis für Analytische Spektroskopie

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Determination of Elemental Composition of Copper-Zinc Ores on Micro- and Macro-Level by Total Reflection X ray Fluorescence Analysis

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Determination of elemental composition of ores is an important technological task. Obtaining the operative information on the elemental composition during the process of ore extraction and enrichment allows optimizing technological processes and increasing the efficiency of production. Receiving the data about the toxic elements is also important for the environmental protection.

Unfortunately, considerable part of analysis time is spent on sample preparation, in particular for its dissolution. In our work we propose to use the method of Total Reflection X-ray Fluorescence analysis (TXRF) for rapid direct determination of elemental composition of ores. TXRF is a modern method of determination of elemental composition which is able to analyze solid and liquid (after drying) state samples. It provides high sensitivity, wide dynamic ranges of concentrations, simultaneous determination of several elements, high reproducibility. Due to low influence of matrix effects no standard samples are required for quantitative analysis, so it can be performed using internal standard technique.

In present work the following analysis sequence is proposed. Sample picked up from conveyor belt is successively grounded along with mass decreasing by using jaw crusher, rod mill and iron mortar until 1 2 mm fragments are obtained. This fraction is milled by Pulverisette 7 (Fritsch, Germany) planetary mill using 10 mm zirconium oxide balls in zirconium oxide bowl at 650 rpm rotation speed during 10 min. A weighted portion (5-6 mg) of finely milled ore sample is placed into the polyethylene vial and filled with ethylene glycol and shaken. We propose to use ethylene glycol as a dispersion medium in order to provide necessary stability of obtained suspension. A nickel internal standard is added and the suspension is treated by ultrasonic radiation to destroy ore agglomerates. The vial is finally shaken to obtain homogeneous suspension. Then a droplet rapidly sampled by micropipette and placed onto quartz reflector. The droplet is being dried on electrical hot plate at 70 80 °C during 1 2 min. Measurements is carried out with energy-dispersive TXRF spectrometer S2 PICOFOX (Bruker Nano, Germany) with Mo Kα (17.5 keV) excitation. Acquisition time was 500 s. The spectra obtained were processed by Spectra 7 (Bruker Nano, Germany) software using optimized Bayes fit technique.

Samples from Uchaly copper-zinc ores deposit located in South Ural Mountains, (Bashkortostan, Russia) were analyzed by proposed technique. Concentration of 13 elements (Al, S, K, Ca, Mn, Fe, Cu, Zn, As, Rb, Sr, Ba, Pb) were determined simultaneously. It was found that copper concentration varies between 0.5 4 mass. % and zinc concentration varies between 2 3 mass. %. All of the ore samples contain high amount of iron (20 25 mass %) and sulfur (15 20 %, 13 % in one sample). They can be classified as sulfide ores (FeS2 matrix) which is the most widespread copper ore. The relative standard deviation of measurements basically does not exceed 10 %.

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