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Geochemical analysis of the European Kupferschiefer – from method development to data assessment

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The European Kupferschiefer is currently the world's most important source of Ag and an important source of Cu, Au, Mo, Ni, Pb, Se and Re. Cu is the main product, with grades of 2.5 wt% Cu and 62 ppm Ag on average. The Kupferschiefer sensu strictu is a very fine-grained, finely laminated and fissile carbonaceous shale. Complex sulphide mineralogy, a great variety of clay minerals and very high organic carbon contents render mineralogical as well as geochemical characterization of the Cu-rich Kupferschiefer difficult. The availability of high quality whole rock geochemical data sets in the published literature is strictly limited. A detailed geochemical study was thus undertaken within the framework of the Ecometals project [1]. Within the context of this study, different analytical methods were compared and a suitable approach for a routine analytical protocol developed.

X-ray fluorescence (XRF) and inductively coupled plasma optical emission spectrometry (ICP-OES) were tested as potential techniques based on their availability and adaptability to analyse main (Al, Ca, Cu, Fe, K, Mg, Pb, Si, Zn) and trace (Ag, As, Ba, Co, Mo, Ni, Ti, V) element concentrations. It is known that both techniques can be susceptible to errors either caused by matrix effects in the material itself, sample preparation or interferences. To ascertain the accuracy of XRF and ICP-OES analysis, instrumental neutron activation analysis (INAA) was carried out at the TRIGA research reactor in Mainz.

Two approaches were tested in the digestion process for this material. Especially the organic carbon in the samples is known to be resistant, which is why Na-peroxide digestion within an HCl solution was used in addition to classic acid digestion based on HF and HNO₃. The ICP-OES results show great accordance to the ones measured by INAA for both acid and peroxide digestion, although a complete digestion was only achieved by peroxide digestion and is reflected in slightly increased Cu concentrations.

Further information needs to be taken into consideration when working with XRF pressed pellets. To process the data correctly, knowledge about the mineralogical composition is necessary to distinguish between oxide and sulphide compounds. The total organic carbon content (TOC) needs to be determined independently and used to correct the XRF data. A preceding calcination of the material can not be recommended and should be approached with caution. The copper sulphides start sintering at temperatures above 780°C and ruin any crucible.

In accordance with the results, both acid and peroxide digestion with subsequent ICP-OES analysis can be recommended for the analysis of Kupferschiefer. Whilst ICP-OES measurements and the sample preparation through digestion are more time consuming compared to the ones used for XRF, the data quality is better and there is no explicit requirement for additional TOC data, mineralogical measurements or data correction.

References:

- [1] Kutschke S et al. (2015) Miner Eng 75:116-125

