Trace element analysis of high-purity graphite materials

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Abstract

The sample preparation for the quantitative elemental analysis of graphite materials according to international standards is typically based on an oxidation process (Ashing). Depending on the applied temperature, the ashing step results in a complex mixture of oxides, sulfates, carbides, etc. Subsequent to ashing, a larger number of additional process steps is performed: a chemical digestion of the ash by melting with borates, dissolving of the resulting melt in acids, preparation of a dilution and, finally, the analysis by spectroscopic methods like ICP-MS, ICP-OES or AAS.

Due to the oxidative and thermal load, some elements (e.g. Na, K, P, S, Hg, Pb, Mo) form volatile components resulting in at least a partial vaporization of these elements and, consequently, in a loss of analyte. The large number of process steps introduces a significant risk of contamination and additional analyte loss.

Nowadays, highly purified graphites are very important materials, applied in large quantities e.g. by the semiconductor industry. The rapid progress in electronic applications has set new and high requirements and standards to the purity level of these graphites. As a result, analytical methods based on the above mentioned procedure do not meet the requirements of up-to-date graphites in terms of analytical precision, reproducibility and detection limits. Solid sampling methods, eliminating the disadvantages of conventional methods, are required.

Electrothermal vaporization in combination with ICP-OES is a very promising method for the analysis of high-purity graphites, exhibiting both high sensitivity and high reproducibility without the risk of cross-contamination or analyte loss. Additionally, this method offers the possibilty for a true quantification and multi-element analysis.