ACCURATE ANALYSIS OF TRACE METALS IN FLY ASH USING ELECTRO-THERMAL–VAPORIZATION IN COMBINATION WITH ICP-MS DETECTION

G. Bauer*, A. Limbeck*

* Vienna University of Technology, Institute of Chemical Technologies and Analytics, Getreidemarkt 9/164-IAC, 1060 Vienna, Austria

INTRODUCTION

Coal fly ash is known for its enriched metal contents but was studied mostly in the field of pollution monitoring and influences on the ecological system [1]. Our goal is to examine the possible recycling potential by determining the contents of target elements, usually metals like Au, Pt or U, which are present in trace concentration levels. Due to its harsh matrix properties, fly ash analysis is a challenging topic, usually demanding intensive sample pre-treatment. Typically some kind of digestion step is involved to separate the organic matrix. These approaches are labor and time demanding and propose the risk of sample-contamination during pre-treatment, furthermore sample dilution accomplished during sample digestion hampers sensitive measurement of target analytes.

In this work, an ETV-based method for direct, fast and accurate ICP-MS-analysis of trace elements and noble metals is proposed. Sample preparation is reduced to simple dispersion of the particulate sample in diluted nitric acid. An aliquot of the derived slurry solution is transferred to a graphite-boat and introduced to an electro thermal vaporization unit coupled to an ICP-MS. The developed method was validated using certified reference material BCR®-176 R from EIC-JRC-IRMM and was applied to fly ash samples originating from the Vienna community heating combustion plant.

EXPERIMENTAL

Analytical procedure

- The fly ash samples were dispersed (concentration < 1 mg/10 ml) in 1% (v/v) nitric acid and engaged in a slurry state by means of a vortexer and ultrasonic agitation.
- 20 µl of slurry solution were transferred into a graphite boat and the solvent was slowly evaporated by means of an IR-Lamp.
- The graphite boat was planted into the ETV-4000 graphite furnace (Spectral Systems, Germany) and an temperature program (see Figure I) was applied.
- Analytes were transferred from ETV 4000 to ICP-MS and emission signals were recorded in transient signal mode (intensity vs. time).

RESULTS

The presence of Uranium was confirmed for all samples. Comparing the sample results against each other the following relation was found:

KSA:FLA:Schlacke = 100:20:65

| Figure I: optimized temperature program | Figure II: transient signal and peak shapes in ETV-4000 EM, Pt graph: 1 ppm, Au 1 ppm | Table I: instrument parameters |

METHOD VALIDATION

Standard reference material SRM BCR 176R was used
- to evaluate the signal quantification and
- to test the accuracy of the method

| Table II: correlation between ICP-MS analysis and SRM certified values | Table III: ICP-MS analysis results for SRM certified values |

REFERENCES


Small amounts of Gold are found in all samples but are still at or below LOD (see Table II). Results for Co and Pt are displayed in Table III.

| Table II: ICP-MS analysis results for SRM certified values | Table III: ICP-MS analysis results for SRM certified values |

SOURCES:

- Quantification of Gold and Uranium
- Validation of ETV-ICP-MS analysis with REE CRM
- Explaining the number of analytes