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Evaluation of a sample preparation procedure for total-reflection X-ray fluorescence analysis of directly collected airborne particulate matter samples

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Abstract

This paper investigates in detail a sample preparation procedure for the total-reflection X-ray fluorescence (TXRF) analysis of airborne particles using a three-stage Dekati™ PM10 cascade impactor. The impactor was modified in such a way, that 30 mm diameter quartz reflectors suitable for TXRF spectrometers can be inserted as collection plates below the impactor's nozzle arrays with cut-off diameters of 10, 2.5 and 1 µm. Smaller particles are collected in a backup filter, which can be analyzed using classical energy-dispersive XRF. As an internal standard 5 ng Y were pipetted centrally onto each reflector after collection. The reflector surface has to be coated in order to reduce particle bounce-off. Two surface coatings, silicone oil and petroleum jelly ("Vaseline"), were compared in this study. Before TXRF analysis this coating layer was removed by low-temperature oxygen plasma ashing. A new approach was introduced, which involves masking of the quartz reflectors in order to remove the outer ring of the spot pattern, which consists of two concentric circles, and to produce a sample, which lies entirely inside the detectable area. Results of this approach were compared to those obtained from unmasked samples and discussed in detail. Angle scans show residue-like behavior of the samples in each stage. Samples were taken on the roof of the Atominstitut building in January 2017 on 10 days during working hours.

1. Introduction

Total-reflection X-ray fluorescence analysis (TXRF) is a simple, fast and accurate analytical method for trace elements, even if the sample is present only in minute quantities. A few nanograms of the sample applied on the surface of a sample carrier are sufficient for a reliable qualitative and quantitative analysis. Samples are usually present as aqueous or acidic solutions, of which a droplet of a few microliters is applied on the reflector surface. After evaporation of the solvent, the sample can be inserted into a TXRF spectrometer for analysis. For quantification, an element of known concentration has to be added to the sample solution as an internal standard. Given the small sample amount, in TXRF the sample can be considered as infinitely thin, so that self-absorption effects within the sample matrix can be neglected. [1]

However, also solid samples can be analyzed, if the sample amount transferred to the sample carrier is sufficiently low (in the ng range). These samples are typically present in particulate or granular form with grain sizes of a few micrometers and below. Applications range from forensics (gunshot residue) or cultural heritage (wipe tests of paint or metallic alloys) to environmental samples (soil, airborne particles). [2]

Airborne particles are classically collected on filters, but can also be collected directly onto TXRF reflectors (quartz, silicon etc.). Typically, a multi-stage cascade impactor, which separates particles from different size fractions, is used for this purpose. The reflectors