DETERMINATION OF GAS-PHASE MERCURY USING TXRF

S.Böttger[1,2], U. E. A. Fittschen[2*]

[1] *Chemistry Departement, Europa-Universität Flensburg, Auf dem Campus 1, 24943 Flensburg, Germany*
[2] *Institute of Inorganic and Analytical Chemistry, TU Clausthal, Clausthal-Zellerfeld, Germany*
**Ursula.fittschen@tu-clausthal.de*

Mercury compounds like $HgCl_2$ were often used to protect cultural heritage specimens e.g. herbaria and paintings from damage. Over time, Hg^0 is formed by bacterial activity and released into the air [1,2]. Accordingly, access to archives may need to be controlled because of the hazard originating from high Hg gas phase concentrations. Marcotte et al. found mercury in dust in museums using an atomic mercury analyser (AAS) [3]. Our aim is to develop a reliable and accurate procedure to analyze airborne Hg using Total Reflection X-ray fluorescence (TXRF); a small footprint and efficient micro-analytical tool already available in many laboratories. The airborne mercury was enriched from the gas phase on silver nanoparticles (AgNPs) prepared directly on TXRF carriers. A similar method has been applied by Romero et al. to enrich Hg from the aqueous phase [4]. We optimized the synthesis of our AgNP preparation method. The last step involved was a washing procedure. The efficiency and reproducibility of the Hg-capture of washed and non-washed AgNP-specimens was studied. The washing step removed approx. 40% of Ag. Interestingly, Hg capture of the washed carriers was significantly higher than of the ones that were just dried. Initially, we used a Ga standard solution as an internal standard on ‘non-washed’ specimens. We found that a low pH during the drying process results in the formation of large Ag crystals. Accordingly, we tested alternative standard solutions having basic to neutral pH i.e. Cr and Mo STD solutions. The TXRF results showed a good reproducibility using Mo (L-lines) however a lower reproducibility for Cr. Micro-XRF studies on the spatial correlation confirmed a good Ag to Mo correlation however poor Ag to Cr correlation. The calibration of “washed” AgNPs using an internal standards proofed to be more problematic. Using the Micro-XRF we found a rather poor correlation of internal standard and Ag. In a next step external calibration using Ag-STD and Hg-STD was evaluated. To determine gas phase Hg concentrations, the Hg uptake on the AgNPs was studied under control room conditions and varying Hg air born concentrations.

[1] D. Briggs, P.D. Sell, M. Block, R.D. I’ons, New Phytol. 94, 1983, 453-457.

[2] J.W. Fellowes, R.A.D. Pattrick, D.I. Green, A. Dent, J.R. Lloyd, C.I. Pearce, Journal of Hazardous Materials 189 (2011), 660-669.

[3] S. Marcotte, L. Estel, S. Minchin, S. Leboucher, S. Le Meur, Atmospheric Pollution Research 8(3), 2017, 483-489.

[4] V. Romero, I. Costas-Mora, I. Lavilla, C. Bendicho, J. Anal. At. Spectrom. 29, 2014, 696-706.