**Abstract**

Experimental conditions have been optimized for volatile species generation (VSG) of four mercury species and their subsequent atomization with detection by atomic absorption spectrometry (AAS). The species investigated were mercury (+II), methylmercury (+I), ethylmercury (+I) and phenylmercury (+I). Flow injection construction of volatile species generator and externally heated quartz tube atomizer were employed. The optimized parameters were the concentration of hydrochloric acid and sodium borohydride, the length of the reaction coil, carrier gas flow rate, atomization temperature and the amount of oxygen present in the atomizer. Furthermore, the effect of the drying tube on the signal of Hg species as well as the applicability of tin chloride as an alternative reductant instead of sodium borohydride were studied. Calibration curves were measured and the basic analytical figures of merit were determined, under optimum experimental conditions, for all four mercury species investigated. Limits of detection for Hg species ranged from 0.12 to 0.25 ng $cm^{-3}$, while sensitivity reached ca 0.10 s $ng^{-1}$ at atomization temperature of 500 °C. Experiments focused on atomization temperature optimization have revealed that volatile compounds generated from organic Hg species decompose significantly. Speciation analysis of mercury in hair samples, quantification of methylmercury and total mercury contents, was performed in the second part of the thesis. Five hair samples and a certified IAEA-086 reference material were studied. Methylmercury was selectively extracted by 2 mol $dm^{-3}$ HCl, which was proven by high-performance liquid chromatography (HPLC) with inductively coupled plasma mass spectrometry (ICP-MS) as a detector. Four analytical techniques were compared for methylmercury determination in the extract: AMA-254, VSG-AAS, ICP-MS and HPLC-ICP-MS. In all cases a good match was achieved between the found values and the certified value for IAEA-086 reference material. Real hair samples contained around 70% of methylmercury.

**Keywords:** atomic absorption spectrometry, mercury species, chemical generation of volatile species, extraction