Abstract

The diploma thesis deals with the study of interferences in the method of UV-photochemical vapour generation of volatile Se species in formic acid media catalysed by nanocrystalline titanium dioxide. High resolution continuum source atomic absorption spectrometry was used for detection.

It was observed that Se^{VI} was less resistant to pH change relative to Se^{IV}, while at pH = 2.3, a 20% increase in response was observed for this specie. It was further observed that both the generated volatile Se^{IV} and Se^{VI} species are resistant to UV radiation in the gas phase.

The Cu^{2+} and Ag^{+} ions were particularly significant interferents in the Se^{IV} determination. Interference behaviour was also observed for Cd^{2+} ions and silver nanoparticles. In the case of Cd^{2+} ions, the generation of cadmium selenide was likely observed. For Zn^{2+}, Fe^{2+}, Co^{2+} ions, there was an increase in response of more than 30% and 40%, in case of Ni^{2+} ions. The Co^{2+} and Ni^{2+} ions were chosen as reaction modifiers.

For the UV-photochemical vapour generation of selenium species with photocatalyst and reaction modifiers, there was a 4-fold improvement in detection limits and limits of quantification compared to the method without these modifiers. In addition, the overall generation efficiencies for Se^{IV} (64.7 %) and Se^{VI} (68.6 %) were determined. Based on the ratio of slopes of the calibration curves with and without modifiers, the one with modifiers has approximately 3-fold generation efficiency.

The NO_3^- ions were a significant interferent for both nitric acid and sodium nitrate because they not only decreased the response but also changed the peak shape. Interference behaviour was observed at higher SO_4^{2-} ions concentrations for both sulfuric acid and sodium sulphate. For Cl^- ions, on the other hand, there was an increase in the relative response up to the highest measured concentration of 1 mol dm^{-3}.

Keywords

Selenite, selenate, interference, titanium dioxide