

Speciation analysis of inorganic antimony in natural waters using the combination of extraction procedures and electrothermal atomic absorption spectrometry

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The aim of the present work was to propose an optimal procedure for the speciation analysis of inorganic antimony in drinking and natural waters prior its determination by electrothermal atomic absorption spectrometry (ET AAS).

Solid phase extraction (SPE) using nano-sized titanium dioxide (which has a high surface area/body weight ratio, high adsorption capacity, and strong coordination of titanium) was used for the separation and preconcentration of total inorganic antimony. Three different modes for the separation, preconcentration and determination of inorganic antimony can be compared: (I) direct TiO₂-slurry ET AAS sampling after adsorption of antimony onto TiO₂, (II) batch mode with the elution of antimony from TiO₂ by a mixture of EDTA and HNO₃, and (III) minicolumn system using TiO₂ with the elution by a mixture of EDTA and HNO₃. Some advantages and drawbacks of these three procedures are discussed and evaluated. Direct TiO₂-slurry sampling offers relatively easy preparation but the high attention has to be pay to the stability of TiO₂-slurry and a serious problem with the damage of a graphite tube leads to the worst reproducibility. Batch mode is the most laborious (tending to the higher risk of sample contamination) but in spite of that it offers higher reproducibility than direct TiO₂-slurry sampling mode. Minicolumn system is flexible, since different initial volumes of a sample can be used (resulting in different enrichment factors and detection limits), reuse of the column is possible after regeneration and high reproducibility is achieved. Briefly, all three procedures are effective for the separation and preconcentration of inorganic antimony in water samples.

Cloud point extraction (CPE) was used for selective separation and preconcentration of inorganic Sb(III) species. After their complexation with ammonium pyrrolidinedithiocarbamate (APDC), the analyte was quantitatively extracted to the surfactant-rich phase in the non-ionic surfactant octyl phenoxy polyethoxy ethanol (Triton X-114). Then the surfactant-rich phase separated by the centrifugation was diluted by HNO₃/ethanol to reduce its high viscosity and the concentrated analyte was introduced into graphite tube of ET AAS. The effects of pH, extraction temperature, extraction and centrifugation time, ionic strength, potential interferences and APDC, Triton X-114, HNO₃ and ethanol concentrations on obtained results were investigated also.

The accuracy of the both optimized methods was checked by certified reference material (CRM) for trace elements in riverine water SLRS-4 (for CPE, the reduction of Sb(V) to Sb(III) with L-cysteine was firstly made). Finally, the proposed methods were used for the speciation analysis of inorganic antimony in drinking and natural waters.

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