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Determination of Sb(III) and Sb(V) by HPLC—Online isotopic dilution—ICP MS

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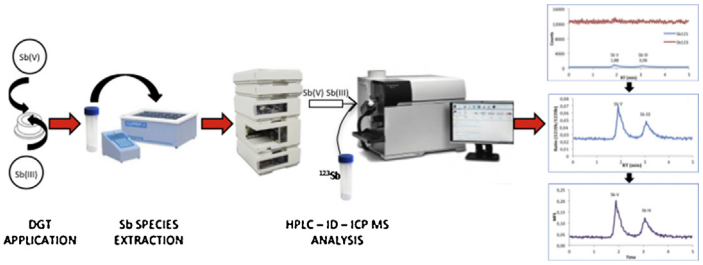
GRAPHICAL ABSTRACT



ABSTRACT

This work provides a method with application of valid techniques to extract and determinate inorganic species of antimony (Sb) for water. The procedure involves

- the simultaneous accumulation of Sb(III) and Sb(V) on passive samplers like Diffusive Gradient in Thin Films (DGT) with iron (Fe) oxide gel, eliminating the risk of speciation changes due to transport and storage;
- application of less concentrated acid (50 mM Na_2EDTA) for elution and preservation of Sb species from DGT resin;
- subsequent analytical determination of inorganic species with High Performance Liquid Chromatography–Isotopic Dilution–Inductively Coupled Plasma Mass Spectrometer (HPLC-ID-ICP MS) based on determination of the isotope ratio ($^{123}Sb/^{121}Sb$) of isotopes in the samples after spiking with 123Sb enriched standard solution, reducing the effect of signal drift and matrix effect on the final value.

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Method details

Most of the analytical methods for antimony assessment are based on the determination of total antimony concentrations. However, it is widely accepted that the impact of a toxic element on the environment is linked to the presence of its chemical forms [1].

Coupled techniques, based on the combination of a separation method with a suitable element adsorption system, have become reliable in speciation analysis to discriminate specific forms of an element.

We report the first investigation of use of Fe-oxide gels in Diffusive Gradient in Thin Films (DGT) for incorporation of inorganic chemical forms of antimony, especially Sb(III), coupled with High Performance Liquid Chromatography–Isotopic Dilution–Inductively Coupled Plasma mass spectrometer (HPLC-ID-ICP MS).

Development of method

To develop the method, test of kinetics of binding and elution efficiency of less concentrated acid (50 mM Na_2EDTA) were applied to ensure an appropriate quantitative recovery of the element from the resin gel of DGT (see SUPPLEMENTARY INFORMATION, Table 1S and Fig. 1S).

The diffusion coefficients of each inorganic species, measured in laboratory using DGT devices in aqueous solution with know concentration of Sb species at pH 5 and 0.01 M, were 7.60 ± 0.05 $10^{-6}\,cm^2\,s^{-1}$ for Sb(III) and 5.23 ± 0.02 7.60 ± 0.05 $10^{-6}\,cm^2\,s^{-1}$ for Sb(V) (see SUPPLEMENTARY INFORMATION, Table 2S and Fig. 2S). These values reflect the ionic characteristics of antimony species [2] and they were applied in DGT equation (Eq. (2)) for Sb species determination aqueous solutions.

The effects of pH and ionic strength were observed. At higher pH, measurements of Sb species in Fe oxide gel agreed with the solution concentrations (see SUPPLEMENTARY INFORMATION, Fig. 4S).

Strong negative effects on Sb species accumulation, especially for Sb(III), by resin gel of DGT were observed when electrolyte concentration was particularly low or absent (see SUPPLEMENTARY INFORMATION, Fig. 3S).

For one day deployment the Method Detection Limit (MDL) for a typical DGT device (0.78 mm thick diffusive gel, 0.13 mm filter) were 0.2 ng mL^{-1} for Sb(V) and 0.4 ng mL^{-1} for Sb(III).

DGT devices application

Materials

- Stock solutions of antimony species (1000 mg l^{-1} for Sb) prepared by antimony (III) potassium tartrate hemihydrate ($C_4H_4KO_7Sb \cdot ½H_2O$), potassium hexahydroxoantimonate ($KSb(OH)_6$) (Carlo Erba Reagents).
- Ultra-pure water prepared by a Milli-Q system (18 MΩ-cm resistance, Millipore® system, Millipore, Bedford, MA).
- Boxes for sampling waters with holders for DGTs devices.
- Iron (Fe) oxide DGTs (0.60 mm Fe-oxide gel, 0.78 mm open pore diffusive gel) (DGT Research Ltd., Lancaster, UK).
- Stirrer.
- Thermostatic chamber.
- Timer.