

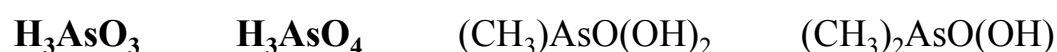
Research Method Development

In-situ separation of As^{III} & As^V during sampling procedure in natural water

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In natural water (groundwater, drinking water and soil pore water) different arsenic compounds like arsenite, arsenate, mono- and dimethylarsinic acids are found depending on conditions like bacterial activity, Eh, and pH. These compounds are characterised by different toxicology, solubility and the adsorption on solid phases.

The following arsenic compounds are found in natural water:



To determine the different species in water samples hydride- generation atomic absorption spectrometry for the routine speciation of the compounds listed above and ICP-MS for total arsenic were used (see also Poster: "Speciation of arsenic compounds in natural water").

In natural waters a changing ratio between As^(III) and As^(V) was observed probably caused by bacterial activity as well as by oxidising or reducing components in the sampled water. In some samples arsenate was reduced to arsenite within days, so that the initial ratio of the samples is unknown. Common methods of stabilising water samples (e.g. addition of acids) affected the As^(III) / As^(V) ratio as well.

To overcome sampling problems, a simple method for a quantitative separation of arsenate and arsenite was developed.

Trace separation of arsenite (AsO₃³⁻) and arsenate (AsO₄³⁻) in natural water samples

Preparation of the anion exchange cartridge

The anion exchange resin Dowex 1 X 8 (100- 200 mesh; Cl⁻ form) was stirred in a batch procedure with 2 M HCl to ensure the complete conversion into the Cl⁻ form. After stirring the resin was filtered and washed with water. 0.6 g of the wet resin was filled into small plastic cartridges and stored until the treatment of the water samples in the field.

Sampling of water

50 ml of water sample were taken using a plastic syringe. 1 ml of a 0,25 M EDTA solution (pH 10 - 11) was added to raise the pH of the sample while avoiding the precipitation of metal hydroxides. At alkaline pH values arsenate (AsO₄³⁻) as a stronger acid is fixed to the exchange resin while arsenite (AsO₃³⁻) and methylated As- compounds (MMAA, DMAA) pass the cartridge and remain in solution. The alkaline water sample must pass the resin with low flow rate for complete separation of the As species. The loaded cartridges were stored, the treated water samples were filled in sealed bottles with no further conservation steps.

Determination of As^(III), As^(V) & As_(total)

As^(III): The alkaline water samples (after separation) were acidified with HCl and reduced with KI. Arsenic (As^(III)) was analysed by HG- AAS with a standard procedure.

As^(V): The elution of arsenate from the exchange cartridges was performed with 25 ml of 2 M HCl. Arsenate elution was proofed to be complete.

As^(V) was reduced to arsenic (As^(III)) by adding KI to the sample-solution and analysed with HG- AAS in a standard procedure.

As_(total) and other elements in the water samples were analysed by ICP- MS analysis from a second set of samples acidified with HNO₃ in the field.

As^(III) / As^(V) speciation results are in good agreement with the ICP- MS data.

As^(III) / As^(V) ratio in drinking water from Hungary, September 1998

In a basic medium, arsenate can be retained on anion exchange resins at pH >10. The arsenic acid is deprotonated and the formed anions (HAsO₄²⁻ / AsO₄³⁻) interact with the anion exchanger. The oxidation of arsenous acid in alkaline solutions is too slow to form arsenate within minutes.

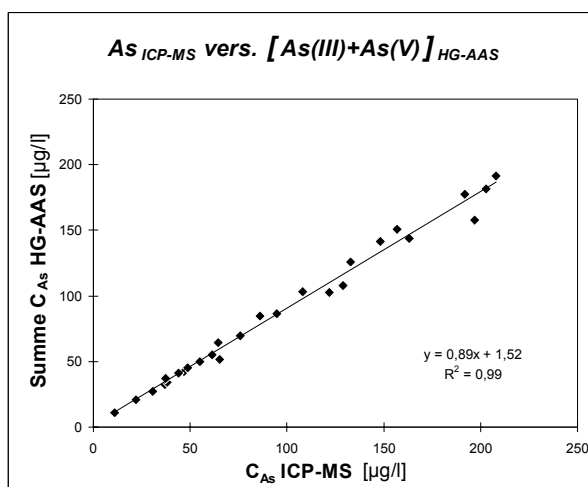


Fig 1

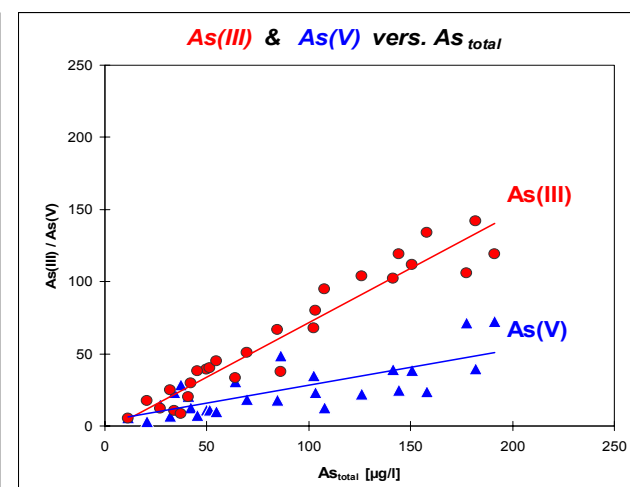


Fig 2