

Speciation of inorganic antimony by stripping voltammetry: advantages of a gold wire electrode.

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Antimony in seawater and fresh water is mainly present as inorganic species, Sb(V) being much more predominant in oxic systems. The total concentrations are usually very low with levels around 0.2 ppb in sea water and even much lower in uncontaminated freshwater[1]. Sb(III), when detected, is rarely present as more than 10% of the total concentration. However, much higher concentrations have been found in mineral water due to release of Sb present within the plastic container PET (polyethylene terephthalate) [2]. This gives an indication of the potential contamination problems related to sample storage of water sample with low levels of antimony.

On-site analysis greatly reduces these problems and voltammetry is ideally suited because of its portability, miniaturisation and sensitivity. Using a gold microwire electrode previously used for Cu, Hg[3] and As[4] detection, the optimum voltammetric procedures for antimony speciation will be presented. The speciation scheme is very similar to the one developed for arsenic[4], is rapid and simple to implement. Sb(III) is first detected by anodic stripping voltammetry (ASV) either at natural pH or in acidic conditions. Sb(III) is then oxidised into Sb(V) by chlorine which is produced electrochemically at the auxiliary electrode after acidification to 0.1 M HCl. The total antimony present as Sb(V) is then directly determined and the original Sb(V) concentration retrieved by simple subtraction. This speciation scheme presents several advantages over other electrochemical methods. For instance, mild acidic conditions (0.1 M) are sufficient for the determination of Sb(V) at the sub ppb level which would usually require either a chemical reduction step to Sb(III) or a very acidic media (5 M HCl) to be achieved. In addition, Sb(III) can be measured at natural pH which not only preserves the original speciation of Sb(III) but also facilitates the analytical procedure: no acid and no stabilising agent (e.g. ascorbic acid) are required.

Particular attention will be given to the interference of arsenic. Arsenic is naturally present at much higher levels than Sb and gives an ASV signal at similar potentials. However, using an adapted deposition potential, Sb does not suffer from As. For instance, at low deposition potential (e.g. -1.8 V) and in acidic conditions ($\text{pH} \leq 1$), arsenic is reduced to arsine which does not remain adsorbed at the electrode and only Sb(V) is accumulated and stripped. Under optimum conditions, low detection limits are obtained for both Sb(III) and Sb(V) (< 0.1 ppb) with short deposition times (c.a. 30s). Total Sb levels in Liverpool Bay and in fresh/mineral water favourably compare with literature values and ICP-MS analysis respectively. Relatively high levels but still lower than the European maximum contaminant level (MCL) of 5 ppb (40 nM) were detected in all tested mineral water.

References:

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