

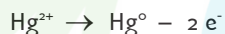
Determination of total Arsenic and Mercury in waters

Types of samples:

All types of water samples

Principle:

The inorganic As(III) and As(V) species, as well as Hg(II) species are electrochemically deposited from the flowing sample solution on the working electrode:



The deposition is performed by applying a suitable constant potential. In the next step, the deposit is stripped galvanostatically, whereas the stripping chronopotentiogram is recorded and evaluated.

Reagents:

KMnO₄, p.a.

Certified reference materials for As(V) and Hg(II)

Solutions:

R-101s:

KMnO₄, 0.01 mol/l:

Carrier electrolyte solution

Reagent solution

Standard solutions for the calibration curve:

The standard solutions are prepared in pure water from the certified reference material:

Standard No. 1: 10 µg/dm³ As(V); 10 µg/dm³ Hg(II);

Standard No. 2: 50 µg/dm³ As(V); 50 µg/dm³ Hg(II);

Standard No. 3: 100 µg/dm³ As(V); 100 µg/dm³ Hg(II);

Note: If appropriate, adjust the composition of the standard solutions to the sample !

Standard solution for the additions:

The standard solution is prepared in pure water from the certified reference material:

100 µg/dm³ As(V); 100 µg/dm³ Hg(II);

Note: If appropriate, adjust the composition of the standard solution to the sample !

Sample preparation:

Sample

Add dropwise KMnO₄ solution to the sample solution until it turns light violet for at least one minute.

Blank sample

The same procedure as that for the sample, just use water instead of the sample.

Electrode:

E-T Au

Experimental:

Potentiostatic deposition

Calibration <input type="radio"/> Calibrationless <input checked="" type="radio"/> Calibration curve <input type="radio"/> Standard addition	Deposition <input type="radio"/> GST <input checked="" type="radio"/> PST	Background reading <input type="radio"/> With the first measurement only <input type="radio"/> With each measurement <input checked="" type="radio"/> With each new sample or standard <input type="radio"/> With each 0 th sample or standard
Calibration curve type <input checked="" type="radio"/> Linear <input type="radio"/> Quadratic curve	Autosampler <input checked="" type="radio"/> Off <input type="radio"/> On	

Experimental:

Setup Parameters

General | Preparation | Regeneration | Measure | Calibration | Calculation | Samples

Deposition
 Edepos: -1600 mV
 Idepos: 0 uA

Potencial (mV)
 Estart1: -200
 Estart2: -200
 Estop: 750
 Eregen: 800
 Estndby: 1000

Pause (s)
 Quiesc1: 10
 Quiesc2: 10
 Regen: 5

Stripping:
 Istrip: 5 uA
 Timeout: 60 s
 Pump: Off On

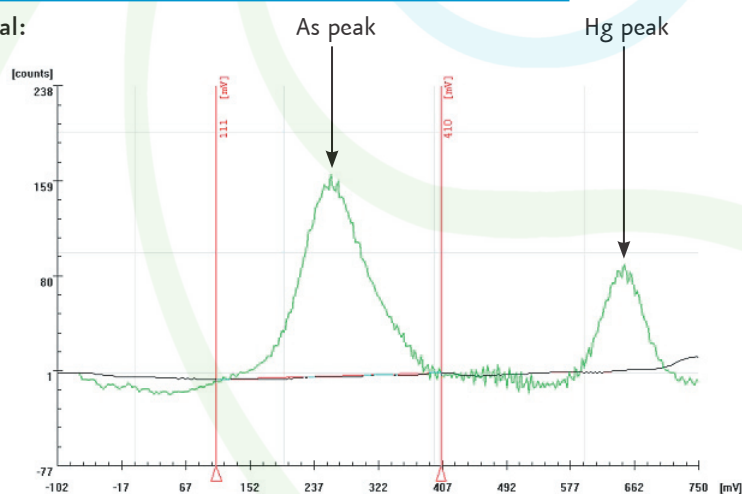
Flow (ml)
 Sample: 1
 Back: 1
 Rinse: 3

Sample segmentation
 No Yes

Measure mode
 Mode A Mode B

Flow: 3 ml/min

Typical signal:



Data evaluation:

Calibration curve or standard additions method

Metrological data:

Concentration range: 0.5 to 500 µg/dm³ As
 0.1 to 1000 µg/dm³ Hg

Reproducibility: 4.2 % at 50 µg/dm³ As
 2.0 % at 50 µg/dm³ Hg

Interferences:

- Some metal ions such as Cu(II), Sb(III), Bi(III), etc., may interfere at higher concentrations though partial or complete overlapping of the As stripping peak. These interferents should be removed with a strong acidic ion exchanger.
- High contents of humic materials may decrease the sensitivity of the measurements.

Notes:

- Samples for higher Arsenic and/or Mercury concentrations than given above should be suitably diluted.
- By a suitable setting of the deposition potential (e.g. to -400 mV instead of -1600 mV), the As(III) species could selectively be deposited. Hence, there is a possibility for speciation measurements. Of course, the permanganate solution must not be added in this case and the calibration should be performed with standard solutions of As(III) !