

Determination of mercury in water with the scTRACE Gold

Summary

Mercury and its compounds are toxic. Acute poisoning is rather seldom. More likely is a chronic poisoning with mercury compounds ingested with the food. A natural source of mercury in the environment are volcanos. But a significant part of the mercury present in the environment is human-generated. Considerable anthropogenic sources are coal-fired power plants, steel, and nonferrous metal production, waste incineration plants or the chemical industry, e.g., with the chlor-alkali plants namely the still used mercury-cell process, which is used for the production of important base chemicals such as sodium hydroxide and chlorine. Also not negligible is the contribution of artisanal gold mining where the use of elemental mercury for the extraction of gold from the ore is still common. Once in the biosphere, mercury is highly mobile and can accumulate in organisms. The WHO (World Health Organization) guideline value for inorganic mercury is 6 µg/L, but the limit value for drinking water in many countries is much lower, e.g., USA 2 µg/L, European Union and Switzerland 1 µg/L.

This Application Bulletin describes the determination of inorganic mercury in water samples by anodic stripping voltammetry using the scTRACE Gold sensor. With a deposition time of 60 s, the limit of detection is $\beta(\text{Hg}^{2+}) = 0.1 \text{ µg/L}$; the calibration is linear up to a concentration of $\beta(\text{Hg}^{2+}) = 15 \text{ µg/L}$.

Samples

Surface water, ground water, waste water

Instruments

797 VA Computrace	2.797.0020
<i>Accessories</i>	
Stirrer	6.1204.200
Driving belt	6.1244.020
Measuring vessel 5 mL	6.1415.150
SGJ Stopper B-14	6.1446.000
Cap	6.2753.210

Electrodes

scTRACE Gold	6.1258.000
Electrode shaft	6.1241.080

Note! It is recommended to have a separate measuring vessel and stirrer for this application which have not been used in combination with a mercury or platinum electrode. It is also not recommended to use the same sensor for the determination of arsenic according to Application Bulletin 416.

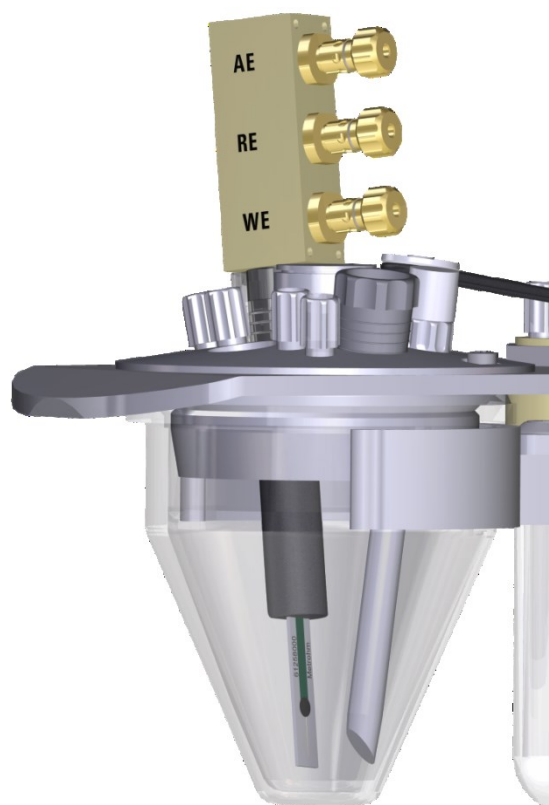


Fig. 1 scTRACE Gold in a voltammetric measuring cell

The scTRACE Gold

The scTRACE Gold sensor holds all three electrodes required for a voltammetric determination. Together with the electrode shaft, it makes a complete electrode system which can be used in any Metrohm voltammetric measuring stand. No further electrodes are required. The working electrode is a gold microwire. Reference and auxiliary electrode are screen-printed electrodes.

Different from other solid state electrodes, the scTRACE Gold does not need extensive conditioning before it can be used. A new sensor only needs to be activated as described in paragraph «Conditioning of the scTRACE Gold». This takes about 20 minutes and the sensor is ready for the first determination.

Due to its construction the scTRACE Gold is maintenance-free. It can be electrochemically cleaned as described in paragraph «Conditioning of the scTRACE Gold», but mechanical cleaning is neither necessary nor possible. As any electrode, the performance of the scTRACE Gold will deteriorate with the number of determinations. Signals will get smaller and curves are less reproducible. Then, it is time to replace the sensor.

Reagents

- Hg standard stock solution, $\beta(\text{Hg}^{2+}) = 1 \text{ g/L}$, commercially available
- Fe standard stock solution $\beta(\text{Fe}^{3+}) = 1 \text{ g/L}$, commercially available
- Nitric acid, $w(\text{HNO}_3) = 65\%$, for trace analysis*, CAS 7697-37-2
- Formic acid, $w(\text{HCOOH}) = 98 - 100\%$, for trace analysis*, CAS 64-18-6
- Sodium chloride, NaCl, for trace analysis*, CAS 7647-14-5
- Ammonium hydroxide solution, $w(\text{NH}_3) = 25\%$, for trace analysis*, CAS 1336-21-6
- Ultrapure water, resistivity $>18 \text{ M}\Omega \cdot \text{cm}$ (25 °C), type I grade (ASTM D1193)

In addition, if UV digestion is required:

- Sulfuric acid, $w(\text{H}_2\text{SO}_4) = 96\%$, for trace analysis*, CAS 7664-93-9
- Hydrogen peroxide solution, $w(\text{H}_2\text{O}_2) = 30\%$, for trace analysis*, CAS 7722-84-1

* e.g., Merck suprapur®, Sigma-Aldrich TraceSelect® or equivalent

Solutions

Conditioning solution	$w(\text{NH}_3) = 2.5\%$ 5 mL ammonium hydroxide solution is made up to 50 mL with ultrapure water.
Electrolyte	$w(\text{HCOOH}) = 3\%$ $c(\text{NaCl}) = 0.06 \text{ mol/L}$ $\beta(\text{Fe}^{3+}) = 1 \text{ mg/L}$ 0.175 g sodium chloride is

dissolved in approx. 40 mL ultrapure water in a 50 mL volumetric flask. 1.5 mL formic acid and 0.05 mL Fe standard stock solution are added. The mixture is made up to the mark with ultrapure water.

Electrolyte for preliminary tests

$w(\text{HCOOH}) = 3\%$
 $c(\text{NaCl}) = 0.06 \text{ mol/L}$
0.175 g sodium chloride is dissolved in approx. 40 mL ultrapure water in a 50 mL volumetric flask. 1.5 mL formic acid is added. The mixture is made up to the mark with ultrapure water.

Standard solutions

Hg standard solution 10 mg/L

$\beta(\text{Hg}^{2+}) = 10 \text{ mg/L}$
Approx. 5 mL ultrapure water is filled into a 10 mL volumetric flask. 0.01 mL $w(\text{HNO}_3) = 65\%$ and 0.1 mL Hg standard stock solution is added. The solution is made up to the mark with ultrapure water.

Hg standard solution 1 mg/L

$\beta(\text{Hg}^{2+}) = 1 \text{ mg/L}$
Approx. 40 mL ultrapure water is filled into a 50 mL volumetric flask. 0.05 mL $w(\text{HNO}_3) = 65\%$ and 0.05 mL Hg standard stock solution is added. The solution is made up to the mark with ultrapure water.

Hg standard solution 0.1 mg/L

$\beta(\text{Hg}^{2+}) = 0.1 \text{ mg/L}$
0.5 mL Hg standard 10 mg/L and 0.05 mL $w(\text{HNO}_3) = 65\%$ are made up to 50 mL in a volumetric flask with ultrapure water.

Sample preparation

- Ground water, drinking water, sea water, and mineral water can usually be analyzed directly.
- Water that is polluted with organic substances is digested using the 909 UV Digester:
10 mL acidified water sample ($\text{pH} = 2$) with $10 \mu\text{L}$ $w(\text{H}_2\text{SO}_4) = 96\%$ and $50 \mu\text{L}$ $w(\text{H}_2\text{O}_2) = 30\%$ is irradiated for 90 min at 90 °C.

Conditioning of the scTRACE Gold

Prior to use, the scTRACE Gold has to be activated and conditioned. This applies to a new electrode as well as to an electrode that has not been used for more than 1 hour. The electrode is first conditioned in diluted ammonium hydroxide solution. After the contact with the alkaline conditioning solution, some dummy sweeps in the determination electrolyte are required to make the electrode ready for use.

The same procedure can also be used to clean the sensor in between determinations.

Conditioning

10 mL ultrapure water and 1 mL conditioning solution are pipetted into the measuring vessel. The conditioning is carried out using the parameters given under «Parameters for conditioning». The objective is that the curve shape only changes very little within a series of 5 replications.

Measuring solution for conditioning

10 mL ultrapure water

1 mL conditioning solution

Parameters for conditioning

Determination	
No. of additions	1
No. of replications	5
Voltammetric	
Electrode	RDE/SSE
Measuring mode	DC – Sampled direct current
Stirring speed	2000 min ⁻¹
Hydrodynamic measurement	No
Conditioning cycles	
Start potential	-0.3 V
End potential	0.7 V
No. of cycles	20
Pretreatment	
Cleaning potential	0 V
Cleaning time	0 s
Deposition potential	0 V
Deposition time	0 s
Sweep	

Equilibration time	5 s
Start potential	0.1V
End potential	0.55 V
Potential step	0.006 V
Potential step time	0.2 s
Sweep rate	0.03 V/s

Example curves for conditioning

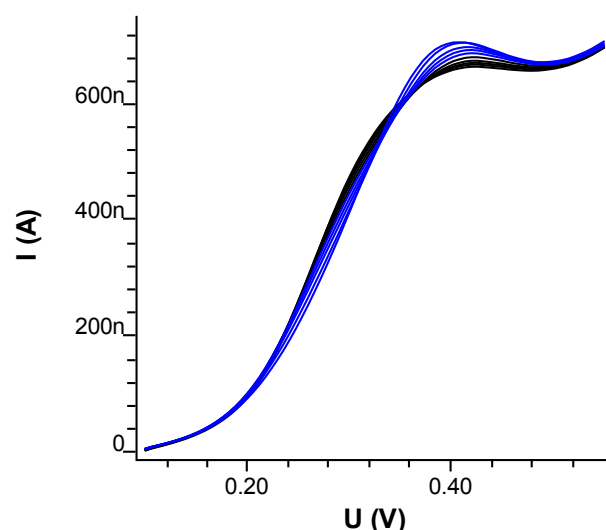


Fig. 2 Example for the conditioning sweeps of a new scTRACE Gold. Blue – first series of 5 sweeps; black – second series of five sweeps

Dummy sweeps

10 mL ultrapure water, 1 mL electrolyte and 0.1 mL Hg standard solution (1 mg/L) are pipetted into the measuring vessel. The dummy sweeps are carried out using the same parameters as for a determination.

Measuring solution for dummy sweeps

10 mL ultrapure water

1 mL electrolyte

0.1 mL Hg standard solution 1 mg/L

Parameters for dummy sweeps

See «Parameters for determination of mercury» in method 1.

Determination	
No. of additions	0
No. of replications	2

Example curve for dummy sweeps

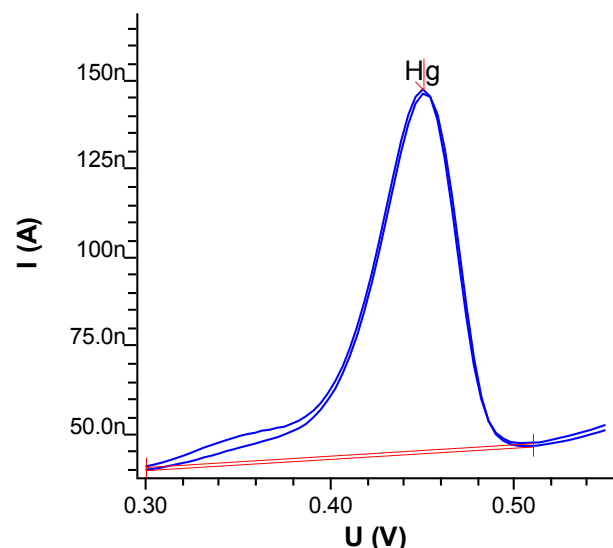


Fig. 3 Example for the curves obtained from the dummy sweeps

Comments

- If the electrode should be cleaned in between two determinations usually 5 sweeps in ammonium hydroxide solution are sufficient.
- After conditioning in ammonium hydroxide solution, it is always necessary to run some dummy sweeps in the determination electrolyte.

Method 1: Determination of mercury with the scTRACE Gold

Analysis

10 mL (diluted) sample and 1 mL electrolyte are pipetted into the measuring vessel. The determination is carried out using the parameters given under «Parameters for determination of mercury».

The concentration of mercury is quantified by two additions of Hg standard solution.

Measuring solution

10 mL (diluted) sample

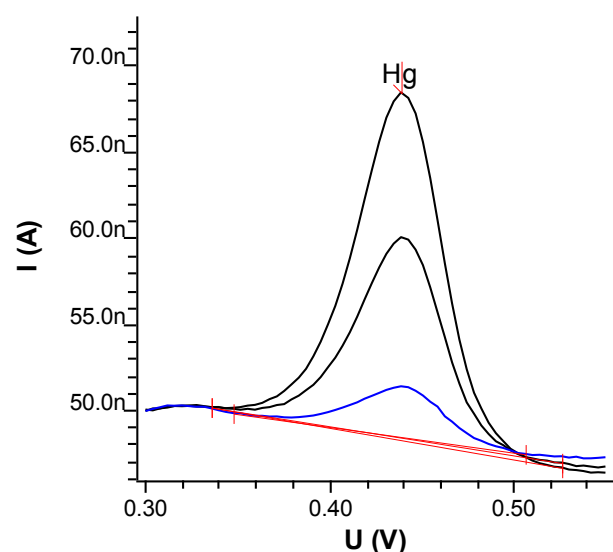
1 mL electrolyte

Parameters for determination of mercury

Determination	
No. of additions	2
No. of replications	1
Voltammetric	
Electrode	RDE/SSE
Measuring mode	DP – Differential Pulse
Stirring speed	2000 min ⁻¹
Hydrodynamic measurement	No
Conditioning cycles	
Start potential	0.3 V
End potential	0.75 V
No. of cycles	10
Pretreatment	
Cleaning potential	0 V
Cleaning time	0 s
Deposition potential	0.3 V
Deposition time	60 s
Sweep	
Equilibration time	5 s
Start potential	0.3 V
End potential	0.55 V
Pulse amplitude	0.05 V
Pulse time	0.03 s
Potential step	0.004 V
Potential step time	0.4 s
Sweep rate	0.01 V/s

Substance + calibration	
Calibration	Standard addition
Name	Hg
Peak potential	0.44 V
Tolerance	0.05 V
Baseline	Linear Automatic

Example for Hg determination in tap water



Hg
c = 0.332 µg/L
+/- 0.001 µg/L (0.36%)

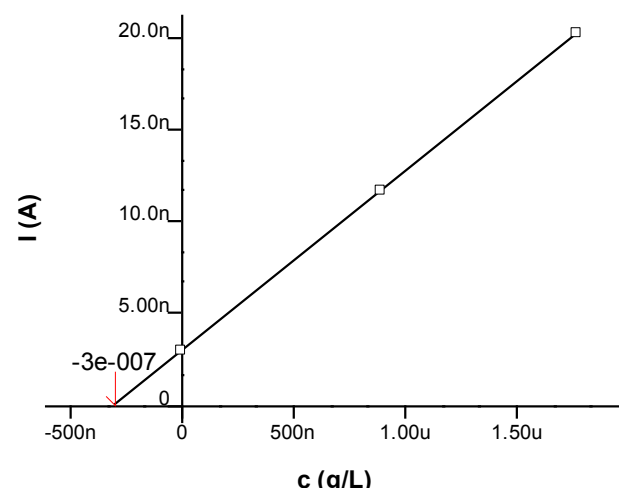


Fig. 4 Determination of mercury in tap water (spiked sample)

Results

Sample	$\beta(\text{Hg})$
Tap water spiked	0.33 µg/L
Reagent blank	0.16 µg/L

Recovery of spiked amount $\beta(\text{Hg}) = 0.2 \mu\text{g/L}$	85%
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Limit of detection and linear working range

The limit of detection was determined using the «regression approach» [1], where the limit of detection is calculated as « $3 \cdot s_y$ », with s_y as the residual standard deviation of a linear regression. The limit of quantification is calculated with « $10 \cdot s_y$ ».

The linear working range was read out from a calibration curve.

The following table gives an overview over limit of detection and linear working range depending on the time of deposition.

Deposition	Limit of detection	Linear working range
30 s	0.2 µg/L	30 µg/L
60 s	0.1 µg/L	15 µg/L
90 s	0.07 µg/L	10 µg/L

The limit of detection and linear working range were determined in standard solutions with a new sensor. Depending on the condition of the electrode or the sample matrix, these values can differ.

Interferences

Anions

The influence on the determination of $\beta(\text{Hg}) = 1 \mu\text{g/L}$ was tested. If nothing else is mentioned the interference was examined up to a concentration of 1 mg/L (equals 1000 fold excess). The sensitivity mentioned in the table below corresponds to the slope of the standard addition curve.

Cl^-	Chloride facilitates the oxidation of the gold electrode. Besides, it has an influence on the potential of the reference electrode. For concentrations $c(\text{Cl}^-) > 100 \text{ mmol/L}$, adapted method parameters should be used. Refer to «Method 2: Determination of mercury in sea water or other chloride-containing samples».
Br^-	Shifts the Hg peak up to 50 mV into cathodic direction. A ratio of $\text{Br}^-:\text{Hg}$ of 1000:1 reduces the sensitivity by approx. 20%.

I^-	Peak at approx. 0.55 V. Shifts the Hg peak up to 200 mV into cathodic direction. Higher concentrations shift the peak back into anodic direction due to the influence on the reference electrode potential. Determination in the presence of $\beta(I^-) = 10 \mu\text{g/L}$ possible but strong influence on the shape of the background current.
NO_2^-	Broad peak at approx. 0.45 V. A ratio of NO_2^- :Hg of 4000:1 does not interfere. With higher excess the NO_2^- peak overlaps with the Hg peak.
NO_3^-	A ratio of NO_3^- :Hg of 10^6 :1 does not interfere.
SO_4^{2-}	A ratio of SO_4^{2-} :Hg of 10^5 :1 does not interfere.
PO_4^{3-}	A ratio of PO_4^{3-} :Hg of 10^5 :1 does not interfere.

Cations

The influence on the determination of $\beta(\text{Hg}) = 1 \mu\text{g/L}$ was tested. If nothing else is mentioned the interference was examined up to a concentration of 1 mg/L (equals 1000 time excess). The sensitivity mentioned in the table below corresponds to the slope of the standard addition curve.

Ag^+	Does not interfere up to a concentration of $\beta(Ag) = 10 \mu\text{g/L}$ in the measuring cell. Higher concentrations alter the working electrode due to the formation of a gold-silver alloy which inhibits the determination of mercury.
As^{3+}	Does not interfere.
As^{5+}	Does not interfere.
Bi^{3+}	Sharp peak at approx. 0 V. A ratio of Bi^{3+} :Hg of 1000:1 reduces the sensitivity by approx. 40%.
Ca^{2+}	Does not interfere. A ratio of Ca^{2+} :Hg of $5 \cdot 10^5$:1 reduces the sensitivity by approx. 20%.
Cd^{2+}	Does not interfere.
Co^{2+}	Does not interfere.
Cu^{2+}	Peak at approx. 0.15 V. Does not interfere.

Cr^{3+}	Does not interfere.
Cr^{6+}	Broad peak at approx. 0.14 V. Does not interfere.
Fe^{2+}	Peak at approx. 0.3 V. A ratio of Fe^{2+} :Hg of 1000:1 reduces the recovery by approx. 20% due to an overlapping with the Hg peak.
Fe^{3+}	Peak at approx. 0.3 V. A ratio of Fe^{3+} :Hg of 1000:1 reduces the recovery by approx. 20% due to an overlapping with the Hg peak.
Mg^{2+}	Does not interfere. A ratio of Mg^{2+} :Hg of $5 \cdot 10^5$:1 reduces the sensitivity by approx. 25%.
Mn^{2+}	Does not interfere.
Ni^{2+}	Does not interfere.
Pb^{2+}	Does not interfere. A ratio of Pb^{2+} :Hg of 1000:1 reduces the sensitivity by approx. 20%.
Sb^{3+}	Broad peak at approx. 0.25 V. A ratio of Sb^{3+} :Hg of 10:1 reduces the sensitivity by approx. 30%. Higher excess inhibits the determination of mercury.
Sb^{5+}	Does not interfere.
Se^{4+}	Peak at approx. 0.55 V. Does not interfere. Solubility product $K_{sp}(\text{HgSeO}_3) = 1.5 \cdot 10^{-14}$.
Se^{6+}	Does not interfere.
Sn^{2+}	Does not interfere.
Sn^{4+}	Does not interfere up to a ratio of Sn^{4+} :Hg of 100:1. Higher concentrations deteriorate the shape of the background current.
Zn^{2+}	Does not interfere.

Miscellaneous

The influence on the determination of $\beta(\text{Hg}) = 1 \mu\text{g/L}$ was tested. If nothing else is mentioned the interference was examined up to a concentration of 1 mg/L (equals 1000 time excess). The sensitivity mentioned in the table below corresponds to the slope of the standard addition curve.

O_2	Peak at approx. -0.25 V. Not quantified. Does not interfere.
EDTA	Peak at approx. 0.55 V. A ratio of EDTA:Hg of 1000:1 does not interfere. Higher excess can

	completely cover the Hg peak.
Humic acids	Do not interfere. A ratio of humic acid:Hg of 1000:1 shows a slightly reduced sensitivity (approx. 10%).
Triton X-100	Does not interfere. A ratio of Triton X-100:Hg of 1000:1 shows a slightly reduced sensitivity (approx. 10%).

Comments

Electrolyte

The iron in the electrolyte is used as a baseline modifier. Usually the Hg peak is placed on a hump which makes it difficult to detect the start of the Hg peak. The presence of $\beta(\text{Fe}) = 100 \mu\text{g/L}$ causes a peak right in front of the Hg peak which facilitates the evaluation of the Hg peak but does not interfere with the determination. In general, the application also works without iron in the electrolyte.

Reagent blank

Since formic acid as well as sodium chloride can contain traces of mercury, it is recommended to determine a reagent blank on a regular basis.

Measuring solution for the reagent blank:

10 mL ultrapure water + 1 mL electrolyte

Measuring parameters are the same as those used for the mercury determination.

Electrode storage

When the electrode is not used for a short period of time (e.g., overnight), it can be stored in slightly acidified ultrapure water ($\sigma(\text{HNO}_3) = 0.1 \text{ mL/L}$). When the electrode should not be used for a longer time, it should be thoroughly rinsed and stored dry.

Measurement artefact

After a few determinations, a measurement artefact can appear, which looks like a blank, but with a 10 to 20 mV more negative peak potential. The occurrence of the measurement artefact is usually an indication, that the end potential of the cleaning cycles is too positive.

Standard solution

Standard solutions with concentrations of less than $\beta(\text{Hg}^{2+}) = 0.1 \text{ mg/L}$ are not very stable and should be daily prepared.

Automation

If Dosinos are used for automatic addition of electrolyte and standard, those parts of the Dosing Unit that get in contact with the solution have to be completely free of grease. If the Dosing Unit had been greased it can be cleaned with n-hexane. For cleaning, the Dosing Unit has to be disassembled and completely dried. Then, piston, glass dosing cylinder, cylinder plate, valve, and distributor disk and the four ports of the distributor can be cleaned with n-hexane. Before reassembling, the parts have to be dried again. For disassembling and assembling of the Dosing Unit, please refer to the manual of the 807 Dosing Unit.

Method 2: Determination of mercury in sea water or other chloride-containing samples

Chloride facilitates the oxidation of the gold electrode. Besides that, higher concentrations have an influence on the potential of the reference electrode. Therefore, the gold electrode would be damaged if samples such as sea water which contains approx. 0.5 mol/L chloride were analyzed using the parameters given in method 1.

Analysis

10 mL (diluted) sample and 1 mL electrolyte are pipetted into the measuring vessel. The determination is carried out using the parameters given under «Parameters for chloride-containing samples».

The concentration of mercury is quantified by two additions of Hg standard solution.

Measuring solution

10 mL (diluted) sample

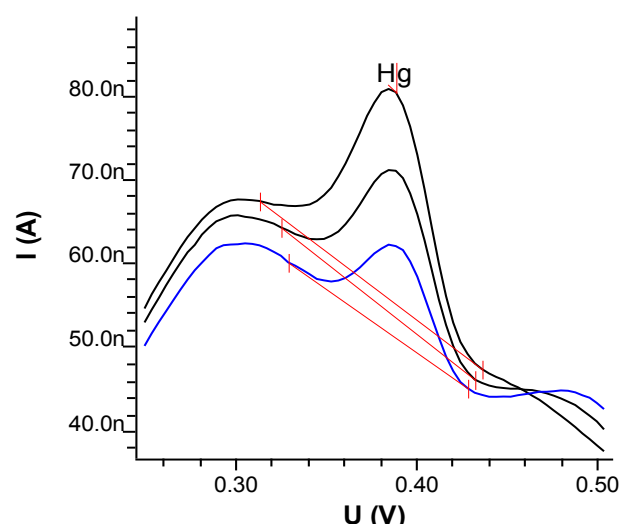
1 mL electrolyte

Parameters for chloride-containing samples

Determination	
No. of additions	2
No. of replications	1
Voltammetric	
Electrode	RDE/SSE
Measuring mode	DP – Differential Pulse
Stirring speed	2000 min ⁻¹
Hydrodynamic measurement	No
Conditioning cycles	
Start potential	0.25 V
End potential	0.7 V
No. of cycles	10
Pretreatment	
Cleaning potential	0 V
Cleaning time	0 s
Deposition potential	0.25 V
Deposition time	60 s
Sweep	
Equilibration time	5 s

Start potential	0.25 V
End potential	0.5 V
Pulse amplitude	0.05 V
Pulse time	0.03 s
Potential step	0.004 V
Potential step time	0.4 s
Sweep rate	0.01 V/s
Substance + calibration	
Calibration	Standard addition
Name	Hg
Peak potential	0.4 V
Tolerance	0.05 V
Baseline	Linear Automatic

Example for Hg determination in sea water



Hg
c = 1.519 µg/L
+/- 0.264 µg/L (17.36%)

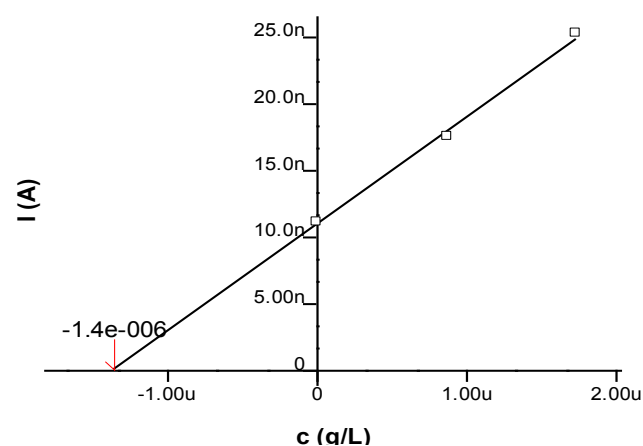


Fig. 5 Determination of mercury in artificial sea water (spiked sample)

Results

Sample	$\beta(\text{Hg})$
Sea water spiked	1.52 $\mu\text{g/L}$
Recovery of spiked amount $\beta(\text{Hg}) = 2 \mu\text{g/L}$	76%

Comments

Sensitivity

The sensitivity of the determination in samples with high chloride content is significantly lower than of samples with low chloride content. In artificial sea water, the sensitivity, represented by the slope of the standard addition curve, was reduced by more than 50% compared to a standard solution.

Composition of artificial sea water

The artificial sea water was prepared according to Römpf [2] with:

28 g/L NaCl
7 g/L $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$
5 g/L $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$
2.4 g/L $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$
0.2 g/L NaHCO_3
pH 7.8 ... 8.2

Adaption of method parameters to an unknown sample matrix

Halides, pseudo-halides and other substances forming complexes with gold can facilitate the oxidation and therefore the degradation of the working electrode. Since the electrode surface cannot be renewed, this usually means the electrode has to be replaced. Also the potential of the reference electrode can be influenced by the sample matrix since it is an electrode of the first kind.

To avoid damage to the electrode and to ensure that the mercury signal is found within the potential window, it can be useful to do some preliminary tests, if the sample matrix is not known in detail.

For the preliminary tests, use the electrolyte without iron «Electrolyte for preliminary tests».

Analysis

10 mL (diluted) sample and 1 mL electrolyte for preliminary tests are pipetted into the measuring vessel. Run a sweep using the parameters given under «Parameters for preliminary tests». Manually stop the sweep when the current exceeds 50 nA.

Add 0.1 mL $\beta(\text{Hg}^{2+}) = 10 \text{ mg/L}$ and repeat the sweep to identify the position of the Hg peak.

Measuring solution for preliminary tests

10 mL (diluted) sample

1 mL electrolyte for preliminary tests

Parameters for preliminary tests

Voltammetric	
Electrode	RDE/SSE
Measuring mode	DC – Sampled Direct Current
Stirring speed	2000 min^{-1}
Hydrodynamic measurement	No
Conditioning cycles	No
Pretreatment	No
Sweep	
Equilibration time	5 s
Start potential	0.0 V
End potential	0.8 V

Potential step	0.004 V
Potential step time	0.4 s
Sweep rate	0.01 V/s

Example for preliminary tests

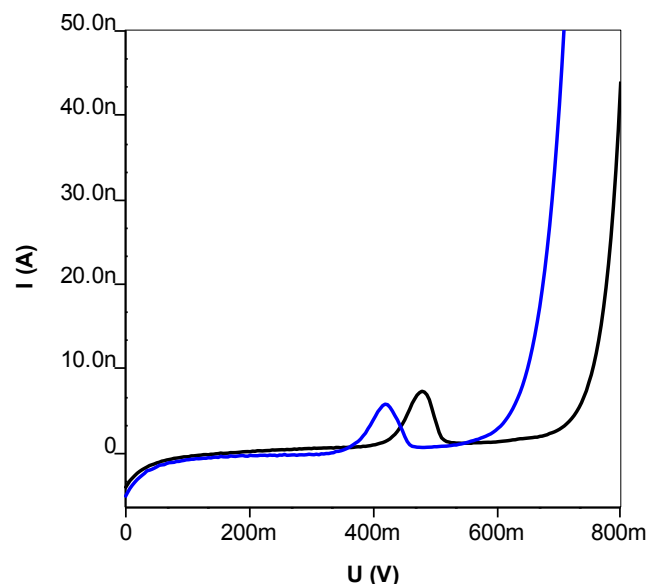


Fig. 6 Black line – curve in standard solution (Hg peak at 0.48 V, oxidation of gold starts at approx. 0.7 V); blue line – curve in artificial sea water (Hg peak at 0.42 V, oxidation of gold starts at approx. 0.6 V)

Adjustment of method parameters

Conditioning cycles

Chose a start potential which ensures that the potential of the Hg peak will be included in the conditioning cycles. Chose an end potential which ensures that the background current increase has started, but the current does not exceed 50 nA.

Deposition potential

Chose a deposition potential 150 to 200 mV more negative than the peak potential of the Hg peak.

Sweep

Chose start and end potentials which make sure that the Hg peak is well inside the potential interval.

References

- [1] J. Mocak, A. M. Bond, S. Mitchell, and G. Scollary, "A statistical overview of standard (IUPAC and ACS) and new procedures for determining the limits of detection and quantification: Application to voltammetric and stripping techniques," *Pure and Applied Chemistry*, vol. 69, no. 2, pp. 297-328, 1997.
- [2] Römpp. (1995) Chemie Lexikon (Version 1.0). CD.

Appendix

Method print «Conditioning of the scTRACE Gold»

Method parameters	
Method	: AB422 Conditioning scTRACE Gold in NH3-solution.mth
Title	: Conditioning scTRACE Gold in NH3-solution
Remark1	: 10 mL H2O + 1 mL w(NH3) = 2.5%
Remark2	:
Calibration	: Standard addition
Technique	: Batch
Addition	: Manual
Sample ID	: Conditioning scTRACE
Sample amount (mL)	: 10.000
Cell volume (mL)	: 11.000
Voltammetric parameters	
Mode	: DC - Sampled Direct Current
Highest current range	: 10 mA
Lowest current range	: 100 nA
Electrode	: SSE/RDE
Stirrer speed (rpm)	: 2000
Initial electr. conditioning	: No
No. of additions	: 1
No. of replications	: 5
Measure blank	: No
Addition purge time (s)	: 10
Initial purge time (s)	: 10
Conditioning cycles	
Start potential (V)	: -0.300
End potential (V)	: 0.700
No. of cycles	: 20
Hydrodynamic (measurement)	: No
Cleaning potential (V)	: 0.000
Cleaning time (s)	: 0.000
Deposition potential (V)	: 0.000
Deposition time (s)	: 0.000
Sweep	
Equilibration time (s)	: 5.000
Start potential (V)	: 0.100
End potential (V)	: 0.550
Voltage step (V)	: 0.006
Voltage step time (s)	: 0.200
Sweep rate (V/s)	: 0.030
Cell off after measurement	: Yes
Peak evaluation	
Regression technique	: Linear Regression
Peak evaluation	: Height
Minimum peak width (V.steps)	: 5
Minimum peak height (A)	: 1.000e-010
Reverse peaks	: No
Smooth factor	: 4
Eliminate spikes	: Yes
Substances	
Baseline	
Substance Addition	automatic start (V) end (V) type scope

Method print «Method 1: Determination of mercury with the scTRACE Gold»

Method parameters						

Method	: AB422_1 Determination Hg with scTRACE.mth					
Title	: Determination of Hg with scTRACE					
Remark1	: 10 mL H2O + 1 mL electrolyte					
Remark2	: Electrolyte: c(CH2O2)=0.78mol/L; c(NaCl)=0.06mol/L, c(Fe3)=1mg/L					
Calibration	: Standard addition					
Technique	: Batch					
Addition	: Manual					
Sample ID	: sample					
Sample amount (mL)	: 10.000					
Cell volume (mL)	: 11.000					
Voltammetric parameters						

Mode	: DP - Differential Pulse					
Highest current range	: 10 mA					
Lowest current range	: 100 nA					
Electrode	: SSE/RDE					
Stirrer speed (rpm)	: 2000					
Initial electr. conditioning	: No					
No. of additions	: 2					
No. of replications	: 1					
Measure blank	: No					
Addition purge time (s)	: 10					
Initial purge time (s)	: 10					
Conditioning cycles	: 0.300					
Start potential (V)	: 0.750					
End potential (V)	: 10					
No. of cycles	: No					
Hydrodynamic (measurement)	: 0.000					
Cleaning potential (V)	: 0.000					
Cleaning time (s)	: 0.300					
Deposition potential (V)	: 60.000					
Deposition time (s)	: 5.000					
Sweep	: 0.300					
Equilibration time (s)	: 0.550					
Start potential (V)	: 0.004					
End potential (V)	: 0.400					
Voltage step (V)	: 0.010					
Voltage step time (s)	: 0.050					
Sweep rate (V/s)	: 0.030					
Pulse amplitude (V)	: Yes					
Pulse time (s)	: Yes					
Cell off after measurement	: Yes					
Peak evaluation						

Regression technique	: Linear Regression					
Peak evaluation	: Height					
Minimum peak width (V.steps)	: 5					
Minimum peak height (A)	: 1.000e-010					
Reverse peaks	: No					
Smooth factor	: 4					
Eliminate spikes	: Yes					
Substances						

Hg	: 0.440 V +/- 0.050 V					
Standard solution	: 1 0.100 mg/L					
Addition volume (mL)	: 0.100					
Mercury	: Final result (Hg) = Conc * (11 / 10) * (1e+006 / 1) + 0 - 0					
Baseline						

Substance	Addition	automatic	start (V)	end (V)	type	scope

Hg	Sample	yes	---	---	linear	wholePeak
	Addition 1	yes	---	---	linear	wholePeak
	Addition 2	yes	---	---	linear	wholePeak

Method print «Method 2: Determination of mercury in sea water or other chloride-containing samples»

Method parameters						

Method	: AB422_2_Determination Hg with scTRACE in sea water.mth					
Title	: Determination of Hg with scTRACE in sea water					
Remark1	: 10 mL H2O + 1 mL electrolyte					
Remark2	: Electrolyte: c(CH2O2)=0.78mol/L; c(NaCl)=0.06mol/L, c(Fe3)=1mg/L					
Calibration	: Standard addition					
Technique	: Batch					
Addition	: Manual					
Sample ID	: sample					
Sample amount (mL)	: 10.000					
Cell volume (mL)	: 11.000					
Voltammetric parameters						

Mode	: DP - Differential Pulse					
Highest current range	: 10 mA					
Lowest current range	: 100 nA					
Electrode	: SSE/RDE					
Stirrer speed (rpm)	: 2000					
Initial electr. conditioning	: No					
No. of additions	: 2					
No. of replications	: 1					
Measure blank	: No					
Addition purge time (s)	: 10					
Initial purge time (s)	: 10					
Conditioning cycles	: 0.250					
Start potential (V)	: 0.700					
End potential (V)	: 10					
No. of cycles	: No					
Hydrodynamic (measurement)	: 0.000					
Cleaning potential (V)	: 0.000					
Cleaning time (s)	: 0.250					
Deposition potential (V)	: 60.000					
Deposition time (s)	: 5.000					
Sweep	: 0.250					
Equilibration time (s)	: 0.500					
Start potential (V)	: 0.004					
End potential (V)	: 0.400					
Voltage step (V)	: 0.010					
Voltage step time (s)	: 0.050					
Sweep rate (V/s)	: 0.030					
Pulse amplitude (V)	: Yes					
Pulse time (s)	: Yes					
Cell off after measurement	: Yes					
Peak evaluation						

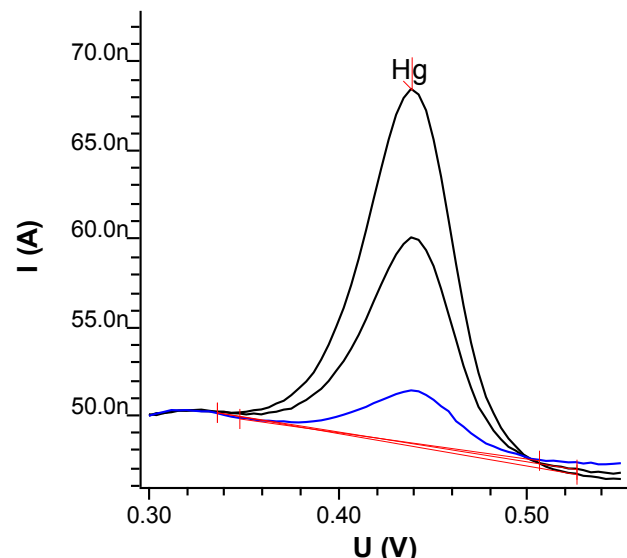
Regression technique	: Linear Regression					
Peak evaluation	: Height					
Minimum peak width (V.steps)	: 5					
Minimum peak height (A)	: 1.000e-010					
Reverse peaks	: No					
Smooth factor	: 4					
Eliminate spikes	: Yes					
Substances						

Hg	: 0.400 V +/- 0.050 V					
Standard solution	: 1 0.100 mg/L					
Addition volume (mL)	: 0.100					
Mercury	: Final result (Hg) = Conc * (11 / 10) * (1e+006 / 1) + 0 - 0					
Baseline						

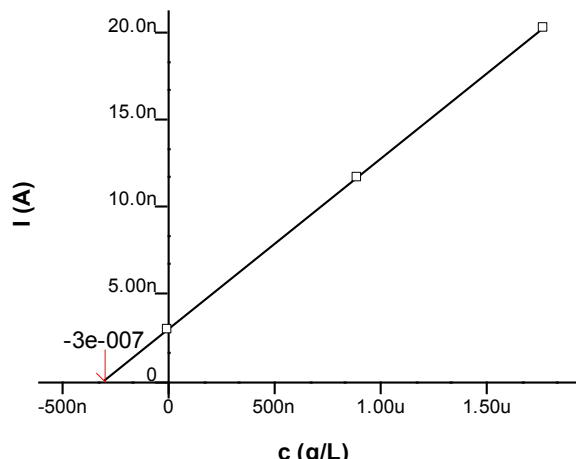
Substance	Addition	automatic	start (V)	end (V)	type	scope

Hg	Sample	yes	---	---	linear	wholePeak
	Addition 1	yes	---	---	linear	wholePeak
	Addition 2	yes	---	---	linear	wholePeak

Report «Example for Hg determination in tap water»



Hg
c = 0.332 ug/L
+/- 0.001 ug/L (0.36%)



===== METROHM 797 VA COMPUTRACE (Version 1.3.2.85) (Serial No. 9161) =====
Determination : 1404160919_Tap water spiked 0.2ppb Hg.dth
Sample ID : Tap water spiked 0.2ppb Hg
Creator method : zu Date : 2014-04-01 Time: 15:58:03
Creator determ.: zu Date : 2014-04-16 Time: 09:31:43
Modified by : zu Date : 2014-04-24 Time: 13:55:39

Method : Hg Recovery scTRACE Gold Dosino.mth
Title : Determination of Hg with scTRACE
Remark1 : 10 mL H2O + 1 mL electrolyte
Remark2 : Electrolyte: c(CH2O2)=0.78mol/L; c(NaCl)=0.06mol/L, c(Fe3)=1mg/L

Sample amount : 10.000 mL
Cell volume : 11.020 mL

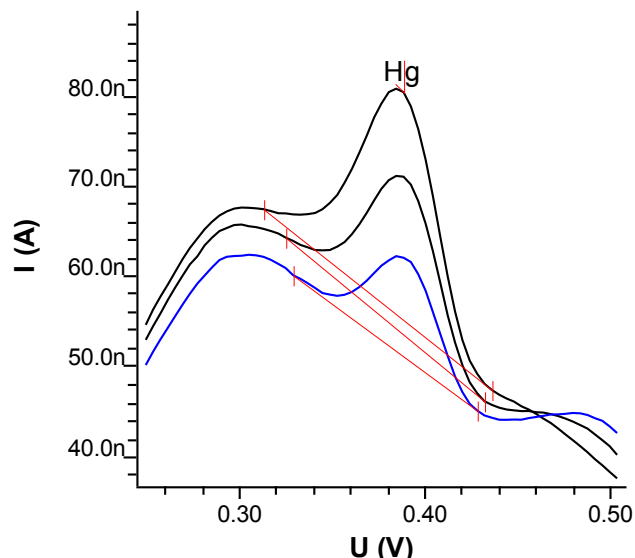
Substance : Hg
Conc. : 300.931 ng/L
Conc.dev. : 1.071 ng/L (0.36%)
Amount : 3.316 ng
Add.amount : 10.000 ng

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	0.443	2.92	2.92	---	0.00	
2 - 1	0.439	11.61	11.61	---	8.69	
3 - 1	0.439	20.16	20.16	---	8.55	

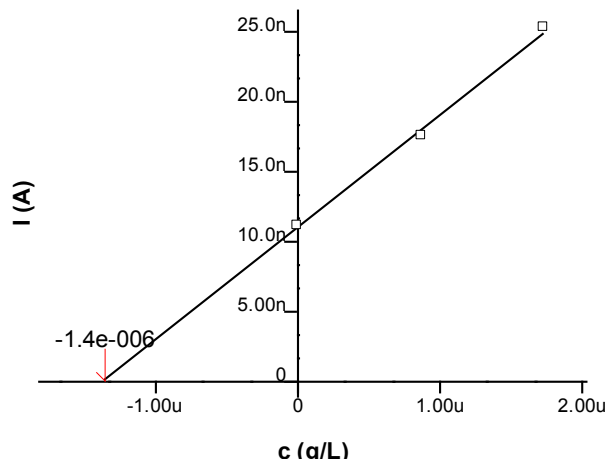
Substance	Calibr.	Y.reg/offset	Slope	Mean deviat.	Corr.Coeff.
Hg	std.add.	2.920e-009	9.702e-003	5.388e-012	1.00000

Final results	+/-	Res. dev.	%	Comments
Hg:				
Mercury	=	0.332 ug/L	0.001	0.356
Blank corrected	=	0.272 ug/L	0.001	0.435

Report «Example for Hg determination in sea water»



Hg
c = 1.519 ug/L
+/- 0.264 ug/L (17.36%)



```
===== METROHM 797 VA COMPUTRACE (Version 1.3.2.85) (Serial No. 9161) =====
Determination : 1404161348 Sea water Römpp spike 2ppb Hg.dth
Sample ID : Sea water Römpp spike 2ppb Hg
Creator method : zu Date : 2014-04-16 Time: 13:32:24
Creator determ.: zu Date : 2014-04-16 Time: 13:48:41
Modified by : --- Date : Time:
```

```
-----
Method : Hg scTRACE Gold sea water.mth
Title : Determination of Hg with scTRACE in sea water
Remark1 : 10 mL sample + 1 mL electrolyte
Remark2 : Electrolyte: c(CH2O2)=0.78mol/L; c(NaCl)=0.06mol/L, c(Fe3)=1mg/L
```

```
-----
Sample amount : 10.000 mL
Cell volume : 11.200 mL
```

```
-----
Substance : Hg
Conc. : 1.357 ug/L
Conc.dev. : 0.236 ug/L (17.36%)
Amount : 15.195 ng
Add.amount : 10.000 ng
```

VR	V	nA	I.mean	Std.Dev.	I.delta	Comments
1 - 1	0.393	11.07	11.07	---	0.00	
2 - 1	0.389	17.52	17.52	---	6.45	
3 - 1	0.389	25.29	25.29	---	7.77	

Substance	Calibr.	Y.reg/offset	Slope	Mean deviat.	Corr.Coeff.
Hg	std.add.	1.092e-008	8.048e-003	5.701e-010	0.99836

Final results	+/-	Res. dev.	%	Comments
---------------	-----	-----------	---	----------

Hg:				
Mercury	=	1.519 ug/L	0.264	17.363