

# Preconcentration of Hg in Waters for ET AAS in a Flow-Through Electrochemical Cell

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## Abstract

Mercury in water samples was determined by graphite furnace atomic absorption spectrometry after the preconcentration of Hg(II) on a gold working electrode in a flow system coupled on-line to the AAS instrument. The porous electrode is made of glassy carbon particles coated with gold. Tap, river, and wastewater samples were analyzed using this method. The method enables us to determine Hg in the concentration range of 0.05 to 60  $\mu\text{g}\cdot\text{dm}^{-3}$ .

**Keywords:** preconcentration of Hg, water samples, ET AAS, electrochemical cells

## Introduction

Wastewaters, e.g. from chloro-alkali electrolytic plants, may contain mercury and mercury compounds and therefore it is important to have reliable and simple methods to determine their content. There are few methods enabling the determination of traces of mercury [1-10]. Most of them are based on atomic absorption spectrometry and make use of the easy reduction and evaporation of mercury to its atomic form. These analyzers chemically convert the different mercury species present in the sample solutions to elementary mercury, which is then stripped by a gas to the absorption tube for measurements. The measurement step is simple but the sample preparation procedure may be rather tedious. For complex compounds the sample preparation may include a digestion of the sample to convert all

mercury compounds into Hg(II), which is then reduced to elementary mercury and is transported by a gas to the absorption cuvette. Mercury can be easily preconcentrated on gold electrodes, which are successfully used for its electrochemical determination [11-15]. This principle may significantly simplify the procedure owing to the fact that the reduction of Hg species and the determination can be done with the same electrode. The chemical reduction step can be omitted. The main advantages of the use of flow-through cell are simplicity, high speed, and low costs. However, like other methods, this method is also vulnerable to matrix interferences and electrode fouling. The aim of this paper is to show the utility of preconcentration determinations of Hg in waters by making use of a flow system coupled on-line to the AAS as measurement principle.

Mercury in water samples was determined by graphite furnace atomic absorption spectrometry after the preconcentration of Hg(II) on gold working electrode in a flow

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Table 1. Comparison of various parameters (preconcentration factor, sample volume, total time of analysis, and detection limit) preconcentration of Hg(II) in water samples by various methods from the literature.

Method	Volume (mL)	Time (min)	PF	LOD	Reference
ICP-MS	-	10	200	15.86 pg·mL <sup>-1</sup>	16
Visible spectrophotometry	1000	-	100	5 µg·L <sup>-1</sup>	17
CV AAS	-	-	25	2.1 ng·L <sup>-1</sup>	18
FI-CV-AAS	20	-	36	2.3 ng·L <sup>-1</sup>	19
CV AAS	1500	-	500	3.8 ng·L <sup>-1</sup>	20
Visible spectrophotometry	500	-	33	2 µg·L <sup>-1</sup>	21
Visible spectrophotometry	1000	-	100	4 µg·L <sup>-1</sup>	22
Spectrophotometry	-	-	152	10.92 ng·mL <sup>-1</sup>	23
CV AAS	-	10	120	-	24
Spectrophotometry	-	-	50	0.015 µg·mL <sup>-1</sup>	25
Spectrophotometry	-	-	25	0.2 µg·mL <sup>-1</sup>	26
ET AAS	8.5	3	14	0.27 µg·L <sup>-1</sup>	27
UV-visible and IR spectroscopy	-	10-20	100	-	28
UV-visible and IR spectroscopy	-	5-15	50	4 ng·mL <sup>-1</sup>	29
CVAAS	-	-	200	0.3 µg·L <sup>-1</sup>	30
UV-visible and IR spectroscopy	-	15-20	>50	-	31
ICP-MS	-	30	100	0.44 pg·mL <sup>-1</sup>	32
X-ray fluorescence	-	-	107	0.5 mg·L <sup>-1</sup>	33
CVAAS	-	-	100	7.8 ng·L <sup>-1</sup>	34
CVAAS	-	-	100	-	35

system coupled on-line to the AAS instrument. A flow-through electrochemical analyzer EcaFlow (model GLP 150, Istran, Slovakia) was used as the preconcentration unit with a two-electrode cell. The porous electrode was made of glassy carbon particles coated with gold. An AAS spectrometer (model AA 6300, Shimadzu, Japan) with graphite furnace atomizer was used to measure the preconcentrated Hg species. The preconcentration parameters for the electrochemical process were optimized. The deposition was run at a constant current of -3500 µA and the optimum stripping current was found to be 100 µA. The deposition was made from a hydrochloric acid solution, which was then on-line replaced by a diluted nitric acid facilitating the AAS measurement of Hg. The advantage of the used preconcentration system was in an automatic and reliable matrix exchange facility. Optimum electrolyte concentrations were found to be 0.1 mol·dm<sup>-3</sup> HCl and 0.1 mol·dm<sup>-3</sup> HNO<sub>3</sub> for the deposition and stripping/AAS measurement, respectively.

The trueness was determined by means of a certified reference material CRM DWPS with Hg content of 20.0±0.2 µg·dm<sup>-3</sup>. Detection limit and precision were found to be 53 ng·dm<sup>-3</sup> and 5.9%, respectively.

The comparison of various parameters (preconcentration factor, sample volume, total time of analysis and

detection limit) preconcentration of Hg(II) in water samples by various methods from the literature are shown in Table 1.

Tap, river, and wastewater samples were analyzed by this method. The method enables it to determine Hg in the concentration range of 0.05 to 60 µg·dm<sup>-3</sup>.

## Material and Methods

### Instrumentation

The flow system (Fig. 1) was constructed of PTFE tubing. The sampling valve was 6-way (Latek, Eppelheim, Germany). A peristaltic pump drove the liquids. The flow-through cell (FTC, Fig. 2) for preconcentration was prepared from a perspex body. The working electrode was prepared from reticulated vitreous carbon "RVC of 100 pores per inch porosity" (Electrosynthesis Co., East Amherst, NY, USA) crushed to particles of 10 to 100 µm size. The surface of the working electrode was plated with a thin layer of gold. For the detection of Hg a ET AAS instrument AA-6300 (Shimadzu, Japan) coupled on-line to the flow system was used.

## Reagents and Samples

Analytical-grade reagents and deionized, boiled, cooled water were used throughout the experiments. The carrier electrolyte solution contained  $0.1 \text{ mol}\cdot\text{dm}^{-3}$  of HCl or  $0.1 \text{ mol}\cdot\text{dm}^{-3}$  of  $\text{HNO}_3$ . Standard solution  $1.000 \text{ g}\cdot\text{dm}^{-3}$  of Hg was used. Wastewater samples were obtained from plant Grafia and from a chloro-alkali electrolytic plant.

## Procedure

The flow system and cell were purged with  $1 \text{ mg dm}^{-3}$  HCl solution for a few min. to remove possible contamination. The working electrode was brought at the deposition potential. A given volume of the sample solution filtered through a  $0.45 \mu\text{m}$  membrane filter and pumped through the system at a flow rate  $4 \text{ cm}^3\cdot\text{min}^{-1}$  and mixed with the carrier electrolyte. This solution passed through the cell where mercury in the sample was preconcentrated. The system was subsequently rinsed with the carrier electrolyte, the flow was stopped and the deposit was stripped at constant current. After this step the system was rinsed again with the carrier electrolyte. The next step included the ignited furnace of the AAS instrument and the preconcentrated Hg was eluted to the instrument by making use of the valve. The integrated signal was measured and the corresponding Hg concentration was calculated by means of a calibration plot. The cell was washed again with  $1 \text{ mol}\cdot\text{dm}^{-3}$  HCl to prepare the system for the next run.

## Results and Discussion

Mercury can easily be determined in various samples by ET AAS after the preconcentration of Hg(II) species on gold working electrode. However, the porous working electrode used in this technique may rapidly be clogged when analyzing wastewater with high colloidal particle content. Moreover, dissolved gases may be liberated from waters, which tend to form bubbles in the pores of the working electrode, decreasing its active surface and efficiency. Hence, for direct analysis of such samples an oxygen scavenger is desirable. Preliminary measurements have demonstrated the utility of a cell with a gold working electrode. To minimize the clogging of the electrode, a membrane filter was used for the samples inlet to the target cell. Most waters, especially wastewaters, contain organic matter, which may reduce mercury to its elementary form. However, elementary mercury cannot be preconcentrated electrochemically. To determine total Hg content, the sample should be treated first to transform all Hg species into an electrochemically active, ionic form. For most water samples (which do not contain reducing species), acidification with a mineral acid proved to be sufficient to liberate mercury from complex compounds. In the presence of reducing substances, the acidification caused poor recoveries, probably as the result of a rapid reduction of Hg species to elementary mercury. On the other hand, excellent results were obtained with the addition of an acidified potassium permanganate solution. This is probably due to the oxida-

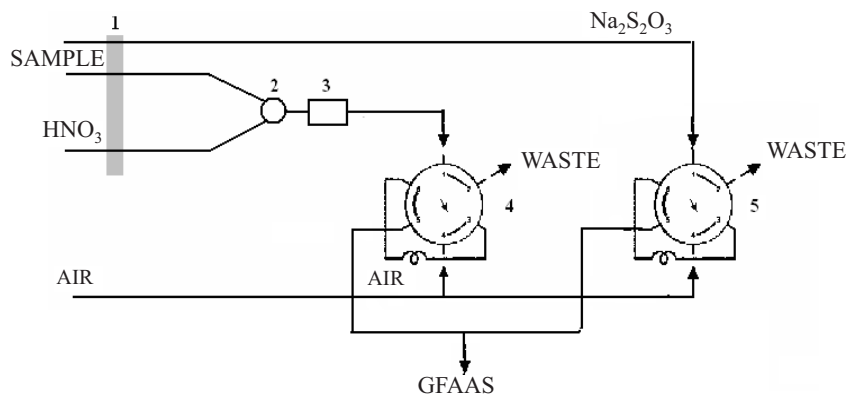


Fig. 1. Scheme of the flow-through system.

1 – pump, 2 – valve, 3 – preconcentration cell, 4, 5 – 6-way valve with sampling loop.

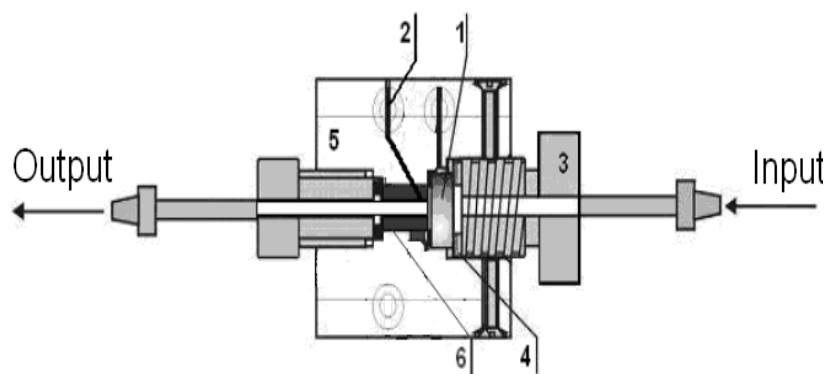


Fig. 2. Scheme of the flow-through cell with dual electrode connection.

1 – working electrode E-53 Au, 2 – auxiliary Pt electrode, 3 – screw, 4 – packing, 5 – plexiglass body, 6 – inert inset.

Table 2. Linear ranges, equations of the linear regressions and their coefficients for different feeding volumes Hg.

Volume [mL]	Linear range [ $\mu\text{g}\cdot\text{dm}^{-3}$ ]	Equation of the linear regression	Coefficient of determination
1	0.053-6.1	$A = 0.0057 + 0.3387 \cdot \rho$	0.9918
5	0.012-1.1	$A = 0.2347 + 0.568 \cdot \rho$	0.9856
10	0.00517-0.52	$A = 0.0458 + 1.354 \cdot \rho$	0.9945

tion of various Hg species to Hg(II), which can be preconcentrated on the electrode. Flow-through systems enable a simple and elegant online sample pretreatment just by mixing a suitable modifier solution to the flowing sample. The flow system used in this work enabled to mix the sample solution with a carrier electrolyte just through segmenting the sample and carrier electrolyte flows by switching the valve periodically once to the sample and then to the electrolyte. The carrier electrolyte contained diluted hydrochloric acid. The role of the hydrochloric acid was to facilitate the stripping of mercury and to shift its stripping peak to a potential range with minimum interference arising from an oxidation of the gold electrode and a signal produced by stripping frequently present Cu. The presence of potassium permanganate in the carrier electrolyte ensured the oxidation of reducing species as well as various mercury species in the sample solution before they entered the cell. The main problem was its interference during the stripping step as a result of the rapid chemical oxidation of the deposit competing with electrochemical stripping. Fortunately, the system made it possible to stop the flow before the stripping started and the permanganate in the diffusion layer could be reduced electrochemically. We used modifier  $\text{Na}_2\text{S}_2\text{O}_3$ .

Then, the applied current could perform the stripping of the deposit to the permanganate-free diffusion layer.

Modifier  $\text{Na}_2\text{S}_2\text{O}_3$  was used in this work. The optimum potential for preconcentration was  $-1200$  mV (Fig. 3). At more negative potentials high currents occur, which adversely influence the noise level as a result of bubble formation at the working electrode.

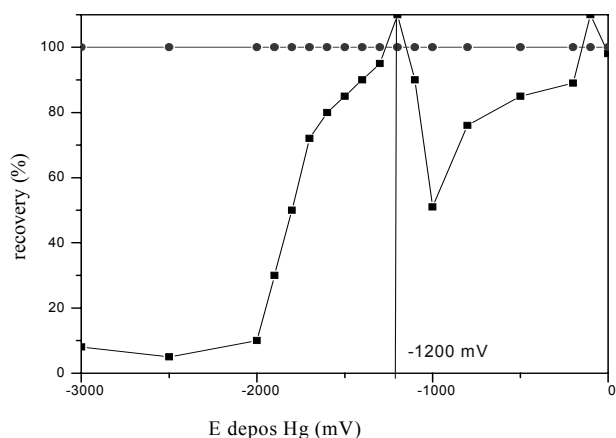


Fig. 3. Influence of the preconcentration potential on Hg signal.

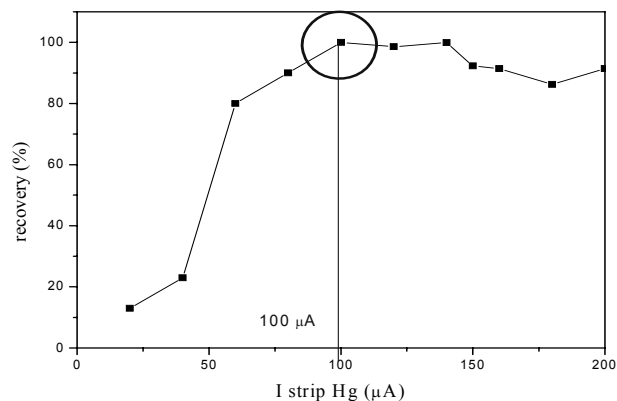


Fig. 4. Influence of the stripping current on Hg signal.

The stripping current in the range of  $20 \mu\text{A}$  to  $200 \mu\text{A}$  has only a negligible influence on the results. However, stripping currents higher than  $300 \mu\text{A}$  produced small signals with enhanced relative noise level. Hence a current of  $100 \mu\text{A}$  (Fig. 4) was used in further experiments. The gold electrode needed little care. The electrode surface was automatically cleaned after each run. After a few weeks of use the electrode was polished and used further. The influence of various inorganic substances on the determination of Hg was tested (Fig. 5) and (Fig. 6). The influence of the sample volume for various concentrations of Hg on the signal is presented in Fig. 7. In Fig. 8 repeatability for the determination of Hg,  $\rho = 20.0 \mu\text{g}\cdot\text{dm}^{-3}$ , RSD = 5.9 % for 15 measurements is shown.

Samples with low conductivity can also be analyzed since they are mixed in the flow system with the carrier electrolyte to form a conductive solution.

Linear ranges, equations of the linear regressions and their coefficients for different feeding volumes Hg are shown in Table 2; limit of detection (LOD) and limit of quantification (LOQ) for different feeding volumes Hg are shown in Table 3. The trueness was determined by means of a certified reference material DWPS with Hg content of  $20.0 \pm 0.2 \mu\text{g}\cdot\text{dm}^{-3}$  (Table 4).

The method enables us to determine Hg in the concentration range  $0.05$  to  $60 \mu\text{g}\cdot\text{dm}^{-3}$ .

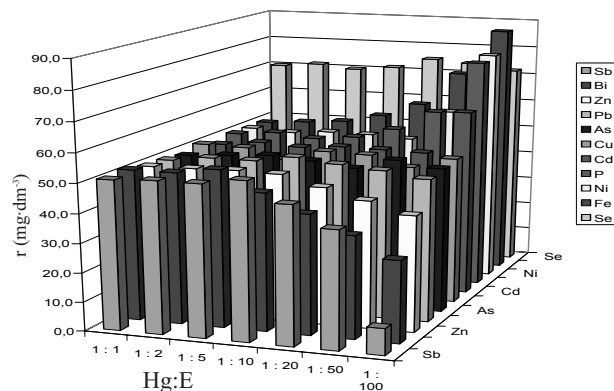


Fig. 5. Influence of the interfering ions on the determination of Hg before preconcentration.

Table 3. Limit of detection (LOD) and limit of quantification (LOQ) for different Hg feeding volumes.

Volume [mL]	LOD [ $\text{ng}\cdot\text{dm}^{-3}$ ]	LOQ [ $\text{ng}\cdot\text{dm}^{-3}$ ]
1	16	53
5	3.6	12
10	1.6	5.3

Table 4. Trueness determined in standard reference material DWPS.

FTC-ET AAS	$\rho$ Hg [ $\text{mg}\cdot\text{dm}^{-3}$ ] (found)	$\rho$ Hg [ $\text{mg}\cdot\text{dm}^{-3}$ ] (certified)
DWPS	$20.1\pm 0.1$	$20.0\pm 0.2$

## Conclusions

The aim of this work was to develop an online coupling of electrochemical preconcentration to AAS method and the utilization of the developed system for the determination of ultra-trace amounts of Hg in water samples.

The preconcentration parameters for the electrochemical process were optimized. The deposition runs at a constant preconcentration potential of  $-1\ 200$  mV and the optimum stripping current was found to be  $100\ \mu\text{A}$ . The deposition was made from a hydrochloric acid solution that was then replaced online by a diluted nitric acid facilitating the AAS measurement of Hg. The advantage of the used preconcentration system was in an automatic and reliable matrix exchange facility. Optimum electrolyte concentrations were found to be  $0.1\ \text{mol}\cdot\text{dm}^{-3}$  HCl and  $0.1\ \text{mol}\cdot\text{dm}^{-3}$   $\text{HNO}_3$  for the deposition and stripping/AAS measurement, respectively. Trueness was determined by means of a certified reference material DWPS with Hg content of  $20.0\pm 0.2\ \mu\text{g}\cdot\text{dm}^{-3}$ .

ET AAS in connection with flow-through electrochemical cell provides a useful tool for determination of mercury in water samples. The main advantage of the method lies in its simplicity and low cost.

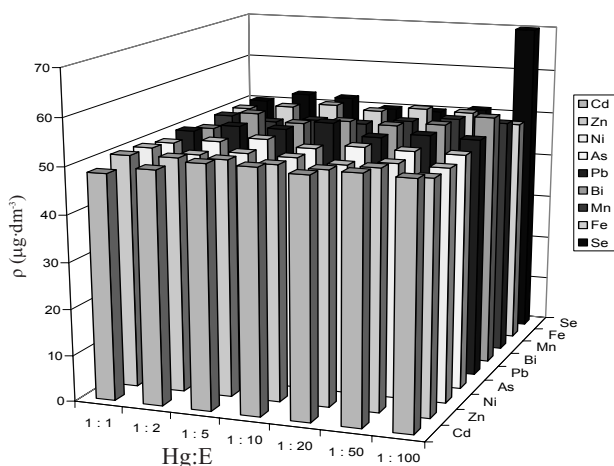


Fig. 6. Influence of the interfering ions on the determination of Hg after of preconcentration.

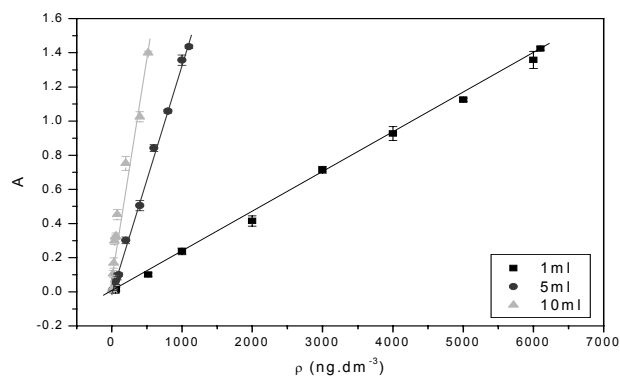
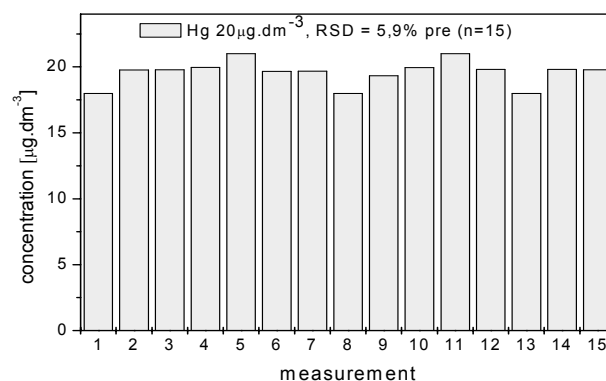


Fig. 7. Linear ranges for 1 mL, 5 mL, and 10 mL of feeding volume of the samples.

Fig. 8. Repeatability for the determination of Hg,  $\rho = 20.0\ \mu\text{g}\cdot\text{dm}^{-3}$ , RSD = 5.9% for 15 measurements.

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