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MERCURY DETECTION IN SURFACE AND WASTEWATER USING ATOMIC FLUORESCENCE SPECTROMETRY

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Introduction

Monitoring of mercury content in the environment samples is very important due to its toxicity.

The sources of mercury in the environment includes natural and anthropogenic sources. Natural sources are volcano eruptions and forest fires. In the anthropogenic sources are included incineration of wastes, emissions from coal power plants, urban discharges, atmospheric depositions, mining and agricultural activities.

Mercury is used in fluorescent lamps, thermometers, manometers, barometers, electrodes. Mercury was used in the mining industry in order to extract and concentrate precious metals such as gold and silver.

Different analytical methods were proposed in the last decade for mercury detection, the applied techniques used spectrometry (AAS-HG, AAS-CV, ICP-EOS-HG, ICP-MS-HG), chromatography or even electrochemistry.

The present paper proposes a sensitive and selective analytical method for mercury determination in surface and wastewater samples using atomic fluorescence spectrometry.

Materials and methods

Equipment: M-8000 Tedelyne Cetac Quick Trace Analyzer with Hg lamp (without gold trap mode)

Reagents: Mercury (II) in nitric acid 5%, Certified Reference Material, 1000 mg/L, Merck quality; potassium bromide; potassium bromate; hydrochloric acid; tin chloride; ascorbic acid. All reagents were of analysis quality (max. 0.000001% Hg).

Procedure: The samples and the standards were mineralized using chemically generated bromine and bromine chloride (BrCl), which decompose all organomercury species to mercury (II). Before the analysis, excess bromine was removed with ascorbic acid. Elemental mercury vapors were generated from the mineralized sample by reduction with tin (II) chloride and were purged from the solution using a carrier gas (argon). The elemental mercury was measured at 253.7 nm. The program applied to the Tedelyne equipment is described in Table 1.

Table 1. Parameters set-up for Hg determination using *without gold trap mode*

Sample uptake (s)	Rinse (s)	Read delay (s)	Replicates	Replicate time (s)	Pump speed (%)
55	95	60	4	5	100
GLS Gase	Purge Gas	#1 Start time	#1 End time	#2 Start time	#2 End time
High	High	8	18	130	140

Results and conclusions

The performance parameters such as: detection limit, quantification limit, repeatability, intermediate precision, linearity, recovery were estimated.

The results indicated that detection limit of the method is 4 ng/L, the quantification limit being 15 ng/L. The calibration curve was set-up in the range $0.2 \div 1.0 \mu\text{g/L}$. Value of the correlation coefficient for the calibration curve is 0.9985.

The precision of the method at $0.5 \mu\text{g/L}$ is 2.03% and the intermediate precision (repeatability data obtained by 2 analysts in 4 different days) is 6.75%.

The recovery percentage (wastewater sample) was estimated after participation in a LGC Proficiency Test Scheme. The obtained value for PT-AQ-17C was $5.9 \mu\text{g/L}$, the recovery percentage was 98.33%, Z score being -0.17.

The uncertainty budget was evaluated, overall uncertainty being 9.5%.

The performance parameters indicate that the method could be used not only for wastewater monitoring (NTPA 001, maximum admissible value: 0.05 mg/L), but also for surface water quality evaluation (lowest value for Class I quality according to 161/2006 Romanian Order is $0.1 \mu\text{g/L}$).

Figure 1a) present de calibration curve and figure 1b) indicate the shape of the peak for $0.6 \mu\text{g/L}$.

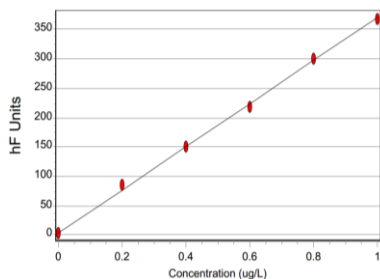


Fig.1a) Calibration curve

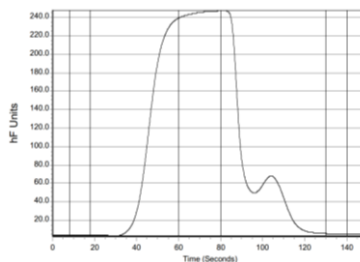


Fig.1b) Shape of the peak

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