

Determination of mercury in selected environmental components using cold vapour atomic absorption spectrometry

Oznaczanie rtęci w wybranych komponentach środowiska z wykorzystaniem atomowej spektrometrii absorpcyjnej z generowaniem zimnych par

Authors' Contribution:

A – Study Design
B – Data Collection
C – Statistical Analysis
D – Data Interpretation
E – Manuscript Preparation
F – Literature Search
G – Funds Collection

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Article history: Received: 30.11.2015 Accepted: 25.04.2016 Published: 10.05.2016

Abstract: Research on the determination of mercury in various environmental components has grown in significance over the past few years as the element's ubiquity in nature was recognized. One of the most common techniques for determining mercury content in environmental samples is cold vapour atomic absorption spectrometry (CV-AAS) because of its simplicity, high sensitivity and relatively low operating costs. This paper describes the methodology for the determination of mercury content in water, wastewater, bottom sediments and soils using CV-AAS. The main objective of this study was to calculate following the validation parameters: limit of detection, limit of quantification, linearity, range, selectivity, repeatability, accuracy. The uncertainty of the developed method also was assessed.

Keywords: mercury, cold vapour atomic absorption spectrometry, validation parameters, environment

Streszczenie: W ostatnich latach badania nad oznaczaniem rtęci w różnych komponentach środowiska zyskały na znaczeniu z uwagi na poznaną wszechobecność tego pierwiastka w przyrodzie. Jedną z najczęściej stosowanych technik oznaczania rtęci w próbkach środowiskowych jest atomowa spektrometria absorpcyjna z generowaniem zimnych par z uwagi na swoją prostotę, wysoką czułość oraz względnie niskie koszty eksploatacji. Artykuł opisuje metodykę oznaczania rtęci w wodach, ściekach, gruntach i osadach dennych z wykorzystaniem atomowej spektrometrii absorpcyjnej z generowaniem zimnych par. Głównym celem niniejszej pracy było wyznaczenie parametrów walidacyjnych: granicy wykrywalności, granicy oznaczalności, zakresu liniowości, zakresu roboczego, selektywności, powtarzalności oraz dokładności. Oceniono również niepewność opracowanej metody.

Słowa kluczowe: rtęć, atomowa spektrometria absorpcyjna z generowaniem zimnych par, parametry walidacyjne, środowisko

Introduction

Contamination of the natural environment is a serious problem with a highly negative impact on human health as well as other living organisms. Particularly dangerous are heavy metals, the introduction of which into a food chain result in their accumulation in an organism, the effects of which may become visible many years after the intoxication. Due to its

chemical and biological activity, durability, and high toxicity of the chemical forms that it takes, mercury is a metal of specific toxicity [1]. These forms may be classified as highly volatile ones and ones readily or poorly soluble in water. An example of the first ones are elemental mercury (Hg^0) and dimethylmercury ($(\text{CH}_3)_2\text{Hg}$). Among the ones readily soluble in water are mercury (II) chloride (HgCl_2) and mercury (II) sulfide ($[\text{HgS}_2]^{-2}$), while methylmercury (CH_3Hg^+) and mercury (II) cyanide

($\text{Hg}(\text{CN})_2$) belong to the group of insoluble forms. The ease with which mercury converts to various chemical forms is the source of its long-range transport properties. Gaseous mercury evaporates into the atmosphere and may be transported with air mass to very distant regions (its durability in the air may last up to 360 years), where after conversion to appropriate forms it is deposited into the land and aquatic environment. Mercury can take a myriad of pathways through the environment (Fig. 1/Rys.1) [2,3].

In the natural environment, mercury may be biotransformed into methylmercury ion, and may next transform back into the elemental form (e.g. methylation of mercury followed by ion reduction caused by certain yeasts). Methylmercury is the most mobile form mercury takes, which easily becomes accumulated in living organism tissues. That is why eating fish, amongst others, is the primary source of exposure to mercury [2,3] for humans. Harmful effects of mercury are very long-lasting as its compounds join together with enzymes and proteins thus causing negative changes in blood and blood vessels, which, as we all know, has an influence on the whole organism. The most sensitive to the effects of mercury is the central nervous system, proper functioning of which is of crucial importance. The negative impact of mercury may be observed also in the other systems, i.a. the cardiovascular, immune, and reproductive systems [4]. Emission of mercury from natural sources (e.g. volcanic eruptions, and emission from the oceans), as well as man-made ones (i.a. metals industry, agriculture, industrial waste management) has contributed to creating a great risk of exposure to mercury and its accumulation in the environment. Elevated mercury concentrations occur in various parts of the world. Due to mercury's transboundary nature (which has already been mentioned), contamination has affected the areas where no or minor sources of mercury occur, such as for example the Arctic [5,6,7]. Since the early 60's, the growing awareness of the dangers that mercury constitutes has led to the development of accurate and precise methods of its determination in various environmental samples. Mercury present in soil or water may very easily enter into the trophic chain of the ecosystem and get into a human body as a result. That is why, the monitoring of environmental mercury pollution is a very important issue and it shouldn't be neglected [8,9].

Modern analytical chemistry presents many diverse methods for the determination of mercury in environmental samples. They are, amongst others spectrophotometry, voltammetry, mass spectrometry, inductively coupled plasma emission spectrometry and neutron activation analysis. The most common analytical approaches for determining mercury content in environmental samples is cold vapour atomic absorption spectrometry (CV-AAS). The main advantages of this method are fast sample preparation (most type of samples can be analyzed without acid digestion), relatively short analysis time and low consumption of reagents. However, in case of ultratraces of mercury in water, the CV-AAS method is more and more often superseded

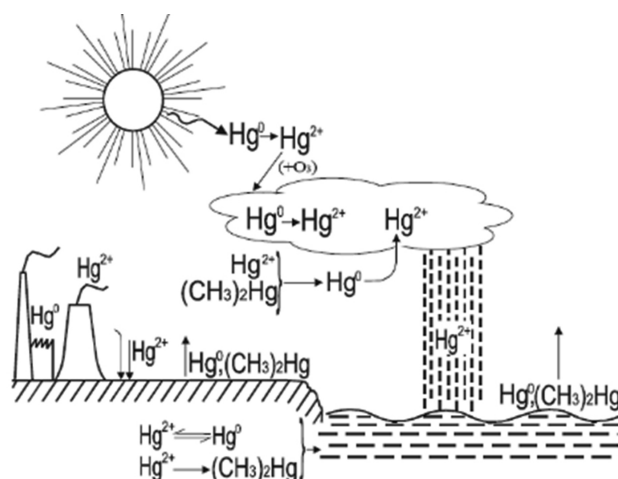


Fig1. Scheme of mercury cycle in nature [2].

by the cold vapour atomic fluorescence spectrometry (CV-AFS). Competitiveness of the method lies in its greater sensitivity and selectivity, as well as smaller interferences, but even so the most common analytical approach for mercury determination is CV-AAS, which has received great attention owing to its simplicity, high sensitivity and relatively low operating costs [1,10].

This paper describes the way of determining mercury in water, wastewater, soils and bottom sediments using cold vapour atomic absorption spectrometry. The materials selected for the purposes of the study constitute the main sources of mercury in the natural environment, the presence of which poses a big threat to all the living organisms. As a part of developing the above-mentioned methodology, there was optimization of the measurement conditions of the mercury analyzer (MA-2, NIC, Japan), validation parameters were calculated and the uncertainty of the method was evaluated. The limit of detection and quantification, repeatability, accuracy, linearity and selectivity were assessed as well. Uncertainty was determined with the use of an experimental method taking into account all the possible sources of measurement error. The aim of this paper was to validate a method for determination of mercury that would make it possible to eliminate the time-consuming stage of digestion of a sample from the process, as well as to reduce amounts of reagents.

Materials and Methods

Equipment and Analytical Procedure

A mercury analyzer MA-2 (NIC, Japan) was used for the study. It is a very simple and user-friendly instrument. Operating diagram of the MA-2 analyzer is shown in Fig. 2/Rys. 2.

Depending on the sample type, two types of method for the determination of mercury content can be applied. In the case of wastewater, bottom sediments and soils, mercury is deter-

mined using thermal decomposition of the sample inside a ceramic tube. The sample under analysis undergoes thermal decomposition inside the H1 oven. Next, mercury is subject to atomization in the H2 oven, and the free mercury vapors are absorbed on the golden collector, thus producing an amalgamate. Subsequently, the amalgamate is heated in order for the free (atomic) mercury to get released, which is next determined by CV-AAS. The golden collector is next cooled before another cycle is initiated. In order to remove any interfering substances (produced during the thermal decomposition of a sample), gas washing was performed.

While determining mercury content in water, a method of reduction was used (with 10% tin (II) chloride solution as a reducing agent). A water sample is introduced into a wash column, and then sulfuric acid solution and tin (II) chloride are added. Tin chloride reduces mercury ions into atomic mercury. Mercury vapors, after being dried inside the scrubber are next directed into the amalgamate, where their concentration takes place. Amalgamate is next heated in order for the free (atomic) mercury to get released, which is next determined with the use of the atomic absorption spectrometry.

Reagents

Working mercury standard solutions were prepared by appropriately diluting the stock standard solution with a concentration of 100 ppm in 2% hydrochloric acid of 99.999% purity (CPA-chem) with deionized water containing 0.001% L-cysteine of 99% purity (ACROS Organics). It is very important to use L-cysteine as a stabilizer to preparing appropriate standard solutions.

For determining the mercury content in wastewater, soils and bottom sediments, there were applied an activated alumina - Additive B (Wako Pure Chemical Industries), a mixture of sodium carbonate and calcium hydroxide - Additive M (Avantor), buffer solution pH 7.00 ± 0.05 (Avantor) and hydrochloric acid 36%, MerckTracepur (for bottom sediment's digestion with hydrochloric acid solution). When mercury content in water was being determined, the reagents used were sodium hydroxide (POCh), tin (II) chloride (Merck) and sulfuric acid (VI) 96% (Merck).

Samples preparation

The first stage of preparation of solid samples (soils, bottom sediments) to be tested was lyophilization. Next, the dried samples were sifted through a 2 mm mesh sieve in order to separate redundant elements, such as pebbles. Finally, the obtained samples were homogenized (grated inside a laboratory sample mill). After all those activities had been performed, moisture of the bottom sediments and soils under investigation was measured for the purposes of calculation of the final result on dry matter. Mercury content in soils was determined directly on the base of the prepared solid samples, while the bottom sediments were subject to digestion in 20% hydrochloric acid solution, after

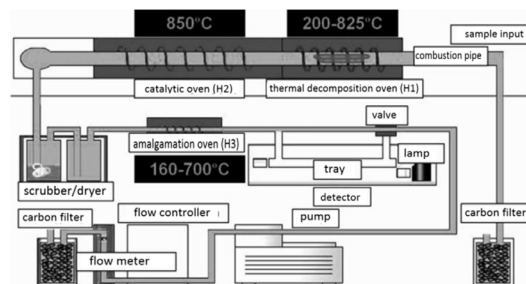


Fig 2. Schematic diagram of the MA-2 analyzer (NIC, Japan) [11].

which the concentration of mercury was determined basing on the received mineralizers (straining through filter paper into 25 cm³ flasks). Mercury content in water and wastewater samples were determined directly, without digestion of the samples. The only step before analysis of mercury in water was preparing reagents (tin chloride and sulfuric acid solution).

Optimization of the measurement conditions

Before the process of validation of the analytical method could be initiated, the measurement conditions of the instrument must be optimized. Following the measurement conditions to determine mercury content in environmental samples by CV-AAS using thermal decomposition or the reduction method described in Tab. I.

Results and Discussion

Validation parameters

While developing a new analytical method it is absolutely necessary to perform its validation. The aim of validation is to set parameters characterizing the given method and next, to document that the given methodology meets all the quality requirements and provides reliable results [12]. As a part of the validation process, amongst others, such quantities as accuracy, precision, selectivity, linearity, limit of detection and quantification are also assessed. It needs to be determined which of them have to be necessarily calculated for the best characterization of a given method [12,13]. It is crucial to find the optimal number of parameters, in order to limit both the length of time a validation process is to take (too many parameters to calculate), and a necessity to perform a "post-validation control" (when the scope of parameters is too narrow). It is noteworthy that validation doesn't refer only to new methods. It also pertains to cases where there occurs a need to extend the scope of application of an already known method (the same analytical method, but a different matrix), change its parameters or measurement conditions (a different laboratory, equipment, analyst) [14].

Within the framework of this paper, the following validation parameters were calculated: limits of detection and quantification, precision (repeatability), accuracy, range, linearity and

Tab. I. Optimization of measurement conditions (MA-2 analyzer; NIC, Japan).

ANALYSIS PARAMETER	TYPE OF TECHNIQUE BY MA-2 ANALYZER	
	THERMAL DECOMPOSITION	REDUCTION METHOD
Concentration level:	LOW/HIGH	LOW/HIGH
Flow rate of the carrier gas:	0,5 dm ³ min ⁻¹	0,5 dm ³ min ⁻¹
Temperature program:	MODE 2	RA-MODE
Decomposition temperature:	8500C	-
Time of decomposition:	70 sec.	-

Tab. II. Validation parameters for developed method (water and wastewater)

PARAMETER	DRINKING WATER	SURFACE WATER	GROUNDWATER	WASTEWATER
LOD [µg·dm ⁻³]	0.006	0.006	0.006	0.006
LOQ [µg·dm ⁻³]	0.05	0.05	0.05	0.5
Range [µg·dm ⁻³]	0.05-1000	0.05-1000	0.05-1000	0.5-3000
Repeatability [%]	4.25	2.33	2.63	3.38
Accuracy [%]		4.44		12
			r=0.9999	
Linearity	0-2 ng	0-2 ng	0-2 ng	0-2 ng
			r=0.9949	
	2-20 ng	2-20 ng	2-20 ng	2-20 ng
Expanded uncertainty, k=2[%]	27	25	29	29

selectivity. It was assumed that the above-mentioned parameters provide the fullest image of the method, and constitute a sufficient criterion for its identification. Additionally, measurement uncertainty was estimated for the developed method. Validation parameters and expanded uncertainty of the developed method are presented in Tab. II (water and wastewater) and Tab. III (soils and bottom sediments).

Limit of Detection (LOD) and Quantification (LOQ)

Limit of detection (LOD) is the lowest concentration of an analyte that may possibly be detected by means of a given analytical technique with a stated probability. It is determined most often on a basis of measurement blank samples. A multiple of this value is the limit of quantification (LOQ), which is defined as the lowest concentration that may be quantified via a given method assuming certain accuracy and precision [12,14].

LOD for water and waste water was taken to be 0.006 µg dm⁻³ and for soils and bottom sediments was 0.00022 mg kg⁻¹. However, LOQ was determined to be 0.05 µg dm⁻³ for water and 0.5 µg dm⁻³ for wastewater. For soils and bottom sediments it was 0.01 mg kg⁻¹.

Linearity

Linearity of the analytical method indicates whether the obtained measurement results are directly proportional to their

concentration within the given range. Preparation of calibration curves and determination of their correlation coefficient are the basis for conducting every analytical measurement [12,13].

The calibration curves was carried out as a dependence of peak height and mercury content (in the range 0-2 µg·dm⁻³ and 2-20 µg·dm⁻³) or peak surface area and mercury content (in the range 20-200 µg·dm⁻³). Regression coefficient *r* for water and wastewater were *r*=0.9999 (0-2 µg·dm⁻³) and *r*=0.9949 (2-20 µg·dm⁻³), however for soils and bottom sediments were *r*=0.9996 (0-2 µg·dm⁻³); *r*=0.9991 (2-20 µg·dm⁻³) and *r*=0.9999 for mercury content in the range 20-200 µg·dm⁻³. Therefore, it can be assumed that the developed method is linear over a wide concentration range.

Repeatability

The precision of the analytical method expresses the closeness of agreement (degree of scatter) between a series of measurements obtained from multiple sampling of the same homogeneous sample under the prescribed conditions. Precision may be considered at three levels: repeatability, intermediate precision and reproducibility. The precision of an analytical procedure is usually expressed as the variance, standard deviation or coefficient of variation of a series of measurements. Repeatability expresses the precision under the same operating conditions over a short interval of time [14, 15].

Repeatability for determination of mercury content in water amounted to 4.25% for drinking water; 2.33% for surface water and 2.63% for groundwater respectively. For wastewater, the precision was similar to water and it was 3.38%. However, for soils and bottom sediments the precision of mercury content was calculated at 4.56% and 9.54% respectively.

Accuracy

Accuracy of a method is defined as conformity of the obtained result with the reference value. On this basis, the percentage of error, which is a determinant of a given analytical method's precision, is next calculated. The term "accuracy" is often used interchangeably with the concept of "correctness" [15]. Basing on reference materials (from inter-laboratory comparisons), accuracy was calculated for different matrices in the developed method and it was 4.44% and 12.0% for water and wastewater respectively. Accuracy for soils and bottom sediments was 0.88% and 7.76% respectively. The following recoveries were obtained: 95.6% (water); 88% (waste water); 99.1% (bottom sediments); 92.2% (soils) (Tab. IV).

Selectivity

Selectivity of a method defines the degree to which it is possible to quantify the studied component in the presence of other substances contained in the sample, thus also assessing how the potential interferences impact on the analytical signal. Selectivity gives us information on to what degree does a result come directly from the analyte and not from the other substances, e.g. contaminants [16].

In order to avoid potential interferences, it is absolutely crucial to prepare laboratory containers in an appropriate way. In the case of the determination of mercury in water and waste water, the containers assigned to sampling are acid-etched with acid beforehand. Because of mercury vapour's volatility, the containers should be made of glass, or preferably of perfluorinated ethylene-propylene (FEP), which is characterized by a very high chemical resistance. Volatile organic compounds which absorb within UV region may lead

Tab. III. Validation parameters for developed method (soils and bottom sediments)

PARAMETER	SOILS	BOTTOM SEDIMENTS
LOD [mg kg ⁻¹]	0.00022	0.00022
LOQ [mg kg ⁻¹]	0.01	0.01
Range [mg kg ⁻¹]	0.01-100	0.01-100
Repeatability [%]	4.56	9.54
Accuracy [%]	0.88	7.76
Linearity		r=0.9996
	0-2 ng	0-2 ng
		r=0.9991
	2-20 ng	2-20 ng
		r=0.9999
	20-200 ng	20-200 ng
Expanded uncertainty, k=2 [%]	23.5	30.2

to obtaining overestimated results. To eliminate the arising interferences, potassium permanganate is added to water and to waste water in particular, which enables a partial decomposition of organic compounds. Additionally, background compensation is applied [17].

Contaminations such as: organic matter, halogens (halogen derivative compounds) and acids may have an influence on the selectivity of the method for determination of Hg in soils, sediments, and wastewater. These compounds may lead to obtaining overestimated results. Interferences caused by the compounds are eliminated in the procedure of determining Hg by the supplements M (mixture of sodium carbonate and calcium hydroxide) and B (activated alumina), which are placed together with the sample inside a ceramic boat. They cause neutralization, adsorption, and decomposition of the above-mentioned contaminations [18].

Uncertainty

Uncertainty is defined as a parameter connected with measurement result, which characterizes dispersion of values that may be reasonably classified as the measured value [19]. Although uncertainty is not counted among the basic valida-

Tab. IV. Comparison of the accuracy and recovery results as obtained for water, waste water, bottom sediments and soils

REFERENCE MATERIALS FROM INTER-LABORATORY COMPARISONS					
Type of sample	Organizer of the inter-laboratory comparisons	Reference value	Determined value	Accuracy [%]	Recovery [%]
Water [mg dm ⁻³]	Cracow University of Technology, Poland	0.00090	0.00086	4.44	95.6
Waste water [mg dm ⁻³]	Cracow University of Technology, Poland	0.0150	0.0132	12.0	88.0
Bottom sediments [mg kg ⁻¹]	Wageningen University, Netherlands	4.57	4.53	0.88	99.1
Soils [mg kg ⁻¹]	Wageningen University, Netherlands	0.889	0.820	7.76	92.2

tion parameters, ascribing its value to the obtained results is a necessary element of running a comparison of results between laboratories, clients, and institutions that make use of the measurement results [14, 20].

There can be distinguished, two methods of estimating the measurement uncertainty (type A and type B). The type A method of estimating uncertainty is based on statistical analysis of a series of measurement results. It is calculated on the basis of frequency distribution of the obtained results of multiple measurements, e.g. normal distribution. An example of this type of uncertainty estimation may be calculation of calibration uncertainty (one resulting from accuracy of measuring instrument). The type B way of estimating uncertainty makes use of methods other than the statistical ones, e.g. based on the experimenter's scientific judgement. Contrary to the type A method, the type B method of estimating uncertainty allows for a choice of distribution pattern (e.g. rectangular distribution). While calculating the value of uncertainty of this kind, all the possible sources of measurement error must be taken into account [20, 21].

It should be emphasized that the type A method has certain limitations, namely it may be used only when there is no constitutive systematic series of measurements, which is anyway established with the use of the type B estimation. The latter may in turn be applied in every situation, without any restrictions, which is due to the fact that it may be "supported" with an infinite (possible) number of factors, that is, e.g. the experiments conducted so far, former research results, certificates provided by the manufacturer, certificates of calibration and other documents on characteristics of given instruments, reports from the previous studies including for example, results connected with the calibration curves, and estimations of uncertainty conducted on the basis of reference materials [14, 22]. Standard uncertainty for the type A method is the standard deviation for the mean value of a series of measurement results, while determination of standard uncertainty for the type B refers to only one measurement result or to a situation where the obtained results do not display a dispersion. In this case, standard uncertainty is calculated on the basis of the knowledge of a given quantity or the interval to which this quantity should belong [21]. While estimating measurement uncertainty it is crucial to assess all the considerable elements of uncertainty. In the group there belong: standard uncertainty, relative standard uncertainty, composite standard uncertainty, and expanded uncertainty. The last one is the final stage of measurement uncertainty estimation and it defines the interval around the measurement, including a large fraction of the value range that can justifiably be attributed to the measurand. The final record of the result, together with a statement of the expanded uncertainty is a complete information characterizing the particular analytical measurement which indicates credibility of the given result, and so the ability to estimate uncertainty correctly is an indispensable tool in an analyst's everyday work [20, 21, 22].

In the discussed paper, the expanded uncertainty for water, waste water, soils, and bottom sediments was estimated with the use of the type B method for estimating uncertainty. In case of water and wastewater, the following values of expanded uncertainty were evaluated: 27% (drinking water); 25% (surface water); 29% (groundwater and wastewater). The expanded uncertainty calculated for soils and bottom sediments amounted to 23.5% and 30.2%, respectively.

Summary

The validation parameters are similar to those reported by other authors based on the CV-AAS method for determination of mercury in environmental samples. The recovery of determination mercury content by U.S. EPA Method 245.5 using CV-AAS were varied about 102.1%; 100.7%; 101.5%; 98.7% for various soils and sediments respectively. The calibration also demonstrates perfect linearity with a correlation coefficient of 0.9999 [23]. The detection limit falls in the similar range to described in other articles [24]. However, in comparison with another method, for example ICP-AES, the LOD by CV-AAS is much lower, so that CV-AAS describes higher sensitivity. In addition, ICP-AES involves several steps for the analysis of mercury (e.g. soils), among others sample digestion using acids or acid mixtures. However, determination of mercury content in soil samples by CV-AAS is a very easy and fast method, because the only step for preparing the samples is their freeze drying and homogenization before analysis [25].

Conclusions

The determination of mercury in environmental samples by CV-AAS is a simple, precise and accurate method. The linear range of calibration, repeatability, accuracy, detection and quantification limits obtained for the proposed method show its suitability for determining mercury in water, wastewater, bottom sediments and soils. At the same time, it is advisable that after the method is implemented for the intended use, the obtained validation parameters should be constantly improved by means of controlling their changeability while performing analyses. Considering the uncomplicated selection of specific samples in relation to choosing the type of CV-AAS method (decomposition or reduction), it is also possible to extend the scope of the method's applicability to other matrices, i.e. water extracts, plants, etc. The developed method for determination of mercury in environmental samples might also find application for example, in the analysis of mercury content in aquatic organisms, which could be a vital element in the reduction of the dangers that i.a. human consumption of fish poses. CV-AAS method requires a low consumption of reagents, as well as resulting in the production of insignificant amounts of by-products, it is eco-friendly. For these reasons, this method may be successfully applied for mercury determination in many various samples.

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Word count: 3720 Page count: 7 Tables: 4 Figures: 2 References: 25

Scientific Disciplines: Geoscience

DOI: 10.5604/12307424.1201262

Full-text PDF: www.bullmaritimeinstitute.com/fulltxt.php?ICID=1201262

Cite this article as: Szczepańska K., Dembska G., Zegarowski Ł., Pazikowska-Sapota G., Galer-Tatarowicz K., Aftanas B: Determination of mercury in selected environmental components using cold vapour atomic absorption spectrometry; *BMI*, 2016; 31(1): 73-80

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Competing interests: The authors declare that they have no competing interests.

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