

COMPARISON OF SOME WET DECOMPOSITION METHODS FOR MERCURY DETERMINATION IN INDUSTRIAL WASTE

JERZY CIBA, JOANNA KLUCZKA, MARIA ZOŁOTAJKIN

Silesian University of Technology, Chemistry Department, B. Krzywoustego 6, 44-100 Gliwice, Poland

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PORÓWNANIE MOKRYCH METOD MINERALIZACJI W CELU OZNACZANIA RTECI W ODPADACH PRZEMYSŁOWYCH

Streszczenie

Przedmiotem pracy były dwa różne odpady przemysłowe składowane na terenie zakładu chemicznego: zużyta w procesie elektrolizy solanki metodą rtęciową elektroda grafitowa i przepracowany katalizator rtęciowy na węglu aktywnym po syntezie chlorku winylu. Wymienione odpady charakteryzowały się dużą niejednorodnością i w zależności od partii zawierały 1–3% rtęci. Konieczność ich utylizacji wymagała opracowania szybkiej i powtarzalnej procedury oznaczania rtęci w poszczególnych partiach odpadowego grafitu i węgla aktywnego. W dostępnej literaturze znaleziono procedury mineralizacji próbek węgla i oznaczania w nich niskich stężeń rtęci metodą atomowej spektrometrii absorpcyjnej techniką zimnych par (CV AAS). Przebadano sześć procedur przeprowadzenia rtęci z badanych odpadów do roztworów, w których oznaczono rtęć metodą miareczkową opisaną przez Wickbolda i CV AAS. Wyniki poddano ocenie statystycznej. Stwierdzono, że cztery z przebadanych sposobów mineralizacji próbek odpadów przemysłowych są możliwe do wykorzystania.

Summary

The research was carried out on two different industrial wastes deposited on the premises of a chemical plant: used graphite electrode after electrolysis of brine applying the mercury-cathode method and coal catalyst past the usage period after the synthesis of vinyl chloride. The need for utilization of the waste necessitated development of a fast and reliable procedure for mercury determination. We have found procedures for mineralization of coal samples and determination of small concentrations of mercury by the cold vapour of atomic absorption spectrometry (CV AAS) in the available literature. Six procedures for passing mercury from the examined waste into solutions were tested, and mercury was assayed using the titration method of Wickbold and CV AAS. The results were evaluated statistically. It has been found that four ways to mineralize the examined industrial waste samples can be used.

INTRODUCTION

One of the extensively used analytical methods for mercury determination in waste materials is the cold vapour of atomic absorption spectrometry (CV AAS) [5, 25, 29]. The

determination with CV AAS does not pose methodological problems. However, the way of mineralization of waste samples and the process of passing all present metal forms into an analytical solution are of key importance in order to achieve reproducible and accurate results. The mineralization can be carried out by employing the wet method that uses acids or oxidizing substances [2, 6, 7, 11, 15, 20, 21, 24, 26]. Another method is dry mineralization, which consists in matrix combustion in oxygen and absorption of mercury vapour in an acid solution or its deposition in a filter or bed [12, 15, 18, 21, 24, 27]. Microwave digestion and thermal decomposition of the matrix are also successfully applied [1, 8, 15, 17, 19].

The aim of this research was to analyze mercury in two industrial wastes deposited on the premises of a chemical factory producing polyvinyl chloride and chlorine applying the mercury-cathode method. The first waste material – graphite electrode (ground after preliminary thermal treatment) used during electrolysis of brine applying the mercury-cathode method, and the second waste material – mercury catalyst (past usage period) in the coal matrix after the synthesis of vinyl chloride (granules) were characterized by high heterogeneity and contained 1–3% of unused mercury residue, depending on a batch [12]. The need for utilization of the waste necessitated development of a fast and reliable procedure for mercury determination in particular batches of the waste graphite and activated coal that differed in mercury content.

We have not found any procedure for mineralization of graphite and activated coal samples in the available literature so that we could assay mercury in the obtained solution applying the CV AAS method. However, some reports on decomposition of coal samples were written in the last three decades. Their authors [11, 24] claim that wet methods using mixtures of nitric(V), chloric(V) and sulfuric(VI) acids produced satisfactory results. Yet, they were time consuming; decomposition of a sample lasted 24 hours and a shorter heating (approx. 3 hours) did not result in a clear but dark brown solution that foamed during the CV AAS analysis and rendered the measurements impossible. The application of other oxidizing agents produced various results: worse with an addition of CrO_3 , KMnO_4 [2] or better with H_2O_2 [20], $(\text{NH}_4)_2\text{S}_2\text{O}_8$ [6]. Murphy [20] employed oxidation of coal samples (0.2 g) in a conical flask with concentrated H_2SO_4 at 100°C and was feeding 30% H_2O_2 until the mixture became clear. He determined the amount of mercury applying CV AAS with Ag amalgamation in the range of 0.03 ± 1 mg/g and obtained a satisfactory repeatability of results. The coal samples were treated with a mixture of HNO_3 – HClO_4 – $(\text{NH}_4)_2\text{S}_2\text{O}_8$ at $250\div 300^\circ\text{C}$ and then the solution obtained was analyzed using CV AT AAS. The insoluble silicates were eliminated through filtration because they caused interferences. The decomposition was being carried out for 30 minutes and the solution obtained was stable for 24 hours. The determination limit of the method was 0.08 mg Hg/g coal sample [6]. Zeng and Yao [28] suggested decomposition of the coal samples with H_2SO_4 – HClO_4 in the presence of Mo as a catalyst over $20\div 60$ min period recovering 97.8% and 96.4% of inorganic and organic mercury respectively. Chen [3] initially wetted $0.1\div 0.2$ g coal samples with a mixture of ethanol with 80 mg V_2O_5 and 10 cm^3 of concentrated HNO_3 , then slightly heated them for 30 minutes and added 4 cm^3 of concentrated H_2SO_4 .

Since the procedures of coal decomposition with the mixture of acids carried out in the open reflux system cause certain losses of inorganic mercury [7, 26] numerous papers

dealing with the closed system have been published. In the investigations coal and ash samples were decomposed in a pressure bomb and HNO_3 or KMnO_4 or their mixture are applied as absorbents [12, 24, 27].

The Polish Standard [21] recommends two mineralization methods for the determination of mercury in hard and brown coal. The first one involves the decomposition of coal in a flask equipped with a reflux condenser applying a mixture of concentrated sulfuric(VI) and nitric(V) acids until a light clear solution is obtained. The latter consists in combustion of a sample in a bomb calorimeter and adsorption of mercury vapour in nitric(V) acid (1:10). Mineralization of the coal sample in acids with microwave system is also allowed.

Determination of mercury in ultra pure coal was performed also by Rievaj [23]. He presented two ways of preliminary preparation of samples. The first method involved incineration of the coal matrix in oxygen flux and digestion of the residue in concentrated HNO_3 . The latter consisted in elimination of sorptive properties of the coal matrix through pulverization and extraction with a mixture of concentrated $\text{HCl} - \text{HNO}_3$ (2:1), at 160°C for 2 hours in a hermetic teflon vessel. Rievaj determined mercury applying the DP ASV method (Differential Pulse Anodic Stripping Voltammetry), and AAS for comparison reasons, obtaining satisfactory results.

Lengyel [15] compared the results of mercury determination obtained in 12 separate laboratories applying different analytical techniques (CV AAS, NAA and CV AFS) and ways of pre-treatment of model coal samples (combustion, microwave decomposition, mineralization with acids). The repeatability and reproducibility of the results were 0.02 mg/g and 0.04 mg/g respectively, mercury content in coal being 0.08 mg/g.

Furthermore, mercury was determined in hard coal and lignite after the samples had been burned in the closed system (burning time was 10 minutes). The process was carried out not in an oxygen bomb but in a specially constructed installation and the combustion gases were absorbed in HNO_3 solution (1:10) with the addition of KMnO_4 . Certified standards were applied to verify the method and satisfactory results were obtained (RSD = 5.8%) [18].

In Richaud's study [22] mercury levels in coals, coal-derived products, biomass materials, sand and kaolin have been determined by an atomic-absorption-based instrument without pretreatment of solid samples. The results have been compared with certified values of reference materials and instrument gave correct mercury concentration for certified reference materials.

There are some papers in which their authors propose the method of leaching mercury from investigated coal materials. Study on continuous extraction with acidified (4% HNO_3) subcritical water of mercury from coal prior to on-line derivatisation-atomic fluorescence detection was presented by Fernandez-Perez [9]. The method was validated using a reference material. It characterized good precision RSD = 6.5% ($n = 6$), safety and rapidity. Mercury was also quantitatively leached out of the coal reference materials into 1 mol/dm^3 nitric acid within 48–72 h and determined cold vapor technique after treated with gold or rhodium modifier [10]. The greatest advantage of the method is that only a minimum of reagents and sample handling steps are required, a prerequisite for accurate results in routine analysis.

Unfortunately, we did not have any information about mercury forms present in the coal material tested in the research, which might be very useful when selecting the mineralization methods. The conducted preliminary tests (sequential extraction procedure –

Table 1) revealed that the largest amounts of mercury in the graphite material are leached with hydrochloric acid – 47% and nitric(V) acid – 44%, while in the case of the activated coal, the largest amounts are extracted with nitric(V) acid – 70% [12, 13].

Table 1. Sequential extraction of mercury in the industrial waste

Mercury content in the fractions [mg/g]								
	Graphite electrode				Coal catalyst			
	H ₂ O	HCl	HNO ₃	HCl, HNO ₃ (3:1)	H ₂ O	HCl	HNO ₃	HCl, HNO ₃ (3:1)
\bar{X} (n=4)	0.260	4.160	3.962	0.535	0.365	2.523	8.357	0.697
SD	0.015	0.185	0.123	0.055	0.022	10.56	0.132	0.033
RSD [%]	5.8	4.4	3.1	10.2	6.0	4.2	1.6	4.7
W _{Hg} [%]	3	47	44	6	3	21	70	6

^a The values in the brackets denote the results obtained after the samples had been homogenized and averaged

n – number of determinations

\bar{x} – arithmetic mean

SD – standard deviation

RSD – relative standard deviation

t – Student's t-distribution

F – test on homogeneity of variance for CV AAS and titration

t_{crit} – critical value of the Student's t-distribution

F_{crit} – critical value of the F-test for a given number of degrees of freedom and significance level of 0.95

W_{Hg} – percentage of mercury in a given fraction

A literature survey and the preliminary tests – sequential extraction of mercury in the waste – convinced us to examine 4 procedures of wet mineralization: HNO₃ under various conditions (A, C, D), and aqua regia (B). The efficiency of those procedures was compared with the results obtained by means of the procedures recommended by the Polish Standard [21].

EXPERIMENTAL

MATERIALS

Graphite electrode (after preliminary thermal treatment) used during electrolysis of the brine applying the mercury-cathode method, mercury catalyst (beyond usage period) on the coal matrix after the synthesis of vinyl chloride (granules), the waste was ground under 0.2 mm of diameter.

APPARATUS AND REAGENTS

Atomic absorption spectrometer 3300 (Perkin Elmer) equipped with a hydride and mercury vapour forming unit (MHS-10), a cathode mercury-vapour lamp (Photron Perkin Elmer), nitric(V) acid, aqua regia, sulfuric(VI) acid (POCh, Poland), mercury standard solution 1 g/dm³ (Merck)

PROCEDURES FOR MERCURY DIGESTION IN INDUSTRIAL WASTES

PROCEDURE A – COLD EXTRACTION IN NITRIC(V) ACID

A 1-g sample was placed in a conical flask with a tapered glass joint and shaken for 24 hours in 20-cm³ of nitric(V) acid (conc. 6 mol/dm³). The extract was filtered, transferred into a 50-cm³ volumetric flask and filled to volume with distilled water.

PROCEDURE B – COLD EXTRACTION IN AQUA REGIA

Procedure A was followed replacing nitric acid with aqua regia.

PROCEDURE C – MACERATION WITH NITRIC(V) ACID IN OPEN SYSTEM

A 1-g sample was placed in tall 100-cm³ beakers and heated with 20-cm³ of nitric(V) acid (conc. 6 mol/dm³) at boiling point for 2 hours. After filtration the eluate was transferred into a 50-cm³ volumetric flask and filled to volume with distilled water. The precipitate was macerated again in nitric(V) acid. After filtration, the separated solution was transferred into a flask that was filled to volume with distilled water.

PROCEDURE D – MACERATION WITH NITRIC(V) ACID IN THE GLASS SET

A 1-g sample was placed in a conical flask of a glass set and heated with 20-cm³ of nitric(V) acid (conc. 6 mol/dm³) at boiling point for 2 hours. After filtration, the eluate was transferred into a 50-cm³ volumetric flask and filled to volume with distilled water. The precipitate was macerated in nitric(V) acid for 1 hour. After filtration, the separated solution was transferred into a flask that was filled to volume with distilled water.

PROCEDURE E – ACID MINERALIZATION IN GLASS SET

A 1-g sample was placed in a round-bottomed flask of a glass set and heated with 20-cm³ of concentrated sulfuric(VI) acid at boiling point for 2 hours. Next 2-cm³ of concentrated nitric(V) acid was added and heated 1 hour. The proportioning of concentrated nitric(V) acid was repeated three times. After filtration, the eluate was transferred into a 50-cm³ volumetric flask and filled to volume with distilled water. The precipitate was macerated in nitric(V) acid for 1 hour. After filtration, the separated solution was transferred into a flask that was filled to volume with distilled water.

PROCEDURE F – COMBUSTION IN THE BOMB CALORIMETER

A 0.4-g sample was mixed with 0.4-g of benzene carboxylic acid, tableted and burned in oxygen (2.5 MPa). Mercury vapour was absorbed in nitric(V) acid. Then the solution from the calorimeter was transferred quantitatively into a 100-cm³ volumetric flask that was filled to volume with distilled water.

Procedure E – mineralization of a sample in a glass set applying sulfuric(VI) acid and dosing nitric(V) acid – and Procedure F – combustion of a sample in a bomb calorimeter adsorbing mercury vapour in nitric(V) acid – were conducted according to the Polish Standard [21]. Mercury was assayed in the obtained solutions using CV AAS.

RESULTS AND DISCUSSION

The results of the research that aimed at determining mercury level in the waste are presented in Table 2. The pretreatment of the samples was performed by applying Procedure C. Mercury was assayed in the solutions using the titration method developed by Wickbold [16] and, for comparison, CV AAS. The wide scatter of the results around the calculated mean value (RSD > 20%) points to the high heterogeneity of the examined waste. Therefore, the waste was ground and homogenized before further tests in the laboratory in order to improve the precision of the determination. Grinding of the samples prior to the mineralization enabled us to obtain a higher mercury recovery, which was suggested by the data found in the literature [23]. It was found out that the examined wastes contained about 1% of total mercury. The comparison of the analytical methods showed that both the titration method as well as CV AAS can be applied to determine mercury in the solutions, and the differences between the mean values should be regarded as negligible ($t < t_{crit}$, $F < F_{crit}$). The application of atomic absorption spectrometry necessitated a hundred-fold dilution of the solutions, which might result in additional errors e.g. systematic or random. Nevertheless, CV AAS rather than the titration method were selected for further measurements due to its fast performance.

Table 2. Mercury content in the industrial waste after mineralization according to procedure C – comparison of CV AAS and titration methods

Procedure:	Mercury content in the waste [mg/g]			
	Graphite electrode		Coal catalyst	
	CV AAS	Titration	CV AAS	Titration
\bar{X} (n=8)	7.488 (8.634) ^a	8.691 (8.826) ^a	10.270 (11.057) ^a	12.213 (11.400) ^a
SD	1.822 (0.397) ^a	2.145 (0.423) ^a	2.392 (0.530) ^a	2.801 (0.583) ^a
RSD [%]	24.3 (4.6) ^a	24.7 (4.8) ^a	23.3 (4.8) ^a	22.9 (5.1) ^a
t	1.60 (1.24) ^a < t_{crit}		1.97 (1.63) ^a < t_{crit}	
F	1.03(1.09) ^a < F_{crit}		1.03 (1.14) ^a < F_{crit}	

^a The values in the brackets denote the results obtained after the samples had been homogenized and averaged

n – number of determinations

\bar{x} – arithmetic mean

SD – standard deviation

RSD – relative standard deviation

t – Student's t-distribution

F – test on homogeneity of variance for CV AAS and titration

t_{crit} – critical value of the Student's t-distribution

F_{crit} – critical value of the F-test for a given number of degrees of freedom and significance level of 0.95

W_{Hg} – percentage of mercury in a given fraction

The results of the industrial waste mineralization according to procedures A–F are shown in Table 3. Six parallel tests were performed which were followed by the calculation of the arithmetic mean and measurement error. The Dixon's test was also conducted to eliminate questionable results. The procedures for matrix digestion were selected on the basis of the following criteria:

- repeatability of measurement results in a given series,
- Student's t-test which compares the results of two measurement series,
- percentages of mercury recovery,
- simplicity of a given procedure.

Table 3. Mercury content in the industrial waste, depending on the applied procedure of sample mineralization

Mercury content in graphite electrode [mg/g]						
Procedure:	A	B	C	D	E	F
n	6	6	8	6	6	6
\bar{X}	4.117	4.159	8.643	9.074	8.933	8.696
SD	0.086	0.094	0.397	0.240	0.370	0.537
RSD [%]	2.1	2.3	4.6	2.6	4.7	8.0
Mercury content in coal catalyst [mg/g]						
Procedure:	A	B	C	D	E	F
n	6	6	8	6	6	6
\bar{X}	2.471	2.453	11.057	11.982	12.116	11.660
SD	0.062	0.076	0.530	0.336	0.399	0.481
RSD [%]	2.5	3.1	4.8	2.8	3.3	4.1

^a The values in the brackets denote the results obtained after the samples had been homogenized and averaged

n – number of determinations

\bar{x} – arithmetic mean

SD – standard deviation

RSD – relative standard deviation

t – Student's t-distribution

F – test on homogeneity of variance for CV AAS and titration

t_{crit} – critical value of the Student's t-distribution

F_{crit} – critical value of the F-test for a given number of degrees of freedom and significance level of 0.95

W_{Hg} – percentage of mercury in a given fraction

The application of procedures C, D, E, F produced satisfactory results. The optimum way to mineralize examined samples was their double maceration in a glass set applying nitric acid (procedure D). The average mercury content was 9.1 mg Hg/g in the waste graphite and 12.0 mg Hg/g in waste catalyst. This method is characterized by good reproducibility (RSD = 2.6; 2.8%). The results were comparable to the ones obtained by applying the mineralization recommended by the standard [21] – procedures E (sample mineralization in concentrated sulfuric(VI) and nitric(V) acids) and F (combustion of the sample in

bomb calorimeter). Procedure C (hot maceration of sample in nitric(V) acid) resulted in smaller mercury content, probably, owing to the bigger losses of mercury while heating the solutions in the open system. The 24-hour cold extraction, both with nitric(V) acid – procedure A – as well as aqua regia – procedure B – produced lower results, at least by half. Presumably, under the conditions of room temperature, the acids did not leach mercury and its compounds present in the deeper parts of the tested materials, which is confirmed by the literature [4].

Finally, procedure D – hot maceration with nitric(V) acid (conc. 6 mol/dm³) in a glass set – was selected for the pretreatment of the industrial waste. The selection of the procedure for matrix digestion depended upon the efficiency of mercury recovery, reproducibility of results and the procedure's ease of use.

CONCLUSIONS

The conducted research and interpretation of the results enable a selection of the most suitable analytical procedure for determination of the total mercury content in the waste tested in this research. The mineralization of the waste graphite and coal with nitric(V) acid in a glass set carried out proved to be the most satisfactory. Although the tested waste is of coal origin, there is no need to apply the more time-consuming procedures recommended for this matrix [11, 12, 21, 24].

The proposed method enables a fast and reproducible determination of mercury in the contaminated waste investigated in this research.

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