The MAK Collection for Occupational Health and Safety

Mercury – Method for the determination of mercury vapours in workplace air using atomic absorption spectrometry

Air Monitoring Method – Translation of the German version from 2017

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Mercury – Method for the determination of mercury vapours in workplace air using atomic absorption spectrometry

Air Monitoring Methods

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Abstract

This analytical method is a validated measurement procedure for the determination of mercury vapours in workplace air using atomic absorption spectrometry in a concentration range of one thousandth up to twice the currently valid Occupational Exposure Limit (OEL) or MAK value of 0.02 mg/m^3. Both personal and stationary sampling can be performed for risk assessment at work. Sampling is carried out by drawing a defined volume of air through an adsorption tube filled with hopcalite (mixture of MnO_2 and CuO) using a flow-regulated pump. The flow rate is set to 250 mL/min with a recommended air sample volume of approx. 30 L at a sampling period of 2 hours. After sampling the loaded hopcalite is transferred into a quartz boat and the collected mercury can be analysed directly via atomic absorption spectrometry (AAS). The quantitative determination is based on a calibration function obtained by means of a 10-point calibration. The limit of quantification is 0.013 mg/m^3 based on an air sample volume of approx. 30 L with an expanded uncertainty between 18 and 21%.

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Keywords

Mercury vapours; hopcalite; air analysis; workplace measurement; hazardous substances; workplace monitoring; air sampling; atomic absorption spectrometry; AAS

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Mercury – Method for the determination of vapour from mercury and its inorganic compounds in workplace air using atomic absorption spectrometry

Method number	1
Application	Air analysis
Analytical principle	Atomic absorption spectrometry (AAS)
Completed in	March 2016

Summary

The analytical procedure described here permits the determination of mercury vapour and the vapours of its inorganic compounds in workplace air in a concentration range of 0.001 up to twice the currently valid Occupational Exposure Limit (OEL) or MAK value for mercury of 0.02 mg/m^3 [1, 2]. A suitable pump draws a defined volume of air through a collection tube filled with hopcalite (a mixture of MnO_2 and CuO) for the sampling procedure. After sampling is complete, the hopcalite loaded with mercury is transferred into a quartz combustion boat and analysed directly by means of atomic absorption spectrometry. The quantitative determination is based on a 10-point calibration using mercury calibration solutions.

Characteristics of the method

Precision:	Standard deviation (rel.):	$s = 2.6$ to 11.5%
	Expanded uncertainty:	$U = 18$ to 21%
	in the concentration range of a tenth up to twice the Occupational Exposure Limit and $n = 6$ determinations	
Limit of quantification:	Absolute:	0.4 ng
	Relative:	0.013 µg/m^3 at an air sample volume of 30 L and a sampling period of 2 h
Recovery:	98 to 111%	
Sampling recommendations:	Sampling period:	2 h
	Air sample volume:	30 L
	Flow rate:	250 mL/min

Description of the substances

Mercury [7439-97-6]

Mercury is a naturally occurring element with the chemical symbol of Hg, an atomic number of 80 and an atomic mass of 200.59 u. It is a silvery-white, liquid heavy metal with a melting point of -38.83 °C and a boiling point of 357 °C. Its density is 13.5 g/cm^3. Mercury has a high surface tension, therefore it forms into drops on inert surfaces due to its strong cohesion. Although mercury occurs in its pure form in nature, it is usually found as a mineral ore in the form of cinnabar (HgS). Furthermore, it is contained in bituminous coal. It forms alloys with many metals, the so-called amalgams. In the past mercury was widely used in liquid-filled and contact thermometers as well as in barometers. Since 2009 the sale of new fever thermometers, barometers and sphygmanometers containing mercury has been banned within the EU; devices for scientific or medical use as well as antique and second-hand devices are exempt from the ban.

In the chemical industry mercury is used in chloralkali electrolysis according to the amalgam process. Mercury is used on a large scale for the extraction of gold. It is also employed in the field of medicine, e.g. as a dental filling material in the form of amalgam and as a preservative for vaccines due to its bactericidal properties. Mercury is used in illuminants (“energy-saving light bulbs”) that are a replacement for the previously market-domineering incandescent light bulb [3]. Nowadays the increasing use of LED illuminants is reducing its use in this application. In Germany approx. 9.5 t of mercury were emitted in 2011. Around 70% of air emissions are caused by energy generation [4]. In 2010 the world-wide mercury emissions were approx. 1960 t. The main sources of emission were gold panning and the small-scale extraction of gold as well as the combustion of coal (power stations and households) with approximately 37% and 24% respectively [5].

The Occupational Exposure Limit (OEL) or MAK value for mercury is 0.02 mg/m^3 I [1, 2]. The peak limit has been allocated an excursion factor of 8 in Category II. Furthermore, mercury has been classified as a Category 3 B carcinogen and due to its good absorption through the skin is marked with H and Sh [2]. Detailed information on the toxicity of mercury can be found in the toxicological-occupational health documentation of the MAK values [6].

Contents

1 General principles

A flow-regulated sampling pump draws a defined volume of air from the breathing zone through a collection tube filled with hopcalite. In this process the mercury vapours present in the air are adsorbed onto the hopcalite, a mixture of copper and manganese oxides. Separate sample preparation is not necessary.

The hopcalite loaded with mercury can be directly analysed by means of atomic absorption spectrometry after removal from the collection tube. For this purpose the loaded hopcalite is transferred into a quartz combustion boat and heated in accordance with a predefined temperature programme. After passing over a catalyst in order to reduce the mercury compounds present, the elemental mercury thus released is captured on a gold trap. Then the mercury is transferred into the measurement cell and analysed by means of atomic absorption spectrometry. The quantitative determination is based on a 10-point calibration using mercury calibration solutions.

2 Equipment, chemicals and solutions

2.1 Equipment

- Analytical balance
- Mercury analytical system (e.g. DMA-80, incl. external control unit and autosampler, from MLS GmbH, 88299 Leutkirch, Germany) with two cuvettes (measurement cells) of different sensitivities arranged in series (working ranges 0 – 20 ng and 20 – 1100 ng)
- Quartz combustion boats for loading into the analytical device
- Collection tubes filled with hopcalite (Anasorb® C300, Carulite® (HYDRAR)), NIOSH type (standardised 70 x 6 mm, adsorbent filling of 200 mg, 20/40 mesh) (e.g. SKC, from Analyt MTC, 79379 Müllheim, Germany)
- Tube holder
- Pump for personal sampling, suitable for a flow rate of 250 mL/min (e.g. GilAir plus, from Sensidyne, LP, St. Petersburg, USA)

- Flow meter (e.g. TSI model 4199, from TSI Inc., Shoreview, U.S.A)
- Weighing boats
- Volumetric flasks, 50 and 100 mL
- Glass beakers, 10 mL
- Microlitre pipettes, 10 to 100; 100 to 1000 and 500 to 2500 µL
- Pipettes, 5 and 25 mL
- Pointed-tip tweezers made of metal
- Tube cutter
- Metal spatula
- Wash bottle
- Small brush (for washing the quartz combustion boats)

2.2 Chemicals

The substances to be determined should have the highest possible purity.

- Oxygen 5.0 (gas to operate the atomic absorption spectrometer)
- Hydrochloric acid, 30%, Suprapur (e.g. from Merck KGaA, 64293 Darmstadt, Germany)
- Potassium bromide p.a. (e.g. from Merck)
- Potassium bromate p.a. (e.g. from Merck)
- Mercury standard (AAS standard solution, Suprapur), 1000 µg/mL (e.g. from Alfa Aesar GmbH & Co. KG, 76185 Karlsruhe, Germany)
- Ultrapure water
- Wheat flour, 405 type (German standard flour)

2.3 Solutions

The procedure for the preparation of the solutions necessary for calibration is described below.

Potassium bromide solution:

3 g of KBr are added into a 50 mL volumetric flask, into which approximately 20 mL of ultrapure water have been previously placed. Then 25 mL of the 30% hydrochloric acid are added using a pipette and, after cooling to room temperature, the volumetric flask is filled to the mark with ultrapure water and shaken.

Potassium bromate solution:

2 g of $KBrO_3$ are diluted with ultrapure water to 30 mL in a 50 mL volumetric flask. Then 5 mL of the 30% hydrochloric acid are added using a pipette and the volumetric flask is filled to the mark with ultrapure water and shaken.

Mercury solutions:

The following standard solutions 1 and 2 are prepared from the stock solution (mercury standard with 1000 µg/mL) in each case before carrying out the analytical determinations.

Standard solution 1: Mercury (0.01 µg/µL)

1 mL of stock solution is placed into a 100 mL volumetric flask, then 200 µL each of potassium bromide and potassium bromate solutions are added, the flask is then filled to the mark with ultrapure water and shaken.

Standard solution 2: Mercury (0.1 ng/µL)

1 mL of standard solution 1 is placed into a 100 mL volumetric flask, then 200 µL each of potassium bromide and potassium bromate solutions are added, the flask is then filled to the mark with ultrapure water and shaken.

The freshly prepared standard solutions are stable for up to 14 days under normal laboratory conditions!

Calibration solutions:

The calibration solutions are prepared in 50 mL volumetric flasks in accordance with the instructions given in Table 1. All calibration solutions are stabilised by adding 100 µL each of the potassium bromide and potassium bromate solutions. The calibration solutions cover the entire working range from the limit of determination up to double the Occupational Exposure Limit (OEL, MAK value) when both cuvettes in the analytical system are used. The appropriate calibration solutions can be selected according to the subject under investigation and the anticipated concentration range.

The calibration solutions should be used in sufficient quantities – *a 10-point calibration should be carried out, if possible* – as the resulting calibration functions are not linear (second degree polynomials). It is important to ensure that the transition between the measurement ranges of the two cuvettes is covered. The calibration range can also be covered with fewer calibration solutions if pertinent information on the anticipated mercury concentration in the analytical samples is available. The calibration solutions must always be freshly prepared.

3 Sampling and sample preparation

3.1 Sampling

Sampling can be carried out as stationary or personal sampling. Immediately before sampling, an adsorption tube filled with hopcalite is opened and connected to a flow-regulated pump. A flow rate of 250 L/min is then set. At a recommended sampling period of 2 hours this is equivalent to an air sample volume of approx. 30 litres. After sampling, the flow rate must be checked for constancy. If the deviation from the adjusted flow rate is greater than ± 5%, it is advisable to discard the measured value [7]. After sampling, the collection tubes loaded with mercury are sealed with the plastic caps that are supplied with them and unambiguously labelled.

Table 1 Preparation of the calibration solutions

Calibration solution No.	Content of mercury [ng/250 µL]	Stock solution [µL]	Standard solution 1 [µL]	Standard solution 2 [µL]	KBr solution [µL]	$KBrO_3$ solution [µL]
1	0.5	0	0	1000	100	100
2	0.75	0	0	1500	100	100
3	1.0	0	0	2000	100	100
4	1.75	0	0	3500	100	100
5	2.5	0	50	0	100	100
6	5.0	0	100	0	100	100
7	7.5	0	150	0	100	100
8	10.0	0	200	0	100	100
9	12.5	0	250	0	100	100
10	15.0	0	300	0	100	100
11	17.5	0	350	0	100	100
12	20.0	0	400	0	100	100
13	22.5	0	450	0	100	100
14	25.0	0	500	0	100	100
15	27.5	0	550	0	100	100
16	30.0	0	600	0	100	100
17	40.0	0	800	0	100	100
18	50.0	0	1000	0	100	100
19	75.0	0	1500	0	100	100
20	100.0	0	2000	0	100	100
21	250.0	50	0	0	100	100
22	500.0	100	0	0	100	100
23	750.0	150	0	0	100	100
24	1000	200	0	0	100	100

Note:
Enough collection tubes filled with hopcalite must be included to ensure that there are additional field blank samples.[1]

The method was checked at a flow rate of 250 L/min up to an air sample volume of 120 L (≙ 8 h) [8]. No breakthrough could be observed in a collection tube connected downstream (≤ 1%) at mercury concentrations of up to approx. double the OEL at a sampling period of up to eight hours.

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[1] Recommended number of field blank samples: (Number of analytical samples)/4 + 1

3.2 Sample preparation

Sample preparation is carried out in the laboratory by transferring the mercury-loaded hopcalite from the collection tube into a quartz combustion boat. For this purpose the collection tube is carefully opened with a tube cutter, the glass wool is removed from the collection tube with pointed-tip tweezers and then the loaded hopcalite is transferred into a quartz combustion boat; if necessary the opened collection tube must be tapped from the outside to facilitate the transfer. The quartz combustion boat is then placed into the autosampler of the mercury analytical system and analysed in accordance with the temperature programme described in Table 2.

4 Operational conditions for atomic absorption spectrometry

The instrumental parameters for the mercury analytical system are summarised below.

Apparatus: DMA-80 mercury analytical system with autosampler
Analytical wavelength: 253.7 nm
Autosampler: integrated into the analytical system; 40 insertion places
Combustion gas: Oxygen 5.0 (6 - 10 L/h; 400 kPa working pressure)

Table 2 lists the temperature programme of the mercury analytical system for checking the system without quartz combustion boats as well as for the calibration solutions and analytical samples (including blank samples and flour).

Note:
It is important to note that, in contrast to the calibration solutions, the analytical samples are solid substances that can, when heated too rapidly, begin "to jump or dance" in the combustion boats, which can lead to a loss of analyte.

5 Analytical determination

Before the start of the analytical determination the analytical system must be operated twice without insertion of the quartz combustion boats (for system check see Table 2). The catalyst within the analytical system is then cleaned by subjecting a quartz combustion boat filled with approx. 100 µg of flour to the entire temperature programme as the analytical samples undergo (see Table 2). The filling of an unused hopcalite collection tube is analysed at regular intervals in order to determine the blank value.

For the purpose of the analytical determination the entire content of a hopcalite collection tube is transferred into a quartz combustion boat and analysed under the conditions stated in Section 4. The quantitative determination is based on a calibration function that was obtained using the mercury calibration solutions listed in Table 1. The peak heights are used for the evaluation of the calibration solutions and analytical samples (when the peak areas are used for evaluation, comparable results are obtained).

Table 2 Temperature programme of the mercury analytical system

	System check (blank value method)	Calibration solution	Analytical samples (hopcalite)
Max. starting temperature*	500 °C	200 °C	200 °C
Heating rate 1	max.	max.	100 °C/60 s
Temperature 1:	750 °C	300 °C	300 °C
Dwell time at T1	180 s	120 s	0
Heating rate 2	–	max.	450 °C/180 s
Temperature 2:	–	750 °C	750 °C
Dwell time at T2	–	180 s	180 s
Purge time (Oxygen, 6 – 10 L/h)	30 s	60 s	60 s
Gold trap			
Heating time	12 s	12 s	12 s
Heating temperature	900 °C	900 °C	900 °C
Recording time of the measurement signals of the measurement cells	24 s	30 s	30 s

* A maximum starting temperature is stated, as the analytical system must cool down after each previous measurement. If the process is begun at a lower starting temperature, then the waiting time to the next analysis may increase significantly. All other working steps remain unchanged.

6 Calibration

The calibration function based on the calibration solutions listed in Table 1 is stable in the long-term. The calibration function must be checked by recalibration every working day by means of analysis of two calibration samples. It is advisable to carry out the check using calibration solutions with a content of 10 or 100 ng of mercury in 250 µL.[2] Two quartz combustion boats loaded with 250 µL of ultrapure water must be analysed according to the temperature programme for the calibration samples (see Table 2) before each calibration/recalibration. The calibration must be performed anew if the analytical conditions change or the quality control results indicate that this is necessary.

7 Calculation of the analytical result

Based on the peak heights obtained the corresponding mass X in µg is derived from the respective calibration curve. As the filling of a hopcalite collection tube is ana-

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2) This is equivalent to calibration solutions 8 and 20 in Table 1.

lysed directly without further processing steps, the recovered mass X in ng of mercury is equivalent to the absolute mass collected during sampling. The corresponding mass concentration (ρ) is calculated according to Equation (1) below as follows:

$$\rho = \frac{X_{Hg}}{V} \tag{1}$$

where:

ρ is the mass concentration of mercury in µg/m^3

X_{Hg} is the mass of mercury in the analytical sample in ng

V is the air sample volume in litres (calculated from the flow rate and the sampling period)

8 Reliability of the method

The characteristics of the method were calculated as stipulated in EN 482 [9] and DIN 32645 [10] with SKC hopcalite collection tubes, Carulite (HYDRAR), lot 8679.

An atmosphere containing mercury was generated in order to determine the characteristics of the method with mercury concentrations in the minimum measurement range (approx. one tenth, one and two times the OEL). At the same time the mercury concentration was recorded using the Tracker 3000 IP direct-reading measurement device (from Mercury Instruments GmbH, 85757 Karlsfeld, Germany) and used as a reference value. The measured temperature and relative humidity were continuously 21 °C and 55%.

8.1 Precision and expanded uncertainty

Six hopcalite collection tubes were each spiked with three different mercury concentrations (2.6; 20.7 and 38.8 µg/m^3) from a test gas atmosphere to determine the precision. Clean air was then drawn through the tubes at a flow rate of 250 mL/min for 30 minutes. The theoretical values were monitored with the Tracker 3000 IP and were constant during sampling. The loaded collection tubes were then processed as described in Sections 3.2, 4 and 5 and analysed. Two hopcalite collection tubes that were not loaded were included per series as blanks. The resulting air concentrations as well as the corresponding standard deviations and recoveries at these mercury concentrations are listed in Table 3.

Table 3 Characteristics of the validation

Concentration nominal value [µg/m^3]	Concentration found [µg/m^3]	Repeated standard deviation (rel.) [%]	Recovery [%]	Expanded uncertainty U [%]
2.6	2.87	2.8	111	20.8
20.7	20.5	1.7	98.9	17.6
38.8	38.1	1.1	98.4	17.7

The expanded uncertainty was estimated as stipulated in EN 482 [9] and calculated according to [11] taking all relevant influencing factors into consideration. The values listed in Tables 3 and 4 for the standard deviation under repeatability conditions, recovery and storage stability were used for this purpose. The expanded uncertainty lies in the range between 18 and 21%. The individual values can be found in Table 3.

8.2 Recovery

The recovery was determined in the course of determining the precision in the minimum measurement range (Section 8.1) from six parallel samples in each case. The investigated concentrations as well as the determined recovery rates are listed in Table 3. The mean recovery for mercury was 103 ± 6%. The recovery should not be taken into account in the calculation of the results due to this result.

8.3 Limit of quantification

The limit of quantification was determined from a 10-point calibration with 0.5 to 5 ng of mercury in an aqueous solution as stipulated in DIN 32645 [10]. The resulting absolute limit of quantification was 0.4 ng of mercury. The relative limit of quantification is 0.013 µg/m^3 under the recommended sampling conditions (2 h, 250 mL/min).

8.4 Storage stability

The storage stability was tested by spiking 12 hopcalite collection tubes for 30 minutes at a flow rate of 250 mL/min from a controlled test gas atmosphere with mercury concentrations of 1.95; 18.7 and 37.2 µg/m^3. The collection tubes sealed with protective caps were then stored in daylight under normal laboratory conditions.

Two hopcalite collection tubes spiked as described above were prepared and analysed in accordance with Sections 3.2, 4 and 5 after one day as well as after one, two, three, four and five weeks. The resulting measurement values are listed in Table 4. The storage stability of the samples in the adsorbed state without losses is at least 5 weeks at room temperature. The collection tubes must be sealed with the appropriate caps for storage.

8.5 Influence of the humidity

All tests were carried out in an air-conditioned laboratory at a room temperature of 21 °C and a relative humidity of 55%. An investigation on the influence of the relative humidity was not carried out.

Tests with adsorption tubes that are filled with 200 mg of Carulite (Hydrar) have been described in the literature [12]. The investigations showed that the influence of the temperature in the range from 5 to 40 °C and a relative humidity in the range from 20 to 70% at a mercury concentration of 50 µg/L can be considered negligible.

Table 4 Investigation of the storage stability

Nominal value Tracker (mean value)	Concentration of mercury [µg/m³]		
	1.95	18.7	37.2
Storage	Recovery [%]		
1 day	110	95.9	99.3
1 day	116	95.1	102
1 week	105	97.5	102
1 week	106	96.0	98.9
2 weeks	114	90.2	102
2 weeks	102	97.0	95.8
3 weeks	114	95.9	99.4
3 weeks	106	97.2	102
4 weeks	115	97.9	101
4 weeks	115	99.9	103
5 weeks	117	96.4	102
5 weeks	108	100	103
Mean value	111	96.6	101
Standard deviation	5.05	2.53	2.16
Standard deviation (rel.)	4.6%	2.6%	2.1%

8.6 Interference

Interference has not been observed for the analytical method described here.

Whether organic mercury compounds can be sampled in the same manner was not investigated. If such investigations are undertaken, it can be assumed that the measurement signal will increase.

9 Discussion

The analytical method described here permits a rapid and reliable determination of mercury in workplace air.

Personal sampling is carried out using hopcalite as a collection phase. In this case the analytical determination is carried out directly from the collection phase by means of atomic absorption spectrometry without further processing. The determined characteristics of the method fulfilled the requirements stipulated in DIN EN 482 [9]. The limit of quantification at a sampling period of two hours and an air sample volume of 30 litres was 0.013 µg/L. The expanded uncertainty of the method lies in the range between 18 and 21%.

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