BRITISH STANDARD

BS ISO 20552:2007

Workplace air — Determination of mercury vapour — Method using gold-amalgam collection and analysis by atomic absorption spectrometry or atomic fluorescence spectrometry

ICS 13.040.30

British Standards

National foreword

This British Standard was published by BSI. It is the UK implementation of ISO 20552:2007.

The UK participation in its preparation was entrusted by Technical Committee EH/2, Air quality, to Subcommittee EH/2/2, Work place atmospheres.

A list of organizations represented on EH/2/2 can be obtained on request to its secretary.

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This British Standard was
published under the authority
of the Standards Policy and
Strategy Committee
on 30 March 2007

Amendments issued since publication

Amd. No.	Date	Comments

ISBN 978 0 580 50333 7

INTERNATIONAL STANDARD

ISO 20552

First edition
2007-02-15

Workplace air — Determination of mercury vapour — Method using gold-amalgam collection and analysis by atomic absorption spectrometry or atomic fluorescence spectrometry

Air des lieux de travail — Détermination de la vapeur de mercure — Méthode combinant un prélèvement par amalgamation à l'or et une détection par spectrométrie d'absorption atomique ou par spectrométrie de fluorescence atomique

Reference number
ISO 20552:2007(E)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20552 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

Introduction

The health of workers in many industries is at risk through exposure by inhalation of mercury and inorganic mercury compounds. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure, and this is generally achieved by making workplace air measurements. This International Standard has been published in order to make available a method for making measurements of mercury vapour in the workplace environment, i.e. by static sampling. It is also of use for making short-term personal exposure measurements. The standard will be of benefit to: agencies concerned with health and safety at work; industrial hygienists and other public health professionals; analytical laboratories; industrial users of mercury and inorganic mercury compounds and their workers, etc.

The procedure described in this International Standard is based upon several published papers [1][2][3][4][5][6] that describe methodology for the determination of mercury vapour in air. This procedure has been fully validated and the resulting back-up data are presented in this standard.

It has been assumed in the drafting of this International Standard that the execution of its provisions and the interpretation of the results obtained, is entrusted to appropriately qualified and experienced people.

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Workplace air — Determination of mercury vapour — Method using gold-amalgam collection and analysis by atomic absorption spectrometry or atomic fluorescence spectrometry

1 Scope

This International Standard specifies a procedure for determination of the mass concentration of mercury vapour in workplace air using a method of gold-amalgam collection with analysis by either cold vapour atomic absorption spectrometry (CVAAS) or cold vapour atomic fluorescence spectrometry (CVAFS).

The procedure specifies a number of sampling methods for different applications.

a) When it is known that no particulate inorganic mercury compounds are used in the workplace and that none are produced in the processes carried out, samples of mercury vapour are collected using a pumped sorbent tube containing porous gold-coated diatomaceous earth. Suitable sorbent tubes are commercially available or they can be made from sorbent prepared by pyro-decomposition of chloroauric acid [hydrogen tetrachloroaurate(III)] sintered on diatomaceous earth.

b) When both mercury vapour and particulate inorganic mercury compounds could be present in the test atmosphere, samples of mercury vapour are collected using a pumped sorbent tube fitted with a quartz fibre prefilter to remove particulate inorganic mercury compounds. If desired, the procedure described in ISO 17733 can be used to collect and analyse separate samples for measurement of particulate inorganic mercury compounds.

c) When it is known that no elemental mercury is used in the workplace and that no mercury vapour is produced in the processes carried out, the procedure described in ISO 17733 can be used, if desired, to collect and analyse samples for measurement of particulate inorganic mercury compounds.

The procedure is highly sensitive and suitable for static sampling or for determination of short-term personal exposure to mercury vapour in workplace air. The lower limit of the working range of the procedure is governed by the lower limit of the analytical range of the CVAAS or CVAFS instrument, which is approximately 0,01 ng of mercury for a sorbent tube containing 80 mg of sorbent (see 13.1). The upper limit of the working range of the procedure is governed by the upper limit of the analytical range of the CVAAS or CVAFS instrument, e.g. about 1 μg of mercury (see 13.2). The sampling capacity of one commercially available sorbent tube has been shown to be greater than 2 μg. The concentration ranges of mercury in air for which the procedure is applicable are determined in part by the sampling method selected by the user.

The procedure is suitable for making short-term measurements (e.g. 15 min) when sampling at a flow rate of between 100 ml min^{-1} and 1 000 ml min^{-1} using a commercially available sorbent tube. For assessment of long-term exposure, such as 8 h, this procedure can be used with sampling flow rate of 100 ml min^{-1} in workplaces where the concentration of mercury vapour is expected to be lower than 20 μg m^{-3}. If the expected concentration of mercury vapour is higher than 20 μg m^{-3}, it is necessary to use the procedure prescribed in ISO 17733.

The method is unsuitable for making measurements of mercury vapour in air when chlorine is present in the atmosphere, e.g. in chloralkali works (see 13.8.1). Gaseous organo-mercury compounds can cause a positive interference (see 13.8.2).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 17733, *Workplace air — Determination of mercury and inorganic mercury compounds — Method by cold-vapour atomic absorption spectrometry or atomic fluorescence spectrometry*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 General definitions

3.1.1
chemical agent
any chemical element or compound, on its own or admixed as it occurs in the natural state or as produced by any work activity, whether or not produced intentionally and whether or not placed on the market

[EN 1540:1998] [7]

3.1.2
breathing zone
⟨general definition⟩ space around the worker's face from where he or she takes his or her breath

3.1.3
breathing zone
⟨technical definition⟩ hemisphere (generally accepted to be 0,3 m in radius) extending in front of the human face, centred on the mid-point of a line joining the ears; the base of the hemisphere is a plane through this line, the top of the head and the larynx

NOTE 1 The definition is not applicable when respiratory protective equipment is used.

NOTE 2 Adapted from EN 1540 [7].

3.1.4
exposure (by inhalation)
situation in which a chemical agent is present in air, which is inhaled by a person

3.1.5
measuring procedure
procedure for sampling and analysing one or more chemical agents in the air, and including storage and transportation of the sample

3.1.6
operating time
period during which a sampling pump can be operated at specified flow rate and back pressure without recharging or replacing the battery

[EN 1232:1997] [8]

3.1.7
limit value
reference figure for concentration of a chemical agent in air

NOTE An example is the Threshold Limit Value® (TLV) for a given substance in workplace air, as established by the ACGIH [9]. (Threshold Limit Value® is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.)

3.1.8
reference period
specified period of time stated for the limit value of a specific chemical agent

NOTE Examples of limit values for different reference periods are short-term and long-term exposure limits, such as those established by the ACGIH [9].

3.1.9
workplace
defined area or areas in which the work activities are carried out

[EN 1540:1998] [7]

3.2 Sampling definitions

3.2.1
personal sampler
device attached to a person that samples air in the breathing zone

[EN 1540:1998] [7]

3.2.2
personal sampling
process of sampling carried out using a personal sampler

[EN 1540:1998] [7]

3.2.3
sampling instrument
sampler
device for collecting airborne particles or gaseous materials (vapour), or both

3.2.4
sorbent tube, pumped
tube, usually made of metal or glass, containing an active sorbent or reagent-impregnated support, through which sampled atmosphere is passed at a rate controlled by an air sampling pump

[EN 1076:1997] [10]

3.2.5
static sampler
area sampler
device, not attached to a person, that samples air in a particular location

3.2.6
static sampling
area sampling
process of air sampling carried out using a static (area) sampler

3.3 Statistical terms

3.3.1
analytical recovery
ratio of the mass of analyte measured when a sample is analysed, to the known mass of analyte in that sample, expressed as a percentage

3.3.2
bias
consistent deviation of the results of a measurement process from the true value of the air quality characteristic itself

3.3.3

precision

closeness of agreement of results obtained by applying the method several times under prescribed conditions

3.3.4

true value

value which characterizes a quantity or quantitative characteristic, perfectly defined in the conditions which exist when that quantity or quantitative characteristic is considered

NOTE The true value of a quantity or quantitative characteristic is a theoretical concept and, in general, cannot be known exactly.

[ISO 3534-2:2006, definition 3.2.5] [11]

3.3.5

uncertainty (of measurement)

parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand

NOTE 1 The parameter may be, for example, a standard deviation (or a given multiple of it), or the width of a confidence interval.

NOTE 2 Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of the results of a series of measurements, and can be characterized by standard deviations. The other components, which also can be characterized by standard deviations, are evaluated from assumed probability distributions based on experience or other information. The *Guide to the expression of uncertainty in measurement (GUM)* [12] refers to these different cases as Type A and Type B evaluations of uncertainty, respectively.

NOTE 3 Adapted from the *International vocabulary of basic and general terms in metrology (VIM)* [13].

4 Principle

Mercury vapour is collected by drawing a known volume of air through a sorbent tube containing porous gold-coated diatomaceous earth using a pump. The sorbent tube is preceded by a quartz fibre filter to trap particulate inorganic mercury compounds when these could be present in the test atmosphere. If desired, the procedure described in ISO 17733 is used to collect and analyse separate samples for measurement of particulate inorganic mercury compounds.

The sample tube is transported to the laboratory and installed in a mercury analyser, consisting of a double gold-amalgam unit, or sample applicator unit, and a CVAAS or CVAFS analyser unit. The sample applicator unit comprises two heaters, separated by a gas washer, and a charcoal filter. The sample tube used for sampling mercury vapour is installed in the first heating unit and the temperature is raised to about 300 °C. At this temperature any volatile organic compounds collected with the mercury vapour are driven off, but the mercury is retained on the sample tube. The released volatile organic compounds pass through a second gold sorbent tube, which is preheated to 150 °C in the other heating unit, before being exhausted through the charcoal filter. The sample tube is then heated to the vaporisation temperature of mercury (about 700 °C) and the mercury vapour released is trapped on the second gold sorbent tube (a mercury-gold amalgam is formed up to about 200 °C). Finally, the trapping tube is heated to 700 °C and the mercury vapour is released into the CVAAS or CVAFS analyser unit. An important characteristic of double gold-amalgam technique is that the mercury peak is sharp and stable due to the reproducible analysis conditions that result from repeated use of the same gold trap. Sample tubes can be reused up to 3 000 times if no damage occurs from exposure to chlorine or ammonia.

The results may be used for the assessment of workplace exposure to mercury vapour (see EN 689 [14]).

5 Reactions

The porous gold-coated sorbent used in the method described in this International Standard has been shown to have a reversible affinity for mercury. The trapped mercury forms an alloy, called an amalgam, from which mercury vapour is easily released by heating.

NOTE Other amalgam-based mercury sorbents are known, e.g. silver wool and gold wool. However, silver wool has many problems. It is easily oxidized, has a low sorption capacity and poor collection and recovery efficiencies. Similarly, mercury is not released efficiently from gold wool by heating and does not give sharp analytical peaks [1].

6 Requirement

The measuring procedure shall comply with any relevant International, European or national standard that specifies performance requirements for procedures for measuring chemical agents in workplace air.

7 Reagents

During the analysis of mercury vapour by this method, only analytical grade mercury reagent is needed for generation of a standard mercury vapour to make a calibration curve.

7.1 Argon, suitable for use in CVAAS.

7.2 Mercury, for preparation of a mercury vapour standard.

8 Apparatus

8.1 Sampling equipment

8.1.1 Sorbent tubes, glass, containing a single section of porous gold-coated diatomaceous earth, stored in a glass tube sealed with a butyl rubber stopper.

The construction of such a sorbent tube is illustrated in Figure B.1.

NOTE A sorbent tube suitable for use in this method is commercially available [2][3][15]. This is 160 mm long, with a 6 mm outer diameter and a 4 mm internal diameter, and contains 80 mg of porous granular gold sorbent retained by small quartz wool plugs. Similar custom-made sorbent tubes can also be used if it can be demonstrated that they give equivalent performance. Custom-made sorbent tubes can be manufactured from porous gold-coated sorbent consisting of diatomaceous earth on which pyro-decomposition of chloroauric acid has been carried out by sintering at about 800 °C.

8.1.2 Sorbent tube and prefilter assemblies, consisting of a quartz fibre filter (8.1.3) mounted in a suitable sampler, connected to the inlet of a sorbent tube (8.1.1) with a minimum length of inert plastic tubing, e.g. polytetrafluoroethylene (PTFE) or modified polyvinyl chloride tubing.

NOTE A sorbent tube and prefilter assembly is only required if the concentration of airborne particles could be so high that there is a risk of blockage of the quartz wool plugs that retain the gold-coated sorbent in the tubes.

8.1.3 Quartz fibre filters, of a diameter suitable for use in the samplers used for construction of prefilter assemblies (see 8.1.2), with a collection efficiency of not less than 99,5 % for particles with a 0,3 μm diffusion diameter (see 2.2 of ISO 7708 [16]).

NOTE Quartz fibre filters have been shown [17] not to absorb mercury vapour from the sampled air. They are therefore suitable for use as prefilters. It has been reported [18] that there can be significant loss of mercury vapour if mixed cellulose ester filters are used as prefilters, although these findings were not reproduced in later work [19].

8.1.4 Sampling pumps, with an adjustable flow rate, capable of maintaining the selected flow rate (see 10.1.3) to within ± 5 % of the nominal value throughout the sampling period (see 10.1.4).

NOTE 1 A flow-stabilized pump may be required to maintain the flow rate within the specified limits.

For personal sampling the pumps shall be capable of being worn by the worker without impeding normal work activity. Sampling pump flow meters shall be calibrated using either a primary or secondary standard; if a secondary standard is used, it shall be calibrated using a primary standard.

The pump should have, as a minimum, the following features:

- an automatic control that keeps the volume flow rate constant in the case of a changing back pressure;
- either a malfunction indicator which, following completion of sampling, indicates that the air flow has been reduced or interrupted during sampling; or an automatic cut-out, which stops the pump if the flow rate is reduced or interrupted; and
- a facility for the adjustment of flow rate, such that it can only be actuated with the aid of a tool (e.g. screwdriver) or requires special knowledge for operation (e.g. via software), so as to preclude inadvertent readjustment of the flow rate during use.

An integral timer is a highly desirable additional feature.

NOTE 2 EN 1232 [8] requires that the performance of the pumps is such that:

- the pulsation of the flow rate does not exceed 10 %;
- a flow rate set within the nominal range does not deviate by more than ± 5 % from the initial value under increasing back pressure;
- within the range of ambient temperatures from 5 °C to 40 °C, the flow rate measured under operating conditions does not deviate by more than ± 5 % from the flow rate at 20 °C;
- the operating time is at least 2 h, and preferably 8 h; and
- the flow rate does not deviate by more than ± 5 % from the initial value during the operating time.

If the sampling pump is used outside the range of conditions specified in EN 1232 [8], appropriate action should be taken to ensure that the performance requirements are met. For instance, at sub-zero temperatures it might be necessary to keep the pump warm by placing it under the worker's clothes.

8.1.5 Flow meter, portable, with an accuracy that is sufficient to enable the volume flow rate (see 10.1.3) to be measured to within ± 5 %.

The calibration of the flow meter shall be checked against a primary standard, i.e. a flow meter whose accuracy is traceable to national standards. If appropriate (see 10.1.2), record the atmospheric temperature and pressure at which the calibration of the flow meter was checked.

It is advisable that the flow meter used is capable of measuring the volume flow rate to within ± 2 % or better.

8.1.6 Ancillary equipment.

8.1.6.1 Flexible tubing, of a diameter and length suitable for making a leak-proof connection between the sampling pumps and the sorbent tubes and/or the samplers.

8.1.6.2 Belts or harnesses, to which the sampling pumps can conveniently be fixed for personal sampling (except where the sampling pumps are small enough to fit inside worker's pockets).

8.1.6.3 Forceps, flat-tipped, for loading and unloading of filters and sorbent capsules into and out of samplers.

8.1.6.4 Filter transport cassettes, or similar, if required to transport samples for laboratory analysis.

8.1.6.5 Barometer, suitable for measurement of atmospheric pressure, if required (see 10.1.2).

8.1.6.6 Thermometer, 0 °C to 50 °C, graduated in divisions of 1 °C or better, for measurement of atmospheric temperature, if required (see 10.1.2).

For applications at temperatures below freezing, the range of the thermometer shall extend to the appropriate desired range.

8.2 Analytical instrumentation

A mercury analyser consisting of a double gold-amalgam unit (or sample applicator unit), comprising two heaters, a gas washer and a charcoal filter, connected to an analyser unit employing a cold vapour atomic absorption spectrometer or cold vapour atomic fluorescence spectrometer. A block diagram of an instrument employing a double gold amalgam system with CVAAS is illustrated in Figure B.2.

NOTE A mercury analyser suitable for use in this method is commercially available [2][3][15].

9 Occupational exposure assessment

9.1 General

This standard pertains to the taking both of static (area) and personal samples. Refer to relevant International, European or national standards (e.g. EN 689 [14], ASTM E 1370-96 [20], etc.) for guidance on how to develop an appropriate assessment strategy and for general guidance on measurement strategy.

9.2 Static (area) sampling

Static (area) sampling may be carried out, if appropriate, at points systemically selected in the workplace for one or more of the following purposes:

a) to assess the concentration of mercury vapour in the working environment,

b) to give an indication of the efficiency of ventilation or other engineering controls,

c) to characterize or to provide information on the location and intensity of an emission source, and/or

d) to estimate the exposure of workers in situations where personal sampling is not possible.

9.3 Personal sampling

Exposure of workers to mercury shall normally be determined by personal sampling, since the concentration of mercury vapour in the breathing zone can be different from the background level in the workplace.

9.4 Selection of measurement conditions and measurement pattern

9.4.1 General

9.4.1.1 Sampling shall be carried out in such a way as to cause the least possible interference with the worker during the normal performance of the job, and to provide samples that are representative of normal working conditions and that are compatible with the analytical method.

9.4.1.2 The pattern of sampling shall take into consideration practical issues, such as the nature of the measurement task and the frequency and duration of particular work activities.

9.4.2 Screening measurements of variation of concentration in time and/or space

Screening measurements of variation of concentration in time and/or space may be performed to provide information on the likely pattern of concentration of chemical agents. They can be used to identify locations and periods of elevated exposure, and to set the duration and frequency of sampling for measurements for comparison with limit values. Emission sources can be located and the effectiveness of ventilation or other technical measures can be estimated (see 4.3 of EN 482:1994 [21]).

9.4.3 Screening measurements of time-weighted average concentration and worst case measurements

9.4.3.1 Screening measurements of time-weighted average concentration may be performed to obtain relatively crude information on the exposure level in order to decide whether an exposure problem exists at all, and if so to appraise its possible seriousness. They may also be used to determine if the exposure is well below or well above the limit value (see 4.2 of EN 482:1994 [21]).

9.4.3.2 Screening measurements of time-weighted average concentration are typically carried out in the initial stages of a survey to assess the effectiveness of control measures. Sampling may be carried out during representative work episodes to obtain clear information about the level and pattern of exposure, or worst case measurements may be made.

NOTE Screening measurements of time weighted average concentration made to clearly identify work episodes during which highest exposure occurs are typically referred to as “worst case measurements” (see 5.2.3.2 of EN 689:1995 [14]).

9.4.4 Measurements near an emission source

Measurements may be performed near an emission source to provide information on the location and intensity of the source. In association with other information they can allow the elimination of a suspected source as a significant contributor to exposure (see 4.4 of EN 482:1994 [21]).

9.4.5 Measurements for comparison with limit values and periodic measurements

9.4.5.1 Measurements for comparison with limit values

9.4.5.1.1 Measurements for comparison with limit values are performed to provide accurate and reliable information on, or allow the prediction of, the time-weighted average concentration of a specific chemical agent in the air, which could be inhaled (see 4.5 of EN 482:1994 [21]).

9.4.5.1.2 For making measurements for comparison with a long-term exposure limit, samples shall be collected for the entire working period, if possible, or during a number of representative work episodes (see 10.1.4.1 for the minimum sampling time).

9.4.5.1.3 For making measurements for comparison with a short-term exposure limit, the sampling time shall be as close as possible to the reference period, which is typically 15 min, but can be anything between 5 min and 30 min (see 10.1.4.2).

NOTE The best estimate of long-term exposure is obtained by taking samples for the entire working period, although this might not always be practicable.

9.4.5.2 Periodic measurements

Periodic measurements are performed to determine whether exposure conditions have changed since measurements for comparison with limit values were made, or whether control measures remain effective. (see 4.6 of EN 482:1994 [21]).

10 Sampling

10.1 Preliminary considerations

10.1.1 Selection of sampling method

10.1.1.1 Measurement of mercury vapour

For measurement of mercury vapour, or for measurement of total inorganic mercury (mercury vapour and inorganic mercury compounds) when it is known that no particulate inorganic mercury compounds are used in the workplace and that none are produced in the processes carried out, use the pumped sorbent tube method specified in this International Standard to collect samples.

10.1.1.2 Measurement of mercury vapour and particulate inorganic mercury compounds

When both mercury vapour and particulate inorganic mercury compounds could be present in the test atmosphere, use the pumped sorbent tube method specified in this International Standard to collect samples of mercury vapour and, if desired, use the procedure described in ISO 17733 to collect and analyse separate samples for measurement of particulate inorganic mercury compounds.

10.1.1.3 Measurement of particulate inorganic mercury compounds

For measurement of inorganic mercury compounds, or for measurement of total inorganic mercury (mercury and inorganic mercury compounds) when it is known that no elemental mercury is used in the workplace and that no mercury vapour is produced in the processes carried out, use the procedure described in ISO 17733, if desired, to collect and analyse samples.

10.1.2 Consideration of temperature and pressure effects

10.1.2.1 Effect of temperature and pressure on volume flow rate

Refer to the manufacturer's instructions to determine if the indicated volume flow rate of the flow meter (8.1.5) is dependent upon temperature and pressure. Consider whether the difference between the atmospheric temperature and pressure at the time of calibration of the flow meter and during sampling, is likely to be great enough to justify applying a correction to take this into account, e.g. if the error could be greater than ± 5 %. If a correction is necessary, measure and record the atmospheric temperature and pressure at which the calibration of the flow meter was checked (see 8.1.5), and measure and record the atmospheric temperature and pressure at the start and at the end of the sampling period (10.4.1 and 10.4.3). Then calculate a temperature and pressure corrected volume flow rate following the procedure given in A.1.

10.1.2.2 Expression of results

Consider whether it is necessary to recalculate mercury in air concentrations to reference conditions (such as in high altitude situations). If so, measure and record the atmospheric temperature and pressure at the start and at the end of the sampling period (10.4.1 and 10.4.3) and follow the procedure given in A.2 to apply the necessary correction to the mercury in air concentrations calculated in 12.2.1.

NOTE The concentration of mercury in air is generally stated for actual environmental conditions (temperature, pressure) at the workplace during the sampling period.

10.1.3 Volume flow rate

Select a flow rate within the range recommended by the manufacturer of the sorbent tube (normally between 100 ml min^{-1} and 1 000 ml min^{-1}).

10.1.4 Sampling period

10.1.4.1 Select a sampling period that is appropriate for the measurement task (see 9.4), but ensure that it is long enough to enable mercury to be determined with acceptable uncertainty (see 3.3.5) at levels of industrial hygiene significance. For example, estimate the minimum sampling time required to ensure that the amount of mercury collected is above the lower limit of the working range of the analytical method when it is present in the test atmosphere at a concentration of 0,1 times its limit value, using the following equation:

$$t_{\text{min}} = \frac{m_{\text{lower}}}{q_V \times 0{,}1 \times \rho_{\text{LV}}}$$

where

t_{min} is the minimum sampling time, in minutes;

m_{lower} is the lower limit of the analytical range, in nanograms;

q_V is the design flow rate of the sampler, in millilitres per minute;

ρ_{LV} is the limit value, in milligrams per cubic metre.

10.1.4.2 The sampling time shall not be less than 15 min when the sorbent tube is used at a flow rate of 100 ml min^{-1}.

10.1.4.3 Sample handling

To minimize the risk of damage or contamination, only handle sorbent tubes (8.1.1) and quartz fibre filters (8.1.3), if used, in a clean area where the concentration of mercury in air minimal, and only handle quartz fibre filters using flat-tipped forceps (8.1.6.3).

10.2 Preparation for sampling

10.2.1 Preconditioning of sorbent tubes

Take out the required number of sorbent tubes (8.1.1) from their sealed glass tubes. Precondition the sorbent tubes prior to sampling by heating to 700 °C in the mercury analyser and passing air through them at a flow rate of 500 ml min^{-1} for 2 min to reduce the mercury blank. Replace each sorbent tube in its stoppered glass tube and label the glass tube uniquely.

Preconditioning of sorbent tubes to reduce the mercury blank may be done automatically by commercial instruments.

10.2.2 Setting the volume flow rate

Perform the following in a clean area, where the concentration of mercury in air is minimal.

Remove each sorbent tube (8.1.1) from its storage tube and connect it to a sampling pump (8.1.4) using flexible tubing (8.1.6.1), ensuring that no leaks can occur. Switch on the sampling pump, attach the flow meter (8.1.5) to the sorbent tube so that it measures the flow through the inlet orifice, and set the required volume flow rate (see 10.1.3). Switch off the sampling pump, replace each sorbent tube in its storage tube and close with a butyl rubber stopper to prevent contamination during transport to the sampling position.

If necessary, allow the sampling pump operating conditions to stabilize before setting the volume flow rate (refer to the manufacturer's instructions).

10.2.3 Field blanks

Retain as blanks, one unused sorbent tube (10.2.1) from each batch of ten prepared, subject to a minimum of two. Treat these in the same manner as those used for sampling with respect to storage and transport to and from the sampling position, but draw no air through them.

10.2.4 Sorbent tube and prefilter assemblies

If required, construct sorbent tube and prefilter assemblies (see 8.1.2).

10.3 Sampling position

10.3.1 Static (area) sampling

10.3.1.1 If static sampling is carried out to assess the exposure of a worker in a situation where personal sampling is not possible (e.g. due to the need to sample in confined spaces), position a sorbent tube (10.2.1) or sorbent tube and prefilter assembly (10.2.4) in the immediate vicinity of the worker and at breathing height. If in doubt, take the sampling position to be the point where the risk of exposure is considered to be greatest.

10.3.1.2 If static sampling is carried out to assess the background level of mercury in the workplace, select a sampling position that is sufficiently remote from the work processes, such that results will not be directly affected by mercury from emission sources.

10.3.1.3 If static sampling is carried out to characterize the concentration of mercury in the working environment, select a number of sampling points systematically to cover the workplace.

NOTE This working environment measurement employing static sampling has been performed in some countries by regulation, comparing the measured concentration values statistically with a specific value which is established by an authority, e.g. an administrative control level (ACL) by the Japanese government (see http://www.jicosh.gr.jp/english/index.html).

10.3.2 Personal sampling

Position the sorbent tube (10.2.1) or sorbent tube and prefilter assembly (10.2.4) in the worker's breathing zone, as close to the mouth and nose as is reasonably practicable, e.g. fastened to the worker's lapel or collar. Attach the sampling pump to the worker in a manner that causes minimum inconvenience, e.g. to a belt (8.1.6.2) around the waist or place it in a convenient pocket.

10.4 Collection of samples

10.4.1 When ready to begin sampling, switch on the sampling pump. Record the time and volume flow rate at the start of the sampling period. If the sampling pump is fitted with an integral timer, check that this is reset to zero. If appropriate (see 10.1.2), measure the atmospheric temperature and pressure at the start of the sampling period using the thermometer and barometer, and record the measured values.

Integral timers built into sampling pumps can be imprecise and should only be used to provide evidence that the sampler has been operating properly throughout the sampling period (see 10.4.3).

If the temperature or pressure at the sampling position is different from that where the volume flow rate was set (see 10.2.2), the volume flow rate could change and it might need to be re-adjusted before sampling.

10.4.2 Monitor the performance of the pumps frequently, a minimum of once every two hours. Measure the flow rate using the flow meter (8.1.5) and record the measured value. Terminate sampling and consider the sample to be invalid if the flow rate is not maintained to within ± 5 % of the nominal value throughout the sampling period.

10.4.3 At the end of the sampling period (see 10.1.4), record the time. Check the malfunction indicator and/or the reading on the integral timer, if fitted, and consider the sample to be invalid if there is evidence that the sampling pump was not operating properly throughout the sampling period. Measure the volume flow rate at the end of the sampling period using the flow meter (8.1.5), and record the measured value. If appropriate (see 10.1.2), measure the atmospheric temperature and pressure at the end of the sampling period using the thermometer and barometer, and record the measured values.

10.4.4 Carefully record the sample identity and all relevant sampling data (see Clause 14). Calculate the mean volume flow rate by averaging the volume flow rates at the start and at the end of the sampling period and, if appropriate (see 10.1.2), calculate the mean atmospheric temperature and pressure. Calculate the volume of air sampled, in litres, at atmospheric temperature and pressure, by multiplying the mean flow rate in litres per minute by the duration of the sampling period in minutes.

10.5 Transportation of samples

10.5.1 Disassemble the sorbent tube and prefilter assemblies, if used, and discard the prefilters. Place each sorbent tube in a labelled glass tube and close it with a butyl rubber stopper. Follow the same procedure for the blanks (10.2.3).

10.5.2 Transport the samples (10.5.1) to the laboratory in a container which has been designed to prevent damage to the samples in transit and which has been labelled to assure proper handling.

10.5.3 Follow sampling chain of custody procedures to ensure sample traceability. Ensure that the documentation which accompanies the samples is suitable for a “chain of custody” to be established (see, for example, ASTM D 4840-99 [22]).

10.5.4 It is recommended that the samples be analysed within four weeks of sampling.

11 Analysis

11.1 Set up the analytical instrument following the manufacturer's instructions.

11.2 Calibrate the instrument using the mercury vapour generation procedure outlined below:

a) Insert a small amount of metallic mercury into a suitable glass container, as shown in Figure B.4, or a commercially available mercury gas box.

b) Calculate the concentration of saturated mercury vapour in the air from the equation of state, assuming ideal gas behaviour, using the following equation:

$$\rho = \frac{2{,}412 \times p}{T}$$

where

ρ is the concentration of mercury in air, in grams per litre;

2,412 is the necessary conversion factor, at normal temperature and pressure;

p is the saturated vapour pressure of mercury, in pascals;

T is the temperature, in kelvins.

c) Withdraw a known volume of the saturated mercury vapour using a gas-tight microsyringe, inject it into the standard injecting port of the instrument and initiate the analysis.

For instruments that do not have a standard injecting port, inject the mercury vapour into a sorbent tube whilst drawing air through it using a sampling pump. Then install the tube in the instrument and initiate the analysis.

d) Calculate the mass of mercury injected into the instrument from the injection volume and the concentration of saturated mercury vapour.

e) Repeat c) and d) using a minimum of five different injection volumes to cover a suitable range of masses of mercury and calculate a calibration function.

11.3 Remove each sample sorbent tube from its glass storage tube, immediately install it in the analytical instrument and initiate the analysis.

11.4 Calculate the amount of mercury in each sample using the calibration function.

12 Expression of results

12.1 Calculation of the air sample volumes

Calculate the mean volume flow rate by averaging the measurements taken at the start and end of the sampling period. If appropriate, (see 10.1.2.1), calculate the mean atmospheric temperature and pressure by averaging the measurements taken at the start and end of the sampling period, and apply a temperature and pressure correction to the mean volume flow rate. Follow the guidance given in A.1. Then calculate the volume of the air sampled, in litres, by multiplying the mean volume flow rate, in litres per minute, by the duration of the sampling period, in minutes.

12.2 Calculation of airborne mercury concentrations

12.2.1 Calculate the mass concentration of mercury in the air samples, at ambient conditions, using the equation:

$$\rho_{\mathrm{Hg}} = \frac{(m_{\mathrm{Hg}_1} - m_{\mathrm{Hg}_0})}{V} \times 1000$$

where

ρ_{Hg} is the calculated mass concentration of mercury in the air sample, in milligrams per cubic metre, at ambient conditions;

m_{Hg_0} is the mass of mercury in the sample sorbent tube, in nanograms;

m_{Hg_1} is the average mass of mercury in the blank sorbent tubes, in nanograms;

V is the volume, in litres, of the air sample (see 12.1).

12.2.2 If it is necessary to recalculate mercury in air concentrations to reference conditions (see 10.1.2.2), calculate the mean atmospheric temperature and pressure by averaging the measurements taken at the start and end of the sampling period, and apply a temperature and pressure correction to mercury in air concentrations calculated in 12.2.1 using the equation given in A.2.

13 Method performance

13.1 Detection and quantification limits

Using CVAAS, the method detection limit and quantification limit for mercury (defined as three times and ten times the standard deviation of a blank determination, respectively) have been determined [23] to be 0,003 ng and 0,01 ng, respectively, for samples of mercury vapour collected on sorbent tubes containing 80 mg of porous gold-coated diatomaceous earth. For the minimum air sample volume of 5 l, this corresponds to airborne mercury concentrations of 0,000 6 μg m^{-3} and 0,00 2 μg m^{-3}, respectively.

13.2 Upper limits of the analytical range

The upper limit of the useful analytical range is governed by the linear dynamic range of the spectrometer.

NOTE The upper limit of the analytical range of one commercial mercury analyser is 1 µg of mercury [23]. (The upper limit of the analytical range cited is for one particular model of instrument [15]).

13.3 Blank values

Gold-coated diatomaceous earth does not generally contain mercury at measurable concentrations. When the sorbent tubes are preconditioned prior to use, as described in 10.2.1, the blank value of mercury is negligible.

13.4 Bias and precision

13.4.1 Analytical bias

Laboratory experiments have shown that the analytical method does not exhibit significant bias. The mean analytical recovery has been determined [2] to be 98 % for sorbent tubes containing 80 mg of gold-coated diatomaceous earth dosed in the range 0,02 µg to 1 µg of mercury.

13.4.2 Analytical precision

The component of the coefficient of variation of the method that arises from analytical variability, CV (analysis), is dependent on a number of factors, including the analytical instrumentation used. Laboratory experiments have been carried out to obtain figures of merit for CV (analysis). Using CVAAS, the CV (analysis) has been determined [2] to be 0,6 % for sorbent tubes containing 80 mg of gold-coated diatomaceous earth dosed with 0,05 µg to 0,2 µg of mercury.

13.5 Effects on sampler performance

13.5.1 Effect of concentration and time on sampler performance

Laboratory experiments have been performed to determine the effect of concentration and time on the performance of sorbent tubes containing 80 mg of gold-coated diatomaceous earth. These experiments were carried out by sampling mercury vapour generated at concentrations between 0,001 mg m^{-3} and 0,015 mg m^{-3} at a temperature of 20 °C and a relative humidity of 50 %. Results demonstrated that the effect of exposure concentration and time is negligible for sampling periods up to 8 h.

13.5.2 Effect of atmospheric temperature and humidity on sampler performance

Laboratory experiments have been performed to determine the effect of atmospheric temperature and humidity on the performance of sorbent tubes containing 80 mg of gold-coated diatomaceous earth. These experiments were carried out by sampling mercury vapour at a concentration of 0,005 mg m^{-3} at temperature extremes of 5 °C and 40 °C and relative humidity extremes of 20 % and 70 %. Results demonstrated that the effect of temperature and humidity is negligible within these extremes.

13.6 Collection efficiency, breakthrough volume and sampling capacity of sorbent tubes

Laboratory experiments have shown that the collection efficiency of sorbent tubes is close to 100 %. Breakthrough was found to be less than 1 % when sampling mercury vapour at a concentration of 0,05 mg m^{-3} for 1,5 h using an elevated flow rate of 500 ml min^{-1} at a temperature of 20 °C and a relative humidity of 50 %. The breakthrough volume is therefore greater than 54 l for mercury vapour at a concentration of 0,05 mg m^{-3}. This corresponds to a sampling capacity of at a maximum 2,7 µg of mercury vapour.

13.7 Storage stability

Laboratory experiments have shown [6] that samples of mercury vapour collected using pumped sorbent tubes are stable for at least six months when the tubes are sealed with their plastic caps.

13.8 Interferences

13.8.1 In the chloralkali process, mercury vapour commonly co-exists in the atmosphere with chlorine. Chlorine can react with mercury vapour in the air to form particulate mercuric chloride [18] and, therefore, this procedure is unsuitable for making accurate measurements of mercury vapour in chloralkali works. However, the particulate mercuric chloride formed may be collected separately on a quartz prefilter, analysed by the method prescribed in ISO 17733 and the two results combined to obtain an estimate of total inorganic mercury in the atmosphere of chloralkali works.

13.8.2 Gaseous organo-mercury compounds are collected by diatomaceous earth and can therefore cause a positive interference on the measurement of mercury vapour in the air. Some collected organo-mercury compounds decompose on the sample tube when it is preheated to 300 °C and are determined as mercury vapour in the normal manner. Others are released from the sample tube when it is heated to 300 °C, but are then retained on the trapping tube, which is maintained at a lower temperature of 150 °C. Such organo-mercury compounds will also be determined as mercury vapour by this method. However, organo-mercury compounds that are released from the sample tube when it is preheated to 300 °C, but which then pass through the trapping tube at 150 °C, will not interfere. See Reference [24].

13.8.3 Any compound with the same absorption wavelength as mercury (253,7 nm) can cause interference in CVAAS. Some volatile organic compounds (e.g. hexane, benzene, toluene, acetone, carbon tetrachloride, isopropyl alcohol, etc.) absorb at this wavelength and are therefore potential analytical interferences. These compounds are expected to be retained on gold-coated diatomaceous earth during sample collection and they could therefore cause interference in the determination of mercury vapour by single-amalgam CVAAS [2]. However, the double amalgam method employed in this International Standard is an effective means of measuring mercury vapour in the presence of volatile organic compounds. The excellent efficiency with which mercury is separated from volatile organic compounds is illustrated in Figure B.3. This shows that only a mercury peak is detected in the presence of a volatile organic compound (hexane). CVAFS does not suffer from interference by volatile organic compounds that absorb at 253,7 nm, since they do not re-emit at this wavelength [25].

14 Test report

The test report shall contain the following information:

a) a statement to indicate the confidentiality of the information supplied, if appropriate;

b) a complete identification of the air sample, including the date of sampling, the place of sampling, the type of sample (personal or static), either the identity of the individual whose breathing zone was sampled (or other personal identifier) or the location at which the general occupational environment was sampled (for a static sample), a very brief description of the work activities that were carried out during the sampling period, and a unique sample identification code;

c) a reference to this International Standard (ISO 20552);

d) the make and type of sorbent tube and/or sampler used;

e) the make, type and diameter of filter used, if appropriate;

f) the make and type of sampling pump used, and its identification;

g) the make and type of flow meter used, the primary standard against which the calibration of the flow meter was checked, the range of flow rates over which the calibration of the flow meter was checked, and the atmospheric temperature and pressure at which the calibration of the flow meter was checked, if appropriate;

h) the time at the start and at the end of the sampling period, and the duration of the sampling period in minutes;

i) the mean flow rate during the sampling period, in litres per minute;

j) the mean atmospheric temperature and pressure during the sampling period, if appropriate;

k) the volume of air sampled, in litres, at ambient conditions;

l) the name of the person who collected the sample;

m) the time-weighted average mass concentration of mercury vapour and/or particulate inorganic mercury compounds, or total inorganic mercury (mercury and inorganic mercury compounds) found in the air sample (in milligrams per cubic metre, mg m^{-3}), at ambient temperature and pressure, or, if appropriate conditions, adjusted to reference conditions;

n) the analytical variables used to calculate the result, including the concentrations of mercury in the sample and blank solutions, the volumes of the sample and blank solutions, and the dilution factor, if applicable;

NOTE If the necessary data (e.g. the volume of air sampled) are not available to the laboratory for the above calculations to be carried out, the laboratory report may contain the analytical result in micrograms of mercury per sample.

o) the type(s) of instrument(s) used for sample preparation and analysis, and unique identifier(s);

p) the estimated instrumental detection limit, method detection limit and quantification limit under the working analytical conditions; the measurement uncertainty determined in accordance with the GUM [12]; and, if requested by the customer, quality control data;

q) any operation not specified in this International Standard, or regarded as optional;

r) the name of the analyst(s) or other unique identifier(s);

s) the date of the analysis; and

t) any inadvertent deviations, unusual occurrences or other notable observations.

Annex A
(informative)

Temperature and pressure correction

A.1 Temperature and pressure correction for the indicated volume flow rate of flow meters

A.1.1 Bubble flow meters are preferred for measuring the volume flow rate because the readings they give are independent of temperature and pressure. For other flow meters, it might be necessary to apply a correction to the indicated volume flow rate if the temperature and pressure at the time of measurement is different to when the calibration of the flow meter was checked.

A.1.2 A typical example of the need for a temperature and pressure correction is when a constant pressure drop, variable area, flow meter is used to measure the volume flow rate. In this instance, use the following equation to calculate a corrected air sample volume:

$$V_{\text{corr}} = \quad q_V \times t \times \sqrt{\frac{p_1 \times T_2}{p_2 \times T_1}}$$

where

V_{corr} is the calculated corrected air sample volume, in litres;

q_V is the mean volume flow rate, in litres per minute;

t is the duration of the sampling period, in minutes;

p_1 is the atmospheric pressure, in kilopascals, during calibration of the flow meter (see 8.1.5);

p_2 is the mean atmospheric pressure, in kilopascals, during the sampling period;

T_1 is the temperature, in kelvin, during calibration of the flow meter (see 8.1.5);

T_2 is the mean temperature, in kelvin, during the sampling period.

A theoretical calculation shows that a 5 % deviation in the air sample volume at the reference atmospheric pressure of 101,3 kilopascal occurs at 91,9 kPa and 112,2 kPa. Both these values are outside the normal weather conditions at sea level, but this pressure difference corresponds to an altitude change of about 800 m ($-$0,1 kPa $\approx$ 8 m increased altitude) at normal atmospheric pressure at sea level. Similarly, a 5 % deviation in the air sample volume at the reference temperature of 293 K occurs at 264 K and 323 K.

A.1.3 Any other flow meter can also require a correction for variation in pressure and temperature. Follow the manufacturer's instructions for such corrections.

A.2 Recalculation of airborne mercury concentrations to reference conditions

If necessary (see 10.1.2.2), recalculate the mercury in air concentrations to reference conditions (e.g. 293 K and 101,3 kPa) using the following equation:

$$\rho_{\text{Hg, corr}} = \rho_{\text{Hg}} \times \frac{(101{,}3 \times T_2)}{(p_2 \times 293)}$$

where

$\rho_{\text{Hg, corr}}$ is the corrected concentration of mercury in the air sample, in milligrams per cubic metre, at reference conditions;

ρ_{Hg} is the concentration of mercury in the air sample, in milligrams per cubic metre, at ambient conditions;

T_2 is the mean temperature, in kelvins, during the sampling period;

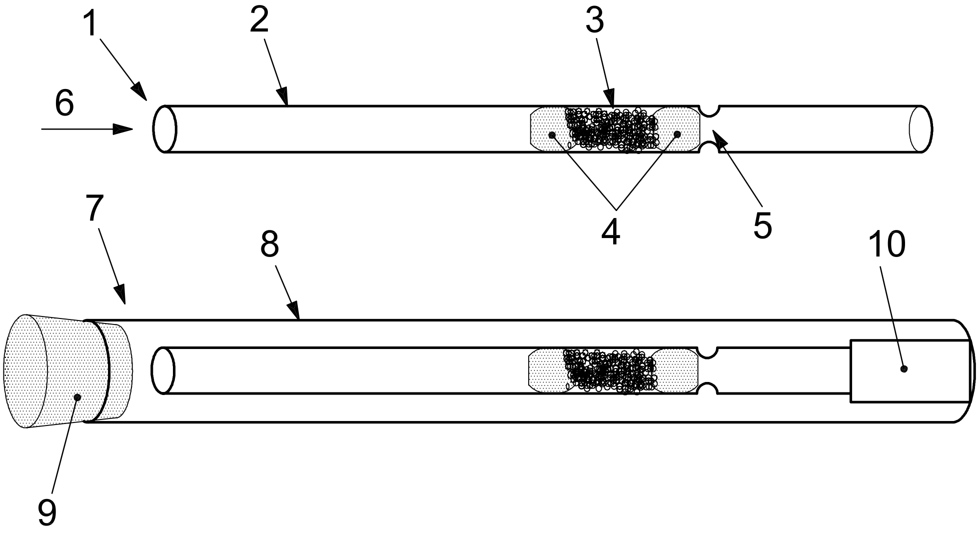
p_2 is the mean atmospheric pressure, in kilopascals, during the sampling period;

293 is the reference temperature, in kelvins;

101,3 is the reference atmospheric pressure, in kilopascals.

Annex B
(informative)

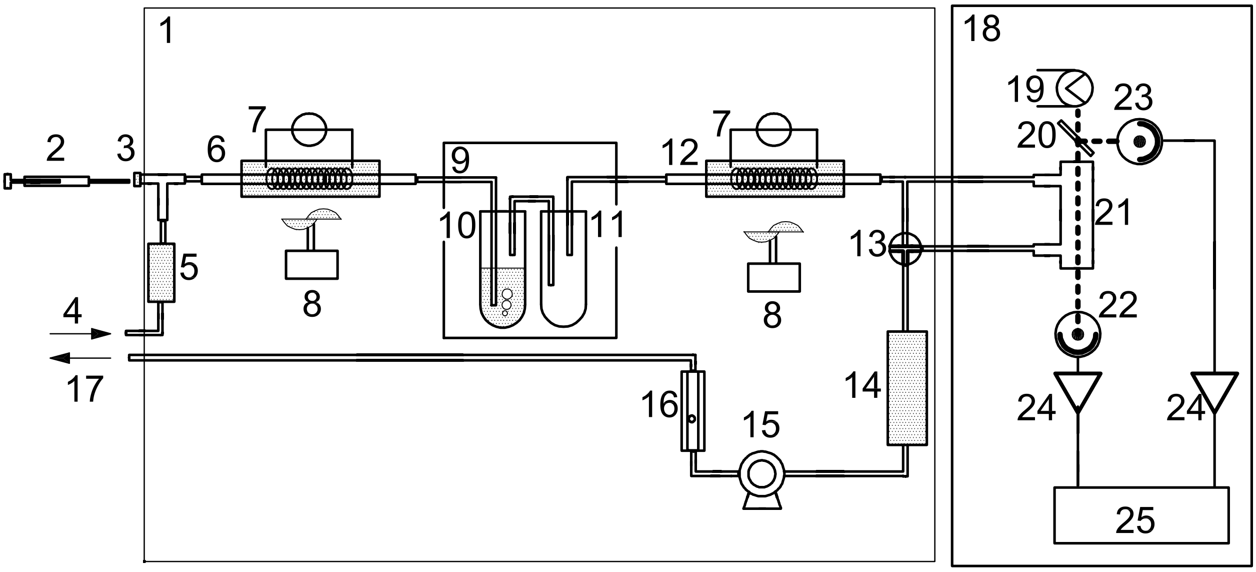
Figures



Key

1	sample tube
2	glass tube
3	mercury sorbent, consisting of gold-coated diatomaceous earth
4	quartz fibre wool
5	dimple to stop reagents
6	direction of air flow (to pump, during sampling, or in instrument, during analysis)
7	carrying container including sample tube
8	glass tube
9	butyl rubber stopper
10	polyethylene tube as sample tube protector

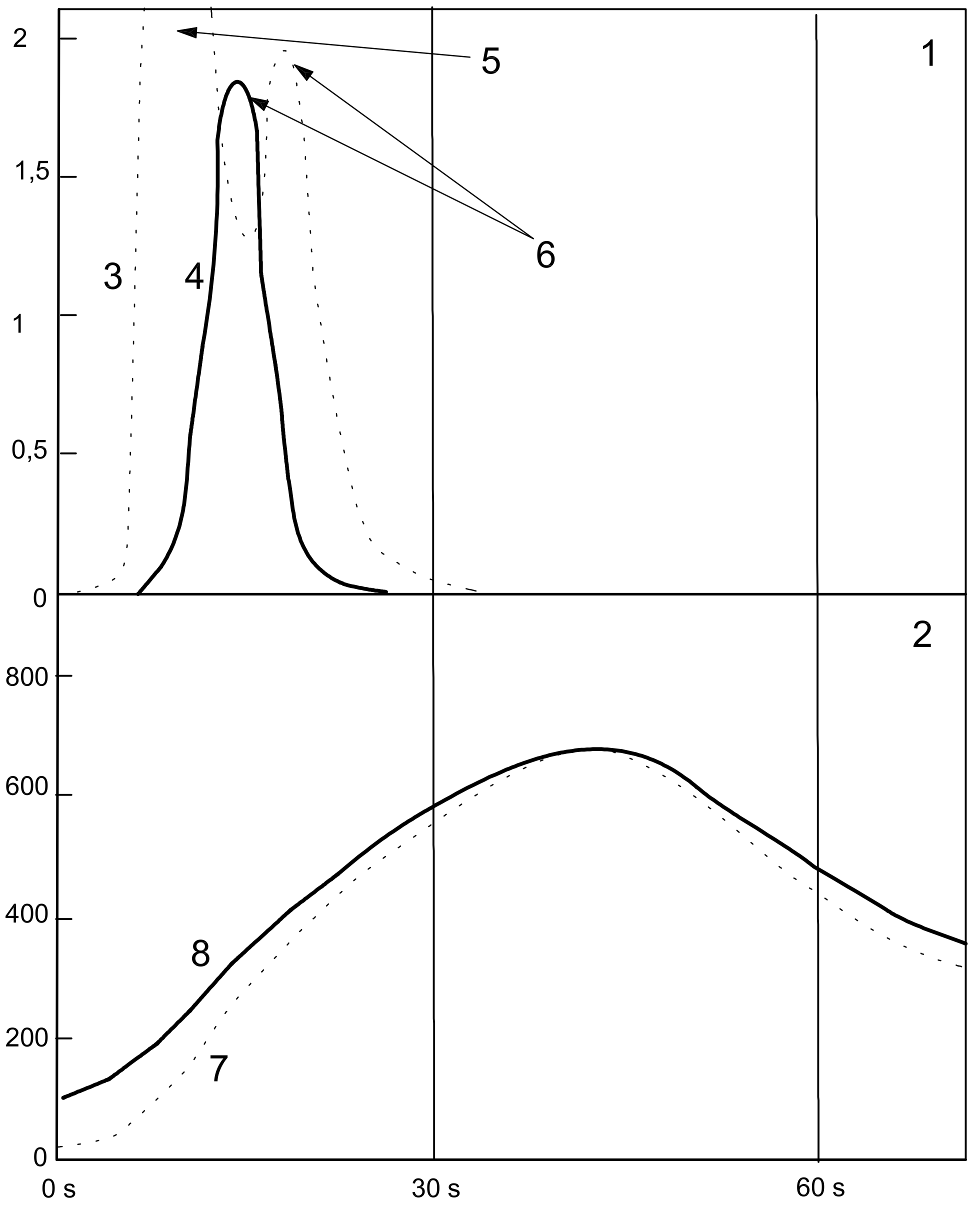
Figure B.1 — Mercury sorbent tube



Key

1	double amalgam sample applicator
2	microsyringe
3	standard injection port
4	carrier gas (air)
5	carrier gas cleaner (gold-coated diatomaceous earth)
6	sampling tube
7	computer controlled heating furnace
8	computer controlled cooling fan
9	cooling chamber
10	gas washer with phosphate buffer
11	dehumidifier
12	second trapping tube
13	three-way valve
14	charcoal filter
15	pump
16	flow meter
17	exhaust
18	CVAAS
19	mercury glow discharge lamp
20	half mirror
21	gas cell
22	photomultiplier (signal)
23	photomultiplier (reference)
24	analogue/digital converter
25	computer

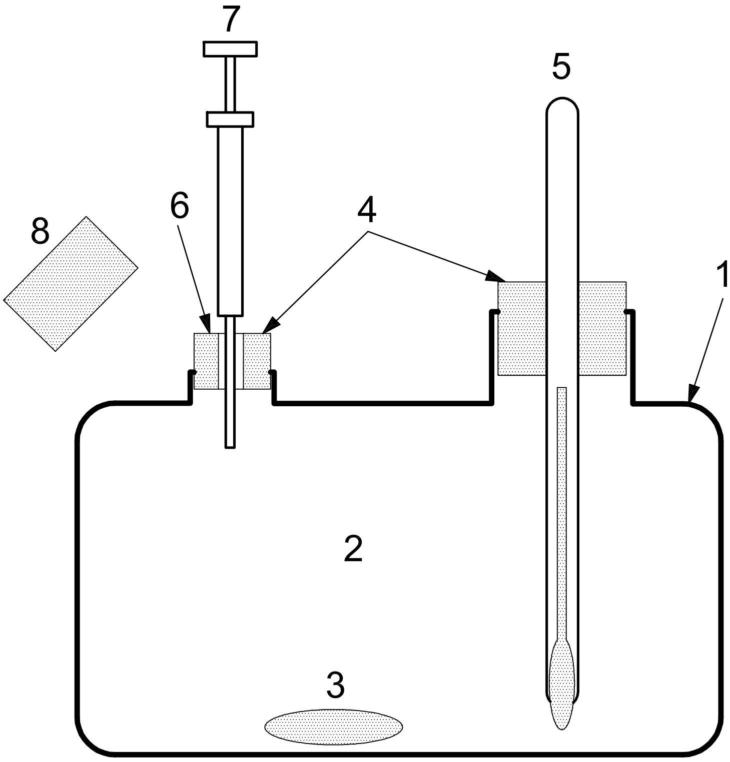
Figure B.2 — Block diagram of a typical double gold amalgam CVAAS mercury analyser



Key

1	observed signal at 254 nm, in arbitrary units
2	temperature of tube heating device, in degrees Celsius, °C
3	CVAAS signal without preheating of the trapping tube (dot curve). Two peaks are observed. The first peak is due to hexane interference, which overlaps the mercury signal.
4	CVAAS signal with preheating of the trapping tube (solid curve). Only a single peak due to mercury is observed, because hexane passes through the trapping tube without being adsorbed onto the heated gold sorbent.
5	hexane peak
6	mercury peak
7	temperature of tube heating device without preheating (dot curve)
8	temperature of tube heating device with preheating (solid curve)

Figure B.3 — Illustration showing elimination of hexane interference using a heated trapping tube



Key

1	glass chamber
2	saturated mercury vapour
3	mercury droplet
4	butyl rubber stoppers
5	thermometer
6	sampling port
7	microsyringe
8	cap to seal the sampling port

Figure B.4 — Mercury vapour standard generation apparatus

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1) Model WA-4 mercury analyzer is the trade name of a product supplied by Nippon Instrument Corporation. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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