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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**

*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*

ISO 20552:2007

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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*.

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## 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 <sup>[1][2][3][4][5][6]</sup> 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<sup>-1</sup> and 1 000 ml min<sup>-1</sup> 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<sup>-1</sup> in workplaces where the concentration of mercury vapour is expected to be lower than 20 µg m<sup>-3</sup>. If the expected concentration of mercury vapour is higher than 20 µg m<sup>-3</sup>, 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] <sup>[7]</sup>

#### 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 <sup>[7]</sup>.

#### 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] <sup>[8]</sup>

#### 3.1.7

##### **limit value**

reference figure for concentration of a chemical agent in air

NOTE An example is the Threshold Limit Value<sup>®</sup> (TLV) for a given substance in workplace air, as established by the ACGIH <sup>[9]</sup>. (Threshold Limit Value<sup>®</sup> 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] <sup>[11]</sup>

### 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)* <sup>[12]</sup> 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)* <sup>[13]</sup>.

## 4 Principle

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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 <sup>[14]</sup>).

## 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 <sup>[1]</sup>.

## 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 <sup>[2][3][15]</sup>. 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 <sup>[16]</sup>).

NOTE Quartz fibre filters have been shown <sup>[17]</sup> not to absorb mercury vapour from the sampled air. They are therefore suitable for use as prefilters. It has been reported <sup>[18]</sup> 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 <sup>[19]</sup>.

**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 <sup>[8]</sup> 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  $\pm 5\%$  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  $\pm 5\%$  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  $\pm 5\%$  from the initial value during the operating time.

If the sampling pump is used outside the range of conditions specified in EN 1232 <sup>[8]</sup>, 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  $\pm 5\%$ .

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  $\pm 2\%$  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.