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

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*Air des lieux de travail — Détermination du mercure et des composés
minéraux de mercure — Méthode par spectrométrie d'absorption
atomique ou spectrométrie de fluorescence atomique de la vapeur
froide*

[ISO 17733:2004](#)

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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 17733 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 presents a method for making valid exposure measurements for mercury and inorganic mercury compounds in use in industry. It 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 a method published by the United Kingdom Health and Safety Executive^[1], which was developed after a thorough review of sampling and analysis techniques available for determination of mercury and inorganic mercury compounds in air^[2]. This procedure has been fully validated and the resulting back-up data are freely available^{[3],[4]}. Similar methods have been published by the United States Occupational Safety and Health Administration (OSHA)^{[5],[6]} and the United States National Institute of Occupational Safety and Health (NIOSH)^[7].

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

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

1 Scope

This International Standard specifies a procedure for determination of the time-weighted average mass concentration of mercury vapour and inorganic mercury compounds in workplace air. Mercury vapour is collected on a solid sorbent using either a diffusive badge or a pumped sorbent tube. Particulate inorganic mercury compounds, if present, are collected on a quartz fibre filter. Samples are analysed using either cold vapour atomic absorption spectrometry (CVAAS) or cold vapour atomic fluorescence spectrometry (CVAFS) after acid dissolution of the mercury collected.

This International Standard is applicable to the assessment of personal exposure to mercury vapour and/or particulate inorganic mercury compounds in air for comparison with long-term or short-term exposure limits for mercury and inorganic mercury compounds and for static (area) sampling.

The lower limit of the working range of the procedure is the quantification limit. This is determined by the sampling and analysis methods selected by the user, but it is typically in the range 0,01 µg to 0,04 µg of mercury (see 13.1). The upper limit of the working range of the procedure is determined by the capacity of the diffusive badge, sorbent tube or filter used for sample collection, but it is at least 30 µg of mercury (see 13.2). The concentration range of mercury in air for which this International Standard is applicable is determined in part by the sampling method selected by the user, but it is also dependent on the air sample volume.

The diffusive badge method is not applicable to measurements of mercury vapour when chlorine is present in the atmosphere, e.g. in chloralkali works, but chlorine does not interfere with the pumped sorbent tube method (see 13.11.1). Gaseous organo-mercury compounds could cause a positive interference in the measurement of mercury vapour (see 13.11.2). Similarly, particulate organo-mercury compounds and gaseous organo-mercury compounds adsorbed onto airborne particles could cause a positive interference in the measurement of particulate inorganic mercury compounds (see 13.11.3).

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 648, *Laboratory glassware — One-mark pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3585, *Borosilicate glass 3.3 — Properties*

ISO 3696:1987, *Water for laboratory use — Specifications and test methods*

ISO 7708:1995, *Air quality — Particle size fraction definitions for health-related sampling*

ISO 8655-1, *Piston-operated volumetric apparatus — Part 1: Terminology, general requirements and user recommendations*

ISO 8655-2, *Piston-operated volumetric apparatus — Part 2: Piston pipettes*

ISO 8655-5, *Piston-operated volumetric apparatus — Part 5: Dispensers*

ISO 8655-6, *Piston-operated volumetric apparatus — Part 6: Gravimetric methods for the determination of measurement error*

EN 13205:2002, *Workplace atmospheres — Assessment of performance of instruments for measurement of airborne particle concentrations*

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

NOTE This definition is taken from the "Council Directive 98/24/EC of 7 April 1998 on the protection of the health and safety of workers from the risks related to chemical agents at work".

[EN 1540^[8]]

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3.1.2

breathing zone

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

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3.1.3

breathing zone

⟨technical⟩ hemisphere (generally accepted to be 0,3 m in radius) extending in front of the human face, centred on the midpoint of a line joining the ears, whose base is a plane through this line, the top of the head and the larynx

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

NOTE 2 Adapted from EN 1540^[8].

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, including storage and transportation of the sample

3.1.6

operating time

⟨of a sampling pump⟩ period during which a sampling pump can be operated at specified flow rate and back-pressure without recharging or replacing the battery

[EN 1232^[9]]

3.1.7**time-weighted average concentration
TWA concentration**

concentration of a chemical agent in the atmosphere, averaged over the reference period

NOTE A more detailed discussion of TWA concentrations has been published by the American Conference of Government Industrial Hygienists (ACGIH)^[10].

3.1.8**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^[10].

3.1.9**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^[10].

3.1.10**workplace**

defined area or areas in which work activities are carried out

[EN 1540^[8]]

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3.2 Particle size fraction definitions**3.2.1****inhalable convention**

target specification for sampling instruments when the inhalable fraction is of interest

[ISO 7708]

3.2.2**inhalable fraction**

mass fraction of total airborne particles which is inhaled through the nose and mouth

NOTE The inhalable fraction depends on the speed and direction of air movement, on breathing rate and other factors.

[ISO 7708]

3.2.3**total airborne particles**

all particles surrounded by air in a given volume of air

NOTE Because all measuring instruments are size-selective to some extent, it is often impossible to measure the concentration of total airborne particles.

[ISO 7708]

3.3 Sampling definitions

3.3.1

diffusive badge

dosimeter

badge-type diffusive sampler

passive badge

diffusive sampler in which the gas or vapour passes to the sorbent by permeation through a thin solid membrane or diffusion across a porous membrane

NOTE The cross-sectional area of a diffusive badge is large in relation to the internal air gap.

3.3.2

diffusive sampler

passive sampler

device which is capable of taking samples of gases or vapours from the atmosphere at a rate controlled by a physical process such as gaseous diffusion through a static air layer or permeation through a membrane, but which does not involve the active movement of air through the sampler

[EN 838^[11]]

3.3.3

personal sampler

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

[EN 1540^[8]]

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3.3.4

personal sampling

process of sampling carried out using a personal sampler

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[EN 1540^[8]]

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3.3.5

sampler

device for collecting airborne particles

NOTE Instruments used to collect airborne particles are frequently referred to by a number of other terms, e.g. sampling heads, filter holders, filter cassettes, etc.

3.3.6

pumped sorbent tube

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^[12]]

3.3.7

static sampler

area sampler

device, not attached to a person, that samples air in a particular location

3.3.8

static sampling

area sampling

process of air sampling carried out using a static sampler

3.4 Analytical definitions

3.4.1

blank solution

solution prepared by taking a reagent blank, laboratory blank or field blank through the same procedure used for sample preparation

3.4.2

calibration blank solution

calibration solution prepared without the addition of any working standard solution

NOTE The concentration of mercury in the calibration blank solution is taken to be zero.

3.4.3

calibration solution

solution prepared by dilution of the working standard solution, containing mercury at a concentration that is suitable for use in calibration of the analytical instrument

NOTE The technique of matrix-matching is normally used when preparing calibration solutions.

3.4.4

field blank

sorbent capsule, filter or sorbent tube that is taken through the same handling procedure as a sample, except that it is not used for sampling

NOTE For the purposes of this International Standard, the field blank is loaded into a diffusive badge, sampler or sorbent tube holder, transported to the sampling site and then returned to the laboratory for analysis.

3.4.5

laboratory blank

unused sorbent capsule, filter or sorbent tube, taken from the same batch used for sampling, that does not leave the laboratory

3.4.6

linear dynamic range

range of concentrations over which the calibration curve for mercury is linear

NOTE The linear dynamic range extends from the detection limit to the onset of calibration curvature.

3.4.7

matrix interference

matrix effect

non-spectral interference

interference of a non-spectral nature caused by a difference between the matrices of the calibration and test solutions

3.4.8

matrix-matching

technique used to minimize the effect of matrix interferences on analytical results, involving the preparation of calibration solutions in which the concentrations of acids and other major solutes are matched with those in the test solutions

3.4.9

reagent blank

combination of all reagents used in sample dissolution, in the same quantities used for preparation of laboratory blank, field blank and sample solutions

3.4.10

sample dissolution

process of obtaining a solution containing mercury from a sample, which might or might not involve complete dissolution of the sample

3.4.11

sample preparation

all operations carried out on a sample, after transportation and storage, to prepare it for analysis, including transformation of the sample into a measurable state, where necessary

3.4.12

sample solution

solution prepared from a sample by the process of sample dissolution

NOTE A sample solution might need to be subjected to further operations, e.g. dilution, in order to produce a test solution that is ready for analysis.

3.4.13

stock standard solution

solution, used for preparation of the calibration solutions, containing mercury at a certified concentration that is traceable to national standards

3.4.14

test solution

blank solution or sample solution that has been subjected to all operations, including dilution, required to bring it into a state in which it is ready for analysis

NOTE The blank test solution is the blank solution and the sample test solution is the sample solution, if these solutions are not subjected to any further operations before analysis.

3.4.15

working standard solution

solution, prepared by dilution of the stock standard solution, that contains mercury at a concentration that is better suited to preparation of calibration solutions than the concentration of mercury in the stock standard solution

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3.5 Statistical terms

3.5.1

analytical recovery

ratio of the mass of analyte measured when a sample is analysed to the known mass of analyte in that sample

NOTE It is expressed as a percentage.

3.5.2

bias

consistent deviation of the measured value from the value of the air quality characteristic itself, or the accepted reference value

[ISO 6879^[13]]

3.5.3

overall uncertainty

(of a measuring procedure or of an instrument) quantity used to characterize as a whole the uncertainty of a result given by an apparatus or measuring procedure

NOTE It is expressed, as a percentage, by a combination of bias and precision, usually according to the formula:

$$\frac{|\bar{x} - x_{\text{ref}}| + 2s}{x_{\text{ref}}} \times 100$$

where

\bar{x} is the mean value of results of a number (n) of repeated measurements;

x_{ref} is the true or accepted reference value of the measurement;

s is the standard deviation of the measurements.

[EN 482^[14]]

3.5.4

precision

closeness of agreement between independent test results obtained under stipulated conditions

[ISO 6879^[13]]

3.5.5

true value

value which characterizes a quantity perfectly defined in the conditions which exist when that quantity is considered

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

[ISO 3534-1^[15]]

3.5.6

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

[VIM^[16]]

NOTE 1 The parameter might 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 can be evaluated from the statistical distribution of the results of 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 GUM^[17] refers to these different cases as Type A and Type B evaluations of uncertainty, respectively.

4 Principle

Mercury vapour is collected either actively or passively. Active sampling involves drawing a known volume of air through a sorbent tube using a pump, whilst passive sampling relies upon the principle of controlled diffusion into a badge. In both the pumped and diffusive sampling methods, mercury vapour entering the sampling device is collected on a proprietary solid sorbent that is widely known as Hydrar (see 8.1.2) but is currently marketed as Anasorb C 300.

Different sampling methods are used depending on the specific application.

- a) If it is known that no particulate inorganic mercury compounds are present in the test atmosphere, mercury vapour is collected using a diffusive badge or by drawing a known volume of air through a sorbent tube using a pump.
- b) If it is known that no mercury vapour is present in the test atmosphere, particulate inorganic mercury compounds are collected by drawing a known volume of air through a quartz fibre filter mounted in a sampler designed to collect the inhalable fraction of airborne particles, as defined in ISO 7708, using a pump.

- c) A pumped sorbent tube is also used for sampling air that contains both mercury vapour and particulate inorganic mercury compounds. The sorbent tube, which collects mercury vapour, is preceded by a quartz fibre filter to collect particulate inorganic mercury compounds, unless these do not make up a significant proportion (e.g. > 10 %) of the total inorganic mercury (mercury vapour and particulate inorganic mercury compounds) present in the test atmosphere (see 10.1.3).

After sampling, the sorbent and/or filter are treated with 2 ml of concentrated nitric acid and 2 ml of concentrated hydrochloric acid and heated in a thermostatically-controlled water bath at 50 °C for 1 h to dissolve the collected mercury. Sample solutions are mixed with tin(II) chloride solution in a continuous flow, flow injection, or discrete injection cold-vapour generation system. Mercury vapour is formed by reduction of divalent mercury ions and this is flushed by a stream of inert gas into the measurement cell of an atomic absorption spectrometer or an atomic fluorescence spectrometer equipped with a mercury hollow cathode lamp or electrodeless discharge lamp. Absorbance or fluorescence measurements are made at 253,7 nm and analytical results are obtained by the analytical curve technique (see 6.1 of ISO 6955:1982^[18]).

The results may be used for the assessment of workplace exposure to mercury vapour, inorganic mercury compounds or total inorganic mercury (see EN 689^[19]).

5 Interferences

The diffusive badge method is unsuitable for making measurements of mercury vapour when chlorine is present in the atmosphere, e.g. in chloralkali works. However, chlorine does not interfere with the pumped sorbent tube method (see 13.12.1). Gaseous organomercury compounds could cause a positive interference in the measurement of mercury vapour (see 13.12.2). Particulate organomercury compounds contained within airborne particles and gaseous organomercury compounds adsorbed onto airborne particles could cause a positive interference in the measurement of particulate inorganic mercury compounds (see 13.12.3). Spectral interferences are not significant when measuring mercury by CVAAS using the procedure described in this International Standard and they do not occur when measuring mercury by CVAFS (see 13.12.4).

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6 Requirement

The measurement procedure shall comply with any relevant international, european or national standard that specifies performance requirements for procedures for measuring chemical agents in workplace air (e.g. ISO 6879^[13]).

7 Reagents

During the analysis, use only reagents of recognized analytical grade and only water as specified in 7.1.

7.1 Water, complying with the requirements for ISO 3696 grade 2 water (electrical conductivity less than 0,1 mS·m⁻¹ and resistivity greater than 1 MΩ·cm at 25 °C).

It is recommended that the water used be obtained from a water purification system that delivers ultrapure water having a resistivity greater than 18 MΩ·cm.

7.2 Nitric acid (HNO₃), concentrated, $\rho \sim 1,42$ g·ml⁻¹, mass fraction ~ 70 %.

The concentration of mercury shall be less than 0,002 mg·l⁻¹.

WARNING — Concentrated nitric acid is corrosive and oxidizing, and nitric acid fumes are irritant. Avoid contact with the skin or eyes, or inhalation of fumes. Use suitable personal protective equipment (including gloves, face shield or safety glasses, etc.) when working with nitric acid, and carry out sample dissolution with concentrated nitric acid in open vessels in a fume hood.

7.3 Nitric acid, diluted 1:9.

Add approximately 700 ml of water (7.1) to a 1 000 ml one-mark volumetric flask (8.3.1.4). Carefully add 100 ml of concentrated nitric acid (7.2) to the flask and swirl to mix. Allow to cool, dilute to the mark with water, stopper and mix thoroughly.

7.4 Hydrochloric acid (HCl), concentrated, $\rho \sim 1,18 \text{ g}\cdot\text{ml}^{-1}$, mass fraction $\sim 36 \%$.

The concentration of mercury shall be less than $0,002 \text{ mg}\cdot\text{l}^{-1}$.

WARNING — Concentrated hydrochloric acid is corrosive and hydrochloric acid vapour is irritant. Avoid contact with the skin or eyes, or inhalation of the vapour. Use suitable personal protective equipment (including gloves, face shield or safety glasses, etc.) when working with hydrochloric acid. Handle open vessels containing concentrated hydrochloric acid in a fume hood. The vapour pressure of hydrochloric acid is high, therefore beware of pressure build-up in stoppered flasks when preparing hydrochloric acid/water mixtures.

7.5 Nitric acid and hydrochloric acid, diluted 1:1:23.

Add approximately 700 ml of water (7.1) to a 1 000 ml one-mark volumetric flask (8.3.1.4). Carefully add 40 ml of concentrated nitric acid (7.2) and 40 ml of concentrated hydrochloric acid (7.4) to the flask and swirl to mix. Allow to cool, dilute to the mark with water, stopper and mix thoroughly.

NOTE This reagent is only required for analysis of sorbent tube samples.

7.6 Nitric acid and hydrochloric acid, diluted 2:2:21.

Add approximately 700 ml of water (7.1) to a 1 000 ml one-mark volumetric flask (8.3.1.4). Carefully add 80 ml of concentrated nitric acid (7.2) and 80 ml of concentrated hydrochloric acid (7.4) to the flask and swirl to mix. Allow to cool, dilute to the mark with water, stopper and mix thoroughly.

NOTE This reagent is only required for analysis of diffusive badge samples.

7.7 Tin(II) chloride dihydrate ($\text{SnCl}_2\cdot 2\text{H}_2\text{O}$).

The concentration of mercury shall be less than $10 \mu\text{g}\cdot\text{kg}^{-1}$.

Tin(II) chloride of a lower purity may be used if mercury contamination is removed from the tin(II) chloride solution before use by bubbling through nitrogen or clean air.

7.8 Tin(II) chloride solution, $\rho = 10 \text{ g}\cdot\text{l}^{-1}$.

Weigh 10 g of tin(II) chloride (7.7) into a 500 ml beaker (8.3.1.1), add 30 ml of hydrochloric acid (7.4), cover with a watchglass and leave for approximately 15 min until dissolved. Transfer to a 1 000 ml one-mark volumetric flask (8.3.1.4), dilute to the mark with water (7.1), stopper and mix thoroughly. Prepare this solution fresh on the day of use.

7.9 Stock mercury standard solution, $\rho = 1\,000 \text{ mg}\cdot\text{l}^{-1}$.

Use a commercial standard solution with a certified mercury concentration traceable to national standards. Observe the manufacturer's expiration date or recommended shelf life.

WARNING — Mercury compounds are toxic by skin absorption. Take great care when working with solutions containing mercury to avoid skin contamination.

A stock mercury standard solution with a different concentration of mercury may be used, if desired. However, in such circumstances, the volumes of solution used to prepare working mercury standard solution A (7.10) and mercury standard solution B (7.11) shall be adjusted accordingly.