
**Workplace air — Determination of
particulate lead and lead compounds —
Flame or electrothermal atomic absorption
spectrometric method**

*Air des lieux de travail — Dosage du plomb particulaire et des composés
particulaires du plomb — Méthode par spectrométrie d'absorption atomique
dans la flamme ou méthode par spectrométrie d'absorption avec
atomisation électrothermique*

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 8518 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

This second edition cancels and replaces the first edition (ISO 8518:1990), which has been technically revised.

Annexes A and B of this International Standard are for information only.

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Introduction

The health of workers in many industries, e.g. mining, metal refining, battery manufacture, construction, etc., is at risk through exposure by inhalation of particulate lead and lead 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 provides a method for making valid exposure measurements for lead. 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 and workers of metals and metalloids, etc.

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 particulate lead and lead compounds — Flame or electrothermal atomic absorption spectrometric method

1 Scope

1.1 This International Standard specifies flame and electrothermal atomic absorption spectrometric methods for the determination of the time-weighted average mass concentration of particulate lead and lead compounds in workplace air.

1.2 The method is applicable to personal sampling of the inhalable fraction of airborne particles, as defined in ISO 7708, and to static (area) sampling.

1.3 The sample dissolution procedure specifies hot plate or microwave digestion, or ultrasonic extraction (11.2). The use of an alternative, more vigorous dissolution procedure is necessary when it is desired to extract lead from compounds present in the test atmosphere that are insoluble using the dissolution procedures described herein.

1.4 The flame atomic absorption method is applicable to the determination of masses of approximately 1 µg to 200 µg of lead per sample, without dilution [1]. The electrothermal atomic absorption method is applicable to the determination of masses of approximately 0,01 µg to 0,5 µg of lead per sample, without dilution [1].

1.5 The ultrasonic extraction procedure has been validated for the determination of masses of approximately 20 µg to 100 µg of lead per sample, for laboratory-generated lead fume air filter samples [2].

1.6 The concentration range for lead in air for which this procedure is applicable is determined in part by the sampling procedure selected by the user (see 10.1).

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3585:1998, *Borosilicate glass 3.3 — Properties*

ISO 3696:1987, *Water for analytical laboratory use — Specification 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*

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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*

ISO 15202-2:2001, *Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 2: Sample preparation*

EN 13205¹⁾, *Workplace atmospheres — Assessment of performance of instruments for measurement of airborne particle concentrations*

3 Terms and definitions

For the purposes of this International Standard, 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, used or released, including release as waste, by any work activity, whether or not produced intentionally and whether or not placed on the market

[EN 1540]

3.1.2

breathing zone

space around a person's face from where he or she takes his or her breath

NOTE For technical purposes, a more precise definition is as follows: 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; the base of the hemisphere is a plane through this line, the top of the head and the larynx. This definition is not applicable when respiratory protective equipment is used.

[EN 1540]

3.1.3

exposure by inhalation

situation in which a chemical agent is present in air which is inhaled by a person

3.1.4

measuring procedure

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

3.1.5

operating time

period during which a sampling pump can be operated at specified flowrate and back-pressure without recharging or replacing the battery

[EN 1232]

3.1.6

time-weighted average concentration

TWA concentration

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

1) To be published.

NOTE A more detailed discussion of TWA concentrations and their use can be found in [3].

3.1.7

limit value

reference figure for concentration of a chemical agent in air

EXAMPLE Threshold Limit Value[®] (TLV) for a given substance in workplace air, as established by the ACGIH [3].

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 [3].

3.1.9

workplace

defined area or areas in which work activities are carried out

[EN 1540]

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]

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3.2.2

inhalable fraction

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

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

[ISO 7708]

3.2.3

respirable convention

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

[ISO 7708]

3.2.4

respirable fraction

mass fraction of inhaled particles penetrating to the unciliated airways

[ISO 7708]

3.2.5

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 total concentration of airborne particles.

[ISO 7708]

3.3 Sampling definitions

3.3.1

personal sampler

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

[EN 1540]

3.3.2

personal sampling

process of sampling carried out using a personal sampler

[EN 1540]

3.3.3

sampling instrument 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.

3.3.4

static sampling area sampling

process of air sampling carried out in a particular location

3.3.5

static sampler area sampler

device, not attached to a person, used in static sampling

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3.4 Definitions used in analysis

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3.4.1

sample dissolution

process of obtaining a solution containing the analytes of interest from a sample

NOTE This may or may not involve complete dissolution of the sample.

3.4.2

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

3.4.3

sample solution

solution prepared by the process of sample dissolution, but possibly needing to be subjected to further operations in order to produce a test solution that is ready for analysis

3.4.4

test solution

solution prepared by the process of sample dissolution and, if necessary, having been subjected to any further operations required to bring it into a state in which it is ready for analysis

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 results of a measurement process from the true value of the air quality characteristic itself

[ISO 6879]

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 concentration; and

s is the standard deviation of repeated measurements.

[EN 482]

3.5.4**precision**

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

[ISO 6879]

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NOTE Precision is often expressed in terms of the relative standard deviation.

3.5.5**true value**

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

[ISO 3534-1]

NOTE The true value of a quantity is a theoretical concept and, in general, cannot be known exactly (see EN 1540).

4 Principle

4.1 A known volume of air is drawn through a filter to collect particulate lead and lead compounds. For personal sampling, a sampler designed to collect the inhalable fraction of airborne particles is used.

4.2 The filter and collected sample are subjected to a dissolution procedure in order to extract lead. The sample dissolution procedure may use one of three techniques: hot plate digestion, microwave digestion or ultrasonic extraction.

4.3 Sample solutions are analysed for lead content by aspirating into the oxidizing air-acetylene flame of an atomic absorption spectrometer equipped with a lead hollow-cathode lamp or electrodeless discharge lamp. Absorbance measurements are made at 283,3 nm, and analytical results are obtained by the analytical curve technique (see 6.1 of ISO 6955:1982). Potential interference by anions that form precipitates with lead is overcome by the addition of the disodium salt of ethylenediamine tetraacetic acid (EDTA) when necessary.

4.4 For accurate lead determination when the concentration of lead in the solution is low, the analysis may be repeated using electrothermal atomic absorption spectrometry. Aliquots of the test solution are injected into a

graphite furnace, and after drying and sample ashing stages, the sample is atomized electrothermally. Absorbance measurements are made at 283,3 nm with background correction, and results are obtained by the analytical curve technique (see 6.1 of ISO 6955:1982).

4.5 The results may be used for the assessment of workplace exposures to airborne particulate lead (see EN 689).

5 Reactions

In general, the overwhelming majority of particulate lead compounds that are commonly found in samples of workplace air are converted to water-soluble lead ions (Pb^{2+}) by the sample dissolution procedures described in 11.2. However, certain lead compounds, for example lead silicate, might not be dissolved. If necessary, a dissolution procedure employing hydrofluoric acid should be used to dissolve silicate lead. If there is any doubt about the effectiveness of these procedures for the dissolution of particulate lead compounds that may be present in the test atmosphere, then this shall be investigated before proceeding with the analytical method described in clause 11.

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 (e.g. EN 482).

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 and resistivity greater than 0,01 $M\Omega \cdot m$ at 25 °C).

The concentration of lead in the water shall be less than 0,01 $\mu g/ml$.

It is recommended that the water used be obtained from a water purification system that delivers ultrapure water having a resistivity greater than 0,18 $M\Omega \cdot m$ (usually expressed by manufacturers of water purification systems as 18 $M\Omega \cdot cm$).

7.2 Nitric acid (HNO_3), concentrated, $\rho \approx 1,42$ g/ml (about 70 % mass fraction).

The concentration of lead shall be less than 0,01 $\mu g/ml$.

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

7.3 Nitric acid, diluted 1 + 1.

Carefully add 500 ml of concentrated nitric acid (7.2) to 450 ml of water (7.1) in a 2-litre beaker. Swirl to mix, allow to cool and transfer to a 1-litre one-mark volumetric flask (8.6.1.4). Dilute to the mark with water, stopper and mix thoroughly.

7.4 Nitric acid, diluted 1 + 9.

Place approximately 800 ml of water (7.1) in a 1-litre one-mark volumetric flask (8.6.1.4). Carefully add 100 ml of concentrated nitric acid (7.2) to the flask and swirl to mix. Allow to cool, dilute to 1 litre with water and mix thoroughly.

7.5 Hydrofluoric acid (HF), concentrated, $\rho \approx 1,16$ g/ml (about 48 % mass fraction), if required, for digestion of samples containing lead silicates.