



**SLOVENSKI STANDARD**  
**SIST ISO 8518:2023**

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**SIST ISO 8518:2002**

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**Zrak na delovnem mestu - Določevanje svinca in svinčevih spojin v delcih -  
Plamenska in elektrotermijska atomska absorpcijska spektrometrijska metoda**

Workplace air - Determination of particulate lead and lead compounds - Flame and  
electrothermal atomic absorption spectrometric methods

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

**Ta slovenski standard je istoveten z: ISO 8518:2022**

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

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

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

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

This third edition cancels and replaces the second edition (ISO 8518:2001), which has been technically revised.

The main changes are as follows:

- a new [Annex B](#) (informative) has been added concerning sampler wall deposits;
- references and definitions have been updated;
- additional editorial changes have been made.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

The health of workers in many industries, for example, mining, metal refining, battery manufacture, construction, 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 document 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.

During the development of this document, it has been assumed 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 and electrothermal atomic absorption spectrometric methods

## 1 Scope

This document 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.

These methods are typically applicable to personal sampling of the inhalable fraction of airborne particles, as defined in ISO 7708, and to static (area) sampling. It can be applied to other health-related fractions as required.

The sample dissolution procedure specifies hot plate or microwave assisted digestion, or ultrasonic extraction (see 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 (see Clause 5).

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

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<sup>[2]</sup>.

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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3585, *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: Pipettes*

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

ISO 8655-6, *Piston-operated volumetric apparatus — Part 6: Gravimetric reference measurement procedure for the determination of volume*

ISO 13137, *Workplace atmospheres — Pumps for personal sampling of chemical agents — Requirements and test methods*

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

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 17034, *General requirements for the competence of reference material producers*

ISO 18158, *Workplace air — Terminology*

ISO 20581, *Workplace air — General requirements for the performance of procedures for the measurement of chemical agents*

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

### 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

#### 3.1 sample dissolution

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

[SOURCE: ISO 15202-2:2020, 3.1]

#### 3.2 sample solution

solution prepared from a sample by the process of *sample dissolution* (3.1)

Note 1 to entry: A sample solution might need to be subjected to further operations, e.g. dilution, or addition, or both, of an internal standard(s), in order to produce a *test solution* (3.3).

[SOURCE: ISO 15202-2:2020, 3.2]

#### 3.3 test solution

blank solution or *sample solution* (3.2) that has been subjected to all operations required to bring it into a state in which it is ready for analysis

[SOURCE: ISO 15202-2:2020, 3.3, modified — Note 1 to entry has been deleted.]

### 4 Principle

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

**4.2** The sampling substrate and collected sample are subjected to a dissolution procedure in order to extract lead. The sample dissolution procedure can use one of three techniques: hot plate digestion, microwave assisted 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 with background correction (e. g. Zeeman mode or deuterium background correction), and analytical results are obtained by the analytical curve technique. 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 can 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.

**4.5** The results can be used for the assessment of workplace exposures to airborne particulate lead (see ISO 21832<sup>[3]</sup>).

## 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 ( $\text{Pb}^{2+}$ ) by the sample dissolution procedures described in 11.2. However, certain lead compounds, for example, lead-containing silicates, can possibly 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 can 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 applicable requirements of ISO 20581 and with any relevant national standard that specifies performance requirements for procedures for measuring chemical agents in workplace air.

## 7 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water as specified in [7.1](#).

**7.1 Water**, conforming with the requirements for ISO 3696:1987, grade 2 water (electrical conductivity less than 0,1 mS/m and resistivity greater than 0,01  $\text{M}\Omega\cdot\text{m}$  at 25 °C).

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

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  $\text{M}\Omega\cdot\text{m}$  (usually expressed by manufacturers of water purification systems as 18  $\text{M}\Omega\cdot\text{cm}$ ).

**7.2 Nitric acid** ( $\text{HNO}_3$ ), concentrated,  $\rho \approx 1,42 \text{ g}/\text{ml}$  (about 70 % mass fraction).

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

**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.**

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**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. 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. 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, with the density,  $\rho$ , almost equal to 1,14 g/ml (about 38 % mass fraction), if required, for digestion of samples containing lead silicates.

The concentration of lead in the HF shall be less than 0,1  $\mu\text{g/ml}$ .

**WARNING — Concentrated hydrofluoric acid and hydrogen fluoride vapour are extremely toxic and intensely corrosive, and diluted hydrofluoric acid can also cause serious and painful burns that can possibly not be felt until up to 24 h after contact. Avoid exposure by contact with the skin or the eyes, or by inhalation of the vapour. Use of personal protection (for example, impermeable gloves, face shield or safety glasses) is essential when working with concentrated or diluted hydrofluoric acid, and concentrated hydrofluoric acid should be used in a fume hood. It is essential that hydrofluoric acid antidote gel containing calcium gluconate is readily available to workers, both during and for 24 h after use of hydrofluoric acid.**

**7.6 Matrix modifier**,  $\text{NH}_4\text{H}_2\text{PO}_4$ ,  $\text{Mg}(\text{NO}_3)_2$  or  $\text{Pd}(\text{NO}_3)_2$ , or a combination of these, if required, for analysis by electrothermal atomic absorption spectrometry.**7.7 Stock lead standard solution**, 1 000 mg/l of lead.

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

Alternatively, prepare a lead standard solution by one of the following procedures.

- Dissolve 1,598 g  $\pm$  0,001 g of lead(II) nitrate [ $\text{Pb}(\text{NO}_3)_2$ ], previously dried to constant mass at 110 °C and cooled in a desiccator, in 200 ml of 1 + 1 nitric acid (7.3). Quantitatively transfer the solution to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water (7.1), stopper and mix thoroughly. Store in a suitable container, for example, a polypropylene bottle (8.6.2.2), for a maximum period of one year.
- Dissolve 1,000 g  $\pm$  0,001 g of lead wire (99,9 % mass fraction Pb) in 200 ml of 1 + 1 nitric acid (7.3). Quantitatively transfer the solution into a 1 000 ml one-mark volumetric flask, dilute to the mark with water (7.1), stopper and mix thoroughly. Store in a suitable container, for example, a polypropylene bottle (8.6.2.2), for a maximum period of one year.

**7.8 Working lead standard solution**, 1 mg/l of lead, if required, for analysis by electrothermal atomic absorption spectrometry.

Accurately pipette 100  $\mu\text{l}$  of stock lead standard solution (7.7) into a 100 ml one-mark volumetric flask (8.6.1.4). Add 1 ml of concentrated nitric acid (7.2), dilute to the mark with water (7.1), stopper and mix thoroughly. Store in a suitable container, for example, a polypropylene bottle (8.6.2.2), for a maximum period of one month.

**7.9 Hydrogen peroxide ( $\text{H}_2\text{O}_2$ )**, approximately 30 % mass fraction solution, if required, for use in the hot-plate sample digestion method.

The concentration of lead in the hydrogen peroxide solution shall be less than 0,01  $\mu\text{g/ml}$ .