



SLOVENSKI STANDARD

SIST ISO 8518:1996

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

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

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

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

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Reference number
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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.

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.

International Standard ISO 8518 was prepared by Technical Committee ISO/TC 146, *Air quality*.

Annex A of this International Standard is for information only.

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

1 Scope

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

The method is applicable to the determination of masses of approximately 10 µg to 200 µg of lead per sample using the normal procedure without dilution. 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 7.1).

The method is applicable to personal and fixed location sampling.

The normal sample dissolution procedure (8.2.2) is not effective for all lead compounds (see clause 4). The use of an alternative more vigorous dissolution procedure is necessary when lead compounds which are insoluble in normal sample dissolution procedures are present in the test atmosphere (see 10.1).

Anions which give a precipitate with lead interfere, but this interference is overcome by the addition of the disodium salt of ethylenediamine tetraacetic acid (EDTA) when necessary (see 10.2).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3585:1976, *Glass plant, pipeline and fittings — Properties of borosilicate glass 3.3*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 6955:1982, *Analytical spectroscopic methods — Flame emission, atomic absorption, and atomic fluorescence — Vocabulary*.

ISO/TR 7708:1983, *Air quality — Particle size fraction definitions for health-related sampling*.

ISO 8655-1:—¹⁾, *Piston and/or plunger operated volumetric apparatus (POVA) — Part 1: Definitions*.

ISO 8655-2:—¹⁾, *Piston and/or plunger operated volumetric apparatus (POVA) — Part 2: Operating considerations*.

ISO 8655-3:—¹⁾, *Piston and/or plunger operated volumetric apparatus (POVA) — Part 3: Methods of test*.

ISO 8655-4:—¹⁾, *Piston and/or plunger operated volumetric apparatus (POVA) — Part 4: Specifications*.

3 Principle

A known volume of air sample is drawn through a filter to collect particulate lead and lead compounds. The filter and collected sample are then wet-washed using nitric acid and hydrogen peroxide^[1]. Sample solutions are aspirated into the oxidizing air-

1) To be published.

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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 6.1.1 of ISO 6955:1982).

4 Reactions

In general, the overwhelming majority of particulate lead compounds which are commonly found in samples of workplace air are converted to water-soluble lead ions (Pb^{2+}) by the wet-ashing procedure described in 8.2.2. However, certain lead compounds, for example lead silicate, are not dissolved. If there is any doubt about the effectiveness of this procedure for the dissolution of particulate lead compounds which may be present in the test atmosphere, then investigate this before proceeding with the method (see 10.1).

5 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water as specified in (5.1).

5.1 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 $\text{M}\Omega\cdot\text{m}$ at 25 °C).

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

5.2 Nitric acid (HNO_3), concentrated, ρ about 1,42 g/ml, 69 % (m/m) to 71 % (m/m).

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

5.3 Nitric acid, diluted 1 + 9.

Add 100 ml of concentrated nitric acid (5.2) to 500 ml of water (5.1), dilute to 1 litre with water and mix thoroughly.

5.4 Lead standard solution, corresponding to 1000 mg of Pb per litre.

Use these standard solutions at a concentration of 1000 mg/l. Observe the manufacturer's expiry date or recommended shelf life.

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

a) 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 dilute nitric acid (5.3). Quantitatively transfer the solution to a 1000 ml one-mark volumetric flask (6.2.1.5).

Dilute to the mark with dilute nitric acid (5.3), and mix thoroughly.

b) Dissolve 1,000 g \pm 0,001 g of lead wire [99,9 % (m/m) Pb] in 200 ml of nitric acid diluted 1 + 1, prepared by adding 100 ml of concentrated nitric acid (5.2) to 100 ml of water (5.1). Quantitatively transfer the solution into a 1000 ml one-mark volumetric flask (6.2.1.5), dilute to the mark with water (5.1), and mix thoroughly.

NOTE 1 Lead standard solutions prepared according to the instructions in a) and b) may be stored in polypropylene bottles (6.2.2) for a maximum period of one year.

5.5 Hydrogen peroxide (H_2O_2), approximately 30 % (m/m) solution.

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

5.6 Acetylene, cylinder.

5.7 Air, compressed and filtered.

6 Apparatus

6.1 Sampling equipment

6.1.1 Filters, soluble, using the wet-ashing procedure described in 8.2.2, and with a retentivity not less than 99 % for 0,3 μm mass median particle aerodynamic diameters (see subclause 2.1 of ISO/TR 7708:1983).

The concentration of lead shall be less than 0,1 μg per filter.

NOTE 2 Cellulose ester membrane filters of 0,8 μm to 1,2 μm pore size are generally most suitable. Cellulose (paper) filters may have retentivities below 99 % and therefore may be unsuitable. Glass fibre filters will not dissolve, and are therefore also unsuitable.

6.1.2 Filter holders, suitable for collecting the inspirable fraction of airborne particles, as defined in ISO/TR 7708 and compatible with the filter and other sampling equipment used.

NOTES

3 The filter holder used may be designed so that the inspirable fraction of airborne particles is collected specifically on the filter, or it may be designed so that airborne particles which pass through the entry orifice or orifices constitute the inspirable fraction.

4 Filter holders not designed to collect the inspirable fraction of airborne particles may be used when this is necessary to comply with legislation, for example to comply with the requirements of European Council Directive 82/605/CEE[2].

6.1.3 Sampling pumps, with an adjustable flow rate, incorporating a flowmeter and capable of maintaining the selected flow rate (see 7.1.1) to within $\pm 5\%$ of the nominal value throughout the sampling period (see 7.1.2). For personal sampling the pumps shall be capable of being worn without impeding normal work activity.

NOTE 5 A flow-stabilized pump may be required to maintain the flow rate within the specified limits.

6.1.4 Flowmeter, portable, capable of measuring the selected flow rate (see 7.1.1) to within $\pm 5\%$, and calibrated against a primary standard i.e. a flowmeter whose accuracy is established according to national standards.

NOTES

6 The flowmeter incorporated in the pump may be used provided it has adequate sensitivity, but it should be calibrated against a primary standard with a loaded filter holder (7.2.2) in line, and read with the flowmeter vertical if it is of the supported float type. A flowmeter in the pump or elsewhere in the line can be misleading if there are leaks in the sampling train between the filter holder and the flowmeter.

7 A soap bubble flowmeter may be used as a primary standard, provided its accuracy is established according to national standards.

8 If appropriate, note the ambient temperature and pressure at which the flowmeter was calibrated (see 7.1.3).

6.1.5 Rubber or plastics tubing, of a diameter suitable for ensuring a leak-proof fit to both pump and filter holder.

6.1.6 Barometer.

6.1.7 Thermometer, 0 °C to 50 °C, graduated in divisions of 1 °C or better.

6.2 Analytical or laboratory apparatus.

Ordinary laboratory apparatus, and

6.2.1 Glassware, made of borosilicate glass 3.3 complying with the requirements of ISO 3585.

NOTE 9 It is preferable to reserve a set of glassware for analysis of lead by this method, to ensure that problems do not arise from incomplete removal of heavy metal contamination by cleaning (8.1.4).

6.2.1.1 Beakers, of capacities between 50 ml and 150 ml.

6.2.1.2 Watch glasses, to fit the beakers (6.2.1.1).

6.2.1.3 One-mark pipettes, complying with the requirements of ISO 648.

6.2.1.4 Measuring cylinder, of capacity 100 ml.

6.2.1.5 One-mark volumetric flasks, of capacities between 10 ml and 1000 ml, complying with the requirements of ISO 1042.

6.2.2 Polypropylene bottles, of capacities between 100 ml and 1 litre.

6.2.3 Piston-operated volumetric apparatus, complying with the requirements of ISO 8655-1 to ISO 8655-4. Pipetters, as an alternative to one-mark pipettes for the preparation of calibration solutions (see 8.3.2) and dilution of over-range samples (see 8.5.4), and dispensers for dispensing acids (see 8.2.2 and 8.2.3).

6.2.4 Hot plate, thermostatically controlled, capable of maintaining a surface temperature of approximately 140 °C.

NOTE 10 The efficiency of thermostating of hot plates is sometimes deficient, and the surface temperature can also vary considerably with position on a hot plate with a large surface area. It may therefore be useful to characterize the performance of the hot plate before use.

6.2.5 Atomic absorption spectrometer, equipped with a lead hollow cathode lamp or an electrodeless discharge lamp and a burner for use with an air-acetylene flame.

6.2.6 Analytical balance, capable of weighing to the nearest 0,1 mg.

7 Sampling

7.1 Sampling procedure

7.1.1 Collection characteristics and flow rate

Select a sampling procedure suitable for collecting the inspirable fraction of airborne particles as defined in ISO/TR 7708. In particular, select a flow rate at which the filter holder (6.1.2) exhibits the required collection characteristics. A typical flow rate is 2 l/min.

NOTE 11 A sampling procedure in which the inspirable fraction of airborne particles is not collected may be used when this is necessary to comply with legislation, for example to comply with the requirements of European Council Directive 82/605/CEE^[2].

7.1.2 Sampling time

Select a sampling period long enough to ensure that the amount of lead collected is adequate to enable lead-in-air concentrations to be determined at the required level. However, the sampling time shall not be longer than a representative work period and not

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long enough to risk overloading the filter with particulate matter.

NOTE 12 In calculating the minimum sampling time required it is necessary to consider the selected flow rate (see 7.1.1) and the lower limit of the recommended analytical working range of the method i.e. 10 µg of lead per filter (see 9.3.1). For example, to determine concentrations of lead in air with a lower limit for the working range of 0,03 mg/m³, the minimum sampling time at a flow rate of 2 l/min is approximately 3 h.

7.1.3 Temperature and pressure effects

7.1.3.1 The indicated flow rate of certain types of flowmeter is dependent upon temperature and pressure. Therefore, refer to the manufacturer's directions for the particular flowmeter used, and consider whether it is necessary to make a correction to take into account any difference between the temperature and pressure at the time of calibration of the flowmeter and at the time of sampling. Make such a correction if it is considered possible that an error of greater than 5 % will be introduced by not doing so. If a correction is to be made, note the temperature and pressure at which the flowmeter was calibrated (see 6.1.4) and the ambient temperature and pressure at the start and end of the sampling period (see 7.2.3 and 7.3.5).

NOTES

13 In many instances a correction is unnecessary because the difference between the temperature and pressure at the time of calibration of the flowmeter and the temperature and pressure at the time of sampling is not great enough for the correction to be significant.

14 An example of a temperature and pressure correction for the indicated flow rate is given in 9.1.1, for a constant pressure drop, variable area flowmeter.

7.1.3.2 The time-weighted average mass concentration of lead in the air is usually expressed at ambient conditions. However, if results are to be reported at reference conditions, note the ambient temperature and pressure at the start and end of the sampling period (see 7.2.3 and 7.3.5) and use the correction given in 9.1.2.

7.1.4 General

Take precautions in accordance with the recommendations for dust sampling methods. (These recommendations will be published later in an International Standard.)

7.2 Preparation of sampling equipment

Perform the following in a clean area where lead contamination is known to be very low.

7.2.1 Clean the filter holders (6.1.2) before use by soaking them in detergent solution, rinsing them thoroughly with water, and then drying them.

7.2.2 Load the filters (6.1.1) into clean, dry filter holders (see 7.2.1). Handle the filters only with clean flat-tipped forceps, or with clean gloves for larger filters. Seal each loaded filter holder with a cover or plug to protect it from lead contamination during transportation to and from the sampling position.

7.2.3 Connect each sampling pump (6.1.3) to a loaded filter holder (see 7.2.2) using rubber or plastics tubing (6.1.5), and ensuring that no leaks can occur. Remove the protective cover or plug, turn on the pump, and set the selected flow rate (see 7.1.1) with an accuracy of ± 5 % using the calibrated flowmeter (6.1.4). Finally, turn off the pump, and re-seal the filter holder with its cover or plug. If appropriate, note the ambient temperature and pressure (see 7.1.3).

NOTE 15 If a flow-stabilized pump is not used (see note 5), allow the pump to operate for 15 min before setting the flow rate. This enables the pump to warm up and the flow rate to stabilize. Then discard the used filter and load a new one into the filter holder for collecting the sample. Finally, readjust the flow rate to the selected value, turn off the pump, and re-seal the filter holder.

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7.3 Collection of samples

7.3.1 For personal sampling, fix the filter holder to the lapel of the worker, as close to the mouth and nose as practicable. Then attach the sampling pump to the operator as appropriate, to minimize inconvenience. For fixed location sampling, select a suitable sampling site.

7.3.2 When ready to commence sampling, remove the cover or plug from the filter holder and turn on the pump to begin sample collection. Note the time and the initial reading of the flowmeter incorporated in the pump.

7.3.3 The flow rate shall be maintained to within ± 5 % of the nominal value throughout the sampling period. Since it is possible for a filter to become clogged by heavy particulate loading or by the presence of oil mists or other liquids in the air, observe the flowmeter incorporated in the pump frequently (a minimum of once per hour) to check for any drop in flow rate (see note 16). Terminate sampling at the first sign of a problem.

NOTE 16 Regular observation of the flowmeter of a flow-stabilized pump is not essential, however it is useful to provide a check that the pump is continuing to function correctly.

7.3.4 Consider the samples to be invalid if the flow rate is not maintained within the limits given in 7.3.3.

7.3.5 At the end of the sampling period (see 7.1.2), measure the flow rate with an accuracy of $\pm 5\%$ using the calibrated flowmeter (6.1.4), turn off the pump, and note the flow rate and the time. If appropriate, also note the ambient temperature and pressure (see 7.1.3). Reseal the filter holder with its cover or plug, and disconnect the filter holder from the pump.

7.3.6 Carefully record the sample identity and all relevant sampling data (see clause 11). Calculate the mean flow rate during the sampling period by averaging the flow rate measurements taken at the start and end of the sampling period. If appropriate, calculate the mean ambient temperature and pressure, (see 7.1.3). Calculate the volume of air sampled, in litres, at ambient temperature and pressure, by multiplying the mean flow rate in litres per minute by the sampling time in minutes.

7.3.7 For each batch of ten samples, submit two filters from the lot of filters used for sample collection. Subject these filters to exactly the same handling procedure as the samples, except that no air should be drawn through them. Label these filters as blanks.

7.4 Transportation

Transport the samples in a container which has been designed to prevent damage to the samples in transit, and which has been appropriately labelled to ensure proper handling. If possible, transport the samples in the filter holders in which they were collected, with the protective covers or plugs still in position.

8 Procedure

8.1 Cleaning of glassware and polypropylene bottles

8.1.1 Before use, clean all glassware to remove any residual grease or chemicals, by firstly soaking in detergent solution and then rinsing thoroughly with water.

8.1.2 After the initial cleaning (see 8.1.1), clean all beakers with hot nitric acid. Fill them to one-third capacity with concentrated nitric acid (5.2), heat then to approximately 140 °C on the hot plate (6.2.4) in a fume cupboard until most of the liquid has evaporated, allow to cool, and then rinse them thoroughly with water (5.1).

8.1.3 After initial cleaning (see 8.1.1), clean all glassware other than beakers by soaking in dilute nitric acid (5.3) for several days and then rinsing thoroughly with water (5.1).

8.1.4 Glassware which has been previously subjected to the entire cleaning procedure described in 8.1.1, 8.1.2 and 8.1.3, and which has been reserved for the analysis of lead by this method, can be cleaned adequately by rinsing thoroughly with dilute nitric acid (5.3) and then with water (5.1).

8.1.5 Before use, clean polypropylene bottles by soaking them in dilute nitric acid (5.3) for several days and then rinsing them thoroughly with water (5.1).

8.2 Preparation of sample and blank solutions

8.2.1 Open the filter holders, and transfer the samples and blanks (see 7.3.7) into individual clean beakers (6.2.1.1).

NOTE 17 If the filter holders used are designed so that the airborne particles which pass through the entry orifice constitute the inspirable fraction (see note 3), wash any particulate matter adhering to the interior walls of the filter holder into the beakers using a minimum volume of water (5.1).

8.2.2 Add 3 ml of concentrated nitric acid (5.2) and 1 ml of hydrogen peroxide (5.5) to each beaker (see note 18), cover each beaker with a watch glass (6.2.1.2), and heat to approximately 140 °C (see note 19) on the hot plate (6.2.4) in a fume cupboard until most of the liquid has evaporated. Repeat this step twice, adding 3 ml of concentrated nitric acid and 1 ml of hydrogen peroxide each time. Continue to heat each beaker on the hot plate at approximately 140 °C, until it is barely dry and a white ash appears. Do not bake the residue (see note 20). When each beaker is dry, remove it from the hot plate and allow it to cool. Carefully rinse the watch glass and the sides of each beaker with 3 ml to 5 ml of dilute nitric acid (5.3), replace it on the hot plate at approximately 140 °C, and again allow the solution to evaporate just to dryness.

NOTES

18 If lead dioxide is not present in the sample, then the hydrogen peroxide (5.5) need not be added.

19 The exact temperature of the hot plate is not critical. A temperature of 140 °C is used because it is high enough to enable the liquid to be evaporated at an acceptable rate, without being high enough to evaporate the liquid at a rate at which there is a significant risk of inadvertently baking the residue.

20 If the residue ignites, lead will be lost. In this event, report the mass concentration of lead in the air sample as greater than or equal to the test result, and state why