



# Standard Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry<sup>1</sup>

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## 1. Scope

1.1 This standard specifies flame and graphite furnace 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 sample dissolution procedure is not effective for all lead compounds (see Section 5). 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. For example if it is desired to determine silicate lead, a hydrofluoric acid dissolution procedure is required.

1.4 The flame atomic absorption method is applicable to the determination of masses of approximately 1 to 200  $\mu\text{g}$  of lead per sample, without dilution (1).<sup>2</sup> The graphite furnace atomic absorption method is applicable to the determination of masses of approximately 0.01 to 0.5  $\mu\text{g}$  of lead per sample, without dilution (1).

1.5 The ultrasonic extraction procedure has been validated for the determination of masses of approximately 20 to 100  $\mu\text{g}$  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 Section 10).

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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<sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

1.7 Anions that form precipitates with lead may interfere, but this potential interference is overcome by the addition of the disodium salt of ethylenediamine tetraacetic acid (EDTA) when necessary.

1.8 The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.9 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D3195 Practice for Rotameter Calibration

D4840 Guide for Sample Chain-of-Custody Procedures

D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1370 Guide for Air Sampling Strategies for Worker and Workplace Protection

E1792 Specification for Wipe Sampling Materials for Lead in Surface Dust

### 2.2 Other Standards:<sup>4</sup>

ISO 648 Laboratory Glassware—One-Mark Pipettes

ISO 1042 Laboratory Glassware—One-Mark Volumetric Flasks

ISO 3534-1 Statistics—Vocabulary and Symbols—Part 1: General Statistical Terms and Terms Used in Probability

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

- ISO 3585** Glass Plant, Pipelines and Fittings—Properties of Borosilicate Glass 3.3
- ISO 6879** Air Quality—Performance Characteristics and Related Concepts For Air Quality Methods
- ISO 6955** Analytical Spectroscopic Methods—Flame Emission, Atomic Absorption, and Atomic Fluorescence—Vocabulary
- ISO 7708** Particle Size Definitions for Health Related Sampling
- 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 13137** Workplace Atmospheres—Pumps for Personal Sampling of Chemical and Biological Agents—Requirements and Test Methods
- EN 482** Workplace Exposure—General Requirements for the Performance of Procedures for the Measurement of Chemical Agents
- EN 689** Workplace Atmospheres—Guidance for the Assessment of Exposure to Chemical Agents for Comparison with Limit Values and Measurement Strategy
- EN 1232** Workplace Atmospheres—Pumps for Personal Sampling of Chemical Agents—Requirements and Test
- EN 1540** Workplace Atmospheres—Terminology
- EN 12919** Workplace Atmospheres—Pumps for Sampling of Chemical Agents with a Volume Flow Rate of Over 5 L/Min—Requirements and Test Methods
- EN 13205** Workplace Atmospheres—Assessment of Performance of Instruments for Measurement of Airborne Particle Concentrations
- EN ISO 8655-1**, Piston-Operated Volumetric Instruments—Part 1: Terminology, General Requirements and User Recommendations
- EN ISO 8655-2** Piston-Operated Volumetric Instruments—Part 2: Piston Pipettes
- EN ISO 8655-5** Piston-Operated Volumetric Instruments—Part 5: Dispensers
- EN ISO 8655-6** Piston-Operated Volumetric Instruments—Part 6: Gravimetric Test Methods

### 3. Terminology

3.1 *General Definitions*—For definitions of other terms used in this test method, refer to Terminology **D1356**.

3.1.1 *breathing zone*—the space around the worker’s face from where he or she takes his or her breath. 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, centered 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. The definition is not applicable when respiratory protective equipment is used. **EN 1540**

3.1.2 *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**

3.1.3 *exposure (by inhalation)*—a situation in which a chemical agent is present in air which is inhaled by a person.

3.1.4 *occupational exposure limit value*—limit of the time-weighted average of the concentration of a chemical agent in the air within the breathing zone of a worker in relation to a specified reference period. **EN 1540**

3.1.4.1 *Discussion*—An example is the Threshold Limit Value (TLV) for a given substance in workplace air, as established by the ACGIH **(3)**.

3.1.5 *measuring procedure*—procedure for sampling and analyzing one or more chemical agents in the air and including storage and transportation of the sample.

3.1.6 *operating time*—the period during which a sampling pump can be operated at specified flow rate and back pressure without recharging or replacing the battery. **EN 1232**

3.1.7 *reference period*—the specified period of time stated for the limit value of a specific chemical agent.

3.1.7.1 *Discussion*—Examples of limit values for different reference periods are short-term and long-term exposure limits, such as those established by the ACGIH **(3)**.

3.1.8 *time weighted average (TWA) concentration*— the concentration of a chemical agent in the atmosphere, averaged over the reference period.

3.1.8.1 *Discussion*—A more detailed discussion of TWA concentrations and their use can be found in the American Conference of Government Industrial Hygienists publication *Threshold Limit Values for Chemical Substances and Physical Agents; Biological Exposure Indices* **(3)**.

3.1.9 *workplace*—the defined area or areas in which the work activities are carried out. **EN 1540**

#### 3.2 Particle Size Fraction Definitions:

3.2.1 *inhalable convention*—a target specification for sampling instruments when the inhalable fraction is of interest. **ISO 7708**

3.2.2 *inhalable fraction*—the mass fraction of total airborne particles which is inhaled through the nose and mouth.

3.2.2.1 *Discussion*—The inhalable fraction depends on the speed and direction of air movement, on breathing rate and other factors. **ISO 7708**

#### 3.3 Sampling Definitions:

3.3.1 *personal sampler*—a device attached to a person that samples air in the breathing zone. **EN 1540**

3.3.2 *personal sampling*—The process of sampling carried out using a personal sampler. **EN 1540**

3.3.3 *sampling instrument; sampler*—for the purposes of this standard, a device for collecting airborne particles.

3.3.3.1 *Discussion*—Instruments used to collect airborne particles are frequently referred to by a number of other terms, for example, sampling heads, filter holders, filter cassettes etc.

3.3.4 *static sampler; area sampler*—a device, not attached to a person, used in static (area) sampling.

3.3.5 *static sampling; area sampling*—the process of air sampling carried out in a particular location.

#### 3.4 Analytical Definitions:

3.4.1 *sample dissolution*—the process of obtaining a solution containing the analytes of interest from a sample. 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 analyzed to the known mass of analyte in that sample, 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.

3.5.4 *precision*—the closeness of agreement of results obtained by applying the method several times under prescribed conditions. **ISO 6879**

3.5.4.1 *Discussion*—Precision is often expressed in terms of the relative standard deviation.

3.5.5 *true value*—the value which characterizes a quantity perfectly defined in the conditions which exist when that quantity is considered. **ISO 3534-1**

3.5.5.1 *Discussion*—The true value of a quantity is a theoretical concept and, in general, cannot be known exactly. **EN 1540**

## 4. Summary of Test Method

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 may be 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 analyzed 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.

4.4 For accurate lead determination when the concentration of lead in the solution is low, the analysis may be repeated

using graphite furnace 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 may be used for the assessment of workplace exposures to airborne particulate lead (see Guide **E1370** and **EN 689**).

## 5. Reactions

5.1 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 method (see Section **11**).

## 6. Significance and Use

6.1 The health of workers in many industries, for example, 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 standard has been published in order to make available 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 of metals and metal-oids and their workers, etc. It has been assumed in the drafting of this standard that the execution of its provisions, and the interpretation of the results obtained, is entrusted to appropriately qualified and experienced people.

6.2 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 (for example, **EN 482**).

## 7. Reagents

NOTE 1—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 Specification **D1193**, grade 2 water (electrical conductivity less than 0.1 mS/m and resistivity greater than 0.01 M $\Omega$ .m at 25°C). The concentration of lead shall be less than 0.01  $\mu$ g/mL.

NOTE 2—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 $\Omega$ .m (usually expressed by manufacturers of water purification systems as 18 M $\Omega$ .cm).

7.2 *Nitric Acid (HNO<sub>3</sub>)*, concentrated,  $\rho$  about 1.42 g/mL, about 70 % (m/m). The concentration of lead shall be less than