Designation: D6785 – 20

Standard Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry[1]

This standard is issued under the fixed designation D6785; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 (10.2). 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 µg of lead per sample, without dilution (**1**).[2] The graphite furnace atomic absorption method is applicable to the determination of masses of approximately 0.01 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 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 Section 9).

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.10 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

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:*[4]

ISO 648 Laboratory Glassware—One-Mark Pipettes

[1] 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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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

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

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

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
ISO 3585 Glass Plant, Pipelines and Fittings—Properties of Borosilicate Glass 3.3
ISO 6955 Analytical Spectroscopic Methods—Flame Emission, Atomic Absorption, and Atomic Fluorescence—Vocabulary
ISO 7708 Particle Size Definitions for Health Related Sampling
ISO 13137 Workplace Atmospheres—Pumps for Personal Sampling of Chemical and Biological Agents—Requirements and Test Methods
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 18158 Workplace Atmospheres—Terminology
ISO 20581 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 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 *Definitions*—For definitions of other terms used in this test method, refer to Terminology D1356.

3.1.1 *occupational exposure limit value, n*—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. **ISO 18158**

3.1.1.1 *Discussion*—An example is the Threshold Limit Value (TLV) for a given substance in workplace air, as established by the American Conference of Governmental Industrial Hygienists (ACGIH) (**3**).

3.1.2 *personal sampler, n*—a device attached to a person that samples air in the breathing zone. **ISO 18158**

3.1.3 *sample dissolution, n*—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.

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

3.1.4.1 *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**).

3.1.5 *workplace, n*—the defined area or areas in which the work activities are carried out. **ISO 18158**

4. Summary of Test Method

4.1 A known volume of air is drawn through a sampler containing 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.

NOTE 1—Other collection substrates, such as foams, may also be suitable.

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 10.2. 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 10).

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