



Designation: **D6785—13** **D6785 – 20**

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

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 ( $\epsilon$ ) 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 (~~H-210.2~~). 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 ~~409~~).

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:*<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](#)

<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. Current edition approved Oct. 1, 2013; March 1, 2020. Published October 2013; May 2020. Originally approved in 2002. Last previous edition approved in 2008; 2013 as D6785—08; D6785 – 13. DOI: 10.1520/D6785-13; 10.1520/D6785-20.

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

<sup>3</sup> 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.

~~D7035~~D5337 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) 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:*<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

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 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 13137~~18158 Workplace Atmospheres—Pumps for Personal Sampling of Chemical and Biological Agents—Requirements and Test MethodsAtmospheres—Terminology

~~EN 482~~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 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 8655-1,8655-1 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—Definitions* For—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.

3.1.1 *occupational exposure limit value*—*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. **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). ISO 18158

3.1.1.1 *Discussion*—

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

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 *measuring procedure—personal sampler, n*—procedure for sampling and analyzing one or more chemical agents in the air and including storage and transportation of the sample; a device attached to a person that samples air in the breathing zone. ISO 18158~~

~~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~~

~~3.1.3 *reference period—sample dissolution, n*—the specified period of time stated for the limit value of a specific chemical agent; process of obtaining a solution containing the analytes of interest from a sample. This may or may not involve complete dissolution of the sample.~~

~~3.1.7.1 *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).~~

~~3.1.4 *time weighted average (TWA) concentration—concentration, n*—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).~~

~~3.1.4.1 *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—workplace, n*—the defined area or areas in which the work activities are carried out. EN 1540 ISO 18158~~

~~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 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<sup>2+</sup>) by the sample dissolution procedures described in **H-210.2**. 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 **H10**).

#### **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 metalloids 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](#))-ISO 20581).

## 7. Reagents

NOTE 2—*Purity of 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 Specification [D1193](#), grade 2 water (electrical conductivity less than 0.1 mS/m and resistivity greater than 0.01 MΩ.m at 25°C). The concentration of lead shall be less than 0.01 µg/mL.

NOTE 3—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 MΩ.m (usually expressed by manufacturers of water purification systems as 18 MΩ.cm).

7.2 *Nitric Acid (HNO<sub>3</sub>)*, concentrated, ρ about 1.42 g/mL, about 70 % (m/m). The concentration of lead shall be less than 0.01 µg/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 diluted 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 L beaker. Swirl to mix, allow to cool and transfer to a 1 L one-mark volumetric flask (8.7.1.4). Dilute to the mark with water, stopper and mix thoroughly.

7.4 *Nitric Acid, Diluted 1 + 9*—Add approximately 800 mL of water (7.1) to a 1 L one-mark volumetric flask (8.7.1.4). Carefully add 100 mL of concentrated nitric acid (7.2) to the flask and swirl to mix. Allow to cool, dilute to 1 L with water and mix thoroughly.

7.5 *Hydrofluoric Acid (HF)*, concentrated, ρ about 1.16 g/mL, about 48 % (m/m), if required, for digestion of samples containing lead silicates. The concentration of lead shall be less than 0.1 µg/mL. (**Warning**—Concentrated hydrofluoric acid and hydrogen fluoride vapor are extremely toxic and intensely corrosive, and diluted hydrofluoric acid can also cause serious and painful burns that might 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 vapor. Use of personal protection (for example, impermeable gloves, face shield or safety glasses, etc.) 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*, NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>, Mg(NO<sub>3</sub>)<sub>2</sub> or Pd(NO<sub>3</sub>)<sub>2</sub>, or a combination of these, if required, for analysis by graphite furnace atomic absorption spectrometry.

7.7 *Stock Lead Standard Solution*, 1000 mg/L of lead.

7.7.1 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:

7.7.1.1 Dissolve 1.598 g ± 0.001 g of lead (II) nitrate [Pb(NO<sub>3</sub>)<sub>2</sub>], 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 1000 mL one-mark volumetric flask (8.7.1.4). Dilute to the mark with water (7.1), stopper and mix thoroughly. Store in a suitable container, for example, a polypropylene bottle (8.7.2.2), for a maximum period of one year.

7.7.1.2 Dissolve 1.000 g ± 0.001 g of lead wire [99.9 % (m/m) Pb] in 200 mL of 1 + 1 nitric acid (7.3). Quantitatively transfer the solution into a 1000 mL one-mark volumetric flask (8.7.1.4), dilute to the mark with water (7.1), stopper and mix thoroughly. Store in a suitable container, for example, a polypropylene bottle (8.7.2.2), for a maximum period of one year.

7.8 *Working Lead Standard Solution*, 1 mg/L of lead, if required, for analysis by graphite furnace atomic absorption spectrometry. Accurately pipet 100 µL of stock lead standard solution (7.7) into a 100 mL one-mark volumetric flask (8.7.1.4). 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 (8.7.2.2), for a maximum period of one month.

7.9 *Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>)*, approximately 30 % (m/m) solution, if required, for use in the hot plate sample digestion method. The concentration of lead shall be less than 0.01 µg/mL.

7.10 *Acetylene*, cylinder, if required, for use in analysis by flame atomic absorption spectrometry.

7.11 *Air*; compressed and filtered, if required, for use in analysis by flame atomic absorption spectrometry.

## 8. Apparatus

8.1 *Inhalable Samplers*, designed to collect the inhalable fraction of airborne particles, ~~complying with the provisions of EN 13205, for use when the exposure limits of interest apply to the inhalable fraction of airborne particles.~~

NOTE 4—In general, personal samplers for collection of the inhalable fraction of airborne particles do not exhibit the same size selective characteristics if used for static (area) sampling.

NOTE 5—Some inhalable samplers are designed to collect the fraction of airborne particles on a filter, and any particulate matter deposited on the internal surfaces of the sampler is not of interest. Other inhalable samplers are designed such that all airborne particles that pass through the entry orifice(s) ~~match the inhalable convention, in which case orifice(s), including~~ particulate matter deposited on the internal surfaces of the sampler ~~does also~~ form part of the sample. (Samplers of this second type generally incorporate an internal filter cassette or cartridge that can be removed from the sampler to enable this material to be easily recovered.) See [Appendix X1](#) for guidance on handling of wall deposits within sampling cassettes.

8.2 *Adapter*, if necessary, for connecting the sampler to the calibration apparatus or sampling pump.

8.3 *Filters*, of a diameter suitable for use with the samplers (see 8.1), with a collection efficiency of not less than 99.5 % for particles with a 0.3 µm diffusion diameter (see 2.2 of ISO 7708), with a minimum lead content (typically less than 0.1 µg Pb), and compatible with the selected sample preparation method.

NOTE 6—See [Appendix X2](#) for guidance on filter selection.

8.4 *Sampling Pumps*:

8.4.1 Sampling pumps with an adjustable flow rate and capable of maintaining the selected flow rate (between 1 and 5 L/min for personal sampling pumps, and between 5 and 400 L/min for high-volume sampling pumps) to within  $\pm 5$  % of the nominal value throughout the sampling period (see [10.1.29.1.2](#)). Sampling pumps shall have their flow rate set so that the measured flow is traceable to a primary standard (Practice [D3195](#)).

8.4.1.1 Pumps shall conform to the requirements of ISO 13137.

NOTE 6—A flow-stabilized pump may be required to maintain the flow rate within the specified limits.

8.4.2 For personal sampling the pumps shall be capable of being worn by the worker without impeding normal work activity. Sampling pump flowmeters shall be calibrated using either a primary or secondary standard; if a secondary standard is used, it shall be calibrated using a primary standard.

NOTE 7—The pump should have, as a minimum, the following features:

- (1) An automatic control that keeps the volumetric flow rate constant in the case of a changing back pressure,
- (2) Either a malfunction indicator which, following completion of sampling, indicates that the air flow has been reduced or interrupted during sampling; or an automatic cut-out, which stops the pump if the flow rate is reduced or interrupted; and
- (3) A facility for the adjustment of flow rate, such that it can only be actuated with the aid of a tool (for example, screwdriver) or requires special knowledge for operation (for example, by means of software), so as to preclude inadvertent readjustment of the flow rate during use.

An integral timer is a highly desirable additional feature.

NOTE 8—EN 12919 requires that the performance of the pumps is such that:

- (1) The pulsation of the flow rate does not exceed 10 %;
- (2) A flow rate set within the nominal range does not deviate by more than  $\pm 5$  % from the initial value under increasing back pressure;
- (3) Within the range of ambient temperatures from 5 to 40°C, the flow rate measured under operating conditions does not deviate by more than  $\pm 5$  % from the flow rate at 20°C;
- (4) The operating time is at least 2 h, and preferably 8 h, and
- (5) The flow rate does not deviate by more than  $\pm 5$  % from the initial value during the operating time.

If the sampling pump is used outside the range of conditions specified in EN 1232 or EN 12919, or both, appropriate action should be taken to ensure that the performance requirements are met. For instance, at sub-zero temperatures it might be necessary to keep the pump warm by placing it under the worker's clothes.

8.5 *Flowmeter*, portable, with an accuracy that is sufficient to enable the volumetric flow rate (see [10.1.29.1.1.2](#)) to be measured to within  $\pm 5$  %. The calibration of the flowmeter shall be checked against a primary standard, that is, a flowmeter whose accuracy is flowmeter calibration, by a provider accredited to ISO/IEC 17025 for such calibrations, shall be traceable to national standards. If appropriate or international standards (see [10.1.3.1](#)), Practice [D5337](#) record the atmospheric temperature and pressure). Retain the calibration certificate, including the pressure and temperature at which the calibration of the flowmeter was checked, was performed, and identifying and performance documentation for the flowmeter.

NOTE 7—It is recommended that the flowmeter used should be capable of measuring the volumetric flow rate to within  $\pm 2$  % or better.

8.6 *Ancillary Equipment*:

8.6.1 *Flexible Tubing*, of a diameter suitable for making a leak-proof connection from the samplers to the sampling pumps.

8.6.2 *Belts or Harnesses*, to which the sampling pumps can conveniently be fixed for personal sampling (except where the sampling pumps are small enough to fit inside worker's pockets).

8.6.3 *Flat-tipped Forceps*, plastic or with plastic tips, for loading and unloading filters into samplers.

8.6.4 *Filter Transport Cassettes*, or similar, if required to transport samples for laboratory analysis.

8.6.5 *Barometer*, suitable for measurement of atmospheric pressure, if required (see [10.1.39.1.3](#)).

8.6.6 *Thermometer*, minimum temperature range of 0 to 50°C, with graduated divisions minimum resolution of 1°C or less, for measurement of atmospheric temperature. For applications at temperatures below freezing, the range of the thermometer shall extend to the appropriate desired range.

8.7 *Analytical or Laboratory Apparatus*—Ordinary laboratory apparatus, and:

8.7.1 *Glassware*, made of borosilicate glass 3.3 and complying with the requirements of ~~ISO 3585~~:ISO 3585.

NOTE 8—It is preferable to reserve a set of glassware for analysis of lead by this method, in order to ensure that problems do not arise from incomplete removal of lead contamination by cleaning.

8.7.1.1 *Beakers*, of capacities between 50 and 150 mL, with watch glasses to fit the beakers; for hot plate procedures.

8.7.1.2 ~~One-mark~~*One-Mark Pipets*, complying with the requirements of ~~ISO 648~~:ISO 648.

8.7.1.3 *Measuring Cylinder*, of capacity between 10 and 1000 mL. (Also often referred to as a graduated cylinder.)

8.7.1.4 ~~One-mark~~*One-Mark Volumetric Flasks*, of capacities between 10 and 1000 mL, complying with the requirements of ~~ISO 1042~~:ISO 1042.

8.7.2 *Plastic Labware*:

8.7.2.1 *Heatable Beakers, Beaker Covers, etc.*, if required, made of a material that is resistant to corrosion by hydrofluoric acid, for example, a fluorocarbon polymer such as polytetrafluoroethylene (PTFE), and suitable for performing dissolutions using hydrofluoric acid.

8.7.2.2 *Polypropylene Bottles*, of capacities from 100 to 1000 mL.

8.7.3 ~~Piston-operated~~*Piston-Operated Volumetric Instruments*, complying with the requirements of EN ISO 8655-1, and tested in accordance with EN ISO 8655-6: pipetters, complying with the requirements of EN ISO 8655-2, as an alternative to one-mark pipets, for the preparation of standard solutions, calibration solutions and dilution of samples; and dispensers, complying with the requirements of EN ISO 8655-5, for dispensing acids.

8.7.4 *Hot Plate*, thermostatically controlled, capable of maintaining a surface temperature of approximately 150°C; for hot plate procedures.

NOTE 9—The efficiency of thermostating of hot plates is sometimes deficient, and the surface temperature can also vary considerably with position on hot plates with large surface areas. It is therefore recommended that the performance of the hot plate be characterized prior to use.

8.7.5 *Microwave Digestion Apparatus*: (**Warning**—Ensure that manufacturer’s safety recommendations are followed.)

NOTE 10—The specified method is for closed vessel microwave digestion systems with a temperature control system. Microwave digestion systems that are equipped only with a pressure control system or with lower pressure vessels, or both, may be used provided that a suitable sample dissolution procedure is developed and a prior assessment of dissolution efficiency is carried out.

NOTE 11—Open vessel microwave digestion systems can give equivalent results to closed vessel microwave digestion systems. They may, therefore, be used provided that a suitable sample dissolution procedure is developed and a prior assessment of dissolution efficiency is carried out.

8.7.5.1 *Microwave Digestion System*, designed for closed vessel sample digestion in the laboratory, with power output regulation, fitted with a temperature control system capable of sensing the temperature to within  $\pm 2^\circ\text{C}$  and automatically adjusting the microwave power output within 2 s. The microwave cavity shall be corrosion resistant and well ventilated, with all electronics protected against corrosion to ensure safe operation. (**Warning**—Domestic (kitchen) microwave ovens shall not be used, since there are very significant hazards associated with their use for the procedure described in this standard. For example, acid vapors released into the cavity can corrode safety devices that prevent the magnetron from shutting off when the door is opened, potentially exposing the operator to microwave energy. Also, the fumes generated can be extremely hazardous.)

NOTE 12—A pressure control system is also very useful, since it provides a safeguard against the possibility of sample loss due to excessive pressure build-up and partial venting of the sample vessels.

8.7.5.2 *Vessels*, designed for carrying out microwave digestions, capable of withstanding a temperature of 180°C, and with an internal volume of at least 50 mL. The vessels shall be transparent to microwave energy and shall be capable of withstanding internal pressures up to at least 3000 kPa (435 psi) or greater, and temperatures up to at least 180°C, or greater. Closed vessels shall also be equipped with a safety relief valve or disc that will prevent vessel rupture or ejection of the vessel cap. Such vessels consist of an inner liner and cover made of a microwave transparent and chemically resistant material (usually a fluorocarbon polymer such as tetra-fluoro methoxil polymer (TFM)), which contains and isolates the sample solution from a high strength, outer pressure vessel structure. Other types of sample vessels designed to operate at equivalent or higher temperatures or pressures, or both, may be used. (**Warning**—For closed vessel designs, the material from which the outer vessels are made is usually not as chemically resistant as the liner material. Since the outer vessels provide the strength required to withstand the high pressures within the inner liners, they shall be inspected regularly to check for any chemical or physical degradation.)

8.7.6 *Ultrasonic Bath (Sonicator)*, for performing ultrasonic extractions; capable of delivering sufficient power to effect the quantitative dissolution of particulate lead under the conditions described in ~~H-2.4.10.2.4~~ (typically 1 W/cm<sup>2</sup> power density or greater).

8.7.7 *Plastic Centrifuge Tubes*, 50 mL, with screw caps (for ultrasonic procedure).

8.7.8 *Atomic Absorption Spectrometer*, fitted with an air-acetylene burner supplied with compressed air and acetylene, and equipped with either a lead hollow cathode lamp or electrodeless discharge lamp (4, 5). If sample dissolution is carried out with the aid of hydrofluoric acid (see notes in ~~H-2.2.3.10.2.2.3~~ and ~~H-2.3.2.10.2.3.2~~), the atomic absorption spectrometer shall be hydrofluoric acid-compatible. If graphite furnace atomic absorption is to be carried out, the atomic absorption spectrometer shall

be capable of carrying out simultaneous background correction at 283.3 nm, either by using a continuum source such as a deuterium lamp to measure non-specific attenuation (see 5.1.5 of ISO 6955), or by using Zeeman or Smith-Hieftje background correction systems (6).

8.7.9 *Electrothermal Atomizer*, fitted with a solid, pyrolytic graphite platform mounted in a pyrolytically-coated graphite tube, supplied with argon purge gas, and equipped with an autosampler capable of injecting microlitre volumes onto the platform.

NOTE 13—Some manufacturers of atomic absorption spectrometers use an alternative design of electrothermal atomizer to achieve a constant temperature environment during atomization, and some use aerosol deposition as a means of sample introduction. The use of such accessories is acceptable, provided satisfactory method performance is verified. Likewise, atomizers made from heat-resistant metal, for example, tungsten, might also be suitable.

8.7.10 *Analytical Balance*, capable of weighing to  $\pm 0.1$  mg, if required, for use in preparation of stock standard lead solution.

8.7.11 *Disposable Gloves*, for prevention of sample contamination.

8.7.12 *Forceps*, plastic or with plastic tips, flat-tipped, for loading and unloading of filters into and out of samplers.

## 9. Occupational Exposure Assessment

9.1 *Assessment Strategy*—Refer to relevant International or National Standards (for example, EN 689, Guide E1370) for guidance on how to develop an appropriate assessment strategy.

9.2 *Measurement Strategy*:

9.2.1 *General*—Refer to relevant International or National Standards (for example, EN 689, Guide E1370) for general guidance on measurement strategy.

9.2.2 *Personal Sampling*—Exposure of workers to lead shall normally be determined by personal sampling, since the concentration of lead and lead compounds in the breathing zone is usually higher than their background levels in the workplace.

9.2.3 *Static (Area) Sampling*—Static (area) sampling may be carried out, if appropriate, to assess the exposure of workers in a situation where personal sampling is not possible; to characterize the background level of lead in the workplace to give an indication of the efficiency of ventilation or other engineering controls; or to provide information on the location and intensity of an emission source.

9.3 *Selection of Measurement Conditions and Measurement Pattern*:

9.3.1 *General*:

9.3.1.1 The sampling procedure shall be devised to cause the least possible interference with the worker and the normal performance of the job, and to provide samples that are representative of normal working conditions and that are compatible with the analytical method.

9.3.1.2 The pattern of sampling shall take into consideration practical issues, such as the nature of the measurement task and the frequency and duration of particular work activities.

9.3.2 *Screening Measurements of Variation of Concentration in Time or Space, or Both*—Screening measurements of variation of concentration in time or space, or both, may be carried out in the initial stages of a survey to identify locations and periods of elevated exposure, and to set the duration and frequency of sampling for measurements for comparison with limit values.

NOTE 16—For making screening measurements of variation of concentration in time or space, or both, the sampling time used is normally between 5 and 30 min.

9.3.3 *Screening Measurements of Time-Weighted Average Concentration and Worst Case Measurements*—Screening measurements of time-weighted average concentration may be carried out in the initial stages of a survey to assess the effectiveness of control measures. This may involve sampling during representative work episodes to obtain clear information about the level and pattern of exposure, or worst case measurements can be made.

9.3.4 *Measurements for Comparison with Limit Values and Periodic Measurements*—For making long-term measurements, samples shall be collected for the entire working period or during a number of representative work episodes (3).

NOTE 17—The best estimate of long term exposure is obtained by taking samples for the entire working period, but this is often not practicable or it is not desirable (for example, because of the possibility of overloading the filter).

## 9. Sampling

9.1 *Preliminary Considerations*:

9.1.1 *Selection and Use of Samplers*:

9.1.1.1 Select samplers (8.1), for example, those designed to collect the inhalable fraction of airborne particles, as defined in ISO 7708.

NOTE 14—If possible, the samplers selected should be manufactured from conducting material, since samplers comprised of non-conducting material have electrostatic properties that can influence representative sampling. For the purposes of this standard, the use of 25- or 37-mm close-faced sampling cassettes is acceptable (29 CFR 1910.1025) cassettes, which sample the inhalable fraction of particles <35  $\mu$ m aerodynamic equivalent diameter, is acceptable in the absence of significant numbers of larger particles.

9.1.1.2 Use the samplers at their design flow rate and in accordance with the manufacturer's instructions.

9.1.2 *Sampling Period*:



9.1.2.1 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 (see level 9.3).

9.1.2.2 In calculating the minimum sampling time required, it is necessary to consider the selected flow rate and the lower limit of the analytical working range of the method (7).

9.1.2.3 When high concentrations of airborne particles are anticipated, select a sampling period that is not so long as to risk overloading the filter with particulate matter.

NOTE 15—If filter overloading is an observed or suspected problem and it is desired to sample for the entire working day, it might be necessary to collect consecutive samples (8).

#### 9.1.3 *Temperature and Pressure Effects:*

9.1.3.1 *Expression of Results*—Consider whether it is necessary to recalculate the concentration of lead in air to reference conditions (such as in high altitude situations). If so, measure and record the atmospheric temperature and pressure at the start and at the end of the sampling period (see 10.4.1 and 10.4.29.4.1 and 9.4.2) and use the equation given in Appendix X3 to apply the necessary correction.

NOTE 16—The concentration of lead in air is generally stated for actual environmental conditions (temperature, pressure) at the workplace during the sampling period.

9.1.3.2 *Effect of Temperature and Pressure on Flow Rate Measurements*—Refer to the manufacturer's instructions to determine if the indicated volumetric flow rate of the flowmeter (8.5) is dependent upon temperature and pressure. Consider whether the difference between the atmospheric temperature and pressure at the time of calibration of the flowmeter and during sampling is likely to be great enough to justify making a correction to take this into account for example, if the error could be greater than  $\pm 5\%$ . If a correction is necessary, measure and record the atmospheric temperature and pressure at which the calibration of the flowmeter was checked (see 8.5) and measure and record the atmospheric temperature and pressure at the start and at the end of the sampling period (see 10.4.1 and 10.4.29.4.1 and 9.4.2).

NOTE 17—An example of temperature and pressure correction for the indicated mass flow rate is given in Appendix X2 for a constant pressure drop, variable area, flowmeter.

#### 9.2 *Preparation of Sampling Equipment:*

9.2.1 *Cleaning of Samplers*—Unless disposable filter cassettes are used, clean the samplers (8.1) before use. Disassemble the samplers, soak in detergent solution, rinse thoroughly with water, wipe with absorbent tissue, and allow to dry before reassembly. Alternatively, use a laboratory washing machine.

9.2.2 *Loading the Samplers with Filters*—Load clean samplers (see 10.2.19.2.1) with filters (8.3), label each sampler so that it can be uniquely identified, and seal with its protective cover or plug to prevent contamination.

NOTE 18—Alternatively, commercially available pre-loaded filter cassettes may be used.

9.2.3 *Setting the Volumetric Flow Rate*—Perform the following in a clean area, where the concentration of lead is low. Connect each loaded sampler (see 10.2.29.2.2) to a sampling pump (8.4) using flexible tubing (8.6.1), ensuring that no leaks can occur. Remove the protective cover or plug from each sampler, switch on the sampling pump, attach the flowmeter (8.5) to the sampler so that it measures the flow through the sampler inlet orifice(s), and set the required volumetric flow rate (see 10.1.1.29.1.1.2). Switch off the sampling pump and seal the sampler with its protective cover or plug to prevent contamination during transport to the sampling position.

9.2.3.1 If necessary, allow the sampling pump operating conditions to stabilize before setting the volumetric flow rate.

NOTE 23—If necessary, allow the sampling pump operating conditions to stabilize before setting the volumetric flow rate.

9.2.4 *Blanks*—Retain as blanks, one unused loaded sampler from each batch of ten prepared, subject to a minimum of three. Treat these in the same manner as those used for sampling in respect of storage and transport to and from the sampling position, but draw no air through the filters.

### 9.3 Sampling Position:

9.3.1 *Personal Sampling*—Position the sampler in the worker’s breathing zone, as close to the mouth and nose as is reasonably practicable, for example, fastened to the worker’s lapel. Attach the sampling pump to the worker in a manner that causes minimum inconvenience, for example, to a belt (8.6.2) around the waist, or place it in a convenient pocket.

#### 9.3.2 Static (Area) Sampling:

9.3.2.1 If static sampling is carried out to assess the exposure of a worker in a situation where personal sampling is not possible (for example, due to the need to sample at a volumetric flow rate higher than the design flow rate of available personal samplers), position the sampler in the immediate vicinity of the worker and at breathing height. If in doubt, take the sampling position to be the point where the risk of exposure is considered to be greatest.

9.3.2.2 If static sampling is carried out to characterize the background level of lead in the workplace, select a sampling position that is sufficiently remote from the work processes, such that results will not be directly affected by lead from emission sources.

### 9.4 Collection of Samples:

9.4.1 When ready to begin sampling, remove the protective cover or plug from the sampler and switch on the sampling pump. Record the time and volumetric flow rate at the start of the sampling period. If the sampling pump is fitted with an integral timer, check that this is reset to zero. If appropriate (see 10.1.1.29.1.1.2), measure the atmospheric temperature and pressure at the start of the sampling period using the thermometer (8.6.5) and barometer (8.6.6), and record the measured values.

NOTE 19—If the temperature or pressure at the sampling position is different from where the volumetric flow rate was set (see 10.2.39.2.3), the volumetric flow rate could change and it might need to be re-adjusted before sampling.

9.4.2 At the end of the sampling period (see 10.1.29.1.2), record the time and calculate the duration of the sampling period. Check the malfunction indicator or the reading on the integral timer, or both, if fitted, and consider the sample to be invalid if there is evidence that the sampling pump was not operating properly throughout the sampling period. Measure the volumetric flow rate at the end of the sampling period using the flowmeter (10.1.3.29.1.3.2), and record the measured value. If appropriate (see 10.1.39.1.3), measure the atmospheric temperature and pressure at the end of the sampling period using the thermometer (8.6.5) and barometer (8.6.6), and record the measured values.

9.4.3 Carefully record the sample identity and all relevant sampling data (see Section 1413). Calculate the mean volumetric flow rate by averaging the volumetric flow rates at the start and at the end of the sampling period and, if appropriate (see 10.1.39.1.3), calculate the mean atmospheric temperature and pressure. Calculate the volume of air sampled, in litres, at atmospheric temperature and pressure, by multiplying the mean flow rate in litres per minute by the duration of the sampling period in minutes.

### 9.5 Transportation:

9.5.1 For samplers which collect airborne particles on the filter (see Note 45 in 8.1), remove the filter from each sampler, place in a labelled filter transport cassette (8.6.4), and close with a lid. Take particular care to prevent the collected sample from becoming dislodged from heavily loaded filters. Alternatively, transport samples to the laboratory in the samplers in which they were collected.

9.5.2 For samplers which have an internal filter cassette (see Note 45 in 8.1), remove the filter cassette from each sampler and fasten with its lid or transport clip.

9.5.3 For samplers of the disposable cassette type, transport samples to the laboratory in the samplers in which they were collected.

9.5.4 Transport the samples (10.5.19.5.1 – 10.5.39.5.3) to the laboratory in a container that has been designed to prevent damage to the samples in transit and which has been labelled to assure proper handling.

9.5.5 Follow sampling chain of custody procedures to ensure sample traceability. Ensure that the documentation which accompanies the samples is suitable for a “chain of custody” to be established (see, for example, Guide D4840).

## 10. Procedure

NOTE 20—Perform all of the following while wearing gloves.

### 10.1 Cleaning of Glassware and Plasticware:

10.1.1 Before use, clean all glassware, microwave digestion vessels, and plasticware to remove any residual grease or chemicals by first soaking in laboratory detergent solution and then rinsing thoroughly with water (7.1).

10.1.2 After initial cleaning with detergent and water, clean all beakers with nitric acid. This can be accomplished either by soaking for a minimum of 24 h in concentrated nitric acid (7.2), or by the following procedure. Fill beakers to one-third capacity with concentrated nitric acid (7.2), and then heat them on a hot plate with a surface temperature of 140°C in a fume hood until most of the liquid has evaporated, and allow to cool. Rinse beakers thoroughly with water (7.1).

10.1.3 Glassware that has been previously subjected to the entire cleaning procedure described in the previous steps, and which has been reserved for the analysis of lead, can be cleaned adequately by rinsing with 1 + 9 nitric acid (7.4) and then with water (7.1).

10.1.4 Before use, clean polypropylene bottles, microwave digestion vessels, and other plasticware by soaking them in 1 + 9 nitric acid (7.4) for at least 24 h and then rinse thoroughly with water (7.1).