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

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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 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 10).

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:*[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
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:*[4]

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

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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.

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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 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).

## 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 MΩ.m at 25°C). The concentration of lead shall be less than 0.01 µg/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 MΩ.m (usually expressed by manufacturers of water purification systems as 18 MΩ.cm).

7.2 *Nitric Acid ($HNO_3$),* 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,* $NH_4H_2PO_4$, $Mg(NO_3)_2$ or $Pd(NO_3)_2$, 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 [$Pb(NO_3)_2$], previously dried to constant mass at 110°C and cooled in a dessicator, 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 ($H_2O_2$),* 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 3—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 4—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 airborne particles that pass through the entry orifice(s) match the inhalable convention, in which case particulate matter deposited on the internal surfaces of the sampler does 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 5—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 ±5 % of the nominal value throughout the sampling period (see 10.1.2). Sampling pumps shall have their flow rate set so that the measured flow is traceable to a primary standard (Practice D3195).

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 12919ISO 13137 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 ±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 ±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 ±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 10.1.1.2) to be measured to within ±5 %. The calibration of the flowmeter shall be checked against a primary standard, that is, a flowmeter whose accuracy is traceable to national standards. If appropriate (see 10.1.3.1), record the atmospheric temperature and pressure at which the calibration of the flowmeter was checked.

NOTE 9—It is recommended that the flowmeter used should be capable of measuring the volumetric flow rate to within ±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 10.1.3).

8.6.6 *Thermometer,* minimum temperature range of 0 to 50°C, with graduated divisions 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.

NOTE 10—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 Pipets,* complying with the requirements of 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 Volumetric Flasks,* of capacities between 10 and 1000 mL, complying with the requirements of 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 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 11—The efficiency of thermostatting 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 12—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 13—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 ±2°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 14—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 tetrafluoro 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 11.2.4 (typically 1 W/cm$^2$ 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 11.2.2.3 and 11.2.3.2), 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 15—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 ±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).

## 10. Sampling

10.1 *Preliminary Considerations:*

10.1.1 *Selection and Use of Samplers:*

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

NOTE 18—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)[5].

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

10.1.2 *Sampling Period:*

10.1.2.1 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 9.3).

10.1.2.2 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)**.

10.1.2.3 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 19—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)**.

10.1.3 *Temperature and Pressure Effects:*

10.1.3.1 *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 10.4.2) and use the equation given in Appendix X3 to apply the necessary correction.

NOTE 20—The concentration of lead in air is generally stated for actual environmental conditions (temperature, pressure) at the workplace during the sampling period.

10.1.3.2 *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 ±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 10.4.2).

NOTE 21—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.

10.2 *Preparation of Sampling Equipment:*

10.2.1 *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.

10.2.2 *Loading the Samplers with Filters*—Load clean samplers (see 10.2.1) 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 22—Alternatively, commercially available pre-loaded filter cassettes may be used.

10.2.3 *Setting the Volumetric Flow Rate*—Perform the following in a clean area, where the concentration of lead is low. Connect each loaded sampler (see 10.2.2) 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 10.1.1.2). Switch off the sampling pump and seal the sampler with its protective cover or plug to prevent contamination during transport to the sampling position.

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

10.2.4 *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.

10.3 *Sampling Position:*

10.3.1 *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.

10.3.2 *Static (Area) Sampling:*

10.3.2.1 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

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[5] Occupational Exposure to Lead—General Industry Standard. *Code of Federal Regulations*, 29 CFR Part 1910.1025. Washington, DC: U.S. Government Printing Office, 1978; pp. 114-151.

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.

10.3.2.2 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.

10.4 *Collection of Samples:*

10.4.1 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 10.1.1.2), 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 24—If the temperature or pressure at the sampling position is different from where the volumetric flow rate was set (see 10.2.3), the volumetric flow rate could change and it might need to be re-adjusted before sampling.

10.4.2 At the end of the sampling period (see 10.1.2), 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 (10.1.3.2), and record the measured value. If appropriate (see 10.1.3), 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.

10.4.3 Carefully record the sample identity and all relevant sampling data (see Section 14). 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 10.1.3), 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.

10.5 *Transportation:*

10.5.1 For samplers which collect airborne particles on the filter (see Note 4 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.

10.5.2 For samplers which have an internal filter cassette (see Note 4 in 8.1), remove the filter cassette from each sampler and fasten with its lid or transport clip.

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

10.5.4 Transport the samples (10.5.1 – 10.5.3) 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.

10.5.5 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).

## 11. Procedure

NOTE 25—Perform all of the following while wearing gloves.

11.1 *Cleaning of Glassware and Plasticware:*

11.1.1 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).

11.1.2 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).

11.1.3 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).

11.1.4 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).

NOTE 26—Plasticware (possibly disposable) can be received in clean condition directly from the vendor, thereby precluding the need for cleaning prior to use.

11.2 *Preparation of Sample and Blank Solutions:*

11.2.1 *Selection of Sample Dissolution Method*—Prepare samples and blanks for analysis using one of the three sample preparation methods described below: either hot plate digestion, microwave digestion, or ultrasonic extraction.

11.2.2 *Hot Plate Digestion Method:*

11.2.2.1 Open the samplers, sampler filter cassettes or transport filter cassettes (see 10.5), and transfer each filter sample or blank into a clean, labelled 50 mL beaker (8.7.1.1) using flat-tipped forceps (8.6.3). If the sampler used was of a type in which airborne particles deposited on the internal surfaces of the sampler form part of the sample, wash any particulate matter adhering to the internal surfaces into the beaker using a minimum volume of 1 + 9 nitric acid (7.3).

NOTE 27—An alternative procedure entails wiping the inside surfaces of the sampler with a wipe meeting the specifications of ASTM E1792 and including this wipe as part of the sample to be digested and analyzed; see Appendix X1 for more details.

11.2.2.2 To each beaker, add 3 mL of concentrated nitric acid (7.2) and 1 mL of hydrogen peroxide (7.9), and cover with a watch glass.

11.2.2.3 Heat on a hot plate (8.7.4) with a surface temperature of approximately 140°C in a fume hood, and allow the solution to evaporate until the final solution volume is reduced to approximately 1 mL. Avoid taking to dryness. Remove beakers from the hot plate and allow to cool.

NOTE 28—The exact hot plate temperature 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. This temperature is also useful for minimizing the risk of taking samples to dryness.

NOTE 29—Use of hydrofluoric acid (HF) is needed in the digestion process if it is desired to dissolve silicate lead. If the material in the test atmosphere is believed to contain a significant amount of silicate material, its dissolution can be facilitated by adding 1 mL of hydrofluoric acid at the same time as the nitric acid. However, it will be necessary to use heatable beaker and beaker covers, etc., that are made of plastic that is resistant to corrosion by HF, for example, a fluorocarbon polymer such as polytetrafluoroethylene (PTFE). (**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 which may 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 HF.)

11.2.2.4 Carefully rinse each watch glass and the sides of each beaker with water, and transfer each solution quantitatively to a 10 mL one-mark volumetric flask (8.7.1.4). If necessary, remove any undissolved particulate by filtration or centrifugation. Dilute to the mark of the volumetric flask with water (7.1), seal the flask with a stopper, and mix thoroughly.

11.2.3 *Microwave Digestion Method:*

11.2.3.1 Open the samplers, sampler filter cassettes or transport filter cassettes (see 10.5), and transfer each filter into the clean liner of a labelled microwave digestion vessel (8.7.5.2) using flat-tipped forceps (8.6.3). Follow the same procedure for blank filters. If the sampler used was of a type in which airborne particles deposited on the internal surfaces of the sampler form part of the sample, wash any particulate matter adhering to the internal surfaces into the vessel liner using a minimum volume of water (7.1).

NOTE 30—An alternative procedure entails wiping the inside surfaces of the sampler with a wipe meeting the specifications of ASTM E1792 and including this wipe as part of the sample to be digested and analyzed; see Appendix X1 for more details.

11.2.3.2 Carefully add 5 mL of concentrated nitric acid (7.2) to the inside of the liner of the microwave digestion vessel containing the filter sample or blank. Seal the vessels.

NOTE 31—The use of hydrofluoric acid is required to dissolve lead silicates. If the material present in the test atmosphere is believed to contain a significant amount of silicate material, its dissolution can be facilitated by adding 1 mL of hydrofluoric acid at the same time as the nitric acid. (**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.)

11.2.3.3 Load the vessels into the microwave oven (8.7.5.1) in accordance with manufacturer's instructions. Vessels containing samples shall be evenly and symmetrically placed in the microwave oven.

NOTE 32—Even, symmetrical spacing of vessels is needed to ensure uniform microwave heating of all vessel solutions.

11.2.3.4 Program the microwave digestion system to reach 180°C in less than 10 min, and then hold at this temperature for 15 min.

NOTE 33—If hydrofluoric acid is used to dissolve the samples and the temperature sensor is not resistant to attack by this acid, the vessel in which the temperature sensor is fitted should contain a filter blank in which an equal volume of nitric acid is substituted for the hydrofluoric acid used for dissolution of the samples.

11.2.3.5 At the end of the digestion period, remove the vessels from the microwave oven, place them in a fume hood, and allow the solutions to cool to room temperature.

11.2.3.6 For closed vessels, carefully detach the vent tubing, and carefully shake the vessels to vent any excess gas pressure that may be present inside the vessels. Carefully open each sample vessel.

11.2.3.7 Quantitatively transfer the contents of each vessel to 10 mL one-mark volumetric flasks (8.7.1.4). Carefully rinse each vessel with water, and dilute to volume with water (7.1). If necessary, remove any undissolved particulate by filtration or centrifugation. Seal each flask with a stopper and mix thoroughly.

11.2.4 *Ultrasonic Extraction Method:*

NOTE 34—The following method is not applicable to samples containing silicates. In such a case, use the method employing hydrofluoric acid described in Appendix D of ISO 15202-2.

11.2.4.1 Open the samplers, sampler filter cassettes or transport filter cassettes (see 10.5), and transfer each filter sample or blank into a clean 50 mL centrifuge tube (8.7.7) using flat-tipped forceps (8.6.3). Label each centrifuge tube with a unique identifier. If the sampler used was of a type in which airborne particles deposited on the internal surfaces of the sampler form part of the sample, wash any particulate matter adhering to the internal surfaces into the centrifuge tube using a minimum volume of 1 + 9 nitric acid (7.4). Using a clean glass or plastic rod, push the filter to the bottom of the centrifuge tube.

NOTE 35—An alternative procedure entails wiping the inside surfaces of the sampler with a wipe meeting the specifications of ASTM E1792 and including this wipe as part of the sample to be extracted and analyzed; see Appendix X1 for more details. Another alternative includes performing the ultrasonic extraction directly within the sampling cassette.

11.2.4.2 Introduce 10 mL of 1 + 9 nitric acid (7.4) into each centrifuge tube containing a filter sample or blank, and cap each tube.

11.2.4.3 Place each centrifuge tube upright into an ultrasonic bath (8.7.6), and ensure that the water level within the bath is at or above the level of liquid within the tube.

NOTE 36—Depending on the size of the ultrasonic bath, many centrifuge tubes may be immersed in the bath at one time. A custom rack for the centrifuge tubes can be purchased or constructed to allow for the regular and orderly placement of multiple tubes in the sonicator bath.

11.2.4.4 Apply ultrasonic energy to the acid-immersed filter samples for a minimum of 30 min.

11.2.4.5 Remove centrifuge tubes from the bath. Keep tubes in upright position, and allow to cool to room temperature.

11.3 *Instrumental Analysis:*

11.3.1 *Selection of Analytical Line*—The 283.3 nm lead analytical line shall be used for making absorbance measurements.

NOTE 37—The most sensitive lead line is at 217.0 nm. However, this line is subject to possible spectral interference from antimony, and the significant spectral background at 217.0 nm makes correction for non-specific attenuation (see 5.1.5 of ISO 6955) essential at this wavelength. The 283.3 nm line exhibits somewhat lower sensitivity than the 217.0 nm line, but it is not subject to spectral interference. In addition, while the detection limits obtained are dependent upon the instrument used, absorbance measurements made at 283.3 nm generally have a better signal-to-noise ratio than those made at 217.0 nm, and hence a better detection limit.

11.3.2 *Flame Atomic Absorption Spectrometry:*

11.3.2.1 *Instrument Set-up*—Set up the atomic absorption spectrometer (8.7.8) to make absorbance measurements at 283.3 nm, following the manufacturer's instructions for specific instrument operating parameters. Use a lead hollow cathode lamp or electrodeless discharge lamp and an oxidizing air-acetylene flame. Allow an appropriate warm-up period for the source lamp.

11.3.2.2 *Preparation of Calibration Solutions:*

*(1)* Use 1 + 9 nitric acid (7.4) as the solvent blank.

*(2)* Prepare at least four calibration solutions, including a blank calibration solution, to cover a suitable concentration range, for example, from 0 to 20 µg/mL of lead. Accurately pipet appropriate volumes of stock lead standard solution (7.7) into separate, labelled 100 mL one-mark volumetric flasks (8.7.1.4). Dilute to the mark with 1 + 1 nitric acid (7.3), for test solutions prepared by the microwave digestion method, or with 1 + 9 nitric acid (7.4) for test solutions prepared by the hot plate or ultrasonic sample digestion methods. Stopper and mix thoroughly. Prepare these calibration solutions fresh daily.

NOTE 38—The concentration range of calibration solutions is given as a guide. The upper limit of the working range is dependent upon which wavelength is used, and it is also governed by instrumental factors that affect sensitivity and the linearity of the calibration. Accordingly, the range of the set of calibration solutions may be varied, but when making any changes, ensure that the response of the spectrometer over the alternative range of concentrations selected is such that it is linear.

11.3.2.3 *Calibration Measurements:*

*(1)* Adjust the spectrometer zero while aspirating the solvent blank (see 11.3.2.2, Item *(1)*) into the flame. Then aspirate the calibration solutions into the flame, and make absorbance measurements for each solution.

NOTE 39—Use of an autosampler is recommended, since precision is maximized and the volume of solution consumed is minimized.

*(2)* Analyze all blank solutions, and calculate the mean concentration of the blank solutions.

11.3.2.4 *Calibration Function*—For instruments controlled by a microprocessor or personal computer, use a suitable algorithm to generate the calibration function. For instruments without this capability, prepare a calibration graph by plotting the absorbance of the calibration solutions versus the concentration of lead (µg/mL) in the respective solutions.

NOTE 40—In general, it is best to work within the linear range of the calibration, where absorbance is proportional to the lead concentration in solution.

11.3.2.5 *Determination:*

*(1)* Adjust the spectrometer zero while aspirating the solvent blank (see 11.3.2.2) into the flame. (Repeat this procedure regularly throughout the determination and readjust the zero if the baseline drifts.) Then aspirate the sample and blank test solutions into the flame and make absorbance measurements for each solution. For instruments controlled by a microprocessor or personal computer, use the calibration function to calculate the concentration of lead in the test solutions, and obtain a direct read-out of the results in concentration units. For instruments without this capability, determine the concentration of lead in the test solutions from the calibration graph.

*(2)* Aspirate a mid-range calibration solution after each five to ten test solutions and make an absorbance measurement. If this indicates that the sensitivity has changed by more than ±5 %, take one of the following corrective measures: either use the available software facilities of the microprocessor or personal computer to correct for the sensitivity change (reslope facility); or suspend analysis and recalibrate the spectrometer. In either case, reanalyze the test solutions that were analyzed during the period in which the sensitivity change occurred.

*(3)* If high concentrations of lead are found, dilute the sample test solutions to bring the concentration within the calibration range. Make all dilutions so that the final nitric acid concentration is 1 + 9, and record the dilution factor DF.

*(4)* Calculate the mean lead concentration of the blank test solution.

*(5)* If the concentration of lead in the sample test solutions is less than 0.5 µg/mL, consider repeating the analysis using graphite furnace atomic absorption spectrometry, since this technique gives more precise measurements at low concentrations.

11.3.3 *Graphite Furnace Atomic Absorption Spectrometry:*

NOTE 41—Lead is present ubiquitously in the environment, and therefore it is imperative that strict standards of cleanliness are observed to avoid contamination of labware. This is particularly important when carrying out graphite furnace atomic absorption spectrometry since the technique exhibits a very low detection limit. Ensure that all glassware is cleaned thoroughly before use, and autosampler cups are stored in 1 + 9 nitric acid until required.

11.3.3.1 *Preparation of Working Calibration Solutions:*

*(1)* Prepare a working calibration solution at a concentration of 2.5 ng/mL of lead. Accurately pipet 250 µL of working lead standard solution (7.8) into a 100 mL one-mark volumetric flask (8.7.1.4). Dilute to the mark with 1 + 1 nitric acid (7.3), for test solutions prepared by the microwave digestion method, or with 1 + 9 nitric acid (7.4) for test solutions prepared by the hot plate or ultrasonic sample digestion methods. Stopper and mix thoroughly. Prepare this solution fresh weekly.

*(2)* Prepare a working calibration blank solution following the procedure in the preceding paragraph, but omitting the 250 µL of working lead standard solution. Prepare this solution fresh weekly.

*(3)* For instruments controlled by a microprocessor or personal computer, use a suitable algorithm to generate the calibration function. For instruments without this capability, prepare a calibration graph by plotting the absorbance of the calibration solutions versus the concentration of lead in micrograms per millilitre in the respective solutions.

NOTE 42—In general it is best to work in the linear range of the calibration, where absorbance is proportional to the concentration of lead in solution.

11.3.3.2 *Calibration and Determination:*

*(1)* Set up the atomic absorption spectrometer (8.7.8) and the electrothermal atomizer (8.7.9) to measure lead at a wavelength of 283.3 nm, using background correction to correct for non-specific attenuation (see 5.1.5 of ISO 6955). Follow the manufacturer's instructions for specific operating parameters. Allow a suitable warm-up period for the hollow cathode lamp or equivalent source.

NOTE 43—The operating parameters for graphite furnace atomic absorption vary considerably between different instruments, much more so than for flame atomic absorption spectrometry.

*(2)* Program the autosampler to prepare matrix-modified calibration solutions, sample test solutions and blank test solutions in situ on a pyrolytic graphite platform mounted in the pyrolytically coated graphite tube of the electrothermal atomizer. Prepare at least four matrix-modified calibration solutions to cover the range 0 to 50 ng/mL of lead using the working calibration solution (11.3.3.1, Item *(1)*), the working calibration blank solution (11.3.3.1, Item *(2)*), and matrix modifier (7.6). Also prepare matrix modified sample and blank test solutions using the unmodified sample and blank solutions and matrix modifier solution. Matrix modified calibration and test solutions shall be prepared by an autosampler or manually by means of one-mark volumetric flasks.

NOTE 44—The procedure described above may be varied to accommodate the use of electrothermal atomizers of alternative design.

*(3)* Set up the analytical sequence in the microprocessor or personal computer interfaced to the graphite furnace atomic absorption spectrometric instrument. Specify an appropriate number of replicate analyses for each solution, and insert a calibration blank solution and a mid-range calibration solution after each five to ten test solutions in order to monitor for baseline drift and sensitivity change, respectively.

*(4)* Place the working calibration solution (11.3.3.1, Item *(1)*), the working calibration blank solution (11.3.3.1, Item *(2)*), the matrix modifier (7.6), and the unmodified sample and blank solutions in separate acid-washed autosampler cups and position as appropriate in the autosampler carousel. Analyze the matrix modified calibration and test solutions, using the microprocessor or personal computer software to generate a calibration. Obtain a direct readout of sample and blank results in ng Pb/mL.

*(5)* If significant baseline drift is observed during the course of analysis, or if the sensitivity changes by more than ±5 %, take one or more of the following corrective measures: either use the available software facilities of the microprocessor or personal computer to correct for sensitivity change ("reslope" facility); or suspend analysis and recalibrate the spectrometer. In either case, reanalyze the solutions that were analyzed during the period in which the sensitivity change occurred.

*(6)* If concentrations of lead above the upper limit of the linear calibration range are found, dilute the sample test solutions in order to bring them within the range of the calibration, and repeat the analysis. Make all dilutions so that the final nitric acid concentration is 1 + 1 or 1 + 9, as appropriate, and record the dilution factor (DF). Alternatively, analyze a smaller aliquot of sample, and make a correction for the amount of sample that is analyzed.

*(7)* Calculate the mean concentration of lead in the blank test solutions.

11.4 *Estimation of the Instrumental Detection Limit:*

11.4.1 Estimate the instrumental detection limit under the working analytical conditions following the procedure described below, and repeat this exercise whenever the experimental conditions are changed.

11.4.2 Prepare test solutions at a concentration of 0.1 µg/mL of lead for flame atomic absorption analysis or 1 ng/mL for graphite furnace atomic absorption analysis by diluting the working lead standard solution (7.8). Make these dilutions so that the final nitric acid concentration is 1 + 9.

11.4.3 Make at least twenty absorbance measurements on the test solution and calculate the instrumental detection limit as three times the sample standard deviation of the mean concentration value (see 6.2.3 of ISO 6955).

NOTE 45—The limit of detection calculated from results using this procedure is an instrumental detection limit. This is of use in identifying changes in instrument performance, but it is not a method detection limit (**7**). The instrumental detection limit is likely to be unrealistically low because it only takes into account the variability between individual instrumental readings; determinations made on one solution do not take into consideration contributions to variability from the matrix or sample.

11.5 *Estimation of the Method Detection Limit:*

11.5.1 Estimate the method detection limit under the working analytical conditions following the procedure described in 11.5.2 and 11.5.3, and repeat this exercise whenever the experimental conditions are changed significantly.

11.5.2 Fortify at least ten filters (8.3) with lead near the anticipated detection limit, for example, 1 µg of lead for flame atomic absorption analysis or 0.01 µg of lead for graphite furnace atomic absorption analysis, by spiking the filter with 0.1 mL of a suitable calibration solution (11.3.3.1) diluted by an appropriate factor with 1 + 9 nitric acid (7.4).

11.5.3 Make atomic absorption measurements on the test solutions derived from each spiked filter (11.5.2) (after carrying out digestion of the filters), and calculate the method detection limit as three times the sample standard deviation of the mean concentration value.

NOTE 46—An alternative procedure for estimating the method detection limit involves the analysis of filter samples fortified with the analyte of interest at values spanning the predicted detection limit (**7**).

11.6 *Quality Control*—Quality control (QC) samples to process with each batch of field samples are summarized below:

11.6.1 *Reagent Blanks and Media Blanks*—Carry reagent blanks (water and reagents) and media blanks (unspiked filters) throughout the entire sample preparation and analytical process to determine whether the samples are being contaminated from laboratory activities. Process reagent blanks in accordance with a frequency of at least 1 per 20 samples or a minimum of one per batch.

11.6.2 *Spiked Samples and Spiked Duplicate Samples:*

11.6.2.1 Process these samples on a routine basis to estimate the method accuracy on the sample batch, expressed as a percent recovery relative to the true spiked value. Spiked samples and spiked duplicate samples consist of filters to which known amounts of analyte were added. (This can be accomplished by spiking known volumes of known concentrations of lead solution at amounts within the dynamic range of the instrument. The lead solution used shall be prepared from a stock standard solution from a different source than that used for preparing the calibration solutions.) Process these QC samples at a frequency of at least 1 per 20 samples or minimum of one per batch.

11.6.2.2 Monitor the performance of the method by plotting control charts of the relative percent recoveries and of the relative percent differences between the spiked samples and the spiked duplicate samples. If QC results indicate that the method is out of control, investigate the reasons for this, take corrective action and reanalyze the samples if possible. See Guide E882 for general guidance on the use of quality control charts.

11.6.3 *Certified Reference Materials (CRMs)*—Certified reference materials (CRMs) for lead shall be analyzed prior to routine use of the method, and periodically thereafter, to establish that the percent recovery relative to the certified value is satisfactory. Suitable CRMs are available from many sources. A minimum of one CRM sample shall be analyzed at least six times quarterly.

11.6.4 *External Quality Assessment*—If laboratories carry out lead in air analysis on a regular basis, it is strongly recommended that they participate in a relevant external quality assessment scheme or proficiency testing scheme.

## 12. Calculation

12.1 *Calculation of Lead Concentration:*

12.1.1 Calculate the mass concentration of lead in the air sample, ρ[Pb], in $mg/m^3$, at ambient conditions, using the equation:

$$\rho[Pb] = \frac{(\rho[Pb]_1 \times V_1 \times DF) - (\rho\,[Pb]_0 \times V_0)}{V}$$

where:

$\rho[Pb]_0$ = mean lead concentration in the blank test solutions, µg/mL,

$\rho[Pb]_1$ = lead concentration in the sample test solution, µg/mL,

$V$ = volume of the air sample, L,

$V_0$ = volume of the blank test solution, mL,

$V_1$ = volume of the sample test solution, mL, and

$DF$ = dilution factor ($DF$ = 1 in the absence of dilution).

## 13. Special Cases

13.1 If there is any doubt as to the suitability of the digestion or extraction procedure used for the dissolution of particulate lead compounds (for example, silicates) that may be present in the test atmosphere, determine its effectiveness by analyzing a bulk sample of known lead content that is similar in nature to the material being used or produced. If the efficiency of recovery is less than 90 %, use an alternative, more vigorous dissolution procedure, for example, by using hydrofluoric acid. Do not use a correction factor to compensate for an apparently ineffective dissolution procedure.

NOTE 47—It should be recognized that the recovery of lead can be dependent upon the particle size distribution of a bulk sample.

13.2 Anions that give rise to precipitates can interfere with lead analysis. If such interferents are likely to be present in sample solutions, add the disodium salt of ethylenediamine tetraacetic acid (EDTA) to the sample and blank solutions and to the calibration standard solutions, such that these solutions have a concentration of 0.1 mol/L of EDTA.

NOTE 48—The addition of EDTA usually prevents precipitation, but high levels of phosphate can diminish the lead signal even in the presence of EDTA. If high levels of phosphate are suspected in the sample solutions, then the method of standard addition should be used to obtain accurate results (see 6.1.3 of ISO 6955).

13.3 It has been postulated (**9, 10**) that gaseous lead can be present in significant concentrations in certain work environments, for example, when high temperature processes are used. In such circumstances, the sampling method described in this test method might not be fully effective because gaseous lead could pass through the filter. If necessary, this possibility can be investigated using a sampling train consisting of a filter and bubbler (see (**9, 10**)).

## 14. Report

14.1 The test report shall contain the following information:

14.1.1 A statement to indicate the confidentiality of the information supplied, if appropriate;

14.1.2 A complete identification of the air sample, including the date of sampling, the place of sampling, the type of sample (personal or static (area)), either the identity of the individual whose breathing zone was sampled (or other personal identifier) or the location at which the general occupational environment was sampled (for a static (area) sample), a brief description of the work activities that were carried out during the sampling period, and a unique sample identification code;

14.1.3 A reference to this test method;

14.1.4 The make, type and diameter of filter used;

14.1.5 The make and type of sampler used;

14.1.6 The make and type of sampling pump used, and its identification;

14.1.7 The make and type of flowmeter used, the primary standard against which the calibration of the flowmeter was checked, the range of flow rates over which the calibration of the flowmeter was checked, and the atmospheric temperature and pressure at which the calibration of the flowmeter was checked, if appropriate (see 10.1.3);

14.1.8 The time at the start and at the end of the sampling period, and the duration of the sampling period in minutes;

14.1.9 The mean flow rate during the sampling period, in litres per minute;

14.1.10 The mean atmospheric temperature and pressure during the sampling period, if appropriate (see 10.1.3);

14.1.11 The volume of air sampled, in litres, at ambient conditions;

14.1.12 The name of the person who collected the sample;

14.1.13 The time-weighted average mass concentration of lead found in the air sample (in mg/$m^3$), at ambient temperature and pressure, or, if appropriate, adjusted to reference conditions;

14.1.14 The analytical variables used to calculate the result, including the concentrations of lead in the sample and blank solutions, the volumes of the sample and blank solutions, and the dilution factor, if applicable;

NOTE 49—If necessary data (for example, the volume of air sampled) are not available to the laboratory for the above calculations to be carried out, the laboratory report may contain the analytical result in micrograms of lead per filter sample.

14.1.15 The type(s) of instrument(s) used for sample preparation and analysis, and unique identifier(s);

14.1.16 The estimated detection limit under the working analytical conditions;

14.1.17 Any operation not specified in this standard, or regarded as optional;

14.1.18 The name of the analyst(s) (or other unique identifier(s));

14.1.19 The date of the analysis; and

14.1.20 Any inadvertent deviations, unusual occurrences, or other notable observations.

## 15. Precision and Bias

15.1 *Sample Collection*—A collection efficiency of 1.00 was determined for the filter collection step for laboratory-generated lead nitrate aerosols and for lead fume (**11**).

15.2 *Hot Plate Digestion and Flame Atomic Absorption Spectrometry:*

15.2.1 The detection limit of flame atomic absorption measurements depends in part on the instrument used. However, the detection limit of the method has been estimated to be 0.25 µg per sample, and the precision of the measurement procedure was < 0.1 for samples in the range 0.9 to 2.25 µg and < 0.03 for samples in the range 3.6 to 288 µg (**4**). No bias has been identified. The applicable range is 1 to 200 µg Pb per sample, without dilution.

15.2.2 In tests using laboratory-generated lead nitrate aerosols (**11**), the coefficient of variation for a similar procedure was found to be 0.072 for the overall sampling and analytical method in the range 130 to 400 µg Pb/$m^3$. The bias of the method was found to be insignificant. Also, data from interlaboratory proficiency testing for samples of paint, soil and dust (**12**, **13**) have indicated insignificant bias.

NOTE 50—If the sample dissolution procedure using concentrated nitric acid and hydrogen peroxide is ineffective for the dissolution of particulate lead compounds present in the test atmosphere (for example, lead silicates), and an alternative, more vigorous dissolution procedure has not been used (for example, employing hydrofluoric acid), then the analytical results will be subject to a negative bias. If it is desired to determine lead in samples containing high concentrations of silicates, consider the use of hydrofluoric acid in the digestion procedure.

15.3 *Microwave Digestion and Flame Atomic Absorption Spectrometry*—Interlaboratory evaluations of lead determinations in reference materials (paint, soil, and dust) have shown that microwave digestion with concentrated nitric acid performs equivalently to hot plate digestion when followed by lead analysis using flame atomic absorption spectrometry (**13**).

15.4 *Ultrasonic Extraction and Flame Atomic Absorption Spectrometry*—Ultrasonic extraction with electrochemical determination of lead has been shown to perform equivalently to microwave digestion and flame atomic absorption spectrometry in the determination of lead from laboratory-generated lead fume atmospheres (**2**).

15.5 *Hot Plate Digestion and Graphite Furnace Atomic Absorption Spectrometry*—The detection limit of graphite furnace atomic absorption measurements depends in part on the instrument used. However, the detection limit of the method has been estimated to be 0.003 µg per sample, and the precision of the measurement procedure was < 0.05 for samples in the range 0.1 to 4.5 µg. No bias has been identified. The applicable range is 0.01 to 0.5 µg Pb per sample (**4**), without dilution.

15.6 *Microwave Digestion and Graphite Furnace Atomic Absorption Spectrometry*—Interlaboratory analysis of lead in reference materials has demonstrated the equivalence of microwave digestion followed by graphite furnace atomic absorption spectrometry and hot plate digestion followed by graphite furnace atomic absorption spectrometric determination of lead (**13**).

15.7 *Ultrasonic Extraction and Graphite Furnace Atomic Absorption Spectrometry*—Ultrasonic extraction with electrochemical detection of lead was evaluated against hot plate extraction and graphite furnace atomic absorption spectrometry for air samples collected from construction sites (**14**). The test procedure using ultrasonic extraction was found to be equivalent to that using hot plate digestion.

# APPENDIXES

## (Nonmandatory Information)

## X1. SAMPLER WALL DEPOSITS (15, 16)

X1.1 Samplers for aerosols typically consist of a filter supported in a holder, though other collection substrates are also used, for example, impaction plates, foams. The entire device is considered to be an aerosol sampler. The sampling efficiency of the aerosol sampler is considered to be the air concentration calculated from the particulates collected by the sampler compared to the undisturbed concentration in air. All aerosol samplers exhibit a decrease in sampling efficiency with increasing particulate aerodynamic diameter. Some size-selective samplers are designed for a specific sampling efficiency over a range of aerodynamic diameters, in which case the actual sampling efficiency of the sampler is considered in reference to the stated efficiency. In some sampler designs (for example, cyclones) there is an internal separator to achieve the required size separation.

X1.2 The collection efficiency of an aerosol sampler has three components: aspiration (or entry efficiency), passage within the sampler (either from entry plane to collection substrate or, if an internal separator is present, both from entry plane to internal separator and from internal separator to collection substrate) and penetration (through the internal separator, if present). For any given design of sampler, the three components are functions of particle aerodynamic size and air flow-rate through the sampler. The aspiration efficiency also depends on wind speed and direction, while the sampler's angle to the vertical influences both aspiration and transport efficiency. Part of the sample will deposit on internal surfaces of the sampler as a result of losses during passage within the sampler. In addition, if the sampler is transported after sampling, particles already deposited on the substrate may become dislodged and add to deposits already on the internal surfaces (although this is likely of lesser importance). If the design specification for the sampler is to include all aspirated particles, these losses should be taken into account unless it can be shown that they can be disregarded. Table X1.1 below provides median and maximum values of deposits on the walls for two commercially available samplers in common use. No pattern can be discerned from these data that would allow the use of correction factors without introducing a very large uncertainty.

**TABLE X1.1 CFC Maximum and Median Wall Deposits of Lead (15)**

| Environment | n | Maximum Wall Deposit (%) | Median Wall Deposit (%) |
|---|---|---|---|
| Copper smelter | 17 | 55 | 21 |
| Lead ore mill | 28 | 35 | 19 |
| Solder manufacture | 30 | 74 | 29 |
| Battery production | 16 | 66 | 28 |
| Bronze foundry | 6 | 17 | 13 |

X1.3 For some samplers, such as the GSP and CIP, the sample deposited on the collection substrate is considered to be the entire sample; that is, there are no wall deposits. Internal capsules that are digestible automatically include aerosol deposits not collected on the filter (**17**). For other samplers, it is recommended that the wall deposits should be evaluated (**18**).

X1.4 There exist several procedures that could be used to account for wall deposits. One method is digestion within the body of the sampler, which is the practice in some French standard methods. This procedure needs to be carefully designed with respect to the composition of the acid media, the composition of the substrate and the stability and integrity of the sampler. Another procedure, often followed, is to rinse the internal deposit into the digestion vessel containing the collection substrate. This may be quantitative if the deposit is very soluble or easily displaced, but that may not be the case, even when acid is used for the rinse. Brushing the deposit into the digestion vessel may not be quantitative, and may be a source of contamination. A procedure that has been tested in a limited evaluation and shown to be quantitative is wet-wiping of the internal surfaces.

X1.5 Wiping the internal surfaces of a sampler with a wetted wipe allows a combination of mechanical removal with wetting or solubilization. The choice of wipe is important. It must be free of significant contamination, and it must be compatible with the digestion and analytical procedure. The area of the wipe should be as small as possible in order not to unduly compromise the detection limits of the analysis, and quality control samples should be matched to the same matrix. Typically, the same material should be used as would be selected to perform a surface wipe sample for the element(s) of interest. If the most appropriate wipe material cannot be digested and analyzed in the same way as the collection substrate, it can be analyzed as a separate sample and the results combined. Where the procedure has not been validated to provide quantitative results for a first wipe, the analysis of a second wipe can be used as a guide to recovery.

X1.6 Where the validation of an air sampling and analytical method has not included a specific procedure for recovering and analyzing wall deposits, any procedure selected for this purpose will add an unknown amount to the uncertainty budget of the method. It is therefore recommended that any procedure be validated to determine the contribution to uncertainty.

## X2. GUIDANCE ON FILTER SELECTION

### INTRODUCTION

The following guidance is intended to help the user choose the most suitable filter for a particular application. It is not an exhaustive treatise on the subject, and covers only the basics of those matters that merit consideration. In many instances, similar considerations apply to the selection of other sampling substrates, such as polyurethane foams.

### X2.1 Collection Efficiency

X2.1.1 Most filters that are typically used for sampling airborne particulate matter have the required collection efficiency (see 8.3) for sampling both the respirable and the inhalable fractions of airborne particles. Such filters include depth filters, for example, glass or quartz fiber filters, and membrane filters, for example, mixed cellulose ester membrane filters and membrane filters made from polymers such as polyvinyl chloride (PVC) or polytetrafluoroethylene (PTFE).

X2.1.2 Cellulose (paper) filters can have a collection efficiency below 99 %, and are generally unsuitable for sampling airborne particles containing lead.

X2.1.3 Certain processes carried out at elevated temperatures can produce ultrafine particles condensed from the vapor phase, known as fume. Filters used to sample airborne particulate matter can have a reduced collection efficiency for these very small particles, which are significantly less than 1 µm in diameter. However, the particles usually agglomerate soon after formation to produce larger particles that are efficiently collected. In general, filters that have a collection efficiency that meets the specification given in 8.3 are therefore suitable for sampling fume.

### X2.2 Dust-loading Capacity

X2.2.1 Membrane filters are manufactured from a variety of polymeric materials by a number of different processes. In each case the result is a thin, flexible disc of microporous material, with well-defined pore size, pore structure, pore density, etc. Retention of particles takes place on the surface of membrane filters, which results in their having a relatively low dust-loading capacity in comparison with depth filters. If an excessive amount of dust is collected on a membrane filters, this can result in blockage of the pores, and failure of the sampling pump. In addition, sample can be lost from the filter during handling or in transport. Sampling times should therefore be kept reasonably short when sampling with membrane filters in dusty environments, or depth filters should be used.

X2.2.2 Depth filters consist of fibers that have been pressed together to form an irregular three-dimensional mesh. Particles are not only retained at the surface, but also within the structure of the filter. This gives them a significantly higher dust loading capacity than membrane filters. In this respect, depth filters are a better choice than membrane filters when sampling for long periods in dusty environments. However, depth filters tend to have a higher metal content than membrane filters, and this needs to be considered when selecting the filter to be used if metals other than lead are to be determined.

### X2.3 Lead Content

X2.3.1 The lead content of the filters should be as low as possible, since it can make a significant contribution to the blank, the variability of which determines (in part) the lower limit of the working range of the analytical method. Exactly how low the lead content of the filters should be depends upon the applicable limit value. The lower limit of the working range of the analytical method should be less than the amount of lead that would be collected when sampling air at 0.1 times the limit value over the selected sampling period at the selected flow rate.

X2.3.2 Membrane filters generally have a very low lead content, and in this respect are suitable for nearly all applications.

X2.3.3 Glass fiber filters are unsuitable for use when measuring certain metals for which they have a high blank value. This is also true of quartz fiber filters, but to a lesser extent. Glass and quartz fiber filters do not present problems for lead measurements, but their potential use needs consideration if metals other than lead are to be measured.

### X2.4 Weight Stability

X2.4.1 If the filters are to be weighed in order to determine the amount of dust collected, it is important that they are reasonably resistant to moisture retention, so that blank weight changes that can occur as a result of changes in atmospheric conditions (temperature, humidity) are as low and as repeatable as possible.

X2.4.2 If glass or quartz fiber filters are used, it is important that these are not excessively friable, since this can introduce weighing errors due to loss of filter material. Quartz fiber filters can be more friable than glass fiber filters. However, this disadvantage is counterbalanced by their lower metal content.

### X2.5 Solubility

X2.5.1 The filters should be either wholly soluble or wholly insoluble using the selected sample preparation method. Partially dissolved filters can make subsequent handling of the sample solutions difficult, or they can cause analytical error, or both, because of a matrix mismatch between sample solutions and calibration solutions. If polyvinyl chloride (PVC) filters are chosen for sampling, it is recommended that microwave or ultrasonic dissolution procedures are used.

X2.5.2 If the sample preparation method selected involves quantitative transfer of the sample solution to volumetric glassware prior to analysis, the filters used for sampling should preferably be soluble using the sample preparation method concerned. This reduces the chance of incomplete transfer of the sample solution.

X2.5.3 Mixed cellulose ester membrane filters of 0.8 µm mean pore diameter are soluble in nitric acid, and these are suitable for use when this acid is used in the selected sample preparation environment. Quartz fiber filters are soluble in hydrofluoric acid, and are suitable for use when this acid is used. Other filters might be equally suitable. Certain types of filters, for example, PVC, may be partially soluble, and may cause problems for analysis.

X2.5.4 If sample solutions are to be made to volume in the sample dissolution vessel (for example, a graduated centrifuge tube), it is unimportant whether or not the filters are soluble using the selected sample preparation procedure.

## X3. TEMPERATURE AND PRESSURE CORRECTION

### X3.1 Temperature and Pressure Correction for the Indicated Volumetric Flow Rate

X3.1.1 Bubble flowmeters are preferred for measuring the volumetric flow rate because the readings they give are independent of temperature and pressure. For other flowmeters, it might be necessary to apply a correction to the indicated volumetric flow rate if the temperature and pressure at the time of measurement is different to when the calibration of the flowmeter was checked.

X3.1.2 A typical example of the need for a temperature and pressure correction is when a constant pressure drop, variable area, flowmeter is used to measure the volumetric flow rate. In this instance, use the following equation to calculate a corrected air sample volume:

$$V_{corr} = q_v \times t \times \sqrt{\frac{p_1 \times T_2}{p_2 \times T_1}}$$

where:

$V_{corr}$ = corrected volume, L,
$q_v$ = mean flow rate, L/min,
$t$ = sampling time, min,
$p_1$ = atmospheric pressure during calibration of the sampling pump flowmeter, kPa,
$p_2$ = mean atmospheric pressure during the sampling period, kPa,
$T_1$ = temperature during calibration of the sampling pump flowmeter, °K, and
$T_2$ = mean temperature during the sampling period, °K.

Any other flowmeter can also require a correction for variation in pressure and temperature; follow the manufacturer's instructions for such corrections.

### X3.2 Recalculation of Lead in Air Concentrations to Reference Conditions

X3.2.1 If necessary (see 10.1.3.2), recalculate lead in air concentrations to reference conditions (for example, 273°K and 101.3 kPa), using the following equation:

$$\rho[Pb]_{corr} = \rho[Pb] \times \frac{(101.3 \times T_2)}{(p_2 \times 273)}$$

where:

$\rho[Pb]_{corr}$ = concentration of lead in the air sample at ambient conditions, mg/m$^3$,
$\rho[Pb]$ = concentration of lead in the air sample at ambient conditions, mg/m$^3$,
$T_2$ = mean temperature during the sampling period, °K
$p_2$ = mean atmospheric pressure during the sampling period, kPa,
273 = reference temperature, °K, and
101.3 = reference atmospheric pressure, kPa.

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