



Designation: D 6785 – 02

Standard Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry¹

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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 *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

D 4840 Guide for Sample Chain-of-Custody Procedures

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E 1370 Guide for Air Sampling Strategies for Worker and Workplace Protection

2.2 Other Standards:⁴

ISO 648:1977, Laboratory Glassware—One-Mark Pipettes

ISO 1042:1983, Laboratory Glassware—One-Mark Volumetric Flasks

ISO 3585:1991, Glass Plant, Pipelines and Fittings—Properties of Borosilicate Glass 3.3

ISO 3696:1987, Water for Laboratory Use—Specifications and Test Methods

ISO 6879:1983, Air Quality—Performance Characteristics and Related Concepts For Air Quality Methods

ISO 6955:1982, Analytical Spectroscopic Methods—Flame Emission, Atomic Absorption, and Atomic Fluorescence—Vocabulary

ISO 7708:1995, Particle Size Definitions for Health Related Sampling

ISO 15202-2: 2001, Workplace Air—Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry—Part 2: Sample Preparation

EN 689:1995, Workplace Atmospheres—Guidance for the Assessment of Exposure to Chemical Agents for Comparison with Limit Values and Measurement Strategy

EN 1232:1997, Workplace Atmospheres—Pumps for Personal Sampling of Chemical Agents—Requirements and Test Methods

EN 1540:1998, Workplace Atmospheres—Terminology

¹ This test method is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.04 on Workplace Atmospheres.

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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, 25 W. 43rd St., 4th Floor, New York, NY 10036.

EN 12919: 1999, Workplace Atmospheres—Pumps for Sampling of Chemical Agents with a Volume Flow Rate of Over 5 L/Min—Requirements and Test Methods

EN 13205: 2001, 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 Pipettes⁵

EN ISO 8655-5 Piston-Operated Volumetric Instruments—Part 5: Dispensers⁵

EN ISO 8655-6 Piston-Operated Volumetric Instruments—Part 6: Gravimetric Test Methods⁵

3. Terminology

3.1 General Definitions:

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 *limit value*—reference figure for concentration of a chemical agent in air. **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**

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

3.2.4 *respirable fraction*—the mass fraction of inhaled particles penetrating to the unciliated airways. **ISO 7708**

3.2.5 *total airborne particles*—all particles surrounded by air in a given volume of air.

3.2.5.1 *Discussion*—Because all measuring instruments are size-selective to some extent, it is often impossible to measure the total airborne particles concentration. **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.

⁵ To be published.

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. It is expressed, as a percentage, by a combination of bias and precision, usually in accordance with the formula:

$$\frac{|\bar{x} - x_{ref}| + 2s}{x_{ref}} \times 100$$

where:

\bar{x} = mean value of results of a number (n) of repeated measurements,

x_{ref} = true or accepted reference value of concentration, and

s = standard deviation of repeated measurements. **EN 482**

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.

NOTE 1—The inhalable convention may not be the regulated sampling convention in all countries where this standard may be applicable. In the USA, for example, limit values promulgated by the Occupational Safety and Health Administration (OSHA) (29 CFR 1910.1025) are based on the use of samplers that were not specifically designed to meet the inhalable convention.

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 (see 6.1 of ISO 6955).

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 (see 6.1 of ISO 6955).

4.5 The results may be used for the assessment of workplace exposures to airborne particulate lead (see EN 689).

5. Reactions

5.1 In general, the overwhelming majority of particulate lead compounds that are commonly found in samples of workplace air are converted to water-soluble lead ions (Pb^{2+}) by the sample dissolution procedures described in 11.2. However, certain lead compounds, for example lead silicate, might not be dissolved. If necessary, a dissolution procedure employing hydrofluoric acid should be used to dissolve silicate lead. If there is any doubt about the effectiveness of these procedures for the dissolution of particulate lead compounds that may be present in the test atmosphere, then this shall be investigated before proceeding with the method (see Section 11).

6. Significance and Use

6.1 The health of workers in many industries, for example, mining, metal refining, battery manufacture, construction, etc., is at risk through exposure by inhalation of particulate lead and lead compounds. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure, and this is generally achieved by making workplace air measurements. This standard has been published in order to make available a method for making valid exposure measurements for lead. It will be of benefit to: agencies concerned with health and safety at work; industrial hygienists and other public health professionals; analytical laboratories; industrial users of metals and metal-oids and their workers, etc. It has been assumed in the drafting of this standard that the execution of its provisions, and the interpretation of the results obtained, is entrusted to appropriately qualified and experienced people.

6.2 The measuring procedure shall comply with any relevant International, European or National Standard that specifies performance requirements for procedures for measuring chemical agents in workplace air (for example, EN 482).

7. Reagents

NOTE 2—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 ISO 3696 grade 2 water (electrical conductivity less than 0.1 mS/m and resistivity greater than 0.01 M Ω .m at 25 °C). The concentration of lead shall be less than 0.01 μ g/mL.

NOTE 3—It is recommended that the water used be obtained from a water purification system that delivers ultrapure water having a resistivity greater than 0.18 M Ω .m (usually expressed by manufacturers of water purification systems as 18 M Ω .cm).

7.2 *Nitric Acid (HNO₃)*, concentrated, ρ about 1.42 g/mL, about 70 % (m/m). The concentration of lead shall be less than 0.01 μ g/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 (8.6.1.4). 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 (8.6.1.4). 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 $\mu\text{g/mL}$. (**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*, $\text{NH}_4\text{H}_2\text{PO}_4$, $\text{Mg}(\text{NO}_3)_2$ or $\text{Pd}(\text{NO}_3)_2$, 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 \pm 0.001 g of lead (II) nitrate [$\text{Pb}(\text{NO}_3)_2$], previously dried to constant mass at 110°C and cooled in a desiccator, in 200 mL of 1 + 1 nitric acid (7.3). Quantitatively transfer the solution to a 1000 mL one-mark volumetric flask (8.6.1.4). Dilute to the mark with water (7.1), stopper and mix thoroughly. Store in a suitable container, for example, a polypropylene bottle (8.6.2.2), for a maximum period of one year.

7.7.1.2 Dissolve 1.000 g \pm 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 (8.6.1.4), dilute to the mark with water (7.1), stopper and mix thoroughly. Store in a suitable container, for example, a polypropylene bottle (8.6.2.2), 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

(8.6.1.4). 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 (8.6.2.2), 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 $\mu\text{g/mL}$.

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

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

8. Apparatus

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

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

NOTE 5—Some inhalable samplers are designed to collect the fraction of airborne particles on a filter, and any particulate matter deposited on the internal surfaces of the sampler is not of interest. Other inhalable samplers are designed such that 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.) The operating instructions supplied by the manufacturer should be consulted to find out whether particulate matter deposited on the internal surfaces of the sampler forms part of the sample.

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

NOTE 6—See Appendix X1 for guidance on filter selection.

8.3 Sampling Pumps:

8.3.1 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).

NOTE 7—A flow-stabilized pump may be required to maintain the flow rate within the specified limits.

8.3.2 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 8—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 9—EN 1232 and EN 12919 require 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.

8.4 *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 10—It is recommended that the flowmeter used should be capable of measuring the volumetric flow rate to within ± 2 % or better.

8.5 Ancillary Equipment:

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

8.5.2 *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).

8.5.3 *Flat-tipped Forceps*, for loading and unloading filters into samplers.

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

8.5.5 *Barometer*, suitable for measurement of atmospheric pressure, if required (see 10.1.3).

8.5.6 *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.

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

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

NOTE 11—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.

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

8.6.1.2 *One-mark Pipets*, complying with the requirements of ISO 648.

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

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

8.6.2 *Plastic Labware*:

8.6.2.1 *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.

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

8.6.3 *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.

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

NOTE 12—The efficiency of thermostating of hot plates is sometimes deficient, and the surface temperature can also vary considerably with position on hot plates with large surface areas. It is therefore recommended that the performance of the hot plate be characterized prior to use.

8.6.5 *Microwave Digestion Apparatus*:

(**Caution**—Ensure that manufacturer's safety recommendations are followed.)

NOTE 13—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 14—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.

8.6.5.1 *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. (**Caution**—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 15—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.

8.6.5.2 *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. (**Caution**— 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.)

8.6.6 *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/cm² power density or greater).

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

8.6.8 *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 Note 29 in 11.2.2.3 and Note 30 in 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).

8.6.9 *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 16—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.

8.6.10 *Analytical Balance*, capable of weighing to ±0.1 mg, if required, for use in preparation of stock standard lead solution.

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

8.6.12 *Forceps*, 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 E 1370) 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 E 1370) 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 17—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.