



Designation: D8568 – 24

# Standard Test Method for Determination of Lead by Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques<sup>1</sup>

This standard is issued under the fixed designation D8568; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of lead (Pb) in airborne particulate, dust by wipe and micro-vacuuming, paint, and soil collected in and around buildings and related structures using graphite furnace atomic absorption spectrometry (GFAAS).

1.2 This test method contains directions for sample analysis, as well as quality assurance (QA) and quality control (QC), and may be used for purposes of laboratory accreditation and certification.

1.3 No detailed operating instructions are provided because of differences among various makes and models of suitable GFAAS instruments. Instead, the analyst shall follow the instructions provided by the manufacturer of the particular instrument.

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

1.4.1 *Exception*—The SI and inch-pound units shown for wipe and micro-vacuuming sampling data are to be individually regarded as standard for wipe and micro-vacuuming sampling data.

1.5 This test method contains notes which are explanatory and not part of the mandatory requirements of this standard.

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

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

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.12 on Sampling and Analysis of Lead for Exposure and Risk Assessment.

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## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- D1193 Specification for Reagent Water
- D1356 Terminology Relating to Sampling and Analysis of Atmospheres
- D3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry
- D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn 2002)<sup>3</sup>
- D4532 Test Method for Respirable Dust in Workplace Atmospheres Using Cyclone Samplers
- D4697 Guide for Maintaining Test Methods in the User's Laboratory (Withdrawn 2009)<sup>3</sup>
- D4840 Guide for Sample Chain-of-Custody Procedures
- D6785 Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry
- D6966 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals
- D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D7144 Practice for Collection of Surface Dust by Microvacuum Sampling for Subsequent Determination of Metals and Metalloids
- D8358 Guide for Assessment and Inclusion of Wall Deposits in the Analysis of Single-Stage Samplers for Airborne Particulate Matter
- E456 Terminology Relating to Quality and Statistics
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E1188 Practice for Collection and Preservation of Information and Physical Items by a Technical Investigator

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

- [E1583 Practice for Evaluating Laboratories Engaged in Determination of Lead in Paint, Dust, Airborne Particulates, and Soil Taken From and Around Buildings and Related Structures](#)
- [E1605 Terminology Relating to Lead in Buildings](#)
- [E1644 Practice for Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead](#)
- [E1645 Practice for Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis](#)
- [E1726 Practice for Preparation of Soil Samples by Hotplate Digestion for Subsequent Lead Analysis](#)
- [E1727 Practice for Field Collection of Soil Samples for Subsequent Lead Determination](#)
- [E1728 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination](#)
- [E1729 Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination](#)
- [E1741 Practice for Preparation of Airborne Particulate Lead Samples Collected During Abatement and Construction Activities for Subsequent Analysis by Atomic Spectrometry \(Withdrawn 2009\)<sup>3</sup>](#)
- [E1775 Guide for Evaluating Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead](#)
- [E1792 Specification for Wipe Sampling Materials for Lead in Surface Dust](#)
- [E1864 Practice for Evaluating Quality Systems of Organizations Conducting Facility and Hazard Assessments for Lead in Paint, Dust, Airborne Particulate, and Soil in and around Buildings and Related Structures \(Withdrawn 2011\)<sup>3</sup>](#)
- [E1908 Practice for Sample Selection of Debris Waste from a Building Renovation or Lead Abatement Project for Toxicity Characteristic Leaching Procedure \(TCLP\) Testing for Leachable Lead \(Pb\)](#)
- [E1979 Practice for Ultrasonic Extraction of Paint, Dust, Soil, and Air Samples for Subsequent Determination of Lead](#)
- [E2115 Guide for Conducting Lead Hazard Assessments of Dwellings and of Other Child-Occupied Facilities](#)
- [E2239 Practice for Record Keeping and Record Preservation for Lead Hazard Activities](#)
- [E2271/E2271M Practice for Clearance Examinations Following Lead Hazard Reduction Activities in Multifamily Dwellings](#)
- [E2913/E2913M Practice for Hotplate Digestion of Lead from Composited Wipe Samples](#)
- [E2914/E2914M Practice for Ultrasonic Extraction of Lead from Composited Wipe Samples](#)
- [E3074/E3074M Practice for Clearance Examinations Following Lead Hazard Reduction Activities in Single Family Dwellings, in Individual Units of Multifamily Dwellings, and in Other Child-Occupied Facilities](#)

2.2 *ISO/IEC Standard*:<sup>4</sup>

[ISO/IEC 17025](#) General requirements for the competence of testing and calibration laboratories

### 3. Terminology

3.1 *Definitions*—For definitions of terms not appearing here, see Terminologies [D1356](#), [E456](#), and [E1605](#).

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *analysis run, n*—a period of measurement time on a given analytical instrument during which data are calculated from a single calibration curve (or single set of such curves).

3.2.1.1 *Discussion*—Recalibration of a given GFAAS instrument produces a new analysis run.

3.2.2 *calibration standards, n*—solutions of known analyte (Pb) concentrations used to calibrate instruments.

3.2.2.1 *Discussion*—Calibration standards are matrix matched to the acid content present in sample digestates or extracts and are measured prior to analyzing samples.

3.2.3 *initial calibration blank (ICB), n*—a standard containing no analyte (Pb) that is used for the initial calibration and zeroing of the instrument response.

3.2.3.1 *Discussion*—The ICB is matrix matched to the acid content of sample extracts and digestates. The ICB is measured during and after calibration. The measured Pb value is to be (at maximum) less than five times the IDL (see [3.2.7](#)).

3.2.4 *initial calibration verification (ICV), n*—a solution (or set of solutions) of known analyte (Pb) concentration used to verify calibration standard levels; the concentration of analyte is to be near the mid-range of the linear curve that is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards.

3.2.4.1 *Discussion*—The ICV is matrix matched to the acid content of sample extracts or digestates. The ICV is measured after calibration and before measuring any sample digestates or extracts. The measured Pb value is to fall within  $\pm 10\%$  of the known value.

3.2.5 *instrumental detection limit (IDL), n*—the lowest concentration at which the instrumentation can distinguish analyte (Pb) content from the background generated by a minimal matrix.

3.2.5.1 *Discussion*—The IDL is usually determined by the instrument manufacturer. The IDL can be determined from blank, acidified, deionized, or ultrapure water as the matrix and from the same calculation methods used to determine a method detection limit (MDL) (see [3.2.10](#)). Typical lead (Pb) IDLs for GFAAS are near 0.002  $\mu\text{g Pb/mL}$ .

3.2.6 *method blank, n*—a digestate or extract that reflects the maximum treatment given to any one sample within a sample batch, except that no sample is placed into the digestion or extraction vessel.

3.2.6.1 *Discussion*—The same reagents and processing conditions that are applied to field samples within a batch are also

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

applied to the method blank. Analysis results from method blanks provide information on the level of potential contamination experienced by samples processed within the batch.

3.2.7 *method detection limit (MDL), n*—the minimum concentration of analyte (Pb) that, in a given matrix and with a specified analytical method, has a 99 % probability of being identified and is reported to be greater than zero concentration.

3.2.7.1 *Discussion*—As an example, the MDL for lead in paint is the smallest measurable (that is, nonzero) concentration of lead within the paint sample as determined by the validated extraction and analysis method used. Note that there would be a different MDL for different sample matrices (such as dust wipes, air particulates, and soils), even if the sample preparation and analysis process is the same for all types of matrices. Thus each sample matrix has a unique MDL, given in units specific to the matrix, even if the analyte content is the same for each.

NOTE 1—For instance, for dust wipe samples, different brands of wipe media could have different Pb MDLs. Dust wipes and paint samples will have lead contents expressed in different units.

3.2.7.2 *Discussion*—There are four component inputs to defining an MDL: (1) the analyte of interest (that is, lead (Pb) for our purposes here); (2) the sample matrix (for example: paint, dust, or brand *x* wipe, soil, or air particulate collected on type *x* sampling media); (3) the extraction/digestion procedure used; and (4) the analysis procedure (using the type of GFAAS instrumentation and components) used for quantification of analyte content. The MDL is established prior to reporting analysis data.

3.2.8 *quantitation limit, n*—an instrumental measurement value that is used to provide a lower Pb concentration limit for reporting quantitative analysis data for a given analytical method.

3.2.8.1 *Discussion*—Any sample that generates a lead measurement below the quantitation limit is reported as a less-than value using the quantitation limit value multiplied by the appropriate dilution factors resulting from preparation of the sample for instrumental analysis.

3.2.9 *quantitative analysis, n*—an analysis run on sample digestates or extracts (or serial dilutions thereof) that includes instrumental QC standards.

3.2.9.1 *Discussion*—Data from this analysis run are used to calculate and report final lead analysis results.

3.2.10 *semiquantitative analysis, n*—an analysis run that is performed on highly diluted sample digestates or extracts for the purpose of determining the approximate analyte (Pb) level in the digest.

3.2.10.1 *Discussion*—This analysis run is generally performed without inserting instrumental QC standards except for calibration standards. Data from this run are used for determining serial dilution requirements for sample digestates or extracts to keep them within the linear range of the instrument.

3.2.11 *serial dilution, n*—a method of producing a less-concentrated solution through one or more consecutive dilution steps.

3.2.11.1 *Discussion*—A dilution step for a standard or sample solution is performed by volumetrically placing a small

aliquot (of known volume) of a higher concentrated solution into a volumetric flask and diluting to volume with water containing the same acid levels as those found in original sample digestates or extracts.

3.2.12 *spiked duplicate sample, n*—two portions of a homogenized sample that were targeted for addition of analyte (Pb) and fortified with all the target analytes before preparation.

3.2.12.1 *Discussion*—Analysis results for these samples are used to provide information on the precision and bias of the overall analysis process.

3.2.13 *spiked sample, n*—a sample portion (split from an original sample) that is spiked with a known amount of analyte (Pb).

3.2.13.1 *Discussion*—Analysis results for spiked samples are used to provide information on the precision and bias of the overall analysis process.

3.2.14 *un-spiked sample, n*—a portion of a homogenized sample that was targeted for the addition of analyte (Pb) but is not fortified with target analytes before sample preparation.

3.2.14.1 *Discussion*—Analysis results for this sample are used to correct for native analyte (Pb) levels in the spiked and spiked duplicate samples.

## 4. Summary of Test Method

4.1 A sample digestate or extract of a sample of air particulate, dust (wipe or vacuum sample), paint or soil is analyzed for lead content using graphite furnace atomic absorption spectrometric (GFAAS) instrumentation (Practice D3919; **1, 2, 3**).<sup>5</sup> Quality control (QC) samples are analyzed along with sample digestates or extracts in order to ensure adequate instrumental performance.

NOTE 2—Digestion is an example of an extraction process. Other examples of extraction processes are ultrasonic extraction (**4**) and leaching (Guide E1908).

## 5. Significance and Use

5.1 Environmental (including workplace) samples obtained during the assessment or mitigation of lead hazards from buildings and related structures are analyzed to determine lead content in media of concern. This test method is intended for use with other ASTM standards (see 2.1) that address the collection and preparation of samples (airborne particulate, dusts by wipe and micro-vacuuming, dried paint chips, and soils) that are obtained during the assessment or mitigation of lead hazards. This test method may be used to analyze samples collected from various environments, such as workplaces, buildings, indoor or outdoor settings, construction sites, housing, and so on.

5.2 This test method may also be used to analyze similar samples from other environments such as toxic characteristic extracts of waste sampled using Guide E1908, and soil and sludge as prepared for analysis using U.S. EPA SW-846 Test Method 1311 (**5**).

<sup>5</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.



5.3 This test method can be relied upon by laboratories seeking accreditation for lead analysis by means of GFAAS.

## 6. Interferences

6.1 Interferences for GFAAS can be manufacturer- and model-specific. The following are general guidelines:

6.1.1 Molecular absorption is a potential interference in GFAAS (6). These interferences can be minimized by using techniques such as D<sub>2</sub> or H<sub>2</sub> continuum background correction (7).

6.1.2 High concentrations (for example, 100- to 1000-fold excess compared to lead concentration) of calcium, sulfate, phosphate, iodide, fluoride, or acetate can interfere with lead determination by GFAAS (7, 8). These interferences can be corrected by standard addition techniques (9).

6.1.3 Other sources of interference may be found for various matrices; these are discussed in more detail elsewhere (6, 8).

## 7. Apparatus and Materials

7.1 *Analytical Instrumentation*—The instrumentation used shall consist of one or more of the following apparatus:

7.1.1 *Graphite Furnace Atomic Absorption Spectrometer (GFAAS)*, equipped with background correction, lead hollow cathode lamp, or discharge lamp without electrodes, and capable of making lead absorption measurements at the 283.3 nm absorption line (see Practice D3919).

NOTE 3—For lead, the 283.3 nm line is normally preferred over the 217.0 nm line because of the increased noise levels commonly observed at 217.0 nm for GFAAS.

NOTE 4—GFAAS is sometimes referred to as electrothermal atomic absorption spectrometry (ETAAS).

NOTE 5—The operating parameters for graphite furnace atomic absorption can vary considerably between different instruments.

7.2 *Argon*, compressed, in grade specified by the manufacturer of the GFAAS instrument used.

7.3 *Vinyl Gloves*, powderless.

7.4 *Micropipettors with Disposable Plastic Tips*, in sizes necessary to make reagent additions, serial dilutions, and spiking standards. In general, the following sizes should be readily available: 1 mL to 5 mL adjustable and 1000 µL, 500 µL, 250 µL, and 100 µL.

7.5 *Volumetric Flasks*, in sizes necessary to make calibration standards, serial dilutions, and instrumental QC standards.

## 8. Reagents

8.1 *Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification D1193. (ASTM Type I Water: minimum resistance of 16.67 MΩ-cm or equivalent.)

8.2 *Nitric Acid*, concentrated, suitable for atomic spectrometry analysis (such as spectroscopic grade).

8.3 *Calibration Stock Solution*, 100 µg/mL of lead in dilute nitric acid or equivalent (such as a multielement stock containing lead).

8.4 *Check Standard Stock Solution (for ICV)*, 100 µg/mL of lead in dilute nitric acid or equivalent. This shall be from a

different lot number (or manufacturer) than the calibration stock solution (see 8.3).

## 9. Preparation of Labware

9.1 Wash glassware and plastic equipment with laboratory detergent, rinse with tap water, soak for at least 4 h in volume fraction 35 % (volume/volume 1+1) nitric acid and water, rinse three times with ASTM Type I Water, and allow to dry preferably in a fume hood. Commercial, automatic systems are available that perform a similar process.

9.2 Alternatively, soak glassware and plastic equipment in volume fraction 35 % (volume/volume 1+1) nitric acid and water in a plastic tub preferably in a working hood with the hood sash down, rinse three times with ASTM Type I Water, and allowed to dry preferably in a fume hood.

## 10. Sample Collection and Preparation

10.1 *Sample Collection*—Samples should be collected, as appropriate to the matrix of interest, using Test Method D4532, Practices D6966, D7144, E1727, E1728, E1729, E2271/E2271M, E3074/E3074M, or Guide D8358, or combinations thereof.

10.2 *Sample Preparation*—Samples should be prepared for analysis, as appropriate to the matrix of interest, using Practices E1644, E1645, E1726, E1741, E1979, E2913/E2913M, or E2914/E2914M.

## 11. Requirement for Laboratory Analysis

11.1 Laboratories analyzing samples obtained during the assessment or mitigation of lead hazards from buildings and related structures shall conform to Practice E1583, or shall be recognized for lead analysis as promulgated by authorities having jurisdiction, or both.

NOTE 6—In the United States of America, laboratories performing analysis of samples collected during lead-based paint activities are required to be accredited to ISO/IEC 17025 and to other requirements promulgated by the Environmental Protection Agency (EPA).

## 12. Procedure

12.1 *Laboratory Records*—Record all reagent sources (lot numbers and vendors) used for sample preparation and analysis in a laboratory notebook. Record any inadvertent deviations, unusual happenings, or observations on a real-time basis as the samples are processed. Use these records to add supplemental information when reporting the results.

12.2 *Instrumental Setup*:

12.2.1 Set the GFAAS spectrometer up for the analysis of lead at 283.3 nm, in accordance with the instructions given by the manufacturer. Allow an appropriate warm-up of the system prior to analysis.

12.3 *Preparation of Calibration and Instrumental QC Standards*:

12.3.1 *Calibration Standards*—Prepare a series of calibration standards (minimum of three) covering the linear range of the GFAAS instrumentation. Prepare these standards using