INTERNATIONAL

Designation: ~~E3193 – 19~~ E3193 – 20

Standard Test Method for Measurement of Lead (Pb) in Dust by Wipe, Paint, and Soil by Flame Atomic Absorption Spectrophotometry (FAAS)[1]

This standard is issued under the fixed designation E3193; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of lead (Pb) in dust by wipe, paint, and soil collected in and around buildings and related structures by flame atomic absorption spectrophotometry (FAAS) and is derived from Test Methods D4185 and E1613. For determination of lead (Pb) and other metals in air by FAAS, see Test Method D4185.

1.2 The sensitivity, detection limit, and optimum working concentration for lead (Pb) are given in Table 1.

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

1.3.1 *Exception*—The SI and inch-pound units shown for wipe sampling data are to be individually regarded as standard for wipe sampling data (13.4.1).

1.4 *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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water
D1356 Terminology Relating to Sampling and Analysis of Atmospheres
D4185 Test Method for Measurement of Metals in Workplace Atmospheres by Flame Atomic Absorption Spectrophotometry
D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn 2002)[3]
D4697 Guide for Maintaining Test Methods in the User's Laboratory (Withdrawn 2009)[3]
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)
E456 Terminology Relating to Quality and Statistics
E631 Terminology of Building Constructions
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
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

[1] 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,~~Analysis of ~~Lead,~~Lead for Exposure and Risk Assessment.

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[2] 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

TABLE 1 FAAS Instrumental Detection Limit and Optimum Working Concentration for Lead (Pb)

Element	Detection Limit, µg/mL (approximately three times standard deviation of blank)[A]	Optimum Linear Range Upper Limit, µg/mL	Elements, Compound Classes, and Oxides
Pb	0.02	10	Inorganic compounds, paint, dust by wipe, soil

[A] Detection limit represents ideal laboratory conditions; variability due to sampling, digestion, reagents, and sample handling has not been taken into account.

E1605 Terminology Relating to Lead in Buildings
E1613 Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques
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)[3]
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)[3]
E1908 Guide 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 *Code of Federal Regulations:*[4]
40 CFR 745 Lead-Based Paint Poisoning Prevention in Certain Residential Structures

2.3 *Governmental Agency Guidance:*[5]
U.S. Environmental Protection Agency National Lead Laboratory Accreditation Program (NLLAP)
U.S. Environmental Protection Agency SW-846 Test Method 1311: Toxicity Characteristic Leaching Procedure

2.4 *ISO Standard:*[6]
ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminologies D1356, E456, E631, 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 curves).

3.2.1.1 *Discussion*—

Recalibration of a given instrument produces a new analysis run.

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

3.2.2.1 *Discussion*—

[4] Available from U.S. Government Publishing Office, 732 N. Capitol St., NW, Washington, DC 20401, http://www.gpo.gov.

[5] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

[6] Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

Calibration standards must be matrix matched to the acid content present in sample digestates or extracts and must be measured prior to analyzing samples.

~~3.2.3 *continuing calibration blank (CCB), n*—a solution containing no analyte that is used to verify blank response and absence of carryover.~~

~~3.2.3.1 *Discussion*—~~

~~The CCB must be analyzed after the continuing calibration verification (CCV). The measured value is to be (at most) less than five times the instrumental detection limit (IDL).~~

~~3.2.4 *continuing calibration verification (CCV), n*—a solution (or set of solutions) of known analyte concentration used to verify absence of excessive instrumental drift.~~

~~3.2.4.1 *Discussion*—~~

~~The CCV must be matrix matched to the acid content present in sample digestates or extracts at a concentration near the mid-range of a linear calibration curve. The CCV must be analyzed before and after all samples and at a frequency of not less than every ten samples. The measured value is to fall within ±10 % (±20 % for graphite furnace atomic absorption (GFAA)) of the known value.~~

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

3.2.3.1 *Discussion*—

The ICB must be matrix matched to the acid content of sample extracts and digestates. The ICB must be measured during and after calibration. The measured value is to be (at most) less than five times the IDL.

3.2.4 *initial calibration verification (ICV), n*—a solution (or set of solutions) of known analyte 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 must be matrix matched to the acid content of sample extracts or digestates. The ICV must be measured after calibration and before measuring any sample digestates or extracts. The measured value is to fall within ±10 % of the known value.

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

3.2.5.1 *Discussion*—

The IDL is usually determined by the 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). Typical lead (Pb) IDL for FAAS is 0.05 µg/mL.

3.2.6 *instrumental QC standards, n*—these provide information on measurement performance during the instrumental analysis portion of the overall analyte measurement process. They include CCBs, CCVs, ICBs, and ICVs.

~~3.2.9 *limit of detection (LOD), n*—the MDL or the IDL, depending on the context.~~

3.2.7 *method blank, n*—a digestate or extract that reflects the maximum treatment given any one sample within a sample batch, except that no sample is placed into the digestion or extraction vessel. (The same reagents and processing conditions that are applied to field samples within a batch are also applied to the method blank.)

3.2.7.1 *Discussion*—

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

~~3.2.11 *method detection limit (MDL), n*—the minimum measured concentration of a substance that can be reported with 99 % confidence that the measured concentration is distinguishable from method blank results (**1**).[7]~~

~~3.2.11.1 *Discussion*—~~

(a) 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, paints, 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.

(1) For instance, for dust wipe samples, different brands of wipes could have different MDLs. Dust wipes, soil, and paint samples would have lead contents expressed in different units.

(b) There are thus 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, or soil); (*3*) the *extraction/digestion procedure* used; and (*4*) the *analysis procedure* (includes the type of instrument) used for quantification of analyte content. The MDL must be established prior to reporting analysis data.

3.2.8 *quantitation limit, n*—an instrumental measurement value that is used to provide a lower 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 *reporting limit, n*—the lowest concentration of analyte in a sample that can be reported with a defined, reproducible level of certainty.

3.2.10.1 *Discussion—*

This value is usually based on the low standard used for instrument calibration. For analyses in the United States of America subject to 40 CFR 745 and the National Lead Laboratory Accreditation Program (NLLAP), the reporting limit for a specific sample matrix must be at least twice the MDL for that same sample matrix.

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

3.2.11.1 *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.

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

3.2.12.1 *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.

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

3.2.13.1 *Discussion—*

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

3.2.14 *spiked sample, n*—a sample portion (split from an original sample) to which an additional known amount of analyte has been added.

3.2.14.1 *Discussion—*

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

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

3.2.15.1 *Discussion—*

Analysis results for this sample are used to correct for native analyte levels in the spiked and spiked duplicate samples.

4. Summary of Test Method

4.1 A sample digestate or extract is analyzed for lead content using FAAS. Instrumental QC samples are analyzed along with sample digestates or extracts in order to ensure adequate instrumental performance.

NOTE 1—Digestion is an example of an extraction process. Other examples of extraction processes are ultrasonic extraction and leaching.

4.2 Samples and standards are aspirated into the flame of an absorption spectrophotometer. A lead (Pb) hollow cathode or electrodeless discharge lamp provides the characteristic radiation energy for lead. The absorption of this characteristic energy by lead atoms in the flame is related to the concentration of lead in the aspirated sample. The flame and operating conditions for lead are listed in Table 2.[7]

5. Significance and Use

5.1 This test method is intended for use with other standards that address the collection and preparation of samples (dusts by wipe, dried paint chips, and soils) that are obtained during the assessment or mitigation of lead hazards from buildings and related structures.

5.2 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 2—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).

5.3 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.

6. Interferences

6.1 In FAAS, the occurrence of interferences is less common than in many other analytical techniques. Interferences can occur, however, and when encountered are corrected as indicated in the following sections. The known interferences and correction methods for each metal are indicated in Table 2. The methods of standard additions and background monitoring and correction (**2-5**) are used to identify the presence of an interference. Insofar as possible, the matrix of sample and standard are matched to minimize possible interference.

TABLE 2 FAAS Flame and Operating Conditions for Lead (Pb)

Element	Type of Flame	Analytical Wavelength, nm	Interferences[A]	Remedy[A]	Reference
~~Pb~~	~~Air-C_2H_2 (oxidizing)~~	~~217.0~~ ~~283.3~~	~~Ca, high concentration~~ ~~SO_4^{-2}~~	[B]	(~~2~~)

TABLE 2 FAAS Flame and Operating Conditions for Lead (Pb)

Element	Type of Flame	Analytical Wavelength, nm	Interferences[A]	Remedy[A]	Reference
Pb	Air-C_2H_2 (oxidizing)	217.0 283.3	Ca, high concentration SO_4^{-2}	[B] –	(**1**)

[A] High concentrations of silicon in the sample can cause an interference for many of the elements in this table and may cause aspiration problems. No matter what elements are being measured, if large amounts of silica are extracted from the samples, the samples should be allowed to stand for several hours and centrifuged or filtered to remove the silica.
[B] Samples are periodically analyzed by the method of additions to check for chemical interferences. If interferences are encountered, determinations must be made by the standard additions method or, if the interferent is identified, it may be added to the standards.

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

6.2 Background or nonspecific absorption can occur from particles produced in the flame, which can scatter light and produce an apparent absorption signal. Light scattering may be encountered when solutions of high salt content are being analyzed. They are most severe when measurements are made at shorter wavelengths (for example, below about 250 nm). Background absorption may also occur as the result of the formation of various molecular species which can absorb light. The background absorption can be accounted for by the use of background correction techniques (42).

6.3 Spectral interferences are those interferences which result from an atom different from the one being measured that absorbs a portion of the radiation. Such interferences are extremely rare in FAAS. In some cases, multi-element hollow cathode lamps may cause a spectral interference by having closely adjacent emission lines from two different elements. In general, the use of multi-element hollow cathode lamps is discouraged.

6.4 Ionization interference occurs when easily ionized atoms are being measured. The degree to which such atoms are ionized is dependent upon the atomic concentration and the presence of other easily ionized atoms. This interference can be controlled by the addition of a high concentration of another easily ionized element which will buffer the electron concentration in the flame.