Designation: E1613 – 04

Standard 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[1]

This standard is issued under the fixed designation E1613; 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 is intended for use with extracted or digested samples that were collected during the assessment, management, or abatement of lead hazards from buildings, structures, or other locations.

1.2 This test method covers the lead analysis of sample extracts or digestates (for example, extracted or digested paint, soil, dust, and airborne particulate) using inductively coupled plasma atomic emission spectrometry (ICP-AES), flame atomic absorption spectrometry (FAAS), or graphite furnace atomic absorption spectrometry (GFAAS).

1.3 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.

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

1.5 The values stated in SI units are to be regarded as the standard.

1.6 This practice contains notes which are explanatory and not part of the mandatory requirements of this standard.

1.7 *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:*[2]

D1193 Specification for Reagent Water

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[3]

D4697 Guide for Maintaining Test Methods in the User's Laboratory[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

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

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

[1] This test method is under the jurisdiction of ASTM Committee E06 on Performance of Buildings and is the direct responsibility of Subcommittee E06.23 on Lead Hazards Associated with Buildings.

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] Withdrawn.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

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[3]
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
E1973 Practice for Collection of Surface Dust by Air Sampling Pump Vacuum Technique for Subsequent Lead Determination[3]
E1979 Practice for Ultrasonic Extraction of Paint, Dust, Soil, and Air Samples for Subsequent Determination of Lead
E2239 Practice for Record Keeping and Record Preservation for Lead Hazard Activities

3. Terminology

3.1 *Definitions:* For definitions of terms not appearing here, see Terminology E1605.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *analysis run*—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*—solutions of known analyte concentrations used to calibrate instruments.

3.2.2.1 *Discussion*—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)*—a solution containing no analyte that is used to verify blank response and freedom from carryover.

3.2.3.1 *Discussion*—The CCB must be analyzed after the CCV (see 3.2.4) and after the ICkS (see 3.2.9). The measured value is to be (at most) less than five times the instrumental detection limit (IDL) (see 3.2.7).

3.2.4 *continuing calibration verification (CCV)*—a solution (or set of solutions) of known analyte concentration used to verify freedom from excessive instrumental drift; the concentration is to be near the mid-range of a linear calibration curve.

3.2.4.1 *Discussion*—The CCV must be matrix matched to the acid content present in sample digestates or extracts. 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 GFAA) of the known value.

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

3.2.5.1 *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 (see 3.2.7).

3.2.6 *initial calibration verification (ICV)*—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.

3.2.6.1 *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.

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

3.2.7.1 *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) (see 3.2.12). Typical lead (Pb) IDLs for FAAS, ICP-AES, and GFAAS are 0.05, 0.03, and 0.002 µg/mL, respectively.

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

3.2.9 *interference check standard (ICkS)*—a solution (or set of solutions) of known analyte concentrations used for ICP-AES to verify an accurate analyte response in the presence of possible spectral interferences from other analytes that may be present in samples; the concentration of analyte is to be less than 25 % of the highest calibration standard, and concentrations of the interferences will be 200 µg/mL of aluminum, calcium, iron, and magnesium.

3.2.9.1 *Discussion*—The ICkS must be matrix matched to the acid content of sample digestates or extracts. The ICkS must be analyzed at least twice, once before and once after the analysis of all sample extracts or digestates. The measured analyte value is expected to be within ±20 % of the known value.

3.2.10 *method blank*—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.)

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

3.2.11 *limit of detection (LOD)*—the MDL (see 3.2.12) or the IDL (see 3.2.7), depending on the context.

3.2.12 *method detection limit (MDL)*—the minimum concentration of analyte 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.

3.2.12.1 *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, air filters, 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 wipes could have different MDLs. Dust wipes 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, soil, or air particulate collected on type x filter); (*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.

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

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

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

3.2.14.1 *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.

3.2.15 *semiquantitative analysis*—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.

3.2.15.1 *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.

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

3.2.16.1 *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.

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

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

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

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

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

3.2.19.1 *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 ICP-AES, FAAS, or GFAAS techniques (**4, 1, 2**)[4]. Instrumental 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 (**3**) and leaching.

5. Significance and Use

5.1 This test method is intended for use with other standards (see 2.1) that address the collection and preparation of samples (dried chips, dusts, soils, and air particulates) that are obtained during the assessment or mitigation of lead hazards from buildings and related structures.

5.2 This test method may also be used to analyze similar samples from other environments.

6. Interferences

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

6.1.1 Special interferences may be encountered in ICP-AES analysis (**5**). These interferences can be minimized by proper wavelength selection, interelement correction factors, and background correction (**6**).

6.1.2 Molecular absorption is a potential interference in both FAAS and GFAAS (**7**). These interferences can be minimized by using techniques such as D_2 or H_2 continuum (FAAS and GFAAS) or Zeeman (GFAAS) background correction (**8**).

6.1.3 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 FAAS or GFAAS (**8**). These interferences can be corrected by standard addition techniques (**9**).

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

6.1.4 Other sources of interference may be found for various matrices; these are discussed in more detail elsewhere (**7**, **10**).

7. Apparatus and Materials

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

7.1.1 *ICP-AES*, either sequential or simultaneous, axial or radial, and capable of measuring at least one of the primary ICP lead emission lines. The emission line used must be demonstrated to have freedom from common major interferants such as aluminum, calcium, iron, and magnesium; alternatively, the instrument must have the capability to correct for these interferants.

NOTE 3—The use of direct current plasma atomic emission spectrometry (DCP-AES) is not within the scope of this test method.

7.1.2 *Flame Atomic Absorption Spectrometer (FAAS)*, equipped with an air-acetylene burner head, lead hollow cathode lamp or equivalent, or a discharge lamp without electrodes, and capable of making lead absorption measurements at the 283.3-nm and 217-nm absorption lines.

NOTE 4—The 283.3-nm line is preferred over the 217.0-nm line because of the increased noise levels commonly observed at 217.0 nm for FAAS and GFAAS.

7.1.3 *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 Test Method D3919).

NOTE 5—GFAAS is sometimes referred to as electrothermal atomic absorption spectrometry.

7.2 *Gases*, compressed in grades specified by the manufacturer of the instrument used.

7.2.1 Compressed air and acetylene for FAAS.

7.2.2 Compressed or liquid argon for ICP-AES and GFAAS.

7.2.3 Minimum of two-stage regulation of all compressed gases.

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 to 5 mL adjustable and 1000, 500, 250, 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. It must be from a different lot number (or manufacturer) than the calibration stock solution (see 8.3).

8.5 *Interferant Stock Solution* (for ICkS and ICP-AES only), 10 000 µg/mL of aluminum, calcium, iron, and magnesium in dilute nitric acid or equivalent.

9. Procedure

9.1 *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.

9.2 *Instrumental Setup*:

9.2.1 *FAAS/GFAAS*—Set the 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.

9.2.2 *ICP-AES*—Set up the spectrometer for the analysis of lead at a primary lead emission line (such as 220.2) in accordance with the instructions given by the manufacturer. Be sure to allow at least a 30-min warm-up of the system prior to starting the calibration and analysis.

9.3 *Preparation of Calibration and Instrumental QC Standards*:

9.3.1 *Calibration Standards*—Prepare a series of calibration standards (minimum of three) covering the linear range of the instrumentation. Prepare these standards using serial dilution from the calibration stock solutions and obtaining the same final nitric acid concentration present in the sample digestates or extracts. Also prepare an ICB (see Table 1).

NOTE 6—The ICP-AES analysis can be performed using one high-calibration standard and an ICB. However, more calibration standards are generally preferred.

9.3.2 *Instrumental QC Standards*—Prepare instrumental QC standards as summarized in Table 1 using serial dilution from the required stock solutions. Prepare these standards using the same final nitric acid concentration present in the sample digestates/extracts.

NOTE 7—The ICV is used to assess the accuracy of the calibration standards. It must therefore be made from a different original source of stock solutions than the stock used to make the calibration standards. Use of a different serial dilution of the same original stock solution is not acceptable.

9.4 *Calibration and Instrumental Measurement*—Perform the calibration and quantitative lead measurement of sample digestates or extracts and instrumental QC samples in the sequential order outlined in Table 2.

NOTE 8—It is generally recommended to perform a semiquantitative screen prior to quantitative analysis for sample digestates/extracts containing unknown levels of lead. The purpose of this screen is to determine the serial dilution requirements of each sample digestate/extract necessary to keep the instrumental response within the calibration curve. All digestates are diluted to a constant large value (1 to 100 for ICP-AES and FAAS and 1 to 1000 for GFAAS) during a semiquantitative screen. The instrument is calibrated, and diluted digestates/extracts are analyzed without inserting the instrumental QC used for a quantitative analysis run. Data from this screen are then reviewed to calculate the optimum serial dilution required for each digestate or extract sample solution. The