Designation: E1613 – 12

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 standard. No other units of measurement are included in this 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D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn 2002)[3]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

D7144 Practice for Collection of Surface Dust by Microvacuum Sampling for Subsequent Metals Determination

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] 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 (Withdrawn 2009)[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 (Withdrawn 2011)[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

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