Designation: E 1613 – 99

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 E 1613; 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 originally 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 can be provided because of differences among various makes and models of satisfactory instruments. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument.

1.5 The use of analytical instrumentation other than ICP-AES, FAAS, and GFAAS is not within the scope of this test method.

1.6 The values stated in SI units are to be regarded as the 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:*

D 1193 Specification for Reagent Water[2]

D 3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry[2]

D 4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data[2]

D 4697 Guide for Maintaining Test Methods in the User's Laboratory[3]

D 4840 Guide for Sampling Chain of Custody Procedures[2]

E 456 Terminology Relating to Quality and Statistics[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

E 1188 Practice for Collection and Preservation of Information and Physical Items by a Technical Investigator[4]

E 1553 Practice for Collection of Airborne Particulate Lead During Abatement and Construction Activities[5]

E 1605 Terminology Relating to Abatement of Hazards from Lead-Based Paint in Buildings and Related Structures[5]

E 1644 Practice for Hot Plate Digestion of Dust Wipe Samples for Subsequent Determination of Lead by Atomic Spectrometry[5]

E 1645 Practice for Preparation of Dried Paint Samples for Subsequent Lead Analysis by Atomic Spectrometry[5]

E 1726 Practice for Sample Digestion of Soils for the Determination of Lead by Atomic Spectrometry[5]

E 1727 Practice for Field Collection of Soil Samples for Lead Determination by Atomic Spectrometry Techniques[5]

E 1728 Practice for Field Collection of Settled Dust Samples Using Wipe Sampling Methods for Lead Determination by Atomic Spectrometry Techniques[5]

E 1729 Practice for Field Collection of Dried Paint Samples for Determination by Atomic Spectrometry Techniques[5]

E 1741 Practice for Preparation of Airborne Particulate Lead Samples Collected During Abatement and Construction Activities for Subsequent Analysis by Atomic Spectrometry[5]

[1] This test method is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.23 on Lead Hazards Associated with Buildings.

Current edition approved Oct. 10, 1999. Published December 1999. Originally published as E 1613 – 94. Last previous edition E 1613 – 94.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 07.02.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] *Annual Book of ASTM Standards*, Vol 04.11.

E 1775 Guide for Evaluating Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead[5]

E 1792 Specification for Wipe Sampling Materials for Lead in Surface Dust[5]

E 1864 Practice for Evaluating Quality Systems of Organizations Engaged in Conducting Facility and Hazard Assessments to Determine the Presence and Extent of Lead in Paint, Dust, Airborne Particulate, and Soil in Buildings and Related Structures[5]

E 1973 Practice for Collection of Surface Dust by Air Sampling Pump Vacuum Technique for Subsequent Lead Determination[5]

E 1979 Practice for Ultrasonic Extraction of Paint, Dust, Soil, or Air Samples for Subsequent Determination of Lead[5]

2.2 *Federal Documents:*[6]

40 CFR 261 Appendix II-Method 1311 Toxic Characteristic Leaching Procedure (TCLP)

40 CFR 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants

3. Terminology

3.1 Definitions—For definitions of related terms not appearing here, see Terminology E 1605.

3.1.1 *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). Recalibration of a given instrument produces a new analysis run.

3.1.2 *batch*—a group of field or QC samples that are processed together using the same reagents and equipment.

3.1.3 *calibration standards*—solutions of known analyte concentrations used to calibrate instruments.

3.1.3.1 *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.

3.1.4 *continuing calibration blank (CCB)*—a solution containing no analyte that is used to verify blank response and freedom from carryover.

3.1.4.1 *Discussion*—The CCB must be analyzed after the CCV (see 3.1.5) and after the ICkS (see 3.1.13). The measured value is to be (at most) less than five times the instrumental detection limit (IDL) (see 3.1.1).

3.1.5 *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.

3.1.5.1 *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.

3.1.6 *digestate*—an acidified aqueous solution that results from digestion of the sample.

3.1.7 *digestion*—a high temperature sample preparation process that solubilizes targeted analytes that may be present in the sample and results in an acidified aqueous solution called the digestate (see 3.1.6).

3.1.7.1 *Discussion*—Digestion normally entails the use of a hot plate or microwave oven for subjecting the acidified sample solution to high temperatures. Digestion is a type of extraction (see 3.1.8).

3.1.8 *extraction*—the dissolution of target analytes from a solid matrix into a liquid form, and results in a solution called the extract.

3.1.8.1 *Discussion*—Target analytes are extracted (solubilized) into a solution during sample extraction. Digestion (see 3.1.7) is an example of an extraction process. Apart from digestion, other examples of extraction processes include ultrasonic extraction **(1)**[7] and leaching (for example, 40 CFR 261 Appendix II Method 1311).

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

3.1.9.1 *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 3.1.11).

3.1.10 *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.

3.1.10.1 *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.

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

3.1.11.1 *Discussion*—The IDL is usually determined by the manufacturer for use in advertising and promotion. 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 3.1.15). Typical IDLs for FAAS, ICP-AES, and GFAAS are 0.05, 0.03, and 0.002 µg Pb/mL, respectively.

3.1.12 *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.

3.1.13 *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

[6] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

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

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.

3.1.13.1 *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.

3.1.14 *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 initially. (The same reagents and processing conditions that are applied to field samples within a batch are also applied to the method blank.)

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

3.1.15 *limit of detection (LOD)*—the MDL (see 3.1.16) or the IDL (see 3.1.11), depending on the context.

3.1.16 *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.

3.1.16.1 *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.

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

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

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

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

3.1.19 *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.

3.1.19.1 *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.

3.1.20 *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.

3.1.20.1 *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.

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

3.1.21.1 *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.

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

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

3.1.23 *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.

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

4. Summary of Test Method

4.1 A sample digestate or extract is analyzed for lead content using ICP-AES, FAAS, or GFAAS techniques **(2, 3, 4)**. Instrumental QC samples are analyzed along with sample digestates or extracts in order to ensure adequate instrumental performance.

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. 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)**.

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 2—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 electrodeless discharge lamp, and capable of making lead absorption measurements at the 283.3-nm absorption line.

NOTE 3—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 electrodeless discharge lamp, and capable of making lead absorption measurements at the 283.3-nm absorption line (see Test Method D 3919).

NOTE 4—GFASS 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 D 1193. (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. Be sure to allow at least a 30-min warmup of the system prior to starting the calibration and 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 warmup 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 5—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 6—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

TABLE 1 Instrumental QC Standards and Specifications

Name	Use	Specification
initial calibration blank (ICB)	initial calibration and zeroing instrument.	calibration standard containing no analyte must be measured before and after calibration measured value must be less than five times method detection limit (MDL)
calibration standards	instrument calibration high standard rerun used to check for carryover and instrumental drift	must be matrix matched to digestates/extracts must be measured prior to measuring any sample digestates or extracts correlation coefficient of ≥0.995, as measured using linear regression on instrument response versus concentration highest level calibration standard must be measured after calibration; measured value within ±10 % of known value
initial calibration verification (ICV)	verify calibration standard levels	analyte concentration near mid-range of calibration line made from stock solution from different lot or vendor than calibration standards must be measured after calibration and before measuring sample digestates/extracts measured value within ±10 % of known value
interference check standard (ICkS) (for ICP-AES only)	verify accurate analyte response in presence of possible spectral interference(s).	analyte concentration less than 25 % of highest calibration standard; interferant concentration 200 µg of Al, Ca, Fe, & Mg must analyze at least twice, once before and once after all sample digestates/extracts measured analyte value within ±20 % of known value
continuing calibration verification (CCV)	verify freedom from excessive instrumental drift	analyte concentration near mid-range of calibration line must be analyzed before and after all sample digestates/extracts, and at a frequency not less than once every ten samples measured value within ±10 % of known value (±20 % for GFAAS)
continuing calibration blank (CCB)	verify blank response and freedom from carryover	calibration standard containing no analyte must be analyzed after the CCV and after the ICkS (if applicable) measured value less than five times MDL

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 7—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 optimum dilution is one that achieves the maximum lead response that is still within the calibration curve. For ICP-AES, levels of possible interferants (aluminum, calcium, iron, and magnesium) may also have to be considered in order to make interference corrections. For ICP-AES, digestates or extracts must be diluted sufficiently to ensure that levels of possible interferants are at or below the levels present in the ICkS.

9.5 *Instrumental QC Evaluation and Corrective Action*—Examine the data generated from the analysis of calibration standards and instrumental QC standards. Evaluate the analysis run using the criteria given in Table 1. Failure to achieve the specifications given in Table 1 will require corrective action to be performed as described as follows:

9.5.1 *ICB, Calibration Standards, or ICV*—Failure to meet the specifications for these instrumental QC standards requires complete recalibration. Sample digestates or extracts cannot be

TABLE 2 Example Recommended Analysis Run Order

Run Order No. (Relative[A])	Sample Identification	Comments	
1	ICB	calibration blank	instrument calibration
2–4	low, med, high, standards	calibration standards	
5	ICB	calibration blank	calibration verification
6	ICV	made from different stock, level near midpoint of curve	
7	high standard	calibration standard	linearity check
8	CCB	same as calibration blank	
9	ICkS	interference check standard	interferant check for ICP-AES only
10	CCB	continuing calibration blank	carryover check
11	CCV	carryover check	drift check; same as near midpoint calibration standard
12	CCB	carryover check	
*** start repeating cycle of samples—instrumental QC here ***			
13–22	sample IDs	sample digestates/ extracts	maximum of 10 samples
23–24	CCV CCB	drift check + carryover check	see run Nos. 11–12
25–34	sample IDs	sample digestates/ extracts	maximum of 10 samples
35–36	ICkS CCB	interferant check + carryover check	see run Nos. 9–10
37–38	CCV CCB	drift check + carryover check	see run Nos. 11–12
*** end repeating cycle of samples—QC standards here ***			

[A] Depending on the analysis technique, more or fewer actual solutions may be required to perform the calibration and instrument QC requirements.

measured under these conditions. It is recommended that standards be prepared anew prior to re-calibration.

9.5.2 *High-Calibration Standard Rerun*—Failure to meet specifications for this instrumental QC standard requires complete re-calibration. Sample digestates/extracts cannot be measured under conditions where these specifications are not met. It is recommended that the standard range be reduced prior to recalibration.

9.5.3 *ICkS (for ICP-AES Analysis)*—Failure to meet the specifications for these instrumental QC standards requires reanalysis of the standard until the specifications are met. Sample digestates/extracts cannot be measured under conditions where these specifications are not met. Under these conditions, it is recommended that the standard be prepared anew. Continued failure of the ICkS may require interference correction investigation or changing instrument parameters. Consult the manufacturer's recommendations under these conditions. Any change in instrument parameters must be accompanied by recalibration. The interference levels in the ICkS can be lowered if measured aliquots of sample digestates/extracts can be shown not to contain interferants as high as those recommended for preparing the ICkS. Such changes must be documented in laboratory records with data supporting the justification for the change. All measurements on sample digestates or extracts must be bracketed by an ICkS that meets specifications of Table 1 (called a "passing" ICkS). Failure to meet the specifications on the ICkS run after the sample digestates/extracts requires reanalysis of all sample digestates since the last passing ICkS was measured. Since only the ICkS is required to be analyzed twice, much data could be lost if the analytical run were long and the second ICkS failed specifications. This is good reason for including periodic analysis of the ICkS as indicated in Table 2.

9.5.4 *CCV*—Failure to meet the specifications for these instrumental QC standards indicates excessive instrumental drift. Sample digestates cannot be measured under these conditions, and any sample digestates/extracts cannot be measured under conditions of excessive instrumental drift, and any sample digestates or extracts measured since the last passing CCV must be reanalyzed. This situation requires either reanalysis of the standard until specifications are met or recalibration. All measurements on sample digests or extracts must be bracketed by a CCV that meets specifications.

9.5.5 *CCB*—Failure to meet the specifications for these instrumental QC standards suggests the presence of possible instrumental carryover or baseline shift. Such a failure will have the most impact on sample digestates or extracts having lead concentrations in the low range of the calibration line at the lower end of the calibration curve. The first corrective action is to reanalyze the CCB. The rinse time between the samples should be increased and the analysis run continued if the CCB passes. If the instrument response remains elevated and has not changed significantly, the instrument can be re-zeroed. This shall be followed by a CCV-CCB and reanalysis of all samples since the last passing CCB that are within five times the response of the failed CCB.

10. Calculation

10.1 *Determination of Method Detection Limit*—The MDL shall be determined at least annually. There are many ways to determine an MDL. Each involves the use of sampling media digestates/extracts at low analyte concentration (see Note 8). The two methods discussed below are in common use.

NOTE 8—Liquid standard spiking of clean matrix material is allowed for the determination of an MDL.

10.1.1 To determine an MDL based on the method found in 40 CFR Part 136, extract/digest a minimum of seven spikes with concentration of no more than five times the expected MDL (this necessitates making an educated guess as to the MDL) and determine the standard deviation of the results. The MDL is the standard deviation multiplied by 3.143, a factor from the Tables of Student "t" Values at the 99 % confidence limit:

$$\text{MDL} = 3.143\,S \tag{1}$$

10.1.2 Another method that may be used to determine an MDL but does not require an estimate of the (actual) MDL can be found in several references and texts **(8)**. The process involves analysis of the digestates or extracts from at least seven examples of the blank matrix. The standard deviation of the results is calculated and entered into a relationship that considers the degrees of freedom of the process:

$$\text{MDL} = t\,S\,[(N_i + N_b)/(N_i N_b)]^{0.5} \tag{2}$$

where:

MDL = the method detection limit,
t = the Student T statistic for $n=7$ ($t = 3.143$),
S = the standard deviation of the analyte concentration found in the blank media digestates/extracts,
N_i = the number of times an unknown sample is to be analyzed (usually one), and
N_b = the number of blank media digestates/extracts analyzed, which is greater than or equal to seven.

10.1.2.1 When $N_i = 1$ and $N_b = 7$, Eq 2 is simplified to:

$$\text{MDL} = 3.360\,S \tag{3}$$

10.2 *FAAS/GFAAS*—Prepare a calibration curve to convert the instrument response (absorbance) to concentration of lead (µg/mL) using a linear regression fit. Convert all instrumental measurement on instrumental QC standards and sample digests or extracts to lead concentration (µg/mL) using the calibration line.

NOTE 9—Some instruments will automatically prepare a calibration curve based on a linear regression fit. All modern ICP-AES instruments automatically prepare a calibration curve to convert instrument response (emission intensity) to concentration (µg/g), so 10.2 is unnecessary for ICP-AES analysis.

10.3 *Calculation of Lead Concentration in Sample Digestate/Extract*—Calculate the lead concentration in the sample digest or extract after instrumental analysis as follows:

$$\text{measured lead in sample solution, µg/mL} = (A_i)(D) \tag{4}$$

where:

A_i = instrumentally measured lead concentration, µg/mL, and

D = dilution factor, mL/mL, required during instrumental analysis to produce a measured lead level within the calibration curve.

10.4 *Calculation of Lead Concentration in Original Samples*—Calculation of the lead levels in the originally digested or extracted samples is dependent on the sample matrix (dust, soil, air filter or paint) and sample preparation procedure. The following are calculations for each of these matrices:

NOTE 10—For sample digestates or extracts generating lead measurements falling below the quantitation limit, the quantitation limit value should be used for performance calculations. A less than sign (<) should be used on lead analysis results from such calculations to indicate the uncertainty of these values.

10.4.1 *Dust Wipes*:

NOTE 11—Dust wipe samples should be collected in accordance with Practice E 1728, using wipes meeting the specifications of Specification E 1792. Wipes should be prepared for subsequent analysis following Practice E 1644 or E 1979.

$$\text{lead concentration, } \mu g/cm^2 = [(A)(B)]/(C) \quad (5)$$

where:
A = measured lead concentration in sample digestate or extract from 10.3,
B = final dilution volume, mL, and
C = collection area, cm^2.

10.4.1.1 *Lead Mass Per Sample*:

$$\text{lead content, } \mu g/\text{wipe} = [(A)(B)] \quad (6)$$

where:
A = measured lead concentration in sample digestate or extract from 10.3, and
B = final dilution volume, mL.

10.4.2 *Lead in Soil*:

NOTE 12—Soil samples should be collected following Practice E 1727, and they should be prepared for subsequent analysis in accordance with Practice E 1726 or E 1979.

$$\text{lead concentration, } \mu g/g = [(A)(B)]/(C) \quad (7)$$

where:
A = measured lead concentration in sample digestate or extract, from 10.3,
B = final dilution volume, mL, and
C = sample weight, g.

10.4.3 *Lead in Paint*:

NOTE 13—Paint samples should be collected following Practice E 1729, and they should be prepared for subsequent analysis using Practice E 1645 or E 1979.

10.4.3.1 *Lead Mass Per Unit Sample Area*:

$$\text{lead concentration, } mg/cm^2 = [(A)(B)]/[(C)(1000)] \quad (8)$$

where:
A = measured lead concentration in sample digestate or extract from 10.3,
B = final dilution volume, mL, and
C = collection area, cm^2.

10.4.3.2 *Lead Mass Per Unit Mass of Sample*:

$$\text{lead content, } \mu g/g = [(A)(B)]/[(C)(1000)] \quad (9)$$

where:
A = measured lead content in sample digestate or extract from 10.3,
B = final dilution volume, mL, and
C = sample weight, g.

10.4.4 *For Lead in Airborne Particulate*:

NOTE 14—Air particulate samples should be collected in accordance with Practice E 1553 and prepared for subsequent analysis by following Practice E 1741 or E 1979.

10.4.4.1 *Mass of Lead Per Unit Volume of Sampled Air*:

$$\text{lead concentration, } \mu g/m^3 = [(A)(B)] / (C) \quad (10)$$

where:
A = measured lead concenntration in sample digestate or extract from 10.3,
B = final dilution volume, mL, and
C = collection volume, m^3.

10.4.4.2 *Lead Mass Per Sample*:

$$\text{lead concentration, } \mu g/\text{filter} = [(A)(B)] \quad (11)$$

where:
A = measured lead content in sample digestate or extract from 10.3, and
B = final dilution volume, mL.

10.4.5 *Lead in Dust Vacuum Samples*

NOTE 15—Dust vacuum samples should be collected using Practice E 1973, and they should be prepared for subsequent analysis using Practice E 1741 or E 1979.

10.4.5.1 *Lead Mass Per Sample*:

$$\text{lead concentration, } \mu g/\text{filter} = [(A)(B)] \quad (12)$$

where:
A = measured lead content in sample digestate or extract from 10.3, and
B = final dilution volume, mL.

10.4.5.2 *Dust Wipes*:

$$\text{lead concentration, } \mu g/cm^2 = [(A)(B)]/(C) \quad (13)$$

where:
A = measured lead concentration in sample digestate or extract from 10.3,
B = final dilution volume, mL, and
C = collection area, cm^2.

10.5 Calculation of the lead levels in the originally digested QC samples is dependent on the sample matrix (dust, soil, or paint) and sample preparation procedure. The previous calculations in 10.3are examples of each of these matrices.

11. Quality Assurance (QA)

11.1 *Analysis Procedures*—The performance of the analysis procedures on QC samples shall meet the specifications listed in Tables 1 and 2.

NOTE 16—Performance criteria for lead analyses of environmental samples have been recommended **(11)**. Performance criteria for measurement accuracy, precision, sample size, and working range for lead analyses have been delineated in Practice E 1775. Although this Test Method does not deal specifically with sample preparation aspects of the overall analysis, the performance of the overall analytical method should

meet the minimum performance criteria stated in Practice E 1775.

11.2 *QA System*—The QA system shall meet the requirements of Practice E 1864. Follow QA/QC procedures delineated in Practices D 4210 and E 1188 and Guides D 4697 and D 4840.

12. Report

12.1 Data to report include sample receipt information, all final field sample analysis results, and instrument QC data. (QC analysis data to report shall include results for method blanks, spike and spike duplicate recoveries, and range of duplicate percent recoveries.)

13. Precision and Bias

13.1 The precision and bias for this analysis method are dependent on both the choice of analytical instrumentation (FAAS, ICP-AES, or GFAAS) and the sample digestion or extraction procedures used for preparing the samples.

13.2 *Precision*—Practice E 691 was used to estimate method precision as applied to a subset of data from the Environmental Lead Proficiency Analytical Testing (ELPAT) Program **(11)** for which repeatability data were available **(12)**. Practice E 691 specifies a minimum of six laboratories, four materials, and two determinations for each of the four materials. The precision estimate was made for certified reference materials (CRMs), which were dust wipes containing a certified concentration of lead. Sample preparation and methods that were used were equivalent to Practice E 1644 (hot plate digestion) and Test Method E 1613 (FAAS or ICP-AES). The CRMs consisted of dust wipes having different lead loadings.

13.2.1 *Hot Plate Digestion and FAAS or ICP-AES Analysis*—Four materials, two determinations each, were digested and analyzed by 38 and 23 laboratories, respectively. Precision, characterized by repeatability, S_r, r, and reproducibility, S_R, R, were determined, and the results are shown in Table 3.

NOTE 17—The definitions of S_r, r, S_R, R are given in Terminology E 456. S_r and S_R are standard deviations for repeatability and reproducibility, respectively, whereas $_r$ and $_R$ are the repeatability and reproducibility 95 % confidence limits (coefficients of variation).

13.2.2 *Other Procedures and Sample Matrices*—Data for other sample matrices (such as paint, soil, and air filters) and analysis methods (that is, GFAAS) have been published **(12, 13,14)**, but repeatability data are not yet available for those cases. Interlaboratory testing data showing results for reproducibility are available from ELPAT **(11)**. For air filter samples, precision data for reproducibility based on equivalent procedures are published **(2)**, but again, repeatability data are not available.

TABLE 3 Precision Data

(A) FAAS Analysis (38 laboratories):

Material	Average (µg Pb)	S_r	S_R	r	R
A	478.09	34.19	51.77	95.73	144.97
B	1244.29	75.45	122.77	211.26	343.77
C	454.84	10.02	22.31	28.04	64.64
D	126.45	16.09	17.14	45.04	48.00

(B) ICP-AES Analysis (23 laboratories):

Material	Average (µg Pb)	S_r	S_R	r	R
A	478.13	36.78	44.61	102.97	124.90
B	1198.67	38.94	128.84	109.02	360.75
C	398.00	10.43	43.11	29.21	120.72
D	122.18	6.24	10.89	17.47	30.49

13.3 *Bias*—For lead determinations, bias depends on both the sample preparation procedure and analysis procedures used. Biases for analytical methods used in procedures that are equivalent to ASTM sample preparation and analysis procedures are typically less than ±5 % **(12, 13,14)**; the principal contributor to overall method bias ordinarily arises from the sample preparation procedure. Hot plate digestion of environmental CRMs by procedures that are equivalent to Practices E 1644, E 1645, E 1726, and E 1741 can yield recoveries that are significantly less that 100 % **(12)**. For example, some soil CRMs have been found to give lead recoveries of 80 to 85 %, and fly ash CRMs may yield recoveries of lead in the vicinity of only 50 %. This is due to the possible presence of silicates, which are insoluble when using the reference sample preparation procedures; the use of hydrofluoric acid (HF) is needed to achieve 100 % recovery from such samples. However, the use of HF is generally discouraged for safety reasons. During spectrometric analysis, matrix matching is crucial to minimizing bias.

14. Keywords

14.1 FAAS; GFAAS; ICP-AES; instrumental analysis; lead

REFERENCES

(**1**) Ashley, K., *Trends in Analytical Chemistry*, 17, 1998, p. 366.

(**2**) Eller, P. M., Cassinelli, M.E., Eds., *NIOSH Manual of Analytical Methods*, 4th ed., Methods 7082, 7105, and 7300, National Institute for Occupational Safety and Health, Cincinnati, OH, 1994.

(**3**) Environmental Protection Agency, *Standard Operating Procedures for Lead in Paint by Hotplate- or Microwave-Based Acid Digestions and Atomic Absorption or Inductively Coupled Plasma Spectrometry*, U.S. EPA, Research Triangle Park, NC, 1991. EPA Contract No. 68-02-4550.

(**4**) U.S. EPA, *Standard Operating Procedure for the Laboratory Analysis of Lead in Paint, Bulk Dust, and Soil by Ultrasonic Acid Digestion and ICP-AES Measurement* (EPA 600/R-95/111), U.S. Environmental Protection Agency: Research Triangle Park, NC, 1997.

(**5**) Larson, G. F., Fassel, V. A., Scott, R. H., Kniseley, R. N., *Analytical Chemistry, 47*, 1975, p. 238.

(**6**) Boumans, P. W. J. M., *Spectrochimica Acta, Part B*, 31, 1976, p. 90.

(**7**) Slavin, W., *Atomic Absorption Spectroscopy*, 2nd ed., Wiley Interscience, New York, 1978.

(**8**) Zander, A. T., *American Laboratory, 8*(11), 1976, p. 11.

(**9**) Skoog, D., West, D. M., Holler, F.J., *Fundamentals of Analytical Chemistry*, 5th ed., Saunders: Philadelphia, 1990.

(**10**) Smith, S. B., and Hieftje, G. M., *Spectroscopy*, Vol 37, 1983.

(**11**) U.S. EPA, *Laboratory Accreditation Guidelines: Measurement of Lead in Paint, Dust and Soil*, U.S. Environmental Protection Agency: Washington, DC, 1992.

(**12**) Schlecht, P. C., Groff, J. H., Feng, A., Song, R., *American Industrial*

Hygiene Association Journal, 57, 1996, p. 1035.
(**13**) Millson, M., Eller, P. M., and Ashley, K., *American Industrial Hygiene Association Journal*, 55, 1994, p. 339.
(**14**) Ashley, K., Schlecht, P. C., Song, R., Feng, A., Dewalt, G., and McKnight, M. E., *Sampling Environmental Media*, Morgan, J. H., Ed., *ASTM STP 1282*, ASTM, 1996, p. 125.