



Standard Guide for Evaluating Performance of On-Site Extraction and Field- Portable Electrochemical or Spectrophotometric Analysis for Lead¹

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1. Scope

1.1 This standard provides guidelines for determining the performance of field-portable quantitative lead analysis instruments.

1.2 This guide applies to field-portable electroanalytical and spectrophotometric (including reflectance and colorimetric) analyzers.

1.3 Sample matrices of concern herein include paint, dust, soil, and airborne particulate.

1.4 This guide addresses the desired performance characteristics of field-based sample extraction procedures for lead, as well as on-site extraction followed by field-portable analysis.

1.5 *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 5438 Practice for Collection of Floor Dust for Chemical Analysis²

E 1553 Practice for Collection of Airborne Particulate Lead During Abatement and Construction Activities³

E 1605 Terminology Relating to Abatement of Hazards from Lead-Based Paint in Buildings and Related Structures³

E 1613 Test Method for Analysis of Digested Samples for Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption (FAAS), or Graphite Furnace Atomic Absorption (GFAAS) Techniques³

E 1644 Practice for Hot Plate Digestion of Dust Wipe Samples for Determination of Lead by Atomic Spectrometry³

E 1645 Practice for Preparation of Dried Paint Samples for Subsequent Lead Analysis by Atomic Spectrometry³

E 1726 Practice for Sample Digestion of Soils for Determination of Lead by Atomic Spectrometry³

E 1727 Practice for Field Collection of Soil Samples for Lead Determination by Atomic Spectrometry Techniques³

E 1728 Practice for Field Collection of Settled Dust Samples Using Wipe Sampling Methods for Lead Determination by Atomic Spectrometry Techniques³

E 1729 Practice for Field Collection of Dried Paint Samples for Lead Determination by Atomic Spectrometry Techniques³

E 1741 Practice for Preparation of Airborne Particulate Lead Samples Collected During Abatement and Construction Activities for Subsequent Analysis by Atomic Spectrometry³

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 Soil³

2.2 U.S. EPA Documents:

EPA 600/R-93/200 Standard Operating Procedure for the Field Analysis of Lead in Paint, Bulk Dust, and Soil by Ultrasonic, Acid Digestion and Colorimetric Measurement (1993)⁴

EPA 747-R-92-001 Laboratory Accreditation Guidelines: Measurement of Lead in Paint, Dust, and Soil (1992)⁵

3. Terminology

3.1 For definitions of terms not listed here, see Terminology E 1605.

3.2 *anodic stripping voltammetry*—an electroanalytical technique in which the concentration of analyte metal species dissolved in solution is determined in the following manner. The analyte is first deposited (preconcentrated) electrochemically by reducing the dissolved ion in solution to immobilized metal species at a mercury electrode surface. The metal is

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² *Annual Book of ASTM Standards*, Vol 11.03.

³ *Annual Book of ASTM Standards*, Vol 04.11.

⁴ Available from U.S. EPA, Office of Research and Development, Research Triangle Park, NC.

⁵ Available from U.S. EPA, Office of Pollution Prevention and Toxic Substances, Washington, DC.

deposited in the form of an amalgam (with Hg) at an applied potential (voltage) which is negative of the standard oxidation potential for the metal/ion redox couple. After deposition, the preconcentrated metal species is then “stripped” from the mercury electrode by applying a positive potential sweep, which causes anodic oxidation of the analyte metal species to dissolved ion. The current associated with this reoxidation is measured. The peak current is proportional to the original concentration of dissolved analyte species over a wide range of concentrations.

3.3 *colorimetry*—an analytical technique that is similar to spectrophotometry (see 3.5), except that ultraviolet-visible light of a single, narrow wavelength range is passed through a sample cell containing dissolved analyte, and the absorption measured.

3.4 *reflectance*—a measurement technique (subset of spectrophotometry; see 3.5) in which light is reflected off of a reflecting surface and measured by a detector. The amount of reflected light may be a function of analyte concentration.

3.5 *spectrophotometry*—an analytical technique in which a spectrum of analyte species is obtained and used to determine the analyte concentration in the following manner. Light is directed onto or through analyte species, and the absorption of this light across a range of wavelengths is measured by a detector. The amount of absorbed light is a function of the concentration of analyte species.

4. Significance and Use

4.1 This guide is intended for use in evaluating the performance of field-portable electroanalytical or spectrophotometric devices for lead determination, or both.

4.2 Desired performance criteria for field-based extraction procedures are provided.

4.3 Performance parameters of concern may be determined using protocols that are referenced in this guide.

4.4 Reference materials to be used in assessing the performance of field-portable lead analyzers are listed.

4.5 Exhaustive details regarding quality assurance issues are outside the scope of this guide. Applicable quality assurance aspects are dealt with extensively in references that are cited in this guide.

5. Performance Evaluation Materials

5.1 *Certified Reference Materials (CRMs)*—These may consist of NIST Standard Reference Materials (SRMs) and are also known as Primary and Secondary Reference Materials:

5.1.1 *Paint*—Examples are NIST paint SRMs, for example, NIST SRM 2582, and new SRMs currently under development and numerous other CRMs.

5.1.2 *Dust*—Examples are NIST 1648 (urban particulate matter), other NIST dust SRMs, and new materials under development.

5.1.3 *Soil*—Examples are NIST 2704 (river sediment) and NIST soil standards: SRMs 2709, 2710, and 2711 and numerous other CRMs.

5.1.4 *Airborne Particulate*—No NIST SRMs are available for airborne particulate collected on filters. In lieu of this, use NIST urban particulate SRM 1648 (see 5.1.2) or one of numerous other CRMs.

5.2 Real-World Materials:

5.2.1 *Paint*, collected using Practice E 1729 (or equivalent). To obtain reference values, determine lead concentration using Test Method E 1613 and Practice E 1645 (or equivalent).

5.2.2 *Dust Wipes*, collected using Practice E 1728. To obtain reference values, determine lead concentration using Test Method E 1613 and Practice E 1644 (or equivalent).

5.2.3 *Vacuumed Dust*, collected using Practice D 5438. To obtain reference values, determine lead concentration using Test Method E 1613 and Practice E 1726 (or equivalent).

5.2.4 *Soil*, collected using Practice E 1727. To obtain reference values, determine lead concentration using Test Method E 1613 and Practice E 1726 (or equivalent).

5.2.5 *Airborne Particulate*, collected using Practice E 1553 (or equivalent). To obtain reference values, determine lead concentration using Test Method E 1613 and Practice E 1741 (or equivalent).

5.3 *Secondary Reference Materials*—Examples of these CRMs include samples from the Environmental Lead Proficiency Analytical Testing (ELPAT) program (paints, dusts spiked on wipes, and soils) and the Proficiency Analytical Testing (PAT) program (air filters).⁶ Other examples include secondary reference materials (for example, bag house dust, sludge, and solid waste).⁷

5.3.1 *Paint*—Examples include ELPAT paint samples.

5.3.2 *Dust*—Examples include ELPAT wipe samples spiked with lead-containing dusts.

5.3.3 *Soil*—Examples include ELPAT soil samples.

5.3.4 *Air Filters*—Examples include PAT air filter samples.

6. Performance Criteria

6.1 *Extraction Procedures*—The extraction procedure chosen shall have a demonstrated recovery of at least 80 % for the matrix of concern, and shall be compatible with the lead analysis technique used (EPA 600/R-93/200). (Reference extraction and analytical techniques include those ASTM standards listed in 2.1.)

6.2 Field-Portable Analysis:

6.2.1 *Accuracy*—Overall measurement accuracy of the field-portable analytical technique following field-based extraction: within 25 % of the values obtained by the applicable ASTM extraction procedure (see 2.1) and Test Method E 1613.

6.2.2 *Precision*—For field-based extraction followed by field-portable analysis, total uncertainties for standard reference materials: 25 % relative standard deviation (RSD) (EPA 747-R-92-001); for real-world materials: 25 % RSD (EPA 747-R-92-001); for secondary reference materials: 25 % RSD.

6.2.3 *Working Range*—Minimum working concentration range for the overall method shall extend from 0.1 times the applicable action level to 2 times the applicable action level (NIOSH SOP 018)⁸ for the sample matrix of concern.

6.3 *Sample Size*—Follow applicable ASTM sample collection and sample preparation practices listed in 2.1.

⁶ Available from the American Industrial Hygiene Association, Fairfax, VA.

⁷ Available from Fisher Scientific and Resource Technology Corp., Laramie, WY.

⁸ National Institute for Occupational Safety and Health (NIOSH), “Guidelines for Air Sampling and Analysis,” available from NIOSH Publications, 4676 Columbia Parkway, Cincinnati, OH 45226, www.cdc.gov/niosh.

7. Quality Assurance (QA) and Quality Control (QC)

7.1 *Field and Laboratory QA/QC*—Follow QA/QC procedures delineated in the applicable ASTM sample preparation practices for the pertinent sample matrix (2.1) and in Test Method E 1613.

7.2 *QA System*—Follow the requirements delineated in Practice E 1864.

NOTE 1—Participation in proficiency testing programs, for example, ELPAT and other similar round robin schemes, is recommended.

8. Keywords

8.1 electroanalysis; extraction; lead; portable analysis; spectrophotometry

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