



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 particles.

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:²

D 5438 Practice for Collection of Floor Dust for Chemical Analysis

D 6785 Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry

D 6966 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals

D 7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D 7144 Practice for Collection of Surface Dust by Micro-

vacuum Sampling for Subsequent Metals Determination

E 1605 Terminology Relating to Lead in Buildings

E 1613 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

E 1644 Practice for Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead

E 1645 Practice for Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis

E 1726 Practice for Preparation of Soil Samples by Hotplate Digestion for Subsequent Lead Analysis

E 1727 Practice for Field Collection of Soil Samples for Subsequent Lead Determination

E 1729 Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination

E 1792 Specification for Wipe Sampling Materials for Lead in Surface Dust

E 1864 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

2.2 U.S. EPA Documents:³

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)

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

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:

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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 Available from United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

The analyte is first deposited (preconcentrated) electrochemically by reducing the dissolved ion in solution to immobilized metal species at an electrode surface (such as mercury or bismuth). The metal is deposited 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 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 Example 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 SRMs 2582, 2583, and numerous other CRMs.

5.1.2 Dust—Examples are NIST 1648 (urban particulate matter), other NIST dust SRMs, and other CRMs.

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 Matter—An example is NIST SRM 2783, which is available for airborne particulate matter collected on filters. Other CRMs are also available.

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 D 6966, with wipes meeting the requirements of Specification E 1792. 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 Practices D 7144 or 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 Matter, collected using Test Method D 6785 (or equivalent). To obtain reference values, determine lead concentration using Test Method E 1613 and either Test Methods D 6785 or D 7035 (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).⁴ Other examples include secondary reference materials (for example, bag house dust, sludge, and solid waste).⁵

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

⁴ Available from the American Industrial Hygiene Association (AIHA), 2700 Prosperity Ave., Suite 250, Fairfax, VA 22031

⁵ Available from Fisher Scientific and Resource Technology Corp., Laramie, WY.

applicable action level to 2 times the applicable action level (NIOSH SOP 018)⁶ for the sample matrix of concern.

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

NOTE 1—Performance data from interlaboratory testing of a portable anodic stripping voltammetric procedure have been published⁷

⁶ 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 Ashley, K., Song, R., Esche, C.A., Schlecht, P. C., Baron, P.A., and Wise, T.J., “Ultrasonic extraction and portable anodic stripping volumetric measurement of lead in pain, dust wipes, soil, and air—An interlaboratory evaluation. *Journal of Environmental Monitoring*, Vol. 1, pp. 459–464 (1999).

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 2—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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