



# Standard Guide for Evaluating Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead<sup>1</sup>

This standard is issued under the fixed designation E1775; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This guide 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:<sup>2</sup>

**D5438** Practice for Collection of Floor Dust for Chemical Analysis

**D6785** Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry

**D6966** Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals

**D7035** Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively

Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

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

**D7439** Test Method for Determination of Elements in Airborne Particulate Matter by Inductively Coupled Plasma–Mass Spectrometry

**E1605** Terminology Relating to Lead in Buildings

**E1613** 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

**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

**E1729** Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination

**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)<sup>3</sup>

### 2.2 U.S. EPA Documents:<sup>4</sup>

**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)

<sup>1</sup> This guide 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.

Current edition approved March 1, 2016. Published April 2016. Originally approved in 1996. Last previous edition approved in 2016 as E1775 – 07 (2016). DOI: 10.1520/E1775-16.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>4</sup> Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

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

2.3 ISO Document.<sup>5</sup>

ISO Guide 30 Reference materials -- Selected terms and definitions

### 3. Terminology

3.1 For definitions of terms not listed here, see Terminology E1605.

3.2 *anodic stripping voltammetry*—an electroanalytical technique in which the concentration of analyte metal species dissolved in solution is determined.

3.2.1 *Discussion*—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 in which 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 in which light reflected off of a surface containing the analyte of interest is measured by a detector.

3.5 *spectrophotometry*—an analytical technique in which a spectrum of a sample containing the analyte of interest is obtained and used to determine the analyte concentration.

3.5.1 *Discussion*—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)*—Reference material accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realization of the unit in which the property values are expressed: each certified value is accomplished by an uncertainty at a stated level of confidence (ISO Guide 30).

NOTE 1—These may consist of NIST Standard Reference Materials (SRMs) and are also known as Primary and Secondary Reference Materials:

(1) *Paint*—Examples are NIST paint SRMs, for example, NIST SRMs 2582, 2583, and numerous other CRMs.

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

(3) *Soil*—Examples are NIST 8704 (river sediment) and NIST soil standards: SRMs 2709a, 2710a, and 2711a and numerous other CRMs.

(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 E1729 (or equivalent). To obtain reference values, determine lead concentration using Test Methods D7439 and E1613 and Practice E1645 (or equivalent).

5.2.2 *Dust Wipes*, collected using Practice D6966, with wipes meeting the requirements of Specification E1792. To obtain reference values, determine lead concentration using Test Methods D7439 and E1613 and Practice E1644 (or equivalent).

5.2.3 *Vacuumed Dust*, collected using Practices D7144 or D5438. To obtain reference values, determine lead concentration using Test Methods D7439 and E1613 and Practice E1726 (or equivalent).

5.2.4 *Soil*, collected using Practice E1727. To obtain reference values, determine lead concentration using Test Methods D7439 and E1613 and Practice E1726 (or equivalent).

5.2.5 *Airborne Particulate Matter*, collected using Test Method D6785 (or equivalent). To obtain reference values, determine lead concentration using Test Methods D7439 and E1613 and either Test Methods D6785 or D7035 (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).<sup>6</sup> Other examples include

<sup>5</sup> Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

<sup>6</sup> Available from American Industrial Hygiene Association Proficiency Analytical Testing Programs, LLC (AIHA-PATP), 3141 Fairview Park Drive, Suite 777, Falls Church, VA 22042, <http://www.aiha.org>.

secondary reference materials (for example, bag house dust, sludge, and solid waste).<sup>7</sup>

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 E1613.

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)<sup>8</sup> for the sample matrix of concern.

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

NOTE 2—Performance data from interlaboratory testing of a portable anodic stripping voltammetric procedure have been published.<sup>9</sup>

## 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 E1613.

7.2 *QA System*—Follow the requirements delineated in Practice E1864.

NOTE 3—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

<sup>7</sup> Available from Fisher Scientific and Resource Technology Corp., Laramie, WY.

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<sup>8</sup> National Institute for Occupational Safety and Health (NIOSH), “Guidelines for Air Sampling and Analysis,” available from NIOSH Publications, 1150 Tusculum Ave., Cincinnati, OH 45226, Mail Stop C14, <http://www.cdc.gov/niosh>.

<sup>9</sup> 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).