Designation: E 2051 – 01

Standard Practice for the Determination of Lead in Paint, Settled Dust, Soil and Air Particulate by Field-Portable Electroanalysis[1]

This standard is issued under the fixed designation E 2051; 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 practice covers the analysis of extracts of environmental samples for lead content using field-portable electroanalytical devices.

1.2 Matrices of concern in this practice include paint, settled dust, soil, and air particulate.

1.3 *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 5438 Practice for Collection of Floor Dust for Chemical Analysis[3]

E 1553 Practice for Collection of Airborne Particulate Lead During Abatement and Construction Activities[3]

E 1605 Terminology Relating to Lead in Buildings[4]

E 1613 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[4]

E 1644 Practice for Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead[4]

E 1645 Practice for Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis[4]

E 1726 Practice for Preparation of Soil Samples by Hotplate Digestion for Subsequent Lead Analysis[4]

E 1727 Practice for Field Collection of Soil Samples for Lead Determination by Atomic Spectrometry Techniques[4]

E 1728 Practice for Field Collection of Settled Dust Samples Using Wipe Sampling Methods for Lead Determination by Atomic Spectrometry Techniques[4]

E 1729 Practice for Field Collection of Dried Paint Samples for Lead Determination by Atomic Spectrometry Techniques[4]

E 1741 Practice for Preparation of Airborne Particulate Lead Samples Collected During Abatement and Construction Activities for Subsequent Analysis by Atomic Spectrometry[4]

E 1775 Guide for Evaluating the Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead[4]

E 1973 Practice for Collection of Surface Dust by Air Sampling Pump Vacuum Technique for Subsequent Lead Determination[4]

2.2 *Federal Standard:*

Title 40, CFR Part 261, Appendix II—Method 1311, Toxic Characteristic Leaching Procedure (TCLP)[5]

3. Terminology

3.1 *Definitions*—For definitions of terms relating to this practice that do not appear in this section, refer to Terminology E 1605.

3.1.1 *anodic stripping voltammetry (ASV)*—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 metal ion in solution to immobilized discharged metal species at a mercury electrode surface, for instance, Pb^{2+} (solution) → Pb° (electrode). The metal is deposited in the form of an amalgam (with Hg) at an applied potential (voltage) that is negative of the standard oxidation potential of the metal/ion redox couple. After deposition for a given time period, the preconcentrated metal species is then "stripped" from the mercury electrode surface by applying a positive potential sweep, which causes anodic oxidation of the analyte metal species to dissolved ion; for example, Pb° (Hg) → Pb^{2+}(soln). During the stripping step, the current associated with this reoxidation is measured. The peak current arising from the reoxidation of discharged,

[1] This practice is under the jurisdiction of ASTM Committee E06 on Performance of Buildings and is the direct responsibility of Subcommittee E06.23 on Lead Paint Abatement.

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

[3] Annual Book of ASTM Standards, Vol 11.03.

[4] Annual Book of ASTM Standards, Vol 04.11.

[5] Available from U.S. Government Printing Office, Washington D.C.

amalgamated metal species is proportional to the original concentration of dissolved analyte species over a wide range of concentrations.

3.1.2 *disposable electrode*—In anodic stripping voltammetry and potentiometric stripping analysis, this is a solid-state electrode probe that is used once for electroanalysis and is then discarded.

3.1.2.1 *Discussion*—Since solid state electrode response may change significantly after one analysis, results of additional analyses with this electrode may not be reliable and cannot be used.

3.1.3 *field-portable*—For the purpose of this practice, this term refers to an electroanalytical device that is battery-powered and may be carried by hand into the field for use.

3.1.4 *on-site*—For the purposes of this practice, this term refers to activities that are carried out at the location where samples are taken.

3.1.5 *oxygen scavenger*—a chemical that is purposely dissolved in solution in order to reduce dissolved oxygen.

3.1.5.1 *Discussion*—Dissolved O_2 may interfere with the electroanalytical determination of lead, so its presence in sample solution is minimized by the addition of an oxygen scavenger such as L-ascorbic acid.

3.1.6 *potentiometric stripping analysis (PSA)*—an electroanalytical technique which is similar to anodic stripping voltammetry (ASV—see 3.1.1), but differs from ASV in the method used for stripping the amalgamated metals (**1**).[6] In the case of potentiometric stripping analysis (PSA), the system is open-circuited following the deposition (preconcentration) step; that is, potential (voltage) control of the system is lifted. The concentrated metals are then reoxidized by an oxidizing agent (such as O_2 or Hg(II)) which is present in solution; for example, Pb° (electrode) + dissolved oxidant → Pb^{2+}(solution). The potential due to this reoxidation is monitored as a function of time. The time required for the oxidation of a given analyte metal is proportional to the concentration of deposited metal, which in turn is proportional to the original analyte concentration.

3.1.7 *renewable electrode*—In anodic stripping voltammetry and potentiometric stripping analysis, this is a glassy carbon (or other material) electrode upon which a mercury (or other material) film is deposited and used for electrochemical analysis.

3.1.7.1 *Discussion*—After proper conditioning (as demonstrated by acceptable quality control analysis), many successive analyses may be conducted before the Hg film must be removed and then redeposited onto the glassy carbon electrode (as demonstrated by acceptable quality control analysis).[7]

3.1.8 *supporting electrolyte*—an inert salt that is dissolved in sample solution in order to maximize the conductivity of the solution for electrochemical analysis.

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

[7] For specific information on conditioning of electrodes and on Hg film cleaning and redeposition, refer to manufacturers literature or: Wang, J.," Electrochemical Preconcentration," in *Laboratory Techniques in Electroanalytical Chemistry*, 2nd ed.; Kissinger, P.T., and Heineman, W.R., Eds., Marcel Dekker: New York, 1996.

4. Summary of Practice

4.1 Sample extracts of dry paint film, settled dust, soil, and air particulate are analyzed for lead content by field-portable electroanalytical techniques (**2**).

4.2 Electroanalytical techniques under consideration here for lead determination in sample extract solutions include anodic stripping voltammetry (ASV) (**3**) and potentiometric stripping analysis (PSA) (**4**).

5. Significance and Use

5.1 Lead contamination in paint, dust, soil and air represents a potential health hazard to people, and field-portable analytical methods for the determination of this toxic metal in environmental samples are needed for the on-site assessment of lead hazards.

5.2 Field-portable techniques for the determination of lead in environmental samples may allow for rapid assessments of lead hazards and corresponding cost reductions compared to traditional laboratory-based analyses.

5.3 Field-portable techniques for the measurement of lead content in environmental matrices may be used for compliance with applicable federal, state, and local regulations and guidelines, providing accepted performance criteria are met (see Guide E 1775).[8]

5.4 This practice may be used in the field as an alternative to laboratory-based methods such as Test Method E 1613.

5.5 It is assumed that samples will have been collected by the pertinent ASTM sample collection methods: paint by Practice E 1729, dust wipes by Practice E 1728, vacuumed dust by Practice E 1973 or Practice D 5438, soil by Practice E 1727, and air particulate by Practice E 1553.

5.6 It is generally assumed that samples will have been prepared by a field-based sample preparation technique such as ultrasonic extraction in diluted nitric acid (**2,5**). However, this practice may also be employed in the laboratory on samples prepared by traditional laboratory-based sample preparation methods using heat and strong nitric acid, namely Practices E 1644, E 1645, E 1726, and E 1741 (**6**). Also, this practice may be used to analyze TCLP extracts for lead content that are subsequently acidified with HNO_3 (see 2.2).

5.7 Sample extracts that are analyzed by portable electroanalysis must be acidic: a minimum acidity of pH < 2 is required. Acidification is accomplished through the addition of nitric acid.

6. Apparatus and Materials

6.1 *Analytical Instrumentation*:

6.1.1 Field-portable anodic or potentiometric stripping instrument.

6.1.2 Disposable electrodes for voltammeter, if applicable.

6.1.3 Chemically inert hard-walled plastic sample extract containers.

6.1.4 Chemically inert voltammetric cell, if applicable: contains working electrode (for example, glassy carbon),

[8] Determination of instrumental detection limit and dynamic range are discussed in Guide E 1775.