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.

Current edition approved May 10, 2001. Published August 2001. Originally published as E 2051 - 99. Last previous edition E 2051 - 99.

[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.

auxiliary (counter, or secondary, for example, platinum) electrode, and reference (for example, silver/silver chloride or saturated calomel) electrode.

6.1.5 Computer or printer, or both, interfaced to instrument (optional).

6.2 *Materials and Reagents*:

6.2.1 ASTM Type I water, in accordance with Specification D 1193.

6.2.2 Supporting electrolyte, consisting of inert salt mixture such as NaCl and NaOH (reagent grade or equivalent).

6.2.3 Dissolved oxygen scavenger, such as L-ascorbic acid (tissue culture grade or equivalent).

6.2.4 Class A volumetric glassware or pipets, or both, of desired volumes, as needed.

6.2.5 Mercuric nitrate (reagent grade), if required.

6.2.6 Chemically inert sample containers, 5 mL minimum volume.

6.2.7 Calibration stock solution, 100 ± 1 mg/L of lead in dilute nitric acid solution, pH < 2.

6.2.8 Check stock solution, 100 ± 1 mg/L of lead in dilute nitric acid solution, pH < 2. This lead source must be from a different lot number or manufacturer than the calibration stock solution (see 6.2.7).

6.2.9 Nitric acid (reagent grade).[9]

6.2.10 Lead nitrate (reagent grade).

7. Procedure

7.1 *Records*—All records must be entered by hand into a laboratory notebook; these records may be augmented by electronic data entry. Record all reagent sources (manufacturers and lot numbers) used for sample preparation of calibration and check stock solutions. Reagent and calibration solution preparations used shall also be recorded and maintained. Record any inadvertent deviations, unusual happenings, or observations in real time as the samples are processed. Record all analysis data in a laboratory notebook.

7.2 *Instrumental Setup*—Ensure that the batteries of the field-portable electroanalytical instrument are sufficiently charged, and that the instrument is programmed for analysis of lead, in accordance with manufacturer's instructions.

7.2.1 *Temperature Range*—A maximum temperature range of 0 to 40°C is specified.

NOTE 1—A lesser range may be specified by the instrument manufacturer. Do not operate the instrument outside its specified operation range.

7.2.2 *Temperature of Samples*—All samples and standards shall be run at the same temperature (to within± 2°C).

NOTE 2—Changes and differences may influence analytical results.

7.3 *Calibration Standards*—Prepare a series of calibration standards, using lead nitrate, nitric acid, and water, covering the linear range of the instrument. Prepare a minimum of three calibration standards plus a blank for instrument calibration. The concentrations of calibration standards prepared shall cover the range of concentrations from 0.1 to 10 times the action level of interest.[10]

7.4 *Instrumental QC Standards*—Prepare instrumental quality control standards as described in 7.3, except the QC standards must be prepared from a different stock solution lot or manufacturer.

7.5 *Preparation of Supporting Electrolyte/Oxygen Scavenger Mixture*—an example—Where applicable, prepare this solution in the following manner: First add 117 g NaCl, then add 35.5 g L-ascorbic acid, and finally add 7.7 g NaOH to 400 mL ASTM Type I water, and dissolve in the above order. Bring final solution volume to 500 mL with water.

NOTE 3—If the supporting electrolyte/O_2 scavenger mixture is provided by the manufacturer of the instrument, it is not necessary to prepare this mixture. Also, the addition of L-ascorbic acid may not be required for PSA experiments. Furthermore, alternative supporting electrolyte mixtures to the example provided above may be called for by various manufacturers; in such instances, follow manufacturer instructions for electrolyte preparation.

7.6 *Instrument Calibration*:

7.6.1 Add mixture of supporting electrolyte and oxygen scavenger (liquid if not provided in solid form by the manufacturer; see 7.5) to the blank and calibration solutions. Account for effect of dilution on lead concentrations if the dilution effect is greater than or equal to 5 %.

7.6.2 *Initial Calibration*:

7.6.2.1 For instruments that are not calibrated electronically in the factory prior to being obtained, check the calibration of the instrument using calibration standards (instrumental QC standards) to which supporting electrolyte and oxygen scavenger (if applicable) have been added. The measured value of calibration standards for lead concentrations greater than 20 mg/L must be within 10 % of the known value; otherwise the instrument must be re-calibrated. Additionally, the measured value of calibration standards between 10 and 20 mg/L must be within 15 % of the known value, or else the instrument must be re-calibrated. Further, the measured value of calibration standards between 0 and 10 mg/L must be within 20 % of the known value; failing this, the instrument shall be re-calibrated. Also, the measured value of a calibration blank must be within 2 mg/L of zero; otherwise the instrument must be re-calibrated.

NOTE 4—O_2 scavenger is not needed in the measurement of lead by PSA.

7.6.2.2 For instruments that are calibrated by the manufacturer prior to being obtained by the user of the instrument, check the calibration as described in 7.6.2.1.

NOTE 5—Although initial calibration was made by the manufacturer, this calibration must be checked with calibration standards. It is necessary to check initial instrument calibration with calibration standards for the following reasons. Although the instrument may be performing adequately by using electronic calibration checks, it is necessary to check the calibration using solution calibration checks in order to investigate any problems that may arise in the use of the electrodes provided by the manufacturer. As described in 7.6.2.1, a measurement of the calibration

[9] If this practice is to be used for trace analysis of lead, spectroscopic grade nitric acid is required.

[10] For example, if the action level of interest for lead translates to 50 mg/L (after accounting for sample digestion and dilution), then the calibration standards must cover the range of 5 to 500 mg/L.

blank must be within ±2 mg/L of zero. Also, calibration standards of lead concentration between 0 and 10 mg/L must give readings of no more than ±20 % of the known value; failing this, the instrument must be re-calibrated. Further, calibration standards of lead concentration between 10 and 20 mg/L must give readings of no more than ±15 % of the known value; otherwise the instrument must be re-calibrated. Additionally, calibration standards with lead concentrations greater than 20 mg/L must give readings of no more than ±10 % of the known value; otherwise the instrument must be re-calibrated.

7.7 *Instrumental Quality Control*:

7.7.1 Add mixture of supporting electrolyte and oxygen scavenger (0.1 M minimum final concentration) to calibration standards. Account for dilution effect on the final lead concentrations in the QC standards if the change in volume is greater than or equal to 5 %.

7.7.1.1 To ensure reliability of QC data during analysis runs, QC samples and calibration standards shall be treated in the same manner as field samples.

7.7.2 During sample analyses runs, test the instrument with a calibration standard after a maximum of every twenty sample analyses or one per batch, whichever is smaller. For calibration standards of lead concentration between 0 and 20 mg/L, the measured value must be within 20 % of the known value; otherwise measures must be taken to ensure that the analysis of QC standards meets this specification. For calibration standards of lead concentration greater than 20 mg/L, the measured value must be within 15 % of the known value; otherwise measures must be taken to ensure that the analysis of QC standards meets this specification. For calibration standards of lead concentration of zero (that is, calibration blanks), the measured value must be within 2 mg/L of the known value (which is 0 mg/L); otherwise measures must be taken to ensure that the analysis of QC standards meets this specification.

7.8 *Instrument Calibration Check After Analyses Runs*:

7.8.1 After analyses are completed, check the calibration of the instrument to ensure that no significant instrumental drift occurred during sample runs. For calibration standards of greater than 20 mg/L, the measured value must be within 15 % of the known value. For calibration standards of between zero and 20 mg/L, the measured value must be within 20 % of the known value. For calibration blanks, the measured value must be within 2 mg/L of zero. If these final calibration checks do not meet the prescribed performance specifications, then the instrument must be re-calibrated and the samples re-analyzed.

7.9 *Sample Analysis*:

7.9.1 *Analysis Using Disposable Electrodes*—Follow the procedure below if disposable, single-use electrodes are employed for electroanalytical lead measurements.

7.9.1.1 Choose the desired lead analysis program (for example, for analysis of paint, dust, or soil, if applicable), in accordance with manufacturer instructions. If no pre-set program is provided by the manufacturer for the determination of lead in specific matrices, choose or design a program to effect lead analysis by ASV or PSA, in accordance with manufacturer guidelines.

7.9.1.2 Open the sample extract solution and, if necessary, transfer to a suitable chemically inert sample analysis vessel.

7.9.1.3 Place the sample analysis vessel into a metal support sleeve, in accordance with manufacturer instructions.

7.9.1.4 Add mixture of supporting electrolyte and oxygen scavenger (0.1 M minimum final concentration) to the sample, in accordance with instrument manufacturer instructions. Account for dilution effect on the final lead concentrations in the samples if the change in volume is greater than or equal to 5 %.

NOTE 6—O_2 scavenger is not needed in the measurement of lead by PSA. Also, electroanalytical devices based on the use of ultramicroelectrodes or microelectrode array[11] may not require the addition of supporting electrolyte.

7.9.1.5 Insert a clean, fresh disposable electrode into the electrode holder so as to make electrical contact with the field-portable electroanalytical instrument (in accordance with manufacturer instructions).

7.9.1.6 Introduce a fresh, clean electrode into the sample solution so as to immerse the electrode probe into the solution, in accordance with manufacturer instructions.

7.9.1.7 Conduct the analysis, and record results in terms of desired units of lead content (for example, mg/mL, µg/sample, etc.). Alternatively, signal from current due to lead peaks in an ASV voltammogram, or due to potential arising from lead oxidation in a potentiometric stripping experiment, may be recorded and mathematically converted to desired units of lead content. (See Section 8.)

NOTE 7—If desired, a computer may be interfaced to the portable electroanalytical instrument for conducting analyses and downloading data.

7.9.2 *Analysis Using Reusable or Renewable Electrodes*:

7.9.2.1 *Deposition of a Mercury Film*:

(a) Assemble the electrochemical cell consisting of a working (for example, glassy carbon) electrode, auxiliary (for example, platinum) electrode, and reference (for example, Ag/AgCl or saturated calomel reference) electrode, in accordance with manufacturer instructions.

(b) Introduce ASTM Type I water into the cell, and add supporting electrolyte into the cell to effect a minimum concentration of 0.1 M.

(c) Pipet a sufficient quantity of mercuric nitrate stock solution into the cell containing a minimum of 0.1 M supporting electrolyte to effect a final $Hg(NO_3)_2$ concentration of 100 ppb. Mix to ensure homogeneity.

(d) Choose or create the desired mercury film deposition program to effect the deposition of a mercury film onto the working (for example, glassy carbon) electrode, in accordance with manufacturer instructions.

(e) Drain the cell of its contents ($Hg(NO_3)_2$ in 0.1 M or greater supporting electrolyte).

(f) Rinse and empty the cell three times with ASTM Type I water.

7.9.2.2 *Sample Analysis*:

(a) If necessary, clean the cell thoroughly, in accordance with manufacturer's instructions, and drain the cell.

(b) Rinse the cell three times with ASTM Type I water.

(c) Pipet a selected volume of ASTM Type I water into the electroanalytical cell. Mix to ensure homogeneity.

[11] M. Fleischmann, S. Pons, D. R. Rolison, and P. P. Schmidt, Ultramicroelectrodes; Datatech: Morganton, NC, 1987.

(d) Open the sample extract solution and transfer an aliquot to the cell. The total volume of solution in the cell shall be at least half the cell volume.

NOTE 8—It may be necessary to either increase or decrease the amount of sample solution and accordingly decrease or increase the volume of dilution water that is introduced into the cell, depending on the analytical results. That is, if the concentration is near or below the detection limit of the instrument, then larger sample aliquots must be used and the concentration of the electrolyte adjusted. Alternatively, if the lead signal exceeds the dynamic range of the instrument, then smaller sample aliquots must be analyzed.

(e) Pipet an equal volume of supporting electrolyte/O_2 scavenger solution into the cell containing sample extract solution. This is accomplished by adding the mixture (see 7.5) to the diluted or undiluted sample in a 1:1 ratio. Mix to ensure homogeneity.

(f) Choose the desired lead analysis program (for example, for analysis of paint, dust, or soil, if applicable), in accordance with manufacturer instructions. If no pre-set program is provided by the manufacturer for the determination of lead in specific matrices, choose or design a program to effect lead analysis by ASV or PSA, in accordance with manufacturer guidelines.

(g) Conduct the analysis, and record results in the desired units of lead content (for example, mg/mL, µg/sample, etc.). Alternatively, signal from current due to lead peaks in an ASV voltammogram, or due to potential arising from lead oxidation in a potentiometric stripping experiment, may be recorded and mathematically converted to desired units of lead content.

NOTE 9—If desired, a computer may be interfaced to the portable electroanalytical instrument for conducting analyses and downloading data.

(h) Drain the cell of its contents.

(i) Rinse the cell three times with ASTM Type I water.

7.9.3 *Renewal of Electrodes*—If it is necessary to renew the mercury electrode surface (as indicated by the inability to meet calibration specifications (see 7.6.2.1) after prolonged use of the instrument), follow manufacturer instructions.

NOTE 10—Generally, this requires the following protocol: (1) Remove electrode from electrochemical cell. (2) Physically, but gently, remove mercury film from electrode using a wetted laboratory wipe. (3) Re-insert electrode into cell. (4) Introduce mercury nitrate solution into cell. (5) Re-deposit Hg film by applying a potential sufficient to effect reduction of dissolved mercuric cations to mercury metal on the electrode surface. (6) Run analyses of calibration standards of known concentration until a plateau of performance is reached where the response is consistently within QC specifications; this should require no more than two or three successive analyses.

7.10 *Instrument Storage*—Store the instrument in accordance with manufacturer instructions; avoid exposure to extremes of temperature.

NOTE 11—For instruments that employ renewable electrodes, the electrochemical cell may need to be stored with solution inside the cell in order to prevent electrode fouling.

7.11 *Disposal of Test Materials*—Dispose of all waste materials left over from testing according to applicable federal, state, and local regulations.

8. Calculation

8.1 Prepare a calibration curve of instrument response (recorded result) using a linear regression fit.[12] Convert all instrumental measurements on instrumental QC standards and sample extracts to lead concentration (in desired units) using the calibration curve. Compute the standard error about the regression line using instrumental QC data.[13]

NOTE 12—Some instruments will automatically prepare a calibration curve based on a linear regression fit.

8.1.1 Convert all measurements for instrumentally measured sample extracts (including QC sample extracts) to lead concentration using the calibration line.

8.2 Calculate the lead concentration in the sample extract after electrochemical analysis as follows:

$$(Pb) = (E)(D) \quad (1)$$

where:

Pb = measured lead concentration in sample extract,
E = electroanalytically measured lead concentration, and
D = dilution factors required during analysis to produce a measured lead level within the calibration curve.

8.2.1 For sample extracts generating lead measurements below the instrumental detection limit (IDL),[14] the IDL shall be used for performing calculations. A less-than sign (<) shall be used on results from such calculations to indicate the uncertainty of these values.

8.3 Calculation of the lead levels in the originally extracted samples is dependent on the sample matrix (paint, dust, soil, or air particulate) and sample preparation procedure. (Instrumental software may perform these calculations automatically.) The following calculations give examples for each of these matrices:

8.3.1 *Lead in Paint*—For lead content in paint on an area basis,

$$(Pb) = [(A)(B)]/[(C)(1000)] \quad (2)$$

where:

Pb = lead content in mg/cm^2,
A = lead concentration in sample extract in mg/L,
B = final solution volume in mL, and
C = sample collection area in cm^2.

For lead content in paint on a concentration basis, use Eq 2 except replace C with the sample mass in grams. This will give a measured lead concentration in units of milligrams of lead per gram of sample.

8.3.2 *Lead in Dust Wipes*—For lead content in dust wipe samples:

[12] The protocol for preparation of calibration lines using a linear regression fit can be found in many texts, for example: Wernimont, G. T., *Use of Statistics to Develop and Evaluate Analytical Methods*; Association of Official Analytical Chemists: Arlington, VA (1985).

[13] Computation of calibration lines, and error about the calibration line, is described in many texts, for example: Miller, J. C., and Miller, J. N., *Statistics for Analytical Chemistry*; Ellis Horwood: Chichester, U.K. (1984).

[14] For the purposes of this standard, the IDL shall be determined as described in the following paper: American Chemical Society on Environmental Improvement-,"Principles of Environmental Analysis;" *Analytical Chemistry*, Vol 55, p. 2210 (1983).

$$(Pb) = [(A)(B)]/(C) \quad (3)$$

where:
Pb = lead content in µg/cm^2
A = lead concentration in sample extract in mg/L,
B = final solution volume in mL, and
C = sample collection area in cm^2.

The lead mass per wipe or vacuum sample, then the lead content in micrograms per wipe, is simply A times B.

8.3.3 *Lead in Soil*—For lead concentration in terms of mass of lead per mass of sample, the applicable expression to be used is:

$$(Pb) = [(A)(B)]/(C) \quad (4)$$

where:
Pb = lead concentration in µg/g,
A = lead concentration in the sample extract in mg/L,
B = the final solution volume, and
C = the mass of soil sample in g.

8.3.4 *Lead in Air Particulate*—For lead concentration in terms of mass of lead per unit sampled volume, the applicable expression is:

$$(Pb) = [(A)(B)]/(C) \quad (5)$$

where:
Pb = lead content in µg/m^3,
A = lead concentration in the sample extract in mg/L,
B = final solution in mL, and
C = sample volume in m^3.

For lead content in terms of micrograms per sample, use Eq 1, with Pb = measured lead in µg/filter, A = measured lead in sample extract in mg/L, and B = final solution volume in mL.

9. Report

9.1 Data to report include sample collection and preparation information, all final sample analysis results, sample preparation QC analysis results (method blanks, calibration standards), and instrumental QC data. Record temperature at which samples were run. Report dilution factors, analysis volumes, and any noteworthy observations that may influence the analysis results. Also, document type of field-portable electroanalytical equipment used, manufacturer and serial number.

10. Keywords

10.1 anodic stripping voltammetry; electroanalysis; field-portable; lead; potentiometric stripping analysis

REFERENCES

(**1**) Wang, J., *Analytical Electrochemistry*; VCH Publishers: New York, 1994.
(**2**) Ashley, K., "Electroanalytical Applications in Occupational and Environmental Health;" *Electroanalysis*, Vol 6, 1994, pp. 805–820.
(**3**) Ashley, K., "Ultrasonic Extraction and Field-Portable Anodic Stripping Voltammetry of Lead from Environmental Samples;" *Electroanalysis*, Vol 7, 1995, pp. 1189–1192.
(**4**) Wang, J., *Stripping Voltammetry*; VCH Publishers: New York, 1985.
(**5**) EPA, *Standard Operating Procedure for the Field Analysis of Lead in Paint, Bulk Dust, and Soil by Ultrasonic, Acid Digestion and Colorimetric Measurement*; EPA: Research Triangle Park, NC, 1993.
(**6**) Ashley, K., Schlecht, P.C., Song, R., Feng, A., Dewalt, G., and McKnight, M.E., "ASTM Sampling Methods and Analytical Validation for Lead in Paint, Dust, Soil, and Air," in *Sampling Environmental Media* (ASTM STP 1282), Morgan, J. H., Ed., American Society for Testing and Materials: Philadelphia, 1996.