Designation: E1979 – 17

Standard Practice for Ultrasonic Extraction of Paint, Dust, Soil, and Air Samples for Subsequent Determination of Lead[1]

This standard is issued under the fixed designation E1979; 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 an ultrasonic extraction procedure for the extraction of lead from environmental samples of interest in lead abatement and renovation (or related) work, for analytical purposes.

1.2 Environmental matrices of concern include dry paint films, settled dusts, soils, and air particulates.

1.3 Samples subjected to ultrasonic extraction are prepared for subsequent determination of lead by laboratory analytical methods.

1.4 This practice includes, where applicable, descriptions of procedures for sample homogenization and weighing prior to ultrasonic extraction.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This practice 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 practice to establish appropriate safety and health practices and determine the applicability of regulatory limitation prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

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

D7144 Practice for Collection of Surface Dust by Micro-vacuum Sampling for Subsequent Metals Determination

E631 Terminology of Building Constructions

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

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

E1728 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination

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

E1775 Guide for Evaluating Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead

E1792 Specification for Wipe Sampling Materials for Lead in Surface Dust

3. Terminology

3.1 *Definitions:* For definitions of terms relating to this practice that do not appear in this section, refer to Terminologies E631 and E1605.

3.1.1 *extraction*—the dissolution of target analytes from a solid matrix into a liquid form.

3.1.1.1 *Discussion*—During sample digestion or extraction, target analytes are extracted (solubilized) into solution to enable subsequent determination by analytical techniques (for example, see Test Method E1613).

3.1.2 *ultrasonic extraction*—the use of ultrasonic energy and acidic or basic solution to extract targeted analytes from samples.

3.1.2.1 *Discussion*—The extract solution is subsequently analyzed for the determination of targeted analytes.

4. Summary of Practice

4.1 Samples of paint, settled dust (wipe or vacuum), soil, or airborne particles, obtained by ASTM sample collection methods (see Practice E1729; Practice E1728, Practice D5438 and

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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.

Practice D7144; Practice E1727, respectively), are subjected to ultrasonic extraction in dilute nitric acid for a delineated time period.

4.2 Paint samples are ground, homogenized and weighed, if necessary, prior to ultrasonic extraction.

4.3 Soil and bulk dust vacuum samples are sieved, homogenized, and weighed prior to ultrasonic extraction.

4.4 If applicable, dust filter samples are weighed prior to ultrasonic extraction.

4.5 Sample extracts are diluted with ASTM Type I water (see Specification D1193) in preparation for subsequent determination of lead by analytical techniques (for example Test Method E1613, Guide E1775, and NIOSH methods 7082, 7105, and 7300 (**1**)[3]).

4.6 This practice was developed based on an EPA standard operating procedure (ultrasonic extraction followed by colorimetric lead analysis (**2**)) and published protocols (ultrasonic extraction followed by anodic stripping voltammetric determination of lead) (**3**) (**4**).

5. Significance and Use

5.1 Ultrasonic extraction using dilute nitric acid is a simpler and easier method for extracting lead from environmental samples than are traditional digestion methods that employ hot plate or microwave digestion with concentrated acids (**1**), (**3**), (**5**), (**6**). Hence, ultrasonic extraction may be used in lieu of the more rigorous strong acid/high temperature digestion methods (for example, see Ref (**1**) and Test Method E1613), provided that the performance is demonstrated using accepted criteria as delineated in Guide E1775.

5.2 In contrast with hot plate or microwave digestion techniques, ultrasonic extraction is field-portable, which allows for on-site sample analysis.

6. Apparatus and Materials

6.1 *Sonicator,* 50 W minimum power; proper operation of the sonicator shall be confirmed prior to initial use and periodically thereafter.

6.2 *Plastic Centrifuge Tubes,* 50 mL, with screw caps.

6.3 *Analytical Balance,* accuracy better than or equal to ±0.002 g.

6.4 *Mortar and Pestle,* pug mill, or wigglebug.

6.5 *Dry Ice* (CO_2(s)).

6.6 *Cooler,* for storage of dry ice.

6.7 *Plastic or Glass Rods,* 0.6 to 1.0 cm diameter, 15 to 20 cm length; tapered at one end to conform to the shape of the bottoms of the 50 mL centrifuge tubes.

6.8 *Powderless Plastic Gloves*.

6.9 *Tweezers*.

6.10 *Class A Pipets,* 5 or 10 mL or both, and other volumes as needed.

NOTE 1—Precision digital mechanical pipettors using replaceable tips meet Class A requirements.

6.11 *Laboratory Wipes,* wet or dry, or both.

6.12 *Power Source for Sonicator*.

6.13 *Sieves,* Number 10 (1.9 mm) and 250 µm.

6.14 *Collection Tray*.

6.15 *Rubber Mallet*.

6.16 *Nylon Brush*.

6.17 *Aluminum Pie Tins*.

6.18 *Dust Mask*.

6.19 *Glass or Plastic Stirring Rods*.

6.20 *Aluminum Foil* (conventional thickness for household use, that is, not heavy duty).

6.21 *Graphite Pencil*.

7. Reagents

7.1 *ASTM Type I Water,* in accordance with Specification D1193.

NOTE 2—Commercially available distilled or deionized water meets ASTM Type I water specifications.

7.2 *Extraction Solution,* 10 % or 25 % v/v HNO_3/H_2O; prepared from reagent grade concentrated nitric acid (70 to 71 % HNO_3) and ASTM Type I water.

NOTE 3—Air filter samples require 10 % nitric acid (v/v) for extraction, while all other sample matrices require 25 % v/v HNO_3/H_2O.

8. Procedure

8.1 *Testing of Sonicator:*

8.1.1 Before use, ensure proper operation of the sonicator by employing the following diagnostic test.

8.1.1.1 Turn on the sonicator and allow for a reasonable warm-up period, as recommended by the instrument manufacturer.

8.1.1.2 Insert the tip of a graphite pencil into the bath.

8.1.1.3 If the sonication device is operating properly, graphite in solution will be observed streaming off the tip of the pencil.

8.1.2 Alternatively, demonstrate proper operation of the sonicator according to the following procedure:

8.1.2.1 Fill the sonicator bath with warm water (ca. 45 °C) to a level about half-full, and add a small amount (for example, three drops) of surfactant

8.1.2.2 Turn on the sonicator for a minimum of 5 min to degas the solution. Turn off the sonicator.

8.1.2.3 Place aluminum foil (that is cut to a size conforming to ½ to ¾ of the area of the bottom of the sonicator bath) on the bottom of the sonicator bath. Lower the foil at an angle to prevent the trapping of air beneath the foil. Ensure that a narrow layer of solution remains between the foil and the bottom of the sonicator bath. The foil shall be parallel and centered to the bottom of the sonicator.

8.1.2.4 Turn on the sonicator for a period of 45 s.

8.1.2.5 Examine the aluminum foil after sonication. The foil should be observed to contain a myriad of small holes and

[3] The boldface numbers given in parentheses refer to a list of references at the end of the text.

bumps, and may be torn apart. Also, the perforation observed should be uniform; that is, all portions of the foil should be observed to have a high density of holes and perhaps tears. If the foil is not affected in this manner, then the sonicator performance is inadequate for the purposes of this practice.

8.2 *Dry Paint Film Samples:*

NOTE 4—This practice assumes paint samples collected in accordance with Practice E1729.

8.2.1 Don a pair of gloves.

8.2.1.1 If the desired reported lead concentration units are to be in terms of lead mass per unit area of sample, and the estimated sample mass is greater than or equal to 0.25 g, then quantitatively transfer the entire sample to an analytical balance and weigh to the nearest ±0.002 g. Under these circumstances, do quantitative transfer and sample weighing prior to grinding and homogenization as described later in this practice.

8.2.1.2 If the desired reporting units of lead concentration are to be in terms of lead mass per unit mass of sample, or if the desired reporting units are to be in terms of lead mass per unit area and the estimated mass of the sample is less than 0.25 g, then quantitatively transfer the sample to a mortar and pestle or a labeled 50 mL centrifuge tube. Use tweezers if necessary for quantitative sample transfer. Clean tweezers before and after use with laboratory wipes.

8.2.2 *Sample Grinding and Homogenization:*

8.2.2.1 Grind and homogenize paint film sample to a fine powder using a mortar and pestle, pug mill, or wigglebug. Quantitatively transfer the ground and homogenized a paint sample to a weighing vessel or weighing paper (see below). Thoroughly clean mortar and pestle, wigglebug, or pug mill with moistened laboratory wipes prior to and following grinding, and then dry.

8.2.2.2 Alternatively, place paint film sample in a clean, dry 50 mL centrifuge tube, and place the tube in dry ice. Using a plastic or metal rod, grind and homogenize the super-cooled, brittle paint sample to a fine powder. Thoroughly clean rod with laboratory wipes prior to and following grinding procedure. Avoid condensation of water onto the sample by sealing the sample and allowing it to warm to ambient temperature before weighing.

NOTE 5—The use of dry ice as described assists greatly in the grinding and homogenization of dry paint film samples.

8.2.3 If the total sample mass is greater than or equal to 0.25 g, or if the total sample mass is less than 0.25 g and the desired reported lead concentration is to be in units of mass of lead per unit mass of sample, weigh 0.05 to 0.25 g of ground and homogenized paint sample to the nearest ±0.002 g, and record the sample mass.

NOTE 6—If the desired reporting units are to be in terms of mass of lead per unit area of sample, and the sample mass is estimated to be less than 0.25 g, a larger sample may be collected in order to obtain more mass (thereby enabling the sample to be treated as described in 8.2.1.1).

8.2.4 If the total sample mass is less than 0.25 g and the desired reported lead concentration is to be in terms of mass of lead per unit area of collected sample, then the sample need not be weighed. However, the entire ground and homogenized sample must be placed in the tube for extraction as described below.

8.2.5 Place the sample in a clean, dry, labeled 50 mL polypropylene centrifuge tube. To ensure quantitative sample transfer, rinse materials used for transferring samples with a minimum of dilute (5 %) nitric acid, and direct the rinsate into the centrifuge tube.

8.2.6 Using a class A pipet, introduce 5 mL of 25 % HNO_3 to the centrifuge tube containing the ground and homogenized paint sample. Shake the centrifuge tube briefly to ensure that no solid paint material remains stuck to the bottom of the tube. Rinse the inside of the centrifuge tube with a minimum quantity of 5 % HNO_3 in order to ensure immersion of all solid material.

8.2.7 Cap the centrifuge tube, and place in an upright position in the sonicator bath.

8.2.8 Ensure that the bath of the sonicator contains enough water so that the water level is at least 2.5 cm above the level of liquid within the centrifuge tube.

8.2.9 Repeat steps 8.2.1 – 8.2.8 as needed, depending on the size of the ultrasonic bath and the number of samples.

NOTE 7—Depending on the size of the sonicator, many centrifuge tubes may be immersed in the bath at one time. A custom rack for the centrifuge tubes may be purchased or constructed to allow for the regular and orderly placement of multiple tubes in the sonicator bath.

8.2.10 Apply ultrasonic energy to the crushed and acidified samples within the immersed centrifuge tubes for at least 30 min.

8.2.11 Remove centrifuge tubes from the bath. Keep tubes in upright position.

8.2.12 Remove caps from centrifuge tubes that contain samples that were subjected to ultrasonic agitation, and dilute acidified extracts to the 50 mL mark with ASTM Type I water.

8.2.13 Re-cap and then shake the tubes for 5 to 10 s, and allow the contents to settle. The samples are now ready for analysis for lead content.

NOTE 8—The sample solutions may require filtration or centrifugation prior to analysis.

8.3 *Settled Dust Wipe Samples:*

NOTE 9—This procedure assumes dust wipe samples were collected in accordance with Practice E1728 using wipes that conform to Specification E1792.

8.3.1 Don a pair of gloves.

8.3.2 If not already placed in a labeled 50 mL centrifuge tube, remove the wipe sample from the sample container with a pair of clean tweezers, and place it in a labeled clean and dry 50 mL centrifuge tube. Shove the wipe to the bottom of the tube with a clean glass or plastic rod. Rinse the original sample container (if applicable), tweezers and rod with a minimum of dilute (5 %) nitric acid to ensure quantitative sample transfer; make sure that the rinsate falls into the centrifuge tube. Clean tweezers and rods before and after use with laboratory wipes.

8.3.3 Using a class A pipet, introduce 15 mL of 25 % HNO_3 to the centrifuge tube containing the wipe sample, and cap the tube. If needed, add more diluted 25 % HNO_3 in 5 mL aliquots to ensure that the wipe is completely covered by acid extraction

solution. If bubbles are observed inside of the immersed wipe, apply pressure to the wipe with a clean stirring rod in order to force the bubbles up and out.

8.3.4 Place centrifuge tube (containing wipe sample immersed in dilute acid) upright in the sonicator bath, and ensure that the water level in the bath is at least 2.5 cm above the liquid level within the tube.

8.3.5 Repeat 8.3.1 – 8.3.4 for successive samples as needed, depending on the size of the ultrasonic bath.

NOTE 10—Depending on the size of the sonicator, many centrifuge tubes may be immersed in the bath at one time. A custom rack for the centrifuge tubes may be purchased or constructed to allow for the regular and orderly placement of multiple tubes in the sonicator bath.

8.3.6 Apply ultrasonic energy to the acid-immersed wipe sample for at least 20 min.

8.3.7 Turn off the sonicator.

8.3.8 Carefully uncap each centrifuge tube.

8.3.9 Using a clean glass or plastic stirring rod for each sample, push down on each wipe within each centrifuge tube in order to force bubbles (formed during sonication) up and out.

8.3.10 Recap the tubes.

8.3.11 Apply ultrasonic energy again for a minimum of 20 min.

NOTE 11—Longer extraction periods may be required for wipe samples. Quantitative recoveries from spiked wipes must be demonstrated using performance evaluation materials traceable to a primary standard (**7**).

8.3.12 Remove centrifuge tubes from the bath. Keep tubes in upright position.

8.3.13 Remove caps from centrifuge tubes that contain samples which were subjected to ultrasonic agitation, and dilute acidified extracts to the 50 mL mark with ASTM Type I water.

8.3.14 Cap and then shake the tubes for 5 to 10 s, and allow the contents of the tubes to settle. The samples are now ready for analysis for lead content.

NOTE 12—The sample solutions may require filtration or centrifugation prior to analysis.

8.4 *Settled Dust Vacuum Filter Samples:*

NOTE 13—This assumes dust samples collected according to Practice D7144.

8.4.1 Don a pair of gloves.

8.4.2 If a pre-weighed filter cassette was employed for sample collection, weigh the cassette to the nearest ±0.002 g and record the mass.

8.4.3 Open filter cassette, and, with gentle tapping, transfer into a labeled 50 mL centrifuge tube all dust that does not adhere to the filter within the filter cassette.

8.4.4 Using clean tweezers, remove the filter from the filter cassette and place in the same centrifuge tube as that containing the un-adhered dust previously transferred. Using a plastic or glass rod, shove the filter sample to the bottom of the centrifuge tube. To effect quantitative sample transfer, rinse the tweezers and rod with a minimum of dilute (5 %) nitric acid, ensuring that the rinsate is directed into the centrifuge tube. Clean tweezers and rod prior to and following use with laboratory wipes.

8.4.5 Using a class A pipet, introduce 10 mL of 25 % HNO_3 into the centrifuge tube containing the transferred dust and filter, and cap the tube.

8.4.6 Place centrifuge tube (containing dust and filter sample immersed in diluted nitric acid) upright in the sonicator bath, and ensure that the water level in the bath is at least 2.5 cm above the liquid level within the tube.

8.4.7 Repeat 8.4.1 – 8.4.6 for successive samples as needed, depending on the size of the ultrasonic bath.

NOTE 14—Depending on the size of the sonicator, many centrifuge tubes may be immersed in the bath at one time. A custom rack for the centrifuge tubes may be purchased or constructed to allow for the regular and orderly placement of multiple tubes in the sonicator bath.

8.4.8 Apply ultrasonic energy to the acid-immersed dust vacuum filter sample for at least 30 min.

8.4.9 Remove centrifuge tubes from the bath. Keep tubes in upright position.

8.4.10 Remove caps from centrifuge tubes that contain dust vacuum filter samples that were subjected to ultrasonic agitation, and dilute acidified extracts to the 50 mL mark with ASTM Type I water.

8.4.11 Cap and then shake the tubes for 5 to 10 s, and allow the contents to settle. The samples are now ready for analysis for lead content.

NOTE 15—The sample solutions may require filtration or centrifugation prior to analysis.

8.5 *Settled Dust Vacuum Bulk Samples:*

NOTE 16—This assumes dust samples collected according to Practice D5438.

8.5.1 Don a pair of gloves, and a dust mask.

8.5.2 Clean the sieve using the following procedure: First, brush the sieve with the nylon brush, and then wipe the sieve with a moist laboratory wipe and allow to dry.

8.5.3 Quantitatively transfer the dust sample to a clean 250 µg sieve that is placed atop a clean collection tray.

NOTE 17—Some bulk dust vacuum samples may not require sieving.

8.5.4 Remove large foreign objects from the dust with tweezers.

8.5.5 Gently and repeatedly tap the side of the sieve with a rubber mallet for about 30 s.

8.5.6 Stir the dust with a clean stirring rod, and repeat the tapping process.

8.5.7 Repeat 8.5.6 three more times.

8.5.8 Remove the sieve and stir the sieved dust (with a clean stirring rod) in the collection tray to facilitate homogenization.

8.5.9 Weigh 0.25 to 0.50 g of sieved, homogenized bulk dust sample to the nearest ±0.002 g, and record the sample mass.

8.5.10 Quantitatively transfer the weighed sample to a labeled 50 mL polypropylene centrifuge tube. Rinse materials used for sample transfer with a minimum of dilute (5 %) nitric acid, ensuring that the rinsate falls into the centrifuge tube.

8.5.11 Using a class A pipet, introduce 5 mL of 25 % HNO_3 into the centrifuge tube containing the weighed dust sample. Rinse the sides of the container with a minimum quantity of

5 % nitric acid solution to ensure immersion of all solid material that may otherwise be stuck to the inside of the tube.

8.5.12 Cap the centrifuge tube, and place the tube (containing dust sample immersed in acid solution) upright in the sonicator bath, and ensure that the water level in the bath is at least 2.5 cm above the liquid level within the tube.

8.5.13 Repeat 8.5.1 – 8.5.12 for successive samples as needed, depending on the size of the ultrasonic bath.

NOTE 18—Depending on the size of the sonicator, many centrifuge tubes may be immersed in the bath at one time. A custom rack for the tubes may be purchased or fabricated to allow for the regular and orderly placement of multiple tubes in the sonicator bath.

8.5.14 Apply ultrasonic energy to the acid-immersed dust samples for at least 30 min.

8.5.15 Remove centrifuge tubes from the bath. Keep tubes in upright position.

8.5.16 Remove caps from centrifuge tubes containing dust samples that were subjected to ultrasonic energy while immersed in acid solution. Dilute to the 50 mL mark with ASTM Type I water.

8.5.17 Cap the tubes and shake for 5 to 10 s, and then allow the contents to settle. The samples are now ready for analysis for lead content.

NOTE 19—The sample solutions may require filtration or centrifugation prior to analysis.

8.6 *Soil Samples:*

NOTE 20—This assumes soil samples collected according to Practice E1727.

8.6.1 Don a pair of gloves.

8.6.2 Quantitatively transfer soil sample from sample container to a clean aluminum pie tin or comparable alternative for subsequent drying of sample.

8.6.3 *Drying of Soil Samples:*

8.6.3.1 Allow sample to air dry. Ensure that drying sample is not placed in a location where it may be contaminated.

NOTE 21—Soil samples may be split after homogenization or dried according to Practice E1726 to determine the water content of air-dried samples, or both.

8.6.3.2 Alternatively, dry soil samples as described in Practice E1726.

8.6.4 After sample is dry, sieve it as follows, or as described in Practice E1726. Place a clean No. 10 (1.9 mm) sieve on the collection tray. Clean as described in 8.6.6.

8.6.5 Place the soil sample on the 1.9 mm (No. 10) sieve and, using a gloved hand, gently rub the soil round and round to break up clumps and facilitate sieving. Continue until no more material passes through the sieve.

8.6.6 Remove the No. 10 (1.9 mm) sieve and discard the material in the sieve. Clean the sieve thoroughly. Sieves are to be cleaned between samples by first brushing with a nylon brush, secondly wiping with moist laboratory wipes, and then allowing them to dry.

8.6.7 Mix the soil sample on the collection tray to facilitate homogenization. Transfer the soil sample to a mortar, and using a pestle, crush the soil sample as finely as possible. Homogenize further by mixing and crushing.

8.6.8 Quantitatively transfer the crushed, homogenized soil sample to a clean 250 µm sieve, that is placed atop a clean collection tray.

8.6.9 Don a clean pair of gloves. Using gloved fingers, gently rub the soil round and round to facilitate sieving. Continue until no more material passes through the 250 µm sieve. When finished with sieving, clean the sieve. Sieves are to be cleaned between samples by first brushing with a nylon brush, secondly wiping with moist laboratory wipes, and then allow to dry.

8.6.10 Material that does not pass through the 250 µm sieve shall be returned to the mortar and pestle for additional grinding, in accordance with 8.6.7.

8.6.11 Repeat 8.6.8 and 8.6.9 for the additional material from 8.6.10.

8.6.12 Mix (homogenize) sieved material and weigh 0.25 to 0.50 g of the soil sample (following sieving through the 250 µm sieve) to the nearest ±0.002 g, and record the sample mass.

8.6.13 Quantitatively transfer the weighed soil sample to a labeled 50 mL polypropylene centrifuge tube. To ensure quantitative sample transfer, rinse materials used in sample transfer with a minimum of 5 % nitric acid, and direct the rinsate into the centrifuge tube.

8.6.14 Using a class A pipet, introduce 5 mL of 25 % HNO_3 into the centrifuge tube containing the soil sample. Rinse the inside of the tube with a minimum of 5 % HNO_3 to ensure that no solid material remains stuck on the sides of the tubes.

8.6.15 Cap the centrifuge tube (containing the soil sample immersed in dilute acid), and place upright in the sonicator bath. Ensure that the water level in the bath is at least 2.5 cm above the level of liquid inside the tube.

8.6.16 Repeat 8.6.1 – 8.6.15 for successive samples as needed, depending on the size of the ultrasonic bath.

NOTE 22—Depending on the size of the sonicator, many centrifuge tubes may be immersed in the bath at one time. A custom rack for the centrifuge tubes may be purchased or constructed to allow for the regular and orderly placement of multiple tubes in the sonicator bath.

8.6.17 Apply ultrasonic energy to the sieved, homogenized, weighed, and acidified soil samples within the immersed centrifuge tubes for a minimum of 30 min.

8.6.18 Remove centrifuge tubes from the bath. Keep tubes in upright position.

8.6.19 Remove caps from centrifuge tubes that contain samples that were subjected to ultrasonic agitation, and dilute acidified extracts to the 50 mL mark with ASTM Type I water.

8.6.20 Re-cap tubes, shake the tubes for 5 to 10 s, and then allow the contents to settle. The samples are now ready for analysis for lead content.

NOTE 23—The sample solutions may require filtration or centrifugation prior to analysis.

8.7 *Airborne Particulate Samples:*

NOTE 24—This assumes samples collected according to Test Method D6785.

8.7.1 Don a pair of gloves.

8.7.2 Open filter cassette, and, with gentle tapping, transfer into a labeled 50 mL centrifuge tube all particulate that does not adhere to the filter within the filter cassette.

8.7.3 Using clean tweezers, remove the filter from the filter cassette and place in the same centrifuge tube as that containing the un-adhered particulate previously transferred. Using a clean polypropylene rod, shove the filter sample to the bottom of the centrifuge tube. Clean tweezers and rod prior to and following use with laboratory wipes.

8.7.4 Using a class A pipet, introduce 10 mL of 10 % nitric acid into the centrifuge tube containing the transferred particulate and filter, and cap the tube.

8.7.5 If necessary, rinse the inside of the centrifuge tube with a minimum of 5 % HNO_3 to ensure immersion of all solid particulate material.

8.7.6 Place centrifuge tube (containing particulate and sample immersed in 10 % nitric acid) upright in the sonicator bath, and ensure that the water level in the bath is at least 2.5 cm above the liquid level within the tube.

8.7.7 Repeat 8.7.1 – 8.7.6 for successive samples as needed, depending on the size of the ultrasonic bath.

NOTE 25—Depending on the size of the sonicator, many centrifuge tubes may be immersed in the bath at one time. A custom rack for the centrifuge tubes may be purchased or constructed to allow for the regular and orderly placement of multiple tubes in the sonicator bath.

8.7.8 Apply ultrasonic energy to the acid-immersed air filter sample for at least 30 min.

8.7.9 Remove centrifuge tubes from the bath. Keep tubes in upright position.

8.7.10 Remove caps from centrifuge tubes that contain air filter samples that were subjected to ultrasonic agitation, and dilute acidified extracts to the 50 mL mark with ASTM Type I water.

8.7.11 Cap tubes, shake the tubes for 5 to 10 s, and allow the contents to settle. The samples are now ready for analysis for lead content.

NOTE 26—The sample solutions may require filtration or centrifugation prior to analysis.

9. Quality Assurance/Quality Control

9.1 Quality assurance/quality control procedures shall be followed as described in Guide E1775. All performance evaluation materials and samples shall be handled in the same manner as field samples. The frequency and nature of QA/QC samples is given in Test Method E1613.

10. Records

10.1 Field and laboratory data related to sample preparation must be documented in a sample log form or field/laboratory notebook, or recorded electronically.

NOTE 27—Field or laboratory notebooks are useful for recording field data even when preprinted sample data forms are used.

10.1.1 If field or laboratory notebooks are used, then the notebooks shall be bound with numbered pages. All entries on sample data forms and field or laboratory notebooks shall be made using ink with signature and date of entry. Any entry errors shall be corrected using a single line through the erroneous entry (no scratch outs), accompanied by the initials of the person making the correction, and the date of the correction. The use of electronic notebooks is also acceptable as long as similar security and record control precautions are in place.

NOTE 28—These procedures are important to properly document and trace field and laboratory data.

10.2 At a minimum, the following information shall be documented:

10.2.1 Project or client name, address, city/state location,

10.2.2 General description of sample preparation site,

10.2.3 Specific information on type of sample (paint, dust wipe, soil, air),

10.2.4 Information as to what specific sample preparation protocol was used,

10.2.5 For each sample treated, an individual and unique sample identifier, and date of preparation. This shall be recorded on the sample container in addition to the field documentation, and

10.2.6 For each sample handled, a personal identifier for the person having collected the sample, the person preparing the sample, and the sampling location information.

10.3 Record all information regarding the preparation of samples (both QA/QC samples and samples collected in the field) as follows:

10.3.1 Record all reagent sources and lot numbers used for sample preparation. For each entry, include the date(s), personal identifier(s), and signature(s) of the person(s) making the entry. Record any inadvertent deviations, unusual occurrences, and observations on a real-time basis as samples are processed. Use the records to supplement information when reporting results.

11. Keywords

11.1 airborne particulate matter; dry paint film; lead; sample preparation; settled dust; soils; ultrasonic extraction

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