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Designation: E 1645 – 94

An American National Standard

Standard Practice for the Preparation of Dried Paint Samples for Subsequent Lead Analysis by Atomic Sprectrometry[1]

This standard is issued under the fixed designation E 1645; 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 sample preparation procedures for paint samples that are collected during the assessment or abatement of lead hazards in and around buildings and related structures.

1.2 This practice describes the digestion procedures for paint samples that are to be analyzed for lead content.

1.3 This practice covers the general considerations for quantitative sample extraction for total recoverable lead in dried paint samples (either bulk paint or paint powder) using hot plate or microwave heating techniques, or both.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 This practice describes an alternative sample preparation procedure to that given in Test Method D 3335. The procedure described in this practice is a wet digestion method, while that described in Test Method D 3335 employs dry ashing. Also, unlike the procedure outlined in Test Method D 3335, this practice includes a microwave digestion procedure for dried paint samples.

1.6 This practice contains notes that are explanatory and not part of the mandatory requirements of the standard.

1.7 This practice is based on two NIOSH Methods, 7082 and 7105, and on EPA standard operating procedure for lead in paint.

1.8 *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.* For specific precautionary statements, see 6.2.4.4 and 7.2.1.1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

D 3335 Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy[3]

E 1605 Terminology Relating to Abatement of Hazards from Lead-Based Paint in Buildings and Related Structures[4]

2.2 *Other Documents:*

Environmental Protection Agency, *Standard Operating Procedures for Lead in Paint by Hotplate- or Microwave-based Acid Digestions and Atomic Absorption or Inductively Coupled Plasma Emission Spectrometry*; U.S. EPA, Research Triangle Park, NC (1991).[5]

NIOSH Manual of Analytical Methods, P.M. Eller, Ed., 3rd ed., Methods 7082 and 7300; National Institute for Occupational Safety & Health, Cincinnati, OH (1984).[5]

3. Terminology

3.1 *Definitions*—For definitions of terms relating to the preparation of dried paint samples that are not given here, refer to Terminology D 1129, Terminology E 1605, or Test Method D 3335.

3.1.1 *batch*—a group of field or quality control samples that are processed together using the same reagents and equipment.

3.1.2 *digestate*—an acidified aqueous solution that results from digestion of the sample.

3.1.3 *digestion*—the sample preparation process that solubilizes (extracts) targeted analytes present in the sample, and results in an acidified aqueous solution called the digestate.

3.1.4 *extraction*—the dissolution of target analytes from a solid source matrix into a liquid form. During sample digestion, target analytes are extracted (solubilized) into an acid solution.

3.1.5 *method blank*—a sample, devoid of analyte, that is analyzed to determine its contribution to the total blank (background) reading.

3.1.6 *non-spiked sample*—a sample, devoid of analyte, that is targeted for addition of analyte but is not fortified with all target analytes prior to sample preparation.

3.1.6.1 *Discussion*—Analysis results for this sample are used to correct for background levels in the blank medium that is used for spiked and spiked duplicate samples.

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[1] This practice is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.23 on Lead Paint Abatement.

Current edition approved Dec. 15, 1994. Published February 1995.

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

[3] *Annual Book of ASTM Standards*, Vol 06.01.

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

[5] Available from National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22161.

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3.1.7 *reagent blank*—a digestate that reflects the maximum treatment given any one sample within a batch of samples, except that it has no sample placed initially into the digestion vessel. (The same reagents and processing conditions that are applied to field samples within a batch are also applied to the reagent blank.)

3.1.7.1 *Discussion*—Analysis results from this sample provide information on the level of potential contamination resulting from only laboratory sources that are experienced by samples processed within the batch.

3.1.8 *reference material (standard reference material) (SRM)*—a material of known composition where the lead level is certified by the manufacturer.

3.1.9 *sample set*—a group of samples (one or more).

3.1.10 *spiked sample or spiked duplicate sample*—a blank medium that contains no purposely added analyte to which a known amount of analyte is added before preparation.

3.1.10.1 *Discussion*—Analysis results for these samples are used to provide information on the precision and accuracy of the overall process.

4. Summary of Practice

4.1 Lead in dried paint samples (chips, powder, etc.) is solubilized (extracted) by digestion with nitric acid and hydrogen peroxide facilitated by heat, or by a mixture of nitric acid and hydrochloric acid facilitated by microwave energy. The lead content of the digested sample is then in a form ready for measurement by atomic spectrometric methods.

5. Significance and Use

5.1 Paint in buildings and related structures needs to be monitored for lead content in order to determine the potential lead hazard. Hence, effective and efficient methods are required for the preparation of paint samples that may contain lead.

5.2 This practice is to be used for the digestion of paint samples that are collected during various construction and renovation activities associated with lead abatement in and around buildings and related structures. This practice may be used for the preparation of paint samples collected in other environments as well.

5.3 This practice may be used to prepare samples that have been obtained in order to ensure compliance with federal laws that govern lead content in paints.

5.4 This practice may be used to prepare samples that have been collected for risk assessment purposes.

5.5 This practice is intended for use with paint samples that are prepared for subsequent analysis by laboratory-based quantitative analytical methods.

6. Apparatus

6.1 *Heating Equipment*:

6.1.1 *Electric Hot Plate*—suitable for operation at surface temperatures up to at least 140°C. A temperature of at least 100°C, as measured by a thermometer placed inside a borosilicate glass container (on the hot plate) filled with digestion solution, should be attainable. (See Note 1.)

NOTE 1—Provided that the hot plate is capable of handling the extra heating required, use of a 12 to 25-mm (approximately 0.5 to 1-in.) thick aluminum plate placed on the burner head can help reduce the presence of hot spots common to electric hot plates.

6.1.2 (Alternatively) *Microwave Digestion System*, nominal 550 W power minimum, with turntable, 120-mL TFE-fluorocarbon-lined vessels and capping station. The power available for heating must be evaluated weekly in order to ensure that the microwave unit has not degraded, and to compare absolute power settings for different microwave digestion apparatuses. (See Annex A1 for information on evaluation and calibration of microwave power level.)

6.2 *Reagents, Glassware and Supplies*:

6.2.1 *Apparatus-Hot Plate Digestion*:

6.2.1.1 *Borosilicate glass beakers*, 125-mL or 50-mL with watchglass covers,

6.2.1.2 *Class A borosilicate volumetric flasks*, 100 mL and 200 mL,

6.2.1.3 *Class A borosilicate volumetric pipets*, volume as needed,

6.2.1.4 *Linear polyethylene bottles with caps*, 100 mL,

6.2.1.5 *Analytical balance*, accurate to ±0.0001 g,

6.2.1.6 *Glass funnels*, and

6.2.1.7 *Filter paper*.

6.2.2 *Apparatus-Microwave Digestion*:

6.2.2.1 *Centrifuge*, with 30 mL polysulfone centrifuge tubes and polypropylene screw closure,

6.2.2.2 *Class A volumetric and graduated pipets*,

6.2.2.3 *Mechanical shaker*, and

6.2.2.4 *Analytical balance*, accurate to ±0.0001 g.

6.2.3 *Reagents-Hot Plate Digestion*:

6.2.3.1 *Concentrated nitric acid*, ACS reagent grade or spectrographic grade 16.0 M HNO_3,

6.2.3.2 *Nitric acid*, 10 % (w/v): Add 100 mL concentrated HNO_3 to 500 mL ASTM Type I or Type II water (see Specification D 1193). Dilute to 1 L with ASTM Type I or Type II water,

6.2.3.3 *Hydrogen peroxide*, 30 % H_2O_2 (w/w); ACS reagent grade, and

6.2.3.4 *ASTM Type I or Type II water* (see Specification D 1193).

6.2.4 *Reagents-Microwave Digestion*:

6.2.4.1 *Concentrated nitric acid*, ACS reagent grade or spectrographic grade 16.0 M HNO_3,

6.2.4.2 *Concentrated hydrochloric acid*, ACS reagent grade 12.3 M HCl,

6.2.4.3 *ASTM Type I or Type II water* (see Specification D 1193), and

6.2.4.4 *Extraction Solution*—In a 1-L volumetric flask, combine the following in order and mix well: 500 mL ASTM Type I or Type II water, 60 mL concentrated HNO_3 and 180 mL concentrated HCl. Cool to room temperature and dilute to 1 L with ASTM Type I or Type II water. **Caution:** Nitric and hydrochloric acid fumes are toxic. Prepare in a well-ventilated fume hood.

7. Sample Treatment

7.1 *Sample Preparation*:

7.1.1 *Sample Mass and Area*—After analysis, report the final results in area concentration (mg Pb/cm^2) or mass concentration (ppm Pb, percent Pb by mass, or alternative units). If area concentration is desired, sample areas must be

provided (by the person submitting the samples) for each paint sample (chip, powder, etc.). The mass of area concentration samples must be determined and processed in total; mass concentration samples may be subsampled (after grinding and homogenization), depending on the sample mass.

7.1.2 *Area Samples*—For each field sample, homogenize the dried paint sample (inside the original sample container, if possible) as described in the following:

7.1.2.1 Don a new clean pair of vinyl gloves to perform sample handling.

7.1.2.2 Remove any large amounts of substrate present in the sample. Exercise care when removing substrate to avoid any losses of paint. If required, use a clean safety razor blade or equivalent tool to aid in substrate removal.

7.1.2.3 *Determination of Total Collected Sample Mass*—This determination is most accurate when the sample has been collected in a rigid-walled sampling container. Determinations of total sample mass for samples collected in plastic bags are less accurate due to difficulties with complete removal of the sample from the container. However, procedures for this determination for samples collected in both these types of containers is presented because of limits in laboratory control over field collection activities. Use the following procedure to determine total collected sample mass for samples collected in rigid walled containers:

(1) Label the original sample container cap to match that of the container body using indelible ink and allow the ink to dry. This is performed to avoid errors in determing mass resulting from a mismatch of caps to container bodies.

(2) Determine the mass of the area concentration sample in total (sample plus original container with cap) to the nearest 0.1 mg. This mass is the total container plus sample mass. This is performed while wearing clean vinyl gloves prior to homogenization (grinding).

(3) Transfer the collected paint sample into a clean, labeled container such as a beaker or plastic centrifuge tube. Transfer as much of the paint sample as possible to the container by carefully tapping or by using a clean spatula (or rubber policeman). Any visible traces of paint left in the original container may result in bias of final lead analysis results. In general, most losses caused by fine powder remaining in the original container will not result in any significant bias (particularly with respect to the large sampling variability that normally accompanies the field collection practices). However, any visible material that can not be transferred should be documented in sample preparation records.

(4) Rinse out the inside surface of original sample container and cap with ASTM Type I or Type II water. Set the original sample container and cap aside and allow them to dry at room temperature.

(5) After the original sample container has dried completely, re-determine the mass of the empty container with cap. This mass is the empty container mass.

(6) Determine the total sample mass by subtracting the empty container from the total container plus sample mass.

Use the following procedure to determine total collected sample mass for samples collected in plastic bags:

(1) Determine the mass of the area concentration sample in total (sample plus plastic bag) to the nearest 0.1 mg. This mass is the total container plus sample mass. This is performed while wearing clean vinyl gloves prior to homogenization (grinding).

(2) Transfer the collected paint sample into a clean, labeled container such as a beaker or plastic centrifuge tube. Transfer as much of the paint sample as possible to the container by carefully tapping or by using a clean spatula (or rubber policeman). Any visible traces of paint left in the original container may result in bias of final lead analysis results. In general, most losses caused by fine powder remaining in the original container will not result in any significant bias (particularly with respect to the large sampling variability that normally accompanies the field collection practices). However, any visible material that can not be transferred should be documented in sample preparation records.

(3) Re-determine the mass of the empty plastic bag. This mass is the empty container mass.

(4) Determine the total sample mass by subtracting the empty container mass from the total container plus sample mass.

7.1.2.4 *Homogenization of Samples*—Breakup the paint sample into small pieces using a clean sharp blade, or by crushing with a clean plastic or glass rod. Sample should be broken down to a fine powder. Use of a mortar and pestle or other grinding device may be needed to achieve a fine powder. Other techniques, for example, the use of dry ice to assist in the breakup of the sample, may also be employed.

7.1.2.5 *Hot Plate Digestions*—Determine the mass, to the nearest 0.1 mg, of a 0.25 to 0.50 g subsample of the homogenized sample into a clean, labeled 125-mL or 50-mL beaker.

7.1.2.6 *Microwave Digestions*—Determine the mass, to the nearest 0.1 mg, of a 0.1 to 0.2 g subsample of the homogenized sample into a clean, labeled 30-mL polysulfone centrifuge tube.

7.1.3 *Mass Samples*—For each field sample, perform the homogenization and subsample mass determining steps using the same general procedure described for the area samples (7.1.2.1, 7.1.2.2, 7.1.2.4, 7.1.2.5 and 7.1.2.6). If possible, perform the homogenization in the original sample container. If not, include the performance of the transfer part of the total mass determination procedure described above; for samples collected in rigid walled containers or for samples collected in plastic bags.

7.2 *Sample Extraction*:

7.2.1 *Hot Plate Extraction*—For each sample in a beaker having a known mass, plus any quality control samples, perform HNO/H_2O_2 hot plate extraction as described below. **Caution:** nitric acid fumes are toxic; perform the following operations in a fume hood.

7.2.1.1 Add 3 mL concentrated HNO_3 and 1 mL 30 % H_2O_2, and cover with a watch glass. Heat on a hot plate (surface temperature approximately 140°C; 85 to 100°C initially) until most of the acid has evaporated (see Note 2). Remove the beaker containing sample from the hotplate and allow it to cool to room temperature.

NOTE 2—Initial hotplate surface temperature should be between 85 and 100°C to prevent spattering of the solution.

7.2.1.2 Repeat step 7.2.1.1 two more times using 2 mL concentrated HNO_3 and 1 mL 30 % H_2O_2. Heat (surface temperature approximately 140°C) until the sample is nearly dry (see Note 3).

NOTE 3—Evaporate gently to dryness or near dryness; to avoid potential sample losses caused by spattering, some solution should be left in the digestion vessels.

7.2.1.3 Rinse the watch glass and beaker walls with 3 to 5 mL 10 % HNO_3, and allow the solution to evaporate gently to dryness (surface temperature approximately 140°C). Cool to near room temperature.

7.2.1.4 Add 1 mL concentrated HNO_3 to the residue; swirl to dissolve soluble species.

7.2.1.5 Rinse the beaker walls and bottom of the watch glass with ASTM Type I water, and quantitatively transfer to a 100 mL volumetric flask. Dilute to volume with ASTM Type I water.

7.2.1.6 Remove any particulate in the digestate by filtration, by centrifugation, or by allowing the sample to settle prior to instrumental measurement. The diluted digestate solution contains approximately 1 % (v/v) nitric acid. Calibration standards used for instrumental measurement should be made with this level of nitric acid.

7.2.2 *Microwave Extraction*—For each sample in a microwave digestion vessel having a known mass, plus any quality control samples, perform HNO_3/HCl microwave extraction as described as follows:

7.2.2.1 Add 10 mL of extraction solution (6.2.4.4) using a Class A volumetric pipet. Cap the tube tightly.

7.2.2.2 Pipet 31 mL of ASTM Type I or Type II water into a 120 mL TFE-fluorocarbon microwave digestion vessel. Place a centrifuge tube containing the sample in the digestion vessel. Place a safety valve and cap on the vessel and tighten the cap (see Note 4).

NOTE 4—Capping stations may be used to tighten vessel caps to the proper torque.

7.2.2.3 Fill the microwave turntable with vessels containing centrifuge tubes. Follow manufacturer's instructions on loading of sample vessels into the microwave unit. Place the turntable in the microwave oven and activate the oven and turntable. Set the exhaust fan to maximum flow rate. Program the microwave oven for a time of 23 min. and a power of 522 W before initiating the digestion.

7.2.2.4 At the end of the microwave heating program, remove the turntable containing the microwave vessels and allow to cool to room temperature.

7.2.2.5 Open the microwave vessels and discard the water they contain. Open the centrifuge tubes and add 10 mL of ASTM Type I water using a Class A volumetric pipet. Cap the tubes tightly, and mechanically shake for 5 min.

7.2.2.6 Centrifuge the digestates at 2000 RPM for 25 min. Open the centrifuge tubes and decant or pipet off the clear solution for instrumental measurement. Use a final sample dilution volume of 20 mL to calculate analytical results. The sample is ca. 1.03 M in HCl and 0.45 M in HNO_3. Calibration standards used for instrumental measurement should be made with this level of acid.

7.3 *Supplemental Information—Lead Result Calculations for Area Samples*:

7.3.1 Instrumental measurements for lead in the digestates are converted to final area results using a ratio of the total collected sample mass to the digested subsample mass. These masses were generated using the total sample mass determination procedures described in 7.1.2.3. An example of the final results calculation is as follows:

$$\text{mg of lead per cm}^2 = [(A)(B)(C)][D/E)]/[F)] \quad (1)$$

where:

A = measured lead in sample digest, mg/mL,
B = final digestion volume, mL,
C = additional dilution factors from instrumental measurement, mL/mL,
D = total collected sample mass, g,
E = mass of sample digestion for lead measurement, g, and
F = area of collect sample, cm^2.

8. Quality Assurance

8.1 *Quality Control Samples*—Quality control (QC) samples to process with each batch of samples are summarized in Table 1.

8.1.1 *Reagent blanks*—Carry reagent blanks (water and reagents) throughout the entire sample preparation and analytical process to determine if the samples are being contaminated from laboratory activities. Process reagent blanks according to the frequency listed in Table 1.

8.1.2 *Non-spiked samples, spiked samples, and spiked duplicate samples*—Process these samples on a routine basis to estimate the method accuracy on the sample batch, expressed as a percent recovery relative to the true spiked value. Since paint samples cannot be easily split uniformly, method blanks are used for non-spike samples; spiked and spiked duplicates would consist of method blanks to which known amounts of

TABLE 1 Quality Control Samples

QC Samples	Definition	Frequency
Method blank or non-spiked sample	A sample known to contain no lead. This sample is carried through sample preparation along with other samples. It should reflect the maximum treatment given any one sample within the batch.	1 per 20 samples, minimum of 1 per batch.
Reagent blank	Type I or Type II water—digest as sample with addition of all reagents. It should reflect the maximum treatment given any one sample within the batch.	1 per batch
Spiked sample	A sample containing no lead which is spiked with lead before preparation.	1 per 20 samples, minimum of 1 per batch.
Spiked duplicate	A blank sample (containing no lead) which is fortified with lead before preparation.	1 per 20 samples, minimum of 1 per batch.
Reference material (standard reference material)	A material of known composition where the lead levels are certified by the manufacturer.	1 per batch.

analyte are added. Run these QC samples at the frequency listed in Table 1.

8.1.3 *Standard reference materials (SRMs)*—Process certified standard reference materials on a routine basis to determine an estimate of method accuracy on the sample batch, expressed as a percent recovery relative to the certified value. Incorporate SRMs into each analytical batch according to the frequency listed in Table 1. Use an SRM that has a matrix which is similar to or identical to paint with a certified lead concentration level. Process a known amount of SRM along with other samples.

8.2 *Laboratory Records*—Record all information regarding the preparation of samples (both QC samples and those submitted to the analyst) as follows:

8.2.1 Record all reagent sources (lot numbers) used for sample preparation in a laboratory notebook. Include the date(s) and identification and signature(s) of the person(s) making all entries. Record any inadvertent deviations, unusual occurrences, or observations on a real-time basis as samples are processed. Use the records to add supplemental information when reporting results.

8.2.2 Laboratory notebooks must be bound with prenumbered pages, and all entries must be made in ink. Any entry errors must be corrected by using only a single line through the incorrect entry, accompanied by the initials of the person making the correction, and the date of the correction.

9. Keywords

9.1 hot plate; lead; microwave; paint; sample preparation

ANNEX

(Mandatory Information)

A1. MICROWAVE POWER CHECK

A1.1 Procedure

A1.1.1 Remove the turntable, drive lug, and all vessels from the instrument cavity.

A1.1.2 Adjust the instrument cavity exhaust to minimum air flow.

A1.1.3 Program the instrument for 4 min at 100 % power.

A1.1.4 Transfer 2000 mL ± 2 mL of room temperature (19 to 25°C) water into a 2-L polypropylene beaker.

A1.1.5 Measure and record the initial water temperature (T_i) to the nearest 0.1°C.

A1.1.6 Place the beaker in the right front corner of the instrument cavity (as you face the front of the instrument).

A1.1.7 Heat the water for the programmed time.

A1.1.8 When the heating cycle is complete, immediately remove the beaker from the cavity, thoroughly stir the water to ensure even heat distribution, and measure the final temperature (T_f) to the nearest 0.1°C.

A1.1.9 Calculate the delivered power according to the following equation:

$$\text{Power, W} = \Delta T \times [K \times C_p \times M)/t] \quad \text{(A1.1)}$$

where:

ΔT = $T_f - T_i$, where:
T_f = final water temperature,° C, and
T_i = inital water temperature, °C,
K = 4.2, the conversion factor for thermochemical calories to Watts,
C_p = 1.0, the heat capacity for water in $cal \cdot g^{-1} \cdot deg^{-1}$,
M = mass of water, g, and
t = time, s.

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