



Standard Practice for the Preparation of Dried Paint Samples for Subsequent Lead Analysis by Atomic Spectrometry¹

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 Water²

D 1193 Specification for Reagent Water²

D 3335 Test Method for Low Concentrations of Lead,

Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy³

E 1605 Terminology Relating to Abatement of Hazards from Lead-Based Paint in Buildings and Related Structures⁴

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.

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.

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 HNO₃,

6.2.3.2 *Nitric acid*, 10 % (w/v): Add 100 mL concentrated HNO₃ 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 % H₂O₂ (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 HNO₃,

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 HNO₃ 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/cm²) or mass concentration (ppm Pb, percent Pb by mass, or alternative units). If area concentration is desired, sample areas must be