



Designation: E 1645 – 01

Standard Practice for Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis¹

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, management or control of lead hazards.

1.2 This practice describes the digestion procedures using a hot plate or microwave oven or apparatus 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 contains notes that are explanatory and not part of the mandatory requirements of the standard.

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

1.7 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.

2. Referenced Documents

2.1 ASTM Standards:

D 1129 Terminology Relating to Water²

D 1193 Specification for Reagent Water²

E 1605 Terminology Relating to Lead in Buildings³

E 1729 Practice for Field Collection of Dried Paint Samples for Lead Determination by Atomic Spectrometry Techniques³

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). (NTIS No. PB92-114172)⁴

NIOSH Manual of Analytical Methods, P.M. Eller and M.E. Cassinelli, Eds., 4th ed., Methods 7082 and 7105; National Institute for Occupational Safety & Health, Cincinnati, OH (1994).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, or Terminology E 1605.

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 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.

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.)

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

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

³ *Annual Book of ASTM Standards*, Vol 04.11.

⁴ Available from National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22161.

⁵ Available from NIOSH Publications, 4676 Columbia Parkway, Cincinnati, OH 45226; (800)35-NIOSH.

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 (certified reference material) (CRM)*—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. (It is assumed that the paint samples were collected in accordance with Practice E 1729; however, this practice can be used for any collected paint sample.) The lead content of the digested sample is then in a form ready for measurement.

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 may be used for the digestion of paint samples that are collected during various lead-hazard control and risk assessment activities associated with lead abatement in and around buildings and related structures. This practice is also suitable for the digestion of paint samples collected from locations such as commercial buildings.

5.3 This practice may be used to prepare samples that have been obtained in order to ensure compliance with 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 Microwave Extraction Apparatus

Caution: Ensure that manufacturer's safety recommendations are followed.

NOTE 2—The procedure described is for microwave digestion systems with a temperature control system. Microwave digestion systems that are equipped only with a pressure control system or lower pressure vessels, or both, may be used, provided that a prior assessment of the dissolution efficiency is carried out.

6.1.2.1 *Microwave Digestion System*—designed for closed vessel digestion, with power output regulation, fitted with a temperature control system capable of sensing the temperature to within $\pm 2^\circ\text{C}$, and automatically adjusting the microwave power output within 2 s. The microwave cavity shall be resistant to chemical attack, and equipped with exhaust ventilation for acid vapor protection of the unit and operator. All electronics shall be protected against corrosion to ensure safe operation. Safety interlocks, to shut off magnetron power output, shall be contained within the oven door opening mechanism.

Caution: Domestic (kitchen) microwave ovens shall not be used, since there are very significant hazards associated with their use for the procedure described in this standard. For example, acid vapors released into the cavity can corrode safety devices that prevent the magnetron from shutting off when the door is opened, potentially exposing the operator to microwave energy. Also, the fumes generated can be extremely hazardous.

NOTE 3—A pressure control system is also very useful, since it provides a safeguard against the possibility of sample loss due to excessive pressure buildup and partial venting of the sample vessels.

6.1.2.2 *Lined Sample Vessels*—closed, designed for carrying out microwave digestions, capable of withstanding a temperature of at least 180°C and with an internal volume of at least 50 mL. The vessels must be transparent to microwave energy, and vessel liners shall be chemically inert. The vessels must be capable of withstanding high internal pressures (up to at least 3000 kPa) and temperatures (up to at least 180°C). Vessels shall also be equipped with a safety relief valve or disc that will prevent vessel rupture or ejection of the vessel cap. Such vessels consist of an inner liner and cover made of a microwave transparent and chemically resistant material (usually a fluorocarbon polymer such as tetra-fluoromethoxil polymer (TFM), which contains and isolates the sample solution from a high strength, outer pressure structure. Other types of sample vessels designed to operate at equivalent or higher temperatures or pressures, or both, may be used.

Caution: For closed vessel designs, the material from which the outer vessels are made is usually not as chemically inert as the liner material. Since the outer vessels provide the strength required to withstand the high pressures within the inner liners, they must be inspected regularly to check for any chemical or physical degradation.

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,