Designation: E1645 – 21

Standard Practice for Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis[1]

This standard is issued under the fixed designation E1645; 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 This practice contains notes that are explanatory and not part of the mandatory requirements of the standard.

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

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

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 6.1.2, 6.1.2.1, 6.1.2.2, 6.3.2.4, 8.2.1, and 8.2.2.

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D1356 Terminology Relating to Sampling and Analysis of Atmospheres
E288 Specification for Laboratory Glass Volumetric Flasks
E969 Specification for Glass Volumetric (Transfer) Pipets
E1605 Terminology Relating to Lead in Buildings
E1729 Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination

2.2 *ISO Documents:*[3]

ISO Guide 30 Reference materials — Selected terms and definitions
ISO 1042 Laboratory glassware — One-mark volumetric flasks
ISO 8655 Piston-operated volumetric apparatus — Part 1: Terminology, general requirements and user recommendations

2.3 *Other Documents:*

NTIS No. PB92–114172 Standard Operating Procedures for Lead in Paint by Hotplate- or Microwave-based Acid Digestions and Atomic Absorption or Inductively Coupled Plasma Emission Spectrometry[4]
NMAM 7082 and 7105 NIOSH Manual of Analytical Methods, 4th ed.[5]

3. Terminology

3.1 *Definitions*—For definitions of terms relating to the preparation of dried paint samples that are not given here, refer

[1] This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.12 on Sampling and Analysis of Lead for Exposure and Risk Assessment.

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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.

[3] Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

[4] U.S. EPA, Research Triangle Park, NC (1991). Available from National Technical Information Service (NTIS), 5301 Shawnee Rd., Alexandria, VA 22312, http://www.ntis.gov.

[5] P.M. Eller, and M.E. Cassinelli, Eds., National Institute for Occupational Safety & Health, Cincinnati, OH (1994). Available from NIOSH Publications, 1150 Tusculum Ave, Cincinnati, OH 45226, Mail Stop C14, http://www.cdc.gov/niosh.

to Terminologies D1129, D1356, or E1605.

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

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

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

3.1.6 *non-spiked sample, n*—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, n*—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 (certified reference material) (CRM), n*—reference material accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realization of the unit in which the property values are expressed; each certified value is accomplished by an uncertainty at a stated level of confidence. **ISO Guide 30**

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

3.1.10 *spiked sample or spiked duplicate sample, n*—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, and so forth) 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 E1729; 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 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:*

Warning—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 ±2 °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.

Warning—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.

Warning—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 *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 *Borosilicate volumetric flasks* meeting Specification E288 or conforming to ISO 1042, 100-mL and 200-mL,

6.2.1.3 *Borosilicate volumetric pipets* meeting Specification E969 or piston-operated pipets conforming to ISO 8655, volume as needed,

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

6.2.1.5 *Analytical balance,* accurate to ±0.1 mg,

6.2.1.6 *Glass funnels,*

6.2.1.7 *Filter paper,* and

6.2.1.8 *Weighing Paper or Weighing Boat.*

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 *Borosilicate volumetric pipets* meeting Specification E969 or piston-operated pipets conforming to ISO 8655, volume as needed,

6.2.2.3 *Mechanical shaker,* and

6.2.2.4 *Analytical balance,* accurate to ±0.1 mg.

6.2.3 *Mortar and pestle* (porcelain or agate), shatter box, or mixer mill.

6.2.4 *Plastic gloves,* powderless.

6.2.5 *Thermometers,* red alcohol, that cover a range from 0 °C to 110 °C.

6.3 *Reagents:*

6.3.1 *Reagents-Hot Plate Digestion:*

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

6.3.1.2 *Nitric acid,* mass fraction 10 %: Add 100 mL concentrated HNO_3 to 500 mL ASTM Type I water (see Water, 6.3.1.4. References to ASTM Type I water shall mean reagent water as defined by Type I of Specification D1193). (ASTM Type I water: minimum resistivity of 16.7 megohm·cm, or equivalent.) Dilute to 1 L with ASTM Type I water,

6.3.1.3 *Hydrogen peroxide,* mass fraction 30 % H_2O_2; ACS reagent grade, and

6.3.1.4 *ASTM Type I water.*

6.3.2 *Reagents-Microwave Digestion:*

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

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

6.3.2.3 *ASTM Type I water*, and

6.3.2.4 *Extraction Solution*—In a 1-L volumetric flask, combine the following in order and mix well: 500 mL ASTM Type I water, 60 mL concentrated HNO_3 and 180 mL concentrated HCl. Allow to cool to room temperature and dilute to 1 L with ASTM Type I water. (**Warning—**Nitric and hydrochloric acid mists and vapors are toxic. Prepare in a well-ventilated fume hood.)

7. Equipment Preparation

7.1 Wash glassware and plastic equipment with laboratory detergent, rinse with tap water, soak for at least 4 hours in volume fraction 35 % nitric acid and water, rinse three times with ASTM Type I Water, and allow to dry preferably in a fume hood. Commercial, automatic systems are available that perform a similar process.

7.2 Alternatively, soak glassware and plastic equipment in volume/volume 1+1 nitric acid and water in a plastic tub preferably in a working hood with the hood sash down, rinse three times with ASTM Type I Water, and allow to dry preferably in a fume hood.

8. Sample Treatment

8.1 *Sample Preparation:*

8.1.1 *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, and so forth). The total mass of area concentration samples must be determined. Samples may be subsampled (after grinding and homogenization), depending on the sample mass.

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

8.1.2.1 Don a new clean pair of plastic gloves to perform sample handling.

8.1.2.2 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.

8.1.2.3 *Determination of Total Collected Sample Mass*—Accurate determination of the collected sample mass is required to report lead analysis results in terms of area concentration (mass per unit area of paint sample). A complete transfer of the sample is required to whatever preweighed container is used to hold the sample during mass determination (for example, weighing boat or weighing paper). Total mass shall be made to the nearest 0.1 mg.

The following precautions shall be observed during determination of total mass:

(1) Total sample mass can be determined either before or after sample homogenization. Determination of total sample mass is generally advisable prior to homogenization when samples consist of large intact chips that can be easily transferred without incurring losses. Determination of total sample mass is generally advisable after homogenization when samples can be homogenized in the original sample collection container and the samples are not large intact chips.

NOTE 4—In this case, the sample container should be weighed after homogenization.

(2) Any visible traces of paint left in the original container or container used for homogenization (if different from original container) may result in bias of the final lead analysis results. Therefore, such traces shall be minimized. Any visible material that cannot be transferred shall be documented in sample preparation records.

(3) For sample transfers following homogenization, most losses caused by the presence of fine powder remaining in the original container or container used for homogenization (if different from original container) will not result in any significant bias (particularly with respect to the large sampling variability that normally accompanies the field collection practice.)

(4) For sample transfers prior to homogenization (that is, when homogenization cannot be performed in the original container used in sample collection), any losses caused by fine powder remaining in the original container may result in a significant bias. Therefore, sample transfers conducted prior to sample homogenization shall be performed with extra attention to avoiding visible traces of paint left in the original container.

NOTE 5—If sample mass is determined after homogenization in the collection container, the container should be weighed (clean) either before sampling, or after sample homogenization, reweighing (immediately following sample transfer), and recleaning.

8.1.2.4 *Homogenization of Samples*—Samples shall be homogenized as finely as possible, regardless of whether area concentration or mass concentration results are desired. The homogenization of the sample serves two purposes: (*a*) to ensure that the subsamples will be representative of the whole collected sample; and (*b*) to maximize the extraction and digestion efficiency of the sample. Any sample homogenization technique that meets the following criteria may be used:

(1) Samples shall be ground, crushed or broken into a fine powder or small granules consisting of particles no larger than that visually represented by the size of a poppy seed or small grain of sand (no larger than 0.5 mm in diameter).

(2) Samples shall not be contaminated from any other previously processed sample. This means that the sample homogenization technique is carried out such that careful cleaning between samples is performed on the equipment used to process multiple samples.

NOTE 6—Sample homogenization techniques that employ cold temperatures, such as dry ice-assisted grinding or liquid nitrogen shatter box mills, can be extremely effective in homogenizing paint samples, and are recommended, but not required. Such homogenization techniques can be used in lieu of or in addition to the use of a mortar and pestle or other grinding technique.

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

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

8.1.3 *Mass Samples*—For each field sample, perform the homogenization, subsampling, and mass determining steps using the same general procedure described for the area samples (8.1.2). If possible, perform the homogenization in the original sample container. If not, perform homogenization on samples which are quantitatively transferred to a suitable container for homogenization.

8.2 *Sample Extraction:*

8.2.1 *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. (**Warning—**Nitric acid mists and vapors are toxic; perform the following operations in a fume hood.)

8.2.1.1 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 °C to 100 °C initially) until most of the acid has evaporated (see Note 7), leaving less than 1 mL in the bottom of the beaker. Remove the beaker from the hotplate and allow it to cool to room temperature.

NOTE 7—Cross-contamination or sample loss could occur with boiling or splashing of the digestate.

8.2.1.2 Repeat step 8.2.1.1 two more times using 3 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 8). Evaporate to near dryness.

NOTE 8—Potential sample loss caused by spattering is reduced when a small amount of solution is left in the digestion vessel.

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

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

8.2.1.5 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.