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An American National Standard

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 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 Water[2]

D 1193 Specification for Reagent Water[2]

E 1605 Terminology Relating to Lead in Buildings[3]

E 1729 Practice for Field Collection of Dried Paint Samples for Lead Determination by Atomic Spectrometry Techniques [3]

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).[4] (NTIS No. PB92–114172)

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.)

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[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 ±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.

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,

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

6.2.1.6 *Glass funnels*, and

6.2.1.7 *Filter paper*,

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 *Class A volumetric and graduated pipets*,

6.2.2.3 *Mechanical shaker*, and

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

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 water (see Specification D 1193). Dilute to 1 L with ASTM Type I water,

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

6.2.3.4 *ASTM Type I 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 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 water, 60 mL concentrated HNO_3 and 180 mL concentrated HCl. Cool to room temperature and dilute to 1 L with ASTM Type I 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 total mass of area concentration samples must be determined. 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*—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.

7.1.2.4 *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 the 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.

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 and place it 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 and place it into a clean, labeled 30-mL polysulfone centrifuge tube.

7.1.3 *Mass Samples*—For each field sample, perform the homogenization, subsampling, and mass determining steps using the same general procedure described for the area samples (7.1.2). 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.

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 7). Remove the beaker containing sample from the hotplate and allow it to cool to room temperature.

NOTE 7—Initial hotplate surface temperature should be between 85 and 100°C to prevent spattering of the solution. To eliminate the possibility of cross-contamination or sample loss, avoid boiling or splashing of the digestate.

7.2.1.2 Repeat step 7.2.1.1 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).

NOTE 8—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 having a known mass, plus any quality control samples, perform microwave extraction as described below. **Caution:** Nitric acid fumes are toxic; perform the following operations in an appropriately ventilated area.

7.2.2.1 Transfer each sample into the clean liner of a labeled microwave digestion vessel.

7.2.2.2 Carefully add 5 mL of concentrated nitric acid or extraction solution (6.2.4.4) to the inside liner of the digestion vessel containing the sample or blank. Seal the vessels.

7.2.2.3 Load the vessels into the microwave oven in accordance to manufacturer's instructions. Vessels containing samples shall be evenly and symmetrically placed in the microwave oven.

NOTE 9—Even, symmetrical spacing of vessels is desired to ensure uniform microwave heating of all vessel solutions.

7.2.2.4 Program the microwave digestion system to reach at 180°C (±5°C) in less than 10 min, and then hold at this temperature for at least 15 min.

7.2.2.5 At the end of the digestion period, remove the vessels from the microwave oven, place them in a fume hood, and allow the solutions to cool to room temperature.

7.2.2.6 Carefully detach the vent tubing, and mechanically shake the vessels to vent any excess gas pressure that may be present inside the vessels.

NOTE 10—Filtration of digestates can be performed in lieu of centrifugation.

7.2.2.7 Carefully open the vessels. Quantitatively transfer the liquid contents of each vessel to 10–mL one-mark volumetric flasks. Carefully rinse each vessel with ASTM Type I water and bring to volume in the 10–mL volumetric flask with water. Seal each flask with a stopper and mix thoroughly.

7.3 *Supplemental Information—Lead Result Calculations for Area Concentration 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. 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 digested for lead measurement, g, and
F = area of collected sample, cm^2.

8. Quality Assurance

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

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 below.

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 analyte are added. Run these QC samples at the frequency of 1 per 20 samples or minimum of 1 per batch.

8.1.3 *Certified reference materials (CRMs)*—Process certified 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 CRMs into each analytical batch according to a frequency of 1 per 20 samples or mimimum of 1 per batch. Use a CRM that has a matrix which is similar to or identical to paint with a certified lead concentration level. Process a known amount of CRM 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 with signature and date of entry.

8.2.2 Laboratory notebooks must be bound with prenumbered pages. All entries on sample data forms and laboratory notebooks 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; digestion; extraction