



Standard Practice for the Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis by Atomic Spectrometry¹

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1. Scope

1.1 This practice covers the sample preparation procedures for paint samples that are collected during the assessment, management or abatement control of lead hazards in and around buildings and related structures. 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 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 an 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:*

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

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D 1129 Terminology Relating to Water²

D 1193 Specification for Reagent Water²

~~D 3335 Test Method for Low Concentrations of Lead, Cadmium, and Cobalt~~

~~E 1605 Terminology Relating to Lead in Paint by Atomic Absorption Spectroscopy Buildings~~³

~~E 1605 Terminology Relating to Abatement~~ 1729 Practice for Field Collection of Hazards from Lead-Based Dried Paint in Buildings and Related Structures⁴ 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).⁵⁴ (NTIS No. PB92-114172)

NIOSH Manual of Analytical Methods, P.M. Eller and M.E. Cassinelli, Eds., ~~3rd~~ 4th ed., Methods 7082 and 731005; National Institute for Occupational Safety & Health, Cincinnati, OH (1989).⁵

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

³ *Annual Book of ASTM Standards*, Vol 06.01: 04.11.

~~*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 ~~National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22161;~~ NIOSH Publications, 4676 Columbia Parkway, Cincinnati, OH 45226; (800)35-NIOSH.

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~~. 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 ~~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.

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~~ (certified reference material) (SCRM)—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 by atomic spectrometric methods.~~ 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 ~~is to~~ may be used for the digestion of paint samples that are collected during various ~~construction lead-hazard control and renovation risk assessment~~ activities associated with lead abatement in and around buildings and related structures. This practice may be used is also suitable for the preparation digestion of paint samples collected in other environments from locations such as w commercial buildings.

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 *(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, nominal 550 W—designed for closed vessel digestion, with power minimum, output regulation, fitted with turntable, 120-mL TFE-fluorocarbon-lined vessels a temperature control system capable of sensing the temperature to within $\pm 2^{\circ}\text{C}$, and capping station. The automatically adjusting the microwave power available for heating must output within 2 s. The microwave cavity shall be evaluated weekly in order resistant to chemical attack, and equipped with exhaust ventilation for acid vapor protection of the microwave unit has not degraded, and operator. All electronics shall be protected against corrosion to compare absolute 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 digestion apparatuses. (See Annex A1 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 information on evaluation carrying out microwave digestions, capable of withstanding a temperature of at least 180°C and calibration 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 ± 0.0001 g, ± 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 ± 0.0001 g, ± 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 or Type H water (see Specification D 1193). Dilute to 1 L with ASTM Type I or Type H 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 H 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 H 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 H 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 H 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 ($\text{mg Pb}/\text{cm}^2$) 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 and processed in total; mass concentration samples 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*—~~This—Accurate determination is most accurate when of 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 is required to difficulties with report lead analysis results in terms of area concentration (mass per unit area of paint sample). A complete removal transfer 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 required to determine total collected sample mass for samples collected in rigid-walled containers:~~

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

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

~~(3) Transfer 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 collected paint original sample into a clean, labeled collection container such as a beaker or plastic centrifuge tube. Transfer as much of and the paint sample as possible to samples are not large intact chips.~~

~~NOTE 4—In this case, the sample container by carefully tapping or by using a clean spatula (or rubber policeman): 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 practices). However, any visible material that can not be transferred should be documented in sample preparation records. practice.)~~

~~(4) Rinse out the inside surface of original(4) For 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 transfers 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 (that is, when homogenization cannot be performed in the original container may result used in bias of final lead analysis results. In general, most sample collection), any losses caused by fine powder remaining in the original container will not may result in any a significant bias (particularly bias. Therefore, sample transfers conducted prior to sample homogenization shall be performed with respect extra attention to the large sampling variability that normally accompanies the field collection practices). However, any avoiding visible material that can not be transferred should be documented traces of paint left in sample preparation records:~~

~~(3) Re-determine the original container.~~

~~NOTE 5—If sample mass of the empty plastic bag. This mass is determined after homogenization in the empty container mass.~~

~~(4) Determine collection container, the total sample mass by subtracting the empty container mass from the total container plus~~

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—Breakup—Samples shall be homogenized as finely as possible, regardless of whether area concentration or mass concentration results are desired. The homogenization of the paint 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 small pieces using a clean sharp blade, fine powder or small granules consisting of particles no larger than that visually represented by crushing with the size of a clean plastic poppy seed or glass rod. Sample should 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 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. 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 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). (7.1.2). 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 perform homogenization on samples collected in rigid walled containers or which are quantitatively transferred to a suitable container for samples collected in plastic bags. 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 HNO₃/H₂O₂ 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 HNO₃ and 1 mL 30 % H₂O₂, 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). 7). Remove the beaker containing sample from the hotplate and allow it to cool to room temperature.

NOTE 27—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 2–3 mL concentrated HNO₃ and 1 mL 30 % H₂O₂. Heat (surface temperature approximately 140°C) until the sample is nearly dry (see Note 3). 8).

NOTE 38—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 % HNO₃, 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 HNO₃ 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 HNO₃/HCl 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) using a Class A volumetric pipet. Cap to the tube tightly.

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

~~NOTE 4—Capping stations may be used. 9—Even, symmetrical spacing of vessels is desired to tighten vessel caps to the proper torque.~~

~~7.2.2.3 Fill the ensure uniform microwave turntable with vessels containing centrifuge tubes. Follow manufacturer's instructions on loading heating of sample vessels into all vessel solutions.~~

~~7.2.2.4 Program the microwave unit. Place the turntable digestion system to reach at 180°C (±5°C) in the microwave oven less than 10 min, and activate the oven and turntable. Set the exhaust fan to maximum flow rate. Program the microwave oven then hold at this temperature for a time of 23 min. and a power of 522 W before initiating at least 15 min.~~

~~7.2.2.5 At the digestion:~~

~~7.2.2.4 At the end of the microwave heating program, digestion period, remove the turntable containing vessels from the microwave vessels oven, place them in a fume hood, and allow the solutions to cool to room temperature.~~

~~7.2.2.5 Open~~

~~7.2.2.6 Carefully detach the microwave vessels vent tubing, and discard mechanically shake the water they contain. Open vessels to vent any excess gas pressure that may be present inside the centrifuge tubes and add 10 mL 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 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 bring to calculate analytical results. The sample is ea. 1.03 M volume in HCl and 0.45 M in HNO₃. Calibration standards used for instrumental measurement should be made the 10-mL volumetric flask with this level of acid. 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 7.1.2.3. An example of the final results calculation is as follows:~~

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

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

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 digested for lead measurement, g, and

F = area of collected sample, cm².

8. Quality Assurance

~~8.1 Quality Control Samples—Quality control (QC) samples to process with each batch of samples are summarized in Table 1. 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 in Table 1. 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 listed in Table 1. of 1 per 20 samples or minimum of 1 per batch.~~

~~8.1.3 Standard Certified reference materials (SCRMs)—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 SCRMs into each analytical batch according to the a frequency listed in Table 1. of 1 per 20 samples or mimimum of 1 per batch. Use an SRM 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 SRM 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 pre-numbered pages, and all 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; ~~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:

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

T_f

where:

- ΔT = $T_f - T_i$, where:
- T_f = final water temperature, °C, and
- T_i = initial 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·g⁻¹·deg⁻¹,
- M = mass of water, g, and
- t = time, s.

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