



**UNITED STATES
CONSUMER PRODUCT SAFETY COMMISSION
DIRECTORATE FOR LABORATORY SCIENCES
DIVISION OF CHEMISTRY**

**Test Method: CPSC-CH-E1003-09.1
Standard Operating Procedure for Determining Lead (Pb) in Paint and Other
Similar Surface Coatings*
February 25, 2011**

This document provides information on the test methodology that is used by the U.S. Consumer Product Safety Commission's (CPSC) Product Testing Laboratory, Chemistry Division (LSC), in the analysis of paint and certain painted products. The methodology is used to determine the total lead content of paint, or a painted surface, on a dry paint basis. This method supersedes all previously published standard operating procedures for lead in paint testing.

CPSC staff has concluded that this test methodology is sufficient to make appropriate determinations concerning lead (Pb) in paint, as defined in 16 C.F.R Part 1303, with a regulatory limit of 0.009% by weight. Adjustments may be necessary to achieve total digestion for certain paints, and should be based on sound chemistry knowledge and appropriate acids for the sample material being analyzed. Screening tests by X-ray fluorescence may sometimes be employed by CPSC staff to determine samples in need of such testing.

Definitions

1. Sample – an individual consumer product or a group of identical consumer products from a batch to be tested
2. Component Part – individual sub-unit within the total sample. Each separate paint color on a sample is a component part.
3. Composite Testing-like parts – combining like paints from several like parts or products to obtain sufficient sample size for analysis when there is insufficient quantity of paint on one item to perform testing.
4. Composite Testing-different parts – combining different paints (*e.g.*, multiple colors) from one or more samples to reduce the number tested.
5. Instrument Detection Limit (IDL) – 3 times the standard deviation of 10 replicate measurements of reagent blank.
6. Method Detection Limit (MDL) – reagent blank fortified with 2-3 times the IDL. Seven replicate measurements are made. Calculate the MDL as follows: $MDL = t \times S$, $t = 3.14$ (99% confidence level for 7 replicates), S = standard deviation.

* This document was prepared by CPSC staff, has not been reviewed or approved by, and may not necessarily reflect the views of, the Commission.

7. Laboratory Reagent Blank (LRB) – an aliquot of the digestion reagents that is treated exactly as a sample including exposure to glassware, digestion media, apparatus, and conditions used for a particular Pb test, but with no added sample. LRB data are used to assess contamination from the laboratory environment.
8. Calibration Blank – deionized water acidified with nitric acid (3 ml concentrated nitric acid diluted to 100 ml with deionized water).
9. Stock Standard Solution – 1000 µg/ml solution of Pb purchased from reputable commercial source, used to prepare calibration standards. Replace before expiration date.
10. Calibration Standards – solutions containing 0 to 25 µg/ml of Pb in 3% nitric acid matrix are used. A minimum of 4 calibration standards are used. Calibration standards should be prepared weekly.
11. Quality Control Sample (QCS) – a solution containing Pb that is used to evaluate the performance of the instrument system. QCS is obtained from a source external to the laboratory and Stock Standard Solution.
12. Certified Reference Material (CRM) – CRMs are materials with similar matrices as test samples with known lead levels. The CRMs are used to verify digestion and analysis methods. For example, standard reference materials (SRMs) are CRMs that are available from the National Institute of Standards and Technology (NIST), such as those listed in the Equipment and Supplies section below.

Equipment and Supplies: The materials used for sampling and analysis are as follows:

1. Nitric acid, trace metal grade
2. Disposable plastic digestion vessels, 50 ml, or glass test tubes
3. Hot block digester or hot plate with test tube block
4. Disposable razor blade or scalpel
5. Methylene chloride (optional)
6. Distilled water
7. Microwave digestion apparatus
8. CRMs such as NIST SRMs¹ 2581 and 2582 lead in powdered paint. CRMs such as NIST SRM 2569 that are paint films should be used if X-ray fluorescence spectroscopy methods conducted
9. Internal Standard (such as yttrium, from a stock standard solution of that element appropriate to the instrument parameters of the ICP used for the analysis)

Note on composite testing: Composite testing-like parts (as defined above) is appropriate and may be necessary to obtain valid analytical results. Composite testing-different parts (as defined above) must be done with adequate care, planning, and understanding of the limitations and propagations of error in measurements or the test may fail to detect excessive lead in one individual paint because of dilution. If composite testing-different parts is used, great care must be taken to accurately weigh each component paint and account for the propagation of errors due to weighing and measuring, and the analyst must ensure that the compositing will not result in a Pb measurement being below the detection limit for a paint component that would exceed 0.009% Pb.

¹ NIST SRMs 2581 and 2582, and other reference materials, are available from the National Institute of Standards and Technology. See <http://ts.nist.gov/measurementservices/referencematerials/index.cfm>

Each individual paint must be weighed individually with sufficient precision and sufficient safety factors to assure that no false negatives are reported.

Sufficient paint must be used for each of the paints giving proper consideration for the weighing capabilities of the balance used and the detection limits and necessary dilution for the subsequent instrumental analysis. The combined paints could then be digested according to the procedure given below for a single paint, before analysis by Inductively Coupled Plasma (ICP) spectroscopy. Appropriate weights for each of the individual paints that are composited depend on final dilution volumes, weighing accuracy, and detection limits. All of the lead found in the digestate for the combined composite could be attributed to the weight of each of the individual paints as though all of the lead could possibly come from any one of the individual paints. The measured lead should be treated in calculations as if it originated solely from one part of the composite; the lead concentration calculation should be carried out for each part unless identical weights of each sample were used in the analysis. An example is given at the end of this document.

In considering results from such a composite sample, it is imperative that a sufficient “safety factor” be applied to account for weighing inaccuracy and propagation of errors from each step in the analytical procedure to ensure no violative paints are misrepresented as nonviolative. It has been suggested, for example for a composite of three samples, that any sample possibly having greater than 80 percent of the limit for lead in paint in any of the composited paints should be retested²³.

I. Acid Digestion

Method: The digestion method is based on the Association of Official Analytical Chemists (AOAC) standard AOAC 974.02⁴ (Lead in Paint). Alternate microwave digestion based on ASTM E1645⁵ may be used as well. Analysis by Inductively Coupled Plasma (ICP) spectroscopy is based on ASTM E1613⁶.

1. For testing of wet paint, apply a thin coating to a glass slide, and dry completely prior to testing by heating in an oven at nominally 105 °C (105 °C + 2 °C) until the weight is stable for at least two successive readings; the readings should be separated by 30 minutes of heating in the oven.
2. For products coated with paint or a similar surface coating, remove and digest the coating, separately from the substrate material. Care should be taken to remove as little of the substrate

² Intertek Proposed Operating Procedure: Multiple Color Testing for Lead in Paint and Similar Surface Coating Materials (16 CFR 1303), Comment submitted to CPSC, February 13, 2009.

³ Toy Industry Association Inc. Laboratory Testing Technical Work Group, that includes (but is not limited to) representatives of the following laboratories: Specialized Technology Resources (STR), SGS consumer Testing Services; EMC Inc., Bureau Veritas (BV), CMA Testing NSF International, Intertek (ITS) and Toy Safety and Quality, Inc. *Compositing Acceptance Method for Lead in Surface Coatings*, Comment submitted to CPSC, March 18, 2009.

⁴ AOAC Official Method 974.02 *Lead in Paint*

⁵ ASTM E1645 *Standard Practice for Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis*

⁶ ASTM E1613 *Standard Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)*

as possible. It may be necessary to add a few drops of solvent, such as methylene chloride, to soften the paint and aid in its removal from the substrate. If used, such solvent must be evaporated fully prior to analysis. The scraped paint should be finely divided to help in digesting.

3. Scrape approximately 5-100 mg of paint from the product. If it is not possible to collect this much paint, it may be necessary to combine more than one unit of such product to collect sufficient paint.

4. Prepare a reagent blank sample and digest and test a standard reference paint material, such as National Institute of Standards and Technology (NIST) SRM 2581 - Powdered Paint Nominal 0.5% Lead or NIST SRM 2582 - Powdered Paint Nominal 0.02% Lead, with each batch of samples tested.

5. Digest appropriately according to AOAC 974.02 or ASTM E1645 in either a disposable glass test tube with a heating block, a disposable plastic digestion vessel in a hot block digester, or in a suitable digestion vessel and digestion microwave oven system.

6. Dilute samples so that Pb results are within the calibration range of the instrument. Be careful not to dilute a sample that could have 0.009% Pb to a level below the method detection limit.

7. Analyze diluted samples for Pb concentration using an ICP spectrometer (or Atomic Absorption spectrometer). Analysis procedures for ICP-OES, flame atomic absorption spectrometry (FLAA), and graphite furnace atomic absorption spectrometry (GFAA) are based on the methodology in ASTM E1613-04. Inductively coupled plasma mass spectrometry (ICP-MS) may also be employed with appropriate procedures, such as EPA 6020A⁷.

ICP Operating Procedures and Quality Control Measures

Analysis

1. Ignite plasma. Perform wavelength calibration or torch alignments per instrument manufacturer recommendations.
2. Allow the instrument to become thermally stable before continuing.
3. Ensure the following element and wavelength are selected in analytical method:
 - a. Pb 220.353

One other Pb line such as Pb 217.00 should be used to ensure spectral interferences are not occurring during analysis.

4. An internal standard such as 2 µg/ml yttrium should be used.
5. Perform calibration using calibration blank and at least 3 standards. Calibration should be performed a minimum of once a day when used for analysis, or each time the instrument is set up. Results for each standard should be within 5% of the true value. If the values do not fall within this range, recalibration is necessary.
6. Analyze the QCS immediately after the calibration. The analyzed value of Pb should be within ±10% of the expected value. If the Pb value is outside the ±10% limit, recalibration is required.
 - a. At least one LRB must be analyzed with each sample set. If the Pb value exceeds 3 times the MDL, laboratory or reagent contamination should be expected. The source of the contamination should be identified and resolved before continuing analyses. The LRBs should be at the same acid concentration as that of the

⁷ EPA 6020A *Inductively Coupled Plasma-Mass Spectrometry*

sample and should be taken through the same digestion procedure as that of a sample.

7. At least one certified reference material (CRM) should be analyzed with each batch of samples. The CRM should have a known amount of Pb and be of a material similar to a test specimen. Analyte recoveries should be within $\pm 15\%$ of expected values. If recoveries are outside this limit, the source of the problem should be identified and resolved before continuing analyses.
8. Dilute any samples that have Pb values exceeding 1.5 times the high calibration standard, and reanalyze.

Calculations and Results Reported

Results for the Pb test methods are calculated and reported as follows:

1. Total Pb concentration:⁸ $\%Pb \text{ (wt./wt.)} = 0.10cd/w$
 - a. c = concentration of Pb detected ($\mu\text{g/ml}$)
 - b. d = dilution volume (ml)
 - c. w = weight of aliquot digested (mg)

Example 1: Single Paint Tested

An aliquot of 10 mg of yellow paint scrapings is digested in acid and diluted to a final volume of 50 ml. When analyzed on the ICP-OES, the analytical result is 0.080 $\mu\text{g/ml}$ lead in the acid. The paint therefore contained $0.10 \times 0.080 \mu\text{g/ml} \times 50 \text{ ml} / 10 \text{ mg} = 0.04\%$ (400 ppm).

Table 1: Total Pb Analysis

(c)		(d)		(w)	
Item Analytical	result	Dilution	Total Pb	Sample	Pb (%)
	Pb ($\mu\text{g/ml}$)	volume	(μg)	wt (mg)	
		(ml)			
Yellow Paint	0.080	50	4	10	0.04

Example 2: Composite Testing – Different Parts

One example of composite testing of different paints would be as follows, and considers the case of weighing to the nearest 0.01 mg, digesting in acid, diluting to a final volume of 10 ml, and testing on an ICP-OES with an MDL of 0.01 $\mu\text{g/ml}$. A sample bears red, green, and orange paint. The paints are removed from the sample and 4.90 mg of red paint is combined with 5.00 mg of green paint, and 5.10 mg of orange paint. The resulting 15 mg of composite paint from this example is digested in acid and diluted to 10 ml, and then the diluted digest is found to contain 0.05 $\mu\text{g/ml}$ lead. The combined 3 aliquots of paint would have contributed to a total of 0.50 μg of lead for the composite sample. Although the average concentration in this case would be 33 ppm, the individual contributions are not known, and one must calculate the lead

⁸ The factor 0.10 in this formula is the mathematical conversion of the units used for concentration ($\mu\text{g/ml}$), and weight of paint (mg) into percent, and represents $100\% \times 1 \text{ mg} / 1000 \mu\text{g}$.

concentration of each paint as if all of the lead originated from it. Thus, the red paint could contain up to $0.50 \mu\text{g} / 0.0049 \text{ g} = 102 \text{ ppm} (\mu\text{g/g})$, with similarly calculated results of 100 ppm and 98 ppm for the green and orange paints. See Table 2 below.

Table 2: Total Pb Analysis – Composite Testing

(c)		(d)		(w)		
Item Analytical	result Pb ($\mu\text{g/ml}$)	Dilution volume (ml)	Total Pb (μg)	Sample wt (mg)	Potential Pb (%) per component	Pb (%) Composite
Red Paint	*0.05	10	0.5*	4.90	0.102	
Green Paint	*0.05	10	0.5*	5.00	0.100	
Orange Paint	*0.05	10	0.5*	5.10	0.098	
Total Composite	0.05	10	0.5	15.00		0.0033**

*In a composite of different component paints, the analytical result for the total composite would be applied to each component part as if all the Pb was in that component paint.

** In this example, the Pb concentration of the combined paints is <0.009%, but when estimating potential Pb per component, each component potentially could have >0.009% Pb and would require additional testing without compositing to ensure Pb <0.009%

Summary of changes in Revision CPSC-CH-E1003-09.1

Ministerial changes were made to the method as well as a change in the introduction to remove the disclaimer that the rules for accreditation for lead in paint testing did not require the use of the method. Additionally, the following statement was added. “Adjustments may be necessary to achieve total digestion for certain paints and should be based on sound chemistry knowledge and appropriate acids for the sample material being analyzed.”