



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 2582

#### Powdered Paint (low lead concentration)

This Standard Reference Material (SRM) is intended for use in the calibration of apparatus and the evaluation of methods used in the determination of lead in paint. SRM 2582 is intended to mimic or resemble the paint on interior surfaces of modern housing (see section on Collection). It consists of 20 g of a fine homogeneous powder of latex paint which 99+ % passes a 100  $\mu\text{m}$  (No. 145) sieve. The certified value, given below, is based on analysis of at least a 100 mg sample of the dried material. (See Instructions for Drying.)

Lead Content:  $208.8 \pm 4.9$  mg/kg

The certified value is based on measurements by isotope dilution thermal ionization mass spectrometry (ID-TIMS). The uncertainty in the certified value is calculated as

$$U = ku_c$$

where  $u_c$  is the "combined standard uncertainty" calculated according to the CIPM approach [1] and  $k$  is a coverage factor. The value of  $u_c$  is intended to represent at the level of one standard deviation, the combined effect of uncertainty components associated with material inhomogeneity and ID-TIMS measurement uncertainty. Additional detail on the components of uncertainty is given in Table 1. In the absence of Type B uncertainties (which are negligible here in comparison with Type A), the uncertainty given is for a 95% prediction interval, that is, there is a 95% chance that the interval will contain the lead concentration for a single bottle of this SRM. (Note that the coverage factor,  $k=2.57$ , is the Student's  $t$ -value for a 95% prediction interval with five degrees of freedom.)

The overall direction and coordination of the technical measurements leading to this certificate were performed by J.R. DeVoe and P.A. Pella of the NIST Inorganic Analytical Research Division.

Statistical calculations were carried out by E.S. Lagergren of the Statistical Engineering Division.

Financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency, Office of Research and Development, Atmospheric Research and Exposure Assessment Laboratory, S.L. Harper and M.E. Beard, project managers.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.S. Kane.

Gaithersburg, MD 20899  
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Thomas E. Gills, Chief  
Standard Reference Materials Program

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## NOTICE TO USERS

**Stability:** This material is considered to be stable; however, its stability has not been rigorously assessed.

**Use:** To relate analytical determinations to the certified value on this Certificate of Analysis, a minimum sample weight of 100 mg should be used and the sample should be dried according to the Instructions for Drying. Also, sample preparation procedures should be designed to effect complete dissolution in order to relate the determined value to the certified value.

## PREPARATION, TESTING, AND ANALYSIS

**Collection:** The latex paint for this SRM was removed from the corrugated metal ceiling of the lower level of a two-level warehouse in the Winston Salem, NC area under the direction of the Research Triangle Institute and the U.S. Environmental Protection Agency. The paint, which had been sprayed on in a thick single coat, was adhering poorly, and was peeling extensively. This allowed the paint to be removed with only moderate scraping using contoured rubber squeegees. Collection of paint and its initial evaluation for use as SRM 2582 was performed by J.D. Neefus, E.E. Williams, and D.B. Binstock, of the Research Triangle Institute, Research Triangle Park, NC, under the leadership of W.F. Gutknecht.

**Preparation:** The as-received paint material was first cleaned in the laboratory by passing it through a 1000  $\mu\text{m}$  (#70) sieve to remove as much debris and foreign matter as possible. Next the paint was coarsely chipped in a large-capacity blender fitted with a stainless steel blade. The paint was then further ground in small batches in a ball mill for 1/2 h. After that time, each batch was sieved and the fraction which did not pass a 100  $\mu\text{m}$  (#145) sieve was returned for further grinding with a fresh charge of coarse paint chips. All <100  $\mu\text{m}$  powdered paint was combined and blended as a single batch for 1 h before being bottled in 20 g units.

**Analysis:** Certification analyses by ID-TIMS were made by K.E. Murphy and R.D. Vocke of the NIST Inorganic Analytical Research Division. The XRF homogeneity analyses were performed by A.F. Marlow and P.A. Pella and inductively coupled plasma-optical emission spectrometric (ICP-OES) analyses were performed by L.J. Wood, also of the NIST Inorganic Analytical Research Division. The ICP-OES analyses given in Table 2 provide information on the concentration of constituents other than lead in the paint. These values listed below are not certified, but are given for information only, to enable users performing XRF analysis to make appropriate matrix correction calculations.

**Instructions for Drying:** Samples of this SRM should be air dried in an oven at 105 °C for 2 h. At NIST, weight loss on drying according to this procedure was approximately 1% by weight. Thus, the as-received concentration is ~207 mg/kg. However, under different humidity conditions in user laboratories, the weight loss could vary and that variation would affect the lead concentration of the as-received analysis and its uncertainty. Detailed studies of the variation in moisture content as a function of humidity have not been undertaken.

## SUPPLEMENTAL INFORMATION

Table 1. Components of Uncertainty for SRM 2582

Source	$u_i$ in mg/kg	Degrees of Freedom
Type A, Statistically Determined		
Sample measurement, instrumental and material variability	1.91	5
Spike calibration	0.08	3
Fractionation correction	0.04	5
Blank Correction	<0.01	2
Combined Type A	1.91	
Type B, Other		
Purity of Calibrant	0.18	$\infty$
Combined standard uncertainty, $u_c$	1.92	
Degrees of Freedom:	5	
Coverage Factor; K	2.57	
Expanded Uncertainty U:	4.94	

Table 2. Non-certified values, in wt. %

Element	Concentration
Al	1.6
Ca	15.1
Fe	0.21
Ti	8.1
Zn	0.51

## REFERENCES

- [1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Switzerland, 1993.