



National Institute of Standards & Technology

# Certificate of Analysis

Standard Reference Material<sup>®</sup> 2589

Powdered Paint  
(Nominal 10 % Lead)

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and for the calibration of apparatus used to determine lead in paint. SRM 2589 is composed of paint collected from the interior surfaces of housing. A unit of SRM 2589 consists of 35 g of powdered paint material, 99+ % of which passes a 100  $\mu\text{m}$  (No. 145) sieve. The certified mass fraction of lead, given below, is based on measurements by isotope dilution thermal ionization mass spectrometry (ID-TIMS) with a minimum sample size of 100 mg. The certified value is reported on a dry basis (see "Instructions for Drying").

## Certified Mass Fraction

Lead Content: 9.99 %  $\pm$  0.16 %

The uncertainty in the certified value is calculated as

$$U = ku_c$$

where  $u_c$  is the combined standard uncertainty calculated according to the ISO Guide [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. In the absence of Type B uncertainties (which are negligible here in comparison with Type A), the expanded uncertainty ( $U$ ) given is for a 95 % confidence interval. The coverage factor,  $k = 2.09$ , is the Student's  $t$ -value for a 95 % confidence interval with 19 degrees of freedom.

**Expiration of Certification:** The certification of **SRM 2589** is valid, within the measurement uncertainty specified, until **31 December 2020**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

**Maintenance of Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the technical measurements leading to certification of this SRM were performed by J.R. DeVoe, P.A. Pella, and R.L. Watters, Jr. of the NIST Analytical Chemistry Division.

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (EPA) under the direction of project managers S.L. Harper and M.E. Beard of the EPA Office of Research and Development, National Exposure Research Laboratory, Research Triangle Park, NC.

Stephen A. Wise, Chief  
Analytical Chemistry Division

Robert L. Watters, Jr., Chief  
Measurement Services Division

Gaithersburg, MD 20899  
Certificate Issue Date: 08 June 2009  
*See Certificate Revision History on last Page*

Statistical consultation was provided by E.S. Lagergren of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

## INSTRUCTIONS FOR USE

To relate analytical determinations to the certified value on this Certificate of Analysis, a minimum sample mass of 100 mg should be used and the sample should be dried according to the "Instructions for Drying". Sample preparation procedures should also be designed to effect complete dissolution in order to relate the determined value to the certified value. This SRM must be stored in an air conditioned environment or similar cool and dry environment away from sunlight and fumes.

**Instructions for Drying:** Samples of this SRM should be dried in an oven at 105 °C for 2 h. At NIST, loss on drying according to this procedure was less than 1 % relative by mass. However, under different conditions of humidity, the mass loss could vary. In order for users to directly relate their analyses to the certified value, loss on drying corrections should be measured and applied at the time of the analysis.

## COLLECTION, PREPARATION, AND ANALYSIS

**Collection:** The paint material for this SRM was collected primarily in Athens, Ohio from various interior wall surfaces of old housing which, for the most part, were painted prior to 1945. The material was collected under the direction of the Research Triangle Institute and the U.S. Environmental Protection Agency. Collection of paint by dry scraping and its initial evaluation for use as SRM 2589 were 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:** First the largest pieces of debris and foreign material were removed by hand. Next, the material was coarsely chipped in a large-capacity blender fitted with a stainless steel blade. The material was then further ground in small batches in a ball mill. Each batch was sieved and the fraction that did not pass a 100 µm (#145) sieve was returned for further grinding with a fresh charge of coarse paint material. All material of a size less than 100 µm was combined and blended as a single batch before being bottled in 35 g units.

**Analysis:** Certification analysis by ID-TIMS was performed by K.E. Murphy and R.D. Vocke of the NIST Analytical Chemistry Division. The X-ray fluorescence homogeneity analysis was performed by A.F. Marlow and P.A. Pella and the inductively coupled plasma-optical emission spectrometric (ICP-OES) analysis were performed by L.J. Wood of the NIST Analytical Chemistry Division and R. Saraswati, Guest Scientist from the Defense Metallurgical Research Laboratory, India.

The ICP-OES analysis data given in Table 1 provide information on the concentrations of major constituents other than lead in the material. These values listed are not certified, but are given for information only to provide additional information on the matrix.

**Environmental Lead Proficiency Analytical Testing Program Results:** This material was included as an unknown in the Environmental Lead Proficiency Analytical Testing Program (ELPAT) administered by the American Industrial Hygiene Association (AIHA). Conventional dissolution methods employed by participating laboratories include hotplate, microwave, and other techniques such as sealed bomb dissolutions and leaching techniques. Instrumental determinations were performed using inductively coupled plasma-mass spectrometry (ICP-MS), inductively coupled plasma-optical emission spectrometry (ICP-OES), flame atomic absorption spectrometry (FAAS), graphite furnace atomic absorption spectrometry (GFAAS), and X-ray fluorescence spectrometry (XRF). Information from this study is provided to indicate the state of the practice for lead in paint measurements using such methods. A summary of the round robin lead results obtained from ELPAT Reference Laboratories for SRM 2589 is presented in Table 2. The SRM 2589 is identified as round robin 002, paint #4 in the ELPAT report.

## SUPPLEMENTAL INFORMATION

Table 1. Information Values for Major Constituents of SRM 2589

Element	Mass Fraction (%)
Al	1
Ca	12
Fe	0.2
Mg	1
Ti	9
Zn	2

Table 2. Environmental Lead Proficiency Analytical Testing (ELPAT) Program Summary Statistics of Reference Laboratories for Round Robin 002<sup>(a)</sup>

Sample	n	Mean	Minimum	Maximum	S <sup>(b)</sup>
Paint 4	31	9.55 %	7.57 %	10.8 %	0.91

<sup>(a)</sup> These results are provided to demonstrate user experience with this material. They were not used in calculating the certified value of SRM 2589.

<sup>(b)</sup> s is one standard deviation.

## REFERENCE

- [1] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.

**Certificate Revision History:** 08 June 2009 (This revision reflects editorial updates); 10 March 2009 (This revision reports an extension in the certification period); 10 December 1996 (Original certificate date).

*Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*