National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2582

Powdered Paint
Nominal 200 mg/kg 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 2582 is intended to resemble the paint on interior surfaces of housing. It consists of 20 g of powdered latex paint of which 99+ % passes a 100 μm (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: 208.8 mg/kg ± 4.9 mg/kg

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 % prediction interval. The coverage factor, $k = 2.57$, is the Student's t-value for a 95 % prediction interval with 5 degrees of freedom.

NOTICE AND WARNING TO USERS

Expiration of Certification: This certification is valid for five years from the date of shipment from NIST. Should there be any change in the certified value before the expiration of certification, purchasers will be notified by NIST. Return of the attached registration card will facilitate notification.

Stability: This material is considered to be stable; however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

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.

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

Gaithersburg, MD 20899
May 2, 1996
(Revision of certificate dated 6-23-94)

Thomas E. Gills, Chief
Standard Reference Materials Program

The overall direction and coordination of the technical measurements leading to this certificate were performed by J.R. DeVoe, P.A. Pella, and R.L. Watters, Jr. of the NIST Analytical Chemistry Division. Statistical calculations were carried out by E.S. Lagergren of the NIST Statistical Engineering 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.

COLLECTION, PREPARATION, 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 scientists from the Research Triangle Institute and the U.S. Environmental Protection Agency. The paint, which had been sprayed on in a thick single coat, and was already peeling extensively when collection of paint by dry scraping was initiated. Preliminary 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: First the largest piece of debris and foreign matter were removed from the material by passing it through a 500 μm (#35) sieve. 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 20 g units.

Analysis: Certification analyses by ID-TIMS were 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) analyses were performed by L.J. Wood of the NIST Analytical Chemistry Division.

The ICP-OES analyses 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 and are given for information only to provide additional information on the matrix.

Instructions for Drying: Samples of this SRM should be air 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.

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)[a]. 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 2582 is presented in Table 2. The SRM 2582 is identified as round robin 005, paint #2 in the ELPAT report.

SUPPLEMENTAL INFORMATION

Table 1. Information Values for Major Constituents of SRM 2582

Element	Mass Fraction (%)
Al	1
Ca	15
Fe	0.2
Mg	0.2
Ti	15
Zn	0.6

Table 2. Environmental Lead Proficiency Analytical Testing (ELPAT) Program Summary Statistics of Reference Laboratories for Round 005[a]

Sample	n	Mean	Minimum	Maximum	s[b]
Paint 2	36	222 mg/kg	186 mg/kg	271 mg/kg	3 mg/kg

[a]These results are provided to demonstate user experience with this material. They were not used in calculating the certified value of SRM 2582.
[b]s is one standard deviation.

REFERENCE

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993): See also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, (1994).