National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2582

Powdered Paint
(Nominal Mass Fraction of 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 composed of paint collected from the interior surfaces of housing. A unit of SRM 2582 consists of 20 g of powdered paint material, 99+ % of which 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"). Metrological traceability is to the SI derived unit for mass fraction (expressed as mg per kg).

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

Expiration of Certification: The certification of **SRM 2582**is valid, within the measurement uncertainty specified, until **12 June 2022**, provided the SRM is handled and stored 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 SRM 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 or register online) will facilitate notification.

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., formerly of NIST.

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.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 24 August 2020
See Certificate Revision History on last Page

Steven J. Choquette, Director
Office of Reference Materials

Statistical consultation was provided by E.S. Lagergren formerly of NIST.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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 air atmosphere at 105 °C for 2 h. At NIST, loss on drying according to this procedure was about 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 latex paint for this SRM was removed from the corrugated metal ceiling of the lower level of a two-level warehouse in Winston-Salem, NC 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, was already peeling extensively when collection of paint by dry scraping was initiated. Preliminary evaluation for use as SRM 2582 was preformed 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 matter were removed from the material by passing it through a 500 μm (No. 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 (No. 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 analysis by ID-TIMS was performed by K.E. Murphy and R.D. Vocke of the NIST Chemical Sciences Division. The X-ray fluorescence homogeneity analysis was performed by A.F. Marlow of the NIST Chemical Sciences Division, and P.A. Pella, formerly of NIST. The inductively coupled plasma optical emission spectrometric (ICP-OES) analysis was performed by L.J. Wood of the NIST Chemical Sciences Division.

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 only to provide additional information on the matrix. Information values cannot be used to establish metrological traceability.

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 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 Robin 005, Paint 2[(a)]

Sample	n	Mean	Minimum	Maximum	s[(b)]
Paint 2	36	222 mg/kg	186 mg/kg	271 mg/kg	30 mg/kg

[(a)] These results are provided to demonstrate 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] 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Aug 2020).

Certificate Revision History: 24 August 2020 (Change of expiration date; title updated; editorial changes); **08 June 2009** (This revision reflects editorial changes); **10 March 2009** (This revision reports an extension in the certification period); **12 June 1997** (This revision reports a correction to Table 2 ELPAT); **02 May 1996** (Editorial revision); **23 June 1994** (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.