National Institute of Standards and Technology
U.S. Department of Commerce

Date of Issue:
09 September 2022

Standard Reference Material® 2589

Powdered Paint

(Nominal Mass Fraction of 10 % Lead)

CERTIFICATE OF ANALYSIS

Purpose: The certified value delivered by 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.

Description: A unit of SRM 2589 consists of 35 g of powdered paint material, 99+ % of which passes a 100 μm (No. 145) sieve.

Certified Value: 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 International System of Units (SI) derived unit for mass fraction (expressed as a percent) [1,2].

Certified Mass Fraction

Lead Content: 9.99 % ± 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.

Additional Information: Values of potential interest to users and additional information are provided in Appendix A.

Period of Validity: The certified values delivered by **SRM 2589** are valid within the measurement uncertainty specified until **12 June 2032**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM at the time of purchase. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available free of charge through the NIST SRM website.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

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Safety: Please refer to the Safety Data Sheet for this material.

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 the original bottle in a cool (10 °C to 25 °C), dry (relative humidity ≤60 %) environment away from light 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 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.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Sep 2022).

[2] JCGM 100:2008; Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/en/publications/guides (accessed Sep 2022); 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 Sep 2022).

Certificate Revision History: 09 September 2022 (Updated format; updated storage instructions; change of period of validity; editorial changes); **14 August 2020** (Change of expiration date; editorial changes); **08 June 2009** (This revision reflects editorial updates); **10 March 2009** (This revision reports an extension in the certification period); **09 June 1997** (Correction on site material collection; editorial changes); **02 May 1996** (Original certificate date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Values of Potential Interest to Users: Values of potential value to users are shown below.

Table A1. Values of Potential Interest to users for Major Constituents of SRM 2589

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

Table A2. Environmental Lead Proficiency Analytical Testing (ELPAT) Program Summary Statistics of Reference Laboratories for Round Robin 002, Paint 4[(a)]

Sample	N	Mean	Minimum	Maximum	s[(b)]
Paint 4	31	9.55 %	7.57 %	10.8 %	0.91

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

[(b)] s is one standard deviation.

Additional Information:

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 (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 35 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 and R. Saraswati, Guest Scientist from the Defense Metallurgical Research Laboratory, India.

The ICP-OES analysis data given in Table A1 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 2589 is presented in Table A2. The SRM 2589 is identified as round robin 002, paint 4 in the ELPAT report.

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.

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.

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