

Standard Reference Material[®] 3128

Lead (Pb) Standard Solution

Lot No. 101026

CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for use as a primary calibration standard for the quantitative determination of lead.

Description: A unit of SRM 3128 consists of five 10 mL sealed borosilicate glass ampoules of an acidified aqueous solution prepared gravimetrically to contain a known mass fraction of lead. The solution contains nitric acid at a concentration of approximately 1.6 mol/L.

Certified Values: These values are traceable to the International System of Units (SI) [1]. The measurand is the mass fraction of lead, and the certified value is metrologically traceable to the SI derived unit for mass fraction, expressed as milligrams per gram.

Certified Value of Lead: 9.995 mg/g ± 0.014 mg/g

The certified value is a weighted mean [2,3] of the results of (1) gravimetric preparation using high-purity, assayed lead and (2) inductively coupled plasma optical emission spectrometry (ICP-OES) calibrated using four primary standards independently prepared from high-purity, assayed lead.

The uncertainty in the certified value is calculated as

$$U = ku_c$$

where $k = 2.007$ is the coverage factor for a 95 % confidence interval and 52 effective degrees of freedom. The quantity u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guide [4,5]. 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 gravimetric preparation, the ICP-OES determination, any bias observed between the methods' results, and stability of the actual lead mass fraction.

Period of Validity: The certified value delivered by **SRM 3128 Lot 101026** is valid within the measurement uncertainty specified until **30 April 2028**. 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. Registration will facilitate notification. Before making use of the value delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

Metrological Traceability

Metrological traceability of measurement results to a given reference must be established through an unbroken chain of calibrations and/or comparisons, each having stated uncertainties [6], using measurement standards that are appropriate for the physical or chemical property being measured. Comparisons may include validation measurements using various spectroscopic, chromatographic, or classical methods of analysis. Gravimetric or volumetric dilution is also a method of comparison, where the mass or volume of a solution before and after dilution is measured.

This SRM can be used to establish traceability of the results of lead measurements to NIST measurement results and standards. One approach is to calibrate analytical instruments or procedures for the determination of lead using standards, the values of which are traceable to the certified value of lead in this SRM. When the traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of all calibration measurements.

Safety: This SRM is an acid solution contained in tip-sealed borosilicate glass ampoules with pre-scored stems. All appropriate safety precautions, including use of gloves during handling, should be taken. Unopened ampoules should be stored under normal laboratory conditions in an upright position inside the original container supplied by NIST. Consult the Safety Data Sheet (SDS), enclosed with the SRM shipment, for chemical hazard information.

Storage: Unopened ampoules should be stored upright under normal laboratory conditions inside the original container supplied by NIST.

Opening an Ampoule: When an ampoule is to be opened, that area of the stem where the prescored band is located (≈ 5 mm below the encircling metallic band) should be carefully wiped with a clean, damp cloth and the body of the ampoule wrapped in absorbent material. Holding the ampoule steady and with thumb and forefinger grasping the stem at the metallic band, **minimal** thumb pressure should be applied to the stem to snap it. Correctly done, the stem should break easily where prescored. Use of a metal file to break the stem is **NOT** recommended.

Working Standard Solutions: After opening the ampoule, the entire contents should be transferred immediately to another container and *working standard solutions* should be prepared. Working standard solutions in the range of 10 mg/kg to 100 mg/kg are recommended, from which more dilute standards can be prepared. The user should establish internal laboratory procedures that specify a maximum shelf life for a working standard solution. Two procedures for the preparation of working standard solutions follow.

Preparation of Working Standard Solutions by Mass: Each working standard solution should be prepared by emptying one or more ampoules of the SRM into an empty, dry, preweighed, polyethylene bottle and then reweighing the bottle. An appropriate dilute acid must be added by mass to bring the solution to the desired dilution. The dilution need not be exact since the mass of the empty bottle, mass of the bottle plus SRM aliquot, and the final diluted mass of the solution will permit calculation of the exact mass fraction (mass of lead per mass of solution) of the working standard solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true mass fraction in vacuum.

Preparation of Working Standard Solutions by Volume: Volumetric dilutions are **NOT** recommended due to uncertainties in volume calibrations and variations in density. However, for user convenience, a procedure for volumetric preparation that will minimize the major sources of error is given. Each working standard solution should be prepared by emptying one or more ampoules of the SRM into an empty, dry, polyethylene bottle and weighing the bottle. The solution must be transferred to a Class A volumetric flask and the polyethylene bottle reweighed to determine the exact mass of SRM solution transferred. The solution in the flask is diluted to 99 % + volume using an appropriate dilute acid, mixed thoroughly, and the remaining few drops needed to dilute to exact volume carefully added. The concentration (in mg/mL) of the resulting working standard solution can be calculated by multiplying the mass (in g) of the SRM solution amount by the SRM certified value (in mg/g) and dividing the numerical product by the calibrated volume (in mL) of the flask used for dilution. If this procedure is followed, no correction for density is needed. Although the concentration of the resulting working standard solution may be an uneven fraction of the original SRM concentration, it will be known as accurately as a volumetric dilution permits.

Possible Presence of Other Elements: Studies conducted by NIST have shown that components of borosilicate glass ampoules may leach into solution. In *undiluted* solutions, Na and Si mass fractions as large as 20 mg/kg, B and La mass fractions in the range 1 mg/kg to 5 mg/kg, and Al, As, Ca, Ce, Mg, Mn, Rb, and Zn mass fractions in the range 0.05 mg/kg to 1 mg/kg have been found. When diluted to prepare working standard solutions, the levels of these elements become negligible for most purposes. Nevertheless, possible effects should be considered when this SRM is used.

NOTICE TO USERS

NIST encourages the use of its SRMs to establish metrological traceability for the user's measurement results, and NIST strives to maintain the SRM inventory supply. However, NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of SRMs as primary benchmarks for the quality and accuracy of the user's in-house (working) standards. As such, SRMs should be used to validate or otherwise assign values to the more routinely used standards in a laboratory. When the metrologically traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of the calibration measurements for the in-house standard. Comparisons between NIST SRMs and such working measurement standards should take place at intervals appropriate to the conservation of the SRM primary standard and the stability of relevant in-house standards. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

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 (NIST SP) 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 May 2022).
- [2] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; *Metrologia*, Vol. 46; pp. 323–331 (2009).
- [3] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; *Control. Clin. Trials*, Vol. 7; pp. 177–188 (1986).
- [4] Horn, R.A.; Horn, S.A.; Duncan, D.B.; *Estimating Heteroscedastic Variance in Linear Models*; *J. Am. Stat. Assoc.*, Vol. 70; pp. 380–385 (1975).
- [5] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement*; (ISO GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at <https://www.bipm.org/en/publications/guides> (accessed May 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 May 2022).
- [6] JCGM 200:2012; *International Vocabulary of Metrology - Basic and General Concepts and Associated Terms*, 3rd ed.; Joint Committee for Guides in Metrology (JCGM) (2012); available at <https://www.bipm.org/en/publications/guides> (accessed May 2022).

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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

Coordination of the technical measurements leading to the certification of SRM 3128 Lot No. 101026 was provided by M.R. Winchester of the NIST Chemical Sciences Division.

This SRM was prepared by T.A. Butler of the NIST Chemical Sciences Division. The ICP-OES analysis was performed by T.A. Butler and M.R. Winchester. Primary standards for calibration of ICP-OES measurements were prepared by T.A. Butler.

Statistical consultation was provided by S.D. Leigh, formerly of the NIST Statistical Engineering Division.

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

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