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

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Standard Reference Material® 3328
Lead (Pb) Isotopic Standard Solution
CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended primarily as an isotopic reference material for instrument calibration and to correct for mass bias in lead (Pb) isotope ratio measurements on a mass spectrometer. Additionally, this SRM defines the Pb SRM 981 isotope-delta scale ($\delta^{x}Pb_{SRM981}$) for relative isotope ratio measurements of Pb, $R(^{206}Pb/^{204}Pb)$, $R(^{207}Pb/^{206}Pb)$, and $R(^{208}Pb/^{206}Pb)$ [1].

Description: A unit of SRM 3328 consists of two 10 mL sealed borosilicate glass ampoules containing a solution of approximately 10 mg/kg Pb. The solution consists of nitric acid at a volume fraction of approximately 2 %, equivalent to an amount-of-substance concentration (molarity) of approximately 0.47 mol/L. SRM 3328 was produced from a portion of SRM 981 Pb wire.

Certified Values: Certified values for SRM 3328 are reported below in Tables 1–3. Value assignment categories are based on the definitions of terms and modes used at NIST for chemical reference materials [2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been taken into account [2]. The uncertainties associated with the certified values are expanded uncertainties (95 % confidence interval) and are calculated according to the methods described in the ISO/JCGM Guide [3]. All certified values are traceable to the International System of Units (SI).

Table 1. Certified Absolute Isotope-amount Ratios for SRM 3328

Isotope Ratio	Isotope-amount Ratio[(a)]
$n(^{204}Pb)/n(^{206}Pb)$	0.059042 ± 0.000037
$n(^{207}Pb)/n(^{206}Pb)$	0.91464 ± 0.00033
$n(^{208}Pb)/n(^{206}Pb)$	2.1681 ± 0.0008
	Atomic Mass Units (u)
Lead Atomic Weight[(b)]	207.21517 ± 0.00015

[(a)] The measurand is the absolute isotope-amount ratio. Metrological traceability is to the mole, the SI unit of amount-of-substance.

[(b)] The lead atomic weight, expressed in atomic mass units (u), is calculated [4] from the isotope-amount ratios and associated uncertainties listed in Table 1, and on the relative atomic masses of the stable isotopes of Pb and their uncertainties taken from reference 5. This certified value is specific to this particular material and does not coincide with the current standard atomic weight of lead as published by the IUPAC Commission on Isotopic Abundances and Atomic Weights (CIAAW), whose current value is 207.2 ± 1.1 [6].

Additional Information: Additional information is provided in Appendix A.

Period of Validity: The certified values delivered by **SRM 3328** are valid within the measurement uncertainty specified until **15 May 2038**. The certified values assigned are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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Table 2. Certified Isotope-amount Fraction Values for SRM 3328

Isotope	Isotope-amount Fraction[(a)] (mol/mol)
$X_{\text{SRM 3328}}(^{204}\text{Pb})$	0.014255 ± 0.000012
$X_{\text{SRM 3328}}(^{206}\text{Pb})$	0.241442 ± 0.000057
$X_{\text{SRM 3328}}(^{207}\text{Pb})$	0.220833 ± 0.000074
$X_{\text{SRM 3328}}(^{208}\text{Pb})$	0.523470 ± 0.000099

[(a)] The measurand is the isotope-amount fraction expressed as mol/mol, mole fraction. Metrological traceability is to the mole, the SI unit of amount-of-substance. The expanded uncertainties are based on the isotope-amount ratios listed in Table 1 and the atomic masses of the stable isotopes of Pb and their associated uncertainties reported in reference 5, and they take into account the correlations between the isotope-amount ratios and the correlations induced by the constant sum of the isotope-amount fractions.

Table 3. Certified Isotope-delta Values for $\delta^{x}\text{Pb}_{\text{SRM981}}$ SRM 3328

$\delta^{x}\text{Pb}_{\text{SRM981}}$	Isotope-delta Value $\delta^{x}\text{Pb}_{\text{SRM981}}$[(a,b)] (‰)
$\delta^{206/204}\text{Pb}_{\text{SRM981}}$	0 ± 0 (exact)
$\delta^{207/206}\text{Pb}_{\text{SRM981}}$	0 ± 0 (exact)
$\delta^{208/206}\text{Pb}_{\text{SRM981}}$	0 ± 0 (exact)

[(a)] The $\delta^{x}\text{Pb}_{\text{SRM981}}$ values are exact values in units of per mil (‰) and are the common "stated reference" point for the Pb isotope-delta scale [1]. The Pb isotope-delta scale ($\delta\text{Pb}_{\text{SRM981}}$) is realized primarily and explicitly through SRM 3328, where SRM 3328 is $\delta^{x}\text{Pb}_{\text{SRM981}} \equiv 0$ ‰. The isotope-delta values are traceable to the SI, specifically the unit of amount-of-substance, the mole, through the SRM 981 values.

[(b)] The symbol ‰ is part per thousand and is equal to 0.001.

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 any of the values 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).

Safety: SRM 3328 is intended for research use. 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.

Storage: SRM 3328 is stable at room temperature (20 ºC ± 5 ºC). It is recommended that any unused portion of the solution be transferred from the ampoule to a pre-cleaned Teflon container for long term storage. A Teflon container such as FEP or PFA cleaned with a series of dilute nitric acid soaks followed by soaking in double distilled or 18 MΩ cm water is suggested. This applies to working solutions as well as storage of the stock solution.

Use: The recommended sample size for preparing a Pb isotopic working solution will depend on the sensitivity of the instrument used for analysis and the type of balance used for the gravimetric preparation. For planning purposes, a 20 g working solution with an adequate Pb mass fraction (200 µg/kg–300 µg/kg) can be readily prepared from a sample size of approximately 0.4 g–0.5 g (when using a 4 or 5-place analytical balance). Prior to sampling, the ampoule unit should be thoroughly mixed by carefully inverting the ampoule a minimum of five times. The 0.4 g–0.5 g aliquot is then diluted in approximately 20 g of 2 % volume fraction high-purity HNO_3 (e.g., sub-boiling distilled). A Pb purity of +99.9 % should be assumed.

REFERENCES

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If you use this SRM in published work, please reference:

Catanzaro EJ, Murphy TJ, Shields WR, Garner EL (1968) Absolute Isotopic Abundance Ratios of Common, Equal Atom, and Radiogenic Lead Isotopic Standards. J. Res. Natl. Bur. Stand., Vol. 72A, No. 3, pp. 261-267. https://doi.org/10.6028/jres.072A.025

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, Maryland 20899-2300; telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Source and Preparation: The source of the Pb material used to produce SRM 3328 was the Pb wire from the original SRM 981 wire stock. This Pb wire material was purified to >99.9 % Pb by the former Cominco American, Inc. (Spokane, WA). An aliquot of the SRM 981 Pb wire was etched to remove the oxide coating with nitric acid, dried, and weighed. The weighed aliquot of SRM 981 Pb wire was then digested in dilute nitric acid and a stock Pb solution (SRM 3328) with an amount content of 10 mg/kg in 2 % volume fraction HNO_3 was produced. This Pb solution was then ampouled under argon into clear borosilicate ampoules.

Analysis: Trace element impurities of the original Pb wire (SRM 981) used to prepare SRM 3328 were determined by quantitative emission spectrography. The absolute isotope-amount ratios of $n(^{204}Pb)/n(^{206}Pb)$, $n(^{207}Pb)/n(^{206}Pb)$, and $n(^{208}Pb)/n(^{206}Pb)$ of SRM 981 were determined by thermal ionization mass spectrometry. Gravimetrically prepared mixtures of electromagnetically separated and chemically pure Pb end member isotopes obtained from Oak Ridge National Laboratory (Oak Ridge, TN) were used to calibrate the mass spectrometers. Absolute isotope-amount ratios were realized via the bias correction (true isotopic ratio/observed (measured) isotopic ratio) derived from synthetic isotopic mixtures. Details of the measurements of SRM 981 are described in reference [7]. Measurements of the Pb isotope-amount ratios ($n(^{204}P)/n(^{206}Pb)$, $n(^{207}Pb)/n(^{206}Pb)$, and $n(^{208}Pb)/n(^{206}Pb)$) of the new SRM 3328 Lead (Pb) Isotopic Standard Solution were made and compared to concurrently measured isotope-amount ratios of dissolutions of the original SRM 981 Pb wire to ensure that the Pb isotope-amount ratios remained constant during the conversion from a solid piece of wire to a solution. Design of Experiments (DEX) scatter plots and mean plots were used to evaluate this process and determine if the SRM 3328 measurements were in agreement with the SRM 981 certified isotope-amount ratios. Within the propagated uncertainties the values determined for SRM 3328 were indistinguishable from the certified values of SRM 981.

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