



NIST PS1

Primary Standard for quantitative NMR (Benzoic Acid)

CERTIFICATE OF ANALYSIS

Purpose: This NIST Certified Reference Material is intended for use as a primary standard for quantitative nuclear magnetic resonance (qNMR) methods.

Description: A unit of NIST PS1 consists of two vials, each containing approximately 500 mg of purified crystalline benzoic acid.

Certified Values: Certified values, valid for use of quantities ≥ 5 mg, are provided in Table 1. A NIST certified value is a value for which NIST has the highest confidence in that all known or suspected sources of bias have been accounted for. Confidence in the certified values was achieved through acidimetric titration and qNMR primary measurement procedures.

Table 1. Certified Values for Benzoic Acid in NIST PS1

Values are metrologically traceable to the International System of Units (SI) mole and kilogram.

	Mass fraction, g/g ^(a,b,c)	Specific amount, mol/kg ^(a,b,c)
Benzoic Acid ^d	0.99992 -0.00006 +0.00004	8.1880 -0.0005 +0.0004

- (a) Values are expressed as $x - U_{L95\%} + U_{H95\%}$, where x denotes the certified value and $U_{L95\%}$ and $U_{H95\%}$ indicate the low and high boundaries of a 95% coverage interval for the true value of the measurand.
- (b) The standard uncertainty may be approximated as $u(x) = U_{L95\%} / 2$.
- (c) If a Monte Carlo method, as described in the GUM Supplement 1 [1], is employed to propagate the uncertainty of the certified value, this may be accomplished via “The NIST Uncertainty Machine” (<https://uncertainty.nist.gov/>) [2], whereby the certified mass fraction is assigned a beta distribution, Beta (Shape1, Shape2), with parameters Shape1=88873.7, Shape2=7.11. The assignment for the certified specific amount may be derived from this distribution using the molar mass (M) of benzoic acid.
- (d) The value of M was evaluated empirically through stable isotope relative abundance measurements as 122.12204 g/mol with an associated standard uncertainty $u(M) = 0.00019$ g/mol.

Expiration of Certification: The certification of **NIST PS1** is valid, within the stated measurement uncertainties, until **31 December 2037**, provided the CRM is handled and stored in accordance with the instructions given in this certificate. The certification is nullified if the CRM is damaged, contaminated, or otherwise modified.

Source: The benzoic acid used in this CRM was provided, prepared and packaged by Sigma-Aldrich Production GmbH and in collaboration with NIST.

Storage and Handling: Until required for use, NIST PS1 should be stored at room temperature in its original packaging. After use, this CRM should be stored in its original bottle at room temperature. It must be tightly re-capped after use, protected from moisture and light, and placed in a desiccator for long term storage. The certified value should not be considered valid if remaining material is not stored in this manner.

Supporting Measurements: Direct ^1H -qNMR comparisons of NIST PS1 were made to SRM 350b that established confidence in traceability for historical use of SRM 350b for ^1H -qNMR calibrations. Confirmatory ^1H -qNMR analysis by an external standard approach was performed by the National Research Council (NRC) Canada. Confirmatory ^1H -qNMR analysis of bulk purity by an internal standard approach was performed by Sigma-Aldrich Production GmbH. Mass spectrometric determinations of stable isotope ratios were performed by the United States Geological Survey, Reston, VA and NRC Canada. Isotope atom fractions (x) of ^1H and ^{13}C are derived from the isotope delta values of $\delta^2\text{H}_{\text{VSMOW-SLAP}}$ and $\delta^{13}\text{C}_{\text{VPDB}}$ as $x(^1\text{H}) = 0.999\ 860$, $u(x(^1\text{H})) = 0.000\ 003$ and $x(^{13}\text{C}) = 0.010\ 74$, $u(x(^{13}\text{C})) = 0.000\ 09$, respectively [3,4,5].

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.

Distribution of this NIST CRM will occur only via Material Transfer Agreement. For more information, contact the Technology Partnerships Office, 100 Bureau Drive, Stop 2200, Gaithersburg, Maryland 20899-2200; telephone (301) 975-2573; e-mail tpo@nist.gov.

Technical queries regarding the certification and usage of this material may be directed to the NIST PS1 Technical Project Leader, Michael Nelson; e-mail michael.nelson@nist.gov.

Users of this CRM should ensure that the Certificate of Analysis in their possession is current. NIST will monitor this CRM until its certification expires. If substantive technical changes occur that affect the certified values before this certificate expires, NIST will notify the signatory of the Material Transfer Agreement.

Reference

- [1] Joint Committee for Guides in Metrology (2008) "Evaluation of measurement data --- Supplement 1 to the "Guide to the expression of uncertainty in measurement" --- Propagation of distributions using a Monte Carlo method", JCGM 101:2008, International Bureau of Weights and Measures (BIPM), Sevres, France, www.bipm.org/en/publications/guides/gum.html.
- [2] T. Lafarge and A. Possolo (2015) "The NIST Uncertainty Machine", NCSLI Measure Journal of Measurement Science 10(3): 20-27
- [3] Coplen, T. (1994) Reporting of stable hydrogen, carbon, and oxygen isotopic abundances (Technical Report). *Pure and Applied Chemistry* 66(2): 273-276
- [4] Brand W.A., Coplen T.B., Vogl J., Rosner M., Prohaska T. (2014) Assessment of international reference materials for isotope-ratio analysis (IUPAC Technical Report), *Pure and Applied Chemistry* 86(3): 425
- [5] Coplen T.B., Hopple J.A., Böhlke J.K., Peiser H.S., Rieder S.E., Krouse H.R., Rosman K.J.R., Ding T., Vocke R.D. Jr., Révész K.M., Lamberty A., Taylor P., De Bièvre P. (2002) "Compilation of Minimum and Maximum Isotope Ratios of Selected Elements in Naturally Occurring Terrestrial Materials and Reagents", U.S. Geological Survey Water-Resources Investigations Report 01-4222.