National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 350c

Benzoic Acid
(Acidimetric Standard)

This Standard Reference Material (SRM) consists of purified benzoic acid (C_6H_5COOH). SRM 350c is intended for use in acidimetric standardization. This material is NOT suitable for use as a standard in quantitative nuclear magnetic resonance spectrometry (qNMR). A unit of SRM 350c consists of 30 g of benzoic acid.

Certified Values: 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 investigated or taken into account [1]. The certified value, reported in Table 1 as the mass fraction of C_6H_5COOH ($w_{C_6H_5COOH}$), is based on coulometric assays of the dried material (see "Drying Instructions") including the effects of air buoyancy. The certified value is based on the results of 18 determinations from 12 randomly selected bottles from the entire lot of SRM 350c. Each determination was obtained by coulometric acidimetric titration [2] to the inflection point (pH ≈ 8.15).

Table 1. Certified Value for SRM 350c, Benzoic Acid

$w_{C_6H_5COOH}$	99.959 %	±	0.040 %

The uncertainty in the value, calculated according to the method described in the ISO/JCGM Guide [3], is expressed as an expanded uncertainty, U. The expanded uncertainty is calculated as $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor. The quantity u_c represents, at the level of one standard deviation, the potential combined effects of the uncertainty arising from instrumental sources, chemical interferences, uncertainties in fundamental constants, and possible material inhomogeneity. The value of k is calculated from the effective degrees of freedom, ν_{eff}. The value $k = 2.026$, corresponding to $\nu_{eff} = 37$, was used to obtain the cited value of U for $w_{C_6H_5COOH}$. The coverage factor was chosen to obtain an approximate 95 % level of confidence. The measurand is total replaceable hydrogen expressed as the mass fraction of C_6H_5COOH. The certified value is metrologically traceable to the SI units for mass, current, and time via the coulometric assay (expressed as percent).

Expiration of Certification: The certification of **SRM 350c** is valid, within the measurement uncertainty specified, until **14 September 2030**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Overall direction and coordination of the technical activities leading to the certification of this SRM were performed by K.W. Pratt of the NIST Chemical Sciences Division. Coulometric analyses were performed by K.W. Pratt. Mass spectrometric determinations of the isotope ratios were performed by the United States Geologic Survey, Reston, VA. The calculation of the molar mass and its uncertainty from the isotope ratios was performed by R.D. Vocke, Jr., of the NIST Chemical Sciences Division.

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 01 December 2015

Robert L. Watters, Jr., Director
Office of Reference Materials

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.

Calculation of Certified Value: The certified value was obtained using the 2014 CODATA (Committee on Data for Science and Technology) recommended value for the Faraday constant, 96 485.332 89 C mol^{-1} [4]. Corrections for air buoyancy were made using a density of 1.301 g cm^{-3} for SRM 350c. The molar mass of benzoic acid in the SRM 350c material, 122.122 14 g mol^{-1}, was determined by mass spectrometric analysis of the SRM 350c material. This experimentally-determined value was used in the calculation of $w_{C_6H_5COOH}$, and its uncertainty is included in the certified uncertainty of $w_{C_6H_5COOH}$. The certified value of $w_{C_6H_5COOH}$ is calculated under the assumption that the replaceable H^+ derives from a substance of net formula $C_7H_6O_2$. No representation is made as to the mass fraction of any impurities present.

INSTRUCTIONS FOR USE

Drying Instructions: Grind a quantity of approximately two grams of SRM 350c in an agate mortar for a period of at least 60 s. The ground material should be a fine, uniform, flour-like powder. Transfer the ground material to a weighing bottle and dry for 2 h at 70 °C. Store the ground, dried material at room temperature (22 °C to 23 °C) in a desiccator over anhydrous magnesium perchlorate. Previous investigations [5] indicate that benzoic acid will not absorb moisture from the atmosphere if the relative humidity does not exceed 90 %.

Stability and Storage: This SRM should be stored in its original bottle at room temperature. It must be tightly re-capped after use and protected from moisture and light.

Homogeneity: Tests indicate that this SRM is homogeneous within the uncertainty limits for sample sizes greater than 200 mg. Samples less than 200 mg are not recommended in order to avoid possible inhomogeneity with smaller sample sizes.

SOURCE AND ANALYSIS[(1)]

The benzoic acid used for this SRM was obtained from a commercial source. The material was examined for compliance with the specification for reagent grade benzoic acid as specified by the American Chemical Society [6]. The material was found to meet or exceed these specifications in all respects.

REFERENCES

[1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G..; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); http://www.nist.gov/srm/publications.cfm (accessed Dec 2015).

[2] Pratt, K.W.; *Automated, High-Precision Coulometry* II. *Strong and Weak Acids and Bases*; Anal. Chim. Acta, Vol. 289, pp. 135–142 (1994).

[3] 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 (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Dec 2015); 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 http://www.nist.gov/pml/pubs/index.cfm (accessed Dec 2015).

[4] 2014 CODATA recommended value for the Faraday constant; available at http://physics.nist.gov/cgi-bin/cuu/Value?f|search_for=faraday (accessed Dec 2015).

[5] SRM 350a; *Benzoic Acid* (*Acidimetric*); NIST, U.S. Department of Commerce: Gaithersburg, MD (1995); available at https://www-s.nist.gov/srmors/certArchive.cfm (accessed Dec 2015).

[6] *Reagent Chemicals*, 8th ed.; American Chemical Society: Washington, DC (1993).

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 http://www.nist.gov/srm.

[(1)] Certain commercial equipment, instruments or materials are identified in this certificate 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.