



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 185i

Potassium Hydrogen Phthalate

pH Standard

This Standard Reference Material (SRM) is intended for use in preparing solutions for calibrating electrodes for pH measuring systems. SRM 185i Potassium Hydrogen Phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) was prepared to ensure high purity and uniformity. However, this SRM is certified **ONLY** as a pH standard [pH(S)] not as a pure substance. A unit of SRM 185i consists of 60 g of potassium hydrogen phthalate.

Certified Values: The certified pH(S) values provided in Table 1 correspond to $\lg(1/a_{\text{H}})$, where a_{H} is the conventional activity of the hydrogen (hydronium) ion referred to the standard state ($p^\circ = 1 \text{ atm} = 1.01325 \times 10^5 \text{ Pa}$) on the scale of molality. The values were derived from potential measurements of cells without liquid junction by the primary measurement method [1,2]. 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 investigated or taken into account [3]. The certified pH(S) values and their expanded uncertainties, U , are stated in Table 1.

Reference Values: The uncertainty [1,4] of the Bates-Guggenheim convention [5] is excluded from the uncertainty calculation for the reference values provided in Table 2. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty [3].

Traceability: The measurand is the pH of the specified buffer solution. The certified values in Table 1 are metrologically traceable to the International System of Units (SI) of amount-of-substance and mass and to the definition of single ion activity. The reference values in Table 2 are traceable to the SI units of amount-of-substance and mass and to the convention [5] used to define the single ion activity.

Expiration of Certification: The certification of **SRM 185i** is valid, within the measurement uncertainty specified, until **15 July 2023**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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) will facilitate notification.

The experimental work leading to the certification of this material was performed by J.F. Waters and K.W. Pratt 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.

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Certificate Issue Date: 08 August 2013

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

INSTRUCTIONS FOR HANDLING, STORAGE AND USE

Storage: SRM 185i is stable when stored in its original container, with the cap tightly closed, in a dry environment, and under normal laboratory temperatures.

Drying Instructions: SRM 185i should be dried for 2 h at 110 °C and stored in a desiccator over anhydrous $\text{Mg}(\text{ClO}_4)_2$ before use.

Source Water for Solution Preparation: The water used in the preparation of the SRM 185i buffer solution need not be protected from atmospheric carbon dioxide, and elaborate precautions for the exclusion of air from the solution are not necessary. Distilled water with an electrolytic conductivity not greater than 2 $\mu\text{S}/\text{cm}$ or water directly obtained from a point-of-use, deionization-based system, of electrolytic conductivity less than 1 $\mu\text{S}/\text{cm}$, may be used, without boiling. The solution should, however, be protected against evaporation and contamination by mold spores.

Preparation of the 0.05 mol/kg Solution: Quantities denoted by m_{W} and associated numerical factors in this paragraph include the effect of air buoyancy, i.e., they correspond to the balance indication in units of mass obtained in the laboratory (the *balance reading*). Weigh by difference approximately 9.8 g of SRM 185i, $m_{\text{W},185\text{i}}$, to an accuracy of 1 mg, into a clean, dry, 1 L polyethylene bottle. Add a quantity of water, equal to 97.887 multiplied by $m_{\text{W},185\text{i}}$, to an accuracy of 0.1 g. Shake thoroughly until the solid has totally dissolved. Gravimetric preparation in this manner eliminates the need to weigh exactly predetermined masses of solid samples. Proportionately smaller quantities of each SRM may be used in this preparation, provided that $m_{\text{W},185\text{i}}$ exceeds 4.0 g.

Stability of Prepared Solution: Solutions should be discarded after one month or sooner if mold or sediment appears.

PREPARATION⁽¹⁾

Source of Material: The potassium hydrogen phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) was obtained from a commercial company. This material conforms to the specifications of the American Chemical Society for primary standard chemicals [6].

Certified Values: The pH(S) and the expanded uncertainty, U , of this solution as a function of temperature are given in Table 1. The uncertainty in the certified value, U , is calculated as $U = ku_c(y)$, where $u_c(y)$ is the “combined standard uncertainty” calculated according to the ISO/JCGM Guide [7]. The value of $u_c(y)$ represents the combined effect of the following uncertainty components associated with the primary measurement method and material homogeneity: extrapolation to obtain the acidity function, $\text{p}(a_{\text{H}}/\text{Cl})^\circ$; standard electrode potentials, E° ; material homogeneity; molality of HCl, b_{HCl} , used for determining E° ; measured cell potentials; correction to the standard pressure for H_2 gas; mean activity coefficient of HCl at b_{HCl} ; gas constant; temperature; Faraday constant; the molality of NaCl; and the uncertainty of the conventional calculation of $\log \gamma_{\text{Cl}}$ (Bates-Guggenheim convention [5]). Current expert opinion [1,4] has assessed the uncertainty attributable to the Bates-Guggenheim convention as 0.010 pH (95 % confidence interval). The value of $u_c(y)$ has been multiplied by a coverage factor, k , obtained by the Student’s t -distribution for effective degrees of freedom at the given temperature and a 95 % confidence level. A solution of molality 0.05 mol/kg is recommended for the calibration of pH measuring systems.

⁽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.

Table 1. Certified pH(S) Values and Expanded Uncertainties (95 % Confidence)

Temperature (°C)	pH(S)	Combined Uncertainty, $u_c(y)$	Coverage Factor, k	Uncertainty, U
5	4.003	0.0050	1.96	0.010
10	3.999	0.0050	1.96	0.010
15	3.999	0.0050	1.96	0.010
20	4.001	0.0050	1.96	0.010
25	4.005	0.0050	1.96	0.010
30	4.012	0.0051	1.96	0.010
35	4.021	0.0051	1.96	0.010
37	4.025	0.0051	1.96	0.010
40	4.031	0.0051	1.96	0.010
45	4.044	0.0051	1.96	0.010
50	4.058	0.0051	1.96	0.010

Reference Values: To attain traceability to the NIST reference pH(S) values for SRM 185i when traceability to the SI units is not necessary, the uncertainty of the Bates-Guggenheim convention is excluded from the uncertainty calculation. The respective pH(S) values in Table 2 are identical to those in Table 1 but are listed to the number of decimal places reported for the expanded uncertainty, U_R :

$$U_R = k_R u_c(\text{measurement}),$$

where k_R is the coverage factor for U_R . The quantities U_R and $u_c(\text{measurement})$ each include all components associated with the measurement method and assessment of material homogeneity, but **DO NOT** include the uncertainty of the Bates-Guggenheim Convention.

Table 2. Reference pH(S) Values and Expanded Reference Uncertainties (95 % Confidence)

Temperature (°C)	pH(S)	Combined Uncertainty, $u_c(\text{measurement})$	Reference Coverage Factor, k_R	Reference Uncertainty, U_R
5	4.0025	0.0005	2.04	0.0011
10	3.9993	0.0004	1.99	0.0008
15	3.9988	0.0004	2.00	0.0008
20	4.0009	0.0004	1.99	0.0008
25	4.0053	0.0004	2.05	0.0007
30	4.0120	0.0010	1.96	0.0020
35	4.0207	0.0010	1.96	0.0020
37	4.0247	0.0011	1.96	0.0021
40	4.0313	0.0011	1.96	0.0022
45	4.0438	0.0011	1.96	0.0021
50	4.0579	0.0011	1.96	0.0022

REFERENCES

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- [7] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), International Bureau of Weights and Measures, Joint Committee for Guides in Metrology, Sèvres Cedex, France (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2013); 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/tn1297/index.cfm> (accessed Aug 2013).

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