



# Certificate of Analysis

## Standard Reference Material<sup>®</sup> 189b

### Potassium Tetroxalate Dihydrate

#### pH Standard

Standard Reference Material (SRM) 189b is intended for use in preparing solutions for calibrating electrodes for pH measuring systems. SRM 189b *Potassium Tetroxalate Dihydrate* [KH<sub>3</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>2</sub>·2H<sub>2</sub>O], meets the specifications of the American Chemical Society for reagent grade material [1]. A unit of SRM 189b consists of a bottle containing 65 g of crystalline material. **NOTE:** These certified pH(S) values apply **only** to solutions prepared from this current lot (b) of **SRM 189b**. This SRM is certified for pH(S) **only** and not for acidimetric purposes.

**Certified pH Values and Uncertainties:** The pH(S) values listed in Table 1 correspond to  $\log(1/a_{\text{H}})$ , where  $a_{\text{H}}$  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 emf measurements of cells without liquid junction by the method of calculation described in references 2 and 3. The expanded uncertainty,  $U = ku_c$ , at the 95 % confidence level, calculated according to the ISO and NIST Guides [4], is less than 0.005 pH units at all temperatures listed. It includes Type B components due to measurements of temperature, pressure, electrode potential, and the gravimetric preparation of standards. The uncertainty also includes Type A components due to the standard deviation of the pH(S) values after smoothing with respect to temperature as described in reference [3], the homogeneity assessment of the SRM material, and the coulometric determination of the molality of HCl used to standardize the electrodes. However, to allow for extra-thermodynamic assumptions in the assignment of pH(S), an expanded uncertainty ( $k = 2$ ,  $df \geq 60$ ) of 0.005 pH units is assigned to the pH(S) values at all temperatures from 0 °C to 50 °C. The composition of the material used for this SRM is homogeneous with respect to the certified pH(S) values. However, it contains a slight excess of potassium hydrogen oxalate as compared to the theoretical formula. The certified pH(S) values of SRM 189b are, therefore, greater than the theoretical values for this material.

A solution of SRM 189b with a molality of 0.05 mol/kg with respect to KH<sub>3</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>2</sub>·2H<sub>2</sub>O is recommended for the calibration of pH measuring systems (See “Calibrations with SRM 189b in pH Cells with Liquid Junction” and “Preparation of the 0.05 mol/kg Solution” sections). The pH(S) of a 0.05 mol/kg solution of SRM 189b as a function of temperature,  $t$ , is presented in Table 1. No value is certified at  $t = 0$  °C due to solubility restrictions of this material [5].

Table 1. Certified pH(S) Values and Uncertainties for SRM 189b

$t/^\circ\text{C}$	pH(S)	$t/^\circ\text{C}$	pH(S)	$t/^\circ\text{C}$	pH(S)
5.0	1.709 ± 0.005	25.0	1.719 ± 0.005	45.0	1.746 ± 0.005
10.0	1.709 ± 0.005	30.0	1.724 ± 0.005	50.0	1.754 ± 0.005
15.0	1.711 ± 0.005	35.0	1.731 ± 0.005		
20.0	1.714 ± 0.005	40.0	1.738 ± 0.005		

**Expiration of Certification:** The certification of SRM 189b is valid until **30 June 2007**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is damaged, contaminated, or modified.

The experimental work leading to the certification of this SRM was performed by K.W. Pratt of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief  
Analytical Chemistry Division

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

The support aspects involved in the issuance of this SRM was coordinated through the NIST Measurement Services Division.

**Source of Material:** The potassium tetroxalate dihydrate was obtained from a commercial supplier and sieved by NIST to remove coarse particles [1].

**Maintenance of Certification:** NIST will monitor representative solutions from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

## INSTRUCTIONS FOR USE

**Drying Instructions:** Use as received. The crystalline material must **NOT** be dried in an oven or placed in a desiccator before use.

**Storage:** The unit should be stored, as received, in its original container with the cap tightly closed, under normal laboratory conditions. The SRM should **NOT** be stored in a desiccator.

**Calibrations with SRM 189b in pH Cells with Liquid Junction:** This SRM is recommended for calibration and for confirmatory purposes in cells with liquid junction. However, the liquid junction potential of the common pH cell displays a greater variability in solutions of pH less than 2.5 than in solutions of pH between 2.5 and 11.5. Hence, the experimental pH in such cells for SRM 189b may differ by 0.02 to 0.05 pH units from the values of pH(S) given in this certificate.

**Preparation of the 0.05 mol/kg Solution:** Gently crush any large lumps of the crystalline material. Transfer 12.717 g (mass in air) of SRM 189b to a flask and dissolve in 1.000 kg (mass in air) of distilled carbon dioxide-free water. Alternatively, if a volumetric apparatus is to be used, transfer 12.584 g (mass in air) to a 1 L volumetric flask, dissolve in distilled or deionized water, and fill to the mark with water at 25 °C. The water should have a conductivity no greater than 2 µS/cm. The solution, being acidic, does not need to be protected with a soda-lime tube. With either method, verify visually that the SRM material has completely dissolved. **NOTE:** The SRM material dissolves slowly. Shaking for 12 h or longer may be necessary to effect complete dissolution.

**Stability of Prepared Solution:** Solutions prepared from SRM 189b are stable for one month. For the highest accuracy, prepare fresh solutions on a weekly basis.

## REFERENCES

- [1] Reagent Chemicals, 8th ed., American Chemical Society: Washington, DC (1993).
- [2] Wu, Y.C.; Koch, W.F.; Marinenko, G.; *A Report on the National Bureau of Standards pH Standards*; J. Res. Natl. Bur. Stand., Vol. 89, p. 395 (1984).
- [3] Wu, Y.C.; Koch, W.F.; Durst, R.A.; *Standard Reference Materials: Standardization of pH Measurements*; NBS Spec. Publ. 260-53 (1988).
- [4] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/>.
- [5] Baucke, F.G.K.; *Lower Temperature Limit of NBS (DIN) pH Standard Buffer Solution Potassium Tetroxalate*; *Electrochimica Acta*, Vol. 24, pp. 95-97 (1979).

<b>Certificate Revision History:</b> 21 December 2005 (This revision reflects an extension of the certification period); 16 August 2000 (Original certificate date).
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*Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet <http://www.nist.gov/srm>.*