National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 84k

Potassium Hydrogen Phthalate

$KHC_8H_4O_4$
Acidimetric Primary Standard

This Standard Reference Material (SRM) is intended for use as a primary acidimetric standard. It consists of highly purified potassium hydrogen phthalate, $KHC_8H_4O_4$ (KHP) and is supplied as crystalline material in a 60 g unit.

Certified Value and Uncertainty: The certified value is based on the results of coulometric assays of dried material (see "Drying Instructions"). The assay value for this material was obtained by automated coulometric titration [1] to the inflection point (pH ca. 8.4) of weighed KHP samples. The certified value represents the result of 19 such titrations of samples from 10 randomly selected bottles from the entire lot of SRM 84k. The value of the Faraday constant used in this work was 96 485.3383 C/mol [2] and 2001 values for the atomic weights [3] were used.

$KHC_8H_4O_4$ (KHP) Assay (mass fraction): 99.9911 % ± 0.0054 %

The uncertainty in the certified value is expressed as an expanded uncertainty, U, at an approximate 95 % level of confidence and is calculated according to the method described in the ISO and ISO Guides [4]. The expanded uncertainty is calculated as $U = ku_c$, where k is the coverage factor and u_c is the combined uncertainty. The calculation of u_c includes the Type A uncertainty of the 19 titrations of the SRM and the Type B uncertainties attributable to the coulometric method.

Information Value: A NIST information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed. The information value is based on the 2001 Atomic Weights [3]. The information value is given to provide additional characterization of the material.

Total Organic Carbon: 47.05 %

Expiration of Certification: The certification of this SRM is valid until **01 July 2010**, 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 contaminated or modified.

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

Coulometric analyses were performed by K.W. Pratt of the NIST 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 were coordinated through the NIST Standard Reference Materials Program by C.S. Davis of the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 13 May 2005
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Office of Measurement Services

NOTICE AND WARNINGS TO USERS

Storage: This SRM should be stored in its original bottle at temperatures between approximately 20 ºC and 25 ºC. It must be tightly recapped after use and protected from moisture and light.

INSTRUCTIONS FOR USE

Drying Instructions: Dry at 120 ºC for 2 h and store over anhydrous magnesium perchlorate [$Mg(ClO_4)_2$] in a desiccator. The SRM should not be ground or crushed before or after drying. Any large lumps that may develop on storage should be broken up by shaking the bottle prior to withdrawing the SRM sample for drying.

Inappropriate Use: This SRM is certified for acidimetric assay **ONLY** and is not intended for use in pH standardizations.

Homogeneity: Tests indicate that this SRM is homogeneous within the uncertainty limits for sample sizes greater than 500 mg. Samples less than 500 mg are not recommended in order to avoid possible inhomogeneity with smaller sample sizes.

SOURCE AND ANALYSIS

Source of Material: The material used for this SRM was obtained from the Sigma-Aldrich Fine Chemicals[1] (St. Louis, MO). The material was examined for compliance with the specification for reagent grade KHP as specified by the American Chemical Society [5]. The material was found to meet or exceed these specifications in all respects.

REFERENCES

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

[2] Mohr, P.J.; Taylor, B.N.; *The Fundamental Physical Constants*; J. Phys. Chem. Ref. Data, Vol. 28(6), pp. 1713-1852 (1999); Reviews of Modern Physics, Vol. 72(2), pp. 351–495 (2000).

[3] IUPAC Commission on Atomic Weights and Isotopic Abundances; *Atomic Weights of the Elements 2001*; Pure and Appl. Chem., Vol. 75(8), pp. 1107–1122 (2003).

[4] ISO; *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st ed., ISO, 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] *Reagent Chemicals*, 8th ed.; American Chemical Society: Washington, DC (1993).

Certificate Revision History: 13 May 2005 (This revision reflects an extension of the certification period); 23 November 1999 (Original certificate date).

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; or via the Internet at http://www.nist.gov/srm.

[1] Certain commercial equipment, instruments, or materials are identified to specify adequately the 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.