National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 84k

Potassium Hydrogen Phthalate

$KHC_8H_4O_4$
Acidimetric Primary Standard

Standard Reference Material (SRM) 84k 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 96485.309 C/mol [2] and 1995 values for the atomic weights [3] were used.

$KHC_8H_4O_4$ (KHP) Assay, Mass Fraction (in %): 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 Guide [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: The theoretical total organic carbon (TOC) content is 47.05 %, based on the 1995 Atomic Weights [3]. This is a noncertified value with no reported uncertainty, as there is insufficient information to assess the uncertainty. The information value is given to provide additional characterization of the material.

Expiration of Certification: The certification of this SRM is valid until **01 July 2005**, 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: If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card 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 preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.C. Colbert.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 23 November 1999

Thomas E. Gills, Director
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.

Drying Instructions: Dry at 120 ºC for 2 h and store over anhydrous $Mg(ClO_4)_2$ 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. **289**, pp. 135-142, (1994).

[2] Cohen, E.R. and Taylor, B.N., J. Res. Nat. Bur. Stand. **92(2)**, pp. 85-95, (1987).

[3] IUPAC Commission on Atomic Weights and Isotopic Abundances, "Atomic Weights of the Elements 1995," Pure and Appl. Chem. **68**, p. 2339, (1996).

[4] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and 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).

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 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.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.