National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 84L

Potassium Hydrogen Phthalate

$KHC_8H_4O_4$
Acidimetric Primary Standard

This Standard Reference Material (SRM) is intended for use as an acidimetric primary standard. It consists of highly purified potassium hydrogen phthalate (KHP), $KHC_8H_4O_4$. A unit of SRM 84L is supplied as crystalline material in a 60 g unit.

Certified Value: The certified value listed in Table 1 is the mass fraction of total acid (replaceable H^+) expressed as KHP. 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].

Table 1. Certified Value [(a)] for SRM 84L Potassium Hydrogen Phthalate

99.9934 % ± 0.0076 %

[(a)] The certified value is expressed as the value ± its expanded uncertainty, U. The expanded uncertainty is calculated as $U = ku_c$, where k is the coverage factor and u_c is the combined standard uncertainty calculated according to the ISO Guide [2]. The value of u_c represents the combined uncertainty in the certified value, at the level of one standard deviation, and includes the replication uncertainty of the 30 titrations of the SRM and all sources of uncertainty inherent to the coulometric method. The value of k controls the approximate level of confidence associated with U. For this SRM, $k = 2.04$. This value corresponds to a level of confidence of approximately 95 %. The value of k is obtained from the Student's t-distribution with effective degrees of freedom, $\nu_{eff} = 31$.

Information Value: The theoretical total organic carbon (TOC) content is 47.05 %, based on the 2005 Atomic Weights [3]. This TOC value is a noncertified value with no reported uncertainty, as there is insufficient information to assess the uncertainty. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [1].

Expiration of Certification: The certification of **SRM 84L** is valid, within the measurement uncertainties specified, until **01 April 2024**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). This 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 this notification.

The overall direction and coordination of technical measurements leading to certification was provided by T.W. Vetter of the NIST Analytical Chemistry Division.

Coulometric analyses were performed by K.W. Pratt of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 01 February 2010

Robert L. Watters, Jr., Chief
Measurement Services 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 Measurement Services Division.

INSTRUCTIONS FOR USE

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

Homogeneity: Tests indicate that this SRM is homogeneous within the uncertainty limits for test portions with a mass greater than 300 mg. Test portions with a mass less than 300 mg are not recommended, to avoid possible inhomogeneity.

Drying Instructions: As issued, SRM 84L contains some entrapped (occluded) water that is released by a combination of grinding and drying. The following procedure was used in the certification of this material and must be followed to attain the certified value. Grind a test portion to a fine, flour-like powder, taking special care to fragment the larger crystals. Dry the powder at 120 ºC for 2 h and store over anhydrous magnesium perchlorate in a desiccator.

The SRM 84L material was ground by hand for a period of 60 s to 90 s in an agate mortar for this certification. Other methods that do not introduce contaminants may also yield a suitable powder.

Analyses of dried, unground test portions of SRM 84L yielded non-certified values from 99.94 % to 99.99 %, owing to variations in the frequency or size of inclusions of water in the KHP crystals in the given test portion. Such inclusions are well known for KHP [4,5] and a mass fraction of occluded water in the range of 0.01 % to 0.15 % was present in previous issues of this SRM [4].

Intended Use: This SRM is certified for acidimetric assay **ONLY** and is not intended for use as a pH standard. The current issue of SRM 185 Potassium Hydrogen Phthalate, pH Standard, is certified for pH.

SOURCE, PREPARATION, AND ANALYSIS[1]

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

Assay Technique: The certified value is based on the results of coulometric assays of ground and dried material (see Drying Instructions). The assay value for this material was obtained by automated coulometric titration [7] to the inflection point (pH ≈ 8.4) of weighed test portions of KHP. The certified value represents the result of 30 titrations of test portions taken from 16 bottles selected by stratified random sampling from the entire lot of SRM 84L. The value of the Faraday constant used in this work was 96 485.3399 C/mol [8]. The 2005 values for the atomic weights [3] were used.

[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.

REFERENCES

[1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm.

[2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf; 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/.

[3] Wieser, M.E.; *Atomic Weights of the Elements 2005*, IUPAC Technical Report; Pure Appl. Chem., Vol. 78 Issue (11), pp. 2051-2066 (2006).

[4] Certificates for SRM 84a (March 7, 1938) through SRM 84h (September 27, 1966); http://ts.nist.gov/MeasurementServices/ReferenceMaterials/archived_certificates/archived_certificates.htm.

[5] Ester, G.R.; Price, R.; Halfpenny, P.J.; *The Relationship Between Crystal Growth and Defect Structure: A Study of Potassium Hydrogen Phthalate Using X-Ray Topography and Atomic Force Microscopy*; J. Phys. D: Appl. Phys., Vol. 32, pp. A128-A132 (1999).

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

[7] 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).

[8] Mohr, P.J.; Taylor, B.N.; Newell, D.B.; *CODATA Recommended Values of the Fundamental Physical Constants: 2006*; Rev. Mod. Phys., Vol. 80(2), pp. 633-730 (2008); available at http://physics.nist.gov/cgi-bin/cuu/Value?f|search_for=faraday.

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) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.