

Standard Reference Material[®] 84L

Potassium Hydrogen Phthalate



Acidimetric Primary Standard

CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended for use as an acidimetric primary standard.

Description: SRM 84L consists of highly purified potassium hydrogen phthalate (KHP), $\text{KHC}_8\text{H}_4\text{O}_4$. 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 Mass Fraction Value^(a,b) for SRM 84L

99.9934 % \pm 0.0076 %

^(a) The measurand is expressed as the mass fraction of Potassium Hydrogen Phthalate. Metrological traceability of the measurand is to the International System of Units (SI) derived units for mass fraction, expressed as a percentage, and is established through the Faraday constant, the molar mass of KHP, and to the SI-base units for electric current, time, and mass through calibrations maintained by NIST.

^(b) The certified value is expressed as a value and its expanded uncertainty, U . The expanded uncertainty is calculated as $U = k u_c$, where k is the coverage factor and u_c is the combined standard uncertainty calculated according to the ISO/JCGM 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, $\nu_{\text{eff}} = 31$.

Additional Information: A value of potential interest and additional information are provided in Appendix A.

Period of Validity: The certified values delivered by SRM 84L are valid within the measurement uncertainty specified until **01 April 2029**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (<https://www.nist.gov/srm>) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

Safety: FOR RESEARCH USE, see attached SDS for additional information.

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.

Use: 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, due to possible inhomogeneity.

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 by hand for a period of 60 s to 90 s in an agate mortar to a fine, uniform, flour-like powder, taking special care to fragment the larger crystals. Other methods that do not introduce contaminants may also yield a suitable powder. Dry the ground powder at 120 °C for 2 h and store in a desiccator over anhydrous magnesium perchlorate.

Analyses of dried, unground test portions of SRM 84L yielded assay 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 [3,4] and a mass fraction of occluded water in the range of 0.01 % to 0.15 % was present in previous issues of this SRM [3].

This SRM is certified for acidimetric assay **ONLY** and is not intended for use as a pH standard.

REFERENCES

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- [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 https://physics.nist.gov/cgi-bin/cuu/Value?f|search_for=faraday (accessed Feb 2024).

Certificate Revision History: 05 February 2024 (Editorial changes); 25 January 2024 (Change of period of validity; updated format; editorial changes); 01 February 2010 (Original certificate issue date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at <https://www.nist.gov/srm>.

***** End of Certificate of Analysis *****

APPENDIX A

Value of Potential Interest: The theoretical total organic carbon (TOC) content is 47.05 %, based on the 2005 Atomic Weights [5]. This TOC value is considered to be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [1]. Values of potential interest cannot be used to establish metrological traceability.

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 'Use' section above). The assay value for this material was obtained by automated coulometric titration [7] to the inflection point ($\text{pH} \approx 8.4$) 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 relative atomic masses [5] were used.

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