

Certificate of Analysis

Standard Reference Material® 200b

Potassium Dihydrogen Phosphate (Fertilizer Standard)

This Standard Reference Material (SRM) is a highly purified and homogeneous lot of crystalline potassium dihydrogen phosphate (KH_2PO_4). It is intended primarily for use as a working standard in the calibration and standardization of procedures employed in the fertilizer industry for the determination of potassium and phosphorus. A unit of SRM 200b consists of one bottle containing 90 g of crystalline potassium dihydrogen phosphate.

Certified Values: The certified mass fractions of phosphorus and potassium, presented in Table 1, are based on assays of dried material (see "Drying Instructions"), including the effects of air buoyancy. A NIST certified value represents data for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or taken into account [1]. Certified values are based on results obtained by a single primary method with confirmation by other methods, or with two or more critically evaluated independent methods [1]. The certified value for phosphorus is based on the results of the coulometric acidimetric assay of phosphate as phosphorus in SRM 200b and on the relative atomic mass (atomic weight) of phosphorus [2]. The value of the Faraday constant used in this work was 96 485.339 9 C/mol [3]. The certified value for phosphorus represents the results of determinations from 13 randomly selected bottles from the entire lot of SRM 200b. Potassium was determined gravimetrically by separation of potassium and conversion to potassium sulfate (K₂SO₄). The certified value for potassium, sulfur, and oxygen [2], with corrections for contaminants and possible transfer losses. The certified value for potassium represents the result of determinations from six randomly selected bottles from the entire lot of SRM 200b.

Information Values: The information values for impurities shown in Table 2 are noncertified values with no reported uncertainties. 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 [1]. The information values are given to provide additional characterization of the material only. These values should not be used to monitor or assess analytical performance. The information values are reported as mass fractions. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 200b** is valid, within the measurement uncertainty specified, until **30 September 2029**, provided the SRM is handled and stored 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 or register online) will facilitate notification.

Overall direction and coordination of technical measurements leading to certification were provided by T.W. Vetter of the NIST Chemical Sciences Division.

Coulometric analyses were performed by K.W. Pratt of the NIST Chemical Sciences Division. Gravimetric analyses were performed by T.W. Vetter.

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

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Steven J. Choquette, Director Office of Reference Materials

Gaithersburg, MD 20899 Certificate Issue Date: 26 January 2017 *Certificate Revision on Last Page* Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR USE

Drying Instructions: Dry for 2 hours at 110 °C. Store the dried material over a desiccant such as Mg(ClO₄)₂.

Storage: The SRM should be stored, as received, in its original container with the cap tightly closed, under normal laboratory conditions.

SOURCE AND ANALYSIS⁽¹⁾

The potassium dihydrogen phosphate was obtained from a commercial supplier. The material was examined and was found to meet or exceed the American Chemical Society specification for reagent-grade potassium dihydrogen phosphate [5] in all respects. A commercial laboratory analyzed the material for impurities using glow discharge mass spectrometry.

Homogeneity: Tests indicate that this SRM is homogeneous within the uncertainty limits for test portions with a mass greater than 350 mg. Use of test portions with a mass less than 350 mg is not recommended, to avoid possible inhomogeneity.

Table 1. Certified Mass Fractions^(a) of Phosphorus and Potassium

Phosphorus (P) 22.769 % ± 0.010 % Potassium (K) 28.735 % ± 0.018 %

^(a) The certified value is expressed as the value \pm 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/JCGM Guide [4]. The value of u_c represents the combined uncertainty in the certified value, at the level of one standard deviation. The value of *k* controls the approximate level of confidence associated with *U*, which, for this SRM, is approximately 95 %. The value of *k* is obtained from the Student's *t*-distribution using the effective degrees of freedom, v_{eff} . For P, $v_{eff} = 20$ and k = 2.09; for K, $v_{eff} = 2.56$ and k = 3.52. The uncertainty of the acidimetric assay for P includes components for measurement replication, carbon dioxide interference, sample carryover, calibration (current and time), current efficiency, mass determination, relative atomic masses, and the Faraday constant. The uncertainty of the gravimetric assay for K includes components for measurement replication, ignition temperature, contaminants, possible transfer loss, and relative atomic masses. The measurands are the total mass fractions of phosphorus and potassium and the certified value is metrologically traceable to the SI for mass fraction, expressed as percent.

Table 2. Information Values for Selected Elements

Element	(mg/kg)	Element	(mg/kg)	Element	(mg/kg)	Element	(mg/kg)
Aluminum (Al)	2	Europium (Eu)	< 0.1	Mercury (Hg)	<1	Sodium (Na)	18
Antimony (Sb)	1	Fluorine (F)	4	Molybdenum (Mo)	< 0.5	Strontium (St)	< 0.5
Arsenic (As)	1.5	Gadolinium (Gd)	< 0.5	Neodymium (Nd)	< 0.1	Sulfur (S)	13
Barium (Ba)	< 0.1	Gallium (Ga)	<5	Nickel (Ni)	0.75	Tantalum (Ta)	<5
Beryllium (Be)	< 0.1	Germanium (Ge)	<5	Niobium (Nb)	< 0.5	Tellurium (Te)	< 0.5
Bismuth (Bi)	< 0.1	Gold (Au)	<1	Osmium (Os)	< 0.5	Terbium (Tb)	< 0.1
Boron (B)	0.2	Hafnium (Hf)	< 0.5	Palladium (Pd)	< 0.5	Thallium (Tl)	< 0.5
Bromine (Br)	<10	Holmium (Ho)	< 0.1	Platinum (Pt)	< 0.5	Thorium (Th)	< 0.05
Cadmium (Cd)	<1	Indium (In)	< 0.1	Praseodymium (Pr)) <0.1	Thulium (Tm)	< 0.1
Calcium (Ca)	16	Iodine (I)	< 0.5	Rhenium (Re)	< 0.5	Tin (Sn)	<1
Cerium (Ce)	< 0.1	Iridium (Ir)	< 0.1	Rhodium (Rh)	< 0.1	Titanium (Ti)	0.3
Cesium (Cs)	< 0.1	Iron (Fe)	8	Rubidium (Rb)	3.5	Tungsten (W)	< 0.5
Chlorine (Cl)	7.5	Lanthanum (La)	< 0.05	Ruthenium (Ru)	< 0.5	Uranium (U)	< 0.05
Chromium (Cr)	2.7	Lead (Pb)	< 0.1	Samarium (Sm)	< 0.1	Vanadium (V)	< 0.1
Cobalt (Co)	< 0.2	Lithium (Li)	< 0.1	Scandium (Sc)	< 0.5	Ytterbium (Yb)	< 0.5
Copper (Cu)	0.9	Lutetium (Lu)	< 0.1	Selenium (Se)	<5	Yttrium (Y)	< 0.5
Dysprosium (Dy	y) <0.1	Magnesium (Mg)	4	Silicon (Si) <	<100	Zinc (Zn)	2.5
Erbium (Er)	< 0.1	Manganese (Mn)	< 0.4	Silver (Ag)	<1	Zirconium (Zr)	< 0.1

⁽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. SRM 200b Page 2 of 3

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.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at: http://www.nist.gov/srm/publications.cfm (accessed Jan 2017).
- Wieser, M.E.; Atomic Weights of the Elements 2005, IUPAC Technical Report; Pure Appl. Chem., Vol. 81 Issue 11, pp. 2131–2156 (2009).
- [3] Mohr, P.J. and Taylor, B.N.; CODATA Recommended Values of the Fundamental Physical Constants: 2002; Reviews of Modern Physics, Vol. 77 Issue 1, pp. 1–107 (2005).
- [4] JCGM 100:2008; Evaluation of Measurement Data Guide to the Expression of Uncertainty in Measurement; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jan 2017); 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://www.nist.gov/pml/pubs/index.cfm (accessed Jan 2017).
- [5] ACS; Reagent Chemicals, 9th ed., American Chemical Society, Washington, DC (1999).

Certificate Revision History: 26 January 2017 (Editorial changes); 03 May 2010 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.