National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 194a

Ammonium Dihydrogen Phosphate

This Standard Reference Material (SRM) is a highly purified and homogeneous lot of crystalline ammonium dihydrogen phosphate ($NH_4H_2PO_4$). 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 ammoniacal nitrogen and phosphorus. A unit of SRM 194a consists of one bottle containing 90 g of crystalline ammonium dihydrogen phosphate.

Certified Mass Fraction Values: Certified values for nitrogen and phosphorus, expressed as mass fractions [1] on a dry-mass basis, are provided in Table 1. 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 [2]. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories.

Information Mass Fraction Values: Information values for impurities are shown in Table 2. These values are noncertified mass fraction 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 [2]. 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. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 194a** is valid, within the measurement uncertainty specified, until **28 February 2049**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and 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 notification.

Coordination of the technical measurements leading to certification of SRM 194a was provided by T.W. Vetter of the NIST Chemical Sciences Division.

Analytical measurements leading to certification were made by R.G. Brennan, T.A. Butler, A.F. Marlow, M.M. Phillips, K.W. Pratt, J.R. Sieber, and M.R. Winchester of the NIST Chemical Sciences Division. External inter-laboratory analyses were coordinated by J.H. Falls of CF Industries, Inc. in Plant City, FL.

Statistical analyses were performed by W.F. Guthrie of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Certificate Issue Date: 12 March 2014

Robert L. Watters, Jr., Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Sampling: Before it is sampled, the unit should be thoroughly mixed by carefully inverting and rotating the tightly sealed bottle. To relate analytical determinations to the certified values in this Certificate of Analysis, a minimum test portion of 100 mg should be used. The SRM should be stored in its original bottle, tightly sealed, under normal laboratory conditions.

Drying Instructions: Dry at 110 °C for 2 h. Store the dried material over a desiccant such as anhydrous $Mg(ClO_4)_2$.

SOURCE, PREPARATION, HOMOGENEITY, AND ASSESSMENT[(1)]

The ammonium 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 ammonium dihydrogen phosphate [3] in all respects. A commercial laboratory analyzed the material for impurities using inductively coupled plasma mass spectrometry and spark source mass spectrometry.

Homogeneity Testing: Fifteen bottles of SRM 194a were selected for homogeneity assessment. Duplicate test portions from each bottle were analyzed by wavelength-dispersive X-ray fluorescence spectrometry (WDXRF) for nitrogen and phosphorus. Based on the results of one-way analyses of variance (ANOVA) F-tests for a bottle effect, there is insufficient evidence at the 0.05 significance level to claim a difference between one or more of the bottle means, which is consistent with material homogeneity. Additional homogeneity testing using combustion with thermal conductivity detection for nitrogen was performed on test portions of these same 15 bottles. Although there was no evidence of significant material heterogeneity, an uncertainty component for heterogeneity that reflects the current knowledge of its potential magnitude given the available data was incorporated into the expanded uncertainty for each constituent. The uncertainty component calculated for phosphorus heterogeneity was based on the results of WDXRF analyses, and for nitrogen heterogeneity was based on the results of combustion with thermal conductivity detection.

VALUE ASSIGNMENT

Certified values are based on weighted means of three assessments of the mass fraction for each constituent made using analyses performed at NIST and collaborating laboratories. The weighted means were calculated using the DerSimonian-Laird procedure [4], which weights the results from each method in proportion to the inverse of the square of its standard uncertainty and includes estimates of both between- and within-method uncertainties in the weights. The expanded uncertainty listed with these values is expressed as $U = ku_c$ at approximately the 95 % level of confidence, where k is a coverage factor used to control the level of confidence, and u_c is intended to represent, at the level of one standard deviation, the combined effect of all between- and within-method components of measurement uncertainty and material heterogeneity. Within-method uncertainties for each method were assessed using the methods described in the Guide to the Expression of Uncertainty in Measurement [5]. The uncertainty of the certified value was then computed using the methods in Supplement 1 to the Guide to the Expression of Uncertainty in Measurement [6].

Certified Mass Fraction Values: The certified values and their corresponding expanded uncertainties are given in Table 1. The certified value and expanded uncertainty for nitrogen are based on measurements by gravimetric titrimetry, ion chromatography (IC), and an inter-laboratory study, as well as material homogeneity testing. The certified value and expanded uncertainty for phosphorus are based on measurements by inductively coupled plasma optical emission spectrometry (ICP-OES), IC, and an inter-laboratory study, as well as material homogeneity testing. The coverage factor, k, is 2. The measurand is the total mass fraction of the element listed. The certified values are metrologically traceable to the SI unit of grams per 100 grams, expressed as percent.

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for SRM 194a

Element	Mass Fraction (%)
Nitrogen	12.130 ± 0.047
Phosphorus	26.93 ± 0.14

[(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.

Table 2. Information Mass Fraction Values for Selected Elements in SRM 194a

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Aluminum	0.3	Fluorine	<5	Neodymium	<0.01	Sulfur	<10
Antimony	0.01	Gadolinium	<0.01	Nickel	0.8	Tantalum	<0.05
Arsenic	0.4	Gallium	<0.05	Niobium	<0.05	Tellurium	<0.1
Barium	<0.05	Germanium	<0.05	Osmium	<0.01	Terbium	<0.01
Beryllium	<0.01	Gold	<0.05	Palladium	<0.1	Thallium	<0.01
Bismuth	<0.01	Hafnium	<0.05	Platinum	<0.01	Thorium	<0.01
Boron	6	Holmium	<0.01	Potassium	22	Thulium	<0.01
Bromine	<5	Indium	<5	Praseodymium	<0.01	Tin	0.01
Cadmium	<0.1	Iodine	<10	Rhenium	<0.01	Titanium	1
Calcium	1	Iridium	<0.01	Rhodium	<0.01	Tungsten	<0.05
Cerium	<0.01	Iron	2	Rubidium	<0.05	Uranium	<0.01
Cesium	<0.01	Lanthanum	<0.01	Ruthenium	<0.01	Vanadium	<0.01
Chlorine	9	Lead	<0.01	Samarium	<0.01	Ytterbium	<0.01
Chromium	0.3	Lithium	<0.01	Scandium	<0.3	Yttrium	≤0.1
Cobalt	<0.05	Lutetium	<0.01	Selenium	<0.1	Zinc	0.2
Copper	0.4	Magnesium	0.3	Silicon	25	Zirconium	0.2
Dysprosium	<0.01	Manganese	0.2	Silver	<0.1		
Erbium	<0.01	Mercury	<0.05	Sodium	6		
Europium	<0.01	Molybdenum	0.2	Strontium	0.1		

REFERENCES

[1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at http://www.nist.gov/pml/pubs/index.cfm/ (accessed Mar 2014).

[2] May, W.; Parris, R.; Beck, 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 (2000); available at http://www.nist.gov/srm/publications.cfm (accessed Mar 2014).

[3] ACS; *Reagent Chemicals*, 10th ed., American Chemical Society, Washington, DC (2006).

[4] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; Controlled Clin. Trials, Vol. 7, pp. 77–188 (1986).

[5] 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 (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Mar 2014); 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/guidelines/TN1297/tn1297s.pdf (accessed Mar 2014).

[6] JCGM 101:2008; *Evaluation of Measurement Data - Supplement 1 to the "Guide to the Expression of Uncertainty in Measurement" - Propagation of Distributions Using a Monte Carlo Method*; Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Mar 2014).

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.