National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3185

Nitrate Anion Standard Solution

Lot No. 991508

This Standard Reference Material (SRM) is intended primarily for use in anion ion chromatography or any other analytical technique that requires aqueous standard solutions for calibration or as control samples. One unit of SRM 3185 consists of five 10-milliliter sealed borosilicate glass ampoules of a single component solution prepared gravimetrically to contain a nominal 1000 mg/kg of nitrate dissolved in filtered (0.22 μm), deionized (18 MΩ·cm) water.

Certified Value of Nitrate: 976 mg/kg ± 6 mg/kg

The certified value is based on (1) gravimetric preparation using high purity sodium nitrate, and (2) ion chromatography calibrated using three independently prepared gravimetric solutions.

The uncertainty in the certified value is calculated as

$$U = (ku_c + B) \text{ mg/kg}$$

where k is the coverage factor, u_c is the combined standard uncertainty, and B is an allowance for method bias. The coverage factor, $k = 2.00$, is the Student's t-value for a 95 % confidence interval. The combined standard uncertainty, u_c, has been calculated according to the ISO and NIST Guides [1] and is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with gravimetric preparation and the chromatographic measurement. The allowance for method bias, B, has been calculated according to the procedure of Schiller and Eberhardt for combining independent analytical methods [2].

Expiration of Certification: The certification of **SRM 3185 Lot No. 991508** is valid, within the measurement uncertainty specified, until **30 March 2009**, 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 modified.

Maintenance of Certification: NIST will monitor representative solutions from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the technical measurements for certification of this SRM were performed by F.R. Guenther of the NIST Analytical Chemistry Division.

This SRM was prepared gravimetrically and analyzed using ion chromatography by J.M. Smeller of the NIST Analytical Chemistry Division.

Statistical consultation was provided by K.R. Eberhardt of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by C.S. Davis of the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 25 April 2005
See Certificate Revision History on Last Page

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

INSTRUCTIONS FOR USE

CAUTION: This SRM is an aqueous solution contained in tip-sealed borosilicate glass ampoules with prescored stems. Therefore, all appropriate safety precautions, including use of gloves during handling, should be taken to avoid accidental breakage or spillage. Unopened ampoules should be stored under normal laboratory conditions in an upright position inside the original container supplied by NIST.

Opening an Ampoule: When an ampoule is to be opened, that area of the stem where the prescored band is located (~5 mm below the encircling metallic band) should be carefully wiped with a clean, damp cloth and the body of the ampoule wrapped in absorbent material. Then holding the ampoule steady and with thumb and forefinger grasping the stem at the metallic band, **minimal** thumb pressure should be applied to the stem to snap it. Correctly done, the stem should break easily where prescored. Use of a metal file to break the stem is **NOT** recommended.

Working Standard Solutions: After opening the ampoule, the entire contents must be transferred immediately to another container before *working standard solutions* are prepared. Working standard solutions in the range of 10 mg/kg to 100 mg/kg, from which more dilute standards can be prepared, are recommended. The user should establish internal laboratory procedures that specify a maximum shelf life for a working standard solution. Two procedures for the preparation of working standard solutions follow.

Preparation of Working Standard Solutions by Mass: Each working standard solution should be prepared by emptying one or more ampoules of the SRM into an empty, dry, preweighed polyethylene bottle, and then re-weighing the bottle. Deionized water must be added by mass to bring the solution to the desired dilution. The dilution need not be exact since the mass of the empty bottle, mass of the bottle plus SRM aliquot, and the final diluted mass of the solution will permit calculation of the exact mass fraction of the working standard solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true mass fraction in vacuum. The working standard solution will be specified in mass fraction units (mg/kg, mass of phosphate per mass of solution). Volumetric dilutions are **NOT** recommended due to uncertainties in volume calibrations and variations in density. However, for user convenience, a procedure for volumetric preparation that will minimize the major sources of error is given below.

Preparation of Working Standard Solutions by Volume: Each working standard solution should be prepared by emptying one or more ampoules of the SRM into an empty, dry polyethylene bottle and then weighing the bottle. The solution must now be transferred to a Class A volumetric flask and the polyethylene bottle re-weighed to determine the exact mass of SRM solution transferred. The solution in the flask is then diluted to 99 % + volume using deionized water, mixed thoroughly, and the remaining few drops needed to dilute to exact volume carefully added. The concentration (in mg/mL) of the resulting working standard solution can then be calculated by multiplying the mass (in kg) of the SRM solution amount by the SRM certified value (in mg/kg) and dividing the numerical product by the calibrated volume (in mL) of the flask used for dilution. If this procedure is followed, no correction for density is needed, and although the concentration of the resulting working standard solution may be an uneven fraction of the original SRM certified value, it will be known as accurately as a volumetric dilution permits.

REFERENCES

[1] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); 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/.

[2] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results From Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105; p. 571 (2000).

Certificate Revision History: 25 April 2005 (This technical revision reflects an extension in the expiration date); 08 December 2003 (This technical revision reflects an extension in the expiration date.); 24 January 2000 (Original certificate date).

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