National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3186

Phosphate Anion Standard Solution

Lot No. 791209

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 3186 consists of 50 mL of a single component solution prepared gravimetrically to contain a nominal 1000 mg/kg of phosphate dissolved in filtered (0.22 μm) 18 MΩ water.

Certified Value (Y) of Phosphate: 1002 mg/kg ± 8 mg/kg at 22 °C

The certified value (Y) is based on: (1) gravimetric preparation using SRM 186Id Potassium Dihydrogen Phosphate, and (2) ion chromatography calibrated using two independently prepared gravimetric solutions. The certified value has been adjusted upward by 0.1 % relative to compensate for the effect of transpiration losses of solvent through the container walls and packaging during the period of validity of the *unopened* SRM. *No correction has been applied for transpiration that will occur after the SRM bottle has been removed from the sealed bag.* See "Instructions for Use" for more information regarding transpiration.

The uncertainty in the certified value is calculated as

$$U = (2u_c + 0.001Y + B) \text{ mg/kg}$$

where u_c is the "combined standard uncertainty" calculated according to the ISO Guide [1] and the procedure of Schiller and Eberhardt for combining independent analytical methods [2]. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with gravimetric factors, as well as the purity of the starting material and the uncertainty associated with the analytical method. The quantity, $0.001Y$, is an allowance for transpiration of the solution through the container walls and packaging, which is estimated to be ± 0.1 % of the certified value. The quantity, B, is an allowance for between method differences.

Expiration of Certification: The certification of **SRM 3186 Lot No. 791209** is valid, within the measurement uncertainty specified, until **01 October 2000**, 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. Return of the attached registration card will facilitate notification.

This SRM was prepared gravimetrically by T.A. Butler 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 preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by N.M. Trahey.

Willie E. May, Chief
Analytical Chemistry Division

Thomas E. Gills, Director
Office of Measurement Services

Gaithersburg, MD 20899
Certificate Issue Date: 01 October 1999
See Certificate Revision History on Last Page

INSTRUCTIONS FOR USE

Transpiration: After the SRM has been removed from the aluminized bag, transpiration will occur at an accelerated rate resulting in a gradual increase in the phosphate mass fraction. It is the responsibility of the user to account for this effect. The recommended way to reduce the effects of transpiration is to begin the preparation of all working standard solutions by delivering weighed aliquots of the SRM to appropriate vessels as soon as the SRM is removed from the aluminized bag. The aliquots may now be stored and can be diluted to known mass or volume at a later date. Storage of a partially used SRM bottle is **NOT** recommended; however, if such storage is necessary, the cap should be tightly sealed and the SRM bottle kept in an airtight container to slow the rate of transpiration. When the bottle is weighed both before and after being placed in storage, the mass difference observed will be a measure of transpiration mass loss. The user should set a maximum shelf life *for a partially used SRM bottle* commensurate with accuracy requirements.

Preparation of Working Standard Solutions by Mass: Each diluted working standard solution should be prepared by transferring an aliquot of the SRM to an empty, dry, preweighed polyethylene bottle, and then reweighing the bottle. 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 concentration of the working solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true concentration in vacuum. Dilute SRM solution concentration will be in mg/kg units. 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 diluted working standard solution should be prepared by transferring an aliquot of the SRM to 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 reweighed to determine the exact mass of SRM solution transferred. The solution in the flask is then diluted to 99 % + of volume, mixed thoroughly, and the remaining few drops needed to dilute to exact volume carefully added. The concentration (in mg/mL) of the resulting diluted 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 the analyst follows this procedure, 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 concentration, it will be known as accurately as a volumetric dilution permits.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and 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] Schiller, S.B. and Eberhardt, K.R., "Combining Data from Independent Chemical Analysis Methods," Spectrochimica Acta, **46B**, No. 12, pp. 1607-1613, (1991).

Certificate Revision History: 22 September 99 (This revision reflects a change in the certification period); 14 October 97 (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 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.