



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3183

Fluoride Anion Standard Solution

Lot No. 991510

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 3183 consists of 50 mL of a single component solution in a high density polyethylene bottle sealed in an aluminized Mylar¹ bag. The solution is prepared gravimetrically to contain a nominal 1000 mg/kg of fluoride dissolved in filtered (0.22 μm) 18 M Ω water.

Certified Value (Y) of Fluoride: 1000 mg/kg \pm 4 mg/kg at 22 °C

The certified value (Y) is based on: (1) gravimetric preparation using high purity sodium fluoride, and (2) ion chromatography calibrated using four independently prepared gravimetric solutions. The certified value has been adjusted upward by 0.08 % 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

$$U = (ku_c + 0.0008Y + B) \text{ mg/kg}$$

where k is the coverage factor, u_c is the combined standard uncertainty, $0.0008Y$ is the uncertainty of the transpiration correction, 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 Guide [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 maximum increase in concentration due to transpiration predicted to occur in unopened bottles of the SRM over the period of validity is 0.16 %. Accordingly, a correction of $+ 0.0008Y$ has been applied, with an uncertainty of $\pm 0.0008Y$. 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 3183 Lot No. 991510** is valid, within the measurement uncertainty specified, until **30 March 2005**, provided the SRM is handled in accordance with instructions given in this certificate (see “Instructions for Use”). This expiration date extends the original expiration for **Lot No. 991510**. The certified uncertainty is valid only if the aluminized Mylar¹ bag and the bottles have **NOT** been previously opened. 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.

Willie E. May, Chief
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Gaithersburg, MD 20899
Certificate Issue Date: 05 November 2003
See Certificate Revision History on Last Page

John Rumble, Jr., Chief
Measurement Services Division

¹Certain commercial organizations, services, equipment, or materials are identified in this report in order 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 organizations, services, materials, or equipment identified are necessarily the best available for the purpose.

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

Statistical consultation was provided by K.R. Eberhardt and S.D. Leigh 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 and B.S. MacDonald of the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

Transpiration: After the SRM has been removed from the aluminized Mylar¹ bag, transpiration will occur at an accelerated rate resulting in a gradual increase in the fluoride 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.

This SRM can be used to prepare working standard solutions in the range of 10 mg/kg to 100 mg/kg, from which more dilute standards are prepared. 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 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.; 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.; Eberhardt, K.R.; *Combining Data from Independent Chemical Analysis Methods*; *Spectrochimica Acta*, Vol. 46B, No. 12, pp. 1607-1613 (1991).

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| Certificate Revision History: 05 November 2003 (This revision reflects a change in the certification expiration date); 25 February 2000 (Original certificate date). |
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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 <http://www.nist.gov/srm>.