National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3183

Fluoride Anion (F^-) Standard Solution

Lot No. 140203

This Standard Reference Material (SRM) is intended as a primary calibration standard for the quantitative determination of fluoride using anion ion chromatography (IC) or other methods. A unit of SRM 3183 consists of 50 mL of solution in a high density polyethylene bottle sealed in an aluminized bag. The solution is prepared gravimetrically to contain a known mass fraction of fluoride dissolved in filtered (0.22 µm) water having a minimum resistivity of 18 MΩ cm.

Certified Value of Fluoride: 0.9968 mg/g ± 0.0031 mg/g

The certified value was calculated as the weighted mean of the mass fraction values obtained through (1) gravimetric preparation using high-purity sodium fluoride assayed by NIST and (2) analysis by anion IC calibrated using four primary standard solutions independently prepared from high-purity sodium fluoride assayed by NIST [1-2]. *No correction has been applied for transpiration that will occur after the SRM bottle unit has been removed from the sealed bag*. See "Instructions for Handling, Storage, and Use" for more information regarding transpiration.

The uncertainty associated with the certified value, stated as a symmetric interval with a level of confidence of 95 %, was evaluated in accordance with Supplement 1 to the ISO/JCGM Guide [3]. The uncertainty can be expressed as

$$U = ku_c$$

where $k = 1.969$ is the coverage factor for a 95 % confidence interval and 274 effective degrees of freedom. The quantity u_c is the combined standard uncertainty that represents, at the level of one standard deviation, the combined effect of uncertainty components associated with the gravimetric preparation, the IC determination, any difference between the methods' results, and stability of the actual fluoride mass fraction.

Expiration of Certification: The certification of **SRM 3183 Lot No. 140203** is valid, within the measurement uncertainty specified, until **31 December 2024**, provided the SRM is stored and handled in accordance with 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 certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of SRM 3183 was provided by T.W. Vetter of the NIST Chemical Sciences Division.

This SRM was prepared by T.A. Butler of the NIST Chemical Sciences Division. IC analyses were performed by T.A. Butler using primary standard solutions for IC calibration prepared by T.A. Butler and B.E. Lang of the NIST Chemical Sciences Division.

Statistical consultation was provided by A.M. Possolo of the NIST Statistical Engineering Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 16 November 2016

Steven J. Choquette, Director
Office of Reference Materials

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

METROLOGICAL TRACEABILITY

Metrological traceability of measurement results to a given reference must be established through an unbroken chain of calibrations and/or comparisons, each having stated uncertainties [4], using measurement standards that are appropriate for the physical or chemical property being measured. Comparisons may include validation measurements using various spectroscopic, chromatographic or classical methods of analysis. Gravimetric or volumetric dilution is also a method of comparison, where the mass or volume of a solution before and after dilution is measured.

For this SRM, the measurand is the total mass fraction of fluoride and the certified value is metrologically traceable to the derived SI unit for mass fraction. This SRM can be used to establish traceability of the results of fluoride measurements to NIST measurement results and standards. One approach is to calibrate analytical instruments or procedures for the determination of fluoride using standards whose values are traceable to the certified value of fluoride in this SRM. When the traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of all calibration measurements.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

This SRM can be used to prepare working standard solutions with fluoride mass fractions in the range 10 mg/kg to 100 mg/kg, from which more dilute standards can be 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 working standard solution should be prepared by transferring an aliquot of the SRM into an empty, dry, preweighed, polyethylene bottle and then reweighing the bottle. Water of appropriate purity 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 (i.e., mass of fluoride per mass of solution) 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.

Preparation of Working Standard Solutions by Volume: Volumetric dilutions are **NOT** recommended because of 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. Each working standard solution should be prepared by transferring an aliquot 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 must be reweighed to determine the exact mass of SRM solution transferred. The solution in the flask is then diluted to 99 % + volume using water of appropriate purity, mixed thoroughly, and the remaining few drops needed to dilute to exact volume carefully added. The concentration (in milligrams per milliliter) of the resulting working standard solution can then be calculated by multiplying the mass (in grams) of the SRM solution amount by the SRM certified value (in milligrams per gram) and dividing the numerical product by the calibrated volume (in milliliters) of the flask used for dilution. If this procedure is followed, no correction for density is needed. 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.

Transpiration: While stored in the sealed aluminized bag, transpiration of this SRM is negligible. After the SRM has been removed from the aluminized bag, transpiration will occur at a solution mass loss rate of approximately 0.2 % relative per year, resulting in a gradual increase of the mass fraction. It is the responsibility of the user to account for this effect. The recommended way to reduce the effect of transpiration is to deliver all of the SRM as aliquots weighed into appropriate vessels as soon as the SRM is removed from the aluminized bag. The aliquots may 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.

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM to validate or otherwise assign values to the more routinely used standards in a laboratory. When the metrologically traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of the calibration measurements for the in-house standard. Comparisons between NIST SRMs and such working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house standards. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

REFERENCES

[1] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46; pp. 323–331 (2009).

[2] DerSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; Control. Clin. Trials, Vol. 7; pp. 177–188 (1986).

[3] 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*; JCGM (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Nov 2016).

[4] JCGM 200:2012; *International Vocabulary of Metrology - Basic and General Concepts and Associated Terms*; 3rd ed.; JCGM (2012); available at http://www.bipm.org/en/publications/guides/vim.html (accessed Nov 2016).

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.