
3M Environmental Laboratory

Standard Operating Procedure

Salt and Acid Correction Factors for Fluorochemical Anionic Standards

SOP Number: ETS-4-031.1

Adoption Date: Upon Signing

Approved By:

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Technical Director

Effective Date (date of Quality Assurance signature):

Quality Assurance

1 Scope and Application

In general, fluorochemicals (FCs) are analyzed as the anionic form $[M]^-$ of the parent compound in negative-ion electrospray mass spectrometry. As most fluorochemicals are available in acid or salt form as neat materials, the corresponding acid or salt component should be accounted for in the resultant concentration of the standards used for quantitation. This standard operating procedure provides a list of correction factors for common fluorochemicals and their corresponding salts or free acids used in the 3M Environmental Laboratory, and describes how to correct for the salt or acid (H^+) component of a fluorochemical when calculating the resulting anionic concentrations.

Note: The correction factors described in this SOP do not replace nor override the need for purity corrections. The most current version of ETS-4-013 should be followed when preparing and documenting standards and other solutions.

Note: For solution preparation tracked and documented in LabWare® LIMS, the salt/acid correction factor will be incorporated automatically. There is no need for the analyst to apply any correction factor in those instances.

2 Definitions and Examples

Correction Factor: The mass ratio of the target anionic analyte to that of either the salt form or free acid form of the neat material.

Example: Ammonium Perfluorooctanoate (PFOA Ammonium Salt)

Molecular Formula: $C_8F_{15}O_2NH_4^+$

Molecular Weight of the Salt Form ($C_8F_{15}O_2NH_4^+$) (g/mol): 431.10

Molecular Weight of the Anion ($C_8F_{15}O_2^-$) (g/mol): 413.06

$$\text{CorrectionFactor} = \frac{\text{MolecularWeight Anion}}{\text{MolecularWeight SaltForm}}$$

Correction Factor for PFOA Ammonium Salt: 0.958. For purposes of this SOP, correction factors will be limited to 3 significant figures.

3 Precautions

Always wear appropriate personal protective equipment (gloves, safety glasses, lab coats, etc.) when working with solvents, neat materials, standards, and laboratory equipment.

4 Responsibility

It is the responsibility of all laboratory personnel who prepare fluorochemical standards to use this SOP to apply the appropriate salt or acid correction factors.

5 Supplies and Materials

Standards and Solutions Preparation Logbook.

List of Correction Factors, Attachment A

6 Equipment

Attachment A Correction Factor Calculator.

7 Procedures

Note: for solution preparation tracked and documented in LabWare® LIMS, the salt/acid correction factor will be incorporated automatically. There is no need for the analyst to apply any correction factor in those instances.

7.1 Single Component Solutions Prepared from Neat Materials

Identify the appropriate salt/acid correction factor for the material weighed. A list of correction factors is provided in Attachment A. Record the correction factor to three significant figures on the logbook page under "Other Correction Factors". (See ETS-4-013) Multiply the measured mass of neat material by the salt/acid correction factor and the recorded purity. Enter the resultant adjusted mass under "Corrected Weight". Proceed with determining the final concentration. The final corrected concentration should be used for all subsequent standards prepared using that stock.

7.2 Vendor Prepared Solutions

Several vendor prepared FC solutions (primarily, but not limited to, isotopically labeled FCs) have been purchased for use as calibration standards. The vendor prepared standards usually come in sealed ampules as a prepared solution in an organic solvent with a certified concentration (i.e. 50 ug/mL [1,2,3,4-¹³C]PFBA in methanol or 50 ug/mL of sodium perfluoro-1-[1,2,3,4-¹³C₄]octanesulfonate). The certificate of analysis will indicate if the FC material used to make the solution was in a salt or acid form. In general, the concentrations listed on the certificate of analyses have been determined gravimetrically using the mass of the neat material. The analyst should verify whether or not the salt or acid correction factor has been applied. Wellington Laboratories, the supplier of most of the laboratory's vendor prepared FC solutions, has recently started to include both the anion concentration as well as the salt concentration on the certificate of analysis. However, early solutions from this vendor did not apply the salt correction to the concentration listed on the certificate of analysis. In these instances, the correction factor needs to be applied before using the vendor solution to prepare calibration standards for analysis of the corresponding anion. In these situations, the stated vendor concentration should be multiplied by the appropriate correction factor and recorded in the standard logbook preparation. The corrected concentration should then be used for all subsequent standard preparations that use the vendor standard. If the analyst is unsure if the stated concentration has, or has not, been corrected for the salt by the vendor, he/she should contact the vendor for clarification.

Note: Wellington Laboratories now provides anion concentrations corrected for the salt form on their certificates of analysis; however, they do not correct for the proton when the compound is a free acid.

8 Records

The appropriate correction factor should be listed on the standard logbook page used to make the initial standard. The corrected concentration should be used for all subsequent dilutions. A list of correction factors for the most commonly used fluorochemicals is provided as an attachment. If a specific chemical is not found on the list, the analyst should state how the correction factor was determined as a Note to File or as a comment on the standard logbook page. For these calculations it is recommended that the correction factor calculator in Attachment A be used.

9 Attachments

Attachment A: Salt Correction Table and Correction Factor Calculator

10 References

ETS-4-013, "Documentation of Solutions and Standards Preparation."

11 Affected Documents

ETS-4-013 "Documentation of Solutions and Standards Preparation."

12 Revisions

<u>Revision Number</u>	<u>Summary of Changes</u>
1	Attachment A was moved from the SOP to a stand-alone Excel Attachment. A correction factor calculator was added to Attachment A. Sections 6, 8, 9 and the title of attachment A were updated to include a reference to the Correction Factor Calculator added to Attachment A.