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Reference Material 8447
Perfluorinated Sulfonic Acids in Methanol
REFERENCE MATERIAL INFORMATION SHEET

Purpose: This Reference Material (RM) is a solution of three perfluorinated sulfonic acids (PFSAs) in methanol intended primarily for use in the calibration of chromatographic instrumentation.

Description: A unit of RM 8447 consists of three 2 mL ampoules, each containing approximately 1.2 mL of solution.

Non-Certified Values: The non-certified mass fraction values are given in Table 1 for three PFSAs. These values are based on results obtained from the gravimetric preparation of the solution and from the analytical results determined using liquid chromatography. Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the International System of Units (SI) or other higher-order reference system [1]. Each non-certified value is a weighted mean of average mass fractions, with one average from gravimetric preparation and one average from chromatographic measurements [2,3]. The expanded uncertainty is the half-width of a symmetric 95 % parametric bootstrap confidence interval [4], which is consistent with the ISO/JCGM Guide [5,6]. The effective coverage factor, *k*, is 2. Since two methods were used for each compound, the measureand is the average mass fraction for each compound listed.

Table 1. Non-certified Mass Fractions of the Perfluorinated Sulfonic Acids (PFSAs) in RM 8447

PFSA	Compound	Mass Fractions (mg/kg)
PFBS	Perfluorobutane sulfonate	42.3 ± 2.3
PFHxS	Perfluorohexane sulfonate	55.2 ± 1.7
PFOS	Perfluorooctane sulfonate	56.6 ± 2.5

Period of Validity: The non-certified values are valid within the measurement uncertainty specified until **31 January 2034**. The non-certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Reference Material Information Sheet and notify registered users. RM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the RM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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Safety: This material contains perfluoroalkyl substances, many of which have been reported to have toxic and/or carcinogenic properties, and should be handled with care. Use proper disposal methods. Refer to Safety Data Sheet included along with the Reference Material Information Sheet.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures below 30 °C.

Use: Open ampoules carefully to prevent contamination and injury. The ampoules are pre-scored and should **NOT** be opened using a file. Sample aliquots, minimum sample size of 50 µL, for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the non-certified values in Table 1 to be valid within the stated uncertainties. Because of the volatility of methanol, non-certified values are not applicable to material stored in ampoules that have been opened for more than 5 min, even if they are resealed.

Preparation: The PFSAs used in the preparation of this RM were obtained from Alfa Aesar (Ward Hill, MA), Fluka (St. Louis, MO), and Sigma Aldrich (St. Louis, MO). The solution was prepared at NIST by weighing and mixing the individual perfluoroalkyl substances and methanol. The weighed components were added to the methanol and mixed until completely dissolved and homogenized. The total mass of this solution was measured, and the mass fractions were calculated for the components. This bulk solution was then chilled to approximately –5 °C, and 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules that were then flame sealed.

Liquid Chromatographic Analysis: Aliquots from ampoules selected by a stratified random sampling were analyzed by using liquid chromatography tandem mass spectrometry (LC-MS/MS). An internal standard solution containing ^{13}C- labeled PFSAs was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of the perfluoroalkyl substances and internal standard compounds in methanol were chromatographically analyzed to determine analyte response factors.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed March 2023).

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

[3] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46(3), pp. 323–331 (2009).

[4] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall (1993).

[5] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed March 2023); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed March 2023).

[6] 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 https://www.bipm.org/en/committees/jc/jcgm/publications (accessed March 2023).

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Certain commercial equipment, instruments, or materials may be identified in this Reference Material Information Sheet 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 materials or equipment identified are necessarily the best available for the purpose.

Users of this RM should ensure that the Reference Material Information Sheet in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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