National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material® 2377

### Fatty Acid Methyl Esters in 2,2,4-Trimethylpentane

This Standard Reference Material (SRM) is a solution of 26 fatty acid methyl esters (FAMEs) in 2,2,4-trimethylpentane. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of FAMEs. A unit of SRM 2377 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

**Certified Mass Fractions of Constituents:** The certified mass fraction values and estimated uncertainties for the 26 FAMEs are given in Table 1 along with the Chemical Abstract Service (CAS) Registry Numbers. The certified mass fraction values are based on masses used in the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. The measurand is the total mass fraction for each fatty acid methyl esters. Metrological traceability is to the SI derived unit for mass faction (expressed as milligrams per gram).

**Expiration of Certification:** The certification of **SRM 2377** is valid, within the measurement uncertainty specified, until **30 June 2023**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The 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 this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of technical measurements leading to certification were performed by M.M. Schantz and L.C. Sander of the NIST Chemical Sciences Division.

Preparation and analytical measurements of the SRM were performed by M.M. Schantz.

Statistical consultation was provided by S.D. Leigh of the NIST Statistical Engineering Division.

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

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*Certificate Revision History on Last Page*

Steven J. Choquette, Director
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## INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

**Handling:** This material should be handled with care. Use proper disposal methods.

**Storage:** The material should be stored under refrigeration (0 °C to 4 °C), in an unopened ampoule, until required for use.

**Use:** Prior to removal of a test portion for analysis, the contents of an ampoule should be allowed to warm to room temperature and mixed thoroughly. Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified values in Table 1 to be valid within the stated uncertainties. Because of the volatility of 2,2,4-trimethylpentane, certified values are not applicable to material stored in ampoules that have been opened for more than 5 minutes, even if they are resealed.

## PREPARATION AND ANALYSIS[1]

The compounds used in the preparation of this SRM were obtained from commercial sources. The solution was prepared at NIST by weighing and combining the individual compounds and 2,2,4-trimethylpentane. The weighed components were added to the 2,2,4-trimethylpentane and mixed overnight. The total mass of this solution was measured, and the mass fractions were calculated. These gravimetric concentrations were adjusted for the purity estimation of each component, which was determined by using capillary gas chromatography with two stationary phases of different polarities. The bulk solution was chilled slightly, and 1.2 mL aliquots were dispensed into 2-milliliter glass ampoules, which were flushed with argon then flame-sealed.

Aliquots from nine ampoules, selected using a random stratified sampling scheme, were analyzed in duplicate by using capillary gas chromatography with flame ionization detection (GC-FID) on a relatively polar nonbonded, biscyanopropyl polysiloxane phase (SP 2560, 100 m × 0.25 mm id, 0.25 µm film thickness, Supleco, Bellefonte, PA). The internal standard solution added to each sample for quantification purposes contained methyl tridecanoate, methyl heneicosanoate, and methyl octacosanoate. Calibration solutions consisting of weighed amounts of the 26 FAMEs and the internal standard compounds in 2,2,4-trimethylpentane were chromatographically analyzed to determine analyte response factors.

[1]Certain commercial equipment, instruments or materials are identified in this certificate 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.

Table 1. Certified Mass Fraction Values of FAMEs

| FAME | CAS Registry No.[a] | Mass Fraction (mg/g)[b] |
|---|---|---|
| Octanoic Acid Methyl Ester (Caprylic Acid Methyl Ester) | 111-11-5 | 7.286 ± 0.054 |
| Decanoic Acid Methyl Ester (Capric Acid Methyl Ester) | 110-42-9 | 7.499 ± 0.058 |
| Dodecanoic Acid Methyl Ester (Lauric Acid Methyl Ester) | 111-82-0 | 7.93 ± 0.11 |
| Tetradecanoic Acid Methyl Ester (Myristic Acid Methyl Ester) | 124-10-7 | 7.11 ± 0.13 |
| Hexadecanoic Acid Methyl Ester (Palmitic Acid Methyl Ester) | 112-39-0 | 7.38 ± 0.32 |
| Octadecanoic Acid Methyl Ester (Stearic Acid Methyl Ester) | 112-61-8 | 7.68 ± 0.12 |
| Eicosanoic Acid Methyl Ester (Arachidic Methyl Ester) | 1120-28-1 | 3.66 ± 0.21 |
| Docosanoic Acid Methyl Ester (Behenic Acid Methyl Ester) | 929-77-1 | 4.28 ± 0.10 |
| Tetracosanoic Acid Methyl Ester (Lignoceric acid methyl ester) | 2442-49-1 | 1.807 ± 0.081 |
| 9-Tetradecenoic Acid Methyl Ester (Myristoleic Acid Methyl Ester) | 56219-06-8 | 1.894 ± 0.033 |
| 9-Hexadecenoic Acid Methyl Ester (Palmitoleic Acid Methyl Ester) | 1120-25-8 | 5.031 ± 0.039 |
| 9-Octadecenoic Acid Methyl Ester (Oleic Acid Methyl Ester) | 112-62-9 | 7.01 ± 0.31 |
| 9-*trans*-Octadecenoic Acid Methyl Ester (Elaidic Acid Methyl Ester) | 1937-62-8 | 2.02 ± 0.18 |
| 11-Octadecenoic Acid Methyl Ester (Vaccenic Acid Methyl Ester) | 1937-63-9 | 2.31 ± 0.14 |
| 11-*trans*-Octadecenoic Acid Methyl Ester (*trans*-Vaccenic Acid Methyl Ester) | 6198-58-9 | 2.368 ± 0.047 |
| 9,12-Octadecadienoic Acid Methyl Ester (Linoleic Acid Methyl Ester) | 112-63-0 | 7.33 ± 0.14 |
| 9-*trans*,12-*trans*-Octadecadienoic Acid Methyl Ester (Linoelaidic Acid Methyl Ester) | 2566-97-4 | 2.046 ± 0.015 |
| 9,12,15-Octadecatrienoic Acid Methyl Ester (*alpha*-Linolenic Acid Methyl Ester) | 301-00-8 | 4.26 ± 0.26 |
| 6,9,12-Octadecatrienoic Acid Methyl Ester (*gamma*-Linolenic Acid Methyl Ester) | 16326-32-2 | 1.796 ± 0.052 |
| 11-Eicosenoic Acid Methyl Ester (Gondolic Acid Methyl Ester) | 2390-09-2 | 1.919 ± 0.081 |
| 5,8,11,14-Eicosatetraenoic Acid Methyl Ester (Arachidonic Acid Methyl Ester) | 2566-89-4 | 1.511 ± 0.063 |
| 5,8,11,14,17-Eicosapentaenoic Acid Methyl Ester (EPA Methyl Ester) | 2734-47-6 | 1.526 ± 0.068 |
| 7,10,13,16,19-Docosapentaenoic Acid Methyl Ester (DPA Methyl Ester) | 108698-02-8 | 1.426 ± 0.030 |
| 4,7,10,13,16,19-Docosahexaenoic Acid Methyl Ester (DHA Methyl Ester) | 2566-90-7 | 1.621 ± 0.082 |
| 13-Docosenoic Acid Methyl Ester (Erucic Acid Methyl Ester) | 1120-34-9 | 2.204 ± 0.083 |
| 15-Tetracosenoic Acid Methyl Ester (Nervonic Acid Methyl Ester) | 2733-88-2 | 1.74 ± 0.011 |

[a] Chemical Abstracts, Fourteenth Collective Index. Index Guide, American Chemical Society, Columbus, Ohio 2001.

[b] The reference value is a weighted mean of the mass fractions determined by gravimetric and chromatographic measurements [2], corrected for purity. The uncertainty listed with each value is an expanded uncertainty about the mean [3,4], with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method variance following the ISO/JCGM Guide [5].

## REFERENCES


[1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed Feb 2017).

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

[3] Horn, R.A.; Horn, S.A.; Duncan, D.B.; *Estimating Heteroscedastic Variance in Linear Models*; J.Am.Stat. Assoc., Vol. 70, pp. 380–385 (1975).

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

[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 (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Feb 2017); 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://www.nist.gov/physlab/pubs/index.cfm (accessed Feb 2017).



**Certificate Revision History:** 10 February 2017 (Editorial changes); 14 July 2016 (Change of expiration date; editorial changes); 06 October 2010 (Original certificate date).


*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.*