



SLOVENSKI STANDARD

SIST EN 14078:2014

01-junij-2014

Nadomešča:
SIST EN 14078:2010

Tekoči naftni proizvodi - Določevanje metilnih estrov maščobnih kislin (FAME) v srednjih destilatih - Metoda infrardeče spektroskopije

Liquid petroleum products - Determination of fatty methyl ester (FAME) content in middle distillates - Infrared spectrometry method

Flüssige Mineralölprodukte - Bestimmung von Fettsäure Methylester (FAME) in Mitteldestillaten - Infrarotspektrometrisches Verfahren

Produits pétroliers liquides - Détermination de la teneur en esters méthyliques d'acides gras (EMAG) des distillats moyens - Méthode spectrochimique infrarouge

Ta slovenski standard je istoveten z: EN 14078:2014

ICS:

75.160.20 Tekoča goriva Liquid fuels

SIST EN 14078:2014 en,fr,de

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST EN 14078:2014](#)

<https://standards.iteh.ai/catalog/standards/sist/448a561b-761e-435a-b04b-fd3deca98958/sist-en-14078-2014>

EUROPEAN STANDARD

EN 14078

NORME EUROPÉENNE

EUROPÄISCHE NORM

May 2014

ICS 75.160.20

Supersedes EN 14078:2009

English Version

Liquid petroleum products - Determination of fatty acid methyl ester (FAME) content in middle distillates - Infrared spectrometry method

Produits pétroliers liquides - Détermination de la teneur en esters méthyliques d'acides gras (EMAG) des distillats moyens - Méthode par spectrométrie infrarouge

Flüssige Mineralölerzeugnisse - Bestimmung des Gehaltes an Fettsäuremethylester (FAME) in Mitteldestillaten - Infrarotspektrometrisches Verfahren

This European Standard was approved by CEN on 10 April 2014.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Foreword.....	3
1 Scope	4
2 Normative references	4
3 Principle.....	4
4 Reagents and materials	5
5 Apparatus	5
6 Sampling and sample handling.....	5
7 Procedure	5
7.1 Selection and treatment of the cell	5
7.2 Cleaning of the cells	6
7.3 Selection of the path length.....	6
7.4 Calibration	7
7.5 Sample Preparation	8
7.6 Recording of infrared spectra	8
8 Calculation.....	9
8.1 Calculation of FAME content in grams per litre (g/l)	9
8.2 Unit conversion from FAME content from g/l to % (V/V)	9
9 Expression of results	10
10 Precision.....	10
10.1 General.....	10
10.2 Repeatability, r	10
10.3 Reproducibility, R	10
11 Test report	11
Annex A (informative) Specific adjustments for calibration and dilution	12
Bibliography.....	15

iteh STANDARD PREVIEW

(standard.iteh.ai)

SIST EN 14078:2014

<https://standards.iteh.ai/catalog/standards/sist/448a561b-761e-435a-b04b->

[1d3deca98938/sist-en-14078-2014](https://standards.iteh.ai/catalog/standards/sist/448a561b-761e-435a-b04b-1d3deca98938/sist-en-14078-2014)

Foreword

This document (EN 14078:2014) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2014 and conflicting national standards shall be withdrawn at the latest by November 2014.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document will supersede EN 14078:2009. This third version includes the following significant technical changes with respect to the former:

- the test procedure has been updated with an additional range C, and
- data from a newly executed Round Robin test have been integrated in order to expand the scope to blends containing up to 50 % (*V/V*) of FAME.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

[SIST EN 14078:2014](https://standards.iteh.ai/catalog/standards/sist/448a561b-761e-435a-b04b-fd3deca98958/sist-en-14078-2014)

<https://standards.iteh.ai/catalog/standards/sist/448a561b-761e-435a-b04b-fd3deca98958/sist-en-14078-2014>

EN 14078:2014 (E)

1 Scope

This European Standard specifies a test method for the determination of Fatty Acid Methyl Ester (FAME) content in diesel fuel or domestic heating fuel by mid infrared spectrometry, which applies to FAME contents of the three measurement ranges as follows:

- range A: for FAME contents ranging from approx. 0,05 % (V/V) to approx. 3 % (V/V);
- range B: for FAME contents ranging from approx. 3 % (V/V) to approx. 20 % (V/V);
- range C: for FAME contents ranging from approx. 20 % (V/V) to approx. 50 % (V/V).

Principally, higher FAME contents can also be analysed if diluted; however, no precision data for results outside the specified range is available at present.

This test method was verified to be applicable to samples which contain FAME conforming to EN 14214. Reliable quantitative results are obtained only if the samples do not contain any significant amounts of other interfering components, especially esters and other carbonyl compounds which possess absorption bands in the spectral region used for quantification of FAME. If such interfering components are present, this test method is expected to produce higher values.

NOTE 1 For the purposes of this European Standard, the term “% (V/V)” is used to represent the volume fraction (φ) of a material.

NOTE 2 For conversion of grams FAME per litre (g FAME/l) to volume fraction, a fixed density for FAME of 883,0 kg/m³ is adopted.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

WARNING — The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 14214, *Liquid petroleum products - Fatty acid methyl esters (FAME) for use in diesel engines and heating applications - Requirements and test methods*

EN ISO 3170, *Petroleum liquids - Manual sampling (ISO 3170)*

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171)*

3 Principle

The mid infrared absorption spectrum of a test portion of a sample which is diluted as appropriate if necessary with FAME-free solvent is recorded. The absorbance at the peak maximum of the typical absorption band for esters at about $(1\,745 \pm 5) \text{ cm}^{-1}$ is measured. Initially, calibration as well as evaluation of the data will be carried out as grams FAME per litre. For conversion of grams FAME per litre (g/l) to the reporting unit “% (V/V)”, a fixed density of FAME of 883,0 kg/m³ (at 15 °C) is adopted.

Three measurement ranges (A, B or C) have been chosen for which specific adjustments for the calibration and dilution need to be followed. Measurement should be conducted preferably without dilution for range A and a

shorter path length of the measurement cell as well as an appropriate dilution for range B and C. In particular, the lower determination range A is a challenging one; all ensuing details shall be kept as a package without any omissions or additions of individual details. That is the only way to adhere to the improved precision data of range A determined by round robin tests.

Based on the absorbance measured at the maximum of the peak of the absorption band the FAME content is calculated by means of a calibration function which was determined by measuring calibration solutions for which the FAME content is known.

4 Reagents and materials

4.1 FAME for calibration, FAME as specified in EN 14214.

4.2 FAME free middle distillate as solvent for dilution and as reference material for the measurement of the background spectrum. In particular, a middle distillate suitable for the type of sample (diesel fuel or domestic heating oil) shall be used for range A in order to avoid spectral decompensation as far as possible. In this context, the property "FAME free" means middle distillates without any absorption bands in the IR signal range typical for FAME.

4.3 Solvents for cleaning, such as ethanol, n-pentane or cyclohexane.

5 Apparatus

5.1 Infrared spectrometer, dispersive or interferometer type, capable of operating in the wave number range from approx. 400 cm^{-1} to approx. $4\,000\text{ cm}^{-1}$, with a linear absorption in the absorbance range from (0,1 to 1,1) absorbance units, and having a resolution of minimum 4 cm^{-1} .

5.2 Cells, made of KBr, NaCl, or CaF_2 , with known path length, where the additional instructions for the selection and treatment of cells dependent on the applied measurement range as given in 7.1 are followed.

EXAMPLE A standard solution with a FAME concentration of 3 g/l (0,34 % (V/V)) should give an absorbance of about 0,4 at the maximum peak at about $1\,745\text{ cm}^{-1}$ if a cell with a path length of 0,5 mm is used.

6 Sampling and sample handling

Samples shall be taken according to EN ISO 3170 or EN ISO 3171 as well as in accordance with the requirements of national standards or regulations for the sampling of the product under test. If samples are not tested immediately, they shall be stored tightly sealed and in a cool and dark place.

7 Procedure

7.1 Selection and treatment of the cell

The path length of the cell (5.2) shall be selected so that adequate net signal intensities can be obtained (in at least two decimal places, see marked cells in Table A.1). Signal intensities shall still be within the linear detector range.

Specific adjustments shall be followed (see recommendations given in Table A.1) depending on measurement range A, B or C:

- range A: path length as long as possible and measurement preferably without dilution;
- range B: shorter path length and dilution adapted to the anticipated FAME content;

EN 14078:2014 (E)

— range C: shorter path length and a dilution higher than for range B, adapted to the anticipated FAME content.

The path length shall be known or determined exactly. If in use for a longer period of time it should be checked more frequently. A specific and individual calibration shall be made for every cell in use, retaining all other measurement parameters for every applied measurement range. The use of several or different cells during the same calibration is not permissible.

The very same cell shall be used for calibration and for sample measurement. Only the exactly determined path length applies for the cell in use; nominal data (nominal path length, labelling, nominal spacer data or the like) shall not be used without any adequate control or correction measures.

If cells sensitive to water are used the path length shall be checked more frequently. In case the path length has changed, path length determination and calibration shall be carried out anew.

7.2 Cleaning of the cells

After every measurement the cell shall be carefully cleaned with a solvent (4.3). This is particularly important after measuring samples with high FAME content and extremely important when the measurement of samples with low FAME contents is prepared. The cell may also be cleaned by rinsing repeatedly with FAME free middle distillate (4.2).

In persistent cases cyclohexane may also be used for cleaning (yet not for calibration or dilution purposes) as well as the following alternative cleaning procedure:

- a) rinse twice with 5 ml n-pentane each time; then
- b) rinse once with 5 ml ethanol (absolute); then
- c) rinse once again with 5 ml n-pentane and finally dry with suitable equipment.

In case the cleanliness of the cell is in doubt, a reference spectrum of a FAME free sample shall be recorded and checked for control reasons. The spectrum shall not show signals in the neighbourhood of $1\ 745\ \text{cm}^{-1}$.

7.3 Selection of the path length**7.3.1 Range A**

For from approx. 0,05 % (*V/V*) to approx. 3 % (*V/V*) cells with a long path length (e.g. KBr) of approx. 0,5 mm, known to the nearest 0,01 mm) have proven to be well suited. Other materials and path lengths are also permissible, though on no account should the path length be chosen less than about 0,2 mm to enable a signal and signal-to-noise ratio as favourable as possible.

7.3.2 Range B

For FAME contents from approx. 3 % (*V/V*) to approx. 20 % (*V/V*) a path length of approx. 0,1 mm, known to the nearest 0,01 mm, is recommended in combination with a dilution rate of 1 : 5 (refer to data given in Table A.1). Other cells and path lengths are also possible; however, dilution should be arranged within a comparable dimension in order to avoid any major dilution errors.

7.3.3 Range C

For FAME contents above 20 % (*V/V*), the following measurement parameters are recommended:

- path length of approx. 0,05 mm, known to the nearest 0,01 mm, with a dilution rate of 1 : 5;
- path length of approx. 0,1 mm, known to the nearest 0,01 mm, with a dilution rate of 1 : 10.

For detailed information refer to data given in Table A.1.

7.4 Calibration

7.4.1 General instructions

Calibration and ensuing measurement shall be carried out retaining all other measurement parameters.

The lower the FAME content, the smaller is the carbonyl band (even if the absorption intensity is still high) and thus the more critical the background correction. Especially for range A (low FAME contents) the background correction with the calibration samples (and possibly also with spectra of blank samples) should be practiced thoroughly.

7.4.2 Preparation of calibration solutions

The following ranges should be followed as general guidance:

- range A from approx. 0,05 % (V/V) to approx. 3 % (V/V);
- range B from approx. 3 % (V/V) to approx. 20 % (V/V);
- range C from approx. 20 % (V/V) to approx. 50 % (V/V).

A set of at least five calibration solutions (preferably more) with precisely known concentrations of FAME (4.1) in FAME free middle distillate (4.2) shall be prepared for the measurement range of interest by weighing FAME into appropriate graduated flasks and filling to the mark with FAME free middle distillate.

WARNING — This method detects all FAME components by measurement of the C=O IR absorption at approximately 1745 cm⁻¹ and C8 to C22 molecules, as specified in EN 14214. The accuracy of the measured results depends on the best match of molecular weights (proportional to chain length) between the FAME material used for calibration and the FAME material in the analyzed sample. This means that presence of FAME with lower average chain length, determined with a calibration using FAME of longer average chain length, will result in considerable overestimation and vice versa. For a correct result it is recommended to use FAMEs for calibration (4.1) that have a similar average molecular weights to the product being tested.

FAME free middle distillate should be used as an additional calibration sample (nominal FAME content “zero”). Additional calibration samples should not be made by means of dilution due to possible error propagation.

Each calibration solution shall be produced separately by weighing.

7.4.3 Calculation of the calibration function

In every case and without exception the form of the calibration function is

$$Y = F(X) \quad (1)$$

with

Y is the signal (dependent variable), i.e. corrected Extinction E_{CORR} ;

X is the content (independent variable), i.e. FAME content in grams FAME per litre (g FAME/l).

Based on the FAME content (X) and the relevant normalised extinction coefficients $E_{\text{CORR}}(Y)$, for all calibration solutions the straight calibration line is calculated by linear regression using the model according to Formula (2):

$$Y(i) = a \times X(i) + b \quad (2)$$

with