



**SLOVENSKI STANDARD
SIST EN 14078:2004**

01-julij-2004

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Liquid petroleum products - Determination of fatty acid methyl esters (FAME) in middle distillates - Infrared spectroscopy 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 par spectrométrie infrarouge

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Ta slovenski standard je istoveten z: EN 14078:2003

ICS:

75.160.20 V^ \ [æ^ [!ãæ Liquid fuels

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 14078

December 2003

ICS 75.160.20

English version

Liquid petroleum products - Determination of fatty acid methyl esters (FAME) in middle distillates - Infrared spectroscopy method

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This European Standard was approved by CEN on 7 November 2003.

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 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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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EN 14078:2003 (E)

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Foreword

This document EN 14078:2003 has been prepared by CEN/TC 19, "Petroleum products, lubricants and related products", 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 June 2004, and conflicting national standards shall be withdrawn at the latest by June 2004.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard : Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

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EN 14078:2003 (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 in the range from about 1,7 % (V/V) to 22,7 % (V/V). Other FAME contents can also be analyzed in principle, however, no precision data for results outside the specified range are available at this time.

The test method has been verified to be applicable to samples which contain FAME conforming to the European specifications EN 14214 or EN 14213. Reliable quantitative results are obtained only when the samples do not contain significant amounts of other interfering components, especially esters, which possess absorption bands in the spectral region used for quantification of FAME. When such interfering components are present, this test method is expected to produce higher values

NOTE 1 When interfering components are suspected to be present, it is recommended for cases of doubt or dispute to record the full infrared spectrum and to compare it to spectra of samples with well known FAME contents.

NOTE 2 For the purposes of this European Standard, the term "% (V/V)" is used to represent the volume fraction of a material.

NOTE 3 For conversion of g/l to % (V/V), a fixed density of FAME of 880,0 kg/m³ is adopted.

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.

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2 Normative references

This European Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed thereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies (including amendments).

EN 14213, *Heating fuels - Fatty acid methyl esters (FAME) - Requirements and test methods.*

EN 14214, *Automotive fuels – Fatty acid methyl esters (FAME) for diesel engines – Requirements and test methods.*

EN ISO 3170, *Petroleum liquids – Manual sampling (ISO 3170:1988, including Amendment 1:1998).*

EN ISO 3171, *Petroleum liquids – Automatic pipeline sampling (ISO 3171:1988).*

3 Principle

The mid infrared absorption spectrum of a test portion of a sample which has been diluted as appropriate with cyclohexane, is recorded. The absorbance at the peak maximum of the typical absorption band for esters at about $1\,745\text{ cm}^{-1} \pm 5\text{ cm}^{-1}$ is measured. The FAME content is then calculated with a calibration function produced from standard solutions with a known FAME content.

4 Reagents and materials

4.1 FAME for calibration

FAME as specified in EN 14214 or EN 14213

4.2 Cyclohexane, > 99,5 % (V/V)

5 Apparatus

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

5.2 Cell, made of KBr, or NaCl, or CaF_2 , with accurately known path length

EXAMPLE A 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 $1\,745\text{ cm}^{-1}$ when a cell with a path length of 0,5 mm is used.

6 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in EN ISO 3170 or EN ISO 3171 and/or in accordance with the requirements of national standards or regulations for the sampling of the product under test.

7 Procedure

7.1 General

Because of the viscosity of FAME solutions, cleaning the cells used for measurement is of great importance. Cells shall be cleaned thoroughly by repeated rinsing with cyclohexane. The cells are considered as sufficiently clean when the recorded IR spectrum of the cell filled with cyclohexane exactly matches the reference cyclohexane spectrum.

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7.2 Calibration

7.2.1 Preparation of calibration solutions

A set of at least five calibration solutions with precisely known concentrations of FAME (4.1) in cyclohexane (4.2) shall be prepared by weighing FAME into appropriate graduated flasks and filling to the mark with cyclohexane. The nominal FAME concentrations for the set of five calibration solutions shall be selected in such a way that the absorbance at the maximum peak at about $1\,745\text{ cm}^{-1}$ is in the range from 0,1 to 1,1 absorbance units

EXAMPLE For a cell with a nominal path length of 0,5 mm (see also 5.2), the calibration solutions are 1 g/l, 2 g/l, 4 g/l, 6 g/l and 10 g/l.

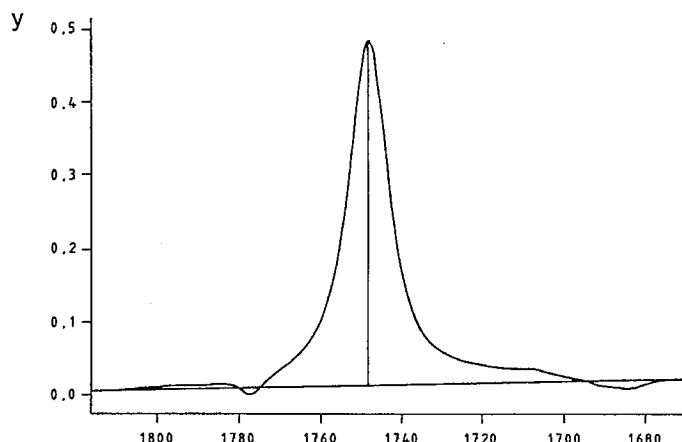
It is important to use identical cells both for calibration and measurement.

7.2.2 Spectrometric measurement

This procedure is identical for the calibration solutions and for the samples under test. The test portion or the calibration solution is filled into the cell and the IR spectrum is recorded against a spectrum of cyclohexane (4.2). The absorbance at the peak maximum at about $1\,745\text{ cm}^{-1}$ is then measured, using a baseline from $1\,670\text{ cm}^{-1}$ to $1\,820\text{ cm}^{-1}$ (see Figure 1).

NOTE Great care should be exercised to accurately perform the measurement against cyclohexane. The IR absorption bands from cyclohexane should either be directly optically compensated (double beam instruments), or subtracted by calculation (single beam instruments).

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Key

y Absorbance

Figure 1 — Typical spectrum for FAME in diesel fuel diluted in cyclohexane (cell path 0,5 mm, concentration: 44 g/l after a 1:10 dilution (V/V))

7.2.3 Calibration function

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Using the absorbance measurements for the set of FAME calibration solutions (see also 7.2.1), a calibration function is calculated by linear regression or by plotting, using the absorbance, A , as the dependent, and the concentration, q , as the independent variable. This gives the calibration function for a calculated standard cell path length of 1 cm as follows:

$$A/L = a * q + b \quad (1)$$

where:

- A is the measured absorbance in units of absorbance;
- L is the actually used cell path length in cm;
- q is the concentration of FAME in g/l;
- a is the slope of the regression line;
- b is the y intercept of the regression line.

NOTE It is strongly recommended to repeat the calibration procedure when the correlation coefficient (R^2) for the regression line is below 0,99.

7.3 Quantitative analysis

7.3.1 Preparation of samples

Samples containing FAME in a middle distillate are analysed after appropriate dilution in cyclohexane. If the absorbance measured on this test solution does not fall in the absorbance range of the calibration, a new sample with a more suitable dilution shall be prepared. For FAME contents below about 100 g/l (11,4 % (V/V)), a dilution ratio of at least 1:10 (V/V) shall be used. For FAME contents above 100 g/l (11,4 % (V/V)) and below about 200 g/l (22,7 % (V/V)), a dilution ratio of at least 1:20 (V/V) shall be used.

NOTE 1 For FAME contents above 200 g/l (22,7 % (V/V)), adequate dilution ratios should be used in order to bring the absorption in the specified absorbance range of the calibration.

NOTE 2 The given dilution ratios are based on a nominal path length of the cell of 0,5 mm

7.3.2 Spectrometric measurement

The spectrometric measurement is performed on the test solution according to 7.2.2. It is important to ensure that the same cells are used both for measurement and calibration.

Due to the great importance of cleaning the cells, it is recommended to record the IR spectrum of the cell filled with cyclohexane between each sample, to check its cleanliness (see also 7.1).

8 Calculation

Calculate the FAME content, Q , in the sample using:

$$Q = \frac{X}{a} \left[\frac{A}{L} - b \right] \frac{100}{d} \quad (2)$$

where:

- Q is the FAME content in % (V/V);
- X is the dilution factor (i.e. $X = 10$ for a dilution of 1:10);
- a is the slope of the regression line;
- b is the y intercept of the regression line;
- A is the absorbance measured according to 7.3.2;
- L is the path length of the cell in cm;
- d is the density of FAME ($d = 880,0 \text{ kg/m}^3$) at $20 \text{ }^\circ\text{C}$ in kg/m^3 .

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9 Expression of results

Report the FAME content in the sample, Q , in % (V/V), rounded off to the nearest 0,1.

10 Precision

10.1 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed 0,3 % (V/V) in absolute value in only one case in twenty.

10.2 Reproducibility

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, for concentrations lower or equal to 11,4 % (V/V), exceed 0,9 % (V/V) in absolute value in only one case in twenty. For concentrations higher than 11,4 % (V/V) and below 22,7 % (V/V), it would exceed 1,4 % (V/V) in absolute value in only one case in twenty.

11 Test report

The test report shall contain at least the following information:

- a) a reference to this European Standard;