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

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Terminology.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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.

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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, <i>r</i>	10
10.3 Reproducibility, <i>R</i>	10
11 Test report	11
Annex A (informative) Specific adjustments for calibration and dilution	12
Bibliography.....	15

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of an identical text or by endorsement, at the latest by November 2014 and conflicting national standards shall be withdrawn at the latest by November 2014.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.

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.

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;

— 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

$$Y = F(X) \tag{1}$$

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):

$$Y(i) = a \times X(i) + b \tag{2}$$

with

$Y(i)$ is the determined corrected extinction E_{CORR} for calibration sample (i);

$X(i)$ is the adjusted FAME content, in grams FAME per litre (g FAME/l), of calibration sample (i);

a, b are the regression coefficients (slope and intercept) obtained from the linear regression.

The regression coefficient b ("Y-axis intercept") should ideally be zero in case of accurate work. However, it shall not be set to zero at random. Deviation from zero results from the regular statistical spread of the measuring points around the line of best fit and from the leverage of calibration samples with higher FAME contents. In case of high or striking values for the y-axis intercept the calibration should be rechecked carefully.

Other calibration models are not permitted. The inversion of the calibration function necessary for the evaluation of measurements is described in 8.1.

7.5 Sample Preparation

Depending on the FAME concentration the samples may need to be diluted with FAME free Diesel fuel. The dilution ratio shall be chosen according to Annex A in order to find the optimal absorbance.

Samples are diluted by weighing the sample volume corresponding to the dilution ratio into an appropriate graduated flasks and fill to the mark with FAME free middle distillate.

7.6 Recording of infrared spectra

7.6.1 General instructions

If multiple scans are possible, at least 16 scans (identical for all records, see also 7.4.1) shall be used. All further steps apply to both the calibration samples and for the samples to be tested. It is important that all other settings of the IR spectrometer are also retained.

7.6.2 Background and reference spectrum

At every calibration a background spectrum shall be taken and recorded as a reference spectrum for the compensation which is carried out with every measurement. For that the appropriate base fuel for the relevant type of sample (refer to 7.4.1 and 4.2) shall be used.

7.6.3 Recording of spectra

When registering spectra of the calibration solutions, blank samples should be registered first, to be followed by rising FAME contents so that memory effects are eliminated as far as possible. Apart from that, the cell shall be cleaned thoroughly before every measurement.

In case of overflowing of the cell it should be cleaned thoroughly with solvent.

Cells and dilution factors are selected and recorded according to the assumed FAME contents. In case of doubt an indicative measurement shall be carried out with a separated sample fraction. The IR spectrum is recorded in the range from $4\,000\text{ cm}^{-1}$ to approximately 400 cm^{-1} .

The background and/or reference spectrum is deducted. Compensation as sound as possible shall be achieved and overcompensation (negative bands) especially in the wave number range required for assessment shall be avoided. The resultant IR spectrum shall be recorded for a possible inspection necessary at a later date (preferably digital recording).

If difficulties occur concerning the measurement and assessment of the absorption bands especially of lower FAME contents, it is strongly recommended to determine the measuring result based on multiple, independent and individual measurements. Additionally, the assessment of the spreads within the individual results may provide good reference as to the quality of both measurement and assessment.

7.6.4 Determination of the Extinction and the corrected Extinction

First of all a tangent is drawn between approximately $1\,670\text{ cm}^{-1}$ and approximately $1\,820\text{ cm}^{-1}$ as base line. Thereafter the extinction E_{meas} is determined as the vertical distance from the base line to the absorption band maximum at $1\,745 \pm 5\text{ cm}^{-1}$. For the assessment, the correct definition of the base line, a background correction as sound as possible and sufficient signal-to-noise ratio shall be given attention to.

During calculation the used dilution rates shall be considered accordingly. The corrected extinction E_{corr} is the extinction measured in the IR spectrum (E_{meas}), recalculated (normalised or adjusted) for the undiluted sample. E_{corr} is calculated according to:

$$E_{\text{corr}} = E_{\text{meas}} \cdot \left(\frac{V_{\text{VF}}}{V_{\text{SV}}} \right) \quad (3)$$

and applies only for the specific cell used.

V_{VF} is the volume of the volumetric flask in millilitres (ml) used for diluting the sample

V_{SV} is the sample volume in millilitres (ml) for dilution.

If an undiluted sample is tested the dilution factor is equal to one. In that case, for both volumes a value of 1,0 shall be used.

NOTE Unlike to the extinction E_{meas} which should be in the linear extinction range of the detector (see above remarks) the normalisation on undiluted samples according to Formula (3) may lead to significantly higher values for E_{corr} if higher FAME contents are tested.

8 Calculation

8.1 Calculation of FAME content in grams per litre (g/l)

The solution of Formula (2) to X for the calculation of the FAME content gives Formula (4):

$$X_{\text{s}} = \frac{E_{\text{corr}} - b}{a} \quad (4)$$

a regression coefficient (slope) obtained from the linear regression (7.4.3);

b regression coefficient (intercept) obtained from the linear regression (7.4.3);

X_{s} the determined FAME content of the tested sample, in grams per litre (g/l).

8.2 Unit conversion from FAME content from g/l to % (w/w)

FAME concentrations given in grams per litre (g/l) shall be converted using a nominal FAME density ($15\text{ }^{\circ}\text{C}$) of $883,0\text{ kg/m}^3$ according to:

$$Y_{\text{s}} = \frac{100 \cdot X_{\text{s}}}{883} \quad (5)$$

Y_{s} is the FAME content in % (w/w);

X_{s} is the FAME content in grams per litre (g FAME/l).

EXAMPLE $23,5\text{ g FAME/l} \rightarrow 2,661\text{ } \%$ (w/w), rounded to two decimals = $2,66\text{ } \%$ (w/w) (range A, see 7.4.2).

9 Expression of results

The unrounded value for the FAME content, in g FAME/l, shall be used as intermediate result.

In order to convert the result to % (V/V) use Formula (5) in 8.2.

The result shall be reported in % (V/V) with two decimal places for measurement range A.

Results for measurement range B and C shall be reported in % (V/V) with one decimal place.

10 Precision

10.1 General

The precision given was derived from statistical analysis by EN ISO 4259 [1] of the results of interlaboratory testing of a matrix of various diesel fuels with both low and high FAME contents, as well as three domestic heating oils (measurement range A only).

NOTE The interlaboratory testing and the statistical evaluation are detailed in a report of a European Commission project called Bioscopes [2].

10.2 Repeatability, *r*

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 the value given in Table 1 in only one case in twenty.

10.3 Reproducibility, *R*

The difference between two individual and independent test results, obtained from identical samples by different operators in different laboratories, implementing the usual and correct procedure, may only exceed the value stated in Table 1 in one case in twenty.

Table 1 — Precision statements

Measurement range and type of product	Repeatability <i>r</i> % (V/V)	Reproducibility <i>R</i> % (V/V)
Middle distillate range A	$r = 0,012\ 6\ X + 0,007\ 9$	$R = 0,049\ 9\ X + 0,023\ 1$
Middle distillate range B	$r = 0,016\ 6\ X - 0,019\ 5$	$R = 0,079\ 3\ X - 0,041\ 3$
Middle distillate range C	$r = 0,003\ 2\ X + 0,418\ 7$	$R = 0,063\ 2\ X - 0,003\ 6$
Domestic heating oil with FAME content of approx. 0,06 % (V/V)	0,004	0,015
Where <i>X</i> is the average of the two results being compared.		

11 Test report

The test report shall contain at least the following information:

- a) a reference to this European Standard, i.e. EN 14078;
- b) the type and complete identification of the product tested;
- c) the result of the test (see Clause 9);
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) the range the test method was applied for (A, B or C);
- f) the date of the test.

Annex A (informative)

Specific adjustments for calibration and dilution

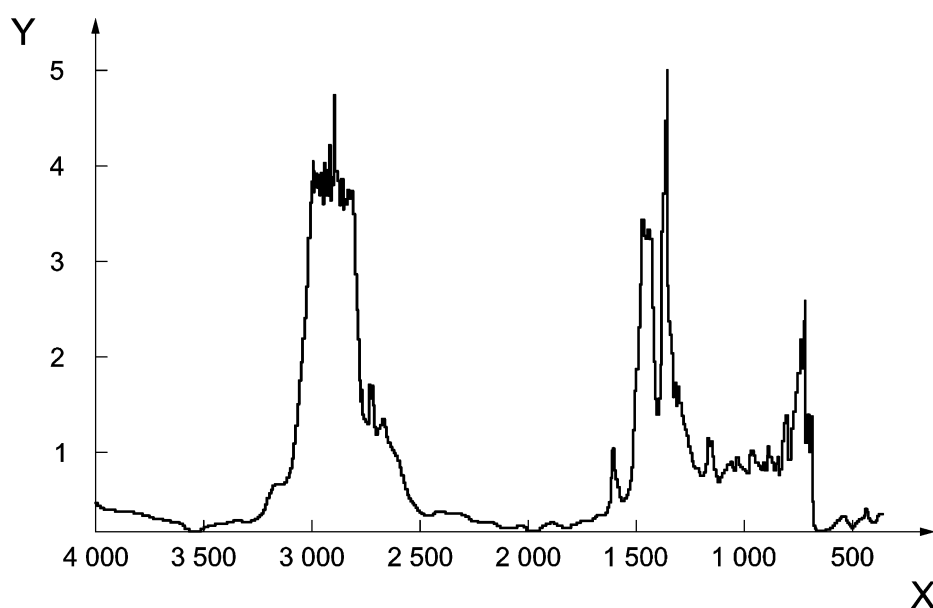
Table A.1 gives additional information for the selection of path length and dilution factor. All values in Table A.1 should be regarded as approximate reference values. The cells with bold figures indicate the presumed possible main range for the test method. Pay attention to the linearity. The best results can be achieved by determining the extinction preferably in the medium extinction range. Therefore, based on preliminary tests the optimum combination of cell and dilution factor should be selected. These approximate values may well differ entirely due to varying cell materials.

Table A.1 — Estimate data for the selection of path length and dilution factor

FAME g/l	FAME % (V/V)	Absorbance at:											
		Dilution											
		no	no	no	1:2	1:2	1:2	1:5	1:5	1:5	1:10	1:10	1:10
		Cell path length mm			Cell path length mm			Cell path length mm			Cell path length mm		
		(1,0)	0,5	0,1	(1,0)	0,5	0,1	(1,0)	0,5	0,1	0,5	0,1	0,05
0,009	0,001	0,003	0,001	—	—	—	—	—	—	—	—	—	—
0,044	0,005	0,012	0,006	0,001	0,01	—	—	—	—	—	—	—	—
0,09	0,010	0,023	0,012	0,002	0,01	0,01	—	—	—	—	—	—	—
0,22	0,025	0,058	0,029	0,006	0,03	0,01	—	0,01	0,01	—	—	—	—
0,44	0,050	0,12	0,059	0,012	0,06	0,03	0,01	0,02	0,01	—	0,01	—	—
0,88	0,100	0,23	0,12	0,023	0,12	0,06	0,01	0,05	0,02	—	0,01	—	—
2,21	0,25	0,59	0,29	0,059	0,29	0,15	0,03	0,12	0,06	0,01	0,03	0,01	—
4,42	0,50	1,17	0,59	0,12	0,59	0,29	0,06	0,23	0,12	0,02	0,06	0,01	0,01
8,83	1,00	2,33	1,17	0,23	1,17	0,58	0,12	0,47	0,23	0,05	0,12	0,02	0,01
13,25	1,50	3,51	1,75	0,35	1,75	0,88	0,18	0,70	0,35	0,07	0,18	0,03	0,02
17,66	2,00	4,67	2,33	0,47	2,33	1,17	0,23	0,93	0,47	0,09	0,23	0,05	0,02
22,08	2,50	5,84	2,92	0,58	2,92	1,46	0,29	1,17	0,58	0,12	0,29	0,06	0,03
26,49	3,00	7,00	3,50	0,70	3,50	1,75	0,35	1,40	0,70	0,14	0,35	0,07	0,04
30,91	3,5	8,17	4,09	0,82	4,09	2,04	0,41	1,63	0,82	0,16	0,41	0,08	0,04
35,32	4,0	9,34	4,67	0,93	4,67	2,34	0,47	1,87	0,93	0,19	0,47	0,09	0,05
39,74	4,5	10,5	5,26	1,05	5,26	2,63	0,53	2,10	1,05	0,21	0,53	0,11	0,05
44,15	5,0	11,7	5,84	1,17	5,84	2,92	0,58	2,34	1,17	0,23	0,59	0,12	0,06
48,57	5,5	12,9	6,43	1,29	6,43	3,21	0,64	2,57	1,29	0,26	0,65	0,13	0,06
52,98	6,0	14,0	7,01	1,40	7,01	3,50	0,70	2,80	1,40	0,28	0,70	0,14	0,07
57,40	6,5	15,2	7,59	1,52	7,59	3,80	0,76	3,04	1,52	0,30	0,76	0,15	0,08
88,30	10,0	23,4	11,7	2,34	11,69	5,84	1,17	4,67	2,34	0,47	1,17	0,23	0,12
132,45	15,0	35,1	17,5	3,51	17,53	8,77	1,75	7,01	3,51	0,70	1,76	0,35	0,18

176,60	20,0	46,7	23,4	4,67	23,37	11,69	2,34	9,35	4,67	0,93	2,34	0,47	0,23
220,75	25,0	58,4	29,2	5,84	29,21	14,61	2,92	11,69	5,84	1,17	2,92	0,58	0,29
264,90	30,0	70,1	35,1	7,01	35,05	17,53	3,51	14,02	7,01	1,40	3,51	0,70	0,35
353,20	40,0	93,5	46,7	9,35	46,75	23,37	4,67	18,70	9,35	1,87	4,68	0,93	0,47
441,5	50,0	—	58,44	11,68	—	29,22	5,84	—	11,69	2,34	7,01	1,17	0,58
423,84	60,0	—	—	14,03	—	0,00	7,01	—	0,00	2,81	9,35	0,00	0,70

Examples of infrared spectra are given in Figure A.1 and Figure A.2. Figure A.1 presents a FAME free diesel and Figure A.2 a regular diesel blend with 5 % (V/V) FAME.

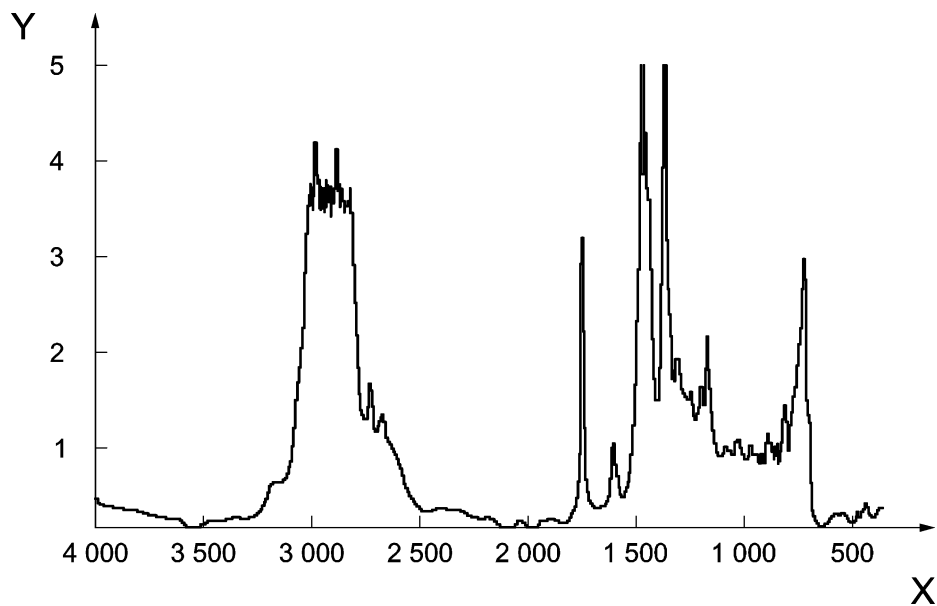


Key

X wave number (cm⁻¹)

Y absorption units

Figure A.1 — Example of an IR spectrum of diesel fuel (FAME free)



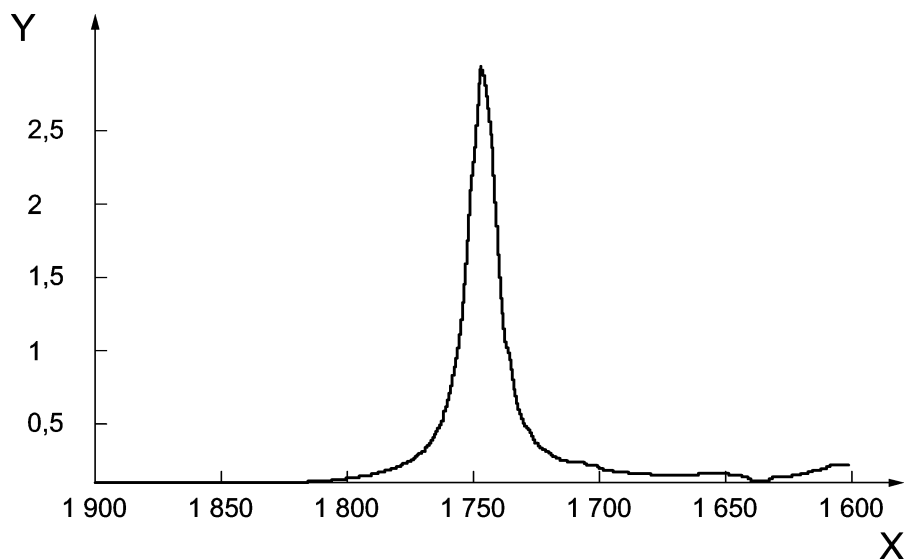
Key

X wave number (cm⁻¹)

Y absorption units

Figure A.2 — Example of an IR spectrum of diesel blend (5 % (v/v) FAME)

Figure A.3 shows the net absorbance after baseline correction of a sample containing 4 % (v/v) of FAME, measured in KBr cell with a path length of 490 μm . Background compensation was performed with FAME free diesel fuel.



Key

X wave number (cm⁻¹)

Y absorption units

Figure A.3 — Absorption according to base line procedure for diesel blend (4 % (v/v) FAME)

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