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Petroleum and related products — Applicability of diesel fuel test methods for Fatty Acid Methyl Esters (FAME) — Information and results on round robin tests

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Petroleum and related products - Applicability of diesel fuel test methods for Fatty Acid Methyl Esters (FAME) - Information and results on round robin tests

Produits pétroliers et produits relié - Application des méthodes d'examination de gazole en Methyl Acides Graz (UMAG) - Information ét résultats d'examination inter-laboratoir

Mineralölerzeugnisse und verwandte Produkte - Anwendbarkeit von Prüfverfahren für Diesel-Kraftstoffe auf Fettsäure-Methylester (FAME) - Informationen und Ergebnisse aus Ringversuchen

This Technical Report was approved by CEN on 18 June 2005. It has been drawn up by the Technical Committee CEN/TC 19.

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Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (CEN/TR 15160:2005) 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 document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

Introduction

This Technical Report gives the results of the round robin series of tests to evaluate different test methods on their compatibility on FAME which are referred to in:

- EN 14213: *Heating fuels – Fatty acid methyl esters (FAME) – Requirements and test methods*, and
- EN 14214: *Automotive fuels – Fatty acid methyl esters (FAME) for diesel engines – Requirements and test methods*.

CEN/TC 19 acknowledges Mrs. M.F. Benassy from Total, all project leaders of each test method as indicated in the annexes and all other participants in CEN/TC 19/WG 26 "FAME related fuel test methods" (see Annex A) for their contribution to this report.

1 Summary

One task under the European Mandate M/245 was to investigate the applicability of existing petroleum test method standards for fatty acid methyl esters (FAME). For this task CEN/TC 19 has founded a separate working group 26, which has validated 24 methods via round robins, including the development of new test methods.

The work of CEN/TC 19/WG 26 was aimed at the drafting of amendments for existing ISO standards (with test methods for fossil fuels). These amendments include the extension of the scope of the standards to biodiesel as well as the new precision data for biodiesel. To get these new precision data (repeatability & reproducibility), WG 26 has done a major effort to validate all relevant test methods on request of the Task Force of WG 24 and WG 25 dealing with the specifications.

2 Background

CEN has been asked by the Commission (Mandate M/245) to elaborate standards for fatty acid methyl esters (FAME) to be used as sole fuel for heating and diesel engines and as an additive to mineral oil based fuels. This work has been divided amongst two Technical Committees, of which CEN/TC 19 was responsible for determining the applicability of already existing petroleum type test method standards to both pure FAME and to blends. This included comparing precision data, generating new reproducibility and repeatability figures and developing new test method standards if necessary.

For this task CEN/TC 19 has founded a separate working group with the following task: “Verification of applicability to biodiesel (for use in diesel engines as well as for use as heating oil) of existing test methods (to be validated via round robins) and development of new test methods, if necessary”. The convenor of this WG 26 was Mrs. Benassy of Total (former ELF Antar), France. For a list of participants in this WG, see Annex A.

The work of WG 26 was aimed at the drafting of amendments for existing ISO standards (with test methods for fossil fuels). These amendments include the extension of the scope of the standards to biodiesel as well as the new precision data for biodiesel. To get these new precision data (repeatability & reproducibility), WG 26 has organised several round robin studies. Besides that it had several consultations with other WG's responsible for existing or revising of test methods and with the WG 24 (automotive diesel) and WG 25 (heating fuels) dealing with the specifications.

Scope of the work was investigating the applicability of each test method towards FAME as 100 % diesel fuel. The repeatability and reproducibility were determined if the test method was applicable. Furthermore, it was determined whether the method was also applicable to blends of FAME and diesel fuel.

This Technical Report gives an overview of the work of WG 26 and of the results in relation to applicability of each test method and the determined precision data, giving the basic conclusions and data. A full report with all data per test method has been presented within CEN/TC 19. This report is the basis of two standards:

1. EN 14213:2003, *Heating fuels — Fatty acid methyl esters (FAME) — Requirements and test methods*

2. EN 14214:2003, *Automotive fuels — Fatty acid methyl esters (FAME) for diesel engines — Requirements and test methods*

Moreover, the results of the study by WG 26 have been input in the revision of the automotive diesel fuel specification standard (EN 590).

NOTE For the purposes of this document, the terms “% (*m/m*)” and “% (*V/V*)” are used to represent respectively the mass fraction and the volume fraction.

3 Basis of the work

The following list of standards[1] has been investigated in the period 1999 until 2002:

— EN 116:1997, *Diesel and domestic heating fuels – Determination of cold filter plugging point*

— EN 590: 1998, *Automotive fuels – Diesel – Requirements and test methods*

— EN 12662:1998, *Liquid petroleum products – Determination of contamination in middle distillates*

— EN 12634:1998, *Petroleum products and lubricants – Determination of acid number – Non-aqueous potentiometric titration method*

— prEN 14078:2001, *Liquid petroleum products – Determination of fatty acid methyl esters (FAME) in middle distillates – Infrared spectroscopy method*

— EN 22719:1993, *Petroleum products and lubricants – Determination of flash point - Pensky-Martens closed cup method (ISO 2719:1988)*

— EN 23015:1994, *Petroleum products – Determination of cloud point (ISO 3015:1992)*

— EN ISO 2160:1998, *Petroleum products – Corrosiveness to copper – Copper strip test (ISO 2160:1998)*

— EN ISO 3104:1998/C2:1999, *Petroleum products – Transparent and opaque liquids – Determination of kinematic viscosity and calculation of dynamic viscosity (ISO 3104:1997)*

— EN ISO 3405:2000, *Petroleum products – Determination of distillation characteristics at atmospheric pressure (ISO 3405:2000)*

— EN ISO 3675:1998, *Crude petroleum and liquid petroleum products – Laboratory determination of density – Hydrometer method (ISO 3675:1998)*

— prEN ISO/DIS 3679:2001[2)], *Petroleum products - Determination of flash point - Rapid equilibrium closed cup method*

— EN ISO 4259:1995, *Petroleum products – Determination and application of precision data in relation to methods of test (ISO 4259:1992/Cor.1: 1993)*

— EN ISO 5165:1998, *Petroleum products – Determination of ignition quality of diesel fuels – Cetane engine method (ISO 5165:1998)*

— EN ISO 10370:1995, *Petroleum products – Determination of carbon residue – Micro method (ISO 10370: 1993)*

— EN ISO 12185:1996, *Crude petroleum and petroleum products – Determination of density – Oscillating U-tube method (ISO 12185:1996)*

— EN ISO 12205:1996, *Petroleum products – Determination of the oxidation stability of middle distillate fuels (ISO 12205:1995)*

— EN ISO 12937:2000, *Petroleum products – Determination of water – Coulometric Karl Fisher titration method (ISO 12937:2000)*

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[1] These standards are referred to in the document as either the EN ISO or the ISO number (given in brackets), but here the correct reference of the actual, updated standard is given.

2 Succeeded by EN ISO 3679:2004, *Petroleum products - Determination of flash point - Rapid equilibrium closed cup method (ISO 3679:2004)*

— EN ISO 13759:1996, *Petroleum products – Determination of alkyl nitrate in diesel fuels – Spectrometric method (ISO 13759:1996)*

— EN ISO 14596:1998/C1:1999, *Petroleum products – Determination of sulfur content – Wavelength-dispersive X-ray fluorescence spectrometry (ISO 14596:1998/C1:1999)*

— prEN ISO/DIS 20846:2002, *Petroleum products – Determination of total sulfur content of liquid petroleum products – Ultraviolet Fluorescence Method*

— ISO 3016:1994, *Petroleum products – Determination of pour point*

— ISO 3987:1994, *Petroleum products – Lubricating oils and additives – Determination of sulfated ash.*

— ISO 6245:2001, *Petroleum products – Determination of ash*

— ISO 12156-1:1997, *Diesel fuels – Assessment of lubricity using the high-frequency reproducing rig (HFRR) – Part 1: Test method (including Cor. 1:1998)*

— ASTM D 1160:99, *Distillation of Petroleum Products at Reduced Pressure*

4 Details of the round robin

After having considered all existing (statistical) data in the standards, WG 26 has decided for which test methods an inter-laboratory study was necessary. For some methods the data were sufficient, for others the decision on applicability was made after internal discussions.

For each of the remaining test methods a round robin study was initiated and a project leader from WG 26 was assigned. The number of participating laboratories changed with each standard, going from 7 to 15. There was a variety in countries but from France, Italy, Austria and Germany there were always laboratories participating.

Samples from actual FAME products from different feed-stocks were supplied by ITERG, France. Distribution of the samples, together with the correct protocol, was organised by WG 26. All data received from the labs were statistically treated according to EN ISO 4259.

For pure FAME, inter-laboratory testing has been completed for the following determination methods[3]: density (EN ISO 3675 and EN ISO 12185), viscosity (EN ISO 3104), flash point (prEN ISO/DIS 3679), sulfur content (EN ISO 20846), carbon residue (EN ISO 10370), cetane number (EN ISO 5165), ash content (ISO 3987), water content (EN ISO 12937), copper corrosion (EN ISO 2160), acid number (EN 12634). For 5 % blends of FAME in diesel fuel only testing on determination of alkyl nitrate content (EN ISO 13759) has been done by WG 26. For sulfur content (five methods) and cetane number (one 5 % blend sample tested among four petroleum type samples) studies have been done by other CEN/TC 19 groups, while they were conducting their round robin investigations.

5 Results

All results are compiled in Table 1 and Table 2. The conclusions on applicability, repeatability and reproducibility are given for pure FAME and one for blends of 5 % in diesel. The results of the sulfur study on the blends have been left out of the table, as this is part of the report of WG 27. However, their results have been incorporated in the drafting of the two FAME specification standards.

Detailed reports on studies on flash point, distillation, carbon residue, corrosiveness to copper and the FAME detection method are given in Annex B to Annex H. Other short reports are not available.

[3] In brackets the documents which have been used as a basis for the lab-protocol are given. After the study these standards might have been updated.

Table 1 - Details of inter-laboratory test programme
Methods for pure FAME

Standard	Applicable	Precision similar to std [a]	Repeatability	Reproducibility	Comments
EN ISO 3675 Density at 15 ºC	Yes	Yes	0,000 5 g/cm^3	0,001 2 g/cm^3	Conversion tables applicable
EN ISO 12185 Density at 15 ºC	Yes	Yes	0,2 kg/m^3	0,5 kg/m^3	Conversion tables not applicable
EN ISO 3104 Viscosity at 40 ºC	Yes	Yes	0,11 %	1,8 %	
EN 22719 Flash point (Pensky-Martens)	No				
prEN ISO/DIS 3679 Flash point (equilibrium)	Yes	r: Yes R: No	0,022$X^{0,9}$ °C	15 °C	- Electronic detection - 2ml test portion
EN ISO 20846 Sulfur content	Yes	Yes	0,028 5 X + 2 (X in mg/kg)	0,108 8 X + 2 (X in mg/kg)	
EN ISO 10370 Carbon residue	Yes	Yes	0,077 $X^{2/3}$ (X in %)	0,245 $X^{2/3}$ (X in %)	10% residue obtained by ASTM D1160 run at 10 mmHg
EN ISO 5165 Cetane number	Yes	r: No R: Yes	2,4	5	
ISO 3987 Ash content (sulphated)	Yes	Yes	0,06 $X^{0,85}$	0,142 $X^{0,.85}$	
EN ISO 12937 Water content	Yes	Yes	0,0187 4 $X^{0,5}$ (X in %)	0,0687 7 $X^{0,5}$ (X in %)	
EN ISO 2160 Copper corrosion	Yes		No value		Not a numerical result
EN ISO 3405 Distillation	No				
ASTM D 1160 Distillation	Yes	Yes	2 °C (90% distilled)	3 °C (90% distilled)	run at 10mmHg
ISO 3016 Pour point	Yes				
EN 116 CFPP	Yes				As stated by CEN/TC19/WG14
EN 12662 Total contamination					Wait for method revision
EN 12634 Acid number	Yes	r: Yes R: No	0,05 X (X in mg KOH/g)	0,065+ 0,281 X (X in mg KOH/g)	

[a] if YES: repeatability and reproducibility quoted are the one of the reference standard,
if NO: repeatability and reproducibility quoted are the one obtained during the inter-laboratory testing

Table 2 - Details of inter-laboratory test programme
Methods for blends of FAME, (up to 5 % volume)

Standard	Applicable	Precision similar to std[a]	Repeatability	Reproducibility	Comments
EN ISO 3675 Density at 15 ºC	Yes	Yes	0,000 5 g/cm^3	0,001 2 g/cm^3	No inter-lab. test
EN ISO 12185 Density at 15 ºC	Yes	Yes	0,2 kg/m^3	0,5 kg/m^3	No inter-lab. test
EN ISO 3104 Viscosity at 40 ºC	Yes	Yes	0,11%	1,8%	No inter-lab. test
EN 22719 Flash point (P.M.)	Yes	Yes	2 °C (at 101°C)	3,5 °C (at 101°C)	Procedure A. Decision made after short study
EN ISO 14596 Sulfur content	Yes	Yes	0,037 X + 1,9 (mg/kg)	0,063 X + 3,2 (mg/kg)	
EN ISO 10370 Carbon residue	Yes	Yes	0,077 $X^{2/3}$ (X in %)	0,245 $X^{2/3}$ (X in %)	No inter-lab. test
EN ISO 5165 Cetane number	Yes	Yes	0,9 at 52	4,3 at 52	
ISO 6245 Ash content	Yes	Yes			Decision made after short study
EN ISO 12937 Water content	Yes	Yes	0,0187 4 $X^{0,5}$ (X in %)	0,068 77 $X^{0,5}$ (X in %)	No inter-lab. test
EN ISO 2160 Copper corrosion	Yes		No value		Decision made after short study
EN ISO 13759 Alkyl nitrate	Yes	Yes			Decision made after short study
ISO 12156-1 Lubricity	Yes				
EN ISO 3405 Distillation	Yes	Yes			Decision made after short study
EN ISO 12205 Oxidation stability	Yes	Yes			No inter-lab. test
ISO 3016 Pour point	Yes	Yes			As advised by CEN/TC19/WG14
EN 23015 Cloud point	Yes	Yes			As advised by CEN/TC19/WG14
EN 116 CFPP	Yes	Yes			As advised by CEN/TC19/WG14
EN 12662 Total contamination					Wait for method revision
EN 14078 Fame content by IR	Yes		r = 2,5 g/l	X = 100 g/l R = 7,7 g/l X > 100 g/l R = 12,7 g/l	

[a] if YES: repeatability and reproducibility quoted are the one of the reference standard,
if NO: repeatability and reproducibility quoted are the one obtained during the inter-laboratory testing

6 Conclusion

6.1 Pure FAME

Flash point by EN 22719 and distillation by EN ISO 3405 where found to be not applicable to FAME samples after an internal study.

All other methods were found applicable to FAME samples with similar precision data as for petroleum products, except for:

- Cetane number: r is significantly higher
- Acid number: R is significantly higher
- Flash point (prEN ISO/DIS 3679): R is significantly higher

No improvement of these methods could be expected in the time given by the Mandate, but information has been given to the different method working groups for possible future work.

No inter-laboratory test has been conducted on cold properties (EN 116), following advice of CEN/TC19/WG14. Pour point and cloud point methods where considered to be applicable.

6.2 5 % blends of FAME in diesel fuel

Considering the work done on pure FAME and the known method on diesel fuel, the experts agreed on the fact that the following methods where applicable with the same precision data than already stated in the standard, without running an inter-laboratory testing:

— density (EN ISO 3675 and EN ISO 12185),

— viscosity (EN ISO 3104),

— flash point (EN 22719),

— carbon residue (EN ISO 10370),

— cetane number (EN ISO 5165),

— ash content (ISO 6245),

— water content (EN ISO 12937),

— copper corrosion (EN ISO 2160),

— distillation (EN ISO 3405),

— pour point (ISO 3016),

— cloud point (EN 23015),

— lubricity (ISO 12156-1), and

— oxidation stability (EN ISO 12205).

EN ISO 13759 (determination of alkyl nitrate content) was tested and found adequate to be used on products containing FAME. Cetane number was tested by another WG and found adequate, too.

Annex A
(informative)

List of participants in working group 26 activities

The following people have been active in WG 26 "FAME related fuel test methods" and in the underlying laboratory work.

Name	Company
Mrs. M.F. BENASSY	TOTALFINAELF (Convenor)
P. FERRARI	EURON
Dr. T. FEUERHELM	DIN/FAM
Dr. Ing. J CONNEMANN	OELMUEHLE LEER CONNEMANN GmbH Co
Dipl. Ing. B. BLAICH	Robert BOSCH GmbH FV/FLA
Dr. J. FISCHER	AG QM Biodiesel e.V.
Dr. Ing. K. SCHARMER	GET - Gesellschaft Für Entwicklungstechnik
Dr. Ing. TH. GOTTSCHAU	FNR
X. MONTAGNE	IFP
Mrs. N. DAVIAS	RENAULT Technocentre
Ms. U. KIISKI	FORTUM Oil and Gas OY, Technology Centre
D. KARNER	OMV
Dr. F. van DIEVOET	BfB Oil Research
Dott. P. TITTARELLI	Stazione Sperimentale Combustibili
Dott. M. VIGO	UNIONE PETROLIFERA
M.L. DAANE	SHELL Research and Technology Centre
J. WOLDENDORP	SHELL Research and Technology Centre
Dr. M. HUTTER	IMU
B. DUFRENOY	NOVAOL
Ms. F. LACOSTE	ITERG
A. FRASER	NOVAOL
R.W. HOOKS	Shell Research Ltd. / R.W. HOOKS consultancy
F. TORT	TOTALFINAELF
Ing. U. JANISCH	OELMUEHLE LEER CONNEMANN GmbH Co
W. DORMER	ARAL AG
J. PHIPPS	INSTITUTE OF PETROLEUM
L. BURMAN	SGS SWEDEN AB
U. OSTAN	SAYBOLT SWEDEN AB
J. BERG	SWEDISH Farmer Supply and Crop Marketing Association
D. MEHLIS	PETRO LAB GmbH
T. WILHARM	ASG
B. CAHILL	PSA CITROEN/PEUGEOT
F. MORDRET	ITERG
F. HEGER	OMV
A. PHILIPPSEN	OELMUEHLE LEER CONNEMANN GmbH Co
J. MUELLER_BELAU	Deutsche Shell AG

Dr. J. BOSSE	VOLKSWAGEN AG
T.J. BERRYMAN	TIM-BER Support
S. PAGLIANTI	NOVOAL Srl
M. MARCHI	IES Raffineria di Mantova
G. ARALDI	TAMOIL
S. CATALANO	SGS Redwood
G. LO BAIDO	Esso Technical Services
G. PINELLI	Stazione Sperimentale Combustibili
F. AVELLA	Stazione Sperimentale Combustibili
E. COLOMBO	SARPOM
Mrs. C. PUEL	ELF ANTAR / TOTALFINAELF

Annex B
(informative)

Report of the result of the round robin on 'determination of flash point using the rapid equilibrium close cup method' (prEN ISO/DIS 3679)

B.1 Scope

In this round robin the applicability of test method EN ISO 3679 "Determination of flash point - Rapid equilibrium close cup method" towards FAME as 100 % diesel fuel is investigated. The precision, repeatability and reproducibility, for this product versus the stated precision for petroleum products were also investigated.

NOTE This study has been supervised by Mr. M.L. Daane and Mr. J. Woldendorp.

B.2 Assessed standard documents

EN 22719:1993, *Petroleum products and lubricants – Determination of flash point – Pensky-Martens closed cup method (ISO 2719:1988)*

prEN ISO/DIS 3679:2002 – *Determination of flash point - Rapid equilibrium closed cup method (ISO/DIS 3679:2002)*

EN ISO 4259:1995, *Petroleum products – Determination and application of precision data in relation to methods of test (ISO 4259:1992, including Cor.1: 1993).*

B.3 Background

The actual test method at the date of investigation for flash point for EN 590 diesel was EN 22719, the Pensky-Martens closed cup test method[4)]. The applicability of this test method for 100 % FAME has been studied and found unsuitable for two reasons.

1) the precision of this method for products containing trace amounts of highly flammable material in relatively high flash point material is very poor;

NOTE The flash point for fatty acid methyl esters is high (> 160 °C). Methanol is used for the manufacturing of FAME. Methanol is to a large extent removed from the final product, but remnants may still be present. The current specification for 100 % FAME allows 0,2 % (*m/m*) methanol max. Methanol as such has a flash point of 11 °C. The use of non-equilibrium test methods for the determination of flash point for products containing trace amounts of highly flammable material (i.e. like FAME containing methanol) give poor repeatable test results due to

i) non homogeneous heat transfer conditions, and

ii) unfavourable V/L (Vapour/Liquid) conditions in the test cup.

2) optical detection is unsuitable, because methanol burns (and flashes) with a colourless, invisible flame.

NOTE The flash point of FAME is caused by methanol, which gives a colourless, invisible flame. Even experienced operators have difficulties to detect this flame using their eyes.

4 At the date of publication of this document a revision (EN ISO 2719) has been published in November 2002.

Both procedures A and B of EN 22719 were tested (see Table B.1). Procedure A gave repeatable results for FAME A, giving a greenish flame, caused by the FAME itself, i.e. all methanol is evaporated during the test without giving a flash. Procedure A gave poorly repeatable results for FAME B. Procedure B gave poorly repeatable results for FAME A and B. These results clearly demonstrated that EN 22719 is not applicable for 100 % FAME.

Based on the above, the most suitable test method is an equilibrium test method with a mandatory non-optical flash detection. According to state of the art, EN ISO 3679 test method was considered to be most appropriate to run a round robin with.

Table B.1 — Flash point determinations of 100 % FAME by EN 22719

FAME A		FAME B	
Procedure A	Procedure B	Procedure A	Procedure B
166	(134 [a]) 160	(118 [b]) 126	116
166	(132 [a]) 154	(122 [a]) 126	120
168	(120 [b]) 156	(118 [b]) 124	122
	(122 [b]) 160	(126 [a]) 128	124
	140	130	
	134	134	
	148		

[a] very weak flash observed, flame blown out, no flame observed

[b] flame blown out (sucked into the cup)

B.4 Pre-study for round robin with prEN ISO/DIS 3679

B.4.1 Set-up

The suitability of prEN ISO/DIS 3679 was initially tested at SRTCA. For this purpose a flash point tester was got on loan. The first tests showed that repeatable flashpoints results for FAME, containing methanol at several concentrations, can be achieved using this apparatus (see Figure B.1, flash point App. 1, 2 ml). Therefore SRTCA decided to get possession of such a flash point tester. The samples, tested on apparatus 1, were also tested on this new apparatus (see Figure B.1, flash point App. 2, 2 ml). It was noted that the flashpoints were somewhat lower on apparatus 2. The curve through the flashpoints is S-shaped; the left end site is limited by the flash point of FAME without methanol, the right end site is asymptotically limited by the flash point of methanol, the steep middle of the curve is around the methanol concentration of 0,2 % (*m/m)* (i.e. the current FAME specification).

According to prEN ISO/DIS 3679, a specified volume is introduced into the test cup, which is maintained at the temperature of the estimated flash point of the material under test. After a specified time, a test flame is applied and the presence or absence of a flash observed. For flashpoints up to and including 100 °C, the specified test portion is 2 ml and the specified time is 1 minute; for flashpoints above 100 °C the specified test portion is 4 ml and the specified time is 2 min.

When 4 ml FAME is introduced instead of 2 ml, then much lower flashpoints are achieved (see Figure B.1, flash point, App. 2, 4 ml). Clearly the two curves through the flashpoints derived by 2 ml and 4 ml do not overlap, which is partly accounted for by differences in V/L (Vapour/Liquid) ratios: V/L for 2 ml and 4 ml test portions are 9/1 and 8/2 respectively. It was noted that the flashpoint results for 100 % FAME without methanol and 100 % FAME with methanol >0,5 % (m/m) were less sensitive to sample volume.

Generally the flashpoints for 100 % FAME will be above 100 °C, so a test portion of 4 ml is required. However, for samples containing more than ca. 0,17 % (m/m) methanol, the flashpoint will be < 100 °C when using 4 ml test portions. For those samples a 2 ml test portion is required according to the test method. However, for samples containing ca. 0,17 % (m/m) to ca. 0,25 % (m/m), flashpoints > 100 °C will be expected when using a 2 ml test portion, so a 4 ml test portion is required, etc.

Obviously the test method cannot be used for FAME samples regarding the specified test portion. Hereto the test method had to be modified, i.e. specifying a single test portion for FAME samples, irrespective of flashpoint. The fixation of the test portion to either 2 ml or 4 ml was up for debate.

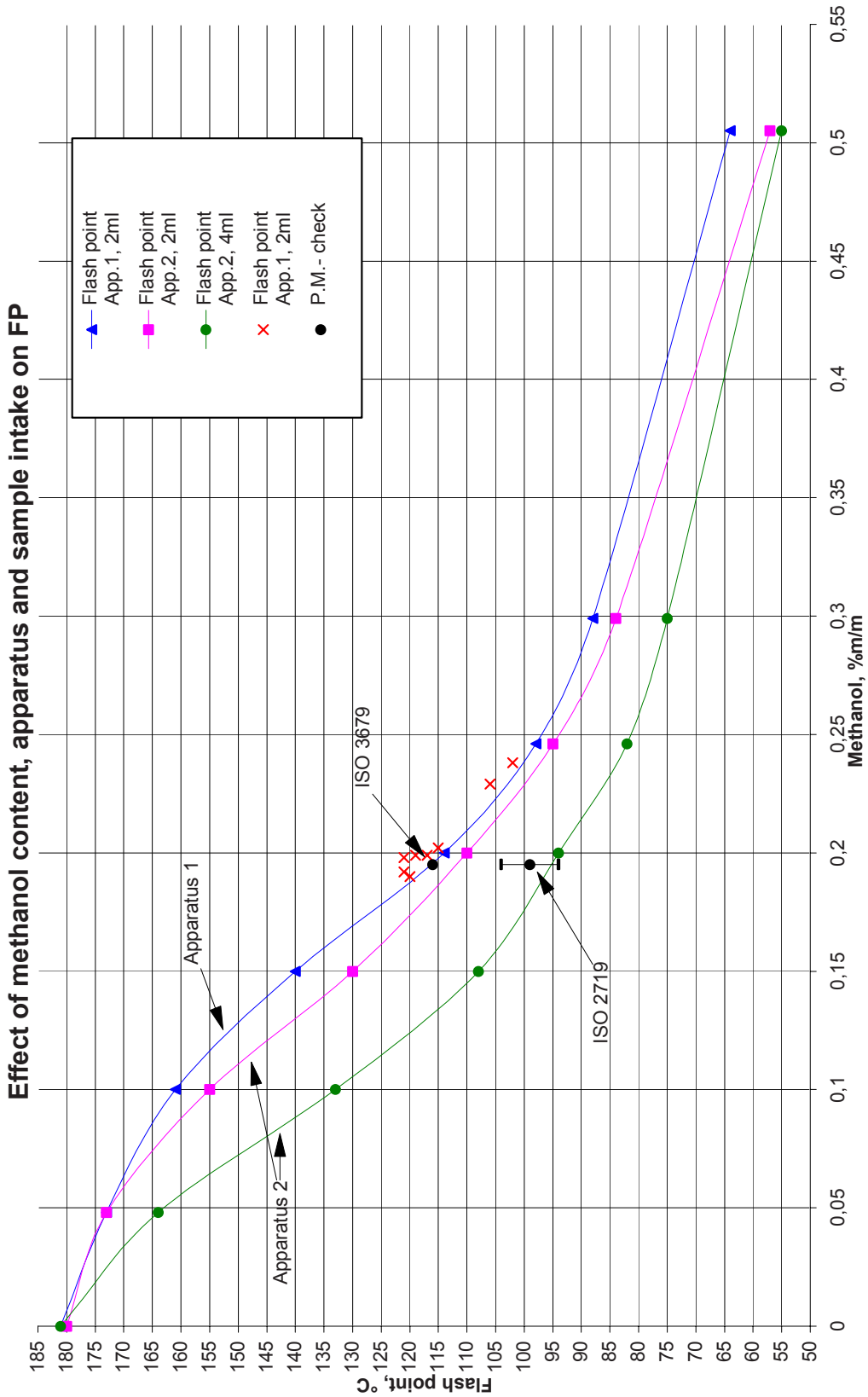

Figure B.1 — EN ISO 3679 at 100 % FAME

B.4.2 2 ml test portion

pro: the flashpoint limit for transportation regulation is around 65 °C, which is based on 2 ml sample intake

contra: flashpoint for 100 % FAME will generally be >110 °C, so test method is not performed accordingly

B.4.3 4 ml test portion

pro: flashpoint for 100 % FAME will generally be >100 °C, so the original test method is performed accordingly most of the time.

contra:

1) for samples containing ca. >0,17 % (m/m) methanol, flashpoint will be <100 °C, so original test method is not performed accordingly, and

2) there is no comparison with other fuels, for which generally 2 ml sample volume is required.

After advice from ISO/TC 28 and ISO/TC 35, it was decided to specify a 2 ml test portion for the flashpoint round robin.

B.5 Details of the round robin

A total of 11 laboratories in 4 countries (i.e. United Kingdom, Germany, France and The Netherlands) took part in the round robin. 10 laboratories used the Seta Flash apparatus and 1 laboratory used the Normalab NPV 210 apparatus. The sample set comprised two types of FAME, i.e. rapeseed (EMC, samples 1-5) and sunflower (EMT, samples 6-10). These FAME's were first purged with nitrogen to remove any methanol and were successively spiked with 0 % (*m/m*), 0,05 % (*m/m*), 0,1 % (*m/m*), 0,2 % (*m/m*) and 0,5 % (*m/m*) methanol. The FAME batches were sampled into 250 ml flasks. All participants received a test protocol and prEN ISO/DIS 3679 (see B.6). The round robin results were statistically treated by EN ISO 4259.

B.6 Round robin protocol specific measurement instructions

In addition to the requirements of prEN ISO/DIS 3679, the following measurement instructions apply:

— 2 ml test portions are used for all flash points tests, even those carried out at temperatures greater than 100 °C.

— The occurrence of a flash is to be detected by the flash point tester's automatic detector only.

— Determine the flash point to the nearest 0,5 °C.

— Perform duplicate flash point determinations. Also report the atmospheric pressure at each test. Fill the duplicate results and the atmospheric pressures in the reporting table.

— For indicative purposes only, the samples' flashpoints are in the neighbourhood of:
Samples 1 and 6: 180 °C
Samples 2 and 7: 170 °C
Samples 3 and 8: 150 °C
Samples 4 and 9: 110 °C
Samples 5 and 10: 60 °C

B.7 Results

The results are displayed in Table B.2. Lab No. 6 was not able to test all samples due to instrument failure. Lab No. 10 used an apparatus suitable for flashpoint testing up to 110 °C, so it was not able to test all the samples. The results from lab No. 4 were clearly outliers; on inquiry, this lab ignored the protocol and applied a 4 ml test portion instead of the required 2 ml test portion.

Table B.2 — Round robin results

	Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
L1	Result 1	180	169,5	159,5	115	61,5	179,5	169,5	159	114	62
	Result 2	179	169,5	157,5	113,5	60	179	170	157,5	114	63
L2	Result 1	177,5	172	166,5	117,6	64,2	176,5	173,5	162	120,6	63,2
	Result 2	177,5	172,5	166	118,6	64,7	177	174	162	121,1	63,7
L3	Result 1	181	173	156	105	59	181	171	190	109	57
	Result 2	182	174	156	105	59	182	173	193	109	58
L4	Result 1	164	151	140	80	57	175	152	107	42	50
	Result 2	164	153	142	78	56	174	150	106	41	50
L5	Result 1	184	177	166,5	126	63	184	175	165	122	61,5
	Result 2	184	177,5	167	127	63	183,5	175,5	165,5	123	62
L6	Result 1	173,5	164				174	159,5			
	Result 2	175	165				172,5	159			
L7	Result 1	181	173	160	116,5	64,5	180	171	158	114	63
	Result 2	180,5	174	159	116,5	64	179,5	171,5	157	113,5	63,5
L8	Result 1	179,7	176,3	169,8	141,3	65,9	183,4	175,8	168,9	143,9	65,4
	Result 2	184,2	176,3	169,3	149,3	64,4	183,4	179,4	168,4	139,4	61,3
L9	Result 1	179,5	177	173	153	89	178,5	174	171,5	164	81,5
	Result 2	179,5	177,5	173	152,5	90	178	174,5	171	164,5	81
L10	Result 1					85					85
	Result 2					85					85
L11	Result 1	176,5	166,5	161,5	112	71,5	177,5	170,5	161	116,5	65
	Result 2	176,5	168	161	111,5	71,5	178	171	161,5	116	65

Samples 1-5:	EMC (rapeseed)	Samples 1 and 6:	0 % (m/m) methanol
Samples 6-10:	EMT (sunflower)	Samples 2 and 7:	0,05 % (m/m) methanol
		Sample 3 and 8:	0,1 % (m/m) methanol
		Sample 4 and 9:	0,2 % (m/m) methanol
		Sample 5 and 10:	0,5 % (m/m) methanol

Since labs No. 4 and 10 were not using the right protocol or the right apparatus, they were excluded from the statistical analysis. The statistical evaluation was performed using EN ISO 4259.

Lab No. 3 was detected as an outlier in the results of sample 8. Lab No.8 was detected as an outlier in the results of samples 1, 4, 7, 9 and 10. Lab No. 9 was detected as an outlier in the results of samples 4, 5, 9 and 10. The statistical analysis is summarised in Table B.3.

Table B.3 — Statistical analysis of flashpoint round robin

Sample	1	2	3	4	5	6	7	8	9	10
Average	179,2	172,4	163,9	115,4	64	179,3	172,4	163,5	116,1	62,2
r	1,8	1,9	2	2,2	2,1	1,7	2,2	1,9	1,4	1,7
r %	1,0	1,1	1,2	1,9	3,3	0,9	1,3	1,2	1,2	2,7
R	10,3	14,7	19,9	26	13,8	10,9	7,2	17,9	18,1	9,4
R %	5,7	8,5	12,1	22,5	21,6	6,1	4,2	10,9	15,6	15,1
n	8	9	8	6	7	9	7	7	6	6
Ex. Lab V	L8			L8			L8		L8	L8
Ex. Lab M				L9	L9		L6	L3	L9	L9

The repeatability can be considered as constant over the interval 62°C - 179°C. The global repeatability is estimated at 1,9 °C, which is somewhat higher than the repeatability stated in prEN ISO/DIS 3679 for petroleum and related products (i.e. 0,022 $X^{0,9}$ for flashpoint > 70 °C) for flashpoints up to 150 °C but is similar to the repeatability stated in prEN ISO/DIS 3679 at higher flashpoints.

The reproducibility (R), however, cannot be considered as constant: it varies between 10 °C and 20 °C, except for one high-flyer at 26 °C. The reproducibilities found for this round robin are significantly higher than the reproducibilities stated in prEN ISO/DIS 3679 (i.e. 0,083 $X^{0,9}$ for flashpoints > 70 °C). The R is maximal around the 0,2 % (m/m) methanol concentration (samples 4 and 9) and decreases (significantly) going to higher and lower concentrations. So there clearly is a relationship of R and the methanol (flashpoint) concentration. However, the standard software is not able to linearise this (Gaussian) relationship and consequently estimates the global reproducibility to 15,8 °C. This implies that the reproducibility is significantly underestimated at the flash point specification (i.e. flashpoint at 0,2 % (m/m) methanol) and is significantly overestimated at higher or lower methanol concentrations.

Based on the physics involved one might expect that this type of error distribution is not that exceptional and will occur more frequently, certainly for any flash point caused by "trace contamination" (mixing a high flash point material with trace amounts of low flash material). And it is exactly this situation, which has the most potential hazard in terms of flammability. This issue has been addressed to the standardization bodies (i.e. ASTM and ISO TC 28 / TC 35).

B.8 Conclusion

prEN ISO/DIS 3679 and its later successor, the test method for the determination of flashpoint, is applicable for 100 % FAME, when the following modifications are implemented:

1) For FAME samples, 2 ml test portion and 1 min stabilisation time are dictated.

2) The use of the flash point detector is mandatory for testing FAME samples.

The repeatability derived for the round robin can be regarded as similar to the method's repeatability. The derived reproducibility, however, was significantly higher than the method's reproducibility.

Annex C
(informative)

Report of the result of the round robin series on 'distillation of petroleum products at reduced pressure' (ASTM D 1160)

C.1 Scope

In this round robin the applicability of test method ASTM D 1160 "Distillation of Petroleum Products at Reduced Pressure" towards FAME as 100 % diesel fuel is investigated. The repeatability and reproducibility are determined if the test method was applicable.

NOTE This study has been supervised by Mr. M.L. Daane.

C.2 Assessed standard documents

EN ISO 3170:1998, *Petroleum liquids – Manual sampling (ISO 3170:1988)*

EN ISO 4259:1995, *Petroleum products – Determination and application of precision data in relation to methods of test (ISO 4259:1992/Cor.1:1993)*

ASTM D1160 :95, *Distillation of Petroleum Products at Reduced Pressure*

C.3 Details of the round robin

A total of 5 laboratories in 4 countries took part in the round robin. They are labelled 1 to 5.

4 laboratories used automatic instrument and 1 laboratory used a manual instrument (laboratory N°3).

5 FAME samples were supplied by Elf. Their composition is given in Table C.1.

Table C.1 — Composition of FAME samples

Sample	Sample WI 18-1	Sample WI 18-2	Sample WI 18-3	Sample WI 18-4	Sample WI 18-5
Type of FAME	100 % FAME from Rape	100 % FAME from Sun Flower	100 % FAME from Soya	100 % FAME from animal (Tallow)	100 % FAME from used oil
Country	Europe	Europe	United Stated	United States	United States

In addition to the requirements of ASTM D 1160 the following measurement instructions were applied:

- Test carried out at a pressure of 10 mm Hg and at a uniform rate of 6 ml/min to 8 ml/min.
- For each sample, the test has been carried out in duplicate.

- Results reported rounded at 0,1°C.

These recommendations came out from an applicability study done by Mr. M.L. Daane and Mr. M. Hutter.

Handling of the samples was done according to EN ISO 3170.

The statistical treatment of this round robin was performed according to EN ISO 4259.

C.4 Results

Table C.2 — Results of test method ASTM D1160 for different laboratories and samples

Labs		Sample WI 18.1	Sample WI 18.2	Sample WI18.3	Sample WI 18.4	Sample WI 18.5
1	Initial	317,9	280,5	305	304,4	295,3
	5 %	348	346	345,5	336,1	338,2
	10 %	348,7	347,7	345,7	338,8	341
	20 %	349	348,1	346,8	341,9	342
	30 %	349,3	348,8	347,9	343,9	340,4
	40 %	350,1	349,6	348,2	345,2	342,1
	50 %	350,1	349,3	349	346,7	344,4
	60 %	351	350,3	349,8	348	345,6
	70 %	351,5	350,7	350,4	350	347,9
	80 %	351,9	351,4	351	351,6	350
	90 %	353,3	352,3	352,4	353,7	352,6
	95 %	358,5	354,5	353,8	356,4	354,4
	Final	436,6	398,6	416	404,6	393,9
1	Initial	316,9	279	313,1	306,3	290,2
	5 %	348,5	345,9	345,5	337,1	337,7
	10 %	349	347,4	345,8	338,9	340,1
	20 %	349,1	348,4	344,8	341,8	338,7
	30 %	350,1	348,8	345,4	343,5	340,7
	40 %	350,6	349,1	347,3	345	342,1
	50 %	350,9	349,1	348,3	346,1	344,3
	60 %	351,6	350,1	348,7	347,4	345,9
	70 %	351,9	351	349,9	349,4	347,8
	80 %	352,6	351,6	350,6	351,1	350,2
	90 %	354,4	352,3	351,5	353,8	352,6
	95 %	359,2	355,2	352,7	356,4	354,4
	Final	450,1	401,6	411,7	404,4	395,7
2	Point Initial	136,2	144,5	336,7	316,2	312,2
	5 %	347,8	346,3	345,5	339,4	337,9
	10 %	348,7	347	345,7	340,4	339,7
	20 %	348,6	348,1	347	341,9	342,5
	30 %	349,5	348,7	347,4	343,5	343,2
	40 %	349,9	349	348,1	345	344,9
	50 %	390,2	349,4	348,7	346,4	346,5
	60 %	350,6	349,9	349,5	348	348

Labs		Sample WI 18.1	Sample WI 18.2	Sample WI18.3	Sample WI 18.4	Sample WI 18.5
	70 %	351,2	350,3	350	349,8	349,5
	80 %	352	350,8	350,7	351,4	351,2
	90 %	353,3	351,9	351,8	353,7	353,5
	Final	437,4	406,5	416,7	408,3	398,9
2	Initial	140,7	143,4	326,6	311,3	303,8
	5 %	347,9	346,4	344,7	338,3	338
	10 %	348,7	347,6	345,5	339,8	339,2
	20 %	349,1	348	347,1	342,1	342
	30 %	349,4	348,4	347,6	343,4	343,4
	40 %	350	348,9	348,2	344,8	344,8
	50 %	350,3	349,5	348,7	346,4	346,2
	60 %	350,6	349,4	349,4	348	347,9
	70 %	351,2	350,8	350,1	349,8	349,5
	80 %	351,7	350,9	350,8	351,4	351,4
	90 %	353,1	352	351,7	353,4	353,9
	Final	437,4	406,3	419,9	408,6	420,4
3	Initial	328,2	291,1	292,3	287,5	315,1
	5 %	350,7	347,2	347,2	341,2	335,3
	10 %	350,7	348,3	348,3	344,8	341,2
	20 %	351,9	349,5	348,3	346	344,8
	30 %	351,9	350,7	349,5	347,2	347,2
	40 %	353,1	350,7	350,7	348,3	347,2
	50 %	353,1	350,7	350,7	349,5	348,3
	60 %	353,1	350,7	350,7	350,7	348,3
	70 %	354,2	351,9	350,7	353,1	350,7
	80 %	354,2	351,9	351,9	353,1	353,1
	90 %	356,6	353,1	353,1	355,4	355,4
	Final	381,2	357,8	362,5	368,3	364,8
3	Initial	319,9	294,7	295,9	286,3	304,3
	5 %	350,7	349,5	348,3	341,2	341,2
	10 %	351,9	349,5	349,5	343,6	342,4
	20 %	351,9	350,7	349,5	344,8	343,6
	30 %	351,9	350,7	350,7	346	344,8
	40 %	351,9	351,9	350,7	348,3	344,8
	50 %	353,1	351,9	350,7	349,5	346
	60 %	353,1	351,9	350,7	350,7	347,2
	70 %	354,2	353,1	350,7	351,9	348,3
	80 %	354,2	353,1	351,9	353,1	350,7
	90 %	356,6	354,2	353,1	355,4	353,1
	Final	381,2	364,8	361,3	369,5	368,3
4	Initial	272,5	242,3	259,2	257,5	265,4
	5 %	348,8	347,7	346,8	340,4	342,9
	10 %	350	349,2	347,8	343,6	343,5
	20 %	350,4	349,5	348,4	344,9	344,6
	30 %	350,6	349,8	348,7	345	345,2

Labs		Sample WI 18.1	Sample WI 18.2	Sample WI18.3	Sample WI 18.4	Sample WI 18.5
	40 %	351,1	350,1	349,4	345,9	345,9
	50 %	351,4	350,7	349,9	347,5	347,9
	60 %	351,9	351	350,4	348,2	348,1
	70 %	352	351,3	351,1	351	350,6
	80 %	352,6	352,5	351,5	352,2	352
	90 %	355,4	352,8	352,1	354,1	354,8
	Final	410,9	366,5	381	364,8	368,4
4	Initial	257,2	242,7	144,9	262,3	152,3
	5 %	348,2	348,6	347,3	340,7	336,3
	10 %	348,7	349,4	348,5	342,6	343,4
	20 %	350,5	349,8	348,6	344	344
	30 %	350,7	349,9	348	345,6	345,4
	40 %	351,2	350,1	348,1	346,7	346,2
	50 %	351,3	350,6	348,7	348,1	347,4
	60 %	351,8	351	350,4	348,9	348,4
	70 %	352,4	351,3	350,8	350,6	350,2
	80 %	353,1	352,2	351,9	352,2	351,5
	90 %	354	353,5	352,2	354	354,5
	Final	405,9	372,5	372,3	361,9	369,9
5	Initial	329,1	285,2	334,6	311,4	297
	5 %	349,7	348,4	347,4	341,4	340,6
	10 %	349,8	348,8	347,6	342,2	341,6
	20 %	350,1	349,3	348,3	343,7	343
	30 %	350,2	349,7	348,9	345,2	344,8
	40 %	350,6	350	349,1	346,6	345,8
	50 %	351	350,4	349,7	347,7	347,1
	60 %	351,7	350,9	350,4	348,9	348,4
	70 %	352,3	351,5	350,9	350,4	349,8
	80 %	353,1	352	351,8	352	351,8
	90 %	355,6	353,6	353,1	355,1	354,3
	Final	464,3	436	434,5	428,6	433,7
5	Initial	337,9	280,7	328,7	305	308,8
	5 %	349,6	348,3	347,5	341,3	341,1
	10 %	349,8	348,9	347,6	342,3	342,2
	20 %	350,3	349,5	348	343,7	343,6
	30 %	350,6	349,6	348,4	344,9	345
	40 %	350,9	350	349,2	346,2	346,2
	50 %	351,1	350,4	349,7	347,2	347,4
	60 %	351,6	350,5	350,3	348,7	348,7
	70 %	352,2	350,9	350,9	350	350,1
	80 %	353	351,7	351,7	352	352
	90 %	355,1	352,8	353	354,6	354,9
	Final	461,6	434,7	435,7	430,4	433,6

Table C.3 — Precision data from round robin on FAME

Volume recovered	Repeatability °C	Reproducibility °C
IBP	23	91
10 % to 90 %	0,2 to 3,9	2,1 to 8,7
FBP	2	4

Table C.4 — Precision Data from standard ASTM D 1160

Volume recovered	Repeatability °C	Reproducibility °C
IBP	15	49
10 % to 90 %	1,7 to 3,8	4 to 13
FBP	7,1	27

C.5 Conclusion

From comparison of the precision data adapted from the standard, which was determined by a round robin on petrol, and the precision data determined in this round robin, it can be concluded that:

- The test method is applicable to 100 % FAME using the following conditions: pressure of 10 mm Hg and at a uniform rate of 6 ml/min to 8 ml/min.

- Concerning the blend of FAME and diesel fuel the method EN ISO 3405 is applicable with its precision (see preliminary study in Annex D).

As can be seen from Table C.3 and Table C.4 the precision on 10 % to 90 % of ASTM D 1160 on FAME from the round robin is not significantly different from the precision data on petroleum products reported in the Standard ASTM D 1160. Precision on IBP is higher than in ASTM D 1160 however in reason of the poor number of laboratories in the FAME round robin (4 to 5) it cannot be concluded that it is significantly different.

Annex D
(informative)

Preliminary study on applicability of 'distillation of petroleum products at atmospheric pressure' (EN ISO 3405) on blends of FAME in diesel fuel

D.1 Subject

In the framework of the mandate CEN/TC 19/WG 26 “Applicability of petroleum test methods and related precision for application to FAME and FAME containing fuels” a study has been done to determine applicability of EN ISO 3405 "*Petroleum products - Distillation of petroleum products at atmospheric pressure*" on blends of FAME with diesel fuel.

NOTE This study has been supervised by Mr. F. Tort.

D.2 Study

Four samples have been analyzed following the conditions of EN ISO 3405:

— standard EN 590 diesel fuel: GO

— 3 blends made with respectively 5 % (m/m); 10 % (m/m) and 30 % (m/m) of FAME from Rape (RME) in EN 590 diesel fuel (GO).

These samples have been analyzed twice in order to have an estimation of the precision using an automatic instrument. A comparison has been done between the diesel fuel and the blends.

D.3 Results

The results are reported in Table D.1 and given in Figure D.1.

Table D.1 — Distillation results following EN ISO 3405 on diesel fuel and blends.

% re-covered	GO		GO+5%RME		GO+10%RME		GO+30%RME	
	°C	°C	°C	°C	°C	°C	°C	°C
5	200,3	200,2	198,8	199,4	200,1	199,6	209,7	207,9
10	210,2	210,9	210,8	212,2	214,1	212,4	223,9	223,4
15	218,8	217	218,2	219	221,4	220,2	234,5	233,1
20	226,2	226,5	227,5	227,3	228,8	229,4	245,1	244,9
25	232,9	233,6	235,1	235,6	237,7	238,3	255	254,1
30	240,6	240,4	242,2	242,6	245,3	245,5	263,9	264
35	247,1	247,1	249,9	250	252,6	253,1	272,7	272,4
40	253,3	253,5	256,4	256,6	260,4	260,1	281,2	280,8
45	259,8	259,4	263	262,9	267,1	267	289	288,7
50	265,6	265,3	269,2	269,5	273,8	273,8	296,8	296,8
55	271,4	271,6	276,2	276,1	280,5	280,5	303,8	303,9
60	277,4	277,4	282,1	282,5	287	287,2	310,5	310,2
65	284,1	283,9	289,3	289	294,4	293,8	316,4	316
70	290,6	290,5	296	296,1	301,7	301,2	321,3	321,1
75	297,5	298	303,2	303,7	308,6	308,4	325,7	325,5
80	305,4	305,5	311,4	311,6	316,2	315,6	329,3	329,3
85	314,7	314,8	320,2	320,1	323,6	323,1	332,9	332,8
90	326,3	326,8	329,7	329,5	331,8	331,3	336,7	336,6
95	342,4	342,8	342,8	342,3	341,9	342,1	343,3	343,2

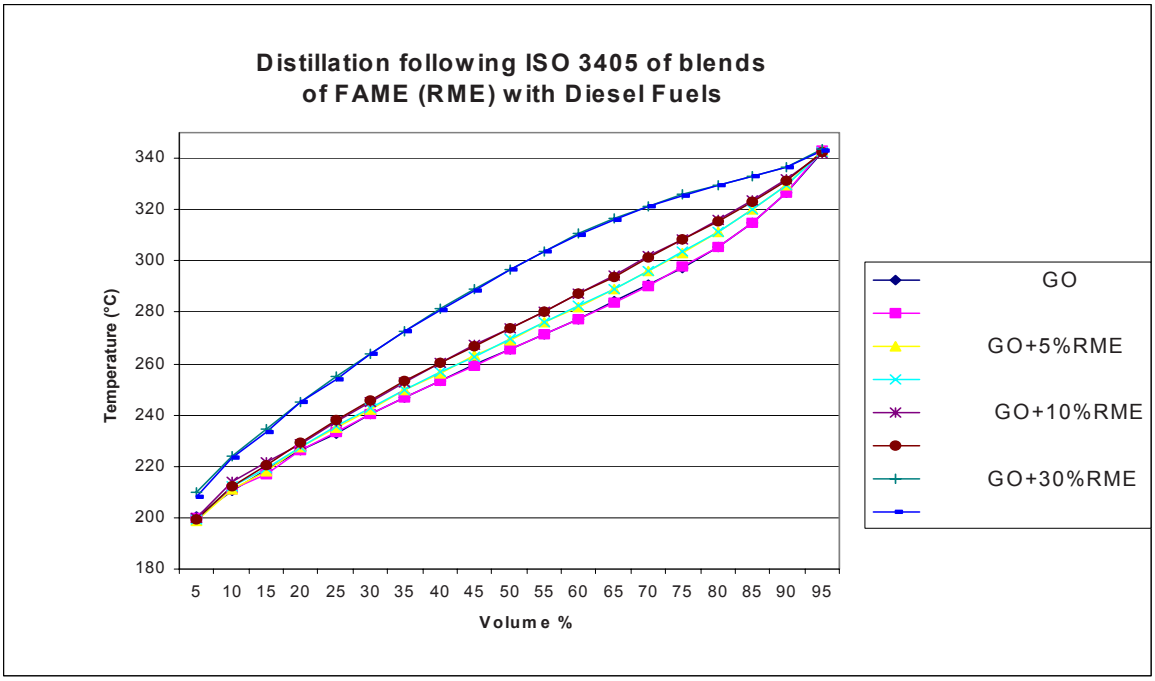

Figure D.1 — Distillation results following EN ISO 3405 on diesel fuel and blends

These results show that there is a decrease of the volatility of the blends when the amount of FAME increases. The superposition of the curves obtained with the repetition on the same sample is also perfect, showing a very good repeatability both on diesel fuel and on blends of FAME. Data for the comparison of the precision are reported in Table D.2 and Figure D.2.

D.4 Conclusion

The results obtained in this study show that EN ISO 3405 is applicable without any modification to blends of FAME in diesel fuel. The study has been limited to blends containing up to 30 % (*m/m*) of FAME. For higher concentration another study would be recommended.

Table D.2 — Estimation of repeatability on diesel fuel and blends

% recovered	GO	GO+5%	GO+10%	GO+30%
	°C	°C	°C	°C
5	0,1	-0,6	0,5	1,8
10	-0,7	-1,4	1,7	0,5
15	1,8	-0,8	1,2	1,4
20	-0,3	0,2	-0,6	0,2
25	-0,7	-0,5	-0,6	0,9
30	0,2	-0,4	-0,2	-0,1
35	0	-0,1	-0,5	0,3
40	-0,2	-0,2	0,3	0,4
45	0,4	0,1	0,1	0,3
50	0,3	-0,3	0	0
55	-0,2	0,1	0	-0,1
60	0	-0,4	-0,2	0,3
65	0,2	0,3	0,6	0,4
70	0,1	-0,1	0,5	0,2
75	-0,5	-0,5	0,2	0,2
80	-0,1	-0,2	0,6	0
85	-0,1	0,1	0,5	0,1
90	-0,5	0,2	0,5	0,1
95	-0,4	0,5	-0,2	0,1

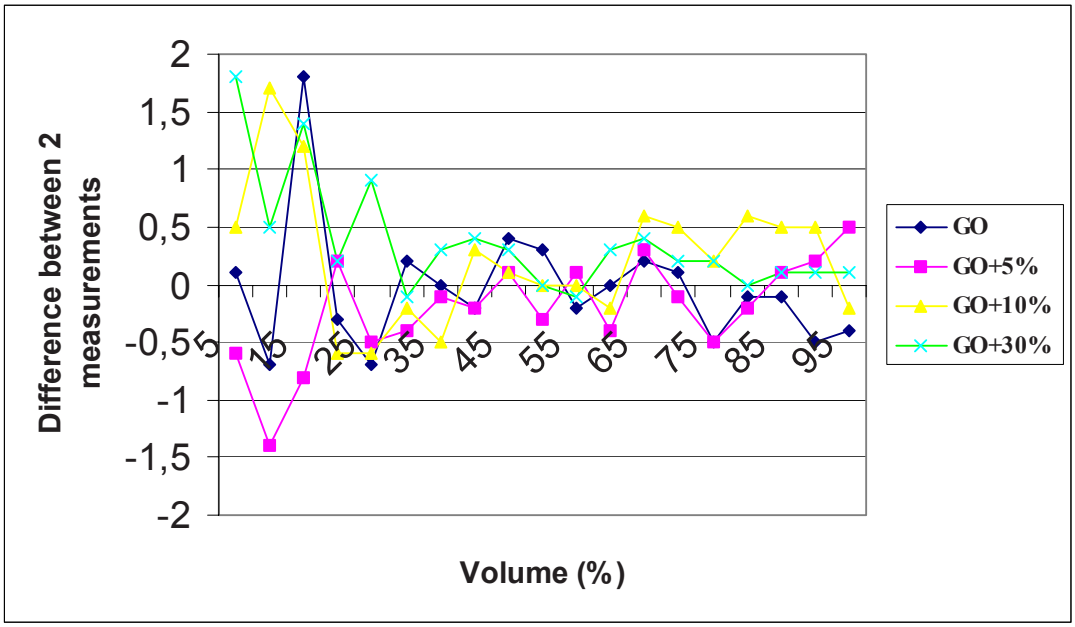

Figure D.2 — Estimation of repeatability on diesel fuel and blends

Annex E
(informative)

Report of the results of the round robin on 'determination of carbon residue via the micro method' (EN ISO 10370)

E.1 Scope

In this round robin the applicability of test method EN ISO 10370 "*Petroleum Products – Determination of carbon residue – Micro method*" towards FAME as 100 % diesel fuel is investigated. The method is also applicable to blends of FAME and diesel fuel.

NOTE This study has been supervised by Mr. P. Tittarelli.

E.2 Assessed standard documents

EN ISO 3405:2000, *Petroleum products – Determination of distillation characteristics (ISO 3405:2000)*

EN ISO 4259:1995, *Petroleum products – Determination and application of precision data in relation to methods of test (ISO 4259:1992/Cor.1:1993)*

EN ISO 10370:1995, *Petroleum products – Determination of carbon residue – Micro method (ISO 10370:1993)*

ASTM D 1160:95, *Standard Test method for the Distillation of Petroleum Products at Reduced Pressure*

E.3 Preliminary study

Carbon residue measurements on 3 FAME samples, 10 % residue after vacuum distillation at 10 mm Hg (ASTM D 1160).

The FAME samples were distilled under vacuum by two laboratories. Vacuum distillation was performed on each FAME sample by both laboratories. Micro Carbon Residue measurements on each distillation residue were performed by both laboratories. MCR was determined using large vials. Table E.1 shows carbon residue results and corrected temperatures at 5 % and 80 % distillation.

The results indicate an influence on the reproducibility of carbon residue measurements mainly due to the vacuum distillation step.

E.4 Details of the round robin

EN ISO 10370 specifies a method for the determination of the amount of carbon residue in the range 0,10 % (*m/m*) to 30,0 % (*m/m*). It also specifies that the method can be applied to petroleum products which may yield a carbon residue below 0,10 % (*m/m*). On such materials, a 10 % (*V/V*) distillation residue is prepared by the procedure described in EN ISO 3405 before analysis.

Table E.1 — Carbon residue results on 10 % (*m/m*) residues after ASTM D 1160 vacuum distillation

FAME	Sample	Distillation	MCR	T_{corr} 5 %,°C	T_{corr} 80 %,°C	Carbon residue, % (*m/m*)	Carbon residue, % (*m/m*)
1 – EMC	I	A	A	348	353	0,23	0,21
	II	A	A	348	353	0,25	0,22
	I	A	B			0,29	0,29
	I	B	B	352	354	0,18	0,17
	II	B	B	350	354	0,17	0,17
	I	B	A			0,16	
2 – EMC	I	A	A	348	352	0,08	0,07
	II	A	A	348	352	0,08	0,06
	I	A	B			0,09	0,09
	I	B	B	351	355	0,18	0,19
	II	B	B	350	354	0,18	0,19
	I	B	A			0,18	
3 – EMT	I	A	A	347	351	0,07	0,04
	II	A	A	347	351	0,08	0,05
	I	A	B			0,07	0,07
	I	B	B	350	354	0,15	0,15
	II	B	B	351	354	0,13	0,13
	I	B	A			0,13	

NOTE The laboratories are indicated as A and B, the residues after the first and second distillation are indicated as I and II, The temperatures corresponding to 5 % and 80 % distillation have been corrected and reported to normal pressure, Ten mm Hg pressure was used during vacuum distillation,

Preliminary measurements of FAME samples showed carbon residue results below 0,10 %, using the large vials indicated in Note 4 of EN ISO 10370:1995. The possible use of very large vials was not considered as these vials are not listed in the current version of EN ISO 10370.

Therefore, is was necessary to perform a preliminary distillation step to obtain FAME samples with carbon residue ≥ 0,10 % (*m/m*).

The application of EN ISO 3405 (distillation at atmospheric pressure) was not suitable for FAME samples, as the boiling range of these products is very limited. Therefore, it was decided to perform under vacuum distillation according to ASTM D 1160 (see Annex C). Preliminary measurements on vacuum residues of FAME products showed that the carbon residue was around 0,1 % (*m/m*) or even lower. It was also shown that the main source of results dispersion was due to the distillation step (E.3).

Hence, it was decided to perform under vacuum distillation in a single laboratory and distribute 10 % residue samples for micro carbon residue measurements.

A total of ten laboratories from five countries participated in the round robin.

Five FAME samples were supplied by Elf CRES Solaize. The samples were numbered from 1 to 5:

— Sample 1 EMC

— Sample 2 EMC

— Sample 3 EMT

— Sample 4 75 % EMC – 25 % EMT

— Sample 5 25 % EMC – 75 % EMT

Ten percentage residues were prepared for each FAME sample, after vacuum distillation at reduced pressure using ASTM D1160. Ten mm Hg pressure was maintained during the distillation under vacuum. An automatic equipment was used to prepare FAME residues. Ten ml aliquots of each residue were delivered to the participants in sealed ampoules.

During the round robin, the participants were required to use the large vials, with a capacity of 4 ml, as indicated in NOTE 4 of EN ISO 10370:1995.

E.5 Results

Table E.2 shows the results obtained in the round robin.

Table E.2 — Results of the round robin

Laboratory	Sample 1		Sample 2		Sample 3		Sample 4		Sample 5	
	% (*m/m*)	% (*m/m*)	% (*m/m*)	% (*m/m*)	% (*m/m*)	% (*m/m*)	% (*m/m*)	% (*m/m*)	% (*m/m*)	% (*m/m*)
1	0,24	0,25	0,09	0,09	0,05	0,05	0,07	0,07	0,06	0,08
2	0,26	0,27	0,14	0,15	0,07	0,07	0,04	0,03	0,09	0,08
3	0,26	0,26	0,12	0,13	0,07	0,06	0,06	0,05	0,06	0,08
4	0,26	0,26	0,14	0,15	0,07	0,07	0,09	0,09	0,08	0,08
5	0,300	0,305	0,130	0,142	0,062	0,079	0,082	0,106	0,067	0,080
6	0,25	0,23	0,11	0,12	0,05	0,05	0,08	0,08	0,07	0,05
7	0,25	0,25	0,10	0,10	0,05	0,06	0,07	0,07	0,06	0,06
8	0,23	0,24	0,09	0,09	0,04	0,04	0,08	0,07	0,05	0,05
9	0,31	0,32	0,12	0,12	0,04	0,04	0,08	0,07	0,11	0,13
10	0,30	0,30	0,12	0,13	0,06	0,07	0,09	0,09	0,08	0,08
Mean	0,263		0,117		0,056		0,071		0,075	

No deviations from the test method were reported by the participants.

The statistical evaluation was performed using EN ISO 4259.

No outliers or stragglers were detected for samples 1 to 4.

Laboratory 9 was detected as a straggler in the results of samples 5.

The precision of EN ISO 10370 is the following:

Repeatability $r = x^{2/3} \cdot 0{,}077\ 0$

Reproducibility $R = x^{2/3} \cdot 0{,}245\ 1$

Table E.3 — Precision of the round robin

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
r EN ISO 10370	0,032	0,018	0,011	0,013	0,014
R EN ISO 10370	0,101	0,059	0,036	0,042	0,044
r round robin	0,019	0,015	0,012	0,013	0,027
R round robin	0,077	0,059	0,034	0,049	0,060

The comparison of precision values obtained in the round robin with those stated in EN ISO 10370 shows that the repeatability on FAME residues differs significantly from EN ISO 10370 only for 1 sample, i.e. sample 5. However, lab. 9 is detected as a straggler in sample 5, and the precision can be affected by the results of this laboratory.

The reproducibility on FAME residues does not differ significantly from EN ISO 10370.

E.6 Conclusions

From the comparison of the precision data obtained in this round robin on FAME residues with the precision data of EN ISO 10370 it can be concluded that:

- The test method is applicable to 100% FAME.

- The test method is applicable to a blend of FAME and diesel fuel.

Annex F
(informative)

Report of the result of the round robin series on 'corrosiveness to copper via the copper strip test' (EN ISO 2160)

F.1 Scope

In this round robin the applicability of test method EN ISO 2160 "Petroleum products – Corrosiveness to copper – Copper strip test" towards FAME as 100 % diesel fuel is investigated. The repeatability and reproducibility are determined if the test method was applicable. The method is *also* applicable to blends of FAME and diesel fuel.

NOTE This study has been supervised by Mr. F. Tort.

F.2 Assessed standard documents

prEN ISO/FDIS 2160:1998 [5)], *Petroleum products – Corrosiveness to copper – Copper strip test (ISO 2160:1998)*

EN ISO 3170:1998, *PetroleumlLiquids – Manual sampling (ISO 3170:1988)*

EN ISO 4259:1995, *Petroleum products – Determination and application of precision data in relation to methods of test (ISO 4259:1992/Cor.1:1993)*

F.3 Details of the round robin

A total of 10 laboratories in 6 countries took part in the round robin. They are labelled 1 to 10.

5 FAME samples were supplied by Elf. Their composition is given in Table F.1.

In addition to the requirements of EN ISO 2160 the following measurement instructions were applied:

— The test was carried out at (50 ± 1) °C during 3 h ± 5 min.

— For each sample, the test was carried out in duplicate preferably on the same day and the results reported in the table attached.

5 Succeeded by EN ISO 2160:1998, *Petroleum products – Corrosiveness to copper – Copper strip test (ISO 2160:1998)*

Table F.1 — Composition of samples for EN ISO 2160

Sample	Sample WI 16-1	Sample WI 16-2	Sample WI 16-3	Sample WI 16-4	Sample WI 16-5
Type of FAME	100 % FAME from Rape Supplier Novaol (RME1)	100 % FAME from Rape Supplier Cognis (RME2)	100 % FAME from Sun Flower Supplier Cognis (SME)	SME/RME2 25/75 (% (*m/m*))	SME/RME2 75/25 (% (*m/m*))

Handling of the samples was done according EN ISO 3170.

The statistical treatment of this round robin was performed according EN ISO 4259.

F.4 Results

The results of the study are given in Table F.2.

Table F.2 — Results of test method EN ISO 2160 for different laboratories and samples

Laboratory	Sample					Date of test
	WI16-1	WI16-2	WI16-3	WI16-4	WI16-5	
1	1a	1a	1a	1a	1a	16-18/06/1998
	1a	1a	1a	1a	1a	
2	1a	1a	1a	1a	1a	07/08/1998
	1a	1a	1a	1a	1a	
3	1a	1a	1a	1a	1a	8-16/06/1998
	1a	1a	1a	1a	1a	
4	1a	1a	1a	1a	1a	8-10/06/1998
	1a	1a	1a	1a	1a	
5	1a	1a	1a	1a	1a	10-11/06/1998
	1a	1a	1a	1a	1a	
6	1a	1a	1a	1a	1a	11-15/06/1998
	1a	1a	1a	1a	1a	
7	1a	1a	1a	1a	1a	28/05-2/06/1998
	1a	1a	1a	1a	1a	
8	1a	1a	1a	1a	1a	3-5/06/1998
	1a	1a	1a	1a	1a	
9	1a	1a	1a	1a	1a	09/06/1998
	1a	1a	1a	1a	1a	
10	1a	1b	1b	1b	1b	24/06/1998
	1a	1a	1b	1b	1b	

On the 5 samples most of the laboratories found a classification of 1a except one laboratory which found 1b on 3 samples. These results show that the test is applicable on pure FAME. Because of the qualitative nature

of this test no precision evaluation is necessary. Tests performed on blends of FAME (up to 30 % (*m/m*) of FAME from Rape) in diesel fuel have shown that the method is also applicable and the values obtained were also of 1.

F.5 Conclusion

From the results obtained in this study, it can be concluded that:

— The test method is applicable to 100 % FAME.

— The test method is applicable to a blend of FAME and diesel fuel.

As for petroleum product no precision data is necessary to be evaluated.

Annex G
(informative)

Report of the result of the round robin series on 'determination of fatty acid methyl esters (FAME) in middle distillates via infrared spectroscopy' (prEN 14078)

G.1 Scope

In this round robin the applicability of draft test method prEN 14078 "Determination of fatty acid methyl esters (FAME) in middle distillates – Infrared spectroscopy method" to blends of FAME and diesel fuel is investigated. The repeatability and reproducibility are determined.

NOTE This study has been supervised by Mrs. M.F. Benassy.

G.2 Assessed standard documents

prEN 14078:2001, *Liquid petroleum products – Determination of fatty acid methyl esters (FAME) in middle distillates – Infrared spectroscopy method*

EN ISO 3170:1998, *Petroleum liquids – Manual sampling (ISO 3170 1988)*

EN ISO 3675:1998, *Crude petroleum and liquid petroleum products – Laboratory determination of density - Hydrometer method (ISO 3765:1998)*

EN ISO 4259:1995, *Petroleum products – Determination and application of precision data in relation to methods of test (ISO 4259:1992/Cor.1:1993)*

EN ISO 12185:1996, *Crude petroleum and petroleum products – Determination of density – Oscillation U-tube method (ISO 12185:1996)*

G.3 Details of the round robin

A total of 7 laboratories in 4 countries took part in the round robin.

They are labelled 1 to 7.

Six FAME in diesel fuel samples were supplied by Elf. Their composition is given in Table G.1.

In addition to the requirements of the following measurement instructions have been applied:

— For sample WI21-1 to WI21-4 a dilution factor of 10 shall be used and for the sample WI21-5 to WI21-6 a dilution factor of 20 shall be used.

— If the cell thickness used for the measure is different from the cell thickness used for the calibration then it is necessary to correct the absorbance at 1 745 cm^{-1} of the sample.

— For each sample, the test shall be carried out in duplicate and the results reported in the table attached.

— Report the results rounded at 0,01g/l.

Table G.1 — Composition of samples used in the round robin test

	% (*m/m*) of FAME (4 origins) in diesel fuel			
	Sunflower	**Tallow**	**Rape**	**Soya**
WI 21-1	2 %			
WI 21-2	10 %			
WI 21-3		8 %		
WI 21-4			5 %	
WI 21-5			20 %	
WI 21-6				15 %
WI stand			100 %	

Handling of the samples was done according EN ISO 3170.

The statistical treatment of this round robin was performed according EN ISO 4259.

G.4 Results

The results of the round robin are given in Table G.2.

The statistical analysis have found result of lab N° 3 suspect with results much higher on sample 5 and higher on sample 2 and 6 than others laboratories. Then for the precision evaluation the results of lab N° 3 on samples 2, 5 and 6 have been withdrawn.

Table G.2 — Results of test method prEN 14078 for different laboratories and samples

Laboratories	Sample WI 21-1	Sample WI 21-2	Sample WI 21-3	Sample WI 21-4	Sample WI 21-5	Sample WI 21-6
1	17,07	89,29	70,04	40,8	170,34	131,05
	17,03	87,46	71,3	41,42	170,48	131,15
2	18,46	85,15	69,03	43,23	167,8	128,8
	18,84	85,28	70,72	43,87	167,9	130
3	19,12	94,3	76,55	48,04	187,79	145,38
	17,8	95,62	74,21	48,54	187,99	144,97
4	20,98	88,66	75,19	47,72	175,16	139,07
	20,6	90	74,38	48,26	176,24	137,46
5	18,82	89,41	72,41	46,28	173,46	134,59
	17,65	88,55	73,5	45,75	174,72	132,94
6	17,89	85,98	70,29	45,25	166,74	129,52
	18,09	88	70,29	46,25	166,94	128,52
7	22,2	89,92	75,13	45,06	172,08	132,32
	22,26	89,8	75,2	44,97	172	132,95
Mean 1	19,2	89,0	72,7	45,2	173,3	134,4
Mean 2	18,9	89,2	72,8	45,6	173,8	134,0
Mean value	**20,6**	**89,5**	**73,9**	**45,2**	**172,8**	**133,4**

The results of precision evaluation are reported in Table G.3 and G.4.

Table G.3 — Precision data from round robin on FAME in diesel fuel blends

Sample	Mean *(g/l)*	Repeatability *(g/l)*	Reproducibility *(g/l)*
WI 21-1	19,1	1,7	6,3
WI 21-2	88,1	3,3	6,7
WI 21-3	72,7	3,2	8,7
WI 21-4	45,4	1,5	8,7
WI 21-5	171,2	1,8	12,7
WI 21-6	132,4	3	12,6

Table G.4 —Precision rata from round robin study on prEN 14078 expressed

Quantity *(g/l)*	Repeatability *(g/l)*	Reproducibility *(g/l)*
≤ 100	2,5	7,7
> 100	2,5	12,7

Precision data can also be expressed in % (*V/V*) using the formula:

$$FAME\ \%(V/V) = \frac{Q\ (g/l) \cdot 100}{d\ (g/l)} \qquad \text{(G.1)}$$

The precision on density (EN ISO 12185 or EN ISO 3675) is negligible on the precision on FAME quantity expressed in % *(V/V)* and is reported in Table G.5.

Table G.5 — Precision data from round robin study on prEN 14078 expressed in % (*V/V*)

Quantity % (*V/V*)	**Repeatability** % (*V/V*)	**Reproducibility** % (*V/V*)
≤ 11	0,28	0,87
> 11	0,28	1,44

G.5 Conclusion

According to the results obtained here this method allows to determine the quantity of FAME in diesel fuel for concentration equal to or lower than 200 g/l. This method is applicable with a good precision whatever the origin of FAME (vegetal or animal origin). This method has been adapted from the French standard NF M 07-084 limited to concentration lower than 100 g/l and at that concentration the precision data are similar.

Annex H
(informative)

Influence of FAME origin on the measure of FAME content in mineral oil using prEN 14078 ('Determination of fatty acid methyl esters (FAME) in middle distillates via infrared spectroscopy method')

H.1 Subject

In the framework of the mandate CEN/TC19/WG26 “Applicability of petroleum test methods and related precision for application to FAME and FAME containing fuels” a study has been done by F. Tort (TotalFinaElf) to determine the influence of the origin of the FAME (type of vegetal, animal) several blends of FAME in diesel fuels have been studied.

H.2 Study

Five types of FAME have been studied to influence of the origin of FAME on the results. The concentrations studied are: 5 %; 10 % and 15 % of FAME in EN 590 diesel fuel. The calibration curve has been built with rape oil.

1) European FAME, Rape (RME)

2) European FAME, Sunflower (SME)

3) American FAME, Soya

4) American FAME, Tallow

5) American FAME, Mixed

H.3 Results

Characteristics of the European FAME are reported in Table H.1. These data show that they are very similar and mainly composed of methyl esters from C18 fatty acid. No data were available for the FAME of US origin. Calibration data for the measurement are given in Table H.2. Results of the study are given in Table H.3 for European FAME and in Table H.4 for the American versions and represented in Figure H.1.

Table H.1 — Characteristics of European FAME

	RME (Rape) 99040182	SME (Sunflower) 99040183
Ester content	99,40%	99,70%
Water content	290 mg/kg	193 mg/kg
MeOH content	<100mg/kg	1089 mg/kg
Iodine indices	116	133
Acid Number	0,15	0,11
Free glycerol	0,02%	0,04%
Mono glycerides	0,16%	0,03%
Diglycerides	0,02%	0,01%
Triglycerides	0,01%	0,06%

Fatty Acid	RME (Rape) 99040182	SME (Sunflower) 99040183
C14:0	0,1	0,1
C16:0	**4,4**	**6,5**
C16:1	0,3	0,1
C17:0	0,1	0,1
C17:1	0,2	<0,1
C18:0	**1,4**	**4,2**
C18:1	**59,6**	**22,6**
C18:2	**19,7**	**64,6**
C18:3	**10,1**	**0,4**
C20:0	0,5	0,3
C20:1	1,4	0,2
C22:0	0,3	0,5
C22:1	1	0,1
C24:0	0,1	0,1
C24:1	0,2	0
NI	0,6	0,2

Table H.2 — Calibration data

Height 1745 cm-1	Rapeseed FAME *kg/m3*	Cell height / thickness	slope 4,770 3327
0,064	0,517	0,12	intersection
0,127	1,017	0,25	-0,141 008 91
0,236	1,993	0,46	
0,445	3,993	0,86	
0,678	6,026	1,31	
1,094	10,004	2,12	

FAME Density	etalon cell thickness mm	meas.cell thickness mm	Dilution Factor
0,88	0,517	0,517	10

Table H.3 — Measurement of FAME content in diesel fuel: European FAME

Sample	Height 1 745 cm-1	FAME *kg/m3*	SME - RME
RME 5% PDS-1	0,461	41,13	
SME 5% PDS-1	0,46	41,03	-0,1
RME 5% PDS-2	0,469	41,86	
SME 5% PDS-2	0,468	41,77	-0,09
RME 10%	0,911	82,65	
SME 10%	0,913	82,83	-0,02
RME 15%*	0,684	123,4	
SME 15%*	0,692	124,88	1,48

Table H.4 — Measurement of FAME content in diesel fuel: American FAME

Sample	height 1 745 cm^{-1}	FAME *kg/m3*	mean	FAME mean
SOYA 5 % (*m/m*)	0,471	42,13		0,98
TALLOW 5 % (*m/m*)	0,454	40,56		-0,59
MIXED 5 % (*m/m*)	0,456	40,75	41,15	-0,4
SOYA 10 % (*m/m*)	0,896	81,42		-0,4
TALLOW 10 % (*m/m*)	0,902	81,98		0,16
MIXED 10 % (*m/m*)	0,903	82,07	81,82	0,25
SOYA* 15 % (*m/m*)	0,699	126,42		3,32
TALLOW* 15 % (*m/m*)	0,676	122,17		-0,93
MIXED* 15 % (*m/m*)	0,668	120,69	123,1	-2,41

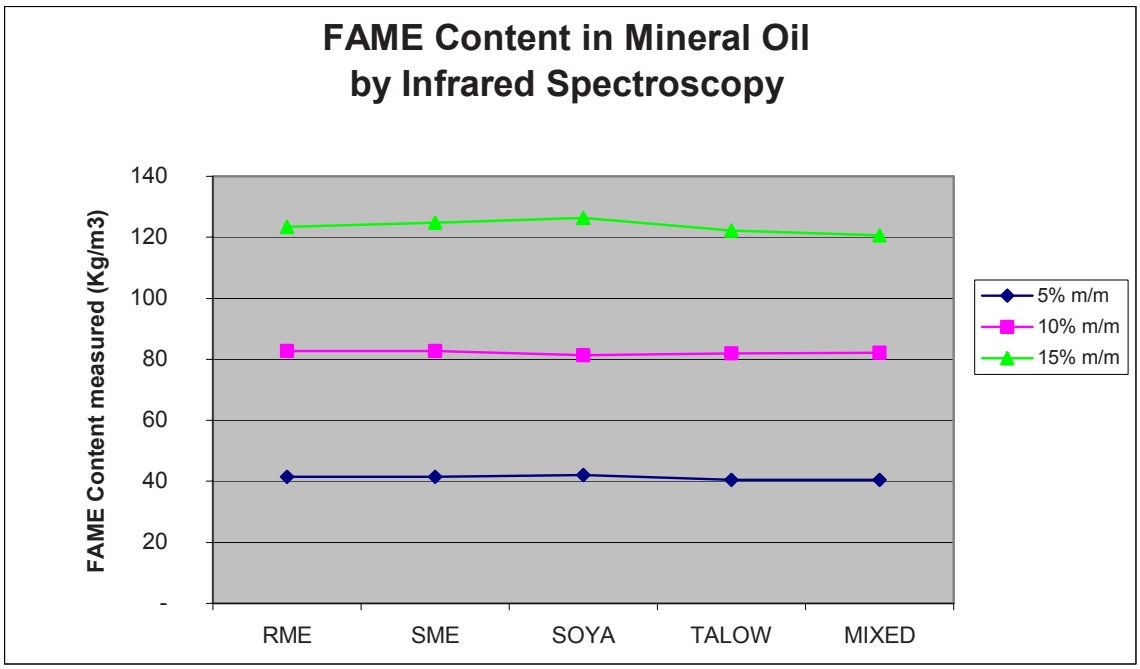

Figure H.1 — Comparison of results according to the origin

H.4 Conclusions

The results show that whatever the origin of FAME (type of vegetal, animal) the method allows to measure the concentration. For concentration higher than 10 % it is necessary to use a higher dilution. This higher dilution lowers the precision. Precision of the method is directly linked to the dilution factor used. A round robin is then recommended to determine the precision of the method.

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