SLOVENSKI STANDARD
SIST-TP CEN/TR 15160:2005

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Naftni in sorodni proizvodi ï Uporabnost preskusnih metod dizelskega goriva za metil estre maġļ obnih kislin (FAME) ï Informacije in rezultati èround robinç preskusov

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

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

Produits pétroliers et produits connexes - Applicabilité des méthodes d'essai des carburants diesel (gazoles) aux esters méthyliques d'acides gras (EMAG) - Information et résultats relatifs aux essais circulaires

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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

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

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	$0{,}022X^{0{,}9}$ °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