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Automotive fuels — High FAME diesel fuel blends (B11 — B30) — Background to the parameters required and their respective limits and determination

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National foreword

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Automotive fuels - High FAME diesel fuel blends (B11 - B30) - Background to the parameters required and their respective limits and determination

Carburants pour automobiles - Mélanges de carburants diesel ayant une teneur en EMAG élevée (B11 - B30) - Contexte de l'élaboration des caractéristiques requises, de leurs déterminations et de leurs limites respectives

Kraftstoffe für Kraftfahrzeuge - Dieselkraftstoffmischungen mit hohem FAME-Anteil (B10 - B30) - Hintergrund zu den geforderten Parametern und deren jeweiligen Grenzwerten und Bestimmungen

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Ref. No. CEN/TR 16557:2013: E

Contents

Page

Foreword

This document (CEN/TR 16557:2013) 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.

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1 Scope

This Technical Report provides background information to the deliberations within CEN that led to establish a specification for blending from more than 10 % (*V/V*) up to 30 % (*V/V*) of fatty acid methyl ester (FAME) in diesel fuel to be used in captive fleet application for designated vehicles. It gives guidance and explanations to the producers, blenders, marketers and users of high FAME diesel blends (B11 to B30).

The sole designation "Bxx" refers to a FAME-diesel blend where "*xx"* is the specific FAME content in volume percentage. The connotation "Byy fuel" is used in this document for a fuel with a defined range of FAME allowed and having "*yy"* volume percentage of FAME content as the maximum of that range.

NOTE For the purposes of this document, the term "% (*m/m*)" and "% (*V/V*)" are used to represent the mass fraction, *μ*, and the volume fraction, *φ,* respectively.

2 Normative references

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

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

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

EN 12662:2008, *Liquid petroleum products - Determination of contamination in middle distillates*

EN 14078:2009, *Liquid petroleum products - Determination of fatty acid methyl ester (FAME) content in middle distillates - Infrared spectrometry method*

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

EN 15195:2007, *Liquid petroleum products - Determination of ignition delay and derived cetane number (DCN) of middle distillate fuels by combustion in a constant volume chamber*

EN 15751:2009, *Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of oxidation stability by accelerated oxidation method*

EN 16091:2011, *Liquid petroleum products - Middle distillates and fatty acid methyl ester (FAME) fuels and blends - Determination of oxidation stability by rapid small scale oxidation method*

EN 16329:2013, *Diesel and domestic heating fuels - Determination of cold filter plugging point - Linear cooling bath method*

EN ISO 2719:2002, *Determination of flash point - Pensky-Martens closed cup method (ISO 2719:2002)*

EN ISO 3104:1996, *Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity (ISO 3104:1994)*

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

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

EN ISO 3405:2011, *Petroleum products - Determination of distillation characteristics at atmospheric pressure (ISO 3405:2011)*

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

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

EN ISO 6245:2002, *Petroleum products - Determination of ash (ISO 6245:2001)*

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

EN ISO 12937:2000, *Petroleum products - Determination of water - Coulometric Karl Fischer titration method (ISO 12937:2000)*

EN ISO 20846:2011, *Petroleum products - Determination of sulfur content of automotive fuels - Ultraviolet fluorescence method (ISO 20846:2011)*

EN ISO 20884:2011, *Petroleum products - Determination of sulfur content of automotive fuels - Wavelength-dispersive X-ray fluorescence spectrometry (ISO 20884:2011)*

3 Background of the High FAME diesel blends (B11 - B30) taskforce work

At the 2009 plenary meeting, CEN/TC 19 decided to register the following preliminary work item as described in its active programme of work: *Automotive fuels — High FAME diesel blends (B10 - B30) — Requirements and test methods*, under responsibility of WG 24, with the scope to finalise its feasibility study in time to allow WG 24 to report to TC19 in order to make a final decision on activation at 2011 plenary meeting at the latest on the basis of existing related national standards and to consider and solve the comments as presented by SUTN during the PWI ballot.

An automatic link has been established between the diesel fuel and FAME standards (EN 590 and EN 14214 respectively) and the High FAME diesel blends standard so that modifications to one will be coherent with the other.

Captive fleets are in general considered as a group of vehicles that possess specific supply logistics, their own dedicated facilities for storage and distribution and adequate maintenance of the vehicles. As the definitions are widespread around Europe and the group does not wish to contradict specific legal situations in some countries, the technical definition of captive fleets is left to the national standardization bodies and will be required.

The provisional scope would thus read: “This European Standard specifies requirements and test methods for marketed and delivered high FAME (B30) fuel for use in diesel engine vehicles designed or subsequently adapted to run on high FAME (B30) fuel. High FAME (B30) fuel is a mixture of more than 10 % (*V/V*) up to 30 % (*V/V*) fatty acid methyl esters (commonly known as FAME) complying to EN 14214 and automotive diesel fuel complying to EN 590. For maintenance and control reasons it is to be used in captive fleets that are intended to have an appropriate fuel management.”

The group agreed that the national captive fleet description requirement needed to be required in a point in the text.

4 Record of the work to date

4.1 Context

The European Commission is following a policy of promoting renewable energy use in Europe, and to this end is encouraging the extension of automotive diesel fuel with a proportion of renewably-sourced fatty acid methyl ester (referred to in this document as FAME).

In order to facilitate a transparent and stable market in FAME, it is necessary to establish a higher FAME diesel blend (B11 - B30) standard for Europe that will ensure a uniform high quality fuel for problem-free blends from more than 10 %(*V/V*) up to 30 % (*V/V*) of FAME in Diesel fuel to be used in captive fleet application for designated vehicles.

To this end, the Comité Européen de Normalisation (CEN) Technical Committee 19, responsible for Automotive Fuels Standards, has accepted a preliminary work item on its programme to be developed in Working Group 24, responsible for Automotive Diesel fuel management. The intention of CEN/TC 19 was to finalise a feasibility study in time to allow WG 24 to report to the Technical Committee in order to make a final decision on activation at 2011 plenary meeting at the latest to define a CEN standard based on the publication of EN 14214 and EN 590, also taking into account the developments in France[1] and Czech Republic[2]. Discussion in WG 24 had given indications that there were still test method questions to solve and thus national initiatives were not halted.

CEN/TC 19, having considered the proposal for a preliminary new work item, as documented in N 1441 and supported by WG 24 decided to register “CEN/TR, Automotive fuels – High FAME diesel blends (B10 - B30) - Requirements and test methods” in its active programme of work, under responsibility of WG 24. The scope of this work to finalise the feasibility study in time to allow WG 24 to report to CEN/TC 19 in order to make a final decision on activation at 2011 plenary meeting at the latest on the basis of existing related national standards and to consider and solve the comments as presented by SUTN during the ballot. The Task Force B30 was created.

The idea of CEN/TC 19 was that the standard should specify requirements and test methods for marketed and delivered high FAME (B30) fuel for use in diesel engine vehicles designed or subsequently adapted to run on high FAME (B30) fuel. High FAME (B30) fuel is a mixture of nominally 30 % (*V/V*) fatty acid methyl esters (commonly known as FAME) complying to EN 14214 and automotive diesel fuel complying to EN 590. HVO and XTL products can be included in this fuel if the final automotive diesel fuel part complies to EN 590.

B30 fuel is a mixture of nominally 30 % FAME and 70 % diesel fuel, but also including the possibility of having different seasonal grades. For maintenance and control reasons the high FAME (B30) fuel is to be used in captive fleets that have appropriate fuel management practices, for example high fuel turn-over.

In 2011, the WG 24 chose to merge the TF FAME and the B30 TF. The new TF had been given the preparation work for EN 14214 revision and the task to continue the development on B30 fuel.

This Technical Report is based on the final report carried out by this B30 TF towards establishing the feasibility to reach a European standard for High FAME diesel blends (B11 - B30). On the basis of the report, CEN/TC 19 decided in November 2012 to activate the work item for a CEN specification for the fuel itself.

4.2 The Task Force

CEN/TC 19/WG 24 decided to convene a task force and to begin studying the feasibility to draft a high FAME diesel blends (B10 - B30) standard. A call was made to the industries concerned by the mandate for experts to participate in the B30 TF. After a first report to WG 24, it was felt that many discussions on

FAME product had overlapping technical content. It was decided in 2011 to merge the TF B30 with the existing TF FAME. The experts that have contributed to the work over the years are listed in Table 1.

Table 1 — Membership of the taskforce

Surname	Christian name	Company or Organisation	Member State
1. Alfredsson	Sara	Scania	Sweden
2. Alvarez	Beatriz	APPA Biofuels	Spain
3. Andreasen	Kjær	Daka Biodiesel Production	Denmark
4. Baldini	Luca	ENI	Italy
5. Balfour	Graham	Infineum	CEFIC-ATC
6. Baumgarten	Jens	Esso Deutschland	Germany
7. Borkes	Ruben	BP	Netherlands
8. Burrows	Aubrey	Downstream Fuel	United Kingdom
9. Buttle	Dermot	EBB	EBB
10. Cajete Garcia	Reyes	Acciona Biocombustibles	Spain
11. Catalano	Salvatore	SGS Italia	Italy
12. Roudot	Florence	AFNOR	France
13. Chrysafi	Sofia	Ford Motors Europe	United Kingdom
14. Costenoble	Ortwin	TF secretary	Netherlands
15. Crépeau	Gérald	PSA Peugeot Citroen	France
16. Diaz Garcia	Carlos	Repsol	Spain
17. Doermer	Wolfgang	BP	Germany
18. Elliott	Nigel	WG 24 convenor	United Kingdom
19. Engelen	Benoit	Total	Belgium
20. Eriksson	Henrik	Scania	Sweden
21. Faedo	Davide	SSC	Italy
22. Falciola	Michele	Assocostieri	Italy
23. Feuerhelm	Thomas	DIN-FAM	Germany
24. Fiolet	Gerard	Shell Nederland	Netherlands
25. Fischer	Juergen	ADM Research	Germany
26. Gomez Guenca	Felix	CLH	Spain
27. Gomez Martinech	Jose	CEPSA	Spain
28. Guirao Galinos	Beatriz	CLH	Spain
29. Guizouarn	Kristell	Diester Industries	France
30. Guizouarn	Gwenael	Diester Industries	France
31. Jackson	Alister	ExxonMobil	United Kingdom
32. Jeuland	Nicolas	IFP	France
33. Keasey	Alan	Biofuels Corp	United Kingdom

Surname	Christian name	Company or Organisation	Member State
34. Kleijntjens	Rene	Argos Oil	Netherlands
35. Kupfermunz	Alain	Cargill	Belgium
36. Kvinge	Frode	Statoil	Norway
37. Lacey	Paul	Delphi Systems	United Kingdom
38. Lois	Evripidis	Technical University of Athens	Greece
39. Macrae	Colin	DFA	United Kingdom
40. Manuelli	Pascal	Total	France
41. Marcos	Jose Luis	Abengoa Bioenergy	Spain
42. Marthinsen	Geir	Exxon Mobil	Norway
43. Mittelbach	Martin	University of Graz	Austria
44. O'Connell	Adrian	EBB	EBB
45. Papachristou	Chariklia	Hellenic Petroleum	Greece
46. Pidol	Ludivine	IFP Energies Nouvelles	France
47. Schuermans	Kurt	Chevron	Netherlands
48. Scott	Mike	Argent Energy	United Kingdom
49. Stikans	Indulis	Bio-VENTA	Latvia
50. Stopper	Ingrid	OMV	Austria
51. Tamm	Ebba	SPBI Service	Sweden
52. Theeuwissen	Jan	ExxonMobil	Belgium
53. Ullmann	Jörg	Robert Bosch	Germany
54. Verdonck	Raf	OLEON Biodiesel	Belgium
55. Verschaeve	Michel	BNPé	France
56. Woldendorp	Jacco	Shell Global Solutions	Netherlands

The experts have discussed B30 fuel on the following occasions:

1) 21 May 2010, Brussels, 1st meeting of the B30 TF

2) 1 October 2010, Brussels, 2nd meeting of the B30 TF

3) 10 January 2011, Hamburg, 3rd meeting of the B30 TF

4) 1 March 2012, Paris, 20th meeting of the TF FAME

5) 9 October 2012, Paris, 21st meeting of the TF FAME

6) 26 March 2013, Brussels, 22nd meeting of the TF FAME

4.3 Planning

The planning was firstly for the B30 TF to finalise the scope and a feasibility study in time to allow WG 24 to report to CEN/TC 19 in order to make a final decision on activation at 2011 plenary meeting at the latest. For the renewed TF FAME, the planning was to establish a specification proposal to WG 24 for blending from more than 10 % (*V/V*) up to 30 % (*V/V*) of FAME in diesel fuel to be used in captive fleet application for designated vehicles.

5 The feasibility study

5.1 Background

Worldwide, energy policy makers are increasingly keen to move away from petroleum based fuels to more diverse and renewable sources of energy for reasons of environmental protection, energy security and continued economic development. As an alternative for diesel fuel many countries use fatty acid methyl ester (FAME) as a biologically based alternative. With this “biodiesel” good experience has been observed and it has resulted in many specification standards, of which EN 14214 (now in its third revision) is the best example. It is a specification fit for all type of diesel engines, where especially the demands of light-duty, Euro V and beyond engines scrutinise the biological product.

Investigation is done towards specifying a FAME quality that could be used at 10 % level (B10), but much higher levels for non-adapted light duty engines are not to be expected soon.

From a sustainability perspective, it would be beneficial to use much higher levels of FAME. In addition, use of FAME blends above 20 % would allow the oil industry to compensate for the supply of regular diesel or low blends for specific cases (protection grade, marine applications) or periods of the year. Especially, now that the EU directives require them to lower overall greenhouse gas emissions (FQD, 2009/30/EC[3]) and to reach 10 % of energy from renewable sources in transport in 2020 (RED, 2009/28/EC[4]).

About the certification fuels, the Euro V certification fuel for heavy duty vehicles is not allowed to contain any FAME at all. A truck manufacturer can, by adapting seals and tubing materials, service intervals, filter change intervals etc., make it possible to run the vehicle on B30 (or even up to B100) without bigger technical risks. This has been done by several European truck and bus manufacturers. With higher FAME-contents, it is possible that the legal limits for NOx emissions will not be fulfilled. Nevertheless, with the Euro V certification fuel for heavy duty vehicles “legal NOx limits are likely not to be met when B30….B100 is used”. Moreover, the Euro VI certification diesel fuel shall contain 6 % - 7 % FAME. In addition, there is new fuel provision in Euro VI legislation: the so called “Universal fuel type-approval”. This means that if a vehicle manufacturer wants to approve other fuels/ fuel blends (for instance B30...B50…B100), the full legal compliance shall be demonstrated also for this new fuel.

Some countries already have quality specification in place for B20 under ASTM, B30 in France[1] or even B31 in the Czech Republic for specific tax reasons[2]. Some car manufacturers already supply heavy or light duty diesel vehicles running on B20 fuel or B30 fuel in some EU countries, with particular lube and maintenance specifications. Moreover, some trucks fleet use B15 fuel.

However, a wide range of blends would make matters really difficult in view of fuel consumption and guarantee emissions. For these reasons, in order to find consensus, the group decided to work on a high FAME diesel blends (B11 - B30) and recommended to WG 24 to activate the work item as an EN specification for B11 - B30 with at least two grades of choice: a B20 fuel grade (11 % (*V/V*) – 20 % (*V/V*)) and a B30 fuel grade (21 % (*V/V*) – 29,5 % (*V/V*)). The taskforce had no specific preference whether the two grades should be presented in a single standard with national choices on the basis of climate or engine warranties, or in two separate standards for more clear pump labelling.

The TF agreed to have a maximum content of FAME of 29,5 % (*V/V*) as a start. This to avoid requesting a change of the definition of diesel in the FQD, or a change of the definition of CN-code via DG Taxud. This option is however still open to CEN.

On the other hand, DG CLIMA had set the maximum in the past to address the negative impact on NOx of higher density. This would need to be assessed from the environmental perspective. The convenor drafted a letter together with the CEN/TC 19 Chairman in order to convince the EC about the need to allow adaptation of the density requirements.

Due to the fact the scope for this B30 fuel is for captive fleet usage, i.e. a niche market, and the wish of car and truck manufacturer is to have low range FAME content to ensure emission regulations, and to respect the current national choices, WG 24 agreed to have two distinct grades, the B20 fuel (more than 15 % (*V/V*) up to 20 % (*V/V*) and the B30 fuel (more than 25 % (*V/V*) up to 30 % (*V/V*)).

In all cases the lack of standards is seen by fleet owners and suppliers as the main barrier for the fuels to reach the market. Also, because the perception is that the actual CEN standards limit the use of biofuel blends. A standard that would allow higher FAME blends would thus support the market.

5.2 Parameters to be included

High FAME diesel blends (B11 - B30) should be based on diesel fuel complying with EN 590 and FAME complying with EN 14214. This leads to on one side a set of limitations to possible contaminants resulting from both production processes and from the logistic chain, and on the other side a set of requirements that allow adapted engines to be correctly tuned for reception of this fuel.

These parameters, which are listed in Table 2, are the same for the two grades except the property that discriminates between the two grades. For the moment FAME content is considered as the most useful discriminator.

NOTE See 5.3. Parameters considered, but not included in the draft specification are discussed in 5.8.

Table 2 — Parameters to be considered for a B11 - B30 specification

	Unit	Values	Method	Applicability
Density at 15°C	kg/m^3	820 min 860 max	EN ISO 12185:1996	**OK** 600 - 1100 ; U tube method. Indicated reproducibility corresponds to that mentioned in the standard EN ISO 12185 for the transparent medium distillates.
			EN ISO 3675:1998	**OK** Aerometer - Indicated reproducibility corresponds to that mentioned in the standard EN ISO 3675 for the transparent and low viscosity products.
Kinematic viscosity at 40°C	mm^2/s	2.0 min 4,5 max	EN ISO 3104:1996	**OK** Indicated reproducibility corresponds to that mentioned in the standard EN ISO 3104:1996.
Distillation 95 % vol	°C	360 max	EN ISO 3405:2011	**OK**
Distillation Final point	°C			
Distillation % at 250°C	% (*V/V*)	65 max		
Distillation % at 350°C	% (*V/V*)	85 min		
Cetane number		51 min	EN ISO 5165:1998	**OK** the result, in terms of standard deviation, were in line with the results obtained with the other fuels tested in the scheme, with both test methods (EN ISO 5165 and EN 15195)
			EN 15195:2007	

	Unit	Values	Method	Applicability
Sulfur content	mg/kg	10 max	EN ISO 20846:2011 EN ISO 20884:2011	**OK** both EN ISO 20846 and EN ISO 20884 are suitable for the determination of sulfur in B30.The UVF (EN ISO 20846) is not affected by the presence of different levels of oxygen containing species. For WDXRF (EN ISO 20884), oxygen content of B30 is about 3,6 % that is very similar to the oxygen contents in the petrols checked during the last RRT made by WG 27. Oxygen effect would correspond to about 0.6 mg/kg sulfur that is lower than the repeatability of the method. So both methods should be applicable and the precision is expected to be similar to that published.
Flash point	°C	above 55	EN ISO 2719:2002	**OK** Pensky Martens Method.
Appearance		C&B at 20 °C		**OK**
Water content	mg/kg	250 max	EN ISO 12937:2000	**OK** Karl Fischer coulometry
Acid number after ageing @115°C	mgKOH/g	More data needed		Method under investigation
Oxidation stability	h	20 min	EN 15751:2009	**OK**
Oxidation stability	min	More data needed	EN 16091:2011	**OK**
Total contamination	mg/kg	24 max	EN 12662:2008	**OK** final paper
FAME content	% (*V/V*)	15 min 20 max (B20) 25 min 30 max (B30)	EN 14078:2009	**OK**
CFPP	°C	EN 590 and EN 16329	EN 116:1997	**OK** Regarding CFPP acc. EN 116 or EN 16329 there seem to be no specific problems for B30 and is similar to other blend ratios up to B110.
Ash content	% (*m/m*)	0,01	EN ISO 6245:2002	**OK**
Na, K, Mg, Ca content	mg/kg	More data needed		**Feasibility study**

5.3 Considerations on the parameters

5.3.1 Density

Biodiesel density is higher than that of fossil diesel fuel. The specific density values depend on fatty acid composition and purity. Most batches of FAME contain only about ten different molecules with densities usually within a very narrow range.

The diesel fuel injection is controlled volumetrically or by timing of the solenoid valve. Variations in fuel density (and viscosity) result in variations in engine power and, consequently, in engine emissions and fuel consumption. Therefore, in order to optimise engine performance and tailpipe emissions, OEMs prefers both minimum and maximum density limits shall be defined in a fairly narrow range. Moreover, the (volumetric) injection quantity is a control parameter for other emission control systems like the exhaust gas recirculation (EGR). Variations in fuel density therefore result in non-optimal EGR-rates for a given load and speed point in the engine map and, as a consequence, influence the exhaust emission characteristics.

The group felt that the same line as for B11 would be followed and that anything between 7 and 29 vol% of FAME shall follow Annex 2 of the FQD. In that case the density requirements needed to be fulfilled, so max 845 kg/m^3. According to the definition of the FQD, it was a biofuel. That gave the group the possibility of choosing the effective percentage of FAME. In case of 30 % you would be out of the FQD definition of Diesel according to the weight% of diesel remaining. Nevertheless, if we would be outside the CN-description we do not have to stick to the FQD requirements.

So two options of blends (up to 29 % and 30 % - 33 %). The range for ≤ B29 would be as in EN 590 and the range for ≥ B30 would be (830 – 860) kg/m^3 the OEMs accepting this difference of 30 kg/m^3.

The EC is asked if they felt that for anything between 10 % and 30 % Annex 2 of the FQD applied. The convenor had forwarded the question to the EC representatives through Ms Szalkowska of IFQC. First of all, DG Energy agreed to further work on the 24 – 30 % FAME in order to support the RED targets. After some discussion, DG Climate allowed further work on 24 % - 30 % FAME and a higher density. They had indicated that the next revision of the RED would include an exemption towards density and FAME content for B30 fuel. Gérald Crépeau concluded that both DGs had authorised the work of this TF.

The range was set from 820 – 860 kg/m^3 measured at 15°C. Just as for B10, the recalculation of densities measured at other temperatures requires the information that has been gathered by the EI, PTB and in France in order to correctly recalculate for FAME blends. The TF agreed that CEN/TC 19 should produce a CEN/TR on the basis of all available data in Europe in order to complement the API petroleum tables (IP 200/08[5] or ASTM D 1250-07[6], which are updates from ISO 91-1:1992[7]) and that ISO should be asked to adopt this work in parallel under the Vienna Agreement. CEN/TC 19 has accepted such a development[8].

5.3.2 Kinematic viscosity

Kinematic viscosity is an inherent property of FAME's different feedstocks that affects injector lubrication and fuel atomisation. Biodiesel fuel blends generally have improved lubricity; however, their higher viscosity levels tend to form larger droplets on injection which can cause poor combustion and increased exhaust smoke under certain operating conditions. At FAME blending levels up to 5 % by volume, the suggested limits provide an acceptable level of fuel system performance for the finished fuel blends and allow blending without changing the viscosity of the base diesel fuel. For temperatures at or below −20°C, viscosity should be at or below 48 mm^2/s to avoid potentially dangerous loads on the fuel injection pump drive system.

The limits of (2,0 – 4,5) mm^2/s as in EN 590 were adopted.

5.3.3 Fatty Acid Methyl Ester (FAME) content

The fuel's ester content indicates the amount of FAME in this high FAME diesel blends fuel (B30). Local law may require measuring the fuel's ester content; ester measurement also can help prevent or minimise fraud under incentive programs.

The group agreed to work on a range of larger than 10 % (*V/V*) up to 30 %(V/V) of FAME complying with EN 14214. However, to avoid a change of the definition of diesel can be done via the FQD, or a change of the definition of CN-code via DG Taxud, the TF agreed to have a maximum content of FAME of 29,5 % (*V/V*)V/V.

A wide range of blends would make matters really difficult in view of fuel consumption and guarantee emissions. For these reasons, in order to find consensus, the group decided to work on a high FAME diesel blends (B11 - B30) and recommended to WG 24 to activate the work item as an EN specification for B11 - B30 with at least two grades of choice: a B20 fuel grade (11 % (*V/V*) – 20 % (*V/V*)) and a B30 fuel grade (21 % (*V/V*) – 29,5 % (*V/V*)).

Due to the fact the scope for this B30 fuel is for captive fleet usage, i.e. a niche market, and the wish of car and truck manufacturer is to have low range FAME content to ensure emission regulations, and to respect the current national choices, WG 24 agreed to have two distinct grades, the B20 fuel (more than 15 % (*V/V*) up to 20 % (*V/V*) and the B30 fuel (more than 25 % (*V/V*) up to 30 % (*V/V*)).

EN 14078 is under revision for an extension of the scope to mention B30 (draft text ready for enquiry). JWG1 confirmed there is no indication that there will be problems.

5.3.4 Cold flow

Diesel fuel can have a high content of paraffinic hydrocarbons or saturated esters or glycerides which have a limited solubility in the fuel and, if cooled sufficiently, will come out of solution as wax. Adequate cold flow performance, therefore, is one of the most fundamental quality criteria for diesel fuels.

The cold flow characteristics are primarily dictated by:

— fuel distillation range, mainly the back-end volatility;

— hydrocarbon composition: content of paraffins, naphthenes, aromatics, saturated esters, Steryl glucosides (SG), saturated monoglycerides (SMG);

— presence of water

— use of cold flow additives.

Specifications for diesel cold flow properties shall be specified according to the seasonal and climatic needs in the region where the fuel is to be used. Wax in vehicle fuel systems is a potential source of operating problems; the low-temperature properties of diesel fuels are therefore defined by wax-related tests:

— Cloud Point, CP: The temperature at which the heaviest paraffins start to precipitate and form wax crystals; the fuel becomes ‘cloudy’.

— Cold Filter Plugging Point, CFPP: The lowest temperature at which the fuel can pass through the filter in a standardised filtration test. CFPP can be influenced by cold flow additives.

The group felt that the B30 fuel market was more controlled and that no immediate action on saturated monoglycerides requirements would be needed. The developments in EN 14214 would be awaited.

The suggestion to stick to the actual EN 590 table and leave the decision for the classes to be used to the NSBs was accepted, including eventual precaution notes towards fleet operations when the temperature drops too low. The fact that there was still need for a warning towards the NSBs that a blend of their diesel and FAME could not at all times reach up to the expectations was accepted too.

According to WG 14, there seemed to be no specific problems for B30 fuel regarding CFPP measurement via EN 116 or EN 16329 as it is similar to other blend ratios up to B10.

5.3.5 Distillation characteristics

The distillation curve of diesel fuel indicates the amount of fuel that will evaporate at a given temperature. The curve can be divided into three parts:

— the light end, which affects startability;

— the region around the 50 % evaporated point, which is linked to other fuel parameters such as viscosity and density; and,

— the heavy end, characterised by the T90, T95 and final boiling points, which affects emissions and engine oil dilution.

It was agreed that the distillation characteristics would remain as set until data were presented to show need for change.

As EN ISO 3405 had just been revised (to include the automatic method) and the procedure would not need to be adapted, the Energy Institute was requested to entertain a short ruggedness test in order to confirm that the precision data would apply. The scope of the test method is expanded to include biodiesel blends in the B20 – B30 range. The EI SC-B-9 committee met on 6th September 2012 and agreed to form a taskforce to coordinate the Inter-Laboratory Study (kick-off meeting to be held in November).

WG 9 recommends adding the SIMDIS method (EN ISO 3924) as alternative to the distillation, like in EN 590. This method has a scope for B10 max, but was not expected to give a different precision for B30. WG 9 and the TF suggest keeping the existing precision and take action if actual data show there is a need to do so. In case of dispute EN ISO 3405 shall be used.

5.3.6 Flash point

The flash point temperature is the minimum temperature at which the fuel will ignite (flash) on application of an ignition source under specified conditions. It is used to classify fuels for transport and storage according to hazard level; minimum flash point temperatures are required for proper safety and handling of the fuel. Flash point varies inversely with the fuel’s volatility, and biodiesel’s flash point can decrease rapidly as the amount of residual alcohol increases.

The group considers that this parameter is needed for safety and transport reasons. Flash point requirement would be “above 55°C”, like in EN 590.

5.3.7 Cetane number

Cetane number is a measure of the compression ignition behaviour of a fuel; it influences cold startability, exhaust emissions and combustion noise. Cetane index, which is based on measured fuel properties, is a calculated value that approximates the ‘natural’ cetane of a fuel. “Natural” cetane equals the cetane number when the fuel does not contain any cetane improver. The cetane number is measured on a test engine and reflects the effects of cetane improver additives. As shown below, natural cetane levels affect vehicle performance differently than cetane levels achieved through additives. To avoid excessive additive dosage, the difference between cetane index and cetane number shall be maintained.

Cetane Index would not work and the requirement was deleted. The reference would be the engine test. The original engine test did not include any Bxx-samples. ISO and ASTM/EI were reviewing and revising the CFR and DCN tests. In 2011 a single B30 fuel was tested in the EI scheme, and the result, in terms of standard deviation, was in line with the results obtained with the other fuels tested in the scheme, with both test methods (EN ISO 5165 and EN 15195). Regarding B30 fuel, in the Italian correlation scheme this fuel had been introduced. The first indications were that the CN and the DCN method were suitable for B30 fuels.

5.3.8 Sulfur content

Many governments regulate sulfur levels in fuel to ensure compatibility with emission control systems and to enable these systems to meet emission standards. Biodiesel blends may not exceed the applicable maximum sulfur levels defined for petroleum diesel fuel; fortunately, most biodiesel naturally contains less than 10 mg/kg sulfur. Feedstocks like used cooking oil and animal fat can have higher sulfur content than vegetable oils, but proper processing ensures the finished FAME respects the 10 mg/kg limit.;

Additives or logistic problems needed to be checked for the final product, so the limit was set at 10 mg/kg.

According to WG 27 and JWG 1, both EN ISO 20846 and EN ISO 20884 are suitable for the determination of sulfur in B30 fuel.The UVF (EN ISO 20846) is not affected by the presence of different levels of oxygen containing species. For WDXRF (EN ISO 20884), oxygen content of B30 fuel is about 3,6 % that is very similar to the oxygen contents in petrol checked during the last RRT executed by WG 27. Oxygen effect would correspond to about 0,6 mg/kg sulfur that is lower than the repeatability of the method. So both methods should be applicable and the precision is expected to be similar to that published.

5.3.9 Oxidation stability

Oxidation stability is very important. Oxidation stability is one of the most important properties because FAME oxidises easily compared with petroleum diesel fuel and produces reaction products that can damage the engine or vehicle. Fuels with a high number of molecules with methylene groups adjacent to double bonds are particularly susceptible to oxidation. The oxidation process begins with the processing of the seed and continues during transesterification and the lifetime of the final product. Oxidation produces peroxides (hydro-peroxides) which undergo further reaction to form acids, which are themselves oxidising agents. Molecules may also polymerise and form gums, sludge or other insoluble compounds; unlike peroxides, which usually disappear at some point during transesterification, polymers that form during oxidation do not disappear and remain in the mixture. The oxidation reaction continues until the reactive sites or available oxygen are depleted. The “Rancimat” test method, EN 15751, provides an indication of the amount of time (induction period) the fuel can be stored before the production of acids indicates the fuel is becoming unstable.

Peroxides can damage or degrade plastics and elastomers, particularly at high temperatures. The acids corrode metals used in vehicles and distribution fuel handling systems. The impact of acids on metal fuel tanks is especially severe. Even light corrosion from carboxylic acid salts will cause sticky deposits inside the fuel pump and injectors. These metals and carboxylic acids react again to form salts. These salts, which are soluble in the fuel, pass through the fuel filter but then stick to the surfaces of the fuel pump and fuel injectors. The salts also can form sludge like injector deposits. Polymers, sludge and other insoluble materials formed during oxidation can cause fuel filter blockage.

Historically, petroleum diesel fuels have been successfully stored for extremely long periods without oxidising, but even low concentrations of FAME can reduce the stability of finished blends. A few cases of lower oxidation stability than expected have been observed with biodiesel blends made with very low sulfur petroleum diesel fuels; some theorise this change may be related to the type of processing required to produce the very low sulfur levels.

Depending on storage conditions (e.g. temperature, humidity in the air space, presence of water, and other factors), FAME that meets the specified limit at the time of retail sale should provide several months of storage capability before unacceptable degradation of the FAME or blends occurs.

To reduce the potential for market problems, the oxidation stability of the blendstock shall exceed the recommended minimum induction period, and the finished fuel also shall meet its own oxidation stability requirement. If the oxidation stability of the blendstock is poor, the oxidation stability of the finished fuel will be poor. Antioxidant additives can help achieve the recommended limit; research is continuing into antioxidant types, effectiveness and dosing levels. When using antioxidants to improve the oxidation stability, producers should add the antioxidants to freshly made FAME — or even during FAME production — for maximum effectiveness. Due to fuel changes over time, fuel providers should monitor the fuel quality and take steps to minimise degradation and avoid the use of degraded fuel.

According to the specification, the 20 h Rancimat limit is more difficult to meet since higher FAME contents result in less stable products. Extra measures are required by the fuel producers. Even by keeping the IP limit value of 20 h minimum for the final fuel, the higher blend rate of 30 % FAME (B30) means, that the average oxidation stability in vehicle tanks will be lower. Although the minimum requirement for the IP of B7 is 20 h, most marketed fuels have significant higher oxidation stability. This safety margin, however, is reduced if the bio content is 30 %. Oxidation stability of fuels in vehicle tanks that have already lost some of their ageing reserve will be less replenished. As the problem was in the fuel tank, including an acid number as an alternative for the high-turnover was suggested.

Nevertheless, B30 is meant for a high turn-over use and several months' storage is not required. In the context with captive fleets, the limit of the IP was set to 20 h minimum.

PetroOxy, EN 16091, was introduced as an alternative after seeing correlation data of that test. Looking back at data presented in WG 24 by Renault, and despite the correlation during the RR was well established, correlation between the two test methods Rancimat and Petroxy was difficult to observe. The fact that hydrotreatment in the refinery takes out the natural anti-oxidants and the cetane improver impacts was discussed in the TF.

In the end, the group had assessed all three test methodologies and recommend further studying of the field data. Moreover, as the enquiry on the revision for EN 590 would start halfway 2011, the group felt that the plan of the programme would need to be debated between JWG 1 and WG 24 to be concluded in May 2011. JWG 1 was requested to draft a test programme proposal for WG 24 to decide upon. On the basis of the suggestions, CONCAWE would be asked to work on the fuels matrix.

The round robin was realised to find correlation between Rancimat and PetroOxy in order to support limit setting for EN 16091. There is a relationship, which is blurred to some extent by the imprecision of both test methods.

EN ISO 12205[9] should not be used.

Acid number after ageing is still under development as pass/fail method. Its precision is not sufficient for a standard test method.

For the newly developed Rancimat at 120 °C test method the precision for B30 seems to be acceptable (equivalent precision to EN 15751 at 110 °C). This new procedure could be an alternative to the current proposed methods. The draft text is ready for enquiry.

5.3.10 Total contamination

The fuel should be clear in appearance and free of sediment/total contamination. The presence of these materials can shorten filter life or plug fuel filters, which can lead to engine fuel starvation.

Esterification and product refining processes, along with feedstock selection, tend to produce many types of contamination, such as soap. Poor fuel handling practices can introduce other contaminants or compounds that are not soluble in FAME. Small amounts of such contamination will cause fuel filter plugging and injector deposits and should be well-controlled to prevent problems.

Engine and fuel injection equipment manufacturers continue to develop fuel systems to reduce emissions and fuel consumption and to improve performance. Injection pressures have increased dramatically; current injectors operate at pressures as high as 2400 bars, and injectors with even higher pressure capabilities are in development. Such levels of injection pressure demand reduced orifice sizes and component clearances, typically ranging from (2 to 5) µm in injectors. Small, hard particles, which may remain in the fuel that are carried into these engine parts, are potential sources of injector and subsequent engine failure.

WG 31 executed a round robin approved by WG 24 and WG 36. Momentarily, EN 12662 specifies a method for the determination of total contamination as the content of undissolved substances in middle distillates, in diesel fuels containing up to 30 % (*V/V*) fatty acid methyl esters (FAME), and in pure FAME. The precision and working range is incorporated in EN 12662 draft under enquiry.

5.3.11 Appearance

The requirement “clear and bright” by visual inspection at 20°C is set. The water content limit that is acceptable for B30 level blending might give problems at B11 level, so an appearance check at the fuelling pump is needed. Also for total contamination effects a visual inspection on insoluble materials is needed.

5.3.12 Water content

Water accelerates oxidation, dramatically increases corrosivity and promotes microbial growth. Since FAME is capable of absorbing significantly more water than petroleum diesel fuel, it is especially important to reduce the water content of the FAME during production and to minimise the potential to create free water during blending or distribution. The level of water specified is within the solubility level of water in fuel and, as such, does not represent free water. 250 mg/kg max water content was accepted by the group.

5.3.13 Ash content

Ash is a measure of the amount of metals and other inorganic contaminants contained in the fuel. Ash precursors may be present in three forms - (i) abrasive solids, (ii) soluble metallic soaps, and (iii) residual biodiesel catalyst; when oxidised during combustion, these materials form ash.

Ash has been linked to engine deposits, particulate filter plugging and oxidation catalyst deactivation. Metal salts and fatty acid soaps can further contribute to deposits in the entire fuel system. These abrasive solids cause increased wear.

Diesel particulate filters (DPF), which began appearing in mid-2000 to achieve extremely low particulate emissions and stringent emission standards, are particularly susceptible to impairment from ash. All ash forming compounds can contribute to the accumulation of material on these filters. Rapid accumulation of ash requires more frequent filter maintenance.

CONCAWE had done extensive measurements on the logistic chain on diesel fuels that had shown very low levels of contribution. So the risks of introducing ashes were low. The latest CONCAWE survey data on the logistic chains with regards to metals are presented. In most cases, the 26 metals measured were below the detection limit.

Problems occurring in common rail systems with internal diesel injector deposits that contain numerous amounts of sodium. OEMs and injection system suppliers proposed to control the sodium in the finished fuel. This hard insoluble deposit affects injector and engine performance (power loss, increased noise, poor starting, rough running, etc…). Back in 2011, the WG 24 convenor expressed the need to better understand the source of the sodium, in order to see where more could be done. He asked the fuel injector manufacturers to try to give more indications to CONCAWE on the effective presence of the problems in order to better understand the problems initiated by the logistics. As a result of these diesel vehicle common rail injector sticking field problems WG 24 recommended and CEN/TC 19 endorsed the formation of an Ad hoc task force to urgently investigate the injector sticking issue (TF IDID).Two types of internal injector deposits reported:

— Carboxylate soap deposits

— Amide deposits (from DDS or HDS)

The TF IDID agreed to focus on carboxylate soap deposits initially since it is most serious and independent of amide deposit issues.

After a year of study, the TF had strong evidence that the use of the sodium nitrite corrosion inhibitor in the French pipeline system is contributing to the high level of injector sticking incidents in France. It was recommended that use of this additive was immediately discontinued and alternate harm free corrosion inhibitor technology deployed. DDS and HDS acid corrosion inhibitors have also been implicated with sporadic injector sticking issues in the USA, Denmark and Spain and it is recommended that alternative corrosion inhibitor technologies (Dimer) be considered for use in multi-product pipeline systems that are believed to be harm free.

The pipeline authorities in France stopped using sodium-containing additives. The TF IDID hoped that the pipeline operators would take steps towards not using those additives anymore as quickly as possible. Similar activities towards alternative additives were undertaken by CLH in Spain. In 2013, the number of customer complaints linked to Internal Diesel Injectors Deposits have greatly decreased. Further details are reported in the TF IDID report[10].

Based on the above assessment,. the TF FAME experts agreed to keep the ash content at 100 mg/kg for B30 and include a requirement on sodium with no levels. WG 27 would be requested to address the test method for sodium and improve the detection limit for sodium in biodiesel blends. For sodium (and alkalines) determination, tests shall be run in order to evaluate the quantification limit that can be reached and to define the specification depending on that result. Some work has been done by WG 27 and should be continued (by WG 27 or by JWG1).

5.4 Dyes and markers

The sentences from the EN 590 should be duplicated:

The use of dyes or markers is allowed.

5.5 Additives

The general additives clause would be completed with the adulterant and the FAME oxidation stability enhancing additives section of EN 590:

In order to improve the performance quality, the use of additives is allowed. Suitable fuel additives without known harmful side-effects are recommended in the appropriate amount, to help to avoid deterioration of driveability and emissions control durability. Other technical means with equivalent effect may also be used.

It is strongly recommended to add oxidation stability enhancing additives in the FAME product, at the production stage and before storage, providing an action similar to that obtained with 1 000 mg/kg of 3,5-di-tert-butyl-4-hydroxy-toluol (butylated hydroxyl-toluene, BHT).

5.6 Sampling

Samples shall be taken as described in EN ISO 3170 or EN ISO 3171 and/or in accordance with the requirements of national standards or regulations for the sampling of automotive diesel fuel. The national requirements shall be set out in detail or shall be referred to by reference in a National Annex to this European Standard.

In view of the sensitivity of some of the test methods referred to in this European Standard, particular attention shall be paid to compliance with any guidance on sampling containers which is included in the test method standard.

5.7 Pump marking

Information to be marked on dispensing pumps used for delivering high FAME diesel blends, and the dimensions of the mark shall be in accordance with the requirements of national standards or regulations for the marking of pumps for high FAME diesel blends. Such requirements shall be set out in detail or shall be referred to by reference in a National Annex to this European Standard.

The group recommends a label that indicates “B30”, includes a notice that it is a special fuel and that one should check his car warranties. A single colour of the dispenser would be ideal.

5.8 Parameters considered and not included in the draft specification

5.8.1 Linolenic Acid Methyl Ester

This ester, which is inherent to certain feedstocks, contains three double bonds which make the molecule highly unstable. Thus, these molecules oxidise or polymerise easily, thereby creating acids or sludge. The group thinks this parameter is not needed and difficult to measure in B30 fuels.

5.8.2 Aromatics

Aromatics are molecules that contain at least one benzene ring. The fuel aromatic content will affect combustion, flame temperature, particulates and PAH emissions and therefore, NOx emissions.

5.8.3 Glycerides: Mono, Di, and Tri

Glycerides, which are high molecular weight reactants in the biodiesel production process, can remain unreacted in the FAME after processing. They are undesirable because they can cause filter plugging, especially at cold temperatures, and injector and engine deposits; they can also limit vehicle operability over a wide range of conditions. Saturated monoglycerides can lead to increased injector deposits. High monoglyceride content has been linked to solid deposits in B5 fuel where the fuel was stored in tanks under Scandinavian winter conditions with MK1 diesel. Other problems related to saturated monoglycerides were observed in France when a high level of palm FAME was introduced during winter in 2009.

Diglycerides can lead to filter plugging. Triglyceride content is a good indicator of unreacted oils or greases in the biodiesel.

The group thinks this parameter is not needed and difficult to measure in B30 fuels.

5.8.4 Glycerine (glycerol), total and free

Glycerine, which is a by-product of the chemical reaction that produces biodiesel, may remain in the fuel if the ester is inadequately separated or washed. Glycerine also may separate out of the liquid during storage after any methanol, which acts as a solvent, has evaporated. Once separated, the glycerine will fall to the tank bottom and attract such polar compounds as water, monoglycerides and soaps that can block filters, damage injectors, cause injector coking and other engine deposits, and otherwise make the fuel incompatible with vehicle materials and reduce engine durability.

The group thinks this parameter is not needed and difficult to measure in B30 fuels.

5.8.5 Total acidity

Acid number is a measure of the acids in the fuel. These acids emanate from two sources: (i) acids used in the production of the biodiesel that are not completely removed in the production process; and (ii) as a product from degradation by oxidation. The acids are measured in terms of amount of KOH required to neutralise a gram of fuel. For biodiesel blends, the acid number will change as a result of the normal oxidation process over time, and recent research has shown that this change is a good indicator of B100 stability. If the fuel is not used immediately after purchase, buyers should monitor their biodiesel fuel blends for changes in acid numbers, as an indicator of fuel degradation. The presence of acids in the fuel can harm injection systems and other metallic components.

Stable products with a remaining induction period do not evolve ageing acids. The total acidity parameter therefore does only cover fatty acids from biodiesel production. Fatty acids are already limited in the B100 EN 14214 fuel specification. It would be redundant to limit total acidity again in the B30 fuel specification. The group agreed to not introduce this parameter.

5.8.6 Iodine number

Iodine number indicates the total number of double bonds (i.e. level of saturation) in the mixture of molecules. Opinions differ regarding whether the iodine number helps indicate oxidation stability, where a higher iodine number represents lower oxidation stability. To the extent it does help indicate oxidation stability, the iodine number may provide information about the fuel's tendency to form sludge, affect lubricant quality and/or cause corrosion.

The FAME follows EN 14214, so the group agreed to not introduce this parameter.

5.8.7 Alkali metals (Na + K)

Biodiesel producers who use sodium and potassium to catalyse biodiesel production should remove these metals before allowing the biodiesel to leave the production process. Moreover, sodium salt can be used as corrosion inhibitors for fuel transport by pipeline. Residual alkali metals can form deposits in fuel injection system components and poison emission control systems. Sodium and potassium are also associated with ash formation.

5.8.8 Alkaline earth metals (Ca + Mg)

Biodiesel producers who use alkaline earth metals as absorbents during biodiesel production should remove these metals before allowing the biodiesel to leave the production process. Residual alkaline earth metals can form deposits in fuel injection system components and poison emission control systems. Calcium soaps can cause injection pumps to stick.

5.8.9 Phosphorus

Phosphorus will be present in biodiesel at trace levels from phospholipid compounds naturally found in plant oils and inorganic salts that may be contained in used cooking oils. Phosphorus can greatly impair the effectiveness of emission control systems. Its influence is cumulative, which means that even very low levels in the fuel may lead to premature deterioration over time, especially when an engine consumes a significant amount of contaminated fuel.

For metals and phosphorus, the group feels the improvement shall be made via EN 14214.

5.8.10 Lubricity

The group feels this parameter is not needed at this stage although being called up in the Italian standard.

5.8.11 Copper strip corrosion

At the beginning of the discussions, with a 10 mg/kg sulfur limit, the copper strip corrosion was still needed. Especially if a grade 2 needed a highly problematic fuel. WG 24 had discussed the replacement in favour of a silver strip test.

After checking the test, experts saw no added value anymore for high FAME diesel blends. The copper strip corrosion test was deleted from the table.

5.8.12 Housekeeping guidance

The group agreed that housekeeping guidance was needed and as a start a reference should be made to CEN/TR 15367-1[11]. WG 24 has been requested to consider revision of the CEN/TR and that was accepted.

6 Conclusions

In general, the group was positive about the feasibility of a B30 specification. On 8 November 2012, it was therefore recommended to WG 24 to activate the existing work item as a full EN specification and adopt a NWI for progressing this report into a CEN/TR in order for it to support the enquiry ballot.

This decision has been adopted by WG 24 and the group has been asked to finalise the specification on the basis of two separate classes, preferably a B20 fuel (more than 15 % (*V/V*) up to 20 % (*V/V*) and a B30 fuel (more than 25 % (*V/V*) up to 30 % (*V/V*))..

7 Acknowledgement

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