BSI Standards Publication

Automotive fuels — Paraffinic diesel fuel from synthesis or hydrotreatment — Requirements and test methods

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National foreword

This British Standard is the UK implementation of EN 15940:2016. National Annexes NA, NB and NC contain sampling requirements, pump marking requirements and climate-dependent requirements.

The UK participation in its preparation was entrusted to Technical Committee PTI/2, Liquid Fuels.

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

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Amendments/corrigenda issued since publication

Date	Text affected
30 November 2016	National Annexes NA, NB and NC added

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EN 15940

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English Version

Automotive fuels - Paraffinic diesel fuel from synthesis or hydrotreatment - Requirements and test methods

Carburants pour automobiles - Gazoles paraffiniques de synthèse ou obtenus par hydrotraitement - Exigences et méthodes d'essais

Kraftstoffe für Kraftfahrzeuge - Paraffinischer Dieselkraftstoff aus Synthese oder Hydrierungsverfahren - Anforderungen und Prüfverfahren

This European Standard was approved by CEN on 15 April 2016.

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European foreword

This document (EN 15940:2016) has been prepared by Technical Committee CEN/TC 19 "Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin", the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2016, and conflicting national standards shall be withdrawn at the latest by December 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 15940:2012.

Significant other technical changes between this document and CEN/TS 15940:2012 are:

a) the limits for distillation at 250 °C and 350 °C are included in line with EN 590 and the EU Common Customs tariff for diesel fuel;

b) EN ISO 3924, also known as simulated distillation, has been incorporated in Table 1 as an additional methodology to determine distillation characteristics;

c) prEN 16906 (EN equivalent to DIN 51773, *Testing of liquid fuels — Determination of ignition quality (cetane number) of Diesel fuels with the BASF-engine*) has been studied and allowed as an additional methodology to determine cetane number;

d) in order to allow fitness for purpose product and to align it with EN 590 product that has proven functionality in diesel engines, the arctic climate viscosity limits and the distillation recovery at 180 °C have been introduced in Table 3;

e) in order to present all relevant requirements within the same fuel specification, the necessary climate dependent properties from EN 590 have been introduced in 5.7; this required reference of some additional test methods in Clause 2;

f) further clarification on the oxidation stability requirement, as a result of recent changes in EN 15751, has been introduced;

g) exclusion of special sampling procedures for clean paraffinic fuel as they apply to diesel fuel in general;

h) to further underline the link with EN 590 that normative reference being stipulated without reference to a particular date of publication;

i) introduction of an annex of the precision data for test methods where different from normal diesel fuel in following the CEN/TC 19 interlaboratory study [1];

j) introduction of an annex covering a test procedure for aromatics content determination being developed as part of a second interlaboratory study funded by the EC on three different HPLC techniques;

k)   introduction of an annex on density – temperature corrections being developed as part of a CEN/TC 19 investigation led by Mr. H. Th. Feuerhelm of DIN-FAM.

In this document, all relevant characteristics, requirements and test methods are specified. These specifications are relevant for the driveability of the vehicles and are currently known to prevent harm to the vehicles and their powertrains. Climate dependent requirements of this document may vary according to national adoptions of EN 590 and EN 14214, but should be indicated by a specific National Annex.

Several assessments of test methods for paraffinic diesel fuel have been executed and the results thereof [1] led to conclusions regarding the applicability of each of the test methods as required in Clause 5. The conclusion of these assessments, partially funded by the European Commission, led to the possibility to upgrade the original Technical Specification into a full European Standard. Although it is its main actual use, the product is now no longer limited to captive fleet usage, but the scope defines the need to check the use of the product with the vehicle manufacturer. There are no EU legislative needs to limit the product to captive fleets. Such restriction is not for the specification but for the market to decide upon. Therefore, and in the light of the defined need to check the use of the product with the vehicle manufacturer, all restrictions towards captive fleet from the CEN/TS text have been deleted

This document is based on current knowledge at the time of publishing, but will require revision when the specification for either regular automotive diesel fuel, EN 590, or FAME, EN 14214, has been determined (revised) by CEN/TC 19 or based on further experiences with the use of paraffinic diesel fuel according to this document. Further background can be found in CEN/TR 16389 [2].

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This document has been laid down to define a quality specification for diesel fuel on the basis of synthesis gas or of hydrotreated bio-oils or -fats. Its main use is as diesel fuel in dedicated diesel vehicle fleets and engines. Paraffinic diesel fuel does not meet the current diesel fuel specification, EN 590. The main differences between paraffinic diesel fuel and automotive diesel fuel are in the areas of density, sulfur, aromatics and cetane. Its density can be outside the regular diesel specification, and the described class A type fuel has a higher cetane number. Paraffinic diesel fuel is not validated for all vehicles, consult vehicle manufacturer before use.

Paraffinic diesel is a high quality, clean burning fuel with virtually no sulfur and aromatics. Paraffinic diesel fuel can be used in diesel engines (see NOTE 1 under Clause 1 and the last paragraph of Clause 4), also to reduce regulated emissions. In order to have the greatest possible emissions reduction, a specific calibration may be necessary. Paraffinic diesel fuel can also offer a meaningful contribution to the target of increased non-petroleum and/or renewable content in transportation fuel pool.

As some production processes result in a fuel containing *cyclo*-paraffins, as well as *n*-paraffins and *iso*-paraffins, they show different cetane number compared to other paraffinic diesel fuels. Hence, in this document, two classes have been defined, one class showing improved ignition quality compared to regular diesel fuel.

Blending of paraffinic diesel with biodiesel (FAME) is covered in this document. Against the background of the EU Renewable Energy Directive (RED, 2009/28/EC [3]) and also the latest developments regarding European regular diesel specification, there is now a pressing requirement to allow for FAME blend variations of those paraffinic fuels, which are not already classified as being from renewable resources.

As with CEN/TS 15940, this document allows for a paraffinic diesel specification other than the former CWA 15940, to "mirror" the current EN 590 diesel fuel quality specification. That is, allowing a blend variant of paraffinic diesel in the same way that CEN diesel quality specifications allow for refinery diesel up to 7 % (*V/V*) FAME blend percentage.

Paraffinic diesel may also be used as a blending component for automotive diesel fuel, but this is not in the scope of this document.

The document will be usable on a voluntary basis for engine clearance, fuel acceptance and fuelling station allowance, supporting both local regulations and international trade.

1 Scope

This European Standard describes requirements and test methods for marketed and delivered paraffinic diesel fuel containing a level of up to 7,0 % (*V/V*) fatty acid methyl ester (FAME). It is applicable to fuel for use in diesel engines and vehicles compatible with paraffinic diesel fuel. It defines two classes of paraffinic diesel fuel: high cetane and normal cetane.

Paraffinic diesel fuel originates from synthesis or hydrotreatment processes.

NOTE 1   For general diesel engine warranty, paraffinic automotive diesel fuel may need a validation step, which for some existing engines may still need to be done (see also the Introduction to this document). The vehicle manufacturer needs to be consulted before use.

NOTE 2   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.

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 116:2015, *Diesel and domestic heating fuels — Determination of cold filter plugging point — Stepwise cooling bath method*

EN 12662:2014, *Liquid petroleum products — Determination of total contamination in middle distillates, diesel fuels and fatty acid methyl esters*

EN 14078:2014, *Liquid petroleum products — Determination of fatty acid methyl ester (FAME) content in middle distillates — Infrared spectrometry method*

EN 14214:2012+A1:2014, *Liquid petroleum products — Fatty acid methyl esters (FAME) for use in diesel engines and heating applications — Requirements and test methods*

EN 15195:2014, *Liquid petroleum products — Determination of ignition delay and derived cetane number (DCN) of middle distillate fuels by combustion in a constant volume chamber*

EN 15751:2014, *Automotive fuels — Fatty acid methyl ester (FAME) fuel and blends with diesel fuel — Determination of oxidation stability by accelerated oxidation method*

EN 16136:2015, *Automotive fuels — Determination of manganese and iron content in unleaded petrol — Inductively coupled plasma optical emission spectrometry (ICP OES) method*

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

prEN 16906, *Liquid petroleum products — Determination of the ignition quality of diesel fuels — BASF engine method*

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

EN ISO 1042:1999, *Laboratory glassware — One-mark volumetric flasks (ISO 1042:1998)*

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

EN ISO 2719:2002, *Determination of flash point — Pensky-Martens closed cup method (ISO 2719:2002)*[1]

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

EN ISO 3170:2004, *Petroleum liquids — Manual sampling (ISO 3170:2004)*

EN ISO 3171:1999, *Petroleum liquids — Automatic pipeline sampling (ISO 3171:1988)*

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 3924:2010, *Petroleum products — Determination of boiling range distribution — Gas chromatography method (ISO 3924:2010)*

EN ISO 4259:2006, *Petroleum products — Determination and application of precision data in relation to methods of test (ISO 4259:2006)*

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 10370:2014, *Petroleum products — Determination of carbon residue — Micro method (ISO 10370:2014)*

EN ISO 12156-1:2016, *Diesel fuel — Assessment of lubricity using the high-frequency reciprocating rig (HFRR) — Part 1: Test method (ISO 12156-1:2016)*

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 Fischer titration method (ISO 12937:2000)*

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

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)*

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1) Under revision.

3 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, should it be adopted on a national level.

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

4 Pump marking

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

Paraffinic diesel fuel shall be distinguished from other diesel fuel by a dedicated marking[2].

Labelling shall be clearly visible, easily legible and displayed at any point where paraffinic diesel with metallic additives is made available to consumers. In that case, the label shall contain "Contains metallic additives" in the national language(s) and shall be laid down in the National Annex to this document.

Further indication on dispensing pumps in the national language of "Not suitable for all vehicles; consult vehicle manufacturer (information) or manual before use" is also recommended.

5 Requirements and test methods

5.1 Dyes and markers

The use of dyes or markers is allowed.

5.2 Additives

5.2.1 General

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.

NOTE   Deposit forming tendency test methods suitable for routine control purposes have not yet been identified and developed.

5.2.2 Methylcyclopentadienyl Manganese Tricarbonyl (MMT)

When methylcyclopentadienyl manganese tricarbonyl (MMT) is used, a specific labelling is required (see Clause 4). The presence of the MMT in paraffinic diesel fuel shall be limited to 2 mg of manganese per litre.

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2) CEN is developing a generic fuel labelling standard, EN 16942 [24], for this.

5.3 Fatty acid methyl ester (FAME)

Paraffinic diesel fuel may contain up to 7,0 % (*V/V*) of FAME complying with EN 14214:2012+A1:2014, in which case the climate-dependent requirements set out in EN 14214:2012+A1:2014, 5.4.2 do not apply.

NOTE   A suitable method for the separation and identification of FAME is given in EN 14331 [4].

5.4 Cavitation prevention

Fuels with an initial boiling point (IBP) below 160 °C, as determined by EN ISO 3405, may impose a risk of cavitation damage.

The IBP of paraffinic diesel fuels shall be measured and reported using EN ISO 3405.

NOTE   This issue is further being studied by CEN. For explanation on the risks, see CEN/TR 16389 [2].

5.5 Seizure protection

There has been successful usage of paraffinic fuels for well over 12 years, whilst no issues due to insufficient lubricity have been reported. However, there are indications that diesel fuel high in paraffin content does not always protect fuel system components sufficiently against seizure. The lubricity requirement in Table 1 ensures protection against wear but not necessarily also against seizure. Appropriate seizure protection shall be provided by using suitable fuel additives or by blending of minimum 2 % (*V/V*) of FAME.

NOTE   For further information, see Annex A.

5.6 Generally applicable requirements and related test methods

5.6.1   When tested by the methods indicated in Table 1, paraffinic diesel fuel shall be in accordance with the limits specified in Table 1 for either Class A (high cetane paraffinic diesel fuel) or Class B (normal cetane paraffinic diesel fuel).

NOTE 1   All values in Table 1 meet the requirements of the European Fuels Directive 98/70/EC [5], including Amendments 2003/17/EC [6], 2009/30/EC [7] and 2014/77/EU [8].

NOTE 2   For further clarification of the Classes, see CEN/TR 16389 [2].

5.6.2   The limiting value for the cetane number given in Table 1 is based on product prior to addition of cetane improver.

5.6.3   The correlation equation in EN 15195:2014, Clause 12 shall be used. This and similar test methods, such as EN 16715 [9] and ASTM D6890 [10], provide for an alternative correlation equation, specifically for low ignition delay (high cetane) diesel fuels, which will produce a different result and which should not be used. There is no precision statement available for the alternative correlation equation. Special care should be taken with the automated equipment to ensure that it does not apply this alternative equation by default.

Table 1 — Generally applicable requirements and test methods

Property	Unit	Limits Class A		Limits Class B		Test method [a]
		minimum	maximum	minimum	maximum	(See Clause 2)
Cetane number [b]		70,0	–	51,0	-	EN 15195 [c] prEN 16906 EN ISO 5165
Density at 15 °C	kg/m^3	765,0	800,0	780,0	810,0	EN ISO 3675 [d] EN ISO 12185
Flash point	°C	Above 55,0	–	Above 55,0	–	EN ISO 2719
Viscosity at 40 °C	mm^2/s	2,000	4,500	2,000	4,500	EN ISO 3104
Distillation % (*V/V*) recovered at 250 °C % (*V/V*) recovered at 350 °C 95 % (*V/V*) recovered at	% (*V/V*) % (*V/V*) °C	– 85 –	< 65 – 360	– 85 –	< 65 – 360	EN ISO 3405 [e] EN ISO 3924
Lubricity, wear scar diameter (wsd) at 60 °C [f]	μm	–	460	–	460	EN ISO 12156-1
FAME content [g]	% (*V/V*)	–	7,0	–	7,0	EN 14078
Manganese content [h]	mg/l	–	2,0		2,0	EN 16136
Total aromatics content [i]	% (*m/m*)	–	1,1	–	1,1	Annex C
Sulfur content	mg/kg	–	5,0	–	5,0	EN ISO 20846 [j] EN ISO 20884
Carbon residue (on 10 % distillation residue)[k]	% (*m/m*)	–	0,30	–	0,30	EN ISO 10370
Ash content	% (*m/m*)	–	0,010	–	0,010	EN ISO 6245
Water content	mg/kg	–	200	–	200	EN ISO 12937
Total contamination	mg/kg	–	24	–	24	EN 12662 [l]
Copper strip corrosion (3 h at 50 °C)	rating	class 1		class 1		EN ISO 2160
Oxidation stability	g/m^3	–	25	–	25	EN ISO 12205
	h	20,0 [m]	–	20,0 [m]	–	EN 15751

[a]   See also 5.8.1. All test methods are applicable to paraffinic diesel fuels without any specific problems. See also Annex B.

[b]   See also 5.6.2.

[c]   See 5.8.3. To ensure the correct correlation equation is applied see 5.6.3.

[d]   See also 5.8.7. For correction of the determined value for paraffinic diesel fuel Annex D shall be applied.

[e]   See also 5.8.4. Limits are in EN ISO 3405 scale. EN ISO 3924 results shall be converted according to EN ISO 3924:2010, Annex A.

[f]   See also 5.5.

[g]   FAME shall meet the requirements of EN 14214, see 5.3.

[h]   See also 5.2.2.

[i]   Total aromatics content includes polycyclic aromatic hydrocarbons. If the product conforms to the limit in the table it also conforms to actual legal limits on polycyclic aromatic hydrocarbons content. See also 5.6.6 and 5.8.2.

[j]   See also 5.8.5.

[k]   See also 5.6.4.

[l]   If the filtration time exceeds 30 min the test should be stopped and the result reported as an incomplete filtration together with the volume filtered. Failure to complete the filtration in 30 min means the product under test does not comply with this European Standard.

[m]   For paraffinic diesel fuel containing FAME above 2 % (*V/V*) this is an additional requirement.

5.6.4 The limiting value for the carbon residue given in Table 1 is based on product prior to addition of ignition improver, if used. If a value exceeding the limit is obtained on finished fuel in the market, EN ISO 13759 shall be used as an indicator of the presence of a nitrate-containing compound. If an ignition improver is thus proved present, the limit value for the carbon residue of the product under test cannot be applied. The use of additives does not exempt the manufacturer from meeting the requirement of maximum 0,30 % (*m/m*) of carbon residue prior to addition of additives.

5.6.5 Tests have shown that EN 15751 is applicable for paraffinic fuels with a FAME content above 2 % (*V/V*) covered under this European Standard. Paraffinic diesel fuels can have an induction period above 48 h thus exceeding the maximum measuring time considered for the precision statement in EN 15751. However, even taking into account that the precision above the 48 h can get worse, such fuels are considered having a oxidation stability significantly above the limit set in this European Standard.

5.6.6 Paraffinic diesel fuel shall be free from any adulterant or contaminant that may render the fuel unacceptable for use in diesel engine vehicles.

NOTE 1   For further information on preventing contamination by water or sediment that can occur in the supply chain, it is advisable to check CEN/TR 15367–1 [11].

Any intentional addition of non-paraffinic material, other than additives and dyes or markers, is not allowed.

NOTE 2   Paraffinic diesel fuel before blending with FAME is expected to contain more than 98,5 % (*m/m)* of paraffinic hydrocarbons.

NOTE 3   As no test method for determination of the paraffinic hydrocarbon content is available at the time of publication of this European Standard, total aromatics are limited in Table 1. See also CEN/TR 16389 [2].

5.7 Climate dependent requirements and related test methods

5.7.1 For climate-dependent requirements options are given to allow for seasonal grades to be set nationally. The options are for temperate climates six CFPP (cold filter plugging point) grades and for arctic or severe winter climates five different classes. Climate-dependent requirements are given in Table 2 (temperate climates) and Table 3 (arctic or severe winter climates). When tested by the methods given in Tables 2 and 3, paraffinic diesel fuel shall be in accordance with the limits specified in these Tables.

Table 2 — Climate-related requirements and test methods - Temperate climates

Property	Unit	Limits						Test method [a]
		Grade A	Grade B	Grade C	Grade D	Grade E	Grade F	(see Clause 2)
CFPP	°C, max.	+5	0	−5	−10	−15	−20	EN 116 [b] EN 16329
[a] See also 5.8.1								
[b] See also 5.8.6.								

5.7.2 When adopting this European Standard, each country shall in a National Annex detail requirements for a summer and a winter grade and may include (an) intermediate and/or regional grade(s) which shall be justified by national meteorological data. It is strongly recommended to follow the same grades as in the National Annex to EN 590, which has detailed requirements that relate to the Tables 2 and 3 of this document.

Table 3 — Climate-related requirements and test methods - Arctic or severe winter climates

Property	Units	Limits					Test method [a]
		class 0	class 1	class 2	class 3	class 4	(see Clause 2)
CFPP	°C, max.	−20	−26	−32	−38	−44	EN 116 [b] EN 16329
Cloud point	°C, max.	−10	−16	−22	−28	−34	EN 23015
Viscosity at 40 °C	mm²/s, min. mm²/s, max.	1,500 4,000	1,500 4,000	1,500 4,000	1,400 4,000	1,200 4,000	EN ISO 3104
Distillation							EN ISO 3405 [c] EN ISO 3924
recovered at 180 °C	% (*V/V*), max.	10,0	10,0	10,0	10,0	10,0	
recovered at 340 °C	% (*V/V*), min.	95,0	95,0	95,0	95,0	95,0	
[a] See also 5.8.1.							
[b] See also 5.8.6.							
[c] See also 5.8.4. Limits are in EN ISO 3405 scale. EN ISO 3924 gives instructions to convert to EN ISO 3405-equivalent data.							

5.8 Precision and dispute

5.8.1 The test methods referred to in this document include a precision statement based on regular diesel fuel matrices. In cases of dispute, the procedures for resolving the dispute and interpretation of the results based on test method precision, described in EN ISO 4259, shall be used. Where precision data are known to be different for paraffinic diesel fuels as listed in Annex B, precision data from Annex B shall be used.

5.8.2 The test method for contents of total aromatics content in Annex C has not been assessed at all required levels, but a first feasibility study regarding actual field experience has indicated that the method is applicable.

NOTE   It is the intention to include data of a pending Round Robin in future publications of this document.

5.8.3 In cases of dispute concerning cetane number, EN 15195 shall be used. For the determination of cetane number, alternative methods to those indicated in Table 1 may also be used, provided that these methods originate from a recognized method series and have a valid precision statement, derived in accordance with EN ISO 4259, which demonstrates precision at least equal to that of the referenced method. The test result, when using an alternative method, shall also have a demonstrable relationship to the result obtained when using the referenced method.

5.8.4 In cases of dispute concerning distillation, EN ISO 3405 shall be used.

5.8.5 In cases of dispute concerning sulfur, EN ISO 20846 shall be used.

5.8.6 In cases of dispute concerning CFPP, EN 116 shall be used.

5.8.7 In cases of dispute concerning density, EN ISO 12185 shall be used.

Annex A
(informative)

Seizure protection

At this time, meeting a minimum scuffing load of 3 500 g as measured by the SL-BOCLE test [12] is found sufficient to avoid seizure. Following years of experience with standard diesel fuels adequate seizure protection under field conditions is given, if fuels pass a limit value of > 3 500 g in the SL-BOCLE test. This is generally the case for conventional crude oil based diesel fuels having sufficient protection against wear i.e. the Wear Scar Diameter (WSD) of the ball does not exceed 460 µm in the HFRR test (EN ISO 12156-1).

In the ASTM standard specification for diesel fuel oils [13], a HFRR wear scar diameter (WSD) of maximum 520 µm has been placed, and a SL-BOCLE value of > 3 100 g has been referenced for giving sufficient lubricity. The diesel vehicle fleet in Europe is generally validated for fuels with a HFRR wear scar diameter of maximum 460 µm thus correlating to a SL-BOCLE value of > 3 500 g [14].

There has been successful usage of paraffinic fuels for well over 12 years in captive fleet and general purpose usage. No complaints or failures due to insufficient lubricity have been reported so far. However, no or low aromatic fuels do not necessarily have the same good lubrication characteristics as crude oil based diesel fuels protecting simultaneously against wear and seizure. Some paraffinic fuels poor in "natural" seizure protection do not protect against seizure even if the WSD in the HFRR test is adjusted to < 460 µm. All lubricity additives reduce the risk of wear. However, depending on the nature of the fuel and the type and concentration of additive used for adjusting the lack of lubricity, adequate seizure protection is not necessarily ensured by low values in the HFRR test on its own.

Appropriate seizure protection of paraffinic fuels shall be provided. Up until now the lubricating characteristics of paraffinic fuels are neither extensively studied in laboratory tests nor fully validated in system and engine tests thus making the assessment of the risk of seizure difficult. As a consequence it is recommended to use only fuels that pass a limit value of > 3 500 g in the SL-BOCLE test. This limit is preliminary and might be revised when indicated by additional investigation results.

The SL-BOCLE method is known to have poor reproducibility and repeatability and for this reason it is not practical to analyse each batch of fuel. Fuel producers may show that sufficient seizure protection is provided for a given fuel and additive combination and dose rate through an initial series of tests for both SL-BOCLE and HFRR. Thereafter, for each batch of fuel additized at the selected dose rate with the same additive, sufficient protection is demonstrated by the HFRR result as is required by this specification in Table 1.

Further work is executed in CEN to understand this phenomenon.

Annex B
(normative)

Details of interlaboratory test programme

The precision data given in Table B.1 apply in the case of paraffinic diesel fuel (with up to 7 % (*V/V*) of FAME), as far as not already indicated in the indicated standard. In Table B.1 only those data for requirements from standardized test methods that differ from CEN/TC 19 or ISO/TC 28 precision data are given. These test methods contain precision for regular diesel fuel or the precision for paraffinic diesel fuel determined by a specific RR test programme has shown to give better results. More details are available in the interlaboratory test report [1].

Table B.1 — Precision data from interlaboratory test programme

Property	Test method	Unit	CEN/TC 19 data for paraffinic diesel fuel[a]
Cloud point	EN 23015	°C	$r = 1{,}433\ 9 - 0{,}007\ 1\ X$ $R = 3{,}958\ 5 - 0{,}066\ 1\ X$
CFPP	EN 116	°C	$r = 1{,}728\ 2 - 0{,}017\ X$ $R = 2{,}067\ 7 - 0{,}029\ 1\ X$
	EN 16329	°C	$r = 0{,}986\ 3 - 0{,}040\ 1\ X$ $R = 0{,}949 - 0{,}223\ 1\ X$
Cetane number	EN ISO 5165		$r = -2{,}914\ 8 + 0{,}079\ 4\ X$ $R = -6{,}873\ 3 + 0{,}199\ 8\ X$
	EN 15195[b]		$r = 1{,}045\ 7 + 0{,}001\ 1\ X$ $R = 0{,}652\ 4 + 0{,}017\ 1\ X$
	prEN 16906		$r = 1{,}474\ 2 + 0{,}004\ 3\ X$ $R = -1{,}054\ 2 + 0{,}069\ 2\ X$

[a] Where
  r repeatability (EN ISO 4259);
  R reproducibility (EN ISO 4259);
  X mean of two results being compared.

[b] This precision statement applies only to the correlation equation in EN 15195:2014, Clause 12.

Annex C
(normative)

Details of HPLC procedure

C.1 Warning

WARNING — The use of this procedure can involve hazardous materials, operations and equipment. This Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this standard to take appropriate measures to ensure the safety and health of personnel prior to application of the standard, and fulfil statutory and regulatory requirements for this purpose.

C.2 Scope

This procedure specifies a test method for the determination of the content of mono-aromatic, di-aromatic and tri+-aromatic hydrocarbons in diesel fuels that may contain fatty acid methyl esters (FAME). The polycyclic aromatic hydrocarbons content is calculated from the sum of di-aromatic and tri+-aromatic hydrocarbons and the total content of aromatic compounds is calculated from the sum of the individual aromatic hydrocarbon types.

The usual HPLC techniques as published use a dilution step. The test method is based on EN 12916 [15] and adapted for paraffinic diesel fuels with and without FAME blending up to 7 % (*V/V*) in order to determine very low levels of aromatic components. Therefore, no dilution step of the sample is required or included (see C10.1).

NOTE 1   By convention, the aromatic hydrocarbon types are defined on the basis of their elution characteristics from the specified liquid chromatography column relative to model aromatic compounds. Their quantification is performed using an external calibration with a single aromatic compound for each of them, which may or may not be representative of the aromatics present in the sample. Alternative techniques and test methods may classify and quantify individual aromatic hydrocarbon types differently.

NOTE 2   This method has been assessed via CEN/TC 19 ILS work executed as part of a programme funded by the European Commission. Further details can be obtained via the CEN/TC 19 Secretariat.

C.3 Terms and definitions

For the purposes of this annex, the following terms and definitions apply.

C.3.1
non-aromatic hydrocarbon
compound having a shorter retention time on the specified polar column than the majority of mono-aromatic hydrocarbons

C.3.2
mono-aromatic hydrocarbon
MAH
compound having a longer retention time on the specified polar column than the majority of non-aromatic hydrocarbons, but a shorter retention time than the majority of di-aromatic hydrocarbons

C.3.3
di-aromatic hydrocarbon
DAH
compound having a longer retention time on the specified polar column than the majority of mono-aromatic hydrocarbons, but a shorter retention time than the majority of tri+-aromatic hydrocarbons

C.3.4
tri+-aromatic hydrocarbon
T+AH
compound having a longer retention time on the specified polar column than the majority of di-aromatic hydrocarbons, but a shorter retention time than chrysene

C.3.5
polycyclic aromatic hydrocarbon
POLY-AH
sum of the di-aromatic hydrocarbons and tri+-aromatic hydrocarbons

C.3.6
total aromatic hydrocarbon
sum of the mono-aromatic hydrocarbons, di-aromatic hydrocarbons and tri+-aromatic hydrocarbons

NOTE   Published and unpublished data indicate that the major constituents for each hydrocarbon type include:

a)   non-aromatic hydrocarbons: acyclic and cyclic alkanes (paraffins and naphthenes), mono-alkenes (if present);

b)   MAHs: benzenes, tetralins, indanes and higher naphthenobenzenes (e.g. octahydrophenanthrenes), thiophenes, styrenes, conjugated polyalkenes;

c)   DAHs: naphthalenes, biphenyls, indenes, fluorenes, acenaphthenes, benzothiophenes and dibenzothiophenes;

d)   T+AHs: phenanthrenes, pyrenes, fluoranthenes, chrysenes, triphenylenes, benzanthracenes.

C.4 Principle

A known mass of sample is taken and a fixed volume of this sample is injected into a high performance liquid chromatograph fitted with a polar column. This column has little affinity for non-aromatic hydrocarbons, whilst exhibiting a strong selectivity for aromatic hydrocarbons. As a result of this selectivity, the aromatic hydrocarbons are separated from the non-aromatic hydrocarbons and into distinct bands according to their ring structure, i.e. MAH, DAH and T+AH compounds.

The column is connected to a refractive index detector which detects the components as they elute from the column. The electronic signal from the detector is continually monitored by a data processor. The amplitudes of the signals from the aromatics in the sample are compared with those obtained from calibration standards in order to calculate the mass fraction of MAHs, DAHs and T+AHs in the sample. The sum of the DAHs and T+AHs mass fractions is reported as the mass fraction of POLY-AH, and the sum of the MAHs, DAHs and T+AHs mass fractions is reported as the mass fraction of total aromatic hydrocarbons.

C.5 Reagents and materials

WARNING — Protective gloves should be worn when handling aromatic compounds.

The highest purity reagents and materials available should be used; those required to be of high performance liquid chromatography (HPLC) grade are commercially available from major suppliers.

C.5.1 **Cyclohexane**, of 99 % *(m/m)* minimum purity.

NOTE   Cyclohexane might contain benzene as an impurity.

C.5.2 **Heptane**, HPLC analytical grade, as the mobile phase.

Batch to batch variation of the solvent water content, viscosity, refractive index, and purity may cause unpredictable column behaviour. Drying (for example, by standing over activated molecular sieve type 5A) and filtering the mobile phase may help reducing the effect of trace impurities present in the solvent.

It is recommended practice to de-gas the mobile phase before use; this can be done conveniently online or off-line by helium sparging, vacuum degassing or ultrasonic agitation. A failure to de-gas the mobile phase may lead to negative peaks.

C.5.3 **1-Phenyldodecane**, of 98 % *(m/m)* minimum purity.

C.5.4 **1,2-Dimethylbenzene (*o*-xylene)**, of 98 % *(m/m)* minimum purity.

C.5.5 **Hexamethylbenzene**, of 98 % *(m/m)* minimum purity.

C.5.6 **Naphthalene**, of 98 % *(m/m)* minimum purity.

C.5.7 **Fluorene**, of 98 % *(m/m)* minimum purity.

C.5.8 **Phenanthrene**, of 98 % *(m/m)* minimum purity.

**C.5.9** **Dibenzothiophene**, of 95 % *(m/m)* minimum purity.

C.5.10 **9-Methylanthracene**, of 95 % *(m/m)* minimum purity.

C.5.11 **Chrysene**, of 95 % *(m/m)* minimum purity.

C.5.12 **FAME** (see 5.3).

C.6 Apparatus

C.6.1 **Liquid chromatograph**, consisting of a high performance instrument capable of pumping the mobile phase at flow rates from 0,5 ml/min to 1,5 ml/min, with a precision better than 0,5 % and a pulsation of < 1 % full scale deflection under the test conditions described in C.8.
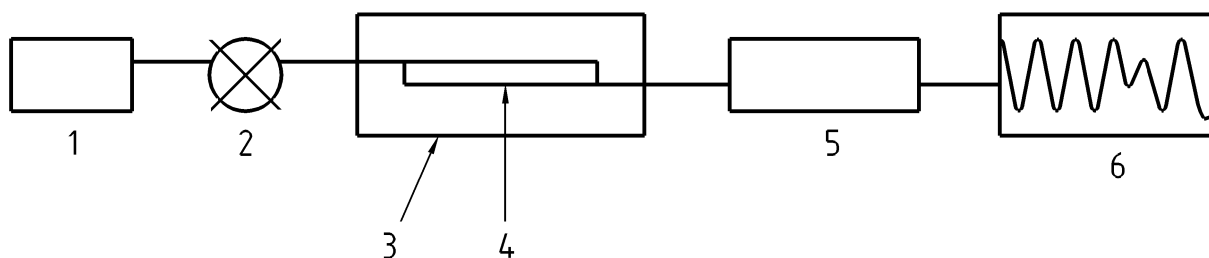
Key

1	pump	4	column
2	injection device	5	refractive index detector
3	oven	6	data acquisition system

Figure C.1 — Diagrammatic representation of liquid chromatograph

C.6.2   Sample injection system, capable of nominally injecting 10 µl of sample solution with a repeatability better than 1 %.

Equal and constant volumes of the calibration and sample solutions are injected into the chromatograph. Both manual and automatic sample injection systems, using either complete or partial filling of the sample loop, can meet these repeatability requirements when used correctly. When using the partial filling mode, it is recommended that the injection volume is less than half the total loop volume. For complete filling of the loop, best results are obtained by overfilling the loop at least six times. The repeatability of the injection system may be checked by comparing peak areas from at least four injections of the system calibration standard (see C.8.6).

Sample and calibration injection volumes different from 10 µl (typically in the range 3 µl to 20 µl) may be used provided they meet the requirements for injection repeatability, refractive index sensitivity and linearity (see C.9.4), and column resolution (see C.8.12).

C.6.3   Sample filter, if required (see C.10.1), consisting of a microfilter of porosity 0,45 µm or less, chemically inert towards hydrocarbon solvents, for the removal of particulate matter from the sample solutions.

NOTE   PTFE filters have been found to be suitable.

C.6.4   Column system, consisting of a stainless steel HPLC column(s) packed with a commercial 3 µm, 5 µm or 10 µm amino-bonded (or amino/cyano-bonded) silica stationary phase meeting the resolution requirements given in C.8.9, C.8.10 and C.8.12.

Column lengths of 150 mm to 300 mm with an internal diameter of 4 mm to 5 mm have been found to be satisfactory. It is good practice to protect the analytical column by using a guard column (e.g. 30 mm by 4,6 mm ID packed with amino-silica) and replacing it regularly.

Batch to batch variations, in terms of resolution and aromatic hydrocarbon type selectivity, have been noted for some commercial stationary phases. Laboratories are advised to test individual columns prior to purchase to ensure they meet the minimum resolution and selectivity requirements of this standard.

New columns will typically be shipped in a solvent different from the mobile phase used in this standard and should be conditioned by purging the column with the mobile phase (heptane). A minimum of two hours conditioning at 1 ml/min is recommended but longer periods of up to two days are sometimes necessary. Alternatively, a reduced flow rate (e.g. 0,25 ml/min) for a minimum of 12 h (e.g. overnight) may be used.

Most of the columns used in daily laboratory practice have exhibited long-term stability and column lifetimes may be two or more years. However, small changes in column performance may go undetected by an operator in the absence of appropriate quality control measures. Laboratories are advised to record, on a regular basis, the column head pressure and calibrant retention times as a simple diagnostic tool for monitoring system and column performance. Participation in inter-laboratory precision monitoring schemes and the regular use of validated and/or internal reference gas oils as part of the test procedure and column evaluation are strongly recommended.

Used columns, which do not meet the requirements of this standard, may be regenerated by flushing the column in backflush mode with a polar solvent (e.g. dichloromethane, 1 ml/min for two hours) and then re-conditioning as for a new column. Before discarding a used column, it is recommended to carefully check all other system components for leaks, dead volumes and/or partial blockage of filters, column frits, tubing, injector needles/seals and valve rotors which may also be contributing to poor column performance.

C.6.5   Temperature control, consisting of either a heating block, or an air-circulating HPLC column oven or an alternative form of temperature control, such as a temperature-controlled laboratory, capable of maintaining a constant temperature in the range 20 °C ± 1 °C to 40 °C ± 1 °C.

The refractive index detector (C.6.6) is sensitive to both sudden and gradual changes in the temperature of the eluent. All necessary precautions should be taken to establish constant temperature conditions throughout the liquid chromatograph system. The temperature should be optimized depending on the stationary phase.

C.6.6   Refractive index detector, capable of being operated over the refractive index range 1,3 to 1,6 and giving a linear response over the calibration range with a suitable output signal for the data system.

If the detector is equipped with a device for independent temperature control, it is recommended that it is set at the same temperature as the column oven.

C.6.7   Computer or computing integrator, compatible with the refractive index detector, having a minimum sampling rate of 1 Hz and capable of peak area and retention time measurements. It shall also have minimum capabilities for post-analysis data processing such as baseline correction and re-integration. The ability to perform automatic peak detection and identification and to calculate sample concentrations from peak area measurements is recommended, but is not essential.

C.6.8   Volumetric flasks, 10 ml and 100 ml capacity, conforming to grade A of EN ISO 1042.

C.6.9   Analytical balance, capable of weighing to the nearest 0,000 1 g.

C.7 Sample handling and storage

Sample handling storage shall be in line with EN ISO 3170 and the following additional instructions because some samples may contain FAME:

— A storage temperature of 19 °C ± 5 °C shall be maintained. If for some reason, the samples have been exposed to temperatures above 25 °C for a long period during storage or in custody (that you are aware of), this shall be reported.

— At least 24 h before a test, the blend shall be placed at ambient temperature.

— When a portion of sample is removed for use in a test, air admitted to the container shall be replaced by nitrogen or helium before closing the container tight.

C.8 Apparatus preparation

C.8.1   Ensure that the equipment and any distribution system of sample is clean and dry before use. Make sure that the equipment for handling or testing the sample is not sensitive to FAME. Polytetrafluoro-ethylene, Viton® and Nylon are materials that should be used.

C.8.2   Set up (see Figure C.1) the liquid chromatograph, sample injection system, column, refractive index detector and computing integrator in accordance with the manufacturer's manuals. If a column oven is used, install the HPLC column in the column oven. Maintain the sample injection system at the same temperature as the sample solution; in most cases this should be at room temperature.

IMPORTANT - Regular maintenance of the liquid chromatograph and its components is important to ensure consistent performance. Leakages and partial blockage of filters, frits, injector needles and valve rotors can produce flow rate inconsistencies and poor injector repeatability.

C.8.3   Adjust the flow rate of the mobile phase to a constant between 0,8 ml/min and 1,2 ml/min and ensure the reference cell of the refractive index detector is full of mobile phase.

C.8.4   In order to minimize instrument drift, the reference cell of the detector should be filled with mobile phase, either by flushing mobile phase through the reference cell immediately prior to the analysis, and then isolating the reference cell to prevent evaporation, or by compensating for

evaporation by supplying a steady flow of mobile phase through the reference cell. The flow should be optimized so that cell mismatch due to drying-out (reference cell) or temperature or pressure gradients (reference or analysis cells, depending the type of detector) are minimized; with some detectors this can be accomplished using a mobile phase flow through the reference cell of one tenth of that through the analysis cell.

C.8.5   Allow the temperature of the column and of the refractive index detector, if it is equipped with temperature control, to stabilize.

C.8.6   Prepare a system calibration standard 1 (SCS1) by weighing, to the nearest 0,000 1 g, 1,0 g ± 0,1 g   cyclohexane   (C.5.1),   0,1 g ± 0,01 g   1-phenyldodecane   (C.5.3),   0,5 g ± 0,05 g 1,2 dimethylbenzene (C.5.4), 0,1 g ± 0,01 g hexamethylbenzene (C.5.5), 0,1 g ± 0,01 g naphthalene (C.5.6), 0,05 g ± 0,005 g dibenzothiophene (C.5.9) and 0,05 g ± 0,005 g 9-methylanthracene (C.5.10) into a 100 ml volumetric flask. Place the flask and its contents into an ultrasonic bath until a visual examination shows that all the components have dissolved into the 1,2 dimethylbenzene/cyclohexane mixture. Remove from the ultrasonic bath and make up to the mark with heptane.

The SCS1 may be kept for at least one year if stored in a tightly stoppered bottle in a cool dark place (for example in a refrigerator).

C.8.7   Prepare a system calibration standard 2 (SCS2) by weighing, to the nearest 0,000 1 g, 0,4 g ± 0,1 g FAME (C.5.12) and 0,04 g ± 0,01 g chrysene (C.5.11) into a 100 ml volumetric flask and making up to the mark with heptane (C.5.2). Keep the solution into an ultrasonic bath at 35 °C.

Ensure the appearance is homogeneous without deposits of chrysene on the bottom.

NOTE   25 min has been found to be a suitable time for all the components to become dissolved.

The SCS2 may be kept for at least one year if stored in a tightly stoppered bottle in a dark place (for example in a refrigerator).

C.8.8   When operating conditions are steady, as indicated by a stable horizontal baseline, inject 10 µl of the SCS1 (C.8.6). Ensure the baseline drift over the period of the HPLC analysis run is less than 1 % of the peak height for cyclohexane.

NOTE   A baseline drift greater than this indicates problems with the temperature control of the column/refractive index detector and/or material eluting from the column.

C.8.9   Ensure the components of the SCS1 are eluted in the order: cyclohexane, phenyldodecane, 1,2 dimethylbenzene, hexamethylbenzene, naphthalene, dibenzothiophene and 9-methylanthracene.

C.8.10 Ensure that baseline separation is obtained between all components of the SCS1 (see Figure C.2).
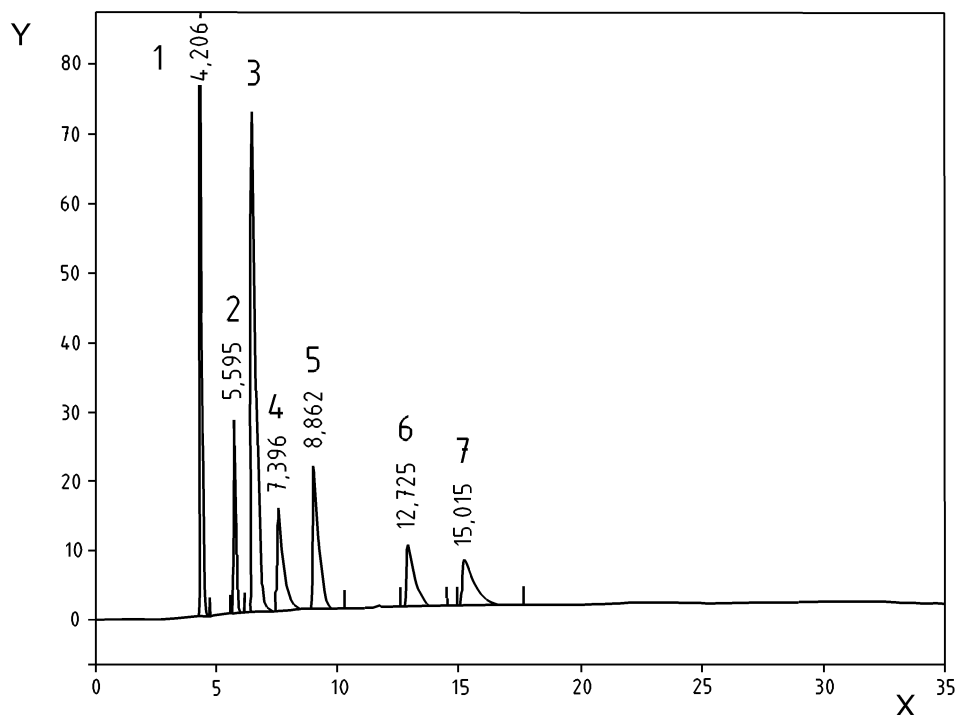
Key

1	cyclohexane	5	naphthalene
2	phenyldodecane	6	dibenzothiophene
3	1,2 dimethylbenzene	7	9-methylanthracene
4	hexamethylbenzene		

Figure C.2— Chromatogram of the system calibration standard SCS1

C.8.11 Measure the retention times of the cyclohexane, phenyldodecane, 1,2 dimethylbenzene, hexamethylbenzene, dibenzothiophene and 9-methylanthracene peaks using the data system.

C.8.12 Ensure that the resolution between cyclohexane and 1,2 dimethylbenzene is between 5,7 and 10 (see C.11.2).

C.8.13 Calculate the cut times using the formulae given in C.11.3.

C.8.14 Ensure the appearance of SCS 2 is homogeneous (C.8.7) and then, inject 10 μl of the SCS2 and check the chrysene peak elutes just before or together with the first peak of FAME.

Ensure the retention time of chrysene peak be higher than the retention time of 9-methylanthracene peak.

Test the column with the SCS2 to verify its performances when starting the method with a new column, after a period of time of inactivity or when samples with FAME should be run.
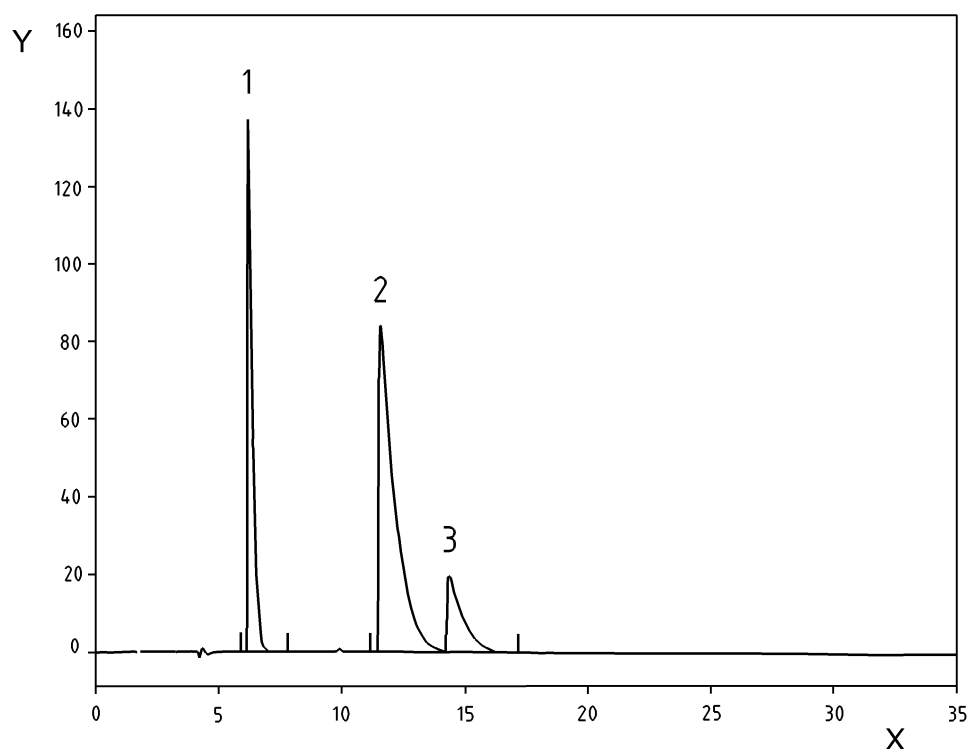
C.9 Calibration

C.9.1 Prepare four calibration standards referenced A, B, C and D at the approximate (but accurately known) concentrations given in Table C.1, by weighing the appropriate materials to the nearest 0,000 1 g into 100 ml volumetric flasks and making up to the mark with heptane (C.5.2).

NOTE   The calibration standards are viable for at least six months if stored in tightly stoppered containers (e.g. 100 ml volumetric flasks) in a cool dark place (for example, in a refrigerator).

Table C.1 — Concentrations of calibration standard components

Calibration standard	1,2 Dimethylbenzene g/100 ml	Fluorene g/100 ml	Phenanthrene g/100 ml
A	4,0	2	0,4
B	1,0	1,0	0,2
C	0,25	0,25	0,05
D	0,05	0,02	0,01

C.9.2   When operating conditions are steady (see C.8.8), inject 10 µl of calibration standard A. Record the chromatogram and measure the peak areas for each aromatic standard (see Figure C.3).

Key

1   1,2 dimethylbenzene

2   fluorene

3   phenanthrene

Figure C.3— Chromatogram of calibration standard A

C.9.3   Repeat C.9.2 for each of the other calibration standards B, C and D. If the peak area for phenanthrene in calibration standard D is too small to be accurately measured, prepare a new calibration standard, D+, with a higher concentration of phenanthrene, e.g. 0,02 g/100 ml, and repeat C.9.2.

C.9.4   Plot concentration in g/100 ml against area counts for each aromatic standard, i.e. 1,2 dimethylbenzene, fluorene and phenanthrene. Calibration plots shall be linear with a correlation coefficient greater than 0,999 and an intercept between ± 0,01 g/100 ml.

A computer or data system may be used to perform these calibrations.

BS EN 15940:2016
EN 15940:2016 (E)

C.10   Procedure

CAUTION   As paraffinic fuels are expected to contain very low or no aromatics, then the diagrams in the procedures of example HPLC traces for typical refinery fuels will not be a good match to the HPLC traces of paraffinic fuels.

C.10.1 Prepare an undiluted fuel sample by filling a 10 ml volumetric flask with the chosen sample. Weigh the sample added to the nearest 0,001 g. Allow the sample to stand for 10 min and, if necessary, use a filter (C.6.3) to remove insoluble material .

C.10.2 When operating conditions are steady (see C.8.8) and identical to those used for obtaining the calibration data (C.9), inject 10 μl of the sample solution (C.10.1) and start data collection.

C.10.3 Identify correctly the MAHs, DAHs and T+AHs:

— MAH are compounds having a retention time between t_b and t_c (see C.11.3);

— DAH are compounds having a retention time between t_c and t_d (see C.11.3);

— T+AH are compounds having a retention time between t_d and t_e (see C.11.3).

C.10.4 Draw a line from just before the beginning of the non-aromatics peak (point A, t_a, Figure C.4) to a point on the chromatogram immediately after the T+AHs (point E, t_e, in Figure C.4), where the signal has returned to its baseline value (i.e. the signal at point A after allowance has been made for any baseline drift, C.8.8).

If DAHs and/or T+AHs are not present in the sample then point E shall be selected at an earlier retention time provided the signal has returned to its baseline value (i.e. the signal at point A after allowance has been made for any baseline drift, C.8.8).

IMPORTANT – Figure C.4 is an example of a diesel type fuel. Paraffinic diesel fuel chromatograms are substantially different from diesel fuels.
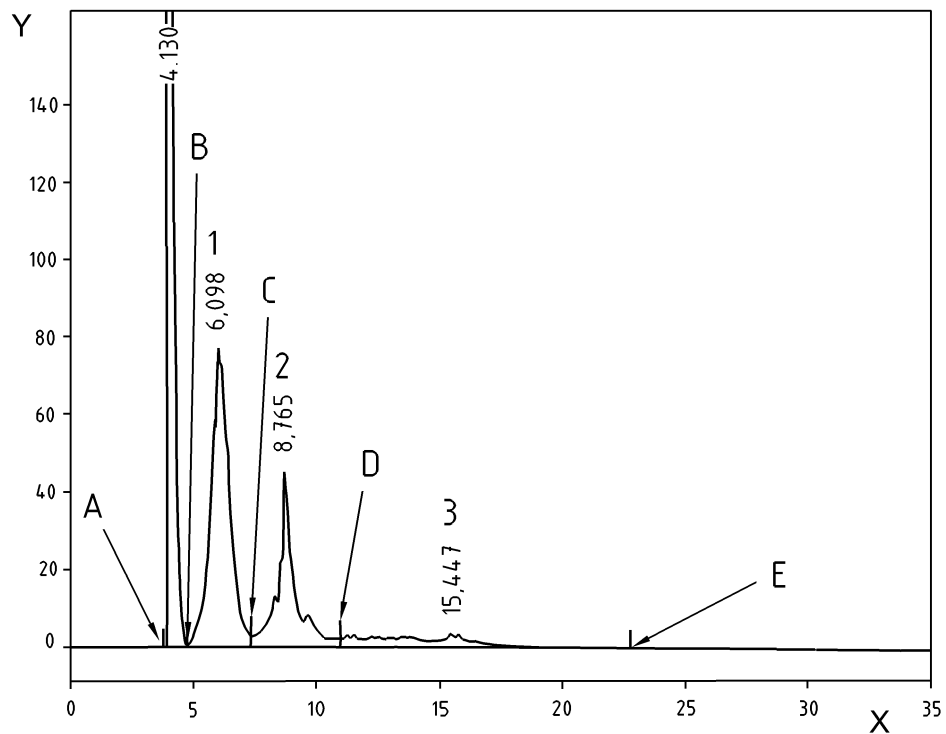
Key

1   mono-aromatic hydrocarbons

2   di-aromatic hydrocarbons

3   tri+-aromatic hydrocarbons

Figure C.4 —Exemplary chromatogram of diesel fuel with the peaks identified showing integration

C.10.5 Drop a vertical line from the valley between the non-aromatics and MAHs (point B, t_b, in Figure C.4) to the baseline (C.10.4). If several valleys are present, use the nearest-one from the time t_b (see C.11.3).

C.10.6 Drop a vertical line from the valley between the MAHs and DAHs (point C, t_c in Figure C.4) to the baseline (C.10.4). If several valleys are present, use the nearest-one from the time t_c (see C.11.3).

C.10.7 Drop a vertical line from the valley between the DAHs and T+AHs (point D, t_d, in Figure C.4) to the baseline (C.10.4). If several valleys are present, use the nearest-one from the time t_d (see C.11.3). If no valley is present, use t_d.

C.10.8 Integrate the area due to MAHs from points B to C.

C.10.9 Integrate the area due to DAHs from points C to D.

C.10.10 Integrate the area due to T+AHs from points D to E.

C.10.11 If the chromatographic data have been processed automatically, check visually that the integration parameters have correctly identified and integrated the peaks.

C.10.12 Practical instructions for paraffinic diesel fuel samples:

Paraffinic diesel fuels have significantly lower levels of total aromatics than in usual diesel fuels. In general samples will be close to zero, and others will be on either side of the total aromatics content limit as in Table 1. There is also the expectation that over 90 % of the total aromatics in a paraffinic fuel will be mono aromatics.

Assuming a theoretical example of a paraffinic fuel with 1 % total aromatics, then it will essentially be a sample of 99 % paraffinic hydrocarbons and 1 % of mono aromatics (as di- and tri-aromatics will be low). This has two consequences when running the samples in an HPLC test:

1) It should be expected that 98 % (*m/m*) or more of the sample is paraffins, and the mono-aromatics peak will be extremely small, in the region of (0 to 2) % (*m/m)*. Experience of those using HPLC of paraffinic fuels report the mono-aromatics peak as very small and also broad.

2) The high level of paraffins in the sample means that they will tend to saturate the HPLC column and it will take a long time for the system to fall back to the base line measurement. A consequence of this is that at times it can be difficult or impossible to achieve a good separation between the paraffins peak and the mono-aromatics peak.

Whether or not good separation is possible between paraffins and mono-aromatics is also a function of the HPLC columns, columns vary from manufacturer to manufacturer, and even batch to batch. Experience of those using HPLC of paraffinic fuels report that sometimes good separation is achievable between the paraffins and the mono-aromatics peak, and sometimes there is a degree of overlap.

Where there is significant overlap of the paraffins and the mono-aromatics peak, it may be necessary to perform an extrapolation to find the true peak areas. The mono-aromatics peak needs to be extrapolated backwards in time to determine the point where the mono-aromatics first started to elute. Similarly, the paraffinic peak needs to be extrapolated forwards in time to determine the point where the paraffins finish eluting.

In some HPLC-systems, another potential consequence of the paraffins saturating the column is that the baseline will not have returned to its true value by the time the mono-aromatics are eluted. This means that the size of the mono-aromatics peak could be overestimated as it is the combination of both mono-aromatics and some of the paraffins still eluting. This might necessitate the use of a pseudo-baseline in order to remove the contribution of these paraffins to the mono-aromatics peak.

C.11   Calculation

C.11.1 Reference time

Retention times to be measured from the chromatogram of SCS1 (C.8.9) are:

— the retention time of cyclohexane (t_1), in seconds;

— the retention time of phenyldodecane (t_2), in seconds;

— the retention time of 1,2 dimethylbenzene (t_3), in seconds;

— the retention time of hexamethylbenzene (t_4), in seconds;

— the retention time of dibenzothiophene (t_6), in seconds;

— the retention time of 9-methylanthracene (t_7), in seconds.

C.11.2 Column resolution

Calculate the resolution, R, between cyclohexane and 1,2 dimethylbenzene using the following formula:

$$R = \frac{2\,(t_3 - t_1)}{1,699\,(y_1 + y_3)}$$

(C.1)

where

y_1 is the width at half-height of the cyclohexane peak, in seconds;

y_3 is the width at half-height of the 1,2 dimethylbenzene peak, in seconds.

C.11.3 Cut times

Determine the cut times, t_a, t_b, t_c, t_d, and t_e, in seconds, using the following:

— t_a is a point on the baseline just before the non-aromatic peak;

— t_b is $0,5(t_1 + t_2)$;

— t_c is t_4;

— t_d is $t_6 + 0,4(t_7 - t_6)$;

— t_e is a point on the baseline when all TAH+ have been eluted.

C.11.4 Aromatic hydrocarbons type content

Determine the content, C, as mass fraction, of MAHs, DAHs and T+AHs, either directly from the data system, or calculated using the following formula:

$$C = \frac{\left[(A \times S) + I\right] \times V}{M}$$

(C.2)

where

A is the MAH or DAH or T+AH peak area of the sample;

S is the slope of the MAH or DAH or T+AH calibration plot (concentration in g/100 ml versus peak area);

I is the intercept of the MAH or DAH or T+AH calibration plot;

M is the mass of sample taken, in grams (C.10.1);

V is the volume of sample solution, in millilitres (C.10.1).

C.11.5 Total aromatic hydrocarbons content

Calculate the total aromatic hydrocarbons content of the sample, as a mass fraction, from the sum of the individual hydrocarbons types (i.e. MAHs, DAHs and T+AHs).

C.12   Test precision

C.12.1 General

The precision given was derived from statistical analysis by EN ISO 4259 of the results of interlaboratory testing of a matrix of fuels including three types of paraffinic diesel fuels containing up to 7 %(V/V) of FAME and levels of up to 2,0 % (m/m) of total aromatics content.

C.12.2 Repeatability, *r*

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

$$r = 0{,}039\ 1\ X + 0{,}077\ 2 \qquad\qquad\qquad\text{(C.3)}$$

where

   X is the average of the two results being compared, in % (m/m).

C.12.3 Reproducibility, *R*

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the: following value in only one case in twenty.

$$R = 0{,}171\ 3\ X + 0{,}346\ 9 \qquad\qquad\qquad\text{(C.4)}$$

where

   X is the average of the two results being compared, in % (m/m).

C.13   Test report

Report the total aromatic hydrocarbons content to the nearest 0,01 % (m/m).

Report as well:

a)   identification of the product under test;

b)   type and identification of the apparatus used;

c)   reference to this procedure (i.e. EN 15940:2016, Annex C);

d)   deviation from the procedure described;

e)   date of the test.

Annex D
(normative)

Measurements and constants for paraffinic fuel products and components

D.1 General

Product densities and volumes of petroleum products vary with temperature. During product transfer, these measurements are normally not carried out at the requested reference temperature (15 °C), but mostly at elevated temperatures. It is therefore necessary to apply prescribed calculation procedures to transform densities or volumes from a given temperature, t, to the prescribed reference temperature, t_{ref}, or vice versa for obtaining results at other temperatures of interest. These calculation routines are part of the so called "petroleum metering" or "petroleum measurement" as defined and described in a number of international standards (see [16] to [22]).

For new products in the market, it is advisable to check if the product constants from earlier publications may be used, or if, for a new product, the constants need updating to allow density/temperature conversions with the requested precision. It has been shown that the latter is the case for the paraffinic diesel product family.

For regular products already in the market, the international standards mentioned above contain constants like thermal expansion coefficients, α15, and extensively tested conversion calculation routines which are in use since many decades. For user's convenience the essential "recipe" for fuels is described in form of a short abstract in D.2. D.3 gives data developed for GTL and XTL paraffinic diesel fuels plus information which constants should be used for this product family for density/volume temperature conversion, at least until these constant have found their way into the mentioned international standards..

D.2 Short abstract of density / temperature conversion formulae and constants

Density / temperature (and in parallel volume / temperature) conversions for fuels are performed using the following set of formulae and constants:

$$VCF = D(t) / D(t_{ref}) = V(t_{ref}) / V(t) \tag{D.1}$$

$$VCF = \exp\left(- \alpha15 * (t - t_{ref}) * (1 + 0{,}8 * \alpha15 * (t - t_{ref}))\right) \tag{D.2}$$

$$VCF = (1 - \alpha15 * (t - t_{ref})) \tag{D.3}$$

$$\alpha15 = K0 / [\, D(t_{ref}) \,]^2 + K1 / D(t_{ref}) \tag{D.4}$$

where

$D(t_{ref})$ is the density at reference temperature, almost always set at 15,0 °C;

NOTE 1   Some countries set the reference temperature to 60 °F, which is somewhat different from 15,0 °C (i.e. 60 °F = 15,666 6 °C, while 15,0 °C = 59 °F)

$D(t)$ is the density at the measured (or desired) temperature;

VCF is the product- and temperature dependent volume correction factor;

α15 is the product-specific thermal expansion coefficient, calculated by regression of at min 10 accurately measured density / temperature pairs over the

temperature range of interest (for fuels normally – 20 °C to 50 °C);

K0, K1   are calculation constants from regression, see [16], over multiple, representative samples in order to bundle calculations for a specific product family, often called "group" or "product family" constants.

While Formula (D.2) represents the standardized calculation normally used for fuels, Formula (D.3) gives a simplification obtained from a Taylor series expansion which can also be used, but only when the density/temperature function has been shown to be sufficiently linear and only when the difference between the results from calculated and measured temperatures are still very small.

NOTE 2   The EU Machinery Directive for instance requires that the measurement uncertainty, expressed here as the relative difference between a measured and calculated density (or volume) is smaller than 0,2 %.

Formula (D.4) may be used directly when the reference density is known, i.e. when density is calculated for any other desired temperature. For the reverse case (i.e. calculation of reference density from a density at measured temperature), Formula (D.3) shall be used in an iteration, which usually does not take more than 6 to 7 cycles.

D.3 Measurements and constants for paraffinic diesel fuel products and components

A set of representative paraffinic diesel fuel samples has been measured by the German metrological office, PTB. These values are shown in Table D.1. The constants proposed for the product family specified in this standard are developed from this table, and a check for the expected max. calculation error (for a target temperature of 50 °C) is also given in Table D.2.

Table D.1 —Density - Temperature measurements for paraffinic diesel fuel samples in kg/m^3

temperature t, °C	Name (t-15)	GTL A	GTL B	GTL C	GTL D	GTL E	GTL F	GTL G	GTL H (Naphtha)
50,00	35,00	784,601	794,425	806,058	763,200	752,716	728,575	723,929	671,721
45,00	30,00	787,846	797,594	809,144	766,599	756,196	732,266	727,616	676,017
40,00	25,00	791,086	800,769	812,239	769,997	759,678	735,945	731,292	680,286
35,00	20,00	794,333	803,949	815,337	773,394	763,153	739,619	734,961	684,529
30,00	15,00	797,580	807,125	818,445	776,787	766,626	743,284	738,624	688,740
25,00	10,00	800,828	810,306	821,555	780,179	770,095	746,941	742,280	692,931
20,00	5,00	804,078	813,494	824,672	783,570	773,560	750,593	745,933	697,101
15,00	0,00	807,335	816,686	827,790	786,962	777,027	754,238	749,582	701,249
10,00	-5,00	810,592	819,884	830,910	790,356	780,491	757,880	753,229	705,377
5,00	-10,00	813,857	823,090	834,018	793,750	783,956	761,518	756,877	709,488
0,00	-15,00	817,125	826,303	837,139	797,147	787,421	765,154	760,525	713,583
-5,00	-20,00	820,401	829,517	840,304	800,543	790,887	768,786	764,175	717,661
-10,00	-25,00	823,684	832,725	843,472	803,946	794,356	772,417	767,834	721,725
-15,00	-30,00	825,974	835,926	846,645	807,357	797,826	776,049	771,501	725,777
-20,00	-35,00	830,261	839,191	849,812	812,618	801,300	779,697	775,183	729,814
-25,00	-40,00	833,536	842,505	853,407	817,323	806,052	783,311	**779,118**	733,841

Table D.2 shows data according to regression from Formula (D.3), including calculation of group constants from regression according to Formula (D.4) and also including a prediction check via calculation of density at 50 °C.

Table D.2 —Calculation of density (15°C) and α15 for paraffinic diesel fuel samples

	GTL A	GTL B	BTL C	GTL D	GTL E	GTL F	GTL G	GTL H
Density 15°C, meas.	807,335	816,686	827,790	786,962	777,027	754,238	749,582	701,249
Density 15°C, calc	807,322	816,743	827,866	787,233	777,086	754,194	749,585	701,047
Calculation error	-0,002%	0,007%	0,009%	0,034%	0,008%	-0,006%	0,000%	-0,029%
alpha15*1000, calc	0,804112	0,783751	0,757696	0,889609	0,900041	0,967141	0,977387	1,180319
Group constant K_0	902,5633							
Group constant K_1	-0,4630	--->>>	alpha15 = 902,5633 / D15 / D15 - 0,4630 / D15					
(R-squared)	0,9974							
Calculation / prediction check:								
Density 50 °C, meas.	784,601	794,425	806,058	763,200	752,716	728,575	723,929	671,721
Density 50°C, calc	784,601	794,339	805,912	762,721	752,606	728,665	723,943	672,086
Calculation error	0,000%	0,011%	0,018%	0,063%	0,015%	-0,012%	-0,002%	-0,054%

NOTE 1   It is noted that the maximum calculation errors for a temperature of 50 °C (i.e. for a temperature difference of 35 K) are well below the allowed maximum error of 0,2 %.

As a further check, duplicate density measurements for three representative paraffinic diesel fuel (XTL) products (see Table D.3) have been executed which are used here as an additional check how well conversion calculations using the constants for "GTL" from Table D.2 do also apply to new XTL samples which have not been included in the modelling. The data in Table D.3 are obtained using K0, K1 from Table D.2 (i.e. from the other sample set) according to Formula (D.3) and also including prediction check via calculation of density at 50 °C.

The results show that such extension to other samples of the same product family can be done without problems for a range of densities at 15 °C from about 700 kg/m³ to 830 kg/m³ in a temperature range of −20 °C up to 50 °C.

Table D.3 —Calculation of density (15 °C); and α15 for additional paraffinic diesel fuel samples

	XTL3-B0	XTL3-B0	XTL2-B3	XTL2-B3	XTL3-B7	XTL3-B7
	Repeat A	Repeat B	Repeat A	Repeat B	Repeat A	Repeat B
Density 15°C, meas.	777,979	777,981	781,753	781,765	785,199	785,204
Density 15°C, calc	777,994	778,689	782,767	782,091	785,204	785,212
Calculation error	-0,002%	-0,091%	-0,130%	-0,042%	-0,001%	-0,001%
alpha15*1000	0,896043	0,893913	0,881542	0,883579	0,874249	0,874224
Calculation / prediction check:						
Density 50°C, meas.	753,756	753,766	757,433	757,443	760,902	760,915
Density 50°C, calc	753,595	754,326	758,616	757,904	761,178	761,187
Calculation error	0,021%	-0,074%	-0,156%	-0,061%	-0,036%	-0,036%

NOTE 2   Measurement of XTL3-B0, repeat B and XTL3-B3, repeat A had some difficulties with the determination at low temperatures (below 0 °C). This is reflected and visualized by the calculation errors. As a comparison, however, see the results of the corresponding second duplicate measurement.

D.4 Conclusions

Constants K0 and K1 from Table D.2 may be used for samples from the paraffinic diesel fuel product family specified in this European Standard to execute density/temperature and volume/temperature conversions with sufficient precision following the standardized calculations given in D.2.

As a summary, Formulae (D.5) to (D.9) should be used for density / temperature conversion without exceeding the required measurement error (max. 0,2 %). Volume conversions may be done accordingly using the definition "density = mass / volume".

$$K0 = 902,533 \tag{D.5}$$

$$K1 = -0,463\,0 \tag{D.6}$$

$$α15 = (K0 / [\,D(15°C)^2\,]) + (K1 / D(15°C)) \tag{D.7}$$

$$D(t) = D(15°C) * \exp[-α15 * (t - 15) * (1 + 0,8 * α15 * (t - 15)] \tag{D.8}$$

or

$$D(t) = D(15°C) * (1 - α15 * (t - 15)) \tag{D.9}$$

Bibliography

[1]   *Assessment for checking the applicability of several petroleum related test methods for paraffinic diesel fuels and components*, CEN/TC 19 RRT report number 400-430, 2013, available from CEN/TC 19 Secretariat, NEN, the Netherlands, energy@nen.nl

[2]   CEN/TR 16389, *Automotive fuels — Paraffinic diesel fuel and blends — Background to the parameters required and their respective limits and determination*

[3]   *Directive 2009/28/EC of the European Parliament and of the Council of 23 April 2009 on the promotion of the use of energy from renewable sources and amending and subsequently repealing Directives 2001/77/EC and 2003/30/EC*

[4]   EN 14331, *Liquid petroleum products — Separation and characterisation of fatty acid methyl esters (FAME) from middle distillates — Liquid chromatography (LC)/gas chromatography (GC) method*

[5]   *Directive 98/70/EC of the European Parliament and of the Council of 13 October 1998 relating to the quality of petrol and diesel fuels and amending Council Directive 93/12/EEC*

[6]   *Directive 2003/17/EC of the European Parliament and of the Council of 3 March 2003 amending Directive 98/70/EC relating to the quality of petrol and diesel fuels and amending Council Directive 93/12/EEC*

[7]   *Directive 2009/30/EC of the European Parliament and of the Council of 23 April 2009 amending Directive 98/70/EC relating to the quality of petrol and diesel fuels and amending Council Directive 93/12/EEC*

[8]   *Commission Directive 2014/77/EU of 10 June 2014 amending Annexes I and II of Directive 98/70/EC of the European Parliament and of the Council relating to the quality of petrol and diesel fuels*

[9]   EN 16715, *Liquid petroleum products — Determination of ignition delay and derived cetane number (DCN) of middle distillate fuels — Ignition delay and combustion delay determination using a constant volume combustion chamber with direct fuel injection*

[10]   ASTM D6890, *Standard Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber*

[11]   CEN/TR 15367-1, *Petroleum products — Guidelines for good housekeeping – Part 1: Automotive diesel fuels*

[12]   ASTM D6078, *Standard Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE)*

[13]   ASTM D975 — 15b, *Standard Specification for Diesel Fuel Oils*

[14]   *Diesel Fuel Lubricity: On the Path to Specifications*, Nikanjam, M., SAE Technical Paper, 1999-01-1479

[15]   EN 12916, *Petroleum products — Determination of aromatic hydrocarbon types in middle distillates — High performance liquid chromatography method with refractive index detection*

[16]   ISO 91-1, *Petroleum measurement tables — Part 1: Tables based on reference temperatures of 15 degrees C and 60 degrees F*

[17]   ASTM D1250, *Standard Guide for Use of the Petroleum Measurement Tables*

[18]   API MPMS Chapter 11; *Physical Properties Data (Volume Correction Factors)*

[19]   OIML R 63, *Petroleum measurement tables*

[20]   OIML R120, *Standard capacity measures for testing measuring systems for liquids other than water*

[21]   OIML R117; *Dynamic measuring systems for liquids other than water*

[22]   *PTB – Anforderungen an Messanlagen für Flüssigkeiten*, PTB-A 5

[23]   prEN 16942:2016, *Fuels — Identification of vehicle compatibility — Graphical expression for consumer information*

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

National Annex NA
(normative)

Sampling requirements

NA.1 Introduction

This National Annex gives the sampling requirements to comply with Clause 3 of this standard.

NA.2 Normative references

The following referenced documents are indispensable for the application of this National Annex. For undated references, the latest edition of the referenced document (including any amendments) applies.

BS EN ISO 3170, *Petroleum liquids — Manual sampling* (dual numbered as BS 2000-475).

BS EN ISO 3171, *Petroleum liquids — Automatic pipeline sampling* (dual numbered as BS 2000-476).

BS EN 14275, *Automotive fuels — Assessment of petrol and diesel fuel quality — Sampling from retail site pumps and commercial site fuel dispensers* (dual numbered as BS 2000-509).

NA.3 Sampling

NA.3.1 Sampling from storage tanks

All sampling from storage tanks shall be carried out in accordance with the relevant procedures given in BS EN ISO 3170/BS 2000-475.

NA.3.2 Sampling from pipelines

All sampling from pipelines shall be carried out in accordance with the relevant procedures given in either BS EN ISO 3170/BS 2000-475 or BS EN ISO 3171/BS 2000-476.

NA.3.3 Sampling from commercial site fuel dispensers

All sampling from commercial site fuel dispensers shall be carried out in accordance with the relevant procedures given in BS EN 14275/BS 2000-509.

NA.3.4 Labelling and transport

Full and legible information relating to the source of the sample shall be attached to the can in such a manner that it shall not easily become detached.

NOTE 1   If required, the sample may be sealed and labelled to maintain its legal integrity.

NOTE 2   Attention is drawn to:

— the European Agreement Concerning the International Carriage of Dangerous Goods by Road (ADR) as amended, particularly with regard to the provisions for Limited Quantities;

— the Carriage of Dangerous Goods (Classification, Packaging and Use of Transportable Pressure Receptacles) Regulations 2004 (as amended);

— the Carriage of Dangerous Goods by Road Regulations 1996 (as amended), with reference to the labelling and transport of samples.

National Annex NB
(normative)

Pump marking requirements

NB.1 Introduction

This National Annex gives the pump marking requirements to comply with Clause 4 and Subclause 5.2.2 of this standard.

NB.2 Requirements

The following information shall be marked on each dispensing pump or container used for delivering paraffinic diesel fuel (conforming to the requirements of BS EN 15940:2016) into the consuming vehicle.

a)   The name or mark of the supplier or vendor of the diesel fuel.

b)   The designations 'Paraffinic Diesel Fuel BS EN 15940:2016' displayed as shown in Figure NB.1. The minimum dimensions of this mark shall be as shown in Figure NB.1 and the colour used for the design and lettering shall be in clear contrast to the background (see Notes 2 and 3).
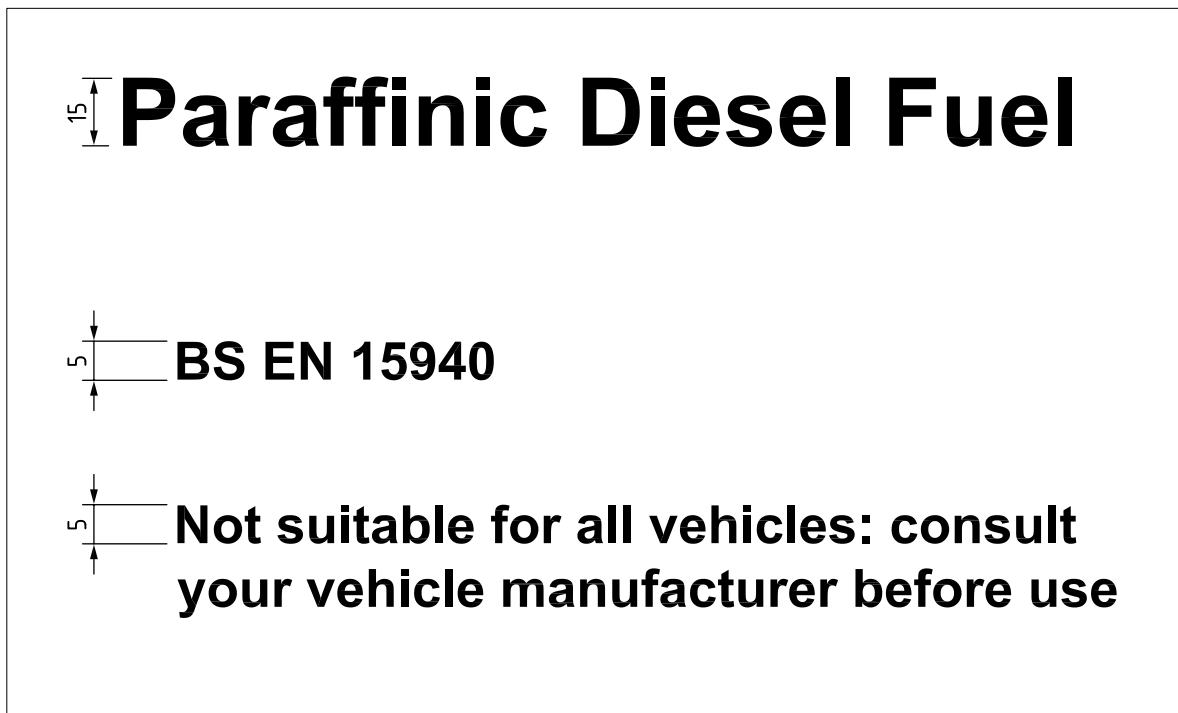
Dimensions are in millimetres

Figure NB.1 — Form of marking for paraffinic diesel fuel

The mark shall be in a position where it can be seen by the person dispensing fuel.

In accordance with Clause 4 and Subclause 5.2.2 the words 'CONTAINS METALLIC ADDITIVES' shall be displayed at any point where paraffinic diesel fuel containing metallic additives is made available to

consumers. The form and minimum dimensions are shown in Figure NB.2. Where this label is necessary it is not necessary for this mark to form an integral part of the label shown in Figure NB.1, although it shall also be in a position where it can be seen by the person dispensing fuel.

Dimensions are in millimetres

Figure NB.2 — Form of marking for paraffinic diesel fuel containing metallic additives

The dispensing nozzle shall be predominantly colour-coded black, forward of the grip.

NOTE 1   Marking BS EN 15940 on or in relation to a product represents a manufacturer's declaration of conformity, i.e. a claim by the manufacturer that the product meets the requirements of the standard. The accuracy of the claim is solely the claimant's responsibility. Such a declaration is not to be confused with third-party certification of conformity, which may also be desirable.

NOTE 2   The minimum dimensions of the marks given in Figures NB.1 and NB.2 have been selected to enable their application to pumps of the smallest anticipated dimensions. It is strongly recommended that, in the interests of visibility to the motorist, larger dimensions be used wherever possible.

NOTE 3   It is recommended that the prominent colour of the lettering should be black. The preferred colour is colour number 642, 'Night', of BS 381C:1996.

NOTE 4   Attention is drawn to the relevant marking provisions of the Measuring Instruments legislation: SI 2006 No. 1266 and SI 2006 No. 2234.

National Annex NC
(normative)

Climate-dependent requirements

NC.1 Introduction

This National Annex gives the climate-dependent requirements to comply with Subclause 5.7 of this standard.

NC.2 Requirements

For the purposes of this British Standard, the following grades shall apply:

	Summer	Winter
For delivery from refineries and imports into the United Kingdom	16 March to 15 October inclusive CFPP Grade C	16 October to 15 March inclusive CFPP Grade E
For delivery from terminals (including refinery-based terminals)	16 March to 31 October inclusive CFPP Grade C	01 November to 15 March inclusive CFPP Grade E
For sales from the dispenser	16 March to 15 November inclusive CFPP Grade C	16 November to 15 March inclusive CFPP Grade E

NOTE 1   The date for the change from summer grade to winter grade will be reviewed in the light of experience.

NOTE 2   At the seasonal change-over points, product may be deemed to conform to BS EN 15940:2016, providing it

i) conforms to the previous season's quality, and

ii) is stored in a tank to which fewer than three deliveries have been made in the four weeks prior to the relevant sales date in BS EN 15940:2016.

NOTE 3   A 'refinery-based terminal' is a facility located within a refinery boundary which is used to load fuel onto road tanker vehicles for delivery to retail service stations and other end-consumers.

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