BS EN 16715:2015

BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of EN 16715:2015.

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.

ISBN 978 0 580 85393 7

ICS 75.160.20

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 August 2015.

Amendments issued since publication

Date	Text affected

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16715

August 2015

ICS 75.160.20

English Version

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

Produits pétroliers liquides - Détermination du délai d'inflammation et de l'indice de cétane dérivé (ICD) des distillats moyens - Détermination du délai d'inflammation et de combustion en utilisant une chambre à volume constant avec injection directe de gazole

Flüssige Mineralölerzeugnisse - Bestimmung des Zündverzugs und der abgeleiteten Cetanzahl (ACZ) von Kraftstoffen aus Mitteldestillaten - Bestimmung des Zündverzugs und des Verbrennungsverzugs in einer Verbrennungskammer mit konstantem Volumen und direkter Kraftstoffeinspritzung

This European Standard was approved by CEN on 20 June 2015.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of 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 United Kingdom.

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Page

European foreword 3

Introduction 4

1 Scope 5

2 Normative references 5

3 Terms and definitions 6

4 Principle 7

5 Reagents and materials 7

6 Apparatus 8

7 Sampling 8

8 Apparatus assembly and installation 10

9 Preparation of apparatus 10
9.1 System start-up and warm-up 10
9.2 Standard operating and test conditions 10

10 Calibration, verification and quality control 11
10.1 General 11
10.2 Calibration 11
10.3 Apparatus verification 12
10.4 Quality control (QC) 12

11 Test procedure 13

12 Calculation 13

13 Expression of results 13

14 Precision 14
14.1 General 14
14.2 Repeatability 14
14.3 Reproducibility 14

15 Test report 15

Annex A (normative) **Combustion analyser description** 16

A.1 General 16

A.2 Apparatus description and assembly 16

A.3 Control and data acquisition 18

Annex B (normative) **Operational details in support to the standard test procedure** 19

B.1 General 19

B.2 Cleaning procedure to prepare fuel system for calibration 19

B.3 Automatic calibration procedure 19

B.4 Test sequence 20

B.5 Unit shutdown 22

Bibliography 23

European foreword

This document (EN 16715:2015) 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 February 2016 and conflicting national standards shall be withdrawn at the latest by February 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.

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 European Standard is derived from joint standardization work within the Energy Institute and ASTM International. It is based on and technically equivalent with ASTM D7668 [1].

The described method is an alternative quantitative determination of the cetane number of middle distillate fuels intended for use in compression ignition engines. A correlation study between this method and EN ISO 5165:1998 [2] has been done and the results of this are incorporated in this European Standard. Research Report RR: D02-1771 [3].

The basis of this method is the derived cetane number (DCN) correlation equation as given in Clause 12. The on-going validation of the equation is monitored and evaluated through the existing American and European fuel exchange programs. The validation data will be reviewed by CEN/TC 19 with a frequency of at least every two years. As a result of the review, CEN/TC 19 may make the decision to, if necessary, modify the existing equation/correlation or develop a new one. As part of this review, the sample types will be examined, and if certain types are underrepresented, further steps may be taken to evaluate how they perform.

The ignition delay (ID) and combustion delay (CD) values and the DCN value determined by this test method can provide a measure of the ignition characteristics of diesel fuel oil used in compression ignition engines. This test is for use by engine manufacturers, petroleum refiners and marketers, and in commerce as a specification aid to relate or match fuels and engines. This test is also applicable to non-conventional diesel fuels.

For the moment the basics of one type of apparatus are described. Once more correlation data on different types of derived cetane number testing equipment is available, CEN/TC 19 will consider revising this European Standard.

1 Scope

This European Standard specifies a test method for the quantitative determination of ignition and combustion delays of middle distillate fuels intended for use in compression ignition engines. The method utilizes a constant volume combustion chamber with direct fuel injection into heated, compressed synthetic air. A dynamic pressure wave is produced from the combustion of the product under test. An equation is given to calculate the derived cetane number (DCN) from the ignition and combustion delays determined from the dynamic pressure curve.

This European Standard is applicable to middle distillate fuels, fatty acid methyl esters (FAME) and blends of diesel fuels and FAME. The method is also applicable to middle distillate fuels of non-petroleum origin, oil-sands based fuels, blends of fuel containing biodiesel material, diesel fuel oils containing cetane number improver additives and low-sulfur diesel fuel oils. However, users applying this standard especially to unconventional distillate fuels are warned that the relationship between derived cetane number and combustion behaviour in real engines is not yet fully understood.

This European Standard covers the ignition delay range from 2,47 ms to 4,09 ms and combustion delay from 3,71 ms to 6,74 ms (67 DCN to 39 DCN).

NOTE 1 The combustion analyser can measure shorter or longer ignition and combustion delays, but precision is not known.

NOTE 2 There is no information about how DCNs outside the 67 to 39 range compare to EN ISO 5165.

NOTE 3 For the purpose of this European Standard, the expression “% (*V/V*)” is used to represent the volume fraction (φ), and “% (*m/m*)” the mass fraction (ω).

WARNING — The use of this standard 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.

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

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

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

ISO 1998-2, *Petroleum industry - Terminology - Part 2: Properties and tests*

IP 537, *Determination of the purity of Derived Cetane Number reference materials - Gas chromatography method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1998-2 and the following apply.

3.1
cetane number
CN
measure of the ignition performance of a diesel fuel in a standardized engine test on a scale defined by reference fuels

Note 1 to entry: It is expressed as the percentage by volume of hexadecane (cetane) in a reference blend having the same ignition delay as the fuel for analysis. The higher the cetane number, the shorter the ignition delay.

3.2
ignition delay
ID
period of time, in milliseconds (ms), between the start of fuel injection and the start of combustion

Note 1 to entry: In the context of this test method, the start of fuel injection is interpreted as the rise in the electronic signal that opens the injector and the combustion start is interpreted as the first increase of the chamber pressure during the combustion cycle, as measured by a pressure sensor in the combustion chamber.

3.3
combustion delay
CD
period of time, in milliseconds (ms), between the start of fuel injection and mid-point of the combustion pressure curve

Note 1 to entry: In the context of this test method, the start of fuel injection is interpreted as the rise in the electronic signal that opens the injector and the combustion pressure curve mid-point is interpreted as the part of the pressure curve midway between the initial chamber pressure and the maximum pressure generated during the combustion cycle, as measured by a pressure sensor in the combustion chamber. The combustion delay CD measures the time between the injection of the sample and phase of combustion controlled by the diffusive mixing of the air and fuel.

3.4
derived cetane number
DCN
number calculated by using an equation that correlates a combustion analyser’s ignition and combustion delays to the cetane number

3.5
accepted reference value
ARV
value agreed upon as a reference for comparison

Note 1 to entry: The value is derived as (1) a theoretical or established value, based in scientific principles, (2) an assigned value, based on experimental work of some national or international organization, or (3) a consensus value, based on collaborative experimental work under the auspices of a scientific or engineering group.

3.6
quality control sample
QC sample
stable and homogenous material(s) similar in nature to the materials under test, properly stored to ensure integrity, and available in sufficient quantity for repeated long-term testing

3.7
calibration reference fluid
stable and homogenous fluid used to calibrate the performance of the combustion analyser

3.8
verification reference fluid
stable and homogenous fluid used to verify the performance of the combustion analyser

4 Principle

A test portion of the material under test is injected into a temperature and pressure controlled, constant volume combustion chamber, which has previously been charged with synthetic air of a specified quality. Each injection produces a compression ignition combustion cycle detected using a pressure sensor. The ignition delay and combustion delay are measured from the rise of the electronic signal that activates the injector solenoid to two specific points along the combustion pressure wave produced by the combustion cycle.

A complete sequence comprises 5 preliminary injection cycles and 15 subsequent injection cycles used for the sample analysis. The ID and CD measurements for the last 15 injection cycles are statistically reviewed and the outlying ID's and CD's are eliminated using Peirce's Criterion [4]. The remaining ID's and CD's are averaged to produce the ID and CD results. An equation is given to calculate the derived cetane number (DCN) from the ignition and combustion delays determined from the dynamic pressure curve. The DCN obtained by this procedure is an estimate of the cetane number (CN) obtained from the conventional large-scale engine test EN ISO 5165 [2].

5 Reagents and materials

5.1 Calibration reference fluid, 40:60 mixture by weight of hexadecane and 2,2,4,4,6,8,8-heptamethylnonane, respectively, measured with an accuracy of 0,01 percent. For peroxide-free material the assigned ID_{ARV} is 2,96 ms and the assigned CD_{ARV} is 4,90 ms.

5.1.1 Hexadecane, minimum purity of 99,0 % (*m/m*).

5.1.2 2,2,4,4,6,8,8-Heptamethylnonane, minimum purity of 98,0 % (*m/m*).

IMPORTANT — Hydrocarbons can form peroxides and other free radicals forming contaminants that can influence the ID and CD. Experience has found some 40:60 blends of hexadecane and 2,2,4,4,6,8,8-heptamethylnonane meeting the purity specification can contain peroxides and other free radically form contaminants. Typically, the peroxides and other free radically formed contaminants can be removed from the 40:60 mixture of hexadecane and 2,2,4,4,6,8,8-heptamethylnonane by subjecting the blend to activated 4 Å molecular sieves.

5.2 Verification reference fluid, methylcyclohexane (MCH) of a purity of minimum 99,0 % (*m/m*) to be used as the designated 11,0 ms ignition delay (ID_{ARV}) and the designated 17,0 ms combustion delay (CD_{ARV}) assigned accepted reference value material.

If the initial purity is not known the purity shall be checked in accordance with IP 537.

Even if the verification reference fluid meets the purity specification, it may not meet the Ignition and Combustion delay requirements (see Table 2). It is recommended to either pass the suspect MCH through a filter column to remove peroxide based impurities or to test a bottle of MCH that has been shown to meet the ID and CD requirements. It is recommended that each bottle of MCH is tested prior to its use as a verification reference fluid to confirm it is of acceptable quality.

5.3 Quality control sample, stable and homogenous distillate fuel, similar in nature to the materials under test (see 3.6).

5.4 Combustion charge air, a compressed synthetic air mixture containing (20,0 ± 0,5) % (V/V) oxygen with the balance nitrogen, less than 0,003 % (V/V) hydrocarbons, and less than 0,025 % (V/V) water. It is recommended that a quality control test be performed after an air cylinder has been changed.

NOTE Oxygen content of combustion charge air can vary between batches (cylinders). Significant variation will lead to changes in ignition and combustion delay (higher oxygen content leads to shorter ignition and combustion delays).

5.5 Heptane, (n-Heptane) with a minimum purity of 99,5 % (*m/m*).

5.6 Water, unless otherwise specified, meeting the requirements of grade 3 of EN ISO 3696.

5.7 Coolant system fluid, 50:50 volumetric mixture of commercial grade ethylene glycol-type radiator antifreeze with water (5.6).

5.8 Compressed nitrogen, of minimum purity 99,9 % (*V/V*), capable of delivering a pressure of (0,6 to 1,0) MPa to the instrument.

6 Apparatus

6.1 Combustion analyser

The apparatus is described in more detail in Annex A. For the installation and set-up procedures, and for detailed system description, refer to the manufacturer’s manual.

The system described in this standard comprises a temperature and pressure controlled combustion chamber (6.1.1) with fluid cooling of designated areas, chamber inlet and exhaust valves and associated piping, an electronically controlled fuel injection system, a fuel delivery system, a recirculating coolant system, solenoids, sensors, controls and connection fittings for the compressed gas utilities. Figure 1 gives a schematic outline of the analyser.

6.1.1 Combustion chamber, a steel combustion chamber of capacity 0,473 l ± 0,005 l. Annex A gives further details.

6.1.2 Filter medium, a removable polytetrafluoroethylene filter with a 5 µm pore size is placed downstream from the sample vessel to filter particulate matter from the test portion.

7 Sampling

7.1 Unless otherwise specified, obtain samples in accordance with the procedures given in EN ISO 3170 or EN ISO 3171.

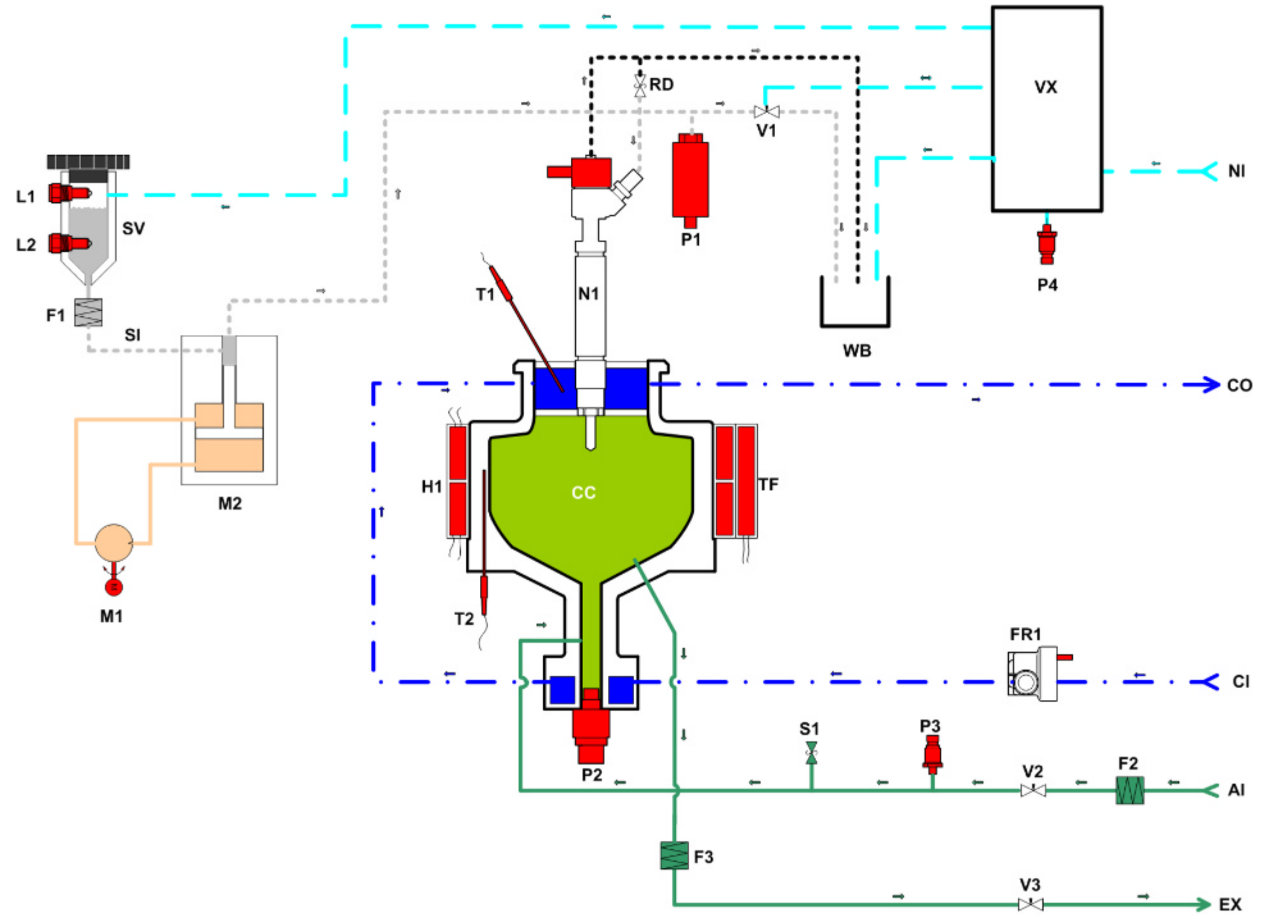
7.2 To minimize exposure to UV emissions that can induce chemical reactions, which may affect ignition and combustion delays measurement, collect and store samples in sample containers that are either constructed of materials that minimize light reaching the sample such as a dark brown bottle, metal can or containers that shall be wrapped or boxed in light-proof containers immediately after filling. If the fuel is not to be analysed within 24 h, retain in a dark, cool environment, and preferably under an inert gas.

NOTE 1 Exposure of petroleum fuels to UV wavelengths of less than 550 nm for even a short period of time has been shown to affect ignition delay [5].

NOTE 2 The formation of peroxides and radicals, which affect the ignition delay and the combustion delay, is minimized when the sample is stored in the dark, under a nitrogen blanket in a cool environment.

7.3 Condition the diesel fuel oil sample before opening the storage container, so that it is at room temperature, typically 18 °C to 32 °C.

7.4 Inspect the sample for wax precipitation. If precipitants are present, bring the test sample to a temperature of at least 14 °C above the expected cloud point of the material being tested, taking care not to lose any lower boiling range components. Agitate the sample to return precipitants back in to the solution, ensuring the sample is homogeneous before proceeding.



Key

Digital signals	**Analogue signals**
L1: upper level sensor	T1: coolant temperature
L2: lower level sensor	T2: inner wall temperature
TF: thermal fuse	P1: fuel pressure
M1: hydraulic pump	P2: chamber dynamic pressure
N1: injector	P3: chamber static pressure
V1: flush valve	P4: nitrogen pressure
V2: air inlet valve	FR1: coolant flow rate
V3: exhaust valve	
Vx: nitrogen circuit valves	
Analyser lines	**Analyser parts (no signal registration)**
AI: Air inlet	CC: Combustion chamber
CI: Coolant input	F1, F2, F3: Filter
CO: Coolant output	M2: Multiplier
EX: Air exhaust	RD: Rupture disk
NI: Nitrogen input	S1: Safety valve
SI: Sample inlet	SV: Sample vessel
	WB: Sample waste drain
	H1: Clam shell heater

Figure 1 — Schematic overview of combustion analyser

8 Apparatus assembly and installation

Annex A gives more details on the apparatus assembly and installation. The apparatus requires placement on a bench with facilities for the hook-up of all utilities and gases. The user shall ensure compliance with all local and national codes. The apparatus requires an environment with a temperature of 10 °C to 35 °C. The exhaust gases shall be directed into a low suction pressure fume extraction system.

9 Preparation of apparatus

9.1 System start-up and warm-up

9.1.1 Open the valve at the source of the combustion charge air supply and adjust the pressure regulator as needed to provide the specification pressure (5.4).

9.1.2 Open the valve at the source of the nitrogen supply and adjust the pressure regulator as needed to provide the specification pressure (5.8).

9.1.3 Switch on power to the combustion analyser and the coolant pump.

9.1.4 After the chamber wall temperature has stabilized a chamber leakage test will be performed to determine the chamber leakage rate. Combustion chamber leakage rate shall be less than 0,75 kPa/s, as measured during the automated check of the sealing integrity of the combustion chamber. If the leakage test fails, a warning is issued.

9.1.5 For more details and in case of error messages refer to the manufacturer’s manual.

9.2 Standard operating and test conditions

9.2.1 Operation of the combustion analyser requires setting a series of testing variables to prescribed specifications. Some of these settings are established by the operator, others are operating conditions that are monitored or controlled by the computer software.

9.2.2 The set points for chamber wall temperature and the injection time are determined during the calibration procedure.

9.2.3 Check that the parameters are according to Table 1. Refer to manufacturer’s manual in case the parameters are outside the limits.

Table 1 — Standard operating test conditions

Parameter	Limits
Chamber static pressure	(2,00 ± 0,02) MPa
Chamber wall temperature Stability during the 15 injections	560 °C to 640 °C ± 0,2 °C
Injector nozzle coolant temperature	(50 ± 2) °C
Injection Pressure	(100 ± 1,5) MPa
Injection time	2000 µs to 2700 µs

10 Calibration, verification and quality control

10.1 General

Calibrate and verify the apparatus at each of the following occasions:

- after it is installed and commissioned,
- after replacement of critical parts or components of combustion chamber assembly, fuel injection system, fuel injection system instrument sensors, chamber static pressure or chamber dynamic pressure sensors,
- after calibration of the chamber static pressure, or chamber dynamic pressure sensors, or
- whenever QC sample determinations are not in statistical control, and the reasons for QC non-compliance have been suitably addressed.

10.2 Calibration

10.2.1 Clean the fuel system in accordance with B.2.

10.2.2 Bring the calibration reference fluid (5.1) to a temperature greater than 20 °C and agitate before use.

10.2.3 Remove the fuel reservoir cap and wash the stem and threads and the fuel reservoir with approximately 50 ml of the calibration reference fluid. Reinstall the fuel reservoir cap.

10.2.4 Flush the entire aliquot of the calibration reference fluid through the fuel system. Refer to the manufacturer's instruction manual.

10.2.5 Charge the instrument with the calibration reference fluid (at least 160 ml) and wipe the stem and threads of the fuel reservoir cap with a clean dry towel and secure the fuel reservoir cap to the fuel reservoir.

10.2.6 Carry out the automatic calibration procedure (see B.3).

If the average ID value or the average CD value is outside the acceptance limits, the combustion chamber inner surface temperature controller set-point is adjusted by the computer to change the combustion chamber wall temperature or the fuel injection period is adjusted by the computer to inject the appropriate quantity of fuel into the combustion chamber, or both. The automatic calibration procedure performed by the processor controlling the instrument is summarized in B.3.

NOTE ID increases when the combustion chamber inner surface temperature decreases and vice versa. *CD* decreases when a larger sample volume is injected into the combustion chamber and vice versa.

If the temperature controller set-point adjustment from the previous setting exceeds ± 4 °C or the injection period adjustment from the previous setting exceeds ± 100 μs, a system malfunction is suspected and diagnostic procedures to determine and remedy the problem are recommended. Refer to the instruction manual of the manufacturer.

10.2.7 The combustion analyser calibration is complete when the calibration reference fluid average delays are within the specified acceptance limits as given in Table 2.

10.2.8 Without flushing refill the fuel reservoir with the calibration reference fluid and perform a single determination of the calibration reference fluid. The result shall satisfy the acceptance limits as given in Table 2. If the single determination exceeds the acceptance limits for either ID or CD, perform the calibration procedure again.

Table 2 — Limits for calibration and Apparatus verification

Parameter	Limit
ID for the calibration reference fluid during calibration	(2,96 ± 0,16) ms
CD for the calibration reference fluid during calibration	(4,90 ± 0,08) ms
ID for the calibration reference fluid for a single result	(2,96 ± 0,16) ms
CD for the calibration reference fluid for a single result	(4,90 ± 0,08) ms
ID for the verification reference fluid for a single result	(11,00 ± 1,30) ms
CD for the verification reference fluid for a single result	(17,00 ± 1,40) ms
ID for the verification reference fluid for the average of 2 results	(11,00 ± 1,10) ms
CD for the verification reference fluid for the average of 2 results	(17,00 ± 1,20) ms

10.3 Apparatus verification

10.3.1 Verify the calibration by using the verification reference fluid (5.2).

10.3.2 Carry out two consecutive ignition delay and combustion delay measurements using the verification reference fluid (5.2). Perform the second determination by refilling the fuel reservoir without flushing.

10.3.3 To pass the verification test, each single result of the ID and CD measurements shall be within the limits and tolerances as specified in Table 2.

10.3.4 To pass the verification test, the averaged result of the ID and CD measurements shall be within the limits and tolerances as specified in Table 2.

10.3.5 If any of the two single results or the average result is outside their tolerance limit, system performance is unacceptable. This can either be a problem with the MCH verification reference fluid (5.2) or the result of a malfunction of the system.

10.3.6 The diagnostic procedures shall be used to determine and remedy the problem before performing a new calibration. Refer to the instruction manual of the manufacturer.

10.4 Quality control (QC)

10.4.1 Proper quality control procedures shall be in place to ensure continuous satisfactory operation of the analyser. Quality control samples (5.3) shall be tested at intervals and records shall be kept of the results.

10.4.2 Carry out quality control measurements on one or more quality control samples at least daily after apparatus preparation, and after every adjustment or replacement of the combustion charge air.

Take into account the volume of the pipe work connected to the instrument after every replacement of combustion charge air cylinders.

In continuous use, the recommended QC interval is at least every 10 samples.

10.4.3 When quality control results indicate an out-of-control situation, an investigation shall be initiated, with corrective actions that can include a repeat of calibration and verification procedures.

11 Test procedure

11.1 Remove the fuel reservoir cap and wash the stem and threads and the fuel reservoir with approximately 50 ml of the test portion. Reinstall the fuel reservoir cap hand tight.

11.2 Flush the entire test portion through the fuel injection system. Refer to the instruction manual of the manufacturer.

11.3 Fill the fuel reservoir past the upper level sensor with the test portion (at least 160 ml). Wipe the stem and threads of the reservoir cap with a clean, dry towel. Reinstall the fuel reservoir cap hand tight.

11.4 Flush the entire test portion through the fuel injection system.

11.5 Remove the fuel reservoir cap and refill the fuel reservoir past the upper level sensor with the test portion (at least 160 ml). Wipe the stem and threads of the reservoir cap with a clean, dry towel. Reinstall the fuel reservoir cap hand tight.

11.6 Check if the parameters are according to Table 1. Refer to manual in case there is a problem.

11.7 Start the automatic test sequence. At the end of the test, a test output summary is automatically displayed on the computer screen.

11.8 The delay results are obtained by averaging the ID and CD measurements of the last 15 cycles. If either of the ID and CD pairs are identified as a statistical outlier according to Peirce’s Criterion [4] that pair of ID and CD measurements are removed from the 15 measurements and are not included in calculating the average values. The outlying delays, if any, are noted in the result record. A maximum of three outlier pairs of ID and CD measurements for a specific test are allowed.

11.9 Flush the remaining test portion from the fuel reservoir through the fuel injection system.

12 Calculation

The DCN result is obtained by converting the average ID and CD result from 11.8 DCN using the multivariate Formula (1):

$$\mathrm{DCN} = 13{,}028 + \left(-5{,}337\,8 / \mathrm{ID}\right) + \left(300{,}18 / \mathrm{CD}\right) + \left(-1\,267{,}90 / \mathrm{CD}^2\right) + \left(3\,415{,}32 / \mathrm{CD}^3\right) \qquad (1)$$

NOTE The derivation of Formula (1) is given in Research Report ASTM RR:D02-1771 [3].

13 Expression of results

Report the average ignition delay (ID), in ms, rounded to the nearest 0,01 ms.

Report the average combustion delay (CD), in ms, rounded to the nearest 0,01 ms.

Report the derived cetane number (DCN) rounded to the nearest 0,1.

14 Precision

14.1 General

The precision given was derived from statistical analysis by EN ISO 4259 [6] from a 2013 interlaboratory cooperative test program. Participants analysed 20 sample sets comprised of 13 distillate fuels, 2 blends of FAME in distillate fuel (B7 and B20), 4 neat FAME's (B100, soy, canola, tallow and a 30/70 blend of soy and rapeseed, respectively) and 1 aviation turbine fuel. The DCN range was 39 to 67. Sixteen laboratories participated using this standard and eleven laboratories participated using the EN ISO 5165 [2] method (which is equivalent to ASTM D613 [7]).

NOTE 1 The inter-laboratory testing, the information on the samples and the statistical evaluation are detailed in Research Report ASTM RR:D02-1771 [3].

NOTE 2 The precision of test results obtained using CRM blended by the test operator has not been determined.

14.2 Repeatability

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 normal and correct operation of the test method, exceed the values given in Table 3 only in one case in twenty. Examples of precision are shown in Table 4 for user information.

14.3 Reproducibility

The difference between two test results independently obtained by different operators operating in different laboratories on identical test material would, in the normal and correct operation of the test method, exceed the values given in Table 3 only in one case in twenty. Examples of precision are shown in Table 4 for user information.

Table 3 — Precision values

	r	***R***	**Applicable range**
DCN	0,019 82 (DCN – 21)	0,046 31 (DCN – 21)	(39,4 – 66,8) DCN
ID	0,003 035 $\mathrm{ID}^{2,5}$	0,009 86 $I\mathrm{D}^{2,5}$	(2,47 – 4,09) ms
CD	0,002 81 CD^{2}	0,006 44 CD^{2}	(3,71 – 6,74) ms

Table 4 — Calculated precision values for information

ID *ms*	**r**	**R**	**CD** *ms*	**r**	**R**	**DCN**	**r**	**R**
2,50	0,03	0,10	3,90	0,04	0,10	40,0	0,38	0,88
3,00	0,05	0,15	4,50	0,06	0,13	45,0	0,48	1,11
3,25	0,06	0,19	5,25	0,08	0,18	50,0	0,57	1,34
3,50	0,07	0,23	6,00	0,10	0,23	55,0	0,67	1,57
4,00	0,10	0,32	6,75	0,13	0,29	60,0	0,77	1,81
						65,0	0,87	2,04

15 Test report

The test report shall contain the following information:

a) a reference to this European Standard, i.e. EN 16715;

b) the type and complete identification of the product tested;

c) the result of the test (see Clause 13);

d) any deviation, by agreement or otherwise, from the procedures specified;

e) the date of the test.

Annex A
(normative)
Combustion analyser description

A.1 General

The apparatus consists of a combustion chamber that is supported by sub-systems to supply a charge of air and fuel, and to measure temperature, pressures, ignition and combustion delay and injection time.

A.2 Apparatus description and assembly

A.2.1 Combustion chamber

A cylindrical chamber having a volume of (0,473 ± 0,005) l, with external heating elements, heat shield, and electrically actuated intake and exhaust valves. There is an opening at one end of the chamber to accommodate insertion of the fuel injection nozzle assembly and there are openings at the other end of the chamber to insert air, remove exhaust, and attach a pressure sensor.

See Figure 1 for details. A stainless steel vessel manufactured from three pieces. The top piece accepts the common rail injector (N1) and provides for a coolant passage to cool the common rail injector. A type K thermocouple (T1) is used to monitor cooling fluid temperature and used to set the bath temperature for the closed loop circulating coolant system. Fitted to the bottom of the combustion chamber is the dynamic pressure sensor (P2) used to record the combustion pressure curve. The dynamic pressure sensor is also cooled using the circulated cooling fluid. The combustion chamber wall is heated using a clam shell heater (H1). The wall temperature of the combustion chamber is measured using a type K thermocouple (T2). A thermal fuse is fitted to the back of the clam shell heater to prevent overheating of the combustion chamber.

A.2.2 Fuel injection system

The material under test is supplied to the pressure multiplier from a fuel reservoir and is delivered to an electronic diesel fuel injector at high pressure, generated using a hydraulic pump and pressure multiplier.

The system includes:

A.2.2.1 **Fuel sample reservoir assembly**, metal reservoir having a nominal volume of 200 ml.

A.2.2.2 **Hydraulic pump**, capable of producing fuel pressures up to 19 MPa.

A.2.2.3 **Pressure multiplier**, 10:1 ratio.

A.2.2.4 **Fuel injector**, solenoid-based common rail diesel fuel injector.

The common rail injector is manufactured by Bosch and has the part number 0445110181.1)

The total number of jets is six. Jet orifice diameter: 0,17 mm.

---

1) The common rail injector is the trade name of a product supplied by Bosch. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN or CENELEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

The flow volumes are measured using hexadecane. The values are the average volume per injection from four injectors. See Table A.1 for further guidance.

Table A.1 — Injection volume vs. injection time

Injection pulse width (µs)	**Injection volume** (µL)
2 000	101
2 250	113
2 500	125
2 750	138

A.2.2.5 Safety burst disk, attached to the high pressure sample system manifold block opposite the injector, for relieving the high pressure if the sample pressure exceeds 180 MPa.

A.2.2.6 Flush valve, high pressure air actuated valve used to exchange samples.

A.2.2.7 Functioning, see Figure 1: The fuel vessel is the fuel reservoir and is fitted with two level sensors (L1, L2). The upper level sensor (L1) warns the user when the fuel vessel is getting full. When triggered, the lower level sensor (L2) stops the analysis process preventing air from being introduced into the high pressure fuel loop. A removable polytetrafluoroethylene filter with a 5 µm pore size is placed downstream from the fuel vessel to filter particulate matter from the fuel. The fuel is forced through the fuel injection system using low pressure nitrogen gas above the fuel in the fuel reservoir.

The fuel is pushed into the high pressure multiplier (Multiplier) through a one-way valve by the head pressure of nitrogen in the sealed fuel vessel. The pressure multiplier is controlled by the hydraulic pump and the pressure control circuit (M1). The pressure multiplier produces a fuel pressure that is 10-times the pressure generated by the hydraulic pump.

The high pressure fuel generated by the Multiplier enters the high pressure fuel manifold. Attached to the high pressure manifold is the common rail fuel injector (N1), the fuel pressure sensor (P1) and the air operated high pressure flush valve (V1), and the high pressure safety burst disk (Rupture Disk). The fuel pressure sensor measures the pressure of the fuel before the fuel is injected into the combustion chamber. If the measured fuel pressure is not within the tolerance limits, the solenoid on the common rail injector will not be energized and the fuel will not be injected into the combustion chamber. If the fuel pressure exceeds the safety margin of the high pressure system, the rupture disk will break and the fuel pressure will be relieved into the waste bottle. Opening the flush valve (V1) allows the fuel to be flushed from the fuel vessel and the fuel lines to the fuel waste bottle.

A.2.3 Coolant system, a closed loop circulating coolant system filled with a 50:50 volume percent mixture of commercial grade ethylene glycol type vehicle radiator antifreeze and water. The temperature output from the thermocouple T1 is used to adjust the bath temperature of the chiller such that T1 reports a temperate of (50 ± 2) °C. The flow sensor (FR1) is a safety device to ensure that coolant is flowing through the cooling spaces in the combustion chamber. The combustion chamber clam shell heater is de-energized if the flow sensor does not detect flow.

A.2.4 Combustion Charge Air Circuit. The synthetic air used to charge the combustion chamber is first filtered to protect the air inlet solenoid valve (V2). The combustion chamber static pressure sensor (P3) is used to correct for the temperature offset of the dynamic pressure sensor (P2). The safety valve ensures the pressure inside the combustion chamber does not exceed 5 MPa. The filter placed in front of the exhaust solenoid valve (V3) prevents particulate matter generated during the combustion from fouling exhaust valve.

A.3 Control and data acquisition

A.3.1 Instrument sensors, to measure and either indicate the value of a variable or transmit the condition for control or data acquisition purposes such as:

A.3.1.1 Combustion chamber static pressure sensor, calibrated, to correct the temperature offset of the dynamic pressure sensor.

A.3.1.2 Combustion chamber dynamic pressure sensor, calibrated, to measure the pressure within the combustion chamber.

A.3.1.3 Sample pressure sensor, calibrated, to measure the pressure of the fuel injected into the combustion chamber.

A.3.1.4 Nitrogen pressure sensor, to measure the inlet pressure from the nitrogen regulator.

A.3.1.5 Combustion chamber inner wall temperature sensor, Type K thermocouple with a stainless steel sheath.

A.3.1.6 Injector nozzle cooling jacket temperature sensor, Type K thermocouple with stainless steel sheath, inserted in the injector nozzle coolant passage.

A.3.2 Data processing

A microprocessor controlled system with a keyboard for manual entry of operating instructions, an LCD monitor for visual observation of all testing functions, and a printer for printed copy output of test results. The computer-based system provides automated control of the relevant combustion analyser and subsystem component functions and to collect and process all relevant signals from the temperature and pressure sensors.

A.3.3 Compressed gas pressure regulators

A.3.3.1 Combustion charge air regulator, two-stage, capable of controlling the downstream pressure to a minimum pressure of 2,2 MPa.

A.3.3.2 Nitrogen regulator, two-stage, cap able of controlling the downstream pressure to a minimum pressure of 0,7 MPa.

Annex B
(normative)

Operational details in support to the standard test procedure

B.1 General

This annex specifies operational details in support to the standard procedure given in Clause 11.

B.2 Cleaning procedure to prepare fuel system for calibration

B.2.1 Clean the fuel reservoir, the fuel reservoir cap, and the sample system.

B.2.2 Wash the fuel reservoir cap stem and threads, fuel reservoir funnel, the inside walls of the fuel reservoir and the protruding level sensor housings with approximately 50 ml of n-heptane (5.5) and flush the solvent from the fuel reservoir.

B.2.3 Replace the fuel reservoir cap and flush the solvent completely from the fuel reservoir allowing the flush cycle to run its full course.

B.2.4 Repeat steps B.2.2 and B.2.3 once.

B.2.5 Remove the fuel reservoir cap and wipe the stem and threads of the fuel reservoir cap with a clean dry towel.

B.2.6 Leave off the fuel reservoir cap and allow the solvent to evaporate.

NOTE Dry, oil-free compressed air to assist with evaporation of the solvent can be used.

B.3 Automatic calibration procedure

B.3.1 This procedure summarizes the adjustments to the combustion chamber wall temperature setpoint and the injection period made by the processor controlling the instrument during the calibration procedure.

B.3.2 After cleaning and filling the fuel vessel with the calibration reference fluid, the calibration procedure is initiated by pressing the appropriate softkey.

B.3.3 The fuel is loaded into the high pressure sample loop and injections of the fuel into the combustion chamber begin.

B.3.4 Using a moving average, ID and CD are tested for stability.

B.3.5 Once ID and CD are determined to be stable the ID’s and CD’s from the last 15 injections are averaged to calculate an average ID and an average CD using Pierce’s Criterion to identify outlying ID and CD pairs.

B.3.6 The average ID is compared to the target ID value of (2,96 ± 0,16) ms and the average CD is compared to the target CD value of (4,90 ± 0,08) ms. If the ID and CD meet their respective target values within the acceptance tolerance intervals, the calibration procedure is terminated.

B.3.7 If ID and/or CD exceed their respective tolerance interval, the magnitude and sign of the difference between the average value and the target value causes the combustion wall temperature setpoint and/or the injection period, respectively, to be adjusted on a sliding scale and in a particular direction.

B.3.8 If an ID adjustment is made, the chamber wall temperature is allowed to stabilize, and an additional 15 fuel injections are acquired to calculate average ID and average CD values.

B.3.9 If the ID and CD meet their respective target value within the acceptance tolerance interval the calibration procedure is terminated. See B.3.6.

B.3.10 If the average ID is outside the tolerance limit, the magnitude and sign of the difference between the average value for ID and the ID target value causes the combustion wall temperature setpoint to be adjusted on a sliding scale and in a particular direction, respectively.

B.3.11 After the chamber wall temperature has stabilized, an additional 15 fuel injections are made to calculate average ID and CD values.

B.3.12 Steps B.3.10 and B.3.11 are repeated until the average value for ID falls within the tolerance limit for the ID target value.

B.3.13 If the average CD value is outside the tolerance limit, but the average ID is within the tolerance limit, the magnitude and sign of the difference between the average value for CD and the CD target value causes the injection period to be adjusted on a sliding scale and in a particular direction, respectively. See B.3.6.

B.3.14 After the chamber wall temperature has stabilized, an additional 15 fuel injections are acquired and the average ID and CD are calculated.

B.3.15 Steps B.3.13 and B.3.14 are repeated until the average value for CD falls within the tolerance limit for the CD target value. See B.3.6.

B.3.16 The iterative calibration procedure continues until the average ID and average CD value are within their respective target value acceptance interval for a set of 15 injections.

B.4 Test sequence

B.4.1 A complete automated test run consists of 5 preliminary (pre-injections) plus 15 subsequent (test injections) automated combustion cycles. A combustion cycle involves firstly charging the combustion chamber, with combustion charge air, to the chamber static pressure, then injecting a test portion of fuel into the temperature and pressure controlled combustion chamber, and finally, releasing the combustion gases. During the combustion cycle, the combustion chamber pressure sensor measures the combustion chamber pressure.

B.4.2 The signal of the combustion chamber pressure sensor defines the start of injection and the start of combustion. An example output of these signals against time for a single combustion cycle during a test sequence, is given in Figure B.1.

The first 5 combustion cycles are performed in order for the apparatus to attain equilibrium conditions and to clean the injector. The ignition and combustion delays of the successive 15 combustion cycles are accumulated and then averaged to produce the analytical ignition and combustion delay results (see Clause 12).

B.4.3 Data record

During each of the 15 combustion test cycles, the following parameters shall be recorded:

a) ignition delay (ID);

b) combustion delay (CD);

c) derived cetane number (DCN);

d) combustion chamber inner wall temperature;

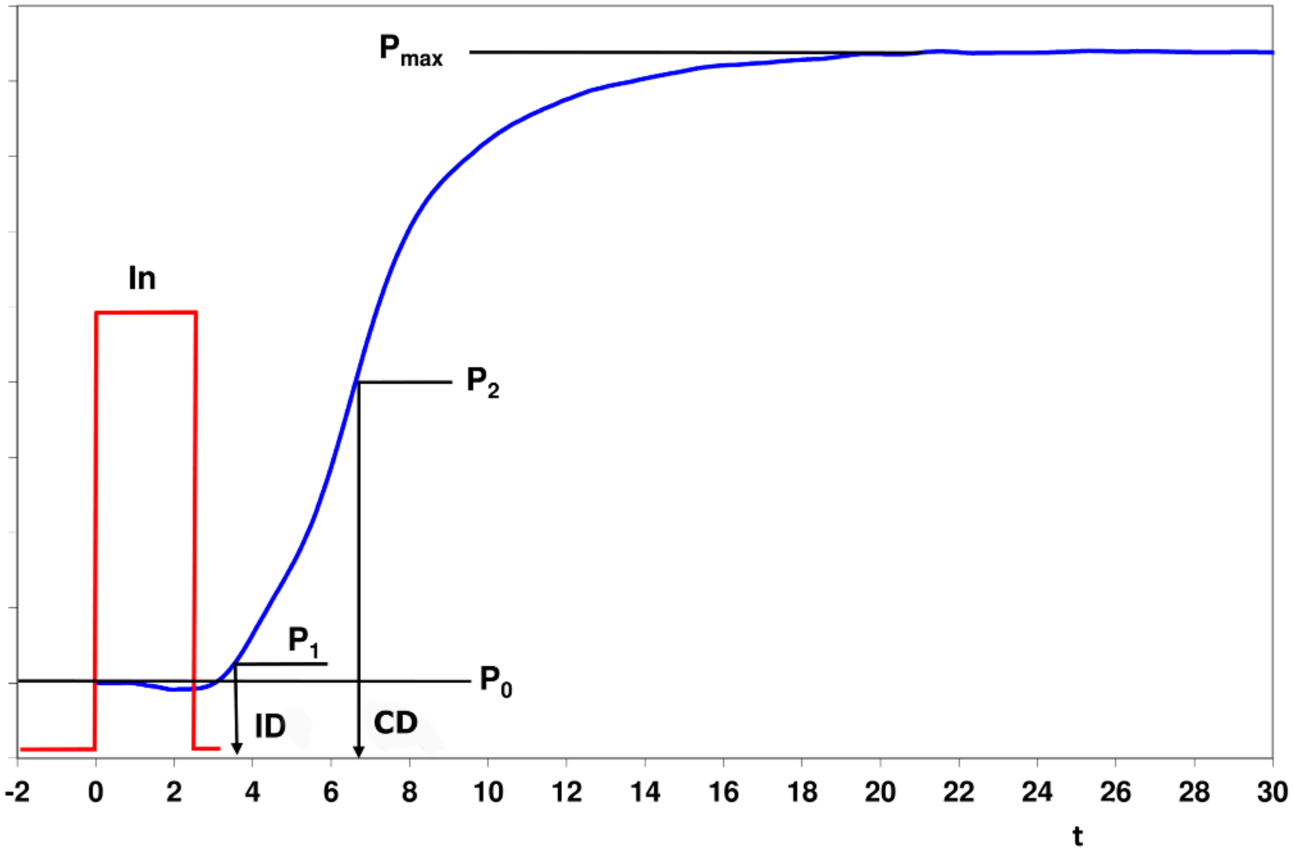
e) chamber static pressure;

f) injection pressure;

g) injector nozzle coolant temperature.

NOTE 1 The individual values of the above parameters, together with their average are automatically reported by the equipment.

NOTE 2 The instrument records the derived cetane number, but the indicated parameters are needed to determine the DCN according to the formula under Clause 12 and to determine whether test conditions are within acceptable limits (see 9.2).



Key

P_0	initial chamber pressure
P_1	P_0 + 0,02 MPa (pressure to detect ID)
P_2	(P_0 + P_{max}) / 2 (pressure to detect CD)
P_{max}	max combustion chamber pressure
In	signal to open the injector
ID	ignition delay time
CD	combustion delay time
t	time (ms)

Figure B.1 — Typical signal output of Injector and pressure sensor for a single combustion cycle

B.5 Unit shutdown

The shutdown procedure decompresses the combustion chamber and switches off the heating element to allow the combustion chamber to cool down.

Confirm that the entire specimen has been discharged from the fuel system and that the fuel reservoir is empty.

Close the valve at the source of the charge air supply and nitrogen supply. Use the applicable computer command to shut down the combustion analyser. Do not turn off the circulation coolant system until 1,5 h has elapsed or until prompted to do so by the appropriate message on the LCD.

After 1,5 h or after the appearance of appropriate prompt on the LCD position the combustion analyser power switch to OFF.

Bibliography

[1] ASTM D7668-13, *Standard Test Method for Determination of Derived Cetane Number (DCN) of Diesel Fuel Oils - Ignition Delay and Combustion Delay Using a Constant Volume Combustion Chamber Method*

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

[3] ASTM Research report RR: D02-1771:2013, available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA.

[4] Ross, Stephen, Peirce's Criterion for the Elimination of Suspect Experimental Data, J. Engr. Technology, Fall 2003.

[5] ASTM RR: D02-1502, Sunlight and Air Exposure Effects on Octane Number or Cetane Number of Petroleum Product Samples, available from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA.

[6] EN ISO 4259, *Petroleum products - Determination and application of precision data in relation to methods of test (ISO 4259)*

[7] ASTM D613-10a, *Standard Test Method for Cetane Number of Diesel Fuel Oil*

This page deliberately left blank

This page deliberately left blank

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards -based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email bsmusales@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Copyright

Useful Contacts:

Customer Services
Tel: +44 845 086 9001
Email (orders): orders@bsigroup.com
Email (enquiries): cservices@bsigroup.com

Subscriptions
Tel: +44 845 086 9001
Email: subscriptions@bsigroup.com

Knowledge Centre
Tel: +44 20 8996 7004
Email: knowledgecentre@bsigroup.com

Copyright & Licensing
Tel: +44 20 8996 7070
Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

...making excellence a habit.™