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

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

Produits pétroliers liquides - Détermination de délai d'inflammation et de l'indice de cétane dérivé (ICD) des distillats moyens par combustion dans une enceinte à volume constant

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

This European Standard was approved by CEN on 20 September 2014.

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Foreword

This document (EN 15195:2014) 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of an identical text or by endorsement, at the latest by May 2015 and conflicting national standards shall be withdrawn at the latest by May 2015.

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 EN 15195:2007.

Based on new data sets used and experience in the field, the major updates towards the former version are:

— based on recent data from EI and ASTM correlation schemes precision of the method has been improved (by around 25 %) and a common global precision statement for EN 15195 has been incorporated (see also the Introduction) [9];

— the ignition delay range has been expanded to 2,8 ms to 6,3 ms (71 DCN to 34 DCN), where it used to be 3,3 ms to 6,4 ms (61 DCN to 34 DCN);

— the scope has been expanded to from diesel blends with 7 % (*V/V*) up to 30 % (*V/V*) of FAME;

— the test procedure has been updated following experience in the market;

— the standard operating and test conditions have been more precisely defined;

— the calibration information has been improved;

— an alternative system cleaning procedure has been introduced in Annex B.

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 is derived from joint standardization work in the Energy Institute and ASTM International. It has originally been based on IP 498/06 [1] published by the Energy Institute and harmonized with equivalent ASTM [2] Standards.

The described method is an alternative quantitative determination of the cetane number of middle distillate fuels intended for use in compression ignition engines. Correlation studies between this method and EN ISO 5165 have been done and the results of this are incorporated in this European Standard.

The basis of this method is the derived cetane number correlation equation as given in Clause 13. The on-going validation of the equation is monitored and evaluated through the existing monthly 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.

For the moment the basics of one type of apparatus are described[1]. 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] The injection pump in the currently described apparatus is covered by a patent.

1 Scope

This European Standard specifies a test method for the quantitative determination of ignition delay of middle distillate fuels intended for use in compression ignition engines. The method utilizes a constant volume combustion chamber designed for operation by compression ignition, and employing direct injection of fuel into compressed air that is controlled to a specified pressure and temperature. An equation is given to calculate the derived cetane number (DCN) from the ignition delay measurement.

This European Standard is applicable to diesel fuels, including those containing fatty acid methyl esters (FAME) up to 30 % (*V/V*). 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.

The test method is also applicable to the quantitative determination of the ignition characteristics of FAME, especially the ignition delay. However the correlation data available were inconclusive about the precision of the equation. So the determination of derived cetane number for FAME fuel, also known as B100, has not been included in the precision determination as in Clause 12[2)].

This European Standard covers the ignition delay range from 2,8 ms to 6,3 ms (71 DCN to 34 DCN). The combustion analyser can measure shorter or longer ignition delays, but precision is not known. For these shorter or longer ignition delays the correlation equation for DCN is given in Annex D.

NOTE 1 There is no information about how DCNs outside the 34 to 71 range compares to EN ISO 5165.

NOTE 2 For the purpose of this European Standard, the expression "% (*V/V*)" is used to represent the volume fraction and "% (*m/m*)" the mass fraction.

WARNING — The use of this standard may 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 the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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)*

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

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

ISO 4010, *Diesel engines — Calibrating nozzle, delay pintle type*

2) A further Round Robin study for B100 samples is being considered by CEN.

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 1998-2:1998 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.

Note 2 to entry: ISO 1998-2 expresses it as “number on a conventional scale, indicating the ignition quality of a diesel fuel under standardized conditions”, but for this document the definition as given is chosen as with new equipment on the market since 1998 the reference to an engine has become essential.

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

Note 1 to entry: In the context of this standard, this period is determined by movement and pressure sensors in the instrument.

3.3
derived cetane number
DCN
number calculated by using an equation that correlates a combustion analyser's ignition delay to the cetane number

3.4
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.

3.5
quality control sample
QC
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

3.6
calibration reference fluid
stable and homogenous fluid used to calibrate the performance of the combustion analyzer

3.7
verification reference fluid
stable and homogenous fluid used to verify the performance of the combustion analyzer

4 Principle

A test portion of the material under test is injected into a heated temperature- and pressure-controlled constant volume combustion chamber which has previously been charged with compressed air. Sensors detect the start of injection and the start of combustion for each single-shot cycle. A complete test sequence consists of 15 preliminary combustion cycles to ensure apparatus equilibrium and 32 subsequent test cycles to obtain ignition delay values. The average ignition delay (ID) of these 32 cycles is inserted into an equation to obtain the derived cetane number (DCN). 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.

5 Reagents and materials

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

5.2 Coolant system fluid, 50:50 (V/V) mixture of commercial grade radiator antifreeze (aluminium-compatible, ethylene glycol-type) with water (5.1).

NOTE This mixture meets the boiling point requirements and gives adequate protection of the coolant system against corrosion and mineral scale that can alter heat transfer and rating results. See the manufacturer's manual for the correct ethylene glycol-type antifreeze quality.

5.3 Calibration reference fluid, heptane of a purity of minimum 99,5 % (*m/m*) to be used as the designated 3,78 ms ignition delay accepted reference value material.

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

5.4 Verification reference fluid, methylcyclohexane of a purity of minimum 99,0 % (*m/m*) to be used as the designated 10,4 ms ignition delay 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 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 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.

5.5 Quality control sample, stable and homogeneous material(s), similar in nature to the materials under test (see 3.5)

5.6 Combustion charge air, of oxygen content 20,9 % (*V/V*) ± 1,0 % (*V/V*), and containing less than 0,003 % (*V/V*) hydro-carbons and less than 0,025 % (*V/V*) water.

NOTE 1 Oxygen content of combustion charge compressed air can vary between batches (cylinders). Significant variation will lead to changes in ignition delay (higher oxygen content leads to a shorter ignition delay).

NOTE 2 The effects of oxygen concentration have been investigated [3].

5.7 Actuating air, oil-free compressed air containing less than 0,1 % (*V/V*) water supplied at a minimum sustained pressure of 1,5 MPa.

5.8 Compressed nitrogen, of minimum purity 99,9 % (*V/V*).

6 Apparatus

6.1 Combustion analyser

6.1.1 General

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: an insulated heated, constant volume combustion chamber (see 6.1.2) with fluid cooling of designated areas; external, pneumatically actuated, chamber inlet and exhaust valves, and associated piping; a heated, pneumatically-actuated, fuel injection pump; a constant pressure fuel delivery system; a re-circulating coolant system; solenoids; sensors; controls; connection fittings for the compressed gas utilities; and a computer to control test sequencing. Figure 1 gives a schematic outline of the analyser.

6.1.2 Combustion chamber, steel combustion chamber of capacity 0,213 l ± 0,002 l, further detailed in Annex A.

6.2 Filter medium, with a nominal pore size 3 µm to 5 µm, made of glass fibre, polytetrafluorethylene (PTFE) or nylon, of a size appropriate to the apparatus being used for sample filtration (see 7.5).

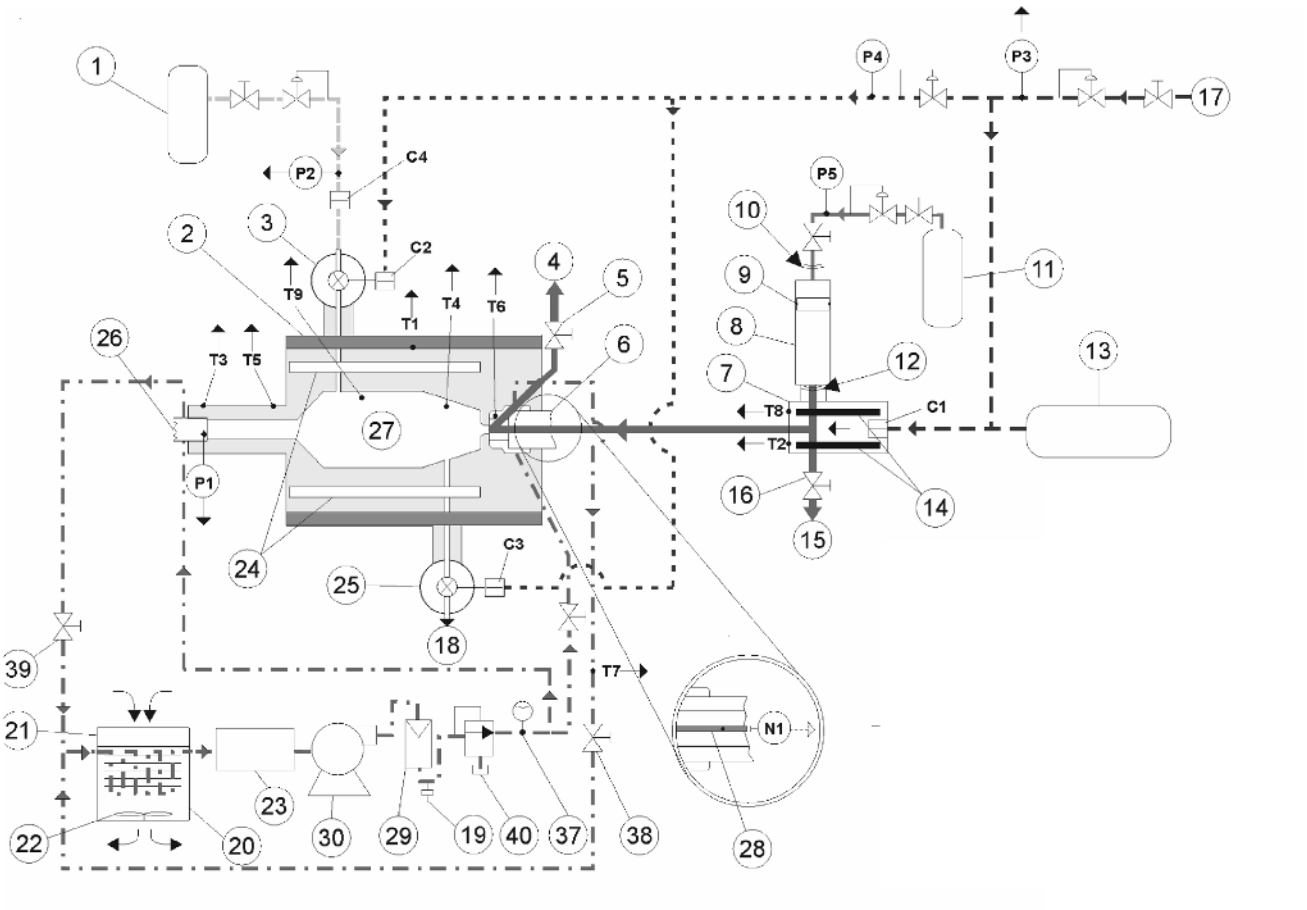


Figure 1 — Schematic overview of combustion analyser

Key

P1: combustion chamber pressure
P2: combustion charge air pressure
P3: injection actuator air pressure
P4: inlet/exhaust valve actuator air pressure (gauge)
P5: sample fuel reservoir pressure (gauge)
T1: combustion chamber outer surface temperature
T2: fuel injection pump temperature
T3: combustion chamber pressure sensor temperature
T4: charge air temperature
T5: (used for diagnostic functions)
T6: injector nozzle coolant passage temperature
T7: coolant return temperature
T8: (used for diagnostic functions)
T9: combustion chamber air back temperature
N1: injector nozzle needle motion sensor
C1: digital signal - fuel injection actuator
C2: digital signal - inlet valve actuator
C3: digital signal - exhaust valve actuator
C4: digital signal – charge air valve actuator

: charge air line
: fuel injection pump driver air line
: inlet/exhaust valve actuator air line
: coolant system line
: fuel reservoir utility nitrogen line
: high pressure fuel line

Mechanical system

1.charge air supply
2.insulation
3.inlet valve
4.hydrocarbon waste
5.nozzle bleed
6.injector nozzle
7.fuel injection pump
8.fuel sample reservoir with or without a check valve[3)]
9.plunger
10.quick connect valved fitting
11.fuel reservoir utility compressed nitrogen supply[4)]
12.quick connect valved fitting
13.pneumatic driver air surge tank
14.pump heating elements
15.hydrocarbon waste
16.pump bleed
17.actuator utility compressed air supply
18.exhaust to ventilation system
19.drain
20.liquid to air heat exchanger
21.air filter
22.fan
23.coolant reservoir
24.chamber heating elements
25.exhaust valve
26.combustion chamber pressure sensor coolant housing
27.combustion chamber
28.injector nozzle needle extension pin
29.coolant filter
30.coolant pump
37.coolant flow indicator
38.injector nozzle coolant flow control valve
39.pressure sensor coolant flow control valve
40.pressure relief valve

Figure 1 — Schematic overview of combustion analyser *(continued)*

[3)] The standard fuel sample reservoir does not have a check valve. A larger volume fuel sample reservoir does have a check valve and permits the fuel sample reservoir to be filled and cleaned in a remote, well-ventilated area when used in conjunction with remote filling/cleaning station. Refer to manufacturer's instructions for the details of this larger volume fuel reservoir and filling station.

[4)] May also be used with an associated nitrogen adaptor. The fuel system flushing adaptor is used as in B.4 to permit nitrogen to be blown through the fuel injection system when using a larger volume fuel sample reservoir with a check valve.

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 delay 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 sample 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 [4].

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

7.3 Bring the laboratory sample to 18 °C to 32 °C before testing.

7.4 Inspect the sample before testing for wax precipitation. If precipitants are present, bring the test sample to a temperature of approximately 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 filtering.

7.5 Filter the laboratory sample through the filter medium (see 6.2) at ambient temperature, without vacuum. Use a positive pressure filtration system. Immediately collect the filtered sample in a container as described in 7.2.

WARNING — If a glass syringe is used to filter the sample, ensure that the filter capsule is correctly located on the syringe fitting. Do not apply excessive force to the plunger as this could result in the glass syringe shattering. It is recommended that protective gloves are worn during the filtering operation.

8 Apparatus assembly and installation

Annex A and Annex B give more details on the apparatus assembly and installation. The apparatus requires placement on a level floor with facilities for the hook-up of all utilities and gasses. The user shall ensure compliance with all local and national codes. The apparatus requires an environment with a temperature of 18 °C to 32 °C. The exhaust gases shall be directed into a low suction pressure fume extraction system.

NOTE The heat exchange of the coolant system and the injection pump operate satisfactorily at 18 °C to 32 °C.

CAUTION 1 — The apparatus requires high-pressure compressed air at high flow for intermittent short periods of time.

CAUTION 2 — The noise level without a noise reduction system is approximately 86 dB, measured at 1,5 m distance, and approximately 77 dB with noise reduction. Local regulations may apply to high noise levels, but ear protectors should be worn when equipment is in operation.

9 Preparation of apparatus

9.1 System start-up and warm-up

9.1.1 For more details refer to the manufacturer’s manual.

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

9.1.3 Warm up the system.

NOTE At the end of the automated warm-up sequence, the ramp-up and total warm-up times will be indicated on the computer monitor. Typical values are 1 300 s to 1 800 s for ramp-up time and 1 500 s to 2 300 s for the total warm-up time. Significant increases in the average ramp-up time (more than 5 %) or total warm-up time (more than 10 %) indicates a potential malfunction of the heating elements of the combustion chamber. For diagnostic procedures, refer to the manufacturer's manual.

9.2 Standard operating and test conditions

9.2.1 Adjust the pressure (P5) of the nitrogen supply to the sample fuel reservoir to approximately 345 kPa (50 psi).

9.2.2 Adjust the pressure (P3) of the injection actuating air to meet the requirements of Table 1.

9.2.3 Check that the coolant temperatures (T6 and T7) are in accordance with the temperatures and tolerances as given in Table 1. If they are not then follow the diagnostic procedures given in the manufacturer's manual.

Table 1 — Standard operating and test conditions

Parameter	Limits
Charge air pressure (P2)	2,137 MPa ± 0,007 MPa (310 psi ± 1 psi)
Injection actuator air pressure (P3)	1,21 MPa ± 0,03 MPa (175 psi ± 4 psi)
Injector nozzle coolant passage temperature (T6)	50 °C ± 4 °C
Coolant return temperature (T7)	40 °C ± 10 °C
• Charge air temperature (T4)	$(T4_{max} - T4_{min}) < 2,5$ °C
• Combustion chamber pressure sensor temperature (T3)	$(T3_{max} - T3_{min}) < 8$ °C
Fuel injection pump temperature (T2)	35 °C ± 3 °C

9.2.4 Adjust the fuel injection pump temperature (T2) to meet the requirements of Table 1.

9.2.5 Set the charge air pressure (P2) in accordance with the requirement of Table 1.

9.2.6 Check the sealing of the combustion chamber by measuring the pressure drop during a charge test in accordance with the manufacturer's manual. If the pressure drop is greater than 3,5 kPa/s (0,5 psi/s) follow the diagnostic procedures as given in the manufacturer's manual.

9.2.7 Check that the operating temperatures and pressures are within the tolerances given in Table 1 and that the tolerances in Table 2 for a single test are met, by conducting a test using the calibration reference fluid (5.3), in accordance with Clause 11.

NOTE $T6$, $T6_{min}$, $T6_{max}$, $T3_{min}$ and $T3_{max}$ are printed out as a supplemental output result. T6 is also shown on the computer display during the test run.

9.2.8 If one or more conditions in Table 1 are not met, follow the diagnostic procedures in the manufacturer's manual to identify, and then remedy the problem.

NOTE 1 The charge air temperature is initially set by factory calibration and subsequently tuned by user calibration of the apparatus performance characteristics. The charge air temperature typically ranges from 545 °C ± 30 °C.

NOTE 2 Typical combustion chamber pressure sensor temperatures for the design described in this standard are in the range of 130 °C ± 20 °C.

9.2.9 If the requirements of Table 2 are not met for a single calibration reference fluid (5.3) test, the cause should be investigated and 9.2.7 repeated.

9.2.10 If the requirements of Table 1 and Table 2 are met, test the quality control sample (5.5) or, calibrate and verify the instrument in accordance with Clause 10.

Table 2 — Tolerance limits for apparatus calibration and verification

Fluid	Test mode	Tolerance limits ms
Calibration (heptane)	Single	3,78 ± 0,06
Calibration (heptane)	Average of three	3,78 ± 0,01
Verification (MCH)	Single	10,4 ± 0,6
Verification (MCH)	Average of two	10,4 ± 0,5

10 Calibration, verification and quality control

10.1 General

Calibrate the combustion analyser at each of the following occasions:

1) after it is installed and commissioned,

2) after replacement of critical parts or components of combustion chamber assembly, fuel injection system or instrument sensors,

3) after calibration of the data acquisition board, and

4) whenever quality control sample determinations are not in statistical control, and the reasons for quality control non-compliance have been suitably addressed.

NOTE For further maintenance advice see Annex C.

10.2 Calibration

10.2.1 Measure the ignition delay of the calibration reference fluid (see 5.3) three times following the procedure given in Clause 11.

10.2.2 Check the three single test results and the average of the three results against the tolerances given in Table 2. If the tolerances are met, the apparatus is calibrated and fit to proceed to verification.

10.2.3 If the values in 10.2.2 deviate by more than the tolerance limits given in Table 2, the apparatus is not acceptable for use, and adjustment of the charge air temperature is required. Re-set the combustion chamber skin temperature to adjust the air temperature, allow a stabilization time of at least 10 min, and repeat 10.2.1.

NOTE 1 The ignition delay increases as combustion charge air temperature decreases and conversely decreases as combustion charge air temperature increases.

NOTE 2 If the temperature controller set-point adjustment from the previous setting exceeds ± 4°C, a system malfunction is suspected and diagnostic procedures to determine and remedy the problem are recommended. Refer to the instruction manual of the manufacturer.

10.3 Apparatus verification

10.3.1 Measure the ignition delay of the verification reference fluid (see 5.4) twice following the procedure given in Clause 11.

10.3.2 If the two single results and the average of the two results are within the tolerances given in Table 2, the calibration is verified and the apparatus is acceptable for use.

10.3.3 If the values in 10.3.2 deviate by more than the tolerance limits given in Table 2 the apparatus is not acceptable for use. This can either be a problem with the MCH verification reference fluid, see 5.4 or the result of a malfunction of the system. Follow the diagnostic procedures in the manufacturer's manual to identify and remedy the problem.

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 (5.5) shall be tested at intervals (see 10.4.2) 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.

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

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

10.4.3 When quality control results are outside the control limits, carry out corrective action starting with a repeat of the calibration and verification procedures.

11 Test procedure

11.1 Flush the fuel injection system with the filtered sample (see B.1).

11.2 Fill and purge the fuel injection system with filtered sample (see B.2).

11.3 Start the test sequence (see B.3).

11.4 Check that during the test all conditions are within the required limits given in Table 1. If the conditions are in compliance, proceed to 11.5. If one or more conditions are not within the stated limits, follow the diagnostic procedures in the manufacturer's manual to identify and remedy the source of variability, and discard the test result.

NOTE Standard test conditions are reached after 15 (preliminary) combustion cycles. Only the test conditions during the next 32 (measurement) combustion cycles are recorded and considered.

11.5 Record the average ignition delay, *ID*, in milliseconds, to the nearest 0,001 ms.

11.6 Clean the fuel injection system (see B.4) or according the alternative cleaning procedure when samples with unknown or high levels of 2-EHN are used (see B.5).

NOTE A carryover effect has been observed after testing samples containing more than 2 000 µl/l of 2-ethylhexylnitrate (2-EHN), commonly called cetane improver.

12 Calculation

Calculate the derived cetane number, *DCN*, from the average ignition delay, *ID*, in milliseconds (recorded as in 11.5), using the following formula:

$$DCN = 4{,}460 + 186{,}6/ID \quad (1)$$

NOTE The equation that relates ignition delay to derived cetane number was originally developed in 1997 [5]. In 2005 the equation was re-evaluated by the EI and ASTM through the correlation of cetane number data from the IP and the National Exchange Group (NEG) Diesel Fuel Engine Correlation Schemes and ignition delay data on the same samples from the IP and NEG IQT Correlation Schemes collected over a number of years [6]. In 2006 another ASTM evaluation [7] led to the actual equation, which showed an optimal fit over the range of the scope (see Introduction).

13 Expression of results

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

Report the derived cetane number (DCN), calculated in Clause 12, to the nearest 0,1.

14 Precision

14.1 General

The precision given was derived from statistical analysis by EN ISO 4259 [8] of the results of interlaboratory testing of a matrix of fuels including petroleum and unconventional fuels and fuels with and without ignition improving additives within the ignition delay range of 2,8 ms to 6,3 ms.

NOTE 1 The interlaboratory testing and the statistical evaluation are detailed in Research Report IP 498/13 [9]. The DCN precision has been recalculated [9] following the current DCN equation (see Clause 12, Formula (1)).

NOTE 2 Due to sample specific bias effects observed this standard may systematically over-predict EN ISO 5165 for some fuels, while for other fuels, it may systematically under-predict EN ISO 5165.

NOTE 3 The repeatability for testing FAME samples (see also Clause 1) has been found to be similar, whereas the reproducibility may differ about 7 %.

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 20. Examples of precision are shown in Tables 4A and 4B 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 20. Examples of precision are shown in Tables 4A and 4B for user information.

Table 3 — Precision values

Category	Ignition delay (ID) *milliseconds*	Derived Cetane Number (DCN)
Repeatability, *r*	0,015 23 * *ID*	0,013 80 * *DCN*
Reproducibility, *R*	0,052 38 * *ID*	0,046 82 * *DCN*

Table 4 — Exemplary precision data

Table 4A — Ignition delay data

ID ms	**r** ms	**R** ms
2,8	0,043	0,147
3,2	0,049	0,168
3,7	0,056	0,194
4,2	0,064	0,22
4,7	0,072	0,246
5,2	0,079	0,272
5,7	0,087	0,299
6,3	0,096	0,330

Table 4B — Derived cetane number data

DCN	**r**	**R**
35	0,483	1,639
40	0,552	1,873
45	0,621	2,107
50	0,690	2,341
55	0,759	2,575
60	0,828	2,809
65	0,897	3,043
70	0,966	3,277

15 Test report

The test report shall contain at least the following information:

a) reference to this document, i.e. EN 15195:2014;

b) type and complete identification of the product tested;

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

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

e) date of the test.

Annex A
(normative)

Test apparatus 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 and nozzle needle motion.

A.2 Apparatus description and assembly

A.2.1 Combustion chamber, as illustrated in Figure A.1, consisting of a stainless steel chamber of capacity 0,213 l ± 0,002 l, with heaters (A.2.1.1), and equipped with temperature sensor ports, a pressure sensor port, inlet and exhaust servo-valves and designated combustion chamber coolant areas.

A.2.1.1 Heaters, cartridge heaters embedded in the combustion chamber walls.

A.2.1.2 Combustion chamber valves, pneumatically driven inlet and outlet valves permitting charging of the combustion chamber with compressed air and the release of the combustion gases.

A.2.2 Fuel injection system, comprising all components required for repeatable injection of fuel into the combustion chamber. It includes a sensor to detect the exact moment of fuel injection.

A.2.2.1 Fuel reservoir, floating piston type of stainless steel, mounted on top of the fuel injection pump, with appropriate fittings to connect to the nitrogen gas supply.

A.2.2.2 Fuel reservoir piston, with a fuel-compatible O-ring to allow movement of the piston and to prevent direct contact between fuel and the pressurizing gas during the test.

A.2.2.3 Fuel injection pump, pneumatically-driven, consisting of:

A.2.2.3.1 Heating system, to heat and control the temperature of the injection pump.

A.2.2.3.2 Surge reservoir, to minimize pressure fluctuations during actuation of the injection pump.

A.2.2.4 Fuel injector assembly, comprising an inward-opening, pintle-type injector nozzle tip, a nozzle opening adjustment screw and lock nut, and a mechanism to permit the sensing of the injector nozzle needle movement.

A.2.2.4.1 Injector nozzle, calibrating nozzle, delay pintle-type, meeting the requirements of ISO 4010.

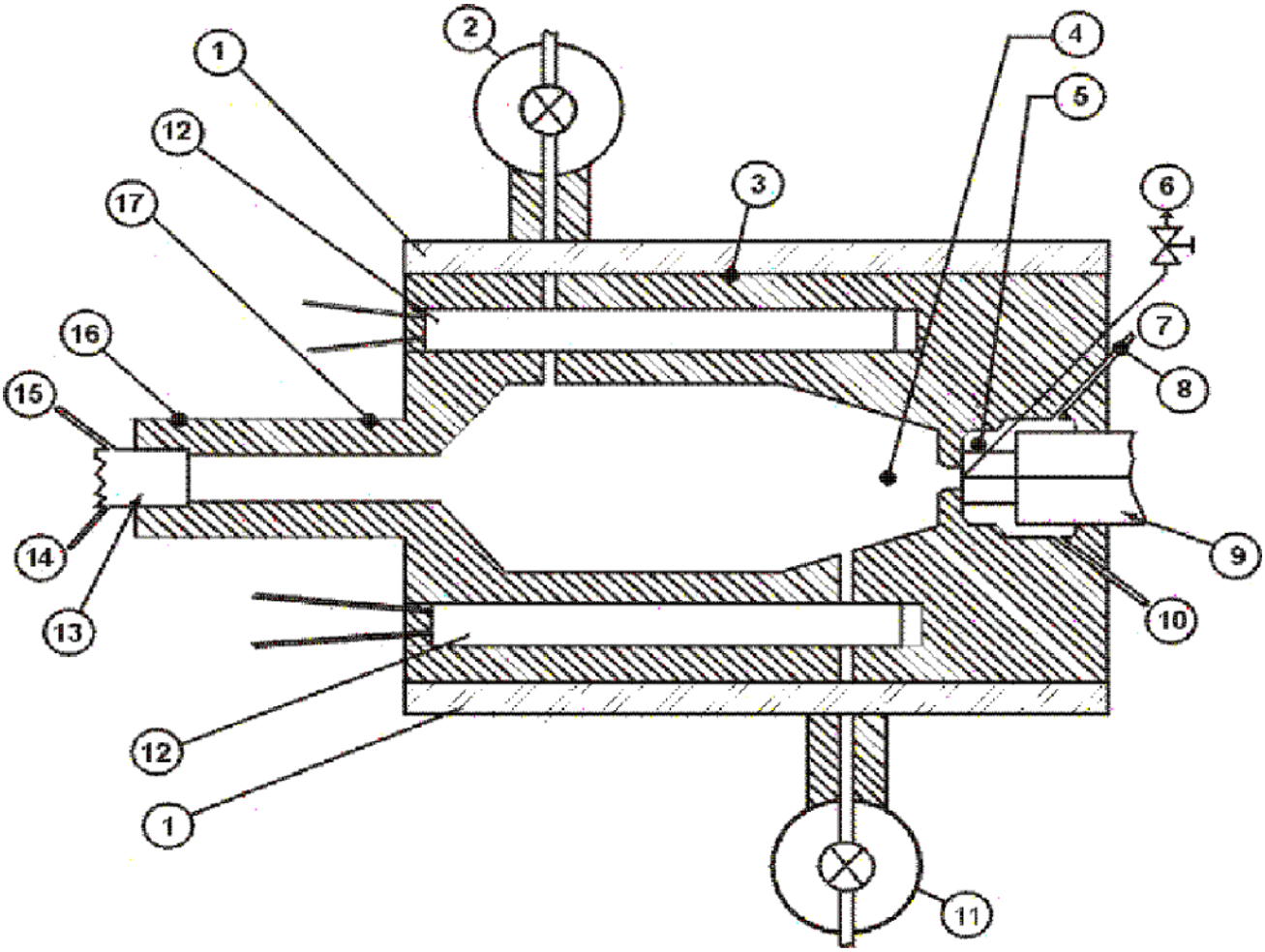
A.2.2.4.2 Pressure adjusting nut, set to release fuel in conformance with the conditions set out in the manufacturer's manual each time the nozzle assembly is reassembled and/or replaced, using an injection nozzle opening tester, preferably with a transducer to more accurately determine nozzle opening pressure.

A.2.2.4.3 Nozzle needle motion sensor, set with its sensing surface just before contact with the surface of the injector needle follower. Refer to the manufacturer's manual for exact details.

A.2.3 Combustion chamber air and injector coolant temperature sensors, installed at a specified depth using the supplied depth-setting tools.

NOTE Depth-setting instructions are given in the manufacturer's manual.

A.2.4 Cooling system, to serve as a heat transfer agent, to control and maintain the temperature of critical parts of the equipment, such as the injector nozzle and combustion chamber pressure sensor, and to serve as a safety against overheating of the system (see note under A.3.1).



Key

1. insulation blanket
2. inlet valve
3. combustion chamber outer surface temperature (T1)
4. charge air temperature (T4)
5. injector nozzle coolant passage temperature (T6)
6. hydrocarbon waste
7. coolant return
8. coolant return temperature (T7)
9. injector nozzle
10. coolant in
11. exhaust valve
12. combustion chamber heating elements
13. combustion chamber pressure sensor and coolant housing
14. coolant in
15. coolant return
16. combustion chamber pressure sensor temperature (T3)
17. temperature sensor used for diagnostics functions (T5)

Figure A.1 — Schematic of combustion chamber

A.3 Utilities

A.3.1 Electricity, consisting of a supply capable of delivering a current of 20 A. A back-up uninterruptible power supply (UPS), which provides power to the coolant system during power outage, is recommended to prevent damage of combustion chamber pressure sensor and high temperature gaskets.

A.3.2 Compressed air system, used to charge the combustion chamber, to drive the injection pump and to actuate the chamber valves.

A.3.2.1 Combustion charge air, used to charge the combustion chamber (see notes under 5.6), with a calibrated pressure sensor.

A.3.2.2 Actuating air, used for actuating the pneumatically-driven fuel injection pump and the combustion chamber inlet and exhaust valves. It is recommended to refer to the manufacturer's instruction manual for the correct pressure settings.

A.3.3 Inert gas system, nitrogen gas (5.8) to supply pressure to the fuel reservoir. It is recommended to refer to the manufacturer's instruction manual for the correct pressure setting.

A.3.4 Exhaust ventilation system, low (less than 125 Pa) suction pressure fume extraction system to dispose of exhaust gases.

NOTE The user is responsible for compliance with local regulations with regard to the safe disposal of exhaust gases.

A.4 Control and data acquisition

A.4.1 System control, enabling automatic control of the relevant system and sub-system devices. It is recommended to refer to the manufacturer's manual for a detailed description of the electronic control systems.

A.4.2 Data processing system, enabling collecting and processing all relevant signals from the temperature sensors, pressure sensors and nozzle needle lift sensor.

A.5 Auxiliary apparatus

A.5.1 Fuel reservoir piston tools, to insert and remove the fuel reservoir piston into and from the fuel reservoir.

A.5.2 Depth-setting tools

A.5.3 Other tools, refer to the manufacturer's manual.

Annex B
(normative)

Operational details in support to the standard test procedure

B.1 Fuel injection system flushing

B.1.1 The standard fuel sample reservoir as described in A.2.2.1 and Figure 1, items 8, 9 and 10 does not have a check valve.

B.1.1.1 If the fuel reservoir does not have a check valve, completely fill the fuel sample reservoir with filtered sample.

B.1.1.2 If the fuel sample reservoir is larger than the standard fuel sample reservoir, and has a check valve, fill the fuel sample reservoir with a volume of filtered sample that is at least equivalent to the volume of the standard fuel sample reservoir taking care to wet the walls of the reservoir during filling. Shake (by hand) the reservoir with its cap on (and stopper in cap) for at least 5 s.

NOTE All fuel sample reservoirs with a volume larger than the standard fuel sample reservoir, that also have a check valve, allow removal of the filled or partially filled reservoir from both the instrument and a filling/cleaning station.

B.1.2 If the fuel reservoir does not have a check valve and this part of the procedure is done with the fuel sample reservoir on the instrument, follow the following procedure (refer to the manufacturer's instructions for the details).

B.1.2.1 Flush the entire contents of the reservoir through the fuel injection system.

B.1.2.2 Use the compressed nitrogen supply to blow a sufficient amount of nitrogen through the fuel injection pump and injector body bleed valves to remove residual test sample from the fuel injection system.

B.1.3 If the fuel sample reservoir has a check valve, it may be filled in a well, ventilated location using a filling/cleaning station remote from the instrument. Then the following flushing procedure shall be followed (refer to the instructions provided by the manufacturer for the details).

B.1.3.1 Connect the reservoir to the filling/cleaning station and fill it as directed in B.1.1.2.

B.1.3.2 Flush a small volume of the filtered sample through the filling/cleaning station and refill the reservoir so that it again contains a volume of filtered sample at least equivalent to the volume of a standard fuel sample reservoir. Install the reservoir cap.

B.1.3.3 Remove the fuel sample reservoir from the filling station and install it onto the instrument.

B.1.3.4 Flush the entire contents of the fuel sample reservoir through the fuel injection system.

B.1.3.5 Use the compressed nitrogen supply to blow a sufficient amount of nitrogen through the fuel injection pump and injector body bleed valves to remove residual sample from the fuel injection system.

B.2 Fuel injection system filling and purging

B.2.1 If the fuel reservoir does not have a check valve, the following filling and purging procedure shall be followed (refer to the manufacturer's instructions for the details).

B.2.1.1 Fill the fuel sample reservoir with filtered sample as in B.1.1.1.

B.2.1.2 Re-Install the reservoir cap.

B.2.1.3 Reconnect the nitrogen line to the cap.

B.2.1.4 Open the nitrogen valve.

B.2.1.5 Pressurize the fuel injection system with compressed nitrogen to force a small volume of sample (± 10 ml) through the system to purge any air from the fuel injection system.

B.2.1.6 Remove the reservoir cap, and refill the reservoir to the required fuel level.

B.2.1.7 Insert the plunger, and re-install the reservoir cap.

B.2.2 If the fuel sample reservoir has a check valve, it may be filled in a well, ventilated location remote from the instrument and the following filling and purging procedure shall be followed (refer to the manufacturer's instructions for the details).

B.2.2.1 Fill the fuel sample reservoir by installing it on a filling/cleaning station as in B.1.1.2.

B.2.2.2 Remove the reservoir cap, insert the plunger and re-install the reservoir cap.

B.2.2.3 Install the filled reservoir onto the instrument.

B.2.2.4 Reconnect the nitrogen line to the cap.

B.2.2.5 Pressurize the fuel injection system with compressed nitrogen to force a small volume of sample (± 10 ml) through the system and purge the system of air.

B.2.3 The fuel system is now ready for the measurement procedure.

B.3 Test sequence

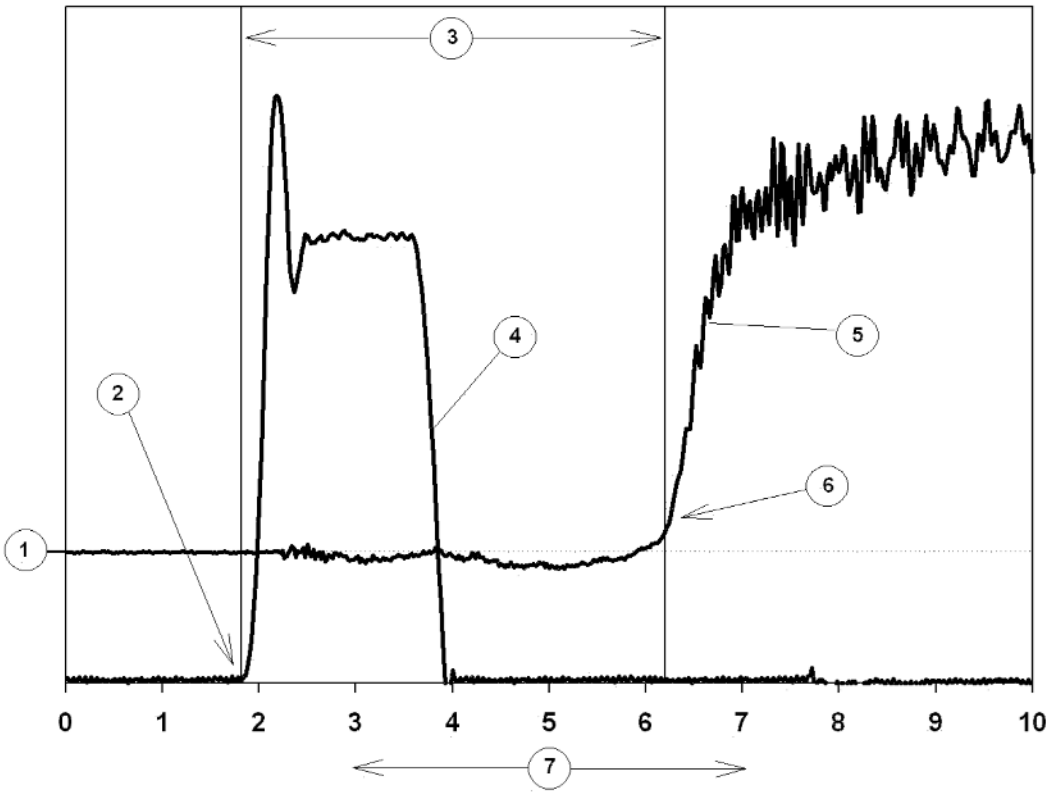
B.3.1 General

B.3.1.1 A complete automated test run consists of 15 preliminary (pre-injections) plus 32 subsequent (test injections) automated combustion cycles. A combustion cycle involves firstly charging the chamber, with compressed air, to the test pressure, then injecting a test portion of fuel into the heated combustion chamber, and finally, releasing the combustion gases. During the combustion cycle, the nozzle needle motion sensor measures the movement of the injector nozzle needle, and the combustion chamber pressure sensor measures the combustion chamber pressure.

B.3.1.2 The signals of the needle nozzle motion sensor and the combustion chamber pressure sensor define 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.

B.3.2 Test sequence

The first 15 combustion cycles are performed in order for the apparatus to attain equilibrium conditions. The ignition delays of the successive 32 combustion cycles are accumulated and then averaged to produce the analytical ignition delay result (see 11.5).



Key

1. initial chamber pressure
2. start of Injection
3. ignition delay time
4. nozzle needle movement
5. combustion chamber pressure
6. start of combustion
7. time (milliseconds)

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

B.3.3 Data record

During each of the 32 combustion test cycles, the following parameters shall be recorded:

a) ignition delay (*ID*);

b) derived cetane number (*DCN*)

c) charge air pressure (P2);

d) injection actuator air pressure (P3);

e) charge air temperature (*T*4);

f) combustion chamber pressure sensor temperature (*T*3);

g) injector nozzle coolant passage temperature (*T*6).

h) coolant return temperature (T7);

i) combustion chamber air back temperature (T9);

j) fuel injection pump temperature (T2).

NOTE 1 The individual values of the above parameters, together with their average, minimum and maximum, are usually automatically reported by the equipment data system output.

NOTE 2 Most instruments calculate and record 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 11.4).

B.4 Fuel injection system cleaning

B.4.1 Discharge any unused specimen from the fuel sample reservoir, and clean the fuel injection system.

B.4.1.1 If the fuel sample reservoir does not have a check valve, blow a sufficient amount of nitrogen from the compressed nitrogen system to remove unused sample from the reservoir and fuel injection system. Refer to manufacturer's instructions for the details of this procedure.

B.4.1.2 If the fuel sample reservoir has a check valve, the following cleaning procedure shall be followed (refer to manufacturer's instructions for the details).

B.4.1.2.1 Remove the reservoir from the instrument and connect it to the filling/cleaning station.

B.4.1.2.2 Use compressed nitrogen to flush all residual sample from the reservoir.

B.4.1.2.3 Blow a sufficient amount of nitrogen from the compressed nitrogen system, using the fuel system flushing adaptor, through the fuel injection system to remove unused sample from the system.

B.4.1.3 The apparatus and fuel system are now prepared for the next test method sequence, which includes flushing and purging the fuel injection system prior to testing (see 11.1 and 11.2).

B.5 Alternative fuel injection system cleaning

B.5.1 This procedure shall be used for unused specimen, after fuel samples containing 2-EHN cetane improver at either unknown concentrations or concentrations greater than 2 000 µl/l that have just been tested.

B.5.1.1 If the test sample contains 2 ethyl hexylnitrate (commonly called cetane improver or 2-EHN), at a concentration greater than 2 000 µl/l the cleaning procedure in B.4 can be insufficient. Discharging unused sample and cleaning the reservoir and fuel injection system after these samples includes use of either toluene or n-heptane solvent.

B.5.2 Discharge any unused sample from the fuel sample reservoir and fuel system and follow steps according to B.4.1.1 or B.4.1.2, depending on whether the reservoir has a check valve.

B.5.3 If the fuel sample reservoir does not have a check valve, the following discharging procedure is then to be followed (refer to manufacturer's instructions for the details),

B.5.3.1 Completely fill the reservoir with toluene or n-heptane.

B.5.3.2 Slowly flush the entire contents of the fuel sample reservoir through the fuel injection system, taking a minimum of 2 minutes to complete the flushing.

B.5.3.3 Using the compressed nitrogen supply, blow a sufficient amount of nitrogen through the reservoir and fuel injection system to remove residual toluene or n-heptane from the system.

B.5.4 If the fuel sample reservoir has a check valve, the following discharging procedure is then to be followed (refer to manufacturer's instructions for the details)

B.5.4.1 Connect the reservoir to the filling/cleaning station.

B.5.4.2 Fill the reservoir with a volume of toluene or heptane that is at least equivalent to the volume of the standard fuel reservoir.

B.5.4.3 Flush a small amount of solvent through the filling/cleaning station, then add enough solvent to restore the original volume in the reservoir.

B.5.4.4 Remove the fuel sample reservoir from the filling/cleaning station and shake it with its cap on (and stopper in cap) for 5 seconds to completely wet the walls of the reservoir.

B.5.4.5 Connect the fuel sample reservoir to the instrument.

B.5.4.6 Slowly flush the entire contents of the fuel sample reservoir through the fuel injection system, taking a minimum of 2 min to complete the flushing.

B.5.4.7 Using the compressed nitrogen supply, blow a sufficient amount of nitrogen through the reservoir and fuel injection system to remove residual toluene or n-heptane from the system.

B.5.5 The fuel injection system is now prepared for the next test method sequence, which includes flushing and purging the fuel injection system prior to testing. (See 11.1 and 11.2).

Annex C
(informative)

Apparatus maintenance

C.1 General

This annex does not deal in detail with the maintenance and repair procedures. For further detail, refer to the manufacturer's manual, and failing that, to the manufacturer. The quality of the test result is particularly dependent upon the care used in inspection and adjustment.

C.2 Daily maintenance

C.2.1 Check that the connection between the combustion chamber pressure sensor cable and the combustion chamber pressure sensor is tight, and has not become loosened by vibration.

C.2.2 Check the operation of the temperature acquisition system.

C.2.3 Check the sealing of the coolant system.

C.3 Weekly maintenance

Verify that the torque of the three brass nuts of the end cap is as specified in the manufacturer's manual. Adjust as necessary.

C.4 Yearly maintenance

Verify and calibrate the data acquisition system.

Annex D
(informative)

Equation outside scope of method

The conversion equation for derived cetane number outside the ignition delay range 2,8 ms to 6,3 ms is:

$$DCN = 83{,}99(ID\text{-}1{,}512)^{(-0{,}658)} + 3{,}547 \quad \text{(D.1)}$$

There is no precision for this equation for derived cetane number outside the range of 2,8 ms to 6,3 ms.

NOTE The equation was derived from a correlation test programme, comprising ASTM National Exchange Group (NEG) check fuels, heptamethylnonane, cetane and in-house check fuel [5].

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