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Standard Test Method for Determination of Indicated Cetane Number (ICN) of Diesel Fuel Oils using a Constant Volume Combustion Chamber— Reference Fuels Calibration Method¹

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1. Scope

1.1 This test method covers the quantitative determination of the indicated cetane number (ICN) of conventional diesel fuel oils, and diesel fuel oils containing cetane number improver additives; it is applicable to products typical of Specification [D975](#), Grades No.1-D and 2-D diesel fuel oils, European standard EN 590, and Canadian standards CAN/CGSB-3.517 and CAN/CGSB-3.520. The test method is also applicable to biodiesel, blends of diesel fuel oils containing biodiesel material (for example, materials as specified in Specifications [D975](#), [D6751](#), [D7467](#) and European standards EN 14214, EN 16734, and EN 16709), diesel fuels from non-petroleum origin, hydrocarbon oils, diesel fuel oil blending components, aviation turbine fuels, and polyoxymethylene dimethyl ether (OME).

1.2 This test method utilizes a constant volume combustion chamber (CVCC) with direct fuel injection into heated compressed air. The apparatus is calibrated using blends of reference fuels. ICN is determined directly from ignition delay using an instrument specific reference fuel calibration curve.

1.3 This test method and its precision cover the calibrated range of 35 ICN to 85 ICN, inclusive. The analyzer can measure ICN outside the calibrated range, but the precision has not been determined.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Some specific hazards statements are given in Section 7 on Hazards.*

¹ This test method is under the jurisdiction of ASTM Committee [D02](#) on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee [D02.01](#) on Combustion Characteristics.

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1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

- [D613 Test Method for Cetane Number of Diesel Fuel Oil](#)
- [D975 Specification for Diesel Fuel Oils](#)
- [D1193 Specification for Reagent Water](#)
- [D3703 Test Method for Hydroperoxide Number of Aviation Turbine Fuels, Gasoline and Diesel Fuels](#)
- [D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter](#)
- [D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)
- [D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants](#)
- [D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products](#)
- [D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products](#)
- [D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)
- [D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants](#)
- [D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material](#)
- [D6751 Specification for Biodiesel Fuel Blend Stock \(B100\) for Middle Distillate Fuels](#)
- [D7467 Specification for Diesel Fuel Oil, Biodiesel Blend \(B6 to B20\)](#)

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

2.2 European Standards:³

EN 590 Automotive fuels—Diesel—Requirements and test methods

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

EN 16709 Automotive Fuels—High FAME diesel fuel (B20 and B30)—Requirements and test methods

EN 16734 Automotive Fuels—Automotive B10 Fuel—Requirements and test methods

2.3 Energy Institute Standards:⁴

IP 41 Ignition quality of diesel fuels—Cetane engine test method

IP 617 Determination of indicated cetane number (ICN) of fuels using a constant volume combustion chamber—Primary reference fuels calibration (PRFC) method

2.4 Canadian Standards:⁵

CAN/CGSB-3.517 Diesel Fuel—Specification

CAN/CGSB-3.520 Automotive Low-Sulphur Diesel Fuel Containing Low Levels of Biodiesel Esters (B1-B5)—Specification

2.5 ISO Standards:⁶

ISO 868 Plastics and ebonite—Determination of indentation hardness by means of a durometer (Shore hardness)

ISO Guide 35 Certification of reference materials—General statistical principles (Reference materials—Guidance for the characterization and the assessment of the homogeneity and stability of the material).

3. Terminology

3.1 Definitions:

3.1.1 *cetane number, n*—a measure of the ignition performance of a diesel fuel oil obtained by comparing it to reference fuels in a standardized engine test. **D4175**

3.1.1.1 *Discussion*—In the context of this test method, cetane number is measured and reported by Methods **D613/IP 41**.

3.1.2 *quality control (QC) sample, n*—for use in quality assurance programs to determine and monitor the precision and stability of a measurement system, a stable and homogeneous material having physical or chemical properties, or both, similar to those of typical samples tested by the analytical measurement system; the material is properly stored to ensure sample integrity, and is available in sufficient quantity for repeated, long term testing. **D6299**

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *analyzer, n*—an integrated compression ignition apparatus to measure the ignition and combustion characteristics of diesel fuel oil.

3.2.2 *calibration and verification fluids, n*—volumetric blends of n-hexadecane and 1-methylnaphthalene, at 20 °C, define an ICN scale in specific volume ratios according to the relationship shown in **Eq 1**.

3.2.2.1 *Discussion*—Seven blends of n-hexadecane and 1-methylnaphthalene are used to create an analyzer specific calibration curve. A single blend is used to create a verification fluid.

3.2.3 *calibration curve, n*—plot of ID versus the ICN (see **3.2.6**) of reference fuel blends obtained by making ignition delay measurements with calibration fluids for each analyzer.

3.2.3.1 *Discussion*—The calibration curve comprises seven calibration points covering the 35 ICN to 85 ICN range; see research report⁷ for the format of the equation used.

3.2.3.2 *Discussion*—Calibration curves are different for each analyzer and can change each time calibration is carried out.

3.2.4 *combustion charge air, n*—compressed air at a specified pressure introduced into the combustion chamber.

3.2.5 *ignition delay (ID), n*—period of time, in milliseconds, between the start of fuel injection and the start of combustion.

3.2.5.1 *Discussion*—In the context of this standard, this period is represented by the mean of ID₀ and ID₁₅₀.

3.2.5.2 *ID₀, n*—the time in milliseconds (ms) between the start of fuel injection and the point where the relative pressure recovers to 0 kPa, as shown in **Fig. A3.1**.

3.2.5.3 *ID₁₅₀, n*—the time in milliseconds (ms) between the start of fuel injection and the point where the relative pressure reaches 150 kPa, as shown in **Fig. A3.1**.

3.2.5.4 *Discussion*—Start of fuel injection is interpreted as the rise in the electronic signal that opens the injector for the time given in Table 2; timings for ID₀ and ID₁₅₀ commence at this start point.

3.2.5.5 *Discussion*—IDs are recorded but not reported as they are converted into ICN by an instrument specific calibration curve.

3.2.6 *indicated cetane number (ICN), n*—measure of the ignition performance of a diesel fuel obtained by comparing it to reference fuels that have been blended to a scale; where 0 and 100 are represented by 1-methylnaphthalene and n-hexadecane respectively, to create a calibration curve.

3.2.6.1 *Discussion*—It is in principle a number indicated from a calibration curve that has been generated on the analyzer under test using reference fuel blend calibration points. The calibration curve, ICN = function of ignition delay (ID); see research report⁷ for the format of the equation used. The scale is defined by the relationship shown in **Eq 1**:

$$\text{indicated cetane number} = \% \text{ n-hexadecane (volume fraction)} \quad (1)$$

for any blends of n-hexadecane and 1-MN.

3.2.7 *injection time, n*—the period of time, in microseconds (μs), that the fuel injector nozzle is open as determined by the

³ Available from European Committee for Standardization (CEN), Rue de la Science 23, B-1040, Brussels, Belgium, <http://www.cen.eu>.

⁴ Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., <http://www.energyinst.org>.

⁵ Available from Canadian General Standards Board (CGSB), 11 Laurier St., Phase III, Place du Portage, Gatineau, Quebec K1A 0S5, Canada, <http://www.tpsgc-pwgsc.gc.ca/ongc-cgsb>.

⁶ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁷ Research Report reference IP 617 (ILS): Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., <http://www.energyinst.org.uk>.

length of the electronic signal (injection pulse), in microseconds, that opens the injector.

3.2.8 *reference fuels*, *n*–1-methylnaphthalene and *n*-hexadecane.

3.3 Abbreviations:

3.3.1 *ICN*—indicated cetane number

3.3.2 *ID*—ignition delay

3.3.3 *I–MN*—reference fuel 1-methylnaphthalene

3.3.4 *QC*—quality control

4. Summary of Test Method

4.1 A sub-sample of the sample under test is automatically drawn from a sample vial located in the auto-sampler carousel and heated during pressurization. At the start of a combustion cycle, a small specimen of the sub-sample is injected into a temperature and pressure controlled, constant volume combustion chamber, which has previously been charged with compressed air of a specified quality. Each injection, and its resulting combustion, causes a rapid pressure rise in the combustion chamber that is detected by the dynamic pressure sensor.

4.2 The complete test sequence comprises a cleaning stage and multiple combustion cycles (see Section 13, A3.1.5, and A3.1.5.1) to obtain ignition delay (ID) values. The ICN result is determined using the mean of the combustion cycles' IDs, and the reference fuel calibration curve.

4.3 Each analyzer is calibrated with seven fluids created from blends of reference fuels, with known ICNs calculated from the ICN scale; test results outside the calibration range are determined by extrapolating the calibration curve, but are subject to increased uncertainty.

5. Significance and Use

5.1 The ICN value determined by this test method provides a measure of the ignition characteristics of diesel fuel oil used in compression ignition engines.

5.2 This test can be used by engine manufacturers, petroleum refiners, fuel producers and in commerce as a specification aid to relate or match fuels and engines.

5.3 The relationship of diesel fuel ICN determinations to the full scale, variable speed, variable load diesel engine is not completely understood.

5.4 This test can be applied to non-conventional diesel fuels.

5.5 This test determines ICN; it requires a sample of approximately 40 mL and a test time of approximately 25 min.

5.6 This test method is based on the Energy Institute Test Method IP 617.

6. Interferences

6.1 Effects of UV light. Minimize exposure of sample fuels, reference fuels, calibration and verification fluids, and QC samples to sunlight or fluorescent lamp UV emissions to minimize induced chemical reactions that can affect the ignition delay measurements.

6.1.1 Exposure of these fuels and materials to UV wavelengths shorter than 550 nm for a short period of time can significantly affect ignition delay measurements.

NOTE 1—The formation of peroxide and free radicals can affect ignition delay. These formations are minimized when the fuel sample is stored in the dark in a cold room at a temperature of less than 10 °C and covered by a blanket of inert gas.

6.2 A limited study (see Appendix X1) demonstrated that an unusually high purity (99 %) reference fuel (I–MN) used for calibration can result in a positive relative bias of 0.3 ICN at a level of 50 ICN.

7. Apparatus

7.1 *Automatic Auto-sampling Analyzer*⁸—The apparatus as shown in Fig. A2.1 and outlined in 7.1.1, 7.1.2, 7.1.3, and 7.1.4, is described in more detail in Annex A2. For the installation and set-up procedures, and for a detailed system description, refer to the manufacturer's instructions.

7.1.1 *Electronics*, comprising power supplies (including an integral uninterruptable power supply) for the programmable logic controllers, data acquisition units, and associated interfaces.

7.1.2 *Combustion Chamber Assembly*, comprising the heated constant volume combustion chamber, cooled dynamic pressure sensor as well as regulating and shut-off valves, and temperature and pressure sensors.

7.1.2.1 *Combustion Chamber*, a stainless steel combustion chamber of capacity 0.390 L ± 0.010 L.

7.1.3 *Common Rail Injection System*, comprising a cooled piezoelectric injector, high pressure piston pump, heated piping, and temperature and pressure sensors.

7.1.4 *Auto Sampler/Carousel*, providing space for up to 36 sample vials and cleaning fluid.

7.2 *Sample Vials*, 40 mL (nominal) headspace vials with screw caps and silicone/PTFE septa or natural rubber/PTFE septa, approximately 1.3 mm thick, Shore A hardness of approximately 45 (see ISO 868). The PTFE side of the septum shall be on the inside of the vial.

7.2.1 The vials shall be amber or brown glass to help protect against the effects of UV light.

7.3 *Syringe Filter*, disposable, 25 mm to 28 mm diameter with a nominal pore size of 0.45 µm or less, PTFE filter media, to be attached onto a syringe (7.4).

7.4 *Syringe*, disposable, ≥20 mL plastic, suitable for use with a syringe filter (7.3).

7.5 *Recirculating Cooler*, capable of recirculating coolant to the injector assembly and the dynamic pressure sensor and maintaining a bath temperature of 65 °C ± 5 °C.

7.6 *Computer*, for inputting and outputting data, printing functions and interfacing with the analyzer and networks.

⁸ The sole source of supply of the analyzer described in this method known to the committee at this time is Stanhope-Seta, London Street, Chertsey, Surrey KT16 8AP UK. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

8. Reagents and Materials

8.1 Calibration and Verification Fluids:

8.1.1 Calibration and verification fluids are available commercially. They comprise volumetric blends of reference fuels n-hexadecane and 1-methylnaphthalene prepared in accordance with Eq 1 to create fluids with known ICNs within 0.5 ICN of the required values to an accuracy of ± 0.01 ICN.

8.1.2 The ICN number of the blend shall be determined by mass measurements and density (Test Method D4052 or equivalent) to provide volumetric equivalence. The volumes of reference fuels used shall be accurately dispensed and recorded by volume so that the validity of the ICN determination can be checked by comparison with the mass/density calculation. A difference of greater than 0.2 ICN between the volumetric and mass measurement methods shall be considered suspect and the blending process started again. If the discrepancy persists check the calibration of the measuring devices.

8.1.2.1 The ICNs determined by the mass/density volumetric equivalence shall be used for calibration and verification.

NOTE 2—At least 40 mL of each calibration or verification fluid is required.

8.1.3 Calibration and verification fluids (8.1), shall also meet the following requirements:

8.1.3.1 Be in accordance with ISO Guide 35.

NOTE 3—ISO Guide 35 permits and clarifies the use of reference materials from a single source.

8.1.3.2 Be prepared from reference fuels that have been checked, immediately before the blending process, for the presence of hydroperoxides using Test Method D3703 or an equivalent national standard. A result ≥ 1.0 mg/kg indicates the hydroperoxide level is too high, and that a new sample from a different batch shall be obtained and tested. (**Warning**—When blending reference fuels, take appropriate safety precautions in keeping with the relevant safety data sheets, such as using a fume hood.)

NOTE 4—Hydrocarbons can form peroxides and other free radical formed contaminants that can influence the ID. Reference fuels meeting the purity specification can contain peroxides and other free radical formed contaminants.

8.1.3.3 Be packaged into sealed vials of less than 100 mL volume under an inert gas blanket.

8.1.3.4 A record of reference fuel batch numbers, hydroperoxide content, and blending details shall be kept for QC purposes.

NOTE 5—Commercially available calibration and verification fluids,⁹ blended from reference fuels that have been analysed for purity and the presence of hydroperoxides, and conform to 8.1 are available.

8.1.4 Calibration and verification fluids shall be stored in a dark cool place to avoid possible effects of UV light. They shall be used within 24 h of opening the sealed vial.

⁹ The sole source of supply of the commercially available calibration and verification fluids described in this method known to the committee at this time is Stanhope-Seta, London Street, Chertsey, Surrey KT16 8AP UK. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

8.1.5 *Calibration Fluids*—A set of seven volumetric blends of reference fuels n-hexadecane and 1-methylnaphthalene, with known ICNs as shown in Table 1 covering the range 35 ICN to 85 ICN, inclusive.

8.1.6 *Verification Fluid*—A volumetric blend of reference fuels n-hexadecane and 1-methylnaphthalene, with a known ICNs. The ICN of each verification fluid shall be different to those shown in Table 1 by at least 1 ICN and be made from different batches of reference fuels.

8.1.7 *n-hexadecane*, minimum purity of 99.0 % (volume fraction) with a certificate of analysis, the designated 100 ICN component. Hydroperoxide level shall be ≤ 1.0 mg/kg as measured by Test Method D3703 (see 8.1.3.2).

8.1.7.1 Store n-hexadecane in a dark cool place to avoid possible effects of UV light and temperature effects. n-hexadecane solidifies at temperatures below approximately 18 °C and can require warming, to ensure it is homogeneous, before use. For sampling and handling, follow the guidelines of Section 9.

NOTE 6—n-hexadecane meeting the requirements of 8.1.7 is known to be commercially available.

8.1.8 *1-methylnaphthalene*—(also known as alpha-methylnaphthalene), minimum purity of 97.0 % (volume fraction) with a certificate of analysis, the designated 0 ICN component. Hydroperoxide level shall be ≤ 1.0 mg/kg as measured by Test Method D3703 (see 8.1.3.2). (**Warning**—Flammable. Vapor harmful. Vapor may cause flash fire, skin irritant.)

8.1.8.1 Store 1-methylnaphthalene in a dark cool place to avoid possible effects of UV light and temperature effects.

8.2 *Quality Control (QC) Sample*—Stable and homogenous distillate fuel, similar to the materials under test. For sampling and handling, see Section 9.

8.3 *Combustion Charge Air*—Compressed air containing 20.9 % \pm 0.5 % (volume fraction) oxygen with the balance nitrogen, less than 0.003 % (volume fraction) hydrocarbons, and less than 0.025 % (volume fraction) water and capable of delivering a regulated pressure of 2.5 MPa to 3.0 MPa, inclusive.

8.3.1 Air compressed locally can meet the above requirements. Follow the manufacturer's instructions regarding filtration and cleanliness of the air to meet the requirements. (**Warning**—Compressed gas under high pressure that supports combustion.)

NOTE 7—The oxygen content of combustion charge air can vary between batches (cylinders). Significant variation, even within the allowed tolerance, can lead to changes in ignition delay (higher oxygen content leads to shorter ignition delays).

8.4 *Cleaning Fluid*—1-decanol, minimum purity 99.0 %, filtered through a 0.45 μ m, or less, PTFE media filter.

8.5 *Water*—Unless otherwise specified, meeting the requirements of Specification D1193 or a similar national or international standard.

TABLE 1 ICN of Calibration Fluids

ICN	35.00	40.00	46.00	53.00	60.00	70.00	85.00

8.6 *Coolant System Fluid*—30:70 (volume fraction) mixture of commercial grade ethylene glycol-type, aluminium compatible, with algae inhibitor, vehicle radiator antifreeze with water (8.5).

8.7 *Inert Gas*—Argon or nitrogen, for use as a blanket over calibration and verification fluids, and samples.

9. Sampling Handling and Preparation

9.1 Sampling:

9.1.1 Collect diesel fuel oil samples in accordance with Practices **D4057** or **D4177**.

9.1.2 Refer to Practice **D5854** for appropriate information relating to the mixing and handling of test samples.

9.1.3 To minimize exposure to UV emissions that can induce chemical reactions and affect ignition delay measurement, collect and store samples in containers that are constructed of materials that minimize light reaching the sample, such as dark brown/amber bottles or epoxy lined metal containers or minimally-reactive plastic containers. If samples are collected in containers that do not minimize light reaching the sample, they shall be wrapped or boxed in light-proof containers immediately after filling. If the sample is not to be analyzed within 24 h, retain in a dark, cool environment, and preferably under an inert gas (see Interferences, Section 6).

NOTE 8—The formation of peroxides and free radicals, which affect the ignition delay, is minimized when the sample is stored in the dark, under an inert gas blanket, in a cool environment. Exposure of petroleum fuels to UV wavelengths for even a short period of time has been shown to affect ignition delay.

9.2 Test Sample Preparation:

9.2.1 *Sample Fuel Temperature*—Condition the diesel fuel oil sample before opening the storage container, so that it is at room temperature, typically 18 °C to 32 °C.

9.2.1.1 Fuel sample shall be homogeneous before testing. If in doubt, fuel temperature shall be raised at least 14 °C above the fuel's cloud point.

9.2.2 Collect the sample in a dark brown or amber bottle, suitable metal can or nonreactive plastic container.

9.2.3 Filter at least 40 mL of sample through the syringe filter (7.3) using the syringe (7.4) at ambient temperature, without vacuum, into a new brown/amber sample vial (7.2) and immediately fit a new screw cap and septum (PTFE side inside the vial).

10. Basic Apparatus Settings and Standard Operating Conditions

10.1 *Apparatus Installation*—Follow the manufacturer's instructions regarding apparatus assembly and installation. **Annex A2** gives more details of the apparatus.

10.2 System Start-up and Warm-up:

10.2.1 Open the valve at the source of the combustion charge air supply and adjust the pressure regulator as needed to provide the required 2.5 MPa to 3.0 MPa pressure (see 8.3).

10.2.2 Follow the manufacturer's instructions to switch on power to the analyzer. This activates the electronic components in the analyzer and the uninterruptable power supply that supplies the recirculating cooler's (7.5) pump.

10.2.3 Check that the computer and the recirculating cooler are switched on.

10.2.4 Sample vials may be loaded into the carousel at any time.

10.2.5 Immediately after switch on, sample details may be entered into the computer program.

10.2.6 After the chamber wall and chamber air temperatures have stabilized, the analyzer can commence testing samples in the carousel automatically.

10.2.7 For more details and in case of error messages, refer to the manufacturer's instructions.

10.2.8 Run a test using a QC sample at the beginning of each new operating period, before commencing routine tests.

10.3 Standard Operating and Test Conditions:

10.3.1 The operation and test conditions are pre-set and are not user changeable. Sensors, and their tolerances, used to measure these parameters are listed in **Annex A2** as well as other sensors used for diagnostic and safety purposes.

10.3.2 Key operator and test conditions are shown in **Table 2**. If these conditions are not met, then follow the manufacturer's instructions.

11. Calibration, Verification, and Quality Control Testing

11.1 *General*—Calibration uses seven reference fuel blended fluids (see 8.1.1 to 8.1.6). Verification uses one or more reference fuel blended fluids (see 8.1.1 to 8.1.6).

11.2 Reference Fuel Calibration: /astm-d8183-18

11.2.1 Calibrate the analyzer after one of the following:

11.2.1.1 Installation and commissioning.

11.2.1.2 Replacement or adjustment of critical parts or components of the combustion chamber assembly, fuel injection system, temperature or pressure sensors.

11.2.1.3 When quality control or verification checks indicate a calibration is required and the reasons for non-compliance have been suitably addressed; see manufacturer's recommended checks.

11.2.2 Follow the manufacturer's instructions to select reference fuel calibration mode and load the seven calibration fluids into the carousel and enter details of the calibration fluids' ICNs into the computer. The details of the calibration fluids' ICNs can be preloaded into the computer.

TABLE 2 Key Operating and Test Conditions

Key from Fig. A2.1	Parameter	Limits
C14	Chamber static pressure during cleaning	Nominally 1.75 MPa
	Chamber static pressure before each of 12 test injections	1.75 MPa ± 0.01 MPa
T4 and T5	Chamber air temperature (Indicated)	580 °C
	Stability before each injection	± 0.4 °C
P1	Injection Pressure	100.0 MPa ± 1.5 MPa
	Injection time (quartz crystal timed electronic pulse) See Fig. A3.1	1500 μs ± 1 μs

11.2.3 Before commencing calibration ensure that the cleaning fluid reservoir is completely filled with cleaning fluid.

11.2.4 The calibration procedure includes the automatic flushing of the injector lines and cleaning of the injector using the cleaning fluid (8.4).

11.2.5 In the automatic calibration process, all calibration fluids are tested twice. The IDs for each of the calibration fluids are averaged and used to enable a calibration curve of ID versus ICN to be automatically computed for use during normal testing.

11.2.6 The calibration curve is automatically checked for “fit quality” using the R-square statistic (coefficient of determination), with a pass criteria of ≥ 0.998 . Follow the manufacturer’s instructions, before attempting another calibration, if the criterion is not met.

11.2.7 The equation of the calibration curve, the associated IDs and ICNs are available although not required by the test method; follow the manufacturer’s instructions, if desired, to access this data.

11.3 Apparatus Verification:

11.3.1 Verify the apparatus performance by using a verification fluid with a ICN closest to the typical ICN of fuels being tested. Do not use an ICN value listed in Table 1. The apparatus is operating within specification if the ICN test result is within $R/\sqrt{2}$ of the ICN of the verification fluid being used, where R is the reproducibility of the test method.

11.3.2 Verify the apparatus after one of the following:

11.3.2.1 Calibration.

11.3.2.2 When the QC checks are out of tolerance.

11.3.3 Follow the Preparation of Apparatus and Procedure (10 and 12) to test the verification fluid. In the event that the verification test result falls outside of the performance stated in 11.3.1, follow the manufacturer’s instructions regarding diagnostic procedures, before performing a new reference fuel blend calibration.

11.3.3.1 In the event that the verification test results fall within the performance requirements stated in 11.3.1 but the QC checks are out of tolerance, follow the manufacturer’s instructions regarding diagnostic procedures. If the QC checks remain out of tolerance then perform a new reference fuel blend calibration.

11.4 Quality Control (QC):

11.4.1 Proper quality control procedures, such as those described in Practice D6299 or equivalent, shall be in place to ensure continuous satisfactory operation of the analyzer. Quality control samples (8.2) shall be tested at intervals and records shall be kept of the results.

11.4.2 Carry out quality control measurements on one or more quality control samples at least before each new operating period before commencing routine tests, after every replacement of the combustion charge air cylinder and after calibration.

11.4.2.1 When replacing the combustion charge air supply, take into account the volume of the external combustion charge air lines connected to the instrument. Extremely long high pressure air lines can result in many tests being completed before the new air reaches the apparatus.

11.4.2.2 In continuous use, it is good practice to perform a QC check at least every 10 samples.

11.4.2.3 Follow the Preparation of Apparatus and Procedure (Section 10 and 12) to test the quality control sample.

11.4.3 Whenever QC sample determinations are not in statistical control, as determined by Practice D6299 or equivalent, carry out corrective action starting by following the manufacturer’s instructions to run a test using the cleaning fluid (8.4). If the use of the cleaning fluid does not bring the QC sample back into statistical control then continue with apparatus verification (11.3) and then reference fuel calibration (11.2) if necessary.

12. Procedure

12.1 Ensure that the analyzer meets the requirements for calibration, verification, and quality control given in Section 11 and preparation of apparatus given in Section 10.

12.2 Ensure that the sample in the sample vial has been prepared in accordance with the requirements for sample handling and test sample preparation given in Section 9.

12.3 Follow the manufacturer’s instructions to fill the sample vial with sample, to input the sample details, and to place the vial in the auto-sampler’s carousel.

12.4 Follow the manufacturer’s instructions to start the test, which continues to the end of the test automatically. If there are further samples in the carousel these can be tested without operator intervention.

NOTE 9—Annex A3 describes the automatic procedures carried out by the apparatus during the test.

13. Calculation

13.1 The 12 combustion cycle IDs are statistically analysed by Peirce’s Criterion¹⁰ for outliers. If there are one or more outliers, a 13th combustion cycle is carried out. If there are 12 valid measurements out of the 13, then the outlier is removed. If the 13 measurements contain two or more outliers then a 14th combustion cycle is carried out. However if there are still remaining outliers, they are removed. The remaining IDs are averaged (see A3.1.5 and A3.1.5.1). If the number of valid measurements is less than 10, the whole test shall be repeated. The ICN result is obtained automatically from the reference fuel calibration curve.

14. Report

14.1 Report the following information:

14.1.1 A reference to this standard,

14.1.2 The complete identification of the product tested,

14.1.3 The ICN result rounded to the nearest 0.1,

14.1.4 Any deviation, by agreement or otherwise, from the procedures specified, and

14.1.5 The date of the test.

15. Precision and Bias

15.1 *Precision*—The precision statements were derived from a 2016 interlaboratory cooperative test program using

¹⁰ Ross, S., “Peirce’s Criterion for the Elimination of Suspect Experimental Data, *J. Engr. Technology*, Fall 2003.