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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[1]

This standard is issued under the fixed designation D7668; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers the quantitative determination of the derived cetane number of conventional diesel fuel oils, diesel fuel oils containing cetane number improver additives, and is applicable to products typical of Specification D975, Grades No.1-D and 2-D regular, low and ultra-low-sulfur diesel fuel oils, European standard EN590, and Canadian standards CAN/CGSB-3.517 and CAN/CGSB3.6. The test method may be applied to the quantitative determination of the derived cetane number of biodiesel, blends of diesel fuel oils containing biodiesel material (for example, Specifications D975, D6751, and D7467), and diesel fuel oil blending components.

1.2 This test 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 sample. An equation converts the ignition delay and the combustion delay determined from the dynamic pressure curve to a derived cetane number (DCN).

1.3 This test method covers the ignition delay ranging from 1.9 ms to 25 ms and combustion delay ranging from 2.5 ms to 160 ms (30 DCN to 70 DCN). However, the precision stated only covers the range of DCN from 39 to 67.

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.*

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
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)
E456 Terminology Relating to Quality and Statistics

2.2 *EN Standards:*[3]

EN590 Automotive Fuels—Diesel—Requirements and Test Methods

[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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[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.

[3] Available from European Committee for Standardization. Central Secretariat: rue de Stassart, 36,B-1050 Brussels, Belgium.

*A Summary of Changes section appears at the end of this standard

2.3 *Energy Institute Standards:*[4]
IP41 Ignition Quality of Diesel Fuels—Cetane Engine Test Method

2.4 *Canadian Standards:*[5]
CAN/CGSB-3.517 Regular Sulphur Diesel Fuel—Specification
CAN/CGSB 3.6 Automotive Low-Sulphur Diesel Fuel—Specification

2.5 *DIN Standards:*[6]
DIN 73372 Einspritzdüsen Grösse T und U

3. Terminology

3.1 *Definitions:*

3.1.1 *accepted reference value (ARV), n*—a value that serves as an agreed-upon reference for comparison and that is derived as *(1)* a theoretical or established value, based on scientific principles, *(2)* an assigned value, based on experimental work of some national or international organization, such as the U.S. National Institute of Standards and Technology (NIST), or *(3)* a consensus value, based on collaborative experimental work under the auspices of a scientific or engineering group. **E456**

3.1.1.1 *Discussion*—In the context of this method, accepted reference value is understood to apply to the ignition delay and the combustion delay of specific reference materials determined under reproducibility conditions by collaborative experimental work.

3.1.2 *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**

3.1.2.1 *Discussion*—In the context of this test method, cetane number is that defined by Test Method D613/IP41.

3.1.3 *check standard, n*—in QC testing, a material having an accepted reference value used to determine the accuracy of a measurement system.

3.1.3.1 *Discussion*—In the context of this test method, check standard refers to the calibration reference material.

3.1.4 *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 *calibration reference material, n*—a pure chemical or a specified mixture of pure chemicals having an assigned ignition delay accepted reference value and an assigned combustion delay accepted reference value.

3.2.2 *chamber wall temperature, n*—temperature, in °C, of the combustion chamber wall.

3.2.3 *charge air, n*—compressed synthetic air at a specified pressure introduced into the combustion chamber at the beginning of each test cycle.

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

3.2.5 *combustion delay (CD), n*—that period of time, in milliseconds (ms), between the start of fuel injection and mid-point of the combustion pressure curve.

3.2.5.1 *Discussion*—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 chamber static 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.

3.2.6 *derived cetane number (DCN), n*—a number calculated using a conversion equation to determine a cetane number.

3.2.6.1 *Discussion*—The conversion equation relates a measured ignition delay or ignition delay and combustion delay from a combustion analyzer, to a cetane number.

3.2.7 *ignition delay (ID), n*—that period of time, in milliseconds (ms), between the start of fuel injection and the start of combustion as determined using the specific combustion analyzer applicable for this test method.

3.2.7.1 *Discussion*—In the context of this test method, start of fuel injection is interpreted as the rise in the electronic signal that opens the injector; combustion is interpreted as the part of the pressure curve generated during the combustion cycle when significant (+0.02 MPa above the chamber static pressure) and sustained increase in rate-of-change in pressure, as measured by a pressure sensor in the combustion chamber.

3.2.8 *injection period, n*—the period of time, in microseconds (µs), that the fuel injector nozzle is open as determined by the length of the electronic signal, in microseconds, that opens the injector.

3.2.9 *operation period, n*—the time, not to exceed 12 h, between successive calibration or QC testing, or both, of the combustion analyzer by a single operator.

3.3 *Abbreviations:*

3.3.1 *ARV*—accepted reference value

3.3.2 *CD*—combustion delay

3.3.3 *CN*—cetane number

3.3.4 *DCN*—derived cetane number

3.3.5 *ID*—ignition delay

3.3.6 *QC*—quality control

4. Summary of Test Method

4.1 A small specimen of sample is injected into a heated, temperature-controlled, constant volume chamber, which has

[4] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

[5] Available from the Canadian General Standards Board, Sales Centre, Gatineau, Canada, K1A1G6. www.ongc-cgsb.ca.

[6] Available from Beuth Verlag GmbH (DIN-- DIN Deutsches Institut fur Normung e.V.), Burggrafenstrasse 6, 10787, Berlin, Germany, http://www.en.din.de.

previously been charged with compressed 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.[7] The remaining ID's and CD's are averaged to produce the two independent results. An equation converts the average ID result and the average CD result into a DCN.

5. Significance and Use

5.1 The ID and CD values and the DCN 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 and marketers, and in commerce as a specification aid to relate or match fuels and engines.

5.3 The relationship of diesel fuel oil DCN determinations to the performance of full-scale, variable-speed, variable-load diesel engines is not completely understood.

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

5.5 This test determines ignition characteristics and requires a sample of approximately 370 mL and a test time of approximately 30 min using a fit-for-use instrument.

6. Interferences

6.1 **Warning—**Minimize exposure of sample fuels, calibration reference materials, QC samples, and check standards to sunlight or fluorescent lamp UV emissions to minimize induced chemical reactions that can affect the delay measurements.[8]

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 radicals can affect ignition delay measurement. These formations are minimized when the sample or material is stored in the dark in a cold room at a temperature of less than 10 °C and covered by a blanket of nitrogen.

7. Apparatus

7.1 *General*—This test method uses an integrated automated analytical measurement system[9] comprised of:

7.1.1 *Combustion Chamber*—A cylindrical chamber having a volume of 0.473 L ± 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.

7.1.2 *Fuel Injection System*—A high pressure sample, generated using a hydraulic pump and pressure multiplier, is delivered to a commercial electronic diesel fuel injector. A sample reservoir supplies the pressure multiplier with sample to ensure proper and repeatable injection of calibration, QC material, and test specimens into the combustion chamber. The system includes:

7.1.2.1 *Fuel Sample Reservoir*—A metal reservoir having a nominal volume of 200 mL.

7.1.2.2 *Hydraulic Pump*—Capable of producing fuel pressures up to 19 MPa.

7.1.2.3 *Pressure Multiplier*—10:1 ratio.

7.1.2.4 *Fuel Injector*—A solenoid-based common rail diesel fuel injector from Bosch with the part number 0445110181 (Annex A6).

7.1.2.5 *Safety Burst Disk*—Relieves the high pressure if the sample pressure exceeds 180 MPa. The burst disk is attached to the high pressure sample system manifold block opposite the injector.

7.1.2.6 *Flush Valve*—High pressure air actuated valve used to exchange samples.

7.1.3 *Coolant System*—A closed loop circulating coolant system to control the temperature of the combustion injector nozzle and dynamic pressure sensor. The system includes an auxiliary heat exchanger with built-in circulating pump and flow control valves.

7.1.4 *Instrument Sensors*—Sensors used to measure and either indicate the value of a variable or transmit the condition for control or data acquisition purposes such as:

7.1.4.1 *Combustion Chamber Static Pressure Sensor*—A calibrated sensor installed to correct the temperature offset of dynamic pressure sensor.

7.1.4.2 *Combustion Chamber Dynamic Pressure Sensor*—A calibrated sensor installed to measure the pressure within the combustion chamber.

7.1.4.3 *Sample Pressure Sensor*—A calibrated sensor installed to measure the pressure of the sample injected into the combustion chamber.

[7] Ross, Stephen, "Peirce's Criterion for the Elimination of Suspect Experimental Data," *Journal of Engineering Technology*, Fall 2003.

[8] Supporting data, "Sunlight and Air Exposure Effects on Octane Number or Cetane Number of Petroleum Product Samples," have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1502.

[9] The sole source of supply of the analyzer described in this method known to the committee at this time is PAC LP, 8824 Fallbrook Drive, Houston, TX 77064. 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.

7.1.4.4 *Nitrogen Pressure Sensor*—A sensor installed to measure the inlet pressure from the nitrogen regulator.

7.1.4.5 *Combustion Chamber Inner Wall Temperature Sensor*—Type K thermocouple with a stainless steel sheath.

7.1.4.6 *Injector Nozzle Cooling Jacket Temperature Sensor*—Type K thermocouple with stainless steel sheath, inserted in the injector nozzle coolant passage.

7.1.5 *Computerized Control, Data Acquisition, Data Analysis and Reporting System*—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 analyzer and subsystem component functions and collects and processes all relevant signals from the temperature and pressure sensors.

7.2 *Instrument Schematic*—A schematic of the instrument is reproduced in Annex A4.

7.3 *Compressed Gas Pressure Regulators:*

7.3.1 *Charge Air Regulator*—A two-stage regulator capable of controlling the downstream pressure to a minimum pressure of 2.2 MPa.

7.3.2 *Nitrogen Regulator*—A two-stage regulator capable of controlling the downstream pressure to a minimum pressure of 0.7 MPa.

8. Reagents and Materials

8.1 *Calibration Reference Material:*

8.1.1 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 of:

8.1.1.1 *Hexadecane*—With a minimum purity of 99.0 volume percent. (**Warning—**Combustible. Vapor harmful.)

8.1.1.2 *2,2,4,4,6,8,8-Heptamethylnonane*—With a minimum purity of 98.0 volume percent. (**Warning—**Combustible. Vapor harmful.)

8.1.1.3 For peroxide-free material, the assigned ID_{ARV} is 2.96 ms and the assigned CD_{ARV} is 4.90 ms.

NOTE 2—Hydrocarbons can form peroxides and other free radically formed 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 4Å molecular sieves.

8.1.2 *Methylcyclohexane (MCH)*—With a minimum purity of 99.0 volume percent. The assigned ID_{ARV} for this material is 11.00 ms and the assigned CD_{ARV} for this material is 17.00 ms. (**Warning—**Flammable. Vapor harmful. Vapor may cause flash fire.)

NOTE 3—Hydrocarbons can form peroxides and other free radically formed contaminants that can influence the ID and CD. Experience has found some MCH meeting the purity specification but which does not meet the ID_{ARV} or CD_{ARV}. It is recommended that new material be qualified prior to use.

8.2 *Check Standard:*

8.2.1 *Calibration Reference Material*—40:60 mixture by weight of hexadecane and 2,2,4,4,6,8,8-heptamethylnonane (see 8.1). (**Warning—**Combustible. Vapor harmful.)

8.2.2 *Quality Control Sample*—A stable and homogeneous diesel fuel oil having physical and chemical properties similar to those of typical sample fuels routinely tested (**Warning—**Combustible. Vapor harmful.)

8.3 *Charge Air*—A compressed synthetic air mixture containing 20.0 ± 0.5 volume percent oxygen with the balance nitrogen, less than 0.003 volume percent hydrocarbons, and less than 0.025 volume percent water. It is suggested that a quality control test be performed after an air cylinder has been changed (**Warning—**Compressed gas under high pressure that supports combustion.)

8.4 *Compressed Nitrogen*—Compressed nitrogen having a minimum purity of 99.9 volume percent (**Warning—**Asphyxiant. Compressed gas under high pressure.)

8.5 *Coolant System Fluid*—A 50:50 volume mixture of water and commercial ethylene glycol-based antifreeze (**Warning—**Poison. Maybe harmful or fatal if inhaled or swallowed.)

8.5.1 *Antifreeze*—A commercial automotive cooling system ethylene glycol-based solution.

8.5.2 *Water*—A distilled or reagent-grade, conforming to Specification D1193, Type IV.

8.6 *Heptane*—(*n*-Heptane) with a minimum purity of 99.5 volume percent. (**Warning—**Flammable. Vapor harmful. Vapor may cause flash fire.)

9. Sampling and Test Specimen Preparation

9.1 *Sampling:*

9.1.1 Collect diesel fuel oil samples in accordance with Practices D4057 or D4177. (**Warning—**Collect and store diesel fuel oil samples in a suitable container such as a dark brown glass bottle, a metal can, or a minimally reactive plastic container to minimize exposure to UV emissions.)

9.1.2 Refer to Practice D5854 for appropriate information relating to the mixing and handling of diesel fuel oil samples.

9.2 *Test Specimen 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 temperature should be raised at least 14 °C above the fuel's cloud point. Fuel sample should be homogeneous before testing.

NOTE 4—Give consideration to the fuel composition related to sample temperature to avoid the loss of lower boiling components that may affect the DCN value.

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

10. Basic Apparatus Settings and Standard Operating Conditions

10.1 Operation of the combustion analyzer, associated equipment, instrumentation, and computer system requires setting a series of testing variables to prescribed specifications.

Some of these settings are established by component specifications, others are operating conditions that are monitored or controlled by the computer software or by operator adjustment.

10.2 *Settings Based on Component Specifications:*

10.2.1 *Combustion Chamber Leakage Rate*—Shall be less then 0.75 kPa/s, as measured during the automated check of the sealing integrity of the combustion chamber.

NOTE 5—The computer system initiates an automatic diagnostic procedure consisting of zero-adjustment of the chamber dynamic pressure sensor and a chamber sealing integrity check.

10.3 *Standard Operating Conditions:*

10.3.1 *Chamber Static Pressure*—The average Chamber Static Pressure for the 15 combustion cycles is required to be within 2.00 MPa ± 0.02 MPa.

10.3.2 *Chamber Wall Temperature,* 560 °C to 640 °C.

10.3.2.1 The Chamber wall temperature is initially set by the manufacturer. The temperature set-point is monitored and controlled by the computer. Adjustment of the controller set-point is required, in accordance with the calibration procedure.

10.3.2.2 The average wall temperature for the 15 combustion cycles is required to be within ±0.2 °C of the set point temperature.

10.3.3 *Injector Nozzle Coolant Jacket Temperature*—Set the coolant reservoir temperature to achieve an injector nozzle coolant passage temperature of 50 °C ± 2 °C. This is determined and recorded by the computer. A temperature outside the range given during a 15 combustion cycle measurement indicates a possible malfunctioning of the cooling system.

10.3.4 *Injection Pressure*—Set by the manufacture to 100 MPa. An individual injection does not occur unless the high pressure sample sensor measures 100 MPa ± 1.5 MPa. If the sample pressure is outside the tolerance limit the hydraulic pressure is adjusted and the injection process is re-initiated. If an appropriate sample pressure is not found after 5 adjustments of the hydraulic pressure the test is aborted and the user is warned of the fault.

10.3.5 *Injection Period*—Set by the instrument using the computer controlled calibration process. The injection period is limited to the range from 2000 µs to 2700 µs.

11. Calibration and Quality Control Testing

11.1 *Calibration*—Calibrate the combustion analyzer: *(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 chamber static pressure, or chamber dynamic pressure sensors, or *(4)* whenever check standard or QC sample determinations are not in statistical control, and the assignable causes for QC non-compliance have been suitably addressed.

11.2 *Pre-calibration Procedure:*

11.2.1 Open the valve at the source of the charge air supply and adjust the pressure regulator as needed to provide the specification pressure. Open the valve at the source of the nitrogen supply and adjust the pressure regulator as needed to provide the specification pressure. Turn on the circulation coolant system.

11.2.2 Position the combustion analyzer power switch to ON and warm-up the combustion analyzer. After the chamber wall temperature has stabilized a chamber leakage test will be performed to determine the chamber leakage rate. If the leakage test fails, a warning is issued.

11.2.3 Clean the sample system (see Annex A2).

11.3 *Hexadecane/Heptamethylnonane Calibration Procedure*—The calibration reference material is tested to affirm that the combustion chamber wall temperature and the sample injection period settings produce ignition delay measurements for this material that are within specification limits.

11.3.1 To ensure homogeneity the calibration reference material CRM) must be above 20 °C. Agitate the calibration reference material before use.

11.3.2 Remove the sample reservoir cap and wash the stem and threads and the sample reservoir with approximately 50 mL of the calibration reference material. Reinstall the sample reservoir cap.

11.3.3 Flush the entire aliquot of the calibration reference material through the fuel injection system by pressing the Flush button. Refer to the instruction manual of the manufacturer.

11.3.4 Charge the instrument with the calibration reference material (at least 160 mL) and wipe the stem and threads of the sample reservoir cap with a clean dry towel and secure the sample reservoir cap to the sample reservoir.

11.3.5 Perform the automatic calibration procedure.

11.3.5.1 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 cause a change in the combustion chamber wall temperature or the sample injection period is adjusted by the computer to inject the appropriate quantity of sample into the combustion chamber, or both. The automatic calibration procedure performed by the processor controlling the instrument is summarized in Annex A5.

NOTE 6—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.

11.3.5.2 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.

11.3.6 The combustion analyzer calibration is complete when the calibration reference material average delays are within the specified acceptance limits of 2.96 ms ± 0.16 ms for ID and 4.90 ms ± 0.08 ms for CD.

11.3.7 Without flushing, refill the sample reservoir with the calibration reference material (CRM) and perform a single determination of the calibration reference material. The result must satisfy the acceptance limits of 2.96 ms ± 0.16 ms for ID and 4.90 ms ± 0.08 ms for CD. If the single determination exceeds the acceptance limits for either ID or CD, perform the calibration procedure again.

11.4 *Methylcyclohexane Calibration Procedure*—Perform two consecutive ignition delay and combustion delay measurements using methylcyclohexane. Perform the second determination by refilling the sample reservoir without flushing.

11.4.1 To pass the calibration test, each single result of the ID and CD measurements must be within 11.00 ms ± 1.30 ms and 17.00 ms ± 1.40 ms, respectively.

11.4.2 To pass the calibration test, the averaged result of the ID and CD measurements must be within 11.00 ms ± 1.10 ms and 17.00 ms ± 1.20 ms, respectively.

11.4.3 If any of the single results or the average results is outside the respective limits, system performance is unacceptable and it is recommended that diagnostic procedures be used to determine and remedy the problem before performing a new calibration. Refer to the instruction manual of the manufacturer.

11.4.4 The combustion analyzer calibration is complete when both the hexadecane/heptamethylnonane and methylcyclohexane datasets are acceptable.

11.5 *Quality Control (QC Testing)*—Conduct a regular statistical quality assurance (quality control) program in accordance with the techniques of Practice D6299 or equivalent.

11.5.1 This test method requires a quality control testing at the beginning of each operation period using a single determination for ID and CD for the calibration reference material or the DCN for at least one QC sample. In continuous use, the recommended QC interval is at least every ten samples.

11.5.2 The QC sample is a typical diesel fuel oil having an ignition delay that represents the primary range of use for the combustion analyzer.

11.5.2.1 If the combustion analyzer is used for testing fuel having a very wide range of ignition delays, it may be useful to have a second QC sample of a different DCN.

11.5.3 Conduct a QC test whenever there is a change from one charge air cylinder to another.

NOTE 7—The oxygen content of the new charge air cylinder may differ from that of the previous source and can have a significant effect on the delay measurements.

11.5.4 *Calibration Reference Material*—Perform a single measurement of the hexadecane/heptamethylnonane calibration reference material.

11.5.4.1 This determination is acceptable if it satisfies the limits protocol specified in Practice D6299 or equivalent.

11.5.4.2 Prior to having established ID and CD tolerances for the calibration reference material in accordance with Practice D6299 or equivalent, use the warning limits of ±0.13 ms and ±0.07 ms for ID and CD, respectively, and action limits of ±0.18 ms and ±0.10 ms for ID and CD, respectively, based on the single determination for the calibration reference material, as per 11.1.[10]

11.5.5 *QC Sample*—Perform a single measurement of the quality control sample.

11.5.5.1 This determination is acceptable if it satisfies the limits protocol specified in Practice D6299 or equivalent.

11.5.6 The combustion analyzer is deemed fit for use when the calibration reference material result or the quality control standard result are acceptable. If the measurement results are not acceptable, conduct a calibration before performing additional sample measurements.

12. Procedure

12.1 *Operating Procedure:*

12.1.1 With the combustion analyzer in shutdown mode, start a new operating period as follows:

12.1.1.1 Open the valve at the source of the charge air supply and adjust the pressure regulator as needed to provide the specification pressure. Open the valve at the source of the nitrogen supply and adjust the pressure regulator as needed to provide the specification pressure. Turn on the circulation coolant system.

12.1.1.2 Position the combustion analyzer power switch to ON and warm-up the combustion analyzer. After the chamber wall temperature has stabilized a chamber leakage test will be automatically performed by the instrument to determine the chamber leakage rate. If the leakage test fails, a warning is issued.

12.2 *Test Procedure:*

12.2.1 Remove the sample reservoir cap and wash the stem and threads and the sample reservoir with approximately 50 mL of the test specimen. Reinstall the sample reservoir cap.

12.2.2 Flush the entire test specimen through the fuel injection system by pressing the Flush button. Refer to the instruction manual of the manufacturer.

12.2.3 Fill the sample reservoir past the upper level sensor with the test specimen (at least 160 mL). Wipe the stem and threads of the reservoir cap with a clean, dry towel. Reinstall the sample reservoir cap.

12.2.4 Flush the entire test specimen through the fuel injection system by pressing the Flush button.

12.2.5 Remove the sample reservoir cap and refill the sample reservoir past the upper level sensor with the test specimen (at least 160 mL). Wipe the stem and threads of the reservoir cap with a clean, dry towel. Reinstall the sample reservoir cap.

12.2.6 Initiate an automatic ID and CD determination procedure using the appropriate computer commands. At the end of the test, a test output summary is automatically displayed on the computer screen. The user can optionally print the result with the printer, store the result in memory and export the result to an external memory device.

12.2.7 The delay results are obtained by averaging the ID and CD measurements of the last 15 cycles to get an average ID and an average CD. If either of the ID and CD pairs are identified as a statistical outlier according to Peirce's Criterion[7] that pair of ID and CD measurements are removed from the 15 measurements and are not included in calculating the average value. 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 injection are allowed.

12.2.8 Flush the remaining test specimen from the sample reservoir through the sample injection system by pressing the Flush button.

[10] Supporting data (the results of the 2010 Intralaboratory Ruggedness Test Program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1704.