

Designation: D 6890 – 03a

Standard Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber^{1,2}

This standard is issued under the fixed designation D 6890; 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 automated laboratory test method covers the quantitative determination of the ignition characteristics of conventional diesel fuel oil, oil-sands based fuels, blends of fuel containing biodiesel material, diesel fuel oils containing cetane number improver additives, and is applicable to products typical of ASTM Specification D 975 grades No. 1-D and 2-D regular and low-sulfur diesel fuel oils, European standard EN 590, and Canadian standards CAN/CGSB-3.517 and 3.6-2000. The test method may also be applied to the quantitative determination of the ignition characteristics of diesel fuel blending components.

1.2 This test method measures the ignition delay and utilizes a constant volume combustion chamber with direct fuel injection into heated, compressed air. An equation converts an ignition delay determination to a derived cetane number (DCN).

1.3 This test method covers the ignition delay range from 3.3 to 6.4 ms (60 to 33 DCN). The combustion analyzer can measure shorter and longer ignition delays, but precision may be affected.

1.4 The values stated in SI units are to be regarded as the 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 and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 613 Test Method for Cetane Number of Diesel Fuel Oil³

D 975 Specification for Diesel Fuel Oils⁴

- D 1193 Specification for Reagent Water⁵
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum ${\rm Products}^6$
- D 4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants⁶
- D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products 6
- D 5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products⁷
- D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance⁸
- D 6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants⁷
- D 6708 Practice for Statistical Assessment and Improvement of the Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material⁸
- E 456 Terminology Relating to Quality and Statistics⁹
- 2.2 ISO Standards:¹⁰
- ISO 4010 Diesel Engines—Calibrating Nozzle, Delay Pintle Type
- ISO 4259 Petroleum products—Determination and application of precision data in relation to methods of test
- 2.3 EN Standard:
- EN 590 Automotive Fuels—Diesel—Requirements and Test Methods¹¹
- 2.4 IP Standard:

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¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.01 on Combustion Characteristics.

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² This test method is based on IP PM CQ/2001, published in the IP Standard Methods for Analysis and Testing of Petroleum and Related Products and British Standard 2000 Parts. Copyrighted by The Institute of Petroleum, 61, New Cavendish Street, London, W1G 7AR. Adapted with permission of The Institute of Petroleum.

³ Annual Book of ASTM Standards, Vol 05.05.

⁴ Annual Book of ASTM Standards, Vol 05.01.

⁵ Annual Book of ASTM Standards, Vol 11.01.

⁶ Annual Book of ASTM Standards, Vol 05.02.

⁷ Annual Book of ASTM Standards, Vol 05.03.

⁸ Annual Book of ASTM Standards, Vol 05.04.

⁹ Annual Book of ASTM Standards, Vol 14.02.

¹⁰ Available from American National Standards Institute, 25 W. 43rd St., 4th floor, New York, NY 10036.

¹¹ Available from European Committee for Standardization. Central Secretariat: rue de Stassart, 36, B-1050 Brussels, Belgium.

- IP 41 Ignition Quality of Diesel Fuels—Cetane Engine Test Method¹²
- 2.5 Canadian Standards:
- CAN/CGSB-3.517-2000 Automotive Low Sulfur Diesel Fuel—Specification¹³
- CAN/CGSB 3.6-2000 Automotive Diesel Fuel— Specification¹³

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. **E 456**

3.1.1.1 *Discussion*—In the context of this method, accepted reference value is understood to apply to the ignition 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. **D 4175**

3.1.2.1 *Discussion*—In the context of this method, cetane number is that defined by Test Method D 613/IP 41.

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. **D 6299**

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

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. **D** 6299

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration reference material*, *n*—a pure chemical having an assigned ignition delay accepted reference value.

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

3.2.3 *charge air temperature*, *n*—temperature, in °C, of the air inside the combustion chamber.

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

3.2.5 *derived cetane number (DCN)*, *n*—a number calculated using a conversion equation that relates a combustion analyzer ignition delay result to cetane number.

3.2.6 *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.

3.2.6.1 *Discussion*—In the context of this test method, start of fuel injection is interpreted as the initial movement or lift of the injector nozzle needle as measured by a motion sensor; start of combustion is interpreted as that point in the combustion cycle when a significant and sustained increase in rate-of-change in pressure, as measured by a pressure sensor in the combustion chamber, ensures combustion is in progress.

3.2.7 *operating 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 CN-cetane number.
- 3.3.3 DCN-derived cetane number.
- 3.3.4 *ID*—ignition delay.
- 3.3.5 QC—quality control.

4. Summary of Test Method

4.1 A small specimen of diesel fuel oil is injected into a heated, temperature-controlled constant volume chamber, which has previously been charged with compressed air. Each injection produces a single-shot, compression ignition combustion cycle. ID is measured using sensors that detect the start of fuel injection and the start of significant combustion for each cycle. A complete sequence comprises 15 preliminary cycles and 32 further cycles. The ID measurements for the last 32 cycles are averaged to produce the ID result. An equation converts the ID result to a DCN.

5. Significance and Use

5.1 The ID and DCN values determined by this test method can provide a measure of the ignition characteristics of diesel fuel oil 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 may be applied to non-conventional fuels. It is recognized that the performance of non-conventional fuels in full-scale engines is not completely understood. The user is therefore cautioned to investigate the suitability of ignition characteristic measurements for predicting performance in full-scale engines for these types of fuels.

5.5 This test determines ignition characteristics and requires a sample of approximately 100 mL and a test time of approximately 20 min on a fit-for-use instrument.

6. Interferences

6.1 Minimize exposure of sample fuels, calibration reference materials, QC samples, and check standard to sunlight or

 $^{^{12}}$ Available from Institute of Petroleum, 61 New Cavendish St., London, W1G 7AR, U.K.

¹³ Available from the Canadian General Standards Board, Gatineau, Canada, K1A 1G6.

fluorescent lamp UV emissions to minimize induced chemical reactions that can affect ignition delay measurements.¹⁴

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

Note 1—The formation of peroxide and radicals can effect 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 system¹⁵ comprised of: (1) a constant volume compression ignition combustion chamber with external electrical heating elements, suitable insulation and pneumatically actuated intake and exhaust valves, (2) a heated, pneumatically actuated fuel injection system¹⁶ with pump, injector nozzle assembly, and associated sample reservoir, (3) a coolant system with a liquid-to-air heat exchanger, filter, circulating pump and flow control valves, (4) temperature thermocouples, pressure gages and sensors, an injector nozzle needle motion sensor, compressed gas pressure regulators, control valves, pneumatic actuator components, and solenoid valves, and (5) a computer to control test sequencing, acquire and accumulate sensor signal data, provide processing calculations, and automatically output a printed report of some important test parameters (see Fig. 1).

7.2 See Annex A2, Combustion Analyzer Equipment Description and Specifications, for detailed information.

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 Actuator Utility Compressed Air Regulator, a twostage regulator capable of controlling the downstream pressure to a minimum pressure of 1.3 MPa.

7.3.3 *Fuel Reservoir Utility Compressed Nitrogen Regulator*, a single or two-stage regulator capable of controlling the downstream pressure to a minimum pressure of 350 kPa.

7.4 Auxiliary Apparatus:

7.4.1 *Diesel Fuel Oil Sample Filter*, a single-use glass fiber, polytetrafluorethylene (PTFE), or nylon filter with a nominal pore size of 3 to 5 micrometers (μ m) for use with a glass syringe.

7.4.2 *Syringe*, a glass syringe of a minimum volume of 100 mL.

8. Reagents and Materials

8.1 Calibration Reference Materials:

8.1.1 *Heptane (n-heptane)*, with a minimum purity of 99.5 volume percent. The assigned ID_{ARV} for this material is 3.78 ms. (**Warning**—Flammable. Vapor harmful. Vapor may cause flash fire.)

8.1.2 *Methylcyclohexane*, with a minimum purity of 99.0 volume percent. The assigned ID_{ARV} for this material is 10.4 ms. (**Warning**—Flammable. Vapor harmful. Vapor may cause flash fire.)

8.2 Check Standard:

8.2.1 *Heptane (n-heptane)*, with a minimum purity of 99.5 volume percent. The assigned ID_{ARV} for this material is 3.78 ms. (**Warning**—Flammable. Vapor harmful. Vapor may cause flash fire.)

8.3 *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.)

8.4 Charge Air, compressed air containing 20.9 ± 1.0 volume percent oxygen, less than 0.003 volume percent hydrocarbons, and less than 0.025 volume percent water. For charge air cylinders supplied with a blend of oxygen and nitrogen, it is required that a quality control test be performed after an air cylinder has been changed. (Warning—Compressed gas under high pressure that supports combustion.)

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

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

8.5.2 *Water*, distilled or reagent-grade, conforming to Specification D 1193, Type IV.

8.6 Actuator Utility Compressed Air, oil free compressed air having less than 0.1 volume percent water supplied at a minimum sustained pressure of 1.5 MPa. (Warning— Compressed gas under high pressure that supports combustion.)

8.7 *Fuel Reservoir Utility Compressed Nitrogen*, compressed nitrogen having a minimum purity of 99.9 volume percent. (**Warning**—Compressed gas under high pressure.)

9. Sampling and Test Specimen Preparation

9.1 Sampling:

9.1.1 Collect diesel fuel oil samples in accordance with Practices D 4057 or D 4177.

9.1.1.1 Collect and store diesel fuel samples in a suitable container such as a dark brown bottle, a metal can, or a minimally reactive plastic container to minimize exposure to UV emissions.

9.1.2 Refer to Practice D 5854 for appropriate information relating to the mixing and handling of diesel fuel oil samples.

9.2 Test Specimen Preparation:

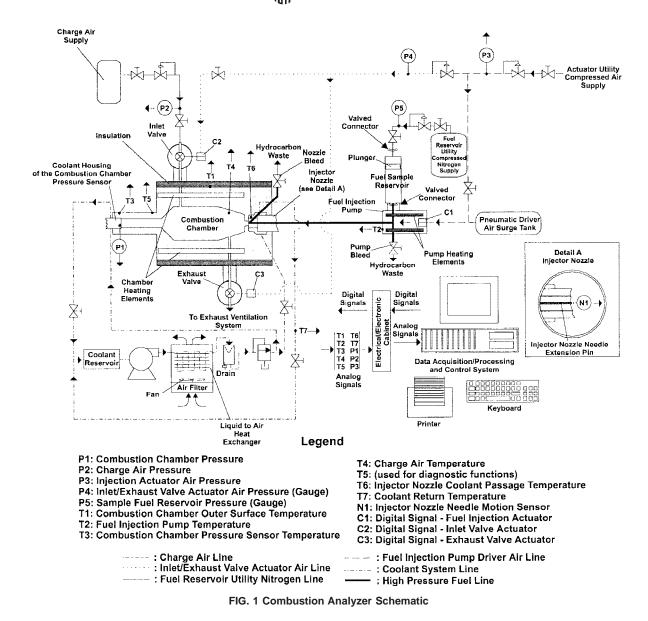
9.2.1 *Sample Fuel Temperature*—Condition the diesel fuel sample before opening the storage container, so that it is at room temperature, typically 18 to 32°C.

¹⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1502.

¹⁵ The sole source of supply of the combustion analyzer known to the committee at this time is Advanced Engine Technology Ltd. (AET), 17 Fitzgerald Road, Suite 102, Ottawa, Canada, K2H 9G1. 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.

¹⁶ The fuel injection system is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to the ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

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9.2.2 *Filtration*—Prepare a test specimen by filtering at least 100 mL of diesel fuel oil sample through a nominal 3 to 5 μ m porosity filter element using a glass syringe.

9.2.2.1 Collect the specimen in a dark brown bottle, metal can or minimally reactive plastic container.

10. Basic Apparatus Settings and Standard Operating Conditions

10.1 Installation of the apparatus requires placement on a level floor and connection of all utilities. Engineering and technical support for this function is required, and the user shall be responsible to comply with all local and national codes and installation requirements.

10.2 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/ controlled by the computer software or by operator adjustment.

10.3 Settings Based on Component Specifications:

10.3.1 Injector Nozzle Opening Pressure—Each time the nozzle assembly is reassembled or replaced, or both, set the pressure-adjusting nut to release fuel in conformance with the requirements in the manufacturer's equipment manual, using an injector nozzle tester. For additional details, refer to the instruction manual of the manufacturer.

10.3.2 *Injector Nozzle Motion Sensor Position*—Manually position the motion sensor while visually observing the nozzle needle movement signal on the computer monitor (see Fig. A4.1). The criteria for optimized setting are as follows:

10.3.2.1 The signal prior to the steep increase in needle lift is required to indicate some signal noise. If the signal trace is flat and constant, the motion sensor is too far away from the nozzle needle extension pin.

10.3.2.2 The peak of the steep increase in signal level is required to be visible on the computer monitor screen. If the signal peak is flat, the motion sensor is too close to the nozzle needle extension pin. For additional details, refer to the instruction manual of the manufacturer.

10.3.3 Injector Nozzle Coolant Passage Thermocouple Position—Proper positioning of the thermocouple in the injector nozzle coolant passage is set by installing a compression fitting nut and associated ferrule on the stainless steel sheath of the thermocouple, crimping the ferrule on the sheath using a specialized depth setting tool to establish the correct depth of penetration. For additional details, refer to the instruction manual of the manufacturer.

10.3.4 *Charge Air Thermocouple Position*—Proper positioning of the thermocouple in the combustion chamber is set by installing a compression fitting nut and associated ferrule on the stainless steel sheath of the thermocouple, crimping the ferrule on the sheath using a specialized depth setting tool to establish the correct depth of penetration. For additional details, refer to the instruction manual of the manufacturer.

10.3.5 *Combustion Chamber Pressure Drop Rate of Charge Air*, less than 3.5 kPa/s, as measured during the check of the sealing integrity of the combustion chamber (see A3.4).

10.4 Standard Operating Conditions:

10.4.1 Charge Air Pressure (P2), 2.137 \pm 0.007 MPa.

10.4.2 Charge Air Temperature (T4), $545 \pm 30^{\circ}$ C.

10.4.2.1 The difference in temperature $(T4_{max} - T4_{min})$ as determined and recorded by the computer, shall be less than 2.5°C during a 32 combustion cycle measurement determination.

10.4.3 Combustion Chamber Outer Surface Temperature (T1)—Initially set by the manufacturer, the surface temperature is monitored and controlled by the computer. Operator adjustment of the controller set-point is required, in accordance with the calibration procedure.

10.4.4 Combustion Chamber Pressure Sensor Temperature (T3), $130 \pm 20^{\circ}$ C.

10.4.4.1 The difference in temperature $(T3_{max} - T3_{min})$ as determined and recorded by the computer, shall be less than 8.0°C during a 32 combustion cycle measurement determination.

10.4.5 Coolant Return Temperature (T7), $40 \pm 10^{\circ}$ C.

10.4.6 *Fuel Sample Reservoir Pressure (P5)*, 345 ± 35 kPa. Visually check the gage reading, as this parameter is not recorded by the data acquisition system.

10.4.7 Fuel Injection Pump Temperature (T2), $35 \pm 3^{\circ}$ C. 10.4.8 Injector Nozzle Coolant Passage Temperature (T6)— The maximum ($T6_{max}$) and minimum ($T6_{min}$) temperatures as determined and recorded by the computer, shall be within $50 \pm 4.0^{\circ}$ C during a 32 combustion cycle measurement determination.

10.4.9 Injection Actuator Air Pressure (P3), 1.21 ± 0.03 MPa.

10.4.10 Inlet/Exhaust Valve Actuator Air Pressure (P4), 480 \pm 35 kPa. Visually check the gage reading, as this parameter is not recorded by the data acquisition system.

11. Calibration and Quality Control Testing

11.1 *Calibration*—Calibrate the combustion analyzer for only the following reasons: (I) after it is installed and commissioned, (2) once a week, (3) after replacement of critical parts or components of combustion chamber assembly

(see A2.2), fuel injection system (see A2.3) or instrument sensors (see A2.4), (4) after calibration of the data acquisition board, injection actuator air pressure sensor or charge air pressure sensor, (5) whenever check standard or QC sample determinations are not acceptable.

11.2 Precalibration Procedures:

11.2.1 Clean the combustion chamber pressure sensor assembly (see A3.3).

11.2.2 If necessary, start and warm-up the combustion analyzer (see A3.1).

11.3 Calibration Procedure—Two filtered calibration reference materials are tested: (1) heptane to affirm that the combustion chamber charge air temperature setting produces ignition delay measurements for this material that are within specification limits and, (2) methylcyclohexane to affirm that the measurement sensitivity of the combustion analyzer produces ignition delay measurements for this material that are within specification limits.

11.3.1 *Heptane Calibration Reference Material*—Perform three consecutive ignition delay determinations.

11.3.1.1 The average of three acceptable ID results is required to be within 3.78 \pm 0.01 ms.

11.3.1.2 If the average ID is outside the limits, the combustion chamber outer surface temperature controller set-point requires adjustment to cause a change in the combustion chamber charge air temperature.

Note 2—ID increases when the combustion chamber outer surface temperature decreases and vice versa.

11.3.1.3 If the temperature controller set-point adjustment from the previous setting, exceeds \pm 4°C, a system malfunction is suspected and diagnostic procedures to determine and remedy the problem are recommended. Refer to the instructions provided by the manufacturer.

Note 3—After a change of charge air cylinders that employ a blend of oxygen and nitrogen, a temperature controller set-point adjustment beyond 4°C can accommodate the extreme limits of the 20.9 \pm 1.0 volume percent oxygen in the blend.

11.3.1.4 After a temperature controller set-point adjustment, wait at least 10 min before initiating a new calibration so that the combustion analyzer attains thermal equilibrium.

11.3.1.5 To be an acceptable data set, each single result is required to be within 3.78 \pm 0.06 ms.

11.3.1.6 If any of the three results is outside the limits, a system malfunction is suspected and diagnostic procedures to determine and remedy the problem are recommended before performing a new calibration. Refer to the instructions provided by the manufacturer.

11.3.2 *Methylcyclohexane* Calibration Reference *Material*—Perform two consecutive ignition delay determinations.

11.3.2.1 To be an acceptable data set, each single result is required to be within 10.4 ± 0.6 ms and the average of the two results is required to be within 10.4 ± 0.5 ms.

11.3.2.2 If either of the two single results or the average of the two 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 instructions provided by the manufacturer.

11.3.3 The combustion analyzer calibration is complete when both heptane and methylcyclohexane data sets are acceptable.

11.4 *Quality Control (QC Testing)*—Conduct a regular statistical quality assurance (quality control) program in accordance with the techniques of Practice D 6299 or equivalent.

11.4.1 This test method requires quality control testing at the beginning of each operating period by a single ignition delay determination for both the check standard (heptane) and one QC sample.

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

11.4.2.1 If the combustion analyzer is used for testing fuels having a very wide range of ignition delay, it may be useful to have a second QC sample of a different ignition delay.

11.4.3 For locations using blends of oxygen and nitrogen as the source for charge air, conduct a QC test whenever there is a change from one cylinder to another.

Note 4—The oxygen content of the new oxygen and nitrogen blend may differ from that of the previous source and can have a significant effect on ID measurements.

11.5 *Check Standard*—Perform a single ignition delay determination for filtered heptane.

11.5.1 This determination is acceptable if it satisfies the limits protocol specified in Practice D 6299 or equivalent.

11.5.2 Prior to having established ignition delay tolerances for heptane in accordance with Practice D 6299 or equivalent, use warning limits of \pm 0.07 ms and action limits of \pm 0.106 ms, based on the average of the three acceptable ID results for heptane, as per 11.3.1.

Note 5—The warning and action limits for heptane were determined by analysis of round robin test data. 17

11.6 *QC Sample*—Perform a single ignition delay determination for the filtered QC sample.

11.6.1 This determination is acceptable if it satisfies the limits protocol specified in Practice D 6299 or equivalent.

11.7 The combustion analyzer is fit-for-use when both the check standard (heptane) and the QC sample ignition delay determinations are acceptable. If the ignition delay determination for either material is not acceptable, conduct a new calibration before performing further ignition delay determinations.

12. Procedure

12.1 Operating Period Procedure:

12.1.1 If necessary, warm-up the combustion analyzer (see A3.1).

12.1.2 Check the sealing integrity of the combustion chamber (see A3.4).

12.1.3 Check that the combustion analyzer is fit-for use by performing a quality control test (see 11.4).

 TABLE 1 Repeatability (r) and Reproducibility (R) for Ignition

 Delay (ID) and Derived Cetane Number (DCN)

	ID (ms)	DCN	
Repeatability (r) Reproducibility (R)	$\begin{array}{l} 0.0465 \times ({\rm ID-2.432}) \\ 0.0777 \times ({\rm ID-0.7679}) \end{array}$	0.76 0.0987 × (DCN − 17.67)	

12.2 Test Procedure:

12.2.1 Filter the diesel fuel sample at room temperature, using a glass syringe and single-use filter element, to prepare a test specimen of at least 100 mL.

12.2.2 Flush the fuel system with the specimen (see A3.2.2).

12.2.3 Fill and purge the fuel system with the specimen (see A3.2.3).

12.2.4 Initiate an automatic ignition delay determination using the appropriate computer command (see Annex A4 for detailed information about the test sequence).

12.2.4.1 During the test sequence the following equation is applied to convert ID to DCN:

$$DCN = 83.99(ID - 1.512)^{(-0.658)} + 3.547$$
 (1)

12.2.5 At the end of the test, a test output summary is automatically printed out. This printout contains detailed information about each of the 32 measurement combustion cycles including ID, DCN, and some of the important operating conditions. At the bottom of the printout, the average value, minimum, maximum, range, and standard deviation of all measurements are given (see Appendix X1).

12.2.5.1 The ID result at the bottom of the printout is obtained by averaging the ID measurements of the last 32 cycles.

12.2.5.2 The DCN result at the bottom of the printout is obtained by converting the ID result to DCN using Eq 1.

12.2.6 Check that all standard operating conditions are in compliance.

12.3 Discharge unused specimen and clean the fuel system (see A3.2.4) to prepare for (1) the next specimen determination, or (2) combustion analyzer shut down (see A3.5).

13. Report

13.1 Report the following information:

13.1.1 A reference to this standard,

- 13.1.2 The sample identification,
- 13.1.3 The date of the test,

13.1.4 The ID result to three significant figures,

13.1.5 The DCN result to the nearest tenth,

13.1.6 The test's average charge air temperature to the nearest tenth $^{\circ}\mathrm{C},$ and

13.1.7 Any deviation, by agreement or otherwise, from the specified procedures.

14. Precision and Bias

14.1 *General*—The precision statements for ID and DCN are based on an interlaboratory study conducted in 2002.¹⁸ The test results for the study were statistically analyzed using

¹⁷ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1532.

¹⁸ Supporting data, produced by equipment using software version 3.40a, have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1545.

	Information			
ID (ms)	Repeatability (r)	Reproducibility (R)		
3.2	0.036	0.189		
3.7	0.059	0.228		
4.2	0.082	0.267		
4.7	0.105	0.306		
5.2	0.129	0.345		
5.7	0.152	0.384		
6.2	0.175	0.422		
DCN	Repeatability (r)	Reproducibility (R)		
33	0.76	1.51		
40	0.76	2.20		
45	0.76	2.70		
50	0.76	3.19		
55	0.76	3.68		
60	0.76	4.18		
63	0.76	4.47		

TABLE 2 Repeatability and Reproducibility Values for Information

TABLE 3 Between Test Method Reproducibility

= (DCN + CN _{D613})/2	Reproducibility	
33	2.51	
40	3.65	
45	4.47	
50	5.29	
55	6.10	
60	6.92	
63	7.41	

ASTM Practice D 6300/ISO 4259 techniques and involved 10 laboratories and 15 test samples covering the ID range from 3.3 to 6.4 ms (DCN range from 60 to 33).

14.2 Precision:

14.2.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus, under constant operating conditions, on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the values in Table 1 only in one case in twenty.

14.2.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test materials, would, in the long run, and in the normal and the correct operation of the test method, exceed the values in Table 1 only in one case in twenty.

14.2.3 Examples of precision are shown in Table 2 for user information.

14.3 *Bias*—The ID determined using this test method has no bias because ID is defined only in terms of this test method.

14.4 *Relative Bias to Test Method D 613*—The degree of expected agreement between DCN results by this test method and CN results by Test Method D 613 has been assessed in accordance with Practice D 6708. The degree of agreement cannot be further improved using bias correction schemes considered in Practice D 6708.

14.4.1 Reproducibility Limit between a Single DCN Result versus a Single CN_{D613} Result:

14.4.1.1 As a consequence of sample-specific biases observed, the 95 % confidence limit on the differences between a single DCN result and a CN_{D613} result can be expected to be larger than the reproducibility of either test method.

14.4.1.2 Based on the results from the interlaboratory study, the difference between the single DCN result and a single CN_{D613} result, over the long-term and correct operation of both test methods, for any sample meeting the scope of both test methods, is estimated to exceed the values in Table 3 no more than one case in twenty.

NOTE 6—For average values between those listed linearly, interpolate between reproducibility figures in Table 3.

15. Keywords

15.1 cetane number; derived cetane number; diesel performance; ignition characteristic; ignition delay

ANNEXES

(Mandatory Information)

A1. HAZARDS INFORMATION

A1.1 Introduction

A1.1.1 In the performance of the standard test method there are hazards to personnel. These are indicated in the text. For more detailed information regarding the hazards, refer to the appropriate Material Safety Data Sheet (MSDS) for each of the applicable substances to establish risks, proper handling, and safety precautions.

A1.2 (Warning—Combustible. Vapor harmful.)

A1.2.1 Applicable Substances:

A1.2.1.1 Diesel fuel oil, and

A1.2.1.2 Quality control sample.

A1.3 (**Warning**—Flammable. Vapors harmful if inhaled. Vapors may cause flash fire.)

A1.3.1 Applicable Substances:

A1.3.1.1 Heptane, and

A1.3.1.2 Methylcyclohexane.

A1.4 (**Warning**—Poison. May be harmful or fatal if inhaled or swallowed.)

A1.4.1 Applicable Substances:

A1.4.1.1 Ethylene glycol based antifreeze.

A1.5 (**Warning**—Compressed gas under high pressure that supports combustion.)

A1.5.1 Applicable Substances:

A1.5.1.1 Compressed air.

A1.6 (Warning—Compressed gas under high pressure.)

A1.6.1 Applicable Substances:

A1.6.1.1 Compressed nitrogen.

A1.7 (Warning—Hot surfaces.)

A1.7.1 Applicable Substances:

A1.7.1.1 Protective cage enclosing the combustion chamber,

A1.7.1.2 Exposed areas of the combustion chamber around the injector nozzle, and

A1.7.1.3 Exposed areas of the combustion chamber near the combustion chamber inside the combustion chamber protective cage.

A2. COMBUSTION ANALYZER EQUIPMENT DESCRIPTION AND SPECIFICATIONS

A2.1 The combustion chamber assembly and fuel injection system are critical to the proper operation of this test method.

A2.2 *Combustion Chamber Assembly*—The principle component of this assembly, illustrated in Fig. A2.1, is a corrosion-protected metal cylindrical block that is precision machined and fabricated to include the following features:

A2.2.1 A cavity along a central axis of the body, having a volume of 0.213 \pm 0.002 L, that constitutes the compression ignition combustion chamber.

A2.2.2 An opening at one end of the chamber to accommodate insertion of the fuel injection nozzle assembly and which includes a passage for circulation of liquid coolant to control the injector nozzle temperature.

A2.2.3 An opening at the other end of the chamber, to accommodate insertion of a pressure sensor liquid-cooled housing.

A2.2.4 Two drilled ports or passages between the combustion chamber cavity and the external surface of the assembly to accommodate an inlet and an exhaust valve.

A2.2.5 Nine passages, drilled from the pressure sensor end of the block, parallel to the chamber axis, to accept individual electric heating elements.

A2.2.6 A series of wells or drilled passages to accommodate temperature sensor elements.

A2.2.7 An external insulation blanket to minimize heat loss from the block and improve heat distribution inside the combustion chamber cavity.

A2.2.8 An inlet valve assembly that includes a digital signal controlled solenoid valve to operate a pneumatically actuated, servo-type valve connected to the inlet port.

A2.2.9 An exhaust valve assembly that includes a digital signal controlled solenoid valve to operate a pneumatically actuated, servo-type valve connected to the exhaust port.

A2.2.10 Combustion Chamber Heating Elements, nine cartridge-type resistance heaters.

A2.3 *Fuel Injection System*,¹⁶ a patented, integrated assembly of components for proper and repeatable injection of calibration reference material, QC sample fuel, check standard, and test specimens into the combustion chamber. The system includes:

A2.3.1 *Fuel Sample Reservoir Assembly*, a corrosionprotected metal reservoir having a volume of 40 mL, a threaded cap, a fuel resistant, internal, floating plunger with fuel-resistant O-ring to separate the pressurizing gas from the fuel specimens, a quick-connect coupling on the cap for

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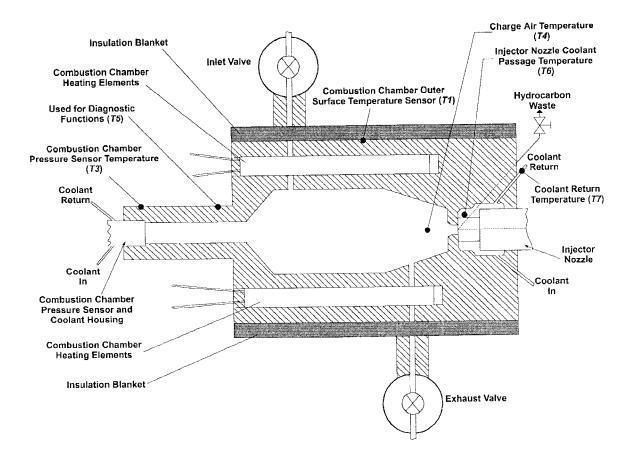


FIG. A2.1 Combustion Chamber Schematic

connection to the pressurizing gas source, and a quick-connect coupling and associated retention pin on the bottom for connection to the fuel injection pump inlet. See Fig. A2.2.

A2.3.2 *Fuel Injection Pump Assembly*, an integrated unit that incorporates a housing with two electric heater elements; a specific constant volume fuel delivery valve; a fuel bleed passage connecting to an external bleed valve for flushing fuel and purging air from the reservoir and fuel injection pump; and a digital controlled three-way solenoid valve that operates a pneumatically-actuated driver mechanism to deliver specimen fuel from the fuel sample reservoir to the injector nozzle and when turned off, discharges air from the driver mechanism to atmosphere.

A2.3.3 *Pneumatic Driver Air Surge Tank*, a tank of a minimum volume of 5.5 L installed in the compressed air line to the pneumatically-actuated fuel pump driver mechanism to minimize pressure fluctuations during the injection process. A suitable protection (that is, pressure relief valves or rupture discs) is installed in the compressed air line to the pneumatically-actuated fuel pump driver mechanism to prevent pressure in the surge tank exceeding 2.4 MPa. The air surge tank shall be pressure tested up to 4.0 MPa in accordance with local regulations.

A2.3.4 Fuel Injector Nozzle and Body Assembly, a specific design pintle-type injector nozzle conforming to the requirements of ISO 4010. The nozzle is assembled to the body that incorporates a spring-loaded needle extension with screw and

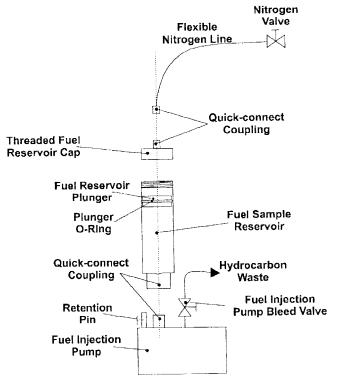


FIG. A2.2 Fuel Reservoir Schematic

lock nut for adjusting the nozzle opening pressure/release setting; a fuel bleed passage connecting to an external bleed valve for bleeding fuel from the nozzle and nozzle body; and an adjusting mechanism that positions a motion sensor near the injector nozzle needle extension pin, to determine when the nozzle needle lifts to initiate the start of injection.

A2.3.5 *Fuel Injector Body End Cap*, a machined plate with associated gaskets and seals, to clamp the injector nozzle body in the combustion chamber block.

A2.3.6 *Fuel Line*, high-pressure fuel line with associated fittings connecting the fuel injection pump assembly to the fuel injector body assembly.

A2.4 *Instrument Sensors*, sensors used to measure and either indicate the value of a variable or transmit the condition for control or data acquisition purposes as follows:

A2.4.1 Combustion Chamber Pressure Sensor (P1), a sensor installed to measure the pressure within the combustion chamber during each testing cycle.

A2.4.2 *Charge Air Pressure Sensor (P2)*, a calibrated pressure sensor installed in the piping between the charge air supply pressure regulator and the combustion chamber inlet valve.

A2.4.3 Injection Actuator Air Pressure Sensor (P3), a calibrated pressure sensor installed in the piping between the utility air supply pressure regulator and the injection pump driver mechanism manual pressure control valve.

A2.4.4 Inlet/Exhaust Valve Actuator Air Pressure Gage (P4), a pressure gage installed in the piping between the inlet/exhaust actuator valves and the associated manual pressure control valve.

A2.4.5 Combustion Chamber Outer Surface Temperature Sensor (T1), a Type K thermocouple with stainless steel sheath, inserted in a well fastened to the outer surface of the block.

A2.4.6 *Fuel Injection Pump Temperature Sensor (T2)*, a Type K thermocouple with stainless steel sheath, inserted in a well of the injection pump body.

A2.4.7 Temperature Sensor Near the Combustion Chamber Pressure Sensor (T3), a Type K thermocouple with stainless steel sheath, inserted in a well fastened to the outer surface of the block, near the combustion chamber pressure sensor.

A2.4.8 *Charge Air Temperature Sensor (T4)*, a Type K thermocouple with stainless steel sheath, inserted in the combustion chamber.

A2.4.9 Injector Nozzle Coolant Passage Temperature Sensor (T6), a Type K thermocouple with stainless steel sheath, inserted in the injector nozzle coolant passage.

A2.4.10 Coolant Return Temperature Sensor (T7), a Type K thermocouple with stainless steel sheath, installed in the coolant return piping of the injector nozzle coolant passage.

A2.4.11 *Injector Nozzle Needle Motion Sensor (N1)*, a motion sensor, that can be adjusted to provide a suitable gap between its sensing surface and the end of injector nozzle needle extension pin to detect the start of fuel injection.

A2.5 Computerized Control, Data Acquisition, Data Analysis and Reporting System, a PC-based computer, signal converters, test sequence control logic, control logic for critical temperatures, computer keyboard for manual entry of operating instructions, a monitor for visual observation of all testing functions, and a printer for printed copy output of test results.

A2.5.1 *Computer*, PC-type computer compatible with Windows¹⁹ operating system.

A2.5.2 *Control System*, a computer-based system to provide automated control of the relevant combustion analyzer and sub-system component functions. Electrical and electronic components of the control system are enclosed in a metal electrical/electronic cabinet.

A2.5.3 *Data Acquisition/Processing System*, a computerbased system with associated instrumentation to collect and process all relevant signals from the injector nozzle needle motion sensor, and temperature and pressure sensors. The system includes an analog-to-digital (A/D) data acquisition board installed in the computer to acquire the output signals from the sensors.

A2.5.4 *Signal Conditioning Components*, located in a metal electrical/electronic cabinet including signal conditioners for the temperature sensors, the combustion chamber pressure sensor, and the injector nozzle needle motion sensor.

A2.6 Circulating Coolant System

A2.6.1 *General*, a closed-loop circulating coolant system to control the temperature of the combustion injector nozzle and combustion chamber pressure sensor. The system includes:

A2.6.2 *Coolant Housing*, liquid cooled housing which is capable of fastening the combustion pressure sensor to the combustion chamber and maintaining its temperature within specifications.

A2.6.3 *Coolant Reservoir*, reservoir having a minimum volume of 1.5 L.

A2.6.4 *Coolant Pump*, centrifugal pump capable of meeting the pressure and flow requirements of the combustion analyzer.

A2.6.5 *Heat Exchanger*, liquid to air heat exchanger with associated fan and air filter.

A2.6.6 *Coolant Filter*, filter installed in the coolant line, capable of removing foreign particles from the coolant system fluid.

A2.6.7 *Manual Flow Control Valve*, Needle valve used to control the coolant flow to the injector nozzle coolant passage.

A2.7 Optional Equipment

A2.7.1 UPS, an electrical unit capable of powering the coolant system fan and pump during a utility power outage.

¹⁹ Windows is a registered trademark of Microsoft Corporation, One Microsoft Way, Redmond, WA 98052-6399.

A3. COMBUSTION ANALYZER OPERATING FUNCTIONS

A3.1 Starting and Warm-up Procedure

A3.1.1 With the combustion analyzer in shut down mode, start a new operating period as follows:

A3.1.1.1 Position the combustion analyzer power switch to ON.

A3.1.1.2 Initiate the automated warm-up sequence using the appropriate computer command.

A3.1.1.3 At the end of the warm-up sequence, the total warm-up time will be indicated on the computer monitor. Typically each warm-up requires an average of 2500 to 2900 s. Significant increases in the average total warm-up time (that is, more than 10 %) are indicative of a potential malfunction of the heating elements of the combustion chamber. For diagnostic procedures, refer to the instructions provided by the manufacturer.

A3.1.1.4 Open the valve at the source of each compressed gas and adjust the individual pressure regulators as needed to provide the specification pressures.

A3.1.1.5 Perform at least one preliminary ignition delay determination for a typical diesel fuel oil sample following the procedure described in 12.2. Check and adjust all operating conditions so that the combustion analyzer complies with the specification values and is ready for fit-for-use qualification testing. Discard the results of all preliminary ignition delay determinations.

A3.2 Fuel Injection System Procedure

A3.2.1 *General*—The sample fuel reservoir is illustrated in Fig. A2.2, Fuel Reservoir Schematic. The floating plunger is inserted between the pressurizing nitrogen and the fuel in the reservoir when a fuel specimen is to be tested. The floating plunger is omitted from the assembly during the sequences involving flushing of fuel when the pressurizing nitrogen is in direct contact with the fuel specimen. One flushing function involves forcing a portion of specimen fuel through the fuel injection pump and injector nozzle passages to ensure that they are full of fuel and free of any trapped air. A second flushing function is utilized to force all specimen fuel out of the injection pump and injector nozzle passages after the completion of a test determination. Details of these functions are as follows:

A3.2.2 Flushing the Fuel System with Specimen:

A3.2.2.1 Close the nitrogen valve, disconnect the flexible nitrogen line and remove the reservoir cap.

A3.2.2.2 Fill the reservoir with the test specimen.

A3.2.2.3 Reinstall the reservoir cap hand tight and reconnect the flexible nitrogen line to the cap.

A3.2.2.4 Open the nitrogen valve.

A3.2.2.5 Open the injection pump manual bleed valve for sufficient time to flush the fuel injection pump and bleed passages before closing the injection pump bleed valve.

A3.2.2.6 Open the injector nozzle manual bleed valve to flush the fuel line and injector nozzle with the remainder of the test specimen before closing the injector nozzle bleed valve.

A3.2.2.7 Close the nitrogen valve, disconnect the flexible nitrogen line and remove the reservoir cap.

A3.2.3 Filling and Purging the Fuel System with Specimen:

A3.2.3.1 Refill the fuel reservoir with the test specimen.

A3.2.3.2 Reinstall the reservoir cap hand tight and reconnect the flexible nitrogen line to the cap.

A3.2.3.3 Open the nitrogen valve and close it after a few seconds.

A3.2.3.4 Purge the injection pump and injector nozzle passages of air as follows: (1) Open the manual injection pump bleed valve until a continuous stream of fuel is discharged to waste and then close the valve; (2) Open the manual injector nozzle bleed valve until a continuous stream of fuel is discharged to waste and then close the valve.

A3.2.3.5 Disconnect the flexible nitrogen line and remove the reservoir cap.

A3.2.3.6 Check that the fuel reservoir still contains some fuel. If not, repeat the filling and purging steps before continuing.

A3.2.3.7 Fill the reservoir with test specimen leaving enough space to insert the reservoir plunger.

A3.2.3.8 Insert the reservoir plunger in the reservoir using the appropriate tools, and press it into the reservoir until test specimen seeps up past the plunger thus purging any air trapped between the test specimen and the plunger. Carefully extract the tools.

A3.2.3.9 Reinstall the reservoir cap hand tight, reconnect the flexible nitrogen line and open the nitrogen valve.

A3.2.4 Discharging Unused Specimen and Cleaning Fuel System:

A3.2.4.1 Close the nitrogen valve, disconnect the flexible nitrogen line and remove the reservoir cap.

A3.2.4.2 Open the injection pump bleed valve to facilitate the removal of the plunger.

A3.2.4.3 Remove the plunger from the reservoir using the appropriate tool.

A3.2.4.4 Clean the plunger and O-ring assembly using a clean, lint-free cloth and set it aside. Blowing across the surfaces using oil-free compressed air in a fume extraction system may be an adequate alternative or useful supplement.

A3.2.4.5 Close the injection pump bleed valve.

A3.2.4.6 Reinstall the reservoir cap hand tight, reconnect the flexible nitrogen line and open the nitrogen valve.

A3.2.4.7 Open the injection pump bleed valve to discharge any specimen from the injection pump passages. Close the bleed valve.

A3.2.4.8 Open the injector nozzle bleed valve to discharge any remaining specimen from the injection system. Close the nitrogen valve. Close the injector nozzle bleed valve.

A3.2.4.9 Disconnect the flexible nitrogen line and remove the reservoir cap.

A3.2.4.10 Remove the reservoir from the fuel injection pump inlet by releasing the reservoir retention pin.

A3.2.4.11 Clean the internal surfaces of the reservoir using a clean, lint-free cloth. Blowing across the surfaces using oil-free compressed air in a fume extraction system may be an adequate alternative or useful supplement.

A3.2.4.12 Clean the injection pump inlet connector fitting using a clean, lint-free cloth.

A3.2.4.13 Reinstall the reservoir on the injection pump inlet and lock it in position with the retention pin.

A3.2.4.14 The fuel system is prepared for the next specimen determination.

A3.3 Pressure Sensor Assembly Cleaning Procedure

A3.3.1 (**Warning**—Avoid skin contact with the surfaces of the pressure sensor assembly and combustion chamber if the combustion analyzer is not at room temperature.)

A3.3.2 Check that the valve at the source of each compressed gas is closed, decompress the combustion chamber using the appropriate computer command, and position the combustion analyzer power switch to OFF.

A3.3.3 Disconnect the pressure sensor signal cable, remove the combustion chamber pressure sensor from its housing, clean the sensing surface of the pressure sensor and the hole of the pressure sensor housing in accordance with the instructions of the manufacturer.

A3.3.4 Reinstall the pressure sensor in its housing.

A3.3.5 Wipe any oily deposits from the sensor signal cable and connector and connect the cable to the pressure sensor.

A3.3.6 Position the combustion analyzer power switch to ON.

A3.3.7 Warm-up the combustion analyzer.

A3.4 Combustion Chamber Sealing Integrity Check Procedure

A3.4.1 Using the appropriate computer command, start an automated sealing integrity check of the warmed-up combustion chamber. This procedure tests the effectiveness of the combustion chamber seals by pressurizing the chamber with a standard charge of compressed air. The pressure variation inside the chamber is monitored for a period of 20 s. The rate of pressure drop is displayed on the computer monitor.

A3.4.2 The operator is responsible to check that the displayed rate of pressure drop is less than the specified 3.5 kPa/s limit.

A3.4.3 If the rate of pressure drop exceeds the limit, inadequate sealing is confirmed and diagnostic procedures to determine and remedy the problem are recommended before performing tests. Refer to the instructions provided by the manufacturer.

A3.5 Combustion Analyzer Shut Down Procedure

A3.5.1 Check that all specimen has been discharged from the fuel injection system and the fuel reservoir and associated components are clean.

A3.5.2 Close the valve at the source of each compressed gas.

A3.5.3 Open the appropriate bleed valves to decompress the piping between the compressed gas regulators and combustion analyzer. Close all bleed valves after decompressing the piping.

A3.5.4 Position the combustion analyzer power switch to OFF.

NOTE A3.1—Electric power for the circulating coolant system will remain on for 3 h after the combustion analyzer is shut down.

A4. SUPPLEMENTAL PROCEDURE INFORMATION

A4.1 Test Sequence

A4.1.1 *General*—An automated test run consists of 15 preliminary (pre-test injections) + 32 subsequent (test injections) combustion cycles. A combustion cycle involves: (1) charging the chamber to the test pressure, (2) injecting a small volume of fuel sample into the combustion chamber, and (3) releasing of the combustion gases. During the combustion cycle, the injector nozzle needle motion sensor measures the motion of the injector nozzle needle and the combustion chamber pressure sensor measures the charge air pressure.

A4.1.2 A simplified example of the output of the nozzle needle motion sensor and the combustion chamber pressure sensor recorded for a single combustion cycle during a test sequence is shown in Fig. A4.1.

A4.1.3 The ignition delays of the 32 test injections are averaged to produce the analytical ID result.

A4.1.4 During each of the 32 test injections the following parameters are recorded:

Parameters

ID DCN Charge air pressure (*P2*) Injection actuator air pressure (*P3*) Charge air temperature (*T4*) Combustion chamber pressure sensor temperature (*T3*) Injector nozzle coolant passage temperature (*T6*) Coolant return temperature (*T7*)

A4.1.5 The individual measured values of the above parameter for each of the 32 combustion cycles as well as their average, minimum and maximum are automatically printed on a test report at the end of each test (see Appendix X1).

A4.1.6 The derived cetane number is calculated using the following equation²⁰:

$$DCN = 83.99(ID - 1.512)^{(-0.658)} + 3.547$$
 (A4.1)

where: ID = ignition dela

ID = ignition delay in milliseconds, and DCN = derived cetane number.

²⁰ The equation was derived from a correlation test program, comprising ASTM National Exchange Group (NEG) check fuels, heptamethylnonane, cetane and an in-house check fuel.

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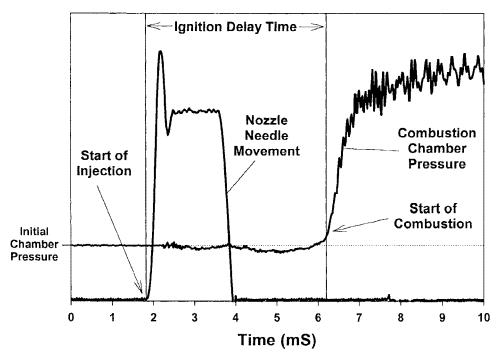


FIG. A4.1 Signals of Motion Sensor and Combustion Chamber Pressure Sensor During a Single Combustion Cycle

APPENDIX

(Nonmandatory Information)

X1. EXAMPLE OF TEST OUTPUT

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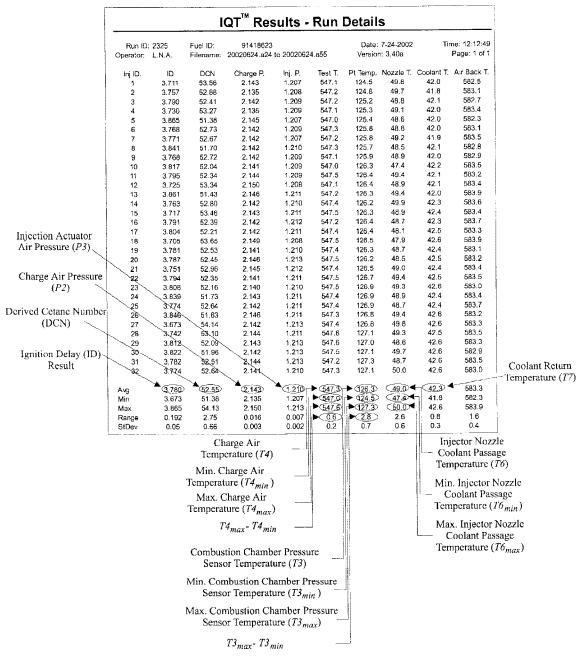


FIG. X1.1 Example of Test Output

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