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Standard Test Method for Determination of Derived Cetane Number (DCN) of Diesel Fuel Oils—Fixed Range Injection Period, Constant Volume Combustion Chamber Method¹

This standard is issued under the fixed designation D7170; 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 ignition characteristics 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 and low-sulfur diesel fuel oils, European standard EN 590, and Canadian standards CAN/CGSB-3.517-2000 and CAN/CGSB 3.6-2000. The test method may also be applied to the quantitative determination of the ignition characteristics of blends of fuel oils containing biodiesel material, and diesel fuel oil 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).

1.3This 1.3 This test method covers the ignition delay range from a minimum value of 35.0 DCN (ignition delay of 4.89 ms) to a maximum value of 59.6 DCN (ignition delay of 2.87 ms). The average DCN result for each sample in the ILS ranged from 37.29 (average ignition delay of 4.5894 ms) to 56.517 (average ignition delay of 3.0281 ms).

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 and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

D613 Test Method for Cetane Number of Diesel Fuel Oil Preview

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, Petroleum Products, and Lubricants 5c-d25ce9d70c83/astm-d7170-11

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

E456 Terminology Relating to Quality and Statistics

E832 Specification for Laboratory Filter Papers

2.2 EN Standard:³

EN 590 Automotive Fuels-Diesel-Requirements and Test Methods

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

2.3 Energy Institute Standard:⁴

IP 41 Ignition Quality of Diesel Fuels-Cetane Engine Test Method

2.4 Canadian Standards:⁵

CAN/CGSB-3.517-2000 Regular Sulphur Diesel Fuel—Specification

CAN/CGSB 3.6-2000 Automotive Low-Sulphur Diesel Fuel—Specification

2.5 DIN Standard: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 of specific reference materials determined under reproducibility conditions by collaborative experimental work.

3.1.2 *cetane number (CN)*, *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 method, cetane number is that defined by ASTM D613/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. D6299

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. **D6299**

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 into 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. —a number calculated using a conversion equation to determine a cetane number.

<u>3.2.5.1</u> *Discussion*—The conversion equation relates a measured ignition delay or ignition delay and combustion delay from a combustion analyzer to a cetane number.

3.2.6 *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 (+0.02 MPa above chamber static pressure) 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 *injection period (IP)*, *n*—the period of time, in milliseconds (ms), that the fuel injector nozzle is open as determined using the specific combustion analyzer applicable for this test method.

3.2.8 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 Acronyms:

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

⁴ Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K.

⁵ Available from the Canadian General Standards Board, Ottawa, Canada, K1A 1G6.

⁶ Available from DIN, Deutsches Institut für Normung, Burggrafenstrasse 6, 10787 Berlin.

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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 2 preliminary cycles and 25 further cycles. The ID measurements for the last 25 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 220 mL and a test time of approximately 20 min on a fit-for-use instrument.

6. Interferences

6.1 **Warning**—Minimize exposure of sample fuels, calibration reference materials, QC samples, and check standard to sunlight or fluorescent lamp UV emissions to minimize induced chemical reactions that can affect ignition delay measurements.⁷

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 system⁸ (see Fig. 1) comprised of:

7.1.1 *Combustion Chamber*—A cylindrical block having a volume of 0.60 ± 0.03 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 an opening at the other end of the chamber to accommodate insertion of intake, exhaust, and various sensors.

7.1.2 *Fuel Injection System*—A pneumatically actuated fuel injection system with fuel injection pump, injector nozzle assembly and associated sample reservoir to assure for 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 100 mL.

7.1.2.2 *Fuel Injector Nozzle*—A standard one-hole nozzle conforming to the requirements of DIN 73372. The nozzle is assembled to the body that incorporates a spring-loaded needle extension with screw and lock nut for adjusting the nozzle opening pressure setting; a fuel bleed passage connecting to an external bleed valve for bleeding fuel from the nozzle and nozzle body; and a positions motion sensor mounted in an adjustable housing near the injector nozzle needle extension pin, to determine when the nozzle needle lifts to initiate the start of injection.

7.1.3 *Coolant System*—A closed-loop circulating coolant system to control the temperature of the combustion injector nozzle. 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:

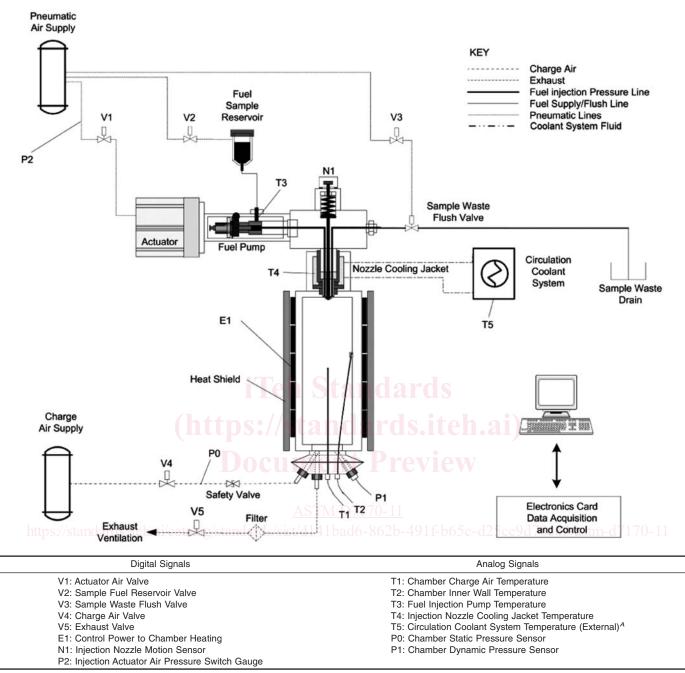
7.1.4.1 *Combustion Chamber Static Pressure Sensor (P0)*—A sensor installed to measure the static pressure within the combustion chamber before and after each combustion cycle.

7.1.4.2 *Combustion Chamber Dynamic Pressure Sensor (P1)*—A sensor installed to measure the pressure within the combustion chamber during each combustion cycle. The pressure sensor is fitted with an integrated temperature sensor to record the operating temperature (T3) at the sensor.

7.1.4.3 *Injection Actuator Air Pressure Regulator (P2)*—A calibrated pressure regulator installed between the pneumatic air supply and the fuel injection pump actuator.

⁷ 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.

⁸ The sole source of supply of the FIT combustion analyzer known to the committee at this time is Waukesha Dresser, Inc., 1101 West St. Paul Avenue, Waukesha, WI 53188-4999. 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, which you may attend.



^A T5 is not located on the instrument. It is the temperature of the auxiliary Circulation Coolant System adjusted to maintain T4. FIG. 1 Combustion Analyzer Schematic

7.1.4.4 *Combustion Chamber Inner Wall Temperature Sensor (T2)*—A type K thermocouple with stainless steel sheath, inserted in a well fastened to the inner surface of the chamber.

7.1.4.5 Chamber Charge Air Temperature Sensor (T1)—A type K thermocouple with stainless steel sheath, inserted in the combustion chamber.

7.1.4.6 Fuel Injection Pump Temperature Sensor (T4)—A PT100 temperature sensor with stainless steel sheath, inserted into the fuel injection pump body.

7.1.4.7 Injector Nozzle Cooling Jacket Temperature Sensor (T5)—A PT100 temperature sensor with stainless steel sheath, inserted in the injector nozzle coolant passage.

7.1.4.8 *Injector Nozzle 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.

7.1.5 Computerized Control, Data Acquisition, Data Analysis and Reporting System—A microprocessor controlled system connected to a computer with keyboard for manual entry of operating instructions, a monitor for visual observation of all testing

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functions, and a printer for printed copy output of test results. The computer-based system provides automated control of the relevant combustion analyzer and sub-system component functions and to collect and process all relevant signals from the injector nozzle needle motion sensor, and temperature and pressure sensors.

7.2 Refer to the instruction manual of the manufacturer⁹ 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 2.40 MPa.

7.3.2 *Pneumatic Air Regulator*—A two-stage regulator capable of controlling the downstream pressure to a minimum pressure of 0.75 MPa.

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 (µm) 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*—With a minimum purity of 99.5 volume percent. The assigned IDARV for this material and this method is 3.15 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 IDARV for this material and this method is 10.1 ms (**Warning**—Flammable. Vapor harmful. Vapor may cause flash fire.)

NOTE 2—Experience has found some MCH meeting the purity specification but which does not meet ignition delay ARV (typically 1 to 1.5 milliseconds shorter). It is recommended that new material be qualified prior to use.

8.2 Check Standard:

8.2.1 *Heptane*—With a minimum purity of 99.5 volume percent (see 8.1.1).

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*—A compressed air containing 20.9 ± 1.0 volume percent oxygen (O₂), less than 0.5 PPM CO, less than 1.0 PPM CO₂, less than 5 PPM H₂O, less than 0.1 PPM NO_X, less than 0.1 PPM SO₂, and less than 0.1 PPM Total Hydrocarbon Content (THC). (Warning—Compressed gas under high pressure that supports combustion.)

NOTE 3-This grade air is typically referred to as Continuous Emissions Monitoring Grade Air (CEM).

8.5 *Coolant System Fluid*—A50:50 volume mixture of water and commercial ethylene glycol-based antifreeze (**Warning**—Poison. May be harmful or fatal if inhaled or swallowed.) M D7170-11

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 *Pneumatic Air*—An oil free compressed air having less than 0.1 volume percent water (**Warning**—Compressed gas under high pressure that supports combustion.)

9. Sampling and Test Specimen Preparation

9.1 Sampling:

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

9.1.1.1 **Warning**—Collect and store diesel fuel oil 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 D5854 for appropriate information relating to the mixing and handling of diesel fuel oil samples. 9.2 *Test Specimen Preparation*:

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 to 32°C.

9.2.2 *Filtration*—Samples may be filtered through a Type I, Class A filter paper at room temperature and pressure before testing (see Specification E832), or through a nominal 3 to 5 μm porosity filter element using a 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 surface 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.

⁹ The instruction manual is available from Waukesha Dresser, Inc., 1101 West St. Paul Avenue, Waukesha, WI 53188-4999.

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 or controlled by the computer software or by operator adjustment.

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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 of the manufacturer, using a pressure sensor connected to the nozzle housing through an appropriate adapter. For additional details, refer to the instruction manual of the manufacturer.⁹

10.3.2 *Injector Nozzle Motion Sensor Position*—Verify Sensor positioning using the appropriate function of the apparatus software. If required, manually adjust the position of the motion sensor by referring to the instruction manual of the manufacturer,⁹ using the appropriate function of the apparatus software.

10.3.3 *Combustion Chamber Leakage Rate*—Shall be less then 2.0 kPa/s, as measured during the automated check of the sealing integrity of the combustion chamber.

NOTE 4—After the warm-up sequence (see 12.1.1) is finished, the computer system initiates an automatic diagnostic procedure consisting of zero-adjustment of the chamber dynamic pressure sensor (P1) and a chamber sealing integrity check. The chamber leakage rate is continuously monitored by the computer system throughout the test and in between tests.

10.4 Standard Operating Conditions:

10.4.1 *Chamber Static Pressure (P0)*—The average of 25 combustion cycles for Chamber Static Pressure is required to be within 2.40 ± 0.02 MPa.

10.4.2 Chamber Charge Air Temperature (T1), $510 \pm 50^{\circ}$ C.

10.4.3 *Chamber Inner Wall Temperature (T2)*—Initially set by the manufacturer, the surface temperature set-point is monitored and controlled by the computer. Operator adjustment of the controller set-point is required, in accordance with the calibration procedure.

10.4.3.1 The difference in temperature $(T2_{max} - T2_{min})$ as determined and recorded by the computer, shall be less than 2.5°C during a 25 combustion cycle measurement determination.

10.4.4 To ensure proper heat distribution and guard against malfunctioning of the heater element the temperature difference (T2 - T1) shall be within the tolerances, as referred to in the instruction manual of the manufacturer⁹ for each of the 25 combustion cycles. The difference (T2 - T1) is monitored by the computer.

10.4.5 Fuel Injection Pump Temperature (T3), $35 \pm 2^{\circ}$ C.

10.4.6 Injector Nozzle Coolant Jacket Temperature (T4)—Set the coolant reservoir temperature (T5) to achieve an injector nozzle coolant passage temperature (T4) of $30.0 \pm 0.5^{\circ}$ C. T4 is determined and recorded by the computer. A temperature outside the range given during a 25 combustion cycle measurement indicates a possible malfunctioning of the cooling system.

10.4.7 Injection Actuator Air Pressure (P2), 0.75 \pm 0.05 MPa.

10.4.8 Injection Period (IP):

10.4.8.1 The average injection period over 25 combustion cycles: 5.00 ± 0.25 ms.

10.4.8.2 The individual injection period for each of the 25 combustion cycles: 5.00 ± 1.00 ms. 70c83/astm-d7170-11

10.4.8.3 Each individual injection period and the updated average is continuously monitored and recorded by the computer. If necessary, to comply with these requirements, the rack setting on the fuel pump can be adjusted manually.

11. Calibration and Quality Control Testing

11.1 Calibration—Calibrate the combustion analyzer (1) after it is installed and commissioned, (2) once a week, (3) after replacement of critical parts or components of combustion chamber assembly, fuel injection system or instrument sensors, (4) after calibration of the injection actuator air pressure, chamber static pressure, or chamber dynamic pressure sensors, or (5) whenever check standard or QC sample determinations are not acceptable.

11.2 Pre-calibration Procedures :

11.2.1 Start and warm-up the combustion analyzer (see 12.1.1).

11.3 *Calibration Procedure*—Two 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 To be an acceptable data set, each single result is required to be within 3.15 \pm 0.04 ms.

11.3.1.2 The average of three acceptable ID results is required to be within 3.15 \pm 0.02 ms.

11.3.1.3 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 instruction manual of the manufacturer.⁹

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

Note 5-ID increases when the combustion chamber inner surface temperature decreases and vice versa.