



Designation: D7545 – 14 (Reapproved 2019)^{ε1}

Standard Test Method for Oxidation Stability of Middle Distillate Fuels—Rapid Small Scale Oxidation Test (RSSOT)¹

This standard is issued under the fixed designation D7545; 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.

^{ε1} NOTE—Editorially updated Footnote 5 and Fig. 1 in December 2019.

1. Scope

1.1 This laboratory test method covers a quantitative determination of the stability of middle distillate fuels such as diesel fuels and heating oils, with up to 100 % biodiesel, under accelerated oxidation conditions, by an automatic instrument.

NOTE 1—This test method is technically equivalent to test method EN 16091

1.2 This test method is designed for products complying with Specification **D975** on Diesel Fuel, Grades No. 1D and 2D; Specification **D396** on Burner Fuel, Grades No. 1 and No. 2; Specification **D6751** on Biodiesel, B100, and Specification **D7467** on Diesel Fuel Oil, B6 to B20.

1.3 This test method measures the induction period, under specified conditions, which can be used as an indication of the oxidation and storage stability of middle distillate fuels.

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.*

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 **D02.14** on Stability, Cleanliness and Compatibility of Liquid Fuels. Current edition approved Dec. 1, 2019. Published December 2019. Originally approved in 2009. Last previous edition approved in 2014 as D7545 – 14. DOI: 10.1520/D7545-14R19E01.

2. Referenced Documents

2.1 ASTM Standards:²

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

2.2 Other Standards:

CEN/TR 16366:2012 Liquid Petroleum Products—Middle Distillates and Fatty Acid Methyl Ester (FAME) Fuels and Blends—Round Robin Report on Applicability of Rapid Small Scale Oxidation Test Method³

EN 590 Automotive Fuels—Diesel—Requirements and Test Methods³

EN 16091 Liquid Petroleum Products—Middle Distillates and Fatty Acid Methyl Ester (FAME) Fuels and Blends—Determination of Oxidation Stability by Rapid Small Scale Oxidation Method³

ISO 4259 Petroleum Products—Determination and Application of Precision Data in Relation to Methods of Test⁴

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *break point, n*—pressure in the test apparatus which is 10 % below the maximum pressure of the actual test run.

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 (CEN), Avenue Marnix 17, B-1000, Brussels, Belgium, <http://www.cen.eu>.

⁴ Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, <http://www.iso.org>.

3.1.2 *induction period, n*—time elapsed between starting the heating procedure of the sample vessel and the break point, measured in minutes.

4. Summary of Test Method

4.1 A 5 mL sample is introduced into a pressure vessel which is then charged with oxygen to 700 kPa at ambient temperature. The test is initiated by starting the heater and heating the pressure vessel to a temperature of 140 °C.

4.2 The pressure is recorded continuously until the break-point is reached. Alternatively, the test may be terminated when a minimum specification requirement is reached.

5. Significance and Use

5.1 The induction period may be used as an indication of the oxidation and storage stability of middle distillate fuel.

5.2 Compared to some other oxidation and storage stability test methods, this method uses a small sample and gives a result in a short time period.

6. Apparatus

6.1 *General*—This test method uses an automatically controlled oxidation tester⁵ (see Fig. 1) comprising an oxidation pressure vessel containing a test sample cup capable of being rapidly heated, fitted with a pressure sensor capable of measuring pressures up to 2000 kPa and a temperature sensor capable of reading to 0.1 °C.

6.1.1 Pressure and temperature in the oxidation vessel are recorded continuously during the test. The oxidation pressure vessel is fitted with filling and relief-valves and a means of automatically releasing the pressure. The integrated cooling fan cools the pressure vessel from the test temperature to ambient temperature by a flow of air. See Annex A1 for detailed information.

6.2 *Volumetric Device*—Clean and free from contaminations of previous samples, capable of delivering 5.0 mL ± 0.1 mL.

6.3 *Temperature Calibration Equipment*—Comprising a cover and a temperature calibration sensor (Fig. 2). The temperature calibration sensor is fixed to a depth of 15 mm ± 0.5 mm. The temperature calibration sensor, calibrated to the nearest 0.1 °C, is calibrated by an approved calibration service, such as one that is traceable to the National Institute of Standards and Technology (NIST) or to a national authority in the country in which the equipment is used or manufactured.

6.4 *Pressure Calibration Equipment*—Comprising a pressure calibration sensor (Fig. 3). The pressure calibration sensor is calibrated to the nearest 10 kPa by an approved calibration service, such as one that is traceable to the National Institute of

Standards and Technology (NIST) or to a national authority in the country in which the equipment is used or manufactured.

7. Reagents and Materials

7.1 Solvent for the removal of oxidation residues from the test vessel. The solvent shall be of suitable purity to leave no residue on the apparatus. Ethanol of 94 % minimum purity has been found to be suitable. Other solvents, such as a mixture of equal volumes of toluene and acetone, may be used if shown to meet the requirements for the removal of oxidation residues from the test vessel without leaving any residue in the sample test cup.

7.2 *Oxygen*—Commercially available extra-dry oxygen of not less than 99.6 % purity.

7.3 *Lint-free Cleaning Tissue*—For sensitive surfaces and that will not scratch the surface.

7.4 “O-ring” Seal—See A1.2.

7.5 *Temperature Calibration Fluid*—Stable middle distillate liquid with a flash point above +60 °C and a boiling point above +150 °C.

8. Hazards

8.1 (**Warning**—To provide protection against possible explosive rupture of the pressure vessel and hazards relating to hot and flammable fuels, the apparatus shall be operated behind an appropriate safety shield.)

9. Sampling

9.1 Sample in accordance with Practice D4057 or D4177.

10. Preparation of Apparatus

10.1 Remove the previous sample by means of a pipette or similar device.

10.2 Remove the used “O-ring” seal and discard.

NOTE 2—To avoid contamination of the new test, it is necessary to discard the used “O-ring” seal, because it might be soaked with oxidation products from the previous test.

10.3 Wipe the test sample cup, the seal groove and the cover of the test vessel with lint-free cleaning tissue (7.3) soaked with solvent until free of gum or other oxidation residues.

10.4 Allow the test sample cup and cover to dry in air and visually inspect for cleanliness.

NOTE 3—Compressed air is generally unsuitable to speed up evaporation of solvent because it can contain traces of oil that could contaminate the next test.

10.5 Insert a new “O-ring” seal.

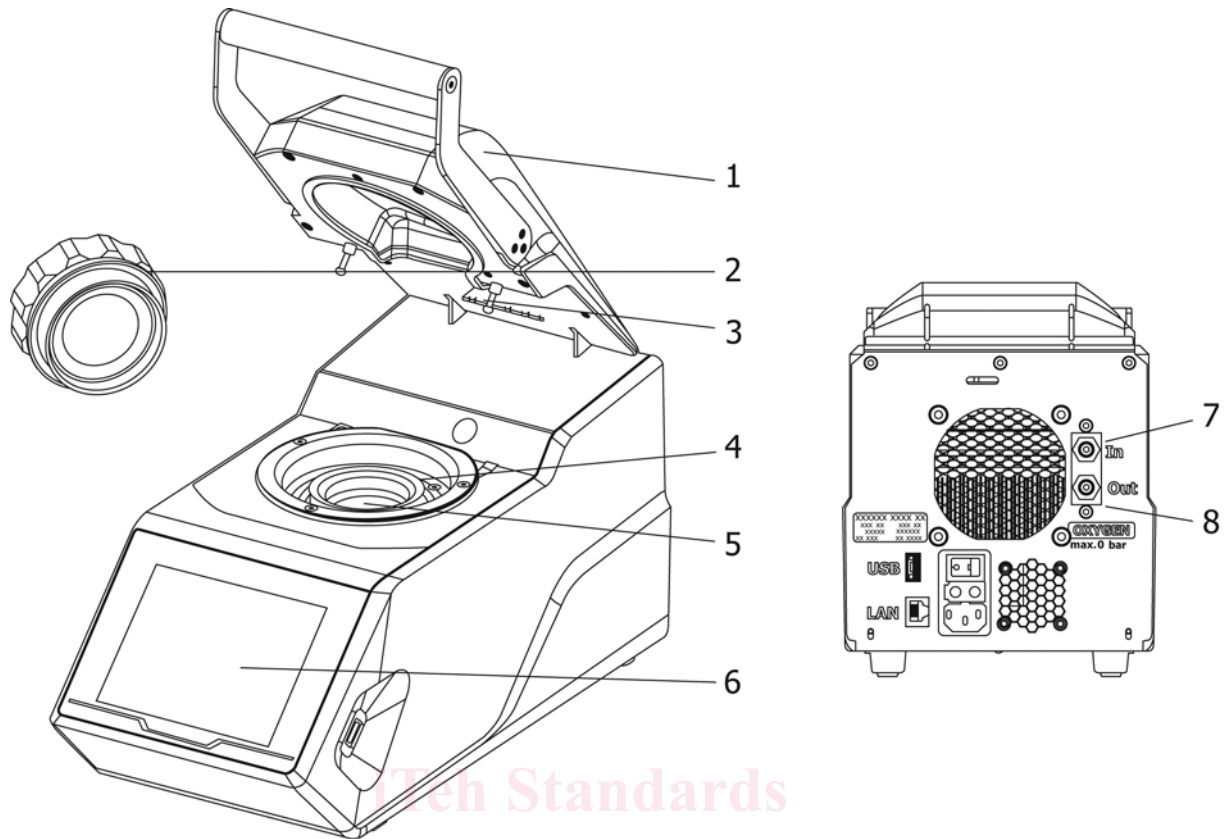
11. Calibration

11.1 Recalibrate the tester every 12 months for correct temperature and pressure detection.

11.2 *Calibration of Temperature Indicator*—Calibrate the temperature sensor (A1.6) to the nearest 0.1 °C using the temperature calibration equipment (6.3) according to A2.1.

11.3 *Calibration of Pressure Detector*—Calibrate the pressure sensor (A1.5) to the nearest 10 kPa, using the pressure calibration equipment (6.4) according to A2.2.

⁵ The sole source of supply of the apparatus known to the committee at this time is Anton Paar's RapidOxy 100 instrument (formerly PetroOxy), available from Anton Paar ProveTec GmbH, Ludwig-Erhard-Ring 13,15827 Blankenfelde-Mahlow, Germany. 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.



Front View

Rear View

1. Safety and insulating hood
2. Screw cap for closing the test container
3. Locking/unlocking mechanism for insulating hood
4. "O-ring" seal for test sample cup
5. Test sample cup
6. Operating panel with display
7. Oxygen inlet
8. Oxygen outlet

FIG. 1 Schematic of Apparatus for Rapid Small Scale Oxidation Test