

Designation: D7548 - 22

Standard Test Method for Determination of Accelerated Iron Corrosion in Petroleum Products¹

This standard is issued under the fixed designation D7548; 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 an accelerated laboratory and field procedure for the determination of corrosion of iron, in the presence of water, on samples such as gasoline and gasoline blended with 10 % ethanol, E10 (Specification D4814); gasoline-blend components (except butane); diesel fuel and biodiesel B5, except Grade No. 4-D (Specification D975); biodiesel B6 to B20 (Specification D7467); diesel-blend component such as light cycle-oil; No. 1 fuel oil, No. 2 fuel oil (Specification D396); aviation turbine fuel (Specification D1655).
- 1.2 The values stated in SI units are to be regarded as the standard.
- 1.2.1 *Exception*—Values in parentheses are for information only.
- 1.3 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.
- 1.4 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

A108 Specification for Steel Bar, Carbon and Alloy, Cold-Finished

D97 Test Method for Pour Point of Petroleum Products

D396 Specification for Fuel Oils

D665 Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water

D975 Specification for Diesel Fuel

D1193 Specification for Reagent Water

D1655 Specification for Aviation Turbine Fuels

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4814 Specification for Automotive Spark-Ignition Engine Fuel

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

G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)³

2.2 Other Standards:

British Standard BS970–1:070M20 ⁴or European Steel C22E Number 1.1151—Carbon and Carbon Manganese Steels including Free Cutting Steels

3. Terminology

- 3.1 Definitions:
- 3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.
- 3.1.2 *corrosion*, *n*—chemical or electrochemical reaction between a material, usually a metal, and its environment that produces a deterioration of the material and its properties. **G15**
- $3.1.3 \ rust, \ n$ —corrosion product consisting primarily of hydrated iron oxide.
- 3.1.3.1 *Discussion*—Rust is a term properly applied only to ferrous alloys. **G15**
 - 3.2 Acronyms:
 - 3.2.1 *TP*—temperature probe.

4. Summary of Test Method

4.1 A polished iron corrosion test-rod is immersed in 50 mL of the sample being tested and heated to 37.8 °C (100 °F), at

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.

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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 The last approved version of this historical standard is referenced on www.astm.org.

⁴ Annual Book of IP Standard Methods for Analysis and Testing of Petroleum and Related Products, Vol 2. Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., http://www.energyinst.org.

which point 5 mL of reagent water, Type III or better, is added to the sample. Sample temperature is maintained between 37 °C to 39 °C (98 °F to 102 °F), with the target being 37.8 °C (100 °F), for 1 h. At the end of the heating period, test-rod is removed, rinsed, and examined for the degree/category of corrosion against the "ASTM Iron Corrosion Rating Chart—Test Method D7548."

5. Significance and Use

- 5.1 In general, wherever the possibility exists of water getting mixed with products/material (covered under 1.1) the results obtained by this test method will indicate the degree to which corrosion of iron components can be expected.
- 5.2 Test also determines the antirust properties of products/material (covered under 1.1) in preparation for the various stages through which the tested product may pass prior to or during its transport through a pipeline.
- 5.3 Test results are also meant to show whether or not the dosage levels and type of iron corrosion inhibitor added to a product/material (covered under 1.1) is sufficient for achieving the desired protection of affected assets such as storage tanks, process lines, and shipment systems.

6. Apparatus

- 6.1 Stirring Hotplate, ^{5,6} Single or multi-position (see Appendix X1), ceramic top, capable of accommodating Accelerated Iron Corrosion Test (AICT) apparatus specified in A1.1. Stirrer should be capable of stirring at a rate of 900 r/min ± 100 r/min.
- 6.2 Water Bath—Beaker, 150 mL to 200 mL capacity, borosilicate glass, containing 100 mL of distilled water, or other type of water bath capable of accommodating Accelerated Iron Corrosion Test (AICT) apparatus specified in A1.1.
- 6.3 Test Jar—Cylindrical, clear glass, 90 mL capacity, flat bottom, 115 mm to 125 mm (4.53 in. to 4.92 in.) in height, 33.2 mm to 34.8 mm (1.31 in. to 1.37 in.) outside diameter, 30.0 mm to 32.4 mm (1.18 in. to 1.28 in.) inside diameter, 1.6 mm (0.06 in.) maximum wall thickness.

Note 1—The pour point test jar specified in Test Method D97 meets this requirement. A test jar meeting requirements of 6.3 and graduated at 50 mL is more suitable.

Note 2—Optionally, to prevent accidental breakage of the test jar, a rubber O-ring, $32 \text{ mm} \ (1.25 \text{ in.})$ inside diameter, and $3 \text{ mm} \ (0.12 \text{ in.})$ thick, could be slipped over the test jar up to about $25 \text{ mm} \ (1.0 \text{ in.})$ below its rim.

6.4 *Cover*,^{7,6} Test Jar—Plastic, such as high density polyethylene (HDPE), with three holes, meeting dimensions and features seen and outlined in A1.2.

6.5 Corrosion Test Rod, ^{8,6} with a plastic or polytetrafluoroethylene (PTFE) holder and nominal dimensions according to Fig. A1.3; round, threaded steel rod, tapered at one end; 81.0 mm (3.19 in.) long, including the 12.7 mm (0.5 in.) long threaded portion, and 12.7 mm (0.5 in.) in diameter. See Fig. A1.3. Test rod should be made of steel conforming to Grade 1015, 1018, 1020, or 1025 of Specification A108, or to European Steel C22E number 1.1151, or to British Standard BS970–1) as per Table 1. If these steels are unavailable, other equivalent steels may be used, provided they are satisfactory according to comparative tests using this test method.

6.5.1 *Holder*, ^{8,6} ethanol-resistant plastic or PTFE with nominal dimensions according to Fig. A1.3. Total length: 63.5 mm (2.5 in.), with a 2-step handle at top that is disc-type, 22.2 mm \pm 0.1 mm (0.875 in. \pm 0.004 in.) in diameter by 15.9 mm (0.625 in.) thick. Below this is a circular step or rim which is 17.1 mm (0.63 in.) diameter by 6.4 mm (0.25 in.) thick. The stem portion of the holder is 41.2 mm (1.622 in.) long by 12.7 mm (0.5 in.) in diameter, with a 6.4 mm (0.25 in.) female-threaded hole in the bottom to accept the male-threaded test rod.

6.6 *Temperature Probe*, Digital, calibrated, with stem about 200 mm (8 in.) long by 3.0 mm (0.125 in.) in diameter and covering a temperature range of about 20 °C (68 °F) to at least 60 °C (140 °F).

Note 3—Calibrated, digital temperature probe, or equivalent calibrated temperature measurement device, covering a range of $-50~^{\circ}$ C ($-58~^{\circ}$ F) to $+300~^{\circ}$ C ($+572~^{\circ}$ F) will be suitable. Alternatively, calibrated thermocouples may be used.

6.7 Stirring Bar—Magnetic, PTFE (polytetrafluoro-ethylene)-coated, 25.4 mm \pm 2.54 mm (1.0 in. \pm 0.1 in.) long by 8 mm \pm 0.8 mm (0.31 in. \pm 0.03 in.) in diameter.

TABLE 1 Chemical Composition of Corrosion Test Rods

Element	Chemical Analysis—Content (%)											
	A108							C22E		BS970-1		
	1015		1018		1020		1025		1.1151 ^A		070 M20	
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Iron, Fe	99.13	99.57	98.81	99.26	99.08	99.53	99.03	99.48	98.365	99.43	98.36	99.24
Carbon, C	0.13	0.18	0.14	0.2	0.17	0.23	0.22	0.28	0.17	0.24	0.16	0.24
Manganese, Mn	0.3	0.6	0.6	0.9	0.3	0.6	0.3	0.6	0.4	0.7	0.5	0.9
Sulfur, S	0	0.05	0	0.05	0	0.05	0	0.05	0	0.035	0	0.05
Phosphorous, P	0	0.04	0	0.04	0	0.04	0	0.04	0	0.03	0	0.05
Silicon, Si	0		0		0		0		0	0.4	0.1	0.4
Nickel, Ni	0		0		0		0		0	0.4	0	
Chromium, Cr	0		0		0		0		0	0.4	0	

 $^{^{}A}$ (Cr + Mo + Ni = 0.63_{max})

⁵ The sole source of supply of the apparatus "Super-Nuova" Multi position stirring hot plate, Model No. SP135935Q known to the committee at this time is Thermo Fisher Scientific, 2000 Park Lane Dr., Pittsburgh, PA 15275-9952.

⁶ 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 The sole source of supply of the test jar cover known to the committee at this time is K & C Manufacturing, 210 S. Main, Newkirk, OK 74647.

⁸ The sole source of supply of the apparatus, with complete set of test accessories, known to the committee at this time is Koehler Instruments Co., 1595 Sycamore Ave, Bohemia, NY 11716.

- 6.8 *Syringe*—Plastic, 5 mL, with 63.5 mm (2.5 in.) long needle, 5 cc syringe and needle, or any other equivalent syringe and needle.
- 6.9 *Timing Device*—Electronic or manual, capable of measuring the test duration with an accuracy of at least 1 %.
- 6.10 Grinding and Polishing Apparatus—Any suitable apparatus, with a mounting or bench stand, capable of rotating at 1700 r/min to 1800 r/min, and having the means to mount a suitable chuck^{9,6} into which the corrosion test rod can be screwed in. See A1.4.

7. Reagents and Materials

- 7.1 Reagent Water—Type III or better, Specification D1193.
- 7.2 Wash Solvent.
- 7.2.1 2,2,4-trimethylpentane (isooctane)—Minimum 99.75 % purity. (Warning—Flammable. Health hazard.)
- 7.2.2 *Acetone*—Minimum 99.5 % purity. (**Warning**—Flammable. Health hazard.)
- 7.3 Hydrochloric Acid (HCl)—Prepare 15 % solution by dissolving 42 mL of concentrated HCl in 100 mL distilled water. (Warning—Corrosive. Burn hazard.)
- 7.4 *Polishing Material*—Abrasive cloth roll;^{10,6} silicon carbide, C-100 open mesh, 38 mm width by 22.5 m length (1.5 in. by 25 yd) roll.
 - 7.5 Magnifier—Lighted, 2× magnification power.
 - 7.6 Lintless Cloth or Tissue.
 - 7.7 Flat Brush, with medium to long soft hairs.
- 7.8 *O-ring*—Rubber, about 32 mm (1.25 in.) internal diameter, and 3 mm (0.12 in.) thick.
- 7.9 *Watch-glass*—For covering 150 mL bath beakers, 65 mm (2.6 in.) in diameter.
- 7.10 Absorbent Paper Towel—Any suitable type, for absorbing water from the outside of the test jar.
- 7.11 *Gloves*—Disposable, acid-resistant to 15 % HCl solution, and resistant to hydrocarbons used in this test method.

8. Preparation of Corrosion Test Rod

- 8.1 Wear clean gloves whenever handling the corrosion test rod.
- 8.2 If the corrosion test rod is new, first clean it with acetone.
- 8.3 Mount it in the chuck of the grinding and polishing apparatus, and rotate it at a speed of 1700 r/min to 1800 r/min.
- 8.3.1 Apply a new strip of the C-100 abrasive cloth perpendicular to the longitudinal axis of the test rod so that circular grooves are formed all around the circumference of the test rod, not criss-cross, or in any other irregular pattern.

- 8.3.2 Polish the test rod just enough to get rid of rust/other spots; do not overpolish. Discard used test rods when the diameter is reduced to 9.5 mm (0.375 in.) as indicated in Fig. A1.3.
- 8.3.3 To ensure that the flat shoulder (that portion of the test rod which is perpendicular to the threaded stem) is free of rust, polish this area by holding a strip of the abrasive cloth between the chuck and the shoulder while rotating the test rod for a brief period.
- 8.3.4 With the polisher still in operation, pass the brush gently over the entire surface of the corrosion test rod to remove the remains of the abrasive cloth and other foreign matter.
- Note 4—Check test rod with a lighted, 2× magnification power magnifier to ascertain freedom from rust/other spots, and completion of polishing operation.
- 8.3.5 Stop the polisher. Discard the gloves, which usually get contaminated with the residue of the abrasive cloth and other foreign matter. Wear a fresh pair of gloves. Remove the corrosion test rod with a clean, lint-free cloth or tissue-paper wrapped around the corrosion test rod.
- 8.3.6 Attach test rod to the holder, and, within 1 min, either immerse it in the sample to be tested through the 12.7 mm (0.5 in.) hole of the test jar cover, or, store it under *iso*octane up to 1 h before use.

9. Preparation of the Test Jar and Magnetic Stirring Bar

- 9.1 If the test jar or stirring bar is found to have a film of rust on it, treat it with a small amount of 15 % hydrochloric acid solution to clean out all the rust, then,
- 9.2 Wash it thoroughly with deionized water to free it from the acid, and finally,
- 9.3 Rinse with acetone and dry with lint-free paper or cloth before using.

10. Procedure

- 10.1 Prepare the corrosion test rod, test jar and magnetic stirring bar in accordance with Sections 8 and 9.
- 10.2 Prepare the test jar cover assembly by inserting the previously polished corrosion test rod with its holder down into the 12.7 mm (0.5 in.) hole up to its ridge. The tip of the test rod should be about 104 mm (4.1 in.) below the top of the cover.
- 10.3 Insert the external thermocouple or the digital TP through one of the 3.2 mm (0.125 in.) holes in the cover such that 57 mm (2.25 in.) of its stem is below the top of the cover. This should place the stem about 12.7 mm (0.5 in.) under the sample surface. Switch on the TP to display in degrees Celsius (degrees Fahrenheit).
- Note 5—Time interval between 10.2 and 10.3 should not exceed 1 min. Note 6—Tests should be run in well-ventilated spaces, or in a vented hood, because of the release of material from samples which are highly volatile, or which may contain potentially hazardous material.
- 10.4 Transfer 50 mL of sample into a clean test jar, gently introduce a stirring bar into the jar, and within 1 min, cover it with the assembly prepared under 10.2 and 10.3.

⁹ The sole source of supply of a semi-automated polishing apparatus known to the committee at this time is Koehler Instrument Co., 1595 Sycamore Ave., Bohemia, NY 11716.

 $^{^{10}}$ The sole source of supply of the polishing material, Part No. 8230A76, known to the committee at this time is McMaster-Carr Supply Co., PO Box 4355, Chicago, IL 60680-4355.