Designation: D7577 – 12 (Reapproved 2016)

Standard Test Method for Determining the Accelerated Iron Corrosion Rating of Denatured Fuel Ethanol and Ethanol Fuel Blends[1]

This standard is issued under the fixed designation D7577; 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 measures the ability of inhibited and uninhibited Ethanol Fuel Blends defined by Specification D5798 and Denatured Fuel Ethanol defined by Specification D4806 to resist corrosion of iron should water become mixed with the fuel, using an accelerated laboratory test method. Corrosion ratings are reported based on a visual, numbered rating scale.

1.2 The values stated in SI units are to be regarded as standard. The 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Sections 7 and 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A29/A29M Specification for General Requirements for Steel Bars, Carbon and Alloy, Hot-Wrought

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

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

D1193 Specification for Reagent Water

D2699 Test Method for Research Octane Number of Spark-Ignition Engine Fuel

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

D4806 Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel

D5798 Specification for Ethanol Fuel Blends for Flexible-Fuel Automotive Spark-Ignition Engines

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.1.2 *Fuel C, n*—a volumetric mixture of 50 volume percent reference fuel grade toluene and 50 volume percent reference fuel grade *iso*octane.

3.1.2.1 *Discussion*—Specifications for reference fuel grade toluene and reference fuel grade *iso*octane can be found in Test Method D2699.

3.2 *Abbreviations:*

3.2.1 *HDPE, n*—high density polyethylene

3.2.2 *PTFE, n*—Polytetrafluoroethylene

4. Summary of Test Method

4.1 A polished steel test rod is immersed in a mixture of the test sample and water at a ratio of 10 parts fuel sample to 1 part water and held at a temperature of 37 °C to 39 °C (98 °F to 102 °F) for 1 h.

4.2 At the end of 1 h, the test rod is removed, rinsed and rated according to a numeric corrosion rating scale.

5. Significance and Use

5.1 This test is designed to be used as a rapid measure of the overall relative corrosivity of Ethanol Fuel Blends (Specification D5798) and Denatured Fuel Ethanol (Specification D4806) to iron (steel).

5.2 The test can be used to compare corrosion inhibitor dosage levels and effectiveness of various corrosion inhibitors as they pertain to protecting iron (steel) materials from corrosion.

6. Apparatus

6.1 *General*—Two test apparatus have been evaluated and found to give comparable results.

[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 and Cleanliness 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.

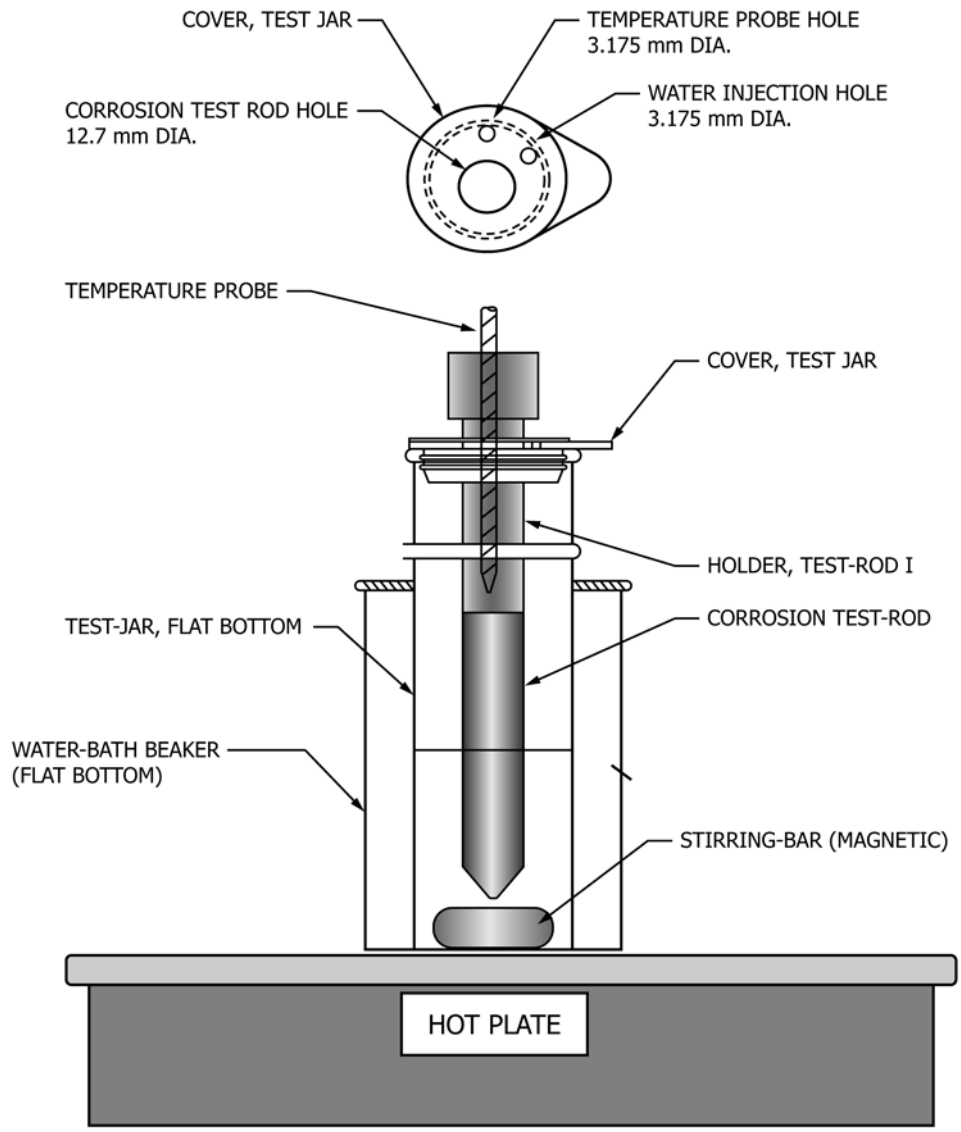

FIG. 1 Recommended Small Volume Test Apparatus

6.1.1 Large sample volume (300 mL) apparatus specified in Test Method D665.

6.1.2 Small sample volume (30 mL to 75 mL) apparatus specified in 6.2.

6.2 *Small Volume Test Apparatus (*Fig. 1*).*

6.2.1 Compared to Test Method D665, the small volume test apparatus is lower in cost and allows for use of smaller volumes of samples to improve the safety of the measurement. Different apparatus and components that achieve the same results may be used.

6.2.2 Hot plate/stir plate or water bath capable of maintaining a temperature of 37 °C to 39 °C (98 °F to 102 °F) and stirring at a rate of 900 r⁄min ± 100 r⁄min.

6.2.3 150 mL to 200 mL borosilicate glass beakers to hold water to serve as a water bath.

6.2.4 50 mL to 150 mL borosilicate, flat bottom, glass test jar to hold test sample.

6.2.5 Jar covers made of HDPE or other material compatible with ethanol, water and gasoline with three holes:

6.2.5.1 A hole to suspend the steel test rod into the test sample,

6.2.5.2 A hole for the thermometer,

6.2.5.3 A hole for inserting a syringe needle to add water to the test sample.

6.2.6 PTFE (polytetrafluoroethylene) coated magnetic stir bar.

6.2.7 The small volume test apparatus shall be designed so that at least 50 % of the test rod surface is below the surface of the test material.

6.3 *Grinding and sanding apparatus,* capable of rotating the steel test rod at 1700 r⁄min to 1800 r⁄min for manual sanding.

6.4 *Timing device,* capable of taking readings with a discrimination of 1 min or better.

6.5 *Analytical balance,* at least 100 g capacity, capable of weighing accurately to at least 0.001 g.

6.6 *Temperature measuring device,* Any thermometer with a temperature range that includes 37 °C to 39 °C (89 °F to

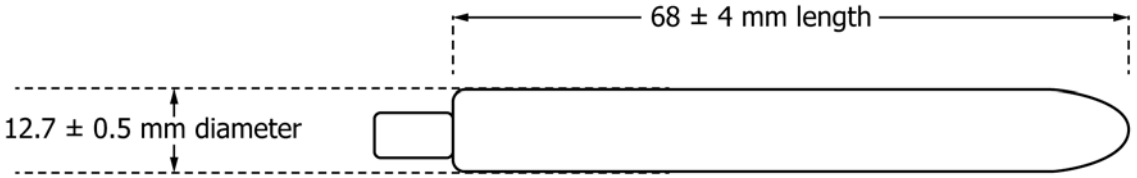

FIG. 2 Dimensions of New Steel Test Rod

102 °F), with one degree graduation subdivisions and conforming to the requirements prescribed in Specification E2251. Alternatively, calibrated thermcouples may be used.

7. Reagents and Materials

7.1 *Water*—References to water shall be understood to mean reagent water of grade Specification D1193 Type II or better.

7.2 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2.1 *Acetic acid*—(**Warning—**Corrosive. Health hazard.)

7.2.2 *Acetone*—(**Warning—**Flammable. Health hazard.)

7.2.3 *Formic acid*—(**Warning—**Corrosive. Health hazard.)

7.2.4 *Iso*octane *(2,2,4-trimethylpentane)*—(**Warning—**Flammable. Health hazard.)

7.2.5 *Reagent alcohol*—(**Warning—**Flammable. Health hazard.)–containing 90 volume % ethanol, 5 volume % isopropanol, 5 volume % methanol and <0.1 volume % water.

NOTE 1—The specified reagent alcohol must be used to achieve equivalent results and ratings to that reported in this test method.

7.2.6 *Sodium chloride.*

7.2.7 *Toluene*—(**Warning—**Flammable. Health hazard.)

7.2.8 *Fuel C*—A mixture of 50 volume percent toluene and 50 volume percent *iso*octane.

7.3 *Polishing Material*[4]—Abrasive cloth, silicon carbide or aluminum oxide, 100 grit.

7.4 *Pipette*—3 mL to 30 mL capacity, dependent on the amount of water required for a ratio of 10 to 1 test sample to water.

7.5 *Graduated cylinder*—50 mL to 300 mL capacity, dependent on the test apparatus, with divisions of 5 % or better of the total volume. For example, 50 mL sample volume should be measured using a graduated cylinder with graduations of 2.5 mL or less.

7.6 *Steel Test Rods:*

7.6.1 The steel test rod, when new, shall be 12.7 mm (0.5 in.) in diameter and approximately 68 mm (2 11⁄16 in.) in length exclusive of the threaded portion that screws into the PTFE holder and shall be tapered at one end as shown in Fig. 2.

7.6.2 The steel test rods shall be made of steel conforming to UNS Grade G10180 (AISI 1018) per Specification A108 (chemistry listed in Specification A29/A29M).

7.6.3 Discard reused rods when the diameter is reduced to 9.5 mm (0.375 in.).

7.7 *PTFE holders for steel test rods*—The PTFE holder screws onto the threaded end of the steel test rod.

8. Hazards

8.1 *Physical*—Care should be taken when manually polishing the steel test rods to avoid injury to hands. This test method also uses aggressive organic solvents; safety glasses should be worn at all times.

8.2 *Chemical*—Flammable, toxic and corrosive chemicals are used in this test procedure. It is the responsibility of the user to follow appropriate handling and storage procedures.

8.2.1 The test shall be run in a well-ventilated space or in a fume hood to avoid build up and exposure to fuel vapors. Test jar covers and secondary spill containers (water bath) are used to reduce the concentration of vapors and contain fuel spills.

9. Standard Preparation

9.1 Standards 1, 2, 3, 4 and 5 in Table 1 shall be prepared and tested when the test method is initially set-up in the laboratory or to demonstrate equivalency of test equipment.

9.2 It is required that one or more of the standards be prepared and tested in the following instances:

9.2.1 When new steel test rods are received.

9.2.2 When new operators are being trained on this procedure.

9.3 Testing of the standards in Table 1 provide the operator and individual laboratory with visual examples of the rating scale.

9.4 Standards are prepared by mixing 84 volume % reagent alcohol, 15 volume % Fuel C and 1 volume % water containing various concentrations of sodium chloride, formic acid and acetic acid. The final concentrations of chloride ion, formic

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the United States *Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[4] The sole source of supply of the abrasive cloth known to the committee at this time is available as Part No. 8230A76 from McMaster-Carr Supply Co., PO Box 4355, Chicago, IL, 60680-4355. 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.

TABLE 1 Concentration of Corrosive Components in 500 mL Standard Solutions Containing 84 Volume % Reagent Alcohol, 15 Volume % Fuel C and 1 Volume % Water

Standard No.	Rating	Chloride, mg/kg ± 10 %	Formic acid, mg/kg ± 10 %	Acetic acid, mg/kg ± 10 %
1	1	0	0	0
2	2	1.5	0	5
3	3	5	1	5
4	4	5	10	5
5	5	15	10	20

acid and acetic acid are shown in Table 1. Standard concentrations shall be prepared within ±10 % of the stated values in Table 1.

9.5 Example standard preparations are shown in Table 2. Water or water solutions containing sodium chloride, formic acid and acetic acid are added to a 500 mL volumetric flask containing approximately 300 mL of reagent alcohol. After stirring to mix, 75 mL of Fuel C is added and reagent alcohol is added to reach the 500 mL volume mark on the flask. The solutions are stirred until mixed adequately.

10. Preparation of Apparatus

10.1 Heat the water bath to a temperature of 37 °C to 39 °C (98 °F to 102 °F).

11. Preparation of Corrosion Test Rod

11.1 It is very important that clean, oil-free gloves are used, or similar precautions are taken, to avoid contamination of the test rod and abrasive cloth with fingerprints or other oils.

11.2 For new test rods, thoroughly clean the surfaces sequentially with acetone, toluene and *iso*octane to remove oils and other contamination before sanding the surface.

11.3 Mount the test rod in the chuck of the grinding and sanding apparatus.

11.4 Rotate the test rod at a speed of 1700 r⁄min to 1800 r⁄min while sanding the surface with a strip of the abrasive cloth.

11.4.1 *Preliminary Sanding*—Hold the 100 grit abrasive cloth strip perpendicular to the long-axis of the test rod so that circular grooves are formed all along the length of the rod. Move the cloth along the axis of the test rod. All rust and irregularities must be removed.

11.4.2 *Surface Marking*—Rub a new piece of abrasive cloth longitudinally over the static test rod until the entire surface shows visible scratches.

11.4.3 *Final Sanding*—Using a new piece of abrasive cloth, hold the 100 grit abrasive cloth strip perpendicular to the long-axis of the test rod so that circular grooves are formed all along the length of the rod. Move the cloth along the axis until all visible surface scratches from 11.4.2 have been removed.

11.5 Using a clean cloth or wipe, remove the test-rod from the chuck. Do not tough the surfaces with fingers.

11.6 Attach the PTFE rod holder.

11.7 Wipe the rod with a clean, lintless cloth or tissue.

11.8 Rinse the rod with acetone.

11.9 Store cleaned and sanded test rods in *iso*octane until use. Time between sanding and use should not exceed 1 h when stored in *iso*octane and should not exceed 5 min when not stored in *iso*octane.

12. Procedure

12.1 Prepare the corrosion test-rod and test apparatus in accordance with Sections 10 and 11.

12.2 Transfer the test sample or standard into the test jar using a graduated cylinder.

12.2.1 Use 300 mL of test sample or standard for the large sample volume apparatus specified in Test Method D665.

12.2.2 Use 30 mL to 75 mL of test sample or standard for the small sample volume apparatus specified in 6.2.

12.3 Place the stir bar or other stirring tool into the test jar and cover with the jar cover.

12.4 It is very important that clean, oil-free gloves are used, or similar precautions are taken, to avoid contamination of the test rod with fingerprints or other oils.

12.5 Insert the previously polished corrosion test rod with its holder down into the jar cover until at least half of the rod is immersed in the test sample or standard. The test rod shall not touch the bottom of the jar.

12.5.1 Do not let the clean test rod rub against the jar cover if a plastic cover is used. Transfer of material to the test rod can affect test results.

12.6 Insert the thermometer (6.6) into the jar cover until the tip is immersed in the test sample or standard.

12.7 Place the test-jar assembly containing the stirring tool, test sample or standard, test rod and thermometer (if used) into the pre-heated heating bath.

12.8 Start stirring the sample or standard at a rate sufficient to uniformly heat the solution.

12.9 When the sample or standard temperature reaches 37 °C to 39 °C (98 °F to 102 °F), inject water into the test sample or standard using a syringe.

NOTE 2—The addition of the water does not cause phase separation; because of the ethanol matrix of the test fuels covered in this scope of this method, the water is completely soluble in the test samples.

12.9.1 A ratio of ten parts test sample or standard to one part water shall be used. For example, 75 mL of test sample or standard is placed into a 150 mL test jar and 7.5 mL of water is added.

12.9.2 No more than 10 min shall pass between heating the sample or standard to temperature and adding the water.

12.10 Set the stirring rate to 900 r⁄min ± 100 r⁄min.

12.11 Heat and stir the test sample or standard for 60 min ± 5 min.

12.12 During the course of the test, if required, adjust the water bath temperature to maintain a sample or standard temperature of 37 °C to 39 °C (98 °F to 102 °F).

12.13 Remove the steel test rod and rinse with acetone.

TABLE 2 Example Standard Preparations

NOTE 1—Each solution is diluted to a total volume of 500 mL using reagent alcohol.

Standard No.	Rating	Fuel C, mL	Water, mL	Sodium chloride solutions	Formic acid solutions	Acetic acid solutions
1	1	75	5	0	0	0
2	2	75	1	3 mL of 325 mg/L of sodium chloride in water	0	1 mL of 1950 mg/L acetic acid in water
3	3	75	0	3 mL of 1070 mg/L of sodium chloride in water	1 mL of 390 mg/L formic acid in water	1 mL of 1950 mg/L acetic acid in water
4	4	75	0	3 mL of 1070 mg/L of sodium chloride in water	1 mL of 3900 mg/L formic acid in water	1 mL of 1950 mg/L acetic acid in water
5	5	75	0	3 mL of 3210 mg/L of sodium chloride in water	1 mL of 3900 mg/L formic acid in water	1 mL of 7800 mg/L acetic water in water

12.14 For standard solutions, within 5 min, photograph or permanently encase (for example, in clear epoxy) the steel test rods to use as the visual rating scale for future testing.

12.14.1 Descriptions of the standard test rods shall be comparable to those in Table 3.

12.14.2 Visual appearance of the standard test rods shall be comparable to Fig. 3.

12.14.3 Significant differences between laboratory results, Table 3 descriptions and Fig. 3 appearance shall be investigated. Reasons for differences include incorrect standard preparations, incorrect temperatures, unacceptable test rod preparation procedures and the use of an incorrect grade of steel test rod material.

12.15 For test samples, within 5 min, rate the discoloration of the test rod using the visual rating scale consisting of the standard test rods recorded or preserved in 12.14.

13. Interpretation of Results

13.1 Ratings shall be based exclusively on the portion of the steel test rod exposed to the test fluid. All discoloration or deposition of solids not removed by rinsing with acetone shall be considered as corrosion products.

13.2 Ratings shall be expressed according to the numerical (1 to 5) scale determined by testing the five standards prepared in Section 9 and tested in Section 12.

13.3 For corrosion in between rating levels, the higher rating level shall be reported.

14. Report

14.1 Report the corrosion rating from Section 13.

14.2 Reference this test method, including revision level, when reporting results.

15. Precision and Bias[5]

15.1 The precision of this test method is based on an interlaboratory study conducted in 2011. A total of six laboratories participated in this study, testing ten gasoline mixtures with ratings from 1 to 5. Each "test result" reported represents an individual determination and all participants were asked to report duplicate test results for each mixture. The details are given in ASTM Research Report RR:D02-1751.[5]

15.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" represents the critical difference between two test results for

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1751. Contact ASTM Customer Service at service@astm.org.

TABLE 3 Rating System[A]

Rating	Section 9 Standard	Appearance of Steel Test Rod Surface
1	1	No corrosion, no dulling and no discoloration of the surface. The surface exposed to the test sample has the same appearance as the unexposed surface. When rubbed on a clean, white cloth or paper towel, no discoloration is visible on the cloth or paper.
2	2	Appearance can range from dulling of the surface to slight discoloration of the exposed surface. When rubbed on a clean, white cloth or paper towel, discoloration is visible on the cloth or paper. Comparison with an unexposed surface of the test rod is helpful for this rating.
3	3	Exposed surface has a golden color, clearly visible without the use of special lighting.
4	4	Exposed surface has an orange/brown color. The polishing marks are clearly visible on the exposed surface.
5	5	Dark orange/brown "rust colored" discoloration of the surface. Polishing marks are less visible or not visible on the exposed surface.

[A]The appearance of these iron corrosion products from exposure to water in high ethanol fuels is very different from that observed in petroleum products using Test Method D665 and NACE TM-072 test methods. A numerical rating scale is used in this test method to signify this difference.

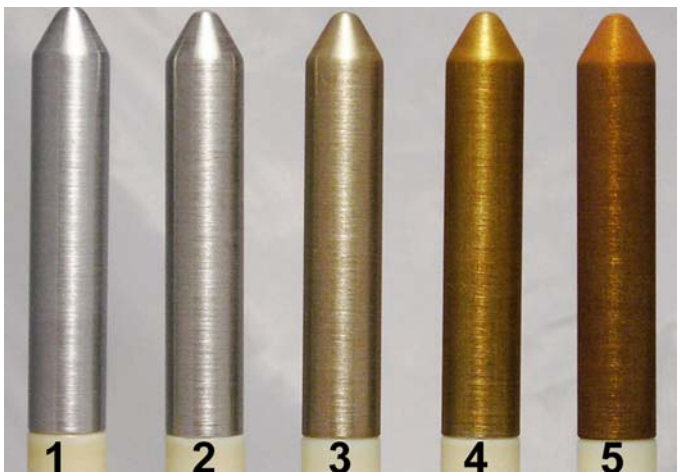

FIG. 3 Example of Visual Rating Scale. Photograph of Steel Test Rods at Each Corrosion Rating, Standards 1 to 5

TABLE 4 Relative Corrosivity (Rating)

Material	Average Rating[A] $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
70 % ethanol/30 % gasoline rating 1	1.25	0.29	0.47	0.81	1.30
95 % ethanol/5 % gasoline rating 1	1.17	0.00	0.41	0.00	1.14
70 % ethanol/30 % gasoline rating 2	1.25	0.65	0.65	1.81	1.81
95 % ethanol/5 % gasoline rating 2	1.25	0.65	0.65	1.81	1.81
70 % ethanol/30 % gasoline rating 3	3.42	0.29	0.53	0.81	1.49
95 % ethanol/5 % gasoline rating 3	2.42	0.50	0.68	1.40	1.91
70 % ethanol/30 % gasoline rating 4	3.83	0.71	0.72	1.98	2.01
95 % ethanol/5 % gasoline rating 4	2.67	1.15	1.15	3.23	3.23
70 % ethanol/30 % gasoline rating 5	4.92	0.29	0.29	0.81	0.81
95 % ethanol/5 % gasoline rating 5	4.42	1.19	1.19	3.33	3.33

[A]The average of the laboratories' calculated averages.

the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

15.1.1.1 Repeatability limits are listed in Table 4.

15.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" represents the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

15.1.2.1 Reproducibility limits are listed in Table 4.

15.1.3 The above terms (repeatability and reproducibility limit) are used as specified in Practice E177.

15.1.4 Any judgment made in accordance with 15.1.1 and 15.1.2 would have an approximate 95 % probability of being correct.

15.2 *Bias*—As there were no available standard reference materials at the time of this study, bias cannot be determined.

15.3 The precision statement was determined through statistical examination of 120 test results, submitted by 6 laboratories, recording corrosivity resistance measurements on 10 gasoline mixtures.

15.4 To judge the equivalency of two test results, it is recommended to choose the mixture that is closest in characteristics to the test material.

16. Keywords

16.1 corrosion; ethanol; steel