Designation: D4350 – 16

Standard Test Method for Corrosivity Index of Plastics and Fillers[1]

This standard is issued under the fixed designation D4350; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method is designed for use in obtaining the specific conductance of a water extract of plastics and fillers. The magnitude of this conductance, called the corrosivity index, is an index of the likelihood that, in a humid atmosphere, metal surfaces in contact with these materials can be corroded due to galvanic action or direct chemical attack.

NOTE 1—There is no known ISO equivalent to this standard.

1.2 The values stated in SI units are to be regarded as standard.

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 precautionary statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing
D883 Terminology Relating to Plastics
D1193 Specification for Reagent Water
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 *Definitions of Terms*—For definitions of terms used in this test method associated with plastics issues refer to the terminology contained in Terminology D883.

4. Significance and Use

4.1 This test method provides a means for comparing the corrosive potential of plastics and fillers in humid atmospheres.

4.2 This test method is intended for use in research and evaluation.

5. Apparatus

5.1 *Conductance Bridge,* Wheatstone type, with a range from 1 to 250 000-Ω measured resistance, a built-in potentiometer, a 1000 ± 50-cycles per second oscillator, and a sensitive null point indicator. The bridge shall be capable of measuring resistance with an accuracy of ±2 %.

5.2 *Conductivity Cell,* dip-type, micro, for solutions of medium conductance. The cell needs to have a cell constant of approximately 1.0 cm^{-1}. The borosilicate glass shall have a maximum outside tube diameter of 12.7 mm, overall length of 177.8 mm, chamber inside diameter of 9.5 mm, and chamber depth of 50.8 mm.[3]

5.3 *Drill,* electric, capable of holding a 10.54-mm drill bit, and rotating at 500-r/min maximum speed.

5.4 *Mill,* such as laboratory Wiley cutting mill or equivalent.

5.5 *Sieves,* standard (alternative) sieve designations 425 µm (No. 40), and 250 µm (No. 60) in accordance with Specification E11.

5.6 *Analytical Balance,* capable of determining mass to the nearest 1.0 mg.

5.7 *Oven,* forced-ventilation type, with uniformity of temperature within ±1 % of the differential between oven and

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.16 on Thermosetting Materials.

Current edition approved Nov. 1, 2016. Published November 2016. Originally approved in 1984. Last previous edition approved in 2015 as D4350 - 15. DOI: 10.1520/D4350-16.

[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 sole source of supply of the conductivity cell (Model No. 3403) known to the committee at this time is Yellow Springs Instrument Co., Inc., P.O. Box 279, Yellow Springs, OH 45387. 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.

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

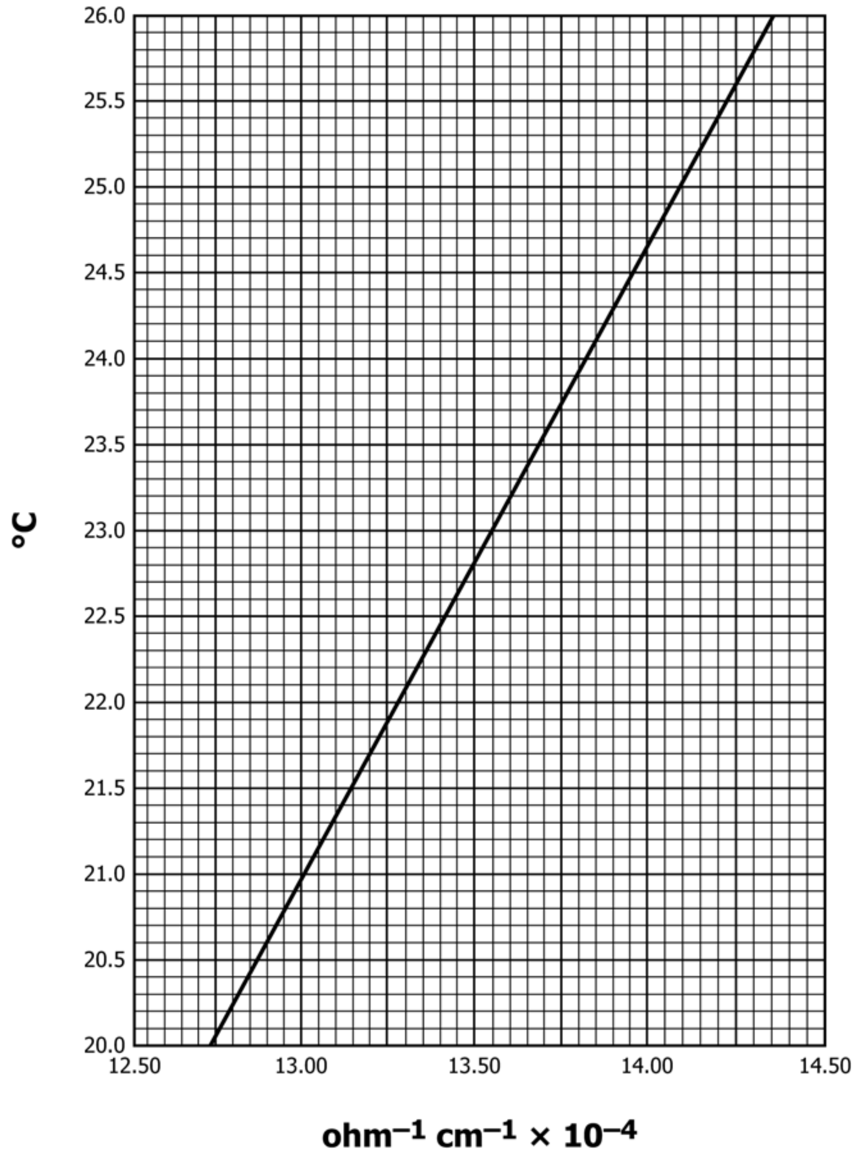

FIG. 1 Specific Conductance of 0.0100 Demal KCl

ambient temperature, with a rate of ventilation of 100 to 200 air changes per hour, in accordance with Specification E145, Type IIA.

5.8 *Thermometer,* solid-stem, precision, ASTM No. S63C, in accordance with Specification E2251. Temperature measuring devices with equivalent accuracy and characteristics, such as RTDs and thermistors, are permitted. Additionally, use of ASTM No. S63C in accordance with Specification E2251 is acceptable.

5.9 *Chemical Glassware:*

5.9.1 *Borosilicate Glass Flask,* nominally 1000-mL size, with ground glass stopper.

5.9.2 *Borosilicate Glass Erlenmeyer Flask,* 65-mL actual capacity to bottom of stopper (nominally 50-mL size), with ground glass stopper No. 19.

5.9.3 *Pipet,* volumetric, 50-mL capacity, calibrated "to deliver."

6. Reagents and Materials

6.1 *Distilled Water,* Type III, reagent water as defined in Specification D1193. When stored in borosilicate glass bottles at 23 ± 2°C, the water shall have a calculated specific conductance of less than 2.0×10^{-6}, ohm^{-1}, cm^{-1}.

6.2 *Potassium Chloride Solution,* consisting of 0.7453 g of reagent grade potassium chloride, previously dried at 105 ± 3°C for at least 24 h, dissolved in 1000 g of distilled water. The solution shall be stored in a borosilicate glass stoppered bottle. The specific conductance of this 0.0100 Demal KCl solution is 0.0007736 ohm^{-1}, cm^{-1} at 0°C, 0.0012205 ohm^{-1}, cm^{-1} at 18°C, and 0.0014087 ohm^{-1}, cm^{-1} at 25°C.[4] Obtain the value *k* from Fig. 1.

6.3 *Grease,* silicone, not soluble in water nor containing any water-soluble constituents. In the control specimens, water exposed to the grease on the stopper shall have a specific conductance less than 7×10^{-6}, ohm^{-1}, cm^{-1}.

7. Safety Hazards

7.1 Some plastics and fillers are known to contain toxic components and special precautions are required in handling. Diligently follow the manufacturer's precautionary instructions and sound laboratory safety practices.

8. Sampling

8.1 Because of the diverse nature of plastics and fillers, and the various forms and packages commercially available, no standard methods of sampling have been established. An adequate amount of material, representative of each ingredient, shall be selected from each lot to permit preparation of specimens as agreed upon between the buyer and the seller.

9. Specimen Preparation

9.1 *Plastics,* either prepared in accordance with the manufacturer's directions, or as received from the manufacturer, shall be drilled with a sharp drill at a rate not exceeding 27.5 mm/s (10.54-mm diameter drill at 500 r/min), and the drillings shall be ground in a mill. Care shall be exercised so as not to overheat the material when drilling or grinding, as overheating shall cause changes in the characteristics of the material. That fraction of ground plastics that passes a 425-µm sieve, but is retained by a 250-µm sieve, is used for the test.

9.2 *Fillers,* shall be used as received from the manufacturer.

10. Conditioning

10.1 Unless otherwise specified, condition all specimens for a minimum of 40 h at the standard laboratory atmosphere (23 ± 2°C, 50 ± 10 % relative humidity), in accordance with Procedure A of Practice D618.

11. Number of Test Specimens

11.1 At least three specimens shall be tested for each material.

12. Procedure

12.1 Place 0.50 ± 0.01 g of the test material in each of three Erlenmeyer flasks. Prepare at least three flasks, without material, as controls for the water and grease.

12.2 Add 50.0 mL of distilled water with a pipet to each flask.

12.3 Grease the flask stopper with silicone grease and stopper flasks tightly. Agitate the flasks until the specimen particles are thoroughly wetted.

12.4 Place the stoppered flasks in an oven at 71 ± 3°C for a total of 288 h (12 days). At the end of the first day of oven storage, examine the flasks to determine that no stoppers have become loose or blown off, with consequent loss of liquid (in which event the specimen shall be discarded). Agitate the flasks in order to break up large aggregates of the test specimen and to dislodge air bubbles that tend to float particles of the test specimen, thus preventing proper wetting.

12.5 At the end of 288 ± 2 h, remove the stoppered flasks from the oven and allow them to cool to 23 ± 2°C. Again agitate the flasks thoroughly and allow the solids to settle.

12.6 Determine the cell constant of the conductivity cell.

12.6.1 Pipet 50.0 mL of 0.0100 Demal KCl into each of three Erlenmeyer flasks, and allow to come to 23 ± 2°C.

12.6.2 Use a thermometer to determine the temperature to the nearest 0.1°C of the liquid in the flask immediately prior to the time of specific resistance measurements.

12.6.3 Dip the conductivity cell vertically into the liquid until the bottom edge of the cell rests on the bottom of the flask.

12.6.4 Measure the specific resistance in ohms of each of the solutions with a conductance bridge at 1000 ± 50 cycles per second.

12.7 Determine the specific resistance of the specimens and controls. Use the same technique and the same conductivity cell used in 12.6.3 and 12.6.4. Measurements shall be made at the same temperature determined in 12.6.2. Measurements shall be made within 4 h after removal from oven.

13. Calculation

13.1 Calculate the cell constant. The conductivity cell constant K is given by $K = kR$, where k is the specific conductance of the standard KCl solution at the temperature determined at the time of measurement,[5] and R is the measured resistance in ohms of the KCl solution. From the measured specific resistance value, calculate the cell constant K for each of the three KCl samples. K needs to be approximately 1.0 cm^{-1}. The three cell constant values shall be averaged as follows:

$$K = (K_1 + K_2 + K_3)/3 \tag{1}$$

No single value shall deviate from the mean value by more than 2 %.

13.2 Calculate the specific conductance S in ohm^{-1}, cm^{-1} for each specimen and control as follows:

$$S = K/R \tag{2}$$

where:

K = conductivity cell constant from 13.1, and

[4] Specific conductance values are based on the work of Jones, G., and Bradshaw, B. C., *J. Amer. Chem. Soc.*, 55 (1933) 1780. For more detailed information on the use of Demal KCl, see "Electrolyte Solutions," by Robinson, R. A., and Stokes, R. H., Academic Press, Inc., New York, 1955, pp. 94–96.

[5] The k value may be obtained from Fig. 1.

R = measured resistance in ohms of the specimen or control from 12.7.

13.3 Calculate the corrosivity index for each material tested. The corrosivity index is the average of the specific conductance S for the replicate specimens of each material, and is calculated as follows:

$$\text{corrosivity index} = (S_1 + S_2 + S_3)/3 \quad (3)$$

where S_1, S_2, and S_3 are the calculated specific conductances of the three replicate specimens from 13.2.

14. Report

14.1 Report the following information:

14.1.1 Dates of test,

14.1.2 Identification of plastics or filler material,

14.1.3 Temperature and total duration of test exposure,

14.1.4 Three cell constant values (K_1, K_2, and K_3) and the average cell constant K for the conductivity cell,

14.1.5 The measured specific resistance R of each specimen and each control,

14.1.6 The calculated specific conductance S of each specimen and each control, and

14.1.7 The calculated corrosivity index for each material in ohms^{-1}, $\text{cm}^{-1} \times 10^{-6}$.

15. Precision and Bias[6]

15.1 Table 1 is based on a round robin conducted in 1985, involving three materials tested by five laboratories. For each material, all the samples were prepared by one source, but each laboratory prepared the individual specimens that it tested. Each test result was based on one individual determination. Each laboratory obtained three test results for each material.

[6] Supporting data are available from ASTM Headquarters. Request RR:D20-1126.

TABLE 1 Precision Values in the Units of 10^{-6} ohm^{-1} cm^{-1}

Material	Average	S_r	S_R	I_r	I_R
Epoxy 1	25.5	3.30	8.35	9.3	23.6
Epoxy 2	30.0	1.80	7.23	5.1	20.5
Epoxy 3	104.0	3.87	19.5	11.0	55.2

15.2 In Table 1, for the materials indicated, and for test results that are derived from testing three specimens:

15.2.1 S_r is the within-laboratory standard deviation of the average; $I_r = 2.83\ S_r$. (See 15.2.3 for application of I_r.)

15.2.2 S_R is the between-laboratory standard deviation of the average; $I_R = 2.83\ S_R$. (See 15.2.4 for application of I_R.)

15.2.3 *Repeatability*—In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, judge those test results as not equivalent if they differ by more than the I_r values for that material and condition.

15.2.4 *Reproducibility*—In comparing two test results for the same material, obtained by different operators using different equipment on different days, judge those test results as not equivalent if they differ by more than the I_R value for that material and condition. (This applies between different laboratories or between different equipment within the same laboratory.)

15.2.5 Any judgment in accordance with 15.2.3 and 15.2.4 will have an approximate 95 % (0.95) probability of being correct.

15.2.6 Other formulations can give somewhat different results.

15.3 For further information on the methodology used in this section, refer to Practice E691.

15.4 There are no recognized standards on which to base an estimate of bias for this test method.

16. Keywords

16.1 corrosivity; plastics; fillers

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4350 - 15) that may impact the use of this standard. (November 1, 2016)

(1) Removed reference to Specification E1 from 2.1.
(2) Replaced Specification E1 with Specification E2251 in 5.8.

Committee D20 has identified the location of selected changes to this standard since the last issue (D4350 - 13) that may impact the use of this standard. (June 1, 2015)

(1) Eliminated permissive language in 1.1.
(2) Added references to Terminology D883 and Specification E2251 in 2.1.
(3) Eliminated old Section 3, Summary of Test Method and replaced with Section 3, Terminology.
(4) Eliminated the use of "should" in 5.2 and reworded the section.
(5) Replaced the word "shall" in 5.8 with vocabulary indicating that any thermometer type is allowed for use. Multiple thermometers are not required.
(6) Eliminated the use of "should" in 7.1 and reworded the section.
(7) Eliminated the use of "should" in 13.1 and reworded the section.

(8) Eliminated the use of "should" in 15.2.3 and reworded the section.
(9) Eliminated the use of "should" in 15.2.4 and reworded the section.
(10) Reworded 15.2.6 to clearly indicate that results may vary.