



Designation: **D 4350 – 9500**

An American National Standard

## Standard Test Method for Corrosivity Index of Plastics and Fillers<sup>1</sup>

This standard is issued under the fixed designation D 4350; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 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 may be taken as an index of the likelihood that, in a humid atmosphere, metal surfaces in contact with these materials may become corroded due to galvanic action or direct chemical attack; this is called the corrosivity index.

NOTE 1—There is no similar or equivalent ISO 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:*

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing<sup>2</sup>

<sup>1</sup> 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. 10, ~~1995~~; 2000. Published January ~~1996~~; 2001. Originally published as D 4350 – 84. Last previous edition ~~D 4350 – 86 (1990)~~. This edition includes an ISO Equivalency Statement and Keywords: D 4350 – 95.

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

D 1193 Specification for Reagent Water<sup>3</sup>

E 1 Specification for ASTM Thermometers<sup>4</sup>

E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>5</sup>

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens<sup>5</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>5</sup>

### 3. Summary of Test Method

3.1 Specimens of plastics or fillers are immersed in distilled water and exposed to specified conditions of temperature and time. The specific resistance of each of the solutions extracted from the test specimens is measured by using the specified conductivity cell. The specific conductance is calculated from the data and is called the corrosivity index of the material.

### 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 should have a cell constant of approximately 1.0 cm<sup>-1</sup>. The borosilicate glass shall have a maximum outside tube diameter of 12.7 ~~mm (0.5 in.)~~, mm, overall length of 177.8 ~~mm (7.0 in.)~~, mm, chamber inside diameter of 9.5 ~~mm (0.375 in.)~~, mm, and chamber depth of 50.8 ~~mm (2.0 in.)~~, mm.<sup>6</sup>

5.3 *Drill*, electric, capable of holding a 10.54-mm (~~0.415 in.~~) drill bit, and rotating at 500-rp/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 E 11.

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 ambient temperature, with a rate of ventilation of 100 to 200 air changes per hour, in accordance with Specification E 145, Type IIA.

5.8 *Thermometer*, solid-stem, precision, ASTM No. 63C, in accordance with Specification E 1.

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 D 1193. When stored in borosilicate glass bottles at 23± 2°C, the water shall have a calculated specific conductance of less than  $2.0 \times 10^{-6}$ , ohm<sup>-1</sup>, cm<sup>-1</sup>.

6.2 *Potassium Chloride Solution*, consisting of 0.7453 g of reagent grade potassium chloride, previously dried at 105 ± 3°C (222 ± 5°F) 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<sup>-1</sup>, cm<sup>-1</sup> at 0°C, 0.0012205 ohm<sup>-1</sup>, cm<sup>-1</sup> at 18°C, and 0.0014087 ohm<sup>-1</sup>, cm<sup>-1</sup> at 25°C.<sup>7</sup> This specific conductance versus temperature is plotted in 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  $7 \times 10^{-6}$ , ohm<sup>-1</sup>, cm<sup>-1</sup>.

### 7. Safety Hazards

7.1 Some plastics and fillers are known to contain toxic components and special precautions are required in handling. The manufacturer’s precautionary instructions and sound laboratory safety practices should be diligently followed.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 08.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.05.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>6</sup> A conductivity cell found suitable for this purpose is a Model No. 3403 conductivity cell, available from Yellow Springs Instrument Co., Inc., P.O. Box 279, Yellow Springs, OH 45387.

<sup>7</sup> 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.

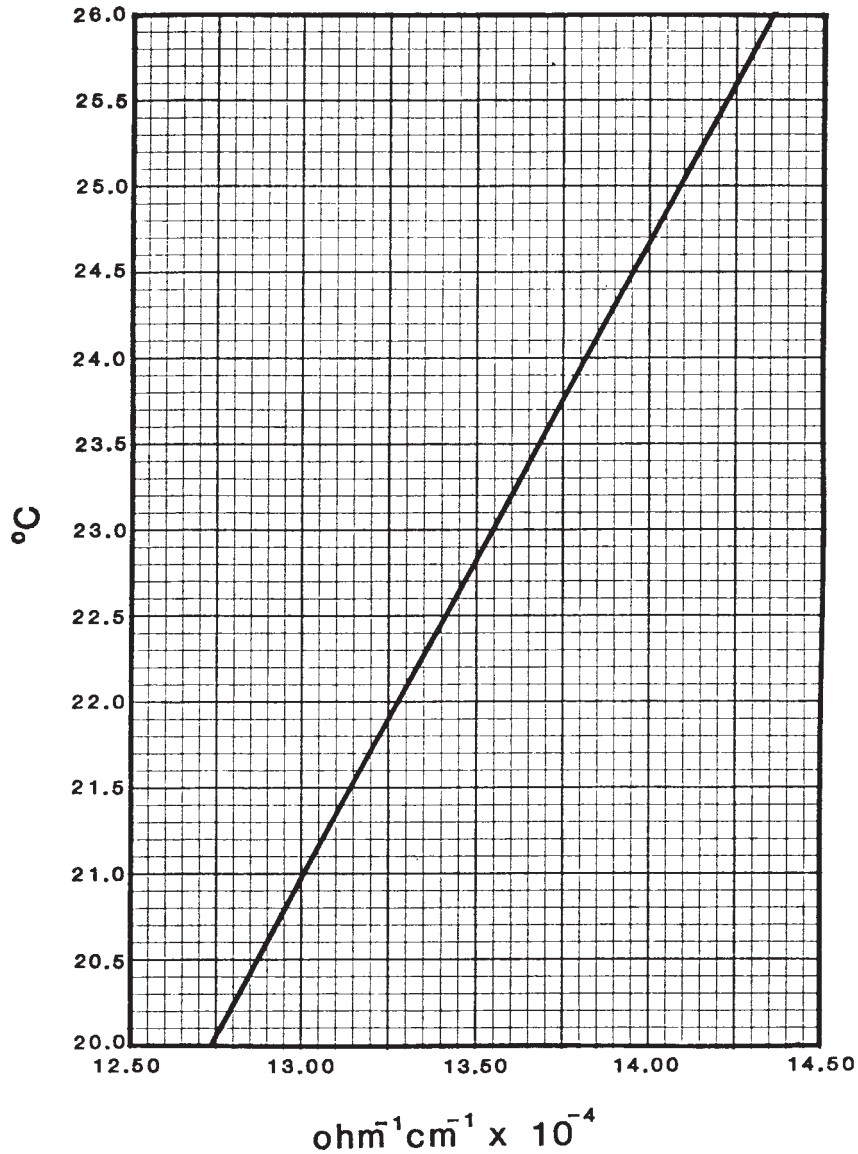


FIG. 1 Specific Conductance of 0.0100 Demal KCl

### 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 according to the manufacturer's directions, or as received from the manufacturer, shall be drilled with a sharp drill at a rate not exceeding  $1.08 \text{ in./s}$   $27.5 \text{ mm/s}$  ( $10.54\text{-mm}$   $\{0.415\text{-in.}\}$  diameter drill at  $500 \text{ rp/min}$ ), 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 may cause changes in the characteristics of the material. That fraction of ground plastics that passes a  $425\text{-}\mu\text{m}$  sieve, but is retained by a  $250\text{-}\mu\text{m}$  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 ( $23 \pm 2^{\circ}\text{C}$ ,  $50 \pm 5\%$  relative humidity), in accordance with Procedure A of Practice D 618.

### 11. Number of Test Specimens

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

## 12. Procedure

12.1 Place  $0.50 \pm 0.01$  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  $71 \pm 3^\circ\text{C}$  ( $160 \pm 5^\circ\text{F}$ ) 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  $288 \pm 2$  h, remove the stoppered flasks from the oven and allow them to cool to  $23 \pm 2^\circ\text{C}$ . 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  $23 \pm 2^\circ\text{C}$ .

12.6.2 Use a thermometer to determine the temperature to the nearest  $0.1^\circ\text{C}$  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  $1000 \pm 50$  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,<sup>8</sup> 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.  $K$  should be approximately  $1.0 \text{ cm}^{-1}$ . The three cell constant values shall be averaged as follows:

$$K = (K_1 + K_2 + K_3)/3 \quad (1)$$

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

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

$$S = K/R \quad (2)$$

where:

$K$  = conductivity cell constant from 13.1, and

$R$  = 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:

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

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

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  $\text{ohms}^{-1}, \text{cm}^{-1} \times 10^{-6}$ .

## 15. Precision and Bias<sup>9</sup>

15.1 Table 1 is based on a round robin conducted in 1985, involving three materials tested by five laboratories. For each

<sup>8</sup> The  $k$  value may be obtained from Fig. 1.

<sup>9</sup> Supporting data are available on loan at from ASTM Headquarters. Request RR:D20-1126.

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

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

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.

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, those test results should be judged not equivalent if they differ by more than the  $I_r$  value 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, those test results should be judged 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 may give somewhat different results.

15.3 For further information on the methodology used in this section, refer to Practice E 691.

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

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

### D 4350–00:

(1) Added 1.2.

(2) Removed inch-pound units throughout.

*ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.*

*This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).*