Designation: D1782 – 17

Standard Test Methods for Operating Performance of Particulate Cation-Exchange Materials[1]

This standard is issued under the fixed designation D1782; 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 These test methods cover the determination of the operating capacity of particulate cation-exchange materials when used for the removal of calcium, magnesium, and sodium ions from water. It is intended for use in testing both new and used materials. The following two test methods are included:

	Sections
Test Method A—Sodium Cycle	8 to 14
Test Method B—Hydrogen Cycle	15 to 21

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered 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, 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]

D1067 Test Methods for Acidity or Alkalinity of Water
D1126 Test Method for Hardness in Water
D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2687 Practices for Sampling Particulate Ion-Exchange Materials

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in these standards, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 Certain terms in these standards that relate specifically to ion exchange are defined as follows:

3.2.2 *free mineral acidity*—the quantitative capacity of aqueous media to react with hydroxyl ions to pH 4.3.

3.2.3 *hydrogen cycle*—the operation of a cation-exchange cycle wherein the removal of specified cations from influent water is accomplished by exchange with an equivalent amount of hydrogen ion from the exchange material.

3.2.4 *theoretical free mineral acidity*—the free mineral acidity that would result from the conversion of the anions of strong acids in solution to their respective free acids.

4. Summary of Test Methods

4.1 Test Method A consists of repeated cycles of backwash, brine regeneration, rinse, and exhaustion of the sample in the form of a bed in a transparent column. The exhausting medium used is an ion-exchange test water.

4.2 Test Method B consists of repeated cycles of backwash, acid regeneration, rinse, and exhaustion of the sample in the form of a bed in a transparent column. The exhausting medium used is an ion-exchange test water.

5. Apparatus

5.1 *Test Assemble* (see Fig. 1), consisting of the following:

5.1.1 *Column,* transparent, vertically supported, 25.4 ± 2.5 mm (1.0 ± 0.1 in.) in inside diameter and approximately 1500 mm (60 in.) long. The bottom of the column shall be closed and provided with an outlet of approximately 6-mm inside diameter. Connections shall be provided at top and bottom for admission and removal of solutions as described in Section 10. Adequate means for measuring and regulating flow shall be provided. Calibrate the column in such a manner that the volume readings required by the test method can be made. Make all measurements at 25 ± 5°C.

5.1.2 *Support,* for the sample, so designed that the distance from the sample to the column outlet is at least 50 mm. A

---

[1] These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

Current edition approved Aug. 1, 2017. Published August 2017. Originally approved in 1960. Last previous edition approved in 2009 as D1782 – 95 (2009). DOI: 10.1520/D1782-17.

[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.

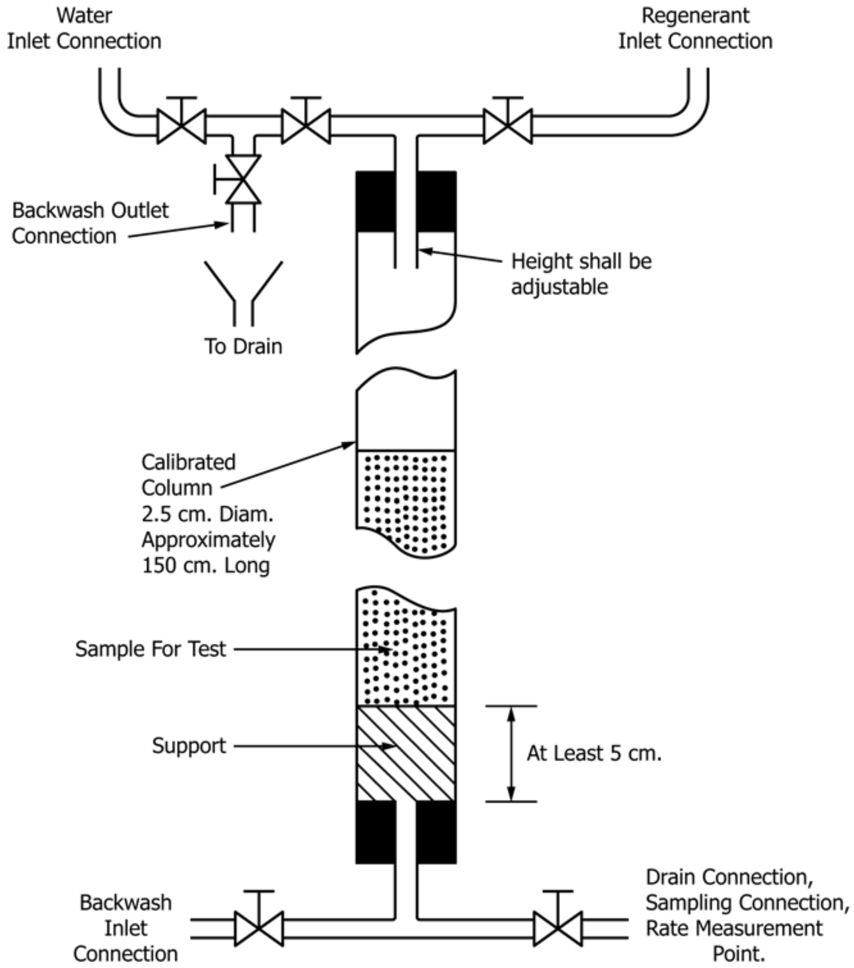

FIG. 1 Typical Arrangement of Apparatus for Performance Testing of Ion-Exchange Materials

suggested supporting bed utilizes quartz, glass beads, or other material 1.5 to 3.5 mm in diameter, insoluble in the reagents used, and retained on a corrosion-resistant screen. However, other supports such as fritted glass or polyester screens may be used at the discretion of the interested parties.

6. Reagents

6.1 *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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, Type IV, conforming to Specification D1193.

7. Sampling

7.1 To obtain a representative sample of particulate ion-exchange material, either from a shipment of new product in the manufacturer's original packages or from a bed of used material, refer to Practices D2687.

7.2 Transfer the laboratory sample to a 2-L beaker and add enough water to bring the water level up to that of the ion-exchange material and soak for 1 h (see Note 1). Mix the sample thoroughly and transfer a sufficient representative portion to fill a 400-mL beaker. Use this portion of sample in the procedure.

NOTE 1—Where new materials are shipped dry, follow the manufacturer's instructions for preconditioning.

TEST METHOD A—SODIUM CYCLE

8. Scope

8.1 This test method is designed to simulate operating conditions on a sodium cycle used for the removal of calcium and magnesium and other divalent ions from water.

9. Significance and Use

9.1 Cation exchange materials are frequently used in the sodium form to exchange divalent and trivalent ions in the

---

[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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

influent water for sodium ions on the resin sites. This process is commonly referred to as *softening* water since it removes those ions that form a "hard" curd of insoluble salts with the fatty acids used in some soaps and that also precipitate when water is boiled. In such a process, sodium chloride is used as the regenerant to return the cation-exchanging groups to the sodium form.

9.2 This test method is intended to simulate the performance of such materials in actual usage. It may be used either to compare the performance of new materials or to compare the performance of a material that has been used with its original performance.

9.3 Regenerant concentrations and dosages used herein are typical for the types of materials used in this application. If different concentrations or amounts of regenerant are agreed upon by parties using this test method, this fact should be stated when the results are reported. Similarly, the test water specified is the agreed upon standard. Where other test waters or the water to be treated are used in the test, the analysis of the water in terms of total solids, sodium, calcium, magnesium, other di- or trivalent metals as well as the major anions present should be reported with the test results.

10. Reagents and Materials

10.1 *Brine Regenerants:*

10.1.1 For synthetic organic ion-exchange materials:

10.1.1.1 *Sodium Chloride* (100 g/L)—Dissolve enough sodium chloride (NaCl) in water to make a solution containing in each litre 100.0 g of NaCl.

10.1.2 For all other ion-exchange materials:

10.1.2.1 *Sodium Chloride* (50 g/L)—Dissolve enough NaCl in water to make a solution containing in each litre 50.0 g of NaCl.

10.2 *Cation-Exchange Test Water A* (10 meq/L)—Dissolve enough calcium chloride ($CaCl_2 \cdot 2H_2O$) and magnesium sulfate ($MgSO_4 \cdot 7H_2O$) in water to make a solution containing, in each litre, 0.49 g of $CaCl_2 \cdot 2H_2O$ and 0.415 g of $MgSO_4 \cdot 7H_2O$. Adjust the pH to 7.5 by the addition of Na_2CO_3 (30 g/L) and determine the hardness of the solution in accordance with Test Method D1126. The hardness of the test water will be 10.0 ± 0.5 meq/L. Use the determined hardness in calculating operating capacity as indicated in 12.1. This test water shall be used for all tests.

10.3 *Hardness Test Reagents*—For reagents used in determining hardness, refer to Test Method D1126. This reagent is used only in preparation of test water (see 10.2).

10.4 *Sodium Carbonate Solution* (30 g/L)—Dissolve 30 g of sodium carbonate (Na_2CO_3) in water and dilute to 1 L.

11. Procedure

11.1 Adjust the temperature of the water and all solutions to be used in this procedure to 25 ± 5°C and maintain this temperature throughout the test.

11.2 Fill the column approximately half full of water and add sufficient sample to give a bed height of 750 ± 75 mm above the top of the support. To avoid drying out of the ion-exchange material, maintain a layer of liquid at least 20 to 30 mm deep above the top of the bed at all times during the procedure.

11.3 Backwash with water for 10 min using a flow rate that will maintain a 50 % expansion of the bed. If the supernatant liquid is clear at this point, proceed to 11.4. If the liquid is cloudy (indicating the presence of light, insoluble, extraneous material), adjust the backwash outlet tube to a height above the bed equal to 75 % of the bed height. Continue backwashing at the same rate until the effluent is clear.

11.4 Allow the bed to settle and then drain at a rate of approximately 100 mL/min until the water level is 20 to 30 mm above the top of the bed. Do not jar. Record the volume, in millilitres, of ion-exchange material. Repeat the 10-min backwash until two successive readings of volume agree within 5 mL. The average of these two readings shall be the sample volume for new materials shipped in the sodium form.

11.5 Exhaust the ion exchanger with cation-exchange test water A at a flow rate of 0.33 mL/min/mL of exchanger, as measured in 11.4. Maintain a head of liquid not less than 50 mm above the top of the bed. Continue the run until the effluent shows 0.2 meq/L (or other agreed-upon hardness level) when tested in accordance with Test Method D1126. Record the volume of test water used.

11.6 Repeat the 10-min backwash and drain as described in 11.3 and 11.4. When testing new material shipped in the sodium form, only one backwash is necessary at this point because a determination of volume has already been made. However, used material other than in the sodium form must have a volume determination made here as described in 11.4. Use this sample volume determined on the exhausted material in calculating the capacity of used ion-exchange materials.

11.7 Determine the amount of brine regenerant and rate required, from Table 1. For use with Table 1, the volume sample for new material shall be that determined in accordance with 11.4 and for used material shall be that determined in accordance with 11.6.

11.8 Pass the specified volume of brine regenerant through the bed at the specified rate until only a 20 to 30-mm layer of liquid remains above the bed. Rinse the bed with water, using

TABLE 1 Amount of Brine Regenerant Required for Use in Test Method A

Type of Exchange Material	Brine Regenerant,	Rate of Flow,	Contact Time,	Regeneration Level	
	g/L	mL brine/min/mL of Exchanger	min	lb/ft^3	g/L
Synthetic organic	100	0.032	30	6.00	96.1
Greensand	50	0.027	15	1.25	20.0
Synthetic siliceous	50	0.080	20	3.00	80.1
Carbonaceous	50	0.067	15	3.15	50.5

the same rate, until one bed-volume of liquid has been displaced. Increase the rinse rate to approximately 100 mL/min. Test for hardness at 3-min intervals by adding 0.5 mL of buffer solution to 50 mL of the effluent followed by three drops of hardness indicator and 0.5 mL of sodium ethylendiamine tetraacetate solution (1 mL = 1.0 mg $CaCO_3$), with stirring. If a blue color develops, the effluent contains 0.2 meq/L or less hardness and the rinse is completed. If the color is red, the end point has not been reached. Continue the rinse until the effluent shows 0.2 meq/L or less hardness.

11.9 Repeat the service run described in 11.5.

11.10 Repeat the cycle, beginning with a single backwash (see 11.6), omitting the determination of bed volume. Continue with a regeneration and rinse (see 11.8), and end with a service run (see 11.5). Repeat the cycle until each of three successive runs agrees within ±5 % of their average capacity as calculated in accordance with Section 12.

12. Calculation

12.1 Calculate the operating capacity, in milliequivalents per millilitre, of the ion exchange material as:

$$\text{capacity, meq/mL} = (A \times B)/S$$

where:

A = hardness of test water, meq/L,
B = volume of test water used in service run, L, and
S = volume of sample in the bed, mL. For new materials, this refers to the average volume of the material in the sodium form as determined in 11.4. For used materials, it is the average volume of the material in the exhausted form as determined in 11.6.

12.2 Calculate the operating capacity, in kilograins of calcium carbonate per cubic foot as follows:

$$\text{capacity, kilograins of } CaCO_3/\text{ft}^3 = C \times 21.8$$

where:

C = capacity in milliequivalents per millilitre of ion-exchange material (see 12.1).

13. Report

13.1 Report the capacity of the tested material as the average of three successive service runs that agree within ±5 % of their average capacity.

14. Precision and Bias[4]

14.1 *Precision*—Precision was determined from the results of the analyses of eight operators in four labs, with triplicate determinations on three materials. The average value determined for each material was as follows:

New Synthetic Organic	1.15 meq/mL
Used Synthetic Organic	1.03 meq/mL
New Synthetic Siliceous	0.43 meq/mL

14.2 The precision of the test method varies with the operating capacity of the material tested and may be expressed as follows:

$$S_t = 0.058\ C$$
$$S_o = 0.024\ C$$

where:

S_t = overall precision, meq/mL,
S_o = single operator precision, meq/mL, and
C = operating capacity, meq/mL.

14.3 *Bias*—Because materials with known operating capacity cannot be prepared, bias cannot be determined.

TEST METHOD B—HYDROGEN CYCLE

15. Scope

15.1 This test method is designed to simulate operating conditions on a hydrogen cycle used for the removal of all other cations from water.

16. Significance and Use

16.1 Cation exchange materials containing sulfonic acid groups are frequently used in the hydrogen form to exchange hydrogen ions from the resin sites for all the cations in the influent water. They are then regenerated with acid to restore the sites to the hydrogen form for reuse. This test is designed to simulate such usage.

16.2 Since each cation has a specific and different exchange equilibrium with hydrogen form exchange groups, the efficiency of the process will vary with the nature and concentration of the ions in the influent water. This test employs a mixture of the most commonly encountered ions in natural waters as its standard test water is intended for comparison of the performance of materials to determine relative performance either between new materials or between new material and the same material after it has been used in the field. The test may also be run, if the parties concerned so agree, using the water to be treated. When this is done, the analysis of the influent water and the end point selected must be included with the reported capacity.

16.3 This test method also provides for the use of either hydrochloric or sulfuric acid as regenerants since both of these acids are routinely used in this application. Concentrations other than those provided in the test method are also used, particularly if the influent water contains only small amounts of calcium. Tests may be made using other acid concentrations and amount, but when this is done that fact and the concentrations and amount used should be stated with the report.

16.4 Nitric acid is also sometimes used as a regenerant in this application. The user should recognize that this acid in combination with metals with catalytic properties such as iron and copper may act as an oxidant, giving rise to dangerous and uncontrollable reactions that liberate toxic gases with explosive potential.

---

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-0161. Contact ASTM Customer Service at service@astm.org.

17. Reagents and Materials

17.1 *Acid Regenerants:*

17.1.1 *Hydrochloric Acid* (100 g/L)—Carefully pour enough concentrated hydrochloric acid (HCl, sp gr 1.19) into water, stirring constantly, to make a solution containing in each litre 227 mL of HCl, sp gr 1.19.

17.1.2 *Sulfuric Acid* (20 g/L)—Carefully pour enough concentrated sulfuric acid (H_2SO_4, sp gr 1.84) into water, stirring constantly, to make a solution containing in each litre 11.2 mL of H_2SO_4, sp gr 1.84.

17.2 *Alkalinity Test Reagents*—For reagents used in determining alkalinity, refer to Test Methods D1067.

17.3 *Cation-Exchange Test Water B* (10 meq/L)—Dissolve enough sodium bicarbonate ($NaHCO_3$) in water to make a solution containing in each litre 0.21 g of $NaHCO_3$. Determine the alkalinity of the solution in accordance with Test Methods D1067, titrating to the methyl purple end point. The alkalinity of the solution shall be 2.50 ± 0.13 meq/L. Dissolve in this solution enough calcium chloride ($CaCl_2 \cdot 2H_2O$) and magnesium sulfate ($MgSO_4 \cdot 7H_2O$) to make a solution containing in each litre 0.368 g of $CaCl_2 \cdot 2H_2O$ and 0.311 g of $MgSO_4 \cdot 7H_2O$. Determine the total hardness of the solution in accordance with Test Method D1126. The total hardness of the solution shall be 7.50 ± 0.38 meq/L. Record the total hardness as meq/L of theoretical free mineral acidity for use in 18.3. Record the total strength of the solution as meq/L of alkalinity plus hardness for use in Section 19. This test water shall be used for all tests.

17.4 *Free Mineral Acidity Test Reagents*—For reagents used in determining the free mineral acidity, refer to Test Methods D1067.

17.5 *Hardness Test Reagents*—For reagents used in determining hardness, refer to Test Method D1126.

18. Procedure

18.1 Refer to 11.1, 11.2, and 11.3.

18.2 Allow the bed to settle and drain at a rate of approximately 100 mL/min until the water level is 20 to 30 mm above the top of the bed. Do not jar. Record the volume, in millilitres, of ion-exchange material.

18.3 Exhaust the ion exchanger with cation-exchange test water B at a flow rate of 0.33 mL/min/mL of exchanger as measured in 18.2. Maintain a head of liquid not less than 50 mm above the top of the bed. Continue the run until the free mineral acidity of the effluent has decreased to 90 % of the theoretical free mineral acidity of the test water. Determine the free mineral acidity of the effluent to the methyl purple end point. Record the volume of test water used.

18.4 Repeat the 10-min backwash and drain as described in 11.3 and 18.2, until two successive readings of volume agree within 5 mL. The average of these two readings shall be the sample volume for all subsequent operations.

18.5 Determine the amount of acid regenerant and the rate required from Table 2, using the sample volume determined in accordance with 18.4. The kind of acid used for synthetic organic materials shall be as agreed upon by the interested parties.

18.6 Pass the specified volume of acid regenerant through the bed at the specified rate until only a 20 to 30-mm layer of liquid remains above the bed. Rinse the bed with water, using the same rate, until one bed volume of liquid has been displaced. Increase the rinse rate to approximately 100 mL/min, and rinse for 20 min. To 25 mL of the effluent add 0.2 mL of methyl purple indicator solution and 0.25 mL of 0.02 *N* NaOH solution (see Note 2). If a green color develops, the effluent contains 0.2 meq/L or less of free mineral acidity and the rinse is completed. If a purple or gray color develops, the rinse is not completed. Continue the rinse until a 25-mL portion of the effluent does show 0.2 meq/L or less free mineral acidity.

NOTE 2—For the methyl purple indicator and standard sodium hydroxide solutions, refer to Test Methods D1067.

18.7 Repeat the service run described in 18.3.

18.8 Repeat the cycle, beginning with a single backwash (see 18.4), omitting the determination of the bed volume. Continue with a regeneration and rinse (see 18.6), and end with a service run (see 18.3). Repeat the cycle until each of three successive runs agrees within ±5 % of their average capacity as calculated in accordance with Section 20.

19. Calculation

19.1 Calculate the operating capacity, in milliequivalents per millilitre, of the ion-exchange materials as follows:

$$\text{capacity, meq/mL} = (A \times B)/S$$

where:

A = total strength of test water in meq/L; it equals the sum of the alkalinity and the hardness,
B = volume of test water used in service run, L, and
S = volume of sample in the bed, mL; this refers to the average volume of the material in the exhausted form as determined in 18.4.

19.2 Calculate the operating capacity, in kilograins of calcium carbonate per cubic foot, as follows:

$$\text{capacity, kilograins of } CaCO_3/\text{ft}^3 = C \times 21.8$$

TABLE 2 Amount of Acid Regenerant Required for Use in Test Method B

Type of Exchange Material	Acid Regenerant, g/L	Rate of Flow, mL acid/min/mL of Exchanger	Contact Time, min	Regeneration Level lb/ft^3	g/L
Synthetic organic	100 (HCl)	0.037	30	7.00	112
	20 (H_2SO_4)	0.212	30	8.00	126
Carbonaceous	20 (H_2SO_4)	0.107	15	2.00	32.6

where:

C = capacity in milliequivalents per millilitre of ion-exchange material (see 19.1).

20. Report

20.1 Report the capacity of the tested materials as the average of three successive service runs that agree within ±5 % of their average capacity.

21. Precision and Bias[4]

21.1 *Precision*—Precision was determined from the results of the analyses of eight operators in four labs, with triplicate determinations on three materials, using hydrochloric and sulfuric acid regenerations. The average value in meq/mL determined for each material was as follows:

	HCl Regen.	H_2SO_4 Regen.
New Synthetic Organic	1.38	0.70
Used Synthetic Organic	1.42	0.75
New Carbonaceous	...	0.23

21.2 The precision of the test method varies with the operating capacity of the material tested and may be expressed as follows:

$$S_t = 0.0615\ C$$

$$S_o = 0.0277\ C$$

where:

S_t = overall precision, meq/mL,
S_o = single operator precision, meq/mL, and
C = operating capacity, meq/mL.

21.3 *Bias*—Because materials with known operating capacity cannot be prepared, bias cannot be determined.

22. Quality Control

22.1 In the analysis of ion exchange resins, it is not possible to prepare a known standard resin for comparison with actual samples. Therefore, it is impossible to test the accuracy of the results, and these test methods do not include a bias statement.

22.2 Analysts are expected to run replicate samples to determine if the results are within the expected precision stated in Sections 14 and 21.

23. Keywords

23.1 cation resin; ion exchange; operating performance

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). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/