

Designation: D6284 - 17

Standard Test Method for Rubber Property—Effect of Aqueous Solutions with Available Chlorine and Chloramine¹

This standard is issued under the fixed designation D6284; 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 procedures for evaluating the ability of rubber and rubber-like materials to withstand the effects of aqueous solutions with available chlorine and chloramine. It is intended to compare the effects of chlorine compounds, present in potable water due to disinfection procedures, on rubber articles.
- 1.2 Test solutions are designed to contain chlorine compounds, including hypochlorous acid (HOCl), hypochlorite ions (OCl), and monochloramine (NH₂Cl).
- 1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.4 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.
- 1.5 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

D471 Test Method for Rubber Property—Effect of Liquids D1193 Specification for Reagent Water

D1253 Test Method for Residual Chlorine in Water

D1415 Test Method for Rubber Property—International Hardness

D2240 Test Method for Rubber Property—Durometer Hardness

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D6284 Test Method for Rubber Property—Effect of Aqueous Solutions with Available Chlorine and Chloramine

D6855 Test Method for Determination of Turbidity Below 5 NTU in Static Mode

3. Terminology

- 3.1 The terminology of chlorine compounds used in this test method is in accordance with Test Method D1253, Section 7.
- 3.2 combined residual chlorine—residual consisting of chlorine combined with ammonia nitrogen and nitrogenous compounds.
- 3.3 free available chlorine residual—residual consisting of hypochlorite ions (OCI) or hydrochlorous acid (HOCl), or both
- 3.4 *total residual chlorine (chlorine residual)*—the amount of available chlorine-induced oxidants present in water at any specified period, subsequent to the addition of chlorine.

4. Summary of Test Method

- 4.1 This test method provides procedures for exposing test pieces to aqueous available chlorine and chloramine solutions under defined conditions of temperature and time.
 - 4.2 This test method includes the following procedures:

 Change in mass after immersion
 Section 10

 Change in volume after immersion
 Section 11

 Change in hardness after immersion
 Section 12

 Visual and turbidity rating after immersion
 Section 13

4.3 The resulting deterioration is measured by changes in mass, volume, hardness, and appearance of the test piece surfaces and test liquid after immersion.

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.40 on Consumer Rubber Products.

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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.

4.4 This test method also describes the preparation of the immersion liquids.

5. Significance and Use

- 5.1 Rubber articles, such as seals, gaskets, and membranes, may be exposed in service to chlorine compounds used in potable water as disinfectants. The exposure may be intermittent or continuous and can occur at various temperatures.
- 5.2 Properties of rubber articles can deteriorate as a result of exposure to water containing these chlorine compounds, affecting their performance for the intended use.
- 5.3 This test method attempts to simulate service conditions through controlled accelerated testing, but may not give a direct correlation with part performance under actual service conditions. It yields comparative data on which to base judgement on expected service quality.
- 5.4 This test method is suitable for compliance testing, quality control, and research and development work.

6. Test Conditions

- 6.1 *Temperature*—Testing can be performed at room temperature out of direct sunlight. If elevated temperatures are required, testing will be performed in a calibrated oven. Unless otherwise specified between the producer and the purchaser, the preferred test temperature shall be $70 \pm 2^{\circ}\text{C}$.
- 6.1.1 If the temperature of the testing room is other than the standard $23 \pm 2^{\circ}$ C, the actual room temperature and any effect on the temperature of the test liquids shall be reported.
- 6.2 *Immersion Periods*—Unless otherwise specified, the immersion shall be performed in one week intervals depending on the anticipated service conditions. Specific time intervals will be outlined by the customer and then listed in report (refer to 15.1.3).
- 6.2.1 To obtain a measure on the rate of deterioration, it is necessary to take measurements after several immersion periods. The tolerance of the immersion times shall be ± 15 min or ± 1 % of the specified time, whichever is greater.

7. Apparatus

- 7.1 An appropriate container able to hold the volume of solution that is no less than 3.13 mL/cm² (ratio of media volume per square centimeter of sample).
- 7.2 Test procedures utilizing continuous monitoring of test liquids and automatic addition of reagents may be used but can be expected to produce different results than those obtained by the procedures of this test method.
- 7.3 As an alternate option to Test Method D1253, a meter may be used to measure total residual chlorine.³

8. Standard Test Liquids

8.1 Standard test liquids are prepared in amber glass jars of sufficient volume to hold 1900 cm³ of the liquid.

- 8.2 Fresh test solutions shall be prepared on a daily basis. At no time throughout the entire test period shall test pieces be allowed to remain in the same solution for more than 48 h.
- 8.2.1 If the test period is to be 166 h or greater, the test solutions shall be left unchanged for one day per week.
- 8.3 Total residual chlorine levels of at least one freshly prepared available chlorine and monochloramine solution shall be determined weekly.
- 8.4 Test solutions are transferred at room temperature unless specified by a specific test protocol to be performed at a higher temperature. If so solutions are heated in loosely sealed amber glass jars in a suitable water bath or hot forced-air oven to 70 \pm 2°C prior to transferring them into test vessels in accordance with Section 9.
- 8.4.1 An acceptable alternative method is the use of a glass container of sufficient volume to prepare a large batch of test solution capable of filling all test vessels with 1900 cm³ of solution each. Prior to filling the test vessels, the liquid shall be stirred and checked with an immersion thermometer to ensure that the specified test temperature has been reached throughout the batch.
 - 8.5 Preparation of Standard Test Liquids and Solutions:
- 8.5.1 *Reagent Water*—Distilled or deionized water (Type III or purer, see Specification D1193) is heated to the test temperature in a loosely sealed amber glass jar and transferred to the test vessel.
- 8.5.2 Available Chlorine and Chloramine Test Solution—A volume of 1900 cm³ of distilled or deionized water (Type III or purer, see Specification D1193) is mixed at room temperature in a loosely sealed amber glass jar with 0.9 cm³ of sodium hypochlorite solution with 10 % minimum total residual chlorine⁴ and 7.0 cm³ of phosphate buffer solution (pH 7.0) is then added using a graduated pipette. The glass jar is conditioned for 10 ± 1 min at room temperature and the solution tested for total residual chlorine content. Solution strength shall be 50 ± 5 ppm (mg/L) of total residual chlorine determined in accordance with Test Method D1253, Section 10 or using the apparatus listed in 7.3. If the solution is not within the required limits, adjust accordingly and retest for total residual chlorine content. The prepared solution is transferred into the test
- 8.5.2.1 Chloramine T shall not be used as a replacement for any of the reagents specified in the preparation of the test solutions.
- 8.5.2.2 Sodium hypochlorite solutions are not stable and will change their concentration in storage. It is therefore recommended to use fresh solutions or store solutions in the dark under refrigeration (about 5°C). The concentration of solutions stored for any length of time should be confirmed by determining their total residual chlorine level in accordance with Test Method D1253, Section 10. If the concentration has

³ The unit produced by Palintest LTD is an example of a meter able to measure both total and free residual chlorine. The "ChloroSense meter" is equipped with ChloroSense sensors and calibration chips. The instructions for proper use of the meter are outlined in the unit's instructions.

⁴ The sole source of supply of the solution (Catalog No. SS-290) known to the committee at this time is Fisher Scientific, 2000 Park Lane Drive, Pittsburgh, PA 15275. 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 Dimensions of Standard Specimens

Туре	Length, mm	Width, mm	Thickness, mm
1	50.0 ± 0.1	25.0 ± 0.1	1.0 ± 0.1
2	50.0 ± 0.1	25.0 ± 0.1	2.0 ± 0.1
3	50.0 ± 0.1	25.0 ± 0.1	4.0 ± 0.1

changed, the necessary adjustments shall be made in the preparation of the test solutions.

8.5.3 Monochloramine Test Solution-A volume of 1900 cm³ of distilled or deionized water (Type III or purer, see Specification D1193) is mixed at room temperature in a loosely sealed amber glass jar with 0.9 cm³ of sodium hypochlorite solution with 10 % minimum total residual chlorine⁴ and 4.0 cm³ of phosphate buffer solution (pH 7.0) is then added using a graduated pipette. The glass jar is conditioned for 10 ± 1 min at room temperature followed by the addition of $2.7 \text{ cm}^3 1 N$ ammonium hydroxide and 8.0 cm³ of phosphate buffer solution⁴ (pH 7.0). The glass jar is conditioned for 10 ± 1 min at room temperature and the solution tested for total residual chlorine content. Solution strength shall be 50 ± 5 ppm (mg/L) of total residual chlorine determined in accordance with Test Method D1253, Section 10 or using the total chlorine probe apparatus listed in 7.3. If the solution is not within the required limits, adjust accordingly and retest for total residual chlorine content. The prepared solution is transferred into the test vessels.

9. Test Specimens

- 9.1 Unless otherwise specified, test pieces shall be prepared in accordance with Practice D3182 or Practice D3183.
- 9.2 Dimensions of standard test pieces shall be as indicated in Table 1. Type 2 specimens shall be cut from standard vulcanized sheets prepared in accordance with Practice D3182. Sheets for Type 1 and 3 specimens shall be vulcanized in molds modified for cavity depth, but having otherwise the same dimensions as specified in Practice D3182 (Fig. 1, 2, 3, or 4).
- 9.3 Type 1 specimens are preferred for accelerated testing. Types 2 and 3 specimens may be specified by mutual agreement between the producer and the purchaser.
- 9.4 Actual parts may be used as specimens instead of dumbbell specimens if it is requested. If parts are used certain measurement procedures must be adjusted to accommodate the shape and size of the specimen. Durometer will be changed to micro hardness.
- 9.5 The same test pieces are used to determine changes in mass, volume, hardness, and visual rating.
- 9.6 If rubber parts or nonstandard test pieces are used, the procedure shall be adjusted to maintain a minimum volume of 20 cm³ of test solutions per 1 cm² of test piece surface area. Data obtained on test pieces of different original thicknesses are not comparable.

10. Procedure for Change in Mass

- 10.1 See Test Method D471, Section 10.
- 10.2 Test three test pieces of a single composition. Calculate the test results of the three test pieces in accordance with Eq 1 and average the results.

- 10.3 Weigh each test piece in air to the nearest 1 mg, record the mass as M_1 , and mount three test pieces on a glass rod so that a minimum of 6 mm separation is maintained between the test pieces through the use of spacers, for example, perforated glass beads. It is important that the materials used to suspend and separate the test pieces do not affect the test liquid and the rubber.
- 10.4 Place the test piece setup in the test vessel (see 9.1) containing the test liquid for the specified time and remove each test piece from the flask at the end of the immersion period.
- 10.5 Cool the test pieces to room temperature by transferring them into a cool and clean portion of the test liquid for 30 min. After removal from the liquid, lightly blot the test pieces dry with lint-free paper, place them in a tared stoppered weighing bottle, weigh each test piece to the nearest 1 mg, record the mass as M_2 .
- 10.6 Return the test pieces to the cool and clean portion of the test liquid before proceeding with hardness testing and visual rating. After completing all testing return the test pieces to the test vessel with the same test liquid, if data are desired on progressive changes that occur with increasing immersion times.
- 10.7 It is important that each manipulation following removal of the test pieces from the test liquid takes place promptly with the least possible lapse in time.
- 10.8 Prepared fresh test liquids daily in accordance with 8.2, except as described in 2.1. Discard the old solutions after evaluating turbidity and add fresh solutions to the test vessel (without removal of the test pieces).

11. Procedure for Change in Volume

- 11.1 See Test Method D471, Section 11.
- 11.2 Test three test pieces of a single composition. Calculate the test results of the three test pieces in accordance with Eq 2 and average the results.
- 11.3 Weight each test piece in air to the nearest 1 mg and record the mass as M_1 . Then obtain the mass M_2 of each test piece immersed in distilled water or methanol (Note 1) at room temperature.

Note 1—Methanol may be employed in place of water if the removal of air bubbles from the test piece surface is a problem.

- 11.4 Quickly blot the test pieces dry using lint-free paper, mount three test pieces on a glass rod in accordance with 10.3, and place the test piece setup in the test vessel (see 9.1) containing the test liquid for the specified time.
- 11.5 Remove each test piece from the flask at the end of the immersion period and cool to room temperature in a cool and clean portion of the test liquid for 30 min. Lightly blot each test piece dry with lint-free paper, place it in a tared stoppered weighing bottle, weigh to the nearest 1 mg, and record the mass as M_3 . Remove each test piece from the weighing bottle, weigh in distilled water or methanol to the nearest 1 mg, and record the mass as M_4 .



- 11.6 Return the test pieces to the cool and clean portion of the test liquid before proceeding with hardness testing and visual rating. After completing all testing return the test pieces to the test vessel with the same test liquid, if data are desired on progressive changes that occur with increasing immersion times.
- 11.7 It is important that each manipulation following removal of the test pieces from the test liquid takes place promptly with the least possible lapse in time.
- 11.8 Prepare fresh test liquids daily in accordance with 8.2, except as described in 2.1. Discard the old solutions after evaluating turbidity (Section 13) and add fresh solutions to the test vessel (without removal of the test pieces).

12. Procedure for Change in Hardness

- 12.1 See Test Method D471, Section 15.
- 12.2 Measure hardness in accordance with Test Method D2240 or D1415 as appropriate based on the tested specimen's dimensions.
- 12.3 Ply Up Three Standard Test Pieces (Section 9) for measuring hardness before and after immersion. Make hardness measurements as close as possible to the center of the test pieces.
- 12.4 Measure the hardness of the top piece and then place it to the bottom of the stack. Repeat the same procedure for the remaining two test pieces and report the median of the three readings. The time interval between removal from the cooling liquid and testing shall not be less than 2 nor more than 3 min.
- 12.5 The hardness before immersion is referred to as $H_{\rm O}$ and the hardness after immersion as $H_{\rm j}$. Calculate hardness change in accordance with Eq 3.
- 12.6 If data on progressive changes with increased immersion times are required, return the test pieces to the vessel with the same test liquid.

13. Procedure for Visual and Turbidity Rating

- 13.1 Inspection of Test Piece Surface:
- 13.1.1 Visually examine the surface appearance of immersed test pieces for signs of progressive degradation in comparison to an unexposed test piece or control specimen of known degradation. Note and record changes based on the following rating system in the test piece surface and immersion media.
- 13.1.1.1 Chloramine/Chlorine Visual Rating System of Elastomers:

No change. Rating = A

Swelling is low to moderate.

Water is clear / No visual changes

Discoloration. Rating = B

Blooming/whitening of sample. Water is clear.

Some Residue may be developing on samples.

Slight Degradation. Rating = C

Edge of sample has degradation / wrinkles
Outline of sample shows on paper / black marks on paper
Some slight surface degradation
Water is clear.

Deformation/Blister, Rating = D

Sample no longer lies flat / edges wrinkled Some slight surface degradation / black marks on paper Blisters form on sample / deformed Water is clear.

Moderate Degradation. Rating = E

Entire surface has degradation / wrinkled edges Surface area of sample shows on paper / black marks on paper No chunks / small pieces / floaters of material in water. Water is slightly cloudy or discolored.

Severe Degradation. Rating = F

Entire surface has degradation.
Sample is swollen/distorted/textured.
Small to Medium floaters of material in water / black pieces on paper
Water is cloudy or discolored.

Severe Fail. Rating = G

Material chunks / noticeable pieces / floaters in water. Water is cloudy or discolored.
Entire surface has degradation.
Sample is noticeably swollen / distorted

Other. Rating = H

Describe other visual observations

- Note 2—The chlorine/chloramine visual rating chart (see Appendix X1) is intended to provide the customer with useful information regarding the observed condition of both the tested sample and the immersion media as testing progresses. It is not a pass/fail criterion.
- 13.1.2 If data on progressive changes with increased immersion times are required, return the test pieces to the vessel with the same test liquid.
 - 13.2 Turbidity of Test Liquid:
- 13.2.1 This test is used to determine the progressive degradation that has occurred in the test pieces since the last test liquid change.
- 13.2.2 At least 24 h after the last test liquid change, remove the test pieces from the solution, vigorously shake the solution in the flask for a minimum of 2 min and immediately remove a sample of the liquid for turbidity testing in accordance with Test Method D6855.
- 13.2.3 Record and report readings in nephelometric turbidity units (NTU).

14. Calculation

14.1 Calculate the percent change in mass as follows (Section 10):

$$\Delta M, \% = \frac{(M_2 - M_1)}{M_1} \times 100 \tag{1}$$

where:

 ΔM = change in mass, %,

 M_1 = initial mass of test piece in air, g, and

 M_2 = mass of test piece in air after immersion, g.

14.2 Calculate the percent change in volume as follows (Section 11):

$$\Delta V, \% = \frac{(M_3 - M_4) - (M_1 - M_2)}{(M_1 - M_2)} \bullet 100$$
 (2)

where:

 ΔV = change in volume, %,

 M_1 = initial mass of test piece in air, g,



 M_2 = initial mass of test piece in water or methanol, g,

 M_3 = mass of test piece in air after immersion, g, and

 M_4 = mass of test piece in water or methanol after immersion, g.

14.3 Calculate the hardness change in hardness units as follows (Section 12):

$$\Delta H = H_i - H_o \tag{3}$$

where:

 ΔH = hardness change after immersion, units,

 H_0 = initial hardness, units, and

 H_i = hardness after immersion, units.

15. Report

15.1 State that the test was conducted in accordance with Test Method D6284 and report the following information:

15.1.1 Description of the sample and its origin,

15.1.2 Date and temperature of testing room or oven,

15.1.3 Duration, temperature, and date and vulcanization of test pieces,

15.1.4 Dates of the various periods of exposure,

15.1.5 Type of immersion liquid(s) used,

15.1.6 Temperature and duration of exposure,

15.1.7 Test results on weight, volume, and hardness changes after immersion,

15.1.8 Visible changes of test piece surface and immersion media after exposure and rated according to 13.1.1.1,

15.1.9 Turbidity of immersion liquid(s) in nephelometric turbidity units (NTU), and

15.1.10 Any deviation from standard test methods.

16. Precision and Bias⁵

Note 3—The interlaboratory test program (ITP) for this standard was not executed according to the guidelines as set forth in Practice D4483. It was a simplified program that did not conduct replicate test result measurements within any laboratory in the program. For any normal evaluation of precision, these replicate test result measurements, usually obtained in a day–1 versus day–2 sequence for duplicates, are required for the evaluation of repeatability.

16.1 The ITP was conducted during the time period 1998-2000 using five different rubber compounds: (1) FKM, (2) EPDM/polypropylene thermoplastic vulcanizate, (3) sulfur vulcanized SBR, (4) peroxide vulcanized NBR, and (5) sulfur vulcanized EPDM.

16.1.1 Each of these was tested once for volume swell, using four different immersion/aging periods: 1, 2, 3 and 4 weeks at 70°C. Other physical properties were not part of the ITP. A test result is the average for three test specimens as specified in 11.2. Only four laboratories volunteered for the program. This is an inadequate number for a valid evaluation of precision. The data that have been obtained have been used however to obtain the best possible estimate for repeatability and reproducibility for this particular program. The values for reproducibility that are given below can be considered as approximate. The special values for repeatability are estimated as discussed below.

TABLE 2 Estimated Type 1 Precision

Note 1—Property = Percent volume swell at 70° C as mean value for 4 weeks immersion.

Material	Mean	Sr	r	(<i>r</i>)	SR	R	(<i>R</i>)	No. Labs
Cmpd. B	4.4	0.11 ^A	0.30 ^A	6.3 ^A	0.37	1.05	22.9	4
Cmpd. A	8.6	0.30	0.83	9.0	5.02	14.04	172.5	4
Cmpd. E	16.3	0.81	2.3	17.0	1.6	4.4	36.6	2
Cmpd. D	26.2	3.3	9.2	53.5	18.5	51.8	99.3	3
Cmpd. C	49.4	2.1	5.8	13.3	8.2	22.9	35.3	3

^A These columns are only estimated; see text of standard for details.

Sr = Estimated repeatability standard deviation, units: % swell.

r = estimated repeatability, in measurement units, % swell.

(r) = estimated relative repeatability, percent of percent.

SR = reproducibility standard deviation, units: % swell (approximate value).

(R) = relative reproducibility, percent of percent (approximate value).

16.2 The precision results as determined by this ITP may not be applied to acceptance or rejection testing for any group of materials or products without documentation that the results of this precision evaluation actually apply to the products or materials tested.

16.3 Special Repeatability Estimation—Since no repeat measurements were conducted in any laboratory, an estimate of the repeatability has been made as follows. The volume swell was plotted versus time. All plots had a good linear character. A simple regression analysis was conducted for each material. The correlation coefficients were all high (0.98 or better) and from the regression the standard deviation of the estimate was evaluated with 2 DF. This standard deviation of the estimate was equated with the average or mean standard deviation in measuring volume swell over the period 1 to 4 weeks.

16.3.1 Once values for repeatability standard deviation (of estimate) were obtained for each material, they were used to calculate duplicate values for each immersion period and from this and the single percent swell values, as reported by each laboratory, the basic data tabulation tables that are required for calculating r and R were generated. The data in these tables were then used to calculate r, (r), R, and (R) for each material by the new revised precision evaluation standard. This new standard is a revised version of Practice D4483 and was published in 2002.

16.3.2 Since (*I*) an average repeatability has been used, and (2) an insufficient number of laboratories participated in this ITP to give really definitive values, the final precision as given in Table 2 are average values over the immersion periods of 1, 2, 3 and 4 weeks. The relative repeatability precision (*r*) varies over the range of 6 to 54 % and the relative reproducibility precision (*R*) has a range of 23 to 199 %. Compounds A and D have very poor reproducibility.

16.4 Repeatability—The approximate repeatability, or local domain precision, of this test method has been established by the values found in Table 2 for each of the materials as listed in the table. Two single test results (obtained by the proper use of this standard) that differ by more than the tabulated values for r, in measurement units, and (r), in percent, shall be considered as suspect, that is, to have come from different populations. Such a decision suggests that some appropriate

⁵ The information in this section is from the original lab work done in 1999-2000. This data will be updated upon completion of the 2017 Precision and Bias.



investigative action be taken. See explanation for the repeatability as described in 16.3.

16.5 Reproducibility—The approximate reproducibility, or global domain precision, of this test method has been established by the values found in Table 2 for each of the materials as listed in the table. Two single test results obtained in different laboratories (by the proper use of this standard) that differ by more than the tabulated values for R, in measurement units, and (R), in percent, shall be considered as suspect, that is, to have come from different populations. Such a decision

suggests that some appropriate investigative action be taken. See comments on reproducibility in 16.1.1.

16.6 *Bias*—Bias is the difference between a test value and a reference or true value. Reference values do not exist for this test method, therefore bias cannot be determined.

17. Keywords

17.1 available chlorine; chloramine; chlorine; disinfection; plumbing; potable water

APPENDIX

(Nonmandatory Information)

X1. CHLORINE/CHLORAMINE VISUAL RATING CHART

X1.1 See Fig. X1.1.

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FIG. X1.1 Chlorine/Chloramine Visual Rating Chart



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