



Standard Test Methods for Rubber Property—Staining of Surfaces (Contact, Migration, and Diffusion)¹

This standard is issued under the fixed designation D925; 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 techniques to evaluate three types of staining that rubber may cause when in contact with, or in proximity to, another surface that may be light colored.

1.2 The test methods also describe how to qualitatively evaluate the degree of staining produced under the conditions of exposure to heat alone or heat and light.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1148 Test Method for Rubber Deterioration—Discoloration from Ultraviolet (UV) or UV/Visible Radiation and Heat Exposure of Light-Colored Surfaces

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

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

G151 Practice for Exposing Nonmetallic Materials in Accelerated Test Devices that Use Laboratory Light Sources

G154 Practice for Operating Fluorescent Ultraviolet (UV) Lamp Apparatus for Exposure of Nonmetallic Materials

¹ These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.15 on Degradation Tests.

Current edition approved May 1, 2014. Published May 2014. Originally approved in 1947. Last previous edition approved in 2006 as D925 – 06². DOI: 10.1520/D0925-14.

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.

G155 Practice for Operating Xenon Arc Light Apparatus for Exposure of Non-Metallic Materials

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *stain, contact*—discoloration of a product by another material or product in the area of direct contact.

3.1.1.1 *Discussion*—A test for direct surface discoloration of rubber is covered by Test Method **D1148**.

3.1.2 *stain, diffusion*—discoloration of a light colored veneer or coating, caused by the diffusion of staining materials from the rubber through the veneer or coating.

3.1.2.1 *Discussion*—The veneer or coating is in direct intimate contact with the underlying rubber.

3.1.3 *stain, migration*—discoloration, caused by constituents of a rubber, that occurs on any portion of the surface of any object in the proximity of or adjacent to but not in direct contact with the rubber causing the stain.

4. Summary of Test Methods

4.1 The test methods are as follows:

4.1.1 *Method A*—Contact Stain.

4.1.2 *Method B*—Migration Stain.

4.1.3 *Method C*—Diffusion Stain.

4.2 *Method A*—Specimens to be tested for *contact* staining are sandwiched between two metal panels. The metal panels are furnished with a desired light colored organic finish, which is in contact with the rubber. The assembly is exposed to elevated temperatures for a specified length of time and the specimens are then removed from the metal. The degree of staining of the surface that was in contact with the rubber is rated against a reference standard and a control panel (a metal plate without rubber) which are simultaneously tested with the specimen.

4.3 *Method B*—Specimens to be tested for *migration* staining are placed on a metal plate with a desired light colored organic finish, and exposed to either fluorescent UVA-340 lamps or filtered xenon arc radiation for a specified period of time. The degree of staining beyond the area that was in contact with the specimens is then rated against a reference standard

and a control panel (a metal plate without rubber) which are simultaneously tested with the specimen.

4.4 *Method C*—Diffusion staining is tested on specimens provided with a light colored rubber veneer or organic coating, deposited from organic solvent or water base. The specimens are exposed to either fluorescent UVA-340 lamps or filtered xenon arc radiation, with their veneer or coated side up, for a specified period of time. Any staining of the light colored veneer or organic coating is considered *diffusion* staining and rated against a reference standard and a control sample of the veneer or organic coating without the staining rubber.

4.5 Specimens used in *all three staining tests* shall include one or more reference samples of known staining characteristics. In addition, the contact and migration staining tests shall include a metal panel, without rubber samples, to establish the effect of the test conditions on the finished metal surface. The diffusion staining test shall include, as controls, a vulcanized sample of the rubber veneer by itself or a sample of the coating applied to aluminum foil.

5. Significance and Use

5.1 Rubber in contact with light colored organic finishes may stain the surfaces in contact with the rubber (contact staining) and surfaces adjacent to or beyond the rubber (migration staining), especially under conditions of heat, pressure, or sunlight. When a light colored rubber veneer or organic coating covers a staining rubber compound, the staining ingredients can diffuse through the veneer or coating and stain the surface (diffusion staining). This staining of light colored surfaces is objectionable to the consumer.

5.2 These test methods provide a means of evaluating staining characteristics of rubber compounds but may not necessarily duplicate the effects of natural exposure conditions.

5.3 Results obtained should not be treated as being equivalent to any natural exposure, unless the degree of quantitative correlation has been empirically established for the material in question.

5.4 These test methods may be used for producer-consumer acceptance, referee purposes, and research and development work.

5.5 The two types of exposures (Fluorescent UV and Xenon Arc) are not equivalent and may produce different test results.

METHOD A—CONTACT STAIN

6. Apparatus

6.1 *Circulating-Air Oven*—The oven shall meet the requirements of Specification E145, Type 11 B.

7. Materials and Manufacture

7.1 Metal panels 50 mm (2 in.) square shall be cut from sheet metal of approximately 0.9 mm (20 gauge) in thickness that has been coated with a desired organic finish. When these test methods are used to determine compliance with specifications, the panels may be supplied by the purchaser and the type of finish used may be the subject of agreement

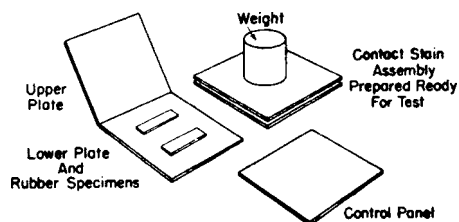


FIG. 1 Method A—Contact Stain Test Assembly

between the purchaser and the seller. The panels shall be aged for at least 48 h after finishing and before use in this test.

NOTE 1—It is recommended that acrylic enamel be employed as an organic coating for the metal panels (Delstar Acrylic Enamel—DAR 2185 white, in conjunction with Satin Primer DPE-1338, light gray-nonsanding).³

8. Test Specimen

8.1 The test specimen shall be prepared from a vulcanized production part or from a test sheet in accordance with Practices D3182 and D3183. The specimen shall be rectangular in shape, 25 by 12 mm (1 by 0.5 in.) and of uniform thickness. If a specimen of this size cannot be prepared from a production part, a modification of this size may be used, when agreed upon between the purchaser and the seller. The specimen shall be tested as received, but extraneous contamination and debris may be removed, without abrading the surface, by washing with a mild soap solution or by mechanical means.

9. Procedure

9.1 Place two rubber specimens from the same material between two metal plates, as shown in Fig. 1, with the finished surfaces of the panels in contact with the rubber.

9.1.1 Place the metal panel-rubber “sandwich” thus formed in the oven maintained at a temperature of $70 \pm 2^\circ\text{C}$ ($158 \pm 4^\circ\text{F}$). Place a 0.5 kg (1 lb) mass on top of the sandwich (or an adjusted mass, if nonstandard specimens are being tested).

9.1.2 Expose the assembly to the oven temperature for a specified time; time intervals frequently used are 6, 24, 48, and 96 h. In lieu of a specified time, 48 h shall be used as the default time interval. Take care that no other volatile or vapor producing materials that might produce staining are in the test chamber.

9.1.3 After the aging period, remove the assembly from the oven and separate the specimens cleanly from the sandwich. Then wash the panels with mild soap solution.

9.2 Simultaneously test a metal control panel without a rubber specimen to check the effect of the test conditions on the finish itself. Expose no control panel more than once.

9.3 One or more reference assemblies using rubber materials of known staining characteristics shall be included in the test.

³ The sole source of supply of the apparatus known to the committee at this time is PPG Industries, Inc., Ditzler Automotive Finishes, Troy, MI 48084. 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.

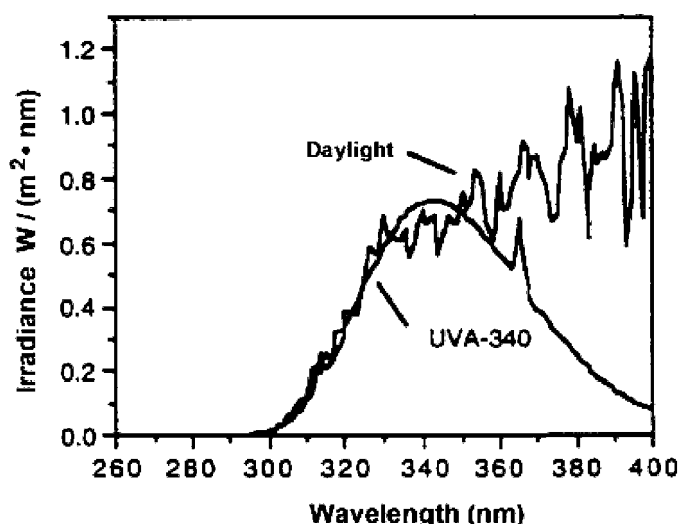


FIG. 2 Spectral Power Distributions (SPD) of UVA-340 Lamp Versus Daylight

9.4 Consider any staining of the area that was in contact with the specimen that is greater than the discoloration of the control panel as contact staining. Judge the degree of this contact stain to be greater or less than that of the reference standard, or a numerical rating based on an arbitrary scale as agreed upon between the purchaser and the seller. Observe and record any staining beyond the immediate area of contact.

METHOD B—MIGRATION STAIN

10. Apparatus

10.1 *Fluorescent UV/Condensation Apparatus (Practice G154)*—Use Fluorescent UV test apparatus that conforms to the requirements defined in Practices G151 and G154; refer to Fig. 2.

10.1.1 The instrumentation shall be calibrated in accordance with the requirements of the manufacturer.

10.2 *Xenon Arc Light Apparatus (Practice G155)*—Use xenon arc test apparatus that conforms to the requirements defined in Practices G151 and G155; refer to Fig. 3.

10.2.1 The instrumentation shall be calibrated in accordance with the requirements of the manufacturer.

11. Materials and Manufacture

11.1 Use metal panels as described in Section 7, except that the size shall be 75 by 100 mm (3 by 4 in.) for the standard specimen and shall be no larger than 300 by 300 mm (12 by 12 in.) for nonstandard specimens. Panels of this size are normally used in purchaser-seller compliance testing.

12. Test Specimens

12.1 Prepare the test specimens in accordance with Section 8.

13. Procedures

13.1 The two procedures (Fluorescent UV and Xenon Arc) use different types of exposure sources and test conditions

which may produce different test results. They shall not be used interchangeably without supporting data that demonstrates equivalency of the procedures for the materials tested.

13.2 For the allowed operational fluctuations of the specified set points for irradiance, temperature and relative humidity, refer to Table A3.1 in Practice G151. If the actual operating conditions do not comply with the maximum allowable fluctuations in Table A3.1 after the equipment has stabilized, discontinue the test and correct the cause of the problem before continuing.

13.3 Specimens should be confined to an exposure area in which the irradiance is at least 90 % of the irradiance at the center of the exposure area. Unless it is known that irradiance uniformity meets this requirement, use one of the procedures described in Practice G151, Section 5.1.4 to ensure equal radiant exposure on all specimens or to compensate for differences within the exposure chamber. If the specimens do not completely fill the racks, fill the empty spaces with blank metal panels to maintain the test conditions within the chamber.

13.4 The apparatus shall be operated continuously. However, if the test needs to be interrupted to perform routine maintenance or inspection, it should be during a dry period.

13.5 *Procedure for Exposure in Fluorescent UV/Condensation Apparatus (Practice G154)*—Unless otherwise specified, operate the fluorescent UV test apparatus with UVA-340 lamps in accordance with Practice G154.

13.5.1 Use the following exposure cycle: Set the irradiance level to 0.77 W/(m²·nm) at 340 nm. Expose specimens to a continuous cycle of 8 h light at 60°C uninsulated black panel temperature followed by 4 h of condensation at 50°C uninsulated black panel temperature.

13.5.2 Periodically reposition specimens in conformance to Practices G151 and G154; refer to 13.3.

13.5.3 Simultaneously test a control panel without rubber to check the effect of the test conditions on the finish itself. Expose no panel more than once.

13.5.4 Also include in the test one or more specimens using rubber materials of known staining characteristics.

13.5.5 Consider any staining beyond the area that was in contact with the specimen that is greater than the discoloration of the control panel as migration staining. Judge the degree of this stain to be greater or less than that of a standard or give a numerical rating based on an arbitrary scale of degree of stain, which may be agreed upon between the purchaser and the seller.

13.6 *Procedure for Exposure in Xenon Arc Apparatus (Practice G155)*—Unless otherwise specified, use the following operating conditions:

13.6.1 The xenon arc test apparatus shall be used with a Daylight Filter and conform to the spectral power distribution specifications in Practice G155.

13.6.2 Set the irradiance level at 0.55 W/(m²·nm) at 340 nm. For equivalent broad band irradiance levels at 300 to 400 nm and 300 to 800 nm, consult the manufacturer of the apparatus.

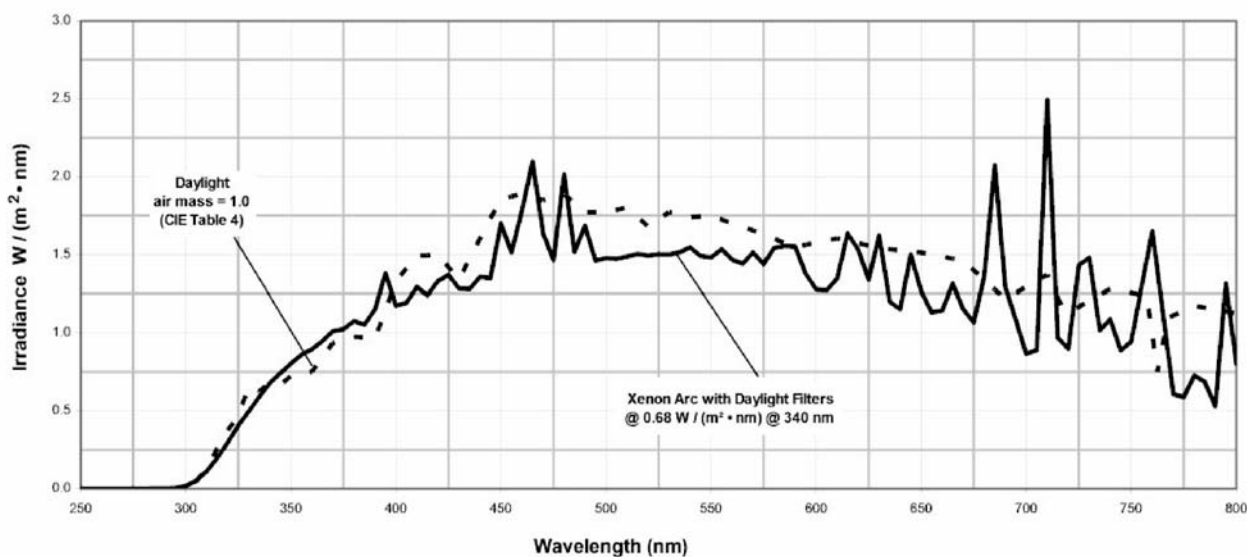


FIG. 3 Spectral Power Distributions of Xenon Arc with Daylight Filter Versus Daylight

13.6.3 The default exposure cycle shall be 102 min light only followed by 18 min light plus either water spray on the front surface or immersion in water (see Note 2 and Note 3).

NOTE 2—In the immersion technique, the test specimens are placed in a chamber that is periodically flooded with either recirculated or running water, which completely covers the specimens. The maximum temperature attained by a black colored specimen is determined with the black standard thermometer (BST) held under water on the same plane and distance from the surface as the test specimens. The immersion system is made from corrosion resistant materials that do not contaminate the water.

NOTE 3—Water spray and immersion in water frequently produce different results. Spray water can be fresh or re-circulated from a holding tank. The temperature of the spray water is uncontrolled and for fresh water is typically $21 \pm 5^\circ\text{C}$. Re-circulated spray water can be at a higher temperature. Immersion water is generally in a holding tank for recirculation. The uncontrolled temperature of the re-circulated immersion water during operation of the weathering device is typically $40 \pm 5^\circ\text{C}$.

13.6.4 Set the uninsulated Black Panel Temperature (BPT) at 63°C during the dry period of exposure to light. Consult the manufacturer of the apparatus for the equivalent insulated black panel temperature (black standard temperature or BST).

13.6.5 Relative humidity shall be set at 60 % during the dry period of exposure to light in xenon arc apparatus that provides for control of relative humidity.

13.6.6 The chamber air temperature shall be set at 44°C in apparatus that provides for adjustment of the chamber air temperature.

13.6.7 Simultaneously test a control panel without rubber to check the effect of the test conditions on the finish. Expose no panel more than once.

13.6.8 Also include in the test one or more reference specimens using rubber materials of known staining characteristics.

13.6.9 Consider any staining beyond the area that was in contact with the specimen that is greater than the discoloration of the control panel as migration staining. Judge the degree of this stain to be greater or less than that of a standard or give a

numerical rating based on an arbitrary scale of degree of stain, which may be agreed upon between the purchaser and the seller.

METHOD C—DIFFUSION STAIN

14. Apparatus

14.1 *Light Source*, as described in 10.1 or 10.2.

14.2 *Test Chamber*, as described in 10.1 or 10.2.

15. Veneer Materials

15.1 *Veneer White Compound*, calendered to a thickness of 0.5 ± 0.05 mm (0.020 ± 0.002 in.). The following is a suggested formulation:

Natural rubber	100.00
Zinc oxide	70.00
Titanium dioxide	20.00
Stearic acid	1.25
Zinc 2-mercaptobenzothiazole	0.38
Di-ortho-tolylguanidine	0.19
Sulfur	3.00
	194.82

15.1.1 Only ASTM standard reference materials (IRMs) should be used.

15.2 *Veneer White Coating* (organic solvent or water based).

15.3 *Aluminum Foil* (preferred), 0.10 mm (0.004 in.), although Holland cloth, No. 1 Grade, may be used.

16. Test Specimen

16.1 The test specimen can be prepared from an unvulcanized compound, that is, a sheet prepared in accordance with Practices D3182 and D3183 or as agreed upon between purchaser and seller.

17. Sample Preparation

17.1 Veneer Method:

17.1.1 All mixing and processing must be done on a well-cleaned mill and a clean work area. Clean the mill with a suitable solvent and follow with a piece of natural rubber allowed to band on the mill. Cut the rubber each way several times or until the mill is completely clean.

17.1.2 After milling the experimental or test compound, obtain from the mill a sheet having a thickness of 2.16 mm (0.085 in.).

17.1.3 Place the compound on aluminum foil or Holland cloth and cut or die out a sample to the curing mold dimensions.

17.1.4 Carefully cover the exposed side of the cut sample with a piece of veneer white stock calendered to a thickness of 0.5 ± 0.05 mm (0.020 ± 0.002 in.). Cover the white veneer with aluminum foil or Holland cloth. Laminate together with a roller and condition the assembly at room temperature for 1 h; then cure with the white veneer, face down in a standard mold in accordance with Practices D3182 and D3183 at the desired temperature.

17.1.5 Cut a rectangular specimen from each cured sheet to a size of 38 ± 1.5 mm (1.5 ± 0.06 in.) width by 75 ± 3 mm (3 ± 0.12 in.) length. Remove 38 mm (1.5 in.) of aluminum foil or Holland cloth from the length of the cut specimen on the veneer side.

17.1.6 A control sample of the same veneer white compound without test rubber shall be prepared, cured, and tested with each group of samples.

17.2 Coating Method:

17.2.1 For tests on a vulcanized sample, prepare samples as in 17.1 except omit the use of white veneer and follow the steps given below for unvulcanized samples.

17.2.2 For tests on unvulcanized samples, obtain from the mill a sheet of the compound having a thickness of 2.0 ± 0.1 mm (0.078 ± 0.003 in.).

17.2.3 Place the compound between aluminum foil or Holland cloth and condition at room temperature for 1 h.

17.2.4 Cut or die out a sample 38 ± 1.5 mm (1.5 ± 0.06 in.) wide by 75 ± 3 mm (3 ± 0.12 in.) long from both vulcanized and unvulcanized specimens and dip in a nonstaining white coating to a depth of 38 mm.

17.2.5 Hang on a suitable rack to drip and dry for 5 min in an area free of ultraviolet light.

17.2.6 Dip in the coating a second time and dry until the surface is nontacky. Aluminum foil dipped in the coating shall be used as a control. Caution should be exercised to avoid contamination of the coating by repeated careless dipping.

18. Procedures

18.1 The two procedures (Fluorescent UV and Xenon Arc) are different types of exposure sources and test conditions which may produce different test results. They shall not be used interchangeably without supporting data that demonstrates equivalency of the procedures for the materials tested.

18.2 For the allowed operational fluctuations of the specified set points for irradiance, temperature and relative

humidity, refer to Table A3.1 in Practice G151. If the actual operating conditions do not comply with the maximum allowable fluctuations in Table A3.1 after the equipment has stabilized, discontinue the test and correct the cause of the problem before continuing.

18.3 Specimens shall be confined to an exposure area in which the irradiance is at least 90 % of the irradiance at the center of the exposure area. Unless it is known that irradiance uniformity meets this requirement, use one of the procedures described in Practice G151, Section 5.1.4 to ensure equal radiant exposure on all specimens or to compensate for differences within the exposure chamber. If the specimens do not completely fill the racks, fill the empty spaces with blank metal panels to maintain the test conditions within the chamber.

18.4 The apparatus shall be operated continuously. However, if the test needs to be interrupted to perform routine maintenance or inspection, it should be during a dry period.

18.5 *Procedure for Exposure in Fluorescent UV/Condensation Apparatus (Practice G154)*—Operate the fluorescent UV test apparatus with UVA-340 lamps in accordance with Practices G151 and G154.

18.5.1 Use the following exposure cycle: Set the irradiance level to $0.77 \text{ W}/(\text{m}^2 \cdot \text{nm})$ at 340 nm. Expose specimens to a continuous cycle of 8 h light at 60°C uninsulated black panel temperature followed by 4 h of condensation at 50°C uninsulated black panel temperature.

18.5.2 Periodically reposition specimens in conformance to Practice G151 and G154; refer to 18.3.

18.5.3 Simultaneously test a control panel without rubber to check the effect of the test conditions on the finish itself. Expose no panel more than once.

18.5.4 Also include in the test one or more specimens using rubber materials of known staining characteristics.

18.6 *Procedure for Exposure in Xenon Arc Apparatus (Practice G155)*—Use xenon arc test apparatus that conforms to the requirements defined in Practices G151 and G155.

18.6.1 The spectral power distribution (SPD) of the filtered xenon arc lamp shall conform to the requirements of Table 1 in Practice G155 (Xenon Arc with Daylight Filter).

18.6.2 Set the irradiance level at $0.55 \text{ W}/(\text{m}^2 \cdot \text{nm})$ at 340 nm. For equivalent broad band irradiance levels at 300 to 400 nm and 300 to 800 nm, consult the manufacturer of the apparatus.

18.6.3 The default exposure cycle shall be 102 min light only followed by 18 min light plus either water spray on the front surface or immersion in water (see Note 2 and Note 3).

18.6.4 Set the uninsulated Black Panel Temperature (BPT) at 63°C during the dry period of exposure to light. Consult the manufacturer of the apparatus for the equivalent insulated black panel temperature (black standard temperature or BST).

18.6.5 Relative humidity shall be set at 60 % during the dry period of exposure to light in xenon arc apparatus that provides for control of relative humidity.

18.6.6 The chamber air temperature shall be set at 44°C in apparatus that provides for adjustment of the chamber air temperature.

18.6.7 Expose the specimens to the light for a specified length of time.

18.6.8 Include one or more standard specimens using rubber stocks of known staining characteristics.

18.7 Consider any staining of the exposed veneer or white coating that is greater than the control compound without the test rubber as diffusion staining. Judge the degree of staining to be greater or less than that of a standard or by an arbitrary rating or scale agreed upon between the purchaser and the seller.

19. Report

19.1 The report shall include the following information:

19.1.1 Type of test used: Method A, Method B, or Method C;

19.1.2 Type of lamp used, such as UVA-340 lamp or xenon arc, employed in Method B or Method C;

19.1.3 Irradiance level used for the test;

19.1.4 Exposure time in hours;

19.1.5 Date of test;

19.1.6 Identification of test and reference specimen(s);

19.1.7 Identification of finish on panels;

19.1.8 Size and shape of specimen(s) used, if not in accordance with the standard sizes; and

19.1.9 An estimate of the degree of stain compared to a standard or its numerical rating that may be agreed upon between the purchaser and the seller.

20. Precision and Bias

20.1 Since all methods yield essentially qualitative comparison data, a candidate test compound versus a control, precision as normally expressed for quantitative measurement test methods is not directly applicable.

21. Keywords

21.1 color; contact stain; diffusion stain; heat discoloration; migration stain; rubber articles; rubber products; rubber stain; ultraviolet discoloration

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 ASTM website (www.astm.org/COPYRIGHT/).