



# Standard Test Method for Rubber Deterioration—Heat and Ultraviolet Light Discoloration of Light-Colored Surfaces<sup>1</sup>

This standard is issued under the fixed designation D 1148; 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.

(This test method was prepared jointly by the Society of Automotive Engineers and the American Society for Testing and Materials.)

## 1. Scope

1.1 This test method describes a technique to evaluate the surface discoloration of white or light-colored vulcanized rubber, that may occur on exposure to heat and ultraviolet radiation.

1.2 This test method also describes how to qualitatively evaluate the degree of discoloration produced under such conditions.

1.3 The term “discoloration” applies to a color change of the rubber sample, as distinguished from staining (see Note 1), that refers to a color change of a metal finish in contact with or adjacent to the rubber specimen.

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

1.5 *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.*

NOTE 1—Tests for staining are covered by Test Methods D 925.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 925 Test Methods for Rubber Property—Staining of Surfaces (Contact, Migration, and Diffusion)<sup>2</sup>

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates<sup>3</sup>

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets<sup>2</sup>

D 3183 Practice for Rubber—Preparation of Pieces for Test Purposes From Products<sup>2</sup>

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries<sup>2</sup>

E 691 Practice for Conducting an Interlaboratory Study to

Determine the Precision of a Test Method<sup>4</sup>

## 3. Summary of Test Method

3.1 Specimens to be tested for discoloration are placed under a sunlamp bulb contained in a test chamber. The specimens shall include one or more reference specimens of known discoloration characteristics.

3.2 After exposing the specimens to radiation from the sunlamp for specified periods of time, the degree of discoloration is rated against the reference standard or the original specimens, or both.

## 4. Significance and Use

4.1 The surface of white or light-colored vulcanized rubber articles, or vulcanized rubber covered with an organic finish, may discolor when exposed to conditions of heat and sunlight. This change in color of light-colored rubber surfaces is objectionable to the consumer.

4.2 This test method does not duplicate natural exposure since the ultraviolet radiation of the sunlamp emits short wave components that are not present in sunlight.

4.3 Results obtained should be treated only as indicating the effect of heat and ultraviolet radiation emitted by the sunlamp and not as equivalent to the result of any natural exposure, unless the degree of quantitative correlation has been established for the material in question.

4.4 This test method may be used for producer-consumer acceptance, referee purposes, and research and development work.

## 5. Apparatus

5.1 *Light Source*—A RSM Type 275 W, 110 to 125 V sunlamp bulb<sup>5</sup> with Mogul screw base shall be used. The RSM bulb shall be calibrated in accordance with Section 7.

5.2 *Alternative Light Sources*—The RS,<sup>6</sup> RS-HUV, and S-1 sunlamp bulbs, formerly employed for this test method, are no longer commercially available, but may be used if they can be obtained. These bulbs shall also be calibrated in accordance with Section 7.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.15 on Degradation Tests.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 09.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 06.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>5</sup> The General Electric Time-A-Tan bulb (RSM Type) has been found satisfactory.

<sup>6</sup> The RS is the same as the RSM bulb, except that it has a standard instead of a Mogul screw base.

5.3 *Test Chamber*—A suitable metal or wooden housing to enclose the sunlamp and test specimens shall be used for control of temperature during the test and shall be equipped with a rotating turntable to produce uniform exposure and a circulating fan to control test temperature. The turntable shall rotate at  $0.05 \pm 0.017$  Hz ( $3 \pm 1$  rpm).

5.4 *Thermocouple*—A thermocouple shall be placed adjacent to the specimens and provided with suitable instruments for accurate temperature measurement.

5.5 *Ultraviolet Intensity Meter*.<sup>7</sup>

5.6 *Voltage Regulator*.

5.7 Color meter capable of measuring tristimulus colors for amber, blue and green with or without automatic calculation for  $L a b$  or  $L^* a^* b^*$  values. If without automatic calculations, a computer program should be available for this calculation.

## 6. Test Specimen

6.1 The test specimen shall be prepared from a vulcanized production part or from a test slab prepared in accordance with Practices D 3182 and D 3183. The specimen shall be rectangular in shape, 62 by 12 mm (2.4 by 0.5 in.). If a specimen of this size cannot be prepared from a production part, a modification of the size may be agreed upon between the purchaser and the seller.

6.2 A control specimen, of the same compound as that stated in 6.1 shall be prepared and reserved for color comparisons without being subjected to the ultraviolet exposure set forth in Section 8.

## 7. Calibration of Sunlamp

7.1 The ultraviolet meter indicates ultraviolet irradiance in watts per square metre ( $W/m^2$ ) when the factor 100 is ignored. Set the sensor unit of the instrument on the turntable of the test chamber and read the irradiance from the scale of the meter when the bulb has been in operation for at least 5 min to be in equilibrium. Then calibrate the lamp to a constant condition, as agreed upon between the purchaser and the seller as the conditions of the test. Use any of the two test methods in 7.1.1 and 7.1.2 to perform this calibration.

7.1.1 *Calibration by Voltage Regulation*—Place a voltage regulator on the powerline between the constant voltage transformer and the sunlamp. The voltage is thereby varied to give the desired ultraviolet light irradiance. In the use of this test method it is necessary to select an irradiance corresponding to an initial voltage of approximately 110 V when using a constant voltage transformer with an output of 120 V. This will give an adequate range to increase the irradiance so as to give satisfactory sunlamp life as the bulb degrades. Recommended irradiance levels are  $25 \pm 0.4$  and  $40 \pm 0.6$   $W/m^2$ . The 25 level will normally be used with the RSM, RS, and S-1 Type lamps and the 40 level with the RS-HUV bulb.

7.1.2 *Calibration by Distance Regulation*—The irradiance can also be varied by adjusting the distance from sunlamp bulb

to the turntable. The irradiance varies as the square of the distance. The actual distance is determined by the intensity meter reading. Recommended level is the same as shown in 7.1.1.

## 8. Procedure

8.1 To operate at the  $25 W/m^2$  level adjust the height of the RSM and RS lamps so that the bottom of the bulb is 250 mm (10 in.) above the surface of the test specimen and the height of the S-1 lamp to 460 mm (18 in.) above the test specimen. The height of the RS-HUV lamp should be adjusted to 330 mm (13 in.), if operated at  $40 W/m^2$ . Increase the height of this lamp proportionately to operate at the  $25 W/m^2$  level.

8.1.1 Change all distances proportionately if calibration method described in 7.1.2 is used.

8.1.2 Place the specimens only in an annular area below the bulb that is described as a circle 75 mm (3 in.) in diameter directly under the bulb and a concentric circle 300 mm (12 in.) in diameter. Do not make exposures in the area within the 75 mm (3 in.) circle. Place the test specimens in the testing area radially. The exposure time shall be a specified time interval, such as 6, 24, 48, and 96 h. Place the hot junction point of the thermocouple adjacent to the test specimens. The temperature recorded at this point should be  $50 \pm 2^\circ C$  ( $122 \pm 4^\circ F$ ).

8.2 One or more control specimens using rubber material of known discoloration characteristics shall be included.

8.3 Any change in color of a test specimen in relation to the original sample shall be considered as discoloration.

8.3.1 The degree of discoloration can be judged visually to be greater or less than the reference standard and can be given a numerical rating based on an arbitrary scale of degree of discoloration which may be agreed upon between the purchaser and the seller.

8.3.2 The change in color can be measured instrumentally using commercial color meters for specimens prepared according to Section 6. Calculate the difference ( $\Delta L$ ) between an unexposed specimen ( $L_U$ ) and the exposed specimen ( $L_E$ ):

$$\Delta L = L_U - L_E \quad (1)$$

$$\Delta L^* = L_U^* - L_E^* \quad (2)$$

NOTE 2—Some color meters calculate  $L$  and others calculate  $L^*$ , while some may calculate both. Either expression is suitable for use in this test method, but one must not mix them together.  $L_U - L_E$  and  $L_U^* - L_E^*$  not  $L_U - L_E^*$ . A complete equation can be found in Test Method D 2244. See Footnote 9 for a list of manufacturers of reflectance equipment.

## 9. Report

9.1 The report shall include the following information:

9.1.1 Type of bulb used, such as RSM, RS, S-1, or RS-HUV,

9.1.2 Ultraviolet light irradiance level used for the test,

9.1.3 Exposure time in hours,

9.1.4 Date of test,

9.1.5 Identification of test specimen(s),

9.1.6 Size and shape of specimen(s) if not in accordance with the standard shape and size, and

9.1.7 An estimate of the degree of discoloration according to a visual estimation (8.3.1) or an instrumental evaluation according to 8.3.2.

<sup>7</sup> Blak-Ray Ultraviolet Intensity meter with Model J-221 sensor has been found satisfactory. This is available from Ultra-Violet Products, Inc., 5114 Walnut Grove Ave., San Gabriel, CA 91778. This type of meter requires recalibration every 6 months to guarantee its accuracy; calibration service is available through the manufacturer listed above.

## 10. Precision and Bias <sup>8</sup>

10.1 These precision and bias statements have been prepared according to D4483-93.

10.2 These precision statements should not be used for acceptance or rejection testing without documentation that they are applicable to the particular materials being tested and that the specific testing protocols adhere to the test method as outlined in this test method.

10.3 The analysis as presented in this report gives realistic estimates of the precision for both the instrumental or  $\Delta L$  technique for evaluating the discoloration of light and colored rubber test specimens as well as the ranking procedure of this test method. The precision for ranking as derived from a special analysis as outlined in Table 1 is not given in the usual format. The rank values are not equivalent to the normal “quantitative variable” values that are required for a strict application of Practice D 4483 calculation procedures.

10.4 Additionally the two evaluation methods (instrumental versus ranking) were compared for sensitivity; that is, how well they detected and expressed realistic differences in discoloration compared to the inherent variation in assessing the level of discoloration on their respective measurement scales. The sensitivity is expressed as the ratio of the range of discoloration values to the standard deviation of the evaluation technique. See Table 1. Higher numbers denote greater sensitivity.

10.5 Although there is reasonable correlation for these two methods of evaluation, the instrumental method proved to be substantially more sensitive to evaluating discoloration. The range from lightest to darkest discoloration is very wide for the instrumental method; the  $\Delta L$  values are distributed on a logarithmic scale for the specimens of this interlaboratory test program. This is in contrast to the more reduced rank differences which do not reflect the true discoloration differences especially at high levels of discoloration. The analysis shows that it is more difficult for evaluators to agree on discoloration rankings for colored rubber specimens (blue, green) compared to white (light) colored rubber. The instrumental method has a reversed behavior; it is more variable for white rubbers compared to colored rubbers.

10.6 *Organization of the Interlaboratory Test Program (ITP)*—Three sets of rubber test specimens (five specimens per set) were used in the ITP. Set 1 (*A to E*) was compounded to have an initial white color, Set 2 (*K to O*) to have an initial blue color, and Set 3 (*F to J*) to have an initial green color.

10.6.1 Each set, with five different formulations, each containing a different material that would give a certain level of

discoloration, was prepared and tested according to Sections 7 and 8. This produced a set of specimens with a uniform gradation of increased discoloration in the *A to E*, the *F to J* and the *K to O* series. These treated and/or exposed sets, suitably protected against additional discoloration over time, were sent in turn in five different laboratories for instrumental ranking according to 8.3.1 and 8.3.2. Each laboratory evaluated the discoloration twice on the usual Day 1–Day 2 basis, as specified in the typical precision ITP format. Visual ranking was carried out in one laboratory with one set of specimens but six different analysts working independently and separately using the same lighting conditions. North light and no fluorescent illumination were mandatory requirements for visual ranking.

10.7 Table 2 gives the results of the multi-analyst visual ranking of exposed specimen sets as compared to an unexposed specimen. Three sets are tabulated here, the white, blue, and green. Each analyst arranged the exposed specimen from light to dark. Therefore numbers from 1 to 5 will be found for each set.

10.8 Four different colormeters<sup>9</sup> were used all of which were capable of determining the *L* or *L\** value along with a number of other CIE color coordinate systems. Although other equations could be used for instrumental evaluation, the task group decided to limit their studies to the  $\Delta L$  value.

10.9 Table 3 gives the precision results for  $\Delta L$  for each of the three sets.

### 10.10 Precision:

10.10.1 *Repeatability*—The difference between two single test results under the conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of this test method.

10.10.2 *Reproducibility*—The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test materials will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of this test method.

10.11 Table 1 gives the sensitivity values as discussed in 10.4.

### 10.12 Bias:

10.12.1 In test method terminology bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore cannot be determined.

## 11. Keywords

11.1 heat discoloration; rubber products; sunlamp; ultraviolet; ultraviolet light discoloration

<sup>8</sup> A complete report on the ITP as discussed in this section can be obtained from ASTM. Request RR: D11-1075.

**TABLE 1 Sensitivity of Discoloration Evaluation<sup>A</sup>**

Rank Method Set	$\Delta L$ Method	
	Within Laboratories	Between Laboratories
White	14.8	92.7
Blue	7.7	29.4
Green	8.6	175
		38.8
		26.5
		73.3

<sup>A</sup>This is based on the ratio of range of discoloration values to the pooled standard deviation of test results over that range for both methods.

<sup>9</sup> Hunter, Photovolt, Minolta, and McBeth color meters were used in the study reported in the appendix. Any color meter which meets the requirements of 5.7 may be used.

**TABLE 2 Multi-analyst Ranking of Exposed Discoloration Specimen Sets (Five White, Five Blue, Five Green)**

Analyst	White Set (A to E)					Blue Set (F to J)					Green Set (K to O)				
	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1	1	3	2	4	5	2	3.5	3.5	5	1	1	2.5	2.5	4	5
2	1	3	2	4	5	2	4	3	5	1	1	3	2	4	5
3	2	3	1	4	5	4	3	2	5	1	2	1	3	4	5
4	1	3	2	4	5	2	4	3	5	1	1	2	3	4	5
5	1	3	2	4	5	2	3	4	5	1	1	2	3	4	5
6	1	3	2	4	5	2	4	3	5	1	1	3	2	4	5
Sum	7	18	11	24	30	14	21.5	18.5	30	6	7	13.5	15.5	24	30
Average	1.17	3.00	1.83	4.00	5.00	2.33	3.58	3.08	5.00	1.00	1.17	2.25	2.58	4.00	5.00
Variation	0.167	0.000	0.167	0.000	0.000	0.667	0.242	0.442	0.000	0.000	0.167	0.575	0.242	0.000	0.000
Standard Deviation	0.408	0.000	0.408	0.000	0.000	0.816	0.492	0.665	0.000	0.000	0.408	0.758	0.492	0.000	0.000
Pooled Set Variation						0.067					0.270				
Pooled Set Standard Deviation						0.258199					0.520				
Coefficient Variation,%						8.6					17.3				
Rank Index	100	256	157	342	427	100	154	132	215	43	100	192	221	342	427
<i>VARIANCE F-RATIO TEST:</i>						Green Set versus White set					Green set versus Blue set				
Blue set versus White set						F(calc) = 0.197/0.067 = 2.93					F(calc) = 0.270/0.197 = 1.38				
F(calc) = 0.270/0.067 = 4.03															
F(crit) at 95 % confidence level = 1.96															
DF = 25, (Both, NUM, DEN)															

**TABLE 3  $\Delta L$  Instrumental Measurement of Color Change<sup>A</sup>**

PART A—WHITE SET:								
Material	Average	Sr	r	(r)	SR	R	(R)	
A	0.28	0.836	2.37	845	1.892	5.35	1912	
C	2.31	0.210	0.59	25.7	0.234	0.66	28.7	
B	2.88	0.102	0.29	10.0	0.218	0.62	21.4	
D	11.00	0.118	0.33	3.0	0.438	1.24	11.3	
E	37.20	0.154	0.44	1.2	0.806	2.28	6.1	
PART B—BLUE SET:								
Material	Average	Sr	r	(r)	SR	R	(R)	
J	0.37	0.072	0.20	55.1	0.094	0.27	71.9	
F	0.40	0.315	0.89	222.9	0.370	1.05	261.8	
G	1.30	0.152	0.43	33.1	0.190	0.54	41.4	
H	1.36	0.080	0.23	16.6	0.101	0.29	21.0	
I	6.33	0.271	0.77	12.1	0.271	0.72	11.3	
PART C—GREEN SET:								
Material	Average	Sr	r	(r)	SR	R	(R)	
K	0.30	0.068	0.19	64.8	0.253	0.72	241.1	
L	1.16	0.091	0.26	22.3	0.344	0.97	84.2	
M	1.26	0.188	0.53	42.3	0.229	0.65	51.6	
N	6.94	0.123	0.35	5.0	0.356	1.01	14.5	
O	22.52	0.129	0.37	1.6	0.312	0.88	3.9	

<sup>A</sup>Laboratories 1 and 2 Material A cell standard deviation (variance) deleted.

## APPENDIX

### (Nonmandatory Information)

#### X1. COLOR METER

X1.1 Set up the color meter<sup>9</sup> according to the directions supplied by the manufacturer for optimum performance. This will include any necessary calibrations.

X1.1.1 If the color meter requires a standard plaque observe any precautions for plaque storage, cleanliness and re-calibration.

X1.1.2 Obtain amber, blue and green tristimulus reflectance readings for each specimen to be measured, including the control specimen (which was not exposed to ultraviolet light).

X1.1.2.1 Color meters present data differently, however,  $L^* a^* b^*$  and  $L^* a^* b^*$  data can be obtained from any color meter

either as part of the instrumental calculations or, if not, computer programs can be used to generate the necessary data.

X1.2 To assess the discoloration of a sample as compared to the control, subtract the  $L_E$  or  $L_E^*$  value of the exposed specimen from the  $L_U$  or  $L_U^*$  value of the unexposed specimen. This is the difference ( $\Delta L$ ) or ( $\Delta L^*$ ) on which the discoloration evaluation is based. See 8.3.2.

X1.3 To obtain precision data found in Table 1 and Table 3, compounds of white, light blue, and light green stock were prepared according to Section 6, exposed to ultraviolet light

according to Section 8 and color evaluations were obtained according to 8.3.2.

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