Designation: D4890 – 13

Standard Test Methods for Polyurethane Raw Materials: Determination of Gardner and APHA Color of Polyols[1]

This standard is issued under the fixed designation D4890; 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 measure visually the color of clear polyester and polyether liquids. They apply only to materials whose colors have light-absorption characteristics similar to those of the standards. An alternative method is Test Method D1209 (see Note 1).

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—Test Method A of this standard is equivalent to ISO 6271-1. Test Method B of this standard is equivalent to ISO 4630-1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics
D1193 Specification for Reagent Water
D1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)
D5386 Test Method for Color of Liquids Using Tristimulus Colorimetry
D6166 Test Method for Color of Pine Chemicals and Related Products (Instrumental Determination of Gardner Color)
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E308 Practice for Computing the Colors of Objects by Using the CIE System
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1164 Practice for Obtaining Spectrometric Data for Object-Color Evaluation

2.2 *ISO Standards:*[3]

ISO 4630-1 Clear Liquids—Estimation of Colour by the Gardner Colour Scale—Part 1: Visual Method
ISO 4630-2 Clear Liquids—Estimation of Colour by the Gardner Colour Scale—Part 2: Spectroscopic Method
ISO 6271-1 Clear Liquids—Estimation of Color by the Platinum-Cobalt Scale—Part 1: Visual Method
ISO 6271-2 Clear Liquids—Estimation of Color by the Platinum-Cobalt Scale—Part 2: Spectroscopic Method

3. Terminology

3.1 For definitions of terms used in these test methods see Terminology D883.

4. Summary of Test Method

4.1 In Test Method A, the color of the material to be tested is compared to a series of color standards with defined chromicity coordinates, prepared on one of three ways. The results are reported as the color standard, which best matches the sample.

4.2 In Test Method B, the color of the material to be tested is compared to a series of platinum-cobalt color standards, designated by mg of Pt/mL of standard solution. The results are reported as the color standard, which best matches the sample (see Note 2).

NOTE 2—Color of liquids are also measured by visible spectroscopy and the results converted to any of several color scales. These results are converted to the APHA scale by appropriate manipulations, as for example in Test Method D5386 and ISO 6271-2. These results are converted to the Gardner scale by appropriate manipulations, as for example in Test Method ISO 4630-2.

5. Significance and Use

5.1 These test methods are suitable for quality control, as specification tests, and for research. Color is an important property of urethane products.

[1] These test methods are under the jurisdiction of Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.22 on Cellular Materials - Plastics and Elastomers.

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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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

TABLE 1 Gardner Reference Standard Color Solutions

Gardner Color Standard Number	Chromaticity Coordinates[A]			Potassium Chloroplatinate, g/1000 mL of 0.1 *N* HCl	Iron-Cobalt Solutions			Potassium Dichromate, g/100 mL Sulfuric Acid[B]
	Y	*x*	*y*		Ferric Chloride Solution, mL	Cobalt Chloride Solution, mL	Hydrochloric Acid, mL	
1	80	0.3177	0.3303	0.550	. . .	. . .	. . .	0.0039
2	79	0.3233	0.3352	0.865	. . .	. . .	. . .	0.0048
3	76	0.3329	0.3452	1.330	. . .	. . .	. . .	0.0071
4	75	0.3437	0.3644	2.080	. . .	. . .	. . .	0.0112
5	74	0.3558	0.3840	3.035	. . .	. . .	. . .	0.0205
6	71	0.3767	0.4061	4.225	. . .	. . .	. . .	0.0322
7	67	0.4044	0.4352	6.400	. . .	. . .	. . .	0.0384
8	64	0.4207	0.4498	7.900	. . .	. . .	. . .	0.0515
9	61	0.4343	0.4640	. . .	3.8	3.0	93.2	0.0780
10	57	0.4503	0.4760	. . .	5.1	3.6	91.3	0.164
11	45	0.4842	0.4818	. . .	7.5	5.3	87.2	0.250
12	36	0.5077	0.4638	. . .	10.8	7.6	81.6	0.380
13	30	0.5392	0.4458	. . .	16.6	10.0	73.4	0.572
14	22	0.5646	0.4270	. . .	22.2	13.3	64.5	0.763
15	16	0.5857	0.4089	. . .	29.4	17.6	53.0	1.041
16	11	0.6047	0.3921	. . .	37.8	22.8	39.4	1.280
17	6	0.6290	0.3701	. . .	51.3	25.6	23.1	2.220
18	4	0.6477	0.3521	. . .	100.0	0.0	0.0	3.00

[A] Chromaticity coordinates for CIE standard illuminant C and the CIE 1931 (2°) standard observer.
[B] The dichromate color standards have been found to be less reliable than chloroplatinate or iron-cobalt color standards. They are included in Table 1 for reference only.

6. Sampling

6.1 Polyesters and polyethers usually contain molecules covering an appreciable range of molecular weights. These have a tendency to fractionate during solidification. Unless the material is a liquid or finely ground solid it is necessary to melt (using no higher temperature than necessary) and mix the resin well before removing a sample for analysis. Many polyols are hygroscopic and care should be taken to provide minimum exposure to atmospheric moisture during the sampling.

7. Purity of Reagents

7.1 *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.[4] 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV or better of Specification D1193.

TEST METHOD A—GARDNER COLOR

8. Apparatus

8.1 *Gardner-Holdt Tubes*, of clear glass, with closed, flat, even bottoms, and having the following approximate dimensions and markings:

8.1.1 A uniform internal length of 112 mm,

8.1.2 A uniform internal diameter throughout the length of the tube of 10.75 mm, and

8.1.3 An etched line around the outside of the tube 5 mm from the open end and a second etched line around the outside of the tube 13 mm from the open end.

9. Reagents

9.1 *Cobalt Chloride Solution*—Prepare a solution containing 1 part by weight of cobalt chloride ($CoCl_2 \times 6H_2O$) to 3 parts of HCl (1 to 17).

9.2 *Ferric Chloride Solution*—Prepare a solution containing approximately 5 parts by weight of ferric chloride ($FeCl_3 \times 6H_2O$) and 1.2 parts of HCl (1 to 17). Adjust to exact color equivalence to a freshly prepared solution containing 3 g of $K_2Cr_2O_7$ in 100 mL of H_2SO_4 (sp gr 1.84).

9.3 *Hydrochloric Acid (1 to 17)*—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 17 volumes of water.

9.4 *Hydrochloric Acid (0.1 N)*—Prepare 0.1 *N* HCl.

9.5 *Potassium Chloroplatinate* ($PtCl_6$).

9.6 *Potassium Dichromate* ($K_2Cr_2O_7$).

9.7 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

10. Gardner Color Reference Standards[5]

10.1 The primary standards for color shall consist of solutions defined by their spectral transmittance in 1-cm cell with parallel sides. The chromaticity coordinates of these solutions

[4] *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[5] The sole source of supply of the glass color standards and color standard solutions known to the committee at this time is BYK-Gardner USA, 9104 Guilford Road, Columbia, MD 21046. 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 2 Gardner Color (Gardner Color Standard Number)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	S_r	S_R	r	R
Jeffol SD-441	5.2	0.2	0.4	0.7	1.3
Jeffol SA-499	9.6	0.2	1.0	0.7	2.7

[A]The average of the laboratories' calculated averages.

shall conform to those given in Table 1 when determined on a 1-cm layer of the solution in accordance with Practice E1164 and Test Methods E308 and D6166.

10.2 For comparison, permanent solutions of known color are more satisfactory. The approximate composition of solutions giving each of the 18 Gardner colors is also given in Table 1. The solutions shall be made from K_2PtCl_6 in 0.1 *N* HCl, or, in the darker colors, from stock solutions of $FeCl_3$, $CoCl_2$, and HCl (see 9.1, 9.2, and 9.3). Pre-prepared Gardner color standard solutions are commonly available for purchase.

10.3 If the solutions in 10.2 are unavailable, solutions of $K_2Cr_2O_7$ (sp gr 1.84) are used as reference standards. The approximate composition of these standards is also given in Table 1. Each solution must be freshly made for the color comparison, using gentle heat, if necessary, to effect solution.

10.4 Secondary reference standards may be obtained in the form of 18 colored glass disks, which are set into a pair of larger, plastic disks and the latter mounted to rotate in a housing for holding the sample tube and glass disk in close and fixed proximity.

11. Procedure

11.1 Fill the tube with sample, free of solid particles or air bubbles, so that the apparent upper edge of the liquid meniscus is even with the lower etched line on the tube.

11.2 Determine the color by comparison with the reference standard solutions prescribed in Table 1, by comparing the sample and the standard in Gardner-Holdt viscosity tubes as described. Make the comparison at 25 ± 5°C by placing tubes close together and looking through them against a white background or by comparison to the standards in a color disk.

12. Report

12.1 Report the color of the sample in terms of the Gardner standard number that is nearest to it in color. If the sample appears exactly halfway between two standards, report the color number of the darker standard.

13. Precision and Bias[6]

13.1 The precision of this test method is based on an interlaboratory study conducted in 2011. Six laboratories tested two different polyurethane materials. Every "test result" represents an individual determination. Each laboratory reported three replicate test results for each material. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D20-1257.

13.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

13.1.1.1 Repeatability limits are listed in Table 2.

13.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

13.1.2.1 Reproducibility limits are listed in Table 2.

13.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

13.1.4 Any judgment in accordance with statements 13.1.1 and 13.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting replicate results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit and the reproducibility limit as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

13.2 *Bias*—At the time of the study, there was no common reference material submitted with the samples for determining the bias for this test method, therefore no statement on bias is being made.

13.3 The precision statement was determined through statistical examination of 36 results, from six laboratories, on two different polyurethane materials.

TEST METHOD B—APHA COLOR

14. Apparatus

14.1 *Nessler Tubes*, matched 100-mL, tall-form.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1257. Contact ASTM Customer Service at service@astm.org.

TABLE 3 APHA Color Standards

Color Standard Number	Number 500 Standard, mL	Water, mL
1	0.2	99.8
3	0.6	99.4
5	1.0	99.0
10	2.0	98.0
15	3.0	97.0
18	3.6	96.4
20	4.0	96.0
25	5.0	95.0
30	6.0	94.0
40	8.0	92.0
50	10.0	90.0
60	12.0	88.0
70	14.0	86.0
80	16.0	84.0
90	18.0	82.0
100	20.0	80.0
120	24.0	76.0
140	28.0	72.0
160	32.0	68.0
180	36.0	64.0
200	40.0	60.0
300	60.0	40.0
400	80.0	20.0
500	100.0	0.0

15. Reagents

15.1 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

15.2 *Potassium Chloroplatinate* (K_2PtCl_6).

15.3 *Cobaltous Chloride Hexahydrate* ($CoCl_2 \cdot 6H_2O$).

16. Preparation of Color Standards

16.1 Pre-prepared platinum-cobalt standards are commonly available for purchase or can be prepared by the following. Measure 500 mL of water into a 1000-mL volumetric flask. Add 100 mL of the HCl and mix well. Weigh 1.245 g of K_2PtCl_6 to the nearest 1 mg and transfer to the flask (see Note 3). Add 1.0 g of crystallized $CoCl_2 \cdot 6H_2O$. Dilute the solution in the flask to the mark with water and mix thoroughly. The color of this standard solution is equivalent to 500 units (500 mg metallic platinum/L), that is each millilitre of standard contains 0.5 mg of metallic platinum.

NOTE 3—If potassium chloroplatinate is not available, dissolve 0.500 g of pure metallic platinum in aqua regia with the aid of heat; then remove HNO_3 by repeated evaporation with fresh portions of HCl. Dissolve this product as directed in 16.1.

16.2 Prepare the required color standards by diluting the No. 500 standard solution as shown in Table 3. If more exact color comparison is desired, prepare additional standards to supplement those given below. One color unit is equivalent to 1 mg metallic platinum/litre. Protect these standards against evaporation and contamination when not in use.

17. Procedure

17.1 Transfer 100 mL of the sample to one of two matched 100-mL tall-form Nessler tubes. Fill the second tube to the mark with the standard that seems to match the color of the sample as indicated by a preliminary estimation. Compare the colors of the sample and the standard by viewing vertically down through the tubes against a white background. Replace the liquid in the second tube with lighter or darker standards until an exact match is obtained.

18. Report

18.1 Report the color of the sample in terms of the color standard number that is nearest to it in color. If the sample appears exactly halfway between two standards, report the color number of the darker standard.

19. Precision and Bias

19.1 The precision of this test method is based on an interlaboratory study conducted in 2011. Six laboratories tested four different polyurethane materials. Every "test result" represents an individual determination. Each laboratory reported three replicate test results for each material. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D20-1257.

19.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

19.1.1.1 Repeatability limits are listed in Table 4.

19.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

19.1.2.1 Reproducibility limits are listed in Table 4.

19.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

19.1.4 Any judgment in accordance with statements 19.1.1 and 19.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting replicate results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit and the reproducibility limit as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

19.2 *Bias*—At the time of the study, there was no common reference material submitted with the samples for determining the bias for this test method, therefore no statement on bias is being made.

19.3 The precision statement was determined through statistical examination of 36 results, from six laboratories, on two different polyurethane materials.

TABLE 4 APHA Color (APHA Color Standard Number)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	S_r	S_R	r	R
Jeffol G 31-35	10.6	0.2	7.4	0.7	20.8
Jeffol S-490	80.8	0.2	11.1	0.7	31.1

[A]The average of the laboratories' calculated averages.

20. Keywords

20.1 APHA; color; Gardner; platinum-cobalt scale; polyols; polyurethane raw materials; Pt-Co scale

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4890–06) that may impact the use of this standard. (April 1, 2013)

(1) Modified Note 1 on ISO equivalency to follow guidelines in Guide D4968.
(2) Added Test Method D6166, Practices E177 and E691, and ISO 6271-2 to Referenced Documents section.
(3) Updated references ISO 4630-1 and ISO 6271-1 in the Referenced Documents section.
(4) Removed nonmandatory language in Note 2 and added references ISO 4630-2 and ISO 6271-2.
(5) Corrected formula for potassium chloroplatinate in reagents section.
(6) Removed footnote containing vendor-specific information.
(7) Added reference to Test Method D6166 in 10.1.
(8) Added that pre-prepared Gardner color standard solutions are commonly available for purchase in 10.2.
(9) Corrected nonmandatory language in 10.3.
(10) Added precision and bias data from ILS to Sections 13 and 19.
(11) Added that pre-prepared platinum-cobalt standards are commonly available for purchase to 16.1.
(12) Updated summary of changes.
(13) Updated chromaticity coordinates in Table 1.