Designation: D 4890 – 98

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

This standard is issued under the fixed designation D 4890; 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 D 1209. (See Note 1.)

1.2 *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—Although Test Method A of these test methods and ISO 4630-1997 differ in some details, data obtained using either are technically equivalent. Although Test Method B of these test methods and ISO 6271-1981 differ in some details, data obtained using either are technically equivalent.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]

D 1193 Specification for Reagent Water[3]

D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)[4]

D 5386 Test Method for Color of Liquids Using Tristimulus Colorimetry[5]

E 308 Practice for Computing the Colors of Objects by Using the CIE System[5]

E 1164 Practice for Obtaining Spectrophotometric Data for Object-Color Evaluation[5]

2.2 *ISO Standards:*

ISO 4630-1997 Binders for Paints and Varnishes—Estimation of Color of Clear Liquids by the Color Scale[6]

ISO 6271-1981 Clear Liquids—Estimation of Color by the Platinum-Cobalt Scale[6]

3. Terminology

3.1 For definitions of terms used in these test methods see Terminology D 883.

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 (Note 2).

NOTE 2—Color of liquids also can be measured by visible spectroscopy and the results converted to any of several color scales. These results can be converted to the APHA scale by appropriate manipulations, as for example in Test Method D 5386.

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.

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.

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[1] These test methods are under the jurisdiction of Committee D-20 on Plastics and are the direct responsibility of Subcommittee D20.22 on Cellular Plastics.

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 06.04.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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.[7] 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 D 1193.

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* ($K_2Cr_2O_7$).

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[8]

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 shall conform to those given in Table 1 when determined on a 1-cm layer of the solution in accordance with Practice E 1164 and Test Method E 308.

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 (9.1, 9.2, and 9.3).

10.3 Solutions of $K_2Cr_2O_7$(sp gr 1.84) may be 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

[7] *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.

[8] Glass color standards and color standard solutions are available from BYK-Gardner USA, 2435 Linden Lane, Silver Spring, MD 20910.

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]
	x	*y*		Ferric Chloride Solution, mL	Cobalt Chloride Solution, mL	Hydrochloric Acid, mL	
1	0.3190	0.3271	0.550	. . .	. . .	. . .	0.0039
2	0.3241	0.3344	0.865	. . .	. . .	. . .	0.0048
3	0.3315	0.3456	1.330	. . .	. . .	. . .	0.0071
4	0.3433	0.3632	2.080	. . .	. . .	. . .	0.0112
5	0.3578	0.3820	3.035	. . .	. . .	. . .	0.0205
6	0.3750	0.4047	4.225	. . .	. . .	. . .	0.0322
7	0.4022	0.4360	6.400	. . .	. . .	. . .	0.0384
8	0.4179	0.4535	7.900	. . .	. . .	. . .	0.0515
9	0.4338	0.4648	. . .	3.8	3.0	93.2	0.0780
10	0.4490	0.4775	. . .	5.1	3.6	91.3	0.164
11	0.4836	0.4805	. . .	7.5	5.3	87.2	0.250
12	0.5084	0.4639	. . .	10.8	7.6	81.6	0.380
13	0.5395	0.4451	. . .	16.6	10.0	73.4	0.572
14	0.5654	0.4295	. . .	22.2	13.3	64.5	0.763
15	0.5870	0.4112	. . .	29.4	17.6	53.0	1.041
16	0.6060	0.3933	. . .	37.8	22.8	39.4	1.280
17	0.6275	0.3725	. . .	51.3	25.6	23.1	2.220
18	0.6475	0.3525	. . .	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.

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

13.1 *Precision*—Attempts to develop a precision and bias statement for this test method have not been successful. For this reason, data on precision and bias cannot be given; however, the precision is expected to be equivalent to that reported in ISO 4630-1997. Because this test method does not contain a numerical precision and bias statement, it shall not be used as a referee test method in case of dispute. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

13.1.1 *Repeatability*—Based on ISO 4630-1997, it would be expected that two replicate test results, run on the same day, using the same equipment, by the same analyst, should be considered to be different only if they differ by more than one color number.

13.1.2 *Reproducibility*—Based on ISO 4630-1997, it would be expected that two test results, run on different days, using different equipment, by the different analysts, should be considered to be different only if they differ by more than two color numbers.

13.2 *Bias*—The bias of this test method has not yet been determined.

TEST METHOD B—APHA COLOR

14. Apparatus

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

15. Reagents

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

15.2 *Potassium Chloroplatinate* (K_2PtCl_6).

16. Preparation of Color Standards

16.1 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). 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 2. 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 *Precision*—Attempts to develop a precision and bias statement for this test method have not been successful. For

TABLE 2 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

this reason, data on precision and bias cannot be given; however, the precision is expected to be equivalent to that reported in ISO 6271-1981. Because this test method does not contain a numerical precision and bias statement, it shall not be used as a referee test method in case of dispute. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

19.1.1 *Repeatability*—Based on ISO 6271-1981, it would be expected that two replicate test results, run on the same day, using the same equipment, by the same analyst, should be considered to be different only if they differ by more than 5.1 % relative.

19.1.2 *Reproducibility*—Based on ISO 6271-1981, it would be expected that two test results, run on different days, using different equipment, by the different analysts, should be considered to be different only if they differ by more than 17 % relative.

19.2 *Bias*—The bias of this test method has not yet been determined.

20. Keywords

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