



Designation: D 4877 – 9804

Standard Test Method for Polyurethane Raw Materials: Determination of APHA Color in Isocyanates¹

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1. Scope*

1.1 This test method measures the color of clear liquids. It is applicable only to materials whose color-producing bodies have

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

light-absorption characteristics similar to those of the platinum cobalt color standards used.² (See Test Method D 1209 and Note 1.)

1.2 The values stated in SI units are to be regarded as the 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. For specific hazards statements see 7.1 and Note 2.*

NOTE 1—Although this test method and ISO 6271-1984⁹⁷ differ in some details, data obtained using either are technically equivalent.

2. Referenced Documents

2.1 *ASTM Standards:*³

D 883 Terminology Relating to Plastics

D 1193 Specification for Reagent Water

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

D 5386 Test Method for Color of Liquids Using Tristimulus Colorimetry⁵

2.2 *ISO Standards:*

ISO 6271-1984⁹⁷ Clear Liquids—Estimation of Color by the Platinum Cobalt Scale⁴

3. Terminology

3.1 For definitions of terms used in this test method see Terminology D 883.

4. Summary of Test Method

4.1 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 This test method can be used for research or for quality control to characterize *isocyanates* used in polyurethane products.

5.2 For toluene diisocyanate, results from this test method may relate to reactivity or performance in polyurethane systems.

6. Apparatus

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

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁵ 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.

7.3 *Cobaltous Chloride Hexahydrate* ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$).

7.4 *Concentrated Hydrochloric Acid* (sp. gr. 1.19).

7.5 *Potassium Chloroplatinate* (K_2PtCl_6).

8. Sampling

8.1 Since organic *isocyanates* react with atmospheric moisture, special precautions must be taken in sampling (**Caution:** Note 3). Usual sampling methods (for example, sampling an open drum with a thief), even when carried out rapidly, can cause contamination of the sample with insoluble urea. Therefore, the sample must be blanketed with dry air or nitrogen at all times.

NOTE 3—**Caution:** Organic *isocyanates* are toxic when they are absorbed through the skin, or when the vapors are breathed. Adequate ventilation should be provided and protective gloves and eyeglasses should be worn.

² See Standard Methods for the Examination of Water, Sewage, and Industrial Wastes, AM. Public Health Assn., 1015 15th St. NW Washington, DC 20005.

³ 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*, Vol 08.01, volume information, refer to the standard's Document Summary page on the ASTM website. *Annual Book of ASTM Standards*, Vol 11.01.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

⁵ *Annual Book of 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*, Vol 06.04, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

9. Preparation of Color Standards (See Note 4)

9.1 Add 500 mL distilled water to a 1000-mL volumetric flask. Add 100 mL HCl and mix well. Weigh to the nearest 1 mg of 1.245 g of K_2PtCl_6 and transfer it to the volumetric flask (Note 5). Add 1.0 g of crystallized $CoCl_2 \cdot 6H_2O$. Dilute the solution in the flask to the mark with distilled water and mix thoroughly. The color of this standard solution is equivalent to 500 color units (500 mg metallic platinum/L).

NOTE 4—Alternatively, use a Hellige Aquatester Model 611-A or equivalent instrument having permanent sealed color standards (Hellige Model 611-10 color comparator disc of APHA cobalt-platinum color standards).

NOTE 5—If potassium chloroplatinate is not available, dissolve 0.500 g of pure metallic platinum in aqua regia with heating; then remove the HNO_3 by repeated evaporations with fresh portions of HCl. Dissolve this product together with 1 g of crystallized $CoCl_2 \cdot 6H_2O$ as directed in 9.1.

9.2 Prepare the required color standards by diluting the No. 500 standard solution as shown in Table 1. If a more exact color comparison is desired, prepare additional standards to supplement those given (one color unit is equivalent to 1 mg metallic platinum/L). When not in use, standards should be sealed to avoid evaporation and contamination.

10. Procedure

~~10.1 Transfer 100 mL of the sample to~~

10.1 Fill one of two matched 100-mL tall-form Nessler tubes to the mark with the sample. Fill the second tube to the mark with the standard that seems to best match the color of the sample.

10.2 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. (See Note 6.)

NOTE 6—Rinse the second tube at least once with the replacement standard before filling the tube to the mark with it.

11. Report

11.1 Report the color number of the standard that is closest in color to the sample. If the sample appears exactly halfway between two standards, report the color number of the darker standard.

12. Precision and Bias

12.1 *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 6271-1981⁹⁷. 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, PO Box C700, West Conshohocken, PA 19428-2959.

12.1.1 *Repeatability*—Based on ISO 6271-1981⁹⁷, 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%~~ 5% relative.

TABLE 1 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
80	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	100.00	0.0

12.1.2 *Reproducibility*—Based on ISO 6271-1981⁹⁷, 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 175 % relative.

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

13. Keywords

13.1 APHA; color; isocyanates; platinum-cobalt scale polyurethane raw materials; Pt/Co scale

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that impact the use of this test method. This section also includes descriptions of the changes or reasons for the changes, or both.

D 4877 - 04:

(1) Paragraph 1.1—Better defined the nature of the standards used in the method.

(2) Note 1, Section 2—Updated the date of the referenced ISO standard.

(3) Paragraph 10.1—Revised to create a command statement and created paragraph 10.2 for emphasis.

(4) Paragraph 10.2—Added a reference to Note 6.

(5) Note 6— Added the note with additional procedural details.

(6) Paragraphs 12.1.1 and 12.1.2—Updated date of referenced ISO standard and repeatability data from new standard.

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