Designation: D4877 – 14

Standard Test Method for Polyurethane Raw Materials: Determination of APHA Color in Isocyanates[1]

This standard is issued under the fixed designation D4877; 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 This test method measures the color of clear liquids. It is applicable only to materials whose color-producing bodies have light-absorption characteristics similar to those of the platinum cobalt color standards used.[2] (See Test Method D1209 and Note 1.) Suitable isocyanates include toluene diisocyanate, and pure or modified monomeric methylene di(phenylisocyanate).

1.2 The values stated in SI units are to be regarded as 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—This standard is equivalent to ISO 6271-1:2004.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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

2.2 *ISO Standards:*

ISO 6271-1:2004 Clear liquids—Estimation of colour by the platinum-cobalt scale—Part 1: Visual method[4]

3. Terminology

3.1 For definitions of terms used in this test method see Terminology D883.

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—Test methods for measuring the platinum-cobalt color of liquids instrumentally are available—for example, Test Method D5386.

5. Significance and Use

5.1 This test method can be used for research or for quality control to characterize *iso*cyanates used in polyurethane products.

5.2 For toluene diisocyanate, results from this test method can 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 can 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.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.22 on Cellular Materials - Plastics and Elastomers.

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[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* volume information, refer to the standard's Document Summary page on the ASTM website.

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

[5] *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.

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

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

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, take special precautions in sampling. Usual sampling methods, even when conducted rapidly, can cause contamination of the sample with insoluble urea. Therefore, blanket the sample with dry air or nitrogen at all times. (**Warning—**Many diisocyanates are known or suspected sensitizers. Over-exposure to diisocyanates can lead to adverse health effects, which may include the development of occupational asthma and other respiratory, skin, and eye effects. Engineering controls, or personal protective equipment or both, including respiratory, skin, and eye protection, are to be used when there is a potential for over-exposure to diisocyanates. The product suppliers' Safety Data Sheet (SDS) provides more detailed information about potential adverse health effects and other important safety and handling information. Always follow the specific instructions provided on the SDS.)

9. Preparation of Color Standards (See Note 3)

9.1 Add 500 mL distilled water to a 1000-mL volumetric flask. Add 100 mL HCl and mix well. Weigh 1.245 g of K_2PtCl_6 to the nearest 1 mg and transfer it to the volumetric flask. Add 1.00 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) and must fall within the limits specified in Test Method D1209. Alternatively, it is acceptable to use commercially available 500 platinum-cobalt stock solutions that meet the specifications of Test Method D1209.

NOTE 3—Color comparators having permanent sealed platinum-cobalt color standards are permissible provided it is first determined that the accuracy and precision of the measurements are not reduced.

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 are to be sealed to avoid evaporation and contamination.

10. Procedure

10.1 Fill one of a matched set of 100-mL tall-form Nessler tubes to the mark with the sample. Fill a second tube from the matched set 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. Fill additional tubes from the matched set with lighter or darker standards until an exact match is obtained. (See Note 4.)

NOTE 4—When properly sealed and stored, standards can be kept in the Nessler tubes between use.

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

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 6271-1:2004. 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. Contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, if you are interested in participating in the development of precision and bias data for this standard.

12.1.1 *Repeatability*—Based on ISO 6271-1:2004, it would be expected that two replicate test results, each the mean of duplicates, run on the same day, using the same equipment, by the same analyst, are to be considered to be different only if they differ by more than 5 % relative.

12.1.2 *Reproducibility*—Based on ISO 6271-1:2004, it would be expected that two test results, run on different days, each the mean of duplicates, using different equipment, by the different analysts, are to be considered to be different only if they differ by more than 15 % 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

Committee D20 has identified the location of selected changes to this standard since the last issue (D4877 - 10) that may impact the use of this standard. August 1, 2014)

(1) Added examples of suitable isocyanates in 1.1.
(2) Editorial changes to be consistent with ISO spelling and format.
(3) Editorial changes made for clarity in Note 2.
(4) Added an additional decimal place for the weight of $CoCl_2.6H_2O$ in 9.1. Added the requirement that the 500 platinum-cobalt stock solution must conform to Test Method D1209. Added that commercially available 500 platinum-cobalt stock solutions are acceptable
(5) Removed vendor specific language and added requirements for the use of a color comparator in Note 3
(6) Modified the procedure in Section 10 and Note 4to allow the use of matched sets of Nessler tubes versus only matched pairs and to allow standards to be kept in Nessler tubes between use