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

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*

1.1This 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.)

- 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 Warning in 8.1.

Note—Although this test method and ISO 6271-1997 differ in some details, data obtained using either are technically equivalent. 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 ISO 6271-1997 Clear 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 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—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 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.

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.



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.
 - 7.3 Cobaltous Chloride Hexahydrate (CoCl₂ · 6H₂O).
 - 7.4 Concentrated Hydrochloric Acid (sp. gr. 1.19).
 - 7.5 Potassium Chloroplatinate (K₂PtCl₆).

8. Sampling

8.1 Since organic isocyanates react with atmospheric moisture, special precautions must be taken in sampling (see Warning below). 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. 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—Organic isocyanates are toxic when they are absorbed through the skin, or when the vapors are breathed. Adequate ventilation must be provided and protective gloves and eyeglasses are to be worn. 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' Material Data Safety Sheet (MSDS) provides more detailed information about potential adverse health effects and other important safety and handling information. Always follow the specific instructions provided on the MSDS.)

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 to the nearest 1 mg of 1.245 g of K_2PtCl_6 and transfer it to the volumetric flask (Note 4). 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 3—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 4—If potassium chloroplatinate is not available, dissolve 0.500 g of pure metallic platinum in aqua regia with heating; then remove the HNO₃ by repeated evaporations with fresh portions of HCl. Dissolve this product together with 1 g of crystallized CoCl₂ · 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 are to be sealed to avoid evaporation and contamination.

10. Procedure

- 10.1Fill 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 5.)

Note 5—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

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.