Designation: D7133 – 21

Standard Test Method for Polyurethane Raw Materials: Instrumental Measurement of Tristimulus CIELAB Color and Yellowness Index of Liquids[1]

This standard is issued under the fixed designation D7133; 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 provides an instrumental method for measuring the CIELAB color and Yellowness Index (YI) of liquid polyurethane raw materials. The CIELAB and YI results are derived from mathematical manipulation of CIE tristimulus values in accordance with Practices E308 and E313, respectively.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions provided for information only and are not considered 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no known ISO equivalent to this standard.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics
D1193 Specification for Reagent Water
E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]
E284 Terminology of Appearance
E308 Practice for Computing the Colors of Objects by Using the CIE System
E313 Practice for Calculating Yellowness and Whiteness Indices from Instrumentally Measured Color Coordinates
E456 Terminology Relating to Quality and Statistics
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E2935 Practice for Evaluating Equivalence of Two Testing Processes

3. Terminology

3.1 *Definitions*—For definitions of terms that appear in this test method, refer to Terminologies E284, D883, and the terminology section of Practice E308. For terms relating to precision and bias and associated issues, the terms used in this standard are defined in accordance with Terminology E456.

4. Summary of Test Method

4.1 The color of the total transmitted light is measured by a spectrophotometer in CIE tristimulus values under CIE standard illuminant D65 and CIE 1964 supplementary standard observer commonly called the 10° standard observer. These values are then converted by the appropriate equations to the CIELAB color scale and the Yellowness Index. L*a*b* and YI values are reported.

5. Significance and Use

5.1 CIELAB is a visual-based scale that is used to specify color and set color tolerances for the polyurethane industry.

5.2 Yellowness Index specifies the degree of departure of the sample from colorless towards yellow. This index is only suitable for clear liquids with degrees of saturation in yellow (dominant transmission wavelength in the 570 to 580 nm range). It is used to set tolerances for appropriate polyurethane raw materials.

[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] 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] The last approved version of this historical standard is referenced on www.astm.org.

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

5.3 This test method does not include provisions for materials with fluorescence or visible haze (usually greater than 5 % haze).

5.4 Before proceeding with this test method, make reference to the specification of the material being tested. Any test specimen preparation, conditioning, dimensions, or testing parameters or combination thereof, covered in the materials specification shall take precedence over those mentioned in this test method. If there are no material specifications, then the default conditions apply.

6. Interferences

6.1 This test method is to be used to compare samples only when they are measured under the same conditions.

6.1.1 The medium in the cuvette used during standardization of the instrument will have an effect on the measured results. Light mineral oil is recommended, however, distilled water is a suitable substitute but a note of the substitution must be included in any report of the results.

6.1.2 The temperature of the sample is also expected to affect the results obtained.

7. Apparatus

7.1 *Instrument*—A hemispherical geometry (integrating sphere) spectrophotometer capable of total transmission CIE tristimulus measurement through a cuvette that includes both the regularly transmitted portion and the diffused portion of the incident light. The instrument must be capable of converting CIE XYZ tristimulus values to the CIELAB color scale as defined in Practice E308 using CIE D65 standard illuminant and 10° standard observer. The instrument must also be capable of converting CIE XYZ tristimulus values to the Yellowness Index value defined in Practice E313 using CIE D65 standard illuminant and 10° standard observer. The instrument is to meet the manufacturer's requirements for calibration. For highly transparent samples, such as the polyols in Tables 1 and 2, spectrophotometers or tristimulus colorimeters without a spherical geometry are acceptable.

7.2 *Sample Cuvettes*—The cuvette must have a 20 ± 0.06 mm pathlength. The entrance and exit windows shall be parallel, colorless, clear and unaffected by the material being analyzed. The optical properties of the cuvette used during standardization of the instrument and the cuvette used for measuring samples (if not the same cuvette) must be matched. This is determined by proving that the variation, if any, in the different cuvettes used do not affect the measured value of a standard sample. Glass or plastic cuvettes are both acceptable.

8. Reagents

8.1 *Mineral Oil*—Colorless NF or FCC grade light mineral oil.

8.2 *Distilled Water*—Colorless distilled water conforming to Type IV of Specification D1193.

9. Hazards

9.1 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—** Diisocyanates are eye, skin and respiratory irritants at concentrations above the occupational exposure limit (TLV or PEL). Diisocyanates can cause skin and respiratory sensitization (asthma) in some people. Once sensitized, it is essential to limit further exposure to diisocyanates. Use a combination of engineering controls and personal protective equipment, including respiratory, skin and eye protection, to prevent overexposure to diisocyanates. Consult the product suppliers' Safety Data Sheet (SDS) for more detailed information about potential health effects and other specific safety and handling instructions for the product.)

10. Sampling, Test Specimens and Test Units

10.1 Test samples are to be homogeneous and representative of the liquid being tested.

10.2 Do not touch the entrance and exit windows of the cuvette through which incident and transmitted light will pass except to clean them.

10.3 The CIELAB values, L*, a*, and b*, have no units associated with them.

10.4 The Yellowness Index value, YI, has no unit associated with it.

11. Calibration and Standardization

11.1 Calibration procedures vary from manufacturer to manufacturer. They are important to ensure accurate data. Include the following periodic system verification steps:

11.1.1 *Wavelength Scale Verification*—One option is to use a didymium filter.

11.1.2 *Photometric Scale Verification*—This is to be done in accordance with manufacturer's instructions with a certified liquid standard. An APHA standard is suitable. Linearity is established using at least two different standard concentrations.

11.1.3 *Selection of Instrument Variables*

11.1.3.1 Select CIE illuminant D65 and 10° standard observer.

11.1.3.2 Select the total transmission mode unless instructed by the manufacturer to use a different mode for the verification procedure.

11.2 Pour mineral oil into a clean cuvette. Ensure that liquid completely covers the measurement area and that no air bubbles remain below the meniscus. Follow the manufacturer's instructions to perform the following steps before sample analyses and at least every four hours when samples are being analyzed.

11.2.1 *Full-Scale Standardization*—Use a cuvette filled with mineral oil to set the top of the neutral axis scale to 100 by simulating the case where all light is transmitted through the sample.

11.2.2 *Zero Scale Standardization*—Set the bottom of the neutral axis scale to 0 by simulating the case where all light is absorbed by the sample. Block the light beam by replacing the cuvette with an opaque object supplied by the manufacturer.

12. Conditioning

12.1 Condition liquids for measurement at 23 ± 2°C unless otherwise specified by contract or relevant material specification.

NOTE 2—The pure 4,4' isomer of methylene di(phenylisocyanate) (MDI) is a solid at 23°C. Condition it for measurement at 50 ± 2°C.

13. Procedure

13.1 *Sample Preparation*—Pour the sample into a clean cuvette. Ensure that liquid completely covers the measurement area and that no air bubbles remain below the meniscus.

13.2 *Selection of Instrument Variables*

13.2.1 Select CIE illuminant D65 and 10° standard observer.

13.2.2 Select the total transmission mode.

13.3 *Selection of Color Scale and Index*—Select the CIELAB color calculated as defined in Practice E308 and the Yellowness Index calculated as defined in Practice E313.

13.4 *Analysis*

13.4.1 The mineral oil is read as a sample to ensure that the instrument is set up and reading correctly prior to sample analysis. Therefore, measure the cuvette of mineral oil in duplicate by following the manufacturer's instructions for sample measurement in total transmission mode. The average of two readings is to meet the following tolerances: L* =100 ± 0.1; a*= 0 ± 0.1; b*= 0 ± 0.1 and YI = 0 ± 0.25.

13.4.2 Measure the cuvette of the sample in duplicate by following the manufacturer's instructions for sample measurement in total transmission mode.

13.4.3 Average the resulting values for L*a*b* and YI.

14. Calculation

14.1 The instrument software is to automatically calculate the L*a*b* values in accordance with the equations defined in Practice E308 and the YI value in accordance with the equation in Practice E313.

15. Report

15.1 The report shall include the following:

15.1.1 Date of measurement.

15.1.2 Sample description and identification.

15.1.3 Any changes from the recommended parameters or conditions (for example, temperature of sample if different from 23 ± 2°C or 50 ± 2°C for pure MDI, the use of a liquid other than mineral oil for standardization).

15.1.4 L*a* b* values and YI value to two significant figures to the right of the decimal point.

16. Precision and Bias

16.1 The precision data presented in this test method are representative of the conditions defined in the standard. However, material preparation and specific test conditions in the material specification can result in a deviation from the precision and bias requiring separate study.

16.2 Table 1 is based on a round robin conducted in 2001 in accordance with Practice E180 involving four materials tested by ten laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each test result was the average of two individual determinations (replicates). Each laboratory obtained two test results for each material.

16.3 Table 2 is based on a limited round robin conducted in 2001 involving two materials tested by four laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each test result was the average of two individual determinations (replicates). Each laboratory obtained two test results for each material. (**Warning—**The explanation of "*r*" and "*R*" (16.4 through 16.4.3) are only intended to present a meaningful way of considering the approximate precision of the test method. The data in Table 1 and Table 2 are not to be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method are to apply the principles outlined in Practice E691 to generate data specific to their material and laboratory (or between specific laboratories). The principles of 16.4 through 16.4.3 would then be valid for such data.)

16.4 *Precision*

16.4.1 *Repeatability, (r)*—It has been determined that the maximum expected difference between two test results for the same material, obtained by the same operator using the equipment on the same day in the same laboratory due solely to the method is *r*.

16.4.2 *Reproducibility, (R)*—It has been estimated that the maximum expected difference between two test results for the same material, obtained by different operators using different equipment in different laboratories due solely to the method is *R*.

16.4.3 Any judgment in accordance with 16.4.1 and 16.4.2 would have an approximate 95 % (0.95) probability of being correct.

16.5 There are no recognized standards by which to estimate the bias of this test method.

16.6 Supporting data are available from ASTM Headquarters. Request RR:D20-1242.

16.7 For information on equivalence, refer to Practice E2935.

17. Keywords

17.1 CIELAB; color; colorimetric analysis; instrumental measurement; isocyanates; L*a*b*; light; methylene di(phenylisocyanate); polyols; polyurethane raw materials; polyurethanes; spectrophotometry; transmission; tristimulus; yellowness index; YI