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## Standard Test Method for Color of Petroleum Products by the Automatic Tristimulus Method<sup>1</sup>

This standard is issued under the fixed designation D6045; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope\*

1.1 This test method covers the automatic determination of color of a wide variety of petroleum products such as undyed motor and aviation gasoline, aviation turbine fuels, naphthas, kerosine, pharmaceutical white oils, diesel fuel oils, heating oils, and lubricating oils by the automatic tristimulus method. This test method correlates to Test Method D156 and Test Method D1500 as calculated by the instrumentation.

NOTE 1—With the appropriate sample handling, this test method would apply to petroleum waxes, but they were not used in the round robin, and the precision of this test method with regard to waxes is unknown.

1.2 This test method reports results in terms of Test Method D156 or Test Method D1500.

1.3 This test method has a one-to-one correlation for the entire range of Test Method D1500 ASTM Color and for the range from 0 to +30 for Test Method D156 Saybolt color.

1.4 This test method does not apply to solid samples, petroleum products containing dye, and petroleum products having extreme fluorescence.

1.5 This test method does not apply to cloudy samples. Such samples shall be filtered so they are clear before measuring.

1.6 *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.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D156 Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)

D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

D2500 Test Method for Cloud Point of Petroleum Products

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

E284 Terminology of Appearance

E308 Practice for Computing the Colors of Objects by Using the CIE System

2.2 *Energy Institute Standard:*

IP 17 Determination of Colour—Lovibond Tintometer<sup>3</sup>

### 3. Terminology

3.1 *Definitions:*

3.1.1 *ASTM color*—the name of an empirical scale of expressing the color of a petroleum liquid darker than Saybolt color based on a scale of 0.5 (lightest) to 8.0 Dil (darkest) and determined by Test Method D1500.

3.1.2 *CIE*—the abbreviation for the French title of the International Commission on Illumination, or Commission Internationale de l'Éclairage. **E284**

3.1.3 *CIE Standard Illuminant C*—Colorimetric illuminant, representing daylight with a correlated color temperature of 6774 K, defined by the CIE in terms of a relative spectral power distribution. **E284**

3.1.4 *CIE 1931 standard observer*—ideal colorimetric observer with color matching functions  $x(\lambda)$ ,  $y(\lambda)$ ,  $z(\lambda)$  corresponding

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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<sup>2</sup> 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.

<sup>3</sup> *Methods for Analysis Testing*, available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K.

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

to a field of view subtending a 2° angle on the retina; commonly called the “2° Standard Observer.”

**E284**

3.1.5 *Saybolt color*—the name of an empirical scale for expressing of the color of a clear petroleum liquid based on a scale of –16 (darkest) to +30 (lightest) and determined by Test Method D156.

3.1.6 *tristimulus values*—The amounts of three specified stimuli required to match a color.

3.1.6.1 *Discussion*—In the CIE system, they are assigned the symbols X, Y, and Z.

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#### 4. Summary of Test Method

4.1 The sample is poured into the glass sample container, and the container is placed into the light path of the automatic instrument. A transmittance measurement is performed in order to determine the CIE tristimulus values (under CIE Standard Illuminant C and the CIE 1931 Standard Observer) of the sample in question. These are then converted instrumentally by the appropriate algorithm to Saybolt color or ASTM color values.

4.2 The color of the sample is reported in either Test Method D156 or Test Method D1500 values, as appropriate.

#### 5. Significance and Use

5.1 Determination of the color of petroleum products is used mainly for manufacturing control purposes and is an important quality characteristic because color is readily observed by the user of the product. In some cases the color may serve as an indication of the degree of refinement of the material. When the color range of a particular product is known, a variation outside the established range may indicate possible contamination with another product. However, color is not always a reliable guide to product quality and should not be used indiscriminately in product specifications.

#### 6. Apparatus

6.1 Either instrument described in 6.1.1 or 6.1.2 may be used in this test method.

6.1.1 *Spectrophotometer*—The spectrophotometer used in the measurement shall satisfy the following specifications:

6.1.1.1 *Wavelength Range*—380–780 nm.

6.1.1.2 *Effective Wavelength Width*—The effective wavelength width of the radiant flux from the slit of the spectrophotometer shall be  $10 \pm 2$  nm or  $5 \pm 1$  nm.

6.1.1.3 *Linearity*— $\pm 0.5$  % of full scale. Photometric reproducibility— $\pm 0.2$  %.

6.1.1.4 *Wavelength Accuracy*— $\pm 1$  nm.

6.1.1.5 *Geometrical Conditions*—Normal illumination and collection. The illumination light flux shall not include a ray which has an angle of five or more degrees toward the central line. The angle of the center line inclination of the illumination flux shall be  $0 \pm 2^\circ$  toward the normal line of the sample surface.

6.1.1.6 The spectrophotometer shall have the capability to compute tristimulus values (CIE XYZ) using CIE Standard Illuminant C and the CIE 1931 Standard Observer.

6.1.1.7 A spectrophotometer that gives results comparable to those of the instrument described in 6.1.1.1 through 6.1.1.6 shall be satisfactory.

6.1.2 *Tristimulus Filter Colorimeter*—Instrument designed for high precision color measurement of clear liquids. The instrument shall be capable of converting the light transmitted by a sample (under normal illumination/normal detection) into tristimulus values (CIE XYZ) using the CIE Standard Illuminant C and the CIE 1931 Standard Observer using Practice E308. A correlation between measured tristimulus values and Test Method D1500 and Test Method D156 numbers shall be used to yield an equivalent instrumental ASTM color and/or color, or Saybolt value, or both. The instrument shall be capable of automatically computing ASTM color or Saybolt values, or both—both (see Section 12).

6.1.3 *Sample Cell*—Sample cell shall be clean, clear, colorless, and unaffected by the petroleum product being measured. Cells with path lengths of 33 mm have been found suitable for measuring ASTM color, and cells with path lengths of 100 mm have been found suitable for measuring Saybolt values. The operator shall follow the instrument manufacturer’s procedures and recommendations.

#### 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 shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society<sup>4</sup> where such specifications are available. 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.

<sup>4</sup>The Minolta CT320 tristimulus transmission colorimeter has been found to be a suitable instrument. The sole source of supply of the colorimeter known to the committee at this time is Konica Minolta Photo Imaging U.S.A., Inc., Instruments Systems Division, 725 Darlington Ave., Mahwah, NJ 07430. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

<sup>4</sup>*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 *Annual 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.

7.2 *Diluent*—Kerosine (**Warning**—Combustible. Vapor harmful.) having a color of +21 saybolt color or lighter by this test method or Test Method D156, or 1.5 by method B of IP 17. This material is used for diluting dark samples to be measured for ASTM color. As an alternative, other solvents, such as white oil or solvent neutral 100 of satisfactory purity that meet the color requirements specified here in 7.2, are also acceptable.

## 8. Sampling

8.1 Samples shall be taken in accordance with Practice D4057.

## 9. Preparation of Sample

9.1 *Liquid Petroleum Products such as Lubricating Oils*—If the sample is not clear, heat it 6°C (10°F) above its cloud point (see Test Method D2500) and observe the color at that temperature. When the sample is darker than ASTM Color 8, mix 15 volumes of the sample into 85 volumes of the kerosine, and test the mixture.

## 10. Procedure

10.1 *Calibration*—Prepare the spectrophotometer or tristimulus filter colorimeter for operation following the manufacturer's instructions.

10.1.1 Periodic measurement of suitable Test Method D1500 or Test Method D156, or both, comparative samples would verify instrumental performance. Refer to the manufacturer's documentation for specific details. A method for creating suitable samples is included in Appendix X1.

10.1.2 Basic calibration procedures are given in Appendix X2.

10.2 *Measurement*—Place the cell filled with the sample in the instrument, and measure according to the manufacturer's instructions.

10.3 Record the ASTM color value or Saybolt number as appropriate.

## 11. Report

11.1 Report the following information:

11.1.1 The color of the sample as either Saybolt color to the nearest whole number or ASTM color according to the following procedure: (1) for results with decimal numerical values of x.1 to x.4, precede the value with the capital letter "L" or with a "<" and change the decimal value to x.5 (for example, for 3.1–3.4, report L3.5 ASTM color or <3.5 ASTM color); (2) for results with decimal numerical values of x.5, report as stated (for example, for 3.5, report 3.5 ASTM color); (3) for results with decimal numerical values x.6–x.9, precede the value with the capital letter "L" or with a "<" and round up to the next larger whole value (for example, for 3.6–3.9, report as L4 ASTM color or <4 ASTM color); (4) for results with decimal numerical values of x.0, report as stated (for example, for 4.0 report as 4.0 ASTM color); (5) for results greater than 8.0, report as D8 ASTM color or >8 ASTM color.

11.1.2 If the sample has been diluted in accordance with 7.2, report the color of the mixture followed by the abbreviation "Dil," for example, "L7.5 Dil ASTM color" or "<7.5 Dil ASTM color" (see Note 2).

NOTE 2—For samples that are diluted in 11.1.2, it is permissible for the laboratory to annotate the report in any suitable manner, provided that the information clearly conveys to the data recipient that the sample analyzed was diluted.

11.1.3 When the sample has been filtered, add the words, "sample filtered."

## 12. Precision and Bias

12.1 A correlation has been derived between the manual Test Method D156 Saybolt color and the manual Test Method D1500 ASTM color, and this automatic method in a cooperative program involving five laboratories and ten petroleum materials. Data were generated in 1993 and details and statistical analyses are on file at ASTM Headquarters.<sup>5</sup>

12.2 *Precision*—The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

12.2.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty):

12.2.1.1  $r = 0.14$  Saybolt color units (Test Method D156).

12.2.1.2  $r = 0.10$  ASTM color units (Test Method D1500).

12.2.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

12.2.2.1  $R = 1.24$  (correlation with Test Method D156).

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1356.

12.2.2.2 R = 0.48 (correlation with Test Method D1500).

12.3 *Bias:*

12.3.1 There is no bias in the correlation between this test method and Test Method D1500.

12.3.2 There is no bias in the correlation between this test method and Test Method D156 in the range from 0 to +30. There is a bias for the range from 0 to -16.

### **13. Keywords**

13.1 ASTM color; automatic colorimeter; color measurement; petroleum products color; Saybolt; tristimulus

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## APPENDIXES

### (Nonmandatory Information)

## X1. PREPARATION PROCEDURE OF THE COLOR STANDARD SAMPLES

### INTRODUCTION

This appendix is an explanation about preparation of color standard samples referred to in RR:D02-1356.<sup>5</sup> It is also an example of the preparation of possible standard samples that can be used for periodic checks (see 10.1.1).

#### X1.1 Scope

X1.1.1 This appendix describes the preparation procedure of the Saybolt color standard samples and ASTM color standard samples.

#### X1.2 Referenced Documents

X1.2.1 *IEC Standard:*

IEC 867 Insulating Liquids—Specifications for Unused Liquids Based on Synthetic Aromatic Hydrocarbons<sup>6</sup>

X1.2.2 *Japanese Industrial Standard:*

K 0510 High Purity Dodecane<sup>7</sup>

#### X1.3 Apparatus

X1.3.1 The apparatus shall conform to the specifications set forth in 6.1 through 6.1.3.

X1.3.1.1 *Cells*—The optical path lengths shall be 10 mm, 33 mm, and 100 mm.

X1.3.1.2 *Balance*—The balance shall have a readability of 0.1 mg or better.

X1.3.1.3 *Pipette*—The capacity shall be 2 mL.

X1.3.1.4 *Volumetric Flasks*—The capacity shall be 200 mL and 250 mL.

X1.3.1.5 *Erlenmeyer Flasks with Standard Taper Joint*—The capacity shall be 100 mL.

X1.3.1.6 *Beaker*—The capacity shall be 50 mL.

#### X1.4 Reagents

X1.4.1 *Dyes:*

X1.4.1.1 3-Methyl-1-phenyl-4-(phenyl azo)-pyrazol-5-ol (hereinafter referred to as Yellow 5GS-EX). The CAS RN (Chemical Abstracts Service Registry Number) is 4314-14-1.

X1.4.1.2 1-(phenyl azo)-2-naphthalenol (Orange EX). CAS RN 842-07-9.

X1.4.1.3 1,4 bis(butylamino)-9,10-anthracenedione (Blue SB). CAS RN 17354-14-2.

X1.4.1.4 1-[[4-[(dimethylphenyl)azo]dimethylphenyl]azo]- 2-naphthalenol (Red 5B-SP). CAS RN 1320-06-5.

X1.4.1.5 1,5 (or 1,8)-bis[(4 methyl phenyl)amino]-9,10-anthracenedione (Violet 3R). CAS RN 8005-40-1.

X1.4.1.6 1-hydroxy-4-[(4-methyl phenyl)amino]-9,10anthracenedione (Violet B-2R). CAS RN 81-48-1.

X1.4.1.7 1,4-bis[(4-butyl phenyl)amino]-5,8-dihydroxy-9, 10-anthracenedione (Green SG). CAS RN 28198-05-2.

X1.4.2 *Alkyldiphenylethane*—Capacitor alkyldiphenylethane described in specification IEC 867, which has a density of 0.9865–0.9877 g/cm<sup>3</sup> at 15°C.

X1.4.3 *Dodecane*—In accordance with JIS K0510 or those with composition as shown in X1.4.3.1, and with tristimulus values XYZ in X1.4.3.2.

X1.4.3.1 The spectral transmittance (value at 5-nm intervals) at 380–780 nm shall be measured by the spectrophotometer (wavelength width: 5 nm, cell: 100 mm). In accordance with Practice E308, calculate the tristimulus values of X, Y, and Z using the values for CIE Standard Illuminant C and the 1931 Standard Observer.

X1.4.3.2 *Composition of Dodecane (unit: volume %):*

Component	Amount
Undecane	0.2 or less

<sup>6</sup> 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 *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

<sup>5</sup> Available from American National Standards Institute (ANSI), 25 West 43rd St., 4th Floor, New York, NY 10036.

<sup>7</sup> Available from American National Standards Institute (ANSI), 25 West 43rd St., 4th Floor, New York, NY 10036.

<sup>8</sup> Available from Japanese Standards Organization (JSA), 4-1-24 Akasaka Minato-Ku, Tokyo, 107-8440, Japan.

Dodecane	99.5 or more
Tridecane	0.2 or less

#### X1.4.3.3 Tristimulus Values of Dodecane:

Stimulus Value	Specification
X	90.00 or more
Y	92.00 or more
Z	105.00 or more

### X1.5 Preparation Procedure for the Saybolt Color Standard Samples

X1.5.1 The Saybolt color standard samples are prepared through the following three steps: (1) preparation of the dye solution, (2) preparation of the Saybolt Color mixed dye solution, and (3) preparation of the Saybolt color standard sample.

#### X1.5.1.1 Preparation and Verification of the Dye Solution:

(1) Measure  $0.2500 \pm 0.0005$  g of yellow 5GS-EX into a 50-mL beaker. Dissolve the dye with 20 mL of alkyldiphenylethane, pour the solution into a 250-mL volumetric flask, add alkyldiphenylethane to the marked line, and mix well. Hereinafter this is called Yellow 5GS-EX Dye Solution.

(2) Repeat the above procedure for Orange EX dye and Blue SB dye. Hereinafter these solutions are called Orange EX Dye Solution and Blue SB Dye Solution.

(3) Check the absorption of the dye solution as follows: Place 2 mL of the Yellow 5GS-EX Dye Solution into a 200-mL volumetric flask. Add dodecane to the marked line and then mix well. In a separate flask, repeat the procedure for Orange EX and Blue SB. Using a spectrophotometer (wavelength width: 5 nm, cell: 10 mm), with dodecane as the control, measure the absorption of the three solutions prepared and compare them to the values in Table X1.1. If they do not meet the specifications, redo the procedures.

#### X1.5.1.2 Preparation of the Saybolt Color Mixed Dye Solution:

(1) Measure the three dye solutions prepared above according to the following specifications, and mix them in a 100-mL Erlenmeyer flask:

Dye Solution	Quantity (g)
Yellow 5GS-EX	$30.000 \pm 0.010$
Orange X	$10.000 \pm 0.005$
Blue SB	$1.000 \pm 0.001$

Measure  $5.000 \pm 0.001$  g of the solution prepared above and place it in a 100-mL Erlenmeyer flask. Add  $45.000 \pm 0.001$  g of dodecane and mix well. Hereinafter this is called Saybolt color mixed dye solution.

#### X1.5.1.3 Preparation and Verification of Saybolt Color Standard Samples:

(1) Measure the quantity of the Saybolt color mixed dye solution as shown in Table X1.2, and place it in a 50-mL beaker. Add 20 mL dodecane and mix. Then place them in a 250-mL volumetric flask, add dodecane to the marked line, and mix well.

(2) For each of the seven solutions above, measure the transmittance between 380–780 nm with a spectrophotometer (wavelength width: 5 nm, cell: 100-mm path length).

(3) Calculate the stimulus value Y of the XYZ color system and chromaticity coordinates x, y, and z from the spectral transmittance (the value at 5 nm intervals) by using the values for CIE Standard Illuminant C and the 1931 Standard Observer according to Practice E308. When the values of Y, x, y, and z meet the specifications in Table X1.3, the samples can be used as Saybolt color standard samples S + 30, S + 25, S + 19, S + 15, S + 12, S0 and S–15. The Saybolt color of each of these color standards is + 30, + 25, + 19, + 15, + 12, 0, and – 15, respectively. If the values of Y, x, y, and z do not meet the specifications in Table X1.3, repeat the above procedure.

X1.5.1.4 Preparation Procedure for the ASTM Color Standard Samples—ASTM Color Standard Samples are prepared through the following three steps: (1) preparation of the dye solution, (2) preparation of the ASTM color mixed dye solution, and (3) preparation of the ASTM color standard sample.

(1) Preparation of the Dye Solution—Measure  $0.2500 \pm 0.0005$  g of Yellow 5GS-EX dye and put it into a 50-mL beaker. Dissolve the dye with 20 mL of alkyldiphenylethane. Place the solution into a 250-mL volumetric flask, add alkyldiphenylethane to the marked line, and mix well. Hereinafter this is called Yellow 5GS-EX dye solution.

**TABLE X1.1 Dye Solution Absorption Range**

Dye Solution	Wavelength, (nm)	Absorption
Yellow 5GS-EX	395	0.881–0.935
Orange EX	465	0.519–0.541
Blue SB	600	0.412–0.438
	645	0.465–0.494
Red 5 B-SP	515	0.673–0.715
Violet 3R	545	0.337–0.358
Violet B-2R	585	0.332–0.353
	635	0.400–0.424
Green SG	680	0.467–0.498