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Standard Test Method for Determination of the Red Dye Concentration and Estimation of Saybolt Color of Aviation Turbine Fuels and Kerosine Using a Portable Visible Spectrophotometer¹

This standard is issued under the fixed designation D7058; 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-Scope*

1.1 This test method covers the determination of the red dye concentration of aviation turbine fuel and kerosine and the estimation of the Saybolt color of undyed and red dyed (<0.750 mg/L of Solvent Red 26 equivalent) aviation turbine fuel and kerosine. The test method is appropriate for use with aviation turbine fuel and kerosine described in Specifications D1655 and D3699. Red dye concentrations are determined at levels equivalent to 0.0260.026 mg/L to 0.7500.750 mg - mg/L/L of Solvent Red 26 in samples with Saybolt colors ranging from +30 to -16. The Saybolt color of the base fuel for samples dyed red with concentration levels equivalent to 0.0260.026 mg/L/L of Solvent Red 26 is estimated in the Saybolt Color for undyed samples is estimated in the Saybolt color range from +30 to -16.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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 safety, health, and healthenvironmental practices and determine the applicability of regulatory limitations prior to use.

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

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

D1319 Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption

D1655 Specification for Aviation Turbine Fuels /sist/226f0eeb-4864-4669-a37d-3db06ffce9f8/astm-d7058-19 D3699 Specification for Kerosine

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D6045 Test Method for Color of Petroleum Products by the Automatic Tristimulus Method

E203 Test Method for Water Using Volumetric Karl Fischer Titration

E1655 Practices for Infrared Multivariate Quantitative Analysis

E2056 Practice for Qualifying Spectrometers and Spectrophotometers for Use in Multivariate Analyses, Calibrated Using Surrogate Mixtures

3. Terminology

3.1 Definitions:

3.1.1 Saybolt color, n—the name of an empirical definition 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.

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

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¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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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.

3.1.2 *surrogate calibration*, *n*—a multivariate calibration that is developed using a calibration set which consists of mixtures with pre-specified and reproducible compositions that contain substantially fewer chemical components than the samples, which will ultimately be analyzed.

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3.1.3 surrogate method, n-standard test method that is based on a surrogate calibration.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *product dyes*—alkyl derivative of azobenzene-4-azo-2-naphthol (methyl derivatives of Color Index No. 26105) which are more soluble in diesel fuel.

3.2.2 *red dye, n*—substance that, when added to fuel, absorbs green light and imparts a red color to the product. For this test method, red dye is:

3.2.2.1 Solvent Red 26—an azobenzene-4-azo-2-naphthol dye of a specific chemical structure that is used to gauge the amount of red dye present in a given sample.

4. Summary of Test Method

4.1 A sample is introduced into the liquid specimen cell. The cell is placed into the light path of the apparatus. A beam of visible light is imaged through the sample onto a detector, and the detector response is determined. Wavelengths of the spectrum, which correlate highly with the red dye concentration and with the estimation of Saybolt color, are selected for analysis using selective bandpass filters. A multivariate mathematical analysis converts the detector response for the selected wavelengths to the red dye concentration and the estimated Saybolt color.

5. Significance and Use

5.1 In the United States, high sulfur content distillate products and diesel fuel used for off-road purposes, other than aviation turbine fuel, are required to contain red dye. A similar dye requirement exists for tax-free distillates. Contamination of aviation turbine fuel by small quantities of red dye has occurred. Such contamination presents major problems because airframe and engine manufacturers have severely limited operation on aviation turbine fuel containing red dye.

5.2 An alternate methodology for the determination of the presence of red dye in aviation turbine fuel is the observation of the color of the fuel when placed in a white bucket. The presence of the dye can be masked in aviation turbine fuels having dark Saybolt color. This test method provides an objective means of quickly measuring red dye concentration, but to avoid confusion with trace levels of other materials which will be indicated by the instrument, the method requires that instrument readings below $\frac{0.0260.026 \text{ mg} - \text{mg/L}}{L}$ be reported as No Dye Present.

5.3 The color of the base fuel is masked by the presence of the red dye. This test method provides a means of estimating the base color of aviation turbine fuel and kerosine in the presence of red dye.

6. Interferences

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6.1 The presence of colorants resulting from the refining process or crude oil or the presence of red dye other than the quantified types (alkyl derivatives of azobenzene-4-azo-2-naphthol) can interfere with the accurate determination of the red dye concentration reported as Solvent Red 26 equivalent, or the accurate estimation of the base fuel color. If there is controversy over whether the indicated dye concentration is from the alkyl derivatives of azobenzene-4-azo-2-naphthol, the procedure described in Annex A5 shall be used to confirm the presence of a red dye.

7. Apparatus

7.1 *Filter Spectrophotometer*, is equipped with specimen chamber, visible wavelength source, three $\frac{1010 \text{ nm} \pm 2 \text{ nm} - 2 \text{ nm}}{100 \text{ nm} \pm 5 \text{ nm}}$, and $\frac{650 \text{ m} \pm 5 \text{ nm}}{100 \text{ nm} \pm 5 \text{ nm}}$. The bandpass filters are used in conjunction with the visible wavelength source to produce light in the blue, green, and red regions of the electromagnetic spectrum. A detector converts the transmitted light to an electronic signal that is processed by an A-D converter and a microprocessor.^{3,4}

7.2 Sample Cell, constructed of polymethacrylate or clear optical glass having a path length of approximately $\frac{12 \text{ cm.}}{12 \text{ cm.}}$. If more than one cell is used for calibration, validation, and sample measurement, the path length of the cells must be matched to $\frac{\pm 0.005 \text{ cm.} \pm 0.005 \text{ cm.}}{12 \text{ cm.}}$.

8. Sampling

8.1 Samples shall be taken in accordance with Practice D4057 or D4177.

8.2 Precautions shall be taken to shield the samples from light prior to analysis.

³ JT100S instruments, manufactured by PAC, LP, 300 Bammel Westfield Road, Houston, TX 77090, were used in the development of this test method. This is not an endorsement or certification by ASTM International.

⁴ The sole source of supply of the apparatus known to the committee at this time is provided. If you are aware of alternative suppliers, please provide this information to ASTM headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.



9. Calibration and Standardization of the Apparatus

9.1 Calibrate the instrument according to the procedure described in Annex A2. Note 1—The instruments³ are calibrated at the factory by the vendor.

9.2 Qualify the instrument according to the procedure described in Annex A3.

Note 2—The instruments³ are qualified at the factory by the vendor.

9.2.1 If the qualification procedure is performed by the vendor, then the user shall perform a quality control check according to the procedure described in Section 10.

10. Quality Control Checks

10.1 To confirm the performance of the instrument periodically, measure the red dye concentration and the estimated Saybolt color of three control samples using the procedure outlined in Section 11. The quality control check standards shall be analyzed at least once a week or before the sample analysis if the instrument is used less frequently than weekly or if the instrument is moved to a different laboratory or field location.

11. Procedure

11.1 Prepare the spectrophotometer for operation in accordance with the manufacturer's instructions.

11.2 Equilibrate the sample to between 2020 °C and 25°C.25 °C.

11.3 Fill a clean, dry, sample cell. The external optical surfaces must be clean. If not, wipe clean, and dry with a piece of lint free paper (for example, lens paper).

11.4 Insert the sample cell into the cell chamber of the instrument.

11.5 Record the Solvent Red 26 equivalent concentration and the estimated Saybolt color.

12. Report

12.1 Report the dye concentration below 0.0260.026 mg -mg/L/L as No Dye Present.

12.2 Report the red dye concentration at or above 0.026 mg/L to the nearest 0.001 /L as Solvent Red 26 equivalent dye.

12.3 Report the color value as units of estimated Saybolt color.

13. Precision and Bias

13.1 Interlaboratory tests of the procedure were carried out using 18 samples covering the red dye concentration range equivalent from 0.0000 mg/L to $0.3740.374 \text{ mg} - \frac{\text{mg/L}}{\text{M}}$ of Solvent Red 26 equivalents and covering the range of Saybolt color from -13 to +30. Seven laboratories participated in the interlaboratory tests. The precision of this procedure, as determined by the statistical examination of the interlaboratory test results,⁵ is as follows:

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

13.1.1.1 for Solvent Red 26 equivalent dye concentrations between $\theta 0 \text{ mg/L}$ to 0.750 mg/L:

r = 0.006 mg/L

13.1.1.2 for samples in the Saybolt color range of -16 to +30:

r = 1.1 Saybolt color units

13.1.2 *Reproducibility*—The difference between two single and independent results obtained from different instruments on identical samples, would in the long run, in normal and correct operation of the test method, exceed the following value in only one case in twenty:

13.1.2.1 for Solvent Red 26 dye concentrations between $\theta 0 \text{ mg/L}$ to 0.750 mg/L:

R = 0.026 mg/L

13.1.2.2 for samples in the Saybolt color range of -16 to +30:

R = 4.6 Saybolt color units

13.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

⁵ Supporting data, results of the 1997 Interlaboratory Cooperative Test Program, have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1521. Contact ASTM Customer Service at service@astm.org.



13.3 *Relative Bias (Dye Concentration)*—Among certain samples, some bias⁵ proportional to the dye concentration was observed when the dye concentration results were compared to the expected concentrations. The observed bias does not appear to be of a systematic nature and is not known to be related to the accuracy of this test method, since the activity levels of the dye in sample preparation have not been determined, only estimated.

13.4 *Relative Bias (Saybolt Color)*—Some bias⁵ was observed when the color results were compared to the Test Method D156 results, however, this bias was observed only for samples that had high concentration of the dye (>0.180 mg - mg/L)./L). The bias for the base fuels was within the standard error of Test Method D156.

13.5 The precision statements in 13.1 were derived from the 1997 interlaboratory test program. Participants analyzed seven sets of undyed base fuels and 13 sets of dyed base fuel/color combinations in duplicate in the Saybolt color range of -16 to +30 and dye concentration from $\theta 0 \text{ mg/L}$ to $\theta .374 \theta .374 \text{ mg} \cdot \text{mg/L}$, ΛL , seven laboratories participated with the automatic apparatus and five laboratories participated with the manual Test Method D156 apparatus.⁵

14. Keywords

14.1 aviation turbine fuel; kerosine; red dye concentration; Saybolt color; visible spectrometry

ANNEXES

(Mandatory Information)

A1. PROCEDURE FOR PREPARATION OF RED DYE/SAYBOLT COLOR STANDARDS, QUALIFICATION SAMPLES, AND QUALITY CONTROL SAMPLES

A1.1 Scope

iTeh Standards

A1.1.1 This annex is a description of the preparation of dye concentration and color standard samples used for calibration and qualification. It also describes the preparation of possible standard samples that can be used for periodic checks.

A1.2 Apparatus

A1.2.1 Spectrophotometer, equipped to measure the absorbance of solutions in the spectral region from 380380 nm to 780 nm with an effective spectral slit width of $1010 \text{ nm} \pm 2 \text{ nm} 2 \text{ nm}$ or $55 \text{ nm} \pm 1 \text{ nm} \cdot 1 \text{ nm}$. Wavelength measurements shall be repeatable and known to be accurate to $0.1 \text{ nm} \cdot 0.1 \text{ nm}$. The photometric linearity is to be $\pm 0.5 \%$ of full scale and a photometric reproducibility of $\pm 0.2 \%$.

A1.2.2 Sample Cells, constructed of optical glass or quartz having a path length of $\pm 1 \text{ cm} \pm 0.001 \text{ cm} \pm 0.001 \text{ cm}$ for use with the spectrophotometer described in A1.2.1.

A1.2.3 Filter Spectrophotometer, see 7.1.

- A1.2.4 Sample Cell, see 7.2. For use with the spectrophotometer described in A1.2.3.
- A1.2.5 *Balance*, with a readability of 0.1 mg, <u>0.1 mg</u>, or better.
- A1.2.6 Pipettes, 0.5 mL, 1 mL, and 2 mL 0.5 mL, 1 mL, and 2 mL capacity, Class A.

•	<u> </u>
Wavelength (nm)	Absorption
395	0.881 to 0.935
465	0.519 to 0.541
600	0.412 to 0.438
645	0.465 to 0.494
	Wavelength (nm) 395 465 600 645

TABLE A1.1 Dye Solution Absorption Range



A1.2.7 Volumetric Flasks, 100 mL, 200 mL, and 250 mL 100 mL, 200 mL, and 250 mL capacity, Class A.

A1.2.8 Beaker, 50 mL capacity.

A1.3 Reagents

A1.3.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless other wise 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.⁶ Other grades may be used, provided it is first asserted that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A1.3.2 Dyes:

A1.3.2.1 3-Methyl-1-(phenyl azo)-pyrazol-5-ol (Yellow 5GS-EX), with CAS Registry No. 4314-14-1.

A1.3.2.2 1-(phenyl azo)-2-naphthalenol (Orange EX), with CAS Registry No. 842-07-9.

A1.3.2.3 1,4 bis (butylamino)-9,10-anthracenedione (Blue SB), with CAS Registry No. 17354-14-2.

A1.3.2.4 1-[[2,5-dimethyl-4[(2-methylphenyl)azo]phenyl] azo]-2-naphthol (Solvent Red 26), with CAS Registry no. 4477-79-6.

A1.3.3 1,1-bis(3,4-dimethylphenyl)ethane, (90 % min purity).

A1.3.4 *Dodecane*, anhydrous (99 % min purity). /standards.iteh.ai) A1.3.5 *Xylenes*, A.C.S. reagent grade.

A1.4 Preparation Procedure for the Saybolt Standard Samples

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A1.4.1 Measure $0.25000.2500 \text{ g} \pm 0.0005 \text{ g} \text{ of Yellow 5GS-EX into a } \frac{50 \text{ mL}}{50 \text{ mL}}$ beaker and dissolve the dye in 20 mL - 20 mL of 1,1-bis(3,4-dimethylphenyl)ethane. Quantitatively transfer the Yellow 5GS- EX solution to a $\frac{250 \text{ mL}}{250 \text{ mL}}$ volumetric flask, dilute to the mark with 1,1-bis(3,4-dimethylphenyl)ethane and mix well. This solution is called the Yellow 5GS-EX dye solution.

A1.4.2 Repeat the above procedure for the Orange EX and Blue SB dyes. These solutions are called Orange EX dye solution and Blue SB dye solution, respectively.

A1.4.3 Pipet <u>2 mL</u> of the Yellow 5GS-EX solution into a <u>200 mL</u> volumetric flask, dilute to the mark with dodecane and mix well. Using separate flasks repeat this procedure with the Orange EX dye solution and the Blue SB dye solution.

A1.4.4 At the wavelengths indicated in Table A1.1, measure the absorbance of these solutions using the spectrophotometer and cells described in A1.2.1 and A1.2.2, respectively. Use dodecane as the reference material. If the measured absorbances are not within the indicated ranges, adjust the solution either by adding more of the solid dye if the measured absorbances are less than the indicated absorbance range or by adding more 1,1-bis(3,4-dimethylphenyl)ethane if the measured absorbances exceed the indicated absorbance range.

A1.4.5 If the dye solutions meet the criteria of Table A1.1, then weigh $\frac{30.00030.000 \text{ g} \pm 0.010 \text{ g} \cdot 0.010 \text{ g}}{1.00010.000 \text{ g} \pm 0.005 \text{ g} \cdot 0.005 \text{ g}}$ of Orange EX dye solution and $\frac{1.0001.000 \text{ g} \pm 0.001 \text{ g}}{1.0001 \text{ g}}$ of Blue SB dye

⁶ 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.



TABLE A1.2 Indicated Dye Mixture Masses (g) for Synthesis of Calibration Standards

Samples	Saybolt Number	Amount of the Saybolt Color Mixed Dye Solution (g)	Amount of the Solvent Red 26 Solution
1	30	0.200 ± 0.001	0.000
2	25	0.472 ± 0.002	0.000
3	19	1.087 ± 0.002	0.000
4	15	1.724 ± 0.003	0.000
5	12	2.083 ± 0.004	0.000
6	0	4.545 ± 0.005	0.000
7	-15	8.772 ± 0.010	0.000
8	30	0.200 ± 0.001	0.050
9	25	0.472 ± 0.002	0.050
10	19	1.087 ± 0.002	0.050
11	15	1.724 ± 0.003	0.050
12	12	2.083 ± 0.004	0.050
13	0	4.545 ± 0.005	0.050
14	-15	8.772 ± 0.010	0.050
15	30	0.200 ± 0.001	0.100
16	25	0.472 ± 0.002	0.100
17	19	1.087 ± 0.002	0.100
18	15	1.724 ± 0.003	0.100
19	12	2.083 ± 0.004	0.100
20	0	4.545 ± 0.005	0.100
21	-15	8.772 ± 0.010	0.100
22	30	0.200 ± 0.001	0.200
23	25	0.472 ± 0.002	0.200
24	19	1.087 ± 0.002	0.200
25	15	1.724 ± 0.003	0.200
26	12	2.083 ± 0.004	0.200
27	0	4.545 ± 0.005	0.200
28	-15	8.772 ± 0.010	0.200
29	30	0.200 ± 0.001	0.500
30	25	0.472 ± 0.002	0.500
31	19	1.087 ± 0.002	0.500
32	15	1.724 ± 0.003	0.500
33	12	2.083 ± 0.004	0.500
34	0	4.545 ± 0.005	0.500
35	-15	8.772 ± 0.010	0.500

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solution into a $\frac{100 \text{ mL}}{100 \text{ mL}}$ amber glass bottle. Weigh $\frac{45.00045.000 \text{ g} \pm 0.001 \text{ g}}{0.001 \text{ g}}$ of dodecane into the same bottle and mix well. This solution is called the Saybolt color mixed dye solution.

A1.4.6 Prepare the Solvent Red 26 solution by weighing $\frac{0.07500.0750 \text{ g} \pm 0.0005 \text{ g} \cdot 0.0005 \text{ g}}{250 \text{ mL}}$ observed the mark with xylenes and mixing well. Calculate the concentration of the solution as follows:

 $C_i = (W/0.250)$ (A1.1)

where:

 C_i = concentration Solvent Red 26 solution in mg/L, and

W = weight mass of the solid Solvent Red 26 in mg.

A1.4.7 To generate the color standard samples, weigh the amount of the Saybolt color mixed dye solution and the Solvent Red 26 solution specified in Table A1.2 into a 250 mL 250 mL volumetric flask, dilute to the mark with dodecane, and mix well. Calculate the concentration of the Solvent Red 26 in the color standard samples as follows:

$$C = C_i \cdot W/(d \cdot 250) \tag{A1.2}$$

where:

- C = concentration Solvent Red 26 in the color standard sample, mg/L,
- C_i = concentration of Solvent Red 26 solution from A1.4.6,
- \dot{W} = weight mass of the Solvent Red 26 solution from A1.4.6, g, and
- $d = \text{density at 15.6}^{\circ}\text{C}$ of the xylenes mixture using Test Method D4052.
- $\underline{d} \equiv \underline{density at 15.6 \ ^{\circ}C}$ of the xylenes mixture using Test Method D4052.