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.

Designation: D6258 – 17

Standard Test Method for Determination of Solvent Red 164 Dye Concentration in Diesel Fuels[1]

This standard is issued under the fixed designation D6258; 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 covers the procedure for determining the concentration of dye Solvent Red 164 in commercially available diesel and burner fuels using visible spectroscopy.

NOTE 1—This test method is suitable for all No. 1 and No. 2 grades in Specifications D396 and D975 and for grades DMA and DMB in Specification D2069.

1.2 The concentration ranges specified for the calibration standards are established in response to the Internal Revenue Service dyeing requirements which state that tax-exempt diesel fuel satisfies the dyeing requirement only if it contains the dye Solvent Red 164 (and no other dye) at a concentration spectrally equivalent to 3.9 lb of the solid dye standard Solvent Red 26 per thousand bbl (11.1 mg/L) of diesel fuel.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

1.5 *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]

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel Oils

D2069 Specification for Marine Fuels (Withdrawn 2003)[3]

D3699 Specification for Kerosine

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

E131 Terminology Relating to Molecular Spectroscopy

E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

E288 Specification for Laboratory Glass Volumetric Flasks

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E969 Specification for Glass Volumetric (Transfer) Pipets

2.2 *Federal Regulation:*

26 CFR 48.4082-1(b) Federal Excise Tax Regulation

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology E131.

4. Summary of Test Method

4.1 The absorbance of each sample is recorded over a specified wavelength range, and the scan is analyzed using derivative analysis software to determine the dye concentration.

4.2 Derivative analysis methodology is employed to minimize interferences caused by variations in the color and composition of the fuel samples regularly tested using this test method.

4.2.1 Naturally occurring diesel test fuels range in color from water white to nearly black, and many of the samples tested using this test method have also had used oils and other products blended with them. These variations in color and composition have a significant effect upon absorbance characteristics of the samples in the region of the visible spectrum where azo dyes absorb. Standard operating procedures to correct for these background variations would involve running

[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] 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

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1-[[2,5-dimethyl-4-[(2-methylphenyl)azo]phenyl]azo]-2-naphthol

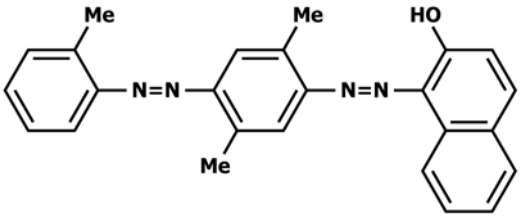

FIG. 1 Structure of Solvent Red 26

a neat (undyed) sample and subtracting out the background absorbance. In most situations involved with the application of this test method, however, neat material is not available, so no background corrections can be made.

4.2.2 The second derivative of the absorbance of these dyes in the visible region is a function of the fine structure of the dye's absorbance peak (versus its height or area) and is relatively unaffected by changes in background absorbance. Further, the specific sections (maxima and minima) of the second derivative spectra employed for this analysis are a function of the fine structure of the top of the absorbance curve which has been found to be unique to the azo dyes.

5. Significance and Use

5.1 This test method was developed to provide for the enforcement of 26 CFR 48.4082-1(b), which mandates that all tax-exempt diesel fuels be dyed with an amount of Solvent Red 164 at a concentration that is spectrally equivalent to 3.9 lb/10^3 bbl (11.1 mg/L) of Solvent Red 26. It is employed to verify that the correct amount of Solvent Red 164 is being added to tax-exempt product at terminals or refineries prior to sale, and to detect the presence of Solvent Red 164 in taxed product intended for on-road use.

5.1.1 Solvent Red 26 is the azo dye shown in Fig. 1. It is the standard against which the concentration of Solvent Red 164 is measured because it is available in a certified pure form. Solvent Red 164 is identical in structure to Solvent Red 26 except that it has hydrocarbon (alkyl) chains incorporated to increase its solubility in diesel and burner fuels. The exact composition of Solvent Red 164 will vary from manufacturer to manufacturer and lot to lot depending upon the extent of alkylation that occurs during production; however, its visible spectrum is virtually identical to the spectrum of Solvent Red 26. Solvent Red 164 is employed in the field (instead of Solvent Red 26) to dye tax-exempt diesel and burner fuels because of its higher solubility and relatively low cost.

6. Apparatus

6.1 *Spectrophotometer,* equipped with automated scanning, background correction, and electronic data storage capabilities, and the ability to automatically record absorbance or transmittance of solutions in the spectral region from 400 nanometers (nm) to 800 nm with a spectral slit width of 1.0 nm or less (Note 2). Wavelength measurements shall be repeatable and known to be accurate to within ±0.2 nm or less at deuterium peak 656.1 nm. In the absorbance range from 0.01 to 1.0, absorbance measurements shall have a photometric accuracy of ±0.005 or less and a photometric repeatability of ±0.002 or less.

NOTE 2—Instruments having different specifications, for example, minimum slit width 2 nm to 4 nm, no data storage, diode array spectrophotometers, and so forth, may be used if they provide demonstrably equivalent results. Equivalence can be demonstrated by successful (within reproducibility limits) participation in inter- or intra-laboratory studies using this test method.

6.1.1 For applicable general techniques and methods of testing spectrophotometers to be used in this test method, refer to Practices E169 and E275.

6.2 *Sample Cells (Cuvettes),* one or more fused silica or glass cells having sample path length of 1.0 cm.

6.3 *Analytical Balance,* 0.1 mg sensitivity, ±0.05 mg precision.

6.4 *Volumetric Pipettes,* 1, 2, 3, 4, and 5 mL, Class A, according to Specification E969.

6.5 *Volumetric Flasks,* 100 mL and 250 mL, Class A, borosilicate glass, according to Specification E288.

7. Reagents

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 where such specifications are available.[4] 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.

7.2 *Solvent Red 26 (Dye Standard)*—Dye, Color Index Solvent Red 26, 1-[[2,5-dimethyl-4-[(2-methylphenyl) azo]phenyl]azo]-2-naphthol, Chemical Abstract Services Registry No. 4477-79-6, dry powder with certified purity, and maximum absorbance at 512 nm ± 20 nm.

7.3 *Kerosine*—1-K, water-white, conforming to Specification D3699, and having a maximum absorbance against air of 0.08 absorbance units over the wavelength range 450 nm to 750 nm (1.0 cm cell, 120 nm/min scan rate, slit width 1.0 nm). (**Warning**—Flammable; harmful if swallowed, inhaled, or brought into contact with skin or eyes.)

7.4 *Xylene*—(**Warning**—Extremely flammable; harmful if swallowed, inhaled, or brought into contact with skin or eyes.)

8. Sampling

8.1 Use the principles of Practice D4057 in acquisition of test sample(s).

8.2 Precautions must be taken to shield the samples from sunlight prior to analysis.

[4] *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.

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