Designation: ~~D7843 – 18~~ D7843 – 21

Standard Test Method for Measurement of Lubricant Generated Insoluble Color Bodies in In-Service Turbine Oils using Membrane Patch Colorimetry[1]

This standard is issued under the fixed designation D7843; 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 extracts insoluble contaminants from a sample of in-service turbine oil onto a patch and the color of the membrane patch is analyzed by a spectrophotometer. The results are reported as a ΔE value, within the ~~CIE LAB~~ CIELAB scale.

1.2 This test method is not appropriate for turbine oils with dyes.

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, health, and environmental 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]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D4378 Practice for In-Service Monitoring of Mineral Turbine Oils for Steam, Gas, and Combined Cycle Turbines
D4898 Test Method for Insoluble Contamination of Hydraulic Fluids by Gravimetric Analysis
D5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration
D7317 Test Method for Coagulated Pentane Insolubles in Used Lubricating Oils by Paper Filtration (LMOA Method)
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E284 Terminology of Appearance
E308 Practice for Computing the Colors of Objects by Using the CIE System
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

[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.C0.01 on Turbine Oil Monitoring, Problems and Systems.

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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.

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

3.1.1 *CIELAB color scales, n*—CIE 1976 *L**, *a**, *b** opponent-color scales, in which *a** is positive in the red direction and negative in the green direction; *b** is positive in the yellow direction and negative in the blue direction; and *L** is positive in the lightness direction and negative in the darkness direction. **E308**

3.1.2 *colorimetry, n*—the science of color measurement. **E284**

3.1.3 *in-service oil, n*—lubricating oil that is present in a machine that has been at operating temperature for at least one hour.

3.1.4 *membrane color, n*—a visual rating of particulate on a filter membrane against ASTM Color Standards.

3.1.5 *membrane filter, n*—a porous article of closely controlled pore size through which a liquid is passed to separate matter in suspension.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *soot, n—in internal combustion engines*, sub-micron size particles, primarily carbon, created in the combustion chamber as products of incomplete combustion.

3.2.2 *varnish, n*—a thin, hard, lustrous, oil-insoluble deposit, composed primarily of organic residue, and most readily definable by color intensity. It is not easily removed by wiping with a clean, dry, soft, lint-free wiping material and is resistant to saturated solvents. Its color may vary, but it usually appears in gray, brown, or amber hues.

4. Summary of Test Method

4.1 Insoluble deposits are extracted from an in-service turbine oil sample using a 47 mm, 0.45 µm membrane nitro-cellulose patch. The color of the patch is then analyzed using a spectrophotometer and the results are reported as a ΔE value in the ~~CIE LAB~~ CIELAB scale.

5. Significance and Use

5.1 This test can be a guide to end-users on the formation of lubricant-generated, insoluble deposits.

5.2 The results from this test are intended to be used as a condition monitoring trending tool as part of a comprehensive program, as outlined in standards such as Practice D4378.

6. Apparatus

6.1 Variations of apparatus, particularly with respect to filter membranes and vacuum pump setting, can affect the test results significantly.

6.2 When the user of this test method uses an alternate membrane filter, it is incumbent upon them to establish that the alternate filter will give equal results.

6.3 *Required Apparatus:*

6.3.1 Membrane Filter, 47 mm nitro-cellulose, 0.45 µm.

6.3.2 Forceps, smooth-tip.

6.3.3 Borosilicate Filter Holder.

6.3.4 Borosilicate Filtering Flask.

6.3.5 Wash Bottle equipped with 0.22 µm membranes.

6.3.6 Vacuum Source, capable of maintaining a vacuum of 71 kPa ± 5 kPa.

6.3.7 Graduated cylinder, 150 mL to 200 mL.

6.3.8 Beaker, 100 mL to 250 mL.

6.3.9 Petri dish.

6.3.10 Spectrophotometer, with capabilities of analyzing a standard 15 mm target with a 0°/45° measuring geometry, 10° observer, 10 nm spectral intervals minimum resolution, the visible spectral range of 400 nm to 700 nm and CIELAB measuring indices.

7. Reagents and Materials

7.1 *Petroleum Spirit* (also known as petroleum ether or IP Petroleum Spirit 40/60) (**Warning—**Extremely flammable. Harmful if inhaled. Vapors are easily ignited by electrostatic discharges, causing flash fire.), having boiling range from 35 °C to 60 °C.

7.2 *Coleman Camp Fuel*—Coleman Fuel is a complex mixture of light hydrocarbons (primarily aliphatic) produced by distillation of petroleum. Carbon number range is C_5 to C_9, and contains less than 0.001 % benzene.

7.3 *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 of Analytical Reagents of the American Chemical Society[3] 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.

8. Sampling, Test Specimens, and Test Units

8.1 Using either Practice D4057 (manual sampling) or Practice D4177 (automatic sampling), obtain a representative sample of at least 60 mL of the material to be tested.

NOTE 1—The sample container used shall preclude exposure to UV light as oil is known to be sensitive to both indoor and outdoor sources. Fluorescent light is known to contain UV components and has been shown to increase deposit levels. This exposure can occur upon drawing of the sample as well as during incubation period. Wide-mouth laboratory quality amber high-density polyethylene (HDPE) bottles have been shown to protect the oil sample from UV light exposure. Other translucent or clear sample bottles are acceptable for use provided that the sample bottle, upon drawing of the sample and throughout the heating/incubation process, is immediately placed and stored into packaging or a storage unit that blocks light. Cylindrical cardboard packaging containers meet this intent.

8.2 The sample shall be heated to 60 °C to 65 °C for 23 h to 25 h then stored between 15 °C to 25 °C, away from UV light for an incubation period of 68 h to 76 h. Samples that are analyzed prior to this aging period may produce fewer color bodies on the patch; thus, producing a lower ΔE value, and may lower the value of trend analysis.

NOTE 2—Samples can be analyzed in longer or shorter intervals with agreement of the end-user. The level of deposit and as a consequence, the test results may be affected by time duration chosen. It is sometimes suggested to additionally analyze the sample at multiple incubation periods to fully understand the operating system.

9. Procedure

9.1 *Preparation of Sample and Materials:*

9.1.1 Document the date and time at the beginning of the test.

9.1.2 Vigorously mix the sample for 15 s minimum to resuspend insolubles uniformly. Visually inspect the inside of the bottle for evidence of material adherent to the surface of the bottle before sampling.

NOTE 3—If adherent material cannot be removed from bottle after repeated attempts to vigorously mix, include as comment in the reporting documentation.

[3] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

TABLE 1 72 h (3 day)

Material	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	s_R	r	R
Sample 1	15.332	0.861	3.169	2.410	8.874
Sample 2	7.409	0.910	2.830	2.547	7.925
Sample 3	6.970	0.839	3.085	2.349	8.637
Sample 4	3.472	0.468	2.047	1.309	5.732
Sample 5	6.993	0.852	2.968	2.386	8.309
Sample 6	34.258	3.656	11.556	10.236	32.357
Sample 7	3.475	0.176	1.647	0.494	4.611

9.1.3 Transfer 50 mL ± 1 mL of sample into clean beaker or Erlenmeyer flask.

9.1.4 Add approximately 50 mL ± 1 mL of petroleum ether to beaker containing sample.

9.1.5 Stir sample for approximately 30 s to ensure that a complete solution (and complete mixing) is attained.

9.1.6 Pour sample into filter funnel within 1 min to 2 min of initial mixing.

9.2 *Filtration Process:*

9.2.1 Using forceps, mount the filter on the center of the filter holder.

9.2.2 Mount and securely clamp the filter funnel to the filter.

9.2.3 Apply a vacuum and ensure a vacuum of less than 76 kPa is attained and held.

9.2.4 Rinse the beaker twice with a minimum of 35 mL of petroleum ether and pour the rinsing into the filter funnel.

9.2.5 Permit the filtrate to completely flow through.

9.2.6 Carefully, remove the clamp and funnel. Wash any adhering insolubles from the funnel onto the membrane with petroleum ether. Wash the membrane gently, particularly the edges, with petroleum ether from the wash bottle.

NOTE 4—If any of the deposits fails to remain on (or falls off) the dry membrane the test must be repeated.

9.2.7 Carefully release the vacuum.

9.2.8 Remove the clamp and filter holder.

9.2.9 Using forceps carefully remove the filter from the filter holder and place into a clean dry petri-dish. To facilitate handling, the membrane filters might be rested on clean glass rods in the petri-dish.

9.2.10 Dry the membrane by placing it in a low-level heat source free of ignition sources for flammable vapors, or air dry (typically, 3 h) in a dust-free location. Dryness can be estimated by comparing the white color of the outer edge of the test membrane with a new membrane.

9.3 *Color Determination of the Membrane Patch:*

9.3.1 Standardize the instrument using a patch developed from clean solvent application of the method to establish the background color white.

9.3.2 Follow the standardization procedure defined by the instrument manufacturer.

9.3.3 Analyze the patch as recommended by the instrument manufacturer.

TABLE 2 120 h (5 day)

Material	Average $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
Sample 1	17.125	1.530	4.287	4.284	12.004
Sample 2	7.618	0.715	2.919	2.003	8.174
Sample 3	7.473	0.710	3.000	1.989	8.401
Sample 4	3.584	0.519	1.872	1.452	5.242
Sample 5	7.883	0.620	3.132	1.736	8.770
Sample 6	38.615	4.889	12.300	13.690	34.439
Sample 7	3.867	0.437	1.568	1.223	4.392

9.3.4 Analyze the color of the patch in accordance with the equipment manufacturer's instructions to produce a ~~CIE LAB~~ CIELAB value.

10. Calculation of Results

10.1 *CIE 1976 L* a* b* Uniform Color Space and Color-Difference Equation*—This is an approximately uniform color space System defined by Practice E308. It is produced by plotting in rectangular coordinates the quantities L^*, a^*, b^*, calculated as follows:

$$L^* = 116^*(Y/Y_n)^{1/3} - 16 \quad (1)$$

$$a^* = 500[(X/X_n)^{1/3} - (Y/Y_n)^{1/3}] \quad (2)$$

$$b^* = 200[(Y/Y_n)^{1/3} - (Z/Z_n)^{1/3}] \quad (3)$$

$$X/X_n\,;\, Y/Y_n\,;\, Z/Z_n > 0.01 \quad (4)$$

The tristimulus values X_n,Y_n,Z_n define the color of the normally white object-color stimulus, in this case the new membrane patch. Under these conditions, X_n,Y_n, and Z_n are the tristimulus values of the standard 10° observer, D65 illuminant.

10.1.1 The total difference ΔE^*_{ab} between two colors each given in terms of L^*, a^*, b^* is calculated as follows:

$$\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (5)$$

10.2 *Reporting:*

10.2.1 Report the spectrophotometer ~~CIE LAB~~ CIELAB ΔE values to one decimal place.

10.2.2 The incubation after heating shall be reported with the ΔE value.

10.2.3 Note in the reporting documentation any deviation from this Standard's methodology.

11. Precision and Bias[4]

11.1 The precision of this test method is based on an interlaboratory study conducted in 2011. Twelve laboratories participated in the study, testing seven different turbine oils, over three and five day periods. Every analyst was instructed to report duplicate test results in this study. Practice E691 was followed for the study design; the details are given in ASTM Research Report RR:D02-1752.

11.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same point, obtained by the same operator using the same equipment on the same day in the same laboratory.

11.1.1.1 Repeatability limits are listed in Tables 1 and 2.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1752. Contact ASTM Customer Service at service@astm.org.