



Designation: D7317 – 07 (Reapproved 2013)

## Standard Test Method for Coagulated Pentane Insolubles in Used Lubricating Oils by Paper Filtration (LMOA Method)<sup>1</sup>

This standard is issued under the fixed designation D7317; 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 determination of coagulated pentane insolubles in used lubricating oils by a paper filtration method.

1.2 This test method was originally developed by the Fuels, Lubricants, and Environmental Committee (FL&E) of the Locomotive Maintenance Officer's Association (LMOA).<sup>2</sup>

1.3 This test method is used primary for testing used diesel engine oils from railroad locomotive service. It may be applied to other samples types but precision, bias, and significance have not been determined for samples other than used railroad locomotive diesel engine oils.

1.4 This test method, in general, does not correlate with Test Method D893 on Insolubles in Lubricating Oils, since it uses separation by centrifugation and a more concentrated solution of anti-coagulant.

1.5 The correlation between this test method and Appendix A4 (Enhanced Thermal Gravimetric Analysis (TGA) Procedure) in Test Method D5967 has not been investigated.

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

1.7 *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.* For specific warning statements, see 7.2, 7.3, and 7.4.

<sup>1</sup> 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 D02.06 on Analysis of Liquid Fuels and Lubricants.

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<sup>2</sup> Locomotive Maintenance Officer's Association (LMOA), 6047 South Mobile Avenue, Chicago, IL 60638.

### 2. Referenced Documents

2.1 *ASTM Standards*:<sup>3</sup>

- D893 Test Method for Insolubles in Used Lubricating Oils
- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
- D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
- D5967 Test Method for Evaluation of Diesel Engine Oils in T-8 Diesel Engine

### 3. Terminology

3.1 *Definitions*:

3.1.1 *coagulate*, *v*—to cause to become viscous or thickened into a coherent mass. **D4175**

3.1.2 *coagulated pentane insolubles*, *n*—in used oil analysis, separated matter that results when a coagulant is added to a solution of used oil in pentane. **D4175**

3.1.2.1 *Discussion*—The addition of a coagulant will aid in separating finely divided materials that may have been held in suspension because of the dispersant characteristics of the oil.

3.1.2.2 *Discussion*—This test method uses a 1 % coagulant solution. Test Method D893 uses a 5 % coagulant solution.

3.1.3 *membrane filter*, *n*—porous article of closely controlled pore size through which a liquid is passed to separate matter in suspension. **D4175**

3.1.4 *pentane insolubles*, *n*—in used oil analysis, separated matter resulting when a used oil is dissolved in pentane. **D4175**

3.1.4.1 *Discussion*—In this test method, the separation is effected by paper filtration.

3.1.5 *used oil*, *n*—any oil that has been in a piece of equipment (for example, an engine, gearbox, transformer, or turbine), whether operated or not. **D4175**

<sup>3</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.

3.1.5.1 *Discussion*—In this test method, the oil can be any oil that has been used for lubrication of a locomotive diesel engine, whether engaged in railroad or other service.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *locomotive diesel engine oil, n*—lubricating oil formulated to meet the chemical, physical, and performance requirements defined by the LMOA for service in diesel engines in railroad locomotives.

### 3.3 Acronyms:

3.3.1 *ILS*—interlaboratory study

3.3.2 *LMOA*—Locomotive Maintenance Officer's Association

3.3.3 *RR*—railroad

3.3.4 *TGA*—thermo-gravimetric analysis

## 4. Summary of Test Method

4.1 A representative sample of used lubricating oil is mixed with pentane-coagulant solution and filtered under vacuum. The filter is washed with pentane, dried, and weighed to give coagulated pentane insolubles.

## 5. Significance and Use

5.1 Coagulated pentane insolubles can include oil-insoluble materials, some oil-insoluble resinous matter originating from oil or additive degradation, soot from incomplete diesel fuel combustion, or a combination of all three.

5.2 A significant change in coagulated pentane insolubles indicates a change in oil, and this could lead to lubrication system problems.

5.3 Coagulated pentane insolubles measurements can also assist in evaluating the performance characteristics of a used oil or in determining the cause of equipment failure.

5.4 High values of coagulated pentane insolubles have been associated with plugged oil filters, leading to opening of the bypass valve and circulation of unfiltered oil in the engine. This can lead to increased piston deposits, increased bearing wear, and premature engine failure.

## 6. Apparatus (see Fig. 1)

6.1 *Smooth-tip Forceps*.

6.2 *Graduated Cylinder*, 50 mL with stopper.

6.3 *Oven*, explosion-proof, capable of maintaining a temperature of  $50 \pm 3^\circ\text{C}$ .

6.4 *Oven*, explosion-proof, capable of maintaining a temperature of  $100 \pm 3^\circ\text{C}$ .

6.5 *Filtering Flask*, 1 L.

6.6 *Filter Holders*, borosilicate glass.

6.7 *Filter Membrane*, 0.45  $\mu\text{m}$ .

6.8 *Weighing Dish*, aluminum.

6.9 *Balance*, capable of weighing to the nearest 0.0001 g (0.1 mg) with a range of 160 g.

6.10 *Vacuum*, capable of maintaining 50.653 kPa (15 in. Hg) minimum.

6.11 *Stopwatch or Other Timing Device*, capable of measuring to the nearest 0.1 s with a range of at least 5 min.

## 7. Reagents and Solvents

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.<sup>4</sup> 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. If industrial grade pentane is used, it shall be filtered using 0.45  $\mu\text{m}$  filter paper.

7.2 *n-butyl diethanolamine* (2,2'-(butylimino) diethanol), 98 %. (**Warning**—May be harmful if inhaled or swallowed.)

7.3 *Pentane* (*n*-pentane), 98 %. (**Warning**—Extremely flammable. Vapors may cause flash fires.)

7.4 *Pentane-Coagulant Solution*, 1 %—Add 5 mL of *n*-butyl diethanolamine (**Warning**—Flammable.) to 500 mL of *n*-pentane (**Warning**—May be harmful if inhaled or swallowed.) and mix. Solution shall be used within one month and should preferably be made within a week of using.

## 8. Sampling

8.1 Obtain a sample using either Practice **D4057** or **D4177**.

8.2 Heat the sample of used oil to  $50 \pm 5^\circ\text{C}$  for  $\frac{1}{2}$  h  $\pm$  5 min in the original container and agitate until all sediment is homogeneously suspended in the oil. If the original container is of opaque material, or if it is more than three-fourths full, transfer the entire sample to a clear glass bottle having a capacity at least one third greater than the volume of the sample, and transfer all traces of sediment from the original container to the bottle by vigorous agitation of portions of the sample in the original container.

## 9. Procedure

9.1 Dry a clean filter membrane for 15 min in a desiccator, place in a labeled aluminum weighing dish, and weigh to the nearest 0.1 mg.

9.2 Place a 50 mL graduated cylinder on the balance, and tare it.

9.3 Remove the oil from the oven. Vigorously shake the sample by hand for a minimum of 30 s.

9.4 Using a medicine dropper, weigh approximately 0.25 g of oil sample into the graduated cylinder. Record the weight to the nearest 0.1 mg.

9.5 Add 10 mL pentane and agitate gently until the oil sample is fully dissolved.

9.6 Bring the volume in the graduated cylinder up to the 50 mL mark with fresh 1 % coagulant solution. Stopper and shake

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, Am. Chemical Soc., 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.