Designation: D7317 – 07

Standard Test Method for Coagulated Pentane Insolubles in Used Lubricating Oils by Paper Filtration (LMOA Method)[1]

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 (ε) 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).[2]

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.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D893 Test Method for Insolubles in Used Lubricating Oils

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum, Petroleum Products, 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**

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

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

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[2] Locomotive Maintenance Officer's Association (LMOA), 6047 South Mobile Avenue, Chicago, IL 60638.

[3] 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.

3.3.2 *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 ± 3°C.

6.4 *Oven*, explosion-proof, capable of maintaining a temperature of 100 ± 3°C.

6.5 *Filtering Flask*, 1 L.

6.6 *Filter Holders*, borosilicate glass.

6.7 *Filter Membrane*, 0.45 µm.

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.[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. If industrial grade pentane is used, it shall be filtered using 0.45 µm 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 ± 5°C for ½ h ± 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 gently, inverting the graduated cylinder four times in 4 to 5 s. Allow to stand for 30 min. Shake the solution similarly every 10 min of the 30 min.

9.7 Mount the filter on a dry holder and apply a vacuum. Mount and securely clamp the filter funnel to the filter holder. Ensure a minimum vacuum of 50.653 kPa (15 in. Hg) is attained and held.

9.8 Shake the sample gently one last time, pour into the filter funnel, and using the stopwatch or other timing device immediately start timing the flow rate.

9.9 Rinse the graduated cylinder twice using a minimum of 35 mL pentane and pour the rinsings into the filter funnel.

9.10 Stop timing when the last of the free liquid on the filter disappears. Record the flow time to the nearest second.

9.11 If filter flow time exceeds 5 min, re-run 9.4 to 9.10 with 0.1 mg sample.

9.12 Rinse the funnel wall with pentane from a squirt bottle.

9.13 Remove the filter funnel from the filter holder and rinse the filter membrane with a stream of pentane from the squirt

[4] *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.

FIG. 1 Paper Filtration Apparatus

bottle. Ensure the edges of the filter funnel are rinsed well to remove any oil trapped beneath the funnel.

9.14 Release the vacuum, and using the smooth-tip forceps transfer the filter membrane to its original weighing dish, and dry in the oven at 100 ± 3°C for 1 h ± 5 min.

9.15 Remove the weighing dish and filter membrane and cool to room temperature in a dessicator for a minimum of 1 h.

9.16 Weigh the filter membrane and weighing dish and record the stabilized weight to the nearest 0.1 mg.

TABLE 1 Calculated Precision at Selected Insolubles Levels

Insolubles Result, %	Repeatability, % (same units)	Reproducibility, % (same units)
0.00	0.11	0.42
0.20	0.12	0.47
0.40	0.13	0.51
0.60	0.14	0.56
0.80	0.16	0.61
1.0	0.17	0.66
1.5	0.20	0.77
2.0	0.23	0.89
2.5	0.26	1.0
3.0	0.29	1.1
3.5	0.32	1.2
4.0	0.35	1.4
4.5	0.38	1.5
5.0	0.41	1.6

10. Calculation

10.1 Calculate the percent coagulated pentane insolubles in the used oil as follows:

$$I = 100\,\frac{(A - B)}{C} \tag{1}$$

where:
I = coagulated pentane insolubles, %,
A = mass of filter and deposit, mg,
B = mass of filter, mg, and
C = mass of oil sample, mg.

11. Report

11.1 Report the percentage of coagulated pentane insolubles to two significant figures.

12. Precision and Bias

12.1 *Precision*—The precision of this test method was determined from the statistical analysis of an interlaboratory study (ILS), comprising six used railroad diesel engine oils measured four times each in 12 laboratories.[5]

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1610.

12.1.1 *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, and in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$0.061*(x + 1.77) \tag{2}$$

where:
x = average of the two results.

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

$$0.237*(x + 1.77) \tag{3}$$

where:
x = average of the two results.

NOTE 1—Precision is finite at a determined value of zero. Since the result of this test method is the difference of two weight determinations, it is possible to obtain negative numbers for a blank sample (true zero result) due to random weighing errors.

12.1.3 Calculated repeatability and reproducibility at selected insolubles values are given in Table 1.

12.2 *Bias*—The procedure in this test method has no bias because the mass percent of insoluble materials can only be defined in terms of this test method.

12.3 *Relative Bias*—Agreement between the results of this test method and those from Test Method D893 has not been investigated. In general, the two test methods are not expected to correlate.

13. Keywords

13.1 diesel; filtration; insolubles; LMOA; lubricating oil; pentane insolubles; used

APPENDIX

(Nonmandatory Information)

X1. TROUBLESHOOTING GUIDE

X1.1 During the development of the test method, a number of observations were made that helped operators recognize problems. These are described below.

X1.2 Repeatability was found to be best if the samples filtered in less than 5 min. With extended filter times, the weights tend to be high, since the insolubles on blocked filters cannot be rinsed enough to remove oil and coagulant residue.

X1.3 Residual coagulant will give high results. The following were found to indicate the presence of residual coagulant: curling of the filter edges with drying, a yellow ring around the edge of the insolubles, or a blotchy surface appearance that is tacky.

X1.4 In humid climates (>65 % relative humidity), the cooling caused by pentane evaporation may cause frost to form on both the filter and inside the holder while rinsing. If this happens, remove the filter after rinsing and dry for at least 1 h in a 100°C oven. An oven or compressed air may be used to thoroughly dry out a holder. Warm the dried filter holders on top of the oven until ready for use. Avoid using any holder that is still hot or damp.