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: D893 – 14 (Reapproved 2018)

Standard Test Method for Insolubles in Used Lubricating Oils[1]

This standard is issued under the fixed designation D893; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of pentane and toluene insolubles in used lubricating oils.

1.2 *Procedure A* covers the determination of insolubles without the use of coagulant in the pentane. It provides an indication of the materials that can readily be separated from the oil-solvent mixtures by centrifuging.

1.3 *Procedure B* covers the determination of insolubles in oils containing detergents and employs a coagulant for both the pentane and toluene insolubles. In addition to the materials separated by using Procedure A, this coagulation procedure separates some finely divided materials that may be suspended in the oil.

NOTE 1—Results obtained by Procedures A and B should not be compared since they usually give different values. The same procedure should be employed when comparing values obtained periodically on an oil in use or when comparing results determined by two or more laboratories.

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

1.5 *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.* For specific warning statements, see Section 7 and 9.1.1.

1.6 *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]

D1193 Specification for Reagent Water

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

3. Terminology

3.1 *Definitions:*

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

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.

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.

3.1.3 *coagulated toluene insolubles, n—in used oil analysis,* coagulated and separated matter not soluble in pentane or toluene.

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

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

3.1.5 *toluene insolubles, n—in used oil analysis,* the portion of pentane insolubles not soluble in toluene.

3.1.6 *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.

3.1.6.1 *Discussion*—In this test method, the oil can be any oil that has been used for lubrication.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *insoluble resins, n—in used oil analysis,* separated matter soluble in toluene but not pentane.

[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 D02.06 on Analysis of Liquid Fuels and Lubricants.

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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**

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3.2.1.1 *Discussion*—Insoluble resins can be calculated for either Procedure A or B by subtracting the toluene insolubles from the pentane insolubles.

4. Summary of Test Method

4.1 *Procedure A*—A representative sample of used lubricating oil is mixed with pentane and centrifuged. The oil solution is decanted and the precipitate washed twice with pentane, dried, and weighed to give the pentane insolubles. For toluene insolubles, a separate sample of the oil is mixed with pentane and then centrifuged. The precipitate is washed twice with pentane, once with toluene-alcohol solution, and once with toluene. The insoluble material is then dried and weighed to give the insolubles.

4.2 *Procedure B*—A representative sample of used lubricating oil is mixed with pentane-coagulant solution and centrifuged. The precipitate is washed twice with pentane, dried, and weighed to give coagulated pentane insolubles. For coagulated toluene insolubles a separate sample of the oil is mixed with pentane-coagulant solution and centrifuged. The precipitate is washed twice with pentane, once with toluene-alcohol solution, and once with toluene. The insoluble material is then dried and weighed to give coagulated toluene insolubles.

5. Significance and Use

5.1 Pentane insolubles can include oil-insoluble materials and some oil-insoluble resinous matter originating from oil or additive degradation, or both.

5.2 Toluene insoluble materials can come from (*1*) external contamination, (*2*) fuel carbon and highly carbonized materials from degradation of fuel, oil, and additives, or (*3*) engine wear and corrosion materials.

5.3 A significant change in pentane insolubles, toluene insolubles (with or without coagulant), and insoluble resins indicates a change in oil which could lead to lubrication system problems.

5.4 Insolubles measured can also assist in evaluating the performance characteristics of a used oil or in determining the cause of equipment failure.

6. Apparatus

6.1 *Centrifuge Tube,* cone-shaped, conforming to the dimensions given in Fig. 1, and made of thoroughly annealed glass. The graduations, numbered as shown in Fig. 1, shall be clear and distinct and the mouth constricted in shape for closure with a cork. Scale error tolerances and smallest graduations between various calibration marks are given in Table 1 and apply to calibrations made with air-free water at 20 °C.

6.2 *Centrifuge,* meeting all safety requirements for normal use and capable of whirling two or more filled centrifuge tubes at a speed that can be controlled to give a relative centrifugal force (rcf) between 600 and 700 at the tips of the tubes. The revolving head, trunnion rings, and trunnion cups, including the rubber cushion, shall be soundly constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall firmly support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to eliminate danger if any breakage occurs. Calculate the speed of the rotating head as follows:

$$\text{Speed, rpm} = 1337\sqrt{\text{rcf}/d} \quad (1)$$

where:

rcf = relative centrifugal force, and
d = diameter swing, mm, measured between tips of opposite tubes when in rotating position.

Table 2 shows the relationship between the diameter of swing, ref and rpm.

6.3 *Oven,* either explosion-proof, Class A, safety rated, or solvent venting, capable of maintaining a temperature of 105 °C ± 3 °C.

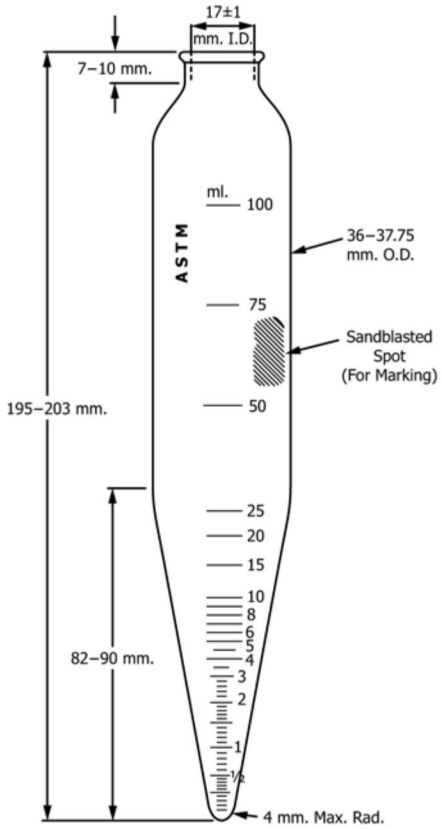

FIG. 1 ASTM Cone-Shaped Centrifuge Tube

TABLE 1 Calibration Tolerances of Cone-Shaped Centrifuge Tube

Range, mL	Smallest Scale Division, mL	Maximum Scale Error, mL
0 to 0.1	0.05	±0.02
Over 0.1 to 0.3	0.05	±0.03
Over 0.3 to 0.5	0.05	±0.05
Over 0.5 to 1.0	0.1	±0.05
Over 1.0 to 2.0	0.1	±0.10
Over 2.0 to 3.0	0.2	±0.10
Over 3.0 to 5.0	0.5	±0.20
Over 5.0 to 10.0	1.0	±0.50
Over 10 to 25	5.0	±1.0
Over 25 to 100	25.0	±1.0

TABLE 2 Rotation Speeds for Centrifuges of Various Diameters of Swing

Diameter of Swing, mm[A]	rpm at 600 rcf	rpm at 700 rcf
483	1490	1610
508	1450	1570
533	1420	1530
559	1390	1500

[A] Measured in millimetres between tips of opposite tubes when in rotating position.

6.4 *Balance,* having a sensitivity of 0.5 mg for weighing the 100 mL beaker and centrifuge tube, and a balance having a sensitivity of 0.1 g for weighing the oil sample.

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.[3] 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 *Purity of Water*—Unless otherwise indicated references to water shall be understood to mean water in accordance with Specification D1193, Type IV.

7.3 *n–butyl diethanolamine* (2,2[1]– (butylimino) diethanol). (**Warning—**May be harmful if inhaled or swallowed.)

7.4 *Ethanol, denatured*—conforming to either Formula 30 or 3A of the U.S. Bureau of Internal Revenue. (**Warning—**Flammable. Denatured. Cannot be made nontoxic.)

7.5 *Pentane*—(**Warning—**Extremely flammable. Vapors may cause flash fires.)

7.6 *Propan–2–ol (isopropyl alcohol)*—(**Warning—**Flammable. Denatured. Cannot be made nontoxic.)

7.7 *Toluene*—(**Warning—**Flammable. Vapor harmful.)

7.8 *Toluene-Alcohol Solution,* wash solvent, made by mixing 1 volume of toluene (7.7) with 1 volume of denatured ethanol conforming to either Formula No. 30 or 3A of the U.S. Bureau of Internal Revenue. (**Warning—**Flammable. Denatured. Cannot be made nontoxic.)

7.9 *Pentane-Coagulant Solution*—Add 50 mL of *n*-butyl diethanolamine (**Warning—**Flammable) and 50 mL of isopropyl alcohol (2-propanol) (**Warning—**As used oil may change appreciably in storage, samples should be tested as soon as possible after removal from the lubricating system and the dates of sampling and testing should be noted) to 1 L of *n*-pentane (**Warning—**May be harmful if inhaled or swallowed) and mix.

[3] *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.

8. Sampling

8.1 Obtain a sample using either Practice D4057 or D4177.

8.2 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 violent agitation of portions of the sample in the original container. Heat the sample of used oil at 60 °C ± 5 °C for 30 min ± 1 min and agitate until all sediment is homogeneously suspended in the oil. After complete suspension of all sediment, strain the sample or a convenient aliquot through a 150 µm (No. 100) sieve to remove large contaminating particles.

NOTE 2—When the sample is clear and transparent and visibly free of sediment, the straining procedure described above can be omitted.

9. Procedure A for Pentane and Toluene Insolubles Without Coagulant

9.1 *Pentane Insolubles:*

9.1.1 Dry a clean centrifuge tube for 30 min ± 1 min at 105 °C ± 3 °C, cool in a desiccator, and weigh to the nearest 1 mg. Weigh 10.0 g ± 0.1 g of the prepared sample of used oil into the tube and fill to the 75 mL mark with pentane (**Warning—**May be harmful if inhaled or swallowed.). Stopper the tube and shake until the mixture is homogeneous. Do not allow the mixture to stand more than 3 h.

9.1.2 Remove the stopper, and using a wash bottle having a fine jet, wash all insolubles from the stopper with pentane into the centrifuge tube and bring the solvent level up to the 100 mL mark. Arrange the tubes symmetrically about the centrifuge to minimize imbalance. In the event of an odd number of tubes, using water, fill a dummy tube to the same mass as the sample to balance the odd tube, and centrifuge for 20 min ± 1 min at a rate calculated in accordance with 6.2, sufficient to produce a relative centrifugal force (rcf) between 600 and 700 at the tips of the whirling tubes. (See Table 2.) Carefully decant the supernatant liquid without disturbing or dispersing the precipitate, leaving not more than 3 mL of liquid in the centrifuge tube.

NOTE 3—Due to safety concerns when handling flammable materials, some laboratories have found it suitable to use refrigerated or explosion-proof centrifuges or hermetically sealed centrifuge cups with screw caps and seals.

9.1.3 Add 10 mL ± 1 mL of pentane to the tube. Dislodge and break up all of the insolubles from the bottom of the tube by means of a clean stiff wire. Wash all insolubles adhering to the wire back into the tube with pentane, filling the tube to the 25 mL mark. Stopper the tube and shake until the mixture is homogeneous. Remove and wash the stopper with pentane, filling the tube to the 50 mL mark. Centrifuge for 20 min ± 1 min. Pour the supernatant liquid from the centrifuge tube, using care to avoid disturbing the cake of insolubles in the bottom of the tube.

9.1.4 Repeat the entire operation described in 9.1.3.

9.1.5 Dry the centrifuge tube containing the washed precipitate for 30 min ± 1 min at 105 °C ± 3 °C, cool in desiccator, and weigh to the nearest 1 mg.

9.1.6 With relatively heavy precipitates there can be some spattering if the tube is placed directly from the centrifuge into the oven. In such cases, the precipitate may be partially dried by weathering at room or slightly higher temperatures before being placed in the oven.

9.2 *Toluene Insolubles:*

9.2.1 Starting with a fresh sample of used oil, precipitate and centrifuge the pentane insolubles as described in 9.1.1 – 9.1.4, but do not dry the insolubles in the centrifuge tube.

9.2.2 Add 10 mL ± 1 mL of toluene-alcohol solution. Break up and dislodge all of the insolubles from the bottom of the centrifuge tube by means of a clean, stiff wire. Wash any insolubles adhering to the wire back into the tube with toluene-alcohol solution, using a sufficient amount of this solvent to fill the tube to the 25 mL mark. Stopper the tube and shake until the mixture is homogeneous. Remove the stopper and wash the adhering insolubles back into the tube with toluene-alcohol solution, and bring the total volume to 50 mL. Centrifuge for 20 min ± 1 min. Decant the clear solution, taking care not to disturb the precipitate.

9.2.3 Repeat the washing procedure described in 9.2.2, substituting toluene for the toluene-alcohol solution.

9.2.4 Dry for 60 min ± 1 min at 105 °C ± 3 °C, cool in a desiccator, and weigh to the nearest 1 mg.

10. Procedure B for Coagulated Pentane and Toluene Insolubles

10.1 *Coagulated Pentane Insolubles*—Proceed in accordance with 9.1.1 and 9.1.2, except in 9.1.1 use pentane-coagulant solution (7.9) in place of pentane. Wash the precipitate twice with pentane, dry and weigh as directed in 9.1.3, 9.1.4, and 9.1.5.

10.2 If the sample is known to contain water, or if water is indicated by a gummy precipitate, repeat the determination on a sample that has been dried by heating to 105 °C ± 3 °C in an open beaker.

10.3 *Coagulated Toluene Insolubles*—Proceed in accordance with 9.1.1 and 9.1.2 except in 9.1.1 use pentane-coagulant solution (7.9) in place of pentane. Wash twice with pentane as directed in 9.1.3 and 9.1.4. Wash with toluene-alcohol solution and toluene, dry, and weigh as directed in 9.2.2 – 9.2.4.

11. Calculation

11.1 Calculate the percentage of insolubles in the used oil as follows:

$$\text{Insolubles, \%} = 10\,(B - A) \tag{2}$$

where:

A = mass of clean, dried, centrifuge tube, g, and

B = mass of dried insolubles and centrifuge tube, g.

12. Report

12.1 If Procedure A (Section 9) was employed, report the percentages of pentane insolubles and toluene insolubles.

12.2 If Procedure B (Section 10) was employed, report the percentages of coagulated pentane insolubles and coagulated toluene insolubles.

12.3 Insoluble resins or coagulated insoluble resins may be reported for either Procedure A or B, respectively, by subtracting toluene insolubles from pentane insolubles.

13. Precision and Bias

13.1 *Precision*—The following criteria should be used for judging the acceptability of results (95 % confidence):

13.1.1 *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:

13.1.1.1 Procedure A, pentane insolubles, and Procedure B, coagulated pentane insolubles:

Insolubles, %	Repeatability, Same Units
0.0 to 1.0	0.07
Over 1.0	10 % of mean

13.1.1.2 Procedure A, toluene insolubles, and Procedure B, coagulated toluene insolubles:

Average Insolubles, %	Repeatability, Same Units[A]
0.1	0.068
0.2	0.097
0.3	0.12
0.4	0.14
0.5	0.15
0.6	0.17
0.7	0.18
0.8	0.19

[A] These precision data were derived as follows:

$$\text{Repeatability} = 0.216\sqrt{I}$$

where I = toluene insolubles or coagulated toluene insolubles.

13.1.2 *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:

13.1.2.1 Procedure A, pentane insolubles, and Procedure B, coagulated pentane insolubles:

Insolubles, %	Reproducibility, Same Units
0.0 to 1.0	0.10
over 1.0	15 % of mean

13.1.2.2 Procedure A, toluene insolubles:

Average Insolubles, %	Reproducibility, Same Units[A]
0.1	0.14
0.2	0.20
0.3	0.24
0.4	0.28
0.5	0.31
0.6	0.34
0.7	0.37
0.8	0.39
0.9	0.42
1.0	0.44
1.1	0.46

[A] These precision data were derived as follows:

$$\text{Reproducibility} = 0.441\sqrt{I_A}$$

where I_A = toluene insolubles.

13.1.2.3 Procedure B, coagulated toluene insolubles (see Note 4):

Average Insolubles, %	Reproducibility, Same Units[A]
0.1	0.30
0.2	0.43
0.3	0.53
0.4	0.60
0.5	0.68
0.6	0.74
0.7	0.80
0.8	0.86
0.9	0.91
1.0	0.96
1.1	1.00

[A] These precision data were derived as follows:

$$\text{Reproducibility} = 0.957\sqrt{I_B}$$

where I_B = coagulated toluene insolubles.

NOTE 4—The poor interlaboratory precision (reproducibility) of this portion of this test is such that Procedure B coagulated toluene insolubles, is unsuitable for the purpose of comparison of interlaboratory results.

13.2 *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.

14. Keywords

14.1 insoluble resins; insolubles; lubricating oil; pentane insolubles; toluene insolubles; used

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