Designation: D 893 – 05

An American National Standard

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

This standard is issued under the fixed designation D 893; 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 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 acceptable SI units are to be regarded as the 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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see 7.7, 7.8, 7.9, 9.1, and 9.7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 1193 Specification for Reagent Water

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 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.

3.2.1.1 *Discussion*—Insoluble resins can be calculated for either Procedue 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,

[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] 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.**

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:

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

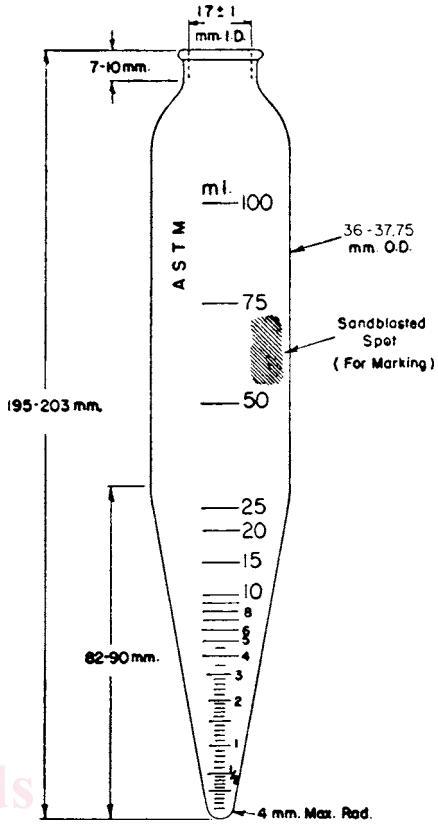

FIG. 1 ASTM Cone-Shaped Centrifuge Tube

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

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*, explosion-proof, capable of maintaining a temperature of 105 ± 3°C.

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.