



Designation: D91 – 02 (Reapproved 2012)^{ε1}

Standard Test Method for Precipitation Number of Lubricating Oils^{1,2}

This standard is issued under the fixed designation D91; 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.

^{ε1} NOTE—Subsection 5.2 was corrected editorially in July 2016.

1. Scope

1.1 This test method covers the determination of the precipitation number of steam cylinder stocks and black oils, and can be used for other lubricating oils.

1.2 The values stated in acceptable SI units are to be regarded as the standard.

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards*:³

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 of Terms Specific to This Standard:*

3.1.1 *ASTM precipitation number, of lubricating oils, n*—the number of millilitres of precipitate formed when 10 mL of lubricating oil are mixed with 90 mL of ASTM precipitation naphtha, and centrifuged under the conditions of the test.

4. Significance and Use

4.1 Fully refined petroleum oils normally contain no naphtha insoluble material. Semirefined or black oils frequently

contain some naphtha insoluble material (sometimes referred to as *asphaltenes*). This test measures the amount of naphtha insoluble material in the oil. This quantity is reported as the precipitation number.

5. Apparatus

5.1 *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 shall be constructed in a shape suitable 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.

5.2 *Centrifuge*, meeting all the safety requirements for normal use and capable of whirling two or more filled centrifuge tubes at a speed which 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 by means of the following equation:

$$\text{speed, r/min} = 1337 \sqrt{\text{rcf}/d} \quad (1)$$

where:

rcf = relative centrifugal force, and

d = diameter of swing, in mm, measured between tips of opposite tubes when in rotating position.

Table 2 shows the relationship between diameter swing, rcf, and revolutions per minute.

6. Reagent

6.1 *Hexanes, Reagent Grade.* (Warning—Extremely flammable, harmful if inhaled.)

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 This test method has been adopted for use by government agencies to replace Method 3101 of Federal Test Method Standard No. 791b.

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.