Designation: D 91 – 02

An American National Standard

Standard Test Method for Precipitation Number of Lubricating Oils[1,2]

This standard is issued under the fixed designation D 91; 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 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:*

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[3]

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 tip 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:

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

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.)

NOTE 1—Precipitation naphtha is sometimes referred to or sold by other names, such as petroleum naphtha, petroleum ether, ligroine, petroleum benzin, and industrial naphtha. One should confirm that it meets the requirements shown in 6.1.

7. Sampling

7.1 For sampling techniques, see Practices D 4057 or Practice D 4177.

8. Procedure

8.1 Add 10 ± 1 mL of the oil to be tested in each of two

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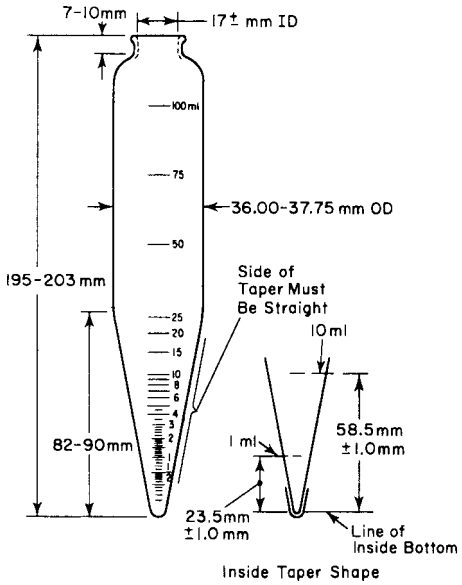
[2] This test method has been adopted for use by government agencies to replace Method 3101 of Federal Test Method Standard No. 791b.

[3] *Annual Book of ASTM Standards*, Vol 05.02.



NOTE—For volumetric tolerances see Table 1.

FIG. 1 ASTM Cone-Shaped Centrifuge Tube

TABLE 1 Calibration Tolerances for 200 mm Centrifuge Tube

Range, mL	Subdivision, mL	Volume Tolerance, mL
0 to 0.1	0.05	±0.02
Above 0.1 to 0.3	0.05	±0.03
Above 0.3 to 0.5	0.05	±0.05
Above 0.5 to 1.0	0.10	±0.05
Above 1.0 to 2.0	0.10	±0.10
Above 2.0 to 3.0	0.20	±0.10
Above 3.0 to 5.0	0.5	±0.20
Above 5.0 to 10	1.0	±0.50
Above 10 to 25	5.0	±1.00
Above 25 to 100	25	±1.00

TABLE 2 Rotation Speeds for Centrifuges of Various Diameters

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.

clean, dry centrifuge tubes at room temperature. Fill each tube to the 100-mL mark with hexanes and close tightly with a softened cork (not a rubber stopper). Then invert each tube at least 20 times, allowing the liquid to drain thoroughly from the tapered tip of the tube each time. Place the tubes in a water bath at 32 to 35°C for 5 ± 1 min. Momentarily remove the corks to relieve any pressure, and invert each tube again at least 20 times exactly as before. The success of this method depends to a large degree upon having a thoroughly homogeneous mixture which will drain quickly and completely from the tapered tip when the tube is inverted.

8.2 Balance the two centrifuge tubes or pairs of tubes with their respective trunnion cups and place them on opposite sides of the centrifuge head. Then whirl them for 10 min at a rate sufficient to produce a relative centrifugal force (rcf) between 600 and 700 at the tips of the whirling tubes (see 5.2). Repeat this operation until the volume of sediment in each tube remains constant for three consecutive readings. In general, not more than four whirlings will be required for oils having a low precipitation number.

9. Calculation and Report

9.1 Read the volume of the solid sediment at the bottom of each centrifuge tube, estimating to 0.1 mL or closer if possible. If the two readings differ by not more than 0.1 mL, report the mean of the two as the *ASTM Precipitation Number*. If the two readings differ by more than 0.1 mL, make two more determinations and report the average of the four determinations.

10. Precision and Bias

10.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.2 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Precipitation Number	Repeatability
0.00 to 1.20	10 % of Mean

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

Precipitation Number	Reproducibility
0.00 to 1.20	30 % of Mean

10.4 *Bias*—This procedure is empirical, precipitation number is defined solely by this procedure, therefore, no statement of bias can be made.

11. Keywords

11.1 asphaltenes; lubricating oils; precipitation number