Designation: D 2273 – 97 (Reapproved 2002)ε1

An American National Standard

Standard Test Method for Trace Sediment in Lubricating Oils[1]

This standard is issued under the fixed designation D 2273; 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.

ε[1] NOTE—Warning notes were editorially moved into the standard text in March 2003.

1. Scope

1.1 This test method covers the determination of trace amounts (less than 0.05 volume %) of sediment in lubricating oils. Since oil-soluble material precipitated by the specified solvent is not intended as part of the measured sediment, the test method is not applicable in cases where precipitated oil-soluble components will appreciably contribute to the sediment readings.

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 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure[2]

D 611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrcarbon Solvents[2]

D 1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method[2]

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

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *trace sediment, n*—the number of millilitres of sediment precipitated from 100 mL of oil sample (volume percent) when equal parts of the oil sample and the specified solvent are mixed and centrifuged under the prescribed conditions.

4. Significance and Use

4.1 This test measures the trace level amount of sediment that is naphtha-insoluble and can be separated by centrifuging. Excessive amounts of sediment in oil could lead to system malfunction in critical applications.

5. Apparatus

5.1 *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 as follows:

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

where:

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

The relationship between the diameter swing, relative centrifugal force, and revolutions per minute is given in Table 1.

5.2 *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 2 and apply to calibrations made with redistilled

[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] *Annual Book of ASTM Standards*, Vol 05.01.

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

TABLE 1 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 mm between tips of opposite tubes when rotating-position.

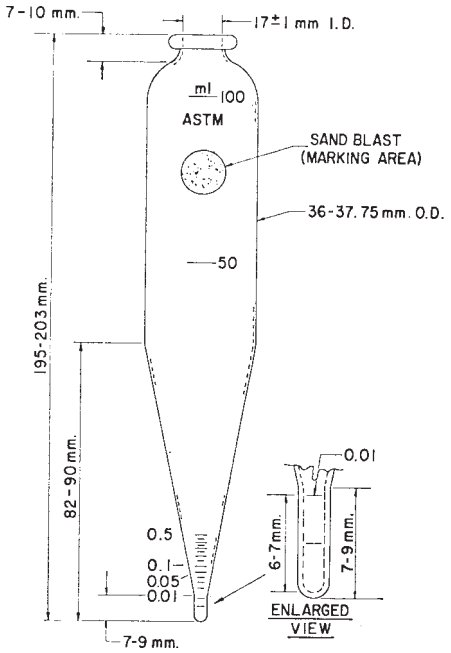

FIG. 1 Trace Sediment Tube

TABLE 2 Trace Sediment Tube Calibration Tolerances

Range, mL	Smallest Scale Division, mL	Scale Error, max, mL
0 to 0.01	0.005	±0.001 at 0.01
0.01 to 0.05	0.01	±0.005
0.05 to 0.15	0.05	±0.01
0.15 to 0.30	0.05	±0.02
0.30 to 0.50	0.05	±0.03
0.50 to 50	none	±1.0
50 to 100	none	±1.0

mercury up to the 0.30-mL mark and distilled water for all remaining marks at 20°C.

6. Reagent

6.1 *ASTM Precipitation Naphtha* —Shall conform to the requirements given in Table 3. (**Warning—**Extremely flammable. See also Note 1.)

TABLE 3 Naphtha

Test	Requirement	ASTM Designation
Density at 15°C	0.692 to 0.702	D 1298
Aniline Point	58 to 60°C	D 611
Initial Boiling Point	not less than 50°C	D 86
50 % Point	70 to 80°C	D 86
End Point	not more than 130°C	D 86

NOTE 1—Precipitation naphtha is sometimes referred to or sold by other names such as petroleum naphtha, petroleum ether, ligroine, petroleum benzin, and industrial naphthas.

NOTE 2—Before use, the naphtha should be free of any extraneous material which might affect the final test readings. For this purpose, it should either be filtered through a membrane filter or centrifuged several times and decanted just prior to its use.

7. Sampling

7.1 Refer to Practice D 4057 (manual) or D 4177 (automatic) for recommended practices for obtaining samples.

7.2 The sample shall be thoroughly representative of the material in question and the portion used for the test shall be thoroughly representative of the sample itself. This requires vigorous agitation of the sample immediately before transferring the sample to the tube. The difficulties in obtaining representative samples for this determination are unusually great; hence, the importance of sampling cannot be too strongly emphasized.

8. Procedure

8.1 Measure 50 ±1 mL of precipitation naphtha. (**Warning—**Extremely flammable.) In each of two clean, dry centrifuge tubes at room temperature. Then fill each tube to the 100-mL mark with the oil sample and close tightly with a softened cork covered with a thin pliable plastic film[4] which is resistant to petroleum products (not a rubber stopper). Shake sample well to ensure complete mixing, then invert each tube at least 20 times allowing the liquid to drain thoroughly from the tip of the tube at each inversion. If the liquid refuses to drain upon inversion, gently tap the inverted tube against the palm of the hand to jolt the liquid out of the tip. 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 completely as possible from the 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 ±1 min at a rate sufficient to produce a relative centrifuge force (rcf) between 600 and 700 at the tips of the whirling tubes (see 5.1). At the end of the 10-min whirling period, decant the mixture carefully, allowing the sediment to remain in the tubes. Measure another 50 ±1 mL of the naphtha into each of the two tubes, then add the oil sample until the total volume reaches the 100 mL mark. Then stopper the tubes, repeatedly invert, heat, and again invert as described in 8.1. Whirl the tubes in the centrifuge for 10 ±1 min as before, repeating the 10-min whirling 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 low sediment values. Record the final reading for sediment in each tube.

[4] Saran film has been found suitable for this purpose.

9. Calculation

9.1 Average the final readings for sediment in the two tubes containing the sample to obtain the volume of sediment per 100-mL sample. Report the result to the nearest 0.001 %.

10. Precision and Bias

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

10.1.1 *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:

Sediment, percent volume	Repeatability
0.000 to 0.002	0.001
0.003 to 0.005	0.001
0.006 to 0.01	0.002

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

Sediment, percent volume	Repeatability
0.000 to 0.002	0.001
0.003 to 0.005	0.001
0.006 to 0.01	0.002

10.1.3 *Bias*—Test Method D 2273 is empirical and no statement of bias can be made.

11. Keywords

11.1 lubricating oils; trace sediment