Designation: D 1698 – 03

Standard Test Method for Sediments and Soluble Sludge in Service-Aged Insulating Oils[1]

This standard is issued under the fixed designation D 1698; 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.

1. Scope

1.1 This test method covers the determination of sediment and soluble sludge in service-aged insulating oils of petroleum origin. Also, provision is made for determining organic and inorganic content of the sediment. The method is intended primarily for oils of comparatively low viscosity; for example 5.7 to 13.0 cSt (mm^2/s) at 40°C (104°F). Suitability for high viscosity oils have not been determined.

1.2 This standard may involve hazardous materials, operations, and equipment. *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 923 Practices for Sampling Electrical Insulating Liquids[2]

D 2440 Test Method for Oxidation Stability of Mineral Insulating Oil[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *inorganic sediment*—that portion of the total sediment which remains after ignition at 500°C.

3.1.2 *organic sediment*—that portion of the total sediment which is lost during ignition at 500°C.

3.1.3 *sediment*—any solid substance or substances that are insoluble in the oil under test at ordinary room temperatures, and that can be separated by centrifuging under certain prescribed conditions.

3.1.4 *soluble sludge*—oil deterioration products or contaminants, or both, which become insoluble upon dilution with *n*-pentane under prescribed conditions.

4. Summary of Test Method

4.1 A sample portion is centrifuged to separate sediment from the oil. The upper, sediment-free portion is decanted and retained for determination of soluble sludge. The sediment is dislodged and filtered through a filtering crucible. After drying and weighing to obtain total sediment the crucible is ignited at 500°C and reweighed. Loss in weight is organic and the remainder is inorganic content of sediment. Soluble sludge is determined on the sediment-free portion by dilution with *n*-pentane to precipitate *n*-pentane insolubles, and filtration through a filtering crucible or 0.45 µm filter membrane.

5. Significance and Use

5.1 Sediment in insulating oil may deposit on transformer parts and interfere with heat transfer and may choke oil ducts; thus hindering oil circulation and heat dissipation. Inorganic sediment usually indicates contamination of some type and organic sediment indicates either deterioration of the oil or contamination.

5.2 Soluble sludge indicates deterioration of the oil, presence of contaminants, or both. It serves as a warning that formation of sediment may be imminent.

5.3 The determination of sediment and soluble sludge in a used insulating oil assists in deciding whether the oil may continue to be used in its existing condition or should be replaced, reclaimed, or reconditioned.

6. Apparatus

6.1 *Centrifuge Tube*, 30-mL capacity, preferably with round or elliptical bottom to facilitate removal of sediment. Pear-shaped tubes are not recommended.

6.2 *Centrifuge*, 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

[1] This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.07 on Physical Tests.

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[2] *Annual Book of ASTM Standards*, Vol 10.03.

case strong enough to eliminate danger if any breakage occurs. Calculate the speed of the rotating head by means of the following equation:

$$\text{rpm} = 265\sqrt{\text{rcf}/d}$$

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

or:
d = diameter of swing cm/2.54, measured between tips of opposite tubes when in rotating position.

6.3 *Electric Muffle Furnace*, capable of maintaining a temperature of 500 ± 15°C.

6.4 *Explosion-Proof Oven*, capable of maintaining a temperature of 105 ± 3°C.

6.5 *Filtering Crucible*—Glass filter crucible having a maximum diameter of the pores of between 5 and 15 µm as determined in accordance with the Appendix in Test Method D 2440.

6.6 *Filter Membrane*—A 47 mm diameter, 0.45 µm pore size cellulose ester type filter.

7. Reagents and Materials

7.1 *Acetone*—Reagent grade.

7.2 *Chloroform*, Reagent grade.

7.3 *Normal Pentane*, Reagent grade.

8. Sampling

8.1 Obtain the sample of used oil in accordance with Practices D 923. In addition, protect the sample from light.

9. Procedure for Total Sediment

9.1 Clean the glass filtering crucible and dry in an air oven at 105 to 110°C until it has reached constant weight. Dry filter membranes in an oven at 105°C for 15 min. Cool and store the crucible in a desiccator and, when needed, weigh to the nearest 0.0001 g.

9.2 Thoroughly agitate the sample of used oil to produce a homogenous mixture, and place 25 g of the well-mixed oil in each of two centrifuge tubes. Balance the two centrifuge tubes and their respective trunnion cups, place them on opposite sides of the centrifuge head, and centrifuge for 30 min at a relative centrifugal force between 600 and 700 at the tips of the tubes.

9.3 Carefully decant the supernatant liquid without dispersing the precipitated sediment, saving the decanted portion for determination of soluble sludge. Dislodge and wash the sediment from the centrifuge tube into a tared, filtering crucible, using n-pentane as the washing agent. Make certain that any sediment adhering to instruments used in cleaning the tube is also transferred to the filtering crucible. A filter membrane may be used instead of the filtering crucible if only total sediment is to be determined.

9.4 Wash the sediment in the filtering crucible thoroughly with n-pentane to remove all traces of oil. Normally, three washes of 25-mL portions each are sufficient. A drop of the final n-pentane washing should show no oil stain on a filter paper.

9.5 Dry the sediment in the filtering crucible by suction, and place in an oven at 105°C for drying to constant weight. Cool in a desiccator and weigh to the nearest 0.0001 g.

10. Procedure for Organic and Inorganic Sediment

10.1 Wash the sediment in the filtering crucible with chloroform or acetone until it is free of chloroform or acetone-soluble material (see Note). Three washes of 25-mL portions each are usually sufficient.

10.2 Dry the sediment by suction and ignite in the crucible in an electric muffle furnace at 500°C for 1 h, cool in a desiccator, and weigh to the nearest 0.0001 g.

NOTE 1—If so desired, the chloroform or acetone washes can be combined and retained for further chemical analysis.

11. Procedure for Soluble Sludge

11.1 Place the crucible in an oven and dry at 105 to 110°C to constant weight or dry a 0.45 µm filter membrane filter in an oven at 105°C for 15 min. Cool in a desiccator and weigh to the nearest 0.0001 g.

11.2 Transfer 20 g of the decanted portion of the centrifuged oil to a 600-mL high-form beaker with pouring spout. Add 200 mL of *n*-pentane, mix thoroughly, and place a cover glass or alumninum foil on the beaker. Place the covered beaker in an area protected from light and drafts, and let it stand overnight, or approximately 16 h, at ordinary room temperature.

11.3 Filter the oil-pentane solution through the tared filtering crucible or filter membrane, making certain that all solid material is transferred to the crucible or filter membrane by thoroughly cleaning and washing the beaker with *n*-pentane.

11.4 Wash the soluble sludge in the filtering crucible thoroughly with *n*-pentane to remove all traces of oil. Normally three washes of 25-mL portions each are sufficient.

11.5 Dry the soluble sludge in the filtering crucible or filter membrane by suction and place in an oven at 105°C for drying to constant weight. Cool in a desiccator and weigh to the nearest 0.0001 g.

12. Calculation

12.1 Calculate the percentage of total sediment in the used oil as follows:

$$\text{Total sediment, \%} = 100/25(B - A) \text{ or } 4(B - A)$$

where:
A = weight of tared ignited filtering crucible, and
B = weight of oven-dried sediment and filtering crucible, g.

12.2 Calculate the percentage of organic sediment in the used oil as follows:

$$\text{Organic sediment, \%} = 100/25(B - C) \text{ or } 4(B - C)$$

where:

B = weight of oven-dried sediment and filtering crucible, g, and
C = weight of ignited sediment and filtering crucible, g.

12.3 Calculate the percentage of inorganic sediment in the used oil as follows:

$$\text{Inorganic sediment, \%} = 100/25(C - A) \text{ or } 4(C - A)$$

where:
A = weight of tared, ignited filtering crucible, g, and
C = weight of ignited sediment and filtering crucible, g.

12.4 Calculate the percentage of soluble sludge in the used oil as follows:

$$\text{Soluble sludge, \%} = 100/20(E - D) \text{ or } 5(E - D)$$

where:
D = weight of tared, oven-dried filtering crucible or filter membrane, g, and
E = weight of soluble sludge and filtering crucible or filter membrane, g.

13. Report

13.1 Report the following information and data, recording percentages below 1 % to three digits and percentages of 1 % or more to two digits:

13.1.1 Date on which the sample of used oil was removed from service,
13.1.2 Date on which the sample was tested,
13.1.3 Total sediment, in %,
13.1.4 Organic sediment, in %,
13.1.5 Inorganic sediment, in %, and
13.1.6 Soluble sludge, in %.

14. Precision and Bias

14.1 *Repeatability*—Two distinct measurements of soluble sludge, made in one laboratory on different days, should differ by no more than 0.013 % soluble sludge, 95 % of the time.

14.2 *Reproducibility*—Two laboratories, each reporting the average of two distinct measurements of soluble sludge, should differ by no more than 0.044 % soluble sludge, 95 % of the time.

14.3 *Bias*—The foregoing statements are restricted to the measurement of soluble sludge, which may be assumed to be uniformly dispersed through the oil sample. Since solid sediment is not uniformly dispersed but occurs in discrete particles, no meaningful statement as to bias can be made.

15. Keywords

15.1 insulating oils; oils; sediments; sludge; soluble-sludge