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Designation: 375/99

Standard Test Method for Determination of Total Sediment in Residual Fuels[1]

This standard is issued under the fixed designation D4870; 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*~~Scope

1.1 This test method covers the determination of total sediment up to 0.40 % m/m for distillate fuel oils containing residual components and to 0.50 % m/m in residual fuel oils having a maximum viscosity of 55 cSt (mm^2/s) at 100°C. Some fuels can exceed the maximum filtration time specified in this test method due to factors other than the presence of significant quantities of insoluble organic or inorganic material. This test method can be used for the assessment of total sediment after regimes of fuel pretreatment designed to accelerate the aging process.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.* For specific warning statements, see 7.2, 7.3, Annex A1, and X1.6.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *total sediment*—the sum of the insoluble organic and inorganic material that is separated from the bulk of the residual fuel oil by filtration through a Whatman GF/A filter medium, and that is also insoluble in a predominantly paraffinic solvent.

4. Summary of Test Method

4.1 A weighed quantity (10 g) of the oil sample is filtered through the prescribed apparatus at 100°C. After solvent washing and drying the total sediment on the filter medium is weighed. The test is to be carried out in duplicate.

5. Significance and Use

5.1 Appreciable amounts of sediment in a residual fuel oil can cause fouling of facilities for handling, and give problems in burner mechanisms. Sediment can accumulate in storage tanks, on filter screens, or on burner parts, resulting in obstruction of the flow of oil from the tank to the burner.

6. Apparatus

6.1 *Filtration Apparatus,* constructed of brass, with copper steam coils attached, suitably supported above a vacuum flask appropriately protected against the effects of implosion. See Figs. 1 and 2.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability and Cleanliness of Liquid Fuels.

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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**

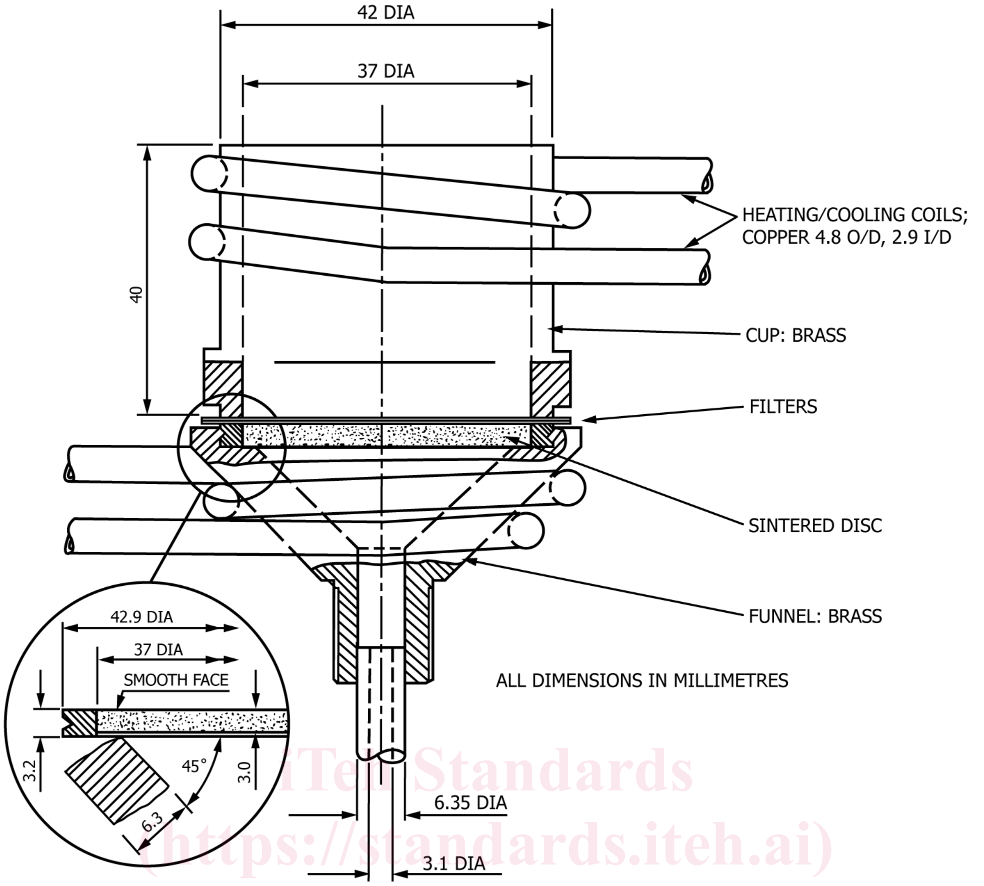

FIG. 1 Detail of Filtration Cell

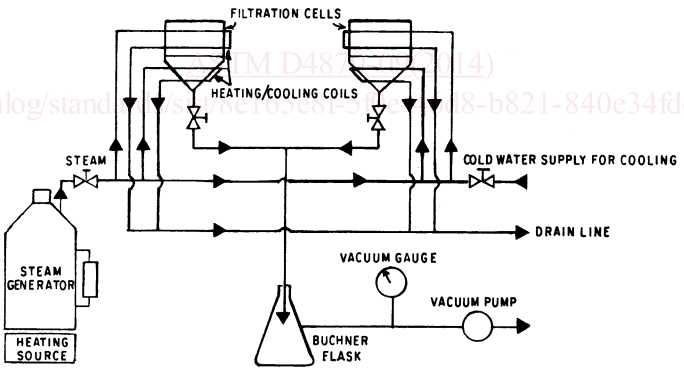

FIG. 2 Arrangement of Filtration Apparatus

6.2 *Temperature Measuring Device* , capable of measuring the temperature in the range from 95 to 105°C with an accuracy of 0.5°C.

6.3 *Oven,* electric, capable of maintaining a temperature of 110 ± 1°C. The oven should be capable of safely evaporating the solvent without risk of fire.

6.4 *Stirring Rod,* glass or polytetrafluoroethylene (PTFE) approximately 150 mm in length and 3 mm in diameter.

6.5 *Beaker,* glass, 30 mL capacity, either squat form with lip or conical.

6.6 *Small Dishes,* such as watch glasses or Petri dishes.

6.7 *Hotplate,* electric.

6.8 *Steam Generator,* to provide steam at 100 ± 1°C.

6.9 *Vacuum Source,* capable of providing the specified vacuum.

6.10 *Vacuum Gauge,* capable of measuring the specified vacuum.

6.11 *Filter Medium,* Whatman glass fiber filter medium, Grade GF/A, 47 mm diameter.

6.12 *High Speed Mixer,* any convenient type, minimum speed 400 rpm.

6.13 *Desiccator.*

6.14 *Cooling Vessel,* a desiccator or other type of tightly covered vessel for cooling the filter media before weighing. The use of a drying agent is not recommended.

6.15 *Syringe or Graduated Wash Bottle* , minimum capacity 25 mL, graduated in 0.5 mL increments.

6.16 *Forceps,* spade-ended.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Normal Heptane,* minimum 99.75 % purity. (**Warning—**Flammable, vapor harmful if inhaled. See A1.1.)

7.3 *Toluene,* at least reagent grade purity. (**Warning—**Flammable, vapor harmful. See A1.2.)

7.4 *Wash Solvent,* consisting of 85 volume % *n*-heptane (7.2) and 15 volume % toluene (see 7.3).

8. Sampling

8.1 Sample in accordance with Practice D4057 or Practice D4177.

9. Procedure

9.1 *Sample Preparation*—Mix the whole sample, as received, thoroughly using a high speed mixer when practicable, for 30 s. In all cases a sample taken on a glass or PTFE rod dipped to the bottom of the container must show a homogeneous appearance. For fuels with a high wax content (high pour point), or of very high viscosity, the sample must be heated before stirring. The temperature must be either 15°C above the pour point in the case of low viscosity fuels, or that equivalent to 150 to 250 cSt in the case of high viscosity fuels. In no case should the temperature exceed 80°C.

9.2 *Filter Preparation*—For each test, dry two filter media for 20 min in the oven at 110°C. Transfer each filter medium, separately, rapidly to a cooling vessel and allow to cool to room temperature for 20 min. Weigh each filter medium to the nearest 0.0001 g.

NOTE 1—The Whatman GF/A filter media are fragile and are to be handled with care. Before use, check each medium for consistency, and the possible presence of small defects (holes).

NOTE 2—For convenience, place the filters on numbered small dishes (6.6) during drying and cooling.

9.3 *Apparatus Assembly*—Before use, check that the filter support screen is clean. If necessary, the screen must be cleaned by boiling in a high boiling point aromatic solvent. When more than 2 % of the sinter area remains blocked by particulate matter after such cleaning, discard the screen and install a new one.

9.3.1 The filtration unit must be clean and dry before assembly. Stack the two previously dried and weighed filter media on top of the sinter support with the mesh imprint side down, using forceps. Apply slight vacuum to aid in centering the filter media, and place the top portion of the filtration apparatus carefully on to the media before clamping. Shut off the vacuum and pass steam at 100 ± 1°C through the heating/cooling coils for 10 min prior to sample addition.

9.4 *Sample Addition*—Into a 30 mL beaker, pour approximately 11 g of the fuel sample prepared as described in 9.1 and weigh to the nearest 0.01 g. Connect the vacuum source and apply vacuum to an absolute pressure of 40 ± 2 kPa minimum (61.3 kPa vacuum). Heat the contents of the beaker to 100 ± 2°C. Then transfer the contents at 100 ± 2°C (Note 3) to the center of the filter medium, taking care that no sample touches the walls during transfer (Note 4). Reweigh the beaker to the nearest 0.01 g. The quantity transferred should be 10± 0.5 g. When filtration is not complete in 25 min, discontinue the test and repeat using 5 ± 0.3 g of sample. If filtration is still not complete in 25 min, report the result as *filtration exceeds 25 min*.

NOTE 3—It is expedient to weigh the beaker plus stirrer plus temperature measurement device before and after transfer to avoid errors incurred by attempting to obtain a net weight. Any convenient means of heating the fuel sample to 100± 2°C may be used, such as hot plate, water or oil bath, or an oven when equipped with a suitable stirrer. Samples that overheat above 105°C must be discarded and not reused.

NOTE 4—For samples of high viscosity or high sediment, level filtration will be aided by small stage or even dropwise addition. It is preferable to avoid

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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

complete coverage of the filter medium with unfiltered oil sample. For samples of low filtration rate the pressure of 40 ± 2 kPa should be maintained for the 25-min period.

9.5 *Filter Washing*—When the filtration is complete and the upper filter medium appears dry, continue the steam and vacuum for a further 5 min. Discontinue the steam supply and cool the apparatus by passing tap water through the coils. Wash the filtration unit carefully with two portions of 25 ± 1 mL of the wash solvent from a syringe or graduated wash bottle with a fine nozzle, taking care to remove any adhered sample from the wall of the upper part of the apparatus. Carefully remove the top portion of the filtration unit and wash the rim of the filter with a further 10 ± 0.5 mL of the wash solvent in a similar manner. Finally wash the whole of the filter medium area with 10 ± 0.5 mL of *n*-heptane.

NOTE 5—If the sample filters very rapidly, the vacuum should be released before the first solvent washing, to ensure complete coverage of the filter medium area by solvent. The vacuum should then be gently reapplied for the subsequent operations.

9.6 *Apparatus Disassembly*—After the filter medium appears dry, discontinue the vacuum supply. Using the forceps, carefully remove each filter medium separately and transfer them to the oven at 110°C. Dry for 20 min and quickly transfer them to the cooling vessel (6.14). Allow them to cool to room temperature for 20 min and reweigh them to the nearest 0.0001 g.

10. Calculation

10.1 Calculate the mass percentage of total sediment for each test specimen using Eq 1. For each test specimen with a calculated total sediment concentration >0.005 % m/m as determined by Eq 1, record the mass percentage of total sediment to the nearest 0.01 % m/m. For each test specimen with a total sediment concentration ≤0.005 % m/m, record the result as 0.00 % m/m.

$$S = \frac{(M_5 - M_4) - (M_3 - M_2)}{10\,M_1} \tag{1}$$

where:

S = total sediment, % m/m,
M_1 = mass of sample, g,
M_2 = mass of lower filter medium before filtration, mg,
M_3 = mass of lower filter medium after filtration, mg,
M_4 = mass of upper filter medium before filtration, mg, and
M_5 = mass of upper filter medium after filtration, mg.

11. Report

11.1 Report the total sediment by hot filtration as the average of duplicate determinations, to the nearest 0.01 % m/m. If the average of the duplicate determinations is <0.01 % m/m, report it as "<0.01 % m/m." If a 5-g sample has been used, report the results as total sediment (5 g) by hot filtration. If filtration is not complete within the specified 25 min, report the results as *filtration time exceeds 25 min*.

11.2 *Test Report*—The test report shall contain at least the following information:

11.2.1 The type and identification of the product tested,
11.2.2 A reference to this test method,
11.2.3 The result of the test (see 11.1),
11.2.4 Any deviation, by agreement or otherwise, from the standard procedures specified (see 11.1), and
11.2.5 The date of the test.

12. Precision and Bias[4]

12.1 *Precision*—The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows. The results ranged from 0.01 to 0.40 % m/m for distillate fuel oils containing residual components and from 0.01 to 0.50 % m/m for residual fuel oils.

12.1.1 *Repeatability*—The difference between successive test results, expressed as the average of duplicate determinations, 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:

$$r = 0.089\sqrt{x} \text{ for residual fuels, and} \tag{2}$$

$$r = 0.048\sqrt{x} \text{ for distillate fuels containing residual components} \tag{3}$$

where x = the average of the test results, % m/m.

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1238.

12.1.2 *Reproducibility*—The difference between two test results, expressed as the average of duplicate determinations independently obtained by different operators operating in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only one case in twenty:

$$R = 0.294\sqrt{x} \text{ for residual fuels, and} \tag{4}$$

$$R = 0.174\sqrt{x} \text{ for distillate fuels containing residual components} \tag{5}$$

where x = the average of the test results, % m/m.

12.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias cannot be determined.

13. Keywords

13.1 ageing; fuel oils; hot filtration; residual fuel; sediment; storage stability; total sediment

ANNEX

(Mandatory Information)

A1. HAZARD STATEMENTS

A1.1 *n*-Heptane

A1.1.1 Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

A1.2 Toluene

A1.2.1 Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

APPENDIX

(Nonmandatory Information)

X1. PREDICTION OF TOTAL SEDIMENT IN RESIDUAL FUEL OILS (STANDARD PROCEDURES FOR AGEING)

X1.1 Scope

X1.1.1 This test method describes two procedures for evaluating the sediment forming tendency of residual fuel oils during storage.

NOTE X1.1—Experience has shown that this test method can be applicable to other fuel oils as well, but precision is unknown and additional testing is required to determine that precision.

X1.1.2 Procedure A covers thermal ageing.

X1.1.3 Procedure B covers chemical ageing.

X1.2 Terminology

X1.2.1 *Definitions of Terms Specific to This Standard:*

X1.2.1.1 *accelerated total sediment*—the total sediment formed in a fuel when it is diluted with hexadecane and stressed at an elevated temperature.

X1.2.1.2 *potential total sediment*—the total sediment formed by stressing a fuel at an elevated temperature.

X1.3 Summary of Test Method

X1.3.1 *Procedure A for Thermal Ageing* —A sample of residual fuel oil is aged at 100°C for 24 h.

X1.3.2 *Procedure B for Chemical Ageing* —A sample of residual fuel oil is diluted with hexadecane (cetane) and then heated to 100°C for 1 h.

X1.3.3 Following ageing by the above procedures, the sample is then filtered at 100°C, solvent washed and dried, and the total sediment on the filter medium weighed, in accordance with Test Method D4870. The test is to be carried out in duplicate.

X1.4 Significance and Use

X1.4.1 The precipitation of asphaltenes from a residual fuel oil during storage and handling can cause severe difficulties which, in extreme cases, can render the fuel unfit for use. Once out of solution, it is extremely difficult to repeptize the asphaltenes into their original state.

X1.4.2 The procedures described in this test method are useful for determining whether sediment is likely to precipitate from a residual fuel oil during storage.

X1.4.3 Thermal ageing is a well established technique for determining whether sediment will precipitate from residual fuel oils during storage and handling.

X1.4.4 Chemical ageing looks at the balance between the required aromaticity of the asphaltenes and the available aromaticity of the oil phase, and tests whether a specified amount of a normal alkane disturbs this balance to the extent that asphaltene precipitation occurs. Experience has shown that such precipitation indicates that sediment can form in the residual fuel oil during storage and handling.

X1.5 Apparatus

X1.5.1 *Oil Bath,* a constant temperature bath capable of being maintained at a temperature of 100 ± 0.5°C, and large enough to hold two or more air wells through cover openings, and with an additional opening in the cover through which the oil bath temperature measuring device can be suspended.

X1.5.2 *Air Well,* cylinder 55 mm in inside diameter and 120 mm in length, provided with a cover having a central opening approximately 10 mm in diameter for supporting the condenser tube in a vertical position.

NOTE X1.2—The air well and oil bath can be made in one assembly as shown in Fig. X1.1.

X1.5.3 *Oil Bath Temperature Measuring Device,* capable of measuring the temperature in the range from 95 to 103°C with an accuracy of 0.5°C.