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

This standard is issued under the fixed designation D 4870; 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 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 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.* For specific warning statements, see 7.2, 7.3, Annex A1, and X1.6.1.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

E 1 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.

6.2 *Thermometer*, partial immersion type ranging from approximately 95 to 103°C. Maximum length 220 mm. Maximum graduation interval 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 *Weighing Bottles*, with ground glass stoppers, numbered, 80 mm diameter by 40 mm.

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.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.14 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.**

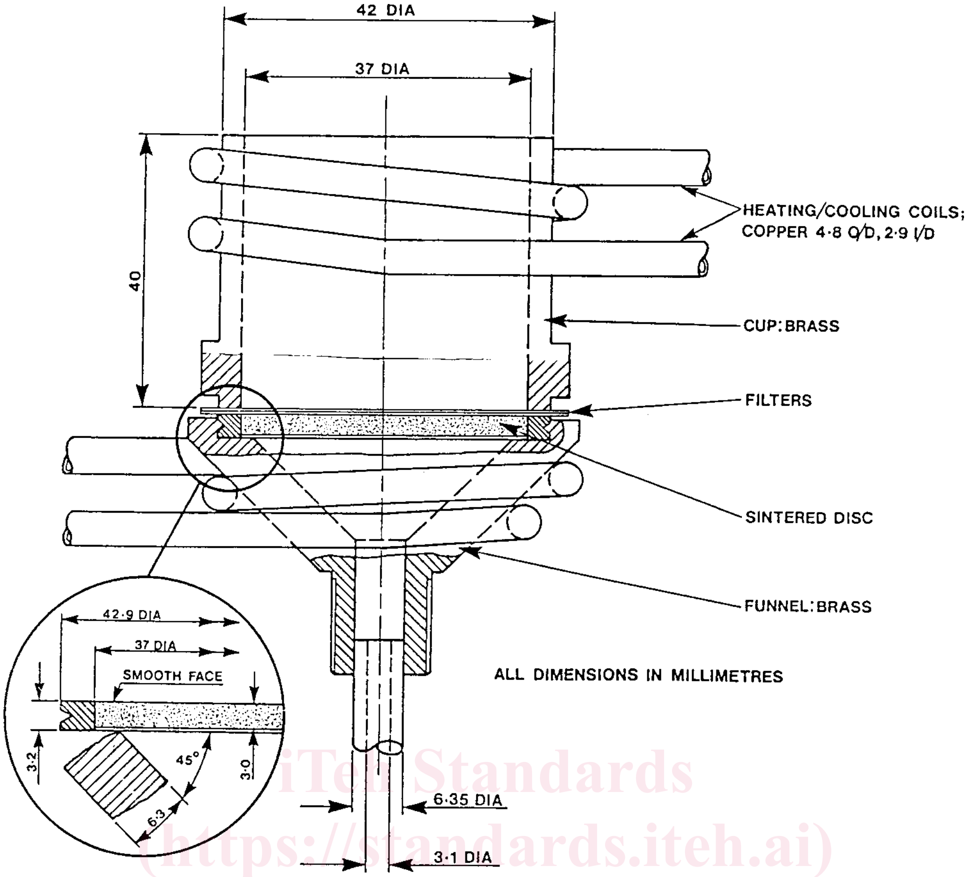

FIG. 1 Detail of Filtration Cell

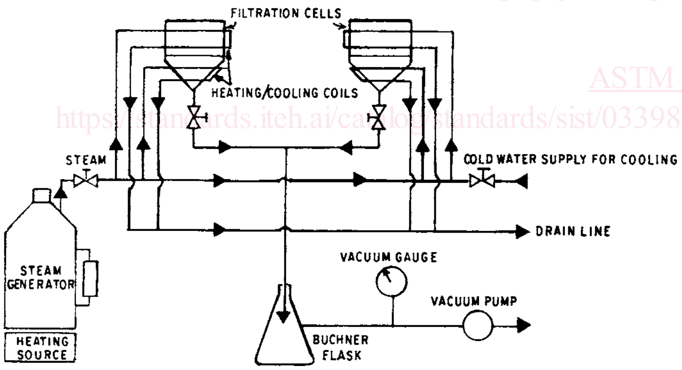

FIG. 2 Arrangement of Filtration Apparatus

6.10 *Vacuum Gage*, 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 D 4057 or Practice D 4177.

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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.

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 paper, separately, rapidly to a numbered weighing bottle and allow to cool in the cooling vessel to room temperature (5 to 10 min). Where two-pan balances are used, weigh each weighing bottle plus filter medium by the tare method against an empty similar bottle, to the nearest 0.001 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, it is useful to have a number of weighing bottles dedicated to the procedure, the lightest of which is chosen as the tare. All weighing bottles should be stored in a desiccator in the vicinity of the balance. Do not place these weighing bottles in the oven since their weights are in equilibrium with the desiccant.

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, placing the one from the lower numbered weighing bottle on the bottom. 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 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 same numbered weighing bottles as used in 9.2. Allow them to cool in the cooling vessel (6.14) to room temperature (5 to 10 min) and reweigh them (against tare) to the nearest 0.0001 g.

10. Calculation

10.1 Calculate the mass percentage of total sediment to the nearest 0.01 % m/m using the following equation:

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

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 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