



Designation: D4740 – 19

# Standard Test Method for Cleanliness and Compatibility of Residual Fuels by Spot Test<sup>1</sup>

This standard is issued under the fixed designation D4740; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope\*

1.1 This test method covers separate procedures for determining the cleanliness of residual fuel oil and the compatibility of a residual fuel oil with a blend stock. It is applicable to residual fuel oils with viscosities up to 50 cSt (1 cSt = 1 mm<sup>2</sup>/s) at 100 °C. This test method describes two protocols: one manual and one automated.

NOTE 1—This test method has not been evaluated for heavy distillate having the propensity to leave a wax sediment on the filter paper and contain no residual asphaltene.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels.

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<sup>2</sup> 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.

2.2 *ASTM Adjuncts:*<sup>3</sup>

Reference Spot Sheet

## 3. Terminology

3.1 For definitions of terms used in this standard, see Terminology D4175.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *blend stock, n*—one component or more components used to make a final fuel product.

3.2.1.1 *Discussion*—In heavy fuel oils, blend stocks include various refinery streams ranging from kerosine to heavy distillates, residuals, and asphalts.

3.2.2 *cleanliness, n*—of residual fuel, the concentration of suspended solids in a finished sample.

3.2.2.1 *Discussion*—The lower the concentration of suspended solids, the cleaner the residual fuel.

3.2.3 *compatibility, n*—of residual fuel, the absence of suspended solids when equal volumes of a sample and a blend stock are mixed together.

## 4. Summary of Test Method

4.1 *Cleanliness Procedures, Manual and Automated*—A drop of the preheated and thoroughly mixed sample is put on a test paper and placed in an oven at 100 °C. After 1 h, the test paper is removed from the oven and the resultant spot is examined for evidence of suspended solids and rated for cleanliness using the D4740 Adjunct Reference Spot Sheet.

4.2 *Compatibility Procedures, Manual and Automated*—A blend composed of equal volumes of the sample fuel oil and the blend stock is tested in the same way as described in 4.1 and rated for compatibility against D4740 Adjunct Reference Spot Sheet.

## 5. Significance and Use

5.1 The four procedures in this test method are used alone or in combination to identify fuels or blends that could result in excessive centrifuge loading, strainer plugging, tank sludge formation, or similar operating problems.

<sup>3</sup> Available from ASTM International Headquarters. Order Adjunct No. ADJD4740. Original adjunct produced in 2000.

\*A Summary of Changes section appears at the end of this standard

5.2 A spot rating of Number 3 or higher on a finished fuel oil by the cleanliness procedure indicates that the fuel contains excessive suspended solids and is likely to cause operating problems.

5.3 Although a fuel may test clean when subjected to the cleanliness procedures (manual and automated), suspended solids can precipitate when the fuel is mixed with a blend stock. Evidence of such incompatibility is indicated by a spot rating of Number 3 or higher in the compatibility procedures (manual and automated).

## 6. Apparatus

### 6.1 Manual Apparatus:

6.1.1 *Test Paper*—Chromatographic or filter paper cut or divided into appropriately sized squares, strips, or circles. Whatman #2 Qualitative filter paper<sup>4</sup> has been found to be suitable and was used in the interlaboratory round robin study. In case of dispute, use Whatman #2 Qualitative filter paper. Store the paper, without folding, rolling, or bending, in a tightly closed container.

6.1.2 *Test Paper Support*—Fig. 1 shows a suitable support for multiple samples spotted on the same test paper, which shall be supported in a horizontal position so nothing touches test areas of the paper.

<sup>4</sup> The sole source of supply of Whatman paper No. 2 known to the committee at this time is Whatman, Inc., 9 Bridewell Place, Clifton, NJ 07014. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

6.1.3 *Oven*—Any convection oven capable of maintaining an air temperature of  $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ .

6.1.4 *Reference Spot Sheet*—Adjunct for D4740.

6.1.5 *Conical Flask*, 100 mL capacity.

6.1.6 *Heating Bath or Hot Plate*, capable of heating sample to a temperature between  $90\text{ }^{\circ}\text{C}$  and  $95\text{ }^{\circ}\text{C}$ .

6.1.7 *Temperature Measuring Device*, capable of accurately measuring the temperature within the tolerances required in 6.1.3 and 6.1.6, such as ASTM 1C thermometer, or liquid-in-glass thermometers, thermocouples, or platinum-resistance thermometers that provide equivalent or better accuracy and precision may be used.

### 6.2 Automated Apparatus:

6.2.1 The automated instrument<sup>5</sup> (Fig. 2) shall include the following:

6.2.1.1 *Test Paper*—Whatman #2 Qualitative filter paper,<sup>4</sup> Grade 2, circles, diameter 50 mm.

6.2.1.2 *Vial*—Disposable, 15 mL capacity, proprietary designed for use in this apparatus.

6.2.2 *Micropipette*—Capable of delivering  $25\text{ }\mu\text{L} \pm 1\text{ }\mu\text{L}$  of sample. Positive displacement type micropipette with capillary piston is preferred for use. Air-displacement type micropipettes are not recommended for viscous samples.

6.2.3 *Oven*—Equipped with three stations, two wells maintained at  $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  for the sample preheating and one station maintained at  $93\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  for the filter paper preheating, proprietary designed for use in this apparatus.

6.2.4 *Digital Camera*—Color, 5 megapixels, connected to a computer to analyze and record the resultant spot.

6.2.5 *Filter Paper Displacement System*—To move the filter paper during the analysis phases, proprietary designed for use in this apparatus.

6.2.6 *Computer*—Associated to a database to analyze the resultant spot, capable to rate the spot according to the rating scale of the adjunct and store the result with the spot image.

## 7. Sampling

7.1 Samples for this test can come from tanks, lines, drums, or small containers. Use the applicable apparatus and techniques described in Practice D4057 or D4177.

## 8. Cleanliness Procedure

### 8.1 Manual Procedure:

8.1.1 Heat the sample in the original container in the heating bath or on a hot plate to above  $90\text{ }^{\circ}\text{C}$  to ensure the sample is in a fluid state.

8.1.2 Mix thoroughly.

NOTE 2—Mechanical shaking or mechanical mixing is recommended.

8.1.3 Pour approximately 50 mL of the sample into a 100 mL conical flask and place the flask in the heating bath or on a hot plate.

<sup>5</sup> The sole source of supply of the automated apparatus known to the committee at this time is AD Systems (www.adsystems-sa.com), Model ST 10 – Portable/on-board heavy fuel compatibility tester, available from AD Systems, P.A. Portes de la Suisse Normande, Allée de Cindais, 14320 Saint-André-sur-Orne, France. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

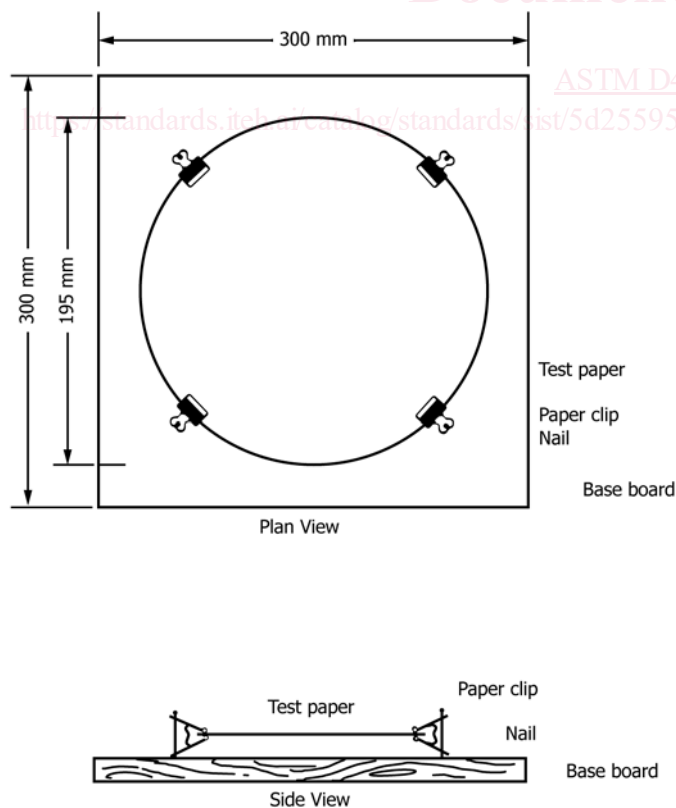


FIG. 1 Support for Test Paper

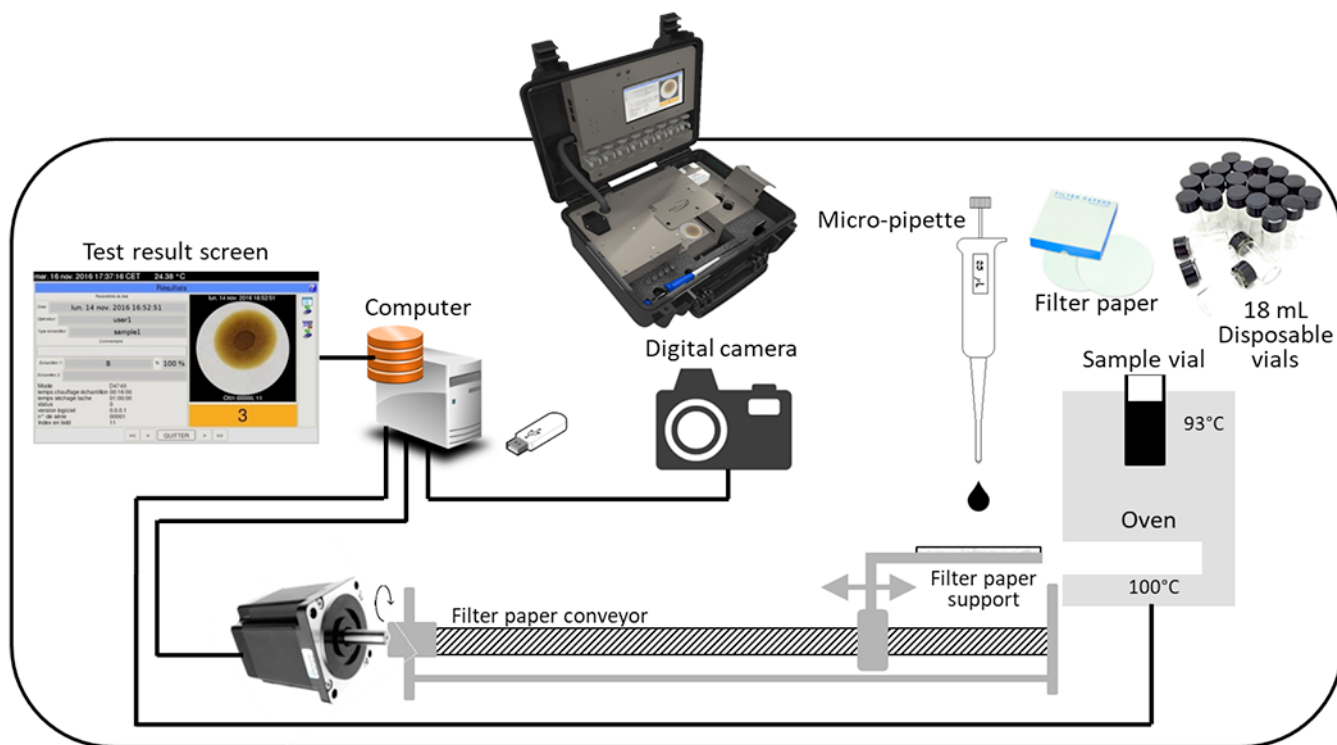


FIG. 2 Principle of the Automated Apparatus

8.1.4 Heat the sample uniformly by stirring and maintain the sample temperature between 90 °C and 95 °C for 15 min to 20 min.

8.1.5 Position the test paper so the test portion is clear of any support (Fig. 1), and place in the oven for a minimum of 5 min, allowing the paper temperature to attain the oven temperature.

8.1.6 Preheat the glass rod by dipping into and stirring the heated sample for about 20 s. Withdraw the rod and allow the first drop to fall back into the conical flask. Allow the second drop to fall onto the test paper which is supported horizontally in the oven heated to a temperature of 100 °C.

8.1.6.1 Multiple samples may be spotted on the same test paper. The spot centers shall not be closer than 50 mm and at least 25 mm from the edge of the test paper.

8.1.7 Dry the test paper in the oven maintained at 100 °C ± 2 °C for 1 h.

8.1.8 After 1 h, remove the test paper from the oven. Refer to the reference spot adjunct and to the reference spot descriptions given in Table 1. Compare the spot with the standard spots on the adjunct and record the spot number having the

closest resemblance. If the sample spot is rated between two consecutive spots on the adjunct, record the larger number.

NOTE 3—Ignore differences in overall darkness, color, size, and appearance of the outer edges.

### 8.2 Automated Procedure:

8.2.1 Prepare the apparatus according to the manufacturer’s instructions.

8.2.2 Heat the sample in the original container in the heating bath or on a hot plate to above 90 °C to ensure the sample is in a fluid state.

8.2.3 Mix thoroughly.

NOTE 4—Mechanical shaking or mechanical mixing is recommended.

8.2.4 Pour approximately 10 mL of the sample into a 15 mL disposable vial and place the vial in one of the two the oven wells.

8.2.5 Position a filter paper on the filter paper support.

8.2.6 Select the CLEANLINESS mode in the test mode menu of the apparatus.

8.2.7 Key in all requested information related to the test (sample number, operator name, etc.) and start the test procedure according to the manufacturer’s instructions. The sample is preheated at 93 °C for 15 min and the filter paper is positioned in the oven at 100 °C.

8.2.8 After 15 min, an audible alarm warns the operator and the filter paper is automatically moved to the sample pouring position.

8.2.8.1 With the micropipette, the operator samples 25 µL from the 15 mL vial previously positioned in the oven well and pours the 25 µL sample on the filter paper.

TABLE 1 Reference Spot Description

Reference Spot No.	Characterizing Features
1	Homogeneous spot (no inner ring)
2	Faint or poorly defined inner ring
3	Well-defined thin inner ring, only slightly darker than the background
4	Well-defined inner ring, thicker than the ring in reference spot No. 3 and somewhat darker than the background
5	Very dark solid or nearly solid area in the center. The central area is much darker than the background