



Designation: ~~D4740~~—19 D4740 – 20

Standard Test Method for Cleanliness and Compatibility of Residual Fuels by Spot Test¹

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 (ϵ) 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 mm²/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:²

- [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](#)
- [D4870 Test Method for Determination of Total Sediment in Residual Fuels](#)

2.2 ASTM Adjuncts:³

Reference Spot Sheet

3. Terminology

3.1 For definitions of terms used in this standard, see Terminology [D4175](#).

¹ 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.

Current edition approved May 1, 2019. Published June 2019. Originally approved in 1992. Last previous edition approved in 2014 as ~~D4740—04 (2014):D4740 – 19~~. DOI: ~~10.1520/D4740-19~~.10.1520/D4740-20.

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.

³ 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

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.

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 paper⁴ 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.

6.1.3 *Oven*—Any convection oven capable of maintaining an air temperature of 100 °C ± 2 °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 °C and 95 °C.

⁴ 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,¹ which you may attend.

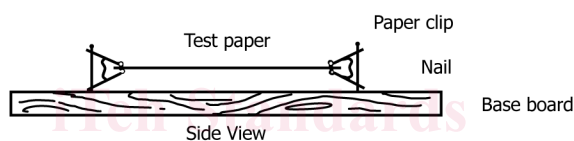
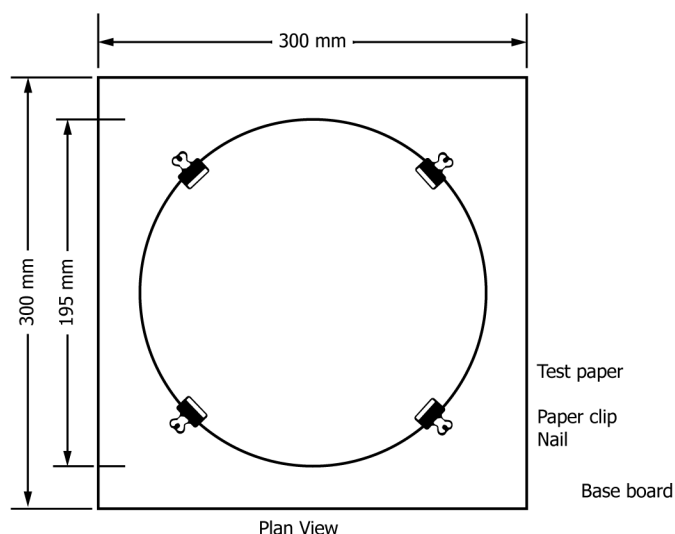


FIG. 1 Support for Test Paper

(<https://standards.iteh.ai>)
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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.

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6.2 Automated Apparatus:

6.2.1 The automated instrument⁵ (Fig. 2) shall include the following:

6.2.1.1 *Test Paper*—Whatman #2 Qualitative filter paper,⁴ Grade 2, circles, diameter 50 ± 5 mm.

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

6.2.2 *Micropipette*—Capable of delivering $25 \mu\text{L} \pm 1 \mu\text{L}$ 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 ± 2 °C for the sample preheating and one station maintained at 93 ± 2 °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.

⁵ 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,¹ which you may attend.

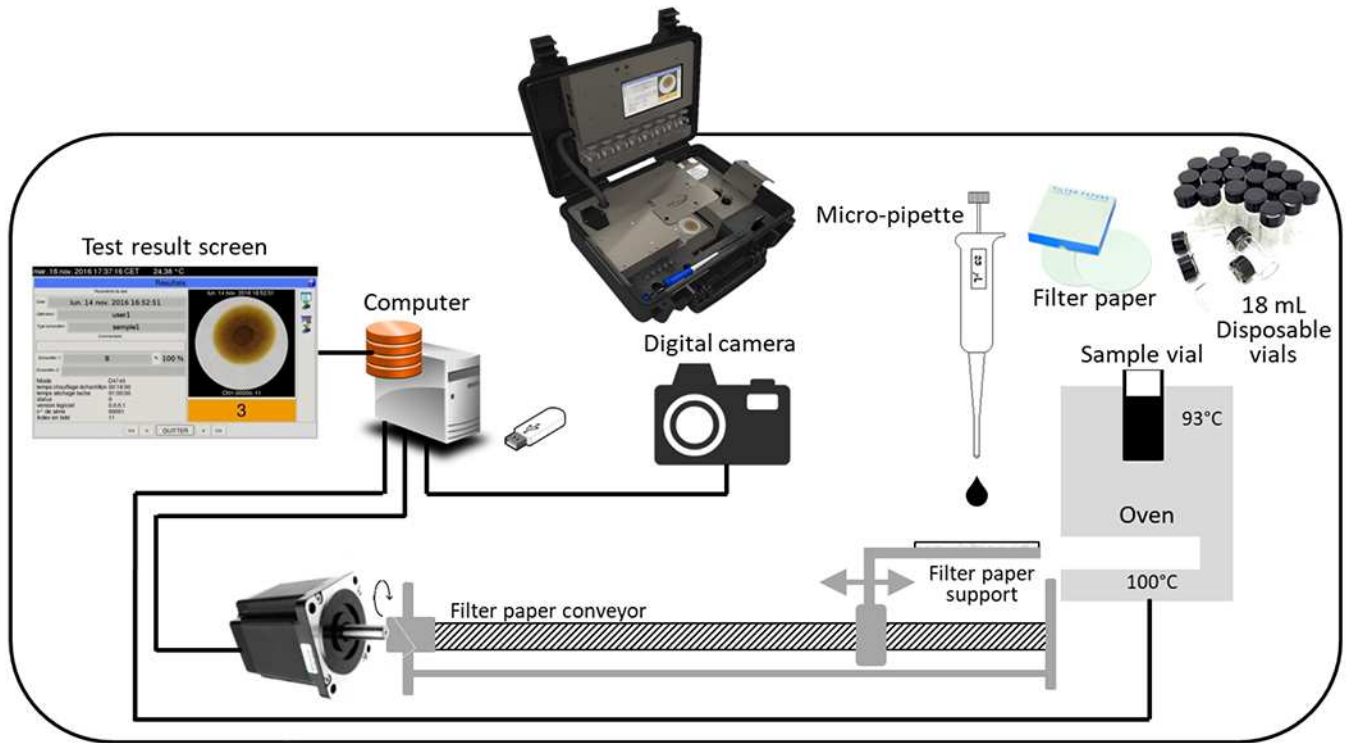


FIG. 2 Principle of the Automated Apparatus

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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

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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 °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.

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 $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ 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 $90\text{ }^{\circ}\text{C}$ 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 $93\text{ }^{\circ}\text{C}$ for 15 min and the filter paper is positioned in the oven at $100\text{ }^{\circ}\text{C}$.

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 $25\text{ }\mu\text{L}$ from the 15 mL vial previously positioned in the oven well and pours the $25\text{ }\mu\text{L}$ sample on the filter paper.

8.2.8.2 The operator instructs the instrument that the sample is dropped on the filter paper.

8.2.8.3 The filter paper is automatically moved back to the $100\text{ }^{\circ}\text{C}$ oven for 60 min. After 60 min, the spot is automatically moved under the camera to be photographed.

8.2.8.4 The dedicated software analyzes the spot picture and the rating is recorded, displayed (**Fig. 3**), and memorized in the database with the color picture of the spot.

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

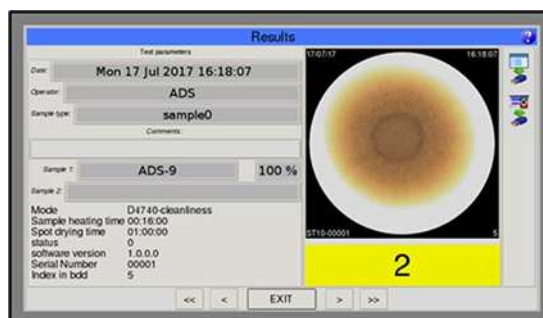


FIG. 3 Example of Cleanliness Test Result Obtained with the Automated Apparatus

9. Compatibility Procedure

9.1 Manual Procedure:

9.1.1 Pour 30 mL of test sample in an appropriate container, heating if necessary.

9.1.2 Add an equal volume of blend stock to the test sample and mix the contents thoroughly.

9.1.3 The equal volume of blend is generally the most severe condition for measuring compatibility. If in actual practice an application requires a blending ratio other than equal parts, conduct the test using the actual blend ratio for the test results to have a good probability of being indicative of the final blend.

9.1.4 Immediately test 50 mL of the mixture as described in 8.1.3 – 8.1.8.

9.2 Automated Procedure:

9.2.1 Prepare the apparatus according to the manufacturer's instructions.

9.2.2 Pour 30 mL of test sample in an appropriate container, heating if necessary.

9.2.3 Add an equal volume of blend stock to the test sample and mix the contents thoroughly.

9.2.4 The equal volume of blend is generally the most severe condition for measuring compatibility.

9.2.4.1 If in actual practice an application requires a blending ratio other than equal parts, conduct the test using the actual blend ratio for the test results to have a good probability of being indicative of the final blend.

9.2.5 Pour approximately 10 mL of the prepared blend into a 15 mL disposable vial and place the vial in one of the two the oven wells.

9.2.6 Position a filter paper on the filter paper support.

9.2.7 Select the COMPATIBILITY mode in the test mode menu of the apparatus and indicate the percentage of each fuel to be tested.

9.2.8 Proceed as described in 8.2.7 and 8.2.8.

10. Report

10.1 Manual Procedure:

10.1.1 Report the cleanliness or the compatibility rating as an integer from 1 to 5, as determined in 8.1.8. Report the blend ratio when other than 50:50 is used to determine the capability rating.

10.2 Automated Procedure:

10.2.1 Report the cleanliness or the compatibility rating (Fig. 4) recorded in 8.2.8. Report the blend ratio when other than 50:50 is used to determine the compatibility rating.

10.3 Report if the rating has been obtained by manual or automated procedure.

11. Precision and Bias

11.1 *Manual Procedure:*

11.1.1 Precision and bias statements have not been determined for the preparation of the spots; however, a precision statement regarding how different individuals rate spots was determined by a modified round robin test. The need for statements pertaining to the preparation of the spots is being considered.

11.1.1.1 The precision of the manual procedure in Test Method D4740, for different individuals using the same D4740 Adjunct Reference Spots in conjunction with Table 1 to rate the same spots prepared in accordance with Test Method D4740 from field samples, as determined at a common site is as follows:

11.1.1.2 *Repeatability*—The difference between successive ratings of the same spot by the same individual viewed under same conditions would, in the long run, not exceed one rating number only in one case in 20.

11.1.1.3 *Reproducibility*—The difference between two single independent ratings obtained by different individuals for the same spot viewed under the same conditions would, in the long run, not exceed one rating number only in one case in 20.

11.1.2 *Bias*—Since there is no accepted reference material suitable for preparing the spots used in the spot rating procedures in Test Method D4740, bias has not been determined.

11.2 *Automated Procedure:*

11.2.1 Final precision has not been determined; however, a ruggedness study has been completed by a single technician at a single laboratory, using five different apparatus, and 21 different samples (see Appendix X1). For 87 % of 105 observations, manual and automated method results agreed. A full ILS will be completed within five years after approval of Test Method D4740 revision to include the automated procedure.

11.2.2 *Bias*—Since there is no accepted reference material suitable for preparing the spots used in the spot rating procedures in Test Method D4740, bias has not been determined.

11.3 *Referee Method:*

11.3.1 In case of dispute, the manual method using the Adjunct Reference Spots in conjunction with Table 1 will be the referee method. The photo recorded by the automated procedure can be used to rate the spot manually.

12. Keywords

12.1 cleanliness; compatibility; residual fuel; spot test

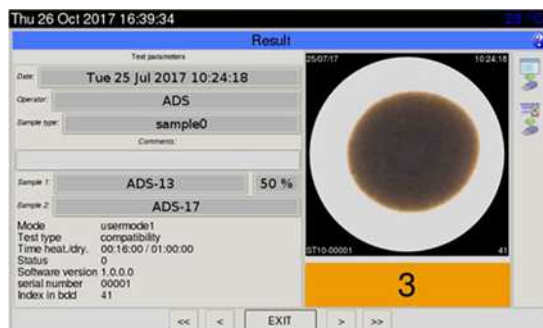


FIG. 4 Example of Compatibility Test Result Obtained with the Automated Apparatus