



Designation: **D7061—06 D7061 – 12**

Standard Test Method for Measuring n-Heptane Induced Phase Separation of Asphaltene-Containing Heavy Fuel Oils as Separability Number by an Optical Scanning Device¹

This standard is issued under the fixed designation D7061; 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 the quantitative measurement, either in the laboratory or in the field, of how easily asphaltene-containing heavy fuel oils diluted in toluene phase separate upon addition of heptane. This is measured as a separability number (%) by the use of an optical scanning device.

1.2 The test method is limited to asphaltene-containing heavy fuel oils. ASTM specification fuels that generally fall within the scope of this test method are Specification **D396**, Grade Nos. 4, 5, and 6, Specification **D975**, Grade No. 4-D, and Specification **D2880**, Grade Nos. 3-GT and 4-GT. Refinery fractions from which such blended fuels are made also fall within the scope of this test method.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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:*²

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel Oils

D2880 Specification for Gas Turbine Fuel Oils

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

3. Terminology

3.1 *Definitions:*

3.1.1 ~~*asphaltene, asphaltenes, n*—(rarely used in the singular), in petroleum technology, a molecule of high molecular mass, high carbon/hydrogen ratio, and containing heteroatoms represent an oil fraction that is soluble in a specified aromatic solvent but separates upon addition of an excess of a specified paraffinic solvent.~~

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.

3.1.1.1 *Discussion*—

~~Asphaltenes are found largely in crude oils and in heavy fuel oils containing residual fractions. They are insoluble in alkanes, such as *n*-heptane and *n*-octane, but soluble in aromatic solvents, such as benzene, toluene, and 1-methylnaphthalene. aromatic solvent is toluene and the paraffinic solvent is heptane.~~

3.1.2 *compatibility, n*—of crude oils or of heavy fuel oils, the ability of two or more crude oils or fuel oils to blend together within certain concentration ranges without evidence of separation, such as the formation of multiple phases.

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

3.1.2.1 Discussion—

Incompatible heavy fuel oils or crude oils, when mixed or blended, result in the flocculation or precipitation of asphaltenes. Some oils may be compatible within certain concentration ranges in specific mixtures, but incompatible outside those ranges.

3.1.3 *flocculation, n—of asphaltenes from crude oils or heavy fuel oils*, the aggregation of colloiddally dispersed asphaltenes into visibly larger masses that may or may not settle.

3.1.4 *peptization, n— of asphaltenes in crude oils or heavy fuel oils*, the dispersion of asphaltenes to produce a colloidal dispersion.

3.1.5 *stability reserve, n—in petroleum technology*, the property of an oil to maintain asphaltenes in a peptized state and prevent flocculation of the asphaltenes.

3.1.5.1 Discussion—

An oil with a low stability reserve is likely to undergo flocculation of asphaltenes when stressed (for example, extended heated storage) or blended with a range of other oils. Two oils each with a high stability reserve are likely to maintain asphaltenes in a peptized state and not lead to flocculation when blended together.

3.1.6 *transmittance, n—of light*, the fraction of the incident light of a given wavelength that is not reflected or absorbed, but passes through a substance.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *Heavy Fuel v. 1.0³, n*—the name of a proprietary computer program designed to allow automatic control of test and calculations of the results in Test Method D7061.

3.2.2 *separability number, n—in petroleum technology*, the standard deviation of the average transmittance, determined in this test method, expressed as a percentage figure.

³ The sole source of supply of the optical scanning device (Turbiscan MA2000 or Turbiscan Heavy Fuel), and corresponding software (Turbisoft or Heavy Fuel v. 1.0), known to the committee at this time is available from Formulation, 10 Impasse Borde Basse, 31240 l'Union, France. This device has been found satisfactory for the purpose of this test method. 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.

3.2.2.1 Discussion—

The separability number estimates the stability reserve of the oil, where a high separability number indicates that the oil has a low stability reserve and a low separability number that the oil has a high stability reserve.

3.2.3 *Turbisoft³, n*—the name of a proprietary computer spreadsheet program, designed to allow automatic calculation of the results in Test Method D7061.

4. Summary of Test Method

4.1 Dilution of oil with toluene followed by addition of heptane causes asphaltenes to flocculate, and the oil to phase separate. The rate of the phase separation is determined by measuring the increase in transmittance in the sample from the bottom of a test tube to the top (or a portion thereof) over time. The standard deviation of the average transmittance from a number of scans gives a separability number (%).

4.2 The oil is first diluted with toluene in ratios that depend on the oil type ([Annex A1](#)). Mix 2 mL of the oil/toluene solution with 23 mL of heptane. Transfer 7 mL of the oil/toluene/heptane mixture into a glass vial that is inserted into an optical scanning device.

4.3 The change in light transmittance through the glass vial containing the oil/toluene/heptane mixture is recorded by scanning the vial vertically with the optical scanning device. One scan is run every 60 s for 15 min. An average of the transmittance is calculated from 1125 readings at 0.04-mm intervals along the glass vial, starting 10 mm above the bottom of the vial and continuing up to 55 mm for each scan. The separability number from 16 scans is calculated and reported.

5. Significance and Use

5.1 This procedure describes a rapid and sensitive method for estimating the stability reserve of an oil. The stability reserve is estimated in terms of a separability number, where a low value of the separability number indicates that there is a stability reserve within the oil. When the separability number is between 0 to 5, the oil can be considered to have a high stability reserve and asphaltenes are not likely to flocculate. If the separability number is between 5 to 10, the stability reserve in the oil will be much lower. However, asphaltenes are, in this case, not likely to flocculate as long as the oil is not exposed to any worse conditions, such

as storing, aging, and heating. If the separability number is above 10, the stability reserve of the oil is very low and asphaltenes will easily flocculate, or have already started to flocculate.

5.2 This test method can be used by refiners and users of oils, for which this test method is applicable, to estimate the stability reserves of their oils. Hence, this test method can be used by refineries to control and optimize their refinery processes. Consumers of oils can use this test method to estimate the stability reserve of their oils before, during, and after storage.

5.3 This test method is not intended for predicting whether oils are compatible before mixing, but can be used for determining the separability number of already blended oils. However, oils that show a low separability number are more likely to be compatible with other oils than are oils with high separability numbers.

6. Apparatus

6.1 *PC-based Computer*, into which the software³ that controls the apparatus can be loaded. Data is acquired and accumulated on the hard disk in the computer.

6.2 *Optical Scanning Device*—The apparatus,³ which should be suitably calibrated to the manufacturer's instructions, consists of a reading head, composed of a pulsed infrared light source that uses a wavelength of 850 nm. A detector is situated opposite from the light source and reads the transmittance through the glass vial containing the specimen. During a scan, the reading head moves up and down along the glass vial and scans the whole vial going up. The transmittance is automatically measured every 0.04 mm. During one measurement, the time interval between each scan shall be 60 s and 16 scans shall be run. The measuring principle is schematically shown in Fig. 1. The measured transmittance along the glass vial is reported every 0.04 mm and is automatically stored on the hard disk in the computer and can be further processed as described in Section 10 and Annex A2 and Annex A3.

6.3 *Cylindrical Clear Glass Vial with Screw Cap*, 15 mL, 16 mm outside diameter, 12 mm inner diameter, and 140 mm high, and of high optical purity shall be used as a sample container. Use once and then discard.

6.4 *Pipette, Graduated or Automatic*, 5 and 10 mL.

6.5 *Graduated Cylinder*, 25 mL.

6.6 *Clear Glass Bottle with Cap*, 250 mL.

6.7 *Clear Glass Bottle with Cap*, 50 mL.

6.8 *Magnetic Bar*, TFE-fluorocarbon-coated.

6.9 *Magnetic Stirrer*.

6.10 *Balance*, precision ± 0.01 g.

6.11 *Stopwatch*, capable of 0.1 s.

6.12 *Burette*, 25 mL, Grade A.

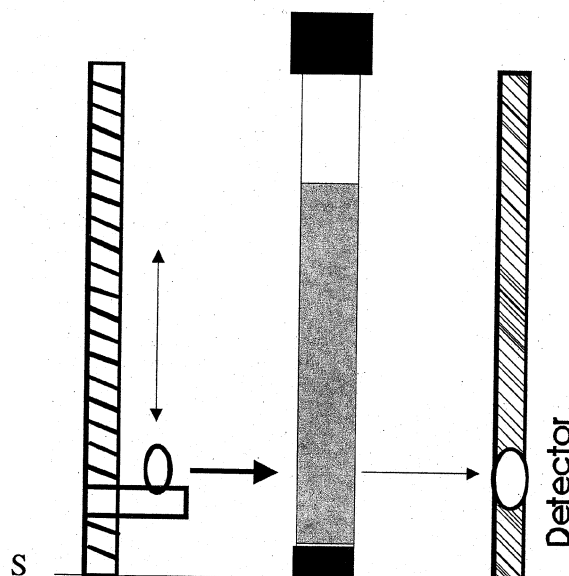


FIG. 1 Schematic Representation of a Typical Measurement Using an Optical Scanning Device

6.13 *Oven*, capable of 60°C at ±0.1°C.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagents of technical grade (95–99% purity) and higher are adequate for this test.

7.2 *Heptane*. (**Warning**—Flammable. Vapor harmful. Vapor may cause flash fire.)

7.3 *Toluene*. (**Warning**—Flammable. Vapor harmful. Vapor may cause flash fire.)

8. Sampling and Test Specimens

8.1 The oil sample drawn for the purpose of this test method shall be representative of the lot of oil. Obtain the sample in accordance with the procedures of Practice [D4057](#) or [D4177](#), if possible.

8.2 When working with the oil sample in the laboratory, the oil shall be stirred either manually or mechanically until the mixture is homogenous and representative for the whole sample before withdrawing oil for testing.

8.3 When working with solid or highly viscous oils, the oil may be heated (for example, on a heating plate, in an oven, or, if a drum is heated, by an electrical heating belt or steam shed) to obtain a lower viscosity prior to weighing and mixing. It is then important that the whole sample is fluid to ensure a homogenous mixture and that the sample withdrawn is representative of the whole sample.

9. Procedure

9.1 Prepare the instrument for measuring by turning it on at least 1 h before use, and make preparation so that one scan can be run automatically every 60 s for 15 min. For more detailed instructions, see [Annex A2](#) and [Annex A3](#).

9.2 Sample to be placed in an oven at 60°C for 24 h to ensure homogeneity.

9.3 Remove the sample from the oven and shake for 1 min to ensure mixing.

9.4 With the aim to increase and achieve a comparable transmittance for all types of oils, weigh in 15.5 ± 0.01 g of the oil sample on a balance. Record the weight of oil added to four decimal places and dilute with toluene, in a weight ratio in accordance with from 1:3 to 1:20 to have a baseline under 15% of transmittance, for guidance see [Annex A1](#), in a bottle with cap (6.6), and shake the bottle well. Add a magnetic bar to the oil-toluene solution ensuring none of the oil/toluene mixture is lost, and seal the bottle. Shake the jar well until the oil/toluene mixture no longer adheres to the bottom of the jar. Put the bottle on a magnetic stirrer and stir the mixture for a minimum of 1 h but not more than 3 h. 90 ± 5 min.

9.2 Prepare the instrument for measuring by turning it on and make preparations so that one scan can be run automatically every 60 s for 15 min. For more detailed instructions, see [Annex A2](#) and [Annex A3](#).

9.5 Using a graduated cylinder, transfer 25 mL burette (6.12), add 23 mL of *n*-heptane into a glass bottle (6.7). Use a pipette to add 22.0 mL of the oil/toluene mixture prepared in 9.4 to the heptane and shake the mixture briskly for 6 s.

9.6 Use a pipette to transfer 7 mL of the oil-toluene-heptane mixture immediately in a period of less than 10 s into the cylindrical clear glass vial and screw the cap on (6.3), taking care not to deposit material on the tube walls. If 10 s is exceeded, the rest should be stopped and the sample retested.

9.7 Immediately place the cylindrical glass vial, with cap, in the instrument at ambient temperature (20 to 25°C) and start to measure at once.

NOTE 1—The transmittance through the cylindrical glass vial is now recorded every 60 s for 15 min and stored by the software on the hard disk of the computer.

9.8 When the measurement is finished, remove the glass vial from the optical scanning device and clean the glass vial with toluene in a fume hood.

10. Calculation and Interpretation of Results

10.1 *Calculation of Results*:

10.1.1 The following calculations may be completed either manually, by using a spreadsheet program (described in [Annex A2](#)) or automatically, using the software Heavy Fuel v. 1.0 (described in [Annex A3](#)).

10.1.2 Analyze the transmittance between 10 to 55 mm (10 mm from the bottom of the glass vial), that is, calculate the average transmittance (X_i) recorded in this region for each minute.

10.1.3 Calculate the total average transmittance (X_T) of each of the 16 scans.

10.1.4 Calculate the separability number using the equation for standard deviation:

$$\text{Separability number} = \sqrt{\frac{\sum_{i=1}^n (X_i - X_T)^2}{n - 1}} \quad (1)$$

where:

X_i = average transmittance for each 60 s,

X_T = average of X_i

$$\left(X_T = \frac{X_1 + X_2 \dots + X_{16}}{16} \right)$$

and

n = the set of replicate measurements (16 in the method).

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10.2 Interpretation of Results:

10.2.1 The separability number is a rate-related factor that gives a measure of how easily an oil phase separates upon addition of heptane. Phase separation is due to asphaltene flocculation and sedimentation. As asphaltenes fall out of solution, the transmittance through the sample increases. There will be a rapid change in transmittance if this process is quick, resulting in a high separability number. A high number shows that the stability reserve of the oil is poor, while a low number shows that there is a stability reserve in the oil. The separability number is presented in percent transmittance.

11. Report

11.1 Report the following information:

11.1.1 The toluene dilution ratio (in accordance with [Annex A1](#)).

11.1.2 The separability number of the oil sample as the deviation in percent transmittance to the nearest 0.1 %.

12. Precision and Bias⁴

12.1 *Precision*—~~The repeatability standard deviation obtained by the same operator with the same apparatus under constant operating conditions is presented below:~~ precision of this test method was determined in an interlaboratory study (ILS) conducted in 2011.

Mean of Separability Number (%)	Standard Deviation (%)	95%-Confidence Interval (%)
0.3	0.02	0.01
6.9	0.6	0.4
17.0	0.4	0.3

12.1.1 *Repeatability*—~~The data displayed above were obtained for three fuel oils (Grade No. 6) and ten measurements were made for each oil. The toluene dilution ratio was 1:9; difference between successive test results 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 this test method, exceed the following, only in one case in twenty:~~

$$\text{Separability Number Repeatability} = 1.3485(X+0.05)^{0.5} Y \quad (2)$$

where:

\bar{X} = mean of replicate analyses, and

\bar{Y} = separation number.

Applicable Nominal Range = 0.1 to 16.

12.1.2 *Reproducibility*—~~The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and the correct operation of the test method, exceed the following, only in one case in twenty:~~

$$\text{Separability Number Reproducibility} = 2.68(X+0.05)^{0.5} Y \quad (3)$$

where:

\bar{X} = mean of replicate analyses, and

\bar{Y} = separation number.

Applicable Nominal Range = 0.1 to 16.

12.1.3 Example of Precision Calculation:

Mean	R	r
0.16	1.1	0.6
1.99	3.6	1.9
5.48	5.9	3.2
6.14	6.2	3.4
7.86	7.0	3.8
9.33	7.6	4.1
9.54	7.7	4.2
10.12	8.0	4.3
10.79	8.2	4.4
15.73	9.9	5.4

12.2 *Reproducibility*—A round robin test is being developed and the reproducibility of this test method will be determined and available within five years.

12.2 *Bias*—Today there is no accepted reference material suitable for testing the stability reserve of oils (in this test method estimated as a separability number) and bias has so far been impossible to determine.

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1748. Contact ASTM Customer Service at service@astm.org.