



# Standard Test Method for Measuring n-Heptane Induced Phase Separation of Asphaltene-Containing Heavy Fuel Oils as Separability Number by an Optical Scanning Device<sup>1</sup>

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 reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 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:<sup>2</sup>

[D396](#) Specification for Fuel Oils

[D975](#) Specification for Diesel Fuel Oils

[D2880](#) Specification for Gas Turbine Fuel Oils

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[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, n*—in petroleum technology, a molecule of high molecular mass, high carbon/hydrogen ratio, and containing heteroatoms.

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 cetane, but soluble in aromatic solvents, such as benzene, toluene, and 1-methylnaphthalene.

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.

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

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

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. 1.0<sup>3</sup>, 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.

3.2.2.1 *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 *Turbisoft<sup>3</sup>, 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 software<sup>3</sup> 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<sup>3</sup> 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.

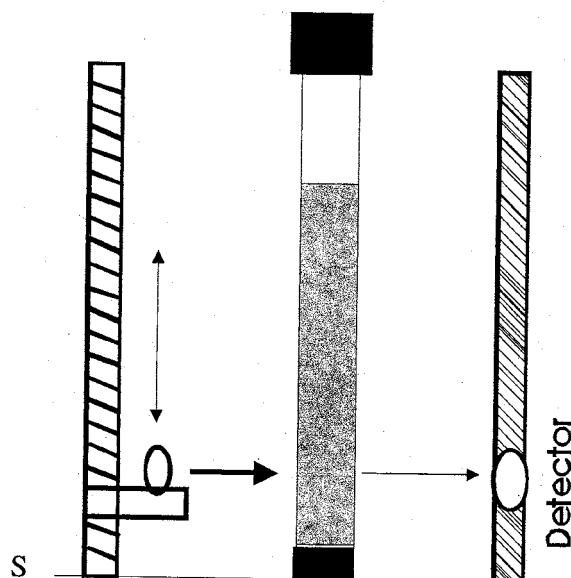


FIG. 1 Schematic Representation of a Typical Measurement Using an Optical Scanning Device

<sup>3</sup> 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,<sup>1</sup> which you may attend.

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, shall be used as a sample container.

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  $\pm 0.01$  g.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Reagents of technical grade (95 % 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 With the aim to increase and achieve a comparable transmittance for all types of oils, weigh in 15 g of the oil sample on a balance and dilute with toluene, in a weight ratio in accordance with [Annex A1](#), in a bottle with cap (6.6), and shake the bottle well. Add a magnetic bar to the oil-toluene solution. Put the bottle on a magnetic stirrer and stir the mixture for a minimum of 1 h but not more than 3 h.

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](#).

9.3 Using a graduated cylinder, transfer 23 mL of *n*-heptane into a glass bottle (6.7). Use a pipette to add 2 mL of the oil/toluene mixture prepared in 9.1 to the heptane and shake the mixture briskly for 6 s.

9.4 Use a pipette to transfer 7 mL of the oil-toluene-heptane mixture immediately into the cylindrical clear glass vial and screw the cap on (6.3).

9.5 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.

9.6 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  $X_i$  ( $X_T = X_1 + X_2 \dots + X_{16} / 16$ ), and

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

### 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 Bias

12.1 *Precision*—The repeatability standard deviation obtained by the same operator with the same apparatus under constant operating conditions is presented below:

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 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.

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.

12.3 *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.

### 13. Keywords

13.1 compatibility; heavy fuels oils; phase separation; separability number; stability reserve; transmittance

## ANNEXES

### (Mandatory Information)

#### A1. OIL:TOLUENE DILUTION RATIOS

**TABLE A1.1 Dilution Ratios (in Weight Ratio) of Oil with Toluene**

Standard Specification for Fuel Oils	Oil:Toluene Ratio (Weight)
<b>D396</b>	
Grade No. 4 <sup>A</sup>	1:3
Grade No. 5	1:6
Grade No. 6	1:9
<b>D975</b>	
Grade No. 4D	1:9
<b>D2880</b>	
Grade No. 3-GT <sup>A</sup>	1:6
Grade No. 4-GT <sup>A</sup>	1:9
Refinery Fractions	
Straight run fuels	1:6
Residue from visbreaker	1:9
Unknown Oil Grade, Refinery Fraction or Blended Oil Samples	1:9

<sup>A</sup> In the presence of asphaltenes in the fuel.

#### A2. MANUAL CALCULATION OF SEPARABILITY NUMBER WHEN USING THE TURBISOFT SOFTWARE

A2.1 To prepare the measurement (9.2), open the software Turbisoft.

A2.1.1 Choose “Acquisition” and “Automatic Acquisitions.”

A2.1.2 A box will appear. Choose 16 for “Number of Acquisitions” and 1 min for “Delay between Acquisitions.”

A2.1.3 Prepare the sample (according to 9.3 and 9.4).

A2.1.4 After the test vial has been inserted in the instrument (9.5), click “Start” immediately.

A2.2 The separability number can be calculated by importing the raw data from Turbisoft to a spreadsheet program and by using pre-programmed functions in that program. This is

done by first choosing “View” then “Transmission.”

A2.2.1 Choose “Edit.”

A2.2.2 Choose “Copy.”

A2.2.3 Open a spreadsheet program, and choose “Paste All.”

A2.2.4 The first column will show the actual number of the measurement. Multiply this column with 0.04 (since one measurement is done every 0.04 mm) to obtain at which height the transmittance was measured.

A2.2.5 Multiply all other values with 100 to get the transmittance in percentage.

	A	B	C	..	Q	
	<b>Height (mm)</b>	<b>Transmittance scan 1 (time=0 min) (%)</b>	<b>Transmittance scan 2 (time=1 min) (%)</b>		<b>Transmittance scan 16 (time=15 min) (%)</b>	
<b>1</b>	10.00	21			89	
<b>2</b>	10.04	19			96	
<b>3</b>	10.08	20			..	
<b>..</b>	..	..			..	
<b>1125</b>	55.00	22			93	
<b>1126</b>		AVERAGE (B1:B1125) (average of all measured values during scan 1)	AVERAGE (C1:C1125) (average of all measured values during scan 2)	..	AVERAGE (Q1:Q1125) (average of all measured values during scan 16)	AVERAGE (B1126:Q1126)
						STDEV (B1126:Q1126)

FIG. A2.1 Illustration of How to Calculate the Separability Number (in a Spreadsheet Program)

A2.3 To calculate the separability number, start by calculating the average of the transmittances measured along the length of the tube from 10 to 55 mm for each scan. This gives ( $X_i$ ).

A2.3.1 Thereafter, calculate the standard deviation of these averages giving the separability number ( $X_T$ ).  $X_T$  can also be calculated using a pre-programmed function for average.

A2.3.2 The abbreviations used in Fig. A2.1, for average (AVERAGE) and standard deviation (STDEV), are the ones used by Microsoft Excel.<sup>4</sup> For other spreadsheet programs, pre-programmed functions may have other names.

<sup>4</sup> Microsoft Excel is a registered trademark of Microsoft Corporation in the United States and other countries.



### A3. PROCEDURE FOR USING THE HEAVY FUEL 1.0 SOFTWARE TO AUTOMATICALLY MEASURE AND CALCULATE THE SEPARABILITY NUMBER

A3.1 To prepare the measurement (9.2), select a user name (see Note A3.1) in the software Heavy Fuel v. 1.0.

A3.1.1 Once the user is selected, the [START] button turns from gray to green, indicating that the software is ready to operate.

A3.1.2 Prepare the sample (in accordance with 9.3 and 9.4).

A3.1.3 Insert the glass vial in the instrument (9.5), and click immediately on the green [START] button.

NOTE A3.1—A new user is created via the user name database by clicking [OPTIONS] and [USER NAME], or directly on the icon [USER NAME]. Once the user name has been written in [NEW NAME], it is validated by clicking on [ADD]. The new name appears in the table and is validated by clicking [OK]. Once the name has been created, it appears automatically in the [USER] scrolling box.

A3.2 Over a 15-min time period, 16 scans are performed at

the frequency of one scan every minute. The time remaining is indicated on the software.

A3.3 The test is finished when the 16 scans have been performed. The separability number is automatically calculated as described in 10.1. The separability number is then displayed together with an estimate of the stability reserve of the oil (low, medium, or high).

A3.4 If more than one test is to be run, insert another sample, and choose [START] again. A report with all the results can then be printed by clicking on [FILE], [PRINT PREVIEW], and [PRINT]. The report includes the name of the file, the separability number, the stability reserve, the name of the user, the date and time of the experiment, potential comments, and the serial number of the instrument.

### SUMMARY OF CHANGES

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D7061–05a) that may impact the use of this standard.

(1) Included definitions of Turbisoft and Heavy Fuel software in 3.2.

(2) Changed Appendix X1 into Annex A2 and provided detailed descriptions of how to use the software to start a measurement.

(3) Added Annex A3 to introduce a new, fully automatic software and instructions for use.

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