Designation: ~~D7827 – 12~~ D7827 – 12 (Reapproved 2017)

Standard Test Method for Measuring n-Heptane Induced Phase Separation of Asphaltene from Heavy Fuel Oils as Separability Number by an Optical Device[1]

This standard is issued under the fixed designation D7827; 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 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. The result is a separability number (%). See also Test Method D7061.

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

1.5 *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]

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
D7061 Test Method for Measuring n-Heptane Induced Phase Separation of Asphaltene-Containing Heavy Fuel Oils as Separability Number by an Optical Scanning Device

3. Terminology

3.1 *Definitions:*

3.1.1 *asphaltenes* (rarely used in the singular),*n—in petroleum technology*, represent an oil fraction that is soluble in a specified aromatic solvent but separates upon addition of an excess of a specified paraffinic solvent.

3.1.1.1 *Discussion—*

In this test method, the aromatic solvent is toluene and the paraffinic solvent is heptane.

[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~~ Stability, Cleanliness and ~~Cleanliness~~Compatibility of Liquid Fuels.

Current edition approved ~~Nov. 1, 2012~~Dec. 1, 2017. Published ~~March 2013~~December 2017. Originally approved in 2012. Last previous edition approved in 2012 as D7827 – 12. DOI: ~~10.1520/D7827-12.~~10.1520/D7827-12R17.

[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.

3.1.2 *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 colloidally 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 *SEPView 6.1, n*—the name of a proprietary computer program designed to allow automatic control of test and calculations of the results in Test Method D7827.

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 *SEPCalc, n*—the name of a proprietary computer program modul of SEPView, designed to allow automatic calculation of the results in Test Method D7827.

3.2.4 *STEP-Technology, n*—parallel light (I_0) illuminates the entire sample cell and the transmitted light I is detected by multiple sensors with a µm-scale resolution arranged linearly from top to bottom. Transmission is recorded time- and space-resolved and may be converted into extinction by lg I/I_0.

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 consecutive automatic measurements gives a separability number (%).

4.2 The oil is diluted with toluene in ratios that depend on the oil type. Mix ~~2 mL~~ 2 mL of the oil/toluene solution with 23 mL of heptane. Transfer ~~3.5 mL~~ 3.5 mL of the oil/toluene/heptane mixture into a disposable optical cell that is inserted into an optical scanning device.

4.3 The change in light transmittance through the cell is recorded by proprietary STEP-Technology instantaneously over the entire sample height without scanning. Measurements are taken periodically every ~~10 s for 15 min.~~ 10 s for 15 min. An average of the transmittance is calculated from each reading of each of the 91 transmission profiles at each ~~0.007 mm~~ 0.007 mm distance along the optical cell, starting from the bottom of the cell and continuing up to ~~44 mm.~~ 44 mm. The separability number from multiple measurements 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 heavy 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, experience shows that oils exhibiting a low separability number are more likely to be compatible with other oils than are oils with high separability numbers.

6. Apparatus

6.1 *Computer executing software SEPView*[3], from a portable storage media or directly from the computer. SEPView controls the apparatus, acquires the data and accumulates it in a database on the portable storage media, the hard disk in the computer or at a server.

6.2 *Optical Device*—The apparatus[3] consists of an illumination system, composed of a pulsed infrared light source that uses a wavelength of ~~870 nm (± 10 nm)~~ 870 nm (± 10 nm) and means to parallelize and expand the light to illuminate the entire specimen height. A high-resolution line detector is situated opposite from the light source and reads the transmittance through the vertical midline of the optical cell (6.3) containing the specimen. The transmittance is automatically and instantaneously recorded at every pixel with a position resolution of ~~0.007 mm~~ 0.007 mm (STEP-Technology (trademarked)[3]). Time interval between each recording shall be ~~10 s.~~ 10 s. Total measurement time shall be ~~15 min.~~ 15 min. The measuring principle is schematically shown in Fig. 1. Each measured transmittance profile along the optical cell is automatically stored on the hard disk in the computer or at a server and can be further processed as described in Section 10 and Annex A2.

6.3 *Rectangular Transparent Disposable Optical Polyamid cells (PA-cells) with PP-stopper,* ~~5 mL~~ 5 mL capacity, cross-section ~~8 mm × 10 mm~~ 8 mm × 10 mm (optical path), wall thickness ~~1 mm and 80 mm~~ 1 mm and 80 mm high, shall be used as a sample container.

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

6.5 *Graduated Cylinder,* ~~25 mL.~~25 mL.

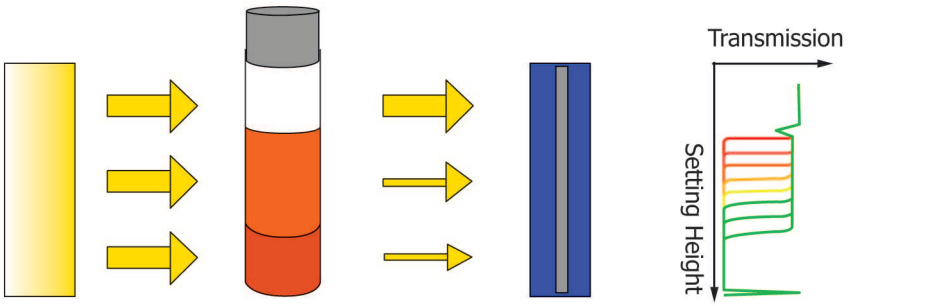
6.6 *Clear Glass Bottle with Cap,* ~~250 mL.~~250 mL.

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

6.8 *Magnetic Bar,* PTFE-coated.

6.9 *Magnetic Stirrer.*

6.10 *Balance,* precision ~~±0.01 g.~~±0.01 g.



NOTE 1—First measured profile after 1 s in red. Last profile after ~~15 min~~ 15 min in green.

FIG. 1 Representation of a Typical Measurement Scheme Using an Optical Device Based on STEP-Technology.

[3] The sole source of supply of the optical device (LUMiReader 413-1 (one channel) or LUMiReader 413-3 (three channel)), and corresponding software (SEPView 6), known to the committee at this time is available from LUM GmbH, Justus-von-Liebig-Str.3, 12489 Berlin, Germany.