Designation: D7060 – 20

Standard Test Method for Determination of the Maximum Flocculation Ratio and Peptizing Power in Residual and Heavy Fuel Oils (Optical Detection Method)[1]

This standard is issued under the fixed designation D7060; 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 a procedure for quantifying the maximum flocculation ratio of the asphaltenes in the oil and the peptizing power of the oil medium, by an automatic instrument using an optical device.

1.2 This test method is applicable to atmospheric or vacuum distillation residues, thermally cracked residue, intermediate and finished residual fuel oils, containing at least 1 % by mass asphaltenes. This test method has not been developed for asphalts.

NOTE 1—An optical probe detects the formation of flocculated asphaltenes. The start of flocculation is interpreted when a significant and sustained increase in rate-of-change of signal, as measured by the optical probe, ensures flocculation is in progress. The start of flocculation can be detected unambiguously when the sample contains at least 1 % mass asphaltenes as measured by Test Method D6560.

NOTE 2—This test method is applicable to products typical of Specification D396—Grades 5L, 5H, and 6, and Specification D2880—Grades 3-GT and 4-GT.

1.3 This test method was evaluated in an interlaboratory study in the nominal range of 32 to 76 for the maximum flocculation ratio and in the nominal range of 36 to 95 for peptizing power.

NOTE 3—The nominal range is determined by (min. sample mean—Reproducibility) to (max. sample mean + Reproducibility).

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

1.5 *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.*

1.6 *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
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
D4870 Test Method for Determination of Total Sediment in Residual Fuels
D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants
D6560 Test Method for Determination of Asphaltenes (Heptane Insolubles) in Crude Petroleum and Petroleum Products
D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories

3. Terminology

3.1 *Definitions:*

3.1.1 *asphaltenes, n*—(rarely used in the singular), *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 1-methylnapthalene, and the paraffinic solvent is n-hexadecane.

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

[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.

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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.

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

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 which 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— of crude oils, heavy fuel oils, and residual streams containing asphaltenes*, the property of an oil to maintain asphaltenes in a peptized (colloidally dispersed) 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.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *critical cetane dilution, n*—number of millilitres of cetane with which 1 g of undiluted sample can be diluted until it just does not flocculate the asphaltenes.

3.2.2 *critical dilution, n*—number of millilitres of 1-methylnaphthalene and cetane with which 1 g of undiluted sample can be diluted until it just does not flocculate the asphaltenes.

3.2.2.1 *Discussion*—The number of millilitres of 1-methylnaphthalene and cetane is variable and depends on the ratio of sample to 1-methylnaphthalene at the starting point and the sample type.

3.2.3 *flocculation ratio, n*—percentage by volume of 1-methylnaphtalene in a mixture of 1-methylnaphthalene and cetane.

3.2.4 *flocculation ratio at critical dilution, n*—percentage by volume of 1-methylnaphthalene in a mixture of 1-methylnaphthalene and cetane at the inflection point.

3.2.5 *inflection point, n*—last step during the titration with cetane, where flocculation of asphaltenes is *not* detected by the optical probe as a significant and sustained increase in rate-of-change of signal.

3.2.6 *maximum flocculation ratio, n—of asphaltenes*, minimum required solvency power, expressed as percentage by volume of 1-methylnaphthalene in a mixture of 1-methylnaphthalene and cetane, to keep the asphaltenes in a colloidal solution.

3.2.6.1 *Discussion*—Maximum flocculation ratio is the flocculation ratio at extrapolated infinite dilution of the sample.

3.2.7 *oil medium, n*—that portion of a sample of heavy fuel oil or crude oil that surrounds and colloidally disperses the asphaltenes.

3.2.7.1 *Discussion*—For purposes of this test method, an oil sample is considered to be composed of an oil medium (sometimes called an *oil matrix* or *maltenes*) and asphaltenes.

3.2.8 *peptizing power, n*—available solvency power, expressed as percentage by volume of 1-methylnaphthalene in a mixture of 1-methylnaphthalene and cetane, to keep asphaltenes in a colloidal solution.

3.2.9 *reciprocal dilution, n*—dilution ratio of sample in solvent mixture of 1-methylnaphthalene and cetane.

3.3 *Symbols:*

FR_{max} = maximum flocculation ratio
FR_x = flocculation ratio at critical dilution
Po = peptizing power
Xmin = critical cetane dilution
Xc = critical dilution

4. Summary of Test Method

4.1 Six portions of the sample are diluted in various ratios with 1-methylnaphthalene. Each solution is inserted into the automatic apparatus, and titrated with cetane until flocculation of asphaltenes is detected by the optical probe. The first two solutions are titrated with cetane in coarse determinations in which the flocculation ratio is decreased in 5 % steps. The coarse determinations help to establish suitable starting values for the fine determinations, in which the flocculation ratio is decreased in 1 % steps. The four flocculation ratios at critical dilution, measured during the fine determinations, are used to calculate the maximum flocculation ratio of the sample's asphaltenes and the peptizing power of the sample's oil medium.

5. Significance and Use

5.1 Asphaltenes are naturally occurring materials in crude petroleum and petroleum products containing residual material. The asphaltenes are usually present in colloidal suspensions, but they may agglomerate and flocculate if the suspension of asphaltene molecules is disturbed through excess stress or incompatibility. This test method provides compatibility parameters, which can be used to assess stability reserve and compatibility.

5.2 A blend is considered stable when the blend's peptizing power is higher than the blend's maximum flocculation ratio;[3,4] both of them can be calculated using empirical blend rules. Refineries and terminal owners can prevent the flocculation of asphaltenes due to incompatibility by assessing the compatibility of fuels beforehand.

NOTE 4—See Appendix X1 for an example of prediction of compatibility.

6. Interferences

6.1 High content of insoluble inorganic matter (sediment) has some interference in this test method. In this case, the insoluble matter shall be removed by filtration according to Test Method D4870.

[3] Berryman, T. J., and Lewis, C. P. G., "The Stability of Residual Fuels. Theory and Practice of the Shell Concept," 16th CIMAC Conference, Oslo, 1985.

[4] Berg van den, F. G. A., "Developments in Fuel Oil Blending," IASH 7th International Conference, Graz, Austria, 2000.