Designation: D7996 – 15

Standard Test Method for Measuring Visible Spectrum of Asphaltenes in Heavy Fuel Oils and Crude Oils by Spectroscopy in a Microfluidic Platform[1]

This standard is issued under the fixed designation D7996; 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 measurement, either in the laboratory or in the field, of visible spectra of asphaltenes and maltenes in gas oil, diesel fuel, fuel oils, residual fuel oils, lubricating oil, bitumen, and crude oil using microfluidics and spectrographic techniques.[2]

1.2 These measurements can be related quantitatively to the mass percent of asphaltenes present in the sample.

1.3 The test method is limited to asphaltene-containing oil with asphaltenes content less than 15 % by mass.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D3279 Test Method for *n*-Heptane Insolubles
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D6560 Test Method for Determination of Asphaltenes (Heptane Insolubles) in Crude Petroleum and Petroleum Products
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, see Terminology D4175.

3.1.2 *absorbance, (A), n*—the molecular property of a substance that determines its ability to take up radiant energy, expressed by:

$$A = \log_{10}(1/T) = -\log_{10}(T) \quad (1)$$

where T is the transmittance.

3.1.2.1 *Discussion*—Absorbance expresses the excess absorption over that of a specified reference or standard. It is implied that compensation has been affected for reflectance losses, solvent absorption losses, and refractive effects, if present, and that attenuation by scattering is small compared with attenuation by absorption.

3.1.3 *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.

3.1.3.1 *Discussion*—In this test method, the aromatic solvent is toluene and the paraffinic solvent is heptane.

3.1.4 *crude oil, n*—a naturally occurring hydrocarbon mixture, generally in a liquid state, which may also include compounds of sulfur, nitrogen, oxygen, metals, and other elements. (Synonym—crude petroleum, crude.)

3.1.5 *dilution factor (f), n*—the proportion of solvent increase made to reduce the concentration and thus the absorbance of a solute, expressed by the ratio of the volume of the diluted solution to the volume of original solution containing the same quantity of solute as the diluted solution.

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[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 and Cleanliness of Liquid Fuels.

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[2] This method and the apparatus are covered by US Patent 8,269,961 and Canadian Patent 2,800,879. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[3] 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.

3.1.6 *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.

3.1.7 *transmittance, (T), n*—the molecular property of a substance that determines its transportability of radiant power, expressed by:

$$T = P/P_o \tag{2}$$

where:

P = the radiant power passing through the sample, and
P_o = the radiant power incident upon the sample.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *concentration (c), n*—the quantity of absorbing substance in a solution in grams per litre.

3.2.2 *flow cell path length (b), n*—the distance in millimetres, measured in the direction of propagation of the beam of radiant energy, between the surface of the specimen on which the radiant energy is incident and the surface of the specimen from which it is emergent.

3.2.2.1 *Discussion*—This distance does not include the thickness of the cell in which the specimen is contained.

3.2.3 *maltenes, n*—the fraction of an oil after precipitation and subsequent removal of asphaltenes when mixed with a paraffinic solvent such as heptane.

3.2.4 *microfluidics, n*—the science and technology of systems that process or manipulate small amounts of fluids (10^{-9} to 10^{-18} L) using channels with dimensions of tens to hundreds of micrometres. [4]

3.3 *Abbreviations:*

3.3.1 *AU*—absorbance unit

3.3.2 *CV*—coefficient of variability

3.3.3 *PTFE*—polytetrafluoroethylene

4. Summary of Test Method

4.1 This test describes a method for measuring the visible spectrum of asphaltenes in crude oil and petroleum products, such as fuel oils, diesel fuel oils, gas turbine fuel oils, heavy fuel oils, residual fuel oils, and lubricating oils. The oil sample is mixed with heptane to induce precipitation of asphaltenes. The precipitated asphaltenes are removed using a porous filter. The change in visible spectrum of the sample before and after precipitation is due to the removal of asphaltenes. The visible spectrum of asphaltenes obtained using this technique is proportional to the concentration of the precipitated asphaltenes.

4.2 In the first step, the visible spectrum of the diluted sample is measured by mixing the sample with toluene at 1 + 40 v/v ratio (1 part sample in 40 parts solvent) using an automated metering system. The two streams of sample and toluene are mixed in a microfluidic mixer for effective mass transfer. The visible spectrum of the mixture is measured downstream of the membrane. The absorbance is recorded once a plateau is reached.

4.3 In the second step, the spectrum of the maltenes is measured. The second portion of the sample is mixed with heptane at 1 + 40 v/v ratio (1 part sample in 40 parts solvent). The asphaltenes precipitate as the sample comes in contact with heptane in the microfluidic mixer. As the mixtures passes through the membrane, the asphaltenes fraction is retained while the maltenes permeate through. The visible spectrum of the maltenes is measured downstream of the membrane.

4.4 The difference between the spectra of the diluted oil sample and maltenes are calculated and correlated to concentration of asphaltenes.

5. Significance and Use

5.1 This procedure describes a rapid and sensitive method for measuring the visible spectrum of asphaltenes in crude oils and petroleum products containing residual material using a microfluidic technique.

5.2 The method is sensitive to small changes in concentration of asphaltenes. Therefore, it can be used as a quality control and chain-of-custody check for fuel or crude oil samples.

5.3 The visible spectrum of asphaltenes obtained using this technique is correlated to the mass concentration of asphaltenes using conventional gravimetric techniques (see X1.3.3).

5.4 This test method can be used by refiners as well as upstream laboratories. The test is intended for samples with asphaltenes content less than 15 % by mass.

6. Apparatus[5]

6.1 *Spectrometer*—Equipped to handle liquid samples in a cell having optical path length of 3 mm and capable of measuring absorbance in the spectral region from 500 nm to 900 nm with a spectral resolution of 2 nm or less. Wavelength measurement shall be repeatable and known to be accurate within ±0.2 nm or less as measured by the argon emission line at 706.722 nm.

6.1.1 The performance of the spectrophotometer can be measured using Practice E275.

6.2 *Syringe Pumps*—Three syringe pumps with 4000 steps per centimeter. The accuracy and precision of the pumps have to be at least 0.10 % CV and 0.03 % CV respectively.

6.3 *Syringes*—5 mL syringe for heptane, 5 mL syringe for toluene, and a 250 µL syringe for toluene.

6.4 *Flow Cell*—3 mm optical path length.

6.4.1 A flow cell with minimal dead volume.

6.5 *Microfluidic Mixer*—Any type of microfluidic mixers may be used. However, since the asphaltenes precipitate in the mixer and eventually clog the mixer if run for too long, a multichannel design is preferred.

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[4] Whitesides, G. M. "The Origins and Future of Microfluidics," *Nature*, Vol. 442, No. 7101, 2006, p. 368–373.

[5] The sole source of supply of the microfluidic asphaltenes content apparatus known to the committee at this time is Schlumberger Canada Limited, 9450 17th Ave., Edmonton, AB, T6N 1M9, Canada, or 14910 Airline Rd., Rosharon, TX 77583, U.S., http://www.slb.com. 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.

6.6 *Microfluidic Membrane Filtration Unit*—200 nm PTFE membrane.

6.7 *Sample Loop*—200 µL stainless steel, PTFE, or chemically compatible sample loop.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagents of HPLC grade are required for this test.

7.2 *Heptane. CAS 142-82-5* (**Warning—**Flammable. Vapor harmful. Vapor may cause flash fire.)

7.3 *Toluene. CAS 108-88-33* (**Warning—**Flammable. Vapor harmful. Vapor may cause flash fire.)

8. Sampling and Test Specimens

8.1 Obtain the sample in accordance with the procedures of Practices D4057 or D4177.

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 an aliquot for testing.

8.3 It is recommended to heat the sample in a vial in an oven at 60 °C for no more than 60 min. The sample vial should then be agitated using a sonicator for 30 s prior to sub-sampling using a syringe.

8.3.1 Perform heating in a closed container such as a glass bottle to minimize oxidative degradation. Loosen the cap of the bottle to avoid building pressure in the container due to evaporation of light ends.

8.4 Use a 500 µL syringe to subsample from the bottle right after sonication of the sample. Inject the sample into the sample loop immediately after subsampling.

9. Procedure

9.1 First, load the sample into the 200 µL sample loop. Half of the sample will be used for spectroscopy of the diluted oil sample and the other half will be used for spectroscopy of the maltenes.

9.2 Prime the system and the flow cells with toluene and acquire light and dark reference spectra.

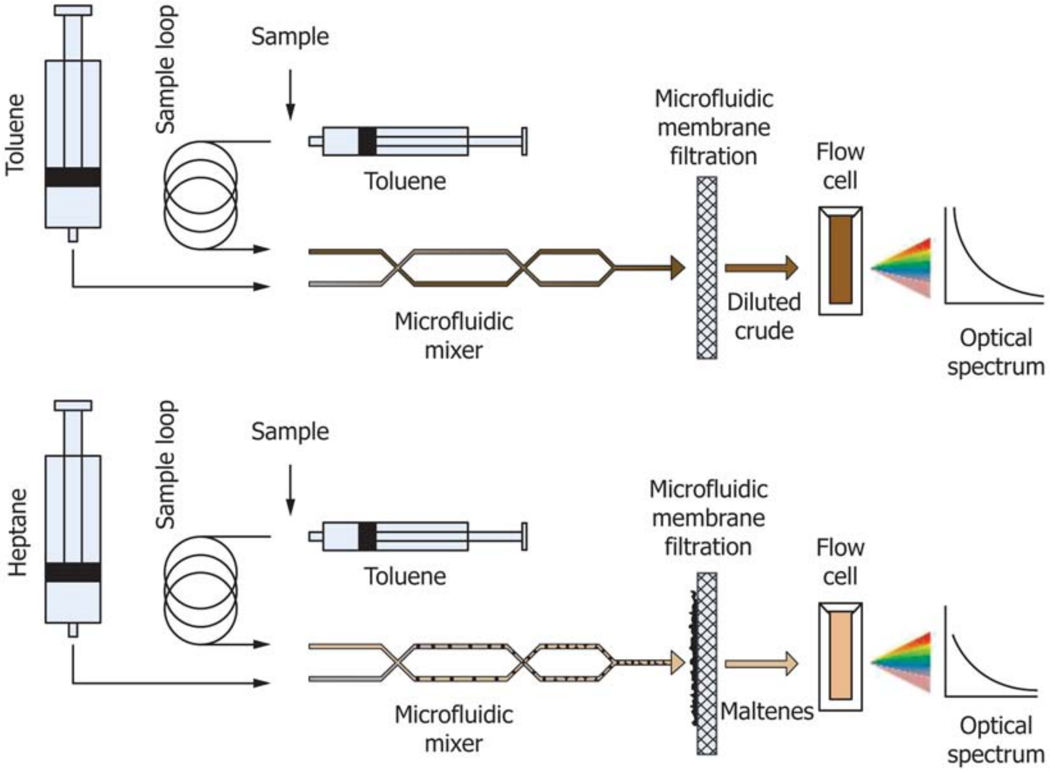
9.3 Check the integrity of the membrane and ensure there are no defects in the membrane. Any ruptures in the membrane could cause the asphaltenes pass through the membrane. The integrity of the membrane may be tested by monitoring the pressure differential across it when a known flow rate is injected into the system.

9.3.1 The temperature of the microfluidic section should be kept at 30 °C.

9.4 Dilute the test sample with toluene at 1 + 40 v/v (1 volume of crude oil sample in 40 volumes of solvent) using the microfluidic mixer. Half of the sample loop volume (100 µL) should be used for this step. The spectrum of the diluted oil is recorded as the fluid passes through the flow cell.

9.4.1 Maintain a flow rate of 10 µL/min for the sample and 400 µL/min for toluene.

9.4.2 Use a dilution ratio of 1 + 80 v/v if the absorbance signal saturates or exceeds the linear range of the spectrometer



NOTE 1—The schematic on the top shows the oil run and the one at the bottom shows that of the maltenes run.

FIG. 1 Schematic Diagram of the System

at 1 + 40 v/v. In such a case, ensure that the extra dilution is considered in the final calculations (see Eq 5 in 10.1.3.1).

9.4.3 Record the spectrum at a rate of at least once every second.

9.4.4 Plot the absorbance at 590 nm and 780 nm ($A_{oil_{(\lambda=590\,nm)}}$ and $A_{oil_{(\lambda=780\,nm)}}$, respectively) as a function of time. A graph such as Fig. 2(a) is expected for each wavelength. The measurement continues until the sample injection is complete.

9.4.5 At 590 nm, standard deviation of the plateau region should be ±0.02 AU or lower for an average absorbance of 1 or less and ±0.05 AU or lower for an average absorbance larger than 1. This criterion can be used to ensure that the heart of the plug (stable region) is used for analysis.

9.5 Prime the system with heptane and acquire light and dark reference spectra.

9.6 Check the integrity of the membrane according to 9.3.

9.7 Mix the second portion of the sample (100 µL) with heptane at 1 + 40 v/v ratio to precipitate asphaltenes. The spectrum of the maltenes is recorded as the fluid passes through the flow cell. Record the spectrum as a function of time.

9.7.1 A flow rate of 10 µL/min for the sample and 400 µL/min for heptane is recommended.

9.7.2 Record the spectrum at a rate of at least once every second.

9.7.3 Plot the absorbance at 590 nm and 780 nm ($A_{malt_{(\lambda=590\,nm)}}$ and $A_{malt_{(\lambda=780\,nm)}}$, respectively) as a function of time. A graph such as Fig. 2(b) is expected for each wavelength. Continue the measurement until the sample injection is complete.

9.8 When the measurement is complete, flush the system and microchips with toluene to remove the remaining oil and asphaltenes residues.

9.9 When the measurements are finished, remove the disposable membrane, and dispose of it in an environmentally safe way.

10. Calculation

10.1 *Calculation of Results:*

10.1.1 The following calculations may be run automatically or the data may be exported to another piece of software for manual analysis.

10.1.2 Time-averaged spectrum of the sample and maltenes are calculated using the following equation:

$$\bar{W} = \frac{1}{n}\sum_{t=t_1}^{t_2} W_t \tag{3}$$

where $\bar{W}$, W_t, and n represent time-averaged spectrum, spectrum at time t, and number of spectra from t_1 to t_2, respectively.

NOTE 1—In this test method, W represents the visible spectrum, whereas A_λ denotes the absorbance at a specific wavelength of λ.

10.1.3 The spectrum of asphaltenes is calculated using the following equation:

$$\bar{W}_{asp} = \bar{W}_{oil} - \bar{W}_{malt} \tag{4}$$

where $\bar{W}_{asp}$, $\bar{W}_{oil}$, and $\bar{W}_{malt}$ denote time-averaged spectrum of asphaltenes, oil, and maltenes, respectively. Fig. 3 shows a typical time averaged spectrum of crude oil, maltenes, and asphaltenes.[6-8]

10.1.3.1 If a volume ratio of 1 + 80 v/v is used for diluting the crude oil sample, then the following formula should be used:

$$\bar{W}_{oil} = \frac{81}{41}\times \bar{W}_{oil_{(1\,+\,80)}} \tag{5}$$

where $\bar{W}_{oil_{(1+80)}}$ is the time-averaged spectrum of oil sample at 1 + 80 v/v dilution.

10.1.4 Calculate:

$$\Delta OD_{asp_{(590\,nm\,-\,780\,nm)}} = \left[\bar{A}_{oil_{(\lambda\,=\,590\,nm)}} - \bar{A}_{oil_{(\lambda\,=\,780\,nm)}}\right] - \left[\bar{A}_{malt_{(\lambda\,=\,590\,nm)}} - \bar{A}_{malt_{(\lambda\,=\,780\,nm)}}\right] \tag{6}$$

where $\bar{A}_{oil_{(\lambda=590\,nm)}}$, $\bar{A}_{oil_{(\lambda=780\,nm)}}$, $\bar{A}_{malt_{(\lambda=590\,nm)}}$, $\bar{A}_{malt_{(\lambda=780nm)}}$, and $\Delta OD_{asp_{(590\,nm\,-\,780\,nm)}}$ represent the time-averaged absorbance of oil at 590 nm and 780 nm, time-averaged absorbance of maltenes at 590 nm and 780 nm, and finally the difference in absorbance of asphaltenes at 590 nm and 780 nm.

10.1.5 Fig. 3 shows an example of optical spectra for an oil sample and its maltenes and asphaltenes. The lines show the

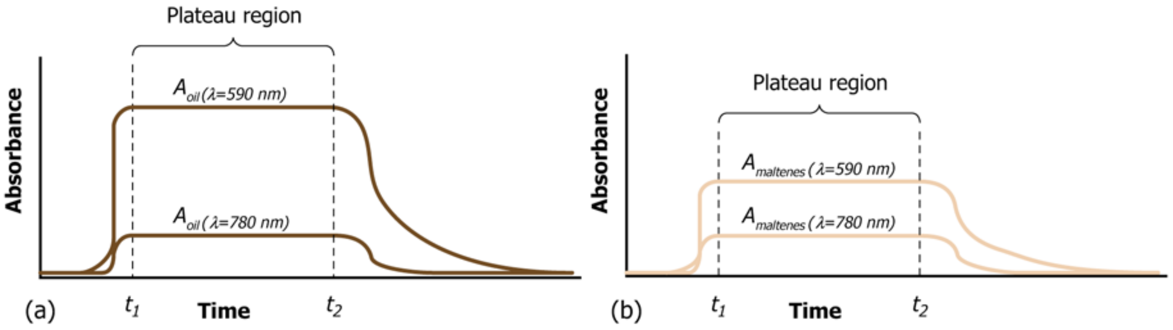


FIG. 2 Temporal Response of the System During (*a*) Oil Measurement and (*b*) Maltenes Measurements

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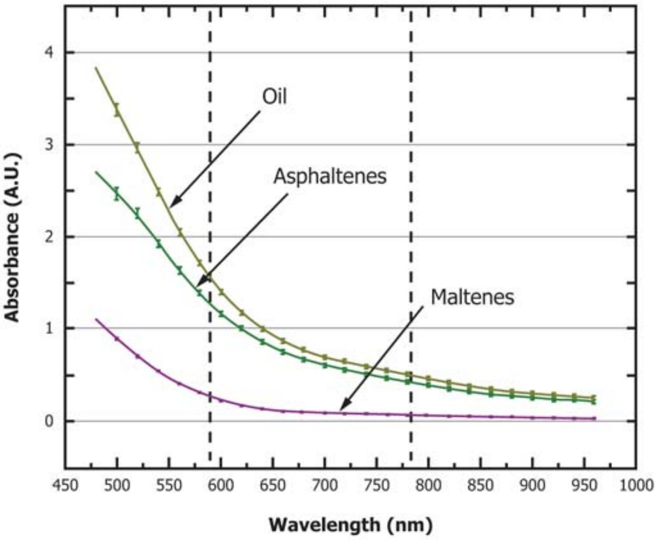
[6] Schneider, M. H., Sieben, V. J., Kharrat, A. M., and Mostowfi, F., "Measurement of asphaltenes using optical spectroscopy on a microfluidic platform," *Analytical Chemistry*, Vol. 85, No. 10, 2013, pp. 5153–5160.

[7] Sieben, V. J., Kharrat, A. M., and Mostowfi, F., "Novel Measurement of Asphaltene Content in Oil Using Microfluidic Technology," SPE Annual Technical Conference and Exhibition, 166394, 2013, New Orleans, LA, USA.

[8] Mostowfi, F and Sieben, V. J., "Determination of Asphaltenes Using Microfluidics," in Analytical Methods in Petroleum Upstream Applications, Edited by Cesar Ovalles and Carl E. Rechsteiner Jr. 2015, Taylor and Francis.



NOTE 1—The error bars show the standard deviation.

FIG. 3 Time-Averaged Spectrum of a Crude Oil and Associated Asphaltenes and Maltenes

time-averaged spectra $\bar{W}$, whereas the error bars show the standard deviation of the signal due to temporal variations in the signal.

10.1.6 The standard deviation due to time averaging for each spectra should be better than 3 % for the 500 nm to 900 nm range.

11. Report

11.1 Report the following information:

11.1.1 $\Delta OD_{asp(590\text{ nm} - 780\text{ nm})}$

11.1.2 Time-averaged visible spectrum of diluted oil ($\bar{W}_{oil}$), diluted maltenes ($\bar{W}_{malt}$), and diluted asphaltenes ($\bar{W}_{asp}$) from 500 nm to 900 nm.

11.1.3 Dilution ratios for oil and maltenes.

11.1.4 Flow cell path length in millimetres.

11.1.5 Reference this test method.

12. Precision and Bias

12.1 The precision statements for asphaltenes in heavy fuel oils and crude oils by spectroscopy in a microfluidic platform are based on results from within a single laboratory study only (Appendix X2). The study involved duplicate measurements by three operators, each using two instruments. An operator/instrument combination was counted as a laboratory.

12.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus, under constant operating conditions, on identical test materials would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$\text{Repeatability: r} = 5.014\text{E-02}(X + 0.0132)^{0.1814}\ \Delta OD_{asp(590\text{ nm} - 780\text{ nm})}$$

12.1.2 *Intermediate Precision*—The difference between single and independent results obtained within a short period of time by different operators working at different stations, using different test equipment at a single laboratory on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$\text{Between Operator/Apparatus Repeatability:} = 7.503\text{E-02}(X + 0.0132)^{0.1814}\ \Delta OD_{asp(590\text{ nm} - 780\text{ nm})}$$

12.1.3 *Reproducibility*—Due to unavailability of the test equipment in other laboratories, it is not possible to determine reproducibility of this test method using interlaboratory studies in accordance with ASTM Form & Style, Section A21.

12.2 *Bias*—Bias cannot be determined for this test method because there are no acceptable reference standards.

12.3 A full interlaboratory study of this test method will be completed and a complete precision statement is expected to be available within five years.

13. Keywords

13.1 asphaltene; asphaltenes content; asphaltenes spectroscopy; crude oils; lab-on-a-chip; microfluidics; residual fuel oils

APPENDIXES

(Nonmandatory Information)

X1. QUANTIFICATION OF ASPHALTENES CONTENT FOR CRUDE OILS

X1.1 Scope

X1.1.1 This test method covers a procedure for measuring the asphaltenes content of a crude oil based on the test method described above.

NOTE X1.1—Experience has shown that this test method can be applicable to other fuel types besides crude oils that are included within the scope, but accuracy is unknown and additional testing is required to determine the accuracy.

X1.1.2 The test method is limited to asphaltene-containing oil with asphaltenes content less than 15 % by mass.

X1.2 Procedure

X1.2.1 Follow the general procedure given in Section 9 for determining the visible spectrum of asphaltenes.

X1.2.2 Ensure the plateau region is selected for analysis. At 590 nm, standard deviation of the plateau region should be ±0.02 AU or lower for an average absorbance of 1 or less and ±0.05 AU or lower for an average absorbance larger than 1. This criterion can be used to ensure that the heart of the plug is used for analysis.

X1.3 Calculation

X1.3.1 *Calculation of Results:*

X1.3.2 Calculate $\Delta OD_{asp(590\ \mathrm{nm}\ -\ 780\ \mathrm{nm})}$ using Eq 6.

X1.3.2.1 If a volume ratio of 1 + 80 v/v is used for diluting the crude oil sample, then the following formula should be used:

$$\Delta OD_{asp(590\,nm\,-\,780\,nm)} = \frac{81}{41} \times \left[\bar{A}_{oil_{(\lambda\,=\,590\,nm)}} - \bar{A}_{oil_{(\lambda\,=\,780\,nm)}}\right] - \left[\bar{A}_{malt_{(\lambda\,=\,590\,nm)}} - \bar{A}_{malt_{(\lambda\,=\,780\,nm)}}\right] \quad (X1.1)$$

X1.3.3 The spectrum of asphaltenes can also be correlated to the mass concentration from a gravimetric measurement such as Test Methods D6560 and D3279. The concentration of asphaltenes is calculated using the following formula for a 3 mm path length flow cell:

$$M_a = 4.95 \times \Delta OD_{asp(590\,nm\,-\,780\,nm)} \quad (X1.2)$$

where M_a is the mass percent concentration of asphaltenes in the sample.

X1.4 Report

X1.4.1 Report the following information:

X1.4.2 The time-averaged absorbances of oil, maltenes, and asphaltenes at 590 nm and 780 nm: $\bar{A}_{oil_{(\lambda=590\ \mathrm{nm})}}$, $\bar{A}_{oil_{(\lambda=780\ \mathrm{nm})}}$, $\bar{A}_{malt_{(\lambda=590\ \mathrm{nm})}}$, $\bar{A}_{malt_{(\lambda=780\ \mathrm{nm})}}$, and $\Delta OD_{asp(590\ \mathrm{nm}\ -\ 780\ \mathrm{nm})}$

X1.4.3 Dilution ratios for oil and maltenes.

X1.4.4 Flow cell path length in millimetres.

X1.4.5 The concentration of asphaltenes in percent, M_a, to the nearest 0.1 % by mass.

X1.4.6 Reference this test method.

X1.5 Precision and Bias

X1.5.1 *Precision*—For repeatability, see 12.1.

X1.5.2 *Reproducibility*—For repeatability, see 12.1.

X1.5.3 *Bias*—Bias cannot be determined for these procedure since there are no acceptable reference standards.

X2. PRELIMINARY INTERLABORATORY [RUGGEDNESS] STUDY

X2.1 This Interlaboratory [ruggedness] Study (ILS) was conducted to establish a preliminary repeatability statement for Test Method D7996.

X2.2 Due to unavailability of the test equipment in other laboratories, this ILS was conducted using a protocol such that the "Reproducibility" calculated is an Intermediate Precision as per ASTM Form & Style.

X2.2.1 Specifically, it is a "Between Operator/Apparatus" repeatability, defined as the value for the absolute difference between two single and independent results obtained by different operators applying the same test method in same laboratory using different apparatus on essentially the same test material within short intervals of time would, in the long run, in the normal and correct operation of the test method, be exceeded only one case in 20.

X2.3 The following laboratories participated in this ILS—Schlumberger – DBR Research Laboratories. Participating analysts were: Drs. Farshid Mostowfi, Vincent Sieben, and Shahnawaz Molla from Schlumberger – DBR research.

X2.4 *Description of Samples*:

X2.4.1 Five samples covering a wide range of fluids were prepared. The samples are listed in Table X2.1.

X2.5 *ILS Instructions*:

X2.5.1 Analysts were instructed to perform the test per the steps provided in Sections 8 and 9 and results were obtained and reported according to Sections 10 and 11.

X2.6 *Statistical Data Summary*:

X2.6.1 Test data and statistical analyses are presented in Table X2.2, Table X2.3, and Fig. X2.1.

TABLE X2.1 Preliminary ILS Sample Matrix

Sample	Source	Dilution Ratio in Toluene (sample + toluene) w/w
Gas Condensate	Schlumberger – DBR	–
Black Oil	Schlumberger – DBR	–
Heavy Oil	Schlumberger – DBR	–
Bitumen	Schlumberger – DBR	1 + 1
Residue	Schlumberger – DBR	1 + 2.5

TABLE X2.2 Input Data for Asphaltenes in Heavy Fuel Oils and Crude Oils by Spectroscopy in a Microfluidic Platform (optical absorbance of asphaltenes ΔOD(590 nm-780 nm))

	Condensate	Black Oil	Heavy Oil	Residue	Bitumen
R-1 1	0.006	0.468	0.791	1.145	0.803
R-1 2	0.023	0.456	0.797	1.184	0.832
R-2 1	0.009	0.466	0.878	1.202	0.838
R-2 2	–0.005	0.474	0.863	1.214	0.818
R-3 1	–0.009	0.459	0.792	1.255	0.847
R-3 2	0.005	0.471	0.849	1.218	0.860
F-1 1	0.004	0.488	0.812	1.206	0.827
F-1 2	0.006	0.493	0.789	1.187	0.833
F-2 1	0.016	0.494	0.846	1.251	0.841
F-2 2	0.016	0.491	0.824	1.251	0.847
F-3 1	–0.013	0.475	0.807	1.252	0.842
F-3 2	0.012	0.492	0.831	1.255	0.864

TABLE X2.3 Test Data and Statistical Analyses

SAMPLE MEANS AND STANDARD DEVIATIONS
After any outlier rejections

Transform Type: Power

			Between Labs			Between Repeats		
Sample	No. of Results	Mean	S.D.	DF	R	S.D.	DF	r
Condensate	12	.37875E-01	.1905E-01	11	.59E-01	.1905E-01	6	.66E-01
Black Oil	12	.55808	.1333E-01	7	.45E-01	.6995E-02	6	.24E-01
Heavy Oil	12	.86391	.2625E-01	8	.86E-01	.1744E-01	6	.60E-01
Bitumen	12	.87614	.1458E-01	8	.48E-01	.1080E-01	6	.37E-01
Residue	12	1.1858	.2916E-01	6	.10	.1332E-01	6	.46E-01

VARIANCE ANALYSIS

Transform Type: Power

	Sum of Squares				
	Estimated	Exact	DF	Mean square	F
Sample Means	9.028292E+00	9.028292E+00			
Lab Means	5.714673E-03	5.714673E-03	5	1.14293E-03	1.879
Interaction	1.216360E-02		20	6.08180E-04	
Pairs	9.046170E+00	9.046170E+00			
Repeats	6.058223E-03		30	2.01941E-04	
Total	9.052229E+00				
Critical F (5 %, 5, 20) =					2.711

	Expectation of Mean Squares
Lab means	1.0000 V(r) + 2 V(I) + 10.0000 V(L)
Interaction	1.0000 V(r) + 2 V(I)
Repeats	1.0000 V(r)

	DF	Variance	Precision
Transformed data			
Repeatability	30	4.038816E-04	4.104E-02
Reproducibility	36	9.170716E-04	6.142E-02

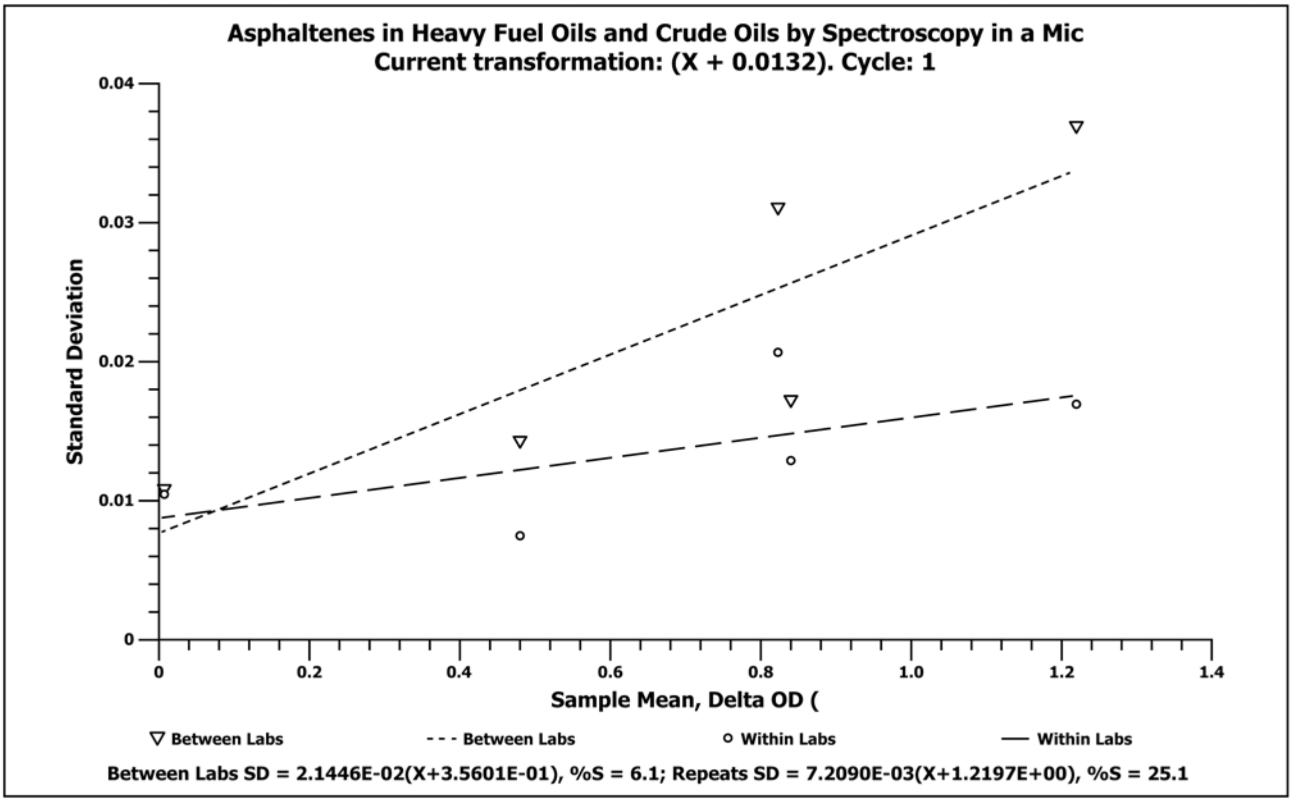


FIG. X2.1 Test Data and Statistical Analyses

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