



Designation: ~~D8253~~—20 D8253 – 21

Standard Test Method for Determination of the Asphaltene Solvency Properties of Bitumen, Crude Oil, Condensate and/or Related Products for the Purpose of Calculating Stability, Compatibility for Blending, Fouling, and Processibility (Manual Microscopy Method)¹

This standard is issued under the fixed designation D8253; 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~~ Scope*

1.1 This test method covers the use of a basic microscope to determine the asphaltene solvency properties of bitumen, crude oil, condensate or related products, or the combinations thereof. These properties can be used to calculate the solubility parameters required to calculate and predict asphaltene stability for blending purposes, refinery unit fouling, processibility, emulsion stabilization, pipeline and tank deposition, and equipment fouling. If the solubility properties are known for two products, then the compatibility of potential blend ratios can be predicted.

1.2 This test method is limited to products that allow handling at atmospheric pressure and room temperature conditions without a significant loss of light end components. Loss of light ends will result in changes in the solubility properties of the product and may alter or bias the results, or both. Samples with vapor pressures (VPCR₄ at 37.8 °C using Test Method **D6377**) greater than 100 kPa are not suitable for use with this test method.

1.3 This test method is primarily suited to products that are freely flowing at test conditions. Samples that are too viscous to flow at test conditions, such as semi-solids, may need to be heated to allow handling (See **Annex A1**.)

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered 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.*

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

2. Referenced Documents

2.1 ASTM Standards:²

- D6560 Test Method for Determination of Asphaltenes (Heptane Insolubles) in Crude Petroleum and Petroleum Products
- D6377 Test Method for Determination of Vapor Pressure of Crude Oil: VPCR_x (Expansion Method)

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 the addition of an excess of a specified paraffinic solvent.

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 heptane and pentane, but soluble in aromatic solvents such as benzene or toluene.

3.1.2 *compatibility, n*—of crude oils or 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 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 *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).

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

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

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *bitumen, n*—a naturally occurring, black, highly viscous liquid or semi-solid form of petroleum.

3.2.1.1 Discussion—

In some parts of the world, the term ‘bitumen’ is also used to describe residues from petroleum refining.

3.2.2 *flocculation ratio at critical dilution, n*—percentage by volume of solvent material to non-solvent material.

3.2.3 *light ends, n*—components that cannot be maintained as a liquid at atmospheric pressure at temperatures greater than 0 °C.

3.2.3.1 Discussion—

This includes any materials that have atmospheric boiling points below 0 °C including methane, ethane, propane, butane, and fixed gases such as H₂S, CO₂, N₂, O₂, H₂, CO.

3.2.4 *maximum flocculation ratio, n*—of asphaltenes, minimum required solvency power, expressed as percentage of toluene in a mixture of toluene and heptane, to keep the asphaltenes in a colloidal solution.

3.2.4.1 Discussion—

Maximum flocculation ratio is the flocculation ratio at the extrapolated infinite dilution of the sample in other words the y-intercept of the linear regression of FR vs Dilution Number.

3.2.5 *peptizing power, n*—available solvency power to keep asphaltenes in a colloidal solution.

3.2.6 *solvency, n*—ability to dissolve precipitated asphaltenes in a hydrocarbon matrix.

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.

3.2.7 *thermally processed material, n*—the product of crude oil that has been processed through a thermal process for the purpose of viscosity and density reduction; also referred to as a product of visbreaking, upgrading, or partial upgrading.

3.3 Abbreviations:

3.3.1 FR—flocculation ratio at critical dilution

3.3.2 FR_{max} —maximum flocculation ratio

3.3.3 P_o —peptizing power

3.3.4 *P-value*—peptization value

3.3.5 GO—gas oil

3.3.6 HVGO—heavy vacuum gas oil

3.3.7 SCO—synthetic crude oil

3.3.8 VGO—vacuum gas oil

4. Summary of Test Method

4.1 The sample type is first determined as either “with asphaltenes” or “without asphaltenes” to identify if it is a source of asphaltenes or not. If the sample type is “without asphaltenes” then a second test is performed to establish if the solvency of the sample is similar in behavior to either toluene or heptane. The sample is contacted with a reference oil known to contain asphaltenes. If the reference oil contacted with the sample results in the precipitation of asphaltenes, then it is considered similar to heptane (low solvency). If the reference oil contacted with the sample does not result in the precipitation of asphaltenes then it is considered similar to toluene (high solvency).

4.2 For samples with asphaltenes, a known mass of sample is contacted with an excess of heptane to force precipitation of asphaltenes. Incrementally toluene is added to the sample/heptane blend and a droplet of the mix is observed under a microscope at each increment. The point at which asphaltenes are no longer visible is recorded as the end point and the procedure is repeated twice more with increasing sample masses each time. The end point from the three tests is plotted and the solubility parameters calculated. (Procedure A)

4.3 For samples without asphaltenes that exhibit behavior similar to heptane (low solvency), a known reference oil containing asphaltenes is contacted with an excess of the sample material to force precipitation of asphaltenes. Incrementally toluene is added to the reference oil/sample blend and a droplet of the mix is observed under a microscope at each increment. The point at which asphaltenes are no longer visible is recorded as the end point and the procedure is repeated twice more with increasing sample masses each time. The end point from the three tests is plotted and the solubility parameters calculated. (Procedure B)

4.4 For samples without asphaltenes that exhibit behavior similar to toluene (high solvency), a known reference oil containing asphaltenes is contacted with an excess of the heptane to force precipitation of asphaltenes. Incrementally the sample is added to the reference oil/heptane blend and a droplet of the mix is observed under a microscope at each increment. The point at which asphaltenes are no longer visible is recorded as the end point and the procedure is repeated twice more with increasing sample masses each time. The end point from the three tests is plotted and the solubility parameters calculated. (Procedure C)

5. Significance and Use

5.1 Understanding the stability and compatibility of a petroleum product (crude oil or refinery stream, or both) is critical to facilities that receive multiple types of products and perform blending and processing operations. Blending incompatible streams can cause asphaltene precipitation with potential consequences such as but not limited to: refinery unit fouling, processing problems, throughput reduction, emulsion stabilization, pipeline and tank deposition, and equipment fouling.

5.2 The ability to predict the results of blending operations allows operators to anticipate potential problems and mitigate those problems prior to receiving the products. It also helps facilities to manage their product movements in the most effective manner to avoid future issues.

5.3 Some petroleum products are unstable without blending and understanding the stability of a product in terms of asphaltene precipitation is an important factor in product selection for refining. Products with poor stability can contribute to refinery unit fouling as well as the overall processibility and yield of that product.

6. Interferences

6.1 Solids materials such as salts, sands, clays and toluene insoluble organic material can mask the asphaltene dissolution endpoint. Samples exhibiting high solids contents prior to solubility testing should be pre-cleaned either via centrifuge or filtration to remove insoluble solids.

7. Apparatus

7.1 Microscope:

7.1.1 Total magnification between 80 and 200 times.

7.1.2 Cross polarization capability.

7.1.3 Digital image capture capability.

7.2 Analytical Balance, 4-digit accuracy.

7.3 Magnetic Stir Plate with Heating Capability.

7.4 *40 mL to 60 mL Glass Vial with Cap*—Vial diameter shall not exceed 25 mm (1 in. nominal).

7.5 Microscope Slides with Cover Plates.

7.6 *Magnetic Stir Bar*, of length to fit within the glass vial in 7.4.

7.7 Pasteur Pipettes.

7.8 Homogenizing Mixer.

7.9 Vortex Mixer.

7.10 Centrifuge:

7.10.1 100 mL conical centrifuge tubes.

7.11 Filtration Apparatus (Vacuum or Pressure):

7.11.1 0.45 μm filter.

8. Reagents and Materials

8.1 *Purity of Reagents*—Use chemicals of at least 99 % purity. Unless otherwise indicated, it is intended that all reagents conform

to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

8.2 Solvents:

8.2.1 *Heptane*, (**Warning**—Heptane is flammable and a health hazard.)

8.2.2 *Toluene*, (**Warning**—Toluene is flammable and a health hazard.)

8.2.3 *Heptane/Toluene Blend at 9:1 ratio*. (**Warning**—Heptane/toluene is flammable and a health hazard.)

8.3 Reference Oil:

8.3.1 Shall have ≥ 5 % heptane insoluble asphaltene content as per Test Method **D6560**.

8.3.2 Shall have known solubility parameters determined using Procedure A.

8.3.3 Duplicate runs of selected reference material shall meet method repeatability criteria.

8.3.4 *P*-value > 2.0 as derived from Procedure A and subsequent calculations.

8.3.5 Reference oil shall not be sourced from thermally processed material.

8.3.6 Known density at test conditions.

9. Sample Preparation

9.1 Sample preparation is carried out to evaluate the sample for solids that may interfere with the test procedure.

9.2 Sample Preparation Procedure:

9.2.1 Homogenize the sample using a high-speed mixer.

9.2.2 Using a Pasteur pipette, take a small sub-sample and transfer one drop to a microscope slide and place a cover plate over the drop(s).

9.2.3 Under the microscope, focus and observe any particulate matter visible under normal light. Capture an image.

9.2.4 Switch to cross-polarized light and observe again. Capture an image.

9.2.5 Compare the two captured images.

9.2.5.1 Crystalline particles (inorganic and waxes) will appear white under cross polarization. See **Fig. 1**.

9.2.5.2 Black particles remaining are amorphous asphaltenes or other insoluble hydrocarbons. See **Fig. 2**.

9.2.6 If there are significant solids present (crystalline or amorphous) that will make differentiation of changing asphaltene populations during the test procedure difficult, then the sample will need to be cleaned as per Section **10**. If there are no solids present or only solids that are easily distinguishable as background materials and will not interfere with the test procedure, then proceed to Section **11**.

³ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

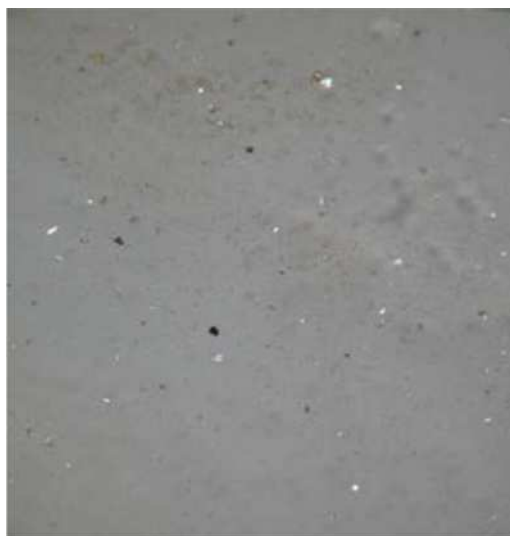


FIG. 1 Cross-polarization

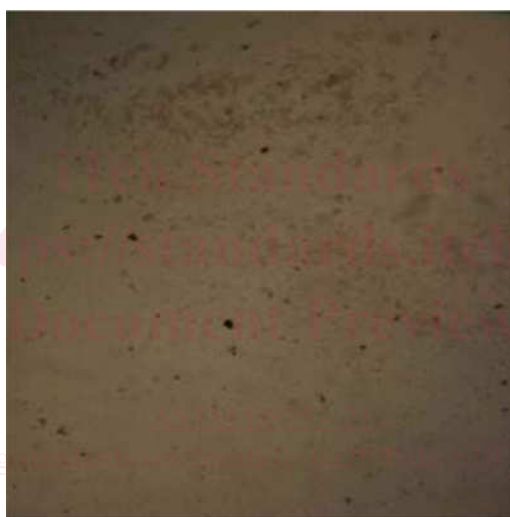


FIG. 2 Normal Light

10. Sample Cleaning

10.1 Since the intention of this test method is to evaluate the solubility properties of the bulk sample, removing already insoluble material will not bias the test results.

10.2 The cleaning method selected will be dependent on what equipment is available as well as the nature of the sample. For example, highly viscous samples may only be possible to filter by pressure filtration.

10.2.1 Highly volatile samples can experience loss of low molecular weight hydrocarbons that can alter the solvency properties of the cleaned product. These types of sample shall only be cleaned if the sample composition will not be compromised. Pressure filtration methods may be suitable.

10.3 The specific cleaning technique selected is not critical as cleaning is simply intended to minimize interferences but also to allow flexibility with respect to equipment availability.

10.4 *Centrifuge Method:*

10.4.1 Transfer 100 mL of sample to a conical centrifuge tube. Do not dilute the sample with solvent.

10.4.2 Weigh and record the mass of the sample and centrifuge tube.

10.4.3 Fill an identical centrifuge tube (balancing tube) with water.

10.4.4 Weigh the balancing tube and add or remove water until it matches the mass of the sample tube.

10.4.5 Place tubes in centrifuge rotor directly opposite one another.

10.4.6 Spin at minimum 1000 rpm for 30 min.

10.4.7 Decant approximately 50 mL of the upper layer into a vial for use in further testing. Use caution not to disturb and redistribute any solids from the bottom of the tube into the bulk phase.

10.4.8 Dispose of the remaining sample in the centrifuge tube.

10.5 *Vacuum Filtration Method:*

10.5.1 Prepare the filtration apparatus as per manufacturer instructions.

10.5.2 Filter approximately 50 mL to 60 mL of sample and start the vacuum. Typical vacuum pressure is 10 mmHg.

10.5.3 Collect the filtrate in a vial for use in further testing.

10.5.4 Dispose of the filter and any remaining residue.

10.6 *Pressure Filtration Method:*

10.6.1 Prepare the filtration apparatus as per manufacturer instructions.

10.6.2 Fill the pressure reservoir with approximately 50 mL to 60 mL of sample.

10.6.3 Pressurize the reservoir as per manufacturer instructions. Do not exceed the maximum working pressure of the filtration apparatus. <https://standards.iteh.ai/catalog/standards/sist/2eeafa7b-0a2f-48da-b2ff-0df493faa3d0/astm-d8253-21>

10.6.4 Open the reservoir slowly to begin filtration and proceed until sample is no longer visible dripping from the filter outlet.

10.6.5 Collect the filtrate in a vial for use in further testing.

10.6.6 Dispose of the filter and any remaining residue.

11. Identification of Sample Type and Solvency

11.1 *Sample Type Evaluation:*

11.1.1 Transfer to a vial, a 1 mL \pm 0.1 mL aliquot of either the neat sample or cleaned sample, depending on the outcome in Section 9.

11.1.2 Add 5 mL \pm 0.5 mL of heptane to the vial containing the sample. Place the cap on the vial and mix using a vortex mixer for 30 s.

11.1.3 Stop mixing and let stand for 5 min.

11.1.4 Using a Pasteur pipette, take a small sub-sample and transfer one or two drops to a microscope slide and place a cover plate over the drop(s).

11.1.5 Using the microscope, focus and observe any particulate matter is visible under normal light.

11.1.5.1 If visible asphaltenes are present proceed to Section 12 (Procedure A).

11.1.5.2 If no asphaltenes are present proceed to 11.2 to evaluate the solvency of the sample.

11.2 Solvency Testing for Samples without Asphaltenes:

11.2.1 Transfer to a vial, a 1 mL \pm 0.1 mL aliquot of reference oil.

11.2.2 Add 5 mL \pm 0.5 mL of sample to the vial containing the reference oil. Place the cap on the vial and mix using a vortex mixer for 30 s.

11.2.3 Stop mixing and let stand for 5 min.

11.2.4 Take a small sub-sample and transfer one or two drops to a microscope slide and place a cover plate over the oil drop(s).

11.2.5 Observe the sample under the microscope.

11.2.5.1 If asphaltenes were observed under the microscope, then the test oil behaves like heptane. Proceed to Section 13 (Procedure B).

11.2.5.2 If asphaltenes were not observed under the microscope, then the test oil behaves like toluene. Proceed to Section 14 (Procedure C).

12. Procedure A – Samples with Asphaltenes

12.1 Measure and record the density of the test oil sample at the test temperature.

12.2 Weigh a vial including a magnetic stir bar and record the tare mass of the vial and stir bar.

12.3 Weigh 1 g \pm 0.01 g of the sample into the tared 40 mL vial containing the magnetic stirring bar.

12.4 Place the vial on the stir plate and begin stirring slowly.

12.5 Using a pipette, slowly add 10 mL \pm 0.01 mL of the 9:1 heptane/toluene mixture to the vial at approximately 1 mL/min.

12.5.1 If the sample stability (P -value) is already reasonably known to the user and it is understood that the sample is typically unstable (P -value $<$ 1.5), then it is permissible to increase the sample sizes and reduce the 9:1 heptane: toluene mixture volume. For example: instead of 1.0 g, 2.0 g, and 3.0 g of sample and 10 mL of heptane/toluene mixture, use 2.5 g, 5.0 g, 7.5 g of sample and 4.5 mL of heptane/toluene mixture.

12.6 Close the cap and stir the mixture for 5 min.

12.7 Weigh and record the mass of vial and sample/heptane mixture.

12.8 Stop stirring and remove the cap from the vial.

12.9 Using a Pasteur pipette, immediately transfer one or two drops of the mixture from the vial to a clean microscope slide and cover the drop(s) with a cover slip.

12.10 Observe the flocculated asphaltenes under the microscope using total magnification of between 80 \times to 200 \times . The observation should be similar to Fig. 3.

NOTE 1—Based on preliminary round robin results, the mass loss from drop removal is negligible and does not impact overall results.

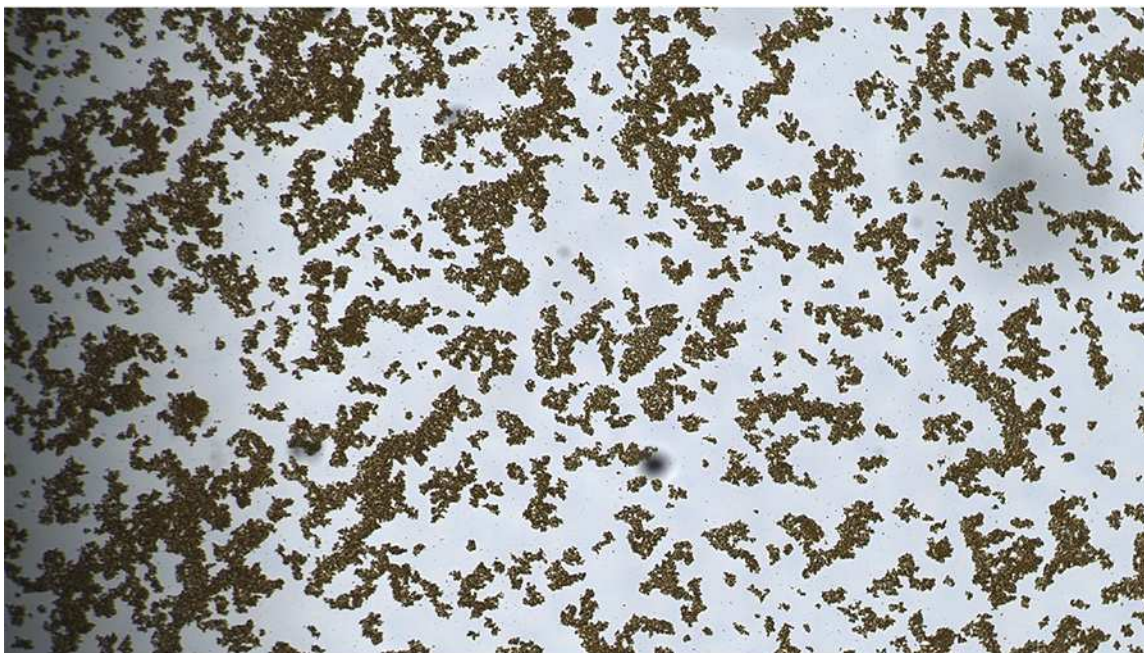


FIG. 3 Flocculated Asphaltenes Under Microscope

- 12.11 Capture multiple images at various positions on the slide and record any observations.
- 12.12 Record the volume of heptane used.
- 12.13 To the same vial, add $1 \text{ mL} \pm 0.01 \text{ mL}$ of toluene.
- 12.14 Weigh and record the mass of the vial, close the cap and stir for 5 min.
- 12.15 Stop stirring and remove the cap from the vial.
- 12.16 Using a Pasteur pipette, immediately transfer one or two drops of the mixture from the vial to a clean microscope slide and cover the drop(s) with a cover slip.
- 12.17 Observe the flocculated asphaltenes under the microscope.
- 12.18 Capture multiple images at various positions on the slide and record observations. The asphaltenes population should be less dense than what is shown in Fig. 3.
- 12.19 After making the microscope observation in 12.17:
- 12.19.1 If there is still a large population of asphaltenes present, then repeat steps 12.14 – 12.20 until the population of asphaltenes is reduced significantly.
- 12.19.2 If asphaltenes are still present but the population density is reduced significantly, then proceed to 12.21.
- 12.20 Reduce the step addition from 1 mL of toluene to $0.5 \text{ mL} \pm 0.01 \text{ mL}$ of toluene and stir the mixture for 5 min.
- 12.21 Stop stirring and capture multiple images at various positions on the slide and record observations.
- 12.22 Continue with the addition of toluene in $0.5 \text{ mL} \pm 0.01 \text{ mL}$ steps until the asphaltenes are almost gone. See Fig. 4.



FIG. 4 Observation Nearing Dissolution End Point

12.23 Record the total volume of toluene used. Capture multiple images at various positions on the slide and record any observations.

12.24 Reduce the toluene addition to $0.2 \text{ mL} \pm 0.01 \text{ mL}$ and repeat until there are no discernible asphaltenes observed under the microscope. See Fig. 5.

12.25 Record the total volume of toluene used. Capture multiple images at various positions on the slide and record observations.

<https://standards.iteh.ai/catalog/standards/sist/2eeafa7b-0a2f-48da-b2ff-0df493faa3d0/astm-d8253-21>



FIG. 5 Observation After Dissolution End Point

12.26 If the endpoint is missed, repeat steps 12.2 – 12.26. This this attempt start step 12.24 at a lower total toluene volume than in the previous attempt.

12.27 Record the total amount of toluene used to just dissolve the precipitated asphaltenes in the oil sample.

12.28 Repeat steps 12.2 – 12.27 using 2 g and 3 g sample in 12.3.

13. Procedure B – Samples, without Asphaltenes, Behaving Like Heptane (Cause Asphaltene Precipitation)

13.1 Measure and record the density of the sample at the test temperature.

13.2 Weigh a vial including a magnetic stir bar and record the tare mass of the vial and stir bar.

13.3 Weigh $1\text{ g} \pm 0.01\text{ g}$ of the reference oil into a tared 40 mL vial containing the magnetic stirring bar.

13.4 Place the vial on the stir plate and begin stirring slowly.

13.5 Using a pipette, slowly add $9\text{ mL} \pm 0.01\text{ mL}$ of the sample to the vial at approximately 1 mL/min.

13.6 Close the cap and stir the mixture for 5 min.

13.7 Weigh and record the mass of vial and reference oil/sample mixture.

13.8 Stop stirring and remove the cap from the vial.

13.9 Using a Pasteur pipette, immediately transfer one or two drops of the mixture from the vial to a clean microscope slide and cover the drops with a cover slip.

NOTE 2—Based on preliminary round robin results, the mass loss from drop removal is negligible and does not impact overall results.

13.10 Observe the flocculated asphaltenes under the microscope using total magnification of between 80 to 200 times. The observation should be similar to Fig. 3.

13.11 Capture multiple images at various positions on the slide and record observations.

13.12 Record the volume of sample used.

13.13 To the same vial, add $1\text{ mL} \pm 0.01\text{ mL}$ of toluene.

13.14 Weigh and record the mass of the vial, close the cap and stir for 5 min.

13.15 Stop stirring and remove the cap from the vial.

13.16 Using a Pasteur pipette, immediately transfer one or two drops of the mixture from the vial to a clean microscope slide and cover the drops with a cover slip.

13.17 Observe the flocculated asphaltenes under the microscope.

13.18 Capture multiple images at various positions on the slide and record observations. The asphaltenes population should be less dense than what is shown in Fig. 3.