



Standard Test Methods for Separation of Asphalt into Four Fractions¹

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1. Scope

1.1 These test methods cover the separation of four defined fractions from petroleum asphalts. The four fractions are defined as saturates, naphthene aromatics, polar aromatics, and nC_7 -asphaltenes. These methods can also be used to isolate saturates, naphthene aromatics, and polar aromatics from distillate products such as vacuum gas oils, lubricating oils, and cycle stocks. These distillate products usually do not contain asphaltenes.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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.* Specific precautionary statements are given in Section 8 and 15.

2. Referenced Documents

2.1 ASTM Standards:

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials²

C 802 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials²

D 140 Practice for Sampling Bituminous Materials³

D 3279 Test Method for n -Heptane Insolubles³

2.2 Other Documents:

Manual on Hydrocarbon Analysis⁴

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *asphaltenes or n -heptane insolubles*—insoluble matter that can be separated from asphalt following digestion of the asphalt in n -heptane under the specified conditions in these test methods.

3.1.2 *naphthene aromatics*—material that is adsorbed on calcined CG-20 alumina in the presence of n -heptane, and desorbed by toluene, after removal of the saturates under the conditions specified.

3.1.3 *petrolenes*—the n -heptane-soluble matter recovered following separation of the asphaltenes from the digested mixture under the specified conditions in these test methods.

3.1.4 *polar aromatics*—material desorbed from calcined CG-20 alumina absorbent, after the saturates and naphthene aromatics have been removed, using toluene and trichloroethylene eluants under the conditions specified.

3.1.5 *saturates*—material that, on percolation in a n -heptane eluant, is not adsorbed on calcined CG-20 alumina absorbent under the conditions specified.

METHOD A

4. Summary of Test Method

4.1 The sample containing the four defined fractions is first separated into n -heptane-insoluble asphaltenes and the n -heptane-soluble petrolenes. Petrolenes are then adsorbed on calcined CG-20 alumina and further fractionated into the saturate, naphthene aromatic and polar aromatic fractions by downward solvent elution in a glass chromatographic column. Eluted fractions are recovered by solvent removal prior to final weighing. The three eluted fractions plus the n -heptane-precipitated (nC_7) asphaltenes comprise the four fractions defined in Section 3.

5. Significance and Use

5.1 This test method separates asphalts into four well-defined fractions. Analysis of these fractions can be used to evaluate asphalt composition. For example, one can compare the ratios of the fractions with other asphalt systems to evaluate processing and aging parameters that relate to performance properties of the asphalt.

6. Apparatus and Materials

6.1 *Glass Chromatographic Column*,⁵ 1000 mm long and 31 mm in inside diameter with features as specified in Fig. 1.

6.2 *Utilities*—Steam bath, vacuum source, nitrogen source, and drying oven (Rotavapor solvent stripper and electric heating mantles optional).

¹ This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 in Miscellaneous Asphalt Tests.

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² *Annual Book of ASTM Standards*, Vol 04.02.

³ *Annual Book of ASTM Standards*, Vol 04.03.

⁴ Available from ASTM as PCN 03-332030-12.

⁵ Can be custom-made by any professional glassblower.

METHOD A

Funnel, Equal-Pressure, Graduated 500 ml With 24/40 Standard Taper Joint or Equivalent Sized Ball Joints

24/40 Standard Taper Joint or Equivalent Sized Ball Joints

Borosilicate Glass Column
31 mm ID x 1000 mm

Glass Wool Plug Concave Filter Pad Cut From 33/94 mm Extraction Thimble

TFE-Fluorocarbon 2 mm Stopcock With Vernier Adjustment

24/40 Standard Taper Joint or Equivalent Sized Ball Joints

Funnel, Same As Above

Tared Receiver (Beaker or Flask)

METHOD B

Funnel, Equal-Pressure, Graduated 125 ml With 24/40 Standard Taper Joint or Equivalent Sized Ball Joints

24/40 Standard Taper Joint or Equivalent Sized Ball Joints

Borosilicate Glass Column
25 mm ID x 510 mm

Glass Wool Plug Plus Concave Filter Pad Cut From 26/60 mm Extraction Thimble

TFE-Fluorocarbon 2 mm Stopcock With Vernier Adjustment

24/40 Standard Taper Joint or Equivalent Sized Ball Joints

Funnel, Same As Above

Tared Receiver (Beaker or Flask)



FIG. 1 Chromatographic Column for Separation of Asphalt by Elution-Adsorption (Method A or B)

6.3 Beakers, graduated; Erlenmeyer flasks, 400-mL; Round-Bottom Flasks, 500-mL, if Rotavapor is used for solvent removal.

6.4 Funnels, two, pressure-equalizing, 500-mL.

6.5 Funnel, Büchner, 12.5-cm.

6.6 Funnel, Separatory, 1-L, TFE-fluorocarbon stopcock preferred.

6.7 Flask, Suction, 2-L.

6.8 Flask, Erlenmeyer, 2-L, with foil-covered rubber stopper.

6.9 Rinse Squeeze Bottle, 0.5 L size, polyethylene or TFE-fluorocarbon.

6.10 Evaporating Dishes, porcelain, 16 and 28-cm.

6.11 Analytical Balance.

6.12 Filter Paper, slow to medium filter speed, qualitative grade, 12.5-cm diameter.

6.13 Extraction Thimble, 33/94 mm.

6.14 Stirrer, air-powered.

6.15 Stirring Rod with suitable foil-covered rubber stopper to fit 2-L Erlenmeyer flask.

6.16 *Glass Wool*, borosilicate.

6.17 *Electric Heat Lamp or Hot Plate*.

7. Reagents and Absorbent

7.1 *Alumina*,⁶ CG-20 chromatographic grade, calcined at 413°C for 16 h and stored in an evacuated desiccator or airtight bottles.

7.2 *n-Heptane* (Note 1), 99 minimum mol % (pure grade).

NOTE 1—*n*-Heptane should be totally free of moisture. Pretreatment of *n*-heptane with 5A molecular sieves or by refluxing over calcium hydride may be necessary to remove residual moisture in the solvent.

7.3 *Methanol*, anhydrous, reagent grade.

7.4 *Toluene*, reagent grade.

7.5 *Trichloroethylene*, boiling point 86.5 to 87.5°C.

8. Safety Precautions

8.1 Most organic solvents used in these methods are flammable and to some degree toxic. Reference should be made to Material Safety Data Sheets available from the supplier. These solvents should be handled with care and only in well-ventilated areas. All working areas should be kept free of sparks, flames, or other sources of high temperature.

9. Sampling and Sample Preparation

9.1 Bulk samples taken in accordance with Practice D 140 shall be representative and free of foreign substances. Samples for testing in 10.1.1 can be transferred by chilling to facilitate fracturing the sample or by heating the sample until it has become sufficiently fluid to pour. **Caution:** In no case shall the samples be heated more than 110°C above the expected softening point. Transfer of a representative portion from the bulk sample to a smaller container may be necessary for determination of sample mass to the nearest 0.01 g in 10.1.1.

10. Procedure

10.1 *Separation of Asphaltenes and Petrolenes:*

10.1.1 Weigh to the nearest 0.01 g and place into a 2-L Erlenmeyer flask a sufficient quantity of asphalt (Note 2) so that it yields about 10 g of petrolenes as indicated by the following equation:

$$\text{Sample mass, g} = 1000/100 - \% \text{ asphaltenes} \quad (1)$$

For paving asphalts, this would be 11 to 13 g of asphalt and slightly more for airblown asphalts. Unless the asphalt is in granular form, warm the flask gently with a heat lamp or hot plate and disperse the asphalt over the bottom and lower sides of the flask before adding the *n*-heptane solvent in the ratio of 100 mL of solvent per 1 g of sample.

NOTE 2—The quantity of asphalt required for 10 g of petrolenes can easily be predetermined in accordance with Test Method D 3279.

10.1.2 Install an air-powered stirrer assembly into the flask prior to placing flask and its contents on a steam bath. The stirring rod should rotate inside a foil-covered rubber stopper that is used to seal the Erlenmeyer flask to reduce the evaporation of *n*-heptane. Maintain the solvent temperature near its boiling point and stir the contents of the flask at a moderate rate until there is no visual evidence of undispersed asphalt adhering to the sides of the flask. Begin timing and continue stirring for an additional 1 h. Normally 1 h is sufficient time for straight reduced asphalts but for airblown or chemically modified asphalts the digestion time should be extended to 1.5 h. After digestion, remove the flask and stirring assembly from the steam bath. Rinse the stirring assembly as it is removed from the flask with *n*-heptane from a squeeze bottle. Cover the flask with a foil-covered rubber stopper and set aside overnight at ambient temperature so the precipitated asphaltenes can settle to the bottom of the flask.

10.1.3 Set up a 12.5-cm diameter Büchner funnel appropriately fitted with a slow to medium filter speed, qualitative-grade filter paper and a 2-L suction flask. The 1-L separatory funnel should be suspended about 25 mm above the center of the filter paper.

10.1.4 Decant as much of the clear heptane-petrolene solution as possible from the mixture prepared in 10.1.2 and place it directly in the separatory funnel.

10.1.5 Wet the filter paper in the Büchner funnel with *n*-heptane from a squeeze bottle and apply sufficient suction to the flask to secure the filter paper firmly to the funnel surface before beginning the filtration step. Add petrolene solution from the separatory funnel at a closely controlled rate and in such a manner that all of the filtering takes place in the center of the paper. The filter paper should be wetted periodically from the squeeze bottle to ensure a tight seal with the funnel surface. After the filtering is completed, it is advisable to empty or replace the suction flask before proceeding with the final phase of filtration process.

10.1.6 Test the filtrate from 10.1.5 for insolubles by placing a drop of the filtrate on a filter paper. Refilter if a ring appears.

10.1.7 Transfer the contents remaining in the Erlenmeyer flask directly to the Büchner funnel, using additional solvent from the squeeze bottle and repeatedly wash the asphaltene cake until the filtrate becomes colorless. Take care to ensure that no insolubles creep over the edges of the filter paper into the filtrate. Next, transfer the filter paper and its contents to a 500-mL beaker and add 150 mL of *n*-heptane. Heat the contents in the beaker for about 30 min with occasional stirring to remove *n*-heptane-soluble materials entrained in the asphaltene cake. Filter the hot solution through the same Büchner funnel fitted with a tared, fresh piece of filter paper, using the prescribed procedures. Continue washing the asphaltene cake until the filtrate is colorless. Test the filtrate for insolubles as in 10.1.6 and repeat this task if a ring appears.

10.2 *Solvent Evaporation:*

10.2.1 Transfer the asphaltene cake on the filter papers (10.1.5 and 10.1.7) to a 16-cm evaporating dish and dry in a 104°C oven until a constant mass is achieved. Record the net mass of asphaltenes recovered and store if desired in a screw-cap bottle.

⁶ F-20 grade alumina originally referenced in this standard is no longer available from Alcoa, the manufacturer. The CG-20 alumina now referenced is the replacement supplied by the manufacturer. The test results are very sensitive to the quality and grade of alumina used. Currently there is no data available indicating difference in test results between the F-20 and the CG-20 alumina. If users of this method have historical data, it may be useful to do a comparison between data derived from tests using the two aluminas.

10.2.2 Heptane can be removed from filtrates containing petroleins (10.1.5 and 10.1.7) using standard laboratory procedures. Concentrate the petroleum solution to about 50 mL and then transfer the concentrate to a 150-mL beaker. Sufficient fluidity should remain in the petroleum concentrate to permit easy transfer of concentrate to a chromatographic column for subsequent separation into the three defined fractions.

10.3 Separation of Petroleum Concentrate into Three Defined Fractions:

10.3.1 Set up the chromatographic column with appurtenances as shown in Fig. 1. Place a concave filter pad, cut from the bottom of an extraction thimble (6.13), on the bottom of the column, and follow by 25 mm of a glasswool plug. Add 450 g of calcined alumina while gently tapping the column with a rubber-coated object. Place a glass wool plug on top of the alumina bed, add 50 mL of *n*-heptane to prewet the column, and proceed immediately to 10.3.2.

10.3.2 Transfer the petroleum concentrate from 10.2.2 to the column, using minimum amount of *n*-heptane. Start addition of eluants with 200 mL of *n*-heptane (see Table 1) from the equal-pressure funnel at a drip rate of about 5 mL/min (Fig. 1); simultaneously collect eluate at the rate of 5 ± 1 mL/min in the receiving funnel; and subsequently drain the eluate into tared containers (weighed to 0.01 g) in accordance with the schedule in Table 1.

10.3.2.1 **Caution:** At no time should the liquid level in the column be allowed to drop below the top of the alumina bed until the final hold-up is removed.

10.3.3 Add the new eluant to the column before the final eluate cut from the previous eluant is taken. Based on earlier studies, such procedure regulates fractionation, provided eluate cuts are taken in accordance with the schedule in Table 1. This procedure requires close attention, especially during the collection of the saturates and naphthene aromatics fractions. The cut point between the naphthene aromatics and polar aromatics fractions is more obvious and may be improved by either cutting short or increasing the volume of this cut as observed by the descent of the black polar aromatic ring. The elution volume will vary with the composition of the feed, and the cut point should be made as the black ring reaches the bottom of the alumina bed. The naphthene aromatics fraction produces a yellow to deep red color eluate whereas the polar aromatics eluate is almost black. The hold-up in the column is that solvent collected by gravity drainage at the end of the elution process and should be essentially colorless.

10.3.4 Recover the three fractions by solvent removal procedures using standard laboratory procedures and record the net mass of each fraction.

TABLE 1 Separation Schedule

Column Feed Volumes		Fractions Received in Tared Containers	
Eluant Solvent	mL	Eluate Fraction	mL ^A
<i>n</i> -Heptane	200	Saturates (S)	300
Toluene	100		
Toluene	300	Naphthene-aromatics	600
Methanol/toluene 50/50	300	(N-A)	
Trichloroethylene	600	Polar-aromatics (P-A)	600 + hold up
Column hold-up	...		

^A Approximate eluate volumes since cut points may be adjusted (10.3.3) and hold-up can vary.

TABLE 2 Repeat Composition Analysis (Asphalt Cement, 89 Penetration)

Fraction	Mass on Sample	
	Run 1	Run 2
Saturates	10.8	11.1
Naphthene aromatics	39.3	39.9
Polar aromatics	36.6	35.5
Asphaltenes	13.0	12.8
Total	99.7	99.3

TABLE 3 Acceptability Criteria of Test Results

	Weight % of Sample	
	Standard Deviation	Acceptable Range of Two Results
Single Operator Precision:		
Asphaltenes 9 to 29 %	0.32	0.9
Saturates 8 to 16 %	0.44	1.2
Naphthene Aromatics 27 to 41 %	1.03	2.9
Polar Aromatics 20 to 41 %	0.78	2.2
Multilaboratory Precision:		
Asphaltenes 9 to 29 %	0.95	2.7
Saturates 8 to 16 %	0.70	1.9
Naphthene Aromatics 21 to 41 %	2.26	6.4
Polar Aromatics 20 to 41 %	2.37	6.7

11. Calculation and Report

11.1 Calculate the weight percent of the fractions, *F*, as the mass percent of the original samples as follows:

$$F, \% = (A/B) \times 100 \quad (2)$$

where:

A = mass of the fraction, and

B = mass of the sample.

11.2 Report percentages to the nearest 0.1 % mass.

12. Precision and Bias

12.1 Criteria for judging the acceptability of test results obtained by this test method are provided in Table 3. Data were obtained from tests by seven rather than ten laboratories as recommended in Practice C 802.

12.2 Material and range covered are described in Table 3. These numbers represent the (1S) and (D2S) limits as described in Practice C 670. This precision statement applies only to the ranges covered in the precision study. The results of tests which are far outside the ranges studied may have different precision values.

METHOD B—SHORT PROCEDURE

13. Apparatus and Materials

13.1 *Glass Chromatographic Column*⁷, 510 mm long and with a 25-mm inside diameter and features as specified in Fig. 1.

13.2 *Utilities*—Steam bath, vacuum source, nitrogen source and drying oven.

⁷ Available from Ace Glass Inc., Vineland, NJ, or can be custom-made by any professional glassblower.

- 13.3 *Beakers*, graduated; 100 and 150-mL.
- 13.4 *Funnels*, two pressure-equalizing, 125-mL.
- 13.5 *Crucible, Gooch*⁸.
- 13.6 *Funnel, Separatory*, 500-mL (TFE-fluorocarbon stop-cock preferred).
- 13.7 *Funnel, Erlenmeyer*, wide-mouth, 500-mL, with foil-covered rubber stopper.
- 13.8 *Flask*, suction, 500-mL.
- 13.9 *Evaporating Dish*, porcelain, 16-cm.
- 13.10 *Glass-Fibre Filter Pad*, 3.2-cm Diameter⁹.
- 13.11 *Extraction Thimble*, 26/60 mm.
- 13.12 *Stirring Rod*, with suitable foil-covered rubber stopper to fit a 500-mL Erlenmeyer flask.
- 13.13 *Glass Wool*, borsilicate.

14. Reagents and Absorbent

14.1 *Alumina*,⁵ CG-20 chromatographic grade, calcined at 775°F (413°C) for 16 h and stored in an evacuated desiccator or airtight bottles.

14.2 *n-Heptane* (Note 3), 99 minimum mol % (pure grade).

NOTE 3—*n*-Heptane should be totally free of moisture. Pretreatment of *n*-heptane with 5A molecular sieves or by refluxing over calcium hydride may be necessary to remove residual moisture in the solvent.

14.3 *Methanol*, anhydrous, reagent grade.

14.4 *Toluene*, reagent grade.

14.5 *Trichloroethylene*, boiling point 86.5 to 87.5°C.

15. Safety Precautions

15.1 Most organic solvents used in this method are flammable and to some degree toxic. Reference should be made to Material Safety Data Sheets available from the supplier. These solvents should be handled with care and only in well-ventilated areas. All working areas should be kept free of sparks, flames, or other sources of high temperature.

16. Sampling and Sample Preparation

16.1 Bulk samples taken in accordance with Practice D 140 shall be representative and free of foreign substances. Samples for testing in 10.1.1 can be transferred by chilling to facilitate fracturing the sample or by heating the sample until it has become sufficiently fluid to pour. **Caution:** In no case shall the samples be heated more than 110°C above the expected softening point. Transfer of a representative portion from the bulk sample to a smaller container may be necessary for determination of sample mass to the nearest 0.01 g in 10.1.1.

17. Procedure

17.1 *Separation of Asphaltenes and Petrolenes:*

17.1.1 Weigh to the nearest 0.001 g and place into a 500-mL Erlenmeyer flask a sufficient quantity of asphalt so that it yields about 2 g of petrolenes as indicated by the following equation:

$$\text{Sample weight, g} = 200 / (100 - \% \text{ asphaltenes}) \quad (3)$$

17.1.1.1 For paving asphalts, this would be 2.3 to 2.5 g of asphalt and slightly more for airblown asphalts. Unless the asphalt is in granular form, warm the flask gently with a heat lamp or hot plate and disperse the asphalt over the bottom and lower sides of the flask before adding the *n*-heptane solvent in the ratio of 100 mL solvent per 1 g of sample.

NOTE 4—The quantity of asphalt required for 2 g of petrolenes can easily be predetermined in accordance with Test Method D 3279.

17.1.2 Install an air-powered stirrer assembly into the flask prior to placing flask and its contents on a steam bath. The stirring rod should rotate inside a foil-covered rubber stopper that is used to seal the Erlenmeyer flask to reduce the evaporation of *n*-heptane. Maintain solvent temperature near the boiling point and stir contents at a moderate rate for ½ h. After digestion, remove the flask and stirring assembly from the steam bath. Rinse the stirring assembly, as it is removed from the flask, with *n*-heptane from a squeeze bottle. Cover the flask with a foil-covered rubber stopper and set aside overnight at ambient temperature so the precipitated asphaltenes can settle to the bottom of the flask.

17.1.3 Set up a tared Gooch crucible appropriately fitted with a glass-fibre filter pad and a 500-mL suction flask. The 500-mL separatory funnel shall be suspended about 25 mm above the center of glass-fibre filter pad.

17.1.4 Decant as much of the clear heptane-petroleum solution as possible from the mixture prepared in 17.1.2 and place it directly in the separatory funnel.

17.1.5 Wet the glass-fibre pad in the Gooch Crucible with *n*-heptane from a squeeze bottle and apply sufficient suction to the flask to firmly secure the filter pad to the crucible surface before commencing the filtration step. Add petroleum solution from separatory funnel at a closely controlled rate.

17.1.6 Transfer the contents remaining in the Erlenmeyer flask directly to the Gooch Crucible using additional solvent from the squeeze bottle and repeatedly wash the asphaltene cake until the filtrate becomes colorless. Test the filtrate for insolubles by placing a drop of the filtrate on a filter paper. If a ring appears, the filtrate must be refiltered.

17.1.7 Asphaltene components adhering to flask, stirrer, etc., can be recovered by dissolving residue in a solvent.

17.1.8 Remove the solvent using standard laboratory procedures, record net mass, and add to net mass determined in 17.2.1.

17.2 *Solvent Evaporation:*

17.2.1 Remove the crucible from the holder and allow it to stand at room temperature for about 10 min to allow most of the heptane to evaporate. Next, place the Gooch crucible containing the asphaltenes in an oven at 104°C and dry until a constant mass is achieved. Record the net mass of asphaltenes recovered.

17.2.2 Filtrates containing petrolenes (14.1.5) shall be transferred to a 160-mm evaporating dish and the solvent volume reduced using a steam bath and a light nitrogen sparge. Do not overfill the evaporating dish. Concentrate the petroleum solution to about 20 mL, then quantitatively transfer the concentrate to a 100-mL beaker. Sufficient fluidity should remain in the petroleum concentrate to permit easy transfer of concentrate

⁸ Gooch Crucible, Coor No. 27009 available from Gooch Porcelain.

⁹ Glass Filter Pads, No. 934AH, available from Reeve Angel and Co., Clifton, NJ.

to chromatographic column for subsequent separation into the three defined fractions.

17.2.3 The column with appurtenances is set up as shown in Fig. 2. A concave filter pad, cut from the bottom of an extraction thimble (9.13), is placed on the bottom of the column followed by a 25-mm plug of glass-wool. Loosen the top clamp-holding column about 0.5 in., then add 150 g of calcined CG-20 alumina while gently shaking the column. After alumina is added, gently tap the column with a rubber-coated object if necessary to reduce the height of the alumina bed to 380 ± 10 mm. Place a glass-wool plug on top of the alumina bed.

17.2.4 Immediately prewet the column with 20 mL of *n*-heptane, then quantitatively transfer the petroleum concentrate with 17.2.2 to the column using a minimum amount of *n*-heptane. Start the addition of eluants with *n*-heptane (see Table 4) from the equal-pressure funnel at a drip rate of 2 to 3 mL/min (Fig. 1); simultaneously collect the eluate at the rate of 2 to 3 mL/min in the receiving funnel; and subsequently drain the eluate into tared 150-mL beakers (weighed to 0.001 g) in accordance with the schedule in Table 4.

17.2.5 Add the new eluant to the column before the final eluate, cut from the previous eluant, is taken. Based on earlier studies, such procedures regulate fractionation, provided eluate

cuts are taken in accordance with the schedule in Table 1. This procedure requires close attention, especially during the collection of the saturates and naphthene aromatics fractions. The eluate volume of the saturates may vary and the cut point should be made if a yellow color is present before the prescribed eluate volume is obtained. The cut point between the naphthene aromatics and polar aromatics fractions is more obvious and may be improved by either cutting short or increasing the volume of this cut as observed by the descent of the black polar aromatic ring. The elution volume will vary with the composition of the feed, and the cut point should be made as the black ring reaches the bottom of the alumina bed. The naphthene aromatics fraction produces a yellow to deep red-color eluate, whereas the polar aromatics eluate is almost black. The hold-up in the column is that solvent collected by gravity drainage at the end of the eluation process and should be essentially colorless.

18. Calculation and Report

18.1 Calculate the mass percent of the fractions, F as the percentage by mass of the original sample as follows:

$$\% F = (A/B) \times 100 \quad (4)$$

where:

A = mass of the fraction, and

B = mass of the sample.

18.2 Report percentages to the nearest 0.1 % mass.

19. Precision and Bias

19.1 Criteria for judging the acceptability of test results obtained by Method B are provided in Table 5.

20. Keywords

20.1 asphaltenes; naphthene; aromatics; polar aromatics; saturates

TABLE 4 Separation Schedule

Column Feed Volumes Eluant Solvent	Fractions Received in Tared Containers of Eluate		
	mL	Fraction	Total mL ^A
<i>n</i> -Heptane	65		
Toluene	35	saturates (S)	100
Toluene	100		
Methanol/toluene 50/50	100	naphthene aromatics (N-A)	200
Trichloroethylene ^B	200		
Column hold-up		polar aromatics (P-A)	200 hold-up

^A Approximate eluate volumes since cut points may be adjusted (14.2.5) and hold-up can vary.

^B Asphalt components remaining on the column packing following the Trichloroethylene eluate can be removed by a 100-mL eluate of methanol.

TABLE 5 Acceptability Criteria for Method B

NOTE 1—The numbers represent the (1S and D2S) limits as described in Practice C 670. This precision statement applies only to the ranges covered in the precision study. The results of the tests that are far outside the ranges studied may have different precision values.

Material and Range Covered	Standard Deviation	Acceptable Range of Two Results
<i>Single Operator Precision:</i>		
Asphaltenes	9 to 29 %	0.32
Saturates	8 to 16 %	0.44
Naphthene aromatics	27 to 41 %	1.03
Polar aromatics	20 to 41 %	0.78
<i>Multilaboratory Precision:</i>		
Asphaltenes	9 to 29 %	0.95
Saturates	8 to 16 %	0.70
Naphthene aromatics	21 to 41 %	2.26
Polar aromatics	20 to 41 %	2.37

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