~~Designation:D4124–01~~

~~Standard Test Methods for~~ Designation: D 4124 – 09

Standard Test Method for Separation of Asphalt into Four Fractions[1]

This standard is issued under the fixed designation D 4124; 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

~~1.1These~~1.1 This test method~~s~~ covers 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~~ iso-octane insoluble asphaltenes. This method 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~~.

1.4 Since a precision estimate for this standard has not been developed, this test method is to be used for research or informational purposes only. Therefore, this standard should not be used for acceptance or rejection of a material for purchasing purposes.

2. Referenced Documents

2.1 *ASTM Standards:*~~*C670Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials*~~
~~C802Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials[2]~~ [2]
D 140 ~~Practice for Sampling Bituminous Materials~~
~~D3279Test Method for *n*-Heptane Insolubles[3]~~ Practice for Sampling Bituminous Materials

2.2 *Other Documents:*
Manual on Hydrocarbon Analysis [3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *asphaltenes* or ~~n-heptane~~ alkane insolubles—insoluble matter that can be separated from asphalt following digestion of the asphalt in ~~n-heptane~~ n-alkane (and in some cases, branched alkanes) under the specified conditions in ~~these~~this test ~~methods.~~ method.

3.1.2 *naphthene*—any of a group of hydrocarbon ring compounds of the general formula, C_nH_{2n}, derivatives of cyclopentane and cyclohexane, found in certain petroleum stocks.

3.1.3 *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.~~ -heptane, and desorbed by toluene, after removal of saturates under the conditions specified.

---

[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.

~~Current edition approved Aug. 10, 2001. Published October 2001. Originally published as D4124–82. Last previous edition D4124–97.~~

Current edition approved July 1, 2009. Published August 2009. Originally approved in 1982. Last previous edition approved in 2001 as D 4124 – 01.

[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* ~~, Vol 04.02.~~volume information, refer to the standard's Document Summary page on the ASTM website.

~~[3] Annual Book of ASTM Standards, Vol 04.03.~~

[3] Available from ASTM as PCN 03-332030-12.

~~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~~ *petrolenes (*also referred to as maltenes) —*(1)* any of the constituents of a bitumen, as asphalt, that are soluble in *n*~~-heptane eluant, is not absorbed on calcined CG-20 alumina absorbent under the conditions specified.~~

~~METHOD A~~

~~4.Summary of Test Method~~

~~4.1The sample containing the four defined fractions is first separated into~~ *-alkanes (and in some cases, branched alkanes), which generally range in carbon number between n*~~*-heptane-insoluble asphaltenes and the*~~ *-C_5 to 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*~~ *-C_{10} alkanes, n*~~*-heptane-precipitated (nC_7) asphaltenes comprise the four fractions defined in Section 3.*~~ *-heptane being the most common solvent used; (2)* the low molecular weight alkane-soluble matter recovered following separation of asphaltenes from the digested mixture under the specified conditions described in this and similar test methods.

3.1.5 *polar aromatics (resins)*—material desorbed from calcined CG-20 alumina absorbent, after the saturates fraction and naphthenic aromatics fraction have been removed, using toluene:methanol (50:50, vol:vol) and trichloroethylene eluate under the conditions specified.

3.1.6 *saturates*—material that, on percolation in an alkane eluate, is not absorbed on calcined CG-20 alumina absorbent under the conditions specified.

4. Summary of Test Method

4.1 The sample containing the four defined fractions is first separated into alkane-insoluble asphaltenes and alkane-soluble petrolenes. Petrolenes are then adsorbed onto calcined CG-20 alumina and further fractionated into saturate, naphthene aromatic, and polar aromatic fractions by pumping an eluotropic series of elution solvents upwards through a glass chromatographic column packed with calcined alumina. Eluted fractions are recovered by solvent removal prior to final weighing. The three eluted fractions plus the alkane-precipitated asphaltenes comprise the four fractions as 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 (1, 2). 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* , 1000 mm long and 31 mm in inside diameter with features as specified in~~

6.1 *Reflux Apparatus for Asphaltene/Maltene Separation,* 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).~~ . (see 6.1.1).

6.1.1 *Apparatus (*Fig. 1)— *(a)* ring stand with clamp; *(b)* heater-stirring plate; *(c)* 500-mL Erlenmeyer flask with 29/42 sintered glass neck; *(d)* reducer, 29/42-to-24/40 sintered glass necks; *(e)* Allihn-type reflux condenser with 24/40 sintered glass neck.

6.2 *Chromatographic Column Apparatus* , with features as specified in Fig. 2, (see 6.2.1-6.2.5).

6.2.1 *LC-Apparatus (*Fig. 2)— *(a)* ring stand w/clamp, flask (25-mL round-bottom or Erlenmeyer flask); *(b)* metering pump; *(c)* sealed glass LC-column; *(d)* UV Detector w/ Data Acquisition System (wavelength range 200-500 nm @ 0.1-nm); *(e)* graduated cylinder (Fig. 2).

6.2.2 *UV Detector with Data Acquisition System (*Fig. 2)—A UV Detector with Data Acquisition System suitable for use with liquid chromatography or HPLC used to detect the occurrence of material fractions (peak response) as they are eluted from the column.

6.2.3 *Metering Pump (*Fig. 2*)*—Piston and piston chamber will be constructed of materials resistant to deterioration by solvents that will be used to perform the method. Flow rate range of the pump will be 0.1 to 5.0-mL/min ± 0.1-mL/min flow rate stability.

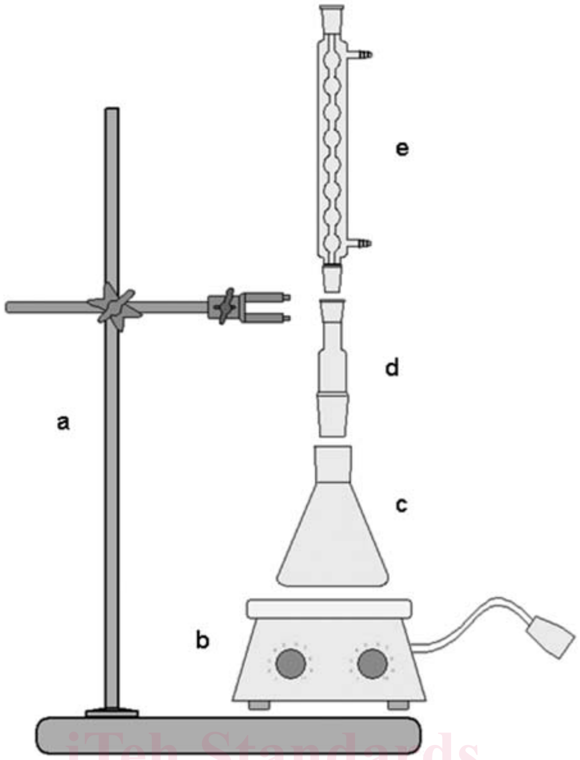
6.2.4 *LC-Column with Water-jacket*—Closed glass liquid chromatography column, 70 cm long and 1.5 cm inside diameter (volume, 124-cc). The LC-column will be a closed column with endplates containing solvent permeable diaphragms and fitting ports for 6.35-mm (¼-in.) tubing fittings.

6.2.5 *Refrigerated/Heating Circulator*— a refrigerated/heating circulator, temperature range between 0°C and 100°C ± 0.1°C stability, with water circulation through the LC-column water jacket via high pressure hose 15.9-mm (⅝ in. ID).

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.~~



N~~OTE~~OMATOE—Key: a. ring stand w/large test-tube clamp; b. heater/stie ~~Co~~rring pl~~u~~ate; c. 500-mL Erlenmeyer f~~o~~lask with 29/42 sinter ~~Sep~~d glass neck; d. r~~a~~educer 29/42-t~~io~~-24/40 sin ~~of A~~tered glass sp~~ha~~out-to-neck; e. Allihn-t ~~by E~~pe reflu~~tix~~ con~~-A~~dens~~oerp~~ with 24/40 si~~on (Metho~~ered ~~A~~ glass spo~~r B)~~ut.

FIG. 1 Asphaltene/Maltene Separation Apparatus

~~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*.~~ Materials:

6.3.1 *Utilities*—Fume hood, vacuum source, nitrogen gas source, cold water source, nitrogen gas-purged vacuum drying oven, rotary solvent evaporator (water-bath and oil-bath type), nitrogen gas stream evaporator with heater water bath.

6.3.2 *Erlenmeyer Flasks*, 25-mL with Glass Stopper (1), 500-mL with Glass Stoppers (5).

6.3.3 *Pear-shaped Flask*,

6.3.4 *Graduated Cylinder*,

6.3.5 *Büchner-style Funnel, Fritted-glass* , 60 to 100-mL, ASTM 10-15 µL medium porosity.

6.3.6 *Flask, Suction*, 1 L to 2 L.

6.3.7 *Rinse Squeeze Bottle*, 0.5-L size, TFE-fluorocarbon.

6.3.8 *Analytical Balance*, 0.0001 g to 250 g ± 0.0001 g.

6.3.9 *Stirrer/heater Plate*, electric.

6.3.10 *Sample Vials*, Borosilicate, 25-mL (6) and 50-mL (2), clear, with Teflon-lined cap.

6.3.11 *Glass Funnels* (2), small.

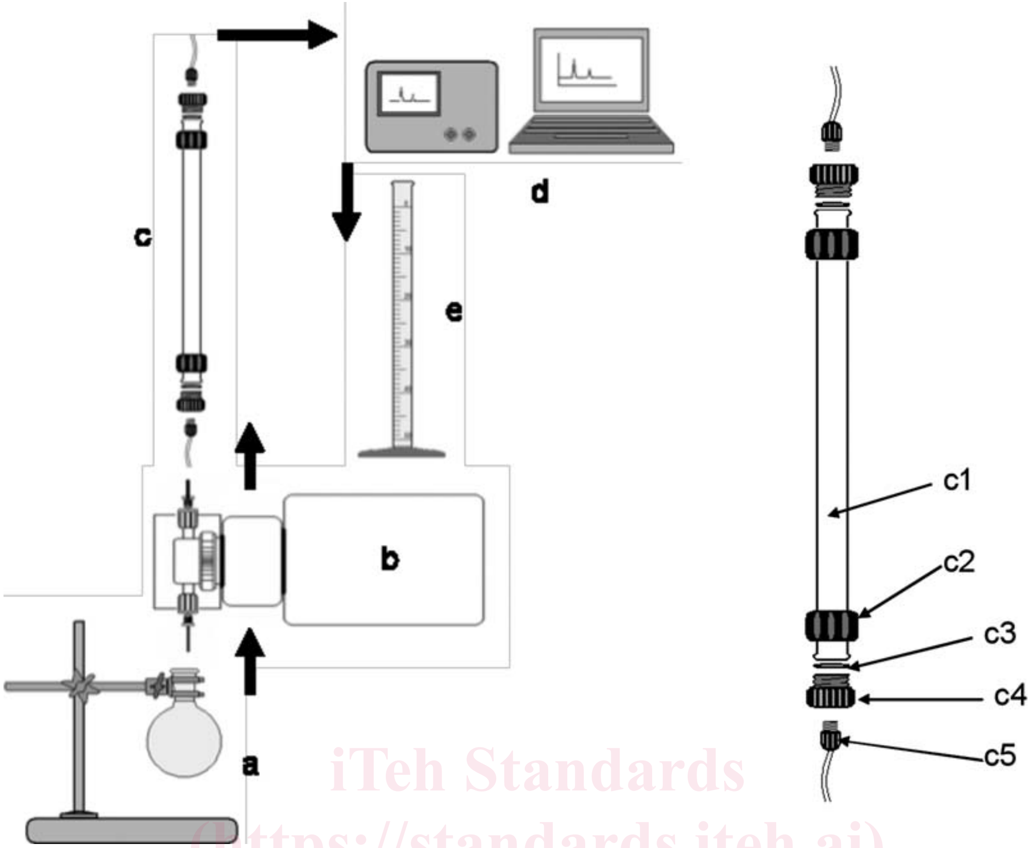
6.3.12 *Teflon or Solvent-resistant Funnels* (1), small.

6.3.13 *Teflon Flask-neck Sleeves*, (3) 24/40-size, (1) 29/42-size.

6.3.14 *Hose*, reinforced, high pressure, 1.59-mm ID (5⁄8 in. ID).

6.3.15 *Tubing*, clear, resistant to organic solvents, 1.59-mm ID/3.17-mm OD (1⁄16 in. ID/1⁄8 in. OD).

6.3.16 *Tubing Fittings*, standard 6.35-mm (1⁄4 in.) nut with 3.17-mm OD (1⁄8 in. ID)-hole and 3.17-mm (1⁄8 in.) ferrule.



NOTE—Key: LC-Apparatus: a. clamp stand w/sample or solvent flask (round-bottom or Erlenmeyer type); b. metering pump; c. sealed glass LC column packed with alumina; c1. glass column; c2. collar; c3. diaphragm; c4. endplate; c5. tubing nut/ferrule and tubing; d. UV-VIS spectrophotometric detector (200-500 nm @ 0.1-nm); e. graduated cylinders of various sizes for fraction collection.

FIG. 2 Chromatographic Column for Separation of Asphalt by Elution-Adsorption

7. ~~Reagents and Absorbent~~

~~7.1~~*Absorbent and Reagents*

7.1 *Alumina,*~~CG-20 chromatographic grade, calcined at 413°C for 16 h and stored in an evacuated desiccator or airtight bottles.~~[4] CG-20 chromatographic grade, calcined at 425°C for 16 h and stored in an evacuated desiccator in airtight bottles for 3 to 5 h.

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.~~ Purity of Reagents—HPLC grade chemicals shall be used in all sample preparations and tests. 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.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.3 *Reagents*

7.3.1 *iso-Octane (2,2,4–trimethyl pentane)*, HPLC Grade, 0.01 % water.

7.3.2 *n-Heptane*, HPLC Grade, 0.01 % water.

7.3.3 *Methanol*~~, anhydrous, reagent grade.~~

~~7.4~~*, anhydrous, HPLC Grade, 0.01 % water.*

7.3.4 *Toluene*~~, reagent grade.~~

~~7.5~~*, HPLC Grade, 0.001 % water.*

~~[4] Available from ASTM as PCN 03-332030-12.~~

[4] Aluminum Oxide available from EMD Chemicals, Inc., P.O. Box 70, 480 Democrat Road, Gibbstown, NJ 08027 (Product Code AX0612), CAS Number:1344-28-1, 2.5 kg Chromatographic Grade Alumina (Al_2O_3) 80-200 mesh CAS 1344-28-1, Cat # EM-AXO612-3.

~~[5] Can be custom-made by any professional glassblower.~~

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual 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.

7.3.5 *Trichloroethylene*~~, boiling point 86.5 to 87.5°C.~~ , ACS Grade, 0.02 % water, 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 such as a fume hood. All working areas should be kept free of sparks, flames, or other sources of high temperature.

9. ~~Sampling and Sample Preparation~~

~~9.1Bulk samples taken in accordance with Practice D140 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*:~~

~~10.1.1Weigh to the nearest 0.01 g and place into a 2-L Erlenmeyer flask a sufficient quantity of asphalt~~ (Separation of Asphalt into Asphaltenes and Petrolenes (Maltenes)

9.1 Representative asphalt samples free of foreign substances will be collected in accordance with Practice D 140. Samples for testing can be transferred by chilling to facilitate fracturing the sample or by heating the sample until it becomes sufficiently fluid to pour. **Warning:** In no case shall the samples be heated more than 50°C above the expected softening point of the material, which is approximately 100°C.

9.2 Transfer to the nearest 0.001 g, 2.000 g of the asphalt into a tared 500-mL Erlenmeyer flask and record the mass of the asphalt, $M_{asphalt}$. Allow the sample to cool if transferred by heating and pouring prior to adding the iso-octane in the ratio of 100 mL of solvent per 1 g of sample. Add 200 mL ± 0.1 mL of HPLC grade iso-octane to the sample asphalt in the 500 mL Erlenmeyer flask.

9.3 In a fume hood of sufficient size to accommodate all required components of the apparatus and supplies used to perform this procedure, assemble a reflux apparatus in accordance with the setup shown in Fig. 1. Place a stir bar into the 500-mL Erlenmeyer flask containing the sample solution. Place the 500-mL Erlenmeyer flask on a stirrer/heater plate. Place an Allihn-type reflux condenser into the neck of 500-mL Erlenmeyer flask (Note 1). Ensure that the reflux apparatus is secured with lab clamps to a heavy ring stand or a laboratory mounting assembly housed in the fume hood by clamping both the neck of the 500-mL Erlenmeyer flask and the Allihn-type reflux condenser. Connect the Allihn-type reflux condenser to a cold water source using high pressure hose 15.9 mm (⅝ in. ID) and secure with hose clamps at all connections.

NOTE 1—Place Teflon flask-neck sleeves between sintered glass fittings to ensure easy disassembly of the reflux apparatus.

9.4 Slowly heat the sample solution on a stirrer/heater plate until the solution is observed to begin to reflux. Maintain the solution temperature near the boiling point of iso-octane (99°C). Begin to stir the contents of the flask at a moderate rate once the solution begins to reflux. Stir the contents of the 500-mL Erlenmeyer flask for 1 to 2 h or until no visual evidence of un-dissolved (un-digested) asphalt adheres to the sides of the flask. Once the sample has been observed to go into solution, stir the solution with refluxing for an additional 1 h (Note 2~~) so that it yields about 10 g of petrolenes as indicated by the following equation:~~

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

~~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~~ 2—Normally two hours is a sufficient amount of time to dissolve (digest) straight reduced asphalts, but for air blown or chemically modified asphalts, the dissolution time may need to be extended to three hours.

9.5 After the 2 to 3-h period of time required for refluxing with stirring, turn off the heater element of the stirrer/heater plate and allow the sample solution to cool with stirring. Continue to stir the cooled sample solution for an additional 2 h after cooling, at which time discontinue stirring, remove the Allihn-type reflux condenser, stopper the flask, and allow the sample solution to settle for 2 h prior to filtering (Note 3).

NOTE 3—At some time during the stirring process, after heating has been discontinued, rinse down the reflux assembly with 10 to 20 mL of iso-octane by dispensing solvent from the top of the Allihn-type reflux condenser into the flask using a squeeze bottle.

9.6 Set up a Büchner-style fritted-glass funnel (60-mL, ASTM 10–15-μL medium porosity) by placing the funnel on a 1-L suction flask with a filter ring. Attach the suction flask to a cold-trapped vacuum source with high pressure hose and hose clamps and secure the suction flask to a ring stand or mount rack with a clamp. Prior to filtration, tare the Büchner-style fritted-glass funnel.

9.7 Filter the sample solution prepared in 9.2-9.5, by quantitatively decanting the liquid contents of the sample solution from the 500-mL Erlenmeyer flask directly into the Büchner-style fritted-glass funnel while applying suction to the 1-L suction flask.

9.8 Quantitatively transfer the filtered decant, using a glass funnel, to a 1-L pear-shaped flask clamped to a ring stand, (this decant represents the first portion of petrolenes dissolved in iso-octane). Transfer the 1-L pear-shaped flask to a rotary evaporator and distill the iso-octane off of the petrolene fraction. Return the Büchner-style fritted-glass funnel to the suction flask.

9.9 Wash the asphaltenes which remain in the 500-mL Erlenmeyer flask with 100 mL of iso-octane. Place the 500-mL Erlenmeyer flask back onto the heater/stirrer plate and gently heat the contents of the Erlenmeyer flask for approximately 30 min with stirring to remove the remaining soluble materials potentially entrained in the asphaltene phase. Allow the solution to cool and settle (1 to 2 h). Quantitatively transfer all of the remaining contents of the 500-mL Erlenmeyer flask into the Büchner-style fritted-glass funnel while applying suction to the 1-L suction flask in order to filter off the asphaltenes. Toward the end of this transfer step, use a squirt bottle containing iso-octane to rinse the final remaining loose material from the 500-mL Erlenmeyer flask into the Büchner-style fritted-glass funnel. Set the empty 500-mL Erlenmeyer flask aside until 9.15 .

~~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.~~

9.10 Continue washing the asphaltene filter cake with additional iso-octane dispensed from a squeeze bottle until the eluate draining from the filter cake is observed to become colorless (Note 4).

NOTE 4—Do not allow the filter cake to dry and crack during this step. The filter cake should remain wet with iso-octane during this step to ensure a tight seal between the filter cake and the funnel surface.

9.11 When the eluate draining from the filter cake becomes colorless, discontinue washing and allow the remaining elution solvent to drain off of the filter so that the filter cake dries and cracks, then discontinue the vacuum.

9.12 Quantitatively transfer the eluate collected in the 1-L suction flask to the 1-L pear-shaped flask containing the petrolenes previously recovered from the filtration of the decant (see 9.7 and 9.8). Attach the 1-L pear-shaped flask to a rotary evaporator and distill off all but 10 to 15 mL of the iso-octane solvent to retain a liquid concentrate. Quantitatively transfer the concentrate to a tared 50-mL Borosilicate screw-cap vial. Remove the remaining iso-octane solvent present in the petrolene concentrate by placing the vial on a 35°C heated nitrogen gas-stream evaporator for 24 to 48 h.

9.13 After solvent removal via the nitrogen gas-stream evaporator, remove the petrolene sample from the nitrogen gas-stream evaporator and further dry the sample to a constant mass on an oil-bath rotary evaporator (22-mm Hg vacuum, 120°C, 30 min to 1 h). Record the mass of recovered pertolenes, $M_{petrolenes}$. Purge the vial with nitrogen or argon gas and store away from light for long-term storage.

9.14 Place the Büchner-style fritted-glass funnel containing the asphalt filter cake prepared in 9.9-9.11 onto a 1-L Erlenmeyer flask to hold the funnel upright. Place the Erlenmeyer flask with the Büchner-style fritted-glass funnel into a pre-heated 80°C nitrogen gas-purging vacuum oven. Purge the oven of air with nitrogen gas, then apply full vacuum to the oven. Dry the asphaltene filter cake for 2 to 3 h, or until a constant mass is achieved for the Büchner-style fritted-glass funnel plus asphaltene filter cake. Record the net mass of recovered asphaltenes, $M_{asphaltenes}$ by subtracting the tare weight of the Büchner-style fritted-glass funnel. Quantitatively transfer the dry contents (asphaltenes) from the Büchner-style fritted-glass funnel to a tared 25-mL Borosilicate screw-cap vial. Weigh the vial plus contents, subtract the vial tare, and record the mass of dry asphaltenes, $M_{asphaltenes}$ (dry). Purge the vial with nitrogen or argon gas and store away from light for long-term storage.