RECOVERY OF ASPHALT FROM SOLUTION BY ABSON OR ROTARY EVAPORATOR

LS-284 R33

3. APPARATUS

3.1 EXTRACTION

3.1.1 EXTRACTOR: Electrically operated, equipment with an electronic panel to provide accurate speed control from at least 1500 rpm up to 3600 rpm; complete with a minimum 3000 g capacity bowl, filter rings and a spout (drain) to collect the asphalt solution (Fig.1 of the LS) ....______

3.1.2 OVEN: A gravity convection oven of suitable capacity and capable of maintaining the temperature at 110 ± 5°C..______

3.1.3 RIFFLE SPLITTER: For splitting hot mix samples. Recommended width of the individual chutes is approximately 38 mm for all types of paving mix..______

3.1.4 FLASK: Flat bottom flask or clean container of sufficient size suitable to collect the asphalt solution..______

3.1.5 TRICHOLOROETHYLENE: Reagent grade trichloroethylene, more than 99.0% pure (>99%).....______

3.2 REMOVAL OF MINERAL FINES

3.2.1 HIGH SPEED CENTRIFUGE: Continuous unit, electrically operated, capable of exerting a minimum force of 3000 times gravity at a minimum speed of 9000 rpm, complete with a feed funnel equipped with a control valve, aluminum centrifuge cups and a spout (drain) to collect the asphalt solution (Fig. 2 of the LS) ....______

3.2.2 FLASKS: Flat bottom flasks or clean containers of sufficient size suitable to collect the asphalt solution..______

3.3 ROTARY EVAPORATOR RECOVERY

3.3.1 ROTARY EVAPORATOR: Complete with distillation flask with a minimum capacity of 1L, variable speed motor to rotate the distillation flask from a rate of at least 30 rpm up to at least 200 rpm...______

Vacuum and inert gas connections, inert gas flowmeter capable of indicating a gas flow up to 1000 ml/min, condenser, solvent recovery flask with a minimum capacity of 1L....................................______

And a heated oil bath capable to maintain the desired temperature from at least 25 ± 1°C up to at least 180 ± 1°C..______

The angle of the distillation flask from horizontal to the bath is set at approximately 30 ± 15°........______

The distillation flask when fully immersed, should be at a depth of approximately 40 mm.............______

Equipped with an electronic module capable to control and display heating bath temperature, flask rotation speed and vacuum level. (Fig 3 of the LS) ....______

3.3.2 VACUUM SYSTEM: Capable of maintaining a vacuum of 700 ± 5 mm of Hg (93.3 ± 0.7 kPA) down to 25 ± 5 mm of Hg (3.3 ± 0.7 kPa) ....______

3.3.3 NITROGEN GAS: Cylinder of nitrogen gas fitted with a pressure reducing valve......................______

3.4 ASH CONTENT

3.4.1 The apparatus is described in ASTM D8078

ASTM 5. Apparatus

5.1 *Crucible*—Porcelain or quartz-fiber, of sufficient size (normally 30 to 100-cm^3 capacity) and suitability for muffle furnace, may be used..______

5.2 *Crucible Tongs*—Sufficient size to transfer the crucible to and from the muffle furnace safely..______

5.3 *Balance,* capable of weighing a minimum of 200 g to within 0.001 g..______

5.4 *Muffle Furnace*—Electric resistance heated or microwave heated furnace, capable of maintaining a temperature of 900 + 25 °C.._______

5.5 *Desiccator,* of suitable size to hold the crucibles during the cooling process............................._______

LS 3.5 ABSON RECOVERY

3.5.1 The apparatus is described in ASTM D1856

ASTM 5. Apparatus

5.1 *Centrifuge,* batch unit capable of exerting a minimum centrifugal force of 770 times gravity or continuous unit capable of exerting a minimum force of 3000 times gravity................................._______

5.2 *Centrifuge Tubes*—A supply of wide-mouth bottles or centrifuge tubes may be used for the batch unit. A tube as illustrated in Fig. 1 of the ASTM has been found satisfactory for the continuous unit______

5.3 *Distillation Assembly,* as shown in Fig. 2 of the ASTM, and consisting of the following items: ..._______

5.3.1 *Extraction Flasks*—Two 250-ml, wide-mouth, heat resistant flasks, one for distillation and the other for the receiver.._______

5.3.2 *Glass Tubing*—Heat-resistant glass tubing, having 10-mm inside diameter and gooseneck shaped (as shown in Fig. 2) for connecting the flask to the condenser.._______

5.3.3 *Inlet Aeration Tube,* at least 180 mm in length, having a 6-mm outside diameter with a 10-mm bulb carrying six staggered side holes approximately 1.5 mm in diameter.._______

5.3.4 *Electric Heating Mantle,* with variable transformer, oil bath, or fluidized sand bath, to fit a 250-ml flask.._______

5.3.5 *Water-Jacketed Condenser,* Allihn type, with 200-mm minimum jacket length or equivalent._______

5.3.6 *Thermometer*—An ASTM Low Distillation Thermometer 7E or 7F, as specified, having a range from −2 to 300°C or 30 to 580°F, respectively, and conforming to the requirements in Specification E1.._______

5.3.7 *Gas Flowmeter,* as shown in Fig. 2, or any flowmeter capable of indicating a gas flow of up to 1000 ml/min.._______

5.3.8 *Corks,* No. 20, drilled as shown in Fig. 2. 5.3.9 *Flexible Elastomeric Tubing,* resistant to chlorinated solvents having sufficient length and size to connect the aeration tube to flowmeter, and equipped with a pinch clamp or stopcock to close aeration tube prior to introducing carbon dioxide...................._______

5.3.10 *Separatory Funnel,* (*Alternative Procedure*, see 9.3.1) 125-ml capacity..............................._______

4. PROCEDURES:

4.1 EXTRACTION

4.1.1 Obtain asphalt mixture samples in accordance with specified procedures............................_______

4.1.2 Place the asphalt mixture into a large flat pan and heat to achieve sufficient workability for quartering or splitting to the minimum sample size shown in Table 1 (Note 2) ...._______

A conventional oven maintained at a temperature of 110 + 5°C or a microwave oven (Note3) may be used for this purpose.._______

Table 1: Size of Test Sample

Mix Type	Designated Large Sieve Size (mm)	Minimum Mass of Sample (kg)
HL 2	2.36	0.5
SMA 9.5, Superpave 9.5	4.75	1.0
HL 1, HL 3, HL 3A, DFC, OFC, SMA 12.5, Superpave 12.5, 12.5FC1, 12.5FC2	9.5	1.5
HL 4 (Binder and Surface)	13.2	1.5
Superpave 19.0, SMA 19.0	12.5	2.0
HL 8, MDBC, HDBC	16.0	2.0
Superpave 25.0	19.0	3.0
Superpave 37.5	25.0	4.0

Note 2: The Designated Large Sieve is a sieve size specifically designated for testing purposes.

Note 3: Caution: Frequent mixing may be necessary to prevent localized overheating when using a microwave oven to heat paving mixtures. Also, the presence of metal particles in some mixtures may render the microwave oven unsafe for the heating of these mixtures. Mixes made with polymer modified asphalt cement will have to be heated to a temperature higher than 110°C to achieve sufficient workability. The actual temperature should be the minimum required to achieve this and should be determined to trial and error.

4.1.3 Once the desired workability is achieved, remove the sample from the oven and obtain a control sample close to the minimum quantity from Table 1 through either quartering or splitting..........._______

4.1.4 Transfer the sample into a tared pan, measure and record the mass of the mix...................._______

4.1.5 Clean and flush the extractor bowl, the inside of the extractor and the drain tube with fresh trichloroethylene.._______

4.1.6 Place the measured mass of mix in the bowl and distribute evenly......................................_______

Add enough trichloroethylene and break up the mix further until all the particles are submerged.._______

Make a note of the quantity of trichloroethylene used.._______

4.1.7 Place the dried filter paper and the lid on the extractor bowl and tighten the lid to 11.3 N-m of torque. Allow the sample to soak for 15 minutes..._______

4.1.8 At the end of the soaking period, ensure that the extractor's cover is secured and a clean flat bottom flask or another container of sufficient size is placed under the extractor drain tube to collect the asphalt solution (extract) ...._______

4.1.9 Start the extractor, slowly increasing the speed up to the maximum speed (Note 4) ...._______

Maintain this rate until the solvent ceases to flow and just drips.._______

Collect and retain the extract in the extract containers.._______

Note 4: The extractor's speed should be calibrated periodically. The maximum speed depends on the extractor used. Operate larger size extractors in accordance with manufacturer's instructions.

4.1.10 Stop the extractor..._______

4.1.11 Add the required quantity of trichloroethylene (Note 5) to the mix allowing it to soak for 10 minutes and repeat section 4.1.8 to 4.1.10..._______

Note 5: The required quantity is determined in 4.1.6.

4.1.12 Add the required quantity of trichloroethylene to the mix (Note 5). Allow to soak for 5 minutes and repeat section 4.1.8 to 4.1.10.._______
4.1.13 The extract container(s) may now contain the total extract and washings from each extraction cycle. Make a note of the volume obtained and procced to section 4.2 for removal of fines.........._______

4.2 REMOVAL OF FINES
4.2.1 Ensure that all components of the centrifuge are clean of any asphalt cement......................_______
4.2.2 Place a clean, previously weighed, centrifuge cup in the high-speed centrifuge and close the top cover.._______
4.2.3 Prepare 4 (four) clean flat bottom flasks or other containers, each able to hold the extract volume obtained in section 4.1.13.._______
4.2.4 Number and label each flask (container) (e.g. Flask #1 for 1 pass through centrifuge etc.) ….._______
4.2.5 Place Flask #1 under the centrifuge effluent tube to collect the asphalt solution (extract) ….._______
4.2.6 Start the centrifuge and allow it to reach constant operational speed of at least 9000 rpm (Note 6) ...._______
Note 6: The centrifuge's speed should be calibrated periodically. The maximum speed depends on the centrifuge used.
4.2.7 Close the control valve on the feed funnel.._______
4.2.8 Fill the feed funnel with extracted solution obtained in section 4.1.13................................_______
Open the control valve at the bottom of the funnel to allow a flow of 100 to 150 ml/min (Note 7)._______
Note 7: The centrifuge's speed should be calibrated periodically. The maximum speed depends on the centrifuge used.
4.2.9 Continue to top up the feed funnel until all the extracted solution has been transferred and passed through the centrifuge..._______
4.2.10 Close the control valve at the bottom of the funnel and stop the centrifuge......................._______
4.2.11 Remove Flask #1 and re-place it with Flask #2..._______
4.2.12 Start the centrifuge and allow it to reach constant operational speed of at least 900 rpm (Note 6) ...._______
4.2.13 Fill the feed funnel with contents of Flask #1 and repeat section 4.2.9 to 4.2.11.................._______
4.2.14 Remove Flask #2 and re-place it with Flask #3..._______
4.2.15 Fill the feed funnel with contents of Flask #3 and repeat section 4.2.9 to 4.2.11.................._______
4.2.16 Remove Flask #3 and re-place it with Flask #4..._______
4.2.17 Fill the feed funnel with contents of the Flask #1 and repeat section 4.2.9 to 4.2.11............_______
4.2.18 Once all the extracted solution passes through the centrifuge and collected in Flask #4 proceed to asphalt cement recovery..._______

4.3 ROTARY EVAPORATOR RECOVERY
4.3.1 Heat the oil bath to a temperature of 160 ± 1°C..._______
4.3.2 Circulate cold water through the condenser, connect the distillation and the solvent recovery flasks.._______
4.3.3 Set the vacuum to 640 ± 5 mm of Hg (85.3 ± 0.7 kPa) and draw enough asphalt solution from the sample container into the distillation flask through the sample line (Note 8) ...._______

Note 8: Fill the flask to around 60% of the capacity. The sample quantity will depend of the flask size.

4.3.4 Begin a nitrogen flow of approximately 500 ml/min through the system (Note 9) ....______

Begin rotating the distillation flask at approximately 40 rpm and lower flask into the oil bath.........______

Initially the immersion depth of the flask will be determined by the need to achieve a controlled solvent evaporation rate..______

The evaporation rate will be observed as a steady controlled stream of condensed solvent collected in the recovery flask...______

Note 9: Vacuum and nitrogen flow valves may require adjusting depending on location. Also, low flow values are recommended at the beginning of the rotary process when sample volume is large because of the possibility of back flow into the vacuum system.

4.3.5 When the amount of asphalt solution within the distillation flask appears low enough so that more solution may be added, discontinue the nitrogen flow...______

Draw another portion of asphalt solution from the sample container in the distillation flask and readjust the nitrogen flow (Note 10) ....______

Note 10: The equipment may be modified to allow a continuous flow of solution from the sample container into the distillation flask such that the volume in the distillation flask is maintained at approximately 60% of the capacity. The nitrogen flow is not started until all the solution has entered the distillation flak.

4.3.6 When the bulk of the solvent has been distilled from the asphalt and no obvious condensation is occurring on the condenser, immerse the distillation flask to maximum immersion depth.............______

A one to two minute delay before applying the vacuum is recommended.....................................______

4.3.7 Gradually, using 50-100 mm Hg increments, bring the vacuum to 160 ± 5 mm of Hg (21.3 ± 0.7 kPa) ....______

Increase the nitrogen flow to approximately 600 ml/min and the spin rate to about 45 rpm (Note 9) ....______

Hold or reduce the vacuum if foaming or a bubbly formation occurs...______

4.3.8 When foaming subsides, gradually bring the vacuum to 30 ± 5 mm of Hg (40 ± 0.7 kPa) ....______

Hold or reduce the vacuum if foaming or a bubbly formation occurs...______

4.3.9 Maintain the vacuum of 30 ± 5 mm of Hg (4.0 ± 0.7 kPa) until the drip from the condenser coils is approximately 5 drops per minute...______

4.3.10 Apply maximum vacuum...______

After the condensate drip rate returns to approximately 5 drops per minute, maintain, maintain this condition for 15 ± 1 minutes...______

4.3.11 At the end of the 15 ± 1 minutes, remove the distillation flask from the apparatus and cover the neck loosely with aluminium foil...______

CAUTION: Nitrogen flow should always be discontinued, and system opened to atmosphere before the vacuum pump is turned off to ensure that a positive pressure does not build up in the system.

4.3.12 Place the flask in an oven at 163 ± 2°C for 30 ± minutes (Note 11) ....______

Note 11: The heating period can be increased with an additional 30 ± 1 minutes to facilitate the pouring of the recovered asphalt cement.

4.3.13 After 30 minutes in the oven remove the flask, wipe the neck clean of any oily residue and pour into the containers prepared for subsequent testing of the asphalt cement..................................______

4.4 ABSON RECOVERY

4.4.1 After removal of fines, follow the procedure described in ASTM D1856.

ASTM 9. Procedure

9.1 The entire procedure, from the start of the extraction to the final recovery, must be completed within 8 h..........................._______

9.2 Centrifuge the solution from the previous extraction for a minimum of 30 min at 770 times gravity in either bottles or centrifuge tubes in the specified batch apparatus..........................._______

If a continuous centrifuge is used, the extract solution shall be charged at a rate not to exceed 150 ml/min, while the unit is operating at a speed calculated to produce a centrifugal force of not less than 3000 times gravity..........................._______

9.3 Concentrate the solution to about 200 ml by any primary distillation operation using a flask large enough to hold all the solution from the extraction..........................._______

Transfer the residue from the primary distillation flask, using several washes of solvent to rinse all of the residue into the 250-ml distillation flask..........................._______

Assemble the apparatus as shown in Fig. 2 of the ASTM, except position the bottom of the aeration tube above the surface of the solution..........................._______

Continue distillation until the temperature reaches 135°C (275°F), at which time lower the aeration tube so that the bulb is in contact with the bottom of the flask..........................._______

And introduce carbon dioxide gas at a low rate (approximately 100 ml/min) to provide agitation and prevent foaming..........................._______

If foaming or bumping occurs, introduce carbon dioxide intermittently at the beginning of the distillation at a rate of 100 ml/min to prevent this..........................._______

When the temperature reaches 157 to 160°C (315 to 320°F), increase the carbon dioxide gas flow to approximately 900 mL/min..........................._______

Maintain this gas flow rate for 10 min while also maintaining the temperature of the residue in the flask at 160 to 166°C (320 to 330°F) ...._______

If, after 10 min, dripping of condensed solvent from the delivery tube is still occurring, maintain the gas flow and temperature until 5 min after the dripping ceases in order to flush solvent vapors from the flask..........................._______

In no case shall the time of flow of carbon dioxide gas be less than 15 min. At the end of this period, discontinue gas flow and heat..........................._______

9.3.1 *Alternative Procedure*—Assemble the apparatus as shown in Fig. 2 with the separatory funnel in the thermometer hole in the cork. (It may be advantageous to insert the separatory funnel in a separate hole drilled in the cork stopper.) ...._______

Raise the aeration tube so that the bulb is above the surface of the solution..........................._______

Fill the separatory funnel with the centrifuged solution and open the stopcock to fill the flask approximately one half full of solvent mixture..........................._______

Apply low heat to the flask and start distillation..........................._______

Adjust the funnel stopcock to introduce fresh solvent at a rate that will keep the flask approximately one half full during distillation, adding additional solvent mixture to the funnel until all solvent has been introduced into the distillation flask..........................._______

Wash the solvent mixture container and funnel with fresh solvent to transfer all asphalt into the distillation flask..........................._______

Continue distillation until the temperature reaches 135°C (275°F) ...._______

At which time lower the aeration tube so that the bulb is in contact with the bottom of the flask and introduce carbon dioxide gas at a low rate (approximately 100 ml/min) to provide agitation and prevent foaming..._______

If foaming or bumping occurs, introduce carbon dioxide intermittently at the beginning of the distillation at a rate of 100 ml/min to prevent this.._______

When the temperature reaches 157 to 160°C (315 to 320°F), increase the carbon dioxide gas flow to approximately 900 mL/min..._______

Maintain this gas flow rate for 10 min while also maintaining the temperature of the residue in the flask at 160 to 166°C (320 to 330°F) ...._______

If, after 10 min, dripping of condensed solvent from the delivery tube is still occurring, maintain the gas flow and temperature until 5 min after the dripping ceases in order to flush solvent vapors from the flask..._______

In no case shall the time of flow of carbon dioxide gas be less than 15 min. At the end of this period, discontinue the gas flow and heat.._______

9.4 If the residue in the flask is highly viscous at 163°C (325°F), so that dispersion of the carbon dioxide in the residue is restricted and the recovered asphalt is expected to have a penetration at 25°C (77°F) of less than 30, maintain the carbon dioxide gas flow and temperature for 20 to 22 min.................._______

9.5 The recovered asphalt can be heated to reliquefy and portions taken for penetration, softening point, ductility, ash content, and kinematic and absolute viscosity determinations as required......_______

Ash content determinations shall be conducted on all recovered bitumens in accordance with Test Methods D2939 and reported with other test data on the recovered asphalt..............................._______

Ash contents of recovered asphalts greater than 1 % may affect the accuracy of the penetration, ductility, softening point, or viscosity tests..._______

NOTE 1—Determination of the weight of asphalt recovered serves as a check to assure that all of the solvent has been removed when this weight is compared with the weight of asphalt extracted as determined by Test Methods D2172. The weight of the recovered asphalt should be corrected for the ash content determined.

LS 4.5 ASH CONTENT

4.5.1 After recovery, follow the procedure described in ASTM D8078.

ASTM 6. Procedure

6.1 Preheat the muffle furnace to a temperature of 600 ± 10 °C prior to moving on to the next step in the procedure..._______

6.2 Heat an empty crucible at 600 ± 10 °C for 10 to 15 min. Cool the crucible to room temperature in the desiccator.._______

6.3 Place the empty crucible on the balance and record the weight (*Wc*) to the nearest 0.001 g. Tare the balance prior to placing the sample in the crucible.._______

6.4 Place approximately 2 to 3 g of the emulsified asphalt residue or asphalt to be tested into the crucible and record the sample weight (*Ws*) to the nearest 0.001 g..._______

If the sample is hot when put into the crucible, place the crucible with the sample into the desiccator to cool for 15 min prior to recording the sample weight (*Ws*) to the nearest 0.001 g........................._______

NOTE 2—A pre-ashing procedure may be used prior to placing the sample into the muffle furnace, if deemed necessary by the muffle furnace manufacturer or prior testing of the sample. This requires placing the crucible onto an acceptable stand above a Bunsen burner. Adjust the height of the flame until it is just high enough to touch the crucible. Heat the sample until the sample flames, and discontinue when the flaming of the sample subsides. Using tongs, remove the crucible from the stand and place it into the muffle furnace.

6.5 Place the crucible with the sample into the muffle furnace and incinerate the contents of the crucible for a minimum of 2 h, but no longer than 3 h.._______

6.6 Remove the crucible from the oven and allow to cool in a desiccator at room temperature for a minimum of 30 min.._______

6.7 Record the crucible and ash sample weight (*Wc+a*) to the nearest 0.001 g.............................._______

Return the crucible to the muffle furnace for an additional 30 ± 5 min.._______

6.8 Remove the crucible from the oven and allow to cool in a desiccator at room temperature for a minimum of 30 min.._______

6.9 Record the crucible and ash sample weight (*Wc+a*) to the nearest 0.001 g, and if the weight is within 0.002 g of that recorded in 6.6, it may be considered to be at constant weight.............................._______

6.10 If a constant weight has not been achieved, repeat steps 6.7 – 6.9 until a constant weight is reached.._______

7. Calculation

7.1 This equation is used to determine the percent ash content (*Ac*) from the asphalt or emulsified asphalt residue sample. The ash content is calculated as follows: ...._______

$$\%Ac = \frac{Wc{+}a - Wc}{Ws} \times 100 \qquad (1)$$

where:

Ac = ash content,
Wc+a = weight of crucible and ash sample,
Wc = weight of crucible, and
Ws = weight of sample (prior to testing).

Report the ash content (*Ac*) to the nearest 0.01 %.._______

LS

4.5.2 If the ash content of the first control sample is higher than 1.00 %, prepare and extract another control sample as per section 4.1 and perform the removal of fines by modifying section 4.2 for 6 centrifuge passes.._______

4.5.3 If the ash content of the second control sample is ≤ 1.00 % take a note of the number of centrifuge passes used.._______

4.5.4 Extract the remaining quantity of asphalt mixture (Note 12) as per section 4.1 perform the removal of fines as per section 4.2 using the number of centrifuge passes determined in section 4.5.2 or 4.5.3.._______

Note 12: The total amount of asphalt mixture needed is calculated based on the quantity of asphalt cement required for further testing and extractor's capacity.

4.5.5 Verify that the ash content is $\leq$ 1.00 % as per section 4.5.1 and proceed to further testing...._______

5. REPORTING AND FURTHER TESTING

5.1 DO NOT PROCEED TO FURTHER TESTING IF THE ASH CONTENT IS HIGHER THAN 1.00 %!.........._______

5.2 If ash content is $\leq$ 1.00 %, report ash content, number of centrifuge passes used and proceed to further testing..._______

6. SAFETY

6.1 Read and follow the instructions on the product label and review the Safety Data Sheet (SDS) to understand and avoid the hazards associated with trichloroethylene..._______

6.2 The personal protective equipment and engineering controls include but are not limited to the following (follow the local, provincial and federal safety regulations):

Fume hoods, chemical resistant gloves, heat protective gloves, safety glasses, googles, appropriate respirators, lab coats etc..._______

7. GENERAL NOTES

7.1 All equipment used in this method shall be calibrated periodically..._______

7.2 The Rotary Evaporator Recover using nitrogen is the preferred method................................._______

COMMENTS