Recovery of Asphalt From Solution by Abson Method ASTM D1856 – 09(Reapproved 2015)

4. Significance and Use

4.1 The asphalt should be extracted from the aggregate asphalt mixture in accordance with Method A of Test Methods D2172 (centrifuge method) as there is some experimental evidence that the recovered asphalt may have slightly lower penetration values when recovered from solutions obtained from hot extraction methods.

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.....

6. Reagents and Materials

6.1 *Carbon Dioxide Gas*—A pressurized tank, with pressure-reducing valve or other convenient source.

6.2 *Solvents:* .....

6.2.1 The solvent for extracting the asphalt from mixtures may be trichloroethylene, conforming with Specification D4080.....

6.2.2 The solvent for extracting the asphalt from mixtures may be Normal Propyl Bromides (nPB). This solvent should conform to Specification D6368.....

6.2.3 The solvent for extracting the asphalt from mixtures may be reagent grade Methylene Chloride

7. Precautions

7.1 **Warning**—The solvent listed in 6.2 should be used only under a hood or with an effective surface exhaust system in a well-ventilated area, since they are toxic to various degrees.....

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Consult the current Threshold Limit Concentration Committee of the American Conference of Governmental Industrial Hygienists for the current threshold limit values.....
7.2 These solvents in the presence of heat and moisture may be hydrolyzed to form acids that are extremely corrosive to certain metals, particularly when subject to contact over lengthy periods of time. Proper precautions should be taken to not allow these solvents to remain in small quantities in the effluent tanks of aluminum vacuum extractors.....
7.3 Exposure of these solvents or their vapors to high temperatures such as contact with flames, hot glowing surfaces, or electric arcs can produce decomposition products such as hydrogen chloride.
Steel drums containing these solvents should be stored in a cool, dry location, kept tightly sealed, and opened as infrequently as possible.....
The hydrogen chloride in decomposed solvent may harden an asphalt during the extraction and recovery test.....

8. Sample

8.1 The sample shall consist of the solution from a previous extraction by Method A of Test Methods D2172 of asphalt aggregate mixture of sufficient quantity to result in 75 to 100 g of recovered bitumen.....
More or less quantities of bitumen may be recovered; however, the properties of the recovered bitumen may not be in agreement with those recovered of the aforementioned quantities, and in case of a disagreement, 75 to 100 g should be recovered.....
8.2 During the extraction process, it is important that all of the asphalt in the mixture be extracted as there could be some selective solvency of the asphalt and the harder, more viscous components of the asphalt might be left in the mixture if extraction is not carried to completion.....
8.3 Since heavy petroleum distillates such as mineral spirits or kerosine will affect the properties of the recovered asphalt, it is important to avoid the use of such solvents in cleaning the extraction and recovery apparatus and use only trichloroethylene for cleaning.....
Residues of heavy petroleum solvents on the equipment may contaminate the recovered asphalt and affect its test properties.....
It is also necessary to use new filter rings, clean felt pads, or other uncontaminated filtering media in the extraction process to avoid contamination from a previous extraction.....
8.4 Generally, the bitumen in mixtures will progressively harden when exposed to air, particularly if the mixtures are in a loose condition.....
Therefore, it is important to protect bituminous mixtures from exposure to air and preferably to store them in airtight containers at a temperature below 0°C (32°F) until they can be tested.....
When samples of bituminous mixture are warmed for preparing representative proportions for extraction tests in accordance with Method A of Test Methods D2172, they should be placed in an oven in covered containers and heated to a maximum temperature of 110°C (230°F) for the minimum time to obtain workability, but no longer than 30 min.....
If the samples have been stored at a low temperature, they should be allowed to reach room temperature before placing them in the oven.....

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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, 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.....

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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.

Comments