



Designation: D 5404 – 02

Standard Practice for Recovery of Asphalt from Solution Using the Rotary Evaporator¹

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1. Scope

1.1 This practice is intended to recover asphalt from a solvent using the rotary evaporator to ensure that changes in the asphalt properties during the recovery process are minimized.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

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.*

2. Referenced Documents

2.1 ASTM Standards:

D 92 Test Method for Flash and Fire Points by Cleveland Open Cup²

D 1856 Test Method for Recovery of Asphalt from Solution by Abson Method³

D 2939 Test Methods for Emulsified Bitumens Used as Protective Coatings⁴

D 6368 Specification for Vapor-Degreasing Grade and General Solvent Grade Normal-Propyl Bromide⁵

2.2 Federal Specification:

O-T-634 (latest) Trichloroethylene, Technical⁶

3. Summary of Practice

3.1 The solution of solvent and asphalt from a prior extraction is distilled by partially immersing the rotating distillation flask of the rotary evaporator in a heated oil bath while the solution is subjected to a partial vacuum and a flow of nitrogen gas or carbon dioxide gas. The recovered asphalt can then be

subjected to testing as required.

4. Significance and Use

4.1 In order to determine the characteristics of the asphalt in an asphalt paving mixture, it is necessary to extract the asphalt from the aggregate by means of a suitable solvent and then to recover the asphalt from the solvent without significantly changing the asphalt's properties. The asphalt recovered from the solvent by this practice can be tested using the same methods as for the original asphalt cement, and comparisons between the properties of the original and recovered asphalt can be made.

5. Apparatus

5.1 *Rotary Evaporator* (see Fig. 1), equipped with a distillation flask, a variable speed motor capable of rotating the distillation flask at a rate of at least 50 rpm, condenser, solvent recovery flask, and heated oil bath. The angle of the distillation flask from the horizontal to the bath is set at approximately 15°. The distillation flask (Note 1), when fully immersed, should be at a depth of approximately 40 mm (1.5 in.).⁷

NOTE 1—A flask having a 2000 mL capacity is recommended.

5.2 *Manometer or Vacuum Gage*, suitable for measuring the specified vacuum.

5.3 *Gas Flowmeter*, capable of indicating a gas flow of up to 1000 mL/min.

5.4 *Sample Container*, having adequate volume to hold the sample and added solvent.

5.5 *Vacuum System*, capable of maintaining a vacuum to within ± 0.7 kPa (± 5 mm Hg) of the desired level up to and including 80 kPa (600 mm Hg).

6. Reagents and Materials

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

NOTE 2—Different flow rates may be required depending on whether nitrogen gas or carbon dioxide gas is used.

6.2 *Oil*—The oil for the heated oil bath should be USP

¹ This practice is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.25 on Analysis of Bituminous Mixtures.

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² *Annual Book of ASTM Standards*, Vol 05.01.

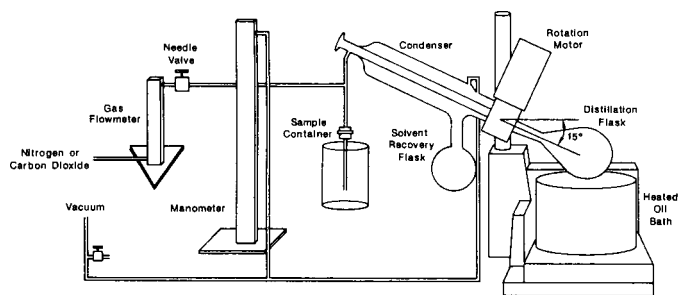
³ *Annual Book of ASTM Standards*, Vol 04.03.

⁴ *Annual Book of ASTM Standards*, Vol 04.04.

⁵ *Annual Book of ASTM Standards*, Vol 15.05.

⁶ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

⁷ The Buchi Rotavapor RE-111A, or its equivalent, has been found satisfactory for this purpose. The Buchi Rotavapor is available from Fischer Scientific, Pittsburgh, PA.



NOTE—It is important that the needle valve is at location shown; not ahead of the flowmeter.

FIG. 1 Rotary Evaporator and Recovery System

White Oil or Silicone Fluid SWS-101 with flash point above 215°C (420°F) or an equivalent. The flash point is determined in accordance with Test Method D 92.

6.3 Solvents:

6.3.1 *Trichloroethylene and Methylene Chloride*—The solvent for extracting the asphalt from mixtures should be reagent grade trichloroethylene or methylene chloride. A technical grade of trichloroethylene may be used, but it is recommended that for each new supply of solvent, a blank should be run on an asphalt with known properties.

6.3.2 *Normal Propyl Bromides (nPB)*—The solvent for extracting the asphalt from the mixtures should conform to Specification D 6368. Because there may be some interactions due to differences in nPB products, it is recommended that for a new supply of solvent, a blank should be run on an asphalt with known properties.

7. Precautions

7.1 **Caution**—The solvents listed in 6.3 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. Consult the current Threshold Limit Concentration Committee of the American Conference of Governmental Industrial Hygienists⁸ 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 Preparation

8.1 The sample shall be obtained and handled in accordance with Test Method D 1856. This includes the procedure for

centrifuging the solution from a previous extraction.

9. Procedure

9.1 Heat the oil bath to a temperature of $140 \pm 3^\circ\text{C}$ ($285 \pm 5^\circ\text{F}$). Circulate cold water through the condenser.

9.2 Apply a vacuum of 5.3 ± 0.7 kPa (40 ± 5 mm Hg) below atmospheric pressure and draw approximately 600 mL of asphalt solution from the sample container into the distillation flask by way of the sample line. Begin a nitrogen or carbon dioxide flow of approximately 500 mL/min through the system (Note 3). Begin rotating the distillation flask (at approximately 40 rpm) and lower the 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 correct rate of evaporation can be observed as a steady controlled stream of condensed solvent being collected in the recovery flask.

NOTE 3—The vacuum and nitrogen or carbon dioxide flow values may need to be adjusted depending on the location. Also, low flow values are recommended at the beginning of the rotary process when the sample volume is large because of the possibility of back flow into the vacuum system. The possibility of back flow can also be minimized by regulating the depth of immersion of the flask.

9.3 When the amount of asphalt solution within the distillation flask appears low enough so that more solution may be added, discontinue the nitrogen or carbon dioxide flow. Draw the remaining asphalt solution from the sample container into the distillation flask and readjust the nitrogen or carbon dioxide flow (Note 4).

NOTE 4—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 600 mL. The nitrogen or carbon dioxide flow is not started until all the solution has entered the distillation flask.

9.4 When the bulk of the solvent has been distilled from the asphalt and no obvious condensation is occurring on the condenser, immerse the flask to the recommended maximum immersion depth of approximately 40 mm (1.5 in.). Slowly apply a vacuum of 80.0 ± 0.7 kPa (600 ± 5 mm Hg) below atmospheric pressure. Increase nitrogen or carbon dioxide flow to approximately 600 mL/min and the spin rate of the distillation flask to about 45 rpm (Note 5). A 2 min delay before applying the vacuum is recommended. Hold or reduce vacuum if foaming or a bubbly formation occurs. When foaming subsides apply maximum vacuum. Maintain this condition for 15 ± 1 min.

NOTE 5—A faster flask spin rate exposes more surface area of the asphalt and, hence, dislodges more traces of solvent from the asphalt. The rotation speed of the flask may also be varied or the flask rotation may be stopped to enhance the removal of solvent.

NOTE 6—Due to the cooling effect of the increased nitrogen or carbon dioxide flow, an increase in the temperature of the oil bath is generally needed to maintain a constant sample temperature. Experience has shown that a typical oil bath temperature range of 300 to 315°F (148.9 to 157.2°C) is satisfactory for this purpose.

9.5 At the end of the 15 min period, remove the distillation flask from the apparatus and wipe the flask clean of oil. Pour the asphalt into a proper size container. If needed, the flask may be inverted and placed into an oven at $165 \pm 1^\circ\text{C}$ ($329 \pm 2^\circ\text{F}$) for 10 to 15 min to cause the asphalt to flow into the container.

⁸ Available from American Conference of Governmental Industrial Hygienists, 1330 Kemper Meadow Drive, Cincinnati, OH 45240, (513) 742-2020, (www.acgi-h.org).



The asphalt should not be allowed to be distributed over the interior of the flask.

9.6 Portions of the recovered asphalt, while still in a liquid state, can be taken for penetration, softening point, ductility, and kinematic and absolute viscosity determinations as required. If ash content determinations are to be conducted, they shall be determined in accordance with Methods D 2939 and reported with other test data on the recovered asphalt.

NOTE 7—Ash contents of recovered asphalts greater than 1 % may affect the accuracy of the penetration, ductility, softening point, or viscosity tests.

10. Keywords

10.1 bituminous paving mixtures; recovery; rotary evaporator

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