



Designation: D5404/D5404M – 21

# Standard Practice for Recovery of Asphalt Binder from Solution Using the Rotary Evaporator<sup>1</sup>

This standard is issued under the fixed designation D5404/D5404M; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 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 either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.2.1 Residual pressure measurements are shown in both the SI unit of kPa and the commonly used non-standard equivalent unit of “mm of Hg.”

1.2.2 Measurements of volume and mass are only given in SI units because they are the only units typically used in practice when performing this standard practice.

1.3 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

<sup>1</sup> 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 Asphalt Mixtures.

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## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D1856 Test Method for Recovery of Asphalt from Solution by Abson Method

D2939 Test Methods for Emulsified Bitumens Used as Protective Coatings (Withdrawn 2012)<sup>3</sup>

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials

D6368 Specification for Vapor-Degreasing Solvents Based on *normal*-Propyl Bromide and Technical Grade *normal*-Propyl Bromide

E1 Specification for ASTM Liquid-in-Glass Thermometers

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

## 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

<sup>2</sup> 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* volume information, refer to the standard’s Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

methods as for the original asphalt cement, and comparisons between the properties of the original and recovered asphalt can be made.

NOTE 1—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Specification D3666 are generally considered capable of competent and objective testing, sampling, inspection, etc. Users of this standard are cautioned that compliance with Specification D3666 alone does not completely ensure reliable results. Reliable results depend on many factors; following the suggestions of Specification D3666 or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.

## 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 2), when fully immersed, should be at a depth of approximately 40 mm [1.5 in.].

NOTE 2—A flask having a 2000-mL capacity is recommended.

5.2 *Thermometer*, used for verifying the temperature of the oil bath, with a range of at least 135 to 145 °C [275 to 298 °F] and an accuracy of ±1.5 °C [±2.5 °F]. The thermometer shall be one of the following:

5.2.1 A liquid-in-glass thermometer conforming to the requirements of Specification E1 or E2251, readable to 0.1 °C [0.2 °F].

5.2.2 A digital metal stem thermometer with a thermocouple sensor and a stem length of at least 150 mm [6 in.] paired with an appropriate meter capable of displaying temperature to the nearest 0.1 °C [0.2 °F]. The sensor shall be encased in a

stainless steel sheath that has a length of at least 150 mm [6 in.] and a minimum immersion depth of not more than 40 mm [1.6 in.].

5.2.3 A Class A Pt-100 RTD (Specification E1137/E1137M) sensor with a three- or four-wire configuration at the connection terminal and paired with an appropriate meter capable of displaying the temperature to the nearest 0.1 °C [0.2 °F]. The sensor shall be encased in a stainless steel sheath that has a length of at least 150 mm [6 in.] and a minimum immersion depth of not more than 40 mm [1.6 in.].

5.3 *Manometer or Vacuum Gauge*, suitable for measuring the specified vacuum.

5.4 *Gas Flow Meter*, capable of indicating a gas flow of up to 1000 mL/min.

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

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

5.7 *Oven*, capable of maintain a temperature of 165 ± 5 °C [329 ± 10 °F].

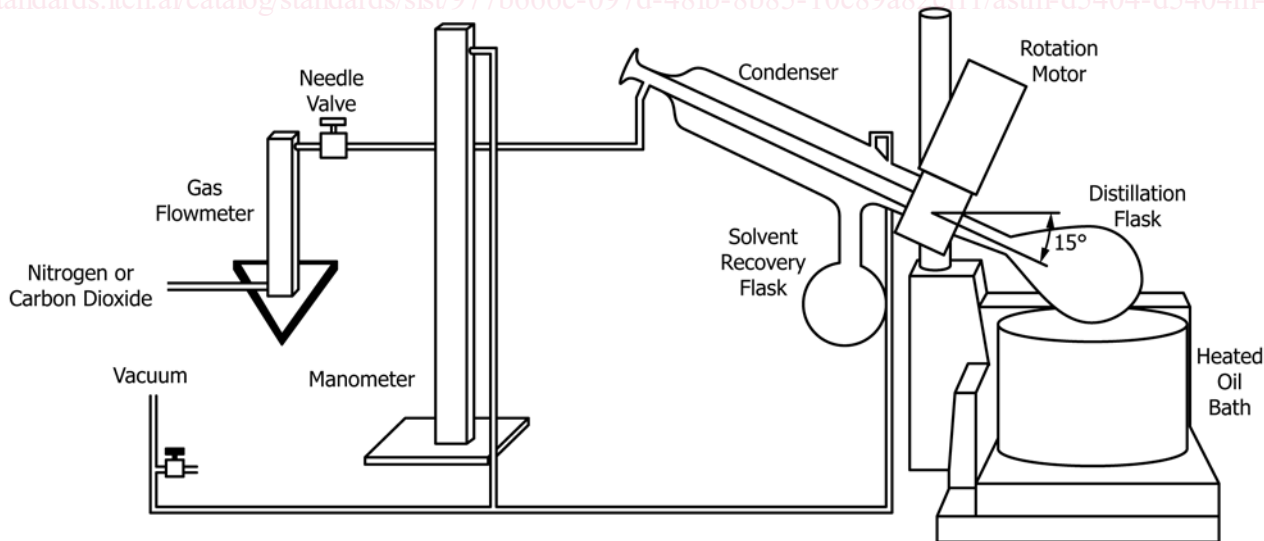
## 6. Reagents and Materials

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

NOTE 3—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 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 D92.

<https://standards.iteh.ai/catalog/standards/sist/977b666c-097d-48fb-8b85-10c89a82c1f1/astm-d5404-d5404m-21>



It is important that the needle valve is located as shown instead of being placed ahead of the flow meter.

FIG. 1 Rotary Evaporator and Recovery System

### 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 D6368. 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 Hygienists<sup>4</sup> 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. Standardization and Verification

8.1 The following standardizations and verifications shall be made every twelve months, and records of these verifications shall be maintained:

8.1.1 *Vacuum*—Using a calibrated manometer or vacuum gauge, verify that the equipment is capable of achieving and holding a vacuum of  $80.0 \pm 0.7$  kPa [ $600 \pm 5$  mm of Hg] down to  $5.3 \pm 0.7$  kPa [ $40 \pm 5$  mm of Hg].

8.1.2 *Gas Flow Meter*—Using a calibrated flow meter, verify the gas flow meter at 500 mL/min or other flow rates being used (Note 5).

8.1.3 *Thermometer*—Using a calibrated reference thermometer, standardize the thermometer at  $140$  °C [ $285$  °F] or at temperatures bracketing  $140$  °C [ $285$  °F].

## 9. Sample Preparation

9.1 The sample shall be obtained and handled in accordance with Test Method D1856. This includes the procedure for centrifuging the solution from a previous extraction.

<sup>4</sup> Available from American Conference of Governmental Industrial Hygienists, 1330 Kemper Meadow Drive, Cincinnati, OH 45240, (513) 742-2020, <http://www.acgih.org>.

## 10. Procedure

10.1 Heat the oil bath to a temperature of  $140 \pm 3$  °C [ $285 \pm 5$  °F] (Note 4). Using the thermometer, verify the temperature of the oil bath near the location where the distillation flask will be immersed. Circulate cold water through the condenser.

NOTE 4—Due to the high temperature of the oil bath and no circulation of the oil, it is not recommended to leave the oil bath on for an extended period of time without immersing and rotating the distillation flask.

10.2 Apply a vacuum of  $5.3 \pm 0.7$  kPa [ $40 \pm 5$  mm of 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 gas or carbon dioxide gas flow of approximately 500 mL/min through the system (Note 5). 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 5—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 backflow into the vacuum system. The possibility of backflow can also be minimized by regulating the depth of immersion of the flask.

10.3 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 6).

NOTE 6—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.

10.4 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  $80.0 \pm 0.7$  kPa [ $600 \pm 5$  mm of 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 7). Reduce the vacuum if foaming or a bubbly formation occurs. When foaming subsides, proceed once again with slowly increasing the vacuum until  $80.0 \pm 0.7$  kPa [ $600 \pm 5$  mm of Hg] below atmospheric pressure is reached. Maintain this condition for  $10 \pm 1$  min.

NOTE 7—It is recommended that the flask be immersed for up to 2 min before slowly applying the vacuum.

NOTE 8—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 9—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