Designation: D7906 – 22

Standard Practice for Recovery of Asphalt Binder from Solution Using Toluene and the Rotary Evaporator[1]

This standard is issued under the fixed designation D7906; 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

1.1 This practice is intended to be used 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 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

D2172/D2172M Test Methods for Quantitative Extraction of Asphalt Binder from Asphalt Mixtures

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

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

3. Summary of Practice

3.1 The solution of solvent and asphalt from a prior extraction is first 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. During the final stage of the process, vacuum is increased and nitrogen or carbon dioxide gas is introduced to the solution to remove remaining toluene and prevent further aging of the asphalt. The recovered asphalt can then be tested as required.

4. Significance and Use

4.1 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.

4.2 Users are cautioned that the recovered binder properties from an asphalt mixture sample may not exactly represent the properties of the original asphalt binder due to factors outside of laboratory control such as aging, contamination, and molecular changes caused by exposure to heat and solvents. Consequently, recovered asphalt binder properties should not be used as a basis for acceptance.

NOTE 1—The quality of the results produced by this is 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, and so forth. Users of this practice 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 distillation flask, variable speed motor capable of rotating the distillation flask at a rate of at least 40 rpm, condenser (Note 2), solvent recovery flask, and heated oil bath. The angle of the

[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 Asphalt Mixtures.

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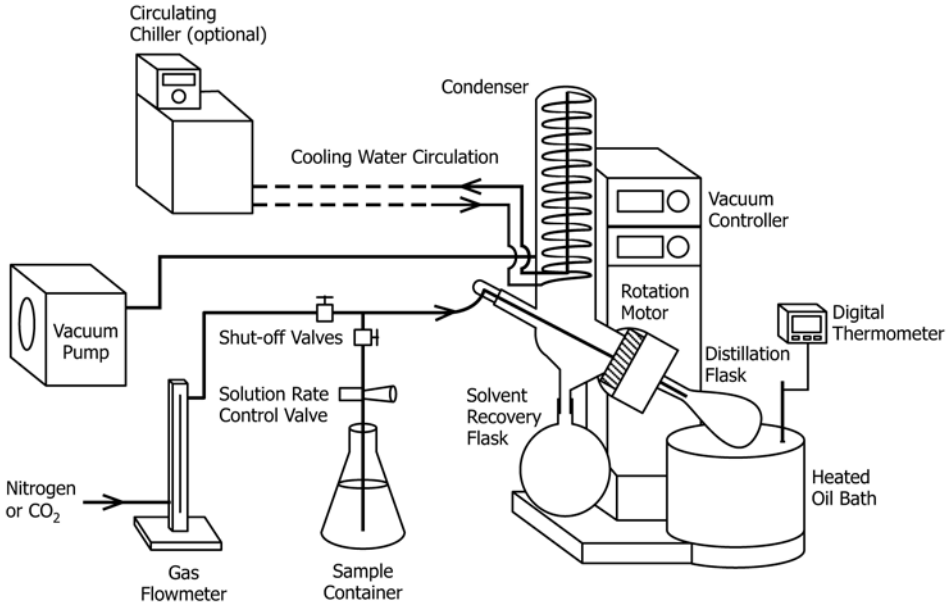
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



NOTE 1—The vacuum pump and vacuum controller are recommended for maintaining the required vacuum in 5.1. The diagram displays a Büchi model rotary evaporator.

FIG. 1 Rotary Evaporator and Recovery System

distillation flask from the horizontal to the bath is set at approximately 30 ± 10°. The distillation flask when fully immersed in the oil bath should be at a depth of approximately 40 mm.

5.2 *Distillation and Receiving Flasks,* a distillation flask with a minimum capacity of 1 L is required for the sufficient recovery of asphalt binder. It is recommended for the receiving flask to have a minimum capacity of 1 L to maximize the amount of solvent recovery.

NOTE 2—A vertical condenser is recommended for solvents with higher boiling points such as toluene.

5.3 *Vacuum Pump and Controller,* capable of maintaining a vacuum of 72.0 ± 0.7 kPa down to 6.7 ± 0.7 kPa.

5.3.1 *Vacuum Controller,* built into rotary evaporator system controlling and measuring the specified vacuum.

NOTE 3—Alternatively, a house vacuum system and vacuum gauge may be used if the rotary evaporator system does not include vacuum pump and controller.

5.4 *Gas Flow Meter,* capable of indicating a gas flow of up to 1 L/min.

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

5.6 *Thermometer,* capable of measuring the oil bath temperature up to 180 °C with an accuracy of ±1.5 °C (2.5 °F) at a depth of 40 ± 5 mm. The thermometer shall be one of the following:

5.6.1 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.).

5.6.2 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.).

5.7 *Circulating Chiller,* capable of cooling circulating water to increase condensation of toluene.

NOTE 4—Circulating chiller is not required if condensing water is normally below 25 °C, as per manufacturer's recommendation. Colder circulating water temperatures below 25 °C can increase the rate of condensation of the toluene vapor.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all 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.[3] 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.

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

6.3 *Oil*—The oil for the heated oil bath should be USP White Oil, Silicone Fluid SWS-101 with flash point above

[3] 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 *Analar 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.

215 °C or an equivalent (Note 5) or high-temperature cooking oil. The flash point is determined in accordance with Test Method D92.

NOTE 5—Silicone oil will expand when heated.

6.4 *Toluene*—The solvent for extracting the asphalt from the mixtures should be an ACS grade that does not have 1,2-epoxybutane as a stabilizer. The stabilizer can cause problems with asphalt containing acids.

7. Hazards

7.1 Toluene is a toxic solvent and should be used under a hood in a well-ventilated area to avoid high vapor levels. Consult the current Threshold Limit Concentration Committee of the American Conference of Governmental Industrial Hygienists for the current threshold limit values.

7.2 Toluene is a highly flammable substance; avoid exposure of the solvent to open flames and sources of ignition. Store container in a cool, dry location that is well ventilated. Keep container closed and sealed until ready for use. Refer to the manufacturer's material safety data sheet (MSDS) for more detailed information.

8. Sampling

8.1 The sample shall be obtained and handled in accordance with Test Method A in Test Methods D2172/D2172M, with the exception that toluene will be used as the solvent. Included in the procedure is a micro-centrifugation of the solution after extraction to remove further fine particles.

9. Calibration

9.1 *Vacuum*—Using a calibrated manometer or vacuum gauge, annually check vacuum pressure capable of achieving and holding a vacuum of 72.0 ± 0.7 kPa down to 6.7 ± 0.7 kPa.

9.2 *Gas Flow Meter*—should be able to read up to 1 L/min and calibrated annually at 600 mL/min with a calibrated flow meter.

9.3 *Thermometer*—Calibrate thermometer with calibrated reference thermometer at least annually to read bath temperature at sample level accurately.

10. Procedure

10.1 *Primary Distillation:*

10.1.1 Heat the oil bath to a temperature of 150 ± 5 °C. Circulate cooling water through the condenser (Note 6).

NOTE 6—The silicone oil bath temperature should be checked at the approximate location of the submerged flask.

10.1.2 While the flask is above the oil bath, draw in the asphalt-toluene solution into the distillation flask by applying a vacuum of 72.0 ± 0.7 kPa. Draw 200 to 300 mL of asphalt-toluene solution into the flask (Note 7). An excessive amount solution in the flask may result in a backflow of solution into the condenser.

NOTE 7—The amount of solution may be increased depending upon the size of the distillation flask.

10.1.3 Begin rotating the flask at 40 rpm and lower the flask into the oil. Immerse the flask approximately 40 mm into the oil bath (Note 8). Gradually increase vacuum to 45.3 ± 0.7 kPa without causing the solution to backflow into the condenser while applying vacuum.

NOTE 8—The rotation speed of the flask may need to be reduced to 30 rpm for stiffer materials. The spin rate should be lowered to expose more surface area of the asphalt and dislodge more traces of the solvent from the asphalt. The 40 rpm speed could cause the asphalt to not turn in the flask, such as with a stiff binder such as PG xx-16, xx-10, recycled asphalt pavement (RAP), or recycled asphalt shingles (RAS). Exposure of a new asphalt surface enhances the diffusion of the solvent.

10.1.4 Maintain 200 to 300 mL of asphalt-toluene solution in the flask until all the solution has entered the flask. Continuously and slowly feed the solution into the distillation flask. If this cannot be done, add the solution every 3 to 5 min (Note 9).

NOTE 9—Be cautious to not let the asphalt solution go below 200 mL until all the solution has been introduced to the rotary evaporator. This could cause overheating and premature aging of the asphalt if the volume falls below 200 mL.

10.1.5 Remove the recovered solvent in the recovery flask when needed to prevent overfill solvent into the condenser (Note 10). When emptying the recovery flask, stop intake of the solution into the distillation flask and release the vacuum. Release the vacuum slowly as not to cause the asphalt to backflow into the condenser.

NOTE 10—Recovered solvent should not be reused with other extractions because it contains trace elements of the previous extraction.

10.2 *Final Distillation to Remove Remaining Solvent:*

10.2.1 After the bulk of the solvent has been distilled from the asphalt and the drip from the condenser coils is approximately one drop every 15 s, slowly increase vacuum to 6.7 ± 0.7 kPa. Hold or release vacuum if foaming or bubbling occurs. When foaming subsides, continue to increase vacuum until target vacuum of 6.7 ± 0.7 kPa is achieved.

10.2.2 After the condensate drip rate returns to approximately one drop every 15 s, turn on the nitrogen or carbon dioxide gas at a flow of 600 mL/min. Maintain this condition of nitrogen or carbon dioxide gas purge and maximum vacuum for 45 ± 2 min (Note 11 and Note 12).

NOTE 11—The purpose of the nitrogen or carbon dioxide is to remove the oxygen from the asphalt to help reduce premature aging of the sample.

NOTE 12—Because of the cooling effect of the nitrogen or carbon dioxide, an increase in the oil bath temperature is generally needed to maintain a constant sample temperature.

10.2.3 At the end of the 45 min, wipe the distillation flask clean of oil and remove from the apparatus. Inverting the flask, place into the oven at 165 ± 1 °C to allow the asphalt binder to drain into a sample cup (Note 13).

NOTE 13—This temperature and time is typical for asphalts such as PG 58-34s to PG 82-16s. The temperature or time or both for draining the flask will need to be increased to 180 °C for stiffer RAP and 200 °C for RAS. Not recommended to exceed 200 °C.

11. Keywords

11.1 bituminous paving mixtures; RAP; RAS; recovery; recycled asphalt pavement; recycled asphalt shingles; rotary evaporator