Designation: D7944 – 15

Standard Practice for Recovery of Emulsified Asphalt Residue Using a Vacuum Oven[1]

This standard is issued under the fixed designation D7944; 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 suitable for recovery of the residue of emulsified asphalts composed principally of a semisolid or liquid asphaltic base, water and an emulsifying agent. Asphalt base may be pre-modified with polymeric modifiers or latex polymer modifiers may be incorporated into the emulsified asphalt through co-milling or post emulsified asphalt production blending.

1.2 *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:*[2]

D244 Test Methods and Practices for Emulsified Asphalts

D7175 Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer

D7405 Test Method for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binder Using a Dynamic Shear Rheometer

2.2 *AASHTO Standards:*[3]

R28 Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)

PP 72 Standard Practice for Recovery Residue from Emulsified Asphalt Using Low-Temperature Evaporative Techniques

3. Summary of Practice

3.1 A sample of emulsified asphalt is spread in a thin film on a silicone rubber mat and conditioned in a vacuum oven or other suitable vacuum chamber to remove the water.

4. Significance and Use

4.1 This practice can be used to obtain residues of emulsified asphalts. The residue may be used for further testing.

4.2 As approximately 5 g of residue is typically obtained, limited testing is possible.

NOTE 1—Rheological characterization as described in Test Methods D7175 and D7405 are suitable test methods. Additional test methods are being developed suited to the small amounts of residue obtained.

5. Apparatus

5.1 Silicone rubber mat[4] approximately 30 by 20 by 0.3 cm (approximately 12 by 9 by 0.15 in.), minimum of 40A durometer.

5.2 *Supports for Silicone Rubber Mat*—Provide adequate support of the silicone rubber mat during draw down of the film, while transporting to the vacuum oven or other suitable vacuum conditioning chamber, and while recovering the emulsified asphalt residue. The procedure uses two separate supports, one at ambient temperature for draw down and transfer, and a second to support the silicone rubber mat and emulsified asphalt during residue recovery. Adequate supports include but are not limited to a metal plate or tile with dimensions that are larger than the silicone rubber mat.

5.3 Draw down applicator[5] with the ability to draw down a wet film approximately 0.38 mm (15 mils) in thickness. (See Fig. 1.)

[1] This practice is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.42 on Emulsified Asphalt Test.

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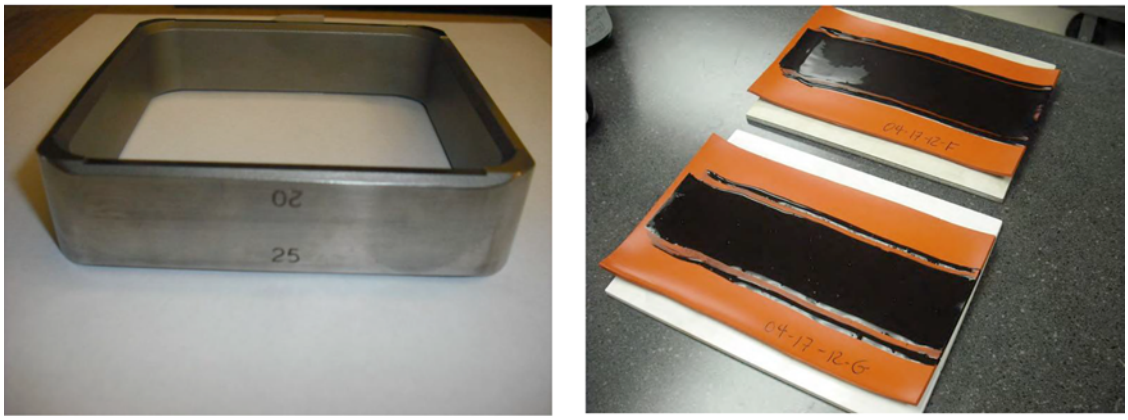
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001, http://www.transportation.org.

[4] The sole source of supply of the apparatus (referred to as "Silicone Rubber Sheeting" in catalog) known to the committee at this time is McMaster Carr, 200 New Canton Way, Robbinsville, NJ 08691-2343, www.mcmaster.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] The sole source of supply of the apparatus (8-Path Wet Film Applicator) known to the committee at this time is Paul N. Gardner Company, Inc., 316 N.E. First Street, Pompano Beach, FL 33060, www.gardco.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.



(a) (b)

FIG. 1 Example of Thin Film Applicator (a) and Emulsified Asphalt Film Produced After Draw Down (b)

5.4 Vacuum oven or any suitable heated pressure reduction vessel, capable of maintaining a temperature of 60 ± 5°C (140 ± 10°F) and holding a vacuum of 1300 ± 500 Pa (0.4 ± 0.2 inHg) absolute pressure for the specified test time.

5.5 Tool capable of removing the emulsified asphalt residue from the silicone rubber mat after recovery without the use of heat or chemical reagents. Recommended tools include a metal spatula to scrape off the material or rolling by a glass rod. Use removal tools with dimensions suitable for the area of the drawn down emulsified asphalt film.

6. Reagents and Materials

6.1 Cleaning agent, capable of removing residual asphalt and other oils from the silicone rubber mat. Halogenated solvents should not be used on the silicone rubber mat material.

6.2 Remove any remaining oils from the silicone rubber mat. Do not use a soap-water solution.

NOTE 2—VM&P naptha, heptane, or other mineral spirits are suggested to remove the residual asphalt from the silicone rubber mat. Following the use of these materials wipe the silicone rubber mat with acetone or warm at 60 ± 5°C (140 ± 10°F) for at least 15 min. Alternative methods can be used provided they are effective at removing residual asphalt and traces of solvents or mineral spirits from the silicone rubber mat.

7. Preparation of Apparatus

7.1 Pre-heat both the support material used during emulsified asphalt recovery and the vacuum oven or other suitable pressure reduction vessel to 60 ± 5°C (140 ± 10°F) for a minimum of 30 min.

NOTE 3—Preheating the vessel 10 to 15°C above the 60°C recovery temperature can be used to reduce the drop in chamber temperature during loading and to minimize the time required to stabilize the system after loading to attain the required temperature range. The specific pre-heat temperature offset used is equipment specific so select the pre-heat temperature based on the value that minimizes the time delay between sample loading and the system returning to the operating range provided in 7.1.

7.2 Wipe clean the material that will support the silicone rubber mat during draw down of the emulsified asphalt film and inspect to ensure the surface is absent any particles or other irregularities. It is not necessary to preheat the support material used for draw down of the emulsified asphalt film.

8. Calibration and Standardization

8.1 *Vacuum Oven:*

8.1.1 *Vacuum Oven Thermometric Device*—Verify the thermometric device used in the vacuum oven to within 60 ± 5°C (140 ± 10°F) at least every twelve months using a calibrated thermometric device traceable to a national standard. Verification shall be performed at a temperature that is within 5°C of the use temperature.

8.1.2 *Absolute Pressure Gauge*—Verify the absolute pressure gauge to equate to a reading within 1300 ± 500 Pa (0.4 ± 0.2 inHg) pressure at least every twelve months using a calibrated vacuum or pressure indicator traceable to a national standard.

NOTE 4—If a vacuum pressure gauge is used, the gauge reading represents the difference between atmospheric pressure and the pressure in the vessel. This value must be converted to absolute pressure by adding the atmospheric pressure to the gauge pressure. Standard atmospheric pressure at sea level is 101.3 kPa (29.92 inHg), correction for higher altitudes is required. A procedure for correcting gauge readings based on altitude is provided in Table 1 of AASHTO R28.

9. Conditioning

9.1 Handle emulsions in accordance with Test Methods D244 subsection 3.1 with the following modifications: Increase the sample heating temperature to 55 ± 5°C (131 ± 10°F) for all emulsions regardless of emulsion viscosity testing requirements.

NOTE 5—The conditioning temperature specified in 9.1 applies to all emulsified asphalt grades, regardless of temperature requirements for other test methods.

10. Procedure

10.1 Place the silicone rubbermat on the support material. Both should be at ambient temperature.

10.2 Place draw down applicator with correct thickness dimension on the silicone rubber mat. Pour sufficient emulsified asphalt inside the draw down applicator to coat the desired length of the silicone rubber mat at the width of the draw down applicator.

NOTE 6—Required amount is approximately 10 to 15 g in the example shown in Fig. 2. Specific quantities will vary depending on the dimensions of the applicator and length of draw down. The location of the emulsified asphalt relative to the applicator (that is, in front of leading edge, inside, etc.) could vary by thin-film applicator geometry.

10.3 Use a wet film applicator capable of generating a film of 0.38 mm (15 mils) nominal film thickness to draw down the emulsified asphalt into a thin film on the silicone rubber mat.

NOTE 7—It has been observed that de-wetting, pooling, or beading occurs for some emulsified asphalts, particularly low viscosity emulsified asphalts, after drawdown. Examples are shown in Fig. 3. In this instance do not proceed with the recovery as the film thickness of the emulsified asphalt is not consistent. Wipe the emulsified asphalt from the silicone rubber mat and re-apply immediately (example provided in Fig. 4). If it is expected that the emulsified asphalt will pool prior to recovery, wiping and re-application of the emulsified asphalt for draw down can be performed immediately without inspecting the results of the initial draw down. If wiping and re-application does not result in a suitable film, discard the silicone rubber mat and replace with mat of different durometer rating or from different supplier, or both.

10.4 Transfer the silicone rubber mat and emulsified asphalt to the support located in the preheated vacuum oven or other suitable device according to 7.1. Limit the time between thin film draw down and loading into the vacuum oven to a maximum of 10 min.

10.5 If the vacuum oven or pressure reduction vessel was pre-heated to other than the required recovery temperature, reset the temperature control of the device to 60 ± 5°C (140 ± 10°F) and adjust the pressure to 1300 ± 500 Pa (0.4 ± 0.2 inHg). If the required temperature or pressure range, or both, is not achieved within 40 min the test is considered invalid and new material will need to be recovered to obtain an acceptable emulsified asphalt recovery. Consistently failing these requirements indicates that the vacuum oven or pressure reducing vessel used for the test is inadequate or in need of repair.

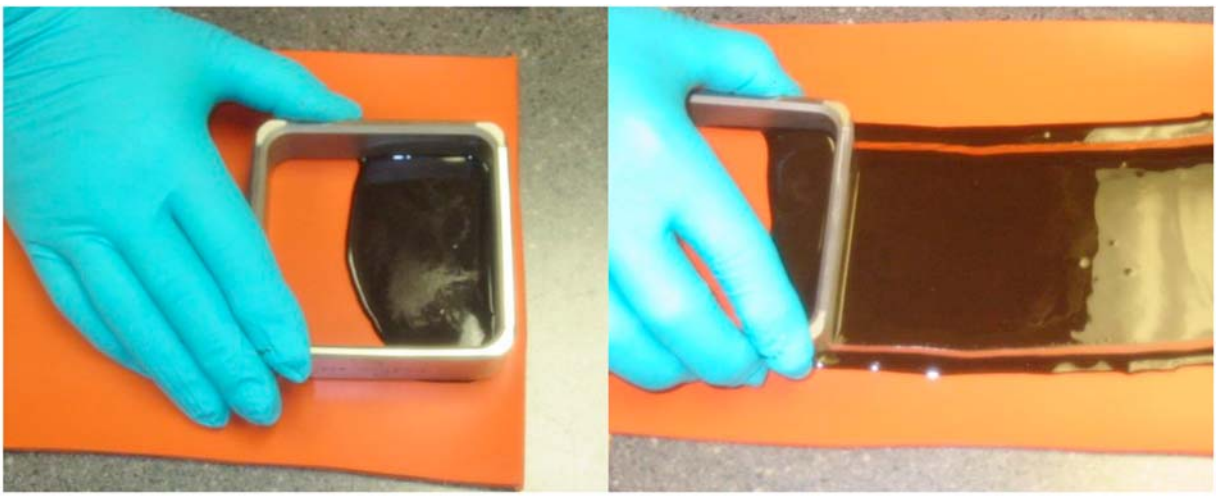
10.6 Maintain sample in the vacuum oven or other conditioning device at 60 ± 5°C (140 ± 10°F) and a pressure of 1300 ± 500 Pa (0.4 ± 0.2 inHg) for 3 h ± 5 min. The 3 h conditioning time required for recovery does not begin until the required vacuum is achieved.

10.7 During recovery as moisture is drawn out of the emulsified asphalt the pressure will increase, necessitating re-application of the vacuum to maintain the pressure within the 1300 ± 500 Pa (0.4 ± 0.2 inHg) range.

NOTE 8—The majority of moisture loss occurs within the first 30 min, during this timeframe it is recommended that the vacuum pressure be monitored in 10 min intervals and adjusted as necessary. After the 30 min time period has elapsed regularly monitor the level of vacuum to verify that the vacuum oven or other suitable vessel is operating within the required pressure range throughout the recovery.

10.8 At the end of the emulsified asphalt residue recovery time period, release the vacuum and remove the silicone rubber mat from the oven. Removal of the emulsified asphalt residue from the silicone rubber mat shall be completed within 1 h of removing the sample from the vacuum oven. Place silicone rubber mat on a laboratory bench or other flat surface, scrape emulsified asphalt residue off, deposit it into a clean container and cover. An example of the emulsified asphalt residue after recovery and depositing the material into a container is provided in Fig. 5. Approximately 5 g of emulsified asphalt residue will be obtained from a drawdown dimension of 9 by 28 cm. Do not collect the material that tracked outside of the wet film applicator during draw down. This material can be discarded.

NOTE 9—Store the recovered emulsified asphalt residue in a covered container at ambient laboratory temperatures to prevent further aging until it is tested. It is recommended that the material be tested within 24 h of recovery. Reheat sample using an oven set to a maximum temperature of 90°C for no more than 5 min. The objective of the reheating process is to



(a) Sample Loading (b) Draw Down in Progress

FIG. 2 Example of Pouring Emulsified Asphalt Into Thin Film Applicator (a), and Drawing Down of Thin Film (b)

NOTE 1—Ideal shape is a rectangle the width of the applicator. Signs of pooling/beading include migration of the emulsified asphalt towards the center of the mat (a), or presence of voids and irregularities on surface of film (b).

FIG. 3 Examples of Pooling After Thin Film Draw Down

FIG. 4 Example of Silicone Rubber Mat Surface After Wiping Clean, Prior to Re-application of Emulsified Asphalt

get the material to a semi-solid state to facilitate molding and manipulation of the sample. The sample should not be heated such that the emulsified asphalt residue is in liquid form. Reheating the sample at conditions more severe than those suggested above will cause changes to the rheological properties of the material and may result in emulsified asphalt residue that is not representative of the material placed in the field.

NOTE 10—After the vacuum conditioning described in this procedure the emulsified asphalt residue will generally contain a small amount of residual moisture. This procedure produces emulsified asphalt residues with moisture contents similar to emulsified asphalt residues recovered using the low temperature evaporative technique described in AASHTO PP 72.

10.9 Clean silicone rubber mat to remove the remaining emulsified asphalt residue followed by wiping. After cleaning dry the silicone rubber mat for approximately 15 min at 60°C.

NOTE 11—Other cleaning procedures may be adopted such that the silicone rubber mat is free of residual oils and a uniform, non-beading film of emulsified asphalt can be applied to the silicone rubber mat.

10.10 Clean draw down applicator and allow to dry.

11. Keywords

11.1 emulsified asphalt; emulsified asphalt recovery; emulsified asphalt residue

(a) Scraping of emulsified asphalt residue from silicone rubber mat

(b) Depositing emulsified asphalt residue in sample tin

NOTE 1—a) Removing from Silicone Rubber Mat, b) Depositing into Sample Container

FIG. 5 Handling Emulsified Asphalt Residue After Recovery