Designation: D7403 – 09

Standard Test Method for Determination of Residue of Emulsified Asphalt by Low Temperature Vacuum Distillation[1]

This standard is issued under the fixed designation D7403; 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 method covers the quantitative determination of residue in emulsified asphalts composed principally of a semisolid or liquid asphaltic base, water, and an emulsifying agent. The emulsified asphalts will generally contain polymeric materials. It is especially suitable for emulsified asphalt residue properties that may be altered at the high temperature 260°C (500°F) distillation. Since there is currently not a precision statement for this procedure, it is recommended to the user that this procedure not be used for buy/sell purposes at the present time.

1.2 The values stated in SI units are to be regarded as the standard. Values in parentheses in inch-pound units are provided for informational purposes 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:*[2]

D6997 Test Method for Distillation of Emulsified Asphalt

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Significance and Use

3.1 This test method can be used for quantitative determination of residue in emulsified asphalts at a temperature of 135°C (275°F) with a 60 minute distillation test using current distillation apparatus. This method is suitable to obtain residues for service evaluation, quality control, and research. This distillation method is not intended to produce residues equivalent to the Test Method D6997 260°C (500°F) distillation procedure.

4. Apparatus

4.1 *Aluminum Alloy Still,* conforming to Test Method D6997 except that a 13 mm (½ in.) diameter hole is drilled between the two existing 13 mm (½ in.) thermometer holes for the connection of a vacuum gauge (see Fig. 1). The joint between the still and lid shall be air tight with the gasket in place. Other heating devices may be used provided they employ the intended control of temperature during the distillation procedure.

4.2 *Connection Apparatus,* Test Method D6997, with modifications as shown in Fig. 2. Connection tubing may be of suitable materials provided the intended vacuum, temperature, and method is maintained. Other forms of condensers may be used provided they have an equivalent or greater interior surface area for condensation of distillate.

NOTE 1—Silicone rubber stoppers are recommended.

4.3 *Thermometric Device,* ASTM 7C (7F) thermometers, or thermocouple devices calibrated at 135°C (275°F). The thermocouple probe, Type T with exposed junction, should be of sufficient length (approximately 300 mm (12 in.)) to be positioned approximately 6 mm (0.25 in.) off the bottom of the assembled still. Use of equivalent thermometric devices is permitted.

4.4 *Balance,* capable of weighing 3500 grams to ± 0.1 gram.

4.5 *Vacuum Pump,* rotary vane type, capable of maintaining a vacuum of 88 kPa below gauge pressure or greater.

NOTE 2—88 kPa below gauge pressure is equal to 26 in. Hg (660 mm) below atmospheric pressure.

4.6 *Gasket,* of silicone rubber, 3 mm (⅛ in.) thick, cut to fit flanged opening on still. Other gasket materials may be used provided they withstand the maximum temperature reached during distillation and are able to maintain the specified vacuum within the still.

4.7 *Vacuum Gauges,* dial type or other suitable type connected to vacuum tubing and/or fittings to allow attachment to both vacuum pump and to still apparatus and capable of

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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.

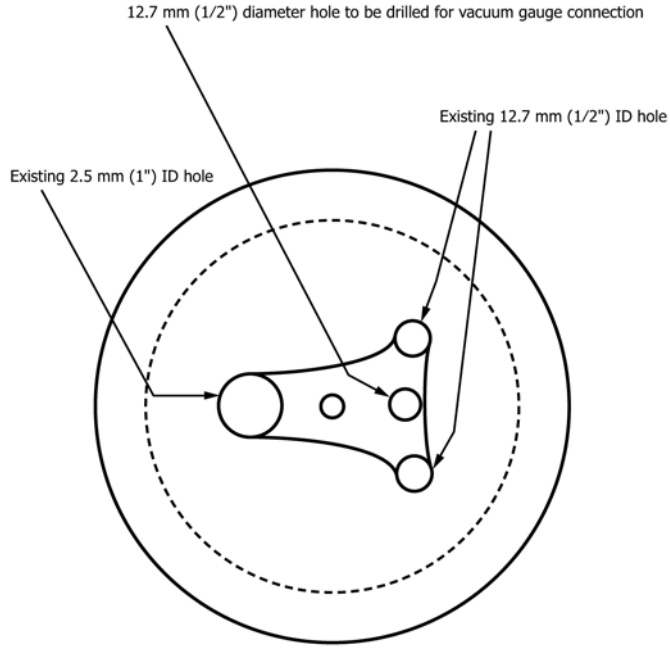

FIG. 1 Aluminum Alloy Still Lid Showing Location of the Hole for Vacuum Gauge Connection

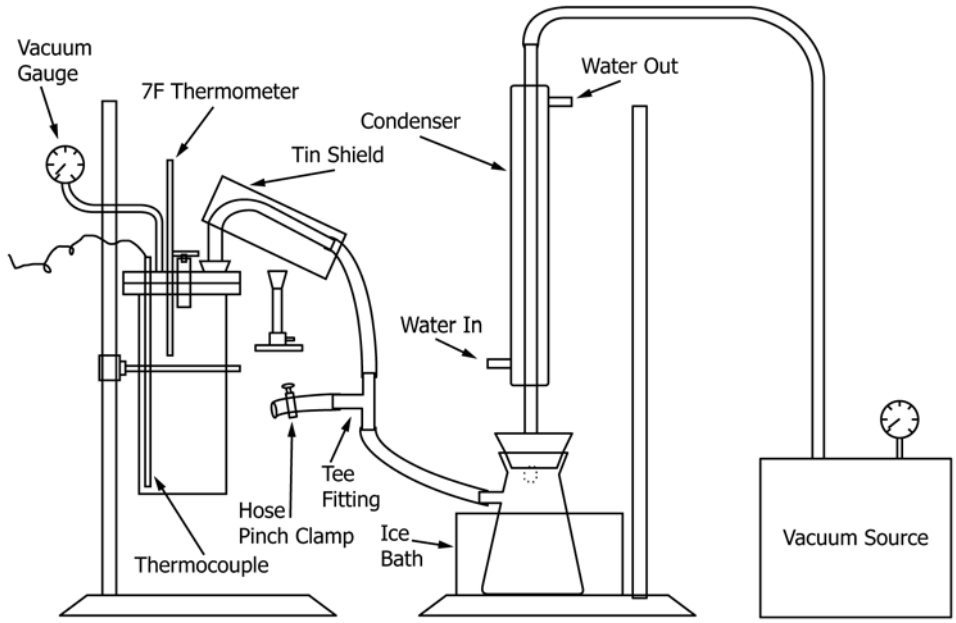

FIG. 2 Connection Apparatus Schematic

reading a minimum vacuum of 88 kPa below gauge pressure (660 mm or 26 in. Hg below atmospheric pressure).

4.8 *Freezer,* maintained at approximately –10°C.

4.9 *Disposable Plastic Drink Cups,* of convenient size and make to serve as a container during freezing of emulsified asphalt.

5. Hazards

5.1 **Warning—**Mercury has been designated by the United States Environmental Protection Agency (EPA) and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should

be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website (www.epa.gov/mercury/faq.htm) for additional information. Users should be aware that selling mercury and/or mercury containing products into your state may be prohibited by state law.

6. Procedure

6.1 Weigh 200 to 201 grams of a representative sample of the emulsified asphalt into the disposable plastic drink cup. Place cup and sample in freezer until thoroughly frozen.

NOTE 3—Typically 0.5 grams of frozen emulsion is lost in handling the frozen emulsified asphalt. If the desired amount of frozen emulsified asphalt to be tested is 200.0 grams then weigh into the drink cup 200.5 grams.

6.2 Prepare still by inserting thermometer and thermocouple probe through size #00 rubber stoppers, and then into the 13 mm (½ in.) holes provided for in the still cover. Adjust the thermometric device so the end is 6 mm (0.25 in.) from the bottom of the still. The bulb of the second thermometric device should be adjusted to 165 mm (6.5 in.) from the bottom of the still.

6.3 Prepare the frozen emulsified asphalt sample by splitting edge of cup and partially peeling sides away from sample. Place frozen sample in previously weighed still apparatus (including lid, clamp, thermometric devices, stoppers, and gasket) and weigh to 0.1 gram.

6.4 Assemble apparatus and insert vacuum gauge with tubing and stopper attachment into the 13 mm (½ in.) hole located between the thermometer holes. Immediately apply a minimum vacuum of 88 kPa below gauge (atmospheric) to the still and contents. Maintain this vacuum throughout the distillation. The elapsed time from removing the sample from the freezer to applying vacuum should not exceed two minutes.

6.5 After 10 minutes apply heat from a ring burner or other suitable heating device. The ring burner at the beginning of the heating period shall be positioned around the still approximately 200 mm (8 in.) from the bottom of the still. Also apply a small amount of heat to the connecting tube from a Bunsen burner or other suitable heating device. Other heating devices may require a different procedure to apply heat in the intended manner.

NOTE 4—The location of the ring burner or heating device at the start of the test is flexible.

6.6 As the temperature rises to approximately 149°C (300°F) on the upper thermometric device, lower the burner and continue heating until the lower thermocouple reads 135 ± 5°C (275 ± 10°F). This temperature should be achieved within 45 minutes of initially applying the heat.

6.7 Maintain the temperature of the still contents at 135 ± 5°C (275 ± 10°F) for 15 minutes.

NOTE 5—Cooling of the outside of the still may be done to prevent temperature overshoot. This can be performed by application of water soaked toweling momentarily applied to base of the still with due precaution.

6.8 At the end of the heating period immediately reweigh the still apparatus and residue. The aluminum alloy still at room temperature weighs 0.8 grams more than at 135°C (275°F). Correct for this error by adding 0.8 grams to the gross weight before calculating the percentage of residue by distillation. Record results.

6.9 Remove the lid from the still and if the residue is sufficiently fluid, stir, and transfer suitable portions of the residue into molds or containers for further testing. If the residue is not sufficiently fluid for pouring without the incorporation of air, quickly heat the sample to the lowest temperature at which this can be achieved. Record the pouring temperature.

7. Calculation

7.1 Calculate the residue content as follows:

$$\text{Emulsified asphalt residue, } \% = \left(\frac{A}{B}\right) \times 100 \quad (1)$$

where:

A = weight of residue, grams, and
B = weight of emulsified asphalt, grams.

8. Report

8.1 Report the following information:
8.1.1 Sample identification,
8.1.2 Date of test,
8.1.3 Pouring temperature,
8.1.4 Emulsified asphalt residue weight percent, and
8.1.5 Distillation test method.

9. Precision and Bias

9.1 *Precision*—The research necessary to report precision has not been conducted.

9.2 *Bias*—The research necessary to report bias has not been conducted.

10. Keywords

10.1 asphalt; distillation; emulsified asphalt; emulsion; low-temperature; vacuum