



Designation: D7403 – 19

Standard Test Method for Determination of Residue of Emulsified Asphalt by Low Temperature Vacuum Distillation¹

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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 The text of this standard reference 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.*

2. Referenced Documents

2.1 ASTM Standards:²

¹ This test method 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.

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials
D6997 Test Method for Distillation of Emulsified Asphalt

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-min 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.

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.

4. Apparatus

PROCEDURE A

4.1 *Aluminum Alloy Still*, conforming to Test Method D6997 except that a 13-mm (1/2-in.) diameter hole is drilled between the two existing 13-mm (1/2-in.) thermometer holes for the connection of a vacuum gauge (see Fig. 1). The joint between the still and lid shall be airtight 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 are maintained. Other forms of condensers may be used, provided they have an equivalent or greater interior surface area for condensation of distillate.

NOTE 2—Silicone rubber stoppers are recommended.

4.3 *Thermometer*—One of the following: (1) ASTM 7C (7F) thermometer, or (2) thermocouple(s) calibrated at 135 °C

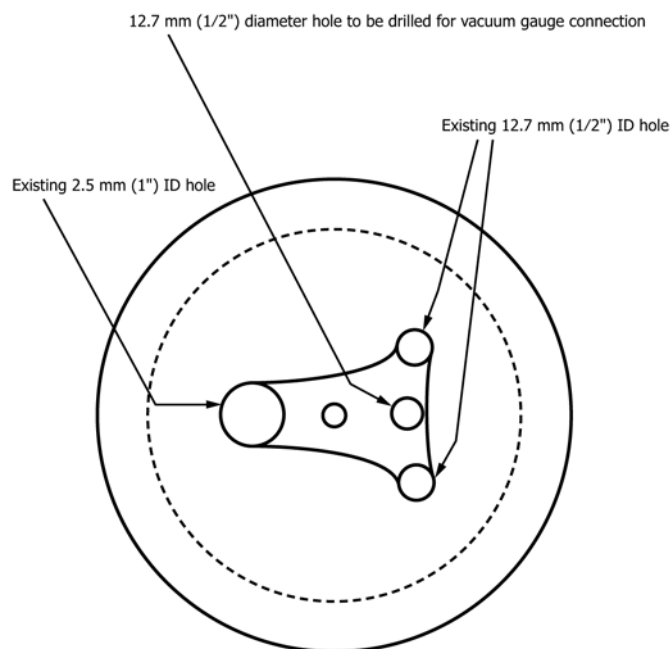


FIG. 1 Aluminum Alloy Still Lid Showing Location of the Hole for Vacuum Gauge Connection

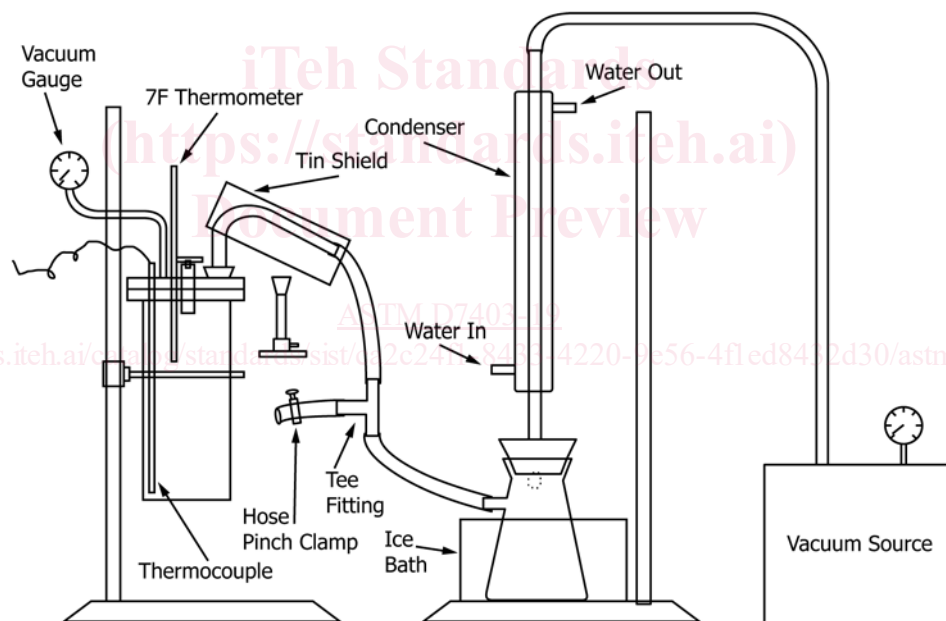


FIG. 2 Connection Apparatus Schematic

(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.

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

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

NOTE 3—88 kPa below gauge pressure is equal to 26 in. Hg (660 mm) below atmospheric pressure.

4.6 *Gasket*, of silicone rubber, 3 mm ($1/8$ 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, fittings, or both, to allow attachment to both vacuum pump and to still apparatus and capable of

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 $-10\text{ }^{\circ}\text{C}$ ($14\text{ }^{\circ}\text{F}$).

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

4.10 *Oven*, maintained at $150 \pm 5\text{ }^{\circ}\text{C}$ ($302 \pm 9\text{ }^{\circ}\text{F}$).

PROCEDURE B

4.11 *Rotary Evaporator*, with an oil bath capable of maintaining a temperature of at least $165\text{ }^{\circ}\text{C}$ ($329\text{ }^{\circ}\text{F}$), maintaining a vacuum as stipulated in 4.5 and being able to rotate the distillation flask at $25 \pm 5\text{ rpm}$.

NOTE 4—The recommended flask for this test is a 2-L Morton flask, also referred to as a modified German rolling flask; see Fig. 3. The 2-L Morton flask is recommended because there is a fair amount of foaming when the emulsified asphalt is above $100\text{ }^{\circ}\text{C}$ and under vacuum. As can be seen in Fig. 3, the Morton flask has indentations arranged at 90° intervals around the flask perimeter. These indentations facilitate agitation of the emulsified asphalt during the vacuum distillation procedure. A standard pear-shaped rotary evaporator flask would not provide the same level of agitation and is therefore not recommended. The bath must be of sufficient volume and depth such that the Morton flask can be submerged in the bath liquid so that the full volume of emulsified asphalt when fluid is below the surface of the bath oil when the flask is not being rotated.³ A food-grade mineral oil with a nominal viscosity of 350 cs at $60\text{ }^{\circ}\text{C}$ ($140\text{ }^{\circ}\text{F}$) has been found acceptable as a bath fluid. The rotary evaporator bath manufacturer for the specific conditions of this test may recommend other bath fluids. The rotary evaporator condenser can be cooled by tap water or by using a closed-system circulating chiller to ensure condensation of water vapor from the vacuum distillation procedure.

³ It is known that the OA-SYS bath available from Organomation Associates, Inc. (Fig. 5) will accommodate the 2-L Morton flask, although any bath capable of submerging the flask as described and maintaining the temperature as required is acceptable.

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

6. Procedure

PROCEDURE A

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

NOTE 5—Typically, 0.5 g 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 g, then weigh into the drink cup 200.5 g.

6.2 Prepare still by inserting thermometer and thermocouple probe through size #00 rubber stoppers, and then into the 13-mm ($\frac{1}{2}$ -in.) holes provided 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



FIG. 3 Two-Liter Morton Flask on Cork Ring