Designation: ~~D8188 – 18~~ D8188 – 23

Standard Test Method for Determination of Density and Relative Density of Asphalt, Semi-Solid Bituminous Materials, and Soft-Tar Pitch by Use of a Digital Density Meter (U-Tube)[1]

This standard is issued under the fixed designation D8188; 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 test method covers the determination of the relative density and density of semi-solid asphalt materials, asphalt binders, asphalt cements, and soft-tar pitches by use of a digital density meter (U-tube).

NOTE 1—Alternate methods for determining the density of semi-solid and solid asphalt materials and asphalt binders include Test Methods D3289, D3142/D3142M, and ~~D70~~D70/D70M.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only. No other units of measurement are included in this standard, with the exception of temperature measurements. Units provided in degrees Fahrenheit are for reference purposes only. Temperatures given in degrees Celsius are to be considered standard.

~~1.3 The between-laboratory reproducibility of this test method is being determined and will be available on or before June 2021. Therefore, this standard should not be used for acceptance or rejection of a material for purchasing purposes.~~

1.3 This test method should not be applied to petroleum distillates other than asphalt and asphalt binders. For the determination of density of all other petroleum distillates and viscous oils, use Test Method D4052 or D5002, or ISO 12185.

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

1.5 *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.*

1.6 *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.*

[1] This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.47 on Miscellaneous Asphalt Tests.

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

D8 Terminology Relating to Materials for Roads and Pavements
~~D70~~D70/D70M Test Method for Specific Gravity and Density of Semi-Solid Asphalt Binder (Pycnometer Method)
D140/D140M Practice for Sampling Asphalt Materials
D287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer/Method)
D1250 Guide for the Use of the Joint API and ASTM Adjunct for Temperature and Pressure Volume Correction Factors for Generalized Crude Oils, Refined Products, and Lubricating Oils: API MPMS Chapter 11.1
D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D3142/D3142M Test Method for Specific Gravity, API Gravity, or Density of Cutback Asphalts by Hydrometer Method
D3289 Test Method for Density of Semi-Solid and Solid Asphalt Materials (Nickel Crucible Method)
D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials
D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
D4311/D4311M Practice for Determining Asphalt Volume Correction to a Base Temperature
D5002 Test Method for Density, Relative Density, and API Gravity of Crude Oils by Digital Density Analyzer
D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories
D7578 Guide for Calibration Requirements for Elemental Analysis of Petroleum Products and Lubricants
D7961 Practice for Calibrating U-tube Density Cells over Large Ranges of Temperature and Pressure
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standard:*[3]

ISO 12185 Crude Petroleum and Petroleum Products—Determination of Density—Oscillating U-Tube Method

3. Terminology

3.1 *Definitions:*

3.1.1 *certified reference material (CRM), n*—a reference material whose one or more property values are certified by a technically valid procedure, accompanied by a traceable certificate or other documentation which is issued by a certifying body, as defined in Practice D6792 and Guide D7578.

3.1.2 *density, n*—the mass per unit volume of a material at a specified temperature.

3.1.3 *relative density, n*—the ratio of the density of a material at a stated temperature to the density of water at a stated temperature.

3.1.3.1 *Discussion*—

Relative density is also known as "specific gravity." Common stated temperatures are 20 °C/20 °C, 15 °C/15 °C, 20 °C/4 °C, and 60 °F/60 °F.

3.1.4 For definitions of general terms used in this standard, refer to Terminology D8.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *adjustment, v*—the operation of bringing the instrument to a state of performance suitable for its use, by setting or adjusting the density meter constants.

3.2.1.1 *Discussion*—

On some digital density meters, an "adjustment" procedure may be performed rather than "calibration." The adjustment procedure uses air and oil-based certified reference material as standards to establish the linearity of measurements over a range of operating temperatures.

3.2.2 *test specimen, n*—the volume of sample aliquot residing in the U-tube during the measurement cycle.

3.2.2.1 *Discussion*—

Sample material residing in filling nozzles, tubing, and valve manifolds is not considered a "test specimen." A test specimen can be measured only once.

[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 International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

4. Summary of Test Method

4.1 A small volume, approximately 3 to 4 mL, of liquid hot sample is introduced into an oscillating U-shaped tube. Introduction of samples into the U-tube changes the oscillating frequency. The change in the frequency is used to determine the density, relative density, or API gravity of the sample.

5. Significance and Use

5.1 Values of density are used for converting volumes to units of mass and for correcting measured volumes at the measured temperature to a standard temperature using Practice D4311/D4311M.

NOTE 2—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.

6. Apparatus

6.1 *Digital Density Meter*—A digital density meter consists of a U-shaped oscillating sample tube, temperature control unit, and a system for electronic excitation, frequency counting, and display. The analyzer shall accommodate the accurate measurement of the sample temperature during measurement. The instrument shall be capable of meeting the precision requirements described in this test method.

6.2 *Syringes,* made of glass or PFA (perfluoroalkoxy alkane). For use primarily in manual injections, which withstand elevated temperatures, at least 10 mL in volume, with a tip or an adapter tip that will fit the opening of the U-tube.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] 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.

7.2 *Cleaning Solvent,* such as petroleum naphtha, or other materials (for example, toluene or mesitylene) (**Warning—**Petroleum naphtha, toluene, and mesitylene are extremely flammable) that are capable of flushing and removing sample residue entirely from the U-tube and have a boiling point higher than the measuring temperature.

8. Sampling, Test Specimens, and Test Units

8.1 Take samples in accordance with Practice D140/D140M. The sample shall be free of foreign substances.

8.2 Thoroughly mix and homogenize the sample before removing a representative portion for testing. Ensure mixing does not introduce air bubbles into the sample.

8.3 *Test Specimen*—A portion or volume of homogeneous sample aliquot delivered to the density analyzer U-tube.

9. Preparation of Apparatus

9.1 Set up the oscillating U-tube density meter, following the manufacturer's instructions. Adjust the instrument at the same temperature at which the density or relative density of the sample is to be measured.

[4] 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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

10. Adjustment of Apparatus

10.1 The adjustment routine for digital density meters involves using a minimum of two reference media. Air and oil-based certified reference materials (CRMs) with density values at relevant temperatures are usually used for adjustment under atmospheric conditions. Other materials such as decane, dichlorotoluene, or toluene can also be used as appropriate adjustment materials, provided that the reference materials have density values that are certified and traceable to national standards (compare to Practice D7961).

10.2 Follow the manufacturer's instructions for the proper adjustment of the apparatus. The density of air varies with ambient pressure and relative humidity. The relative humidity of air above 100 °C is 0, therefore, the formula is simplified. The air density can be calculated using this formula by F. Spieweck and H. Bettin:[5]

$$\rho = \frac{0.34844 * p}{273.15 + T} \quad (1)$$

where:

ρ = density (kg/m^3),
p = pressure (mbar), and
T = temperature (°C).

11. Procedure

11.1 *Preparation of Sample*—Heat the sample with care, stirring to prevent local overheating, until the sample has become sufficiently fluid to pour. In no case should the temperature be raised to more than 55 °C (131 °F) above the expected softening point for soft-tar pitch, or to more than 110 °C (230 °F) above the expected softening point for asphalt. Do not heat for more than 60 min, and avoid incorporating air bubbles into the sample.

11.2 Draw an aliquot of sample slowly into a clean and dry syringe. It is recommended to pre-heat glass and PFA syringes to the same temperature as the test specimen; however, caution should be exercised in heating medical plastic syringes. Take precautions to keep the tip of the syringe clean and prevent inclusion of air bubbles. Fill the sample slowly into the U-tube density analyzer and start the measurement. Ensure sample does not experience significant temperature drop during injection, as the injection port may get clogged or sample might lose its homogeneity.

11.2.1 After filling the syringe with the test specimen, it can be placed back in the oven for 5 min in a vertical position with the piston bottom facing down. This ensures that any bubbles present can collect at the tip of the syringe and be discarded before the sample is injected into the density analyzer.

11.3 Ensure that the U-tube sample tube is properly filled and that no gas bubbles are present. The sample must be free of even the smallest gas bubbles. Check the integrity of the filled sample by measuring a duplicate test specimen.

NOTE 3—The presence of bubbles may result in the instrument failing to stabilize, which is necessary to provide a density reading.

11.4 Start the measurement according to the manufacturer's instructions.

11.5 Allow the sample to equilibrate to the test temperature before recording the density result or relative density. For instruments that can print out results from the display, the printout can be used to meet the recording requirements.

11.6 Flush the U-tube with a proper cleaning solvent (see 7.2) and dry with dry air. Allow the dry air in the U-tube to come to thermal equilibrium with the test temperature and compare the density value to the density of air using Eq 1.

NOTE 4—Some solvents may give vapors at higher temperatures, which might require a fume hood. Take precautions when flushing the U-tube with cleaning solvent. The boiling point of the used cleaning solvent shall be at least 10 °C above the measuring temperature. Handling solvents at boiling or near-boiling temperature can present a safety hazard. Wear appropriate personal protective equipment.

[5] Spieweck, F. and Bettin, H., "Review: Solid and Liquid Density Determination," *tm – Technisches Messen*, Vol 59, No. 7, 1992, pp. 285–292.