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Standard Test Method for Rubber Chemicals—Density[1]

This standard is issued under the fixed designation D1817; 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 density of solid chemicals used as rubber additives during processing and manufacture. It is intended for determining the density of the rubber chemical itself and not for the determination of the effective density of the chemical in a rubber.

1.2 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E1 Specification for ASTM Liquid-in-Glass Thermometers

E300 Practice for Sampling Industrial Chemicals

3. Summary of Test Method

3.1 The density of the test specimen is obtained via wet pycnometry.

4. Significance and Use

4.1 The density of a rubber chemical is used for calculating the rubber compound volume, which is used to determine the cost of a rubber product. The density may also be used as a raw material control tool.

5. Apparatus

5.1 *Pycnometer,* 50-cm^3 capacity.

NOTE 1—The weld type with the cap seal on the outside of the neck of the bottle is preferred because there is less danger of trapping air just under the capillary tube than with types having the ground seal on the inside of the neck. The stopper may contain a thermometer. However, the control of the temperature is through the adjustment of the water bath rather than by this thermometer.

5.2 *Water Bath,* maintained at 23 ± 0.5°C and equipped with a stirring device.

5.3 *Pressure Meter*—A gage or manometer that is accurate to 0.2 kPa, is required to measure the differential pressure between the outside and inside of the system.

5.3.1 Alternatively, a device for measuring the absolute pressure inside the system can be used. The range of the measuring device should be at least 100 kPa for differential pressure meters and at least 10 kPa for absolute pressure meters.

5.4 *Desiccator*—A glass desiccator constructed with heavy walls to withstand a differential pressure of 100 kPa and with an opening at the side or top. The desiccator should be enclosed in a sturdy box or shield to prevent possible injury to the operator.

5.5 *Vacuum Pump*—An oil-filled, motor-driven pump, capable of reducing the absolute pressure of the system to 2 kPa.

5.6 *Thermometer,* having a minimum range from 10 to 30°C and graduated in 0.1°C divisions. ASTM Solidification Point Thermometer having a range from 0 to 30°C and conforming to the requirements for Thermometer 90C as prescribed in Specification E1, may be used.

5.7 *Weighing Bottle*—A wide-mouth, cylindrical, glass weighing bottle (about 30 mm in height and 70 mm in diameter) provided with a ground-glass stopper.

6. Sampling

6.1 Select a representative sample of the chemical to be tested in accordance with the appropriate section of Practice E300.

6.2 *Mesh Size*—Rubber chemicals are generally in the form of powders that require no further treatment. Grind any lumps, pellets, and so forth, to pass a 149-µm sieve prior to the determination.

6.3 *Drying*—It is not necessary to dry rubber chemicals, unless it is known that they contain sufficient water to interfere with an accurate density measurement. If necessary, dry to constant mass at least 10°C below the melting pointing but not above 110°C.

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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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.

7. Immersion Liquid

7.1 An immersion liquid should be chosen in which no portion of the rubber chemical (major component, impurity, or added component) is soluble and which will not react with any portion of the rubber chemical. Refined white kerosene of narrow boiling range and low evaporation rate may sometimes be used. Other immersion liquids such as ethylene glycol, tetrahydronaphthalene, and so forth, may be suitable. If an immersion liquid cannot be found that meets these solubility requirements, an immersion liquid saturated with the soluble components of the sample shall be used.

8. Standardization of Pycnometer

8.1 Fill the pycnometer with freshly boiled distilled water at 20 to 22°C; gradually bring to 23 ± 0.5°C in the water bath, and then remove, dry, and weigh it as described in 9.1. Empty the pycnometer, then clean, dry, and reweigh it. Next, fill the pycnometer with the immersion liquid at 22°C or less, and bring to 23 ± 0.5°C in the water bath. Remove from the bath, dry, and weigh as before. Calculate the density of the immersion liquid as follows:

$$D\text{ensity at } 23°C,\ M\text{g/m}^3 = (A/B) \times 0.997 \quad (1)$$

where:

A = mass of immersion liquid, and
B = mass of water,
0.9976 = density of water at 23°C, Mg/m^3.

9. Procedure

9.1 *Weighing*—Transfer a sufficient amount of the test specimen to a clean, dry, weighed pycnometer to form a layer approximately 19 mm (¾ in.) deep and reweigh. Weigh rubber chemicals of a hygroscopic nature from a weighing bottle.

9.2 *Addition of Immersion Liquid*—Add sufficient immersion liquid to the pycnometer to form a thin layer above the rubber chemical. When necessary, swirl the contents of the pycnometer by hand, to wet the sample.

9.3 *Removal of Occluded Air*—Place the pycnometer in the desiccator, close, and attach to the pump for the removal of air. Take care not to decrease the pressure too quickly, otherwise some of the sample may be lost due to the sudden removal of the entrapped air. A pinch-cock may be used to control the rate of evacuation. The pressure meter is used to indicate whether the oil pump is giving the proper vacuum, which is an absolute pressure of 0.2 kPa. Bubbles of air rise from the sample rapidly at first, then decrease and finally stop. The time required for complete removal of air may vary from 30 min to 24 h. When no more bubbles can be seen, it may be assumed that occluded air has been removed and the rubber chemical is thoroughly wet with immersion liquid. Slowly readmit air to the desiccator.

NOTE 2—If an immersion liquid is used whose vapor pressure is greater than 0.2 kPa at room temperature, the liquid will evaporate, often with "bumping." Therefore it is necessary to adjust the vacuum to a higher pressure with some loss in effectiveness of air removal. The use of such immersion liquids should be limited to those cases in which no low vapor pressure liquid can be used. In no case should a mixed liquid be used in which any component has an appreciable vapor pressure.

9.4 *Final Adjustment*—Remove the pycnometer from the desiccator, fill with immersion liquid at 22°C or less, taking care to add a sufficient quantity to prevent air bubbles remaining in the pycnometer when closed. Insert the stopper, being careful not to trap any air bubbles. Place the pycnometer in the water bath and permit it to come to constant temperature at 23 ± 0.5°C. Remove from the water bath, wipe the end of the capillary with lint-free toweling or lens paper, making sure not to suck any liquid from the capillary. Cap the capillary. Dip the pycnometer (up to the side arm) in a beaker of alcohol to remove any residual immersion liquid. Thoroughly dry the outside of the pycnometer and weigh.

9.5 *Number of Specimens*—Make duplicate tests on all specimens.

10. Calculation

10.1 Calculate the density of the rubber chemical as follows:

$$D\text{ensity at } 23°C,\ M\text{g/m}^3 = PS/[(P+K) - F] \quad (2)$$

where:

P = mass of rubber chemical used,
S = density of the immersion liquid,
K = mass of the pycnometer filled with immersion liquid, and
F = final mass of the pycnometer with rubber chemical and immersion liquid.

11. Report

11.1 Report the following information:
11.1.1 Sample identification
11.1.2 Density at 23°C, Mg/m^3, and
11.1.3 Immersion liquid used.

12. Precision and Bias

12.1 *Precision*—This test method has not been tested for reproducibility or repeatability, but duplicate determinations on the same sample should not differ by more than 0.02 Mg/m^3 at 23°C.

12.2 *Bias*—No statement about bias is being made at this time.

13. Keywords

13.1 density; rubber chemicals