Designation: D1025 – 10

Standard Test Method for Nonvolatile Residue of Polymerization-Grade Butadiene[1, 2]

This standard is issued under the fixed designation D1025; 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 nonvolatile material in polymerization-grade butadiene.

1.2 **WARNING**—Mercury has been designated by many regulatory 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—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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 and health practices and to determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 A measured volume of liquid butadiene is allowed to evaporate at room temperature from a small glass evaporating dish until only residue remains. The evaporation is then completed by heating the dish to a constant weight.

3. Significance and Use

3.1 This test method is used to determine if there is any heavy material in the butadiene. It is possible that these materials could be deleterious to a polymerization reaction.

4. Apparatus

4.1 *Balance, Analytical*, capable of weighing to the nearest 0.1 mg.

4.2 *Evaporating Dish*, glass, 80 mm in diameter and 45 mm in height.

4.3 *Graduated Cylinder*, capacity of 25 mL, graduated in 0.2-mL subdivisions.

4.4 *Oven*, capable of maintaining a temperature of 105 ± 5°C.

4.5 *Cooling Vessel*, tightly covered, such as a glass desiccator, for cooling the evaporating dish before weighing.

4.6 *Thermometer*, range from −40 to 50°C, graduated in 1°C intervals, mercury-filled. Temperature measuring devices that cover the temperature range of interest, such as the ASTM 1C thermometer or liquid-in-glass thermometers, thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision may be used.

5. Preparation of Apparatus

5.1 Clean the evaporating dishes with a chromic-acid solution or other suitable cleaning agent before each test. (**Warning—**Causes severe burns. A recognized carcinogen. Strong oxidizer. Contact with organic material may cause fire.) Remove the dishes from the cleaning solution with stainless-steel forceps and handle only with forceps thereafter. Wash the dishes thoroughly, first with tap water, then with distilled water, and dry in the oven at 105°C for about 1 h, or until constant weight is obtained. Before weighing, cool the dishes for at least 30 min in the cooling vessel.

6. Procedure

6.1 Weigh the evaporating dish to the nearest 0.1 mg on the analytical balance.

6.2 Chill the evaporating dish to ice temperature. Cool the butadiene and the graduated cylinder to about −20°C. (**Warning—**Extremely flammable gas under pressure. May form explosive peroxides upon exposure to air. Harmful if inhaled. Irritating to eyes, skin, and mucous membranes.) Determine the sample temperature to the nearest 1°C and transfer 25 ± 1 mL of sample to the evaporating dish. Record the sample volume and temperature.

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[1] This test method is under the jurisdiction of Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.D0.04 on C4 Hydrocarbons.

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[2] This test method is an adaptation of one developed and cooperatively tested by the Butadiene Producer's Committee on Specifications and Methods of Analysis of the Office of Rubber Reserve. It appears in the Butadiene Laboratory Manual, Office of Rubber Reserve, as Method No. 2.1.56.2.

6.3 Allow the sample to evaporate at room temperature in a well-ventilated hood. When evaporation is complete, place the evaporating dish in an oven at 105 ± 5°C until a weight constant to 0.1 mg is obtained. Before each weighing, cool the dish for at least 30 min in the cooling vessel. Between each two weighings, place the dish in the oven for at least 30 min.

7. Calculation

7.1 Calculate the amount of nonvolatile residue by means of the following equation:

$$\text{Nonvolatile residue, weight \%} = [(B - A)/ Sd] \times 100$$

where:

A = weight of the evaporating dish, g
B = weight of the evaporating dish plus residue, g,
S = volume of the liquid butadiene sample, mL, and
d = density of the sample at the temperature of measurement, g/mL, found by using Table 1.

8. Precision and Bias

8.1 *Precision*—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

8.1.1 *Repeatability*—The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

TABLE 1 Density of Butadiene at Various Temperatures

NOTE—These data may be used in a graphical manner for better interpolation between data points.

Temperature, °C	Density, g/mL
–45	0.6958
–40	0.6903
–35	0.6848
–30	0.6793
–25	0.6737
–20	0.6681
–15	0.6625
–10	0.6568
–5	0.6510
0	0.6452

0.02 %

8.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

0.04 %

8.1.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method for measuring non-volatile residue, no statement on bias is being made.

9. Keywords

9.1 butadiene; nonvolatile residue