

Designation: D1025 – 10 (Reapproved 2015)^{ε1} D1025 – 10 (Reapproved 2021)

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.

 ε^1 NOTE—Subsection 5.6 was revised editorially in May 2015.

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 materialsubstance that can cause eentral nervous system, kidney and liver damage. serious medical issues. Mercury, or its vapor, may has been demonstrated to be hazardous to health and corrosive to materials. Caution should be taken—Use Caution when handling mercury and mercury eontaining 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 (SDS) for additional information. The potential exists that selling mercury and/or mercury containing products into your state or country may be prohibited by law-or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.
- 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 safety, health, and health environmental practices and to 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:³

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Summary of Test Method

3.1 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.

¹ This test method is under the jurisdiction of Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.D0.04 on C4 and C5 Hydrocarbons.

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.

³ 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.

4. Significance and Use

4.1 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.

5. Apparatus

- 5.1 Balance, Analytical, capable of weighing to the nearest 0.1 mg.
- 5.2 Evaporating Dish, glass, 80 mm in diameter and 45 mm in height.
- 5.3 Graduated Cylinder, capacity of 25 mL, graduated in 0.2 mL subdivisions.
- 5.4 Oven, capable of maintaining a temperature of 105 °C \pm 5 °C.
- 5.5 Cooling Vessel, tightly covered, such as a glass desiccator, for cooling the evaporating dish before weighing.
- 5.6 Thermometer, range from -40 °C to 50 °C, graduated in 1 °C intervals, conforming to the requirements for ASTM Thermometer 1C as prescribed in Specification E1 is satisfactory. 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.

6. Preparation of Apparatus

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6.1 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.

7. Procedure

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- 7.1 Weigh the evaporating dish to the nearest 0.1 mg on the analytical balance.
- 7.2 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 mL \pm 1 mL of sample to the evaporating dish. Record the sample volume and temperature.
- 7.3 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 $105 \,^{\circ}\text{C} \pm 5 \,^{\circ}\text{C}$ 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.

8. Calculation

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

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.