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Standard Guide for Analysis of 1,3–Butadiene Product[1]

This standard is issued under the fixed designation D5274; 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 guide covers the analysis of 1,3–butadiene products produced in North America. It includes possible components and test methods, both ASTM and other, either actually used or believed to be in use, to test for these components. This guide is not intended to be used or construed as a set of specifications for butadiene products.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

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]

D1025 Test Method for Nonvolatile Residue of Polymerization-Grade Butadiene
D1157 Test Method for Total Inhibitor Content (TBC) of Light Hydrocarbons
D1550 Standard ASTM Butadiene Measurement Tables
D2384 Test Methods for Traces of Volatile Chlorides in Butane-Butene Mixtures
D2426 Test Method for Butadiene Dimer and Styrene in Butadiene Concentrates by Gas Chromatography
D2593 Test Method for Butadiene Purity and Hydrocarbon Impurities by Gas Chromatography
D3246 Test Method for Sulfur in Petroleum Gas by Oxidative Microcoulometry
D3700 Practice for Obtaining LPG Samples Using a Floating Piston Cylinder
D4178 Practice for Calibrating Moisture Analyzers
D4423 Test Method for Determination of Carbonyls in C_4 Hydrocarbons
D4468 Test Method for Total Sulfur in Gaseous Fuels by Hydrogenolysis and Rateometric Colorimetry
D4629 Test Method for Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection
D4864 Test Method for Determination of Traces of Methanol in Propylene Concentrates by Gas Chromatography
D5799 Test Method for Determination of Peroxides in Butadiene

3. Terminology

3.1 *Definitions:*

3.1.1 *1,3-butadiene*—hydrocarbon product containing more than 99 % 1,3-butadiene.

3.2 *Symbols:*

3.2.1 *BHT*—butyl hydroxy toluene.

3.2.2 *GC*—gas chromatography.

3.2.3 *pTBC*—paratertiary butyl catechol.

3.2.4 *4VCH-1*—4-vinyl cyclo hexene (1,3-butadiene dimer).

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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.

4. Significance and Use

4.1 This guide is intended to provide information on the possible composition of 1,3-butadiene products and possible ways to test them. Since there are currently not enough ASTM standards for determining all components of interest, this guide provides information on other potentially available test methods.

4.2 Although this guide is not to be used for specifications, it can provide a starting point for parties to develop mutually agreed-upon specifications that meet their respective requirements. It can also be used as a starting point in finding suitable test methods for 1,3-butadiene components.

5. Sampling

5.1 *General:*

5.1.1 1,3-butadiene is a very reactive hydrocarbon. It reacts with oxygen to form peroxides and to polymerize. It also dimerizes at a rate that is temperature dependent. Below 10°C (50°F), the dimerization rate is less than 1 mg/kg by mass/h; but, at 20°C (77°F), it increases to 3 to 4 mg/kg mass/h; and at 40°C (104°F), to 14 to 20 mg/kg mass/h. 1,3-butadiene is also classified as toxic and as a potential health hazard, having been found carcinogenic to laboratory animals. Therefore, sampling of 1,3-butadiene must adhere to the following three principles:

5.1.1.1 Minimize personnel exposure. See the appropriate OSHA Material Safety Data Sheet for guidance,

5.1.1.2 Eliminate or keep to an absolute minimum the inclusion of oxygen during and after sampling, and

5.1.1.3 Sample the product at as low a temperature as possible, maintain the sample at a low temperature, and analyze it as soon as possible. Do not allow it to sit outdoors in the sun after sampling.

5.1.2 In addition to 5.1.1.1 – 5.1.1.3, 1,3-butadiene to be analyzed for trace components should be sampled by a technique that minimizes or eliminates loss of light components and concentration of heavy ones. The subsections below list some different sampling methods and principles. However, it is not the intent of this guide to list procedures that are applicable to all sampling situations. It is strongly recommended that samples be obtained under the supervision of a person with wide knowledge and experience in sampling 1,3-butadiene.

5.1.3 Also, even though this guide does not address the location of a sampling point in a line or vessel, the importance of the proper sampling location cannot be overemphasized.

5.2 *Floating Piston Cylinder*—Practice D3700 meets the criterion of minimizing or eliminating loss of light components and concentration of heavy ones. However, some labs have safety codes preventing use of rupture-disc piston containers. Alternative procedures must be used in these labs.

5.3 *Conventional "Outaging" Method*—The widely used "outaging" technique (that is, the practice of removing a portion of the fluid contents from a conventional sampling cylinder after filling in order to provide expansion room) causes a partial loss of light components into the vapor space. Subsequent handling to recapture these light ends in the liquid phase of the sample, such as repressurization of the cylinder contents with an inert gas, is usually successful, since 1,3-butadiene seldom contains noncondensables. However, if permanent gases are present and are to be determined, an alternate procedure may be required.

5.4 *Vaporization Methods*—Vaporization of the sample, either at the source or in the lab prior to analysis, may cause loss of heavier components, if present, and concentration of lighter ones. Also, since 1,3-butadiene is so reactive, the heat required to vaporize may cause undesirable changes in the composition of the sample. For these reasons, vaporization is not recommended for 1,3-butadiene.

5.5 *Reactive Components*—Determination of reactive components, such as certain sulfur compounds, is generally believed to require special sample containers, such as TFE-fluorocarbon-lined cylinders.

6. Composition and Test Methods

6.1 Table 1 indicates possible composition ranges and ASTM methods for 1,3-butadiene product. Table 2 lists other test methods known or believed to be in use.

7. Keywords

7.1 1,3-butadiene; 1,3-butadiene product; 1,3-butadiene test methods