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An American National Standard

Standard Test Method for Butadiene Dimer and Styrene in Butadiene Concentrates by Gas Chromatography¹

This standard is issued under the fixed designation D 2426; 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 butadiene dimer (4-vinylcyclohexene-1) and styrene in butadiene concentrates, both "recycle" and specification grade.
- 1.2 The values stated in SI units are to be regarded as the 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. For specific precautionary statements see Note 1, Note 2, and Note 4.

2. Referenced Documents

2.1 ASTM Standards:

- D 1657 Test Method for Density or Relative Density of Light Hydrocarbons by Pressure Thermohydrometer²
- D 1945 Test Method for Analysis of Natural Gas by Gas Chromatography³
- D 2593 Test Method for Butadiene Purity and Hydrocarbon Impurities by Gas Chromatography² ASTM D24

3. Summary of Test Method

3.1 The sample is introduced into a gas-liquid partition column. The components of interest are separated as they are transported through the column by a carrier gas, and their presence in the effluent is detected and recorded as a chromatogram. Packed columns are used, and either thermal conductivity or ionization detectors are permissible. The quantity of the components of interest present in the sample is determined from the chromatogram by comparing their peak areas or heights with those obtained from a synthetic sample.

4. Significance and Use

4.1 Butadiene dimer and styrene may be present as impurities in commercial butadiene. This test method is suitable for use in internal quality control and in establishing product specifications.

5. Apparatus

- 5.1 Chromatograph—Any chromatograph having either a thermal conductivity or flame ionization detector may be used. The detector system shall have sufficient sensitivity to obtain a deflection of at least 2 mm at a signal-to-noise ratio of at least 5:1 for 0.01 weight % of butadiene dimer and styrene under the operating conditions prescribed in this test method.
- 5.2 Recorder—A 0 to 1-mV, 0 to 5-mV, or 0 to 10-mV recorder with a full-scale response time of 2 s or less, and with sufficient sensitivity to meet the requirements of 5.1.
- 5.3 Column—Any column may be used that is capable of resolving the butadiene dimer and styrene as discrete peaks, quantitatively proportional to concentration and within an elapsed time sufficiently short to be practical. (See Note 1.)
- 5.4 Liquid Sampling Valve—Any liquid sampling valve may be used that will permit the reproducible introduction of the butadiene concentrate as a liquid under its vapor pressure or higher and in a quantity sufficient to meet the sensitivity and resolution requirements in 5.1 and 5.3, respectively.⁴

6. Reagents and Materials

- 6.1 4-Vinylcyclohexene-1 and Styrene, for calibration, purity of not less than 99 %.
- 6.2 Carrier Gas—Helium or hydrogen for use on thermal conductivity detector units; or nitrogen, helium, or argon for use on ionization detector units.

Note 1—Warning: Compressed gas. Hazardous pressure.

Note 2—Warning: Hydrogen gas is flammable. Hazardous pressure.

6.3 Liquid Phase, for column.

Note 3—The following materials have been used successfully as liquid phases:

 $^{^1}$ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.D0.04on C_ς Hydrocarbons.

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² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 05.05.

⁴ Suitable valves are commercially available.