

SUPERSEDED
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Standard Method of Test for

SEPARATION OF RESIDUE FROM BUTADIENE^{1, 2}



APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
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REAFFIRMED 1960

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ADOPTED, 1952.³

This Standard of the American Society for Testing Materials is issued under the fixed designation D 1023; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test describes a procedure for the separation of polymerization grade or recycle butadiene into a distillate consisting of butadiene together with C_3 and lighter hydrocarbons, and a residue containing butadiene dimer (4-vinyl-1-cyclohexene), nonvolatile matter, and styrene if present.

NOTE 1.—If the butadiene sample is to be analyzed only for dimer or styrene, or both, so that it is unnecessary to collect the distillate, use the distillation procedure of ASTM Method D 1024, Test for Butadiene Dimer and Styrene in Butadiene Concentrates.⁴

¹ Under the standardization procedure of the Society, this method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.

² This method is an adaptation of one developed and cooperatively tested by the Butadiene Producer's Committee on Specifications and Methods of Analyses 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 Prior to adoption as standard, this method was published as tentative from 1949 to 1952.

Published first as information in *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 309 (1948).

⁴ 1959 Supplement to Book of ASTM Standards, Part 7.

Outline of Method

2. The sample is distilled through a small, simply designed fractionating column with rapid throughput and slight reflux (Fig. 1). The overhead vapors are condensed for use in analyses requiring samples free from dimer, styrene, and nonvolatile matter. The residue is further purified for analyses for dimer or styrene, or both.

NOTE 2.—The separation is sufficiently quantitative so that the distillate may be used in analyses, such as those for butadiene and C_3 hydrocarbons, in which dimer, nonvolatile matter, and styrene interfere. However, the residue must be further purified to remove traces of C_3 and C_4 hydrocarbons before its analysis for dimer or styrene, or both.

Apparatus

3. (a) *Fractionating Column*, vacuum-jacketed and packed with $\frac{1}{8}$ -in. glass helices, constructed as detailed in Fig. 1.

(b) *Distillation Flask*, flat-bottomed, 125-ml capacity, with 24/40 standard taper ground-glass joint.

(c) *Receiver*, as shown in Fig. 1.

(d) *Graduated Cylinder*, capacity of