



Standard Test Method for OXYGEN IN LIGHT HYDROCARBON VAPORS^{1,2}

This Standard is issued under the fixed designation D 1021; 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.

1. Scope

1.1 This method² covers the determination of low concentrations of oxygen (0.02 to 6.0 percent) in the vapor above liquid butadiene when the sample does not contain such oxidizing or reducing gases as hydrogen sulfide, sulfur dioxide, and the oxides of nitrogen.

2. Summary of Method

2.1 A vapor-phase sample of butadiene is shaken with a freshly prepared aqueous suspension of manganous hydroxide, and the resulting manganese hydroxide is titrated iodometrically with standard thiosulfate solution, using starch indicator.

3. Apparatus

3.1 *Flask*, 500-ml Erlenmeyer, with 24/40 standard-taper joint, heavy-walled to withstand pressure, as shown in Fig. 1. An adaptor shall be provided having a side-connecting tube with a stopcock connected with elastic tubing, and having a graduated open-top separatory funnel of 25-ml capacity and calibrated every 5 ml, sealed on top. The stem of the funnel shall extend to within approximately 5 mm of the bottom of the flask.

3.2 *Vacuum Source*, able to evacuate to 0.1 mm Hg pressure.

3.3 *Mechanical Agitator*, giving vigorous agitation, in which the liquid shall be thrown from side to side in the flask, breaking and reestablishing the liquid surface on each cycle.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the

reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.1.1 *Manganous Chloride Solution (50 g MnCl₂/liter)*—Dissolve 78.6 g of manganous chloride (MnCl₂·4H₂O) in water and dilute to 1 liter.

4.1.2 *Potassium Dichromate, Standard Solution (0.1 N)*—Dissolve 4.9035 g of dried potassium dichromate (K₂Cr₂O₇) in water and dilute to 1 liter in a volumetric flask.

4.1.3 *Potassium Iodide Solution (150 g/liter)*—Dissolve 150 g of potassium iodide (KI) in water and dilute to 1 liter.

4.1.4 *Sodium Hydroxide Solution (100 g/liter)*—Dissolve 100 g of sodium hydroxide (NaOH) pellets in water and dilute to 1 liter.

4.1.5 *Sodium Thiosulfate, Standard Solution (0.1 N)*—Dissolve 25 g of sodium thiosulfate (Na₂S₂O₃·5H₂O) in 1 liter of water and allow to stand 1 week before standardization. To standardize, pipet 25 ± 0.05 ml of 0.1 N K₂Cr₂O₇ solution into a flask containing 10 ml of KI solution (150 g/liter), 5 ml of HCl (sp gr 1.19), and 200 ml of water. After allowing the mixture to stand 5 to 10 min, titrate with the Na₂S₂O₃ solution. Toward the end of the titration, add 1 ml of starch indi-

¹ This method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants.

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² This method is an adaptation of one developed and cooperatively tested by the Butadiene Producers' 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 2.1.8.1.

³ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N.Y., and the "United States Pharmacopeia."