



Designation: D1157 – 91(Reapproved 2009)

Standard Test Method for Total Inhibitor Content (TBC) of Light Hydrocarbons^{1,2}

This standard is issued under the fixed designation D1157; 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 total *p*-tertiary-butylcatechol inhibitor added to polymerization and recycle grades of butadiene or to other C₄ hydrocarbon mixtures containing no phenolic material other than catechol or no oxidized phenolic material other than that derived from oxidation of catechol. In general, all phenols and their quinone oxidation products are included in the calculated catechol content. Small amounts of polymer do not interfere. This test method is applicable over the range of TBC from 50 to 500 mg/kg.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.*

2. Referenced Documents

2.1 *ASTM Standards*:³

D1265 Practice for Sampling Liquefied Petroleum (LP) Gases, Manual Method

3. Summary of Test Method

3.1 The catechol is separated from the butadiene by evaporation. The residue is dissolved in water and an excess of ferric chloride is added. The intensity of the yellow-colored complex is compared in a photoelectric colorimeter with that produced by known concentrations of the catechol.

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

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² This test method was derived from the method developed and cooperatively tested by the Butadiene Producers' Committee on Specifications and Methods of Analysis of the Office of Rubber Reserve, which appears in the Butadiene Laboratory Manual as Method 2.1.9.1.

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 *p*-tertiary-butylcatechol is commonly added to commercial butadiene in amounts of 50 to 250 mg/kg as an oxidation inhibitor. This test method is suitable for use by both producers and users of butadiene within the limitations described in Section 1.

5. Apparatus

5.1 *Photometer*—A sensitive photoelectric photometer capable of producing light of narrow spectral range that is predominantly blue (425 nm).

5.2 *Graduates*, 100-mL.

5.3 *Volumetric Flasks*, 100-mL; or stoppered graduated mixing cylinder, 100-mL.

5.4 *Erlenmeyer Flasks*, 250-mL.

5.5 *Funnels*, 75-mm diameter.

5.6 *Pipet*, 5-mL.

6. Reagents

6.1 *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.⁴ 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.

6.2 *Purity of Water*—References to water shall be understood to mean distilled water.

6.3 *Ferric Chloride, Standard Solution*—Dissolve 20.0 g of ferric chloride (FeCl₃·6H₂O) in ethanol (95 %). Add 9.2 mL of HCl (sp gr 1.19), and then dilute with ethanol (95 %) to 1000 mL in a volumetric flask.

6.4 *p-Tertiary-Butylcatechol, Standard*—(Warning—Potentially hazardous. May cause skin irritation or burns; can

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.