



Designation: D8301 – 21

Standard Test Method for Determination of the Content of Nitroxide Radical (4-Hydroxy-2,2,6,6-tetramethylpiperidine-1-oxyl) in Butadiene, Styrene and Isoprene by Cyclic Voltammetry Method¹

This standard is issued under the fixed designation D8301; 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 is designed to determine the content of nitroxide radical 4-hydroxy-2,2,6,6-tetramethylpiperidine-1-oxyl (H-Tempo) in butadiene, isoprene, and styrene.

1.2 This test method is applicable to samples with nitroxide radical 4-hydroxy-2,2,6,6-tetramethylpiperidine-1-oxyl (H-Tempo) with concentrations to 100 mg/kg. The limit of detection (LOD) is 0.47 mg/kg for nitroxide radical 4-hydroxy-2,2,6,6-tetramethylpiperidine-1-oxyl (H-Tempo) and the limit of quantitation (LOQ) is 1.6 mg/kg nitroxide radical 4-hydroxy-2,2,6,6-tetramethylpiperidine-1-oxyl (H-Tempo).

1.3 The following applies for the purposes of determining the conformance of the test results using this test method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

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

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

2.2 Other Documents:

OSHA Regulations, 29 CFR, paragraph 1910.1000 Air Contaminants³

OSHA Regulations, 29 CFR, paragraph 1910.1200 Hazard Communication³

3. Summary of Test Method

3.1 A sample of butadiene is dissolved in acetonitrile. Butadiene is separated by evaporation. The intensity of the cathode current signal is compared in the cyclic voltammetry (CV) with that produced by the known concentration of the H-Tempo.

3.2 A sample of isoprene and styrene is dissolved in acetonitrile. The intensity of the cathode current signal is compared in the cyclic voltammetry (CV) with that produced by the known concentration of the H-Tempo.

4. Significance and Use

4.1 Nitroxide radicals (H-Tempo, O-Tempo (4-oxo-2,2,6,6-tetramethylpiperidine-1-oxyl), etc.) are widely used as inhibitors of thermopolymerization in the processes of transportation, storage, and separation of monomers (isoprene, butadiene, styrene, etc.). This test method provides a procedure for assaying nitroxide radicals in monomers.

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.07 on Styrene, Ethylbenzene and C9 and C10 Aromatic Hydrocarbons.

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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.

³ Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, <http://www.access.gpo.gov>.