



Standard Test Method for Assay of Di-*tert*-Butyl Peroxide Using Gas Chromatography¹

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1. Scope*

1.1 This test method covers the assay of di-*tert*-butyl peroxide using gas chromatography. It is applicable to commercial di-*tert*-butyl peroxide which may contain small amounts of isobutylene, *tert*-butanol, *tert*-butyl hydroperoxide, triisobutylenes, and water as impurities.

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.* Specific hazards statements are given in Section 9.

2. Referenced Documents

2.1 *ASTM Standards*:²

E203 Test Method for Water Using Volumetric Karl Fischer Titration

3. Terminology

3.1 *Definitions*:

3.1.1 *active oxygen*—the oxidizing power present in organic peroxides expressed as oxygen (equivalent = 8.00).

4. Summary of Test Method

4.1 A sample is diluted in dodecane and injected into a gas chromatograph containing a wide-bore capillary column with a nonpolar stationary phase. A temperature program is used to separate di-*tert*-butyl peroxide from impurities using helium as a carrier gas. The flame ionization detector response, proportional to component concentration, is recorded and the peak

areas are measured. The concentration of di-*tert*-butyl peroxide is determined by area normalization after correcting for the area of the solvent peak. The assay of di-*tert*-butyl peroxide is corrected for water, which is determined using a separate Karl Fischer titration since water is not detected using flame ionization detection. The area percent of di-*tert*-butyl peroxide is assumed to be equal to mass percent.

5. Significance and Use

5.1 Di-*tert*-butyl peroxide is widely used as a catalyst and reaction initiator. Knowledge of the peroxide content is important in such applications. This test method provides a procedure for determining the active peroxide content of commercial di-*tert*-butyl peroxide.

6. Interferences

6.1 Interferences will be encountered if other components are present in the sample that have the same retention time as di-*tert*-butyl peroxide.

7. Apparatus

7.1 *Instrumentation*—Gas chromatograph, capable of column oven temperature programming from 40 to 160°C at a rate of 10°C/min and from 160 to 240°C at a rate of 35°C/min.

7.2 *Injection System*—Glass-lined sample injection port, maintained at 150°C.

7.3 *Sample Introduction*—Microliter syringes or automatic syringe injectors, capable of delivering 0.5 μ L of a liquid sample, have been used successfully.

7.4 *Detection*—Flame ionization detector, maintained at 250°C.

7.5 *Data Acquisition System*—Electronic data acquisition and area integration capabilities are recommended.

7.6 *Chromatographic Column*—30 m wide-bore capillary column (0.53 mm inside diameter) with a 5.0 μ m dimethylpolysiloxane stationary phase.

8. Reagents and Materials

8.1 *Carrier Gas (Helium)*, chromatographic grade.

8.2 *Hydrogen*, chromatographic grade.

8.3 *Compressed Air*, oil-free.

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.16 on Industrial and Specialty Product Standards.

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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.