



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 Review the current appropriate Material Safety Data Sheets (MSDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

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:

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals²

E 203 Test Method for Water Using Karl Fischer Reagent²

3. Terminology

3.1 Definitions:

3.1.1 *active oxygen*—the oxidizing power present in organic peroxides expressed as oxygen (equivalent weight = 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 weight 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 µL 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 µm dimethylpolysiloxane stationary phase.³

8. Reagents and Materials

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

8.2 *Hydrogen*, chromatographic grade.

8.3 *Compressed Air*, oil-free.

8.4 *Dodecane*, for use as a sample diluent, 99 % or greater chromatographic purity.

9. Hazards

9.1 Organic peroxides are strong oxidizing agents and present potential fire and explosion hazards. While di- *tert*-butyl peroxide is one of the more stable peroxides, contact with

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² *Annual Book of ASTM Standards*, Vol 15.05.

³ A J & W Scientific DB-1, available from J & W Scientific, 91 Blue Ravine Rd., Folsom, CA 95630-4714, was utilized to develop this test method.