



# Standard Test Method for Analysis of 2,6-Ditertiary-Butyl Para-Cresol and 2,6-Ditertiary-Butyl Phenol in Insulating Liquids by Gas Chromatography<sup>1</sup>

This standard is issued under the fixed designation D 4768; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination by gas chromatography of 2,6-ditertiary-butyl para-cresol and 2,6-ditertiary-butyl phenol in new and used insulating liquids at concentrations up to 0.5 %. It includes the determination in Type I and II insulating mineral oils as specified in Specification D 3487, but has also been used to measure these inhibitors in other insulating liquids, such as esters and high fire-point hydrocarbons.

1.2 *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:

- D 923 Test Method for Sampling Electrical Insulating Liquids<sup>2</sup>
- D 3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus<sup>2</sup>
- D 5222 Guide for High-Fire Point Electrical Insulating Oils of Petroleum Origin<sup>2</sup>
- E 260 Practice for Packed Column Gas Chromatography<sup>3</sup>

## 3. Summary of Test Method

3.1 The test specimen is placed onto a column containing activated alumina and extracted to remove interfering substances. The inhibitors are then eluted from the column with suitable solvent and analyzed by gas chromatography. The inhibitor type and quantity are determined by comparison of each component with a working standard tested under similar conditions.

## 4. Significance and Use

4.1 In new electrical insulating oil, this test method provides a quantitative measure of the amounts of 2,6-ditertiary-butyl para-cresol and 2,6-ditertiary-butyl phenol that have been added to the oil. In a used oil, the test measures the amount of these inhibitors remaining in the oil. This test method is suitable for manufacturing control, specification acceptance, and service evaluation.

4.2 This test method is used to separate, identify, and quantify the inhibitors with minimal interference and matrix effects.

4.3 This test method has also been used successfully to determine the inhibitor concentrations in other insulating liquids such as esters and high-temperature hydrocarbons.

## 5. Apparatus

5.1 *Gas Chromatograph*, equipped with oven temperature control constant to 1°C and with heated injector port.

5.1.1 *Means to Record the Chromatogram*, such as a pen recorder or a digital integrator to determine peak areas, is recommended. An automated sample injector may be used.

5.2 *Flame Ionization Detector*, with appropriate hydrogen/air gas flows, is preferred over a thermal conductivity detector to provide maximum sensitivity.

5.3 *Column*, a suitable stainless steel or glass column packed with a nonpolar silicone on an appropriate support.

NOTE 1—A 3 % OV-1<sup>4</sup> on 100/120 Mesh Supelcoport,<sup>5</sup> 1.83 m (6 ft) long, 3.2 mm (0.125 in.) in outside diameter has been used successfully.

5.3.1 Condition columns in accordance with manufacturer's recommendations. Disconnect columns from detector prior to conditioning and reconnect after conditioning.

5.4 *Precision Syringe*, glass, 10.0  $\mu$ L.

5.5 *Volumetric Glassware*, appropriate for making dilutions.

5.6 *Pipets*, Pasteur, disposable, 146 by 7.5 mm.

5.7 *Analytical Balance*.

5.8 *Automatic Pipetter*, 1 mL calibrated, adjustable.

5.9 *Oven*, capable of maintaining a temperature of 275  $\pm$  5°C for conditioning extraction columns.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.03 on Analytical Tests.

Current edition approved March 10, 2003. Published May 2003. Originally approved in 1988 as D 4768 – 88. Last previous edition approved in 1996 as D 4768 – 96.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 10.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.01.

<sup>4</sup> Registered trademark of Ohio Valley Specialty Co.

<sup>5</sup> Registered trademark of Supelco, Inc.