



Designation: D3156 – 96 (Reapproved 2021)

Standard Practice for Rubber—Chromatographic Analysis of Antidegradants (Antioxidants, Antiozonants and Stabilizers)¹

This standard is issued under the fixed designation D3156; 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 practice covers the detection and identification by thin-layer chromatography of antidegradants (antioxidants, antiozonants, and stabilizers) that may be present in raw rubber or rubber products. Analysis for other types of antidegradants is possible as long as the requirements of the practice are met.

1.2 The values stated in SI units are to be regarded as the 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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 Document

2.1 *ASTM Standards:*²

D297 Test Methods for Rubber Products—Chemical Analysis

3. Summary of Practice

3.1 A simplified procedure (Method A, Section 13.1) based on a single-solvent system is presented, which provides for the identification of known materials. This may be used to check the presence or absence in a rubber vulcanizate or raw rubber, of an antidegradant which should be present. However, since

the chromatograms obtained may not be absolutely specific for any given antidegradant, a more detailed scheme is given in 13.2.

3.2 Method B, Section 13.2, using additional solvents and sprays is included, which enables a greater degree of separation of the spots to be made and therefore may enable detection and identification of an unknown antidegradant.

3.3 Other techniques, for example: gas chromatographic (with or without treatment to obtain derivatives), spectroscopic (ultraviolet and infrared), more sophisticated thin-layer variations (two dimensional techniques), may be applied to identification of spots for improved detection and identification of an unknown antidegradant.

3.4 It is possible that the chromatographic pattern of one antidegradant may overlap that of another antidegradant; therefore each laboratory must prepare its own reference standards, based on the technique chosen from this practice.

4. Significance and Use

4.1 This practice is useful for the examination of rubber compounds or products for the presence of chemicals that prevent or greatly reduce degradation due to oxygen, ozone, or other agents.

4.2 This practice is suitable for quality assurance, factory control, and research and development applications.

5. Interferences

5.1 In the absence of extender oils, antidegradants are extracted from the rubber by a solvent and the evaporated extract is applied directly to a thin-layer chromatographic plate.

5.2 In the presence of extender oils, the oils are removed by either a pre-treatment of the plate, with light petroleum ether or by a column chromatographic technique.

5.3 Identification of the antidegradant is made by the standard technique of thin-layer chromatography, herein described.

6. Apparatus

6.1 *Spreading Device*, for making thin layer chromatographic plates with a coating 250 to 300 μm thick.

¹ This practice is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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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.