



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

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

2. Referenced Document

2.1 ASTM Standards:

D 297 Test Methods for Rubber Products—Chemical Analysis²

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.

6.2 *Glass Plates*, 200 by 200-mm, or suitable for the selected tank. As an alternative to preparing plates, the use of precoated plates with a coating of 250 to 300 μm is permitted. Precoated film-backed plates are not recommended.

6.3 *Drying Oven*, 105°C minimum.

6.4 *Desiccator*, or drying box for storing plates at fixed humidity.

6.5 *Micro-pipets*, 5 and 10-mm³ (μL).

6.6 *Chromatographic Developing Tank*, of a size suitable to hold plates, approximately 250 by 250 by 70 mm to 330 by 240 by 110 mm. Small “sandwich-type” tanks are not recommended. Add about 200 cm³ of developing solvent (7.4) to the tank, swirl, cover, and allow to stand 15 min before using. Tank may be reused by repeating the swirling and standing step, as long as the solvent ratio remains constant.

6.7 *Extraction Apparatus*, in accordance with Section 18 or 25 of Test Methods D 297.

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

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² *Annual Book of ASTM Standards*, Vol 09.01.