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Rubber and rubber products — Identification of antidegradants — Thin layer chromatographic methods

Caoutchouc et produits à base de caoutchouc — Identification des agents de protection — Méthodes par chromatographie en couche mince

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis.*

This second edition cancels and replaces the first edition (ISO 4645:1984), which has been technically revised. It also incorporates the Technical Corrigendum ISO 4645:1984/Cor.1:1991.

The main changes are as follows:

— the description of the principle has been improved;

— method A has been modified.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rubber and rubber products — Identification of antidegradants — Thin layer chromatographic methods

1 Scope

This document describes two methods for identification of antidegradants (antioxidants, antiozonants and stabilizers) which can be present in raw rubber, unvulcanized compounded rubber, or rubber products, by thin layer chromatography.

Method A is a simplified method that provides for the identification of known materials and can be used to check the presence or absence of a particular antidegradant which is expected to be present.

Method B is a more detailed method that enables a greater degree of separation of the spots to be obtained and therefore can be used to detect and identify an unknown antidegradant.

Antidegradants to which these methods are applicable include phosphited polyalkyl phenols, substituted bisphenols, secondary amines, substituted cresols and substituted p-phenylenediamines. Examination for other types of antidegradants is possible under the same condition when there is a standard chromatogram.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1407, *Rubber — Determination of solvent extract*

ISO 4661-2, *Rubber, vulcanized — Preparation of samples and test pieces — Part 2: Chemical tests*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

Due to the different chemical structures of antioxidants and their transformations, the partition coefficients in the liquid-solid phase are different. Antidegradants are extracted from the rubber with a solvent. The extraction solution is deposited in the form of spots on a thin layer silica gel chromatographic plate or a glass plate coated with silica gel.

If extender oil is present, the oil is removed either by column chromatography of the extract prior to the completion of the evaporation of the original solvent or by the development of the plate in light petroleum prior to the normal development in an appropriate solvent.

The colour and the shape are reported. The ratio shift value R_f of the spots of the corresponding antioxidant in the colour map is calculated