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Rubber — Identification of antidegradants by gas chromatography/mass spectrometry

Caoutchouc — Identification des antidégradants par chromatographie en phase gazeuse/spectrométrie de masse

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

For an explanation on 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 the following URL: 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 10638:2010), which has been technically revised with the following changes:

— trap coolant has been removed from the reagents;

— a calibration procedure for the apparatus has been added;

— test conditions have been added;

— a data analysis clause (Clause 7) has been added.

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Introduction

Most rubber products contain antidegradants to extend the life of the product, the type of antidegradant depending on the service conditions to which a particular product will be exposed. Doubts are increasingly being expressed about the negative impact which rubber containing certain antidegradants can have on the environment. However, demonstrating the presence of antidegradants in rubber products is not easy.

There are methods of qualitative analysis, specified in International Standards such as ISO 4645, which use thin-layer chromatography. This requires a highly skilled operator with a great amount of knowledge and experience, as well as the use of standard reference materials.

The gas chromatography/mass spectrometry technique specified in this document is an efficient method suitable for identifying antidegradants contained in rubber products, as well as in the raw-rubber and the unvulcanized-rubber compounds used to make such products.

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Rubber — Identification of antidegradants by gas chromatography/mass spectrometry

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

WARNING 2 — Certain procedures specified in this document might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies a method using gas chromatography/mass spectrometry, for the identification of antidegradants in raw rubbers, latices, unvulcanized-rubber compounds and vulcanized-rubber products. It is applicable to the 31 types of antidegradant listed in Annex A. The method specified is qualitative and is not intended for quantitative analysis.

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 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

ISO 1407:2011, *Rubber — Determination of solvent extract*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

Antidegradants are recovered from samples by thermal desorption or solvent extraction. For thermal-desorption method, the fume generated from heating test samples is to be analysed after being separated through the gas chromatograph/mass spectrometer which is connected to a heating and desorbing device. For solvent-extraction method, the extract of test samples obtained by solvent extraction process is to be separated and analysed by the gas chromatograph/mass spectrometer. The type of antidegradant recovered is identified by the mass spectrum that is produced after passing the antidegradant through a gas chromatograph and mass spectrometer connected in tandem. The retention index can be used as a supplementary means of identification if necessary.

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