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Rubber compounding ingredients — Organic vulcanizing agents — Determination of organic peroxide content

Ingrédients de mélange du caoutchouc — Agents vulcanisants organiques — Détermination de la teneur en peroxyde organique

Reference number
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Foreword

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This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products,* Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 14932:2012), which has been technically revised.

The main changes are as follows:

— gas chromatography using packed column has been added in 8.3;

— the solvent has been changed from chloroform to toluene and isopropyl alcohol;

— tetrahydrofuran has been removed due to toxicity;

— CAS Registry Numbers (CAS RN) have been added;

— Annex D and the former Annex E have been merged as Annex D;

— Formula (D.1) has been corrected.

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Rubber compounding ingredients — Organic vulcanizing agents — Determination of organic peroxide content

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all 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 determine the applicability of any other restrictions.

1 Scope

This document specifies four methods for the determination of the content of the following groups of organic peroxides used as rubber vulcanizing agents. There are three titration methods and one gas-chromatography method.

a) titration method A for group a: Peroxyketals:

1,1-Di(*tert*-butylperoxy)cyclohexane (DTBPC; CAS Registry Number®[1]:3006-86-8)

1,1-Di(*tert*-butylperoxy)-2-methylcyclohexane (DBPMC; CAS RN 147217-40-1);

1,1-Di(*tert*-butylperoxy)-3,3,5-trimethylcylcohexane (DBPTC; CAS RN 6731-36-8);

2,2-Di(*tert*-butylperoxy)butane (DBPB; CAS RN 2167-23-9);

Butyl −4,4-di(*tert*-butylperoxy)valerate (BPV; CAS RN 995-33-5);

b) titration method B for group b: Diacyl peroxides:

Dibenzoyl peroxide (CAS RN 94-36-0);

Di(2,4-dichlorobenzoyl) peroxide (CAS RN 133-14-2);

Di(4-methylbenzoyl) peroxide (CAS RN 895-85-2);

c) titration method C for group c: Diaralkyl and alkyl-aralkyl peroxides:

Di(tert-butylperoxyisopropyl)benzene (CAS RN 2212-81-9);

Dicumyl peroxide (CAS RN 80-43-3);

tert-Butyl cumyl peroxide (CAS RN 3457-61-2);

d) gas-chromatography for dialkyl peroxides, using a capillary or packed column.

2,5-Dimethyl-2,5-di(tert-butylperoxy)hexane (CAS RN 78-63-7)

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 385, *Laboratory glassware — Burettes*

1) Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6353-1, *Reagents for chemical analysis — Part 1: General test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/

3.1
active oxygen
oxygen-centred radicals, liberated by organic peroxide, capable of initiating vulcanization of rubber compounds

3.2
peroxyketal
peroxide obtained by the reaction of ketone with *tert*-butyl hydroperoxide (TBHP) as follows:

$$2tert-\text{butyl}-\text{OOH}+\text{R}-\text{C}(\text{O})-\text{R}'\rightarrow(tert-\text{butyl}-\text{OO})_2-\text{CR}(\text{R}')+\text{H}_2\text{O}$$

3.3
diacyl peroxide
peroxide obtained by the reaction of benzoyl chloride with hydrogen peroxide as follows:

$$2\text{C}_6\text{H}_5-\text{C}(\text{O})-\text{Cl}+\text{H}_2\text{O}_2\rightarrow\text{C}_6\text{H}_5-\text{C}(\text{O})-\text{OO}-\text{C}(\text{O})-\text{C}_6\text{H}_5+2\text{HCl}$$

3.4
alkyl-aralkyl peroxide
diaralkyl peroxide
peroxide obtained by the reaction of benzyl alcohol with hydrogen peroxide in presence of sulfuric acid as follows:

$$2\text{C}_6\text{H}_5-\text{C}(\text{CH}_3)_2-\text{OH}+\text{H}_2\text{O}_2\rightarrow\text{C}_6\text{H}_5-\text{C}(\text{CH}_3)_2-\text{OO}-(\text{CH}_3)_2\text{C}-\text{C}_6\text{H}_5+2\text{H}_2\text{O}$$

3.5
dialkyl peroxide
peroxide obtained by the reaction of *tert*-butyl alcohol with hydrogen peroxide in presence of sulfuric acid as follows:

$$2\text{CH}_3-\text{C}(\text{CH}_3)_2-\text{OH}+\text{H}_2\text{O}_2\rightarrow\text{CH}_3-\text{C}(\text{CH}_3)_2-\text{OO}-(\text{CH}_3)_2\text{C}-\text{CH}_3+2\text{H}_2\text{O}$$

4 General

Some organic peroxides are treated as diluted with an inert solvent, or mixed with an inorganic filler, a raw or an uncured rubber compound as master batches for explosion protection. The undiluted or diluted peroxides are directly used for its content analysis, however the mixed peroxides with the filler or rubber need to be pre-treated to prepare a test sample for the content analysis. The pre-treatment procedure and the determination of the peroxide content in the mixture shall be as specified in Annex D.

The choice of the properties to be determined and the values required shall be agreed between the interested parties.

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