INTERNATIONAL STANDARD

ISO 14932

First edition
2012-01-15

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
ISO 14932:2012(E)

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Published in Switzerland

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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.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 14932 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*.

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

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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.

1 Scope

This International Standard 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 using capillary gas-chromatography.

1.1 Titration method A for group a) peroxyketals

— DTBPC: 1,1-Di(*tert*-butylperoxy)cyclohexane;

— DBPMC: 1,1-Di(*tert*-butylperoxy)-2-methylcyclohexane;

— DBPTC: 1,1-Di(*tert*-butylperoxy)-3,3,5-trimethylcylcohexane;

— DBPB: 2,2-Di(*tert*-butylperoxy)butane;

— BPV: Butyl -4,4-di(*tert*-butylperoxy)valerate.

1.2 Titration method B for group b) diacyl peroxides

— Dibenzoyl peroxide;

— Di(2,4-dichlorobenzoyl) peroxide;

— Di(4-methylbenzoyl) peroxide.

1.3 Titration method C for group c) diaralkyl and alkyl-aralkyl peroxides

— Di(*tert*-butylperoxyisopropyl)benzene;

— Dicumyl peroxide;

— *tert*-Butyl cumyl peroxide.

1.4 Capillary gas-chromatography for dialkyl peroxides

— 2,5-Dimethyl-2,5-di(*tert*-butylperoxy)hexane.

2 Normative references

The following referenced documents are indispensable for the application 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*

ISO 3696:1987, *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.

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

3.2
peroxyketal
peroxide obtained by the reaction of a ketone with *tert*-butyl hydroperoxide (TBHP) as shown in the following equation:

$$2\,tert\text{-}\mathrm{butyl-OOH} + \mathrm{R-C(O)-R'} \rightarrow (tert\text{-}\mathrm{butyl-OO})_2\mathrm{-CR(R')} + \mathrm{H_2O}$$

3.3
diacyl peroxide
peroxide obtained by the reaction of benzoyl chloride with hydrogen peroxide as shown in the following equation:

$$2\,\mathrm{C_6H_5-C(O)-Cl} + \mathrm{H_2O_2} \rightarrow \mathrm{C_6H_5-C(O)-OO-C(O)-C_6H_5} + 2\,\mathrm{HCl}$$

3.4
di-aralkyl peroxide
alkyl-aralkyl peroxide
peroxide obtained by the reaction of a benzyl alcohol with hydrogen peroxide in presence of sulfuric acid as shown in a simple case in the following equation:

$$2\,\mathrm{C_6H_5-C(CH_3)_2-OH} + \mathrm{H_2O_2} \rightarrow \mathrm{C_6H_5-C(CH_3)_2-OO-(CH_3)_2C-C_6H_5} + \mathrm{H_2O}$$

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

$$2\,\mathrm{CH_3-C(CH_3)_2-OH} + \mathrm{H_2O_2} \rightarrow \mathrm{CH_3-C(CH_3)_2-OO-(CH_3)_2C-CH_3} + \mathrm{H_2O}$$

4 Titration method A

4.1 Purpose

This test method specifies the procedure for the determination of the content of peroxyketals used as rubber organic vulcanizing agents and is applicable to DTBPC, DBPTC, DBPMC, DBPB and BPV.

4.2 Principle

Peroxyketales react with iodide in an acetic acid-hydrochloric acid medium, liberating an equivalent amount of iodine which is titrated with a standard sodium thiosulfate solution:

$$\mathrm{R-OO-R'} + 2\mathrm{I^-} + 2\mathrm{H^+} \rightarrow \mathrm{ROH} + \mathrm{R'OH} + \mathrm{I_2}$$