INTERNATIONAL STANDARD

ISO 9924-2

Second edition
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Rubber and rubber products — Determination of the composition of vulcanizates and uncured compounds by thermogravimetry —

Part 2: Acrylonitrile-butadiene and halobutyl rubbers

Caoutchouc et produits à base de caoutchouc — Détermination de la composition des vulcanisats et mélanges non vulcanisés par thermogravimétrie —

Partie 2: Caoutchoucs acrylonitrile-butadiène et butyl halogéné

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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 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.

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 9924-2:2000), of which it constitutes a minor revision. It also incorporates the amendment ISO 9924-2:2000/Amd 1:2012. The following changes have been made:

— precision clause moved to Annex A.

ISO 9924 consists of the following parts, under the general title *Rubber and rubber products — Determination of the composition of vulcanizates and uncured compounds by thermogravimetry*:

— *Part 1: Butadiene, ethylene-propylene copolymer and terpolymer, isobutene-isoprene, isoprene and styrene-butadiene rubbers*

— *Part 2: Acrylonitrile-butadiene and halobutyl rubbers*

— *Part 3: Hydrocarbon rubbers, halogenated rubbers and polysiloxane rubbers after extraction*

Rubber and rubber products — Determination of the composition of vulcanizates and uncured compounds by thermogravimetry —

Part 2: Acrylonitrile-butadiene and halobutyl rubbers

WARNING — Persons using this part of ISO 9924 should be familiar with normal laboratory practice. This part of ISO 9924 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

1.1 This part of ISO 9924 specifies a thermogravimetric method for the determination of the total organic content, carbon black content, carbonaceous residue and ash in vulcanizates and uncured compounds containing polymers which form a carbonaceous residue on pyrolysis (see 1.2). The loss in mass at around 300 °C is an approximate guide to the volatile-matter content of the compound.

1.2 The method is suitable for the analysis of rubber compounds and vulcanizates containing the hydrocarbon polymers covered by ISO 9924-1 plus the following copolymers with polar groups which form a carbonaceous residue on pyrolysis:

a) acrylonitrile-butadiene copolymers (NBRs);

b) carboxylated acrylonitrile-butadiene copolymers (XNBRs);

c) hydrogenated acrylonitrile-butadiene copolymers (HNBRs);

d) chlorinated isobutylene-isoprene copolymers (CIIRs);

e) brominated isobutylene-isoprene copolymers (BIIRs).

NOTE 1 The field of application of the method may be extended to include the analysis of compounds containing rubbers different from those given in this subclause, provided that the applicability of the method is tested beforehand using known compounds or vulcanizates having similar composition.

NOTE 2 The method is not suitable for compounds containing mineral fillers, such as carbonates or hydrated aluminium oxides, which decompose in the temperature range from 25 °C to 800 °C, unless suitable corrections based on prior knowledge of filler behaviour can be made.

NOTE 3 The method is not suitable for the determination of the total polymer content of compounds containing non-rubber organic ingredients that cannot be completely removed by solvent extraction carried out in accordance with ISO 1407.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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*

3 Principle

3.1 A weighed test portion is heated, following a pre-set programme, from 40 °C to 600 °C in a stream of nitrogen until all the organic material is pyrolysed. If nitrile-butadiene copolymers are present, a small amount of the polymer will be pyrolysed to form a carbonaceous residue that has to be determined in order to obtain the correct total organic content.

3.2 For this purpose, the oven temperature is decreased to 400 °C in a stream of nitrogen and maintained at 400 °C for 5 min. The gas stream is then changed from nitrogen to air or oxygen and the oven temperature raised slowly to 800 °C. During this heating in an oxidizing atmosphere, two distinct losses in mass occur due to combustion. The first is due to the combustion of the carbonaceous residue, and the amount is determined and added to the previously determined loss in nitrogen at 600 °C to obtain the correct value of the total organic content. The second is due to the normal combustion of the carbon black.

NOTE The total rubber content is calculated by subtracting the value of the solvent extract, determined in accordance with ISO 1407, from the total organic content, provided that all non-rubber ingredients can be extracted.

3.3 The mass of the residue at 800 °C represents the ash.

4 Reagents

4.1 **Dry nitrogen**, with an oxygen content of less than 10 mg/kg (ppm).

4.2 **Dry air or oxygen**.

5 Apparatus

5.1 **Thermogravimetric analyser**.

There are many types of analyser commercially available. All should be suitable for use with this part of ISO 9924, but their suitability should be checked using the procedure in Clause 6. Calibrate and operate the thermogravimetric analyser in accordance with the manufacturer's instructions.

The basic components of an analyser are as follows.

5.1.1 **Thermogravimetric balance**.

5.1.2 **Electrically heated, thermo-regulated oven**.

5.1.3 **Temperature programmer**, for the oven.

5.1.4 **Switching device**, allowing either a stream of nitrogen or, alternatively, a stream of air or oxygen to flow through the oven at a predetermined and constant flow rate.

5.1.5 **X/Y recorder**, for recording the temperature/mass plot. Alternatively, temperature/time and mass/time plots may be recorded simultaneously using a two-pen Y/T recorder. Digital data acquisition stations with related software for drawing the temperature/mass and/or time/mass curves are also suitable; in this case, better accuracy in the calculation of the mass losses can be achieved.

5.1.6 **Auxiliary equipment for producing differential curves** (mandatory for this part of ISO 9924).