BS ISO 9924-3:2024

BSI Standards Publication

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

Part 3: Hydrocarbon rubbers, halogenated rubbers and polysiloxane rubbers

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National foreword

This British Standard is the UK implementation of ISO 9924-3:2024. It supersedes BS ISO 9924-3:2009, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/22, Testing and analysis of rubber.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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Published by BSI Standards Limited 2024

ISBN 978 0 539 20074 4

ICS 83.060

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2024.

Amendments/corrigenda issued since publication

Date	Text affected

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International Standard

ISO 9924-3

Second edition
2024-02

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

Part 3: Hydrocarbon rubbers, halogenated rubbers and polysiloxane rubbers

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

Partie 3: Caoutchoucs hydrocarbonés, caoutchoucs halogénés et caoutchoucs polysiloxanes

Reference number
ISO 9924-3:2024(en)

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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 document 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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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 9924-3:2009), which has been technically revised.

The main changes are as follows:

— extension of the scope by deleting the mandatory preliminary extraction;

— the result of 2006 ITP has been deleted, the result of 2022 ITP has been added in Annex C.

A list of all parts in the ISO 9924 series can be found on the ISO website.

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.

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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 determine the applicability of any other restrictions.

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 thermogravimetric method for the determination of the main constituents of rubber compounds such as elastomer(s), carbon black and mineral filler.

It establishes the "fingerprint" of the tested material. However, the result does not always correspond exactly to the theoretical formula of the rubber.

This method applies to raw or compounded rubbers, vulcanized and unvulcanised, with or without extraction.

This method applies to rubbers with hydrocarbon backbones (NR, BR, SBR, IIR, EPDM, ACM, AEM, etc.) used alone or as mixtures. For the mixtures, the polymer content corresponds to the total rubber and it is not usually possible to identify individual polymers.

This method applies to rubbers with halogenated hydrocarbon backbones (CR, CSM, FKM, CM, CO, ECO, etc.) or containing nitrogen (NBR, HNBR, NBR/PVC, etc.), as well as to their mixtures. However, these rubbers often form carbonaceous residues which interfere with the analysis. Application of an appropriate procedure minimizes these interferences.

This method also applies to rubbers with a polysiloxane backbone (VMQ, etc.) and to rubbers not listed above.

2 Normative references

There are no normative references in this document.

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

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

4 Principle

A weighed test portion is heated following a pre-set programme in a known atmosphere.

Initial pyrolysis in an inert atmosphere (nitrogen) is followed by combustion in an oxidizing atmosphere.

Generally, the reactions that generate mass variations are decompositions, oxidations or reactions volatilizing a constituent.

The loss in mass as a function of temperature indicates a quantitatively usable thermogram which is characteristic of the material.

5 Reagents

5.1 Nitrogen of minimal purity 99,995 % mass fraction, with an oxygen content of less than 10 mg/kg (ppm) and hydrocarbon content less than 1,5 mg/kg (ppm).

5.2 Dry air, with no detectable trace of oil.

The air used may be reconstituted nitrogen and oxygen of purity minimum 99,5 % mass fraction. In some cases, pure oxygen may be used.

6 Apparatus

6.1 Thermogravimetric analyser (TGA).

6.1.1 General. Various types of analyser are commercially available. The basic components of an analyser are listed in 6.1.2 to 6.1.8.

6.1.2 Thermogravimetric balance, comprising a microbalance provided with a pan made from a nonoxidizable material that can weigh up to 50 mg, is readable to the nearest 1 µg and equipped with an oven capable of being maintained at temperatures from room temperature to approximately 1 000 °C.

6.1.3 Appropriate enclosure, allowing the sample to be kept under a specified atmosphere.

6.1.4 Pan or **crucible,** of size suitable to accommodate the sample and small enough to reduce the influence of buoyancy.

6.1.5 Temperature-control system, allowing heating rates to be controlled from 10 °C/min to 50 °C/min.

6.1.6 Gas selector, allowing successive introduction of the inert gas and oxidizing gas while controlling the flow rate.

6.1.7 Measurement equipment, for gas flow rate in the range 10 cm^3/min to 250 cm^3/min.

6.1.8 Data acquisition and processing system.

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