
**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**

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

*Partie 1: Caoutchoucs butadiène, copolymères et terpolymères éthylène-
propylène, isobutène-isoprène, isoprène et butadiène-styrène*



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Contents

Page

Foreword.....	iv
1 Scope	1
2 Normative references	2
3 Principle	2
4 Reagents	2
5 Apparatus	2
6 Thermogravimetric analyser checks	3
7 Procedure	4
8 Expression of results	5
9 Precision	7
10 Test report	8

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 part of ISO 9924 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 9924-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analyses*.

This second edition cancels and replaces the first edition (ISO 9924-1:1993), which has been technically revised.

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*

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

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 and ash in vulcanizates and uncured compounds. The loss in mass at 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 following rubbers occurring alone or as mixtures:

- a) polyisoprene of natural or synthetic origin;
- b) polybutadiene;
- c) styrene-butadiene copolymers;
- d) isobutylene-isoprene copolymers;
- e) ethylene-propylene copolymers and related terpolymers.

NOTE The field of application of the method may be extended to 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 a similar composition. Other compounds are covered in ISO 9924-2.

1.3 The method is not suitable for rubbers containing polymers which form a carbonaceous residue during pyrolysis, such as many chlorine- or nitrogen-containing rubbers.

1.4 The method is also not suitable for materials containing additives which cause the formation of carbonaceous residues during pyrolysis, such as cobalt and lead salts or phenolic resins.

1.5 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 650 °C, unless suitable corrections based on prior knowledge of filler behaviour can be made.

1.6 The method is not suitable for the determination of the total polymer content of compounds or vulcanizates 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 normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 9924. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 9924 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1407:1992, *Rubber — Determination of solvent extract*.

ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards*.

3 Principle

3.1 A weighed test portion is heated, following a pre-set programme, from 70 °C to 300 °C in a stream of nitrogen and is maintained at 300 °C for 10 min. The loss in mass indicates approximately the total content of non-rubber organic matter volatile at 300 °C. In general, this value is not equivalent to the value of the solvent extract.

3.2 The oven temperature is then raised to 550 °C, still in a stream of nitrogen, and maintained at 550 °C for 15 min. The organic matter which was undistilled at 300 °C and the polymer distill off, and the loss in mass between 70 °C and 550 °C represents the total organic-matter content.

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 oven temperature is raised from 550 °C to 650 °C in a stream of nitrogen, then the gas is changed from nitrogen to air or oxygen, or a mixture of air or oxygen, and the temperature is maintained at 650 °C for 15 min or until no further loss in mass is observed. The carbon black is burnt off, and the loss in mass in the oxidizing atmosphere at 650 °C thus represents the carbon black content. A balanced flow of gas is maintained throughout the changeover to avoid buoyancy effects.

3.4 The mass of the residue at 650 °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.