
**Rubber, raw natural and raw
synthetic — Sampling and further
preparative procedures**

*Caoutchouc, naturel brut et synthétique brut — Méthodes
d'échantillonnage et de préparation ultérieure*

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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).

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For an explanation on the voluntary nature of standards, 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

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 fifth edition cancels and replaces the fourth edition (ISO 1795:2007), which has been technically revised. The main changes compared to the previous edition are as follows:

- the normative references in [Clause 2](#) have been updated;
- in [Clause 4](#), it has been clarified that the number of bales to be chosen at random represent the lot;
- in [Clause 5](#), the note stating that the surface layer may be removed if it is contaminated with talc or a release agent, has been deleted;
- the title of [7.2.1](#) has been changed from “milling” to “homogenizing”;
- in [7.3.1](#), a sentence has been added to specify to take directly the test samples from the laboratory sample when using thermogravimetric methods in ISO 248-2 for the determination of volatile-matter content.

Rubber, raw natural and raw synthetic — Sampling and further preparative procedures

WARNING — 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 ensure compliance with any national regulatory conditions.

1 Scope

This document specifies a method for the sampling of raw rubber in bales, blocks or packages and further procedures carried out on the samples to prepare test samples for chemical and physical tests.

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 248-1, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

ISO 248-2, *Rubber, raw — Determination of volatile-matter content — Part 2: Thermogravimetric methods using an automatic analyser with an infrared drying unit*

ISO 289-1, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*

ISO 1658, *Natural rubber (NR) — Evaluation procedure*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*

ISO 2930, *Rubber, raw natural — Determination of plasticity retention index (PRI)*

ISO 3951-2, *Sampling procedures for inspection by variables — Part 2: General specification for single sampling plans indexed by acceptance quality limit (AQL) for lot-by-lot inspection of independent quality characteristics*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at <http://www.electropedia.org/>

— ISO Online browsing platform: available at <http://www.iso.org/obp>

NOTE All references to “bales” include blocks and packages of rubber in chip, powder or sheet form.

3.1

lot

assembly of bales of rubber bearing the same grade and lot marks

3.2

sample

group of bales selected to represent the lot

3.3

laboratory sample

rubber taken from a bale of the sample to represent the bale

3.4

combined laboratory sample

quantity of rubber which represents the sample, prepared by blending together equal parts of each of the laboratory samples to give a homogeneous sample

3.5

test sample

rubber taken from the laboratory sample or the combined laboratory sample for testing, including the preparation of test pieces

3.6

test piece

rubber taken from a test sample in order to carry out a specific test

Note 1 to entry: Some standards use the synonymous term "test specimen".

4 Method of selecting the sample

The greater the number of bales in the sample, the more representative is the sample of the lot, but in most cases practical considerations impose a limit on what is possible. The number of bales to be chosen at random, to represent the lot, shall be agreed between customer and supplier. If applicable, a statistical sampling plan chosen from ISO 3951-2 shall be used.

5 Method of taking the laboratory sample

The preferred method of taking a laboratory sample from each of the selected bales is the following.

Remove the outer wrapping sheets, polyethylene wrapping, bale coating or other surface material from the bale and make two cuts, without the use of lubricant, through the entire bale, normal to the bale faces of largest surface area, so that a cross-sectional slice is removed from the middle of the bale. For referee purposes, this preferred method shall be used.

Alternatively, a laboratory sample may be taken from any convenient part of the bale.

In each case, the total mass of the laboratory sample shall be between 350 g and 1 500 g, depending on the tests to be carried out. If the rubber is in chip or powder form, a similar quantity shall be taken at random from the package.

Unless the laboratory sample is used immediately, it shall be placed in a light-proof and moisture-proof container or package of not more than twice its volume until it is required.

6 Testing

6.1 Each laboratory sample shall be tested and reported upon separately.

6.2 For quality-control purposes, a combined laboratory sample (see [3.4](#)) may be used for the determination of chemical properties and vulcanization characteristics.

7 Preparation of test samples

7.1 General

A roll mill having characteristics as described in ISO 2393 shall be used for all milling operations.

If possible, the laboratory temperature and humidity shall be in accordance with ISO 23529.

7.2 Natural rubber

7.2.1 Homogenizing

Prepare a test sample as follows. Weigh $250 \text{ g} \pm 5 \text{ g}$ of the laboratory sample to the nearest 0,1 g and then homogenize it by passing it 6 times between the surfaces of the mill rolls with the nip set at $1,69 \text{ mm} \pm 0,17 \text{ mm}$ and with the surface temperature of the rolls maintained at room temperature. The water passing through the rolls shall not be heated. In passes 2 to 5 inclusive, roll up the rubber after passing it through the nip and present the roll endwise to the nip for the next pass. Return to the rubber any solid matter separating from it. On the sixth pass, sheet the rubber, allow it to cool in a desiccator, and weigh it again to the nearest 0,1 g.

The initial and final masses are used in the calculation of the volatile matter since some of the volatile constituents are lost during homogenization (see the oven method of ISO 248-1). If the volatile matter cannot be determined immediately, store the homogenized rubber in an airtight container of not more than twice its volume, or wrap it tightly in two layers of aluminium foil until required for testing.

NOTE A laboratory sample for homogenizing which is larger than 250 g is acceptable depending on the tests to be carried.

7.2.2 Chemical and physical tests

Cut test pieces from the homogenized test sample (see 7.2.1) and allocate them to such of the specific tests as may be required. These tests shall be performed in accordance with the appropriate International Standards. The determination of volatile-matter content shall be carried out by the oven method specified in ISO 248-1.

7.2.3 Mooney viscosity

Prepare two test pieces from the homogenized test sample (see 7.2.1) in the manner specified in ISO 289-1 and measure the Mooney viscosity in accordance with ISO 289-1.

7.2.4 Plasticity retention index (PRI)

Take a test piece of $20 \text{ g} \pm 2 \text{ g}$ from the homogenized test sample (see 7.2.1) and prepare in accordance with the procedure given in ISO 2930. Determine the plasticity retention index (PRI) in accordance with ISO 2930.

7.2.5 Vulcanization characteristics

Take a test piece from a homogenized test sample (see 7.2.1) and determine the vulcanization characteristics in accordance with ISO 1658.

7.3 Synthetic rubbers

7.3.1 Chemical and physical tests

Cut a test sample of $250 \text{ g} \pm 5 \text{ g}$ (or, if the product is in chip or powder form, take a similar sample at random) from the laboratory sample and use for the determination of volatile-matter content in

accordance with the hot-mill method of ISO 248-1, where specified. Take portions from the material subjected to the determination of volatile-matter content sufficient to carry out the other chemical tests that may be required.

Certain rubbers tend to stick to the rolls during the hot-mill method. If sticking occurs, the oven method of ISO 248-1 shall be used. Even if the oven method is used for the determination of volatile-matter content, the rubber shall still be dried by the hot-mill method prior to carrying out chemical tests. If this is not possible, then the test samples shall be taken directly from the laboratory sample. When thermogravimetric methods in ISO 248-2 are used for the determination of volatile-matter content, the test samples shall be taken directly from the laboratory sample.

If the procedure given in 6.2 is followed, the combined laboratory sample may be prepared by blending together material remaining from each determination of volatile-matter content so that a combined laboratory sample of $250 \text{ g} \pm 5 \text{ g}$ is formed. Blend the individual pieces together using the procedure described in 7.3.2.2.

7.3.2 Mooney viscosity

7.3.2.1 Preparation without milling

This is the preferred procedure.

Cut a test sample of appropriate thickness from the laboratory sample. Prepare two test pieces in the manner specified in ISO 289-1 and measure the Mooney viscosity in accordance with ISO 289-1. The test pieces shall be as free as possible from air and pockets that may trap air against the rotor and die surface. Rubber in chip or pellet form shall be evenly distributed above and below the rotor.

7.3.2.2 Preparation with milling

In some cases, it may be necessary to mass the rubber on a mill prior to testing (for a particular rubber type, the appropriate evaluation procedure specifies whether milling is necessary). Milling shall be carried out in accordance with the following procedure.

Take a test sample of rubber of about $250 \text{ g} \pm 5 \text{ g}$ from the laboratory sample. Pass the test sample 10 times between the surfaces of the mill rolls with the nip set at $1,4 \text{ mm} \pm 0,1 \text{ mm}$ and with the mill roll surface temperature maintained at $50 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$ (see, however, the special procedures for butadiene rubber, ethylene-propylene-diene rubber and some types of butadiene-acrylonitrile rubber given below). In passes 2 to 9 inclusive, double the rubber upon itself. On the tenth pass, sheet the rubber without doubling. Prepare two test pieces in the manner specified in ISO 289-1 and determine the Mooney viscosity in accordance with ISO 289-1.

For butadiene rubber (BR), and for ethylene-propylene-diene rubber (EPDM) of low Mooney viscosity (<35), the mill roll surface temperature shall be $35 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$.

For some types of butadiene-acrylonitrile rubber (NBR), it is necessary to set the nip at $1,0 \text{ mm} \pm 0,1 \text{ mm}$ and the mill roll surface temperature at $50 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$.

By agreement between the interested parties, other conditions (e.g. nip width or temperature) may be used for massing. These conditions shall be reported.

NOTE Cases in which preparation with milling can be necessary:

- rubber showing a high degree of porosity or inhomogeneity;
- rubber of very high viscosity;
- in-process rubber crumb;
- carbon black masterbatches.