INTERNATIONAL STANDARD

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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.

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

International Standard ISO 1795 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 third edition cancels and replaces the second edition (ISO 1795:1992), which has been technically revised.

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

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 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 normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard 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 248:1991, *Rubbers, raw — Determination of volatile-matter content.*

ISO 289-1:1994, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*.

ISO 1629:1995, *Rubber and latices — Nomenclature*.

ISO 1658:1994, *Natural rubber (NR) — Evaluation procedure.*

ISO 2393:1994, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 2930:1995, *Rubber, raw natural — Determination of plasticity retention index (PRI).*

ISO 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

ISO 3951:1989, *Sampling procedures and charts for inspection by variables for percent nonconforming*.

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply (all references to “bales” include blocks and packages of rubber in chip, powder or sheet form).

3.1
lot
an assembly of bales of rubber bearing the same grade and lot marks

3.2
sample
a group of bales selected to represent the lot

3.3
laboratory sample
the rubber taken from a bale of the sample to represent the bale

3.4
combined laboratory sample
a quantity of rubber which will represent the sample, prepared by blending together equal parts of each of the laboratory samples to give a homogeneous sample

3.5
test sample
the rubber taken from the laboratory sample or the combined laboratory sample for testing, including the preparation of test pieces

3.6
test piece
the rubber taken from a test sample in order to carry out a specific test

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 shall be agreed between customer and supplier. If applicable, a statistical sampling plan chosen from ISO 3951 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 to be 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.

NOTE The surface layer may be removed if it is contaminated with talc or a release agent.

6 Sampling report

The sampling report shall include at least the following information:

a) all details necessary for full identification of the sample, e.g. lot identification;

b) the type and grade of rubber;

c) the number of bales or packages forming the lot, and the kind of bale or package;

d) the number of bales or packages forming the sample;

e) any deviation from this International Standard;

f) the date of sampling.

7 Testing

Each laboratory sample shall be tested and reported upon separately.

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

8 Preparation of test portions

8.1 General

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

8.2 Natural rubber

8.2.1 Milling

Weigh 250 g ± 5 g of the laboratory sample to the nearest 0,1 g and then homogenize it by passing it 10 times between the surfaces of the mill rolls with the nip set at 1,3 mm ± 0,15 mm and with the surface temperature of the rolls maintained at 70 °C ± 5 °C. In passes 2 to 9 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 tenth 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). 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.

8.2.2 Chemical and physical tests

Cut test samples from the homogenized laboratory sample (see 8.2.1) 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.

8.2.3 Mooney viscosity

Take two 30 g to 40 g portions of the homogenized laboratory sample (see 8.2.1) and measure the Mooney viscosity in accordance with ISO 289-1.

8.2.4 Plasticity retention index (PRI)

Take a test sample of 20 g ± 2 g from the homogenized laboratory sample (see 8.2.1) and prepare in accordance with the procedure given in ISO 2930. Determine the plasticity retention index (PRI) in accordance with ISO 2930.

8.2.5 Vulcanization characteristics

Determine the characteristics on a portion of the homogenized laboratory sample (see 8.2.1), in accordance with ISO 1658 and ISO 3417.

8.3 Synthetic rubbers

8.3.1 Chemical and physical tests

Cut a test sample of 250 g ± 5 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, 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 shall be used. Even if the oven method is used for 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.

If the procedure given in the second paragraph in clause 7 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 g ± 5 g is formed. Blend the individual pieces together using the procedure described in 8.3.2.2.

8.3.2 Mooney viscosity

8.3.2.1 Preparation without milling (preferred procedure)

Cut a test sample of appropriate thickness from the laboratory sample and determine the Mooney viscosity in accordance with ISO 289. The test sample 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.

8.3.2.2 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 will specify whether milling is necessary). Milling shall be carried out in accordance with the following procedure:

Take a test sample of rubber of about 250 g ± 5 g from the laboratory sample for determination of Mooney viscosity. Pass the test sample 10 times between the surfaces of the mill rolls with the nip set at 1,4 mm ± 0,1 mm and with the mill roll surface temperature maintained at 50 °C ± 5 °C (see, however, the special procedures for butadiene rubber, ethylene-propylene-diene rubber, chloroprene 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 and determine the Mooney viscosity in accordance with ISO 289.

For butadiene rubber (BR), and for ethylene-propylene-diene rubber (EPDM) of low Mooney viscosity (< 35), the mill roll surface temperature shall be 35 °C ± 5 °C.

For chloroprene rubber (CR), the mill roll surface temperature shall be 20 °C ± 5 °C. Set the nip at 0,4 mm ± 0,05 mm and make only two passes. (If condensation of moisture is experienced on the mill, the lowest suitable temperature shall be used. This temperature shall be reported.)

For some types of butadiene-acrylonitrile rubber (NBR), it will be necessary to set the nip at 1,0 mm ± 0,1 mm and the mill roll surface temperature at 50 °C ± 5 °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 1 Cases in which preparation with milling may be necessary:

— rubber showing a high degree of porosity or inhomogeneity;

— rubber of very high viscosity;

— in-process rubber crumb;

— carbon black masterbatches.

NOTE 2 When rubber is prepared with milling, the value of the Mooney viscosity obtained may not be the same as when the preferred procedure is used, and the results have been shown to be less reproducible.

8.3.3 Vulcanization characteristics

Cut a test sample (or physically select, if the rubber is in chip or powder form) from the laboratory sample and determine the vulcanization characteristics in accordance with the evaluation procedure applicable to the rubber to be tested.

If the procedure given in the second paragraph in clause 7 is followed, take sufficient material from each laboratory sample to form a combined laboratory sample of the correct size. Carry out the blending operation in the initial part of the mixing procedure.