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

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*Caoutchouc brut, naturel et synthétique — Méthodes d'échantillonnage
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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.

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.

International Standard ISO 1795 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Sub-Committee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition of ISO 1795 (ISO 1795:1974), as well as the third edition of ISO 1796 (ISO 1796:1982) and the first edition of ISO/TR 2630 (ISO/TR 2630:1978), of which it constitutes a technical revision.

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

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 those samples to prepare test portions for chemical and physical tests.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 248:1991, *Rubbers, raw — Determination of volatile-matter content*.

ISO 289:1985, *Rubber, unvulcanized — Determination of Mooney viscosity*.

ISO 1658:1973, *Natural rubber (NR) — Test recipes and evaluation of vulcanization characteristics*.

ISO 2393:1973, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*.

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 Definitions

For the purposes of this International Standard, the following definitions apply. All references to bales in this standard 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 the laboratory samples.

3.5 test portion: 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 portion 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 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

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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 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 600 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 moisture-proof container or package of not more than twice its volume until it is required.

NOTE 1 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 standard.

7 Testing

Each laboratory sample shall be tested and reported upon separately.

NOTE 2 For quality-control purposes, a combined laboratory sample may be used for the determination of chemical properties and vulcanization characteristics.

8 Preparation of test portions

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

8.1 Natural rubber

Weigh 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 $1,3 \text{ mm} \pm 0,15 \text{ mm}$ and with the rolls maintained at $70 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$. 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.

NOTE 3 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 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.

8.1.1 Chemical and physical tests

Cut test portions from the homogenized laboratory sample 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.

8.1.2 Mooney viscosity

Take a 30 g to 40 g portion of the homogenized rubber and measure the Mooney viscosity in accordance with ISO 289.

8.1.3 Vulcanization characteristics

Determine the characteristics on a portion of the homogenized rubber in accordance with ISO 1658 and ISO 3417.

8.2 Synthetic rubbers

8.2.1 Chemical and physical tests

Cut a test portion 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, 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 portions shall be taken directly from the laboratory sample.

NOTE 4 If the procedure given in note 2 (see clause 7) is to be followed, a 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 8.2.2.2.

8.2.2 Mooney viscosity

8.2.2.1 Preparation without milling (preferred procedure)

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

8.2.2.2 Preparation with milling

In some cases it may be necessary to mass the rubber on a mill prior to testing (see note 5 below) (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 portion of rubber of about $250 \text{ g} \pm 5 \text{ g}$ from the laboratory sample for determination of Mooney viscosity. Pass this test portion 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, 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 ethylene-propylene-diene rubber (EPDM), the mill roll surface temperature shall be $35 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$.

For chloroprene rubber (CR), the mill roll surface temperature shall be $20 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$. Set the nip at $0,4 \text{ mm} \pm 0,05 \text{ mm}$ and make only two passes.

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

NOTES

5 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 masterbatch.

6 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.

8.2.3 Vulcanization characteristics

Cut a test portion (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 note 2 (see clause 7) is to be 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.

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