
International Standard



1796

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Rubber, raw — Sample preparation

Caoutchoucs bruts — Préparation des échantillons

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Descriptors : rubber, crude rubber, synthetic rubber, tests, test specimen conditioning.

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1796 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the second edition (i.e. ISO 1796-1980), which had been approved by the member bodies of the following countries :

Australia	Germany, F. R.	South Africa, Rep. of
Austria	Hungary	Spain
Belgium	India	Sri Lanka
Brazil	Italy	Sweden
Bulgaria	Korea, Rep. of	Thailand
Canada	Mexico	Turkey
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	Poland	USA
France	Romania	USSR

No member body had expressed disapproval of the document.

Rubber, raw — Sample preparation

1 Scope and field of application

This International Standard specifies a method of preparing raw rubbers for test from samples taken in accordance with ISO 1795.

A diagram showing the sequence of operations in sampling and further preparation is included in an annex.

ISO 1655, *Raw rubber and rubber latex — Determination of manganese content — Potassium periodate photometric method.*

ISO 1656, *Raw natural rubber and natural rubber latex — Determination of nitrogen.*

ISO 2007, *Rubber, unvulcanized — Determination of plasticity — Rapid plastimeter method.*

2 References

2.1 General

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

ISO/R 289, *Determination of viscosity of natural and synthetic rubbers by the shearing disk viscometer.*

ISO 1795, *Raw rubber in bales — Sampling.*

ISO 2007, *Rubber, unvulcanized — Determination of plasticity — Rapid plastimeter method.*

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

2.2 Natural rubber

2.2.1 Chemical and physical tests

ISO 247, *Rubber — Determination of ash.*

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

ISO 249, *Raw natural rubber — Determination of dirt.*

ISO/R 1654, *Raw rubber and rubber latex — Determination of copper.*

ISO 2930, *Rubber, raw natural — Determination of plasticity — Rapid plastimeter method.*

ISO 4660, *Rubber, raw natural — Colour index test.*

2.2.2 Vulcanization characteristics

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

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

2.3 Synthetic rubbers

2.3.1 Chemical tests

ISO 247, *Rubber — Determination of ash.*

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

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

ISO 2002, *Raw styrene-butadiene rubber (SBR) — Determination of organic acid content.*

ISO 2003, *Raw styrene-butadiene rubber (SBR) — Determination of soap content.*

ISO 2453, *Styrene-butadiene copolymers — Determination of bound styrene content.*

2.3.2 Vulcanization characteristics

ISO 2302, *Rubber, isobutene-isoprene (IIR) — Evaluation procedures.*

ISO 2303, *Rubber, isoprene (IR) — Non oil-extended, solution-polymerized types — Test recipe and evaluation of vulcanization characteristics.*

ISO 2322, *Rubber, styrene-butadiene (SBR) — Emulsion-polymerized general purpose types — Test recipe and evaluation of vulcanization characteristics.*

ISO 2475, *Rubber, chloroprene (CR) — General purpose types — Evaluation procedures.*

ISO 2476, *Rubber, butadiene (BR) — Solution-polymerized types — Test recipe and evaluation of vulcanization characteristics.*

ISO 4097, *Rubber, ethylene-propylene-diene (EPDM) — Non-oil extended raw general purpose types — Evaluation procedures.*

ISO 4658, *Rubber, acrylonitrile-butadiene (NBR) — Test recipe and evaluation of vulcanization characteristics.*

ISO 4659, *Rubber, raw styrene-butadiene (carbon black or carbon black and oil masterbatches) — Test recipe and method of evaluation.*

3 Definitions

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 piece : The rubber taken from a bale (or package) of the sample.

3.4 test portion : The rubber taken from a piece for testing including the preparation of the test pieces.

4 Apparatus

A roll mill having characteristics as described in ISO 2393 shall be used for homogenization of the piece or portion of the piece.

5 Natural rubber

5.1 Method of preparation of test portions

Weigh the piece to the nearest 0,1 g and then homogenize it by passing ten times between the surfaces of the mill rolls with the nip set at $1,3 \pm 0,15$ mm and with the rolls maintained at

70 ± 5 °C. In passes 2 to 9 inclusive, roll the rubber after passing 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 and weigh it again to the nearest 0,1 g.

NOTE — The initial and final masses are used in the calculation of volatile matter since some of the volatiles 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 the volume of the piece or wrap it tightly in two layers of aluminium foil until required for test.

5.2 Chemical and physical tests

Cut test portions from the homogenized piece and allocate them to such of the specific tests as may be required from those indicated in the figure under "Natural rubber". The tests shall be performed in accordance with the International Standards listed in 2.2.1. The determination of volatile matter content shall be carried out by the oven method specified in ISO 248 at a temperature of 100 ± 5 °C.

5.3 Mooney viscosity

Take a 250 g portion of the homogenized rubber for testing in accordance with ISO/R 289.

5.4 Vulcanization characteristics

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

6 Synthetic rubbers

6.1 Chemical tests

Cut a piece of at least 250 g (or, if the product is in chip or powder form, a similar sample taken at random) from the bale or package 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 remaining from the determination of volatile matter content in such amounts as are required for such other chemical tests as may be required; perform the tests in accordance with the International Standards listed in 2.3.1. Certain rubbers tend to stick to the rolls during the hot mill method; if so, the oven method at 100 ± 5 °C may be used. If the oven method is used for determination of volatile matter content, the rubber shall be dried by the hot mill method prior to carrying out chemical tests.

6.2 Mooney viscosity

6.2.1 Synthetic rubbers (except butyl-IIR)

Take a test portion of rubber of about 250 ± 5 g from the bale or package for determination of Mooney viscosity. Pass this test portion ten times between the surfaces of the mill rolls with the nip set at $1,4 \pm 0,1$ mm and with the mill roll surface temperature maintained at 50 ± 5 °C. In passes 2 to 9 inclusive, double the rubber upon itself. On the tenth pass,

sheet the rubber without doubling for testing in accordance with ISO/R 289.

NOTES

- 1 For butadiene rubber (BR) and ethylene-propylene-diene rubber (EPDM), the mill roll surface temperature should be 35 ± 5 °C.
- 2 For chloroprene rubber (CR), the mill roll surface temperature should be 20 ± 5 °C. Set the nip at $0,4 \pm 0,05$ mm and make only two passes.
- 3 For some types of butadiene acrylonitrile rubber (NBR), the nip should be set at $1,0 \pm 0,1$ mm and the mill roll surface temperature should be 50 ± 5 °C.
- 4 Crumb samples tested during production should be massed according to the procedure specified in 6.2.1.

6.2.2 Butyl rubber (IIR)

Select a piece direct from the sample bale, avoiding areas which contain many bubbles. Cut a circular test portion from this piece approximately 6 mm thick and 50 mm in diameter for testing in accordance with ISO/R 289¹⁾.

6.3 Vulcanization characteristics

Cut a sufficient portion (or physically select, if the rubber is in chip or powder form) from the bale or package to determine the vulcanization characteristics in accordance with the International Standards applicable for the rubber to be tested as selected from the listing in 2.3.2.

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1) ISO/R 289 is not in line with current practice (8 mm thick, 45 mm diameter).

Annex

Sequence of operations in sampling and further preparation

A detailed sequence of operations in the sampling and further preparation of raw rubber is given in the figure. Attention is drawn to the two routes available for determining the volatile matter and chemical properties of synthetic rubbers. The route chosen will depend on the suitability of the material for processing on a hot mill (see 6.1).

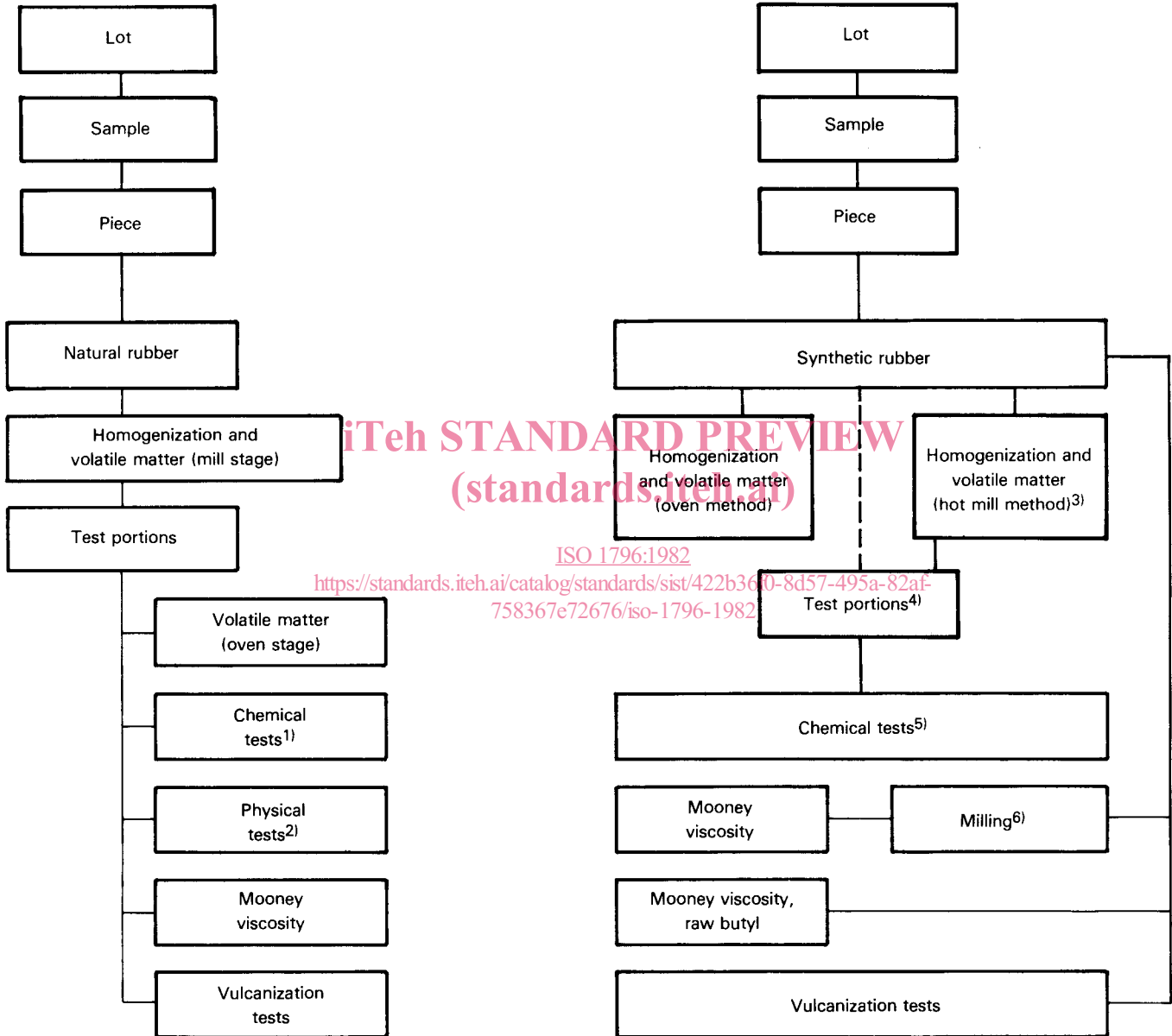


Figure — Schematic breakdown of operations

- 1) Dirt, nitrogen, copper, manganese, ash, solvent extract.
- 2) Rapid plasticity, plasticity retention index, colour.
- 3) Preferred method.
- 4) The test portion can be taken directly from the piece, if the rubber is difficult to hot mill.
- 5) Ash, solvent extract, soap content, organic acid, bound styrene.
- 6) Omit for IIR (butyl).

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