INTERNATIONAL STANDARD

ISO 1658

Fourth edition
2015-08-01

Natural rubber (NR) — Evaluation procedure

Caoutchouc naturel (NR) — Méthode d'évaluation

Reference number
ISO 1658:2015(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This fourth edition cancels and replaces the third edition (ISO 1658:2009), of which it constitutes a minor revision to update the normative references (in Clause 2 and throughout the text).

Natural rubber (NR) — Evaluation procedure

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

— physical and chemical tests on raw natural rubbers;

— standard materials, standard test formulae, equipment and processing methods for evaluating the vulcanization characteristics of natural rubber (NR).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 37, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

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

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

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

ISO 2000:2014, *Rubber, raw natural — Guidelines for the specification of technically specified rubber (TSR)*

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

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

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

ISO 6502, *Rubber — Guide to the use of curemeters*

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

3 Sampling and further preparative procedures

3.1 A laboratory sample of mass approximately 1,5 kg shall be prepared by the method described in ISO 1795.

3.2 The rubber shall be homogenized in accordance with ISO 1795.

3.3 Preparation of the test samples shall be in accordance with ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1 on a test sample prepared as indicated in 3.3. Record the result as ML(1+4) at 100 °C.

4.2 Volatile-matter content

Determine the volatile-matter content by the oven method specified in ISO 248-1 on a test sample prepared as indicated in 3.3.

4.3 Other specifications on requirements

Any specific values for physical and chemical properties other than the above (4.1 and 4.2) shall be based upon the grades given in Table 2 of ISO 2000:2014.

5 Preparation of test mix

5.1 General

The following standard formulae and mixing procedures are recommended:

a) two gum-stock formulae for comparative testing of the vulcanization characteristics of natural rubber for use in non-black-filled compounds;

b) a black-filled formula for comparative testing of natural rubber for use in black-filled compounds;

c) a mill mixing procedure including masterbatches for the two gum-stock formulae;

d) a mill mixing procedure and a laboratory internal mixer (LIM) mixing procedure for the black-filled formula.

NOTE The black-filled formula (formula 3 in Table 1) can also be used for comparative testing of isoprene rubbers (IRs).

5.2 Standard test formulae

The standard test formulae are given in Table 1.

The materials shall be national or international standard reference materials.

Table 1 — Standard test formulae

Material	**Number of parts by mass**		
	Formula (1) **ACS 1**	**Formula (2)** **TBBS 1**	**Formula (3)** **Black-filled**
Natural rubber	100,00	100,00	100,00
Zinc oxide	6,00	6,00	5,00
Sulfur	3,50	3,50	2,25
Stearic acid	0,50	0,50	2,00
Industry reference black [a]	—	—	35,00
MBT [b]	0,50	—	—
TBBS [c]	—	0,70	0,70
Total	110,50	110,70	144,95

[a] The current IRB shall be used.

[b] 2-Mercaptobenzothiazole.

[c] *N*-tert-butyl-2-benzothiazole-sulfenamide. This shall be in powder form having an initial ether-insoluble or ethanol-insoluble matter content of less than 0,3 % (by mass). The material shall be stored at room temperature in a closed container and the ether-insoluble or ethanol-insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 % (by mass), the material shall be discarded or recrystallized.

5.3 Procedure

5.3.1 Equipment and procedure

Equipment and procedure for the preparation, mixing and vulcanizing shall be in accordance with ISO 2393.

5.3.2 Mill mixing procedure for formulae 1 and 2 (gum compounds)

The standard laboratory mill batch mass, in grams, shall be four times the formulation batch mass (i.e. 110,5 g × 4, = 442 g, for formula 1). Maintain the surface temperature of the rolls at 70 °C ± 5 °C and a good rolling bank at the nip of the rolls during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to the mill openings might be necessary.

	Duration (min)
a) Pass the rubber twice between the rolls without banding, with the mill opening set at 0,2 mm.	—
b) Band the rubber with the mill opening set at 1,4 mm. When a smooth band has been obtained, adjust the mill opening to 1,8 mm.	4
c) Add the zinc oxide, the stearic acid, the sulfur and the MBT or TBBS.	4
d) Make three 3/4 cuts from each side.	3
e) Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch endwise through the rolls six times.	2

Total time	13

f) Check the mass of the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than + 0,5 % or − 1,5 %, discard the batch and remix.

g) Cut sufficient material from the batch for curemeter testing and, if required, for determination of the Mooney viscosity of the unvulcanized batch in accordance with ISO 289-1. Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring test pieces.

h) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 23529.

5.3.3 Mill mixing procedure for formulae 1 and 2 (gum compounds) using masterbatches

Compounding materials such as accelerators, sulfur or fillers can be incorporated into the rubber as masterbatches. This technique improves the accuracy of compounding-material incorporation and is also more convenient.

The procedure for preparing masterbatches and test mixes for the gum compounds is given in Annex A.

5.3.4 Mixing procedures for formula 3 (black-filled compound)

5.3.4.1 Mill mixing

5.3.4.1.1 Measurement of rapid plasticity number

Load the rubber on to the mill with the mill opening set at 0,5 mm. Masticate until a smooth band and rolling bank are obtained.

After mastication, determine the rapid plasticity number in accordance with ISO 2007. The rapid plasticity number shall not exceed 45, which is approximately equivalent to a viscosity of 70 Mooney units determined in accordance with ISO 289-1.

5.3.4.1.2 Mixing

The standard laboratory mill batch mass, in grams, shall be four times the formulation batch mass (i.e. 144,95 g × 4, = 579,8 g, for formula 3). Maintain the surface temperature of the rolls at 70 °C ± 5 °C and a good rolling bank at the nip of the rolls during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to the mill openings may be necessary.

	Duration (min)
a) Band the rubber with the mill opening set at 1,4 mm.	1
b) Add the stearic acid. Make one 3/4 cut from each side.	1
c) Add the zinc oxide and the sulfur. Make one 3/4 cut from each side.	2
d) Add the carbon black evenly across the mill at a uniform rate. When about half the black has been incorporated, open the mill to 1,9 mm and make one 3/4 cut from each side. Then add the remainder of the carbon black. When all the black has been incorporated, make one 3/4 cut from each side. Be certain to add the black that has dropped into the mill pan.	10
e) Add the TBBS. Make three 3/4 cuts from each side.	3
f) Cut the batch from the mill. Set the mill opening to 0,8 mm and pass the rolled batch lengthways through the mill six times.	3
Total time	**20**

g) Check the mass of the batch. If the mass of the batch differs from the theoretical value by more than + 0,5 % or − 1,5 %, discard the batch and remix.

h) Cut sufficient material from the batch for curemeter testing and, if required, for determination of the Mooney viscosity of the unvulcanized batch in accordance with ISO 289-1. Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring test pieces.

i) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 23529.

5.3.4.2 Mixing using a laboratory internal mixer (LIM)

5.3.4.2.1 General

For laboratory internal mixers having nominal capacities of 65 cm^3 to about 2 000 cm^3, the batch mass shall be equal to the nominal mixer capacity, in cubic centimetres, multiplied by the density of the compound, in grams per cubic centimetre. This means that, if the laboratory internal mixer has a nominal capacity of 750 cm3, the batch mass for formula 3 is 750 × 1,103 g (= 827,25 g).

For each batch mixed during the preparation of a series of identical mixes, the laboratory internal mixer conditions shall be the same. At the beginning of each series of test mixes, a machine-conditioning batch shall be mixed using the same formulation as the mixes under test. The laboratory internal mixer shall be allowed to cool down to 60 °C between the end of one test batch and the start of the next. The temperature setting shall not be altered during the mixing of a series of test batches.

5.3.4.2.2 Single-stage mixing

The mixing technique shall be such as to obtain a good dispersion of all the ingredients.

The final temperature of the batch discharged after mixing shall not exceed 120 °C. If necessary, adjust the batch mass or the head temperature so that this condition is met.

NOTE 1 The mixing conditions given in Table B.5 for each size of laboratory internal mixer may be helpful.

NOTE 2 Compounding materials other than rubber, carbon black and oil can be added to laboratory internal mixer batches more precisely and with greater ease if they are previously blended together in the proportions required by the formulation. Such blends can be made using a mortar and pestle, by mixing for 10 min in a biconical blender with intensifier bar turning, or by mixing in a blender for five 3 s periods and scraping the inside of the blender to dislodge materials stuck to the sides after each 3 s mix. A Waring blender has been found suitable for this method. Caution: if mixed for longer than 3 s, the stearic acid can melt and prevent good dispersion.

An example of a mixing procedure for a laboratory internal mixer is given in a) to h) hereafter.

	Duration (min)	**Cumulative time** (min)
a) Load the rubber, lower the ram and allow the rubber to be masticated.	1,0	1,0
b) Raise the ram and add the pre-blended zinc oxide, sulfur, stearic acid and TBBS, taking care to avoid any loss. Then add the carbon black, sweep the orifice and lower the ram.	1,0	2,0
c) Allow the batch to mix.	7,0	9,0

d) Turn off the motor, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature.

e) After discharging the mixed batch, pass it through a mill set at 70 °C ± 5 °C once at a 0,5 mm mill opening and then twice at a 3,0 mm mill opening.

f) Determine the batch mass and record. If it differs from the theoretical value by more than + 0,5 % or − 1,5 %, discard the batch and remix.

g) Cut a test piece for determining the vulcanization characteristics in accordance with ISO 6502 or ISO 3417, if required. Condition the test piece for 2 h to 24 h, if possible at standard temperature and humidity as defined in ISO 23529, before testing.

h) If required, sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37. To obtain the effects of mill direction, pass the folded batch four times between mill rolls set at 70 °C ± 5 °C and the appropriate mill opening. Cool on a flat, dry surface.

i) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 23529.

5.3.4.2.3 Two-stage mixing including mill for final mixing

5.3.4.2.3.1 General

The laboratory internal mixer shall be allowed to cool down to 60 °C between the end of one test batch and the start of the next.

5.3.4.2.3.2 Stage 1 — Initial mixing stage

The mixing technique shall be such as to obtain a good dispersion of all the ingredients.

The final temperature of the batch discharged after mixing shall be between 150 °C and 170 °C. If necessary, adjust the batch mass or head temperature so that this condition is met.

An example of a mixing procedure for the initial mixing is given in a) to k) hereafter.

	Duration (min)	**Cumulative time (min)**
a) Adjust the temperature of the laboratory internal mixer to a starting temperature of 50 °C ± 3 °C. Close the discharge door, set the rotor and raise the ram.	—	—
b) Load the rubber, lower the ram and allow the rubber to be masticated.	0,5	0,5
c) Raise the ram and load the zinc oxide, stearic acid and carbon black. Lower the ram.	0,5	1,0
d) Allow the batch to mix.	2,0	3,0
e) Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0,5	3,5
f) Allow the batch to mix.	1,5	5,0
g) Discharge the batch.		

h) After discharging the batch, immediately check the temperature of the batch with a suitable measuring device. If the temperature as measured falls outside the range 150 °C to 170 °C, discard the batch.

i) Pass the batch three times through a mill with a mill opening of 2,5 mm and at a roll temperature of 70 °C ± 5 °C.

j) Sheet the batch to an approximate thickness of 10 mm and determine the mass of the batch. If the mass differs from the theoretical value by more than + 0,5 % or − 1,5 %, discard the batch and remix.

k) Leave the batch for at least 30 min and up to 24 h, if possible at standard temperature and humidity as defined in ISO 23529.

The smaller laboratory internal mixers do not provide enough compound for the final mill mixing as a batch mass of three times the formula mass is required. In these cases, the laboratory internal mixer may be used for the final mixing. The head temperature or the batch mass may be adjusted so that the final temperature of the discharged batch does not exceed 120 °C.

5.3.4.2.3.3 Stage 2 — Final mixing stage

The mixing technique shall be such as to obtain a good dispersion of all the ingredients. The final temperature of the batch discharged after mixing shall not exceed 120 °C.

When a laboratory internal mixer (LIM) is used, adjust, if necessary, the batch mass or the head temperature so that this condition is met.

When mill mixing is used, set the surface temperature of the rolls at 70 °C ± 5 °C and maintain it at this value during mixing. The standard laboratory mill batch mass, in grams, shall be based on three times the formula mass unless otherwise specified in the appropriate rubber evaluation procedure.

An example of an **LIM mixing procedure for the final mixing** is given in a) to h) hereafter.

	Duration (min)	**Cumulative time** (min)
a) Close the discharge door, set the rotor and raise the ram.	—	—
b) Load the rubber, the sulfur and the accelerator. Lower the ram and allow the rubber to be masticated.	0,5	0,5
c) Allow the batch to mix.	1,5	2,0

d) Raise the ram, open the mixing chamber and discharge the batch. Record the maximum batch temperature.

e) After discharging the mixed batch, pass it four times through the mill at a roll temperature of 70 °C ± 5 °C and with a mill opening of 0,8 mm.

f) Determine the batch mass and record. If it differs from the theoretical value by more than + 0,5 % or − 1,5 %, discard the batch and remix.

g) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37.

h) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 23529.

An example of a **mill mixing procedure for the final mixing** is given in a) to g) as follows.

	Duration (min)	**Cumulative time** (min)
a) Set the mill opening to 1,5 mm.	—	—

b) Band the rubber.	1,0	1,0
c) Add the sulfur and the accelerator. Do not cut the band until the sulfur and accelerator are completely dispersed.	1,5	2,5
d) Make three 3/4 cuts from each side, allowing 15 s between each cut.	2,5	5,0
e) Cut the batch from the mill. Set the mill opening at 0,8 mm and pass the rolled batch endwise through the rolls six times, introducing it from each end alternately.	2,0	7,0

f) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37. Determine the batch mass and record. If it differs from the theoretical value by more than + 0,5 % or − 1,5 %, discard the batch and remix.

g) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 23529.

6 Evaluation of vulcanization characteristics by a curemeter test

6.1 Using an oscillating-disc curemeter

Measure the following standard test parameters:

M_L, M_H at defined time, t_{s1}, $t'_c(50)$, $t'_c(90)$

in accordance with ISO 3417, using the following test conditions:

- oscillation frequency: 1,7 Hz (100 cycles per minute);
- amplitude of oscillation: 1° of arc (3° of arc optional);
- selectivity: to be chosen to give at least 75 % of full-scale deflection at M_H (note that, with some rubbers, 75 % may not be attainable);
- die temperature: 160 °C ± 0,3 °C;
- pre-heat time: none.

6.2 Using a rotorless curemeter

Measure the following standard test parameters:

F_L, F_H at defined time, t_{s1}, $t'_c(50)$, $t'_c(90)$

in accordance with ISO 6502, using the following test conditions:

- oscillation frequency: 1,7 Hz (100 cycles per minute);
- amplitude of oscillation: 0,5° of arc (1° of arc optional);
- selectivity: to be chosen to give at least 75 % of full-scale deflection at F_H (note that, with some rubbers, 75 % may not be attainable);
- die temperature: 160 °C ± 0,3 °C;
- pre-heat time: none.

7 Evaluation of tensile stress-strain properties of vulcanized test mixes

Vulcanize sheets at 140 °C for periods of 20 min, 30 min, 40 min and 60 min. Condition the vulcanized sheets for 16 h to 96 h, if possible at standard temperature and humidity as defined in ISO 23529. Measure the stress-strain properties in accordance with ISO 37.

8 Test report

The test report shall include the following:

a) a reference to this International Standard;

b) all details necessary for the identification of the sample;

c) the standard test formula and the mixing procedure used;

d) the reference materials used;

e) the curemeter test used in Clause 6 (ISO 3417 or ISO 6502);

f) any unusual features noted during the determination;

g) any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional, e.g. whether masterbatches were used;

h) the results and the units in which they have been expressed;

i) the date of the test.

Annex A
(normative)

Procedure for preparing gum compounds through use of masterbatches

A.1 Masterbatch formulation

The rubber for preparing masterbatches shall be of similar quality to that being tested. The proportions by mass of the compounding materials used are given in Table A.1.

Table A.1 — Masterbatch formulation

Material	Number of parts by mass		
	MBT	Sulfur	TBBS
Natural rubber	100	100	100
Zinc oxide	120	120	120
Stearic acid	10	10	10
MBT	20	—	—
TBBS	—	—	28
Sulfur	—	140	—
Total	250	370	258

A.2 Test mix formulation

The test mixes are made up as given in Table A.2.

Table A.2 — Test mix formulation

Material	Number of parts by mass	
	ACS 1	TBBS
Test rubber	95,00	95,00
MBT masterbatches	6,25	—
TBBS masterbatches	—	6,45
Sulfur masterbatches	9,25	9,25
Total	110,50	110,70

A.3 Masterbatch preparation by mill mixing procedure

The mill rolls are kept cool with running water throughout the mixing.

	Duration (min)
a) Band the rubber with the mill opening set at 0,6 mm.	1
b) Add the zinc oxide, stearic acid and MBT or TBBS or sulfur. Gradually widen the mill opening so as to maintain a rolling bank. When about 80 % of the material has been incorporated (mill opening approximately 1,0 mm), make one 3/4 cut from each side.	2
c) Incorporate the rest of the material and, when no free powder is evident, make 3/4 cuts from each side until dispersion appears fully uniform.	5

Total time	8

d) Cut the masterbatch from the mill and check the mass. If the mass differs from the theoretical value by more than + 0,5 % or − 1,5 %, discard the batch and remix.

e) Cool the rolls to 27 °C ± 5 °C using running water at ambient temperature.

f) Close the rolls of the mill to a tight nip and grind the masterbatch three times through the rolls, rolling the rubber into a ball between each pass.

g) Sheet the masterbatch through a mill opening of 1,4 mm.

h) Store the masterbatches in airtight containers at an ambient temperature of 23 °C ± 2 °C. The storage period should preferably not exceed three months.

A.4 Test mix preparation

The surface temperature of the mill rolls shall be maintained at 70 °C ± 5 °C throughout the mixing.

	Duration (min)
a) Band the rubber with the mill opening set at 0,8 mm. Make two 3/4 cuts from each side.	0,75
b) Add the MBT or TBBS and sulfur masterbatches. Make six 3/4 cuts from each side.	2,00
c) Cut the batch from the mill and pass the rolled batch endwise through the rolls two times.	0,25

Total time	3,00

d) Check the mass of the batch. If the mass differs from the theoretical value by more than + 0,5 % or − 1,5 %, discard the batch and remix.

e) Sheet the batch through a mill opening of 1,4 mm.

f) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 23529.

Annex B
(informative)

Precision statement for both mill and internal mixer

B.1 Background

This precision statement was obtained by an interlaboratory test programme (ITP) conducted in 2005 on natural rubber (STR 20 CV 60) compounds mixed in the laboratory mill and in various sizes of laboratory internal mixer (LIM). The carbon black used was IRB7.

NOTE 1 The LIMs used covered those types often described as “miniature”, “intermediate” and “laboratory”.

The ITP was carried out using the precision procedures and guidelines described in ISO/TR 9272. Reference should be made to this International Standard for other details and for terminology on precision determination.

Two measurement methods were evaluated for each type of mixing procedure: stress-strain testing and curemeter testing. Stress-strain property precision was evaluated for stress (modulus) at 100 %, 200 % and 300 % elongation, elongation at break and tensile strength. Curemeter precision was evaluated for M_H (maximum torque), M_L (minimum torque), t_{s1} (scorch time or time to a 1 dN·m rise), $t'_c(50)$ (time to 50 % of full torque) and $t'_c(90)$ (time to 90 % of full torque).

A type 2 precision was evaluated. Each compound was mixed and prepared twice on each of the two test days and tested separately for test day 1 and test day 2 at an interval of one week apart.

The test result for each property was taken as the average of the two replicated test values obtained on the two mixes prepared on a test day and the precision is expressed in the units of measurement of the test results.

For the LIM testing, nine laboratories participated in the test programme. However, each laboratory did not have the same type or size of mixer. The sizes of the mixers used in the ITP were 80 cm^3, 85 cm^3, 242 cm^3, 270 cm^3, 588 cm^3, 1 200 cm^3, 1 500 cm^3, 1 530 cm^3, 1 600 cm^3 and 3 000 cm^3.

NOTE 2 Two of the laboratories used FEF black instead of IRB7 and one laboratory used the oscillating-disc rheometer instead of the moving-die rheometer. The results from these laboratories were not included in the analysis.

In addition to being used to determine the precision, the data from the ITP was analysed to determine if, and if so how, the type of mixer (size and other operating conditions) influenced the test results. Some laboratories submitted data from more than one size of mixer, and the data from these separate mixers were incorporated into the ITP as data from individual laboratories. This gave a database equivalent to 12 laboratories. Each of the mixer-laboratory combinations was designated as a pseudo-laboratory.

The precision results as determined from this ITP may not be applied to acceptance or rejection testing for any group of materials or products without documentation that the results of this precision determination actually apply to the materials or products tested.

B.2 Precision results

B.2.1 General

The precision results for both types of mixer and for both stress-strain and curemeter testing are given in Tables B.1, B.2, B.3 and B.4. These results were obtained using the outlier deletion procedures described in ISO/TR 9272:2005. General statements for the use of the precision results are cited below. These are given in terms of the absolute precision, *r* and *R*, and also for relative precision, (*r*) and (*R*).

B.2.2 Repeatability

The repeatability, or local-domain precision, for each of the test methods has been established by the values found in Tables B.1, B.2, B.3 and B.4 for each measurement parameter listed in the tables. Two individual test results obtained in the same laboratory (by the proper use of this International Standard) that differ by more than the tabulated values for *r*, in measurement units or (*r*) in percent, shall be considered suspect, i.e. to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.

B.2.3 Reproducibility

The reproducibility, or global-domain precision, for each of the test methods has been established by the values found in Tables B.1, B.2, B.3 and B.4 for each measurement parameter listed in the tables. Two individual test results obtained in different laboratories (by the proper use of this International Standard) that differ by more than the tabulated values for *R*, in measurement units, or (*R*), in percent, shall be considered suspect, i.e. to have come from different populations. Such a decision suggests that some appropriate investigative action be taken.

B.3 Mixing conditions

Table B.5 gives mixing conditions for each mixer used in the ITP, i.e. head temperature, rotor speed, rotor type, etc.

B.4 Comparison of relative precision

As a general point, comparisons of (r) and (R) are the only valid type of comparison because of the differences in absolute property values. The precision results from mill and laboratory internal mixer (LIM) mixing can be summarized as follows.

For stress-strain testing (see Table B.1 and Table B.3), the overall average values of the repeatability (r) for mill and LIM mixing are 6,98 % and 4,76 %, respectively. The values of the overall average reproducibility (R) are 20,7 % and 20,9 %, respectively.

For curemeter testing (see Table B.2 and Table B.4), the overall average values of (r) for mill and LIM mixing are 8,82 % and 5,92 %, respectively. For reproducibility (R), the average values are 38,1 % and 29,5 %, respectively.

The small numerical differences in (*r*) and in (*R*) are typical (and not really significant) of comparisons of this type, given the inherent variations in the results of ITP testing.

As a result, it can be stated that this ITP shows that there is no significant difference between the mill and the LIM mixing procedure.