BS ISO 1407:2011

BSI Standards Publication

Rubber — Determination of solvent extract

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National foreword

This British Standard is the UK implementation of ISO 1407:2011. It supersedes BS ISO 1407:2009, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/23, Test methods for rubber and non-black compounding ingredients.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

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Rubber — Determination of solvent extract

Caoutchouc — Détermination de l'extrait par les solvants

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1407 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 1407:2009), which has been technically revised with the addition of a Method D for raw rubbers only.

Rubber — Determination of solvent extract

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.

IMPORTANT — Certain procedures specified in this International Standard may involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This International Standard specifies four methods for the quantitative determination of the material extractable from raw rubbers, both natural and synthetic; two of the methods are also applicable to the unvulcanized and vulcanized compounds of raw rubbers.

Method A measures the mass of the solvent extract, after evaporation of the solvent, relative to the mass of the original test portion.

Method B measures the difference in the mass of the test portion before and after extraction.

Method C, which is for raw rubbers only, measures the difference in the mass of the test portion before and after extraction using boiling solvent.

Method D, which is for raw rubbers only, measures the difference in the mass of the test portion before and after extraction relative to the mass of the original test portion.

NOTE 1 Depending on the test method used, the conditioning of the test portion and the solvent used, the test result is not necessarily the same.

NOTE 2 Method C generally gives results which are lower than those obtained with methods A and B due to the fact that an equilibrium is set up, particularly if large test portions are used, depending on the content and the nature of the extractable matter. Method C is, however, a quicker method than method A or method B.

NOTE 3 Methods C and D are not suitable if the test portion disintegrates during the extraction.

NOTE 4 Method D is normally used for production controls.

Recommendations as to the solvent most appropriate for each type of rubber are given in Annex A.

2 Normative references

The following referenced documents are indispensable for the application 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 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

ISO 4661-2, *Rubber, vulcanized — Preparation of samples and test pieces — Part 2: Chemical tests*

3 Principle

A rubber test piece is extracted with an appropriate solvent in suitable equipment.

The amount extracted is obtained by measuring either the mass of the extract or the mass of the test piece after extraction and comparing it to the mass of the original test piece.

4 Solvent

During the analysis, use only solvents of recognized analytical grade.

One of the solvents recommended in Table A.1 should preferably be used, unless otherwise specified or agreed between the interested parties.

IMPORTANT — Persons using this International Standard should consult the safety data sheet for the solvent before its use and take appropriate measures.

5 Apparatus

5.1 Balance, precision ±0,1 mg.

5.2 Extraction apparatus: Examples of suitable types of extraction apparatus are shown in Figure 1. Any other type of apparatus which performs the same extraction function may be used, provided it can be demonstrated to give results which are the same as those obtained using the types of apparatus shown in Figure 1.

NOTE The apparatus used for methods C and D is the same as that used for methods A and B, but does not have an extraction cup.

5.3 Regulated heating system.

5.4 Rotary evaporator or any other suitable type of evaporation equipment.

5.5 Drying equipment, of the oven or vacuum desiccator type.

6 Preparation of test pieces

6.1 Methods A and B

For raw rubber and unvulcanized compounds, select a laboratory sample in accordance with ISO 1795 and pass it at ambient temperature through a two-roll laboratory mill or a press to obtain sheets about 0,5 mm in thickness.

For vulcanized rubber, select a laboratory sample in accordance with ISO 4661-2. Since the efficiency of the extraction is a function of the solid-solvent contact area, comminute the laboratory sample, if necessary, to give fragments of maximum surface area 2 mm^2.

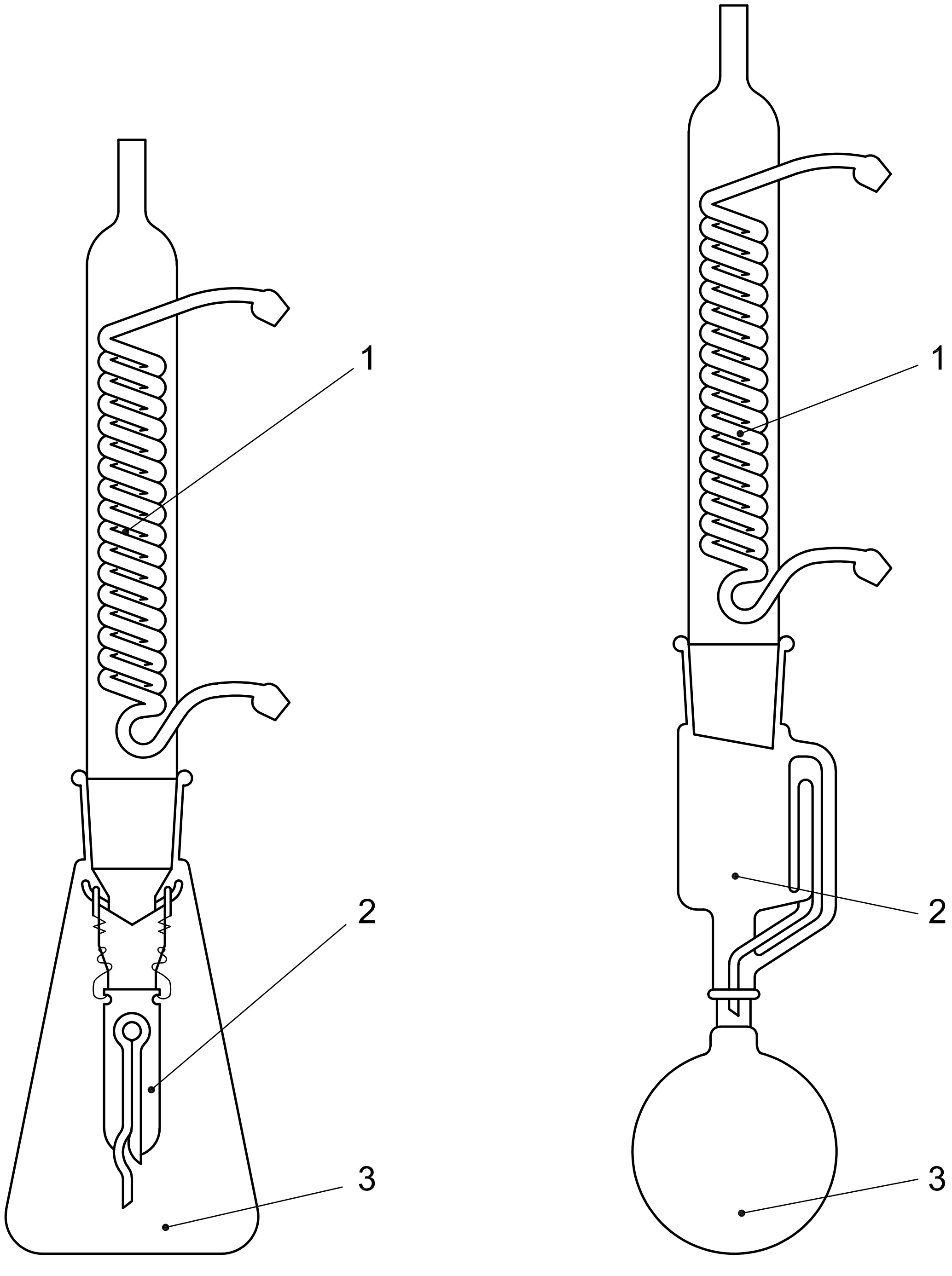
6.2 Method C (raw rubbers only)

6.2.1 Select a laboratory sample in accordance with ISO 1795 and prepare it by either procedure 6.2.2 or procedure 6.2.3.

6.2.2 Pass the laboratory sample at ambient temperature through a two-roll laboratory mill or a press to obtain sheets of 0,5 mm or less in thickness.

The thickness of the sheets is important for the efficiency of the extraction. If it is difficult to obtain sheets of thickness 0,5 mm or less, the roll or press temperature may be increased to a maximum of 100 °C.

6.2.3 Comminute the laboratory sample to give fragments of maximum surface area 2 mm^2.



a) Kumagawa all-glass extraction apparatus

b) Soxhlet all-glass extraction apparatus

Key

1 condenser
2 extraction chamber
3 receiver flask

It is recommended that the ground joints be ungreased.

Figure 1 — Suitable types of extraction apparatus

6.3 Method D (raw rubbers only)

Select a laboratory sample in accordance with ISO 1795. Press a test piece into chromium-nickel wire gauze and roll the gauze into a tube.

7 Procedure

7.1 General

Carry out the determination in duplicate.

7.2 Method A

7.2.1 Dry the empty receiver flask to constant mass and weigh it (m_1).

7.2.2 Weigh, to the nearest 1 mg, a test piece of 2 g to 5 g (m_0), depending on the amount of extract expected.

7.2.3 Place the weighed test piece in a cellulose extraction thimble or wrap it in a filter paper or in a woven wire cloth with a mesh suited to the nature and size of the fragments in the test piece (e.g. 150 µm or 100 mesh). These wrapping materials shall have been previously cleaned in the solvent to be used. Place the wrapped test portion in the extraction chamber of the apparatus.

7.2.4 Introduce a quantity of solvent equal to two or three times the extraction chamber volume into the receiver flask. Then assemble the extraction apparatus.

7.2.5 Carry out the extraction for 16 h ± 0,5 h (or for a shorter time if it can be demonstrated that the extraction is complete). Adjust the heating conditions during the extraction so that the solvent distils at a rate at which the extraction cup is filled at least five times per hour.

7.2.6 At the end of the heating period, turn off the heater, allow the apparatus to cool, remove the extraction chamber from the apparatus and discard the rubber test portion unless it is required for further testing.

7.2.7 Using a rotary evaporator and/or other suitable equipment, remove the solvent from the extract until a constant mass is obtained (i.e. until the difference between two successive weighings at 30 min intervals is 0,2 % or less). Record the mass of the receiver flask plus extract (m_2).

The solvent may also be evaporated from the open flask by gentle heating on the heater used for the extraction.

CAUTION — This may be done only where local health and safety regulations permit and only in a well-ventilated fume cupboard.

7.2.8 Carry out a blank test, going through the entire procedure using the same type of apparatus and same quantity of solvent as for the test piece, but omitting the test piece.

7.3 Method B

7.3.1 Weigh, to the nearest 1 mg, a test piece of 0,5 g to 5 g (m_0), depending on the amount of extract expected.

7.3.2 Place the weighed test piece in a cellulose extraction thimble or wrap it in a filter paper or in a woven wire cloth with a mesh suited to the nature and size of the fragments in the test piece (e.g. 150 µm or 100 mesh). These wrapping materials shall have been previously cleaned in the solvent to be used. Place the wrapped test piece in the extraction chamber of the apparatus.

7.3.3 Introduce a quantity of solvent equal to two or three times the extraction chamber volume into the receiver flask. Then assemble the extraction apparatus.

7.3.4 Carry out the extraction for 16 h ± 0,5 h (or for a shorter time if it can be demonstrated that the extraction is complete). Adjust the heating conditions during the extraction so that the solvent distils at a rate at which the extraction cup is filled at least five times per hour.

7.3.5 At the end of the heating period, turn off the heater, allow the apparatus to cool, remove the extraction chamber from the apparatus and take out the test piece. Discard the solvent in an appropriate manner.

7.3.6 Dry the test piece in an oven to constant mass (i.e. until the difference between two successive weighings at 30 min intervals is 0,2 % or less) at a temperature higher than the boiling point of the solvent used. A drying time of about 1 h at 100 °C in a ventilated oven is usually sufficient.

CAUTION — For safety reasons, gently blot the test piece with absorbent tissue to remove excess solvent or air dry it in a drying chamber before placing it in the oven.

Allow the dried test piece to cool in a desiccator and weigh.

It is recommended that the dried test piece be checked to verify that oxidation has not occurred.

NOTE In the presence of air, too high a drying temperature can cause degradation of the test piece and influence the result. The use of a vacuum lowers the boiling point of the solvent.

7.4 Method C

7.4.1 Cut the laboratory sample of raw rubber into sheets weighing between 90 mg and 110 mg.

7.4.2 Weigh, to the nearest 0,1 mg, a test piece of 200 mg to 600 mg (m_0), depending on the amount of extract expected.

7.4.3 Place the weighed test piece in a 150 ml to 300 ml receiver flask.

7.4.4 Add 25 cm^3 of solvent for each 100 mg of test piece. Connect the condenser to the receiver flask and reflux for 30 min.

7.4.5 At the end of the reflux period, allow the apparatus to cool and remove the receiver flask from the condenser. Decant the solvent and add the same amount of fresh solvent as was added in 7.4.4. Connect the condenser to the receiver flask and reflux for another 30 min.

7.4.6 At the end of the second reflux period, allow the apparatus to cool and remove the receiver flask from the condenser. Decant off the solvent and add the same amount of fresh solvent as was added in 7.4.4. Connect the condenser to the receiver flask and reflux for another 30 min.

7.4.7 At the end of the third reflux period, turn off the heater, allow the apparatus to cool and remove the receiver flask from the condenser. Pour the contents of the receiver flask into a clean-woven cloth with a mesh suited to the nature and size of the fragments in the test piece in order to recover the extracted test piece. Discard the solvent in an appropriate manner.

If particles are observed to be passing through the cloth, either repeat the whole procedure from 7.4.1 to 7.4.7 using a more suitable cloth or use method A or B.

If the mass of the cloth is known, the following drying and weighing operations can be carried out with the test piece on the cloth.

7.4.8 Dry the test piece in an oven to constant mass (i.e. until the difference between two successive weighings at 30 min intervals is 0,2 % or less) at a temperature higher than the boiling point of the solvent used. A drying time of about 1 h at 100 °C in a ventilated oven is usually sufficient.

CAUTION — For safety reasons, gently blot the test piece with absorbent tissue to remove excess solvent or air-dry it in a drying chamber before placing it in the oven.

Allow the dried test piece to cool in a desiccator and weigh.

It is recommended that the dried test piece be checked to verify that oxidation has not occurred.

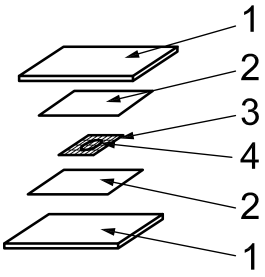
NOTE In the presence of air, too high a drying temperature can cause degradation of the test piece and influence the result. The use of a vacuum lowers the boiling point of the solvent.

7.5 Method D

7.5.1 Weigh a test piece of 0,5 g ± 0,05 g to the nearest 1 mg.

7.5.2 Prepare the chromium nickel gauze[1)] tube in the following way.

a) Clean the gauzes in the chosen solvent for 8 h and dry them in an oven maintained at 105 °C ± 5 °C for 1 h. Weigh the gauze to the nearest 0,1 mg (m_7) and prepare a press assembly as shown in Figure 2, taking care that the test piece is in the centre gauze.



Key
1 press plate
2 polyester film
3 chromium nickel gauze
4 test piece

Figure 2 — Press assembly for method D

b) Press at 150 °C ± 5 °C for 10 s at 130 kN.

c) Remove from the press and cool to room temperature.

d) Weigh the gauze and test piece to the nearest 0,1 mg (m_5).

e) Carefully roll the gauze into a tube form.

f) Close one end of the tube by folding it, so it does not unroll.

7.5.3 Place the gauze tube in the Soxhlet extractor.

7.5.4 Introduce 125 ml of solvent into the receiver flask. Then assemble the system.

[1)] Chromium nickel gauze (woven wire cloth), dimensions: 80 mm × 80 mm; meshes per 25,4 mm: 100; nominal aperture size: 0,14 mm; wire diameter: 0,11 mm.

7.5.5 Carry out the extraction for a length of time that allows a sufficient number of extraction cycles to take place. The number of extraction cycles and/or extraction time may be different for each raw rubber plus solvent combination and the heating equipment used and shall be determined beforehand. See the example given in Annex C for ethylene-propylene-diene (EPDM) in 2-butanone.

Extraction starts the moment the extraction liquid starts to boil.

7.5.6 At the end of the extraction, turn off the heating system and allow the apparatus to cool. Discard the solvent in an appropriate manner.

7.5.7 Take out the gauze tube from the extractor and dry it in an oven to constant mass (m_6) at a temperature higher than the boiling point of the solvent used, i.e. until the difference between two successive weighings at 30 min intervals is 0,2 % or less. A drying time of about 1 h at 100 °C in a ventilated oven is usually sufficient. Cool the gauze tube in a desiccator at room temperature for 15 min ± 1 min.

8 Calculation and expression of results

8.1 Method A

The amount of solvent-extractable material extracted, w_{ex1}, expressed as a percentage mass fraction, is given by the equation:

$$w_{\text{ex1}} = \frac{(m_2 - m_1) - (m'_2 - m'_1)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test piece;

m_1 is the mass, in grams, of the empty receiver flask;

m_2 is the mass, in grams, of the receiver flask plus extract;

m'_1 is the mass, in grams, of the empty receiver flask in the blank test;

m'_2 is the mass, in grams, of the receiver flask in the blank test after the extraction.

8.2 Method B

The amount of solvent-extractable material extracted, w_{ex2}, expressed as a percentage mass fraction, is given by the equation:

$$w_{\text{ex2}} = \frac{m_0 - m_3}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test piece;

m_3 is the mass, in grams, of the test piece after the extraction.

8.3 Method C

The amount of solvent-extractable material extracted, w_{ex3}, expressed as a percentage mass fraction, is given by the equation:

$$w_{ex3} = \frac{m_0 - m_4}{m_0} \times 100$$

where

m_0 is the mass, in milligrams, of the test piece;

m_4 is the mass, in milligrams, of the test piece after the extraction.

8.4 Method D

The amount of solvent-extractable material extracted, w_{ex4}, expressed as a percentage mass fraction, is given by the equation:

$$w_{ex4} = \frac{m_5 - m_6}{m_5 - m_7} \times 100$$

where

m_5 is the mass, in milligrams, of gauze and test piece before extraction;

m_6 is the mass, in milligrams, of gauze and test piece after extraction;

m_7 is the mass, in milligrams, of gauze.

8.5 Expression of results

For all four methods, take the test result as the average of the two determinations carried out.

9 Precision

See Annex B.

10 Test report

The test report shall include the following information:

a) laboratory sample details:

    1) a full description of the laboratory sample and its origin,

    2) details of the method of preparation of the test piece from the laboratory sample;

b) test method:

    1) a full reference to this International Standard (i.e. ISO 1407:2011),

    2) the method used (method A, B, C or D),

    3) the type of apparatus used,

    4) the extraction solvent(s) used;

c) test details:

    1) the laboratory temperature and, if necessary, the relative humidity,

   2) the temperature used to dry the test piece after the extraction,
   3) details of any procedures not specified in this International Standard as well as details of any incidents which could have influenced the results;
   4) the number of extraction cycles and the extraction time (method D);

d) test results:
   1) the individual test results,
   2) the mean of the individual results;

e) the date(s) of testing.

Annex A
(informative)

Recommended solvents

Table A.1 gives the solvents recommended for each type of rubber.

Table A.1 — Recommended solvents (by rubber family)

Rubber family (the various groups are defined in ISO 1629)	**Solvent**[a]	
	Raw rubber/ unvulcanized rubber	**Vulcanized rubber**
Group M		
Copolymer of ethyl acrylate (or other acrylates) and a small amount of a monomer which facilitates vulcanization (ACM) (usually known as acrylic rubber)	**Heptane** or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Copolymer of ethyl acrylate (or other acrylates) and ethylene (AEM)	**Ethanol**	**Ethanol**
Chloropolyethylene (CM)	**Acetone**	**Acetone**
Chlorosulfonylpolyethylene (CSM)	**Acetone**	**Acetone**
Alkyl chlorosulfonylpolyethylene (ACSM)[b]	**Acetone**	**Acetone**
Ethylene-propylene copolymer (EPM)	**Acetone**, 2-butanone or ETA[c]	**Acetone**
Ethylene-propylene-diene terpolymer (EPDM)	**Acetone**, 2-butanone or ETA[c]	**Acetone**
Copolymer of ethylene and vinyl acetate (EVM)	**Ethanol**	**Ethanol**
Fluorocarbon rubbers (FKM)	**Ethanol**	**Ethanol** or acetone
Group O		
Polychloromethyloxirane (CO)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Copolymer of ethylene oxide and chloromethyloxirane (ECO)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Copolymer of epichlorohydrin and allyl glycidyl ether (GCO)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Terpolymer of epichlorohydrin, ethylene oxide and allyl glycidyl ether (GECO)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Copolymer of propylene oxide and allyl glycidyl ether (GPO)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Group Q		
Polymethylvinylsiloxane (VMQ)	**Acetone**	**Acetone**
Polymethylvinylsiloxane with fluorine substituent groups (FVMQ)	**Acetone**	**Acetone**
Group R		
Natural rubber (NR) or synthetic isoprene rubber (IR)	**ETA**[c] or acetone	**Acetone** or ETA[c]
Polybutadiene (BR)	**ETA**[c] or acetone	**Acetone** or ETA[c]
Isobutene-isoprene rubber (IIR)	**Acetone**	**Acetone**
Styrene-butadiene rubber (SBR)	**ETA**[c] or acetone	**Acetone** or ETA[c]

Table A.1 (*continued*)

Rubber family (the various groups are defined in ISO 1629)	**Solvent**[a]	
	Raw rubber/ unvulcanized rubber	**Vulcanized rubber**
Hydrogenated styrene-butadiene rubber (HSBR)[b]	**ETA**[c] or acetone	**Acetone** or ETA[c]
Acrylonitrile-butadiene rubber (NBR)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Hydrogenated acrylonitrile-butadiene rubber (HNBR)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Carboxylic-acrylonitrile-butadiene rubber (XNBR)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*- hexane
Chloroprene rubber (CR)	**Ethanol**, acetone, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Brominated or chlorinated isobutene-isoprene rubber (BIIR or CIIR)	**Acetone**	**Acetone**
Norbornene rubber (NOR)	**Acetone**	**Acetone**
Group U		
Polyester urethane (AU) and polyether urethane (EU)	**Ethanol**	**Ethanol**
Thermoplastic elastomers (TPEs)		
Styrene-butadiene block copolymer (TPS-SBS)	**Acetone** or ETA[c]	**Acetone** or ETA[c]
Styrene-ethylene-butylene block copolymer (TPS-SEBS)	**Acetone** or ETA[c]	**Acetone** or ETA[c]
Styrene-isoprene block copolymer (TPS-SIS)	**Acetone** or ETA[c]	**Acetone** or ETA[c]
Styrene-ethylene-propylene block copolymer (TPS-SEPS)	**Acetone** or ETA[c]	**Acetone** or ETA[c]
Acrylonitrile-butadiene rubber/poly(vinyl chloride) blend [TPZ-(NBR+PVC)]	**Ethanol**	**Ethanol**
Syndiotactic 1,2-polybutadiene (TPZ)[b]	**Acetone**, ethanol or ETA[c]	**Acetone**, ethanol or ETA[c]
Olefin TPE (TPO)	**Acetone**	**Acetone**
Polyamide TPE (TPA)	**Ethanol**	
Copolyester TPE (TPC)	**Ethanol**	
Polyurethane TPE (TPU)	**Ethanol**	

When acetone is used for the extraction, it may be partially converted into high-boiling diacetone alcohol. If it is noticed that the solvent is not evaporating well during the drying step, repeat the extraction using a different solvent or use one of the other methods.

[a] Boldface indicates the preferred solvent.

[b] Not yet standardized.

[c] Ethanol-toluene azeotrope (70 + 30 volume mixture of ethanol and toluene).

Annex B

(informative)

Precision

B.1 General

The precision calculations to provide repeatability and reproducibility values were performed in accordance with ISO/TR 9272, the guidance document for ISO/TC 45 test methods. Consult this for precision concepts and nomenclature.

B.2 Precision results from test programmes in 2006 and 2007

Interlaboratory test programmes (ITPs) for the determination of the precision of methods A to C specified in this International Standard were conducted in 2006 and 2007, using both cured compounds and raw rubbers.

NOTE The ITP on raw rubbers (see Table B.2) was carried out in laboratories in Japan. The ITP on cured compounds (see Table B.1) involved laboratories in various countries.

The raw rubbers SBR 1712, EPDM and NBR were tested using methods A, B and C with the following solvents: ETA for SBR 1712, acetone for EPDM and ethanol for NBR.

Cured compounds of SBR 1500 and SBR 1712 were tested using methods A and B with acetone and ethanol-toluene azeotrope (ETA) as solvents.

For the cured-rubber extraction with acetone as solvent, seven laboratories tested both SBR 1500 and SBR 1712 using method A while nine laboratories tested both SBR 1500 and SBR 1712 using method B. With ETA as solvent, seven laboratories tested both rubbers using method A and eight laboratories tested both rubbers using method B. For the raw-rubber extraction, the number of laboratories which tested EPDM using acetone as solvent was 8, 11 and 4 for methods A, B and C, respectively. The number which tested SBR 1712 using ETA as solvent was 7, 10 and 4 for methods A, B and C, respectively. The number which tested NBR using ethanol as solvent was 8, 9 and 4 for methods A, B and C, respectively.

The testing was conducted on a day 1 vs day 2 repeatability basis: on each day, one measurement was made for that day. Each measurement was designated as a “test result” and all data analysis was conducted on the test results. The precision data resulting from the ITPs are set forth in Table B.1 for the cured rubbers and in Table B.2 for the raw rubbers.

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

General statements for the use of the precision results are given below. These are given in terms of both the absolute precision r and R and the relative precision (r) and (R).

Repeatability — The repeatability, or local domain precision, for each of the test parameters has been established for each material or polymer as the values given in Table B.1 or B.2. Two individual test results (obtained by the proper use of this International Standard) that differ by more than the tabulated values of r, in measurement units, or (r), as a percentage, should be considered suspect, i.e. to have come from different populations. Such a situation should suggest that some appropriate investigative action be taken.

Reproducibility — The reproducibility, or global domain precision, for each of the test parameters has been established for each material or polymer as the values given in Table B.1 or B.2. Two individual test results obtained in different laboratories (by the proper use of this International Standard) that differ by more than the tabulated values of R, in measurement units, or (R), as a percentage, should be considered suspect, i.e. to have come from different populations. Such a situation should suggest that some appropriate investigative action be taken.

Table B.1 — Precision data for cured rubber (percentage extract)

Material	Solvent/method	Mean value	Within lab			Between labs			No. of labs[a]
			s_r	r	(r)	s_R	R	(R)	
SBR 1500	Acetone, method A	7,48	0,130	0,36	4,87	0,22	0,63	8,4	5
	Acetone, method B	7,42	0,076	0,21	2,86	0,29	0,81	11,0	7
SBR 1500	ETA, method A	7,65	0,125	0,349	4,56	0,106	0,30	3,88	5
	ETA, method B	7,85	0,065	0,181	2,31	0,24	0,67	8,55	6
SBR 1712	Acetone, method A	22,80	0,437	1,22	5,36	0,66	1,86	8,16	6
	Acetone, method B	22,63	0,152	0,43	1,88	0,36	1,02	4,49	7
SBR 1712	ETA, method A	22,78	0,500	1,40	6,15	0,644	1,804	7,92	6
	ETA, method B	23,02	0,147	0,41	1,79	0,147	0,412	1,79	4

Notation used:

s_r = within-laboratory standard deviation (in measurement units)
r = repeatability (in measurement units)
(r) = repeatability (as a percentage of mean value)
s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units)
R = reproducibility (in measurement units)
(R) = reproducibility (as a percentage of mean value)
ETA= ethanol-toluene azeotrope

Note that the measurement units are percentages; therefore (r) and (R) are expressed as a percentage of a percentage.

[a] The final number of laboratories in the ITP after deletion of outliers (option 1 of ISO/TR 9272:2005).

Table B.2 — Precision data for raw rubber (percentage extract)

Material	Solvent/method	Mean value	Within lab			Between labs			No. of labs[a]
			s_r	r	(r)	s_R	R	(R)	
SBR 1712	ETA, method A	33,25	0,179	0,50	1,51	0,27	0,76	2,3	6
	ETA, method B	33,13	0,218	0,61	1,84	0,73	2,04	6,2	8
	ETA, method C	33,63	0,085	0,24	0,72	0,23	0,66	1,97	3
EPDM	Acetone, method A	41,91	0,381	1,07	2,55	2,02	5,65	13,5	6
	Acetone, method B	41,79	0,612	1,71	4,10	1,79	5,01	12,0	9
	Acetone, method C	43,30	0,279	0,79	1,82	0,21	0,60	1,39	4
NBR	Ethanol, method A	1,33	0,153	0,43	32,3	0,19	0,52	39,0	7
	Ethanol, method B	1,60	0,058	0,16	10,2	0,43	1,21	75,3	6
	Ethanol, method C	2,72	0,063	0,18	6,57	0,77	2,18	80,26	3

Notation used:

s_r = within-laboratory standard deviation (in measurement units)
r = repeatability (in measurement units)
(r) = repeatability (as a percentage of mean value)
s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units)
R = reproducibility (in measurement units)
(R) = reproducibility (as a percentage of mean value)
ETA= ethanol-toluene azeotrope

Note that the measurement units are percentages, therefore (r) and (R) are expressed as a percentage of a percentage.

[a] The final number of laboratories in the ITP after deletion of outliers (option 1 of ISO/TR 9272:2005).

B.3 Precision results from test programmes in 2009

An interlaboratory test programme (ITP) for the determination of the precision of method D specified in this International Standard was conducted in 2009 using only raw rubbers.

The raw rubbers SBR 1723, EPDM and NBR were tested using method D with the following solvents: ETA for SBR 1723, 2-butanone and acetone for EPDM and ethanol for NBR.

In total, six laboratories carried out this ITP, and the testing was conducted on a day 1-vs-day 2 repeatability basis: on each day, one measurement was made for that day. Each measurement was designated as a “test result” and all data analysis was conducted on the test results. The precision data for raw rubbers resulting from this ITP are set forth in Table B.3.

It is possible that the precision results as determined by this ITP are not applicable to acceptance or rejection testing of any group of materials or products without documentation that the results of the ITP actually apply to the products or materials tested.

General statements for the use of the precision results are given below. These are given in terms of both the absolute precision r and R and the relative precision (r) and (R).

Repeatability — The repeatability, or local domain precision, for each of the test parameters has been established for each raw rubber as the values given in Table B.3. Two individual test results (obtained by the proper use of this International Standard) that differ by more than the tabulated values of r, in measurement units, or (r), as a percentage, should be considered suspect, i.e. to have come from different populations. Such a situation should suggest that some appropriate investigative action be taken.

Reproducibility — The reproducibility, or global domain precision, for each of the test parameters has been established for each raw rubber as the values given in Table B.3. Two individual test results obtained in different laboratories (by the proper use of this International Standard) that differ by more than the tabulated values of R, in measurement units, or (R), as a percentage, should be considered suspect, i.e. to have come from different populations. Such a situation should suggest that some appropriate investigative action be taken.

Table B.3 — Precision data for raw rubber (percentage extract)

Material	Solvent/method	Mean value	Within lab			Between labs			No. of labs[a]
			s_r	r	(r)	s_R	R	(R)	
SBR 1723	ETA, method D	32,12	0,120	0,33	1,01	0,28	0,79	2,46	4
EPDM	2-Butanone, method D	50,11	0,070	0,20	0,40	0,08	0,23	0,45	5
	Acetone, method D	46,10	5,266	1,88	11,42	3,20	8,95	19,41	6
NBR	Ethanol, method D	1,91	0,529	0,19	27,70	0,64	1,79	93,93	4

Notation used:

s_r = within-laboratory standard deviation (in measurement units)
r = repeatability (in measurement units)
(r) = repeatability (as a percentage of mean value)
s_R = between-laboratory standard deviation (for total between-laboratory variation in measurement units)
R = reproducibility (in measurement units)
(R) = reproducibility (as a percentage of mean value)
ETA= ethanol-toluene azeotrope

Note that the measurement units are percentages; therefore (r) and (R) are expressed as a percentage of a percentage.

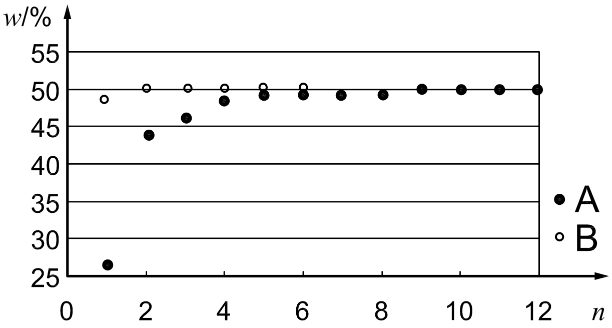
[a] The final number of laboratories in the ITP after deletion of outliers (option 1 of ISO/TR 9272:2005).

Annex C
(informative)

Example of determination of minimum number of extraction cycles for a raw rubber plus solvent combination of EPDM and 2-butanone

One basic condition for method D is the determination of the minimum number of extraction cycles for the raw rubber plus solvent combination. This number of extraction cycles as well as the extraction time can differ for each chosen combination of raw rubber and solvent and according to the heating system used.

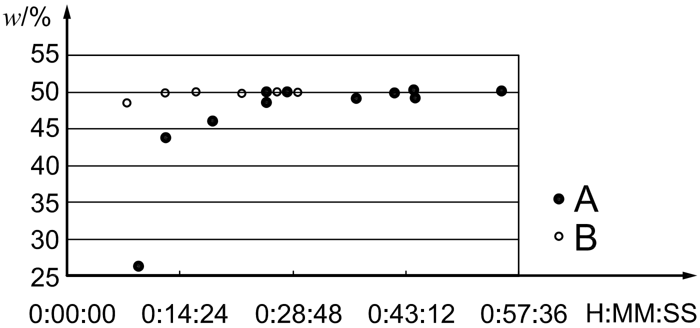
In a first case, tests were carried out at two laboratories to determine the required number of extraction cycles for raw rubber EPDM in a 2-butanone solution. The extractions were carried out with an isomantle (lab A) and with an oil bath (lab B) as heating system. On the basis of the outcome of these tests, the minimum number of extraction cycles required to reach equilibrium was set to five (see Figure C.1), with corresponding extraction times (see Figure C.2).



Key

A	lab A
B	lab B
n	number of extraction cycles

Figure C.1 — Number of extraction cycles for EPDM in 2-butanone



Key

A	lab A
B	lab B
H:MM:SS	extraction time

Figure C.2 — Extraction time for EPDM in 2-butanone

In a second case, tests were carried out at six laboratories which all used different heating systems, such as oil baths and different kinds of electrical devices, which resulted in different extraction times and numbers of extraction cycles (see Table C.1).

Table C.1 — Extraction times and number of extraction cycles for EPDM sample in 2-butanone solvent depending on heating system

Lab No.	No. of extraction cycles	Extraction time min	Heating system
1	5	84	mantle heating
2	≥5	100	electric heater
3	>5	30	oil bath
4	10	30	hot plate
5	6	450	info not available
6	7	30	electrical

Bibliography

[1] ISO 1629, *Rubber and latices — Nomenclature*

[2] ISO/TR 9272:2005, *Rubber and rubber products — Determination of precision for test method standards*

[3] ISO 18064, *Thermoplastic elastomers — Nomenclature and abbreviated terms*

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