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Rubber — Determination of solvent extract

Caoutchouc — Détermination de l'extrait par les solvants

Reference number
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Foreword

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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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

The fifth edition cancels and replace the fourth edition (ISO 1407:2011), which has been technically revised.

The main changes are as follows:

- corrections to clarify mass measurement accuracy have been made;
- warnings to ensure safety in operations have been added;
- the CAS Registry Numbers®[1] have been added for all chemicals listed in this document

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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[1] Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Rubber — Determination of solvent extract

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document 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 determine the applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document can 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 document 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 rubber compounds.

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 an equilibrium which 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 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.

5 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.

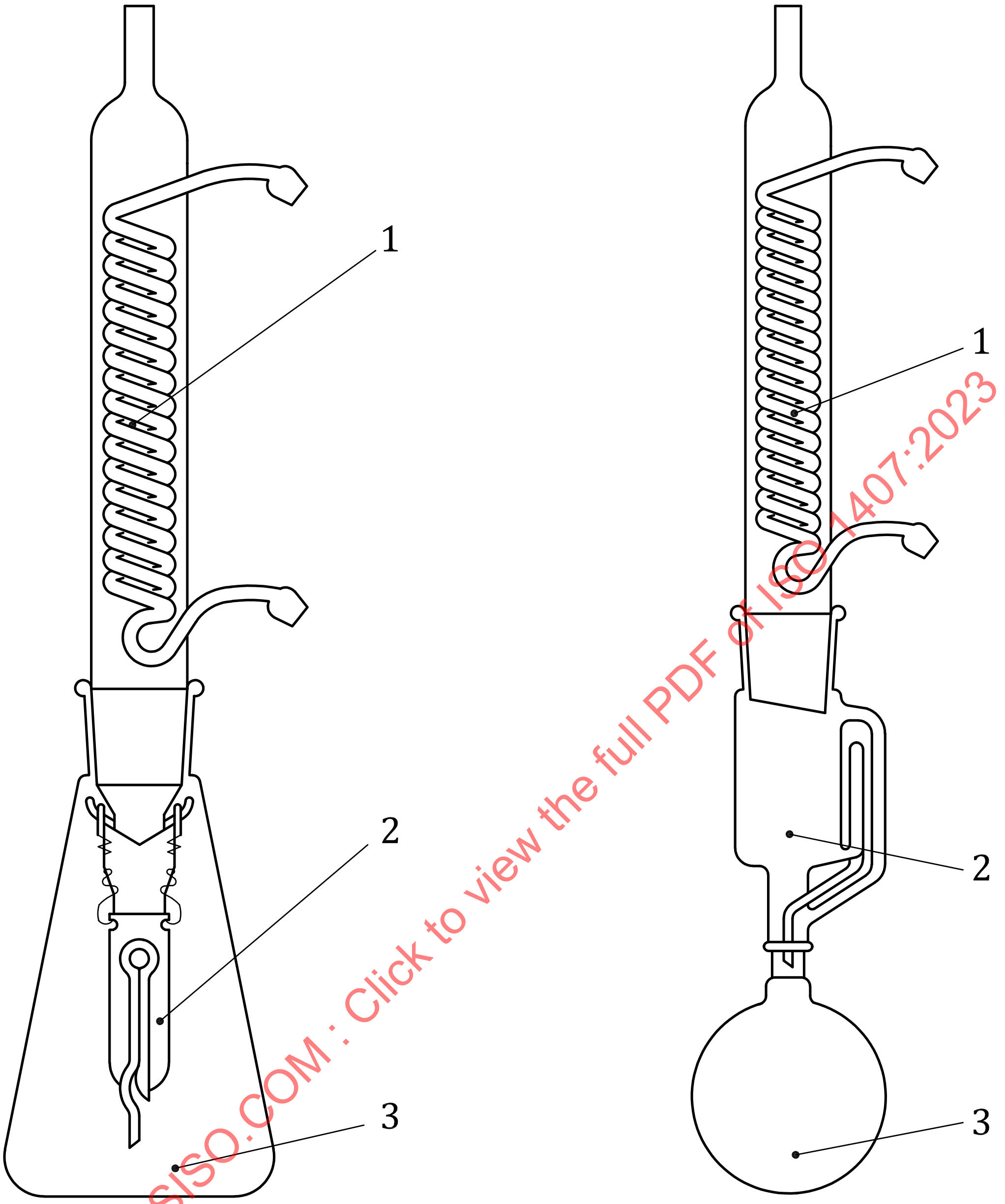
IMPORTANT — Persons using this document should consult the safety data sheet for the solvent before its use and take appropriate measures.

6 Apparatus

6.1 Balance, precision ±0,1 mg.

6.2 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. It is recommended that the ground joints be ungreased.

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



a) Kumagawa all-glass extraction apparatus

b) Soxhlet all-glass extraction apparatus

Key

1	condenser
2	extraction chamber
3	receiver flask

Figure 1 — Suitable types of extraction apparatus

6.3 **Regulated heating system,** with abilities of refluxing for the solvents.

6.4 **Rotary evaporator,** or any other suitable type of evaporation equipment.

6.5 **Drying equipment,** of the oven or vacuum desiccator type.

6.6 **Press,** available setting temperature and pressure.

7 Preparation of test pieces

7.1 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.

7.2 Method C (raw rubbers only)

7.2.1 Select a laboratory sample in accordance with ISO 1795 and prepare it by either the procedure specified in 7.2.2 or 7.2.3.

7.2.2 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.

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

7.3 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.

8 Procedure

8.1 General

Carry out the determination in duplicate.

8.2 Method A

8.2.1 Dry the empty receiver flask to constant mass and weigh it to the nearest 0,1 mg(m_1).

8.2.2 Weigh, to the nearest 0,1 mg, a test piece of 2 g to 5 g (m_0), depending on the amount of extract expected.

8.2.3 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.

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

8.2.5 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 chamber is filled at least five times per hour.

8.2.6 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.

8.2.7 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). Weigh the mass of the receiver flask plus extract to the nearest 0,1 mg(m_2), and record it.

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.

8.2.8 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.

8.3 Method B

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

8.3.2 Place the weighed test piece in a cellulose (CAS 9004-34-6) 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.

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

8.3.4 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 chamber is filled at least five times per hour.

8.3.5 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.

8.3.6 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 to the nearest 0,1 mg (m_3).

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

When the test piece cannot be weighed due to the collapsing of the test piece, try again with a new test piece or apply Method A.

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.

8.4 Method C

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

8.4.2 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.

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

8.4.4 Add 25 ml of solvent for each 100 mg of test piece. Connect the condenser to the receiver flask and reflux for 30 min.

8.4.5 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 8.4.4. Connect the condenser to the receiver flask and reflux for another 30 min.

8.4.6 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 8.4.4. Connect the condenser to the receiver flask and reflux for another 30 min.

8.4.7 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 8.4.1 to 8.4.7 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.

8.4.8 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 to the nearest 0,1 mg (m_4).

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

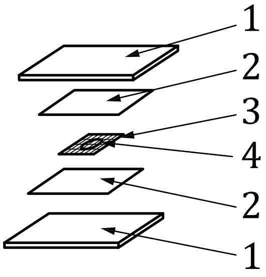
NOTE In the presence of air, too high a drying temperature can cause degradation of the test piece and influence the result. The use of a vacuum lowers the boiling point of the solvent.

8.5 Method D

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

8.5.2 Prepare the chromium nickel gauze[2] (CAS 11106-97-1) 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 (CAS 23038-59-9) film or polytetrafluoroethylene (CAS 9002-84-0) 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.

8.5.3 Place the gauze tube in the Soxhlet extractor.

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

8.5.5 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 can 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) [CAS 25038-36-2] in 2-butanone (CAS 78-93-3).

Extraction starts the moment the extraction liquid starts to boil.

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

8.5.7 Take out the gauze tube from the extractor and dry it in an oven to constant mass 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 and weigh to the nearest 0,1 mg (m_6).

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[2] 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.

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.

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.

9 Calculation and expression of results

9.1 Method A

The amount of solvent-extractable material extracted, w_{ex1}, expressed as a percentage mass fraction, is given by Formula (1):

$$w_{ex1} = \frac{(m_2 - m_1) - (m_2' - m_1')}{m_0} \times 100 \tag{1}$$

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.

9.2 Method B

The amount of solvent-extractable material extracted, w_{ex2}, expressed as a percentage mass fraction, is given by Formula (2):

$$w_{ex2} = \frac{m_0 - m_3}{m_0} \times 100 \tag{2}$$

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.

9.3 Method C

The amount of solvent-extractable material extracted, w_{ex3}, expressed as a percentage mass fraction, is given by Formula (3):

$$w_{ex3} = \frac{m_0 - m_4}{m_0} \times 100 \tag{3}$$

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.

9.4 Method D

The amount of solvent-extractable material extracted, w_{ex4}, expressed as a percentage mass fraction, is given by Formula (4):

$$w_{ex4} = \frac{m_5 - m_6}{m_5 - m_7} \times 100 \quad (4)$$

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.

9.5 Expression of results

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

10 Precision

See Annex B.

11 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 reference to this document (i.e. ISO 1407:2023);
  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 document 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** (CAS 142-82-5) or *n*-hexane (CAS 110-54-3)	**Ethanol** (CAS 64-17-5), acetone (CAS 67-64-1), heptane or *n*-hexane
Copolymer of ethyl acrylate (or other acrylates) and ethylene (AEM)	**Ethanol**	**Ethanol**
Chloropolyethylene (CM) (CAS 64754-90-1)	**Acetone**	**Acetone**
Chlorosulfonylpolyethylene (CSM) (CAS 68037-39-8)	**Acetone**	**Acetone**
Alkyl chlorosulfonylpolyethylene (ACSM)[b]	**Acetone**	**Acetone**
Ethylene-propylene copolymer (EPM) (CAS 9010-79-1)	**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) (CAS 24937-78-8)	**Ethanol**	**Ethanol**
Fluorocarbon rubbers (FKM) (CAS 9011-17-0)	**Ethanol**	**Ethanol** or acetone
Group O		
Polychloromethyloxirane (CO) (CAS 24969-06-0)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Copolymer of ethylene oxide and chloromethyloxirane (ECO) (CAS 24969-10-6)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Copolymer of epichlorohydrin and allyl glycidyl ether (GCO) (CAS 24969-09-3)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Terpolymer of epichlorohydrin, ethylene oxide and allyl glycidyl ether (GECO) (CAS 26587-37-1)	**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) (CAS 67762-94-1)	**Acetone**	**Acetone**
Polymethylvinylsiloxane with fluorine substituent groups (FVMQ)	**Acetone**	**Acetone**
Group R		

[a] Boldface indicates the preferred solvent.

[b] Not yet standardized.

[c] Ethanol-toluene azeotrope (70 + 30 volume mixture of ethanol and toluene) (toluene: CAS 108-88-3).

Table A.1 *(continued)*

Rubber family (the various groups are defined in ISO 1629)	**Solvent**[a]	
	Raw rubber/ unvulcanized rubber	**Vulcanized rubber**
Natural rubber (NR) or synthetic isoprene rubber (IR) (CAS 9006-4-6)	**ETA**[c] or acetone	**Acetone** or ETA[c]
Polybutadiene (BR)(CAS 9003-17-2)	**ETA**[c] or acetone	**Acetone** or ETA[c]
Isobutene-isoprene rubber (IIR) (CAS 9010-85-9)	**Acetone**	**Acetone**
Styrene-butadiene rubber (SBR) (CAS 9003-55-8)	**ETA**[c] or acetone	**Acetone** or ETA[c]
Hydrogenated styrene-butadiene rubber (HSBR)[b] (CAS 66070-58-4)	**ETA**[c] or acetone	**Acetone** or ETA[c]
Acrylonitrile-butadiene rubber (NBR) (CAS 9003-18-3)	**Ethanol**, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Hydrogenated acrylonitrile-butadiene rubber (HNBR) (CAS 88254-10-8)	**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)(CAS 9010-98-4)	**Ethanol**, acetone, heptane or *n*-hexane	**Ethanol**, acetone, heptane or *n*-hexane
Brominated or chlorinated isobutene-isoprene rubber [BIIR (CAS 68441-14-5) or CIIR (CAS 68081-82-3)]	**Acetone**	**Acetone**
Norbornene rubber (NOR)	**Acetone**	**Acetone**
Group U		
Polyester urethane (AU) and polyether urethane (EU)	**Ethanol**	**Ethanol**
Thermoplastic elastomers (TPEs) (See ISO 18064)		
Styrene-butadiene block copolymer (TPS-SBS) (CAS 9003-55-8)	**Acetone** or ETA[c]	**Acetone** or ETA[c]
Styrene-ethylene-butylene block copolymer (TPS-SEBS) (CAS 66070-58-4)	**Acetone** or ETA[c]	**Acetone** or ETA[c]
Styrene-isoprene block copolymer (TPS-SIS) (CAS 25038-32-8)	**Acetone** or ETA[c]	**Acetone** or ETA[c]
Styrene-ethylene-propylene block copolymer (TPS-SEPS) (CAS 68648-89-5)	**Acetone** or ETA[c]	**Acetone** or ETA[c]
Acrylonitrile-butadiene rubber/poly(vinyl chloride) blend [TPZ-(NBR+PVC)], (PVC; CAS 9002-86-2)	**Ethanol**	**Ethanol**
Syndiotactic 1,2-polybutadiene (TPZ)[b] (CAS 9003-17-2)	**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) (CAS 9009-54-5)	**Ethanol**	
When acetone is used for the extraction, it may be partially converted into high-boiling diacetone alcohol (CAS 123-42-2). 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) (toluene: CAS 108-88-3).

Annex B
(informative)

Precision

B.1 General

The precision calculations to provide repeatability and reproducibility values were performed according to ISO/TR 9272.[3)]

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 document 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 versus 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 if there is no documentation proving that the results of the ITP 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 Tables B.1 or B.2. Two individual test results (obtained by the proper use of this document) 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.

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3) Withdrawn.

— 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 Tables B.1 or B.2. Two individual test results obtained in different laboratories (by the proper use of this document) 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

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

Key

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)

NOTE The measurement units are percentages; therefore (r) and (R) are expressed as a percentage of a percentage.

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

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

Key

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)

NOTE The measurement units are percentages; therefore (r) and (R) are expressed as a percentage of a percentage.

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 document 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 versus 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.

The precision results as determined by this ITP may not be applied to acceptance or rejection testing of any group of materials or products if there is no documentation proving that the results of the ITP 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 document) 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 document) that differ by more than the tabulated values of R, in measurement units, or (R), as a percentage, should be considered suspect,