INTERNATIONAL STANDARD

ISO 2006-2

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Rubber latex, synthetic — Determination of mechanical stability —

Part 2: Moderate-speed method under load

Latex de caoutchouc synthétique — Détermination de la stabilité mécanique —

Partie 2: Méthode à vitesse modérée sous charge

Reference number
ISO 2006-2:2009(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 2006-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

ISO 2006 consists of the following parts, under the general title *Rubber latex, synthetic — Determination of mechanical stability*:

— *Part 1: High-speed method*

— *Part 2: Moderate-speed method under load*

Introduction

The mechanical stability of synthetic latices is important in a variety of manufacturing processes, and a number of empirical methods are used for testing. This part of ISO 2006 provides a method of determining the mechanical stability by stirring a latex test portion at a moderate speed under load with shear. It can provide a more accurate indication of latex performance by simulating the actual service conditions.

The design of the mechanical stability test machine utilized in this part of ISO 2006 was originally developed by Maron and Ulevitch [1], and the mechanical stability of various latices has been studied. It has been concluded that the test is rapid and reliable.

Rubber latex, synthetic — Determination of mechanical stability —

Part 2:
Moderate-speed method under load

WARNING — Persons using this part of ISO 2006 should be familiar with normal laboratory practice. This part of ISO 2006 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 part of ISO 2006 specifies a method for the determination of the mechanical stability of synthetic rubber latex. This method measures the mass of coagulum formed when a test portion of latex is stirred for a specified length of time at moderate speed under a relatively high shear stress achieved by applying a load.

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 123, *Rubber latex — Sampling*

ISO 124, *Latex, rubber — Determination of total solids content*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
mechanical stability
resistance to coagulation of latex when subjected to mechanical shear under specified conditions

NOTE The greater the percentage of coagulum formed (w_c as defined in Clause 9), the poorer the mechanical stability.

4 Principle

A test portion of latex is stirred at a moderate speed under load for a given time, and the coagulum formed is separated and weighed. The mass of coagulum formed is inversely proportional to the mechanical stability.

5 Reagents

During the analysis, use only carbonate-free distilled water or water of equivalent purity.

5.1 Surfactant solution: 5 % (by mass) solution of potassium oleate of pH value 10 ± 0,5 or, for use with a latex which is coagulated by potassium oleate solution, a 5 % (by mass) solution of a synthetic anionic or non-ionic surfactant.

6 Apparatus

6.1 Mechanical stability tester[1)], designed to rotate a disc attached to a spindle shaft at a constant stirring speed of 1 000 min^{-1} ± 20 min^{-1} while applying a constant load to the latex container (6.2). The device shall be capable of maintaining a load of up to 500 N to within 2 N. An example of a tester is shown in Figure 1.

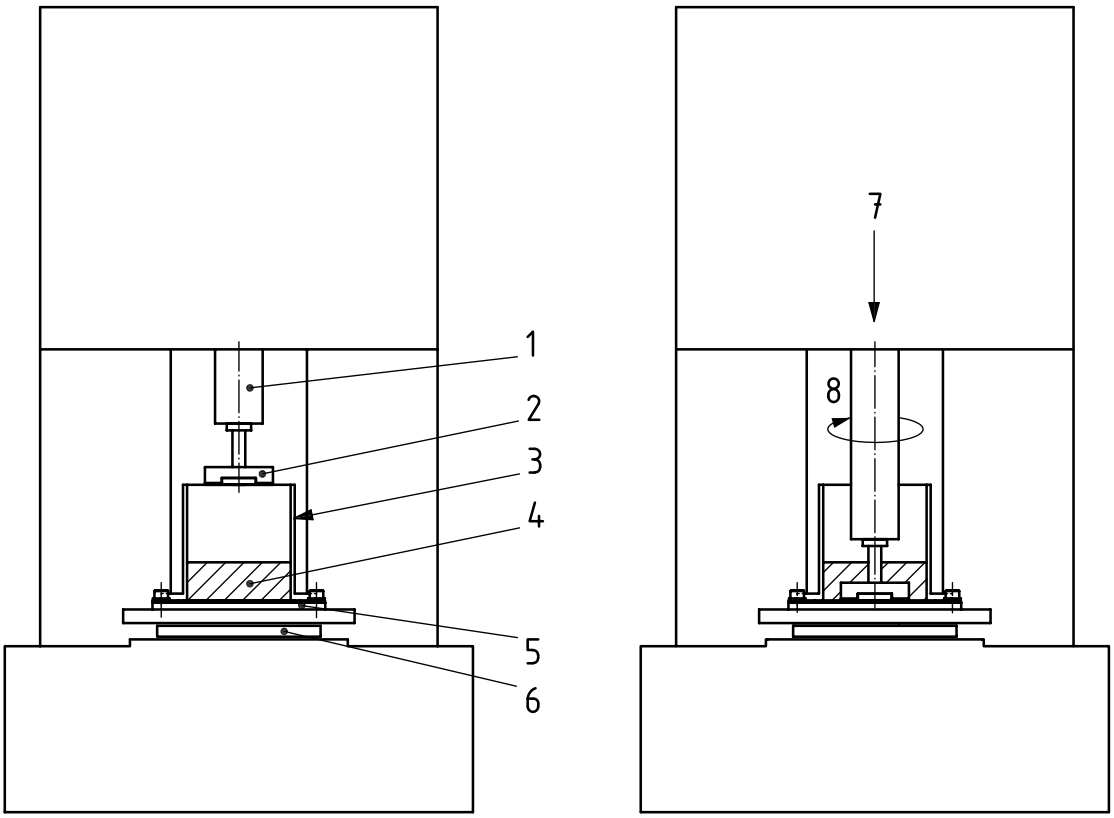
6.2 Latex container, comprising a flat-bottomed plate with a grooved pattern cut into it as shown in Figure 2, a polyethylene liner disc 1,6 mm thick with four holes as shown in Figure 3 and a cylindrical wall with flange assembled with the flat-bottomed plate as shown in Figures 4 and 5.

6.3 Rotating disc, consisting of a vertical stainless-steel shaft 9,5 mm in diameter attached to a disc (also made of stainless steel) with four grooves cut into it. Detailed dimensions are shown in Figure 6.

6.4 Preliminary filter, of stainless-steel wire cloth with an average aperture width of 180 µm ± 10 µm, complying with ISO 3310-1.

6.5 Test filter, consisting of a disc of stainless-steel wire cloth with an average aperture width of 180 µm ± 10 µm, complying with ISO 3310-1, dried to constant mass and weighed to the nearest 1 mg, firmly clamped between two stainless-steel rings of equal internal diameter between 25 mm and 50 mm.

1) Suitable instruments are available commercially, e.g. from Ueshima Seisakusho Co., Ltd., 1053-1 Yaho Kunitachi-shi, Tokyo 186-0011, Japan, Fax:+81-4-2573-1520, and Kumagai Riki Kogyo Co., Ltd., 2-4, Toyotama-kita 3-Chome, Nerima-ku, Tokyo 176-0012, Japan, Fax: +81-3-3994-0520. This information is given for the convenience of users of this part of ISO 2006 and does not constitute an endorsement by ISO of these instruments.

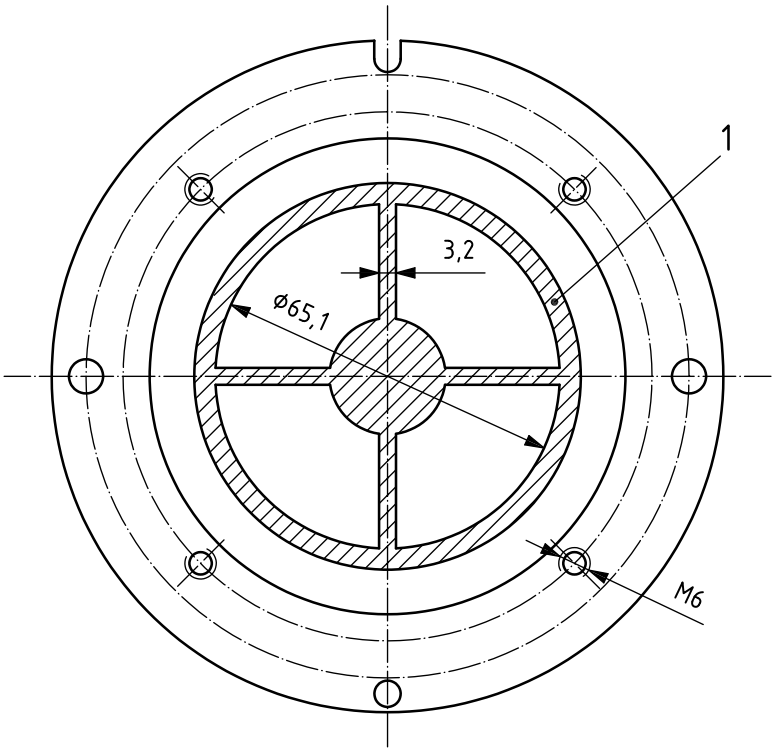


Key

1 spindle shaft
2 rotating disc
3 latex container
4 test portion of latex
5 flat-bottomed plate
6 load cell or scale
7 direction of loading
8 direction of rotation

Figure 1 — Mechanical stability tester

Dimensions in millimetres

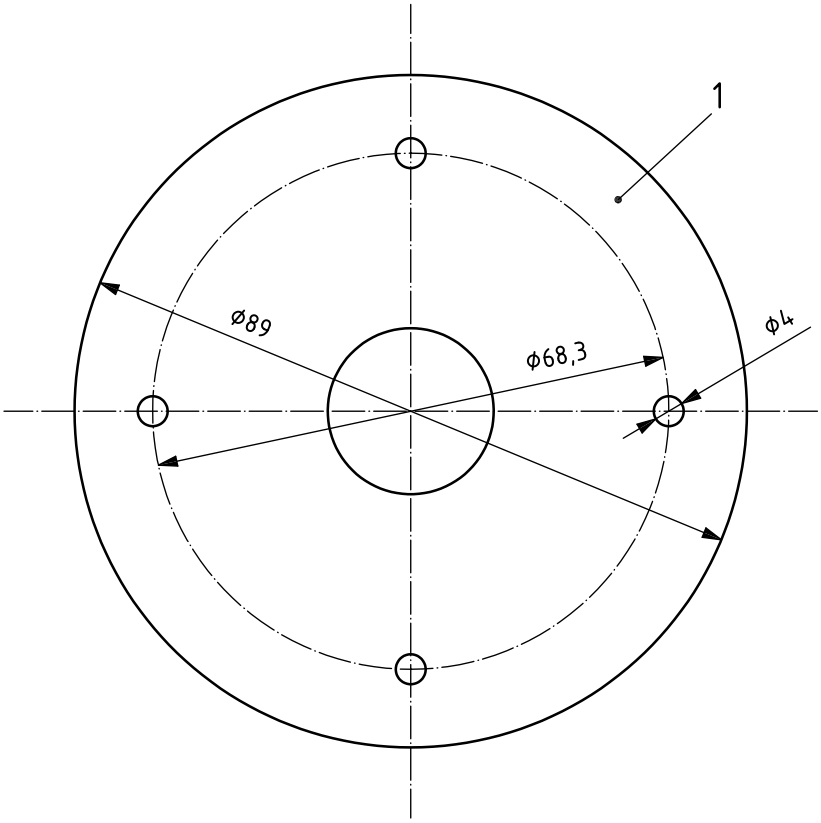


Key

1 groove, 1,6 mm in depth

Figure 2 — Flat-bottomed plate

Dimensions in millimetres



Key

1 disc, 1,6 mm in thickness

Figure 3 — Polyethylene liner disc

Dimensions in millimetres

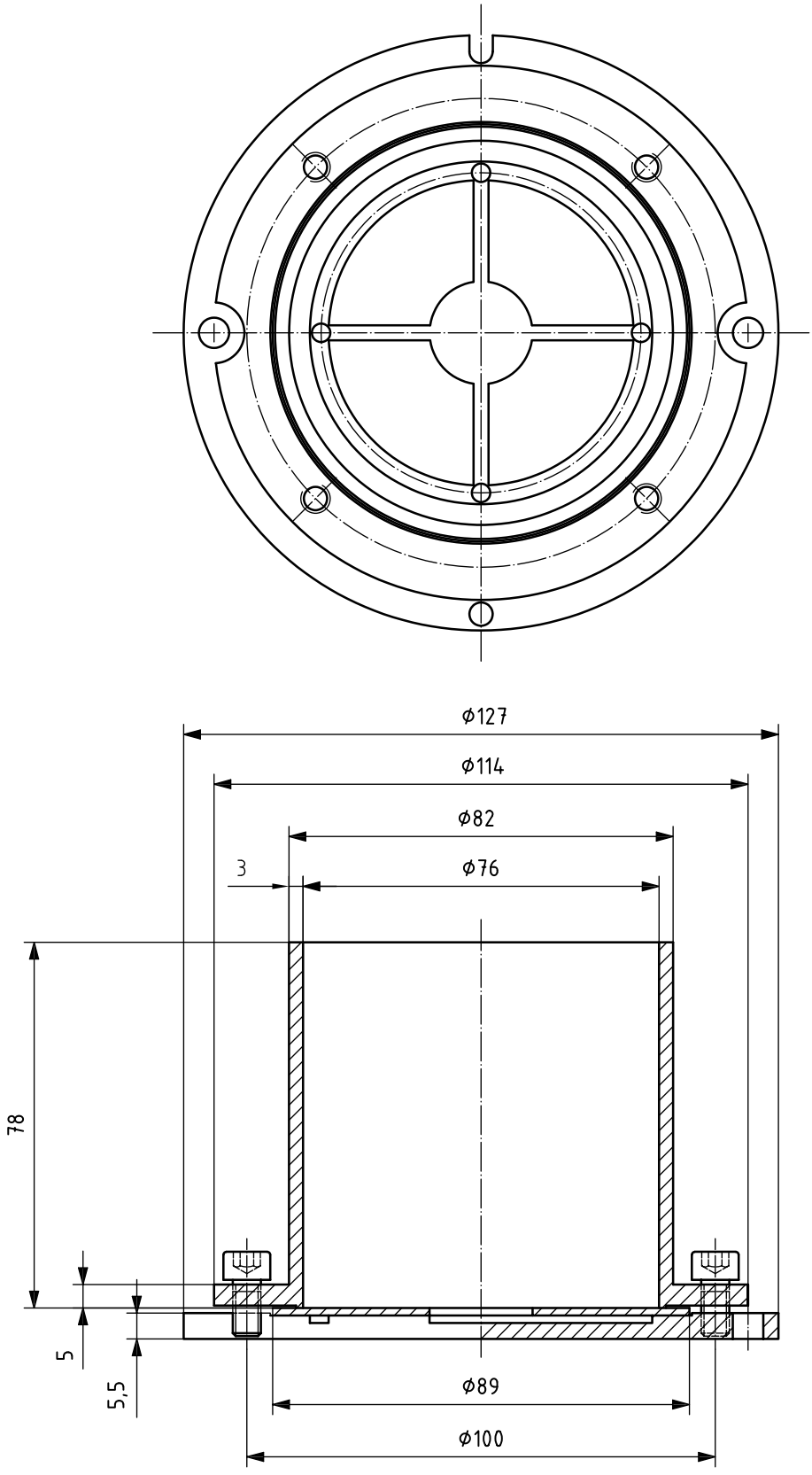
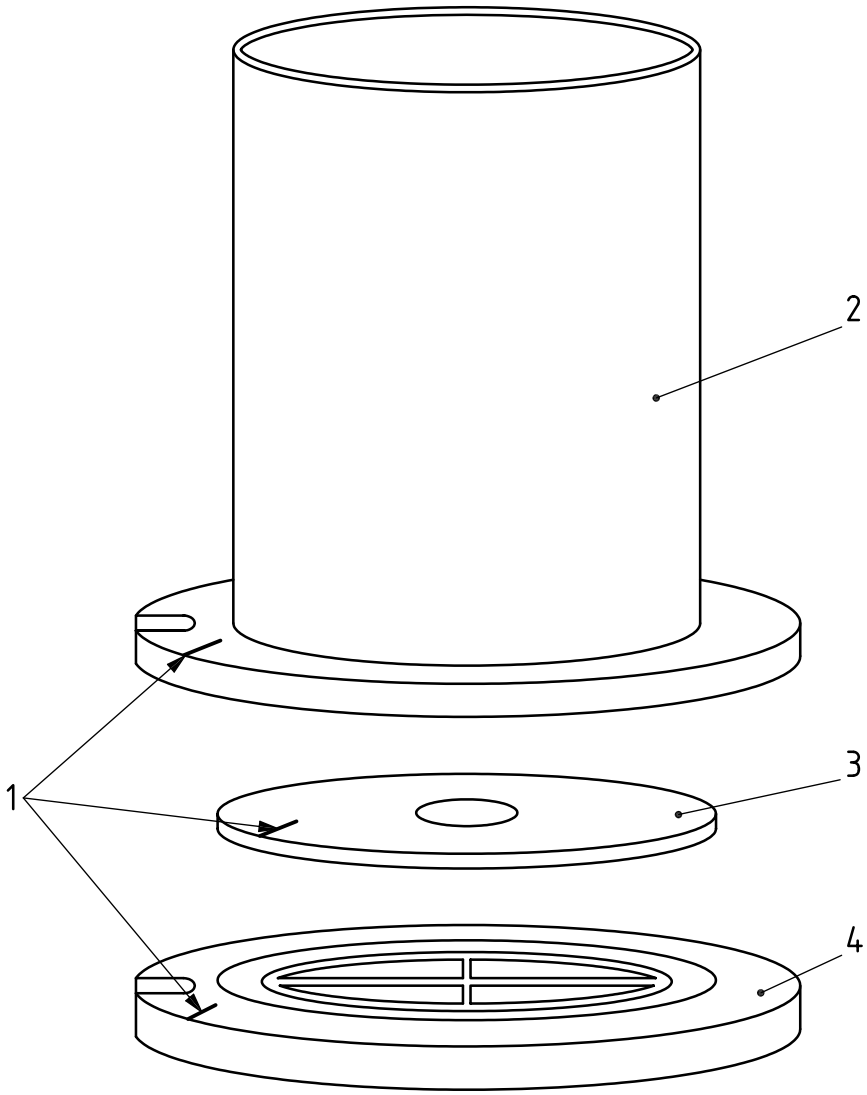


Figure 4 — Latex container



Key

1 positioning lines
2 cylindrical wall with flange
3 polyethylene liner disc
4 flat-bottomed plate

Figure 5 — Assembling the latex container

Dimensions in millimetres

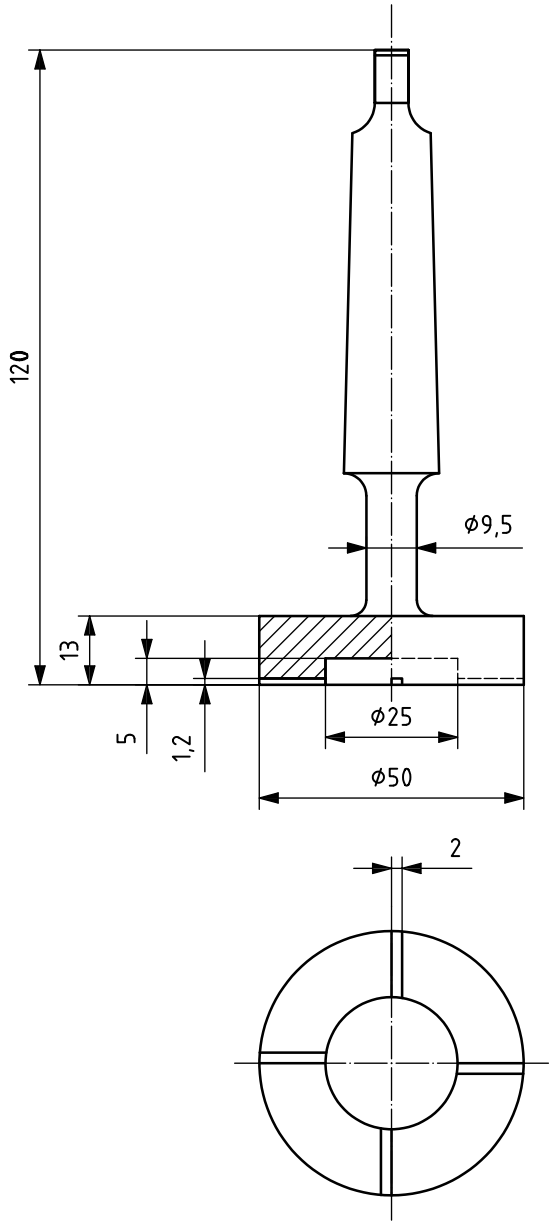


Figure 6 — Shaft and rotating disc

7 Sampling

Carry out sampling in accordance with one of the methods specified in ISO 123.

8 Procedure

8.1 General

A test portion is stirred under shear stress created by the patterned disc which is maintained in contact with the polyethylene liner disc under constant load. The mass of coagulum formed after a specified time is measured.

If the total solids content of the latex is not known, determine it in accordance with ISO 124.

8.2 Preliminary run of the apparatus

8.2.1 Assemble the latex container (6.2) by mounting the polyethylene liner disc between the flange of the cylindrical wall and the flat-bottomed plate and fix them together with bolts as shown in Figures 4 and 5, aligning the positioning marks on the polyethylene disc, the flat-bottomed plate and the flange of the cylindrical wall so that the polyethylene disc can be inserted in exactly the same position again. When a new polyethylene disc is used, mark a positioning line on it with a scratch or paint mark to facilitate maintaining the exact alignment when reassembling.

8.2.2 Place the latex container filled with water in position in the tester and set the rotating disc on the spindle. Then apply a force of 98 N to the container. Start to stir at a rate of 1 000 $min^{-1} \pm 20$ min^{-1}. Adjust the apparatus if the applied load varies by more than $\pm$ 5 N.

8.2.3 Stop running the apparatus after a few minutes. When a new polyethylene liner disc is used, run for at least one hour to condition it to the rotating disc.

8.3 Test

8.3.1 Depending on the latex under test, the load used can be 49 N, 98 N or 147 N and the duration of the test either 300 s or 600 s. The precise conditions shall be established for a given type of latex in a preliminary trial.

8.3.2 Pass the test sample through the preliminary filter (6.4) and cover it to protect it from dust and drying. Note the temperature of the latex.

8.3.3 Place a 50 g $\pm$ 0,5 g test portion of the latex in the latex container.

8.3.4 Place the latex container securely in the tester and mount the rotating disc and shaft. Apply the appropriate load of 49 N $\pm$ 2 N, 98 N $\pm$ 2 N or 147 N $\pm$ 2 N (see 8.3.1). Reset the load scale to zero.

8.3.5 Start the stirrer motor and record the starting time. Adjust the apparatus if the variation in the measured applied load exceeds $\pm$ 5 N.

8.3.6 Stop the test after 300 s or 600 s, unless otherwise specified. Measure and record the temperature of the latex in the container.

NOTE The duration selected for the test depends on the mechanical stability of the latex.

8.3.7 Remove the shaft and rotating disc and wash them free from latex deposits with surfactant solution (5.1) or water (see Clause 5).

8.3.8 Wet the test filter (6.5) with surfactant solution or water and pour the latex and washings onto the test filter. Use surfactant solution or water to ensure quantitative transfer of all the latex and deposits of coagulum, including any skin which might have formed.

8.3.9 Wash the residue on the test filter with surfactant solution or water until it is free from latex and then with water until the washings are neutral to litmus.

8.3.10 Carefully remove the test filter containing the wet solid matter and blot the underside with filter paper.

8.3.11 Dry the test filter and coagulum on a watch glass at 100 °C $\pm$ 5 °C. After 15 min of drying, transfer to a desiccator and allow to cool to ambient temperature, then weigh. Repeat the drying procedure for periods of 15 min until the loss in mass between two successive weighings is less than 1 mg.

8.3.12 If the amount of coagulum formed is too small or too large for an accurate evaluation of the mechanical stability of the latex, repeat the procedure from 8.3.2 to 8.3.11, changing either the load or the duration of the run or both.

NOTE In practice, once meaningful conditions (see 8.3.4 and 8.3.6) have been established for a given latex, it should be possible to adhere to them on a routine basis.

8.3.13 Carry out the procedure described in 8.3.2 to 8.3.11 in duplicate under the same test conditions.

9 Expression of results

The percentage by mass of coagulum formed, w_c, is expressed as a percentage by mass of the total solids content of the latex, using the equation:

$$w_c = \frac{m_c}{\left(\frac{m \times TSC}{100}\right)} \times 100$$

where

m_c is the mass of the dried coagulum, in grams;

m is the mass of the test portion, in grams;

TSC is the total solids content of the latex sample, as a percentage by mass (see 8.1).

NOTE The greater the percentgae of coagulum formed, the poorer the mechanical stability.

10 Test report

The test report shall include the following:

a) a reference to this part of ISO 2006;

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

c) the total solids content at which the latex was tested;

d) the test run duration, the load used and the temperature of the latex in the container before and after the test;

e) the test result, i.e. the percentage by mass of coagulum formed, rounded up to the second decimal place;

f) any unusual features noted during the test;

g) details of any operation not included in this part of ISO 2006 or in the International Standards to which reference is made, as well as details of any operation regarded as optional;

h) the date of the test.

Annex A
(informative)

Precision

A.1 General

An interlaboratory test programme (ITP) to determine the precision of the moderate-speed method with shear described in this part of ISO 2006 (the so-called Maron method) and the high-speed method without shear described in ISO 2006-1 (the so-called Klaxon method) was conducted in 2006 in accordance with ISO/TR 9272, using three synthetic latices and one natural latex (see Table A.1). A type 1 precision was determined, measuring the percentage by mass of coagulum formed relative to the total solids content of the sample.

Of the laboratories participating in the ITP, five laboratories tested the samples using the Maron method, four laboratories used the Klaxon method, while two laboratories used both.

The test result was the mean value from determinations on two test portions of each latex material (i.e. $n = 2$). Test results were obtained on two different days, at intervals of 7 days between each of the two tests.

The test conditions used for each type test are given in Table A.2.

When testing natural rubber latex using the Klaxon method (ISO 2006-1), only two laboratories succeeded in measuring the percentage of coagulum due to clogging of the filter.

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 this precision determination actually apply to the materials or products tested.

Table A.1 — Samples provided for testing

Sample	Concentration %	Viscosity mPa·s
NBR	51	50
X-SBR-1	50	100
X-SBR-2	51	100
NR	61	30

Table A.2 — Test conditions

Item	Maron method (ISO 2006-2)	Klaxon method (ISO 2006-1)
Total solids content	As specified in ISO 124	As specified in ISO 124
Stirring speed	1 000 $min^{-1} \pm 20$ min^{-1}	14 000 $min^{-1} \pm 200$ min^{-1}
Load	98 N	No load applied
Stirring time	300 s	30 min

A.2 Precision results

A.2.1 General

The results of the ITP are given in Tables A.3 and A.4. These results were obtained using the outlier deletion procedures described in Clauses 8, 9 and 10 of ISO/TR 9272:2005.

A.2.2 Repeatability

The repeatability, r, of the two test methods has been established as the appropriate value tabulated in Table A.3 or A.4 for each material. Two single test results that differ by more than this value should be considered suspect and suggest that some appropriate investigative action be taken.

A.2.3 Reproducibility

The reproducibility, R, of the two test methods has been established as the appropriate value tabulated in Table A.3 or A.4 for each material. Two single test results that differ by more than this value should be considered suspect and suggest that some appropriate investigative action be taken.

Table A.3 — Precision data for Maron method (ISO 2006-2)

Material	Mean value	Within-laboratory			Between laboratories		
	%	s_r	r	(r)	s_R	R	(R)
NBR	5,116	0,600	1,697	33	3,312	9,37	183
X-SBR-1	0,124	0,002	0,007	6	0,200	0,57	457
X-SBR-2	0,002	0,001	0,002	98	0,002	0,01	306
NR	1,38	0,133	0,378	33	0,613	1,73	152

s_r is the repeatability standard deviation;

r is the repeatability, in measurement units;

(r) is the repeatability, in percent (relative);

s_R is the reproducibility standard deviation;

R is the reproducibility, in measurement units;

(R) is the reproducibility, in percent (relative).

Table A.4 — Precision data for Klaxon method (ISO 2006-1)

Material	Mean value	Within-laboratory			Between laboratories		
	%	s_r	r	(r)	s_R	R	(R)
NBR	0,142	0,006	0,001 6	12	0,146	0,41	291
X-SBR-1	0,088	0,014	0,040	45	0,013	0,04	43
X-SBR-2	0,003	0,001	0,002	88	0,002	0,01	271
NR [a]	42,171	6,350	17,970	43	10,979	31,07	74

s_r is the repeatability standard deviation;

r is the repeatability, in measurement units;

(r) is the repeatability, in percent (relative);

s_R is the reproducibility standard deviation;

R is the reproducibility, in measurement units;

(R) is the reproducibility, in percent (relative).

[a] Calculated from data from only two laboratories.

Bibliography

[1] MARON, S.H. and ULEVITCH, I.N. *Analytical Chemistry*, Vol. 25 (1953), No. 7, pp. 1087-1091

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

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