INTERNATIONAL STANDARD

ISO 17278

First edition
2013-08-15

Rubber, raw natural — Determination of the gel content of technically specified rubber (TSR)

Caoutchouc naturel brut — Détermination de la teneur en gel des caoutchoucs spécifiés techniquement (TSR)

Reference number
ISO 17278:2013(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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. 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. 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.

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*.

Rubber, raw natural — Determination of the gel content of technically specified rubber (TSR)

1 Scope

This International Standard specifies a method for the determination of gel content for technically specified rubbers (TSR).

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable to 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 2000,[1)]*Rubber, raw natural — Guidelines for the specification of technically specified rubber (TSR)*

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

3 Principle

An LoV-TSR sample is dissolved in toluene under specified conditions, and the gel content is calculated as the percentage mass fraction of the insoluble part of the rubber.

4 Terms and definitions

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

4.1
technically specified rubber
TSR
natural rubber obtained from the latex of *Hevea brasiliensis* (typically processed into block rubber), and having properties complying with the criteria for the grade concerned

5 Reagent

Use only reagents of recognized analytical grade.

5.1 **Toluene**, analytical grade.

CAUTION — The solvent might evaporate from the flask or tube when it is handled. Therefore, handling this solvent should be done only when permitted by local health and safety regulation and only in a well-ventilated appropriate place.

1) Under preparation. (Revision of ISO 2000:2003)

6 Apparatus

6.1 Laboratory centrifuge, capable of rotating at a minimum of 14 000 r/min (revolutions per minute).

If the above speed cannot be attained a minimum speed of 8 000 r/min may be used with a longer running time (8.2.4).

NOTE When the centrifugal rotational frequency (rotational speed) is 14 000 r/min, the gravitational acceleration is 22 000 G. When the centrifugal rotation speed is 8 000 r/min, the gravitational acceleration is 7 000 G.

6.2 Screw-cap centrifuge tubes, with a minimum capacity of 30 cm^3, capable of withstanding the centrifuge conditions and of being heated to a temperature greater than 110 °C.

6.3 Balance, capable of accurately weighing to ±0,1 mg.

6.4 Laboratory oven, gravity convection type, capable of controlling the temperature to ±10 °C.

6.5 Weighing container, i.e. an aluminium box or plate for weighing.

7 Conditions

Laboratory conditions shall be controlled in accordance with ISO 23529.

8 Procedure

8.1 Number of test samples

Two samples shall be taken in accordance with Clause 7 of ISO 2000.[1)]

8.2 Procedure

8.2.1 Take a test sample from a bale without milling of mass calculated at the ratio of 0,1 g per 30 cm^3 of toluene.

NOTE When the volume of a centrifuge tube is 50 cm^3, the volume of toluene is 30 cm^3 (see 8.2.3), and the sample weight is 0,1 g.

8.2.2 Cut the test sample into approximately 1 mm^3-sized pieces using clean scissors. Weigh the prepared test sample to the nearest 0,1 mg (m_0). Place the pieces in a clean centrifuge tube (6.2), which has previously been heated at 100 °C for 1 h and stored in a desiccator.

8.2.3 Add toluene to the tube until it is 60 % full. Cap it and shake by hand for a few seconds. Then, allow it to stand for 16 h to 20 h in dark conditioms without stirring at (25 ± 2) °C.

8.2.4 After this period, shake the tube up and down for 60 s to disperse the jelly-like precipitate on the bottom.

Before placing the tubes in the centrifuge machine, all tubes shall be filled up with fresh toluene, so that the volume of solution is the same in all tubes.

NOTE A centrifuge tube is usually filled up with solution to minimize the dead space created at the top of the tube because of the vacuum-pressured environment during rotation.

Place the tubes in the centrifuge machine and operate the machine at 14 000 r/min for 2 h.

If the rotating capacity of the centrifuge machine is below 14 000 r/min, operate the machine at 8 000 r/min for 6 h.

A set temperature of centrifuge is usually indicated in the operation manual for safety reasons. If not, it may be set to between 0 °C and 25 °C.

8.2.5 Remove the tube from the machine and pipette the liquid from the tube held at approximately 45°. Caution shall be taken to prevent pipetting the precipitate.

8.2.6 Add 1 ml to 3 ml of acetone to the precipitate so that it can be easily peeled from the bottom of the tube. Leave to stand for over 30 s and then pipette the liquid from the tube.

8.2.7 Peel off the precipitate remaining at the bottom of the tube with a spatula or a spatula-like stick and move it into the container (6.5) which has been cleaned and weighed to the nearest 1 mg (m_1). A limited amount of acetone may be used to rinse the spatula, if necessary.

8.2.8 Store the container in a fume cupboard for 30 min to allow the solvent to evaporate, in order to prevent flashing of the solvent during heating. Place the container containing the precipitate in the oven and dry it at the temperature of 110 °C for 1 h.

8.2.9 Take the container out of the oven and allow it to cool in a desiccator for 30 min.

8.2.10 Weigh the container containing the dry precipitate to the nearest 0,1 mg (m_2).

8.2.11 Repeat the procedure in 8.2.8, 8.2.9 and 8.2.10 until the loss in mass between two successive weighings is less than 0,2 mg. Note the period of drying time for the first test piece and use it for the next test piece for the series of testing. Record the final mass (m_2).

9 Expression of the result

To determine the gel content, use Formula (1) and round the result to one decimal place.

$$G = \frac{(m_2 - m_1)}{m_0} \times 100 \quad (1)$$

where

G is the gel content, as a percentage (%);

m_0 is the mass of the original test piece (8.2.2), in grams (g);

m_1 is the mass of the empty container (8.2.7), in grams (g);

m_2 is the mass of the container containing dry precipitate (8.2.10), in grams (g).

Take an average of two gel content values.

10 Precision

For the precision, see Annex A.

11 Test report

The test report shall include the following:

a) a reference to this International Standard, i.e. ISO 17278.

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

c) the test method;

d) the laboratory temperature;

e) the centrifuge diameter, the temperature, the speed and the running time of the test;

f) details of any operation not specified in this International Standard;

g) the results;

h) the number of test pieces tested;

i) the individual test results;

j) the mean result;

k) the details of any unusual features noted during the determination;

l) the date of the test.

Annex A
(informative)

Precision

A.1 General

An interlaboratory test programme (ITP) for the precision evaluation of the test method for TSR gel content was planned and conducted in January and February of 2011. The calculations were carried out in accordance with ISO/TR 9272. Ten laboratories from five countries participated in this ITP.

Three TSR samples with different levels of gel content were prepared for this ITP as shown in Table A.1. They were sent out to each laboratory with instructions to carry out the test as soon as the samples were received. The test was carried out on two different days at one-week intervals with two tests each day (n = 2).

The precision results as determined by this ITP should not be used for acceptance/rejection testing of any group of materials or products without documentation that the results are applicable to those particular materials or products and the specific test protocol of this test method.

Table A.1 — TSR sample

	Sample A	Sample B	Sample C
Gel content level	Low	Medium	High
NOTE Each sample should be appropriately stored in a conditioned place until all the tests are completed.			

The precision results are given in Table A.2. These results were obtained using outlier deletion procedures as described in ISO/TR 9272.

A.2 Repeatability

The repeatability, r, of the test method was established as the appropriate value tabulated in Table A.2 for each material. Two single test results that differ by more than the value shall be considered suspect, and it is recommended that some appropriate investigative action be taken.

A.3 Reproducibility

The reproducibility, R, of the test method was established as the appropriate value tabulated in Table A.2 for each material. Two single test results that differ by more than the value shall be considered suspect, and it is recommended that some appropriate investigative action be taken.

Table A.2 — Precision for gel content

Material	Mean	Within laboratory			Between laboratories			Number of laboratories
	%	s_r	r	(r)	s_R	R	(R)	
A	0,80	0,14	0,39	48,8	0,45	1,27	158,8	8
B	5,20	0,26	0,75	14,4	1,52	4,31	82,9	8
C	8,20	0,56	1,57	19,1	2,02	5,71	69,6	9

Number of replicates n = 2;

s_r is the repeatability standard deviation;

s_R is the reproducibility standard deviation;

r is the repeatability, in measurement units;

(r) is the repeatability, as a percentage (these values represent relative percentage, i.e. per cent of a percent);

R is the reproducibility, in measurement units;

(R) is the reproducibility, as a percentage (these values represent relative per cent, i.e. per cent of a percent).

Bibliography

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

ICS 83.040.10

Price based on 7 pages