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Rubber, raw natural — Determination of dirt content

Caoutchouc naturel brut — Détermination de la teneur en impuretés

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 249:1995), which has been technically revised.

This corrected version of ISO 249:2014 incorporates the following changes:

- in the key to [Figure 1](#), item c, a typo “fs” has been deleted;
- in [Table B.2](#), second cell of the header, the text has been corrected to read “Average of dirt content”.

Rubber, raw natural — Determination of dirt content

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.

1 Scope

This International Standard specifies a method for the determination of the dirt content of raw natural rubber.

It is not applicable to dirt present as surface contamination.

2 Normative references

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

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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

3 Reagents

WARNING — All recognized health and safety precautions shall be exercised during the operations of this analysis, with particular emphasis on safe handling of the flammable solvents required. All solvents shall be free from water and dirt.

During the analysis, wherever possible, use only reagents of recognized analytical grade.

3.1 Mixed xylenes, boiling range 139 °C to 141 °C.

3.2 High-aromatic hydrocarbon solvent known as white spirit, boiling range 155 °C to 198 °C, or other hydrocarbon solvents of similar boiling range.

3.3 Light petroleum, boiling range 60 °C to 80 °C or other hydrocarbon solvents of similar boiling range.

3.4 Toluene.

3.5 Rubber peptizing agents.

3.5.1 Xylyl mercaptan solution, 36 % (m/m) in mineral oil.

3.5.2 2-mercaptobenzothiazole.

3.5.3 Di-(2-benzamidophenyl) disulfide.

3.5.4 Toly mercaptan solution, 20 % (m/m) to 40 % (m/m) in mineral oil.

3.5.5 Other fully soluble rubber peptizing agent.

4 Apparatus

Ordinary laboratory equipment, and the following.

4.1 Conical flask, of capacity 250 cm³ or 500 cm³ fitted with a suitable stopper; or beaker, of capacity 250 cm³ or 500 cm³, and a clock glass of appropriate diameter as cover.

4.2 Short air condenser (optional).

4.3 Thermometer, reading to at least 200 °C.

4.4 Heater, for heating the conical flask or beaker (4.1) and its contents (see the Note to 5.3.4).

Hotplates which provide uniform heating surfaces, or infrared lamps, are recommended. Infrared lamps (250 W) can be placed in rows, with the base of the conical flask about 20 cm from the top of the lamp. Individual control of each lamp is recommended to prevent localized overheating. Alternatively, a sand bath may be used.

4.5 Sieve, of nominal size of openings 44 µm to 45 µm (325 mesh) of corrosion-resistant wire gauze, preferably stainless steel, complying with ISO 565.

4.5.1 The wire gauze shall be mounted across the end of a metal tube about 25 mm in diameter and greater than 20 mm long.

4.5.2 The sieve shall be constructed in such a way that the gauze is free from distortion and is protected from accidental damage. A suitable construction is shown in Figure 1.

4.5.3 Sieves and holders may also be constructed by removing the bottom of a metal crucible having the appropriate dimensions, and soldering the screen to the crucible. This results in an ample container for the rubber solution during filtering.

4.5.4 A coarse screen may also be soldered under the 44 µm to 45 µm (325 mesh) gauze to protect it from accidental damage. This "guard" screen shall not hinder the filtration in any way but only provide a support for the gauze.

4.5.5 Commercially available filtration apparatus having 44 µm to 45 µm (325 mesh) gauze is acceptable, provided it can be used as specified in this International Standard.

4.6 Ultrasonic equipment, for cleaning sieves (optional but desirable).

5 Procedure

5.1 Preparation of the test portion

5.1.1 Prepare a homogenized laboratory sample of 250 cm³ or 500 cm³ diameter as cover and a clock glass of appropriate raw natural rubber in accordance with ISO 1795. From the homogenized laboratory sample, take about 30 g, and pass it twice between the cold rolls of a laboratory mill, the nip being adjusted to 0,5 mm ± 0,1 mm by means of a lead strip (see ISO 2393).^[1]

5.1.2 Immediately weigh a test portion of 10 g to 20 g to the nearest 0,1 g. (For “clean” rubbers of low dirt content, a 20 g test portion is recommended. For heavily contaminated rubbers, a smaller test portion should be used.)

5.1.3 Carry out the determination in duplicate.

5.2 Preparation of the peptizer

5.2.1 If xylyl mercaptan ([3.5.1](#)) is used, use 1 g of the solution per test portion and 150 cm³ to 230 cm³ of solvent ([3.1](#) or [3.2](#)).

5.2.2 If 2-mercaptobenzothiazole ([3.5.2](#)) or di-(2- benzamidophenyl) disulfide ([3.5.3](#)) is used, use 0,5 g per test portion. Prepare a solution by dissolving 0,5 g of solid in 200 cm³ of solvent ([3.1](#) or [3.2](#)) and filtering off any insoluble material.

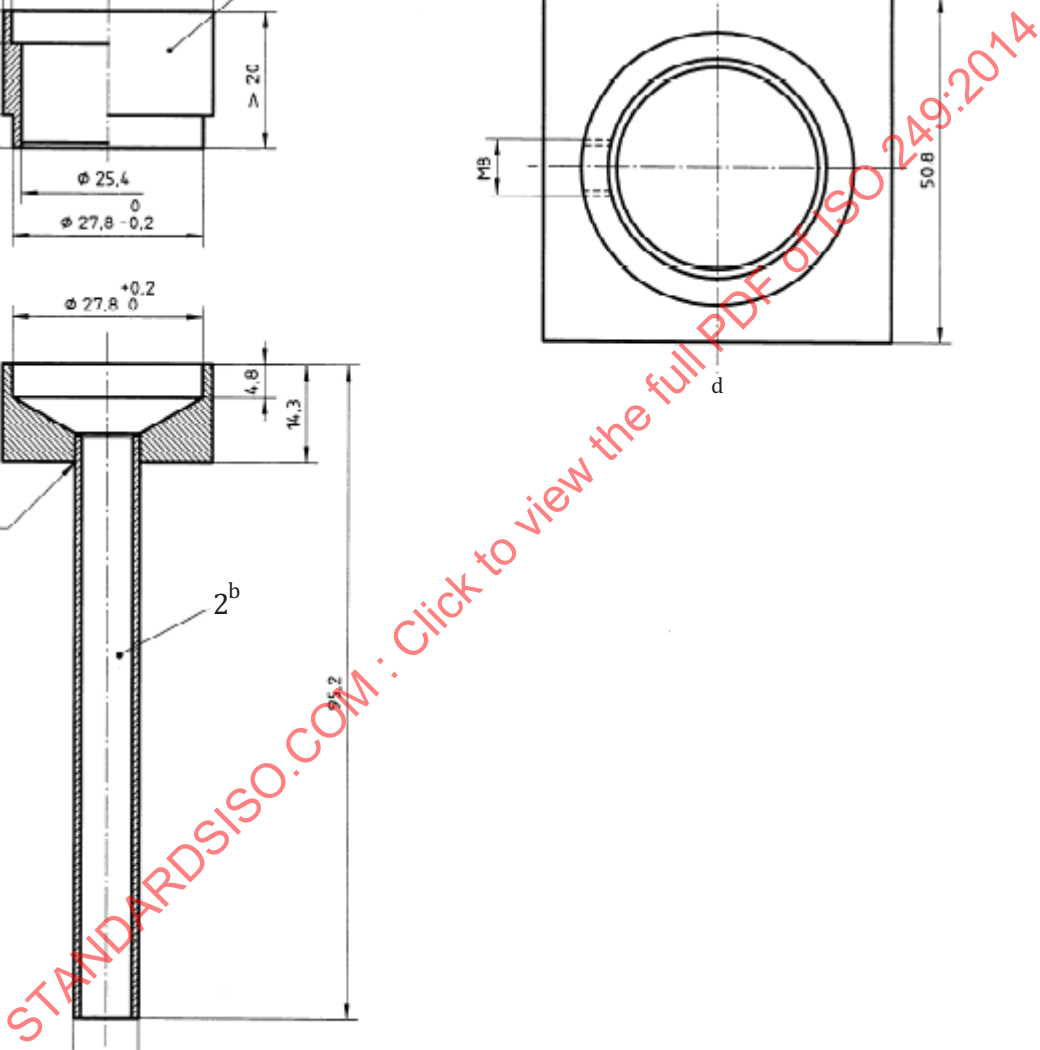
5.2.3 If tolyl mercaptan ([3.5.4](#)) is used, use 1 g to 1,5 g of the solution per test portion and 200 cm³ of solvent ([3.1](#) or [3.2](#)).

5.3 Determination

5.3.1 To the conical flask or the beaker ([4.1](#)), add solvent and peptizer according to [5.2.1](#), [5.2.2](#), and [5.2.3](#).

5.3.2 Cut the test portion into pieces, each of mass about 1 g, and drop each piece, separately, into the flask or beaker containing solvent ([5.3.1](#)).

5.3.3 Heat the flask or beaker and its contents (see [4.4](#)) at 125 °C to 130 °C until a smooth solution is obtained, or stopper the flask or cover the beaker with a clock glass and stand for several hours at room temperature before heating to 125 °C to 130 °C. A short air condenser ([4.2](#)) can be used during the heating, to reduce evaporation of the solvent.



Key

- 1 sieve (stainless sieve ring with wire gauze soldered on to it).
- 2 sieve holder (stainless steel or brass cylinder).
- a A recess of 1 mm around the inner edge of the top and the outer edge of the lower end is allowed for easy stacking of the sieves.
- b Dimensions: 30 mm external diameter, 2 mm to 3 mm wall thickness, and 3 mm height.
- c Braze on outside.
- d Sieve holder for inspection of sieve.

Figure 1 — Details of suitable sieve and holder for dirt determination

5.3.4 Agitate the flask or beaker occasionally by hand.

Boiling or overheating of the rubber solution can result in the formation of a gel-like substance which renders subsequent filtration difficult and can result in a higher apparent dirt content; hence, avoid apparatus and conditions which can cause local overheating.

5.3.5 When the rubber is completely dissolved (and the solution is adequately mobile), decant the hot solution through the sieve (4.5), which has been weighed to the nearest 0,1 mg, retaining the bulk of the dirt in the flask or beaker.

5.3.6 Wash the flask or beaker and the retained dirt with hot solvent (3.1 or 3.2) until the rubber has been completely removed. Again, retain the bulk of the dirt in the flask or beaker. (About 100 cm³ of hot solvent is normally required for effective washing.) During the later stages of the washing operation, rinse the dirt from the flask or beaker into the sieve. Loosen any dirt adhering to the flask or beaker with a glass rod, so it can be rinsed on to the sieve.

5.3.7 Remove any gelled rubber which will not pass through the sieve by one of the following methods:

- a) gently brushing the underside of the gauze with a small sable brush while hot solvent remains in the sieve;
- b) standing the sieve in a beaker containing about 10 mm depth of toluene (3.4) and gently boiling for 1 h, covering the beaker with a clock glass.

These operations should preferably be carried out under a hood.

5.3.8 Wash the sieve twice, either with light petroleum (3.3), in which case dry at 100 °C for 30 min, or with white spirit (3.2), in which case dry at 100 °C for 1 h.

5.3.9 The dirt on the sieve after drying should be loose and, apart from fibrous matter, be free-flowing. It should be readily dislodgeable from the wire gauze. If this is not so, treat the sieve with boiling toluene as in 5.3.7 item b.

5.3.10 If gelled rubber still remains, abandon the determination and carry out a repeat determination.

5.3.11 Cool the sieve and residue in a desiccator and weigh to the nearest 0,1 mg.

5.4 Care of sieves

5.4.1 At all stages, handle the sieve carefully. Inspect it after each determination to check for damage, for example under a microscope, with a slide projector (to throw an image of the gauze on a screen) or with magnifying glass (X 10). If noticeable distortion of the wire gauze has occurred, replace it with new gauze.

5.4.2 After each determination, remove loose dirt by careful brushing. Partially blocked sieves can usually be cleaned by boiling in xylene, but more effectively with ultrasonic equipment (4.6). If, in spite of this treatment, the gauze is badly blocked and the mass of the sieve has increased more than 1 mg, replace the wire gauze.

5.4.3 Sieves can be stored in warm toluene to lessen build-up of rubber.

6 Expression of results

The dirt content, expressed as a percentage by mass, of the test portion is given by the formula

$$\frac{m_1}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the dirt.

Express the result to the nearest 0,01 %.

7 Precision

See [Annex B](#).

8 Test report

The test report shall contain the following information:

- a) reference to this International Standard, i.e. ISO 249;
- b) all details necessary for identification of the sample;
- c) mean of the two results;
- d) solvent and peptizer used;
- e) any particular points observed in the course of the test;
- f) any operation not specified in this International Standard or in the International Standards to which reference is made, and any operation regarded as optional.

Annex A (informative)

Guidance for using precision results

A.1 The general procedure for using precision results is as follows, with the symbol $|x_1 - x_2|$ designating a positive difference in any two measurement values (i.e. regardless of the sign).

A.2 Enter the appropriate precision table (for any test parameter being considered) at an average value (of the measured parameter) nearest to the “test” data average under consideration. This line will give the applicable r , (r) , R or (R) for use in the decision process.

A.3 With these r and (r) values, the following general repeatability statements may be used to make decisions.

A.3.1 For an absolute difference: The difference $|x_1 - x_2|$ between two test (value) averages, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability r on average not more than once in 20 cases.

A.3.2 For a percentage difference between two test (value) averages: The percentage difference between two test values, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability (r) on average not more than once in 20 cases.

$$\left[|x_1 - x_2| / (x_1 + x_2) / 2 \right] \times 100$$

A.4 With these R and (R) values, the following general reproducibility statements may be used to make decisions.

A.4.1 For an absolute difference: The absolute difference $|x_1 - x_2|$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility R not more than once in 20 cases.

A.4.2 For a percentage difference between two test (value) averages: The percentage difference between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility (R) not more than once in 20 cases.

$$\left[|x_1 - x_2| / (x_1 + x_2) / 2 \right] \times 100$$

Annex B (informative)

Precision

B.1 General

Precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272.^[2] Consult that Technical Report for precision concepts and nomenclature. [Annex A](#) of this International Standard gives guidance on the use of repeatability and reproducibility.

An interlaboratory test programme was organized in late 2010 and 2011 by the Malaysian Rubber Board. Two separate programmes were conducted, one in March and one in September. Two types of materials were sent to each laboratory:

- a) blended samples of two rubbers “A” and “B”;
- b) unblended samples of the same two materials “A” and “B”.

For both blended and unblended samples, a test result was taken as the mean of five separate determinations.

NOTE Blended samples are samples blended before they are given to participants; unblended samples are not blended before being given to participants.

“Type 1” precision was measured in the interlaboratory test programme. The time period for repeatability and reproducibility was on a scale of days. A total of 11 laboratories participated in the programme for blended samples and a total of 10 laboratories in the programme for unblended samples.

B.2 Precision results

Precision results for the blended sample programme are given in [Table B.1](#) and the results for the unblended sample programme are in [Table B.2](#).

Table B.1 — Type 1 precision — Blended sample testing using turpentine as the solvent and using a 44 µm sieve

Rubber sample	Average of dirt content % (m/m)	Within-laboratory repeatability		Interlaboratory reproducibility	
		<i>r</i>	(<i>r</i>)	<i>R</i>	(<i>R</i>)
A	0,049 1	0,012 8	26,07	0,0369	75,15
B	0,149 4	0,019 4	12,99	0,1273	85,21
<i>r</i> = repeatability, in percent by mass (<i>r</i>) = repeatability, in percent (relative) of the average <i>R</i> = reproducibility, in percent by mass (<i>R</i>) = reproducibility, in percent (relative) of the average					