

INTERNATIONAL
STANDARD

ISO
249

Third edition
1995-12-15

**Rubber, raw natural — Determination
of dirt content**

Caoutchouc naturel brut — Détermination de la teneur en impuretés

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Reference number
ISO 249:1995(E)

Foreword

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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.

International Standard ISO 249 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 249:1987), which has been technically revised.

Annex A of this International Standard is for information only.

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Rubber, raw natural — Determination of dirt content

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.

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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 1795:1992, *Rubber, raw, natural and synthetic — Sampling and further preparative procedures*.

ISO 2393:1994, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*.

ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards*.

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 196 °C, or other hydrocarbon solvent of similar boiling range.

3.3 Light petroleum, boiling range 60 °C to 80 °C, or other hydrocarbon solvent 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 Tolylyl 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

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 **clockglass** 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 infra-red lamps, are recommended. Infra-red 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 45 µm, 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 45 µm 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 45 µm 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 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).

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 or 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 clockglass and stand for several hours at room temperature before heating to 125 °C to 130 °C. A short air condenser (4.2) may be used during the heating, to reduce evaporation of the solvent.

Dimensions in millimetres

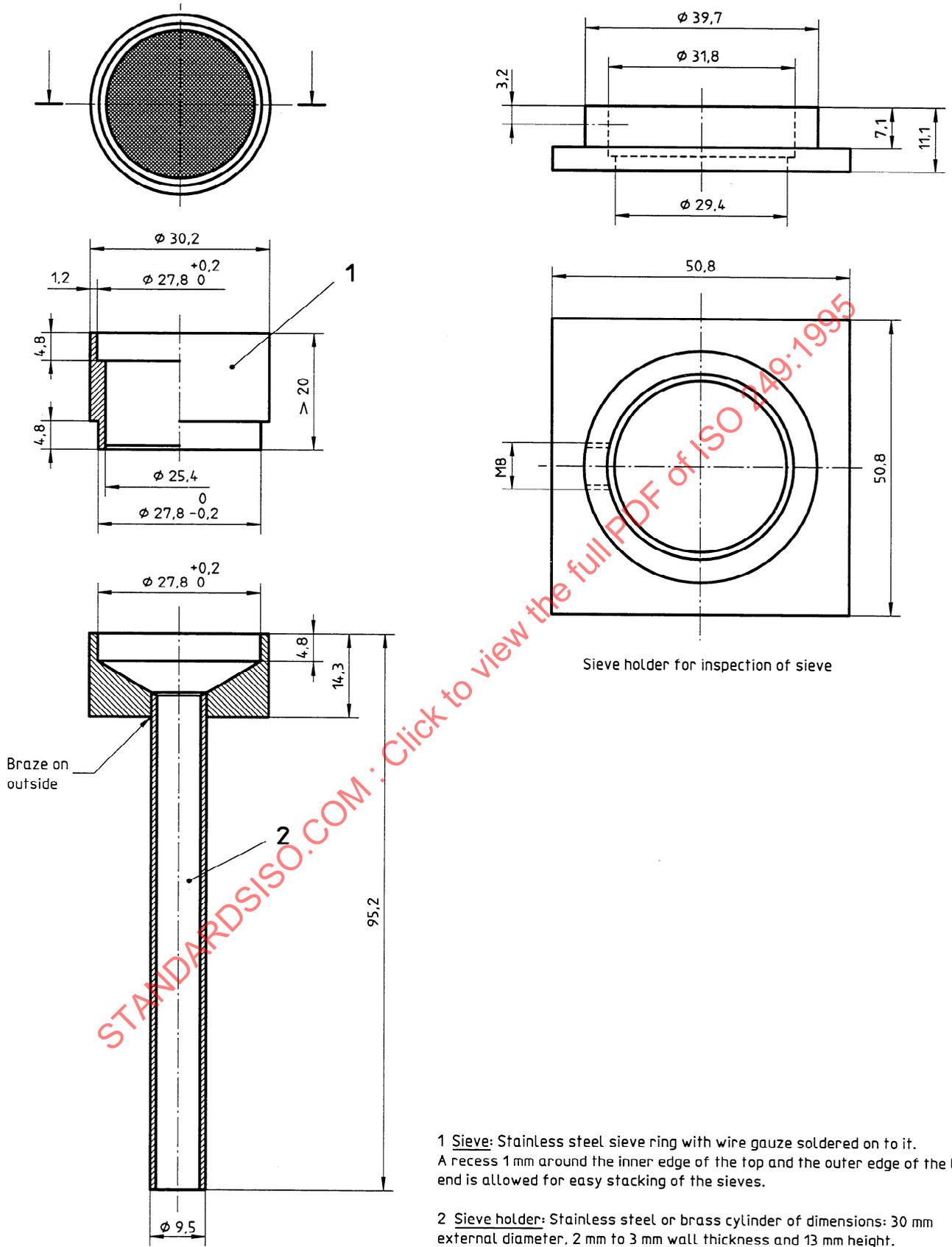


Figure 1 — Details of suitable sieve and holder for dirt determination

5.3.4 Agitate the flask or beaker occasionally by hand.

Boiling or overheating of the rubber solution may result in the formation of a gel-like substance which renders subsequent filtration difficult and may 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 clockglass.

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 dislodgable from the wire gauze. If this is not so, treat the sieve with boiling toluene as in 5.3.7 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 or with a slide projector (to throw an image of the gauze on a screen). If noticeable distortion of the wire gauze has occurred, replace it by a 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 may 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

7.1 General

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272. Consult this Technical Report for precision concepts and nomenclature. Annex A of this International Standard gives guidance on the use of repeatability and reproducibility.

7.2 Precision details

7.2.1 An interlaboratory test programme was organized in late 1984 by the Rubber Research Institute of Malaysia. Two separate programmes were conducted, one in March and one in July. Two types of material were sent to each laboratory:

- a) blended samples of two rubbers "A" and "B";

b) unblended (normal) samples of the same two materials "A" and "B".

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

7.2.3 A "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 14 laboratories participated in the programme for blended samples and a total of 13 laboratories in the programme for unblended samples.

7.3 Precision results

The precision results for the blended-sample programme are given in table 1 and the results for the unblended-sample programme in table 2.

8 Test report

The test report shall contain the following information:

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

Table 1 — Type 1 precision — Blended-sample testing

Rubber sample	Average dirt content % (m/m)	Within-laboratory repeatability		Interlaboratory reproducibility	
		<i>r</i>	(<i>r</i>)	<i>R</i>	(<i>R</i>)
A	0,11	0,018 5	16,4	0,031	27,0
B	0,16	0,038 5	24,4	0,065	40,9
Pooled values	0,14	0,031	22,4	0,051	37,1

r = repeatability, in percent by mass
(*r*) = repeatability, in percent (relative) of the average
R = reproducibility, in percent by mass
(*R*) = reproducibility, in percent (relative) of the average

Table 2 — Type 1 precision — Unblended-sample testing

Rubber sample	Average dirt content % (m/m)	Within-laboratory repeatability		Interlaboratory reproducibility	
		<i>r</i>	(<i>r</i>)	<i>R</i>	(<i>R</i>)
A	0,04	0,013	31,5	0,035	86,2
B	0,04	0,017	39,3	0,029	67,7
Pooled values	0,04	0,015	35,8	0,032	77,1

See table 1 for symbol definitions.

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. without regard to sign).

A.2 Enter the appropriate precision table (for whatever test parameter is 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

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

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.

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

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

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.

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