INTERNATIONAL STANDARD

ISO 249

Fourth edition
2014-08-01

Corrected version
2015-09-15

Rubber, raw natural — Determination of dirt content

Caoutchouc naturel brut — Détermination de la teneur en impuretés

Reference number
ISO 249:2014(E)

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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 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 Tolyl 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 cm^3 or 500 cm^3 fitted with a suitable stopper; or beaker, of capacity 250 cm^3 or 500 cm^3, 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 cm_3 or 500 cm_3 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 cm^3 to 230 cm^3 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 cm^3 of solvent (3.1 or 3.2) and filtering off any insoluble material.

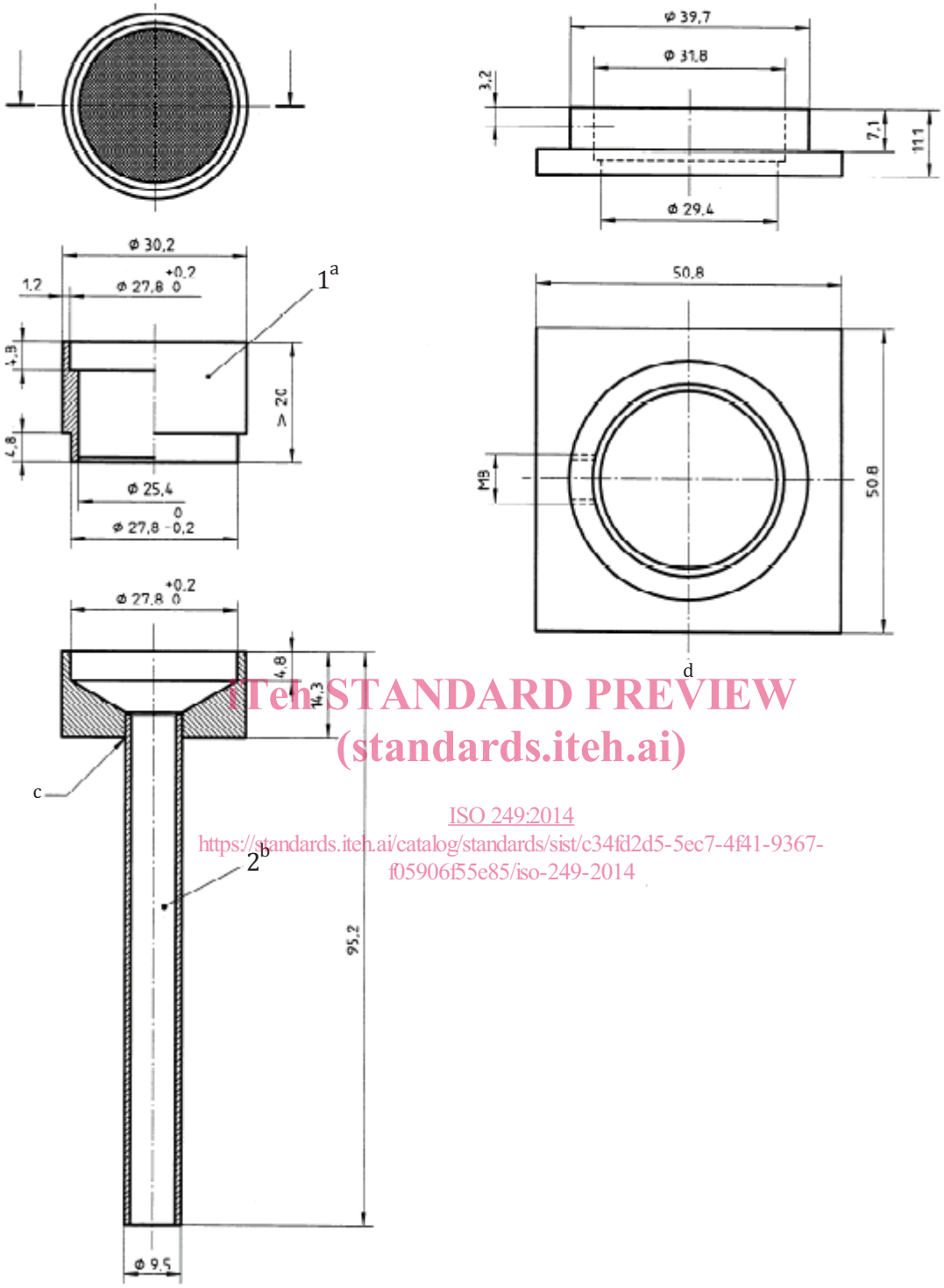
5.2.3 If tolyl mercaptan (3.5.4) is used, use 1 g to 1,5 g of the solution per test portion and 200 cm^3 of solvent (3.1 or 3.2).

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 cm^3 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.