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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Rubber, raw natural — Determination of dirt content

Caoutchouc naturel brut — Détermination de la teneur en impuretés

Reference number
ISO 249 : 1987 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

This second edition cancels and replaces the first edition (ISO 249 : 1974), of which it constitutes a technical revision. It specifies more closely the rubber solvents and gives suitable peptizing agents for the rubber. In addition, specifications for the dimensions of a suitable sieve and sieve holder have been included.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Rubber, raw natural — Determination of dirt content

1 Scope and field of application

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 References

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

ISO 1795, *Raw rubber in bales — Sampling.*

ISO 1796, *Rubber, raw — Sample preparation.*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and 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 to 141 °C.

3.2 High aromatic hydrocarbon solvent known as white spirit, boiling range 155 to 196 °C, or other hydrocarbon solvent of similar boiling range.

3.3 Light petroleum, boiling range 60 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 Tolyl mercaptan solution, 20 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 or 500 cm^3, fitted with a suitable stopper; or **beaker**, of capacity 250 or 500 cm^3, 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 sandbath 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 the figure.

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 should 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 and ISO 1796. 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 ± 0,1 mm by means of a lead strip (see ISO 2393).

5.1.2 Immediately weigh a test portion of 10 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, the 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 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 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 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 120 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 to 130 °C. A short air condenser (4.2) may be used during the heating, to reduce evaporation of the solvent.

5.3.4 Agitate the flask or beaker occasionally by hand.

NOTE — 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 apparatus and conditions which can cause local overheating should be avoided.

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 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 dislodgeable from the wire gauze. If this is not so, the sieve shall be treated with boiling toluene as in 5.3.7 b).

5.3.10 If gelled rubber still remains, the determination shall be abandoned and a repeat determination carried out.

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, the sieve shall be handled carefully. It shall be inspected 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, it shall be discarded and replaced 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 by ultrasonic methods. If, in spite of this treatment, the gauze is badly blocked and the mass of the sieve has increased more than 1 mg, the wire gauze shall be replaced.

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 Test report

The test report shall contain the following information:

a) reference to this International Standard;

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

c) the mean of the two results;

d) any particular points observed in the course of the test;

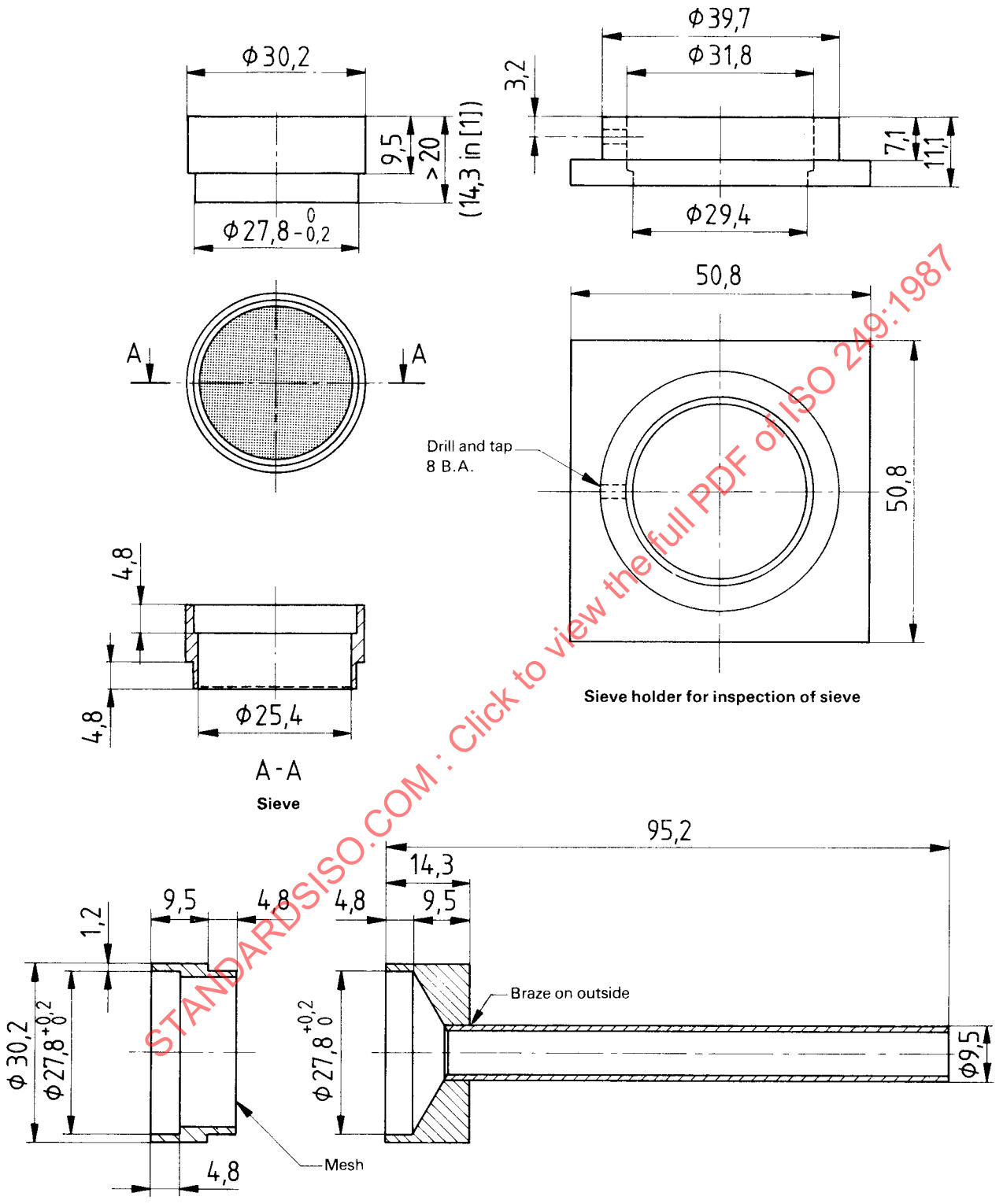
e) the solvent and peptizer used;

f) any operations not specified in this International Standard, or regarded as optional, which might have affected the results.

8 Bibliography

[1] R.R.I.M. Test Methods for Standard Malaysian Rubbers. *SMR Bulletin*, No. 7.

Dimensions in millimetres



Sieve: Stainless steel sieve ring with wire gauze soldered on to it. A recess 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.

Sieve holder: Stainless steel or brass cylinder of dimensions: 30 mm external diameter, 2 to 3 mm wall thickness and 13 mm height.

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