INTERNATIONAL STANDARD 249

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Raw natural rubber — Determination of dirt

Caoutchouc naturel brut — Détermination de la teneur en impuretés

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Raw natural rubber — Determination of dirt

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of dirt contained in raw natural rubber. It is not applicable to dirt present as surface contamination.

2 REFERENCE

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

ISO 1796, *Raw rubber — Sample preparation.*

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

3 REAGENTS

3.1 Xylene or **white spirit.**

3.2 Petroleum spirit, boiling range 60 to 80 °C.

3.3 Rubber peptising agent, such as xylyl mercaptan.

3.4 Toluene

4 APPARATUS

4.1 Conical flask, stoppered, capacity 250 to 500 ml.

4.2 Short air condenser (optional).

4.3 Thermometer, reading to 150 °C.

4.4 Means for heating the conical flask and its contents (see note in 5.2).

4.5 Sieve, nominal aperture 45 μm, of non-corrodible wire gauze, preferably stainless steel, complying with ISO 565, *Test sieves — Woven metal wire cloth and perforated plates — Nominal sizes of apertures.*

The wire gauze shall be mounted across the end of a metal tube about 25 mm diameter and 10 to 30 mm long. The sieve shall be constructed in such a way that the gauze is free from distortion and is protected from accidental damage (see the figure).

5 PROCEDURE

5.1 Preparation of the test portion

Prepare a homogenized piece in accordance with ISO 1795 and ISO 1796. From the homogenized piece take about 30 g and pass it twice between the cold rolls of a laboratory mill, the nip being adjusted at 0,5 ± 0,1 mm by means of a lead strip (see ISO 2393).

Immediately weigh a test portion of 10 to 20 g. (For "clean" rubbers of low dirt content, a 20 g test portion is recommended. For heavily contaminated rubbers, a smaller test portion is preferable.)

5.2 Determination

Cut the test portion into pieces each weighing about 1 g, and drop each piece separately into 150 to 250 ml of xylene or white spirit (3.1) contained in a conical flask (4.1). Add 1 g of the rubber peptising agent (3.3).

Heat the flask and its contents at 125 to 130 °C until a smooth solution is obtained, or stopper the flask and stand for several hours at room temperature before heating at 125 to 130 °C. A short air condenser may be fitted during the heating if desired.

It is advantageous to agitate the flask occasionally during the standing and heating periods.

NOTE — To minimize the formation of gel and consequent filtration difficulties, apparatus and conditions which can cause local overheating should not be used; heating by infra-red lamps is recommended.

When the rubber is completely dissolved, decant the solution through the sieve, which has been weighed to the nearest 0,1 mg, retaining the bulk of the dirt in the conical flask. Wash the flask and the retained dirt with hot rubber solvent until the rubber has been completely removed, again retaining the bulk of the dirt in the conical flask. About 100 ml of hot solvent are normally required for effective washing. During the later stages of the washing operation, rinse the dirt from the flask into the sieve. Loosen any dirt adhering to the flask with a glass rod.

It is essential to remove any gelled rubber which will not pass through the sieve. This may be accomplished by either

a) gently brushing the underside of the gauze with a small sable brush while hot solvent remains in the sieve, or

b) standing the sieve in a beaker containing about 10 mm depth of toluene (3.4) and gently boiling for 1 h.

Wash the sieve twice with petroleum spirit (3.2) and dry at about 100 °C for 30 min.

The dirt on the sieve after drying should be loose, and apart from fibrous matter, free-flowing. It should be readily dislodged from the filter cloth. If this is not so, the sieve shall be treated with boiling toluene. If gelled rubber still remains, the determination shall be abandoned and a repeat determination carried out.

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

At all stages the sieve shall be handled carefully; it shall be inspected after each determination under a microscope. If noticeable distortion of the wire cloth has occurred, it shall be discarded and replaced by a new one.

After each determination, remove loose dirt by carefully 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 cloth shall be replaced.

6 EXPRESSION OF RESULTS

Calculate the dirt content, as a percentage by mass of the dry rubber, from the following 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.

7 TEST REPORT

The test report shall include the following particulars :

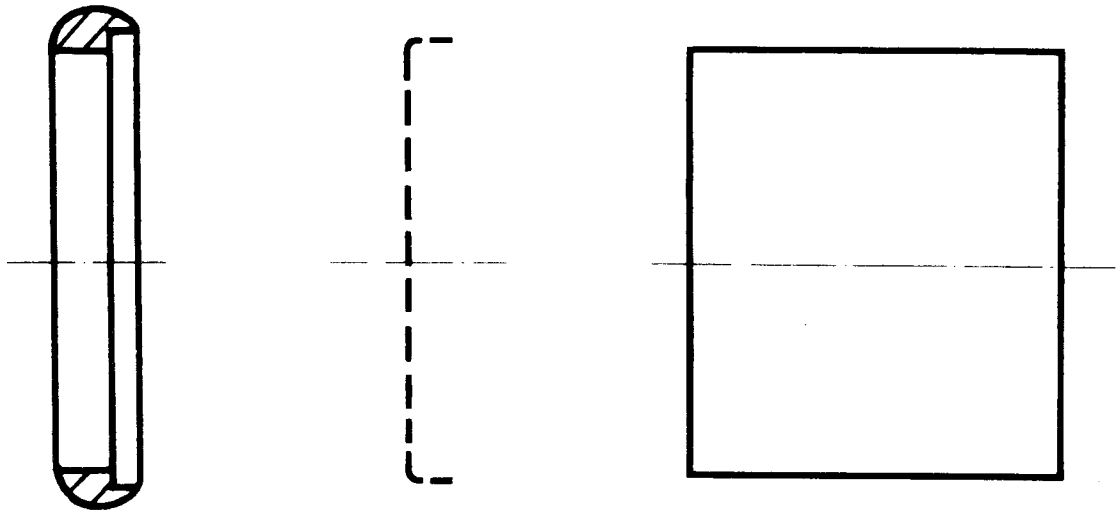
a) reference to this International Standard;

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

c) the results, and the form in which they are expressed;

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

e) any operations not specified, or optional, which might affect the results.



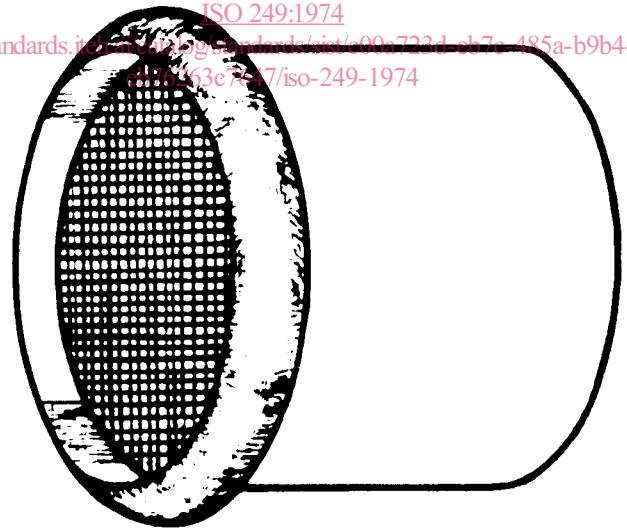


FIGURE — **Details of sieve construction**