UDC 678.4.032 : 543.869

Ref. No. : ISO/R 249-1971 (E)

Translated

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 249

RAW NATURAL RUBBER

DETERMINATION OF DIRT

2nd EDITION

May 1971

This second edition supersedes the first edition

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 249, *Determination of dirt in raw natural rubber*, was drawn up by Technical Committee ISO/TC 45 *Rubber*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 342, which was circulated to all the ISO Member Bodies for inquiry in December 1959. It was approved, subject to a few modifications of an editorial nature, by 22 Member Bodies. One Member Body opposed the approval of the Draft (Italy).

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

BRIEF HISTORY RELATIVE TO THE 2nd EDITION

During the 13th meeting of ISO/TC 45 held at Budapest in 1965, it was proposed to revise ISO Recommendation R 249 in view of some of its weaknesses. Draft ISO Recommendation No. 1406 was adopted to this effect, and it was circulated to the Members of ISO/TC 45 and to all the ISO Member Bodies for enquiry in July 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Germany	Poland
Austria	Hungary	Sweden
Brazil	India	Switzerland
Canada	Iran	Thailand
Ceylon	Israel	U.A.R.
Colombia	Italy	United Kingdom
Czechoslovakia	Netherlands	U.S.A.
France	New Zealand	U.S.S.R.

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as the second edition of ISO Recommendation R 249, under the modified title : *Raw natural rubber – Determination of dirt.*

This second edition cancels and replaces the first edition of ISO Recommendation R 249.

ISO Recommendation R 249 May 1971

RAW NATURAL RUBBER

DETERMINATION OF DIRT

1. SCOPE

This ISO Recommendation describes a method for the determination of dirt contained in raw natural rubber; it is not applicable to dirt present as surface contamination. In order that the method will give a satisfactory result the raw rubber should be sampled in accordance with ISO Recommendation R 250, *Sampling of raw natural rubber.*

2. REAGENTS

2.1 *Xylene* or *white spirit.*

2.2 *Petroleum spirit*, boiling range 60 to 80 °C.

2.3 *Rubber peptising agent*, such as xylyl mercaptan.

3. APPARATUS

3.1 *Conical flask*, stoppered, capacity 250 to 500 ml.

3.2 *Short air condenser* (optional).

3.3 *Thermometer*, reading to 150 °C.

3.4 *Means for heating the conical flask and its contents* (see Note in clause 4.2).

3.5 *Sieve*, nominal aperture 45 μm, of non-corrodible wire gauze, preferably stainless steel, complying with ISO Recommendation R 565, *Woven wire cloth and perforated plates in test sieves – Nominal sizes of apertures.*

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

4. PROCEDURE

4.1 Preparation of the test sample

Weigh the piece of rubber from the sample bale, or the strips from the loose sheets, to the nearest 0.1 g. Set the nip of a laboratory mill to 1.25 mm and the roll temperature to 70 ± 5 °C and homogenize the rubber by passing it ten times between the rolls, which should preferably be running at an even speed, but may have a friction ratio not exceeding 1.4 : 1. As the rubber passes through the mill, roll it into a cylinder and present it endways to the mill for the next pass. Collect any solid matter parting from the mass and re-incorporate it in the rubber. Cool the rubber and weigh again to the nearest 0.1 g.

Record the mass of the rubber before and after milling. These values are required later for the calculation of volatile matter. Store the rubber in an airtight container unless further testing can be carried out immediately.

4.2 Determination

From the homogenized test sample take about 30 g and pass it twice between the cold rolls of a laboratory mill, the nip of the roll being set at 0.5 ± 0.1 mm by means of a lead strip.

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.)

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 (2.1) contained in a conical flask (3.1). Add 1 g of the rubber peptising agent (2.3).

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 and gently boiling for 1 hour.

Wash the sieve twice with petroleum spirit (2.2) and dry at about 100 °C for 30 minutes.

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 should be treated with boiling toluene. If gelled rubber still remains, the determination should 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 should be handled carefully, it should be inspected after each determination under a microscope. If noticeable distortion of the wire cloth has occurred it should be discarded and replaced by a new one.