INTERNATIONAL
STANDARD

ISO
6225-2

Second edition
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Rubber, raw, natural — Determination of castor oil content —

Part 2 :
Determination of total ricinoleic acid content by gas chromatography

Caoutchouc naturel brut — Détermination de la teneur en huile de ricin —

Partie 2 : Détermination de la teneur en acide ricinoléique total par chromatographie en phase gazeuse

Reference number
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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 6225-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 6225-2 : 1983), of which it constitutes a minor revision.

ISO 6225 consists of the following parts, under the general title *Rubber, raw, natural — Determination of castor oil content*:

- *Part 1: Determination of castor oil glycerides content — Thin layer chromatographic method*
- *Part 2: Determination of total ricinoleic acid content by gas chromatography*

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Introduction

Certain grades of natural rubber are treated with castor oil to facilitate crumbing of the rubber during production. ISO 6225 is intended to facilitate estimation of the amount of castor oil remaining in the rubber.

The principal constituent of castor oil, making up about 80 % (m/m) to 85 % (m/m), is the triglyceride of ricinoleic acid. This glyceride may partly hydrolyse to ricinoleic acid and glycerol. Provided the rubber is stored under normal conditions, determination of the castor oil glycerides content will give a good indication of the amount of castor oil added to the rubber.

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Rubber, raw, natural — Determination of castor oil content —

Part 2 :

Determination of total ricinoleic acid content by gas chromatography

1 Scope

This part of ISO 6225 specifies a gas chromatographic method for the determination of the total ricinoleic acid content of raw rubber. The result may be expressed in terms of ricinoleic acid or of castor oil glycerides.

It is applicable to all grades of natural rubber.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 6225. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 6225 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 1795 : 1974, *Raw rubber in bales — Sampling.*

ISO 3696 : 1987, *Water for analytical laboratory use — Specification and test methods.*

3 Principle

Extraction of any free ricinoleic acid and conversion to the acetate of methyl ricinoleate.

Extraction of any castor oil glycerides also present, hydrolysis to ricinoleic acid, and conversion to the corresponding acetate of methyl ricinoleate.

Determination of the total acetate of methyl ricinoleate by gas chromatography by comparison either with a ricinoleic acid standard, or with ricinoleic acid prepared by hydrolysis of a castor oil standard.

4 Reagents

All recognized health and safety precautions shall be taken when carrying out the procedure specified in this part of ISO 6225.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water (grade 3 as defined in ISO 3696) or water of equivalent purity.

4.1 Potassium hydroxide, ethanolic solution.

Carefully dissolve 65 g of potassium hydroxide (KOH) in 1 dm^3 of 95 % (V/V) ethanol.

4.2 Sodium chloride, solution.

Dissolve 10 g of sodium chloride in 100 cm^3 of hot water.

4.3 Hydrochloric acid, ϱ = 1,19 Mg/m^3.

4.4 Dichloromethane.

4.5 Toluene.

4.6 Sulfuric acid, methanolic solution.

Carefully mix 4 g of sulfuric acid, ϱ = 1,84 Mg/m^3, with 100 cm^3 of methanol.

4.7 Pyridine/acetic anhydride solution.

Carefully mix equal volumes of acetic anhydride (minimum purity 97 %) and pyridine (boiling range 113 °C to 117 °C).

4.8 Castor oil reference solution.

Weigh, to the nearest 0,1 mg, 0,05 g to 0,10 g of castor oil (pharmaceutical grades have been found satisfactory) and place it in a flask containing 70 cm^3 of the ethanolic potassium hydroxide solution (4.1).

Reflux for 6 h, using a condenser (5.2) fitted with a carbon dioxide absorbing guard tube at the upper end.

4.9 Ricinoleic acid reference solution.

Weigh, to the nearest 0,1 mg, 0,05 g to 0,10 g of ricinoleic acid (practical or technical grade has been found satisfactory) and dissolve it in 25 cm^3 of the methanolic sulfuric acid solution (4.6) in a flask.

Reflux the solution for 2 h, using a condenser (5.2) fitted with a moisture absorbing guard tube at the upper end.

5 Apparatus

Usual laboratory equipment, and:

5.1 Water-bath.

5.2 Reflux condensers, fitted with guard tubes to absorb carbon dioxide or moisture.

5.3 Gas chromatograph, equipped with dual flame-ionization detectors.

For optimum performance, the chromatograph shall be operated by a competent person in accordance with the manufacturer's instructions.

5.4 Gas chromatographic columns.

Different types of gas chromatographic columns may be used provided that there is a sharp separation of the acetate of methyl ricinoleate from other components.

5.4.1 Polar column.

Stainless steel, 2,5 m long, internal diameter 4 mm, packed with 10 % (*m*/*m*) Carbowax 20 M[1)] on Chromosorb AW-HMDS[1)].

5.4.2 Non-polar column.

Stainless steel, 2 m long, internal diameter 4 mm, packed with 10 % (*m*/*m*) silicone rubber (SE 30[1)]) on Chromosorb AW-HMDS[1)].

5.5 Balance, accurate to 0,1 mg.

5.6 Round-bottom flasks, of capacity 50 cm^3.

6 Preparation of test sample

Take a piece of rubber, in accordance with ISO 1795, weighing at least 12 g, from the bale, and pass it once between the tightly closed rolls of a laboratory mill to give a thin sheet. Avoid extensive milling to minimize loss of castor oil. If a thin sheet cannot be obtained, use the lace or crumbs as the test sample.

If the bale appears non-uniform, select a sufficient number of pieces, each of at least 12 g, to provide adequate representation. Prepare and analyse each piece separately, making sure that cross-contamination does not occur during the preparation.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,1 mg, 10 g ± 0,1 g of the thinly milled test sample and cut it into small pieces. Place the pieces in a flask containing 70 cm^3 of the ethanolic potassium hydroxide solution (4.1). Swirl occasionally, while adding the rubber, to keep the pieces separated.

7.2 Determination

7.2.1 Reflux for 6 h, using a condenser (5.2) fitted with a carbon dioxide absorbing guard tube at the upper end.

7.2.2 At the end of this period, remove the guard tube and pour a few cubic centimetres of methanol through the condenser into the flask. Remove the flask from the heat source, cool, and decant the extract from the flask into a porcelain evaporating dish. Keep the rubber in the flask.

7.2.3 Replace the flask and rubber on the reflux stand, connect the condenser and pour 50 cm^3 of water through the condenser into the flask.

Reflux for 30 min and transfer the cooled extract to the same evaporating dish.

7.2.4 Repeat the operation described in 7.2.3, combine the extracts in the same evaporating dish and discard the rubber. Concentrate the solutions on the water-bath (5.1) until the volume is approximately 30 cm^3.

7.2.5 Transfer the solution to a separating funnel and wash the dish several times with water. Add the washings to the contents of the separating funnel.

Acidify the aqueous solution with the hydrochloric acid solution (4.3) and extract with three 25 cm^3 portions of the dichloromethane (4.4). Combine the dichloromethane washings in another separating funnel.

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1) Carbowax 20 M, Chromosorb AW-HMDS and SE 30 are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 6225 and does not constitute an endorsement by ISO of these products.