International Standard 6225/2

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Rubber, raw, natural — Determination of castor oil content — Part 2 : Determination of total ricinoleic acid content — Gas chromatographic method

Caoutchouc naturel brut — Détermination de la teneur en huile de ricin — Partie 2 : Détermination de la teneur en acide ricinoléique total — Méthode par chromatographie en phase gazeuse.

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Foreword

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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.

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It has been approved by the members bodies of the following countries :

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INTERNATIONAL STANDARD **ISO 6225/2-1983 (E)**

Rubber, raw, natural — Determination of castor oil content — Part 2 : Determination of total ricinoleic acid content — Gas chromatographic method

0 Introduction

Certain grades of natural rubber are treated with castor oil to facilitate crumbing of the rubber during production. This part of 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 to 85 % by mass, 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.

ISO 6225/1 specifies a method for the determination of the castor oil and castor oil glycerides content of raw rubber.

1 Scope and field of application

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 References

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

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

ISO 6225/1, *Rubber, raw, natural — Determination of castor oil content — Part 1 : Determination of castor oil and castor oil glycerides content — Thin layer chromatographic method.*[1]

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 International Standard.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water 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 to 117 °C).

4.8 Castor oil reference solution.

Weigh, to the nearest 0,1 mg, 0,05 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).

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[1] At present at the stage of draft.

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

5 Apparatus1)

Usual laboratory equipment, and :

5.1 Water-bath.

5.2 Reflux condensers, fitted with guard tubes to absorb carbon dioxide and 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 on Chromosorb AW-HMDS.

5.4.2 Non-polar column.

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

5.5 Balance, accurate to 0,000 1 g.

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 and ISO 1796, 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.

NOTE — As some castor oil may be lost when rubber is extensively milled, the homogenization procedure described in ISO 1796 should not be used.

7 Procedure

7.1 Test portion

Accurately weigh 10 ± 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 transfer the contents of 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 a water-bath 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.

Wash the dichloromethane three times with 25 cm^3 of the sodium chloride solution (4.2) and discard the aqueous layer.

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1) The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm^3), in accordance with a decision of the Twelfth Conférence Générale des Poids et Mesures. The term millilitre is acceptable, in general, for references in International Standards to capacities of volumetric glassware and to liquid volumes.

Evaporate the dichloromethane on a water-bath and dissolve the residue in 25 cm^3 of the methanolic sulfuric acid solution (4.6). Reflux the resulting solution for 2 h, using a condenser fitted with a moisture absorbing guard tube at the upper end.

7.2.6 Cool the flask, remove the guard tube and pass 100 cm^3 of hot water through the condenser into the flask. Transfer the solution to a separating funnel. Wash the flask with small portions of dichloromethane and combine all the washings in the separating funnel.

Extract the aqueous solution with four 25 cm^3 portions of the dichloromethane and, if the aqueous layer is still turbid, repeat the extraction with three 25 cm^3 portions of dichloromethane.

Wash the combined dichloromethane solutions with 100 cm^3 of the sodium chloride solution (4.2).

Transfer the combined dichloromethane solution to a conical flask and evaporate all the dichloromethane.

7.2.7 Dissolve the residue in 2 cm^3 of the pyridine/acetic anhydride solution (4.7).

Reflux the solution in a 50 cm^3 round bottom flask (5.6) for 3 h, using a condenser fitted with a moisture absorbing guard tube at the upper end.

7.2.8 At the end of this period, remove the guard tube, allow the flask to cool and pour 25 cm^3 of hot water through the condenser into the flask. Reflux for 10 min.

7.2.9 Cool the flask, transfer the aqueous solution to a separating funnel and wash the flask with a few cubic centimetres of dichloromethane.

Add the washings to the contents of the separating funnel.

Extract the aqueous solution with four 25 cm^3 portions of dichloromethane and combine the washings in a separating funnel.

Wash the dichloromethane solution with 100 cm^3 of the sodium chloride solution (4.2).

Transfer the washed dichloromethane solution to a beaker and evaporate until the volume of the solution is reduced to about 2 cm^3.

7.2.10 Transfer the dichloromethane solution to a 10 cm^3 volumetric flask with the toluene (4.5) and make up to the mark with the toluene. (This is the test solution.)

7.2.11 Set the gas chromatograph to appropriate operating conditions. Column and inlet port temperatures of around 200 °C are likely to be optimal. Inject a suitable quantity of the test solution (7.2.10) into the gas chromatograph (5.3) and record the chromatogram. Measure the area of the peak for the acetate of methyl ricinoleate (A_X).

7.2.12 If the castor oil content is to be determined, treat the castor oil reference solution (4.8) as described in 7.2.2 to 7.2.10.

If the total ricinoleic acid content is to be determined, treat the ricinoleic acid reference solution (4.9) as described in 7.2.6 to 7.2.10.

Using the same quantity of the treated reference solution as was used for the determination (see 7.2.11), inject the reference solution into the chromatograph and record the chromatogram. Measure the area of the peak for the acetate of methyl ricinoleate (A_R).

8 Expression of results

The total ricinoleic acid or castor oil content, expressed as a percentage by mass, is given by the formula

$$\frac{m_R \times A_X}{m_X \times A_R} \times 100$$

where

m_R is the mass, in grams, of ricinoleic acid or castor oil in the reference solution;

m_X is the mass, in grams, of the test portion;

A_R is the area of the reference peak;

A_X is the area of the test portion peak.

NOTE — If the castor oil used in the reference solution is different from that used in the rubber, the castor oil content value may not be accurate.

Express the result to the nearest 0,05 % (m/m).

9 Test report

The test report shall include the following information :

a) a reference to this part of ISO 6225;

b) identification of the sample;

c) the results obtained;

d) any unusual features which may have affected the results;

e) the date of test.