INTERNATIONAL STANDARD

ISO 6225-1

Second edition
2006-10-01

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

Part 1: Determination of castor oil glyceride content by thin-layer chromatography

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

Partie 1: Détermination de la teneur en glycérides d'huile de ricin par chromatographie en couche mince

Reference number
ISO 6225-1:2006(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6225-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 6225-1:1984), which has been revised to update the normative references.

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 glyceride content by thin-layer chromatography*

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

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.

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

Part 1: Determination of castor oil glyceride content by thin-layer chromatography

WARNING — Persons using this part of ISO 6225 should be familiar with normal laboratory practice. This part of ISO 6225 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 6225 specifies a thin-layer chromatographic method for the determination of the castor oil and castor oil glyceride content of raw rubber.

It is applicable to all grades of natural rubber.

The lower limit of detection is approximately 0,05 % of castor oil glycerides.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1407:1992, *Rubber — Determination of solvent extract*

3 Principle

A test portion of rubber is extracted with acetone and the castor oil glycerides separated from the other extracted compounds by thin-layer chromatography. The castor oil glyceride spots are developed with phosphomolybdic acid or anisaldehyde and evaluated visually or spectrometrically.

4 Reagents

During the analysis, except where otherwise specified, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Silica gel, TLC grade.

4.2 Developing solvent.

Prepare a mixture of light petroleum (boiling range 40 °C to 60 °C), diethyl ether and glacial acetic acid, in the proportions, by volume, of 50:50:1, respectively.

4.3 Spray reagents.

4.3.1 Phosphomolybdic acid, ethanolic solution.

Dissolve 15 g of phosphomolybdic acid in 100 cm^3 of 95 % (by volume) ethanol.

4.3.2 Anisaldehyde, solution.

Mix 10 cm^3 of ethanol, 0,5 cm^3 of sulfuric acid (ρ = 1,84 Mg/m^3) and 0,5 cm^3 of anisaldehyde.

4.4 Solvents.

4.4.1 Acetone, redistilled.

4.4.2 Dichloromethane.

4.5 Standard castor oil solutions.

4.5.1 Weigh accurately 0,5 g ± 0,01 g of castor oil (pharmaceutical grades have been found satisfactory) and prepare a stock solution by diluting to 100 cm^3 with dichloromethane (4.4.2) in a one-mark volumetric flask (5.8).

4.5.2 Dilute 2 cm^3; 4 cm^3; 6 cm^3; 8 cm^3 and 10 cm^3 aliquot portions of the stock solution (4.5.1) to 10 cm^3 with dichloromethane (4.4.2) in one-mark volumetric flasks to give solutions corresponding to 0,2 %; 0,4 %; 0,6 %; 0,8 % and 1,0 % (by mass) of castor oil, based on the rubber, when 5 g of rubber is taken for analysis.

5 Apparatus[1)]

Ordinary laboratory equipment, plus the following:

5.1 Extraction unit, all-glass (see Figure 1, 2 or 3 in ISO 1407:1992).

5.2 Water-bath or **electric heating mantle**.

5.3 Thin-layer chromatography (TLC) plate, of dimensions 200 mm × 200 mm, coated with a layer approximately 0,25 mm thick of silica gel (4.1). Commercial plates may also be used.

5.4 Thin-layer applicator.

5.5 Developing tank, of sufficient size to hold the TLC plate (5.3).

5.6 Spray apparatus, for the spray reagent (4.3).

5.7 Oven, capable of being maintained at 100 °C ± 5 °C.

5.8 One-mark volumetric flasks, of capacity 5 cm^3; 10 cm^3 and 100 cm^3.

5.9 Spectrometer, capable of accurate measurement (±1 % of total absorbance at 700 nm ± 1 nm), equipped with cells of optical path length 10 mm.

Operate the spectrometer in accordance with the manufacturer's instructions for optimum performance.

---

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. Apparatus with either type of marking is satisfactory for use with this part of ISO 6225.