



SLOVENSKI STANDARD
oSIST prEN ISO 17059:2018
01-oktober-2018

Oljnice - Ekstrakcija olja in priprava metil estrov iz trigliceridnih maščobnih kislin za analizo s plinsko kromatografijo (hitra metoda) (ISO/DIS 17059:2018)

Oilseeds - Extraction of oil and preparation of methyl esters of triglyceride fatty acids for analysis by gas chromatography (Rapid method) (ISO/DIS 17059:2018)

Ölsamen - Extraktion von Öl und Herstellung von Methylester aus den Fettsäuren der Triglyceride für die Analyse durch Gaschromatographie (Schnellverfahren) (ISO/DIS 17059:2018)

Graines oléagineuses - Extraction de l'huile et préparation des esters méthyliques d'acides gras de triglycérides pour analyse par chromatographie en phase gazeuse (Méthode rapide) (ISO/DIS 17059:2018)

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67.200.20 Oljnice Oilseeds

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Oilseeds — Extraction of oil and preparation of methyl esters of triglyceride fatty acids for analysis by gas chromatography (Rapid method)

Graines oléagineuses — Extraction de l'huile et préparation des esters méthyliques d'acides gras de triglycérides pour analyse par chromatographie en phase gazeuse (Méthode rapide)

ICS: 67.200.20

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34/SC 2, Food products, Subcommittee SC 2, Oleaginous seeds and fruits and oilseed meals.

This second edition cancels and replaces the first edition (ISO 17059:2007), which has been technically revised.

The main changes compared to the previous edition are as follows:

- Description of the preparation of methyl esters in part 8.5. This part referred to ISO 5509:2000 in the previous version

Introduction

Chromatographic analysis of the fatty acid methyl esters (FAME) of oilseeds requires oil extraction from the oilseeds. To date, no International Standard has specified a method for extracting oil from oilseeds for FAME analysis. The methods usually performed in laboratories involve oil extraction for the determination of oil content and are tedious or time consuming[2], [3]. Consequently, the total duration and cost of the analysis of triglyceride fatty acids in oilseeds, including oil extraction, preparation and gas chromatography of the FAME are considerably increased by the oil extraction step.

This International Standard specifies a rapid and optimized method for a combined oil extraction and FAME preparation. The oil is only partially extracted from the seeds and the extracted fraction remains representative enough of the total content when the method is applied to the seeds specified in the Scope[4], [5]. The FAME are prepared according to the transesterification method described in ISO 5509, (2000) and slightly modified to be applied to iso-octane solutions of oil.

Taking into account that no reference method for oil extraction exists, the oil extraction method specified in this International Standard was compared to ISO 659[2] in an interlaboratory test[6]. Results showed very good agreement between the two methods except when applied to rapeseed with high erucic acid content. In this case, this method led to values of erucic acid content higher by approximately a mass fraction of 1 %.

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Oilseeds — Extraction of oil and preparation of methyl esters of triglyceride fatty acids for analysis by gas chromatography (Rapid method)

1 Scope

This International Standard specifies a rapid method for extraction of oil and for preparation of the methyl esters of fatty acids. The methyl esters thus obtained can be used for gas chromatography.

This International Standard is applicable to the following oilseeds: rape, sunflower, soya beans, mustard, linseed.

NOTE Applying this rapid method to high erucic acid content rapeseed leads to an overestimation of erucic acid content by approximately a mass fraction of 1 %. This difference was observed in the study [6] and may be due to the partial extraction of the oil from the sample (yield around 70%). High content of erucic acid in triglycerides could increase their solubility in hexane because of the lipophilic effect of the carbon long-chain (C22). However, as this effect was not checked on a large set of high erucic rapeseed samples, it is not recommended to apply a correction factor to the erucic acid content when analysing high erucic acid rapeseed.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 664, *Oilseeds — Reduction of laboratory sample to test sample*

ISO 21294, *Oilseeds — Manual or automatic discontinuous sampling*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Principle

The oil is cold extracted from previously crushed grains by shaking in iso-octane. After filtration, the triglyceride fatty acids present in the iso-octane solution are transesterified with potassium hydroxide into methyl esters.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Iso-octane (2,2,4-trimethylpentane) of chromatographic quality. See Annex A.1.

5.2 Anhydrous sodium sulfate.

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5.3 Potassium hydroxide, methanolic solution, approximately 2 mol/l. Since potassium hydroxide in practice contains about 15 % water, proceed as follows. Dissolve with gentle heating, 13,1 g of potassium hydroxide in 100 ml of absolute methanol. Add a quantity of anhydrous sodium sulfate to the solution to dry it. Filter to obtain a clear solution. If the solution has to be stored for a considerable time, a small amount of white precipitate of sodium carbonate may be formed; this has no effect on the preparation of the methyl esters when using the clear supernatant.

5.4 Sodium hydrogen sulfate monohydrate.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Blade crusher, coffee grinder type.

6.2 Test tubes, of glass, of capacity 10 ml, with ground or screw type stopper and PTFE cap.

6.3 Graduated pipette, of capacity 5 ml.

6.4 Pipette or automatic pipette, of capacity 200µl.

6.5 Pasteur pipettes, of length 150 mm, filled with a glass wool wick and anhydrous sodium sulfate up to a height of 20 mm.

6.6 Test tubes, of glass, of capacity 5 ml, with ground or screw type stopper and PTFE cap.

6.7 Glass vial, of capacity 2 ml, with screw type stopper and PTFE cap.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 21294^[1].

8 Procedure

8.1 Preparation of the test sample

Reduce the sample in accordance with ISO 664 and crush a quantity of approximately 10 g using a blade crusher (6.1) during 15 s.

NOTE For samples that are non-homogeneous in nature, i.e. contain significant quantities of unseparable seeds (such as *Sinapis arvensis* in canola) a larger sample (25 g) may be required to ensure an accurate estimate of fatty acids.

8.2 Test portion

8.2.1 General

The crushed material test portion shall be adapted as a function of the oil content of the sample in order to permit the extraction of approximately 100 mg of oil.