
**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)*





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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 of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *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 with the previous edition are as follows:

- the description of the preparation of methyl esters in 8.5 has been updated to remove the reference to ISO 5509:2000, which has been withdrawn.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Chromatographic analysis of the fatty acid methyl esters (FAME) of oilseeds requires oil extraction from the oilseeds. 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 document 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 5509:2000¹⁾ 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 document 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 %.

1) Withdrawn standard. Replaced by ISO 12966-2:2011 and ISO 12966-3:2009.

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

1 Scope

This document 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 document is applicable to the following oilseeds: rape and mustard with low erucic acid content (< 2 %), sunflower, soya beans, 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 Reference [6] and could 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 appropriate 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 12966-4, *Animal and vegetable fats and oils — Gas chromatography of fatty acid methyl esters — Part 4: Determination by capillary gas chromatography*

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:

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

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, in accordance with [A.1](#).