

ICS 67.050

English Version

Foods of plant origin - Multimethod for the determination of pesticide residues in vegetable oils by LC-MS/MS

Aliments d'origine végétale - Multiméthode de détermination de la teneur en résidus de pesticides dans les huiles végétales par CL-SM/SM

Pflanzliche Lebensmittel - Multiverfahren zur Bestimmung von Pestizidrückständen in pflanzlichen Ölen mit LC-MS/MS

This Technical Specification (CEN/TS) was approved by CEN on 11 May 2017 for provisional application.

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European foreword

This document (CEN/TS 17062:2017) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

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WARNING — The application of this Technical Specification may involve hazardous materials, operations and equipment. This Technical Specification does not claim to address all the safety problems associated with its use. It is the responsibility of the user of this Technical Specification to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

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1 Scope

This Technical Specification describes a method for the analysis of pesticide residues in plant oils (fat content > 90 %, water content < 5 %). It has been validated in an interlaboratory test with olive oil. However, laboratory experiences are available also for other kind of oils such as sunflower seed oil, sesame oil, flax seed oil, rape seed oil, grape seed oil, thistle oil and pumpkin seed oil.

2 Normative references

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

CEN/TS 17061:2017, *Foodstuffs — Guideline for the calibration and quantitative determination of chromatographic methods for the determination of pesticide residues and organic contaminants*

3 Principle

The homogeneous sample is extracted with acetonitrile. After centrifugation, an aliquot of the organic phase is cleaned-up by dispersive solid phase extraction (D-SPE; sorbents PSA and C18). To separate co-extracted fat a freeze-out step of the acetonitrile phase can be applied. After clean up an additional centrifugation step is performed. The extracts are acidified by adding a small amount of formic acid, to improve the storage stability of certain base-sensitive pesticides. The final extract can be directly used for LC-MS/MS analysis. A scheme of the procedure is given in Annex C.

NOTE In contrast to the method described in EN 15662 [1], this procedure does not include any addition of water.

4 Reagents

Unless otherwise specified, use reagents of recognized analytical grade. Take every precaution to avoid possible contamination of water, solvents, sorbents, inorganic salts, etc.

4.1 Water, HPLC quality.

4.2 Acetonitrile, HPLC quality.

4.3 Methanol, HPLC quality.

4.4 Acetic acid.

4.5 Ammonium formate.

4.6 Formic acid solution in acetonitrile, volume concentration $\sigma = 5 \text{ ml formic acid}/100 \text{ ml}$:

Dilute 5 ml of formic acid (mass fraction $w \geq 95 \%$) to 100 ml with acetonitrile (4.2).