



National Standards Authority of Ireland

IRISH STANDARD

I.S. EN 14524:2004

ICS 67.120.30

**FOODSTUFFS - DETERMINATION OF
OKADAIC ACID IN MUSSELS - HPLC METHOD
WITH SOLID PHASE EXTRACTION CLEAN-UP,
DERIVATIZATION AND FLUORIMETRIC
DETECTION**

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EUROPEAN STANDARD
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English version

**Foodstuffs - Determination of okadaic acid in mussels - HPLC
method with solid phase extraction clean-up, derivatization and
fluorimetric detection**

Produits alimentaires - Dosage de l'acide okadaïque dans
les moules - Méthode par CLHP avec purification par
extraction sur phase solide, dérivation et détection
fluorimétrique

Lebensmittel - Bestimmung von Okadasäure in Muscheln -
HPLC-Verfahren mit Reinigung durch
Festphasenextraktion, Derivatisierung und fluorimetrischer
Bestimmung

This European Standard was approved by CEN on 21 May 2004.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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Foreword

This document (EN 14524:2004) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2005, and conflicting national standards shall be withdrawn at the latest by February 2005.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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

EN 14524:2004 (E)

1 Scope

This document specifies a method for the quantitative determination of the content of okadaic acid in mussels and mussel products. The content of okadaic acid is determined as free extractable acid of mussel hepatopancreas. Okadaic acid, a fat-soluble toxin from dinophysis algae, is a main component of dinophysis toxins.

The method has been validated in an interlaboratory study according to ISO general principles on assessing accuracy of measurement methods and results. The limit of determination of this method (signal/noise = 10) is 100 µg/kg for okadaic acid in mussel hepatopancreas. The method has been validated for okadaic acid in cooked mussels at levels of 441 µg/kg to 1 467 µg/kg.

Laboratory experiences have shown that this method can also be used to determine other dinophysis toxins, e.g. dinophysis toxins 1, 2 and 3 (DTX-1, DTX-2 and DTX-3), see [1], [2], [3], [4], [5] and [6]

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.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*.

3 Principle

Mussel hepatopancreas is separated from cooked mussels and homogenized. The toxins are extracted using methanol, derivatized with 9-anthryldiazomethane and the extract is cleaned up using a solid phase extraction (SPE) cartridge with silica gel. Chromatographic separation is performed on a HPLC-gradient system, followed by fluorescence measurement of the 9-anthryldiazomethyl ester of the toxin at 412 nm with excitation at 365 nm. Determination of okadaic acid is performed using external standards.

4 Reagents and materials

4.1 General

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water according to grade 1 of EN ISO 3696.

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