

Foodstuffs — Determination of saxitoxin and dc-saxitoxin in mussels — HPLC method using pre-column derivatization with peroxide or periodate oxidation

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British Standard

ICS 67.120.30

National foreword

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 19 August 2004

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 17 and a back cover.

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Amendments issued since publication

Amd. No.	Date	Comments

© BSI 19 August 2004

ISBN 0 580 44311 6

English version

**Foodstuffs - Determination of saxitoxin and dc-saxitoxin in
mussels - HPLC method using pre-column derivatization with
peroxide or periodate oxidation**

Produits alimentaires - Détermination de la teneur en
saxitoxine et en dc-saxitoxine dans les moules - Méthode
par CLHP avec dérivation pré-colonne et par oxydation au
peroxyde ou au périodate

Lebensmittel - Bestimmung von Saxitoxin und DC-Saxitoxin
in Muscheln - HPLC-Verfahren mit
Vorsäulenderivatisierung mit Peroxid- oder
Periodatoxidation

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

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Foreword

This document (EN 14526:2004) 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of an identical text or by endorsement, at the latest by February 2005, and conflicting national standards shall be withdrawn at the latest by February 2005.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.

Introduction

This document is based on a procedure described by Lawrence and Ménard [1]. In this original procedure, the C₁₈ solid phase extraction is followed by a solid phase extraction with a weak cation exchanger. In the latter step, three different groups of shellfish toxins are being separated by elution with mobile phases of different ionic strength and polarity. The different groups of toxins are then derivatized with periodate or peroxide depending on the peak responses of the different toxins to the respective derivatization reactions. The method described in this document is specifically for saxitoxin and decarbamoyl (dc)-saxitoxin, so only the C₁₈ clean up is applied.

The method described in this document offers the option to choose between an extraction with acetic acid (HAc) or an extraction with hydrochloric acid (HCl). The analyst's choice depends on his desires and final needs.

HCl extraction with boiling leads to partial hydrolysis of the PSP toxins, leading to conversion of some of the PSP toxins (C-1, C-2, GTX-5) into more toxic analogues (GTX-2, GTX-3, saxitoxin). This process also occurs more or less in the human stomach. This extraction can therefore be considered a reasonable approach to estimate potential toxicity.

HAc extraction without boiling is a milder extraction procedure, which leaves the toxin profile of the sample practically intact. This extraction procedure can be applied if it is considered more important to estimate the actual toxin profile. The HAc extraction has been applied in the development of (certified) reference material for PSP toxins [2] and [3].