Foodstuffs - Determination of vitamin K1 by HPLC

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EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN 14148:2003 sisaldab Euroopa standardi EN 14148:2003 ingliskeelset teksti. Käesolev dokument on jõustatud 14.08.2003 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes. Standard on kättesaadav Eesti standardiorganisatsioonist.	This Estonian standard EVS-EN 14148:2003 consists of the English text of the European standard EN 14148:2003. This document is endorsed on 14.08.2003 with the notification being published in the official publication of the Estonian national standardisation organisation. The standard is available from Estonian standardisation organisation.

Käsitlusala: This draft European Standard specifies a method for the determination of vitamin K1 in foodstuffs by high performance liquid chromatography (HPLC). The determination of Vitamin K1 content is carried out by measurement of reduced phylloquinone. The method has been validated for milk and milk products, however laboratory experiences exist which show that the method is also applicable to other type of foodstuffs	**Scope:** This draft European Standard specifies a method for the determination of vitamin K1 in foodstuffs by high performance liquid chromatography (HPLC). The determination of Vitamin K1 content is carried out by measurement of reduced phylloquinone. The method has been validated for milk and milk products, however laboratory experiences exist which show that the method is also applicable to other type of foodstuffs

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14148

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English version

Foodstuffs - Determination of vitamin K1 by HPLC

Produits alimentaires - Dosage de la vitamine K1 par CLHP

Lebensmittel - Bestimmung von Vitamin K1 mit HPLC

This European Standard was approved by CEN on 2 May 2003.

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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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Contents

Foreword

This document (EN 14148:2003) 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 January 2004, and conflicting national standards shall be withdrawn at the latest by January 2004.

Annexes A, B and C are informative.

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.

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

1 Scope

This European Standard specifies a method for the determination of vitamin K_1 in foodstuffs by high performance liquid chromatography (HPLC). The determination of Vitamin K_1 content is carried out by measurement of reduced phylloquinone. The method has been validated for milk and infant formula, however laboratory experiences exist which show that the method is also applicable to other type of foodstuffs [10].

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

3 Principle

After enzymatic removal of fat from the sample vitamin K_1 is determined in an appropriate sample solution by high performance liquid chromatographic separation coupled with post-column reduction and subsequent fluorometric detection. Vitamin K_1 isomers are quantified as a single unresolved peak with a C_{18} column [1] to [4].

4 Reagents

4.1 General

During the analysis, unless otherwise stated, use only reagents of recognised analytical grade and water of at least grade 1 according to EN ISO 3696 or use distilled water.

4.2 Chemicals and solutions

4.2.1 Methanol, mass fraction $w(CH_3OH) \geq 99{,}8$ %

4.2.2 Ethanol, volume fraction $\varphi(C_2H_5OH) \geq 99{,}8$ %

4.2.3 Reagent alcohol, $\varphi(C_2H_5OH) = 95$ %

Mix 950 ml of ethanol (4.2.2) with 50 ml of methanol (4.2.1).

4.2.4 Dichloromethane, $w(CH_2Cl_2) \geq 99{,}5$ %

4.2.5 n-Hexane, $w(C_6H_{14}) \geq 97$ %

4.2.6 Light petroleum, bp. 35 °C to 60 °C, p.a.

4.2.7 Potassium hydroxide, $w(KOH) \geq 85$ %

4.2.8 Potassium hydroxide solution, substance concentration $c(KOH) = 10$ mol/l