

**Foodstuffs - Determination of patulin in
clear and cloudy apple juice and puree
- HPLC method with liquid/liquid
partition clean-up**

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cloudy apple juice and puree - HPLC method with
liquid/liquid partition clean-up

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

<p>Käesolev Eesti standard EVS-EN 14177:2004 sisaldab Euroopa standardi EN 14177:2003 ingliskeelset teksti.</p> <p>Käesolev dokument on jõustatud 18.05.2004 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.</p> <p>Standard on kättesaadav Eesti standardiorganisatsioonist.</p>	<p>This Estonian standard EVS-EN 14177:2004 consists of the English text of the European standard EN 14177:2003.</p> <p>This document is endorsed on 18.05.2004 with the notification being published in the official publication of the Estonian national standardisation organisation.</p> <p>The standard is available from Estonian standardisation organisation.</p>
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<p>Käsitlusala:</p> <p>This European Standard specifies a method for the determination of patulin in apple juices and apple puree using high performance liquid chromatography (HPLC). The method has been validated for the determination of patulin via the analysis of naturally contaminated and spiked samples in clear apple juice at levels ranging from 26 mg/l up to 128 mg/l, in cloudy apple juice at levels ranging from 26 mg/l up to 106 mg/l and in apple puree at levels ranging from 23 mg/kg up to 121 mg/kg.</p>	<p>Scope:</p> <p>This European Standard specifies a method for the determination of patulin in apple juices and apple puree using high performance liquid chromatography (HPLC). The method has been validated for the determination of patulin via the analysis of naturally contaminated and spiked samples in clear apple juice at levels ranging from 26 mg/l up to 128 mg/l, in cloudy apple juice at levels ranging from 26 mg/l up to 106 mg/l and in apple puree at levels ranging from 23 mg/kg up to 121 mg/kg.</p>
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ICS 67.080.10, 67.160.20

Võtmesõnad: analysis, chemical analysis and testin, food product, food technology, food testing, fruit juices, fruit-and vegetable juices, fruits, high performance liquid chromatography, hplc, liquid chromatography, patulin, purees (food), quantitative analysis, testing

ICS 67.080.10; 67.160.20

English version

Foodstuffs - Determination of patulin in clear and cloudy apple
juice and puree - HPLC method with liquid/liquid partition clean-
up

Produits alimentaires - Détermination de la teneur en
patuline dans le jus de pommes et dans la compote de
pommes, limpides et troubles - Méthode par
chromatographie en phase liquide à haute performance
avec purification par partage liquide/liquide

Lebensmittel - Bestimmung von Patulin in klarem und
trübem Apfelsaft und Apfelpüree - HPLC-Verfahren mit
Reinigung durch Flüssig/Flüssig-Verteilung

This European Standard was approved by CEN on 3 November 2003.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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.



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Contents

Page

Foreword.....	4
1 Scope	5
2 Normative references	5
3 Principle	5
4 Reagents	5
4.1 General.....	5
4.2 Sodium carbonate solution, mass concentration $\rho(\text{Na}_2\text{CO}_3) \approx 15 \text{ g/l}$	5
4.3 Acetic acid, volume concentration $\phi(\text{CH}_3\text{COOH}) \approx 98 \%$	5
4.4 Sodium sulfate anhydrous.....	5
4.5 pH 4 water	5
4.6 Absolute ethanol, $\phi(\text{CH}_3\text{CH}_2\text{OH}) \geq 99,7 \%$	6
4.7 Ethyl acetate.....	6
4.8 Perchloric acid, $\phi(\text{HClO}_4) = 60 \%$	6
4.9 Acetonitrile	6
4.10 Endogalacturonase enzyme solution, typical activity 1 400 U/g	6
4.11 HPLC mobile phase	6
4.12 Patulin stock solution.....	6
4.13 Patulin standard solution.....	6
4.14 Patulin standard solution for calibration.....	7
5 Apparatus	7
5.1 General.....	7
5.2 Displacement pipettes of 5 ml, 1 ml, 200 μl and 50 μl capacity with appropriate pipette tips.....	7
5.3 Analytical balance.....	7
5.4 UV spectrophotometer, double beam and recording suitable for measurement at 250 nm to 350 nm	7
5.5 Quartz cells, of optical path length 1 cm	7
5.6 Centrifuge, capable of operating at 4 500 g	7
5.7 Centrifuge tubes of 50 ml capacity with screw cap lids	7
5.8 Rotary evaporator, with water bath set at 40 °C	7
5.9 Filter paper, with pore size 20 μm to 25 μm	7
5.10 Disposable syringe filters, of 0,2 μm pore size (optional)	7
5.11 HPLC apparatus, comprising the following	7
5.12 Round bottom flasks, of 250 ml capacity with ground glass joint.	8
6 Procedure	8
6.1 Preparation of test samples.....	8
6.2 Extraction of patulin	8
6.3 Removal of interfering acidic compounds	8
6.4 Preparation of the sample test solution	9
7 Spiking procedure	9
7.1 Clear apple juice.....	9
7.2 Cloudy apple juice	9
7.3 Apple puree	9
8 HPLC determination	9
8.1 Calibration graph	9
8.2 HPLC operating conditions.....	10
9 Calculation.....	10

10	Precision.....	10
10.1	Interlaboratory test	10
10.2	Repeatability	11
10.3	Reproducibility.....	11
10.4	Test report	12
Annex A	(informative) Precision data	13
Bibliography	15

Foreword

This document (EN 14177:2003) 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 June 2004, and conflicting national standards shall be withdrawn at the latest by June 2004.

Annex A is 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 patulin in apple juices and apple puree using high performance liquid chromatography (HPLC). The method has been validated for the determination of patulin via the analysis of naturally contaminated and spiked samples in clear apple juice at levels ranging from 26 µg/l up to 128 µg/l, in cloudy apple juice at levels ranging from 26 µg/l up to 106 µg/l and in apple puree at levels ranging from 23 µg/kg up to 121 µg/kg.

2 Normative references

This European Standard incorporates by dated or undated reference, provision 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

Cloudy apple juice and apple puree are treated with a pectinase enzyme. Patulin is extracted from apple juice or enzyme treated puree with ethyl acetate. The solvent extract is cleaned up by liquid-liquid extraction with aqueous sodium carbonate solution. The ethyl acetate extract is dried with anhydrous sodium sulfate. After evaporation of ethyl acetate, patulin is quantitatively determined by high performance liquid chromatography (HPLC) with ultra violet (UV) detection.

4 Reagents

4.1 General

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of grade 1 as defined in EN ISO 3696. Solvents shall be of quality for HPLC analysis.

4.2 Sodium carbonate solution, mass concentration $\rho(\text{Na}_2\text{CO}_3) \approx 15 \text{ g/l}$

Dissolve 1,5 g of sodium carbonate in 100 ml of water.

4.3 Acetic acid, volume concentration $\phi(\text{CH}_3\text{COOH}) \approx 98 \%$

4.4 Sodium sulfate anhydrous

4.5 pH 4 water

Adjust water to pH 4 with acetic acid.