

**Toiduained. Patuliini sisalduse määramine
puuviljamahlas ja väikelastele mõeldud
puuviljapüreedes vedelikkromatograafilisel meetodil UV
detektoriga ja eelneva vedelik/vedelik puastamise ning
tahke faasi ekstraktsiooniga**

Foodstuffs - Determination of patulin in fruit juice and fruit based puree for young children - HPLC method with liquid/liquid partition cleanup and solid phase extraction and UV detection

EESTI STANDARDI EESSÕNA

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English Version

Foodstuffs - Determination of patulin in fruit juice and fruit based
purée for infants and young children - HPLC method with
liquid/liquid partition cleanup and solid phase extraction and UV
detection

Denrées alimentaires - Dosage de la patuline dans le jus
de fruits et la compote de fruits en alimentation infantile -
Méthode par CLHP avec purification par partition liquide-
liquide et extraction en phase solide et détection UV

Lebensmittel - Bestimmung von Patulin in Fruchtsaft und
Obstbrei für Säuglinge und Kleinkinder - HPLC-Verfahren
mit Reinigung durch Flüssig/Flüssig-Verteilung,
Festphasenextraktion und UV-Detektion

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Foreword

This document (EN 15890:2010) 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of an identical text or by endorsement, at the latest by March 2011, and conflicting national standards shall be withdrawn at the latest by March 2011.

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1 Scope

This European Standard specifies a method for the determination of patulin in fruit juices and fruit-based purée, such as baby food purée, using high performance liquid chromatography with ultra-violet detection (HPLC-UV). Using naturally contaminated and spiked samples this method has been validated for the determination of patulin in apple juice, at levels ranging from 3,0 µg/kg to 15,5 µg/kg, and in fruit-based baby food purée, at levels ranging from 3,4 µg/kg to 17,9 µg/kg. Baby food fruit purée used in this study contained a mixture of the following ingredients which are commercially available on the European market: blueberry; apple; banana; lemon; wheat biscuits; wheat syrup; whole milk; and vegetable oil. A detailed listing, including the fractions, of each product used in this study is given in [1].

Further information on validation, see Clause 9 and Annex B.

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 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Principle

Patulin is extracted from apple juice, or fruit-based purée, with a mixture of ethyl-acetate and hexane in the presence of sodium sulfate and sodium hydrogen carbonate. An aliquot of the extract is purified by solid-phase extraction and evaporated. The residue is re-dissolved in water of pH = 4 and patulin is separated by reverse phase (RP)-HPLC and quantitatively determined by UV detection.

4 Reagents

4.1 General

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696:1995, unless otherwise specified. Solvents shall be of quality for HPLC analysis, unless otherwise specified. Commercially available solutions with equivalent properties to the reagents listed may be used.

4.2 Perchloric acid, the mass fraction $w(\text{HClO}_4) \geq 60\%$ in water.

4.3 Sand, 50 mesh to 70 mesh particle size.

4.4 Silicagel solid phase extraction (SPE) cartridges (500 mg SiO₂).

4.5 Sodium sulfate anhydrous, Na₂SO₄.

4.6 Sodium hydrogen carbonate, NaHCO₃.

4.7 Glacial acetic acid, $w(\text{CH}_3\text{COOH}) \approx 98\%$ in water.