

Foodstuffs - Determination of nitrate and/or nitrite content - Part 2: HPLC/IC method for the determination of nitrate content of vegetables and vegetable products

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

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English Version

**Foodstuffs - Determination of nitrate and/or nitrite
content - Part 2: HPLC/IC method for the determination of
nitrate content of vegetables and vegetable products**

Produits alimentaires - Détermination de la teneur en
nitrates et/ou en nitrites - Partie 2 : Méthode de
détermination par CLHP/CI de la teneur en nitrates des
légumes et des produits à base de légumes

Lebensmittel - Bestimmung des Nitrat- und/oder
Nitritgehaltes - Teil 2: HPLC/IC-Verfahren für die
Bestimmung des Nitratgehaltes in Gemüse und
Gemüseerzeugnissen

This European Standard was approved by CEN on 15 October 2017.

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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 CEN-CENELEC Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (EN 12014-2:2017) 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of an identical text or by endorsement, at the latest by June 2018, and conflicting national standards shall be withdrawn at the latest by June 2018.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 12014-2:1997.

The following changes have been made to the former edition:

- a) the scope has been reduced from 50 mg/kg nitrate to 25 mg/kg nitrate and the upper limit has been deleted;
- b) the purification method 1 and 2 used in the preparation of sample test solutions has been deleted;
- c) a new matrix (iceberg lettuce) has been verified in an interlaboratory test;
- d) update of the HPLC/IC-conditions and chromatograms in Annex A;
- e) the procedure has been extensively revalidated and precision data in Annex B have been revised;
- f) editorially revised.

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Introduction

The method described in this standard has been developed and validated for investigations regarding the European legislation for nitrate in vegetables and vegetable products. Laboratory experience has shown that this analytical method is also suitable for the determination of nitrite in other matrices; however, this has not been validated in the interlaboratory test scheme cited here.

1 Scope

This European Standard specifies a high-performance liquid chromatographic (HPLC) and an ion chromatographic (IC) method for determination of the nitrate level in vegetables and vegetable products. This method is applicable for samples with a content of 25 mg/kg or greater.

It has been validated on naturally contaminated and spiked samples as beetroot juice with nitrate mass fractions of 194 mg/kg and 691 mg/kg, pureed carrots with nitrate mass fractions of 26 mg/kg and 222 mg/kg and with iceberg lettuce with nitrate mass fractions of 623 mg/kg and 3 542 mg/kg.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

The nitrate is extracted from the foodstuff with hot water. The determination is performed either by reverse-phase HPLC and UV detection, or by IC and conductivity or UV detection.

4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified. Water shall be nitrate-free and shall comply with grade 1 of ISO 3696.

4.1 Potassium nitrate.

4.2 Sodium nitrate.

4.3 Sulfuric acid, 96 % ($\rho = 1,84 \text{ g/ml}^1$).

4.4 Regenerator for suppressor, e.g. sulfuric acid ($c = 0,0125 \text{ mol/l}^2$).

Carefully pipette 20 ml of sulfuric acid (4.3) into a 1 000 ml volumetric flask containing 800 ml of water. Mix the solution, dilute to the mark with water and mix again. Transfer 33 ml of this solution to a 1 000 ml volumetric flask containing 500 ml of water. Dilute to the mark with water and mix.

Alternatively, use a method with electrolytic suppressor.

4.5 Nitrate stock solution, $\rho = 1\,000 \text{ mg/l}$ (expressed as ion NO_3^-).

Weigh 1,630 7 g of potassium nitrate (4.1) to the nearest 0,1 mg and dissolve in water in a 1 000 ml volumetric flask.

Alternatively, 1,370 9 g of sodium nitrate (4.2) can be used. If required, 15 ml of sulfuric acid (4.3) can be added as a preservative. Mix the solution, dilute to the mark with water and mix again. This solution is stable for at least two months if stored at +4 °C in the dark (refrigerator).

Alternatively, commercially available standard solutions can also be used.

1) ρ = mass concentration

2) c = substance concentration