Non-fatty foods - Determination of bromide residues - Part 1: Determination of total bromide as inorganic bromide

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EESTI STANDARDI EESSÕNA **NATIONAL FOREWORD**

Käesolev Eesti standard EVS-EN 13191-1:2000 sisaldab Euroopa standardi EN 13191-1:2000 ingliskeelset teksti. Käesolev dokument on jõustatud 13.10.2000 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes. Standard on kättesaadav Eesti standardiorganisatsioonist.	This Estonian standard EVS-EN 13191-1:2000 consists of the English text of the European standard EN 13191-1:2000. This document is endorsed on 13.10.2000 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 European Standard specifies a gas chromatographic (GC) method for the determination of bromide residues (including some organic bromide present) as inorganic bromide in non-fatty foods. Generally, the maximum residue levels are expressed in terms of bromide ion from all sources but not including covalently bound bromide. The method is applicable to beets, carrots, chicory, endives, cereal grains, lettuce, potatoes, spinach, strawberries and tomato. It has been validated in an interlaboratory test on lettuce.	**Scope:** This European Standard specifies a gas chromatographic (GC) method for the determination of bromide residues (including some organic bromide present) as inorganic bromide in non-fatty foods. Generally, the maximum residue levels are expressed in terms of bromide ion from all sources but not including covalently bound bromide. The method is applicable to beets, carrots, chicory, endives, cereal grains, lettuce, potatoes, spinach, strawberries and tomato. It has been validated in an interlaboratory test on lettuce.

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English version

Non-fatty food – Determination of bromide residues

Part 1: Determination of total bromide as inorganic bromide

Aliments non gras – Détermination des résidus de bromures – Partie 1: Détermination des bromures totaux en tant que bromures inorganiques

Fettarme Lebensmittel – Bestimmung von Bromidrückständen – Teil 1: Bestimmung von Gesamtbromid als anorganisches Bromid

This European Standard was approved by CEN on 2000-04-08.

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 Central Secretariat or to any CEN member.

The European Standards exist 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 Central Secretariat has the same status as the official versions.

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

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

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Contents

Foreword

This European Standard 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 November 2000, and conflicting national standards shall be withdrawn at the latest by November 2000.

This European Standard "Non-fatty foods - Determination of bromide residues" consists of two parts:

Part 1: Determination of total bromide as inorganic bromide

Part 2: Determination of inorganic bromide

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, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

The Annexes A and B are informative.

1 Scope

This European Standard specifies a gas chromatographic (GC) method for the determination of bromide residues (including some organic bromine present) as inorganic bromide in non-fatty foods.

Generally, the maximum residue levels are expressed in terms of bromide ion from all sources but not including covalently bound bromine.

The method is applicable to beets, carrots, chicory, endives, cereal grains, lettuce, potatoes, spinach, strawberries and tomato. It has been validated in an interlaboratory test on lettuce [1].

2 Normative reference

This draft 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 draft European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN ISO 3696 Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)

3 Principle

An aqueous ethanolic extract of the test portion is evaporated to dryness and the residue is ashed in the presence of sodium hydroxide. The ash is solubilized with sulfuric acid and the solution is treated with ethylene oxide in di-isopropyl ether. Inorganic bromide is converted to 2-bromoethanol, which is analyzed by gas chromatography with electron-capture detection [2].

4 Reagents

4.1 General and safety aspects

Unless otherwise specified, use reagents of recognized analytical grade, preferably for pesticide residue analysis, and water of grade 2 according to EN ISO 3696.

Take every precaution to avoid possible contamination of water, solvents, inorganic salts, etc. by plastics and rubber materials. Use only glass containers for storage and handling of all water and reagents.

4.2 Ethanol, $\varphi(C_2H_5OH) = 96\ \%\ (V/V)$

4.3 Sodium hydroxide solution, $c(NaOH) = 0{,}2$ mol/l

4.4 Sulfuric acid, $c(H_2SO_4) = 0{,}6$ mol/l

4.5 Acetone

4.6 Di-isopropyl ether, peroxide-free.

Before use, check each newly opened bottle by injecting the same volume, e.g. 5 µl, into the gas chromatograph as used in 6.3. If interfering peaks are observed, distill over potassium hydroxide.

WARNING: Di-isopropyl ether is extremely flammable. Store protected from light. Unstable peroxides can form upon longer standing or exposure to sunlight in bottles and can result in explosion risk. Work always in a well-ventilated fume hood.

4.7 Ethylene oxide, $\varphi(C_2H_4O)$ of at least 99,5 % volume fraction, in pressurized can fitted with valve. Store at approximately - 20 °C.

WARNING: Ethylene oxide is a highly reactive and cancerogenic gas. Work always in a well-ventilated fume hood. Consult the safety data sheets of the manufacturer for information.

To destroy excess ethylene oxide solution, add a surplus of sodium chloride solution, shake several times and allow to stand for some hours.