

Non-fatty food — Determination of bromide residues —

Part 2: Determination of inorganic bromide

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British Standard

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 11 and a back cover.

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Fettarme Lebensmittel – Bestimmung von
Bromidrückständen – Teil 2: Bestimmung von
anorganischem Bromid

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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of an identical text or by endorsement, at the latest by November 2000, and conflicting national standards shall be withdrawn at the latest by November 2000.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

The Annexes A and B are informative.

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.

1 Scope

This European Standard specifies a gas chromatographic method for the determination of inorganic bromide residues 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 cereals, dried fruit, dried vegetables, dried mushrooms, fresh fruit and vegetables. It has been validated in interlaboratory studies on maize flour, carrot flakes, lettuce, potatoes, cereal flour and hazelnuts [1], [2].

2 Normative reference

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.

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

3 Principle

The test portion is suspended in an aqueous solution of propylene oxide acidified with sulfuric acid whereupon inorganic bromide is extracted simultaneously and converted to a mixture of 1-bromo-2-propanol and 2-bromo-1-propanol [3] (derivatization A). The derivatives are partitioned into ethyl acetate and determined by gas chromatography with electron-capture detection [4], [5].

As an alternative, ethylene oxide which is more difficult to handle and which is somewhat more toxic, can be used instead of propylene oxide. In this case (derivatization B) inorganic bromide is converted to 2-bromoethanol [4], [5].

4 Reagents

4.1 General and safety aspects

Unless otherwise specified, use reagents of recognized analytical grade, preferably for pesticide residue analysis, and only 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.

WARNING: Ethylene oxide and propylene oxide are highly reactive and cancerogenic. Work always in a well-ventilated fume hood. Consult the safety data sheet of the manufacturer for information and follow local instructions.

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

4.2 Propylene oxide, Φ (C_3H_6O) of at least 99,5 % volume fraction. Store at approximately 4 °C.

4.3 Propylene oxide solution

In a well-ventilated fume hood, pour 95 ml of ice-cold water into a 100 ml volumetric flask, and add propylene oxide (4.2) dropwise to the mark and mix well. Store in a refrigerator at 4 °C. Prepare fresh daily.

4.4 Ethylene oxide (optional), Φ (C_2H_4O) of at least 99,5 % volume fraction, in pressurized can fitted with valve. Store at approximately - 20 °C.

4.5 Ethylene oxide solution (optional)

In a well-ventilated fume hood, pour 96 ml of ice-cold water into a 100 ml volumetric flask, and add ethylene oxide (4.4) dropwise to the mark from the completely inverted, ice-cold pressurized can and mix well. Store at approximately 4 °C. Prepare fresh daily.

4.6 Sulfuric acid, $c(H_2SO_4) = 3 \text{ mol/l}$.