

Non fatty foods — Determination of benzimidazole fungicides carbendazim, thiabendazole and benomyl (as carbendazim) —

Part 2: HPLC method with gel permeation chromatography clean up

The European Standard EN 14333-2:2004 has the status of a British Standard

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee AW/-/3, Horizontal analysis, which has the responsibility to:

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English version

Non fatty foods - Determination of benzimidazole fungicides
carbendazim, thiabendazole and benomyl (as carbendazim) -
Part 2: HPLC method with gel permeation chromatography clean
up

Aliments non gras - Détermination des benzimidazoles
antifongiques, le carbendazime, le thiabendazole et le
bénomyl en tant que carbendazime - Partie 2: Méthode
CLHP avec purification par chromatographie par
perméation de gel

Fettarme Lebensmittel - Bestimmung der Benzimidazol-
Fungizide Carbendazim, Thiabendazol und Benomyl (als
Carbendazim) - Teil 2: HPLC-Verfahren mit Reinigung
durch Gelpermeationschromatographie

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Foreword

This document (EN 14333-2:2004) 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of an identical text or by endorsement, at the latest by April 2005, and conflicting national standards shall be withdrawn at the latest by April 2005.

EN 14333 consists of the following parts, under the general title *Non fatty foods – Determination of benzimidazole fungicides carbendazim, thiabendazole and benomyl (as carbendazim)*:

- *Part 1: HPLC method with solid phase extraction clean up;*
- *Part 2: HPLC method with gel permeation chromatography clean up;*
- *Part 3: HPLC method with liquid/liquid-partition clean up.*

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1 Scope

This document specifies a high performance liquid chromatographic method for the determination of the benzimidazole fungicides carbendazim and thiabendazole in fruits, vegetables and processed products.

When benomyl is present, it is completely degraded to carbendazim and is also determined as carbendazim. Thiophanate-methyl is partly decomposed and therefore not quantitatively determined.

The method has been validated for carbendazim and thiabendazole in an interlaboratory test with homogenates of apples, French beans, mushrooms, lemons and fruit based infant food.

2 Principle

The sample is homogenized with ethyl acetate, sodium hydroxide solution and anhydrous sodium sulfate and the homogenate is filtered. An aliquot portion of the ethyl acetate extract is cleaned up by gel permeation chromatography (GPC) on a polystyrene gel using a cyclohexane/ethyl acetate mixture for elution. In the GPC eluate, carbendazim and thiabendazole are determined by high performance liquid chromatography (HPLC) on a normal phase column and with UV or UV and fluorescence detectors.

3 Reagents

3.1 General

Unless otherwise specified, use reagents of recognized analytical grade, preferably for HPLC and pesticide residue analysis, and only distilled or demineralized water.

3.2 Safety aspects associated with reagents

Vapours from some volatile solvents are toxic. Several of these solvents are readily absorbed through skin. Use an effective fume hood to remove vapours of these solvents as they are set free. Carbendazim and thiabendazole are toxic; avoid contact with skin and eyes.

3.3 Ethyl acetate

3.4 Cyclohexane

3.5 Sodium sulfate, anhydrous

3.6 Sodium hydroxide solution, mass concentration ρ (NaOH) = 26 g/100 ml

3.7 Diluted sodium hydroxide solution, ρ (NaOH) = 2,6 g/100 ml

3.8 GPC eluting mixture: Cyclohexane (3.4) / ethyl acetate (3.3) 1 + 1 (V/V)

3.9 Solvent mixture: dilute 5 ml ethyl acetate (3.3) with cyclohexane (3.4) to 100 ml

3.10 Mobile phase A for HPLC: Cyclohexane (3.4)

3.11 Mobile phase B for HPLC: Cyclohexane (3.4) / isopropanol / methanol 85 + 15 + 5 (V/V/V)

To 250 ml of this mixture, add one drop of ammonia solution (25 g/100 g). Prior to use, filter the mixture through a membrane filter (4.7).