

Foodstuffs — Determination of 3-monochloropropane- 1,2-diol by GC/MS

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National foreword

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Produits alimentaires - Dosage du 3-monochloropropane-
1,2-diol par GC/SM

Lebensmittel - Bestimmung von 3-Monochlorpropan-1,2-
diol mit GC/MS

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Contents

	Page
Foreword.....	3
1 Scope	4
2 Principle	4
3 Reagents	4
4 Apparatus	5
5 Procedure	6
6 Evaluation of results	8
7 Precision	8
8 Test report	9
Annex A (informative) Example of appropriate GC/MS operating conditions.....	10
Annex B (informative) Precision data.....	13
Bibliography	14

Foreword

This document (EN 14573:2004) has been prepared by Technical Committee CEN/TC 275 "Food Analysis - Horizontal methods", the secretariat of which is held by DIN.

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1 Scope

This document specifies a gas chromatographic method using mass spectrometric detection for the determination of 3-monochloropropane-1,2-diol (3-MCPD) in hydrolysed vegetable proteins and other foodstuffs [1]. The method has been validated in interlaboratory studies for malt extract, soup powder, bread crumbs, salami sausage, cheese alternative and hydrolysed vegetable protein.

2 Principle

The sample is mixed with a deuterated internal standard, with sodium chloride solution and with a solid support material. The mixture is transferred to a chromatographic column and is first extracted with a mixture of n-hexane and diethyl ether for removing non-polar components. Then, 3-MCPD is eluted with diethyl ether, the eluate is concentrated and an aliquot portion is derivatized using heptafluorobutyrylimidazole. The solution is analyzed by gas chromatography using mass spectrometric detection [2].

3 Reagents

3.1 General

Unless otherwise specified, use reagents of recognized analytical quality and distilled or demineralized water.

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 — The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

3.2 n-Hexane, glass distilled

3.3 Diethyl ether, glass distilled

3.4 2,2,4-Trimethylpentane

3.5 Ethyl acetate

3.6 Solvent mixture, n-hexane (3.2) and diethyl ether (3.3) 9+1 (V/V)

3.7 Sodium chloride solution, 290 g of sodium chloride dissolved in 1 l of water

3.8 Sodium sulfate, anhydrous

3.9 Heptafluorobutyrylimidazole

3.10 Solid support material, Extrelut®¹, refill packs, approx. 20 g

¹ Extrelut® is the trade name of a suitable product available commercially from E. Merck, Darmstadt (Germany). This information is given for the convenience of the users of this European Standard and does not constitute an endorsement by CEN of this product.