BRITISH STANDARD

BS EN 15055:2006

Non fatty foods — Determination of chlormequat and mepiquat — LC-MS/MS method

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ICS 65.060

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National foreword

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 19 and a back cover.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 June 2006

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ISBN 0 580 48618 4

Amendments issued since publication

Amd. No.	Date	Comments

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15055

May 2006

ICS 65.060

English Version

Non fatty foods - Determination of chlormequat and mepiquat - LC-MS/MS method

Aliments non gras - Détermination de la teneur en chlormequate et mepiquate - Méthode LC-MS/MS

Fettarme Lebensmittel - Bestimmung von Chlormequat und Mepiquat - LC-MS/MS-Verfahren

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Ref. No. EN 15055:2006: E

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Foreword

This document (EN 15055:2006) 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of an identical text or by endorsement, at the latest by November 2006, and conflicting national standards shall be withdrawn at the latest by November 2006.

As an alternative, there is also EN 15054 "Non fatty foods - Determination of chlormequat and mepiquat - LC-MS method" available.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This draft European Standard specifies a method using high performance liquid chromatography/tandem mass spectrometry (LC-MS/MS) for the determination of the growth regulators chlormequat and mepiquat in non-fatty foods as chlormequat and mepiquat cation, respectively.

The method is applicable to all kinds of fruits, vegetables and cereal products. It has been collaboratively studied on mushrooms, pears, wheat flour, fruit puree and, additionally, on infant formula, see [1].

2 Principle

The sample is mixed with deuterated internal standards, water and methanol and the homogenate is centrifuged. An aliquot portion of the supernatant is filtered. An aliquot portion of the filtrate is analysed by liquid chromatography with tandem mass spectrometric detection after positive electrospray ionisation. To achieve the required selectivity the mass spectrometer is operated in the multi reaction mode, see [2] to [7].

3 Reagents

3.1 General and safety aspects

Unless otherwise specified, use reagents of recognised analytical grade. Take every precaution to avoid possible contamination of water, solvents, inorganic salts, etc.

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 to determine the applicability of regulatory limitations prior to use.

3.2 Ammonium acetate

3.3 Ammonium formate

3.4 **Filter aid,** for example Celite® 545[1)]

3.5 **Glacial acetic acid,** mass fraction *w* at least 96 g/100 g

3.6 **Acetonitrile,** HPLC quality

3.7 **Methanol,** HPLC quality

3.8 **Water,** suitable for HPLC

3.9 **Chlormequat chloride stock solution,** mass concentration $\rho(C_5H_{13}NCl_2)$ = 4,30 µg/ml in methanol

This stock solution contains 3,33 µg/ml chlormequat cation.

1) Celite 545 is a product supplied by Celite Corp. (World Minerals Inc., Santa Barbara, CA, USA). This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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3.10 Mepiquat chloride stock solution, $\rho(C_7H_{16}NCl)$ = 4,37 µg/ml in methanol

This stock solution contains 3,33 µg/ml mepiquat cation.

3.11 Internal standard solution 1 (d4-Chlormequat chloride), $\rho(C_5H_9D_4NCl_2)$ = 21,33 µg/ml in methanol

This stock solution contains 16,67 µg/ml d_4-chlormequat cation. For the availability of the standard, contact your national standardisation institute.

3.12 Internal standard solution 2 (d3-Mepiquat iodide), $\rho(C_7H_{13}D_3NI)$ = 34,70 µg/ml in methanol

This stock solution contains 16,67 µg/ml d_3-mepiquat cation. For the availability of the standard, contact your national standardisation institute.

3.13 Calibration solutions

Prepare six calibration solutions (no. 1 to no. 6) in 10 ml volumetric flasks as follows. Into the flasks, pipette 10 µl, 50 µl, 100 µl, 300 µl, 1 000 µl or 3 000 µl each of chlormequat chloride stock solution (3.9) and of mepiquat chloride stock solution (3.10). To all flasks add 10 µl of the internal standard solution 1 (3.11) and 10 µl of the internal standard solution 2 (3.12). Dilute the solutions no. 1 to no. 5 to the mark with a methanol/water mixture 1 + 1 (*V*+*V*) and solution no. 6 with water.

All six calibration solutions contain 16,7 ng/ml of d_4-chlormequat cation and 16,7 ng/ml of d_3-mepiquat cation. Solutions no. 1 to no. 6 contain 3,3 ng/ml, 16,7 ng/ml, 33,3 ng/ml, 100 ng/ml, 333 ng/ml and 1 000 ng/ml each of chlormequat cation and of mepiquat cation respectively. On the basis of a 20 g test portion, this corresponds to 0,01 mg/kg, 0,05 mg/kg, 0,10 mg/kg, 0,30 mg/kg, 1,00 mg/kg or 3,00 mg/kg each of chlormequat cation and mepiquat cation.

4 Apparatus

Usual laboratory apparatus and, in particular, the following:

4.1 Homogenizer or high speed blender, fitted with jar

4.2 Centrifuge, capable of producing a centrifugal acceleration of at least 3 000 *g* at the bases of the centrifuge tubes

4.3 Syringe, capacity of at least 2 ml

4.4 Syringe filter, pore size 0,45 µm (polyamide or polytetrafluoroethylene)

4.5 Glass vials, 1,8 ml volume; applicable for automatic injection into an autosampler

4.6 LC-MS/MS system, equipped with electrospray interface

5 Procedure

5.1 Preparation of the samples

Where possible, carry out the analysis of samples immediately upon their arrival in the laboratory. Do not analyse a laboratory sample which is wholly or extensively spoiled.

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