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BSI Standards Publication

Food analysis — Determination of pesticide residues by GC-MS — Retention times, mass spectrometric parameters and detector response information

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National foreword

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Food analysis - Determination of pesticide residues by GC-MS - Retention times, mass spectrometric parameters and detector response information

Analyse des produits alimentaires - Détermination des résidus de pesticides par CG-SM - Temps de rétention, paramètres de spectrométrie de masse et information sur la réponse des détecteurs

Lebensmitteluntersuchung - Bestimmung von Pestizidrückständen mit GC-MS - Retentionszeiten, Parameter für die Massenspektrometrie und Detektionsempfindlichkeit

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Foreword

This document (CEN/TR 16468:2013) 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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Introduction

Pesticide residue analysis employs multiple methods involving extraction of residues from foods and clean-up of the extract to obtain as many analytes as possible in the purified extracts. Afterwards the extracts can be analysed by different kinds of instruments.

The hyphenation of gas chromatography (GC) and mass spectrometry (MS) is one of the most often used universal and selective analysis techniques for identification and quantification of pesticide residues in food extracts.

For the ionisation of the analytes (pesticides and/or their metabolites) in GC-MS, electron impact ionisation (EI) is most commonly used. If the typical electron energy of 70 eV is used, very often molecular ions (cation radicals) and several fragment ions are formed simultaneously.

The selective determination of each target analyte is performed by simultaneous aquisition of typically three ions formed by the analyte in the selected ion monitoring (SIM) mode. The diagnostic value of an ion depends on its mass. Usually even-numbered, high mass ions are less frequently formed and their recording results in more specific chromatograms. A reduction of selectivity may be caused by background ions formed during ‘column bleeding’ or by sample matrix (e.g. typical ions from fatty of fruit acids or hydrocarbons).

1 Scope

This Technical Report lists mass spectrometric parameters which are useful for the application of European Standards for the determination of pesticide residues in foods that use GC-MS, such as the following standards:

— EN 1528 (all parts), *Fatty food — Determination of pesticides and polychlorinated biphenyls (PCBs)*;

— EN 12393 (all parts), *Foods of plant origin — Multiresidue methods for the gas chromatographic determination of pesticide residues*;

— EN 15662, *Foods of plant origin — Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE — QuEChERS-method*.

To facilitate the determination of pesticides and/or metabolites using GC-MS, Table 1 specifies the diagnostic ions suitable for quantification, which can be used.

2 Analyte specific parameters for gas chromatographic determination of pesticides

2.1 General

All values indicated in Table 1 were acquired using GC-MS, GC-ECD or GC-NPD systems under the experimental conditions as outlined in 2.2. Comparative investigations showed that these parameters can be transferred simply on instruments of other types of the same or other manufacturers [1].

2.2 GC-MS, GC-ECD and GC-NPD Parameters

The following GC operating conditions have been proven to be satisfactory. This is an example for appropriate experimental conditions. Equivalent conditions may be used if they can be shown to lead to the same results.

Gas chromatograph	Agilent model 6890
Carrier gas	Helium, constant flow 1,0 ml/min (typical pressure at 70 °C is 61 kPa)
Injection technique	Pulsed splitless, 200 kPa; Pulse time 1,0 min
Injector temperature	240 °C
Injection liner	single taper, splitless (Agilent 5181-3316)
Injection volume	1 µl or 2 µl
Purge Gas	Helium; purge flow to split vent 50 ml/min; Purge time 1,5 min
Column	Fused silica capillary column HP-5MS, length 30 m, inner diameter 0,25 mm, film thickness 0,25 µm (Agilent Nr. 19091S-433)
Temperature programme	2 min 70 °C, programmed to rise at 25 °C/min to 170 °C, at 3 °C/min to 210 °C, at 30 °C/min to 290 °C, then isothermal for 9 min
Transfer Line Temperatur	280 °C
Mass spectrometer	Agilent MSD 5973N inert
MS temperatures	Quadrupole 150 °C; Source 230 °C;
Ionisation	Electron impact 70 eV
Solvent Delay	4,0 min
Retention time of parathion	15,85 min (parathion is used for calculation of relative retention times)