SLOVENSKI STANDARD
SIST-TP CEN/TR 16699:2014

01-oktober-2014

Živila - Določevanje ostankov pesticidov z GC-MS/MS - Parametri tandemske masne spektrometrije

Foodstuffs - Determination of pesticide residues by GC-MS/MS - Tandem mass spectrometric parameters

Lebensmittel - Bestimmung von Pestizidrückständen mit GC-MS/MS - Parameter für die Tandem-Massenspektrometrie

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Produits alimentaires - Détermination des résidus de pesticides par CG-SM/SM - Paramètres pour la spectrométrie de masse en tandem

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Ta slovenski standard je istoveten z: **CEN/TR 16699:2014**

ICS:

65.100.01	Pesticidi in druge agrokemikalije na splošno	Pesticides and other agrochemicals in general
67.050	Splošne preskusne in analizne metode za živilske proizvode	General methods of tests and analysis for food products

SIST-TP CEN/TR 16699:2014 **en,fr,de**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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TECHNICAL REPORT

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Foodstuffs - Determination of pesticide residues by GC-MS/MS - Tandem mass spectrometric parameters

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (CEN/TR 16699:2014) 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 residues analysis employs multi methods involving extraction of residues from the homogenized sample and clean-up of the extract in order to determine as many components as possible in the extracts. Afterwards the extracts can be analysed by different kind of instruments. The hyphenation of gas chromatography (GC) and tandem mass spectrometry (MS/MS) is a highly selective technique for identification and quantification of pesticide residues in extracts of plant and animal origin. This technique may be used to replace GC-MS detection in existing methods without the need for additional validation provided that calibration was successful and has demonstrated the required sensitivity and precision.

For the ionization of the analytes (pesticides and/or their metabolites) in GC-MS/MS, electron impact ionization (EI) is widely used because it offers sufficient ionization of most compound classes. However, very often molecular ions (cation radicals) and several fragment ions are formed simultaneously. For that reason, a rich variety of potential parent ions for MS/MS transitions exists compared to the soft ionization techniques applied in LC-MS/MS. This results in a greater freedom for the selection of an appropriate transition for a given situation. Such freedom is useful and often necessary in GC-MS/MS because a higher number of (fragment) ions are often produced by the matrix, which may interfere with the signal of the target analyte. On the other hand, this higher number of options may be a bit confusing for less experienced analysts.

To simplify the selection of suitable GC-MS/MS transitions in that situation, this Technical Report lists those MS/MS transitions, which have been reported most often in the scientific literature or are most often proposed by suppliers of GC-MS/MS instruments.

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1 Scope

This Technical Report lists the mass spectrometric parameters which are useful for the application of European Standards for the determination of pesticide residues in foods of plant origin that use GC-MS. These European Standards are as follows:

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 — QuEChERS-method*

To facilitate the determination of pesticides and/or metabolites using GC-MS/MS, Table 2 specifies the diagnostic ion transitions suitable for identification and quantification, which can be used.

2 Normative References

None.

3 Parameters for GC-MS/MS determination of pesticides following electron impact ionization

3.1 General

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All parameters given in Table 2 were carefully selected from different sources (open literature, application notes of instrument providers or tested by experts of CEN/TC 275/WG 4). Only transitions that were applicable on instruments of at least two different manufacturers (e.g. Agilent Technologies, Bruker Daltonics, Thermo Scientific, former Varian Inc., Waters Corporation) were selected.

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GC parameters and retention times are not described in this Technical Report. If needed, this information can be taken from CEN/TR 16468 "Food analysis - Determination of pesticide residues by GC-MS - Retention times, mass spectrometric parameters and detector response information".

3.2 General MS/MS Parameters

All listed MS/MS transitions were obtained from measurements with electron impact ionization at 70 eV. Other parameters (e.g. ion source temperature, ion source voltages, source pressure and the type of carrier gas) were not identical. It is well recognized that variations of these parameters may influence the intensity of selected precursor ions and individual optimization may be necessary. However, despite these differences very often the same preferred precursor ions were reported. This demonstrates that in many cases parameters can be transferred between instruments of other types of the same or other manufacturers, even if these instruments are operated under slightly different conditions.

3.3 Analyte specific MS/MS Parameters

The most often reported analyte specific ion transitions of pesticides are listed in Table 2. Transitions applicable for instruments of three suppliers are reported first and those transitions reported for instruments of two suppliers are written in italics. If available, at least two precursor ions with their transitions have been selected for each analyte. In general, transitions starting from ions with higher mass were preferred to transitions from low mass precursor ions.

Pesticide names (and those of some metabolites) are supplemented by the molecular mass and the CAS number (Chemical Abstracts Service), which is useful for searches in databases. The CAS number is usually taken from [1], but in various cases more than one number exists, e.g. for isomers and racemates.

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In addition to the above mentioned parameters, an indication on the appropriate collision energy for each transition is given. Instead of definitive voltages, a classification into low, medium and high collision energies was chosen, because transitions are based on data from different instruments, which do not require identical voltages to obtain the optimal intensity for a given transition. Therefore, from each reference (application note, article, etc.) all parameter sets were sorted by the collision energy in order to obtain the categories. Afterwards, the sorted list was divided in three groups of equal size. The resulting meaning of low, medium and high collision energy is listed in Table 1.

Table 1 — Categories of collision energy (CE)

Instrument	Voltage corresponding to low CE [V]	Voltage corresponding to medium CE [V]	Voltage corresponding to high CE [V]
Agilent 7000 Series	< 13	13 to 21	> 21
Bruker SCION TQ	< 8	8 to 16	> 16
Thermo Quantum	< 15	15 to 19	> 19
Varian 1200 (L)	< 13	13 to 20	> 20
Waters Quattro micro	< 9	9 to 17	> 17

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Table 2 — MS/MS Parameters

Pesticide name	CAS-Nr.	MRM No. 1	CE	MRM No. 2	CE	MRM No. 3	CE	MRM No. 4	CE	MRM No. 5	CE
Acephate	305-19-1	136 → 94	m	136 → 42	l						
Acetochlor	34256-82-1	146 → 130	h	223 → 132	h	223 → 146	l	*224 → 148*	*l*		
Aclonifen	74070-46-5	212 → 182	l	264 → 194	m	*264 → 211*	*l*	*264 → 182*	*h*	*264 → 212*	*l*
Acrinathrin	101007-06-1	181 → 152	m	208 → 181	l	289 → 93	l	*181 → 127*	*h*	*208 → 152*	*h*
Alachlor	15972-60-8	188 → 130	h	188 → 160	l						
Aldrin	309-00-2	263 → 191	h	263 → 193	h	263 → 228	m	*265 → 193*	*h*	*293 → 258*	*h*
Allethrin	584-79-2	123 → 81	l	*123 → 79*	*m*	*136 → 108*	*l*				
Atrazine	1912-24-9	200 → 94	m	200 → 104	m	200 → 122	l	215 → 173	l	215 → 200	l
Azinphos-ethyl	2642-71-9	132 → 77	l	160 → 77	m	160 → 104	l	160 → 132	l	132 → 104	l
Azinphos-methyl	86-50-0	132 → 77	m	160 → 77	m	160 → 104	l	160 → 132	l		
Azoxystrobin	131860-33-8	344 → 156	h	344 → 329	m	388 → 345	m	*388 → 300*	*m*		
Benfluralin	1861-40-1	292 → 160	m	292 → 206	m	292 → 264	l				
Bifenthrin	82657-04-3	181 → 115	h	181 → 165	h	181 → 166	m	*165 → 115*	*h*	*166 → 165*	*m*
Biphenyl	92-52-4	154 → 152	h	154 → 153	m	*153 → 152*	*m*	*154 → 128*	*m*		
Bitertanol	70585-36-3	141 → 115	m	170 → 115	h	170 → 141	m				
Boscalid (Nicobifen)	188425-85-6	140 → 76	h	140 → 112	l	342 → 140	m	*342 → 112*	*m*		
Bromophos	2104-96-3	329 → 314	m	331 → 286	h	331 → 316	m	*331 → 93*	*h*		
Bromophos-ethyl	4824-78-6	303 → 285	m	359 → 303	m	359 → 331	l	*357 → 301*	*m*	*358 → 303*	*m*
Bromopropylate	18181-80-1	185 → 157	h	341 → 155	h	341 → 157	h	341 → 183	m	341 → 185	m
Bupirimate	41483-43-6	273 → 108	m	273 → 193	l	316 → 208	l				
Buprofezin	69327-76-0	172 → 57	l	175 → 132	l	*105 → 77*	*m*	*105 → 104*	*l*		
Cadusafos	95465-99-9	159 → 97	m	159 → 131	l	*158 → 114*	*m*				
Captafol	2425-06-1	79 → 51	h	79 → 77	l	313 → 79	m	*150 → 79*	*m*	*151 → 79*	m
Captan	133-06-2	79 → 77	l	149 → 70	m	149 → 79	m	149 → 105	l		

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