EUCHINASAFE
中欧食品安全

Deliverable number: D4.5

Deliverable title:

Report on the establishment and application of a validated multianalyte / multi matrix screening method for pesticide residues in fruits and vegetables (and related products) set-up in Chinese and EU laboratories

WorkPackage No.:	WP4
Lead beneficiary:	VSCHT & CAIQ
Due date (project month - dd/mm/yyyy):	M42 - 31/12/2020
Actual delivery date (project month - dd/mm/yyyy):	M54 - 28/02/2022

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 727864 and from the Chinese Ministry of Science and Technology (MOST).

Delivering an Effective, Resilient and Sustainable EU-China Food Safety Partnership

Grant Agreement number:

727864 — EU-China-Safe

Acknowledgements

This report forms part of the deliverables of the project "EU-China-Safe" which has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 727864 and from the Chinese Ministry of Science and Technology (MOST).

EU-China-Safe aims at reducing food fraud and improving food safety through focusing on improving food legislation, food inspection and increasing access to information across Europe and China. State-of-the-art technologies including a virtual laboratory will create a unique space to share and demonstrate best practice. The use of innovative technologies will result in improved detection of adulteration of food products as well as increased traceability and transparency of global supply chains.

The project runs from September 2017 to August 2021. It involves 33 partners and is coordinated by QUB (The Queen's University of Belfast, UK).

More information on the project can be found at www.euchinasafe.eu

Document control page:

Deliverable Title	**Report on the establishment and application of a validated multianalyte / multi matrix screening method for pesticide residues in fruits and vegetables (and related products) set-up in Chinese and EU laboratories**
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Version number (VX.Y)	V1.1
Version date (dd/mm/yyyy)	22/04/2022
Last modified by (person and organisation name)	Jana Hajslova (VSCHT)
Rights (e.g. NA, Intellectual Property Rights, copyright, …)	NA

Revision history:			
Version	**Date**	**Modified by**	**Comments**
1	22/04/2022	Jana Hajslova	

Nature of the deliverable		
ORDP	Open Research Data Pilot	
R	Document, report (excluding the periodic and final reports)	x
DEM	Demonstrator, pilot, prototype, plan designs	
DEC	Websites, patents filing, press & media actions, videos, etc.	
E	Ethics	
OTHER	Software, technical diagram, etc.	

Dissemination Level		
PU	Public, fully open, e.g. web	x
CO	Confidential, restricted under conditions set out in Model Grant Agreement	
CI	Classified, information as referred to in Commission Decision 2001/844/EC	

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1. SUMMARY

Occurrence of pesticide residues in internationally traded food commodities is of high concern both in EU and China. To support export/ import of various food items, the mutual acceptance of certificates on results of contamination control is an important assumption.

The aim of this deliverable was establishment and application of a validated multi-analyte / multi matrix screening method for pesticide residues in fruits and vegetables and related products set-up in Chinese and EU laboratories. This method allows simultaneous determination of 323 pesticide residues and potentially co-occurring mycotoxins (56 compounds) in food of plant origin such as cereals, fruit, vegetables, and dried tea leaves.

The suitability of multi-analyte / multi-matrix screening method for pesticide residues in fruits and vegetables and related products, developed within activities of Task T4.2 (Multi-analyte / multi-matrix screening method for pesticide residues in fruits and vegetables (including tea) and fruit juices), was then evaluated by the Interlaboratory Comparison Study on Pesticide Residues in Food (ILC), see deliverable D4.6: Report on the application of multi-analyte method to inter-laboratory and survey samples. This exercise enabled check a feasibility of proposed approach and helped in overcoming of possible barriers in collaboration among EU and China regions.

2. INTRODUCTION

Food crops and products thereof may contain various potentially hazardous compounds, e.g. residues of plant protection products, or mycotoxins, secondary metabolites of parasitic fungi. The comprehensive control of these contaminants is not an easy task since, currently, there are over 1000 active pesticide substances registered in the European Union, and mycotoxins represent a large group of diverse compounds. Up to now, more than 400 toxic metabolites of filamentous fungi of the *Fusarium, Penicillium, Aspergillus,* and *Claviceps genera* have been reported. Although only several mycotoxins are currently regulated, many of them are on the list of 'emerging' mycotoxins, for which more occurrence data are required, since, they are subjects of health risk assessment process performed by EFSA.

A wide range of methods, nowadays mostly based on LC–MS, with up to hundreds analytes on the target list, has been developed for the above mentioned contaminants, nevertheless, simultaneous determination of their various groups of hazardous chemicals within a single run has been only rarely considered. The current 'gold' standard for target analytes detection in routine laboratories concerned with food safety control is represented by unit resolution tandem mass spectrometric detectors (MS/MS) such as triple quadrupole thanks to high specificity, as well as sensitivity n. However, the key limitation of MS/MS based methods is that, due to the monitoring of only specific ion transitions, neither post acquisition data re-interrogation, nor screening of unidentified unknowns, is possible. In this context, the growing interest in employing of high-resolution mass analyzers is not surprising. In the recent decade, full-scanning high resolution mass analyzers represented mainly by the time-of-flight (TOF) and orbital ion trap (orbitrap) mass analyzers have offered new challenges in food contaminants analysis. Especially sufficient separation of isobaric compounds, thus, enabling reliable analysis of even very complex matrices represents the key benefit. In this context, achievable mass resolution of measurement is a very important parameter related to the mass accuracy obtained. While TOF analyzers available at the market provide mass resolution in order of thousands up to tens of thousands of full width at half maximum (FWHM) units, ultra-high resolution power values higher than hundreds of thousands of FWHM are enabled even by benchtop orbitrap mass spectrometers. Besides the quantitative analysis and targeted screening, identification of 'unknowns' and retrospective data evaluation are the main advantages of these mass analyzers.

In the recent years, application potential of high resolution mass spectrometry has been expanded by introduction of hybrid tandem mass spectrometric systems that combine quadrupole ion analyzer, the

collision cell, and either TOF or orbitrap as high resolution mass analyzers. Such MS set-up allows acquiring of high resolution MS/MS spectra of specific precursor ions, thus providing additional information on their structure. The availability of hybrid tandem mass analyzers has obviously introduced new possibilities in the target analysis of pesticides, mycotoxins, and other food/feed contaminants. The requirements of SANCO/11312/2021 regarding the number of identification points necessary to achieve for a satisfactory analysis (i.e., gaining of two product ions with $\Delta m/z < 5$ ppm) can be fulfilled even at very low concentration level.

Within this task, we focused on an assessment of performance characteristics of newly developed multi-analyte/multi-group method employing high performance liquid chromatography coupled with tandem high resolution mass spectrometry with Q-orbitrap mass analyzer. To enable confirmation and demonstration of non-target screening potential, MS/MS spectral library was created using pure standards of 323 pesticides and 56 mycotoxins. Validation of the method was realized in QuEChERS-based extracts obtained from matrices representing fruits and vegetables, cereals, and tea.

3. ACTIVITIES AND RESULTS

3.1 DESCRIPTION OF ACTIVITIES

3.1.1. Activity 1: Development and validation of methodology for multianalyte / multi matrix screening method for pesticide residues in food of plant origin

A new reliable and highly sensitive method based on high performance liquid chromatographic (HPLC) separation and high resolution tandem mass spectrometric detection (HRMS/MS) was developed and validated for determination of 323 pesticide residues, and potentially co-occurring mycotoxins (56 compounds) in food of plant origin such as fruits and vegetables, cereals and teas. The method was validated for these matrices differing in nature/amount of co-extracts that may cause various matrix effects.

For extraction of target analytes with a wide range of physico-chemical properties from fruit / vegetable / cereals / tea samples, QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) like approach was used. The extraction mixture was acetonitrile:water; separation of organic and aqueous phase was induced by addition of inorganic salts. Acetonitrile phase was then analyzed using U-HPLC-MS/MS method, separately in ESI+/ESI− ionization mode.

Spectral HRMS/MS library was established providing an entire spectrum of fragmentation ions for each analyte, which allows unbiased identification and confirmation of target compounds. The limits of quantification (LOQs) of target analytes were below 10 µg kg^{-1} for 82%, 81%, and 61% for matrices representing fruits and vegetables, cereals, and tea, respectively. Recoveries were in the acceptable range (70-120%) according to SANCO/12571/2013 for most of target analytes, except for highly polar 'masked' mycotoxin deoxynivalenol-3-glucoside with recoveries 35%, 47%, and 42% for matrices representing fruits and vegetables, cereals, and tea, respectively. The linearities of calibration curves expressed as coefficients of determination were in the range of 0.9661-1.000, and repeatabilities expressed as relative standard deviations (RSDs) at LOQs lied in the range of 0.25-13.51%. The trueness of the method was verified using several certified reference materials (CRMs) and proficiency test samples.

The standard operation procedure (SOP) for the sample preparation, extraction, clean-up, separation, identification and quantification of pesticide residues and mycotoxins, and U-HPLC-MS/MS parameters was prepared. This SOP also included database of screened pesticide residues and mycotoxins, and performance characteristics (LOQ, Recovery, Repeatability) for developed method.

Standard operation procedure on “Simultaneous determination of pesticide residues and mycotoxins using multi-detection LC−MS method” is part of **Annex I** of this deliverable.

3.1.2. Activity 2: Test sample analysis

Test material for interlaboratory comparison / method transfer

For the purpose of method transfer at CAIQ, contaminated dried tea leaves test sample (origin from Asia) available at the VSCHT ISO 17025 accredited Metrological and Testing Laboratory was used.

The presence of pesticide residues in this material was determined by VSCHT using developed multiresidue method (see **Annex I**). In the **Table I** an overview of pesticide residues occurring in tea sample, together with contamination level are presented.

To minimise sample damage during transportation to China, dried material selected as test sample was found as appropriate one. It was transferred into 50 ml plastic centrifuge tube and tightly closed. In addition, the vial with the standard solution of pesticide standards mixture with more than 400 analytes (all pesticides present in the test material were contained) was also added to the shipment.

Table I: List of pesticide residues occurring in test material - green tea leaves (determined by VSCHT)

Pesticide residues detected	Elemental formula	Ionization, polarity	Concentration	EU MRL
			[mg/kg]	
acetamiprid	C10H11ClN4	ESI+	0,044	0,05
bifenthrine (sum of isomers)	C23H22ClF3O2	ESI+	0,117	30
buprofezin	C16H23N3OS	ESI+	0,016	0,05
diafenthiuron	C23H32N2OS	ESI+	0,104	0,01
difenoconazole	C19H17Cl2N3O3	ESI+	0,019	0,05
dinotefuran	C7H14N4O3	ESI+	0,023	0,01
fenbuconazole	C19H17ClN4	ESI+	0,016	0,05
fenpropathrin	C22H23NO3	ESI+	0,023	2
flonicamid	C9H6F3N3O	ESI+	0,041	-
flonicamid metabolite: TFNG	C9H7F3N2O3	ESI+	0,050	-
flonicamid: sum of flonicamid, TFNA and TFNG expressed as flonicamid	-	-	0,087	0,1
flufenoxuron	C21H11ClF6N2O3	ESI+	0,087	15
imidacloprid	C9H10ClN5O2	ESI+	0,032	0,05
lufenuron (any ratio of constituent isomers)	C17H8Cl2F8N2O3	ESI+	0,054	0,05
methoxyfenozide	C22H28N2O3	ESI+	0,011	0,05
pyridaben	C19H25ClN2OS	ESI+	0,036	0,05
tebuconazole	C16H22ClN3O	ESI+	0,100	0,05
thiacloprid	C10H9ClN4S	ESI+	0,068	10
tolfenpyrad	C21H22ClN3O2	ESI+	0,120	0,01

3.1.3. Activity 3: Methodology transfer

Sharing the methodology with the CAIQ

VSCHT represents EU laboratory accredited according to the International standard EN ISO/IEC 17025:2018, Moreover, VSCHT Accredited laboratory has status of Nominated laboratory closely collaborating with Czech Agricultural Food Inspection Authority (CAFIA), and generate data for EFSA database within EU coordinated EU program.

Alike VSCHT, CAIQ laboratory in China is well equipped for routine control / inspection and research activities in the field of pesticide multi-residues analysis. Examples of CAIQ activities are:

- HPLC/MS-MS and GC/MS-MS analytical methods of multi-residue pesticides in vegetables and fruits were established, then, the methods were described in detail in the Standard operation procedure and applied in the supervision and inspection of vegetables exported to Hongkong for 6 years.
- HPLC/MS-MS analytical method for multi-residue pesticides in tea was established and also widely used in China.
- Spectral database of high resolution MS data for 400 pesticides was established and applied in the screening of pesticide residues in vegetables. There are several types of high resolution mass spectrometers available in CAIQ laboratory including those equipped with Q-Orbitrap MS and Q-TOF mass analyzers. The data for spectral database were generated using Thermo Q-Exactive.

Thanks to availability of comparable instrumentation to that employed in EU partners´ laboratories, no specific obstacles were expected for method transfer in spite of the fact that such process, even when using the same analytical instrumentation, might be challenging.

However, due to COVID-19 pandemic, it was not possible to organise the visit of VSCHT scientists in Chinese CAIQ lab, focused on the method implementation and hands on experiments performed within on-site training.

Under such conditions, alternative tools, such as Skype meetings and emails exchange remained the only option to assist in the method transfer from VSCHT at CAIQ. Worth to notice that this communication platform was really challenging.

Standard operation procedure on “Simultaneous determination of pesticide residues and mycotoxins using multi-detection LC−MS method” (see **Annex I**, as used for D4.6) was provided to CAIQ via e-mail to work on the method implementation.

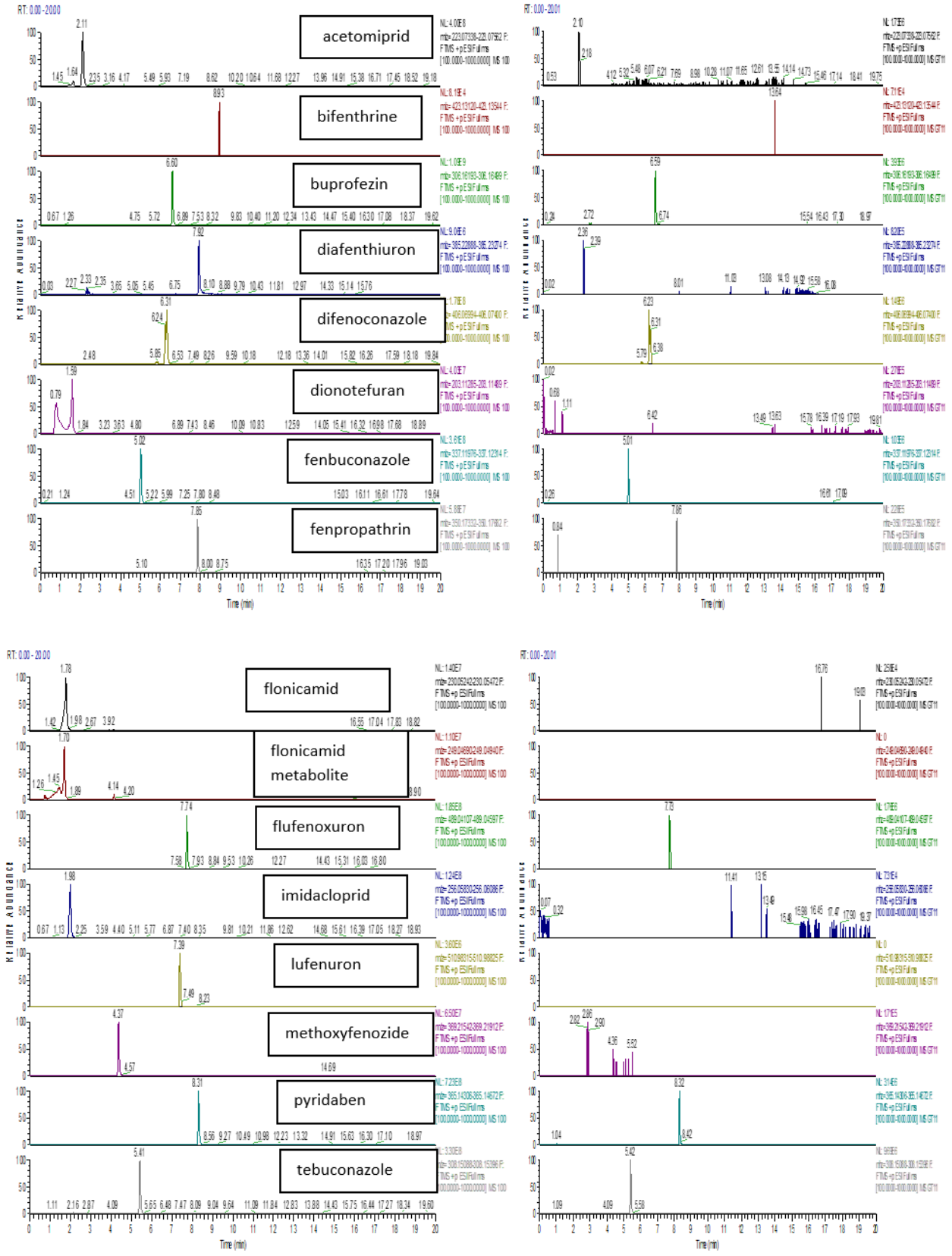
At VSCHT and CAIQ similar LC/MS instrumental platforms were used what enabled more effective method transfer. Comparable instrumental settings in terms of mass range, mass accuracy, linear dynamic range, sensibility and mass resolution/mass resolving power could be employed.

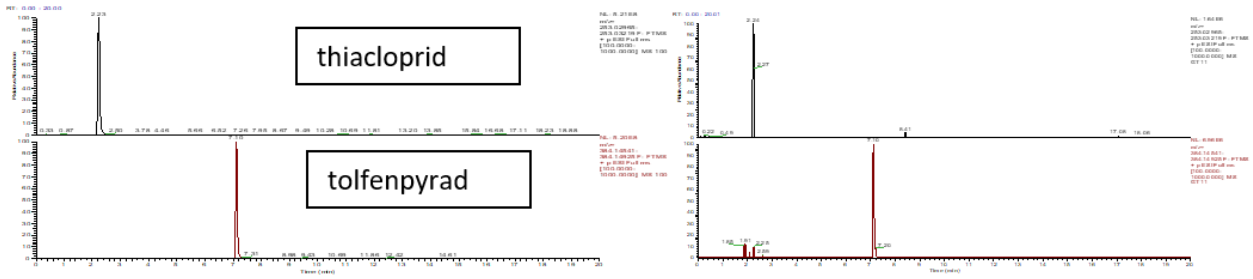
Once the first analytical results were obtained by CAIQ and shared with VSCHT via e-mail (MS PowerPoint file, see **Annex II**), Skype meeting focused on discussion of these results was organized. According to the provided presentation and following discussion, partial match in obtained results was achieved. In order to help our colleagues from CAIQ to enhance success rate within the interlaboratory comparison, list of pesticide residues present in the test material (see **Table I**) was shared via e-mail.

Once updated analytical results obtained by CAIQ were shared with VSCHT via e-mail (see e.g. **Figure 1**), another intensive discussions on these results were organized. Finally, major match in obtained results was achieved in this case. As a final step, the report was prepared by CAIQ demonstrating transferability of proposed multimethod (see **Annex III**) and in-house validation results for selected matrices.

Within this stepwise process, based on sharing the MS PowerPoint slides, interim results and reports with the demonstration of the results of analyses and follow-up discussion via Skype, more in-depth understanding of individual analytical steps was clarified, accuracy of obtained results was discussed, follow-up measures were proposed to achieve successful identification and quantification of analytes presented in the test sample.

Figure 1: Identification (screening) of individual pesticides in standards and tea sample (analysed and provided by CAIQ)





Left: chemical standards; Right: samples

3.2 DESCRIPTION OF RESULTS

3.2.1. Development and validation of methodology for multianalyte / multi matrix screening method for pesticide residues in food of plant origin and its transfer at Chinese laboratories

A multi-analyte UHPLC-HRMS method was developed by VSCHT and validated for rapid screening of a wide range of pesticide classes and mycotoxins in fruits and vegetables, cereals and tea. The method uses a rapid QuEChERS-like sample preparation, which allows the processing of large numbers of samples in a single day. Developed innovative method will allow not only a reliable control of compliance to existing maximum limits, but also retrospective data mining when required.

This efficient fully validated methodology was successfully transferred to CAIQ Chinese laboratories.

3.2.2 Dissemination

Information about results of the method transfer was shared with delegates of the CIFSQ 2021 conference:

- The information on the outcome of interlaboratory study was presented by Jana Hajslova in her lecture at China International Food Safety & Quality Conference (CIFSQ 2021), October 27 - 28, 2021, Beijing, China, during the session “EU-China Safe: China Intergovernmental Cooperation on S&T Innovation / EU Horizon 2020 Food, Agriculture and Biotechnology Flagship Project” on October 28, 2021

  Jana Hajslova, Leos Uttl, Dana Schusterova, Michal Stupak, Vladimir Kocourek, Monika Tomaniova: Current Challenges in Pesticide Residues Analysis

4. CONCLUDING REMARKS

The activities performed within Task T4.2 (Multi-analyte / multi-matrix screening method for pesticide residues in fruits and vegetables (including tea) and fruit juices) demonstrated feasibility of analytical method transfer for simultaneous determination of pesticide residues using multi-detection LC−MS method.

The results of internal interlaboratory comparison were jointly critically assessed, troubleshooting issues resolved, report on the method implementation completed.

Pesticide residues contained in the test material (dried tea leaves), shared within internal interlaboratory comparison, were successfully detected by CAIQ, thus, the transfer of the analytical method developed by VSCHT for pesticide residues determination can be considered as successful.

To document the feasibility of proposed analytical approach, this methodology was further verified within Interlaboratory Comparison Study on Pesticide Residues in Food organised by VSCHT, in collaboration with selected Chinese laboratories. For the detailed description of this Interlaboratory Comparison Study see deliverable D4.6: Report on the application of multi-analyte method to inter-laboratory and survey samples.

5. REFERENCES

We wish to acknowledge the contribution of all project partners who contributed to the completion of this deliverable.

6. APPENDIX

Annex I: Standard Operating Procedure – Simultaneous determination of pesticide residues and mycotoxins using multi-detection LC-MS method, including Supplementary document

Annex II: Presentation on method transfer provided by CAIQ

Annex III: Report demonstrating transferability of developed multimethod by CAIQ

Annex I: Standard Operating Procedure – Simultaneous determination of pesticide residues and mycotoxins using multi-detection LC-MS method, including Supplementary document

Simultaneous determination of pesticide residues and mycotoxins using multi-detection LC−MS method

SOP code	ILC-Multires-2021-SOP
Version / date of issue	2 / 07-07-2021
Institute / Laboratory	University of Chemistry and Technology / Testing Laboratory
Approved by:	J. Hajslova (UCT)
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CONTENTS

1. Method applicability

This method allows simultaneous determination of 323 pesticide residues and potentially co-occurring mycotoxins (56 compounds) in food of plant origin such as cereals, fruit, vegetables, herbal teas, and feedingstuffs. The list of target analytes is shown **Table S1.**

2. Principle

2.1 *Determination of pesticides and mycotoxins in fruits and vegetables*

For extraction of target analytes with a wide range of physico-chemical properties from fruit / vegetable samples, QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) like approach is used. The extraction mixture is acetonitrile:water; separation of organic and aqueous phase is induced by addition of inorganic salts. Acetonitrile phase is then analyzed using U-HPLC-MS/MS method, separately in ESI+/ESI− ionization mode.

2.2 *Determination of pesticides and mycotoxins in cereals and cereal-based products*

Analysis is based on the same principle as described in 2.1, the only difference is soaking of low moisture matrix by acidified water before extraction.

2.3 *Determination of pesticide and mycotoxins in tea*

Analysis is based on similar principle as described in 2.1. As a tea is highly complex matrix, some interfering co-extracts have to be removed prior to instrumental analysis by freezing out.

3. Chemicals and consumables

3.1 *Consumables*

- Automatic micropipettes, 100-1000 µL, 1-5 mL, 1-10 mL (e.g. TreffLab, Switzerland)
- Micro-filters, 0.2 µm (e.g. Ciro, USA)
- Plastic (PTFE) centrifuge tubes
- Crimp top vials, crimp-top caps
- Common laboratory glassware and equipment

3.2 *Chemicals*

- Deionized water, Milli-Q system, Millipore, (Germany)
- Acetonitrile, HPLC grade (e.g. Merck, USA)
- Methanol, LC−MS grade (e.g. Merck, USA)
- Hexane, HPLC grade (e.g. Merck, USA)
- Ammonium acetate, LC−MS grade (e.g. Merck, USA)
- Formic acid, LC−MS grade (e.g. Merck, USA)
- Ammonium formate, LC−MS grade (e.g. Merck, USA)
- Sodium chloride, p.a. (e.g. Merck, USA)
- Magnesium sulphate, p.a. (e.g. Merck, USA)
- Bondesil-C18, p.a. (Agilent Technologies, USA)

3.3 *Reference materials – calibration standards*

- Certified standards of target analytes listed in Table S1 (e.g. Merck, USA; Cayman Chemicals, USA; Romer Labs, AUT; TRC, USA; Santa Cruz, USA)

Preparation of standard stock solutions
Analytical standards of individual pesticides and mycotoxins are supplied either in acetonitrile or in solid state. Solid standards are dissolved in methanol or acetonitrile prior to further dilutions and stored at −20 °C. Separate working standards of pesticides and mycotoxins are prepared at concentration 1 000 ng/mL and then mixed to prepare calibration standards and solutions for method validation.

Preparation of matrix-matched calibration standards
From the abovementioned standard stock solutions (c = 1 000 ng/mL), additional lower concentration levels (500, 200, 100, 50, 20, 10, 5, 2 and 1 ng/ml) are prepared. Particular calibration standards (calibration points) at concentration levels 100, 50, 20, 10, 5, 2, 1, 0.5, 0.2 and 0.1 ng/mL are then prepared by pipetting 100 µl of adequate working standards into vials and addition of 900 µL of blank (analyte-free) sample extract. Matrix-matched calibration standards must be stored in a freezer at −20 °C.

4. Instruments and laboratory equipment

- Homogenizer, Grindomix GM200, Retsch (Germany)
- Laboratory shaker, HS 250 basic, IKA Labortechnik (Germany)
- Rotary vacuum evaporator R100, Büchi (Switzerland)
- Centrifuge, Rotina 35R, Hettich (Germany)
- Milli-Q system, Millipore (Germany)
- LC-MS instrumentation
  - ✓ Ultra-high performance liquid chromatograph UltiMate™ 3000 (Thermo Scientific, USA) or equivalent
  - ✓ High resolution tandem mass spectrometer Q-Exactive™ Plus (Thermo Scientific, USA)
  - ✓ Analytical column Accucore aQ (150 mm × 2.1 mm; 2.6 µm; Thermo Scientific, USA)

5. LC−MS analysis

5.1 *Liquid chromatography conditions*

Separation of analytes is performed using reverse-phased core-shell analytical column Accucore aQ (150 x 2.1; 2.6 µm; Thermo Scientific, USA; Phenomenex). Different mobile phases composition and gradient programs are used depending on an ionization mode, ESI+/ESI−.
To stabilize analytical column, its washing (after reaching the set column temperature) is performed for 30 min using initial composition of mobile phases.

Preparation of mobile phases
Preparation of 1 L of mobile phases (A/C and B/D):
Mobile phase A - 5 mM aqueous solution of ammonium formate is prepared by dissolving of 0.3125 g of ammonium formate in 1 L of 0.2 % formic acid in deionized water (*v/v*).

Mobile phase B - 5 mM methanol solution of ammonium formate is prepared by dissolving of 0.3125 g of ammonium formate in 1 L of 0.2 % formic acid in methanol (*v/v*).
Mobile phase C - 5 mM aqueous solution of ammonium acetate is prepared by dissolving of 0.3854 g of ammonium acetate in 1 L of deionized water.
Mobile phase D – 5 mM methanol solution of ammonium acetate is prepared by dissolving of 0.3854 g of ammonium acetate in 1 L of methanol.

Table 1: Liquid chromatography conditions used in U-HPLC-MS/MS analysis, both ionization modes

Column temperature	40 °C	
Analytical column	10 °C	
Injection volume	3 µL	
Mobile phases	ESI (+)	A: 5 mM ammonium formate in water (0.2 % formic acid) B: 5 mM ammonium formate methanol (0.2 % formic acid)
	ESI (−)	C: 5 mM ammonium acetate in water D: 5 mM ammonium acetate in methanol
Mobile phases gradient	**Table 2**	

Table 2: Gradient of mobile phases for U-HPLC-(ESI−/ESI+)-MS/MS

Time [min]	**Flow rate [mL/min]**	**Mobile phases composition [%]**	
		A/C	**B/D**
0	0.30	90	10
1	0.35	50	50
11	0.45	0	100
12	0.50	0	100
14	0.50	0	100
16	0.40	90	10

5.2 *Mass spectrometry conditions*

The data are acquired in *fullMS-data dependentMS*^{2} (*fullMS-ddMS*^{2}) mode under conditions shown in **Tables 3 – 5**. Monitored analytes are identified according to detection of their precursor ions (*fullMS*) and by comparison of analytes retention times in sample with calibration standards. Their presence is confirmed through detection of at least one fragment ion detection (*ddMS*^{2}) and comparison of theoretical and experimental isotopic pattern detection. The overview of exact masses of precursor and fragment ions of target analytes are summarized in **Table S1**. For the system control and data evaluation, Xcalibur® 4.0 (Thermo Scientific, USA) software is used.

Table 3: ESI ionization conditions

Ionization	ESI+/ESI−
Sheath/auxiliary gas (N_2)	45/10 arb. u.
Capillary temperature	300 °C
Heater temperature	300 °C
ESI needle voltage	+/− 3.3 kV

Table 4: Conditions of acquisition mode *fullMS*

Resolution	70 000 FWHM
Acquisition speed	1.5 Hz
Mass range	*m/z* 100-1000
Automatic gain control (AGC target)	$3e^6$
Maximum inject time (max IT)	200 ms

Table 5: Conditions of acquisition mode *ddMS2*

Resolution	17 500 FWHM
Acquisition speed	12 Hz
Mass range	*m/z* 50 - *m/z* fragmented analyte (+25)
Isolation window width	1 *m/z*
Automatic gain control (AGC target)	$1e^5$
Maximum inject time (max IT)	50 ms
Normalized collision energy (NCE)	**Table S1**
Dynamic exclusion	3 s
Intensity threshold	$2e^3$

5.3 *Quantification of analytes*

Quantitative evaluation is based on external standard calibration method performed by comparison of the signals of analytes with the relevant standards of pesticides and mycotoxins using the calibration curves constructed for each analyte.

Calibration procedure

Calibration should be constructed for at least three calibration levels. In case of a large sample set, series of calibration solutions are analyzed after each 20 – 30 injections of real samples. If the analyte response is not in the range of calibration, it is necessary to dilute the sample to fit the calibration range.

Calculation of the analyte concentration

Calibration is performed in Microsoft Excel program; linearity of calibration is verified through determination coefficient (r^2). Analyte signal must be enveloped by signals of calibration standards, extrapolation is not recommended.
Concentration of analyte in the tested sample is calculated as follows:

$$C_s = \frac{C_{cal} \times V}{m}$$

C_s final content of analyte in sample (µg/kg)
C_{cal} concentration of analyte calculated from calibration curve in sample (ng/mL)
V acetonitrile volume
m sample weight (g)

Notice:
Residues results do not have to be adjusted for Recovery when the mean Recovery is within the range of 80-120%. In case of Recovery correction, the initial result obtained for the applicable pesticide after analysis is multiplied with a factor [100% / Recovery%]. The mean Recovery obtained during validation / verification procedure should be used.

5.4 *Method performance characteristics*

Method trueness (bias) is verified by an analysis of a respective material spiked with standards mixture at two concentration levels (e.g. 20 and 100 ng/mL). Prior to analysis the sample should be conditioned at room temperature for 30 min.
The method performance characteristics comprising recovery, repeatability, and limits of quantification (LOQ) are summarized in **Table S2**.

6. Operation procedures

6.1 *Determination of pesticides and mycotoxins in fruits and vegetable*

Sample pre-treatment

Sample is homogenized and thoroughly mixed before weighing.

Extraction

Homogenized sample (10 g) is weighed into a 50 mL centrifuge tube and vigorously shaken in hand with 10 mL of acetonitrile for 2 min. Magnesium sulphate (4 g) and sodium chloride (1 g) are then added and tube shaken for another 1 min. Sample is then centrifuged at 10 000 RPM (5 min) and extract from the top acetonitrile layer micro-filtered (0.2 µm filter) prior to instrumental analysis.
In case of samples with lower moisture content (< 70 %), 5 g of sample can be weighed with 5 g of water.

6.2 *Determination of pesticides and mycotoxins in cereals and cereal-based food*

Sample pre-treatment

Mechanical impurities are removed from a laboratory sample, homogenization and thorough mixing follow prior to weighing. Samples with higher moisture content are carefully dried for 16 h at 40 °C).

Extraction

Acidified water (0.2% formic acid, *v/v*) is added to 2 g of a cereal sample in a 50 mL centrifuge tube and let to soak into the matrix (30 min). For voluminous materials, consider decreasing of sample weight to 1 g. Extract sample with 10 mL of acetonitrile (30 min) using a horizontal laboratory shaker. Magnesium sulphate (4 g) and sodium chloride (1 g) are then added and the tube vigorously shaken in hand for 1 min. Sample is then centrifuged at 10 000 RPM (5 min) and the upper acetonitrile layer is micro-filtered (0.2 µm filter) prior to instrumental analysis.

6.3 *Determination of pesticide residues and mycotoxins in tea*

Sample pre-treatment

Laboratory sample is homogenized and thoroughly mixed prior to weighing.

Extraction and clean-up

Sample (1 g) is weighed into a 50 mL centrifuge tube, 10 mL of acidified water (0.2 % formic acid, *v/v*) is then added, sample shaken and left 30 min to soak the sample matrix. After addition of 10 mL of acetonitrile, sample is shaken using horizontal laboratory shaker for 30 min at 240 RPM followed by addition of magnesium sulphate (4 g) and sodium chloride (1 g) and tube vigorously shaken in hand for 1 min. Sample is then centrifuged at 10 000 RPM (5 min) and 2 mL of the extract are removed from the upper acetonitrile layer to another centrifuge tube containing 0.1 g of Bondesil-C18 and 0.3 g of magnesium sulphate and hand-shaken for 1 min followed by centrifugation at 10 000 RPM (5 min). Purified extract is placed into a freezer (−20 °C) for 2 h to let freeze out the interfering matrix components and micro-filtered (0.2 µm filter) prior to instrumental analysis.

7. References

- ISO/IEC technical standard: *General requirements for the competence of testing and calibration laboratories ISO/IEC 17025:2018 (015253)*.
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Simultaneous determination of pesticide residues and mycotoxins using multi-detection LC−MS method
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Supplementary document

SOP code	ILC-Multires-2021-SOP_supplementary
Version / date of issue	1 / 31-05-2021
Institute / Laboratory	University of Chemistry and Technology / Testing Laboratory
Approved by:	J. Hajslova (UCT)
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Table S1 Exact masses *m/z* of precursor ions and fragments of mycotoxins and pesticide residues, retention times and normalized collision energies. Precursor ions showing the highest signal are highlighted in yellow.

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
MYCOTOXINS															
1	3+15-Acetyl-DON	C17H22O7	338.1360	2.27	337.1293	397.1504	339.1438	356.1704	10	C17H23O7	339.1438	C17H21O6	321.1333	C8H9O2	137.0597
2	aflatoxin B1	C17H12O6	312.0628	2.92	311.0561	371.0772	313.0707	330.0972	60	C14H9O4	241.0495	C15H9O5	269.0444	C13H10O3	214.0624
3	aflatoxin B2	C17H14O6	314.0785	2.77	313.0718	373.0929	315.0863	332.1129	70	C14H11O5	259.0601	C14H11O4	243.0652	C12H11O3	203.0703
4	aflatoxin G1	C17H12O7	328.0578	2.58	327.0510	387.0722	329.0656	346.0921	60	C13H11O3	215.0703	C14H11O4	243.0652	C13H10O3	214.0624
5	aflatoxin G2	C17H14O7	330.0734	2.45	329.0667	389.0878	331.0812	348.1078	60	C14H13O4	245.0808	C13H13O3	217.0859	C16H13O5	285.0758
6	agroclavine	C16H18N2	238.1465	2.23	237.1397	297.1609	239.1543	256.1808	50	C15H14N	208.1121	C12H11N2	183.0917	C14H16N	198.1277
7	alpha-zearalenol	C18H24O5	320.1618	4.87	319.1551	379.1762	321.1697	338.1962	60	C9H4O3	160.0166	C9H6O	130.0424	C11H11O	159.0815
8	alternariol	C14H10O5	258.0523	3.74	257.0455	317.0667	259.0601	276.0866	70	C12H7O4	215.0350	C9H7O2	147.0452	C11H7O2	171.0452
9	alternariol-methylether	C15H12O5	272.0679	5.80	271.0612	331.0823	273.0758	290.1023	60	C14H8O5	256.0377	C13H8O4	228.0428	C14H7O5	255.0299
10	beauvericin	C45H57N3O9	783.4089	8.60	782.4022	842.4233	784.4168	801.4433	30	C15H18O2N	244.1332	C9H12N	134.0964	C15H20O3N	262.1438
11	beta-zearalenol	C18H24O5	320.1618	4.16	319.1551	379.1762	321.1697	338.1962	60	C9H6O	130.0424	C9H4O3	160.0166	C10H6O3	174.0322
12	citrinin	C13H14O5	250.0836	2.95	249.0768	309.0980	251.0914	268.1179	50	C13H13O4	233.0808	C13H15O5	251.0914	C12H13O3	205.0859
13	cyclopiazonic acid	C20H20N2O3	336.1468	2.81	335.1401	395.1612	337.1547	354.1812	50	C6H6O3N	140.0353	C11H8N	154.0662	C9H10O3N	180.0666
14	deoxynivalenol	C15H20O6	296.1254	1.86	295.1187	355.1398	297.1333	314.1598	10	C2H3O2	59.0139	C15H19O6	295.1187	C14H17O5	265.1081
15	deoxynivalenol-3-glucoside	C21H30O11	458.1783	1.8	457.1715	517.1927	459.1861	476.2126	20	C20H27O10	427.1610	C21H29O11	457.1715	C14H15O4	247.0976
16	diacetoxyscirpenol	C19H26O7	366.1673	2.92	365.1606	425.1817	367.1751	384.2017	20	C17H23O5	307.1540	C15H17O2	229.1223	C15H19O3	247.1329
17	enniatin A	C36H63N3O9	681.4559	9.00	680.4492	740.4703	682.4637	699.4903	20	C12H20O2N	210.1489	C36H64O9N3	682.4637	C12H22O3N	228.1594
18	enniatin A1	C35H61N3O9	667.4402	8.71	666.4335	726.4546	668.4481	685.4746	20	C35H62O9N3	668.4481	C12H20O2N	210.1489	C11H18O2N	196.1332
19	enniatin B	C33H57N3O9	639.4089	8.06	638.4022	698.4233	640.4168	657.4433	30	C11H18O2N	196.1332	C11H20O3N	214.1438	C16H28NO5	314.1962
20	enniatin B1	C34H59N3O9	653.4246	8.42	652.4179	712.4390	654.4324	671.4590	30	C34H60O9N3	654.4324	C11H18O2N	196.1332	C12H20O2N	210.1489
21	ergocornine	C31H39N5O5	561.2946	3.09	560.2878	620.3090	562.3024	579.3289	30	C16H18ON3	268.1444	C15H15N2	223.1230	C19H17O2N2	305.1285
22	ergocorninine	C31H39N5O5	561.2957	3.62	560.2878	620.3090	562.3024	579.3289	30	C19H17O2N2	305.1285	C15H15N2	223.1230	C17H13N2O2	277.0972
23	ergocristine	C35H39N5O5	609.2957	3.62	608.2878	668.3090	610.3024	627.3289	30	C15H15N2	223.1230	C16H18N3O	268.1444	C19H17O2N2	305.1285
24	ergocristinine	C35H39N5O5	609.2957	4.08	608.2878	668.3090	610.3024	627.3289	30	C15H15N2	223.1230	C19H17O2N2	305.1285	C19H21O3N2	325.1547
25	ergocryptine	C32H41N5O5	575.3102	3.52	574.3035	634.3246	576.3180	593.3446	30	C16H18N3O	268.1444	C15H15N2	223.1230	C17H19N2O2	283.1441
26	ergocryptinine	C32H41N5O5	575.3102	3.99	574.3035	634.3246	576.3180	593.3446	30	C15H15N2	223.1230	C17H19N2O2	283.1441	C16H23N2O3	291.1703
27	ergometrine	C19H23N3O2	325.1785	2.07	324.1718	384.1929	326.1863	343.2129	50	C14H10NO	208.0757	C15H15N2	223.1230	C13H10N	180.0808
28	ergosine	C30H37N5O5	547.2789	2.85	546.2722	606.2933	548.2867	565.3133	30	C15H15N2	223.1230	C16H18N3O	268.1444	C14H10NO	208.0757
29	ergosinine	C30H37N5O5	547.2789	2.90	546.2722	606.2933	548.2867	565.3133	30	C15H15N2	223.1230	C16H18N3O	268.1444	C14H10NO	208.0757
30	ergotamine	C33H35N5O5	581.2633	2.98	580.2565	640.2777	582.2711	599.2976	30	C15H15N2	223.1230	C17H17N2O3	297.1234	C17H13N2O2	277.0972
31	ergotaminine	C33H35N5O5	581.2633	3.04	580.2565	640.2777	582.2711	599.2976	30	C15H15N2	223.1230	C14H10NO	208.0757	C16H18N3O	268.1444
32	fumonisin B1	C34H59NO15	721.3879	4.24	720.3812	780.4023	722.3957	739.4223	30	C22H40ON	334.3104	C22H44O3N	370.3316	C22H38N	316.2999
33	fumonisin B2	C34H59NO14	705.3930	5.07	704.3863	764.4074	706.4008	723.4274	30	C22H42ON	336.3261	C22H40N	318.3155	C22H46O3N	372.3472
34	fumonisin B3	C34H59NO14	705.3930	5.76	704.3863	764.4074	706.4008	723.4274	30	C22H42ON	336.3261	C22H44O2N	354.3367	C22H40N	318.3155
35	fusarenon X	C17H22O8	354.1309	2.02	353.1242	413.1453	355.1387	372.1653	10	C2H3O2	59.0139	C17H21O8	353.1242	C14H15O5	263.0925

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
36	gliotoxin	C13H14N2O4S2	326.0389	5.61	325.0322	385.0534	327.0468	344.0733	10	C2H6O2NS	108.0114	C5H5O4NS	174.9934	C13H15O2N2S	263.0849
37	HT-2 toxin	C22H32O8	424.2092	3.69	423.2024	483.2236	425.2170	442.2435	10	C15H19O4	263.1278	C14H15O2	215.1067	C14H17O3	233.1172
38	meleagrin	C23H23N5O4	433.1745	3.07	432.1677	492.1889	434.1823	451.2088	30	C17H12O3N5	334.0935	C22H21O3N5	403.1639	C16H9O2N4	289.0720
39	mycophenolic acid	C17H20O6	320.1254	4.12	319.1187	379.1398	321.1333	338.1598	50	C11H11O4	207.0652	C10H9O3	177.0546	C10H7O2	159.0441
40	neosolaniol	C19H26O8	382.1622	2.07	381.1555	441.1766	383.1700	400.1966	10	C17H21O5	305.1384	C15H17O3	245.1172	C14H15O2	215.1067
41	nivalenol	C15H20O7	312.1204	1.63	311.1136	371.1348	313.1282	330.1547	10	C2H3O2	59.0139	C14H17O6	281.1031	C15H19O7	311.1136
42	ochratoxin A	C20H18ClNO6	403.0817	5.04	402.0750	462.0961	404.0895	421.1161	20	C19H17O4NCl	358.0841	C11H8O4Cl	239.0106	C11H10O5Cl	257.0211
43	patulin	C7H6O4	154.0261	1.63	153.0193	213.0405	155.0339	172.0604	30	C6H5O2	109.0295	C5H5O	81.0346	C4H3O2	83.0139
44	paxilline	C27H33NO4	435.2415	7.28	434.2337	494.2548	436.2482	453.2748	20	C27H32O3N	418.2377	C9H8N	130.0651	C27H30O2N	400.2271
45	penicillic acid	C8H10O4	170.0574	2.08	169.0506	229.0718	171.0652	188.0917	40	C7H9O2	125.0597	C6H9O	97.0648	C6H8O2	112.0519
46	penitrem A	C37H44NO6Cl	633.2852	7.01	632.2784	692.2996	634.2930	651.3195	40	C32H33O5NCl	546.2053	C26H23O2NCl	416.1423	C18H13NOCl	294.0691
47	phomopsin A	C36H45ClN6O12	788.2778	5.86	787.2711	847.2923	789.2857	806.3122	10	C12H15O5N3	281.1006	C9H18O8N2	282.1058	C33H39O10N5Cl	700.2380
48	roquefortine C	C22H23N5O2	389.1846	3.86	388.1779	448.1990	390.1925	407.2190	40	C8H9O2N4	193.0720	C17H16O2N5	322.1299	C14H16N	198.1277
49	stachybotrylactam	C23H31NO4	385.2248	6.19	384.2180	444.2392	386.2326	403.2591	60	C9H8O3N	178.0499	C8H8O2N	150.0550	C9H10O3N	180.0655
50	sterigmatocystin	C18H12O6	324.0628	5.73	323.0561	383.0772	325.0707	342.0972	50	C17H10O6	310.0472	C16H9O5	281.0444	C16H10O5	282.0523
51	T-2 toxin	C24H34O9	466.2197	6.92	465.2130	525.2341	467.2276	484.2541	10	C21H31O7	395.2064	C12H17O4	225.1121	C24H35O9	467.2276
52	tentoxin	C22H30N4O4	414.2262	3.79+ **4.02**	413.2194	473.2406	415.2340	432.2605	30	C18H22O2N3	312.1707	C9H19ON2	171.1492	C18H24O3N3	330.1812
53	verrucarol	C15H22O4	266.1513	2.18	265.1445	325.1657	267.1591	284.1856	10	C15H21O3	249.1485	C15H19O2	231.1380	C15H17O	213.1274
54	verruculogen	C27H33N3O7	511.2313	5.93	510.2246	570.2457	512.2391	529.2657	20	C19H18O4N3	352.1292	C19H20O5N3	370.1397	C13H12ON	198.0913
55	zearalenone	C18H22O5	318.1462	5.02	317.1394	377.1606	319.1540	336.1805	50	C9H7O	131.0502	C10H7O3	175.0401	C9H4O3	160.0166
56	tenuazonic acid	C10H15NO3	197.1046	3.62	196.0979	256.1190	198.1125	215.1390	45	C6H5O3	125.0233	C9H13O2	153.0910	C6H8NO3	142.0499
	PESTICIDES														
57	2,4,5-T	C8H5Cl3O3	253.9299	2.61	252.9232	312.9443	254.9377	271.9643	60	C6H2OCl3	194.9177	C6HOCl2	158.9410	-	-
58	2,4-D	C8H6Cl2O3	219.9689	2.23	218.9621	278.9833	220.9767	238.0032	50	C6H3OCl2	160.9566	C6H2OCl	124.9800	-	-
59	2,4-DB	C10H10Cl2O3	248.0002	3.06	246.9934	307.0146	249.0080	266.0345	50	C6H3OCl2	160.9566	C6H2OCl	124.9800	-	-
60	2-NOA	C12H10O3	202.0624	2.22	201.0557	261.0768	203.0703	220.0968	30	C10H11O2	163.0754	C9H11O	135.0804	C7H7O	107.0491
61	4-CPA	C8H7ClO3	186.0078	1.94	185.0011	245.0222	187.0156	204.0422	40	C6H4OCl	126.9956	C2H3O2	59.0139	C4H7O	71.0502
62	acephate	C4H10NO3PS	183.0114	1.61	182.0046	242.0258	184.0192	201.0457	50	C2H8O3PS	142.9926	CH6O3PS	128.9770	CH7O3NP	112.0158
63	acetamiprid	C10H11ClN4	222.0667	2.28	221.0599	281.0811	223.0745	240.1010	40	C6H5NCl	126.0105	C3H6N	56.0495	C10H11N4	187.0978
64	acetochlor	C14H20ClNO2	269.1177	5.55	268.1110	328.1321	270.1255	287.1521	20	C13H17ONCl	238.0993	C12H15ONCl	224.0837	C10H14N	148.1121
65	acrinathrin	C26H21F6NO5	541.1324	9.16	540.1251	600.1462	542.1397	559.1662	20	C14H10ON	208.0757	C13H9O	181.0648	C5H7O	83.0491
66	alachlor	C14H20ClNO2	269.1177	5.53	268.1110	328.1321	270.1255	287.1521	30	C13H17ONCl	238.0993	C12H15ONCl	224.0837	C10H14N	148.1121
67	aldicarb	C7H14N2O2S	190.0770	2.64	189.0703	249.0915	191.0849	208.1114	30	C4H9S	89.0419	C5H10NS	116.0528	C4H8N	70.0651
68	aldicarb sulfone	C7H14N2O4S	222.0669	1.80	221.0602	281.0813	223.0747	240.1013	10	C4H8ON	86.0600	C2H6O2N	76.0393	C5H10O2NS	148.0427
69	aldicarb sulfoxide	C7H14N2O3S	206.0720	1.74	205.0652	265.0864	207.0798	224.1063	20	C5H10ONS	132.0478	C4H9S	89.0419	C4H9OS	105.0369
70	ametryn	C9H17N5S	227.1199	4.04	226.1132	286.1343	228.1277	245.1543	60	C6H12N5S	186.0808	C4H6N3	96.0556	C2H7N2S	91.0324
71	atrazine	C8H14ClN5	215.0932	3.80	214.0865	274.1076	216.1010	233.1276	60	C5H9N5Cl	174.0541	C4H6N3	96.0556	C2H3N3Cl	104.0010
72	avermectin-b1a	C48H72O14	872.4917	9.14	871.4849	931.5061	873.4995	890.5260	10	C19H29O3	305.2111	C34H47O7	567.3316	C19H31O3	307.2268
73	azadirachtin	C35H44O16	720.2624	3.35	719.2557	779.2768	721.2702	738.2968	20	C5H7O2	99.0452	C34H39O15	687.2294	C10H11O5	211.0612
74	azinphos-ethyl	C12H16N3O3PS2	345.0365	5.43	344.0298	404.0509	346.0443	363.0709	40	H4O3PS	114.9613	C3H8ONPS	137.0059	CH4O2PS2	142.9385

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
75	azinphos-methyl	C10H12N3O3PS2	317.0052	5.28	315.9985	376.0196	318.0130	335.0396	30	C2H8O3PS	142.9926	C8H6ON	132.0444	C8H10O3N	168.0655
76	azoxystrobin	C22H17N3O5	403.1163	4.46	402.1095	462.1307	404.1241	421.1506	30	C21H14O4N3	372.0979	C20H14O3N3	344.1030	C19H11O3N3	329.0795
77	benalaxyl	C20H23NO3	325.1672	6.27	324.1605	384.1816	326.1751	343.2016	20	C12H18O2N	208.1332	C10H14N	148.1121	C19H20O2N	294.1489
78	bendiocarb	C11H13NO4	223.0839	3.00	222.0772	282.0983	224.0917	241.1183	20	C9H11O3	167.0703	C6H5O2	109.0284	C3H7O	59.0491
79	bentazone	C10H12N2O3S	240.0563	1.79	239.0496	299.0707	241.0641	258.0907	60	C7H4ON2	132.0329	C7H5O3N2S	197.0026	C7H5ON2	133.0407
80	beta-cyfluthrin	C22H18Cl2FNO3	433.0642	8.71	432.0575	492.0786	434.0721	451.0986	20	C9H2O3NF	191.0013	-	-	-	-
81	bifenthrin	C23H22ClF3O2	422.1255	9.90	421.1188	481.1399	423.1333	440.1599	40	C14H13	181.1012	C13H10	166.0777	C8H9ClF2	178.0355
82	bitertanol	C20H23N3O2	337.1785	6.58	336.1718	396.1929	338.1863	355.2129	10	C2H4N3	70.0400	C18H21O2	269.1536	C6H11O	99.0804
83	boscalid	C18H12Cl2N2O	342.0321	4.80	341.0254	401.0465	343.0399	360.0665	40	C18H12ON2Cl	307.0633	C18H11ON2	271.0866	C18H12ON2	272.0944
84	bromacil	C9H13BrN2O2	260.0155	3.02	259.0088	319.0299	261.0233	278.0499	50	Br	78.9189	C5H4O2N2Br	202.9462	C4H3ONBr	159.9403
85	bromoxynil	C7H3Br2NO	274.8576	2.04	273.8509	333.8720	275.8654	292.8920	80	Br	78.9189	C7HONBr	193.9247	-	-
86	bromuconazole	C13H12BrCl2N3O	374.9535	5.25+6.02	373.9468	433.9679	375.9614	392.9879	30	C7H5Cl2	158.9763	C4H6O	70.0413	C7H3OCl2	172.9555
87	bupirimate	C13H24N4O3S	316.1564	5.41	315.1496	375.1708	317.1642	334.1907	40	C8H12ON3	166.0975	C2H6O2NS	108.0114	C11H20ON3	210.1601
88	buprofezin	C16H23N3OS	305.1556	7.57	304.1489	364.1700	306.1635	323.1900	20	C9H17ON2S	201.1056	C5H10NS	116.0528	C5H9ON2S	145.0430
89	cadusafos	C10H23O2PS2	270.0872	6.90	269.0804	329.1016	271.0950	288.1215	10	C2H8O2PS2	158.9698	C6H16O2PS2	215.0324	H4O2PS2	130.9385
90	carbaryl	C12H11NO2	201.0784	3.13	200.0717	260.0928	202.0863	219.1128	60	C10H9O	145.0648	C9H9	117.0699	C8H7	103.0542
91	carbendazim	C9H9N3O2	191.0689	2.06	190.0622	250.0833	192.0768	209.1033	80	C8H6ON3	160.0505	C7H6N3	132.0556	C6H5N2	105.0447
92	carbofuran	C12H15NO3	221.1046	3.04	220.0979	280.1190	222.1125	239.1390	30	C10H13O2	165.0910	C7H7O2	123.0441	C8H9O2	137.0597
93	carbofuran-3-hydroxy	C12H15NO4	237.0996	2.22	236.0928	296.1140	238.1074	255.1339	20	C12H14O3N	220.0968	C10H13O3	181.0859	C10H11O2	163.0754
94	carbophenothion	C11H16ClO2PS3	341.9733	8.14	340.9666	400.9877	342.9811	360.0077	20	C7H6ClS	156.9873	C3H8O2PS2	170.9698	CH4O2PS2	142.9385
95	cinerin I	C20H28O3	316.2033	8.46	315.1966	375.2177	317.2111	334.2377	20	C10H13O	149.0961	C8H11	107.0855	C9H13	121.1012
96	cinerin II	C21H28O5	360.1931	6.97	359.1864	419.2075	361.2010	378.2275	20	C10H13O	149.0961	C8H11	107.0855	C9H13	121.1012
97	clofentezine	C14H8Cl2N4	302.0121	7.00	301.0053	361.0265	303.0199	320.0464	50	C7H5NCl	138.0105	C7H4N3	130.0400	C5H4N	78.0338
98	clomazone	C12H14ClNO2	239.0708	4.36	238.0640	298.0852	240.0786	257.1051	100	C7H6Cl	125.0153	C7H5	89.0386	C5H4Cl	98.9996
99	clothianidin	C6H8ClN5O2S	249.0082	2.16	248.0014	308.0226	250.0160	267.0425	50	CNS	57.9757	C6H5N4S	165.0240	C5H2N4S	150.0006
100	cyanazine	C9H13ClN6	240.0885	2.80	239.0817	299.1029	241.0963	258.1228	50	C8H13N5Cl	214.0854	C4H7N3Cl	132.0323	C4H6N3	96.0556
101	cyazofamid	C13H13ClN4O2S	324.0442	5.61	323.0375	383.0586	325.0521	342.0786	10	C2H6O2NS	108.0114	C13H14N4Cl	261.0902	C11H8N3Cl	217.0401
102	cymoxanil	C7H10N4O3	198.0747	2.38	197.0680	257.0891	199.0826	216.1091	60	C3H3ON2	83.0240	C4H5O3N2	129.0295	C4H3O2N2	111.0189
103	cypermethrin	C22H19Cl2NO3	415.0737	8.88+8.97	414.0669	474.0881	416.0815	433.1080	10	C8H9OCl2	191.0025	C22H20O3NCl2	416.0815	-	-
104	cyproconazole	C15H18ClN3O	291.1133	4.97+5.23	290.1066	350.1277	292.1211	309.1477	40	C4H6O	70.0413	C7H6Cl	125.0153	C7H4OCl	138.9945
105	cyprodinil	C14H15N3	225.1260	6.03	224.1193	284.1405	226.1339	243.1604	90	C6H7N	93.0573	C7H7	91.0542	C7H6N2	118.0525
106	DEET	C12H17NO	191.1305	3.91	190.1237	250.1449	192.1383	209.1648	60	C8H7O	119.0491	C7H7	91.0542	C7H9O	109.0648
107	deltamethrin	C22H19Br2NO3	502.9726	8.96	501.9659	561.9870	503.9804	521.0070	10	C22H20O3NBr2	503.9804	C8H9OBr2	278.9015	C13H11O2	199.0754
108	demeton-S-methyl	C6H15O3PS2	230.0195	3.13	229.0127	289.0339	231.0273	248.0538	40	H4O3PS	114.9613	C2H8O3PS	142.9926	C2H8O3PS2	174.9647
109	demeton-S-methylsulfone	C6H15O5PS2	262.0093	1.94	261.0026	321.0237	263.0171	280.0437	50	C2H8O4P	127.0155	C2H8O3PS	142.9926	C4H10O3PS	169.0083
110	desmedipham	C16H16N2O4	300.1105	3.99+4.12	299.1037	359.1249	301.1183	318.1448	30	C8H10O3N	168.0655	C7H6O2N	136.0393	C9H12O3N	182.0812
111	desmetryn	C8H15N5S	213.1043	3.36	212.0975	272.1187	214.1121	231.1386	60	C5H10N5S	172.0651	C3H4N3	82.0400	C2H7N2S	91.0324
112	diazinon	C12H21N2O3PS	304.1005	6.32	303.0938	363.1149	305.1083	322.1349	40	C8H13N2S	169.0794	C8H13ON2	153.1022	H4O3PS	114.9613
113	diclofop-methyl	C16H14Cl2O4	340.0264	7.69	339.0196	399.0408	341.0342	358.0607	10	C14H11O2Cl2	281.0131	C16H15O4Cl2	341.0342	_	-
114	dicrotophos	C8H16NO5P	237.0761	2.07	236.0693	296.0905	238.0839	255.1104	30	C6H10ON	112.0757	C2H8O4P	127.0155	C3H6ON	72.0444

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
115	diethofencarb	C14H21NO4	267.1465	4.37	266.1398	326.1609	268.1543	285.1809	30	C10H14O2N	180.1019	C11H16O4N	226.1074	C8H10O2N	152.0706
116	difenoconazole	C19H17Cl2N3O3	405.0641	6.96+7.05	404.0574	464.0785	406.0720	423.0985	30	C13H9OCl2	251.0025	C17H15O3Cl2	337.0393	C16H13OCl2	291.0338
117	diflubenzuron	C14H9ClF2N2O2	310.0315	5.77	309.0248	369.0459	311.0393	328.0659	20	C14H7O2N2ClF	289.0186	C7H4ONF2	156.0266	C7H4N2Cl	151.0068
118	diflufenican	C19H11F5N2O2	394.0735	7.21	393.0668	453.0879	395.0813	412.1079	60	C13H7O2NF3	266.0423	C12H7ONF3	238.0474	C13H6O2NF2	246.0361
119	dichlofluanid	C9H11Cl2FN2O2S2	331.9618	5.40	330.9550	390.9762	332.9696	349.9961	10	C7H5NCl2FS	223.9498	C9H12O2N2Cl2FS2	332.9696	C3H6NCl2	125.9872
120	dichlormid	C8H11Cl2NO	207.0212	3.20	206.0145	266.0356	208.0290	225.0556	20	C3H4ONCl2	139.9664	C6H9	81.0699	C6H12N	98.0964
121	dichlorprop	C9H8Cl2O3	233.9845	2.46	232.9778	292.9989	234.9923	252.0189	50	C6H3OCl2	160.9566	C6H2OCl	124.9800	-	-
122	dichlorvos	C4H7Cl2O4P	219.9454	3.05	218.9386	278.9598	220.9532	237.9797	50	C2H8O4P	127.0155	C2H4OCl	78.9945	C2H7O3ClP	144.9816
123	dimethachlor	C13H18ClNO2	255.1021	4.12	254.0953	314.1165	256.1099	273.1364	30	C12H15ONCl	224.0837	C10H14N	148.1121	-	-
124	dimethenamide	C12H18ClNO2S	275.0741	4.67	274.0674	334.0885	276.0820	293.1085	50	C9H14NS	168.0841	C11H15ONClS	244.0557	C6H7S	111.0263
125	dimethoate	C5H12NO3PS2	228.9991	2.27	227.9923	288.0135	230.0069	247.0334	20	C4H8O3PS2	198.9647	C3H8O2PS2	170.9698	C3H6NS	88.0215
126	dimethomorph	C21H22ClNO4	387.1232	4.65+4.98	386.1165	446.1376	388.1310	405.1576	50	C9H9O3	165.0546	C17H14O3Cl	301.0626	C7H4OCl	138.9945
127	dimoxystrobin	C19H22N2O3	326.1625	5.92	325.1558	385.1769	327.1703	344.1969	10	C11H13O2N2	205.0972	C16H16ON	238.1226	C8H6N	116.0495
128	diniconazole	C15H17Cl2N3O	325.0743	6.77	324.0676	384.0887	326.0821	343.1087	50	C2H4N3	70.0400	C7H5Cl2	158.9763	C7H3OCl2	172.9555
129	disulfoton	C8H19O2PS3	274.0279	6.74	273.0212	333.0423	275.0358	292.0623	10	C4H9S	89.0419	C5H8O2S	132.0240	-	-
130	disulfoton-sulfone	C8H19O4PS3	306.0178	3.61	305.0110	365.0322	307.0256	324.0521	10	C8H20O4PS3	307.0256	C4H12O3PS	171.0239	C6H14O3PS3	260.9837
131	disulfoton-sulfoxide	C8H19O3PS3	290.0228	3.57	289.0161	349.0372	291.0307	308.0572	10	C4H10O2PS2	184.9854	C6H14O2PS2	213.0167	C4H12O3PS	171.0239
132	diuron	C9H10Cl2N2O	232.0165	4.06	231.0097	291.0309	233.0243	250.0508	60	C3H6ON	72.0444	C6H4NCl2	159.9715	C5H3Cl2	132.9606
133	DMSA	C8H12N2O2S	200.0614	2.68	199.0547	259.0758	201.0692	218.0958	30	C6H6N	92.0495	C6H7N	93.0573	C8H13N2	137.1073
134	DMST	C9H14N2O2S	214.0770	3.11	213.0703	273.0915	215.0849	232.1114	50	C7H8N	106.0651	C6H7	79.0542	C7H9N	107.0730
135	dodine	C15H33N3O2	287.2567	7.17	286.2500	346.2711	288.2646	305.2911	10	C4H12O2N	106.0863	C4H10ON	88.0757	C14H23	191.1794
136	EPN	C14H14NO4PS	323.0376	7.11	322.0308	382.0520	324.0454	341.0719	30	C2H8O3PS	142.9926	C6H9	81.0699	C6H6OPS	156.9871
137	epoxiconazole	C17H13ClFN3O	329.0726	5.60	328.0658	388.0870	330.0804	347.1069	30	C8H6F	121.0448	C7H4OF	123.0241	C7H6OCl	141.0102
138	ethiofencarb	C11H15NO2S	225.0818	4.64	224.0751	284.0962	226.0896	243.1162	40	C8H9O	121.0648	C9H13OS	169.0682	C8H10O	122.0726
139	ethion	C9H22O4P2S4	383.9871	7.90	382.9803	443.0015	384.9949	402.0214	10	C5H12O2PS2	199.0011	C3H8O2PS2	170.9698	C6H16O2PS2	215.0324
140	ethirimol	C11H19N3O	209.1523	2.72	208.1455	268.1667	210.1601	227.1866	70	C8H14ON	140.1070	C5H8ON	98.0600	C9H13ON2	165.1022
141	ethofumesate	C13H18O5S	286.0869	4.49	285.0802	345.1013	287.0948	304.1213	10	C13H19O5S	287.0948	C11H13O4S	241.0529	C11H15O5S	259.0635
142	ethoprophos	C8H19O2PS2	242.0559	5.54	241.0491	301.0703	243.0637	260.0902	30	C3H10O2PS2	172.9854	H4O2PS2	130.9385	C3H8O2PS	138.9977
143	etofenprox	C25H28O3	376.2033	9.71	375.1966	435.2177	377.2111	394.2377	30	C12H17O	177.1274	C9H11O	135.0804	C13H17O	189.1274
144	etrimfos	C10H17N2O4PS	292.0641	6.15	291.0574	351.0785	293.0719	310.0985	40	C2H8O3PS	142.9926	C8H14O4N2PS	265.0406	C2H6O2PS	124.9821
145	famoxadone	C22H18N2O4	374.1261	6.43	373.1194	433.1405	375.1339	392.1605	20	C21H19O2N2	331.1441	C15H14ON2	238.1101	C15H13ON2	237.1022
146	fenamiphos	C13H22NO3PS	303.1053	5.78	302.0985	362.1197	304.1131	321.1396	30	C8H10O3PS	217.0083	C8H13O3NPS	234.0348	C11H19O3NPS	276.0818
147	fenamiphos sulphone	C13H22NO5PS	335.0951	3.10	334.0884	394.1095	336.1029	353.1295	30	C8H13O5NPS	266.0247	C11H19O5NPS	308.0716	C7H11O3NP	188.0471
148	fenamiphos-sulfoxide	C13H22NO4PS	319.1002	3.03	318.0934	378.1146	320.1080	337.1345	30	C11H19O4NPS	292.0767	C8H11O2S	171.0474	C8H13O4NPS	250.0297
149	fenarimol	C17H12Cl2N2O	330.0321	5.42	329.0254	389.0465	331.0399	348.0665	50	C4H5N2	81.0447	C16H11ONCl	268.0524	C7H4OCl	138.9945
150	fenazaquin	C20H22N2O	306.1727	9.37	305.1659	365.1871	307.1805	324.2070	40	C4H9	57.0699	C12H17	161.1325	C8H7ON2	147.0553
151	fenbuconazole	C19H17ClN4	336.1136	5.72	335.1069	395.1280	337.1215	354.1480	30	C7H6Cl	125.0153	C9H9N3Cl	194.0480	C7H7	91.0542
152	fenbutatin-oxide	C60H78OSn2	1054.4080	10.58	517.1922	577.2133	519.2068	536.2333	40	C7H7	91.0542	C18H15Sn	351.0190	C13H13Sn	289.0034
153	fenhexamid	C14H17Cl2NO2	301.0631	5.29	300.0564	360.0775	302.0709	319.0975	40	C7H13	97.1012	C6H6ONCl2	177.9821	C6H5ONCl	142.0054
154	fenoprop	C9H7Cl3O3	267.9455	2.79	266.9388	326.9599	268.9534	285.9799	50	C6H2OCl3	194.9177	C6HOCl2	158.9410	-	-

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
155	fenoxaprop	C16H12ClNO5	333.0399	5.80	332.0331	392.0543	334.0477	351.0742	50	C7H7	91.0542	C8H9O	121.0648	C13H9O3NCl	262.0265
156	fenoxaprop-ethyl	C18H16ClNO5	361.0712	7.45	360.0644	420.0856	362.0790	379.1055	30	C15H11O3NCl	288.0422	C8H9O	121.0648	C8H7O	119.0491
157	fenoxycarb	C17H19NO4	301.1309	5.89	300.1241	360.1453	302.1387	319.1652	30	C3H6O2N	88.0393	C5H10O2N	116.0706	C15H14O3N	256.0968
158	fenpropathrin	C22H23NO3	349.1672	8.59	348.1605	408.1816	350.1751	367.2016	20	C8H13O	125.0961	C14H10ON	208.0757	C22H24O3N	350.1751
159	fenpropidin	C19H31N	273.2451	4.44	272.2384	332.2595	274.2529	291.2795	70	C11H15	147.1168	C5H12N	86.0964	C9H11	119.0855
160	fenpropimorph	C20H33NO	303.2557	4.65	302.2489	362.2701	304.2635	321.2900	60	C11H15	147.1168	C6H12N	98.0964	C9H11	119.0855
161	fenpyroximate	C24H27N3O4	421.1996	8.69	420.1929	480.2140	422.2074	439.2340	20	C20H20O4N3	366.1448	C12H12ON3	214.0975	C12H13ON3	215.1053
162	fensulfothion	C11H17O4PS2	308.0300	3.88	307.0233	367.0444	309.0379	326.0644	30	C7H10O4PS2	252.9753	C9H14O4PS2	281.0066	C7H8O3PS2	234.9647
163	fenthion	C10H15O3PS2	278.0195	6.27	277.0127	337.0339	279.0273	296.0538	40	C8H5O3	149.0233	C8H9S2	169.0140	C8H9	105.0699
164	fenthion-sulfone	C10H15O5PS2	310.0093	3.32	309.0026	369.0237	311.0171	328.0437	40	C2H8O3PS	142.9926	C2H8O4P	128.0233	C8H9O2PS	200.0055
165	fenthion-sulfoxide	C10H15O4PS2	294.0144	3.22	293.0077	353.0288	295.0222	312.0488	40	C9H13O4PS2	279.9987	C10H15O3PS2	278.0195	C2H8O4P	127.0155
166	fipronil	C12H4Cl2F6N4OS	435.9382	5.76	434.9314	494.9526	436.9460	453.9725	30	C11H2ON4ClF3S	329.9595	C7H4OCl2F4	249.9581	C11H3N4Cl2F3	317.9692
167	flonicamid	C9H6F3N3O	229.0457	1.97	228.0390	288.0601	230.0536	247.0801	30	C3HON2	81.0094	C6H3NF3	146.0223	-	-
168	florasulam	C12H8F3N5O3S	359.0294	2.49	358.0227	418.0438	360.0373	377.0638	30	C6H5NF2	129.0385	C6H4O2NF2S	191.9925	C6H4ONF2	144.0255
169	fluacrypyrim	C20H21F3N2O5	426.1397	6.94	425.1330	485.1541	427.1475	444.1741	10	C12H13O3	205.0859	C10H9O	145.0648	-	-
170	fluazifop	C15H12F3NO4	327.0713	4.56	326.0646	386.0857	328.0791	345.1057	40	C14H11O2NF3	282.0736	C7H7	91.0542	C13H11ONF3	254.0787
171	fluazifop-P-butyl	C19H20F3NO4	383.1339	7.56	382.1272	442.1483	384.1417	401.1683	30	C14H11O2NF3	282.0736	C15H13O4NF3	328.0791	C13H11ONF3	254.0787
172	fluazinam	C13H4Cl2F6N4O4	463.9508	7.12	462.9441	522.9652	464.9587	481.9852	30	C13H2O2N3Cl2F6	415.9434	C13H3O3N3ClF6	397.9773	C13H3ON2Cl2F6	386.9532
173	flucythrinate	C26H23F2NO4	451.1590	8.40	450.1522	510.1734	452.1668	469.1933	20	C26H22O4N	412.1543	C11H13OF2	199.0929	C14H10ON	208.0757
174	fludioxonil	C12H6F2N2O2	248.0392	4.79	247.0325	307.0536	249.0470	266.0736	80	C11H4ON2	180.0329	C9H4N	126.0349	C10H5ON2	169.0407
175	flufenacet	C14H13F4N3O2S	363.0659	5.45	362.0592	422.0803	364.0737	381.1003	10	C11H13ONF	194.0976	C8H7ONF	152.0506	C10H13NF	166.1027
176	flufenoxuron	C21H11ClF6N2O3	488.0357	8.39	487.0290	547.0501	489.0435	506.0701	30	C13H7ONClF4	304.0158	C14H6ON2ClF4	329.0110	C13H6ONClF3	284.0095
177	flumioxazin	C19H15FN2O4	354.1010	4.16	353.0943	413.1154	355.1089	372.1354	20	C19H16O4N2F	355.1089	C18H16O3N2F	327.1139	-	-
178	fluopyram	C16H11ClF6N2O	396.0459	5.32	395.0391	455.0603	397.0537	414.0802	30	C8H6NClF3	208.0135	C8H4OF3	173.0209	C8H6ONF2	170.0412
179	fluoxastrobin	C21H16ClFN4O5	458.0788	5.36	457.0720	517.0932	459.0866	476.1132	20	C20H13O4N4ClF	427.0604	C10H5ON2F	188.0380	C18H9O2N4ClF	367.0393
180	fluquinconazole	C16H8Cl2FN5O	375.0084	5.28	374.0017	434.0228	376.0163	393.0428	40	C14H6ON2Cl2F	306.9836	C15H8ON4Cl2F	349.0054	C8H4ON2F	163.0302
181	fluroxypyr	C7H5Cl2FN2O3	253.9656	2.72	252.9588	312.9800	254.9734	272.0000	30	C6H4ON2Cl2F	208.9679	C5H4ON2Cl2F	196.9679	C5H4N2Cl2F	180.9730
182	flusilazole	C16H15F2N3Si	315.0998	5.90	314.0931	374.1142	316.1076	333.1342	40	C8H8N3F	165.0697	C8H9N3F	166.0775	C8H9N3F2	185.0759
183	flutriafol	C16H13F2N3O	301.1021	3.68	300.0954	360.1165	302.1099	319.1365	30	C2H4N3	70.0400	C7H4OF	123.0241	C14H11OF2	233.0772
184	fomesafen	C15H10ClF3N2O6S	437.9895	3.19	436.9827	497.0039	438.9973	456.0238	40	C7H3OClF3	194.9830	C2H2O2N	72.0091	C7H8NClF3	198.0303
185	fonofos	C10H15OPS2	246.0296	6.36	245.0229	305.0440	247.0375	264.0640	10	C4H10OPS	137.0184	C2H6OPS	108.9871	C2H8O2PS	126.9977
186	foramsulfuron	C17H20N6O7S	452.1109	3.16	451.1041	511.1253	453.1187	470.1452	20	C7H8O3N3	182.0560	C10H11O4N2S	255.0434	C10H14O4N3S	272.0700
187	formetanate	C11H16ClN3O2	257.0926	1.65	220.1092	280.1303	222.1237	239.1503	70	C9H13ON2	165.1022	C7H6ON	120.0444	C6H7O2	111.0441
188	formothion	C6H12NO4PS2	256.9940	2.70	255.9873	316.0084	258.0018	275.0284	10	C4H8O3PS2	198.9647	C3H8O2PS2	170.9698	C4H6O2N	100.0393
189	fosthiazate	C9H18NO3PS2	283.0460	3.47	282.0393	342.0604	284.0538	301.0804	10	C5H11O3NPS2	227.9912	C3H6ONS	104.0165	C3H7O3NPS2	199.9599
190	furathiocarb	C18H26N2O5S	382.1557	7.50	381.1490	441.1701	383.1635	400.1901	30	C10H11O2S	195.0474	C9H11OS	167.0525	C10H12O2	164.0832
191	haloxyfop	C15H11ClF3NO4	361.0323	5.88	360.0256	420.0467	362.0401	379.0667	30	C14H10O2NClF3	316.0347	C13H10ONClF3	288.0398	C7H7	91.0542
192	haloxyfop-2-ethoxyethyl	C19H19ClF3NO5	433.0898	7.45	432.0831	492.1042	434.0977	451.1242	30	C14H10O2NClF3	316.0347	C4H9O	73.0648	C13H10ONClF3	288.0398
193	haloxyfop-methyl	C16H13ClF3NO4	375.0480	6.95	374.0412	434.0624	376.0558	393.0823	40	C7H7	91.0542	C14H10O2NClF3	316.0347	C13H10ONClF3	288.0398
194	heptenophos	C9H12ClO4P	250.0156	4.03	249.0089	309.0300	251.0234	268.0500	30	C2H8O4P	127.0155	C7H6Cl	125.0153	C7H5	89.0386

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
195	hexaconazole	C14H17Cl2N3O	313.0743	6.45	312.0676	372.0887	314.0821	331.1087	30	C4H6O	70.0413	C7H5Cl2	158.9763	C9H7Cl2	184.9919
196	hexazinone	C12H20N4O2	252.1581	3.15	251.1513	311.1725	253.1659	270.1925	40	C6H11O2N4	171.0877	C3H7N2	71.0604	C4H9N2	85.0760
197	hexythiazox	C17H21ClN2O2S	352.1007	8.08	351.0939	411.1151	353.1085	370.1351	30	C10H11ONClS	228.0244	C9H11NCl	168.0575	C10H9ONCl	194.0367
198	chlorantraniliprole	C18H14BrCl2N5O2	480.9702	4.10	481.9615	541.9826	483.9760	501.0026	20	C17H10O2N4[81]BrCl2	452.9338	C9H4ON3[81]BrCl	285.9200	C9H4ON3BrCl	283.9221
199	chlorfenvinphos	C12H14Cl3O4P	357.9690	6.36	356.9623	416.9834	358.9768	376.0034	20	C4H12O4P	155.0468	C2H8O4P	127.0155	C7H4OCl2P	204.9371
200	chloridazon	C10H8ClN3O	221.0350	2.31	220.0283	280.0494	222.0429	239.0694	70	C6H6N	92.0495	C7H6N	104.0495	C5H5	65.0386
201	chlorotoluron	C10H13ClN2O	212.0711	3.68	211.0644	271.0855	213.0789	230.1055	50	C3H6ON	72.0444	C7H7NCl	140.0262	C8H7ONCl	168.0211
202	chloroxuron	C15H15ClN2O2	290.0817	5.30	289.0749	349.0961	291.0895	308.1160	50	C3H6ON	72.0444	C12H9ONCl	218.0367	C8H8N	118.0651
203	chlorpyrifos-methyl	C7H7Cl3NO3PS	320.8944	7.00	319.8877	379.9088	321.9023	338.9288	40	C2H8O3PS	142.9926	C6H4O2NCl3PS	289.8760	C3H8O3Cl	127.0156
204	chlorsulfuron	C12H12O4N5SCl	357.0293	3.20	356.0226	416.0437	358.0371	375.0637	30	C5H9ON4	141.0771	C6H7O2N4	167.0564	C3H6N	56.0495
205	imazalil	C14H14Cl2N2O	296.0478	3.75	295.0410	355.0622	297.0556	314.0821	50	C7H5Cl2	158.9763	C3H5N2	69.0447	C8H7Cl2	172.9919
206	Imazamethabenz-methyl	C16H20N2O3	288.1468	2.99	287.1401	347.1612	289.1547	306.1812	50	C5H12N	86.0964	C9H9ON2	161.0709	C9H6ON	144.0444
207	imazamox	C15H19N3O4	305.1370	2.31	304.1303	364.1514	306.1448	323.1714	50	C9H9O3N2	193.0608	C5H9	69.0699	C5H12N	86.0964
208	imazapyr	C13H15N3O3	261.1108	2.13	260.1041	320.1252	262.1186	279.1452	40	C12H13O2N2	217.0972	C10H10O3N3	220.0717	C10H8O2N3	202.0611
209	imazaquin	C17H17N3O3	311.1264	3.00	310.1197	370.1408	312.1343	329.1608	50	C11H7O2N2	199.0502	C5H12N	86.0964	C5H12N	86.0964
210	imazethapyr	C15H19N3O3	289.1421	2.72	288.1354	348.1565	290.1499	307.1765	50	C9H9O2N2	177.0659	C12H12O2N3	230.0924	C5H12N	86.0964
211	imazosulfuron	C14H13ClN6O5S	412.0351	4.80	411.0284	471.0495	413.0429	430.0695	20	C6H10O2N3	156.0768	C7H6N2Cl	153.0214	C7H7O2N3ClS	231.9942
212	imidacloprid	C9H10ClN5O2	255.0518	2.14	254.0450	314.0662	256.0596	273.0861	30	C9H10N4Cl	209.0589	C9H11N4	175.0978	C3H6N3	84.0556
213	indoxacarb	C22H17ClF3N3O7	527.0702	7.08	526.0634	586.0846	528.0780	545.1045	20	C9H7O2NF3	218.0423	C12H10O2N2Cl	249.0425	C8H5NCl	150.0105
214	iodosulfuron-methyl	C14H14IN5O6S	506.9704	4.09	505.9637	565.9848	507.9782	525.0048	30	C6H7O2N4	167.0564	C5H9ON4	141.0771	C8H6O4IS	324.9026
215	ioxynil	C7H3I2NO	370.8299	2.28	369.8231	429.8443	371.8377	388.8642	50	I	126.9050	C7H2ON	116.0142	C6H2ONI	230.9187
216	iprovalicarb	C18H28N2O3	320.2094	5.30+5.23	319.2027	379.2238	321.2173	338.2438	10	C9H11	119.0855	C9H19O3N2	203.1390	C9H16O3N	186.1125
217	isofenphos	C15H24NO4PS	345.1158	6.61	344.1091	404.1302	346.1236	363.1502	30	C7H6O4PS	216.9719	C9H10O4PS	245.0032	C8H8O2PS	198.9977
218	isofenphos-methyl	C14H22NO4PS	331.1002	6.02	330.0934	390.1146	332.1080	349.1345	50	C7H5O2	121.0284	C8H8O4PS	230.9875	C7H6O4PS	216.9719
219	isoprocarb	C11H15NO2	193.1097	3.72	192.1030	252.1241	194.1176	211.1441	20	C6H7O	95.0491	C9H13O	137.0961	C8H10O2N	152.0706
220	isoprothiolane	C12H18O4S2	290.0641	4.95	289.0574	349.0785	291.0719	308.0985	10	C9H11O3S2	231.0144	C6H5O3S2	188.9675	C9H13O4S2	249.0250
221	isoproturon	C12H18N2O	206.1414	4.90	205.1346	265.1558	207.1492	224.1757	50	C3H6ON	72.0444	C9H13ON2	165.1022	C9H12N	134.0964
222	jasmolin I	C21H30O3	330.2189	8.97	329.2122	389.2333	331.2268	348.2533	10	C11H15O	163.1117	C10H15O	151.1117	-	-
223	jasmolin II	C22H30O5	374.2088	7.63	373.2020	433.2232	375.2166	392.2431	10	C11H15O	163.1117	C11H17O4	213.1121	-	-
224	kresoxim-methyl	C18H19NO4	313.1309	6.00	312.1241	372.1453	314.1387	331.1652	10	C15H12ON	222.0913	C17H15O3	267.1016	C16H11O2	235.0754
225	lambda-cyhalothrin	C23H19ClF3NO3	449.1000	8.79	448.0933	508.1144	450.1078	467.1344	10	C9H9OClF3	225.0289	C23H20O3NClF3	450.1078	C22H19O3ClF3	423.0969
226	lenacil	C13H18N2O2	234.1363	3.77	233.1296	293.1507	235.1441	252.1707	70	C7H9O2N2	153.0659	C7H6O2N	136.0393	C7H7ON2	135.0553
227	linuron	C9H10Cl2N2O2	248.0114	4.65	247.0047	307.0258	249.0192	266.0458	40	C6H4NCl2	159.9715	C8H7ON2Cl	182.0241	C6H5NCl2	160.9794
228	lufenuron	C17H8Cl2F8N2O3	509.9779	8.03	508.9711	568.9923	510.9857	528.0122	30	C9H4ONCl2F6	325.9580	C6H3ONCl2	174.9597	C7H2O2NCl2	201.9468
229	malaoxon	C10H19O7PS	314.0584	3.03	313.0516	373.0728	315.0662	332.0927	10	C6H7O3	127.0390	C4H3O3	99.0077	C2H8O3PS	142.9926
230	malathion	C10H19O6PS2	330.0355	4.89	329.0288	389.0499	331.0433	348.0699	10	C8H14O5PS2	285.0015	C6H7O3	127.0390	C10H20O6PS2	331.0433
231	mandipropamide	C23H22ClNO4	411.1232	4.84	410.1165	470.1376	412.1310	429.1576	20	C19H19O2NCl	328.1099	C12H14O2N	204.1019	C20H19O3NCl	356.1048
232	MCPA	C9H9ClO3	200.0235	2.23	199.0167	259.0379	201.0313	218.0578	40	C7H6OCl	141.0113	C6H4OCl	126.9956	C8H8OCl	155.0269
233	MCPB	C11H13ClO3	228.0548	3.12	227.0480	287.0692	229.0626	246.0891	40	C7H6OCl	141.0113	-	-	-	-
234	mecarbam	C10H20NO5PS2	329.0515	5.40	328.0448	388.0659	330.0593	347.0859	20	C6H12O3PS2	226.9960	C5H12O2PS2	199.0011	C4H6O3N	116.0342

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
235	mecoprop	C10H11ClO3	214.0391	2.45	213.0324	273.0535	215.0469	232.0735	40	C7H6OCl	141.0113	C3H3O2	71.0139	-	-
236	mefenpyr-diethyl	C16H18Cl2N2O4	372.0638	6.40	371.0571	431.0782	373.0716	390.0982	40	C14H13O3N2Cl2	327.0298	C6H4NCl2	159.9715	C12H9O3N2Cl2	298.9985
237	mepanipyrim	C14H13N3	223.1104	5.56	222.1037	282.1248	224.1182	241.1448	70	C7H8N	106.0651	C9H11N	133.0886	C6H7	79.0542
238	mepronil	C17H19NO2	269.1410	4.92	268.1343	328.1554	270.1489	287.1754	40	C8H7O	119.0491	C14H14O2N	228.1019	C7H6O2N	136.0393
239	metaflumizone	C24H16F6N4O2	506.1172	7.85	505.1105	565.1316	507.1250	524.1516	40	C16H11N3F3	302.0911	C16H8N2F3	285.0645	C7H5N2	117.0458
240	metalaxyl	C15H21NO4	279.1465	3.85	278.1398	338.1609	280.1543	297.1809	30	C12H18ON	192.1383	C13H18O2N	220.1332	C11H14N	160.1121
241	metamitron	C10H10N4O	202.0849	2.31	201.0782	261.0993	203.0927	220.1193	50	C9H11N4	175.0978	C7H6N	104.0495	C10H10ON2	174.0788
242	metazachlor	C14H16ClN3O	277.0976	3.79	276.0909	336.1120	278.1055	295.1320	20	C11H13ONCl	210.0680	C9H12N	134.0964	-	-
243	metconazole	C17H22ClN3O	319.1446	6.49	318.1379	378.1590	320.1524	337.1790	40	C4H6O	70.0413	C7H6Cl	125.0153	C11H10Cl	177.0466
244	methacriphos	C7H13O5PS	240.0216	4.15	239.0149	299.0360	241.0294	258.0560	30	C2H8O3PS	142.9926	C6H10O4PS	209.0032	C2H6O2PS	124.9821
245	methamidophos	C2H8NO2PS	141.0008	1.49	139.9941	200.0152	142.0086	159.0352	50	C2H6O2PS	124.9821	CH4O2P	78.9943	CH5ONPS	109.9824
246	methidathion	C6H11N2O4PS3	301.9613	4.09	300.9546	360.9757	302.9691	319.9957	30	C4H5O2N2S	145.0066	C3H5ON2	85.0396	C2H3ON2	71.0240
247	methiocarb	C11H15NO2S	225.0818	4.64	224.0751	284.0962	226.0896	243.1162	30	C9H13OS	169.0682	C8H9O	121.0648	C8H10O	122.0726
248	methiocarb sulfoxide	C11H15NO3S	241.0767	2.18	240.0700	300.0911	242.0845	259.1111	40	C9H13O2S	185.0631	C8H10O2S	170.0396	C9H12OS	168.0603
249	methiocarb-sulfone	C11H15NO4S	257.0716	2.29	256.0649	316.0860	258.0795	275.1060	10	C9H13O3S	201.0580	C11H16O4NS	258.0795	C8H10O	122.0726
250	metholcarb	C9H11NO2	165.0784	2.83	164.0717	224.0928	166.0863	183.1128	30	C7H9O	109.0648	C6H9	80.0621	-	-
251	methomyl	C5H10N2O2S	162.0457	1.96	161.0390	221.0602	163.0536	180.0801	20	C3H6NS	88.0215	C3H8ONS	106.0321	CH5OS	65.0056
252	methoxyfenozide	C22H28N2O3	368.2094	5.03	367.2027	427.2238	369.2173	386.2438	10	C9H9O2	149.0597	C18H21O3N2	313.1547	C9H9O	133.0648
253	metobromuron	C9H11BrN2O2	257.9998	3.77	256.9931	317.0142	259.0077	276.0342	40	C6H5NBr	169.9600	C8H8ON2	148.0631	C6H6NBr	170.9678
254	metolachlor	C15H22ClNO2	283.1334	5.63	282.1266	342.1478	284.1412	301.1677	40	C14H19ONCl	252.1150	C12H18N	176.1434	C11H15ONCl	212.0837
255	metosulam	C14H13Cl2N5O4S	417.0060	3.09	415.9993	476.0204	418.0138	435.0404	60	C7H7NCl2	174.9950	C7H7NCl	140.0262	C7H6NCl2	173.9872
256	metoxuron	C10H13ClN2O2	228.0660	2.65	227.0593	287.0804	229.0738	246.1004	50	C3H6ON	72.0444	C7H7ONCl	156.0211	C8H7O2N	149.0471
257	metribuzin	C8H14N4OS	214.0883	3.04	213.0816	273.1027	215.0961	232.1227	60	C7H15N4S	187.1012	C3H6O	58.0413	C5H10N	84.0808
258	metsulfuron-methyl	C14H15N5O6S	381.0738	2.94	380.0670	440.0882	382.0816	399.1081	30	C6H7O2N4	167.0564	C8H7O4S	199.0060	C5H9ON4	141.0771
259	mevinphos	C7H13O6P	224.0444	2.44	223.0377	283.0588	225.0523	242.0788	20	C6H10O5P	193.0260	C2H8O4P	127.0155	C5H7O2	99.0441
260	monocrotophos	C7H14NO5P	223.0604	1.99	222.0537	282.0748	224.0682	241.0948	10	C2H8O4P	127.0155	C5H8ON	98.0600	C6H10O5P	193.0260
261	monolinuron	C9H11ClN2O2	214.0504	3.51	213.0436	273.0648	215.0582	232.0847	50	C6H5NCl	126.0105	C8H8ON2	148.0631	C6H6NCl	127.0183
262	monuron	C9H11ClN2O	198.0554	3.03	197.0487	257.0698	199.0633	216.0898	70	C3H6ON	72.0444	C6H5NCl	126.0105	C5H4Cl	98.9996
263	myclobutanil	C15H17ClN4	288.1136	5.10	287.1069	347.1280	289.1215	306.1480	50	C2H4N3	70.0400	C7H6Cl	125.0153	C9H8Cl	151.0309
264	naled	C4H7Br2Cl2O4P	377.7820	4.05	376.7753	436.7964	378.7899	395.8164	30	C2H8O4P	127.0155	C2HBr2Cl2	252.7817	-	-
265	napropamide	C17H21NO2	271.1567	5.60	270.1500	330.1711	272.1645	289.1911	30	C7H15ON	129.1148	C12H11O	171.0804	C13H11O2	199.0754
266	neburon	C12H16Cl2N2O	274.0634	5.93	273.0567	333.0778	275.0712	292.0978	30	C5H14N	88.1121	C6H12ON	114.0913	C4H9	57.0699
267	nicosulfuron	C15H18N6O6S	410.1003	3.35	409.0936	469.1147	411.1081	428.1347	20	C7H8O3N3	182.0560	C8H9O3N2S	213.0328	C13H12O6N5S	366.0503
268	norflurazone	C12H9ClF3N3O	303.0381	4.01	302.0313	362.0525	304.0459	321.0724	60	C12H9ON3ClF2	284.0397	C7H5NF3	160.0369	C7H4NF2	140.0306
269	omethoate	C5H12NO4PS	213.0219	1.71	212.0152	272.0363	214.0297	231.0563	30	C4H8O4PS	182.9875	C2H8O3PS	142.9926	C3H8O3PS	154.9926
270	oxadixyl	C14H18N2O4	278.1261	2.69	277.1194	337.1405	279.1339	296.1605	10	C12H15O2N2	219.1128	C4H8O2N	102.0550	C8H5O3	149.0233
271	oxamyl	C7H13N3O3S	219.0672	1.84	218.0605	278.0816	220.0750	237.1016	20	C3H6ON	72.0444	C3H8O2N	90.0550	-	-
272	oxydemeton-methyl	C6H15O4PS2	246.0144	1.91	245.0077	305.0288	247.0222	264.0488	30	C4H10O3PS	169.0083	C4H9OS	105.0369	C2H8O3PS	142.9926
273	oxyfluorfen	C15H11ClF3NO4	361.0323	5.87	360.0256	420.0467	362.0401	379.0667	30	C14H10O2NClF3	316.0347	C13H10ONClF3	288.0398	C7H7	91.0542
274	paclobutrazol	C15H20ClN3O	293.1289	4.86	292.1222	352.1433	294.1368	311.1633	60	C2H4N3	70.0400	C7H6Cl	125.0153	C8H8Cl	139.0309

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
275	penconazole	C13H15Cl2N3	283.0638	6.19	282.0570	342.0782	284.0716	301.0981	50	C2H4N3	70.0400	C7H5Cl2	158.9763	C8H7Cl2	172.9919
276	pencycuron	C19H21ClN2O	328.1337	6.80	327.1270	387.1481	329.1415	346.1681	30	C7H6Cl	125.0153	C13H13NCl	218.0731	C14H14ON2Cl	261.0789
277	pendimethalin	C13H19N3O4	281.1370	8.25	280.1303	340.1514	282.1448	299.1714	30	C8H10O4N3	212.0666	C8H8O3N3	194.0560	C8H9O3N3	195.0638
278	permethrin	C21H20Cl2O3	390.0784	9.65	389.0717	449.0928	391.0862	408.1128	10	C13H11O	183.0804	C21H20O3Cl	355.1095	C21H19O3	319.1329
279	phenmedipham	C16H16N2O4	300.1105	3.98+4.12	299.1037	359.1249	301.1183	318.1448	30	C9H12O3N	182.0812	C7H6O2N	136.0393	C7H8O3N	154.0499
280	phenothrin	C23H26O3	350.1876	9.61	349.1809	409.2020	351.1955	368.2220	30	C13H11O	183.0804	C17H17O	237.1274	C18H17O	249.1274
281	phenthoate	C12H17O4PS2	320.0300	5.98	319.0233	379.0444	321.0379	338.0644	20	C10H11O2	163.0754	C9H12O2PS2	247.0011	C9H11O	135.0804
282	phorate	C7H17O2PS3	260.0123	6.61	259.0056	319.0267	261.0201	278.0467	30	C3H7S	75.0263	CH4O2PS2	142.9385	C3H8O2PS2	170.9698
283	phorate-sulfone	C7H17O4PS3	292.0021	3.64	290.9954	351.0165	293.0099	310.0365	20	C4H12O3PS	171.0239	C6H16O4PS2	247.0222	C5H12O2PS2	199.0011
284	phorate-sulfoxide	C7H17O3PS3	260.0300	3.57	275.0005	335.0216	277.0150	294.0416	10	C5H12O2PS2	199.0011	C3H8O2PS2	170.9698	CH4O2PS2	142.9385
285	phosalone	C12H15ClNO4PS2	366.9863	6.64	365.9796	426.0007	367.9941	385.0207	10	C12H16O4NClPS2	367.9941	C8H5O2NCl	182.0003	C11H14O4NClPS	322.0064
286	phosphamidon	C10H19ClNO5P	299.0684	2.69	298.0617	358.0828	300.0762	317.1028	30	C8H13ONCl	174.0680	C6H9O5ClP	226.9871	C2H8O4P	127.0155
287	phoxim	C12H15N2O3PS	298.0536	6.58	297.0468	357.0680	299.0614	316.0879	20	C8H5N2	129.0447	H4O3PS	114.9613	H2O2PS	96.9508
288	picloram	C6H3Cl3N2O2	239.9255	1.92	238.9187	298.9399	240.9333	257.9598	60	C5H4ON2Cl3	212.9384	C4HNCl3	167.9169	C5H2N2Cl3	194.9278
289	picolinafen	C19H12F4N2O2	376.0829	7.95	375.0762	435.0973	377.0908	394.1173	30	C12H9O2NF3	256.0580	C19H11ON2F4	359.0802	C13H9O3NF3	284.0529
290	picoxystrobin	C18H16F3NO4	367.1026	5.92	366.0959	426.1170	368.1104	385.1370	10	C12H13O3	205.0859	C10H9O	145.0648	C11H9O2	173.0597
291	pinoxaden	C23H32N2O4	400.2357	6.66	399.2289	459.2501	401.2435	418.2700	50	C18H25O3N2	317.1860	C16H21O3N2	289.1547	C4H9ON2	101.0709
292	piperonyl butoxide	C19H30O5	338.2088	7.75	337.2020	397.2232	339.2166	356.2431	50	C9H11	119.0855	C11H13O2	177.0910	C10H11O	147.0804
293	pirimicarb	C11H18N4O2	238.1424	2.90	237.1357	297.1568	239.1503	256.1768	60	C3H6ON	72.0444	C9H16ON3	182.1288	C4H9N2	85.0760
294	pirimicarb-desmethyl	C10H16N4O2	224.1268	2.19	223.1200	283.1412	225.1346	242.1612	40	C3H6ON	72.0444	C8H14ON3	168.1131	C8H10O2N3	180.0768
295	pirimiphos-ethyl	C13H24N3O3PS	333.1271	7.62	332.1203	392.1415	334.1349	351.1614	40	C9H16N3S	198.1059	C9H16ON3	182.1288	C7H12N3S	170.0746
296	pirimiphos-methyl	C11H20N3O3PS	305.0958	6.49	304.0890	364.1102	306.1036	323.1301	40	C9H14N3	164.1182	C5H6N3	108.0556	C7H10N3	136.0869
297	profenofos	C11H15BrClO3PS	371.9346	7.47	370.9279	430.9490	372.9424	389.9690	30	C6H6O3BrClPS	302.8642	C5H5OBrPS	222.8977	-	-
298	prochloraz	C15H16Cl3N3O2	375.0303	6.55	374.0235	434.0447	376.0381	393.0646	20	C12H13O2NCl3	308.0006	C9H7O2NCl3	265.9537	C11H13ONCl3	280.0057
299	prometon	C10H19N5O	225.1584	3.59	224.1517	284.1728	226.1662	243.1928	60	C4H8ON5	142.0723	C7H14ON5	184.1193	C2H4ON3	86.0349
300	prometryn	C10H19N5S	241.1356	4.81+4.97	240.1288	300.1500	242.1434	259.1699	60	C4H8N5S	158.0495	C7H14N5S	200.0964	C3H6N3S	116.0277
301	propachlor	C11H14ClNO	211.0758	3.91	210.0691	270.0902	212.0837	229.1102	50	C8H9ONCl	170.0367	C6H8N	94.0651	C7H8N	106.0651
302	propamocarb	C9H20N2O2	188.1519	1.76	187.1452	247.1663	189.1598	206.1863	40	C4H8O2N	102.0550	C7H14O2N	144.1019	C2H4O2N	74.0237
303	propaquizafop	C22H22ClN3O5	443.1243	7.63	442.1175	502.1387	444.1321	461.1586	20	C5H10ON	100.0757	C19H16O4N2Cl	371.0793	C16H12O2N2Cl	299.0582
304	propargite	C19H26O4S	350.1546	8.34	349.1479	409.1690	351.1625	368.1890	10	C16H23O	231.1743	C12H15O	175.1117	C7H7O	107.0491
305	propazine	C9H16ClN5	229.1089	4.56+4.77	228.1021	288.1233	230.1167	247.1432	50	C3H5N5Cl	146.0228	C6H11N5Cl	188.0697	C2H3N3Cl	104.0010
306	propham	C10H13NO2	179.0941	3.69	178.0874	238.1085	180.1019	197.1285	40	C7H6ON	120.0444	C7H8O2N	138.0550	C6H6N	92.0495
307	propiconazole	C15H17Cl2N3O2	341.0692	6.36+6.45	340.0625	400.0836	342.0771	359.1036	40	C7H5Cl2	158.9763	C8H5OCl2	186.9712	C7H5O2Cl2	190.9661
308	propoxur	C11H15NO3	209.1046	3.00	208.0979	268.1190	210.1125	227.1390	20	C6H7O2	111.0441	C8H10O3N	168.0655	C9H13O2	153.0910
309	propoxycarbazone	C15H18N4O7S	398.0891	2.75	397.0823	457.1035	399.0969	416.1234	20	C6H12O2N3	158.0924	C8H7O4S	199.0060	C14H15O6N4S	367.0707
310	propyzamide	C12H11Cl2NO	255.0212	4.91	254.0145	314.0356	256.0290	273.0556	30	C10H8ONCl2	227.9988	C6H3Cl2	144.9617	C8H5OCl2	186.9723
311	proquinazid	C14H17IN2O2	372.0329	8.81	371.0262	431.0473	373.0407	390.0673	40	C8H6O2N2I	288.9468	C8H3O2NI	271.9203	C11H12O2N2I	330.9938
312	prosulfocarb	C14H21NOS	251.1338	7.31	250.1271	310.1482	252.1417	269.1682	30	C7H7	91.0542	C7H14ON	128.1070	C4H8ON	86.0600
313	prothioconazole-desthio	C14H15Cl2N3O	311.0587	5.64	310.0519	370.0731	312.0665	329.0930	40	C4H6O	70.0413	C7H6Cl	125.0153	C8H8Cl	139.0309
314	prothiofos	C11H15Cl2O2PS2	343.9623	9.26	342.9555	402.9767	344.9701	361.9966	20	C6H6O2Cl2PS2	274.8918	C9H12O2Cl2PS2	316.9388	-	-

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
315	pyraclostrobin	C19H18ClN3O4	387.0980	6.56	386.0913	446.1124	388.1059	405.1324	10	C10H12O3N	194.0812	C16H11ON3Cl	296.0585	C9H10O2N	164.0706
316	pyrazophos	C14H20N3O5PS	373.0856	6.61	372.0789	432.1000	374.0934	391.1200	40	C10H12O3N3	222.0873	C8H8O3N3	194.0560	C10H12O2N3S	238.0645
317	pyrethrin I	C21H28O3	328.2033	8.54	327.1966	387.2177	329.2111	346.2377	20	C11H13O	161.0961	C10H13	133.1012	C11H11	143.0855
318	pyrethrin II	C22H28O5	372.1931	7.11	371.1864	431.2075	373.2010	390.2275	10	C11H13O	161.0961	C10H13	133.1012	C11H11	143.0855
319	pyridaben	C19H25ClN2OS	364.1371	9.06	363.1303	423.1515	365.1449	382.1714	20	C15H18ON2ClS	309.0823	C11H15	147.1168	-	-
320	pyridate	C19H23ClN2O2S	378.1163	9.54	377.1096	437.1307	379.1242	396.1507	10	C10H8ON2Cl	207.0320	C18H24ON2ClS	351.1292	C18H24ON2Cl	319.1572
321	pyrifenox	C14H12Cl2N2O	294.0321	5.02+5.34	293.0254	353.0465	295.0399	312.0665	40	C6H7N	93.0573	C13H9N2Cl2	263.0137	C6H6N	92.0495
322	pyrimethanil	C12H13N3	199.1104	4.63	198.1037	258.1248	200.1182	217.1448	90	C5H8N	82.0651	C5H6N	80.0495	C12H10N2	182.0838
323	pyriproxyfen	C20H19NO3	321.1359	7.99	320.1292	380.1503	322.1438	339.1703	30	C5H6ON	96.0444	C12H9O2	185.0597	C15H15O2	227.1067
324	quinalphos	C12H15N2O3PS	298.0536	6.04	297.0468	357.0680	299.0614	316.0879	50	C8H7ON2	147.0553	C8H7N2S	163.0324	H4O3PS	114.9613
325	quinclorac	C10H5Cl2NO2	240.9692	2.53	239.9625	299.9836	241.9770	259.0036	60	C10H4ONCl2	223.9664	C9H6ONCl2	213.9821	C9H4NCl	161.0027
326	quinmerac	C11H8ClNO2	221.0238	2.34	220.0171	280.0382	222.0316	239.0582	60	C11H7ONCl	204.0211	C10H7NCl	176.0262	C10H7N	141.0573
327	quinoclamine	C10H6ClNO2	207.0082	3.00	206.0014	266.0226	208.0160	225.0425	70	C7H5O	105.0335	C8H6N	116.0495	C9H6ON	144.0444
328	quinoxyfen	C15H8Cl2FNO	306.9961	8.32	305.9894	366.0106	308.0040	325.0305	70	C9H6ONCl2	213.9821	C9H5NCl	162.0105	C15H8ONClF	272.0273
329	quizalofop	C17H13ClN2O4	344.0558	5.63	343.0491	403.0702	345.0637	362.0902	40	C16H12O2N2Cl	299.0582	C15H12ON2Cl	271.0633	C14H9O2N2Cl	272.0347
330	quizalofop-p-ethyl	C19H17ClN2O4	372.0871	7.43	371.0804	431.1015	373.0950	390.1215	40	C16H12O2N2Cl	299.0582	C15H12ON2Cl	271.0633	C7H7	91.0542
331	resmethrin	C22H26O3	338.1876	9.27	337.1809	397.2020	339.1955	356.2220	40	C11H11	143.0855	C12H11O	171.0804	C7H7	91.0542
332	rimsulfuron	C14H17N5O7S2	431.0564	3.24	430.0497	490.0708	432.0642	449.0908	20	C7H8O3N3	182.0560	C13H17O4N4S	325.0965	C7H11O4N2S2	251.0155
333	rotenone	C23H22O6	394.1411	5.80	393.1344	453.1555	395.1489	412.1755	40	C14H13O2	213.0910	C11H12O3	192.0781	C12H11O3	203.0703
334	simazine	C7H12ClN5	201.0776	3.13+3.32	200.0708	260.0920	202.0854	219.1119	60	C4H7N3Cl	132.0323	C4H6N3	96.0556	C2H3N3Cl	104.0010
335	simetryn	C8H15N5S	213.1043	3.36	212.0975	272.1187	214.1121	231.1386	60	C5H10N5S	172.0651	C3H4N3	82.0400	C2H7N2S	91.0324
336	spinosyn A	C41H65NO10	731.4603	6.92	730.4536	790.4747	732.4681	749.4947	30	C8H16ON	142.1226	C8H16ON	142.1226	C6H9O	97.0648
337	spinosyn D	C42H67NO10	745.4759	7.42	744.4692	804.4903	746.4838	763.5103	30	C8H16ON	142.1226	C8H16ON	142.1226	C6H9O	97.0648
338	spirodiclofen	C21H24Cl2O4	410.1046	8.65	409.0979	469.1190	411.1124	428.1390	30	C5H11	71.0855	C15H15O3Cl2	313.0393	C19H12O4Cl2	374.0107
339	spiromesifen	C23H30O4	370.2139	8.33	369.2071	429.2283	371.2217	388.2482	40	C17H21O3	273.1485	C17H19O2	255.1380	C12H11O2	187.0754
340	spiroxamine	C18H35NO2	297.2662	4.90+5.03	296.2595	356.2806	298.2741	315.3006	40	C8H18ON	144.1383	C6H14N	100.1121	C4H10N	72.0808
341	sulfosulfuron	C16H18N6O7S2	470.0673	4.27	469.0606	529.0817	471.0751	488.1017	20	C9H11O2N2S	211.0536	C7H11O6N4S	279.0394	C7H9O5N4S	261.0288
342	sulfotep	C8H20O5P2S2	322.0222	6.09	321.0155	381.0366	323.0300	340.0566	30	H4O3PS	114.9613	C2H8O3PS	142.9926	C4H12O3PS	171.0239
343	tau-fluvalinate	C26H22ClF3N2O3	502.1266	9.34	501.1198	561.1410	503.1344	520.1609	30	C13H9O	181.0648	C6H13O3	133.0859	C4H9O2	89.0597
344	tebuconazole	C16H22ClN3O	307.1446	6.16	306.1379	366.1590	308.1524	325.1790	90	C4H6O	70.0413	C7H6Cl	125.0153	C9H8	116.0621
345	tebufenozide	C22H28N2O2	352.2145	5.90	351.2078	411.2289	353.2224	370.2489	10	C9H9O	133.0648	C18H21O2N2	297.1598	C13H19N2	203.1543
346	tebufenpyrad	C18H24ClN3O	333.1602	7.64	332.1535	392.1746	334.1681	351.1946	60	C4H6N2Cl	117.0214	C9H7ON	145.0522	C11H15	147.1168
347	teflubenzuron	C14H6Cl2F4N2O2	379.9737	7.79	378.9670	438.9881	380.9815	398.0081	20	C14H3O2N2Cl2F2	338.9545	C6H2NCl2F2	195.9538	C7H4ONF2	156.0266
348	tepraloxydim	C17H24ClNO4	341.1388	5.33	340.1321	400.1532	342.1467	359.1732	20	C14H20O3N	250.1438	C9H12O2N	166.0863	C14H21O3N	251.1516
349	terbufos	C9H21O2PS3	288.0436	7.62	287.0369	347.0580	289.0514	306.0780	10	C5H11S	103.0576	C4H9	57.0699	C4H12O2PS2	187.0011
350	terbufos-sulfone	C9H21O4PS3	320.0334	4.40	319.0267	379.0478	321.0412	338.0678	20	C4H12O3PS	171.0239	C5H14O4PS3	264.9786	C8H20O4PS2	275.0535
351	terbufos-sulfoxide	C9H21O3PS3	304.0385	4.44	303.0318	363.0529	305.0463	322.0729	40	H4O2PS2	130.9385	C2H8O2PS2	158.9698	C4H12O2PS2	187.0011
352	terbuthylazine	C9H16ClN5	229.1089	4.57+4.78	228.1021	288.1233	230.1167	247.1432	60	C3H5N5Cl	146.0228	C6H11N5Cl	188.0697	C2H3N3Cl	104.0010
353	terbutryn	C10H19N5S	241.1356	4.82+4.98	240.1288	300.1500	242.1434	259.1699	50	C6H12N5S	186.0808	C2H7N2S	91.0324	C4H8N5S	158.0495
354	tetraconazole	C13H11Cl2F4N3O	371.0210	5.54	370.0143	430.0354	372.0288	389.0554	60	C2H4N3Cl2F	158.9761	C4H6O	70.0413	C9H7Cl	150.0231

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
355	tetramethrin	C19H25NO4	331.1778	7.54+ 7.29	330.1711	390.1922	332.1856	349.2122	10	C18H24O2N	286.1802	C19H24O3N	314.1751	C9H10O2N	164.0706
356	thiabendazole	C10H7N3S	201.0355	2.28	200.0288	260.0499	202.0433	219.0699	90	C9H7N2S	175.0324	C8H7N2	131.0604	C6H6N	92.0495
357	thiacloprid	C10H9ClN4S	252.0231	2.44	251.0164	311.0375	253.0309	270.0575	80	C6H5NCl	126.0105	C6H4N	90.0338	C5H4Cl	98.9996
358	thiamethoxam	C8H10ClN5O3S	291.0187	1.98	290.0120	350.0331	292.0266	309.0531	10	C8H11ON4S	211.0648	C4H3NClS	131.9669	C8H10ON4S	210.0570
359	thifensulfuron-methyl	C12H13N5O6S2	387.0302	2.85	386.0234	446.0446	388.0380	405.0646	30	C6H7O2N4	167.0564	C6H5O4S2	204.9624	C5H9ON4	141.0771
360	thiodicarb	C10H18N4O4S3	354.0485	3.45	353.0417	413.0629	355.0563	372.0828	10	C3H6NS	88.0215	C2H6NS2	107.9936	C4H9N2S2	149.0202
361	thiometon	C6H15O2PS3	245.9966	3.67	244.9899	305.0110	247.0045	264.0310	20	C4H9S	89.0419	C2H5S2	92.9827	-	-
362	thiophanate-methyl	C12H14N4O4S2	342.0451	2.88	341.0384	401.0595	343.0529	360.0795	10	C7H7N2S	151.0324	C11H11O3N4S2	311.0267	C9H12O2N3S	226.0645
363	tolclofos-methyl	C9H11Cl2O3PS	299.9538	6.68	298.9471	358.9682	300.9616	317.9882	30	C8H10O3Cl2PS	286.9460	C2H8O3PS	142.9926	C7H5OCl2	174.9712
364	tolylfluanid	C10H13Cl2FN2O2S2	345.9774	6.16	344.9707	404.9918	346.9852	364.0118	10	C8H7NCl2FS	237.9655	C10H14O2N2Cl2FS2	346.9852	C9H13O2N2S	213.0692
365	triadimefon	C14H16ClN3O2	293.0926	5.05	292.0858	352.1070	294.1004	311.1269	20	C11H14OCl	197.0728	C6H11O2	115.0754	C12H14O2Cl	225.0677
366	triadimenol	C14H18ClN3O2	295.1082	5.25	294.1015	354.1226	296.1160	313.1426	10	C4H6O	70.0413	C6H11O	99.0804	C12H16O2Cl	227.0833
367	triasulfuron	C14H16ClN5O5S	401.0555	2.83	400.0488	460.0699	402.0633	419.0899	30	C6H7O2N4	167.0564	C5H9ON4	141.0771	C7H5O2	121.0284
368	triazophos	C12H16N3O3PS	313.0645	5.25	312.0577	372.0789	314.0723	331.0988	60	C8H8ON3	162.0662	C7H7N2	119.0604	H4O3PS	114.9613
369	tricyclazole	C9H7N3S	189.0355	2.75	188.0288	248.0499	190.0433	207.0699	90	C7H6NS	136.0215	C8H7N2S	163.0324	C6H5S	109.0106
370	trifloxystrobin	C20H19F3N2O4	408.1291	7.10	407.1224	467.1435	409.1370	426.1635	40	C9H7NF3	186.0525	C8H6N	116.0495	C9H9N	131.0730
371	triflumuron	C15H10ClF3N2O3	358.0327	6.55	357.0259	417.0471	359.0405	376.0670	30	C7H5ONCl	154.0065	C7H5ONF3	176.0329	COF3	84.9907
372	triforine	C10H14Cl6N4O2	431.9242	4.10+4.30	430.9175	490.9386	432.9321	449.9586	30	C9H12ON3Cl6	387.9106	C8H9N2Cl6	342.8891	C6H10N2Cl3	214.9904
373	trichlorfon	C4H8Cl3O4P	255.9220	2.26	254.9153	314.9364	256.9299	273.9564	20	C2H8O4P	127.0155	C4H8O4Cl2P	220.9532	C2H7O3ClP	144.9816
374	trinexapac ethyl	C13H16O5	252.0992	4.33	251.0925	311.1136	253.1071	270.1336	30	C4H5O	69.0335	C11H11O4	207.0652	C9H14O4	186.0887
375	triticonazole	C17H20ClN3O	317.1289	5.40	316.1222	376.1433	318.1368	335.1633	30	C4H6O	70.0413	C7H6Cl	125.0153	C12H12Cl	191.0622
376	vamidothion	C8H18NO4PS2	287.0409	2.20	286.0342	346.0553	288.0488	305.0753	30	C6H12ONS	146.0634	C4H8ONS	118.0321	C4H8NO	86.0600
377	zoxamide	C14H16Cl3NO2	335.0241	6.27	334.0174	394.0385	336.0319	353.0585	30	C8H5OCl2	186.9712	C8H8NOCl2	203.9977	C6H11NCl	132.0575
378	2-hydroxypropyl-mepanipyrim	C14H17N3O	243.1366	3.71	242.1299	302.1510	244.1444	261.1710	40	C14H16N3	226.1339	C12H14N3	200.1182	C5H8N	82.0651
379	aclonifen	C12H9ClN2O3	264.0302	6.29	263.0229	323.0440	265.0374	282.0640	50	C12H9O2N2Cl	248.0347	C12H8ON	182.0600	C12H9ONCl	218.0367
380	ametoctradin	C15H25N5	275.2115	7.10	274.2037	333.2170	276.2183	293.2448	60	C8H11N5	177.1009	C8H10N5	176.0931	C7H9N4	149.0822
381	asulam	C8H10N2O4S	230.0361	1.74	229.0289	289.0500	231.0434	248.0700	30	C6H6O2NS	156.0114	C8H11O4N2S	231.0434	C6H6ON	108.0444
382	BACC10	C19H34ClN	311.2374	5.59	274.2540	334.2751	276.2686	293.2951	40	C7H7	91.0542	C12H26N	184.2060	C9H14N	136.1121
383	BACC12	C21H38ClN	339.2698	7.12	302.2853	362.3064	304.2999	321.3264	40	C7H7	91.0542	C14H30N	212.2373	-	-
384	BACC14	C23H42ClN	363.2698	8.37	330.3166	390.3377	332.3312	349.3577	40	C7H7	91.0542	C16H34N	240.2686	-	-
385	BACC16	C25H46ClN	391.3011	9.37	358.3479	418.3690	360.3625	377.3890	50	C7H7	91.0542	C18H38N	268.2999	-	-
386	BACC18	C27H50ClN	423.3637	10.16	386.3792	446.4003	388.3938	405.4203	40	C7H7	91.0542	C20H42N	296.3312	-	-
387	BACC8	C17H30ClN	283.2072	3.93	246.2227	306.2438	248.2373	265.2638	50	C7H7	91.0542	C10H22N	156.1747	C11H14N	160.1121
388	bixafen	C18H12Cl2F3N3O	413.0304	5.89	412.0237	472.0448	414.0382	431.0648	40	C13H5ONCl2F	279.9738	C5H3N2	91.0302	C5H5N2F2	131.0426
389	BTS 44595	C12H15N2O2Cl3	324.0205	6.68	323.0126	382.0259	325.0272	342.0537	30	C11H15ONCl3	282.0214	C6H13ON2	129.1022	C5H12N	86.0964
390	BTS 44596	C13H15N2O3Cl3	352.0154	6.48	351.0075	410.0209	353.0221	370.0487	20	C12H13O2NCl3	308.0006	C9H7O2NCl3	265.9537	C11H13ONCl3	280.0057
391	carboxin	C12H13NO2S	235.0662	3.29	234.0594	294.0806	236.0740	253.1005	40	C6H7O2S	143.0161	C6H6NS	124.0215	C10H10O2NS	208.0427
392	cloprop	C9H9ClO3	200.0235	2.24	199.0167	259.0379	201.0313	218.0578	40	C7H6OCl	141.0113	C6H4OCl	126.9956	C8H8OCl	155.0269
393	clopyralid	C6H3Cl2NO2	190.9535	1.72	189.9468	249.9679	191.9614	208.9879	70	C5H4ONCl2	163.9664	C5HNCl	109.9792	-	-
394	cycloxydim	C17H27NO3S	325.1706	7.12	324.1639	384.1850	326.1784	343.2050	30	C15H22O2NS	280.1366	C10H14O2N	180.1019	C5H9S	101.0419

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
395	DDAC	C22H48ClN	361.3481	8.82	324.3635	384.3846	326.3781	343.4046	50	C12H28N	186.2216	C12H26N	184.2060	C6H13	85.1012
396	dithianon	C14H4N2O2S2	295.9720	5.28	294.9641	353.9774	296.9787	314.0052	-	-	-	-	-	-	-
397	diafenthiuron	C23H32N2OS	384.2241	8.82	383.2163	442.2296	385.2308	402.2574	40	C22H21ON2	329.1648	C19H20ON	278.1539	C19H15ON2	287.1179
398	dicamba	C8H6Cl2O3	219.9689	1.81	218.9621	278.9833	220.9767	238.0032	20	C7H5OCl2	174.9723	C6H3Cl2	144.9617	C7H3O2Cl2	188.9516
399	dinotefuran	C7H14N4O3	202.1060	1.74	201.0993	261.1204	203.1139	220.1404	30	C5H11ON3	129.0897	C5H12N3	114.1026	C3H9N3	87.0791
400	empenthrin	C18H26O2	274.1938	8.92	273.1860	332.1993	275.2006	292.2271	30	C14 H19	187.1481	C13H17	173.1325	C17H25	229.1951
401	ethametsulfuron-methyl	C15H18N6O6S	410.1003	3.37	409.0936	469.1147	411.1081	428.1347	20	C7H10O2N5	196.0829	C6H12ON5	170.1036	C5H6O2N5	168.0516
402	etoxazol	C21H23F2NO2	359.1702	8.59	358.1624	417.1757	360.1770	377.2035	30	C7H3OF2	141.0146	C17H16O2NF2	304.1144	C12H17O	177.1274
403	fenamidone	C17H17N3OS	311.1087	4.63	310.1020	370.1231	312.1165	329.1431	30	C6H6N	92.0495	C15H14N3	236.1182	C8H7	103.0542
404	fenpyrazamin	C17H21N3O2S	331.1360	5.30	330.1282	389.1415	332.1427	349.1693	20	C16H22ON3	272.1757	C13H17ON3	231.1366	C13H16ON3	230.1288
405	fensulfothion oxon	C11H17O5PS	292.0540	2.55	291.0462	350.0595	293.0607	310.0873	40	C7H10O5PS	236.9981	C7H9O2S	157.0318	C7H9O4PS	219.9954
406	fensulfothion sulfone	C11H17O5PS2	324.0260	4.08	323.0182	382.0315	325.0328	342.0593	30	C7H10O5PS2	268.9702	C6H8O3PS	190.9926	C7H9O3S	173.0267
407	fensulfothion-PO-sulfon	C11H17O6PS	308.0489	2.58	307.0411	366.0544	309.0556	326.0822	30	C7H10O6PS	252.9930	C6H8O4P	175.0155	C9H14O6PS	281.0243
408	fentin	C18H15Sn	343.0216	6.07	349.0044	409.0255	351.0190	368.0455	80	Sn	119.9017	C6H5Sn	196.9408	C8H13	109.1012
409	fipronil desulfinyl	C12H4Cl2F6N4	387.9723	5.69	386.9644	445.9777	388.9790	406.0055	40	C12H2N4ClF6	350.9878	C11H2N4ClF3	281.9926	C12HN4ClF5	330.9815
410	fipronil sulfone	C12H4Cl2F6N4O2S	451.9331	6.30	450.9263	510.9475	452.9409	469.9674	30	C12H2O2N4ClF6S	414.9497	C8H2N2F3	183.0176	C8H2ON2ClF3	233.9813
411	flubendiamid	C23H22F7IN2O4S	682.0228	6.03	681.0160	741.0372	683.0306	700.0571	40	C10H6NF6	254.0410	C10H4NF4	214.0285	C8H3O2NI	271.9214
412	fluopicolide	C14H8Cl3F3N2O	381.9649	4.95	380.9582	440.9793	382.9727	399.9993	30	C7H3OCl2	172.9555	C14H7N2Cl3F3	364.9621	C7H6ONClF3	212.0085
413	flurochloridone	C12H10Cl2F3NO	311.0086	5.18	310.0019	370.0230	312.0164	329.0430	60	C11H9NF3	212.0682	C4H6Cl	89.0153	C8H5NF3	172.0369
414	flutolanil	C17H16F3NO2	323.1128	4.89	322.1060	382.1272	324.1206	341.1471	40	C14H9O2NF	242.0612	C14H10O2NF2	262.0674	C8H4OF3	173.0209
415	fluxapyroxad	C18H12F5N3O	381.0895	4.91	380.0828	440.1039	382.0973	399.1239	40	C6H3N2	103.0302	C13H5ONF3	248.0329	C5H5N2F2	131.0426
416	hexaflumuron	C16H8Cl2F6N2O3	459.9811	7.25	458.9743	518.9955	460.9889	478.0154	20	C16H6O3N2Cl2F5	438.9681	C8H4ONCl2F4	275.9612	C16H5O3N2ClF5	402.9914
417	chlorbufam	C11H10ClNO2	223.0406	4.64	222.0327	281.0460	224.0473	241.0738	40	C7H5ONCl	154.0054	C6H5NCl	126.0105	-	-
418	chlorpropham	C10H12NO2Cl	213.0551	5.04	212.0484	272.0695	214.0629	231.0895	40	C9H13OCl	172.0649	C7H5ONCl	154.0054	-	-
419	chlorpyrifos (ethyl)	C9H11Cl3NO3PS	348.9257	8.14	347.9190	407.9401	349.9336	366.9601	20	C5H3ONCl3	197.9275	H4O3PS	114.9613	C7H8O3NCl3PS	321.9023
420	iprodione	C13H13Cl2N3O3	329.0328	5.69	328.0261	388.0472	330.0407	347.0672	20	C9H5O2N2Cl2	242.9734	-	-	-	-
421	isopyrazam	C20H23F2N3O	359.1815	7.12	358.1736	417.1869	360.1882	377.2147	30	C20H22ON3	320.1757	C13H11ON3F	244.0881	C20H23ON3F	340.1820
422	meptyldinocap	C18H24N2O6	364.1629	5.38	295.1299	355.1510	297.1445	314.1710	60	C8H5O4N2	193.0255	C9H8O4N	194.0459	C8H5O5N2	209.0204
423	metamitron-desamino	C10H9N3O	187.0751	2.31	186.0673	245.0806	188.0818	205.1084	60	C9H10N3	160.0869	C7H6N	104.0495	C7H7N2	119.0604
424	metominostrobin	C16H16N2O3	284.1155	4.02+3.70	283.1088	343.1299	285.1234	302.1499	20	C13H10ON	196.0757	C15H12O2N	238.0863	C13H8ON	194.0600
425	metrafenone	C19H21BrO5	408.0567	6.69	407.0500	467.0711	409.0645	426.0911	30	C11H13O4	209.0808	C9H8O2Br	226.9702	C10H10O4	194.0574
426	neoquassin	C22H30O6	390.2037	3.03+3.11	389.1970	449.2181	391.2115	408.2381	40	C18H21O3	285.1485	C18H21O	253.1587	C12H15O3	207.1016
427	nitenpyram	C11H15ClN4O2	270.0878	1.88	269.0811	329.1022	271.0956	288.1222	20	C11H16N3Cl	225.1027	C11H15N3	189.1260	C5H12ON3	130.0975
428	oxamyl-oxime	C5H10N2O2S	162.0468	1.70	161.0390	220.0523	163.0536	180.0801	10	C2H4ONS	90.0008	C4H7O2N2	115.0502	C5H9ON2S	145.0430
429	penflufen	C18H24FN3O	317.1909	6.20	316.1831	375.1964	318.1976	335.2242	40	C6H6ON2F	141.0459	C12H13ON3F	234.1037	C5H8N2F	115.0666
430	penthiopyrad	C16H20F3N3OS	359.1285	6.32	358.1206	417.1339	360.1352	377.1617	30	C10H8ON3F2S	256.0351	C10H9ON3F3S	276.0413	C6H4ON2F3	177.0270
431	pethoxamid	C16H22ClNO2	295.1334	5.47	294.1266	354.1478	296.1412	313.1677	30	C10H11	131.0855	C14H17ONCl	250.0993	C4H9O	73.0648
432	phosmet	C11H12NO4PS2	316.9940	4.31	315.9873	376.0084	318.0018	335.0284	60	C9H6O2N	160.0393	C8H5O2	133.0284	C7H5O	105.0335
433	quassin	C22H28O6	388.1880	2.63	387.1813	447.2024	389.1959	406.2224	30	C12H15O4	223.0965	C14H17O4	249.1121	C12H15O2	191.1067
434	spirotetramat	C21H27NO5	373.1884	5.34	372.1816	432.2028	374.1962	391.2227	30	C18H24O3N	302.1751	C17H20O2N	270.1489	C20H28O3N	330.2064

No	Analyte	Formula	*m/z*	RT [min]	ESI(−)		ESI(+)		NCE [%]	Fragment 1		Fragment 2		Fragment 3	
					$[M-H]^-$	$[M+CH3COO]^-$	$[M+H]^+$	$[M+NH4]^+$		Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
435	spirotetramate –enol	C18H23NO3	301.1683	3.56	300.1605	359.1738	302.1751	319.2016	50	C13H14O2N	216.1019	C17H20O2N	270.1489	C11H9O2	173.0597
436	spirotetramate –enol-glukosid	C24H33NO8	463.2212	2.08	462.2133	521.2266	464.2279	481.2544	50	C13H14O2N	216.1019	C17H20O2N	270.1489	C18H24O3N	302.1751
437	spirotetramate –keto-hydroxy	C18H23NO4	317.1633	3.99	316.1554	375.1687	318.1700	335.1965	30	C18H22O3N	300.1594	C17H18O2N	268.1332	C13H12O2N	214.0863
438	spirotetramate –mono-hydroxy	C18H25NO3	303.1840	5.50	302.1762	361.1895	304.1907	321.2173	10	C9H11	119.0855	C9H19O3N2	203.1390	C9H16O3N	186.1125
439	terbuthylazine-desethyl	C7H12ClN5	201.0776	3.13+3.33	200.0708	260.0920	202.0854	219.1119	70	C2H3N3Cl	104.0010	C4H6N3	96.0556	C4H7N3Cl	132.0323
440	TFNA	C7H4F3NO2	191.0200	1.83	190.0121	249.0254	192.0267	209.0532	60	C6H5NF3	148.0369	C5H5NF	98.0401	-	
441	TFNG	C9H7F3N2O3	248.0414	1.87	247.0336	306.0469	249.0482	266.0747	40	C8H5ON2F2	183.0364	C7H5ONF3	176.0318	C6H5NF3	148.0369
442	tolfenpyrad	C21H22ClN3O2	383.1406	8.00	382.1328	441.1461	384.1473	401.1739	40	C14H13O	197.0961	C6H10N2Cl	145.0527	C7H8ON2Cl	171.0320
443	tribenuron-methyl	C15H17N5O6S	395.0894	3.71	394.0827	454.1038	396.0972	413.1238	30	C6H11ON4	155.0927	C7H9O2N4	181.0720	C8H7O4S	199.0060
444	triclopyr	C7H4Cl3NO3	254.9251	2.35	253.9184	313.9395	255.9330	272.9595	20	C5HONCl3	195.9129	C7H2O3NCl2	217.9417	C6H2ONCl2	173.9519
445	vamidothion-sulfon	C8H18NO6PS2	319.0319	1.93	318.0240	377.0373	320.0386	337.0651	30	C6H12O3NS	178.0532	C4H10O3PS	169.0083	C6H10O2NS	160.0427
446	vamidothion-sulfoxid	C8H18NO5PS2	303.0369	1.85	302.0291	361.0424	304.0437	321.0702	10	C4 H10O3PS2	200.9803	C4H10O3PS	169.0083	C2H8O3PS	142.9926

Table S2 Performance characteristics for (i) fruit and vegetables, (ii) cereals, and (iii) tea. The artificial contamination levels for determination of recoveries and repeatability were 200, 1 000 and 2 000 µg/kg (n = 6) for fruit and vegetables, cereals, and tea, respectively.

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
MYCOTOXINS										
1	3+15-Acetyl-DON	50	87	7.14	250	79	6.97	500	78	6.98
2	aflatoxin B1	1	89	4.56	1	82	2.73	1	92	4.17
3	aflatoxin B2	1	87	5.70	1	86	8.37	1	89	8.59
4	aflatoxin G1	1	102	5.90	1	84	6.17	1	91	6.97
5	aflatoxin G2	1	88	6.55	2.5	84	7.84	5	92	9.74
6	agroclavine	1	98	3.62	1	96	8.46	2	78	6.99
7	alpha-zearalenol	1	87	3.85	5	85	5.81	10	88	5.12
8	alternariol	1	82	1.96	1	87	3.55	2	87	4.11
9	alternariol-methylether	1	79	9.51	1	82	6.17	2	84	7.16
10	beauvericin	1	85	5.85	1	96	5.66	2	92	6.14
11	beta-zearalenol	1	88	8.04	5	88	5.34	10	92	8.99
12	citrinin	20	95	2.72	50	77	8.43	100	81	6.78
13	cyclopiazonic acid	100	104	6.84	250	95	13.73	500	86	8.24
14	deoxynivalenol	100	102	6.24	100	87	1.72	500	79	3.47
15	deoxynivalenol-3-glucoside	50	35	9.85	50	79	7.83	100	42	6.87
16	diacetoxyscirpenol	1	84	5.67	1	47	6.91	2	88	6.89
17	enniatin A	1	86	6.13	1	86	7.45	2	82	9.14
18	enniatin A1	1	88	5.66	1	84	3.27	2	86	4.10
19	enniatin B	1	89	5.39	1	81	0.55	1	84	3.58
20	enniatin B1	1	86	1.68	1	78	1.27	1	87	2.14
21	ergocornine	5	94	8.76	10	90	8.68	20	85	6.79
22	ergocorninine	5	89	2.97	10	87	6.59	20	92	3.89
23	ergocristine	5	93	4.14	10	82	8.36	20	88	8.47
24	ergocristinine	5	92	4.98	10	80	7.15	20	86	6.79
25	ergocryptine	5	94	8.95	10	84	7.85	20	87	5.70
26	ergocryptinine	5	103	4.92	10	87	6.12	20	92	9.74
27	ergometrine	5	83	7.40	10	80	6.05	20	74	5.79
28	ergosine	5	97	8.98	10	80	6.11	20	89	6.71
29	ergosinine	5	89	7.82	10	89	4.40	20	88	2.79
30	ergotamine	5	91	8.87	10	95	2.75	20	89	6.73

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
31	ergotaminine	5	102	9.24	10	79	1.90	20	93	3.87
32	fumonisin B1	20	96	8.13	25	72	4.58	100	88	6.57
33	fumonisin B2	20	95	4.98	25	72	9.08	100	98	9.41
34	fumonisin B3	20	91	7.23	25	84	5.35	100	94	6.70
35	fusarenon X	100	84	7.18	100	83	7.54	200	76	8.79
36	gliotoxin	1	93	3.32	5	80	8.65	10	78	4.79
37	HT-2 toxin	1	95	9.90	5	80	6.73	10	94	11.20
38	meleagrin	1	88	3.01	1	92	6.25	2	93	6.14
39	mycophenolic acid	1	95	3.26	1	75	4.99	2	98	6.41
40	neosolaniol	10	91	1.10	25	82	6.22	50	93	2.59
41	nivalenol	100	71	13.38	100	78	9.29	500	70	13.04
42	ochratoxin A	1	88	8.77	2.5	82	3.81	5	92	9.63
43	patulin	100	78	9.54	500	87	6.91	1000	71	7.48
44	paxilline	5	92	9.97	25	91	8.24	50	85	6.99
45	penicillic acid	10	92	6.26	50	88	2.66	100	94	3.14
46	penitrem A	10	107	4.63	50	95	3.46	100	93	4.91
47	phomopsin A	50	101	6.92	250	76	2.08	500	82	6.78
48	roquefortine C	1	105	5.45	2.5	94	5.92	5	103	2.87
49	stachybotrylactam	1	91	6.54	2.5	78	6.20	5	86	6.10
50	sterigmatocystin	1	86	9.65	1	90	9.46	1	92	6.94
51	T-2 toxin	1	86	5.01	2.5	83	6.18	2	91	4.78
52	tentoxin	1	91	5.01	1	71	4.11	2	89	3.96
53	verrucarol	50	101	3.48	250	75	9.28	500	72	5.47
54	verruculogen	50	91	4.92	250	105	5.85	500	84	6.13
55	zearalenone	1	81	9.96	1	89	5.51	1	92	8.69
56	tenuazonic acid	200	80	7.14	500	70	9.81	1000	72	6.91
PESTICIDES										
57	2,4,5-T	1	95	3.49	5	100	7.11	10	83	6.70
58	2,4-D	1	87	4.22	5	98	6.97	10	86	6.75
59	2,4-DB	20	92	7.14	50	89	8.12	100	81	10.13
60	2-NOA	1	97	1.80	1	94	7.27	2	87	3.98
61	4-CPA	1	92	8.23	5	97	7.93	10	93	8.79
62	acephate	1	74	2.42	5	85	8.13	10	76	6.77
63	acetamiprid	1	87	6.40	1	92	9.28	1	82	8.97

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
64	acetochlor	1	82	4.73	2.5	93	2.92	5	88	6.41
65	acrinathrin	5	93	6.15	25	93	2.25	50	90	3.47
66	alachlor	1	87	3.49	5	96	1.87	10	82	2.88
67	aldicarb	10	99	7.84	25	94	4.88	100	82	7.97
68	aldicarb sulfone	1	84	5.42	5	99	6.24	10	72	8.14
69	aldicarb sulfoxide	20	71	5.49	50	87	4.49	100	88	3.47
70	ametryn	1	80	3.20	1	90	3.07	1	84	4.15
71	atrazine	1	72	1.11	1	93	5.50	1	86	5.29
72	avermectin-b1a	50	86	5.07	100	86	6.28	200	89	8.41
73	azadirachtin	5	72	4.78	25	94	5.81	50	91	7.48
74	azinphos-ethyl	1	87	7.74	1	91	4.97	1	80	8.74
75	azinphos-methyl	1	80	2.53	1	93	8.09	1	75	3.79
76	azoxystrobin	1	81	2.26	1	95	4.23	1	88	6.99
77	benalaxyl	1	80	6.66	1	96	10.05	1	81	11.09
78	bendiocarb	1	83	6.67	2.5	96	3.27	2	81	6.84
79	bentazone	1	93	5.79	5	95	9.43	1	82	8.74
80	beta-cyfluthrin	200	92	5.78	500	86	8.74	1000	84	11.04
81	bifenthrin	5	83	8.24	25	80	7.60	100	70	7.86
82	bitertanol	1	90	0.25	1	92	11.21	1	102	8.47
83	boscalid	1	81	2.37	2.5	91	3.66	10	90	2.97
84	bromacil	1	80	7.75	1	90	7.23	1	84	8.98
85	Bromoxynil	1	75	3.12	5	93	3.78	1	82	4.58
86	bromuconazole	1	79	9.66	5	91	5.28	20	85	6.79
87	bupirimate	1	74	4.09	1	94	8.48	1	112	10.24
88	buprofezin	1	81	7.93	1	95	0.49	1	75	4.79
89	cadusafos	1	80	2.41	1	93	5.36	2	78	6.87
90	carbaryl	1	82	5.73	1	91	6.85	2	78	7.67
91	carbendazim	1	98	4.44	1	91	6.22	1	76	6.84
92	carbofuran	1	75	5.65	1	95	8.25	2	84	8.57
93	carbofuran-3-hydroxy	1	80	4.95	5	95	6.70	10	78	7.49
94	carbophenothion	5	78	3.59	25	81	10.69	50	70	11.51
95	cinerin I	10	80	4.50	25	89	6.97	100	84	6.78
96	cinerin II	200	75	4.56	500	82	6.19	500	83	5.74
97	clofentezine	1	74	4.34	2.5	88	7.06	5	80	6.99

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
98	clomazone	1	72	10.39	1	93	8.73	2	91	11.43
99	clothianidin	1	78	5.22	1	91	3.38	1	82	6.41
100	cyanazine	1	81	2.47	10	95	3.38	10	84	3.17
101	cyazofamid	1	70	1.12	1	87	4.65	1	82	4.53
102	cymoxanil	20	82	9.06	100	92	6.03	200	75	6.78
103	cypermethrin	10	95	8.40	10	87	10.26	50	84	13.16
104	cyproconazole	1	85	7.63	5	91	2.94	5	90	5.89
105	cyprodinil	1	81	2.51	1	88	0.28	1	78	4.12
106	DEET	1	82	1.02	1	89	1.65	1	86	3.02
107	deltamethrin	20	87	8.54	50	89	6.09	100	84	4.16
108	demeton-S-methyl	5	78	4.56	25	87	1.58	50	75	4.56
109	demeton-S-methylsulfone	2	79	3.19	2.5	88	6.85	5	82	7.01
110	desmedipham	1	74	4.41	1	90	8.23	2	84	6.98
111	desmetryn	1	73	7.45	1	93	8.71	1	76	10.34
112	diazinon	1	70	7.59	1	92	8.89	1	88	10.41
113	diclofop-methyl	1	80	4.58	1	86	5.94	2	78	6.84
114	dicrotophos	1	80	3.38	2.5	86	3.97	5	79	4.18
115	diethofencarb	1	81	1.05	1	89	3.75	2	81	6.20
116	difenoconazole	1	78	2.71	2.5	84	2.12	2	80	3.54
117	diflubenzuron	1	83	3.30	1	87	1.61	5	85	2.14
118	diflufenican	1	86	4.56	1	94	9.05	1	75	6.57
119	dichlofluanid	1	93	7.06	2.5	77	9.51	10	79	9.68
120	dichlormid	1	85	6.81	5	89	3.19	2	84	6.47
121	dichlorprop	1	94	6.41	2.5	100	8.22	5	84	6.95
122	dichlorvos	1	83	4.56	2.5	102	7.45	5	98	7.45
123	dimethachlor	1	82	3.84	1	93	5.93	2	87	8.14
124	dimethenamide	1	82	2.14	1	96	1.73	1	86	3.58
125	dimethoate	1	81	5.45	5	97	3.21	20	80	6.41
126	dimethomorph	1	82	3.77	5	95	4.86	10	89	3.99
127	dimoxystrobin	1	82	2.24	1	92	0.39	2	84	3.62
128	diniconazole	1	80	6.94	2.5	89	6.31	10	90	7.47
129	disulfoton	2	82	3.14	10	79	11.20	10	76	9.85
130	disulfoton-sulfone	1	84	2.24	2.5	95	9.03	5	82	10.14
131	disulfoton-sulfoxide	1	92	6.22	1	109	4.46	2	99	7.48

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
132	diuron	1	81	4.71	1	91	5.01	2	86	8.61
133	DMSA	1	76	3.96	5	106	7.84	10	88	8.47
134	DMST	1	79	3.15	1	99	5.32	5	94	4.16
135	dodine	200	84	6.36	1000	74	6.46	1000	78	8.61
136	EPN	50	92	2.09	100	85	4.89	100	85	5.61
137	epoxiconazole	1	79	3.72	1	93	7.08	1	86	7.18
138	ethiofencarb	1	76	6.19	1	82	1.43	1	70	8.17
139	ethion	1	87	8.27	1	90	3.99	1	86	7.41
140	ethirimol	1	72	2.79	2.5	88	7.91	5	80	8.24
141	ethofumesate	1	88	7.80	1	94	8.90	1	87	11.64
142	ethoprophos	1	85	8.73	1	93	0.95	2	86	7.54
143	etofenprox	1	84	3.61	1	71	6.41	1	76	8.21
144	etrimfos	1	71	7.52	1	90	7.20	2	82	9.36
145	famoxadone	2	81	8.26	10	90	2.21	20	80	6.87
146	fenamiphos	1	79	4.51	1	85	3.87	2	83	5.61
147	fenamiphos sulphone	1	83	4.15	1	92	6.72	2	84	6.84
148	fenamiphos-sulfoxide	1	87	6.13	1	95	7.07	2	93	11.49
149	fenarimol	1	71	9.65	1	89	10.74	1	87	13.20
150	fenazaquin	1	79	4.25	1	85	3.86	2	76	6.34
151	fenbuconazole	1	82	2.71	1	91	3.70	1	89	4.02
152	fenbutatin-oxide	50	85	6.98	100	67	6.03	200	76	8.09
153	fenhexamid	1	70	0.85	2.5	88	6.70	5	85	9.82
154	fenoprop	1	93	5.59	1	95	3.41	1	84	6.21
155	fenoxaprop	10	76	4.78	50	84	5.67	100	81	6.97
156	fenoxaprop-ethyl	1	80	8.87	1	92	5.59	2	76	10.24
157	fenoxycarb	1	87	5.85	1	90	4.55	2	84	9.64
158	fenpropathrin	1	92	3.05	2.5	91	7.92	5	74	6.54
159	fenpropidin	1	79	6.52	1	90	3.72	1	84	4.89
160	fenpropimorph	1	77	8.01	1	93	4.02	1	79	8.96
161	fenpyroximate	1	89	6.89	1	90	5.52	1	86	6.87
162	fensulfothion	1	88	1.32	1	93	6.43	1	89	4.57
163	fenthion	2	78	2.97	25	84	6.83	50	75	6.85
164	fenthion-sulfone	1	81	4.75	1	95	7.41	1	86	8.29
165	fenthion-sulfoxide	1	87	8.15	1	99	8.51	1	92	10.48

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
166	fipronil	1	77	4.21	1	89	3.07	1	87	5.47
167	flonicamid	1	80	2.10	2.5	93	7.51	1	84	6.87
168	florasulam	1	80	5.09	1	95	7.19	2	94	6.79
169	fluacrypyrim	1	80	3.78	1	95	3.51	2	82	3.71
170	fluazifop	1	77	5.35	2.5	88	2.77	5	90	2.97
171	fluazifop-P-butyl	1	72	3.37	1	91	5.70	1	82	6.14
172	fluazinam	1	86	5.24	1	95	2.78	1	82	6.54
173	flucythrinate	1	85	8.43	5	92	2.26	20	86	7.84
174	fludioxonil	1	86	1.98	1	98	2.13	1	83	2.86
175	flufenacet	1	80	6.31	1	93	8.60	2	88	6.47
176	flufenoxuron	1	73	7.49	2.5	91	8.90	5	84	12.03
177	flumioxazin	50	76	9.20	100	90	5.78	200	84	6.47
178	fluopyram	1	79	7.60	1	93	5.02	1	85	8.74
179	fluoxastrobin	1	80	9.28	5	95	5.17	5	88	11.27
180	fluquinconazole	5	85	8.23	25	92	3.53	100	86	6.69
181	fluroxypyr	10	84	6.48	100	88	3.45	200	84	7.56
182	flusilazole	1	87	3.61	1	92	8.89	2	84	9.11
183	flutriafol	1	81	6.02	2.5	95	3.88	5	85	4.97
184	fomesafen	1	87	7.91	1	96	8.86	2	88	10.23
185	fonofos	2	82	7.17	10	85	5.03	10	76	6.87
186	foramsulfuron	1	84	8.60	1	83	4.60	1	101	9.01
187	formetanate	1	71	0.72	5	82	4.78	10	74	3.61
188	formothion	2	71	5.64	10	94	2.53	20	79	3.58
189	fosthiazate	2	82	4.05	10	95	4.94	20	84	6.10
190	furathiocarb	1	79	2.80	1	92	1.63	2	87	3.14
191	haloxyfop	1	81	8.97	2.5	81	7.39	10	85	10.23
192	haloxyfop-2-ethoxyethyl	1	74	1.13	1	92	7.72	1	83	8.11
193	haloxyfop-methyl	1	79	4.49	1	94	5.59	1	79	6.21
194	heptenophos	1	83	2.64	1	91	6.63	1	87	3.64
195	hexaconazole	1	87	4.41	1	91	2.52	1	100	2.84
196	hexazinone	1	82	6.55	1	89	3.97	2	86	2.99
197	hexythiazox	1	72	9.13	5	88	7.58	5	84	10.78
198	chlorantraniliprole	2	80	8.52	5	90	5.47	10	84	7.87
199	chlorfenvinphos	1	81	11.28	2.5	92	5.47	5	84	12.14

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
200	chloridazon	1	81	6.05	2.5	92	2.89	5	79	6.47
201	chlorotoluron	1	80	2.78	1	96	2.05	1	88	4.15
202	chloroxuron	2	71	5.92	5	91	11.16	10	84	12.67
203	chlorpyrifos-methyl	2	90	8.68	25	94	9.57	50	76	13.07
204	chlorsulfuron	1	82	2.99	2.5	92	5.96	5	94	6.57
205	imazalil	1	71	7.18	1	90	3.14	1	78	6.94
206	Imazamethabenz-methyl	1	80	3.55	1	95	3.37	1	84	4.71
207	imazamox	1	82	3.07	2.5	87	6.92	5	94	4.21
208	imazapyr	1	78	3.87	2.5	82	7.05	2	88	6.72
209	imazaquin	1	72	9.29	1	90	8.75	2	87	11.24
210	imazethapyr	1	88	7.74	1	88	9.22	2	101	10.37
211	imazosulfuron	2	81	4.36	5	88	4.29	10	95	6.54
212	imidacloprid	1	72	5.46	1	93	4.53	1	82	4.82
213	indoxacarb	1	85	2.41	2.5	92	5.35	5	94	3.47
214	iodosulfuron-methyl	1	83	7.73	5	91	10.28	5	92	11.20
215	ioxynil	1	78	3.64	2.5	95	4.54	10	75	6.21
216	iprovalicarb	1	91	3.12	1	91	2.44	2	85	4.00
217	isofenphos	1	79	5.23	5	92	4.92	5	78	5.20
218	isofenphos-methyl	1	83	3.85	5	98	3.72	10	82	3.89
219	isoprocarb	1	84	1.24	1	92	8.07	1	86	8.41
220	isoprothiolane	1	81	6.63	1	96	9.75	1	83	10.08
221	isoproturon	1	81	4.44	1	93	2.14	2	85	4.58
222	jasmolin I	50	76	7.75	100	85	1.15	200	78	6.87
223	jasmolin II	100	74	3.24	250	88	7.82	500	72	6.58
224	kresoxim-methyl	1	76	7.86	1	95	6.08	2	73	8.67
225	lambda-cyhalothrin	10	97	3.69	25	86	4.28	100	82	5.13
226	lenacil	1	79	7.06	1	92	5.52	1	84	6.02
227	linuron	1	80	7.37	2.5	96	7.85	5	87	9.30
228	lufenuron	1	83	5.27	1	93	9.25	2	87	6.89
229	malaoxon	1	90	9.21	1	94	8.59	2	89	13.10
230	malathion	1	83	4.70	2.5	96	6.21	2	86	8.20
231	mandipropamide	1	80	5.82	1	96	2.82	1	80	6.13
232	MCPA	1	75	6.67	5	100	4.06	10	79	6.41
233	MCPB	5	98	8.33	25	94	1.58	50	88	6.34

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
234	mecarbam	1	80	5.60	1	94	9.04	1	85	7.20
235	mecoprop	1	91	8.74	1	100	6.56	1	91	8.69
236	mefenpyr-diethyl	1	78	4.74	1	95	8.67	2	84	9.37
237	mepanipyrim	1	81	7.53	1	89	2.91	1	88	7.10
238	mepronil	1	73	7.43	1	93	6.17	1	83	8.06
239	metaflumizone	1	75	10.00	1	83	11.03	1	76	12.36
240	metalaxyl	1	73	5.95	1	95	1.03	1	86	2.14
241	metamitron	1	80	6.70	5	94	4.11	5	80	3.97
242	metazachlor	1	80	7.76	1	95	9.71	1	84	11.58
243	metconazole	1	87	7.21	1	92	6.35	1	94	8.07
244	methacriphos	1	83	4.30	5	95	4.83	10	81	6.23
245	methamidophos	10	71	5.81	10	80	7.68	10	73	5.87
246	methidathion	1	84	5.35	2.5	95	1.17	5	81	2.86
247	methiocarb	1	82	6.19	1	88	5.94	1	84	9.30
248	methiocarb sulfoxide	1	87	4.60	5	90	7.19	5	82	6.91
249	methiocarb-sulfone	2	81	3.23	10	94	2.70	20	80	4.51
250	metholcarb	20	76	4.64	25	90	5.53	50	83	6.49
251	methomyl	10	79	3.48	25	87	4.62	100	76	3.29
252	methoxyfenozide	1	74	6.94	1	93	7.50	2	82	8.45
253	metobromuron	1	82	5.08	1	94	9.32	2	84	6.19
254	metolachlor	1	82	7.19	1	94	5.39	2	88	9.54
255	metosulam	1	84	7.88	2.5	92	9.71	5	92	8.41
256	metoxuron	1	72	4.36	1	91	8.02	5	85	6.14
257	metribuzin	1	80	2.51	1	105	5.67	1	84	6.00
258	metsulfuron-methyl	1	83	6.09	5	93	3.00	10	93	3.84
259	mevinphos	1	89	4.54	5	94	3.91	10	76	5.84
260	monocrotophos	2	77	1.31	10	83	7.30	20	74	9.74
261	monolinuron	1	74	3.61	1	86	2.01	2	82	5.47
262	monuron	1	74	6.16	1	91	7.67	2	86	2.84
263	myclobutanil	1	81	5.53	1	93	9.41	1	87	6.74
264	naled	10	85	4.57	25	76	7.84	100	84	7.63
265	napropamide	1	79	1.59	1	89	5.58	1	83	3.19
266	neburon	1	83	1.34	1	91	2.36	2	84	2.80
267	nicosulfuron	1	84	8.49	1	78	7.46	1	103	6.89

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
268	norflurazone	1	72	5.41	1	93	3.20	2	85	3.67
269	omethoate	5	76	4.24	10	82	4.29	20	76	2.97
270	oxadixyl	1	81	7.34	1	93	9.34	2	83	11.56
271	oxamyl	20	75	3.51	50	93	8.01	100	74	6.94
272	oxydemeton-methyl	1	79	4.20	2.5	94	5.46	5	78	5.07
273	oxyfluorfen	1	90	4.76	5	90	6.71	10	82	6.79
274	paclobutrazol	1	87	1.27	1	94	3.81	1	86	5.02
275	penconazole	1	81	5.82	2.5	88	2.82	5	85	1.93
276	pencycuron	1	82	5.04	1	86	7.01	2	82	6.08
277	pendimethalin	1	74	5.63	2.5	89	3.96	5	82	5.00
278	permethrin	2	83	1.14	5	81	3.23	10	77	6.41
279	phenmedipham	1	80	4.41	1	94	7.61	1	84	8.08
280	phenothrin	1	74	5.23	5	83	0.77	20	72	2.11
281	phenthoate	1	82	9.43	1	92	7.13	2	83	6.70
282	phorate	50	79	4.85	100	100	6.77	100	73	9.40
283	phorate-sulfone	1	83	3.24	2.5	98	5.36	5	82	3.98
284	phorate-sulfoxide	1	84	5.61	1	100	6.67	1	92	6.64
285	phosalone	1	74	5.86	1	89	11.54	2	76	9.67
286	phosphamidon	1	82	6.41	2.5	94	4.18	5	84	3.00
287	phoxim	1	70	7.26	1	86	2.54	2	72	5.74
288	picloram	100	88	5.99	250	90	6.89	500	84	6.80
289	picolinafen	1	82	7.09	1	88	5.78	2	82	3.67
290	picoxystrobin	1	79	6.40	1	88	3.44	2	82	4.87
291	pinoxaden	1	81	7.17	1	91	9.11	2	84	10.63
292	piperonyl butoxide	1	85	2.91	1	94	2.09	2	79	3.97
293	pirimicarb	1	76	5.88	1	93	6.37	1	80	6.28
294	pirimicarb-desmethyl	1	90	4.23	1	86	2.81	1	83	4.00
295	pirimiphos-ethyl	1	75	4.69	1	90	8.26	1	79	6.74
296	pirimiphos-methyl	1	77	7.62	1	91	3.66	1	75	4.96
297	profenofos	1	72	8.38	1	91	2.08	1	89	2.47
298	prochloraz	1	75	9.07	1	92	6.39	1	84	6.84
299	prometon	1	80	8.30	1	93	1.32	1	83	6.98
300	prometryn	1	71	4.26	1	96	8.52	1	86	4.77
301	propachlor	2	82	2.78	5	95	6.18	5	85	9.80

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
302	propamocarb	1	72	7.86	2.5	82	4.73	2	76	6.00
303	propaquizafop	1	89	6.41	1	90	9.26	1	84	4.70
304	propargite	1	77	0.99	1	87	5.65	1	82	6.98
305	propazine	1	79	3.50	1	92	3.26	1	84	6.59
306	propham	10	104	1.64	50	90	7.08	100	87	4.23
307	propiconazole	1	87	9.02	1	95	6.54	1	93	6.80
308	propoxur	1	76	4.86	1	94	4.32	2	76	8.05
309	propoxycarbazone	1	85	7.78	2.5	89	4.98	1	94	2.14
310	propyzamide	1	78	4.09	1	93	8.69	2	84	6.90
311	proquinazid	1	71	7.31	1	78	7.62	1	72	8.64
312	prosulfocarb	1	79	3.19	1	92	5.01	1	81	4.59
313	prothioconazole-desthio	1	88	6.36	2.5	89	4.42	10	84	6.84
314	prothiofos	50	78	9.21	250	83	4.57	200	71	7.94
315	pyraclostrobin	1	80	2.56	1	91	8.50	1	79	10.54
316	pyrazophos	1	79	4.47	1	96	6.01	2	94	5.76
317	pyrethrin I	5	72	1.76	10	70	3.00	20	78	5.19
318	pyrethrin II	20	73	5.95	50	72	7.68	100	80	9.74
319	pyridaben	1	73	5.54	1	81	7.42	2	71	3.18
320	pyridate	1	93	4.66	1	80	9.54	2	84	10.25
321	pyrifenox	1	81	2.16	1	93	9.67	2	80	6.83
322	pyrimethanil	1	80	8.61	1	96	7.34	2	84	10.36
323	pyriproxyfen	1	80	5.97	1	87	7.43	1	80	6.91
324	quinalphos	1	79	2.43	1	89	2.74	2	81	3.06
325	quinclorac	1	81	9.35	5	82	2.87	10	84	11.09
326	quinmerac	2	82	5.94	10	90	7.53	20	90	6.97
327	quinoclamine	1	80	7.34	5	90	4.11	10	74	4.78
328	quinoxyfen	1	75	4.13	1	87	7.32	5	81	3.69
329	quizalofop	2	80	9.52	10	85	6.78	20	84	6.99
330	quizalofop-p-ethyl	1	72	4.94	1	87	2.25	1	77	4.05
331	resmethrin	1	71	5.27	1	70	6.46	2	71	6.74
332	rimsulfuron	1	81	7.21	2.5	84	8.55	5	88	10.47
333	rotenone	1	81	4.99	1	89	10.70	1	90	12.51
334	simazine	1	80	6.05	1	87	3.88	1	81	1.97
335	simetryn	1	73	7.45	1	95	8.71	1	80	6.87

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
336	spinosyn A	1	74	5.31	5	88	7.77	20	71	6.84
337	spinosyn D	50	71	6.91	100	84	9.10	200	70	10.36
338	spirodiclofen	1	77	2.02	1	89	8.05	2	81	3.67
339	spiromesifen	1	88	2.53	5	90	5.65	10	78	5.47
340	spiroxamine	1	70	4.10	1	95	7.86	2	72	6.99
341	sulfosulfuron	1	81	7.03	5	90	5.19	10	92	6.80
342	sulfotep	1	80	3.45	1	91	5.09	2	83	2.48
343	tau-fluvalinate	50	94	5.86	250	90	5.27	500	86	9.00
344	tebuconazole	1	80	9.26	1	92	5.15	1	87	10.82
345	tebufenozide	1	79	2.74	1	91	6.75	1	78	6.19
346	tebufenpyrad	1	89	3.04	1	88	7.77	2	84	6.31
347	teflubenzuron	1	83	3.96	2.5	83	2.22	5	88	4.51
348	tepraloxydim	1	74	5.39	2.5	91	7.10	5	85	8.67
349	terbufos	2	77	5.72	10	92	7.95	20	73	6.73
350	terbufos-sulfone	1	89	7.32	1	97	5.31	1	86	10.03
351	terbufos-sulfoxide	1	76	7.36	1	95	3.92	2	85	8.31
352	terbuthylazine	1	82	2.09	1	91	9.54	2	83	11.54
353	terbutryn	1	79	4.26	1	95	8.52	1	81	4.61
354	tetraconazole	1	80	5.14	2.5	91	4.80	5	82	6.38
355	tetramethrin	1	79	3.06	1	92	7.59	2	80	8.54
356	thiabendazole	1	70	7.91	1	79	1.27	1	77	2.31
357	thiacloprid	1	81	3.53	1	91	9.09	1	81	4.97
358	thiamethoxam	1	75	7.60	1	82	8.19	1	72	9.15
359	thifensulfuron-methyl	1	81	10.71	5	95	7.09	10	92	11.48
360	thiodicarb	1	87	2.57	2.5	84	9.98	5	79	6.74
361	thiometon	20	80	3.97	100	87	6.09	200	80	4.87
362	thiophanate-methyl	1	70	7.15	2.5	71	7.62	5	87	8.41
363	tolclofos-methyl	20	81	2.36	100	88	7.59	200	80	9.54
364	tolylfluanid	1	88	4.21	5	81	8.21	10	74	7.84
365	triadimefon	1	81	9.76	2.5	94	6.48	5	91	6.87
366	triadimenol	1	78	6.05	5	98	1.76	10	92	3.94
367	triasulfuron	1	81	8.10	1	98	4.85	2	91	6.76
368	triazophos	1	86	3.68	1	92	5.29	1	86	6.40
369	tricyclazole	1	80	2.02	1	84	3.84	1	82	1.59

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		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
370	trifloxystrobin	1	71	3.96	1	95	9.54	1	80	6.75
371	triflumuron	1	85	9.59	1	91	10.29	2	87	10.51
372	triforine	20	75	8.46	100	83	7.82	200	80	5.16
373	trichlorfon	20	72	5.51	50	93	8.41	100	80	9.80
374	trinexapac ethyl	2	82	3.25	10	95	3.46	20	85	4.10
375	triticonazole	1	79	4.42	2.5	96	6.33	2	90	6.81
376	vamidothion	1	81	4.66	5	86	5.24	10	78	5.71
377	zoxamide	1	80	1.52	2.5	94	4.05	2	84	6.17
378	2-hydroxypropyl-mepanipyrim	1	97	3.52	1	91	0.78	1	88	3.58
379	aclonifen	200	92	1.81	500	86	5.35	1000	83	2.59
380	ametoctradin	1	83	2.41	1	79	6.10	1	75	2.51
381	asulam	2	95	2.41	10	89	4.98	10	86	6.69
382	BAC C10	1	81	2.14	1	77	3.67	1	79	1.11
383	BAC C12	1	83	2.34	1	79	1.31	1	82	2.14
384	BAC C14	1	84	2.10	1	80	2.08	1	77	1.94
385	BAC C16	1	81	0.89	1	74	1.01	1	76	2.58
386	BAC C18	1	91	3.00	1	94	1.31	1	90	3.67
387	BAC C8	1	83	1.48	1	82	3.14	1	76	3.40
388	bixafen	1	99	3.71	1	93	3.96	1	90	5.47
389	BTS 44595	1	96	4.54	1	92	3.16	2	86	2.44
390	BTS 44596	2	92	4.10	5	91	2.08	10	88	2.70
391	carboxin	1	73	2.84	1	71	2.59	10	72	4.58
392	cloprop	1	106	6.71	2.5	100	2.91	5	97	3.18
393	clopyralid	5	74	3.14	50	71	2.59	50	73	3.96
394	cycloxydim	1	71	2.41	1	69	3.58	2	72	6.87
395	DDAC	1	79	1.11	1	72	1.97	2	75	1.25
396	dithianon	200	84	3.18	1000	80	9.10	2000	77	10.11
397	diafenthiuron	1	92	2.19	5	88	2.80	10	82	4.22
398	dicamba	1	104	3.64	2.5	101	4.58	5	98	4.98
399	dinotefuran	1	91	6.27	2.5	85	6.87	5	82	5.47
400	empenthrin	5	77	4.57	25	73	3.67	40	75	2.56
401	ethametsulfuron-methyl	1	100	2.14	1	94	3.96	1	91	2.59
402	etoxazol	1	82	3.81	1	75	3.98	2	78	4.47
403	fenamidone	1	98	3.41	1	92	6.69	2	89	3.58

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
404	fenpyrazamin	1	86	2.90	1	82	2.47	2	79	4.21
405	fensulfothion oxon	1	78	2.42	1	81	2.14	2	78	3.61
406	fensulfothion sulfone	1	90	2.47	2.5	89	2.99	5	83	4.23
407	fensulfothion-PO-sulfon	1	97	2.76	1	93	4.42	2	89	4.21
408	fentin	200	71	3.71	500	68	5.47	500	70	3.96
409	fipronil desulfinyl	1	87	3.57	1	90	7.13	1	87	6.30
410	fipronil sulfone	1	100	3.20	1	103	4.98	1	100	5.70
411	flubendiamid	1	99	1.27	1	97	5.35	1	94	5.70
412	fluopicolide	1	98	0.89	2.5	96	4.15	5	93	3.24
413	flurochloridone	2	99	0.81	10	95	4.58	20	92	4.15
414	flutolanil	1	99	3.40	1	97	6.69	2	94	0.39
415	fluxapyroxad	1	99	5.20	1	95	8.45	2	92	5.35
416	hexaflumuron	1	101	2.14	1	97	6.10	1	94	4.98
417	chlorbufam	200	89	9.24	500	85	3.17	1000	88	4.61
418	chlorpropham	200	99	3.11	500	95	3.58	500	92	4.21
419	chlorpyrifos (ethyl)	1	80	7.25	1	91	10.22	5	84	9.89
420	iprodione	10	91	2.13	100	87	4.98	100	84	2.14
421	isopyrazam	1	88	3.67	1	84	2.09	1	81	6.38
422	meptyldinocap	1	97	1.11	2.5	93	4.15	5	90	2.14
423	metamitron-desamino	1	79	5.37	2.5	72	5.16	10	74	7.15
424	metominostrobin	1	98	3.41	1	97	4.98	2	94	6.14
425	metrafenone	1	98	1.84	1	97	6.87	2	94	2.14
426	neoquassin	1	88	2.74	5	84	2.81	10	81	4.00
427	nitenpyram	1	90	3.74	5	86	4.15	20	83	6.14
428	oxamyl-oxime	1	92	5.43	5	88	2.36	10	85	2.81
429	penflufen	1	84	6.01	1	83	4.21	2	80	4.21
430	penthiopyrad	1	90	6.01	1	86	4.23	2	80	6.05
431	pethoxamid	1	98	4.51	1	94	5.07	2	91	6.69
432	phosmet	1	79	4.66	1	95	1.17	1	85	2.59
433	quassin	1	89	4.23	5	85	2.97	10	82	2.81
434	spirotetramat	1	99	4.23	1	97	4.70	1	94	4.23
435	spirotetramate –enol	1	81	7.31	1	80	2.20	1	83	7.45
436	spirotetramate –enol-glukosid	2	80	2.14	10	73	6.01	10	76	3.31
437	spirotetramate –keto-hydroxy	1	86	1.59	2.5	82	5.31	5	79	3.18

No	Compound	(i) Fruit and vegetables			(ii) Cereals			(iii) Tea		
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438	spirotetramate –mono-hydroxy	1	92	5.20	1	88	7.40	2	82	6.84
439	terbuthylazine-desethyl	1	98	5.16	1	94	2.14	1	91	4.23
440	TFNA metab flonicamidu	200	83	6.99	1000	82	9.24	2000	84	2.47
441	TFNG metab flonicamidu	5	77	2.09	25	73	1.76	50	67	2.08
442	tolfenpyrad	1	88	0.92	1	84	4.85	2	81	4.42
443	tribenuron-methyl	1	88	2.14	2.5	84	6.14	5	81	5.1
444	triclopyr	2	100	6.14	10	96	5.10	10	93	2.14
445	vamidothion-sulfon	1	92	2.14	2.5	88	4.21	5	82	4.57
446	vamidothion-sulfoxid	1	81	3.61	2.5	80	2.50	5	82	5.07

Annex II: Presentation on method transfer provided by CAIQ

T4.2. Multi-analyte / multi-matrix screening method for pesticide residues in fruits and vegetables (including tea) and fruit juices

Lead: VSCHT, **contribution** from CAIQ

Liquid chromatography conditions for analytes determined in U-HPLC-(ESI+)-MS/MS

Column temperature	**40 °C**	
Analytical column temperature	10 °C	
Injection volume	3 µL	
Mobile phases	ESI (+)	A: 5 mM ammonium formate in water (0.2 % formic acid) B: 5 mM ammonium formate methanol (0.2 % formic acid)
	ESI (−)	C: 5 mM ammonium acetate in water D: 5 mM ammonium acetate in methanol

Gradient of mobile phases for U-HPLC-(ESI−/ESI+)-MS/MS

Time [min]	Flow rate [mL/min]	Mobile phases composition [%]	
		A/C	B/D
0	0,30	90	10
1	0,35	50	50
11	0,45	0	100
12	0,50	0	100
14	0,50	0	100
16	0,40	90	10

ESI ionization conditions

Ionization	ESI+/ESI−
Sheath/auxiliary gas (N_2)	45/10 arb. u.
Capillary temperature	300 °C
Heater temperature	300 °C
ESI needle voltage	+/− 3,3 kV

Conditions of acquisition mode *fullMS*

Resolution	70 000 FWHM
Acquisition speed	1,5 Hz
Mass range	m/z 100-1000
Automatic gain control (AGC target)	$3e^6$
Maximum inject time (max IT)	200 ms

Conditions of acquisition mode ddMS2

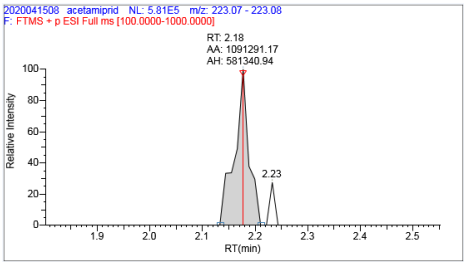
Resolution	17 500 FWHM
Acquisition speed	12 Hz
Mass range	m/z 50 - m/z fragmented analyte (+25)
Isolation window width	1 m/z
Automatic gain control (AGC target)	$1e^5$
Maximum inject time (max IT)	50 ms
Normalized collision energy (NCE)	Table S1
Dynamic exclusion	3 s
Intensity threshold	$2e^3$

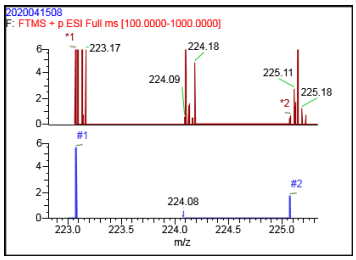
Determination of pesticide residues in tea leaves

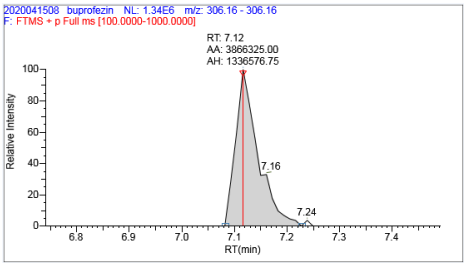
Extraction and clean-up

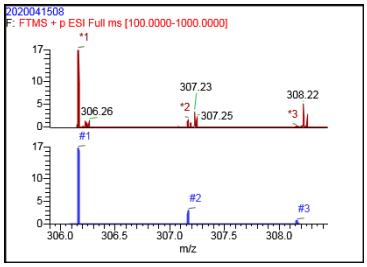
Sample (1 g) is weighed into a 50 mL polypropylene tube, 10 mL of water (0.2 % formic acid, *v/v*) is then added, sample shaken and left to soak the sample matrix. After addition of 10 mL of acetonitrile, sample is shaken on a horizontal laboratory shaker for 30 min at 240 RPM followed by addition of magnesium sulfate (4 g) and sodium chloride (1 g) and tube vigorously shaken in hand for 1 min. Sample is then centrifuged at 10 000 RPM (5 min) and 5 mL of the extract is removed from the top acetonitrile layer to a new polypropylene tube which is placed to a freezer (− 20 ° C) for 2 h to let freeze out the unwanted matrix components. Purified extract is then micro-filtered (0.2 µm filter) prior to instrumental analysis.

Screening Results

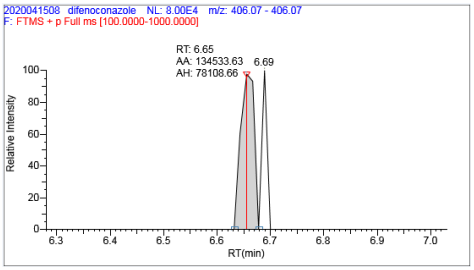


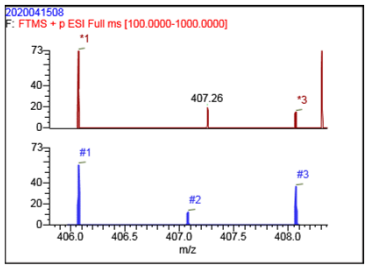


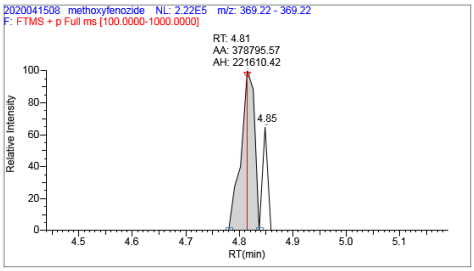


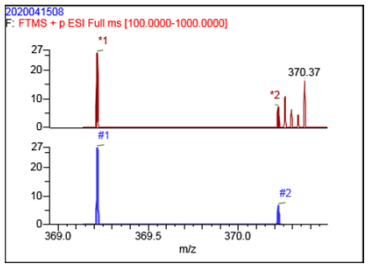


Screening Results









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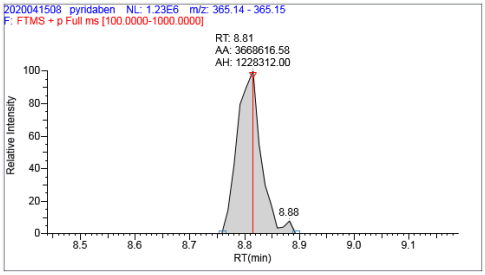

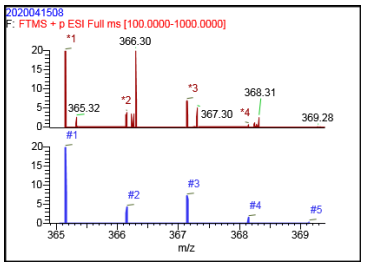

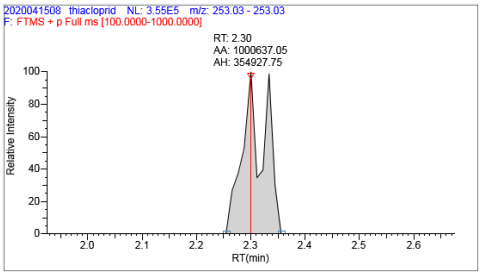

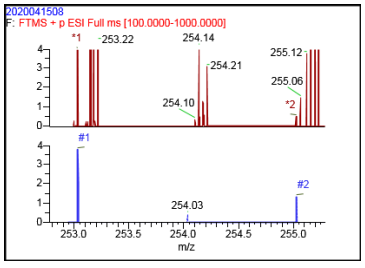

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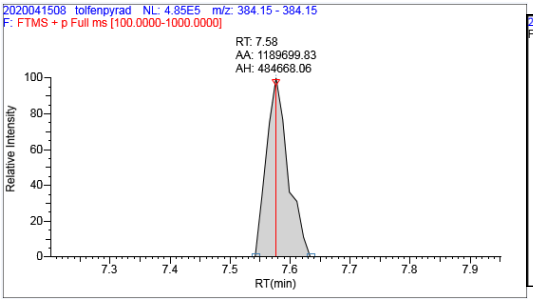

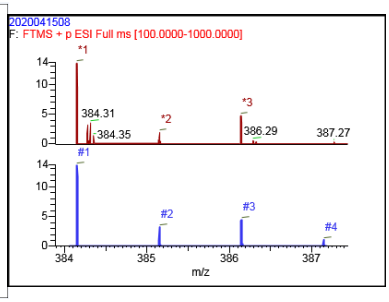

Thank you for your attention

Annex III: Report demonstrating transferability of developed multimethod by CAIQ

Report on applicability of multi-analyte / multi-matrix screening method for pesticide residues in fruits and vegetables and tea at CAIQ

1. Screening of pesticide multi-residues in fruits and vegetables (including tea) and fruit juices (Q-Exactive)

1.1 Scope of application

This method provides a method for the detection of pesticide residues in fruits and vegetables by high resolution liquid chromatography coupled to quadrupole orbitrap mass spectrometry.

This method is suitable for the determination of 115 pesticide residues in fruits and vegetables.

1.2 Principles

The residual pesticides in the samples were extracted by the acidic acetonitrile solution, then centrifuged and cleaned up. The liquid was detected by liquid chromatography coupled to quadrupole orbitrap mass spectrometry (Q-Exactive) and quantified by external standard method.

1.3 Reagents and materials

Only analytical pure reagents are used unless otherwise specified.

1.3.1 Acetonitrile: chromatographic purity.

1.3.2 Formic acid: analytical reagent (AR, 37%).

1.3.3 Sodium chloride: analytical reagent.

1.3.4 Anhydrous Magnesium Sulfate: analytical reagent.

1.3.5 Ultra pure water (MiliQ).

1.3.6 Formic acid: guaranteed reagent.

1.3.7 Ammonium formate: chromatographic purity.

1.3.8 Acetone: analytical reagent, heavy distillation.

1.3.9 4 mmol ammonium formate solution: 0.252 g ammonium formate was diluted to 1000 mL.

1.3.10 0.1% formic acid acetonitrile solution (volume fraction).

1.3.11 Pesticides and related chemicals standard material: purity of not less than 95%.

1.3.12 Standard solution for pesticides and related chemicals

1.3.12.1 Standard stock solution

5 mg-10 mg pesticide standard substances were respectively selected in 10 ml volumetric flask. According to the solubility of standard materials, solvents such as methanol, toluene, acetone, acetonitrile or isooctane were dissolved and fixed to scale. The standard reserve solution is preserved at 0-4 ℃, and can be used for one year.

1.3.12.2 Standard mixed solution

The standard reserve liquid is mixed according to a certain concentration. It can be saved at 4 degrees centigrade for one month.

1.4 Instruments and equipment

1.4.1 liquid chromatography coupled to quadrupole orbitrap mass spectrometry an electrospray ion source (ESI) (Thermo, Q-Exactive).

1.4.2 Analysis of scales: a sense of 0.1 mg and 0.01 g (Mettler ToleDo, XP105DR).

1.4.3 Ultra-pure Water Purifier (Millipore, milli-Q advantage A10)..

1.4.4 Vortex mixer (Vortex, Kylin-Bell)..

1.4.5 Centrifuge (Beckman, avanti J-E)..

1.4.6 Sample Concentrator (Zymark, TurboVap LV)..

1.4.7 Level oscillator (Yamato, SA31)..

1.4.8 Ultrasonic generator (Shengda, SD-8200H)...

1.4.9 Thermostat box (Haier, DW-25L262)..

1.5 Samples preservation

Keep the samples in the refrigerator at 4℃.

1.6 Determination procedure

1.6 .1 Extraction and clean-up

Sample (1 g) is weighed into a 50 mL polypropylene tube, 10 mL of water (0.2 % formic acid, *v/v*) is then added, sample shaken and left to soak the sample matrix. After addition of 10 mL of acetonitrile, sample is shaken on a horizontal laboratory shaker for 30 min at 240 RPM followed by addition of magnesium sulfate (4 g) and sodium chloride (1 g) and tube vigorously shaken in hand for 1 min. Sample

is then centrifuged at 10 000 RPM (5 min) and 5 mL of the extract is removed from the top acetonitrile layer to a new polypropylene tube which is placed to a freezer (−20 °C) for 2 h to let freeze out the unwanted matrix components. Purified extract is then micro-filtered (0.2 µm filter) prior to instrumental analysis.

1.6.2 Analytical conditions for Q-Exactive

1.6.2.1 LC condition

a) Column: Accucore aQ (100 x 2.1, 2.6 µm; Thermo Scientific, USA; Phenomenex) or equivalent;

b) The mobile phase and the gradient elution conditions are shown in Table 1 and 2.

c) Column temperature: 40 °C;

d) The sampling volume: 5 µL;

Table 1: LC conditions for analytes determined in HPLC-(ESI+)-MS/MS

Mobile phases	ESI (+)	A: 5 mM ammonium formate in water (0.2 % formic acid) B: 5 mM ammonium formate methanol (0.2 % formic acid)
	ESI (−)	C: 5 mM ammonium acetate in water D: 5 mM ammonium acetate in methanol
Mobile phases gradient	**Table 2**	

Table 2: ***Gradient of mobile phases for HPLC-(ESI−/ESI+)-MS/MS***

Time [min]	**Flow rate [mL/min]**	**Mobile phases composition [%]**	
		A/C	**B/D**
0	0,30	90	10
1	0,35	50	50
11	0,45	0	100
12	0,50	0	100
14	0,50	0	100
16	0,40	90	10

1.6.2.2 **Mass spectrometry**

The data are acquired in *fullMS-data dependentMS*^{2} (*fullMS-ddMS*^{2}) mode under conditions shown in **Tables 3 – 5**. Monitored analytes are identified according to detection of their precursor ions (*fullMS*) and by comparison of analytes' retention times in samples with calibration standards. Their presence confirmed through detection of at least one fragment ion detection (*ddMS*^{2}) and comparison of theoretical and experimental isotopic pattern detection. For the system control and data evaluation, Xcalibur® 4.0 (Thermo Scientific, USA) software is used.

Table 3: ESI ionization conditions

Ionization	ESI+/ESI−
Sheath/auxiliary gas (N_2)	45/10 arb. u.
Capillary temperature	300 °C
Heater temperature	300 °C
ESI needle voltage	+/− 3,3 kV

Table 4: Conditions of acquisition mode *fullMS*

Resolution	70 000 FWHM
Acquisition speed	1,5 Hz
Mass range	*m/z* 100-1000
Automatic gain control (AGC target)	$3e^6$
Maximum inject time (max IT)	200 ms

Table 5: Conditions of acquisition mode *ddMS2*

Resolution	17 500 FWHM
Acquisition speed	12 Hz
Mass range	*m/z* 50 - *m/z* fragmented analyte (+25)
Isolation window width	1 *m/z*
Automatic gain control (AGC	$1e^5$
Maximum inject time (max IT)	50 ms
Normalized collision energy	**Table S1**
Dynamic exclusion	3 s
Intensity threshold	$2e^3$

1.6.3 Qualitative determination

In this method, the liquid chromatography tandem mass spectrometry was quantified by the external standard calibration curve method. In order to reduce the effect of matrix on quantitative determination, matrix-matched calibration curve was used to quantify the concentration in the samples. And the response value of pesticide in the tested samples was guaranteed to be within the linear range of the instrument.

1.6.4 Parallel test

The same test is carried out in parallel to the same sample according to the above steps.

1.6.5 Blank test

Except for no sample, all of these steps are carried out according to the above steps.

1.7. Result calculation

The content of the pesticides component in the sample was measured using Q-Exactive and quantified by the calibration curve of external standard. The quantitative results of the standard curve method were calculated according to the following formula (1).

X=c×(V/m)×(1000/1000)　（1）

In the formula:

X — The content of the measured component in the sample, mg/kg;

C — The concentration of each substance in the standard working fluid, ug/mL;

V — The final volume of the liquid, mL;

m —The sample weight represented by the final sample solution, g.

The results of the calculation should be deducted from the blank value.

1.8 Determination of LODs values

The LODs (limit of detection) values were 1-1000 μg/kg using this method.

1.9 Recovery rate

The recovery rate of this method is70-112%.

Table 6 Exact masses m/z of precursor ions and fragments of pesticide residues, retention times and normalized collision energies.

No	Analyte	Formula	*m/z*	Fragment 1		Fragment 2		Fragment 3	
				Formula	*m/z*	Formula	*m/z*	Formula	*m/z*
1	acephate	C4H10NO3PS	1,830,114	C2H8O3PS	1,429,926	CH6O3PS	1,289,770	CH7O3NP	1,120,158
2	acetamiprid	C10H11ClN4	2,220,667	C6H5NCl	1,260,105	C3H6N	560,495	C10H11N4	1,870,978
3	atrazine	C8H14ClN5	2,150,932	C5H9N5Cl	1,740,541	C4H6N3	960,556	C2H3N3Cl	1,040,010
4	azinphos-methyl	C10H12N3O3PS2	3,170,052	C2H8O3PS	1,429,926	C8H6ON	1,320,444	C8H10O3N	1,680,655
5	azoxystrobin	C22H17N3O5	4,031,163	C21H14O4N3	3,720,979	C20H14O3N3	3,441,030	C19H11O3N3	3,290,795
6	benalaxyl	C20H23NO3	3,251,672	C12H18O2N	2,081,332	C10H14N	1,481,121	C19H20O2N	2,941,489
7	bitertanol	C20H23N3O2	3,371,785	C2H4N3	700,400	C18H21O2	2,691,536	C6H11O	990,804
8	buprofezin	C16H23N3OS	3,051,556	C9H17ON2S	2,011,056	C5H10NS	1,160,528	C5H9ON2S	1,450,430
9	cadusafos	C10H23O2PS2	2,700,872	C2H8O2PS2	1,589,698	C6H16O2PS2	2,150,324	H4O2PS2	1,309,385
10	carbaryl	C12H11NO2	2,010,784	C10H9O	1,450,648	C9H9	1,170,699	C8H7	1,030,542
11	carbendazim	C9H9N3O2	1,910,689	C8H6ON3	1,600,505	C7H6N3	1,320,556	C6H5N2	1,050,447
12	clofentezine	C14H8Cl2N4	3,020,121	C7H5NCl	1,380,105	C7H4N3	1,300,400	C5H4N	780,338
13	clomazone	C12H14ClNO2	2,390,708	C7H6Cl	1,250,153	C7H5	890,386	C5H4Cl	989,996
14	clothianidin	C6H8ClN5O2S	2,490,082	CNS	579,757	C6H5N4S	1,650,240	C5H2N4S	1,500,006
15	cyproconazole	C15H18ClN3O	2,911,133	C4H6O	700,413	C7H6Cl	1,250,153	C7H4OCl	1,389,945
16	desmedipham	C16H16N2O4	3,001,105	C8H10O3N	1,680,655	C7H6O2N	1,360,393	C9H12O3N	1,820,812
17	desmetryn	C8H15N5S	2,131,043	C5H10N5S	1,720,651	C3H4N3	820,400	C2H7N2S	910,324
18	diazinon	C12H21N2O3PS	3,041,005	C8H13N2S	1,690,794	C8H13ON2	1,531,022	H4O3PS	1,149,613
19	difenoconazole	C19H17Cl2N3O3	4,050,641	C13H9OCl2	2,510,025	C17H15O3Cl2	3,370,393	C16H13OCl2	2,910,338
20	diflubenzuron	C14H9ClF2N2O2	3,100,315	C14H7O2N2ClF	2,890,186	C7H4ONF2	1,560,266	C7H4N2Cl	1,510,068
21	dimethoate	C5H12NO3PS2	2,289,991	C4H8O3PS2	1,989,647	C3H8O2PS2	1,709,698	C3H6NS	880,215
22	dimethomorph	C21H22ClNO4	3,871,232	C9H9O3	1,650,546	C17H14O3Cl	3,010,626	C7H4OCl	1,389,945
23	dimoxystrobin	C19H22N2O3	3,261,625	C11H13O2N2	2,050,972	C16H16ON	2,381,226	C8H6N	1,160,495
24	diniconazole	C15H17Cl2N3O	3,250,743	C2H4N3	700,400	C7H5Cl2	1,589,763	C7H3OCl2	1,729,555
25	diuron	C9H10Cl2N2O	2,320,165	C3H6ON	720,444	C6H4NCl2	1,599,715	C5H3Cl2	1,329,606
26	ethoprophos	C8H19O2PS2	2,420,559	C3H10O2PS2	1,729,854	H4O2PS2	1,309,385	C3H8O2PS	1,389,977
27	fenamiphos	C13H22NO3PS	3,031,053	C8H10O3PS	2,170,083	C8H13O3NPS	2,340,348	C11H19O3NPS	2,760,818
28	fenarimol	C17H12Cl2N2O	3,300,321	C4H5N2	810,447	C16H11ONCl	2,680,524	C7H4OCl	1,389,945
29	fenazaquin	C20H22N2O	3,061,727	C4H9	570,699	C12H17	1,611,325	C8H7ON2	1,470,553
30	fenbuconazole	C19H17ClN4	3,361,136	C7H6Cl	1,250,153	C9H9N3Cl	1,940,480	C7H7	910,542
31	fenbutatin-oxide	C60H78OSn2	10,544,080	C7H7	910,542	C18H15Sn	3,510,190	C13H13Sn	2,890,034
32	fenhexamid	C14H17Cl2NO2	3,010,631	C7H13	971,012	C6H6ONCl2	1,779,821	C6H5ONCl	1,420,054
33	fenthion	C10H15O3PS2	2,780,195	C8H5O3	1,490,233	C8H9S2	1,690,140	C8H9	1,050,699

34	flufenacet	C14H13F4N3O2S	3,630,659	C11H13ONF	1,940,976	C8H7ONF	1,520,506	C10H13NF	1,661,027
35	flufenoxuron	C21H11ClF6N2O3	4,880,357	C13H7ONClF4	3,040,158	C14H6ON2ClF4	3,290,110	C13H6ONClF3	2,840,095
36	flumioxazin	C19H15FN2O4	3,541,010	C19H16O4N2F	3,551,089	C18H16O3N2F	3,271,139	-	-
37	flusilazole	C16H15F2N3Si	3,150,998	C8H8N3F	1,650,697	C8H9N3F	1,660,775	C8H9N3F2	1,850,759
38	flutriafol	C16H13F2N3O	3,011,021	C2H4N3	700,400	C7H4OF	1,230,241	C14H11OF2	2,330,772
39	hexythiazox	C17H21ClN2O2S	3,521,007	C10H11ONClS	2,280,244	C9H11NCl	1,680,575	C10H9ONCl	1,940,367
40	chlorantraniliprole	C18H14BrCl2N5O2	4,809,702	C17H10O2N4[81]BrCl2	4,529,338	C9H4ON3[81]BrCl	2,859,200	C9H4ON3BrCl	2,839,221
41	chlorfenvinphos	C12H14Cl3O4P	3,579,690	C4H12O4P	1,550,468	C2H8O4P	1,270,155	C7H4OCl2P	2,049,371
42	imazalil	C14H14Cl2N2O	2,960,478	C7H5Cl2	1,589,763	C3H5N2	690,447	C8H7Cl2	1,729,919
43	imazosulfuron	C14H13ClN6O5S	4,120,351	C6H10O2N3	1,560,768	C7H6N2Cl	1,530,214	C7H7O2N3ClS	2,319,942
44	imidacloprid	C9H10ClN5O2	2,550,518	C9H10N4Cl	2,090,589	C9H11N4	1,750,978	C3H6N3	840,556
45	indoxacarb	C22H17ClF3N3O7	5,270,702	C9H7O2NF3	2,180,423	C12H10O2N2Cl	2,490,425	C8H5NCl	1,500,105
46	linuron	C9H10Cl2N2O2	2,480,114	C6H4NCl2	1,599,715	C8H7ON2Cl	1,820,241	C6H5NCl2	1,609,794
47	malathion	C10H19O6PS2	3,300,355	C8H14O5PS2	2,850,015	C6H7O3	1,270,390	C10H20O6PS2	3,310,433
48	metalaxyl	C15H21NO4	2,791,465	C12H18ON	1,921,383	C13H18O2N	2,201,332	C11H14N	1,601,121
49	metamitron	C10H10N4O	2,020,849	C9H11N4	1,750,978	C7H6N	1,040,495	C10H10ON2	1,740,788
50	metazachlor	C14H16ClN3O	2,770,976	C11H13ONCl	2,100,680	C9H12N	1,340,964	-	-
51	metconazole	C17H22ClN3O	3,191,446	C4H6O	700,413	C7H6Cl	1,250,153	C11H10Cl	1,770,466
52	methacriphos	C7H13O5PS	2,400,216	C2H8O3PS	1,429,926	C6H10O4PS	2,090,032	C2H6O2PS	1,249,821
53	methamidophos	C2H8NO2PS	1,410,008	C2H6O2PS	1,249,821	CH4O2P	789,943	CH5ONPS	1,099,824
54	methidathion	C6H11N2O4PS3	3,019,613	C4H5O2N2S	1,450,066	C3H5ON2	850,396	C2H3ON2	710,240
55	methiocarb	C11H15NO2S	2,250,818	C9H13OS	1,690,682	C8H9O	1,210,648	C8H10O	1,220,726
56	methomyl	C5H10N2O2S	1,620,457	C3H6NS	880,215	C3H8ONS	1,060,321	CH5OS	650,056
57	methoxyfenozide	C22H28N2O3	3,682,094	C9H9O2	1,490,597	C18H21O3N2	3,131,547	C9H9O	1,330,648
58	metobromuron	C9H11BrN2O2	2,579,998	C6H5NBr	1,699,600	C8H8ON2	1,480,631	C6H6NBr	1,709,678
59	metsulfuron-methyl	C14H15N5O6S	3,810,738	C6H7O2N4	1,670,564	C8H7O4S	1,990,060	C5H9ON4	1,410,771
60	mevinphos	C7H13O6P	2,240,444	C6H10O5P	1,930,260	C2H8O4P	1,270,155	C5H7O2	990,441
61	monuron	C9H11ClN2O	1,980,554	C3H6ON	720,444	C6H5NCl	1,260,105	C5H4Cl	989,996
62	myclobutanil	C15H17ClN4	2,881,136	C2H4N3	700,400	C7H6Cl	1,250,153	C9H8Cl	1,510,309
63	naled	C4H7Br2Cl2O4P	3,777,820	C2H8O4P	1,270,155	C2HBr2Cl2	2,527,817	-	-
64	napropamide	C17H21NO2	2,711,567	C7H15ON	1,291,148	C12H11O	1,710,804	C13H11O2	1,990,754
65	neburon	C12H16Cl2N2O	2,740,634	C5H14N	881,121	C6H12ON	1,140,913	C4H9	570,699
66	nicosulfuron	C15H18N6O6S	4,101,003	C7H8O3N3	1,820,560	C8H9O3N2S	2,130,328	C13H12O6N5S	3,660,503
67	oxamyl	C7H13N3O3S	2,190,672	C3H6ON	720,444	C3H8O2N	900,550	-	-
68	penconazole	C13H15Cl2N3	2,830,638	C2H4N3	700,400	C7H5Cl2	1,589,763	C8H7Cl2	1,729,919
69	pencycuron	C19H21ClN2O	3,281,337	C7H6Cl	1,250,153	C13H13NCl	2,180,731	C14H14ON2Cl	2,610,789
70	pendimethalin	C13H19N3O4	2,811,370	C8H10O4N3	2,120,666	C8H8O3N3	1,940,560	C8H9O3N3	1,950,638

71	permethrin	C21H20Cl2O3	3,900,784	C13H11O	1,830,804	C21H20O3Cl	3,551,095	C21H19O3	3,191,329
72	phenmedipham	C16H16N2O4	3,001,105	C9H12O3N	1,820,812	C7H6O2N	1,360,393	C7H8O3N	1,540,499
73	piperonyl butoxide	C19H30O5	3,382,088	C9H11	1,190,855	C11H13O2	1,770,910	C10H11O	1,470,804
74	pirimicarb	C11H18N4O2	2,381,424	C3H6ON	720,444	C9H16ON3	1,821,288	C4H9N2	850,760
75	pirimicarb-desmethyl	C10H16N4O2	2,241,268	C3H6ON	720,444	C8H14ON3	1,681,131	C8H10O2N3	1,800,768
76	pirimiphos-ethyl	C13H24N3O3PS	3,331,271	C9H16N3S	1,981,059	C9H16ON3	1,821,288	C7H12N3S	1,700,746
77	pirimiphos-methyl	C11H20N3O3PS	3,050,958	C9H14N3	1,641,182	C5H6N3	1,080,556	C7H10N3	1,360,869
78	profenofos	C11H15BrClO3PS	3,719,346	C6H6O3BrClPS	3,028,642	C5H5OBrPS	2,228,977	-	-
79	prochloraz	C15H16Cl3N3O2	3,750,303	C12H13O2NCl3	3,080,006	C9H7O2NCl3	2,659,537	C11H13ONCl3	2,800,057
80	prometon	C10H19N5O	2,251,584	C4H8ON5	1,420,723	C7H14ON5	1,841,193	C2H4ON3	860,349
81	prometryn	C10H19N5S	2,411,356	C4H8N5S	1,580,495	C7H14N5S	2,000,964	C3H6N3S	1,160,277
82	propachlor	C11H14ClNO	2,110,758	C8H9ONCl	1,700,367	C6H8N	940,651	C7H8N	1,060,651
83	propamocarb	C9H20N2O2	1,881,519	C4H8O2N	1,020,550	C7H14O2N	1,441,019	C2H4O2N	740,237
84	propiconazole	C15H17Cl2N3O2	3,410,692	C7H5Cl2	1,589,763	C8H5OCl2	1,869,712	C7H5O2Cl2	1,909,661
85	pyraclostrobin	C19H18ClN3O4	3,870,980	C10H12O3N	1,940,812	C16H11ON3Cl	2,960,585	C9H10O2N	1,640,706
86	pyridaben	C19H25ClN2OS	3,641,371	C15H18ON2ClS	3,090,823	C11H15	1,471,168	-	-
87	pyrimethanil	C12H13N3	1,991,104	C5H8N	820,651	C5H6N	800,495	C12H10N2	1,820,838
88	pyriproxyfen	C20H19NO3	3,211,359	C5H6ON	960,444	C12H9O2	1,850,597	C15H15O2	2,271,067
89	quinalphos	C12H15N2O3PS	2,980,536	C8H7ON2	1,470,553	C8H7N2S	1,630,324	H4O3PS	1,149,613
90	quinoxyfen	C15H8Cl2FNO	3,069,961	C9H6ONCl2	2,139,821	C9H5NCl	1,620,105	C15H8ONClF	2,720,273
91	quizalofop	C17H13ClN2O4	3,440,558	C16H12O2N2Cl	2,990,582	C15H12ON2Cl	2,710,633	C14H9O2N2Cl	2,720,347
92	rimsulfuron	C14H17N5O7S2	4,310,564	C7H8O3N3	1,820,560	C13H17O4N4S	3,250,965	C7H11O4N2S2	2,510,155
93	rotenone	C23H22O6	3,941,411	C14H13O2	2,130,910	C11H12O3	1,920,781	C12H11O3	2,030,703
94	simazine	C7H12ClN5	2,010,776	C4H7N3Cl	1,320,323	C4H6N3	960,556	C2H3N3Cl	1,040,010
95	simetryn	C8H15N5S	2,131,043	C5H10N5S	1,720,651	C3H4N3	820,400	C2H7N2S	910,324
96	spirodiclofen	C21H24Cl2O4	4,101,046	C5H11	710,855	C15H15O3Cl2	3,130,393	C19H12O4Cl2	3,740,107
97	spiromesifen	C23H30O4	3,702,139	C17H21O3	2,731,485	C17H19O2	2,551,380	C12H11O2	1,870,754
98	tebuconazole	C16H22ClN3O	3,071,446	C4H6O	700,413	C7H6Cl	1,250,153	C9H8	1,160,621
99	tetraconazole	C13H11Cl2F4N3O	3,710,210	C2H4N3Cl2F	1,589,761	C4H6O	700,413	C9H7Cl	1,500,231
100	tetramethrin	C19H25NO4	3,311,778	C18H24O2N	2,861,802	C19H24O3N	3,141,751	C9H10O2N	1,640,706
101	fenoxaprop-ethyl	C18H16ClNO5	361,0712	C15H11O3NCl	288,0422	C8H9O	121,0648	C8H7O	119,0491
102	thiabendazole	C10H7N3S	2,010,355	C9H7N2S	1,750,324	C8H7N2	1,310,604	C6H6N	920,495
103	thiacloprid	C10H9ClN4S	2,520,231	C6H5NCl	1,260,105	C6H4N	900,338	C5H4Cl	989,996
104	thiamethoxam	C8H10ClN5O3S	2,910,187	C8H11ON4S	2,110,648	C4H3NClS	1,319,669	C8H10ON4S	2,100,570
105	triadimefon	C14H16ClN3O2	2,930,926	C11H14OCl	1,970,728	C6H11O2	1,150,754	C12H14O2Cl	2,250,677
106	triazophos	C12H16N3O3PS	3,130,645	C8H8ON3	1,620,662	C7H7N2	1,190,604	H4O3PS	1,149,613
107	tricyclazole	C9H7N3S	1,890,355	C7H6NS	1,360,215	C8H7N2S	1,630,324	C6H5S	1,090,106

108	trifloxystrobin	C20H19F3N2O4	4,081,291	C9H7NF3	1,860,525	C8H6N	1,160,495	C9H9N	1,310,730
109	zoxamide	C14H16Cl3NO2	3,350,241	C8H5OCl2	1,869,712	C8H8NOCl2	2,039,977	C6H11NCl	1,320,575
110	carboxin	C12H13NO2S	2,350,662	C6H7O2S	1,430,161	C6H6NS	1,240,215	C10H10O2NS	2,080,427
111	fenamidone	C17H17N3OS	3,111,087	C6H6N	920,495	C15H14N3	2,361,182	C8H7	1,030,542
112	fluopicolide	C14H8Cl3F3N2O	3,819,649	C7H3OCl2	1,729,555	C14H7N2Cl3F3	3,649,621	C7H6ONClF3	2,120,085
113	pethoxamid	C16H22ClNO2	2,951,334	C10H11	1,310,855	C14H17ONCl	2,500,993	C4H9O	730,648
114	phosmet	C11H12NO4PS2	3,169,940	C9H6O2N	1,600,393	C8H5O2	1,330,284	C7H5O	1,050,335
115	spirotetramat	C21H27NO5	3,731,884	C18H24O3N	3,021,751	C17H20O2N	2,701,489	C20H28O3N	3,302,064

Table 7 Validation characteristics for matrices (i) fruit and vegetables, (ii) herbal tea. The artificial contamination levels for determination of recoveries and repeatabilities were 200, 1 000 and 2 000 µg/kg (n = 6) for matrices fruit and vegetables and herbal tea, respectively.

No	Compound	(i) Fruit and vegetables			(ii) Tea		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
1	acephate	1	95	3.49	10	83	6.70
2	acetamiprid	1	87	4.22	10	86	6.75
3	atrazine	20	92	7.14	100	81	10.13
4	azinphos-methyl	1	97	1.80	2	87	3.98
5	azoxystrobin	1	92	8.23	10	93	8.79
6	benalaxyl	1	74	2.42	10	76	6.77
7	bitertanol	1	87	6.40	1	82	8.97
8	buprofezin	1	82	4.73	5	88	6.41
9	cadusafos	5	93	6.15	50	90	3.47
10	carbaryl	1	87	3.49	10	82	2.88
11	carbendazim	10	99	7.84	100	82	7.97
12	clofentezine	1	84	5.42	10	72	8.14
13	clomazone	20	71	5.49	100	88	3.47
14	clothianidin	1	80	3.20	1	84	4.15
15	cyproconazole	1	72	1.11	1	86	5.29

16	desmedipham	50	86	5.07	200	89	8.41
17	desmetryn	5	72	4.78	50	91	7.48
18	diazinon	1	87	7.74	1	80	8.74
19	difenoconazole	1	80	2.53	1	75	3.79
20	diflubenzuron	1	81	2.26	1	88	6.99
21	dimethoate	1	80	6.66	1	81	11.09
22	dimethomorph	1	83	6.67	2	81	6.84
23	dimoxystrobin	1	93	5.79	1	82	8.74
24	diniconazole	200	92	5.78	1000	84	11.04
25	diuron	5	83	8.24	100	70	7.86
26	ethoprophos	1	90	0.25	1	102	8.47
27	fenamiphos	1	81	2.37	10	90	2.97
28	fenarimol	1	80	7.75	1	84	8.98
29	fenazaquin	1	75	3.12	1	82	4.58
30	fenbuconazole	1	79	9.66	20	85	6.79
31	fenbutatin-oxide	1	74	4.09	1	112	10.24
32	fenhexamid	1	81	7.93	1	75	4.79
33	fenthion	1	80	2.41	2	78	6.87
34	flufenacet	1	82	5.73	2	78	7.67
35	flufenoxuron	1	98	4.44	1	76	6.84
36	flumioxazin	1	75	5.65	2	84	8.57
37	flusilazole	1	80	4.95	10	78	7.49
38	flutriafol	5	78	3.59	50	70	11.51
39	hexythiazox	10	80	4.50	100	84	6.78
40	chlorantraniliprole	200	75	4.56	500	83	5.74
41	chlorfenvinphos	1	74	4.34	5	80	6.99
42	imazalil	1	72	10.39	2	91	11.43
43	imazosulfuron	1	78	5.22	1	82	6.41
44	imidacloprid	1	81	2.47	10	84	3.17
45	indoxacarb	1	70	1.12	1	82	4.53

46	linuron	20	82	9.06	200	75	6.78
47	malathion	10	95	8.40	50	84	13.16
48	metalaxyl	1	85	7.63	5	90	5.89
49	metamitron	1	81	2.51	1	78	4.12
50	metazachlor	1	82	1.02	1	86	3.02
51	metconazole	20	87	8.54	100	84	4.16
52	methacriphos	5	78	4.56	50	75	4.56
53	methamidophos	2	79	3.19	5	82	7.01
54	methidathion	1	74	4.41	2	84	6.98
55	methiocarb	1	73	7.45	1	76	10.34
56	methomyl	1	70	7.59	1	88	10.41
57	methoxyfenozide	1	80	4.58	2	78	6.84
58	metobromuron	1	80	3.38	5	79	4.18
59	metsulfuron-methyl	1	81	1.05	2	81	6.20
60	mevinphos	1	78	2.71	2	80	3.54
61	monuron	1	83	3.30	5	85	2.14
62	myclobutanil	1	86	4.56	1	75	6.57
63	naled	1	93	7.06	10	79	9.68
64	napropamide	1	85	6.81	2	84	6.47
65	neburon	1	94	6.41	5	84	6.95
66	nicosulfuron	1	83	4.56	5	98	7.45
67	oxamyl	1	82	3.84	2	87	8.14
68	penconazole	1	82	2.14	1	86	3.58
69	pencycuron	1	81	5.45	20	80	6.41
70	pendimethalin	1	82	3.77	10	89	3.99
71	permethrin	1	82	2.24	2	84	3.62
72	phenmedipham	1	80	6.94	10	90	7.47
73	piperonyl butoxide	2	82	3.14	10	76	9.85
74	pirimicarb	1	84	2.24	5	82	10.14
75	pirimicarb-desmethyl	1	92	6.22	2	99	7.48

76	pirimiphos-ethyl	1	81	4.71	2	86	8.61
77	pirimiphos-methyl	1	76	3.96	10	88	8.47
78	profenofos	1	79	3.15	5	94	4.16
79	prochloraz	200	84	6.36	1000	78	8.61
80	prometon	50	92	2.09	100	85	5.61
81	prometryn	1	79	3.72	1	86	7.18
82	propachlor	1	76	6.19	1	70	8.17
83	propamocarb	1	87	8.27	1	86	7.41
84	propiconazole	1	72	2.79	5	80	8.24
85	pyraclostrobin	1	88	7.80	1	87	11.64
86	pyridaben	1	85	8.73	2	86	7.54
87	pyrimethanil	1	84	3.61	1	76	8.21
88	pyriproxyfen	1	71	7.52	2	82	9.36
89	quinalphos	2	81	8.26	20	80	6.87
90	quinoxyfen	1	79	4.51	2	83	5.61
91	quizalofop	1	83	4.15	2	84	6.84
92	rimsulfuron	1	87	6.13	2	93	11.49
93	rotenone	1	71	9.65	1	87	13.20
94	simazine	1	79	4.25	2	76	6.34
95	simetryn	1	82	2.71	1	89	4.02
96	spirodiclofen	50	85	6.98	200	76	8.09
97	spiromesifen	1	70	0.85	5	85	9.82
98	tebuconazole	1	93	5.59	1	84	6.21
99	tetraconazole	10	76	4.78	100	81	6.97
100	tetramethrin	1	80	8.87	2	76	10.24
101	fenoxaprop-ethyl	1	87	5.85	2	84	9.64
102	thiabendazole	1	92	3.05	5	74	6.54
103	thiacloprid	1	79	6.52	1	84	4.89
104	thiamethoxam	1	77	8.01	1	79	8.96
105	triadimefon	1	89	6.89	1	86	6.87

106	triazophos	1	88	1.32	1	89	4.57
107	tricyclazole	2	78	2.97	50	75	6.85
108	trifloxystrobin	1	81	4.75	1	86	8.29
109	zoxamide	1	87	8.15	1	92	10.48
110	carboxin	1	77	4.21	1	87	5.47
111	fenamidone	1	80	2.10	1	84	6.87
112	fluopicolide	1	80	5.09	2	94	6.79
113	pethoxamid	1	80	3.78	2	82	3.71
114	phosmet	1	77	5.35	5	90	2.97
115	spirotetramat	1	72	3.37	1	82	6.14

2. Determination of pesticide multi-residues in fruits and vegetables (LC/MS-MS)

2.1 Scope of application

This method provides a method for the detection of pesticide residues in fruits and vegetables by liquid chromatography tandem mass spectrometry.

This method is suitable for the determination of 115 pesticide residues in fruits and vegetables.

2.2 Principles

The residual pesticides in the samples were extracted by the acidic acetonitrile solution, then centrifuged and cleaned up. The liquid was detected by liquid chromatography tandem mass spectrometry (LC/MS-MS) and quantified by external standard method.

2.3 Reagents and materials

Only analytical pure reagents are used unless otherwise specified.

2.3.1 Acetonitrile: chromatographic purity.

2.3.2 Hydrochloric acid: analytical reagent (AR, 37%).

2.3.3 Sodium chloride: analytical reagent.

2.3.4 Anhydrous Magnesium Sulfate: analytical reagent.

2.3.5 Ultra pure water (MiliQ).

2.3.6 Formic acid: guaranteed reagent.

2.3.7 Ammonium formate: chromatographic purity.

2.3.8 Acetone: analytical reagent, heavy distillation.

2.3.9 4 mmol ammonium formate solution: 0.252 g ammonium formate was diluted to 1000 mL.

2.3.10 0.1% formic acid acetonitrile solution (volume fraction).

2.3.11 Pesticides and related chemicals standard material: purity of not less than 95%.

2.3.12 Standard solution for pesticides and related chemicals

2.3.12.1 Standard stock solution

5 mg-10 mg pesticide standard substances were respectively selected in 10 ml volumetric flask. According to the solubility of standard materials, solvents such as methanol, toluene, acetone, acetonitrile or isooctane were dissolved and fixed to scale. The standard reserve solution is preserved at 0-4 degrees C, and can be used for one year.

2.3.12.2 Standard mixed solution

The standard reserve liquid is mixed according to a certain concentration. It can be saved at 4 degrees centigrade for one month.

2.4 Instruments and equipment

2.4.1 Liquid chromatography tandem mass spectrometer: an electrospray ion source (ESI) (Thermo, TSQ Quantum Ultra).

2.4.2 Analysis of scales: a sense of 0.1 mg and 0.01 g (Mettler ToleDo, XP105DR).

2.4.3 Ultra-pure Water Purifier(Millipore, milli-Q advantage A10)..

2.4.4 Vortex mixer (Vortex, Kylin-Bell)..

2.4.5 Centrifuge (Beckman, avanti J-E)..

2.4.6 Sample Concentrator (Zymark, TurboVap LV)..

2.4.7 Level oscillator (Yamato, SA31)..

2.4.8 Ultrasonic generator (Shengda, SD-8200H)...

2.4.9 Thermostat box (Haier, DW-25L262)..

2.5 Samples preservation

Keep the samples in the refrigerator at 4℃.

2.6 Determination procedure

2.6.1 Extraction

2 g samples were accurately weighed in 50 mL centrifuge tube, then 0.2% aqueous solution of formic acid 20 mL was added. After ultrasonic extraction for 2 min, 20 ml acetonitrile was added and homogenized 30 s. Then 3 g of sodium chloride was added and oscillated for 10 min. The solution was placed statically for 20 min and then centrifuged at 8000 RPM for 5 min. Finally, 10 mL supernatant was accurately taken for purification.

2.6.2 Purification

10 mL supernatant was accurately taken into the vortex centrifugal tube with 2 g Magnesium Sulfate Anhydrous and vortexed for 30 s. After oscillatation for 2 min, the solution was centrifuged at 8000 r/min for 5 min. Finally the supernatant was filtered through 0.20 μm organic microporous membrane for the detection using high performance liquid chromatography tandem mass spectrometry.

2.6.3 Analytical conditions for LC/MS-MS

2.6.3.1 LC condition

a) Column: Atlantis T3 column (100 mm x 4.6 mm, 5 μm) or equivalent;

b) The mobile phase and the gradient elution conditions are shown in Table 8.

Table 8 The mobile phase and the gradient elution conditions

Time/min	Flow rate /μL/min	A（/%） (4 mmol/L Ammonium formate -0.1% formic acid water solution)	B（/%） (0.1% formic acid acetonitrile solution)
0	300	2	98
3.00	300	2	98
5.00	300	20	80
25.00	300	98	2
28.00	300	98	2
29.00	300	2	98
35.00	300	2	98

c) Column temperature: 35 °C;

d) The sampling volume: 10 μL;

e) Ion source: ESI;

f) Scanning mode: positive ion scanning;

g) Detection methods: selective reaction monitoring (SRM);

h) Pressure of the sheath gas: 275 Kpa;

i) Flow rate of auxiliary air: 3 L/min;

j) Capillary voltage: positive ion mode 3000 V;

k) Capillary temperature: 350 °C;

l) Neubilizer temperature (auxiliary gas): 280 °C;

m) Collision energy of the monitoring ion pair see Appendix 1.

2.6.3.2 Qualitative determination

In this method, the liquid chromatography tandem mass spectrometry was quantified by the external standard calibration curve method. In order to reduce the effect of matrix on quantitative determination, matrix-matched calibration curve was used to quantify the concentration in the samples. And the response value of pesticide in the tested samples was guaranteed to be within the linear range of the instrument.

2.6.4 Parallel test

The same test is carried out in parallel to the same sample according to the above steps.

2.6.5 Blank test

Except for no sample, all of these steps are carried out according to the above steps.

2.7. Result calculation

The content of the pesticides component in the sample was measured using liquid chromatography tandem mass spectrometry method and quantified by the calibration curve of external standard. The quantitative results of the standard curve method were calculated according to the following formula (1).

$$X=c\times(V/m)\times(1000/1000) \quad (1)$$

In the formula:

X — The content of the measured component in the sample, mg/kg;

C — The concentration of each substance in the standard working fluid, ug/mL;

V — The final volume of the liquid, mL;

m —The sample weight represented by the final sample solution, g.

The results of the calculation should be deducted from the blank value.

2.8 Determination of LODs values

The LODs (limit of detection) values were 0.005-0.05 mg/kg using this method.

2.9 Recovery rate

The recovery rate of this method is 68.8-115.8%.

Table 9 Monitoring ion pairs and collision energy of the compounds

No.	Compounds	Parent ion	Daughter ion	CE（ev）
1	Diniconazole	326.2	70.2	35
2	Diniconazole	326.2	148.2	27
3	Diniconazole	326.2	159.2	26
4	Azinphos-methyl	317.9	125.0	19
5	Azinphos-methyl	317.9	261.0	8
6	Fenthion	278.9	247.0	13
7	Fenthion	278.9	169.1	20
8	Tribenuron methyl	396.1	155.0	17
9	Tribenuron methyl	396.1	181.0	28
10	Tribenuron methyl	396.1	364.0	15
11	Difenoconazole	406.2	111.0	55
12	Difenoconazole	406.2	251.0	25
13	Difenoconazole	406.2	187.5	43
14	Benalaxyl	326.2	148.0	22
15	Benalaxyl	326.2	208.0	15
16	Zoxamide	336.2	159.0	38
17	Zoxamide	336.2	187.0	23
18	Fenamiphos	304.0	217.0	24
19	Fenamiphos	304.0	234.0	8
20	Fenamiphos	304.0	217.0	22
21	Fenamiphos	304.0	202.0	35
22	Pyriproxyfen	322.2	96.0	16
23	Pyriproxyfen	322.2	185.3	27
24	Imidacloprid	256.1	175.1	20
25	Imidacloprid	256.1	209.1	18
26	Pymetrozine	218.0	79.0	30
27	Pymetrozine	218.0	105.0	25
28	pyraclostrobin	388.2	163.0	26
29	pyraclostrobin	388.2	194.0	14
30	Propiconazole	342.2	69.2	21
31	Propiconazole	342.2	159.0	29
32	Propiconazole	342.2	273.0	25

33	Flumioxazin	355.1	267.0	26
34	Flumioxazin	355.1	73.0	35
35	Profenophos	372.9	128.0	40
36	Profenophos	372.9	143.9	36
37	Profenophos	372.9	302.8	19
38	Tebufenocide	353.1	133.0	19
39	Tebufenocide	353.1	297.0	10
40	Diflubenzuron	311.0	141.0	32
41	Diflubenzuron	311.0	158.0	14
42	Pyridaben	365.2	147.0	23
43	Pyridaben	365.2	309.1	13
44	Napropamide	272.1	114.2	22
45	Napropamide	272.1	129.2	16
46	Napropamide	272.1	171.1	20
47	Diuron	233.1	46.3	35
48	Diuron	233.1	72.0	20
49	Butylate	218.2	57.1	19
50	Butylate	218.2	156.1	16
51	Butylate	218.2	100.1	17
52	Carbosulfan	381.2	160.1	18
53	Carbosulfan	381.2	118.0	25
54	Carbosulfan	381.2	75.9	36
55	Pyridalyl	490.0	109.0	29
56	Pyridalyl	490.0	163.9	38
57	Acetamiprid	223.1	90.2	36
58	Acetamiprid	223.1	126.1	22
59	Carbendazim	192.1	132.1	33
60	Carbendazim	192.1	160.1	20
61	Dimethenamid-P	276.0	111.2	33
62	Dimethenamid-P	276.0	168.0	23
63	Dimethenamid-P	276.0	244.0	14
64	Pendimethalin	282.1	119.1	25
65	Pendimethalin	282.1	194.1	18
66	Pendimethalin	282.1	212.0	11
67	Diazinon	305.0	153.1	23
68	Diazinon	305.0	169.1	25
69	Flutriafol	302.0	70.0	18
70	Flutriafol	302.0	123.0	29
71	rimsulfuron	432.1	182.0	30
72	rimsulfuron	432.1	325.1	21
73	Flubendiamide	700.0	256.0	58
74	Flubendiamide	700.0	274.0	34
75	Flubendiamide	700.0	408.0	13

76	Fluopicolide	383.0	145.0	47
77	Fluopicolide	383.0	172.9	23
78	Fluopicolide	383.0	365.0	17
79	Norflurazon	304.1	88.0	39
80	Norflurazon	304.1	284.0	25
81	Flufenpyr-ethyl	409.1	335.0	28
82	Flufenpyr-ethyl	409.1	381.0	20
83	Flufenpyr-ethyl	409.1	307.0	39
84	Flusilazole	316.2	165.0	34
85	Flusilazole	316.2	247.1	19
86	Triflumizole	346.2	73.0	18
87	Triflumizole	346.2	278.1	12
88	Flufenacet	364.2	152.0	20
89	Flufenacet	364.2	194.0	12
90	Fluthiacet-methyl	404.1	344.0	30
91	Fluthiacet-methyl	404.1	274.0	36
92	Fluthiacet-methyl	404.1	85.0	30
93	Cyproconazole	291.8	69.4	30
94	Cyproconazole	291.8	124.4	28
95	Fenhexamid	302.1	55.0	36
96	Fenhexamid	302.1	97.0	26
97	Methamidophos	142.0	94.0	20
98	Methamidophos	142.0	125.0	10
99	Pirimiphos-methyl	306.0	108.2	34
100	Pirimiphos-methyl	306.0	164.1	24
101	Isofenfos-methyl	346.0	216.9	23
102	Methiocarb	226.1	169.0	10
103	Carbaryl	202.0	145.0	13
104	Carbaryl	202.0	127.0	33
105	Metalaxyl	280.1	192.1	16
106	Metalaxyl	280.1	220.1	16
107	Methoxyfenozide	369.0	149.0	14
108	Methoxyfenozide	369.0	313.0	10
109	Fenbuconazole	337.2	125.1	35
110	Myclobutanil	289.1	70.2	19
111	Myclobutanil	289.1	125.0	31
112	Pirimicarb	239.1	72.0	21
113	Pirimicarb	239.1	182.0	16
114	Benfuracarb	222.1	165.1	25
115	Benfuracarb	222.1	123.1	14
116	Quinoxyfen	307.9	161.9	47
117	Quinoxyfen	307.9	196.8	33

118	Dimethoate	230.1	125.1	23
119	Dimethoate	230.1	199.1	12
120	Linuron	249.1	160.0	17
121	Linuron	249.1	182.0	18
122	Linuron	250.0	161.0	25
123	Linuron	250.0	183.0	22
124	Linuron	250.0	133.9	46
125	Bitertanol	338.1	99.0	16
126	Bitertanol	338.1	269.0	10
127	Fenarimol	331.1	81.0	34
128	Fenarimol	331.1	268.0	23
129	Chlorantraniliprole	483.6	452.9	17
130	Chlorantraniliprole	483.6	285.8	14
131	Spirotetramat	374.2	302.2	19
132	Spirotetramat	374.2	330.2	17
133	Spiromesifen	371.3	255.3	25
134	Spiromesifen	371.3	273.3	15
135	Spirodiclofen	411.0	213.1	25
136	Malathion	331.0	99.0	25
137	Malathion	331.0	125.0	32
138	Malathion	331.0	127.0	13
139	Prochloraz	376.2	266.0	18
140	Prochloraz	376.2	308.0	14
141	Fenamidone	312.2	236.2	16
142	Fenamidone	312.2	264.2	12
143	Azoxystrobin	404.1	329.1	32
144	Azoxystrobin	404.1	372.1	14
145	Pyrimethanil	200.1	82.0	30
146	Pyrimethanil	200.1	107.0	26
147	Methomyl	163.1	88.1	10
148	Methomyl	163.1	106.1	10
149	Ethoprophos	243.1	97.1	30
150	Ethoprophos	243.1	131.1	40
151	Cyromazine	167.1	68.2	28
152	Cyromazine	167.1	81.2	26
153	Cyromazine	167.1	85.2	19
154	Prometryn	242.2	157.9	24
155	Prometryn	242.2	199.9	20
156	Triforine-a	434.9	390.0	12
157	Clothianidin	250.1	132.1	18
158	Clothianidin	250.1	169.1	14
159	Thiacloprid	253.1	90.2	37
160	Thiacloprid	253.1	126.1	22

161	Thiamethoxam	292.2	132.1	24
162	Thiamethoxam	292.2	211.1	14
163	Thiamethoxam	292.2	181.0	29
164	Thiamethoxam	291.8	210.6	11
165	Thiamethoxam	291.8	131.4	24
166	thiabendazole	202.0	131.1	35
167	Thiabendazole	202.0	175.1	28
168	Buprofezin	306.2	116.0	18
169	Buprofezin	306.2	201.0	12
170	triadimenol	296.1	70.0	20
171	Triazophos	314.0	119.2	36
172	Triazophos	314.0	162.1	21
173	Triadimefon	294.2	197.1	16
174	Triadimefon	294.2	225.1	16
175	Fenitrothion	277.9	125.1	21
176	Fenitrothion	277.9	246.0	17
177	Methidathion	319.2	88.0	36
178	Methidathion	319.2	102.0	35
179	Oxamyl	237.1	72.1	15
180	Oxamyl	237.1	90.1	10
181	Amitraz	293.7	162.6	15
182	Mandipropamid	411.9	328.0	13
183	Mandipropamid	411.9	356.0	7
184	Propamocarb	189.1	102.1	19
185	Propamocarb	189.1	144.1	14
186	tetraconazole	372.2	70.0	24
187	tetraconazole	372.2	159.0	39
188	Mevinphos	225.1	127.1	15
189	Mevinphos	225.1	192.8	8
190	Desmedipham	318.2	136.0	28
191	Desmedipham	318.2	182.0	15
192	Carboxin	236.0	87.0	24
193	Carboxin	236.0	143.0	17
194	Trifloxystrobin	409.3	186.0	21
195	Trifloxystrobin	409.3	206.1	16
196	Penconazole	284.1	70.1	17
197	Penconazole	284.1	159.0	35
198	Tebuconazole	308.2	70.2	21
199	Tebuconazole	308.2	125.0	34
200	simazine	202.1	104.0	27
201	simazine	202.1	132.0	20
202	Dimethomorph	388.1	165.0	34
203	Dimethomorph	388.1	301.0	22

204	Dimethomorph	387.7	300.7	20
205	Dimethomorph	387.7	164.5	31
206	Uniconazole	292.1	70.2	25
207	Uniconazole	292.1	125.0	32
208	Phosmet	317.9	256.2	25
209	Phosmet	317.9	88.0	35
210	Phosmet	317.9	300.3	28
211	Nicosulfuron	411.2	182.0	26
212	Nicosulfuron	411.2	213.0	22
213	Metconazole	320.2	70.1	22
214	Metconazole	320.2	124.9	41
215	Ethalfluralin	334.2	165.2	20
216	Etoxazole	360.2	113.0	52
217	Etoxazole	360.2	141.0	25
218	Etoxazole	360.2	177.1	22
219	Acephate	184.1	95.2	25
220	Acephate	184.1	143.1	10
221	Imazalil	297.2	159.0	24
222	Imazalil	297.2	201.0	18
223	Indoxacarb	528.3	203.0	40
224	Indoxacarb	528.3	293.0	15
225	Atrazine	216.0	174.0	16
226	Piperonyl butoxide	355.7	176.5	12
227	Piperonyl butoxide	355.7	118.5	32
228	Ipconazole	334.1	70.2	22
229	Ipconazole	334.1	125.0	42
230	Carfentrazone ethyl	412.2	384.0	15
231	(E)-Fenpyroximate	422.2	366.0	34
232	(E)-Fenpyroximate	422.2	214.0	15
233	Flumetsulam	326.0	109.0	53
234	Flumetsulam	326.0	109.0	53
235	Kasugamycin	380.3	70.0	31
236	Spinosad	732.2	142.0	28
237	Spinosad	732.2	98.0	35
238	Novaluron	493.3	141.0	42
239	Clofentezine	303.1	102.0	36
240	Clofentezine	303.1	138.0	18
241	phenmedipham	301.2	136.0	22
242	phenmedipham	301.2	168.0	10
243	Spinetoram	748.3	142.0	30
244	Spinetoram	748.3	98.0	31
245	Fluometuron	233.1	72.1	18
246	Fluometuron	233.1	46.3	17

Table 10、Linear range and the correlation coefficient (R^2)

No.	Compounds	R^2	Linear range
1	Diniconazole	0.9985	0.01~0.2
2	Azinphos-methyl	0.9974	0.01~0.2
3	Fenthion	0.9986	0.01~0.2
4	Tribenuron methyl	0.9971	0.01~0.2
5	Difenoconazole	0.9992	0.01~0.2
6	Benalaxyl	0.9983	0.01~0.2
7	Zoxamide	0.9984	0.01~0.2
8	Fenamiphos	0.9975	0.02~0.2
9	Pyriproxyfen	0.9952	0.01~0.2
10	Imidacloprid	0.9979	0.01~0.2
11	Pymetrozine	0.9974	0.01~0.2
12	pyraclostrobin	0.9994	0.01~0.2
13	Propiconazole	0.9937	0.01~0.2
14	Flumioxazin	0.9876	0.01~0.2
15	Profenophos	0.9837	0.01~0.2
16	Tebufenocide	0.9929	0.005~0.01
17	Diflubenzuron	0.9991	0.005~0.01
18	Pyridaben	0.9972	0.01~0.2
19	Napropamide	0.9992	0.01~0.2
20	Diuron	0.9993	0.01~0.2
21	Butylate	0.9956	0.01~0.2
22	Carbosulfan	0.9996	0.005~0.01
23	Pyridalyl	0.9998	0.01~0.2
24	Acetamiprid	0.9932	0.01~0.1
25	Carbendazim	0.9967	0.02~0.2
26	Dimethenamid-P	0.9965	0.01~0.2
27	Pendimethalin	0.9981	0.01~0.2
28	Diazinon	0.9924	0.02~0.2
29	Flutriafol	0.9951	0.01~0.2
30	rimsulfuron	0.9995	0.01~0.2
31	Flubendiamide	0.9992	0.01~0.2
32	Fluopicolide	0.9972	0.01~0.2
33	Norflurazon	0.9972	0.05~0.2
34	Flufenpyr-ethyl	0.9986	0.01~0.2
35	Flusilazole	0.9986	0.01~0.2
36	Triflumizole	0.9982	0.01~0.2
37	Flufenacet	0.9998	0.005~0.2
38	Fluthiacet-methyl	0.9987	0.01~0.2
39	Cyproconazole	0.999	0.01~0.2

40	Fenhexamid	0.9988	0.01~0.2
41	Methamidophos	0.9933	0.01~0.2
42	Pirimiphos-methyl	0.9947	0.01~0.2
43	Isofenfos-methyl	0.9923	0.01~0.2
44	Methiocarb	0.9978	0.01~0.2
45	Carbaryl	0.9993	0.01~0.2
46	Metalaxyl	0.9994	0.01~0.2
47	Methoxyfenozide	0.9982	0.01~0.2
48	Fenbuconazole	0.995	0.01~0.2
49	Myclobutanil	0.9913	0.01~0.1
50	Pirimicarb	0.9994	0.005~0.2
51	Benfuracarb	0.999	0.01~0.2
52	Quinoxyfen	0.9992	0.005~0.2
53	Dimethoate	0.9962	0.01~0.2
54	Linuron	0.9995	0.01~0.2
55	Bitertanol	0.9983	0.01~0.2
56	Fenarimol	0.9999	0.01~0.2
57	Chlorantraniliprole	0.9958	0.01~0.2
58	Spirotetramat	0.9975	0.01~0.2
59	Spiromesifen	0.9997	0.01~0.2
60	Spirodiclofen	0.9988	0.005~0.2
61	Malathion	0.9984	0.02~0.2
62	Prochloraz	0.9935	0.01~0.2
63	Fenamidone	0.9957	0.01~0.2
64	Azoxystrobin	0.9952	0.01~0.2
65	Pyrimethanil	0.9918	0.01~0.2
66	Methomyl	0.9998	0.005~0.2
67	Ethoprophos	0.9993	0.005~0.2
68	Cyromazine	0.9964	0.01~0.2
69	Prometryn	0.9964	0.01~0.2
70	Triforine-a	0.9974	0.01~0.2
71	Clothianidin	0.9993	0.005~0.2
72	Thiacloprid	0.9936	0.05~0.2
73	Thiamethoxam	0.9907	0.02~0.2
74	thiabendazole	0.9984	0.01~0.2
75	Buprofezin	0.9979	0.01~0.2
76	Triadimenol	0.9998	0.005~0.2
77	Triazophos	0.9981	0.01~0.2
78	Triadimefon	0.9995	0.005~0.2
79	Fenitrothion	0.9969	0.01~0.2
80	Methidathion	0.9995	0.01~0.2
81	Oxamyl	0.9954	0.01~0.1
82	Amitraz	0.9974	0.01~0.2

83	Mandipropamid	0.9968	0.01~0.2
84	Propamocarb	0.9984	0.01~0.2
85	tetraconazole	0.9957	0.01~0.2
86	Mevinphos	0.9995	0.01~0.2
87	Desmedipham	0.9979	0.01~0.2
88	Carboxin	0.9958	0.01~0.2
89	Trifloxystrobin	0.9977	0.01~0.2
90	Penconazole	0.9993	0.01~0.2
91	Tebuconazole	0.9935	0.01~0.2
92	simazine	0.998	0.01~0.2
93	Dimethomorph	0.9957	0.01~0.2
94	Uniconazole	0.9982	0.01~0.2
95	Phosmet	0.998	0.05~0.2
96	Nicosulfuron	0.9991	0.01~0.2
97	Metconazole	0.9892	0.01~0.2
98	Ethalfluralin	0.993	0.01~0.2
99	Etoxazole	0.992	0.01~0.2
100	Acephate	0.9929	0.01~0.2
101	Imazalil	0.9986	0.05~0.2
102	Indoxacarb	0.9901	0.01~0.2
103	Atrazine	0.9963	0.01~0.1
104	Piperonyl butoxide	0.9975	0.01~0.2
105	Ipconazole	0.9996	0.01~0.2
106	Carfentrazone ethyl	0.9941	0.01~0.2
107	(E)-Fenpyroximate	0.9932	0.01~0.2
108	Flumetsulam	0.9925	0.01~0.2
109	Kasugamycin	0.9967	0.01~0.2
110	Spinosad	0.9998	0.01~0.2
111	Novaluron	0.9954	0.01~0.2
112	Clofentezine	0.993	0.01~0.2
113	phenmedipham	0.992	0.01~0.2
114	Spinetoram	0.9929	0.01~0.2
115	Fluometuron	0.9986	0.05~0.2

Table11、LOD values, RSD and recoveries of the compounds

No.	Compounds	LOD （mg/kg）	Recovery（%）	RSD（%）	Recovery （%）	RSD（%）
			20.0μgkg		50.0μgkg	
1	Diniconazole	0.01	102.5	13.2	77.2	6.1
2	Azinphos-methyl	0.01	104.1	12.4	92.6	12.6
3	Fenthion	0.01	74.1	16.8	77.8	17.4
4	Tribenuron methyl	0.01	104	18.3	90	9.7
5	Difenoconazole	0.01	75.8	12.2	87.6	11.2
6	Benalaxyl	0.01	70.3	14.5	81.4	9.5
7	Zoxamide	0.01	82.4	12.5	92.6	9.8
8	Fenamiphos	0.01	71.4	6.5	82.7	16.4
9	Pyriproxyfen	0.01	104.6	13.2	76.9	19.4
10	Imidacloprid	0.01	71.6	7.9	97.8	9.5
11	Pymetrozine	0.01	92.9	8.4	89.6	17.9
12	pyraclostrobin	0.01	71	9.3	96.5	7.9
13	Propiconazole	0.01	91.8	11.1	98.4	8.4
14	Flumioxazin	0.01	96.2	16	92.6	12.7
15	Profenophos	0.01	72.8	8.1	82.7	13.5
16	Tebufenocide	0.005	75.6	9.7	76.9	16.2
17	Diflubenzuron	0.005	105.7	11.3	97.8	14.3
18	Pyridaben	0.01	100.2	15.6	89.6	12.1
19	Napropamide	0.01	92.6	17.4	96.5	10.9
20	Diuron	0.01	107.8	19.5	98.4	13.8
21	Butylate	0.01	90	14.7	94.5	9.4
22	Carbosulfan	0.005	87.6	9.4	85.9	12.1
23	Pyridalyl	0.01	81.4	11.2	75.4	17.5
24	Acetamiprid	0.01	93.6	16.4	86.6	17.4
25	Carbendazim	0.01	102.8	14.2	96.7	9.5
26	Dimethenamid-P	0.01	106	6.9	108.4	8.7
27	Pendimethalin	0.01	99.8	9.3	98.1	9.4
28	Diazinon	0.02	69.4	10.3	71.1	8.1
29	Flutriafol	0.01	69.4	15.6	102.5	12.1
30	rimsulfuron	0.01	93.4	6.8	104.1	17.5
31	Flubendiamide	0.01	91.9	9.1	94.1	14.5
32	Fluopicolide	0.01	101.8	10.9	104	9.5
33	Norflurazon	0.05	101.2	11.4	115.8	15.3
34	Flufenpyr-ethyl	0.01	101.8	7.5	90.4	13.8
35	Flusilazole	0.01	90.8	9.5	82.4	9.4
36	Triflumizole	0.01	103.8	12.4	71.4	11.4
37	Flufenacet	0.005	96.9	9.3	104.6	7.5
38	Fluthiacet-methyl	0.01	70.9	6.1	71.6	9.5

39	Cyproconazole	0.01	114.2	12.6	92.9	12.4
40	Fenhexamid	0.01	96.8	17.4	71	9.3
41	Methamidophos	0.01	87.2	9.7	88.9	6.1
42	Pirimiphos-methyl	0.01	107.4	11.2	97.4	12.6
43	Isofenfos-methyl	0.01	99.8	9.5	94.8	17.4
44	Methiocarb	0.01	92.4	9.8	90.1	9.7
45	Carbaryl	0.01	81.2	16.4	87.2	11.2
46	Metalaxyl	0.01	99	19.4	92	8.4
47	Methoxyfenozide	0.01	87.2	9.5	82.7	9.3
48	Fenbuconazole	0.01	87.6	17.9	83.5	11.1
49	Myclobutanil	0.01	107.2	7.9	97.2	11.3
50	Pirimicarb	0.005	95	8.4	89.5	15.6
51	Benfuracarb	0.01	95.6	12.7	91	17.4
52	Quinoxyfen	0.005	98	13.5	96.4	8.4
53	Dimethoate	0.01	80.8	16.2	88.7	12.7
54	Linuron	0.01	88.4	14.3	84.6	13.5
55	Bitertanol	0.01	83.2	12.1	84.9	16.2
56	Fenarimol	0.01	72	9.5	74.1	14.3
57	Chlorantraniliprole	0.01	86	17.9	81.3	12.1
58	Spirotetramat	0.01	94.8	7.9	89.6	10.9
59	Spiromesifen	0.01	83.6	8.4	87.4	12.1
60	Spirodiclofen	0.005	88.2	12.7	82.7	17.9
61	Malathion	0.02	102.3	13.5	96.1	7.9
62	Prochloraz	0.01	84	16.2	79.9	8.4
63	Fenamidone	0.01	101	14.3	94.5	12.7
64	Azoxystrobin	0.01	111.8	12.1	105.6	13.5
65	Pyrimethanil	0.01	74.4	10.9	78.4	16.2
66	Methomyl	0.005	105.2	6.8	95.2	12.4
67	Ethoprophos	0.005	104.4	14.5	98.4	14.6
68	Cyromazine	0.01	83	4.5	87	15.3
69	Prometryn	0.01	107.4	15.3	101.4	7.9
70	Triforine-a	0.01	88.8	13.8	81.8	11
71	Clothianidin	0.005	92.8	9.4	92.8	17.5
72	Thiacloprid	0.05	78.6	12.1	87.2	16.2
73	Thiamethoxam	0.02	78.3	17.5	87.4	12.1
74	thiabendazole	0.01	84.2	14.5	89.8	9.5
75	Buprofezin	0.01	99.8	16	92.4	17.9
76	Tiadimenol	0.005	100.6	8.1	91.2	11.3
77	Triazophos	0.01	98.7	9.7	99	15.6
78	Triadimefon	0.005	102.1	11.3	97.2	7.9
79	Fenitrothion	0.01	98	15.6	87.6	12.7
80	Methidathion	0.01	95.1	17.4	87.2	13.5
81	Oxamyl	0.01	86.9	9.5	83.5	16.2

82	Amitraz	0.01	84.4	8.7	88.4	17.9
83	Mandipropamid	0.01	86	9.4	79.8	7.9
84	Propamocarb	0.01	73.8	8.1	68.9	15.6
85	Tetraconazole	0.01	111.4	12.1	104.4	7.9
86	Mevinphos	0.01	69.6	17.5	75.9	12.7
87	Desmedipham	0.01	80	14.5	81.2	13.5
88	Carboxin	0.01	87.6	13.6	84.1	16.2
89	Trifloxystrobin	0.01	101.2	17.8	104.5	17.9
90	Penconazole	0.01	79.8	7.4	79.8	7.9
91	Tebuconazole	0.01	70.6	4.5	76.4	8.4
92	simazine	0.01	109.4	18.3	104.7	12.7
93	Dimethomorph	0.01	104.6	4.5	98.3	13.5
94	Uniconazole	0.01	95.4	14.5	88.9	16.2
95	Phosmet	0.05	92.1	4.9	89.6	19.7
96	Nicosulfuron	0.01	88	7.9	80	11.3
97	Metconazole	0.01	95.8	8.4	87.6	15.6
98	Ethalfluralin	0.01	107.4	8.3	101.2	7.9
99	Etoxazole	0.01	93.8	11.1	87.6	11.3
100	Acephate	0.01	114.8	16	107.2	15.6
101	Imazalil	0.05	68.8	8.1	72.9	7.9
102	Indoxacarb	0.01	82.2	9.7	95.6	8.4
103	Atrazine	0.01	103.2	11.3	98	12.7
104	Piperonyl butoxide	0.01	100.8	15.6	93.8	11.3
105	Ipconazole	0.01	102.4	9.4	98.4	15.6
106	Carfentrazone ethyl	0.01	69.7	14.3	73.2	7.9
107	(E)-Fenpyroximate	0.01	73.2	12.1	72	7.9
108	Flumetsulam	0.01	72.1	10.9	76	8.4
109	Kasugamycin	0.01	71.6	12.1	74.2	12.7
110	Spinosad	0.01	74.5	17.9	78.7	13.5
111	Novaluron	0.01	73.8	7.9	102.1	16.2
112	Clofentezine	0.01	95.8	8.4	87.6	15.6
113	phenmedipham	0.01	107.4	8.3	101.2	7.9
114	Spinetoram	0.01	93.8	11.1	87.6	11.3
115	Fluometuron	0.05	114.8	16	107.2	15.6