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

Grant Agreement number:

727864 — EU-China-Safe

Acknowledgements

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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

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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

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 evaluated by the Interaboratory Comparison Study on Pesticide Residues in Food (ILC). Test material, prepared from the batch used for another proficiency test (PT), was provided by Fapas (Fera Science Ltd, York, UK).

Evaluation of the results and problems encountered by Chinese laboratories was carried out supporting options for harmonization and wider implementation of the methodology.

Summary of the facts about the Interaboratory Comparison Study on Pesticide Residues in Food:

1. Six laboratories from China and one laboratory from EU agreed to participate in this ILC study.

2. Test materials for the Inter-laboratory Comparison Study (ILC) on "Pesticide Residues in green tea" were dispatched in July 2021. Each participant received one green tea sample to be analysed for pesticide residues using LC-MS multidetection method developed and provided by the ILC Organizers.

3. An assigned value (X_a) was set for each analyte. A fit-for-purpose relative target standard deviation (σ_{FFP}) of 25 % was chosen to calculate the target standard deviations (σ) as well as the z-scores for the individual pesticides.

4. Results for this ILC study are summarised as follows:

analyte	assigned value (X_a) [mg/kg]	number of scores $\|z\| \leq 2.0$	total number of analytes	% $\|z\| \leq 2.0$	number of False Negative
Chlorpyrifos (ethyl)	0.119	5	7	71 %	1
Dimethoate	0.068	7	7	100 %	-
Dinotefuran	0.056	7	7	100 %	-
Fenpropimorph	0.079	6	7	86 %	1
Imidacloprid	0.047	6	7	86 %	1
Malathion	0.107	6	7	86 %	-
Pirimiphos-methyl	0.138	6	7	86 %	-
Pyridaben	0.081	6	7	86 %	-
Tolfenpyrad	0.077	6	7	86 %	-

Total number of False Positive results: 3

Total number of False Negative results: 3

** total number of analytes – total number of laboratories reporting result for certain target analyte*

2. INTRODUCTION

The aim of this ILC was to obtain information regarding the quality, accuracy and comparability of pesticide residue data in food reported within the framework of the EU and China laboratories when implementing multidetection LC-MS method for pesticide residues analysis in food matrix developed within EU-China-Safe project, www.euchinasafe.eu (see D4.5: Report on the establishment and application of the validated multi-analyte / multi matrix screening method for pesticide residues in fruits and vegetables (and related products) set-up in Chinese and EU laboratories).

For this ILC, requirements of the documents below have been taken into account:

- ISO/IEC 17043:2010 "Conformity assessment – General requirements for proficiency testing" [1]
- EA-4/21 INF:2018 Guidelines for the assessment of the appropriateness of small interlaboratory comparisons [2]
- General protocol for EU Proficiency Tests on Pesticide Residues in Food and Feed provided by European Reference Laboratories, 9th Ed., Nov 2019 [3]
- ISO 13528: Statistical methods for use in proficiency testing by interlaboratory comparisons [4]
- Protocol for Proficiency testing Schemes (Part 1: Common Principles), version 7, Jan 2021, FERA Science Ltd, Sand Hutton, York, UK [5]

Participating laboratories were provided with an assessment of their analytical performance that they can use to demonstrate their analytical performance and compare themselves with other participating laboratories [3].

The evaluation and scoring of the results of the participating laboratories was performed on an individual z-score basis and false positive (FP) or false negative (FN) rate. Classification or decisions on eligibility of laboratories for pesticide residues analysis has not been done by the Organiser and was beyond the scope of this ILC.

3. ACTIVITIES AND RESULTS

3.1 DESCRIPTION OF ACTIVITIES

3.1.1. Organizer of the ILC

The laboratory responsible for organization and assessment of this ILC is Metrological and Testing Laboratory UCT Prague (Department of Food Analysis and Nutrition), accredited by the Czech Accreditation Institute (CAI) according to the EN ISO/IEC 17025:2018.

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3.1.2. Identification of participating laboratories in the ILC

Ideally, the ILC would follow requirements of the ISO 13528 [4] when at least seven laboratories have registered. A lower number cannot enable proper statistical evaluation of the data (i.e. calculation of z-scores) and may result in postponement of the ILC. That is why the invitation of candidate laboratories could be expanded to laboratories outside EU-China-Safe project or outside the EU.

Alternatively, small ILC organised among seven or less laboratories will also apply the appropriate ISO/IEC 17043 requirements (see EA-4/21 INF:2018) [2].

The list of laboratories expected to participate in the ICL was proposed by the EU-China-Safe project partner, Professor Wu Yongning, MD, PhD, Chief Technical Officer of China National Center for Food Safety Risk Assessment (CFSA). EU Laboratories participating in the EU-China project were selected and invited by prof. Jana Hajslova, PhD (UCT Prague).

The Invitation letter / announcement (see **Annex I**) together with the ILC calendar, the Pesticides target list (see **Annex II**), and the analytical method description (SOP) (see **Annex III**) were distributed directly to the invited laboratories by e-mail.

Detailed List of registered / participating laboratories is part of the final ILC report (see **Annex IX**).

3.1.3. Test material selected for ILC

The test material was produced from dry green tea leaves. This test material, prepared from the batch used for another proficiency test (PT), was provided by Fapas (Fera Science Ltd, York, UK).

For preparation and homogeneity testing collaboration with FERA Science Ltd (as a participant in the EU-China-Safe project) was agreed as a laboratory meeting the quality requirements of the ISO/IEC 17043:2010. For Fapas QC material data sheet see **Annex IV** and Fapas – Food Chemistry Proficiency Test Report 1903 on Pesticide Residues in Green Tea [6]**.**

Material was homogenized and analysed for incurred residues (none detected). Subsequently, selected analytes were spiked into the blank test material. To test homogeneity, randomly selected test material units were analysed in duplicate.

Stability of the test material was classified by FERA as sufficient for the ILC from previous experience, expert advice, and published literature. Based on this, the test material was supposed to be stable at least until 13th November 2021.

Individual units of the material, 50 g / unit, to be analysed by laboratories participating in this ILC, were labelled by UCT Prague with a unique code and stored at -18 °C until dispatch (see Fig. 1). Blank material was not be distributed to the participants.

The total number of units prepared should exceed the number required for the participating laboratories by 20%, with a minimum of 6 units.

As shipment time can differ between labs/countries the Organiser kept track of the shipment duration and then decided whether it is reasonable to conduct additional stability tests at conditions simulating shipment. The Organiser was also ready to decide to conduct additional stability tests at different storage conditions than those recommended to the participants e.g. at ambient temperature. These additional stability tests were conducted in the UCT Prague laboratory; no effect of storage conditions at ambient temperature (24 °C) for 90 days (July – September 2021) was observed.

The Organiser tried to ensure that all the packages would arrive to each participating laboratory. An information message was sent out by e-mail before shipment. Laboratories had to make their own arrangements for the receipt of the package. This was supported by custom declaration prepared by UCT Prague. Participating laboratories were informed that the Organiser cannot take the responsibility for a parcel if it is retained at customs.

Fig. 1: Test material unit labelled and ready for distribution

3.1.4. Target pesticides list

Participants were provided with the List of target pesticides (see **Annex II**) and were asked to analyse test sample for all 50 potentially present pesticides indicated in the Target pesticides list (see **Annex III**), if possible. For each of the pesticides the Minimum Required Reporting Level (MRRL) was specified and it was supposed that Limit of Quantification LOQ ≤ MRRL.

Participating laboratories were informed that it should not be assumed that only pesticides registered for use on tea are present in the test item.

3.1.5. Procedures implemented for organisation of the ILC

3.1.5.1. General

1. Tasks, responsibilities and requirements of the organiser

For the ILC an expert team was established by the Organizer with the following tasks and responsibilities:

Coordinator, experienced in organization of ILC / studies, responsible for:

- Proposing and coordination of the study
- Contact person for the participating laboratories

Project manager responsible for planning and reporting of the ILC in line with the EU-China-Safe project plan

Scientific expert with sufficient knowledge and experience of the parameter studied: responsible for scientific relevance and quality of the study

Technical staff, experienced with and responsible for:

- Logistics (sample shipment, collection of results and questionnaires)
- Data processing and technical support

Notes:

- *ILC Organiser is the University of Chemistry and Technology Prague, partner of the EU-China-Safe project consortium. Organisation outside the consortium cannot considered as an alternative option.*

2. Communication, confidentiality, collusion

Communication with the laboratories was realized by or under responsibility of the coordinator (Organizer). Participating laboratories remained anonymous in e-mails sent to multiple addresses.

For this ILC, the laboratories were given a unique code (lab code), only known to themselves and the Organisers. In any ILC-Report, the names of participating laboratories were not linked to their laboratory codes.

Communication between participating laboratories during the test on matters concerning the ILC exercise was not permitted from the start of the ILC exercise until the distribution of the preliminary report. If collusion or falsification of results by participating laboratory would be suspected, all its results must be questioned and would be excluded from processing / reporting.

Disclosure of expected or assigned concentrations was done after the deadline of participation laboratories results submission.

The official language used in this ILC study is English.

3.1.5.2. Work flow

The table below describes the steps and indicative timeline set for the conduct of the ILC. Time t=0 corresponds to shipment of the test samples.

Time (d)	Description
t = -40	Drafting plan / protocol
-35 < t < -10	Invitation and registration of candidate participants
t = - 7	**Closure of registration of participants**
- 25 <t < -5	Preparation of list of participants details
-15 < t < 0	Preparation of test sample sets for shipment, notification
- 35 < t <0	Preparation of documentation/shipment arrangements
t = 0	**Shipment of test samples to participants**
2 < t < 12	Collection sample receipt forms, Re-sending if necessary
45 < t < 55	Sending out reminder of deadline approaching
t = 55	**Deadline submission of results**
55 < t < 65	Registration and verification of results; If necessary: inquire additional information from laboratories
60 < t < 70	Analysis for stability verification of test samples
58 < t < 75	Statistical evaluation of results
	If necessary: inquire additional information from laboratories
75 < t < 85	Writing draft ILC report
	Communication of preliminary results to participants
	(reported and assigned values, false positives/false negatives)
85 < t < 99	Discussion/approval of draft report by/with experts

t = 110 **Distribution of final report to participants**

Archiving of ILC documentation/correspondence

1. ILC invitation and registration

An **invitation** to participate in the ILC with a registration form was sent to candidate laboratories by email. The invitation letter (see **Annex I**) included all relevant information on:

- Subject of the ILC (matrix/pesticides)
- Aim of the ILC
- Amount of control material of each sample to be provided
- Calendar with key-dates: registration deadline, sample shipment, deadline for submission analysis results, expected date for delivering preliminary and final report

  ILC calendar (tentative):

Announcement / Invitation of laboratories	02-06-2021
Registration deadline	30-06-2021
Distribution of test samples	07-07-2021
Deadline for submission of results	31-08-2021
Preliminary report (Table with results and Z-scores)	26-09-2021
Final report	27-10-2021

- Any special requirements for participation in the ILC if applicable (e.g. minimum required LOQ, in/exclusion of certain procedures)
- Statement that all laboratory specific information will be treated confidentially, and will never be disclosed to third parties (government, accreditation bodies) except the EU-China-Safe Organizer, without permission of the laboratory
- Statement that participating laboratories themselves are responsible for custom clearance and associated costs if applicable

The **registration form** was available on the Project web page http://www.euchinasafe.eu/form_euchina_2021.html and included:

- Name/address of the Institution / laboratory
- Name of contact person, telephone number and email address
- Address for delivery of the test samples
- Statement to be signed by the contact person to agree with the conditions mentioned in the invitation letter, and that the laboratory will analyse the ILC samples and submit results before the indicated deadline
- Organizer details (institute, coordinator, contact details) and information how to submit the registration form

Upon registration, the participant received the confirmation and the random (unique) lab code. The Organizer kept confidential record linking the participants to the lab codes and results.

Registration closed after the deadline indicated in the invitation letter. However, the Organizer considered to allow additional laboratories to participate until shipment of the samples, if a sufficient number of control materials is available.

2. Specific protocol - Instruction letter

Simultaneously with shipment of the samples, the participants received an instruction letter (see **Annex V**), a sample receipt form (see **Annex VI**) and a result submission (reporting) form (see **Annex VII**) by email.

The instruction letter included:

- Amount of test samples in the package
- The target analytes list
- Way of storage of the samples upon receipt, until analysis
- Any pre-treatment before analysis (e.g. equilibration to room temperature, re-homogenisation)
- Deadline for submission of results and instructions for submission
- Statement that content has to be checked upon receipt and sample receipt form has to be completed and returned
- Statement that the samples need to be analysed in the same way as is done for analysis of samples in the frame of the EU-China-Safe project
- Any specific reporting requirements (e.g. number of significant figures, correction/or not for recovery)

3. Sample receipt form

Upon receipt of the samples by the laboratory, the content had to be checked and this receipt form (template see **Annex VI**) had to be returned by the participant to the Organizer. The sample receipt form included the following items to be filled in by the participant:

- The code of the test sample received
- The condition of the sample upon arrival (e.g. frozen, thawed, any damage/leakage etc.)
- Date of receipt

The form had to be completed and signed by the participant and sent by email to the Organizer (details were provided on the form). The Organizer checked whether the codes of the test samples matches with their records. In case samples did not arrive in good condition, as a contingency an alternative set may be sent immediately.

4. Result submission form

A result submission form (in the MS Excel, see **Annex VII**) was provided by the Organizer and had to be used by the participants to submit the results and method information to the Organizer.

5. Shipment of samples

Samples were preferably shipped on Monday-Tuesday to ensure delivery the same week and avoid storage in a courier distribution centre over the weekend. For the date of shipment, also national holidays were taken into account to ensure the laboratory is able to receive the samples.

6. Sample analysis, reporting results

Laboratories got 4 weeks for sample analysis and reporting the results. Ten days before the deadline, a deadline reminder was sent to those laboratories that did not yet submit their results.

Participants shall normally use the analytical method of their choice. In this ILC, however, participants were instructed to use a specific documented method provided in the frame of the EU-China-Safe project (see **Annex III**). The Laboratory was allowed to modify the original test method (adapted and implemented for their routine use) but the measuring principle (LC-MS) shall be maintained. Any deviations from, additions to, or exclusions from the original test method shall be reported by participants.

Where an analytical method has not yet been established routinely this should be stated.

Participating laboratories were responsible for reporting quantitative results to the Organiser within the stipulated deadline. Any pesticide that was targeted by a participating laboratory should has been reported as "Analysed" and „Detected / Not detected", together with the information on the relevant „Reporting Limit".

Each laboratory was able to report only one result for each analyte detected in the Test Item. The concentrations of the pesticides detected should be expressed in 'mg/kg'.

Laboratories were required to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery rate (in percentage) must also be reported.

7. Evaluation of results

- **False positive results (FP):**

Any results reported lower than the MRRL were not considered as false positives, even though these results should not have been reported. [3]

- **False Negative results (FN)**

These are results for pesticides reported by the laboratories as 'Analysed' but without reporting numerical values although they were detected by the Organiser as well as the majority of the participants that had targeted these specific pesticides at or above the respective MRRLs. Results reported as '< RL' (RL= Reporting Limit of the laboratory) were considered as not detected and were judged as False negatives. [3]

- **Estimation of the assigned value (X_a)**

If a reference material (RM) is available in sufficient amounts for use in the ILC study, the certified values (and associated uncertainties) could be used directly. This approach is quick and simple to implement, and provides a value independent of the participant results.

Test material from the batch used in the recent Proficiency Testing (PT) round provided by Fapas (FERA Science Ltd., UK) was used in this ILC study. Fapas PT materials underwent both homogeneity and stability testing (including long-term stability test for its subsequent use). Datasheet associated with test material provided the same data of assigned values and uncertainties as in the Fapas PT (see **Annex IV**).

The assigned value for each analyte was derived from the consensus of the valid results submitted by 29 - 54 participants (suspect or invalid entries were excluded). Robust mean was chosen as the assigned value X_a [5].

The uncertainty of all assigned values u (X_a) was also calculated by Fapas [5] as:

$$(X_a) = \sigma / \sqrt{n}$$

where σ is the robust standard deviation and n is the number of results.

- **Standard deviation of the assigned value (target standard deviation)**

The target standard deviation of the assigned value (σ) was calculated using a Fit-For-Purpose approach with a fixed Relative Standard Deviation (RSD_{FFP}).

Based on experience, a percentage RSD_{FFP} of 25 % was currently used for all analyte-matrix combination, with the target standard deviation being calculated as follows:

$$\sigma_{FFP} = 0.25 * X_a$$

- **Z-scores**

This parameter was calculated for each analyte and laboratory using the following formula:

$$z = (x - X_a) / \sigma_{FFP}$$

where x is the value reported by the laboratory, X_a is the assigned value, and σ_{FFP} is the standard deviation using the FFP approach. Z-scores will be rounded to one decimal place.

Any z-scores > 5 will be typically reported as '> 5' and a value of '5' will be used to calculate combined z scores (see below).

Z-scores were interpreted in the following way, in line with ISO 17043:2010 [1]:

$|z| \leq 2.0$ Acceptable

$2.0 < |z| < 3.0$ Questionable

$|z| \geq 3.0$ Unacceptable

Nevertheless, the limits mentioned above must not be regarded as strict boundaries but should be treated as action limits. Z-scores are statistics based on the random selection from a hypothetical normal distribution and must be interpreted as such [5].

8. Communication of preliminary results to participants

Data reported by the laboratories were statistically evaluated and z-scores were calculated. A summary table was compiled and sent to the participants as a first feedback on their performance (see **Annex VII**).

9. ILC report

Reporting was done by the Organizer according to EU-China-Safe project requirements. The report will be submitted to the EU-China-Safe project coordinator and after approval made public for all who might be interested.

10. Remarks and complaints

Participants had the possibility to send their remarks or complaints to the Organizer of the ILC. If it had happened, these were examined and feedback was provided. If appropriate, corrective actions were taken.

11. Archiving

All documentation, i.e. ILC plan, invitations, instructions, reports, data files on test materials, email communications, etc. will be archived either electronically (with at least one backup) or as paper files for a period of 5 years.

3.2 DESCRIPTION OF RESULTS

3.2.1. Final report

Final report about Interlaboratory Comparison Study on Pesticide Residues in Food (see **Annex IX**) provides the overview about the participants of the ILC, its organisation and results.

3.2.2 Dissemination

Information about results of the ILC 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

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. Interlaboratory Comparison Study on Pesticide Residues in Food organised within EU-China-Safe activities was designed to check feasibility of proposed approach and help in overcoming of possible barriers in collaboration among EU and China regions. The results of the Interlaboratory Comparison Study on Pesticide Residues in Food were critically assessed, demonstrating potential for harmonisation of used analytical methodology between laboratories in the EU and China.

5. REFERENCES

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

[1] ISO/IEC 17043:2010 "Conformity assessment – General requirements for proficiency testing"

[2] EA-4/21 INF:2018 Guidelines for the assessment of the appropriateness of small interlaboratory comparisons

[3] General protocol for EU Proficiency Tests on Pesticide Residues in Food and Feed provided by European Reference Laboratories, 9th Ed., Nov 2019

[4] ISO 13528: Statistical methods for use in proficiency testing by interlaboratory comparisons

[5] Protocol for Proficiency testing Schemes (Part 1: Common Principles), version 7, Jan 2021, FERA Science Ltd, Sand Hutton, York, UK

[6] Fapas – Food Chemistry Proficiency Test Report 19303, Pesticide Residues in Green Tea, December 2020 – January 2021, FERA Science Ltd, Sand Hutton, York, UK

6. APPENDIX

Annex I: Invitation letter to participate in the Interlaboratory Comparison Study on Pesticide Residues in Food (ILC)

Annex II: Pesticides target list

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

Annex IV: Fapas QC material data sheet T19303QC

Annex V: Instructions for Interlaboratory Comparison Study on Pesticide Residues in Food

Annex VI: Sample receipt form

Annex VII: Reporting form

Annex VIII: Preliminary report - Interlaboratory Comparison Study on Pesticide Residues in Food

Annex IX: Final report - Interlaboratory Comparison Study on Pesticide Residues in Food

Annex I: Invitation letter to participate in the Interlaboratory Comparison Study on Pesticide Residues in Food (ILC)

UNIVERSITY OF CHEMISTRY AND TECHNOLOGY, PRAGUE
Faculty of Food and Biochemical Technology
Department of Food Analysis and Nutrition

Invitation to participate in the Inter-laboratory study (ILC)

Within the frame of EU-China-Safe project (www.euchinasafe.eu) the University of Chemistry and Technology Prague (Czech Republic) in collaboration with Queens University, Belfast and China National Center for Food Safety Risk Assessment (CFSA) announces the **Inter-laboratory Comparison Study (ICL) on pesticide residues in green tea.**

The aim of this ILC is to obtain information regarding the quality, accuracy and comparability of pesticide residues data in food reported within the framework of EU and China laboratories implementing multidetection LC-MS based method for pesticide residues analysis in food matrix developed within this project. This ILC can provide laboratories with an assessment of their analytical performance and reliability of their data in comparison with other laboratories and/or expert laboratories. This will aid in the quality improvement of analysis at each of the involved laboratories.

The list of laboratories expected to participate in the ICL has been proposed by EU-China-Safe project partner, Professor Wu Yongning, MD, PhD, Chief Technical Officer of China National Center for Food Safety Risk Assessment (CFSA).

Test sample

The test matrix will be dry leaves of green tea. Each participant will obtain 50 g sample of homogeneous test material.

Target analytes

The pesticide residues potentially present in the test samples are listed in the Pesticides target list: see *Appendix.* For each of the pesticides, the Minimum Required Reporting Level (MRRL) is specified.
Participants are asked to analyse test sample, if possible, for all 50 potentially present pesticide summarized in the Pesticides target list.

ILC Calendar (tentative):

Announcement / Invitation of laboratories	02-06-2021
Deadline registration	30-06-2021
Distribution of test samples	07-07-2021
Deadline submission of results	31-08-2021
Preliminary report (Table with results and Z-scores)	26-09-2021
Final report	27-10-2021

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).

Registration

For registration please fill in the Registration form available on the Project web page:
http://www.euchinasafe.eu/form_euchina_2021.html
Upon registration, the participant will receive a unique (confident) lab-code for submission of results and detailed Standard Operation Procedure (SOP) of the analytical method to be recommended.

Fee

For partners and linked-third parties of the EU-China-Safe project, participation is free of charge. Please note that the participant is responsible for customs clearance and associated costs, if applicable.

Confidentiality:

All laboratory specific information will be treated confidentially and will never be disclosed to third parties (government, accreditation bodies) without written permission of the laboratory.

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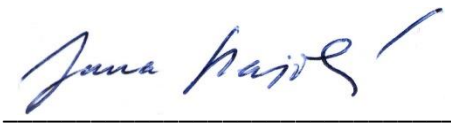

Prof. Jana Hajslova, PhD

Head of Laboratory, ILC Organizer

Appendix: Pesticides target list

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).

Annex II: Pesticides target list

UNIVERSITY OF CHEMISTRY AND TECHNOLOGY, PRAGUE
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Department of Food Analysis and Nutrition

EUCHINASAFE 中欧食品安全

Inter-laboratory Comparison Study on Pesticide Residues in Food (ILC)

PESTICIDES TARGET LIST

SOP code	ILC-Pesticides target list
Version / date of issue	2 / 18-06-2021
Author(s) (Short name of institute)	V. Kocourek (UCT Prague)
Approved by:	J. Hajslova (UCT), M. Tomaniova (UCT)
Institute:	University of Chemistry and Technology Prague Department of Food Analysis and Nutrition Technicka 3/1903; 166 28 Prague 6, Czech Republic https://uapv.vscht.cz/?jazyk=en; http://www.euchinasafe.eu/form_euchina_2021.html

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List of target analytes and their Minimum Required Reporting Levels (MRRLs)

Test material: Green Tea

This list contains all analytes (pesticides) to be sought for !

Any result reported lower than the MRRL will not be considered as False Positive, even though this result should not have been reported.

ANALYTE	MRRL (mg/kg)	ANALYTE	MRRL (mg/kg)
Acetamiprid	0.010	Malathion	0.010
Acetochlor	0.010	Mevinphos	0.010
Ametryn	0.010	Permethrin	0.010
Bifenthrin	0.010	Pirimicarb	0.010
Boscalid	0.010	Pirimiphos-methyl	0.010
Buprofezin	0.010	Prochloraz	0.010
Chlorpyrifos (ethyl)	0.050	Propamocarb	0.010
Chlorpyrifos methyl	0.050	Propargite	0.010
Clothianidin	0.010	Propiconazole	0.010
Cyanazine	0.010	Pyraclostrobin	0.010
Cypermethrin	0.050	Pyridaben	0.010
Cyprodinil	0.010	Pyriproxyfen	0.010
Difenoconazole	0.010	Resmethrin	0.010
Dimethoate	0.010	Rotenone	0.010
Dinotefuran	0.020	Spiromesifen	0.010
Fenazaquin	0.010	Spiroxamine	0.010
Fenpropimorph	0.010	Tebuconazole	0.010
Fenpropathrin	0.010	Thiacloprid	0.010
Fluopyram	0.010	Thiamethoxam	0.010
Flusilazole	0.010	Thiophanate-methyl	0.010
Hexaconazole	0.010	Tolfenpyrad	0.010
Imazalil	0.010	Triazophos	0.010
Imidacloprid	0.010	Trifloxystrobin	0.010
Iprovalicarb	0.010	Triticonazole	0.010
Isoproturon	0.010	Vamidothion	0.010

The MRRLs are typically based upon the LC-MS/MS method and MRLs found in Regulation 396/2005/EC

Annex III: 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

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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

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

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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

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 [%]
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		
		LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]	LOQ [µg/kg]	Recovery [%]	Repeatability [%]
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 IV: Fapas QC material data sheet T19303QC

Fera Science Ltd (Fera)
Sand Hutton, York, YO41 1LZ
Tel: +44 (0)1904 462100 Fax: +44(0)1904 500440
info@fapas.com www.fapas.com

FAPAS QC MATERIAL DATA SHEET	T19303QC
Matrix	Green Tea
Weight / Volume of Contents	50 g

Analyte	Assigned Value, X_a	Range for |z| ≤2	Units	No. of data points producing X_a
2-Phenylphenol (ortho-phenylphenol)	72.2	40.4 - 104.0	µg/kg	42
Anthraquinone	76.4	42.8 - 110.0	µg/kg	27
Chlorfenapyr	94.2	52.7 - 135.6	µg/kg	48
Chlorpyrifos (ethyl)	119	66 - 171	µg/kg	51
Dimethoate	67.9	38.0 - 97.7	µg/kg	49
Dinotefuran	56.7	31.7 - 81.6	µg/kg	29
Endosulfan II (beta)	58.0	32.5 - 83.5	µg/kg	41
Fenpropimorph	78.8	44.1 - 113.4	µg/kg	41
Imidacloprid	46.6	26.1 - 67.1	µg/kg	42
Malathion	107	60 - 154	µg/kg	52
Pirimiphos-methyl	138	78 - 197	µg/kg	54
Pyridaben	81.2	45.5 - 117.0	µg/kg	49
Tolfenpyrad	76.7	43.0 - 110.5	µg/kg	37

This data sheet is applicable until	13 Dec 2021
Recommended Storage on receipt	-20°C

Notes

- Mix the QC material thoroughly before taking a representative analytical sample
- The assigned value has been derived from the consensus of laboratories taking part in this proficiency test, using a variety of methods. This is not a certified reference value.
- The Range for |z| ≤2 is the concentration range within the limits of ±2 z-scores. The assigned value and its range have been established from the proficiency test data and are suitable for use by laboratories as a fit-for-purpose quality control measure.
- Stability of the QC material has been established as sufficient for the scope of the proficiency test from previous experience, expert advice and published literature. FAPAS advises that the QC material is analysed within the recommended date. FAPAS QC materials are intended to be used as single-analysis samples.
- Full details on the proficiency test procedure used to characterise this QC material are available in the Protocol, Part 1 - Common Principles, freely available to download from the FAPAS website.
- You may use any method of analysis you wish.

Annex V: Instructions for Interlaboratory Comparison Study on Pesticide Residues in Food

UNIVERSITY OF CHEMISTRY AND TECHNOLOGY, PRAGUE
Faculty of Food and Biochemical Technology
Department of Food Analysis and Nutrition

EUCHINASAFE 中欧食品安全

Inter-laboratory Comparison Study on Pesticide Residues in Food (ILC)

INSTRUCTIONS

SOP code	ILC-Instructions-2021-SP
Version / date of issue	1 / 30-06-2021
Author (Short name of institute)	V. Kocourek (UCT Prague)
Approved by:	J. Hajslova (UCT), M. Tomaniova (UCT)
Institute:	University of Chemistry and Technology Prague Department of Food Analysis and Nutrition Technicka 3/1903; 166 28 Prague 6, Czech Republic

This project has received funding from the European Union's Horizon 2020 research and innovation programme and from the Chinese Ministry of Science and Technology (MOST).

1 Introduction

The organization of Inter-laboratory Comparison Study (ILC) on Pesticide Residues in Food will be done in the frame of EU-China-Safe project (EU H2020 No. 727864).

The aim of this ILC is to obtain information regarding the quality, accuracy and comparability of pesticide residue data in food reported within the framework of EU and China laboratories implementing multidetection LC-MS method for pesticide residues analysis in food matrix developed within this project.

Participating laboratories will be provided with an assessment of their analytical performance that they can use to demonstrate their analytical performance and compare themselves with other participating laboratories [3].

The evaluation and scoring of the results of the participating laboratories will be done on an individual z-score basis and false positive (FP) or false negative (FN) rate. Classification or decisions on eligibility of laboratories for pesticide residues analysis is not done by the organiser and is beyond the scope of this Protocol.

2 Organizer

The laboratory responsible for organization and assessment of this ILC is **Metrological and Testing Laboratory UCT Prague** (Department of Food Analysis and Nutrition), accredited by the Czech Accreditation Institute (CAI) according to the EN ISO/IEC 17025:2018.

Address:
University of Chemistry and Technology Prague
Department of Food Analysis and Nutrition
Technicka 3/1903, 166 28 Prague 6, Czech Republic

Main contact:
Jana Hajslova, head of Laboratory; tel.: +420 602833424; jana.hajslova@vscht.cz

Technical assistance:
Monika Tomaniova, project management, logistic; monika.tomaniova@vscht.cz
Vladimir Kocourek, quality assurance, statistics; vladimir.kocourek@vscht.cz

3 Participants

The basic list of laboratories expected to participate in the ICL has been proposed by EU-China-Safe project partner, Professor Wu Yongning, MD, PhD, Chief Technical Officer of China National Center for Food Safety Risk Assessment (CFSA). EU Laboratories participating in the EU-China project are selected and invited by prof. Jana Hajslova, PhD (UCT Prague).

The Invitation letter / announcement together with the ILC calendar, the Pesticides target list, and the analytical method description (SOP) were distributed directly to the invited laboratories by e-mail.

4 Test sample and target analytes

The test material was produced from dry green tea leaves by the laboratory meeting the quality requirements of the ISO/IEC 17043:2010. The test material is supposed to be stable at least until November 2021.

Individual units of the material, 50 g / unit, to be analysed by laboratories participating in this ILC, were labelled with a unique code and stored at -18 ºC until dispatch. Blank material is not distributed to the participants.

The Organiser will try to ensure that all the packages arrive to each laboratory. An information message will be sent out by e-mail before shipment. Laboratories must make their own arrangements for the receipt of the package. The Organisers will not take the responsibility for a parcel if it is retained at customs.

4.1 Target pesticides list

Participants are asked to analyse test sample for all 50 potentially present pesticides indicated in the Target pesticide list, if possible. For each of the pesticides the Minimum Required Reporting Level (MRRL) is specified and **it is supposed that Limit of Quantification LOQ ≤ MRRL**. *It should not be assumed that only pesticides registered for use on tea are present in the test item*.

5 Procedures

5.1 General

5.1.1 Communication, confidentiality, collusion

Communication with the laboratories will be done by or under responsibility of the coordinator. Participating laboratories will remain anonymous in e-mails sent to multiple addresses.

For this ILC, **the laboratories are given a unique code (lab code), only known to themselves and the Organiser. In any ILC-Report, the names / identity of participating laboratories will not be linked to their laboratory codes**.

Communication between participating laboratories during the test, on matters concerning an ILC exercise, is not permitted from the start of the ILC exercise until the distribution of the preliminary report. If collusion or falsification of results by participating laboratory is suspected, all its results must be questioned and will be excluded from processing / reporting. Disclosure of expected or assigned concentrations will only be done after the deadline of result submission.

The official language used in this ILC study is English.

5.2 Work flow

5.2.1 Receipt of Test Item

The content of the consignment has to be checked upon receipt by each laboratory. The **Sample receipt form needs to be completed and signed by the participant and sent by email to the organizers** (details to be provided on the form). The organizer checks whether the codes of the test samples matches with their records. In case samples did not arrive in good condition, an alternative set may be sent immediately.

5.2.2 Advice on Test Item Handling

- upon receipt, store the sample frozen until analysis

- equilibration to room temperature and re-homogenisation of the test material is recommended before test portion weighing

- the sample need to be analysed in the same way as is done for analysis of samples in the frame of EU-China-Safe project (i.e. the same method, way of quantification, etc.)

5.2.3 Sample analysis,

Participants shall normally use the analytical method of their choice. In this ILC, however, participants are instructed to use a specific documented method provided in the frame of EU-China-Safe project. **The Laboratory is allowed to modify the original test method (adapted and implemented for their routine use) but the measuring principle (LC-MS) shall be maintained**. Any deviations from, additions to, or exclusions from the original test method shall be reported by participants.

Where an analytical method has not yet been established routinely this should be stated.

The samples need to be analysed in the same way as is done for analysis of samples in the frame of EU-China-Safe project.

5.2.4 Reporting results

Result submission form (in the MS Excel) is provided by the organizer and needs to be used by the participants to submit the results and method information to the organizer.

Participating laboratories are responsible for reporting quantitative results to the Organiser within the stipulated deadline.

Any pesticide that was targeted by a participating laboratory should be reported as "Analysed" and „Detected / Not detected", together with the information on the relevant „Reporting Limit". Symbols (>, <, ±, ≥, ≤, …) will not be accepted. If the result is not correctly expressed it will be considered as 'ND' (Not Detected).

Each laboratory will be able **to report only one result for each analyte detected** in the Test Item. **The concentrations of the pesticides detected should be expressed in 'mg/kg'**.

Laboratories are required to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery rate (in percentage) must also be reported.

When all fields are filled out, laboratories must **submit their final results by E-mail, before 31st August 2021** (details to be provided on the form).

Participants will receive an E-mail confirming the submission of their results. After the final submission it will NOT be possible to edit the results.

5.2.5 Communication of preliminary results to participants

Data reported by the laboratories will be statistically evaluated [3-5] and z-scores will be calculated. A summary table of results will be compiled and sent to the participants as a first feedback on their performance.

5.2.6 ILC report

Reporting will be done by the organizer according to EU-China-Safe project requirements. The organizer will then organise a telecon/web meeting to present and discuss the results. The draft report will be sent after approval to the participants. After that, the report will be finalized and sent to all participants. The report will also be published on the EU-China-Safe project website.

5.2.7 Questions and Remarks

Participants have the possibility to send their questions and remarks to the Organizer. These will be examined and feedback will be provided. If appropriate, corrective actions will be taken.

5.2.8 ILC Calendar

Deadline registration	30-06-2021
Distribution of test samples	07-07-2021
Deadline for submission of results	31-08-2021
Preliminary report (table with results and Z-scores)	26-09-2021
Final report	27-10-2021

6 References

[1] ISO/IEC 17043:2010 "Conformity assessment – General requirements for proficiency testing"

[2] EA-4/21 INF:2018 Guidelines for the assessment of the appropriateness of small interlaboratory comparisons

[3] General protocol for EU Proficiency Tests on Pesticide Residues in Food and Feed provided by European Reference Laboratories, 9th Ed., Nov 2019

[4] ISO 13528: Statistical methods for use in proficiency testing by interlaboratory comparisons

[5] Protocol for Proficiency testing Schemes (Part 1: Common Principles), version 7, Jan 2021, FERA Science Ltd, Sand Hutton, York, UK.

Annex VI: Sample receipt form

Sample receipt form ILC

Interlaboratory Comparison Study: Pesticide residues in green tea leaves

Test Sample No. see the bag	
Contact person:	
Institute:	

Please verify that the items listed below have been received and provide the information requested below.

Content of parcel:
- 1 bag (50 g test sample)
- Sample receipt form ILC
- Instructions (Specific protocol)

Date of receipt (dd-mm-yyyy)		
Conditions	Ambient / Cooled	
Instructions – legible, understandable	Yes / No	
Damaged/Leakage	Yes / No	Notice:

Remarks:

Name	
Signature	

Date:

After signing this form, please scan and send the pdf by E-mail to:
monika.tomaniova@vscht.cz
vladimir.kocourek@vscht.cz

Organiser:
Prof. Jana Hajslova, tel.: +420 602833424; jana.hajslova@vscht.cz
University of Chemistry and Technology Prague
Department of Food Analysis and Nutrition
Technicka 3/1903, 166 28 Prague 6, Czech Republic

Annex VII: Reporting form

Fill or check off Green fields only !

Lab code (see e-mail)	Compound	Analyzed?*	Detected?*	Laboratory LOQ (mg/kg)**	Concentration measured (mg/kg)***	Expanded uncertainty (mg/kg)	Result corrected for recovery?	Recovery (%)	Comments	Sample weight (g)	Extraction/partitioning solvent &	Extraction time	Water addition?	Cleanup 1	Cleanup 2	Cleanup details	Calibration (select one)	LC Analytical column (specification)	Mass spectrometer (select one)	Source of calibration standard (Company, Cat. No.)
	Acetamiprid																			
0	Acetochlor																			
0	Ametryn																			
0	Bifenthrin																			
0	Boscalid																			
0	Buprofezin																			
0	Chlorpyrifos (ethyl)																			
0	Chlorpyrifos methyl																			
0	Clothianidin																			
0	Cyanazine																			
0	Cypermethrin																			
0	Cyprodinil																			
0	Difenoconazole																			
0	Dimethoate																			
0	Dinotefuran																			
0	Fenazaquin																			
0	Fenpropimorph																			
0	Fenpropathrin																			
0	Fluopyram																			
0	Flusilazole																			
0	Hexaconazole																			
0	Imazalil																			
0	Imidacloprid																			
0	Iprovalicarb																			
0	Isoproturon																			
0	Malathion																			
0	Mevinphos																			
0	Permethrin																			
0	Pirimicarb																			
0	Pirimiphos-methyl																			
0	Prochloraz																			
0	Propamocarb																			
0	Propargite																			
0	Propiconazole																			
0	Pyraclostrobin																			
0	Pyridaben																			
0	Pyriproxyfen																			
0	Resmethrin																			
0	Rotenone																			
0	Spiromesifen																			
0	Spiroxamine																			
0	Tebuconazole																			
0	Thiacloprid																			
0	Thiamethoxam																			
0	Thiophanate-methyl																			
0	Tolfenpyrad																			
0	Triazophos																			
0	Trifloxystrobin																			
0	Triticonazole																			
0	Vamidothion																			
0																				
0																				
0																				

* please fill (YES/NO) for all pesticides listed - compulsory field.

** insert your own laboratory LOQ for each Analyzed pesticide - compulsory field if "YES" in the column C.

*** insert your result for each Detected pesticide (4 decimal places) - compulsory field if "YES" in the column D.

Annex VIII: Preliminary report - Interlaboratory Comparison Study on Pesticide Residues in Food

UNIVERSITY OF CHEMISTRY AND TECHNOLOGY, PRAGUE
Faculty of Food and Biochemical Technology
Department of Food Analysis and Nutrition

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This document contains the ILC preliminary results anf their evaluation. Please check if the results submitted by you are correct. If you should find any anomalies, please contact us. If you don´t contact us before September 30th, we will understand that you agree with them and we will proceed with the final evaluation of the results.

Table 1. Concentration of the pesticides in the test item (mg/kg) reported by laboratories

Assigned value *preliminary*	Compound	Lab 10	Lab 11	Lab 12	Lab 13	Lab 14	Lab 15	Lab 16 EU
0,119	Chlorpyrifos (ethyl)	0,1313	0,1821	0,1401	0,1335	0,1411	FN	0,1220
0,068	Dimethoate	0,0720	0,0838	0,0676	0,0712	0,0731	0,0500	0,0570
0,056	Dinotefuran	*0,05*	0,0551	0,0418	0,0502	0,0654	0,0600	0,0470
0,079	Fenpropimorph	0,0664	0,1064	0,0802	0,0784	0,0890	FN	0,0790
0,047	Imidacloprid	0,0447	0,0491	0,0417	0,0402	0,0446	FN	0,0430
0,107	Malathion	0,1121	0,1413	0,1090	0,1072	0,1200	0,0500	0,1190
0,138	Pirimiphos-methyl	0,1455	0,1912	0,1712	0,1709	0,1470	0,0230	0,1410
0,081	Pyridaben	0,0875	0,1070	0,0835	0,0894	0,0881	0,0200	0,0850
0,077	Tolfenpyrad	0,0838	0,1145	0,0720	0,0724	0,0846	0,2100	0,0790
	Properly identified:	**9**	**9**	**9**	**9**	**9**	**6**	**9**

FN - False Negative result

FP - False Positive results: **LAB 14** Fenpropathrin

LAB 15 Cyanazine, Pirimicarb

Table 2: z-scores using Fit-For-Purpose RSD (relative standard deviation for proficiency) = 25 %.

Compound	Lab 10	Lab 11	Lab 12	Lab 13	Lab 14	Lab 15	Lab 16 EU
Chlorpyrifos (ethyl)	0,41	2,12	0,71	0,49	0,74	FN	0,10
Dimethoate	0,24	0,94	-0,02	0,19	0,31	-1,05	-0,64
Dinotefuran	*-0,43*	-0,06	-1,01	-0,41	0,67	0,29	-0,90
Fenpropimorph	-0,63	1,40	0,07	-0,02	0,52	FN	0,01
Imidacloprid	-0,16	0,21	-0,42	-0,55	-0,17	FN	-0,31
Malathion	0,19	1,28	0,08	0,01	0,49	-2,13	0,02
Pirimiphos-methyl	0,22	1,54	0,96	0,95	0,26	-3,33	0,09
Pyridaben	0,31	1,27	0,12	0,40	0,34	-3,01	0,24
Tolfenpyrad	0,37	1,97	-0,25	-0,22	0,41	6,95	0,35

The z scores were calculated using FFP RSD of 25 %. In normal circumstances, about 95 % of z-scores will lie in the range -2 ≤ z ≤ +2.

FN, FP and scores where |z| > 3 very strongly indicate that the result is NOT fit-for-purpose and requires investigation.

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Annex IX: Final report - Interlaboratory Comparison Study on Pesticide Residues in Food

UNIVERSITY OF CHEMISTRY AND TECHNOLOGY, PRAGUE
Faculty of Food and Biochemical Technology
Department of Food Analysis and Nutrition

EUCHINASAFE 中欧食品安全

Inter-laboratory Comparison Study on Pesticide Residues in Food (ILC)

FINAL REPORT

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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).

Summary

1. Six laboratories from China and one laboratory from EU agreed to participate in this ILC study.

2. Test materials for Inter-laboratory Comparison Study (ILC) on “Pesticide Residues in green tea” were dispatched in July 2021. Each participant received one green tea sample to be analysed for pesticide residues using LC-MS multidetection method provided by the ILC organizers.

3. An assigned value (X_a) was set for each analyte. A fit-for-purpose relative target standard deviation (σ_{FFP}) of 25 % was chosen to calculate the target standard deviations (σ) as well as the z-scores for the individual pesticides.

4. Results for this ILC study are summarised as follows:

analyte	**assigned value (X_a) [mg/kg]**	**number of scores $\lvert z\rvert \leq 2.0$**	**total number of analytes**	**% $\lvert z\rvert \leq 2.0$**	**number of False Negative**
Chlorpyrifos (ethyl)	0.119	5	7	71 %	1
Dimethoate	0.068	7	7	100 %	-
Dinotefuran	0.056	7	7	100 %	-
Fenpropimorph	0.079	6	7	86 %	1
Imidacloprid	0.047	6	7	86 %	1
Malathion	0.107	6	7	86 %	-
Pirimiphos-methyl	0.138	6	7	86 %	-
Pyridaben	0.081	6	7	86 %	-
Tolfenpyrad	0.077	6	7	86 %	-

Total number of False Positive results: 3

Total number of False Negative results: 3

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1 Introduction

Organization of this **Inter-laboratory Comparison Study (ILC) on Pesticide Residues in Food** was performed in the frame of the EU-China-Safe project (EU H2020, No. 727864).

The aim of this ILC study was to obtain information on the quality, accuracy and comparability of pesticide residues data in food reported within the framework of EU and China laboratories implementing multidetection LC-MS method developed within the EU-China-Safe project for pesticide residues analysis in specific food matrix.

For this ILC, requirements of the:

- ISO/IEC 17043:2010 "Conformity assessment – General requirements for proficiency testing" [1]
- EA-4/21 INF:2018 Guidelines for the assessment of the appropriateness of small interlaboratory comparisons [2]
- General protocol for EU Proficiency Tests on Pesticide Residues in Food and Feed provided by European Reference Laboratories, 9th Ed., Nov 2019 [3]
- ISO 13528: Statistical methods for use in proficiency testing by interlaboratory comparisons [4]
- Protocol for Proficiency testing Schemes (Part 1: Common Principles), version 7, Jan 2021, FERA Science Ltd, Sand Hutton, York, UK [5]

have been taken into account.

Participating laboratories are provided with an assessment of their analytical performance that they can use to demonstrate their analytical performance and compare themselves with other participating laboratories [3]. This will aid in the quality improvement of analysis at each of the involved laboratories.

The evaluation and scoring of the results of the participating laboratories was done on an individual z-score basis and false positive (FP) or false negative (FN) rate.

Classification or decisions on eligibility of laboratories for pesticide residues analysis is not done by the organisers and is beyond the scope of this Report.

2 Organizer

The ILC was **organised by the Department of Food Analysis and Nutrition, University of Chemistry and Technology Prague (Organizer) (UCT Prague, Czech Republic)**, in collaboration with Queens University Belfast (QUB, UK) and China National Center for Food Safety Risk Assessment,(CFSA, China) and supported by the FERA Science LtD (UK).

3 Participants

The basic list of Chinese laboratories expected to participate in the ILC study was proposed by the EU-China-Safe project Chinese coordinator, Professor Wu Yongning, MD, PhD, Chief Technical Officer of China National Center for Food Safety Risk Assessment (CFSA).

ILC that is organised among seven or less laboratories applies the appropriate ISO/IEC 17043 requirements (see EA-4/21 INF:2018).

The Invitation letter together with the ILC calendar, and the analytical method description (SOP) were distributed directly to the invited laboratories by the e-mail.

Alphabetical list of laboratories participating in the ILC study:

- Academy of National Food and Strategic Reserves Administration, Institute of Cereals and Oils Quality and Safety (China)
- Beijing Center For Disease Control and Prevention, Central Lab (China)
- Chinese Academy of Inspection and Quarantine, Institute of Food Safety (China)
- China National Center for Food Safety Risk Assessment, Food Chemistry (China)
- Metrological and Testing Laboratory, University of Chemistry and Technology Prague (EU)
- Shanghai Customs, China, Animal, Plant and Food Inspection and Quarantine Technical Center (China)
- Shanghai Municipal Center For Disease Control and Prevention (China)

Participants were provided also with the Pesticides target list and were asked to analyse test sample for all 50 potentially present pesticides. For each of the pesticides the Minimum Required Reporting Level (MRRL) is specified; it was supposed that Limit of Quantification LOQ ≤ MRRL.

Each participant was given a laboratory number (in random order) by the Organizer.

4 Test sample

The test material was produced from dry green tea leaves. Material was homogenized and analysed for incurred residues. Subsequently, selected analytes were spiked into the blank test material. **Preparation and homogeneity** testing was subcontracted by FERA Science Ltd (as the participant in the EU-China-Safe project) to a laboratory meeting the quality requirements of the ISO/IEC 17043:2010.

Stability of the test material has been established by FERA as sufficient for the ILC from previous experience, expert advice, and published literature. Based on this, the test material is supposed to be stable at least until 13th November 2021 (-20°C). Nevertheless, the Organiser also decided to conduct additional stability tests at different storage conditions than those recommended to the participants (i.e. -20 °C): 90 days at 25 °C (ambient temperature) and at 4 °C (refrigerator). No LC-MS amenable pesticide residues were detected at, or above, 0.01 mg/kg.

Stability experiment (March – September 2021):

(mg/kg)	**time 0**	**90 days at 4 °C**	**90 days at 25 °C**	*assigned value*	*0.3 x σ*
chlorpyrifos	0.121	0.114	0.122	*0.119*	*0,009*
dimethoate	0.055	0.057	0.055	*0.068*	*0,005*
dinotefuran	0.044	0.046	0.044	*0.057*	*0,004*
fenpropimorph	0.079	0.077	0.079	*0.079*	*0,006*
imidacloprid	0.043	0.039	0.043	*0.047*	*0,004*
malathion	0.108	0.117	0.111	*0.107*	*0,009*
pirimiphos-methyl	0.141	0.151	0.146	*0.138*	*0,010*
pyridaben	0.086	0.085	0.090	*0.081*	*0,006*
tolfenpyrad	0.082	0.077	0.079	*0.077*	*0,006*

Individual units of the material, 50 g / unit, to be analysed by laboratories participating in this ILC, were labelled by UTC Prague with a unique code and identification of the ILC study and stored at -18 ºC until dispatch (see Fig. 1). Blank material was not distributed to the participants.

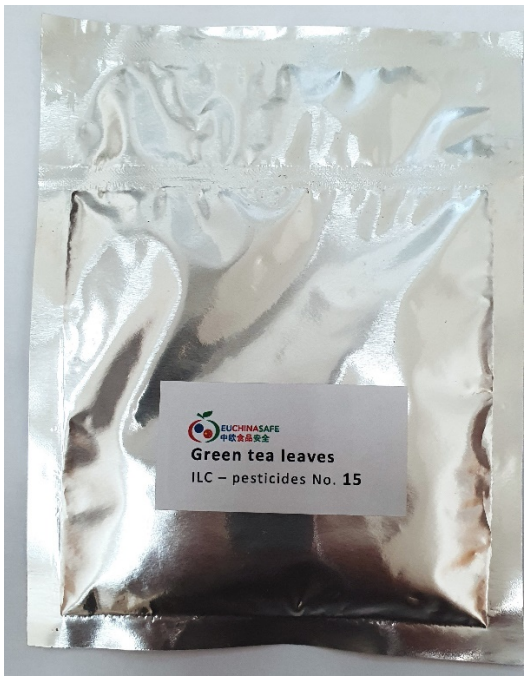

Fig. 1: Test material unit labelled and ready for distribution

Laboratories got about 4 weeks for sample analysis and reporting the results. Due to technical issues announced by some laboratories, extended **closing date for reporting was set on 10th September 2021**.

5 Results

Results were submitted by all 7 laboratories (100 %) before the closing date.

Participants shall normally use the analytical method of their choice. In this ILC, however, **participants were asked to use a specific documented method, provided in the frame of the EU-China- Safe project**. The Laboratory was allowed to modify the original test method (adapted and implemented for their routine use) but the measuring principle (LC-MS) shall be maintained. Any deviations from, additions to, or exclusions from the original test method should be reported.

Any pesticide that was targeted by a participating laboratory was reported as “Analysed” and „Detected” or “Not detected“, together with the information on its own „Laboratory LOQ“.

Each laboratory was allowed to report only one result for each analyte quantified in the test material. The concentrations of the pesticides detected are expressed in mg/kg.

Laboratories were asked to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery rate (in percentage) was also reported.

False positive results (FP):

These are results for pesticides reported by the laboratory as ’Detected’ and with reporting numerical values although they were not detected by the Organiser (using both LC-MS and GC-MS multidetection methods) as well as the absolute majority of the participants.

Any results reported lower than the MRRL given in the List of target pesticides will not be considered as false positives, even though these results should not have been reported. [3]

False Negative results (FN)

These are results for pesticides reported by the laboratory as ’Analysed’ but without reporting numerical values although they were detected by the Organiser as well as the majority of the participants that had targeted these specific pesticides at or above the respective MRRLs. Results reported as either ’< LOQ’ or ’< MRRL’ are considered as ’not detected’ and judged as False negatives. [3]

Estimation of the assigned value (X_a)

Test material from the batch used in the recent Proficiency Testing (PT) round provided by Fapas® (Fera Science Ltd., UK) was used in this ILC study. FAPAS PT materials undergo both homogeneity and stability testing (including long-term stability test for its subsequent use). Datasheet associated with the test material provides the same data of assigned values and uncertainties as in the FAPAS PT.

As a FAPAS PT test material was available in sufficient amounts for use in an ILC study, the reference values (and associated uncertainties) can be confirmed and used directly. This is quick and simple to implement, and provides a value independent of the participant results.

The assigned value for each analyte was derived from the consensus of the valid results submitted by 29 - 54 participants (suspect or invalid entries were excluded). Robust mean was chosen as the assigned value X_a [5].

Standard deviation of the assigned value (target standard deviation)

The target standard deviation of the assigned value (σ_{FFP}) was calculated using a Fit-For-Purpose approach with a fixed Relative Standard Deviation: **FFP-RSD**.

Based on the long-term experience in the Proficiency Tests on pesticide residues in food organised annually by EU Reference Laboratories [3], a percentage **FFP RSD of 25 %** is currently used for all analyte-matrix combination, with the target standard deviation being calculated as follows:

$\sigma_{FFP} = 0.25 * X_a$

Z-scores

This parameter is calculated for each analyte and laboratory using the following formula:

$z = (x - X_a) / \sigma_{FFP}$

where x is the value reported by the laboratory, X_a is the assigned value, and σ_{FFP} is the standard deviation using the FFP approach. Z-scores are rounded to two decimal places.

Z-scores are interpreted in the following way, as is set in the ISO 17043:2010:

|z| ≤ 2.0 Acceptable

2.0 < |z| < 3.0 Questionable

|z| ≥ 3.0 Unacceptable

Nevertheless, the limits mentioned above must not be regarded as strict boundaries but should be treated as action limits. Z-scores are statistics based on the random selection from a hypothetical normal distribution and must be interpreted as such [5].

Data reported by the laboratories were statistically evaluated [3-5] and z-scores were calculated. A summary table of preliminary results, Preliminary Report (v.1, 20th September) was sent to the participants as the first feedback on their performance.

All results reported by participants before the closing date (including original results of the Lab 15) are summarized in **Appendix, Table 1,** and **Figures 1 - 7**.

Corrective actions

Lab 15 analyzed the reasons that caused the unacceptable results and adopted the corrective action:

1) for three pesticides including pirimiphos-methyl, pyridaben, tolfenpyrad which have been screened out successfully but with large deviation in quantitative results, wrong standard solutions were used. New standard solutions have been prepared.

2) for the three pesticides including chlorpyrifos (ethyl), fenpropimorph, imidacloprid which have not been screened out and the two pesticides including cyanazine and pirimicarb which were false positive, the matrix interference occurred.

3) the test sample was re-analyzed and rectified results have been sent to organizers.

Corrected results reported by Lab 15 (after closing date) are presented in **Appendix, Table 2**.

6 Methods

Method origin:

- The SOP provided by the Organizer: Lab 10, 11, 13, 14, 15, 16
- "original AOAC" - no details provided: Lab 12

Chromatographic column:

- Lab 10: Acclaim RSLC 120 C18 (150 x 2.1 mm), 2.2 µm
- Lab 11: *not specified*
- *Lab 11 comment: in the given gradient elution procedure, caffeine in tea can have obvious matrix effect on some detected pesticides, such as imidacloprid, or other unscreened pesticides. Therefore, in the case of complex matrix, it is recommended to prolong the chromatographic analysis time to reduce the matrix effect of matrix interferents on targeted analytes.*
- Lab 12: Accucore aQ (150 x 2.1 mm), 2.7 µm; Thermo Scientific, Phenomenex (USA)
- Lab 13: Poroshell 120 EC-C18 (150 x 3.0 mm), 2.7 µm
- Lab 14: Waters ACQUITY UPLC HSS T3 column (100 x 2.1 mm),1.7 µm
- Lab 15: *not specified*
- Lab 16: Waters ACQUITY UPLC HSS T3 column (100 x 2.1 mm), 1.7 µm

Mass spectrometry:

- MS/MS Triple Quadrupole: Lab 13, 14, 15, 16
- High Resolution MS (Q-)Orbitrap: Lab 11, 12
- High Resolution MS: Lab 10

Recovery, correction:

- recoveries reported, results not corrected: Lab 11, 16
- recoveries reported, results corrected for imidacloprid and pirimiphos methyl (< 80 %): Lab 13
- recoveries not reported, results not corrected: Lab 14
- recoveries not reported, no information on correction: Lab 12, 15

Calibration:

- matrix-matched calibration: Lab 10, 13, 16
- standard addition: Lab 11, 12
- no information provided (matrix matched ?): Lab 15

Source of calibration standards – metrological traceability:

- traceable (LGC / Dr Ehrenstorfer / HPC standards / AccuStandards): Lab 11, 12, 13(?), 16
- "domestic": Lab 10
- no information provided, unknown: Lab 14, 15

7 References

[1] ISO/IEC 17043:2010 "Conformity assessment – General requirements for proficiency testing"

[2] EA-4/21 INF:2018 Guidelines for the assessment of the appropriateness of small interlaboratory comparisons

[3] General protocol for EU Proficiency Tests on Pesticide Residues in Food and Feed provided by European Reference Laboratories, 9th Ed., Nov 2019

[4] ISO 13528: Statistical methods for use in proficiency testing by interlaboratory comparisons

[5] Protocol for Proficiency testing Schemes (Part 1: Common Principles), version 7, Jan 2021, FERA Science Ltd, Sand Hutton, York, UK

8 Appendix

Table 1: Concentration of the pesticides (mg/kg) reported by laboratories and z-scores using Fit-For-Purpose RSD (relative standard deviation for proficiency = 25 %). MRRL: Minimum Required Reporting Level

Laboratory code	chlorpyrifos	Z scores (FFP RSD (25%)	dimethoate	Z scores (FFP RSD (25%)	dinitefuran	Z scores (FFP RSD (25%)	fenpropimorph	Z scores (FFP RSD (25%)	imidacloprid	Z scores (FFP RSD (25%)	malathion	Z scores (FFP RSD (25%)	pirimiphos-methyl	Z scores (FFP RSD (25%)	pyridaben	Z scores (FFP RSD (25%)	tolfenpyrad	Z scores (FFP RSD (25%)
MRRL	**0,050**		**0,010**		**0,020**		**0,010**		**0,010**		**0,010**		**0,010**		**0,010**		**0,010**	
Assigned value	**0,119**		**0,068**		**0,057**		**0,079**		**0,047**		**0,107**		**0,138**		**0,081**		**0,077**	
LAB 10	0,131	0,41	0,072	0,24	*0,050*	-0,47	0,066	-0,63	0,045	-0,16	0,112	0,19	0,146	0,22	0,088	0,31	0,084	0,37
LAB 11	0,182	2,12	0,084	0,94	0,055	-0,11	0,106	1,40	0,049	0,21	0,141	1,28	0,191	1,54	0,107	1,27	0,115	1,97
LAB 12	0,140	0,71	0,068	-0,02	0,042	-1,05	0,080	0,07	0,042	-0,42	0,109	0,08	0,171	0,96	0,084	0,12	0,072	-0,25
LAB 13	0,134	0,49	0,071	0,19	0,050	-0,46	0,078	-0,02	0,040	-0,55	0,107	0,01	0,171	0,95	0,089	0,40	0,072	-0,22
LAB 14	0,141	0,74	0,073	0,31	0,065	0,61	0,089	0,52	0,045	-0,17	0,120	0,49	0,147	0,26	0,088	0,34	0,085	0,41
LAB 15	FN	FN	0,050	-1,05	0,060	0,23	FN	FN	FN	FN	0,050	-2,13	0,023	-3,33	0,020	-3,01	0,210	6,95
LAB 16	0,122	0,10	0,057	-0,64	0,044	-0,90	0,079	0,01	0,043	-0,31	0,108	0,02	0,141	0,09	0,086	0,24	0,084	0,35

Unacceptable results are in red !

False positive:
LAB 14 fenpropathrin
LAB 15 cyanazine, pirimicarb

False negative:

LAB 15 chlorpyrifos, fenpropimorph, imidacloprid

Table 2: Results (mg/kg) and related and z-scores additionally reported by Lab 15 after corrective action.

Assigned value *preliminary*	**Compound**	***Lab 15 rectified results***	***Lab 15 rectified z-score***
0,119	chlorpyrifos (ethyl)	0,132	0,45
0,068	dimethoate	0,050	-1,05
0,056	dinotefuran	0,060	0,23
0,079	fenpropimorph	0,085	0,31
0,047	imidacloprid	0,047	0,00
0,107	malathion	0,050	-2,13
0,138	pirimiphos-methyl	0,143	0,14
0,081	pyridaben	0,082	0,04
0,077	tolfenpyrad	0,082	0,28

Corrected results are in blue.

Both cyanazine and pirimicarb (false positive before ILC closing date) were not detected now.

Figure 1: z-scores for chlorpyrifos

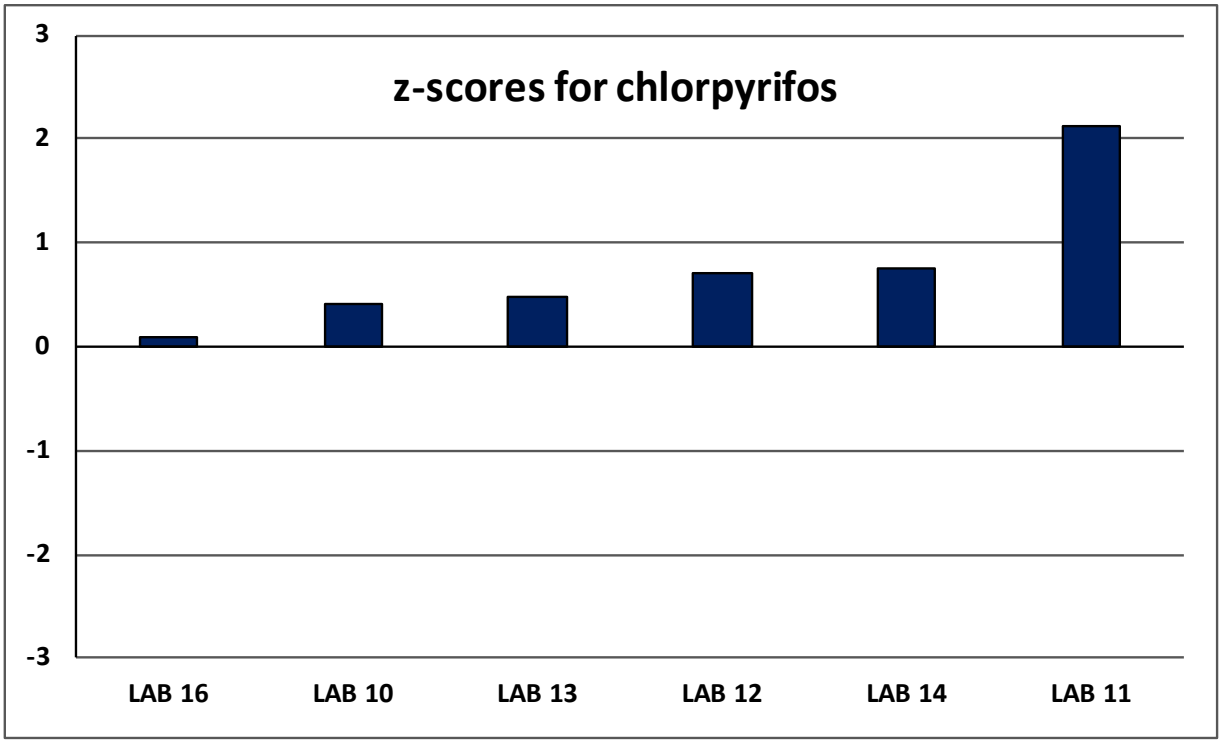

Figure 2: z-scores for dinotefuran

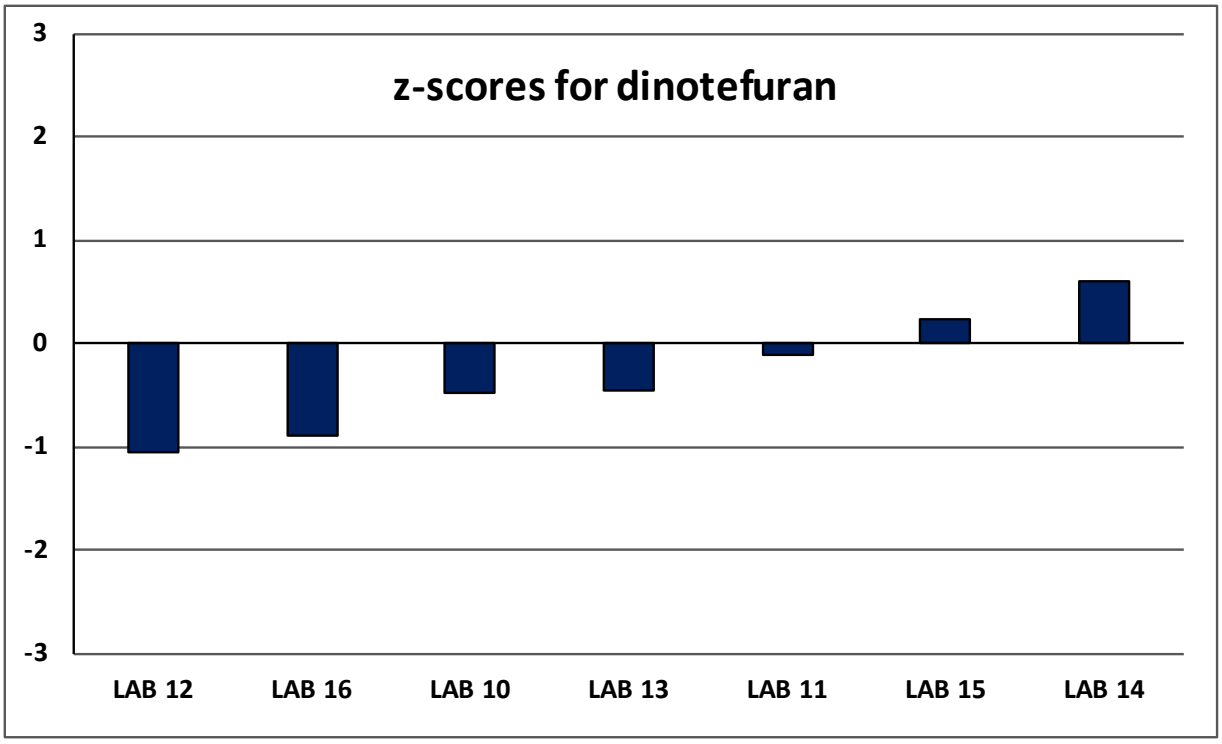

Figure 3: z-scores for fenpropimorph

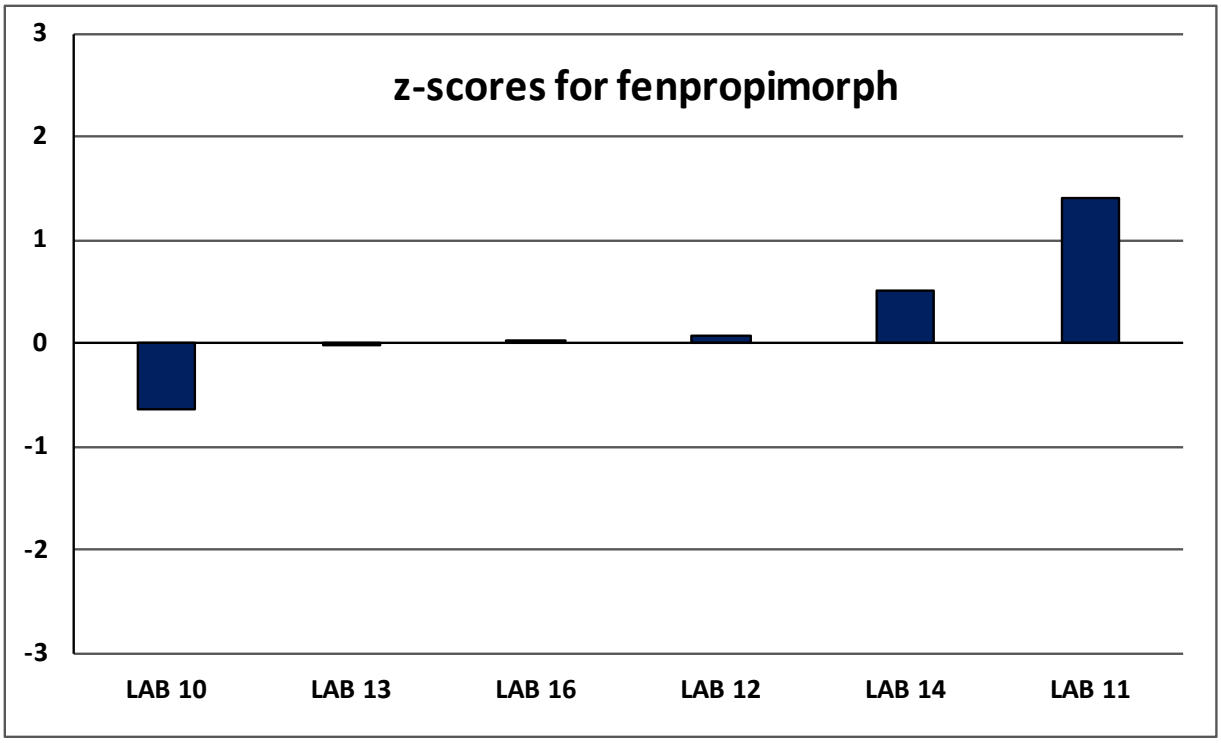

Figure 4: z-scores for imidacloprid

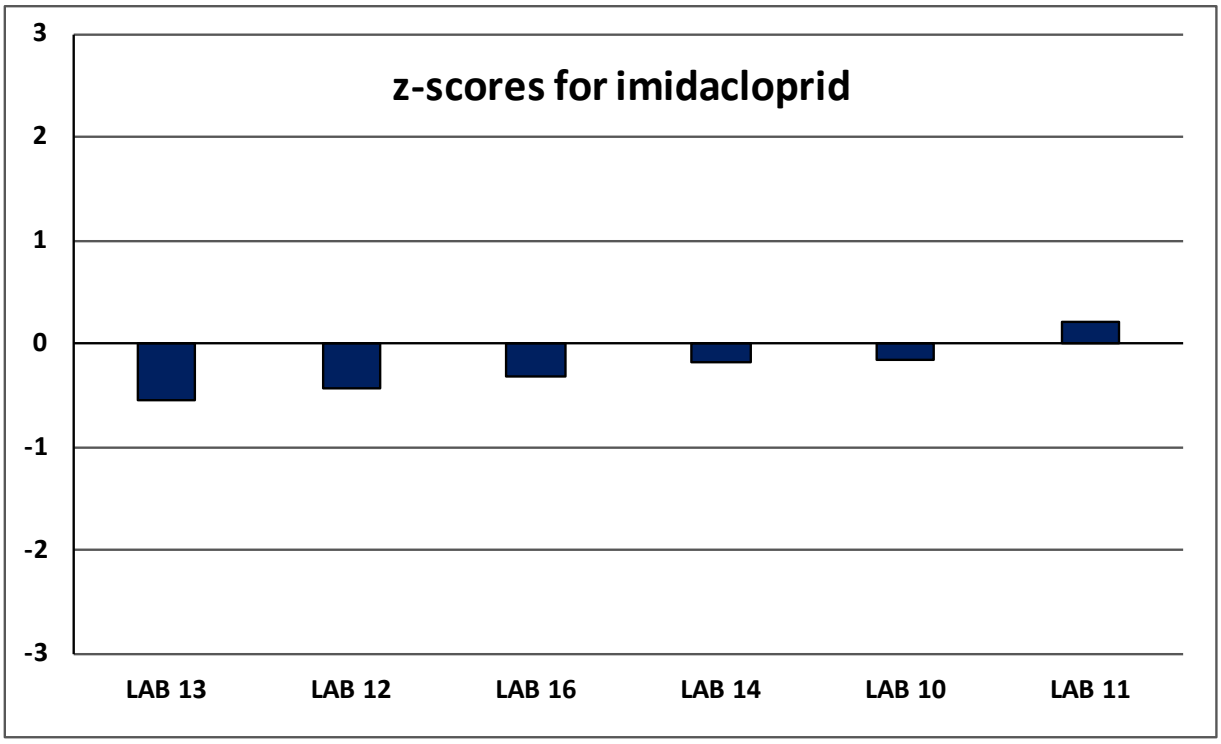

Figure 5: z-scores for malathion

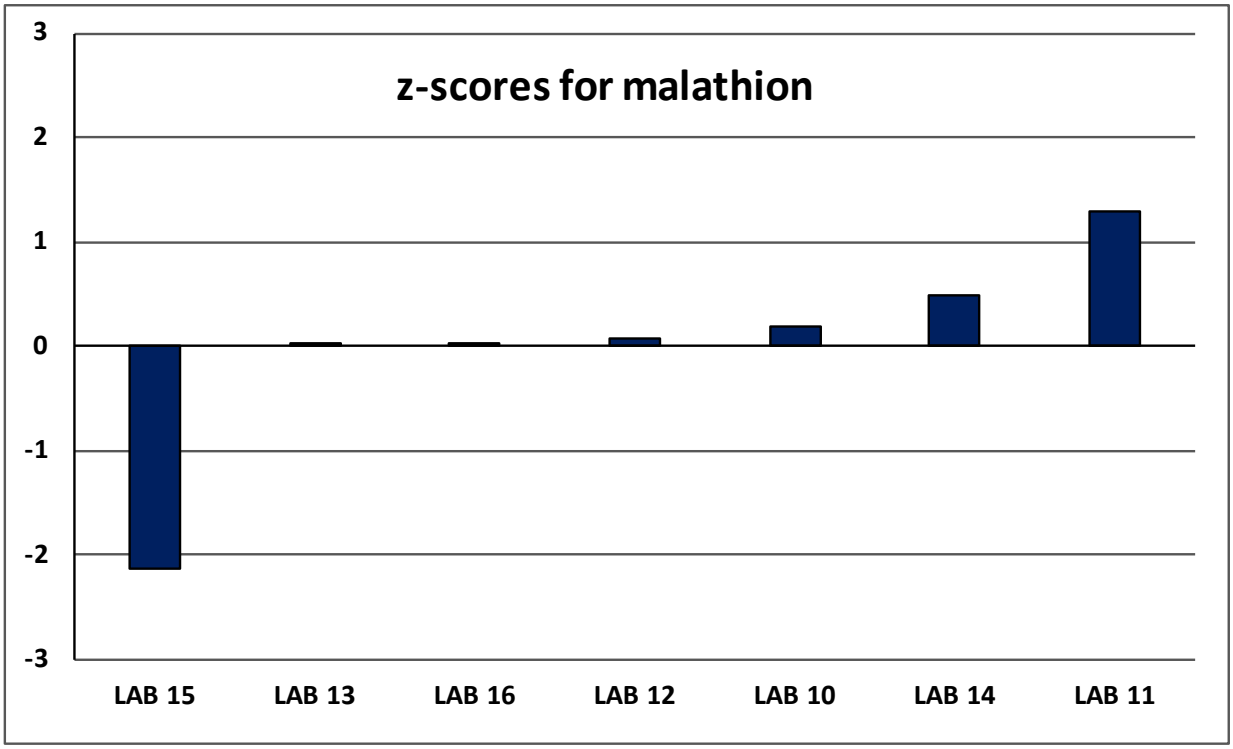

Figure 6: z-scores for pirimiphos-methyl

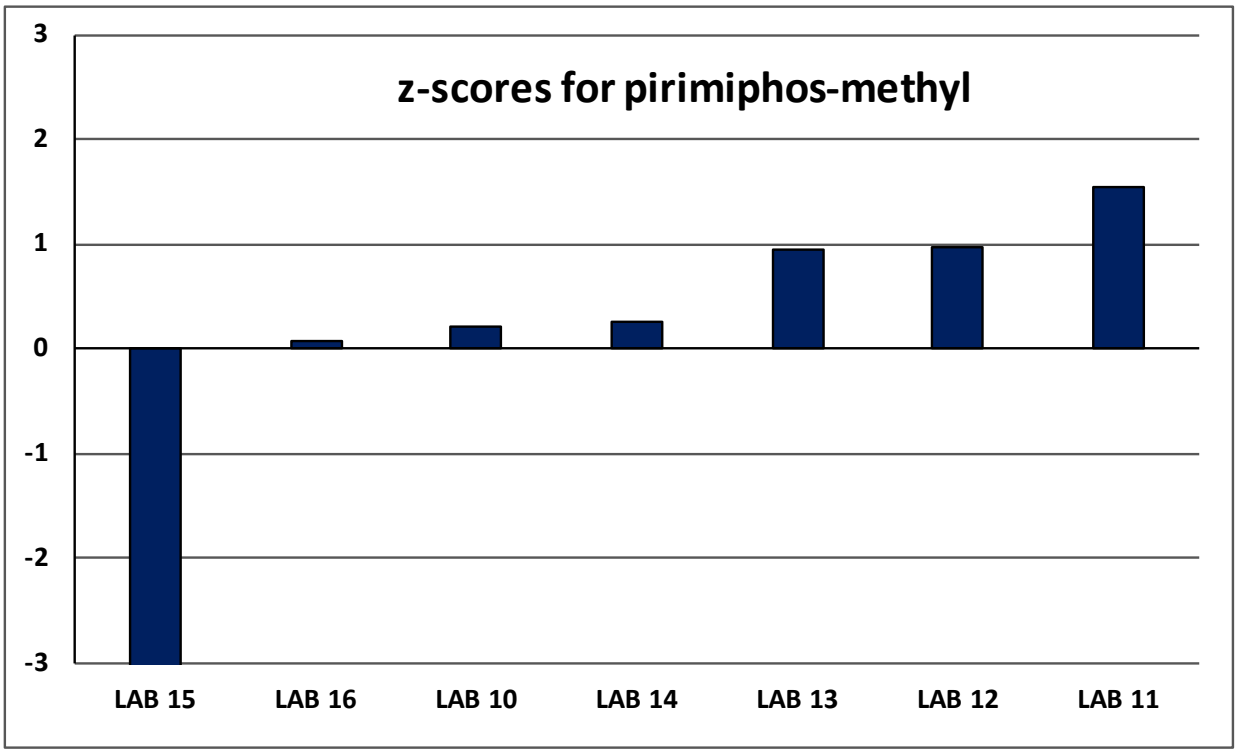

Figure 7: z-scores for pyridaben

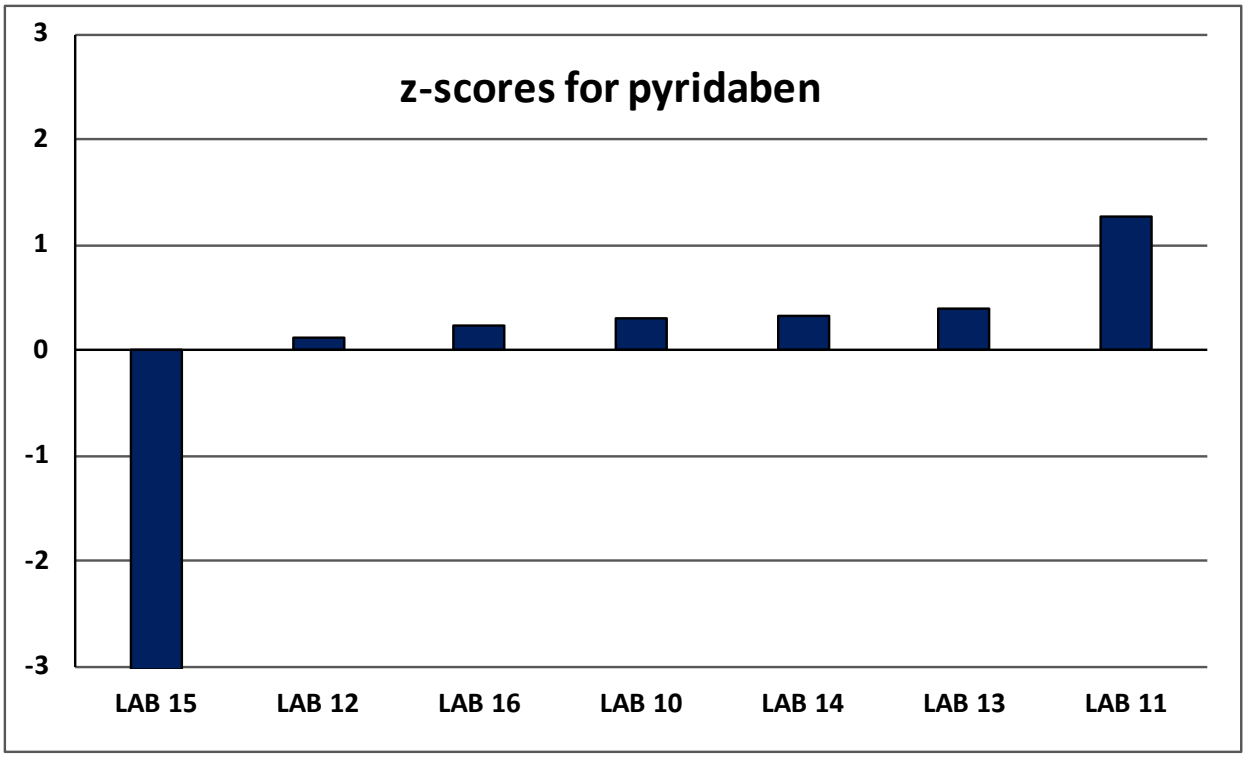

Figure 8: z-scores for tolfenpyrad