

Simultaneous screening and quantification of pesticide residues in potato using GC-Orbitrap MS

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Goal

To develop a combined targeted screening and quantitation method for pesticide residues in potato using gas chromatography coupled to Orbitrap™ mass spectrometry. The optimized method performance was evaluated following the SANTE/12682/2019 guidelines and assessed for compliance with maximum residue levels (MRLs) for potato from the Food Safety and Standards Authority of India (FSSAI) and the European Commission (EC).

Introduction

Potato (*Solanum tuberosum*) is a major root crop that contributes to food security in developing countries.¹ Often, potato cultivation involves unregulated applications of pesticides, thereby leading to non-compliance issues related to trade and potential health hazards to consumers. According to a report provided by the United States Food and Drug Administration, every year around 10% of the



imported potato samples fail to comply with the MRLs. Despite these concerns, there are very few reported validated analytical methods for the analysis of pesticide residues in potato.²

With available technologies like GC-MS/MS, it is possible to detect and quantify the presence of pesticides in potato with unit mass resolution as per the SANTE/12682/2019 quantitation and identification criteria.³ When using triple quadrupole MS, the selectivity required to separate target pesticides from the chemical background is achieved by the use of selected reaction monitoring (SRM). SRM is used in targeted experiments in which the mass spectrometer is pre-programmed utilizing a list of predefined pesticides. During acquisition, the target-specific list of compounds

limits the scope of analysis so pesticides present in the sample but not included in the acquisition list will not be detected and will result in non-detection (false negative) for additional compounds. This limitation has increased the interest for developing methods using high-resolution full scan mass spectrometry, which offers better selectivity due to accurate mass measurement and equal sensitivity. Sample preparation is also equally important for the analysis of residues in food matrices. For sample preparation, there are few generic multi-residue extraction methods reported in the literature. The QuEChERS acetonitrile approach is the most popular and was selected for this study of multi-residue pesticides analysis with respect to their scope.⁴

This work aimed to develop and validate an analytical method for simultaneous screening and quantification of pesticide residues in potato by using the QuEChERS extraction method in combination with the Thermo Scientific™ Exactive™ GC Orbitrap™ GC-MS system operated in full scan mode. The data acquisition and processing were carried out by using Thermo Scientific™ TraceFinder™ software. The optimized method was validated as per the SANTE/12682/2019 guidelines³.

Experimental

GC-Orbitrap analysis

The instrument used was the Thermo Scientific™ TRACE™ 1310 GC coupled to the Exactive GC Orbitrap high-resolution accurate mass spectrometry (HRAM MS) system, with electron impact (EI) ionization and VPI technology. The optimized GC-MS conditions are given in Table 1.

Sample preparation

Reagents and chemicals

- Acetonitrile, Optima™ LC/MS Grade, Fisher Scientific™ (P/N 514 L-16923 U)
- Anhydrous Magnesium Sulfate (MgSO₄), Thermo Scientific™ (P/N 80020-415-500)
- EN 15662 QuEChERS Extraction kit, Thermo Scientific™ (4 g anhydrous MgSO₄, 1 g NaCl, 1 g Na₃Citrate, and 500 mg Na₂Citrate), (P/N 60105-216)
- PSA (Primary Secondary Amine), Thermo Scientific™ (P/N 80020-416-100)

Table 1. GC-Orbitrap instrument conditions

Gas chromatography method	
Instrumentation	Exactive GC Orbitrap system with Thermo Scientific™ TriPlus™ RSH Autosampler
Column	Thermo Scientific™ TraceGOLD™ TG-5SIL MS (30 m × 0.25 mm i.d. × 0.25 μm) (P/N 26096-1420)
Injector	Split/Splitless (SSL)
Liner	Thermo Scientific™ LinerGOLD™ single taper (P/N 453A1345)
Injector temperature	250 °C
Injector mode	Splitless
Splitless time	2.0 min
Split flow	50.0 mL/min
Purge flow	5.0 mL/min
Injection volume	1 μL
Column flow	1.20 mL/min
Carrier gas and purity	Helium (99.999%)
Vacuum compensation	On
Total run time	35.6 min
GC oven program	40 °C, 1.5 min hold, 25 °C/min to 90 °C, 1.5 min hold, 25 °C/min to 180 °C, 5 °C/min to 280 °C, 10 °C/min to 300 °C, 5 min hold
Orbitrap mass spectrometry method	
Acquisition mode	Full Scan
Filament on delay	5.0 min
MS transfer line temp	280 °C
Ion source temp	250 °C
Electron energy	70 eV
Resolving power (FWHM at <i>m/z</i> 200)	60,000
Scan range	50–550 Da
Ionization	Electron Ionization (EI)

Sample extraction and cleanup

Procedure 1: The EN 15662 citrate buffered QuEChERS method⁵

- Weigh 10 g homogenized sub-sample into a 50 mL extraction tube.
- Prepare recovery spike samples (n=6 for each level) by spiking blank samples before the addition of any extraction solvent and salts with the pesticides mix at 0.005, 0.010, and 0.025 mg/kg.
- Add 10 mL acetonitrile.
- Shake vigorously for 1 min on a vortex mixer.

- Add EN 15662 QuEChERS Extraction salts to the tube, and immediately shake vigorously for 1 min on a vortex mixer.
- Centrifuge at 3000 g for 5 min at room temperature.
- Transfer supernatant (1 mL) into a tube containing 150 mg MgSO₄ and 25 mg PSA.
- Vortex for 1 min and centrifuge samples with 5000 rpm for 5 min.
- Transfer the supernatant into a GC vial for instrumental analysis.

Procedure 2: The AOAC 2007.01 QuEChERS method⁶

- Weigh 15 g homogenized sample into a 50 mL extraction tube.
- Prepare recovery spike samples (n=6 for each level) by spiking blank samples with the pesticide mix at 0.025 mg/kg. Recovery samples were spiked before the addition of the extraction solvent.
- Add 15 mL 1% acetic acid in acetonitrile.
- Shake vigorously for 1 min on a vortex mixer.
- Add 6 g MgSO₄ and 1.5 g of sodium acetate, again mix vigorously for 1 min on a vortex mixer.
- Centrifuge at 5000 rpm for 5 min.
- Transfer supernatant (1 mL) into a tube containing 150 mg MgSO₄ and 50 mg PSA.
- Vortex for 1 min and centrifuge samples with 5000 rpm for 5 min.
- Transfer the supernatant into a GC vial for instrumental analysis.

Solvent standard calibration

- The solvent standard calibration was prepared in a range of 0.001 to 0.1 mg/L.
- Prepare matrix blank (un-spiked) extract by following the above protocol for matrix-matched calibration standards.
- Matrix-matched calibration standards: Prepare the matrix-matched calibration standards as per the procedure given in Thermo Scientific Application Note 73039⁶.
- Inject the final extract as well as matrix-matched standards into the Exactive GC Orbitrap system.

Data acquisition and processing

The data acquisition and processing were carried out using Thermo Scientific™ TraceFinder™ 4.1 software. The data were acquired in full scan mode. For data processing, the identification criteria of the analyte, the mass error (± 5.0 ppm) for the base peak and confirmatory ion, retention time (± 0.10 min), and linearity (>0.99 with back-calculated concentration difference $\pm 20\%$), recovery (70–120%), and precision ($\pm 20\%$) were set for quantitation with user-defined filters per the SANTE guidelines³.

Results and discussion

Sample preparation

Potatoes contain 80% water and have low fat content (0.1%) and protein levels (2%). Most of the remaining matter is the edible starch portion of the plant. Because of this high starch content, it is a challenge to extract the pesticides from the potato. The recovery of spiked analyses in potato was evaluated with both methods by using a pre-spiked sample at 0.025 mg/kg. The results showed that there is no significant difference between methods.

In both methods, the mass accuracy observed was within the acceptance criteria of mass error (± 5 ppm). The EN 15662 method has been utilized for extraction and analysis. Signal enhancement was observed due to matrix interferences when the TIC was compared between solvent standards and the matrix-matched standard equivalent (Figure 1A and 1B) at the concentration of 0.01 mg/kg. The high-resolution extracted ion chromatogram (EIC) filtered out the matrix interferences and provided a symmetrical peak. The high selectivity provided by HRAM is illustrated for chlorpropham (exact mass m/z 213.05510 in Figure 1C). At 15,000 and 30,000 chlorpropham could not be fully mass resolved from a co-eluting matrix co-extractive compound correctly due to high mass error (>5 ppm). At a resolving power of 60,000, the m/z 213.06366 impurity mass was isolated from m/z 213.05462, and m/z 213.05511 for the chlorpropham was observed with a mass accuracy of 0.4 ppm (Figure 1C).

The matrix effect was checked by comparing the peak area of the target analytes at a solvent calibration concentration equivalent to 0.01 mg/kg against the matrix-matched standard at 0.01 mg/kg. Ninety-two analytes showed $<20\%$ matrix effect (defined as acceptable matrix influence on the analyte as per the SANTE guidelines), with 105 other analytes showing $>20\%$ ion enhancement that was observed in the range of 20% to 264%. To obtain accurate results, it is necessary to use matrix-matched standards for the accurate quantitation.

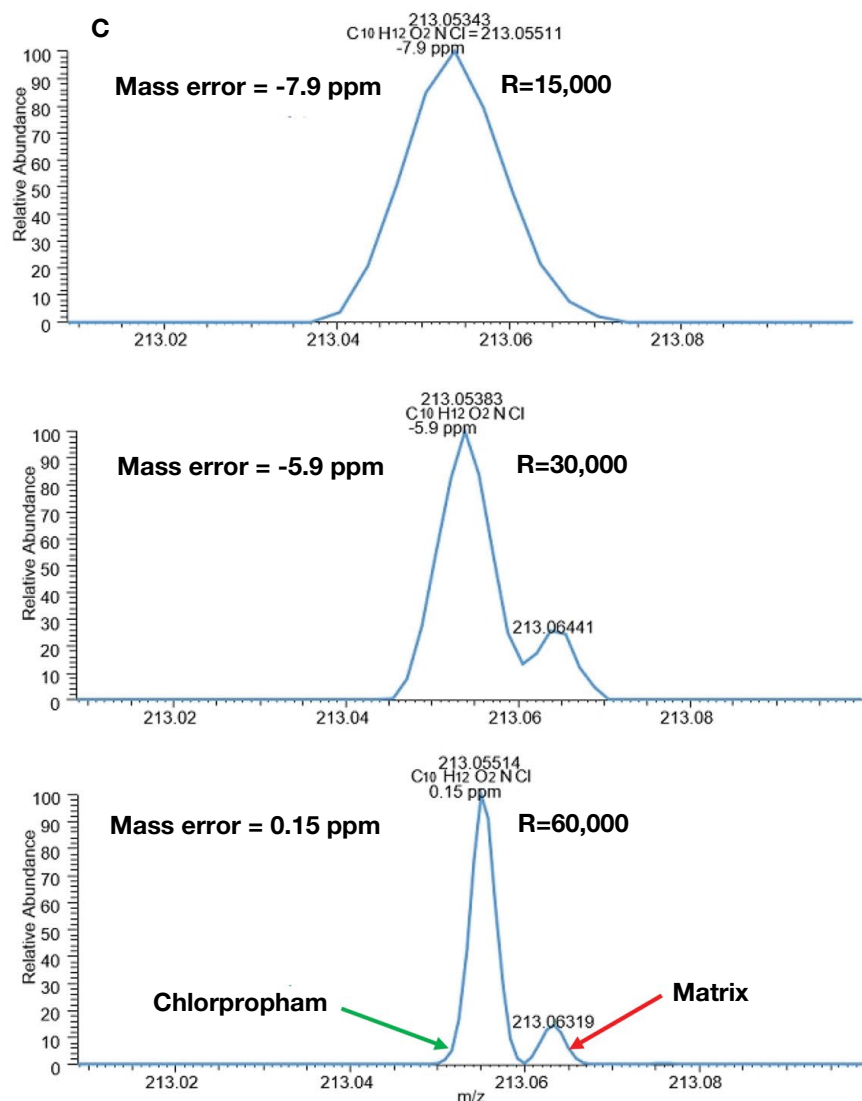
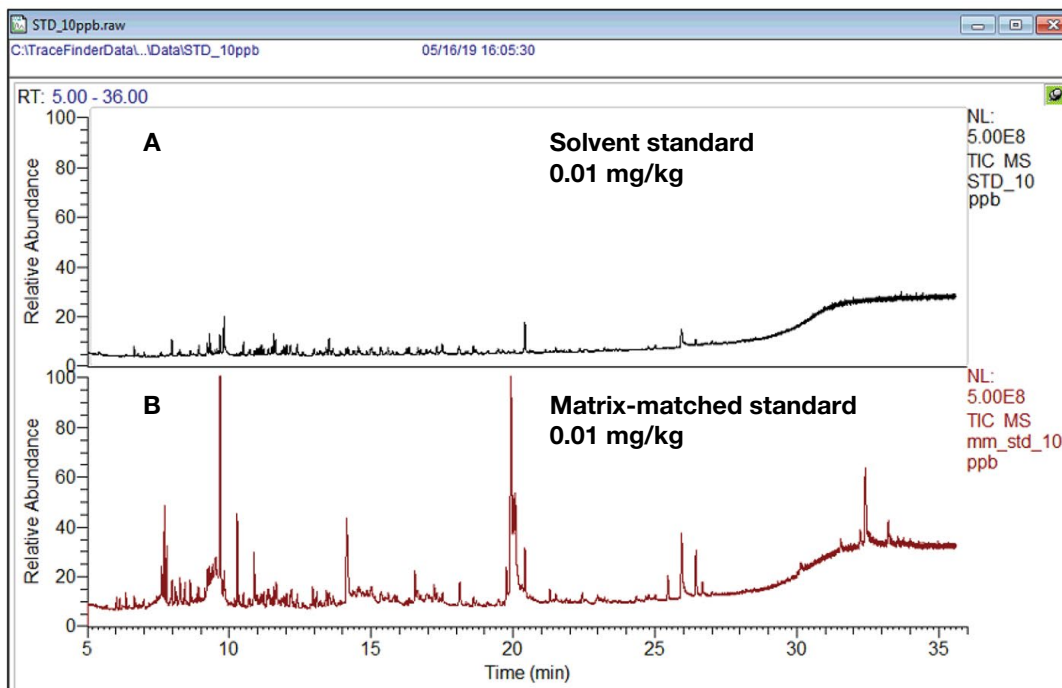


Figure 1. Comparison of total ion chromatogram for solvent standard (A) and matrix-matched standard (B), and the impact of resolving power on chlorpropham ($C_{10}H_{12}ClNO_2$ theoretical mass m/z 213.055116) selectivity at various resolving power settings of 15,000, 30,000, and 60,000 (C)

GC-Orbitrap analysis

Generally, a non-polar solvent is preferred for GC analysis. In this experiment, acetonitrile was used for extraction and as the final solvent prior to GC-MS analysis. The advantage of using acetonitrile as the final solvent is that samples prepared this way can be analyzed on both GC-MS and LC-MS systems without further time-consuming solvent exchange steps. Acetonitrile has a low molecular weight and high polarity. It has a relatively high expansion volume and carries high matrix co-extractives that may disturb the chromatography. By considering these challenges, the splitless injection volume was reduced to 1.0 μ L.

The GC oven program was taken from Thermo Scientific Application Note 10586, which offered excellent chromatographic separation for all target analytes⁷.

Instrument sensitivity

The limit of identification (LOI) was estimated in potato matrix by following retention time criteria and one diagnostic ion with mass accuracy within ± 5 ppm. The IDL was 0.0005 mg/kg for 169 molecules, 0.001 mg/kg for 189 molecules, and 0.0025 for 199 molecules. The total 197 compounds in the range of 0.001–0.005 mg/kg in potato matrix-matched standards were successfully complying the identification and confirmation criteria as per the SANTE/12682/2019 guidelines. All the molecules pass the acceptance criteria for mass accuracy of < 5 ppm. All the parent ions overlap with confirmatory ions at defined retention time (± 0.1 min). An isotopic pattern of chlorpropham showed the chlorinated pattern (m/z 127.01833 and 129.01542) having the chlorinated structure with confirmatory ions m/z 171.00815 and m/z 213.05510 with the mass accuracy 0.26 and 0.07 ppm, respectively, which were within acceptance criteria³ (Figure 2).

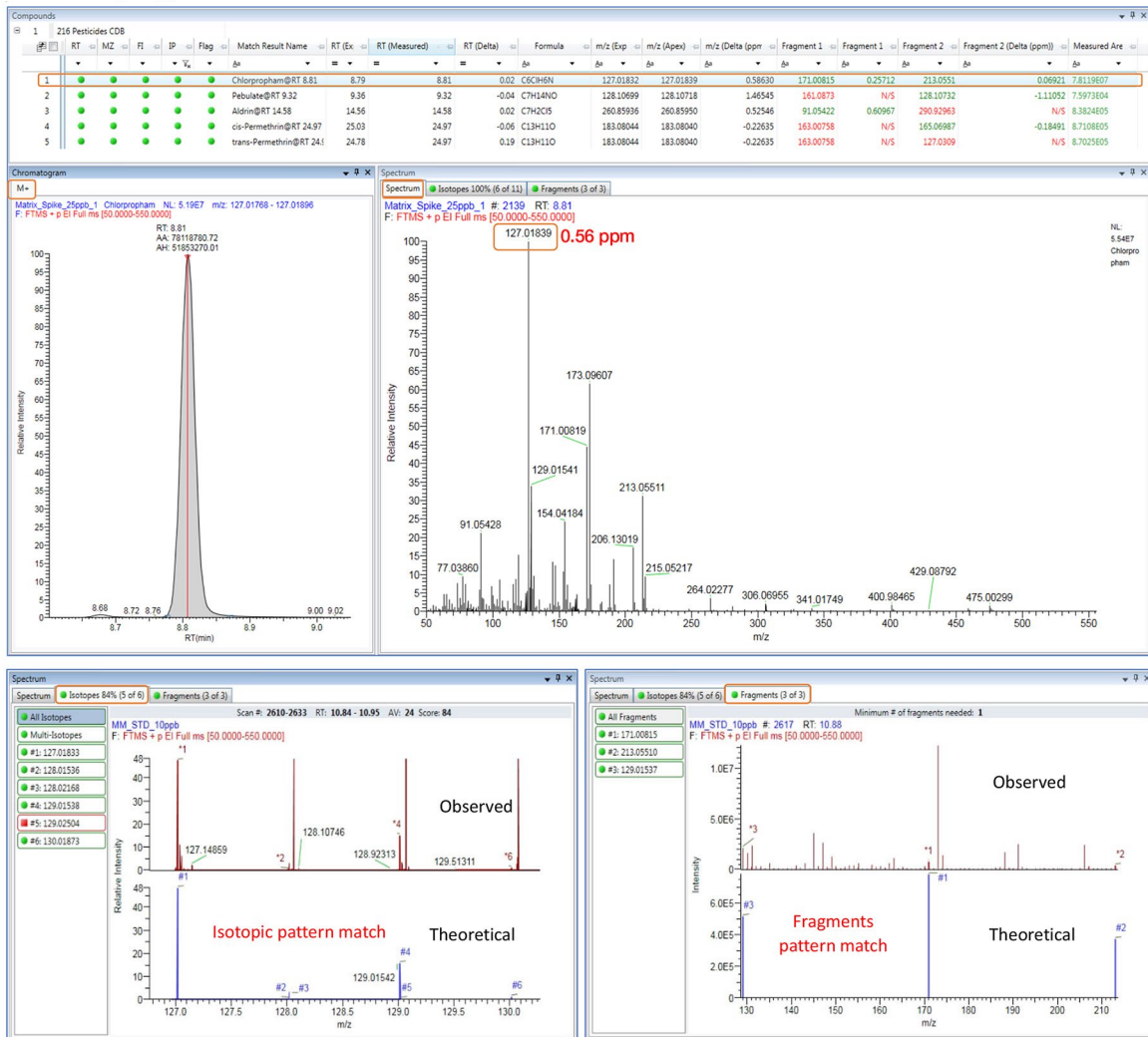


Figure 2. Extracted ion chromatogram along with spectra, isotopic pattern, and fragments for the chlorpropham of the sample post-spiked at 0.01 mg/kg concentration

Targeted quantification

For reliable and confident quantitation, good and symmetrical peak shape is a requirement. An accurate quantitation is reliant upon several factors, one of which is that an acquisition speed should be fast enough to provide at least 12 points across the chromatographic peak. At a resolution of 60K, the Exactive GC Orbitrap system has a scan speed of approximately 7.4 Hz. Because of the high number of scans per peak, better repeatability was achieved.

As a productivity benefit, based on the user-defined criteria, the data was processed automatically with flagging. These flags indicate through color codes whether results pass or fail based on the acceptance criteria given in the processing method. The results that passed under user-defined criteria (SANTE/12682/2019 guidelines) are shown in green (Figure 3).

The parent/base ion (m/z 127.01832) at 0.005 mg/kg was considered the quantitation ion for the chlorpropham. Further, linearity was assessed using matrix-matched standards across a concentration of 0.001–0.1 mg/kg.

The coefficient of determination (R^2) was >0.99 with RF RSD residual values $<20\%$ for all the target analytes in the matrix by plotting the calibration curve.

The matrix effect was checked by injecting the solvent standards linearity and matrix-matched standard linearity (Table 2, page 10). Response enhancement has been observed in matrix-matched standards as compared to solvent standards linearity.

The optimized method was tested for repeatability. A long-term single sequence was assessed for the average mass accuracy by injecting a spiked potato at 0.025 mg/kg level ($n=35$). The mass accuracy observed for molecules along with the isomers between -2.6 and 0.8 ppm without lock mass correction was found to be within an acceptable range (± 5 ppm). The mass error observed for all molecules in spiked potato samples is presented in Figure 4. The ion ratios stability values were also monitored throughout the batch, and all values were within the acceptance criteria ($\pm 30\%$) (Figure 5) and are presented in Table 3, page 15. The ion ratio variation was 3.5% for chlorpropham in one sequence ($n=35$ injections) (Figure 6).

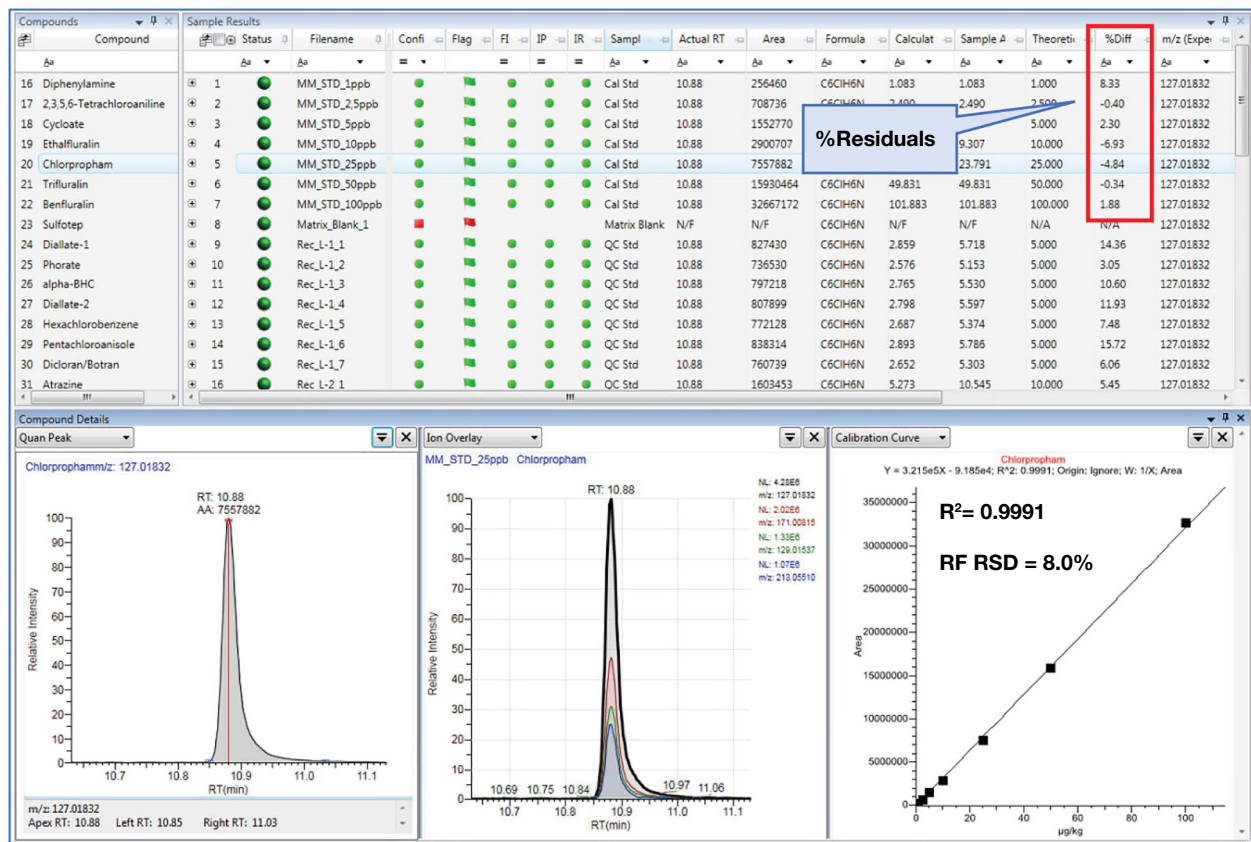


Figure 3. Screening and quantification of chlorpropham based on confirmation criteria

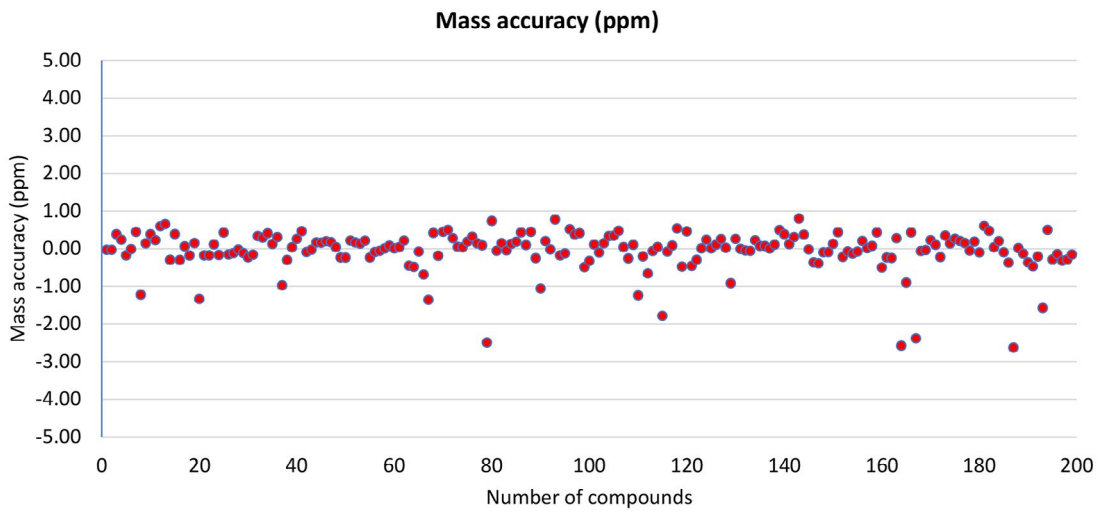


Figure 4. Observed average mass accuracy for all 197 molecules in n=35 replicates of a 0.025 mg/kg pre-spiked potato sample.

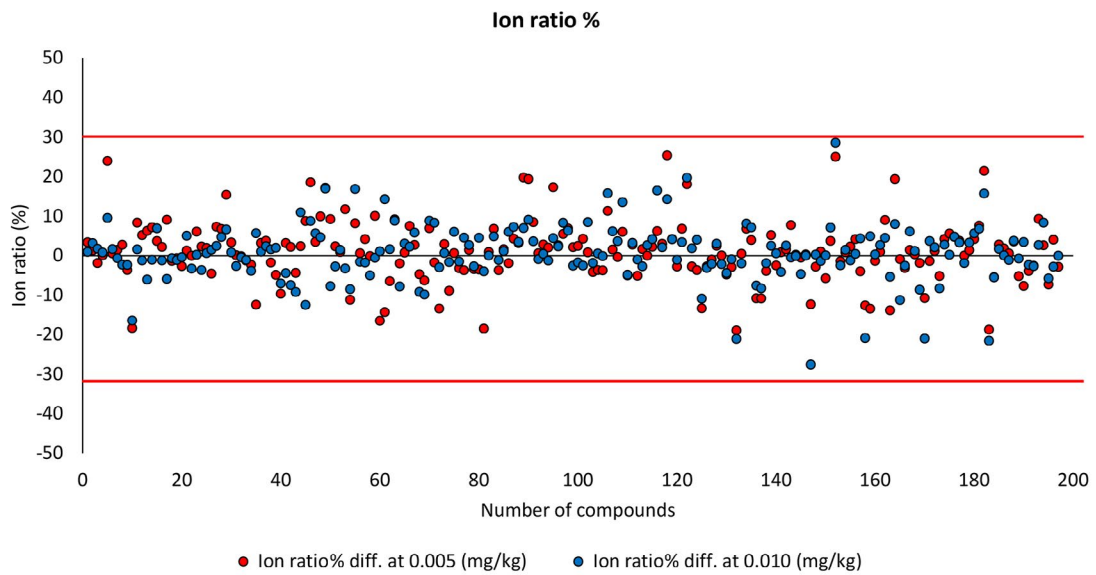


Figure 5. The difference in ion ratio % against the standard reference value in pre-spiked potato matrix at 0.005 mg/kg and 0.01 mg/kg

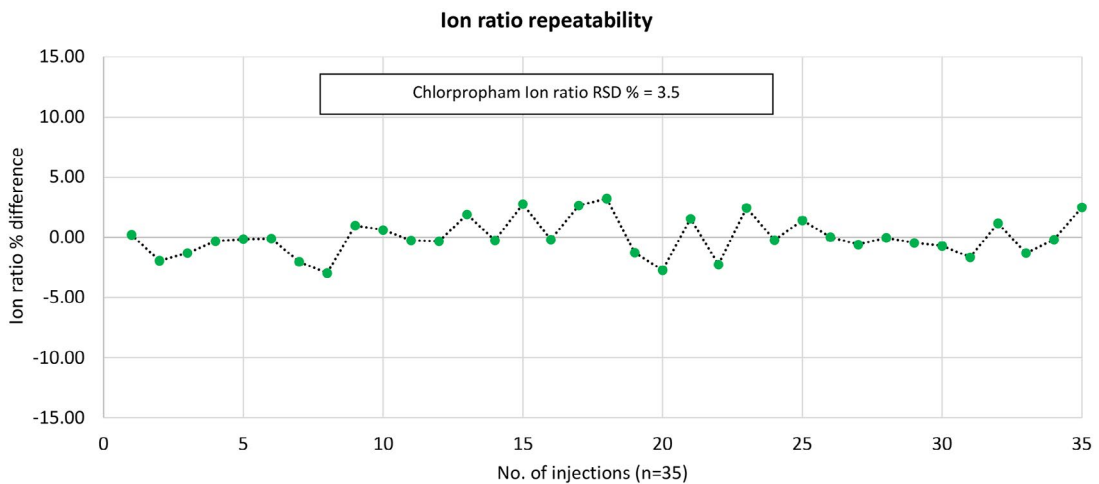


Figure 6. Chlorpropham ion ratio stability across n=35 injections of a potato spiked matrix at 0.025 mg/kg.

To harmonize the results the smallest concentration (0.005 mg/kg) was selected as a limit of quantitation (LOQ) which offered good identification and confirmation criteria.³ The LOQ offered excellent recoveries between 76 and 116% and <13% repeatability (precision). The recovery experiment was carried out at 0.005 (LOQ) and 0.01 (LOQ × 2) mg/kg to demonstrate the method performance

in terms of accuracy and precision (n=6). The average recovery was observed in the range of 76 to 116% with average %RSD of 4.6 and 3.5% for pre-spiked samples at concentration of 0.005 mg/kg and 0.01 mg/kg, respectively (Figures 7 and 8, and Table 2, page 10), which were within acceptance criteria (recovery 70–120% and precision <20%) of the SANTE/12682/2019 guidelines³.

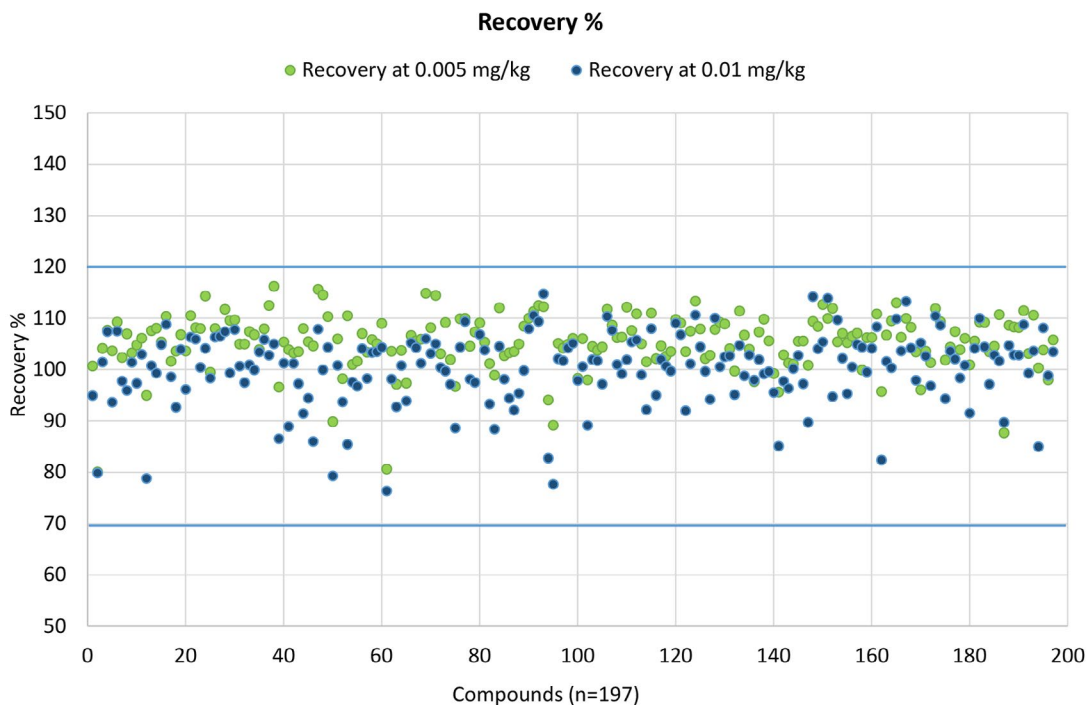


Figure 7. % Recovery of 197 target compounds in potato at 0.005 and 0.01 mg/kg

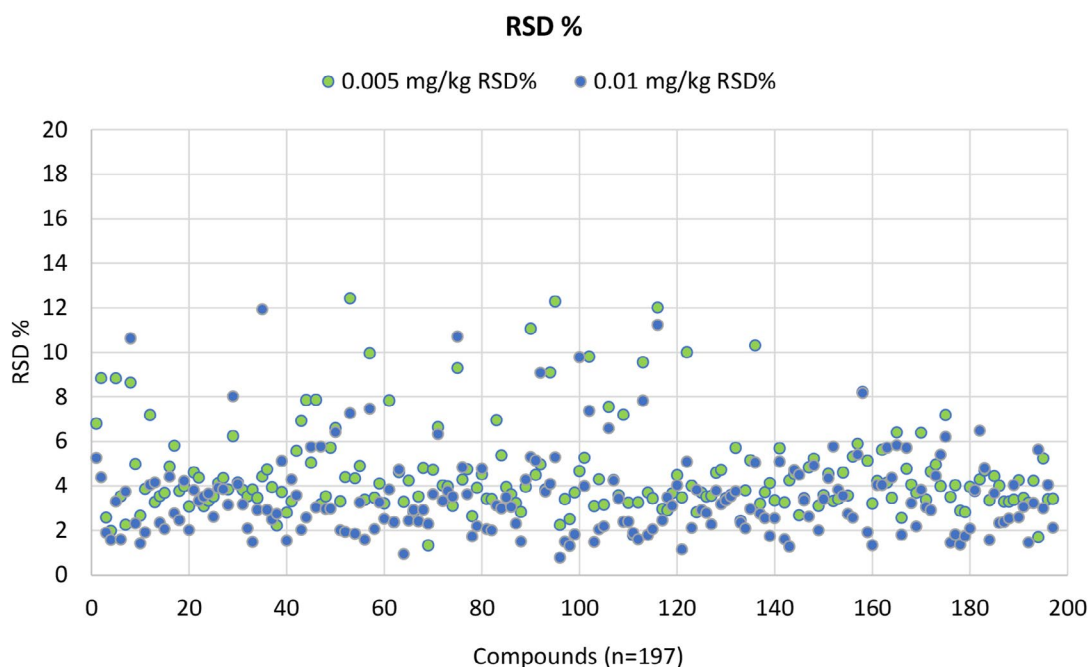


Figure 8. Repeatability (n= 6 injections) as %RSD of peak area for 197 compounds analyzed in potato at 0.005 mg/kg and 0.01 mg/kg, respectively

Conclusion

- The experiments performed demonstrate that the Exactive GC Orbitrap GC-MS high-resolution mass spectrometer, in combination with TraceFinder software, delivers robust and sensitive performance for routine pesticide screening and quantitation in potato in accordance with the SANTE/12682/2019 guidance document.
- The use of the QuEChERS method for extraction, followed by the instrumental analysis, increases the overall throughput and significantly increases the confidence in the results.
- Full scan acquisition allows for easy method setup and enables retrospective data analysis by HRMS.
- The limit of identification (LOI) was observed in the range of 0.0005 mg/kg to 0.0025 mg/kg.
- The observed R^2 value was >0.99 for the plotted calibration curve in the range of 0.001 to 0.1 mg/kg.
- The average recovery was observed in the range of 76 to 116%, with average %RSD of 4.6% and 3.5% for pre-spiked samples at a concentration of 0.005 mg/kg and 0.01 mg/kg, respectively, which were within acceptance criteria (recovery 70–120% and precision $<20\%$) of the SANTE/12682/2019 guidelines.
- The mass error was observed for molecules along with the isomers and metabolites between -2.6 and 0.8 ppm without lock mass correction. The average mass accuracy observed was within ± 1 ppm for 94% of the compounds, whereas 6% (12) compounds were between -1 and -2.6 ppm.

- The ion ratios repeatability values were monitored throughout the batch ($n=35$ injections), and all values were within the acceptance criteria of SANTE guidelines ($\pm 30\%$).
- The method complies with the EU and the FSSAI MRLs requirements.

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Table 2 (part 1). List of pesticides and results (matrix effect, linearity, recovery, and precision at two levels) as per the SANTE guidelines

Compound	RT (min)	R ²	ME#	LOQ (mg/kg)	Recovery at 0.005 (mg/kg)		Recovery at 0.01 (mg/kg)	
					Mean recovery %	RSD%	Mean recovery %	RSD%
2,3,5,6-Tetrachloroaniline	10.72	0.9975	-5.6	0.005	100.73	6.81	94.96	5.26
2-Phenylphenol	10.67	0.9987	1.37	0.005	80.09	8.84	79.86	4.39
4,4'-Dichlorobenzophenone	14.84	0.9976	9.88	0.005	104.13	2.59	101.48	1.9
Acetochlor	13.2	0.9981	14.2	0.005	107.7	1.99	107.41	1.58
Acrinathrin	23.75	0.9941	90.2	0.005	103.67	8.83	93.65	3.3
Alachlor	13.42	0.9966	17.5	0.005	109.28	3.54	107.52	1.59
Aldrin	14.56	0.9982	-4.3	0.005	102.38	2.26	97.79	3.75
Allidochlor	8.23	0.9955	6.96	0.005	106.98	8.65	95.95	10.64
alpha-BHC	11.38	0.9989	14.6	0.005	103.25	4.98	101.41	2.32
Anthraquinone	14.6	0.9925	15.5	0.005	104.81	2.67	97.34	1.43
Atrazine	11.76	0.9976	111	0.005	106.12	3.86	103.01	1.92
Azinphos-ethyl	22.65	0.9925	104	0.005	94.99	7.18	78.83	4.05
Azinphos-methyl	23.77	0.9942	43.8	0.005	107.59	3.27	100.77	4.16
Benfluralin	10.93	0.9936	-1	0.005	108.09	3.55	99.33	2.35
beta-BHC	11.87	0.9984	9.76	0.005	105.39	3.69	104.88	2.07
Bifenthrin	21.51	0.995	47.1	0.005	110.39	4.87	108.83	4.42
Bromfenvinphos-ethyl	16.77	0.9997	14.1	0.005	101.63	5.81	98.6	2.77
Bromfenvinphos-methyl	15.61	0.9941	50.1	0.005	103.6	3.78	92.66	2.48
Bromophos-ethyl	16.19	0.9971	20.3	0.005	106.81	4	103.93	4.23
Bromophos-methyl	15.02	0.9957	27.2	0.005	103.63	3.09	96.16	2.02
Bromopropylate	21.51	0.9946	43.7	0.005	110.52	4.62	106.34	3.81
Bupirimate	17.53	0.9958	32.5	0.005	108.17	4.37	105.94	3.33
Carbophenothion	19.46	0.9924	33.5	0.005	108.02	3.1	100.46	3.52
Carfentrazone-ethyl	19.4	0.9945	28.8	0.005	114.36	3.33	104.13	3.66
Chlorbenside	16.24	0.9979	17.4	0.005	99.48	3.52	98.37	2.62
Chlorfenapyr	17.83	0.9983	20.3	0.005	108.03	4.14	106.33	3.91
Chlorfenson/Ovex	16.96	0.9983	10.6	0.005	106.89	4.36	106.48	3.85
Chlorobenzilate	18.35	0.9958	39.8	0.005	111.75	3.84	107.42	3.15
Chloroneb	9.69	0.9968	-13	0.005	109.6	6.25	99.36	8.03
Chlorpropham	10.88	0.9992	32.7	0.005	109.71	4.16	107.8	4.08
Chlorpyriphos-ethyl	14.42	0.9964	18.9	0.005	104.99	3.82	100.71	3.18
Chlorpyriphos-methyl	13.25	0.9929	33.8	0.005	105	3.53	97.5	2.1
Chlorthal-dimethyl	14.57	0.9935	-3.2	0.005	107.42	3.84	100.92	1.49
Chlorthiophos	18.71	0.992	24.3	0.005	106.81	3.46	99.96	2.93
cis-1,2,3,6-Tetrahydrophthalimide	9.62	0.9947	62.9	0.005	103.91	4.42	103.46	11.94
cis-Chlordane	16.66	0.9984	-1	0.005	107.96	4.75	105.85	2.93
cis-Chlorfenvinphos	16.77	0.9914	57.2	0.005	112.51	3.94	102.8	2.5

* Indicates the MRL has been taken as the LOQ. ME# indicates matrix effect.

Compounds without MRL values were considered to have the default MRL, i.e., 0.01 mg/kg.

Table 2 (part 2). List of pesticides and results (matrix effect, linearity, recovery, and precision at two levels) as per the SANTE guidelines

Compound	RT (min)	R ²	ME [#]	LOQ (mg/kg)	Recovery at 0.005 (mg/kg)		Recovery at 0.01 (mg/kg)	
					Mean recovery %	RSD%	Mean recovery %	RSD%
<i>cis</i> -Nonachlor	18.6	0.9971	5.11	0.005	116.22	2.23	105.02	2.75
<i>cis</i> -Permethrin	25.03	0.9907	39.5	0.005	96.59	3.72	86.55	5.11
Clomazone/Dimethazone	11.85	0.997	19.2	0.005	105.41	2.81	101.26	1.55
Coumaphos	24.95	0.9917	60.2	0.005	103.91	3.3	88.93	4.3
Cycloate	10.73	0.9991	-2.9	0.005	103.25	5.57	101.19	3.58
Cyfluthrin-1	25.84	0.9981	40.4	0.005	103.51	6.93	97.26	2.03
Cyfluthrin-2	26.04	0.9957	46.3	0.005	108.03	7.86	91.46	2.59
Cyfluthrin-3	26.14	0.9964	43.2	0.005	105.45	5.04	94.45	5.76
Cyfluthrin-4	26.23	0.9944	29.8	0.005	104.62	7.87	86.03	3.04
Cypermethrin-1	26.43	0.9975	49.6	0.005	115.65	3.17	107.89	5.77
Cypermethrin-2	26.64	0.9962	48.2	0.005	114.57	3.53	99.99	2.97
Cypermethrin-3	26.73	0.9963	43.9	0.005	110.36	5.72	104.33	2.99
Cypermethrin-4	26.82	0.9956	43.2	0.005	89.87	6.61	79.3	6.42
Cyprodinil	15.36	0.996	13.2	0.005	105.99	3.3	100.82	2.02
delta-BHC	12.61	0.9976	16.2	0.005	98.26	4.39	93.71	1.94
Deltamethrin	29.46	0.9941	22.9	0.005	110.51	12.43	85.47	7.26
Diallate-1	11.22	0.9976	0.84	0.005	101.01	4.35	97.59	1.86
Diallate-2	11.39	0.9972	2.23	0.005	101.64	4.89	96.75	3.26
Diazinone	12.13	0.9971	12.2	0.005	107.03	3.37	104.09	1.59
Diclobenil	8.63	0.9979	-15	0.005	103.4	9.96	98.28	7.47
Dicloran/Botran	11.61	0.9961	24.8	0.005	105.85	3.47	103.35	2.07
Dieldrin	17.46	0.9973	-2.7	0.005	105.07	4.11	103.47	3.27
Diphenamid	15.01	0.9985	12.9	0.005	109.02	3.22	104.32	2.54
Diphenylamine	10.67	0.9977	7.85	0.005	80.63	7.83	76.39	3.84
Disulfoton	12.4	0.9944	29.2	0.005	103.59	2.37	98.19	2.38
Edifenphos	19.52	0.9965	45.5	0.005	97.16	4.63	92.75	4.74
Endosulfan ether	13.01	0.9976	-7.5	0.005	103.73	3.29	100.81	0.95
Endosulfan sulfate	19.64	0.9974	8.59	0.005	97.38	4.24	93.91	2.45
Endosulfan- α	16.66	0.9962	-4	0.005	106.66	2.84	105.23	2.93
Endosulfan- β	18.42	0.9967	9.6	0.005	105.62	3.52	104.27	2.42
Endrin	18.09	0.9938	9.82	0.005	105.93	4.81	101.22	2.93
Endrin ketone	21.15	0.9954	8.94	0.005	114.89	1.34	106.02	2.3
EPN	21.44	0.9955	48.9	0.005	108.17	4.73	103.16	3.63
Esfenvalerate	28.48	0.9935	20.7	0.005	114.47	6.65	105.02	6.34
Ethalfuralin	10.75	0.9936	14.2	0.005	103.11	4.02	100.43	3.32
Ethion	18.61	0.9971	37.5	0.005	109.2	3.97	99.74	3.78
Etofenprox	27.02	0.9948	27.5	0.005	101.96	3.11	97.13	3.52

* Indicates the MRL has been taken as the LOQ. ME# indicates matrix effect.
 Compounds without MRL values were considered to have the default MRL, i.e., 0.01 mg/kg.

Table 2 (part 3). List of pesticides and results (matrix effect, linearity, recovery, and precision at two levels) as per the SANTE guidelines

Compound	RT (min)	R ²	ME [#]	LOQ (mg/kg)	Recovery at 0.005 (mg/kg)		Recovery at 0.01 (mg/kg)	
					Mean recovery %	RSD%	Mean recovery %	RSD%
Etridiazole	9.32	0.995	3.04	0.005	96.7	9.3	88.64	10.71
Fenamiphos	16.81	0.9925	76.1	0.005	109.92	4.29	104.35	4.84
Fenarimol	23.54	0.9922	41	0.005	110.01	4.76	109.37	3.63
Fenchlorphos	13.66	0.9942	22.5	0.005	104.58	2.65	98.16	1.73
Fenitrothion	14	0.9985	50.3	0.005	107.34	3.91	97.5	2.19
Fenpropathrin	21.81	0.9924	43.6	0.005	109.11	4.53	106.84	4.79
Fenson	14.97	0.9937	4.79	0.005	105.38	3.42	103.81	2.07
Fenthion	14.52	0.9938	23.3	0.005	101.17	3.41	93.32	2.01
Fenvalerate	28.08	0.9935	48.2	0.005	98.94	6.96	88.42	3.14
Fipronil*	15.47	0.9919	57.7	0.005	112.06	5.38	104.53	3
Fluazifop butyl	18.11	0.9925	44.9	0.005	102.73	3.95	98.15	3.49
Fluchloralin	12.17	0.9941	10.5	0.005	103.29	3.63	94.4	3.07
Flucythrinate-1	26.77	0.9939	53.6	0.005	103.53	3.23	92.12	2.31
Flucythrinate-2	27.15	0.9988	38.9	0.005	105.01	2.84	95.39	1.52
Fluquinconazole	24.97	0.9938	39.2	0.005	108.48	3.96	99.83	4.29
Fluridone	27.4	0.9965	100	0.005	110.03	11.06	107.96	5.3
Flusilazole	17.5	0.9976	109	0.005	111.33	4.5	110.55	5.14
Flutolanil	16.94	0.9942	64.4	0.005	112.5	4.97	109.38	9.09
Flutriafol	16.76	0.9905	67	0.005	112.26	3.84	114.78	3.74
Fluvalinate-1	28.37	0.9974	44	0.005	94.08	9.1	82.74	4.09
Fluvalinate-2	28.52	0.998	39.9	0.005	89.18	12.29	77.68	5.29
Fonofos	12.15	0.9979	11.6	0.005	105.14	2.25	102.11	0.79
gamma-BHC	12.02	0.998	-1	0.005	104.2	3.41	101.73	1.5
Heptachlor	13.65	0.9976	5.7	0.005	104.95	2.52	104.23	1.32
Heptachlor epoxide	15.6	0.9929	0.9	0.005	106.09	3.71	105.15	1.82
Hexachlorobenzene	11.48	0.9954	-2.8	0.005	98.35	4.67	97.9	9.79
Iodofenfos	16.95	0.9975	45.8	0.005	106.04	5.27	100.61	3.99
Iprodione	21.2	0.9977	47.2	0.005	98.02	9.81	89.15	7.37
Isazophos	12.41	0.9961	1.43	0.005	104.51	3.1	101.96	1.49
Isodrin	15.34	0.9935	-6.5	0.005	103.76	4.31	101.74	2.06
Isopropalin	15.13	0.9963	22.4	0.005	104.42	3.16	97.18	2.19
lambda-Cyhalothrin	23.34	0.9948	75.6	0.005	111.77	7.55	110.37	6.6
Lenacil	19.69	0.996	65.3	0.005	108.69	4.27	107.68	4.25
Leptophos	22.6	0.9943	37.6	0.005	106.23	3.58	101	3.43
Linuron	14.17	0.9979	87.9	0.005	106.34	7.2	99.2	2.39
Malathion	14.2	0.9967	83.6	0.005	112.18	3.24	101.93	2.4
Metalaxyl	13.57	0.9987	15.3	0.005	107.64	1.8	105.41	1.89

* Indicates the MRL has been taken as the LOQ. ME# indicates matrix effect.
Compounds without MRL values were considered to have the default MRL, i.e., 0.01 mg/kg.

Table 2 (part 4). List of pesticides and results (matrix effect, linearity, recovery, and precision at two levels) as per the SANTE guidelines

Compound	RT (min)	R ²	ME [#]	LOQ (mg/kg)	Recovery at 0.005 (mg/kg)		Recovery at 0.01 (mg/kg)	
					Mean recovery %	RSD%	Mean recovery %	RSD%
Metazachlor	15.36	0.9907	15.1	0.005	110.88	3.26	105.77	1.6
Methacrifos	9.59	0.9973	18	0.005	105.05	9.56	99.02	7.83
Methoxychlor	21.7	0.9946	26.4	0.005	101.53	3.71	92.2	1.8
Metolachlor	14.37	0.9978	22.6	0.005	111	3.44	108	2.06
Mevinphos	9.1	0.9976	72.4	0.005	102.15	12.02	95	11.23
MGK 264 Isomer A	15.05	0.9943	9.56	0.005	104.63	2.96	101.98	2.46
MGK 264 Isomer B	15.4	0.994	11.5	0.005	102.65	2.91	100.73	3.48
Mirex	23.23	0.9981	-1.5	0.005	103.53	3.67	99.72	3.11
Myclobutanil	17.42	0.991	59.9	0.005	109.77	4.49	109.07	4.03
N-(2,4-Dimethylphenyl) formamide	12.4	0.9951	19.3	0.005	109.1	3.48	106.82	1.15
Nitralin	20.62	0.9985	76.6	0.005	103.51	10	92.01	5.08
Nitrofen	18.05	0.9962	40.7	0.005	107.51	4.01	101.11	2.12
Norflurazon	19.52	0.9976	49.5	0.005	113.35	2.82	110.65	3.82
o,p-DDD	17.52	0.9936	7.83	0.005	107.95	3.7	104.43	2.92
o,p-DDE	16.35	0.9982	-6.2	0.005	102.16	3.52	99.69	2.79
o,p-DDT	18.69	0.9921	5.4	0.005	102.81	3.56	94.19	2.28
Oxadiazone	17.34	0.9926	18.6	0.005	107.84	4.6	110.05	3.79
Oxyfluorfen	17.52	0.9973	56	0.005	109.23	4.73	100.56	3.19
p,p-DDD	18.63	0.9922	16.6	0.005	108.91	3.44	102.52	3.35
p,p-DDE	17.32	0.9961	-0.7	0.005	104.09	3.6	102.71	3.54
p,p-DDT	19.83	0.9938	23.5	0.005	99.74	5.72	95.13	3.76
Paclobutrazol	16.42	0.9992	19.7	0.005	111.42	2.44	104.73	2.35
Parathion	14.61	0.9993	42.2	0.005	106.73	3.79	98.79	2.1
Parathion-methyl	13.39	0.9927	40.6	0.005	103.99	5.15	102.87	2.97
Pebulate	9.36	0.9953	-2	0.005	97.66	10.32	97.98	5.05
Penconazole	15.49	0.9956	38.8	0.005	107.35	3.21	101.96	2.76
Pendimethalin	15.32	0.9979	22.7	0.005	109.84	3.72	99.17	2.56
Pentachloroaniline	13	0.9977	-1.1	0.005	105.61	4.14	99.64	1.75
Pentachloroanisole	11.56	0.9957	-4.3	0.005	99.24	3.35	95.55	2.56
Pentachlorobenzene	9.87	0.9981	-15	0.005	95.65	5.69	85.11	5.09
Pentachlorobenzonitrile	12.02	0.994	-2	0.005	102.89	3.26	97.76	1.61
Pentachlorothioanisole	14.19	0.9977	0.04	0.005	101.29	4.26	96.35	1.28
Phenothrin	22.52	0.9918	51.8	0.005	101.13	4.56	100.22	4.72
Phorate	11.23	0.9939	12.8	0.005	105.54	2.69	102.79	4.5
Phosalone	22.56	0.9939	46.6	0.005	105.59	3.44	97.25	3.35
Phosmet	21.3	0.9951	79.5	0.005	100.84	4.84	89.74	2.63
Piperonyl butoxide	20.61	0.998	69.7	0.005	109.42	5.23	114.22	4.92

* Indicates the MRL has been taken as the LOQ. ME# indicates matrix effect.
 Compounds without MRL values were considered to have the default MRL, i.e., 0.01 mg/kg.

Table 2 (part 5). List of pesticides and results (matrix effect, linearity, recovery, and precision at two levels) as per the SANTE guidelines

Compound	RT (min)	R ²	ME [#]	LOQ (mg/kg)	Recovery at 0.005 (mg/kg)		Recovery at 0.01 (mg/kg)	
					Mean recovery %	RSD%	Mean recovery %	RSD%
Pirimiphos methyl	13.92	0.9975	17.5	0.005	108.41	3.11	104.05	2.01
Pirimiphos-ethyl	14.98	0.9924	37.5	0.005	112.7	3.6	105.39	3.41
Pretilachlor	17.1	0.999	30.4	0.005	110.01	4.55	113.89	4.35
Prochloraz	25.09	0.993	264	0.005	111.95	3.32	94.73	5.77
Procymidone	15.89	0.9936	2.99	0.005	105.42	3.42	109.71	3.83
Prodiamine	13.98	0.9939	9.42	0.005	107.07	4.61	102.22	3.56
Profenofos	17.17	0.9985	39.5	0.005	105.28	3.57	95.31	2.75
Profluralin	11.92	0.9986	8.27	0.005	106.51	5.32	100.54	2.58
Propachlor	10.48	0.9953	11.5	0.005	107.12	5.9	104.93	5.41
Propargite	20.36	0.997	80.1	0.005	99.93	8.22	104.36	8.17
Propisochlor	12.43	0.998	87.7	0.005	106.23	5.13	99.55	1.93
Propyzamide	12.13	0.9945	14.8	0.005	106.26	3.21	104.16	1.35
Prothiofos	17.05	0.9926	30.4	0.005	110.87	4.23	108.34	4.04
Pyraclofos	24.12	0.994	79	0.005	95.75	5.62	82.42	4.05
Pyrazophos	23.62	0.9905	53.9	0.005	106.73	4.16	101.6	5.72
Pyridaben	25.01	0.9979	42.9	0.005	109.52	3.46	100.36	4.37
Pyridaphenthion	21.11	0.9958	55	0.005	113.03	6.41	109.89	5.83
Pyrimethanil	12.29	0.9971	9.49	0.005	106.32	2.58	103.64	1.81
Pyriproxyfen	22.94	0.9954	40.6	0.005	110	4.77	113.29	5.72
Quinalphos	15.78	0.9951	34.5	0.005	108.26	4.06	104.19	3.23
Quintozene	11.94	0.9976	14.6	0.005	103.45	3.69	97.93	2.18
Resmethrin	20.7	0.9945	74.2	0.005	96.06	6.4	105.22	3.82
Sulfotep	10.98	0.9932	15.6	0.005	103.62	3.38	102.65	3.06
Sulprofos	19.13	0.9955	33.5	0.005	101.36	4.65	96.84	2.92
Tebuconazole	20.28	0.9972	127	0.005	111.89	4.96	110.45	4.46
Tebufenpyrad	22	0.998	48.4	0.005	109.35	3.99	108.62	5.4
Tecnazene	10.4	0.9956	1.84	0.005	101.87	7.19	94.32	6.2
Tefluthrin	12.4	0.9946	1.37	0.005	104.38	3.51	103.6	1.47
Terbacil	12.43	0.9962	60	0.005	107.42	4.04	102.06	1.82
Terbufos	12.04	0.9958	15.9	0.005	103.87	2.89	98.39	1.37
Terbutylazine	12.04	0.9979	13.5	0.005	106.09	2.83	100.85	1.75
Tetrachlorvinphos	16.38	0.9959	40.2	0.005	100.92	3.96	91.51	2.09
Tetradifon	22.36	0.9918	8.2	0.005	105.6	3.76	104.15	3.81
Tetramethrin-1	21.29	0.995	66.4	0.005	109.3	4.29	110	6.49
Tetramethrin-2	21.58	0.9969	58.3	0.005	109.22	4.69	104.45	4.8
Tolclofos-methyl	13.42	0.9944	5.78	0.005	103.47	3.35	97.18	1.57
trans-Chlordane	16.27	0.9978	-4.9	0.005	104.55	4.44	102.87	3.68

* Indicates the MRL has been taken as the LOQ. ME# indicates matrix effect.
Compounds without MRL values were considered to have the default MRL, i.e., 0.01 mg/kg.

Table 2 (part 6). List of pesticides and results (matrix effect, linearity, recovery, and precision at two levels) as per the SANTE guidelines

Compound	RT (min)	R ²	ME [#]	LOQ (mg/kg)	Recovery at 0.005 (mg/kg)		Recovery at 0.01 (mg/kg)	
					Mean recovery %	RSD%	Mean recovery %	RSD%
<i>trans</i> -Chlorfenvinphos	15.6	0.9983	48.5	0.005	110.71	4.01	101.67	2.34
Transfluthrin	13.45	0.9951	4.8	0.005	87.7	3.3	89.74	2.39
<i>trans</i> -Nonachlor	16.75	0.9937	2.12	0.005	108.65	3.29	104.72	2.55
<i>trans</i> -Permethrin	24.78	0.9952	42.7	0.005	108.31	3.38	102.88	4.05
Triadimefon	14.7	0.9957	30.7	0.005	108.21	4.26	102.84	2.6
Triadimenol	15.89	0.9947	65.8	0.005	111.49	3.45	108.78	3.07
Triallate	12.58	0.9903	-1.6	0.005	103.16	3.24	99.3	1.47
Triazophos	19.09	0.9958	58.1	0.005	110.61	4.24	103.64	3.23
Tricyclazole	17.13	0.9947	118	0.005	100.35	1.7	85	5.63
Triflumizole	15.95	0.9976	53.7	0.005	103.81	5.24	108.12	3
Trifluralin	10.88	0.9979	19.6	0.005	98.02	3.4	98.84	4.05
Vinclozolin	13.33	0.9979	4.79	0.005	105.79	3.42	103.49	2.12

* Indicates the MRL has been taken as the LOQ. ME# indicates matrix effect.

Compounds without MRL values were considered to have the default MRL, i.e., 0.01 mg/kg.

Table 3 (part 1). List of pesticides with ion ratio and mass accuracy summary

Compound	Target ion ratio (Standard)	Ion ratio % at 0.005 (mg/kg)	Ion ratio% diff. at 0.005 (mg/kg)	Ion ratio % at 0.01 (mg/kg)	Ion ratio % diff. at 0.010 (mg/kg)
2,3,5,6-Tetrachloroaniline	78.01	75.38	3.37	77.27	0.95
2-Phenylphenol	12.04	11.88	1.31	11.66	3.15
4,4'-Dichlorobenzophenone	24.34	24.80	-1.86	23.92	1.75
Acetochlor	66.06	66.03	0.06	65.53	0.82
Acrinathrin	8.83	6.71	23.98	7.99	9.56
Alachlor	85.52	85.02	0.58	84.10	1.65
Aldrin	39.49	38.92	1.46	39.74	-0.62
Allidochlor	73.77	71.75	2.73	75.45	-2.28
alpha-BHC	16.05	16.63	-3.63	16.42	-2.35
Anthraquinone	103.03	121.91	-18.32	119.96	-16.43
Atrazine	32.45	29.74	8.34	31.94	1.58
Azinphos-ethyl	27.74	26.30	5.18	28.07	-1.20
Azinphos-methyl	31.62	29.63	6.29	33.53	-6.04
Benfluralin	38.25	35.51	7.18	38.66	-1.06
beta-BHC	45.50	43.82	3.69	42.36	6.90
Bifenthrin	46.97	45.94	2.20	47.53	-1.19
Bromfenvinphos-ethyl	37.32	33.93	9.09	39.51	-5.87
Bromfenvinphos-methyl	62.34	63.17	-1.33	62.75	-0.65
Bromophos-ethyl	63.43	63.84	-0.65	64.14	-1.12
Bromophos-methyl	74.39	76.38	-2.66	74.71	-0.42

Table 3 (part 2). List of pesticides with ion ratio and mass accuracy summary

Compound	Target ion ratio (Standard)	Ion ratio % at 0.005 (mg/kg)	Ion ratio% diff. at 0.005 (mg/kg)	Ion ratio % at 0.01 (mg/kg)	Ion ratio % diff. at 0.010 (mg/kg)
Bromopropylate	98.62	97.35	1.29	93.67	5.02
Bupirimate	73.67	73.69	-0.03	76.08	-3.28
Carbophenothion	24.35	22.88	6.07	24.30	0.23
Carfentrazone-ethyl	63.73	62.27	2.29	66.06	-3.65
Chlorbenside	32.52	31.88	1.95	32.31	0.65
Chlorfenapyr	76.56	80.04	-4.54	75.44	1.46
Chlorfenson/Ovex	33.29	30.86	7.32	32.48	2.45
Chlorobenzilate	69.05	64.39	6.75	65.79	4.72
Chloroneb	34.28	29.00	15.39	32.00	6.64
Chlorpropham	46.39	44.84	3.35	46.01	0.83
Chlorpyriphos-ethyl	61.50	61.45	0.09	63.12	-2.63
Chlorpyriphos-methyl	65.57	65.68	-0.16	65.86	-0.45
Chlorthal-dimethyl	79.43	80.41	-1.23	80.29	-1.08
Chlorthiophos	42.80	43.79	-2.30	44.44	-3.83
<i>Cis</i> -1,2,3,6-Tetrahydrophthalimide	62.51	70.26	-12.40	58.96	5.68
<i>cis</i> -Chlordane	97.06	93.95	3.21	96.07	1.02
<i>cis</i> -Chlorfenvinphos	63.73	61.34	3.75	62.20	2.40
<i>cis</i> -Nonachlor	89.37	90.94	-1.75	87.94	1.60
<i>cis</i> -Permethrin	21.10	22.13	-4.87	20.69	1.97
Clomazone/Dimethazone	91.44	100.25	-9.64	97.85	-7.01
Coumaphos	69.31	67.10	3.20	72.37	-4.41
Cycloate	14.63	14.31	2.19	15.71	-7.44
Cyfluthrin-1	90.82	94.76	-4.34	99.17	-9.20
Cyfluthrin-2	94.26	92.00	2.40	83.93	10.96
Cyfluthrin-3	89.87	82.00	8.76	101.07	-12.46
Cyfluthrin-4	71.22	58.00	18.56	64.97	8.77
Cypermethrin-1	97.63	94.25	3.47	92.14	5.63
Cypermethrin-2	93.16	83.91	9.92	88.83	4.65
Cypermethrin-3	323.07	267.56	17.18	268.28	16.96
Cypermethrin-4	89.98	81.68	9.23	96.92	-7.72
Cyprodinil	24.47	23.89	2.37	25.13	-2.73
delta-BHC	36.74	36.39	0.93	36.20	1.46
Deltamethrin	54.40	48.01	11.74	56.16	-3.24
Diallate-1	50.10	55.70	-11.17	54.35	-8.48
Diallate-2	35.12	32.24	8.20	29.19	16.87
Diazinone	81.77	81.28	0.60	83.03	-1.54
Diclobenil	64.02	61.36	4.16	65.15	-1.76
Dicloran/Botran	93.18	93.29	-0.11	97.83	-4.98
Dieldrin	64.57	58.07	10.07	64.91	-0.54

Table 3 (part 3). List of pesticides with ion ratio and mass accuracy summary

Compound	Target ion ratio (Standard)	Ion ratio % at 0.005 (mg/kg)	Ion ratio% diff. at 0.005 (mg/kg)	Ion ratio % at 0.01 (mg/kg)	Ion ratio % diff. at 0.010 (mg/kg)
Diphenamid	41.67	48.53	-16.46	41.22	1.09
Diphenylamine	14.00	16.00	-14.29	12.00	14.29
Disulfoton	93.52	99.57	-6.47	92.01	1.62
Edifenphos	43.98	39.96	9.16	40.10	8.83
Endosulfan ether	127.16	129.67	-1.97	137.08	-7.80
Endosulfan sulfate	81.19	80.56	0.78	78.72	3.05
Endosulfan- α	53.85	49.80	7.51	52.64	2.25
Endosulfan- β	69.26	67.38	2.71	65.22	5.82
Endrin	89.80	94.02	-4.69	98.02	-9.15
Endrin ketone	93.49	99.33	-6.25	102.68	-9.83
EPN	66.24	61.64	6.95	60.39	8.84
Esfenvalerate	31.71	32.26	-1.71	29.08	8.30
Ethalfuralin	43.75	49.61	-13.41	45.04	-2.94
Ethion	26.33	25.56	2.92	26.15	0.69
Etofenprox	28.78	31.33	-8.86	29.24	-1.61
Etridiazole	84.33	83.79	0.64	79.26	6.01
Fenamiphos	63.18	65.18	-3.17	64.11	-1.48
Fenarimol	29.90	31.01	-3.70	28.54	4.55
Fenchlorphos	65.69	64.74	1.45	63.93	2.69
Fenitrothion	77.09	79.72	-3.41	79.17	-2.70
Fenpropathrin	28.47	29.46	-3.49	27.18	4.52
Fenson	117.36	139.02	-18.45	122.01	-3.96
Fenthion	28.51	28.24	0.94	28.50	0.05
Fenvalerate	52.68	49.11	6.78	50.12	4.86
Fipronil	66.58	69.03	-3.68	67.32	-1.12
Fluazifop butyl	54.39	53.50	1.62	53.77	1.14
Fluchloralin	58.84	59.97	-1.91	55.30	6.02
Flucythrinate-1	54.14	51.82	4.29	50.20	7.27
Flucythrinate-2	53.53	51.83	3.19	51.67	3.48
Fluquinconazole	8.68	6.96	19.77	8.07	6.96
Fluridone	12.10	9.76	19.37	11.00	9.09
Flusilazole	26.22	24.00	8.45	25.26	3.64
Flutolanil	25.87	25.72	0.60	26.10	-0.88
Flutriafol	68.63	66.71	2.81	68.32	0.46
Fluvalinate-1	30.33	29.71	2.06	30.71	-1.25
Fluvalinate-2	30.23	25.00	17.30	28.89	4.43
Fonofos	75.98	73.99	2.61	74.20	2.34
gamma-BHC	34.37	32.49	5.46	31.52	8.30
Heptachlor	88.27	82.18	6.89	82.67	6.34

Table 3 (part 4). List of pesticides with ion ratio and mass accuracy summary

Compound	Target ion ratio (Standard)	Ion ratio % at 0.005 (mg/kg)	Ion ratio% diff. at 0.005 (mg/kg)	Ion ratio % at 0.01 (mg/kg)	Ion ratio % diff. at 0.010 (mg/kg)
Heptachlor epoxide	80.21	78.50	2.13	82.26	-2.55
Hexachlorobenzene	77.26	75.35	2.47	78.59	-1.73
Iodofenfos	35.56	34.05	4.26	36.45	-2.49
Iprodione	95.93	95.14	0.82	87.79	8.48
Isazophos	23.33	24.30	-4.15	23.76	-1.83
Isodrin	94.93	98.40	-3.66	94.41	0.54
Isopropalin	87.96	91.22	-3.70	88.07	-0.13
lambda-Cyhalothrin	36.88	32.69	11.35	31.06	15.79
Lenacil	7.81	7.69	1.53	7.33	6.13
Leptophos	43.62	43.78	-0.36	42.02	3.67
Linuron	67.21	63.17	6.02	58.12	13.53
Malathion	69.42	72.88	-4.99	72.77	-4.84
Metalaxyl	59.63	57.98	2.77	57.72	3.20
Metazachlor	95.03	99.87	-5.09	95.97	-0.99
Methacrifos	54.34	53.44	1.67	55.81	-2.70
Methoxychlor	15.41	15.41	0.00	15.00	2.64
Metolachlor	31.47	30.77	2.25	30.16	4.16
Mevinphos	17.81	16.70	6.22	14.87	16.50
MGK 264 Isomer A	25.34	24.58	3.00	24.81	2.10
MGK 264 Isomer B	11.91	8.89	25.36	10.21	14.29
Mirex	60.48	57.94	4.20	57.97	4.16
Myclobutanil	65.93	67.75	-2.77	66.62	-1.05
N-(2,4-Dimethylphenyl) formamide	77.87	72.55	6.83	75.20	3.42
Nitralin	41.86	34.29	18.08	33.61	19.71
Nitrofen	75.54	77.60	-2.72	74.14	1.85
Norflurazon	75.43	78.19	-3.67	72.39	4.02
o,p-DDD	66.31	75.15	-13.33	73.52	-10.88
o,p-DDE	61.71	63.40	-2.74	63.63	-3.12
o,p-DDT	62.93	63.56	-0.99	64.26	-2.10
Oxadiazone	32.02	31.23	2.48	31.05	3.02
Oxyfluorfen	34.83	34.82	0.05	35.59	-2.17
p,p-DDD	58.12	60.90	-4.78	60.67	-4.39
p,p-DDE	62.64	64.45	-2.89	63.21	-0.92
p,p-DDT	58.88	70.00	-18.89	71.29	-21.07
Paclobutrazol	79.78	79.40	0.47	81.35	-1.97
Parathion	48.72	45.40	6.81	44.75	8.14
Parathion-methyl	59.35	57.00	3.96	55.12	7.13
Pebulate	14.65	16.23	-10.77	15.76	-7.56
Penconazole	119.46	132.37	-10.81	129.33	-8.26

Table 3 (part 5). List of pesticides with ion ratio and mass accuracy summary

Compound	Target ion ratio (Standard)	Ion ratio % at 0.005 (mg/kg)	Ion ratio% diff. at 0.005 (mg/kg)	Ion ratio % at 0.01 (mg/kg)	Ion ratio % diff. at 0.010 (mg/kg)
Pendimethalin	52.52	54.56	-3.88	53.52	-1.91
Pentachloroaniline	31.03	29.42	5.20	30.26	2.48
Pentachloroanisole	80.51	82.50	-2.47	80.05	0.58
Pentachlorobenzene	61.14	60.61	0.86	63.67	-4.13
Pentachlorobenzonitrile	63.73	62.73	1.57	62.11	2.54
Pentachlorothioanisole	83.15	76.71	7.75	83.54	-0.47
Phenothrin	84.87	84.68	0.23	84.94	-0.08
Phorate	34.02	34.19	-0.50	35.61	-4.67
Phosalone	30.83	30.74	0.30	30.83	0.00
Phosmet	9.51	10.69	-12.32	12.13	-27.54
Piperonyl butoxide	18.54	19.06	-2.79	18.49	0.26
Pirimiphos methyl	99.99	98.96	1.03	101.35	-1.36
Pirimiphos-ethyl	73.25	77.44	-5.71	73.21	0.06
Pretilachlor	65.09	62.67	3.73	60.47	7.10
Prochloraz	20.94	15.70	25.01	14.96	28.58
Procymidone	75.91	76.98	-1.41	77.76	-2.43
Prodiamine	77.19	76.55	0.83	76.05	1.48
Profenofos	83.47	81.54	2.30	84.48	-1.22
Profluralin	60.56	58.03	4.18	60.29	0.45
Propachlor	72.62	75.49	-3.96	69.45	4.36
Propargite	7.72	8.69	-12.58	9.33	-20.84
Propisochlor	88.98	100.92	-13.42	84.62	4.90
Propyzamide	63.47	64.32	-1.34	63.29	0.28
Prothiofos	67.68	67.02	0.97	65.87	2.67
Pyraclofos	57.26	52.11	9.00	54.69	4.49
Pyrazophos	36.40	41.44	-13.84	38.34	-5.33
Pyridaben	6.20	5.00	19.40	5.71	8.00
Pyridaphenthion	43.87	44.25	-0.88	48.80	-11.23
Pyrimethanil	17.93	18.49	-3.11	18.38	-2.52
Pyriproxyfen	14.23	14.02	1.45	13.36	6.11
Quinalphos	88.28	88.00	0.32	87.23	1.18
Quintozene	88.59	90.22	-1.83	96.22	-8.60
Resmethrin	91.75	101.58	-10.71	110.99	-20.96
Sulfotep	90.20	91.43	-1.36	86.86	3.70
Sulprofos	48.56	47.98	1.19	47.53	2.12
Tebuconazole	127.62	134.20	-5.15	138.17	-8.27
Tebufenpyrad	33.91	32.49	4.20	32.97	2.78
Tecnazene	62.95	59.43	5.61	62.81	0.23
Tefluthrin	91.52	87.18	4.74	87.10	4.83

Table 3 (part 6). List of pesticides with ion ratio and mass accuracy summary

Compound	Target ion ratio (Standard)	Ion ratio % at 0.005 (mg/kg)	Ion ratio% diff. at 0.005 (mg/kg)	Ion ratio % at 0.01 (mg/kg)	Ion ratio % diff. at 0.010 (mg/kg)
Terbacil	48.49	46.67	3.76	46.88	3.31
Terbufos	23.97	23.97	0.02	24.42	-1.86
Terbutylazine	56.28	55.51	1.37	54.44	3.28
Tetrachlorvinphos	21.00	20.12	4.17	19.81	5.65
Tetradifon	54.44	50.31	7.58	50.73	6.81
Tetramethrin-1	51.24	40.25	21.45	43.19	15.72
Tetramethrin-2	20.13	23.89	-18.70	24.46	-21.54
Tolclofos-methyl	32.59	34.36	-5.44	34.38	-5.50
<i>trans</i> -Chlordane	96.62	93.91	2.81	94.98	1.70
<i>trans</i> -Chlorfenvinphos	62.13	61.00	1.81	62.09	0.06
Transfluthrin	50.88	50.59	0.58	51.45	-1.11
<i>trans</i> -Nonachlor	38.29	36.92	3.56	36.85	3.77
<i>trans</i> -Permethrin	15.96	16.78	-5.18	16.07	-0.71
Triadimefon	42.87	46.16	-7.67	41.40	3.44
Triadimenol	77.38	80.39	-3.89	79.18	-2.33
Triallate	73.78	75.66	-2.55	75.75	-2.68
Triazophos	71.88	65.20	9.29	69.96	2.67
Tricyclazole	52.70	51.32	2.61	48.30	8.34
Triflumizole	57.14	61.30	-7.29	60.42	-5.75
Trifluralin	43.70	41.93	4.05	44.91	-2.78
Vinclozolin	85.98	88.40	-2.82	86.01	-0.03

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