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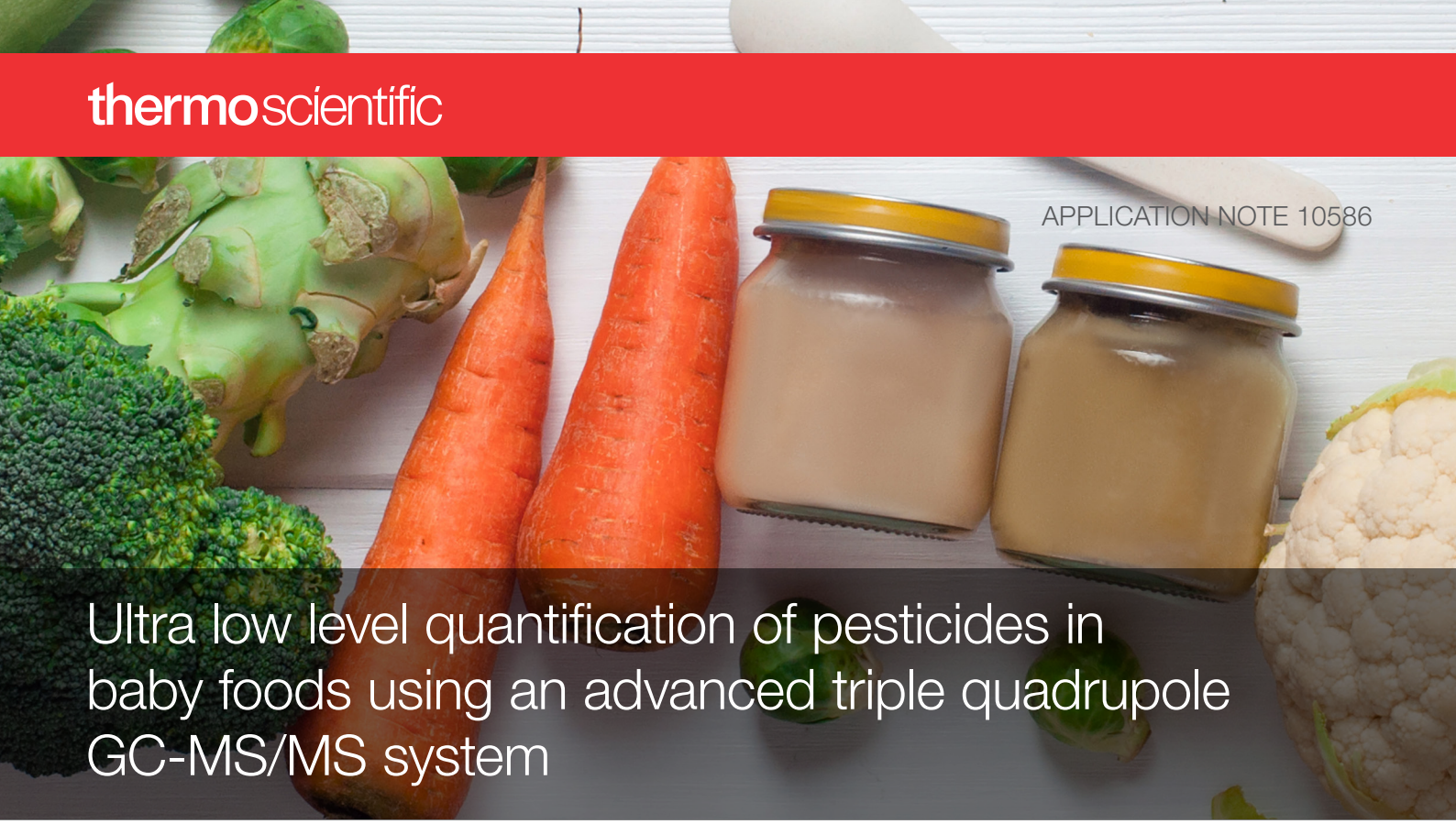

APPLICATION NOTE 10586

Ultra low level quantification of pesticides in baby foods using an advanced triple quadrupole GC-MS/MS system

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Keywords

TSQ 9000, advanced electron ionization, AEI, baby food, sensitivity, robustness, pesticide residue, QuEChERS, triple quadrupole mass spectrometry, GC-MS/MS, Programmable Temperature Vaporization, food safety

Goal

The aim of the study was to assess the quantitative performance of the Thermo Scientific™ TSQ™ 9000 triple quadrupole GC-MS/MS system fitted with the Advanced Electron Ionization (AEI) source for the analysis of pesticide residues at ultra low levels in baby food.

Introduction

The detection and subsequent quantification of pesticides, contaminants, and other chemical residues are of paramount importance, especially when the food stuff is intended to be consumed by infants or young children. The maximum residue level (MRL) for the majority of pesticide-commodity combinations is set at the default level of 10 µg/kg.[1–3] However, the European Union (EU) has established LOD MRLs between 3–8 µg/kg for specific pesticides prohibited in baby foods.[4] These pesticides and their metabolites may cause infants and young children (under worst-case intake conditions) to exceed the acceptable daily intake (ADI) values. The high sensitivity and selectivity of GC-MS/MS enables the detection and identification of residues of prohibited compounds, in compliance with the residue definitions, even when dealing with the diverse composition of multi-ingredient baby foods.

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Also, the increased levels of selectivity and sensitivity provided by triple quadrupole instruments compared to single quadrupole instruments enabled analysts to adopt faster, less specific sample extraction procedures such as QuEChERS (quick, easy, cheap effective, rugged and safe).

The QuEChERS procedure has become the standard approach for sample preparation in many laboratories because of improvement in productivity.[5] The method usually involves extraction with acetonitrile in the presence of various salts followed by dispersive solid phase extraction (dSPE) clean-up with a combination of PSA, C18, and carbon sorbents. The efficiency of the dSPE clean-up is limited so high concentrations of matrix-coextractives can remain in the final extract and cause system contamination. Also, use of acetonitrile (which has a high coefficient of expansion) limits the injection volume and hence the sensitivity of the method.

Taking all of these considerations together, it is evident that an ultra-sensitive, selective, reliable, and robust GC-MS/MS system is needed to address the challenge of routine high-throughput determination of pesticide residues at trace concentrations in baby foods. In this study, the quantitative performance of the Thermo Scientific™ TSQ™ 9000 triple quadrupole GC-MS/MS system was assessed for the analysis of more than 200 pesticides in baby food at ultra low concentrations (as low as 0.025 µg/kg). A complete evaluation of method performance included sample preparation, overall method suitability measured from pesticides recoveries, selectivity, sensitivity, linearity, and long-term robustness.

Experimental

Sample preparation

Samples of carrot/potato and apple/pear/banana baby food samples were extracted using the citrate-buffered QuEChERS protocol using Thermo Scientific™ HyperSep™ dispersive solid phase extraction (dSPE) products.

Homogenized sample (10 g) was extracted with acetonitrile (10 mL) followed by the addition of $MgSO_4$ (4 g), NaCl (1.0 g), disodium hydrogen citrate sesquihydrate (0.5 g), and trisodium citrate dihydrate (1.0 g). Dispersive solid phase extraction (dSPE) [$MgSO_4$ (150 mg), PSA (25 mg) and GCB (25 mg) per 1 mL of extract for carrot/potato and $MgSO_4$ (150 mg) and PSA (25 mg) for apple/pear/banana] was used for sample clean-up.

Preparation of matrix-matched calibrations

Immediately after dSPE clean-up, the final extracts (1 g sample/mL of acetonitrile) were acidified with 5% formic acid in acetonitrile and were spiked with a mixture of 211 pesticides at 14 concentrations spanning a range of 0.025–250 µg/kg. Robustness was tested using repeat injections of samples (carrot/potato) spiked at the 10 µg/kg level.

For method evaluation, samples of carrot/potato and apple/pear/banana baby food samples were each spiked at 1.0, 2.5, and 10.0 µg/kg (n = 6 for each concentration) before extraction, clean-up, and acidification were carried out as described above.

GC-MS/MS analysis

A TSQ 9000 triple quadrupole GC-MS/MS system equipped with a Thermo Scientific™ Advanced Electron Ionization (AEI) source and coupled with a Thermo Scientific™ TRACE™ 1310 GC system was used. The AEI source provides a highly efficient electron ionization of analytes and a more tightly focused ion beam that provides an unparalleled level of sensitivity.

Liquid injections of the sample extracts were performed using a Thermo Scientific™ TriPlus™ RSH™ autosampler, and chromatographic separation was achieved by a Thermo Scientific™ TraceGOLD™ TG-5SilMS 30 m × 0.25 mm I.D. × 0.25 µm film capillary column with 5 m integrated SafeGuard. Additional details of instrument parameters are displayed in Table 1.

Table 1. Gas chromatograph and mass spectrometer parameters.

TRACE 1310 GC System Parameters				
Injection Volume (µL)	1			
Liner	Siltek™ six baffle PTV liner (P/N 453T2120)			
Inlet (°C)	70			
Carrier Gas, (mL/min)	He, 1.2			
Inlet Mode	Splitless (split flow 50 mL/min after 2 min)			
Column	TraceGOLD TG-5SilMS with SafeGuard (30 m × 0.25 mm, 0.25 µm with 5 m integrated guard column (P/N 26096-1425)			
PTV Parameters	**Rate (°C/s)**	**Temp. (°C)**	**Time (min)**	**Flow (mL/min)**
Injection	-	70	0.10	-
Transfer	5.0	300	2.00	-
Cleaning	14.5	320	5.00	75.0
Oven Temperature Program				
Ramp	**RT (min)**	**Rate (°C/min)**	**Target Temp. (°C)**	**Hold Time (min)**
Initial	0	-	40	1.50
1	1.5	25.0	90	1.50
2	5.0	25.0	180	0.00
3	8.6	5.0	280	0.00
Final	28.6	10.0	300	5.00
Run time	35.6	-	-	-
TSQ 9000 Mass Spectrometer Parameters				
Transfer Line (°C)	250			
Ionization Type	EI			
Ion Source (°C)	320			
Acquisition Mode	timed-SRM			
Tuning parameters	AEI SmartTune			
Collision gas and pressure (psi)	Argon at 70			
Peak Width (Da)	0.7 (both Q1 and Q3)			

Data processing

Data were acquired, processed, and reported using Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, which allows instrument control, method development, quantitative/qualitative analysis, and customizable reporting all within one package.

Data review is highly customizable, allowing the user to display the information required on screen in real time. Furthermore, the flexibility of Chromeleon CDS software ensures that SANTE[3] compliance criteria can easily by flagged, tracked, and reported to the user's individual requirements.

Results and discussion

Compliance with EU SANTE criteria

The method performance was tested in accordance to the SANTE/11813/2017 guidance document, which requires that the following criteria are satisfied for identification of pesticide residues:

I. A minimum of two product ions are detected for each pesticide with peak S/N >3 (or, in case noise is absent, a signal should be present in at least five subsequent scans) and with the mass resolution for precursor-ion isolation equal to or better than unit mass resolution.

II. Retention time tolerance of ± 0.1 minutes compared with standards in the same sequence.

III. Ion ratio within ± 30% (relative) of the average of calibration standards from the same sequence.

Wherever SANTE compliance is referenced in this study, all three criteria have been met fully.

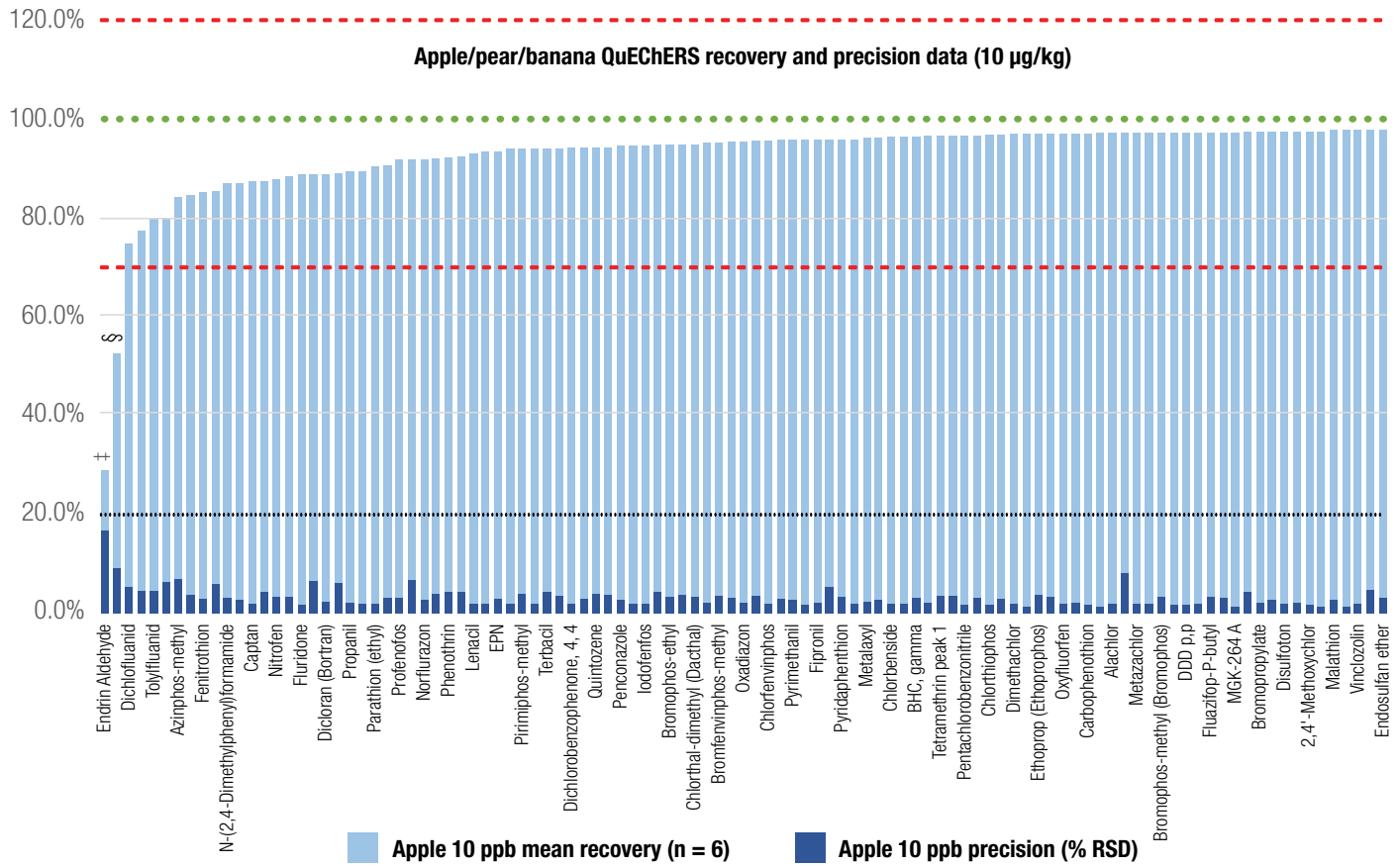
Recoveries

Pesticide recoveries were obtained from the QuEChERS extractions performed on the samples spiked before extraction. All detected compounds, at the three spiking levels in both matrices satisfied all SANTE requirements. More than 97% of the target pesticide residues at 1 µg/kg had recoveries between 70% and 120%. An example of the recovery and precision data for the apple/pear/banana matrix spiked at the default MRL (10 µg/kg) is displayed in Figure 1. A full table of results can be found in Appendix B.

Chromatography and selectivity

Analysis of a large number of pesticides in a single injection requires careful optimization of parameters, especially when injecting acetonitrile. As acetonitrile is a low molecular weight low polarity solvent, it has a relatively high expansion volume and is insoluble in the low polarity phases normally used for routine pesticide analysis (this makes solvent focusing in a standard splitless type injection incredibly difficult). These issues can be addressed by using an optimized programmable temperature vaporisation (PTV) injection. Figure 2 shows an example of three pesticides eluting in the beginning (A–dichlobenil – 0.025 µg/kg), middle (B–dieldrin 0.5 µg/kg), and end (C–deltamethrin 0.05 µg/kg) of the chromatographic run in the lowest detectable standard in carrot and potato matrix, levels at which all compounds detected meet the SANTE requirements. Peak shapes were Gaussian and coefficient of determination (R^2) was >0.990 for all three compounds indicating good chromatography and excellent linear response.

Identification of all 210 component peaks was made using an in-house, commercially available Thermo Scientific SRM pesticide compound database (cdb). In addition to this, retention time alignment of target compounds can be easily performed using the Thermo Scientific RTA tool,[8] eliminating the need for manual correction of compound retention times whenever column maintenance is performed. The cdb database contains >1000 compounds with >3700 unique SRM transitions. Due to the fast scanning speed of the EvoCell technology and the intelligent scheduling of the timed-SRM,[9] it is possible to acquire data with several transitions per compound with minimal loss in sensitivity. This makes it simple to select the most optimal transitions in differing matrices to perform quantitation and qualification on, removing the need to develop matrix-matched SRM compound databases (Figure 3).



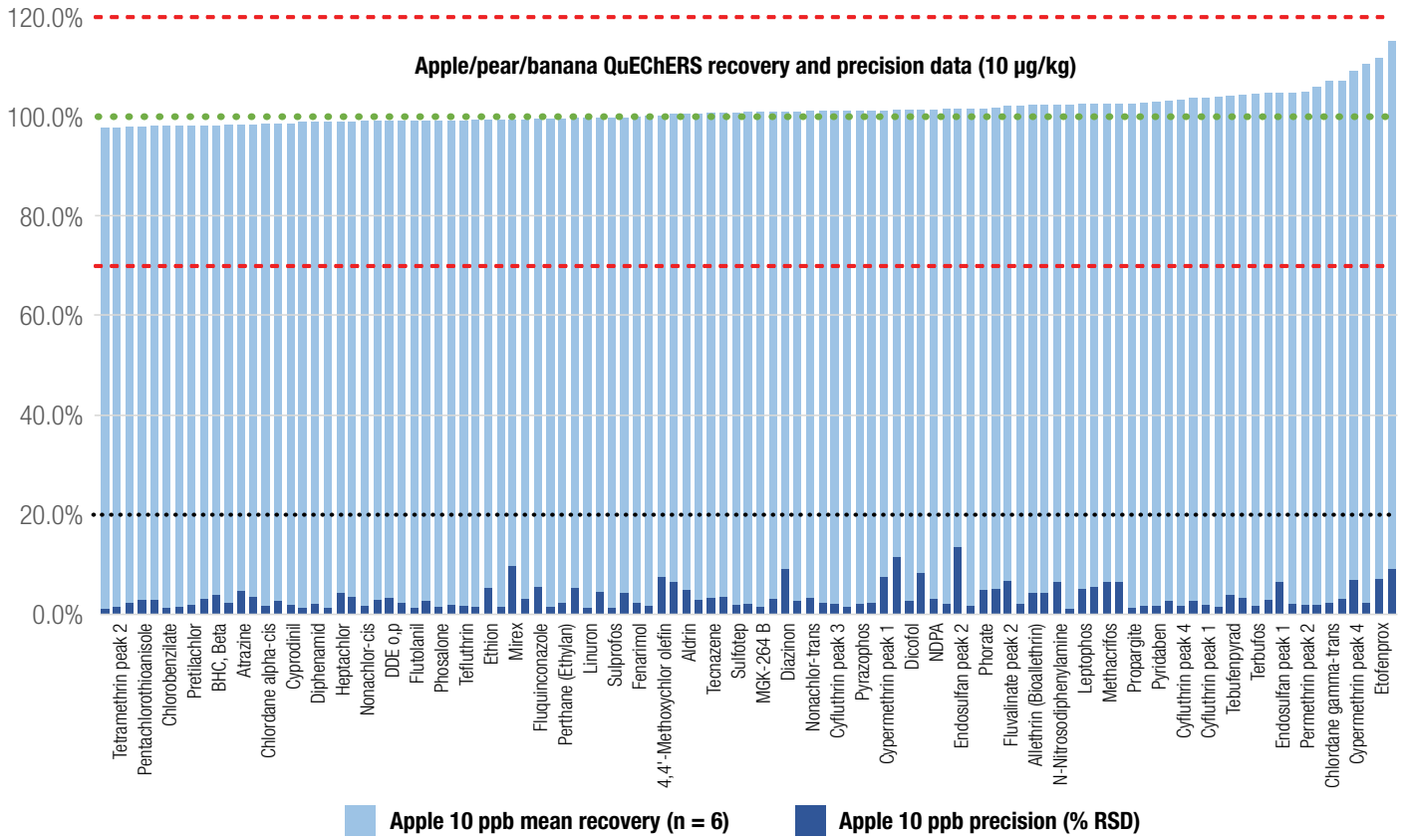


Figure 1. Recovery and precision data for apple/pear/banana extractions (n=6) at a concentration of 10 µg/kg. ‡ Endrin aldehyde recoveries were low, potentially due to reaction with PSA. § Recoveries of chlorothalonil, known to be problematic in QuEChERS extractions,[7] were low.

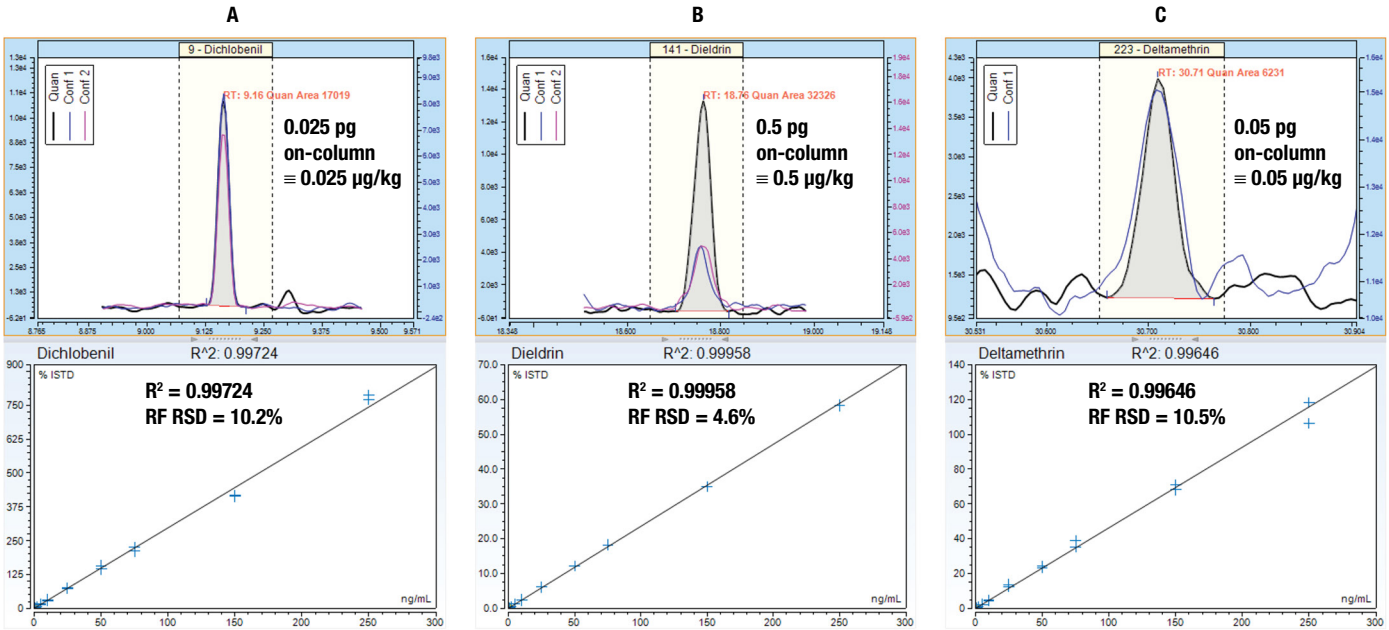


Figure 2. Example (A - Dichlobenil, B - dieldrin and C – deltamethrin) chromatographic peaks showing the lowest detectable matrix matched standard which meets SANTE requirements. The MRLs are 10 µg/kg, 3 µg/kg* and 10 µg/kg respectively. Calibration curves show duplicate injection at 14 discrete levels ranging from 0.025 pg to 250 pg on column. * Dieldrin is classed as a prohibited pesticide and 3 µg/kg considered to be the current limit of quantification, but is subject to regular review.[4]

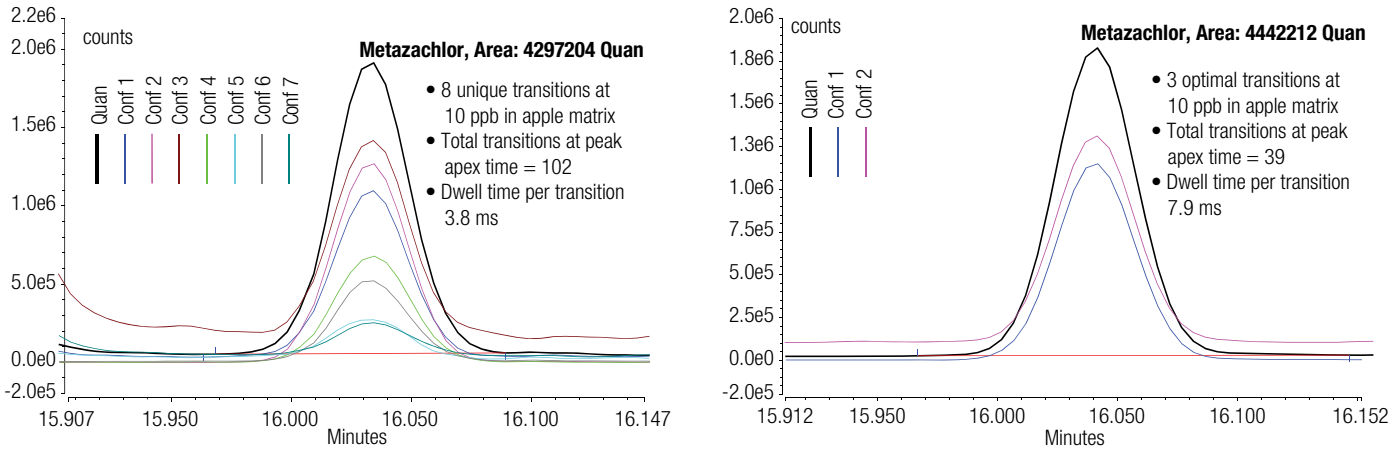


Figure 3. Comparison of Metazachlor SRM chromatographic peaks acquired using an injection containing 1317 unique transitions (left, 8 transitions) and an injection containing 663 (right, 3 transitions). No significant difference in the peak area for quantitation transition is observed indicating no loss in sensitivity.

Sensitivity and linearity

The TSQ 9000 AEI system easily met SANTE criteria (ion ratios ±30%, etc.) at the default MRL of 10 µg/kg for all pesticides targeted. Moreover, over 90% of pesticides detected at < 0.5 µg/kg meet the SANTE requirements and 10% of them meet SANTE criteria even at 0.025 µg/kg level (Figure 4). Resolution settings of 0.7 Daltons for Q1 and Q3 were used, ensuring the optimum combination of selectivity and sensitivity.

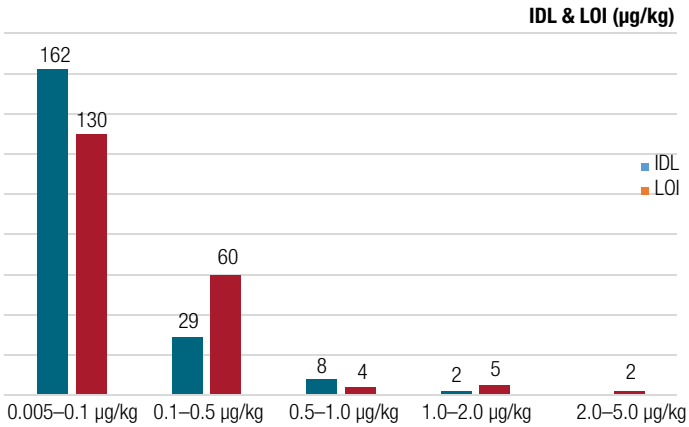


Figure 4. Number of target compounds satisfying the SANTE requirements, with over 90% below 0.5 µg/kg, and over 60% below 0.1µg/kg – 100 times lower than the default MRL [sample matrix – carrot/potato].

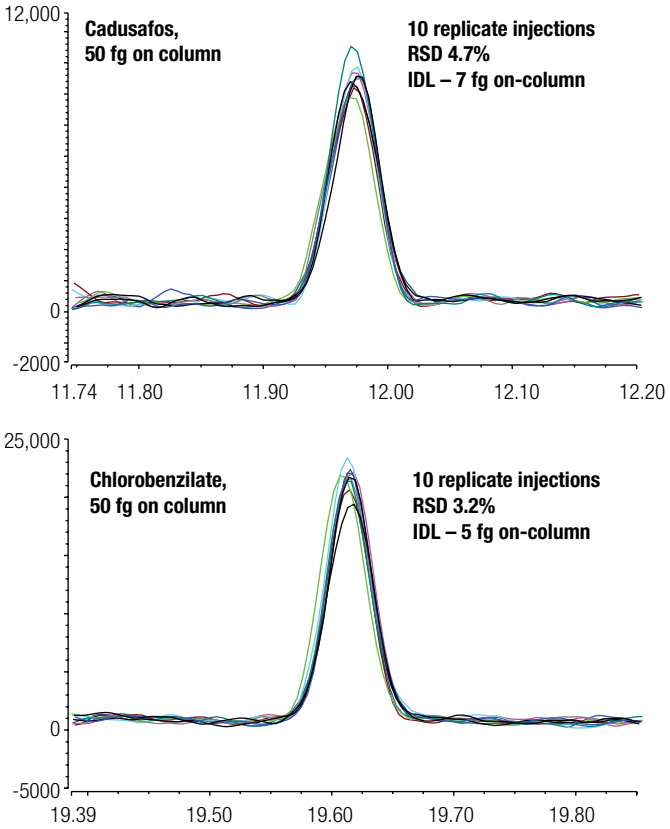


Figure 5. Example quantification SRM overlays of cadusafos and chlorbenzilate injected at the lowest level that met all SANTE criteria. Annotated are on column concentration, %RSD derived from absolute peak area response and calculated IDLs.

Over 90% of the target compounds had a Limit of Identification (LOI) (satisfying all SANTE requirements) below 0.5 µg/kg, and over 60% below 0.1 µg/kg.

System sensitivity, defined as instrumental detection limits (IDLs), was determined experimentally for each compound by performing n=10 replicate injections of the lowest matrix-matched standard of carrot and potato that met all SANTE criteria. Calculations of IDLs were then made using one-tailed student *t*-test at the 99% confidence interval for the corresponding degrees of freedom and taking into account the concentration and absolute peak area %RSD for each compound (Figures 5 and 6).

Fipronil and fipronil-desulfinyl, expressed as fipronil, have a multi-component MRL specified at 4 µg/kg. Therefore, to satisfy the current regulations, each component must be identified at 2 µg/kg. Figure 7 shows fipronil and fipronil-desulfinyl at concentrations of 0.2 µg/kg, ten times lower than the requisite MRL, with back-calculated concentrations versus the linear calibration annotated.

Compound linearity was assessed by injecting matrix-matched standards in the range of 0.025 to 250 µg/kg in duplicate for both carrot/potato and apple/pear/banana. Both sets of linearity data showed $R^2 > 0.990$ and response factor (RF) % RSDs of <20% for over 96% of component peaks indicating excellent linear response. Examples of linearity are shown in Figure 2 and in a comprehensive table provided in Appendix A.

Figure 6. Plot showing the calculated IDLs for all pesticides. IDLs ranged from ~5 fg (chlorobenzilate) to ~2.0 pg (bioallethrin) with >95% of compounds showing an IDL of less than 500 fg on column (equivalent to 0.5 µg/kg in sample extract). See Appendix A for tabulated data.

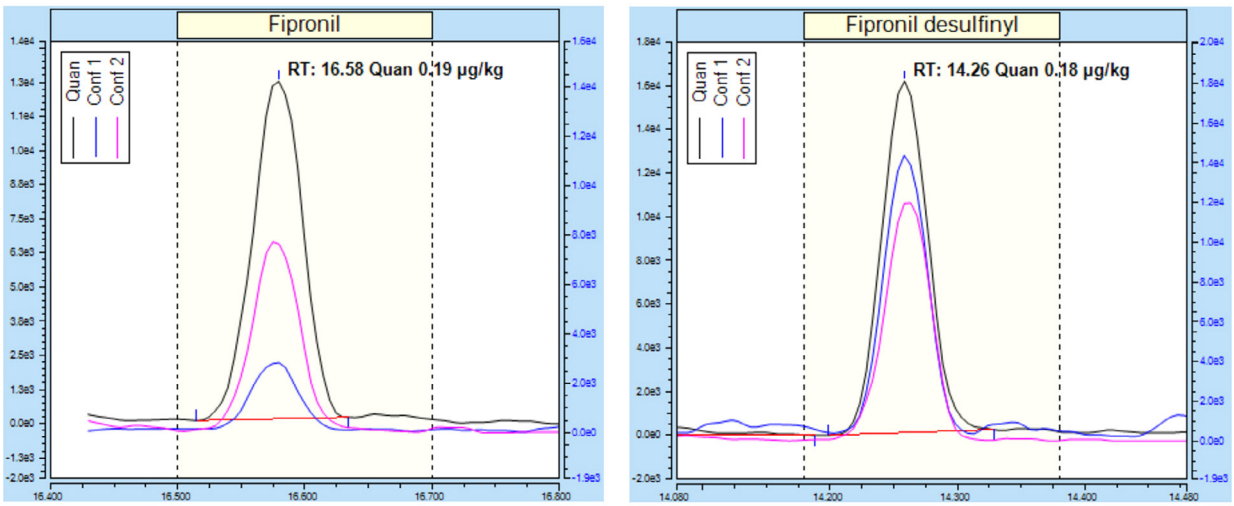


Figure 7. Fipronil and fiproni desulfinyl, at a concentration of 0.2 µg/kg equating to 0.4 µg/kg fipronil (sum), with SANTE compliance throughout.

AEI source robustness

The TSQ 9000 AEI system was set up as described in Table 1. After an initial source cleaning, repeat injections of a QuEChERS sample extract (1 g/mL carrot and potato) spiked at the default MRL (10 µg/kg) were made (Figure 8). Extracts resulting from the QuEChERS methodology contain many undesirable matrix co-extracted components which can easily contaminate the GC inlet, the chromatographic column and the MS ion source. To test the robustness of the AEI ion source only (as far as reasonably practicable), after every 100 sample injections, the PTV liner was replaced along with the injector septum, approximately 10 cm was trimmed from the head of the guard column followed by automatic tuning of the system using the SmartTune feature. SmartTune uses the MS parameters established during the initial tuning on a clean source and intelligently assess the performance of the system, only re-tuning when MS performance has been compromised. No additional maintenance was performed.

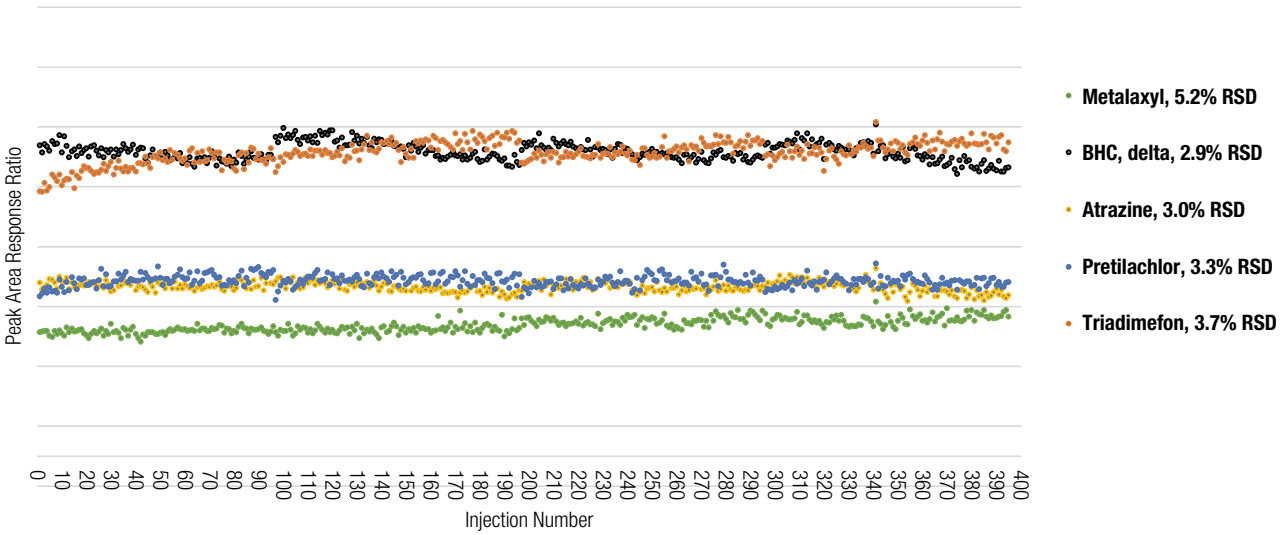


Figure 8A. Example of normalised peak area response for selected compounds across ~400 consecutive injections at the default MRL (10 µg/kg) in carrot/potato matrix.

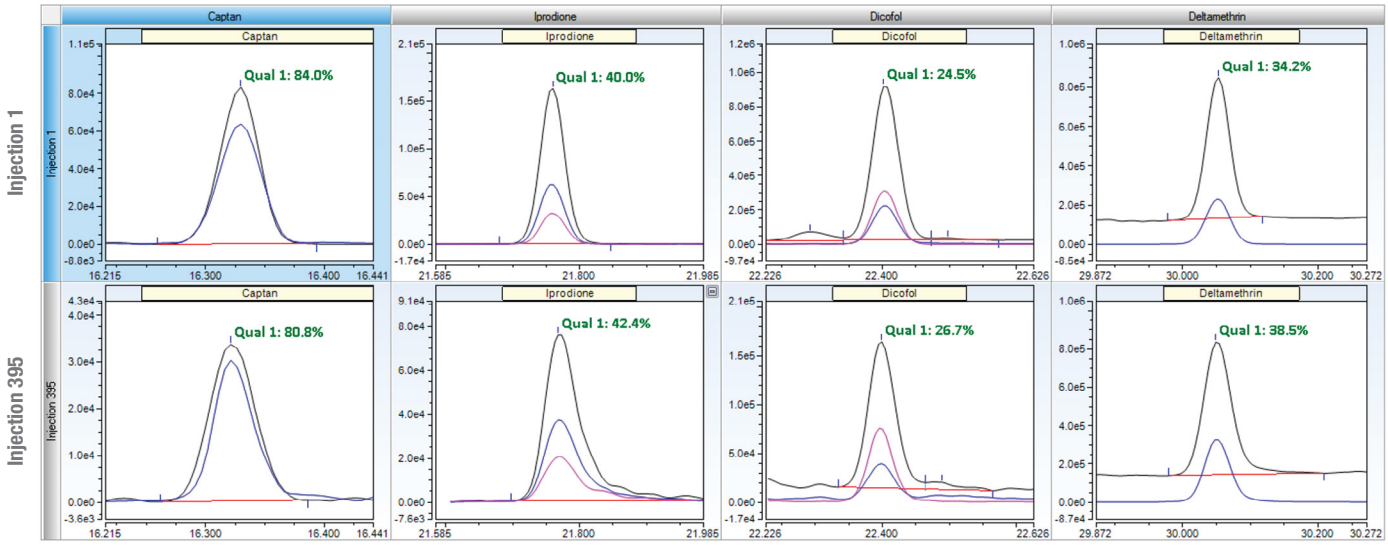


Figure 8B. Peak shapes, intensities and ion ratios of the primary qualifier ion for injection 1 (top row) and injection 395 (bottom row) for captan, iprodione, dicofol and deltamethrin.

Ion ratios at the default MRL were stable, Figure 9 shows pretilachlor ion qualifier ratios 1 and 2 in the first and last batches of injections. Ratios were well within the ±30% SANTE identification criteria.

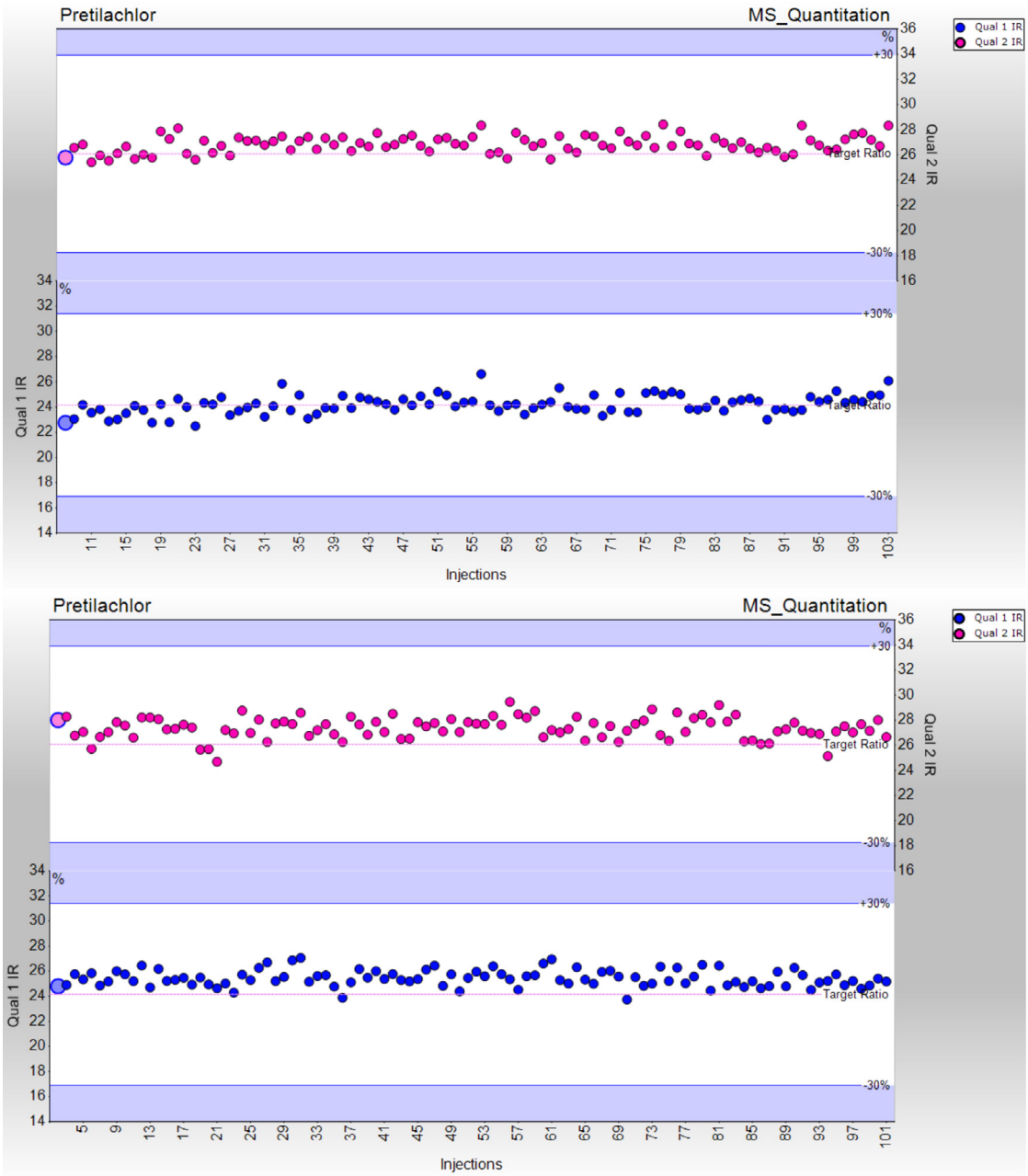


Figure 9. Pretilachlor Ion ratios of robustness injections 1-95 (top) and 295-395 (bottom).

Conclusions

In this work it has been demonstrated that by using QuEChERS with Thermo Scientific HyperSep dSPE products and a direct injection of acetonitrile extracts, the TSQ 9000 AEI system delivers outstanding quantitative performance for low-level pesticide residue analysis in baby food.

- QuEChERS extraction and subsequent clean-up of over 200 pesticides from replicate analysis (n=6 each at three concentrations) of each of two sample matrices, demonstrating excellent accuracy (recovery) and precision.
- Accurate, quantitative analysis of over 200 pesticides over up to five orders of magnitude (0.025–250 µg/kg), showing outstanding LODs and linear response.
- Robustness displayed over approximately 400 consecutive injections of sample matrix (1 g/mL), with SANTE compliance at the default MRL throughout.
- High sensitivity providing the real possibility to dilute the sample extract, thus limiting matrix contamination and system maintenance, leading to an increase in laboratory productivity.

The results of this study establish the TSQ 9000 triple quadrupole GC-MS/MS system, in combination with Chromeleon CDS software and HyperSep dSPE products, as the ideal solution for the routine analysis of pesticides in baby food, providing unprecedented sensitivity, robustness, ease of use, cost effectiveness, and reliability.

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Appendix A – Linearity data sets

Appendix A (Part 1). Linearity data sets.

Chart Number	Compound Name	Apple/Pear/Banana Linearity			Carrot/Potato Linearity			IDL_{99}	
		R^2	RF RSD(%)	Range (ppb)	R^2	RF RSD(%)	Range (ppb)	pg on Column	IDL (fg)
1	2,3,5,6-Tetrachloroaniline	0.99931	6.80	0.1–250	0.99955	7.70	0.05–250	0.05	20
2	2,4'-Methoxychlor	0.99987	3.50	0.1–250	0.99950	7.30	0.025–250	0.05	8
3	4,4'-Methoxychlor olefin	0.99976	5.80	0.1–250	0.99932	4.20	0.05–250	0.05	31
4	Acetochlor	0.99972	3.60	0.2–250	0.99962	3.60	0.2–250	0.20	201
5	Acrinathrin	0.99963	2.70	0.2–250	0.99955	3.90	0.2–250	0.20	57
6	Alachlor	0.99965	5.50	0.1–250	0.99955	5.10	0.2–250	0.20	71
7	Aldrin	0.99959	8.00	0.1–250	0.99983	4.60	0.1–250	0.20	75
8	Allethrin (Bioallethrin)	0.99826	19.40	10–250	0.99888	7.50	5–250	5.00	2007
9	Allidochlor	0.99926	15.20	0.1–250	0.99631	6.30	0.2–250	0.20	145
10	Anthraquinone	0.99966	9.80	0.2–250	0.99988	17.60	0.025–250	0.05	27
11	Atrazine	0.99963	7.40	0.1–250	0.99990	6.70	0.05–250	0.05	19
12	Azinphos-ethyl	0.99465	9.40	0.2–250	0.99935	4.50	0.2–250	0.20	82
13	Azinphos-methyl	0.98165	16.90	1–250	0.99758	19.60	0.5–250	1.00	521
14	BHC, Alpha	0.99981	5.30	0.025–250	0.99949	6.60	0.025–250	0.05	15
15	BHC, Beta	0.99967	6.20	0.05–250	0.99985	8.80	0.025–250	0.05	15
16	BHC, delta	0.99971	4.10	0.05–250	0.99992	7.60	0.025–250	0.05	20
17	BHC, gamma	0.99970	7.30	0.05–250	0.99971	6.70	0.05–250	0.05	31
18	Bifenthrin	0.99989	4.20	0.5–250	0.99976	2.40	0.5–250	1.00	42
19	Biphenyl	0.99822	19.50	2–250	0.99573	14.50	5–250	5.00	865
20	Bromfenvinphos	0.99963	5.10	0.05–250	0.99960	7.60	0.025–250	0.05	31
21	Bromfenvinphos-methyl	0.99917	3.20	0.5–250	0.99971	3.60	0.1–250	0.20	33
22	Bromophos-ethyl	0.99946	3.30	0.1–250	0.99523	5.90	0.05–250	0.05	12
23	Bromophos-methyl (Bromophos)	0.99957	5.60	0.05–250	0.99848	5.90	0.05–250	0.05	24
24	Bromopropylate	0.99960	4.80	0.1–250	0.99806	5.40	0.1–250	0.20	61
25	Bupirimate	0.99947	10.10	0.05–250	0.99961	5.50	0.05–250	0.05	33
26	Cadusafos	0.99982	3.80	0.1–250	0.99952	6.40	0.025–250	0.05	7
27	Captan	0.98233	23.80	1–250	0.98303	16.60	0.5–250	1.00	733
28	Carbophenothion	0.99968	3.50	0.2–250	0.99970	4.40	0.1–250	0.20	30
29	Carfentrazon-ethyl	0.99929	6.10	0.2–250	0.99575	7.50	0.1–250	0.20	41
30	Chlorbenside	0.99981	5.50	0.025–250	0.99984	3.20	0.025–250	0.05	11
31	Chlordane alpha-cis	0.99875	8.10	0.05–250	0.98923	8.10	0.1–250	0.20	61
32	Chlordane gamma-trans	0.99949	6.50	0.05–250	0.99956	7.30	0.025–250	0.05	38
33	Chlorfenapyr	0.99979	6.10	0.2–250	0.99983	3.30	0.2–250	0.20	90
34	Chlorfenson	0.99986	3.60	0.025–250	0.99979	2.30	0.025–250	0.05	10
35	Chlorfenvinphos	0.99966	8.60	0.05–250	0.99987	5.70	0.025–250	0.05	16
36	Chlorobenzilate	0.99990	3.10	0.025–250	0.99976	3.70	0.025–250	0.05	5
37	Chloroneb	0.99962	4.70	0.5–250	0.99907	10.90	0.1–250	0.20	28
38	Chlorothalonil	0.99752	7.40	0.1–250	0.99635	18.40	0.05–250	0.05	24
39	Chlorpropham	0.99985	13.90	0.5–250	0.99981	12.60	2–250	5.00	166
40	Chlorpyrifos-ethyl	0.99948	15.00	0.1–250	0.99916	5.50	0.05–250	0.05	22

Appendix A (Part 2). Linearity data sets.

Chart Number	Compound Name	Apple/Pear/Banana Linearity			Carrot/Potato Linearity			IDL_{99}	
		R^2	RF RSD(%)	Range (ppb)	R^2	RF RSD(%)	Range (ppb)	pg on Column	IDL (fg)
41	Chlorpyrifos-methyl	0.99969	12.30	0.1–250	0.99960	15.70	0.025–250	0.05	24
42	Chlorthal-dimethyl	0.99917	6.20	0.1–250	0.99912	5.80	0.025–250	0.05	20
43	Chlorthiophos	0.99976	7.20	0.1–250	0.99971	3.00	0.2–250	0.20	153
44	Chlozolinate	0.99981	7.90	0.2–250	0.99988	4.30	0.2–250	0.20	67
45	Clomazone	0.99983	4.30	0.025–250	0.99993	6.40	0.025–250	0.05	12
46	Coumaphos	0.99817	4.80	0.5–250	0.99966	8.50	0.2–250	0.20	78
47	Cycloate	0.99815	13.10	2–250	0.99890	4.80	2–250	5.00	789
48	Cyhalothrin I (lambda)	0.99966	7.50	0.2–250	0.99959	2.90	0.2–250	0.20	28
49	Cyprodinil	0.99983	5.00	0.1–250	0.99993	3.90	0.1–250	0.20	86
50	DDD p,p	0.99985	4.30	0.05–250	0.99992	4.00	0.025–250	0.05	22
51	DDD, o, p	0.99988	3.00	0.05–250	0.99985	7.50	0.05–250	0.05	20
52	DDE o,p	0.99974	3.80	0.025–250	0.99976	4.30	0.025–250	0.05	8
53	DDE p, p	0.99957	2.60	0.05–250	0.99989	4.20	0.05–250	0.05	9
54	DDT o,p	0.99988	5.70	0.05–250	0.99960	9.20	0.05–250	0.05	25
55	DDT p,p	0.99962	8.40	0.2–250	0.99937	14.40	0.1–250	0.20	41
56	Deltamethrin	0.99983	6.70	0.05–250	0.99646	11.00	0.05–250	0.05	22
57	Diazinon	0.99949	5.20	0.1–250	0.99906	5.00	0.1–250	0.20	33
58	Dichlobenil	0.99866	5.10	0.025–250	0.99724	10.20	0.025–250	0.05	5
59	Dichlofluanid	0.99949	6.40	0.2–250	0.99966	4.40	0.1–250	0.20	28
60	Dichlorobenzophenone, 4, 4*	0.99979	2.80	0.05–250	0.99957	7.50	0.025–250	0.05	17
61	Dicloran (Bortran)	0.99908	7.10	0.2–250	0.99801	7.00	0.2–250	0.20	58
62	Dicofol*	0.99272	13.50	0.5–250	0.98598	14.70	0.5–250	1.00	973
63	Dieldrin	0.99909	7.20	0.5–250	0.99958	4.60	0.5–250	1.00	162
64	Dimethachlor	0.99964	5.10	0.025–250	0.99968	4.60	0.025–250	0.05	10
65	Dimethoate	0.99903	6.30	0.2–250	0.99973	10.40	0.1–250	0.20	30
66	Diphenamid	0.99974	7.30	0.2–250	0.99974	5.20	0.2–250	0.20	62
67	Diphenylamine	0.99981	9.50	0.2–250	0.99931	17.00	0.1–250	0.20	28
68	Disulfoton	0.99982	6.80	0.2–250	0.99943	4.30	0.2–250	0.20	19
69	Edifenphos	0.99908	4.00	0.05–250	0.99967	11.50	0.025–250	0.05	10
70	Endosulfan ether	0.99983	5.70	0.05–250	0.99982	12.20	0.025–250	0.05	21
71	Endosulfan peak 1	0.99963	4.50	0.2–250	0.99989	4.50	0.2–250	0.20	42
72	Endosulfan peak 2	0.99982	5.20	0.5–250	0.99988	4.00	0.5–250	1.00	190
73	Endosulfan sulfate	0.99981	3.10	0.1–250	0.99980	3.60	0.05–250	0.05	20
74	Endrin	0.99981	3.70	0.5–250	0.99975	4.80	0.2–250	0.20	59
75	Endrin Aldehyde	0.99893	7.30	0.5–250	0.99786	9.30	0.5–250	1.00	209
76	Endrin-Ketone	0.99920	6.00	0.5–250	0.99872	5.80	0.5–250	1.00	353
77	EPN	0.99591	7.10	1–250	0.99334	14.00	1–250	1.00	302
78	Ethion	0.99981	3.60	0.1–250	0.99987	3.90	0.05–250	0.05	29
79	Ethoprop (Ethoprophos)	0.99975	6.10	0.05–250	0.99923	6.40	0.1–250	0.20	39
80	Etofenprox	0.99978	7.40	0.2–250	0.99992	3.70	0.2–250	0.20	42

* - 4,4-dichlorobenzophenone is a breakdown product of dicofol and therefore may be overestimated.

Appendix A (Part 3). Linearity data sets.

Chart Number	Compound Name	Apple/Pear/Banana Linearity			Carrot/Potato Linearity			IDL_{99}	
		R^2	RF RSD(%)	Range (ppb)	R^2	RF RSD(%)	Range (ppb)	pg on Column	IDL (fg)
81	Etridiazole (Terrazole)	0.99954	5.50	0.1–250	0.99599	16.30	0.025–250	0.05	18
82	Fenamiphos	0.99958	3.00	0.5–250	0.99848	3.70	0.5–250	1.00	147
83	Fenarimol	0.99984	3.20	0.2–250	0.99990	2.60	0.2–250	0.20	45
84	Fenchlorfos	0.99964	3.90	0.05–250	0.99956	4.80	0.05–250	0.05	17
85	Fenitrothion	0.99241	13.50	0.1–250	0.99559	13.30	0.1–250	0.20	52
86	Fenpropathrin	0.99969	3.70	0.5–250	0.99976	5.00	1–250	1.00	147
87	Fenson	0.99989	4.40	0.05–250	0.99995	5.60	0.025–250	0.05	11
88	Fenthion	0.99959	10.20	0.05–250	0.99970	10.60	0.05–250	0.05	18
89	Fenvalerate	0.99992	3.10	0.1–250	0.99974	5.80	0.1–250	0.20	36
90	Fipronil	0.99923	5.10	0.1–250	0.99405	8.90	0.05–250	0.05	20
91	Fipronil desulfinyl	0.99826	5.60	0.05–250	0.98489	10.00	0.05–250	0.05	27
92	Fluazifop-P-butyl	0.99971	7.40	0.1–250	0.99976	10.50	0.1–250	0.20	45
93	Fludioxonil	0.99980	9.90	0.05–250	0.99951	8.00	0.025–250	0.05	29
94	Fluquinconazole	0.99953	7.70	0.05–250	0.99609	7.40	0.025–250	0.05	15
95	Fluridone	0.99890	13.60	0.2–250	0.99593	7.10	0.025–250	0.05	22
96	Flusilazole	0.99969	11.20	0.1–250	0.99982	9.00	0.1–250	0.20	66
97	Flutolanil	0.99966	5.80	0.05–250	0.99982	9.30	0.025–250	0.05	51
98	Flutriafol	0.99960	7.20	0.1–250	0.99989	9.50	0.025–250	0.05	24
99	Folpet	0.97866	23.90	0.5–250	0.98874	14.20	0.2–250	0.20	757
100	Fonofos	0.99970	4.80	0.05–250	0.99986	4.20	0.05–250	0.05	14
101	Heptachlor	0.99963	4.20	0.025–250	0.99976	6.40	0.025–250	0.05	7
102	Hexachlorobenzene	0.99862	7.80	0.025–250	0.99939	5.60	0.025–250	0.05	11
103	Hexazinone	0.99971	7.20	0.1–250	0.99983	5.50	0.05–250	0.05	18
104	Iodofenfos	0.99859	6.10	0.05–250	0.99012	11.60	0.05–250	0.05	19
105	Iprodione	0.99976	7.70	0.2–250	0.99536	20.80	0.1–250	0.20	80
106	Isazophos	0.99934	6.60	0.1–250	0.99945	12.10	0.1–250	0.20	46
107	Isodrin	0.99983	5.90	0.1–250	0.99992	6.20	0.1–250	0.20	26
108	Lenacil	0.99928	7.90	0.2–250	0.99971	5.30	0.1–250	0.20	83
109	Leptophos	0.99947	3.30	0.2–250	0.99909	3.80	0.2–250	0.20	36
110	Linuron	0.99913	8.00	0.5–250	0.99831	8.70	0.2–250	0.20	92
111	Malathion	0.99989	5.70	0.05–250	0.99972	6.10	0.025–250	0.05	12
112	Metalaxyl	0.99947	4.70	0.2–250	0.99985	21.80	0.1–250	0.20	54
113	Metazachlor	0.99958	3.80	0.1–250	0.99978	8.10	0.025–250	0.05	32
114	Methacrifos	0.99977	4.50	0.2–250	0.99819	4.80	0.2–250	0.20	91
115	Methoxychlor	0.99918	4.50	0.1–250	0.99921	5.40	0.1–250	0.20	38
116	Metolachlor	0.99978	4.20	0.05–250	0.99992	4.20	0.025–250	0.05	49
117	Mevinphos	0.99985	3.80	0.05–250	0.99937	4.60	0.1–250	0.20	31
118	MGK-264 A	0.99986	5.00	0.2–250	0.99966	4.00	0.2–250	0.20	51
119	MGK-264 B	0.99984	4.50	0.2–250	0.99974	4.40	0.2–250	0.20	65
120	Mirex	0.99980	4.60	0.025–250	0.99981	3.00	0.025–250	0.05	8

Appendix A (Part 4). Linearity data sets.

Chart Number	Compound Name	Apple/Pear/Banana Linearity			Carrot/Potato Linearity			IDL_{99}	
		R^2	RF RSD(%)	Range (ppb)	R^2	RF RSD(%)	Range (ppb)	pg on Column	IDL (fg)
121	Myclobutanil	0.99978	1.90	0.1–250	0.99986	3.50	0.1–250	0.20	40
122	N-(2,4-Dimethylphenyl) formamide	0.99953	4.80	1–250	0.99982	8.30	1–250	1.00	106
123	NDBA	0.99866	18.00	0.5–250	0.99414	21.60	0.05–250	0.05	12
124	NDEA	0.99826	6.90	0.1–250	0.98989	9.80	0.2–250	0.20	74
125	NDPA	0.99865	8.70	0.5–250	0.99133	8.80	0.1–250	0.20	49
126	NEMA	0.99657	8.00	1–250	0.98500	12.80	0.2–250	0.20	87
127	Nitrofen	0.99590	13.50	0.05–250	0.99512	15.00	0.2–250	0.20	30
128	*N*-Nitrosodiphenylamine	0.99971	12.70	0.2–250	0.99931	17.00	0.1–250	0.20	28
129	*N*-Nitrosomorpholine	0.99759	18.50	0.5–250	0.99342	20.00	0.5–250	1.00	198
130	*N*-Nitrosopiperidine	0.99738	7.90	0.5–250	0.99368	14.20	0.5–250	1.00	217
131	*N*-Nitrosopyrrolidine	0.99830	14.70	1–250	0.99359	8.00	0.5–250	1.00	289
132	Nonachlor-cis	0.99591	7.20	0.2–250	0.97664	11.80	0.1–250	0.20	90
133	Nonachlor-trans	0.99924	4.70	0.1–250	0.99968	6.70	0.1–250	0.20	28
134	Norflurazon	0.99886	7.60	0.2–250	0.99937	5.10	0.05–250	0.05	23
135	Ortho-phenylphenol	0.99979	20.20	0.5–250	0.99957	14.70	0.5–250	1.00	102
136	Oxadiazon	0.99964	5.80	0.025–250	0.99970	14.10	0.1–250	0.20	22
137	Oxyfluorfen	0.99610	9.30	0.5–250	0.99445	18.80	0.5–250	1.00	73
138	Paclobutrazol	0.99977	5.40	0.05–250	0.99991	6.70	0.05–250	0.05	49
139	Parathion (ethyl)	0.99534	11.00	0.5–250	0.99395	19.40	0.5–250	1.00	158
140	Parathion-methyl	0.99478	12.00	0.05–250	0.99736	8.20	0.2–250	0.20	111
141	Pebulate	0.99885	14.30	0.5–250	0.99691	13.30	0.5–250	1.00	171
142	Penconazole	0.99985	6.30	0.05–250	0.99992	7.90	0.05–250	0.05	37
143	Pentachloroaniline	0.99973	6.10	0.1–250	0.99961	4.60	0.05–250	0.05	52
144	Pentachloroanisole	0.99939	5.30	0.05–250	0.99956	11.30	0.025–250	0.05	18
145	Pentachlorobenzene	0.99665	8.50	0.025–250	0.99765	11.40	0.025–250	0.05	12
146	Pentachlorobenzonitrile	0.99984	5.10	0.05–250	0.99973	8.40	0.025–250	0.05	23
147	Pentachlorothioanisole	0.99951	8.40	0.025–250	0.99973	6.00	0.05–250	0.05	22
148	Perthane (Ethylan)	0.99994	8.00	0.05–250	0.99982	4.50	0.1–250	0.20	30
149	Permethrin peak 1	0.99971	9.90	1–250	0.99979	12.90	0.2–250	0.20	219
150	Permethrin peak 2	0.99970	6.10	0.5–250	0.99979	5.80	0.5–250	1.00	48
151	Phenothrin	0.99950	17.90	1–250	0.99972	7.40	2–250	5.00	413
152	Phorate	0.99964	3.10	0.5–250	0.99910	13.50	0.025–250	0.05	18
153	Phosalone	0.99862	8.80	0.05–250	0.99982	8.90	0.05–250	0.05	18
154	Phosmet	0.99738	7.00	0.5–250	0.99916	24.50	0.2–250	0.20	54
155	Piperonyl butoxide	0.99977	6.50	0.1–250	0.99990	4.90	0.1–250	0.20	51
156	Pirimiphos-ethyl	0.99964	3.00	0.05–250	0.99967	5.30	0.025–250	0.05	21
157	Pirimiphos-methyl	0.99949	5.50	0.05–250	0.99949	4.80	0.025–250	0.05	18
158	Pretilachlor	0.99984	3.60	0.2–250	0.99989	2.40	0.2–250	0.20	44
159	Prochloraz (parent)	0.99749	14.30	1–250	0.99920	7.80	0.5–250	1.00	320
160	Procymidone	0.99991	4.00	0.1–250	0.99969	7.50	0.05–250	0.05	26

Appendix A (Part 5). Linearity data sets.

Chart Number	Compound Name	Apple/Pear/Banana Linearity			Carrot/Potato Linearity			IDL_{99}	
		R^2	RF RSD(%)	Range (ppb)	R^2	RF RSD(%)	Range (ppb)	pg on Column	IDL (fg)
161	Profenofos	0.99938	9.40	0.1–250	0.99654	10.40	0.1–250	0.20	53
162	Propachlor	0.99977	1.60	1–250	0.99926	15.30	0.025–250	0.05	13
163	Propanil	0.99925	12.30	0.025–250	0.99974	3.70	0.1–250	0.20	32
164	Propargite	0.99970	3.30	2–250	0.99881	19.40	2–250	5.00	1143
165	Propisochlor	0.99972	21.10	0.2–250	0.99953	5.80	0.5–250	1.00	284
166	Propyzamide	0.99966	4.50	0.1–250	0.99985	4.20	0.025–250	0.05	15
167	Prothiofos	0.99881	10.50	0.1–250	0.99842	5.90	0.1–250	0.20	28
168	Pyraclofos	0.99656	15.00	0.1–250	0.99920	20.60	0.05–250	0.05	28
169	Pyrazophos	0.99911	13.30	0.5–250	0.99979	16.60	0.2–250	0.20	163
170	Pyridaben	0.99987	3.10	0.2–250	0.99988	2.50	0.2–250	0.20	186
171	Pyridaphenthion	0.99941	3.80	0.2–250	0.99699	8.90	0.1–250	0.20	24
172	Pyrimethanil	0.99985	19.10	0.1–250	0.99971	8.40	0.05–250	0.05	23
173	Pyriproxyfen	0.99979	9.20	0.1–250	0.99990	2.30	0.2–250	0.20	24
174	Quinalphos	0.99926	10.80	0.5–250	0.99925	4.60	0.5–250	1.00	88
175	Quintozene	0.99912	10.20	0.2–250	0.99774	15.00	0.2–250	0.20	72
176	Sulfotep	0.99970	9.80	0.025–250	0.99962	9.40	0.025–250	0.05	26
177	Sulprofos	0.99986	3.30	0.05–250	0.99850	5.20	0.025–250	0.05	12
178	Tebuconazole	0.99983	9.10	0.5–250	0.99994	4.70	0.025–250	0.05	30
179	Tebufenpyrad	0.99980	4.20	0.05–250	0.99976	4.10	0.05–250	0.05	37
180	Tecnazene	0.99958	8.90	0.05–250	0.99815	13.90	0.025–250	0.05	15
181	Tefluthrin	0.99982	12.50	0.025–250	0.99944	6.60	0.025–250	0.05	34
182	Terbacil	0.99929	7.30	0.2–250	0.99974	5.40	0.1–250	0.20	95
183	Terbufos	0.99973	4.90	0.1–250	0.99978	5.00	0.05–250	0.05	13
184	Terbuthylazine	0.99967	8.60	0.2–250	0.99982	6.10	0.1–250	0.20	72
185	Tetrachlorvinphos	0.99941	8.20	0.05–250	0.99651	10.10	0.025–250	0.05	13
186	Tetradifon	0.99988	3.80	0.2–250	0.99990	17.50	0.025–250	0.05	17
187	Tetrahydrophthalimide (THPI)	0.99744	10.40	0.5–250	0.99985	6.00	0.5–250	1.00	67
188	Tolclofos-methyl	0.99985	4.70	0.05–250	0.99986	10.50	0.05–250	0.05	18
189	Tolylfluanid	0.99911	7.30	0.1–250	0.99952	7.90	0.1–250	0.20	27
190	Triadimefon	0.99965	8.80	0.05–250	0.99973	7.50	0.05–250	0.05	18
191	Triadimenol	0.99983	15.90	0.5–250	0.99973	11.20	0.5–250	1.00	116
192	Triallate	0.99983	2.20	0.1–250	0.99984	6.10	0.025–250	0.05	18
193	Triazophos	0.99937	6.30	0.1–250	0.99983	5.60	0.05–250	0.05	16
194	Tricyclazole	0.99883	9.50	2–250	0.99947	4.50	0.5–250	1.00	367
195	Triflumizole	0.99976	6.70	0.2–250	0.99978	6.00	0.5–250	1.00	147
196	Vinclozolin	0.99967	10.10	0.05–250	0.99969	5.50	0.05–250	0.05	22
197	Tetramethrin peaks 1&2	N/A	N/A	0.5–250	N/A	N/A	0.5–250	5.00	929
198	Resmethrin peaks 1&2	N/A	N/A	2–250	N/A	N/A	2–250	5.00	797
199	Fluvalinate peaks 1&2	N/A	N/A	0.1–250	N/A	N/A	0.1–250	0.20	32
200	Cypermethrin peaks I-IV	N/A	N/A	1–250	N/A	N/A	1–250	1.00	214

Appendix A (Part 6). Linearity data sets.

Chart Number	Compound Name	Apple/Pear/Banana Linearity			Carrot/Potato Linearity			IDL_{99}	
		R^2	RF RSD(%)	Range (ppb)	R^2	RF RSD(%)	Range (ppb)	pg on Column	IDL (fg)
201	Cyfluthrin peaks I-IV	N/A	N/A	0.5–250	N/A	N/A	0.5–250	1.00	91
N/A	Tetramethrin peak 1	0.99965	14.70	N/A	0.99968	16.50	N/A	N/A	N/A
N/A	Tetramethrin peak 2	0.99974	18.80	N/A	0.99984	20.80	N/A	N/A	N/A
N/A	Resmethrin peak 1	0.99950	13.60	N/A	0.99976	19.70	N/A	N/A	N/A
N/A	Resmethrin peak 2	0.99983	6.60	N/A	0.99967	10.50	N/A	N/A	N/A
N/A	Fluvalinate peak 1	0.99946	6.90	N/A	0.99936	5.50	N/A	N/A	N/A
N/A	Fluvalinate peak 2	0.99933	10.20	N/A	0.99886	4.40	N/A	N/A	N/A
N/A	Cypermethrin peak 1	0.99971	4.30	N/A	0.99989	13.40	N/A	N/A	N/A
N/A	Cypermethrin peak 2	0.99987	2.40	N/A	0.99988	5.10	N/A	N/A	N/A
N/A	Cypermethrin peak 3	0.99982	2.80	N/A	0.99975	3.10	N/A	N/A	N/A
N/A	Cypermethrin peak 4	0.99981	2.70	N/A	0.99991	2.90	N/A	N/A	N/A
N/A	Cyfluthrin peak 1	0.99967	2.50	N/A	0.99988	3.90	N/A	N/A	N/A
N/A	Cyfluthrin peak 2	0.99905	4.40	N/A	0.99972	3.00	N/A	N/A	N/A
N/A	Cyfluthrin peak 3	0.99837	10.70	N/A	0.99979	3.30	N/A	N/A	N/A
N/A	Cyfluthrin peak 4	0.99723	8.10	N/A	0.99981	10.50	N/A	N/A	N/A

Appendix B – QuEChERS Recovery data

Appendix B (Part 1). QuEChERS Recovery data.

Component Name	Carrot 1 µg/kg (n=6)		Apple 1 µg/kg (n=6)		Carrot 2.5 µg/kg (n=6)		Apple 2.5 µg/kg (n=3)		Carrot 10 µg/kg (n=6)		Apple 10 µg/kg (n=6)	
	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)
2,3,5,6-Tetrachloroaniline	100.9%	6.2%	97.8%	10.2%	94.7%	5.1%	99.2%	3.4%	87.6%	3.4%	99.4%	5.5%
2,4'-Methoxychlor	98.1%	0.9%	98.4%	1.2%	96.4%	1.0%	98.1%	1.2%	100.1%	1.2%	97.5%	1.1%
4,4'-Methoxychlor olefin	99.9%	2.8%	106.7%	3.5%	98.8%	2.6%	99.4%	1.6%	101.0%	1.1%	100.2%	1.6%
Acetochlor	89.9%	8.2%	90.9%	9.1%	79.1%	8.4%	110.5%	3.4%	97.0%	2.2%	98.2%	3.1%
Acrinathrin	96.5%	4.2%	92.3%	4.5%	112.3%	3.2%	96.4%	4.4%	109.2%	2.0%	97.4%	4.2%
Alachlor	107.8%	6.8%	97.5%	3.4%	96.9%	2.4%	102.0%	1.4%	100.9%	1.7%	97.2%	2.0%
Aldrin	109.8%	7.7%	105.3%	7.6%	93.5%	2.4%	107.7%	2.0%	98.7%	1.6%	100.4%	2.8%
Allethrin (Bioallethrin)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	105.7%	1.2%	102.1%	2.2%
Allidochlor	113.1%	10.1%	93.4%	12.6%	104.7%	12.9%	99.6%	1.9%	88.9%	5.3%	100.4%	4.8%
Anthraquinone	36.3%	27.8%	102.4%	14.5%	39.6%	6.5%	94.6%	3.5%	34.1%	47.7%	95.8%	2.7%
Atrazine	111.1%	4.1%	94.4%	6.0%	90.9%	4.0%	98.7%	5.0%	98.7%	0.9%	98.4%	2.4%
Azinphos-ethyl	95.2%	6.4%	86.1%	2.8%	96.3%	3.3%	92.4%	2.0%	100.9%	3.3%	93.3%	1.9%
Azinphos-methyl	84.5%	4.3%	87.3%	11.4%	87.1%	7.1%	100.2%	0.5%	94.3%	6.3%	84.1%	6.9%
BHC, Alpha	102.0%	3.6%	95.0%	5.6%	94.3%	3.6%	104.2%	3.2%	94.1%	1.9%	99.8%	4.2%
BHC, Beta	98.3%	2.3%	96.3%	7.7%	94.8%	0.9%	96.7%	6.3%	97.7%	1.0%	98.2%	1.9%
BHC, delta	98.5%	1.8%	93.2%	3.2%	95.1%	2.8%	101.1%	0.8%	96.5%	1.5%	97.2%	1.8%
BHC, gamma	99.5%	4.2%	96.3%	5.2%	90.7%	2.9%	109.3%	3.1%	97.4%	2.0%	96.4%	3.2%
Bifenthrin	106.1%	2.2%	106.5%	2.5%	99.9%	1.5%	101.8%	0.9%	99.5%	0.3%	102.6%	1.3%
Biphenyl	<LOQ	<LOQ	<LOQ	<LOQ	86.4%	10.5%	118.1%	3.0%	79.5%	7.7%	111.6%	7.1%
Bromfenvinphos	111.2%	3.2%	97.0%	2.7%	91.8%	1.9%	103.6%	2.8%	97.3%	1.7%	94.6%	2.0%
Bromfenvinphos-methyl	110.5%	3.4%	87.8%	5.1%	92.3%	2.5%	106.4%	2.3%	98.4%	0.9%	95.2%	3.6%
Bromophos-ethyl	113.2%	2.4%	87.2%	6.8%	93.7%	7.1%	113.1%	3.6%	97.2%	3.0%	94.8%	3.4%
Bromophos-methyl (Bromophos)	104.0%	4.3%	90.0%	7.6%	93.2%	2.9%	114.8%	2.5%	95.9%	2.6%	97.3%	3.2%
Bromopropylate	101.2%	14.2%	97.2%	5.3%	96.1%	2.0%	111.1%	2.6%	97.1%	2.3%	97.4%	2.3%
Bupirimate	111.7%	11.6%	98.6%	5.9%	99.7%	2.4%	101.4%	0.7%	100.9%	2.0%	97.1%	2.1%
Cadusafos	101.9%	3.4%	101.4%	3.8%	96.4%	3.8%	101.9%	1.8%	97.6%	1.7%	101.4%	3.1%
Captan	85.0%	16.5%	80.4%	9.1%	96.7%	11.9%	78.8%	2.4%	97.6%	5.6%	87.5%	4.2%
Carbophenothion	103.2%	4.3%	99.3%	3.6%	95.6%	1.5%	96.6%	1.9%	99.2%	1.0%	97.1%	1.5%
Carfentrazon-ethyl	103.9%	11.0%	104.7%	7.6%	94.2%	4.3%	109.0%	3.8%	99.3%	2.7%	94.8%	3.3%
Chlorbenside	88.8%	4.8%	91.1%	4.0%	88.1%	0.8%	94.0%	2.5%	88.0%	6.6%	96.3%	1.8%
Chlordane alpha-cis	110.5%	4.8%	96.1%	2.9%	95.4%	3.0%	100.1%	4.1%	99.3%	3.1%	98.5%	1.7%
Chlordane gamma-trans	112.4%	4.5%	109.7%	5.3%	95.9%	1.9%	116.2%	2.9%	97.3%	1.9%	105.9%	1.8%
Chlorfenapyr	96.9%	6.2%	96.5%	6.0%	97.2%	3.2%	95.4%	4.0%	99.7%	2.3%	99.9%	2.4%
Chlorfenson	97.9%	2.5%	95.3%	3.5%	92.9%	1.8%	94.8%	0.4%	93.1%	2.2%	94.4%	3.7%
Chlorfenvinphos	105.9%	4.3%	92.1%	3.7%	99.5%	3.3%	104.5%	1.6%	100.6%	0.7%	95.7%	2.0%
Chlorobenzilate	102.5%	2.3%	95.7%	4.2%	95.5%	0.9%	95.0%	2.3%	97.4%	0.8%	98.1%	1.5%
Chloroneb	102.6%	4.3%	97.6%	5.5%	99.9%	6.3%	105.6%	4.8%	89.0%	4.6%	102.0%	6.6%
Chlorothalonil	34.1%	8.8%	30.7%	16.9%	35.8%	29.2%	36.4%	19.1%	38.7%	8.0%	52.5%	9.1%
Chlorpropham	92.2%	4.9%	99.7%	4.3%	92.3%	3.7%	92.0%	3.6%	93.1%	2.5%	94.3%	2.7%
Chlorpyrifos-ethyl	100.1%	5.6%	99.5%	6.2%	94.1%	2.1%	101.7%	1.2%	96.7%	1.2%	97.1%	1.2%
Chlorpyrifos-methyl	113.9%	2.8%	88.1%	5.5%	93.3%	5.8%	104.9%	2.5%	95.3%	1.9%	98.0%	2.7%
Chlorthal-dimethyl (Dacthal)	117.1%	4.7%	96.9%	4.0%	93.1%	1.5%	109.9%	5.4%	98.0%	1.4%	94.8%	3.8%
Chlorthiophos	110.4%	3.5%	102.1%	7.0%	96.4%	3.3%	103.8%	0.9%	100.4%	2.0%	96.7%	1.5%
Chlozolinate	117.5%	8.4%	99.7%	8.6%	94.6%	4.1%	98.4%	1.9%	100.6%	1.8%	97.3%	3.2%
Clomazone	99.1%	3.6%	98.3%	4.0%	95.0%	2.8%	96.8%	0.8%	97.8%	0.9%	99.0%	2.9%
Coumaphos	98.6%	6.7%	93.0%	3.5%	88.5%	5.6%	91.4%	2.3%	94.9%	3.7%	92.5%	4.2%
Cycloate	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	114.7%	6.3%	115.2%	9.0%

Appendix B (Part 2). QuEChERS Recovery data.

Component Name	Carrot 1 µg/kg (n=6)		Apple 1 µg/kg (n=6)		Carrot 2.5 µg/kg (n=6)		Apple 2.5 µg/kg (n=3)		Carrot 10 µg/kg (n=6)		Apple 10 µg/kg (n=6)	
	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)
Cyfluthrin peak 1	109.7%	3.2%	103.2%	7.3%	106.0%	3.1%	96.1%	16.7%	106.3%	2.0%	103.6%	2.6%
Cyfluthrin peak 2	95.5%	3.5%	98.5%	2.5%	104.1%	1.7%	103.0%	1.4%	105.7%	2.6%	101.2%	2.3%
Cyfluthrin peak 3	107.8%	4.8%	105.7%	7.2%	103.3%	3.5%	97.5%	2.5%	104.7%	2.2%	101.1%	2.1%
Cyfluthrin peak 4	110.3%	4.9%	98.3%	6.4%	100.5%	2.4%	99.9%	1.8%	105.3%	2.6%	103.3%	2.7%
Cyhalothrin I (lambda)	106.2%	4.5%	100.3%	3.6%	102.8%	3.4%	99.5%	2.4%	104.3%	1.8%	104.5%	1.8%
Cypermethrin peak 1	80.2%	3.6%	94.8%	2.4%	94.8%	4.0%	101.6%	1.2%	101.1%	2.8%	101.2%	2.2%
Cypermethrin peak 2	87.0%	10.2%	80.2%	6.7%	104.7%	2.8%	98.0%	0.8%	106.7%	4.1%	105.1%	1.9%
Cypermethrin peak 3	102.0%	5.6%	99.0%	4.9%	109.1%	1.3%	101.9%	2.7%	105.6%	3.6%	107.0%	2.4%
Cypermethrin peak 4	102.1%	2.9%	102.8%	5.9%	102.2%	4.0%	101.8%	0.5%	107.5%	3.2%	107.1%	3.1%
Cyprodinil	103.5%	3.2%	93.5%	2.1%	96.3%	2.8%	99.2%	2.6%	92.8%	4.8%	98.7%	2.7%
DDD p,p	101.1%	1.9%	102.0%	2.4%	96.6%	1.2%	99.2%	0.8%	99.1%	1.3%	97.3%	1.5%
DDD, o, p	104.1%	2.2%	104.0%	4.1%	95.5%	1.8%	101.4%	1.3%	98.5%	1.3%	96.3%	1.6%
DDE o,p	109.1%	1.7%	97.2%	2.5%	96.9%	0.9%	103.9%	2.9%	99.7%	1.1%	99.0%	1.7%
DDE p, p	106.9%	1.7%	99.3%	4.0%	96.2%	1.5%	97.8%	2.4%	98.8%	1.1%	97.5%	1.5%
DDT o,p	101.4%	2.8%	100.2%	4.5%	95.1%	3.3%	96.4%	2.9%	100.7%	0.8%	97.4%	1.8%
DDT p,p	97.9%	2.6%	97.0%	0.8%	95.4%	2.0%	97.3%	2.1%	100.7%	0.8%	96.6%	1.5%
Deltamethrin	77.2%	5.2%	95.2%	7.0%	114.9%	6.0%	100.0%	8.5%	110.6%	2.3%	101.2%	7.4%
Diazinon	102.0%	3.5%	103.9%	4.4%	102.3%	2.9%	97.4%	4.0%	99.0%	2.1%	100.9%	3.1%
Dichlobenil	97.2%	6.4%	94.6%	10.5%	98.9%	7.3%	103.4%	5.6%	86.1%	5.6%	102.5%	6.5%
Dichlofluanid	58.8%	4.1%	56.8%	4.9%	50.7%	13.9%	62.5%	7.6%	57.5%	3.0%	74.9%	5.3%
Dichlorobenzophenone, 4, 4	115.3%	2.7%	99.1%	4.1%	97.3%	1.8%	95.2%	1.4%	98.0%	2.7%	94.2%	1.7%
Dicloran (Bortran)	86.6%	6.0%	84.9%	6.8%	86.6%	3.8%	91.7%	4.1%	86.4%	3.7%	88.8%	2.4%
Dicofol	34.9%	15.0%	69.0%	12.1%	113.2%	5.2%	92.3%	9.6%	105.9%	15.6%	101.3%	11.5%
Dieldrin	104.9%	17.3%	98.9%	13.4%	103.0%	6.4%	105.5%	3.2%	96.6%	3.2%	95.4%	3.0%
Dimethachlor	102.2%	3.2%	94.3%	1.8%	94.3%	1.7%	98.5%	3.2%	98.6%	2.4%	96.9%	1.2%
Dimethoate	97.7%	10.4%	71.9%	8.5%	87.0%	10.1%	77.3%	1.3%	94.9%	1.8%	85.3%	5.8%
Diphenamid	102.6%	6.1%	82.9%	4.4%	100.8%	1.9%	104.7%	2.3%	99.8%	1.8%	98.8%	1.2%
Diphenylamine	98.1%	2.1%	113.2%	7.1%	97.4%	4.2%	107.7%	4.5%	94.3%	2.0%	102.3%	4.3%
Disulfoton	96.5%	4.0%	94.3%	8.8%	95.8%	1.5%	100.1%	5.1%	98.3%	1.6%	97.4%	2.5%
Edifenphos	96.1%	3.3%	93.5%	2.6%	96.6%	1.5%	92.5%	1.8%	100.3%	2.2%	96.9%	2.0%
Endosulfan ether	105.3%	5.6%	98.8%	3.0%	95.7%	2.6%	101.5%	3.0%	94.5%	1.5%	97.8%	3.0%
Endosulfan peak 1	101.7%	6.6%	107.1%	6.2%	94.8%	2.8%	116.7%	4.6%	99.0%	2.1%	104.7%	2.8%
Endosulfan peak 2	97.9%	6.1%	95.6%	5.4%	100.7%	5.3%	98.2%	2.9%	98.0%	1.8%	101.6%	2.1%
Endosulfan sulfate	101.2%	4.9%	106.0%	8.3%	100.0%	2.3%	108.2%	0.7%	100.5%	1.6%	100.8%	1.5%
Endrin	105.6%	3.5%	93.4%	10.7%	99.3%	2.1%	99.7%	1.2%	98.0%	1.5%	96.8%	2.8%
Endrin Aldehyde	55.6%	14.3%	27.6%	26.3%	40.0%	18.9%	27.2%	28.2%	37.6%	13.2%	29.0%	16.7%
Endrin-Ketone	91.5%	17.1%	105.2%	10.9%	84.3%	16.2%	116.0%	4.9%	96.0%	2.3%	97.3%	3.0%
EPN	116.0%	9.0%	96.9%	7.3%	92.6%	2.2%	87.9%	6.2%	95.5%	2.7%	93.4%	2.8%
Ethion	107.0%	3.7%	98.3%	2.3%	96.8%	1.4%	97.8%	1.4%	100.0%	1.2%	99.3%	1.5%
Ethoprop (Ethoprophos)	97.2%	4.3%	91.4%	4.9%	96.5%	2.6%	96.0%	3.8%	95.0%	3.0%	97.0%	3.8%
Etofenprox	107.0%	4.6%	109.1%	4.3%	106.7%	2.2%	96.3%	5.0%	104.9%	1.6%	110.6%	2.3%
Etridiazole (Terrazole)	100.0%	7.6%	91.9%	11.5%	100.5%	7.2%	102.8%	6.3%	87.1%	6.6%	100.3%	7.4%
Fenamiphos	101.5%	8.3%	87.9%	10.1%	92.1%	4.7%	71.8%	8.3%	97.9%	1.4%	84.7%	3.7%
Fenarimol	107.9%	3.7%	101.3%	1.6%	104.5%	1.4%	102.5%	1.7%	100.3%	0.8%	99.8%	1.1%
Fenchlorfos	112.6%	3.0%	97.7%	3.8%	95.3%	2.6%	103.0%	3.2%	98.1%	2.1%	101.1%	2.3%
Fenitrothion	101.7%	9.5%	79.5%	9.1%	87.0%	6.1%	97.9%	2.8%	91.9%	1.0%	85.0%	2.8%
Fenpropathrin	97.2%	6.0%	80.4%	5.8%	102.5%	6.0%	97.5%	2.7%	98.1%	1.3%	103.4%	1.6%

Appendix B (Part 3). QuEChERS Recovery data.

Component Name	Carrot 1 µg/kg (n=6)		Apple 1 µg/kg (n=6)		Carrot 2.5 µg/kg (n=6)		Apple 2.5 µg/kg (n=3)		Carrot 10 µg/kg (n=6)		Apple 10 µg/kg (n=6)	
	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)
Fenson	98.9%	2.5%	95.5%	3.8%	96.5%	2.2%	96.0%	2.0%	98.8%	1.1%	97.0%	2.0%
Fenthion	109.0%	4.5%	92.3%	2.3%	100.4%	2.7%	108.7%	1.9%	99.6%	2.2%	95.1%	2.2%
Fenvalerate	101.3%	2.9%	102.6%	4.2%	110.1%	2.6%	104.6%	2.6%	110.3%	1.8%	104.2%	4.0%
Fipronil	103.5%	7.8%	83.8%	8.6%	94.9%	3.7%	110.6%	4.4%	100.5%	2.8%	95.9%	3.3%
Fipronil desulfinyl	101.5%	8.4%	92.2%	8.3%	95.2%	5.4%	108.4%	2.1%	99.5%	1.8%	98.5%	3.6%
Fluazifop-P-butyl	111.0%	3.9%	96.2%	6.6%	95.8%	2.2%	100.8%	2.4%	99.7%	2.3%	97.3%	1.4%
Fludioxonil	105.1%	3.2%	93.7%	3.7%	89.3%	4.5%	93.9%	5.0%	92.3%	3.3%	89.6%	1.6%
Fluquinconazole	103.1%	16.6%	102.9%	7.0%	94.3%	3.0%	91.1%	3.9%	98.2%	2.3%	99.4%	3.0%
Fluridone	100.7%	17.2%	85.6%	11.3%	92.1%	4.2%	107.2%	0.4%	100.9%	2.3%	88.7%	1.5%
Flusilazole	95.2%	5.4%	98.4%	9.7%	96.9%	1.9%	92.0%	2.1%	95.6%	1.7%	98.8%	2.1%
Flutolanil	99.0%	2.7%	95.1%	1.9%	95.0%	0.9%	96.7%	0.6%	97.0%	0.8%	99.1%	1.6%
Flutriafol	96.9%	2.5%	99.4%	5.4%	93.3%	2.2%	99.8%	2.4%	95.6%	1.4%	97.8%	1.0%
Fluvalinate peak 1	100.3%	5.9%	115.1%	18.5%	118.9%	3.6%	98.5%	3.5%	120.0%	2.4%	100.6%	3.2%
Fluvalinate peak 2	93.7%	17.3%	108.4%	18.8%	117.6%	5.8%	104.1%	8.2%	113.6%	3.9%	101.9%	5.2%
Folpet	90.2%	11.4%	72.2%	4.1%	86.6%	10.8%	70.7%	7.1%	97.0%	4.2%	77.3%	4.4%
Fonofos	105.9%	3.9%	99.9%	4.6%	99.5%	2.1%	105.4%	4.8%	97.1%	2.7%	101.3%	2.5%
Heptachlor	108.7%	5.4%	97.1%	5.1%	95.9%	3.4%	105.2%	3.6%	94.8%	0.9%	98.9%	3.5%
Hexachlorobenzene	95.1%	7.1%	94.6%	8.3%	86.7%	2.9%	109.1%	4.6%	75.4%	9.7%	99.3%	5.4%
Hexazinone	89.1%	1.9%	87.7%	2.1%	93.5%	1.0%	91.6%	0.9%	95.0%	1.7%	93.9%	2.0%
Iodofenfos	97.6%	4.6%	96.9%	3.2%	83.3%	6.7%	96.7%	0.8%	93.5%	4.2%	94.6%	4.3%
Iprodione	95.2%	16.3%	99.6%	7.6%	97.3%	5.5%	108.6%	1.2%	107.3%	3.8%	90.5%	2.9%
Isazophos	111.1%	9.4%	72.2%	5.0%	105.2%	4.0%	101.5%	2.1%	98.7%	1.5%	99.1%	2.5%
Isodrin	103.3%	4.9%	98.0%	4.6%	96.0%	1.9%	100.4%	4.8%	97.2%	0.8%	98.7%	2.0%
Lenacil	90.0%	10.8%	92.5%	8.2%	93.8%	6.0%	88.3%	4.6%	94.0%	5.3%	92.8%	2.0%
Leptophos	98.9%	6.8%	101.1%	1.8%	97.9%	1.3%	95.0%	1.1%	97.8%	4.0%	102.4%	1.1%
Linuron	104.4%	9.9%	82.4%	3.2%	92.9%	4.7%	97.2%	7.8%	90.9%	3.6%	99.7%	4.5%
Malathion	103.0%	2.1%	82.6%	3.7%	91.5%	2.1%	91.4%	5.1%	94.3%	1.4%	97.6%	1.3%
Metalaxyl	106.0%	6.0%	99.8%	12.5%	96.2%	4.8%	111.5%	1.8%	98.5%	1.9%	96.0%	2.3%
Metazachlor	98.5%	5.0%	99.8%	4.9%	97.4%	1.3%	100.4%	0.2%	98.7%	0.9%	97.2%	1.6%
Methacrifos	95.6%	4.0%	95.7%	5.5%	101.9%	6.6%	103.1%	4.0%	92.6%	3.8%	102.5%	6.6%
Methoxychlor	95.5%	2.3%	97.0%	5.3%	98.2%	2.0%	102.6%	1.7%	102.0%	0.9%	98.9%	1.2%
Metolachlor	98.9%	2.9%	93.3%	2.4%	94.2%	1.1%	96.7%	3.1%	97.8%	1.5%	97.3%	1.2%
Mevinphos	94.2%	4.6%	89.4%	5.4%	99.3%	7.7%	94.4%	4.3%	104.1%	3.6%	95.9%	5.3%
MGK-264 A	109.5%	16.6%	96.3%	10.5%	102.3%	6.8%	94.6%	0.6%	100.0%	2.4%	97.3%	1.9%
MGK-264 B	105.9%	4.6%	100.4%	6.1%	96.7%	2.9%	105.4%	3.4%	97.1%	1.5%	100.8%	2.2%
Mirex	97.6%	4.0%	103.6%	2.2%	93.9%	1.8%	102.0%	1.0%	102.5%	1.7%	99.3%	1.4%
Myclobutanil	100.6%	4.5%	97.6%	5.4%	96.7%	2.7%	101.5%	2.8%	98.1%	2.0%	96.5%	2.1%
N-(2,4-Dimethylphenyl) formamide	86.6%	5.6%	85.0%	8.4%	90.0%	7.6%	77.6%	3.0%	89.8%	5.0%	86.8%	3.0%
NDBA	110.7%	7.6%	110.9%	10.8%	105.2%	9.0%	116.5%	7.8%	90.0%	7.3%	104.8%	6.4%
NDEA	94.9%	7.2%	91.1%	11.1%	97.3%	10.2%	105.3%	5.2%	97.0%	12.2%	101.6%	13.7%
NDPA	97.2%	8.1%	74.8%	13.5%	97.3%	7.7%	90.5%	11.7%	85.4%	9.4%	101.3%	8.4%
NEMA	87.5%	4.1%	85.2%	19.4%	91.7%	15.3%	104.7%	15.3%	83.1%	12.4%	97.2%	8.2%
Nitrofen	109.8%	9.3%	97.4%	2.2%	90.9%	3.3%	93.7%	1.7%	92.8%	1.5%	87.9%	3.2%
N-Nitrosodiphenylamine	98.1%	2.1%	113.2%	7.1%	97.4%	4.2%	107.7%	4.5%	94.3%	2.0%	102.3%	4.3%
N-Nitrosomorpholine	84.9%	8.2%	80.7%	10.3%	85.1%	8.4%	80.5%	3.5%	81.8%	6.9%	91.8%	6.8%
N-Nitrosopiperidine	95.5%	7.2%	81.0%	19.8%	105.0%	11.2%	98.9%	10.5%	86.5%	8.2%	102.3%	6.6%
N-Nitrosopyrrolidine	95.4%	11.0%	114.0%	13.8%	101.6%	6.4%	91.4%	14.2%	82.5%	6.1%	99.4%	9.7%

Appendix B (Part 4). QuEChERS Recovery data.

Component Name	Carrot 1 µg/kg (n=6)		Apple 1 µg/kg (n=6)		Carrot 2.5 µg/kg (n=6)		Apple 2.5 µg/kg (n=3)		Carrot 10 µg/kg (n=6)		Apple 10 µg/kg (n=6)	
	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)
N-Nitrosopyrrolidine	95.4%	11.0%	114.0%	13.8%	101.6%	6.4%	91.4%	14.2%	82.5%	6.1%	99.4%	9.7%
Nonachlor-cis	109.4%	15.3%	114.4%	8.8%	94.6%	10.4%	113.1%	7.2%	100.5%	2.5%	98.9%	4.1%
Nonachlor-trans	107.8%	3.7%	103.7%	8.7%	96.1%	5.1%	107.3%	2.2%	99.4%	2.1%	101.0%	2.6%
Norflurazon	100.4%	7.8%	83.9%	17.3%	93.7%	3.5%	96.7%	1.8%	95.5%	1.8%	91.8%	2.7%
Ortho-phenylphenol	90.6%	5.1%	115.5%	3.5%	82.3%	6.2%	102.6%	2.1%	90.9%	3.2%	99.7%	5.4%
Oxadiazon	100.3%	4.3%	97.7%	4.6%	95.2%	1.9%	92.9%	1.1%	96.9%	2.1%	95.5%	3.6%
Oxyfluorfen	102.4%	15.7%	113.2%	8.9%	97.7%	7.1%	101.6%	4.5%	95.6%	2.0%	97.0%	3.2%
Paclobutrazol	111.2%	2.9%	93.8%	4.4%	98.3%	1.9%	97.4%	2.0%	95.1%	3.9%	94.6%	1.9%
Parathion (ethyl)	118.4%	7.4%	111.1%	5.7%	96.0%	4.0%	111.5%	4.5%	92.0%	3.4%	90.4%	1.8%
Parathion-methyl	119.6%	6.7%	83.7%	4.1%	94.0%	2.5%	95.8%	1.1%	91.0%	3.3%	86.9%	2.7%
Pebulate	112.9%	4.6%	118.8%	5.6%	113.9%	7.6%	119.3%	0.3%	93.4%	5.4%	109.2%	6.9%
Penconazole	100.3%	4.7%	96.3%	3.5%	94.4%	1.7%	100.1%	5.3%	98.1%	0.9%	94.4%	2.7%
Pentachloroaniline	94.9%	2.8%	91.8%	4.3%	85.1%	4.0%	104.1%	0.3%	84.6%	8.2%	96.1%	2.7%
Pentachloroanisole	104.3%	4.1%	92.8%	4.9%	94.8%	4.3%	104.6%	3.7%	90.6%	2.4%	102.5%	5.6%
Pentachlorobenzene	100.1%	4.0%	94.5%	6.3%	95.8%	5.6%	117.9%	6.9%	83.2%	4.4%	100.9%	8.9%
Pentachlorobenzonitrile	102.5%	5.7%	91.4%	3.3%	95.2%	6.4%	103.3%	1.9%	87.7%	2.7%	96.6%	3.5%
Pentachlorothioanisole	106.7%	6.3%	97.4%	3.2%	86.8%	3.4%	106.5%	0.2%	82.8%	9.7%	98.0%	2.3%
Permethrin peak 1	91.6%	13.6%	81.8%	6.2%	82.8%	8.1%	98.9%	2.0%	89.4%	2.3%	97.4%	2.1%
Permethrin peak 2	97.6%	7.0%	98.7%	3.2%	96.2%	2.6%	98.9%	0.3%	100.4%	2.5%	104.8%	2.0%
Perthane (Ethylan)	109.8%	3.1%	101.1%	2.4%	96.6%	0.7%	103.1%	0.8%	101.0%	0.8%	99.4%	1.4%
Phenothrin	<LOQ	<LOQ	<LOQ	<LOQ	89.7%	14.9%	81.7%	13.3%	108.4%	11.3%	92.2%	4.2%
Phorate	116.6%	15.9%	103.1%	7.0%	102.5%	4.8%	103.3%	3.0%	98.9%	3.0%	101.7%	4.8%
Phosalone	100.2%	3.7%	97.2%	4.6%	98.4%	1.8%	70.4%	13.3%	100.4%	2.1%	99.1%	1.2%
Phosmet	85.2%	3.8%	89.6%	6.9%	89.3%	3.6%	89.7%	2.2%	95.3%	3.3%	88.7%	6.4%
Piperonyl butoxide	105.0%	6.8%	99.7%	2.4%	103.1%	1.3%	99.9%	0.5%	102.2%	1.2%	103.8%	1.8%
Pirimiphos-ethyl	104.9%	4.3%	107.5%	4.0%	100.2%	2.0%	102.9%	4.2%	100.0%	2.5%	98.1%	2.8%
Pirimiphos-methyl	117.2%	5.1%	97.0%	4.8%	93.6%	1.9%	110.7%	4.2%	99.1%	1.6%	94.0%	4.0%
Pretilachlor	103.3%	4.1%	95.3%	5.0%	94.6%	2.0%	98.4%	5.2%	99.4%	1.0%	98.1%	1.2%
Prochloraz	104.5%	6.2%	110.6%	12.3%	115.0%	7.9%	98.9%	2.3%	99.3%	4.1%	92.0%	3.9%
Procymidone	115.0%	2.8%	100.8%	5.4%	96.4%	3.0%	103.3%	1.5%	98.7%	1.5%	99.0%	3.2%
Profenofos	114.4%	7.1%	99.0%	6.8%	86.5%	4.3%	99.9%	4.2%	95.7%	3.5%	91.6%	3.0%
Propachlor	94.4%	8.4%	104.0%	2.1%	99.3%	7.2%	99.9%	2.8%	92.7%	1.3%	97.7%	4.6%
Propanil	89.6%	7.5%	78.8%	10.2%	91.3%	5.6%	91.6%	2.3%	95.1%	4.2%	89.5%	2.1%
Propargite	<LOQ	<LOQ	<LOQ	<LOQ	89.1%	7.3%	101.7%	4.9%	103.2%	11.1%	102.5%	5.2%
Propisochlor	97.5%	7.0%	99.2%	3.6%	101.7%	3.2%	99.2%	1.5%	101.3%	3.0%	99.1%	1.6%
Propyzamide	99.8%	6.1%	102.1%	8.9%	100.7%	1.5%	99.5%	2.7%	101.6%	1.3%	99.6%	2.4%
Prothiofos	115.8%	4.8%	92.3%	2.1%	92.1%	3.5%	105.4%	4.7%	96.0%	1.2%	98.2%	4.0%
Pyraclofos	96.9%	8.3%	89.6%	7.2%	92.3%	3.6%	87.6%	1.8%	94.1%	6.6%	94.1%	4.1%
Pyrazophos	110.6%	2.6%	96.0%	3.0%	93.7%	3.8%	97.5%	1.9%	101.9%	3.1%	101.1%	1.4%
Pyridaben	106.8%	2.7%	100.0%	2.7%	98.9%	1.7%	119.5%	1.0%	100.1%	2.1%	102.8%	1.8%
Pyridaphenthion	94.2%	18.0%	96.3%	8.6%	96.3%	3.3%	113.7%	2.6%	100.4%	1.9%	95.9%	2.2%
Pyrimethanil	118.4%	7.6%	80.4%	13.3%	117.0%	9.1%	104.7%	9.1%	101.3%	2.7%	95.8%	2.8%
Pyriproxyfen	103.9%	4.5%	105.7%	1.4%	99.5%	2.9%	80.0%	7.3%	101.2%	1.3%	102.9%	1.6%
Quinalphos	95.2%	4.3%	86.3%	11.5%	78.9%	3.7%	85.1%	4.9%	100.0%	2.3%	95.5%	2.1%
Quintozene	107.3%	8.7%	101.9%	6.1%	97.6%	5.6%	94.6%	2.2%	88.9%	1.6%	94.3%	4.0%
Resmethrin peak 1	<LOQ	<LOQ	<LOQ	<LOQ	94.7%	2.9%	104.0%	7.1%	96.3%	3.7%	87.5%	2.0%
Resmethrin peak 2	<LOQ	<LOQ	<LOQ	<LOQ	92.9%	7.7%	95.4%	4.3%	94.4%	2.6%	89.0%	6.0%

Appendix B (Part 5). QuEChERS Recovery data.

Component Name	Carrot 1 µg/kg (n=6)		Apple 1 µg/kg (n=6)		Carrot 2.5 µg/kg (n=6)		Apple 2.5 µg/kg (n=3)		Carrot 10 µg/kg (n=6)		Apple 10 µg/kg (n=6)	
	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)	Mean Recovery	Precision RSD(%)
Sulfotep	103.2%	2.1%	101.1%	5.1%	96.7%	3.6%	101.8%	0.9%	96.7%	2.5%	100.6%	3.6%
Sulprofos	105.8%	2.6%	98.4%	4.3%	94.2%	1.4%	96.3%	3.0%	101.8%	1.3%	99.7%	1.1%
Tebuconazole	99.4%	3.2%	94.7%	4.5%	94.7%	1.2%	96.6%	1.8%	96.6%	0.6%	95.8%	1.5%
Tebufenpyrad	101.5%	4.7%	105.1%	2.2%	98.2%	1.5%	108.3%	0.7%	104.7%	2.0%	103.8%	1.4%
Tecnazene	100.9%	7.5%	94.3%	9.4%	94.2%	5.0%	104.3%	1.3%	86.9%	3.7%	100.4%	6.5%
Tefluthrin	103.2%	2.2%	101.7%	4.5%	99.0%	2.4%	104.5%	1.5%	100.1%	1.1%	99.1%	2.5%
Terbacil	98.7%	4.7%	89.0%	2.5%	94.6%	2.1%	89.2%	2.9%	95.1%	2.6%	94.1%	3.6%
Terbufos	109.8%	5.4%	97.9%	5.7%	98.0%	2.5%	107.3%	1.4%	98.8%	1.9%	104.3%	3.4%
Terbuthylazine	107.3%	6.2%	99.8%	4.3%	92.7%	4.0%	96.4%	4.9%	101.2%	3.9%	96.6%	3.0%
Tetrachlorvinphos	104.6%	4.4%	86.0%	5.3%	96.5%	3.5%	117.6%	1.7%	102.2%	4.7%	94.1%	1.6%
Tetradifon	92.1%	4.9%	105.0%	9.5%	94.4%	2.2%	99.1%	3.9%	97.5%	2.2%	100.8%	1.9%
Tetrahydrophthalimide (THPI)	88.8%	4.7%	87.6%	4.5%	98.6%	3.8%	89.3%	4.2%	93.8%	1.1%	88.3%	3.3%
Tetramethrin peak 1	<LOQ	<LOQ	<LOQ	<LOQ	108.2%	15.9%	79.5%	12.3%	95.1%	2.7%	96.5%	3.4%
Tetramethrin peak 2	93.4%	6.4%	118.7%	11.5%	96.9%	2.7%	96.1%	3.0%	100.7%	0.8%	97.8%	1.5%
Tolclofos-methyl	111.6%	3.3%	103.2%	5.3%	97.3%	2.5%	106.7%	2.2%	98.1%	1.8%	99.1%	1.9%
Tolylfluanid	70.7%	3.5%	66.5%	5.9%	64.9%	8.7%	70.0%	3.6%	71.7%	2.7%	79.9%	4.4%
Triadimefon	108.2%	5.2%	94.9%	5.8%	97.3%	2.7%	99.0%	0.3%	96.7%	0.7%	97.6%	2.0%
Triadimenol	109.3%	5.6%	105.8%	4.3%	95.9%	2.4%	98.5%	1.8%	99.1%	1.1%	98.4%	4.6%
Triallate	110.7%	2.5%	98.8%	4.6%	97.6%	3.1%	103.0%	3.0%	96.6%	0.6%	101.0%	3.4%
Triazophos	99.1%	4.9%	92.7%	4.1%	97.4%	2.2%	91.5%	1.4%	98.1%	1.6%	95.9%	1.7%
Tricyclazole	88.2%	17.8%	71.2%	5.0%	81.3%	9.1%	78.4%	6.3%	78.1%	6.0%	80.0%	6.3%
Triflumizole	102.5%	8.0%	89.7%	6.8%	96.0%	4.6%	94.4%	4.7%	100.4%	3.1%	101.7%	1.8%
Vinclozolin	106.6%	7.2%	100.3%	6.7%	99.1%	2.3%	98.5%	6.4%	98.2%	1.6%	97.6%	2.5%

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