

Food safety

Increasing productivity in pesticide residues analysis by GC-MS/MS with on-line automated micro solid phase extraction (μ SPE) clean-up

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Keywords

Pesticides, QuEChERS,
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gas chromatography, TriPlus RSH
SMART autosampler

Goal

The aims of this study were to evaluate the overall performance, productivity, and cost savings benefits of using an automated on-line system for the clean-up of QuEChERS extracts of food samples, including the automated preparation of calibration solutions, for pesticide residues analysis by GC-MS/MS.

Introduction

The QuEChERS (quick, easy, cheap, effective, rugged, and safe) extraction method followed by manual dispersive solid phase extraction (dSPE) clean-up was developed two decades ago¹ and is now the most common methodology applied in pesticide residues laboratories. Initially, the method was dedicated to the analysis of pesticides in fruit and vegetables but has since been adapted for other matrices and analytes.² The method is fast and straightforward, but dSPE is not always effective at removing sufficient matrix co-extractives which can quickly contaminate the instrument system and create unwanted downtime for testing laboratories. Additionally, dSPE is a manual multi-step procedure which can be considered for automation to save time.

On-line micro solid phase extraction (μ SPE) is a very interesting alternative for automating the dSPE approach. Application of μ SPE brings substantial improvements to the laboratory workflow since more intimate contact between the sorbent blend, contained in a small cartridge, and the sample extract, along with controlled elution speed, result in cleaner extracts. Reduction of the matrix load improves the robustness of the GC-MS analysis, prolongs column and liner lifetimes increasing the maintenance intervals, and most of all, eliminates interferences that can negatively affect the identification and quantitation of the pesticides. The μ SPE clean-up can be fully automated, so the

analyst only has to transfer the raw extract into the injection vials and to place these vials in the autosampler rack. The remaining procedure is carried out by the robotic arm of the autosampler, resulting in improved repeatability and a substantially reduced risk of human errors.

The main aim of this technical note is to demonstrate the performance of automated sample clean-up in analysis of pesticide residue in fruit and vegetables and discuss the benefits of an automated approach. An extended automated workflow including the calibration set-up is also described.

Experimental

Instrumentation

The on-line automated sample clean-up and sample injection were performed with a Thermo Scientific™ TriPlus™ RSH SMART autosampler (Figure 1). Subsequent analysis of pesticides in the cleaned-up extracts was performed using a Thermo Scientific™ TSQ™ 9610 triple quadrupole GC-MS/MS, where the Thermo Scientific™ TRACE™ 1610 GC connected to the TSQ 9610 mass spectrometer was equipped with a Thermo Scientific™ iConnect™ HeSaver-H₂Safer™ SSL Injector module. The HeSaver-H₂Safer technology decouples the gas used for pressurizing the inlet from the actual carrier gas, which in turn is supplied only to the

column for the gas chromatographic separation of the analytes. This technology can be used in conjunction with helium or hydrogen carrier gas and decreases the carrier gas consumption considerably. The pressurizing gas can be an inexpensive and easier-to-handle alternative (i.e., nitrogen or argon), used for the split and purge flows, which represent the major gas consumption, while it is prevented from entering the column during separation. When the HeSaver-H₂Safer technology is used with helium as carrier gas, as in this application, it brings significant cost savings and extended cylinder lifetime. The use of hydrogen as carrier gas is suitable for pesticide analysis, as demonstrated in a previous application note.³ However, for evaluating the impact of the automated clean-up workflow, helium carrier gas was used to ensure the optimum analytical performance.

The TSQ 9610 GC-MS/MS was operated in timed-SRM mode. All the transitions were carefully optimized with Thermo Scientific™ AutoSRM software to obtain the highest possible sensitivity. The GC-MS method details are listed in Table 1; the transitions along with collision energies and retention times can be found in Appendix 1. For the data acquisition and data processing, the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software (version 7.3.2) was used.

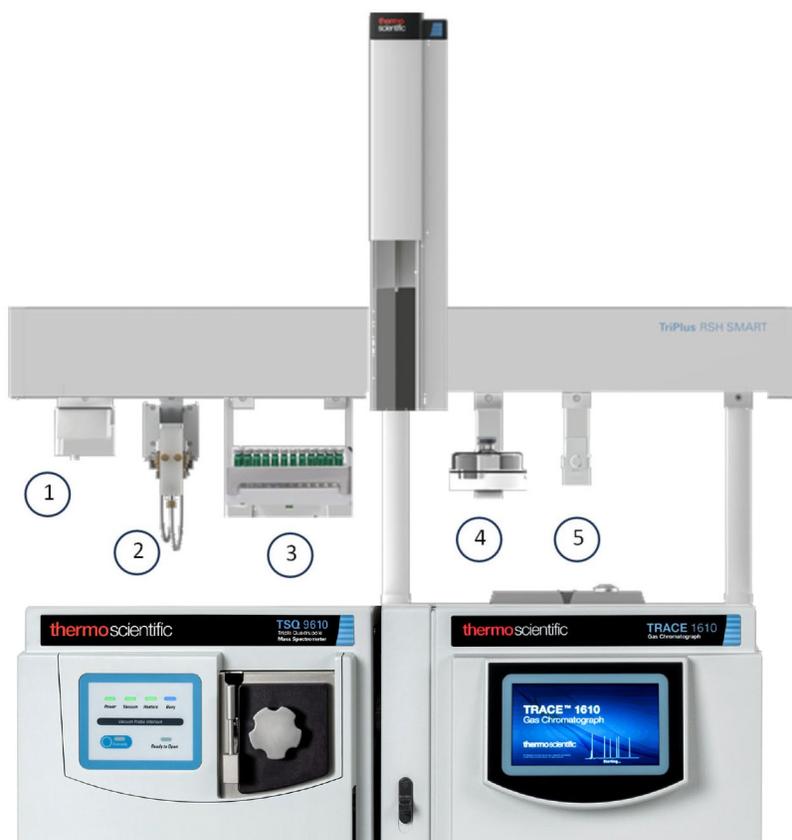


Figure 1. TriPlus RSH SMART autosampler configuration for μ SPE clean-up and automated calibration set-up: 1) automatic tool change station; 2) fast wash module; 3) μ SPE tray holder; 4) solvent station; 5) standard wash station. The detailed configuration is reported in Appendix 3.

Table 1. GC-MS method parameters

TRACE 1610 GC	
Injector	
Injector type	HeSaver-H ₂ Safer SSL Injector Module
Temperature [°C]	260
Pressuring gas	Nitrogen
Injection mode	Splitless
Split flow [mL/min]	50 (Nitrogen) after the injection
Splitless time [min]	1
Septum purge flow [mL/min]	5 (Nitrogen)
Vacuum compensation	On
Liner	Thermo Scientific™ LinerGOLD™ Single Taper Liner with Wool (P/N 453A1925-UI)
Injection volume [μL]	1
Carrier gas	Helium
Carrier gas flow [mL/min]	1.2
Oven	
Analytical column	Thermo Scientific™ TraceGOLD™ TG-5SiIMS with 5 m Safeguard (30 m × 0.25 mm × 0.25 μm) (P/N 26096-1425)
Oven temperature program	
Ready delay [min]	1.3
Temperature 1 [°C]	40
Hold [min]	1.5
Rate [°C/min]	25
Temperature 2 [°C]	90
Hold [min]	1.5
Rate [°C/min]	25
Temperature 3 [°C]	180
Hold [min]	0
Rate [°C/min]	5
Temperature 4 [°C]	280
Hold [min]	0
Rate [°C/min]	10
Temperature 5 [°C]	300
Hold [min]	5
TSQ 9610 GC-MS/MS	
Ion source	Advanced Electron Ionization (AEI) source
Transfer line temp. [°C]	300
Ion source temp. [°C]	320
Emission current [μA]	10
Electron energy [eV]	50

The compound identification criteria were taken from the DG SANTE guidance document.⁴ According to this document, two ion transitions are required to identify a given pesticide. The chromatographic peaks of those transitions must fully overlap, and signal-to-noise ratio must be equal or greater than 3. The ion ratio should be within ±30% (relative) of the average of the calibration standards and the retention time shift should not be greater than ±0.1 min.

Sample preparation

Samples (apple, tomato, lemon, and onion) were extracted using the QuEChERS method.⁵ In short, a 10 g portion of the previously homogenized sample was weighed in a 50 mL PTFE centrifuge tube. Subsequently, 10 mL of acetonitrile were added, and the samples were manually shaken for 5 min. Then, 4 g of magnesium sulfate, 1 g of sodium chloride, 1 g of trisodium citrate dihydrate, and 0.5 g of disodium hydrogen citrate sesquihydrate (Thermo Scientific™ QuEChERS EN 15662 Method Extraction Kit P/N S1-10-EN-KIT) were added, and the samples shaken again for 5 min. The extract was then centrifuged (3,700 rpm) for 5 min. A 1 mL volume of the supernatant was transferred to a standard 2 mL injection vial.

Blank samples spiked with pesticides standards were used to verify the accuracy and repeatability of the workflow. The spiking occurred before extraction, and samples were fortified with a mix of 190 pesticides to obtain concentration levels of 0.01 mg/kg and 0.05 mg/kg.

μSPE clean-up

The automated workflow for extract clean-up executed by the autosampler involved two steps. First, a cartridge (P/N 60101-45GC) containing a mixture of MgSO₄, PSA, C18, and GCB was conditioned with 100 μL of acetonitrile, and then a 300 μL aliquot of the raw extract was loaded onto the cartridge and eluted into an empty 2 mL vial. The syringe allows for controlled low flow rates in the load and elution phases for sharp analyte/matrix separation (Figure 2). Whereas a high proportion of the matrix co-extractives are retained in the cartridge, the pesticides elute with the process.

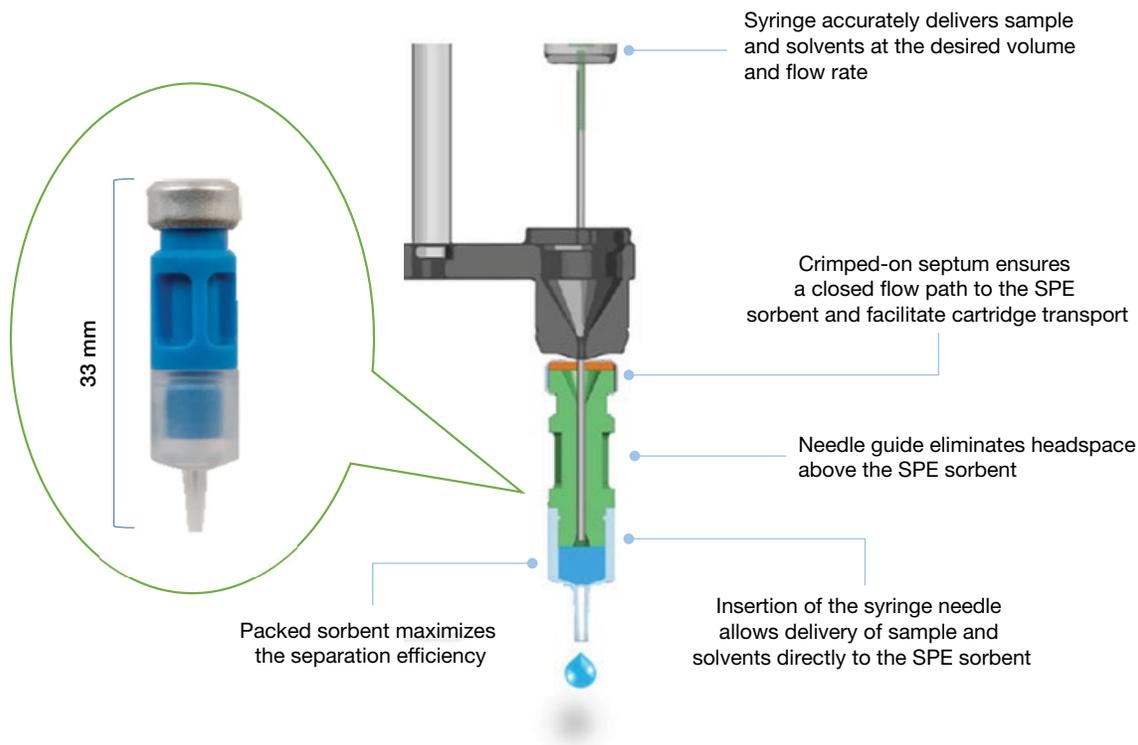


Figure 2. μ SPE cartridge and principle of operation

The collected effluent was subsequently injected into the GC-MS system. As none of the investigated analytes were completely trapped in the cartridge, no additional elution with solvent was applied, avoiding any sample dilution. Figure 3 summarizes the autosampler steps for the clean-up and injection workflow.



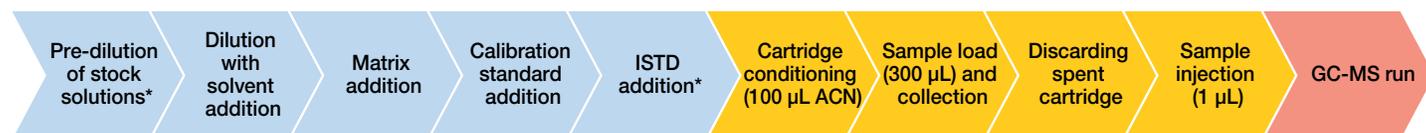
Figure 3. TriPlus RSH SMART extract clean-up and injection

Calibration

The semi-procedural calibration approach was applied, whereby matrix-matched calibration standards were prepared by spiking an aliquot of a blank extract with pesticides prior to μ SPE clean-up. Therefore, any losses of individual pesticides during the clean-up would be automatically corrected.

The use of a robotic autosampler allows automation of most of the sample handling procedures, including the dilution of the calibration standards. Vials containing the stock solution (10 mg/L) and blank matrix were placed in the autosampler rack. The robotic arm performed all the required dilution and the clean-up steps without any additional intervention from the analyst, as shown in the complete workflow represented in Figure 4.

The calibration workflow (applicable up to nine concentration levels) is automatically followed by the clean-up workflow of the samples to be quantified. The entire sequence is controlled by Chromeleon CDS.



*Optional step. The automated workflow offers the possibility to include the addition of the Internal Standard.

Figure 4. Calibration dilution workflow followed by μ SPE clean-up and injection of the standards

Results and discussion

Four commodities, commonly analyzed for pesticide residues on a large scale, were selected for this study. Tomato and apple can be considered easy, since they have a high water content and their raw extracts contain relatively low amounts of coextracted matrix compounds. In contrast, onion and lemon provided extracts with a much higher load of matrix co-extractives. To illustrate the effectiveness of the clean-up, a comparison of the total ion current chromatograms for apple, with and without the clean-up, is shown in Figure 5. The μ SPE clean-up provides a considerable reduction of background compounds even in a relatively simple matrix, improving the identification and confirmation of the target analytes.

The evaluation was focused on three critical analytical parameters: accuracy (expressed as recoveries), precision

(repeatability), and linearity. As mentioned previously, samples were spiked before the QuEChERS-based extraction, so that all results presented in the following are representative of the whole workflow (extraction, clean-up, and analysis).

The quantitative recovery of all pesticide residues under investigation was evaluated at 0.01 mg/kg and 0.05 mg/kg.

At both levels, almost all the evaluated compounds achieved recoveries in the 70–120% range, in compliance with the SANTE guidance criteria. The impact of the clean-up step on recoveries was compensated by using semi-procedural calibration. Matrix-matched calibration standards were run through μ SPE cartridges in the same manner as the samples. Figure 6 shows the summary for all matrices, whereas detailed recovery values can be found in Appendix 2.

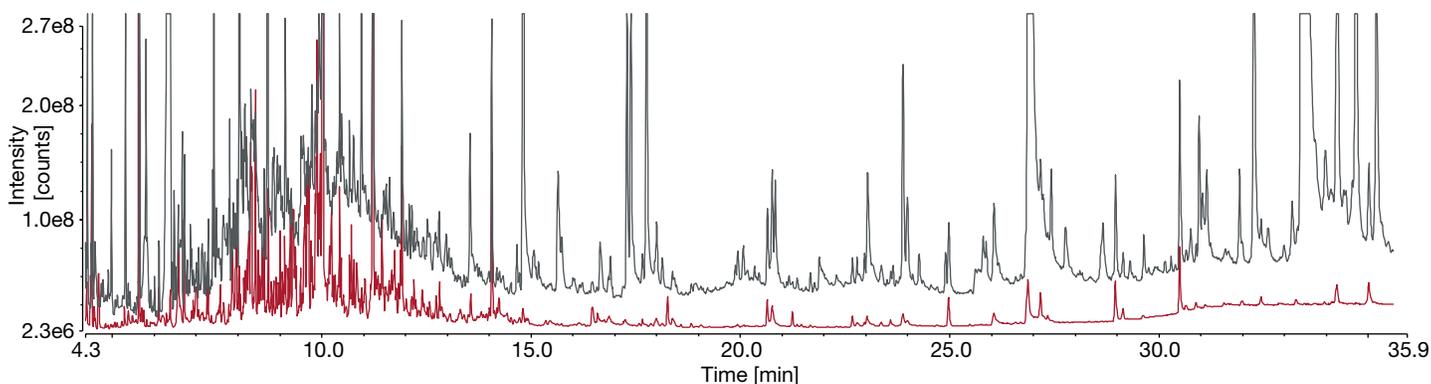


Figure 5. Comparison of total ion current chromatograms of the apple extract before (gray) and after (red) μ SPE clean-up

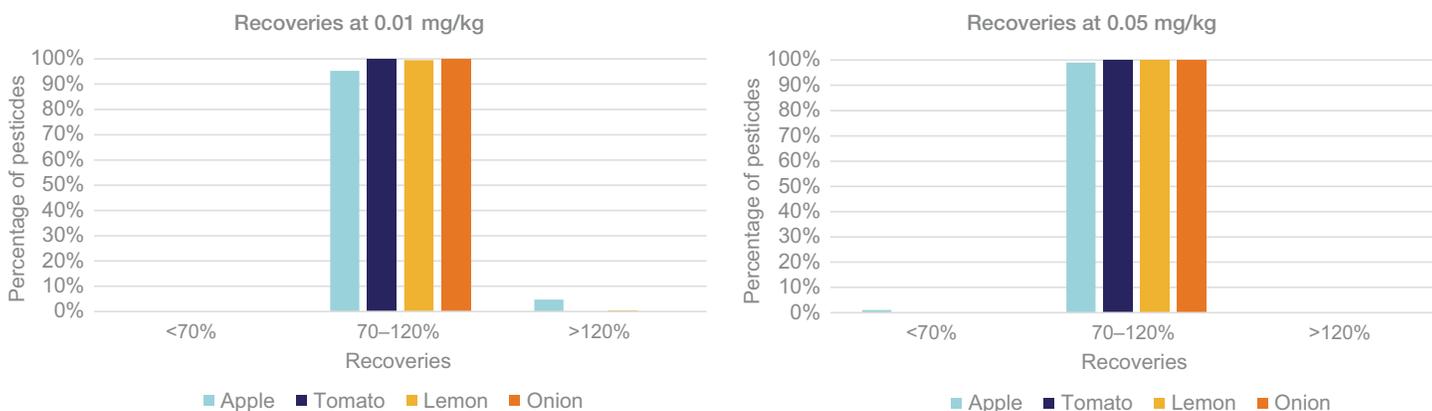


Figure 6. Analyte recovery at two concentration levels across all sample matrices investigated

As can be seen from the results displayed in Figure 7, compliance with the DG SANTE guidance document (RSD < 20%) was achieved for the majority of pesticide matrix combinations. Most pesticides (>97% of all investigated) show RSD of less than 15% at a concentration level of 0.01 mg/kg. Only in few cases (for example, allethrin in onion and in tomato; 3,4-dichloroaniline in onion; prochloraz in apple) the obtained values were above the acceptable threshold. Figure 7 illustrates the precision results in all analyzed matrices.

The linearity of the method was evaluated in the concentration range between 0.005 and 0.200 mg/kg. The response was considered linear when at least five points had the deviation of the back-calculated concentrations of less than 20%. As it is permitted by the SANTE guideline, weighting factors were used for a better adjustment of the equation to the measured peak areas. For all the investigated analyte/matrix combinations, the response was linear across the range, except for anthraquinone in tomato and apple, and parathion ethyl in apple. In these three cases, the signal was linear from 0.005 mg/kg to 0.100 mg/kg. Figure 8 shows heptachlor as a representative case of calibration curve.

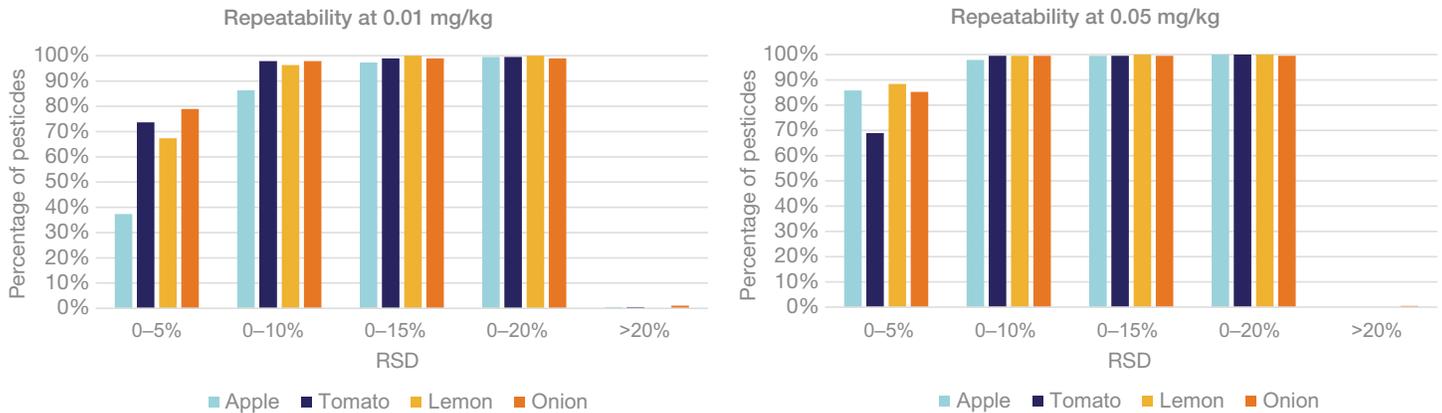


Figure 7. Summary of the repeatability (N = 5) study

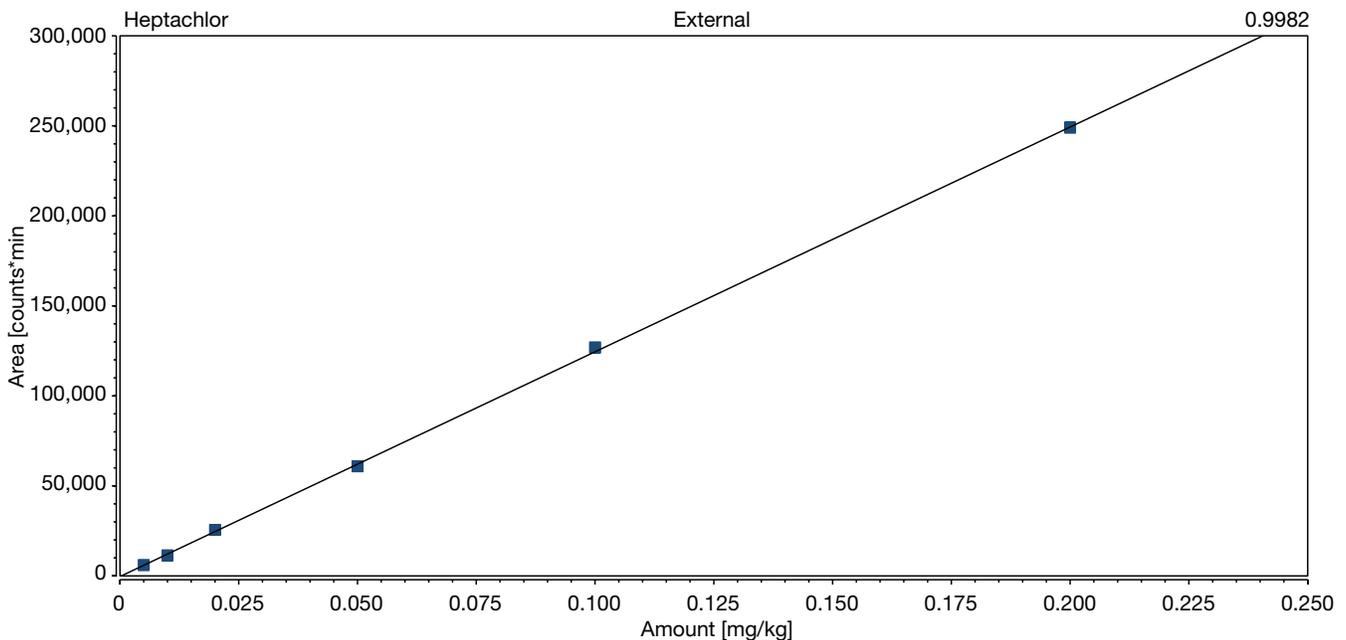


Figure 8. Calibration curve of heptachlor

Impact of automation on lab efficiency

Automating a manual workflow allows extension of unattended operations, running samples over night or over the weekend, with significant labor savings and better use of the analyst's time. The sample throughput can be optimized as well. The comparison reported in Figure 9 shows the reduction of two steps when μ SPE is used. Additionally, the entire instrumental cycle time is optimized thanks to the prep-ahead capability of the

TriPlus RSH SMART autosampler, allowing the extract clean-up to be executed during the chromatographic run of the previous sample.

The estimated labor time savings for the clean-up of 60 samples is about 1.5 hr, as shown in Figure 10. Additional manual labor time can be saved including the calibration standards preparation in the automated workflow, improving the overall efficiency of the laboratory with possible overnight operations.

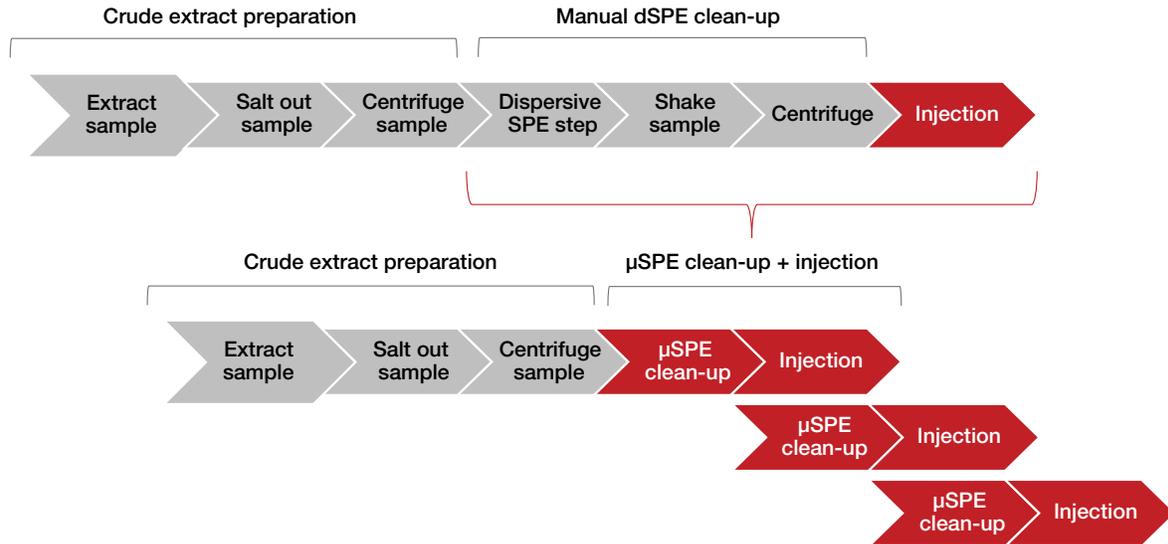


Figure 9. Steps reduction when automated μ SPE is used. After the first sample, the μ SPE clean-up is done during the run time of the previous sample, thanks to the prep-ahead function of the TriPlus RSH SMART autosampler, with additional cycle time optimization

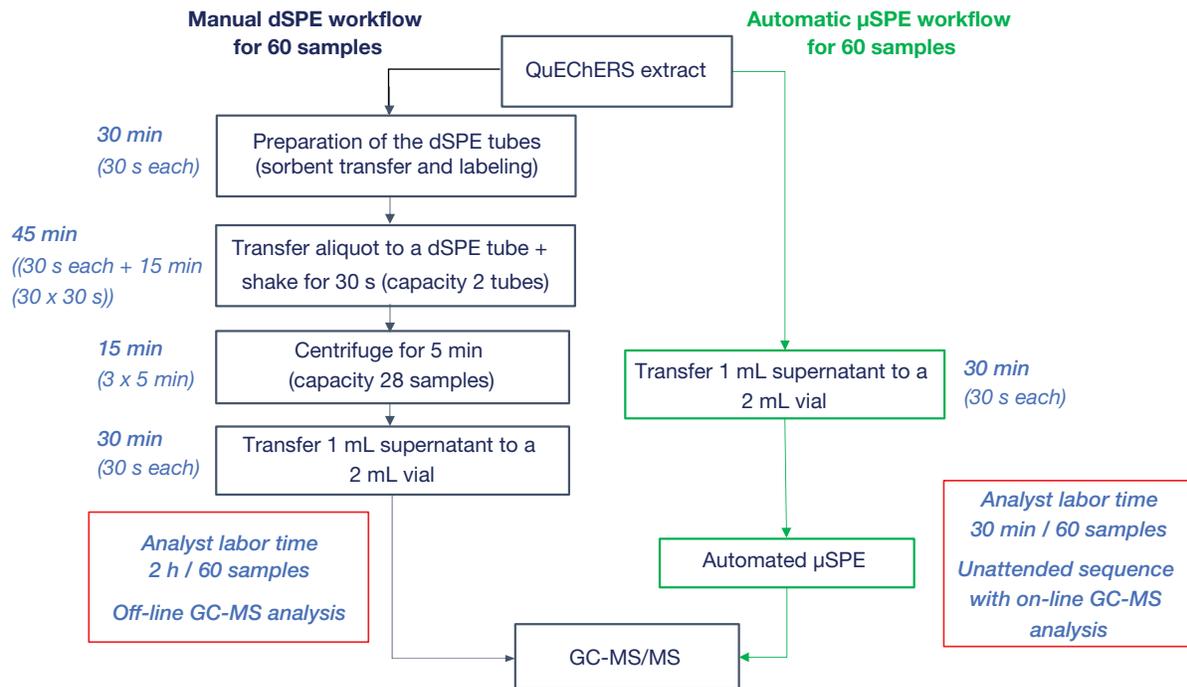


Figure 10. Estimated labor time savings for clean-up of 60 samples

Conclusion

The developed method for pesticides residues analysis in food using μ SPE as an automated clean-up step was demonstrated to be fully compliant with the DG SANTE guidance document in terms of accuracy, precision, and linearity. The automated clean-up workflow was used on-line with a highly sensitive GC-MS/MS system featuring the Advance EI source. Enhanced sensitivity permits the injection of less sample volume, maintaining the system cleanliness for longer and reducing maintenance. Nevertheless, the NeverVent technology available with the AEI source allows, when maintenance is needed, to operate very quickly, maximizing the uptime of the system. Additional benefits of the automated approach can be summarized as follows:

- The μ SPE provides a more efficient clean up than the manual dSPE, thanks to the controlled elution conditions.
- An automated workflow allows for unattended operations (overnight or over the weekend) and offers a significant labor time savings.
- Removing manual steps, especially for calibration standards preparation, minimizes errors and avoids potential costly efforts such as batch re-run.

References

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Appendix 1, part 1. Transitions, collision energies, and retention times

Name	RT	Transition 1			Transition 2			Transition 3		
		Mass	Product mass	Collision energy	Mass	Product mass	Collision energy	Mass	Product mass	Collision energy
Allidochlor	8.39	132	49	26	132	56.1	8	134	56	8
Dichlobenil	8.77	170.9	99.9	24	170.9	136	12	172.8	99.8	24
Biphenyl	9.1	151.8	125.8	24	154.1	115	26	155.1	154.1	12
Mevinphos	9.24	127	95	14	127	109	10	192	127	10
3,4-Dichloroaniline	9.4	160.9	90	18	160.9	99	20	160.9	126	10
Etridiazole (Terrazole)	9.5	182.8	139.9	14	211	139.9	18	211	182.9	10
Pebulate	9.55	128	72	6	128.1	57.1	8	161	128.1	6
N-(2,4-Dimethylphenyl)formamide	9.71	149.1	106.1	16	149.1	120.1	14	149.1	121.1	6
Methacrifos	9.79	124.9	47.1	12	125	79	8	207.9	180.1	6
Tetrahydrophthalimide (THPI)	9.79	151	77.1	32	151	79.9	6	151	122.1	8
Chloroneb	9.9	190.9	113	14	193	53.1	32	193	115	14
Ortho-phenylphenol	10.03	141.1	115.1	14	170.1	115	34	170.1	141.1	22
Pentachlorobenzene	10.08	247.9	212.9	18	248	142	42	249.8	214.8	16
Tecnazene	10.62	202.9	142.9	18	214.8	178.9	8	258.9	201	12
Propachlor	10.71	120	50.9	32	120	77	16	176.1	57.1	8
Diphenylamine	10.91	168.1	167.1	14	169.1	167.1	24	169.1	168.1	12
2,3,5,6-Tetrachloroaniline	10.96	230.8	157.9	18	230.8	159.8	18	230.8	194.8	10
Cycloate	10.99	154.1	55.1	18	154.1	83.1	8	215.1	154.1	6
Chlorpropham	11.1	127	65	20	171	127	8	213	127	14
Trifluralin	11.14	264	160	14	306.1	206	10	306.1	264.1	8
Benfluralin	11.17	292	160	20	292	206	12	292	264	8
Ethalfuralin	11.19	264	160	12	292	160	18	292	264	8
Sulfotep	11.24	202	145.9	10	237.9	145.9	12	322	145.9	22
Ethoprop (Ethoprophos)	11.435	157.9	96.9	16	157.9	113.9	6	200	158	6
Phorate	11.51	75	47	8	121	65	10	260	75	8
Omethoate	11.522	109.8	62.9	24	109.8	64	16	156	110	8
BHC, Alpha	11.65	180.9	144.9	14	216.9	181	8	218.9	183	8
Hexachlorobenzene	11.76	281.8	211.8	28	283.8	213.8	30	283.8	248.8	16
Pentachloroanisole	11.85	264.8	236.9	10	266.8	238.9	10	279.9	236.8	22
Dicloran (Bortran)	11.88	160	124.1	8	176	148	12	206	176	10
Cadusafos	11.94	159	96.9	16	159	130.9	8	213	89.1	12
Atrazine	12.05	200.1	122	8	200.1	132	8	215.1	58.1	10
BHC, Beta	12.09	180.9	145	14	216.9	180.9	8	218.9	183	8
Profluralin	12.24	318.1	55	12	318.1	199.1	12	347.1	330.1	6
Quintozene	12.25	213.8	141.9	28	213.8	178.9	14	294.8	236.9	14
Pentachlorobenzonitrile	12.29	272.9	237.9	16	274.8	204.9	28	274.8	239.9	18
BHC, gamma	12.32	180.9	144.9	12	216.9	180.9	8	218.9	183	8
Terbutylazine	12.33	214.1	104.1	16	214.1	132.1	10	229.1	173.1	10
Terbufos	12.36	231	128.9	20	231	175	10	231	203	8
Dimethoate	12.384	87	42.1	10	93	63	8	125	79	8
Propyzamide	12.42	172.9	109	24	172.9	145	14	174.9	147	14
Fonofos	12.47	109	62.9	10	137	109	6	246	109	14
Diazinon	12.48	137.1	54.1	20	137.1	84.1	12	199	92.9	14

Appendix 1, part 2. Transitions, collision energies, and retention times

Name	RT	Transition 1			Transition 2			Transition 3		
		Mass	Product mass	Collision energy	Mass	Product mass	Collision energy	Mass	Product mass	Collision energy
Fluchloralin	12.49	264	206	8	306	264	8	326	63	12
Pyrimethanil	12.61	198.1	118	32	198.1	158.1	18	198.1	183.1	14
Chlorothalonil	12.64	263.9	132.9	40	265.9	133	36	265.9	170	24
Terbacil	12.71	160	76	12	160	117	8	161	144	12
Clomazone	12.717	125	89	16	125	99	16	204	107	18
Disulfoton	12.72	88	45	18	88	59.8	6	142	81	10
Isazophos	12.73	118.9	76	18	160.9	119	8	256.9	161.9	4
Tefluthrin	12.75	177	127	14	177	137	16	197	141	10
BHC, delta	12.86	180.9	144.9	14	182.9	147	14	218.9	182.9	8
Triallate	12.9	86.1	43.3	6	268	183.9	18	268	226	12
Pentachloroaniline	13.33	262.9	191.9	20	264.9	193.6	18	266.9	193.9	20
Heptachlor	13.35	271.8	236.8	12	273.8	236.8	14	273.8	238.8	14
Endosulfan ether	13.37	238.9	204	12	240.9	206	14			
Propanil	13.45	161	90	24	161	99	24	217	161	8
Acetochlor	13.57	146.1	130	24	146.1	131	12	174.1	146.1	10
Chlorpyrifos-methyl	13.6	285.9	92.9	20	285.9	270.9	14	287.9	92.9	20
Vinclozolin	13.66	186.8	124	18	197.9	145	14	212	172	14
Parathion-methyl	13.72	233	109	10	263	79	30	263	109	12
Tolclofos-methyl	13.75	265	219.9	20	265	250	12	266.9	252	12
Alachlor	13.77	188.1	130	32	188.1	132	14	188.1	160.1	8
Propisochlor	13.88	162.1	91.1	30	162.1	120.1	12	162.1	144.1	8
Metalaxyl	13.92	234.1	146.1	20	234.1	174.1	10	249.1	190.1	6
Fenclorfos	14	284.9	93	24	284.9	269.9	14	286.9	271.9	14
Dimethachlor	13.37	134	77	24	134	105.1	12	197.1	148	10
Fipronil desulinyil	14.219	333.1	231.1	20	333.1	281.1	12	388	333.1	12
Pirimiphos-methyl	14.32	233	151.1	6	290.1	125	20	290.1	233	8
Prodiamine	14.35	275.1	255.1	8	321.1	203	10	321.1	279.1	6
Fenitrothion	14.38	277	109	16	277	109	14	277	260	6
Linuron	14.52	159.8	133	12	187	124	20	248	61.1	8
Dichlofluanid	14.58	123	77	18	224	123	10	226	123	10
Malathion	14.59	127	99	6	158	125	6	173.1	99	12
Pentachlorothioanisole	14.6	262.7	192.9	28	295.7	245.9	30	295.7	262.9	12
Metolachlor	14.75	162.1	133.1	14	238.1	133.1	26	238.1	162.1	10
Chlorpyrifos-ethyl	14.81	196.9	168.9	12	198.9	171	14	313.9	257.9	12
Fenthion	14.89	278	109	18	278	125	14	278	169	14
Chlorthal-dimethyl (Dacthal)	14.93	222.9	166.9	20	300.9	222.9	22	300.9	272.9	12
Anthraquinone	14.96	180.1	152	12	208.1	152	22	208.1	180.1	10
Aldrin	14.97	254.9	219.9	20	262.7	191	30	262.7	192.9	32
Parathion (ethyl)	14.97	138.9	109	6	154.9	125	6	291	109	12
Triadimefon	15.11	208	111	20	208	126.7	12	208	180.8	8
Dichlorobenzophenone, 4, 4	15.23	139	74.9	26	139	111	12	141	113	10
Fenson	15.36	141	50.9	30	141	77	8	268	77	20

Appendix 1, part 3. Transitions, collision energies, and retention times

Name	RT	Transition 1			Transition 2			Transition 3		
		Mass	Product mass	Collision energy	Mass	Product mass	Collision energy	Mass	Product mass	Collision energy
Pirimiphos-ethyl	15.39	304.1	168.1	12	318.1	166.1	12	318.1	182.1	10
Bromophos-methyl (Bromophos)	15.42	328.9	313.8	14	330.8	93	24	330.8	315.8	14
Diphenamid	15.43	167.1	152.1	16	167.1	165.1	20	239.1	167.1	8
MGK-264 A	15.46	164.1	80.1	24	164.1	93.1	10	164.1	98.1	10
Isopropalin	15.55	264.1	222.1	6	280.1	180.2	10	280.1	238.2	8
Isodrin	15.74	146.9	111.1	10	192.9	123	28	192.9	157	20
Pendimethalin	15.75	252.1	161.1	14	252.1	162.1	8	252.1	191.3	8
Metazachlor	15.78	132.1	117.1	14	133.1	132.1	12	209	132.1	16
Fipronil	15.81	366.9	212.9	28	366.9	244.9	20	368.9	214.9	30
Penconazole	15.91	159	123	20	248.1	157	22	248.1	192	12
Chlozolinate	15.92	186	145	14	188	147	14	259	187.9	12
Tolyfluanid	15.98	238	91	40	238	137	10	240	137	14
Bromfenvinphos-methyl	16.02	109	79	6	294.9	79.1	30	294.9	109	16
Allethrin (Bioallethrin)	16.17	107	79	6	107	91	10	123.1	41.1	24
Quinalphos	16.2	146	118.1	10	157.1	102	22	157.1	129	14
Captan	16.23	117	82	30	149	70	20	151	79	14
Procymidone	16.29	283	68.1	24	283	96.1	8	285	96.1	10
Triadimenol	16.31	112	58	8	128	65	18	128	100	10
Triflumizole	16.38	179	144	14	206	179	14	206	186	8
Folpet	16.41	104	76	10	130	102	12	259.9	130	14
Cyprodinil	16.458	224.1	197.1	20	224.1	208.1	18	225.1	210.1	16
MGK-264 B	16.519	164.1	67.1	6	164.1	80.1	22	164.1	98.1	12
Bromophos-ethyl	16.6	302.8	210.9	30	302.8	284.8	14	358.8	302.8	14
Chlorbenside	16.66	125	89	16	125	99	16	268	125	10
DDE o,p	16.78	246	176.1	28	248	176.1	30	317.9	248	18
Tetrachlorvinphos	16.8	328.9	109	18	330.9	109	18	332.9	109	14
Paclobutrazol	16.84	138	103.1	14	236	125	12	236	167	10
Chlordane gamma-trans	17.09	271.8	236.8	12	271.8	236.8	14	372.8	265.9	14
Endosulfan peak 1	17.11	194.9	160	8	240.8	205.8	14	262.8	192.9	30
Flutriafol	17.17	123	75	24	219.1	95	34	219.1	123	12
Chlorfenvinphos	17.18	266.9	159	16	268.9	161	14	323	266.9	14
Bromfenvinphos	17.2	266.9	159	14	268.9	161.1	14	323.1	266.9	10
Nonachlor-trans	17.2	271.8	236.8	14	406.8	299.8	14	408.8	299.8	18
Fenamiphos	17.23	154	139	10	217	202	12	303.1	195	8
Flutolanil	17.38	173	95	28	173	145	14	281	173	10
Iodofenfos	17.38	125	47	12	376.8	361.8	16	378.8	363.8	14
Chlorfenoson	17.4	111	75	14	175	75	28	175	111	10
Fludioxonil	17.44	248	127	26	248	154	18	248	182	10
Chlordane alpha-cis	17.456	271.9	236.9	14	372.8	265.9	20	374.8	265.9	20
Prothiofos	17.47	266.9	220.9	18	266.9	238.9	8	309	238.9	14
Tricyclazole	17.52	162	161	6	189	161	14	189	162	10
Pretilachlor	17.57	162	132.1	20	176.1	147.1	14	202.1	174.2	8
Profenofos	17.61	296.9	268.9	10	336.9	266.9	12	338.9	268.9	14

Appendix 1, part 4. Transitions, collision energies, and retention times

Name	RT	Transition 1			Transition 2			Transition 3		
		Mass	Product mass	Collision energy	Mass	Product mass	Collision energy	Mass	Product mass	Collision energy
DDE p, p	17.77	246	176.1	28	315.9	246	14	317.9	246	20
Oxadiazon	17.8	175	76	28	175	112	12	258	175	6
Myclobutanil	17.85	150	123	14	179	90	28	179	125	14
Dieldrin	17.91	262.9	190.9	30	262.9	192.9	30	262.9	227.8	16
Flusilazole	17.93	233.1	151.9	14	233.1	164.9	16	315.1	233.1	10
DDD, o, p	17.95	235	165	20	235	199	14	237	165	20
Oxyfluorfen	17.96	252	146	30	252	169.8	28	300	223	14
Bupirimate	18	273.1	108	14	273.1	193.2	8	316.2	208.1	10
Chlorfenapyr	18.3	136.9	102	12	248.9	112	24	248.9	137.1	18
Nitrofen	18.49	202	139	24	283	162	20	283	253	10
Fluazifop-P-butyl	18.54	282.1	91.1	18	282.1	238.1	16	383.1	282.1	14
Endrin	18.57	244.9	173	22	262.8	192.9	30	280.8	244.9	8
Perthane (Ethylan)	18.58	223.1	167	12	223.1	179	20	223.1	193	28
Chlorobenzilate	18.76	139	111	12	251	111	34	251	139	14
Endosulfan peak 2	18.84	158.9	123	12	194.9	159	8	236.8	118.9	30
Nonachlor-cis	19.05	262.9	192.9	28	406.8	299.9	14	408.8	299.9	18
Ethion	19.09	120.9	65	10	230.9	128.9	22	230.9	174.9	12
DDD p,p	19.1	235	165	20	235	199	14	237	165	20
DDT o,p	19.14	235	165.1	22	235	199.1	10	237	165.1	22
Chlorthiophos	19.19	268.9	205	14	296.9	268.9	8	324.9	268.9	12
Endrin Aldehyde	19.34	173	138.1	16	249.8	214.9	24	278.9	242.9	10
Triazophos	19.53	161.1	106.1	12	161.1	134.1	8	257	162.1	6
Sulprofos	19.6	156	108	30	156	141	14	322	156.1	10
Carfentrazon-ethyl	19.85	290	99.9	36	311.9	150.7	18	340.1	312.1	10
Carbophenothion	19.89	157	45	12	199	142.9	10	342	157	10
4,4'-Methoxychlor olefin	19.95	238.1	152.1	34	238.1	223.1	10	308	238.2	12
Edifenphos	19.97	172.9	65.1	30	172.9	109	8	310	109	26
Norflurazon	19.97	145	74.7	28	145	95	16	303	145	20
Endosulfan sulfate	20.1	238.7	203.9	12	271.7	234.9	12	271.7	236.8	12
Lenacil	20.11	153	82.1	16	153	110	14	153	135.6	12
DDT p,p	20.31	235	165.1	22	235	199.5	10	236.8	165	22
Hexazinone	20.46	127.7	83	10	171.1	71.1	14	171.1	85.1	12
Tebuconazole	20.72	125	89	16	125	99	16	250	125	20
Propargite	20.85	135.1	77.1	26	135.1	107.1	12	150.1	135.1	8
Triphenylphosphate	20.86	215	168.1	16	326.1	169.1	28	326.1	325.1	10
Piperonyl butoxide	21.08	176.1	103.1	22	176.1	117	18	176.1	131.1	12
Nitralin	21.09	274	169	12	274	216	8	316.2	274	8
Resmethrin peak 1	21.17	123.1	81.1	8	143	128.1	10	171	127.9	14
Resmethrin peak 2	21.18	123.1	81.1	8	143	128	10	171	127.9	14
2,4'-Methoxychlor	21.307	152	126.1	24	152	151.1	16	227.1	121.1	10
Endrin-Ketone	21.64	209.2	138.4	30	316.8	208.9	28	316.8	281	10
Iprodione	21.66	314	245	10	315.7	247	10	315.7	273	8
Captafol	21.776	183.1	79.1	8	150.1	79	6	151.1	79.1	18

Appendix 1, part 5. Transitions, collision energies, and retention times

Name	RT	Transition 1			Transition 2			Transition 3		
		Mass	Product mass	Collision energy	Mass	Product mass	Collision energy	Mass	Product mass	Collision energy
Phosmet	21.79	160	50.9	38	160	76.9	22	160	133	10
EPN	21.94	157	77	22	169	77	22	169	141	8
Bromopropylate	21.97	184.9	75.5	30	184.9	156.9	12	340.8	185	14
Bifenthrin	22.01	165.1	163.6	24	181	165.9	10	181	179	12
Tetramethrin peak 1	22.05	164	77.1	24	164	107.1	12	164	135.1	8
Tetramethrin peak 2	22.08	164	77.1	22	164	107.1	12	164	135.1	8
Methoxychlor	22.19	227.1	141.1	32	227.1	169.1	22	227.1	212.1	12
Fenpropathrin	22.33	181	126.8	28	181	151.9	22	199	77.1	24
Pyridaphenthion	22.372	199	77.1	24	199	92.1	14	340	199.1	8
Tebufenpyrad	22.51	276.1	171	10	318.1	131.1	14	318.1	145.1	14
Tetradifon	22.86	159	74.8	32	159	111	20	159	131	10
Phenothrin	23.02	123.1	41.1	24	123.1	79.1	14	123.1	81.1	8
Phosalone	23.05	121.1	65	10	182	74.8	30	182	111	14
Dicofol	23.098	111	74.9	12	139	111	12	250.9	139	12
Azinphos-methyl	23.18	132	77	12	160	50.9	34	160	77	16
Mirex	23.78	236.8	142.9	26	272	236.8	14	273.8	238.8	14
Acrinathrin	23.82	181	152	22	208.1	180.9	8	289	93.1	8
Leptophos	23.923	171	51	38	171	77.1	18	171	124.3	10
Pyrazophos	24.13	221	148.7	14	221	193.1	8	231.9	204.1	10
Pyriproxyfen	24.225	136.1	78	20	136.1	96	10	226.1	186.1	12
Azinphos-ethyl	24.3	132	51	26	132	77	12	160	77	16
Cyhalothrin I (lambda)	24.615	180.9	152	22	197.1	141.1	10	207.9	180.9	8
Pyraclufos	24.62	194	138	18	360	194.1	12			
Fenarimol	24.869	139	74.9	26	139	74.9	26	139	111	14
Permethrin peak 1	25.28	163	91.1	12	183.1	153	12	183.1	168	12
Coumaphos	25.45	209.9	119	22	209.9	182	10	226	163	18
Fluquinconazole	25.46	340	108.1	36	340	298	16	340	313	14
Permethrin peak 2	25.5	183	153	14	183	165.1	10	183	168.1	10
Pyridaben	26.33	147.1	117.1	20	147.1	119.1	8	147.1	132.1	12
Cyfluthrin peak 1	26.34	163	91.1	12	163	127.1	6	206	151.1	18
Prochloraz	26.434	180	69	14	180.1	138.1	12	308	70	12
Cyfluthrin peak 2	26.55	163	91.1	12	163	127	6	206	151.1	18
Cyfluthrin peak 3	26.65	163	91.1	12	163	127	6	226	206.1	12
Cyfluthrin peak 4	26.75	163	91.1	12	163	127	6	226	206.1	10
Cypermethrin peak 1	26.93	163	91.1	12	163	127.1	6	180.9	152.1	20
Cypermethrin peak 2	27.15	163	91.1	12	163	127	6	180.9	151.9	18
Cypermethrin peak 3	27.24	163	91	12	163	127	6	180.9	152.2	20
Cypermethrin peak 4	27.34	163	91.1	12	163	127.1	6	180.9	152.2	20
Etofenprox	27.55	163.1	77.1	32	163.1	107.1	16	163.1	135.1	10
Fluridone	27.87	328.1	189.1	38	328.1	258.8	24	329.1	328.5	12
Fenvalerate	28.59	125	89	18	167	89	32	167	125	10
Fluvalinate peak 1	28.91	180.8	152.1	22	250	55.1	16	250	199.9	18
Fluvalinate peak 2	29.05	180.8	152.1	20	250	55.1	16	250	200	16
Deltamethrin	29.93	181	152.1	22	252.8	92.9	16	252.8	172	8

Appendix 2A, part 1. Recovery data for apple

Apple	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Chlorpyrifos-ethyl	114%	99%	1%	3%
N-(2,4-Dimethylphenyl)formamide	109%	104%	1%	3%
Pentachlorobenzonitrile	108%	96%	1%	5%
Pyrimethanil	109%	96%	1%	2%
Chlorbenside	105%	97%	2%	3%
Tetrahydrophthalimide (THPI)	118%	101%	2%	5%
Isodrin	101%	95%	2%	2%
MGK-264 B	118%	97%	2%	4%
Tolclofos-methyl	108%	98%	3%	2%
Alachlor	119%	103%	3%	3%
Tetradifon	119%	98%	3%	3%
Isopropalin	101%	94%	3%	5%
Nonachlor-trans	111%	97%	3%	2%
Chlordane alpha-cis	114%	101%	3%	5%
DDD p,p	111%	100%	3%	2%
Mirex	110%	98%	3%	4%
Ethalfuralin	115%	97%	3%	3%
4,4'-Methoxychlor olefin	107%	95%	3%	2%
Chlorfenson	112%	96%	3%	3%
Bupirimate	112%	99%	3%	4%
Fluchloralin	111%	99%	3%	4%
Triazophos	120%	108%	4%	3%
Oxadiazon	117%	97%	4%	3%
Diphenylamine	106%	95%	4%	2%
BHC, gamma	116%	95%	4%	2%
Endrin Aldehyde	118%	107%	4%	6%
Chlordane gamma-trans	102%	94%	4%	3%
Ethion	120%	101%	4%	3%
Fenson	109%	96%	4%	3%
Methacrifos	113%	96%	4%	1%
Triallate	103%	95%	4%	4%
Phenothrin	120%	97%	4%	3%
Sulfotep	119%	99%	4%	2%
Penconazole	113%	98%	4%	4%
Chloroneb	105%	94%	4%	2%
EPN	120%	102%	4%	6%
Nitrofen	120%	99%	4%	3%
Fipronil	119%	105%	4%	4%
Pebulate	105%	94%	4%	3%
Dichlobenil	102%	94%	4%	2%
DDE o,p	112%	95%	4%	3%
DDT o,p	112%	99%	4%	2%
Chlorobenzilate	120%	99%	4%	3%
Tefluthrin	110%	96%	4%	3%
Chlorpyrifos-methyl	118%	99%	4%	2%
Perthane (Ethylan)	116%	99%	4%	2%
BHC, delta	107%	102%	4%	2%
Iodofenfos	120%	99%	5%	3%

Apple	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
DDE p, p	108%	98%	5%	3%
Dicloran (Bortran)	103%	97%	5%	4%
Etofenprox	118%	94%	5%	6%
2,3,5,6-Tetrachloroaniline	104%	94%	5%	3%
Phosalone	118%	101%	5%	3%
Procymidone	112%	98%	5%	1%
Carbophenothion	119%	102%	5%	4%
Bromophos-methyl (Bromophos)	114%	98%	5%	3%
Dichlorobenzophenone, 4, 4	106%	94%	5%	2%
Metazachlor	114%	103%	5%	3%
Propyzamide	109%	96%	5%	3%
Pyriproxyfen	119%	99%	5%	4%
BHC, Alpha	105%	95%	5%	2%
Ortho-phenylphenol	110%	98%	5%	2%
Piperonyl butoxide	120%	101%	5%	2%
Propanil	112%	104%	5%	4%
Aldrin	118%	91%	5%	2%
Pentachloroanisole	106%	95%	5%	2%
DDD, o, p	105%	96%	5%	3%
Diphenamid	110%	95%	5%	4%
Fenchlorfos	112%	98%	5%	3%
BHC, Beta	106%	98%	5%	2%
Chlorfenvinphos	128%	103%	5%	5%
Sulprofos	120%	97%	6%	5%
Acrinathrin	120%	101%	6%	4%
Fenamiphos	113%	99%	6%	7%
Fluquinconazole	116%	100%	6%	1%
Methoxychlor	120%	102%	6%	3%
Terbufos	113%	100%	6%	2%
Clomazone	109%	99%	6%	1%
Fenthion	128%	91%	6%	2%
Tetrachlorvinphos	113%	103%	6%	5%
Fludioxonil	109%	99%	6%	5%
Endrin	117%	103%	6%	2%
Disulfoton	112%	95%	6%	3%
2,4'-Methoxychlor	113%	99%	6%	3%
Phorate	115%	103%	6%	1%
Propargite	119%	107%	6%	4%
Endrin-Ketone	112%	96%	6%	3%
DDT p,p	116%	100%	6%	4%
Allidochlor	106%	98%	6%	2%
Bifenthrin	119%	100%	6%	5%
Chlorthal-dimethyl (Dacthal)	118%	95%	6%	2%
Fluazifop-P-butyl	119%	96%	6%	3%
Flutriafol	109%	102%	6%	4%
Fenpropathrin	111%	100%	6%	4%
Etridiazole (Terrazole)	109%	98%	6%	2%
Resmethrin	85%	67%	6%	4%

Appendix 2A, part 2. Recovery data for apple

Apple	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Nonachlor-cis	108%	95%	6%	1%
Parathion-methyl	110%	103%	7%	2%
Endosulfan sulfate	106%	102%	7%	3%
Pretilachlor	116%	97%	7%	4%
Benfluralin	109%	96%	7%	4%
Permethrin	118%	98%	7%	4%
Lenacil	127%	98%	7%	4%
Tebufenpyrad	111%	99%	7%	3%
Chlorthiophos	112%	100%	7%	5%
Trifluralin	112%	101%	7%	3%
Biphenyl	102%	92%	7%	3%
Prothiofos	117%	99%	7%	4%
Tricyclazole	118%	93%	7%	6%
Propachlor	114%	99%	7%	1%
Heptachlor	103%	97%	7%	1%
Pyridaben	120%	101%	7%	4%
Chlorpropham	112%	99%	7%	3%
Carfentrazon-ethyl	118%	102%	7%	3%
Pyridaphenthion	118%	104%	7%	5%
Mevinphos	115%	105%	7%	2%
Fenarimol	119%	97%	7%	4%
Terbutylazine	116%	97%	7%	4%
Edifenphos	120%	102%	7%	4%
Fonofos	108%	96%	7%	3%
Phosmet	120%	103%	7%	4%
Fluridone	119%	96%	7%	4%
Cyhalothrin I (lambda)	120%	98%	8%	4%
Atrazine	119%	100%	8%	3%
Cyprodinil	113%	98%	8%	5%
Acetochlor	109%	100%	8%	3%
Fenvalerate	120%	101%	8%	4%
Diazinon	120%	97%	8%	3%
Pentachlorobenzene	104%	91%	8%	3%
Hexazinone	109%	99%	8%	5%
Flusilazole	107%	105%	8%	4%
Cyfluthrin peak 1	107%	98%	8%	5%
Leptophos	120%	96%	8%	9%
MGK-264 A	99%	96%	8%	2%
Profluralin	119%	98%	8%	2%
Quintozene	102%	97%	8%	4%
Flutolanil	118%	98%	8%	3%
Bromfenvinphos	115%	103%	8%	2%
Dimethachlor	115%	101%	8%	3%
Metolachlor	111%	98%	8%	2%
Azinphos-methyl	120%	101%	8%	5%
Norflurazon	114%	105%	9%	2%
Endosulfan ether	107%	96%	9%	3%

Apple	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Quinalphos	120%	97%	9%	4%
Tebuconazole	119%	100%	9%	6%
Vinclozolin	108%	96%	9%	3%
Tetramethrin	117%	102%	9%	5%
Malathion	110%	95%	9%	2%
Chlorfenapyr	88%	92%	9%	6%
Myclobutanil	113%	102%	9%	3%
Deltamethrin	120%	102%	9%	4%
Profenofos	127%	103%	10%	6%
Pirimiphos-ethyl	117%	98%	10%	3%
Linuron	110%	90%	10%	4%
Fluvalinate	118%	102%	10%	5%
Pentachloroaniline	102%	97%	10%	5%
Paclobutrazol	115%	94%	10%	5%
Metalaxyl	104%	100%	10%	7%
Azinphos-ethyl	116%	102%	10%	6%
Pendimethalin	110%	96%	10%	4%
Propisochlor	111%	100%	10%	4%
Iprodione	115%	104%	10%	5%
Terbacil	120%	102%	10%	9%
Parathion (ethyl)	116%	88%	10%	5%
Tecnazene	115%	98%	11%	3%
Triadimenol	117%	102%	11%	7%
Chlozolinate	119%	96%	11%	2%
Bromfenvinphos-methyl	116%	103%	11%	3%
Bromopropylate	118%	103%	11%	4%
Pentachlorothioanisole	105%	96%	11%	9%
Cypermethrin peak 1	115%	93%	11%	7%
Endosulfan	112%	97%	11%	4%
Pirimiphos-methyl	110%	98%	11%	2%
Fenitrothion	117%	99%	11%	4%
Prodiamine	119%	96%	11%	3%
Triadimefon	116%	100%	11%	6%
Pyraclufos	119%	102%	12%	7%
Cycloate	113%	95%	12%	3%
Triflumizole	105%	98%	12%	7%
Isazophos	141%	99%	12%	8%
Coumaphos	117%	102%	13%	12%
Pyrazophos	120%	98%	13%	8%
3,4-Dichloroaniline	103%	60%	14%	17%
Dieldrin	110%	91%	14%	3%
Hexachlorobenzene	108%	93%	14%	9%
Oxyfluorfen	127%	93%	16%	8%
Bromophos-ethyl	113%	104%	16%	3%
Anthraquinone	155%	80%	18%	13%
Allethrin (Bioallethrin)	124%	105%	20%	9%
Prochloraz	124%	108%	36%	13%

Appendix 2B, part 1. Recovery data for tomato

Tomato	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Alachlor	98%	96%	5%	1%
Terbutylazine	101%	97%	5%	1%
BHC, Beta	104%	99%	3%	1%
Pentachloroaniline	96%	89%	3%	1%
Tolclofos-methyl	102%	97%	6%	2%
Endosulfan	100%	97%	6%	2%
Oxadiazon	96%	93%	2%	2%
DDE p, p	97%	97%	4%	2%
Ortho-phenylphenol	102%	101%	4%	2%
Metalaxyl	107%	96%	3%	2%
Chlzolinate	100%	97%	3%	2%
Fenson	96%	95%	3%	2%
Endosulfan ether	109%	98%	5%	2%
DDE o,p	106%	96%	1%	2%
Tetradifon	100%	94%	2%	2%
Chlorbenside	99%	98%	4%	3%
4,4'-Methoxychlor olefin	99%	93%	2%	3%
Dichlorobenzophenone, 4, 4	98%	95%	3%	3%
Clomazone	98%	95%	4%	3%
Chlordane gamma-trans	105%	96%	5%	3%
Diphenylamine	95%	92%	3%	3%
Paclobutrazol	99%	91%	7%	3%
Chloroneb	102%	99%	5%	3%
Endrin-Ketone	104%	96%	2%	3%
MGK-264 A	95%	91%	2%	3%
Diazinon	95%	88%	5%	3%
Mirex	98%	97%	2%	3%
BHC, gamma	91%	97%	7%	3%
Procymidone	95%	94%	2%	3%
Chlorthal-dimethyl (Dacthal)	107%	95%	6%	3%
DDD, o, p	98%	96%	5%	3%
Chlorfenson	98%	95%	2%	3%
Acetochlor	100%	95%	6%	3%
Isazophos	95%	90%	4%	3%
2,3,5,6-Tetrachloroaniline	93%	96%	7%	3%
Pyrimethanil	95%	92%	5%	3%
Vinclozolin	100%	98%	3%	3%
Pirimiphos-methyl	94%	93%	2%	3%
Pretilachlor	100%	94%	2%	3%
Triadimefon	84%	97%	6%	3%
Dimethachlor	96%	97%	3%	3%
DDT o,p	102%	95%	1%	3%
Endosulfan sulfate	99%	96%	4%	3%
MGK-264 B	101%	93%	7%	3%
Perthane (Ethylan)	100%	93%	2%	3%
Endrin	101%	93%	4%	3%
Aldrin	109%	99%	7%	3%
Nonachlor-trans	92%	95%	7%	4%

Tomato	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Sulfotep	101%	95%	4%	4%
Malathion	96%	91%	4%	4%
BHC, Alpha	103%	97%	4%	4%
Propachlor	99%	94%	4%	4%
Carbophenothion	99%	93%	3%	4%
DDD p,p	101%	95%	0%	4%
Atrazine	100%	94%	6%	4%
Fipronil	92%	96%	5%	4%
Hexazinone	96%	92%	3%	4%
BHC, delta	93%	92%	3%	4%
Sulprofos	102%	92%	2%	4%
Norflurazon	98%	89%	3%	4%
2,4'-Methoxychlor	99%	93%	2%	4%
Bromophos-methyl (Bromophos)	102%	97%	5%	4%
Dicloran (Bortran)	80%	86%	9%	4%
Chlorthiophos	101%	90%	3%	4%
Endrin Aldehyde	88%	106%	6%	4%
Terbufos	98%	97%	6%	4%
Fludioxonil	93%	94%	4%	4%
Isodrin	107%	99%	4%	4%
Permethrin peak 1	92%	89%	4%	4%
Diphenamid	98%	92%	6%	4%
Metazachlor	95%	95%	3%	4%
DDT p,p	102%	96%	3%	4%
Penconazole	88%	96%	6%	4%
Fonofos	91%	94%	5%	4%
Propyzamide	93%	94%	4%	4%
Propanil	101%	90%	4%	4%
Triflumizole	82%	91%	5%	4%
Chlorpyrifos-ethyl	93%	89%	6%	4%
Quintozene	91%	93%	4%	4%
Metolachlor	97%	92%	2%	4%
Cycloate	105%	98%	4%	4%
Methacrifos	101%	97%	4%	4%
Pirimiphos-ethyl	88%	87%	4%	4%
Phenothrin	93%	90%	2%	4%
Fenchlorfos	104%	94%	4%	4%
Ethion	98%	94%	2%	4%
Resmethrin	94%	86%	3%	4%
Carfentrazon-ethyl	99%	91%	3%	4%
Phosalone	90%	85%	3%	5%
Profenofos	102%	90%	7%	5%
Cyprodinil	89%	94%	6%	5%
Prothiofos	94%	91%	3%	5%
Fluquinconazole	85%	90%	3%	5%
Tetrahydrophthalimide (THPI)	89%	87%	6%	5%
Tefluthrin	104%	97%	4%	5%
Chlorpropham	101%	93%	6%	5%

Appendix 2B, part 2. Recovery data for tomato

Tomato	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Pyridaben	91%	89%	3%	5%
Fenitrothion	93%	85%	5%	5%
Fenarimol	88%	93%	2%	5%
Triallate	107%	95%	3%	5%
Triazophos	92%	89%	4%	5%
Bromfenvinphos	107%	92%	3%	5%
Bifenthrin	99%	89%	2%	5%
Prodamine	99%	90%	7%	5%
Parathion (ethyl)	99%	88%	5%	5%
Nonachlor-cis	96%	93%	3%	5%
Fluazifop-P-butyl	94%	94%	2%	5%
Nitrofen	86%	87%	4%	5%
Phorate	87%	100%	5%	5%
Methoxychlor	97%	90%	2%	5%
Chlorpyrifos-methyl	96%	90%	4%	5%
Chlorfenvinphos	102%	90%	5%	5%
Disulfoton	95%	92%	4%	5%
Propisochlor	101%	93%	3%	5%
Tebufenpyrad	97%	90%	3%	5%
Pentachlorobenzonitrile	100%	89%	6%	5%
Terbacil	112%	90%	7%	5%
Chlordane alpha-cis	95%	102%	9%	5%
Tetramethrin	95%	89%	4%	5%
Flutolanil	94%	91%	3%	5%
Fenpropathrin	96%	93%	5%	5%
Iodofenfos	104%	89%	5%	5%
Piperonyl butoxide	99%	88%	3%	5%
Etofenprox	71%	87%	2%	5%
Bupirimate	97%	91%	4%	5%
Heptachlor	106%	100%	5%	5%
Bromophos-ethyl	95%	91%	12%	5%
Cyhalothrin I (lambda)	94%	89%	1%	5%
Pyriproxyfen	90%	90%	3%	5%
Fluchloralin	83%	87%	8%	5%
Myclobutanil	88%	95%	2%	5%
Trifluralin	91%	95%	7%	6%
Chlorobenzilate	93%	92%	3%	6%
Ethalfuralin	91%	92%	6%	6%
Azinphos-ethyl	89%	85%	2%	6%
Chlorfenapyr	110%	97%	6%	6%
Oxyfluorfen	84%	86%	7%	6%
Fenvalerate	90%	90%	4%	6%
Pentachlorothioanisole	93%	98%	5%	6%
Cyfluthrin peak 1	90%	90%	3%	6%
Bromopropylate	96%	90%	1%	6%
Allethrin (Bioallethrin)	91%	92%	26%	6%
Fenthion	107%	93%	4%	6%

Tomato	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Cypermethrin peak 1	95%	85%	4%	6%
Allidochlor	99%	96%	2%	6%
Parathion-methyl	102%	87%	6%	6%
Quinalphos	100%	85%	7%	6%
Hexachlorobenzene	98%	96%	5%	6%
Pentachloroanisole	104%	98%	5%	6%
Iprodione	97%	92%	5%	6%
EPN	87%	79%	5%	6%
Tecnazene	93%	91%	8%	6%
Bromfenvinphos-methyl	98%	92%	5%	6%
Benfluralin	91%	92%	8%	6%
Leptophos	103%	89%	4%	6%
Phosmet	95%	84%	5%	6%
Fluvalinate	90%	86%	2%	6%
Dichlobenil	95%	92%	5%	7%
Flutriafol	89%	94%	5%	7%
Flusilazole	84%	90%	4%	7%
Propargite	105%	94%	6%	7%
Deltamethrin	91%	90%	2%	7%
Profuralin	102%	87%	6%	7%
N-(2,4-Dimethylphenyl)formamide	92%	89%	3%	7%
Acrinathrin	92%	89%	4%	7%
Lenacil	97%	88%	5%	7%
Tetrachlorvinphos	93%	91%	3%	7%
Tebuconazole	83%	92%	3%	7%
Edifenphos	98%	87%	3%	7%
Fenamiphos	90%	89%	4%	7%
Pendimethalin	102%	84%	3%	7%
Pyrazophos	89%	81%	2%	7%
Pyridaphenthion	88%	81%	4%	7%
Isopropalin	98%	95%	8%	8%
Mevinphos	95%	91%	5%	8%
Fluridone	72%	82%	3%	8%
Biphenyl	93%	94%	8%	8%
Azinphos-methyl	97%	82%	5%	8%
Pyraclufos	88%	79%	6%	8%
Coumaphos	89%	84%	4%	8%
Prochloraz	85%	84%	4%	8%
Pebulate	103%	94%	9%	8%
Etridiazole (Terrazole)	88%	97%	7%	8%
Dieldrin	100%	98%	8%	8%
Anthraquinone	81%	76%	18%	8%
Linuron	76%	78%	7%	9%
Pentachlorobenzene	101%	95%	7%	9%
Triadimenol	87%	94%	4%	9%
Tricyclazole	78%	89%	6%	10%
3,4-Dichloroaniline	91%	107%	13%	19%

Appendix 2C, part 1. Recovery data for onion

Onion	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
2,3,5,6-Tetrachloroaniline	91%	102%	4%	5%
2,4'-Methoxychlor	88%	104%	2%	3%
3,4-Dichloroaniline	88%	83%	29%	35%
4,4'-Methoxychlor olefin	90%	106%	4%	4%
Acetochlor	92%	105%	3%	4%
Acrinathrin	87%	106%	3%	4%
Alachlor	83%	105%	3%	3%
Aldrin	100%	105%	7%	5%
Allethrin (Bioallethrin)	100%	112%	28%	9%
Allidochlor	93%	103%	3%	4%
Anthraquinone	87%	108%	7%	9%
Atrazine	93%	108%	6%	3%
Azinphos-ethyl	87%	105%	3%	4%
Azinphos-methyl	85%	106%	3%	4%
Benfluralin	91%	104%	8%	3%
BHC, Alpha	95%	102%	6%	4%
BHC, Beta	91%	103%	3%	4%
BHC, delta	93%	105%	4%	4%
BHC, gamma	92%	101%	3%	6%
Bifenthrin	92%	107%	2%	3%
Biphenyl	96%	101%	4%	7%
Bromfenvinphos	90%	105%	6%	3%
Bromfenvinphos-methyl	88%	105%	6%	5%
Bromophos-ethyl	88%	103%	7%	5%
Bromophos-methyl (Bromophos)	94%	104%	5%	4%
Bromopropylate	91%	106%	2%	4%
Bupirimate	86%	109%	3%	3%
Carbophenothion	87%	104%	6%	3%
Carfentrazon-ethyl	92%	107%	3%	4%
Chlorbenside	91%	104%	2%	3%
Chlordane alpha-cis	93%	107%	10%	3%
Chlordane gamma-trans	92%	104%	3%	5%
Chlorfenapyr	88%	107%	7%	3%
Chlorfenson	95%	106%	3%	2%
Chlorfenvinphos	88%	106%	6%	4%
Chlorobenzilate	92%	105%	2%	4%
Chloroneb	94%	103%	3%	4%
Chlorpropham	98%	104%	4%	3%
Chlorpyrifos-ethyl	96%	105%	2%	4%
Chlorpyrifos-methyl	90%	105%	4%	3%
Chlorthal-dimethyl (Dacthal)	93%	107%	4%	3%
Chlorthiophos	90%	107%	3%	5%
Chlozolinate	97%	103%	3%	6%
Clomazone	96%	105%	1%	3%
Coumaphos	88%	109%	5%	7%
Cycloate	94%	103%	2%	4%
Cyfluthrin peak 1	91%	107%	4%	7%
Cyhalothrin I (lambda)	91%	105%	4%	4%

Onion	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Cypermethrin peak 1	89%	107%	4%	5%
Cyprodinil	89%	105%	3%	5%
DDD p,p	92%	106%	2%	3%
DDD, o, p	93%	105%	4%	4%
DDE o,p	89%	103%	3%	3%
DDE p, p	90%	103%	3%	3%
DDT o,p	91%	105%	3%	4%
DDT p,p	86%	105%	4%	4%
Deltamethrin	89%	107%	4%	5%
Diazinon	91%	101%	2%	5%
Dichlobenil	95%	102%	2%	5%
Dichlorobenzophenone, 4, 4	88%	104%	2%	3%
Dicloran (Bortran)	95%	105%	6%	2%
Dieldrin	98%	105%	3%	5%
Dimethachlor	90%	108%	3%	4%
Diphenamid	101%	107%	5%	5%
Diphenylamine	94%	104%	2%	4%
Disulfoton	88%	104%	3%	3%
Edifenphos	83%	106%	2%	4%
Endosulfan	99%	106%	5%	5%
Endosulfan ether	94%	106%	1%	4%
Endosulfan sulfate	89%	104%	4%	4%
Endrin	82%	100%	4%	3%
Endrin Aldehyde	95%	107%	11%	5%
Endrin-Ketone	93%	106%	8%	2%
EPN	94%	104%	7%	5%
Ethalfuralin	93%	103%	2%	3%
Ethion	91%	106%	3%	4%
Etofenprox	89%	109%	3%	5%
Etridiazole (Terrazole)	97%	100%	3%	6%
Fenamiphos	94%	101%	4%	7%
Fenarimol	91%	104%	5%	5%
Fenchlorfos	92%	106%	3%	4%
Fenitrothion	96%	109%	2%	4%
Fenpropathrin	87%	104%	3%	4%
Fenson	93%	104%	2%	3%
Fenthion	95%	104%	6%	4%
Fenvalerate	90%	107%	5%	5%
Fipronil	92%	105%	3%	4%
Fluazifop-P-butyl	85%	106%	4%	3%
Fluchloralin	98%	104%	7%	4%
Fludioxonil	91%	104%	3%	3%
Fluquinconazole	90%	104%	3%	4%
Fluridone	85%	95%	5%	9%
Flusilazole	93%	103%	6%	3%
Flutolanil	91%	104%	6%	4%
Flutriafol	91%	108%	2%	3%
Fluvalinate	85%	105%	5%	5%

Appendix 2C, part 2. Recovery data for onion

Onion	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Fonofos	92%	103%	4%	4%
Heptachlor	89%	100%	2%	5%
Hexachlorobenzene	95%	108%	3%	5%
Hexazinone	86%	104%	5%	5%
Iodofenfos	90%	106%	1%	4%
Iprodione	91%	105%	5%	4%
Isazophos	93%	104%	4%	4%
Isodrin	96%	100%	8%	4%
Isopropalin	92%	102%	9%	5%
Lenacil	81%	106%	4%	6%
Leptophos	90%	108%	2%	6%
Linuron	83%	112%	8%	6%
Malathion	90%	107%	4%	4%
Metalaxyl	84%	105%	2%	2%
Metazachlor	89%	106%	5%	4%
Methacrifos	95%	105%	1%	4%
Methoxychlor	85%	104%	3%	5%
Metolachlor	89%	103%	4%	2%
Mevinphos	89%	106%	3%	4%
MGK-264 A	94%	101%	5%	6%
MGK-264 B	96%	109%	5%	4%
Mirex	92%	105%	2%	4%
Myclobutanil	88%	106%	3%	5%
N-(2,4-Dimethylphenyl)formamide	94%	102%	2%	4%
Nitrofen	94%	107%	3%	3%
Nonachlor-cis	86%	102%	5%	4%
Nonachlor-trans	97%	109%	9%	4%
Norflurazon	87%	104%	6%	4%
Ortho-phenylphenol	93%	106%	2%	4%
Oxadiazon	96%	104%	3%	4%
Oxyfluorfen	93%	103%	12%	6%
Paclobutrazol	87%	101%	10%	8%
Parathion (ethyl)	96%	117%	7%	5%
Parathion-methyl	95%	105%	5%	4%
Pebulate	97%	100%	4%	6%
Penconazole	91%	101%	4%	4%
Pendimethalin	94%	106%	4%	3%
Pentachloroaniline	93%	102%	5%	7%
Pentachloroanisole	91%	103%	3%	4%
Pentachlorobenzene	93%	102%	3%	6%
Pentachlorobenzonitrile	94%	102%	4%	6%
Pentachlorothioanisole	94%	104%	2%	5%
Permethrin peak 1	88%	107%	4%	4%
Perthane (Ethylan)	89%	106%	3%	3%
Phenothrin	91%	108%	6%	4%
Phorate	84%	102%	2%	4%
Phosalone	90%	105%	3%	5%

Onion	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Phosmet	92%	104%	2%	4%
Piperonyl butoxide	89%	106%	3%	5%
Pirimiphos-ethyl	91%	104%	3%	3%
Pirimiphos-methyl	91%	103%	2%	4%
Pretilachlor	89%	103%	3%	3%
Prochloraz	83%	115%	8%	6%
Procymidone	89%	110%	5%	3%
Prodiamine	100%	106%	6%	6%
Profenofos	82%	106%	4%	3%
Profluralin	89%	105%	9%	5%
Propachlor	94%	104%	2%	4%
Propanil	97%	106%	3%	4%
Propargite	89%	107%	7%	7%
Propisochlor	92%	103%	5%	3%
Propyzamide	93%	106%	1%	3%
Prothiofos	89%	104%	4%	4%
Pyraclufos	89%	104%	4%	7%
Pyrazophos	90%	108%	3%	5%
Pyridaben	90%	105%	4%	4%
Pyridaphenthion	88%	102%	3%	4%
Pyrimethanil	90%	105%	4%	5%
Pyriproxyfen	90%	104%	2%	3%
Quinalphos	91%	103%	7%	4%
Quintozene	96%	103%	5%	6%
Resmethrin	74%	91%	6%	4%
Sulfotep	90%	106%	5%	3%
Sulprofos	91%	110%	3%	4%
Tebuconazole	85%	108%	5%	6%
Tebufenpyrad	93%	105%	4%	4%
Tecnazene	94%	102%	3%	5%
Tefluthrin	91%	103%	4%	4%
Terbacil	90%	107%	6%	5%
Terbufos	92%	104%	1%	3%
Terbutylazine	94%	103%	5%	5%
Tetrachlorvinphos	88%	107%	9%	3%
Tetradifon	95%	105%	4%	4%
Tetrahydrophthalimide (THPI)	91%	107%	5%	5%
Tetramethrin peak 1	87%	108%	3%	4%
Tolclofos-methyl	90%	102%	2%	4%
Triadimefon	91%	106%	4%	4%
Triadimenol	89%	103%	5%	3%
Triallate	99%	103%	5%	5%
Triazophos	95%	106%	3%	5%
Tricyclazole	84%	105%	5%	4%
Triflumizole	87%	108%	8%	9%
Trifluralin	92%	105%	3%	4%
Vinclozolin	104%	108%	3%	4%

Appendix 2D, part 1. Recovery data for lemon

Lemon	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
2,3,5,6-Tetrachloroaniline	93%	100%	4%	2%
2,4'-Methoxychlor	102%	97%	5%	4%
3,4-Dichloroaniline	92%	110%	4%	4%
4,4'-Methoxychlor olefin	98%	102%	6%	3%
Acetochlor	102%	99%	6%	0%
Acrinathrin	108%	103%	7%	3%
Alachlor	91%	103%	6%	1%
Aldrin	99%	98%	10%	5%
Allethrin (Bioallethrin)	87%	103%	15%	3%
Allidochlor	99%	102%	4%	2%
Anthraquinone	106%	95%	10%	2%
Atrazine	95%	102%	5%	2%
Azinphos-ethyl	107%	102%	8%	3%
Azinphos-methyl	106%	105%	9%	7%
Benfluralin	92%	101%	5%	3%
BHC, Alpha	96%	106%	5%	2%
BHC, Beta	97%	103%	3%	1%
BHC, delta	93%	101%	5%	4%
BHC, gamma	89%	101%	6%	2%
Bifenthrin	103%	99%	6%	8%
Biphenyl	101%	101%	4%	3%
Bromfenvinphos	98%	99%	4%	3%
Bromfenvinphos-methyl	91%	101%	4%	2%
Bromophos-ethyl	98%	108%	6%	3%
Bromophos-methyl (Bromophos)	90%	102%	1%	2%
Bromopropylate	100%	99%	6%	6%
Bupirimate	93%	101%	4%	3%
Carbophenothion	105%	102%	8%	6%
Carfentrazon-ethyl	113%	102%	5%	8%
Chlorbenside	91%	100%	3%	2%
Chlordane alpha-cis	99%	96%	3%	4%
Chlordane gamma-trans	89%	99%	4%	3%
Chlorfenapyr	84%	120%	10%	4%
Chlorfenson	93%	100%	2%	1%
Chlorfenvinphos	97%	101%	3%	4%
Chlorobenzilate	99%	100%	2%	2%
Chloroneb	96%	102%	3%	2%
Chlorpropham	95%	100%	4%	2%
Chlorpyrifos-ethyl	97%	101%	2%	1%
Chlorpyrifos-methyl	92%	102%	8%	1%
Chlorthal-dimethyl (Dacthal)	89%	102%	4%	2%
Chlorthiophos	94%	101%	6%	2%
Chlozolinate	90%	104%	4%	3%
Clomazone	96%	101%	2%	2%
Coumaphos	95%	99%	6%	3%
Cycloate	91%	105%	6%	4%
Cyfluthrin	100%	101%	4%	3%
Cyhalothrin I (lambda)	99%	104%	3%	8%

Lemon	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Cypermethrin	97%	101%	3%	5%
Cyprodinil	94%	99%	1%	2%
DDD p,p	96%	101%	3%	2%
DDD, o, p	96%	101%	4%	1%
DDE o,p	95%	101%	4%	2%
DDE p, p	94%	98%	4%	2%
DDT o,p	94%	101%	2%	2%
DDT p,p	98%	98%	4%	3%
Deltamethrin	100%	99%	4%	3%
Diazinon	99%	104%	5%	3%
Dichlobenil	93%	100%	3%	3%
Dichlorobenzophenone, 4, 4	94%	100%	3%	2%
Dicloran (Bortran)	99%	100%	4%	2%
Dieldrin	87%	99%	5%	2%
Dimethachlor	99%	102%	3%	2%
Diphenamid	86%	100%	4%	2%
Diphenylamine	93%	99%	2%	2%
Disulfoton	92%	102%	3%	2%
Edifenphos	111%	101%	10%	9%
Endosulfan	93%	100%	5%	3%
Endosulfan ether	82%	103%	8%	3%
Endosulfan sulfate	100%	103%	5%	3%
Endrin	101%	103%	4%	2%
Endrin Aldehyde	86%	103%	11%	5%
Endrin-Ketone	96%	96%	6%	3%
EPN	105%	99%	10%	8%
Ethalfuralin	101%	102%	4%	2%
Ethion	97%	102%	4%	2%
Etofenprox	102%	99%	9%	1%
Etridiazole (Terrazole)	99%	103%	3%	3%
Fenamiphos	96%	97%	8%	4%
Fenarimol	101%	102%	3%	3%
Fenchlorfos	92%	101%	2%	1%
Fenitrothion	98%	100%	4%	4%
Fenpropathrin	102%	99%	2%	3%
Fenson	90%	99%	2%	1%
Fenthion	92%	103%	6%	1%
Fenvalerate	98%	101%	2%	2%
Fipronil	99%	103%	4%	3%
Fluazifop-P-butyl	97%	99%	3%	1%
Fluchloralin	94%	102%	5%	3%
Fludioxonil	97%	97%	4%	3%
Fluquinconazole	94%	99%	2%	2%
Fluridone	92%	100%	4%	4%
Flusilazole	98%	105%	5%	8%
Flutolanil	93%	99%	2%	3%
Flutriafol	99%	101%	4%	1%
Fluvalinate	100%	97%	4%	3%

Appendix 2D, part 2. Recovery data for lemon

Lemon	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Fonofos	87%	102%	4%	2%
Heptachlor	105%	98%	4%	2%
Hexachlorobenzene	98%	99%	3%	4%
Hexazinone	104%	96%	6%	5%
Iodofenfos	95%	99%	2%	2%
Iprodione	96%	100%	14%	9%
Isazophos	93%	105%	9%	3%
Isodrin	103%	98%	6%	4%
Isopropalin	93%	105%	10%	3%
Lenacil	109%	97%	13%	8%
Leptophos	102%	100%	3%	3%
Linuron	133%	95%	8%	4%
Malathion	93%	103%	5%	2%
Metalaxyl	98%	104%	6%	1%
Metazachlor	91%	103%	4%	2%
Methacrifos	94%	103%	1%	2%
Methoxychlor	100%	98%	3%	5%
Metolachlor	94%	101%	3%	1%
Mevinphos	101%	99%	5%	6%
MGK-264 A	84%	101%	3%	1%
MGK-264 B	90%	104%	5%	4%
Mirex	98%	100%	2%	2%
Myclobutanil	104%	100%	3%	3%
N-(2,4-Dimethylphenyl)formamide	95%	101%	4%	1%
Nitrofen	90%	99%	4%	3%
Nonachlor-cis	91%	100%	5%	4%
Nonachlor-trans	92%	98%	4%	3%
Norflurazon	109%	98%	8%	6%
Ortho-phenylphenol	90%	100%	3%	2%
Oxadiazon	94%	98%	3%	2%
Oxyfluorfen	99%	101%	6%	3%
Paclobutrazol	92%	106%	6%	4%
Parathion (ethyl)	91%	100%	8%	3%
Parathion-methyl	99%	100%	5%	2%
Pebulate	93%	102%	4%	3%
Penconazole	90%	104%	5%	2%
Pendimethalin	107%	101%	3%	3%
Pentachloroaniline	98%	100%	7%	3%
Pentachloroanisole	92%	98%	5%	3%
Pentachlorobenzene	102%	100%	6%	4%
Pentachlorobenzonitrile	102%	99%	2%	2%
Pentachlorothioanisole	95%	93%	5%	2%
Permethrin	95%	100%	3%	2%
Perthane (Ethylan)	96%	101%	4%	2%
Phenothrin	101%	101%	6%	7%
Phorate	92%	99%	8%	2%
Phosalone	104%	102%	8%	6%

Lemon	Recovery 0.01 mg/kg	Recovery 0.05 mg/kg	RSD 0.01 mg/kg	RSD 0.05 mg/kg
Phosmet	101%	101%	5%	8%
Piperonyl butoxide	109%	91%	11%	7%
Pirimiphos-ethyl	98%	96%	3%	2%
Pirimiphos-methyl	96%	96%	6%	3%
Pretilachlor	90%	104%	6%	2%
Prochloraz	97%	106%	3%	6%
Procymidone	87%	99%	4%	2%
Prodiamine	84%	102%	5%	3%
Profenofos	100%	104%	6%	3%
Profuralin	96%	110%	6%	2%
Propachlor	96%	101%	4%	2%
Propanil	91%	100%	2%	1%
Propargite	104%	95%	6%	3%
Propisochlor	96%	105%	9%	2%
Propyzamide	91%	101%	3%	2%
Prothiofos	97%	105%	4%	3%
Pyraclofos	107%	103%	2%	3%
Pyrazophos	99%	101%	4%	3%
Pyridaben	98%	102%	2%	2%
Pyridaphenthion	108%	103%	9%	10%
Pyrimethanil	89%	97%	4%	2%
Pyriproxyfen	102%	104%	5%	4%
Quinalphos	100%	103%	10%	4%
Quintozene	113%	104%	5%	2%
Resmethrin	113%	91%	15%	12%
Sulfotep	92%	101%	2%	2%
Sulprofos	101%	102%	7%	3%
Tebuconazole	109%	88%	7%	9%
Tebufenpyrad	96%	101%	2%	3%
Tecnazene	101%	104%	5%	2%
Tefluthrin	93%	103%	3%	2%
Terbacil	88%	104%	7%	1%
Terbufos	100%	102%	3%	2%
Terbuthylazine	91%	103%	3%	2%
Tetrachlorvinphos	95%	102%	5%	3%
Tetradifon	92%	99%	2%	2%
Tetrahydrophthalimide (THPI)	85%	104%	5%	1%
Tetramethrin	108%	99%	10%	8%
Tolclofos-methyl	101%	102%	2%	2%
Triadimefon	95%	101%	6%	3%
Triadimenol	115%	97%	6%	4%
Triallate	95%	101%	3%	4%
Triazophos	104%	102%	4%	4%
Tricyclazole	82%	95%	10%	5%
Triflumizole	111%	94%	11%	3%
Trifluralin	94%	102%	2%	1%
Vinclozolin	95%	102%	3%	3%

Appendix 3. Autosampler configuration and consumables

Part number	Description	Qty
TriPlus RSH SMART autosampler minimum configuration		
1R77010-2003	TriPlus RSH SMART Advanced base unit for liquid injection, regular rail including: - One tray holder - Three 54-vial trays for 2 mL vials - Standard wash station - 2x 10 µL syringes - Mounting brackets for TRACE 1300/1600 Series GC	1
1R77010-1019	Automatic Tool Change (ATC) station	1
1R77010-1160	µSPE GC QuEChERS clean-up kit TriPlus RSH autosampler including: - µSPE syringe tool (1 mL) for solvent dispensing - One liquid syringe tool D7/57 mm - 2x 1 mL syringe, 2x 100 µL syringe, 2x 10 µL syringe - Trayholder, vial, and cartridge trays with a 54-sample capacity - Waste tube - Solvent station (3x 100 mL) - Fast wash station - Standard wash station for IS/protectant + 2x 2 mL adapters - USB key driver with dedicated script, SOP and installation and user guide	1
Consumables		
Cartridges for pesticides QuEChERS clean-up		
60101-45GC	µSPE, QuEChERS blend for GC, 45 mg (MgSO ₄ , PSA, C18EC, Carbon), 108/PK	1
Required vials, snap caps, and seals for eluate collection		
6PRV11-1P	11 mm wide opening snap-it vials, 2 mL, clear, pack of 100	1
6PRC11STS1X	11 mm AS snap-it seal star pre-slit caps, pack of 100	1
Suggested vials, caps, and seals for raw sample and standards		
6PSV9-1PSS	Vial, 2 mL clear screw glass (9 mm short thread) with Thermo Scientific™ SureStop™ technology and write-on patch, Performance Level 3, 100/pack	1
6PSC9ST101	Cap, blue screw (9 mm) (PP: blue silicone/clear PTFE soft septa: 1.0 mm thickness) with AVCS technology, Performance Level 3, 100/pack	1
Optional upgrade to 108-vial (to increase the sample capacity)		
1R77010-1021	Trayholder able to house up to 3 different sample trays	1
1R77010-1162	µSPE elution tray cover to lock 2 mL vials in place, matching VT54 rack	1
1R77010-1163	Cartridge tray to hold 54 µSPE cartridges	1
1R77010-1178	µSPE waste receptacle to hold µSPE cartridge tray 54 or 96 positions	1
1R77010-1179	Waste tube, 2 m	1
1R77010-1023	Sample tray for 54 vials of 2 mL	2

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