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Pesticide GC/MSD/SIM Method

Scope and Application

This method will qualitate and quantify pesticide residues found in wines using Gas Chromatography coupled with a Mass Selective Detector in Selected Ion Monitoring mode.

Pesticides are regulated by the US Environmental Protection Agency (EPA). The EPA has the authority to regulate which pesticides and their residue tolerances are allowed for agricultural commodities. The Alcohol and Tobacco Tax and Trade Bureau (TTB) regulates the alcohol industry. Pesticides are monitored to protect consumers. Levels exceeding the EPA limits and unauthorized pesticides are reported to the US Food and Drug Administration (FDA). This method allows TTB to detect the trace residue levels of pesticides in wines and enforce EPA regulations.

Levels and Limitations

This method analyzes approximately 155 pesticides. The limit of detection, limit of quantitation, range and expected r^2 value for these compounds can be found in Table 1.

Equipment

GC:	Hewlett-Packard 6890-5973 Gas Chromatograph-Mass Selective Detector (GC-MSD)		
Column:	HP-5 capillary column, 30m X 0.25 mm x 0.25 μ m film thickness.		
Injector:	Pulsed splitless injection, temp = 250 °C		
Injection volume:	2 μ L		
Carrier Gas:	Ultrapure Helium (99.999% pure) (constant pressure) as set by Retention Time Locking Program on HP6890 GC using methyl-chlorpyrifos as the RTL standard		
Temperature Program:	For RTL, GC should be operating under constant pressure		
	Initial Temperature: 70°C		
	Initial Time: 2.00 min		
	<u>Rate</u>	<u>Final Temp</u>	<u>Final Time</u>
	25 °C/min	150 °C	0 min
	3	200	0
	8	280	10

Total Run Time: 41.87 min

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Mass Spec Ion Source: 230 °C; transfer line 280 °C; quad temp 150 °C
Selected Masses: See Table 2
Ion Groups: See Tables 3, 4, 5
Quantitation: Use Chemstation macro to extract quantitation ions, scale, and integrate peak areas.

Glassware and Supplies:

15 mL graduated conical test tubes, nearest 0.1 mL

50 mL volumetric flasks (class A)

10 mL glass pipets (class A)

Reagent and Sample Preparation and Handling

Reagents:

Pesticide Standards—available from United States Environmental Protection Agency (USEPA) Technical Standards Laboratory, **Ft. Meade**, MD, Crescent Chemicals, Hauppauge, NY; and Chem Service, West Chester, PA, Fluka Chemical Corp. (Milwaukee, WI)

Internal standards--acenaphthene-d10, chrysene-d12 and phenanthrene-d10
Aldrich Chemical Co. (Milwaukee, WI)

Solvents—Methanol, DI water, Hexane, and Ethyl Acetate (analytical, pesticide and chromatography grade solvents)

Solid Phase Extraction Cartridges- Waters Oasis HLB (0.2g/6mL)

Aminopropyl Cartridges – Supelco LC-NH2 (0.5 g/3 mL)

Magnesium Sulfate

Preparation of Individual Stock Standards

1. Measure between 0.100 to 0.500 g of standard.
2. Place the weighed out standard into a 100 mL volumetric flask.
3. Fill to the mark with ethyl acetate.

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Preparation of Working (Mix) Standards:

1. Prepare a twelve level calibration curve for each class of pesticides (organophosphorus, organohalogen, and organonitrogen) as follows:

Standard 1 (~5 ppm): Take 2-10 mL of ~50 individual stocks and place in a 200 mL volumetric. Q.S. with ethyl acetate.

Standard 2 (~2.5 ppm): Take 50 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 3 (~1 ppm): Take 20 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 4 (~0.5 ppm): Take 10 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 5 (~0.250 ppm): Take 5 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 6 (~0.100 ppm): Take 2 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 7 (~0.050 ppm): Take 1 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 8 (~0.025 ppm): Take 0.5 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 9 (~0.010 ppm): Take 0.25 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 10 (~0.005 ppm): Take 0.10 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 11 (~0.0025 ppm): Take 0.05 mL of Standard 10 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

Standard 12 (~0.001 ppm): Take 0.02 mL of Standard 1 and place in a 100 mL volumetric. Q.S. with ethyl acetate.

2. Prepare standards for GC-MS by placing 1 mL of Standards 1-12 in separate GC vials. Add 50 μ L internal standard containing acenaphthene-d10 (21.33 μ g /mL), chrysene-d12 (23.40 μ g /mL) and phenanthrene-d10 (23.46 μ g/mL).

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Sample Preparation (see Figure 1.)

1. Transfer 20 mL wine to a 50 mL volumetric flask. (Fortify the transferred wine at this point for spike/recovery experiment, if necessary.) Quality Control samples (water and laboratory control standard wine) are spiked with 1 mL of 5 ppm pesticide standards.
2. Add 20 mL DI water and mix thoroughly.
3. Condition Waters Oasis HLB cartridge (0.2 g/6 mL) with 1-2 column volumes of 50:50 EtOAc:Hexane, 1-2 column volumes of MeOH, and 1-2 column volumes of HPLC-grade water. Conditioning is performed under gravity and may require a mild vacuum for priming.
4. Load the sample (40 mL diluted wine **or 41 mL for QC samples**) onto the cartridge. Extraction requires little or no vacuum applied. Rinse the volumetric flask with approximately 2 column volumes of DI grade water and load onto the cartridge.
5. Allow the cartridge to dry for at least 15 minutes under vacuum. Prepare second manifold for aminopropyl-magnesium sulfate cartridges.
6. Prepare a Supelco LC-NH2 aminopropyl cartridge (0.5 g/3 mL) and fill about 1/3 full with magnesium sulfate (MgSO₄).
7. Place the MgSO₄-aminopropyl cartridge on the second manifold. Condition the cartridge with 5 mL of 50:50 EtOAc:Hexane. Close the valve when all but ~0.5 mL of EtOAc:Hexane is left in the column volume. Attach an adaptor to this cartridge.
8. Disconnect the Oasis HLB cartridge from the first manifold and attach it to the MgSO₄-aminopropyl cartridge with the adaptor on the second manifold. Insert the tube rack consisting of 15 mL conical tubes into the second manifold.
9. Elute the Oasis HLB/MgSO₄-aminopropyl tandem cartridge with the following under gravity (may require an initial priming with a vacuum):
 - 5 mL 80:20 EtOAc:Hexane
 - 5 mL 50:50 EtOAc:Hexane
 - 5 mL 20:80 EtOAc:Hexane
10. Remove the conical tubes from the manifold and place the tubes in a nitrogen evaporation system at ambient temperature. Evaporate the solvent under a gentle stream of nitrogen to ca. 0.1 mL.

GCMS Procedures

1. **Add** 1 mL of 0.1% corn oil in ethyl acetate, **vortex sample, then** transfer into autosampler vials and add 50 µL internal standard containing acenaphthene-d10 (21.33 µg /mL), chrysene-d12 (23.40 µg /mL) and phenanthrene-d10 (23.46 µg/mL).
2. The sequence generated on the GC-MSD should be as follows:
 - Ethyl Acetate Wash
 - Ethyl Acetate Blank Spiked with Internal Standard solution

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- Standards 1- 12
- Ethyl Acetate Wash
- Water Blanks (QC)
- Water Spike (QC)
- Ethyl Acetate Wash
- Wine Blank (QC)
- Wine Spike (QC)
- Ethyl Acetate Wash
- Samples with Wash injections between each one.
- **250 ppb standard (instrument check)**
- **Ethyl Acetate Wash**
- **10 ppb standard**
- **Ethyl Acetate Wash**

Note: The final ethyl acetate wash should be run on the standby.m method to conserve helium and to decrease the total flow while the instrument is idle.

Quality Control

Quality control blanks and spikes will go through the same sample preparation as the samples. Each run will include the following:

- Three water blanks
- One water spiked with 5.0 ppm standard mix solution (organohalogens, organonitrogens, and organophosphorous compounds.)
- One laboratory control sample wine blank
- One laboratory control standard wine spiked with 5.0 ppm standard mix solution (organohalogens, organonitrogens, and organophosphorous compounds.)

The spiked wine and water quality control samples will actually contain **50 ppb** of pesticides. Eight pesticides in those samples will be control charted. The eight pesticides that will be monitored are:

- Chlorothalonil (organohalogen)
- Iprodione (organohalogen)
- Procymidone (organohalogen)
- Metalaxyl (organonitrogen)
- Oxadixyl (organonitrogen)
- Pyrimethanil (organonitrogen)
- Chlorpyrifos- methyl (organophosphorous)

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- Penconazole (organophosphorous)

Recovery values should be within $\pm 20\%$ of the expected value (**see Table 6**). The control charts and record should be kept with the instrument in the log book.

In the event a control should fail:

- The run must be stopped.
- No results for that run can be reported.
- The standards should be remade, analyzed, and compared to the old standard.
- Once the standards are suitable for use the samples can be re-ran.

Sources of Uncertainty

1. Creation of standards and solutions
2. Spiking of internal standards
3. Splatter of recovered solvent during the clean-up extraction step
4. Splatter of solvent in the concentration of sample via nitrogen evaporation
5. Reconstitution of sample to 1 mL
6. Syringe injection volume during GCMS injection
7. Dirty **inlet (includes inlet liner, gold seal, and/or head of column)**

Calculations

Calculate pesticide levels according to the calibration curve. Use area ratios of (sample area count)/(I.S. area count) using the GC/MS data processing software. The resulting value is divided by 20 because the initial sample was concentrated from 20 mL to 1 mL.

Reporting Results

During the initial screening, pesticide area counts should be compared to the 10 ppb standard area counts. If the analyte's area counts are below the 10 ppb standard, it can be considered "not detected" and a curve does not have to be generated. Otherwise the following rules apply.

Pesticides with a r^2 value of less than 0.99 should be reported qualitatively. The following pesticides are known to produce r^2 values less than 0.99:

Bromoxynil
Dinoseb
Oryzalin

Captafol
Folpet

Carbaryl
Imazalil

Qualitative results are to be reported in the following format:

Pesticide is detected:

D (Detected)

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Pesticide is not detected: ND (Not Detected)

Quantitative results, r^2 greater than 0.99, are to be reported in the following format:

Values below the LOD:	ND (Not Detected)
Values below the LOQ but above LOD:	BQL (Below Quantifiable limit)
Values above the LOQ:	XXX ppb

Recovered pesticide values should be cross referenced with the EPA 40 CFR Section 180. The following actions should be taken:

Pesticides within EPA tolerances:	No Action
Pesticides not within EPA tolerances:	Violation
Pesticides not listed:	Violation

Wines containing pesticides in violation of the EPA tolerances shall be re-extracted and rerun to confirm the violation. If the result confirms the initially obtained value then a report is issued to ALFD.

Safety Notes

Fume hoods should be used throughout the experiment due to the use of solvents.

Consult the MSDS for any chemicals used that are unfamiliar. All chemicals should be considered hazardous—avoid direct physical contact.

Personnel should follow good general laboratory practices such as wearing protective eye wear, gloves and a lab coat.

References

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Location of Validation Package

Quality Management Files

Required Training, Certification and Re-certification.

1. **In-house training by a certified chemist in GC and Chemstation operation. Training on GC (in-house or vendor provided).**
2. Initial certification is achieved by running one blind sample at least in triplicate with results of precision and reproducibility in agreement with the laboratory performance criteria.
3. Proficiency testing.

Revision History

Rev. 1 – general editorial changes and clarification of method, addition of “Sources of Uncertainty”-- 10/31/2008

Rev. 2 -- General editorial changes, addition of 10 ppb calibration check, add table of recoveries for 8 control pesticides

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Table 1.

Pesticide	LOD (ppb)	LOQ (ppb)	Range (ppb)	r ²
Acephate	25	83.3	50 – 6160	0.9957
<i>Acenaphthene-d10 (I.S.)</i>				
Alachlor	1	3.3	2.0 - 3450	0.9999
Aldrin	1.5	5.0	3.0 - 5760	1
Allethrin	3	10.0	6.0 - 6100	0.9998
Atrazine	1	3.3	3.0 - 5120	0.9992
Azinphos-ethyl	1	3.3	3.0 - 5205	0.9992
Azinphos-methyl	3	10	5.5 - 5100	0.9985
Benalaxyl	< 1.0	10	1.0 - 5450	0.9998
Benfluralin	< 1.0	10	1.0 - 4950	0.9996
BHC-a	1	10	2.0 - 3280	0.9992
BHC-d	2	6.7	4.0 - 3820	0.9999
BHC-g (Lindane)	1.5	5	3.0 - 3060	1
Bitertanol I	0.5	1.7	1.5 - 2500	0.9986
Bitertanol II	0.5	1.7	1.5 - 2500	0.9997
Bromophos-ethyl	< 1.0	10	1.0 - 3650	0.9997
Bromophos-methyl	< 1.0	10	1.0 - 4760	0.9999
Bromopropylate	0.5	1.7	1.5 - 2890	0.9995
Bromoxynil	10	33.3	25 - 5070	0.9425
Captafol	25	83.3	50 - 4630	0.9743
Captan	10	33.3	25 - 5140	0.9943
Carbaryl	10	33.3	30 - 5190	0.9911
Carbofuran	2	6.7	2.5 - 5100	0.9901
Carbophenothion	< 1.5	5	1.5 - 5630	0.9997
Chlorbenside	1	3.3	2.0 - 3140	0.9991
cis-Chlordane	< 1.0	3.3	1.0 - 4460	1
trans-Chlordane	< 1.0	3.3	1.0 - 3650	0.9996
Chlorfenvinphos	1	3.3	2.5 - 4425	0.9996
Chlorothalonil	1	3.3	3.0 - 5450	0.9982
Chlorpyrifos	1	3.3	2.0 - 4065	0.9997

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Pesticide	LOD (ppb)	LOQ (ppb)	Range (ppb)	r ²
Chlorpyrifos-methyl	< 1.0	3.3	1.0 - 3900	0.9999
Chlozolinate	1.5	5	3.0 - 5750	0.9999
<i>Chrysene-d12 (I.S.)</i>				
Coumaphos	1	3.3	2.5 - 4500	0.9986
Cyanazine	3	10	6.5 - 6160	0.9998
Cyfluthrin I	1.5	5	3.0 - 2540	0.9982
Cyfluthrin II	1.5	5	3.0 - 2540	0.9983
Cyfluthrin III	2.5	8.3	5.0 - 2540	0.9963
Cyfluthrin IV	2.5	8.3	5.0 - 2540	0.9993
Cyhalothrin	< 1.5	5	1.5 - 7210	0.9999
Cypermethrin I	1	3.3	2.0 - 1625	0.9987
Cypermethrin II	1	3.3	2.0 - 1625	0.9979
Cypermethrin III	1	3.3	2.0 - 1625	0.9981
Cypermethrin IV	1	3.3	2.0 - 1625	0.9977
Cyprodinil	< 1.5	5	1.5 - 7485	0.9996
o,p'-DDT	< 0.5	1.7	1.0 - 850	0.9932
p,p'-DDT	< 1.0	3.3	1.0 - 2770	0.9988
Deltamethrin	10	33.3	20 - 4180	0.9946
Demeton-O	< 1.0	3.3	1.0 - 4900	0.9999
Demeton-S	< 1.0	3.3	1.0 - 4900	0.9997
Desmetryn	< 1.5	5	1.5 - 6040	0.9992
Dialifos	1	3.3	3.0 - 5200	0.9995
Diallate I	< 0.5	1.7	0.5 - 2500	0.9999
Diallate II	< 0.5	1.7	0.5 - 2500	1
Diazinon	< 1.0	3.3	1.0 - 4400	0.9998
Dichlobenil	< 1.5	5	1.5 - 6700	1
Dichlofluanid	< 1.5	5	1.5 - 5425	0.9993
4,4'-Dichlorobenzophenone	0.5	1.7	1.5 - 2590	0.9998
Dichlorvos	< 1.0	3.3	1.0 - 4440	0.9998
Dicloran	4	13.3	8.0 - 3990	0.9985
Dicrotophos	3	10	6.0 - 6210	0.9998

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Pesticide	LOD (ppb)	LOQ (ppb)	Range (ppb)	r ²
Dieldrin	2	6.7	3.5 - 3260	0.9998
Dimethoate	2.5	8.3	5.0 - 5070	0.9998
Dinoseb	150	500	350 - 6620	0.905
Dioxathion	5	16.7	10- 5000	0.9987
Disulfoton	1	3.3	2.5 - 4380	0.9995
Endosulfan-a	1.5	5	3.0 - 2700	0.9999
Endosulfan-b	3	10	5.0 -2700	0.9999
Endrin	3.5	11.7	6.0 - 3220	0.9977
Endrin aldehyde	2	6.7	4.0 - 3630	0.9999
Endrin ketone	< 1.0	3.3	1.0 - 4580	0.9999
EPN	< 1.0	3.3	1.0 - 3350	0.9949
Eptam	1	3.3	2.5 - 4795	1
Ethalfuralin	1	3.3	3.0 - 5300	0.9983
Ethion	1	3.3	2.5 - 4380	0.9993
Fenamiphos	< 1.0	3.3	1.0 - 4110	0.9991
Fenarimol	0.6	2	1.5 - 5560	0.9999
Fenitrothion	1	3.3	2.0 - 3450	0.9993
Fenpropathrin	0.6	2	1.5 - 5780	1
Fenpropimorph	< 0.5	1.7	1.5 - 5580	0.9996
Fenson	10	33.3	30- 3750	0.9998
Fenvalerate I	3	10	6.0 - 5550	0.9992
Fenvalerate II	3	10	6.0 - 5550	0.9995
Fenthion	< 1.5	5	1.5 - 5300	0.9998
Flucythrinate I	1.5	5	2.5 - 2430	0.9985
Flucythrinate II	1.5	5	2.5 - 2430	0.998
Fludioxinil	1	3.3	3.0 - 5335	0.9993
Fluvalinate tau-I	0.5	1.7	1.5 - 2550	0.9968
Fluvalinate tau-II	0.5	1.7	1.5 - 2550	0.9974
Folpet	15	50	30 - 5460	0.9339
Fonofos	< 1.0	3.3	1.0 - 3660	0.9996
Furalaxyl	1	3.3	2.5 - 5050	0.9992

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Pesticide	LOD (ppb)	LOQ (ppb)	Range (ppb)	r ²
Heptachlor	0.5	1.7	1.5 - 2990	0.9998
Heptachlor epoxide	0.5	1.7	1.5 - 2880	0.9999
Hexachlorobenzene	< 0.5	1.7	0.5 - 2780	0.9999
Hexaconazole	1	3.3	3.0 - 5480	0.9997
Hexazinone	0.5	1.7	1.0 - 5050	0.9997
Imazalil	6	20	30- 5550	0.9996
Iprodione	5	16.7	10- 4980	0.9894
Isofenphos	1	3.3	2.5 - 4950	0.9997
Malaoxon	1	3.3	3.0 - 5180	0.9996
Malathion	< 1.5	5	1.5 - 5500	0.9997
Metalaxyl	1	3.3	2.5 - 4880	0.9993
Methidathion	1	3.3	2.0 - 3720	0.9995
Methoxychlor	< 1.0	3.3	1.0 - 5500	0.9998
Metolachlor	< 1.0	3.3	1.0 - 4350	0.9977
Mevinphos	< 1.5	5	1.5 - 6780	0.9998
Mirex	< 1.0	3.3	1.0 - 5500	0.9998
Monocrotophos	3	10	7.0 - 6555	0.9996
Myclobutanil	1	3.3	2.5 - 4455	0.9998
Naled	7	23.3	13 - 6480	0.9984
Napropamide	< 1.0	3.3	1.5 - 5525	0.9999
Nitralin	0.5	1.7	1.0 - 5110	0.9918
Nitrofen	3	10	5.0 - 5120	0.9946
Nitrothal-isopropyl	1	3.3	3.0 - 5520	0.9956
Norflurazon	1	3.3	3.0 - 5120	0.999
Omethoate	6	20	6.0 - 5840	0.9985
Oryzalin	75	250	100 - 5030	0.8945
Oxadiazon	0.6	2	1.5 - 5375	1
Oxadixyl	1.5	5	3.0 - 5310	0.9999
Oxyfluorfen	1	3.3	3.0 - 5790	0.9955
Paraoxon	3	10	6.0 - 5620	0.9996
Parathion	1	3.3	2.5 - 4660	0.9989

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Pesticide	LOD (ppb)	LOQ (ppb)	Range (ppb)	r ²
Parathion-methyl	1	3.3	2.5 - 4500	0.9972
Penconazole	< 1.0	3.3	1.0 - 5220	0.9998
cis-Permethrin	< 0.5	1.7	0.5 - 2530	0.9999
trans-Permethrin	< 0.5	1.7	0.5 - 2900	0.9999
<i>Pheneanthrene-d10 (I.S.)</i>				
Phorate	< 1.0	3.3	1.0 - 4320	0.9996
Phosalone	< 1.0	3.3	1.0 - 3720	0.9996
Phosmet	< 1.0	3.3	1.0 - 4830	0.9994
Prochloraz	6	20	12- 5940	0.997
Procymidone	1	3.3	3.0 - 5920	0.9999
Profenofos	3	10	5.0 - 5100	0.9997
Prometryn	< 1.5	5	1.5 - 5800	0.9992
Propargite	0.5	1.7	1.0 - 4780	0.997
Propazine	< 1.0	3.3	1.0 - 5070	0.9993
Propetamphos	< 1.0	3.3	1.0 - 3720	0.9997
Propyzamide	1.5	5	3.0 - 5750	0.9998
Pyrimethanil	< 1.0	3.3	1.0 - 4970	0.9994
Quinalphos	50	166.7	100 - 9125	0.9997
Quintozene	< 2.0	6.7	2.0 - 8800	0.9997
Simazine	5	16.7	5.0 - 5190	0.999
Tebuconazole	1.5	5	3.0 - 6050	0.9994
Tecnazene	1	3.3	2.0 - 4240	0.9967
Terbufos	< 1.0	3.3	1.0 - 4620	0.9999
Terbutylazine	< 1.5	5	1.5 - 5860	0.9994
Terbutryn	< 1.0	3.3	1.0 - 5000	0.9992
Tetrachlorovinphos	< 1.0	3.3	1.0 - 4020	0.9993
Tetradifon	1	3.3	3.0 - 5180	0.9998
Thiometon	1.5	5	3.5 - 6360	0.9998
Tolyfluanid	2.6	8.7	12- 5220	0.9999
Triadimefon	1	3.3	2.5 - 5060	0.9996
Triadimenol	4	13.3	8.0 - 4050	0.9995

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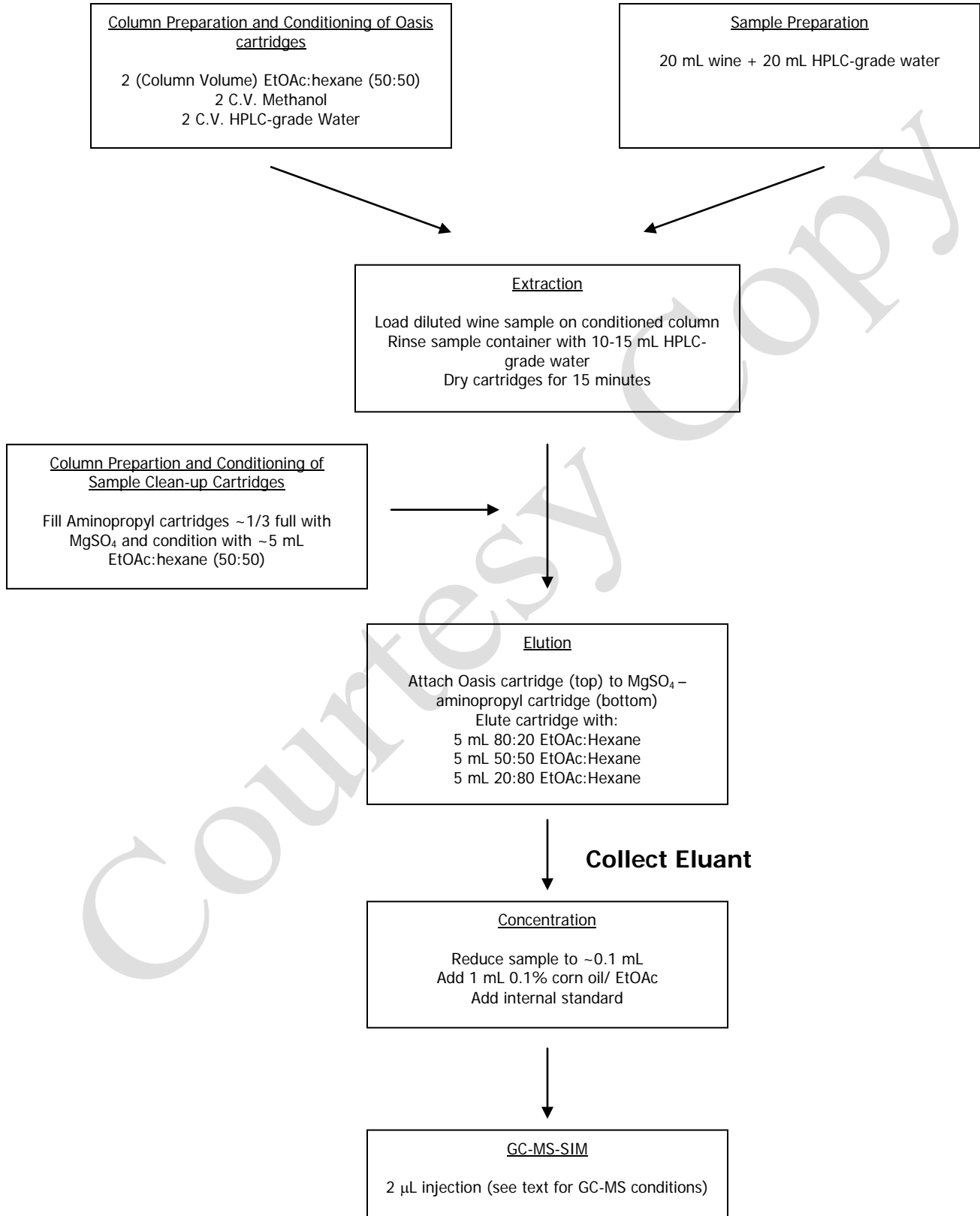
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Pesticide	LOD (ppb)	LOQ (ppb)	Range (ppb)	r²
Tri-allate	< 1.0	3.3	1.0 - 5200	0.9997
Trifluralin	< 1.0	3.3	1.0 - 5580	0.9992
Vinclozolin	1	3.3	3.0 - 5130	0.9992

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Figure 1. Sample Extraction Procedure.



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Table 2. Target to Qualifier Ion Masses and Ratios

Pesticide	MW	RT (min)	Target (T)	Qualifier Ion 1 (Q1)	Q1/T (%)	Qualifier Ion 2 (Q2)	Q2/T (%)	Qualifier Ion 3 (Q3)	Q3/T (%)
Acephate	183.2	7.69	136	94	43.5	95	22.8	125	13.1
Acenaphthene-d10 (I.S.)	164.3	8.39	164	162	92.3	160	42.2	80	15.3
Alachlor	269.8	17.04	160	188	91.1	146	48.2	237	29.9
Aldrin	364.9	18.5	263	265	66.6	261	64.1	66	61
Allethrin	302.4	21.81, 21.86	123	79	27	136	23.4	107	23
Atrazine	215.7	13.2	200	215	53.9	202	35.2	58	25.9
Azinphos-ethyl	345.4	30.65	132	160	87.4	77	51.4	105	26.1
Azinphos-methyl	317.3	29.65	160	132	73.9	77	57	105	22
Benalaxyl	325.4	26.75	148	91	40.1	206	28.6	204	20.8
Benfluralin	335.3	11.71	292	264	17.3	276	12.1	293	11.9
BHC-alpha	290.8	12.1	181	183	94	219	89.5	217	72.7
BHC-delta	290.8	14.68	181	219	103	183	99.2	217	80.8
BHC-gamma (Lindane)	290.8	13.47	181	183	97.4	219	89.6	111	70.6
Bitertanol I	337.4	31.23	170	168	16.3	171	13.3	57	8.5
Bitertanol II	337.4	31.37	170	168	20.5	171	14	57	9
Bromophos-ethyl	394.1	22.53	359	303	81	357	75	301	59.8
Bromophos-methyl	366	20.08	331	329	75.5	333	29.4	125	27.3
Bromopropylate	428.1	28.67	341	183	52.1	339	52	343	49.7
Bromoxynil	276.9	11.63	277	275	54	279	49.3	88	28.5
Captafol	349.1	27.66	79	80	33.4	77	22.2	151	18.2
Captan	300.6	21.3	79	80	23.1	151	18.9	77	16.4
Carbaryl	201.2	16.87	144	115	63.1	116	30.3	145	9.6
Carbofuran	221.3	13.06	164	149	72.9	131	26	123	22
Carbophenothion	342.9	26.09	157	342	56.6	121	44.4	199	27
Chlorbenseide	269.2	21.87	125	127	32.6	268	13.1	270	9.2
cis-Chlordane	409.8	22.85	373	375	94.5	377	49.4	371	43.6
trans-Chlordane	409.8	22.07	373	375	93.7	377	51.9	371	42.6

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Pesticide	MW	RT (min)	Target (T)	Qualifier Ion 1 (Q1)	Q1/T (%)	Qualifier Ion 2 (Q2)	Q2/T (%)	Qualifier Ion 3 (Q3)	Q3/T (%)
Chlorfenvinphos	359.6	21.29	267	323	67.4	269	65.9	325	42.9
Chlorothalonil	265.9	14.92	266	264	79.4	268	46.6	270	10
Chlorpyrifos	350.6	19.03	197	199	94.6	314	88	97	66.8
Chlorpyrifos-methyl	322.5	16.59	286	288	68.9	125	40.4	290	15.2
Chlozolinate	332.1	21.38	188	259	87.8	186	84.4	187	76.6
Chrysene-d12 (I.S.)	240.4	28.42	240	236	24.2	241	19.9	238	5.1
Coumaphos	362.8	31.67	362	226	46.3	109	34.1	210	32.1
Cyanazine	240.7	19.47	212	213	37.7	214	37	68	16.7
Cyfluthrin I	434.3	32.3	163	206	78.7	165	69.4	227	46.6
Cyfluthrin II	434.3	32.45	163	206	63.8	165	66.1	227	46.1
Cyfluthrin III	434.3	32.56	163	206	73.6	165	67.2	227	51.8
Cyfluthrin IV	434.3	32.63	163	206	65.1	199	47.6	227	47.2
Cyhalothrin	449.9	30.37	181	197	79.6	208	50.4	209	25.1
Cypermethrin I	416.3	32.69	181	163	108.5	165	71	209	51.5
Cypermethrin II	416.3	32.85	181	163	122.3	165	79.9	209	57.5
Cypermethrin III	416.3	32.97	163	181	89.5	165	67.1	209	44
Cypermethrin IV	416.3	33.02	163	181	81.5	165	68.2	209	40.9
Cyprodinil	225.3	20.57	224	225	65.3	210	10.2	77	8.7
o,p'-DDT	354.5	25.79	235	237	63.2	165	35.8	236	14
p,p'-DDT	354.5	27.02	235	237	66.2	165	36.6	236	15.1
Deltamethrin	505.2	36	181	253	82.7	251	43.9	255	41.5
Demeton-O	230.3	10.4	88	60	28.7	89	28.5	171	12.7
Demeton-S	230.3	12.63	88	60	28.6	170	17.6	89	13.6
Desmetryn	213.3	16.02	213	198	57.8	171	28	58	17.9
Dialifos	393.9	30.83	208	173	71.6	210	34	76	30.1
Diallate I	270.2	11.94	86	234	78.3	236	30.9	128	30.1
Diallate II	270.2	12.28	86	234	85.7	236	35.5	128	25.9
Diazinon	304.3	14.47	179	137	86	199	61.6	152	60.6
Dichlobenil	172	6.77	171	173	65.4	136	17	100	16.7

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Pesticide	MW	RT (min)	Target (T)	Qualifier Ion 1 (Q1)	Q1/T (%)	Qualifier Ion 2 (Q2)	Q2/T (%)	Qualifier Ion 3 (Q3)	Q3/T (%)
Dichlofluanid	333.2	18.41	123	224	54.4	167	51.3	226	37.8
4,4'-Dichlorobenzophenone	251.1	19.24	139	111	35.4	141	32.9	250	30.7
Dichlorvos	221	5.83	109	185	43.2	79	17.4	187	14.9
Dicloran	207	12.59	206	176	127.6	178	81	208	63.8
Dicrotophos	237.2	11.49	127	67	19.7	193	11.9	72	8.8
Dieldrin	380.9	23.88	79	263	47.8	277	37.2	279	33.4
Dimethoate	229.3	12.68	87	93	61.3	125	58.6	143	12
Dinoseb	240.2	14.57	211	163	33.9	147	20.9	240	18.2
Dioxathion	456	32.23	97	125	80.2	271	66.3	153	29.1
Disulfoton	274.4	14.55	88	89	39.1	97	30	142	18.4
Endosulfan-a	406.9	22.65	241	195	90.1	239	90	237	89.9
Endosulfan-b	406.9	25.2	195	237	91.5	241	88.4	207	82
Endrin	380.9	24.77	317	263	78.9	315	68	319	62.2
Endrin aldehyde	380.9	25.94	67	345	71.8	250	62.2	347	47.5
Endrin ketone	380.9	28.27	317	67	85	315	65	319	62.8
EPN	323.3	28.66	157	169	53.1	141	44.9	185	29.7
Eptam	189.3	6.8	128	43	101.2	86	61.6	132	28.8
Ethalfuralin	333.3	11.27	276	316	92.4	292	47	333	24.8
Ethion	384.5	26	231	153	49.4	97	42.6	125	32.6
Fenamiphos	303.4	23.32	303	154	46	288	28	217	25.2
Fenarimol	331.2	30.42	139	219	75.4	251	69.2	107	66.7
Fenitrothion	277.2	18.07	277	125	97.3	109	72.3	260	55.5
Fenpropathrin	349.4	28.98	97	181	70	125	40.3	265	28
Fenpropimorph	305.5	19.2	128	129	9.2	303	4.4	117	2.9
Fenson	268.7	19.68	77	141	92.8	268	48	51	13.8
Fenvalerate I	419.9	34.4	167	125	98.3	181	74.1	152	55.9
Fenvalerate II	419.9	34.83	167	125	96.8	181	66	169	62.3
Fenthion	278.3	19.12	278	125	33.3	109	26.4	169	24.7
Flucythrinate I	451.4	33.05	199	157	63.1	181	37.6	107	16.6

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Pesticide	MW	RT (min)	Target (T)	Qualifier Ion 1 (Q1)	Q1/T (%)	Qualifier Ion 2 (Q2)	Q2/T (%)	Qualifier Ion 3 (Q3)	Q3/T (%)
Flucythrinate II	451.4	33.38	199	157	61.7	181	38.3	107	14.7
Fludioxinil	248.2	24.23	248	127	30	154	24.2	182	15.7
Fluvalinate tau-I	502.9	34.83	250	252	33.8	181	19.4	208	9.1
Fluvalinate tau-II	502.9	34.97	250	252	33.1	181	20.1	208	9.3
Folpet	296.6	21.66	147	104	95.7	76	80.4	260	69.2
Fonofos	246.3	13.89	109	246	58.5	137	52.1	110	26.5
Furalaxyl	301.3	21.92	95	242	48.6	152	17.9	146	12.4
Heptachlor	373.3	16.77	272	274	82.1	100	72.7	270	54.5
Heptachlor epoxide	389.3	20.78	353	355	81.1	351	51.6	357	35.9
Hexachlorobenzene	284.8	12.37	284	286	82.2	282	51.8	288	36.1
Hexaconazole	352.9	23.57	83	214	60.6	216	38.7	82	34.2
Hexazinone	252.3	27.41	171	83	14.5	128	11.9	71	10.1
Imazalil	297.2	23.78	41	215	67.2	173	50.2	217	42.8
Iprodione	330.2	28.42	314	187	59.5	189	41.4	244	24.9
Isofenphos	345.4	21.62	213	58	81.1	121	54.5	255	44
Malaaxon	314.3	16.78	127	99	39.7	109	22.4	125	19.1
Malathion	330.4	18.8	173	127	84.6	125	77.3	93	56.8
Metalaxyl	279.3	17.35	206	45	62.3	160	52	249	49.5
Methidathion	302.3	22.3	145	85	61.2	93	16.8	125	16.3
Methoxychlor	345.7	28.91	227	228	17.3	152	9	113	3.7
Metolachlor	283.8	18.9	162	238	62	240	21.2	146	13
Mevinphos	224.2	7.59	127	192	59	109	41	67	18.4
Mirex	545.6	29.85	272	274	80	270	52.1	237	44.7
Monocrotophos	223.2	11.74	127	67	17.9	192	16.3	97	14.9
Myclobutanil	280.8	24.49	179	150	49.5	82	35.4	181	31.8
Naled	380.8	11.22	109	185	31.2	79	16.8	145	16.5
Napropamide	271.4	23.46	72	128	51.9	100	35.1	271	30.2
Nitralin	345.4	28.19	316	274	70.6	300	15.1	317	14.7
Nitrofen	284.1	24.9	283	253	85.5	283	66.1	202	46

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Pesticide	MW	RT (min)	Target (T)	Qualifier Ion 1 (Q1)	Q1/T (%)	Qualifier Ion 2 (Q2)	Q2/T (%)	Qualifier Ion 3 (Q3)	Q3/T (%)
Nitrothal-isopropyl	295.3	19.86	236	194	73.4	212	63.6	254	52.2
Norflurazon	303.7	27.01	303	145	90.7	102	41.3	305	34.1
Omethoate	213.2	10.01	156	110	85.7	79	25	109	20.4
Oryzalin	346.4	31.32	317	275	47.2	258	12.5	58	8.8
Oxadiazon	345.2	24.41	175	177	65.8	258	58.9	260	38
Oxadixyl	278.3	25.92	105	163	113.1	45	80.5	132	76.5
Oxyfluorfen	361.7	24.72	252	361	38.3	302	23.3	331	15.6
Paraoxon	275.2	17.34	109	149	39.7	275	33	139	30.8
Parathion	291.3	19.27	291	109	83.4	97	80.9	139	45.9
Parathion-methyl	263.2	16.59	263	109	91.9	125	78.6	79	23.5
Penconazole	284.2	21.08	248	159	93.7	161	60	250	33.5
cis-Permethrin	391.3	31.42	183	163	18.8	165	16.4	184	16.3
trans-Permethrin	281.3	31.61	183	163	28	165	23.1	184	18.1
Pheneanthrene-d10 (I.S.)	188.3	13.7	188	189	16	184	14.5	187	10
Phorate	260.4	11.8	75	121	44	260	24.7	97	22.4
Phosalone	367.8	29.68	182	367	44.4	121	35.6	184	34.2
Phosmet	317.3	28.5	160	161	11.2	77	5.6	93	5.4
Prochloraz	376.7	31.81	180	70	69.9	308	45.2	310	45.1
Procymidone	284.1	21.96	96	283	64.1	285	41.5	67	41.4
Profenofos	373.6	23.9	208	339	91.7	139	84.9	206	78.4
Prometryn	241.4	17.35	241	184	64.5	226	50.8	105	24.8
Propargite	350.5	27.71	135	150	14.6	231	13	64	6.4
Propazine	229.7	13.39	214	229	59.6	172	44.5	58	37
Propetamphos	281.3	13.91	138	194	50.9	236	34.1	222	24.8
Propyzamide	256.1	14.01	173	175	58	145	29.1	255	26.6
Pyrimethanil	199.3	14.15	198	199	47	77	6	200	5.8
Quinalphos	298.3	21.65	146	157	61	118	15.4	156	11.2
Quintozene	295.3	13.69	237	249	75.5	295	65.1	214	63.2
Simazine	201.7	12.97	201	186	58.3	173	39.4	68	25.8

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Pesticide	MW	RT (min)	Target (T)	Qualifier Ion 1 (Q1)	Q1/T (%)	Qualifier Ion 2 (Q2)	Q2/T (%)	Qualifier Ion 3 (Q3)	Q3/T (%)
Tebuconazole	307.8	27.51	125	250	87.4	70	55.4	83	51.3
Tecnazene	260.9	11.4	203	261	83.1	215	81.9	201	80.3
Terbufos	288.4	13.8	231	57	77.5	103	26.9	153	25.4
Terbutylazine	229.7	13.83	214	173	38.3	216	32.3	229	29.8
Terbutryn	241.4	17.99	226	185	64.9	241	62	170	52.4
Tetrachlorovinphos	366	22.95	329	331	97.2	109	57.8	333	33.2
Tetradifon	356.1	29.43	159	111	67.1	229	65	227	63
Thiometon	246.3	12.34	88	125	66.5	89	38.6	93	34.1
Tolyfluanid	347.3	21.27	137	238	41.2	106	5.3	63	3.2
Triadimefon	293.8	19.41	57	208	84	85	31.1	210	27.6
Triadimenol	295.8	21.78	112	168	77	128	48.8	70	26.6
Tri-allate	304.7	14.93	86	268	58.8	270	39.2	128	26.1
Trifluralin	335.3	11.62	306	264	62.9	290	12.4	307	12.1
Vinclozolin	286.1	16.63	212	198	95.6	187	82.9	285	81.4

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Table 3. SIM- 1.

Group	Time (min)	Pesticides and Internal Standards (I.S.)	Ions, amu	Dwell time (ms) Scan rate (cycles/s)
1	5.00	Acenaphthlene-d ₁₀ (I.S.)	80, 160, 162, 164	30 5.41
2	10.00	Tecnazene	201, 203, 215, 261	30 5.41
3	11.75	Diallate I. BHC- α , Diallate II, Hexachlorobenzene, Dicloran	86, 128, 176, 178, 181, 183, 206, 208, 217, 219, 234, 236, 282, 284, 286, 288	25 1.55
4	13.00	Lindane, Pentachloronitrobenzene, Pheneanthrene-d ₁₀ (I.S.), Propyzamide, BHC- δ	111, 145, 173, 175, 181, 183, 184, 187, 188, 189, 214, 217, 219, 237, 249, 295	25 1.46
5	15.50	Vinclozolin, Heptachlor, Alachlor	45, 100, 146, 160, 187, 188, 198, 212, 270, 272, 274, 285	30 1.83
6	17.75	Aldrin, 4,4'-Dichlorobenzophenone	66, 111, 139, 141, 250, 261, 263, 265	30 2.74
7	20.00	Heptachlor epoxide	351, 353, 355, 357	30 5.41

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8	20.90	Penconazole, Captan, Chlozolate	77, 79, 80, 151, 159, 161, 186, 187, 188, 248, 250, 259	30 1.83
9	21.55	Folpet, Triadimenol, Chlorbenseide, Allethrin, Furalaxyl, Procymidone, trans-Chlordane	67, 70, 76, 79, 95, 96, 104, 107, 112, 123, 125, 127, 128, 136, 146, 147, 152, 168, 242, 260, 268, 270, 283, 285, 371, 373, 375, 377	20 1.02
10	22.45	Endosulfan- α , cis-Chlordane	195, 237, 239, 241, 371, 373, 375, 377	30 2.74
11	23.25	Hexaconazole, Dieldrin	79, 82, 83, 214, 216, 263, 277, 279	30 2.74
12	24.35	Endrin, Nitrofen, Endosulfan- β , o,p'-DDT, Endrin aldehyde	67, 165, 195, 202, 207, 235, 236, 237, 241, 250, 253, 263, 283, 285, 315, 317, 319, 345, 347	25 1.24
13	26.35	Benalaxyl, p,p'-DDT	91, 148, 165, 204, 206, 235, 236, 237	30 2.74
14	27.25	Tebuconazole, Captafol	70, 77, 79, 80, 83, 125, 151, 250	30 2.74
15	27.95	Endrin ketone, Chrysene-d ₁₂ , Iprodione, Bromopropylate, Methoxychlor	67, 113, 152, 183, 187, 189, 227, 228, 236, 238, 240, 241, 244, 314, 315, 317, 319, 339, 341, 343	20 1.42

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16	29.15	Tetradifon, Mirex	111, 159, 227, 229, 237, 270, 272, 274	30 2.74
17	31.10	Permethrin I, Permethrin II, Prochloraz	70, 163, 165, 180, 183, 184, 308, 310	30 2.74
18	32.05	Cyfluthrin I-IV	163, 165, 199, 206, 227	30 4.35
19	33.50	Fenvalerate I-II, Fluvalinate tau I-II	125, 152, 167, 169, 181, 209, 250, 252	30 2.74
20	35.50	Deltamethrin	181, 251, 253, 255	30 5.41

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Table 4. SIM- 2.

Group	Time (min)	Pesticides and Internal Standards (I.S.)	Ions, amu	Dwell time (ms) Scan rate (cycles/s)
1	6.00	Acenaphthlene-d ₁₀ (I.S.), Dichlobenil, Eptam	43, 80, 86, 100, 128, 132, 136, 160, 162, 164, 171, 173	30 1.83
2	9.00	Benfluralin, Bromoxynil, Ethalfuralin, Trifluralin	88, 264, 275, 276, 277, 279, 290, 292, 293, 306, 307, 316, 333	30 1.69
3	12.50	Atrazine, Carbofuran, Phenanthrene-d ₁₀ (I.S.), Propazine, Simazine, Terbutylazine	44, 58, 123, 131, 149, 164, 172, 173, 184, 186, 187, 188, 189, 200, 201, 202, 214, 215, 216, 229	30 1.10
4	14.05	Chlorothalonil, Dinoseb, Pyrimethanil, Tri-allate	77, 86, 117, 128, 147, 163, 198, 199, 200, 211, 240, 264, 266, 268, 270	30 1.47
5	15.80	Carbaryl, Desmetryn, Vinclozolin	58, 115, 116, 144, 145, 171, 187, 196, 198, 212, 213, 285	30 1.83
6	17.05	Metalaxyl, Prometryn, Terbutryn	45, 105, 160, 170, 184, 185, 187, 198, 206, 212, 226, 241, 249, 285	30 1.57

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7	18.85	Cyanazine, Cyprodinil, Fenpropimorph, Metolachlor, Nitrothal-isopropyl, Triadimefon	57, 68, 77, 85, 117, 128, 129, 146, 159, 162, 194, 208, 210, 212, 213, 214, 224, 225, 236, 238, 240, 254, 303	20 1.14
8	21.05	Fludioxonil, Imazalil, Myclobutanil, Napropamide, Oxadiazon, Oxyfluorfen, Procymidone	41, 67, 72, 82, 96, 100, 127, 128, 150, 154, 173, 175, 177, 179, 181, 182, 215, 217, 248, 252, 258, 260, 271, 283, 285, 302, 331, 361	20 1.02
9	25.45	Hexazinone, Norflurazon, Oxadixyl, Propargite	45, 71, 83, 102, 105, 128, 132, 135, 145, 150, 163, 171, 231, 303, 305, 350	30 1.38
10	28.00	Chrysene-d ₁₂ (I.S.), Fenpropathrin, Iprodione, Nitalin	97, 125, 181, 187, 189, 236, 238, 240, 241, 244, 265, 274, 300, 314, 316, 317	30 1.38
11	29.95	Cyhalothrin, Fenarimol	77, 107, 139, 181, 197, 208, 209, 219, 251	30 2.44
12	30.95	Bitertanol I- II, Oryzalin	57, 58, 168, 170, 171, 258, 275, 317	30 2.74
13	31.85	Cypermethrin I-IV, Flucythrinate I-II	44, 77, 157, 163, 165, 181, 199, 207, 209	30 2.44

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Table 5. SIM- 3.

Group	Time (min)	Pesticides and Internal Standards (I.S.)	Ions, amu	Dwell time (ms) Scan rate (cycles/s)
1	4.50	Dichlorvos	79, 109, 185, 187	30 5.41
2	6.75	Acephate, Mevinphos, Acenaphthene-d ₁₀	42, 67, 80, 94, 95, 109, 127, 136, 160, 162, 164, 192	30 1.83
3	9.00	Omethoate, Demeton-O	60, 79, 88, 89, 109, 110, 156, 170	30 2.74
4	10.90	Naled (Dibrom), Dicrotophos, Monocrotophos, Phorate	67, 72, 75, 79, 97, 109, 121, 127, 145, 185, 192, 193, 260	30 1.69
5	12.18	Thiometon, Demeton-S, Dimethoate	60, 87, 88, 89, 93, 125, 143, 170	30 2.74
6	13.20	Pheneanthrene-d ₁₀ , Terbufos, Fonophos, Propetamphos	57, 103, 109, 110, 137, 138, 153, 184, 187, 188, 189, 194, 222, 231, 236, 246	30 1.77
7	14.20	Diazinon, Disulfoton	88, 89, 97, 137, 142, 152, 179, 199	30 2.74
8	15.50	Chlorpyrifos-methyl, Parathion-methyl, Malaaxon	79, 99, 109, 125, 127, 263, 286, 288, 290	30 2.44

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9	17.15	Paraoxon	109, 139, 149, 275	30 5.41
10	17.85	Fenitrothion, Dichlofluanid, Malathion	93, 109, 123, 125, 127, 167, 173, 224, 226, 260, 277	30 2.00
11	19.00	Fenthion, Chlorpyrifos, Parathion	97, 109, 125, 139, 169, 197, 199, 278, 291, 314	30 2.20
12	19.85	Bromophos-methyl	125, 329, 331, 333	30 5.41
13	20.90	Chlorvinfenphos, Isofenphos, Quinalphos, Tolyfluanid	58, 63, 106, 118, 121, 137, 146, 156, 157, 213, 238, 255, 267, 269, 323, 325	30 1.55
14	21.95	Methidathion, Bromophos-ethyl, Tetrachlorvinphos	85, 93, 109, 125, 145, 301, 303, 329, 331, 333, 357, 359	30 1.83
15	23.20	Fenamiphos, Profenophos	139, 154, 206, 208, 217, 288, 303, 339	30 2.74
16	25.05	Ethion	97, 125, 153, 231	30 3.64
17	27.50	Carbophenothion, Phosmet, EPN, Azinphos-methyl, Chrysene-d ₁₂ (I.S.)	77, 93, 141, 157, 160, 161, 169, 185, 236, 238, 240, 241	30 1.83

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18	29.15	Phosalone	121, 182, 184, 367	30 5.41
19	30.15	Azinphos-ethyl, Dialifos	76, 77, 105, 132, 160, 173, 208, 210	30 2.74
20	31.20	Coumaphos. Dioxathion	97, 109, 125, 153, 210, 226, 271, 362	30 2.74

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Figure 2. Sample chromatogram for organophosphorous pesticides.

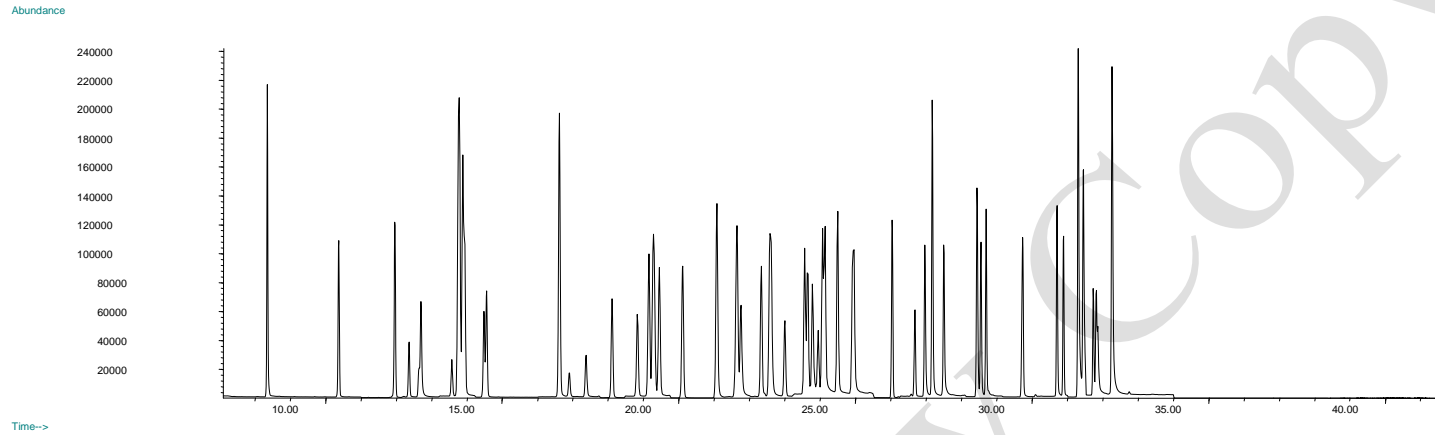
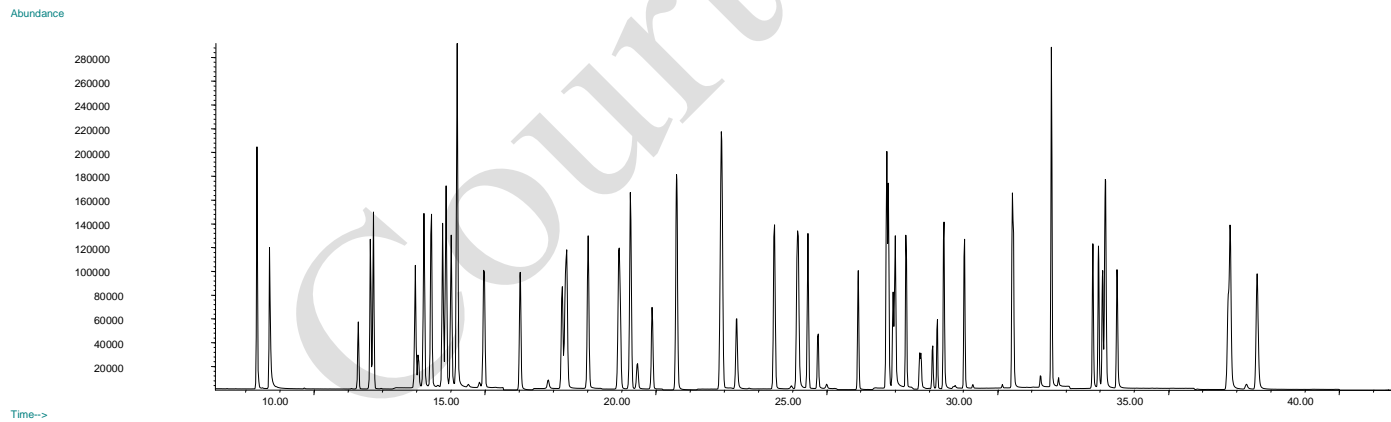


Figure 3. Sample chromatogram for organonitrogen pesticides.

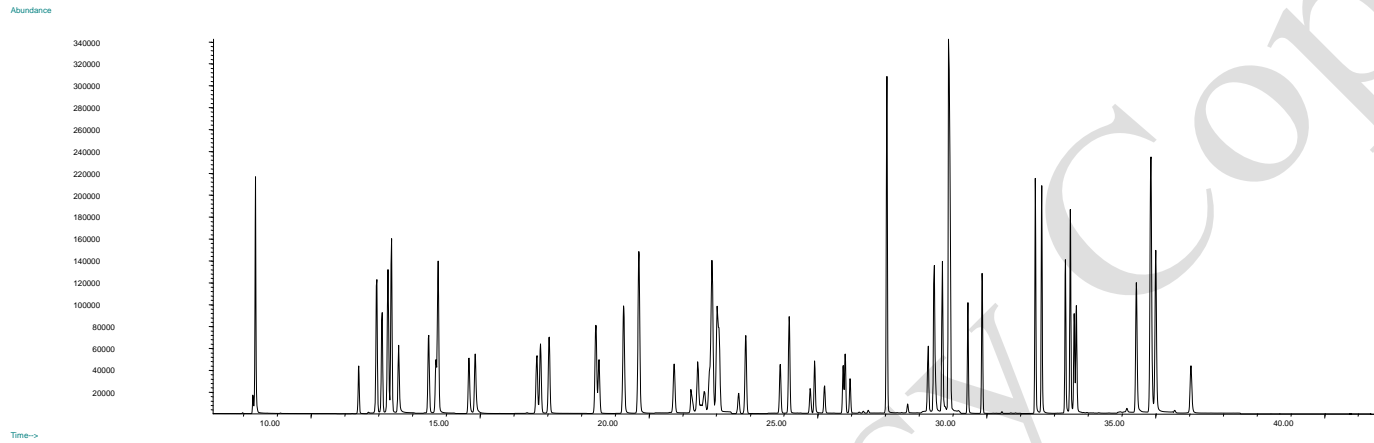


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Figure 4. Sample chromatogram for organohalogen pesticides.



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Table 6: Recoveries at 50 ppb

Compound	Average Spike Recovery (ppb)	Average Spike Recovery (%)	20% of Expected Value (ppb)	20% of Expected Value (% Recovery)
Pyrimethanil	39.87	80	31.90-47.85	64-96
Chlorothalonil	28.14	56	22.51-33.76	45-68
Chlorpyrifos Methyl	30.85	62	24.68-37.02	48-74
Metalaxyl	39.39	79	34.51-47.27	63-95
Procymidone	35.80	72	28.64-42.96	57-86
Penconazole	25.28	51	20.22-30.34	40-61
Oxadixyl	25.29	51	20.23-30.34	40-61
Iprodione	24.42	49	19.54-29.31	39-59