

Analysis of Pesticides in Animal Fluids by LC-MS-MS

Instrumentation: [Agilent HPLC system](#) with [AB Sciex LC/MS/MS System](#)

Column: Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl, 3.0 x 150 mm, 3.5 µm

Elution Type: Gradient

Needle wash: 1:1:1:1 ACN/MeOH/IPA/H₂O w/0.2% FA.

Mobile Phase A: 5 mM ammonium acetate, pH 5.0 in 20:80 MeOH/H₂O

Mobile Phase B: 5 mM ammonium acetate, pH 5.0 in ACN

Gradient Profile:

Step No.	Time (min)	Pct A	Pct B
1	0	80	20
2	0.5	80	20
3	8.0	0	100
4	10.0	0	100
5	13.0	STOP	STOP

Post run: 4 min

Total cycle time: 17 min

Flow Rate: 0.3 mL/min

Col. Temp: 30 °C

Inj. Vol.: 10 µL

Detection: [Tandem Mass Spec \(MS-MS\)](#) @ amu (550 °C) [\(Ctrl + Click to follow link\)](#)

Detector Info: [AB Sciex API 4000](#) [\(Ctrl + Click to follow link\)](#)

MS Conditions

Source: Positive ESI

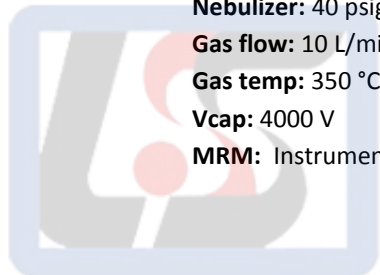
Nebulizer: 40 psig

Gas flow: 10 L/min

Gas temp: 350 °C

Vcap: 4000 V

MRM: Instrument Acquisition Data Used for the Analysis of 13 Pesticides:



Spectralab

Scientific Incorporation

Analyte	MRM channels (m/z)	Fragmentor (V)	CE (V)	RT (min)
Acephate	1) 184.0 > 94.9	60	3	2.55
	2) 184.0 > 111.0		15	
Methamidophos	1) 142.0 > 94.0	60	8	2.54
	2) 142.0 > 124.9		8	
Pymetrozine	1) 218.1 > 105.0	115	20	2.97
	2) 218.1 > 78.0		50	
Carbendazim	1) 192.1 > 160.0	95	18	5.07
	2) 192.1 > 105.0		40	
Imidacloprid	1) 256.1 > 209.1	60	12	5.53
	2) 256.1 > 175.0		18	
Thiabendazole	1) 202.1 > 175.0	110	27	5.65
	2) 202.1 > 131.0		38	
Propoxur	1) 210.1 > 111.0	50	12	6.89
	2) 210.1 > 92.9		15	
Carbaryl	1) 202.0 > 145.0	50	3	7.30
	2) 202.0 > 115.0		40	
Ethoprophos	1) 243.1 > 130.9	80	15	8.50
	2) 243.1 > 172.9		15	
Imazalil	1) 297.1 > 158.9	80	22	8.52
	2) 297.1 > 200.9		15	
Penconazole	1) 284.1 > 158.9	80	32	8.95
	2) 284.1 > 172.9		32	
Cyprodinil	1) 226.1 > 93.0	120	35	9.23
	2) 226.1 > 108.0		35	
Kresoxim methyl	1) 314.0 > 222.1	70	10	9.44
	2) 314.0 > 235.0		10	
TPP (IS)	1) 327.1 > 77.0	70	45	9.49
	2) 327.1 > 151.9		45	

1) Quantifier transition channel
2) Qualifier transition channel

Sample Preparation:

Samples were extracted and cleaned using dispersive SPE. The tubes were capped tightly and vortexed for 1 min. The tubes were centrifuged. A 200 µL aliquot of extract was transferred into an autosampler vial. An aliquot of 10 µL 1% FA in ACN was added immediately. Then 800 µL of water or appropriate standard solutions (prepared in water) were added. The samples were capped and vortexed thoroughly for LC/MS/MS analysis.

Linearity and limit of quantification (LOQ):

The linear calibration range for all of the pesticides tested was 5–250 ng/g. Calibration curves, spiked in matrix blanks, were made at levels of 5, 10, 50, 100, 200, and 250 ng/g. Triphenyl phosphate (TPP) was used as an internal standard at 100 ng/g. A 5 ng/g quantification limit LOQ (5 ppb) was established for all of the pesticides.

References:

- http://www.interchim.fr/cat/QuEChERS_Spinach-5990-4395EN_interchim.pdf
P. Payá, M. Anastasiades; "Analysis of Pesticide Residues Using the Quick Easy Cheap Effective Rugged and Safe (QuEChERS) Pesticide Multiresidue Method in Combination with Gas and Liquid Chromatography and Tandem Mass Spectrometric Detection," *Anal Bioanal Chem.*, 2007, 389, 1697-1714.
L. Zhao, J. Stevens, "Analysis of Pesticide Residues in Spinach Using Agilent SampliQ QuEChERS AOAC Kit by LC/MS/MS Detection. Agilent Technologies publication 5990-4248EN.