


# TN-1112

# APPLICATIONS

## Pesticide Screen from Food Samples using a Synergi™ 2.5 µm Fusion-RP HPLC Column

The chemistry contained within Synergi Fusion-RP HPLC columns offers a balanced combination of hydrophobic and polar selectivities, allowing for separation of compounds that exhibit polar and hydrophobic characteristics such as pesticides.

 For AB SCIEX mass spectrometer users, this method can be instantly implemented by installing the iMethod™ Test. iMethod Tests are verified across several laboratories and contain everything you will need to start running samples including sample preparation recommendations, consumables, LC running conditions, optimized MRM parameters, reporting templates, and complete method documentation.

Visit [www.phenomenex.com/iMethod](http://www.phenomenex.com/iMethod) for more information about available iMethod Tests.

### Sample Preparation

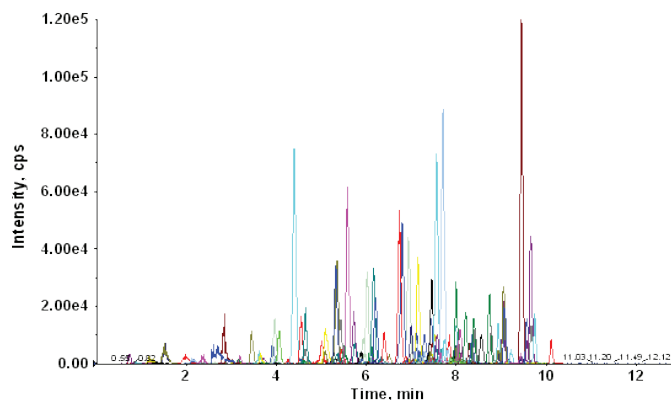
#### QuEChERS Procedure following the EN 15662 Method<sup>1</sup>

- Blend 10 g of fruit or vegetable to be analyzed.
- Shake vigorously in 10 mL of acetonitrile.
- Add 1 pack of pre-packaged QuEChERS extraction salts containing 4 g of anhydrous magnesium sulfate, 1 g of sodium chloride, 1 g of trisodium citrate dihydrate, and 0.5 g of disodium hydrogen citrate sesquihydrate.
- Mix immediately and vigorously for one minute using a vortex mixer.
- Centrifuge the sample at > 3000 g for five minutes.
- Transfer 1 mL aliquots of the resulting raw extracts into a pre-packaged QuEChERS dispersive extraction tube containing 150 mg of anhydrous magnesium sulfate and 25 mg of primary and secondary amine (PSA) sorbent.
- Shake vigorously for 30 seconds then centrifuge at > 3000 g for five minutes.
- Transfer 500 µL of supernatant to an autosampler vial and mix with 500 µL of water.

#### QuEChERS Procedure following the AOAC 2007.01 Official Method<sup>2</sup>

- Homogenize fruit or vegetable using a Molinex mixer or similar apparatus.
- Add 15 mL of acetonitrile to 15 g of homogenized sample, sonicate for two minutes then mix using a roller mixer for five minutes.
- Add pre-packaged QuEChERS dispersive salts containing 6 g of magnesium sulfate and 1.5 g of sodium acetate.
- Mix immediately and vigorously for one minute.
- Centrifuge at > 1500 g for five minutes.
- Transfer 1 mL of the upper layer into a pre-packaged QuEChERS dispersive extraction tube containing 150 mg of magnesium sulfate and 50 mg of primary and secondary amine (PSA) sorbent.
- Shake the tubes well, by hand, for 30 seconds.
- Centrifuge sample at > 1500 g for one minute.
- Evaporate to dryness.
- Dissolve residue in 100 µL of acetonitrile and add 900 µL of water to reconstitute.
- Centrifuge the extract at > 1500 g for one minute.

### HPLC Methodology



#### LC/MS/MS Conditions

**Column:** Synergi Fusion-RP 2.5 µm 100 Å  
**Dimensions:** 50 x 2.0 mm  
**Part No.:** 00B-4423-B0  
**Mobile Phase:** A: Water/Methanol (90:10) + 5 mM Ammonium formate  
 B: Methanol/Water (90:10) + 5 mM Ammonium formate  
**Gradient:**

Time (min)	% B
0	0
1	0
15	100
18	100
18.05	0
20	0

**Flow Rate:** 400 µL/min  
**Temperature:** 40 °C  
**Sample:** For a complete list of analytes visit [www.phenomenex.com/foodsafety](http://www.phenomenex.com/foodsafety)

### Results

Representative recoveries, signal-to-noise (S/N) ratios and estimated detection limits for 130 selected pesticides spiked at 25 ng/mL in cucumber matrix using QuEChERS sample processing technique. For a complete list of analytes visit [www.phenomenex.com/foodsafety](http://www.phenomenex.com/foodsafety).

Analyte	% Recovery	S/N*	% CV	Estimated detection limit (ng/mL)**
Aldicarb-sulfoxide	97.0	26	4.2	9.6
Aldoxycarb	96.4	33.2	10.0	7.5
Alloxydim	60.8	32.2	7.5	7.8
Aminocarb	84.2	84	2.6	3.0
Atrazine	65.1	34	2.4	7.4
Atrazine-2-hydroxy	79.9	77.8	2.9	3.2
Atrazine-desethyl-2-hydroxy	104.1	38.8	1.9	6.4
Benalaxyl	94.9	39.5	18.1	6.3
Bendiocarb	120.2	33.1	16.2	7.6

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Analyte	% Recovery	S/N*	% CV	Estimated detection limit (ng/mL)**
Benzoximate	15.3	29.3	10.0	8.5
Boscalid	99.0	14.3	18.7	17.5
Bromacil	106.2	15.9	8.4	15.7
Bupirimate	102.2	43.8	9.0	5.7
Butocarboxim-sulfoxide	98.6	37.7	2.3	6.6
Buturon	98.4	20.4	4.1	12.3
Carbaryl	94.3	44.7	1.5	5.6
Carbetamide	111.1	58.8	4.9	4.3
Chloridazon	149.2	35.5	1.7	7.0
Chlorotoluron	69.5	38.1	2.4	6.6
Chloroxuron	88.6	24.7	11.0	10.1
Cinosulfuron	107.2	28.8	18.9	8.7
Clomazone	140.8	42.3	1.8	5.9
Cloquintocet-mexyl	120.8	95.8	10.1	2.6
Coumaphos	115.6	24.4	3.6	10.2
Cyanazine	48.6	22.1	20.1	11.3
Cyproconazole	120.2	24.6	7.1	10.2
Cyprodinil	90.0	11.3	7.6	22.1
Demeton-S-methyl-sulfone	100.5	32.8	1.9	7.6
Diazinon	88.3	81.9	7.2	3.1
Diclobutrazol	112.7	26.3	7.6	9.5
Dicrotophos	28.5	58.1	19.1	4.3
Diethofencarb	104.0	16.2	8.0	15.4
Difenoxuron	92.5	51	5.3	5.0
Dimefuron	88.8	20	6.6	12.6
Dimethachlor	100.5	13	11.8	19.5
Dimethenamide	106.0	39	2.7	6.5
Dimetilan	67.0	101	2.5	2.5
Dodemorph	139.0	107	3.0	2.3
Ethidimuron	113.0	28	6.5	8.9
Ethiofencarb-sulfone	99.1	59	14.7	4.3
Ethiofencarb-sulfoxide	113.9	119	3.6	2.1
Ethion	99.6	17	7.2	14.8
Ethirimol	112.9	42	4.9	6.0
Ethoprophos	93.3	16	14.1	15.2
Etrimfos	93.0	64	4.6	3.9
Fenbuconazole	98.0	21	2.2	11.9
Fenfuram	94.9	46	7.7	5.5

Analyte	% Recovery	S/N*	% CV	Estimated detection limit (ng/mL)**
Fenhexamid	47.9	15	15.1	17.0
Fenoxaprop-P-ethyl	113.9	32	7.2	7.8
Fenpropimorph	130.8	152	4.2	1.6
Fenpyroximate	161.6	15	8.4	16.8
Fenuron	102.8	47	5.5	5.3
Flamprop-M-isopropyl	104.1	55	4.1	4.5
Flamprop-M-methyl	90.2	20	3.7	12.4
Flazasulfuron	100.8	74	7.5	3.4
Fluazifop-butyl	144.5	50	3.6	5.0
Flufenacet	89.7	22	12.8	11.5
Fluometuron	120.9	77	2.3	3.2
Fluridone	68.4	30	11.8	8.4
Flurtamone	89.7	20	5.2	12.3
Fuberidazole	102.7	125	6.2	2.0
Haloxifop-P-methyl	106.4	15	3.2	16.9
Hexaconazole	100.9	23	2.1	11.0
Hexazinone	106.3	202	15.8	1.2
Imazalil	117.7	34	18.1	7.3
Isazofos	74.9	13	8.0	19.1
Isoprothiolane	62.1	77	1.2	3.2
Lenacil	111.3	43	6.1	5.8
Malaoxon	44.7	50	9.5	5.0
Malathion	94.7	30	5.9	8.3
Mepanipyrim	79.5	3	7.9	80.6
Metconazole	118.2	19	7.3	13.4
Methamidophos	90.9	33	6.5	7.6
Methfuroxam	66.2	96	1.1	2.6
Methidathion	47.9	35	4.2	7.2
Metobromuron	116.1	19	4.0	12.9
Metolachlor	82.7	22	2.6	11.4
Metosulam	99.7	51	2.7	4.9
Monuron	100.3	22	2.8	11.6
Napropamide	103.7	31	11.5	8.1
Oxasulfuron	87.8	78	6.6	3.2
Oxycarboxin	98.5	110	5.3	2.3
Paclobutrazol	105.4	48	9.2	5.2
Phosphamidon	95.2	104	6.5	2.4
Picoxystrobin	133.1	104	3.8	2.4

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Analyte	% Recovery	S/N*	% CV	Estimated detection limit (ng/mL)**
Pirimiphos-ethyl	129.7	57	4.1	4.4
Pirimiphos-methyl	105.1	63	3.1	4.0
Prometon	100.5	105	6.0	2.4
Prometryne	49.5	14	19.5	18.4
Propamocarb	106.5	20	1.9	12.8
Propazine-2-hydroxy	81.5	19	3.6	12.9
Propazine	72.8	11	10.7	22.5
Prosulfocarb	114.9	145	6.1	1.7
Pymetrozine	101.4	115	2.6	2.2
Pyraclastrobin	141.1	24	9.6	10.5
Pyrazophos	108.9	55	4.8	4.5
Pyridaben	102.8	18	4.1	14.1
Pyridaphenthion	88.6	10	6.3	25.3
Pyrimethanil	40.1	18	5.8	13.6
Pyriproxyfen	153.8	51	4.3	4.9
Quinalphos	124.9	17	7.3	14.8
Quinmerac	104.9	116	1.3	2.2
Sebuthylazine	56.9	55	3.6	4.6
Sebuthylazine-desethyl	88.6	39	7.1	6.5
Sethoxydim	82.5	28	10.1	8.9
Siduron	59.7	14	5.3	17.5
Simazine-2-hydroxy	105.5	99	10.6	2.5
Simetryn	95.7	96	6.6	2.6
Spiroxamine	97.1	230	6.0	1.1
Sulfometuron-methyl	98.2	103	5.1	2.4
Tebuconazol	87.3	45	9.6	5.5
Tebufenpyrad	125.2	13	10.6	18.8
Tebutam	87.2	22	7.3	11.4
Tebuthiuron	97.8	61	14.4	4.1
Terbuthylazine	31.7	18	3.3	14.1
Terbuthylazine-desethyl	88.7	39	8.1	6.5
Terbutryn	31.9	119	6.0	2.1
Tetrachlorvinphos	101.3	20	8.7	12.5

Analyte	% Recovery	S/N*	% CV	Estimated detection limit (ng/mL)**
Tetraconazole	82.1	32	16.3	7.8
Thifensulfuron-methyl	100.8	74	4.2	3.4
Triasulfuron	80.5	35	7.5	7.2
Triazophos	31.9	19	9.9	13.2
Tricyclazole	93.8	72	1.9	3.5
Trietazine	75.4	12	13.6	21.0
Trifloxystrobin	113.9	83	5.8	3.0
Triflumizole	122.4	25	11.9	10.1
Triticonazole	117.1	24	5.8	10.6
Uniconazole	66.5	24	24.0	10.6
Vamidithion	113.1	36	13.9	7.0

\* S/N obtained at three standard deviations on a 3200 QTRAP® LC/MS/MS system.

\*\* Estimated detection limit based on estimated signal at 10x the S/N ratio.

### References

1. EN 15662 (2007), Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and Clean-up by Dispersive SPE—QuEChERS method.
2. AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.

# TN-1112 APPLICATIONS

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## Ordering Information



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Visit [www.phenomenex.com/iMethod](http://www.phenomenex.com/iMethod) for more information about available iMethod Tests.

## Complete iMethod™ Kit\*

Description	Part No.
iMethod Test for Pesticide Screening V2.1 for Cliiquid® Software	KH0-8979

\*Kit contains downloadable iMethod, Synergi™ Fusion-RP 2.5 µm HPLC column with SecurityGuard™ cartridges and holder, in-line filter, check standards, Phenex™ Syringe Filters, Verex™ Vial kit and Sure-Lok™ Fingertight Nut.

## Synergi Fusion-RP HPLC Columns

### 2.5 µm High Speed Technology (HST) Columns (mm)

Phase	30 x 2.0	50 x 2.0	100 x 2.0	50 x 3.0	100 x 3.0	50 x 4.6
Fusion-RP	00A-4423-B0	00B-4423-B0	00D-4423-B0	00B-4423-Y0	00D-4423-Y0	00B-4423-E0

### 2.5 µm MercuryMS LC/MS Cartridges (mm)

Phase	10 x 2.0	10 x 4.0	20 x 2.0	20 x 4.0	20 x 2.0	20 x 4.0
Fusion-RP	00N-4423-B0-CE	00N-4423-D0-CE	00M-4423-B0-CE	00M-4423-D0-CE	00M-4423-B0	00M-4423-D0

Columns (mm)

### 4 µm Synergi Capillary Columns (mm)

Phase	50 x 0.30	150 x 0.30	50 x 0.50	150 x 0.50	250 x 0.50	Guard Column (mm)
Fusion-RP	00B-4424-AC	00F-4424-AC	00B-4424-AF	00F-4424-AF	00G-4424-AF	03M-4424-AC

### 4 µm Microbore and Minibore Columns (mm)

Phase	50 x 1.0	150 x 1.0	250 x 1.0	30 x 2.0	50 x 2.0	75 x 2.0	150 x 2.0	250 x 2.0	4 x 2.0*
Fusion-RP	00B-4424-A0	00F-4424-A0	—	00A-4424-B0	00B-4424-B0	00C-4424-B0	00F-4424-B0	00G-4424-B0	AJ0-7556 for ID: 2.0-3.0 mm

SecurityGuard Cartridges (mm)

### 4 µm Narrow Bore Columns (mm)

Phase	30 x 3.0	50 x 3.0	150 x 3.0	250 x 3.0	4 x 2.0*
Fusion-RP	00A-4424-Y0	00B-4424-Y0	00F-4424-Y0	00G-4424-Y0	AJ0-7556 for ID: 2.0-3.0 mm

SecurityGuard Cartridges (mm)

### 4 µm Analytical Columns (mm)

Phase	30 x 4.6	50 x 4.6	75 x 4.6	150 x 4.6	250 x 4.6	4 x 3.0*
Fusion-RP	00A-4424-E0	00B-4424-E0	00C-4424-E0	00F-4424-E0	00G-4424-E0	AJ0-7557 for ID: 3.2-8.0 mm

SecurityGuard Cartridges (mm)

\* SecurityGuard Analytical cartridges require holder, Part No.: KJ0-4282



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