



## LC-MS/MS Analysis of Anionic Polar Pesticides in Fruits and Vegetables Using a Luna™ 3 µm Polar Pesticides HPLC Column

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### Overview

Many polar pesticides used in conventional agriculture are difficult to retain on standard C18 reversed phase HPLC columns. In this application note, we show a fast and robust method for the LC-MS/MS determination and quantification of several common anionic polar pesticides in fruits and vegetables after sample preparation using the QuPPE method. As the samples we tested were from plant origin, we followed the QuPPE-PO-Method suggested by the EU Reference Laboratories for Residues of Pesticides – Single Residue Methods (EURL-SRM).

The presented gradient reversed phase HPLC method using a Luna Polar Pesticides column for the analysis of polar pesticides is a robust solution for this demanding application. The recovery of the analytes (**Table 3**) and linearity of the method for the tested analytes (**Figure 1**) demonstrates the suitability of this method for the routine analysis in food testing laboratories.

### LC Conditions

**Column:** Luna 3 µm Polar Pesticides

**Dimension:** 100 x 2.1 mm

**Part No.:** OOD-4798-AN

**Mobile Phase:** A: 0.1 % Formic Acid in Water  
B: 0.1 % Formic Acid in Acetonitrile

Gradient: Time (min)	%B
0	5
0.5	5
1	5
5	65
5.1	90
8	90
8.1	5

**Flow Rate:** 0.4 mL/min

**Injection Volume:** 20 µL

**Temperature:** 40 °C

**Instrument:** Shimadzu® Nexera®

**Detection:** MRM

**Detector:** SCIEX® 6500+ Triple Quadrupole MS

### MS Conditions

**Gas Temperature:** 450 °C

**GS1:** 35 psi

**GS2:** 25 psi

**Curtain Gas:** 25 psi

**Ion Spray Voltage:** 4500 V

**CAD:** 9 psi



**Table 1.** MRM Transitions, Including Internal Standards (ISTD).

Analyte	Q1 (m/z)	Q3 (m/z)	Expected t <sub>R</sub> (min)	Start t (min)	End t (min)	CE (V)
AMPA ISTD	112.00	63.00	0.8	0.0	1.6	-28
AMPA 1	110.00	63.00	0.8	0.0	1.6	-24
AMPA 2	110.00	79.00	0.8	0.0	1.6	-34
Chlorate ISTD	89.00	71.00	2.7	1.8	3.5	-28
Chlorate 1	83.00	67.00	2.7	1.8	3.5	-28
Chlorate 2	85.00	69.00	2.7	1.8	3.5	-30
Fosetyl ISTD	114.00	82.00	2.7	1.9	3.5	-20
Fosetyl 1	109.00	81.00	2.7	1.9	3.5	-20
Fosetyl 2	109.00	63.00	2.7	1.9	3.5	-38
Glyphosate ISTD	171.00	63.00	1.5	0.7	2.4	-27
Glyphosate 1	168.00	63.00	1.5	0.7	2.4	-27
Glyphosate 2	168.00	79.00	1.5	0.7	2.4	-50
Glyphosate 3	168.00	81.00	1.5	0.7	2.4	-21
Phosphonic Acid ISTD	87.00	85.00	2.1	1.3	2.9	-21
Phosphonic Acid 1	81.00	78.80	2.1	1.3	2.9	-21
Phosphonic Acid 2	81.01	63.00	2.1	1.3	2.9	-32
Perchlorate ISTD	107.00	89.00	3.4	2.5	4.2	-33
Perchlorate 1	99.00	83.00	3.4	2.5	4.2	-33
Perchlorate 2	101.00	85.00	3.4	2.5	4.2	-35

**Figure 1.** Calibration Curves.

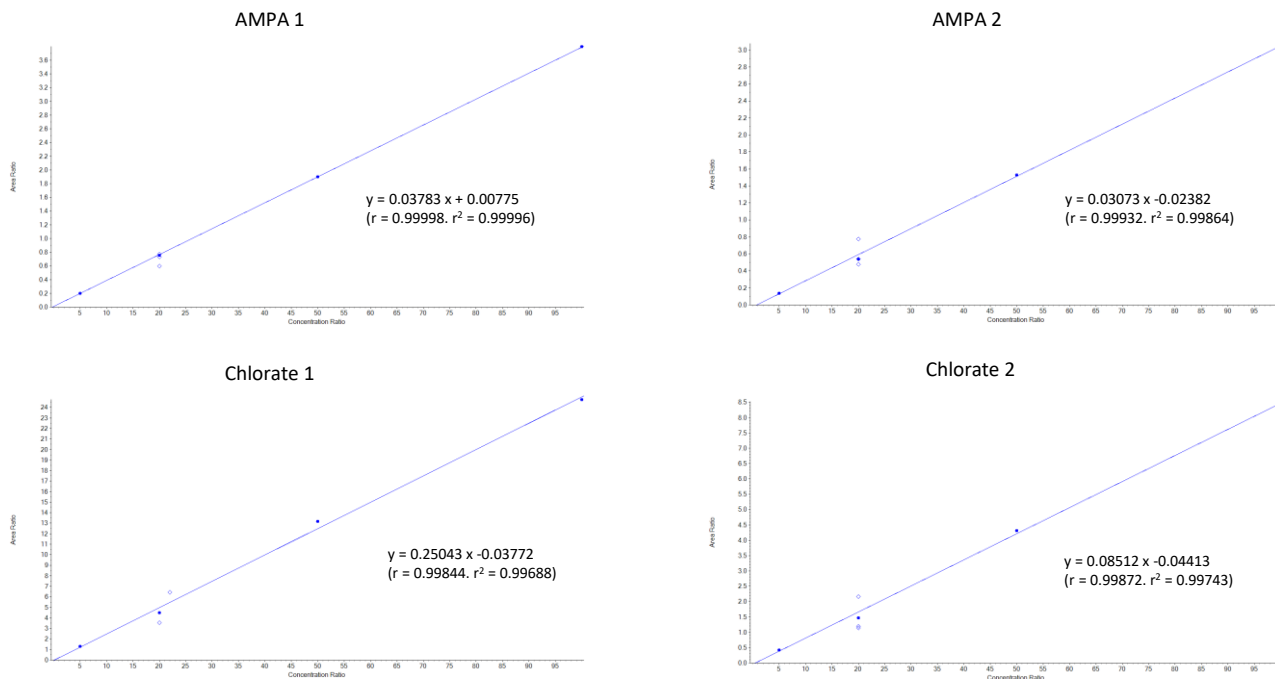


Figure 1 (Cont'd). Calibration Curves.

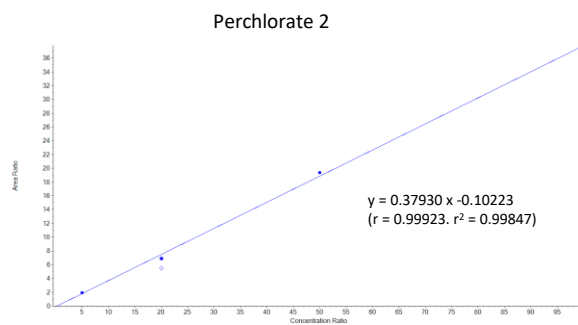
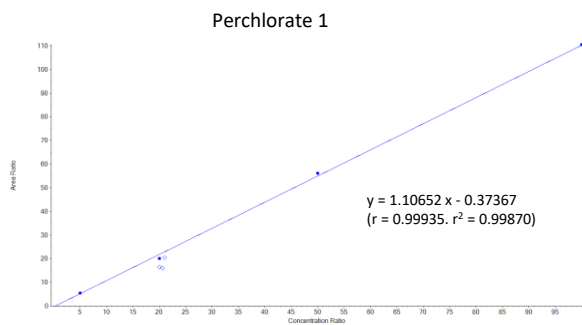
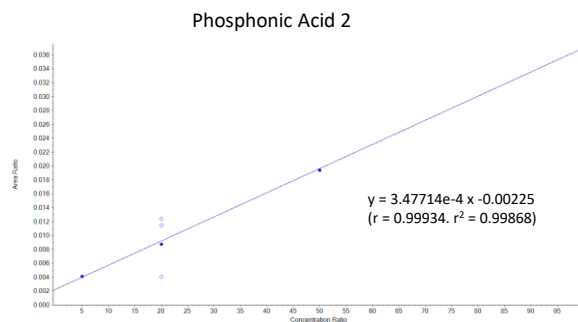
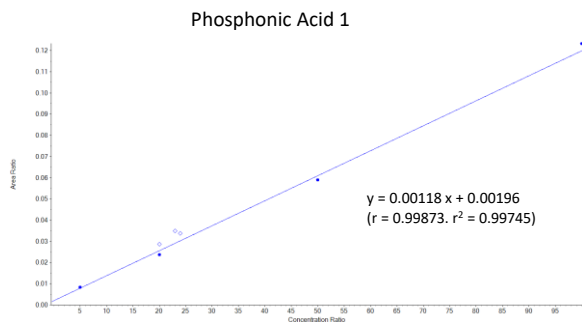
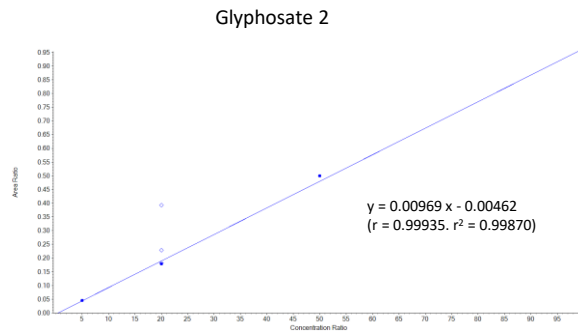
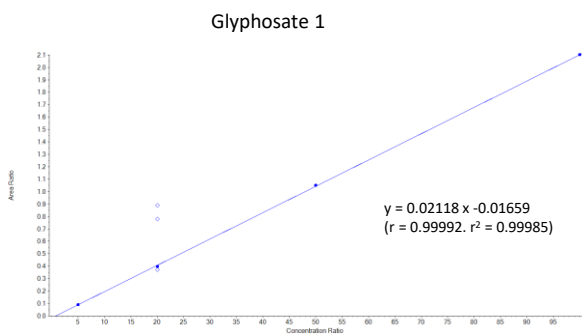
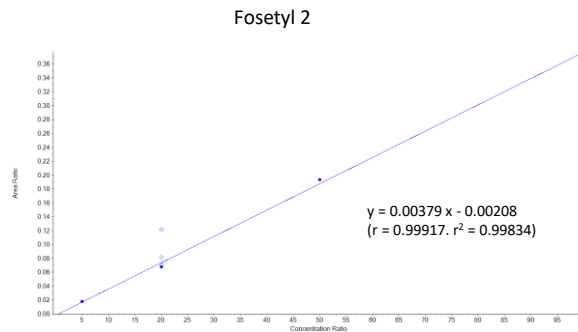
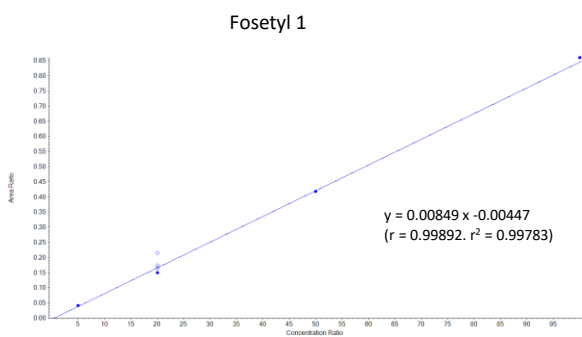


Figure 2. Extracted Ion Chromatograms.

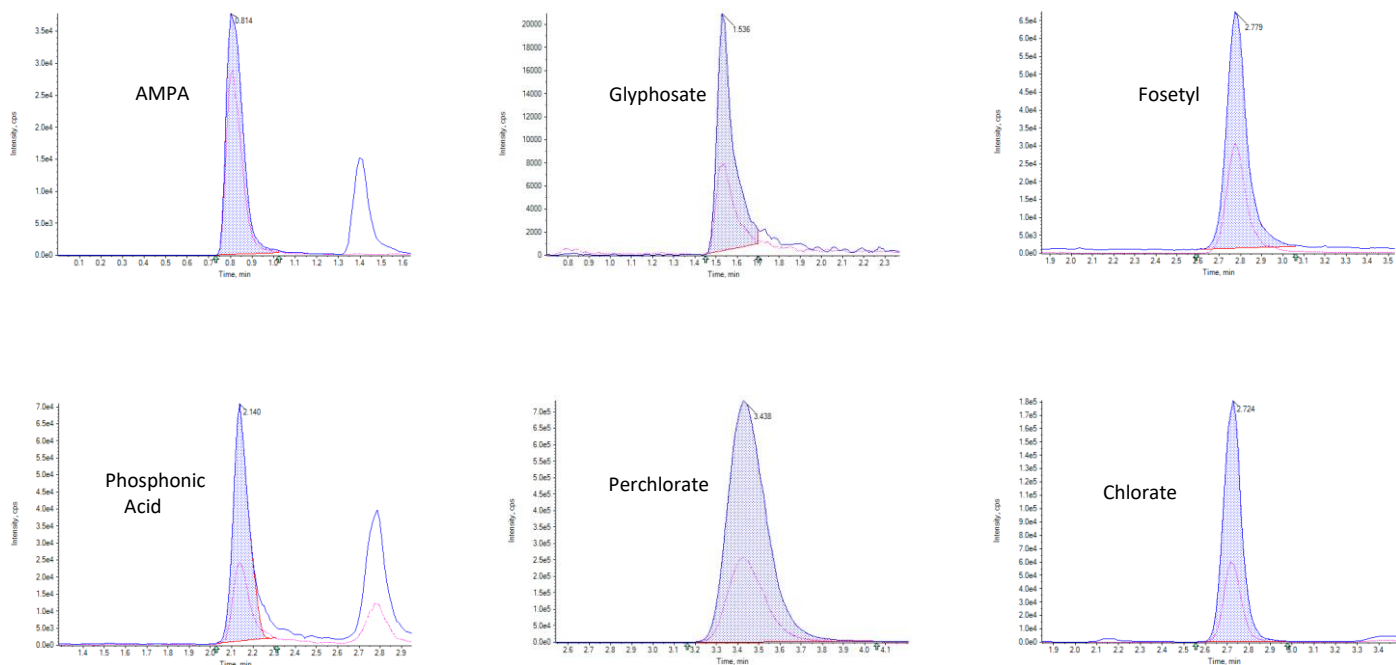


Table 2. Recovery Data for Various Fruit and Vegetable Matrices.

Analyte	Grape			Tomato			Tangerine		
	Spiked Conc. (ppb)	Exp. Conc. (ppb)	Accuracy (%)	Spiked Conc. (ppb)	Exp. Conc. (ppb)	Accuracy (%)	Spiked Conc. (ppb)	Exp. Conc. (ppb)	Accuracy (%)
AMPA 1	20	22.1	110.59	20	16.9	84.56	20	18.8	94.09
AMPA 2	20	31.4	156.95	20	20.5	102.43	20	20.9	104.64
Chlorate 1	20	16.6	83.22	20	14.7	73.36	20	15.1	75.25
Chlorate 2	20	17.1	85.53	20	14.8	74.12	20	15.9	79.37
Fosetyl 1	20	21.6	108.00	20	20.6	102.96	20	19.6	97.80
Fosetyl 2	20	23.1	115.50	20	21.4	106.97	20	21.1	105.34
Glyphosate 1	20	21.7	108.47	20	18.8	94.03	20	16.9	84.55
Glyphosate 2	20	20.6	103.24	20	19.8	98.96	20	19.5	97.72
Phosphonic Acid 1	30	29.4	98.02	20	21.6	108.13	28	29.3	104.65
Phosphonic Acid 2	30	34.2	113.87	20	19.6	98.19	28	29.8	106.37
Perchlorate 1	20	15.6	78.06	20	15.7	78.31	20	19.6	97.94
Perchlorate 2	20	15.4	76.93	20	15.4	76.99	20	19.4	97.04



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