Supporting Information

Development of green and facile sample preparation method for the determination of seven neonicotinoids in fresh vegetables, and dissipation and risk assessment of imidacloprid and dinotefuran

Osama I Abdallah[1*], Rania M. Abd El-Hamid[1], Nevein S. Ahmed[1], Saleh S. Alhewairini[2]. Sherif B. Abdel Ghani[2, 3*]

[1] Department of Pesticide Residues and Environmental Pollution, Central Agricultural Pesticide Laboratory, Agricultural Research Center, Giza, 12618, Egypt.

[2] Department of Plant Protection, College of Agriculture and Food, Qassim University, P.O. Box 6622, Buraydah, Saudi Arabia.

[3] Department of Plant Protection, Faculty of Agriculture, Ain Shams University, P.O. Box 68 Hadayek Shoubra, 11241 Cairo, Egypt.

[*] Corresponding author.

E-mail addresses: oiabdullah@qassim.gov.sa (O. I. A.), sherifbiomy@yahoo.com (S. B. A.)

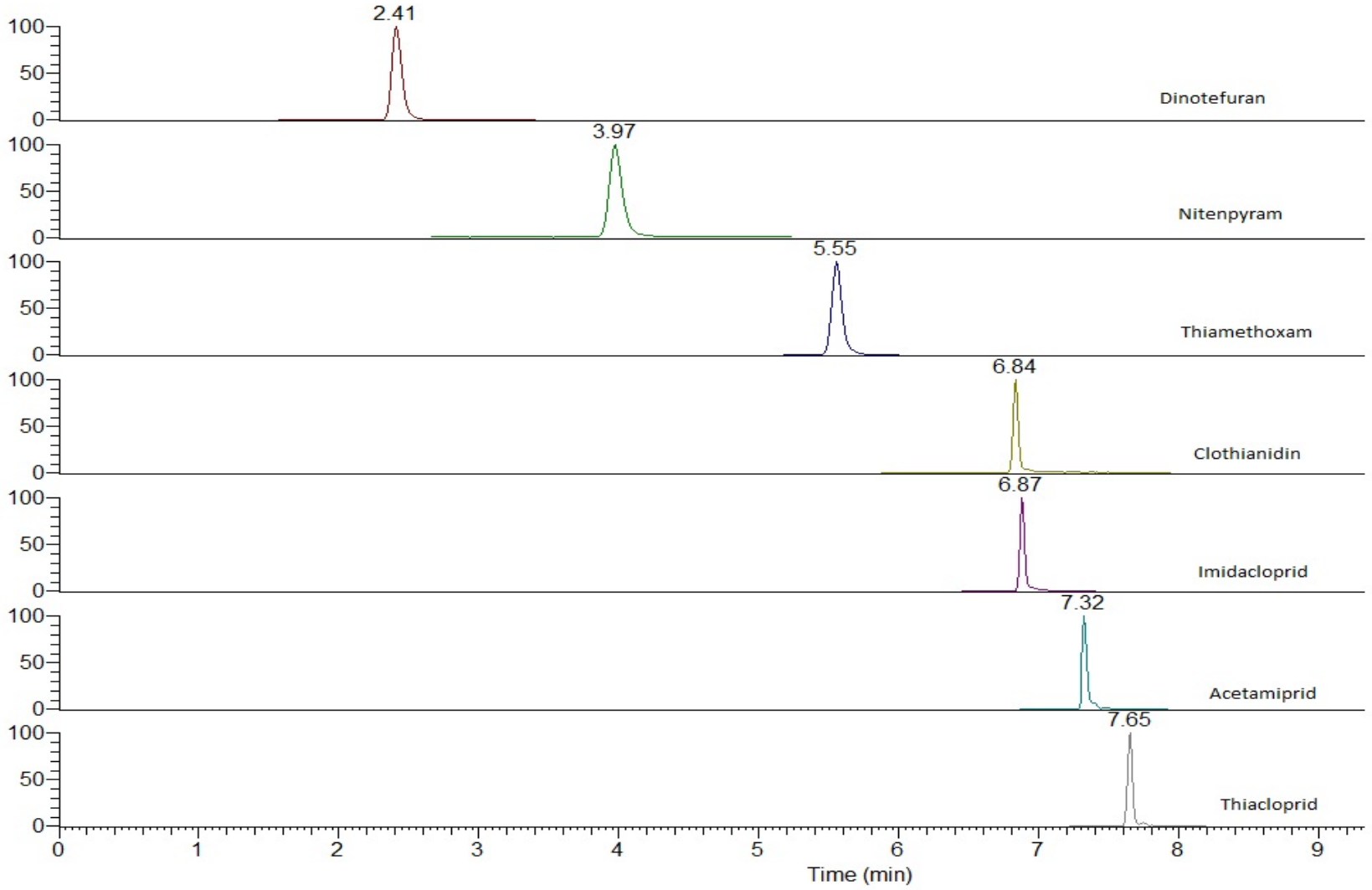
2.41
Dinotefuran
3.97
Nitenpyram
5.55
Thiamethoxam
6.84
Clothianidin
6.87
Imidacloprid
7.32
Acetamiprid
7.65
Thiacloprid
100
50
0
0
1
2
3
4
5
6
7
8
9
Time (min)

Figure S1. Representative chromatograms of the 7 separated neonicotinoids at concentration of 0.01 ةg/L in DI water using LC-MS/MS

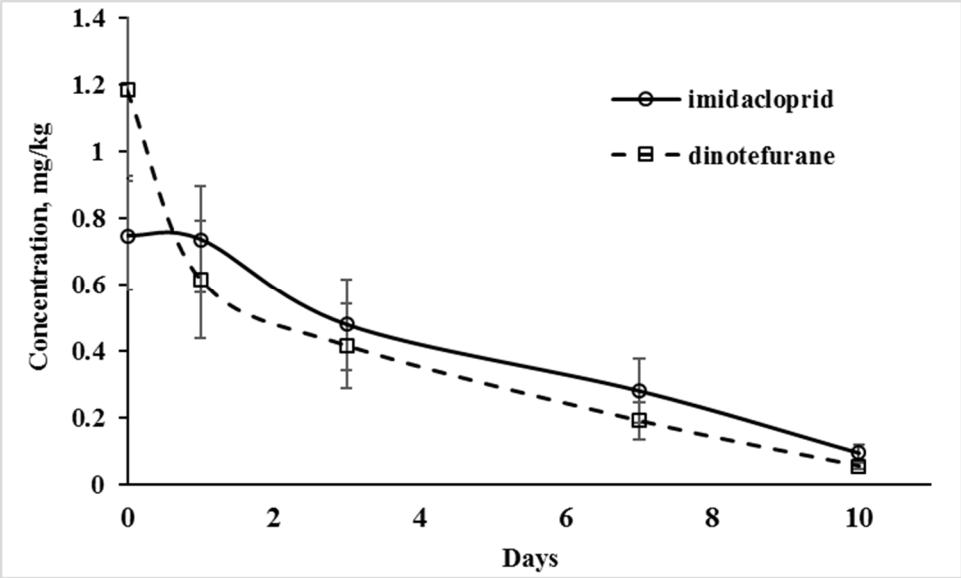

Figure S2. Residues of imidacloprid and dinotefuran in tomatoes under field conditions.

Table S1. LC–MS/MS parameters for determination of neonicotinoids

	Retention Time (min)	Precursor (m/z)	Product (m/z)	Collision Energy (V)	RF Lens (V)
Dinotefuran	2.51	203	113.1	10.2	36
			129.05[a]	12.4	36
Nitenpyram	4.11	271.06	189.1	14.2	50
			225.1	12.09	50
Thiamethoxam	5.67	292.08	181.08	23.7	40
			211.1	12.3	40
Clothianidin	6.88	250.03	131.9	17.09	46
			169.05	13.2	46
Imidacloprid	6.91	256.1	175.08	20.08	50
			209.1	16.7	50
Acetamiprid	7.34	223.03	90.07	34.7	52
			125.9	21.6	52
Thiacloprid	7.66	253	99.04	42.2	59
			126.04	21.8	59

[a] Product ions in bold were utilized for quantification

Table S2. Comparison of the extraction/clean-up step, recovery, and LOQ of neonicotinoids in the previous studies with the current study

No of analytes	Matrices	Instrument	Extraction/cleanup	% Recovery	LOQ (µg/kg)	Ref.
5	Vegetables and fruits	LC-MS (APCI)	-Sample weight: 20 g -Extraction solvents: 95 mL methanol -Clean-up: SPE cartridge	70-95	10 (20 for nitenpyram)	[30]
5	Apple	LC-MS/MS (APCI)	-Sample weight: 10 g -Extraction solvents: 10 mL acetonitrile, followed by salting out step. -Clean-up: dSPE using primary secondary amine (PSA)	93-108	2-3	[33]
3	Cabbage, Tomatoes, Chilli, Peppers, and Potatoes	HPLC-UV	-Sample weight: 1 g -Extraction solvents: Microwave Assisted Extraction (MAE), acetone, n-hexane, and CH_2CL_2	68.1-106	3.2-4.8	[32]
4	Tomatoes Cucumber	HPLC-UV	-Sample weight: 10 g (DSPE) -Extraction solvents: 10 mL acetonitrile, followed by salting out step. -Clean-up: dSPE using PSA and MWCNTs, followed by DLLME -Procedure for enrichment using $CHCL_3$	84.6-97.5	0.5-1	[34]
6	Spinach, Cucumber, Apple, and Pomelo	LC-MS/MS	-Sample weight: 10 g -Extraction solvents: 10 mL acetonitrile, then salting out -Clean-up: dSPE using primary secondary amine (PSA) and graphitized carbon black (GCB)	73.7-103.8	0.66-2.84	[35]
4	cereals, vegetables, fruits	UPLC-MS/MS	-Sample weight: 10 g -Extraction solvents: 5 mL water and 10 mL acetonitrile, followed by salting out step. -Clean-up: dSPE using primary secondary amine (PSA) and graphitized carbon black (GCB)	82.7-103.4	0.71-2.26	[31]
7	Cucumber, Eggplant	HPLC-UV	-Sample weight: 5 g -Extraction solvents: 25 mL water -Clean-up: SPE, Oasis HLB, and Envi-Carb cartridges	82-114	7-17, 3-19	[36]
4	Diverse crops	HPLC-UV	-Sample weight: 10 g -Extraction solvents: 5 mL water and 5 mL acetonitrile, followed by salting out step. -Clean-up: dSPE using PSA and GCB.	77.5-96.4	0.2 µg/mL	[37]
7	Tomatoes Cucumber Lettuce	UPLC-MS/MS	-Sample weight: 3 g -Extraction solvents: 27 mL water -No cleanup step.	91.9-113.7	10	This study