animals

MDPI

Supplementary Materials

Detection of antimicrobial residues in poultry litter: monitoring a risk through a selective and sensitive HPLC-MS/MS method

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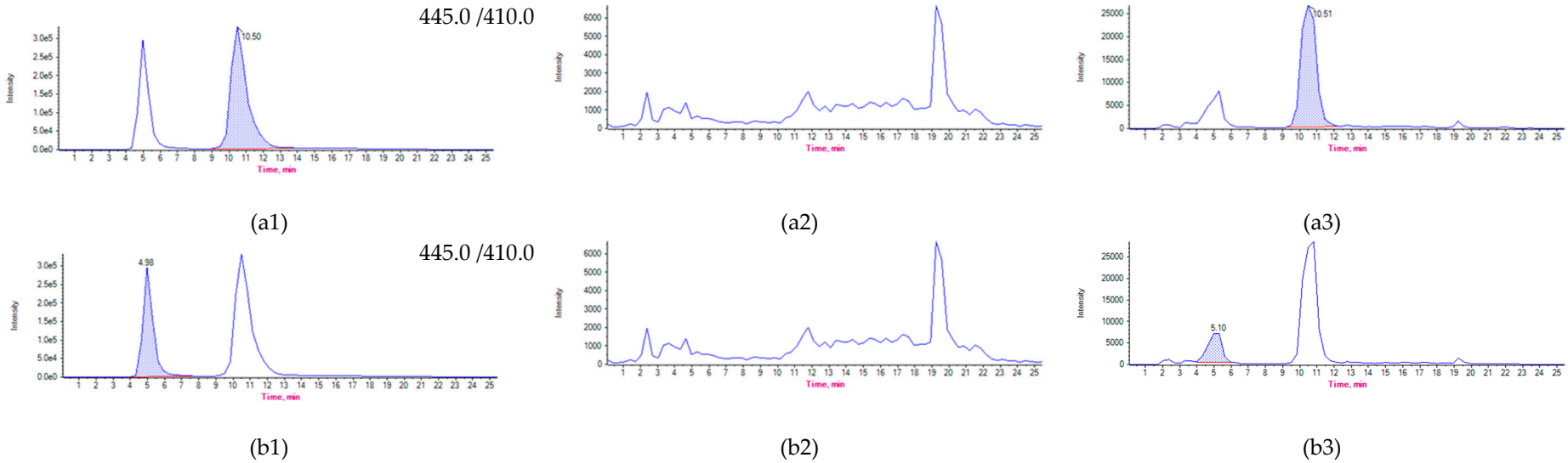

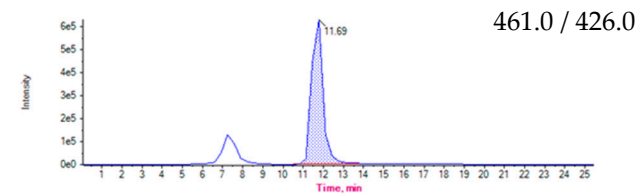

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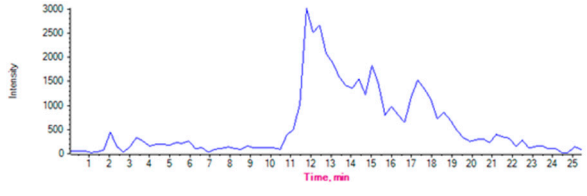

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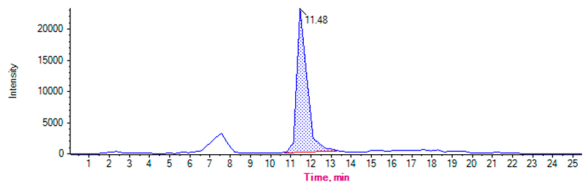

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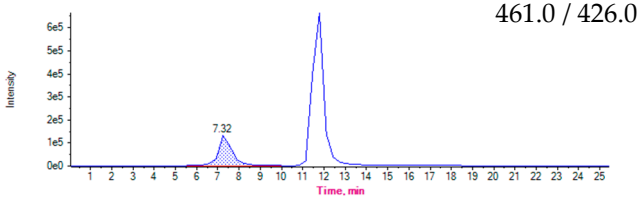

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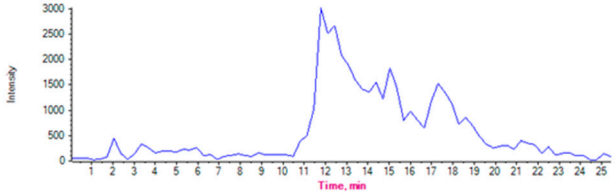

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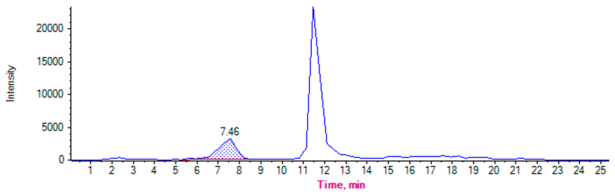

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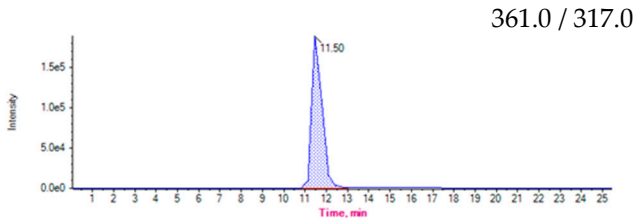

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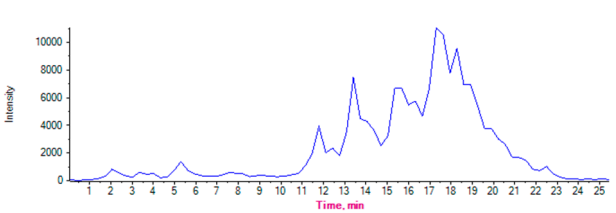

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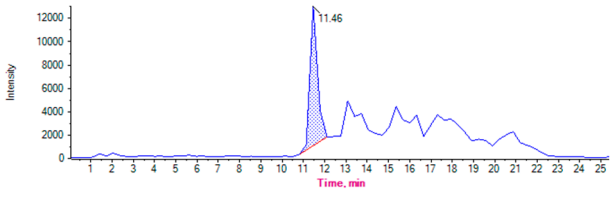

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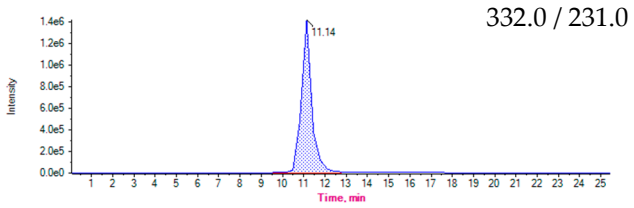

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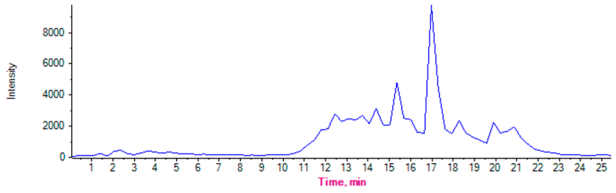

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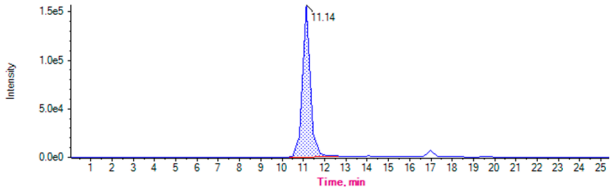

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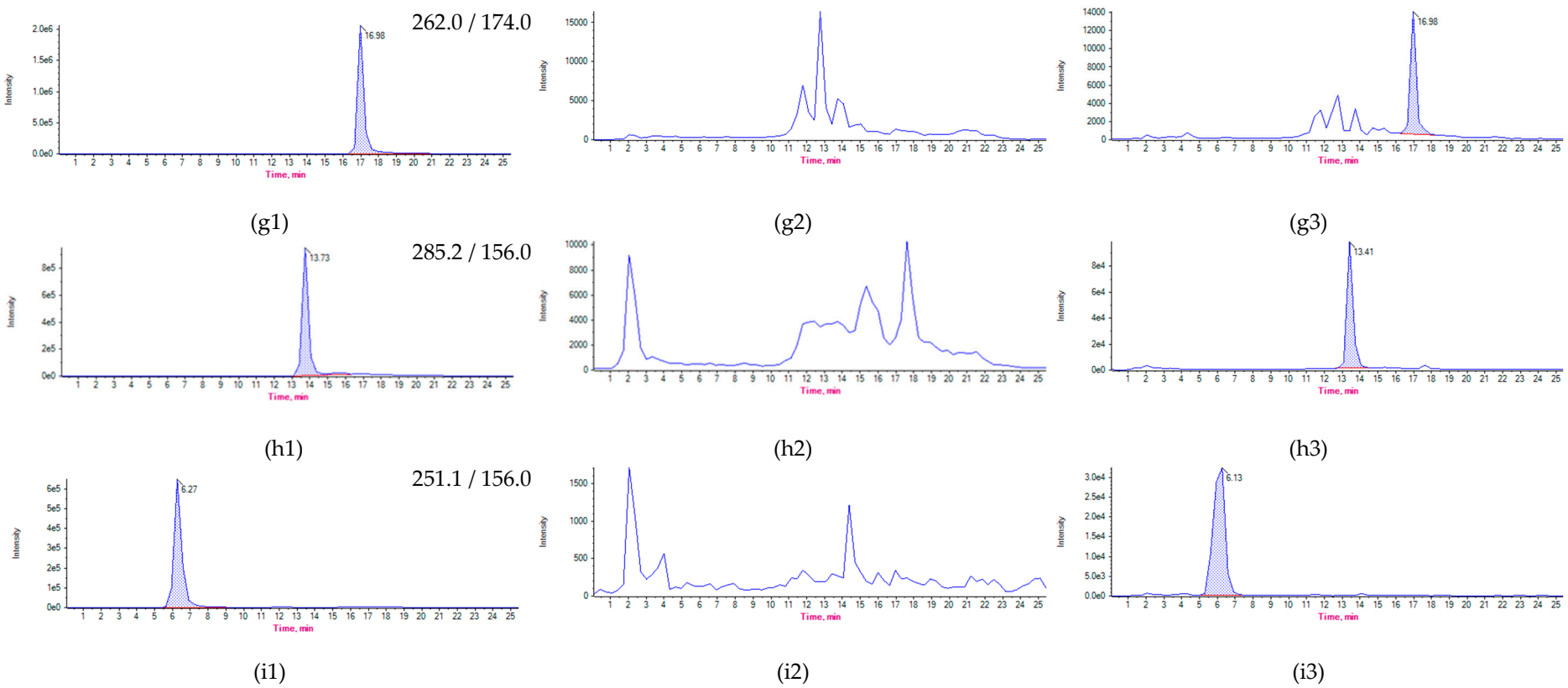

Figure S1. Representative chromatograms from (1) a pure standard solution injection (50 ng mL^{-1}), (2) a blank poultry litter sample and (3) poultry litter sample spiked to 25 ug k^{-1} of (a) tetracycline, (b) 4-epimer-tetracycline, (c) Oxitetracycline, (d) 4-epimer-oxitetracycline, (e) Enrofloxacin, (f) Ciprofloxacin, (g) Flumequine, (h) Sulfachloropyridazine, (i) Sulfadiazine.

Table S1. Scans per peak.

Analytes	Time (msec)
Tetracycline / 4-epimer-Tetracycline	100.0
Oxytetracycline / 4-epimer-Oxytetracycline	100.0
Enrofloxacin	280.0
Ciprofloxacin	280.0
Flumequine	280.0
Sulfachloropyridazine	280.0
Sulfadiazine	200.0
Sulfamethazine-phenyl-13C6	280.0
Enrofloxacin-D5	280.0
Tetracycline-D6	250.0

Table S2. Mass of precursor and fragment ions, and specific mass spectrometry conditions.

Analytes	Precursor Ion (m/z)	Fragment Ions (m/z)	DP [3] (V)	EP [4] (V)	CE [5] (V)	CXP [6] (V)
Tetracycline / 4-epimer-Tetracycline	445.0	410.0[1]	77.0	10.0	28.0	24.0
		392.0	69.0	10.0	38.0	23.0
Oxytetracycline / 4-epimer-Oxytetracycline	461.0	426.0[1]	72.0	10.0	28.0	25.0
		381.0	73.0	10.0	36.0	22.0
Enrofloxacin	361.0	343.0	50.0	13.0	23.0	8.0
		317.0[1]	50.0	13.0	28.0	20.0
Ciprofloxacin	332.0	231.0[1]	56.0	4.5	47.0	4.0
		314.0	56.0	4.5	28.0	4.0
Flumequine	262.0	244.0	20.0	5.0	10.0	13.0
		174.0[1]	20.0	5.0	50.0	7.0
Sulfachloropyridazine	285.2	156.0[1]	61.0	10.0	21.0	12.0
		108.1	61.0	10.0	31.0	8.0
Sulfadiazine	251.1	156.0[1]	46.0	4.0	19.0	4.0
		108.0				
		108.0	46.0	4.0	31.0	4.0
Sulfamethazine-phenyl-13C6[2]	285.0	124.1	71.0	10.0	31.0	12.0
Enrofloxacin-D5[2]	365.0	321.0	50.0	7.0	23.0	8.0
Tetracycline-D6[2]	451.0	160.0	34.0	10.0	25.0	30.0

[1] Quantifier ion; [2] Internal Standard; [3]Declustering potential, [4]Entrance potential, [5]Collision energy, [6]Collision cell exit potential.

Table S3. Instrumental Limit of detection and Limit of quantification.

Analyte	ILOD [1] (μg kg^{-1})	ILOQ [2] (μg kg^{-1})
Enrofloxacin	2.0	6.7
Ciprofloxacin	3.1	10.4
Flumequine	2.9	9.7
Sulfachloropyridazine	3.0	10.1
Sulfadiazine	2.8	9.5
Tetracycline	3.4	11.4
4-epi-Tetracycline	2.9	9.7
Oxytetracycline	2.7	8.9
4-epi-Oxytetracycline	2.3	7.8
Chlortetracycline	3.3	11.1
4-epi-Chlortetracycline	2.9	9.6

[1]Instrumental Limit of detection; [2]Instrumental Limit of quantification.