Application Brief

Food Testing

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Analysis of Olaquindox in Fodder Using SPE with LC/MS/MS

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Abstract

This study developed and validated a method for the quantitative analysis of olaquindox in fodder using SPE followed by LC/MS/MS analysis. Methanol/water (5/95) is used for extraction of olaquindox from fodder followed by SPE cleanup with Agilent Bond Elut PPL. The method provides a reliable solution, with good recoveries and reproducibility, for monitoring olaquindox in fodder.

Experimental

Instrument method

The samples were run on an Agilent 1260 Infinity II LC system coupled to an Agilent 6470 triple quadrupole LC/MS system. The MS was equipped with an Agilent Jet Stream electrospray ion source. Agilent MassHunter workstation software was used for data acquisition and analysis.

HPLC conditions

Parameter	Value
Column	Agilent InfinityLab Poroshell 120 SB-C18, 100 x 2.1 mm, 2.7 µm (p/n 685775-902)
Column Temperature	35 °C
Injection Volume	5 µL
Mobile Phase	A) Water (0.1% formic acid) B) ACN (0.1% formic acid)
Gradient	Time (min) %A %B 0 95 5 0.5 95 5 1.0 85 15 5.0 55 45 5.5 2 98 7.5 2 98 7.6 95 5

MS conditions

Parameter	Value
Gas Temperature	300 °C
Gas Flow	7 L/min
Nebulizer	35 psi
Sheath Gas Heater	350 °C
Sheath Gas Flow	11 L/min
Capillary	3,000 V (POS)
Data Acquisition	MRM as in Table 1.

Sample extraction

The procedure is shown in Figure 1.

Table 1. Target analytes MRM conditions.

Analyte	Precursor Ion (*m/z*)	Product ion (*m/z*)	Fragmentor (V)	CE (V)
Olaquindox	264	212	110	20
		143	110	30

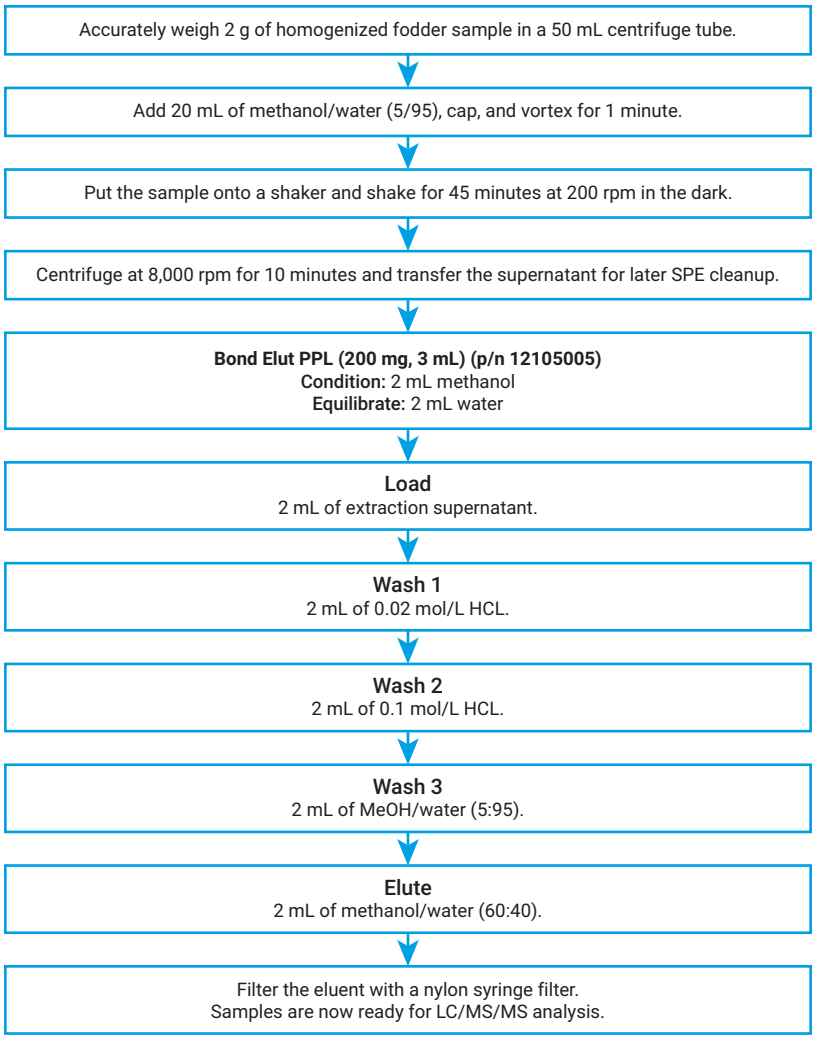

Figure 1. Sample preparation workflow chart.

Results and discussion

The method delivers good linearity for olaquindox in the range of 25 to 1,000 ng/mL (Table 2 and Figures 2 through 4). The recoveries are between 78 and 92 % with RSD ≤3.6 in the spiking levels of 200, 400, and 1,000 µg/kg. The limit of quantitation and limit of detection are 200 µg/kg and 60 µg/kg, respectively.

Table 2. Method recovery and RSDs.

Fodder	Spiking Level (µg/kg)	Recovery (%)	RSD% (n = 3)
Chicken Fodder	200	86.9	1.0
		87.7	
		85.9	
	400	81.4	3.2
		86.2	
		81.8	
	1,000	78.2	1.8
		81.2	
		81.3	
Pig Fodder	200	89.0	1.3
		90.4	
		91.3	
	400	81.8	3.6
		87.5	
		83.2	

Conclusion

A method with Bond Elut PPL cleanup, a polar-modified styrene-divinylbenzene polymer SPE product, coupled with HPLC/MS/MS, delivers excellent recoveries and reproducibility for the analysis of olaquindox. The method was successful in the analysis of both chicken fodder and pig fodder. Bond Elut PPL with large particle size allows ease-of-flow for viscous or particulate-rich water samples. The high surface area and strong hydrophobicity ensure reproducible extractions with high recoveries upon elution.

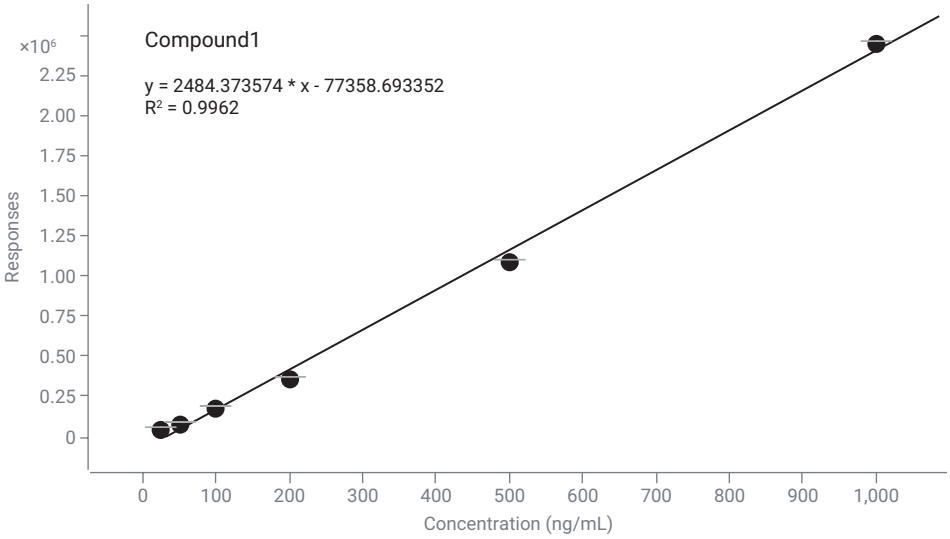

Figure 2. Calibration curves of olaquindox.

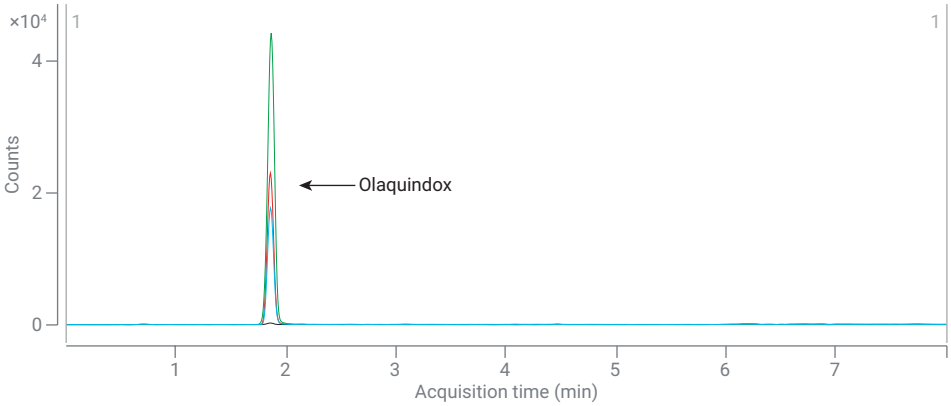

Figure 3. MRM chromatograms of olaquindox for neat standard at 200 ppb (green), postspiked in chicken fodder (red) and prespiked in chicken fodder at 200 µg/kg (blue), matrix blank (black).

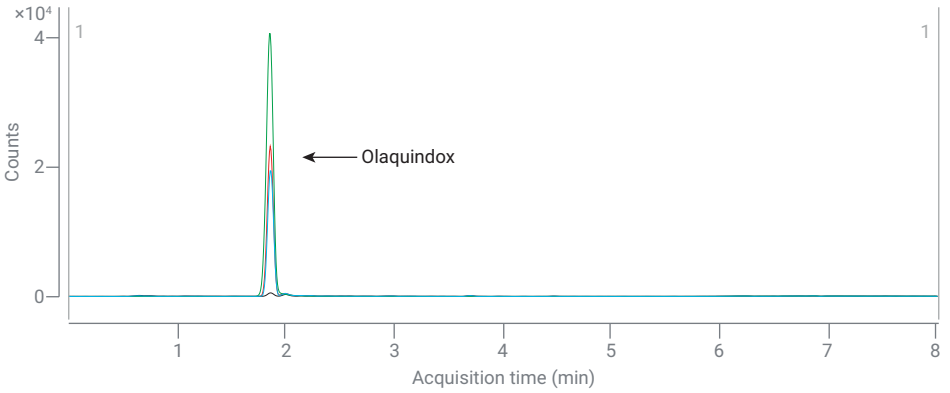

Figure 4. MRM chromatograms of olaquindox for neat standard at 200 ppb (green), postspiked in pig fodder (red) and prespiked in pig fodder at 200 µg/kg (blue), matrix blank (black).

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