

测试报告

样品信息			
样品名称	猪饲料	编号	Z20221230-010
样品重量	/	剂型	/
收样日期	2022/12/30	测试期间	2022/12/30-2023/01/03
样品描述	/		
测试需求			
测试成分	异丙硝唑、甲硝唑、替硝唑、地美硝唑、罗硝唑、唑乙醇、呋喃它酮、唑烯酮		
参考标准			
参考标准	农业部 2349 号公告-6-2015	标样	有
仪器信息			
测试仪器	高效液相色谱串联质谱仪	仪器型号	Xevo TQ-S micro

● 色谱条件:

色谱柱:	月旭 Xtimate® UHPLC C18 2.1×100mm, 1.8μm		
流动相:	时间	流动相 A: (水) %	流动相 B: (甲醇) %
	0.00	95	5
	4.00	5	95
	5.00	5	95
	5.10	95	5
	7.00	95	5
柱温:	40 °C		
流速:	0.3 mL/min		
进样量:	2 μL		
注意事项	本次测试采用基质曲线, 建议采用内标法。		



● 流动相的配置:

流动相 A: 水

流动相 B : 色谱级甲醇。

● 质谱条件:

离子源: ESI

检测方式: MRM

干燥气: 氮气, 500°C, 流速: 1000L/Hr

碰撞气: 氩气

离子喷雾电压: 1.5kV

名称	母离子 (m/z)	子离子 (m/z)	Cone (V)	Collision (V)	Polarity
异丙硝唑	170.1	96.0	22	20	Positive
		109.0		24	
		124.1		16	
甲硝唑	172.1	56.1	28	20	Positive
		82.0		24	
		128.1		12	
替硝唑	248.0	82.1	22	30	Positive
		93.0		20	
		121.0		16	
地美硝唑	142.1	81.0	20	26	Positive
		95.9		18	
罗硝唑	201.0	55.0	8	22	Positive
		140.0		10	
喹乙醇	264.1	102.0	2	50	Positive
		143.0		36	
		221.0		12	
呋喃它酮	325.0	100.0	22	26	Positive
		252.1		14	



		281.1		10	
喹烯酮	307.0	77.0	30	66	Positive
		103.0		44	
		131.1		28	

● 样品溶液的配置:

样品溶液: 称取试样 2.00 g 于 50 mL 离心管中, 准确加入 20.0 mL 提取液 (甲醇: 乙腈: 水 = 3:3:4), 水浴超声提取 10 min, 振荡 15 min, 8000 r/min, 离心 5 min, 取 1.00 mL 上清液于 40°C 氮吹至近干。残余物用磷酸二氢钠溶液 (0.1 mol/L pH=3.0) 5.0 mL 溶解, 超声 10 min, 备用。

BRP 固相萃取柱 (60 mg, 3 mL), 用 3 mL 甲醇, 3 mL 水活化。

备用液过柱, 弃去。

用 3 mL 水, 3 mL 甲醇水溶液 (10%) 淋洗, 弃去, 抽干。

3 mL 乙酸乙酯, 洗脱并收集。

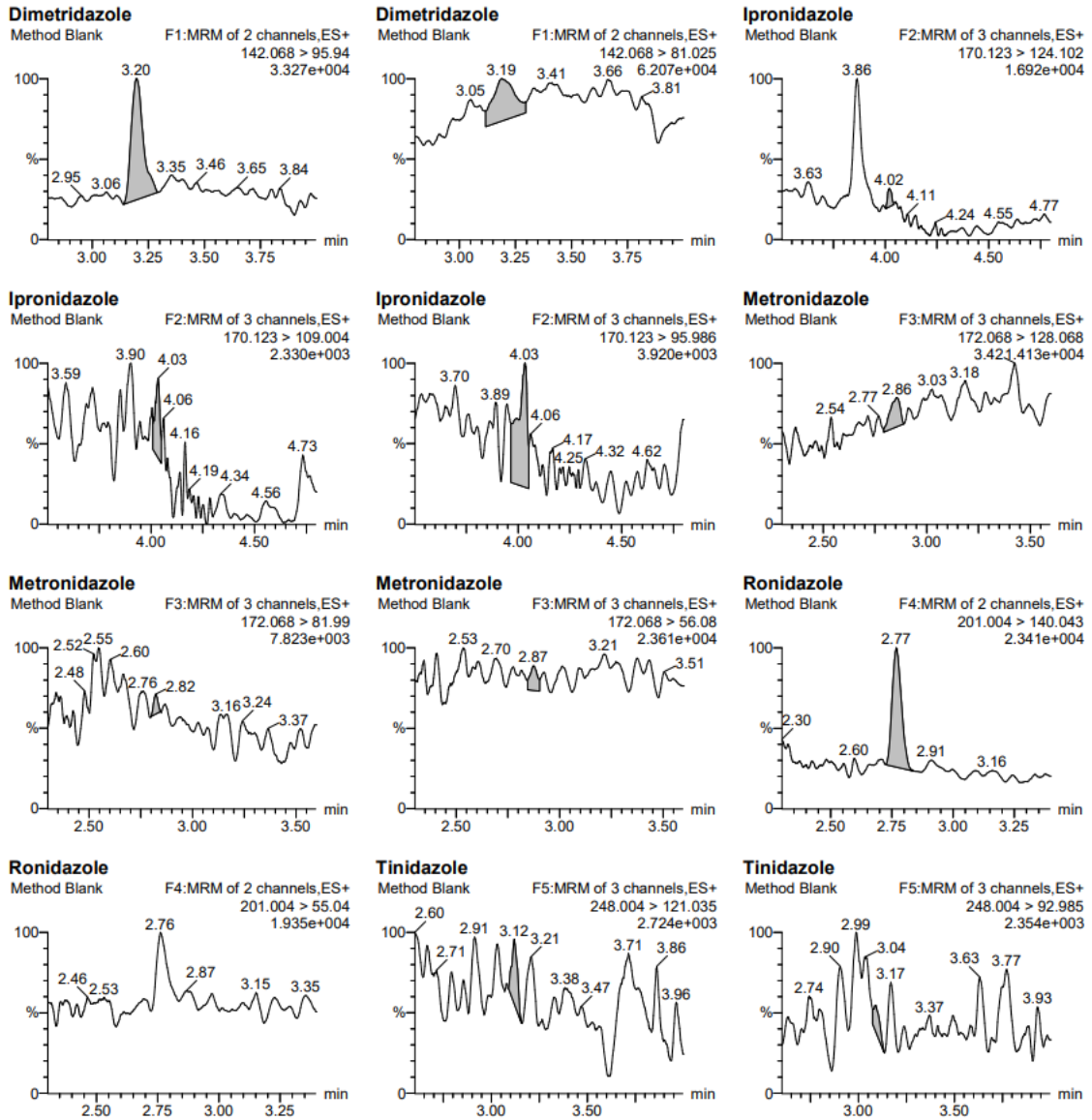
洗脱液于 40°C 氮吹至干, 准确加入 1.00 mL 甲醇水溶液 (5%) 溶解残余物, 涡旋混匀, 超声 10 min, 过 0.22 μm 滤膜, 上机分析。

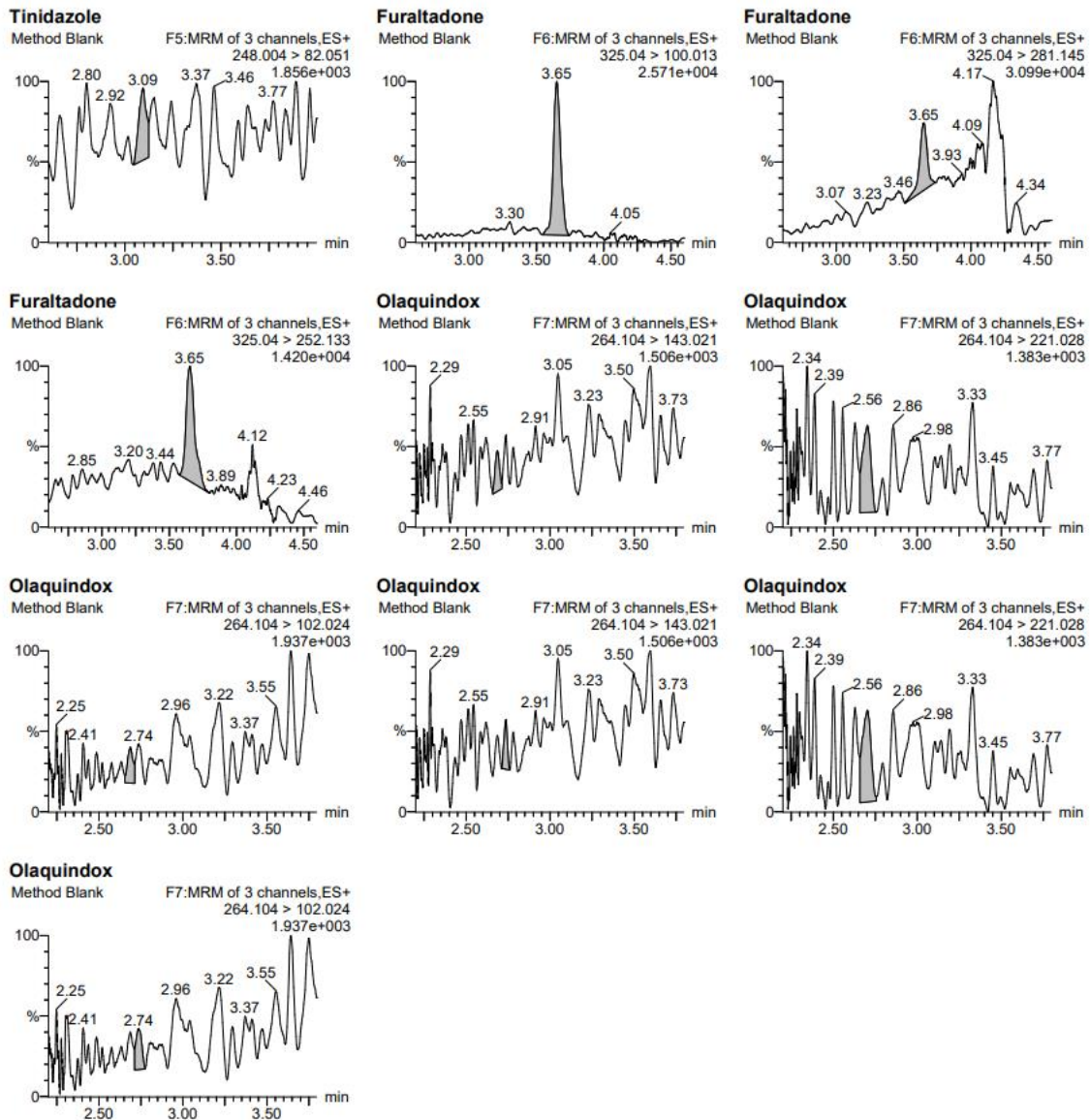
注: 固相萃取柱的流速为每秒 1 滴。

谱图和数据

(1) 空白





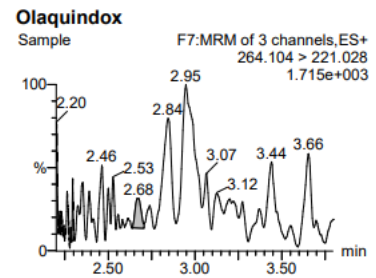
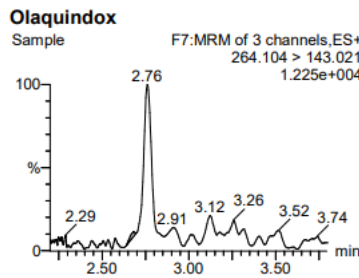
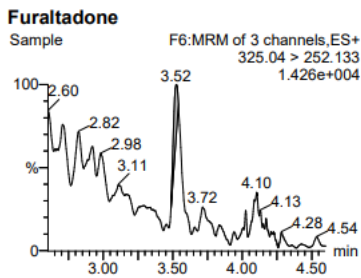
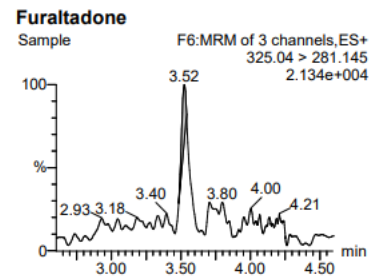
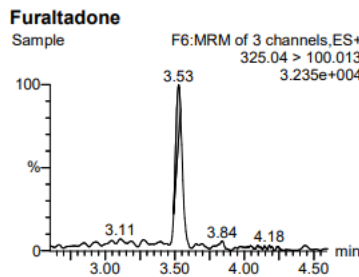
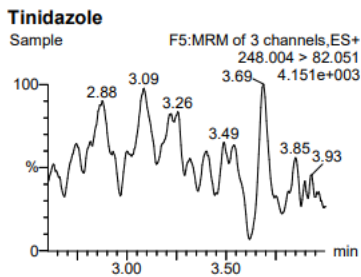
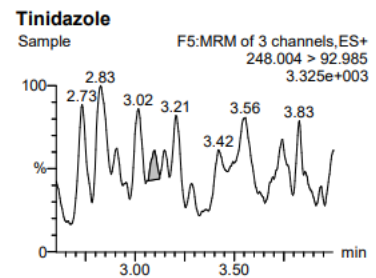
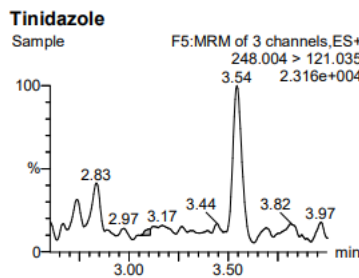
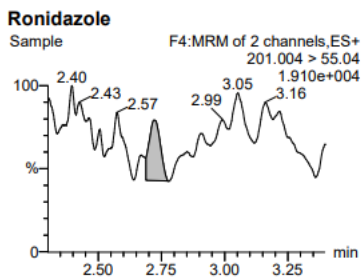
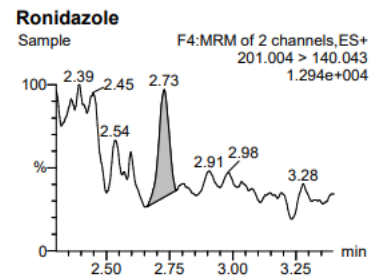
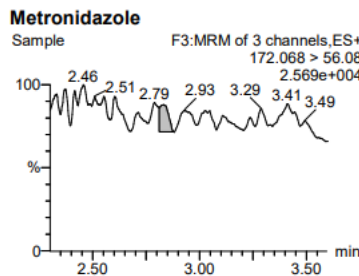
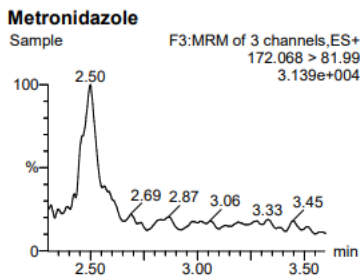
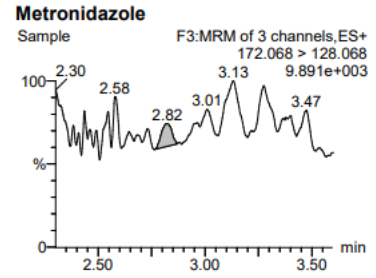
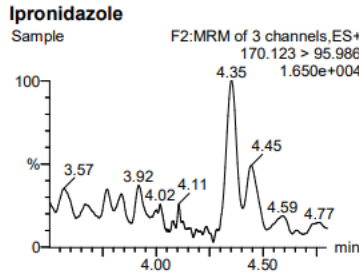
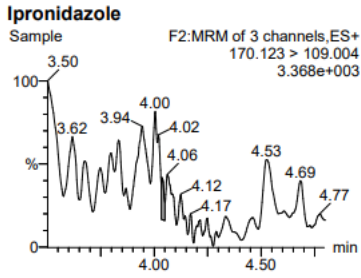
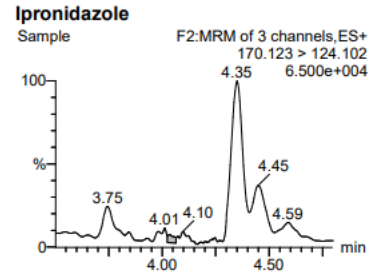
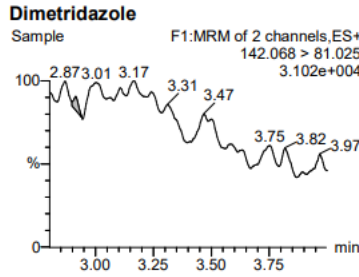
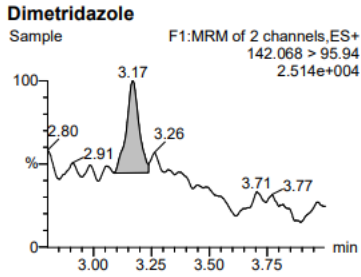


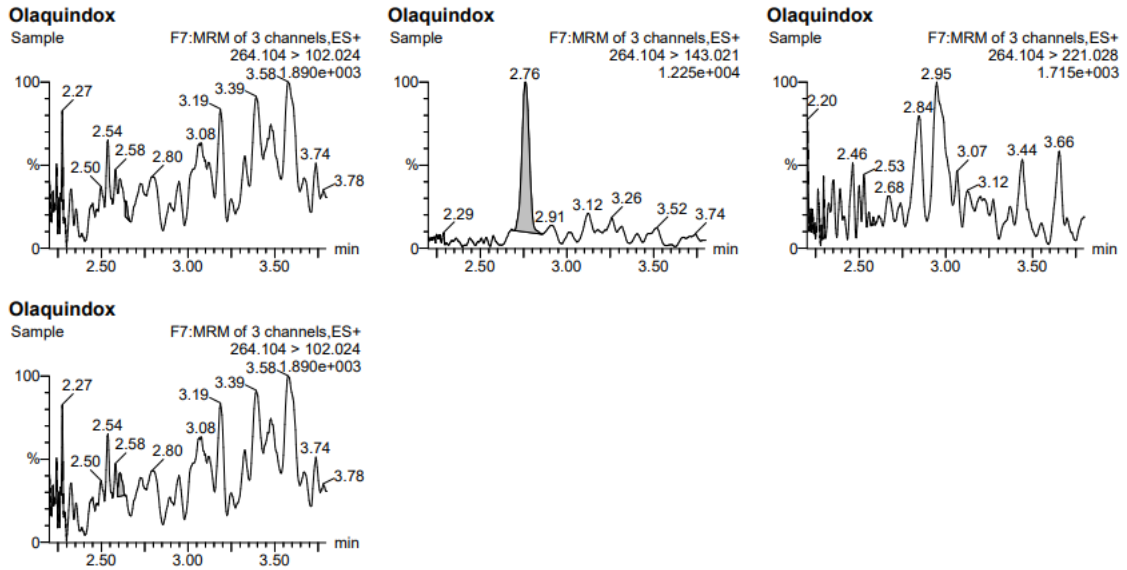
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1° Ratio (A...	2° Ratio (A...
Method Blank	Dimetridazole	142.068 > 95.94	3.20	1540.138	bb	0.043		0.843	
Method Blank	lpronidazole	170.123 > 124.102	4.02	41.580	bb	0.004		1.419	0.255
Method Blank	Metronidazole	172.068 > 128.068	2.86	145.466	db	0.006		6.643	0.983
Method Blank	Ronidazole	201.004 > 140.043	2.77	756.769	bb	0.082			

Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1° Ratio (A...	2° Ratio (A...
Method Blank	Tinidazole	248.004 > 121.035	3.12	35.926	bb	0.004		2.448	0.943
Method Blank	Furaltadone	325.04 > 100.013	3.65	1513.497	MM	0.129		1.784	2.037
Method Blank	Olaquinox	264.104 > 143.021	2.69	11.189	dd	0.017		0.261	0.695
Method Blank	Olaquinox	264.104 > 143.021	2.74	10.663	bd	0.016		0.228	0.541

(2) 样品溶液



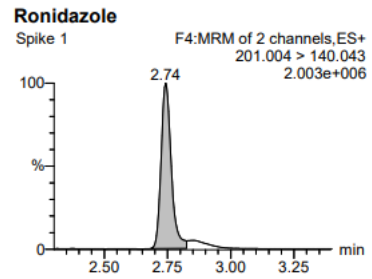
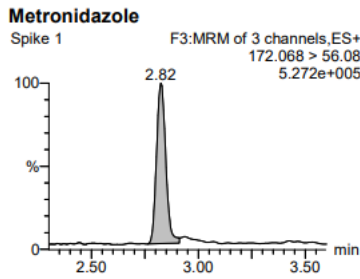
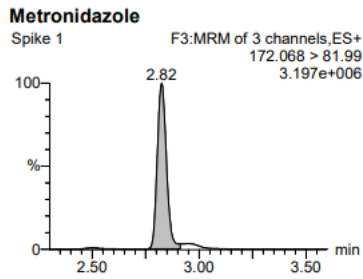
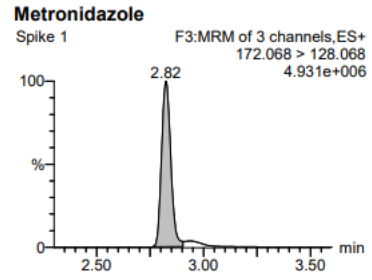
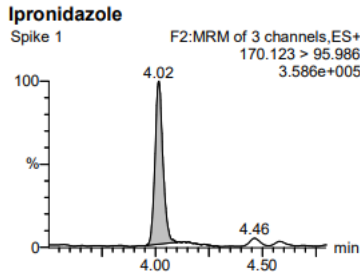
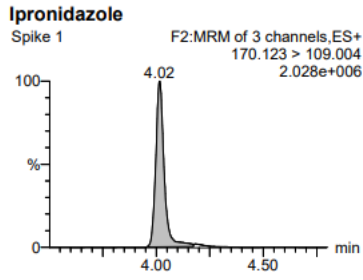
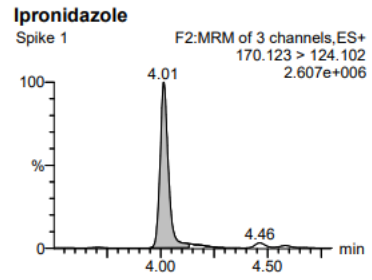
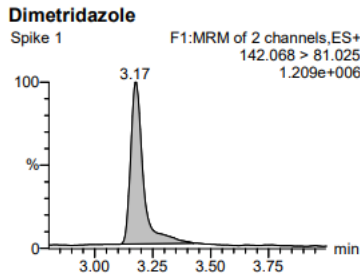
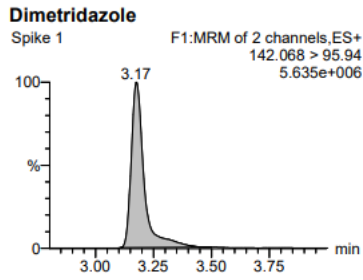


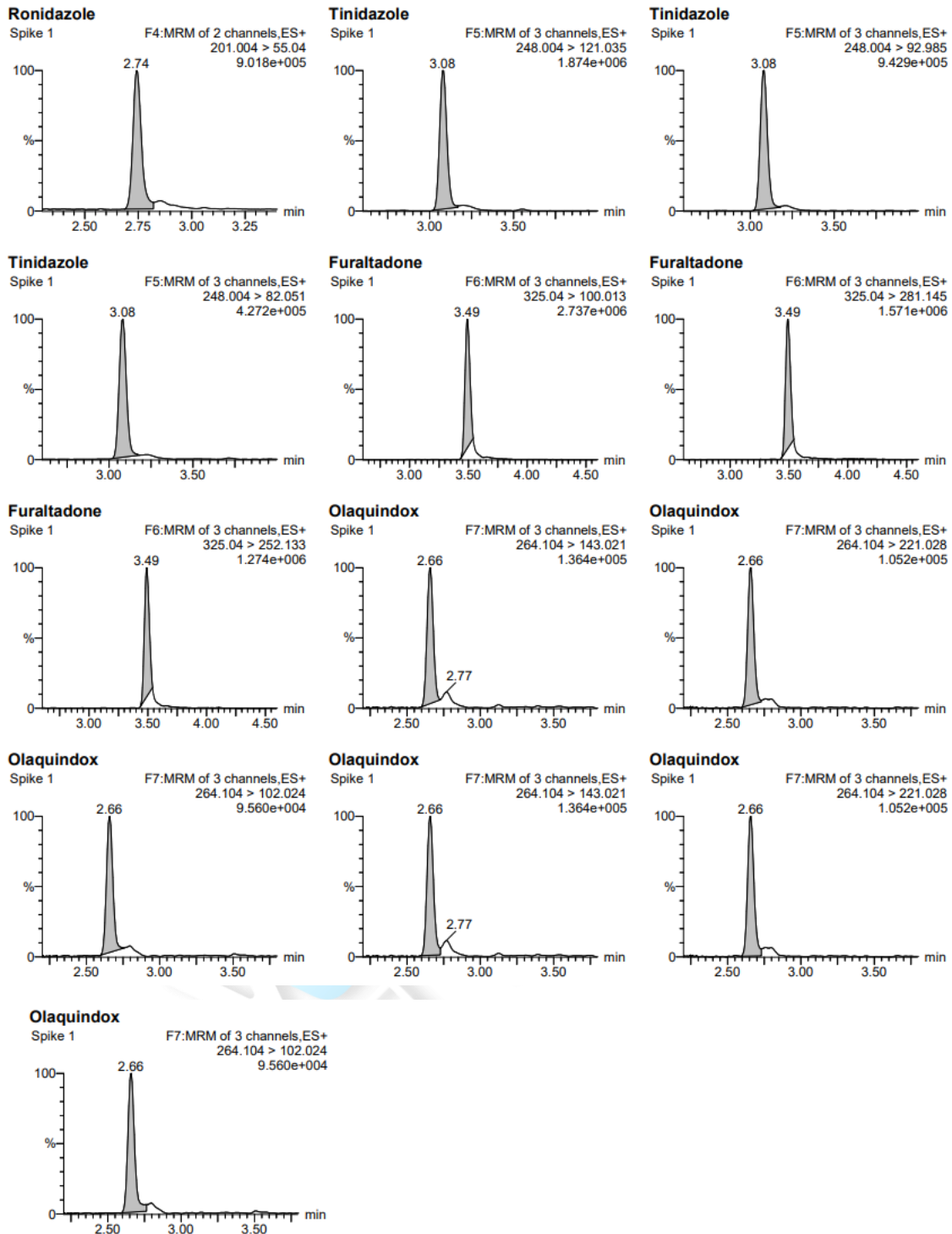


Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1° Ratio (A...	2° Ratio (A...
Sample	Dimetridazole	142.068 > 95.94	3.17	874.680	bd	0.024		14.389	
Sample	Iprnidazole	170.123 > 124.102	4.04	103.740	dd	0.009		10.015	46.251
Sample	Metronidazole	172.068 > 128.068	2.82	76.093	bb	0.003			0.402
Sample	Ronidazole	201.004 > 140.043	2.73	394.263	bb	0.043		1.103	
Sample	Tinidazole	248.004 > 121.035	3.11	24.531	bd	0.003		1.257	
Sample	Furaltadone	325.04 > 100.013	3.53	338.236	bb	0.029		1.455	2.168
Sample	Olaquinox	264.104 > 143.021	2.68	12.076	bb	0.019		1.017	4.702
Sample	Olaquinox	264.104 > 143.021	2.76	543.901	bb	0.797		661.680	87.797

(3) 10 ng/mL 基质曲线



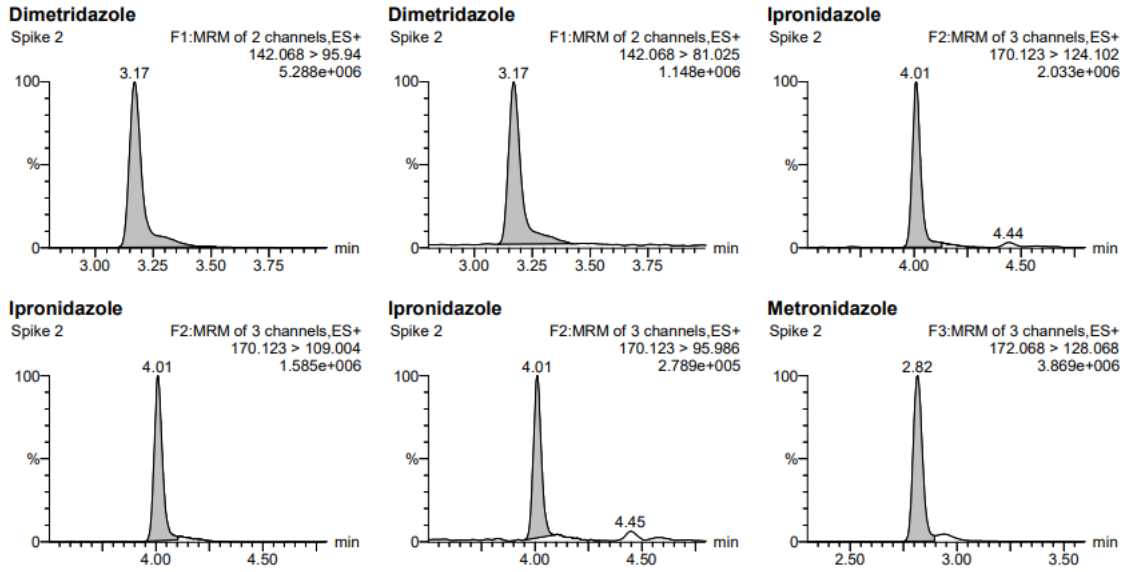




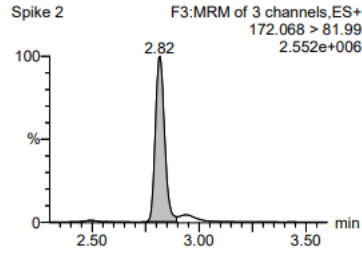
Sample Text	Name	Trace	RT	Area Primar...	ng/mL	%Dev	1 ^o Ratio (A...	2 ^o Ratio (A...
Spike 1	Dimetridazole	142.068 > 95.94	3.17	357740.750	bb	10.000	0.0	4.847
Spike 1	Iprnidazole	170.123 > 124.102	4.01	114511.297	bd	10.000	0.0	1.255
Spike 1	Metronidazole	172.068 > 128.068	2.82	242863.781	bd	10.000	0.0	1.545
Spike 1	Ronidazole	201.004 > 140.043	2.74	91781.281	bd	10.000	0.0	2.251
Spike 1	Tinidazole	248.004 > 121.035	3.08	91599.531	bd	10.000	0.0	1.999
Spike 1	Furaltadone	325.04 > 100.013	3.49	117394.969	bb	10.000	0.0	1.738
Spike 1	Olaquinox	264.104 > 143.021	2.66	6409.544	bd	10.000	0.0	1.296
Spike 1	Olaquinox	264.104 > 143.021	2.66	6825.010	bd	10.000	0.0	1.305



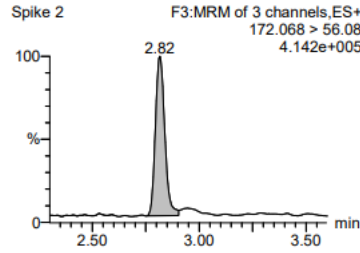
(4) 样品加标, 10 ng/mL。



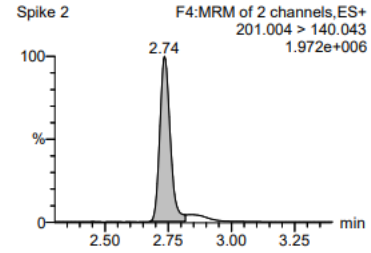
Metronidazole



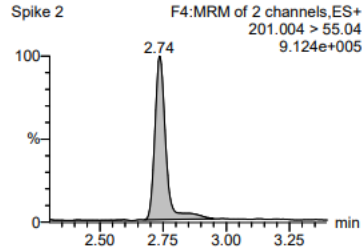
Metronidazole



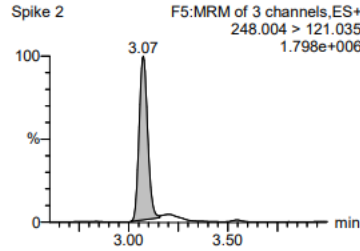
Ronidazole



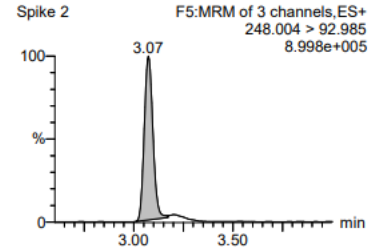
Ronidazole



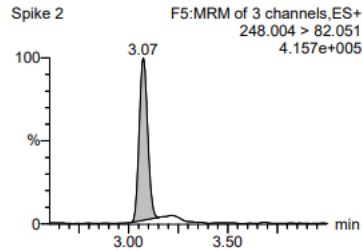
Tinidazole



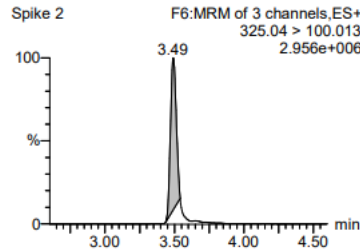
Tinidazole



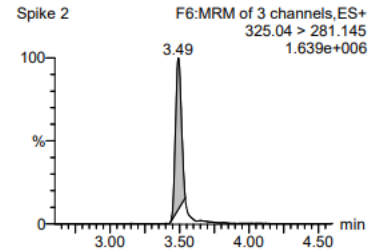
Tinidazole



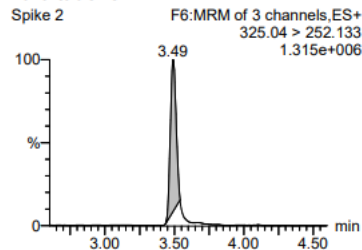
Furaltadone



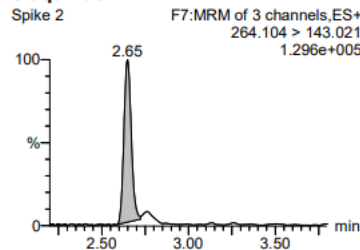
Furaltadone



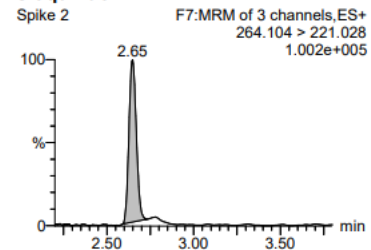
Furaltadone

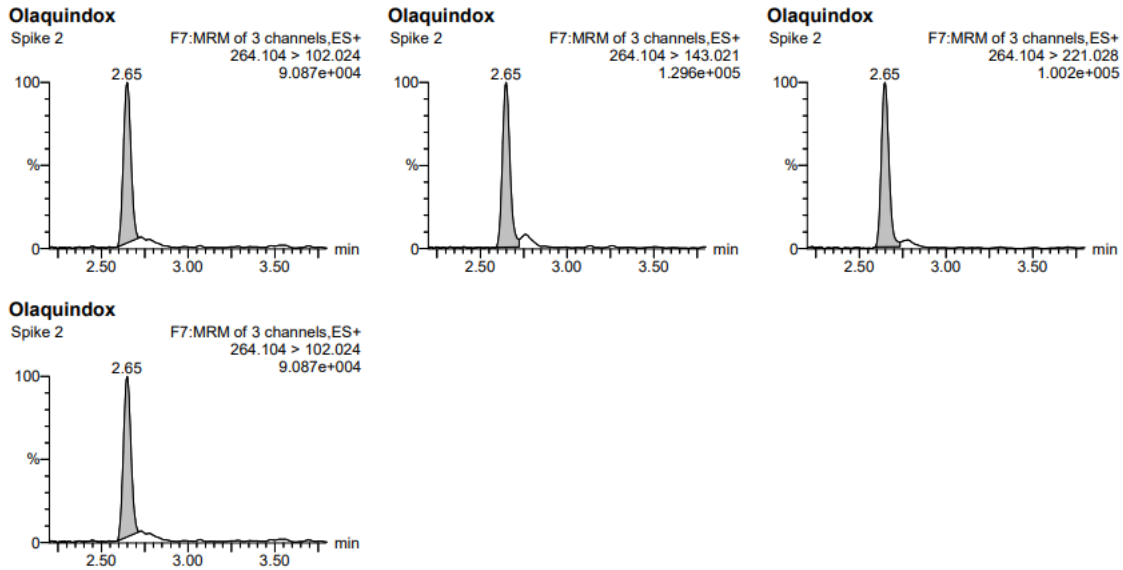


Olaquinox



Olaquinox





Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1 ^o Ratio (A...	2 ^o Ratio (A...
Spike 2	Dimetridazole	142.068 > 95.94	3.17	344439.781	bb	9.628		4.766	
Spike 2	Ipronidazole	170.123 > 124.102	4.01	92180.609	bd	8.050		1.329	7.977
Spike 2	Metronidazole	172.068 > 128.068	2.82	193014.313	bd	7.947		1.545	9.600
Spike 2	Ronidazole	201.004 > 140.043	2.74	92872.828	bd	10.119		2.079	
Spike 2	Tinidazole	248.004 > 121.035	3.07	87557.313	bd	9.559		1.970	4.419
Spike 2	Furaltadone	325.04 > 100.013	3.49	126379.523	bb	10.765		1.778	2.260
Spike 2	Olaquinox	264.104 > 143.021	2.65	6172.080	bd	9.630		1.301	1.413
Spike 2	Olaquinox	264.104 > 143.021	2.65	6441.228	bd	9.438		1.300	1.475

● 结论:

使用月旭 Welchrom® BRP (60 mg, 3 mL) 检测猪饲料中异丙硝唑、甲硝唑、替硝唑、地美硝唑、罗硝唑、喹乙醇、呋喃它酮、喹烯酮回收率为 80.5%、79.5%、95.6%、96.3%、101.2%、96.3%、107.8%、94.4%，样品回收率满足要求。

日期: 2023/01/04

