

## 测试报告

样品信息			
样品名称	火锅底料	编号	Z20220314-006
样品重量	/	剂型	/
收样日期	2022/6/17	测试期间	2022/6/17-2022/6/19
样品描述	/		
测试需求			
测试成分	氧氟沙星、洛美沙星、培氟沙星、氟罗沙星、双氟沙星、司帕沙星、环丙沙星、恩诺沙星		
参考标准			
参考标准	BJS 201909	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 Ultimate® UHPLC LP-C18 (2.1×50mm,1.8μm)		
流动相:	时间	流动相 A% (5mmol/L 乙酸铵 0.1%甲酸水溶液)	流动相 B%乙腈
	0.0	90	10
	3.0	90	10
	8.0	70	30
	10.0	70	30
	10.5	5	95
	13.0	5	95



流动相:	13.1	90	10
	15.0	90	10
柱温:	40°C		
流速:	0.25 mL/min		
进样量:	2 $\mu$ L		
注意事项:	本方法为基质曲线。		

● 流动相的配置:

流动相 A :称取 0.1925 g 乙酸铵加水溶解,移取 0.5 mL 甲酸并加水至 500 mL 混匀,经 0.22  $\mu$ m 滤膜抽滤,即;

流动相 B : 用色谱级乙腈,经 0.22  $\mu$ m 滤膜抽滤,即得。

● 质谱条件:

离子源: ESI

检测方式: MRM

干燥气: 氮气, 450°C, 流速: 1100L/Hr

碰撞气: 氩气

离子喷雾电压: 1.0 kV

名称	母离子(m/z)	子离子(m/z)	Cone (V)	Collision (V)	Polarity
环丙沙星	332.1	314.2*	30	22	Positive
		288.1		18	
双氟沙星	400.1	356.2*	30	20	Positive
		299.1	44	30	
恩诺沙星	360.1	245.1	38	24	Positive
		316.2		19	
		342.2*		23	
氟罗沙星	370.1	269.2	30	26	Positive
		326.3*		18	



洛美沙星	352.1	265.1*	40	23	Positive
		308.2		17	
氧氟沙星	362.1	261.1	25	27	Positive
		318.1*		18	
培氟沙星	334.2	290.1	26	17	Positive
		316.1*		20	
司帕沙星	393.2	292.3	30	25	Positive
		349.0*	20	19	

\*表示定量离子

### ● 样品溶液的配制:

称取样品 2.5 g 置于 50 mL 离心管中, 加入 10 mL 0.1mol/L EDTA-McIlvaine 缓冲溶液, 涡旋 1 min, 超声提取 15 min, 8000 r/min 离心 5 min, 将上清液移入另一个 50 mL 离心管中, 重复提取 2 次, 每次 5 mL, 合并上清液, 定容至 25 mL。

固相萃取柱: BRP 500 mg/6 mL

活化: 6 mL 甲醇, 6 mL 水, 弃去;

上样: 移取上清液 12.5 mL, 弃去;

淋洗: 3 mL 5% 甲醇水溶液, 弃去, 抽干;

洗脱: 6 mL 甲醇, 3 mL 氨化甲醇, 收集。

洗脱液 40℃ 氮吹至近干, 用初始流动相定容至 2 mL, 混匀, 过 0.22 μm 滤膜, 上机。

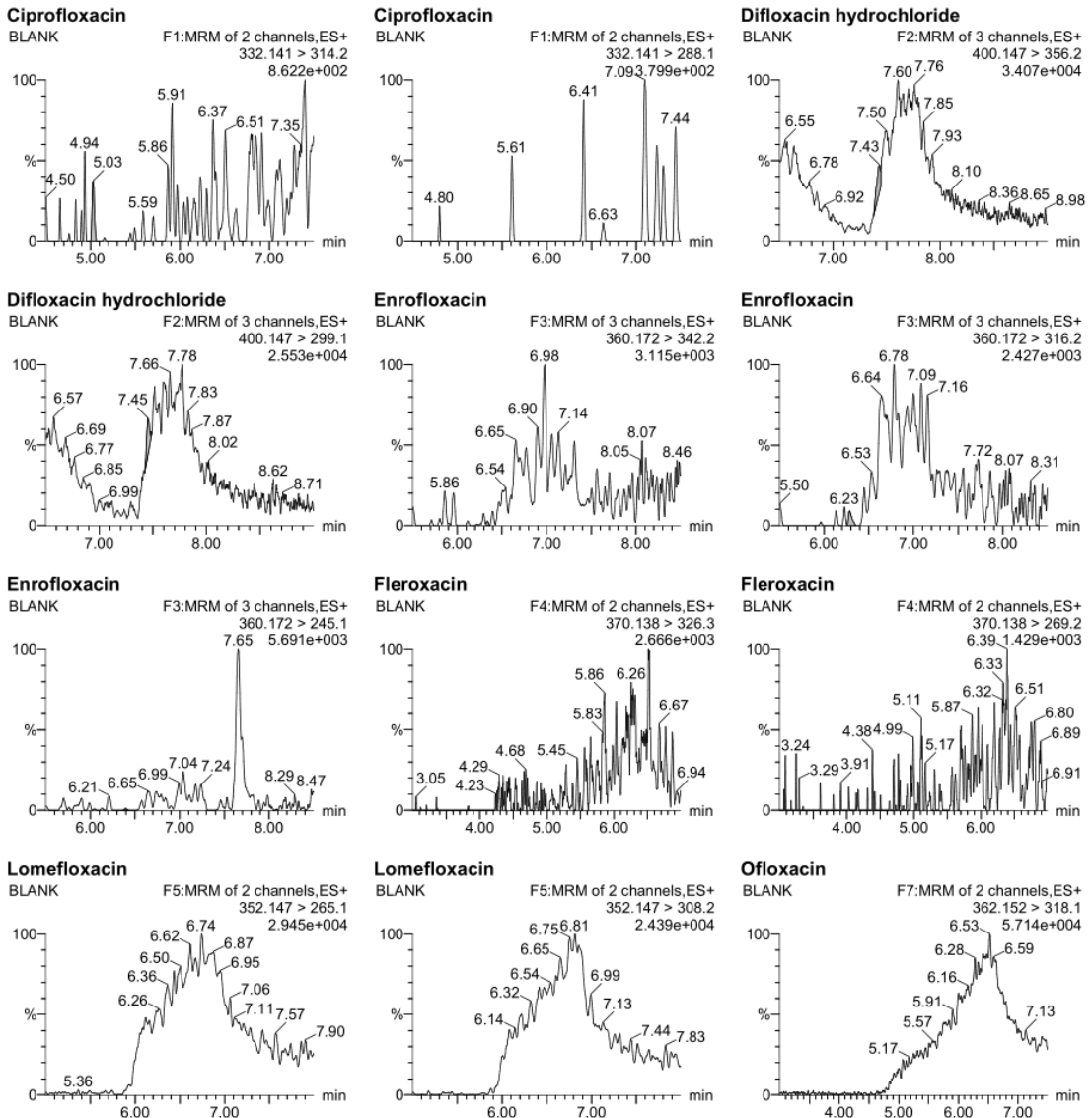
注:

1、固相萃取柱流速为每秒 1 滴。



● 谱图和数据

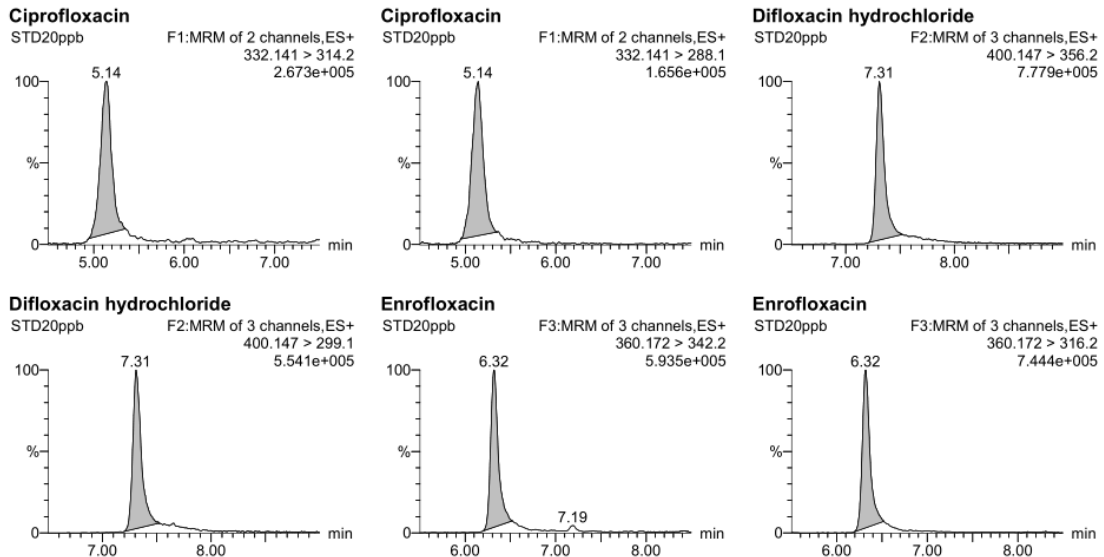
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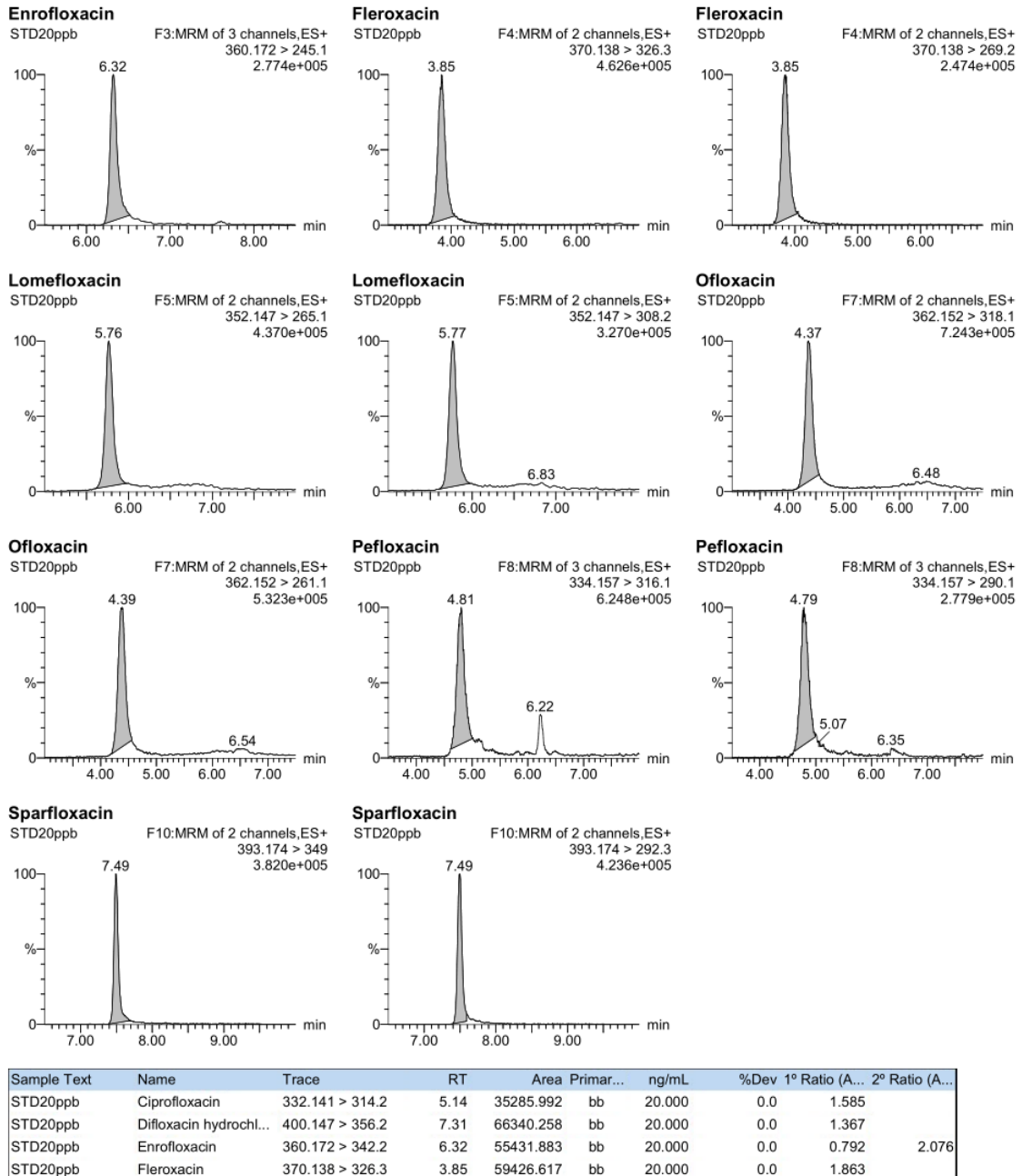




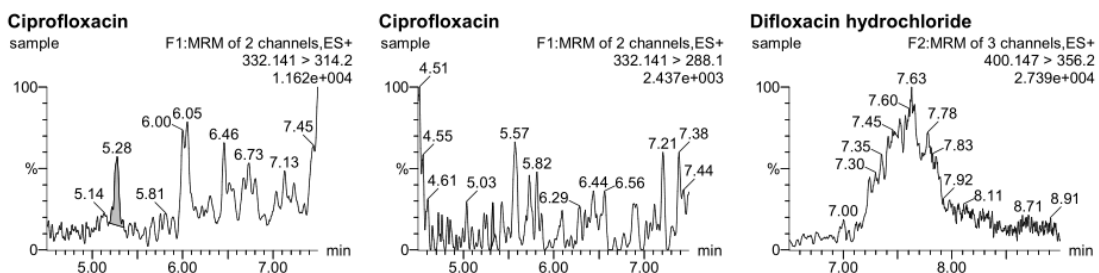
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1° Ratio (A...	2° Ratio (A...
BLANK	Ciprofloxacin	332.141 > 314.2	5.03	9.302	bb	0.005			
BLANK	Difloxacin hydrochl...	400.147 > 356.2	7.43	246.872	bd	0.074		1.408	
BLANK	Enrofloxacin	360.172 > 342.2	6.34	1.953	db	0.001		0.205	1.344
BLANK	Fleroxacin	370.138 > 326.3	3.82	0.738	bb	0.000			
BLANK	Lomefloxacin	352.147 > 265.1							
BLANK	Ofloxacin	362.152 > 318.1	4.37	6.566	db	0.001		0.430	
BLANK	Pefloxacin	334.157 > 316.1							
BLANK	Sparfloxacin	393.174 > 349	7.59	67.541	bd	0.050		0.689	

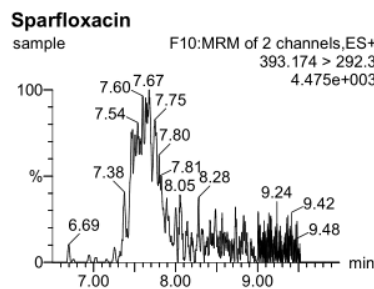
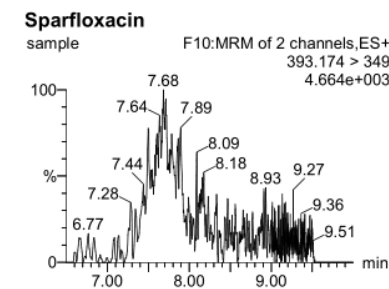
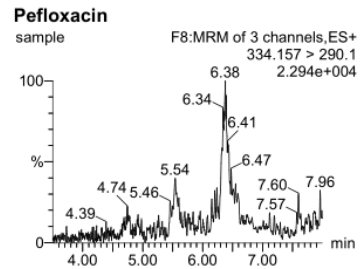
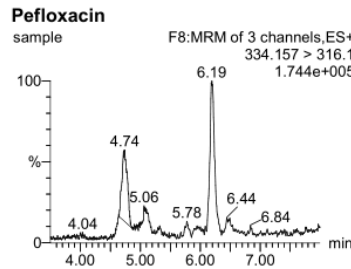
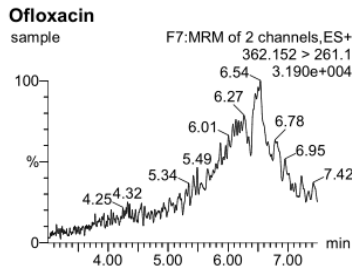
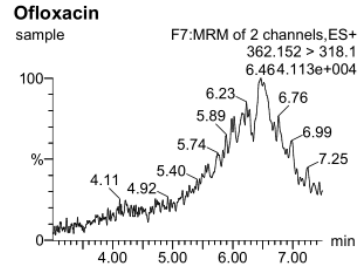
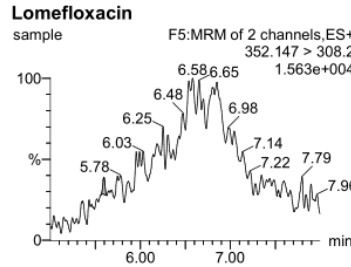
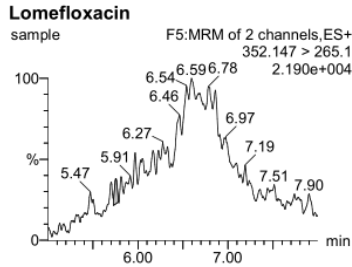
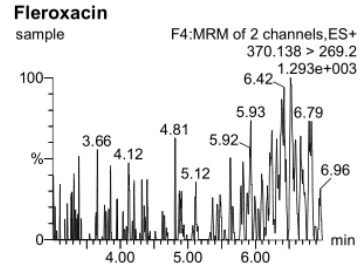
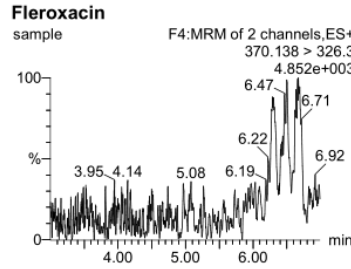
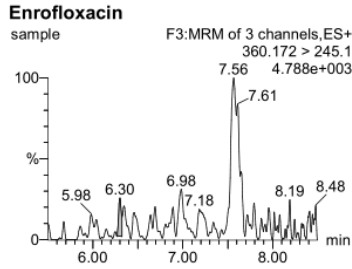
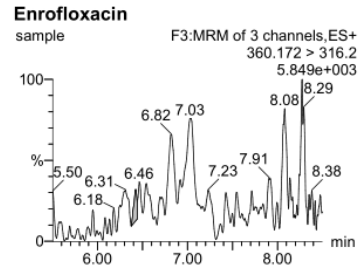
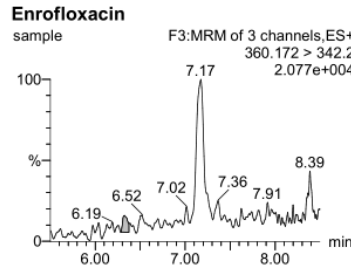
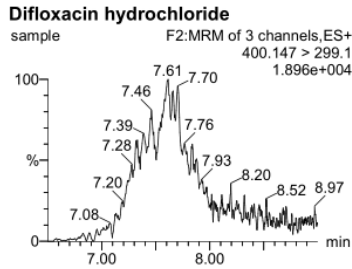
(2) 喹诺酮类化合物标准品溶液浓度为 20 ng/mL。





(3) 火锅底料本底。

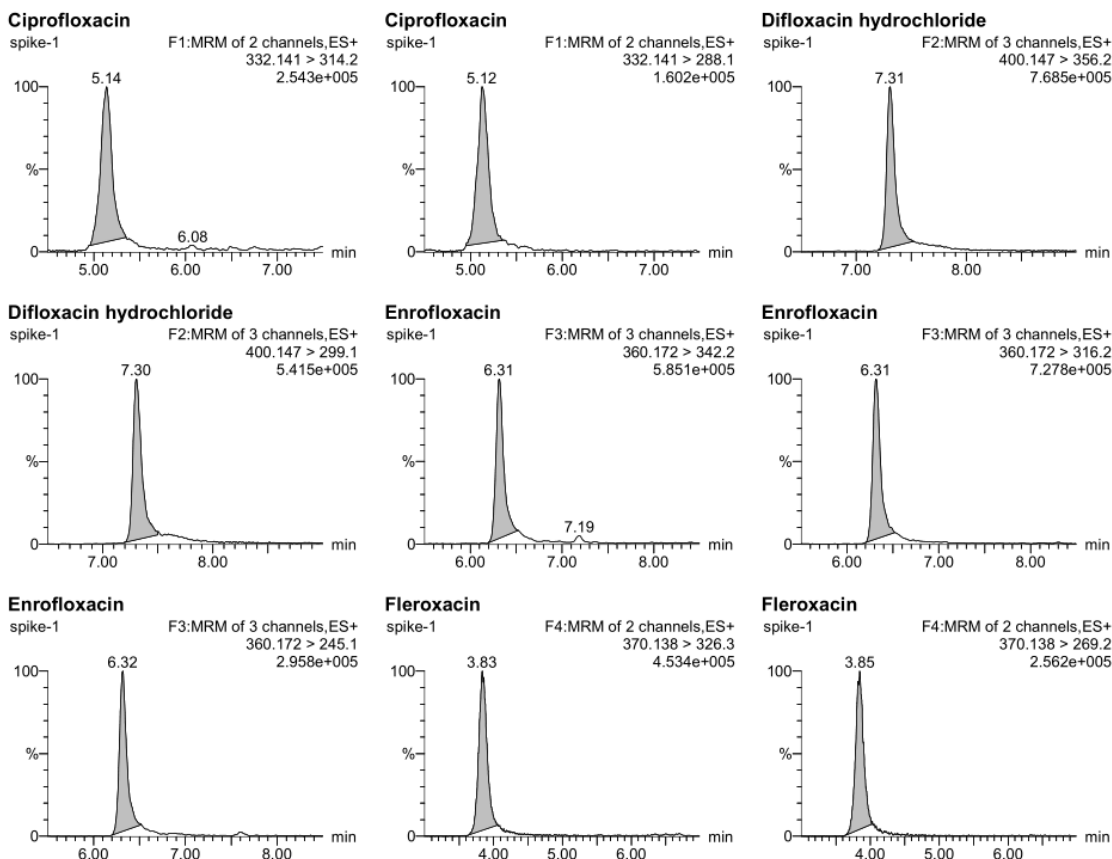


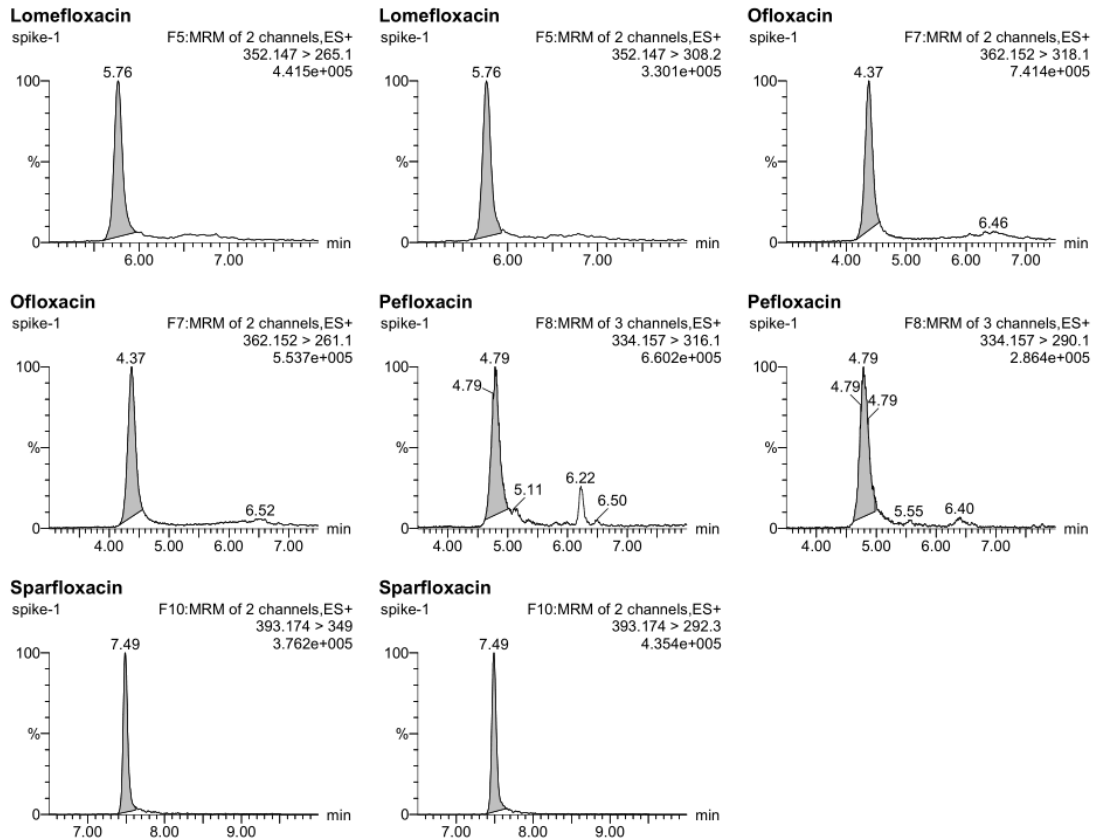


Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1° Ratio (A...	2° Ratio (A...
sample	Ciprofloxacin	332.141 > 314.2	5.28	295.142	bb	0.167		23.796	
sample	Difloxacin hydrochl...	400.147 > 356.2							
sample	Enrofloxacin	360.172 > 342.2	6.32	133.696	bd	0.048		2.991	3.623
sample	Fleroxacin	370.138 > 326.3	3.94	4.151	bd	0.001		1.186	
sample	Lomefloxacin	352.147 > 265.1	5.75	116.574	db	0.052		2.729	
sample	Ofloxacin	362.152 > 318.1	4.35	65.292	dd	0.013		1.765	
sample	Pefloxacin	334.157 > 316.1			MM-			0.000	
sample	Sparfloxacin	393.174 > 349							



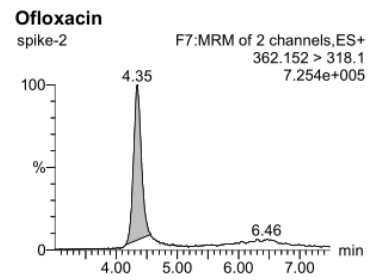
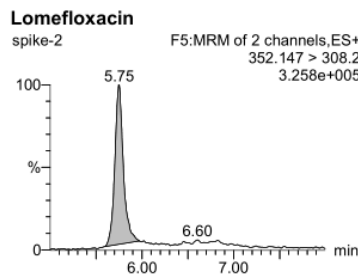
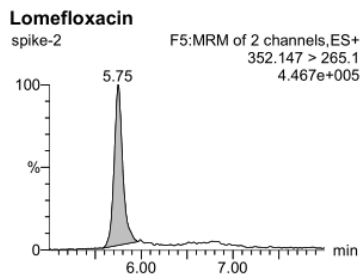
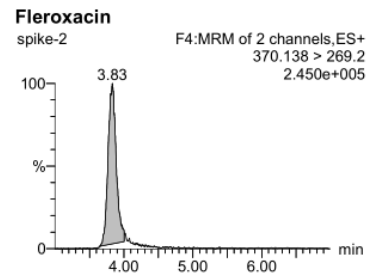
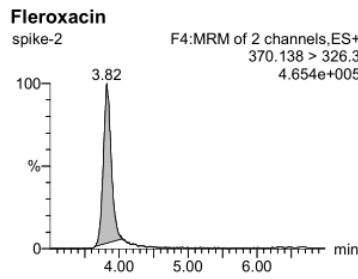
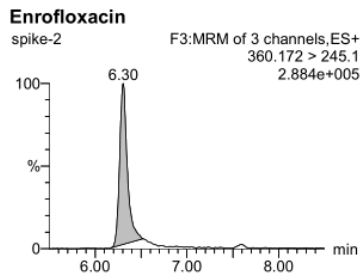
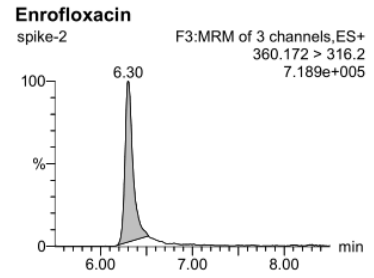
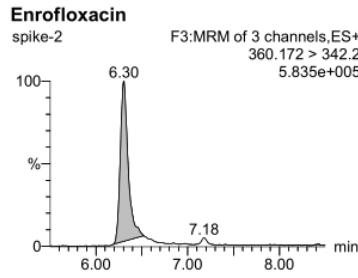
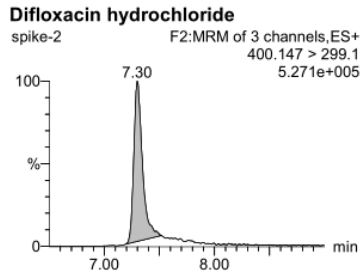
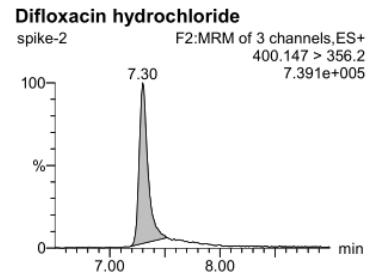
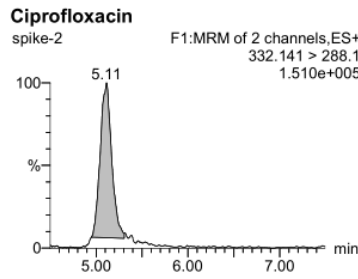
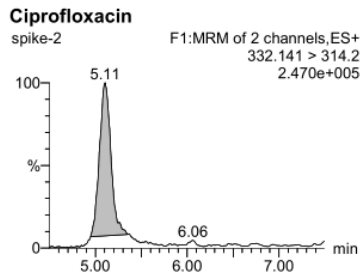
(4) 加标回收: 喹诺酮类标准品加标浓度 20 ng/mL。

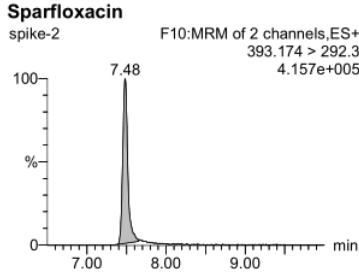
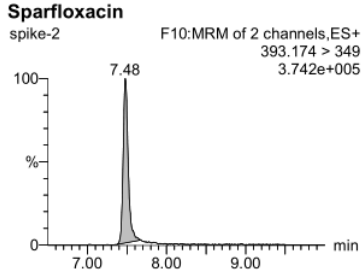
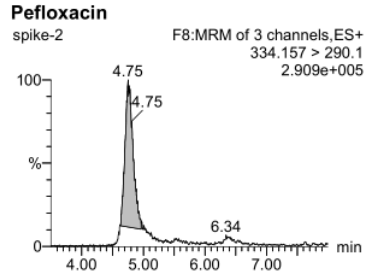
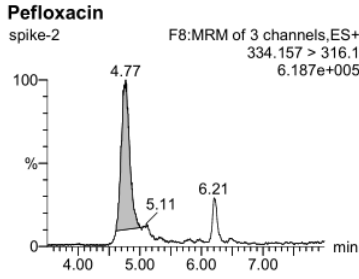
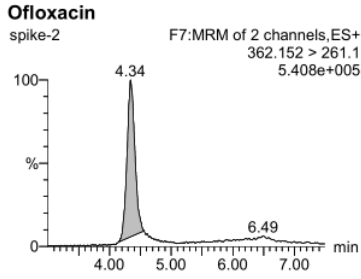




Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1 <sup>o</sup> Ratio (A...	2 <sup>o</sup> Ratio (A...
spike-1	Ciprofloxacin	332.141 > 314.2	5.14	34423.355	bb	19.511		1.645	
spike-1	Difloxacin hydrochl...	400.147 > 356.2	7.31	66187.313	bb	19.954		1.370	
spike-1	Enrofloxacin	360.172 > 342.2	6.31	54623.719	bb	19.708		0.787	2.056
spike-1	Fleroxacin	370.138 > 326.3	3.83	59572.797	bb	20.049		1.854	
spike-1	Lomefloxacin	352.147 > 265.1	5.76	46520.563	bb	20.612		1.351	
spike-1	Ofloxacin	362.152 > 318.1	4.37	97387.680	bb	19.850		1.322	
spike-1	Pefloxacin	334.157 > 316.1	4.79	90000.602	bb	20.658		2.127	
spike-1	Sparfloxacin	393.174 > 349	7.49	25950.359	bd	19.110		0.865	







Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1° Ratio (A...	2° Ratio (A...
spike-2	Ciprofloxacin	332.141 > 314.2	5.11	32807.313	bb	18.595		1.617	
spike-2	Difloxacin hydrochl...	400.147 > 356.2	7.30	65467.813	bb	19.737		1.417	
spike-2	Enrofloxacin	360.172 > 342.2	6.30	55890.293	bb	20.165		0.790	2.053
spike-2	Fleroxacin	370.138 > 326.3	3.82	58599.246	bb	19.722		1.817	
spike-2	Lomefloxacin	352.147 > 265.1	5.75	46813.859	bb	20.742		1.388	
spike-2	Ofloxacin	362.152 > 318.1	4.35	99896.484	bb	20.362		1.344	
spike-2	Pefloxacin	334.157 > 316.1	4.77	85238.969	bb	19.565		2.237	
spike-2	Sparfloxacin	393.174 > 349	7.48	26585.631	bb	19.578		0.893	



● 结论:

样品加标回收

化合物	实际浓度 ng/mL	理论浓度 ng/mL	回收率, %
环丙沙星	19.511	20	97.6
	18.595		93.0
双氟沙星	19.954	20	99.8
	19.737		98.7
恩诺沙星	19.708	20	95.5
	20.165		100.8
氟罗沙星	20.049	20	100.2
	19.722		98.6
洛美沙星	20.612	20	103.1
	20.742		103.7
氧氟沙星	19.850	20	99.2
	20.362		101.8
培氟沙星	20.658	20	103.3
	19.565		97.8
司帕沙星	19.110	20	95.6
	19.578		97.9

使用 Welchrom®BRP 500 mg/6 mL Part#00522-20006 检测火锅底料中喹诺酮类化合物, 回收率良好且稳定, 。

日期: 2022/06/20

