

测试报告

样品信息			
样品名称	虾肉	编 号	Z20221102-003
样品重量	/	剂 型	/
收样日期	2022/11/02	测试期间	2022/11/02-2022/11/03
样品描述	/		
测试需求			
测试成分	磺胺醋酰、磺胺嘧啶、磺胺吡啶、磺胺噻唑、磺胺甲噁啉、磺胺二甲嘧啶、磺胺甲氧嘧啶、磺胺甲二唑、磺胺对甲氧嘧啶、磺胺间甲氧嘧啶、磺胺氯达嗪、磺胺多辛、磺胺苯酰、磺胺地索辛、磺胺苯吡唑		
参考标准			
参考标准	GB/T 21316-2007	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® ODS-3 (4.6×150mm,3µm)		
流动相:	时间	流动相 A% (0.1%甲酸水溶液)	流动相 B%乙腈
	0.0	99.5	0.5
	5.0	90.0	10.0
	25.0	50.0	50.0
	30.0	60.0	40.0
	30.5	99.5	0.5
	40.0	99.5	0.5



柱温:	20°C
流速:	0.8 mL/min
进样量:	1µL
注意事项:	本方法为基质曲线。测试中峰型前沿是由于UHPLC上使用HPLC色谱柱的原因,若使用UHPLC色谱柱,峰型良好。因为ODS-3没有UHPLC色谱柱因此建议使用HPLC串联质谱,其他填料色谱柱分离度弱于ODS-3。

● 流动相的配置:

流动相 A : 移取 0.5 mL 甲酸并加水至 500 mL 混匀,经 0.22 µm 滤膜抽滤,即;

流动相 B : 用色谱级乙腈,经 0.22 µm 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: 1000L/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
磺胺醋酰	215.0	91.9	20	22	Positive
		108.0		18	
		156.0		10	
磺胺嘧啶	251.0	91.9	4	26	Positive
		107.9		22	
		156.0		14	
磺胺吡啶	250.0	92.0	48	24	Positive
		156.1		16	
		184.1		16	
磺胺噻唑	256.0	108.0	32	22	Positive
		156.0		12	
磺胺甲噻唑	265.1	91.9	14	30	Positive



		156.0		16	
		172.0		14	
磺胺二甲嘧啶	279.1	91.9	30	30	Positive
		156.0		20	
		186.0		16	
磺胺甲氧嘧啶	281.1	91.9	14	30	Positive
		108.1		24	
		156.0		16	
磺胺甲二唑	271.0	108.0	32	24	Positive
		271.0		12	
磺胺对甲氧嘧啶	281.1	108.0	16	26	Positive
		156.0		16	
磺胺间甲氧嘧啶	281.1	92.0	6	30	Positive
		108.0		26	
		156.0		16	
磺胺氯达嗪	285.0	91.9	40	24	Positive
		108.0		22	
		156.0		14	
磺胺多辛	311.1	92.0	42	30	Positive
		108.0		28	
		156.0		18	
磺胺苯酰	277.1	108.0	22	22	Positive
		156.0		10	
磺胺地索辛	311.0	92.0	44	30	Positive
		108.0		28	
		156.0		20	
磺胺苯吡唑	315.1	108.0	52	28	Positive
		158.2		28	
		222.1		18	

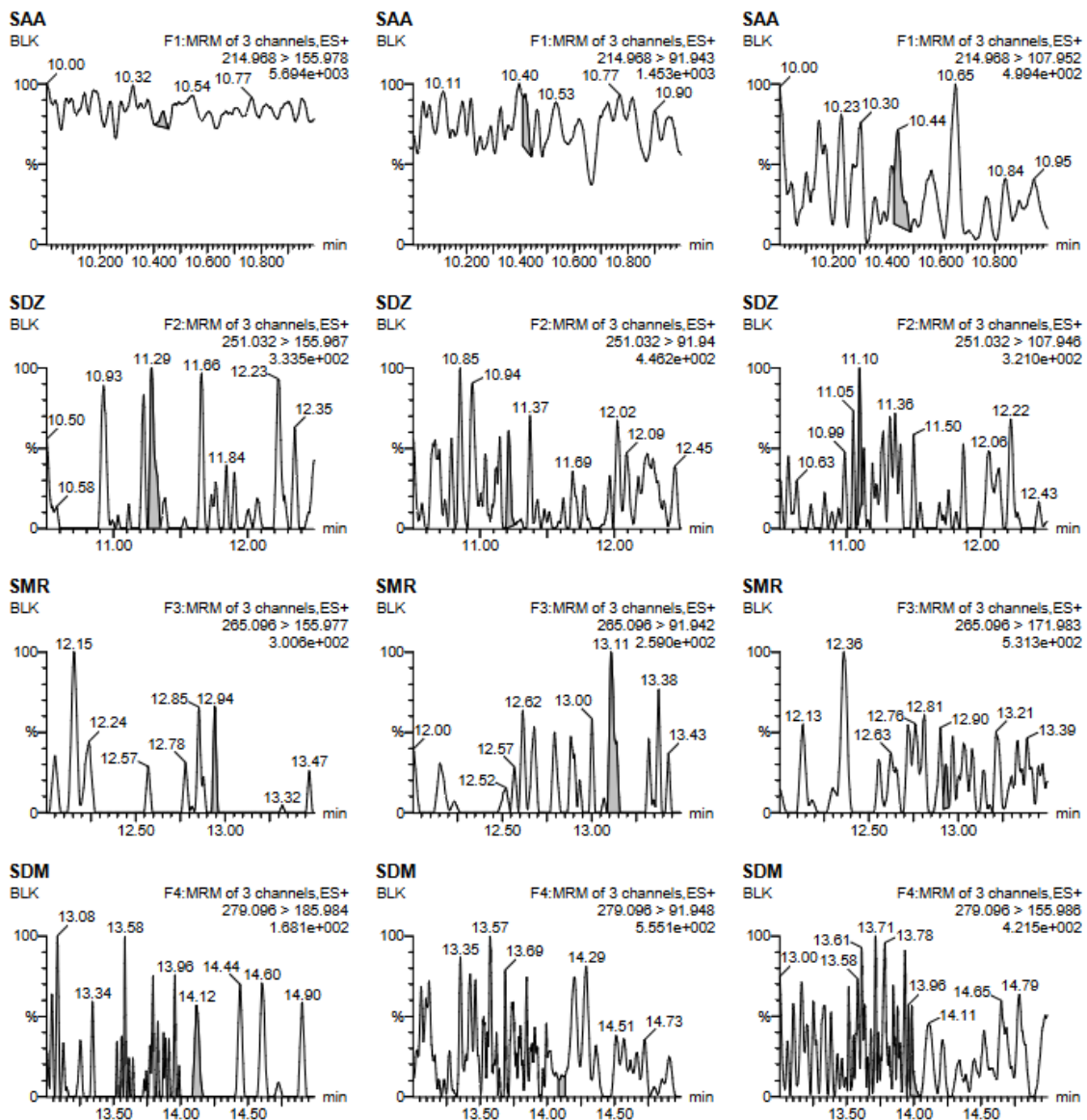


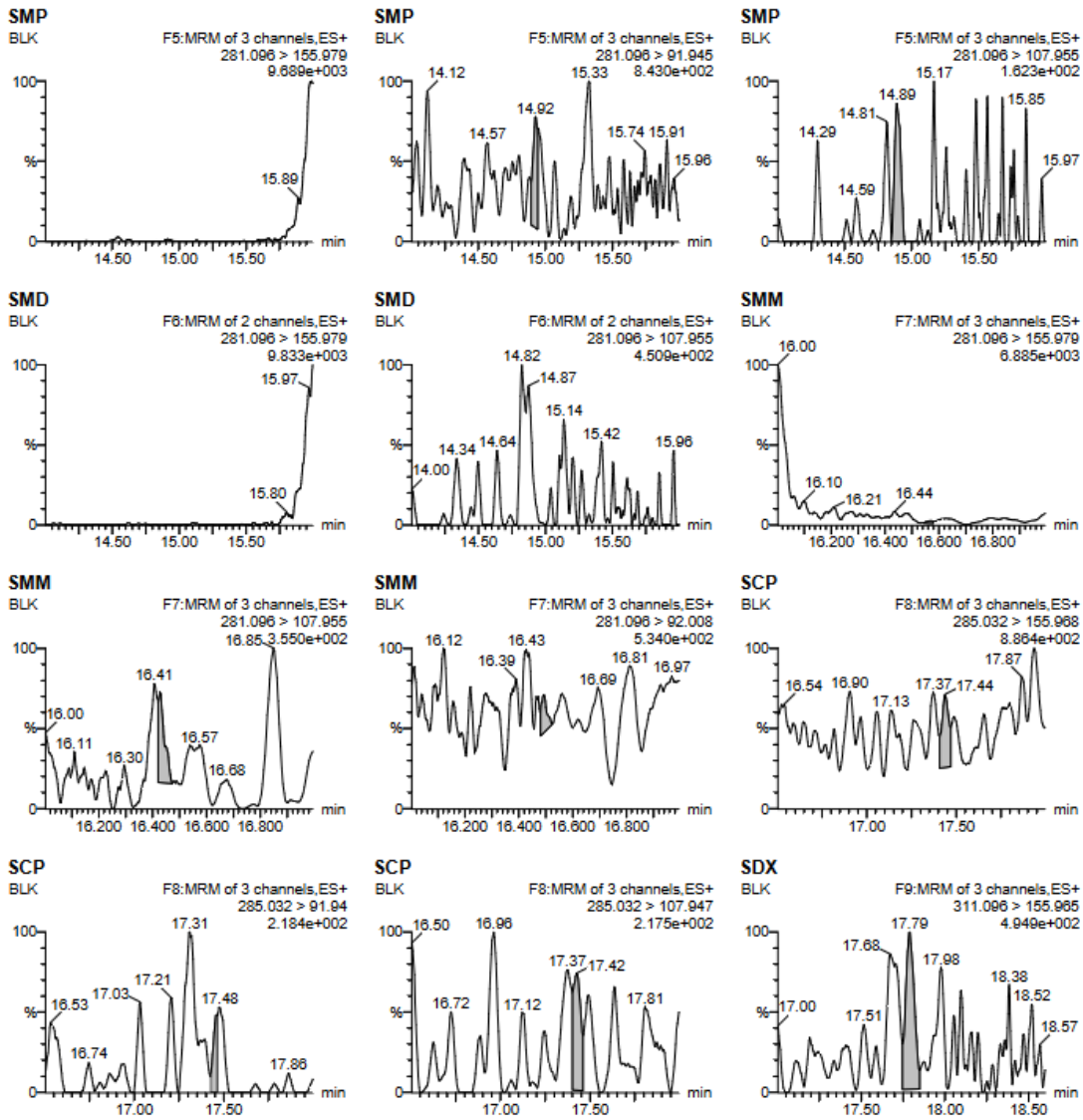
● 样品溶液的配制:

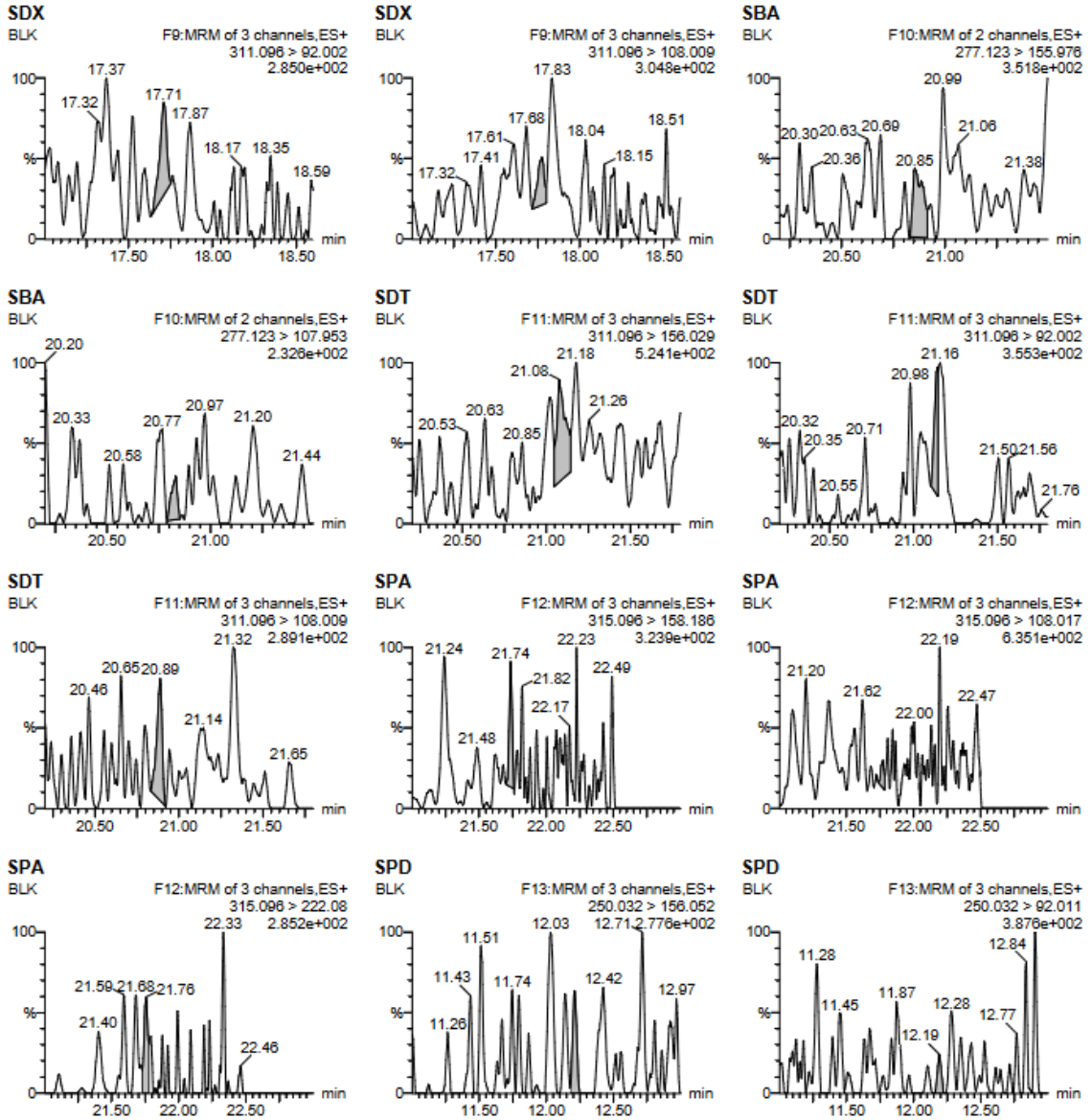
称取样品 2 g, C18 填料 6.00 g, 置于 50 mL 离心管中, 加入 25 mL 乙腈水溶液, 10000r/min 均质 1 min, 6000r/min 离心 5min, 将上清液转移至 100mL 分液漏斗中, 均质机用 25 mL 乙腈均质清洗, 并将溶液倒入沉淀物中, 涡旋混匀 1 min, 6000r/min 离心 5min, 合并上清液于分液漏斗中, 加入 25 mL 乙腈饱和的正己烷溶液, 振摇 5min, 静置分层后, 将乙腈层放入离心管中, 40 °C 氮吹至 0.5 mL 刻度以下, 加入 0.5 mL 水, 用乙腈定容至 1 mL 刻度, 混匀, 过 0.22 μm 滤膜, 上机。

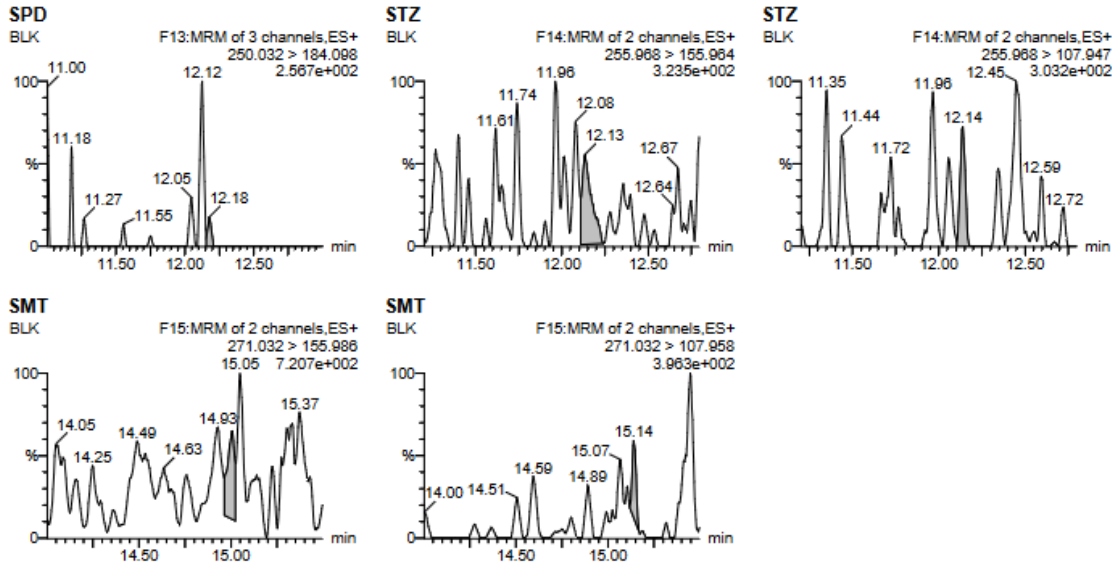
● 谱图和数据

(1) 试剂空白。





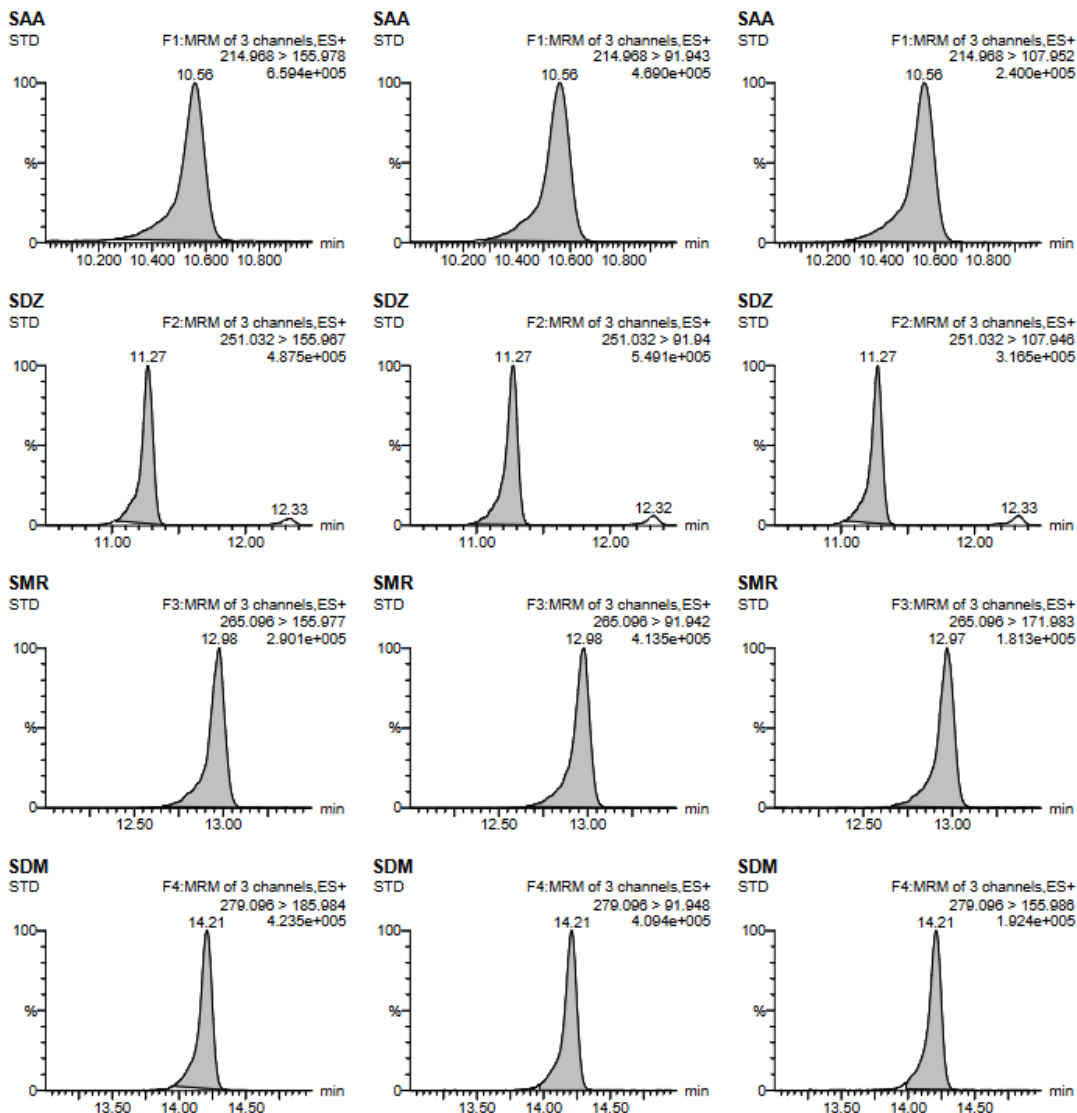


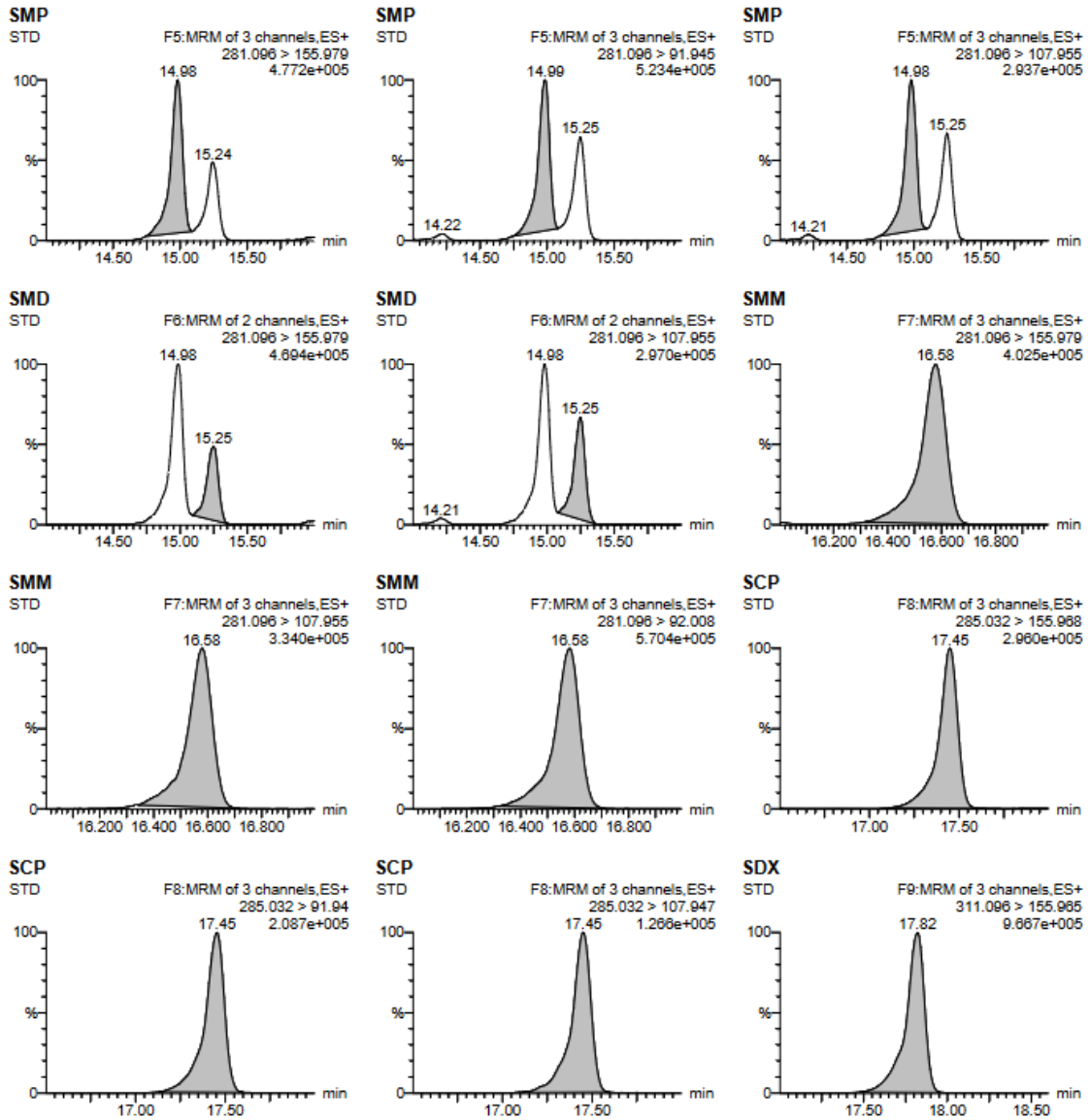


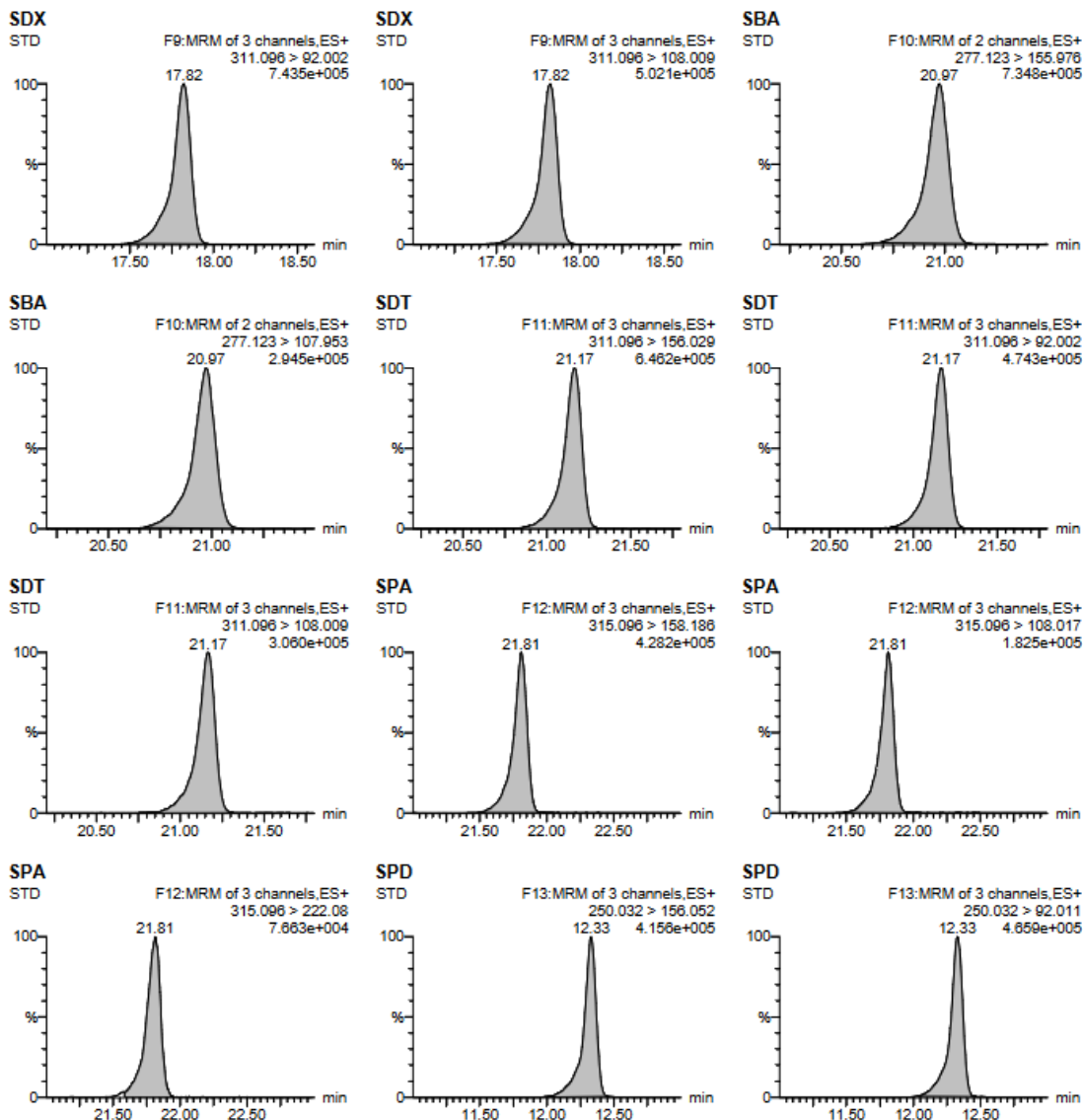
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1 ^o Ratio (A...	2 ^o Ratio (A...
BLK	SAA	214.968 > 155.978	10.43	11.550	bb	0.009		1.183	1.387
BLK	SDZ	251.032 > 155.967	11.28	13.548	bd	0.014		1.930	2.809
BLK	SMR	265.096 > 155.977	12.94	3.561	bb	0.006		0.415	1.388
BLK	SDM	279.096 > 185.984	14.12	3.642	bb	0.004		1.144	0.936
BLK	SMP	281.096 > 155.979	14.91	5.207	dd	0.006		0.242	0.716
BLK	SMD	281.096 > 155.979							
BLK	SMM	281.096 > 155.979	16.57	2.469	bd	0.003		0.491	0.835
BLK	SCP	285.032 > 155.968	17.44	17.928	dd	0.025		6.463	2.401
BLK	SDX	311.096 > 155.965	17.79	28.681	dd	0.011		3.649	7.002
BLK	SBA	277.123 > 155.976	20.85	8.416	bd	0.005		4.091	
BLK	SDT	311.096 > 156.029	21.08	19.893	dd	0.012		3.113	2.516
BLK	SPA	315.096 > 158.186	21.74	6.208	bb	0.006		2.240	1.068
BLK	SPD	250.032 > 156.052	12.21	5.993	db	0.007		2.004	4.387
BLK	STZ	255.968 > 155.964	12.13	11.130	db	0.015		1.493	
BLK	SMT	271.032 > 155.968	15.01	16.523	dd	0.016		3.734	

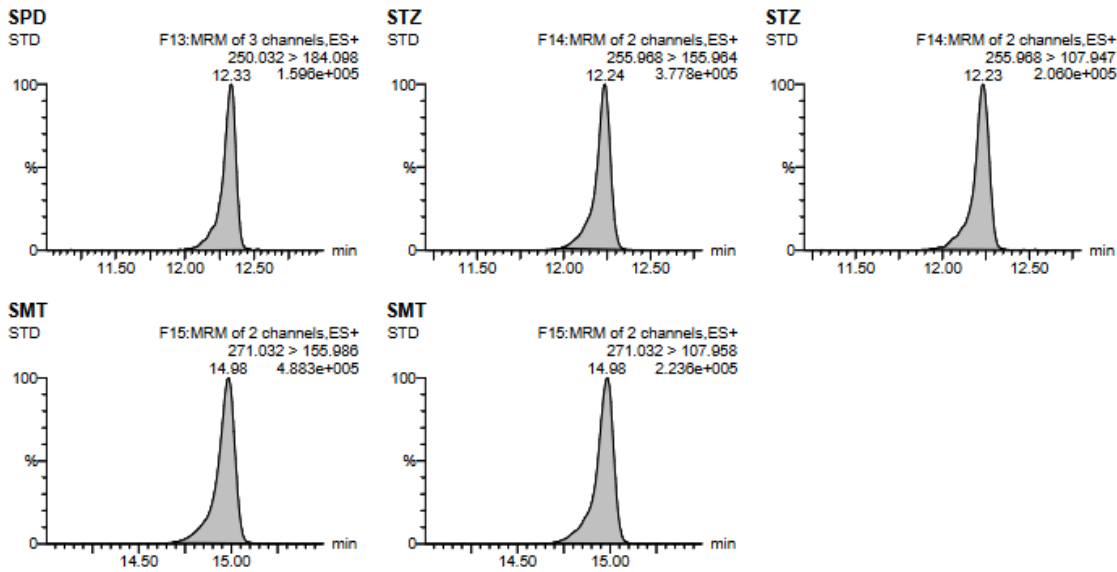
(2) 磺胺类标准品溶液浓度为 50 ng/mL。







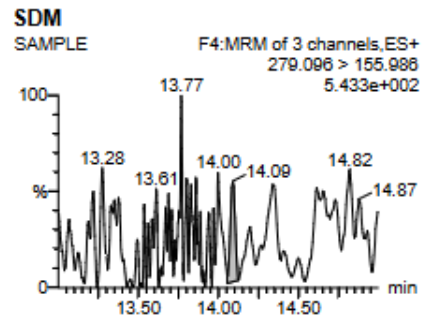
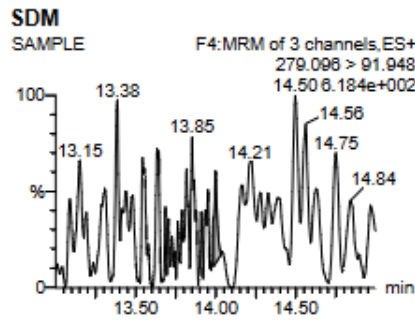
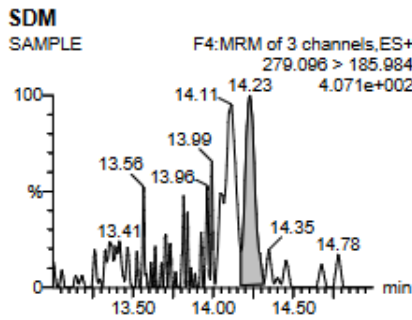
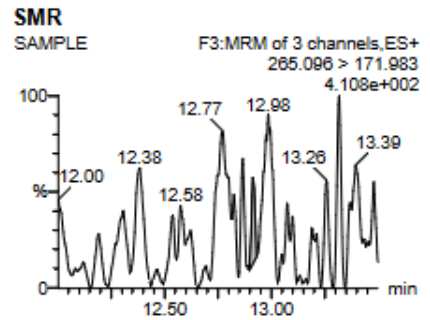
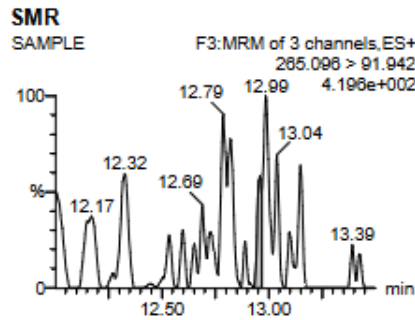
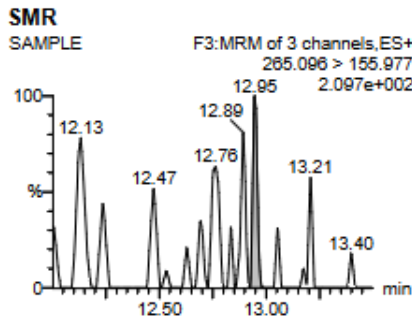
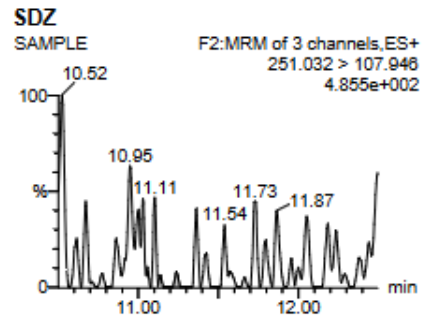
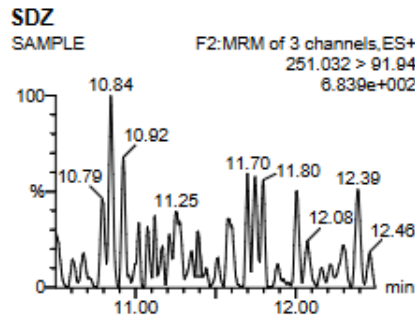
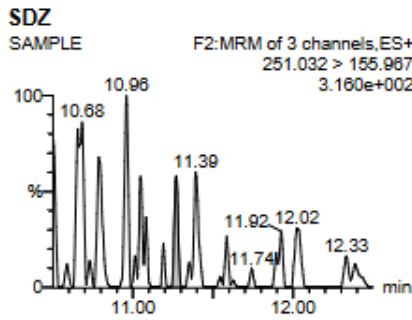
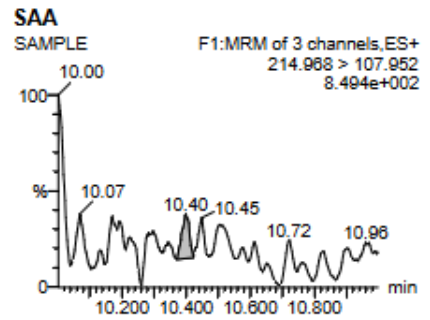
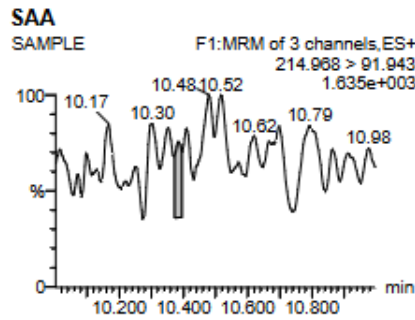
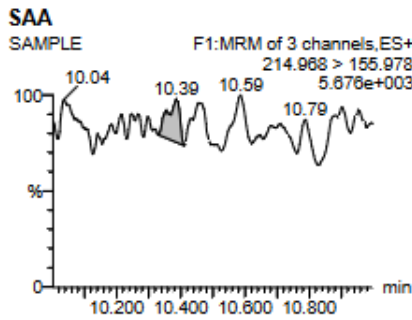


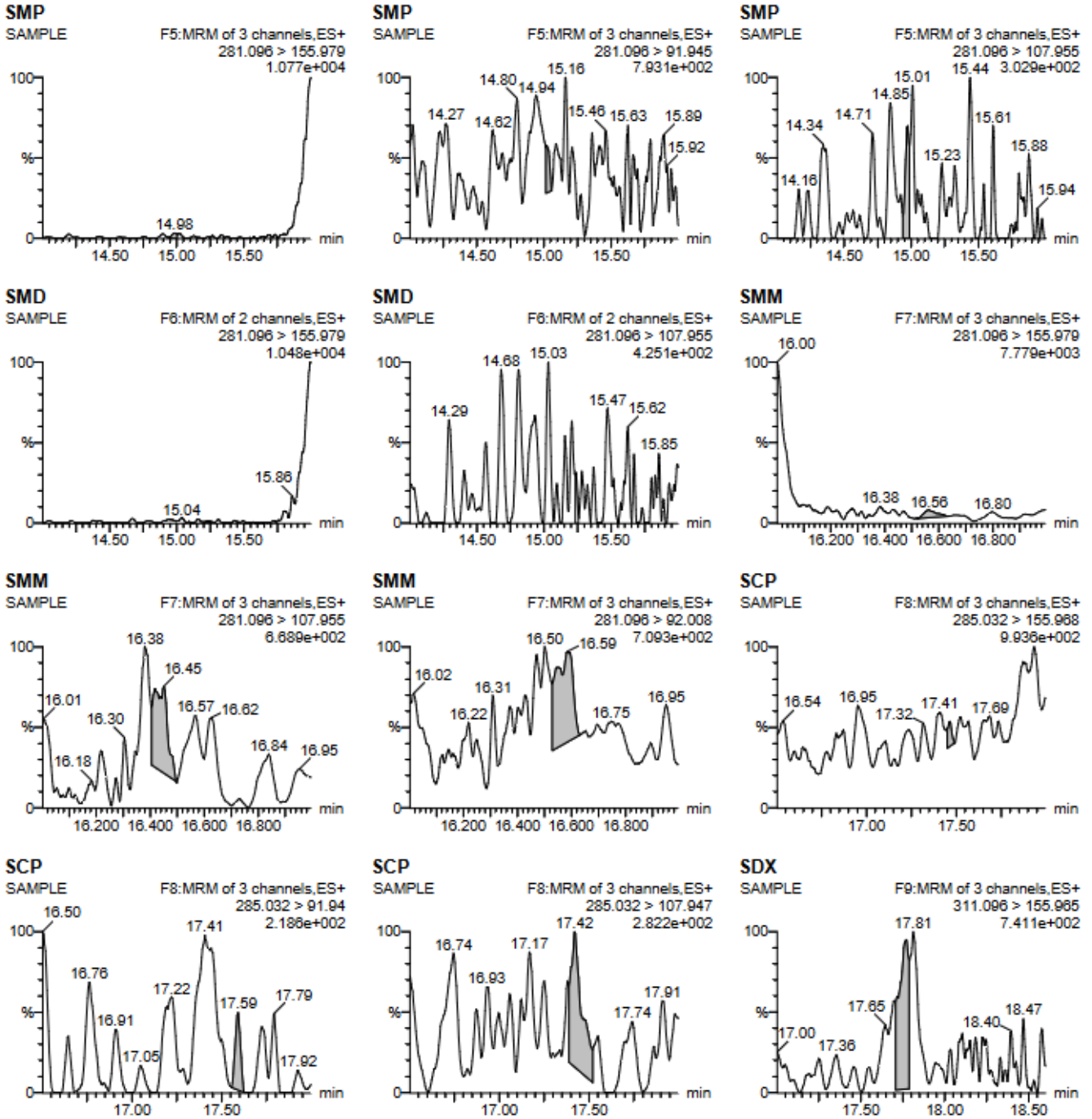


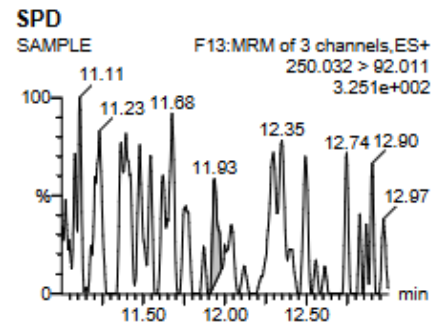
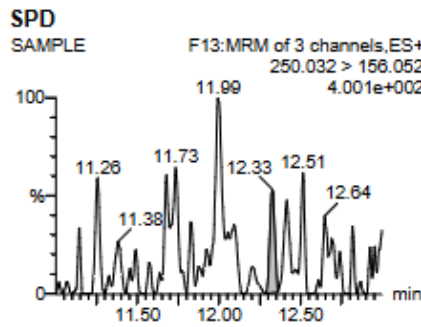
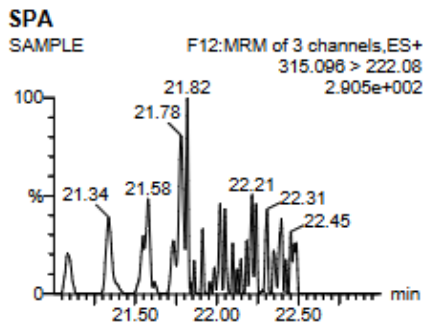
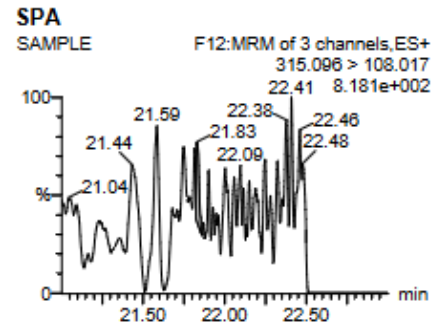
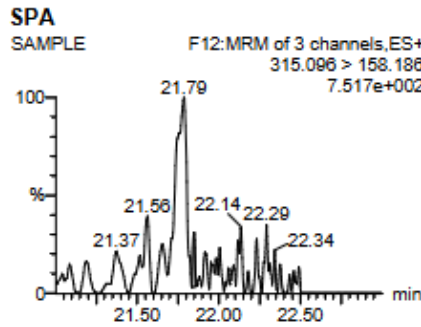
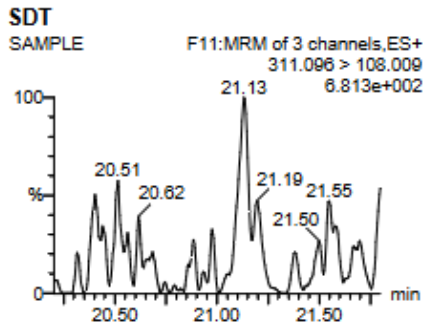
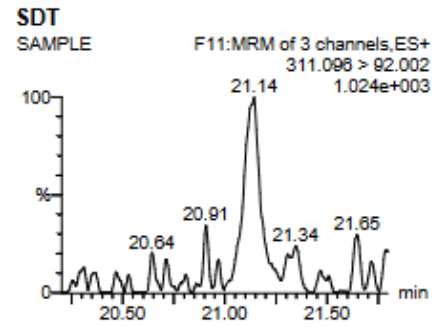
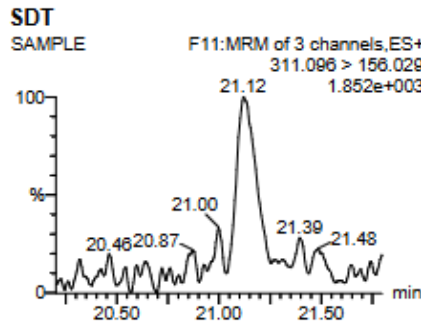
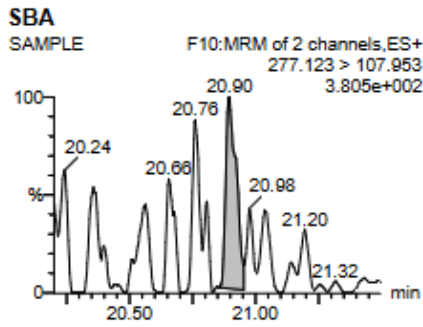
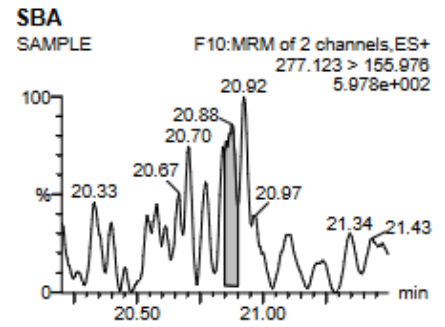
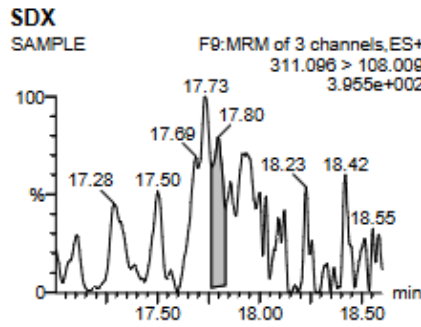
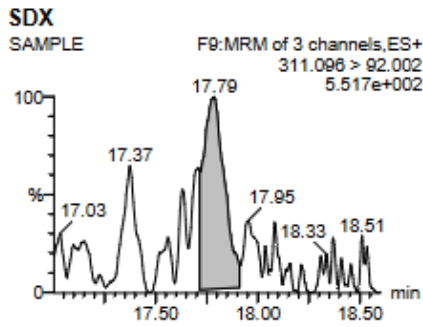
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1 ^o Ratio (A...	2 ^o Ratio (A...
STD	SAA	214.968 > 155.978	10.56	67466.797	bb	50.000	0.0	1.349	2.674
STD	SDZ	251.032 > 155.967	11.27	49161.027	bb	50.000	0.0	0.836	1.584
STD	SMR	265.096 > 155.977	12.98	30452.459	bb	50.000	0.0	0.683	1.547
STD	SDM	279.096 > 185.984	14.21	47248.285	bb	50.000	0.0	0.999	2.140
STD	SMP	281.096 > 155.979	14.98	46735.992	bb	50.000	0.0	0.933	1.670
STD	SMD	281.096 > 155.979	15.25	21610.631	bb	50.000	0.0	1.168	
STD	SMM	281.096 > 155.979	16.58	43751.844	bb	50.000	0.0	1.226	0.707
STD	SCP	285.032 > 155.968	17.45	36300.324	bb	50.000	0.0	1.370	2.274
STD	SDX	311.096 > 155.965	17.82	125140.414	bb	50.000	0.0	1.314	1.967
STD	SBA	277.123 > 155.976	20.97	91753.469	bb	50.000	0.0	2.454	
STD	SDT	311.096 > 158.029	21.17	81804.414	bb	50.000	0.0	1.409	2.147
STD	SPA	315.096 > 158.186	21.81	51034.762	bb	50.000	0.0	2.358	5.414
STD	SPD	250.032 > 158.052	12.33	45246.844	bb	50.000	0.0	0.890	2.535
STD	STZ	255.968 > 155.964	12.24	36823.844	bb	50.000	0.0	1.765	
STD	SMT	271.032 > 155.988	14.98	52901.566	bb	50.000	0.0	2.197	

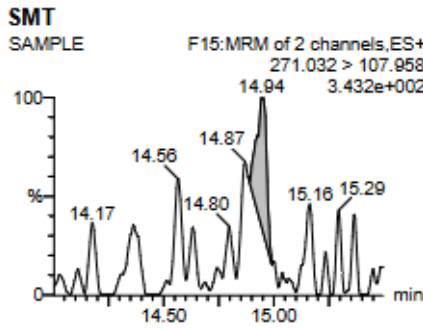
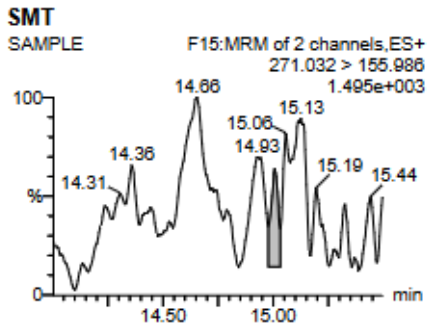
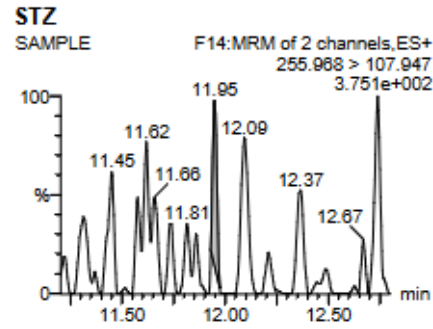
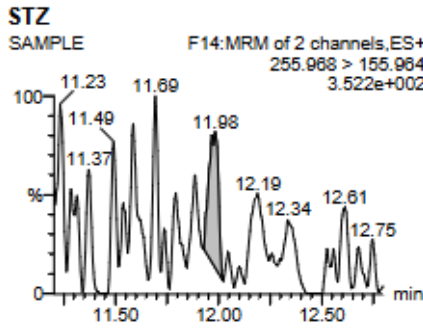
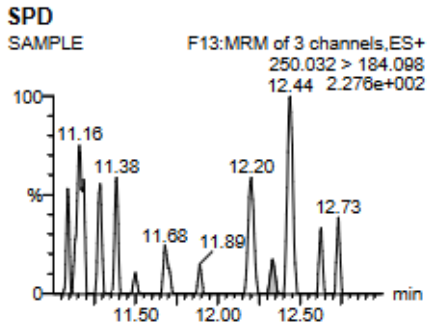
(3) 虾肉样品本底。







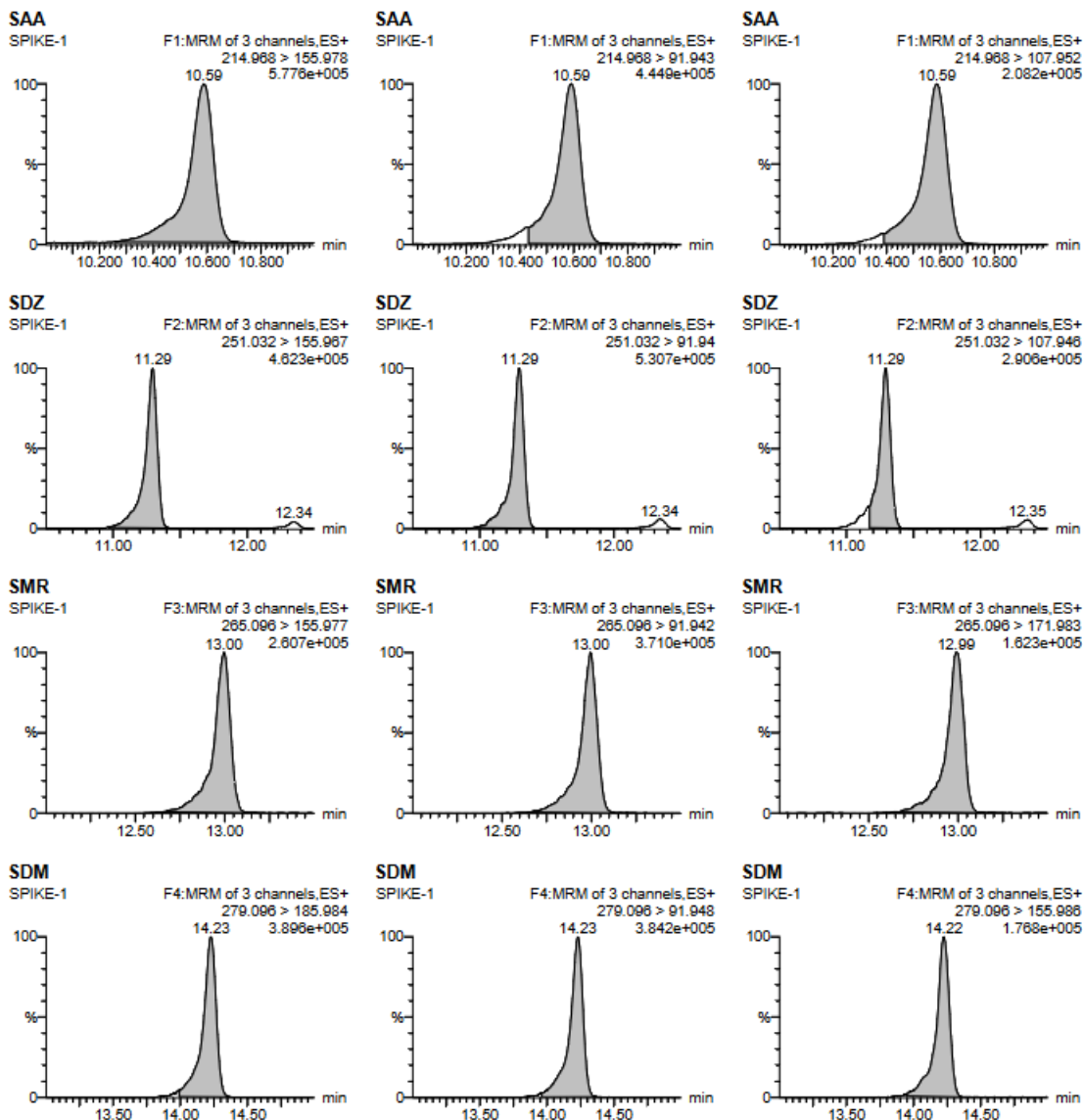


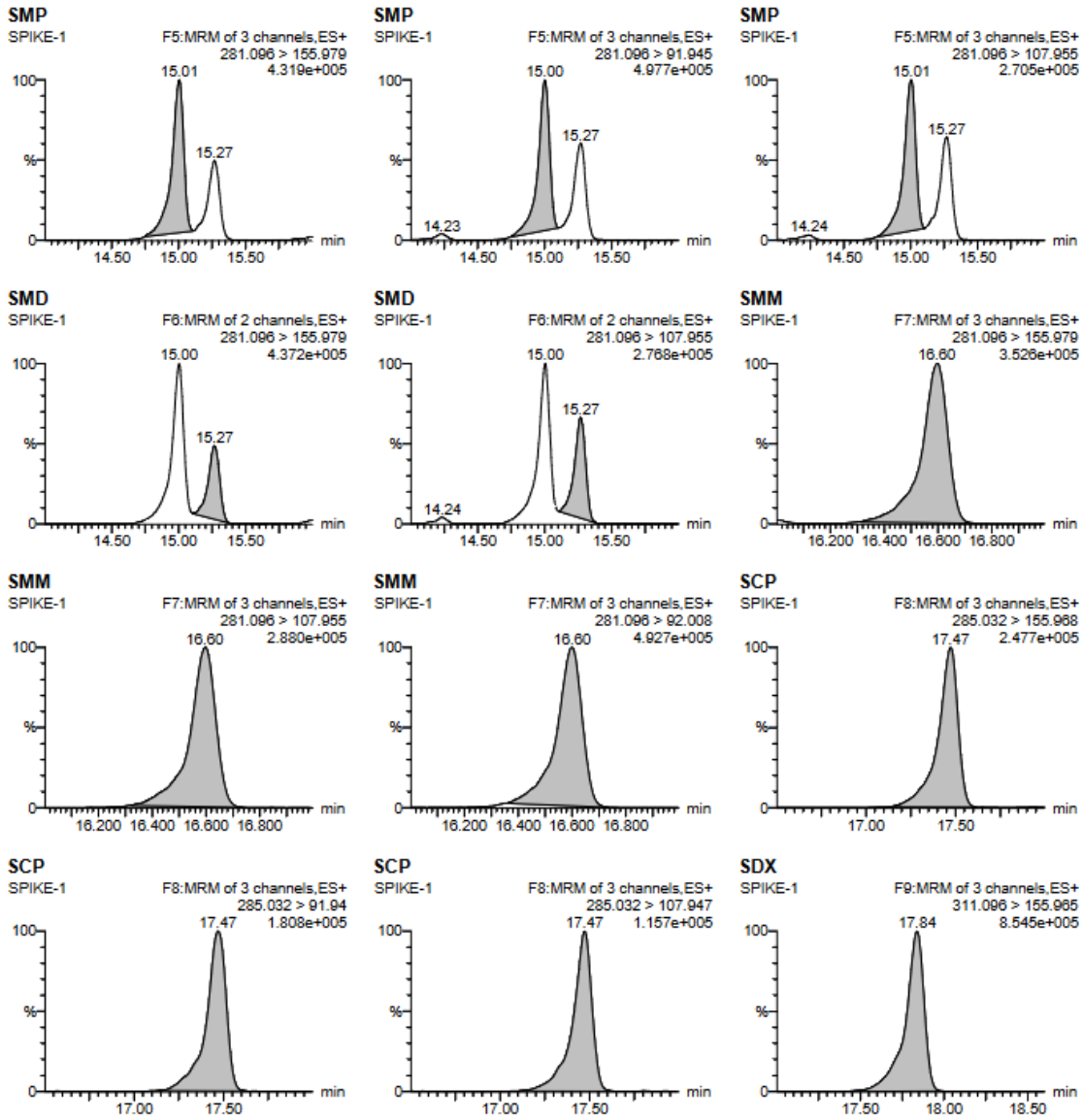


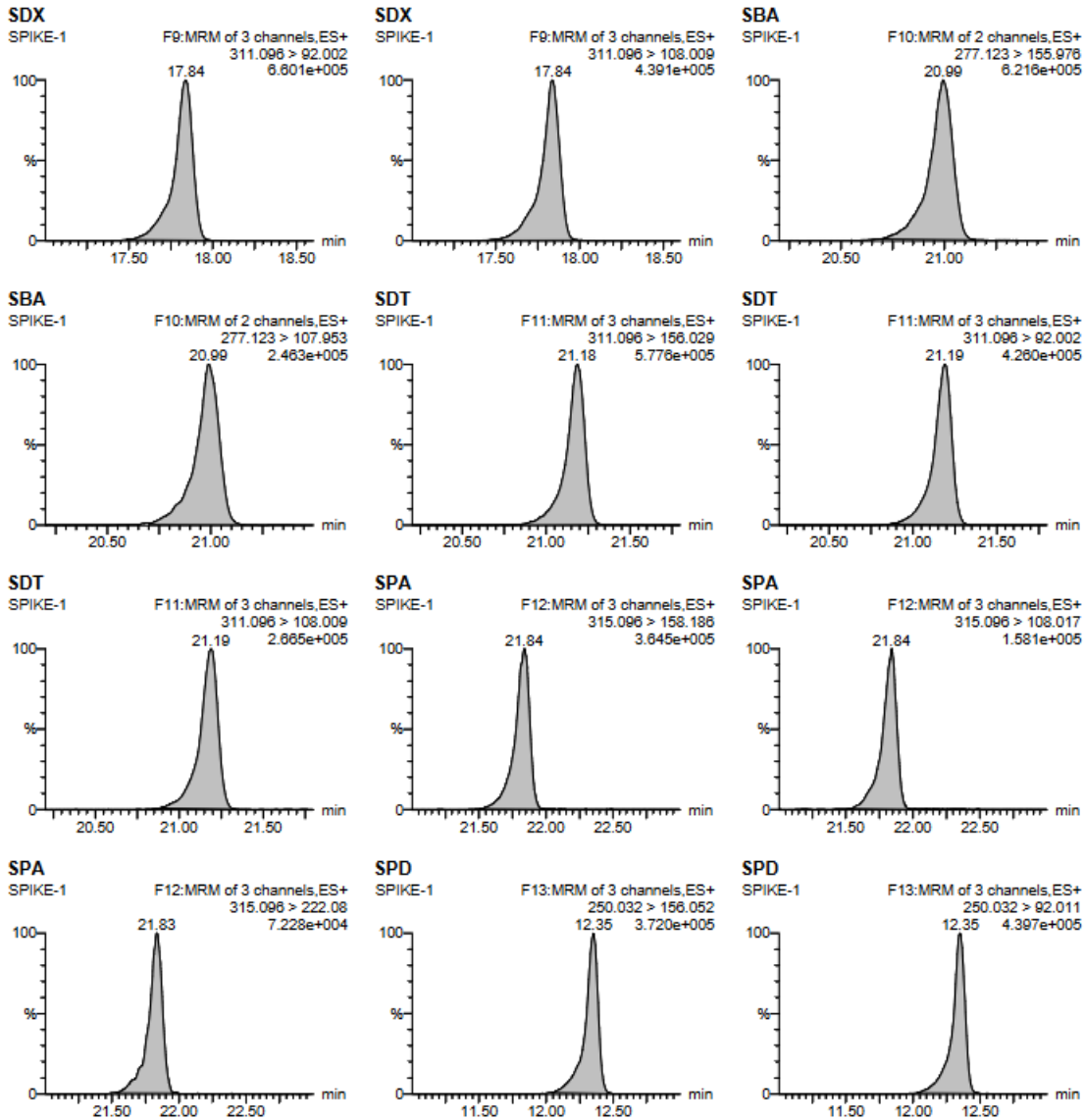
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1 ^o Ratio (A...	2 ^o Ratio (A...
SAMPLE	SAA	214.968 > 155.978	10.39	54.470	bb	0.040		4.118	10.427
SAMPLE	SDZ	251.032 > 155.967	11.27	4.867	bb	0.005		1.324	5.426
SAMPLE	SMR	265.096 > 155.977	12.95	5.038	bb	0.008		0.922	1.568
SAMPLE	SDM	279.096 > 185.984	14.23	28.042	dd	0.030		4.976	2.955
SAMPLE	SMP	281.096 > 155.979	14.98	13.222	dd	0.014		2.010	1.961
SAMPLE	SMD	281.096 > 155.979	15.24	2.106	db	0.005		0.842	
SAMPLE	SMM	281.096 > 155.979	16.56	19.286	bb	0.022		0.943	0.672
SAMPLE	SCP	285.032 > 155.968	17.46	3.650	db	0.005		1.035	0.213
SAMPLE	SDX	311.096 > 155.965	17.77	43.356	dd	0.017		0.677	2.636
SAMPLE	SBA	277.123 > 155.976	20.88	21.750	dd	0.012		1.226	
SAMPLE	SDT	311.096 > 156.029							
SAMPLE	SPA	315.096 > 158.186	21.85	2.918	bb	0.003		0.459	16.770
SAMPLE	SPD	250.032 > 156.052	12.33	7.221	bb	0.008		1.169	5.904
SAMPLE	STZ	255.968 > 155.964	11.98	12.660	bb	0.017		1.771	
SAMPLE	SMT	271.032 > 155.986	15.01	26.906	dd	0.025		2.262	

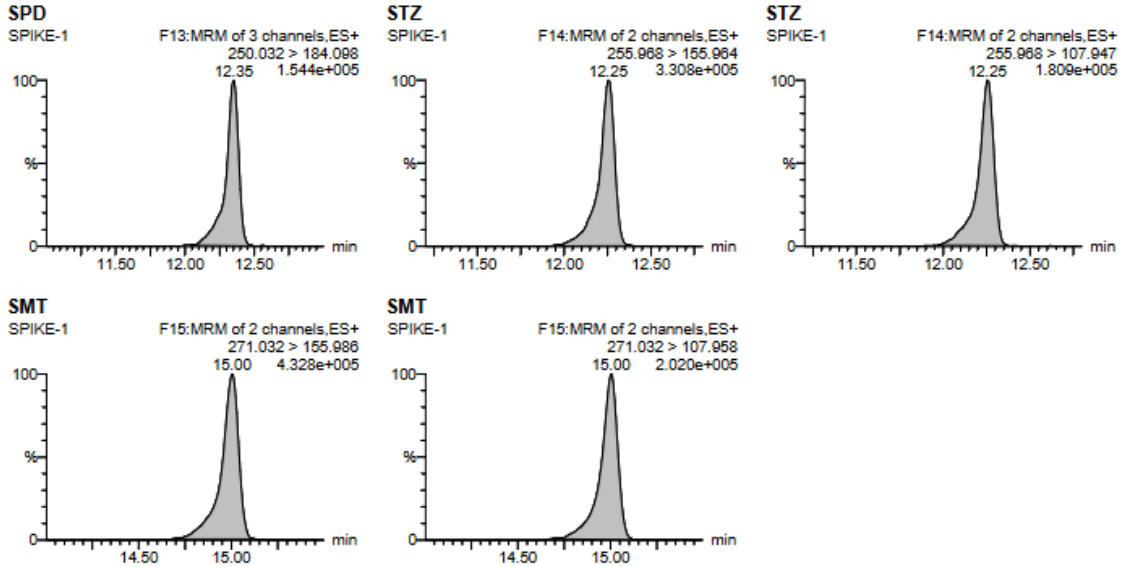
(4) 加标回收: 磺胺类标准品加标浓度 50 ng/mL。





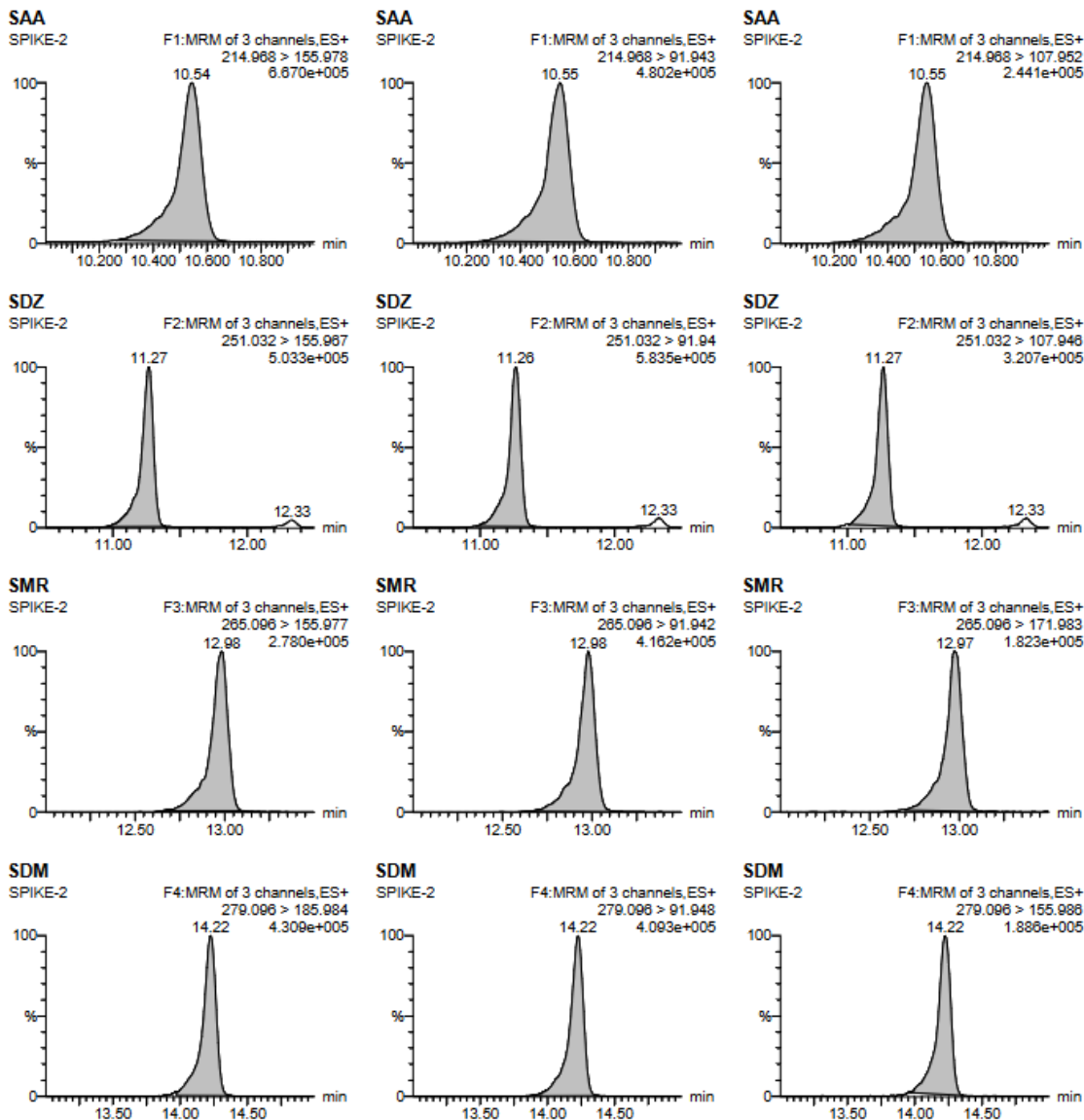


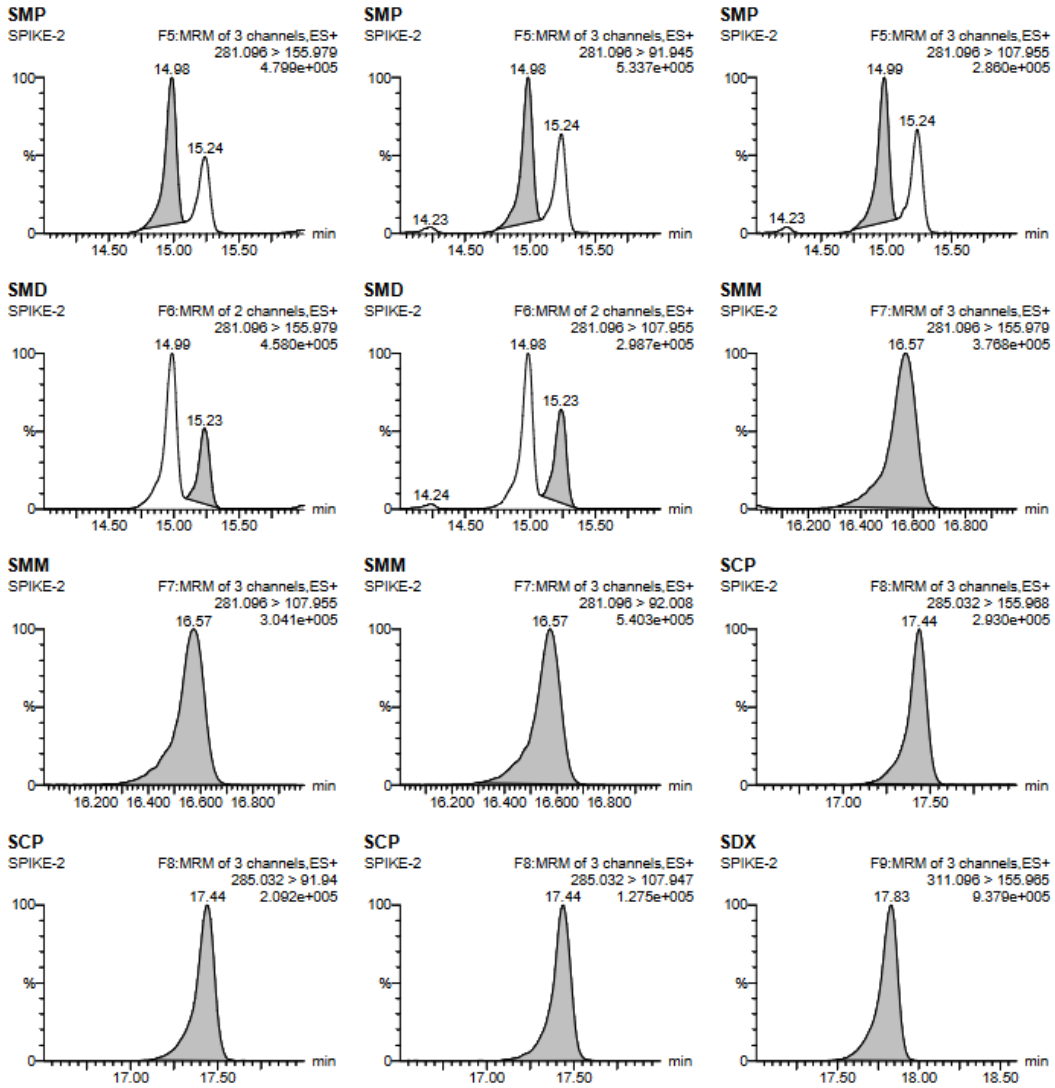


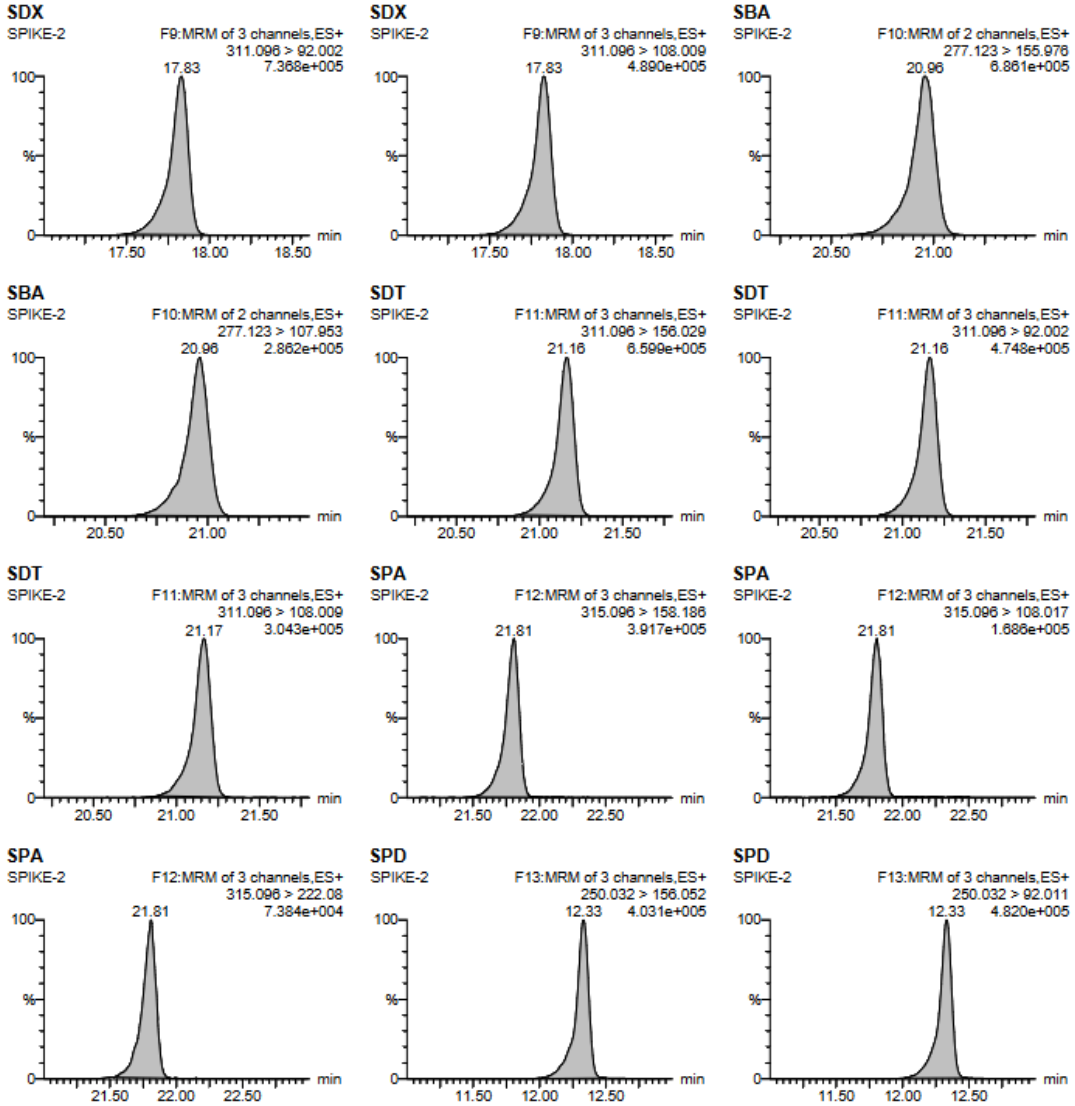


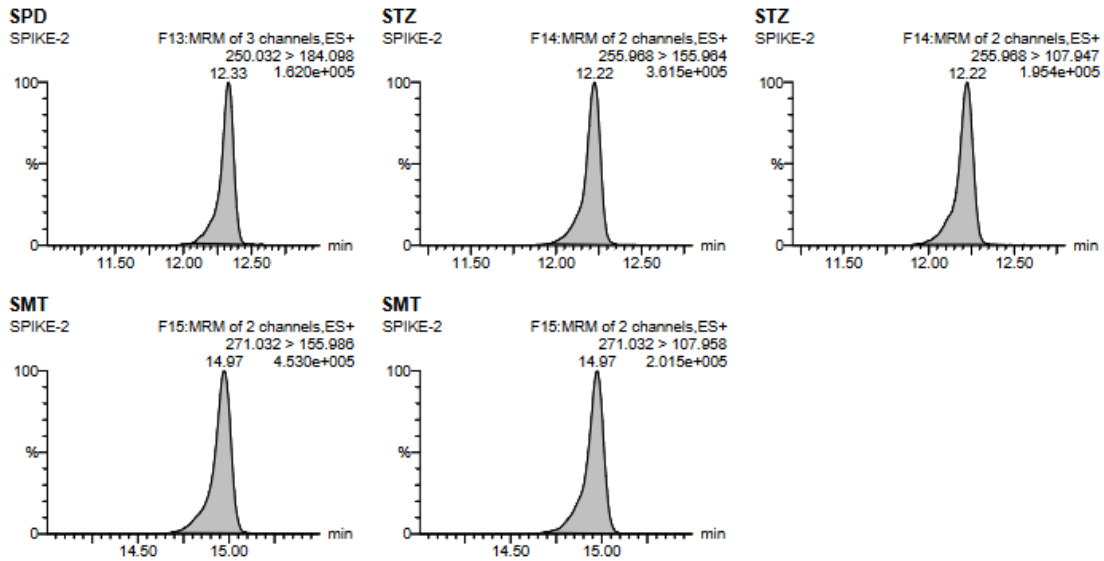
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1 ^o Ratio (A...	2 ^o Ratio (A...
SPIKE-1	SAA	214.068 > 155.978	10.59	62317.375	bb	46.184		1.388	2.782
SPIKE-1	SDZ	251.032 > 155.967	11.29	48491.652	bb	49.319		0.864	1.758
SPIKE-1	SMR	265.096 > 155.977	13.00	28701.861	bb	47.126		0.706	1.567
SPIKE-1	SDM	279.096 > 185.984	14.23	45084.121	db	47.710		1.023	2.223
SPIKE-1	SMP	281.096 > 155.979	15.01	42711.297	bb	45.694		0.915	1.610
SPIKE-1	SMD	281.096 > 155.979	15.27	19748.551	bb	45.692		1.172	
SPIKE-1	SMM	281.096 > 155.979	16.60	40216.531	bb	45.960		1.234	0.739
SPIKE-1	SCP	285.032 > 155.968	17.47	31754.539	bb	43.739		1.352	2.207
SPIKE-1	SDX	311.096 > 155.965	17.84	111785.227	bb	44.664		1.313	1.989
SPIKE-1	SBA	277.123 > 155.976	20.99	80423.172	bb	43.826		2.499	
SPIKE-1	SDT	311.096 > 156.029	21.18	73256.563	bb	44.775		1.381	2.207
SPIKE-1	SPA	315.096 > 158.188	21.84	46194.531	bb	45.258		2.363	5.354
SPIKE-1	SPD	250.032 > 156.052	12.35	41211.301	bb	45.541		0.852	2.477
SPIKE-1	STZ	255.968 > 155.964	12.25	34481.473	db	46.819		1.816	
SPIKE-1	SMT	271.032 > 155.988	15.00	46568.164	bb	44.014		2.175	











Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	%Dev	1 ^o Ratio (A...	2 ^o Ratio (A...
SPIKE-2	SAA	214.968 > 155.978	10.54	68543.320	bb	50.798		1.341	2.698
SPIKE-2	SDZ	251.032 > 155.967	11.27	53129.770	bb	54.036		0.864	1.624
SPIKE-2	SMR	265.096 > 155.977	12.98	30830.842	bb	50.621		0.675	1.537
SPIKE-2	SDM	279.096 > 185.984	14.22	50845.395	db	53.595		1.044	2.326
SPIKE-2	SMP	281.096 > 155.979	14.98	46188.328	bb	49.414		0.928	1.705
SPIKE-2	SMD	281.096 > 155.979	15.23	21697.119	bb	50.200		1.171	
SPIKE-2	SMM	281.096 > 155.979	16.57	43065.637	bb	49.216		1.187	0.706
SPIKE-2	SCP	285.032 > 155.968	17.44	35545.391	bb	48.960		1.341	2.247
SPIKE-2	SDX	311.096 > 155.965	17.83	123040.266	bb	49.161		1.298	1.984
SPIKE-2	SBA	277.123 > 155.976	20.96	89743.727	bb	48.905		2.499	
SPIKE-2	SDT	311.096 > 156.029	21.16	80807.555	bb	49.391		1.366	2.184
SPIKE-2	SPA	315.096 > 158.186	21.81	47573.090	bb	46.609		2.330	5.499
SPIKE-2	SPD	250.032 > 156.052	12.33	45321.680	bb	50.083		0.855	2.535
SPIKE-2	STZ	255.968 > 155.964	12.22	37102.293	bb	50.378		1.841	
SPIKE-2	SMT	271.032 > 155.988	14.97	48974.527	bb	46.288		2.265	

● 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, %
磺胺醋酰	50.798	50	101.6
	46.184		92.4
磺胺嘧啶	54.036	50	108.1
	49.319		98.6
磺胺甲噻啶	50.621	50	101.2
	47.126		94.3
磺胺二甲嘧啶	53.595	50	107.2
	47.71		95.4



磺胺甲氧嗪	49.414	50	98.8
	45.694		91.4
磺胺对甲氧嘧啶	50.2	50	100.4
	45.692		91.4
磺胺间甲氧嘧啶	49.216	50	98.4
	45.96		91.9
磺胺氯达嗪	48.96	50	97.9
	43.739		87.5
磺胺多辛	49.161	50	98.3
	44.664		89.3
磺胺苯酰	48.905	50	97.8
	43.826		87.7
磺胺地索辛	49.391	50	98.8
	44.775		89.6
磺胺苯吡唑	46.609	50	93.2
	45.258		90.5
磺胺吡啶	50.083	50	100.2
	45.541		91.1
磺胺噻唑	50.378	50	100.8
	46.819		93.6
磺胺甲二唑	46.288	50	92.6
	44.014		88.0

使用 Welchrom® C18 Part Number: 00504-11017 填料检测虾肉中磺胺类化合物, 回收率良好且稳定, 。

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