

测试报告

样品信息			
样品名称	化妆水	编号	Z20220810-003
样品重量	/	剂型	/
收样日期	2022/8/8	测试期间	2022/8/8-2022/8/10
样品描述	/		
测试需求			
测试成分	苯甲酸、4-羟甲基苯甲酸戊酯、2-溴-2-硝基丙烷-1,3-二醇、脱氢乙酸、4-羟基苯甲酸苯甲酯、4-羟基苯甲酸苯酯、4-羟基苯甲酸丙酯、4-羟基苯甲酸丁酯、4-羟基苯甲酸乙酯、4-羟基苯甲酸甲酯、4-羟基苯甲酸异丁酯、4-羟基苯甲酸异丙酯		
参考标准			
参考标准	化妆品安全技术规范 2015 版 修订	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% (5mmol/L 乙酸铵水溶液)	流动相 B% (5mmol/L 乙酸铵甲醇溶液)
	0.0	95	5
	4.0	95	5
	8.0	50	50
	16.0	10	90
	20.0	5	95



	20.1	95	5
	24.0	95	5
柱温:	30°C		
流速:	0.35 mL/min		
进样量:	1 μ L		
注意事项:	/		

● 流动相的配置:

流动相 A : 称取 0.1927 g 乙酸铵, 加水至 500 mL 混匀, 经 0.22 μ m 滤膜抽滤, 即得;

流动相 B : 称取 0.1927 g 乙酸铵, 加色谱级甲醇 500 mL 混匀, 经 0.22 μ 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, 400°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
2-溴-2-硝基丙烷 -1,3-二醇 (BNP)	170.1	60.1	30	12	Negative
		81.0		16	
		88.1		8	
4-羟基苯甲酸异丁 酯 (4-HBAIe)	193.2	71.1	14	30	Negative
		92.2		22	
		136.2		16	
4-羟基苯甲酸丁酯 (p-HBAB)	193.2	71.1	2	26	Negative
		92.2		28	
		136.3		16	
4-羟基苯甲酸异丙	179.2	56.7	22	28	Negative



酯（p-HBAI）		92.4		8	
		137.1		12	
4-羟基苯甲酸丙酯 (P p-h)	179.2	57.1	22	30	Negative
		92.2		22	
		136.3		16	
4-羟基苯甲酸乙酯 (E p-h)	165.2	92.2	10	22	Negative
		137.0		12	
4-羟基苯甲酸甲酯 (M p-h)	151.2	92.1	28	22	Negative
		136.1		12	
苯甲酸 (Benzoic acid)	121.2	77.1	4	10	Negative
		121.2		14	
脱氢乙酸（DHAA）	167.2	83.1	20	14	Negative
		123.1		8	
4-羟基苯甲酸苄酯 (Benzyl paraben)	227.3	92.1	28	24	Negative
		136.1		12	
4-羟基苯甲酸苯酯 (Phenyl ester)	213.2	65.0	20	30	Negative
		93.1		12	
4-羟基苯甲酸戊酯 (N-P-4-HB)	207.3	85.2	6	30	Negative
		92.2		26	
		136.3		16	
		158.4		30	
		302.5		30	

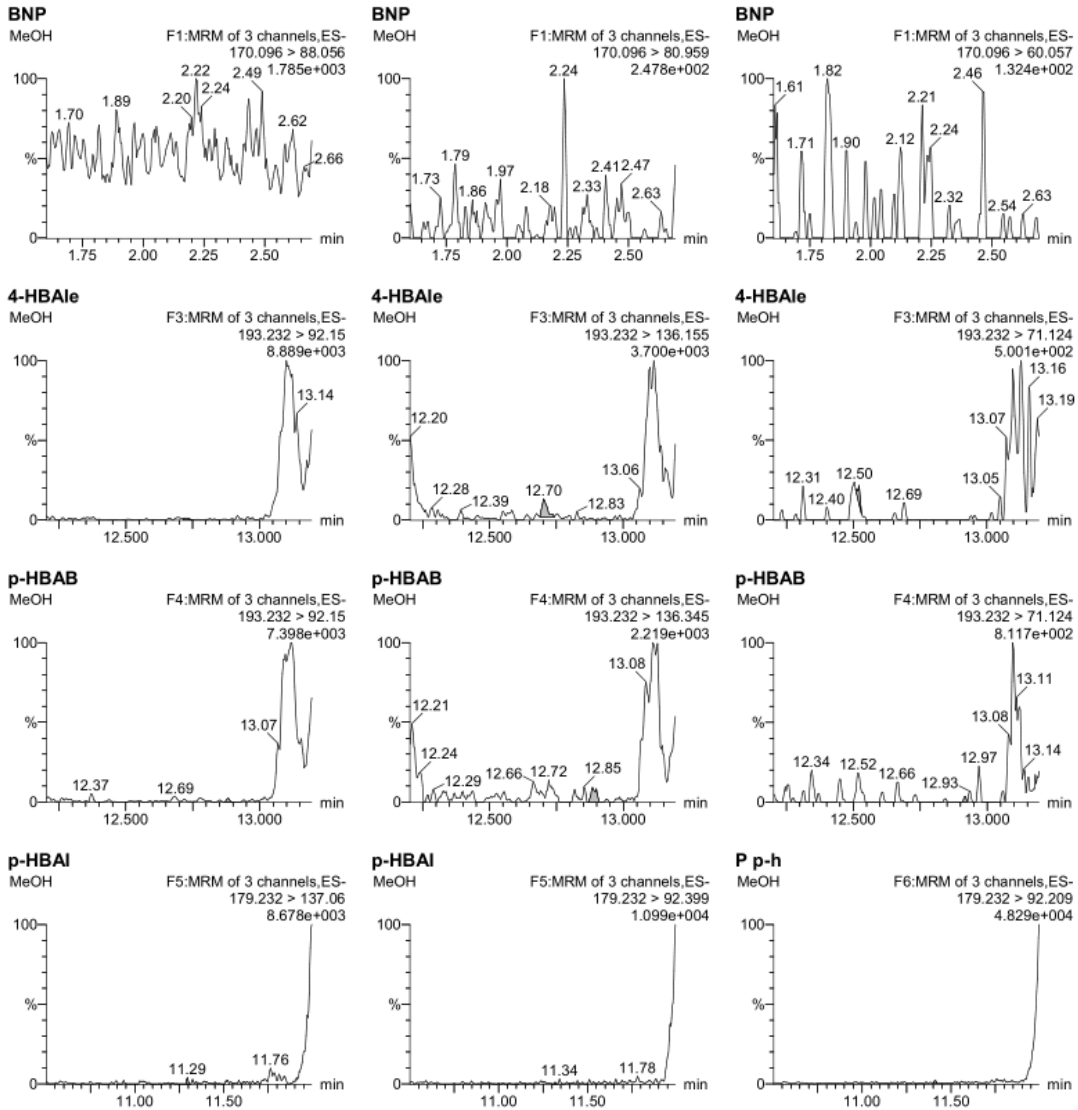
● 样品溶液的配制：

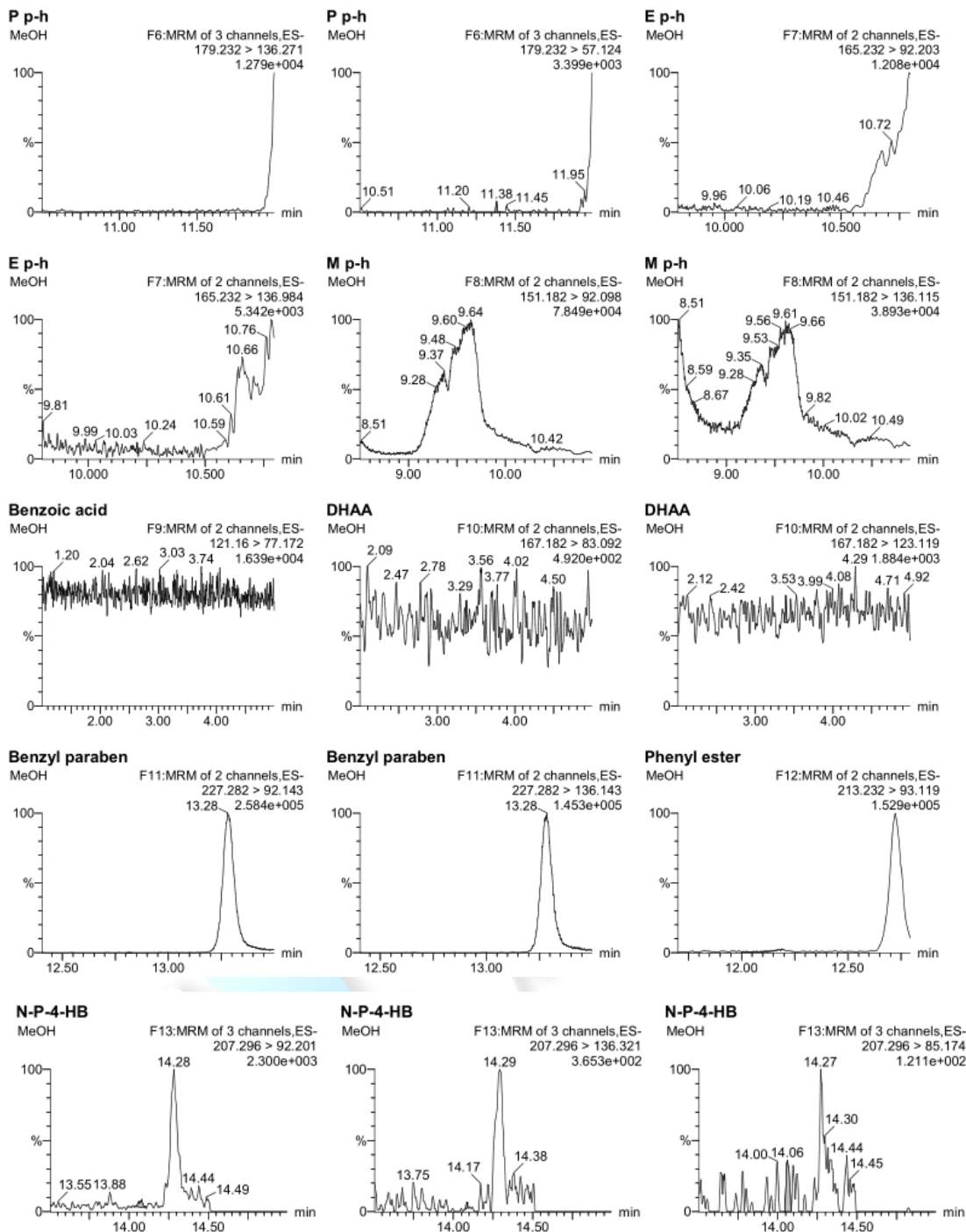
称取样品 1.0 g 置于 10 mL 离心管中，加入 8 mL 甲醇，涡旋振荡 30s，超声提取 20min，用甲醇定容至 10 mL，摇匀，以 10000 r/min 离心 5 min，过 0.22 μm PTFE 滤膜，上机。



● 谱图和数据

(1) 空白

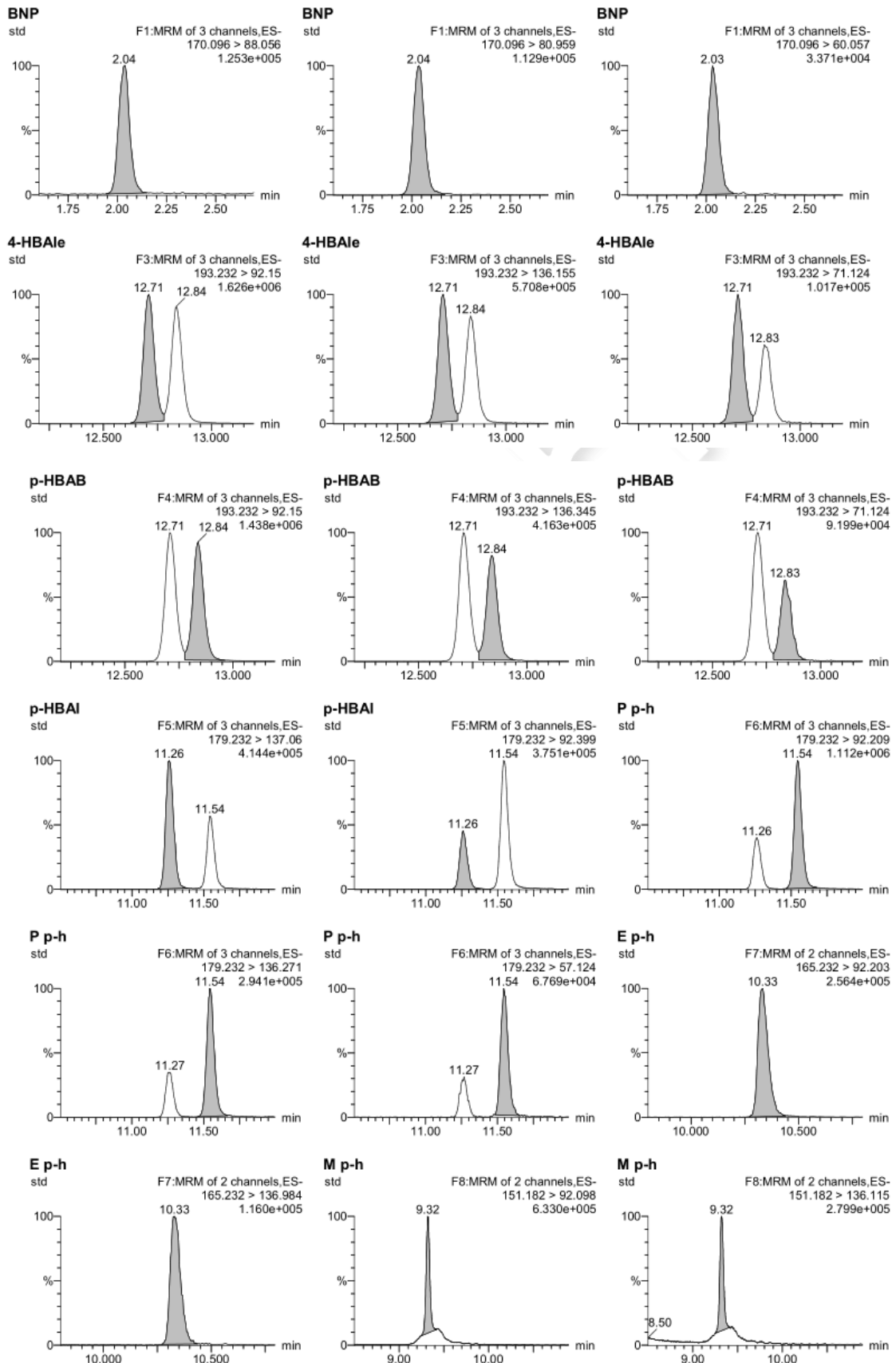


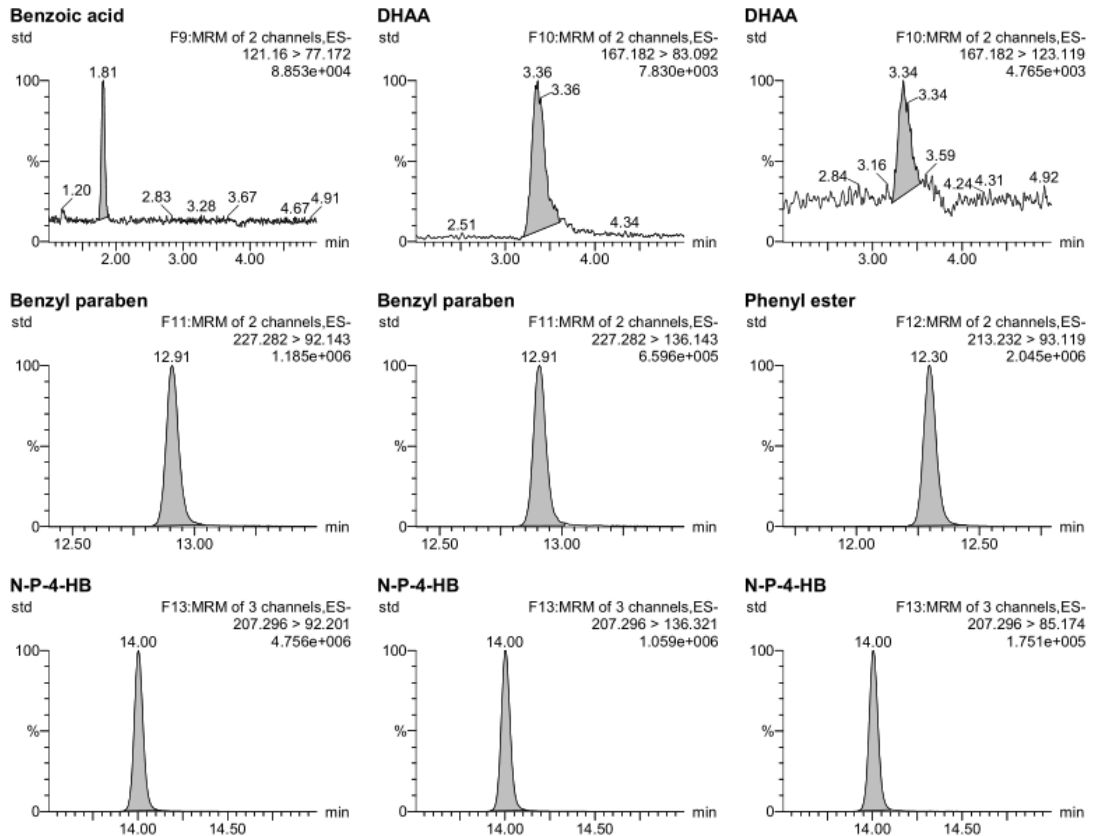


Sample Text	Name	Trace	RT	Area Primar...	ug/mL	%Dev	1° Ratio (A...	2° Ratio (A...
MeOH	BNP	170.096 > 88.056						
MeOH	4-HBAle	193.232 > 92.15	12.72	2.342 db	0.000		0.250	4.361
MeOH	p-HBAB	193.232 > 92.15	12.88	2.139 bb	0.000		0.474	7.325
MeOH	p-HBAI	179.232 > 137.06	11.29	2.906 bb	0.000		3.200	
MeOH	P p-h	179.232 > 92.209	11.41	10.158 bb	0.000		9.969	4.853
MeOH	E p-h	165.232 > 92.203	10.44	2.346 bd	0.000		1.181	
MeOH	M p-h	151.182 > 92.098						
MeOH	Benzoic acid	121.16 > 77.172	1.74	10.899 bb	0.001			
MeOH	DHAA	167.182 > 83.092	3.38	2.356 dd	0.000		0.359	
MeOH	Benzyl paraben	227.282 > 92.143	12.81	4.304 bb	0.000			
MeOH	Phenyl ester	213.232 > 93.119						
MeOH	N-P-4-HB	207.296 > 92.201	14.08	3.247 bb	0.000		19.327	3.128



(2) 2-溴-2-硝基丙烷-1,3-二醇等 12 种组分标准液，浓度见下图。

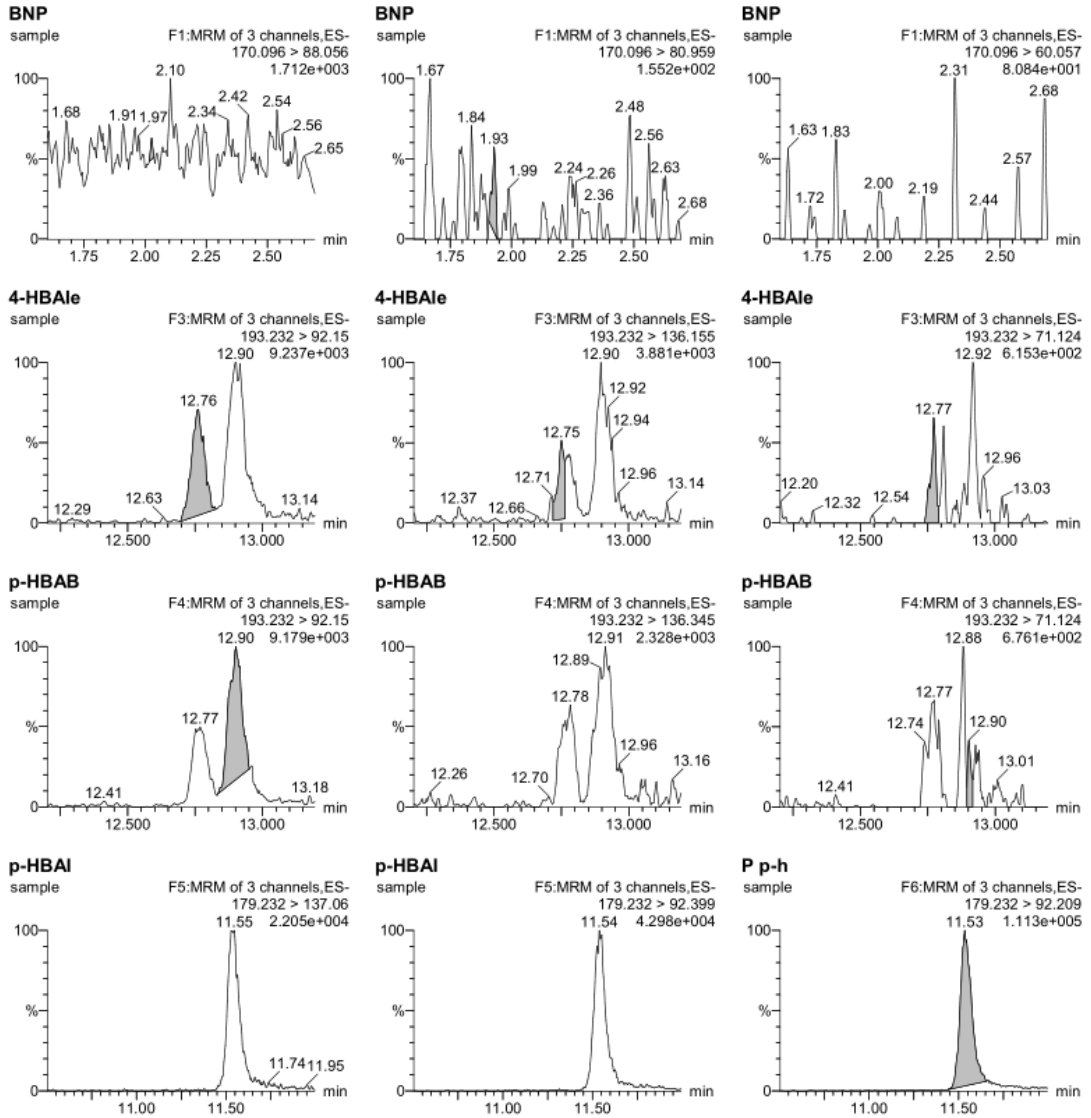


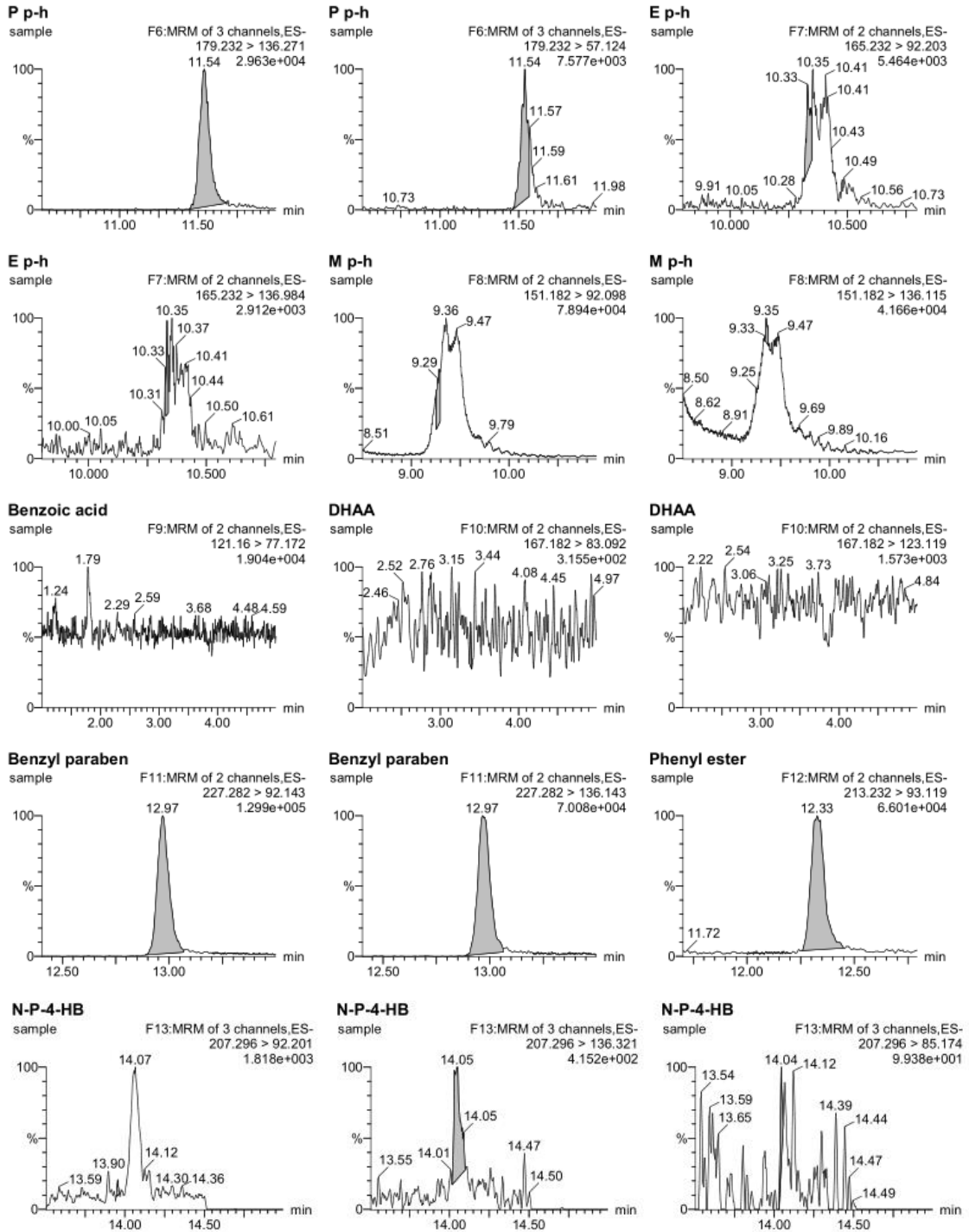


Sample Text	Name	Trace	RT	Area	Primar...	ug/mL	%Dev	1° Ratio (A...	2° Ratio (A...
std	BNP	170.096 > 88.056	2.04	7982.881	bb	0.750	0.0	1.078	3.929
std	4-HBAle	193.232 > 92.15	12.71	93270.398	bd	0.150	0.0	2.874	16.232
std	p-HBAB	193.232 > 92.15	12.84	77091.445	db	0.150	0.0	3.861	23.204
std	p-HBAI	179.232 > 137.06	11.26	26112.938	bb	0.125	0.0	2.523	
std	P p-h	179.232 > 92.209	11.54	66877.891	bb	0.125	0.0	3.786	16.906
std	E p-h	165.232 > 92.203	10.33	13878.793	bb	0.125	0.0	2.171	
std	M p-h	151.182 > 92.098	9.32	27117.453	bb	0.050	0.0	2.276	
std	Benzoic acid	121.16 > 77.172	1.81	4443.116	bb	0.250	0.0		
std	DHAA	167.182 > 83.092	3.36	1202.311	bb	0.150	0.0	2.476	
std	Benzyl paraben	227.282 > 92.143	12.91	69859.359	bb	0.150	0.0	1.763	
std	Phenyl ester	213.232 > 93.119	12.30	121185.539	bb	0.150	0.0		
std	N-P-4-HB	207.296 > 92.201	14.00	270351.031	bb	0.300	0.0	4.443	27.212



(3) 样品本底

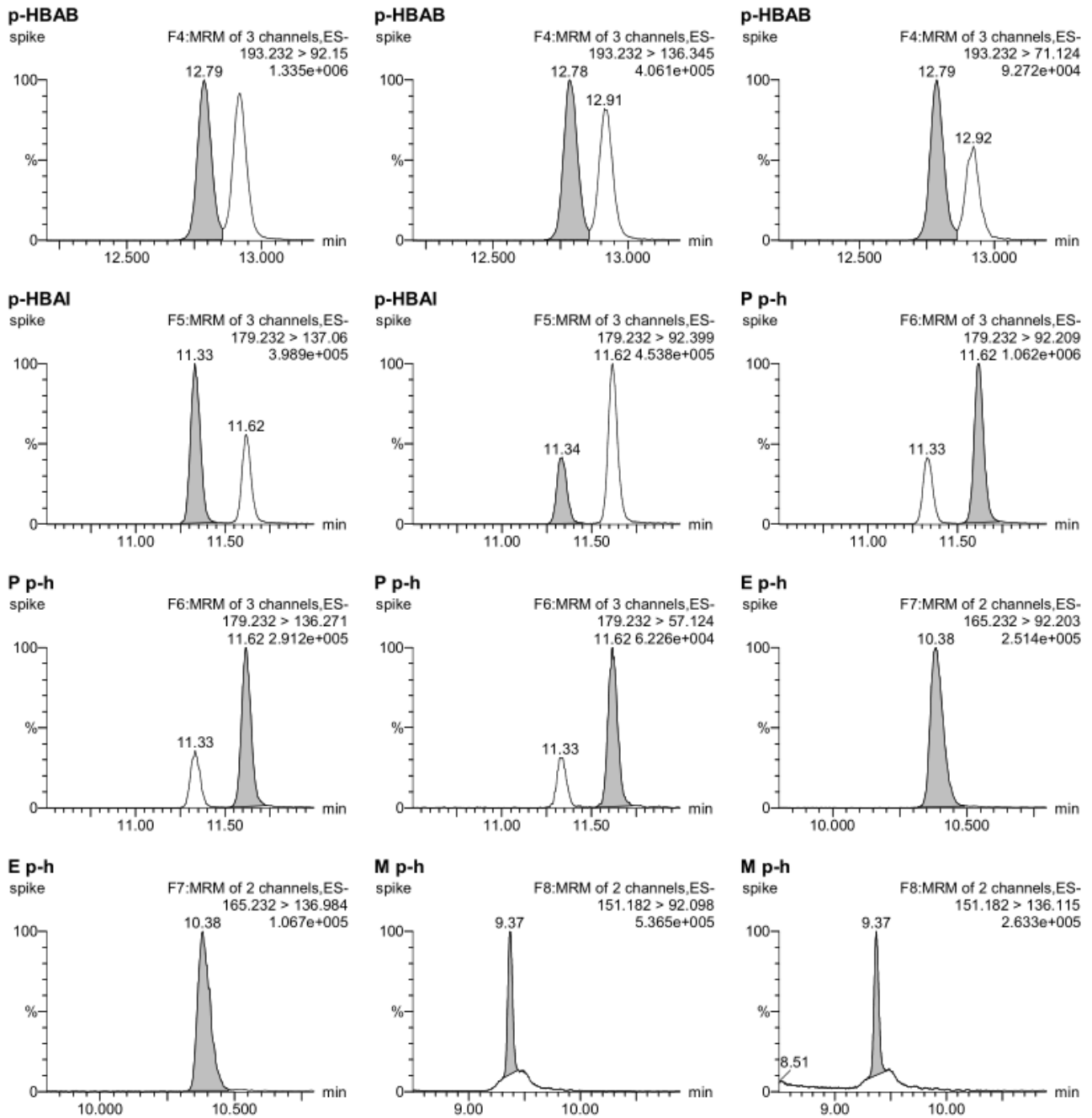


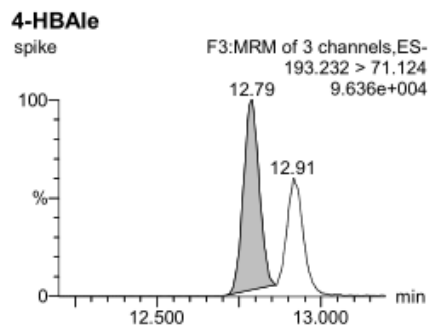
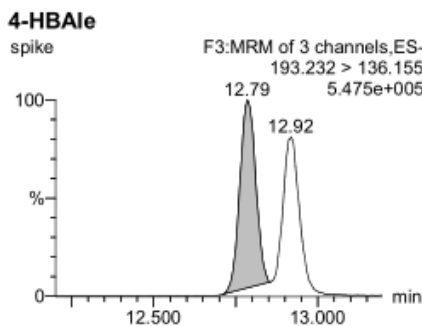
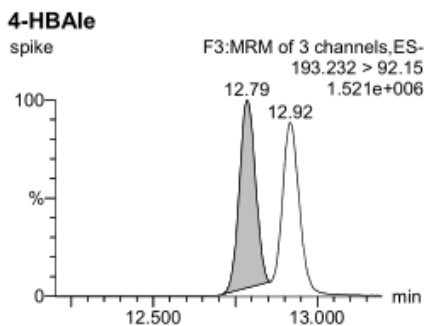
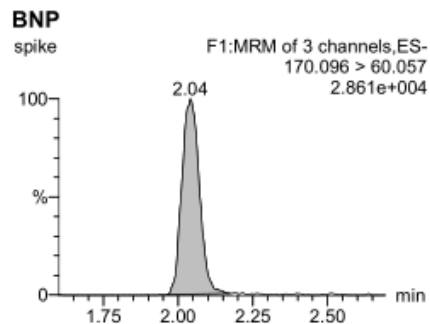
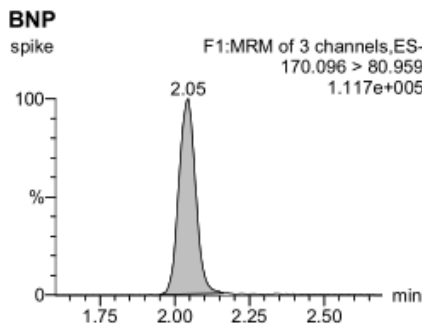
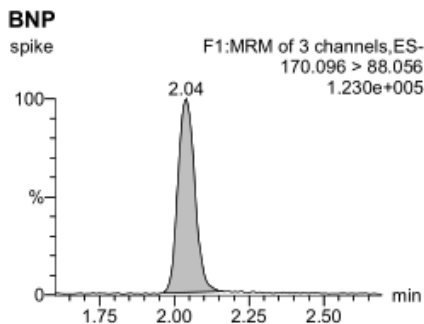


Sample Text	Name	Trace	RT	Area	Primar...	ug/mL	%Dev	1° Ratio (A...	2° Ratio (A...
sample	BNP	170.096 > 88.056	2.03	2.009	bb	0.000		1.399	
sample	4-HBAle	193.232 > 92.15	12.76	333.694	bb	0.001		6.666	38.448
sample	p-HBAB	193.232 > 92.15	12.90	409.884	bb	0.001			102.574
sample	p-HBAI	179.232 > 137.06							
sample	P p-h	179.232 > 92.209	11.53	7764.612	bb	0.015		3.604	24.378
sample	E p-h	165.232 > 92.203	10.33	58.884	bd	0.001		4.452	
sample	M p-h	151.182 > 92.098	9.29	1060.973	dd	0.002		7.560	
sample	Benzoic acid	121.16 > 77.172							
sample	DHAA	167.182 > 83.092	3.38	1.515	bb	0.000		0.713	
sample	Benzyl paraben	227.282 > 92.143	12.97	8018.063	bd	0.017		1.730	
sample	Phenyl ester	213.232 > 93.119	12.33	4615.751	bb	0.006			
sample	N-P-4-HB	207.296 > 92.201	13.95	1.786	bb	0.000		0.118	1.993

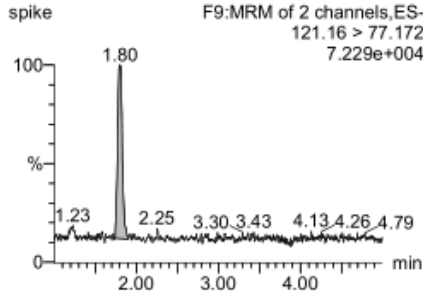


(4) 加标回收：2-溴-2-硝基丙烷-1,3-二醇等 12 种组分，加标浓度同标准品浓度。

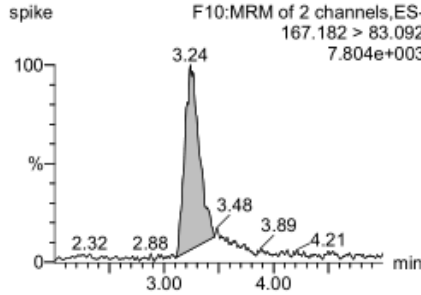




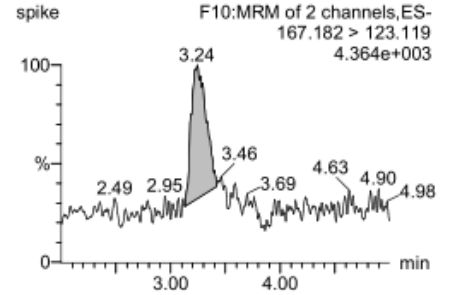
Benzoic acid



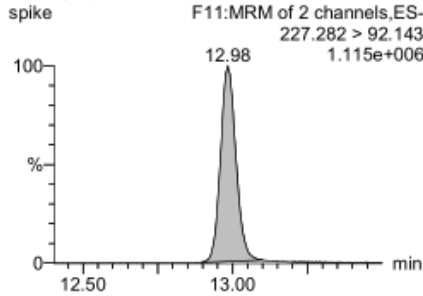
DHAA



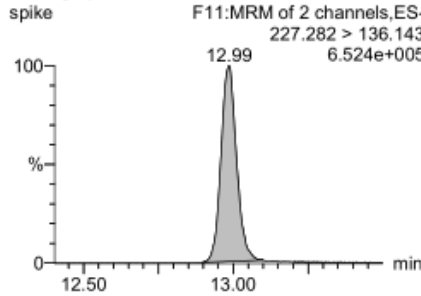
DHAA



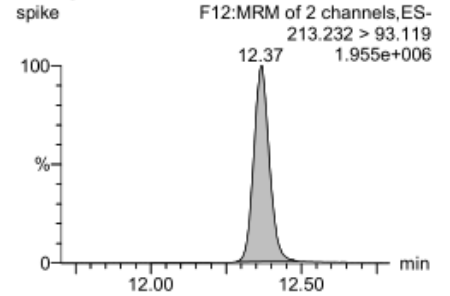
Benzyl paraben



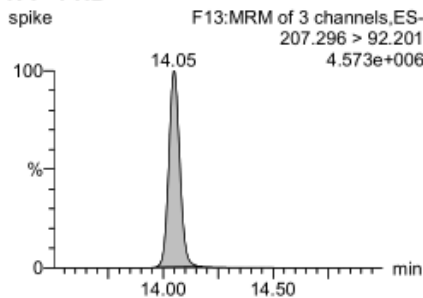
Benzyl paraben



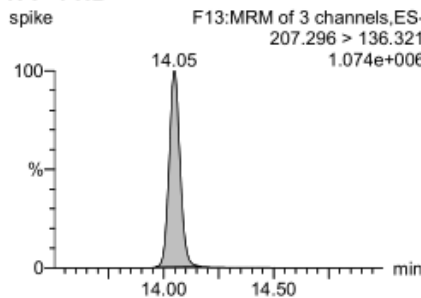
Phenyl ester



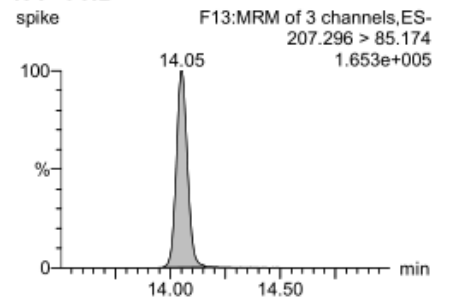
N-P-4-HB



N-P-4-HB



N-P-4-HB



Sample Text	Name	Trace	RT	Area	Primar...	ug/mL	%Dev	1° Ratio (A...	2° Ratio (A...
spike	BNP	170.096 > 88.056	2.04	7876.931	bb	0.740		1.104	4.103
spike	4-HBAle	193.232 > 92.15	12.79	82086.383	bb	0.132		2.812	15.928
spike	p-HBAB	193.232 > 92.15	12.79	78823.477	bd	0.153		3.229	14.731
spike	p-HBAI	179.232 > 137.06	11.33	24540.020	bb	0.117		2.011	
spike	P p-h	179.232 > 92.209	11.62	64636.762	bb	0.121		3.668	17.066
spike	E p-h	165.232 > 92.203	10.38	13781.151	bb	0.124		2.441	
spike	M p-h	151.182 > 92.098	9.37	23345.168	MM	0.043		2.028	
spike	Benzoic acid	121.16 > 77.172	1.80	4113.308	bb	0.231			
spike	DHAA	167.182 > 83.092	3.24	1111.575	bb	0.139		2.408	
spike	Benzyl paraben	227.282 > 92.143	12.98	65038.668	bb	0.140		1.688	
spike	Phenyl ester	213.232 > 93.119	12.37	114581.117	bb	0.142			
spike	N-P-4-HB	207.296 > 92.201	14.05	258235.844	bb	0.287		4.274	27.252



● 结论:

样品加标回收

序号	组分名称	回收率, %
1	2-溴-2-硝基丙烷-1,3-二醇	98.7
2	4-羟基苯甲酸异丁酯	88.0
3	4-羟基苯甲酸丁酯	102.0
4	4-羟基苯甲酸异丙酯	93.6
5	4-羟基苯甲酸丙酯	96.8
6	4-羟基苯甲酸乙酯	99.2
7	4-羟基苯甲酸甲酯	86.0
8	苯甲酸	92.4
9	脱氢乙酸	92.7
10	4-羟基苯甲酸苄酯	93.3
11	4-羟基苯甲酸苯酯	94.7
12	4-羟基苯甲酸戊酯	95.7

使用 Xtimate® UHPLC C18 (2.1×100mm,1.8µm)能有效分离目标化合物,回收率满足要求。

日期: 2022/8/10