

测试报告

样品信息			
样品名称	猪饲料	项目编号	SHZ-20231103-002
样品批号	/	样品性状	/
收样日期	2023/12/04	测试期间	2023/12/04-2023/12/08
标样信息			
名称	规格	数量	
苯乙醇胺 A	10mg	1 瓶	
实验要求			
验证 SPE 小柱回收率			
参考方法			
农业部 1486 号公告-1-2010			
试剂信息			
试剂名称	级别	品牌	
甲醇	HPLC	月旭	
甲酸	HPLC	月旭	
乙腈	HPLC	月旭	
氨水	AR	泰坦	
仪器信息			
仪器厂家	仪器型号		
Waters	Xevo TQ-S micro		

1. 试验过程

1.1. 色谱条件

色谱柱	Ultimate [®] UHPLC LP-C18 (2.1×150mm,1.8μm)
流动相	A: 0.1 %甲酸水; B:乙腈 ;
柱温	30°C
流速	0.2 mL/min
进样量	5 μL

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Add:江苏省南京市六合区天圣路 22 号 F 栋 4 楼

Tel:400-810-6969

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邮编：201600

邮编：321000

邮编：211500



	时间/min	A/%	B/%
参考色谱条件	0	95	5
	9	40	60
	9.1	95	5
	12	95	5

1.2. 质谱条件

● 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：1kV

名称	母离子 (m/z)	子离子 (m/z)	Cone (V)	Collision (V)	Polarity
苯乙醇胺 A	345.2	150.1	4	20	Positive
		327.2		10	

1.3. 溶液配制

1.3.1. 加标浓度：

苯乙醇胺 A：10 ng/mL。

1.3.2. 流动相配制：

0.1%甲酸水溶液：取甲酸 0.05 mL，用水定容至 100 mL，摇匀待用；

1.3.3. 溶液配制：

甲酸甲醇溶液：取甲酸 0.1 mL，用水溶解并定容到 250 mL，混匀备用；

稀释液：取 0.1%甲酸水溶液 90 mL，，用乙腈定容至 100 mL，混匀备用；

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氨化甲醇溶液：量取氨水 5 mL 用甲醇定容至 100 mL，混匀备用；

1.3.4. 标准溶液配制：

将上标准品溶液用甲醇稀释至 0.5 mg/L，供仪器调谐使用；

1.3.5 样品溶液配制：（猪饲料）

称取 2 g 饲料试样（精确至 0.001 g）于 50 mL 离心管中，准确加入甲酸甲醇溶液 20 mL，充分摇动 30 s，再置于超声水浴中超声提取 20 min，期间摇动 2 次；取出后，于离心机上 5000 r/min 离心 5 min，取上清液备用。

Welchrom® P-SCX , 60 mg/3 mL：（实验室领用）

活化：3 mL 甲醇、3 mL 水；

上样：准确移取 5 mL 样品溶液过柱，流速控制在 1 s/滴；

淋洗：3 mL 水、3 mL 甲醇，抽至近干；

洗脱：用 6 mL 氨化甲醇洗脱，收集全部洗脱液；

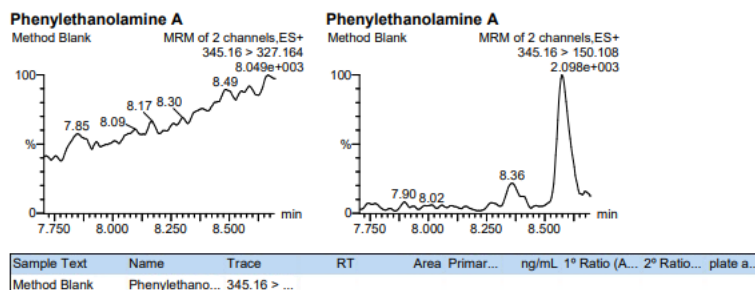
将洗脱液于 50 °C 水浴下氮吹干，用稀释液 1 mL 复溶，混匀过 0.22 μm 尼龙滤膜，待测。

1.3.5.1 样品量

方法空白(Method Blank)、基质空白 (Sample)、基质曲线 (Sample+Std) 基质加标(Spike1、Spike2)、方法加标(Method Spike)

1.3.5.2 谱图和数据

(1) 方法空白(Method Blank)溶液检测图谱



(2) 基质空白 (Sample) 溶液检测图谱

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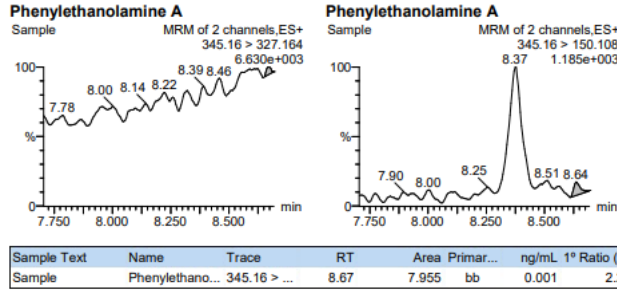
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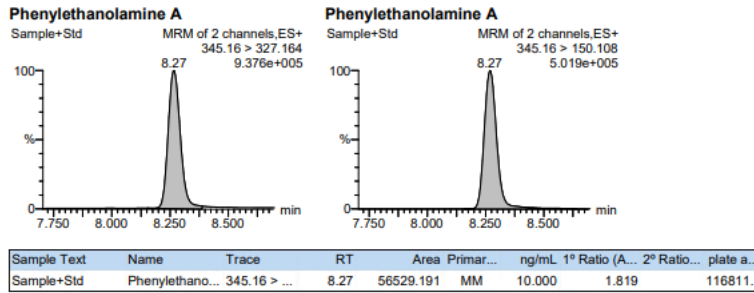
Add:江苏省南京市六合区天圣路 22 号 F 栋 4 楼

Tel:400-810-6969

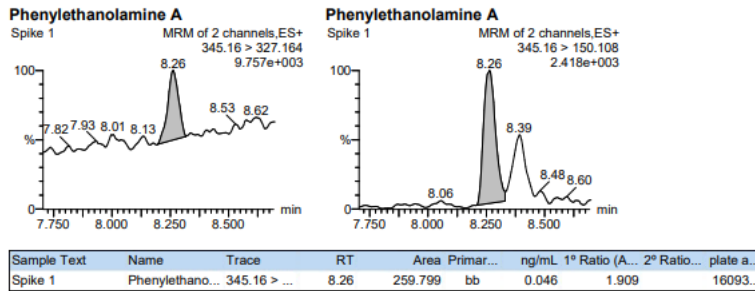




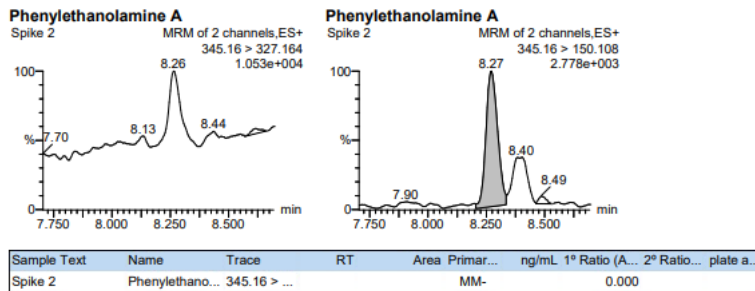
(3) 基质曲线 (Sample+Std) 溶液检测图谱



(4) 基质加标(Spike1、Spike2) 溶液检测图谱

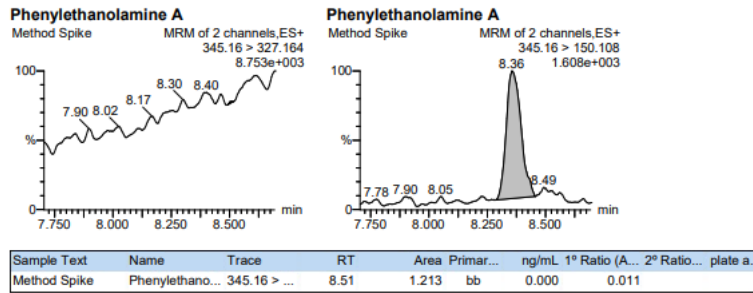


Name: 20231128025, Date: 01-Dec-2023, Time: 14:30:54, Description: Spike 2, Conditions:



(5) 方法加标(Method Spike)溶液溶液检测图谱





1.3.5.3 结果与分析

使用实验室领用 Welchrom[®] P-SCX, 60 mg/3 mL 小柱, 在 50 °C 氮吹下, 目标物苯乙醇胺 A 未检出, 查阅云盘后, 将氮吹温度降至 40 °C 可明显改善回收率, 下一步在 40 °C 水浴氮吹下重新进行实验。

1.3.6 样品溶液配制: (猪饲料)

称取 2 g 饲料试样 (精确至 0.001 g) 于 50 mL 离心管中, 准确加入甲酸甲醇溶液 20 mL, 充分摇动 30 s, 再置于超声水浴中超声提取 20 min, 期间摇动 2 次; 取出后, 于离心机上 5000 r/min 离心 5 min, 取上清液备用。

Welchrom[®] P-SCX, 60 mg/3 mL: (实验室领用)

活化: 3 mL 甲醇、3 mL 水;

上样: 准确移取 5 mL 样品溶液过柱, 流速控制在 1 s/滴;

淋洗: 3 mL 水、3 mL 甲醇, 抽至近干;

洗脱: 用 6 mL 氨化甲醇洗脱, 收集全部洗脱液;

将洗脱液于 40 °C 水浴下氮吹干, 用稀释液 1 mL 复溶, 混匀过 0.22 μm 尼龙滤膜, 待测。

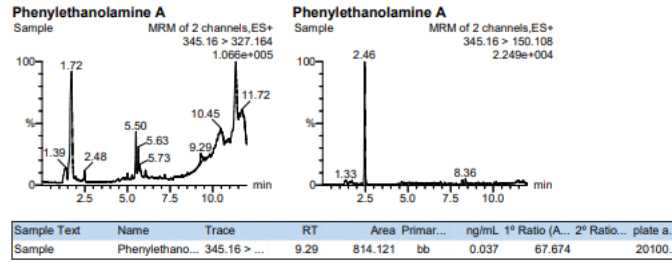
1.3.6.1 样品量

方法空白(Method Blank)、基质空白 (Sample)、基质曲线 (Sample+Std) 基质加标(Spike1、Spike2)、方法加标(Method Spike)

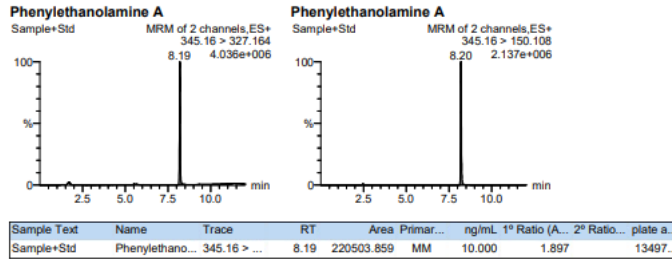
1.3.6.2 谱图和数据

(1) 基质空白 (Sample) 溶液检测图谱

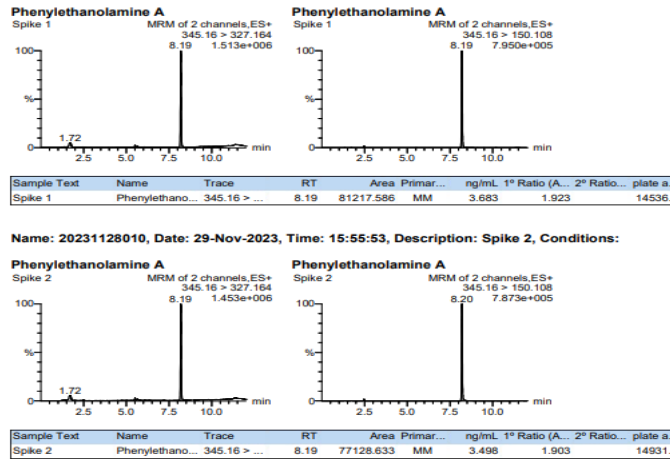




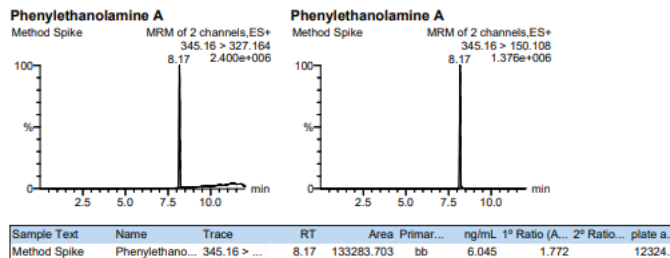
(2) 基质曲线 (Sample+Std) 溶液检测图谱



(3) 基质加标(Spike1、Spike2) 溶液检测图谱

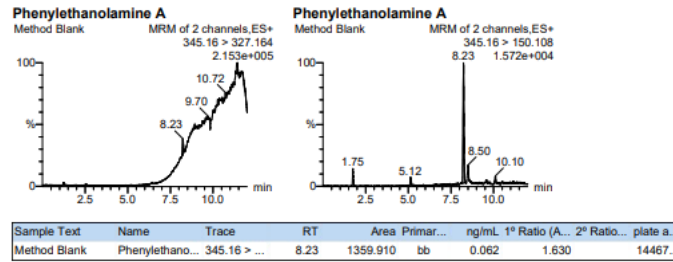


(4) 方法加标(Method Spike)溶液检测图谱



(5) 方法空白(Method Blank)溶液检测图谱





1.3.6.3 结果与分析

使用实验室现有批次 Welchrom[®] P-SCX , 60 mg/3 mL 小柱, 在 40 °C 氮吹下, 以 Sample+Std 计算目标物苯乙醇胺 A 回收率为, 36.8 %、35.0 %, 下一步在使用研发提供小柱进行分步收集实验。

1.3.7 样品溶液配制：（猪饲料）

称取 2 g 饲料试样（精确至 0.001 g）于 50 mL 离心管中, 准确加入甲酸甲醇溶液溶液 20 mL, 充分摇动 30 s, 再置于超声水浴中超声提取 20 min, 期间摇动 2 次; 取出后, 于离心机上 5000 r/min 离心 5 min, 取上清液备用。

Welchrom[®] P-SCX , 60 mg/3 mL：（研发提供小柱）

活化：3 mL 甲醇、3 mL 水；

上样：准确移取 5 mL 样品溶液过柱, 流速控制在 1 s/滴, 收集上样液；

淋洗：3 mL 水、3 mL 甲醇, 抽至近干, 收集淋洗液；

洗脱：用 6 mL 氨化甲醇洗脱, 收集全部洗脱液；

将所收集溶液于 40 °C 水浴下氮吹至近干, 用稀释液定容至 1 mL, 混匀过 0.22 μm 尼龙滤膜, 待测。

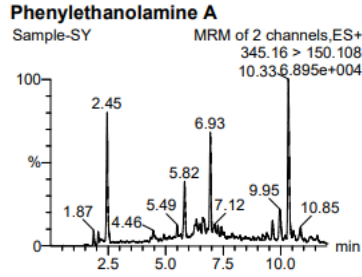
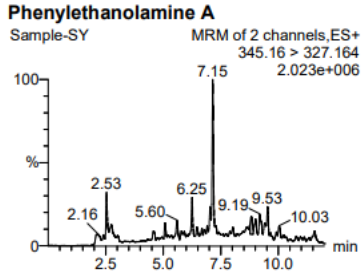
1.3.7.1 样品量

方法空白(Method Blank)、基质空白 (Sample)、基质曲线 (Sample+Std) 基质加标(Spike1、Spike2)、方法加标(Method Spike)

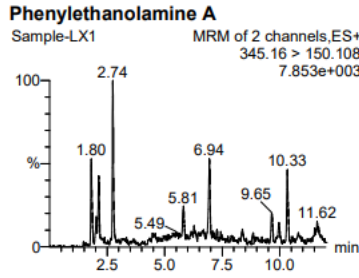
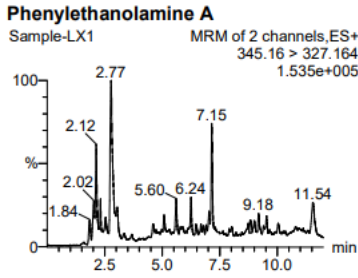
1.3.7.2 谱图和数据

(1) 基质空白 (Sample) 溶液检测图谱（上样液、淋洗液、洗脱液依次）

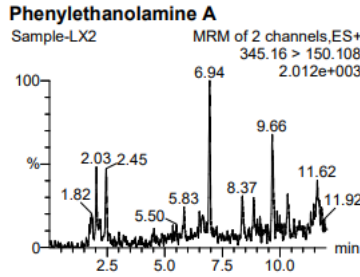
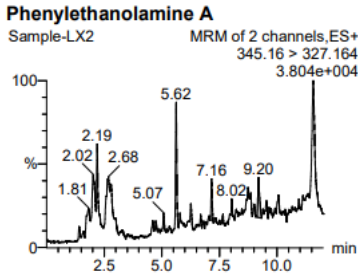




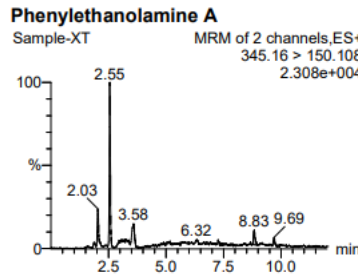
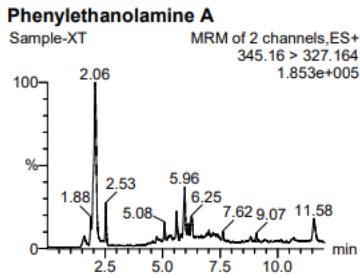
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Sample-SY	Phenylethano	345.16 >	8.82	20.60.617	dd	0.655	0.266		5.4135



Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Sample-LX1	Phenylethano...	345.16 > ...	8.82	958.368	dd	0.031	27.886		20809...



Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Sample-LX2	Phenylethano...	345.16 > ...	9.20	745.315	bb	0.024	106.246		10740...

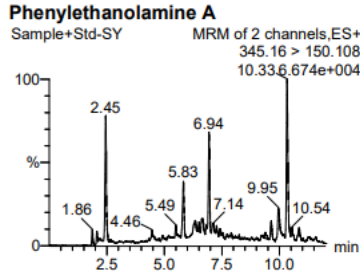
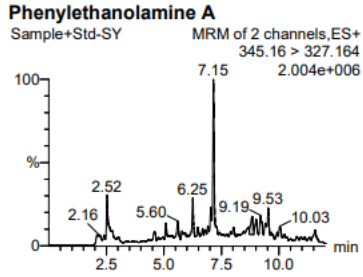


Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Sample-XT	Phenylethano...	345.16 > ...	9.07	672.739	bb	0.022	4.466		14995...

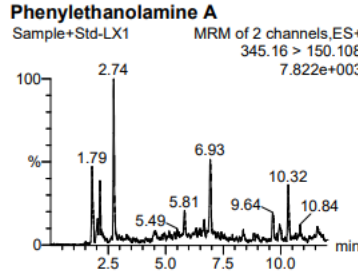
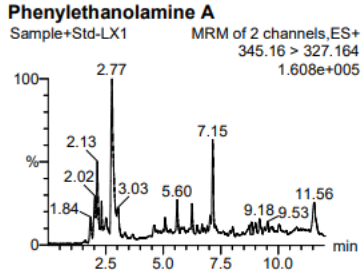
(2) 基质曲线 (Sample+Std) 溶液检测图谱 (上样液、淋洗液、洗脱液依次)



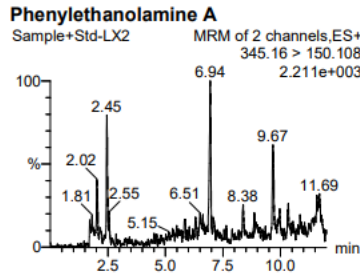
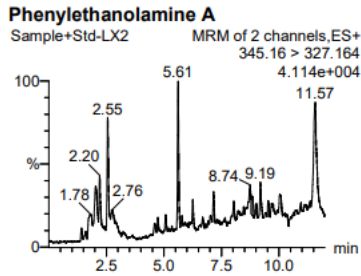
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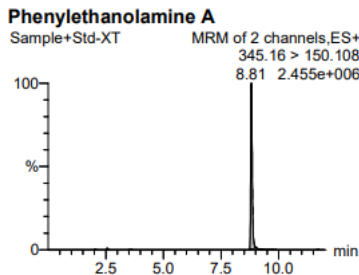
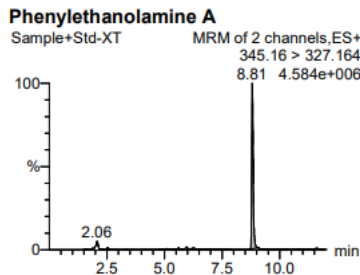
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Sample+Std-SY	Phenylethano...	345.16 > ...	8.83	25883.158	dd	0.828	137.348		59254...



Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Sample+Std-LX1	Phenylethano...	345.16 > ...	8.84	1072.847	dd	0.034	48.125		88226...



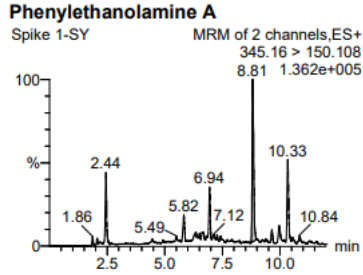
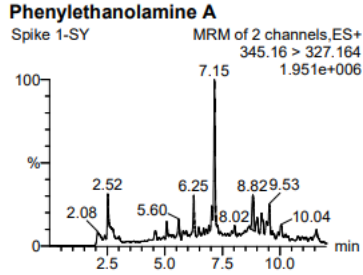
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Sample+Std-LX2	Phenylethano...	345.16 > ...	8.83	419.963	db	0.013	29.409		41508...



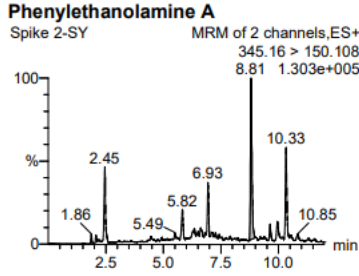
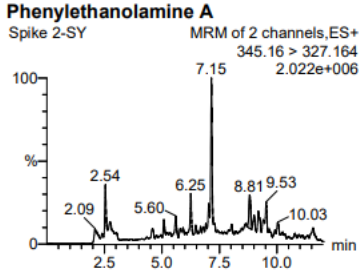
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Sample+Std-XT	Phenylethano...	345.16 > ...	8.81	312482.656	bb	10.000	1.855		12726...

(3) 基质加标(Spike1、Spike2) 溶液检测图谱（上样液、淋洗液、洗脱液依次）

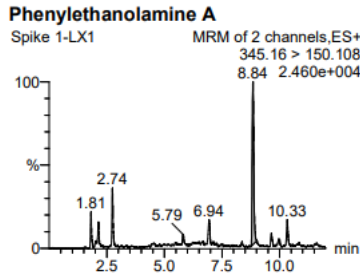
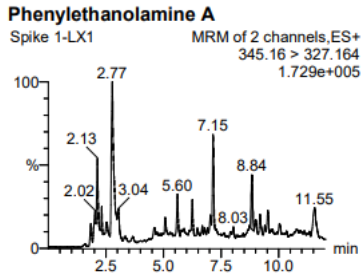




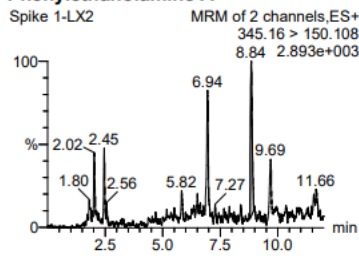
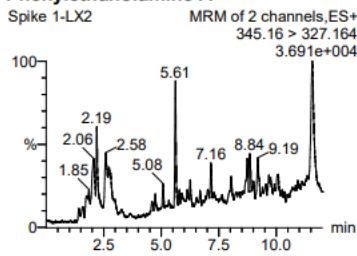
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Spike 1-SY	Phenylethano...	345.16 > ...	8.82	34402.008	bd	1.101 3.661	91751...



Sample Text	Name	Trace	RT	Area Primar...	ng/mL 1° Ratio (A...	2° Ratio...	plate a...
Spike 2-SY	Phenylethano...	345.16 > ...	8.81	34848.266	bd	1.115 3.892	82872...



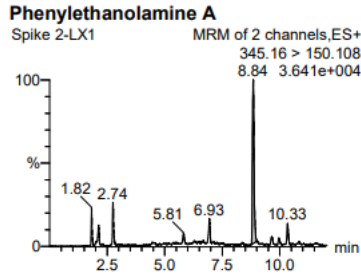
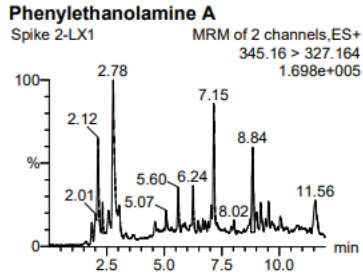
Sample Text	Name	Trace	RT	Area Primar...	ng/mL 1° Ratio (A...	2° Ratio...	plate a...
Spike 1-LX1	Phenylethano...	345.16 > ...	8.84	3978.797	bd	0.127 2.321	98508...



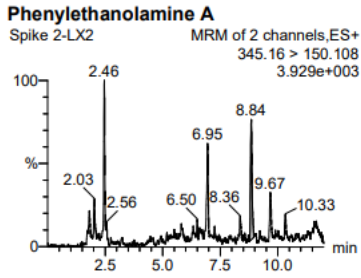
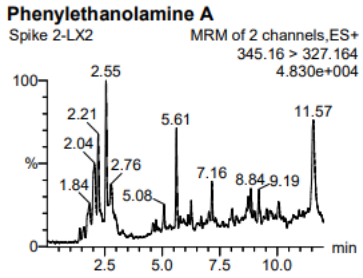
Sample Text	Name	Trace	RT	Area Primar...	ng/mL 1° Ratio (A...	2° Ratio...	plate a...
Spike 1-LX2	Phenylethano...	345.16 > ...	8.84	629.852	db	0.020 3.027	10705...



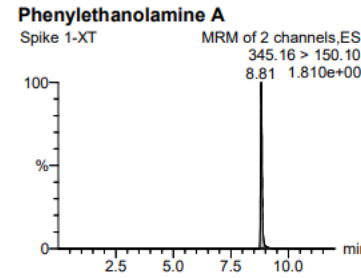
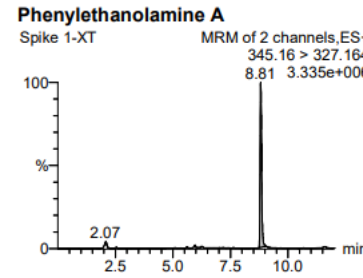
声明: 除非另有说明, 此报告结果仅对该测试样品负责。本报告未经公司许可, 不可复制。
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 Add:浙江省金华市婺城区双林南街 168 号
 Add:江苏省南京市六合区天圣路 22 号 F 栋 4 楼
 Tel:400-810-6969



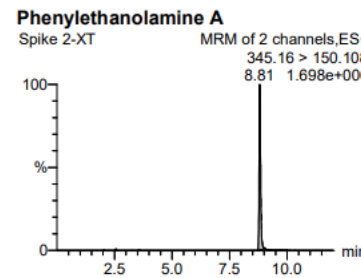
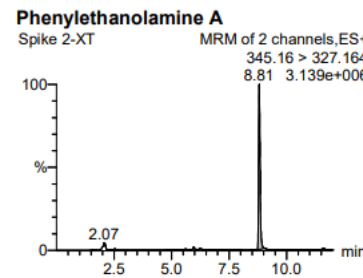
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Spike 2-LX1	Phenylethano...	345.16 > ...	8.84	7101.007	dd	0.227	2.756		97804....



Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Spike 2-LX2	Phenylethano...	345.16 > ...	8.84	434.381	bb	0.014	2.244		15187...



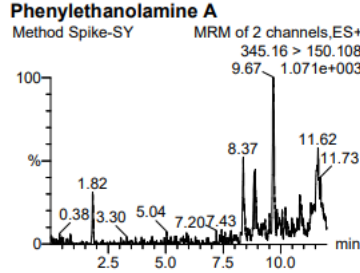
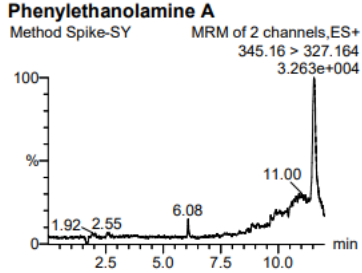
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Spike 1-XT	Phenylethano...	345.16 > ...	8.81	230323.844	bb	7.371	1.829		11510...



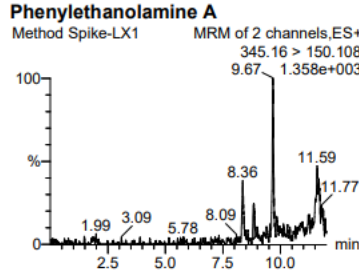
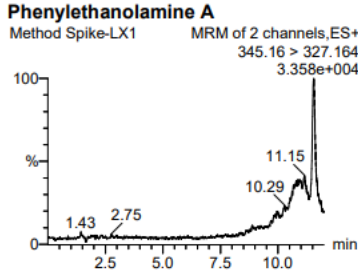
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Spike 2-XT	Phenylethano...	345.16 > ...	8.81	214840.234	bb	6.875	1.838		11847...

(4) 方法加标(Method Spike)溶液检测图谱 (上样液、淋洗液、洗脱液依次)

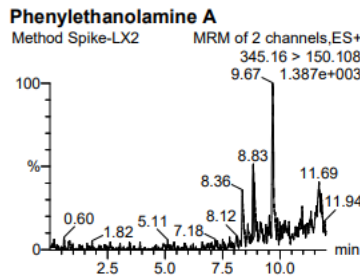
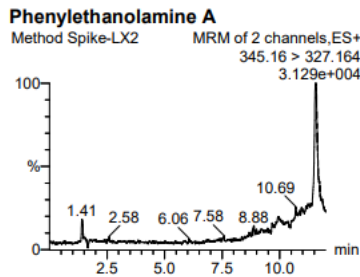




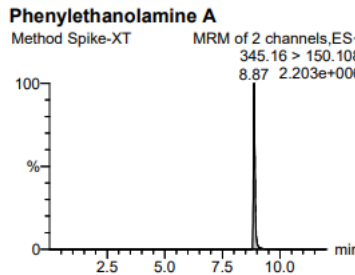
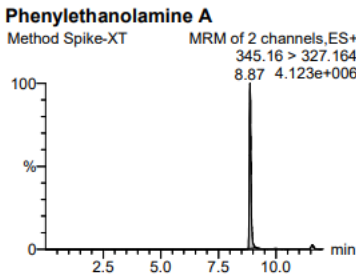
Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Method Spike-SY	Phenylethano...	345.16 > ...	8.79	19.933	bd	0.001	1.306		61614...



Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Method Spike-LX1	Phenylethano...	345.16 > ...	8.81	6.941	dd	0.000	0.387		21958...



Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Method Spike-LX2	Phenylethano...	345.16 > ...	8.83	44.419	bd	0.001	1.475		67975...

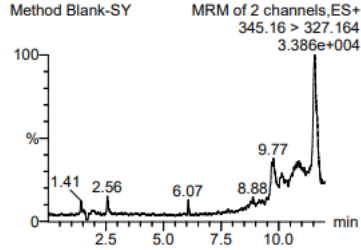


Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Method Spike-XT	Phenylethano...	345.16 > ...	8.87	308154.000	bb	9.861	1.826		10573...

(5) 方法空白(Method Blank)溶液检测图谱 (上样液、淋洗液、洗脱液依次)

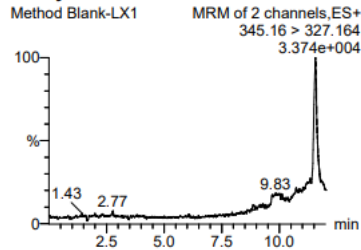
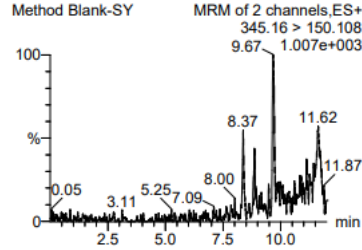


Phenylethanolamine A

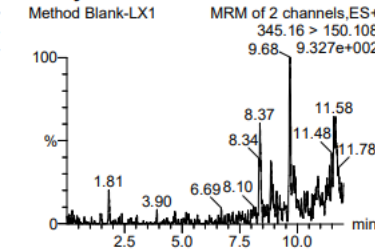


Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Method Blank-SY	Phenylethano...	345.16 > ...	8.88	56.229	bb	0.002	3.594		51714...

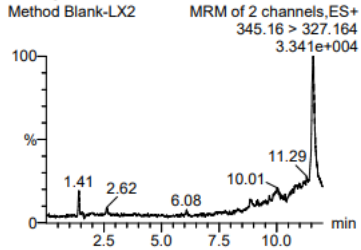
Phenylethanolamine A



Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Method Blank-LX1	Phenylethano...	345.16 > ...	8.86	93.017	bb	0.003	8.722		62766...

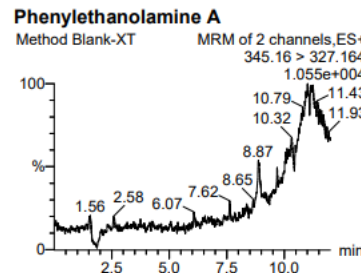
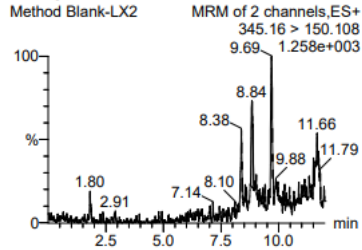


Phenylethanolamine A

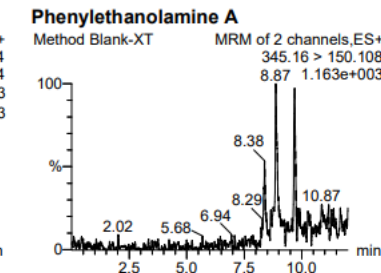


Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Method Blank-LX2	Phenylethano...	345.16 > ...	9.13	21.178	bd	0.001	0.445		14336...

Phenylethanolamine A



Sample Text	Name	Trace	RT	Area	Primar...	ng/mL	1° Ratio (A...	2° Ratio...	plate a...
Method Blank-XT	Phenylethano...	345.16 > ...							



1.3.7.3 结果与分析

使用研发提供批次 Welchrom® P-SCX, 60 mg/3 mL 小柱, 在 40 °C 氮吹下, 以 Sample+Std (基质曲线) 计算目标物苯乙醇胺 A 洗脱液回收率为, 73.7 %、68.8 %, 上样液中回收率为 11.0 %、11.2 %, 其它步骤未检出。

2. 结论

使用实验室领用 Welchrom® P-SCX, 60 mg/3 mL 小柱, 在 40 °C 氮吹下吹干, 以 Sample+Std (基质曲线) 计



算目标物苯乙醇胺 A 回收率为 36.8 %、35.0 %。

使用研发提供批次 Welchrom® P-SCX , 60 mg/3 mL 小柱, 在 40 °C氮吹下吹至近干, 以 Sample+Std (基质曲线) 计算目标物苯乙醇胺 A 洗脱液回收率为 73.7 %、68.8 %, 上样液中回收率为 11.0 %、11.2 %, 其它步骤未检出。

报告人:Chilli

审核人:Tim

日期: 2023/12/14

Welch
月旭科技

