



中华人民共和国出入境检验检疫行业标准

SN/T 2150—2008

进出口食品中涕灭砒威、唑菌胺酯、腈嘧 菌酯等 65 种农药残留量检测方法 液相色谱-质谱/质谱法

Determination of residues of aldoxycarb, pyraclostrobin, azoxystrobin 65
pesticides in foodstuffs for import and export—LC-MS/MS method

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中 华 人 民 共 和 国 发 布
国家质量监督检验检疫总局

前 言

本标准附录 A 和附录 D 为规范性附录,附录 B 和附录 C 为资料性附录。

本标准由国家认证认可监督管理委员会提出并归口。

本标准由中国检验检疫科学研究院和北京出入境检验检疫局负责起草。

本标准主要起草人:彭涛、于静、李晓娟、李刚、严矛、张朝晖、徐超一、李淑娟。

本标准系首次发布的出入境检验检疫行业标准。

进出口食品中涕灭砒威、啮菌胺酯、腈嘧 菌酯等 65 种农药残留量检测方法 液相色谱-质谱/质谱法

1 范围

本标准规定了进出口食品中 65 种农药残留量的液相色谱-质谱/质谱测定方法。

本标准适用于进出口大米、糙米、大麦、小麦和玉米中涕灭砒威、腈嘧菌酯、地散磷、丁苯草酮、联苯肼酯、噻嗪酮、萎锈灵、3-羟基克百威、烯草酮、氰霜唑、噻草酮、环丙酰菌胺、氟啶脲、枯草隆、环虫酰肼、噻虫胺、苄草隆、杀草隆、二甲嘧酚、苄氯三唑醇、除虫脲、敌草隆、乙虫清、氟虫腈、氟啶胺、啮蛄脲、氟虫脲、氟硫灭、苯硫威、啮蛄酯、啮菌胺、氟草隆、氟啶酮、呋线威、氟铃脲、咪草酸甲酯、抗倒胺、异菌脲、茚虫威、吡虫啉、异恶隆、异恶唑草酮、氟丙氧脲、甲基苯噻隆、苯噻草酮、甲氧虫酰肼、敌草胺、双苯氟脲、恶咪唑、噻嗪草酮、辛硫磷、增效醚、啮菌胺酯、吡啶特、苄草唑、戊菌隆、毒草胺、吡丙醚、精喹禾灵、螺螨酯、虫酰肼、氟苯脲、噻酰菌胺、噻虫啉和噻虫嗪残留量的检测。

2 方法提要

试样加水浸泡后用丙酮振荡提取,提取液经液液分配和固相萃取净化后,采用液相色谱-质谱/质谱检测,外标法定量。

3 试剂和材料

除非另有说明,所有试剂均为分析纯,水为去离子水或相当纯度的水。

- 3.1 甲醇:高效液相色谱级。
- 3.2 乙腈:高效液相色谱级。
- 3.3 丙酮:高效液相色谱级。
- 3.4 二氯甲烷:高效液相色谱级。
- 3.5 甲苯:高效液相色谱级。
- 3.6 甲酸:高效液相色谱级。
- 3.7 醋酸铵。
- 3.8 氯化钠。
- 3.9 无水硫酸钠:650 °C 灼烧 4 h,置于干燥器中冷却备用。
- 3.10 助滤剂:celite 545,或相当者。
- 3.11 15%氯化钠水溶液:准确称取 15 g 氯化钠溶于 100 mL 水中。
- 3.12 0.1%甲酸水溶液(含 0.5 mmol/L 醋酸铵):准确量取 1 mL 甲酸和称取 0.038 6 g 醋酸铵于 1 L 容量瓶中,用水定容至 1 L。
- 3.13 SPE 溶液:90 mL 乙腈中加入 30 mL 甲苯,混匀备用。
- 3.14 标准物质:65 种农药标准物质,纯度 $\geq 95\%$ 。标准品信息见附录 A。
- 3.15 标准储备溶液:准确称取适量标准品(精确至 0.000 1 g),用甲醇溶解,配制成浓度为 100 $\mu\text{g/mL}$ 的标准储备溶液,−18 °C 冷冻避光保存。
- 3.16 中间标准溶液:准确移取 1 mL 标准储备溶液(3.15)于 10 mL 容量瓶中,用甲醇定容至刻度,配

制成浓度为 10 $\mu\text{g/mL}$ 的中间标准溶液,4 $^{\circ}\text{C}$ 冷藏避光保存。

3.17 混合标准工作溶液:根据需要用甲醇把中间标准溶液(3.16)稀释成适合浓度的混合标准工作溶液,现用现配。

3.18 石墨化非多孔碳/酰胺丙基甲硅烷基化硅胶为填料固相萃取柱:Envi-Carb/LC-NH₂, 500 mg/500 mg,6 mL,或相当者。

3.19 微孔滤膜:0.22 μm ,有机相。

4 仪器和设备

4.1 液相色谱-质谱/质谱仪:配备电喷雾离子源(ESI)。

4.2 粉碎机。

4.3 样品筛:20 目。

4.4 分析天平:感量为 0.000 1 g 和 0.001 g。

4.5 振荡器。

4.6 减压浓缩仪。

4.7 涡旋混匀器。

5 试样制备与保存

从原始样品取出有代表性样品约 500 g,用粉碎机粉碎并使其全部通过 20 目的样品筛,混和均匀,均分成两份,分别装入洁净容器作为试样,密封,并标明标记。将试样置于 4 $^{\circ}\text{C}$ 冷藏避光保存。

在制样的操作过程中,应防止样品污染或发生残留物含量的变化。

6 测定步骤

6.1 提取

称取约 10 g 试样(精确至 0.01 g)于 300 mL 锥形瓶中。加入 10 mL 水,静置 30 min 后,再加入 40 mL 丙酮,振荡提取 30 min。将试样及提取液转移至抽滤漏斗上[已加入适量助滤剂(3.10)],减压抽滤,收集滤液于 100 mL 梨形瓶中。再用 3 $\times$ 5 mL 丙酮洗涤锥形瓶及试样残渣,合并滤液,并于 40 $^{\circ}\text{C}$ 减压浓缩至约 10 mL。将溶液转移至 125 mL 分液漏斗中,依次加入 30 mL 氯化钠水溶液(3.11)和 30 mL 二氯甲烷,振荡 10 min 后,静置 20 min,取二氯甲烷层。再加入 30 mL 二氯甲烷于分液漏斗中,液液分配后合并二氯甲烷层。二氯甲烷溶液经无水硫酸钠脱水后,在 40 $^{\circ}\text{C}$ 下减压浓缩至近干,氮气吹干后,用 2 mL SPE 溶液(3.13)溶解,待净化。

6.2 净化

固相萃取柱(3.18)用 10 mL SPE 溶液(3.13)预淋洗后,转入样品提取液(6.1),收集流出液。再用 30 mL SPE 溶液(3.13)洗涤固相萃取柱,合并流出液。整个固相萃取净化过程控制流速不超过 2 mL/min。流出液于 40 $^{\circ}\text{C}$ 下减压浓缩至近干,氮气吹干。残留物先用 0.4 mL 乙腈溶解再用 0.1%甲酸水溶液(3.12)定容至 1 mL,涡旋混匀后,过 0.22 μm 微孔滤膜,供仪器检测。

6.3 混合基质标准溶液的制备

称取 5 份约 10 g 空白试样(精确至 0.01 g)于 300 mL 锥形瓶中,按照标准曲线最终定容浓度分别加入中间标准溶液(3.16)或混合标准工作溶液(3.17),余下操作同 6.1 和 6.2。

6.4 测定

6.4.1 液相色谱条件

6.4.1.1 色谱柱:CAPCELL PAK C₁₈, 2.0 mm $\times$ 150 mm(内径),5 μm ,或相当者。

6.4.1.2 流动相:A. 乙腈,B. 0.1%甲酸水溶液(3.12)。梯度洗脱条件见表1和表2。

表1 ESI+模式液相色谱洗脱条件

时间/min	A/%	B/%
0~2	10	90
2~10	10~55	90~45
10~30	55~90	45~10
30~30.1	90~10	10~90
30.1~35	10	90

表2 ESI-模式液相色谱洗脱条件

时间/min	A/%	B/%
0~4	10~90	90~10
4~6	90	10
6~6.1	90~10	10~90
6.1~10	10	90

6.4.1.3 柱温:40℃。

6.4.1.4 流速:0.2 mL/min。

6.4.1.5 进样量:10 μL。

6.4.2 质谱条件

参见附录B表B.1和表B.2。其中氮气和氩气纯度均大于等于99.999%。

6.4.3 色谱测定

根据样液中待测物的含量,选定浓度相近的基质标准溶液,待测样液中农药的响应值应在仪器检测的线性范围内。对基质标准溶液及样液(6.2)等体积参插进样测定。在上述仪器条件下,65种农药的保留时间参见附录B表B.2。液相色谱-质谱/质谱色谱图参见附录C图C.1和C.2。

6.4.4 定性测定

按照上述条件测定样品和基质标准溶液,如果检测的质量色谱峰保留时间与基质标准溶液一致,允许偏差小于±2.5%;定性离子对的相对丰度与浓度相当基质标准溶液的相对丰度一致,相对丰度偏差不超过表3的规定,则可判断样品中存在相应的被测物。

表3 定性测定时相对离子丰度的最大允许偏差

相对离子丰度/%	>50	>20~50	>10~20	≤10
允许的相对偏差/%	±20	±25	±30	±50

6.5 空白试验

除不加试样外,按上述测定步骤进行。

7 结果计算和表达

试样中的残留含量用LC-MS/MS的数据处理机或按式(1)计算(计算结果需将空白值扣除):

$$X_i = \frac{A \times c \times V}{A_s \times m \times 1\,000} \dots\dots\dots (1)$$

式中：

X_i ——试样中残留药物含量，单位为毫克每千克(mg/kg)；

A ——样液中药物的峰面积；

A_s ——基质标准溶液中药物的峰面积；

c ——基质标准溶液中药物的浓度，单位为纳克每毫升(ng/mL)；

V ——样液最终定容体积，单位为毫升(mL)；

m ——最终样液所代表的试样质量，单位为克(g)。

8 测定低限和回收率

测定低限和回收率见附录 D。

附 录 A
(规范性附录)
标准物质信息

表 A.1 65 种农药的基本信息

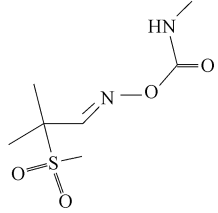
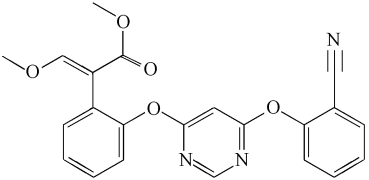
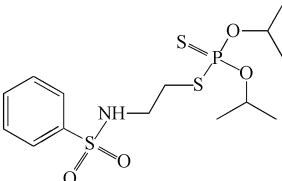
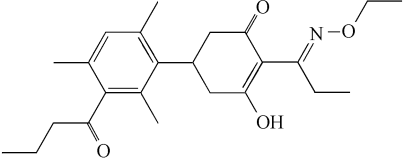
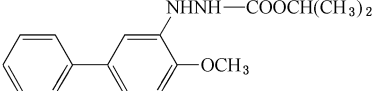
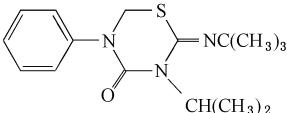
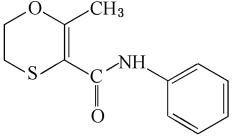
英文名称	中文名称	CAS 号	分子式	相对分子质量	结构式
Aldoxycarb	涕灭氧威	1646-88-4	$C_7H_{14}N_2O_4S$	222.258 6	
Azoxystrobin	腈嘧菌酯	131860-33-8	$C_{22}H_{17}N_3O_5$	403.393 4	
Bensulide	地散磷	741-58-2	$C_{14}H_{24}NO_4PS_3$	397.501 66	
Butoxydim	丁苯草酮	138164-12-2	$C_{24}H_{33}NO_4$	399.53	
Bifenazate	联苯肼酯	149877-41-8	$C_{17}H_{20}N_2O_3$	300.36	
Buprofezin	噻嗪酮	69327-76-0	$C_{16}H_{23}N_3OS$	305.26	
Carboxin	萎锈灵	5234-68-4	$C_{12}H_{13}NO_2S$	235.3	

表 A.1 (续)

英文名称	中文名称	CAS 号	分子式	相对分子质量	结构式
Carbofuran-3OH	3-羟基克百威	16655-82-6	$C_{12}H_{15}NO_4$	237.26	
Clethodim	烯草酮	99129-21-2	$C_{17}H_{26}ClNO_3S$	359.910 3	
Cyazofamid	氰霜唑	120116-88-3	$C_{13}H_{13}ClN_4O_2S$	324.78	
Cycloxydim	噻草酮	101205-02-1	$C_{17}H_{27}NO_3S$	325.465 2	
Carpropamid	环丙酰菌胺	104030-54-8	$C_{15}H_{18}Cl_3NO$	334.67	
Chlorfluazuron	氟啶脲	71422-67-8	$C_{20}H_9Cl_3F_5N_3O_3$	540.660 4	
Chloroxuron	枯草隆	1982-47-4	$C_{15}H_{15}ClN_2O_2$	290.748 7	

表 A.1 (续)

英文名称	中文名称	CAS 号	分子式	相对分子质量	结构式
Chromafenozide	环虫酰肼	143807-66-3	$C_{24}H_{30}N_2O_3$	394.51	
Clothianidin	噻虫胺	205510-53-8	$C_6H_8ClN_5O_2S$	249.67	
Cumyluron	苄草隆	99485-76-4	$C_{17}H_{19}ClN_2O$	302.802 9	
Daimuron	杀草隆	42609-52-9	$C_{17}H_{20}N_2O$	268.357 8	
Dimethirimol	二甲嘧酚	5221-53-4	$C_{11}H_{19}N_3O$	209.290 6	
Diclobutrazol	苄氯三唑醇	75736-33-3	$C_{15}H_{19}Cl_2N_3O$	328.240 6	
Diflubenzuron	除虫脲	35367-38-5	$C_{14}H_9ClF_2N_2O_2$	310.687 1	
Diuron	敌草隆	330-54-1	$C_9H_{10}Cl_2N_2O$	233.096 8	

表 A.1 (续)

英文名称	中文名称	CAS 号	分子式	相对分子质量	结构式
Ethiprole	乙虫清	181587-01-9	$C_{13}H_9Cl_2F_3N_4OS$	397.2	
Fipronil	氟虫腈	120068-37-3	$C_{12}H_4Cl_2F_6N_4OS$	437.146 2	
Fluazinam	氟啶胺	79622-59-6	$C_{13}H_4Cl_2F_6N_4O_4$	465.095 4	
Fluazuron	啶蝉脲	86811-58-7	$C_{20}H_{10}Cl_2F_5N_3O_3$	506.215 3	
Flufenoxuron	氟虫脲	101463-69-8	$C_{21}H_{11}ClF_6N_2O_3$	488.772 9	
Flusulfamide	氟硫灭	106917-52-6	$C_{13}H_7Cl_2F_3N_2O_4S$	415.170 5	
Fenothiocarb	苯硫威	62850-32-2	$C_{13}H_{19}NO_2S$	253.358 6	

表 A.1 (续)

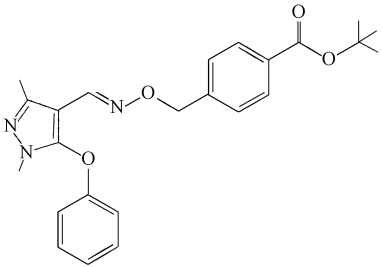
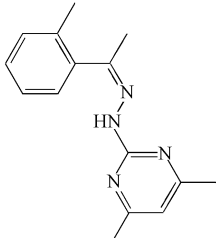
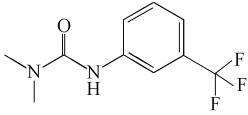
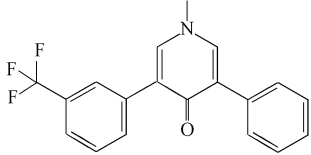
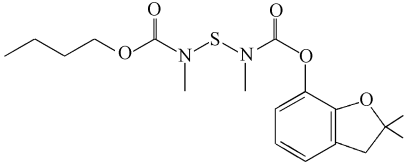
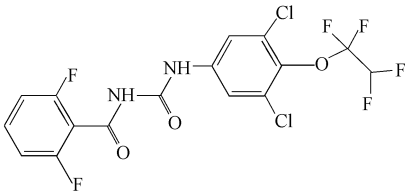
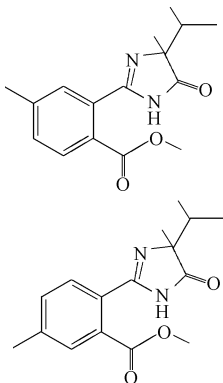
英文名称	中文名称	CAS 号	分子式	相对分子质量	结构式
Fenpyroximate	唑磷酯	134098-61-6	$C_{24}H_{27}N_3O_4$	421.495	
Ferimzone	嘧菌腈	89269-64-7	$C_{15}H_{18}N_4$	254.334	
Fluometuron	氟草隆	2164-17-2	$C_{10}H_{11}F_3N_2O$	232.204 9	
Fluridone	氟啶酮	59756-60-4	$C_{19}H_{14}F_3NO$	329.320 9	
Furathiocarb	呋线威	65907-30-4	$C_{18}H_{26}N_2O_5S$	382.473 8	
Hexaflumuron	氟铃脲	86479-06-3	$C_{16}H_8Cl_2F_6N_2O_3$	461.147 2	
Imazamethabenz-methyl	咪草酸甲酯	81405-85-8	$C_{16}H_{20}N_2O_3$	576.7 (288.35)	

表 A.1 (续)

英文名称	中文名称	CAS 号	分子式	相对分子质量	结构式
Inabenfide	抗倒胺	82211-24-3	$C_{19}H_{15}ClN_2O_2$	338.792 7	
Iprodione	异菌脲	36734-19-7	$C_{13}H_{13}Cl_2N_3O_3$	330.17	
Indoxacarb	茚虫威	144171-61-9	$C_{22}H_{17}ClF_3N_3O_7$	527.83	
Imidacloprid	吡虫啉	138261-41-3	$C_9H_{10}ClN_5O_2$	255.663 3	
Isouron	异恶隆	55861-78-4	$C_{10}H_{17}N_3O_2$	211.263 2	
Isoxaflutole	异恶唑草酮	141112-29-0	$C_{15}H_{12}F_3NO_4S$	359.32	
Lufenuron	氟丙氧脲	103055-07-8	$C_{17}H_8Cl_2F_8N_2O_3$	511.155	
Methabenzthiazuron	甲基苯噻隆	18691-97-9	$C_{10}H_{11}N_3OS$	221.276 4	
Metamitron	苯噻草酮	41394-05-2	$C_{10}H_{10}N_4O$	202.21	

表 A.1 (续)

英文名称	中文名称	CAS 号	分子式	相对分子质量	结构式
Methoxyfenozide	甲氧虫酰肼	161050-58-4	$C_{22}H_{28}N_2O_3$	368.474 8	
Napropamide	敌草胺	15299-99-7	$C_{17}H_{21}NO_2$	271.39	
Novaluron	双苯氟脲	116714-46-6	$C_{17}H_9ClF_8N_2O_4$	492.709 3	
Oxpoconazole	恶咪唑	134074-64-9	$C_{19}H_{24}ClN_3O_2$	361.87	
Oxaziclomefone	噁嗪草酮	153197-14-9	$C_{20}H_{19}Cl_2NO_2$	376.28	
Phoxim	辛硫磷	14816-18-3	$C_{12}H_{15}N_2O_3PS$	298.295 86	
Piperonylbutoxide	增效醚	51-03-6	$C_{19}H_{30}O_5$	338.443	
Pyraclostrobin	唑菌胺酯	175013-18-0	$C_{19}H_{18}ClN_3O_4$	387.82	

表 A.1 (续)

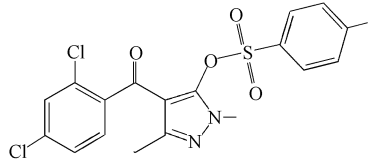
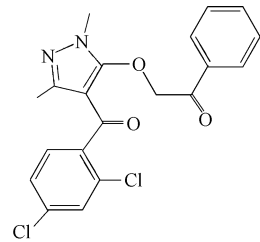
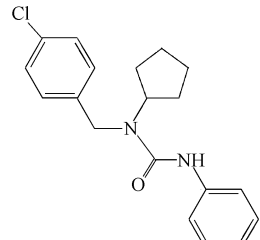
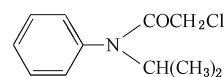
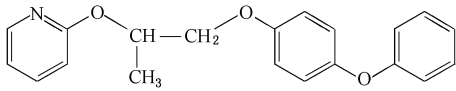
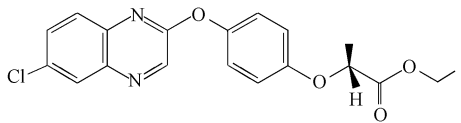
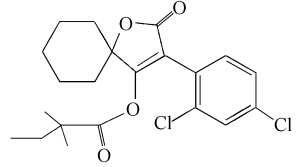
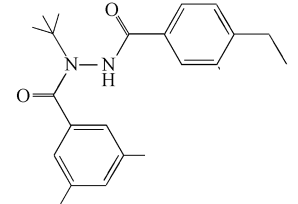
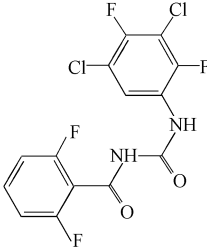
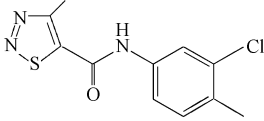
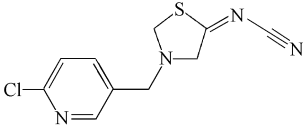
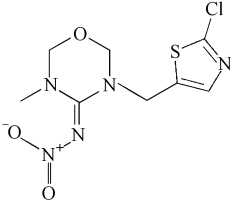
英文名称	中文名称	CAS 号	分子式	相对分子质量	结构式
Pyrazolynate	吡唑特	58011-68-0	$C_{19}H_{16}Cl_2N_2O_4S$	439.312 4	
Pyrazoxyfen	苜草唑	71561-11-0	$C_{20}H_{16}Cl_2N_2O_3$	403.264	
Pencycuron	戊菌隆	66063-05-6	$C_{19}H_{21}ClN_2O$	328.840 7	
Propachlor	毒草胺	1918-16-7	$C_{11}H_{14}ClNO$	211.69	
Pyriproxyfen	吡丙醚	95737-68-1	$C_{20}H_{19}NO_3$	321.4	
Quizalofop-ethyl	精喹禾灵	100646-51-3	$C_{19}H_{17}ClN_2O_4$	372.8	
Spirodiclofen	螺螨酯	148477-71-8	$C_{21}H_{24}Cl_2O_4$	411.33	
Tebufozide	虫酰肼	112410-23-8	$C_{22}H_{28}N_2O_2$	352.475 4	

表 A.1 (续)

英文名称	中文名称	CAS 号	分子式	相对分子质量	结构式
Teflubenzuron	氟苯脲	83121-18-0	$C_{14}H_6Cl_2F_4$ N_2O_2	381.113 2	
Thiadinil	噻酰菌胺	223580-51-6	$C_{11}H_{10}ClN_3OS$	267.7	
Thiacloprid	噻虫啉	111988-49-9	$C_{10}H_9ClN_4S$	252.72	
Thiamethoxam	噻虫嗪	153719-23-4	$C_8H_{10}ClN_5O_3S$	291.71	

附 录 B¹⁾
(资料性附录)
质谱条件

B.1 质谱条件见表 B.1。

表 B.1 质谱条件

电离方式	ESI+	ESI—
毛细管电压	3.0 kV	2.8 kV
源温度	120 °C	
去溶剂温度	350 °C	
锥孔气流	氮气,100 L/h	
去溶剂气流	氮气,600 L/h	
碰撞气压	氩气, 2.40×10^{-6} Pa	
监测模式	多反应监测	

B.2 多反应监测条件见表 B.2。

表 B.2 多反应监测条件

序号	英文名称	中文名称	电离方式 ESI	母离子 m/z	子离子 m/z	驻留时 间/s	锥孔电 压/V	碰撞能 量/eV	保留时 间/min
1	Carpropamid	加普胺	+	335.9	102.7 ^a	0.1	35	40	17.50
					197.8	0.1	35	12	
2	Chlorfluazuron	氟啶脲	+	541.9	385 ^a	0.1	30	22	23.89
					157.7	0.1	30	20	
3	Chloroxuron	枯草隆	+	291	110.6 ^a	0.1	30	40	13.90
					217.9	0.1	30	22	
4	Chromefenozide	环虫酰肼	+	395.3	174.9 ^a	0.1	20	18	15.63
					339.1	0.1	20	8	
5	Clothianidin	噻虫胺	+	249.9	168.8 ^a	0.1	22	10	7.89
					131.7	0.1	22	15	
6	Cumyluron	苄草隆	+	303.2	184.8 ^a	0.1	30	13	14.16
					124.6	0.1	30	30	
7	Diclobutrazol	苄氯三唑醇	+	328	69.9 ^a	0.1	30	20	14.93
					158.6	0.1	30	35	
8	Diflubenzuron	除虫脲	+	311	157.7 ^a	0.1	25	13	15.20
					140.7	0.1	25	30	

1) 附录 B 所列参数是在 Waters Quattro Premier 质谱仪上完成的,此处列出试验用仪器型号仅是为了提供参考,并不涉及商业目的,鼓励标准使用者尝试采用不同厂家或型号的仪器。

表 B.2 (续)

序号	英文名称	中文名称	电离方式 ESI	母离子 m/z	子离子 m/z	驻留时 间/s	锥孔电 压/V	碰撞能 量/eV	保留时 间/min
9	Diuron	敌草隆	+	232.9	71.7 ^a	0.1	30	13	11.95
					159.7	0.1	30	25	
10	Ethiprole	乙虫清	+	397	350.8 ^a	0.1	28	18	13.27
					254.8	0.1	28	33	
11	Fipronil	氟虫腈	—	435	329.8 ^a	0.1	30	15	5.92
					249.8	0.1	30	25	
12	Fluazinam	氟啶胺	—	463	415.7 ^a	0.1	30	18	6.59
					397.8	0.1	30	20	
13	Fluazuron	啶蝼脲	+	506	157.8 ^a	0.1	30	20	21.67
					348.9	0.1	30	20	
14	Flufenoxuron	氟虫脲	+	489.1	157.8 ^a	0.1	20	18	22.78
					140.8	0.1	20	40	
15	Flusulfamide	氟硫灭	—	413	170.9 ^a	0.1	25	38	6.56
					348.9	0.1	25	28	
16	Hexaflumuron	氟铃脲	—	459.1	438.9 ^a	0.1	30	12	6.17
					275.9	0.1	30	20	
17	Imazamethabenz- methyl	咪草酸甲酯	+	289.1	143.7 ^a	0.1	30	32	9.36
					160.8	0.1	30	25	
18	Inabenfide	抗倒胺	+	339.0	321 ^a	0.1	25	16	12.22
					79.7	0.1	25	27	
19	Iprodione	异菌脲	+	330.0	244.9 ^a	0.1	25	15	15.46
					287.9	0.1	25	13	
20	Lufenuron	氟丙氧脲	—	509	326 ^a	0.1	22	20	6.53
					174.7	0.1	22	35	
21	Methoxyfenozide	甲氧虫酰肼	+	369.2	148.7 ^a	0.1	20	15	15.17
					313	0.1	20	10	
22	Novaluron	双苯氟脲	—	491.1	471.1 ^a	0.1	30	14	6.24
					304.9	0.1	30	14	
23	Oxpoconazole	恶咪唑	+	362.3	178.9 ^a	0.1	13	24	14.33
					196.9	0.1	13	16	
24	Pencycuron	戊菌隆	+	329.1	124.6 ^a	0.1	30	20	18.95
					217.9	0.1	30	15	
25	Tebufenozide	虫酰肼	+	353.3	132.7 ^a	0.1	15	22	16.53
					297.1	0.1	15	8	

表 B.2 (续)

序号	英文名称	中文名称	电离方式 ESI	母离子 m/z	子离子 m/z	驻留时 间/s	锥孔电 压/V	碰撞能 量/eV	保留时 间/min
26	Teflubenzuron	氟苯脲	—	378.9	338.8 ^a	0.1	25	12	6.43
					195.7	0.1	25	28	
27	Thiadinil	噻啉菌胺	—	266.1	70.7 ^a	0.1	30	20	5.72
					237.9	0.1	30	10	
28	Aldoxycarb	涕灭氧威	+	222.9	85.6 ^a	0.1	22	15	5.88
					147.7	0.1	22	8	
29	Azoxystrobin	腈嘧菌酯	+	404	372 ^a	0.1	22	15	14.12
					329	0.1	22	30	
30	Bensulide	地散磷	+	398	157.8 ^a	0.1	20	25	17.7
					140.8	0.1	20	33	
31	Butoxydim	丁氧环酮	+	400.1	354.1 ^a	0.1	30	15	22.39
					137.8	0.1	30	26	
32	Carbofuran-3OH	3-羟基克百威	+	238	162.9 ^a	0.1	20	15	8.0
					220.1	0.1	20	6	
33	Clethodim	烯草酮	+	360.0	163.8 ^a	0.1	25	12	20.92
					268	0.1	25	20	
34	Cyazofamid	氰霜唑	+	325	107.8 ^a	0.1	20	15	16.88
					261.1	0.1	20	10	
35	Cycloxydim	噻草酮	+	326.1	280 ^a	0.1	25	12	20.32
					179.8	0.1	25	20	
36	Daimuron	杀草隆	+	269.1	150.9 ^a	0.1	20	12	14.47
					90.8	0.1	20	35	
37	Dimethirimol	二甲嘧酚	+	210.0	70.7 ^a	0.1	35	30	7.10
					139.8	0.1	35	20	
38	Fenothiocarb	苯硫威	+	254	71.7 ^a	0.1	20	15	16.16
					159.8	0.1	20	10	
39	Fenpyroximate	唑螨酯	+	422.1	366.1 ^a	0.1	25	15	23.63
					137.8	0.1	25	30	
40	Ferimzone	嘧菌腈	+	255.1	90.8 ^a	0.1	30	32	9.49
					131.9	0.1	30	22	
41	Fluometuron	氟草隆	+	233.1	71.9 ^a	0.1	30	25	11.57
					160	0.1	30	25	
42	Fluridone	氟啶酮	+	330.1	309.2 ^a	0.1	40	35	13.33
					259.2	0.1	40	45	

表 B.2 (续)

序号	英文名称	中文名称	电离方式 ESI	母离子 m/z	子离子 m/z	驻留时 间/s	锥孔电 压/V	碰撞能 量/eV	保留时 间/min
43	Furathiocarb	呋线威	+	383.1	195 ^a	0.1	30	20	21.45
					252.1	0.1	30	12	
44	Imidacloprid	吡虫啉	+	255.9	174.8 ^a	0.1	20	18	8.21
					208.9	0.1	20	15	
45	Isouron	异恶隆	+	212.1	71.9 ^a	0.1	30	25	10.3
					160	0.1	30	25	
46	Isoxaflutole	异恶唑草酮	+	360.1	251.1 ^a	0.1	25	15	14.04
					220	0.1	25	30	
47	Methabenzthiazuron	甲基苯噻隆	+	222	165 ^a	0.1	22	15	11.24
					150	0.1	22	28	
48	Oxaziclomefone	噁嗪草酮	+	376	189.9 ^a	0.1	20	30	21.69
					160.8	0.1	20	20	
49	Phoxim	辛硫磷	+	298.9	76.7 ^a	0.1	20	12	19.17
					96.6	0.1	20	35	
50	Piperonylbutoxide	增效醚	+	356.2	177 ^a	0.1	20	12	21.32
					119	0.1	20	25	
51	Pyraclostrobin	唑菌胺酯	+	388	193.9 ^a	0.1	20	30	18.39
					162.8	0.1	20	20	
52	Pyrazolynate	吡唑特	+	438.9	90.6 ^a	0.1	30	30	18.95
					172.7	0.1	30	20	
53	Pyrazoxyfen	苋草唑	+	403	90.8 ^a	0.1	30	35	16.69
					104.7	0.1	30	22	
54	Spirodiclofen	螺螨酯	+	411	313 ^a	0.1	22	10	26.1
					70.8	0.1	22	15	
55	Thiacloprid	噻虫啉	+	252.9	125.8 ^a	0.1	30	20	9.58
					89.7	0.1	30	35	
56	Thiamethoxam	噻虫嗪	+	291.9	211 ^a	0.1	20	10	7.09
					180.9	0.1	20	22	
57	Bifenazate	联苯肼酯	+	301	197.9 ^a	0.1	18	8	15.05
					169.9	0.1	18	20	
58	Metamitron	苯噻草酮	+	202.8	174.8 ^a	0.1	30	16	7.77
					103.6	0.1	30	22	
59	Indoxacarb	茚虫威	+	528.0	149.7 ^a	0.1	25	25	19.79
					293.0	0.1	25	15	

表 B.2 (续)

序号	英文名称	中文名称	电离方式 ESI	母离子 m/z	子离子 m/z	驻留时间/s	锥孔电压/V	碰撞能量/eV	保留时间/min
60	Buprofezin	噻嗪酮	+	306	200.9 ^a	0.1	20	12	20.18
					115.7	0.1	20	15	
61	Carboxin	萎锈灵	+	235.9	142.7 ^a	0.1	25	16	11.82
					86.6	0.1	25	25	
62	Propachlor	毒草胺	+	211.9	169.8 ^a	0.1	25	15	12.56
					93.7	0.1	25	25	
63	Pyriproxyfen	吡丙醚	+	322	95.7 ^a	0.1	25	15	22.29
					184.8	0.1	25	22	
64	Napropamide	敌草胺	+	272	128.8 ^a	0.1	25	15	14.99
					170.8	0.1	25	20	
65	Quizalofop-ethyl	精喹禾灵	+	373	299 ^a	0.1	30	20	20.32
					90.7	0.1	30	30	
^a 离子用于定量。									

附录 C
(资料性附录)
标准物质色谱图

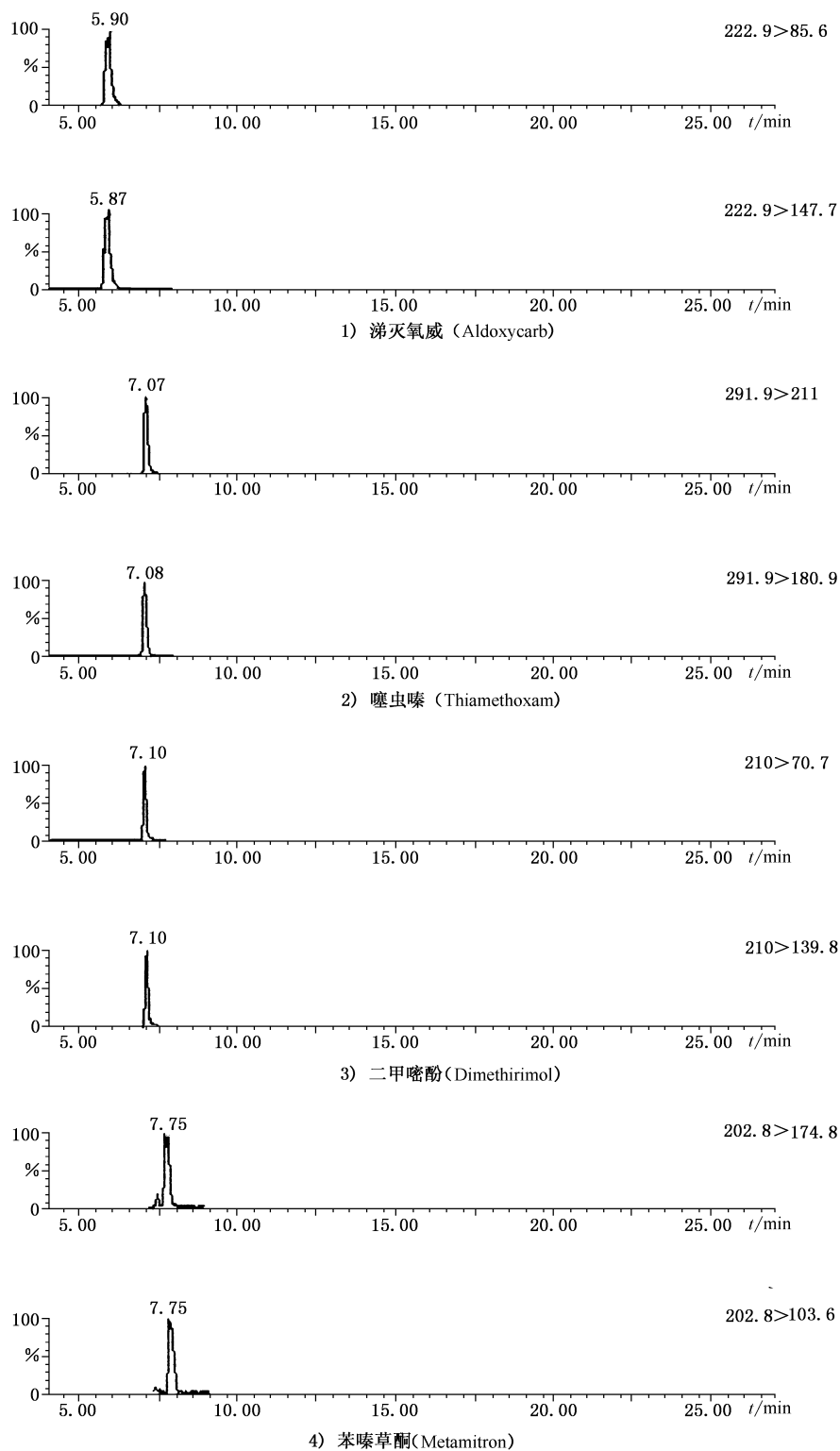
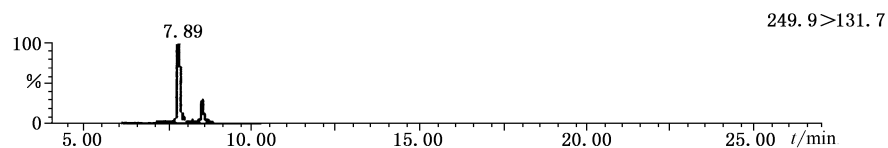
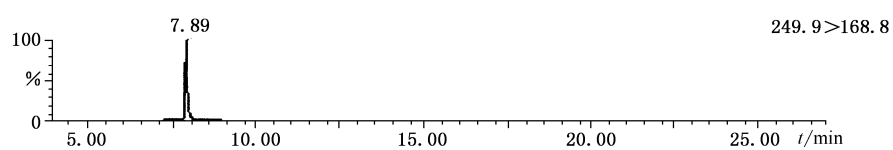
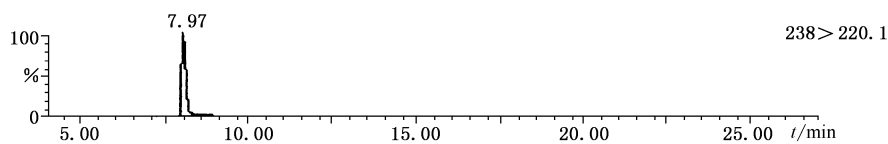
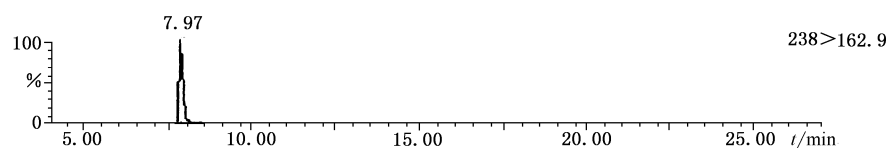


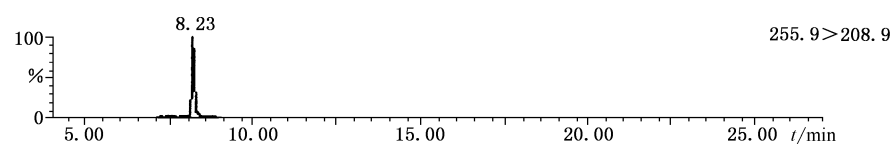
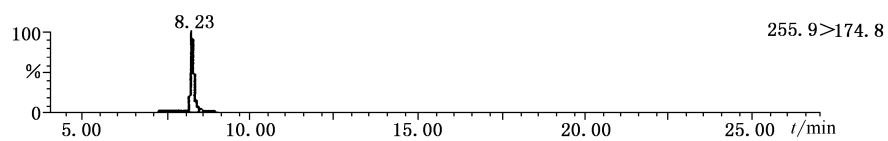
图 C.1 ESI+ 方式 57 种农药液相色谱-质谱/质谱多反应监测色谱图



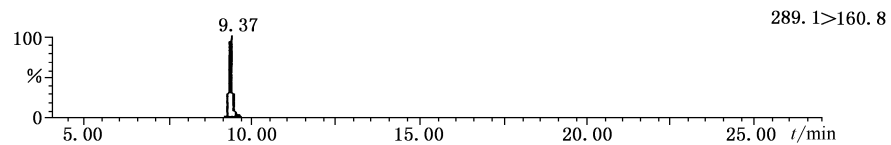
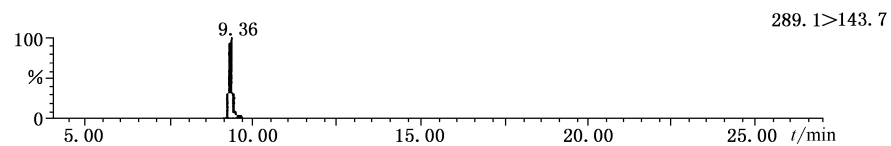
5) 噻虫胺(Clothianidin)



6) 3-羟基克百威(Carbofuran-3OH)

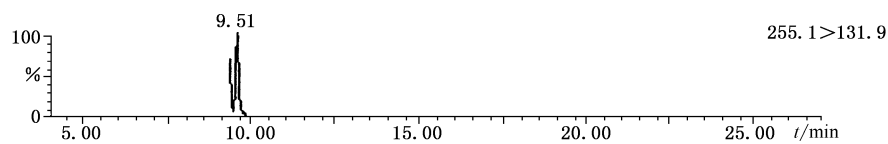
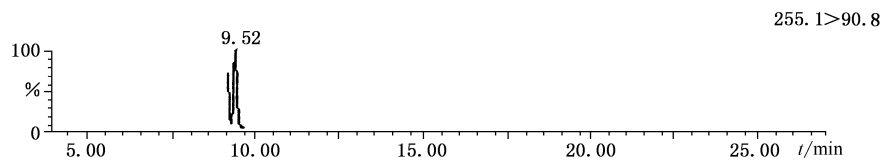


7) 吡虫啉(Imidacloprid)

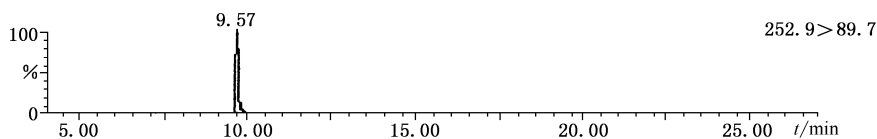
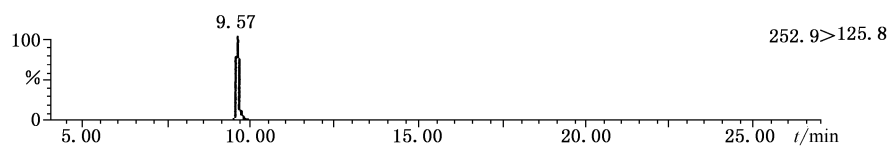


8) 咪草酸甲酯(Imazamethabenz-methyl)

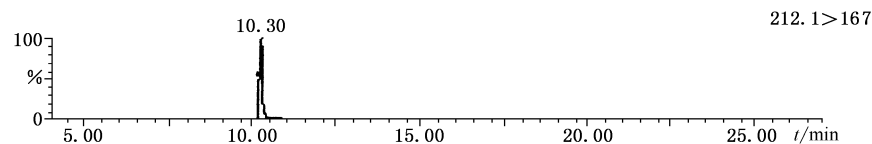
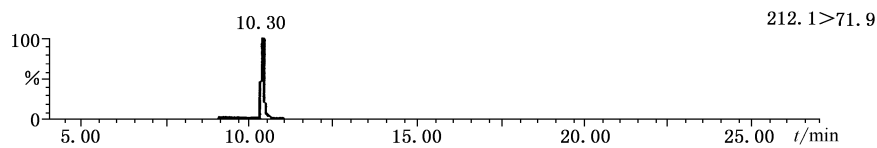
图 C.1 (续)



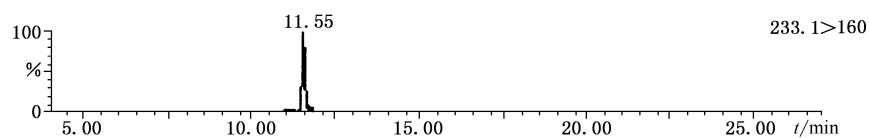
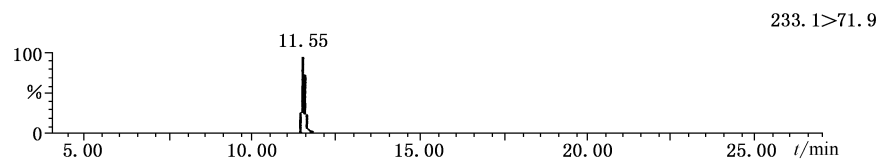
9) 噻菌隆(Ferimzone)



10) 噻虫啉(Thiacloprid)

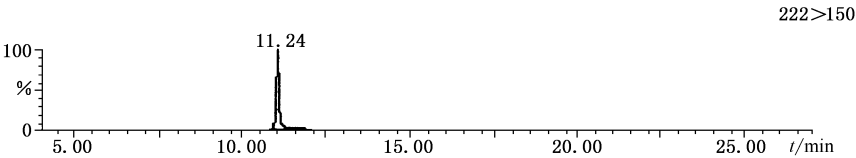
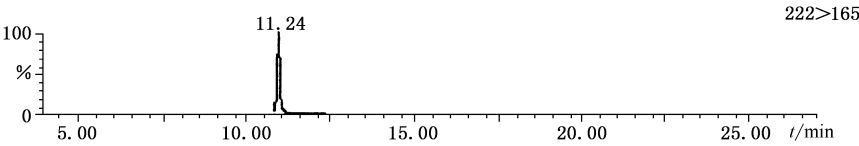


11) 异恶隆(Isouron)

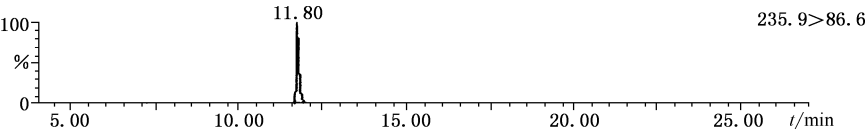
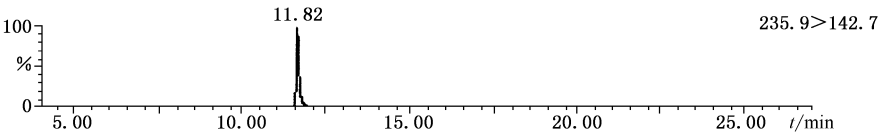


12) 氟草隆(Fluometuron)

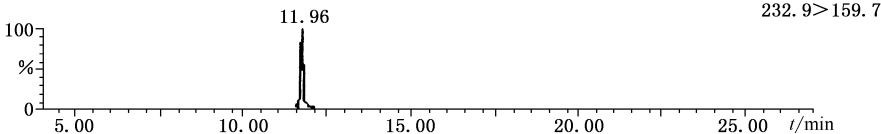
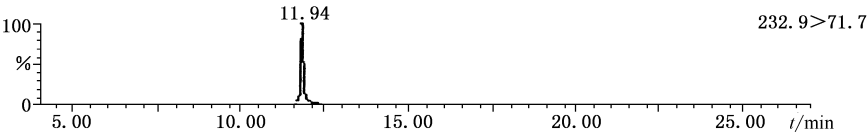
图 C. 1 (续)



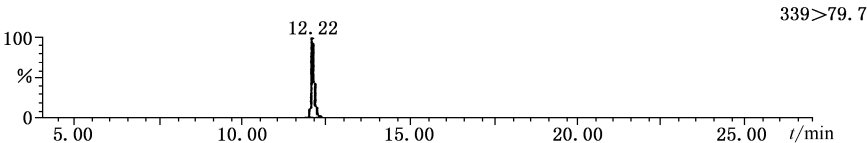
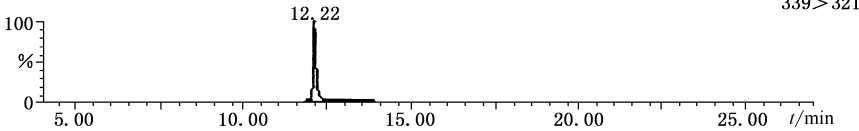
13) 甲基苯噻隆(Methabenzthiazuron)



14) 羧锈灵(Carboxin)

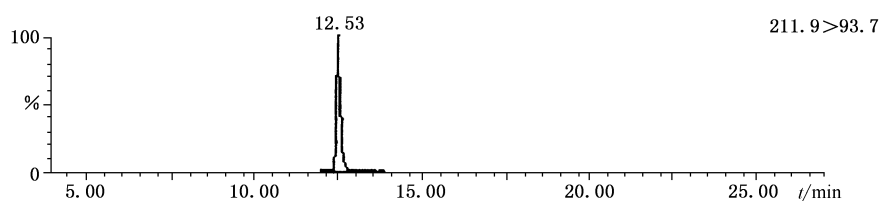
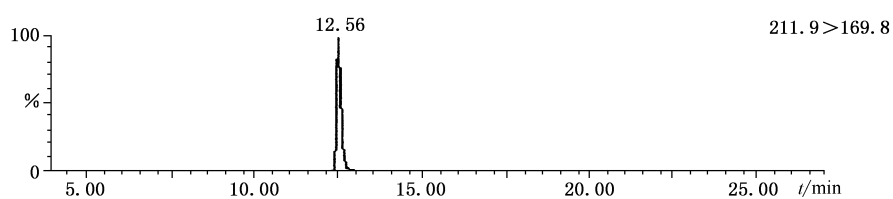


15) 敌草隆(Diuron)

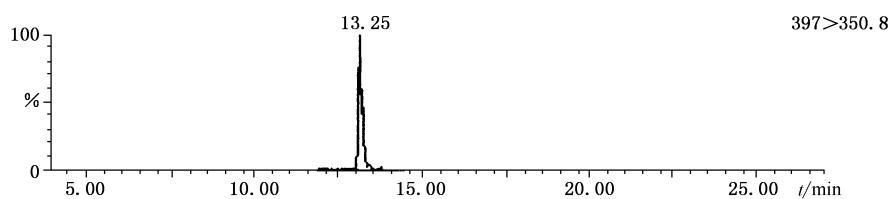
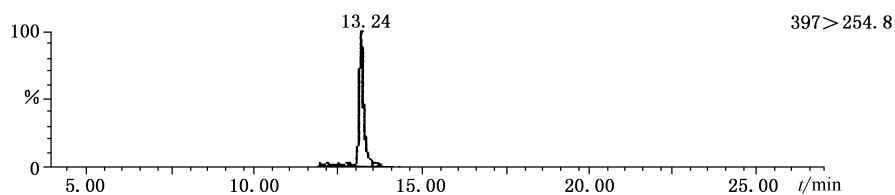


16) 抗倒胺(Inabenzfide)

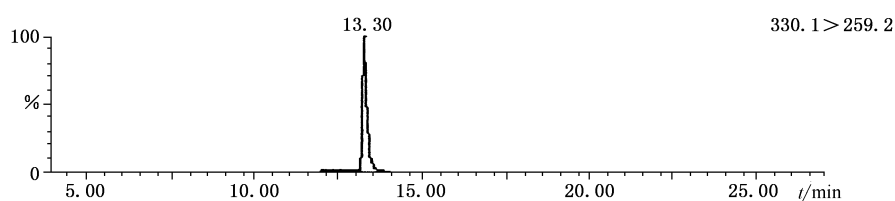
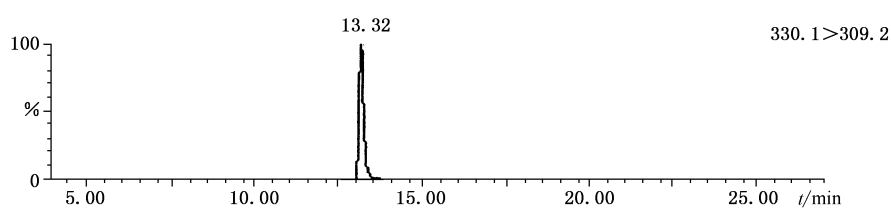
图 C.1 (续)



17) 毒草胺(Propachlor)

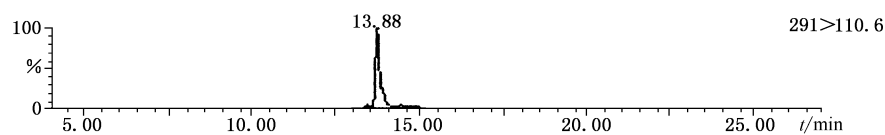
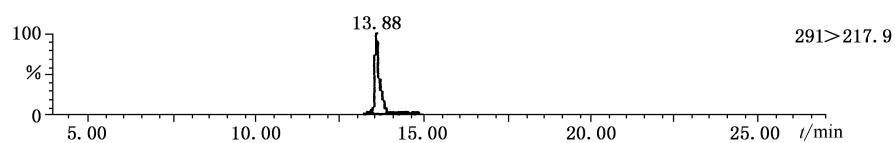


18) 乙虫清(Ethiprole)

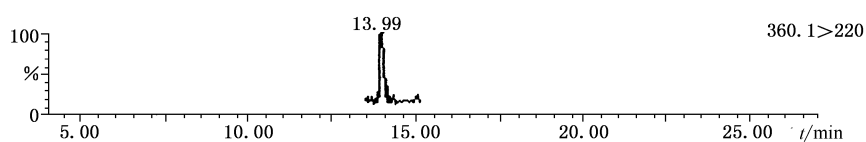
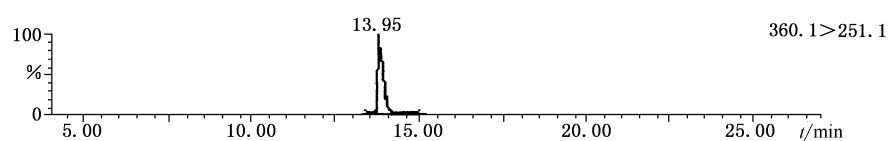


19) 氟啶酮(Fluridone)

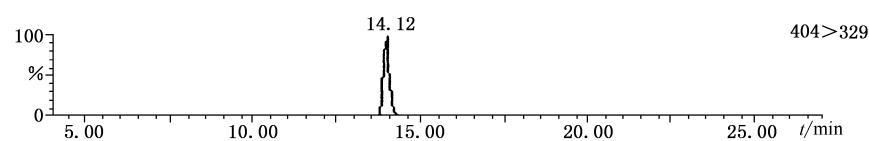
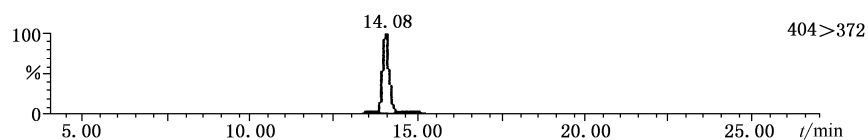
图 C.1 (续)



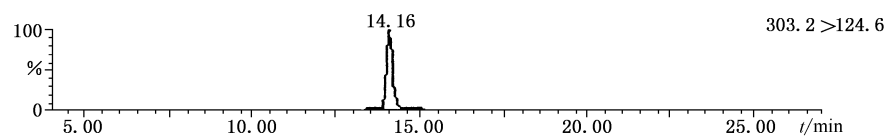
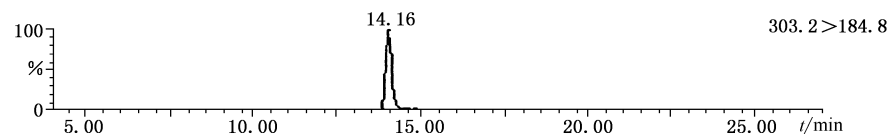
20) 枯草隆 (Chloroxuron)



21) 异恶唑草酮 (Isoxaflutole)

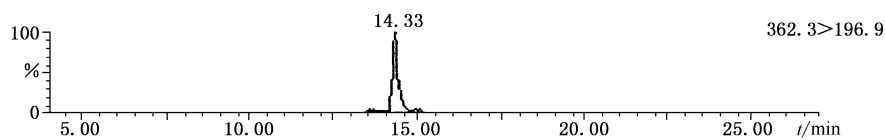
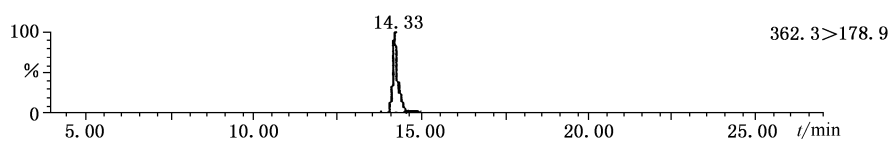


22) 腈嘧菌酯 (Azoxystrobin)

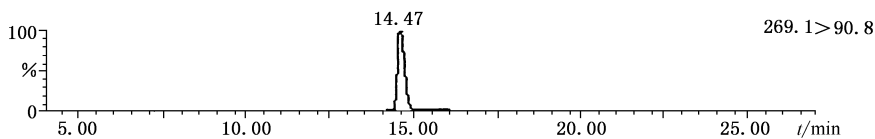
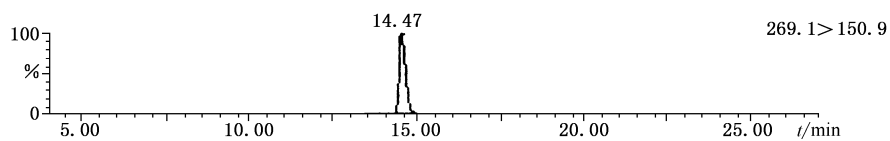


23) 苄草隆 (Cumyluron)

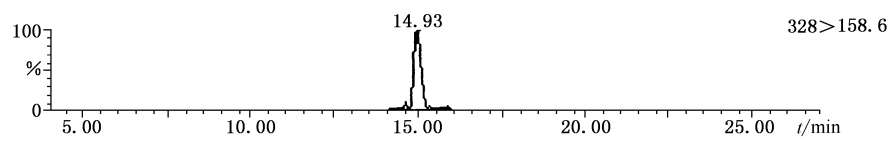
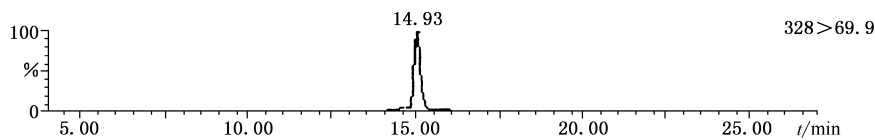
图 C.1 (续)



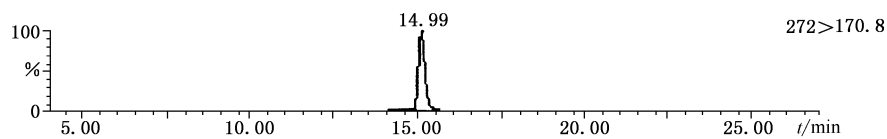
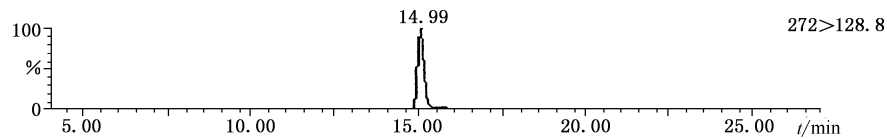
24) 恶咪唑(Oxpoconazole)



25) 杀草隆(Daimuron)



26) 苄氯三唑醇 (Diclobutrazol)



27) 敌草胺(Napropamide)

图 C. 1 (续)

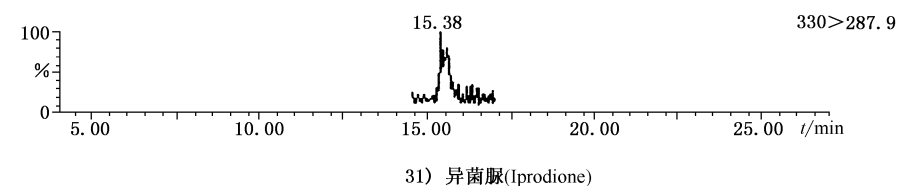
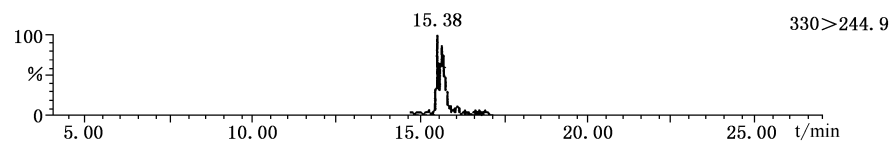
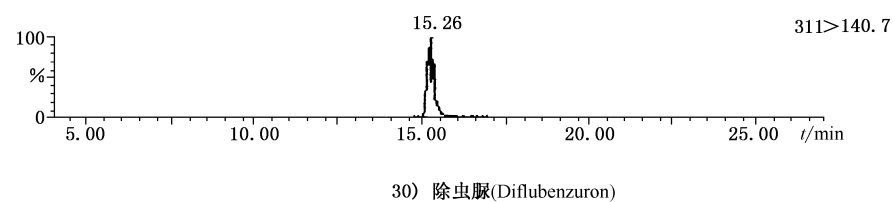
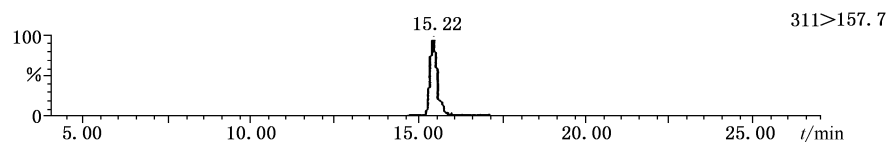
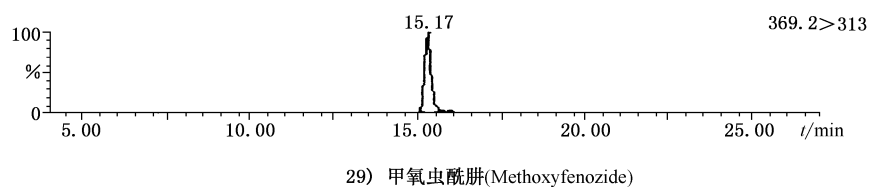
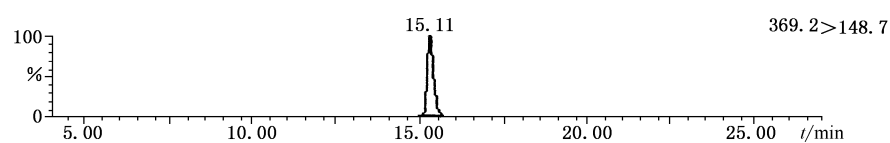
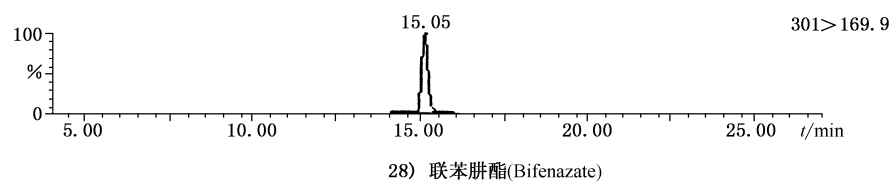
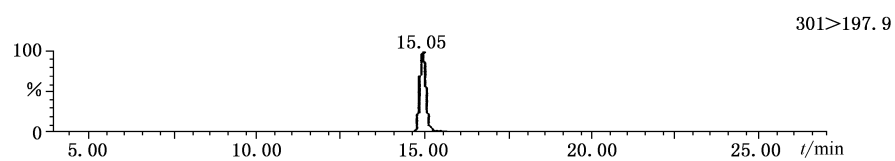
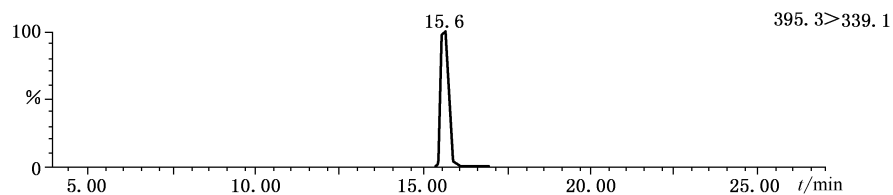
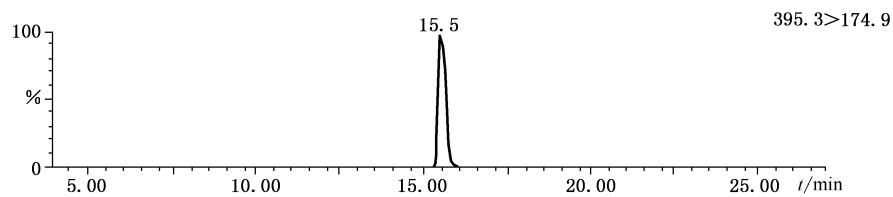
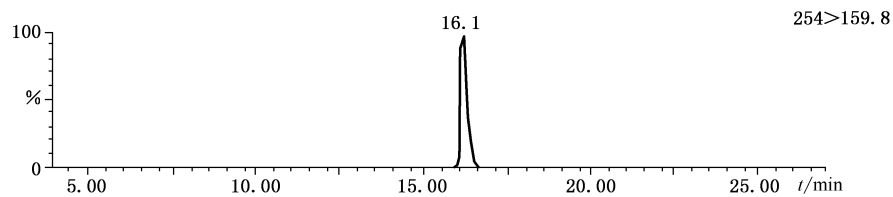
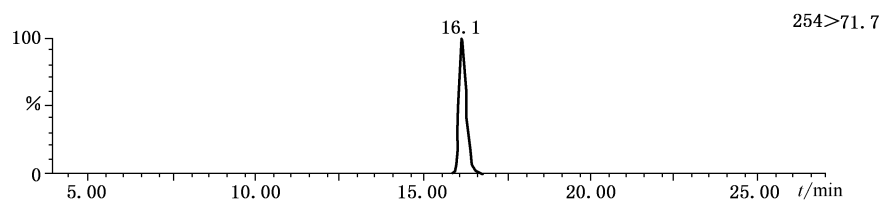


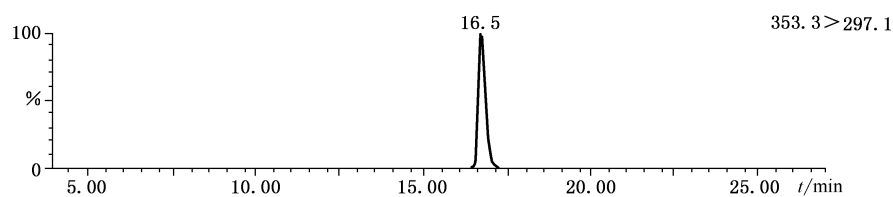
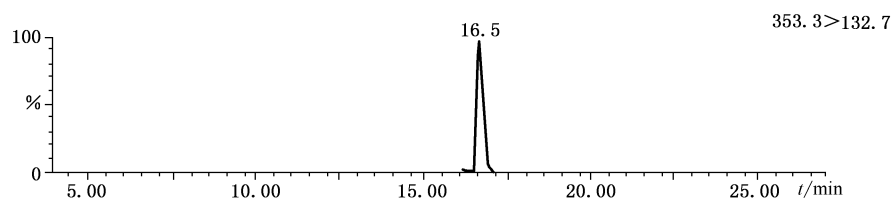
图 C. 1 (续)



32) 环虫酰肼 (Chromafenozide)

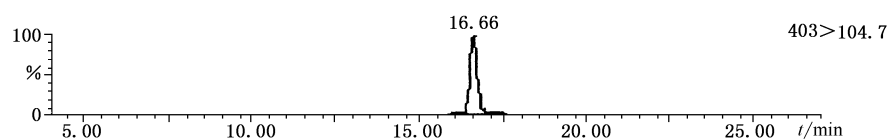
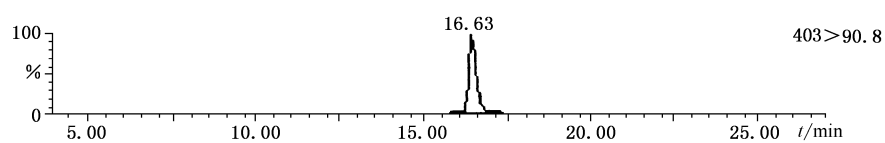


33) 苯硫威 (Fenothiocarb)

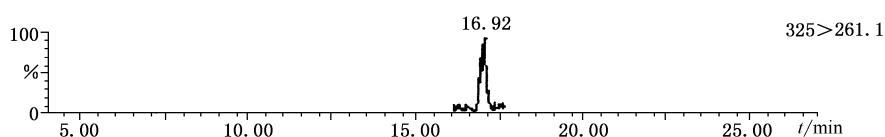
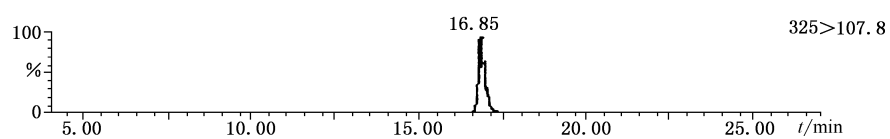


34) 虫酰肼 (Tebufenozide)

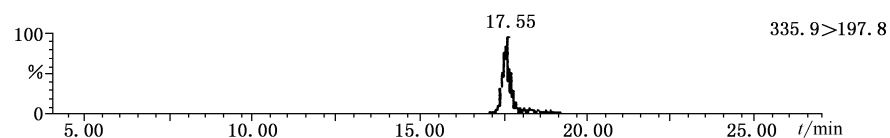
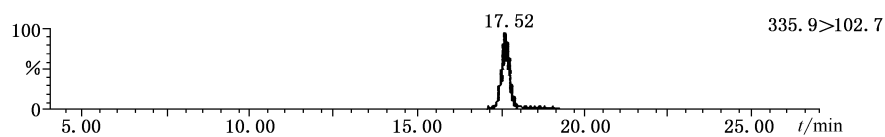
图 C. 1 (续)



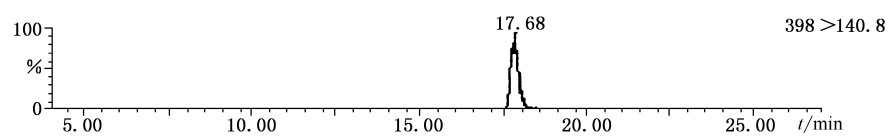
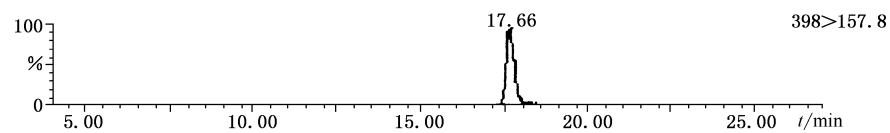
35) 苄草唑 (Pyrazoxyfen)



36) 氰霜唑 (Cyazofamid)

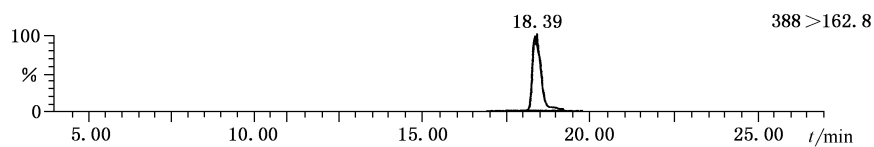
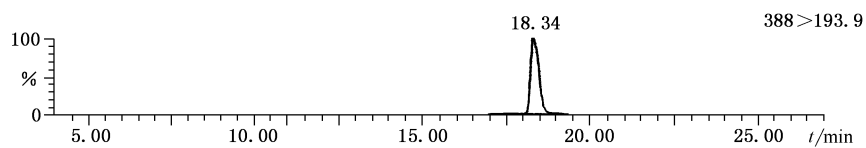


37) 加普胺 (Carpropamid)

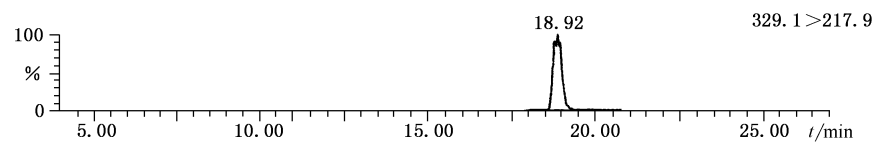
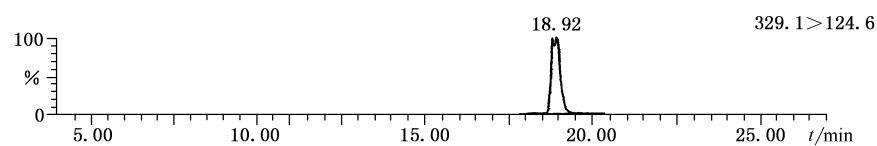


38) 地散磷 (Bensulide)

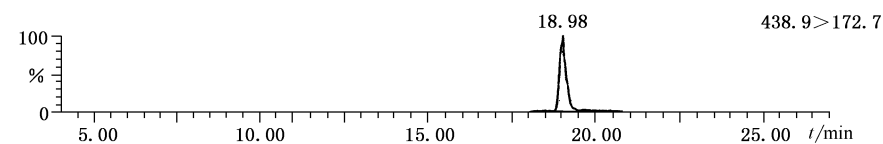
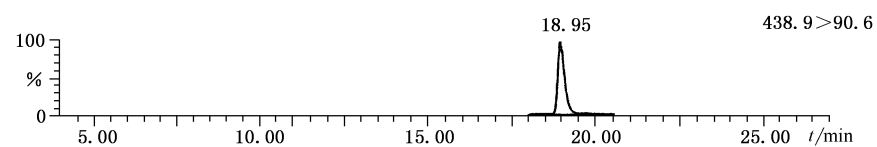
图 C. 1 (续)



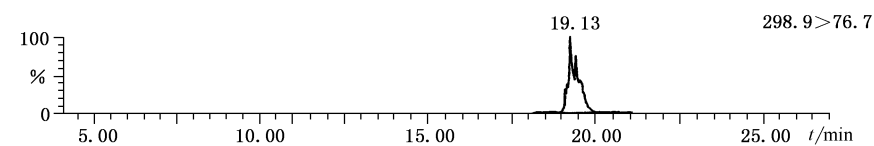
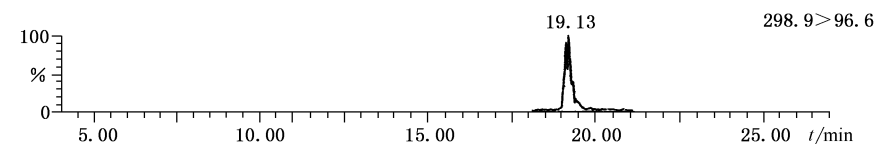
39) 唑菌胺酯(Pyraclostrobin)



40) 戊菌隆(Pencycuron)

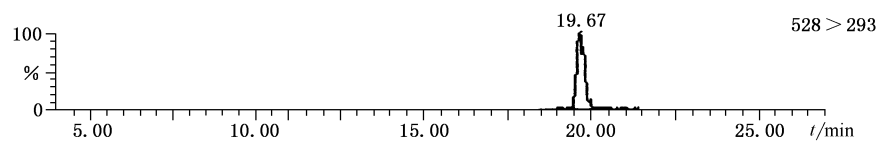
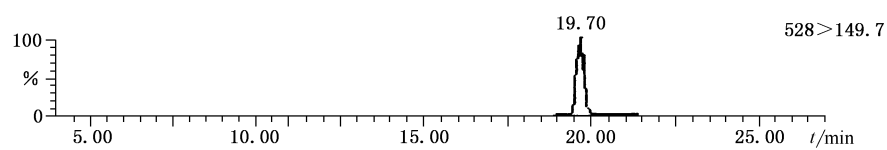


41) 吡唑特(Pyrazolynate)

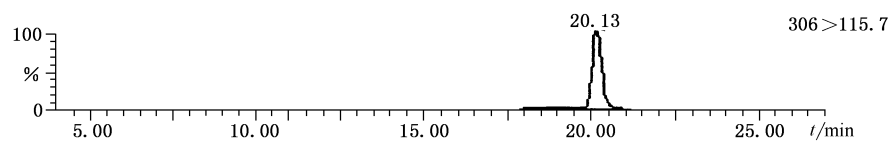
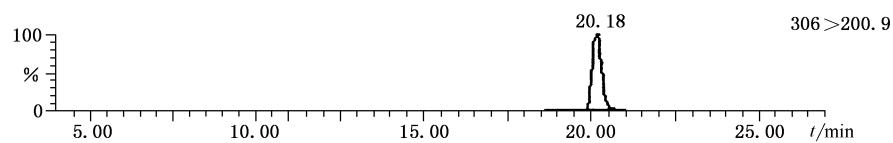


42) 辛硫酸(Phoxim)

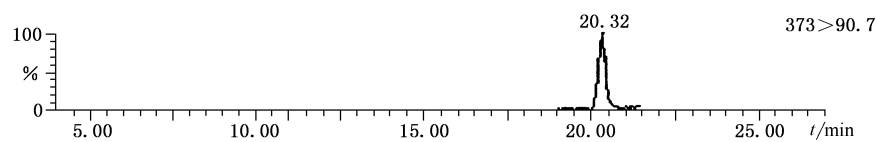
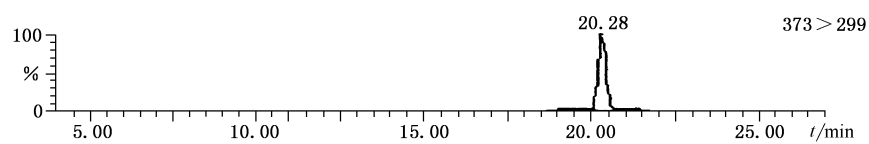
图 C. 1 (续)



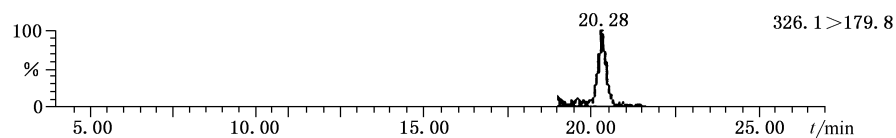
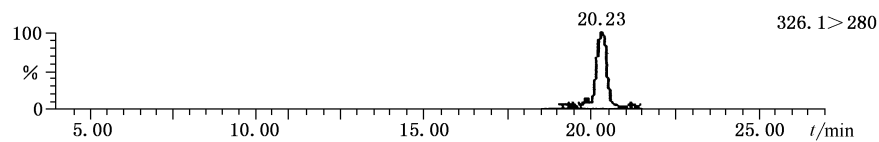
43) 茚虫威(Indoxacarb)



44) 噻嗪酮(Buprofezin)

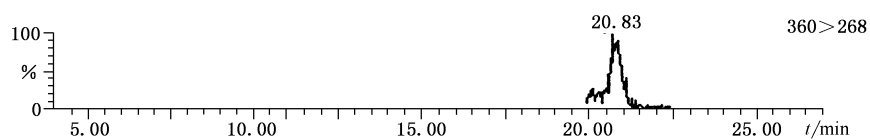
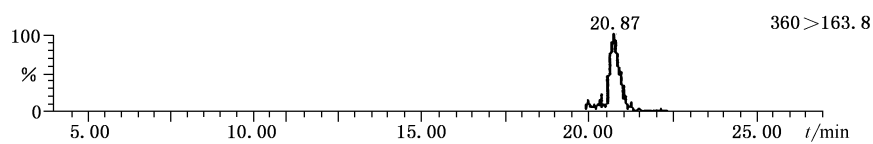


45) 精喹稗(Quizalofop-ethyl)

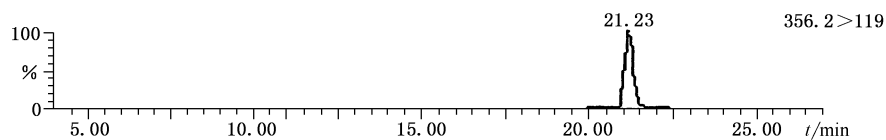
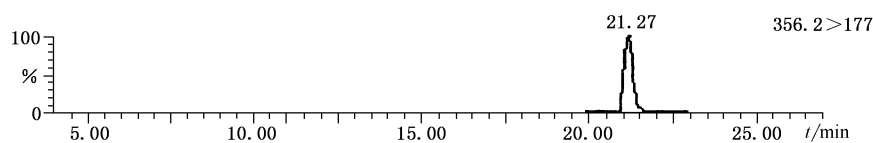


46) 噻草酮(Cycloxydim)

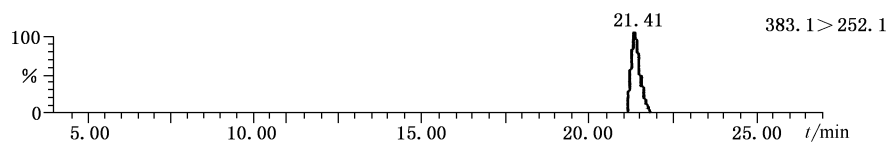
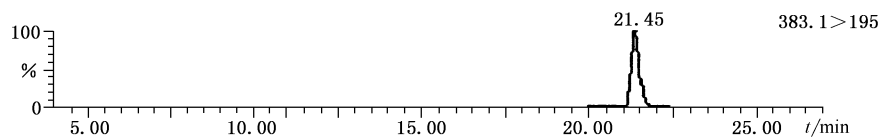
图 C. 1 (续)



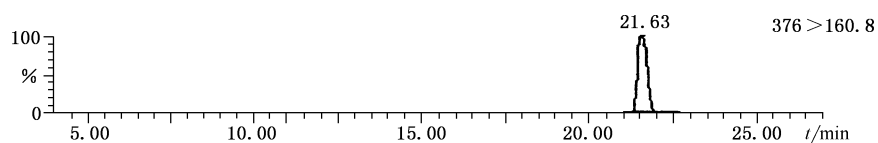
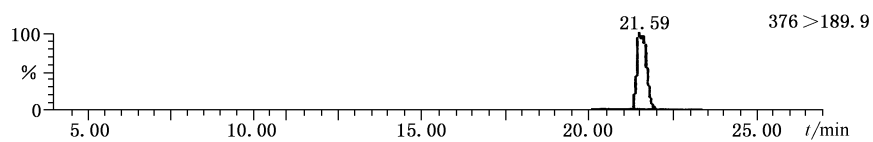
47) 烯草酮(Clethodim)



48) 增效醚(Piperonylbutoxid)

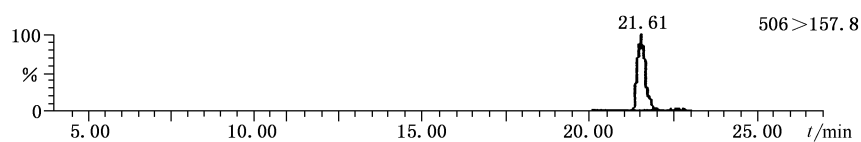
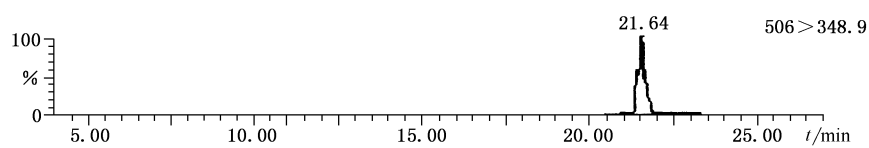


49) 呋线威(Furathiocarb)

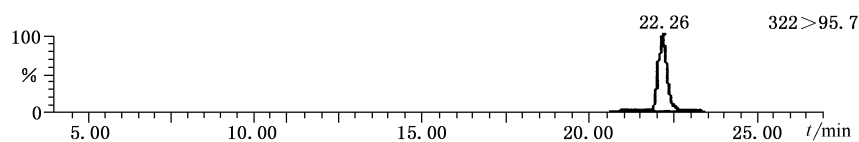
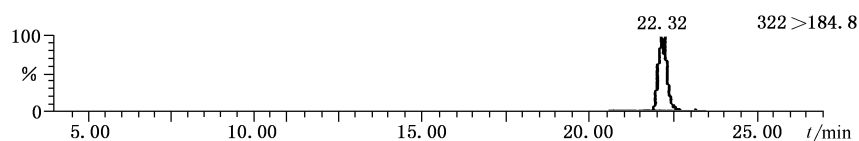


50) 噁嗪草酮(Oxaziclomefone)

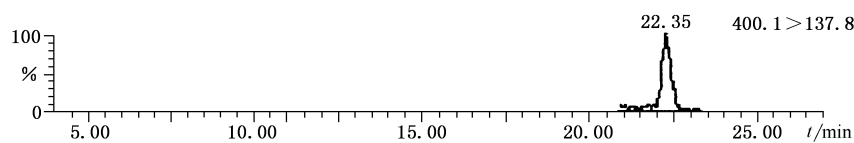
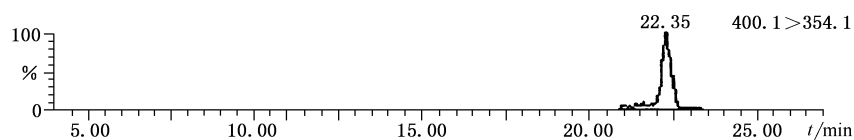
图 C. 1 (续)



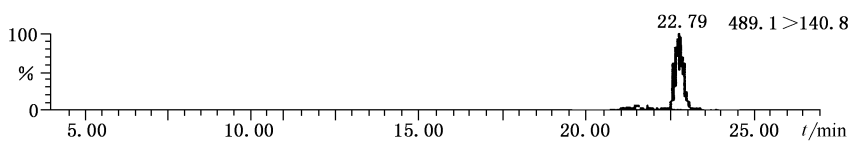
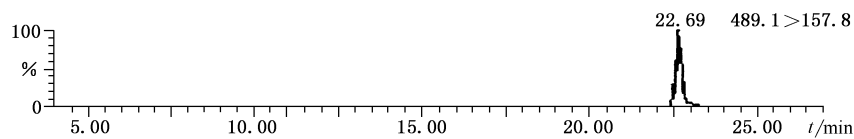
51) 啞蜚脲(Fluazuron)



52) 吡丙醚(Pyriproxyfen)

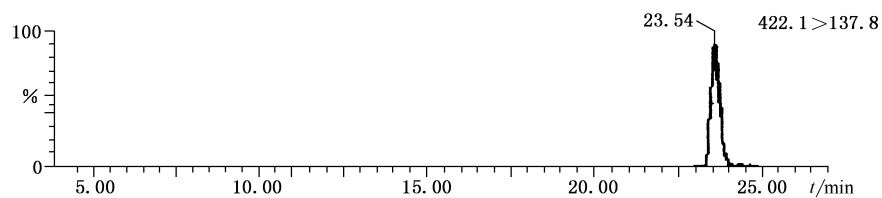
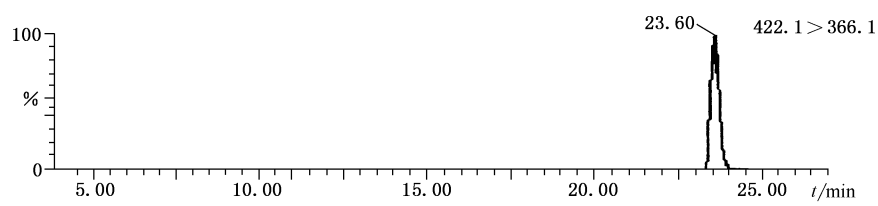


53) 丁氧环酮(Butoxydim)

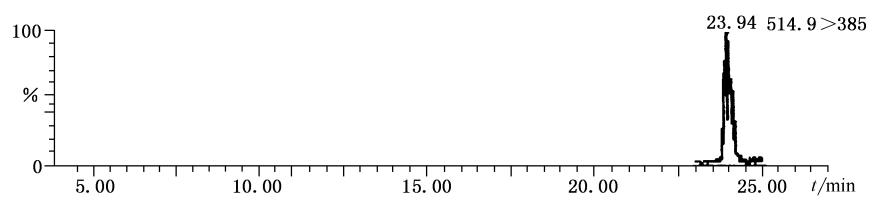
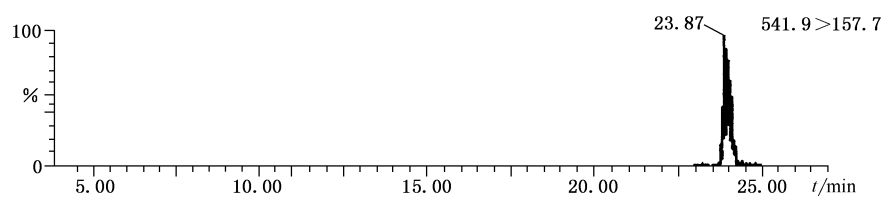


54) 氟虫脲(Flufenoxuron)

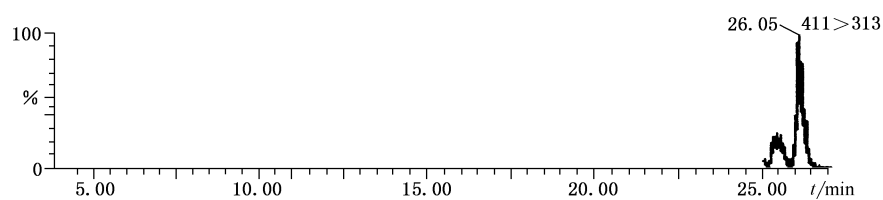
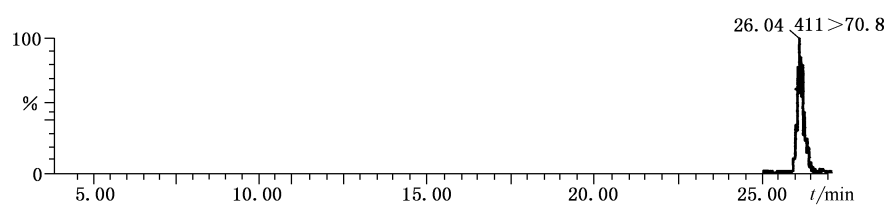
图 C. 1 (续)



55) 唑螨酯(Fenpyroximate)

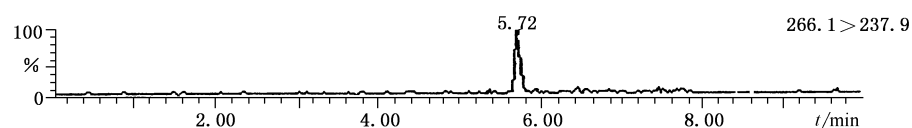
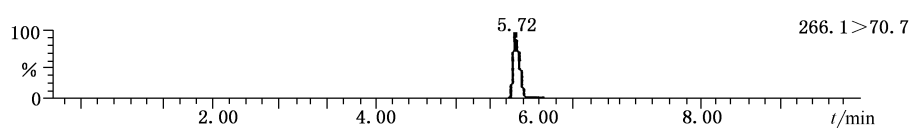


56) 氟啶脲(Chlorfluazuron)

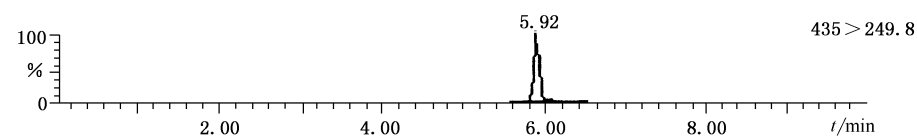
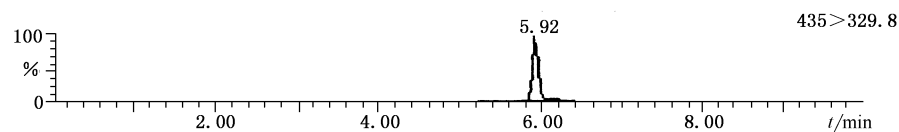


57) 螺螨酯(Spirodiclofen)

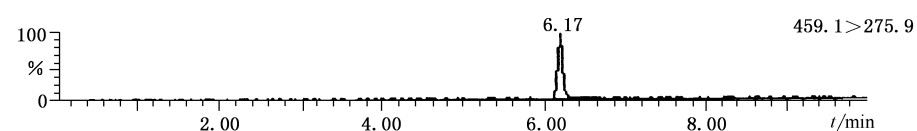
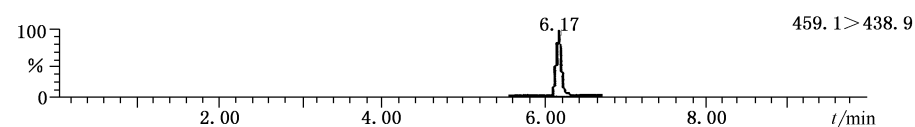
图 C.1 (续)



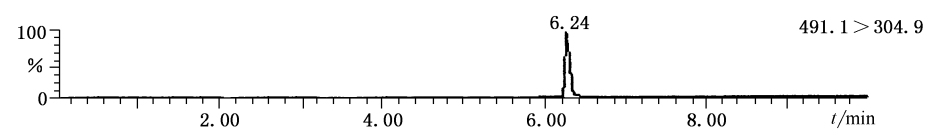
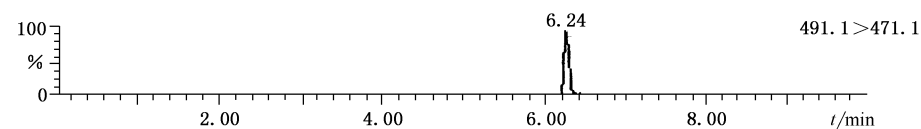
1) 噻酞菌胺(Thiadinil)



2) 氟虫清(Fipronil)

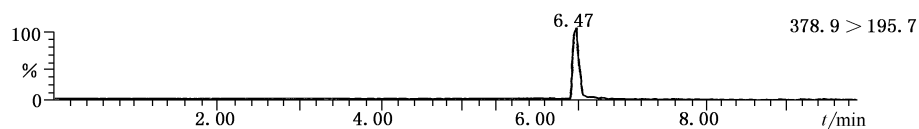
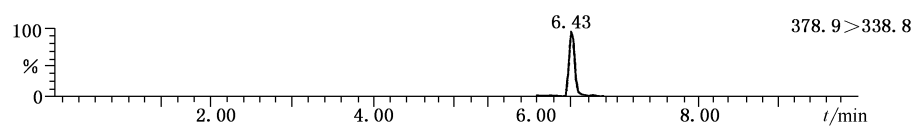


3) 氟铃脲(Hexaflumuron)

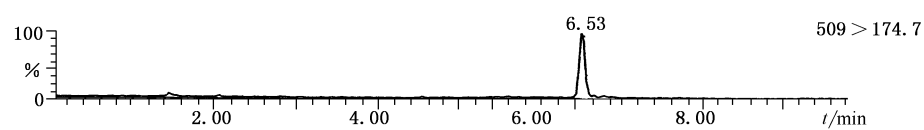
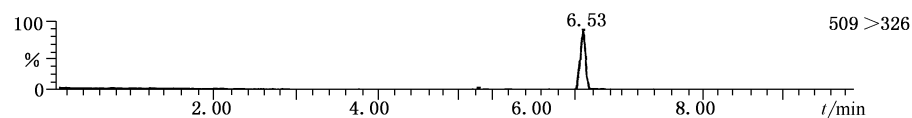


4) 双苯氟脲(Novaluron)

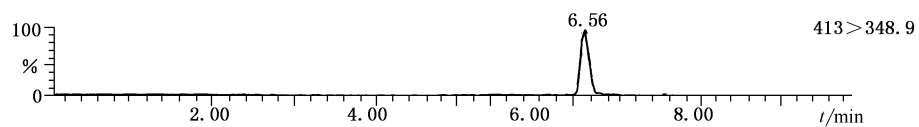
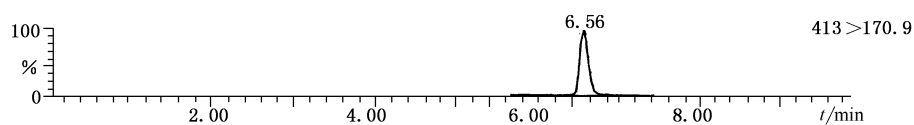
图 C.2 ESI—方式 8 种农药液相色谱-质谱/质谱多反应监测色谱图



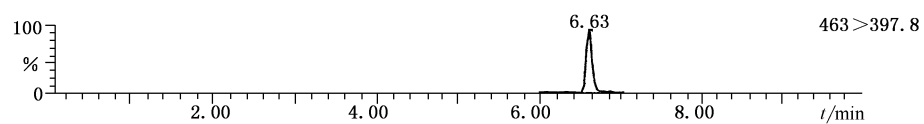
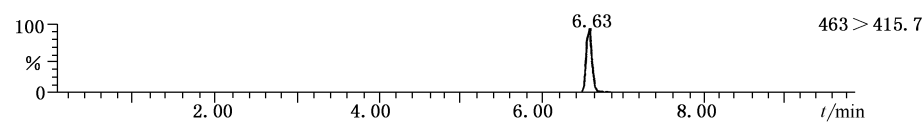
5) 氟苯脲(Teflubenzuron)



6) 氟丙氧脲(Lufenuron)



7) 氟硫灭(Flusulfamide)



8) 氟啶胺(Fluazinam)

按出峰顺序依次为：噻酰菌胺、氟虫腈、氟铃脲、双苯氟脲、氟苯脲、氟丙氧脲、氟硫灭、氟啶胺。

图 C.2 (续)

附 录 D
(规范性附录)
测定低限和添加回收率

表 D.1 65 种农药测定低限和添加回收率($n=10$)

化合物	添加 浓度/ ($\mu\text{g}/\text{kg}$)	玉米		糙米		大米		大麦		小麦		测定 低限/ (mg/kg)
		最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	
涕灭氧威	5	88.0	108.0	85.3	106.8	86.5	106.0	84.5	101.0	86.0	107.6	0.005
	20	90.0	105.5	86.9	108.7	85.3	107.5	80.2	102.8	86.8	106.9	
	200	84.2	107.3	82.7	109.4	85.4	107.4	80.4	95.6	87.6	108.9	
腈嘧菌酯	5	86.0	108.0	85.2	108.3	84.4	106.0	84.8	104.0	87.7	108.7	0.005
	20	85.5	107.5	83.2	106.7	86.5	105.5	84.9	105.3	87.3	108.0	
	200	88.1	107.2	87.3	105.2	86.3	104.4	84.5	100.9	87.5	106.4	
地散磷	5	84.0	108.0	82.4	109.5	83.1	106.0	89.8	107.3	85.3	103.6	0.005
	20	90.0	108.0	85.1	108.1	83.8	105.5	82.6	104.4	87.6	105.3	
	200	86.2	102.8	87.7	109.5	83.5	106.6	80.8	106.8	90.7	106.9	
联苯肼酯	20	85.5	108.5	84.8	108.1	87.2	106.5	87.0	105.3	85.1	108.0	0.02
	100	85.5	106.7	84.6	104.7	83.8	106.7	83.8	99.0	88.1	108.8	
	200	85.0	107.4	83.9	107.3	84.6	107.5	83.5	105.1	87.6	106.5	
噻嗪酮	5	86.0	108.0	82.5	105.5	84.3	106.0	82.9	105.8	89.4	108.8	0.005
	20	85.5	102.5	83.5	107.7	88.3	106.0	81.7	104.9	86.8	106.5	
	200	92.2	108.9	88.9	107.9	87.1	107.4	88.9	104.6	90.1	105.0	
丁苯草酮	5	86.0	108.0	83.1	108.3	86.9	106.0	81.3	103.9	91.6	108.0	0.005
	20	88.5	105.5	89.0	107.8	83.7	105.0	84.4	108.4	85.3	103.8	
	200	85.9	104.5	85.2	108.4	84.2	107.8	86.8	108.4	85.8	108.2	
3-羟基克百威	5	88.0	106.0	85.0	107.6	85.8	106.0	82.6	102.0	86.7	107.7	0.005
	20	85.5	107.0	84.7	108.8	87.4	100.5	83.1	105.3	89.0	106.4	
	200	86.9	108.9	87.9	108.7	83.1	105.9	82.0	102.3	92.0	107.1	
萎锈灵	5	90.0	108.0	82.3	107.3	83.1	106.0	82.7	105.7	88.7	105.7	0.005
	20	85.5	109.5	83.9	104.9	89.3	107.5	83.8	98.6	86.0	104.1	
	200	91.4	107.8	82.4	108.5	88.0	106.6	84.4	104.3	86.8	105.3	
环丙酰菌胺	5	86.0	106.0	84.5	106.7	84.7	106.0	85.8	107.3	87.6	101.2	0.005
	20	85.0	108.5	82.5	104.7	87.0	107.5	83.1	104.9	87.5	105.1	
	200	84.2	106.1	84.1	106.9	85.3	107.4	84.6	105.4	88.7	103.3	

表 D.1 (续)

化合物	添加 浓度/ ($\mu\text{g}/\text{kg}$)	玉米		糙米		大米		大麦		小麦		测定 低限/ (mg/kg)
		最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	
氟啶脲	20	91.0	108.5	87.1	108.8	93.0	107.5	80.4	107.0	88.2	108.5	0.02
	100	87.0	104.5	88.1	105.7	84.2	107.9	81.9	106.8	87.1	107.5	
	200	87.4	106.8	84.3	104.9	83.3	106.2	81.4	101.8	85.1	108.3	
枯草隆	5	88.0	108.0	83.2	107.1	91.2	102.0	81.2	107.5	86.1	106.4	0.005
	20	85.5	109.5	86.3	108.1	85.2	107.0	83.5	105.7	85.6	108.6	
	200	82.0	107.7	88.8	108.6	85.0	105.7	87.6	105.7	88.7	107.9	
环虫酰胺	5	86.0	106.0	82.5	107.4	83.2	98.0	85.8	105.7	85.7	106.3	0.005
	20	87.0	108.5	85.3	106.2	83.1	107.0	82.1	108.0	87.2	108.6	
	200	86.1	109.1	90.7	107.1	83.9	100.4	80.9	105.2	86.2	104.3	
烯草酮	5	86.0	108.0	92.1	106.6	90.1	106.0	81.5	103.4	89.1	101.5	0.005
	20	86.0	97.0	86.9	104.5	88.0	107.0	85.7	104.4	89.6	107.7	
	200	82.7	104.7	83.1	108.3	85.4	106.2	82.1	102.6	89.1	105.2	
噻虫胺	5	86.0	104.0	83.9	108.3	88.3	104.0	84.2	106.9	90.6	101.8	0.005
	20	93.0	108.5	87.5	107.0	83.5	99.0	82.7	103.5	86.3	106.0	
	200	86.3	109.1	82.9	105.9	84.1	107.9	80.3	107.5	91.6	108.4	
苄草隆	5	86.0	108.0	83.1	101.5	84.6	106.0	83.5	106.0	87.1	104.5	0.005
	20	87.5	109.0	83.1	103.0	83.9	104.5	84.9	103.9	87.9	105.5	
	200	85.9	103.4	86.1	104.5	84.0	105.1	82.1	104.8	89.1	105.2	
氰霜唑	5	86.0	108.0	84.1	97.5	84.7	104.0	81.0	103.8	88.3	106.4	0.005
	20	88.0	106.0	86.7	107.9	85.0	106.5	83.8	106.7	89.3	105.7	
	200	89.5	105.9	87.3	108.5	84.8	107.2	81.9	104.2	93.5	108.5	
噻草酮	5	86.0	108.0	81.8	107.4	86.4	106.0	86.0	96.5	92.6	108.2	0.005
	20	85.5	104.0	82.6	108.9	93.7	106.5	83.2	102.6	95.5	107.6	
	200	86.9	109.2	82.9	103.7	86.1	104.5	84.0	107.9	85.9	108.0	
杀草隆	5	86.0	108.0	83.5	106.8	83.5	106.0	80.4	105.0	85.8	106.1	0.005
	20	88.0	108.0	81.1	107.3	83.5	107.5	81.7	107.0	88.9	105.9	
	200	87.0	109.3	84.5	108.9	84.4	105.3	80.9	106.7	85.8	102.9	
苄氯三唑醇	5	86.0	108.0	87.5	102.3	88.0	104.0	81.1	94.5	94.3	108.3	0.005
	20	89.5	108.0	88.2	103.8	84.3	107.0	84.8	106.3	85.4	103.3	
	200	86.8	109.0	86.5	109.2	84.0	106.4	82.8	107.4	85.1	108.3	
除虫脲	20	85.5	109.5	86.4	107.5	85.8	105.5	80.6	104.2	85.3	104.7	0.02
	100	88.0	105.2	82.8	106.0	87.3	105.6	82.9	102.5	88.9	108.6	
	200	87.7	105.5	82.5	108.9	88.7	104.9	86.7	104.0	87.9	108.3	

表 D.1 (续)

化合物	添加 浓度/ ($\mu\text{g}/\text{kg}$)	玉米		糙米		大米		大麦		小麦		测定 低限/ (mg/kg)
		最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	
二甲嘧酚	5	86.0	108.0	89.7	107.9	90.9	106.0	80.8	100.5	87.7	106.6	0.005
	20	91.5	106.0	87.9	108.2	83.8	107.5	82.1	105.8	87.2	106.9	
	200	86.2	107.1	88.9	109.5	83.6	103.5	82.5	104.6	87.9	108.8	
敌草隆	5	86.0	106.0	84.8	106.9	84.1	106.0	84.1	103.3	86.0	104.9	0.005
	20	88.0	109.5	83.3	101.0	83.8	107.0	82.3	106.2	85.7	109.0	
	200	91.5	106.8	84.7	109.0	84.1	102.9	83.8	103.0	86.7	107.1	
乙虫清	5	88.0	106.0	85.8	109.2	83.9	106.0	81.4	107.7	86.4	104.8	0.005
	20	89.0	103.0	85.5	107.6	84.1	102.5	82.8	106.0	88.5	105.6	
	200	85.7	101.4	83.3	108.3	87.6	104.9	85.6	104.4	87.2	102.7	
苯硫威	5	86.0	108.0	86.8	105.7	84.2	106.0	86.4	103.7	85.5	105.0	0.005
	20	86.0	105.0	88.3	108.8	86.9	105.0	84.2	102.5	86.9	105.3	
	200	88.0	106.3	82.3	105.2	87.2	105.4	84.9	103.2	92.2	108.5	
唑螨酯	5	86.0	104.0	82.2	96.7	84.3	100.0	84.6	106.3	86.6	108.6	0.005
	20	85.5	107.5	83.5	106.9	84.2	107.5	85.5	106.9	85.2	106.0	
	200	89.5	109.4	82.9	107.0	87.5	100.0	80.4	107.2	85.3	104.8	
噻菌胺	5	86.0	108.0	82.3	108.4	85.9	106.0	81.1	103.4	87.1	107.1	0.005
	20	89.0	109.5	84.9	106.2	87.5	103.0	86.5	107.9	86.9	98.1	
	200	86.2	109.2	82.2	103.6	84.8	104.9	80.6	102.5	90.9	108.2	
氟虫腈	5	88.0	108.0	84.1	103.3	83.5	104.0	83.7	102.3	88.1	107.2	0.002
	20	86.5	109.5	83.3	108.7	85.2	105.5	86.0	106.8	90.5	108.2	
	200	83.6	107.3	87.1	106.2	83.7	107.1	80.2	107.7	85.6	108.0	
氟啶胺	5	84.0	108.0	84.2	108.5	86.7	106.0	85.1	106.9	87.1	106.4	0.005
	20	85.5	108.5	82.8	108.6	85.2	104.0	83.4	107.2	85.1	105.9	
	200	88.1	108.5	83.0	104.8	83.9	106.7	86.2	99.8	89.5	107.3	
啶蜚脲	20	85.0	106.5	89.6	108.3	83.5	107.0	81.8	107.4	87.3	108.6	0.02
	100	88.6	107.5	85.7	105.5	87.5	107.6	83.3	105.5	88.7	108.2	
	200	86.1	109.4	83.0	101.0	83.5	107.0	82.8	104.4	85.9	106.8	
氟虫脲	20	86.0	107.5	84.1	101.9	86.1	103.0	81.0	107.8	85.2	107.5	0.02
	100	89.8	108.1	82.2	108.7	87.3	107.5	87.1	105.0	85.4	108.2	
	200	88.4	108.3	87.7	106.0	83.2	106.4	81.0	102.0	85.1	108.0	
氟草隆	5	88.0	108.0	82.5	105.7	83.6	106.0	80.1	102.5	88.4	106.2	0.005
	20	90.5	109.0	85.1	108.5	83.4	107.5	86.7	104.1	85.3	109.0	
	200	86.7	108.0	82.2	106.1	83.2	107.8	81.5	99.2	86.9	101.6	

表 D.1 (续)

化合物	添加 浓度/ ($\mu\text{g}/\text{kg}$)	玉米		糙米		大米		大麦		小麦		测定 低限/ (mg/kg)
		最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	
氟啶酮	5	88.0	106.0	83.5	107.2	83.9	106.0	82.6	106.4	85.8	108.6	0.005
	20	88.5	105.0	85.1	108.7	89.1	107.0	85.4	104.0	85.2	104.0	
	200	85.7	108.0	84.5	104.0	85.0	106.4	82.7	106.6	90.8	106.9	
氟硫灭	5	82.0	106.0	85.1	105.1	87.2	104.0	83.6	106.5	85.8	106.2	0.005
	20	87.5	108.0	86.3	108.0	84.6	104.0	84.9	103.4	87.9	107.4	
	200	82.9	105.6	82.1	103.7	92.6	105.5	84.1	103.9	86.9	108.6	
呋线威	5	86.0	106.0	82.4	103.3	87.3	106.0	87.7	103.4	87.9	107.6	0.005
	20	85.5	107.5	83.2	109.1	84.6	107.5	84.9	105.3	90.1	108.3	
	200	84.6	109.2	87.1	109.4	87.6	104.7	81.6	103.1	85.7	108.2	
氟铃脲	5	86.0	104.0	83.1	104.3	87.0	102.0	80.4	103.7	86.4	108.9	0.005
	20	91.0	109.0	84.0	106.7	91.5	103.5	84.0	103.3	85.5	108.0	
	200	87.3	107.1	87.2	109.2	87.6	106.0	80.4	106.5	86.6	105.3	
咪草酸甲酯	5	88.0	108.0	83.2	108.2	87.4	102.0	80.3	102.8	86.1	106.2	0.005
	20	89.0	108.5	86.3	107.1	85.0	99.5	83.8	101.3	89.5	105.9	
	200	93.6	109.0	85.2	108.8	88.2	103.2	81.6	105.4	85.3	108.5	
吡虫啉	5	84.0	108.0	84.6	106.4	84.5	102.0	82.5	106.0	85.2	108.4	0.005
	20	90.0	109.5	84.1	108.3	83.4	106.5	86.1	102.4	85.3	109.0	
	200	85.3	108.9	87.8	106.2	86.7	104.9	81.3	107.8	86.0	108.3	
抗倒胺	5	88.0	108.0	83.3	105.6	86.2	106.0	82.9	99.8	86.4	105.8	0.005
	20	86.0	109.0	82.4	105.5	83.7	104.5	82.2	107.3	87.4	106.5	
	200	92.7	105.5	86.9	109.5	90.5	105.7	80.5	98.0	88.6	109.0	
茚虫威	5	86.0	108.0	85.2	108.4	89.9	106.0	82.4	105.0	85.4	107.2	0.005
	20	87.0	105.5	85.8	102.6	86.1	101.5	87.2	102.6	86.0	103.3	
	200	88.3	107.8	82.9	109.2	83.7	102.8	84.3	105.6	87.4	108.8	
异菌脲	20	86.0	109.5	90.3	108.4	83.4	104.0	82.6	103.7	92.0	108.5	0.02
	100	85.8	108.6	89.3	109.4	84.0	104.4	91.9	106.6	85.2	106.5	
	200	85.1	108.1	90.2	105.7	88.2	107.1	82.7	103.7	85.5	106.5	
异恶隆	5	88.0	104.0	90.4	109.5	84.8	104.0	82.2	105.3	85.2	109.0	0.005
	20	85.0	106.5	88.1	104.6	86.4	106.0	83.6	105.7	87.4	106.2	
	200	89.7	108.5	82.2	109.2	87.0	102.7	84.0	106.7	86.4	105.4	
异恶唑草酮	5	88.0	108.0	86.2	108.8	90.6	106.0	80.3	98.6	85.7	108.3	0.005
	20	85.5	103.0	87.8	107.9	83.7	105.5	84.8	104.4	86.2	108.3	
	200	89.5	108.8	83.1	108.9	86.1	106.4	80.7	106.5	88.5	105.7	

表 D.1 (续)

化合物	添加 浓度/ ($\mu\text{g}/\text{kg}$)	玉米		糙米		大米		大麦		小麦		测定 低限/ (mg/kg)
		最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	
氟丙氧脲	5	88.0	104.0	83.3	103.9	85.0	106.0	80.7	105.5	85.4	108.5	0.005
	20	86.0	105.5	82.6	103.2	86.5	106.0	82.3	106.8	93.0	108.8	
	200	82.2	109.7	87.3	102.8	83.1	106.4	87.6	104.0	86.7	107.4	
苯噻草酮	5	84.0	104.0	84.3	105.5	89.6	104.0	87.6	105.6	85.4	106.8	0.005
	20	88.5	106.5	87.9	109.0	83.2	107.0	84.9	106.4	86.0	107.5	
	200	88.7	109.6	83.2	109.6	84.0	106.4	84.5	106.1	85.6	104.4	
甲基苯噻隆	5	90.0	108.0	85.1	108.1	83.4	104.0	86.4	105.8	85.6	103.6	0.005
	20	87.5	106.5	84.3	105.1	86.4	106.0	86.2	104.3	87.0	108.2	
	200	90.8	106.4	82.5	107.7	83.4	107.9	80.6	107.0	89.7	104.6	
甲氧虫酰肼	5	86.0	108.0	91.4	106.3	84.0	106.0	81.8	108.5	86.8	105.8	0.005
	20	85.5	109.5	86.3	105.0	85.2	106.0	84.5	107.6	86.0	108.1	
	200	87.3	109.4	83.0	104.4	84.5	107.9	82.7	103.0	86.4	106.4	
敌草胺	5	86.0	108.0	82.4	108.3	84.4	106.0	85.8	101.0	87.5	107.1	0.005
	20	93.0	107.0	84.8	109.7	85.9	104.5	84.1	106.2	87.4	109.0	
	200	86.5	108.7	83.1	103.9	86.0	107.3	83.2	100.8	88.7	105.7	
双苯氟脲	5	90.0	108.0	82.4	105.7	88.7	104.0	81.4	104.0	85.6	104.7	0.005
	20	92.5	106.5	83.3	108.8	83.8	105.0	86.7	104.4	85.6	108.3	
	200	85.8	109.4	87.9	105.4	86.1	104.4	86.0	105.7	86.8	105.5	
噁嗪草酮	5	86.0	106.0	83.9	108.9	87.7	106.0	83.9	107.5	87.5	107.1	0.005
	20	82.5	106.5	83.6	106.5	86.7	107.0	82.6	106.4	88.4	108.9	
	200	86.6	109.1	82.3	109.1	92.3	106.2	84.4	104.6	85.9	108.2	
恶咪唑	5	88.0	106.0	82.2	107.1	86.5	102.0	85.4	105.9	87.7	109.0	0.005
	20	86.0	106.5	83.8	104.3	85.8	106.5	85.2	105.0	87.8	106.4	
	200	88.4	109.3	85.6	107.2	84.8	106.6	90.1	100.0	90.5	106.5	
戊菌隆	5	88.0	108.0	84.5	109.3	85.5	104.0	83.2	93.3	89.2	106.0	0.005
	20	87.0	106.0	82.2	107.8	83.5	107.5	84.1	104.7	85.6	108.3	
	200	84.9	107.6	83.9	104.7	86.2	103.1	83.7	104.4	85.9	105.7	
辛硫磷	5	94.0	108.0	83.3	109.8	86.9	106.0	82.5	106.8	85.4	108.3	0.005
	20	89.5	105.0	84.9	106.0	85.5	106.5	84.9	107.9	87.0	104.1	
	200	85.1	107.5	83.5	108.7	83.2	107.5	82.5	101.7	91.1	103.0	
增效醚	5	88.0	106.0	84.3	108.0	83.3	100.0	80.1	105.8	86.3	106.2	0.005
	20	92.0	108.5	86.1	107.2	83.9	104.0	83.9	104.0	87.0	106.9	
	200	88.4	108.7	88.0	108.5	83.5	105.3	86.1	106.9	86.3	108.7	

表 D.1 (续)

化合物	添加 浓度/ ($\mu\text{g}/\text{kg}$)	玉米		糙米		大米		大麦		小麦		测定 低限/ (mg/kg)
		最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	
毒草胺	5	86.0	104.0	84.6	109.1	83.4	106.0	85.0	105.1	89.4	104.0	0.005
	20	89.5	105.5	82.3	99.4	88.2	104.5	84.5	103.9	87.1	107.5	
	200	85.9	108.9	83.1	107.2	87.3	103.0	80.3	101.5	90.6	106.7	
唑菌胺酯	5	84.0	104.0	83.5	108.1	85.1	106.0	81.3	104.4	88.9	107.0	0.005
	20	86.0	109.5	82.6	108.2	84.4	105.5	83.8	105.0	86.5	108.3	
	200	85.7	109.2	84.6	104.0	86.3	104.4	80.4	105.0	89.4	107.3	
吡唑特	5	88.0	108.0	86.0	109.2	84.6	104.0	84.7	104.5	86.6	108.3	0.005
	20	92.0	109.0	89.1	108.6	85.5	105.0	81.4	103.9	90.4	102.3	
	200	86.7	107.4	82.8	108.2	83.9	107.8	84.8	103.8	90.8	107.4	
苄草唑	5	92.0	106.0	82.6	109.7	85.7	104.0	82.6	98.9	89.3	106.3	0.005
	20	85.5	106.5	83.0	103.9	83.8	100.0	82.6	105.3	85.8	100.7	
	200	85.6	106.8	85.5	107.1	84.0	102.9	85.7	105.8	88.3	105.1	
吡丙醚	5	86.0	104.0	83.1	108.5	86.3	104.0	86.8	105.0	86.0	108.6	0.005
	20	89.5	108.0	83.0	106.8	85.2	104.0	80.1	106.6	85.3	104.0	
	200	87.4	106.3	82.5	108.7	83.9	107.4	82.3	106.7	91.8	108.3	
精噻禾灵	5	82.0	106.0	83.6	108.1	85.8	106.0	87.9	106.9	85.4	106.2	0.005
	20	88.0	108.0	82.9	105.1	85.7	104.5	88.1	98.5	93.4	106.2	
	200	86.2	109.7	82.4	98.0	84.6	105.9	84.3	105.9	88.8	106.9	
螺螨酯	20	89.0	109.0	84.7	107.5	84.8	107.5	80.5	106.1	88.0	107.6	0.02
	100	85.8	101.5	83.7	108.2	84.5	107.3	83.0	108.8	89.5	107.6	
	200	86.4	109.0	83.3	109.1	84.0	101.1	84.3	105.3	87.0	104.5	
虫酰肼	5	88.0	104.0	88.2	106.4	83.1	104.0	84.9	102.8	88.4	108.6	0.005
	20	87.0	104.5	82.9	109.6	85.5	106.0	81.4	100.6	89.5	104.3	
	200	85.3	109.7	85.3	108.5	84.5	107.0	80.2	108.9	85.4	106.0	
氟苯脲	5	82.0	108.0	87.1	107.3	83.7	102.0	83.2	106.8	88.4	107.5	0.005
	20	84.5	109.5	85.4	106.9	84.4	104.0	86.0	108.6	85.8	108.9	
	200	85.3	104.0	83.1	108.1	83.9	106.7	80.7	100.7	85.8	107.7	
噻虫啉	5	88.0	106.0	83.1	109.3	87.0	106.0	84.9	104.8	86.9	108.7	0.005
	20	85.0	108.5	86.8	105.4	88.0	107.5	84.1	106.6	86.6	106.0	
	200	89.8	107.8	95.0	107.9	83.6	101.1	80.4	104.6	85.2	107.4	

表 D.1 (续)

化合物	添加 浓度/ ($\mu\text{g}/\text{kg}$)	玉米		糙米		大米		大麦		小麦		测定 低限/ (mg/kg)
		最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	最小 回收 率/%	最大 回收 率/%	
噻酰菌胺	5	86.0	108.0	85.5	103.4	88.2	102.0	86.3	102.9	86.9	107.3	0.005
	20	85.0	109.5	89.0	108.7	93.3	106.5	87.7	106.6	85.2	107.5	
	200	83.3	108.8	84.7	106.8	84.3	107.4	88.0	106.9	87.1	105.6	
噻虫嗪	5	86.0	106.0	83.1	109.7	84.1	106.0	84.2	103.7	88.1	108.3	0.005
	20	84.5	109.5	83.6	106.6	84.5	107.5	81.9	107.8	85.4	107.5	
	200	83.0	109.3	91.2	108.7	84.1	103.7	83.0	108.4	89.1	107.8	