

附二、33种农药的GC-MS/MS检测方法参考

1.1 仪器设备

气相色谱-质谱联用仪 (GC-MS/MS): TQ8040(岛津公司);

1.2 仪器条件

1.2.1 气相条件

色谱柱: 菲罗门 FBX-5MS 15 m × 0.25 mm × 0.25 μm(货号: M005-025-025-15)。

实际应用中推荐标准用柱 FBX-5MS 30 m × 0.25 mm × 0.25 μm(货号: M005-025-025-30)

或者 FBX-1701 MS 30m x 0.25mm x 0.25um(货号: M005-025-025-30)

柱流量: 1.00 mL/min;

进样量: 1μL;

进样口温度: 250 °C;

载气: 高纯氮气;

碰撞气: 高纯氩气, 纯度≥99.999%;

升温程序: 80 °C, 40°C/min 升至 250°C, 20°C/min 升至 290°C, 保持 3.75 min。

1.2.2 质谱条件

电离方式: EI;

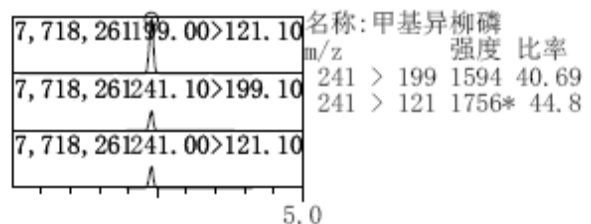
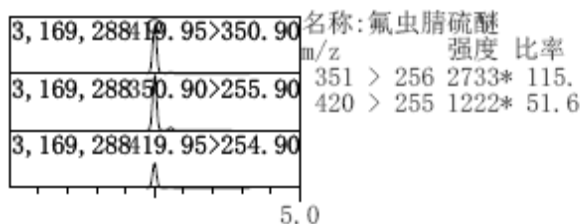
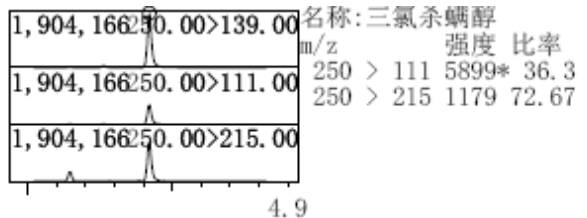
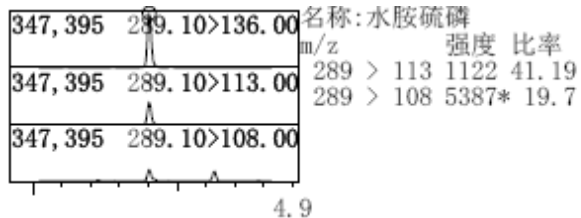
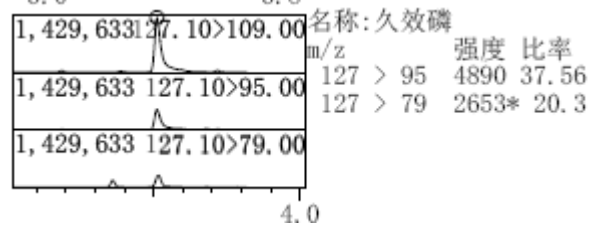
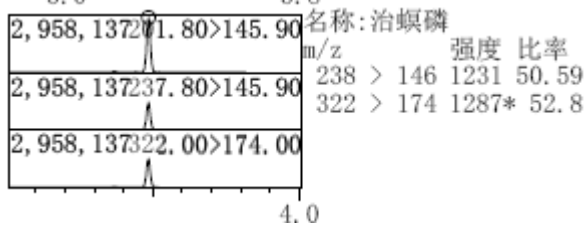
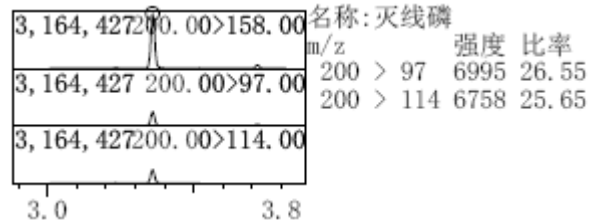
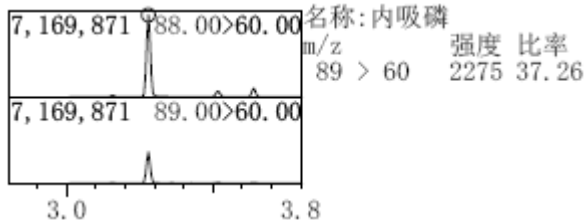
色谱-质谱接口温度: 250 °C;

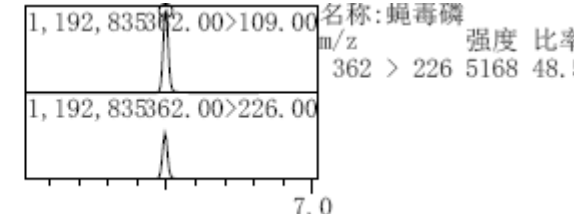
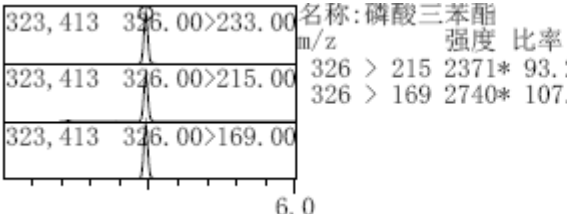
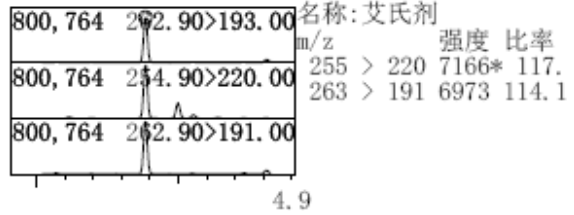
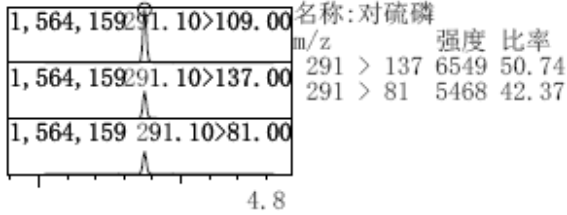
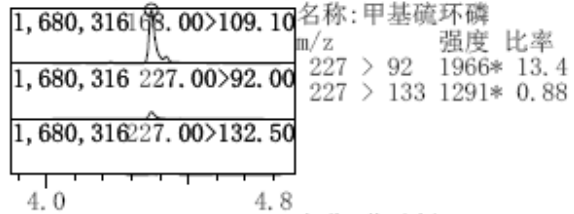
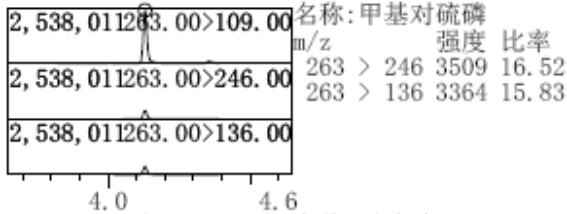
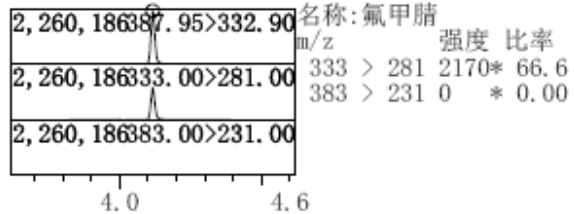
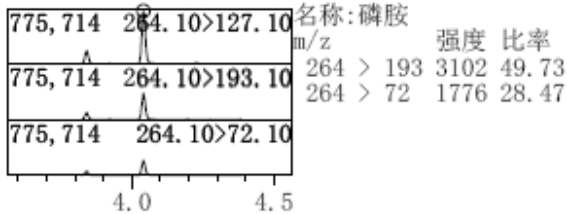
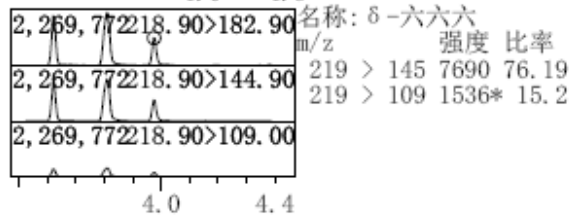
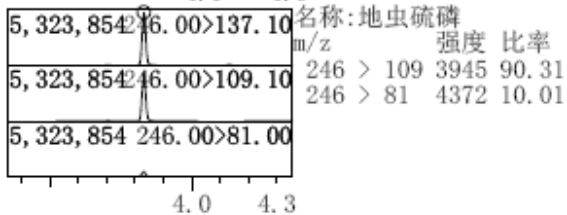
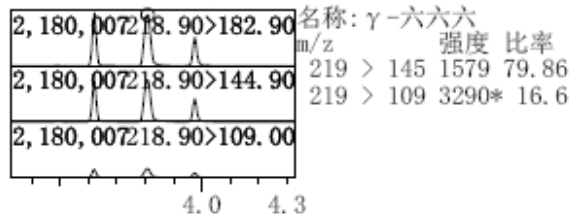
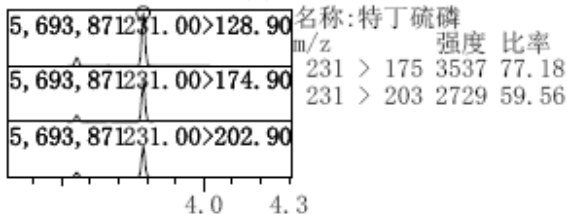
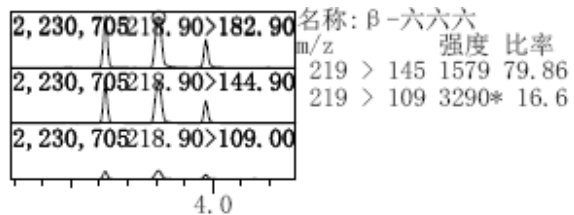
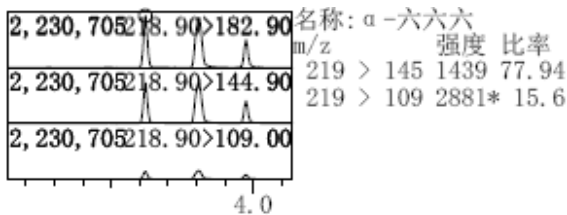
电离能量: 70 eV;

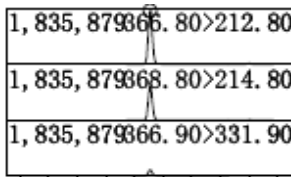
离子源温度: 250 °C;

测定方式: 多重反应监测 (MRM);

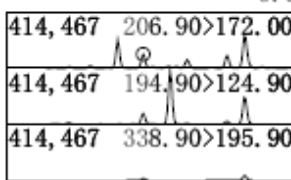
实验结果:



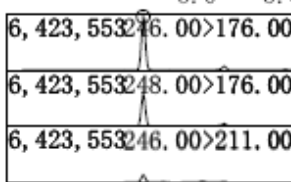




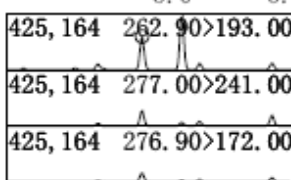
名称: 氟虫腓
m/z 强度 比率
369 > 215 1022* 63.7
367 > 332 1882 11.75



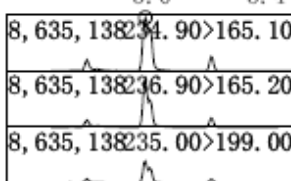
名称: α-硫丹
m/z 强度 比率
195 > 125 7083* 76.6
339 > 196 1309* 14.1



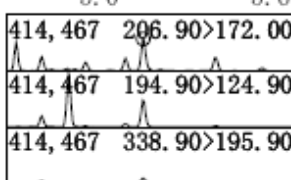
名称: pp'-DDE
m/z 强度 比率
248 > 176 3450 62.85
246 > 211 7712 14.05



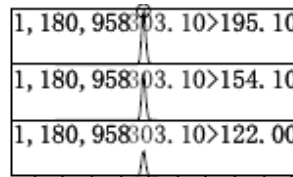
名称: 狄氏剂
m/z 强度 比率
277 > 241 1048* 45.2
277 > 172 6255* 26.9



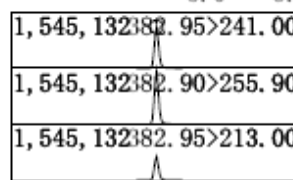
名称: pp'-DDD
m/z 强度 比率
237 > 165 6971 85.52
235 > 199 3119 38.26



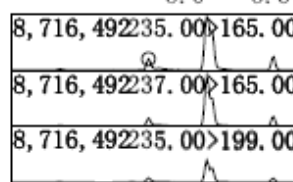
名称: β-硫丹
m/z 强度 比率
195 > 125 1800* 83.9
339 > 196 3208* 14.9



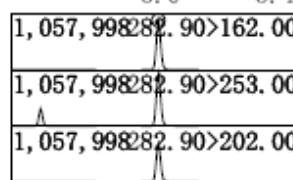
名称: 苯线磷
m/z 强度 比率
303 > 154 8727 84.27
303 > 122 4824 46.58



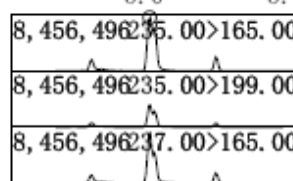
名称: 氟虫腓砜
m/z 强度 比率
383 > 256 1326* 128.
383 > 213 5940* 57.5



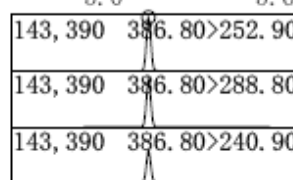
名称: op'-DDT
m/z 强度 比率
237 > 165 7326 70.13
235 > 199 4001 38.30



名称: 除草醚
m/z 强度 比率
283 > 253 9229 108.1
283 > 202 6752 79.15



名称: pp'-DDT
m/z 强度 比率
235 > 199 3100* 39.3
237 > 165 7137 90.68



名称: 硫丹硫酸酯
m/z 强度 比率
387 > 289 1117 88.38
387 > 241 7756 61.34

定量结果

| 名称 | 保留时间 | m/z | 峰面积 | 浓度 | 浓度单位 |
|---------|-------|-----------------|----------|---------|-------|
| 内吸磷 | 3.280 | 88.00 > 60.00 | 6567880 | 0.79948 | ug/mL |
| 灭线磷 | 3.361 | 200.00 > 158.00 | 2832307 | 0.80478 | ug/mL |
| 治螟磷 | 3.485 | 201.80 > 145.90 | 2527831 | 0.80089 | ug/mL |
| 久效磷 | 3.511 | 127.10 > 109.00 | 2406386 | 0.79900 | ug/mL |
| α-六六六 | 3.621 | 218.90 > 182.90 | 2005996 | 0.79837 | ug/mL |
| β-六六六 | 3.809 | 218.90 > 182.90 | 3240902 | 0.80557 | ug/mL |
| 特丁硫磷 | 3.788 | 231.00 > 128.90 | 4588153 | 0.79927 | ug/mL |
| γ-六六六 | 3.809 | 218.90 > 182.90 | 3240902 | 0.80557 | ug/mL |
| 地虫硫磷 | 3.832 | 246.00 > 137.10 | 4445369 | 0.80516 | ug/mL |
| δ-六六六 | 3.975 | 218.90 > 182.90 | 1267088 | 0.80010 | ug/mL |
| 磷胺 | 4.041 | 264.10 > 127.10 | 837836 | 0.80364 | ug/mL |
| 氟甲腈 | 4.117 | 387.95 > 332.90 | 302782 | 0.79951 | ug/mL |
| 甲基对硫磷 | 4.131 | 263.00 > 109.00 | 2322910 | 0.80902 | ug/mL |
| 甲基硫环磷 | 4.370 | 168.00 > 109.10 | 2420486 | 0.80119 | ug/mL |
| 对硫磷 | 4.372 | 291.10 > 109.00 | 1325907 | 0.81108 | ug/mL |
| 艾氏剂 | 4.386 | 262.90 > 193.00 | 644262 | 0.80521 | ug/mL |
| 水胺硫磷 | 4.404 | 289.10 > 136.00 | 241310 | 0.74653 | ug/mL |
| 三氯杀螨醇 | 4.422 | 250.00 > 139.00 | 1944147 | 0.80599 | ug/mL |
| 氟虫腈硫酸酯 | 4.500 | 419.95 > 350.90 | 2644125 | 0.80006 | ug/mL |
| 甲基异柳磷 | 4.480 | 199.00 > 121.10 | 3530606 | 0.80335 | ug/mL |
| 氟虫腈 | 4.555 | 366.80 > 212.80 | 1809759 | 0.79811 | ug/mL |
| 苯线磷 | 4.781 | 303.10 > 195.10 | 1176169 | 0.81106 | ug/mL |
| α-硫丹 | 4.812 | 206.90 > 172.00 | 98187 | 0.80804 | ug/mL |
| 氟虫腈砒 | 4.901 | 382.95 > 241.00 | 1207468 | 0.79456 | ug/mL |
| pp'-DDE | 4.898 | 246.00 > 176.00 | 5981000 | 0.80341 | ug/mL |
| op'-DDT | 4.958 | 235.00 > 165.00 | 1008236 | 0.81117 | ug/mL |
| 狄氏剂 | 4.966 | 262.90 > 193.00 | 265346 | 0.80604 | ug/mL |
| 除草醚 | 5.052 | 282.90 > 162.00 | 1006439 | 0.80631 | ug/mL |
| pp'-DDD | 5.163 | 234.90 > 165.10 | 16329004 | 0.79389 | ug/mL |
| pp'-DDT | 5.163 | 235.00 > 165.00 | 15986622 | 0.79538 | ug/mL |
| β-硫丹 | 5.168 | 206.90 > 172.00 | 263955 | 0.80998 | ug/mL |
| 硫丹硫酸酯 | 5.420 | 386.80 > 252.90 | 149315 | 0.80170 | ug/mL |
| 磷酸三苯酯 | 5.490 | 326.00 > 233.00 | 277223 | 1.00000 | ug/mL |
| 蝇毒磷 | 6.498 | 362.00 > 109.00 | 1442033 | 0.80400 | ug/mL |

