Electronic Supplementary Material (ESI) for Green Chemistry

Supplementary Materials (ESI) for

Efficient and Practical Transition Metal-Free Catalytic Hydration of Organonitriles to Amides

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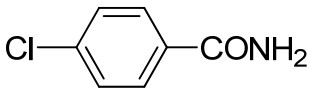
General: All commercial reagents and solvents were used directly as purchased without further purification. All reactions were carried out by CEM Discover microwave instrument unless otherwise noted. ^{1}H-NMR and ^{13}C NMR spectra were recorded on JeOL-ECA 400 spectrometers. GC-MS spectra were recorded on Agilent Technologies 1890A GC system and 5975C inert MSD with Triple-Axis Detector.

General procedure for microwave-assisted catalytic hydration of organonitriles: Organonitriles (1 mmol) and base were added into a 10 mL microwave reaction tube with a stir-bar, and then 8.5 mL deionized water or aqueous solvent was added. After irradiation under microwave at 150 ^{o}C (50 w with CEM Discover microwave instrument) for certain time, the reaction mixture was cooled and then the solvent was evaporated under reduced pressure. The residue was dissolved in acetone and dried over anhydrous sodium sulfate. The crude products were purified by flash column chromatography.

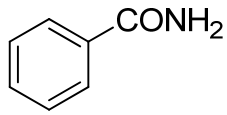
For insoluble solid amide's case, a more convenient isolation process was involved, the product was obtained by simply filtration and then washed with small amount of water to get rid of adsorptive base, which is pure enough for the GC and NMR analysis. The filtrate containing the base could be applied for the next catalytic cycle.

Analytical data of the products:

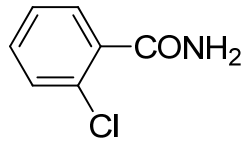


2a[S1]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.75 (d, *J* = 8.5 Hz, 2H), 7.42 (d, *J* = 8.5 Hz, 2H), 5.98 (bd, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 167.3, 136.6, 133.5, 129.9, 128.8. GC-MS: *m/z* = 157 $[M+2]^+$, 155 $[M]^+$, 141

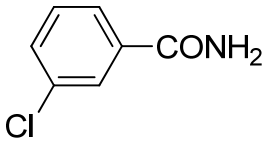
$[M+2\text{-}NH_2]^+$, 139 $[M\text{-}NH_2]^+$, 113 $[M+2\text{-}CONH_2]^+$, 111 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3379, 3177, 1667, 1619, 1573, 1411,1090, 1012 cm^{-1}.



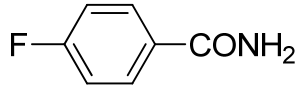
2b[S1]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.81 (d, *J* = 8.5 Hz, 2H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 6.09 (bs, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 168.0, 134.2, 131.2, 128.2, 127.5. GC-MS: *m/z* = 121 $[M]^+$, 105 $[M\text{-}NH_2]^+$, 77 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3366, 3173, 1656, 1625, 1578, 1447, 1402 cm^{-1}.



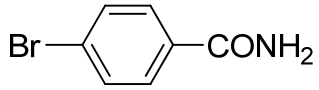
2c[S2]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.77 (dd, *J* = 7.5 and 1.6Hz, 1H), 7.30-7.50 (m, 3H), 6.38 (bs, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 168.1, 134.6, 130.5, 130.0, 129.5, 128.6, 127.0.GC-MS: *m/z* = 157 $[M+2]^+$, 155 $[M]^+$, 141 $[M+2\text{-}NH_2]^+$, 139 $[M\text{-}NH_2]^+$, 113 $[M+2\text{-}CONH_2]^+$, 111 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3368, 3195, 1637, 1616, 1404 cm^{-1}.



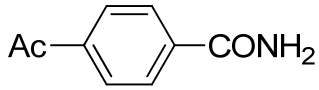
2d[S3]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.81 (t, *J* = 1.8 Hz, 1H), 7.68 (dt, *J* = 7.8 and 1.4Hz, 1H), 7.50 (dq, J_1 = 7.9 and 0.8 Hz, 1H), 7.39 (t, *J* = 7.9 Hz, 1H), 6.12 (bs, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 166.4, 136.2, 133.1, 131.0, 130.2, 127.3, 126.1. GC-MS:*m/z* = 157 $[M+2]^+$, 155 $[M]^+$, 141 $[M+2\text{-}NH_2]^+$, 139 $[M\text{-}NH_2]^+$, 113 $[M+2\text{-}CONH_2]^+$, 111 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3358, 3177, 1658, 1625, 1569, 1432, 1389, 1123 cm^{-1}.



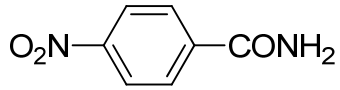
2e[S4]: ^{1}H-NMR (400 MHz, d_6-DMSO, 298 K): δ = 8.02 (bs, 1H), 7.95-7.93 (m, 2H), 7.41 (bs, 1H), 7.25 -7.30 (m, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 167.3, 165.7, 163.2, 130.7, 130.6, 115.8, 115.5. GC-MS: *m/z* = 139 $[M]^+$, 123 $[M\text{-}NH_2]^+$, 95 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3325, 3147, 1676, 1624, 1590, 1512, 1416, 1400 cm^{-1}.



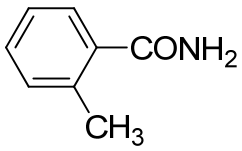
2f[S1]: ^{1}H-NMR (400 MHz, d_6-DMSO, 298 K): δ = 8.03 (bs, 1H), 7.81 (d, *J* = 8.5 Hz, 2H), 7.66 (d, *J* = 8.5 Hz, 2H), 7.46 (bs, 1H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 166.9, 133.3, 131.2, 129.6, 125.0. GC-MS: *m/z* = 201 $[M+2]^+$, 199$[M]^+$, 185 $[M+2\text{-}NH_2]^+$, 183 $[M\text{-}NH_2]^+$, 157 $[M+2\text{-}CONH_2]^+$, 155 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3357, 3174, 1705, 1659, 1622, 1407, 1146 cm^{-1}.



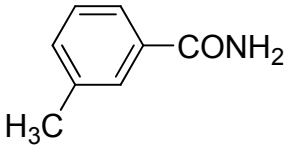
2g[S5]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 8.03 (d, *J* = 8.3 Hz, 2H), 7.90 (d, *J* = 8.3 Hz, 2H), 6.12 (bs, 1H), 5.71 (bs, 1H), 2.65 (s, 3H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 197.7, 167.1, 138.6, 138.0, 128.1, 127.7, 26.9. GC-MS: *m/z* = 163 $[M]^+$, 148 $[M\text{-}CH_3]^+$, 120. IR (KBr): v = 3400, 3190, 1680, 1656, 1416, 1269 3368, 3195, 1637, 1616, 1404 cm^{-1}.



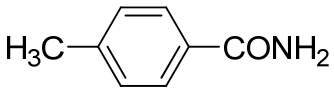
2h[S1]: ^{1}H-NMR (400 MHz, d_6-DMSO, 298 K): δ = 8.31-8.29 (m, 3H), 8.09 (d, *J* = 8.0 Hz, 2H), 7.75 (bs, 1H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 166.2, 149.0, 139.9, 128.9, 123.4. GC-MS: *m/z* = 166 $[M]^+$, 150 $[M\text{-}NH_2]^+$, 120, 76. IR (KBr): v = 3473, 3414, 3308, 3178, 1669, 1591, 1514, 1339 cm^{-1}.



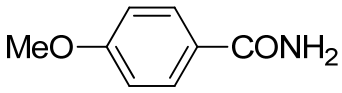
2i[S1]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.45 (dd, *J* = 7.4 and 1.2 Hz, 1H), 7.33 (dt, *J* = 7.5 and 1.2 Hz, 1H), 7.19-7.28 (m, 2H), 5.90 (bd, 2H), 2.50 (s, 3H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 171.0, 137.0, 135.0, 130.4, 129.1, 126.9, 125.3, 19.5. GC-MS: *m/z* = 135 $[M]^+$, 119 $[M\text{-}NH_2]^+$, 91 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3364, 3177, 2803, 1637, 1496, 1454, 1416, 1283 cm^{-1}.



2j[S1]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.65 (s, 1H), 7.58 (d, *J* = 4.0 Hz, 1H), 7.33 (m, 2H), 6.07 (bd, 2H), 2.40 (s, 3H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 168. 1, 137.4, 134.2, 131.8, 128.1, 124.6, 20.9. GC-MS: *m/z* = 135 $[M]^+$, 119 $[M\text{-}NH_2]^+$, 91 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3374, 3194, 2917, 1648, 1615, 1385, 1112 cm^{-1}.

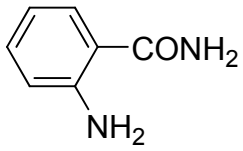


2k[S1]: ^{1}H NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.71 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 6.04 (bs, 2H), 2.40 (s, 3H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 168.3, 141.6, 132.0, 129.2, 128.0, 21.5. GC-MS: *m/z* = 135 $[M]^+$, 119 $[M\text{-}NH_2]^+$, 91 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3336, 3155, 1672, 1616, 1413 cm^{-1}.

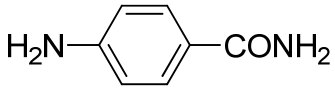


2l[S1]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.78 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 5.91 (bs, 2H), 3.85 (s, 3H). ^{13}C-NMR (100 MHz, d_6-DMSO,

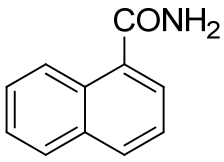
298 K): δ = 168.0, 162.1, 129.9, 127.0, 113.9, 55.8. GC-MS: *m/z* = 151 $[M]^+$, 135 $[M\text{-}NH_2]^+$, 107 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3388, 3168, 2970, 1643, 1572, 1395 cm^{-1}.



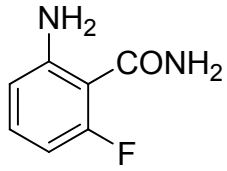
2m[S6]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.36 (dd, *J* = 8.0 and 1.2 Hz, 1H), 7.23 (dt, *J* = 7.7 and 1.2 Hz, 1H), 6.61-6.70 (m, 2H), 5.50-6.00 (m, 4H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 171.3, 150.2, 131.9, 128.7, 116.4, 114.3, 113.6. GC-MS: *m/z* = 136 $[M]^+$, 119 $[M\text{-}NH_3]^+$, 92 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3413, 3319, 3189, 1664, 1628, 1609, 1536, 1401, 1315 cm^{-1}.



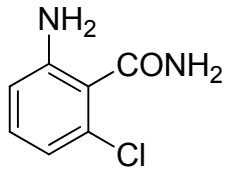
2n[S7]: ^{1}H-NMR (400 MHz, d_6-DMSO, 298 K): δ = 7.57 (d, *J* = 8.4Hz, 2H), 7.50 (bs, 1H), 6.80 (bs, 1H), 6.51 (d, *J* = 8.8 Hz, 2H), 5.59 (bs, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 168.6, 152.2, 129.6, 121.4, 113.0. GC-MS: *m/z* = 136 $[M]^+$, 120 $[M\text{-}NH_2]^+$, 92 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3466, 3350, 3215, 1604, 1389, 1291 cm^{-1}.



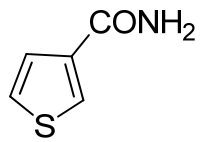
2o[S2]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 8.42 (d, *J* = 8.1 Hz, 1H), 7.95 (d, *J* = 8.2 Hz, 1H), 7.88 (d, *J* = 7.6 Hz, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.47-7.63 (m, 2H), 7.46 (d, *J* = 8.0 Hz, 1H), 6.01 (bs, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 170.5, 134.6, 133.1, 129.7, 129.6, 128.1, 126.6, 126.1, 125.5, 125.1, 124.9. GC-MS: *m/z* = 171 $[M]^+$, 155 $[M\text{-}NH_2]^+$, 127 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3331, 3157, 3048, 1663, 1615, 1507, 1414, 1364, 1151, 1121 cm^{-1}.



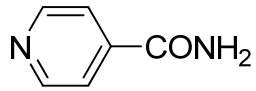
2p[S8]: ^{1}H NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.11 (q, *J* = 7.6 Hz, 1H), 6.67 (bs, 1H), 6.44 (d, *J* = 8.3 Hz, 1H), 6.34 (dd, *J* = 13.2 and 8.0 Hz, 1H), 6.12 (bs, 2H), 5.77 (bs, 1H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 166.7, 162.0, 159.5, 150.5, 150.4, 131.3, 131.2, 111.6, 101.6, 101.4. GC-MS: *m/z* = 154 $[M]^+$, 137 $[M\text{-}NH_3]^+$, 110 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3487, 3423, 1646, 1635, 1455, 1406, 1295 cm^{-1}.



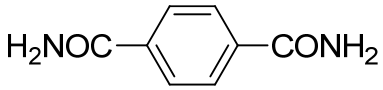
2q[S8]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.06 (t, *J* = 8.1 Hz, 1H), 6.72 (d, *J* = 7.8 Hz, 1H), 6.59 (d, *J* = 8.2 Hz, 1H), 6.21 (bs, 2H), 4.82 (bs, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 167.5, 147.0, 129.9, 129.8, 121.6, 116.1, 113.6. GC-MS: *m/z* = 172 $[M+2]^+$, 170 $[M]^+$, 155 $[M+2\text{-}NH_3]^+$, 153 $[M\text{-}NH_3]^+$, 128 $[M+2\text{-}CONH_2]^+$, 126 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3474, 3429, 3329, 3160, 1650, 1569, 1470, 1449, 1395 cm^{-1}.



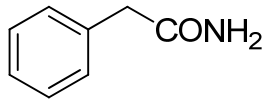
2r[S2]: ^{1}H-NMR (400 MHz, d_6-DMSO, 298 K) : δ = 8.12 (dd, *J* = 2.9 and 1.2 Hz, 1H), 7.78 (bs, 1H), 7.44-7.56 (m, 2H), 7.22 (bs, 1H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 164.3, 137.9, 129.5, 127.4, 127.0. GC-MS: *m/z* = 127 $[M]^+$, 111 $[M\text{-}NH_2]^+$, 83 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3360, 3169, 1658, 1611, 1435 cm^{-1}.



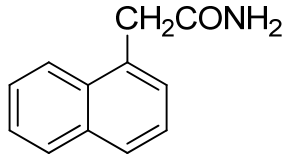
2s[S1]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 8.77 (d, *J* = 4.7 Hz, 2H), 7.65 (d, *J* = 4.6 Hz, 2H), 6.14 (bd, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 166.3, 150.2, 141.2, 121.4. GC-MS: *m/z* = 122 $[M]^+$, 106 $[M\text{-}NH_2]^+$, 78 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3341, 3057, 1682, 1626, 1552, 1388 cm^{-1}.



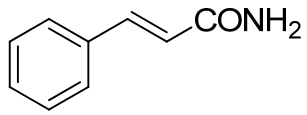
2t[S1]: ^{1}H-NMR (400 MHz, d_6-DMSO, 298 K) : δ = 8.08 (bs, 2H), 7.93 (s, 4H), 7.50 (bs, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 167.8, 137.1, 127.9. MS: *m/z* = 164 $[M]^+$, 148 $[M\text{-}NH_2]^+$, 120 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3360, 3162, 2779, 1661, 1618, 1410, 1388, 1129 cm^{-1}.



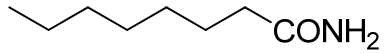
2u[S9]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K): δ = 7.10-7.76 (m, 5H), 5.98 (bs, 1H), 5.47 (bs, 1H), 3.57 (s, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 172.2, 136.5, 129.0, 128.1, 126.2, 42.2. GC-MS: *m/z* = 135 $[M]^+$, 91 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3364, 3177, 2803, 1637, 1496, 1454, 1416, 1283 cm^{-1}.



2v[S10]: ^{1}H-NMR (400 MHz, d_6-DMSO, 298 K) : δ = 7.99 (d, *J* = 8.4 Hz, 1H), 7.82-7.90 (m, 2H), 7.58-7.32 (m, 5H), 7.01 (s, 1H), 3.87 (s, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 172.1, 133.3, 132.9, 132.0, 128.3, 127.8, 126.9, 125.9, 125.6, 125. 5, 124.3, 39.6. GC-MS: *m/z* = 185 $[M]^+$, 141 $[M\text{-}CONH_2]^+$. IR (KBr): v = 3434, 3376, 3185, 1662, 1619, 1397 cm^{-1}.



2w[S1]: ^{1}H-NMR (400 MHz, $CDCl_3$, 298 K) : δ = 7.64 (d, *J* = 15.7 Hz, 1H), 7.51 (q, *J* = 3.4 Hz, 2H), 7.37 (t, *J* = 3.3 Hz, 3H), 6.46 (d, *J* = 15.7 Hz, 1H), 5.78 (bs, 2H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 166.7, 139.1, 134.8, 129.4, 128.9, 127.5, 122.3. GC-MS: *m/z* = 147 $[M]^+$, 146 $[M-H]^+$, 131 $[M-NH_2]^+$, 103 $[M-CONH_2]^+$, 77. IR (KBr): v = 3370, 3166, 1662, 1607, 1398 cm^{-1}.



2x[S6]: ^{1}H-NMR (400 MHz, d_6-DMSO, 298 K) : δ = 7.26 (bs, 1H), 6.67 (bs, 1H), 2.01 (t, *J* = 7.6 Hz, 2H), 1.46 (m, 2H), 1.24 (m, 8H), 0.85 (t, *J* = 6.0 Hz, 3H). ^{13}C-NMR (100 MHz, d_6-DMSO, 298 K): δ = 174.3, 35.0, 31.1, 28.6, 28.4, 25.0, 22.0, 13.9. GC-MS: *m/z* = 143 $[M]^+$, 127 $[M-NH_2]^+$, 114 $[M-CH_3CH_2]^+$. IR (KBr): v = 3429, 2955, 2924, 2866, 1659, 1633, 1469, 1428, 1412 cm^{-1}.

References

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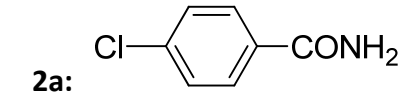

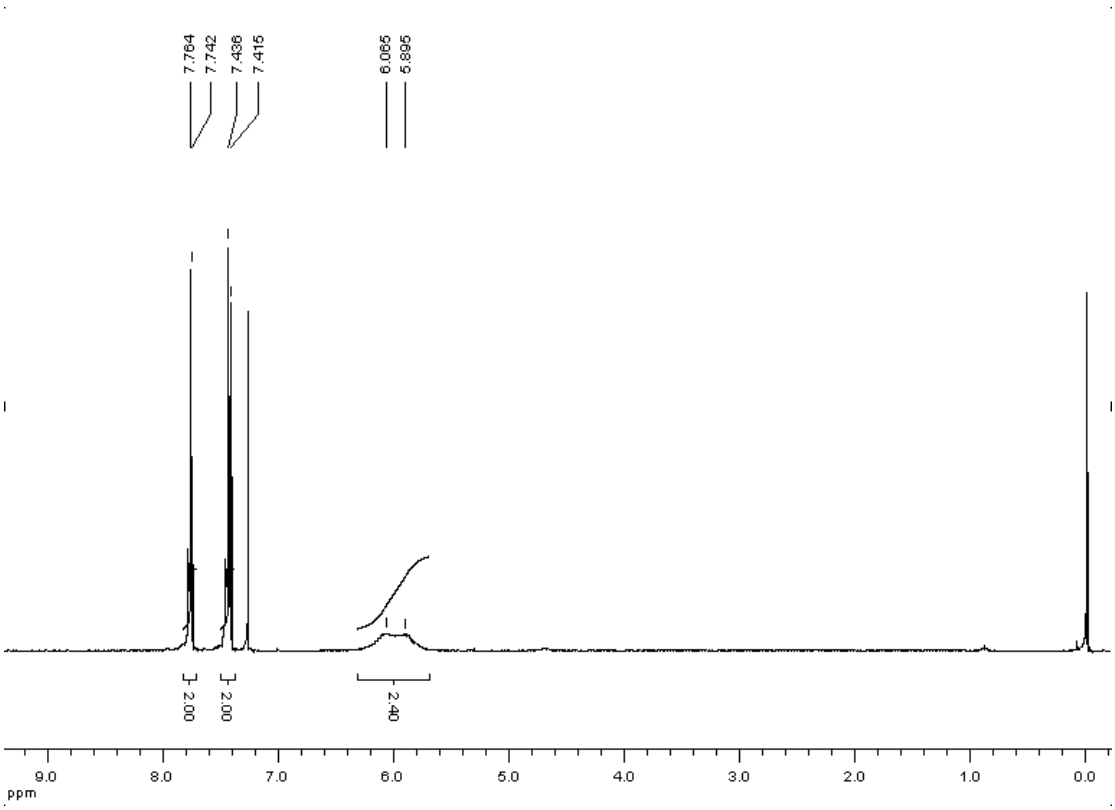

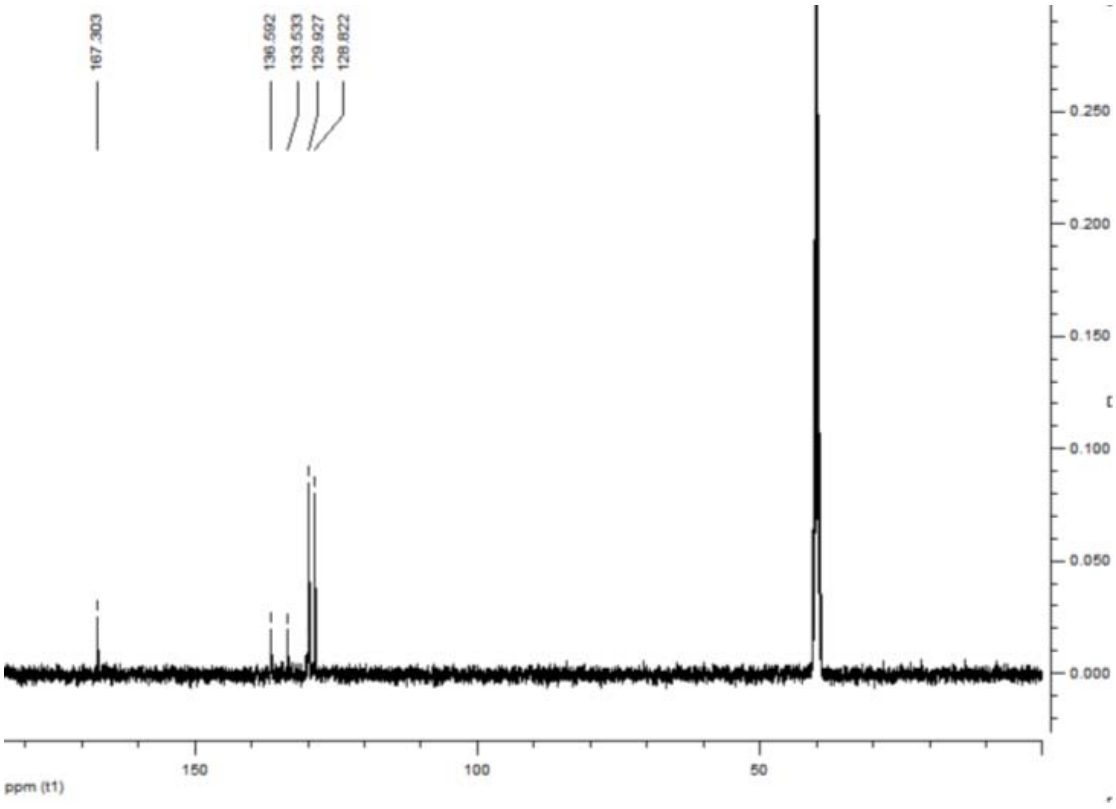

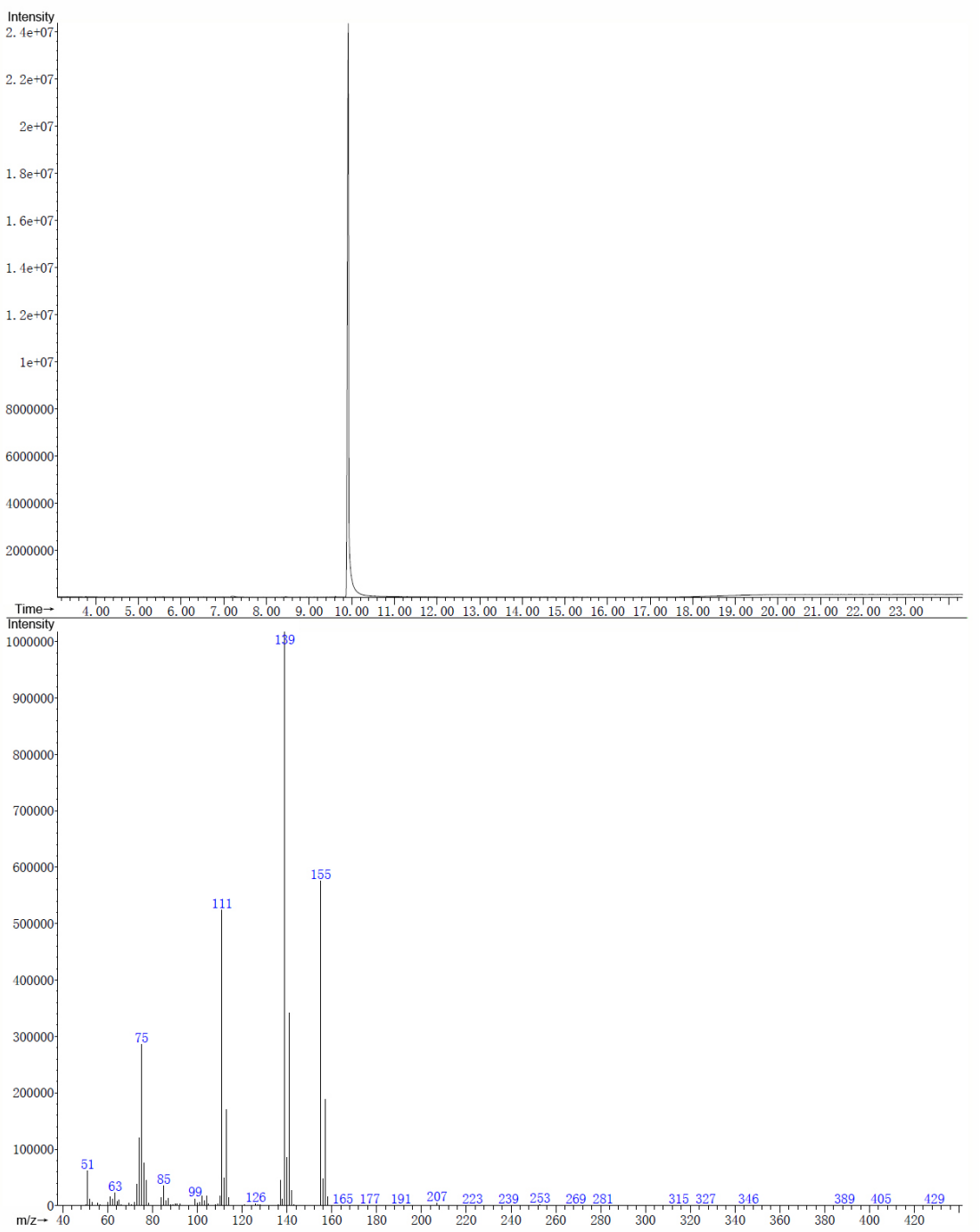
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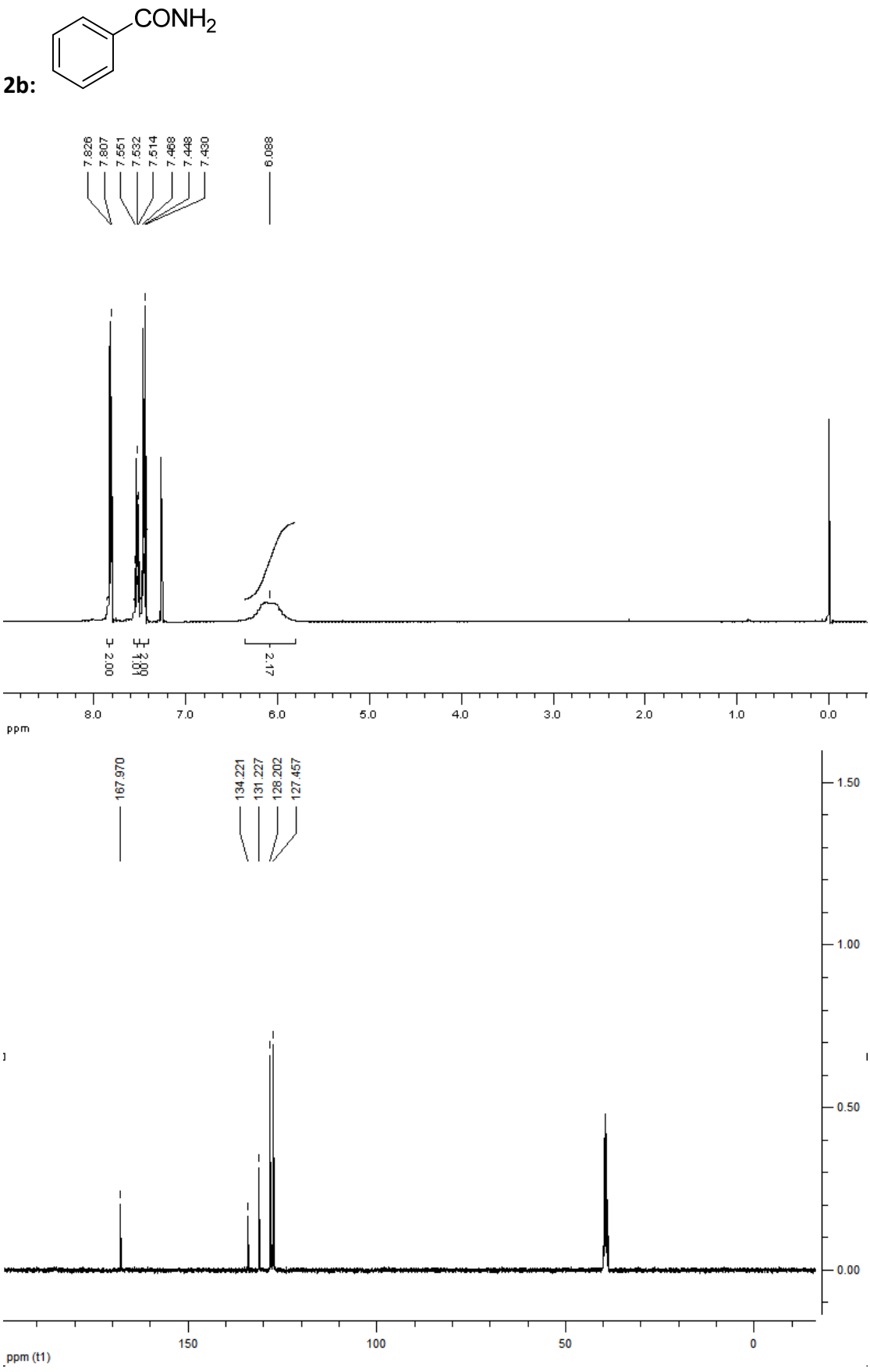
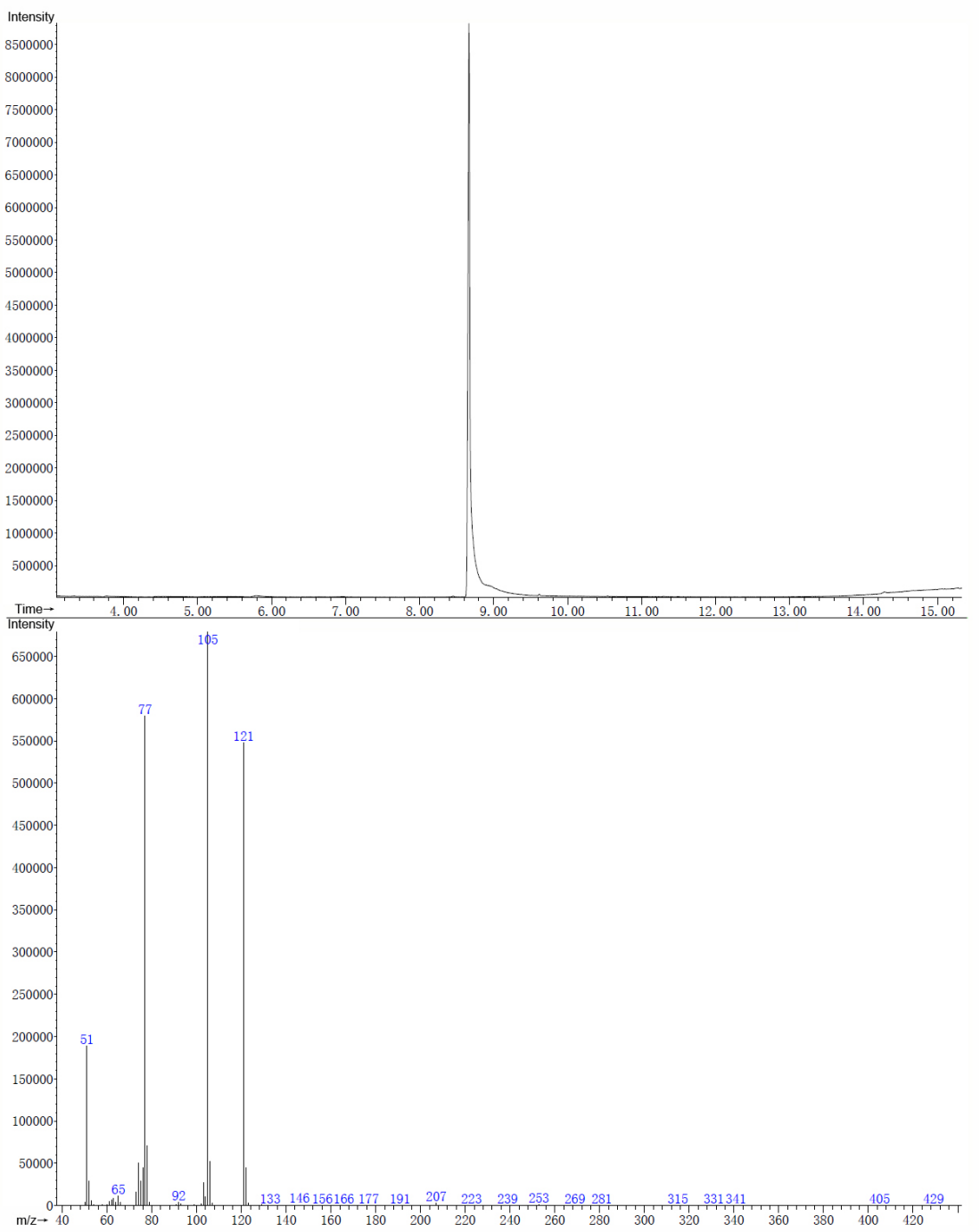
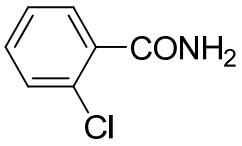
2.2e+07
2e+07
1.8e+07
1.6e+07
1.4e+07
1.2e+07
1e+07
8000000
6000000
4000000
2000000
Time→
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00 21.00 22.00 23.00
Intensity
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
m/z→
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420
139
155
111
75
51
63
85
99
126
165 177 191 207 223 239 253 269 281 315 327 346 389 405 429

2b:







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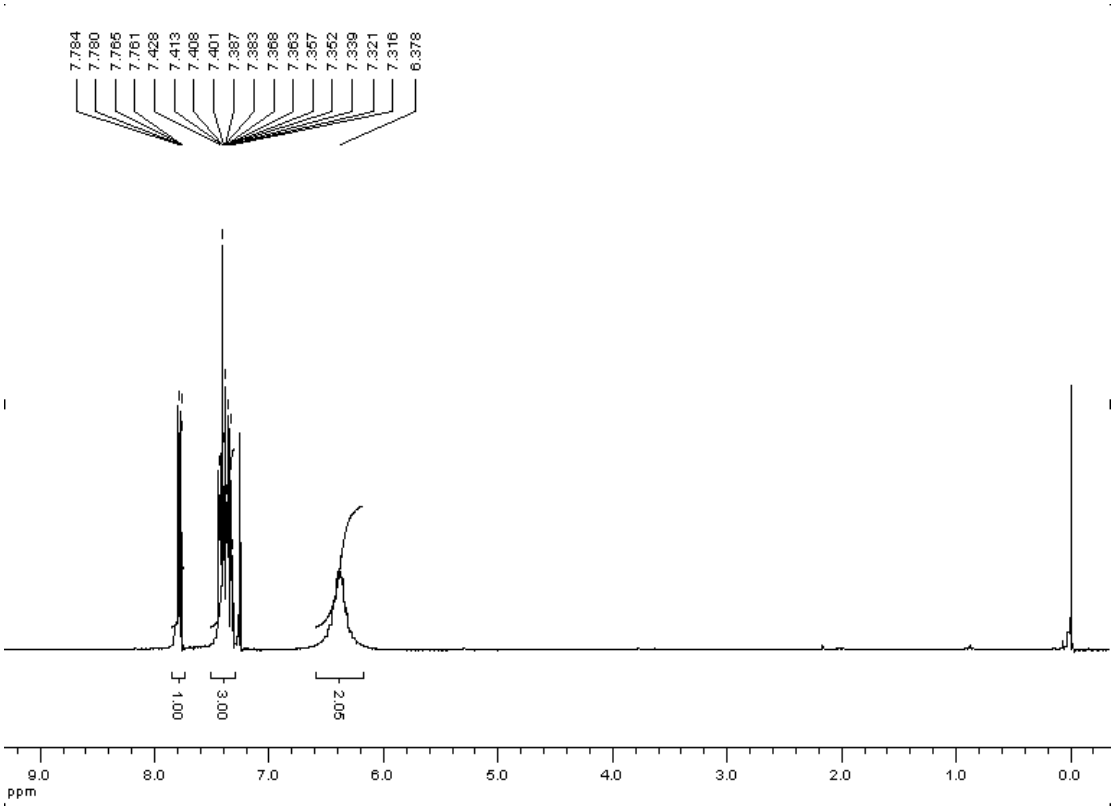

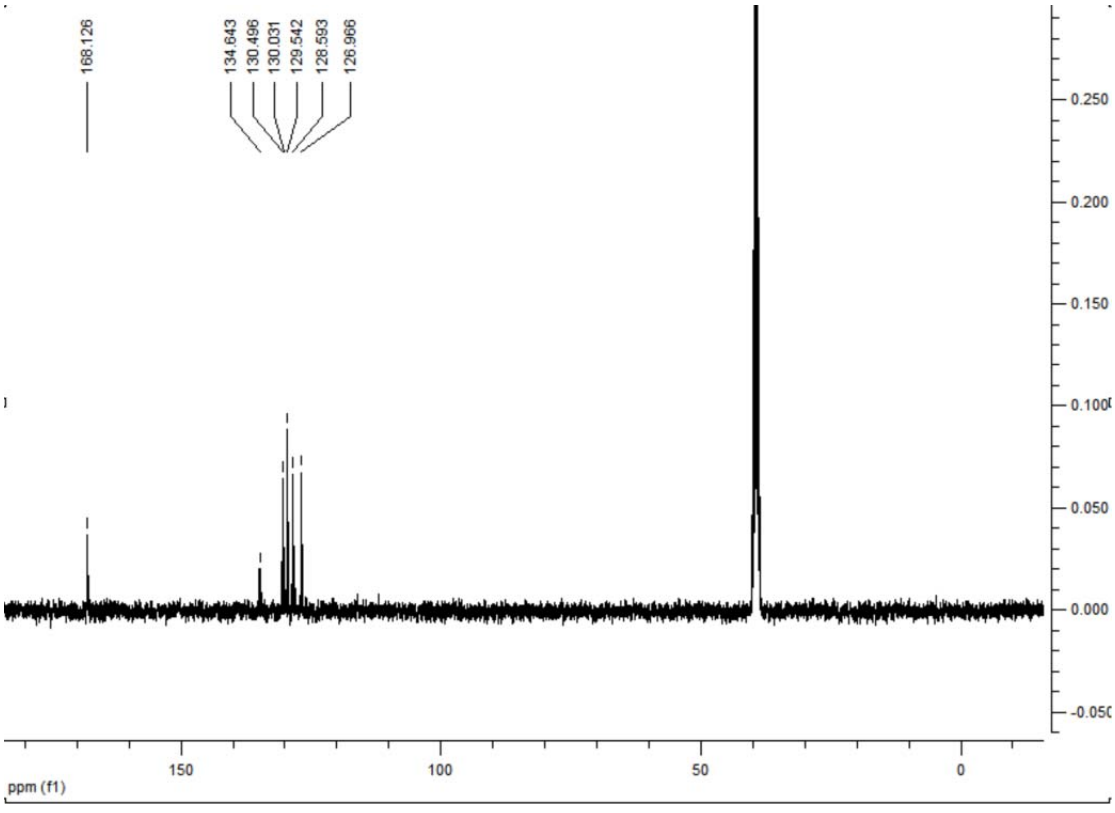

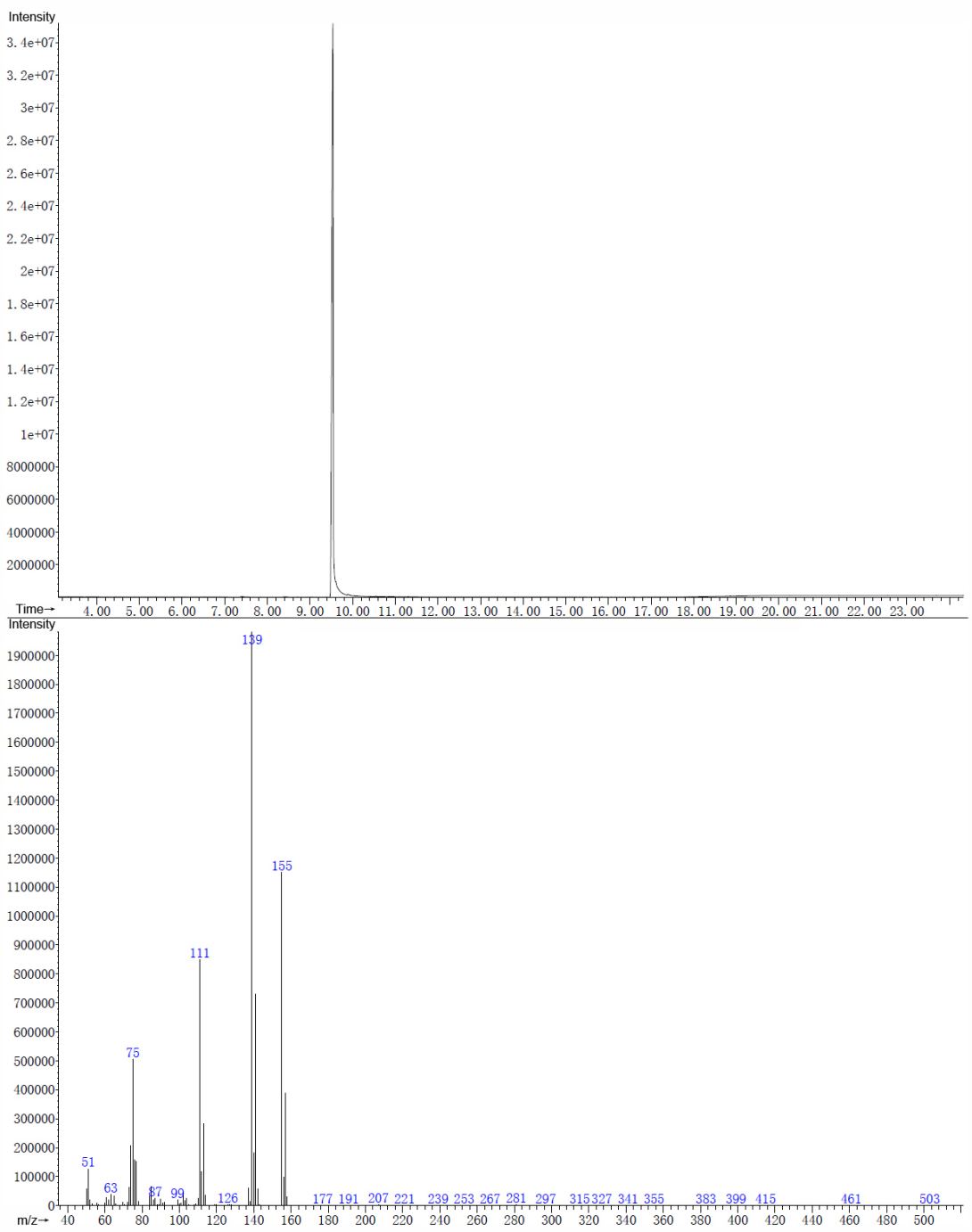

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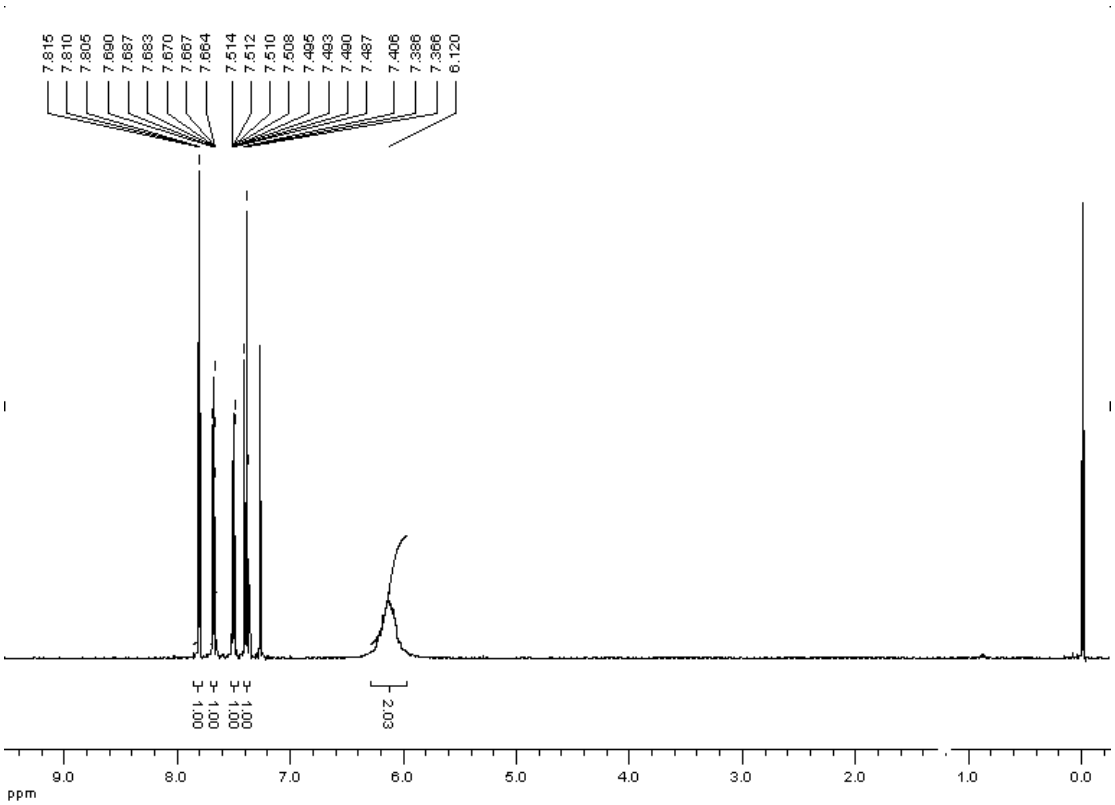

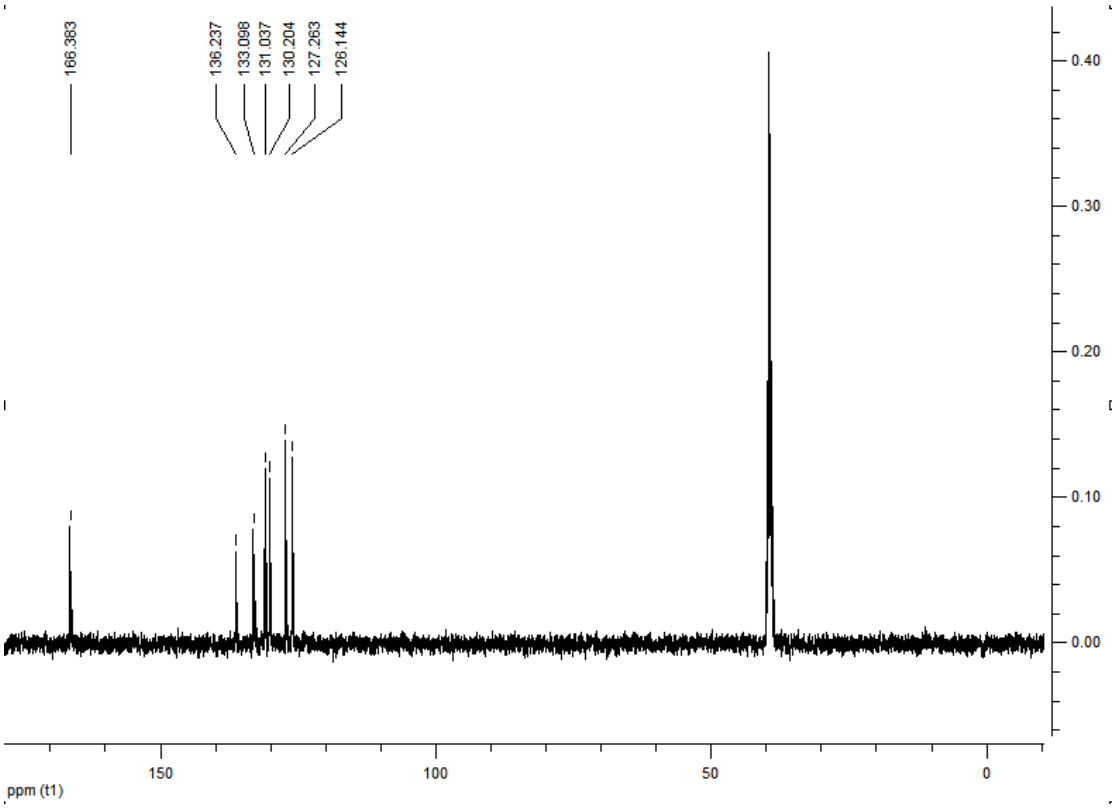

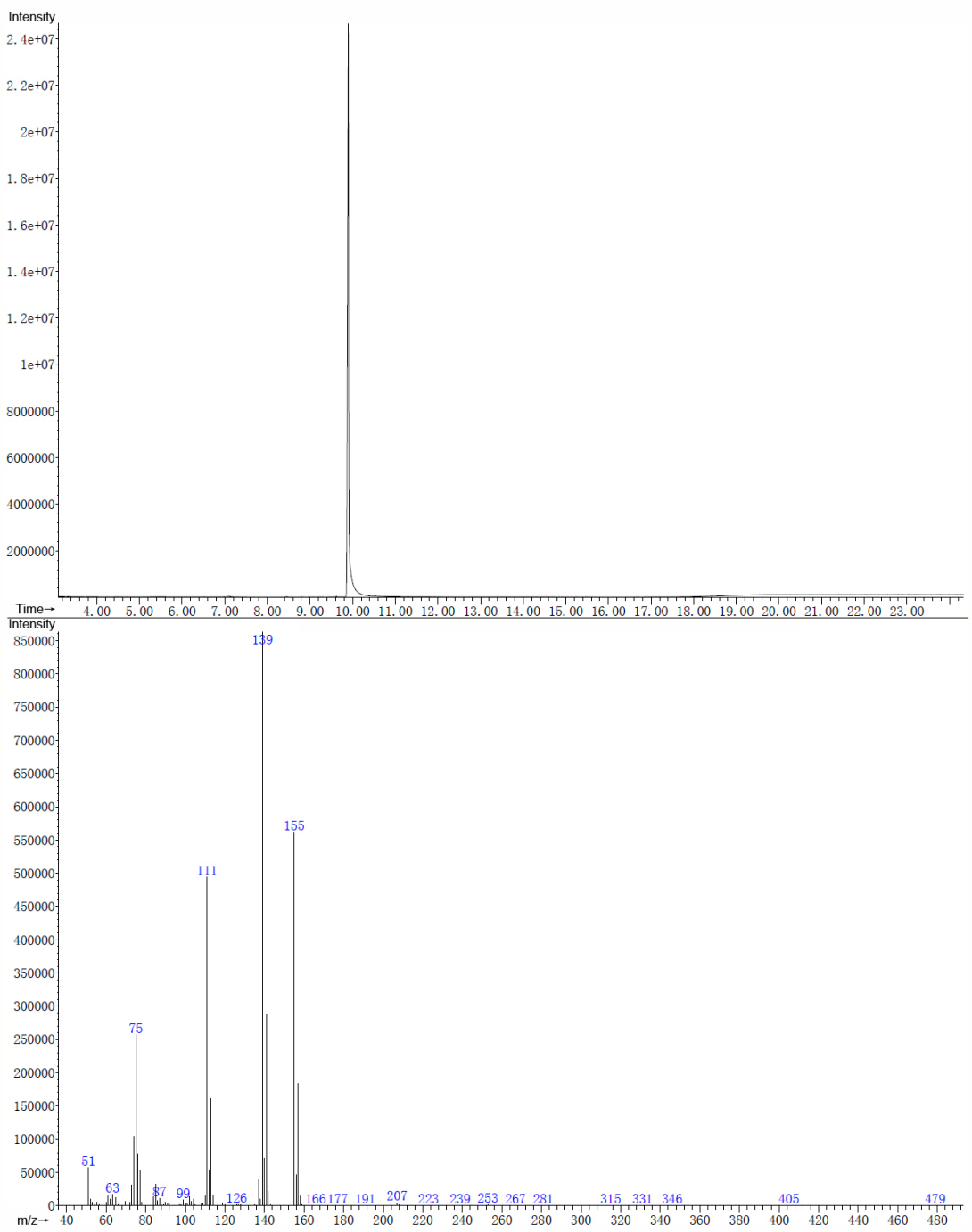

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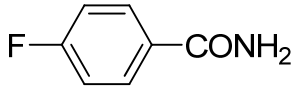
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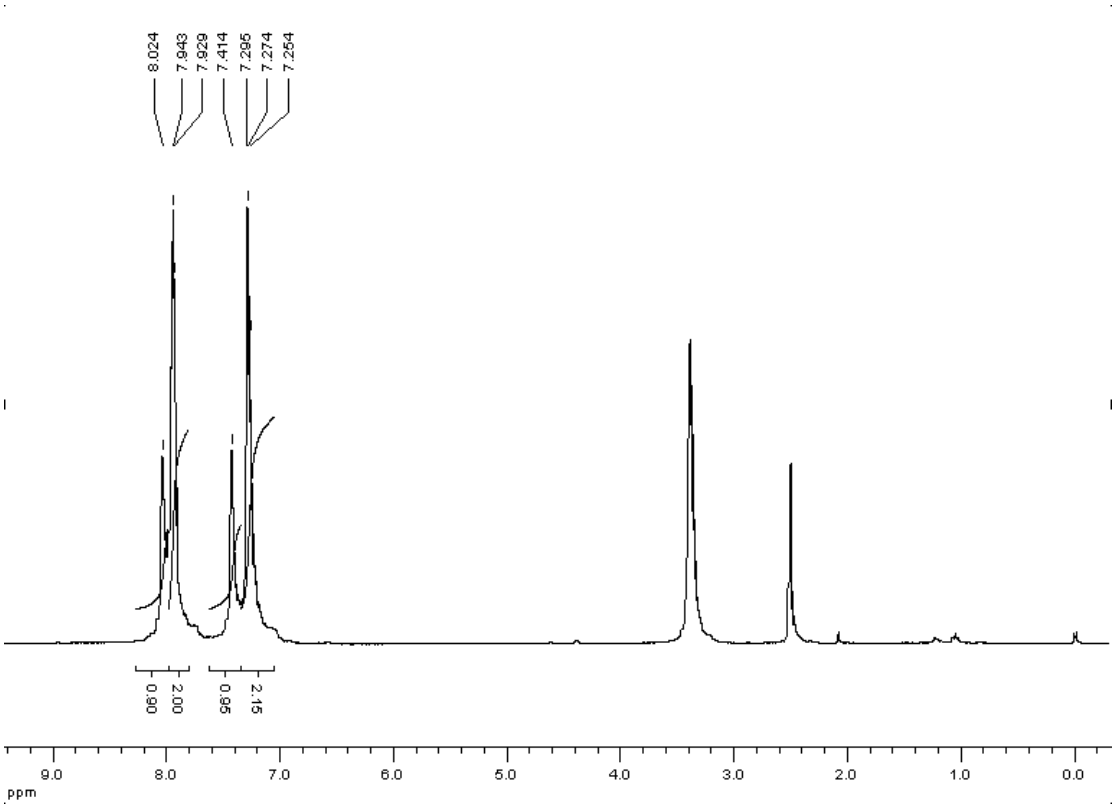
CONH2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7.929
7.414
7.295
7.274
7.254
0.90
2.00
0.96
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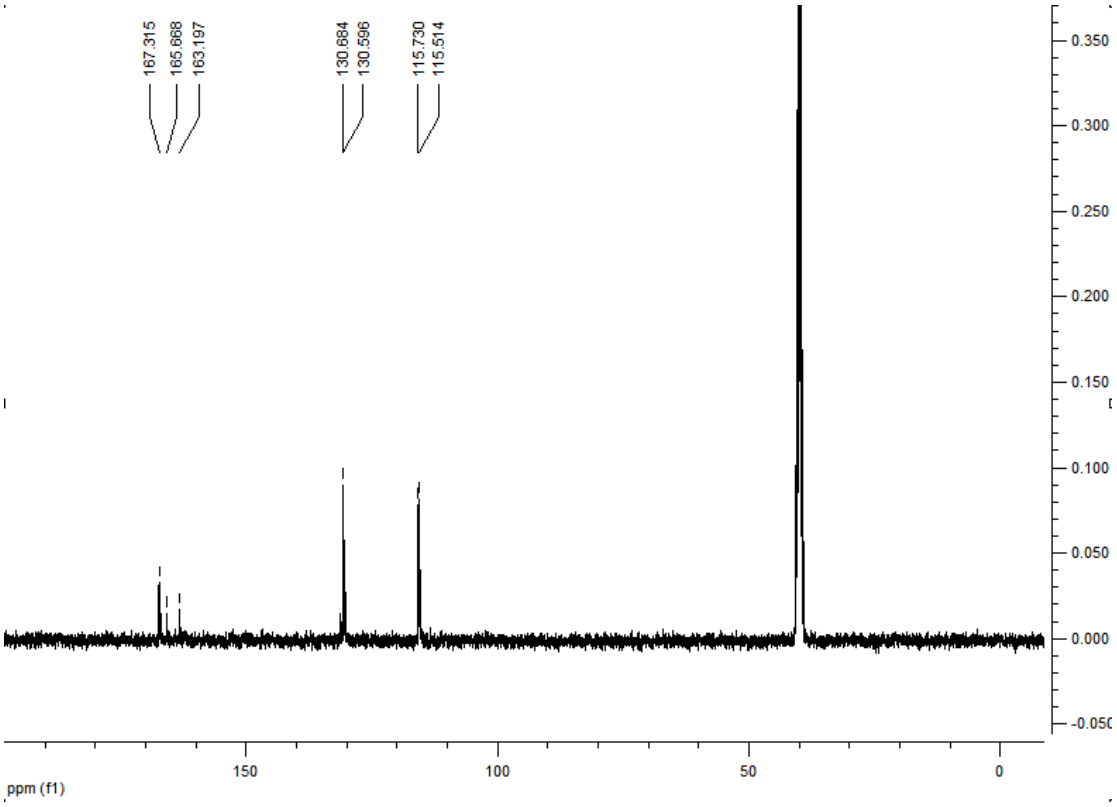
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm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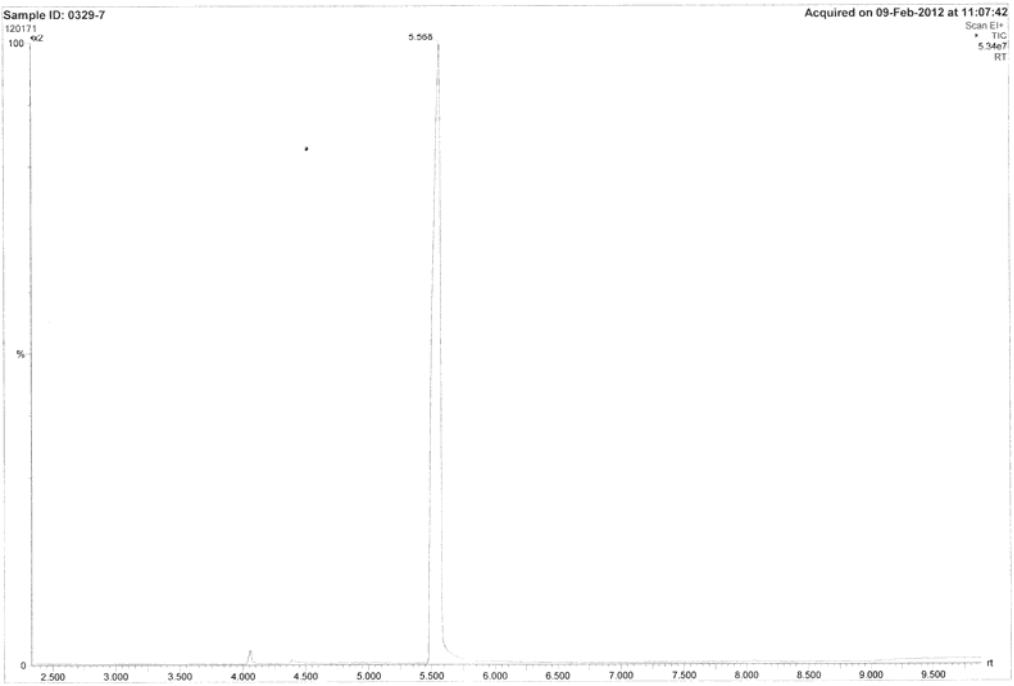
165.668
163.197
130.684
130.596
115.730
115.514
0.350
0.300
0.250
0.200
0.150
0.100
0.050
0.000
-0.050
150
100
50
0
ppm (f1)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Scan EI+
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RT
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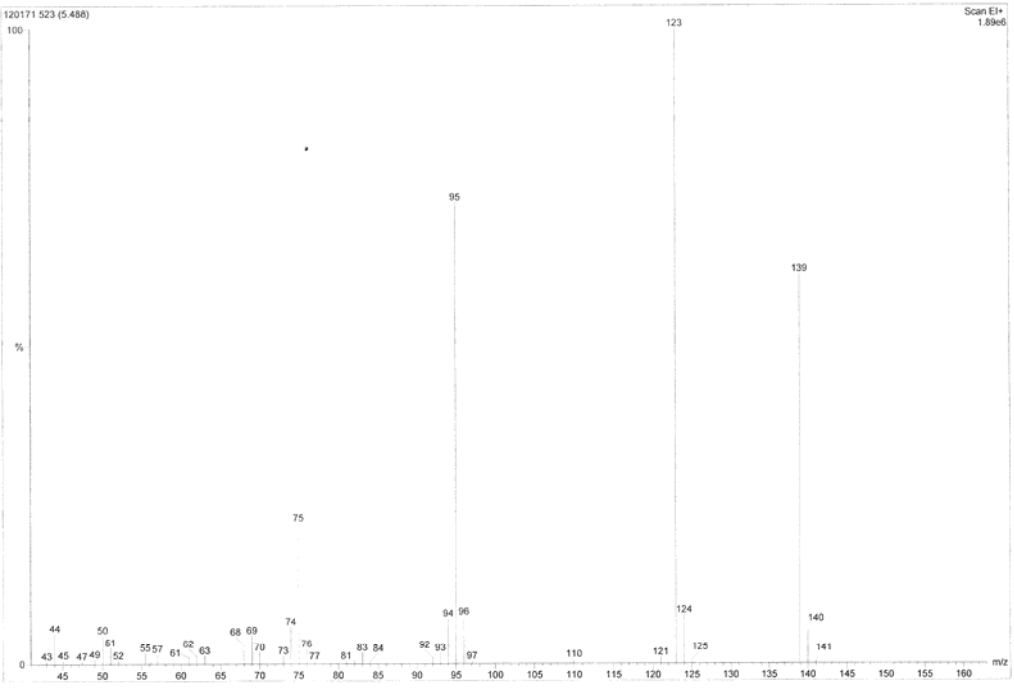
%
rt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Scan EI+
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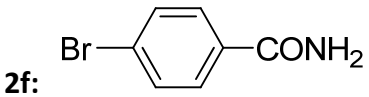
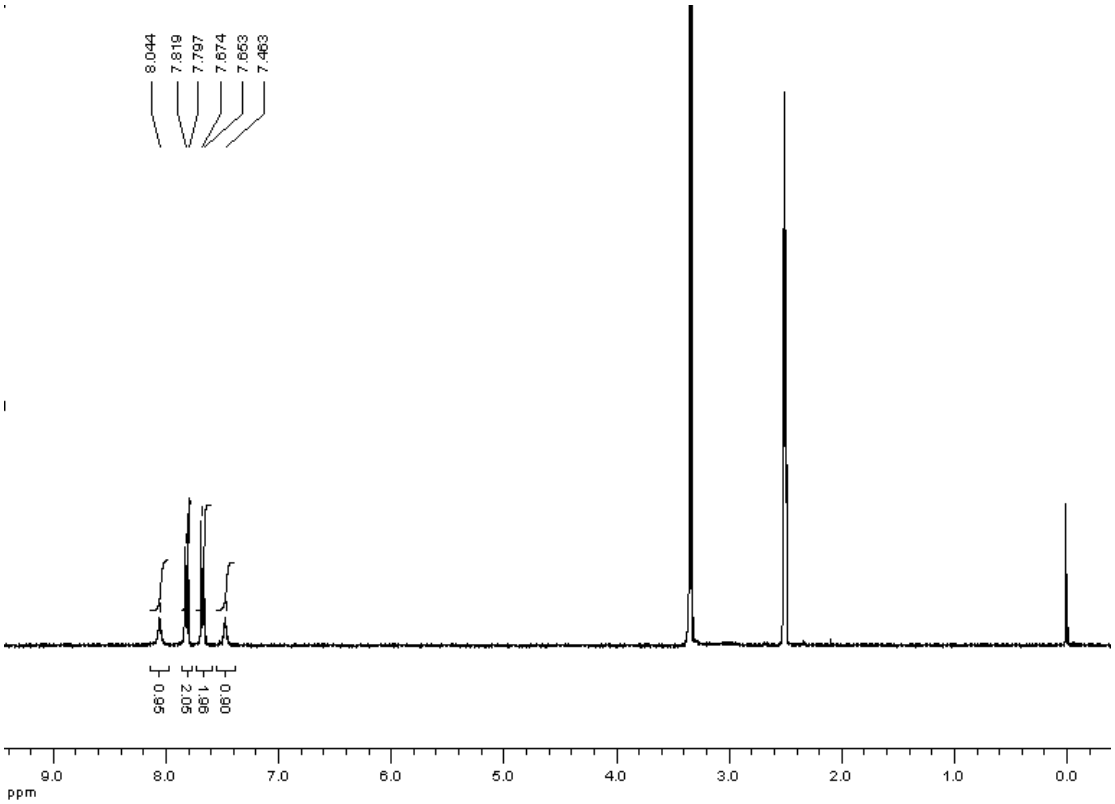
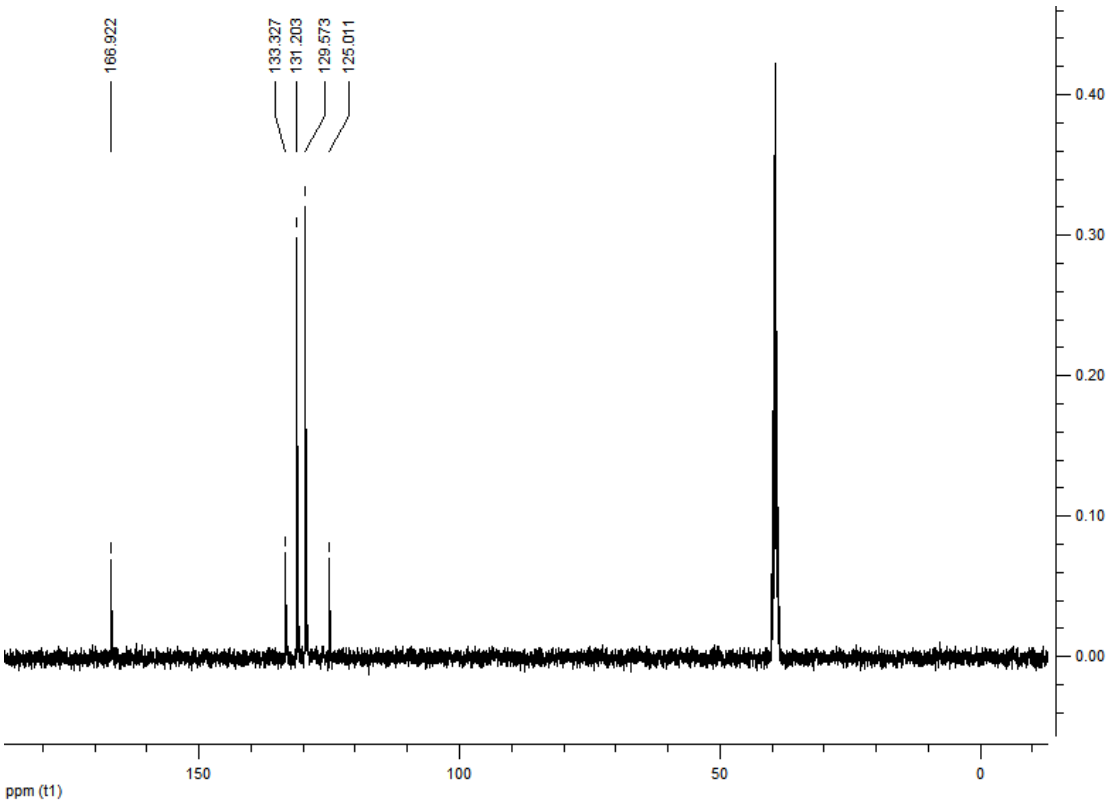
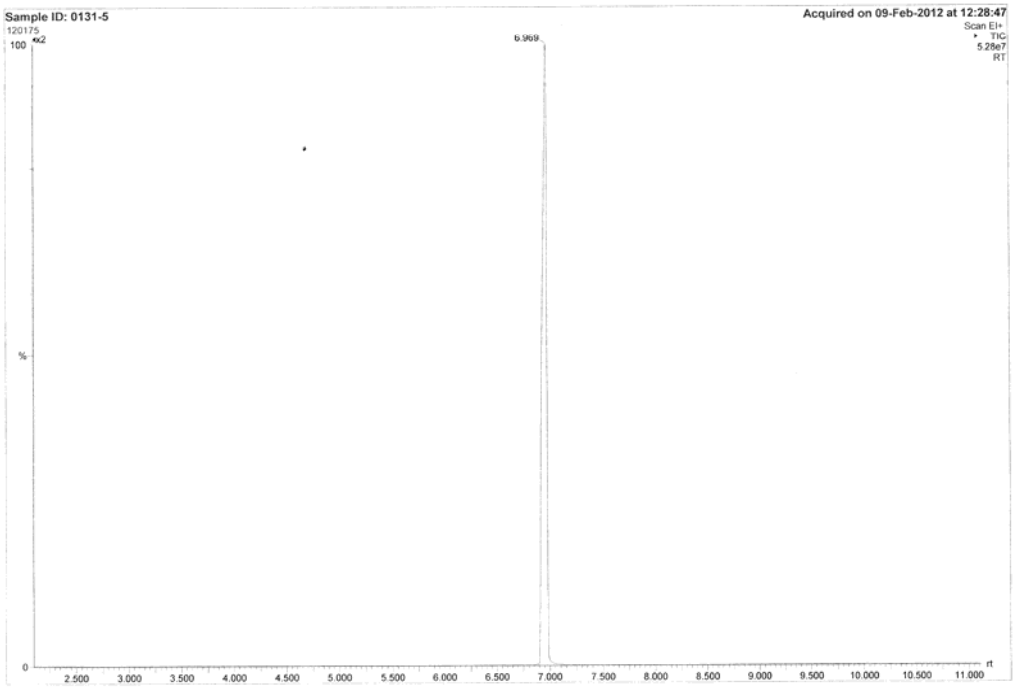
123
95
139
75
%
m/z

2f:





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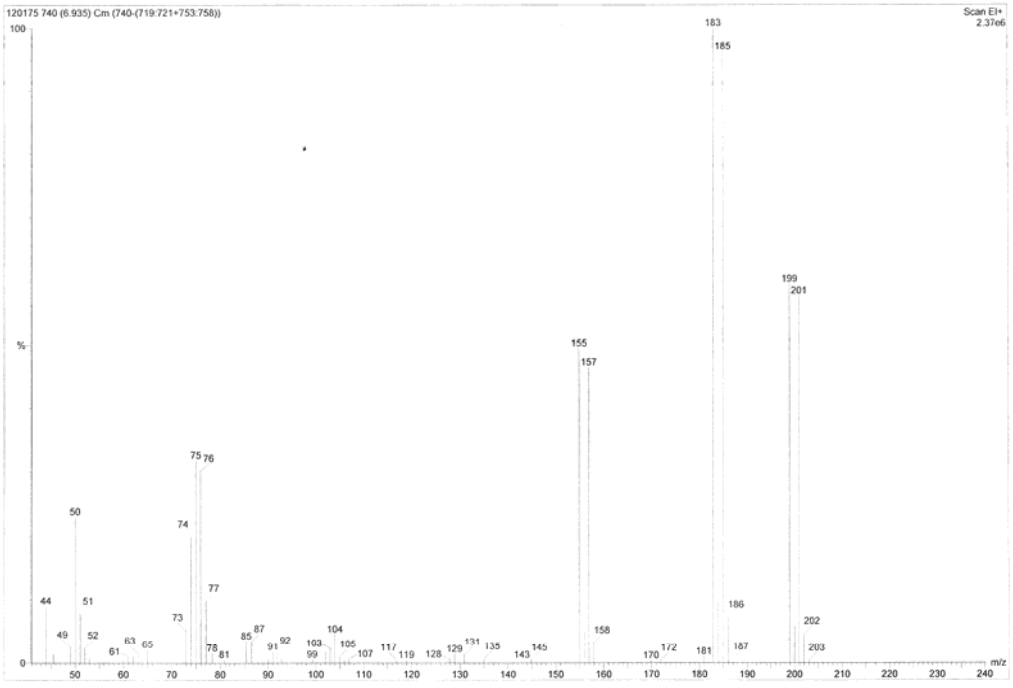
%
rt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Scan EI+
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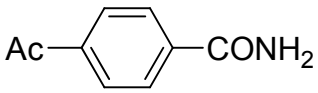
%
m/z

2g: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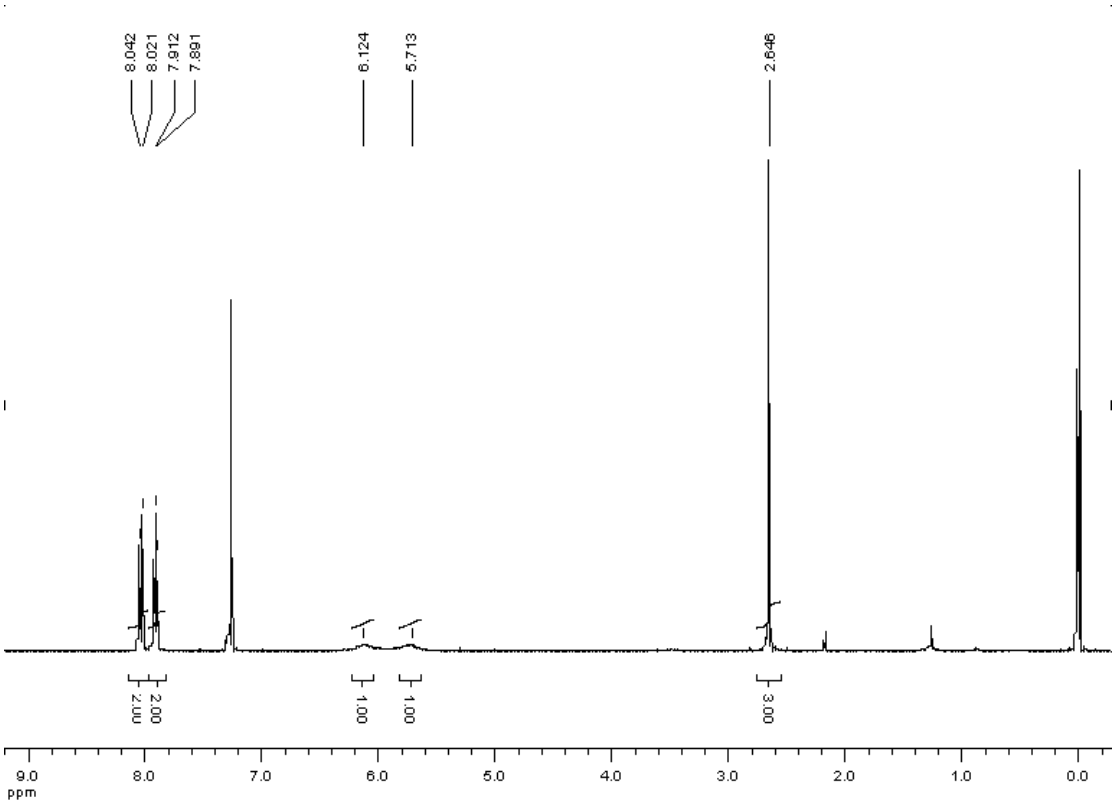
CONH2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2.00
1.00
1.00
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8.0
7.0
6.0
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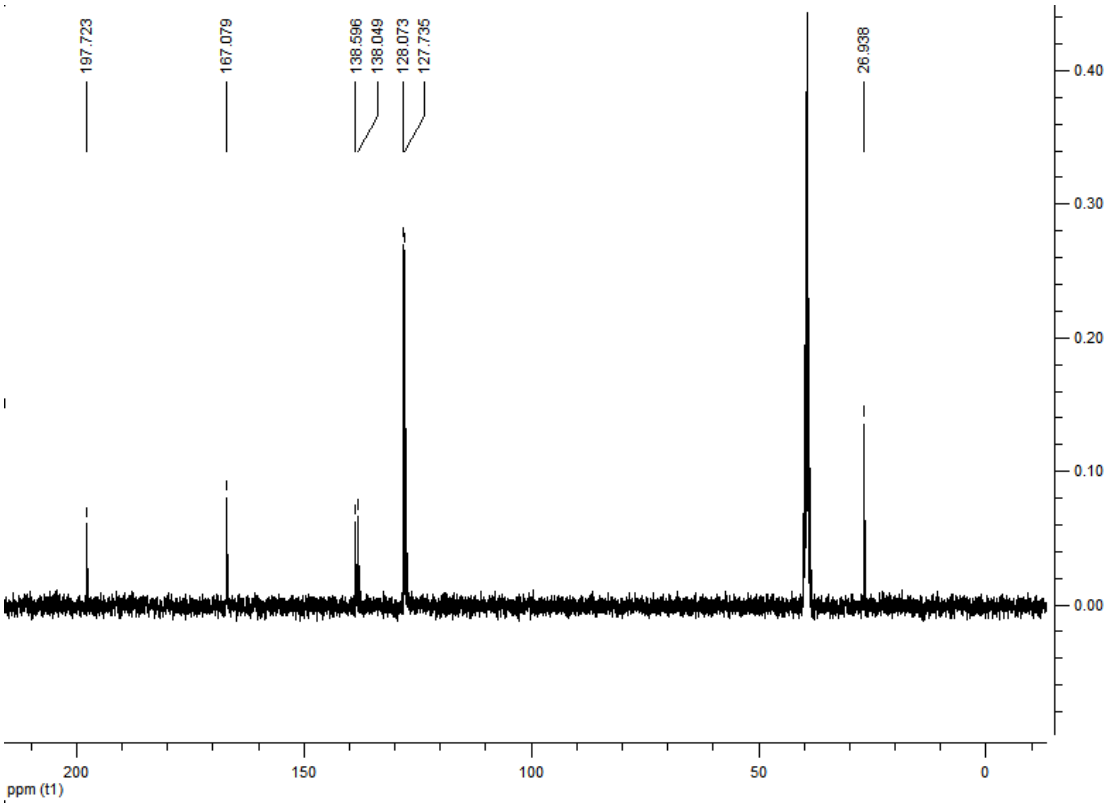
3.0
2.0
1.0
0.0
ppm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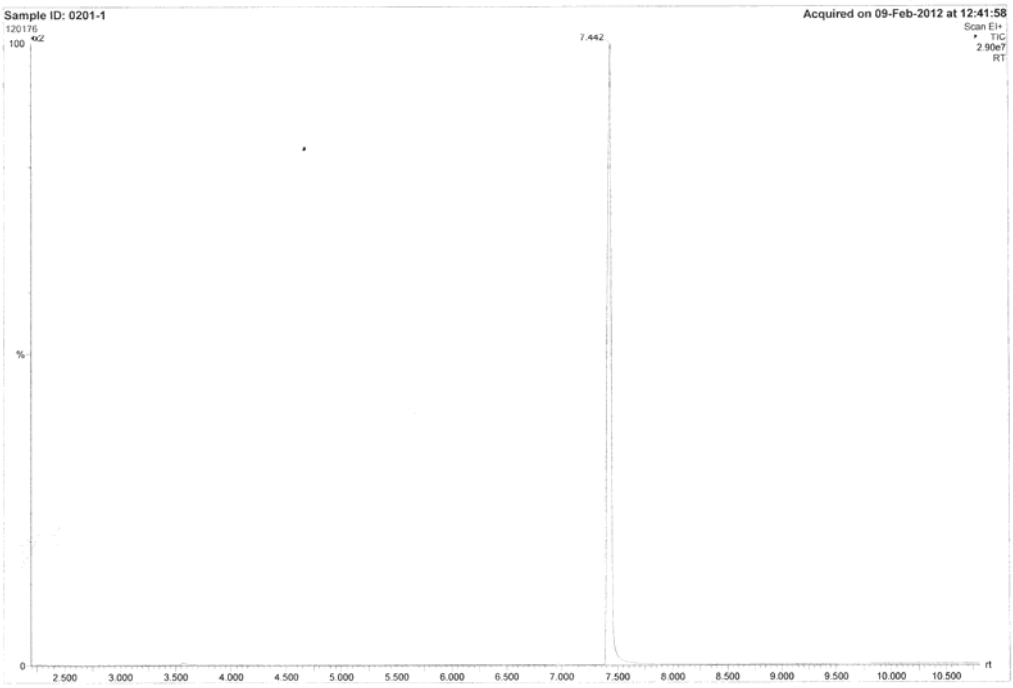
0.30
0.20
0.10
0.00
200
150
100
50
0
ppm (t1)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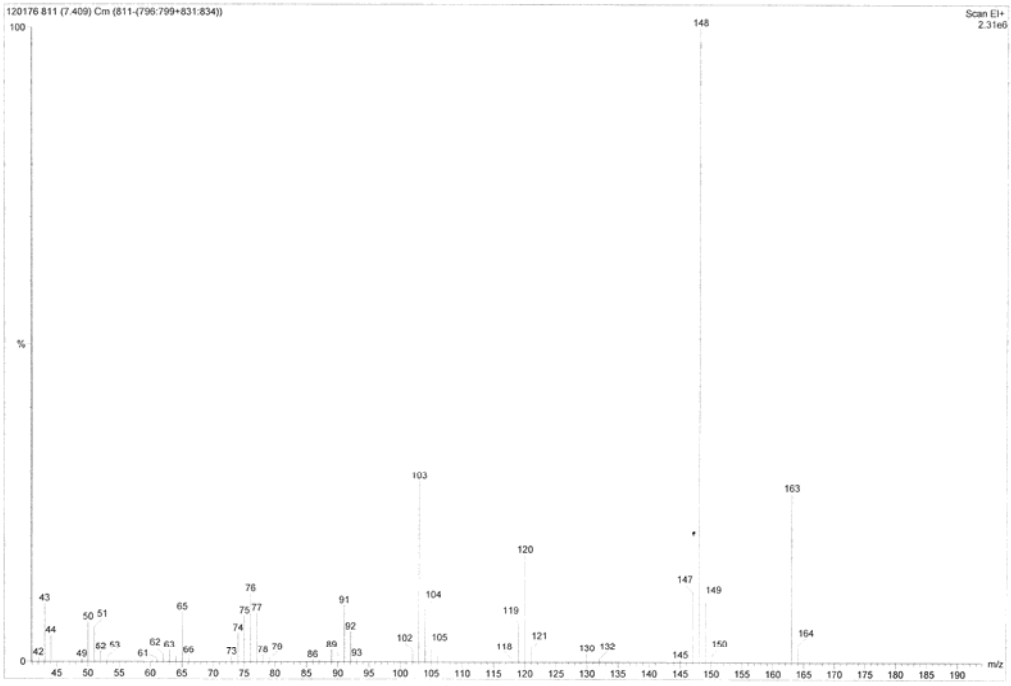
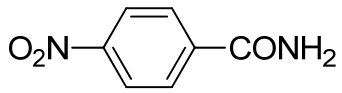
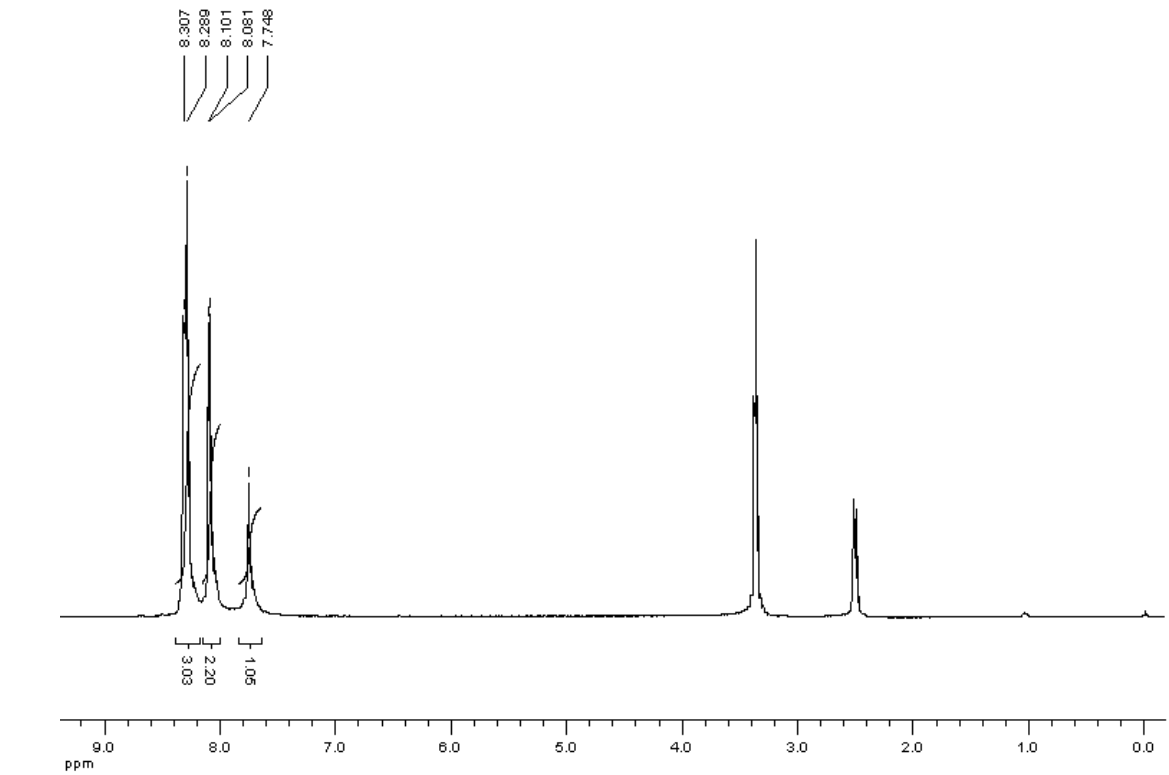
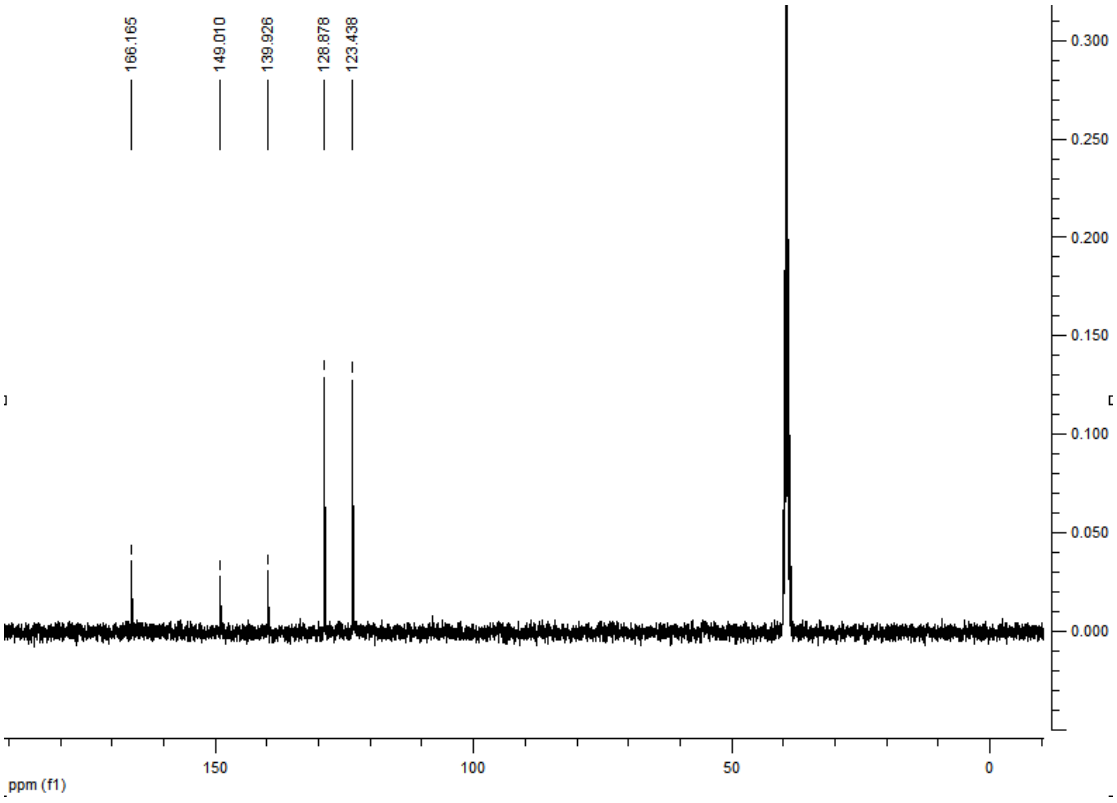
%
rt

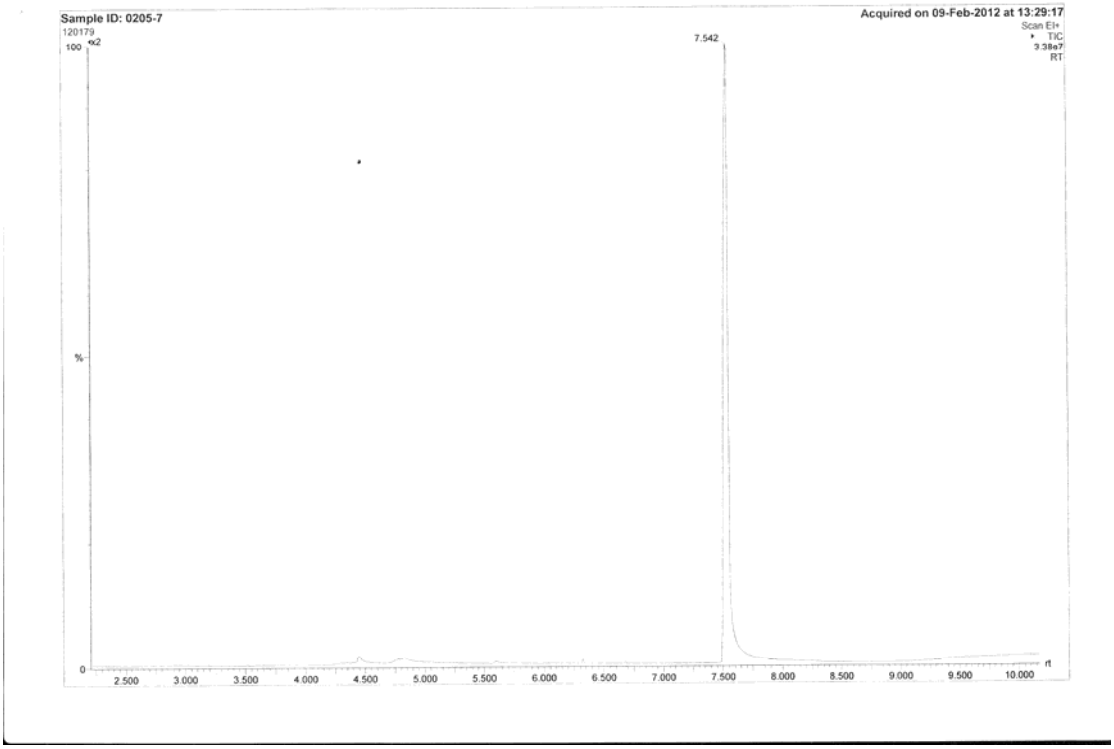

120176 811 (7.409) Cm (811-(796:799+831:834))
Scan EI+
2.31e6
148
103
163
120
%
m/z

2h:





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100
%
0
rt
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3.500
4.000
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5.500
6.000
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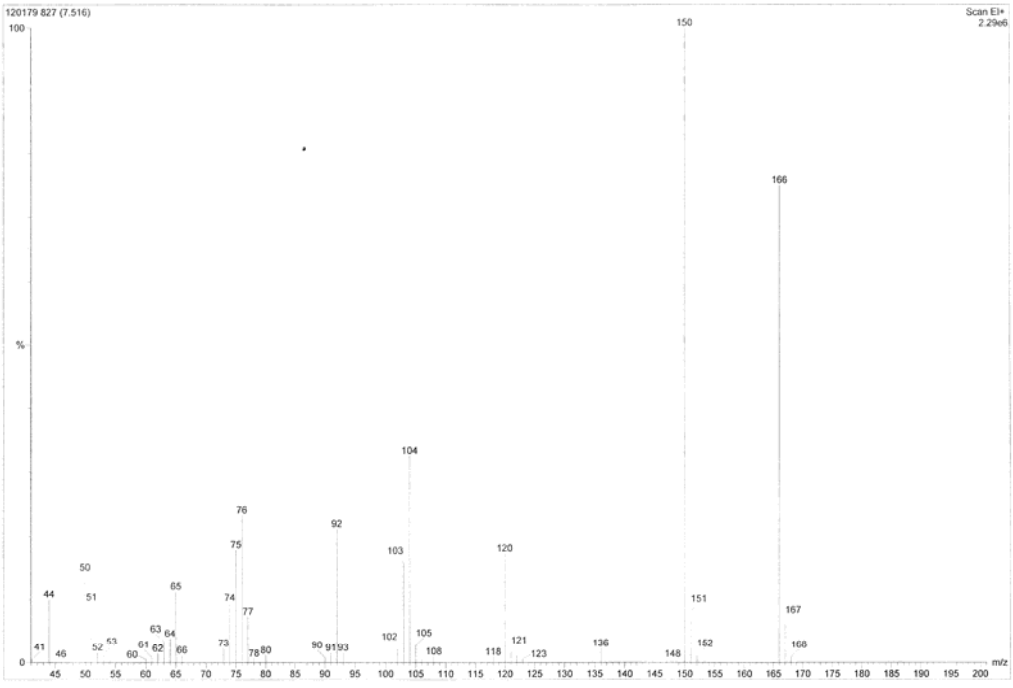
7.000
7.500
8.000
8.500
9.000
9.500
10.000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%
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m/z
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104
76
92
75
103
120
50
65
44
51
74
77
151
167
63
64
41
46
52
53
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66
73
78
80
90
91
93
102
105
108
118
121
123
136
148
152
168
45
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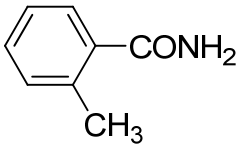

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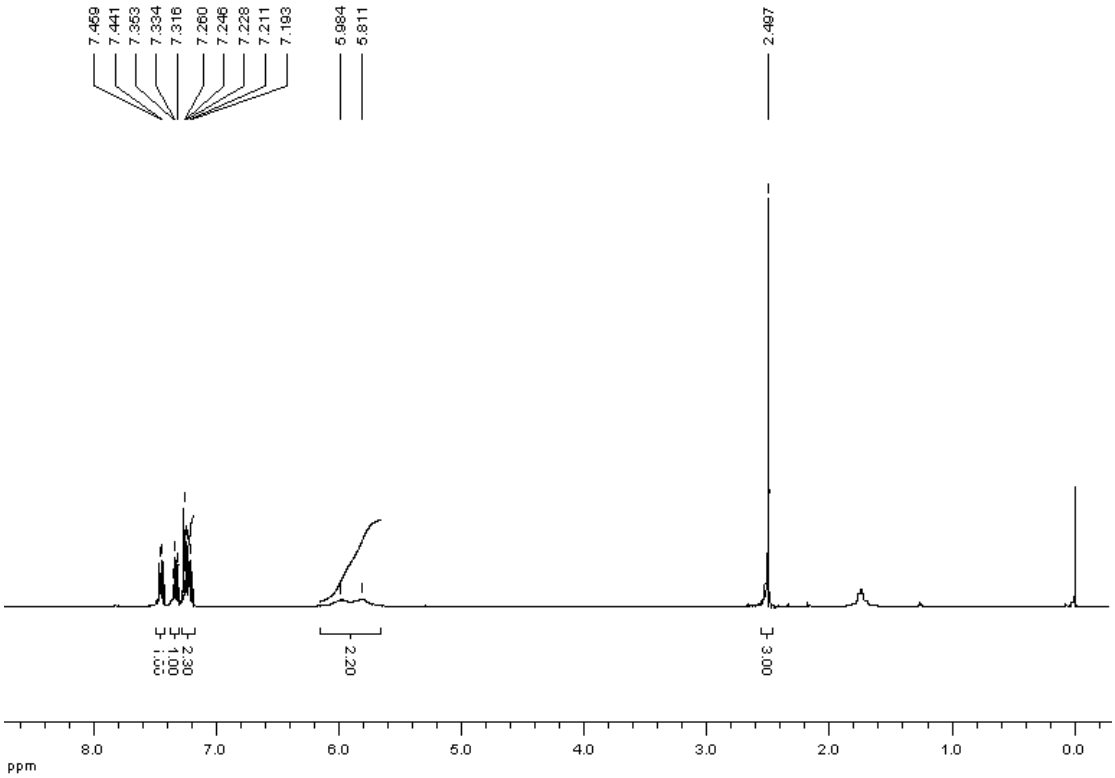
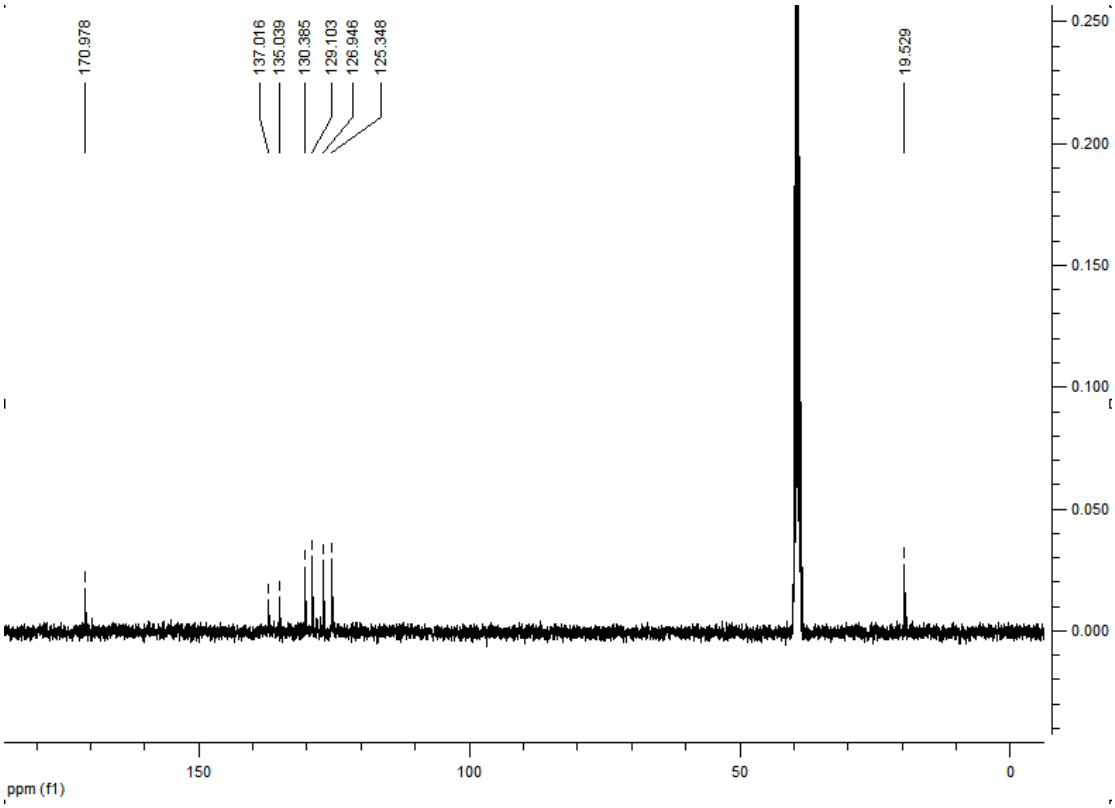
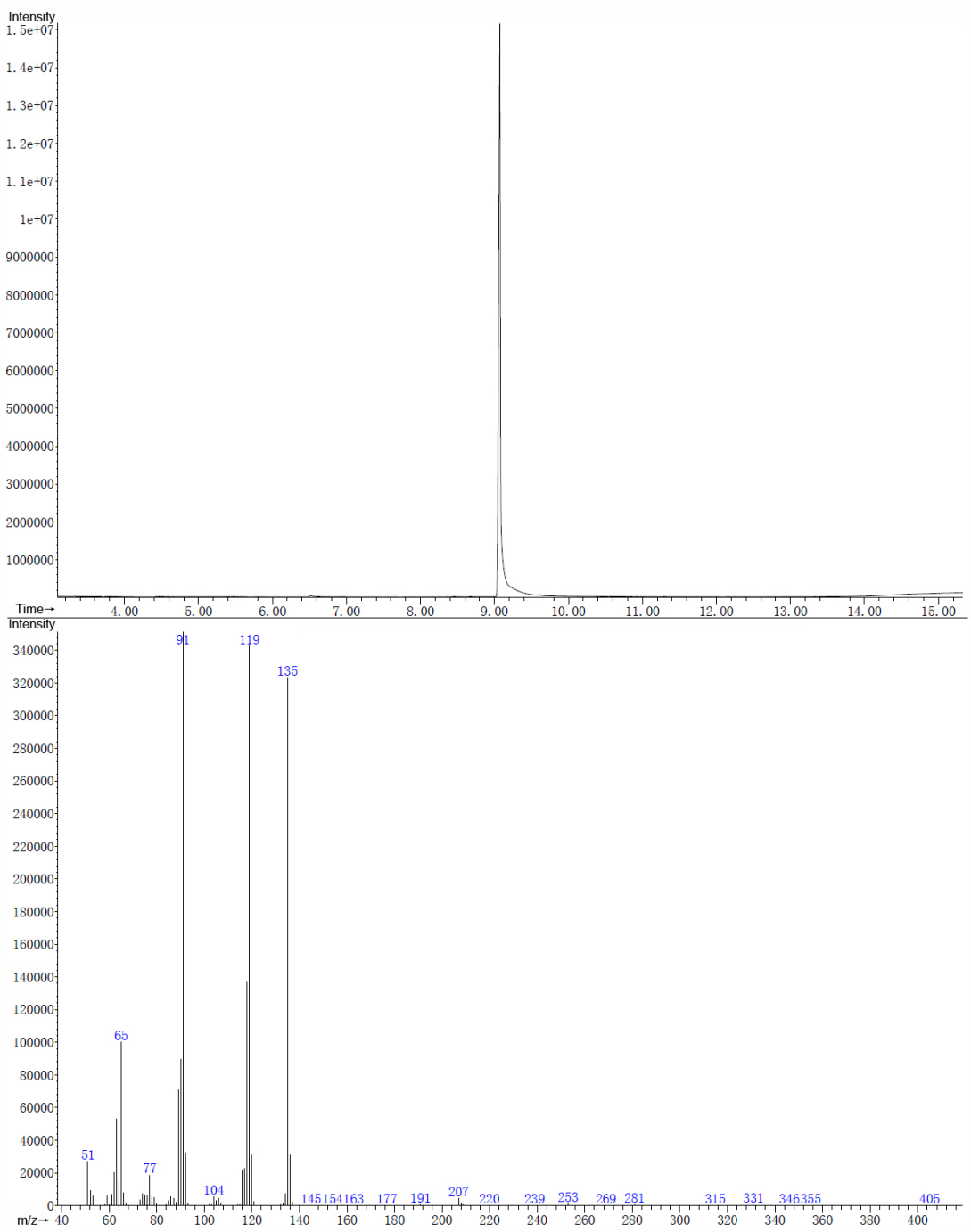
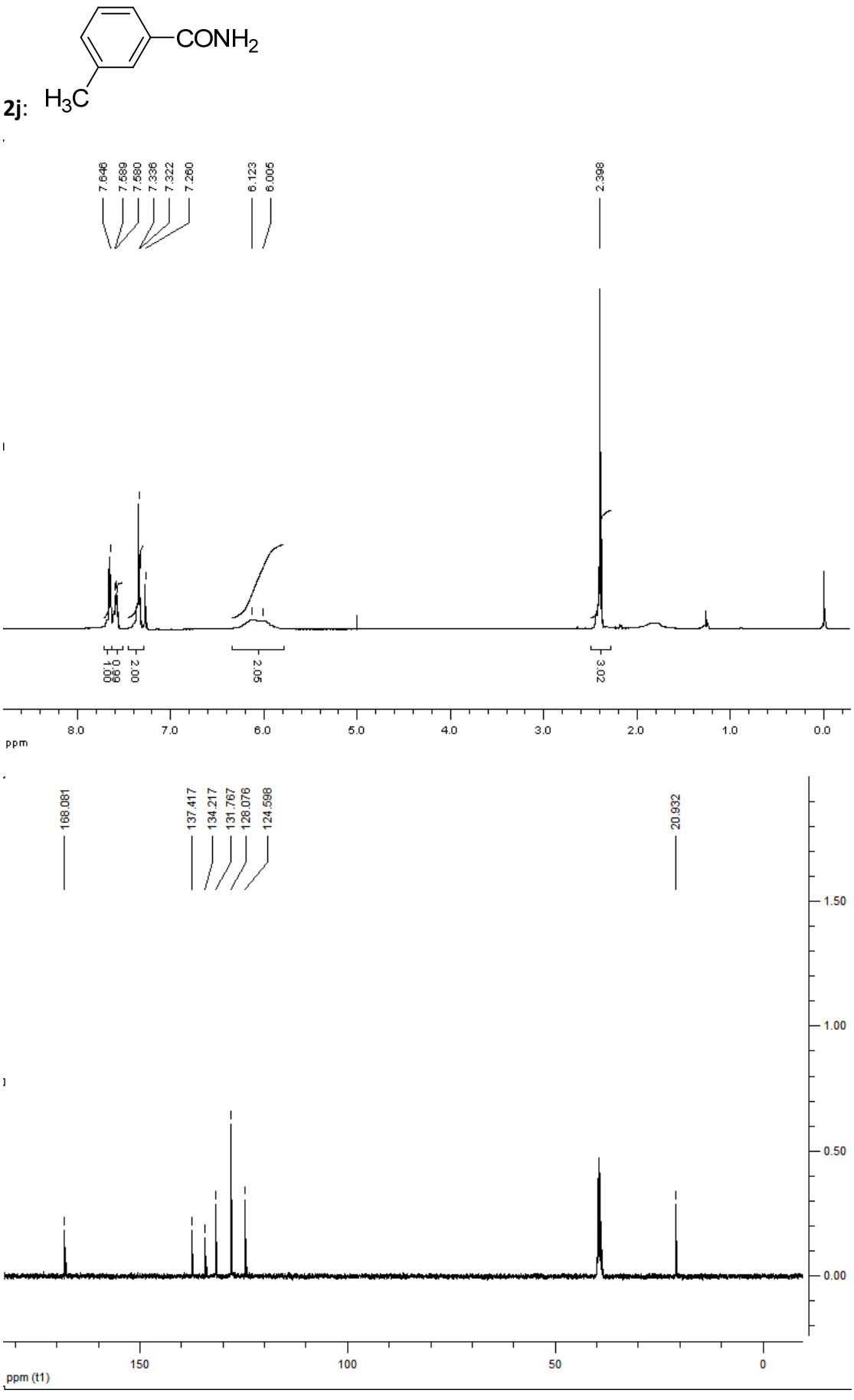
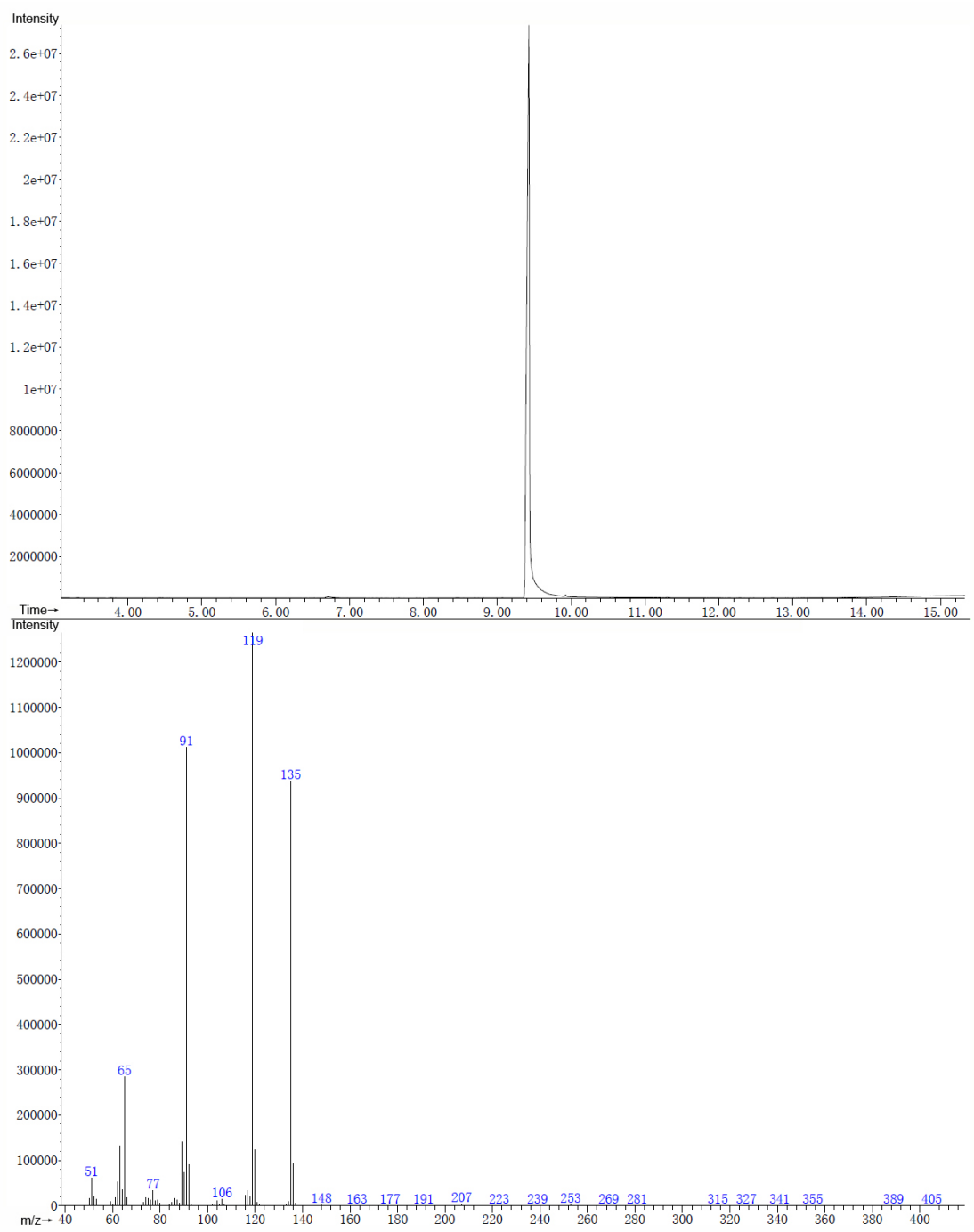






2j:

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Time→
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
Intensity
1200000
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
m/z→
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
119
91
135
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315
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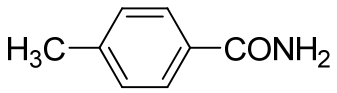
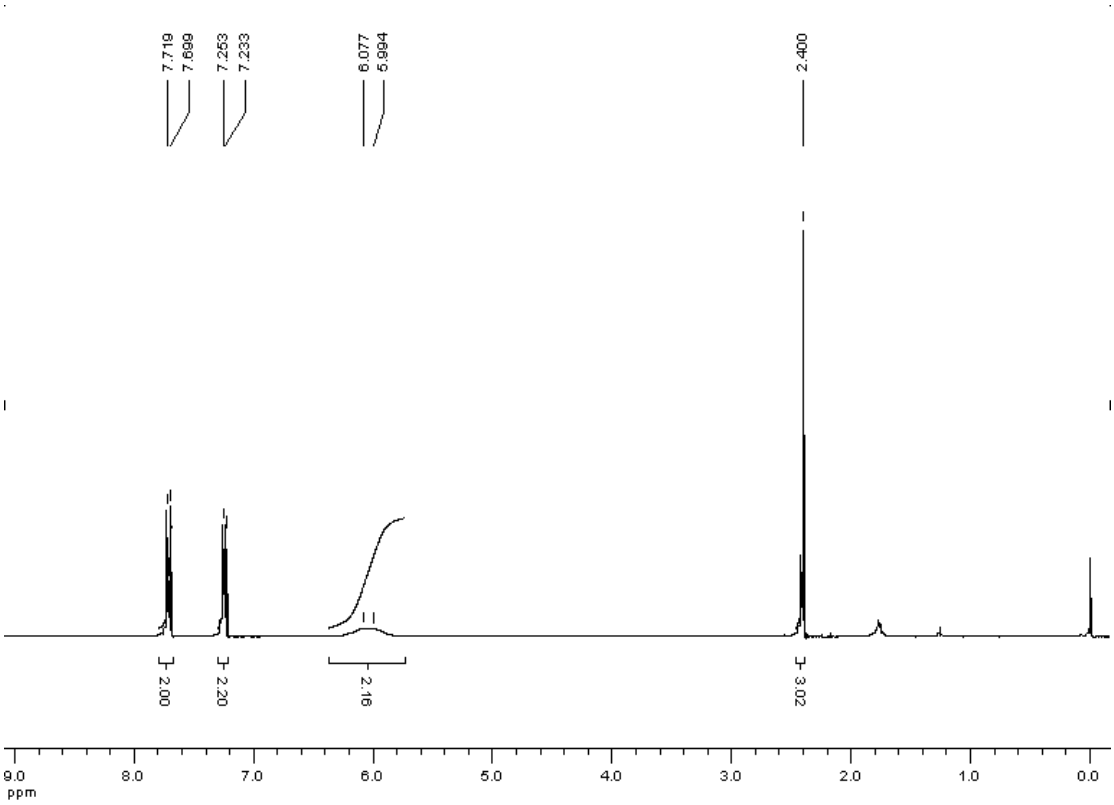
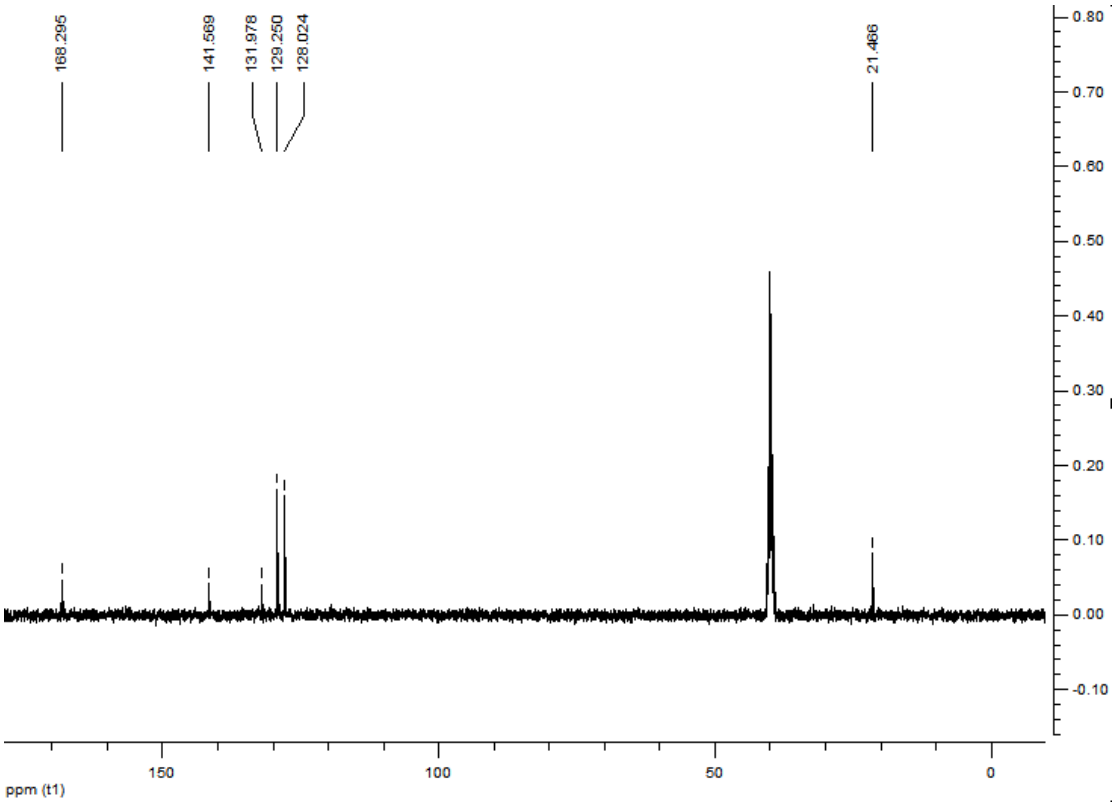
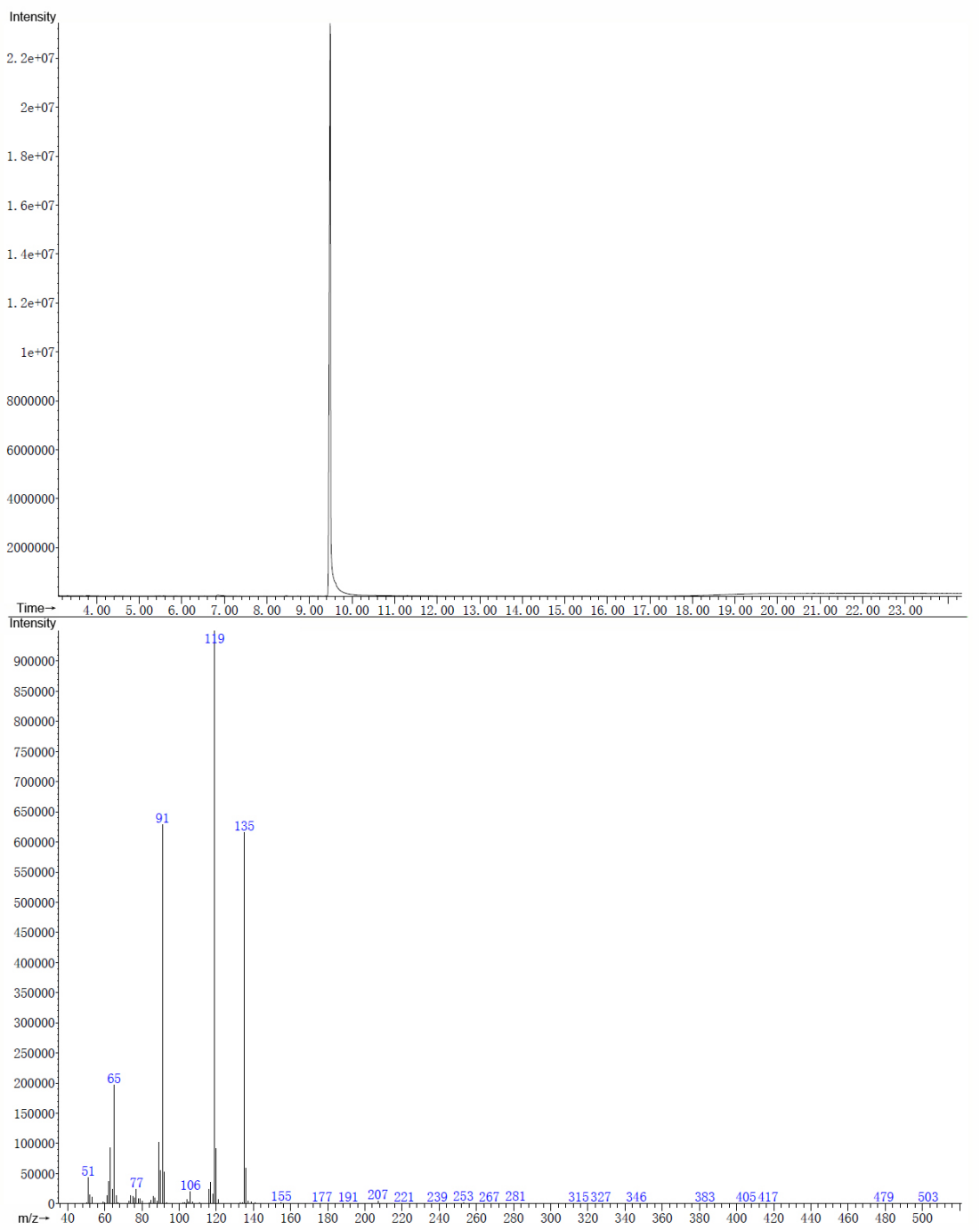
389
405

2k:

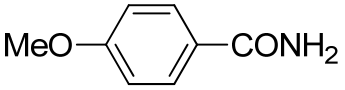

H3C
CONH2

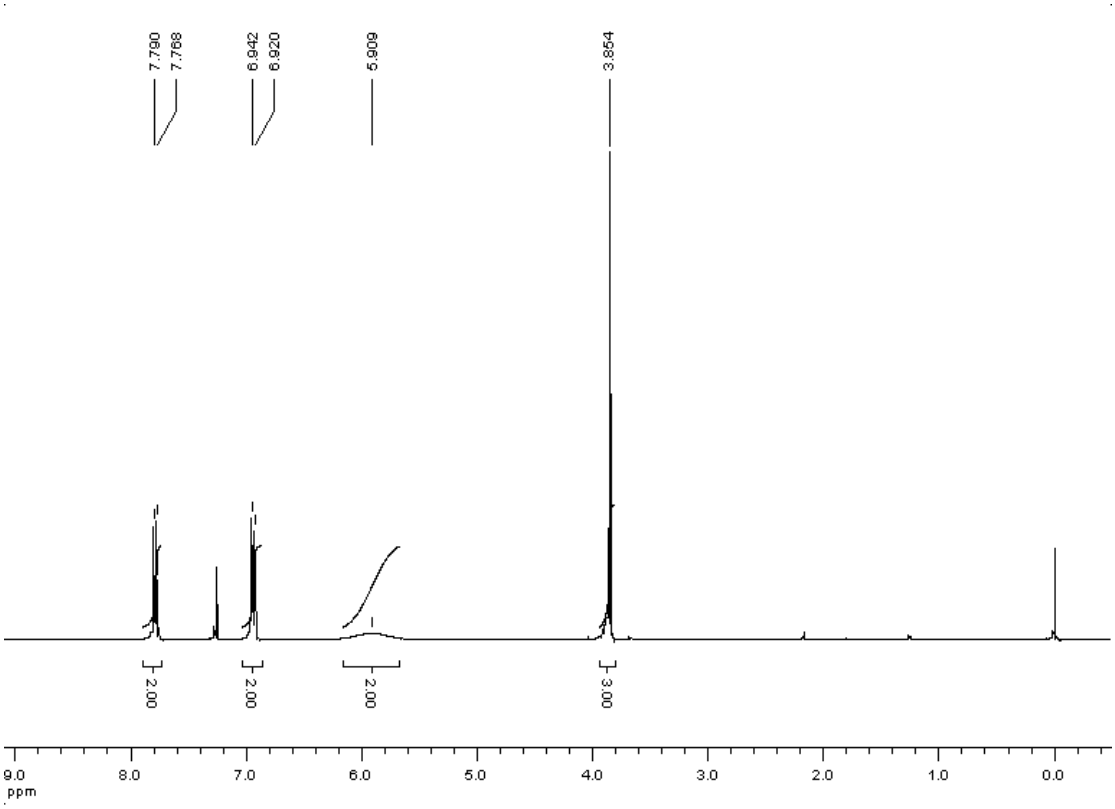

7.719
7.699
7.253
7.233
6.077
5.994
2.400
2.00
2.20
2.16
3.02
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm

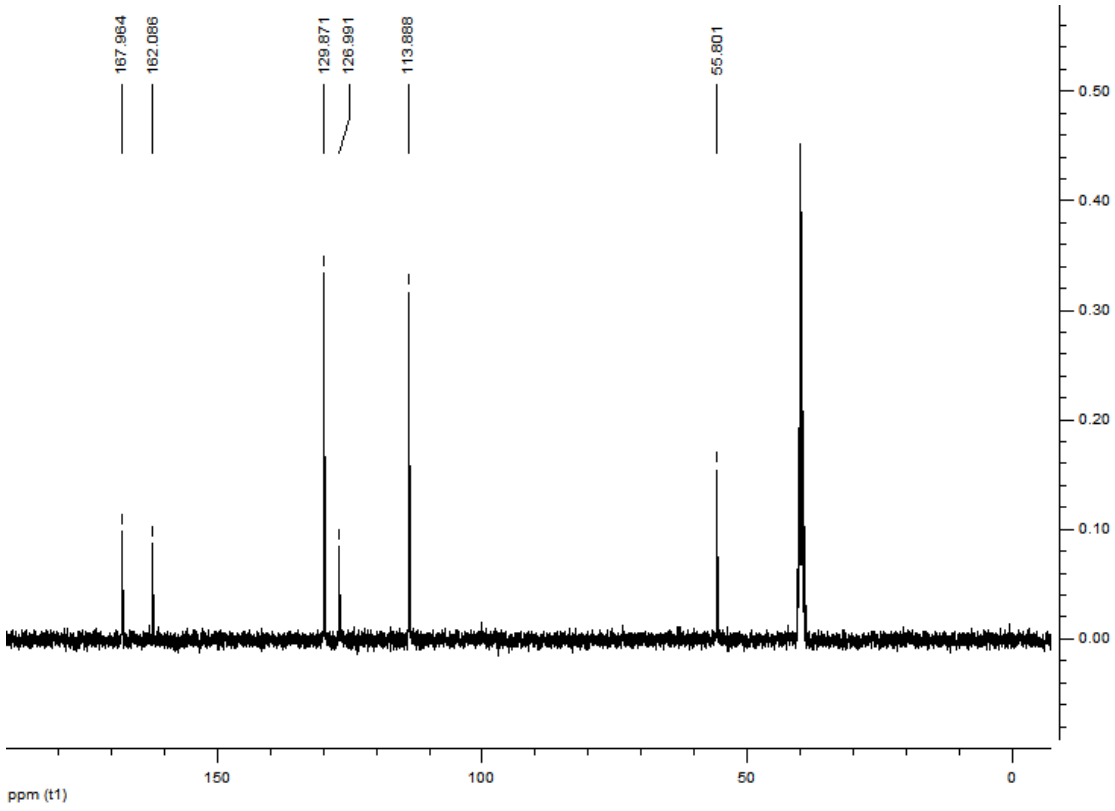

168.295
141.569
131.978
129.250
128.024
21.466
0.80
0.70
0.60
0.50
0.40
0.30
0.20
0.10
0.00
-0.10
150
100
50
0
ppm (t1)

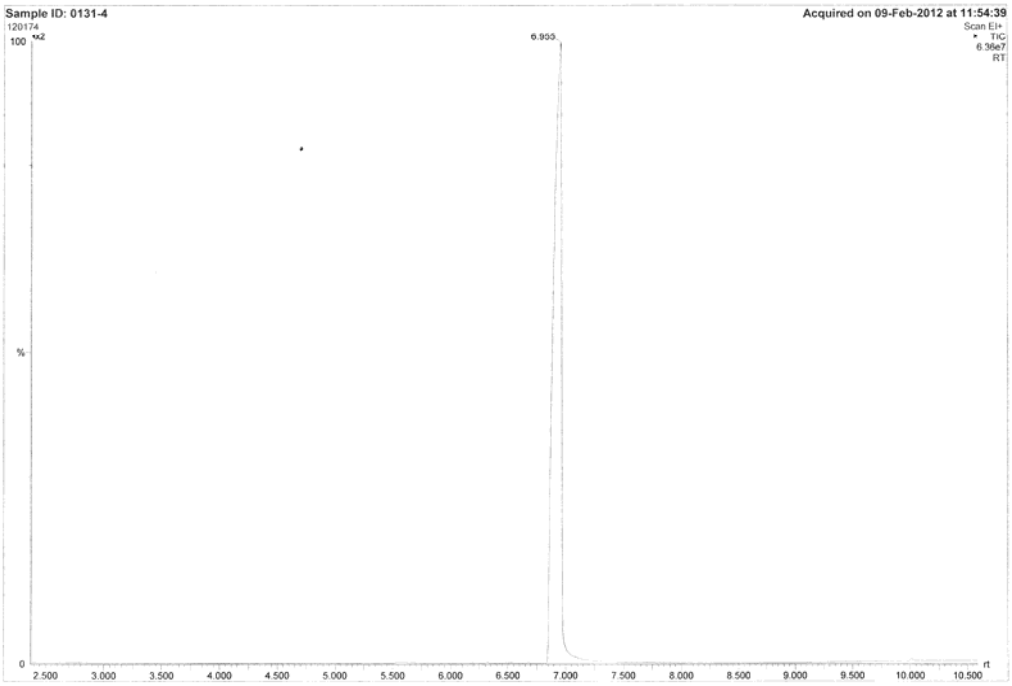

Intensity
2.2e+07
2e+07
1.8e+07
1.6e+07
1.4e+07
1.2e+07
1e+07
8000000
6000000
4000000
2000000
Time→
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00 21.00 22.00 23.00
Intensity
900000
850000
800000
750000
700000
650000
600000
550000
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
m/z→
119
91
135
65
51
77
106
155
177
191
207
221
239
253
267
281
315
327
346
383
405
417
479
503
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500

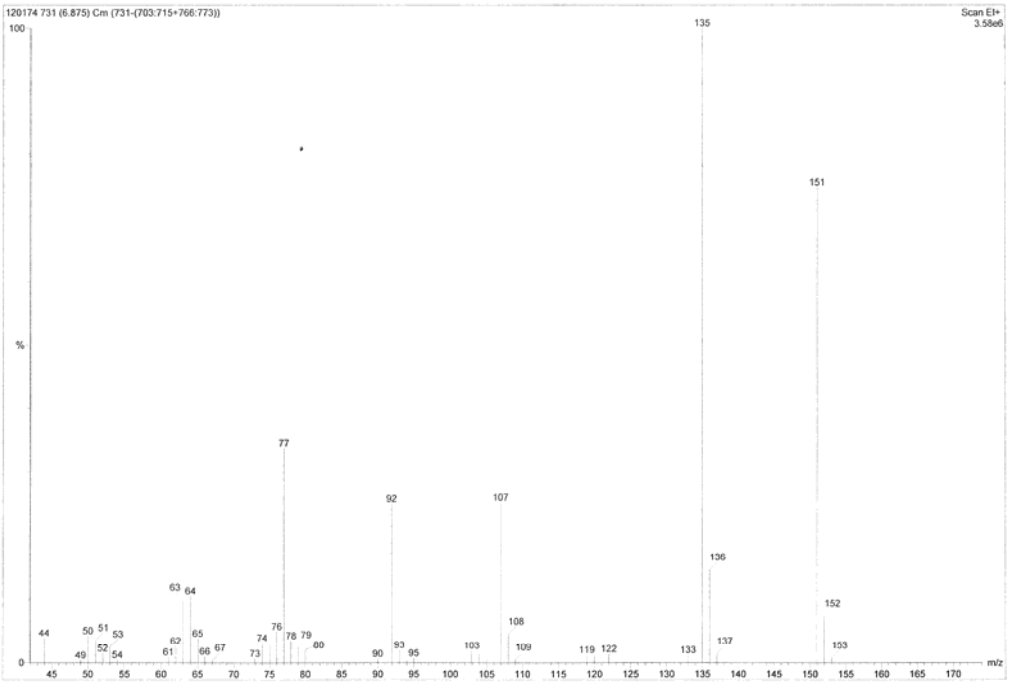
2l:

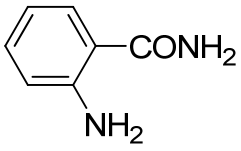




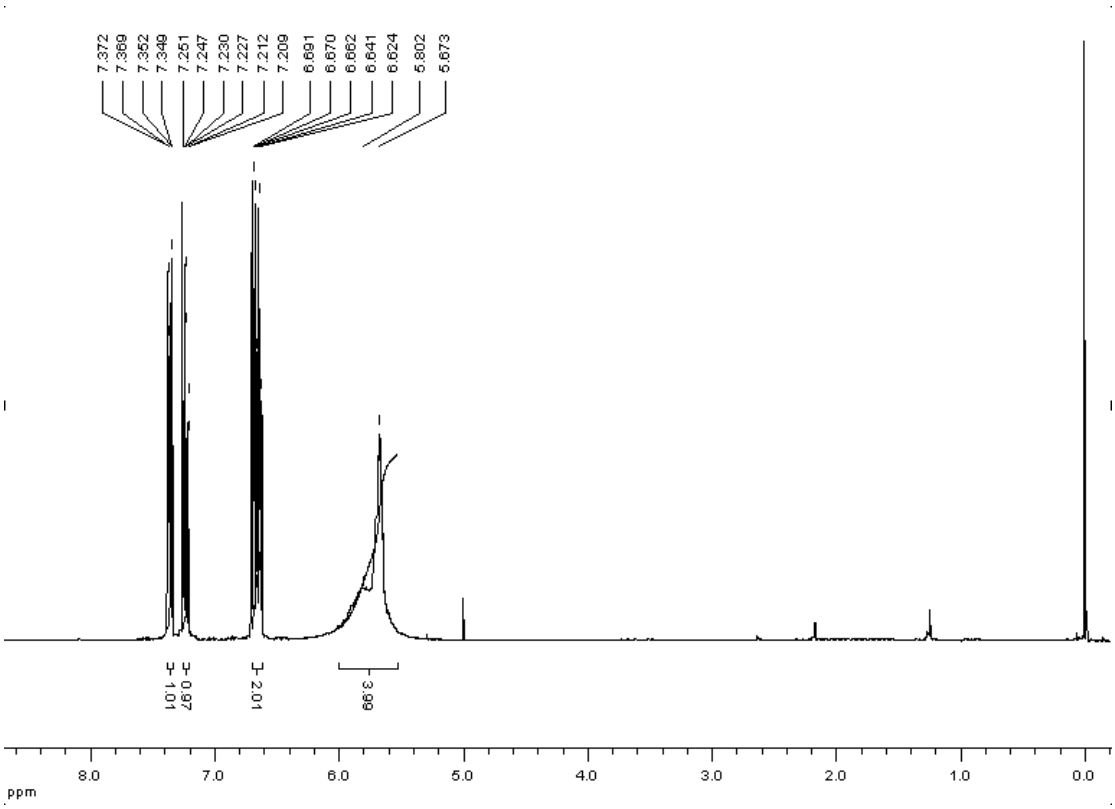


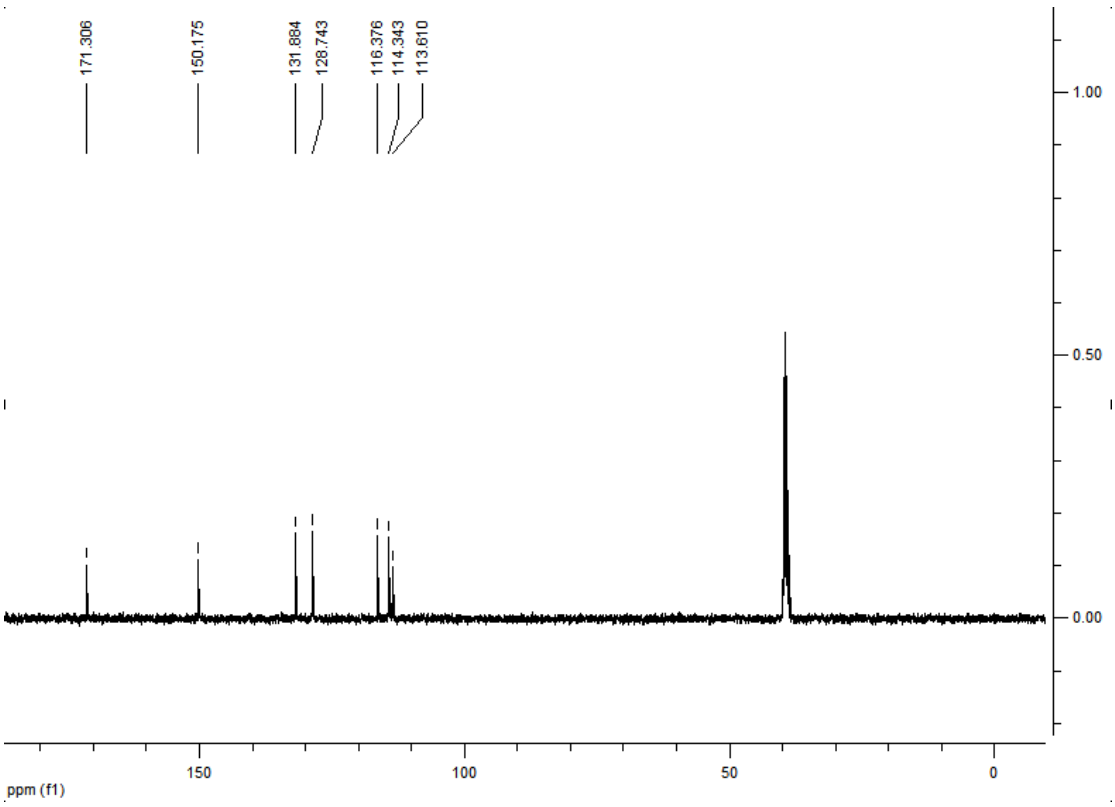

Sample ID: 0131-4
Acquired on 09-Feb-2012 at 11:54:39
120174
Scan EI+
TIC
6.36e7
RT
6.993
100
%
0
rt
2.500 3.000 3.500 4.000 4.500 5.000 5.500 6.000 6.500 7.000 7.500 8.000 8.500 9.000 9.500 10.000 10.500

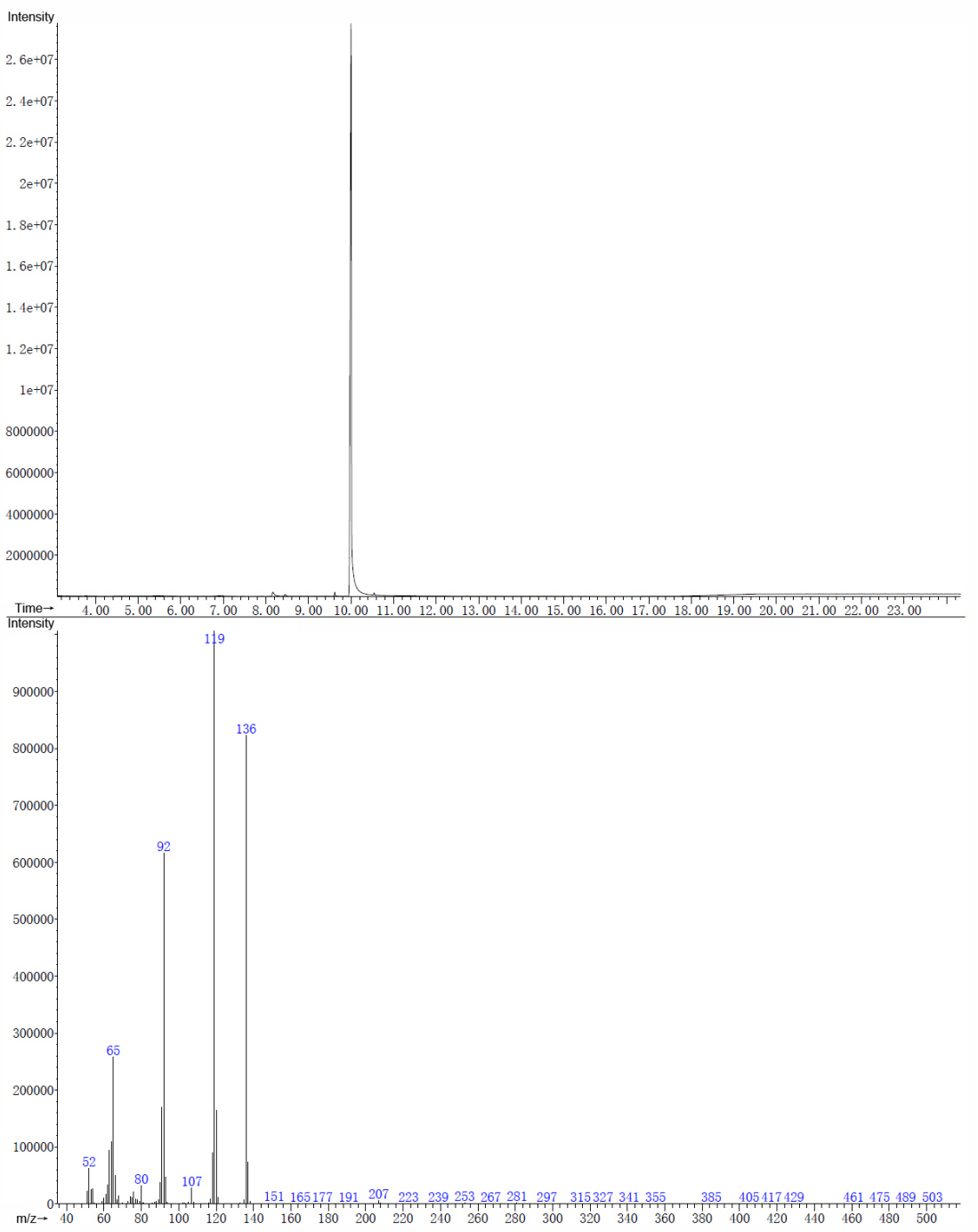

120174 731 (6.875) Cm (731-(703:715+766:773))
Scan EI+
3.58e6
100
%
0
135
151
77
92
107
136
152
63
64
108
44
50
51
53
52
54
49
62
61
65
66
67
74
73
76
78
79
80
90
93
95
103
109
119
122
133
137
153
m/z
45 50 55 60 65 70 75 80 85 90 95 100 105 110 115 120 125 130 135 140 145 150 155 160 165 170

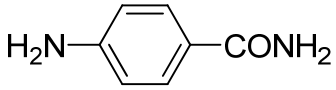


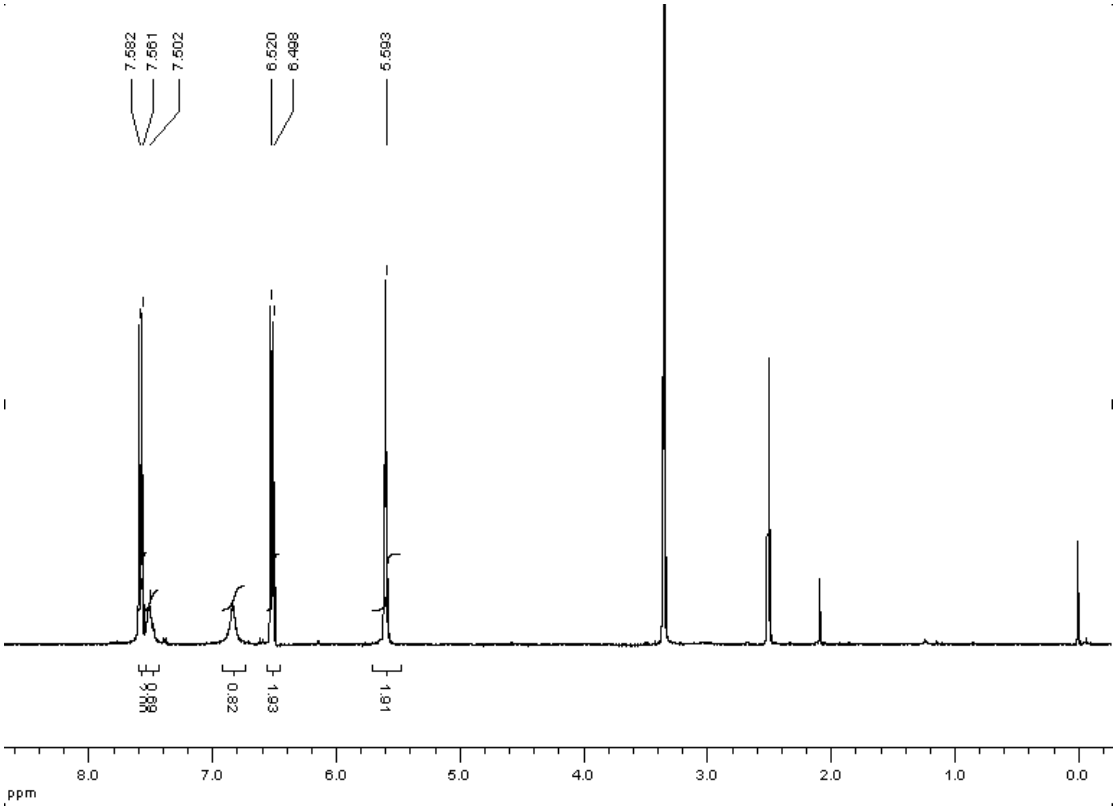
2m:

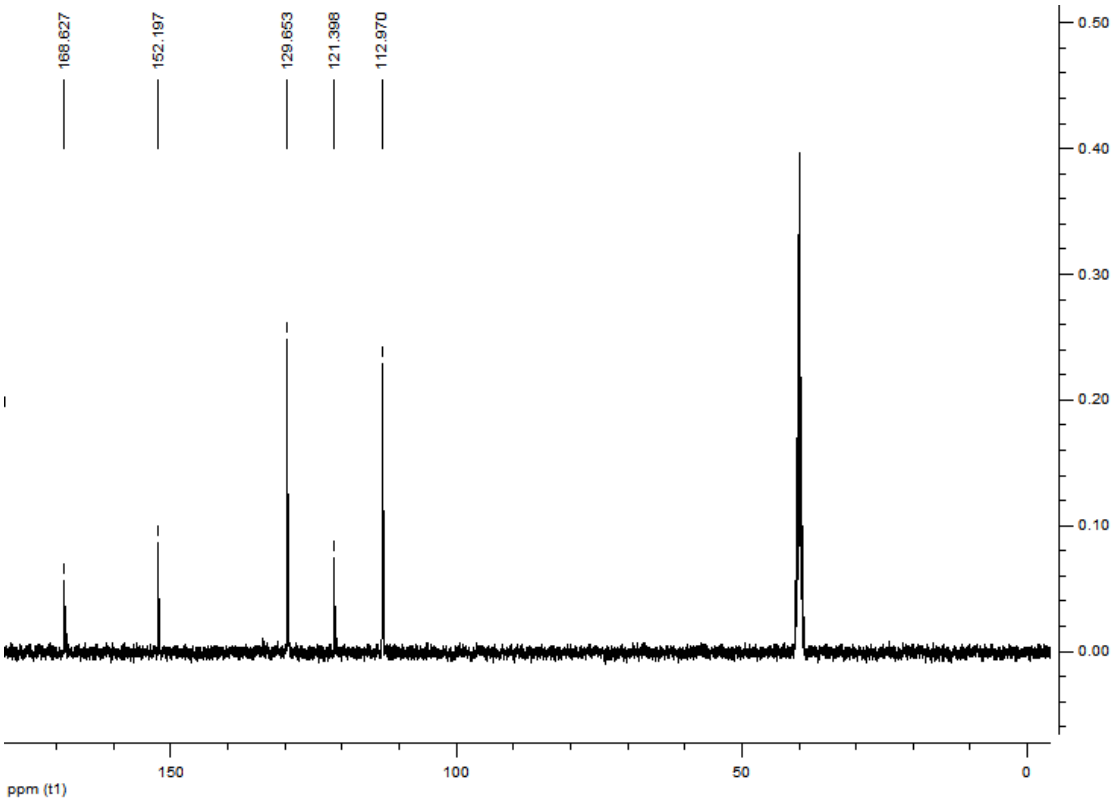


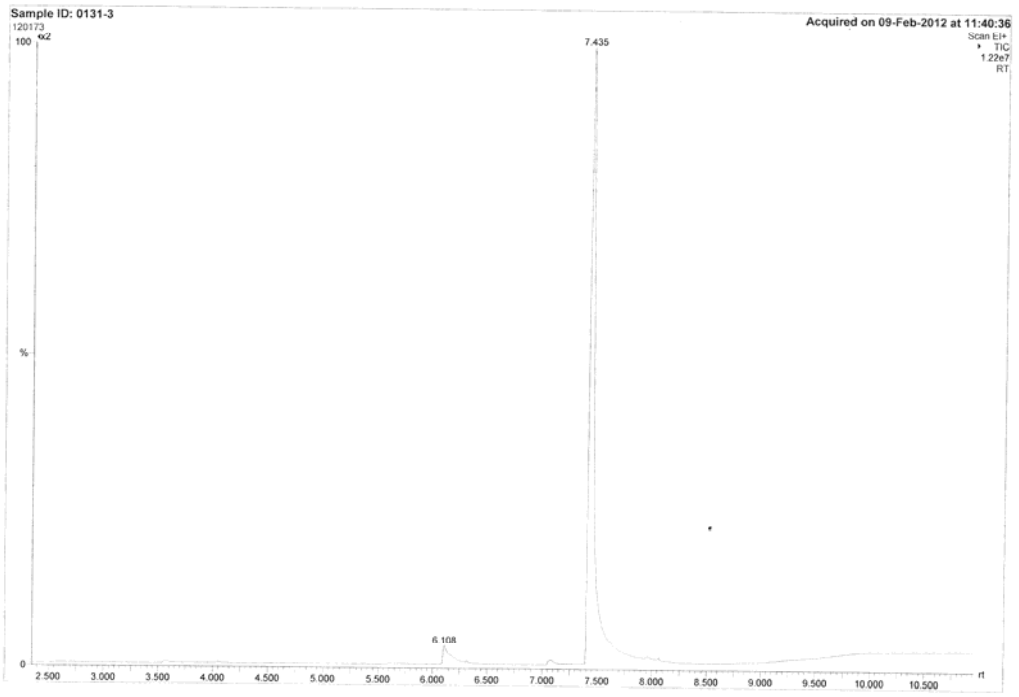


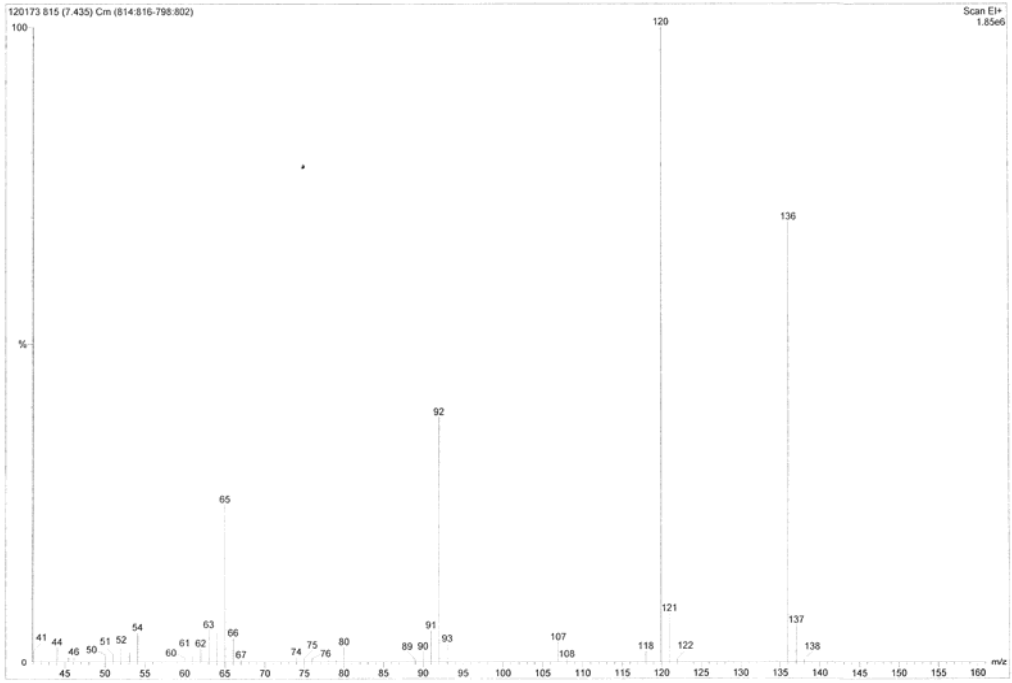

Intensity
2.6e+07
2.4e+07
2.2e+07
2e+07
1.8e+07
1.6e+07
1.4e+07
1.2e+07
1e+07
8000000
6000000
4000000
2000000
Time→
4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00 21.00 22.00 23.00
Intensity
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
119
136
92
65
52
80
107
151 165 177 191 207 223 239 253 267 281 297 315 327 341 355 385 405 417 429 461 475 489 503
m/z→
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500

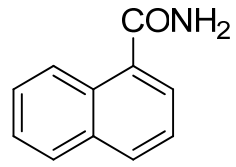
2n: 
H2N
CONH2


7.582
7.561
7.502
6.520
6.498
5.593
2.00
0.82
1.93
1.91
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm

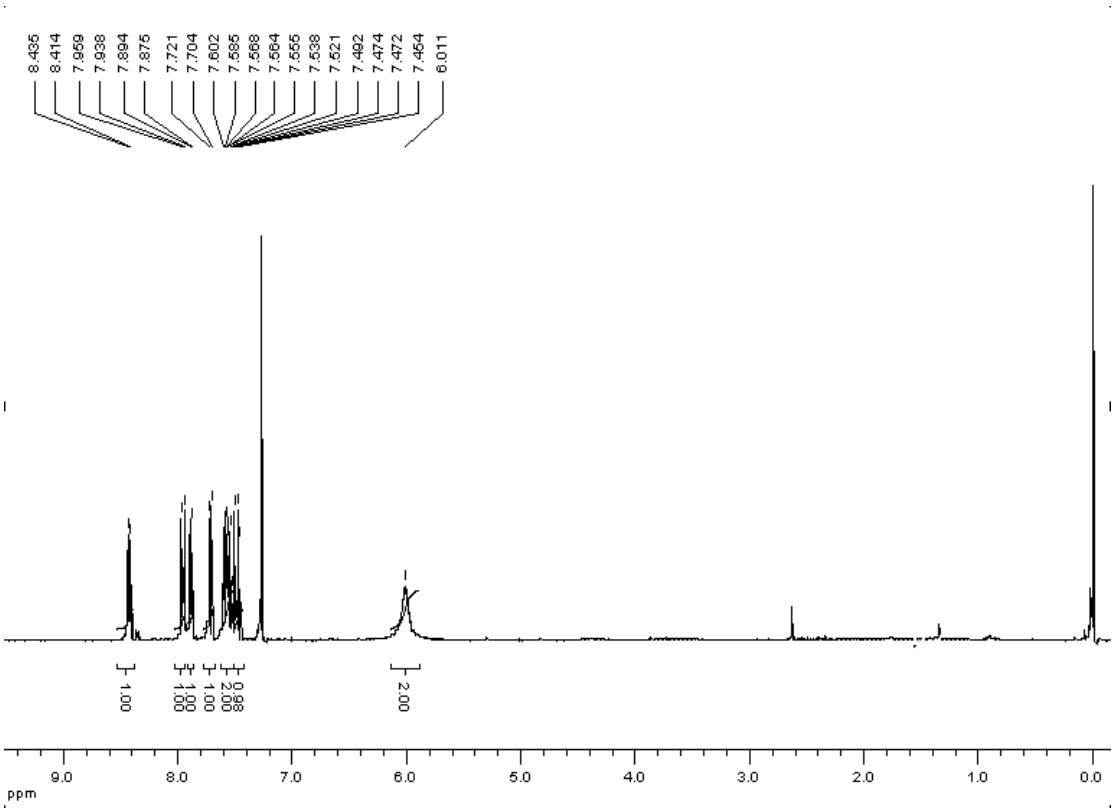

168.627
152.197
129.653
121.398
112.970
0.50
0.40
0.30
0.20
0.10
0.00
150
100
50
0
ppm (t1)

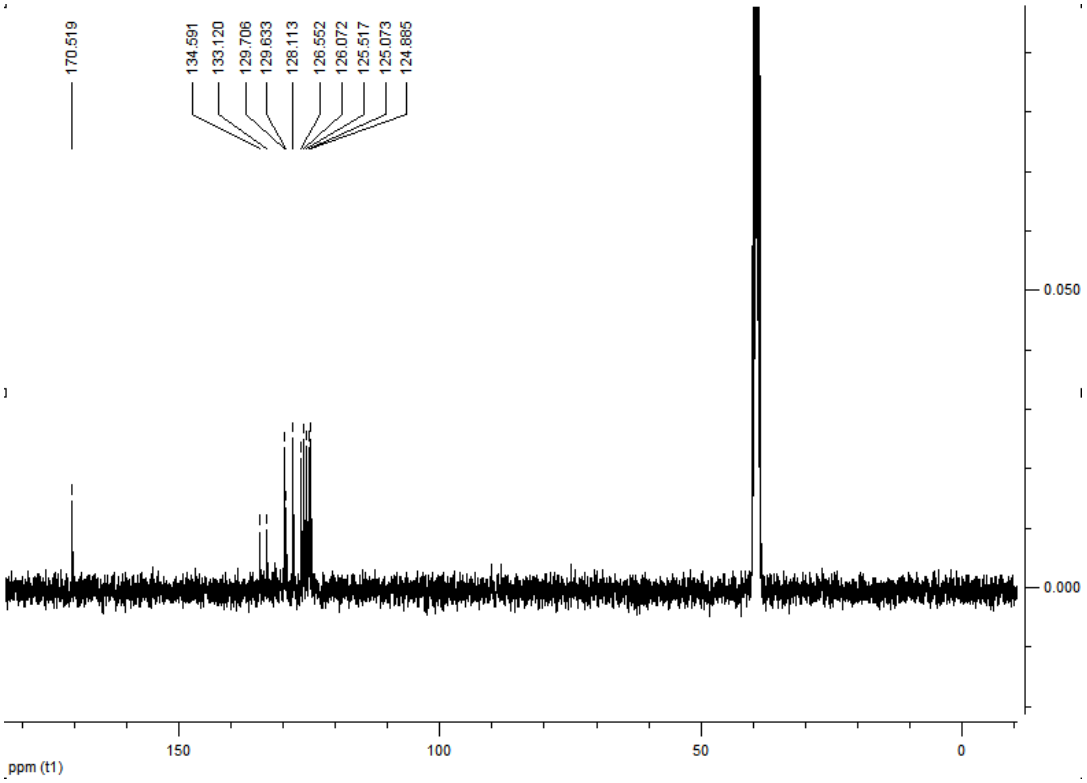


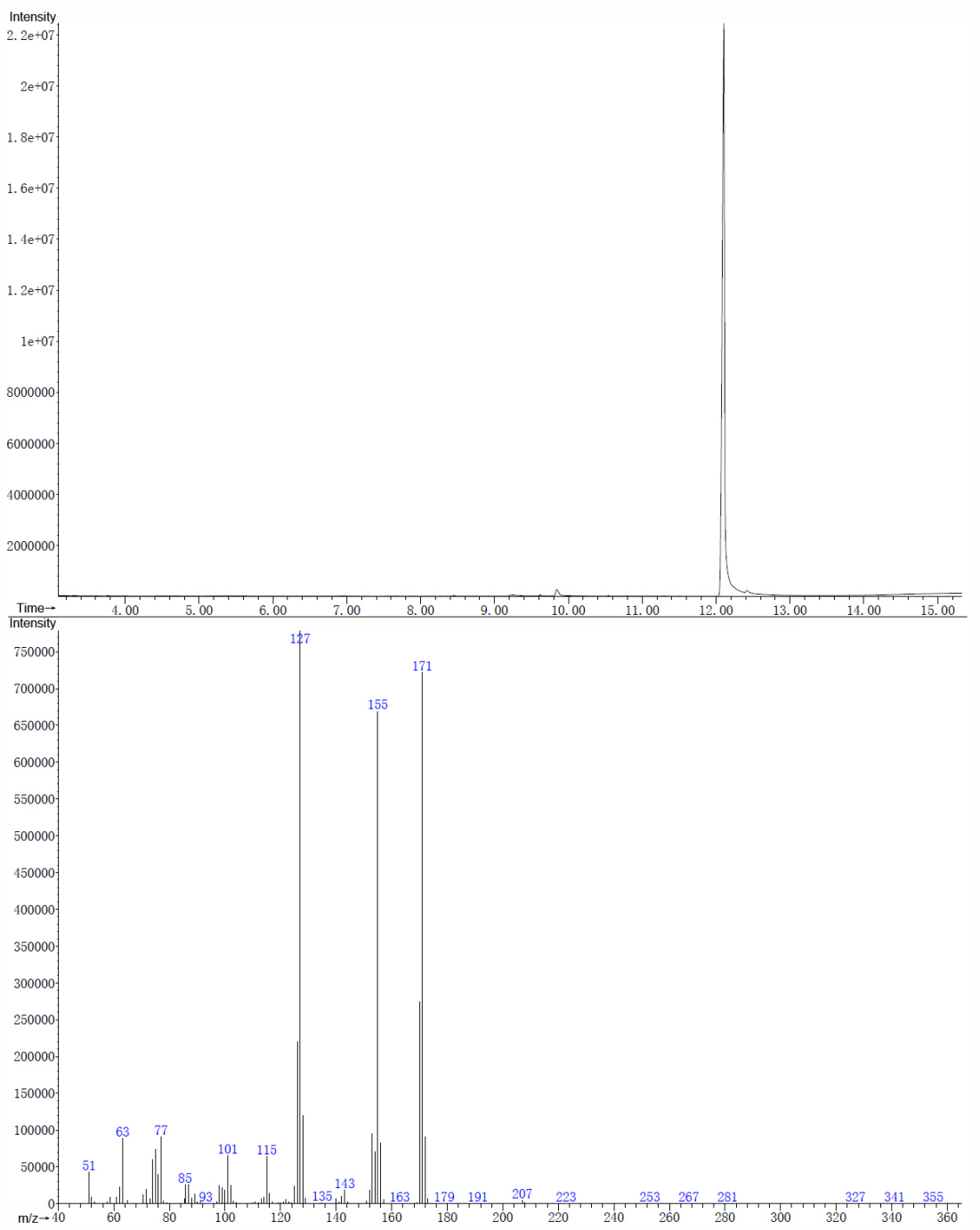




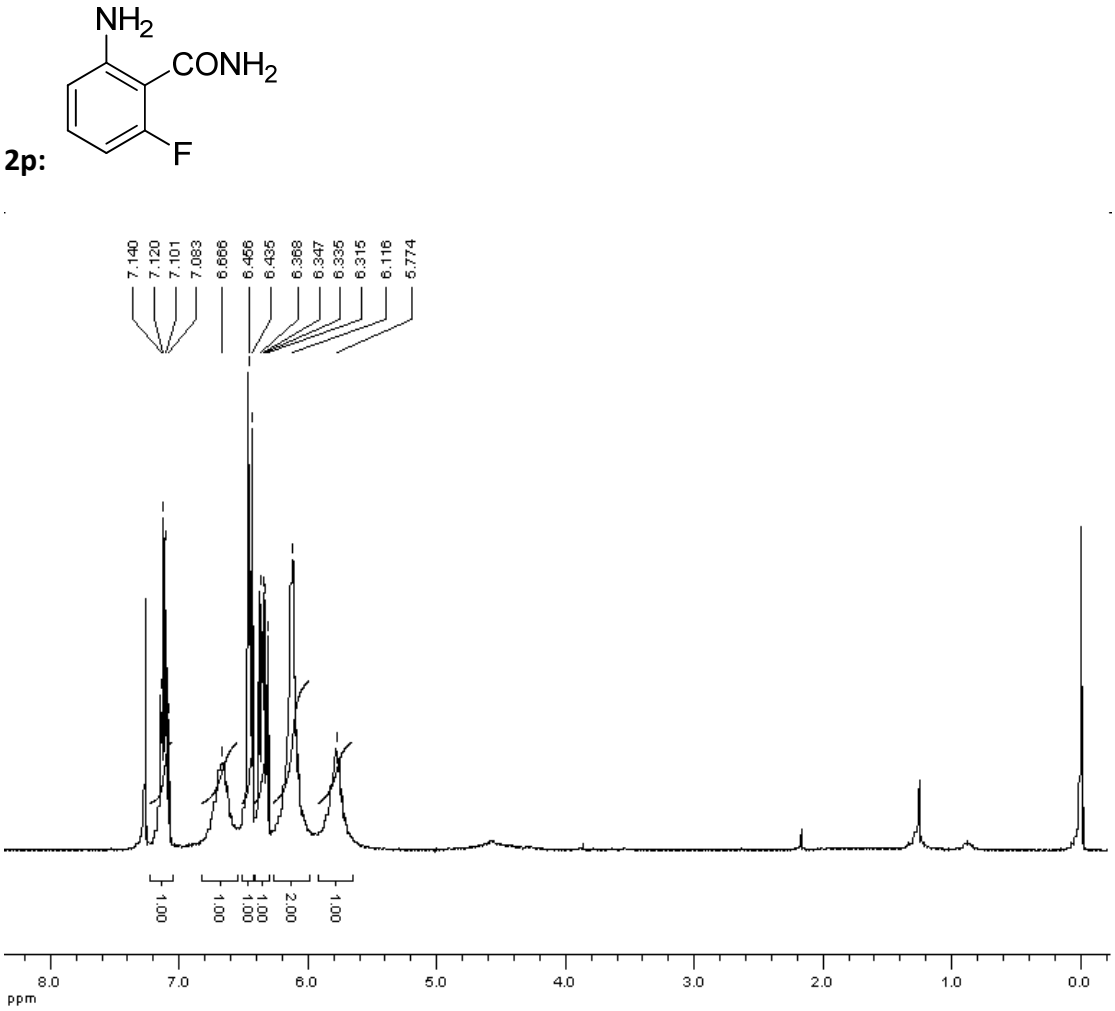
2o:

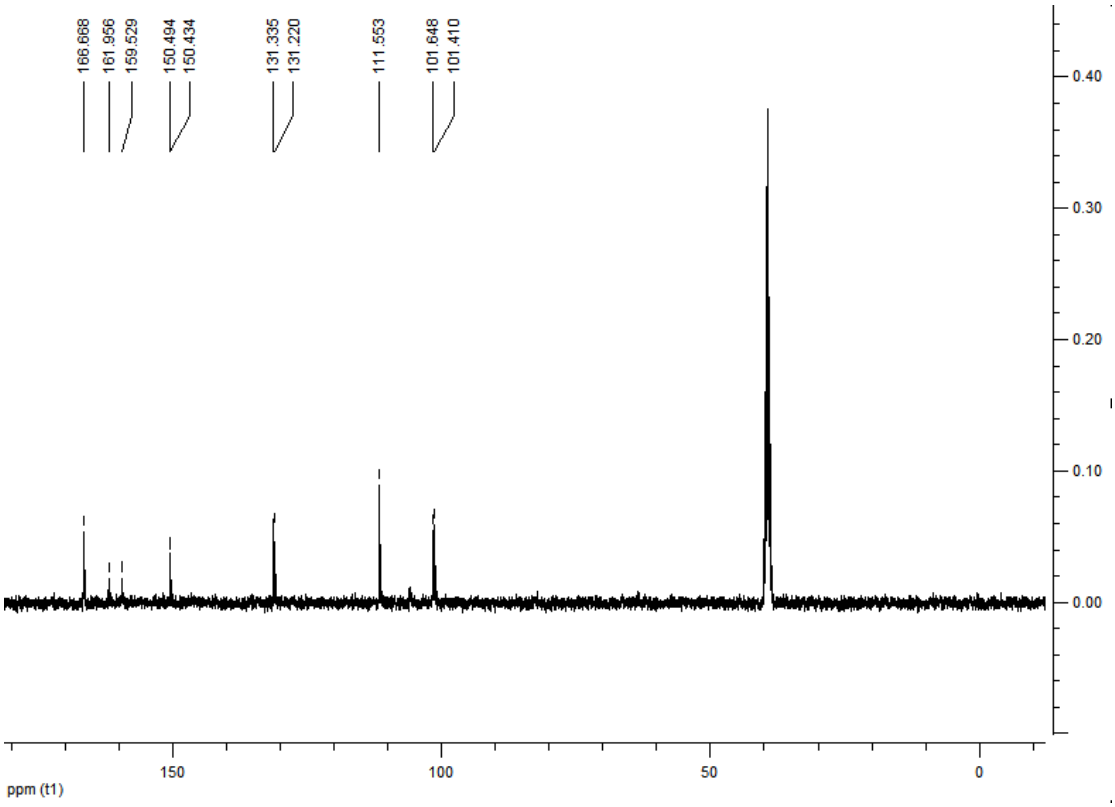


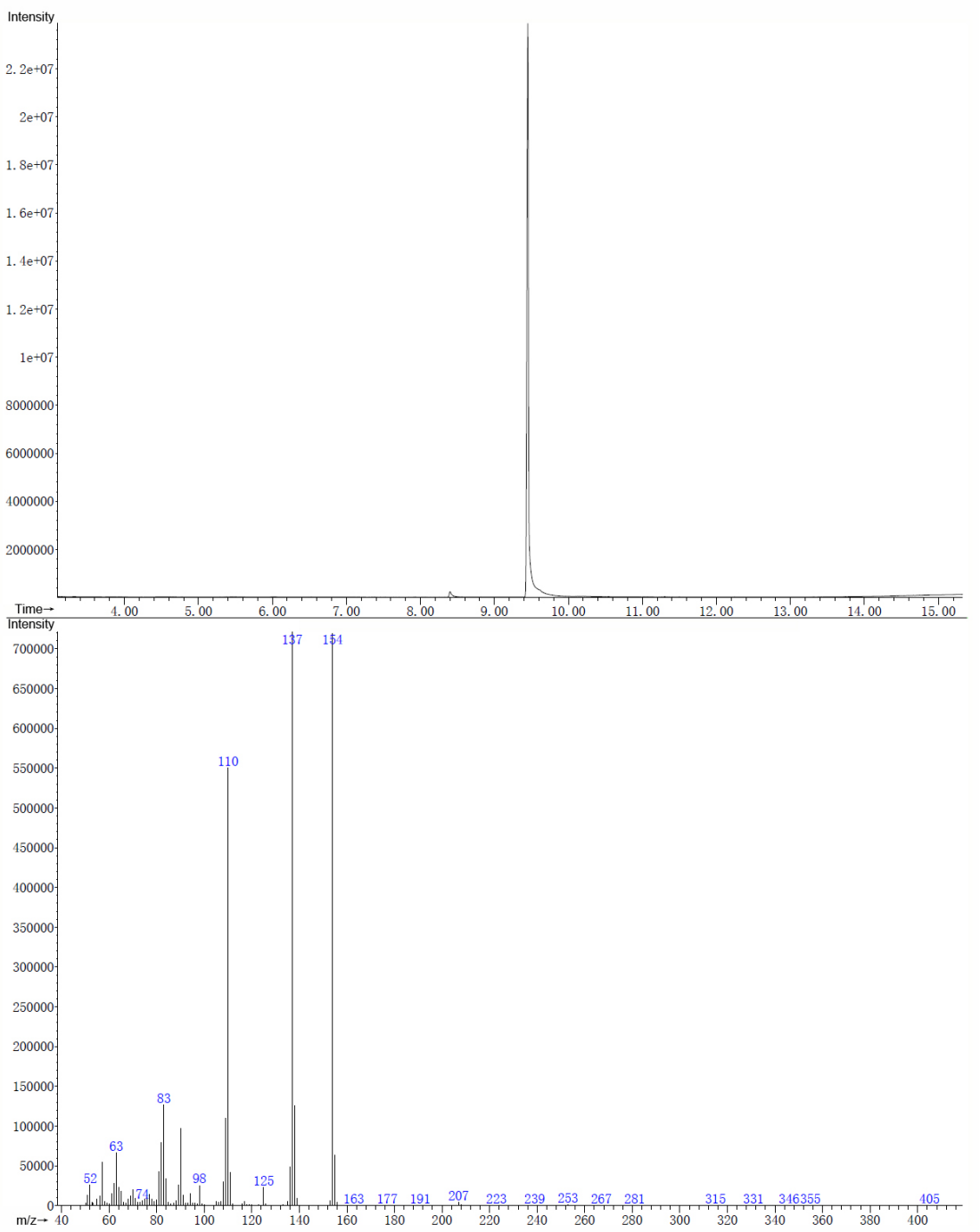



Intensity
2.2e+07
2e+07
1.8e+07
1.6e+07
1.4e+07
1.2e+07
1e+07
8000000
6000000
4000000
2000000
Time→
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
Intensity
750000
700000
650000
600000
550000
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
m/z→
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
127
171
155
63
77
101
115
51
85
93
143
135
163
179
191
207
223
253
267
281
327
341
355

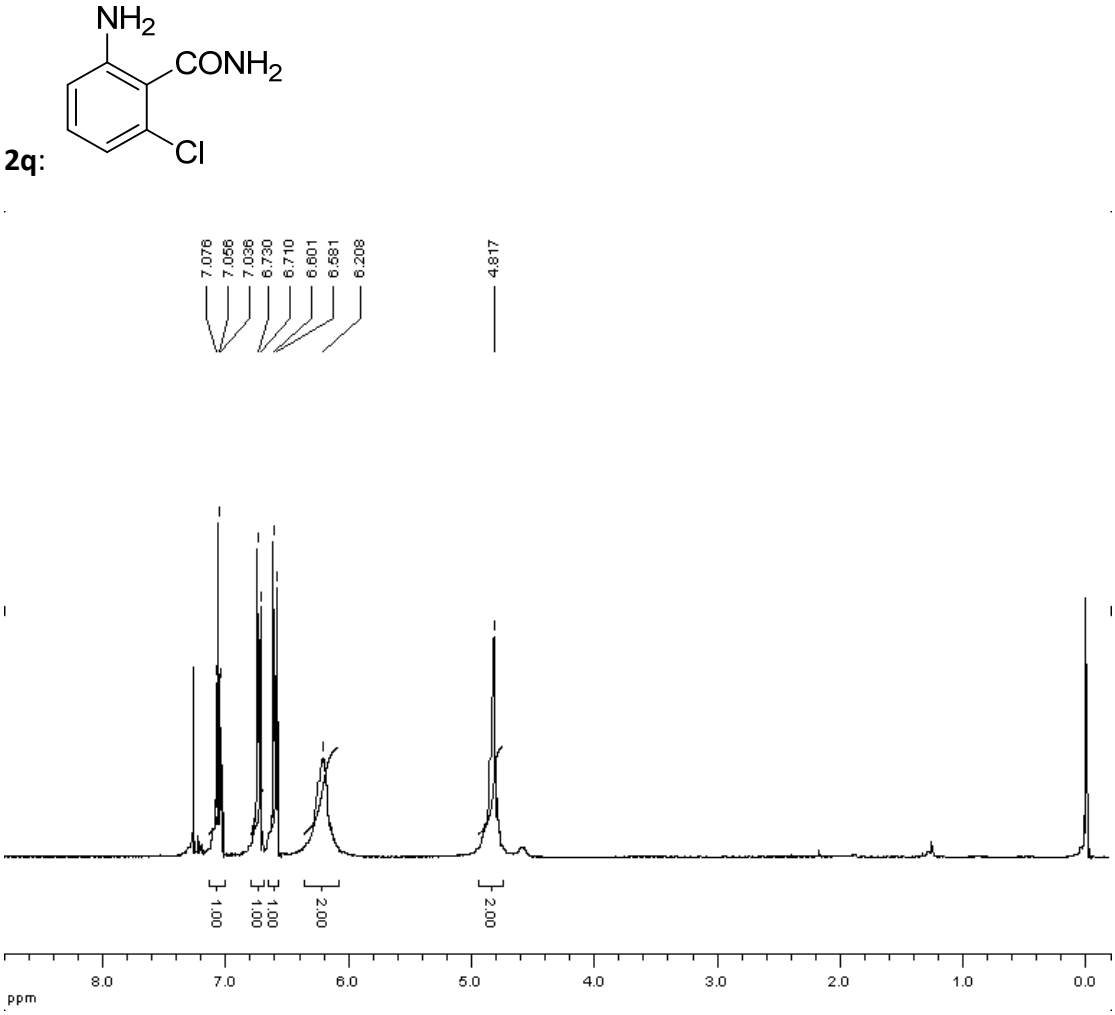
2p:

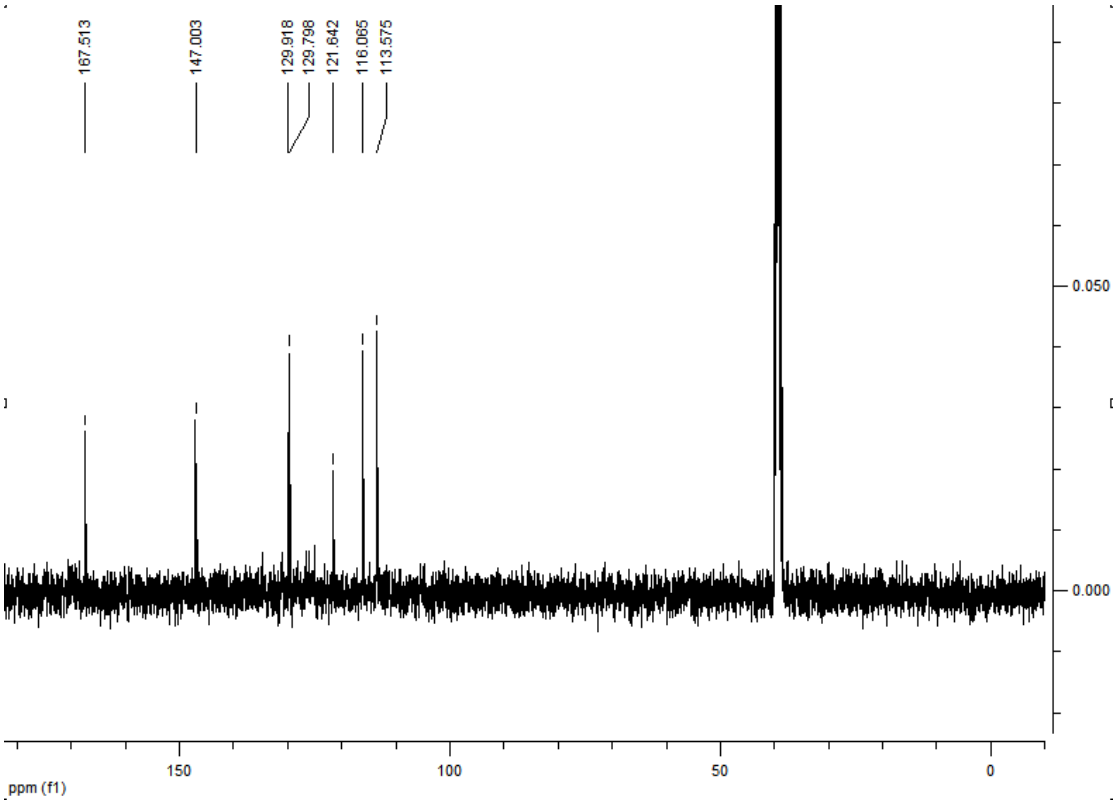


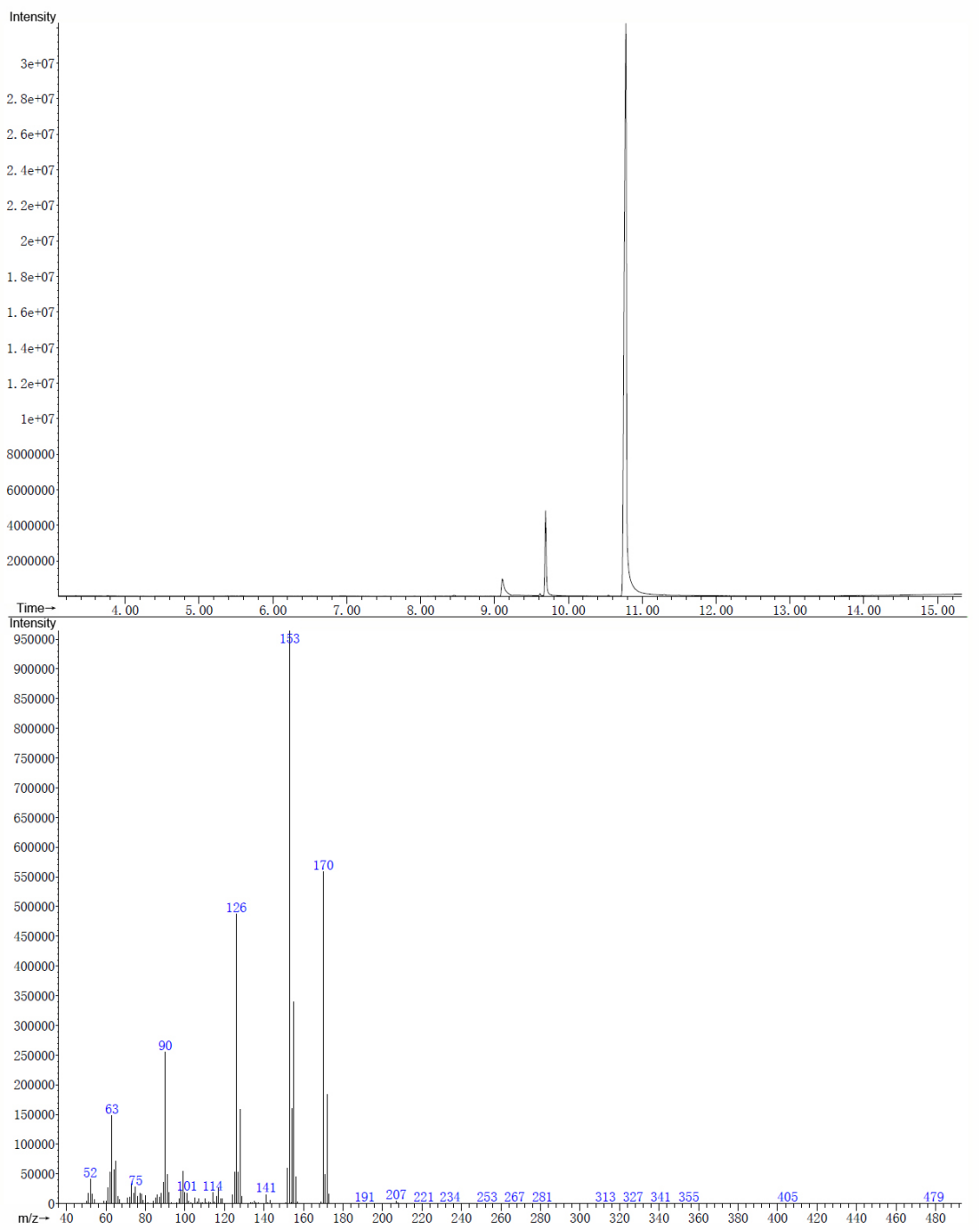

166.668
161.956
159.529
150.494
150.434
131.335
131.220
111.553
101.648
101.410
0.40
0.30
0.20
0.10
0.00
150
100
50
0
ppm (t1)


Intensity
2.2e+07
2e+07
1.8e+07
1.6e+07
1.4e+07
1.2e+07
1e+07
8000000
6000000
4000000
2000000
Time→
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
Intensity
700000
650000
600000
550000
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
m/z→
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
137
154
110
83
63
52
74
98
125
163
177
191
207
223
239
253
267
281
315
331
346
355
405

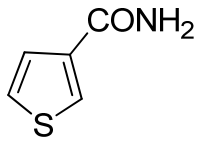
2q:

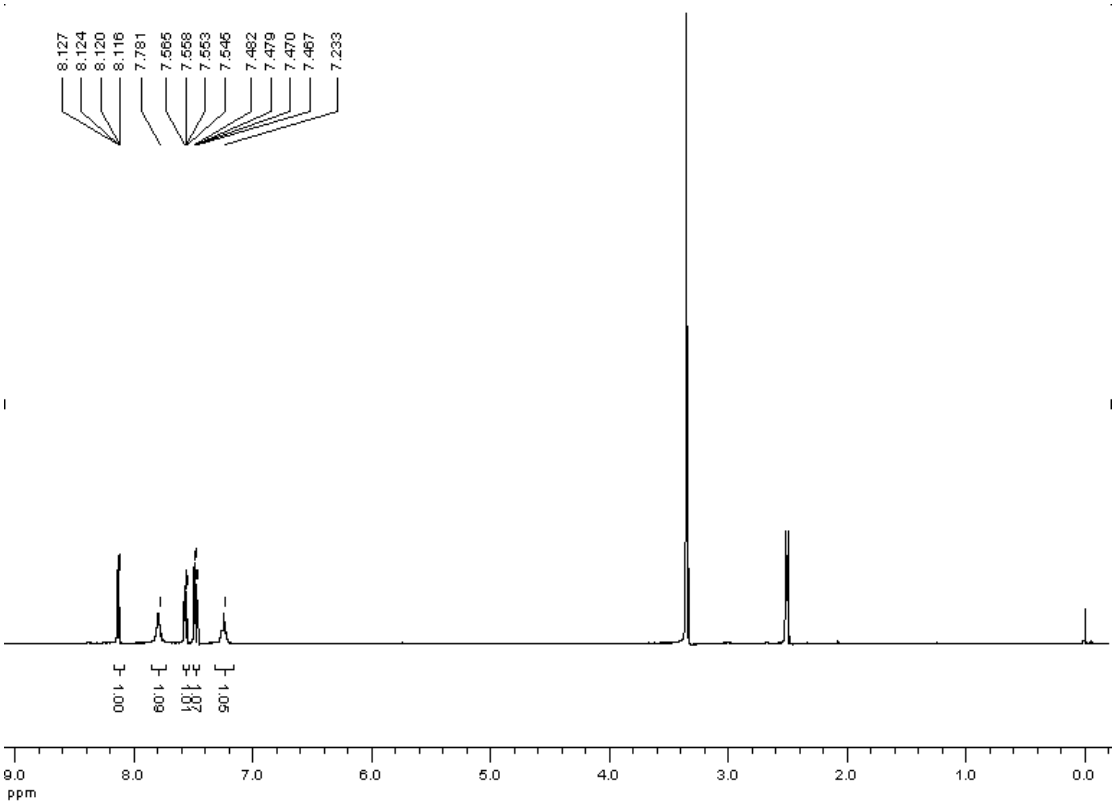


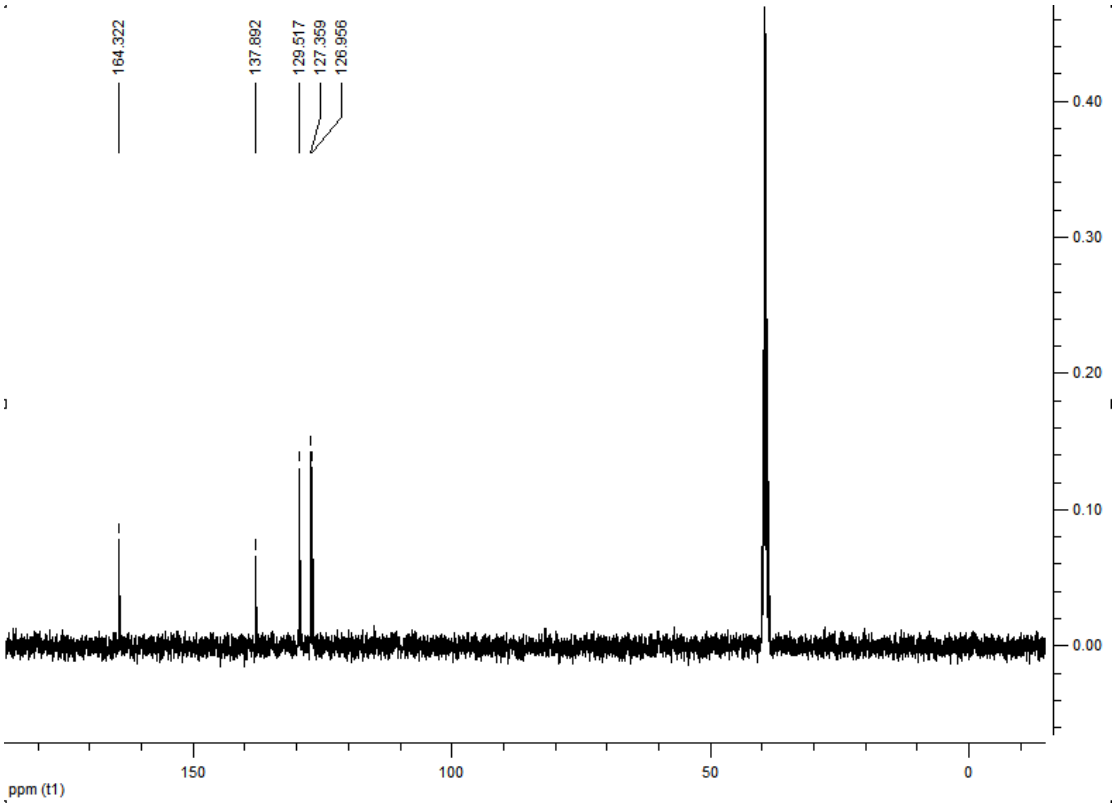

167.513
147.003
129.918
129.798
121.642
116.065
113.575
0.050
0.000
150
100
50
0
ppm (f1)

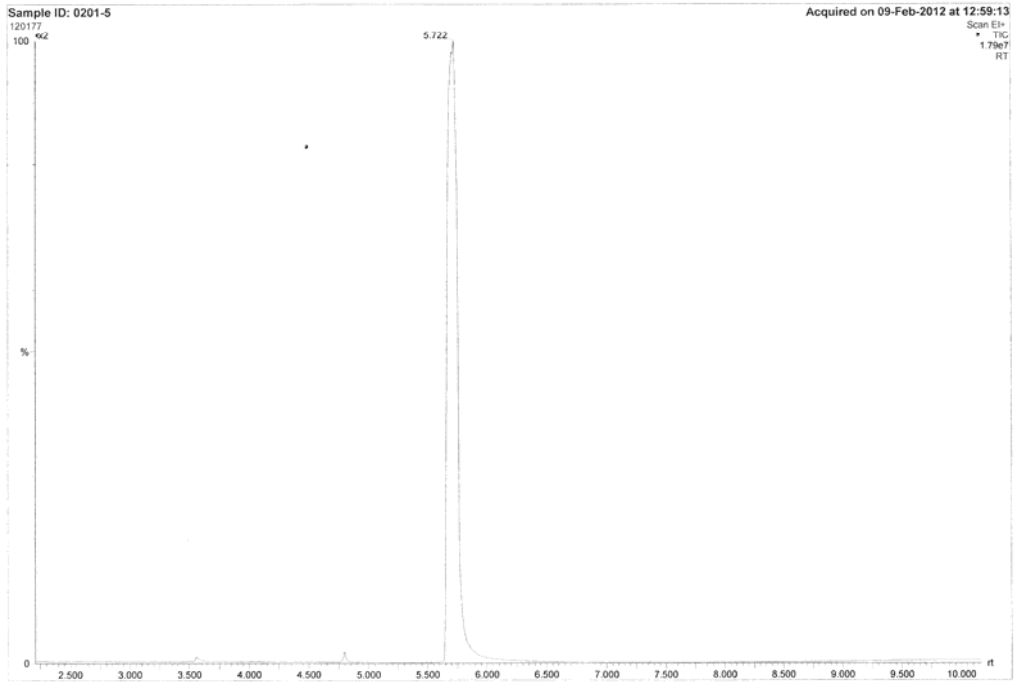

Intensity
3e+07
2.8e+07
2.6e+07
2.4e+07
2.2e+07
2e+07
1.8e+07
1.6e+07
1.4e+07
1.2e+07
1e+07
8000000
6000000
4000000
2000000
Time→
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
Intensity
950000
900000
850000
800000
750000
700000
650000
600000
550000
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
m/z→
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
460
480
153
170
126
90
63
52
75
101
114
141
191
207
221
234
253
267
281
313
327
341
355
405
479

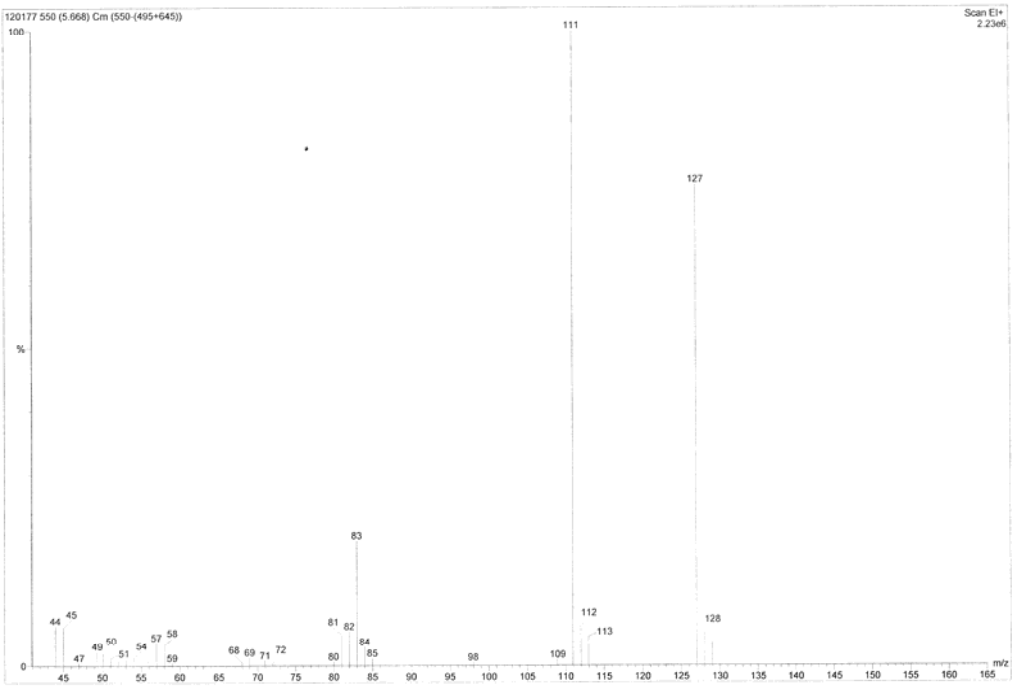
2r:


CONH2
S

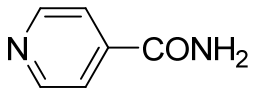

8.127
8.124
8.120
8.116
7.781
7.565
7.558
7.553
7.545
7.482
7.479
7.470
7.467
7.233
1.00
1.09
3.18
2.07
1.06
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm

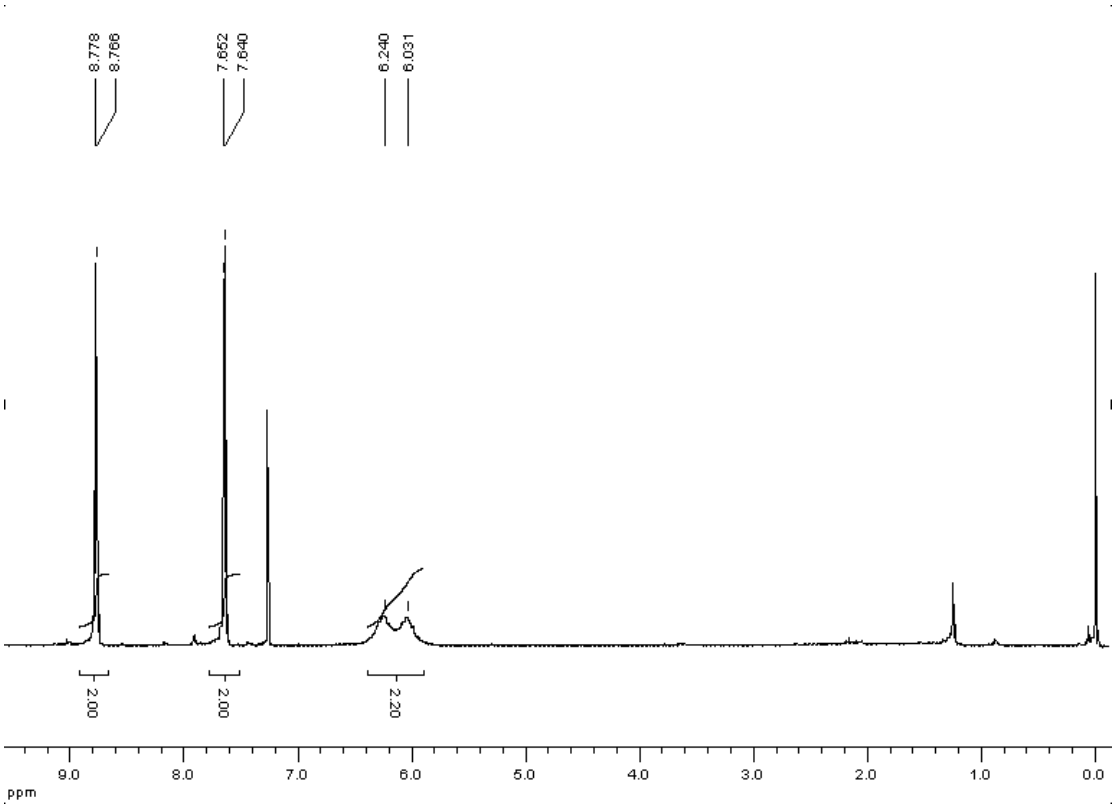

164.322
137.892
129.517
127.359
126.956
0.40
0.30
0.20
0.10
0.00
150
100
50
0
ppm (t1)

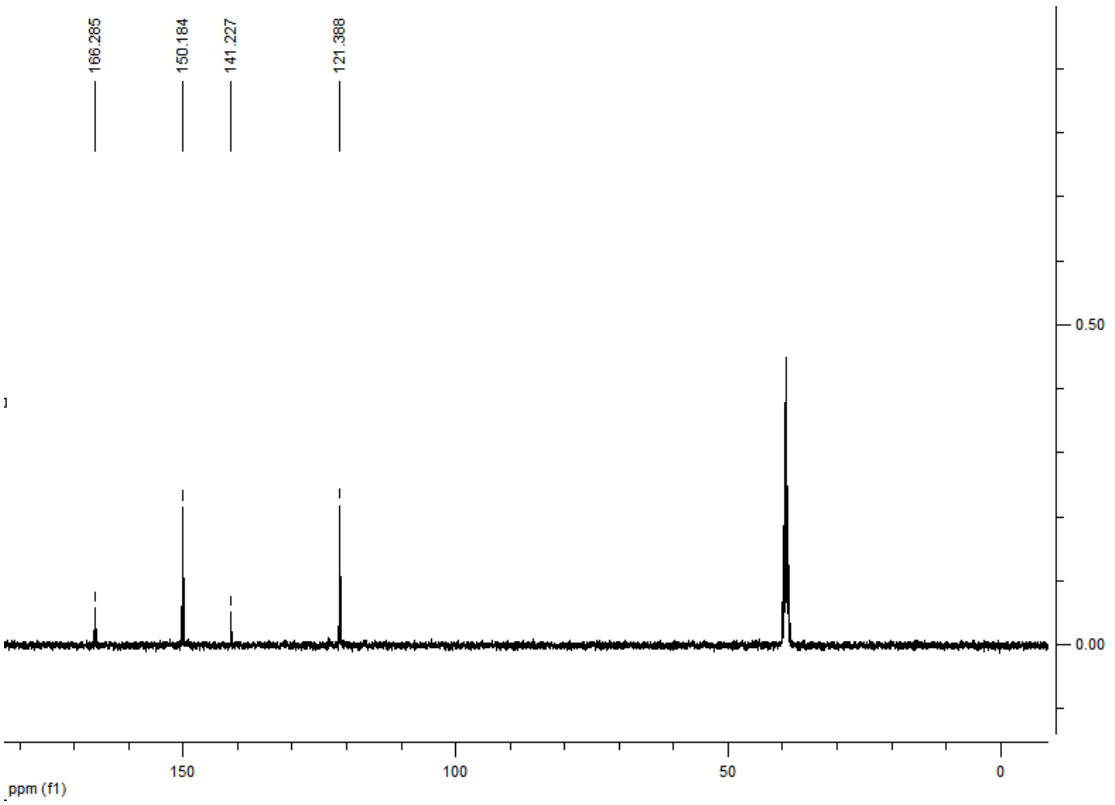

Sample ID: 0201-5
Acquired on 09-Feb-2012 at 12:59:13
120177
Scan EI+
TIC
1.79e7
RT
100
%
0
5.722
2.500
3.000
3.500
4.000
4.500
5.000
5.500
6.000
6.500
7.000
7.500
8.000
8.500
9.000
9.500
10.000
rt

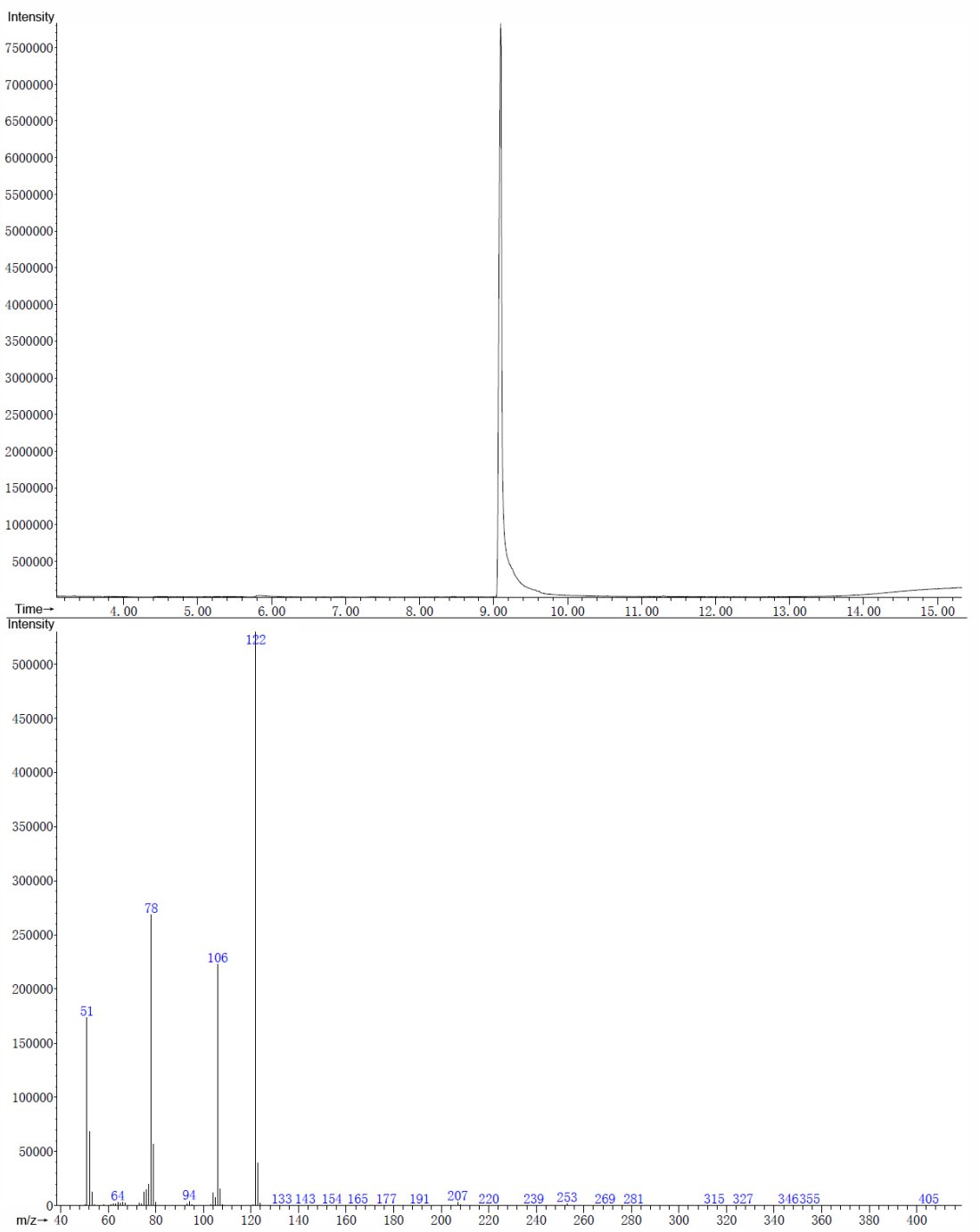

120177 550 (5.668) Cm (550-(495+645))
Scan EI+
2.23e6
100
%
0
111
127
83
112
113
128
44
45
47
49
50
51
54
57
58
59
68
69
71
72
80
81
82
84
85
98
109
45
50
55
60
65
70
75
80
85
90
95
100
105
110
115
120
125
130
135
140
145
150
155
160
165
m/z

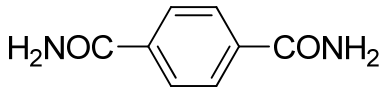
2s:


N
CONH2


8.778
8.766
7.652
7.640
6.240
6.031
2.00
2.00
2.20
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm


166.285
150.184
141.227
121.368
0.50
0.00
150
100
50
0
ppm (f1)


Intensity
7500000
7000000
6500000
6000000
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
Time→
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
Intensity
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
m/z→
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
122
78
106
51
64
94
133
143
154
165
177
191
207
220
239
253
269
281
315
327
346
355
405



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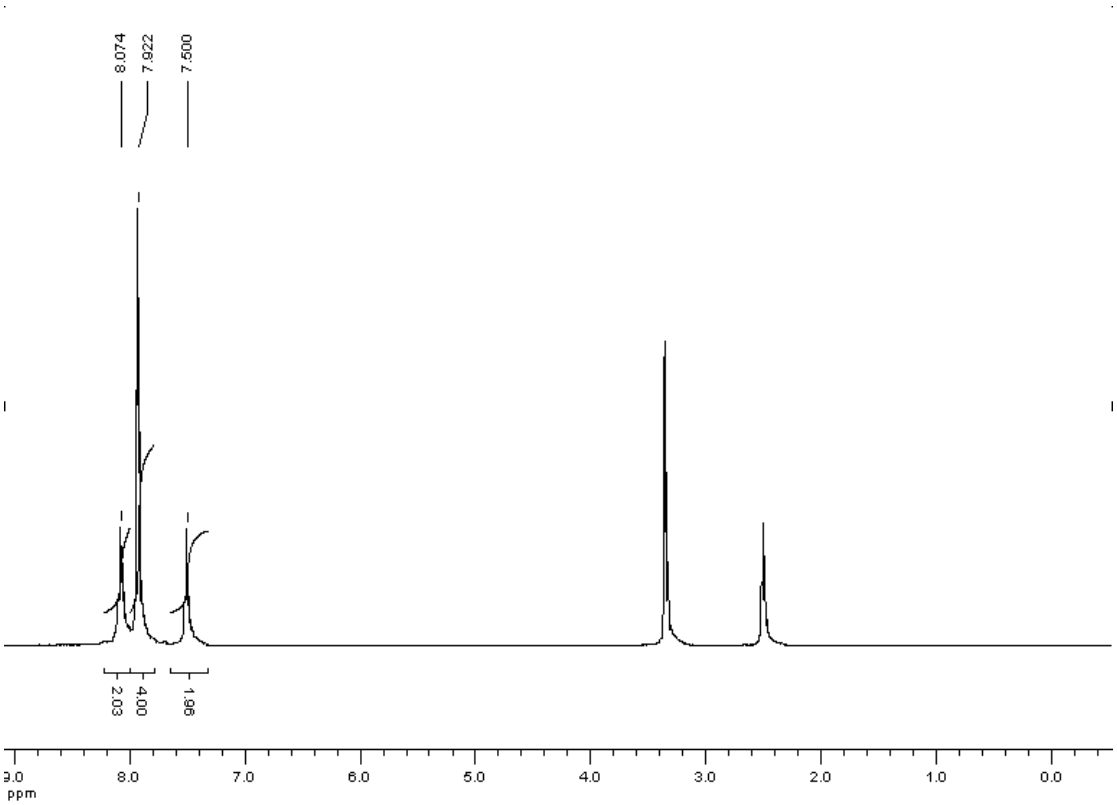

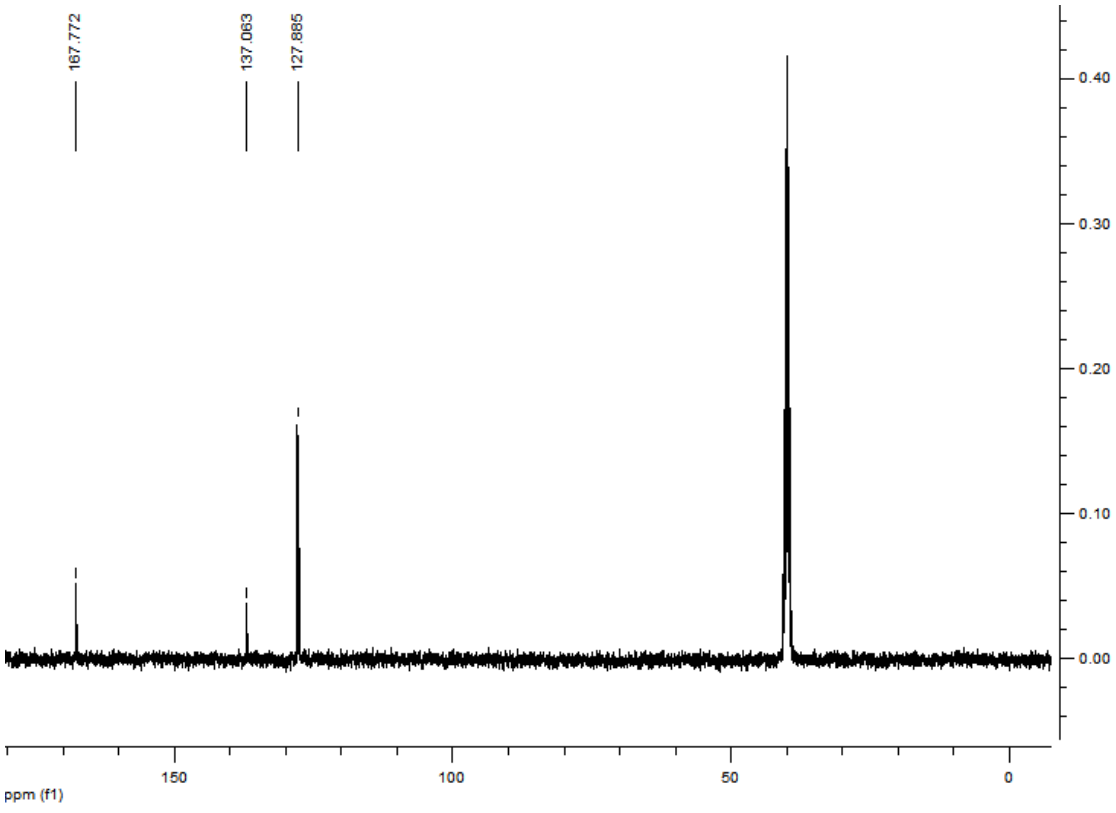

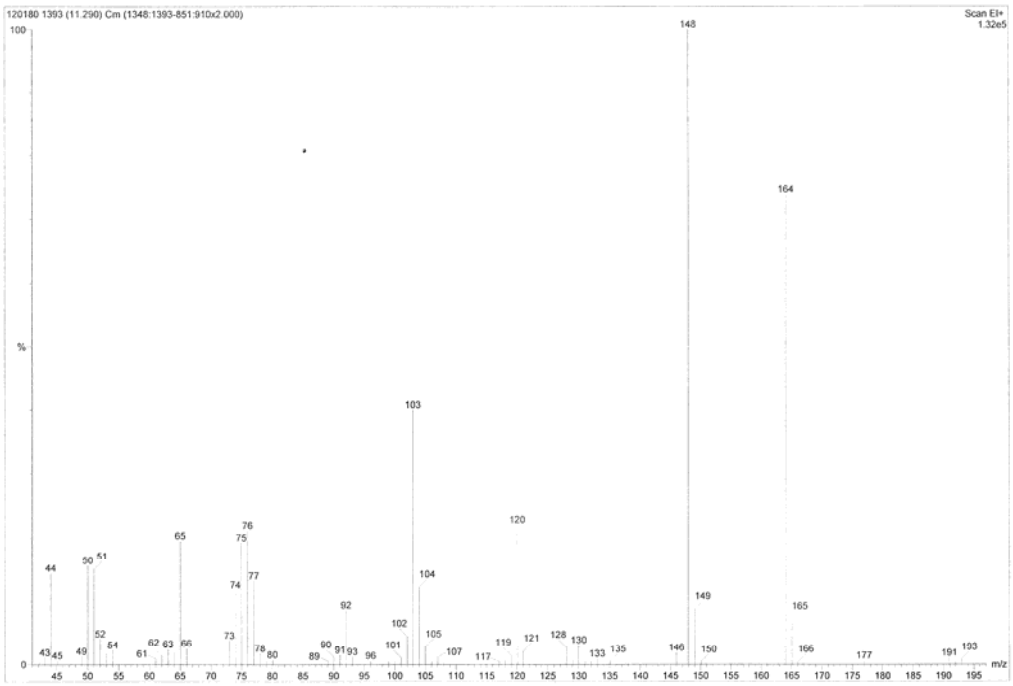

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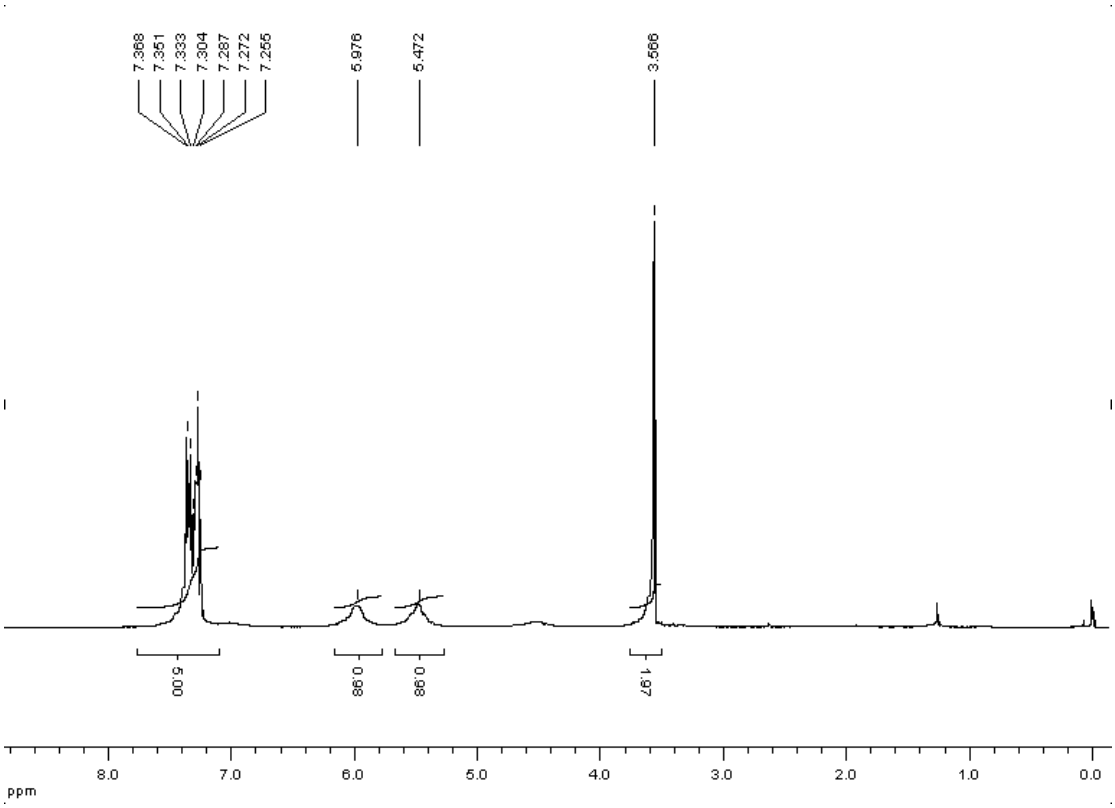

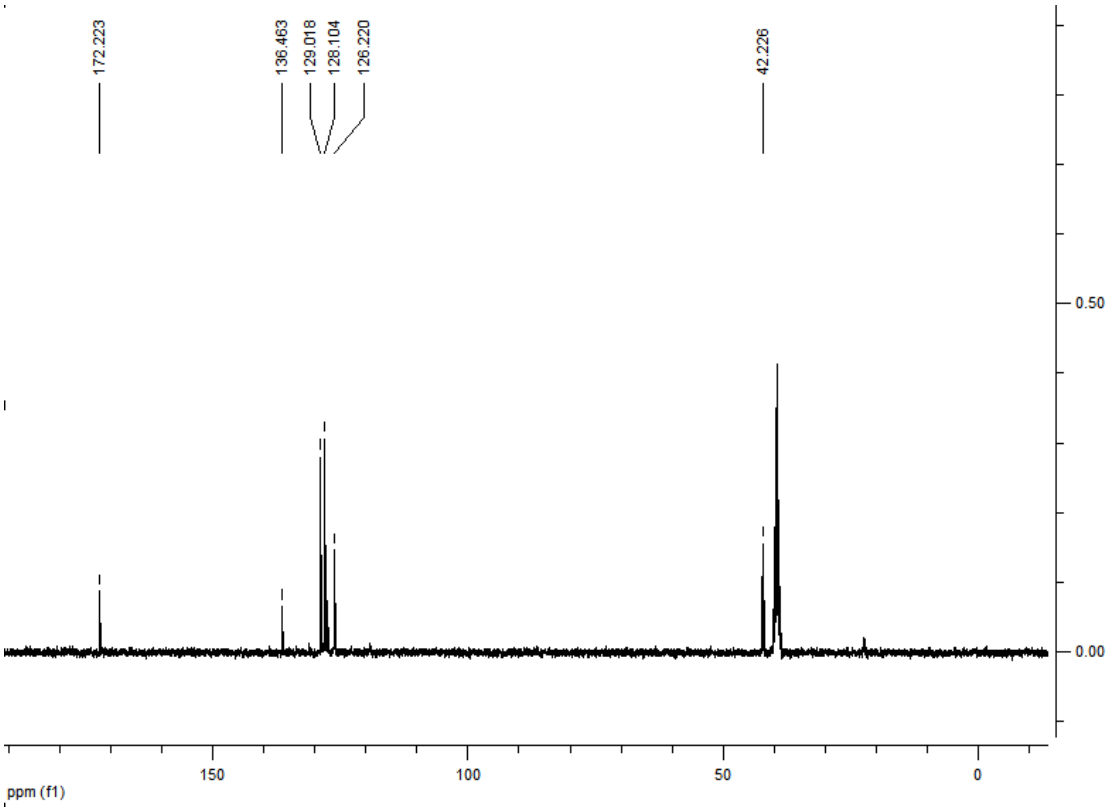

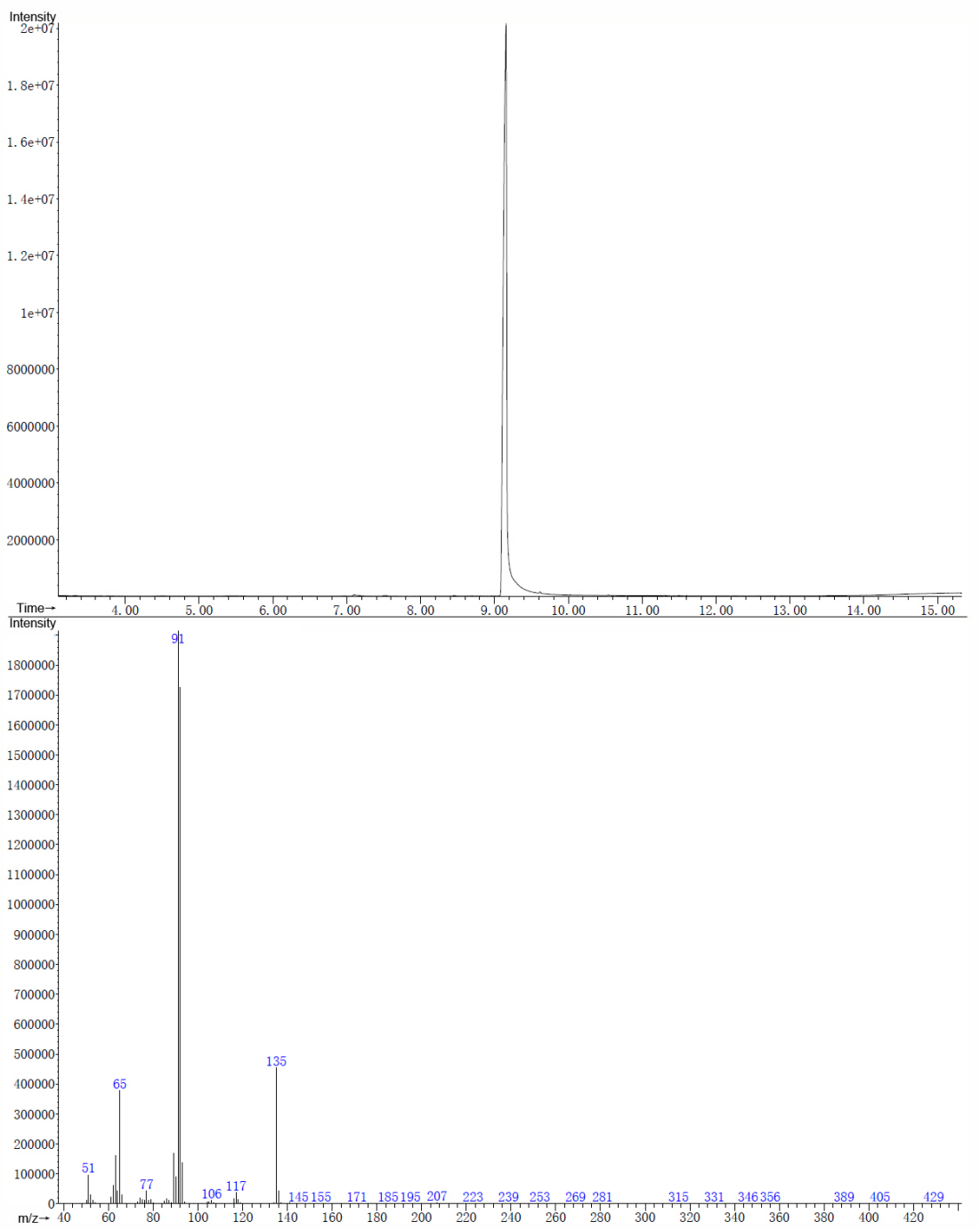
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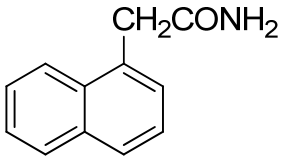
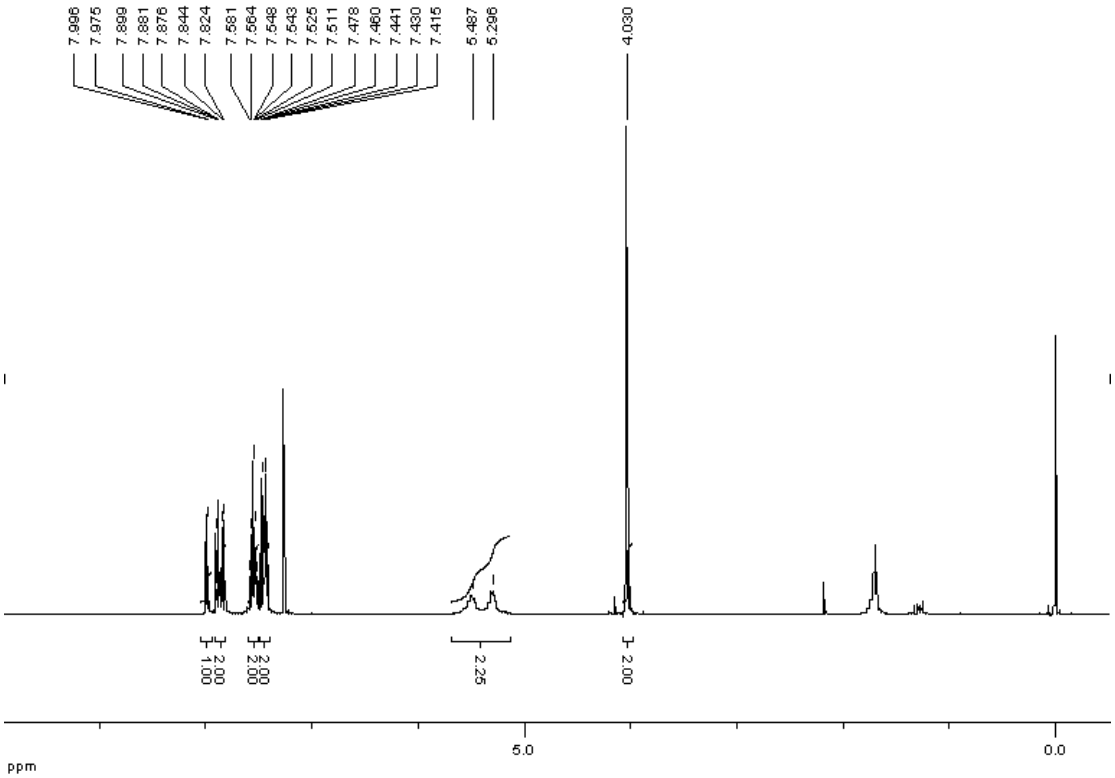
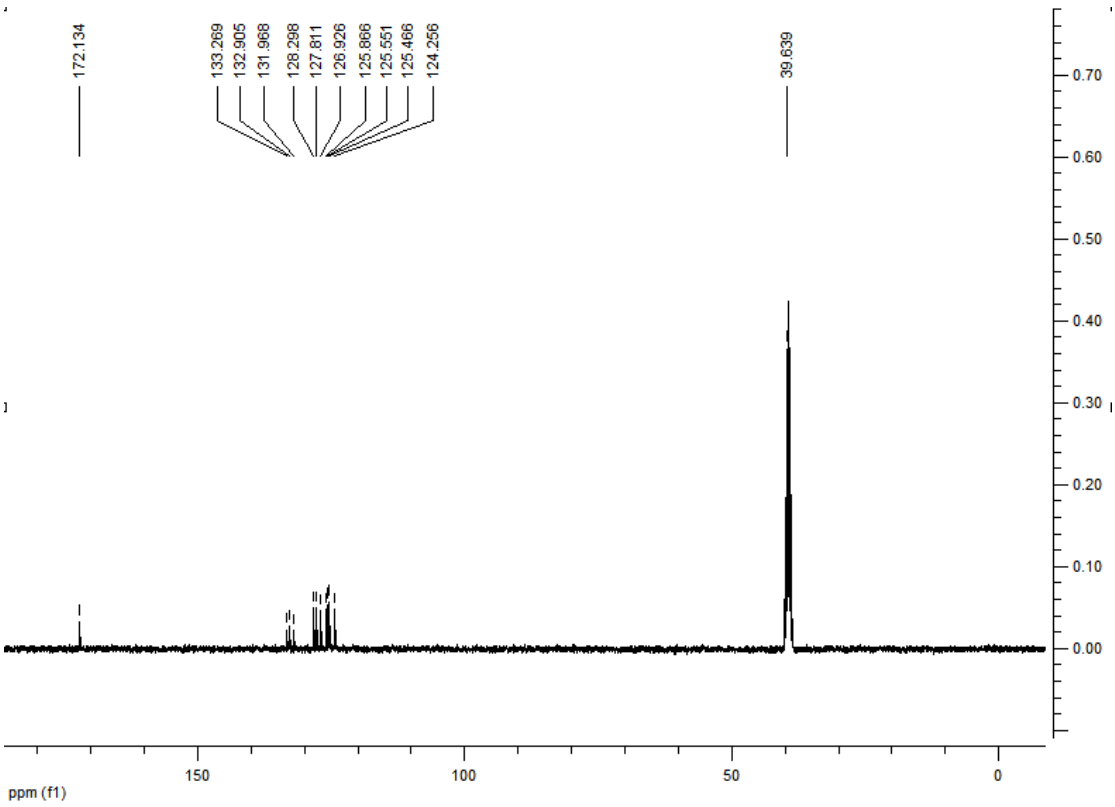
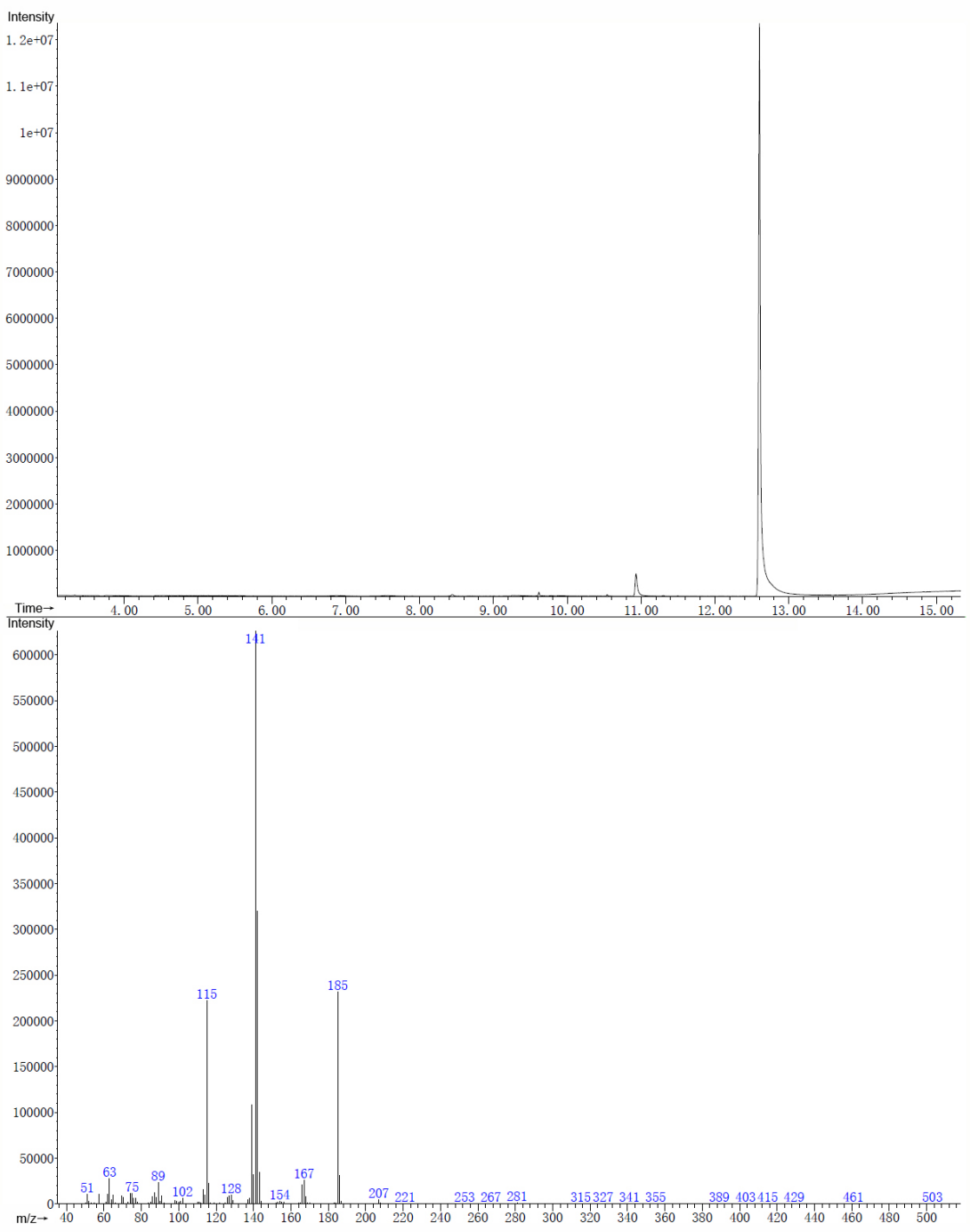
2e+07
1.8e+07
1.6e+07
1.4e+07
1.2e+07
1e+07
8000000
6000000
4000000
2000000
Time→
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
Intensity
91
135
65
51
77
106
117
m/z→
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420

2v:








Intensity
1.2e+07
1.1e+07
1e+07
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
Time→
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00
15.00
Intensity
600000
550000
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
m/z→
141
115
185
63
51
75
89
102
128
154
167
207
221
253
267
281
315
327
341
355
389
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415
429
461
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420
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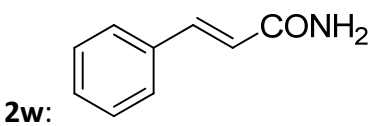
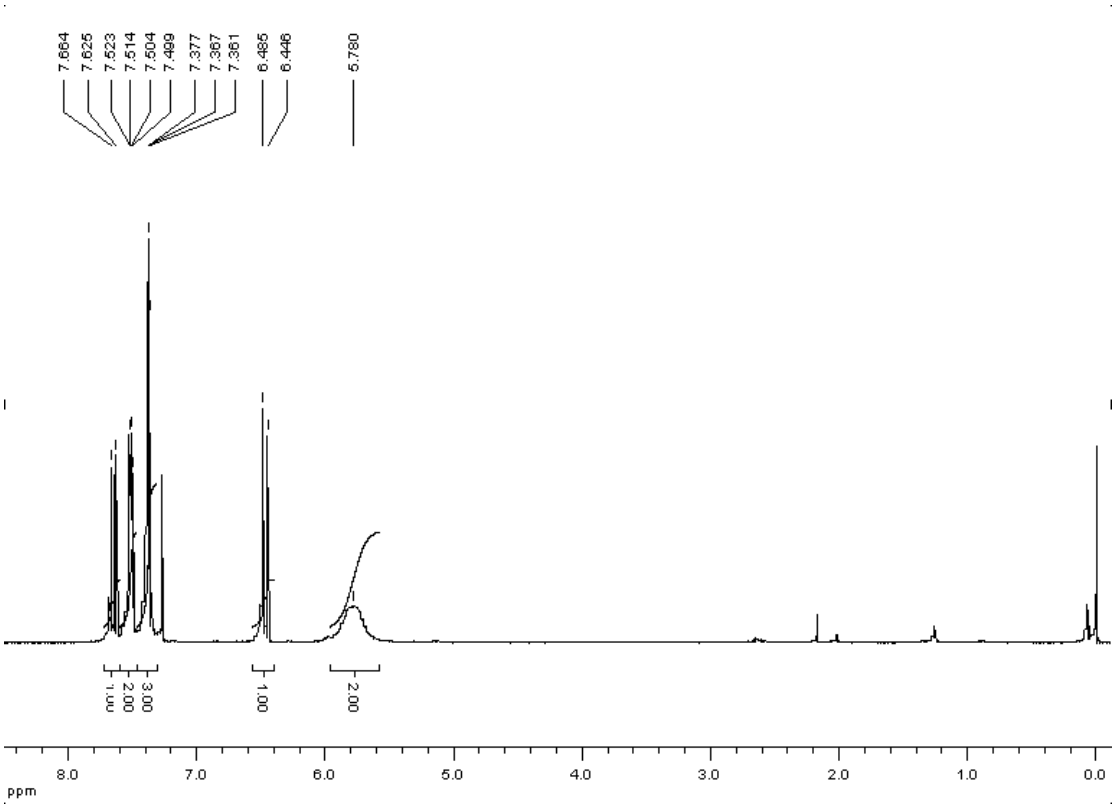
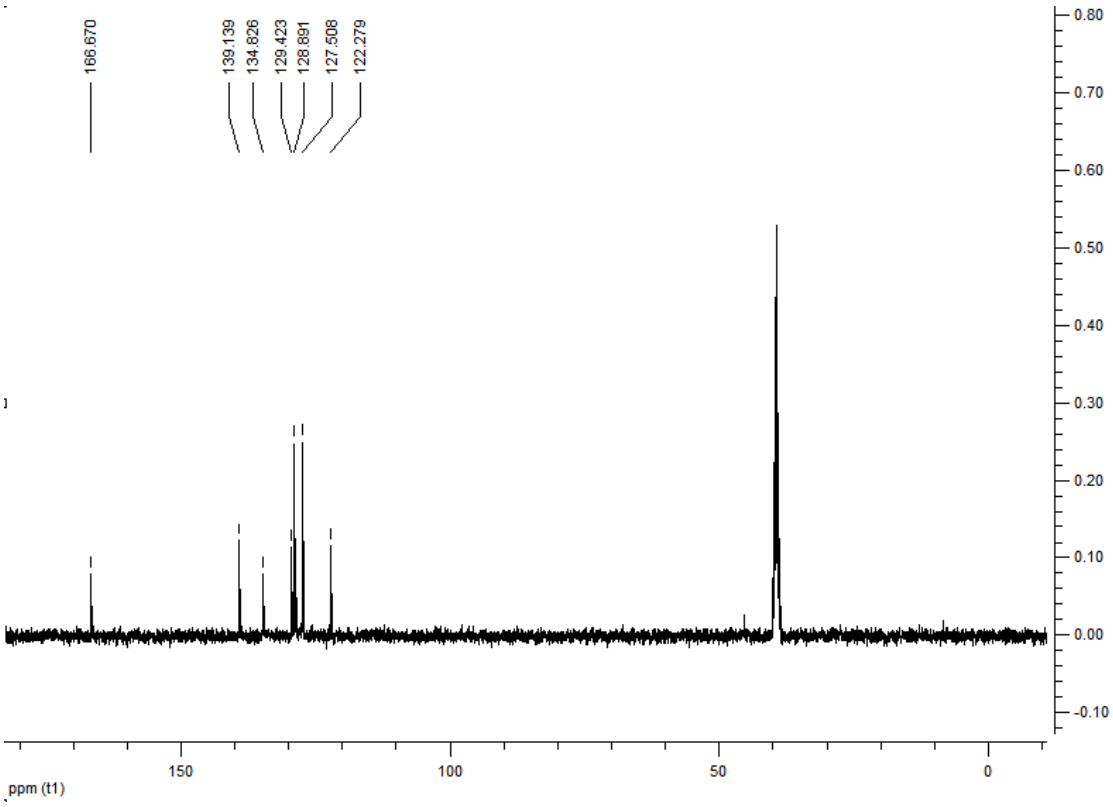
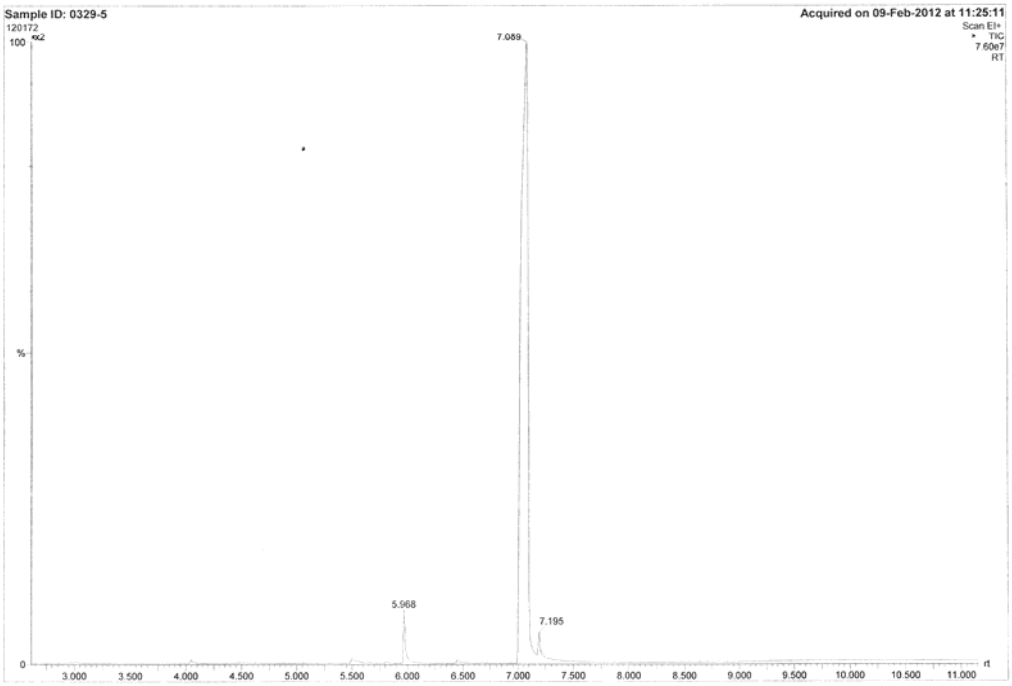
480
500

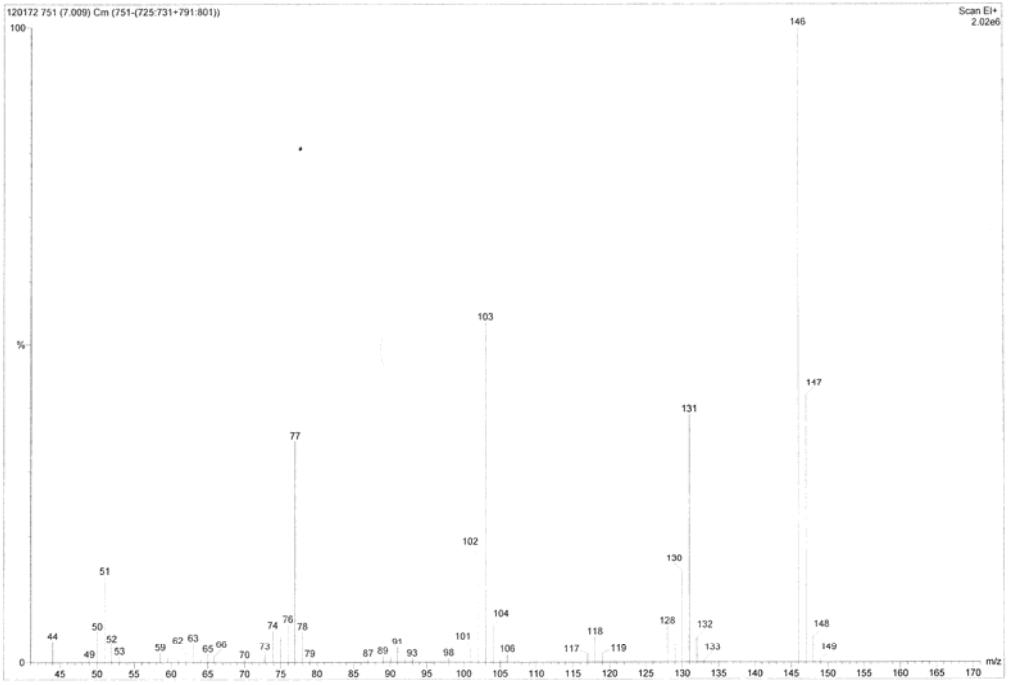
2w:

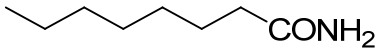


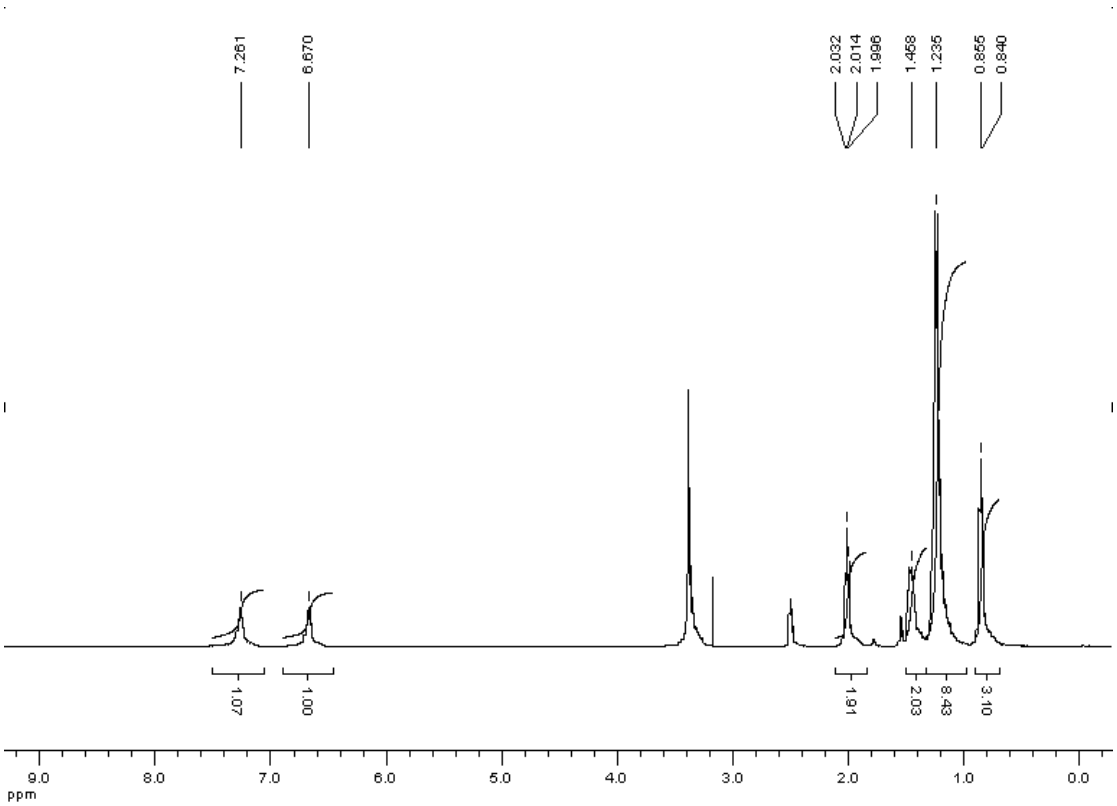


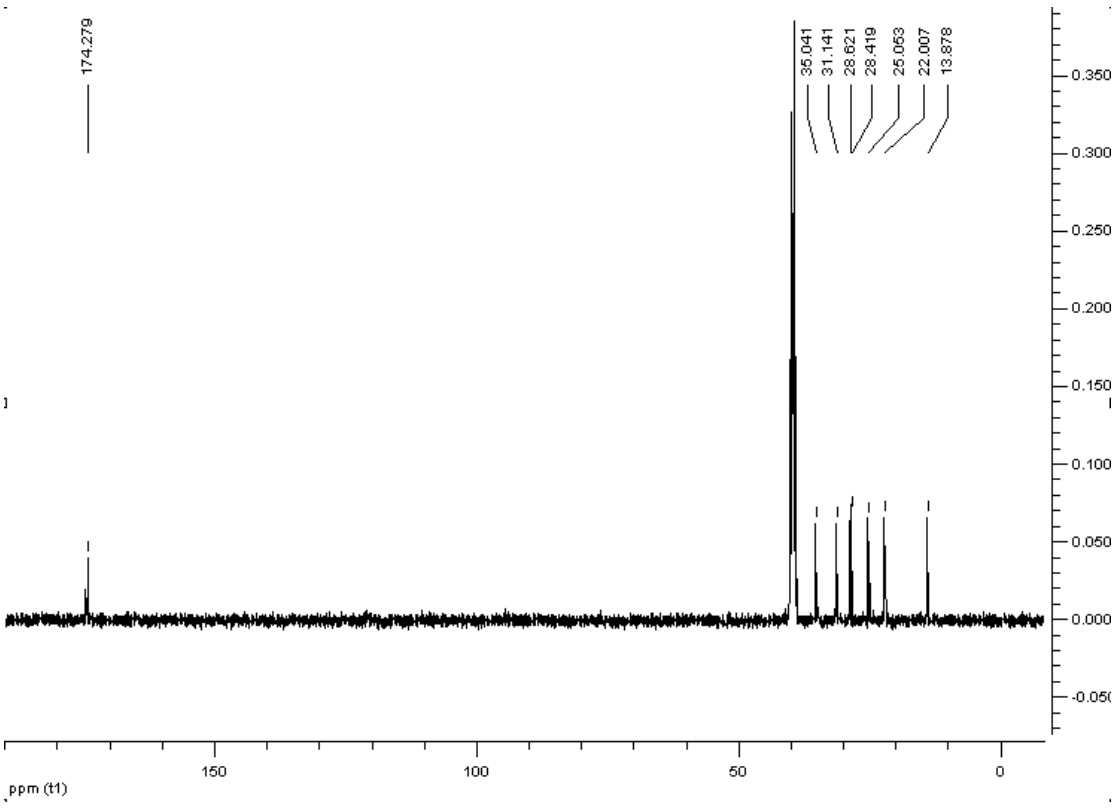


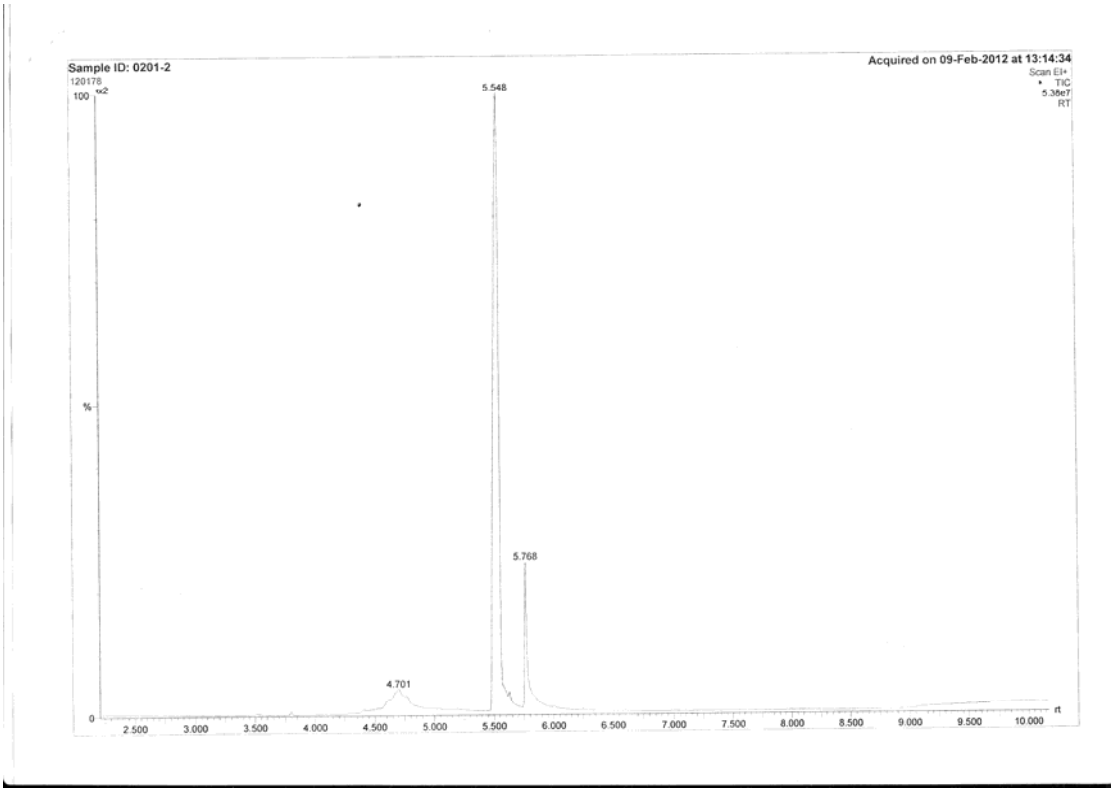

Sample ID: 0329-5
Acquired on 09-Feb-2012 at 11:25:11
120172
Scan EI+
TIC
7.60e7
RT
7.089
5.968
7.195
%
rt

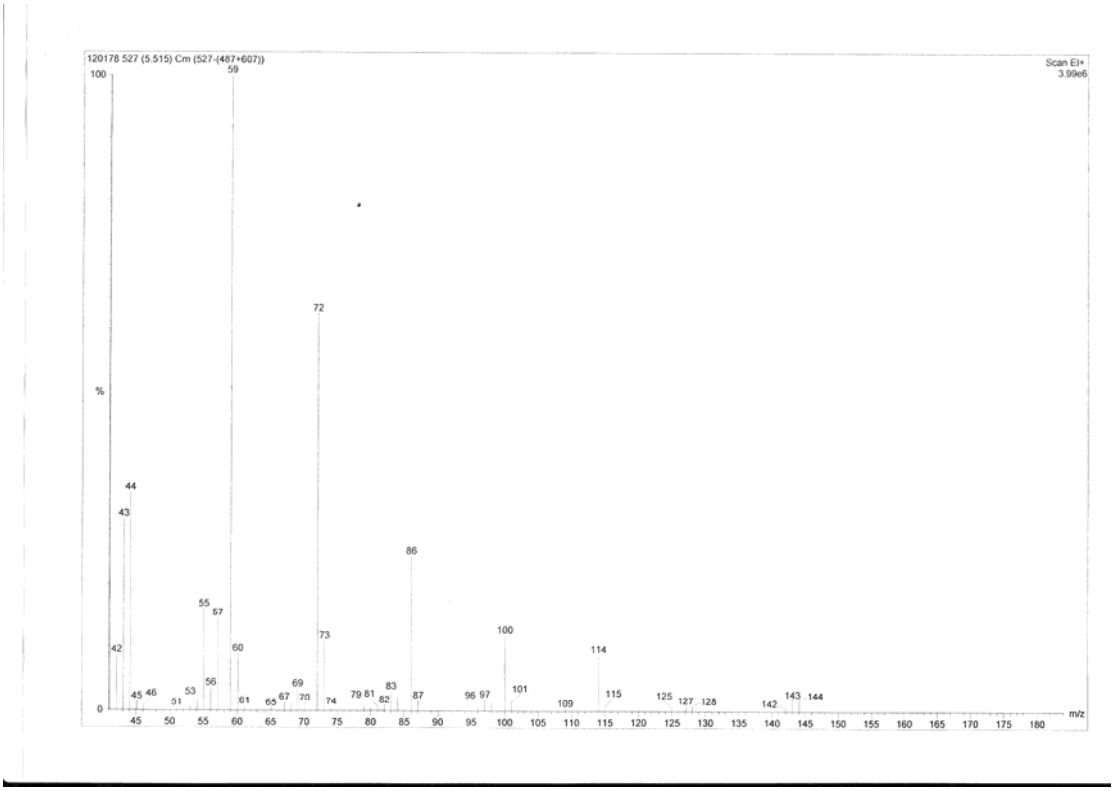

120172 751 (7.009) Cm (751-(725:731+791:801))
Scan EI+
2.02e6
146
103
147
131
77
102
130
51
104
128
132
148
118
101
106
117
119
133
149
%
m/z

2x: 






Sample ID: 0201-2
120178
Acquired on 09-Feb-2012 at 13:14:34
Scan EI+
TIC
5.38e7
RT
100
%
0
5.548
5.768
4.701
rt
2.500
3.000
3.500
4.000
4.500
5.000
5.500
6.000
6.500
7.000
7.500
8.000
8.500
9.000
9.500
10.000


120178 527 (5.515) Cm (527-(487+607))
Scan EI+
3.99e6
100
%
0
59
72
44
43
86
55
57
60
73
100
114
42
56
69
83
45
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