Electronic Supplementary Material (ESI) for Green Chemistry.

Facile synthesis of controllable graphene-coshelled reusable Ni/NiO nanoparticles and application for synthesis of amines under mild conditions

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S1. Methods and materials

All aldehydes, ketones, nitroarenes and amines were obtained commercially from various chemical companies. 1,3,5 trimethoxybenzene was purchased from Sigma Aldrich Co., Ltd.; nickel(II) nitrate hexahydrate (AR, 98%), citric acid (AR,≥99.5%), anhydrous ethanol (99.5%) were obtained from Shanghai Macklin Biochemical Co., Ltd.; H_2SO_4 (GR, 98%) was purchased from Sinopharm Chemical Reagent Co.,Ltd.;Al_2O_3 (basic, 200~300 meshes), 5 wt% Pt/C, 5 wt% Pd/C, 10 wt% Pd/C, and 5 wt% Ru/C catalysts were purchased from Aladdin Co., Ltd.; $CDCl_3$ (99.8% D, containing 0.03% TMS, stabilized with Ag)was applied by Meryer Chemical Technology Co., Ltd. Before using, the purity of aldehydes and ketones has been checked.

GC were recorded by GC-2014C (Shimadzu, Japan) column with HP5 (30 m × 250 mm × 0.25 μm) and FID detector. GC-MS was determined by TRACE 1300ISQ GC-MS (Thermo Fisher Scientific, America). ^{1}H and ^{13}C NMR were recorded on a Bruker AVANCE III 400 MHz spectrometer using $CDCl_3$ solvents. The TEM measurements were performed on a JEM-2100F microscope opeated at 200 kV. The SEM was conducted on Analytical SEM SU-70 (Hitachi, Japan) operated at 10kV. XPS data were conducted on a Escalab250 Xi photoelectron spectrometer (Thermo Fisher Scientific, America) equipped with an Al Kα X-ray source with scan number of 5. The peak position was calibrated with C 1s peak at 284.8 eV. XRD powder patterns were recorded on an X'Pert Pro MPD (PANalytical, Netherlands) using the Cu Kα radiation source. Processing and assignment of the powder patterns was done on the software Jade 6.0 and using the Powder Diffraction File (PDF) database of the International Centre of Diffraction Data (ICDD). Nitrogen adsorption isotherms were measured at -196 °C QUADRASORB SI apparatus (Quantachrome, America) apparatus by using the BET method. Raman spectra were measured at LabRAM HR800 apparatus (HORIBA JY, France) with wavelength 100~3200 cm^{-1}.

S2. General preparation of catalysts

S2.1. Preparation of Ni@C-H_2O

In a typical synthesis procedure, nickel (II) nitrate hexahydrate (Ni $(NO_3)_2$·6 H_2O,0.3 mol) and citric acid ($C_6H_8O_7$, 0.3 mol) were dissolved in deionized water (5 mL). The mixture was then aged at 70ºC for 8 h under stirring (300 rpm) until to obtain a transparent greenish gel, followed by drying at 100ºC for 72 h in a drying oven to remove the excess water. The obtained greenish solid was then calcining at fixed bed at 600 or 700 ºC for 3 h under a high-purity N_2 (99.999 %) flow of 40 mL min^{-1}. The heating rate was controlled at 2 ºC min^{-1}. The obtained black solids were treated in 1M H_2SO_4 aqueous solution at 70ºC until the solution colorless to remove the insecure and uncovered Ni particles. The black solids were then full washed with deionized water until pH of waste solution was 7. Then, the black solids were dried at -48 ºC for 12 h in vacuum by using a freeze dryer. The dried black solids denoted as Ni@C-x-H_2O, where x is calcining temperature of greenish gel, such as Ni@C-600-H_2O or Ni@C-700-H_2O.

S2.2. Preparation of Ni@C-EtOH

In a typical synthesis procedure, nickel nitrate hexahydrate ($Ni(NO_3)_2$·6 H_2O, 0.3 mol) and citric acid ($C_6H_8O_7$, 0.3 mol) were dissolved in anhydrous ethanol (5 mL). The mixture was then aged at 70 ºC for 4 h under stirring (300 rpm) until to obtain a bubble-shaped greenish gel, followed by drying at 100ºC for 24 h in a drying oven to remove the excess solvent. The obtained greenish solid was then calcining at fixed bed at 600 or 700 ºC for 3 h under a high-purity N_2 (99.999 %) flow of 40 mL·min^{-1}. The heating rate was controlled at 2 ºC·min^{-1}. The obtained black solids were treated in 1M H_2SO_4 aqueous solution at 70ºC until the solution colorless to remove the insecure and uncovered Ni particles. The black solids were then full washed with deionized water until pH of waste solution was 7. Finally, the black solids were dried at -48 ºC for 12 h in vacuum by using a freeze dryer. The dried black solids denoted as Ni@C-x-EtOH, where x is calcining temperature of greenish gel, such as Ni@C-600-EtOH and Ni@C-700-EtOH.

S2.3. Preparation of Ni-NiO@Cs

In a typical synthesis procedure, Ni@C-H_2O and Ni@C-EtOH were grinded and sieved to powders of 800 meshes and then were heated at fixed bed at 200 ºC for 1 or 2 h under argon (containing 1 vol% O_2) flow of 30 mL·min^{-1}. All these materials were labeled as M1/M2@C-x-y-z-s, where M1= Ni; M2=NiO; x denotes the pyrolysis temperature; y denotes the oxidation temperature; z denotes the oxidation time; and s denotes the solvent, respectively.

S3. General procedure of the synthesis of amines

S3.1. General procedure for the synthesis of primary amines

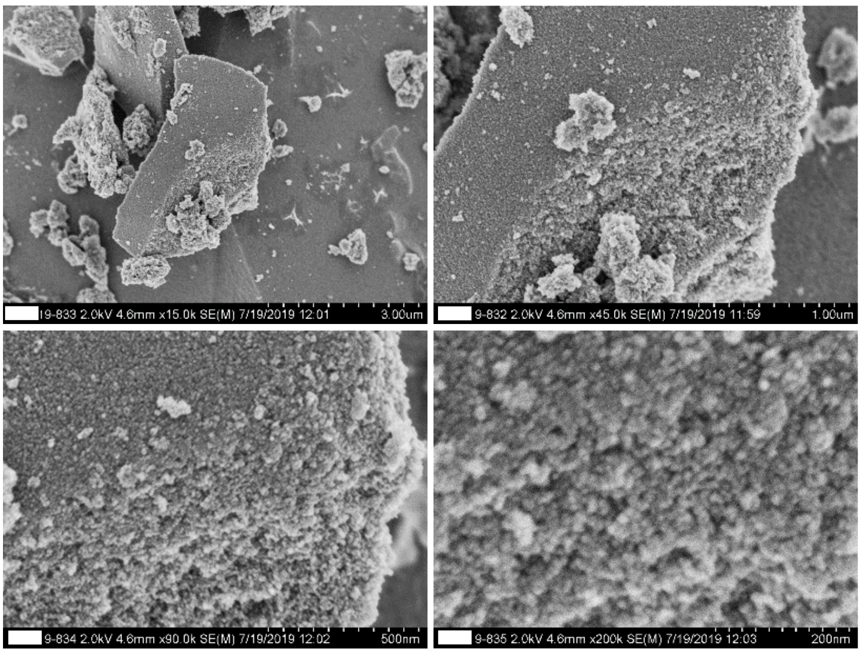
The reaction was proceeding in a stainless-steel autoclave with six wells (20 mL per well), one thermocouple and one circulating water-cooling equipment. Each well was loaded with one 10 mm magnetic stirring bar, 0.5 mmol corresponding carbonyl compound, 0.05~0.1mmol 1,3,5-trimethoxybenzene, 10 mg M1/M2@C-x-y-z-s catalyst, and 5mL ammonia solution (2 M in methanol). Then, the autoclave was sealed and flushed with hydrogen (H_2) three times at 2 MPa pressure and it was pressurized with 2 MPa H_2. The autoclave was placed into a heating mantle and the stirring was set at 400 rpm. The autoclave was heated from room temperature to target temperature (inside temperature) at rate of 2 ºC·min^{-1} and keep the target temperature for require time. The target temperature was used as the reaction temperature. After the reaction, the autoclave was cooled down to room temperature. The waste gas was discharged then the reaction solutions were collected with a dropper and filtered. The catalyst was immobilized on the magnetic stirring bar and can be recycled by a magnet. The catalyst was washed thoroughly with ethanol and water. The reaction products were identified by GC-MS and ^{1}H NMR and the yields of reaction products were determined by ^1H NMR. For ^1H NMR analysis, about 2 mL reaction solution was concentrated under a N_2-flow, and then added 0.5 mL of $CDCl_3$ which preneutralized with basic Al_2O_3.

1 g scale experiments were carried out in a 500 mL stainless steel autoclave with one thermocouple. In a typical experiment, 1g carbonyl compound, 0.35 mmol 1,3,5-trimethoxybenzene, one 30 mm magnetic stirring bar, and Ni/NiO@C-700-200-2h-EtOH (10mg per 0.5 mmol carbonyl compound) and ammonia solution (2 M in methanol, 5 mL per 0.5 mmol carbonyl compound) were loaded into the reactor. The procedure of reaction and product analysis were similar to the 0.5 mmol scale experiments.

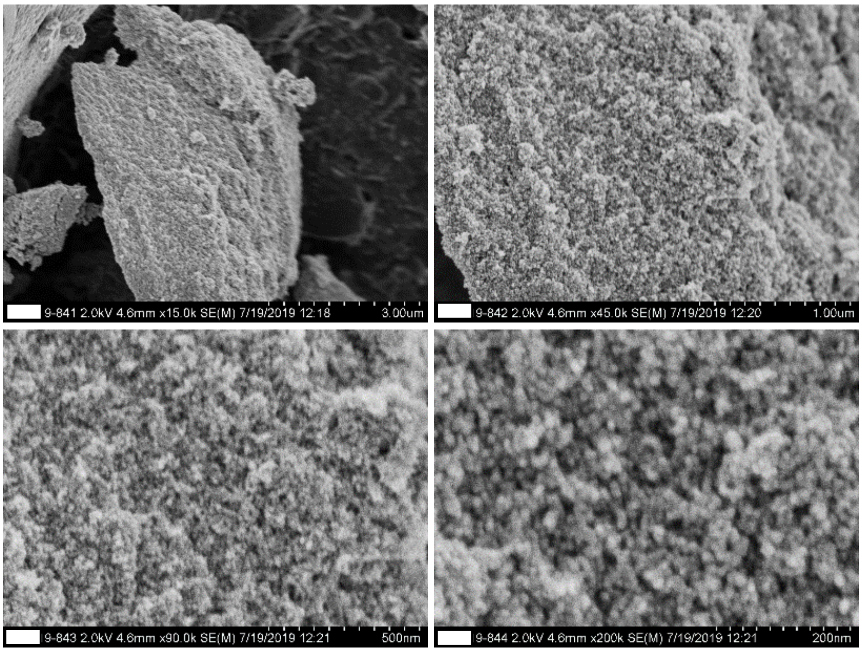
S3.2. General procedure for the synthesis of secondary amines and tertiary amines

The reaction was proceeding in a stainless steel autoclave with six wells (20 mL per well), one thermocouple and one circulating water cooling equipment. Each well was loaded with one 10 mm magnetic stirring bar, 0.5-3 mmol corresponding carbonyl compound, 0.5-0.75 mmol corresponding amine, 0.05~0.1 mmol 1,3,5-trimethoxybenzene, 10 mg Ni-NiO@C-700-200-2h-EtOH, and 5 mL methanol. Then, the autoclave was sealed and flushed with hydrogen (H_2) three times at 1-2MPa pressure and it was pressurized with 1-2MPa H_2. The autoclave was placed into a heating mantle and stirring the reaction solution at 400 rpm. The autoclave was heated from room temperature to target temperature (inside temperature) at rate of $2^oC\cdot min^{-1}$ and keep the target temperature for require time. The target temperature was used as the reaction temperature. After the reaction, the autoclave was cooled down to room temperature. The waste gas was discharged then the reaction solutions were collected with a dropper and filtered. The catalyst was immobilized on the magnetic stirring bar and can be recycled by a magnet. The catalyst was washed thoroughly with ethanol and water. The reaction products were identified by GC-MS and ^1H NMR and the yields of reaction products were determined by ^1H NMR on a BRUKER AVANCE III 400 MHz spectrometer. For ^1H NMR analysis, about 2 mL reaction solution was concentrated under a N_2-flow, and then added 0.5 mL of $CDCl_3$ which preneutralized by Al_2O_3.

S4. TEM spectra



Ni@C-600-H_2O



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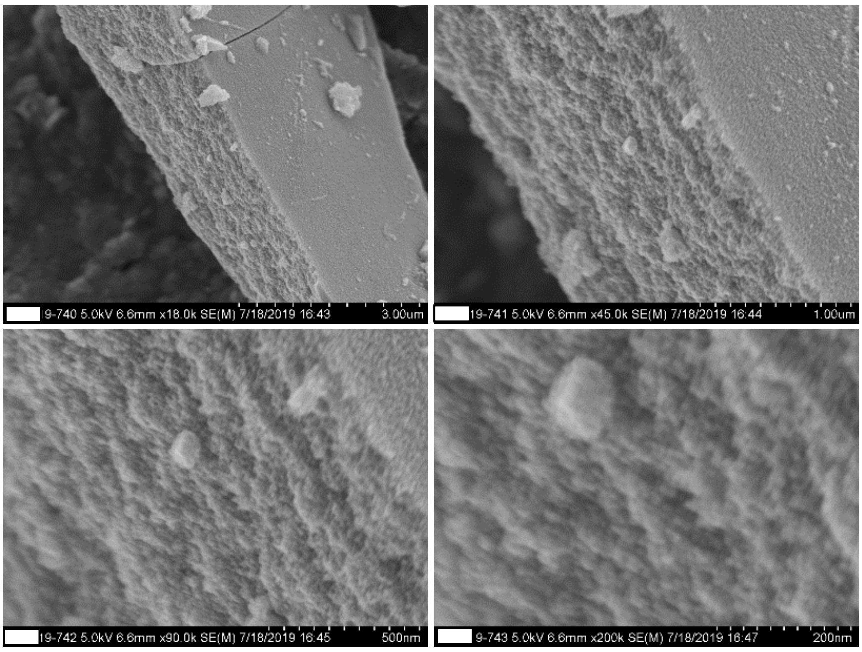

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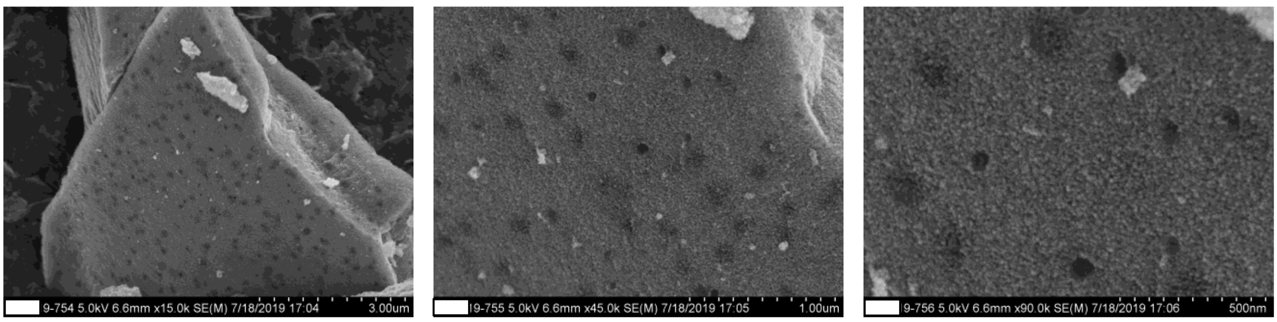

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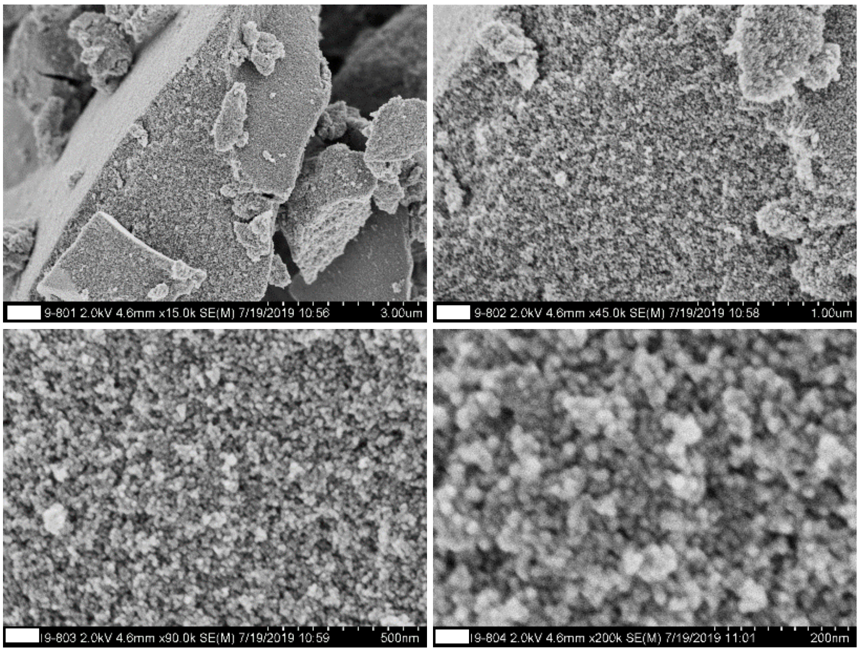

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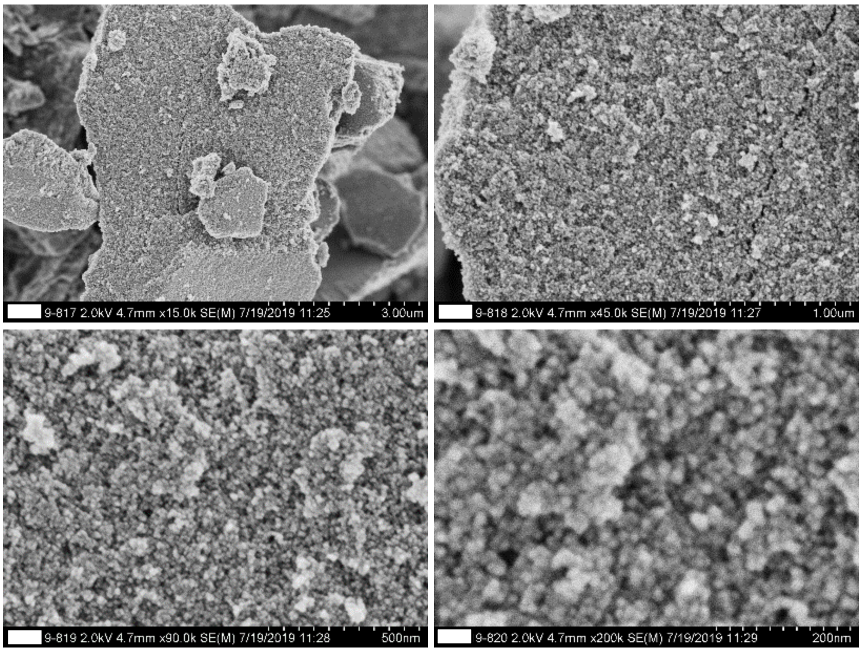

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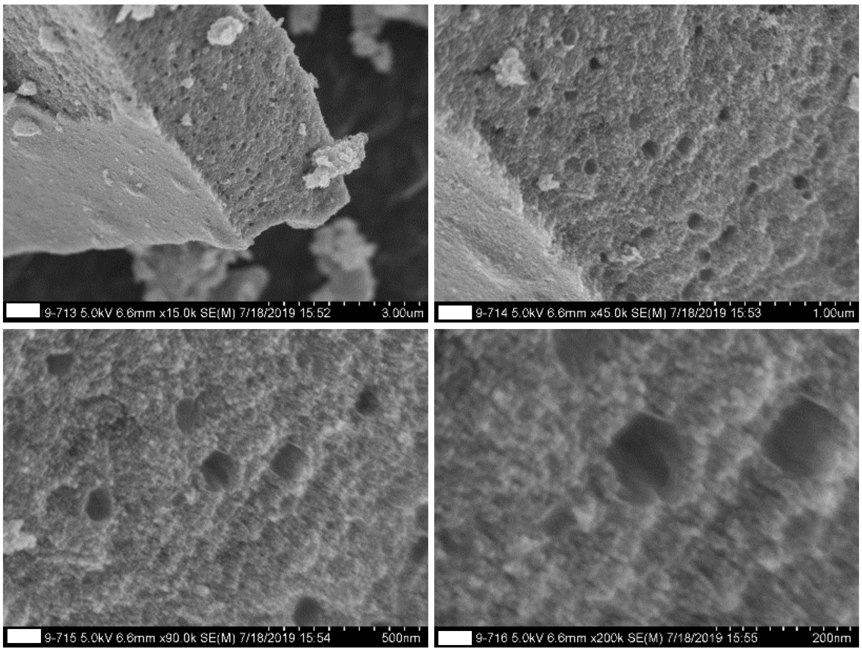

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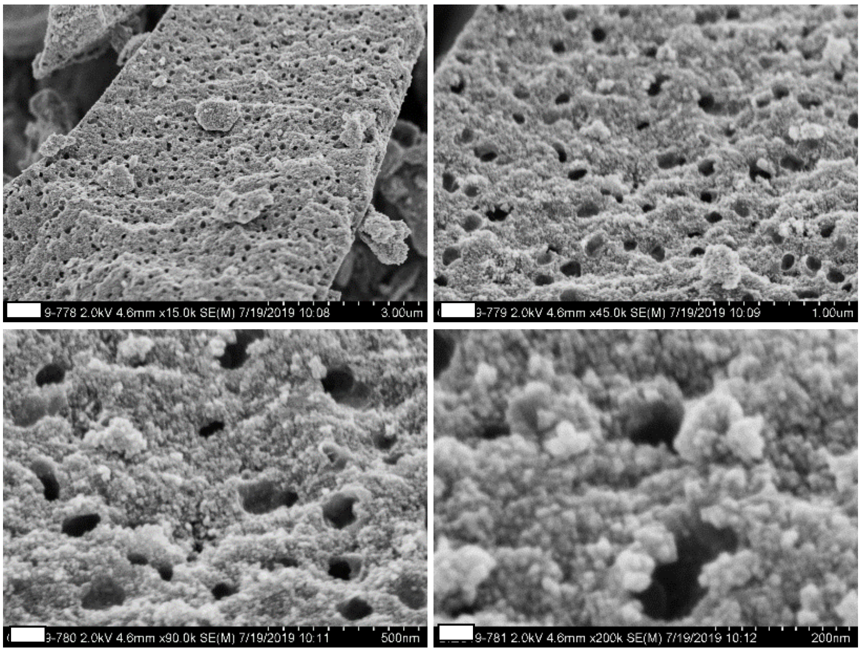
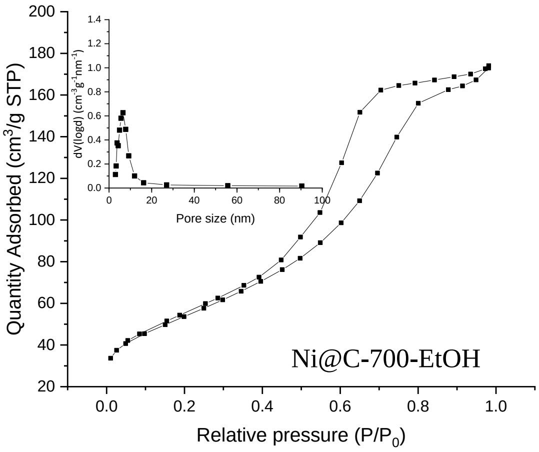
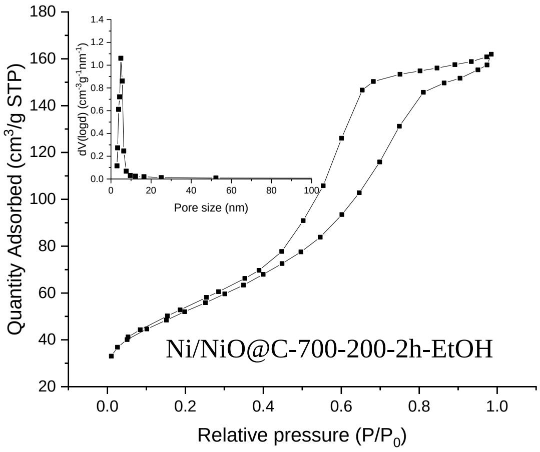
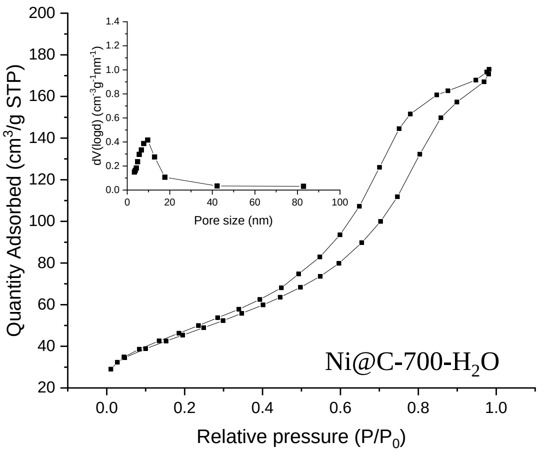


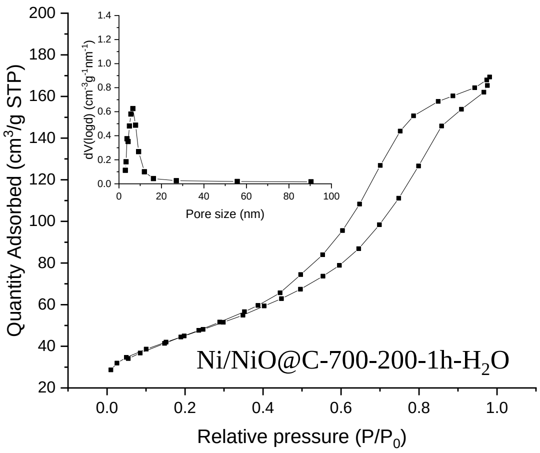
Ni/NiO@C-700-200-2h-EtOH

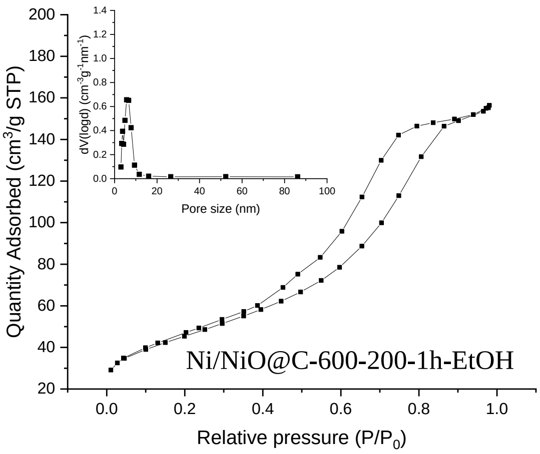
S5. BET measurements







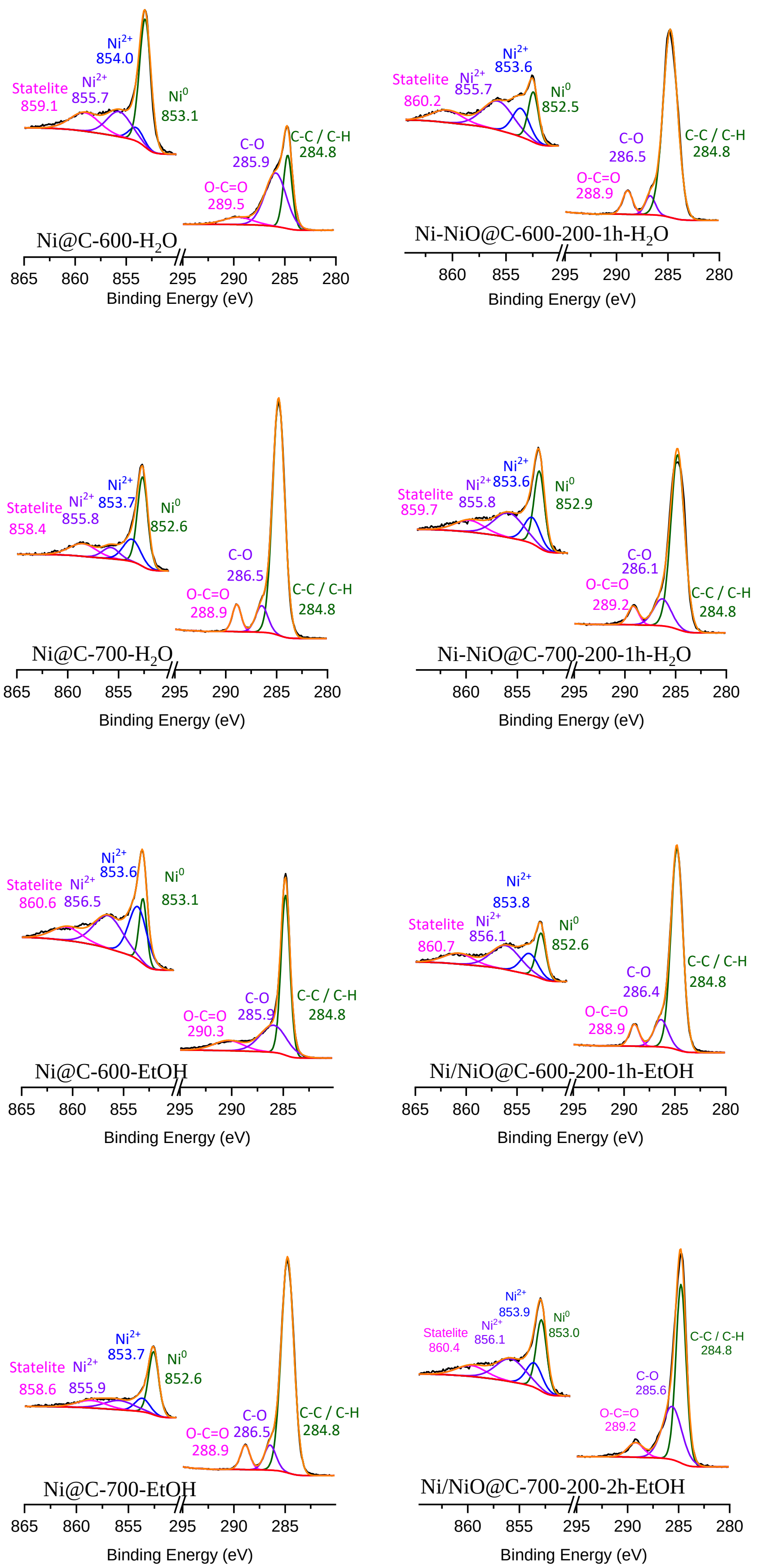




BET table of selective catalysts

Catalyst	Pore size (nm)	Surface area (m^2/g)	Total pore volume ($cm^{-3}g$) micro- and meso-
Ni@C-700-H_2O	6.543	161.937	0.268
Ni@C-700-EtOH	5.609	182.051	0.270
Ni/NiO@C-700-200-1h-H_2O	5.625	160.087	0.262
Ni/NiO@C-600-200-1h-EtOH	5.678	160.172	0.242
Ni/NiO@C-700-200-2h-EtOH	5.131	223.912	0.287

S6. XPS spectra



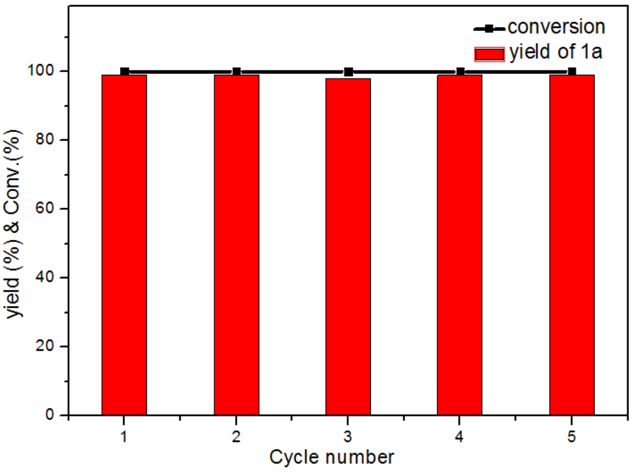
XPS peak assignment

Catalyst	C1s			$Ni2p_{3/2}$			
	C-C/C-H	C-O	O-C=O	Ni^0	Ni^{2+}	Ni^{2+}	Statelite
Ni@C-600-H_2O	284.8	285.9	289.5	853.1	854.0	855.7	859.1
Ni/NiO@C-600-200-1h-H_2O	284.8	286.5	288.9	852.5	853.6	855.7	860.2
Ni@C-700-H_2O	284.8	286.5	288.9	852.6	853.7	855.8	858.4
Ni/NiO@C-700-200-1h-H_2O	284.8	286.1	289.2	852.9	853.6	855.8	859.7
Ni@C-600-EtOH	284.8	285.9	290.3	853.1	853.6	856.5	860.6
Ni/NiO@C-600-200-1h-EtOH	284.8	286.4	288.9	852.6	853.8	856.1	860.7
Ni@C-700-EtOH	284.8	286.5	288.9	852.6	853.7	855.9	858.6
Ni/NiO@C-700-200-2h-EtOH	284.8	286.5	289.2	853.0	853.9	856.1	860.4

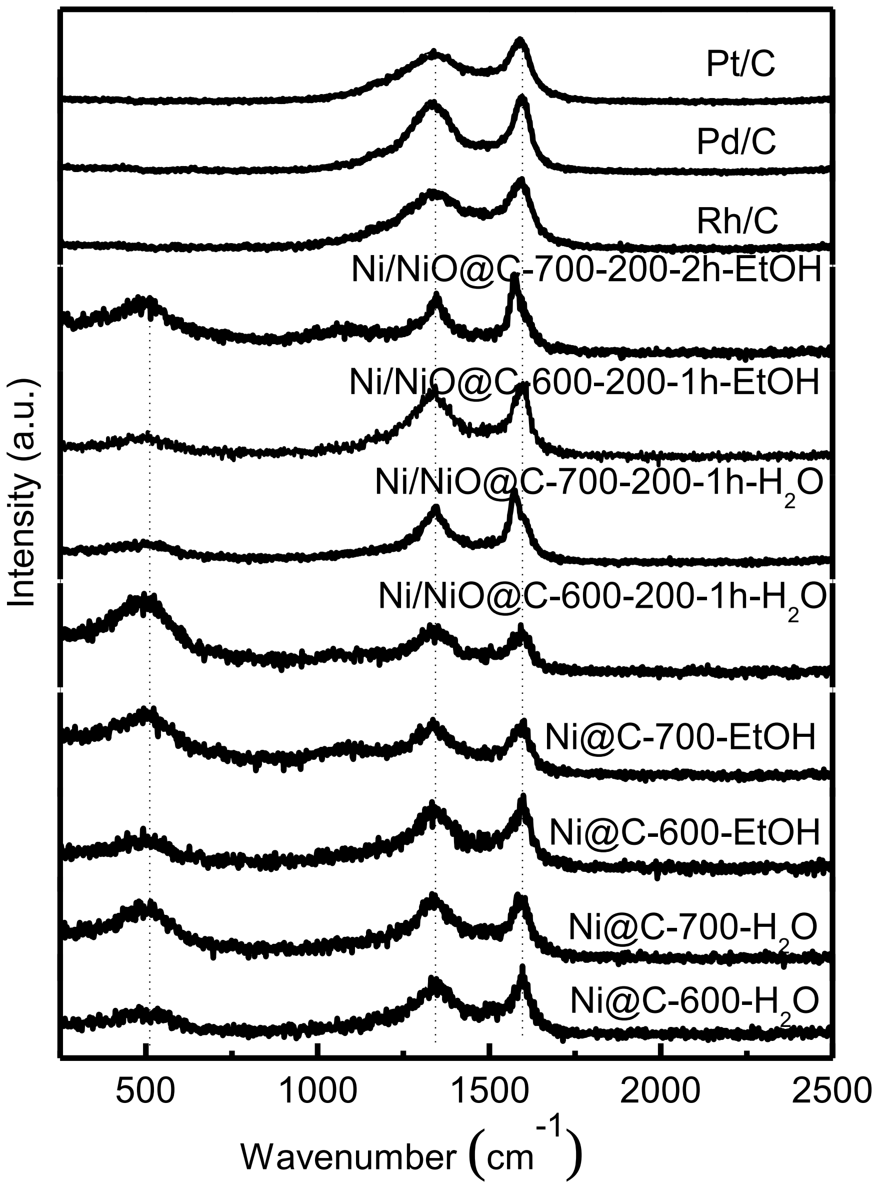
S7. EDS element

Number	Catalyst	Ni(%)	C(%)	O(%)	S(%)
A	Ni@C-600-H_2O	37.83	58.39	3.78	0
B	Ni@C-700-H_2O	33.61	60.98	5.41	0
C	Ni@C-600-EtOH	29.06	68.12	2.82	0
D	Ni@C-700-EtOH	25.35	68.6	5.32	0.73
E	Ni/NiO@C-600-200-1h-H_2O	30.05	60.7	9.24	0
F	Ni/NiO@C-700-200-1h-H_2O	22.9	72.54	4.56	0
G	Ni/NiO@C-600-200-1h-EtOH	26.91	59.24	13.84	0
H	Ni/NiO@C-700-200-2h-EtOH	20.78	64.7	14.02	0.5

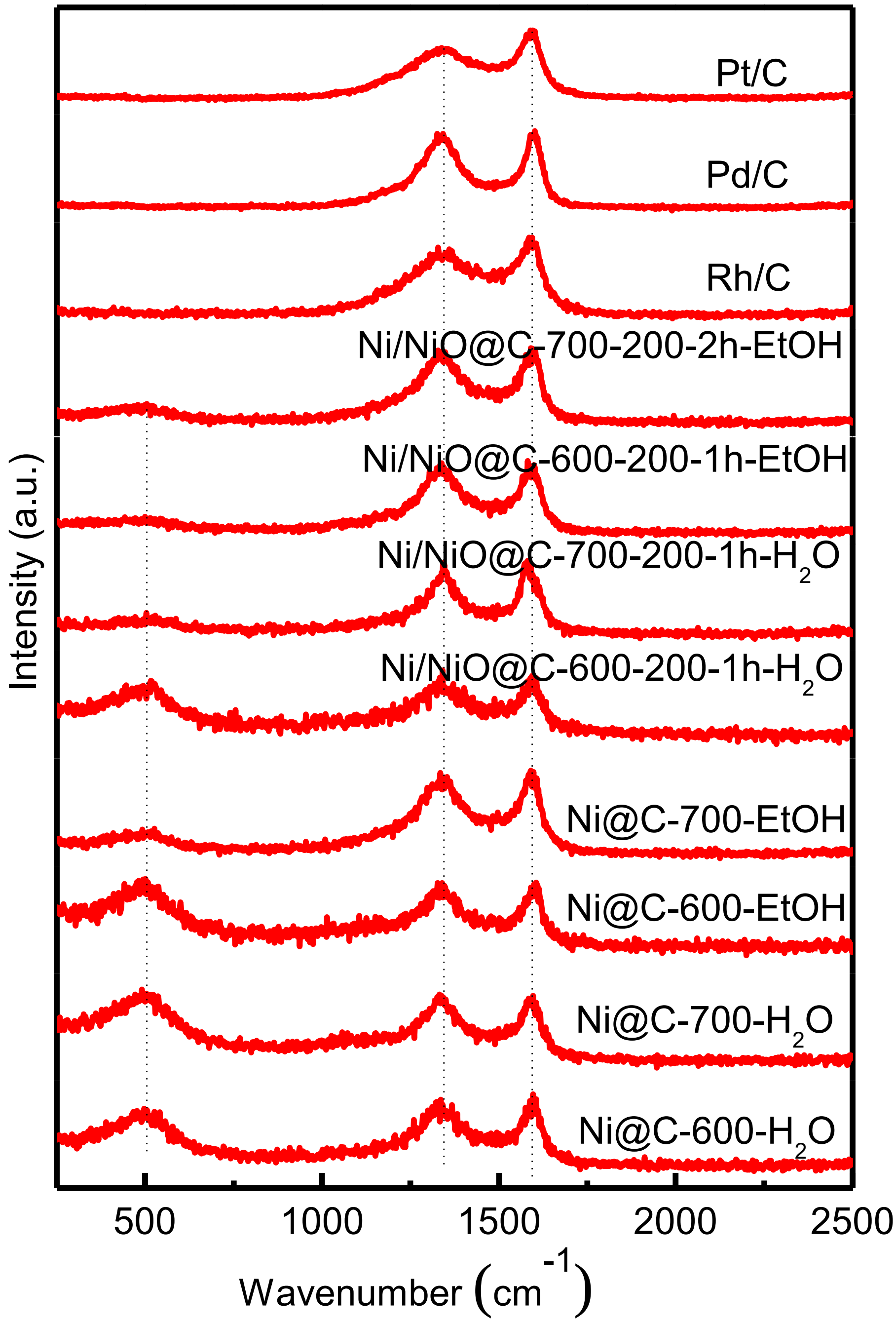
S8. Catalyst recovery using magnetic stir bar and catalytic activity recycle test



S9. Raman spectra of all freshed catalysts used in Table 1 in the manuscript

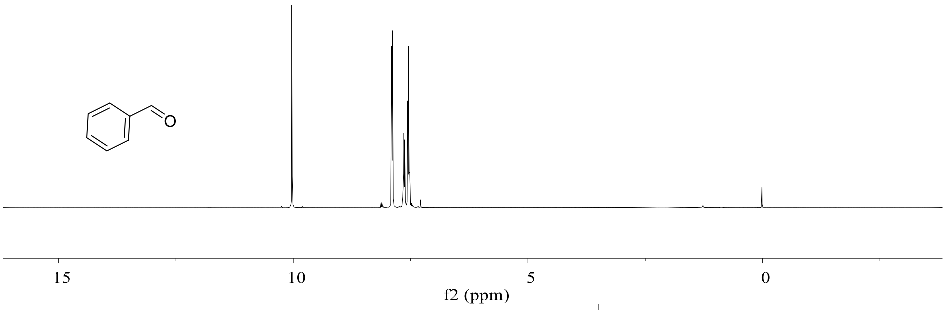


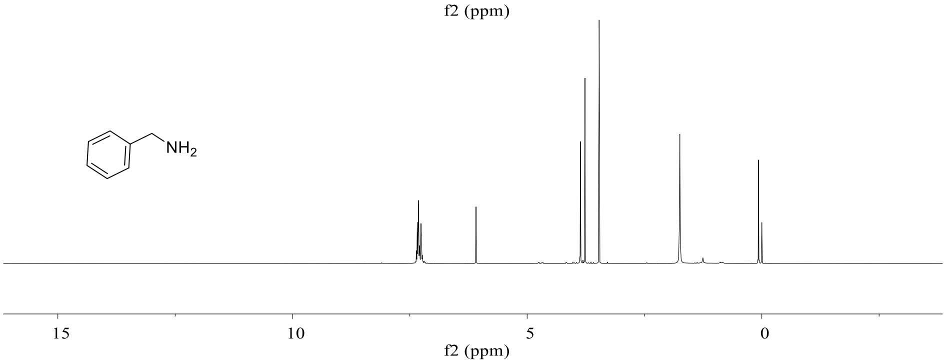
S9. Raman spectra of all catalysts after the reaction used in Table 1 in the manuscript



S10. NMR and GCMS spectra

A136





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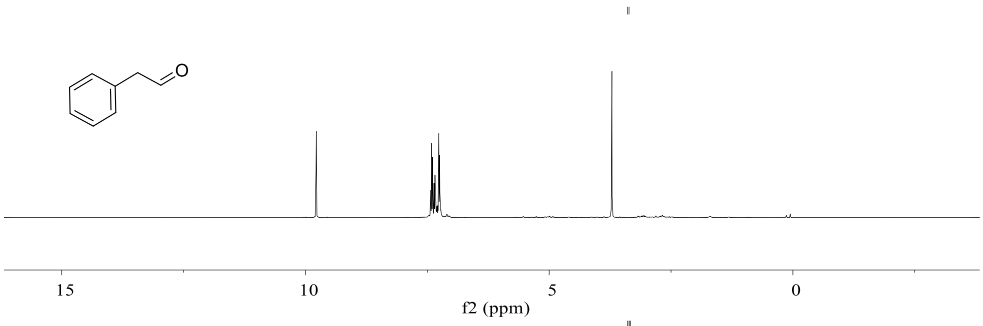

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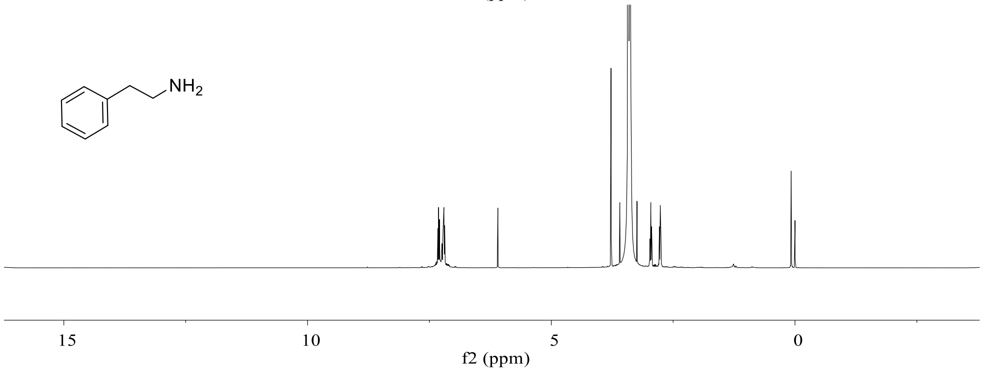

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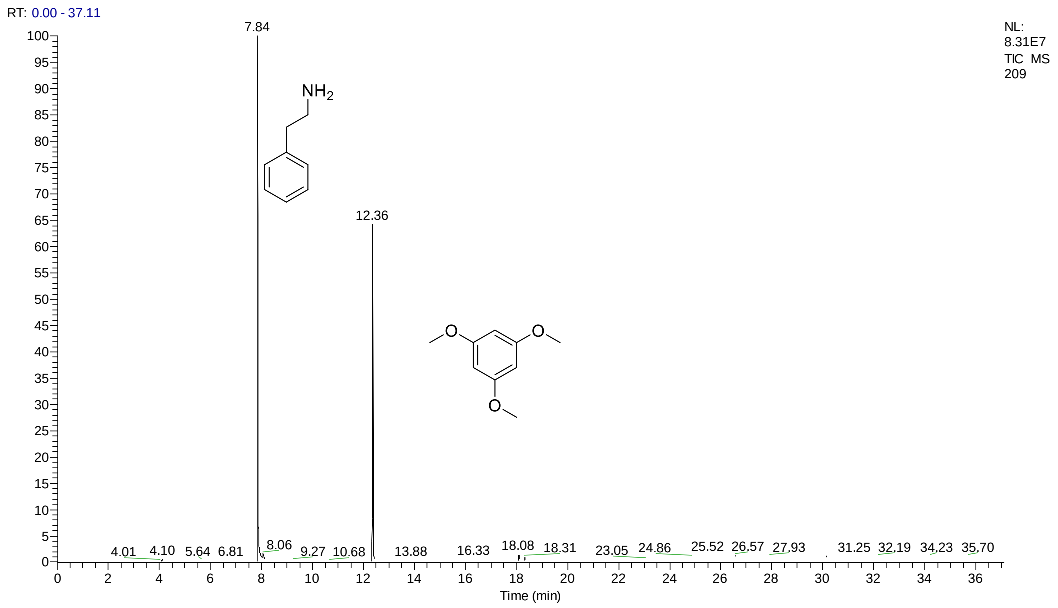

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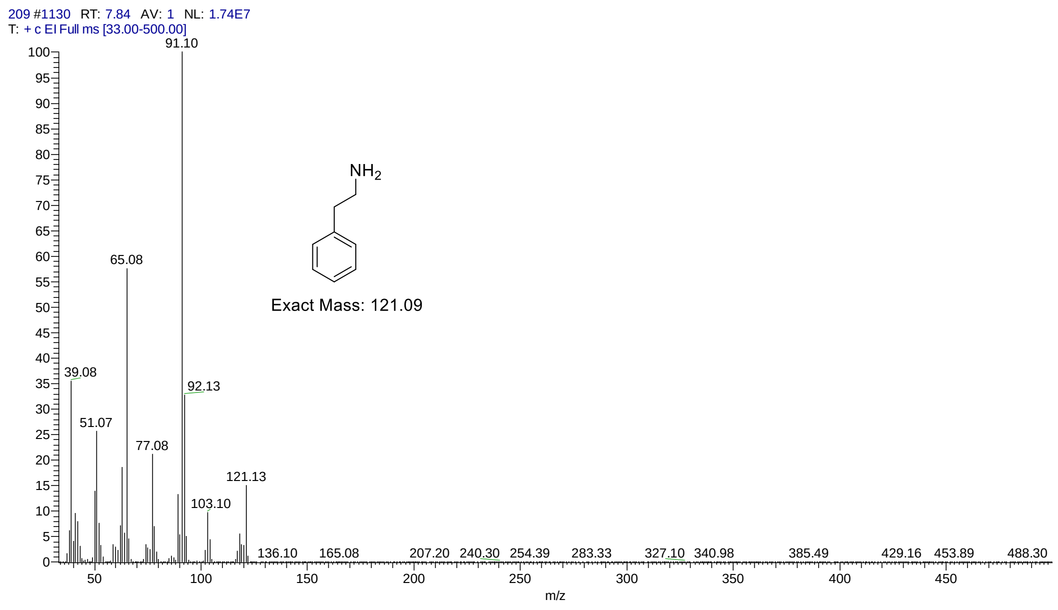

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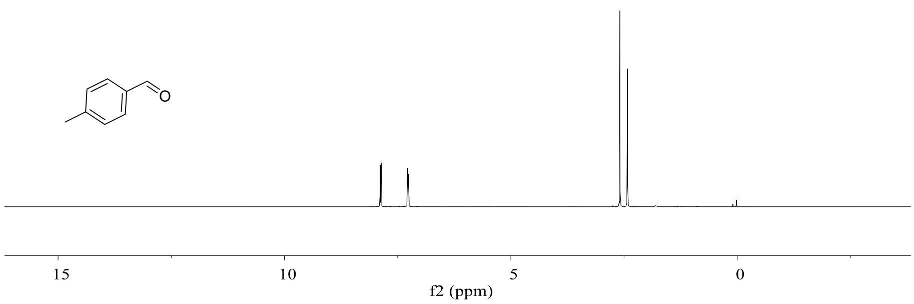
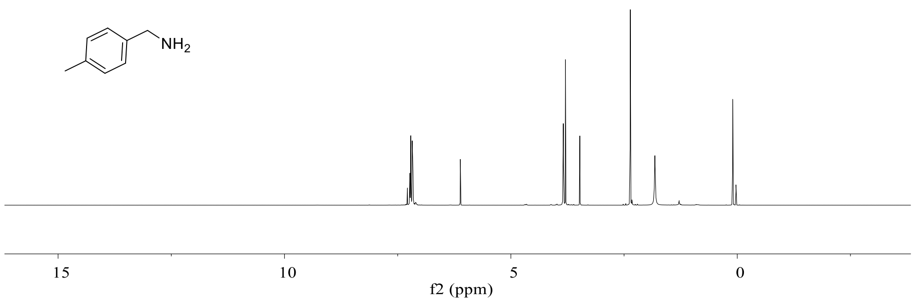
m/z

A170





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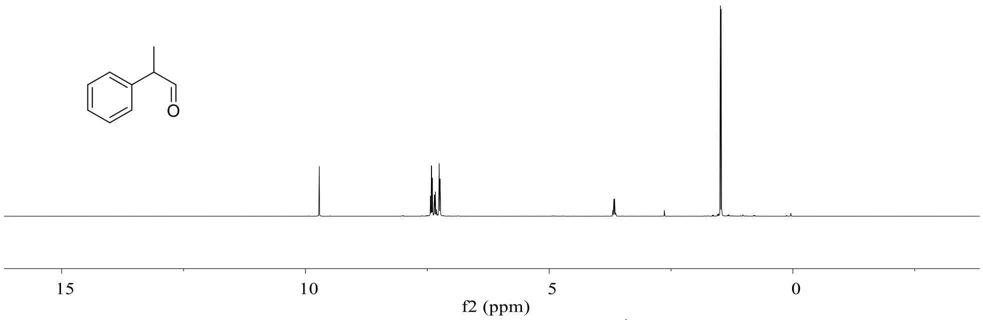

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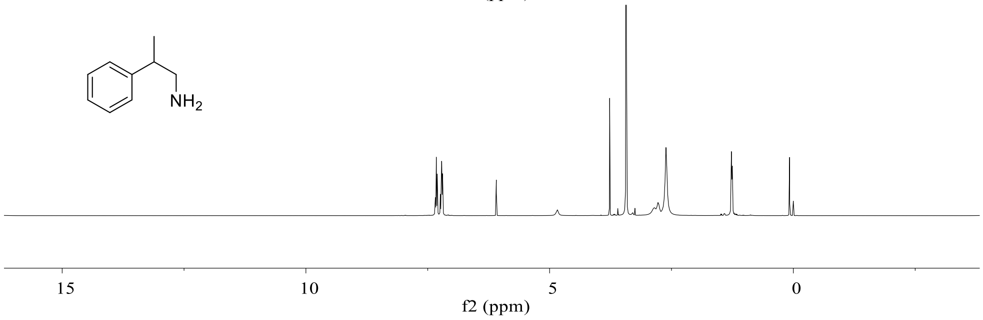

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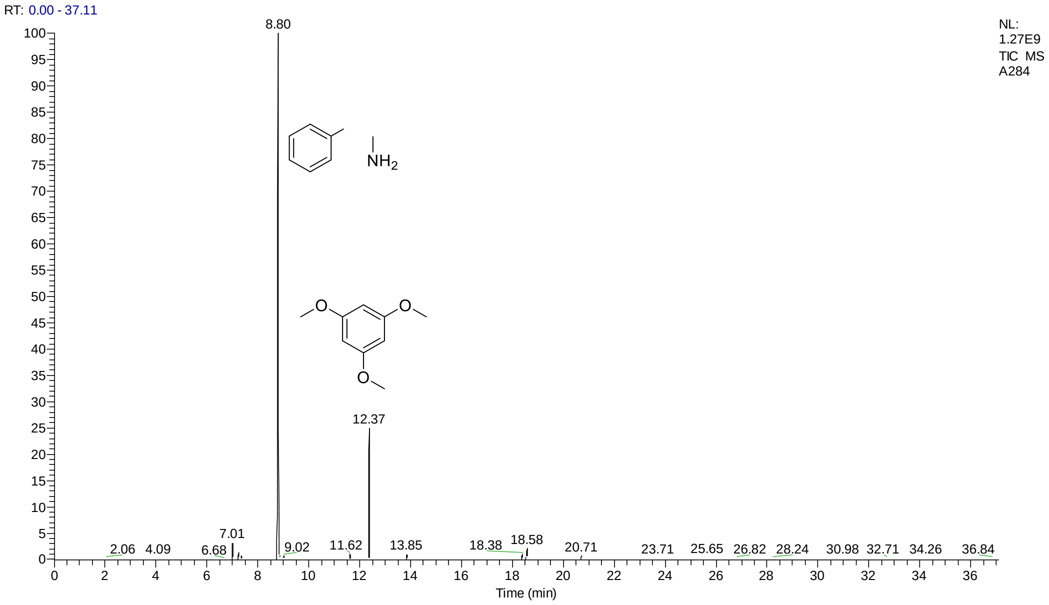

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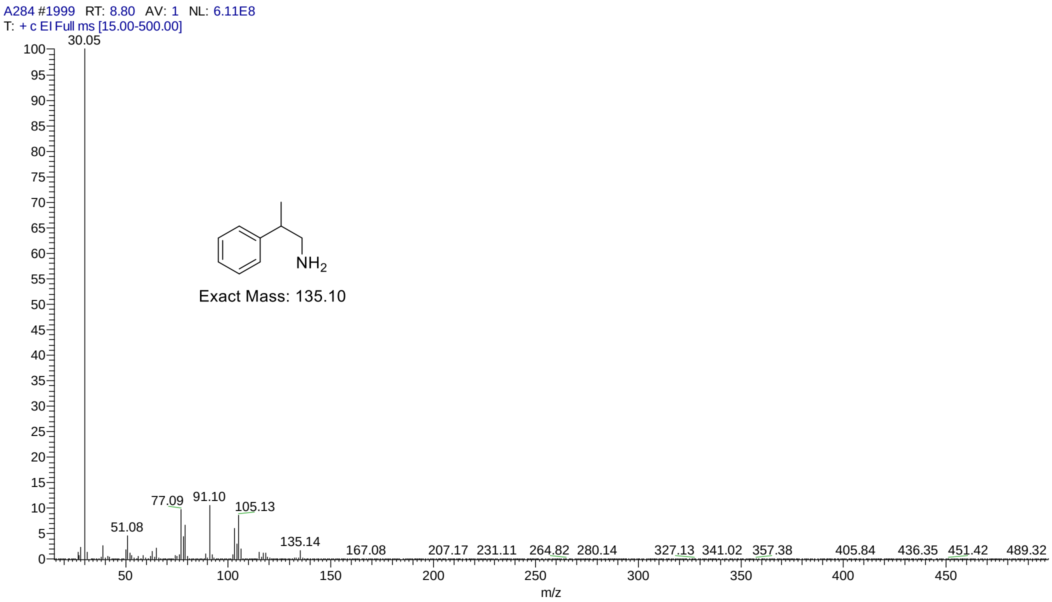

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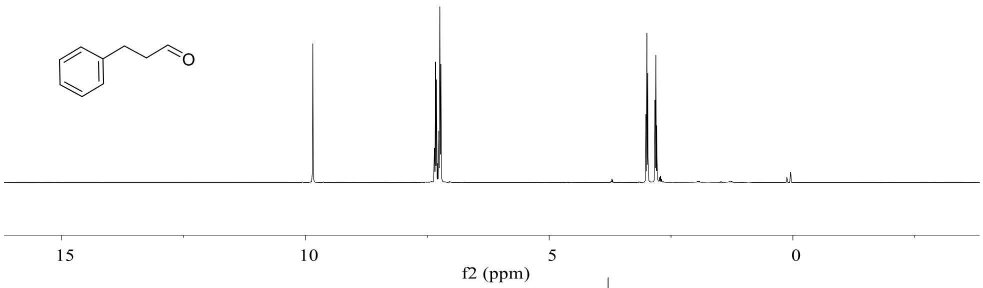
m/z

A216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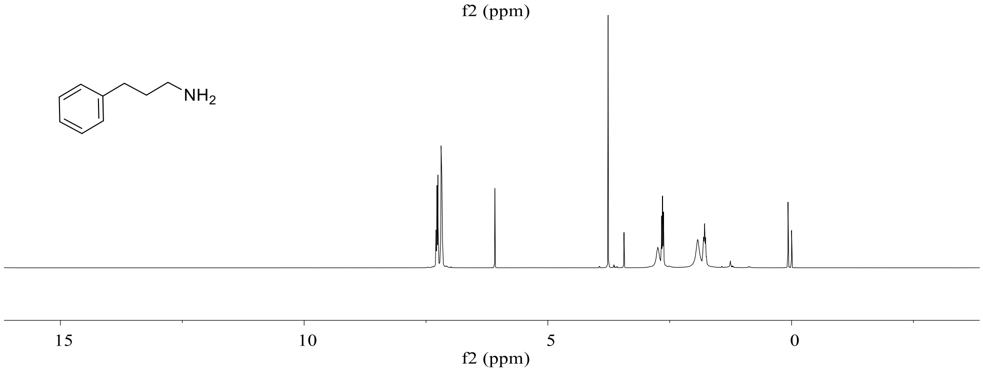
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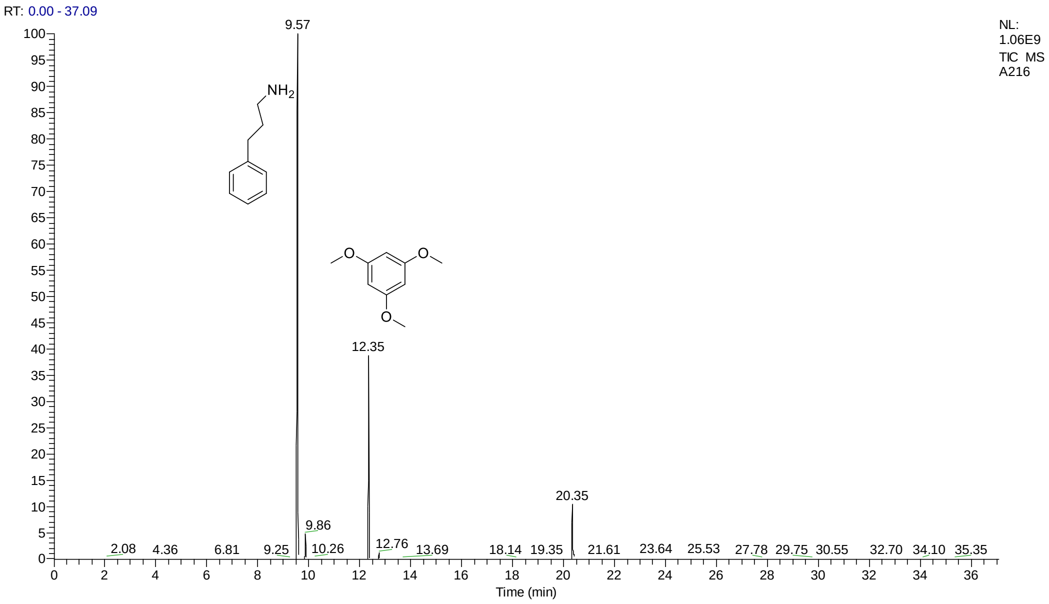
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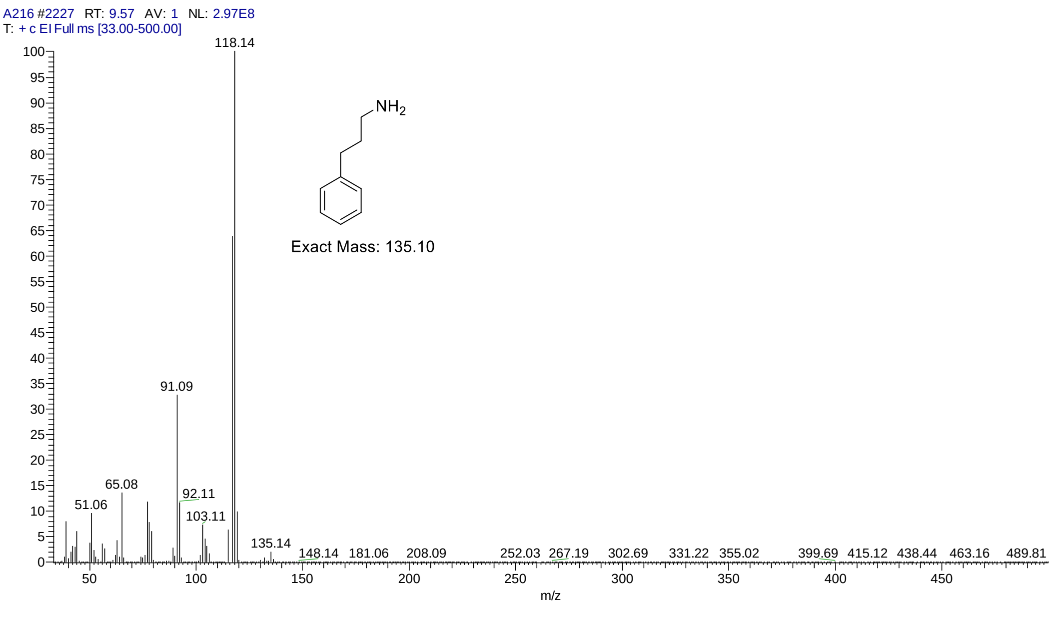
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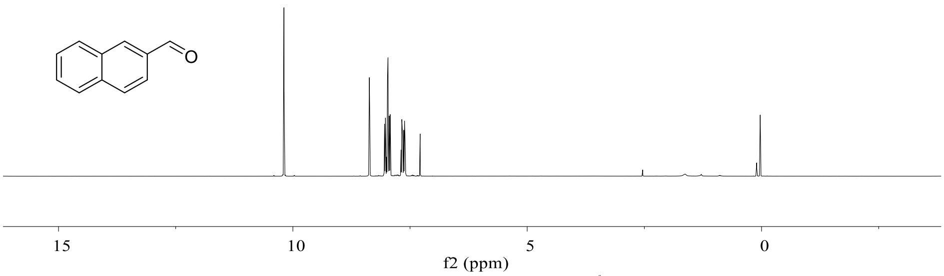
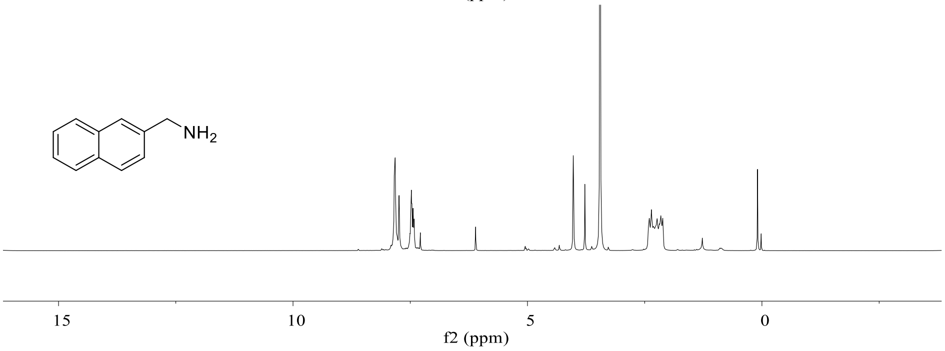
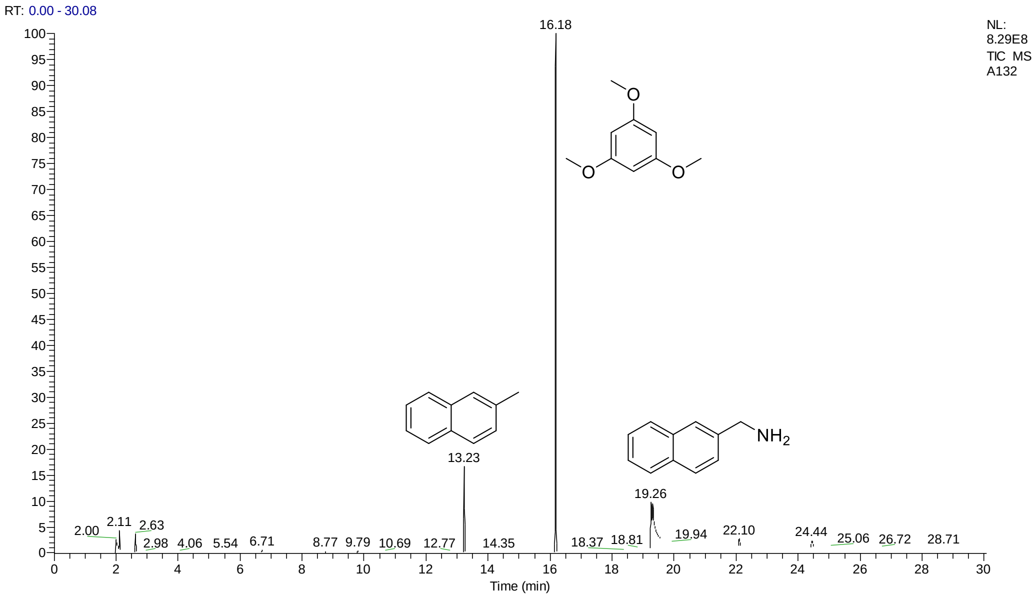
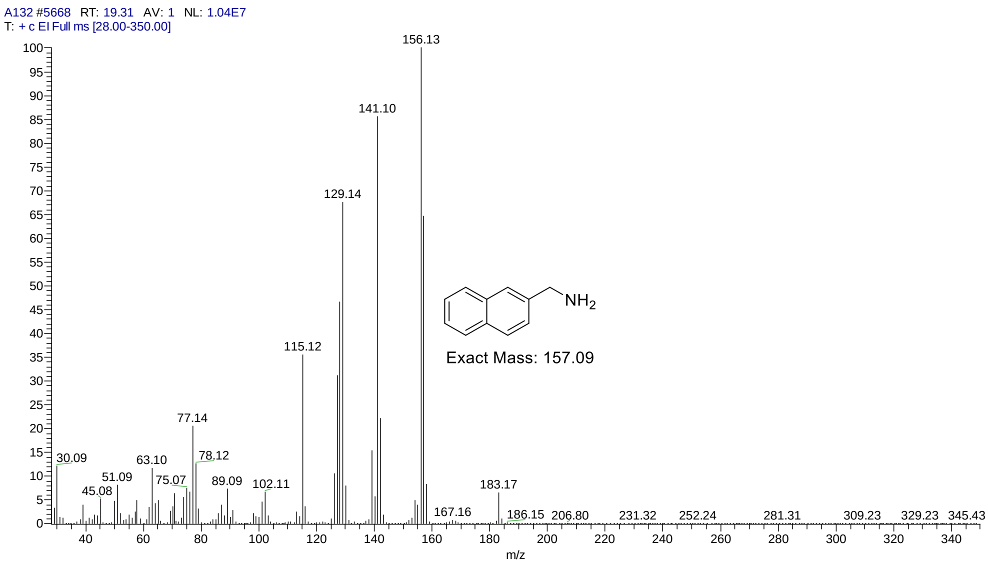
A132



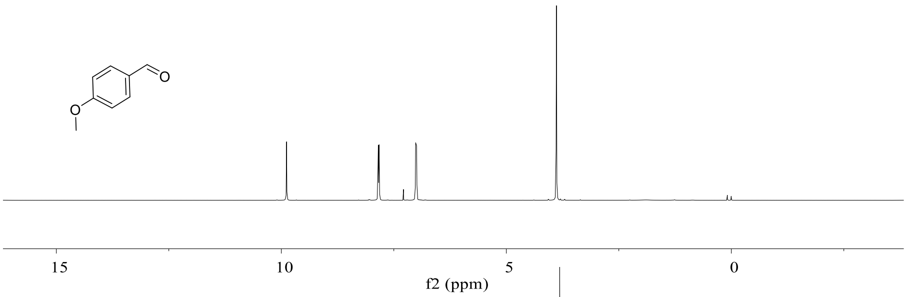


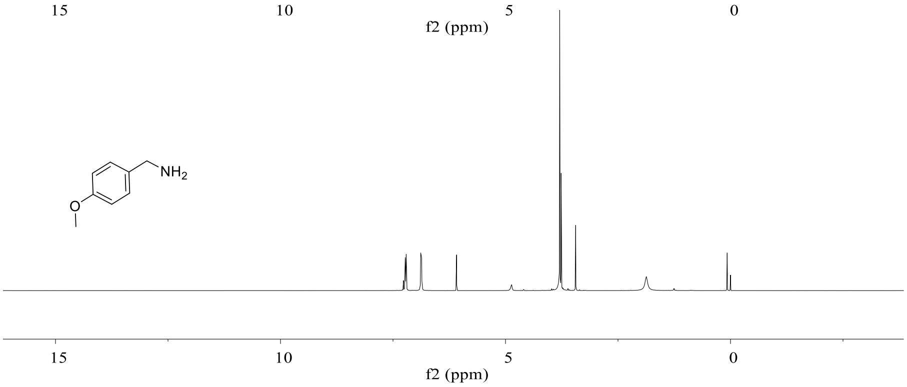


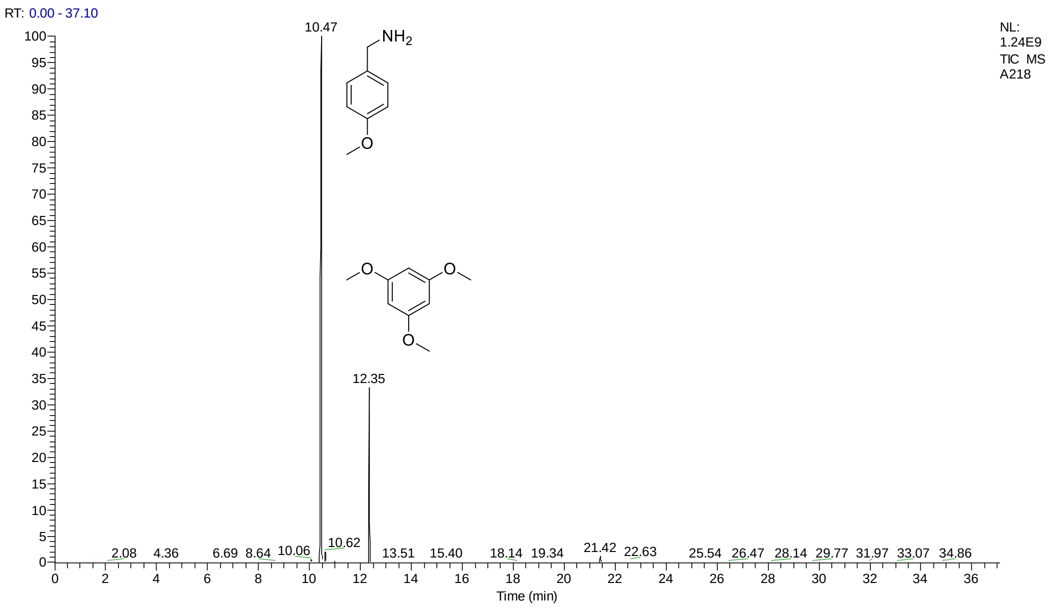
(Innovax column)

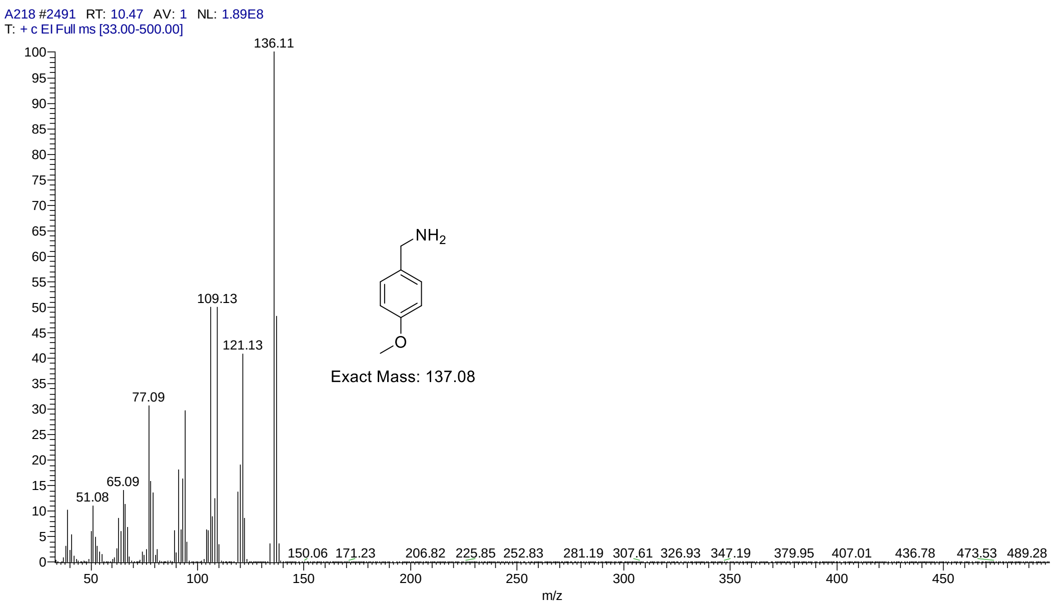


A218

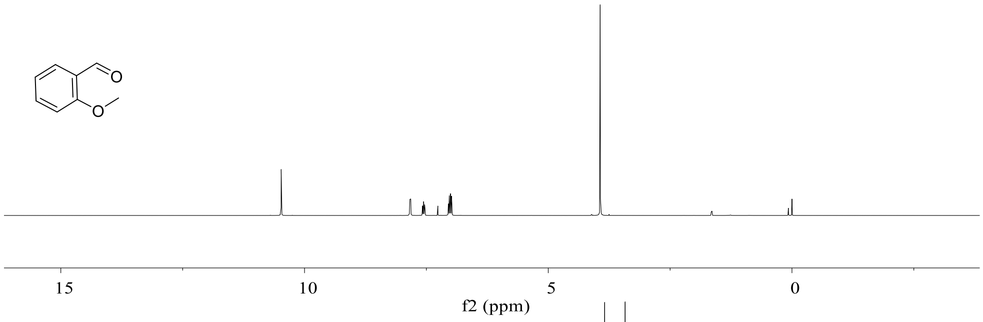

O
O
15
10
5
0
f2 (ppm)


NH2
O
15
10
5
0
f2 (ppm)

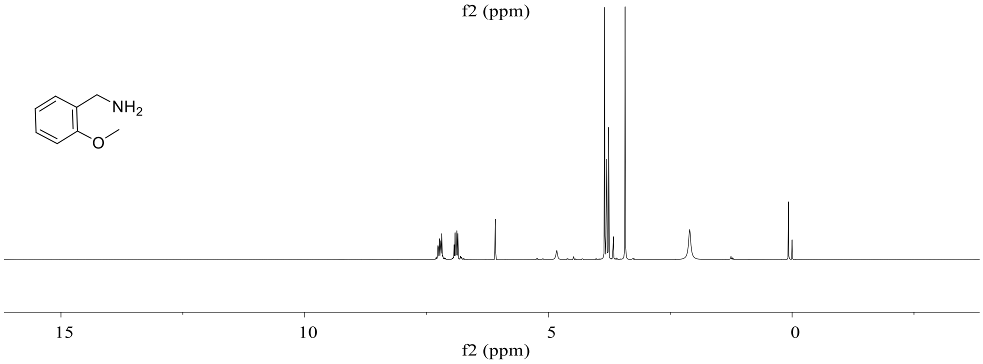

RT: 0.00 - 37.10
NL:
1.24E9
TIC MS
A218
10.47
12.35
2.08
4.36
6.69
8.64
10.06
10.62
13.51
15.40
18.14
19.34
21.42
22.63
25.54
26.47
28.14
29.77
31.97
33.07
34.86
NH2
O
Time (min)


A218 #2491 RT: 10.47 AV: 1 NL: 1.89E8
T: + c EI Full ms [33.00-500.00]
136.11
109.13
121.13
77.09
65.09
51.08
150.06
171.23
206.82
225.85
252.83
281.19
307.61
326.93
347.19
379.95
407.01
436.78
473.53
489.28
NH2
O
Exact Mass: 137.08
m/z

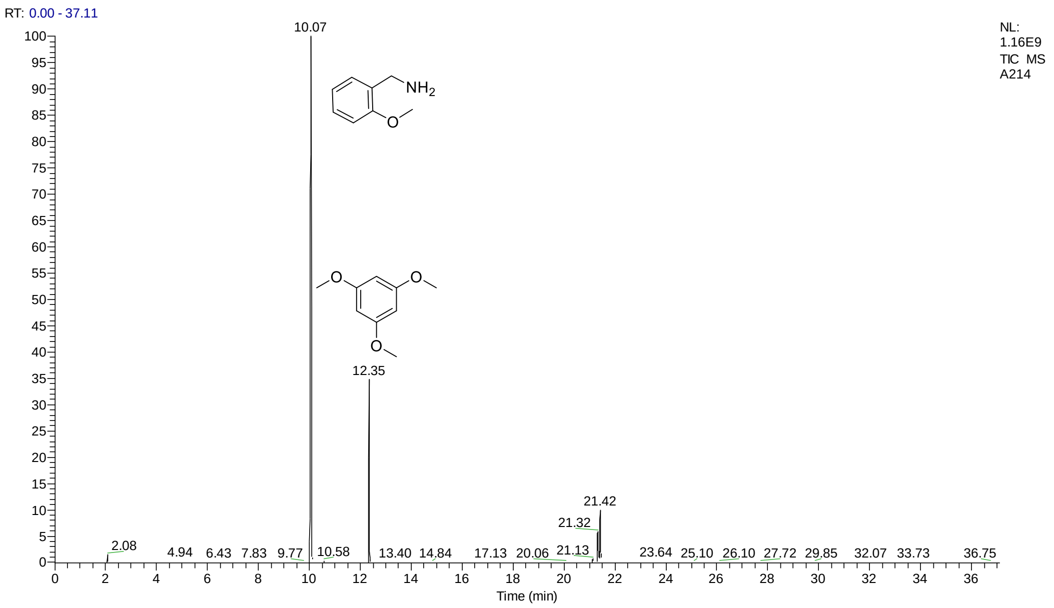
A214



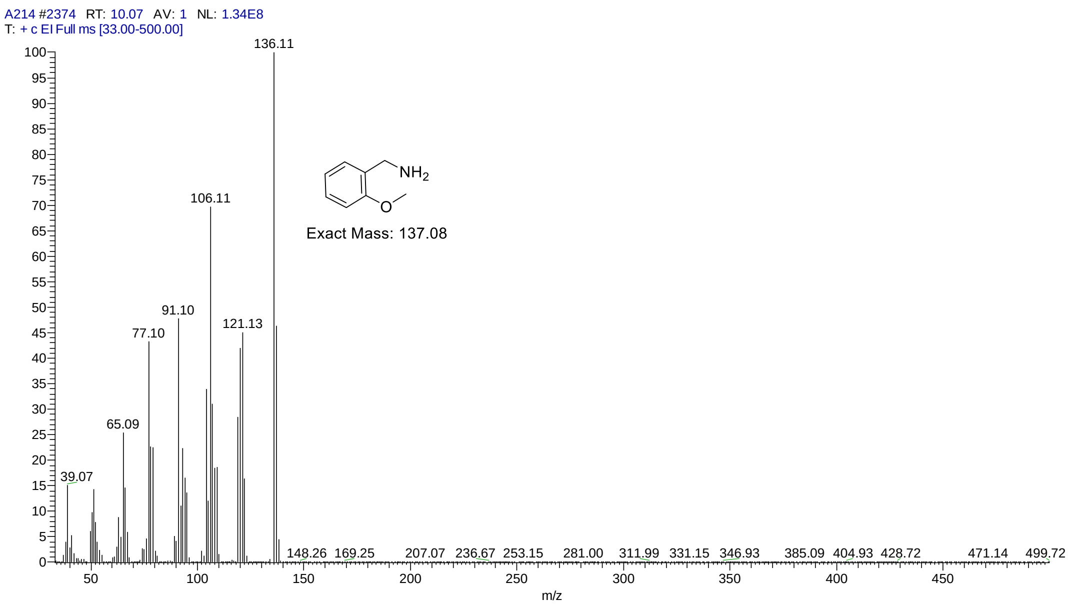
O
15
10
5
0
f2 (ppm)



NH2
O
15
10
5
0
f2 (ppm)

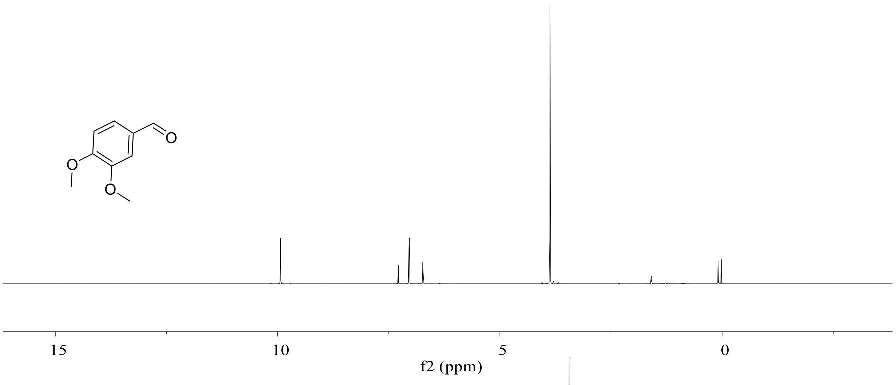


RT: 0.00 - 37.11
NL: 1.16E9 TIC MS A214
10.07
12.35
21.42
21.32
2.08
4.94
6.43
7.83
9.77
10.58
13.40
14.84
17.13
20.06
21.13
23.64
25.10
26.10
27.72
29.85
32.07
33.73
36.75
NH2
O
Time (min)

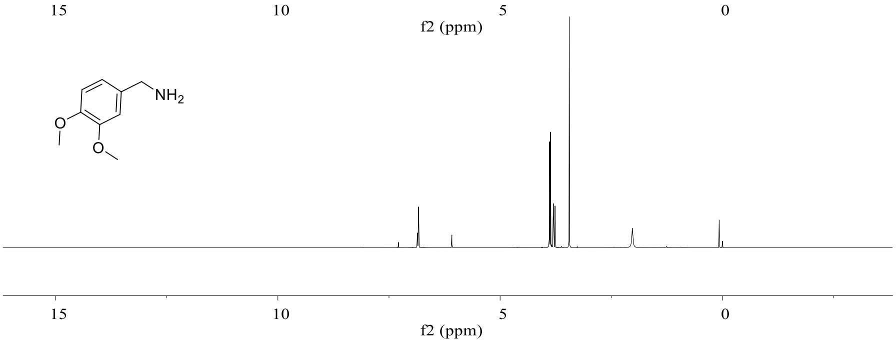


A214 #2374 RT: 10.07 AV: 1 NL: 1.34E8
T: + c EI Full ms [33.00-500.00]
136.11
106.11
91.10
77.10
121.13
65.09
39.07
148.26
169.25
207.07
236.67
253.15
281.00
311.99
331.15
346.93
385.09
404.93
428.72
471.14
499.72
NH2
O
Exact Mass: 137.08
m/z

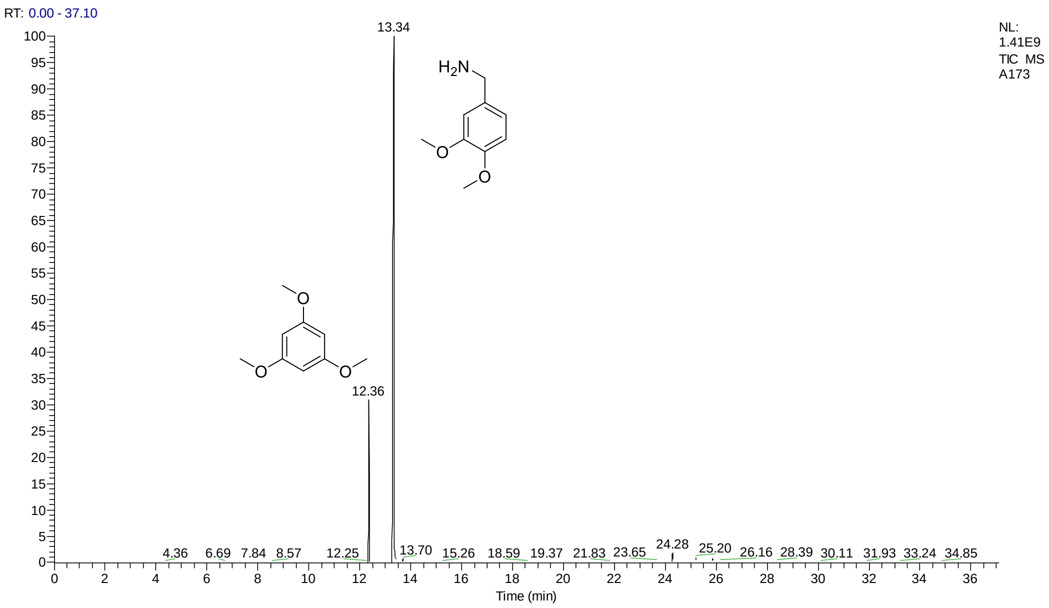
A173



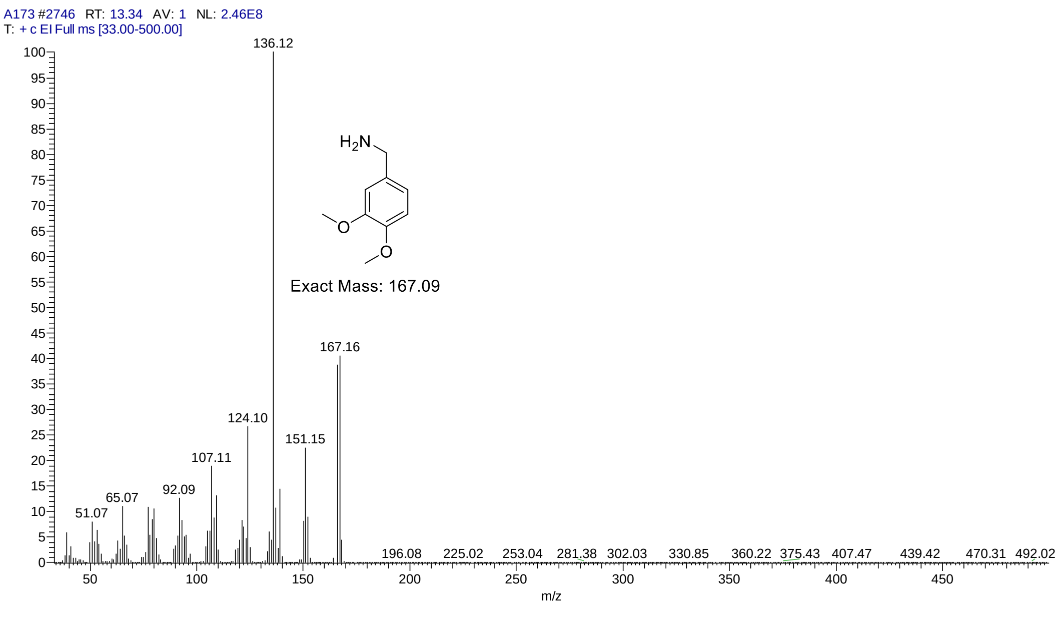
O
O
O
15
10
5
0
f2 (ppm)



NH_2
O
O
15
10
5
0
f2 (ppm)

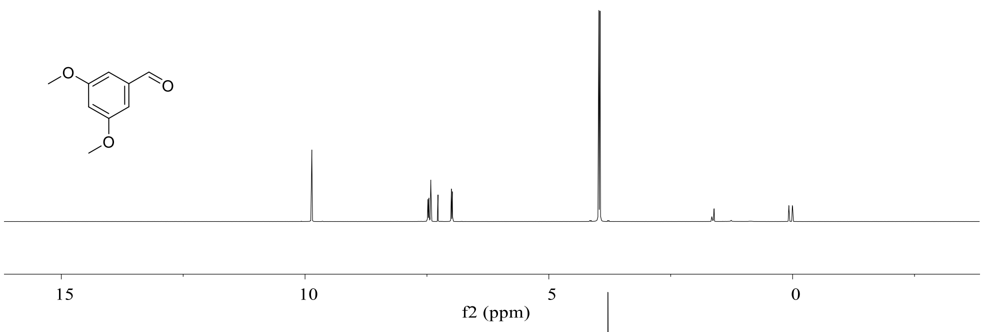


RT: 0.00 - 37.10
NL: 1.41E9
TIC MS
A173
13.34
12.36
4.36 6.69 7.84 8.57 12.25 13.70 15.26 18.59 19.37 21.83 23.65 24.28 25.20 26.16 28.39 30.11 31.93 33.24 34.85
H_2N
O
O
O
O
O
Time (min)

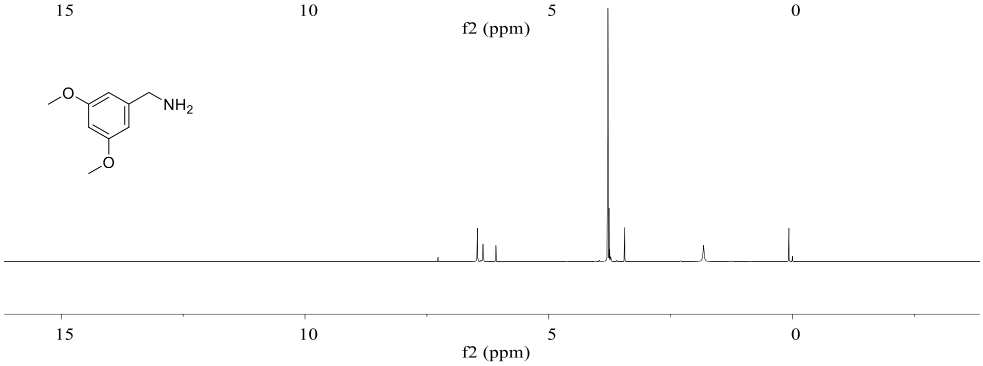


A173 #2746 RT: 13.34 AV: 1 NL: 2.46E8
T: + c EI Full ms [33.00-500.00]
136.12
167.16
124.10
151.15
107.11
92.09
65.07
51.07
196.08 225.02 253.04 281.38 302.03 330.85 360.22 375.43 407.47 439.42 470.31 492.02
H_2N
O
O
Exact Mass: 167.09
m/z

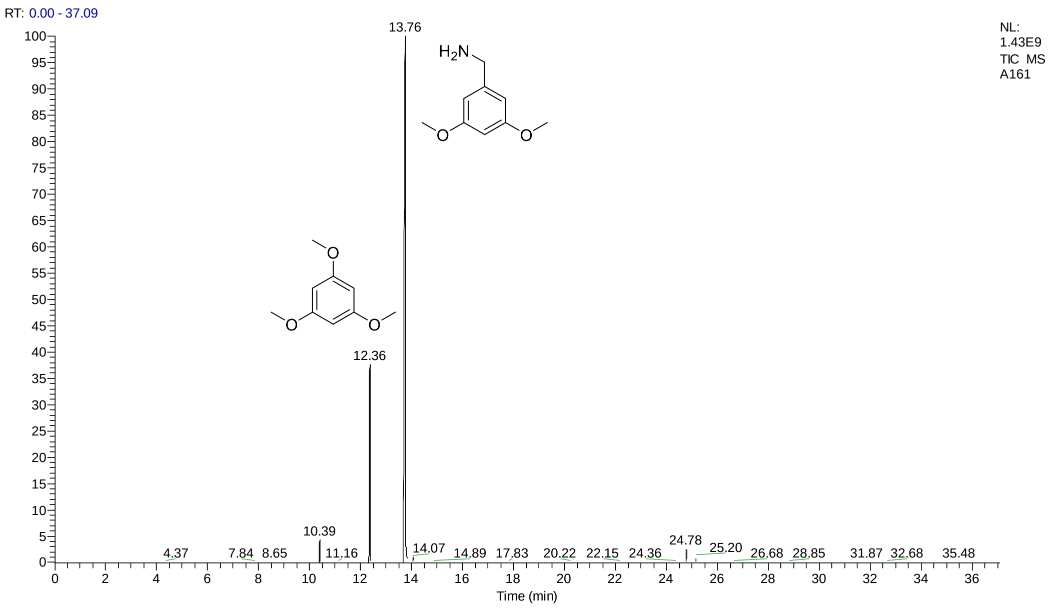
A161



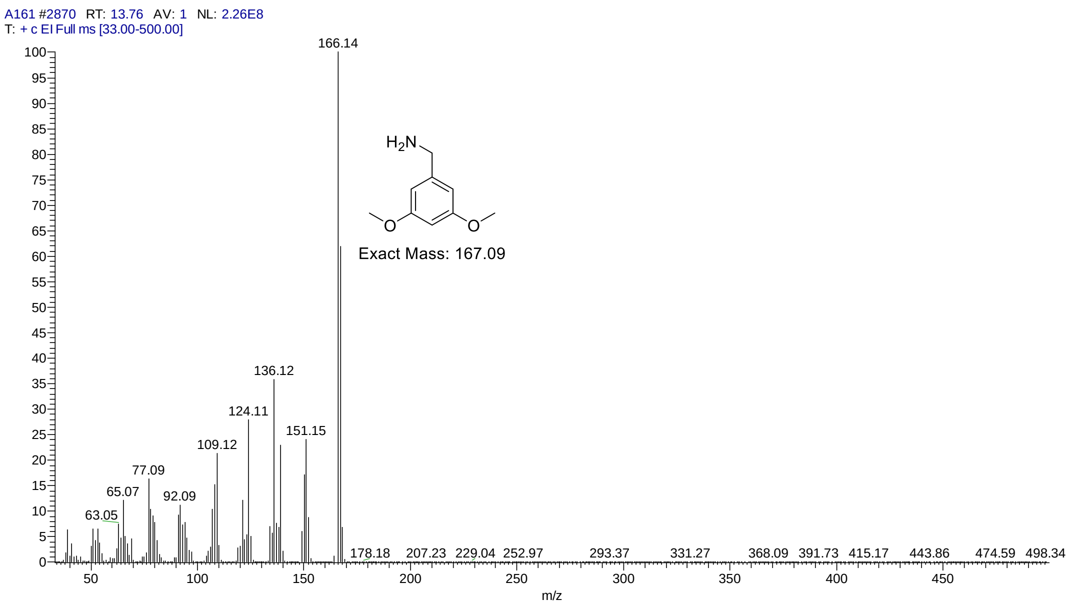
O
O
O
15
10
5
0
f2 (ppm)



O
O
NH2
15
10
5
0
f2 (ppm)

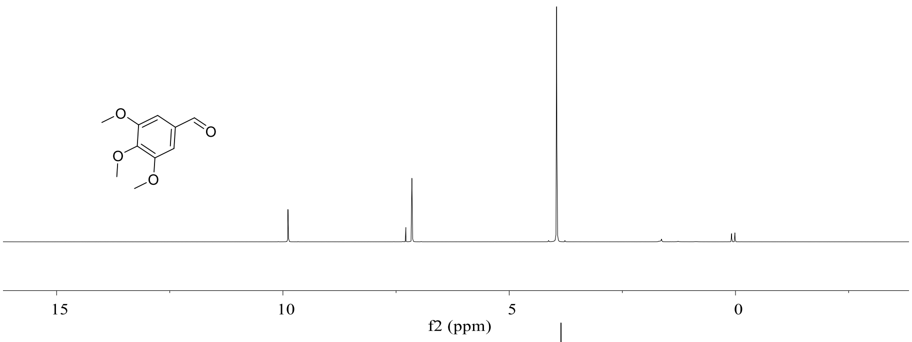


RT: 0.00 - 37.09
NL:
1.43E9
TIC MS
A161
13.76
12.36
10.39
4.37
7.84
8.65
11.16
14.07
14.89
17.83
20.22
22.15
24.36
24.78
25.20
26.68
28.85
31.87
32.68
35.48
H2N
O
O
Time (min)

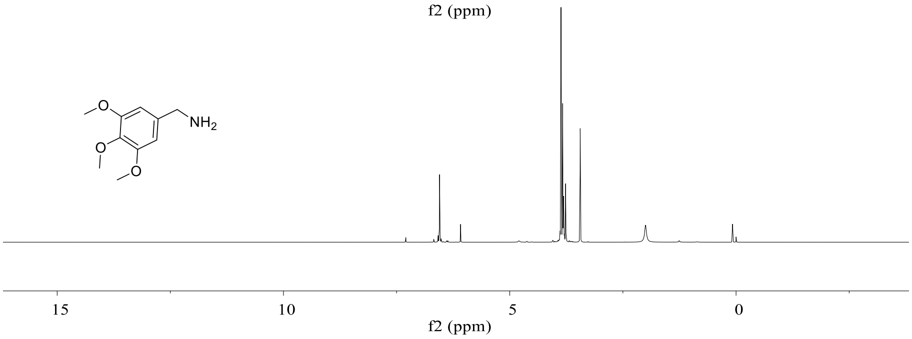


A161 #2870 RT: 13.76 AV: 1 NL: 2.26E8
T: + c EI Full ms [33.00-500.00]
166.14
136.12
124.11
151.15
109.12
77.09
65.07
92.09
63.05
178.18
207.23
229.04
252.97
293.37
331.27
368.09
391.73
415.17
443.86
474.59
498.34
H2N
O
O
Exact Mass: 167.09
m/z

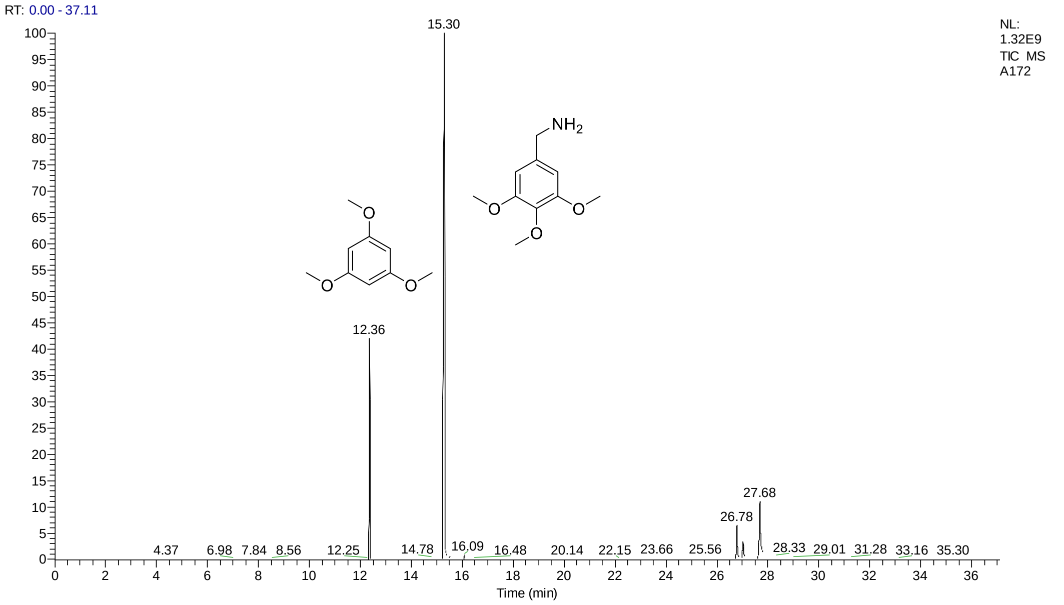
A172



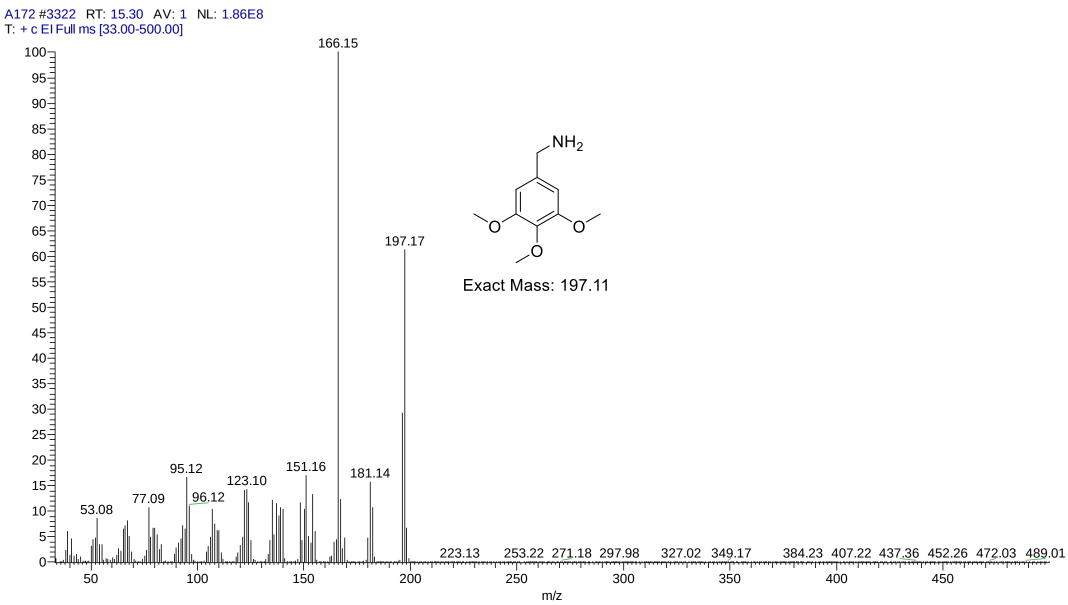
15
10
5
0
f2 (ppm)



NH2
15
10
5
0
f2 (ppm)

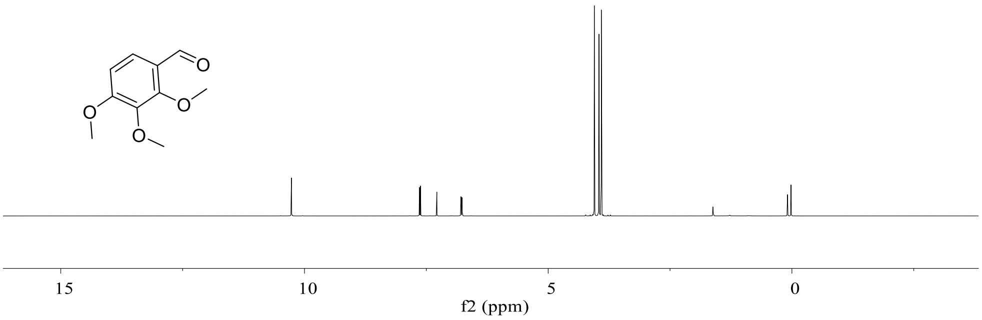


RT: 0.00 - 37.11
NL: 1.32E9 TIC MS A172
15.30
12.36
27.68
26.78
4.37 6.98 7.84 8.56 12.25 14.78 16.09 16.48 20.14 22.15 23.66 25.56 28.33 29.01 31.28 33.16 35.30
NH2
Time (min)

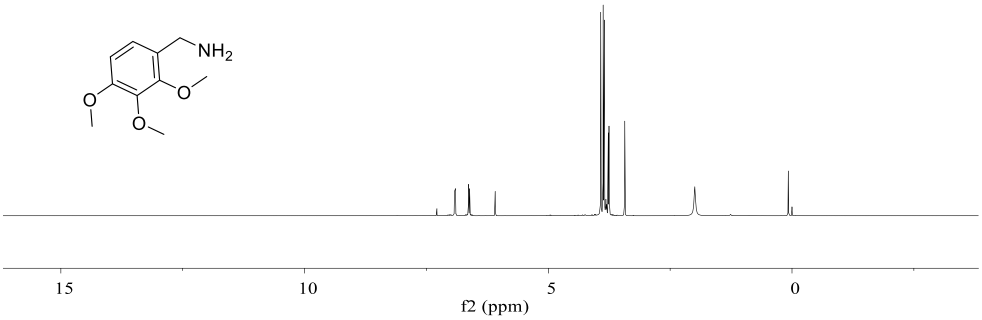


A172 #3322 RT: 15.30 AV: 1 NL: 1.86E8
T: + c EI Full ms [33.00-500.00]
166.15
197.17
181.14
151.16
123.10
95.12
96.12
77.09
53.08
223.13 253.22 271.18 297.98 327.02 349.17 384.23 407.22 437.36 452.26 472.03 489.01
NH2
Exact Mass: 197.11
m/z

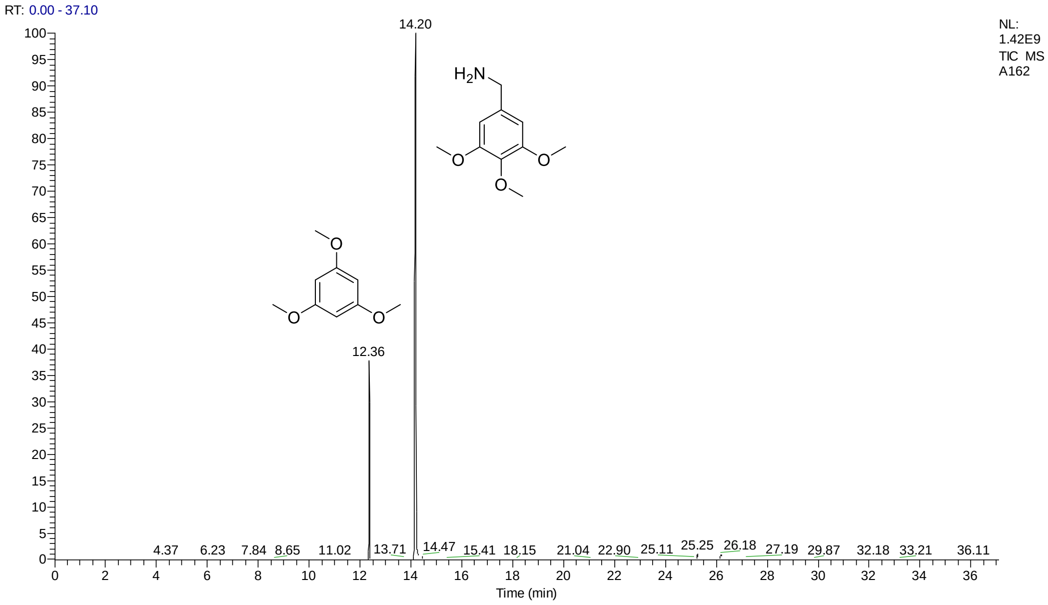
A162



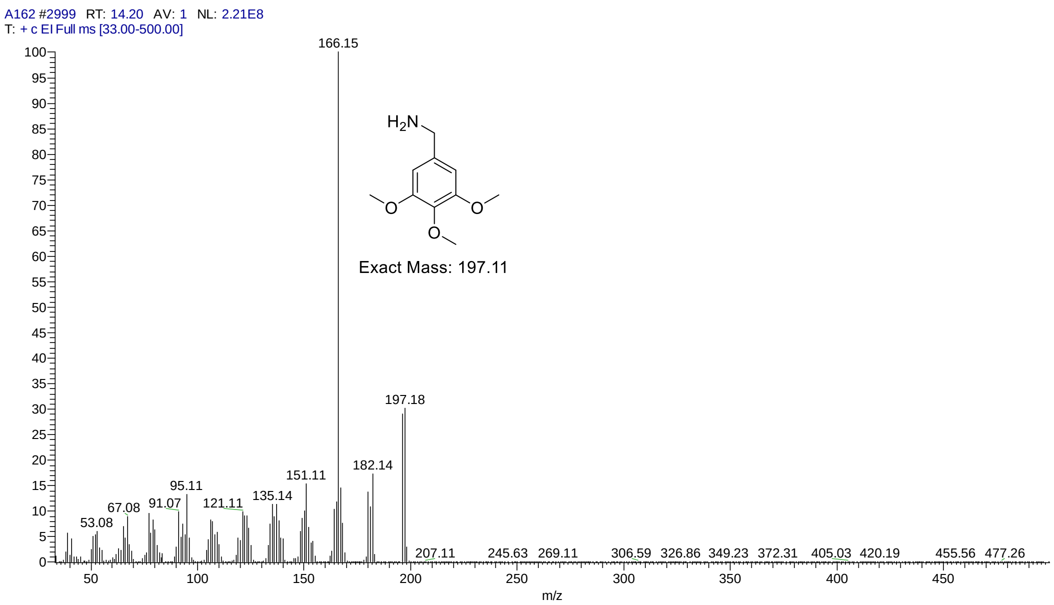
15
10
5
0
f2 (ppm)



NH2
15
10
5
0
f2 (ppm)

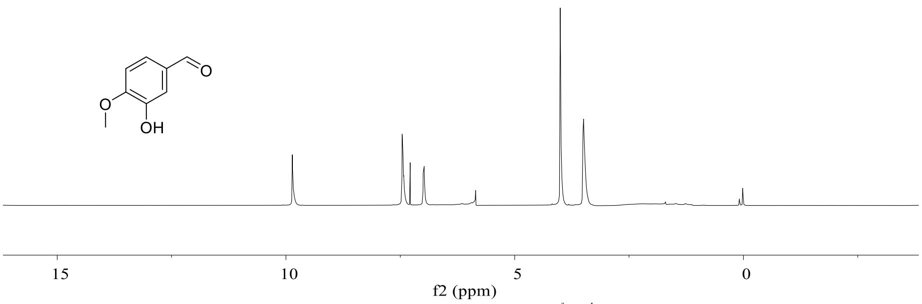


RT: 0.00 - 37.10
NL:
1.42E9
TIC MS
A162
14.20
12.36
H2N
4.37 6.23 7.84 8.65 11.02 13.71 14.47 15.41 18.15 21.04 22.90 25.11 25.25 26.18 27.19 29.87 32.18 33.21 36.11
Time (min)

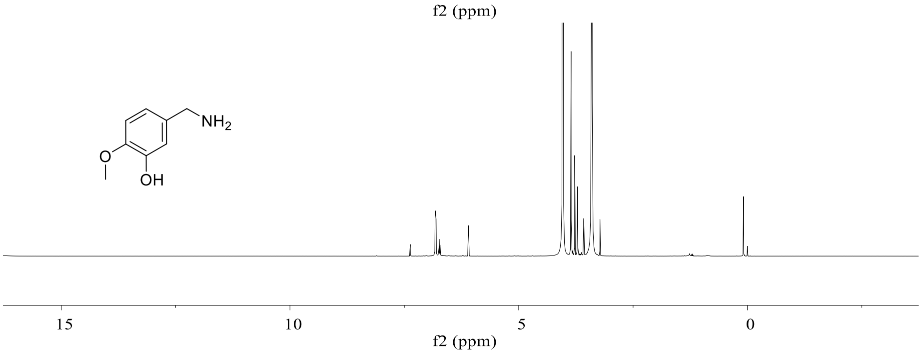


A162 #2999 RT: 14.20 AV: 1 NL: 2.21E8
T: + c EI Full ms [33.00-500.00]
166.15
H2N
Exact Mass: 197.11
197.18
182.14
151.11
135.14
121.11
95.11
91.07
67.08
53.08
207.11 245.63 269.11 306.59 326.86 349.23 372.31 405.03 420.19 455.56 477.26
m/z

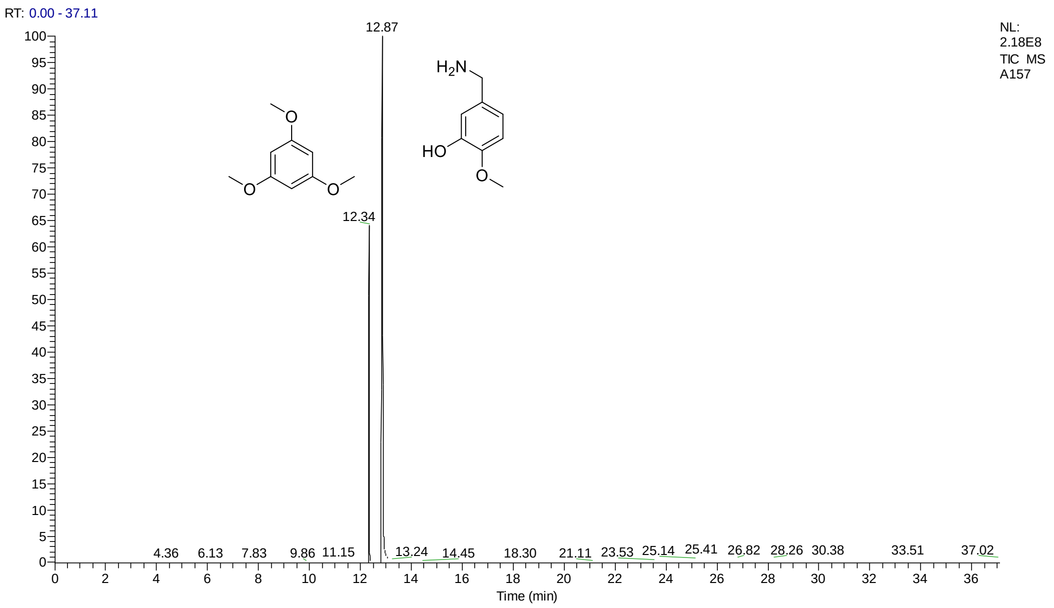
A157



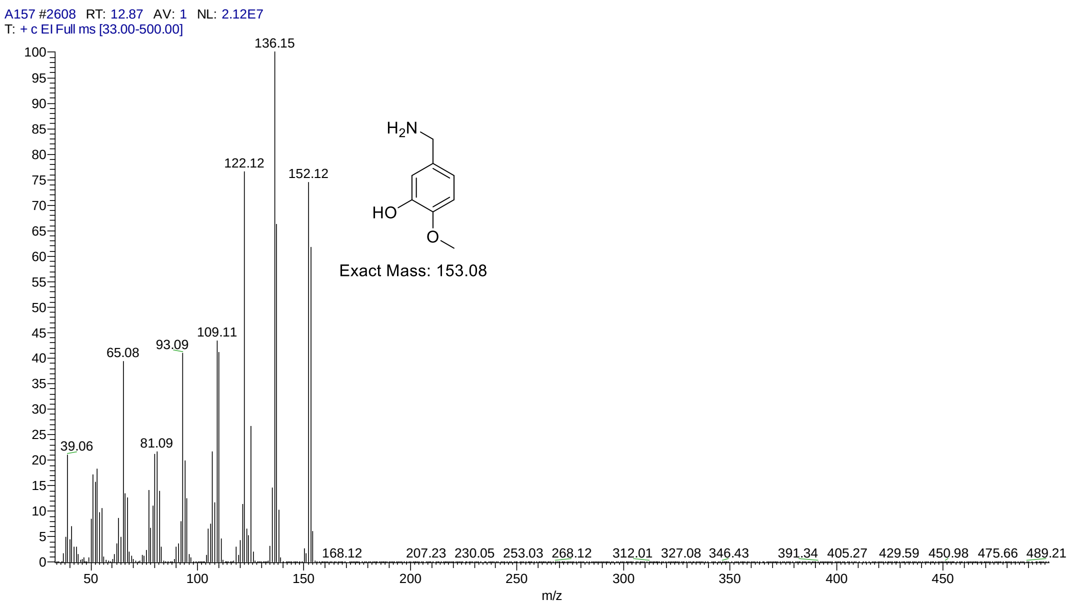
O
OH
15
10
5
0
f2 (ppm)



NH2
O
OH
15
10
5
0
f2 (ppm)

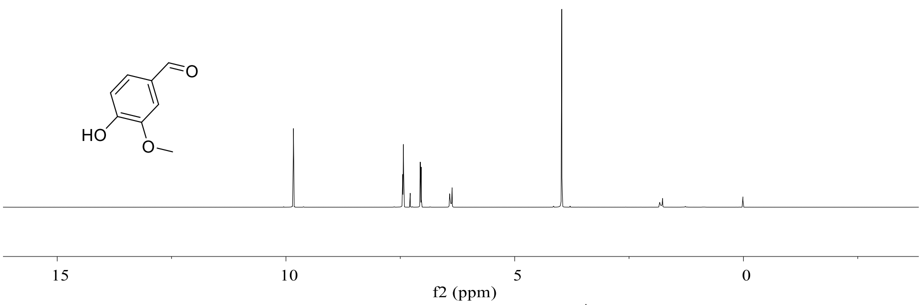


RT: 0.00 - 37.11
NL:
2.18E8
TIC MS
A157
12.87
12.34
H2N
HO
O
Time (min)

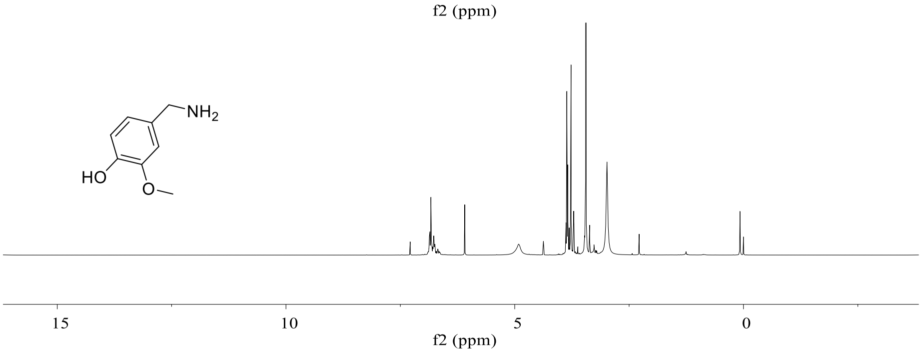


A157 #2608 RT: 12.87 AV: 1 NL: 2.12E7
T: + c EI Full ms [33.00-500.00]
136.15
122.12
152.12
109.11
93.09
65.08
81.09
39.06
H2N
HO
O
Exact Mass: 153.08
m/z

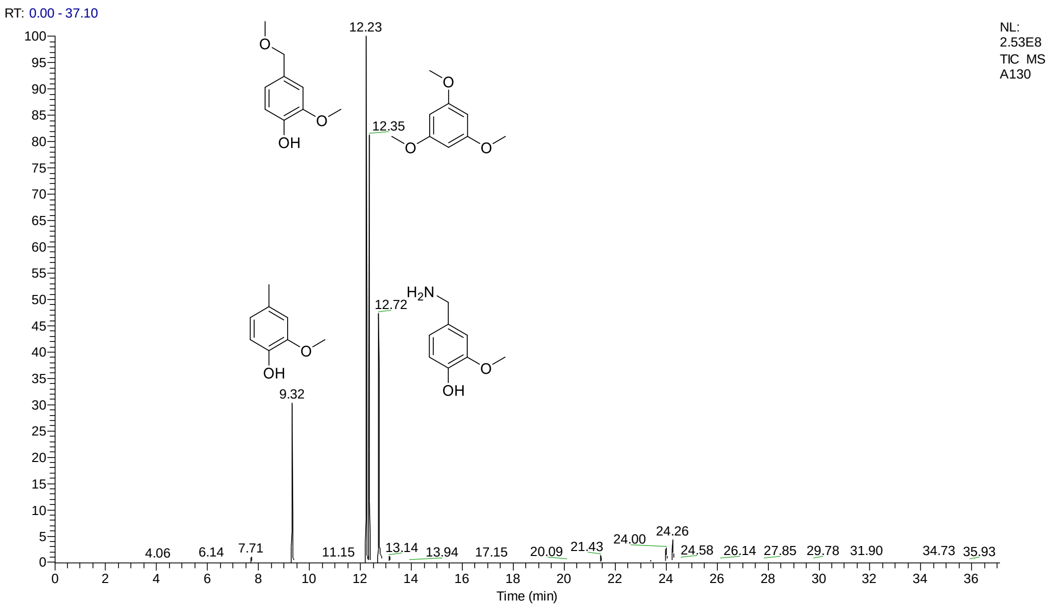
A130



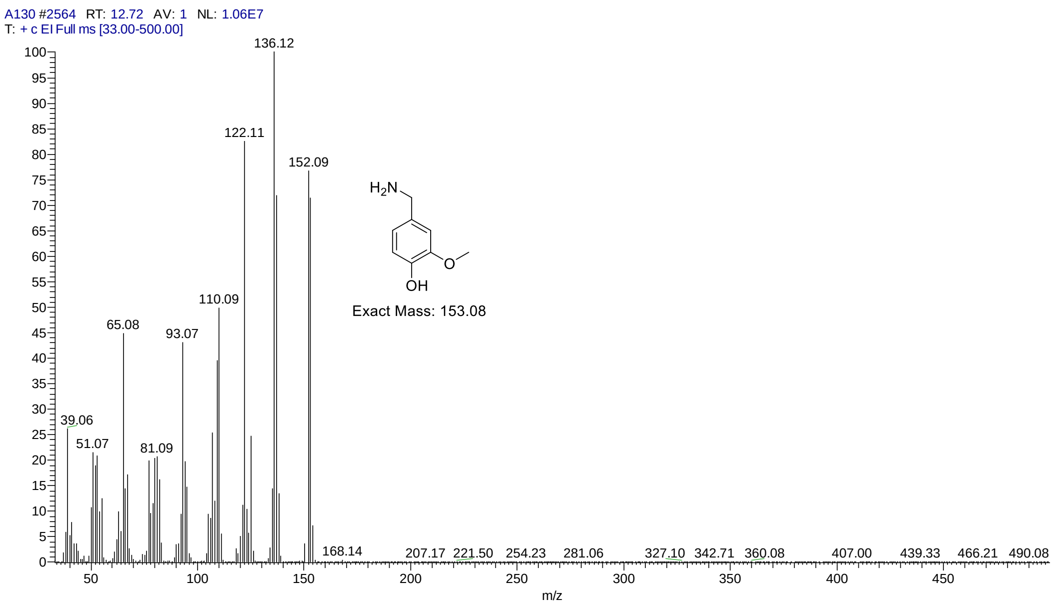
HO
O
O
15
10
5
0
f2 (ppm)



NH2
HO
O
15
10
5
0
f2 (ppm)

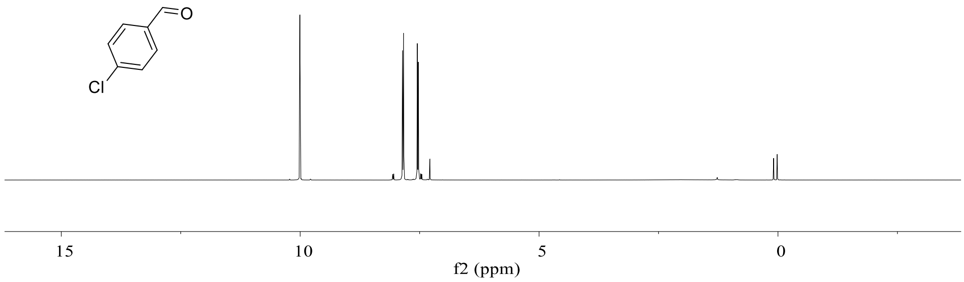


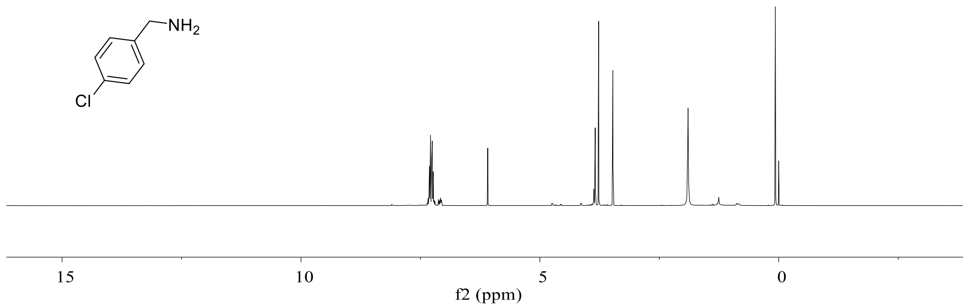
RT: 0.00 - 37.10
NL: 2.53E8 TIC MS A130
12.23
12.35
12.72
9.32
4.06
6.14
7.71
11.15
13.14
13.94
17.15
20.09
21.43
24.00
24.26
24.58
26.14
27.85
29.78
31.90
34.73
35.93
Time (min)

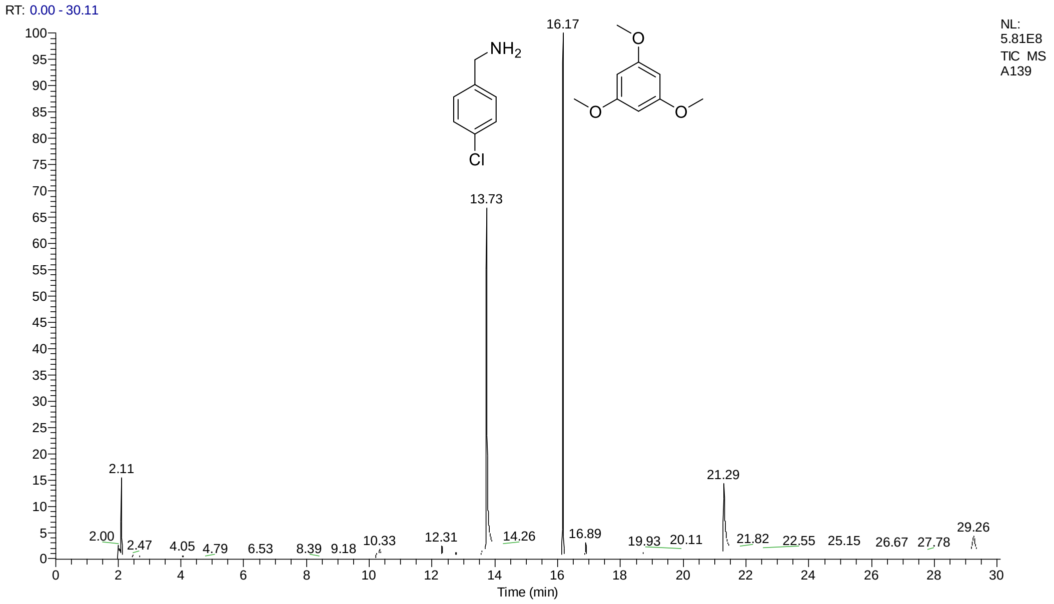


A130 #2564 RT: 12.72 AV: 1 NL: 1.06E7
T: + c EI Full ms [33.00-500.00]
136.12
122.11
152.09
110.09
65.08
93.07
39.06
51.07
81.09
168.14
207.17
221.50
254.23
281.06
327.10
342.71
360.08
407.00
439.33
466.21
490.08
Exact Mass: 153.08
m/z

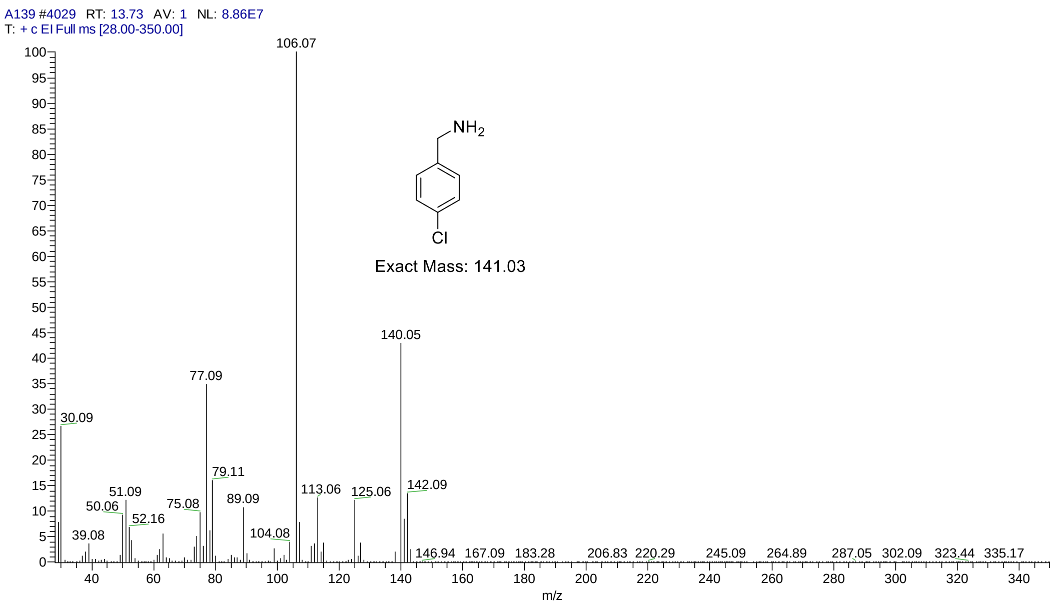
A139



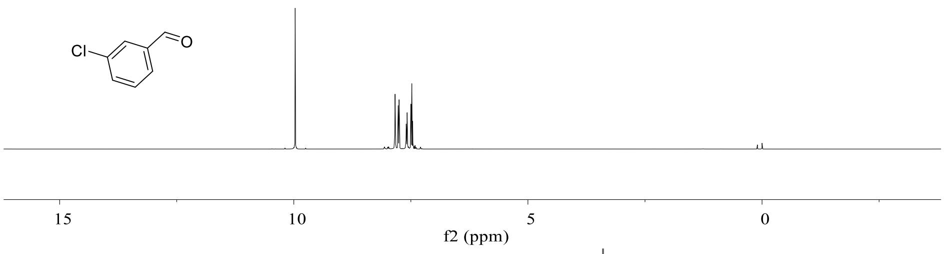




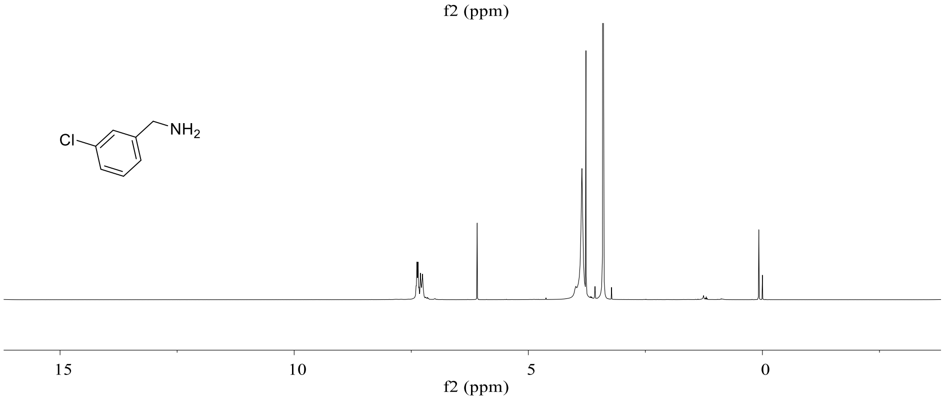
(Innovax column)



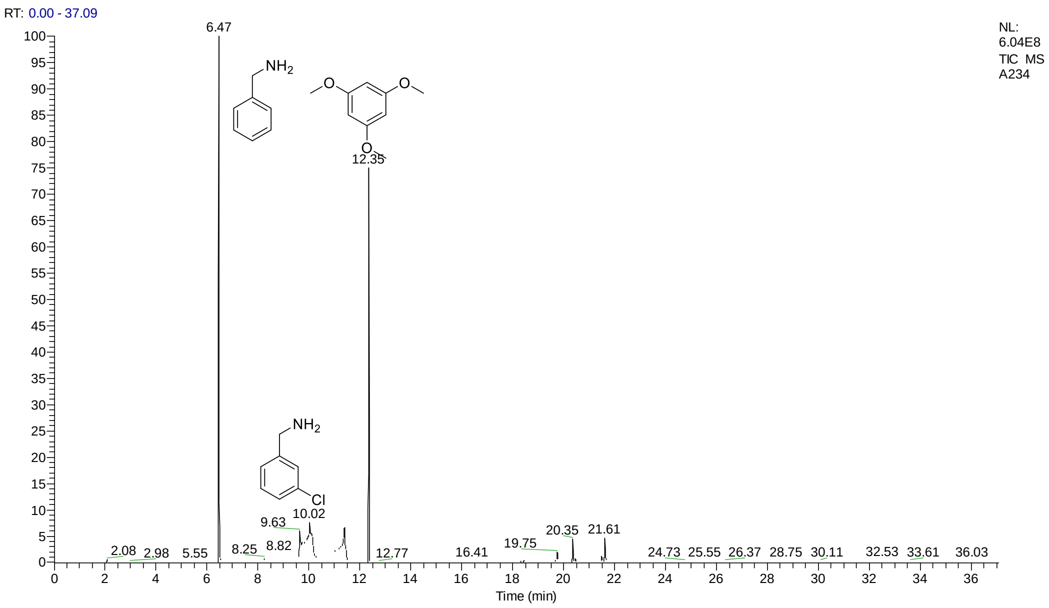
A234



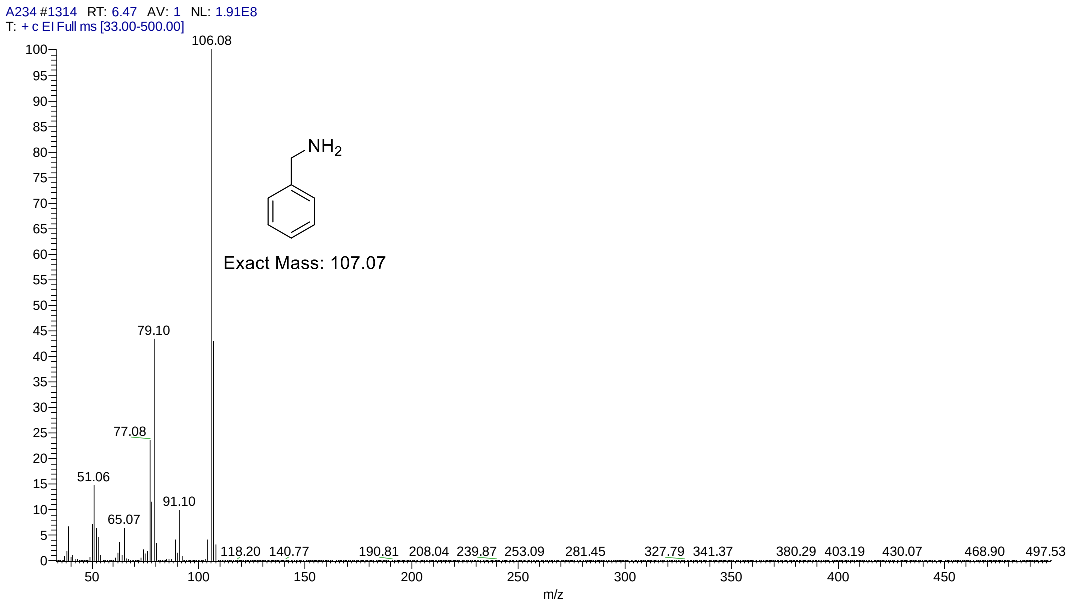
Cl
O
15
10
5
0
f2 (ppm)

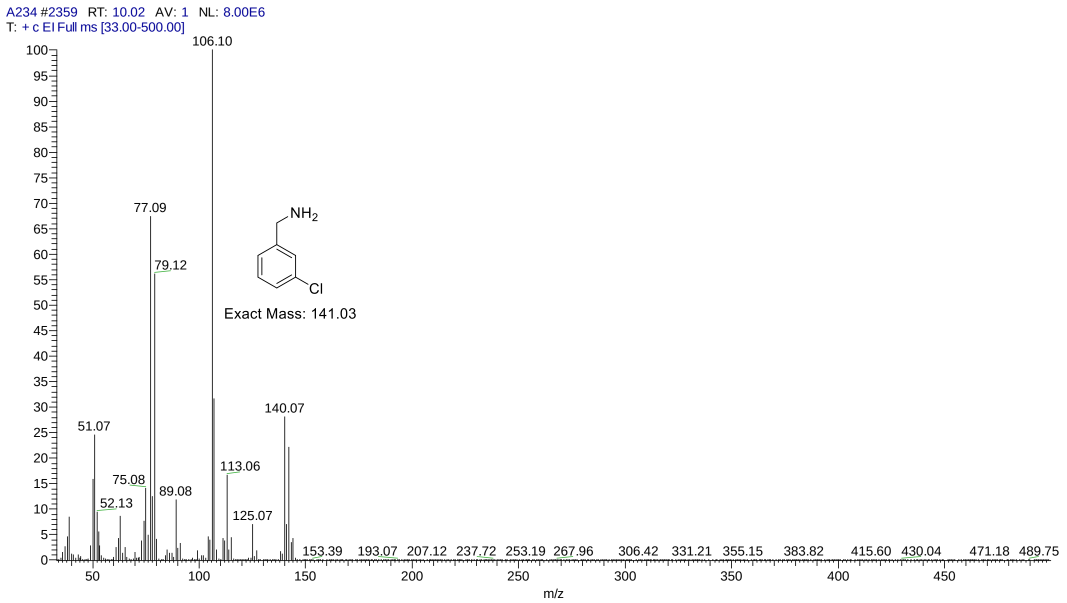


Cl
NH2
15
10
5
0
f2 (ppm)

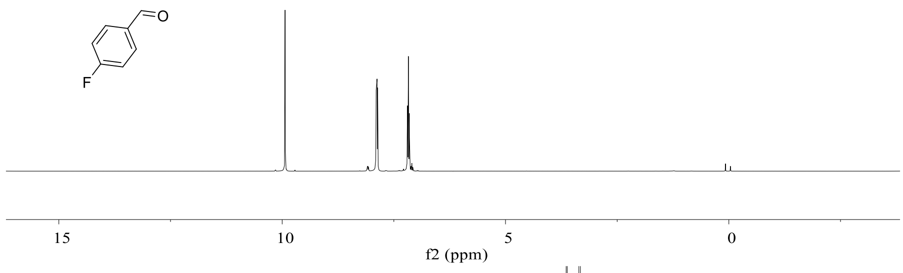


RT: 0.00 - 37.09
NL:
6.04E8
TIC MS
A234
6.47
12.35
10.02
9.63
20.35
21.61
19.75
2.08
2.98
5.55
8.25
8.82
12.77
16.41
24.73
25.55
26.37
28.75
30.11
32.53
33.61
36.03
NH2
O
Cl
Time (min)

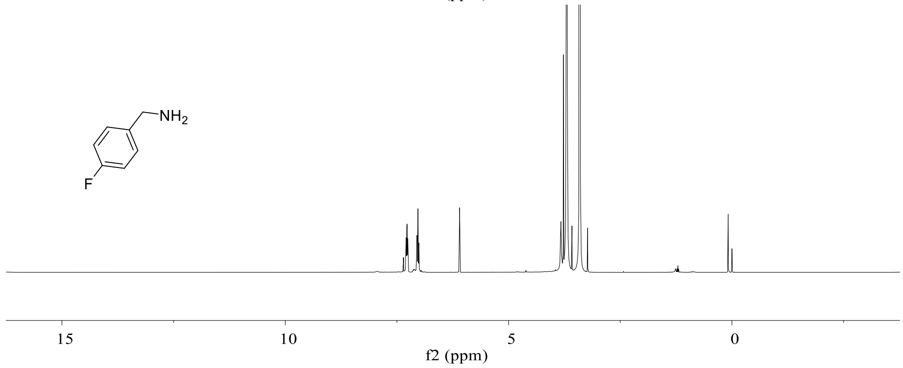

A234 #1314 RT: 6.47 AV: 1 NL: 1.91E8
T: + c EI Full ms [33.00-500.00]
106.08
NH2
Exact Mass: 107.07
79.10
77.08
51.06
91.10
65.07
118.20 140.77 190.81 208.04 239.87 253.09 281.45 327.79 341.37 380.29 403.19 430.07 468.90 497.53
m/z


A234 #2359 RT: 10.02 AV: 1 NL: 8.00E6
T: + c EI Full ms [33.00-500.00]
106.10
77.09
NH2
79.12
Cl
Exact Mass: 141.03
140.07
51.07
113.06
75.08
89.08
52.13
125.07
153.39 193.07 207.12 237.72 253.19 267.96 306.42 331.21 355.15 383.82 415.60 430.04 471.18 489.75
m/z

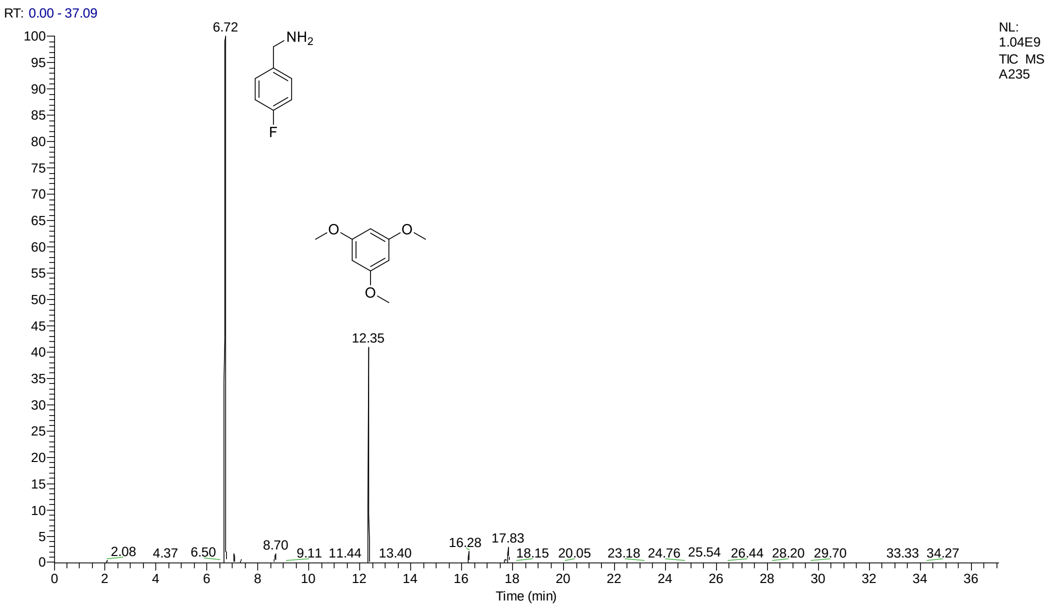
A235



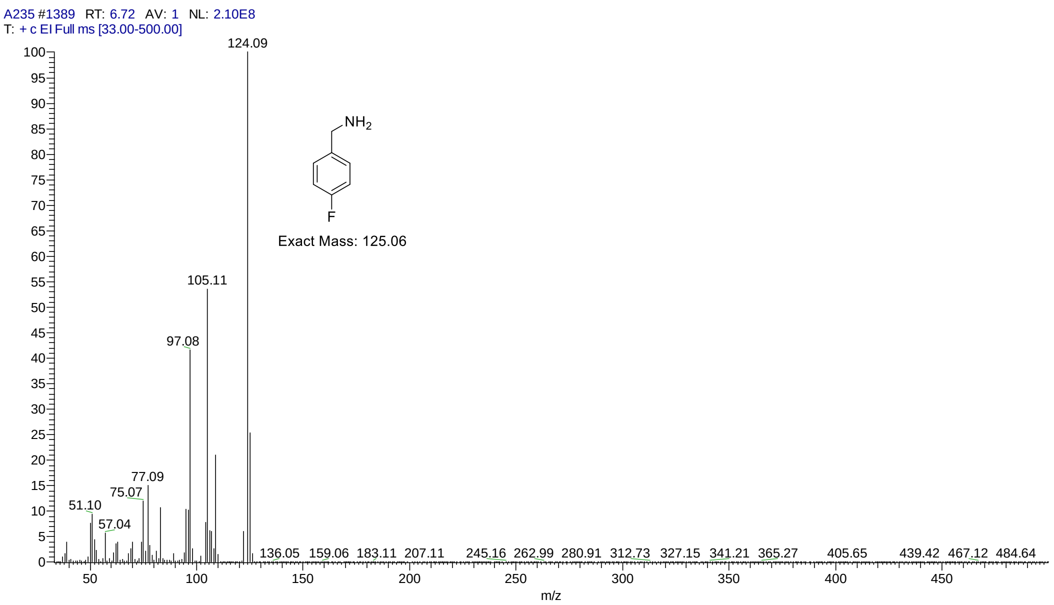
O
F
15
10
5
0
f2 (ppm)



NH2
F
15
10
5
0
f2 (ppm)

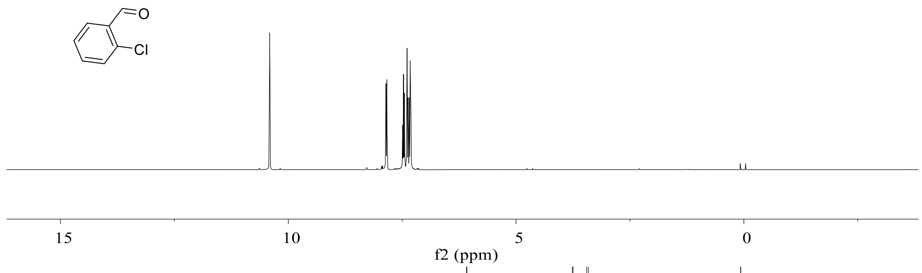


RT: 0.00 - 37.09
NL: 1.04E9 TIC MS A235
6.72
12.35
2.08
4.37
6.50
8.70
9.11
11.44
13.40
16.28
17.83
18.15
20.05
23.18
24.76
25.54
26.44
28.20
29.70
33.33
34.27
Time (min)

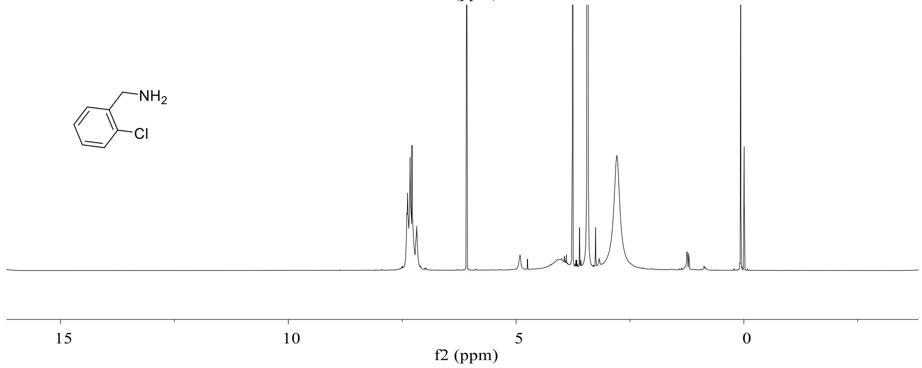


A235 #1389 RT: 6.72 AV: 1 NL: 2.10E8
T: + c EI Full ms [33.00-500.00]
124.09
105.11
97.08
77.09
75.07
51.10
57.04
136.05
159.06
183.11
207.11
245.16
262.99
280.91
312.73
327.15
341.21
365.27
405.65
439.42
467.12
484.64
Exact Mass: 125.06
m/z

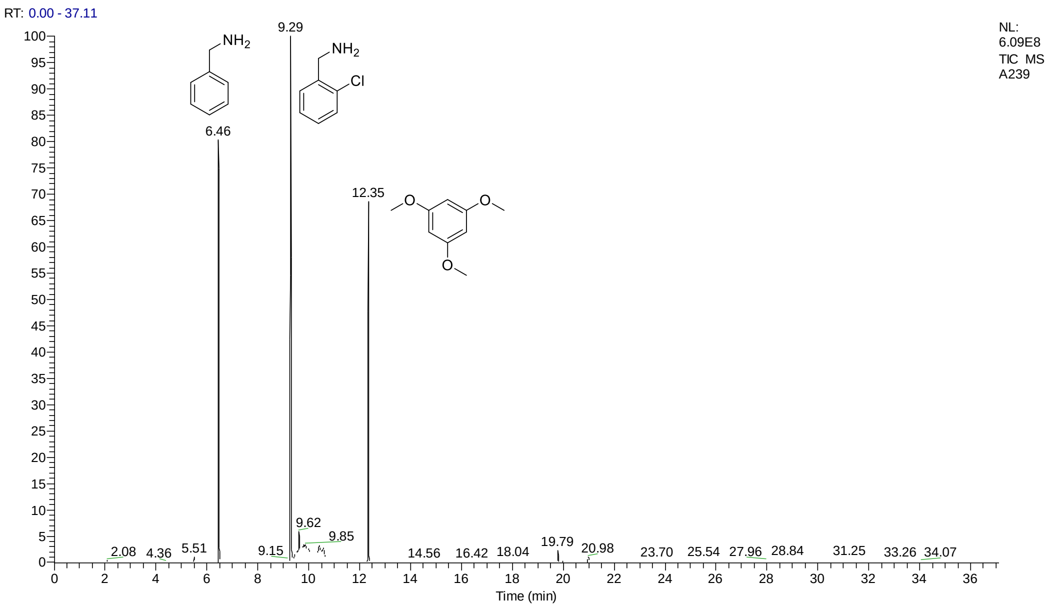
A239



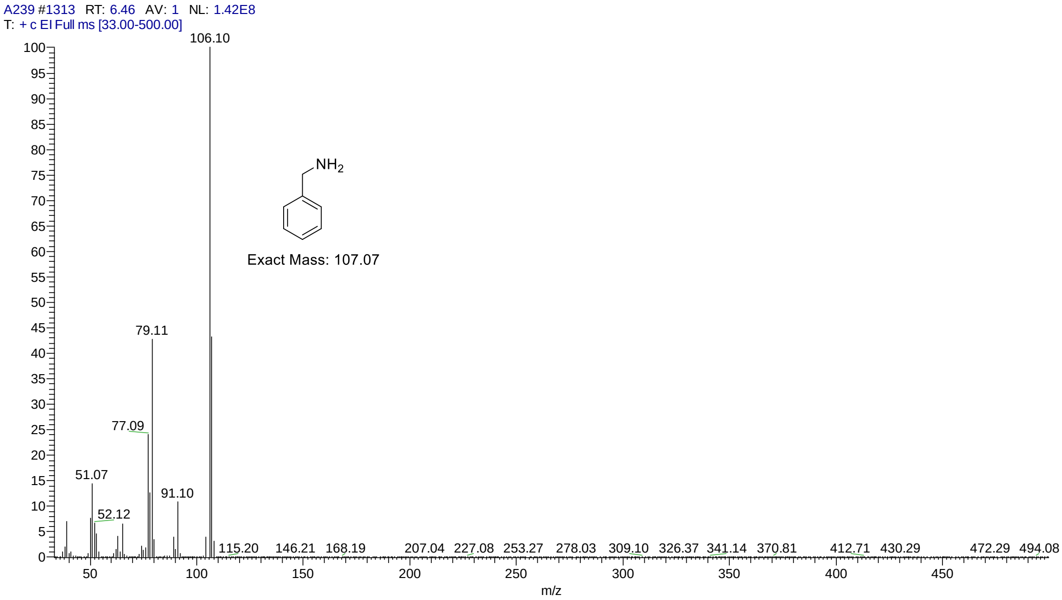
O
Cl
15
10
5
0
f2 (ppm)

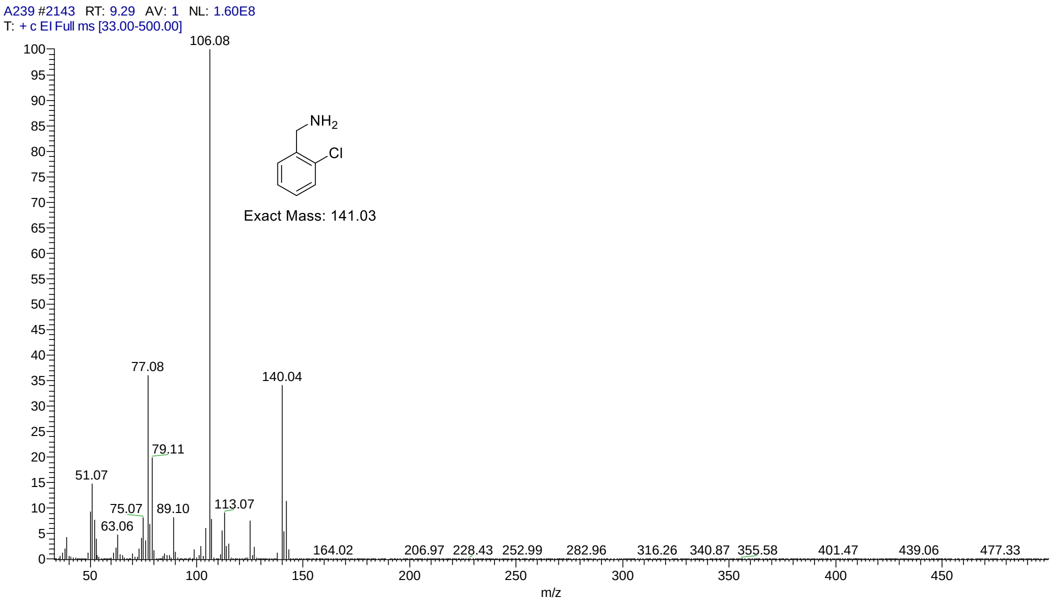


NH2
Cl
15
10
5
0
f2 (ppm)

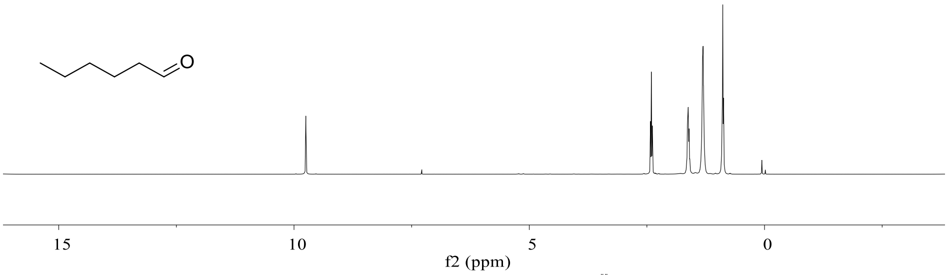


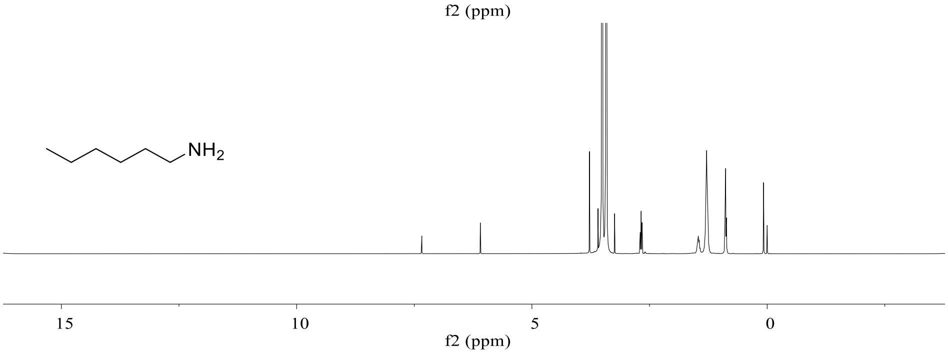
RT: 0.00 - 37.11
NL:
6.09E8
TIC MS
A239
NH2
Cl
6.46
9.29
12.35
9.62
9.85
2.08
4.36
5.51
9.15
14.56
16.42
18.04
19.79
20.98
23.70
25.54
27.96
28.84
31.25
33.26
34.07
Time (min)

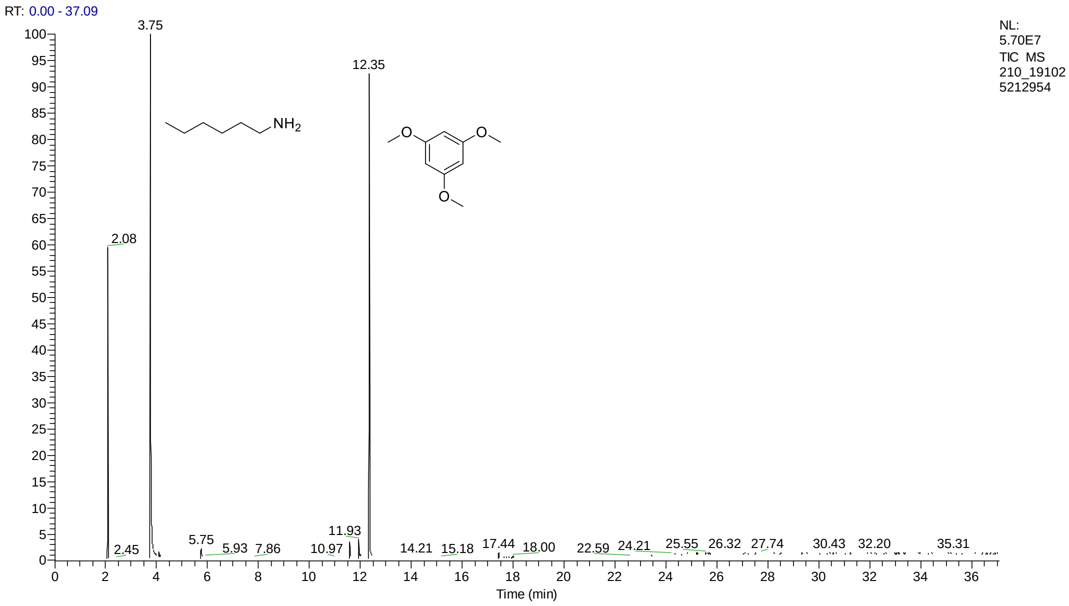

A239 #1313 RT: 6.46 AV: 1 NL: 1.42E8
T: + c EI Full ms [33.00-500.00]
106.10
NH2
Exact Mass: 107.07
79.11
77.09
51.07
52.12
91.10
115.20 146.21 168.19 207.04 227.08 253.27 278.03 309.10 326.37 341.14 370.81 412.71 430.29 472.29 494.08
m/z

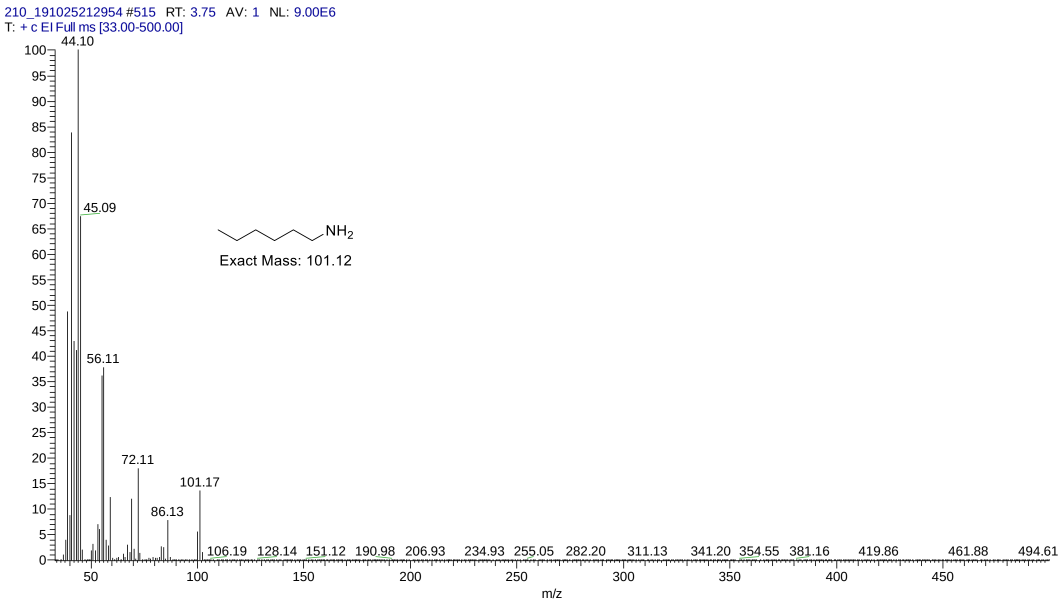

A239 #2143 RT: 9.29 AV: 1 NL: 1.60E8
T: + c EI Full ms [33.00-500.00]
106.08
NH2
Cl
Exact Mass: 141.03
77.08
140.04
79.11
51.07
75.07
63.06
89.10
113.07
164.02 206.97 228.43 252.99 282.96 316.26 340.87 355.58 401.47 439.06 477.33
m/z

A210

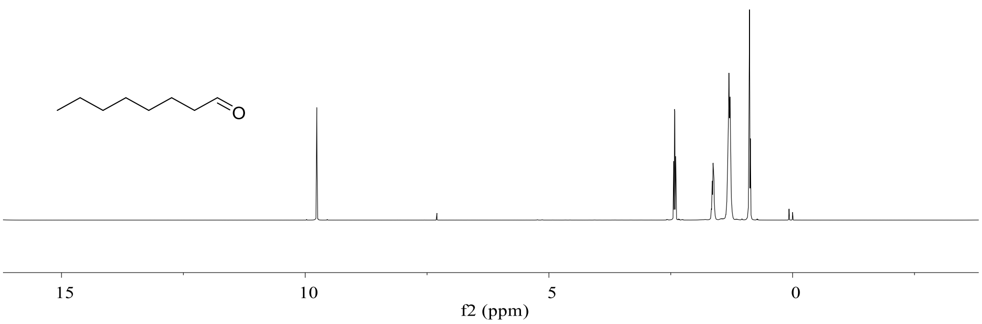

O
15
10
5
0
f2 (ppm)

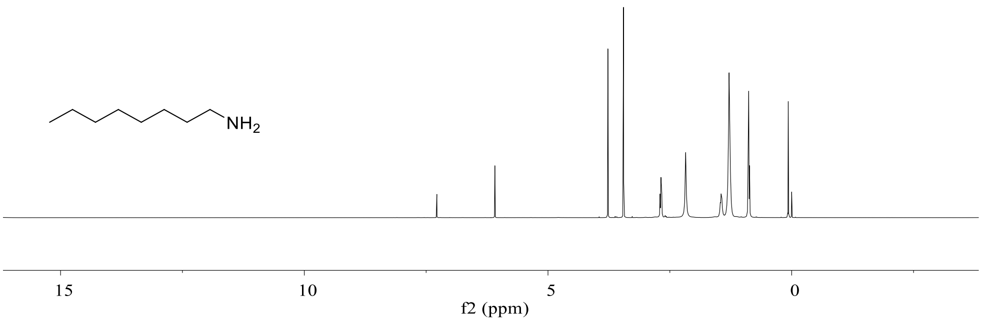

NH2
15
10
5
0
f2 (ppm)

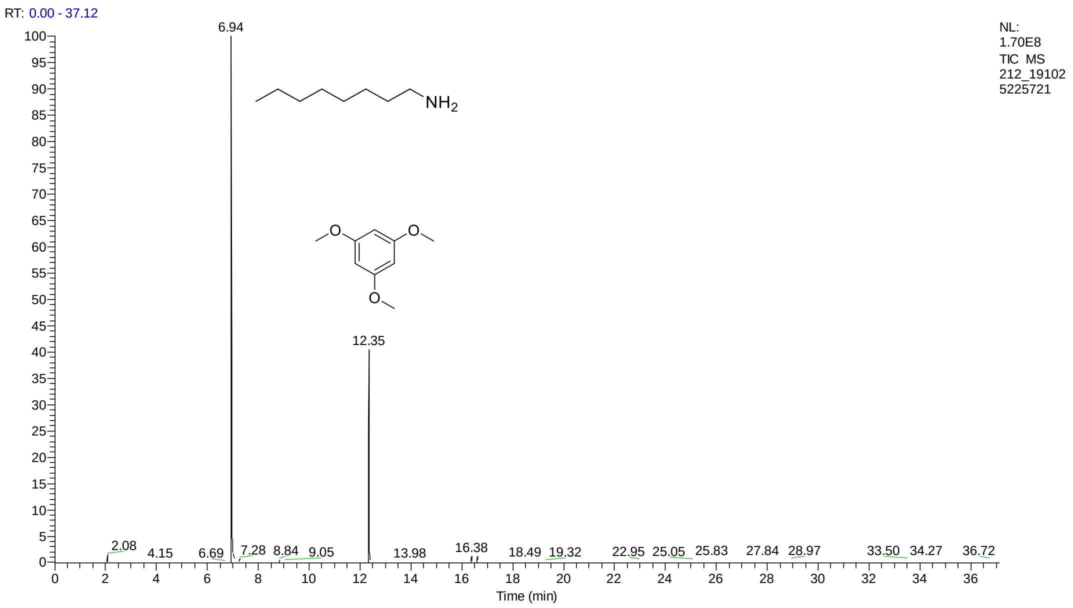

RT: 0.00 - 37.09
NL:
5.70E7
TIC MS
210_19102
5212954
3.75
12.35
2.08
NH2
O
O
O
2.45
5.75
5.93
7.86
10.97
11.93
14.21
15.18
17.44
18.00
22.59
24.21
25.55
26.32
27.74
30.43
32.20
35.31
Time (min)

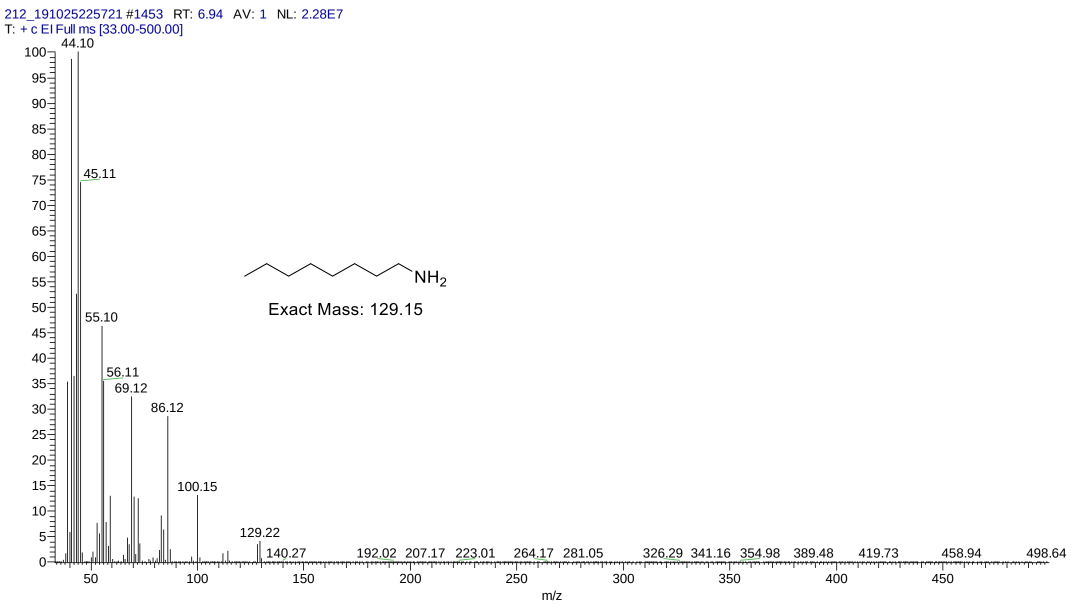

210_191025212954 #515 RT: 3.75 AV: 1 NL: 9.00E6
T: + c EI Full ms [33.00-500.00]
44.10
45.09
56.11
72.11
101.17
86.13
NH2
Exact Mass: 101.12
106.19
128.14
151.12
190.98
206.93
234.93
255.05
282.20
311.13
341.20
354.55
381.16
419.86
461.88
494.61
m/z

A212

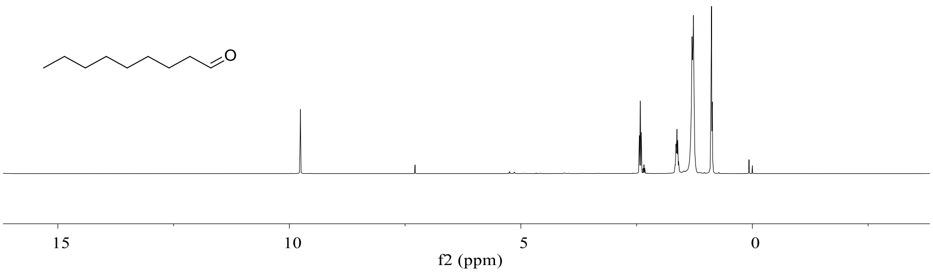

O
15
10
5
0
f2 (ppm)


NH2
15
10
5
0
f2 (ppm)

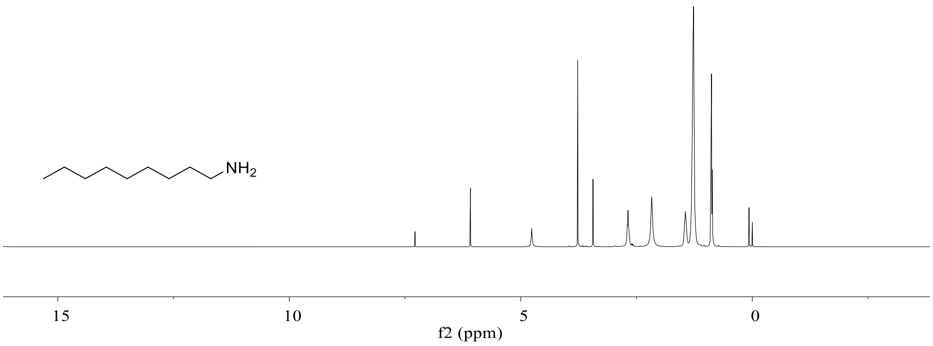

RT: 0.00 - 37.12
NL: 1.70E8 TIC MS 212_191025225721
6.94
NH2
O
O
O
12.35
2.08
4.15
6.69
7.28
8.84
9.05
13.98
16.38
18.49
19.32
22.95
25.05
25.83
27.84
28.97
33.50
34.27
36.72
Time (min)


212_191025225721 #1453 RT: 6.94 AV: 1 NL: 2.28E7
T: + c EI Full ms [33.00-500.00]
44.10
45.11
55.10
56.11
69.12
86.12
100.15
129.22
140.27
192.02
207.17
223.01
264.17
281.05
326.29
341.16
354.98
389.48
419.73
458.94
498.64
NH2
Exact Mass: 129.15
m/z

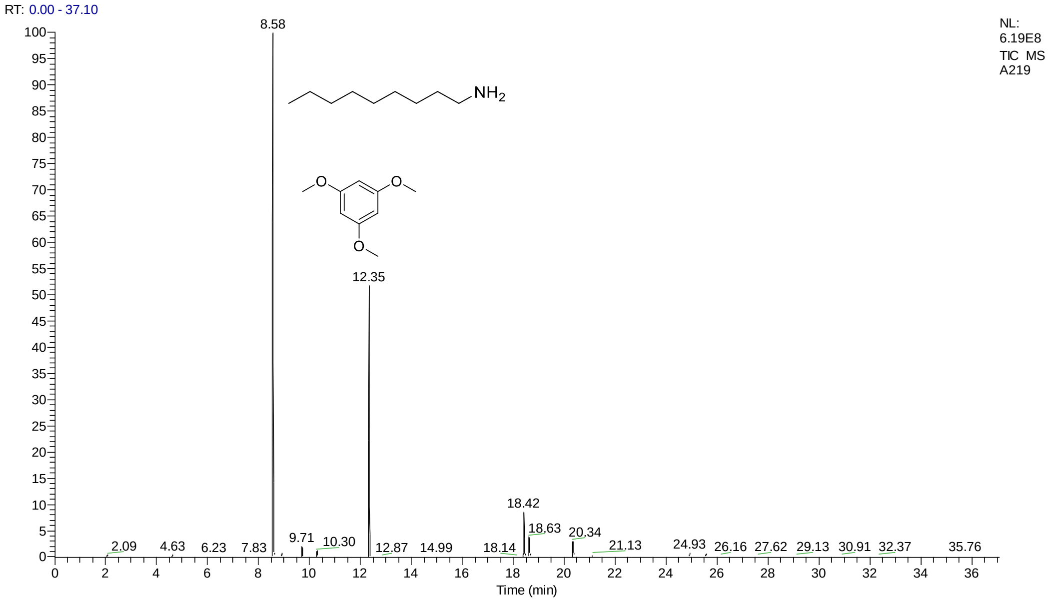
A219



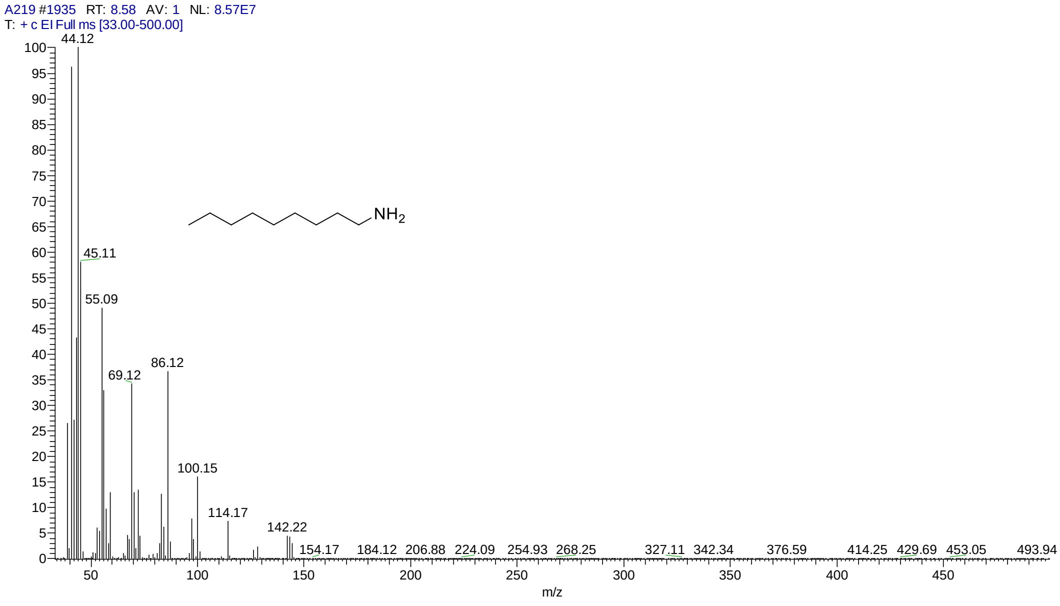
O
15
10
5
0
f2 (ppm)



NH2
15
10
5
0
f2 (ppm)

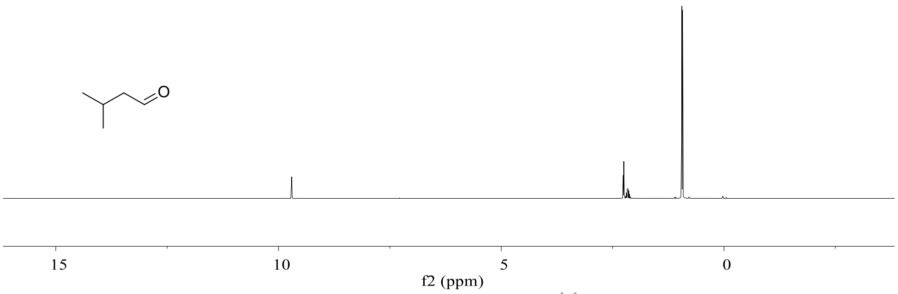


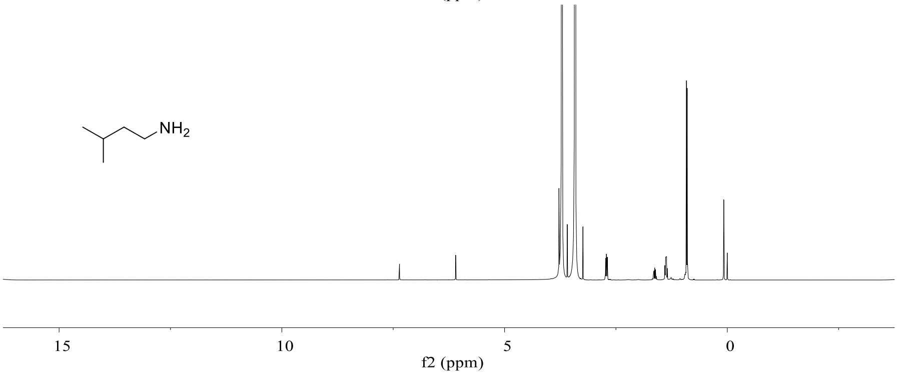
RT: 0.00 - 37.10
NL:
6.19E8
TIC MS
A219
8.58
NH2
O
O
O
12.35
18.42
18.63
20.34
2.09
4.63
6.23
7.83
9.71
10.30
12.87
14.99
18.14
21.13
24.93
26.16
27.62
29.13
30.91
32.37
35.76
Time (min)

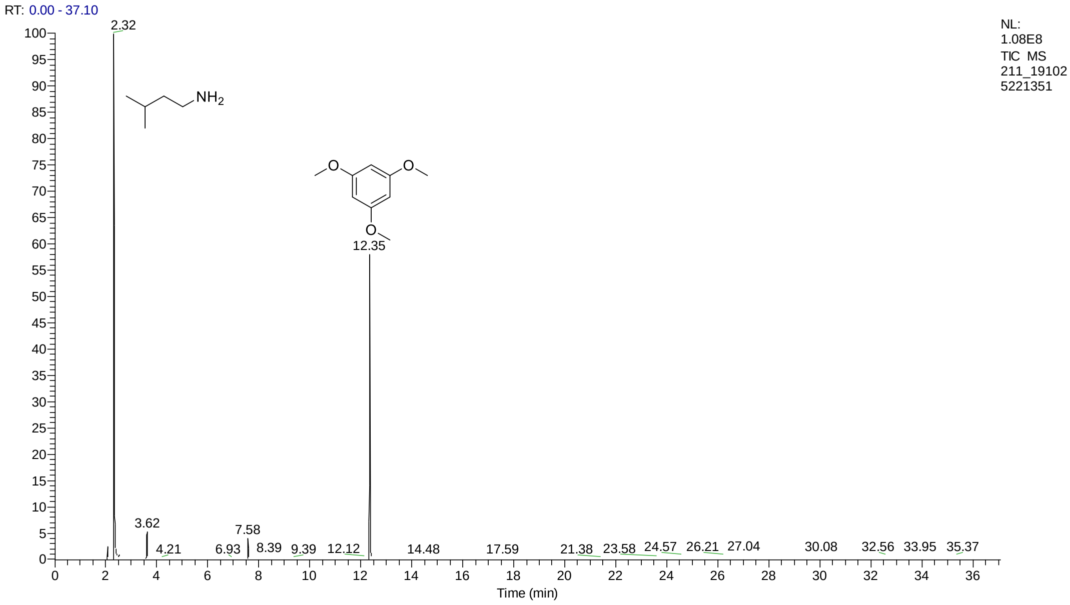


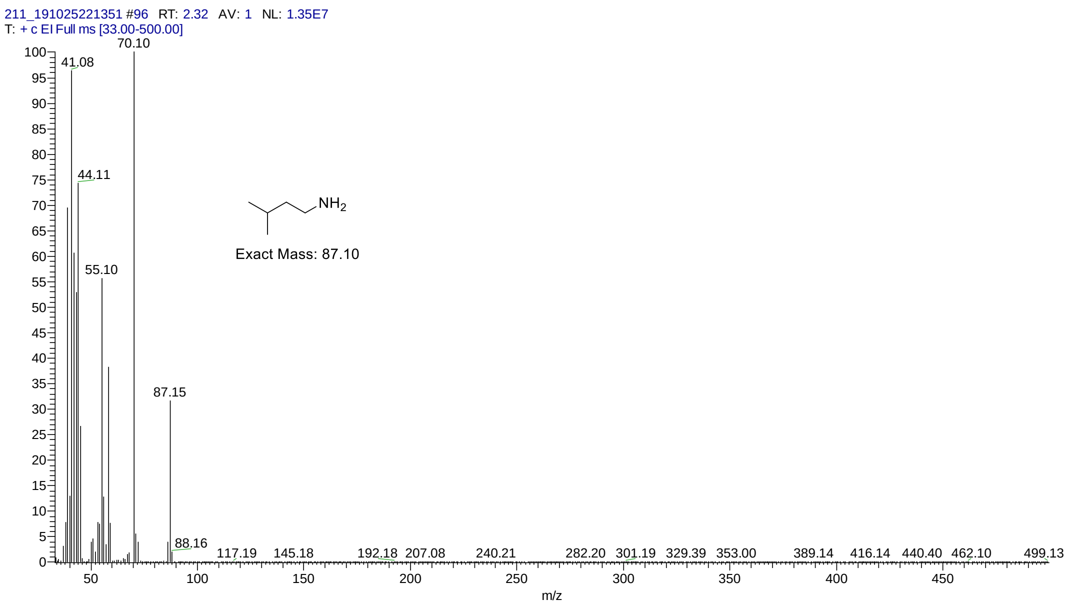
A219 #1935 RT: 8.58 AV: 1 NL: 8.57E7
T: + c EI Full ms [33.00-500.00]
44.12
45.11
55.09
69.12
86.12
100.15
114.17
142.22
154.17
184.12
206.88
224.09
254.93
268.25
327.11
342.34
376.59
414.25
429.69
453.05
493.94
NH2
m/z

A211

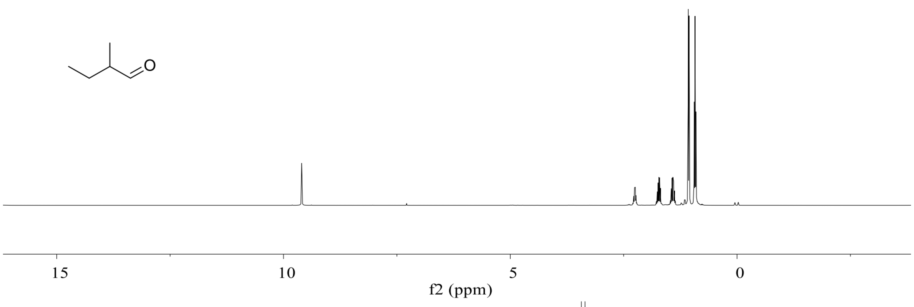

O
15
10
5
0
f2 (ppm)

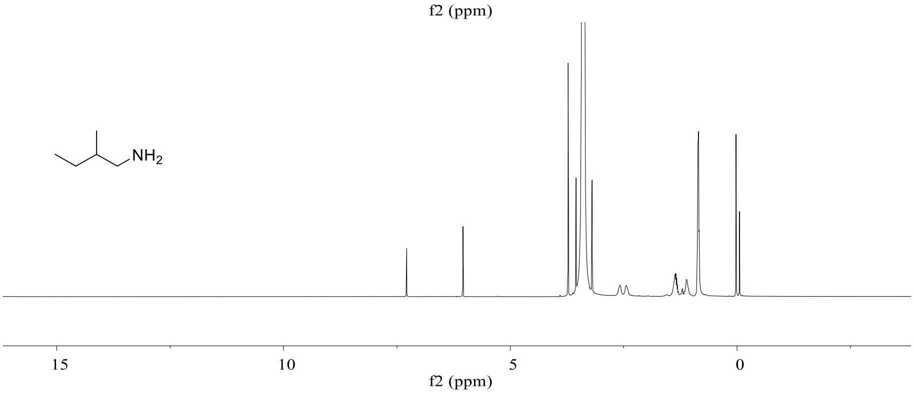

NH2
15
10
5
0
f2 (ppm)

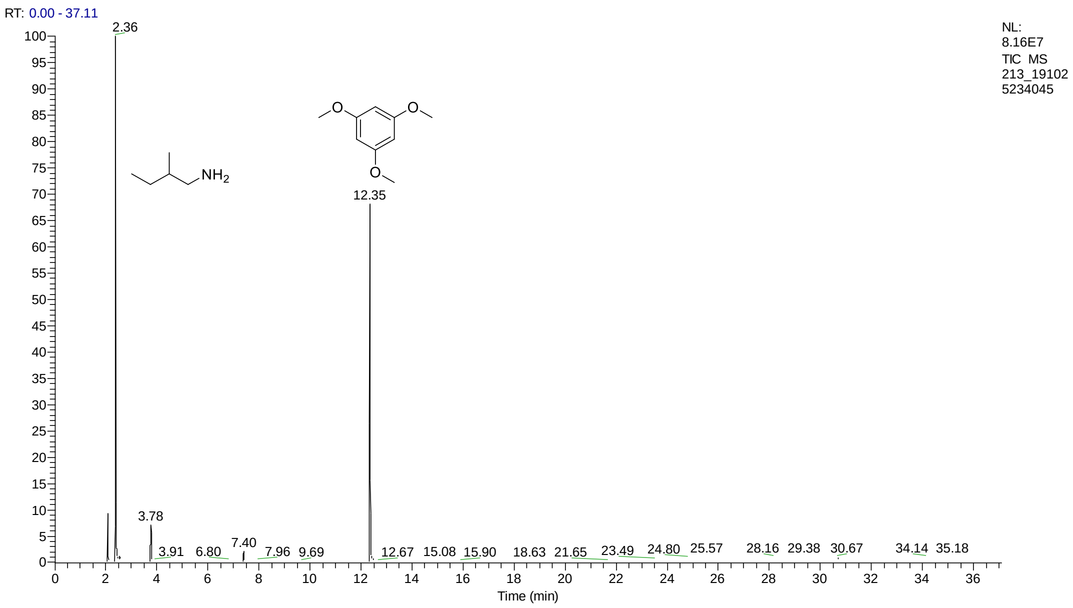

RT: 0.00 - 37.10
NL:
1.08E8
TIC MS
211_19102
5221351
2.32
NH2
O
O
O
12.35
3.62
7.58
4.21
6.93
8.39
9.39
12.12
14.48
17.59
21.38
23.58
24.57
26.21
27.04
30.08
32.56
33.95
35.37
Time (min)

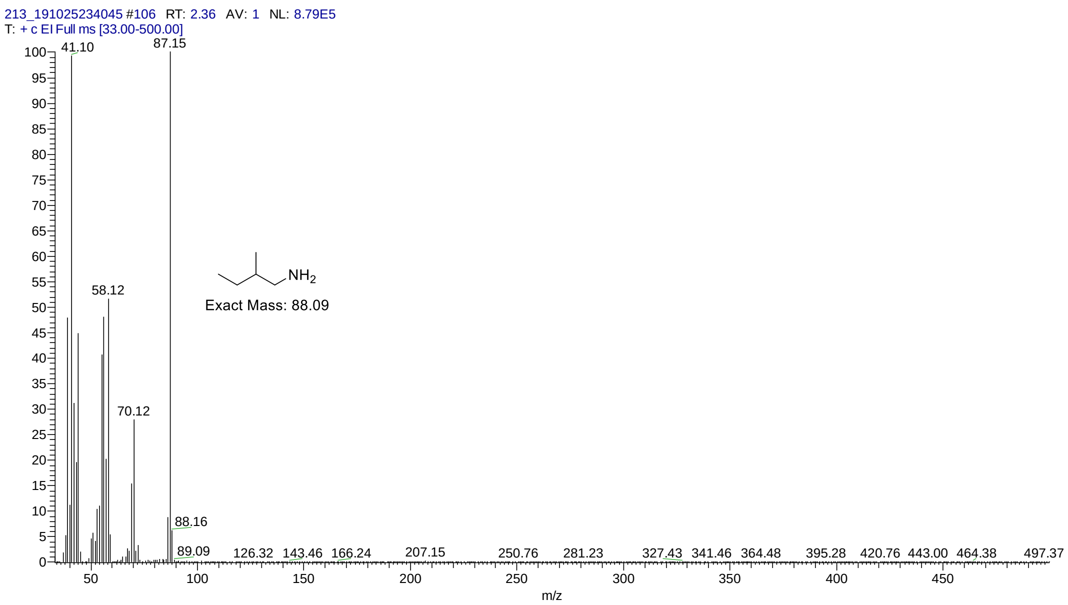

211_191025221351 #96 RT: 2.32 AV: 1 NL: 1.35E7
T: + c EI Full ms [33.00-500.00]
70.10
41.08
44.11
55.10
87.15
88.16
117.19
145.18
192.18
207.08
240.21
282.20
301.19
329.39
353.00
389.14
416.14
440.40
462.10
499.13
NH2
Exact Mass: 87.10
m/z

A213

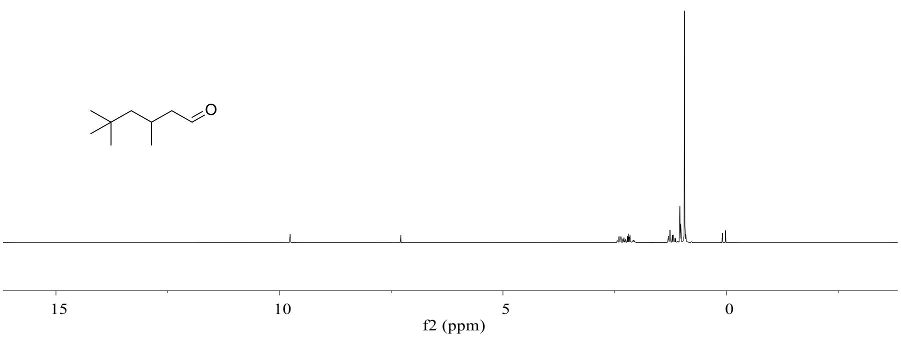

O
15
10
5
0
f2 (ppm)


NH2
15
10
5
0
f2 (ppm)

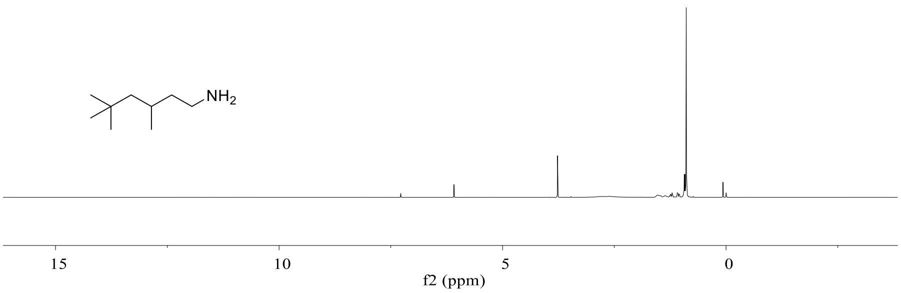

RT: 0.00 - 37.11
NL:
8.16E7
TIC MS
213_19102
5234045
NH2
12.35
2.36
3.78
3.91 6.80 7.40 7.96 9.69 12.67 15.08 15.90 18.63 21.65 23.49 24.80 25.57 28.16 29.38 30.67 34.14 35.18
Time (min)


213_191025234045 #106 RT: 2.36 AV: 1 NL: 8.79E5
T: + c EI Full ms [33.00-500.00]
41.10
87.15
58.12
70.12
88.16
89.09 126.32 143.46 166.24 207.15 250.76 281.23 327.43 341.46 364.48 395.28 420.76 443.00 464.38 497.37
NH2
Exact Mass: 88.09
m/z

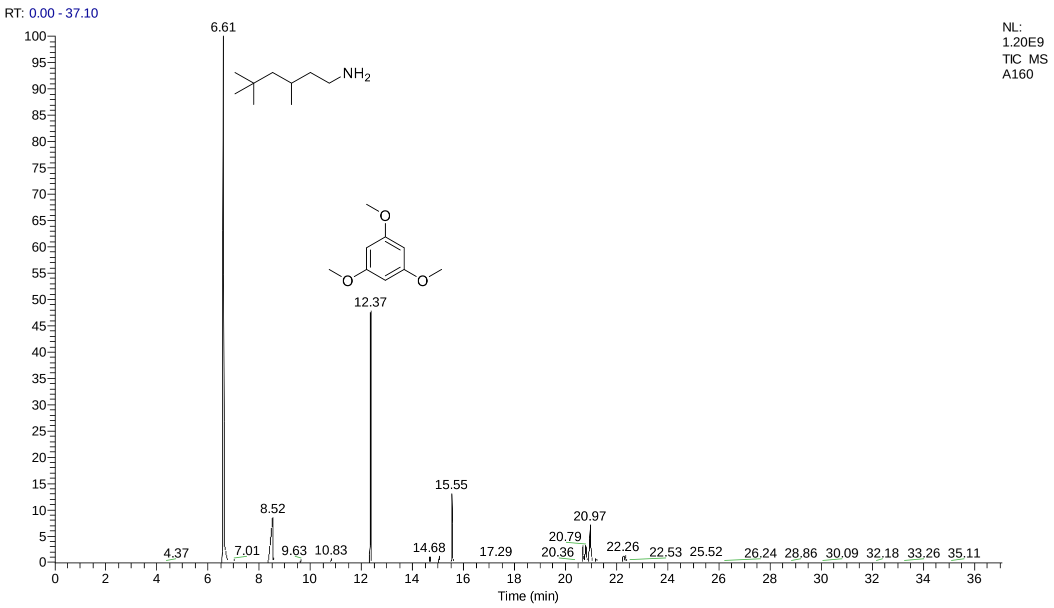
A160



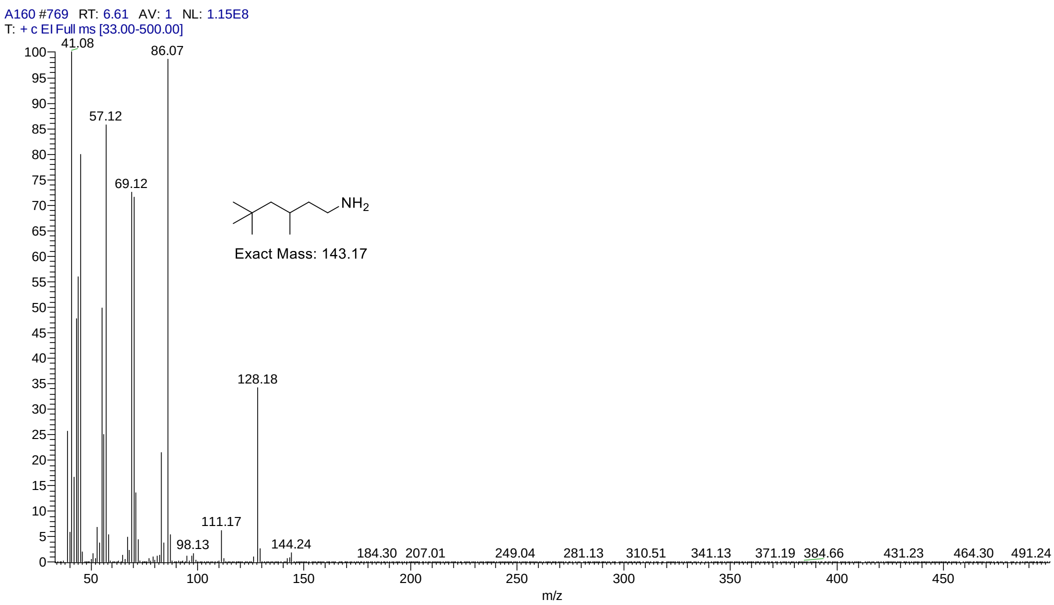
O
15
10
5
0
f2 (ppm)



NH2
15
10
5
0
f2 (ppm)

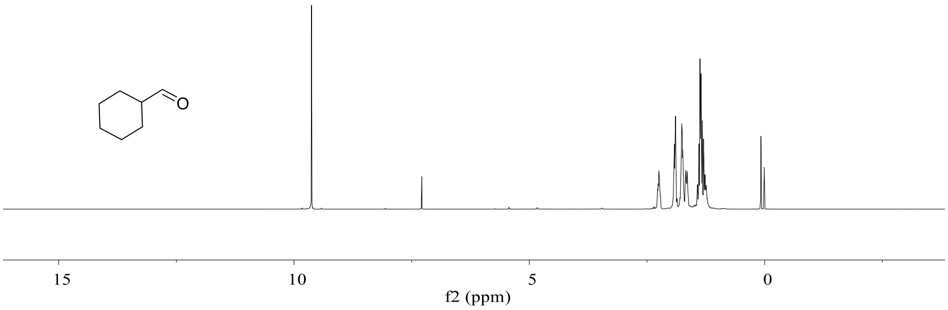


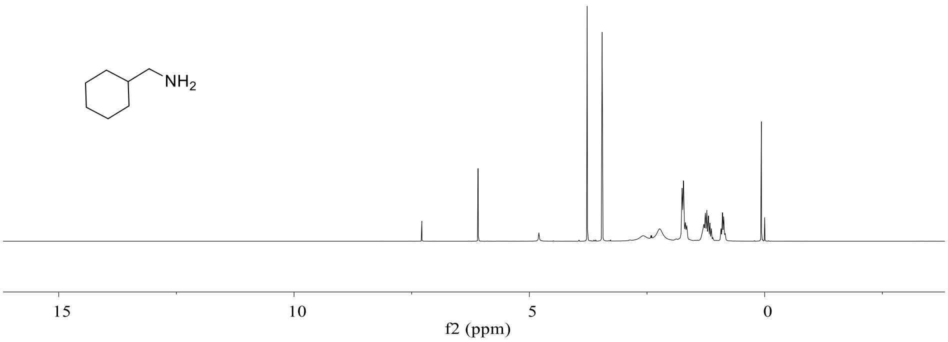
RT: 0.00 - 37.10
NL: 1.20E9 TIC MS A160
NH2
6.61
12.37
8.52
15.55
20.97
20.79
4.37 7.01 9.63 10.83 14.68 17.29 20.36 22.26 22.53 25.52 26.24 28.86 30.09 32.18 33.26 35.11
Time (min)



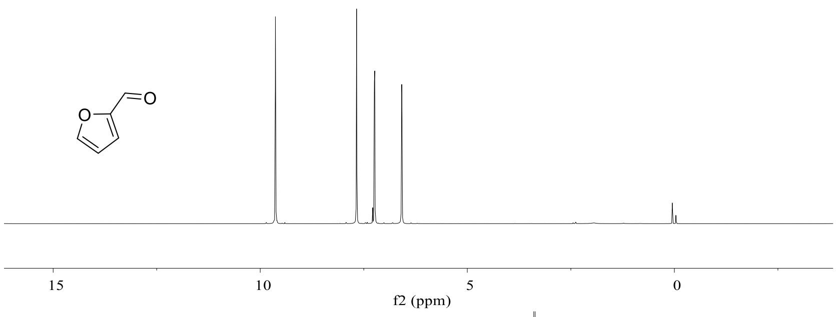
A160 #769 RT: 6.61 AV: 1 NL: 1.15E8
T: + c EI Full ms [33.00-500.00]
41.08
86.07
57.12
69.12
NH2
Exact Mass: 143.17
128.18
111.17
98.13
144.24
184.30 207.01 249.04 281.13 310.51 341.13 371.19 384.66 431.23 464.30 491.24
m/z

A186

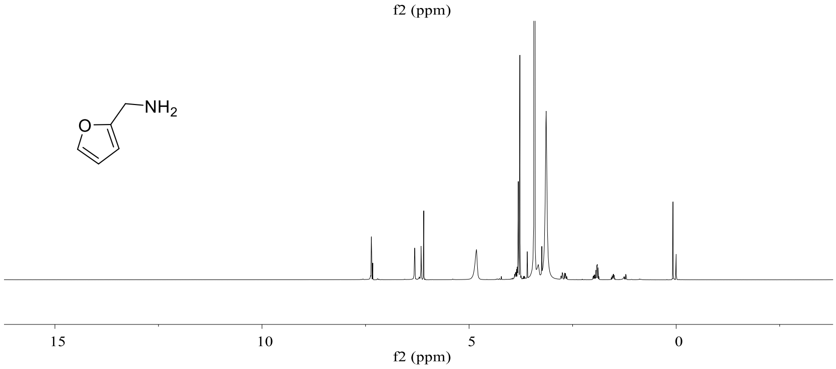




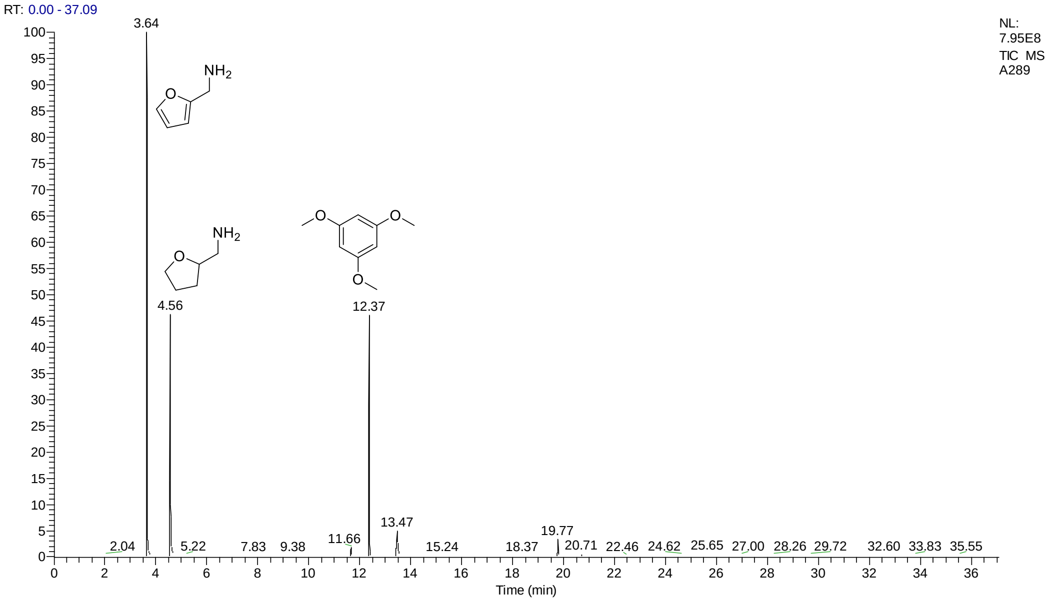
A289



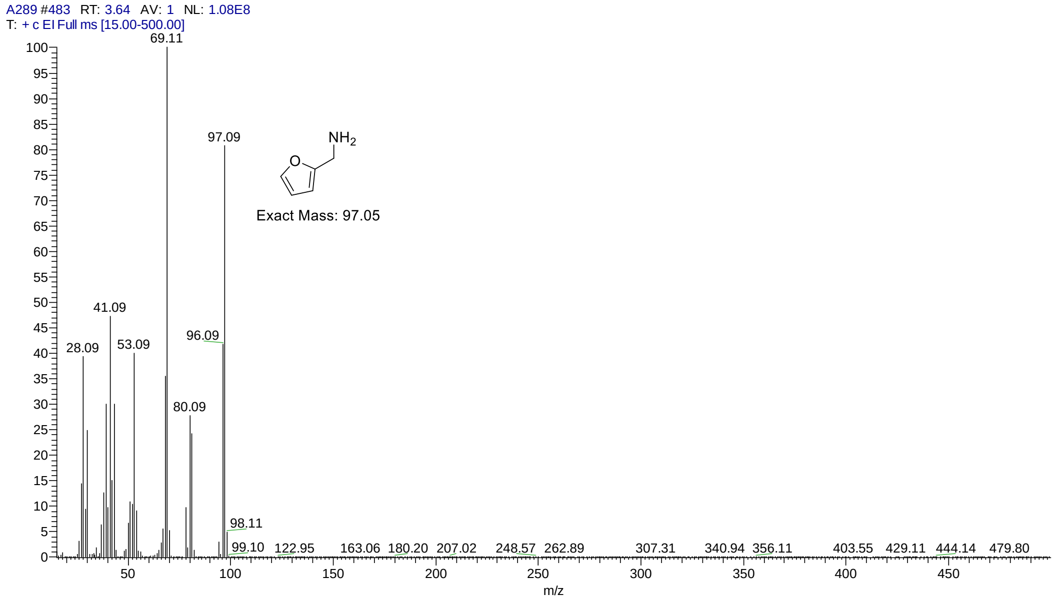
15
10
5
0
f2 (ppm)

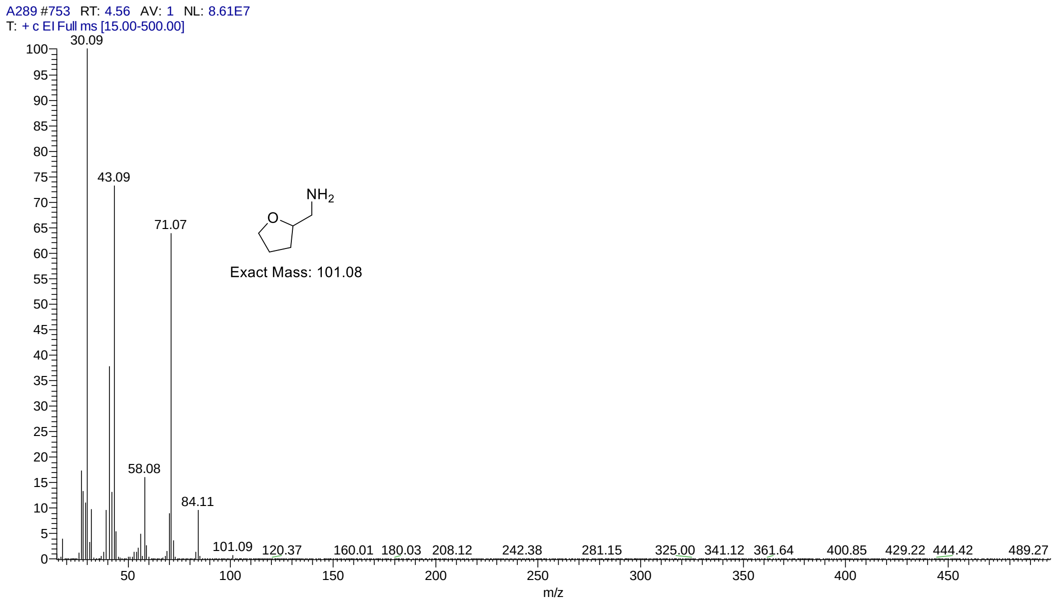


NH2
15
10
5
0
f2 (ppm)

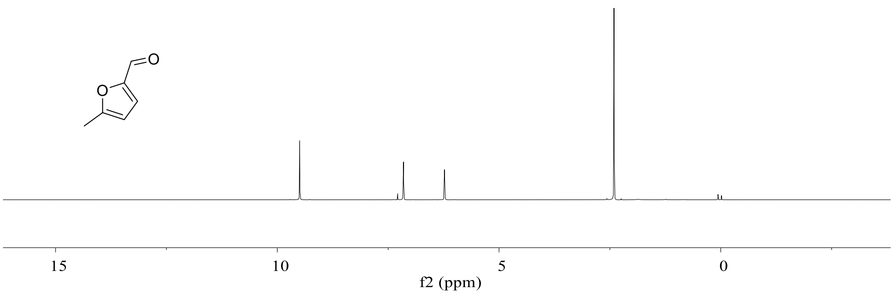


RT: 0.00 - 37.09
NL:
7.95E8
TIC MS
A289
3.64
NH2
NH2
4.56
12.37
13.47
2.04
5.22
7.83
9.38
11.66
15.24
18.37
19.77
20.71
22.46
24.62
25.65
27.00
28.26
29.72
32.60
33.83
35.55
Time (min)

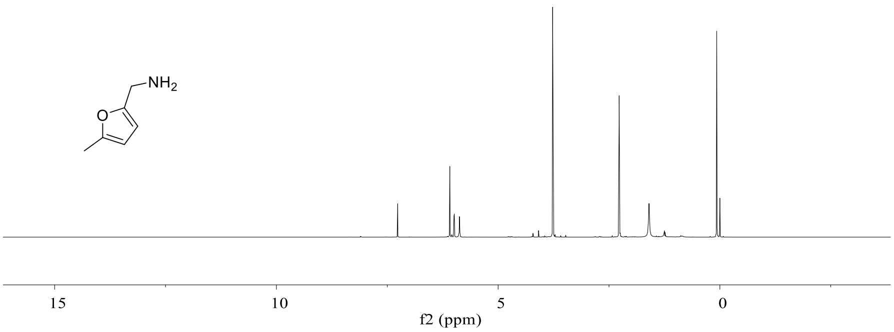

A289 #483 RT: 3.64 AV: 1 NL: 1.08E8
T: + c EI Full ms [15.00-500.00]
NH2
O
Exact Mass: 97.05
69.11
97.09
41.09
28.09
53.09
96.09
80.09
98.11
99.10
122.95
163.06
180.20
207.02
248.57
262.89
307.31
340.94
356.11
403.55
429.11
444.14
479.80
m/z


A289 #753 RT: 4.56 AV: 1 NL: 8.61E7
T: + c EI Full ms [15.00-500.00]
NH2
O
Exact Mass: 101.08
30.09
43.09
71.07
58.08
84.11
101.09
120.37
160.01
180.03
208.12
242.38
281.15
325.00
341.12
361.64
400.85
429.22
444.42
489.27
m/z

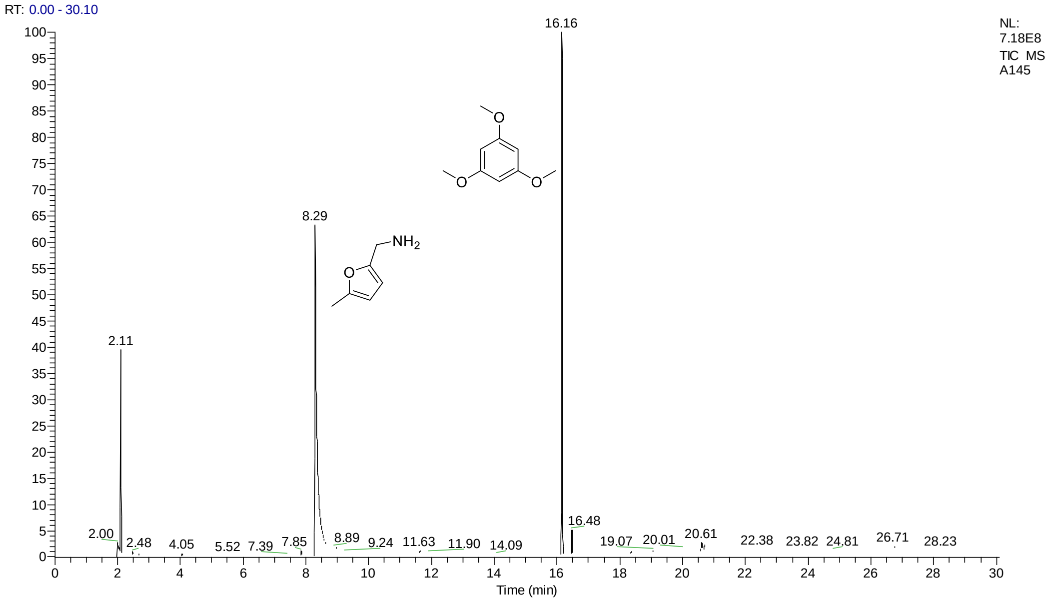
A145



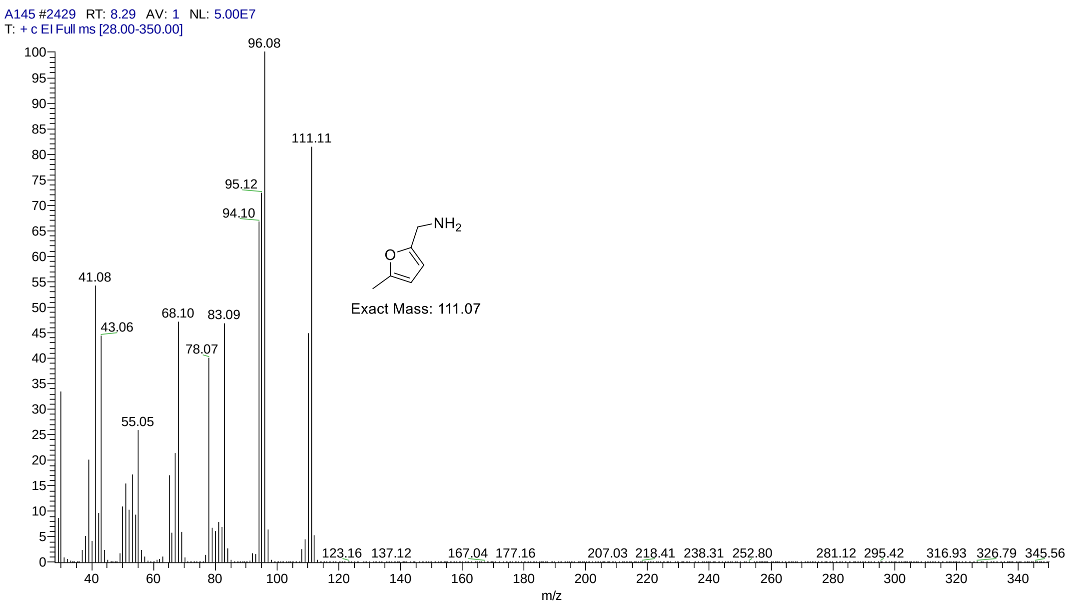
O
15
10
5
0
f2 (ppm)



NH2
O
15
10
5
0
f2 (ppm)

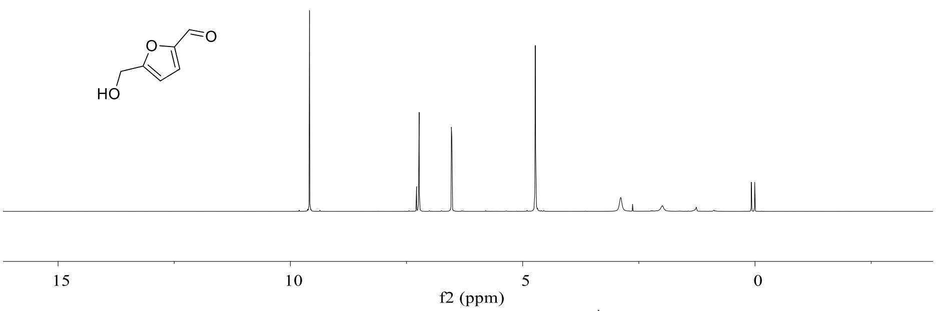


RT: 0.00 - 30.10
NL:
7.18E8
TIC MS
A145
16.16
8.29
2.11
2.00
2.48
4.05
5.52
7.39
7.85
8.89
9.24
11.63
11.90
14.09
16.48
19.07
20.01
20.61
22.38
23.82
24.81
26.71
28.23
NH2
Time (min)

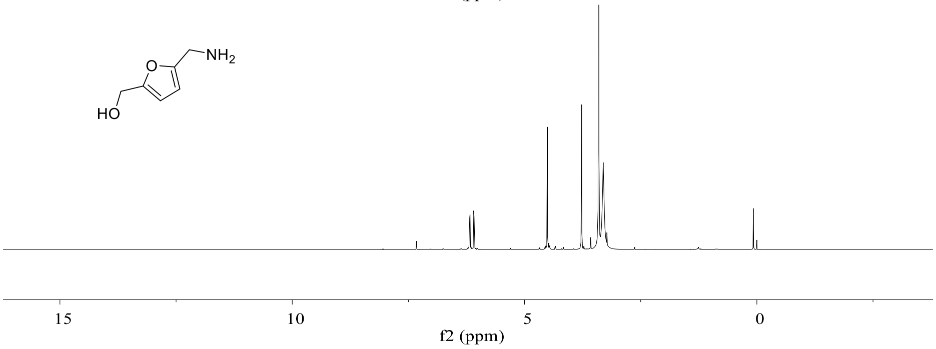


A145 #2429 RT: 8.29 AV: 1 NL: 5.00E7
T: + c EI Full ms [28.00-350.00]
96.08
111.11
95.12
94.10
41.08
43.06
68.10
83.09
78.07
55.05
123.16
137.12
167.04
177.16
207.03
218.41
238.31
252.80
281.12
295.42
316.93
326.79
345.56
NH2
Exact Mass: 111.07
m/z

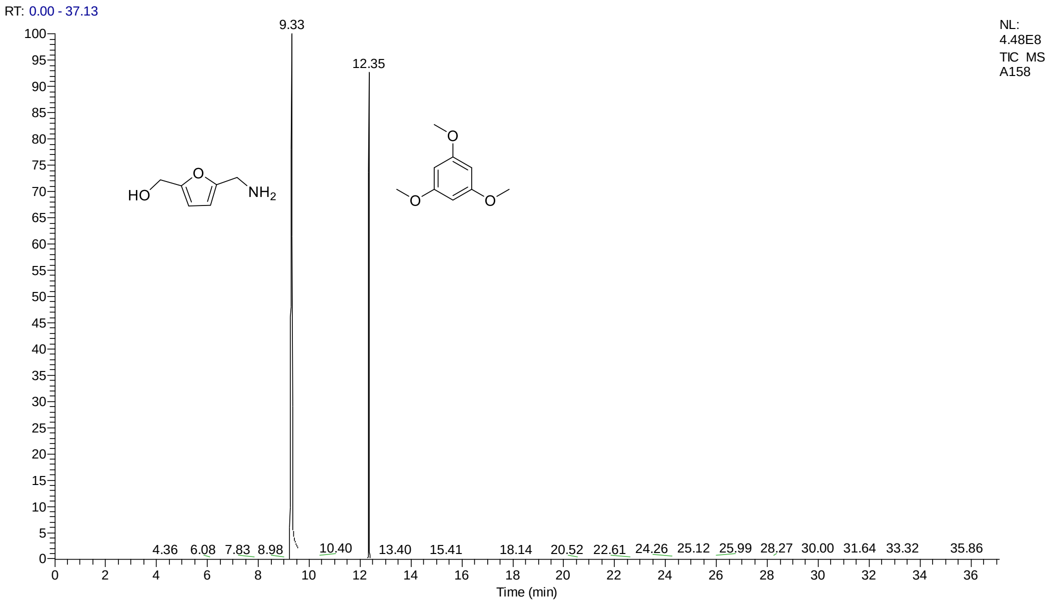
A158



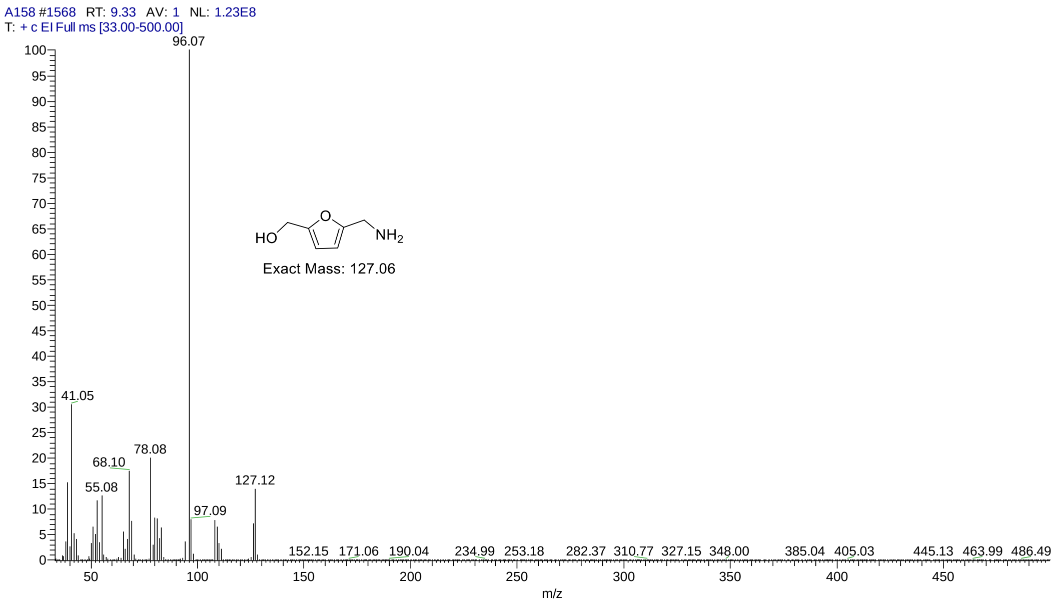
O
HO
15
10
5
0
f2 (ppm)



O
NH2
HO
15
10
5
0
f2 (ppm)

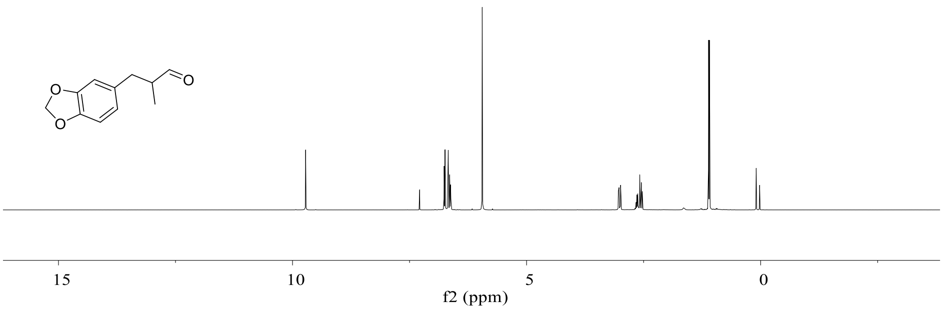


RT: 0.00 - 37.13
NL:
4.48E8
TIC MS
A158
9.33
12.35
HO
O
NH2
O
4.36 6.08 7.83 8.98 10.40 13.40 15.41 18.14 20.52 22.61 24.26 25.12 25.99 28.27 30.00 31.64 33.32 35.86
Time (min)

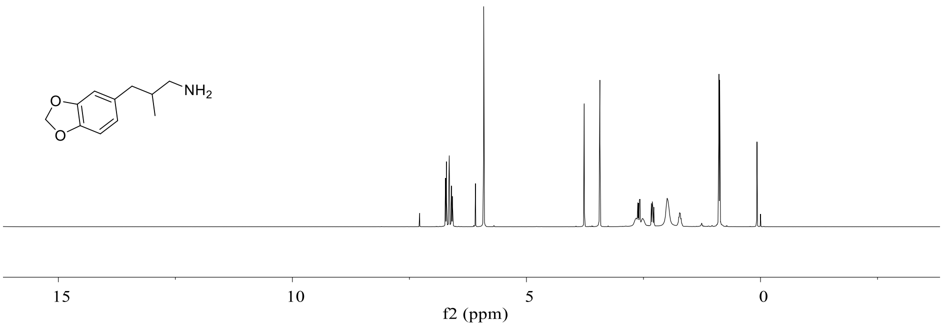


A158 #1568 RT: 9.33 AV: 1 NL: 1.23E8
T: + c EI Full ms [33.00-500.00]
96.07
HO
O
NH2
Exact Mass: 127.06
41.05
78.08
68.10
55.08
127.12
97.09
152.15 171.06 190.04 234.99 253.18 282.37 310.77 327.15 348.00 385.04 405.03 445.13 463.99 486.49
m/z

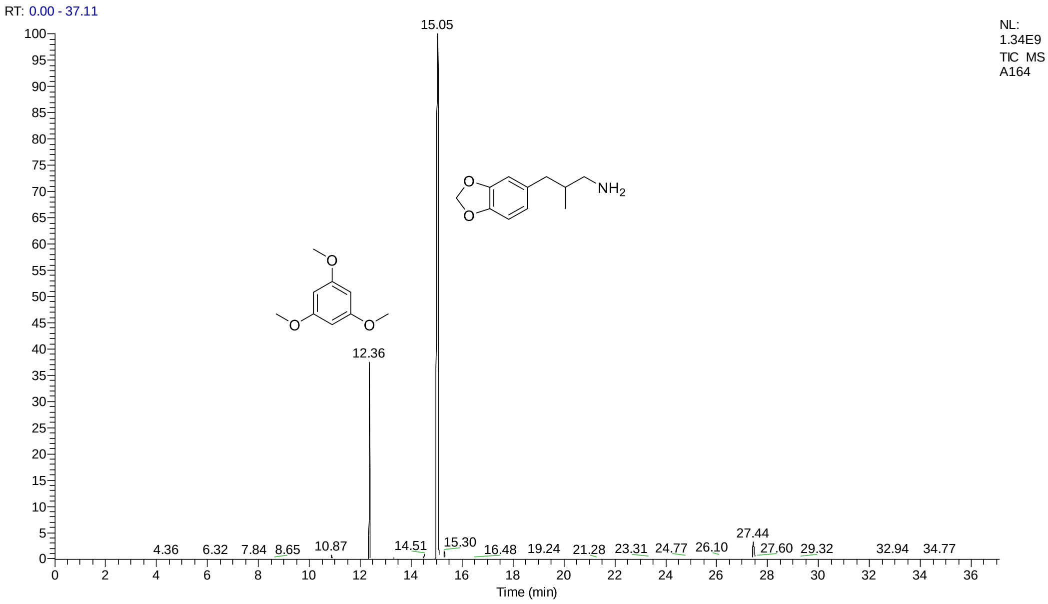
A164



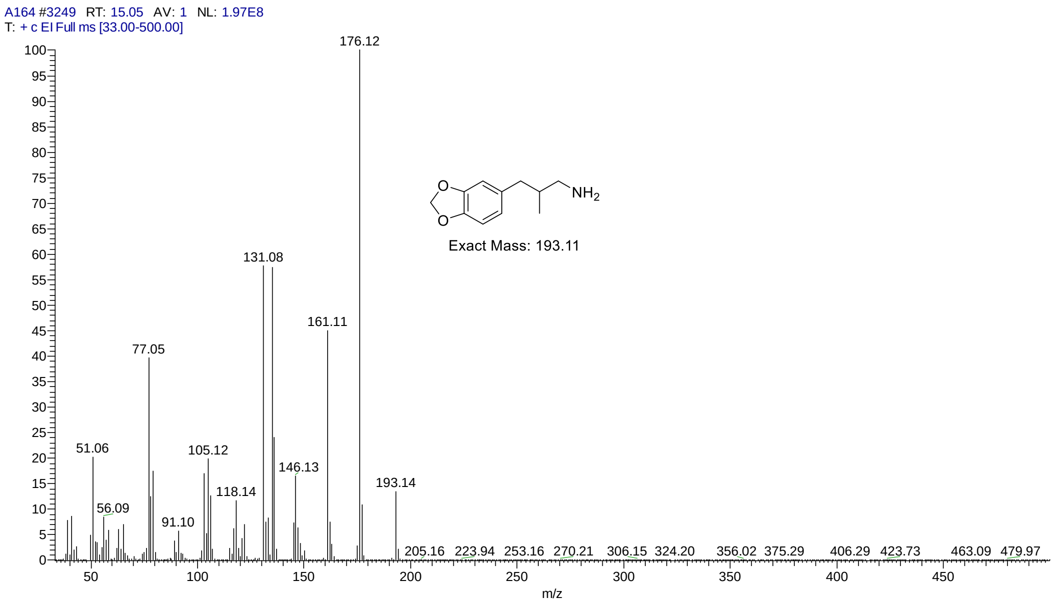
O
O
O
15
10
5
0
f2 (ppm)



O
O
NH2
15
10
5
0
f2 (ppm)

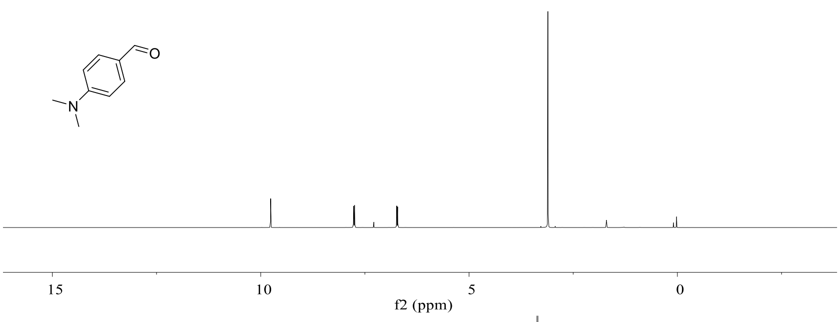


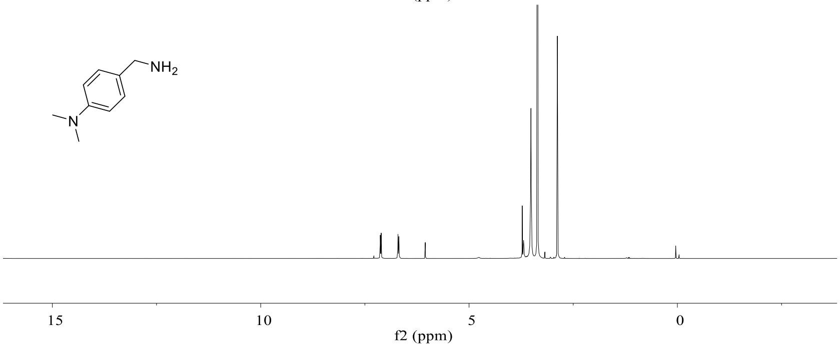
RT: 0.00 - 37.11
NL:
1.34E9
TIC MS
A164
15.05
12.36
4.36 6.32 7.84 8.65 10.87 14.51 15.30 16.48 19.24 21.28 23.31 24.77 26.10 27.44 27.60 29.32 32.94 34.77
NH2
O
Time (min)

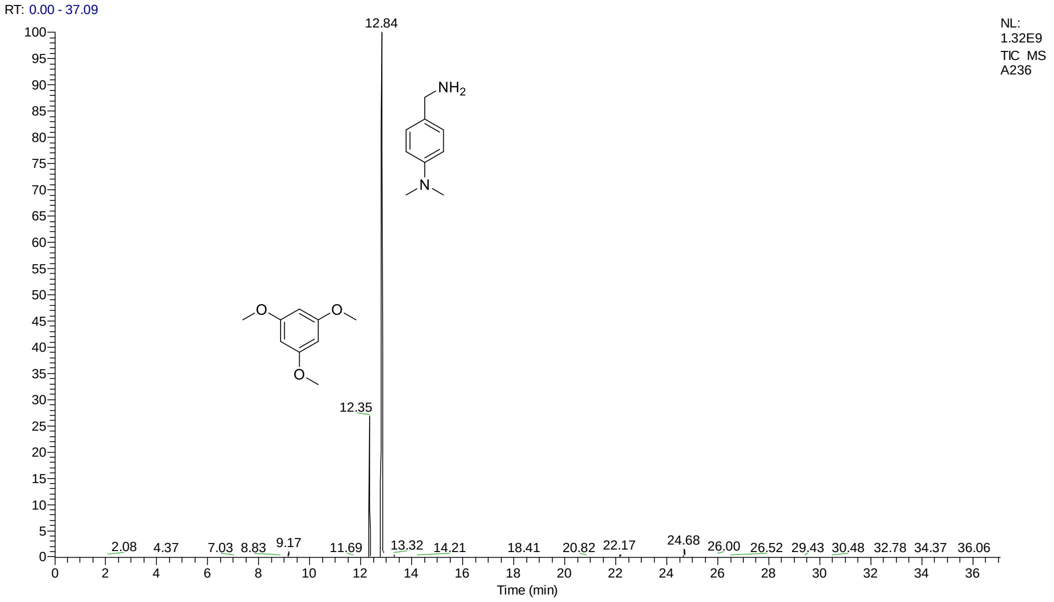


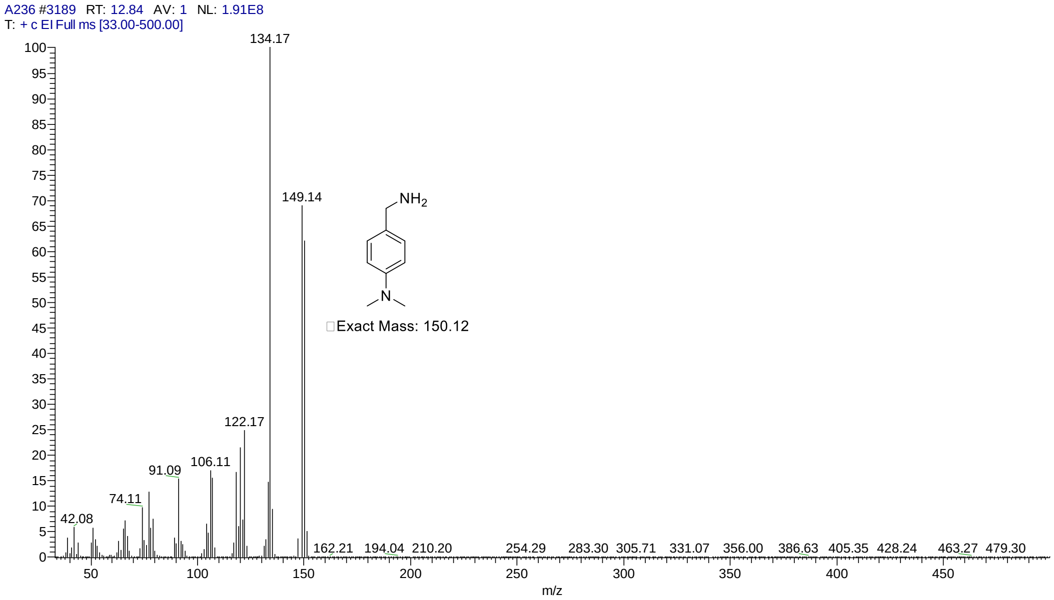
A164 #3249 RT: 15.05 AV: 1 NL: 1.97E8
T: + c EI Full ms [33.00-500.00]
176.12
131.08
161.11
77.05
51.06
105.12
146.13
118.14
193.14
56.09
91.10
205.16 223.94 253.16 270.21 306.15 324.20 356.02 375.29 406.29 423.73 463.09 479.97
NH2
Exact Mass: 193.11
m/z

A236

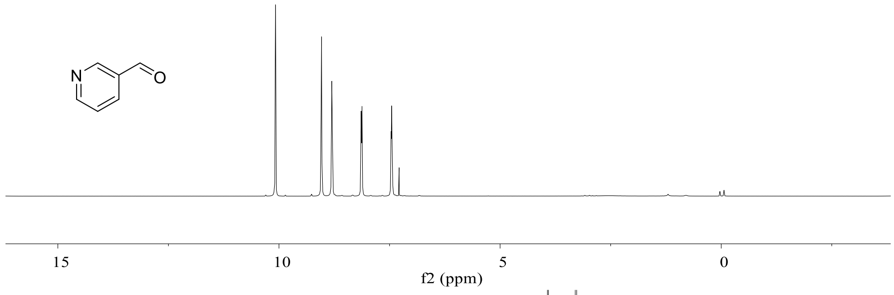

O
N
15
10
5
0
f2 (ppm)

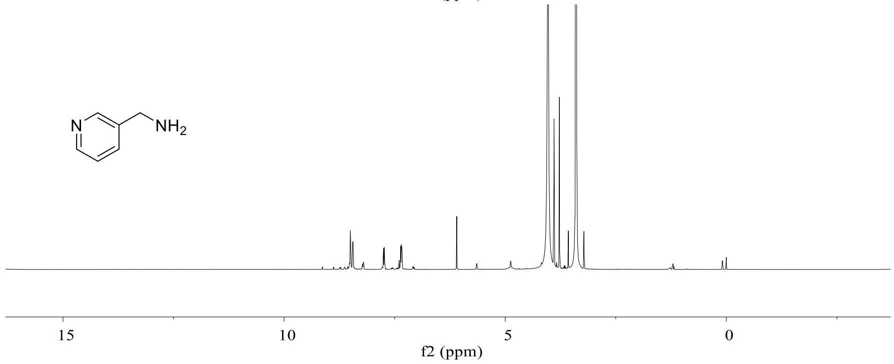

NH2
N
15
10
5
0
f2 (ppm)


RT: 0.00 - 37.09
NL: 1.32E9 TIC MS A236
12.84
12.35
2.08 4.37 7.03 8.83 9.17 11.69 13.32 14.21 18.41 20.82 22.17 24.68 26.00 26.52 29.43 30.48 32.78 34.37 36.06
NH2
N
O
O
O
Time (min)

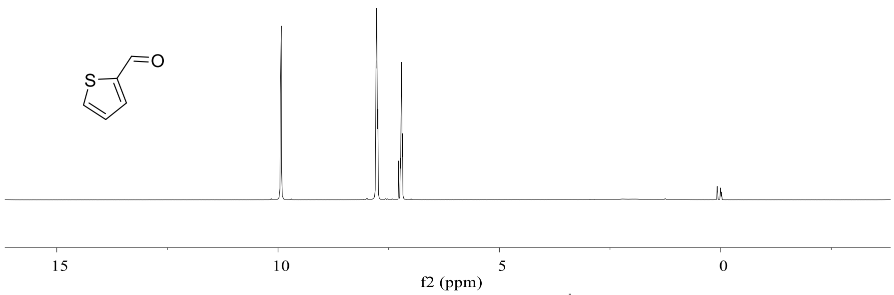

A236 #3189 RT: 12.84 AV: 1 NL: 1.91E8
T: + c EI Full ms [33.00-500.00]
134.17
149.14
122.17
106.11
91.09
74.11
42.08
162.21 194.04 210.20 254.29 283.30 305.71 331.07 356.00 386.63 405.35 428.24 463.27 479.30
NH2
N
Exact Mass: 150.12
m/z

A279

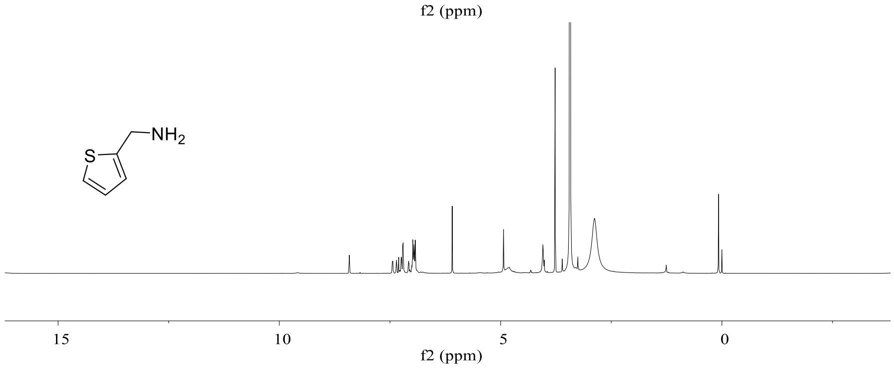




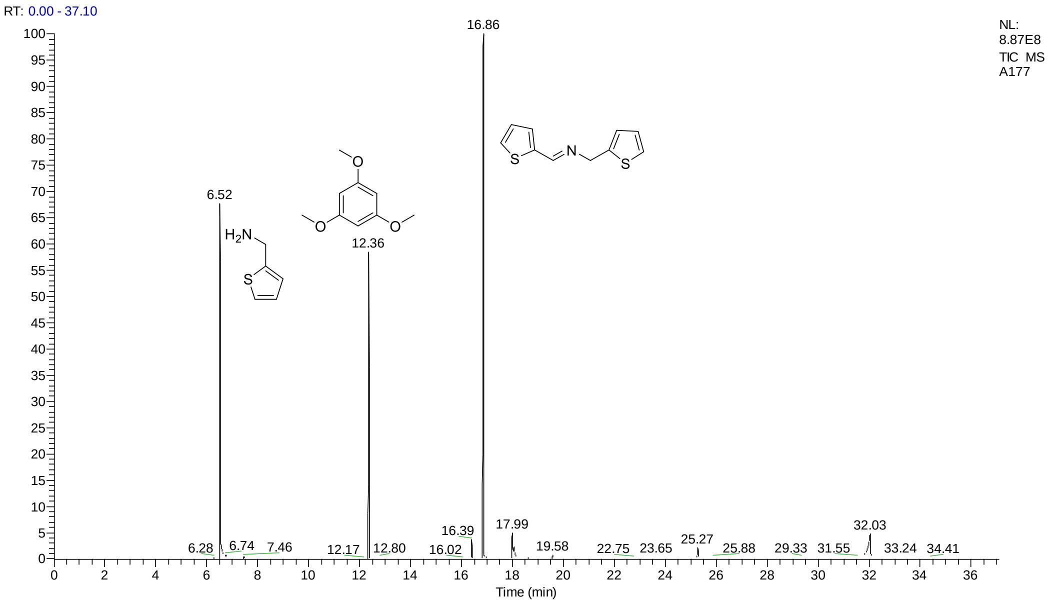
A177



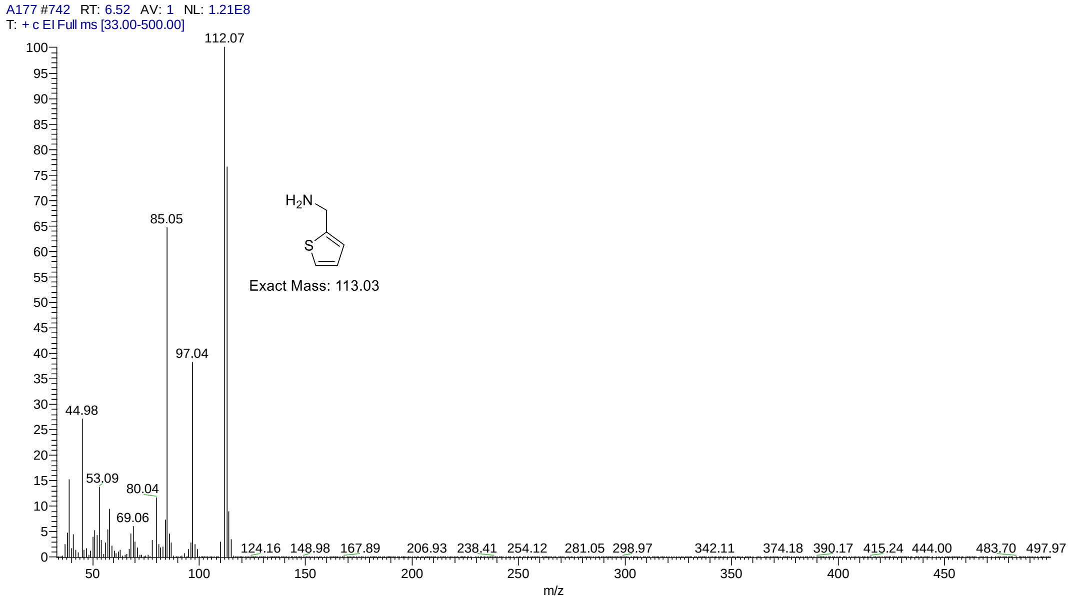
S
O
15
10
5
0
f2 (ppm)

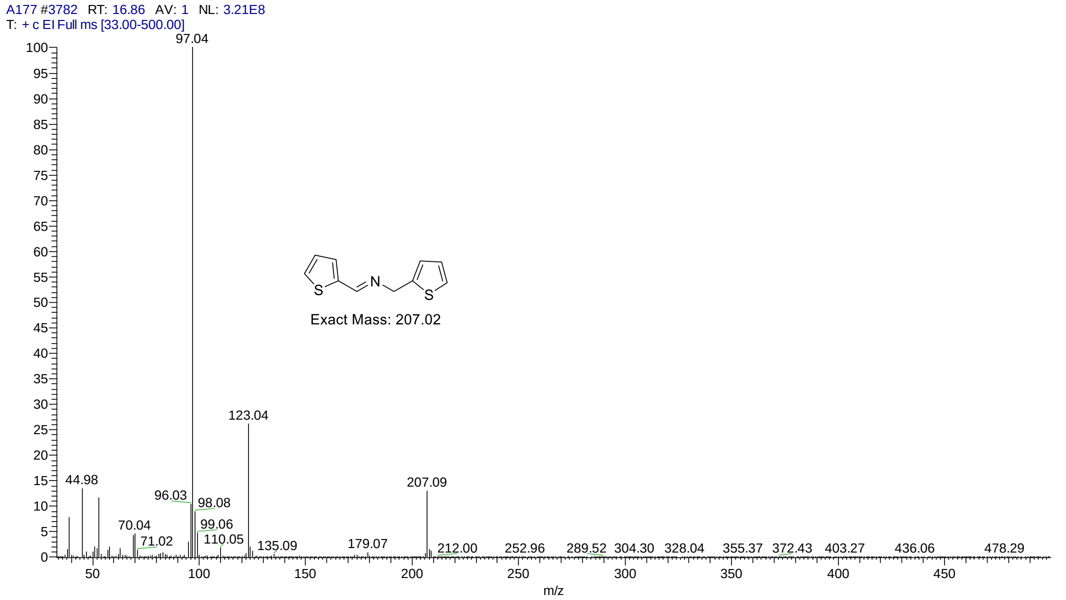


S
NH2
15
10
5
0
f2 (ppm)

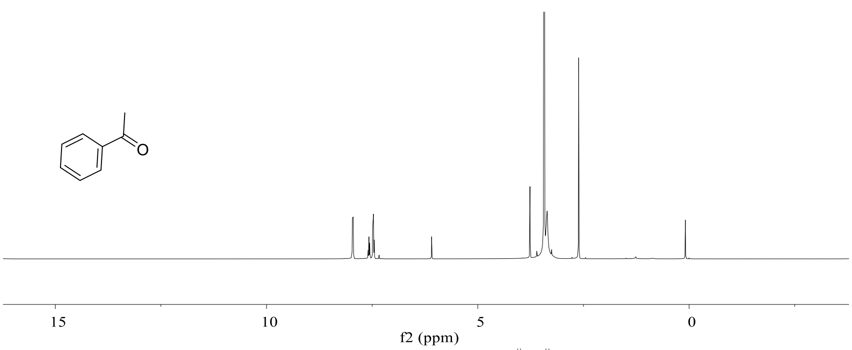


RT: 0.00 - 37.10
NL:
8.87E8
TIC MS
A177
16.86
6.52
12.36
H2N
S
O
N
17.99
16.39
32.03
25.27
6.28
6.74
7.46
12.17
12.80
16.02
19.58
22.75
23.65
25.88
29.33
31.55
33.24
34.41
Time (min)

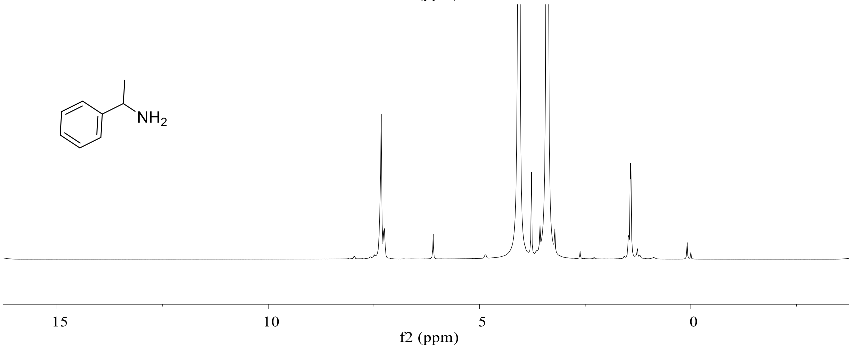

A177 #742 RT: 6.52 AV: 1 NL: 1.21E8
T: + c EI Full ms [33.00-500.00]
112.07
85.05
97.04
44.98
53.09
80.04
69.06
124.16
148.98
167.89
206.93
238.41
254.12
281.05
298.97
342.11
374.18
390.17
415.24
444.00
483.70
497.97
H2N
S
Exact Mass: 113.03
m/z


A177 #3782 RT: 16.86 AV: 1 NL: 3.21E8
T: + c EI Full ms [33.00-500.00]
97.04
123.04
207.09
44.98
96.03
98.08
99.06
110.05
70.04
71.02
135.09
179.07
212.00
252.96
289.52
304.30
328.04
355.37
372.43
403.27
436.06
478.29
N
S
S
Exact Mass: 207.02
m/z

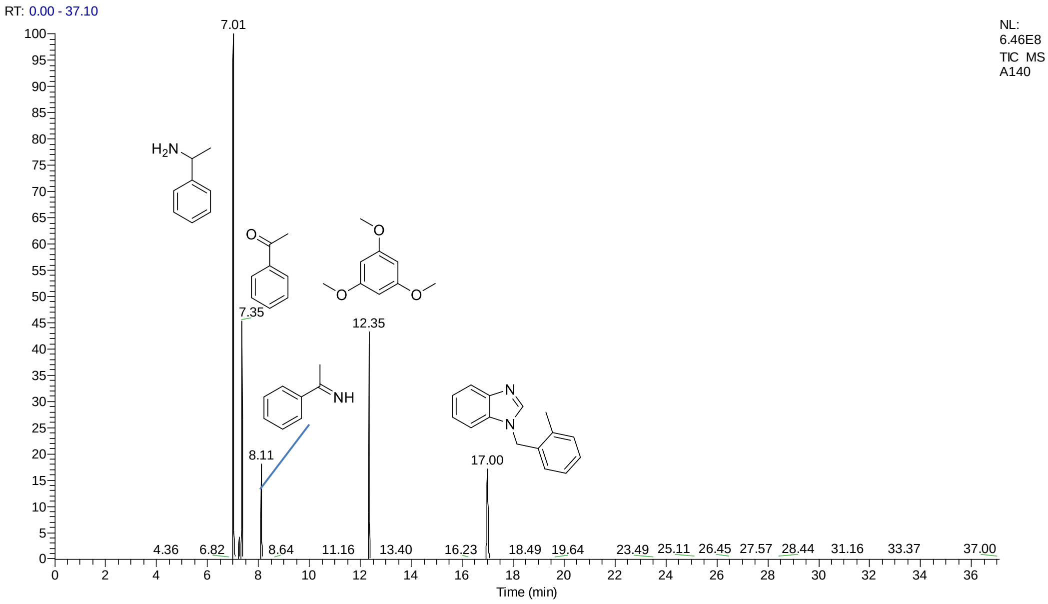
A140



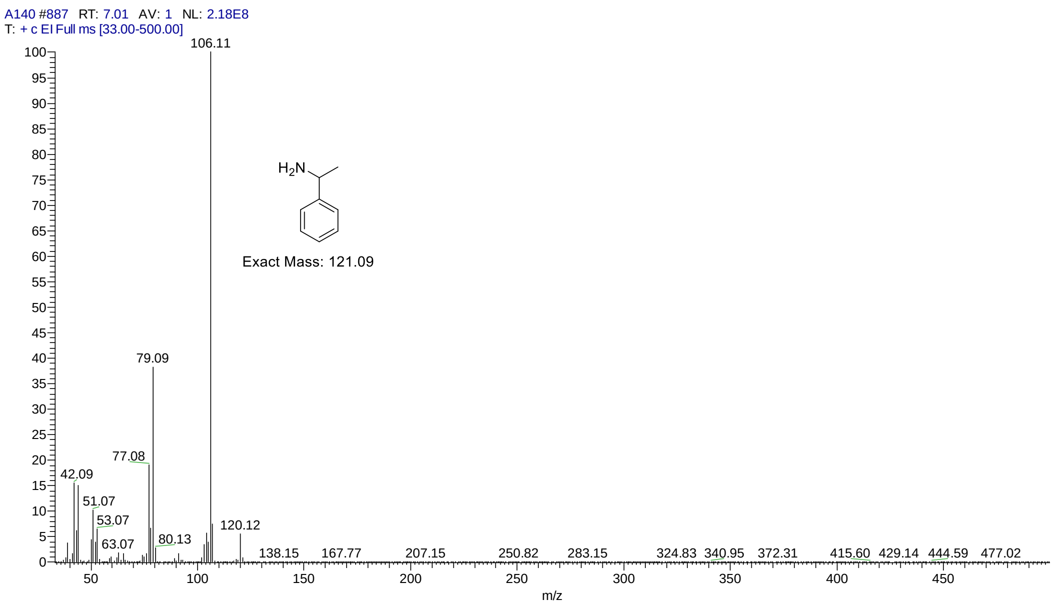
O
15
10
5
0
f2 (ppm)



NH2
15
10
5
0
f2 (ppm)

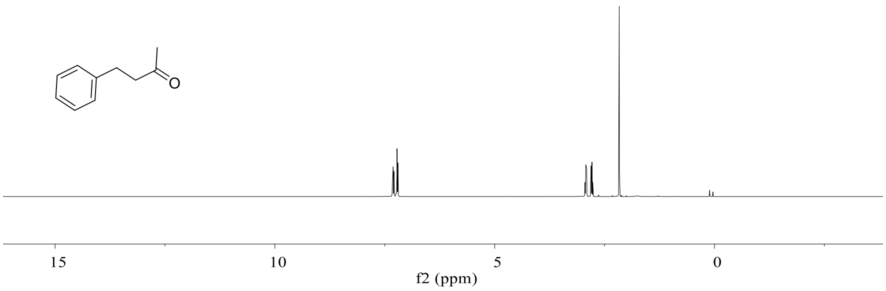


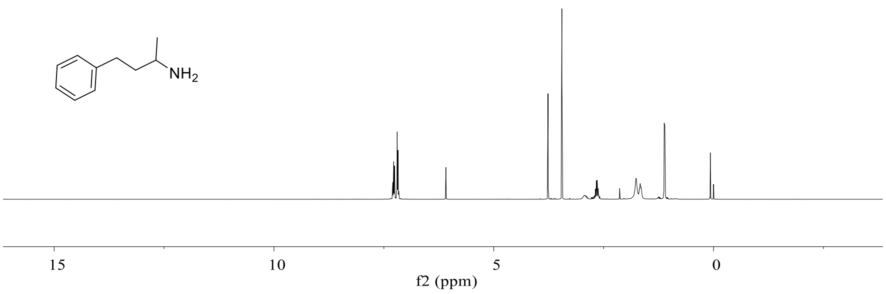
RT: 0.00 - 37.10
NL: 6.46E8 TIC MS A140
7.01
7.35
8.11
12.35
17.00
4.36
6.82
8.64
11.16
13.40
16.23
18.49
19.64
23.49
25.11
26.45
27.57
28.44
31.16
33.37
37.00
H2N
O
NH
N
Time (min)



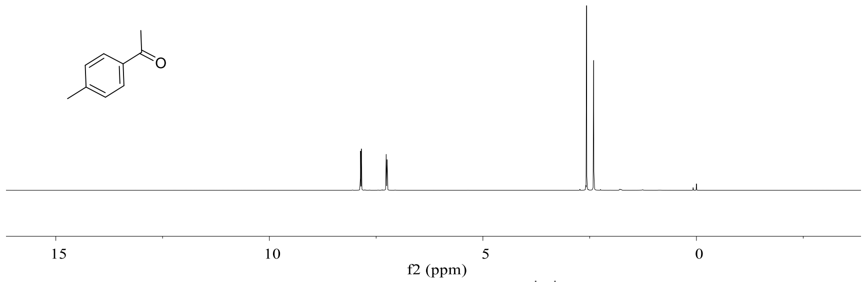
A140 #887 RT: 7.01 AV: 1 NL: 2.18E8
T: + c EI Full ms [33.00-500.00]
106.11
79.09
77.08
42.09
51.07
53.07
63.07
80.13
120.12
138.15
167.77
207.15
250.82
283.15
324.83
340.95
372.31
415.60
429.14
444.59
477.02
H2N
Exact Mass: 121.09
m/z

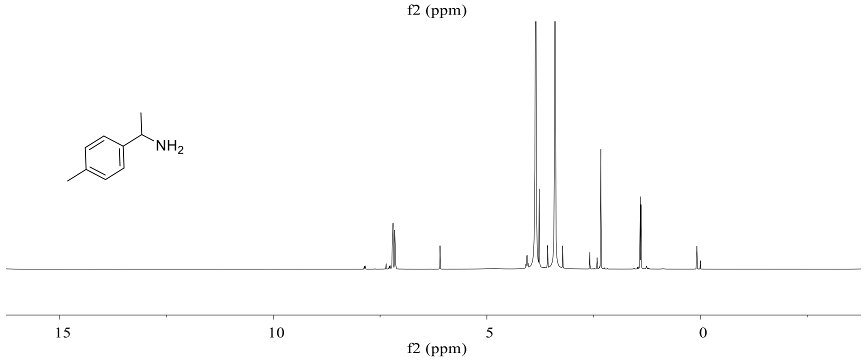
A144



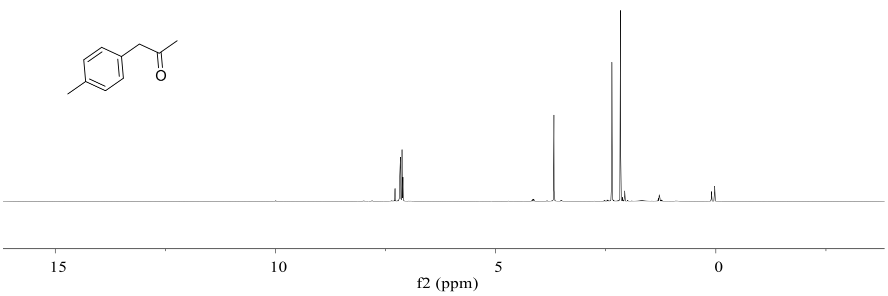


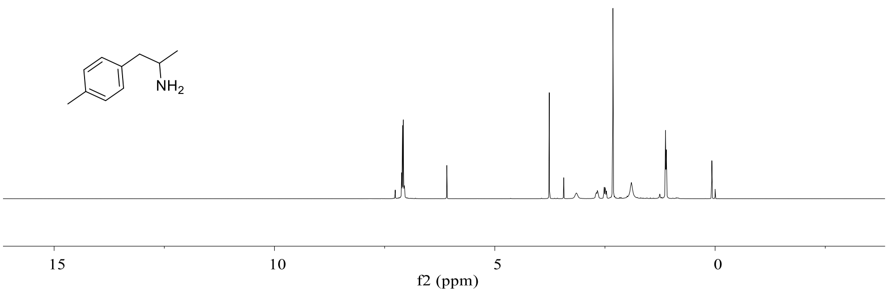
A222



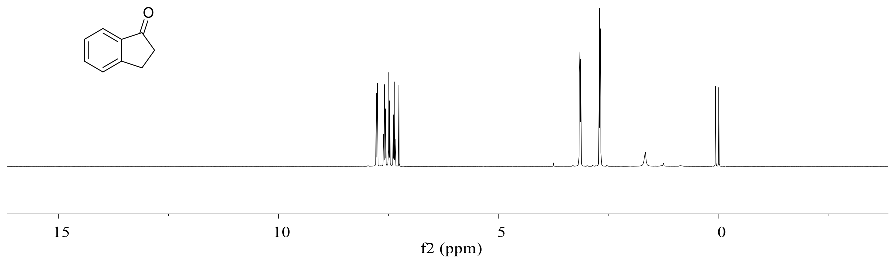


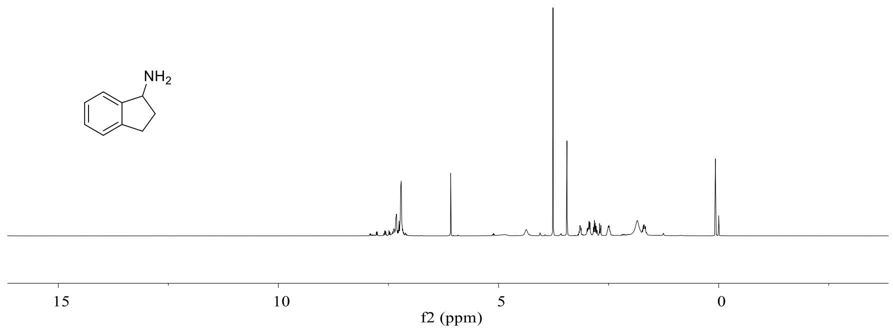
A187



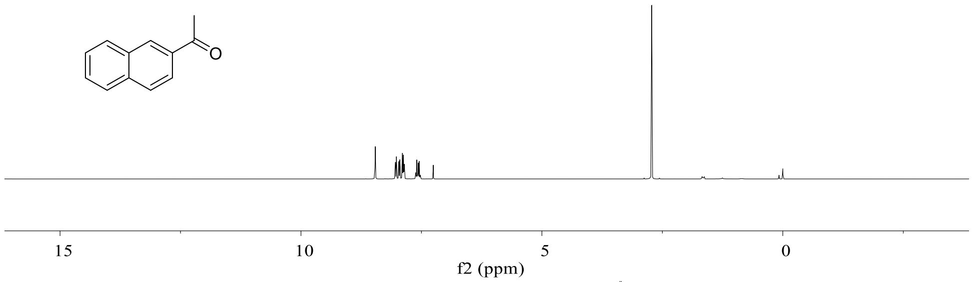


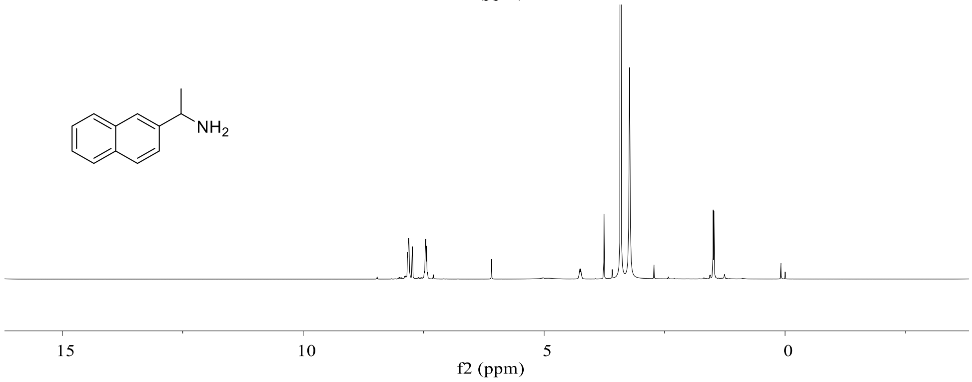
A191

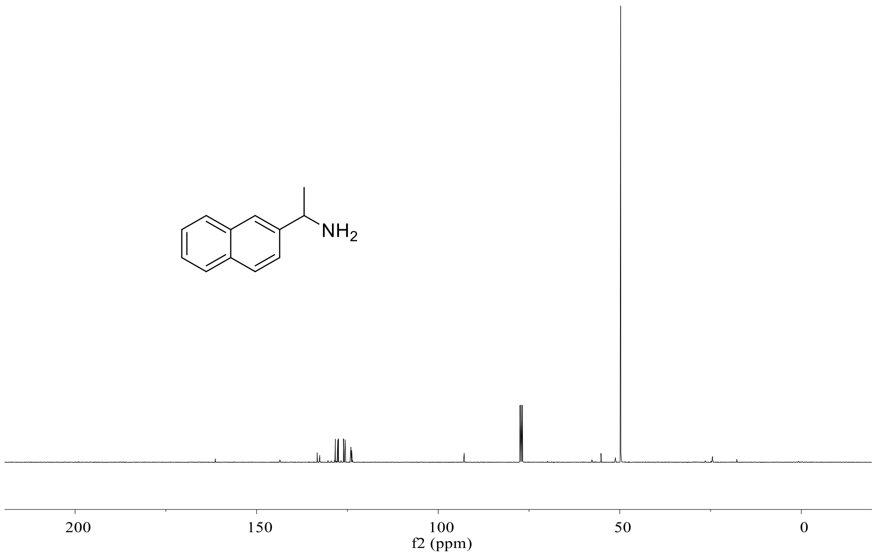




A204

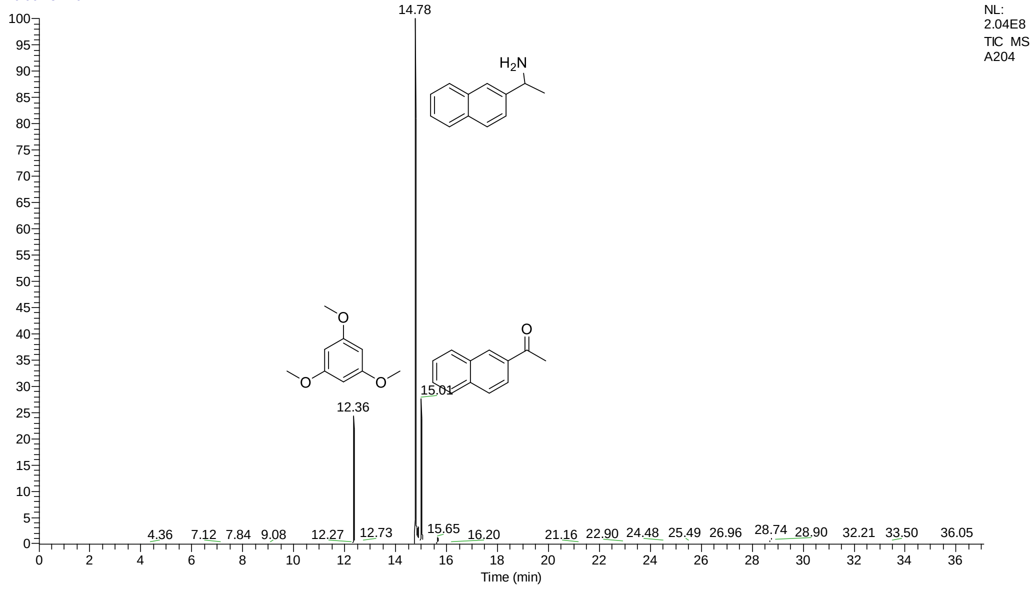




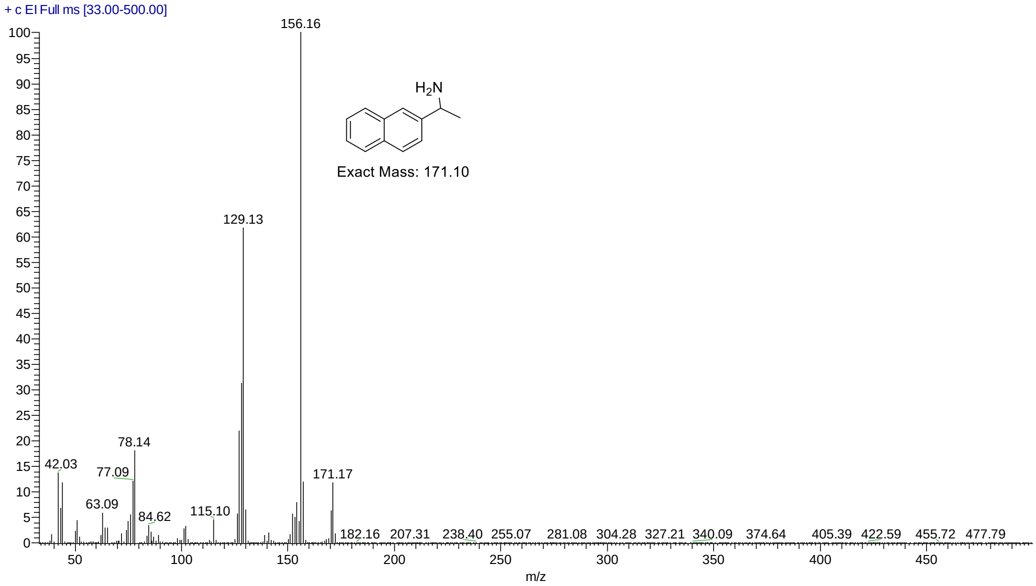


^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (m, 3H), 7.73 (s, 1 H), 7.46 (m, 3H), 4.26 (m, 1 H), 1.48 (d, 3H). ^{13}C NMR (125MHz, $CDCl_3$) δ, 133.34, 132.62, 128.33, 127.66, 127.49, 126.06, 125.62, 124.05, 123.81, 51.23, 24.48.

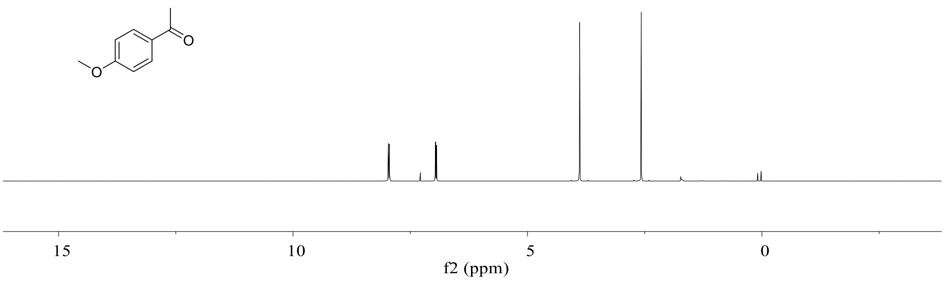
RT: 0.00 - 37.10

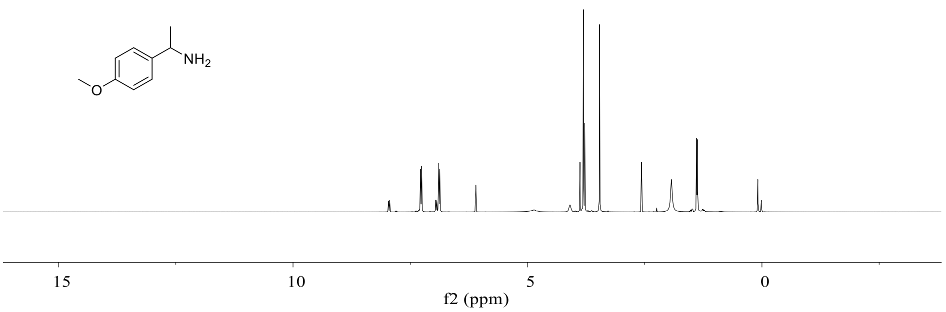


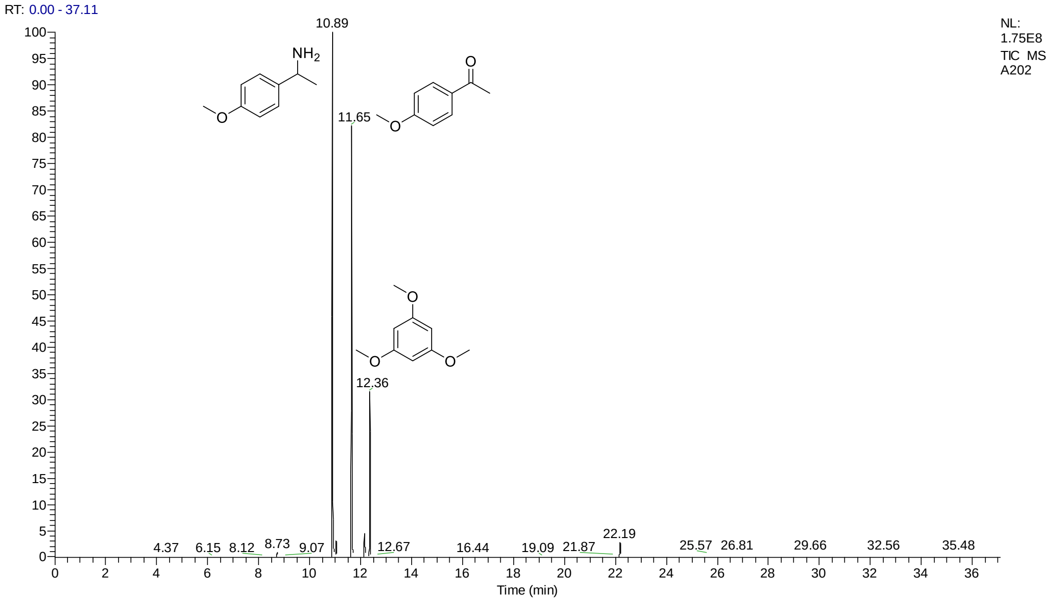
A204 #3170 RT: 14.78 AV: 1 NL: 4.32E7
T: + c EI Full ms [33.00-500.00]

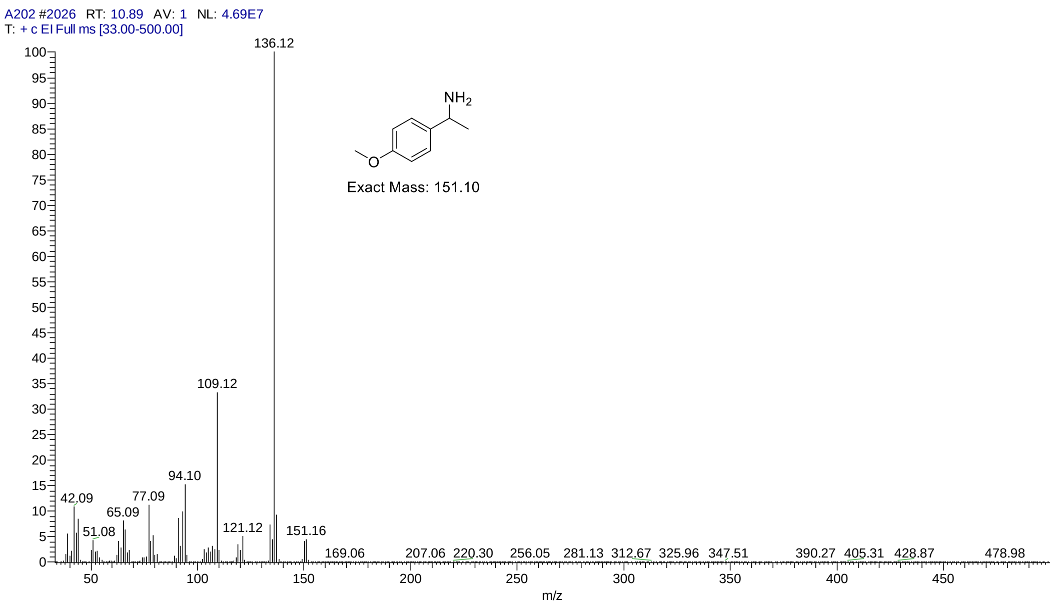


A168 (A202 GC-MS)

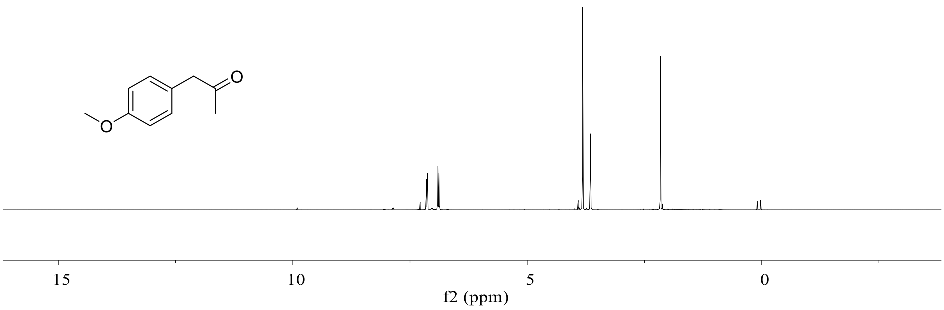




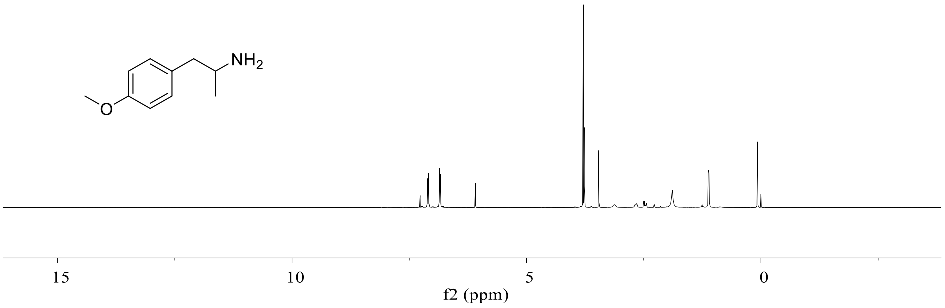




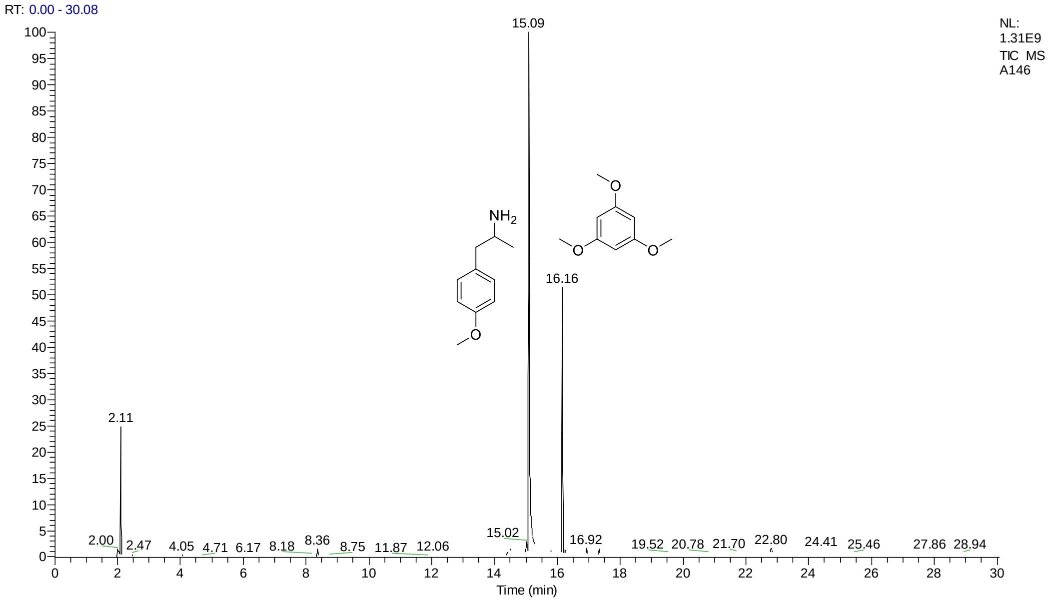
A146



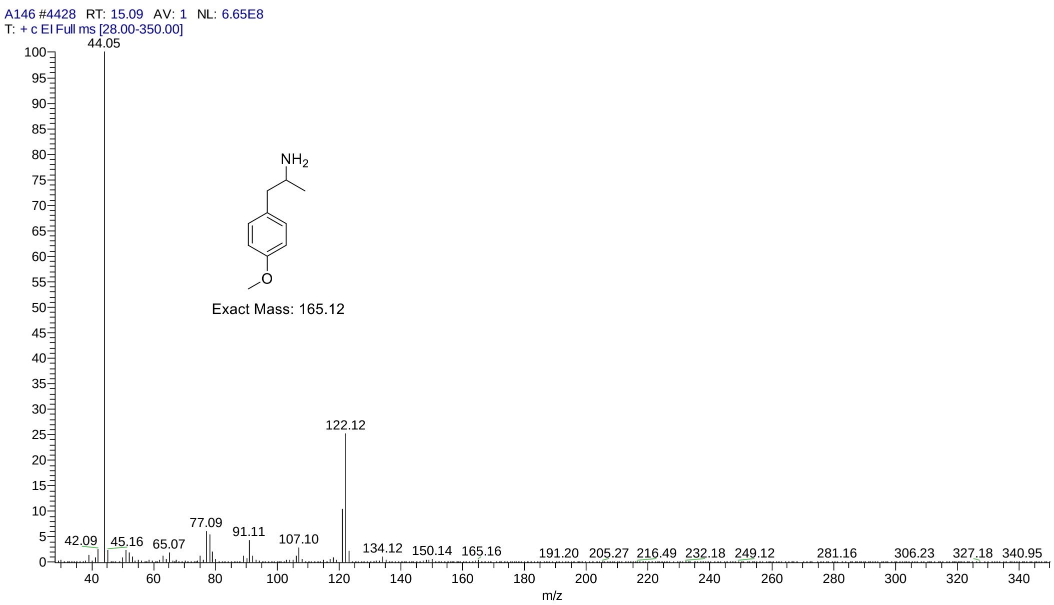
O
O
15
10
5
0
f2 (ppm)



NH2
O
15
10
5
0
f2 (ppm)

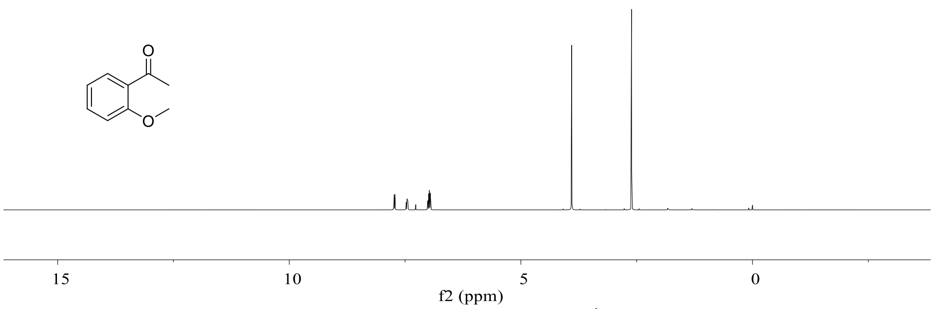


RT: 0.00 - 30.08
NL: 1.31E9 TIC MS A146
15.09
16.16
2.11
2.00 2.47 4.05 4.71 6.17 8.18 8.36 8.75 11.87 12.06 15.02 16.92 19.52 20.78 21.70 22.80 24.41 25.46 27.86 28.94
NH2
O
Time (min)

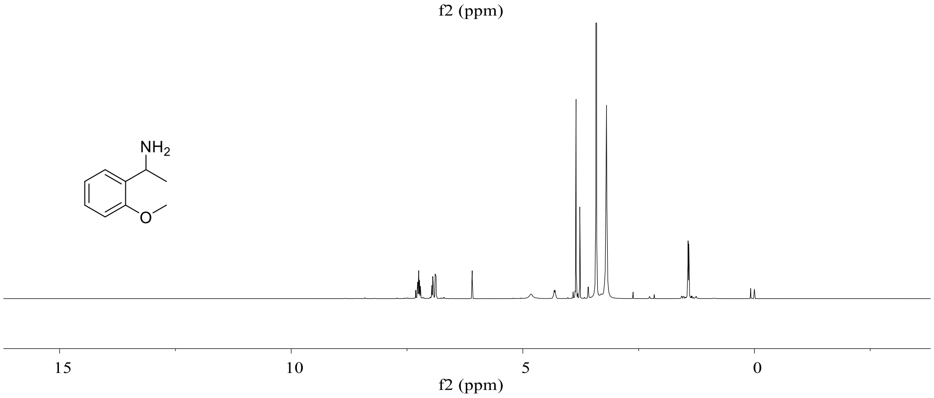


A146 #4428 RT: 15.09 AV: 1 NL: 6.65E8
T: + c EI Full ms [28.00-350.00]
44.05
NH2
O
Exact Mass: 165.12
122.12
42.09 45.16 65.07 77.09 91.11 107.10 134.12 150.14 165.16 191.20 205.27 216.49 232.18 249.12 281.16 306.23 327.18 340.95
m/z

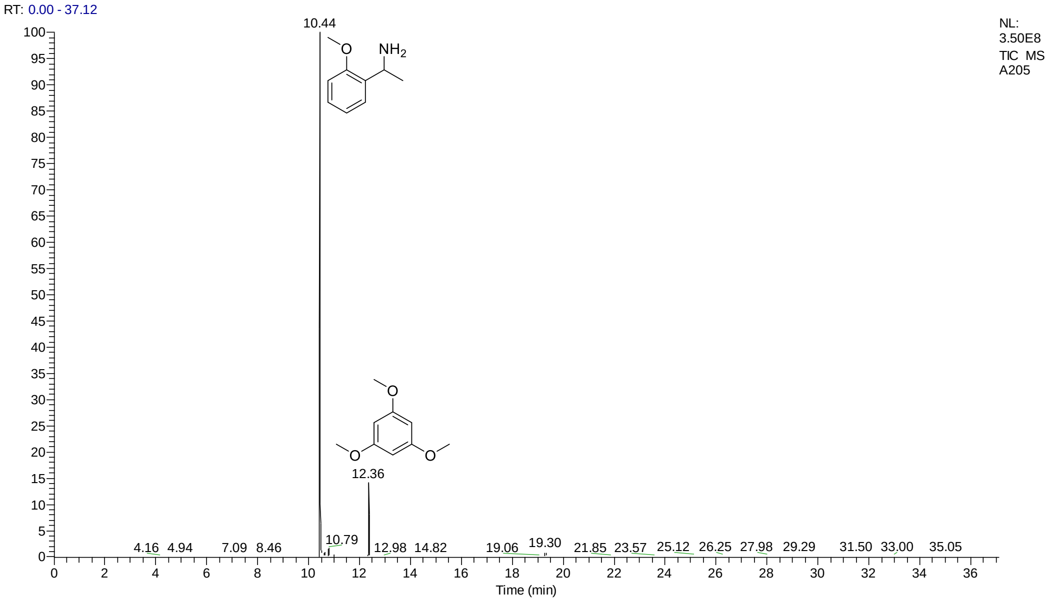
A205



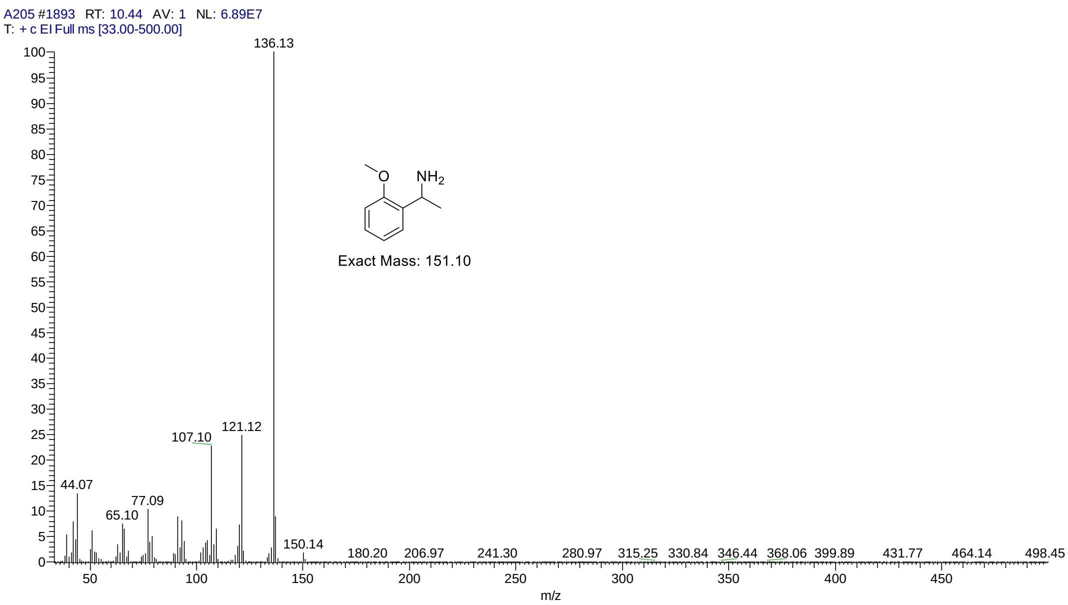
O
15
10
5
0
f2 (ppm)



NH2
O
15
10
5
0
f2 (ppm)

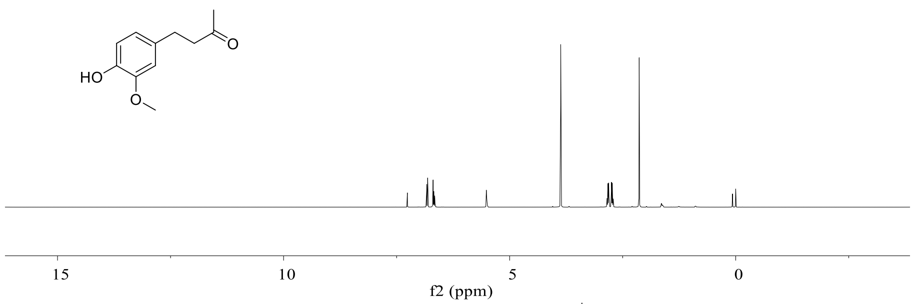


RT: 0.00 - 37.12
NL:
3.50E8
TIC MS
A205
10.44
12.36
4.16 4.94 7.09 8.46
10.79
12.98 14.82
19.06 19.30
21.85 23.57 25.12 26.25 27.98 29.29
31.50 33.00 35.05
Time (min)

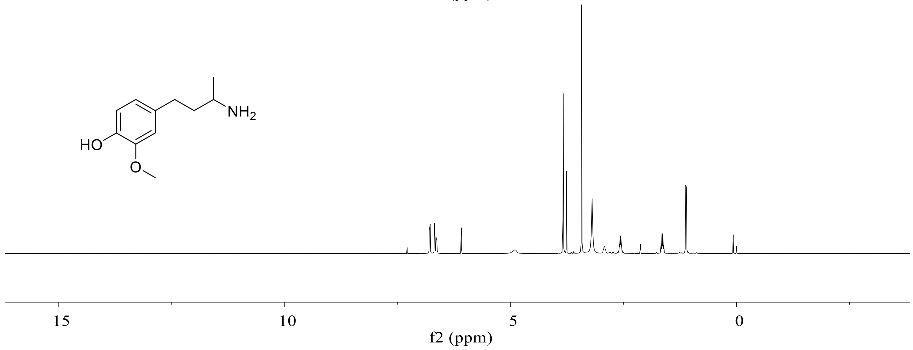


A205 #1893 RT: 10.44 AV: 1 NL: 6.89E7
T: + c EI Full ms [33.00-500.00]
136.13
Exact Mass: 151.10
107.10
121.12
44.07
77.09
65.10
150.14
180.20 206.97 241.30 280.97 315.25 330.84 346.44 368.06 399.89 431.77 464.14 498.45
m/z

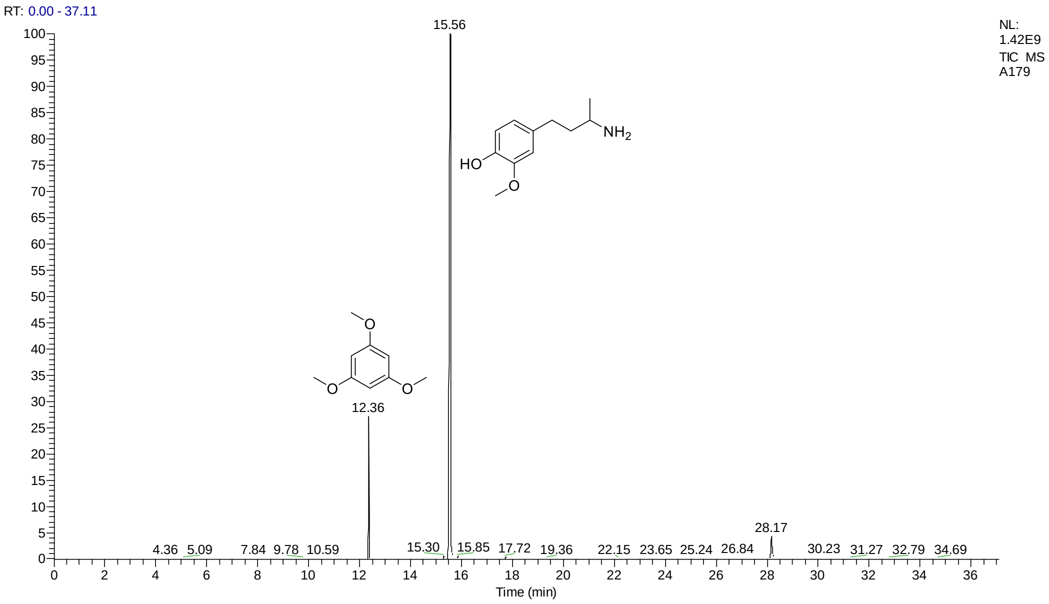
A179



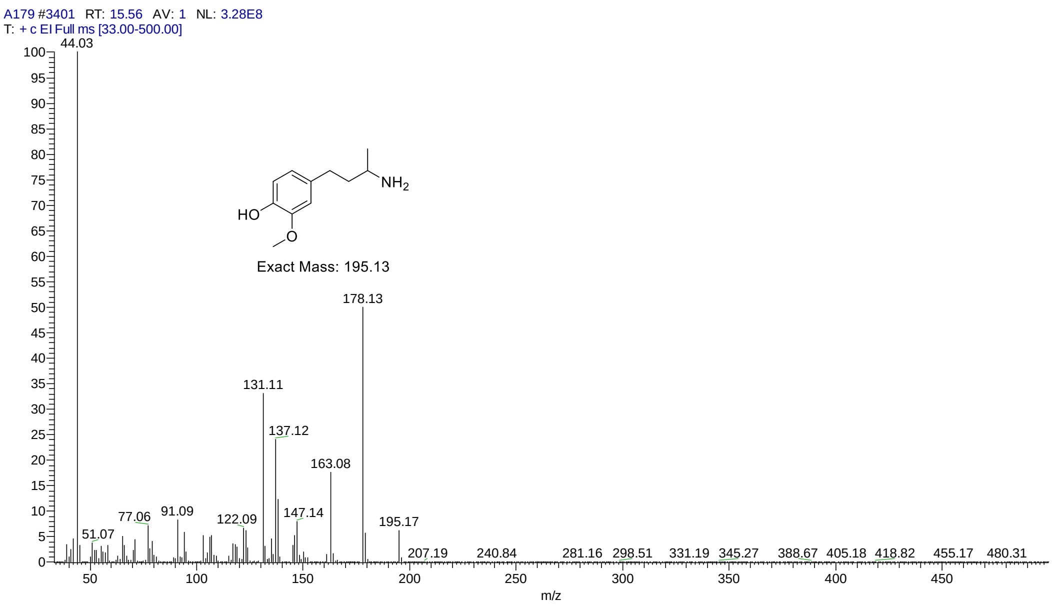
HO
O
15
10
5
0
f2 (ppm)



NH2
HO
O
15
10
5
0
f2 (ppm)

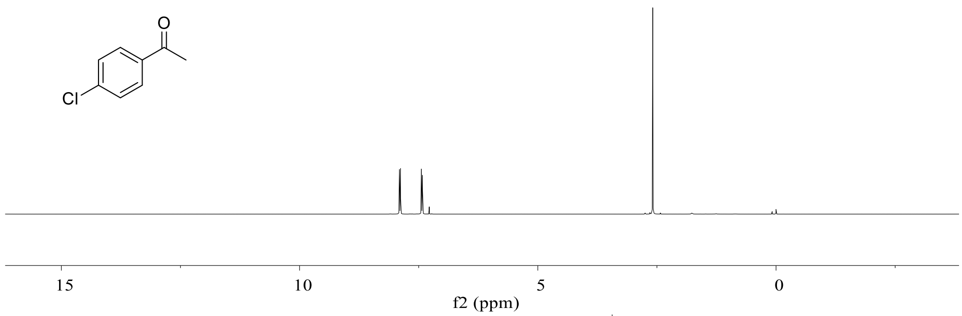


RT: 0.00 - 37.11
NL: 1.42E9 TIC MS A179
15.56
12.36
28.17
4.36 5.09 7.84 9.78 10.59 15.30 15.85 17.72 19.36 22.15 23.65 25.24 26.84 30.23 31.27 32.79 34.69
Time (min)

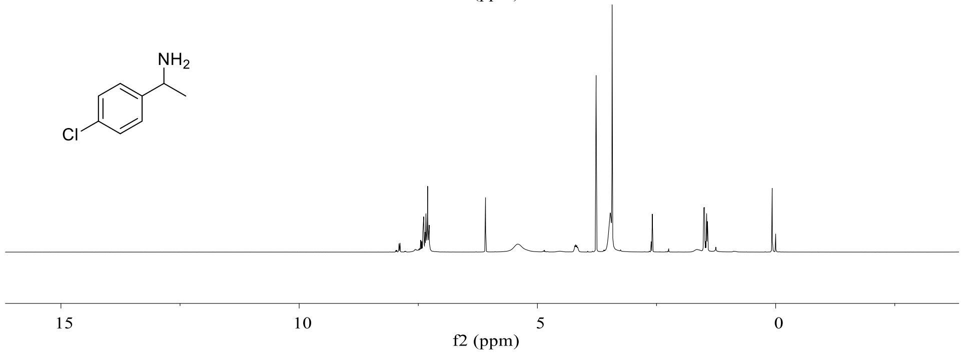


A179 #3401 RT: 15.56 AV: 1 NL: 3.28E8
T: + c EI Full ms [33.00-500.00]
44.03
Exact Mass: 195.13
178.13
131.11
137.12
163.08
147.14
122.09
91.09
77.06
51.07
195.17
207.19 240.84 281.16 298.51 331.19 345.27 388.67 405.18 418.82 455.17 480.31
m/z

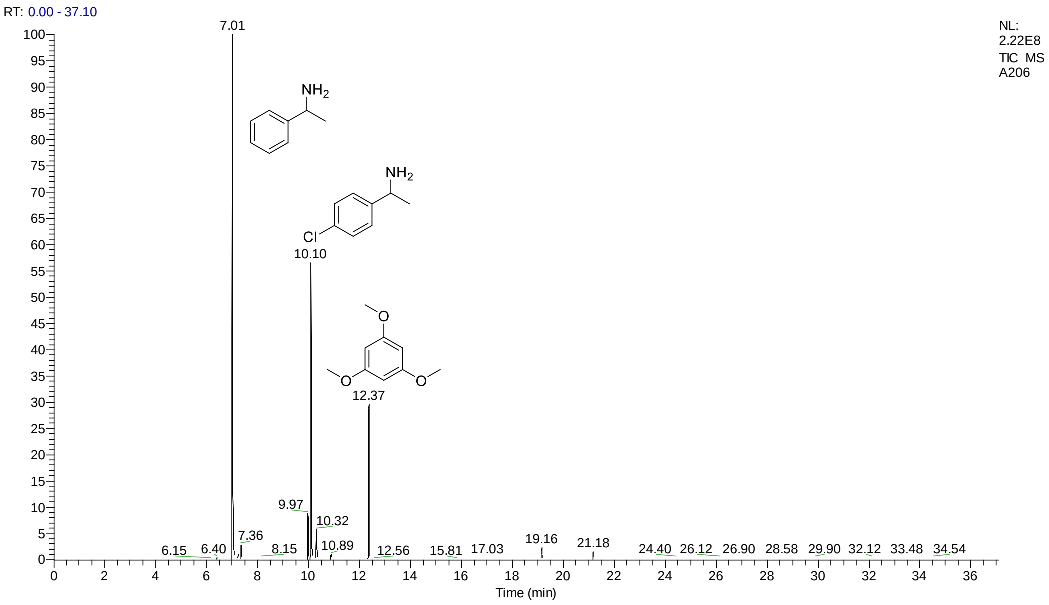
A206



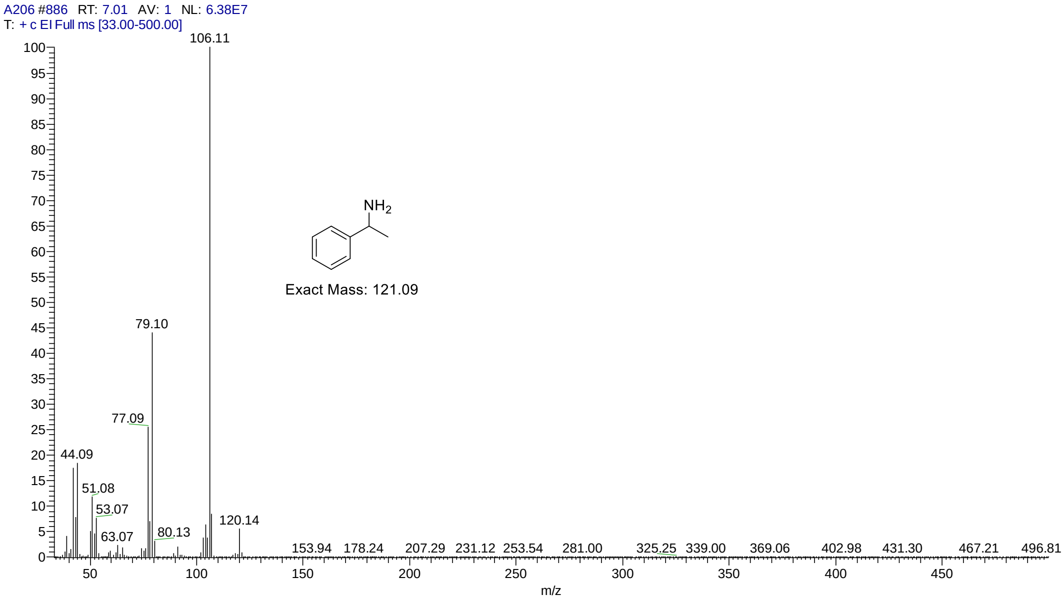
O
Cl
15
10
5
0
f2 (ppm)



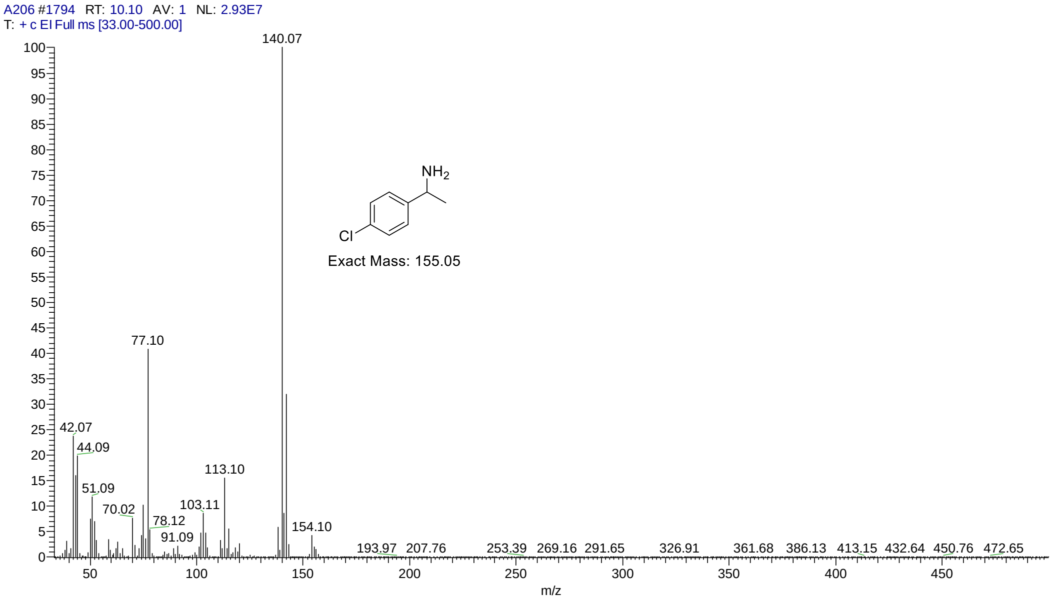
NH2
Cl
15
10
5
0
f2 (ppm)



RT: 0.00 - 37.10
NL:
2.22E8
TIC MS
A206
7.01
NH2
NH2
Cl
10.10
O
O
O
12.37
9.97
10.32
7.36
6.15
6.40
8.15
10.89
12.56
15.81
17.03
19.16
21.18
24.40
26.12
26.90
28.58
29.90
32.12
33.48
34.54
Time (min)

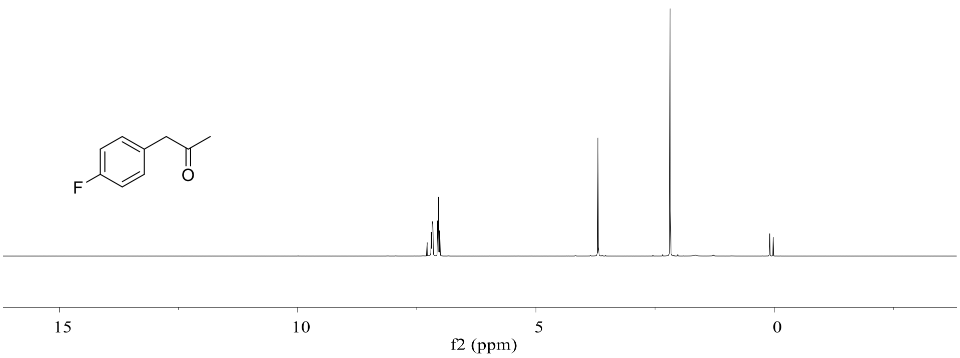


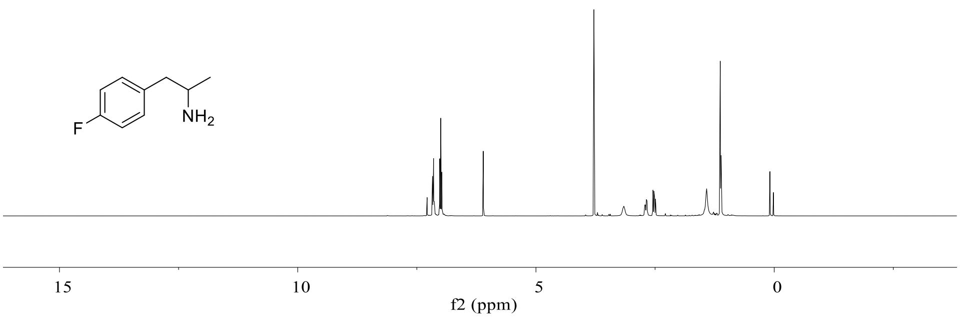
A206 #886 RT: 7.01 AV: 1 NL: 6.38E7
T: + c EI Full ms [33.00-500.00]
106.11
NH2
Exact Mass: 121.09
79.10
77.09
44.09
51.08
53.07
63.07
80.13
120.14
153.94
178.24
207.29
231.12
253.54
281.00
325.25
339.00
369.06
402.98
431.30
467.21
496.81
m/z

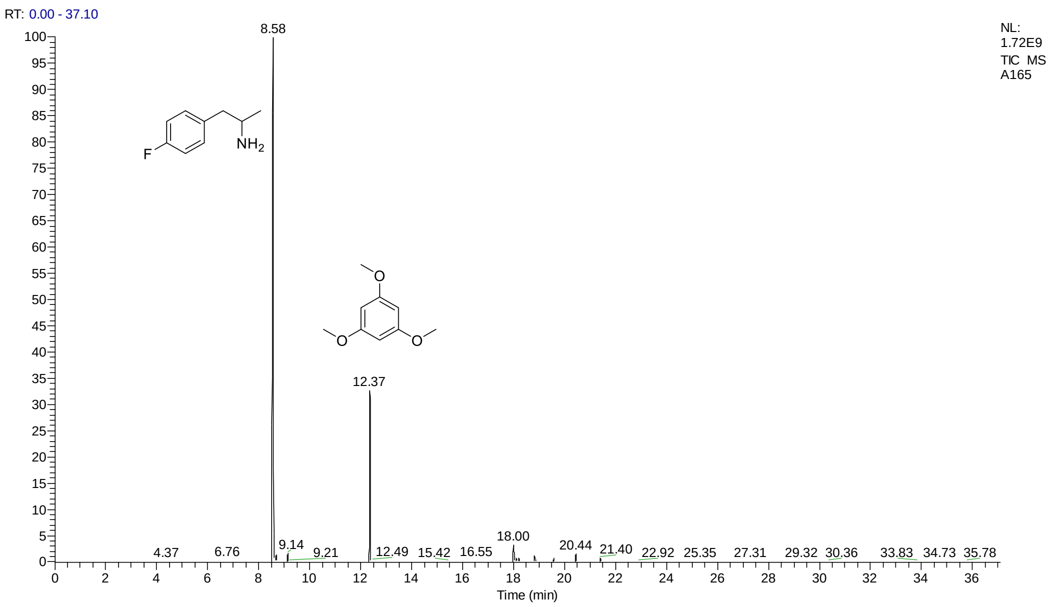


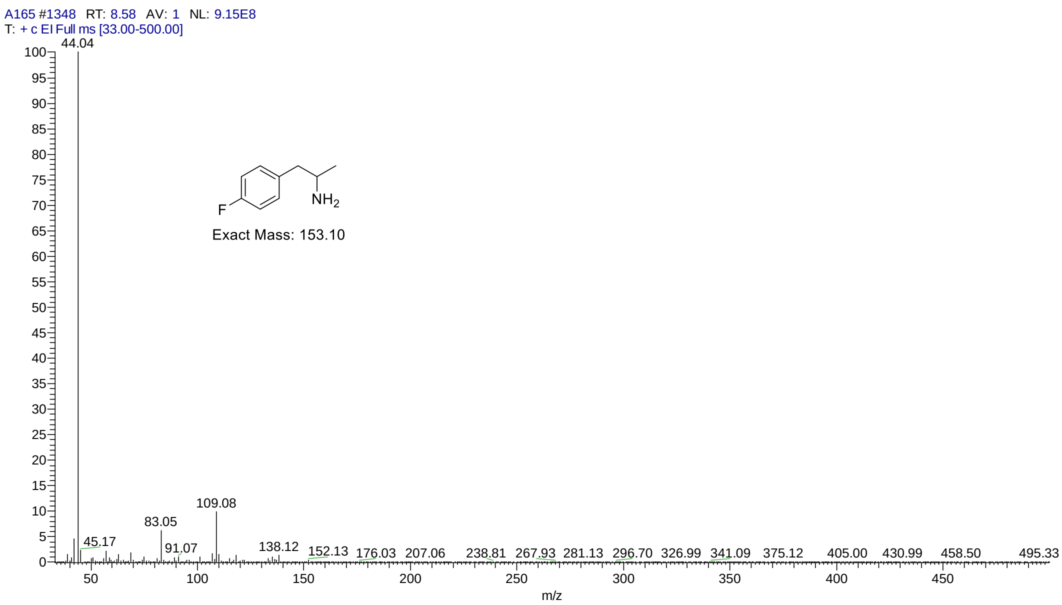
A206 #1794 RT: 10.10 AV: 1 NL: 2.93E7
T: + c EI Full ms [33.00-500.00]
140.07
NH2
Cl
Exact Mass: 155.05
77.10
42.07
44.09
51.09
70.02
78.12
91.09
103.11
113.10
154.10
193.97
207.76
253.39
269.16
291.65
326.91
361.68
386.13
413.15
432.64
450.76
472.65
m/z

A165

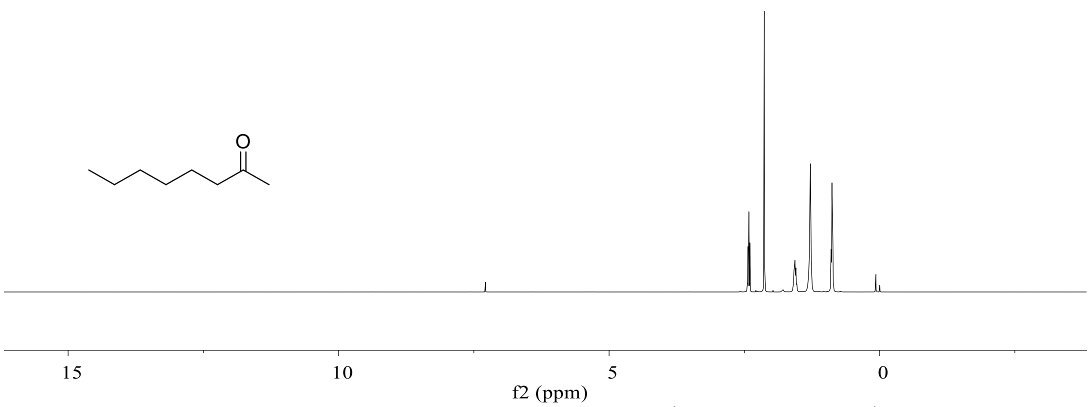

F
O
15
10
5
0
f2 (ppm)


F
NH2
15
10
5
0
f2 (ppm)

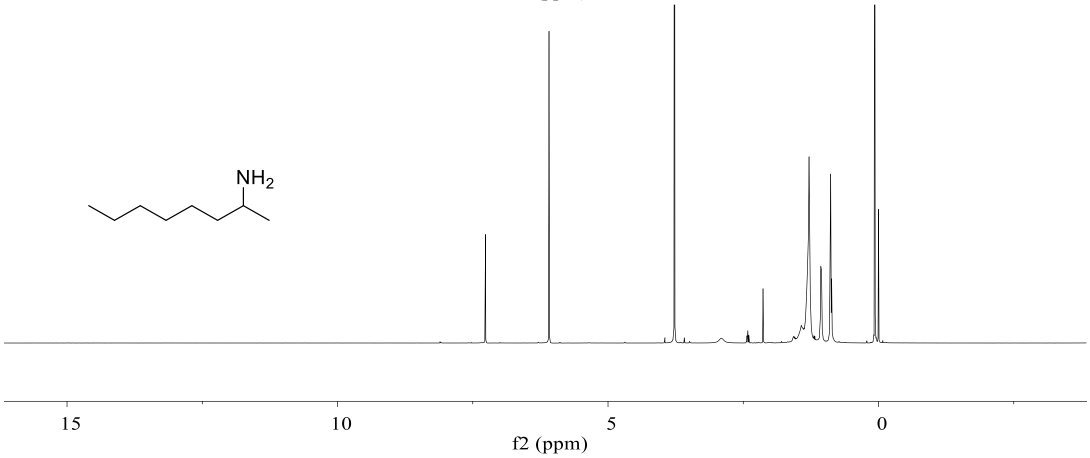

RT: 0.00 - 37.10
NL: 1.72E9 TIC MS A165
8.58
12.37
4.37
6.76
9.14
9.21
12.49
15.42
16.55
18.00
20.44
21.40
22.92
25.35
27.31
29.32
30.36
33.83
34.73
35.78
F
NH2
O
O
O
Time (min)


A165 #1348 RT: 8.58 AV: 1 NL: 9.15E8
T: + c EI Full ms [33.00-500.00]
44.04
45.17
83.05
91.07
109.08
138.12
152.13
176.03
207.06
238.81
267.93
281.13
296.70
326.99
341.09
375.12
405.00
430.99
458.50
495.33
F
NH2
Exact Mass: 153.10
m/z

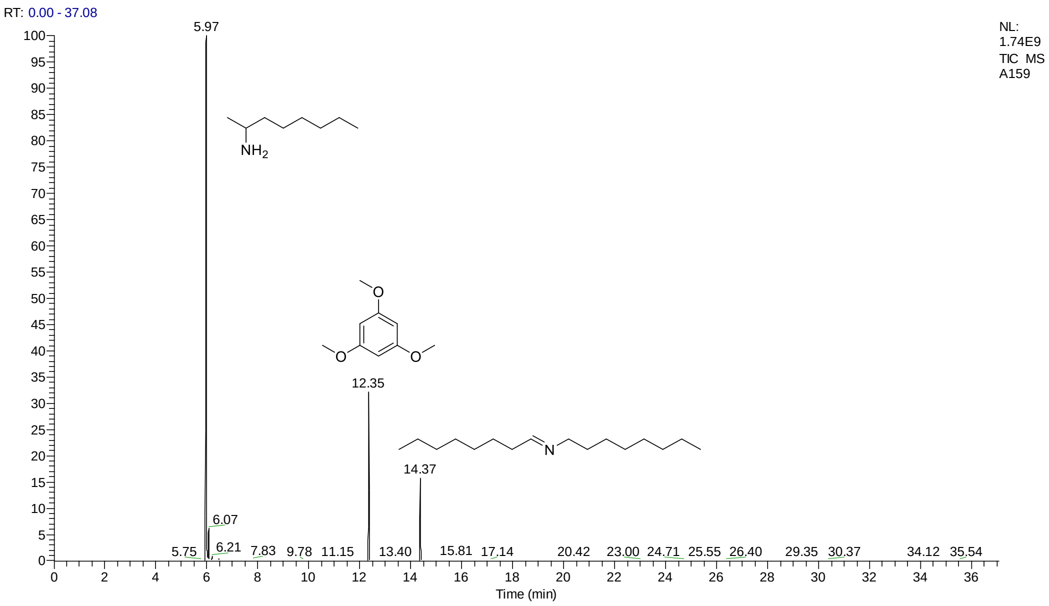
A159



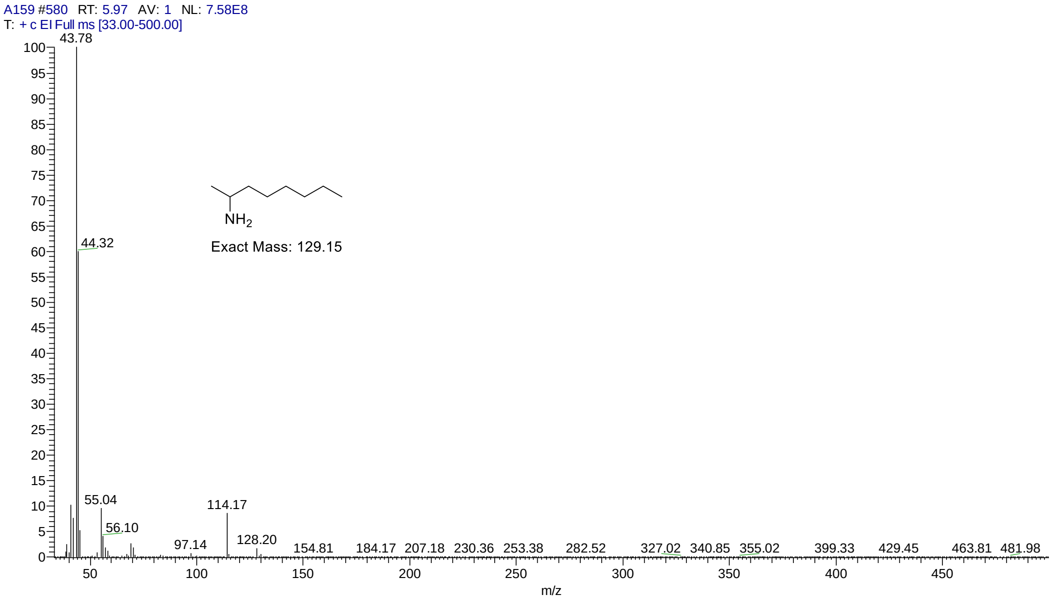
O
15
10
5
0
f2 (ppm)

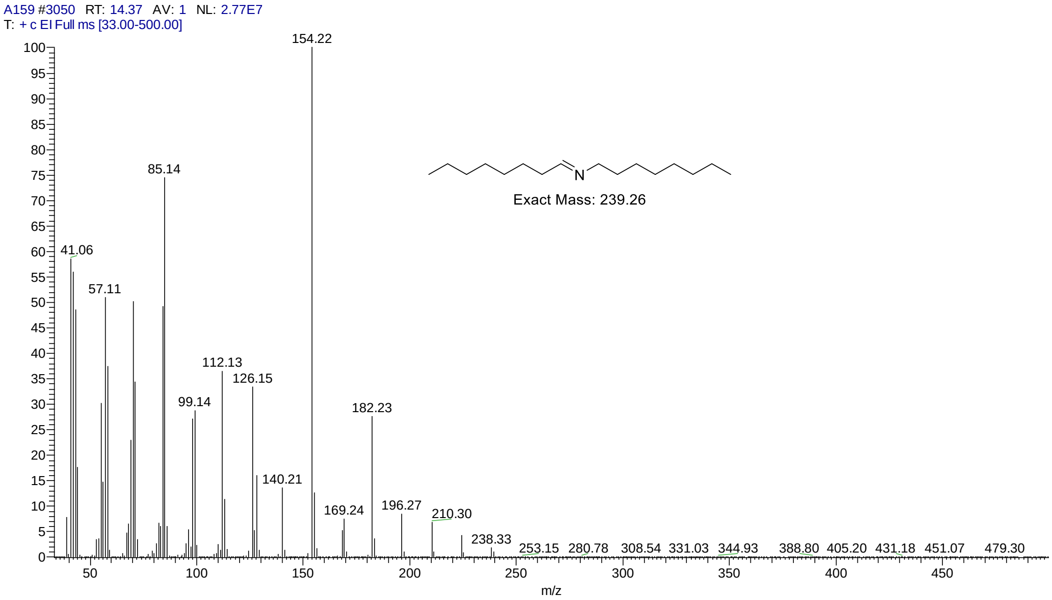


NH2
15
10
5
0
f2 (ppm)

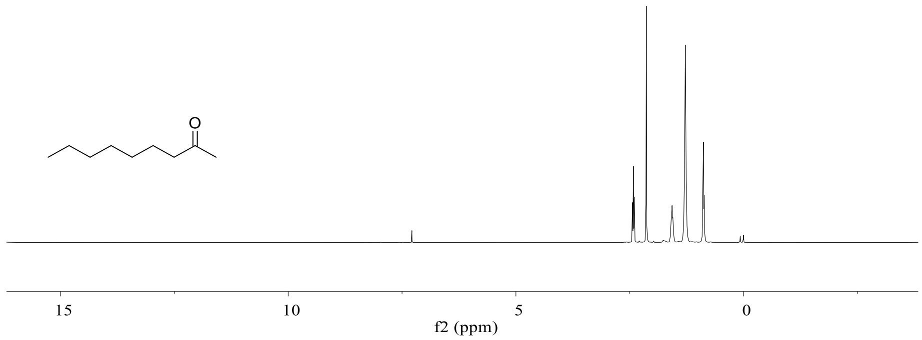


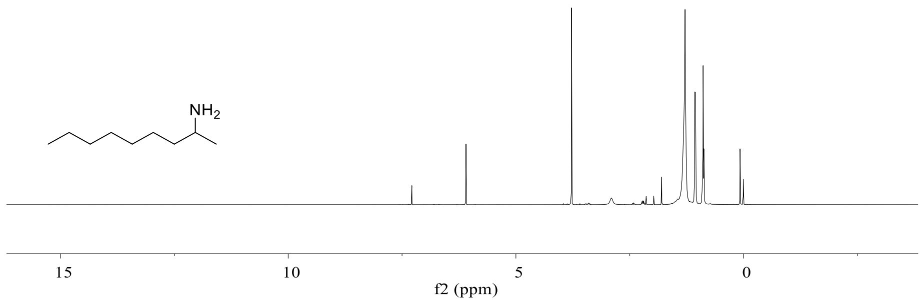
RT: 0.00 - 37.08
NL:
1.74E9
TIC MS
A159
5.97
NH2
12.35
N
14.37
6.07
6.21
5.75
7.83
9.78
11.15
13.40
15.81
17.14
20.42
23.00
24.71
25.55
26.40
29.35
30.37
34.12
35.54
Time (min)

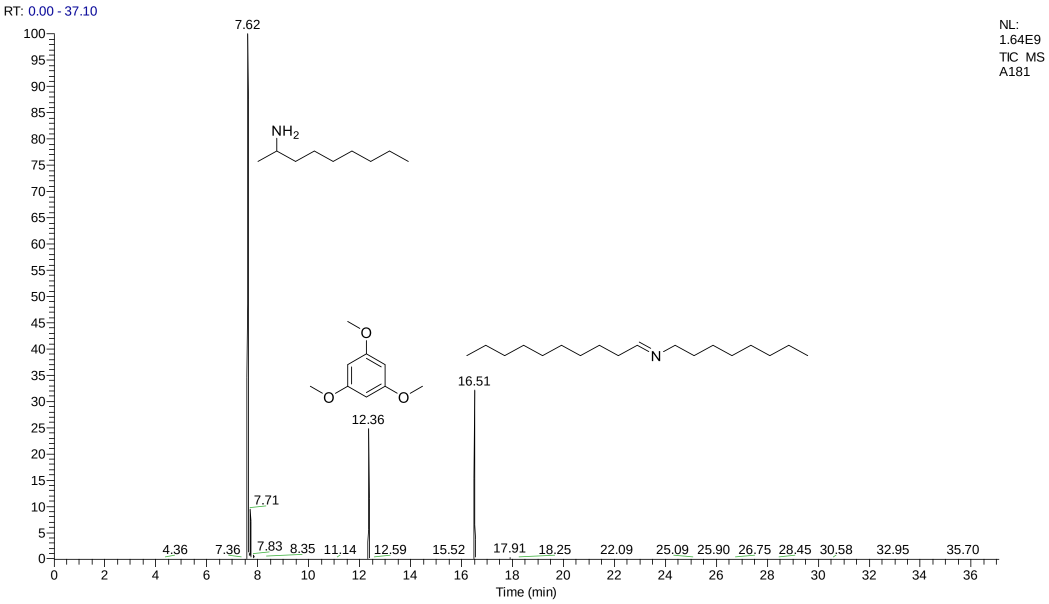

A159 #580 RT: 5.97 AV: 1 NL: 7.58E8
T: + c EI Full ms [33.00-500.00]
43.78
44.32
55.04
56.10
97.14
114.17
128.20
154.81
184.17
207.18
230.36
253.38
282.52
327.02
340.85
355.02
399.33
429.45
463.81
481.98
NH_2
Exact Mass: 129.15
m/z

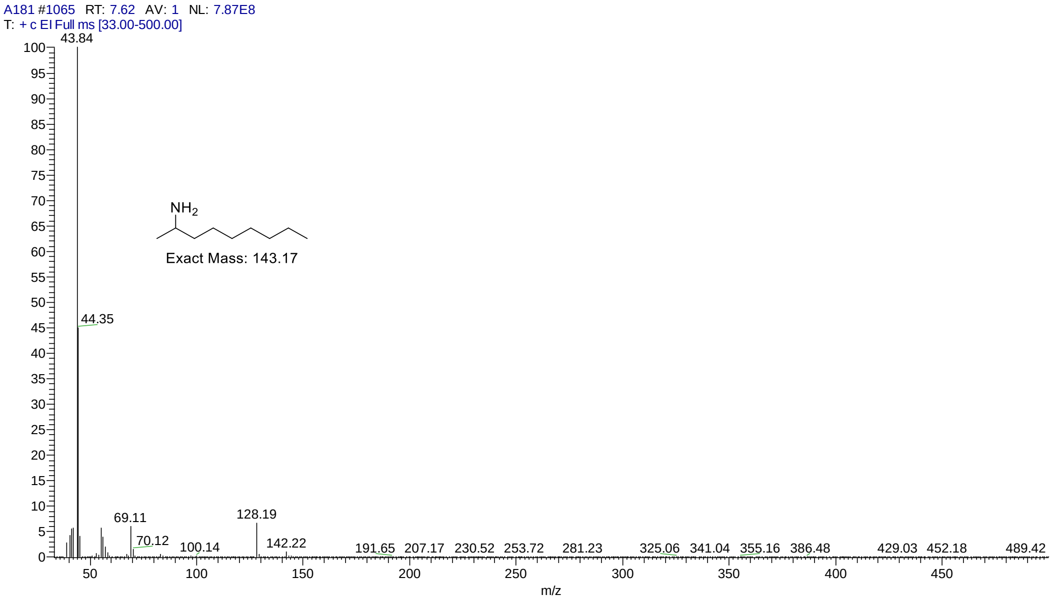

A159 #3050 RT: 14.37 AV: 1 NL: 2.77E7
T: + c EI Full ms [33.00-500.00]
154.22
85.14
41.06
57.11
112.13
126.15
99.14
182.23
140.21
169.24
196.27
210.30
238.33
253.15
280.78
308.54
331.03
344.93
388.80
405.20
431.18
451.07
479.30
N
Exact Mass: 239.26
m/z

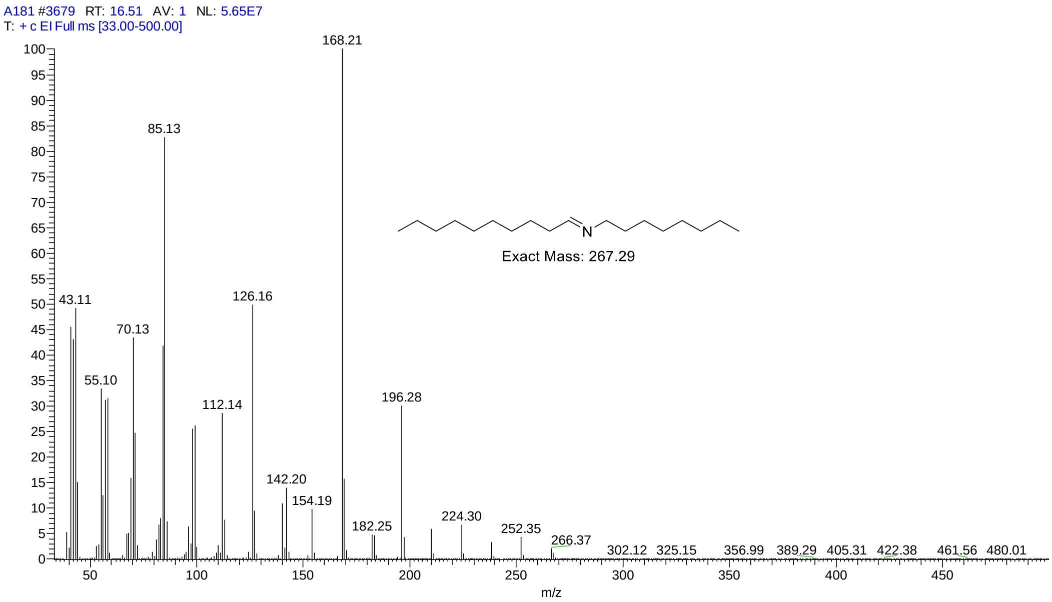
A181


O
15
10
5
0
f2 (ppm)

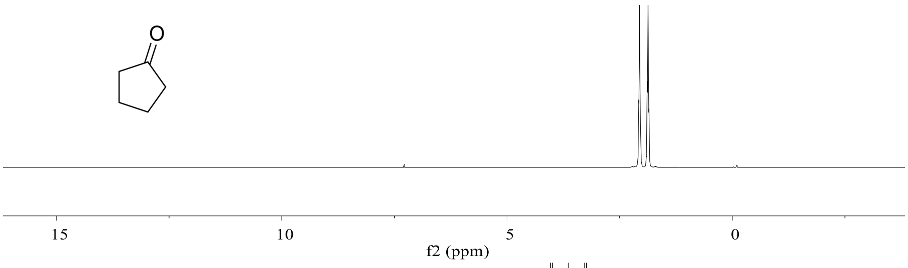

NH2
15
10
5
0
f2 (ppm)

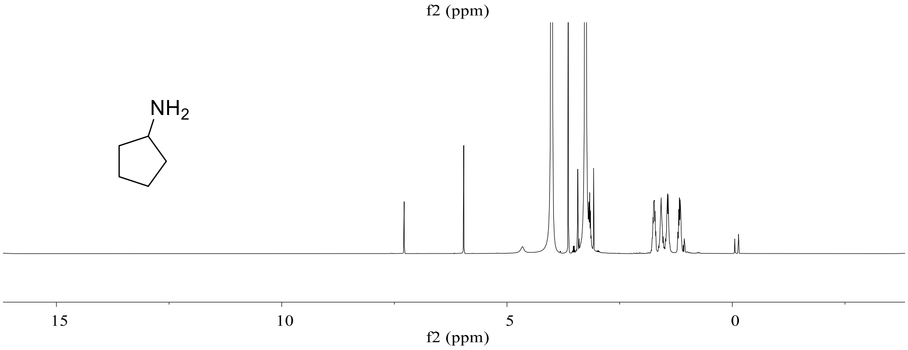

RT: 0.00 - 37.10
NL:
1.64E9
TIC MS
A181
7.62
NH2
O
O
O
N
16.51
12.36
7.71
4.36
7.36
7.83
8.35
11.14
12.59
15.52
17.91
18.25
22.09
25.09
25.90
26.75
28.45
30.58
32.95
35.70
Time (min)


A181 #1065 RT: 7.62 AV: 1 NL: 7.87E8
T: + c EI Full ms [33.00-500.00]
43.84
44.35
69.11
70.12
100.14
128.19
142.22
191.65
207.17
230.52
253.72
281.23
325.06
341.04
355.16
386.48
429.03
452.18
489.42
NH2
Exact Mass: 143.17
m/z

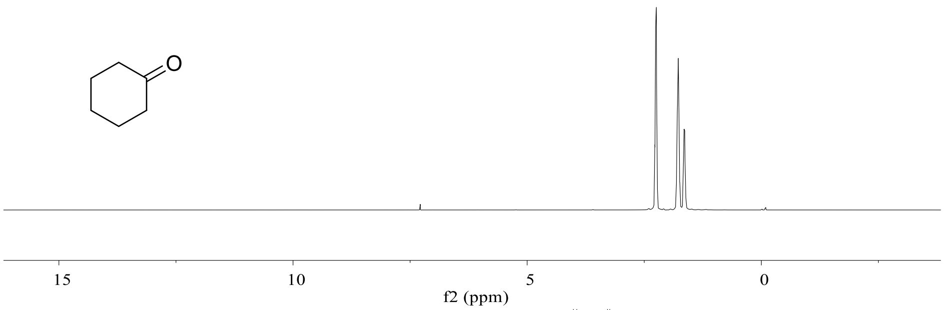

A181 #3679 RT: 16.51 AV: 1 NL: 5.65E7
T: + c EI Full ms [33.00-500.00]
168.21
85.13
43.11
70.13
126.16
55.10
112.14
196.28
142.20
154.19
182.25
224.30
252.35
266.37
302.12
325.15
356.99
389.29
405.31
422.38
461.56
480.01
N
Exact Mass: 267.29
m/z

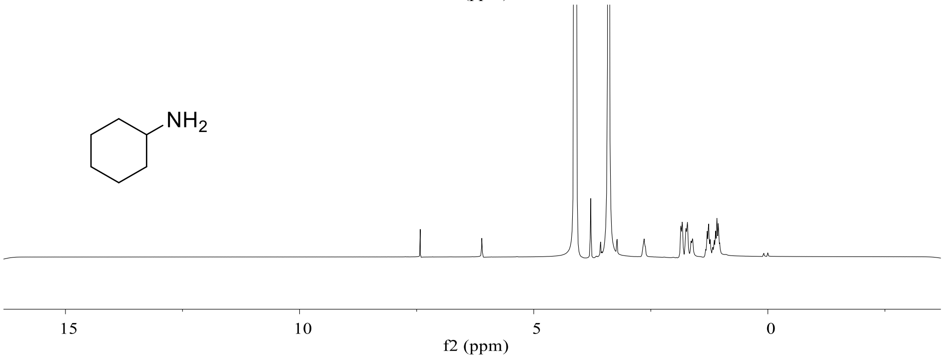
A278



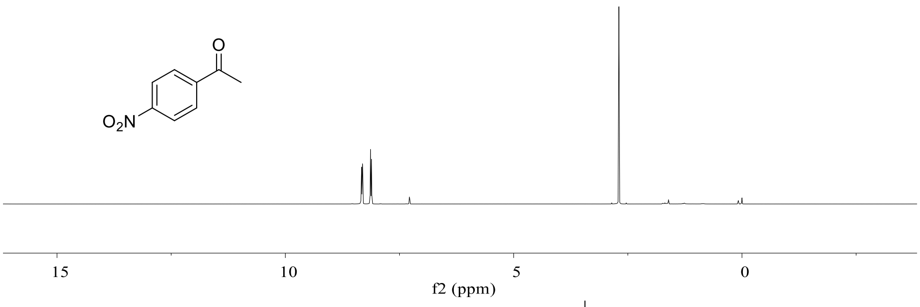


A280

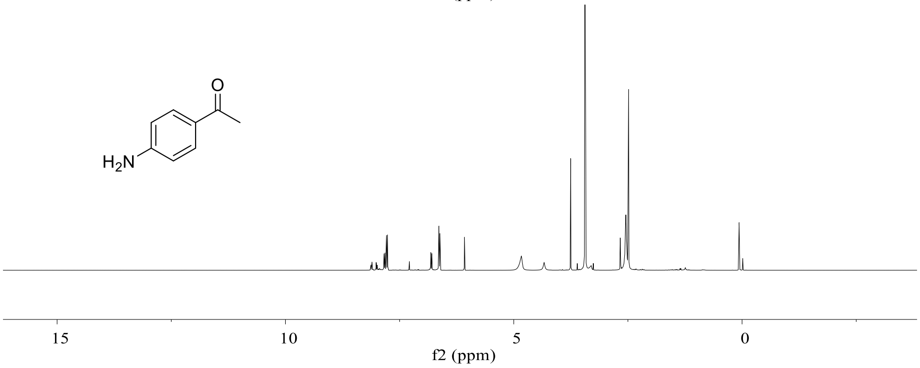




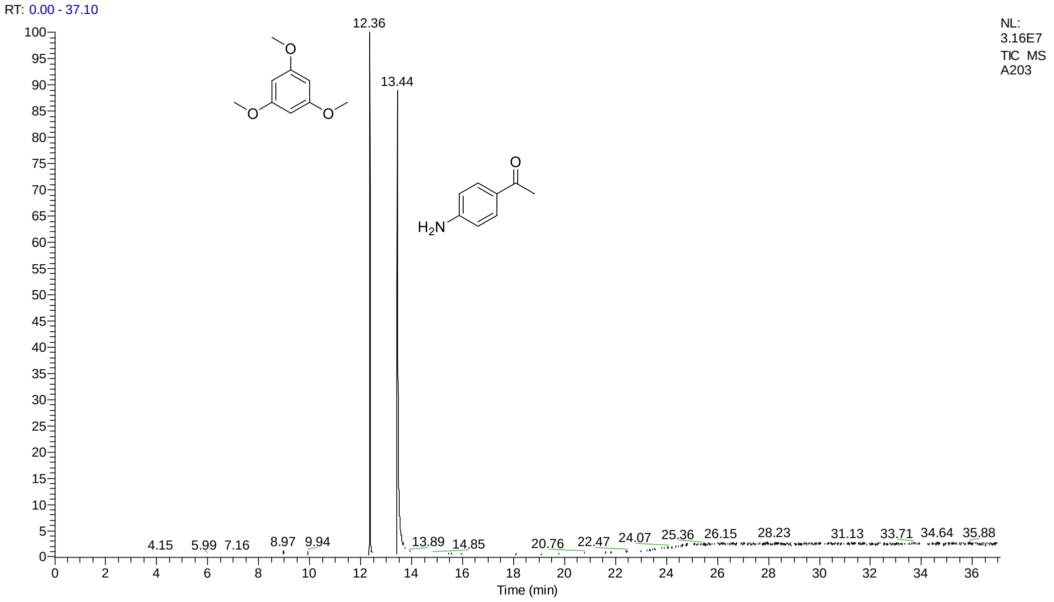
A203



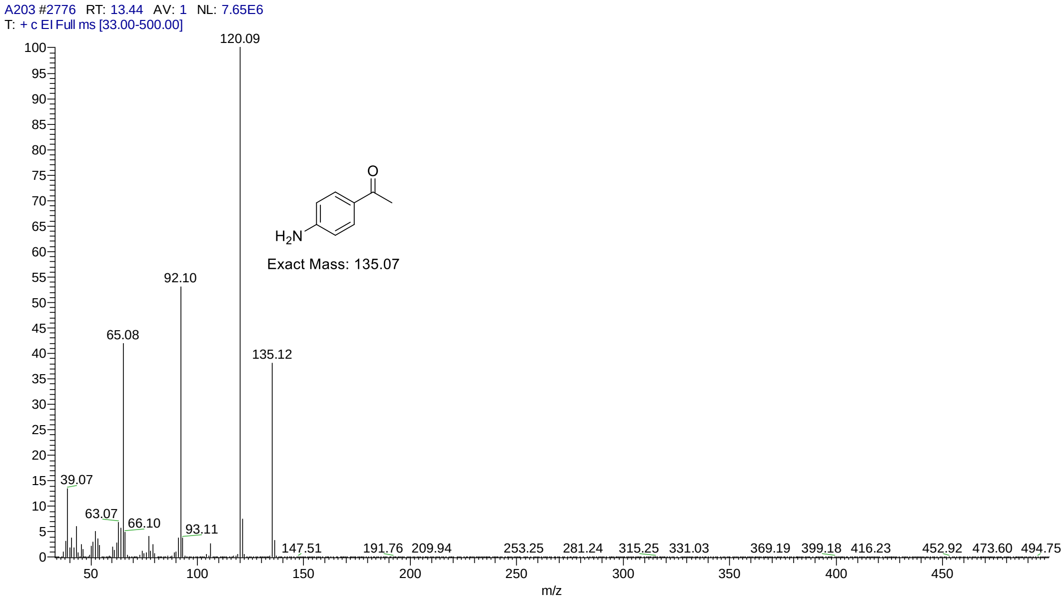
O_2N
O
15
10
5
0
f2 (ppm)



H_2N
O
15
10
5
0
f2 (ppm)

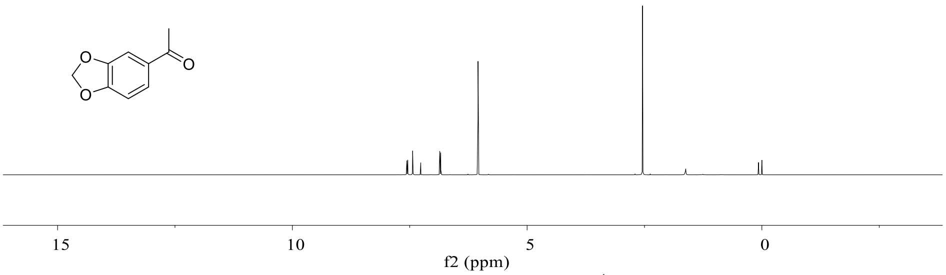


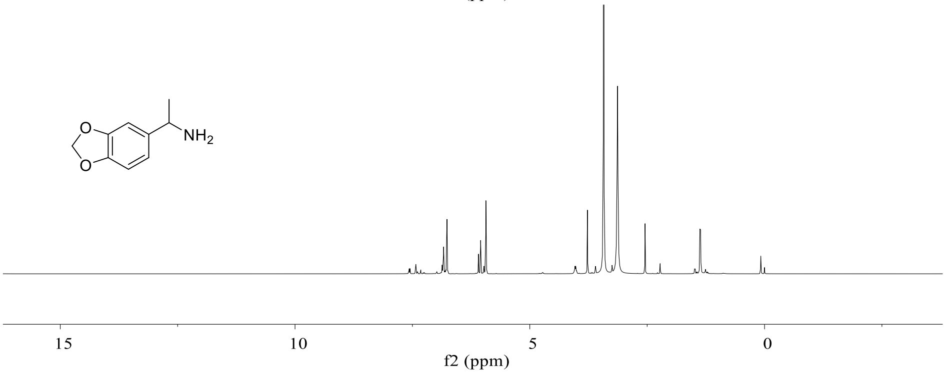
RT: 0.00 - 37.10
NL:
3.16E7
TIC MS
A203
12.36
13.44
4.15 5.99 7.16 8.97 9.94 13.89 14.85 20.76 22.47 24.07 25.36 26.15 28.23 31.13 33.71 34.64 35.88
Time (min)



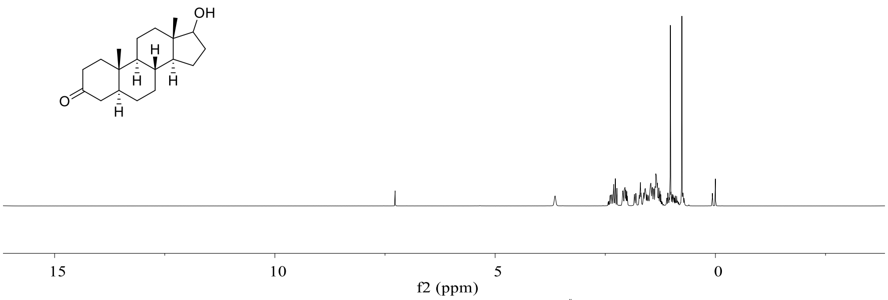
A203 #2776 RT: 13.44 AV: 1 NL: 7.65E6
T: + c EI Full ms [33.00-500.00]
120.09
92.10
65.08
135.12
39.07
63.07
66.10
93.11
147.51 191.76 209.94 253.25 281.24 315.25 331.03 369.19 399.18 416.23 452.92 473.60 494.75
Exact Mass: 135.07
m/z

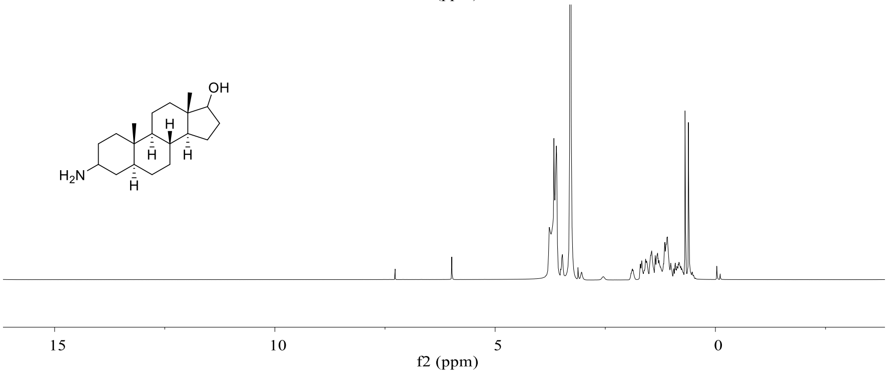
A230

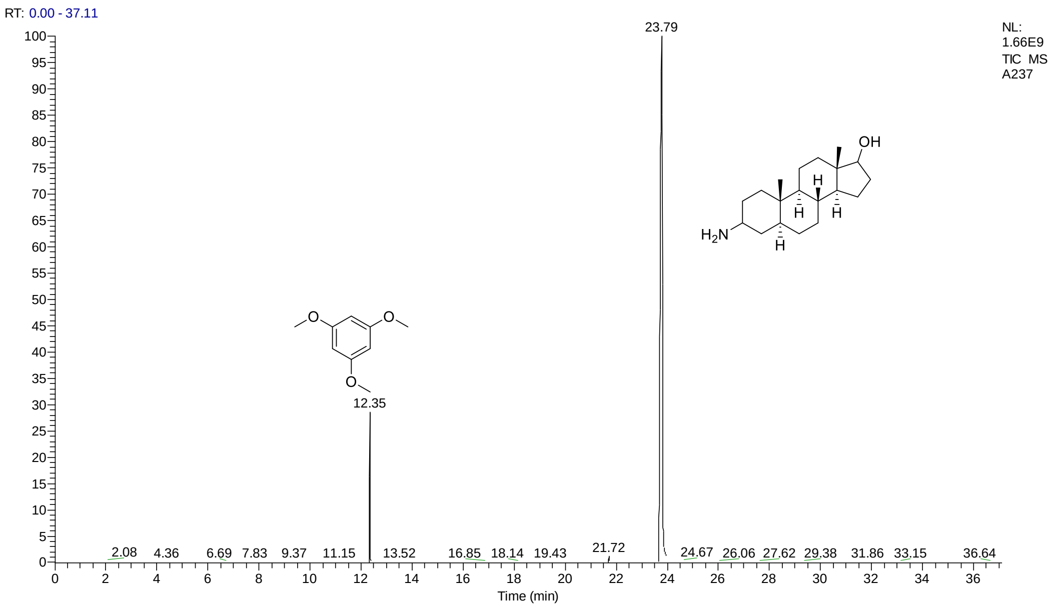


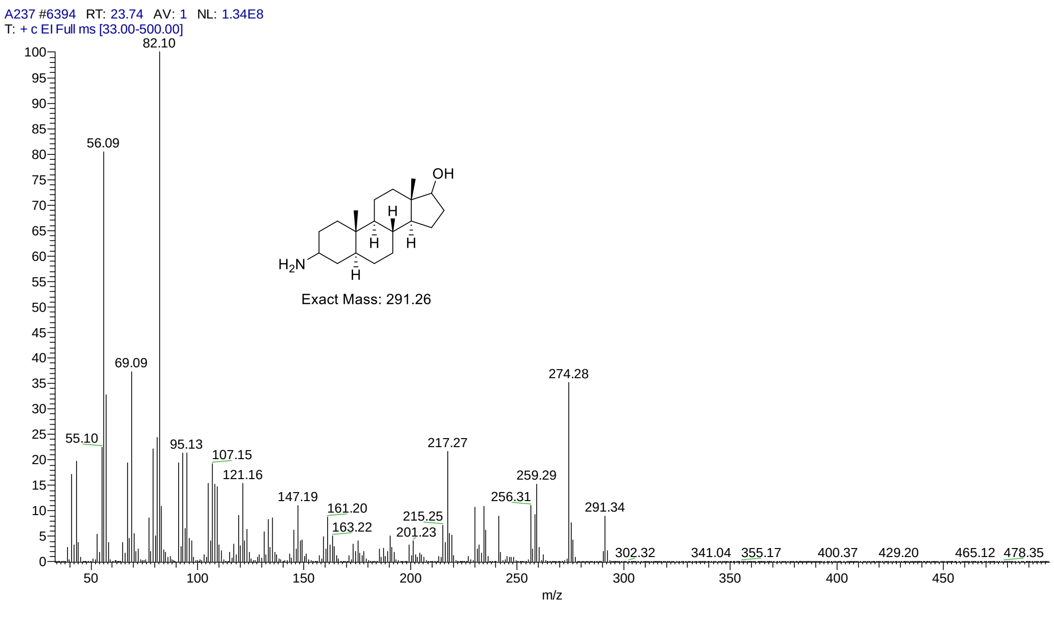


A237

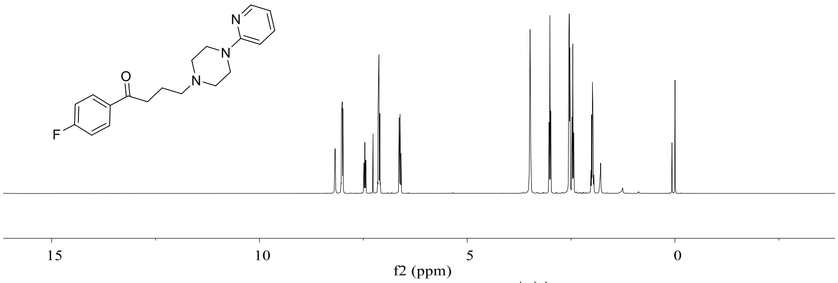

OH
H
H
H
O
H
15
10
5
0
f2 (ppm)

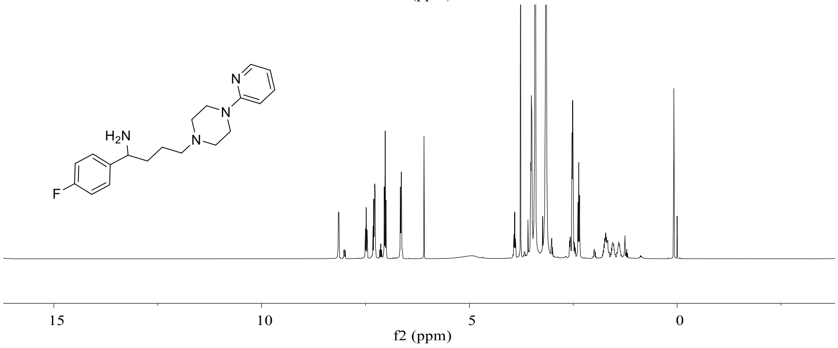

OH
H
H
H
H_2N
H
15
10
5
0
f2 (ppm)

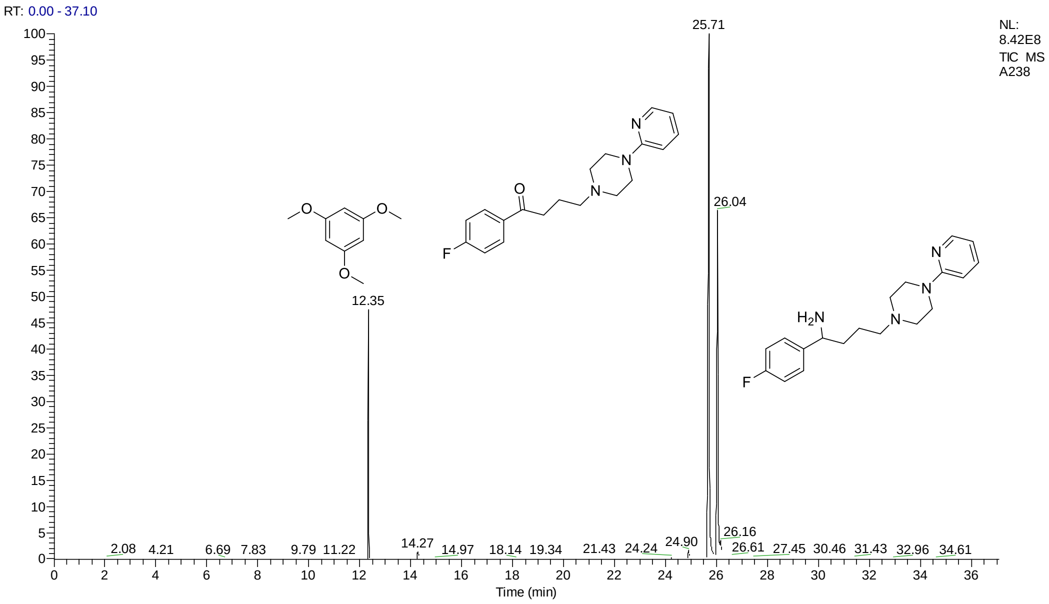

RT: 0.00 - 37.11
23.79
NL:
1.66E9
TIC MS
A237
12.35
2.08
4.36
6.69
7.83
9.37
11.15
13.52
16.85
18.14
19.43
21.72
24.67
26.06
27.62
29.38
31.86
33.15
36.64
Time (min)

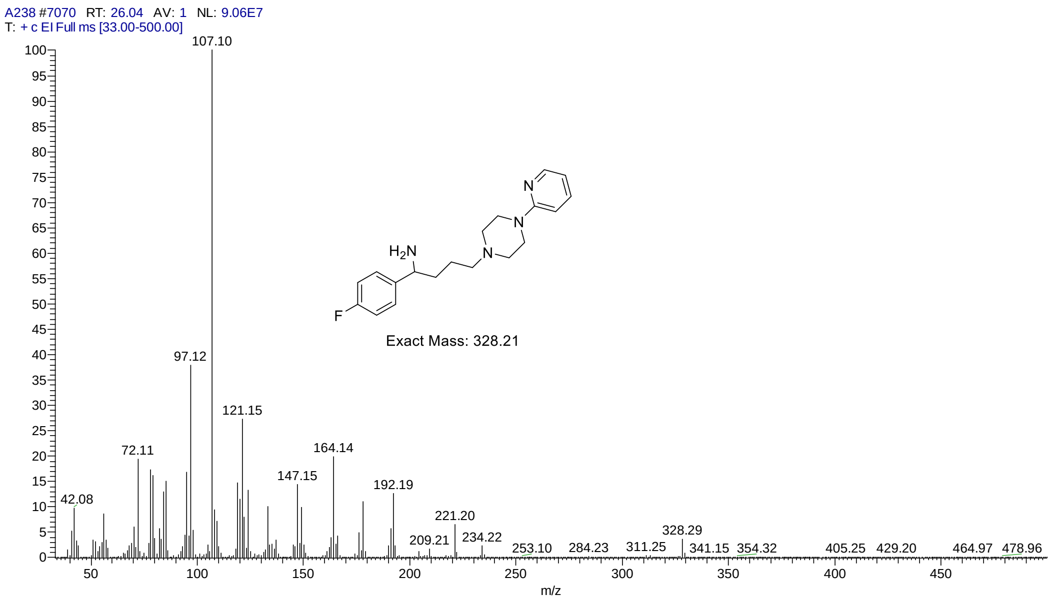

A237 #6394 RT: 23.74 AV: 1 NL: 1.34E8
T: + c EI Full ms [33.00-500.00]
82.10
56.09
69.09
55.10
95.13
107.15
121.16
147.19
161.20
163.22
215.25
201.23
217.27
256.31
259.29
274.28
291.34
302.32
341.04
355.17
400.37
429.20
465.12
478.35
Exact Mass: 291.26
m/z

A268 (A238 GC-MS)

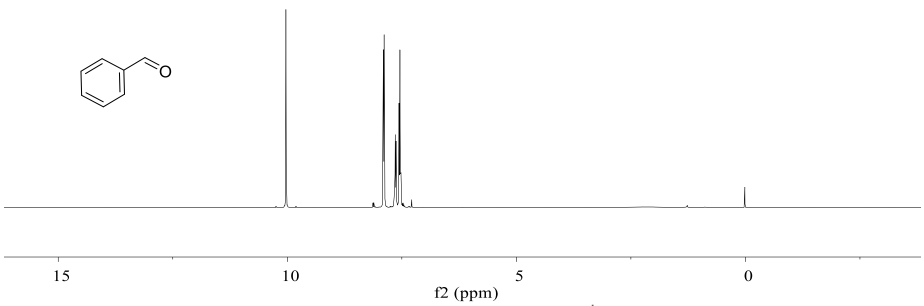




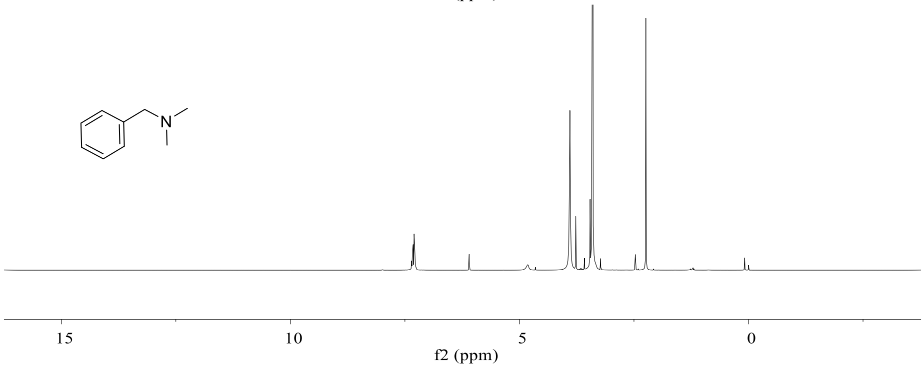




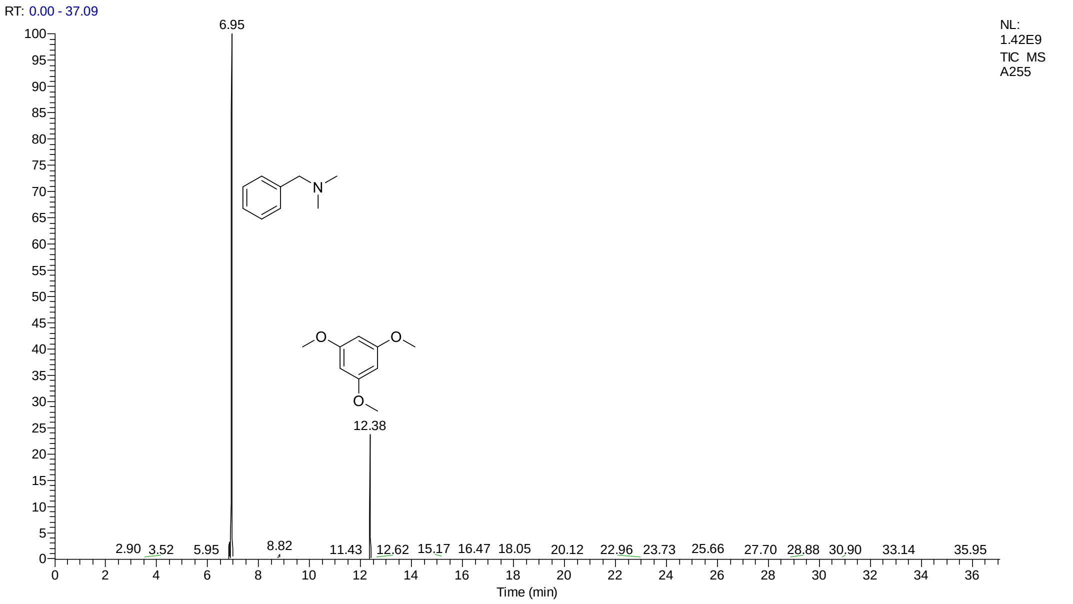
A255



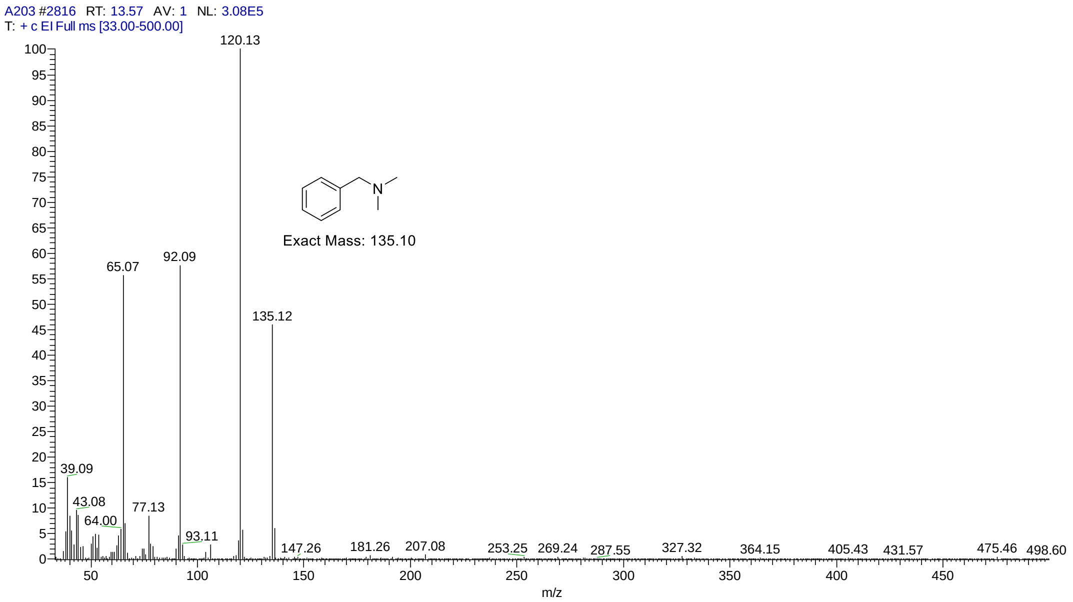
15
10
5
0
f2 (ppm)



15
10
5
0
f2 (ppm)

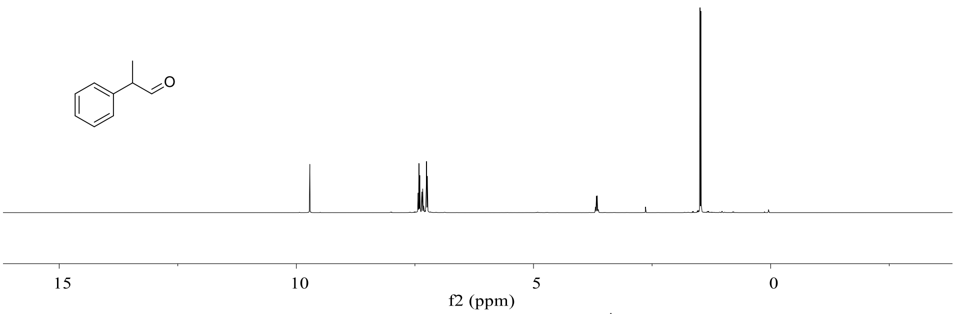


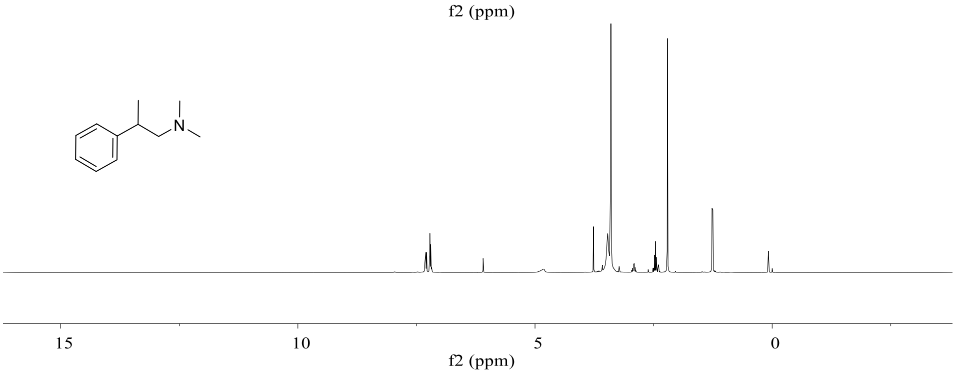
RT: 0.00 - 37.09
NL: 1.42E9 TIC MS A255
6.95
12.38
Time (min)

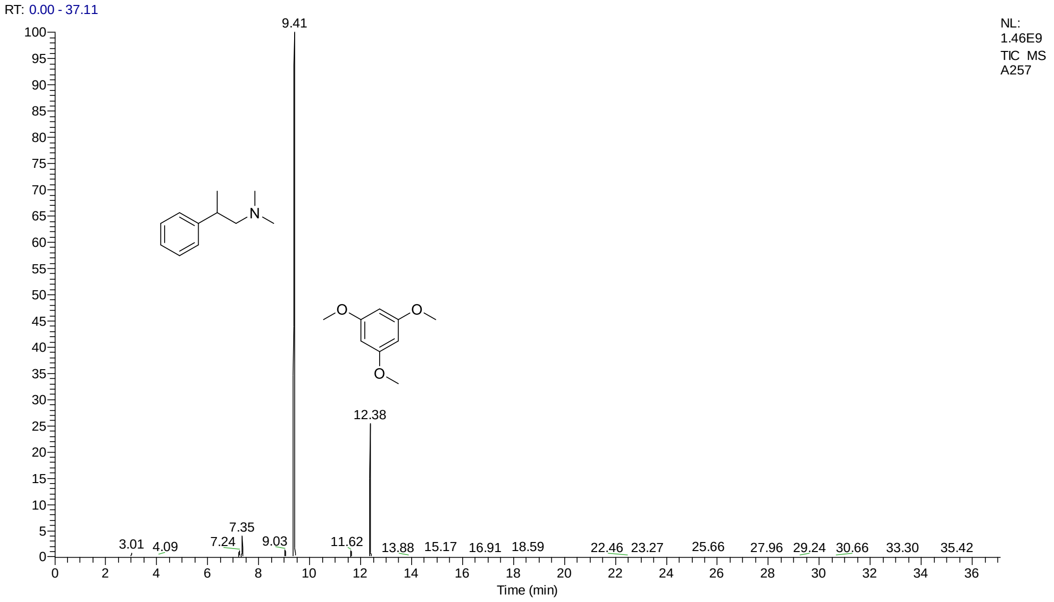


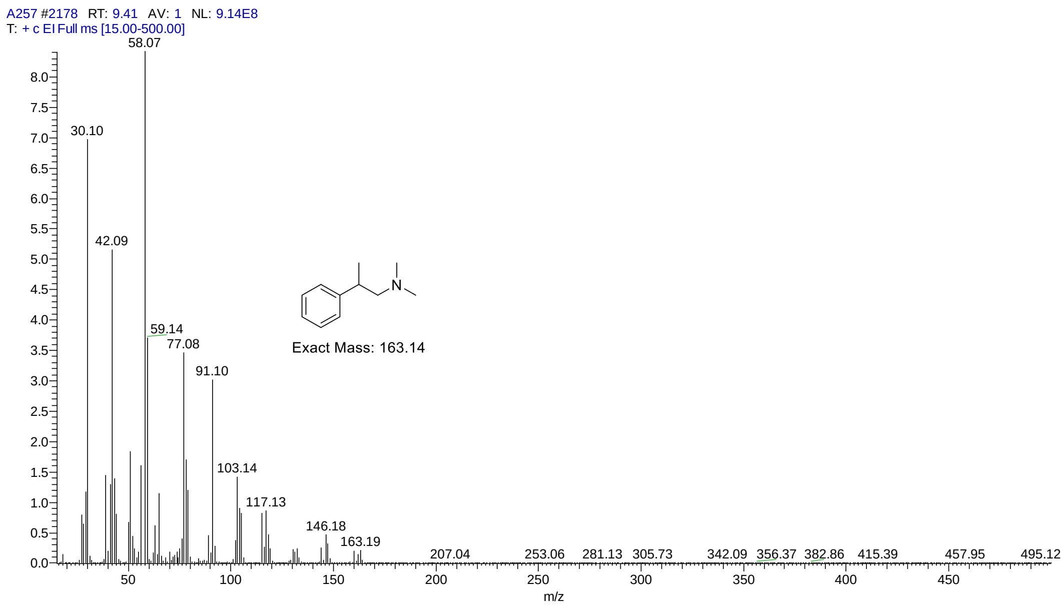
A203 #2816 RT: 13.57 AV: 1 NL: 3.08E5
T: + c EI Full ms [33.00-500.00]
120.13
65.07
92.09
135.12
39.09
43.08
64.00
77.13
93.11
Exact Mass: 135.10
m/z

A257

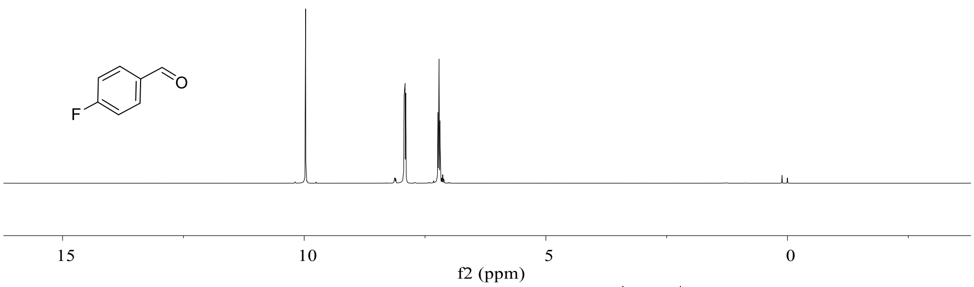

O
15
10
5
0
f2 (ppm)


N
15
10
5
0
f2 (ppm)

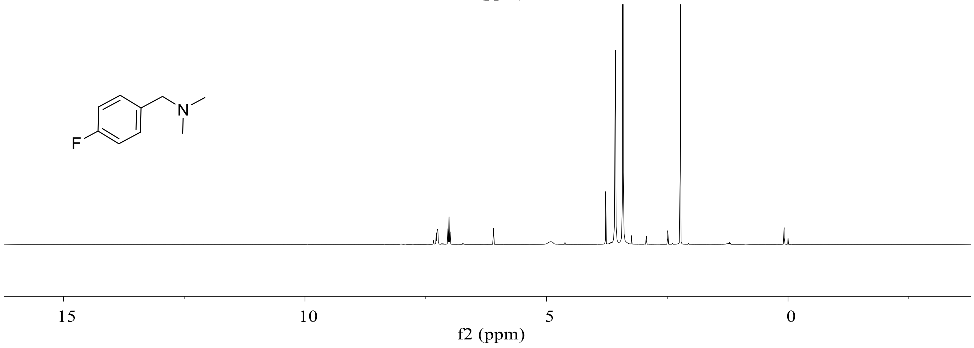

RT: 0.00 - 37.11
NL:
1.46E9
TIC MS
A257
9.41
12.38
7.35
3.01 4.09 7.24 9.03 11.62 13.88 15.17 16.91 18.59 22.46 23.27 25.66 27.96 29.24 30.66 33.30 35.42
Time (min)


A257 #2178 RT: 9.41 AV: 1 NL: 9.14E8
T: + c EI Full ms [15.00-500.00]
58.07
30.10
42.09
59.14
77.08
91.10
103.14
117.13
146.18
163.19
207.04 253.06 281.13 305.73 342.09 356.37 382.86 415.39 457.95 495.12
Exact Mass: 163.14
m/z

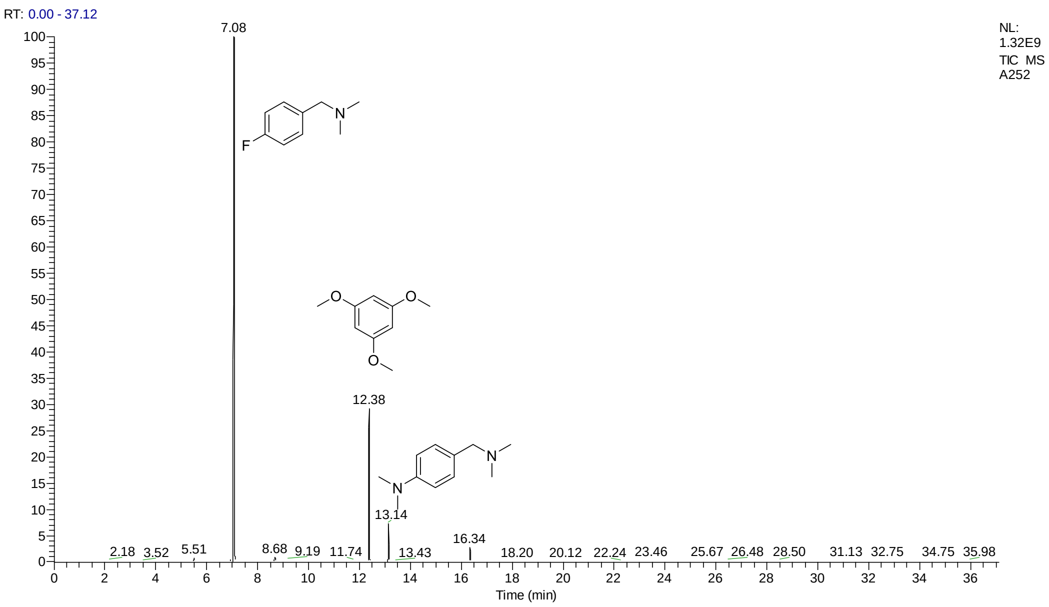
A252



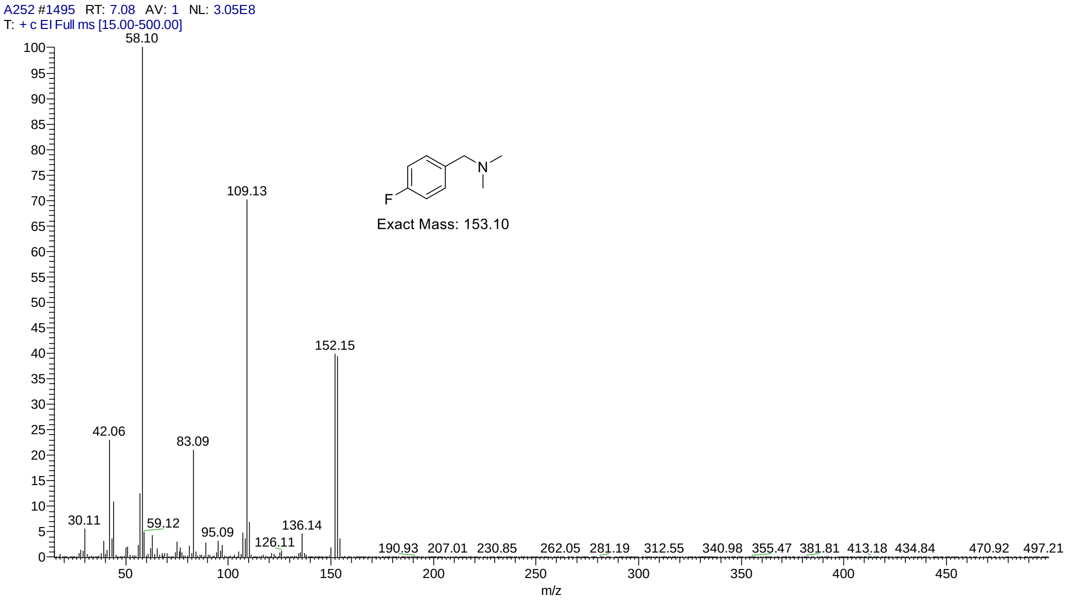
F
O
15
10
5
0
f2 (ppm)

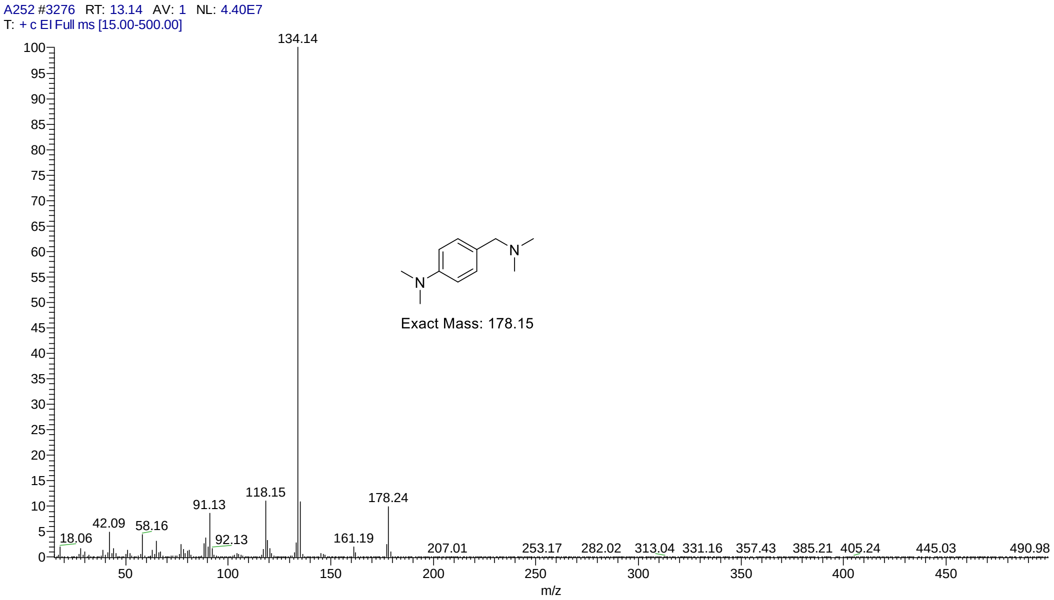


F
N
15
10
5
0
f2 (ppm)

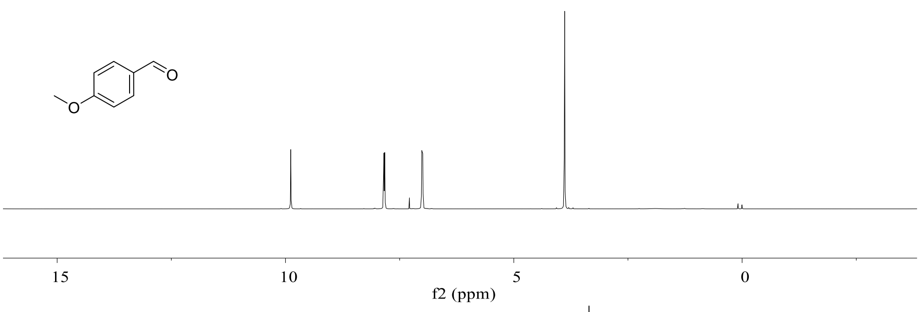


RT: 0.00 - 37.12
NL:
1.32E9
TIC MS
A252
7.08
12.38
13.14
16.34
2.18 3.52 5.51 8.68 9.19 11.74 13.43 18.20 20.12 22.24 23.46 25.67 26.48 28.50 31.13 32.75 34.75 35.98
Time (min)

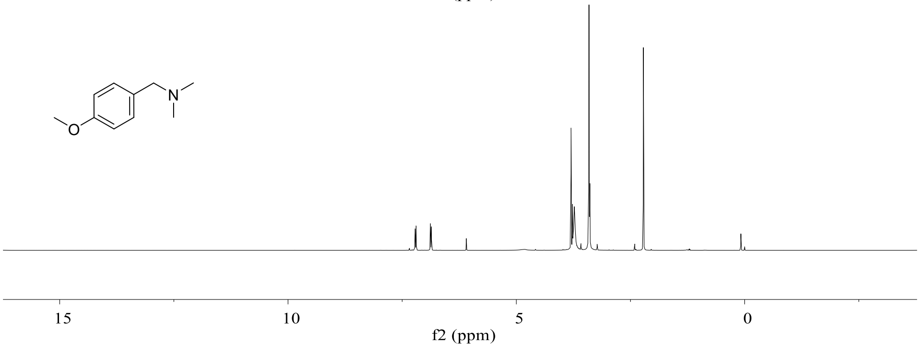

A252 #1495 RT: 7.08 AV: 1 NL: 3.05E8
T: + c EI Full ms [15.00-500.00]
58.10
109.13
152.15
42.06
83.09
30.11
59.12
95.09
126.11
136.14
190.93 207.01 230.85 262.05 281.19 312.55 340.98 355.47 381.81 413.18 434.84 470.92 497.21
m/z
F
N
Exact Mass: 153.10


A252 #3276 RT: 13.14 AV: 1 NL: 4.40E7
T: + c EI Full ms [15.00-500.00]
134.14
178.24
118.15
91.13
42.09
58.16
18.06
92.13
161.19
207.01 253.17 282.02 313.04 331.16 357.43 385.21 405.24 445.03 490.98
m/z
N
N
Exact Mass: 178.15

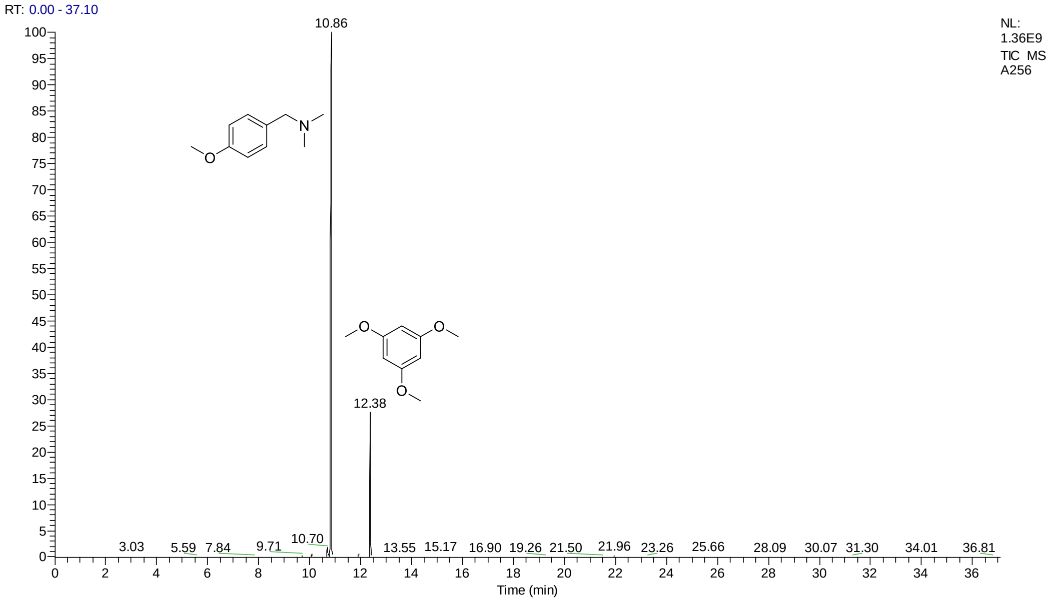
A256



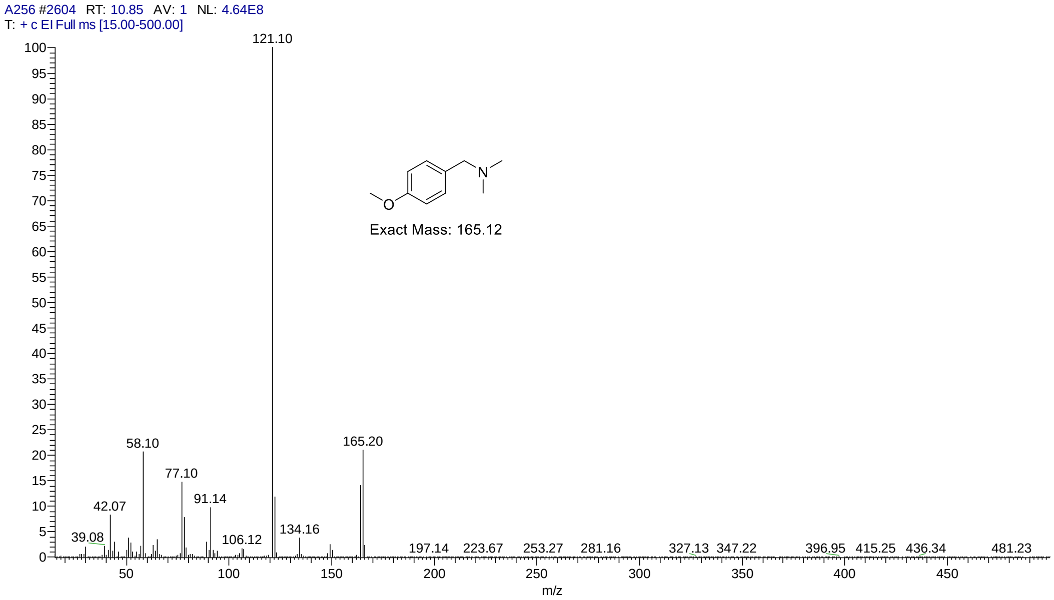
O
15
10
5
0
f2 (ppm)



N
O
15
10
5
0
f2 (ppm)

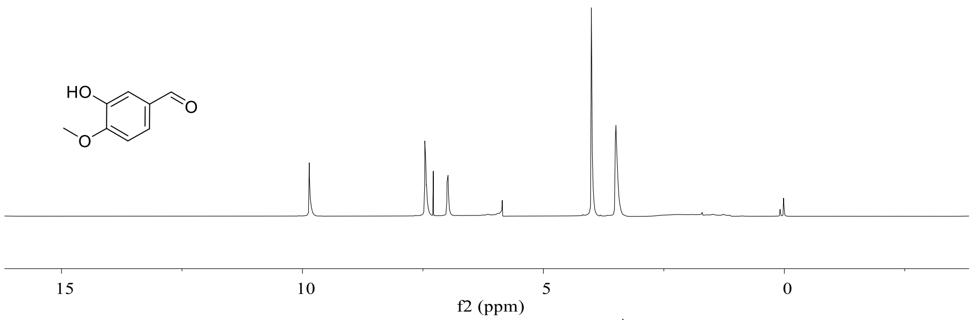


RT: 0.00 - 37.10
NL:
1.36E9
TIC MS
A256
10.86
12.38
3.03
5.59
7.84
9.71
10.70
13.55
15.17
16.90
19.26
21.50
21.96
23.26
25.66
28.09
30.07
31.30
34.01
36.81
Time (min)

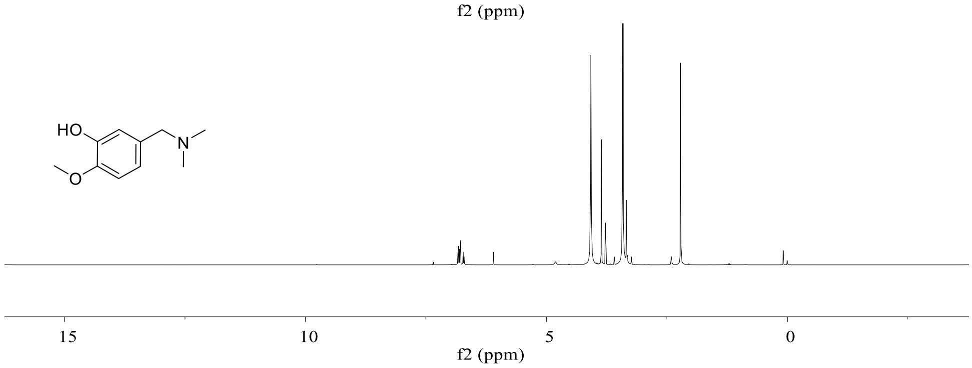


A256 #2604 RT: 10.85 AV: 1 NL: 4.64E8
T: + c EI Full ms [15.00-500.00]
121.10
Exact Mass: 165.12
58.10
165.20
77.10
42.07
91.14
39.08
106.12
134.16
197.14
223.67
253.27
281.16
327.13
347.22
396.95
415.25
436.34
481.23
m/z

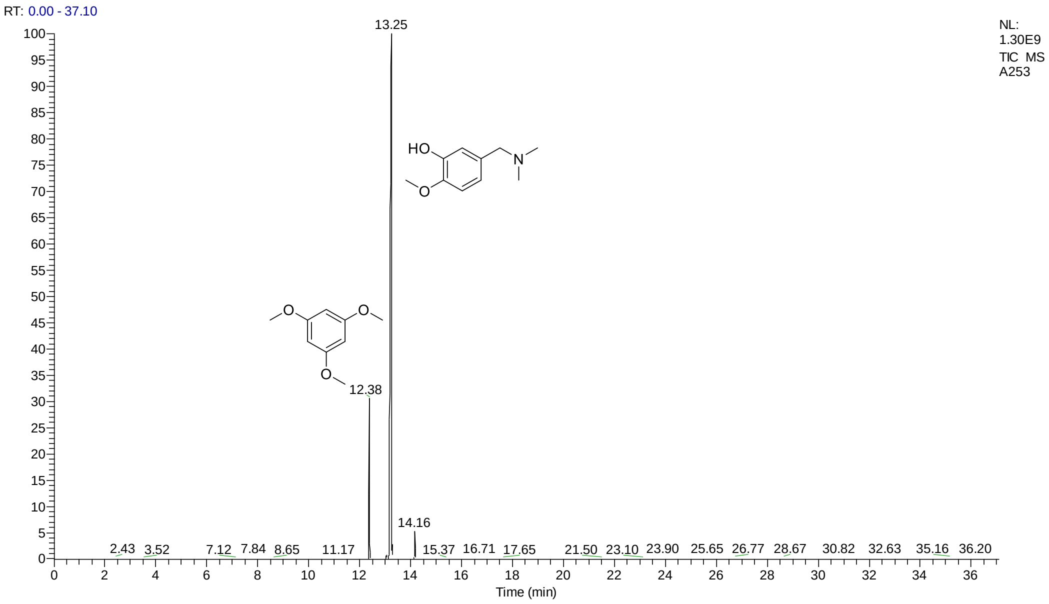
A253



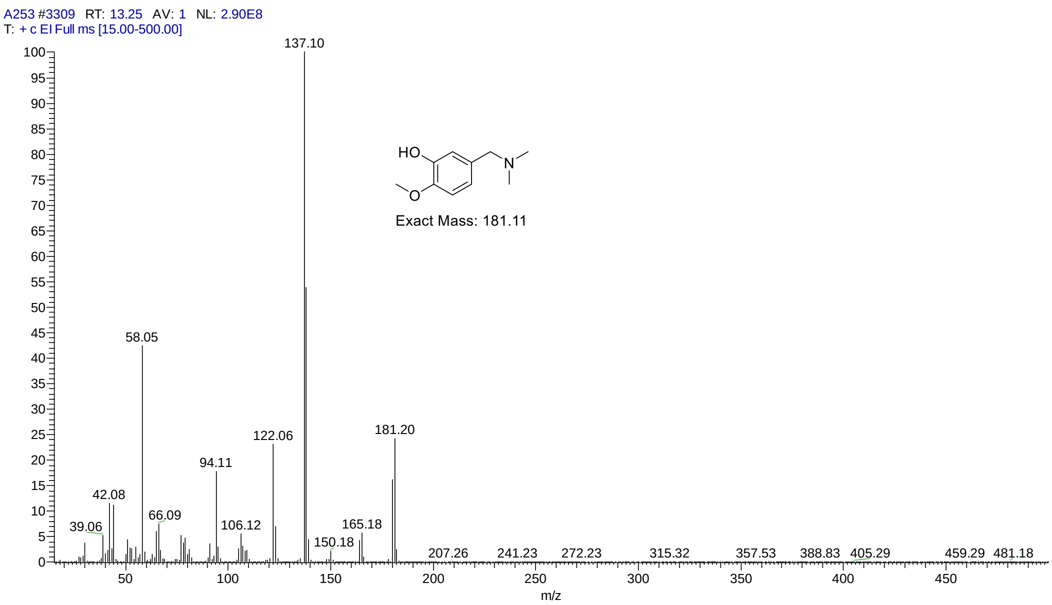
HO
O
O
15
10
5
0
f2 (ppm)



HO
N
O
15
10
5
0
f2 (ppm)

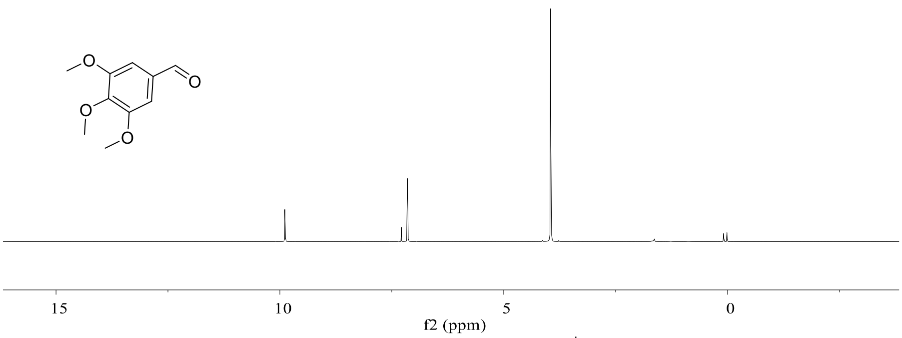


RT: 0.00 - 37.10
NL: 1.30E9 TIC MS A253
13.25
12.38
14.16
2.43 3.52 7.12 7.84 8.65 11.17 15.37 16.71 17.65 21.50 23.10 23.90 25.65 26.77 28.67 30.82 32.63 35.16 36.20
HO
N
O
O
O
O
Time (min)

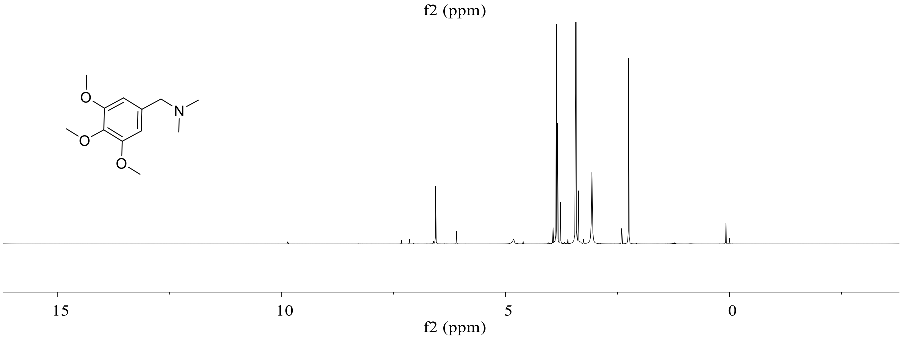


A253 #3309 RT: 13.25 AV: 1 NL: 2.90E8
T: + c EI Full ms [15.00-500.00]
137.10
58.05
181.20
122.06
94.11
42.08
66.09
106.12
39.06
165.18
150.18
207.26 241.23 272.23 315.32 357.53 388.83 405.29 459.29 481.18
HO
N
O
Exact Mass: 181.11
m/z

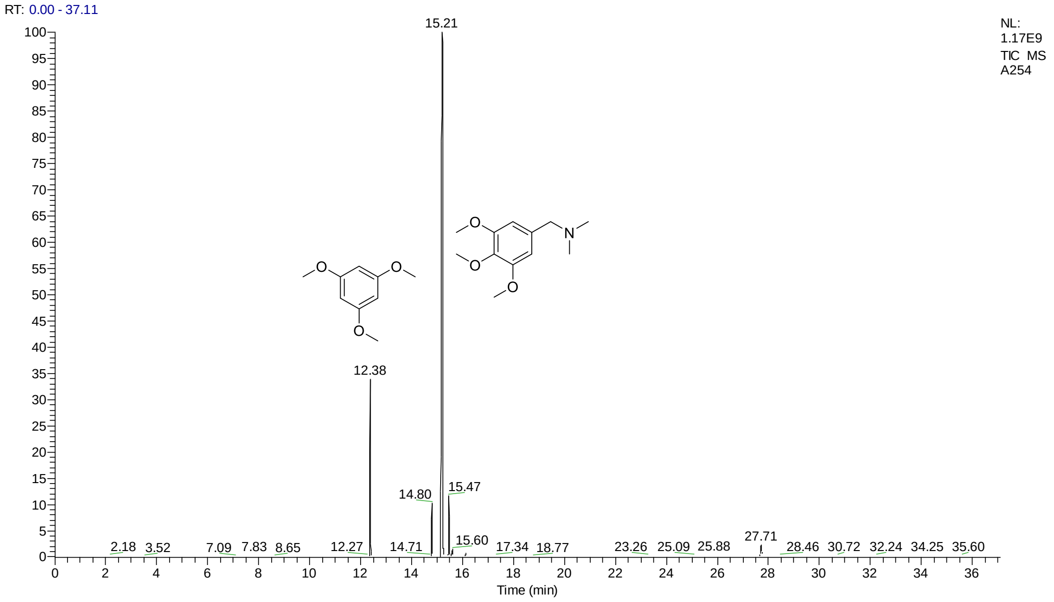
A254



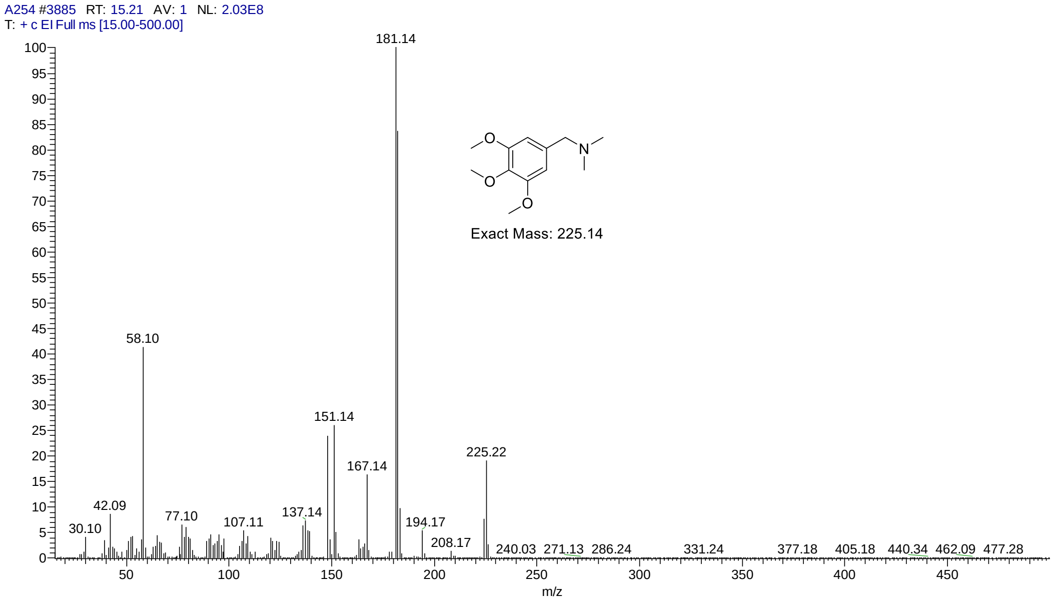
O
O
O
O
15
10
5
0
f2 (ppm)



O
N
O
O
15
10
5
0
f2 (ppm)

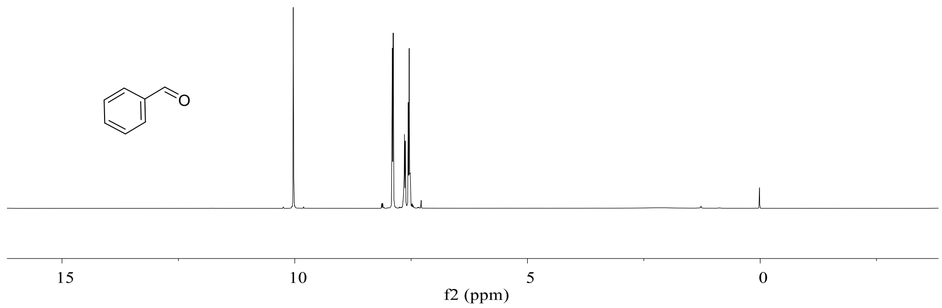


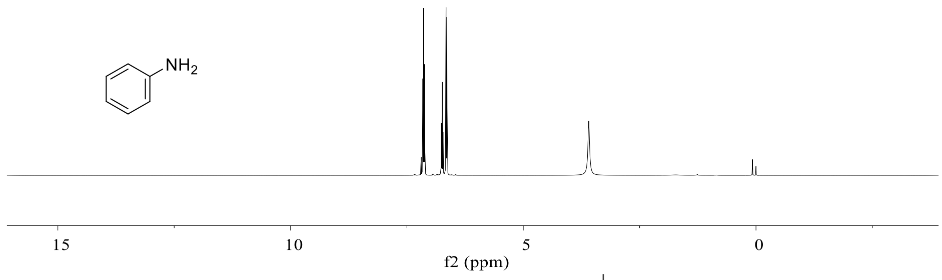
RT: 0.00 - 37.11
NL:
1.17E9
TIC MS
A254
15.21
12.38
14.80
15.47
2.18 3.52 7.09 7.83 8.65 12.27 14.71 15.60 17.34 18.77 23.26 25.09 25.88 27.71 28.46 30.72 32.24 34.25 35.60
Time (min)
O
O
O
N

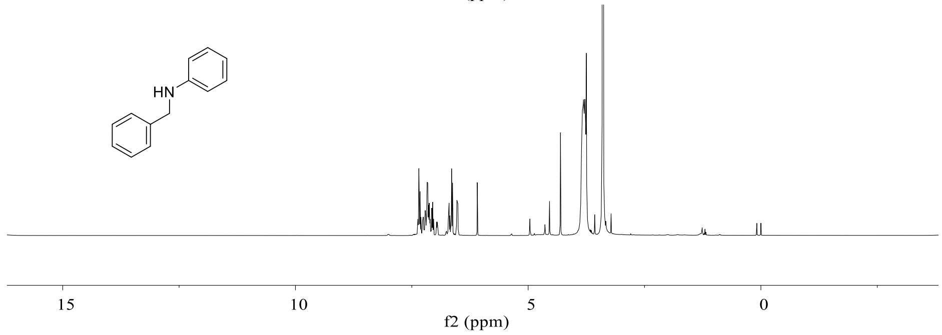


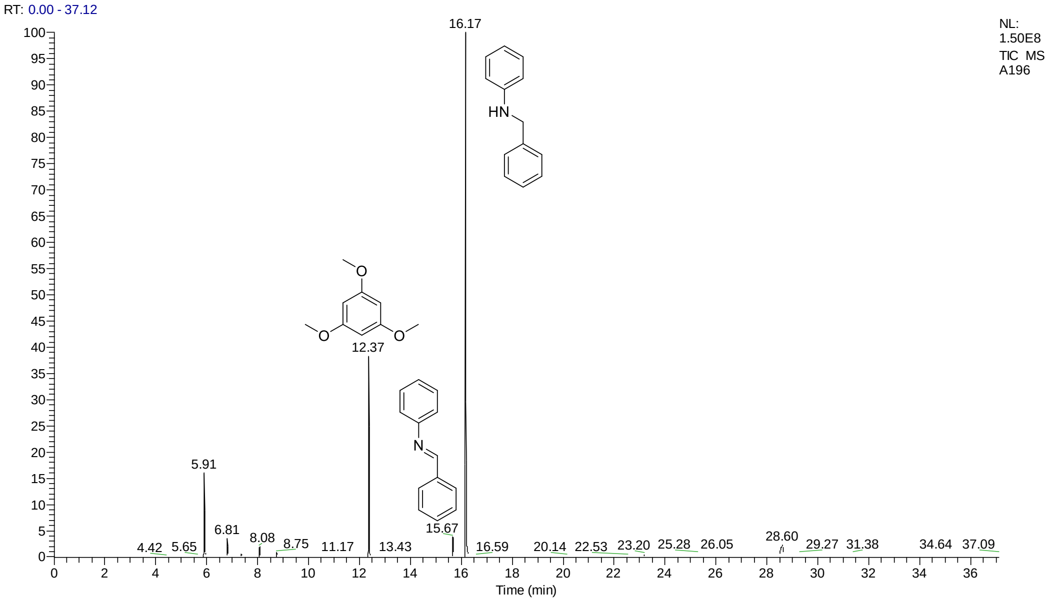
A254 #3885 RT: 15.21 AV: 1 NL: 2.03E8
T: + c EI Full ms [15.00-500.00]
181.14
58.10
151.14
225.22
167.14
42.09
30.10
77.10
107.11
137.14
194.17
208.17
240.03 271.13 286.24 331.24 377.18 405.18 440.34 462.09 477.28
m/z
N
O
O
O
Exact Mass: 225.14

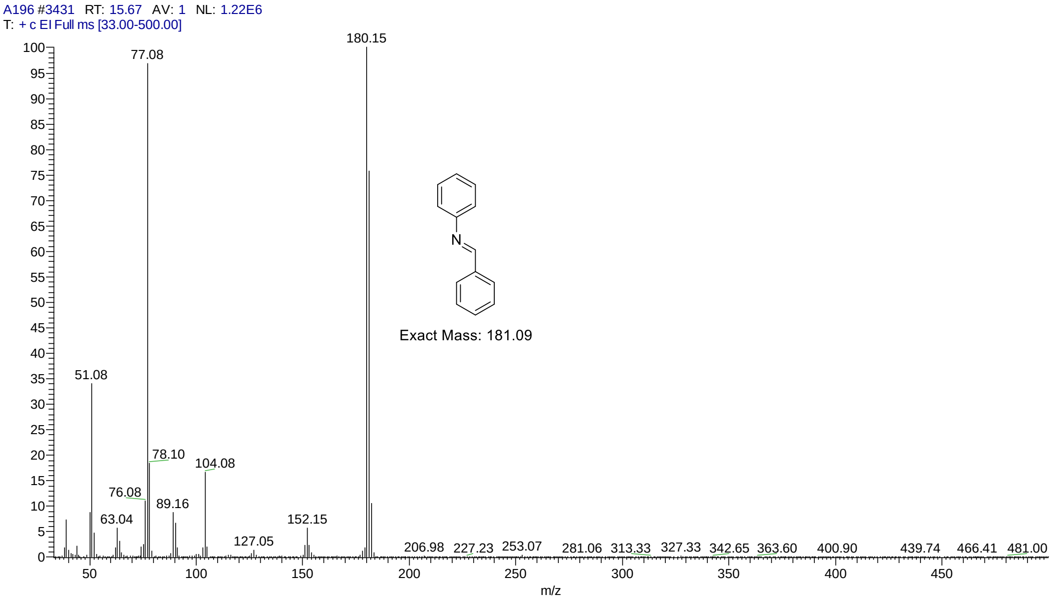
A196

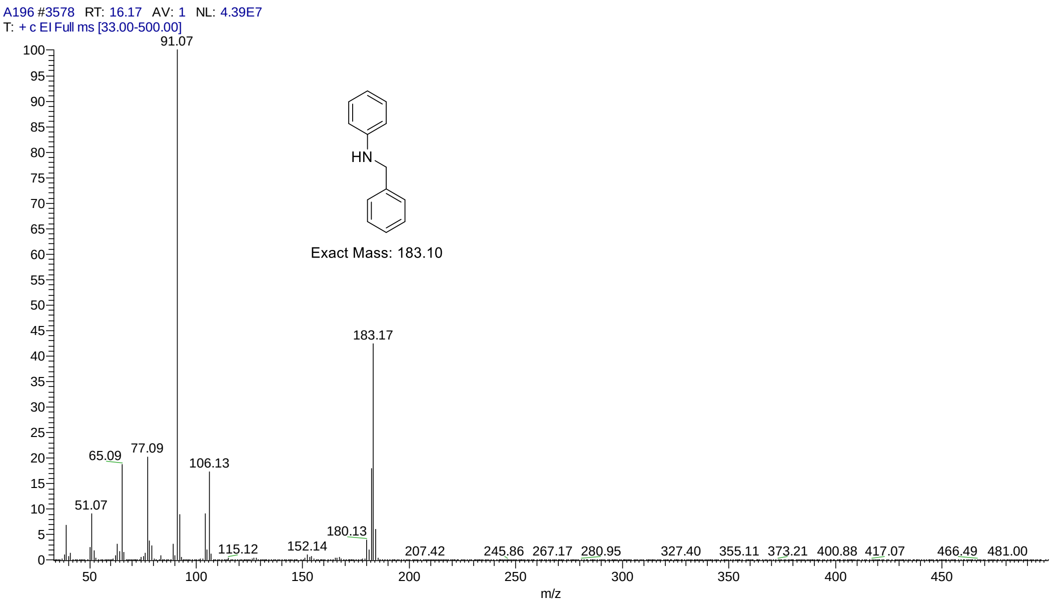

O
15
10
5
0
f2 (ppm)


NH2
15
10
5
0
f2 (ppm)

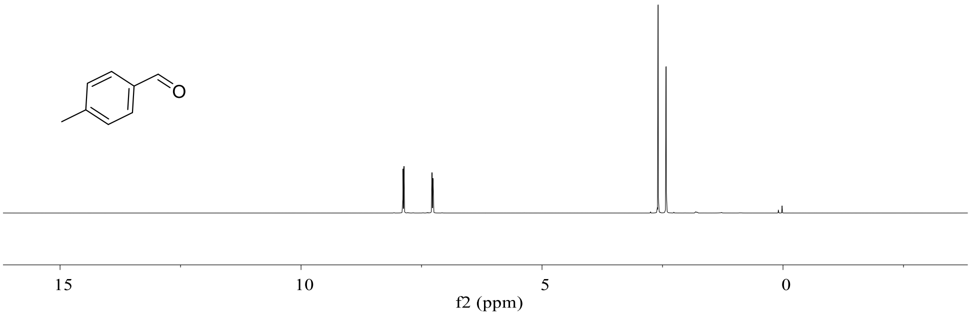

HN
15
10
5
0
f2 (ppm)

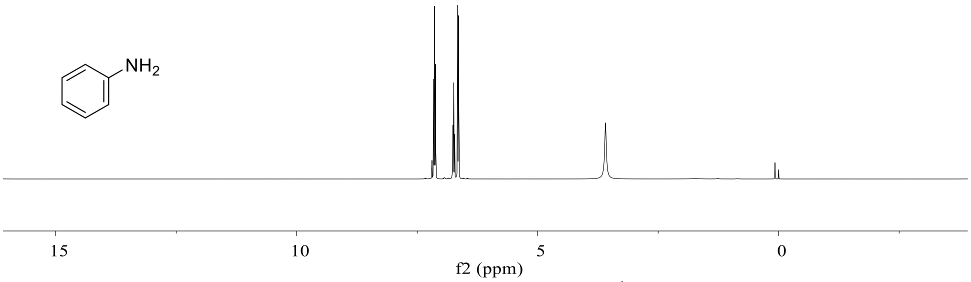

RT: 0.00 - 37.12
NL:
1.50E8
TIC MS
A196
16.17
HN
O
O
O
12.37
N
5.91
6.81
8.08
8.75
4.42
5.65
11.17
13.43
15.67
16.59
20.14
22.53
23.20
25.28
26.05
28.60
29.27
31.38
34.64
37.09
Time (min)

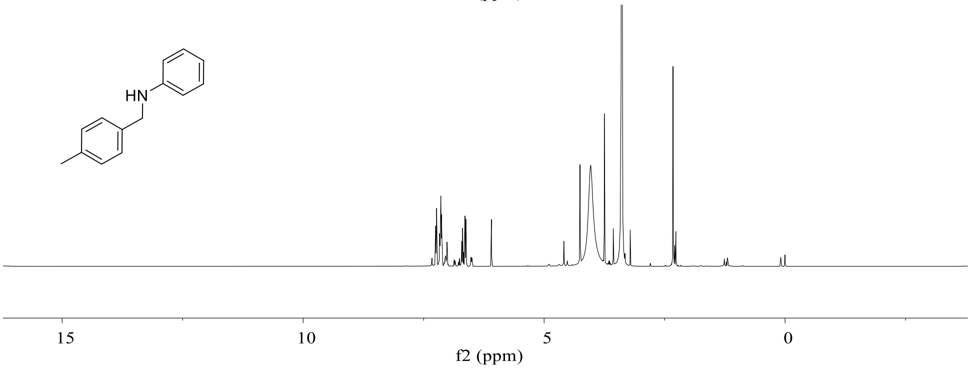

A196 #3431 RT: 15.67 AV: 1 NL: 1.22E6
T: + c EI Full ms [33.00-500.00]
Exact Mass: 181.09
m/z

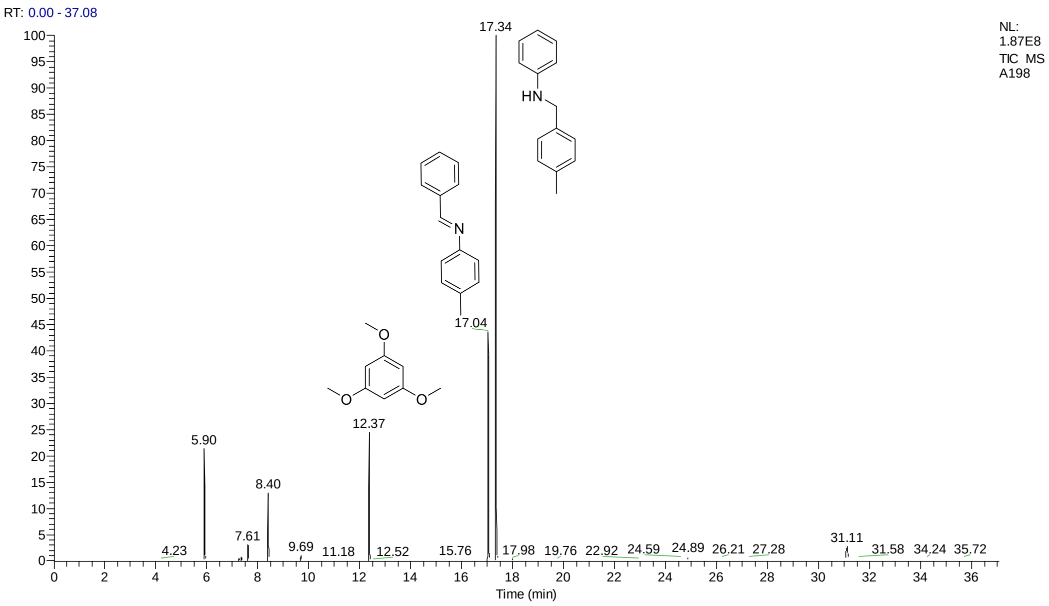

A196 #3578 RT: 16.17 AV: 1 NL: 4.39E7
T: + c EI Full ms [33.00-500.00]
Exact Mass: 183.10
m/z

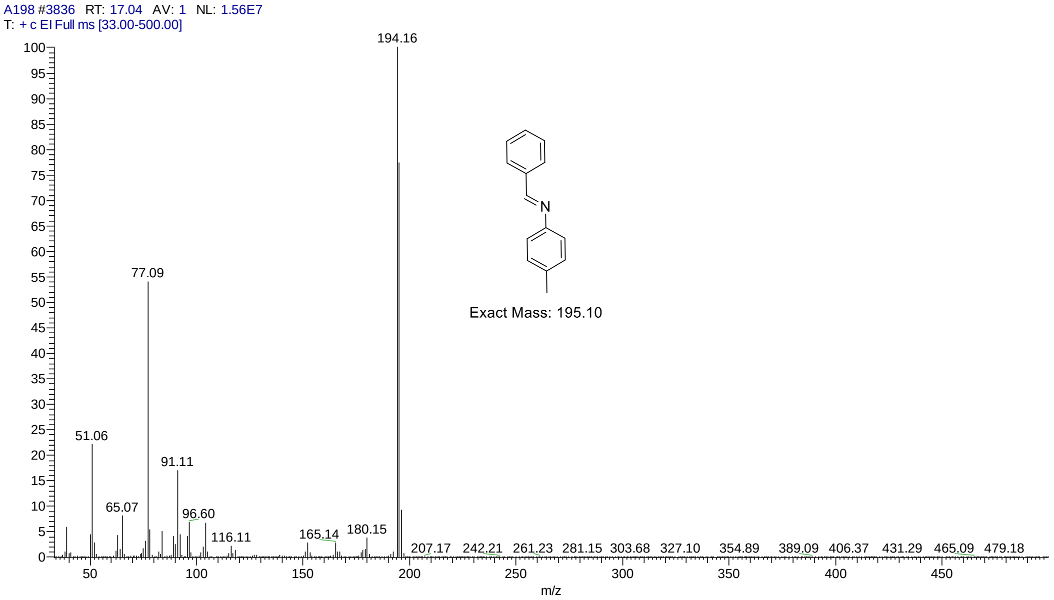
A291 (A198 GC-MS)

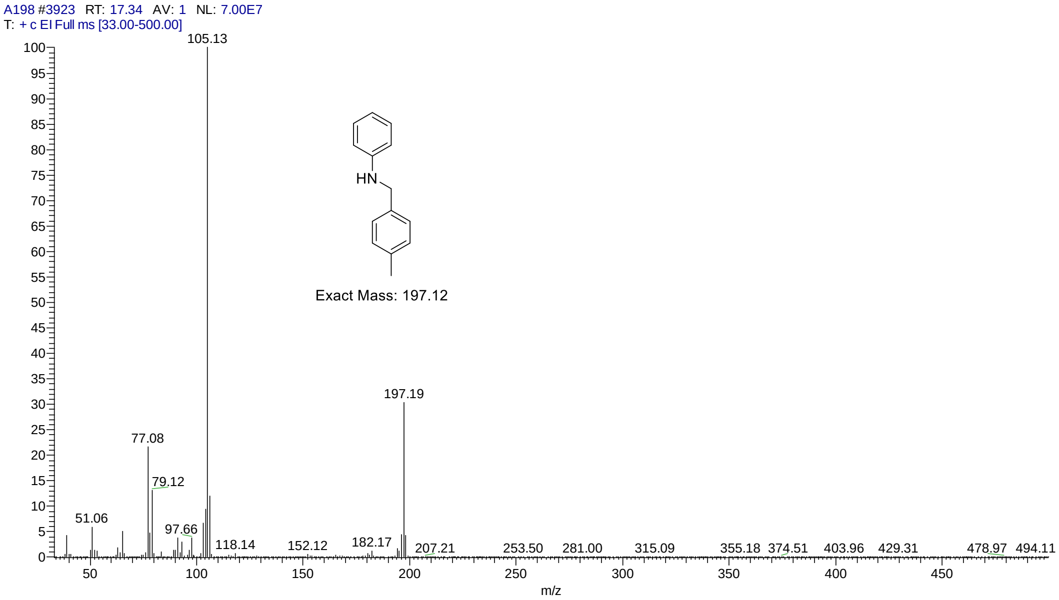




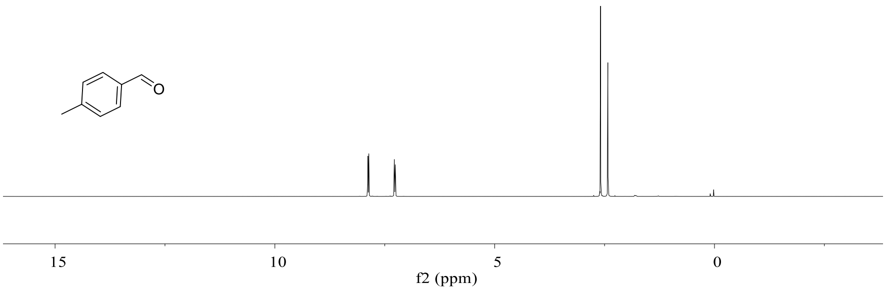


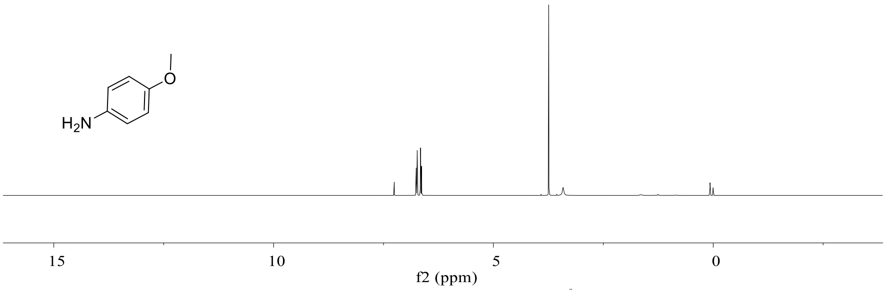


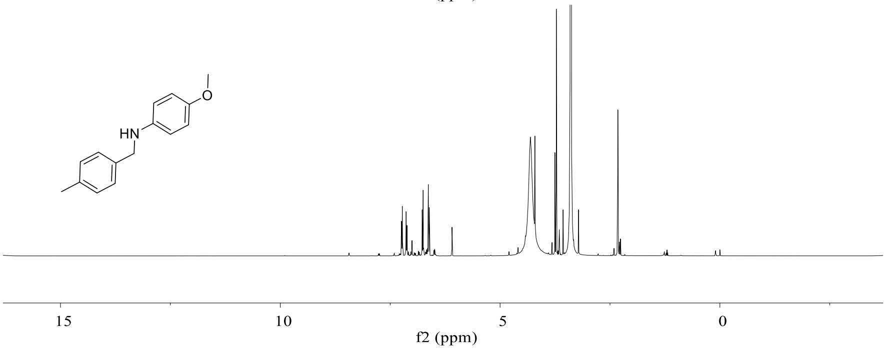

A198 #3836 RT: 17.04 AV: 1 NL: 1.56E7
T: + c EI Full ms [33.00-500.00]
194.16
77.09
51.06
91.11
65.07
96.60
116.11
165.14
180.15
207.17
242.21
261.23
281.15
303.68
327.10
354.89
389.09
406.37
431.29
465.09
479.18
m/z
N
Exact Mass: 195.10

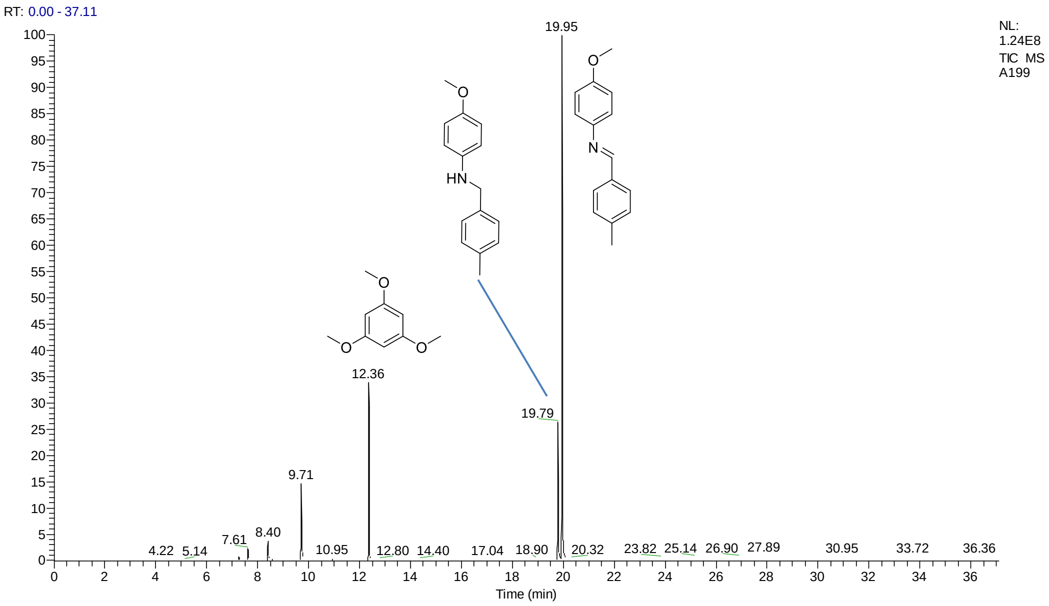

A198 #3923 RT: 17.34 AV: 1 NL: 7.00E7
T: + c EI Full ms [33.00-500.00]
105.13
197.19
77.08
79.12
51.06
97.66
118.14
152.12
182.17
207.21
253.50
281.00
315.09
355.18
374.51
403.96
429.31
478.97
494.11
m/z
HN
Exact Mass: 197.12

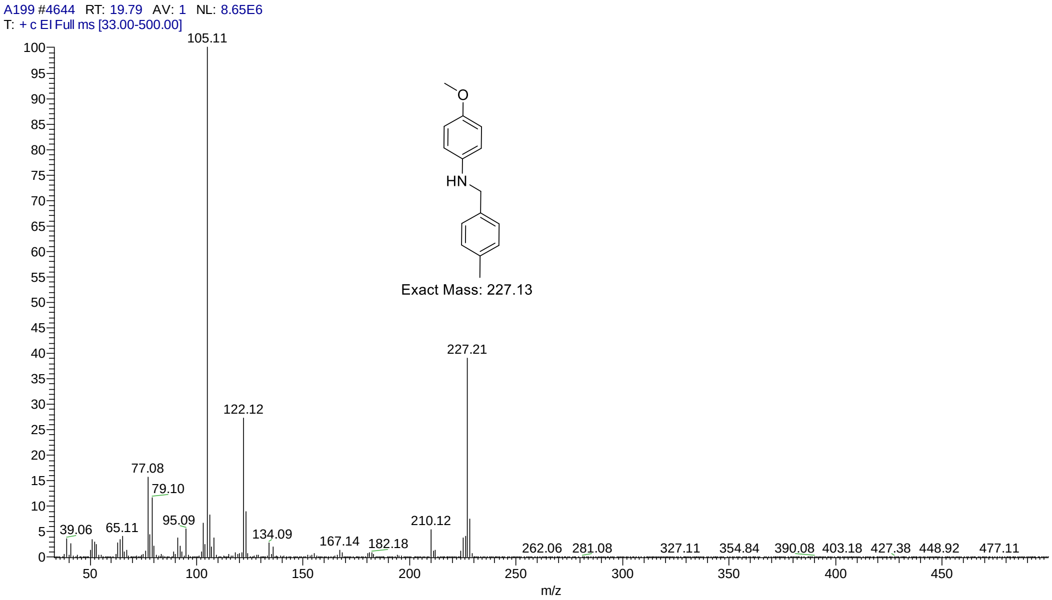
A292(199 GC-MS)

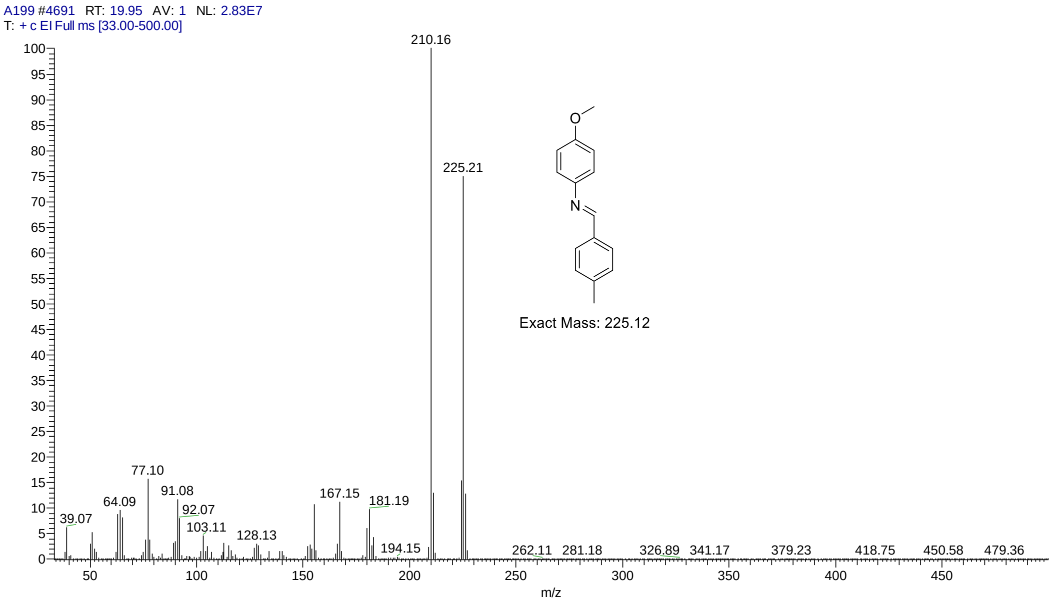




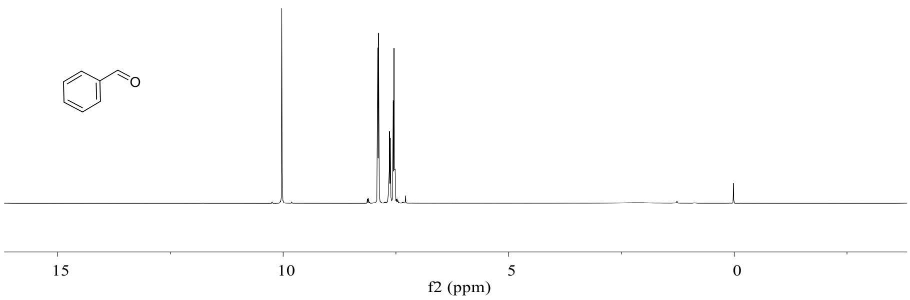


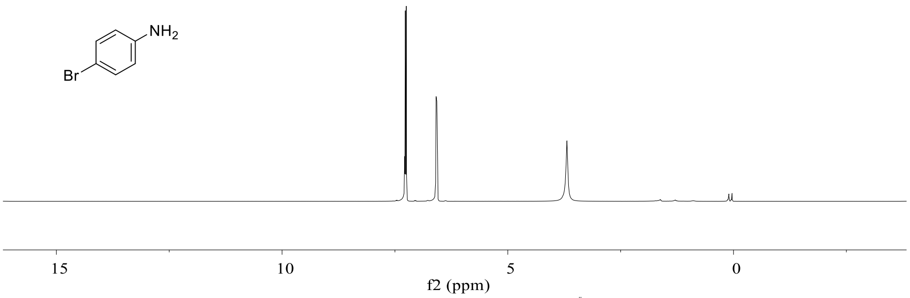


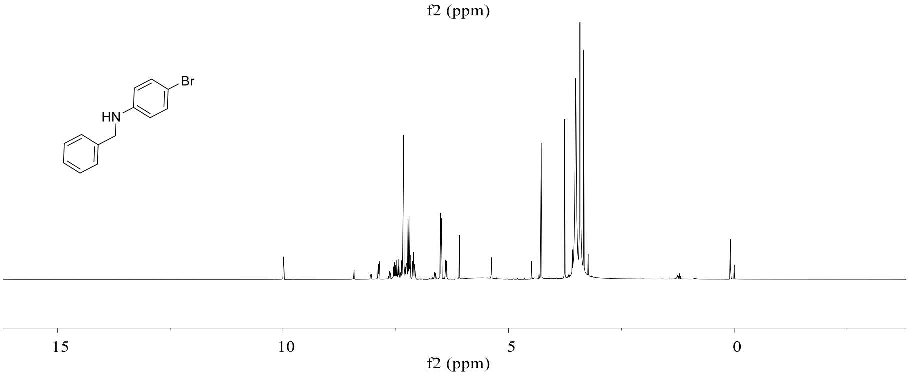

A199 #4644 RT: 19.79 AV: 1 NL: 8.65E6
T: + c EI Full ms [33.00-500.00]
HN
O
Exact Mass: 227.13
105.11
227.21
122.12
77.08
79.10
95.09
65.11
39.06
134.09
167.14
182.18
210.12
262.06
281.08
327.11
354.84
390.08
403.18
427.38
448.92
477.11
m/z

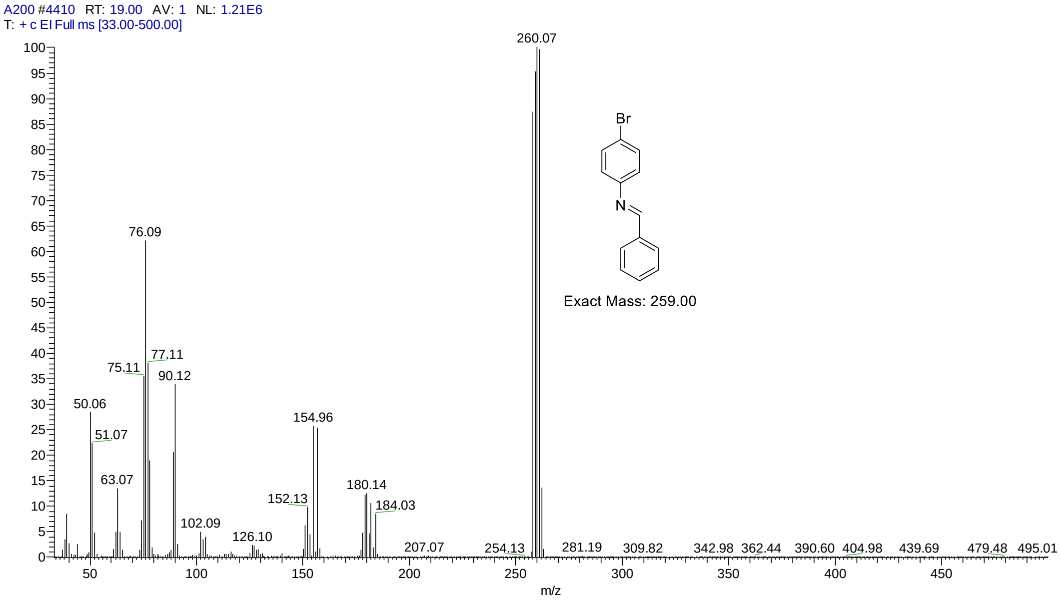

A199 #4691 RT: 19.95 AV: 1 NL: 2.83E7
T: + c EI Full ms [33.00-500.00]
O
N
Exact Mass: 225.12
210.16
225.21
77.10
64.09
91.08
92.07
103.11
39.07
128.13
167.15
181.19
194.15
262.11
281.18
326.89
341.17
379.23
418.75
450.58
479.36
m/z

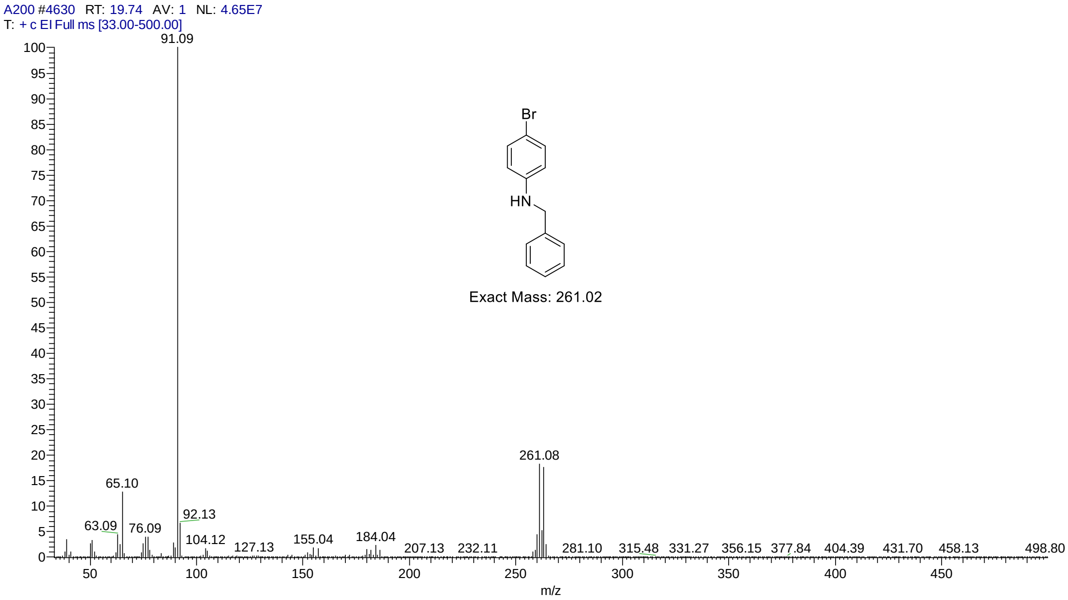
A244 (200 GC-MS)



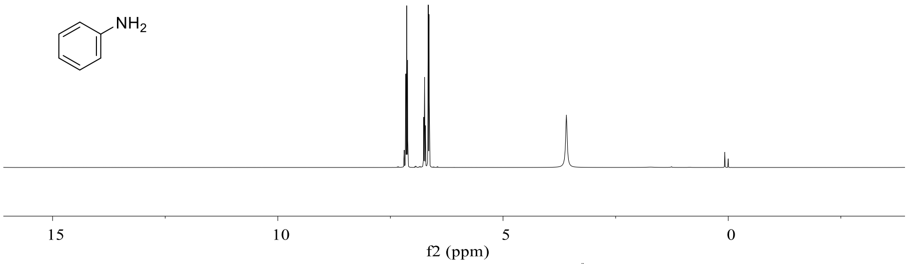


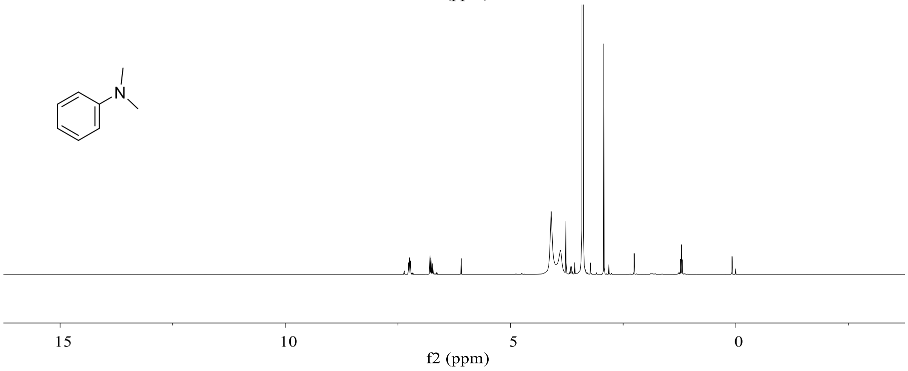



A200 #4410 RT: 19.00 AV: 1 NL: 1.21E6
T: + c EI Full ms [33.00-500.00]
260.07
76.09
77.11
75.11
90.12
50.06
51.07
63.07
154.96
152.13
180.14
184.03
102.09
126.10
207.07
254.13
281.19
309.82
342.98
362.44
390.60
404.98
439.69
479.48
495.01
m/z
Br
N
Exact Mass: 259.00

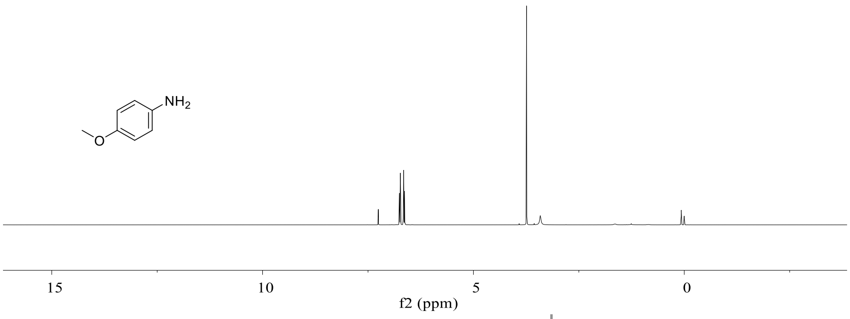

A200 #4630 RT: 19.74 AV: 1 NL: 4.65E7
T: + c EI Full ms [33.00-500.00]
91.09
261.08
65.10
63.09
76.09
92.13
104.12
127.13
155.04
184.04
207.13
232.11
281.10
315.48
331.27
356.15
377.84
404.39
431.70
458.13
498.80
m/z
Br
HN
Exact Mass: 261.02

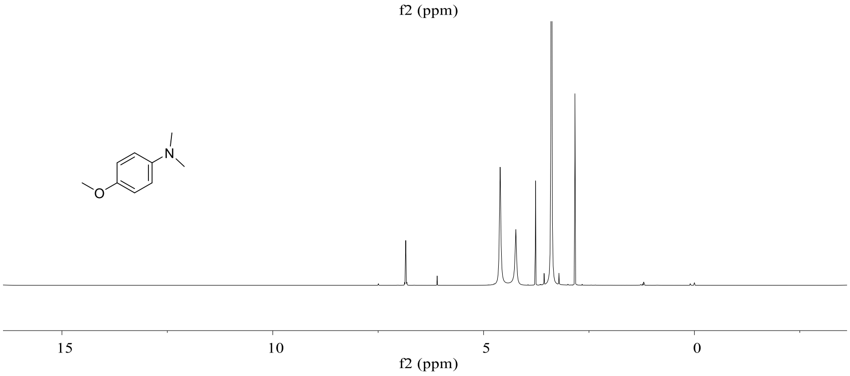
A335

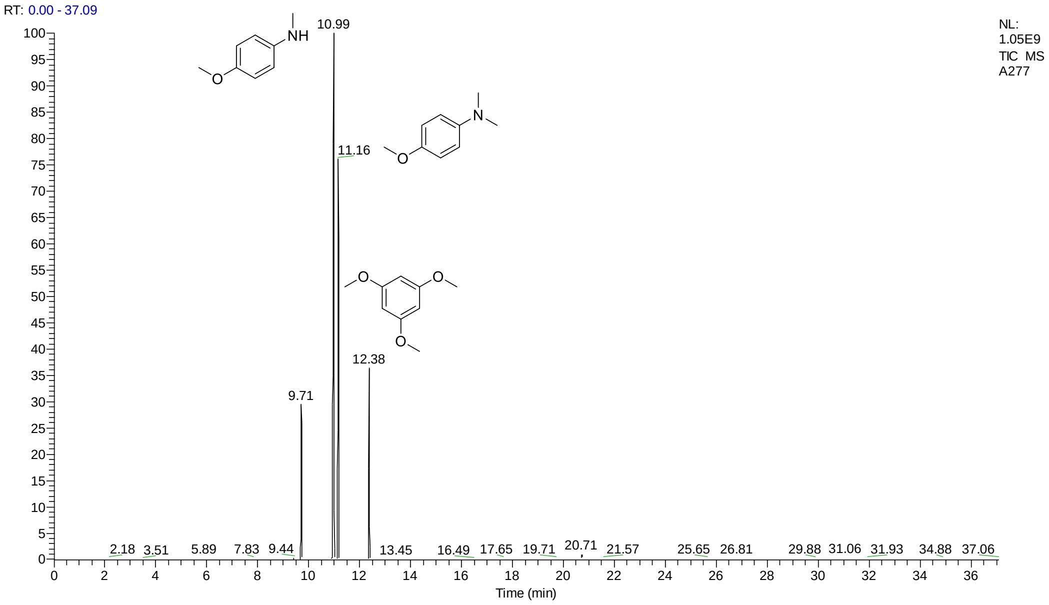


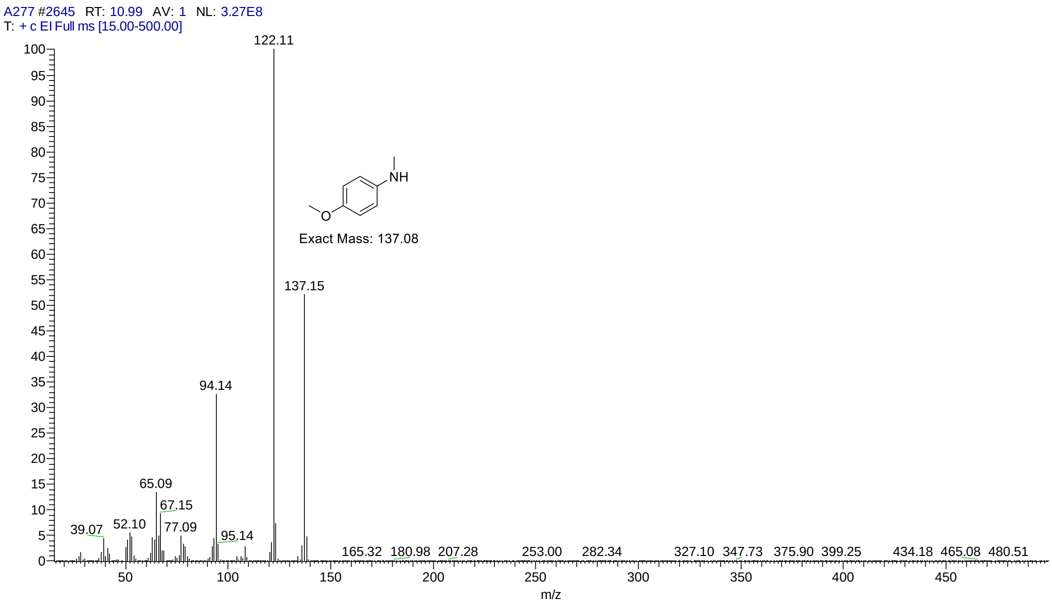


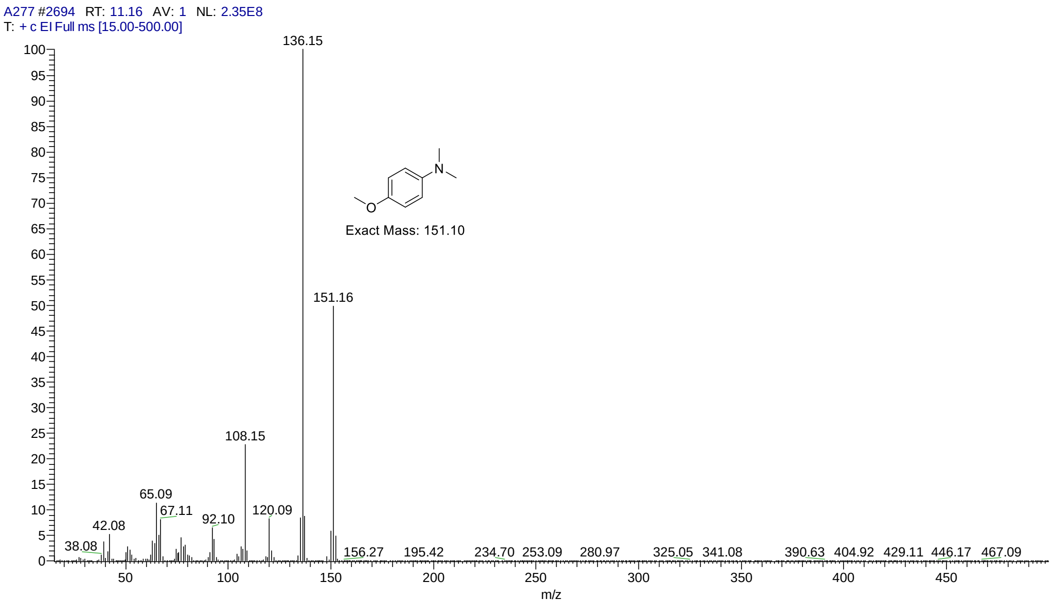
A334 (277 GC-MS)



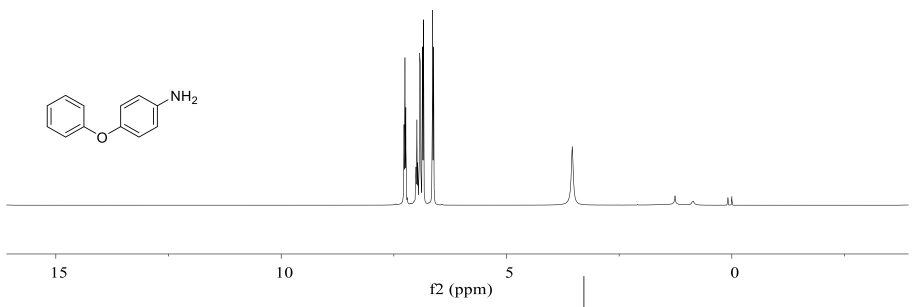


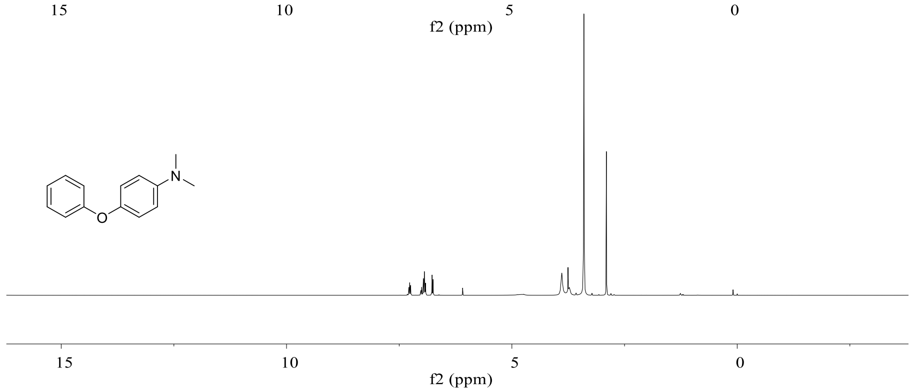


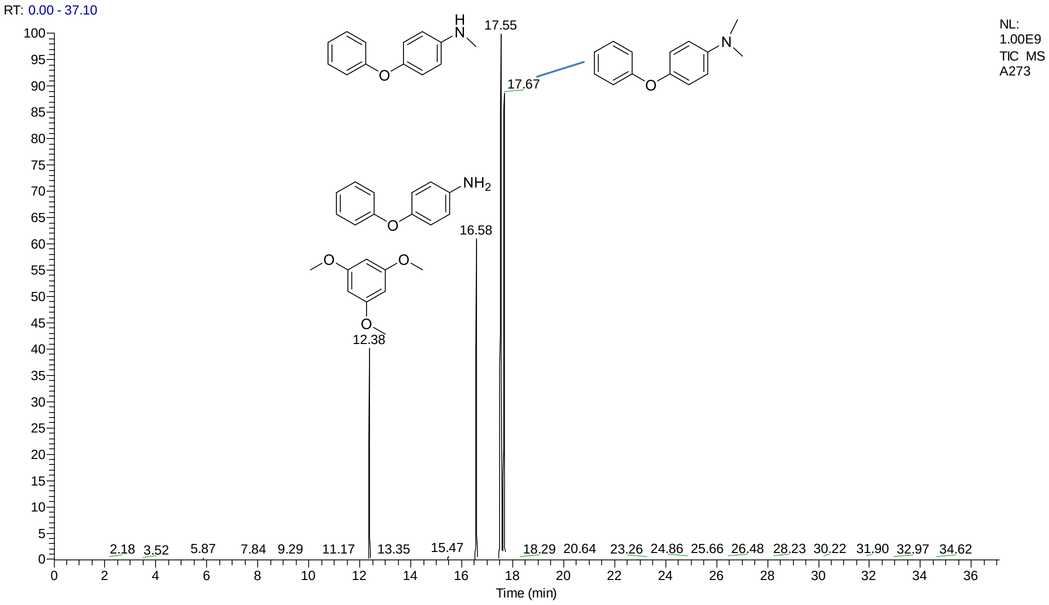

A277 #2645 RT: 10.99 AV: 1 NL: 3.27E8
T: + c EI Full ms [15.00-500.00]
NH
O
Exact Mass: 137.08
122.11
137.15
94.14
65.09
67.15
39.07
52.10
77.09
95.14
165.32 180.98 207.28 253.00 282.34 327.10 347.73 375.90 399.25 434.18 465.08 480.51
m/z

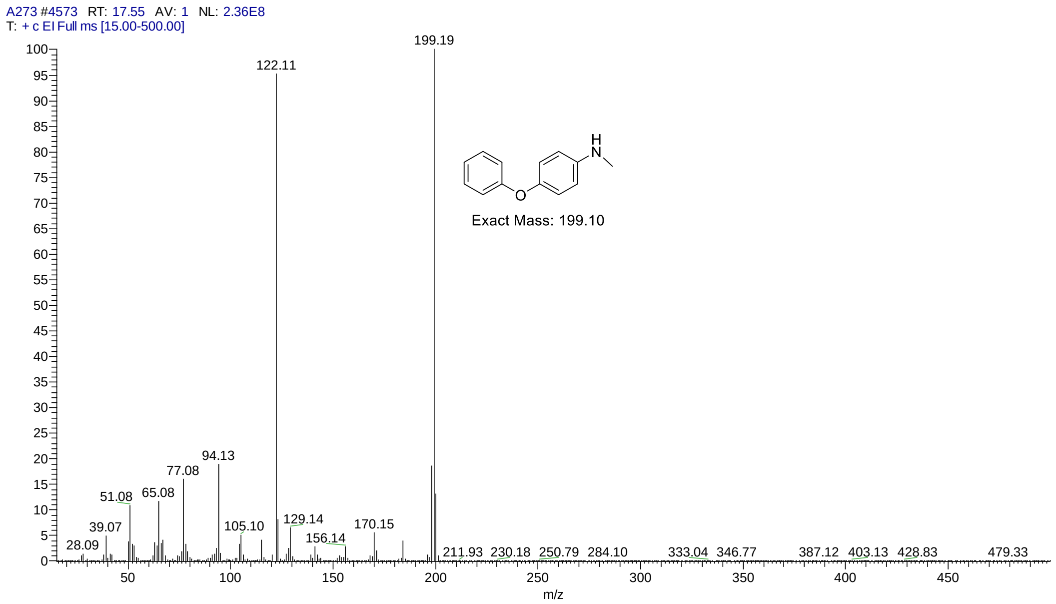

A277 #2694 RT: 11.16 AV: 1 NL: 2.35E8
T: + c EI Full ms [15.00-500.00]
N
O
Exact Mass: 151.10
136.15
151.16
108.15
65.09
67.11
42.08
92.10
120.09
38.08
156.27 195.42 234.70 253.09 280.97 325.05 341.08 390.63 404.92 429.11 446.17 467.09
m/z

A332 (A273 GC-MS)

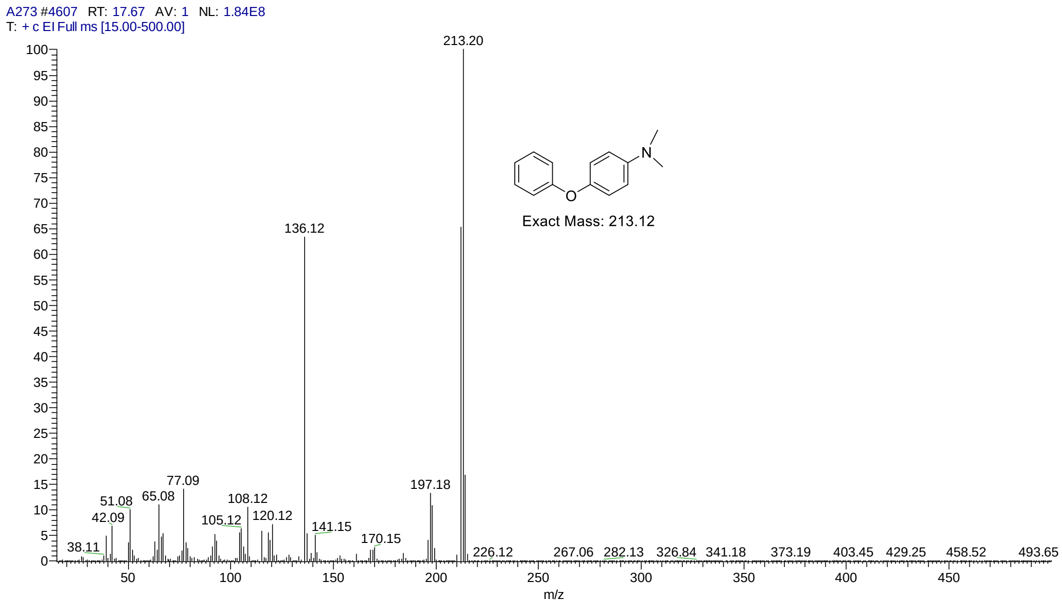






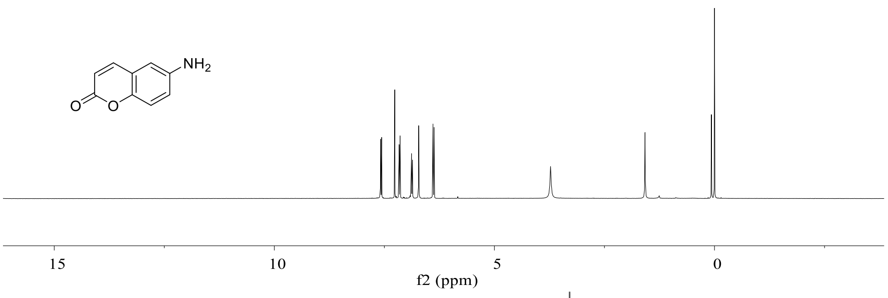


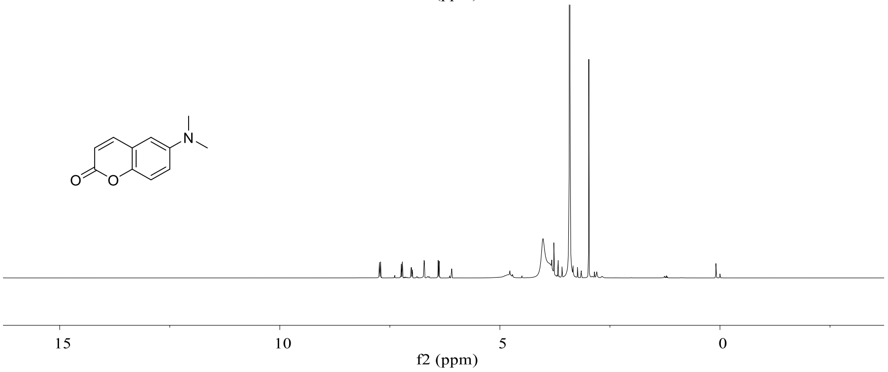
A273 #4573 RT: 17.55 AV: 1 NL: 2.36E8
T: + c EI Full ms [15.00-500.00]
199.19
122.11
94.13
77.08
65.08
51.08
39.07
28.09
105.10
129.14
156.14
170.15
211.93 230.18 250.79 284.10 333.04 346.77 387.12 403.13 428.83 479.33
Exact Mass: 199.10
m/z

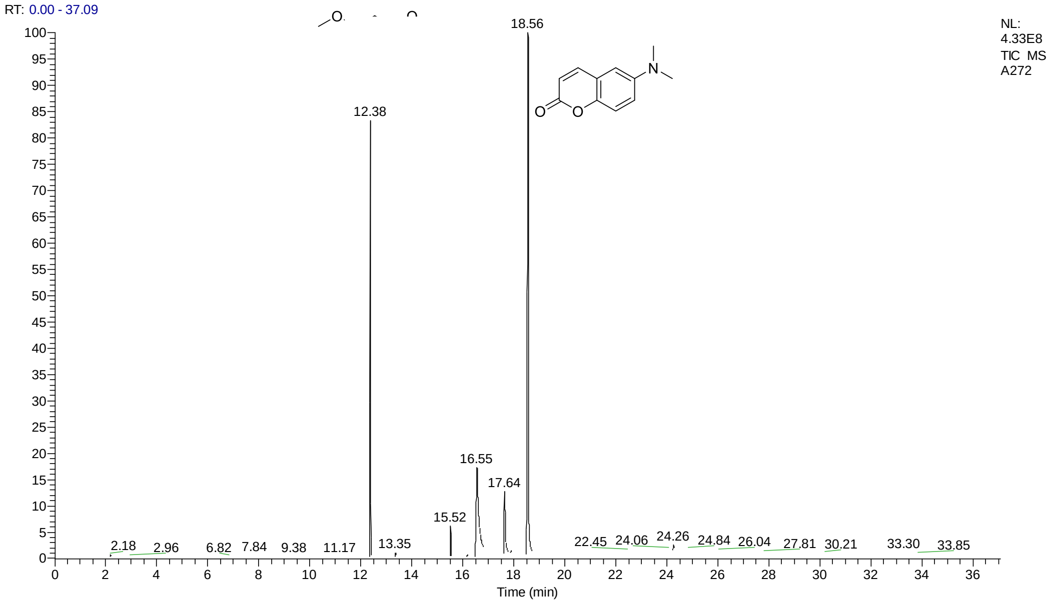


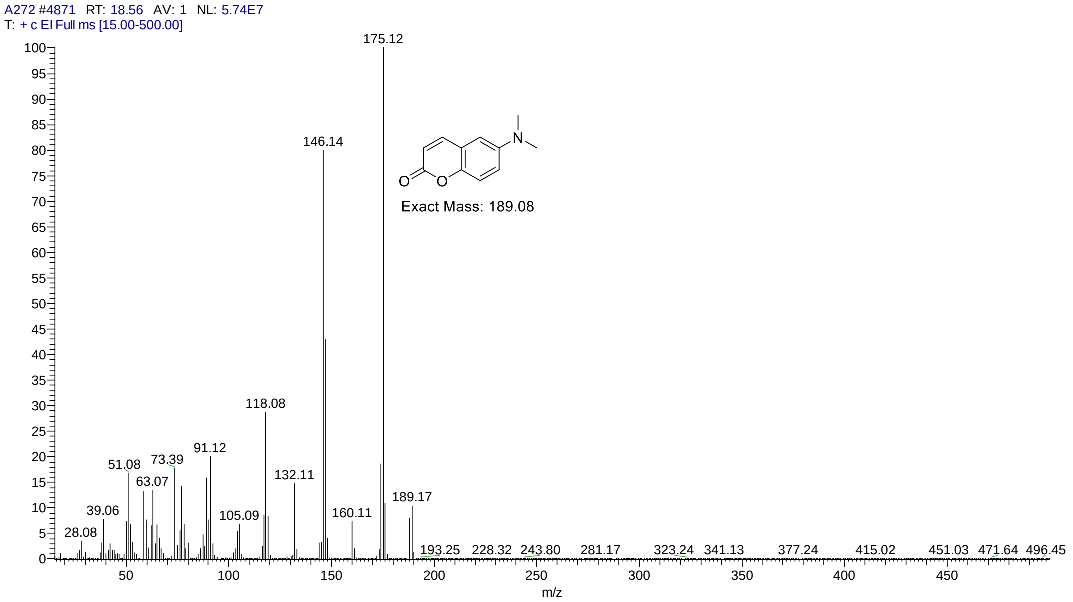
A273 #4607 RT: 17.67 AV: 1 NL: 1.84E8
T: + c EI Full ms [15.00-500.00]
213.20
136.12
197.18
77.09
108.12
51.08
65.08
42.09
38.11
105.12
120.12
141.15
170.15
226.12 267.06 282.13 326.84 341.18 373.19 403.45 429.25 458.52 493.65
Exact Mass: 213.12
m/z

A337 (A272 GC-MS)









A333