Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Efficient Synthesis of Pyrrolo[1,2-a]quinoxalines Catalyzed by Brønsted Acid through Cleavage of C-C Bond

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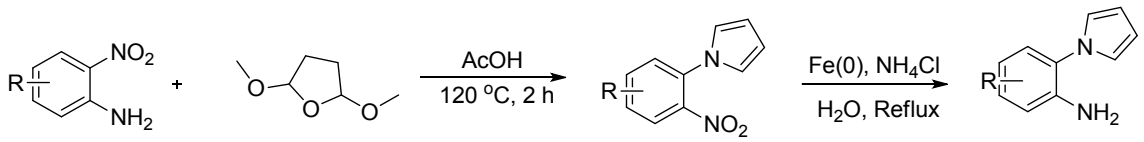
Supporting Information

Table of contents

1. Preparation of starting materials

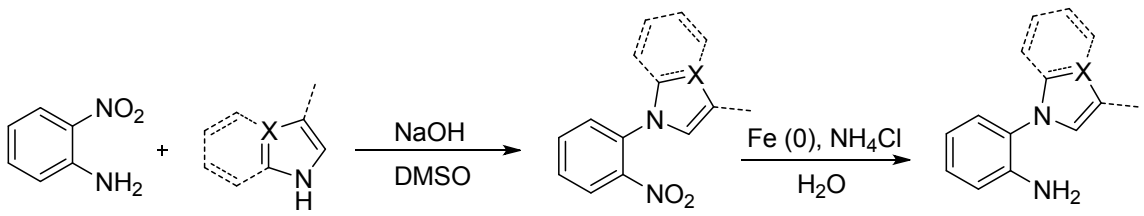
1.1 General procedure for preparation of 2-(1*H*-pyrrol-1-yl)anilines.



Scheme 1 General procedure for preparation of 2-(1*H*-pyrrol-1-yl)anilines.

2-(1*H*-pyrrol-1-yl)anilines were prepared according to a modified literature procedure.[1] A mixture of substituted 2-nitroaniline (20 mmol) and 2,5-dimethoxytetrahydrofuran(20 mmol) in acetic acid (100 mL) was refluxed for 2 h with vigorous stirring. After cooling, the reaction mixture was poured into water (300 mL) and extracted with EtOAc three times (3×50 mL). The combined organic layers were dried with $MgSO_4$ and the solvent was removed in vacuo to afford a residue. The residue was added to iron powder (80 mmol) and NH_4Cl (20mmol) in water (50 mL) and reflux for 4 h. After cooling, the reaction mixture was poured into water (300 mL) and extracted with ethyl acetate time times (3×50 mL). The combined organic layers were dried with $MgSO_4$ and the solvent was removed in vacuo to afford a residue. The residue was purified by column chromatography on silica gel using petroleum ether / EtOAc as eluent to provide the desired product. The spectra data are shown in **2** part.

1.2 General procedure for preparation of 2-(1*H*-indol-1-yl)aniline.

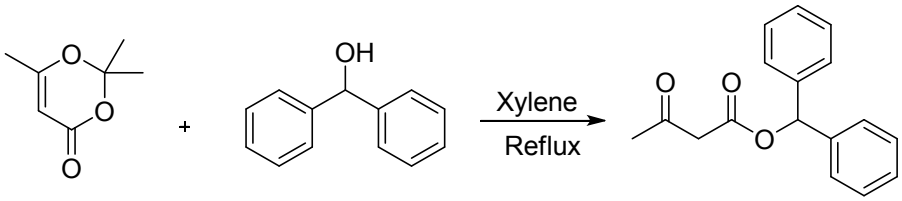


Scheme 2 General procedure for preparation of 2-(1*H*-indol-1-yl)anilines

2-(1*H*-indol-1-yl)anilines were prepared according to a modified literature procedure.[2] A

mixture of 2-nitroaniline (2 mmol) , N-heterocycle (2 mmol) and NaOH (2 mmol) in DMSO (4 mL) was stirred vigorously for 2 h. After cooling, the reaction mixture was poured into water (30 mL) and extracted with EtOAc three times (3×30 mL). The combined organic layers were dried with $MgSO_4$ and the solvent was removed in vacuo to afford a residue. The residue was added to iron powder (16 mmol) and NH_4Cl (1 mmol) in water (30 mL) and refluxed for 4 h. After cooling, the reaction mixture was poured into water (100 mL) and extracted with ethyl acetate twice (3×30 mL). The combined organic layers were dried with $MgSO_4$ and the solvent was removed in vacuo to afford a residue. The residue was purified by column chromatography on silica gel using petroleum ether / ethyl acetate as eluent to provide the desired product. The spectra data are shown in **2** part.

1.3 General procedure for preparation of Benzhydryl 3-oxobutanoate



Scheme 3 General procedure for preparation of Benzhydryl 3-oxobutanoate

Benzhydryl 3-oxobutanoate was prepared according to a modified literature procedure.[3] In a 100 mL round-bottom flask, equipped with a magnetic stir bar and a reflux condenser, 2,2,6-trimethyl-4*H*-1,3-dioxin-4-one (2 mmol, 0.28 g) and benzyl alcohol (2 mmol, 0.36 g) was dissolved in 5 mL xylene. The reaction was heated to vigorous reflux for 4 h, then cooled to room temperature. Xylene was removed *in vacuo*. The residue was purified by column chromatography on silica gel using petroleum ether / ethyl acetate as eluent to provide the desired product. This compound is literature known.[3]

2. Spectra data of starting materials

2-(1*H*-pyrrol-1-yl)aniline (1a)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.16-7.12 (m, 2H), 6.82 (t, *J* = 2.1 Hz, 2H)， 6.79-6.75 (m, 2H), 6.33 (t, *J* = 2.0, 2H), 3.68 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 142.07, 128.59, 127.54, 127.20, 121.74, 118.43, 116.13, 109.42；HRMS calcd for $C_{10}H_{10}N_2$ [$(M+H)^+$]: 159.0917; found, 159.0916.

5-methyl-2-(1*H*-pyrrol-1-yl)aniline (1b)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.03 (d, *J* = 7.7 Hz, 1H), 6.80 (t, *J* = 2.1 Hz, 2H), 6.60 (d, *J* = 9.2 Hz, 2H), 6.32 (t, *J* = 2.0Hz, 2H), 3.61 (s, 2H)， 2.29 (s. 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =141.86, 138.62, 126.98, 125.27, 121.86, 119.20, 116.60, 109.24, 21.20; HRMS calcd for $C_{11}H_{12}N_2$ [$(M+H)^+$]: 173.1073; found, 173.1081.

5-chloro-2-(1*H*-pyrrol-1-yl)aniline (1c)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.05 (d, *J* = 8.3 Hz, 1H), 6.78-6.76 (m, 3H), 6.74 (dd, *J* = 8.3 Hz, 2.2 Hz, 1H), 6.34 (t, *J* = 2.06 Hz, 2H), 3.76 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.15, 133.97, 128.17, 125.95, 121.65, 118.23, 115.62, 109.77；HRMS calcd for $C_{10}H_9ClN_2$ [$(M+H)^+$]: 193.0527; found, 193.0528.

5-fluoro-2-(1*H*-pyrrol-1-yl)aniline (1d)

^{1}H NMR (400 MHz, $CDCl_3$): δ =7.08-7.04 (m, 1H), 6.76 (t, *J*=2.0 Hz, 2H), 6.47-6.42 (m, 2H), 6.33 (t, *J* = 2.0 Hz, 2H), 3.78 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 163.98 ($^1J_{C,F}$ = 243 Hz), 143.93 ($^3J_{C,F}$ = 12 Hz), 128.61 ($^3J_{C,F}$ = 11 Hz), 123.64, 121.91, 109.60, 104.93 ($^2J_{C,F}$ = 23 Hz), 102.57 ($^2J_{C,F}$ = 26Hz)；HRMS calcd for $C_{10}H_9FN_2$ [$(M+H)^+$]: 177.0823; found, 177.0827.

5-methoxy-2-(1*H*-pyrrol-1-yl)aniline (1e)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.07 (dd, *J* = 8.8 Hz, 3.6 Hz, 1H), 6.77 (t, *J* = 2.8Hz, 2H), 6.35-6.31 (m, 4H), 3.78 (s, 3H), 3.67 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.94, 143.36, 128.15, 122.12, 121.24, 109.17, 103.66, 102.13, 55.40; HRMS calcd for $C_{11}H_{12}ON_2$ [(M+H)$^+$]: 189.1022; found, 189.1024.

4-chloro-2-(1*H*-pyrrol-1-yl)aniline (1g)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.13-7.09 (m, 2H), 6.80 (m, *J* = 1.8 Hz, 2H), 6.71 (d, *J* = 8.4 Hz, 1H), 6.33 (d, *J* = 2.1 Hz, 2H), 3.70 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 140.67, 128.38, 128.06, 126.98, 122.58, 121.51, 116.94, 109.88; HRMS calcd for $C_{10}H_9ClN_2$ [(M+H)$^+$]: 193.0527; found, 193.0529.

4-fluoro-2-(1*H*-pyrrol-1-yl)aniline (1h)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.93-6.86 (m, 2H), 6.83 (t, *J* = 2.1 Hz, 2H), 6.75 (dd, *J* = 9.6 Hz, 5.1 Hz, 1H), 6.34-6.31 (m, 2H), 3.65 (s,2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 157.14 (($^1J_{C,F}$ = 236 Hz), 137.91, 127.88 ($^3J_{C,F}$ = 9 Hz), 121.53, 116.89 ($^3J_{C,F}$ = 8 Hz), 115.29 ($^2J_{C,F}$ = 23 Hz), 114.10 ($^2J_{C,F}$ = 23 Hz), 109.85; HRMS calcd for $C_{10}H_9FN_2$ [(M+H)$^+$]: 177.0823; found, 177.0821.

2-(1*H*-indol-1-yl)aniline (1i)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.69-7.67 (m, 1H), 7.23-7.14 (m, 6H), 6.83-6.80 (m, 2H), 6.67 (d, J = 3.16 Hz, 1H), 3.49 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.21, 136.42, 129.24, 128.69, 128.62, 124.90, 122.30, 121.04, 120.24, 118.58, 116.31, 110.83, 103.28. HRMS calcd for $C_{14}H_{12}N_2$ [(M+H)$^+$]: 209.1072; found, 209.1071.

2-(3-methyl-1*H*-indol-1-yl)aniline (1j)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.80-7.77 (m, 1H), 7.35-7.24 (m, 5H), 7.09 (s, 1H), 6.96-6.89 (m, 2H), 3.48 (s, 2H), 2.54 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.27, 136.85, 129.14, 129.00, 128.71, 126.28, 125.24, 122.35, 119.69, 119.20, 118.62, 116.37, 112.50, 110.79, 9.81. HRMS calcd for $C_{15}H_{14}N_2$ [(M+H)$^+$]: 223.1230; found, 223.1232.

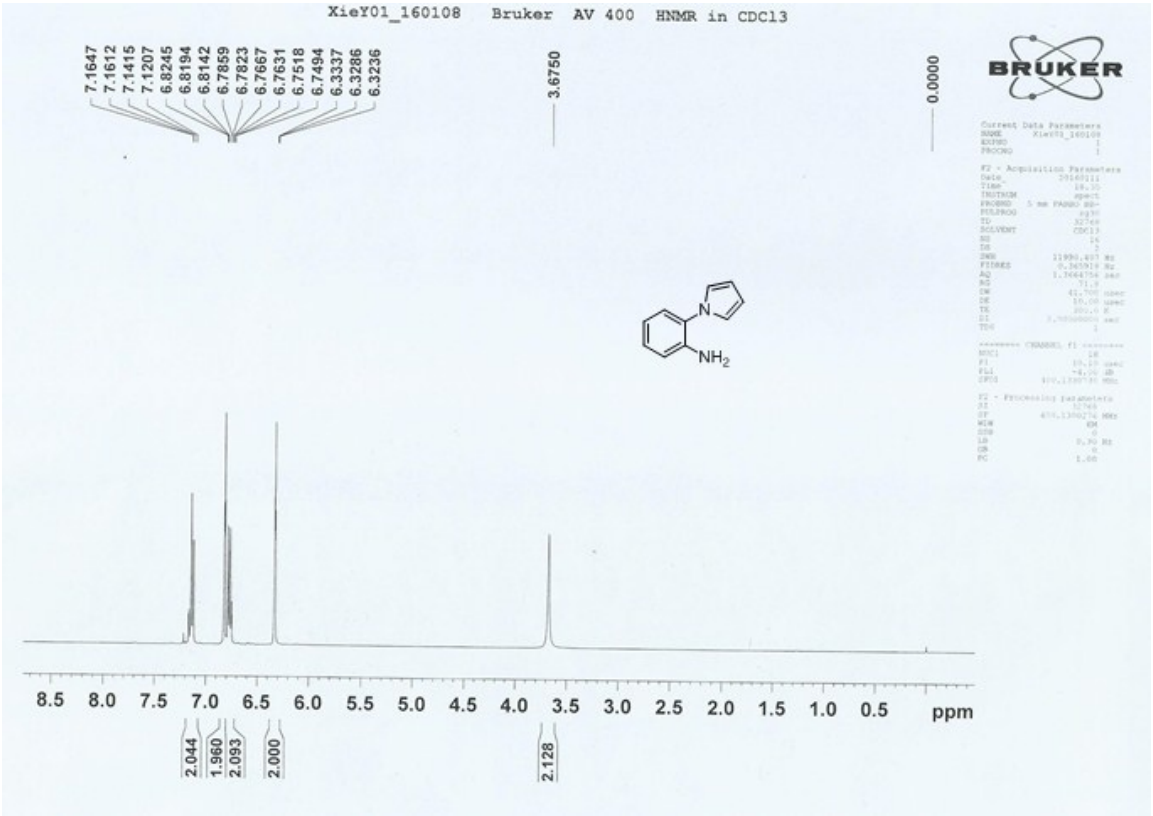
2-(1*H*-imidazol-1-yl)aniline (1k)

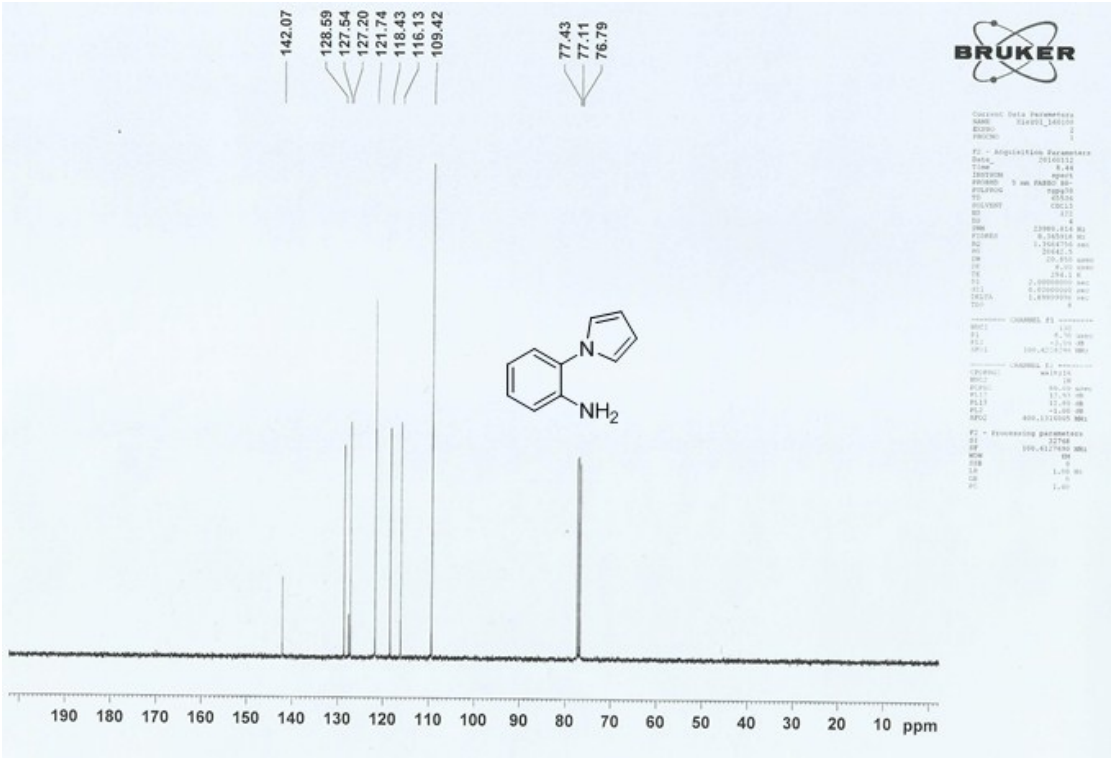
^{1}H NMR (300 MHz, DMSO-d_6): δ =7.75 (s, 1H), 7.30 (s, 1H), 7.17-7.11 (m, 1H), 7.11 (s, 1H), 7.05 (d, J = 7.8 Hz, 1H), 6.89 (d, J = 8.1 Hz, 1H), 6.67 (t, J = 7.5 Hz, 1H), 4.94 (s, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ =143.21, 137.42, 129.02, 128.89, 126.74, 122.44, 120.28, 116.32, 116.05. HRMS calcd for $C_9H_9N_3$ [(M+H)$^+$]: 160.0869; found, 160.0864.

3.^{1}H NMR, ^{13}C NMR and HRMS spectra

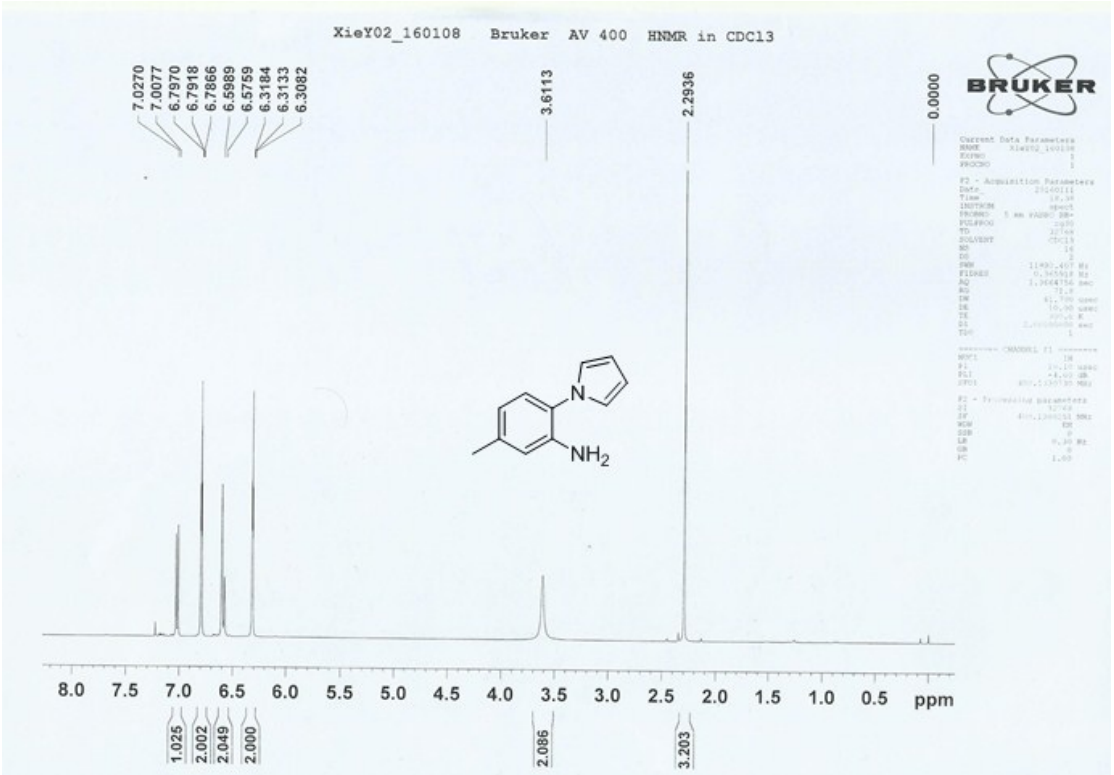
3.1 The spectra of starting materials

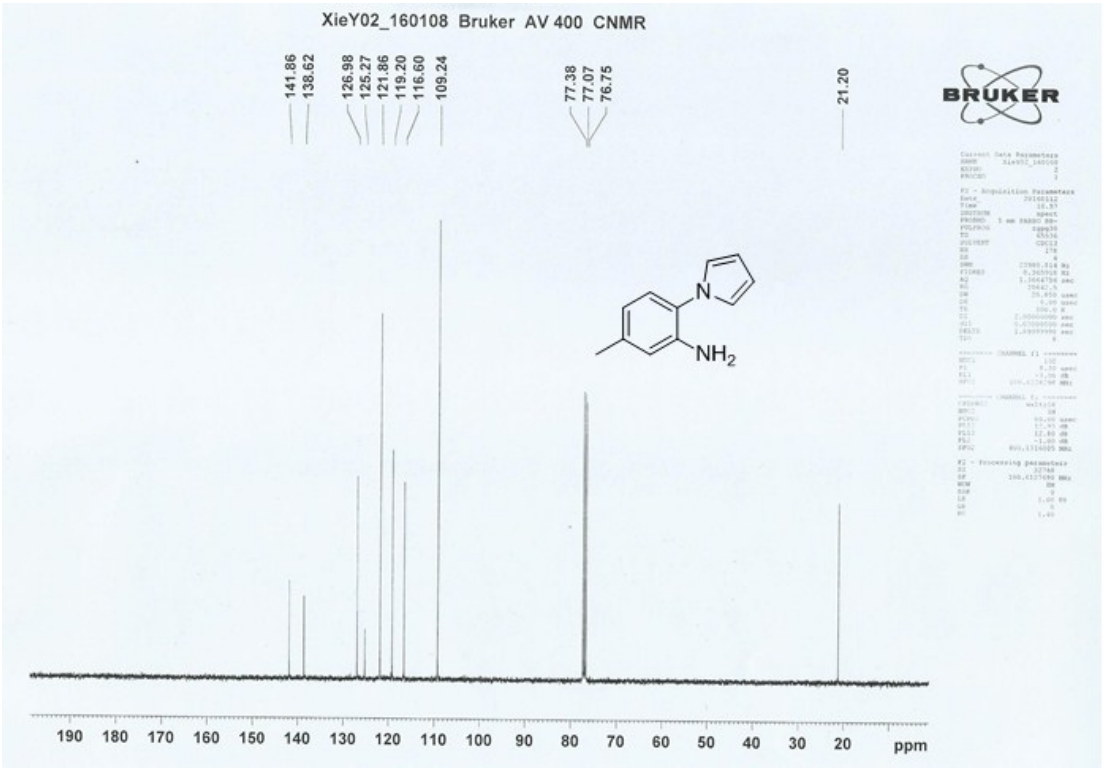
2-(1*H*-pyrrol-1-yl)aniline (1a)



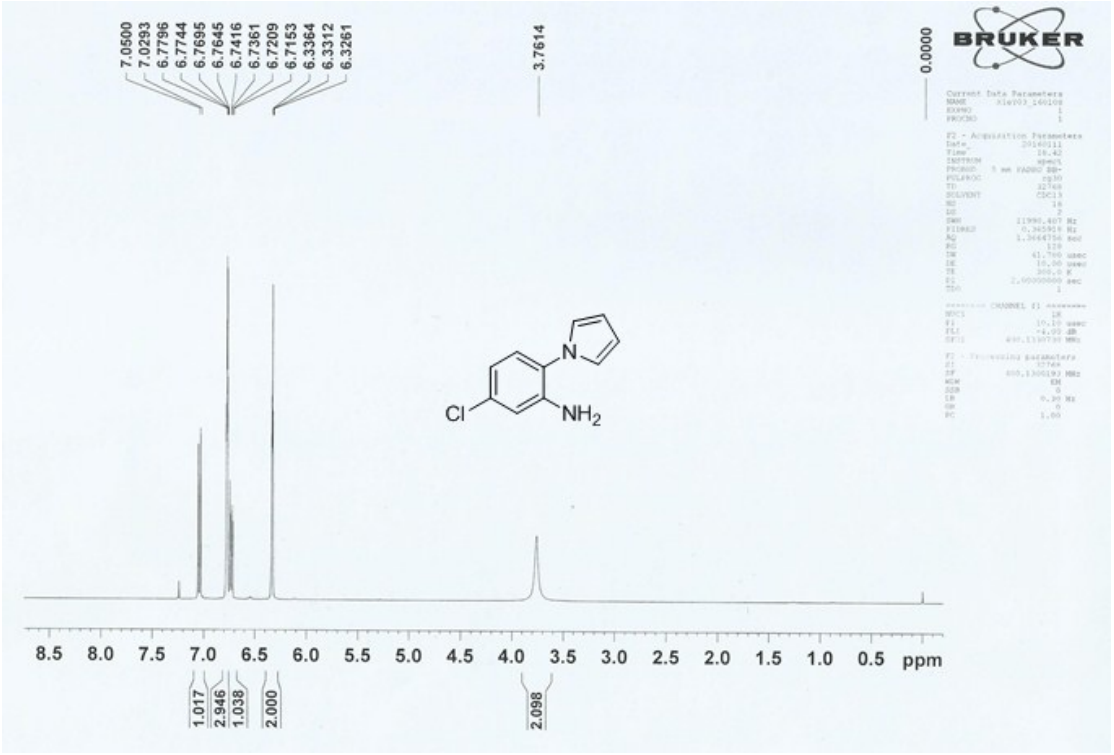


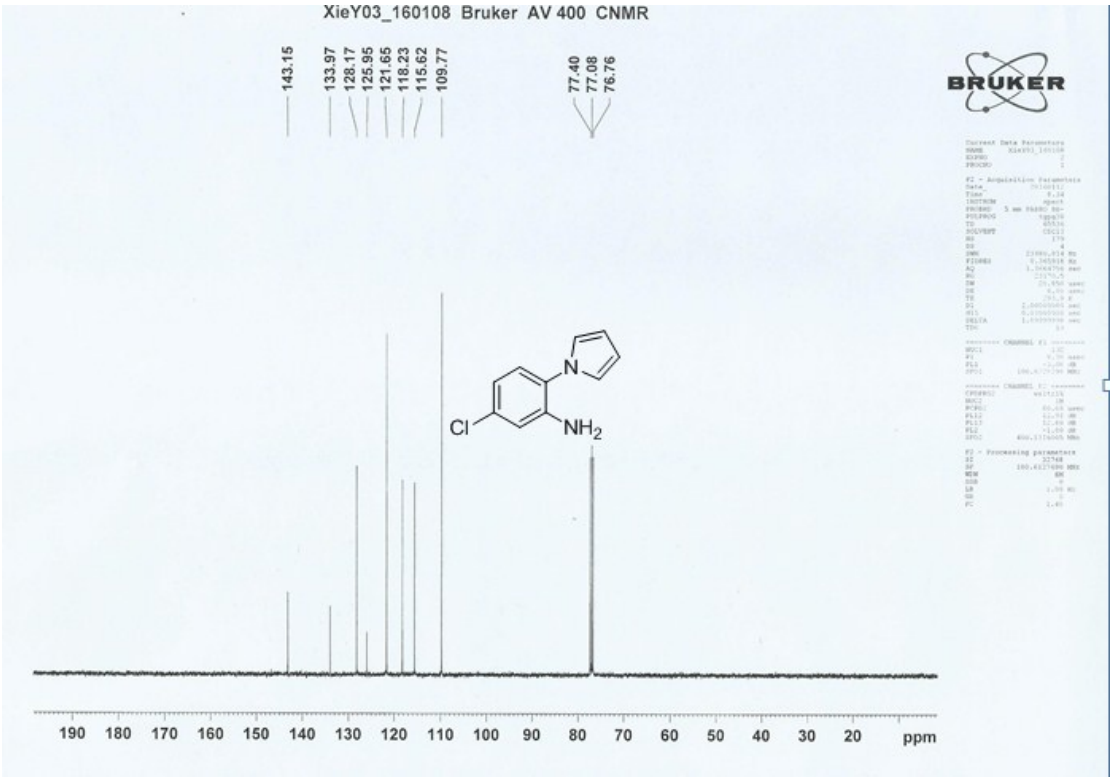
5-methyl-2-(1*H*-pyrrol-1-yl)aniline (1b)



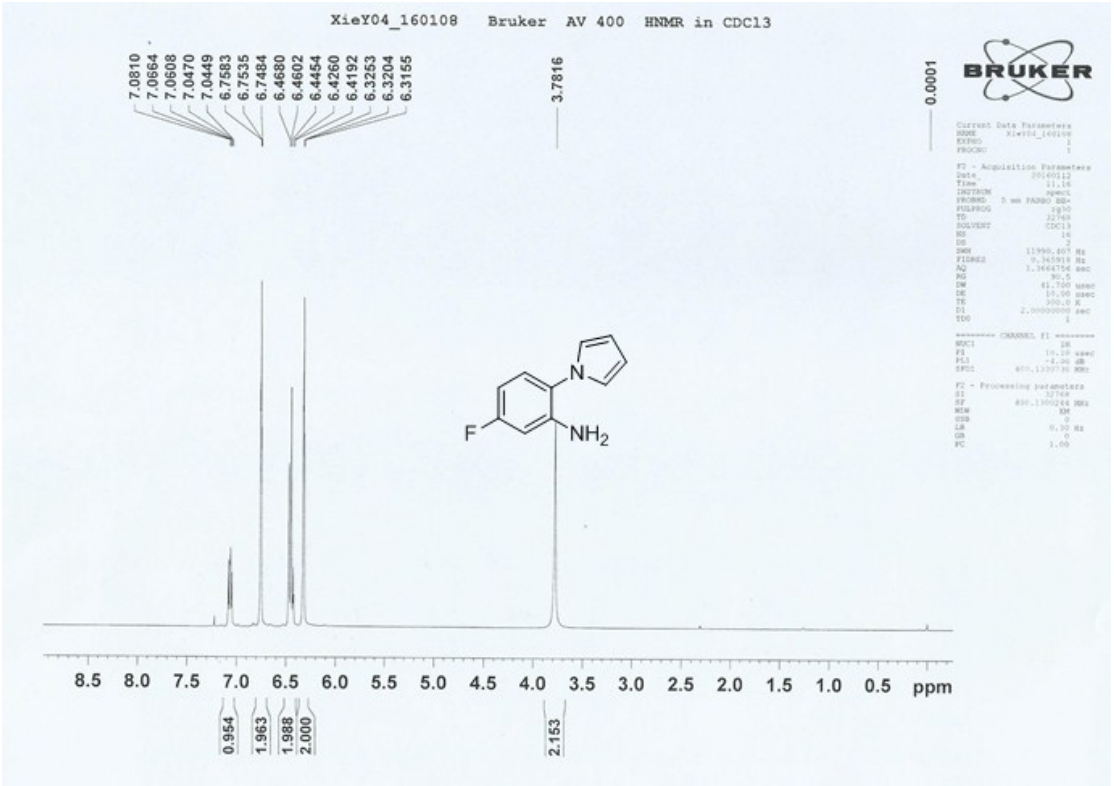


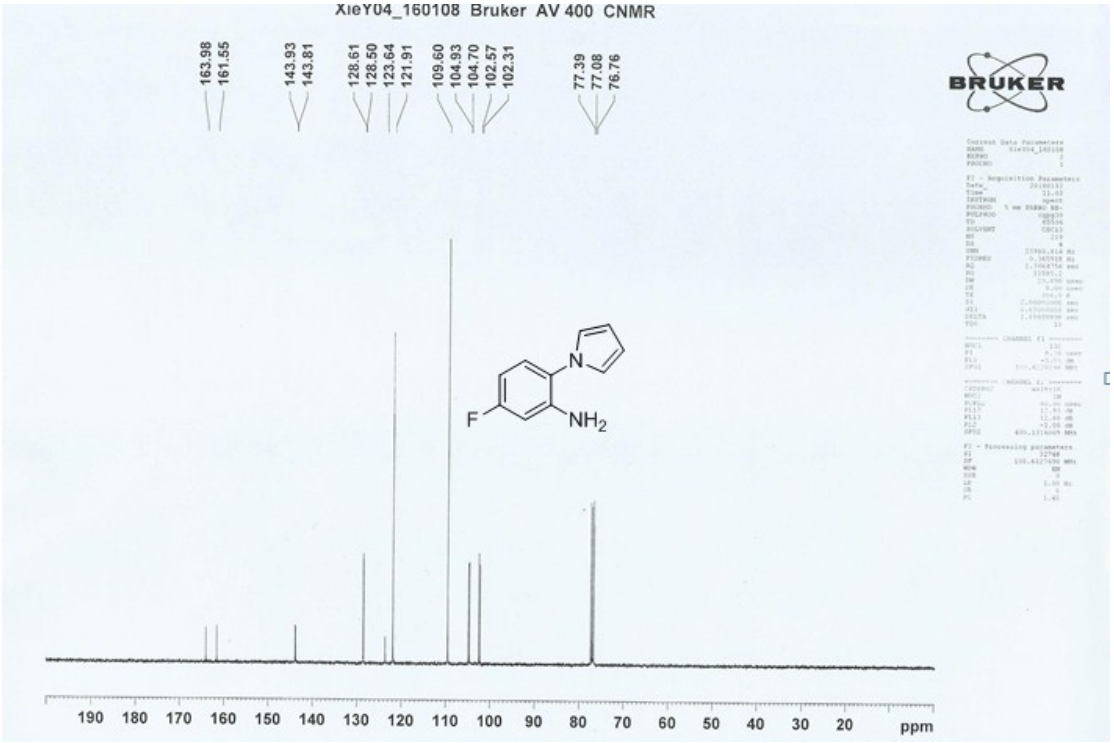
5-chloro-2-(1*H*-pyrrol-1-yl)aniline (1c)





5-fluoro-2-(1*H*-pyrrol-1-yl)aniline (1d)





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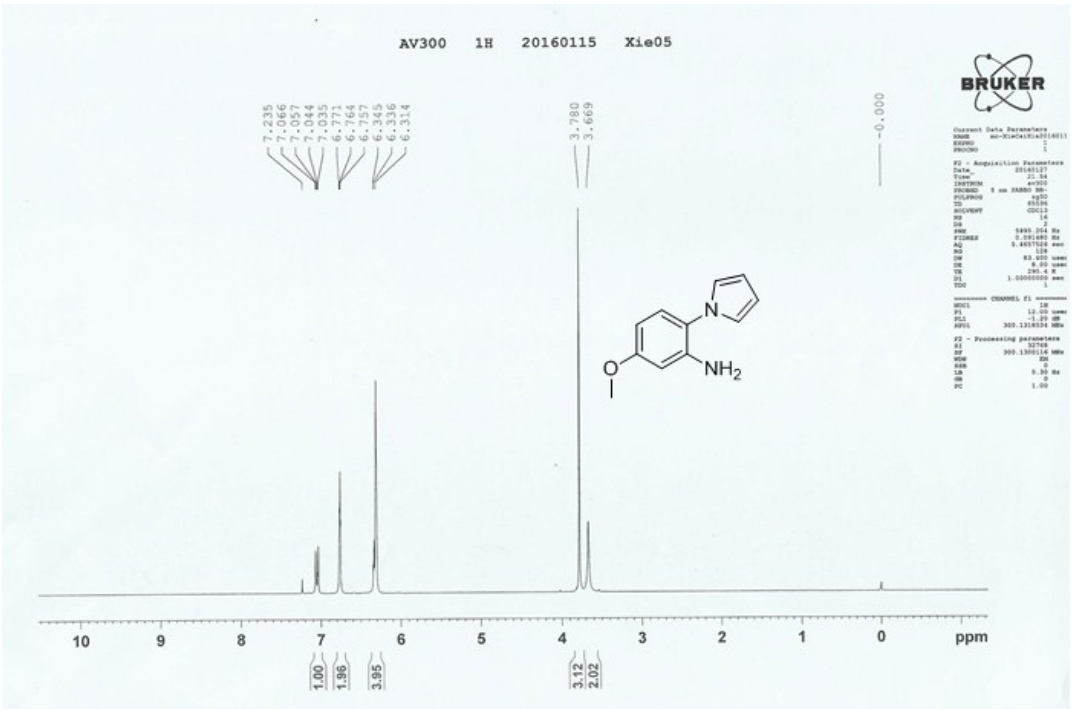

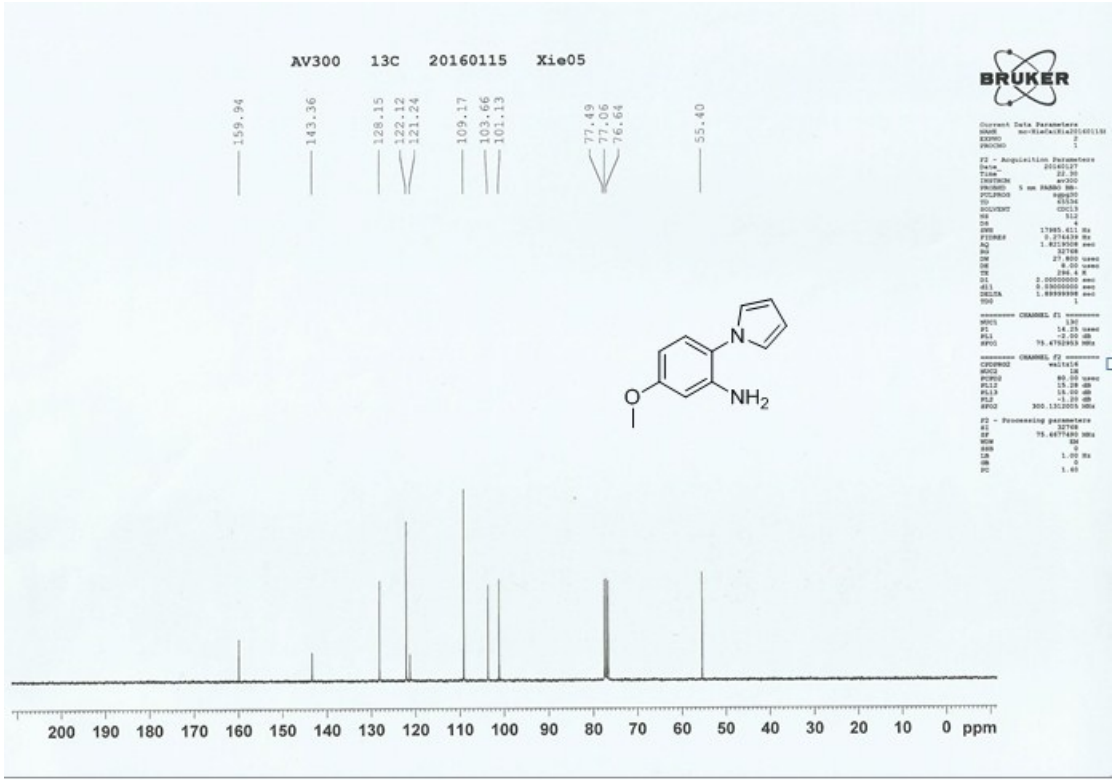

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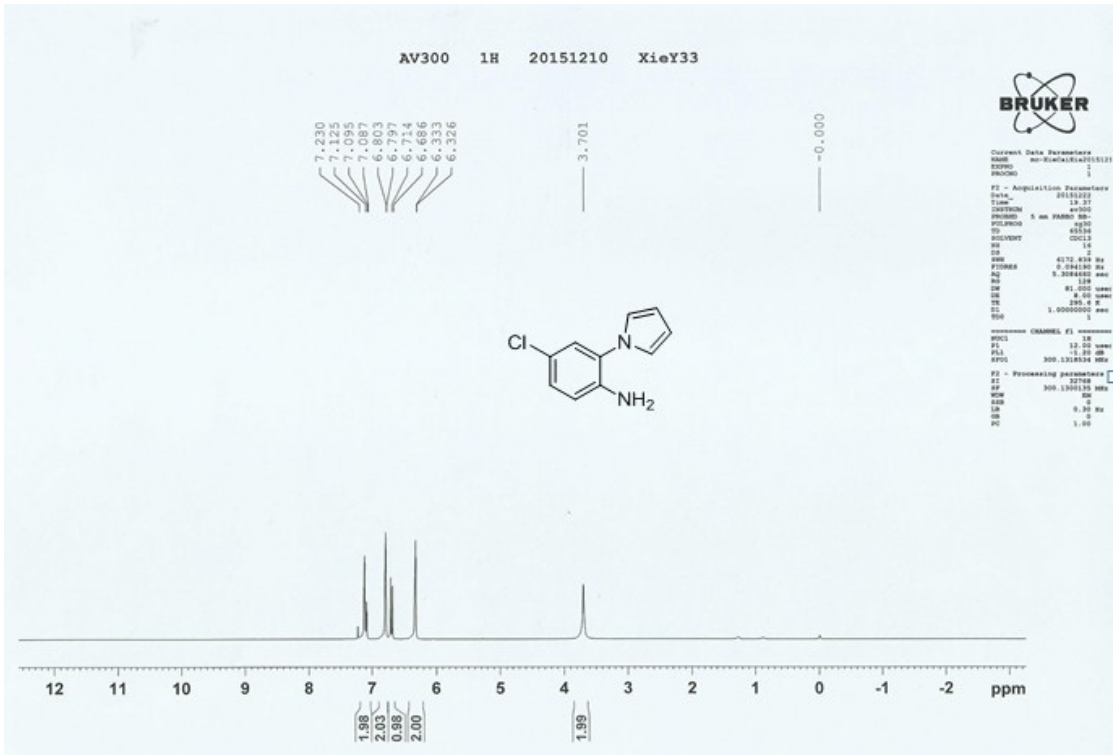

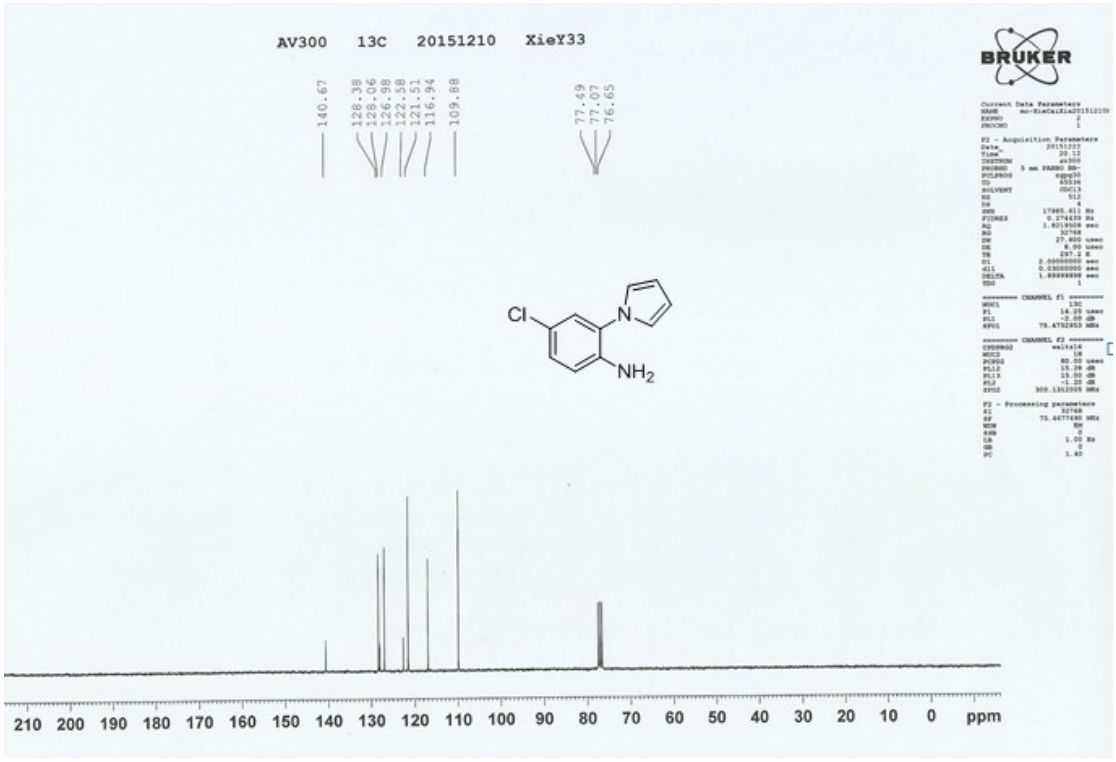

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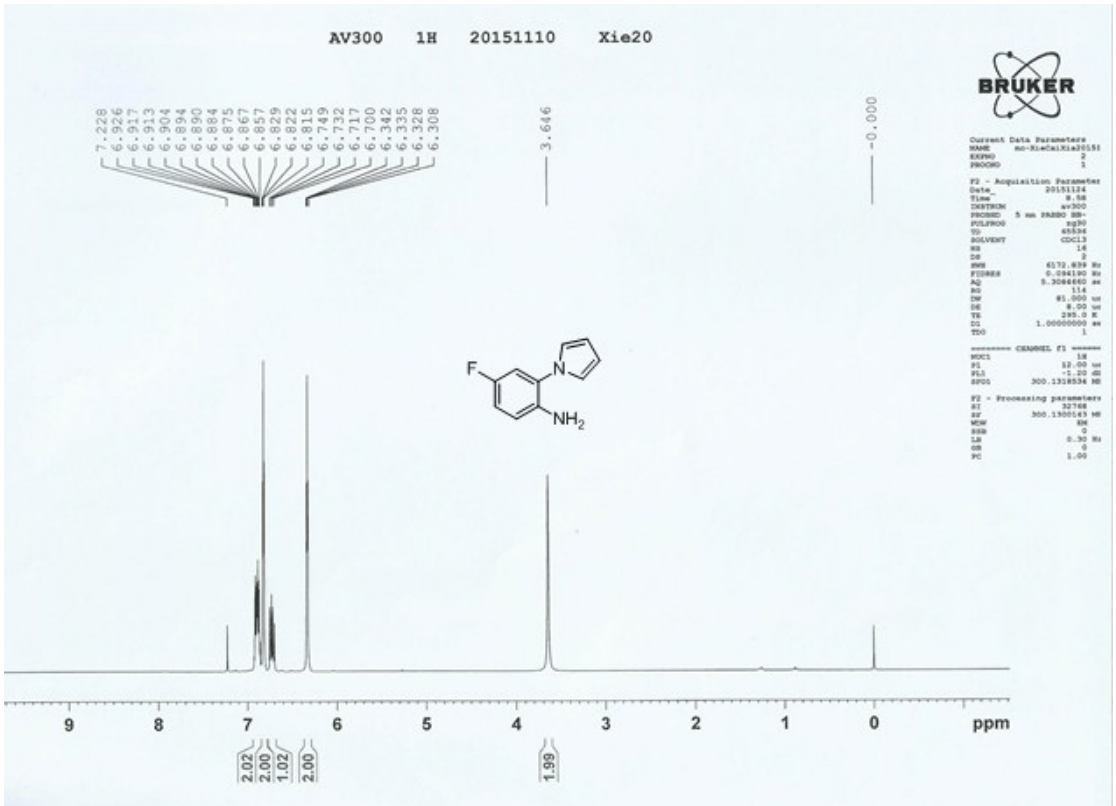
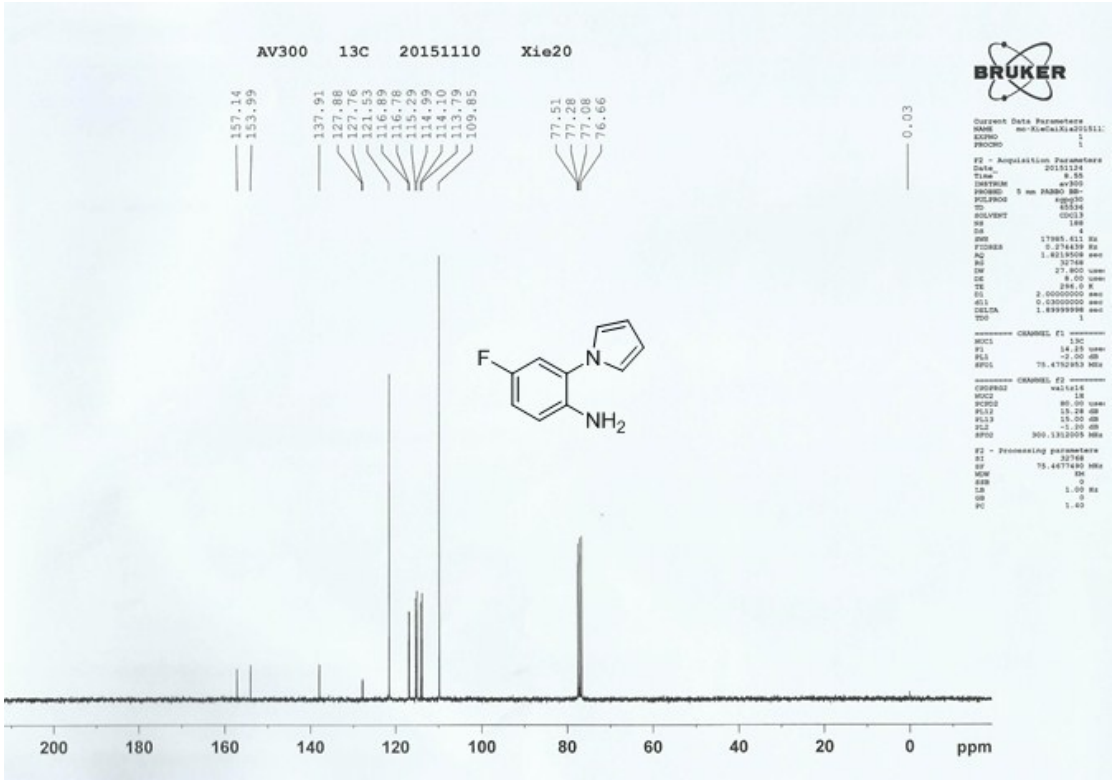
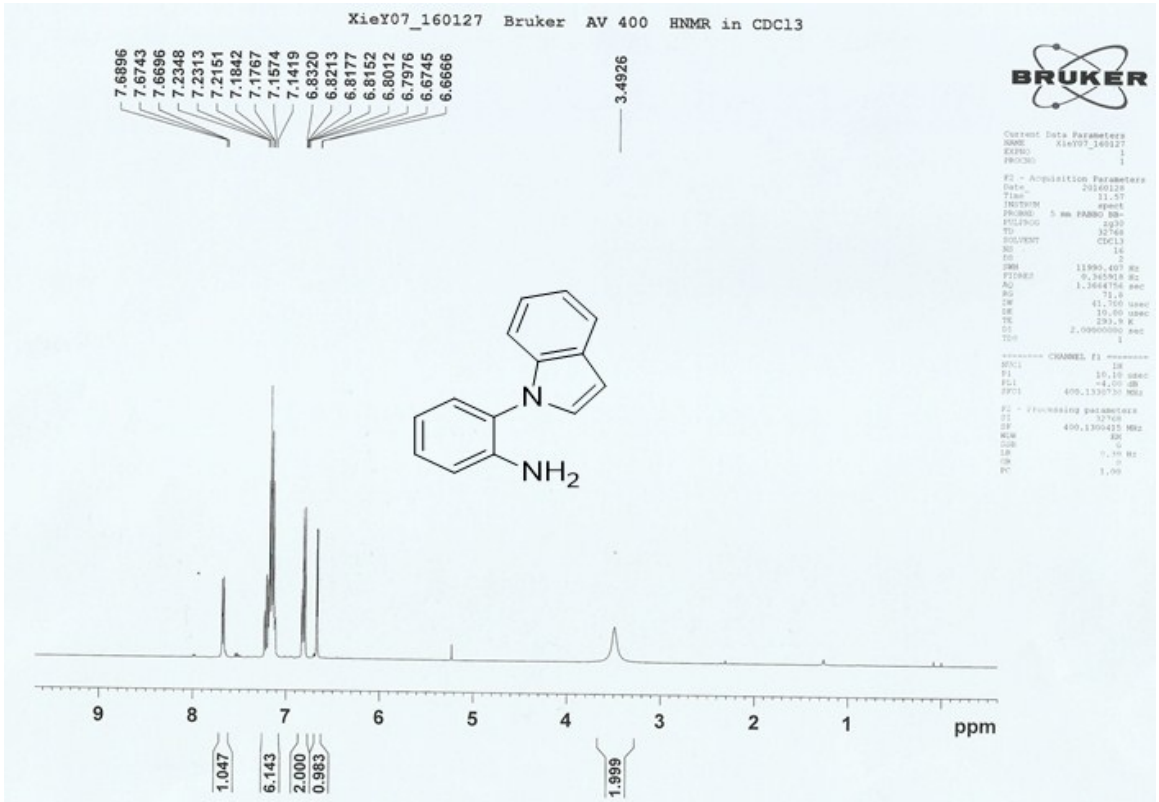
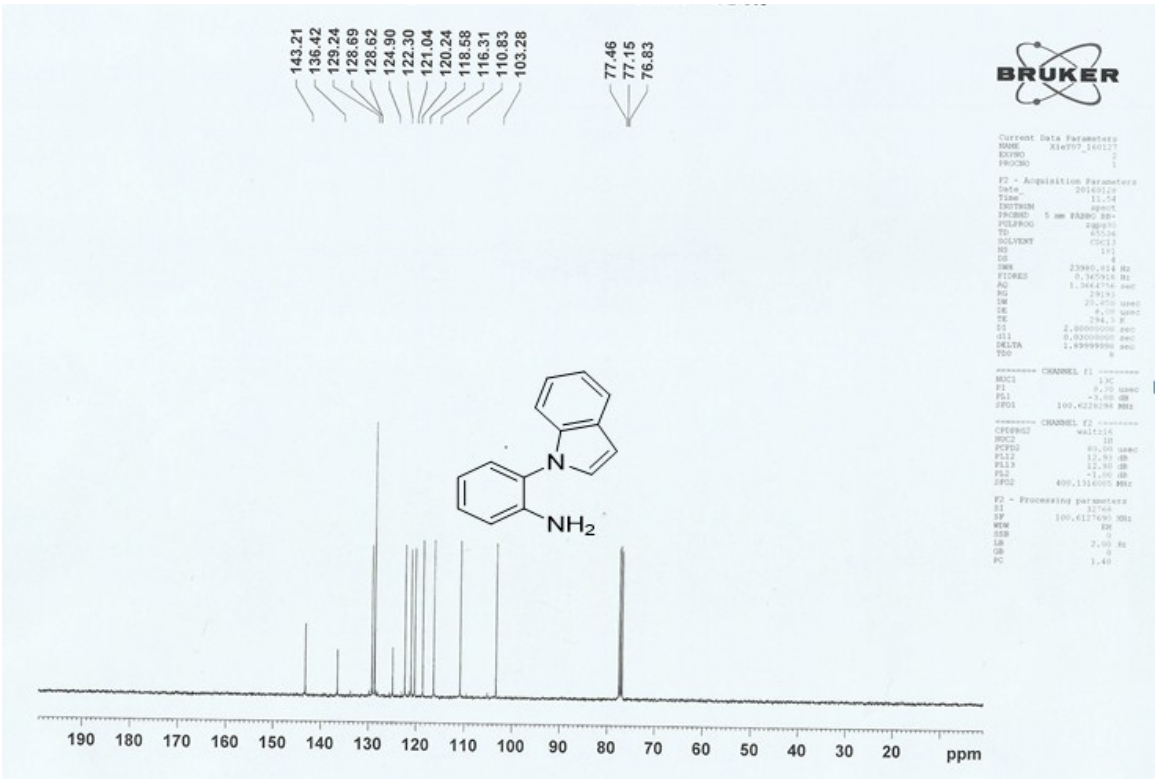


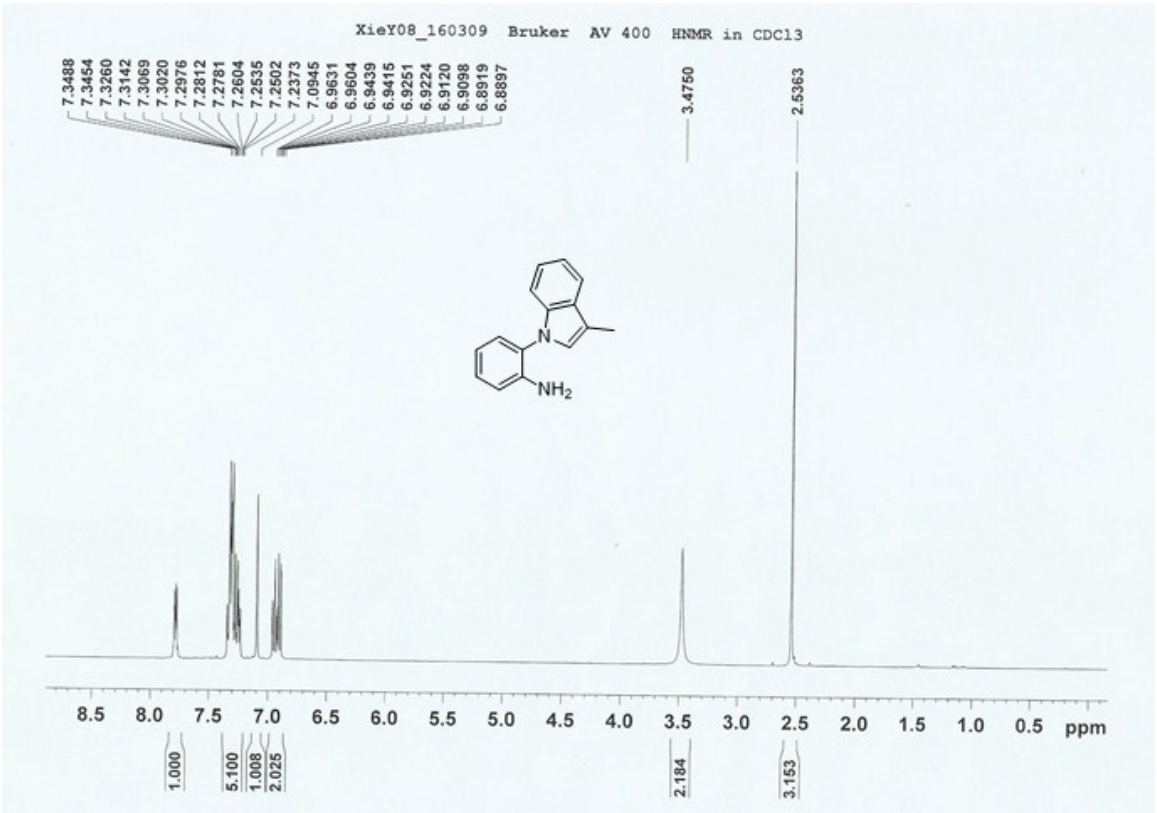


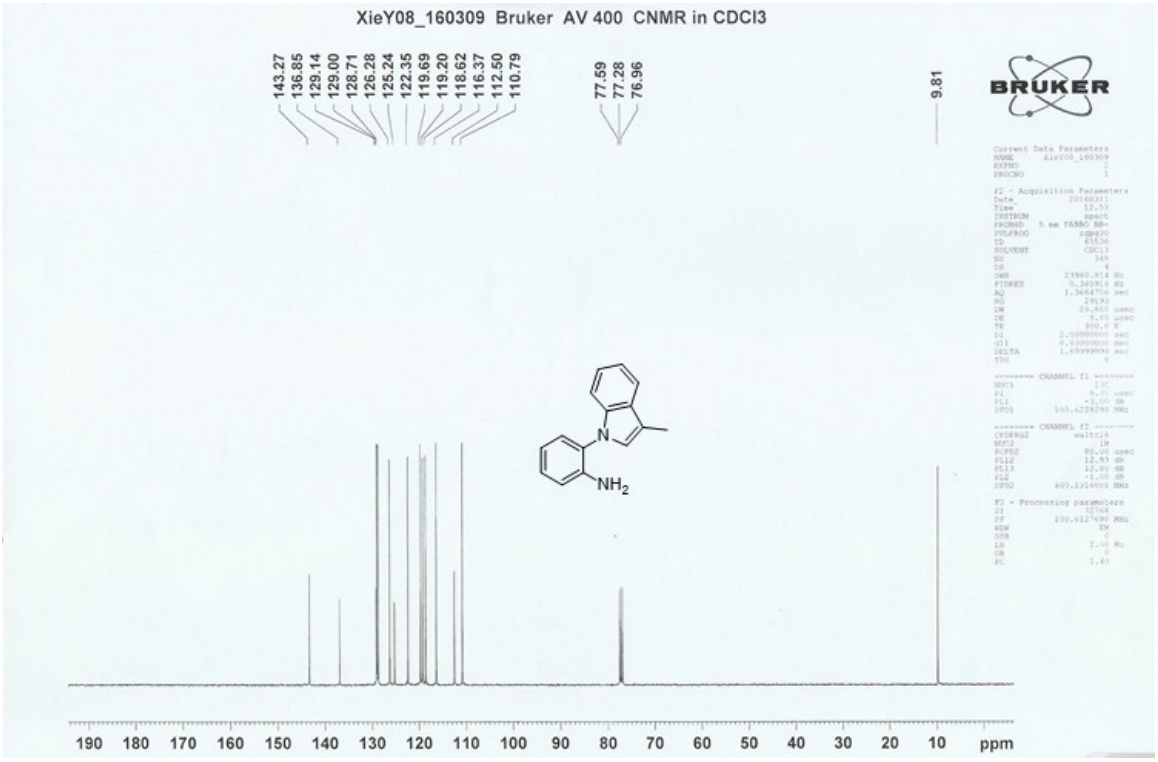
2-(1*H*-indol-1-yl)aniline (1i)



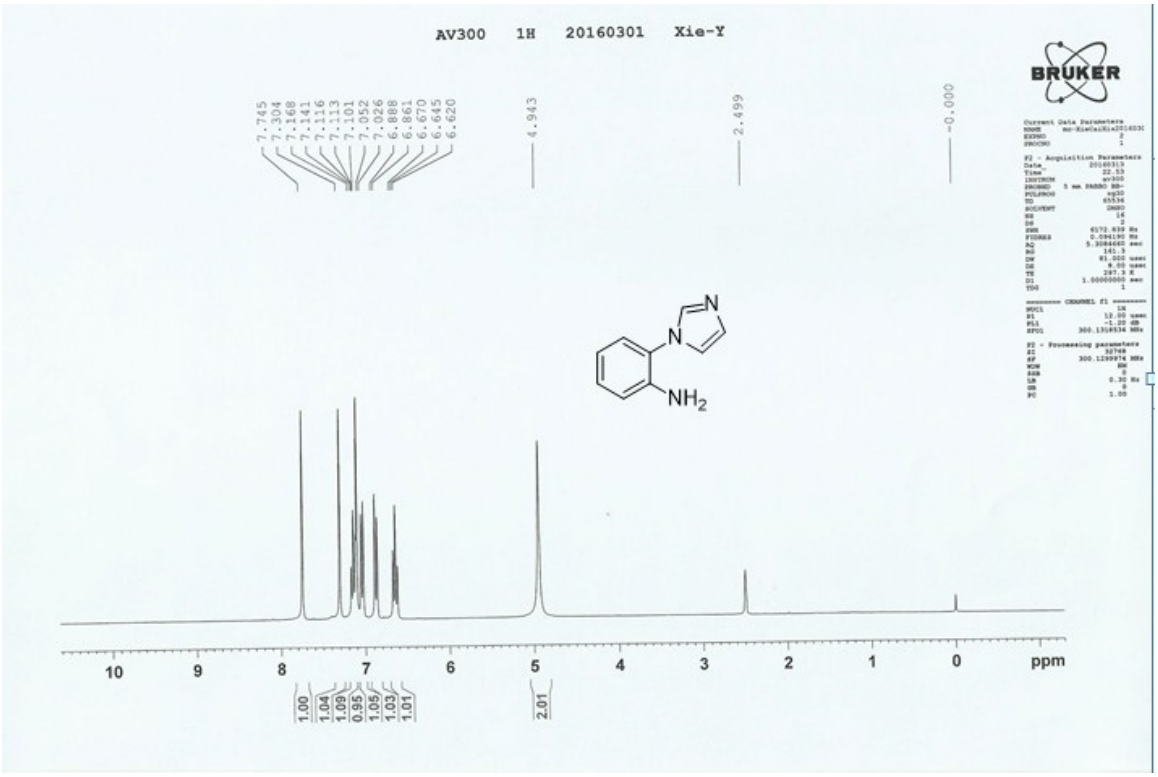


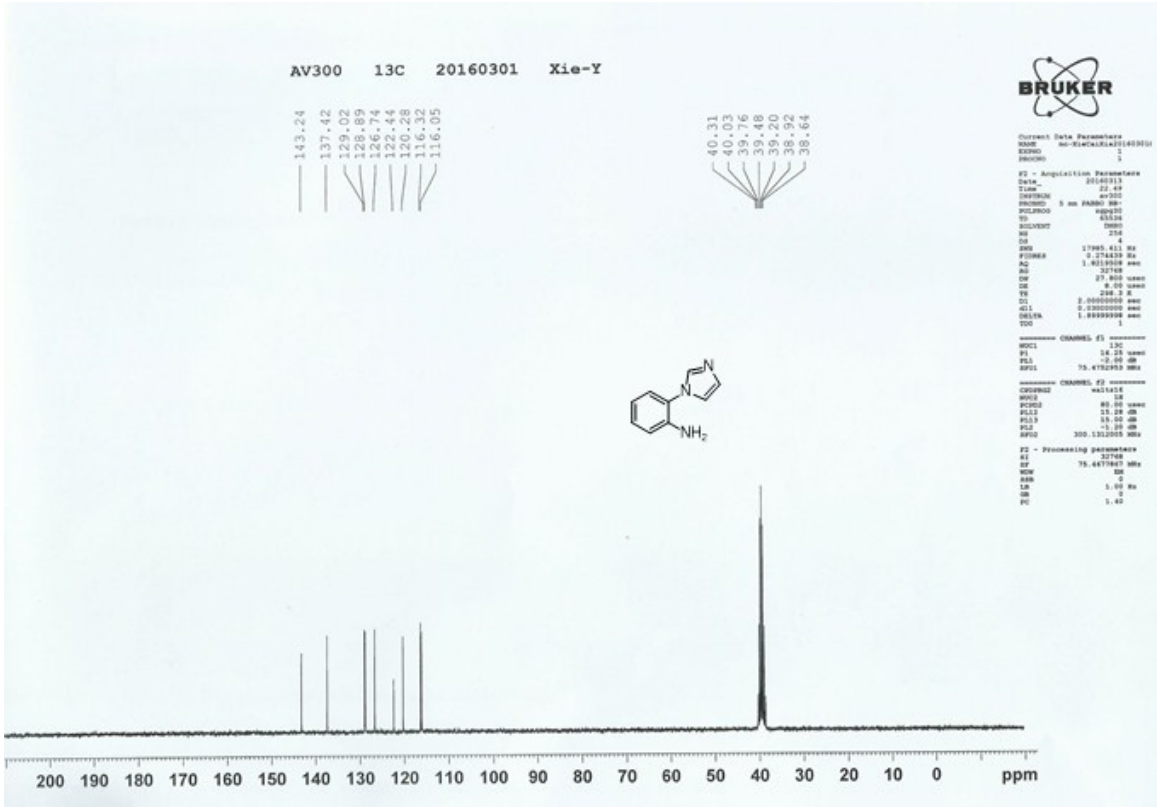
2-(3-methyl-1*H*-indol-1-yl)aniline (1j)





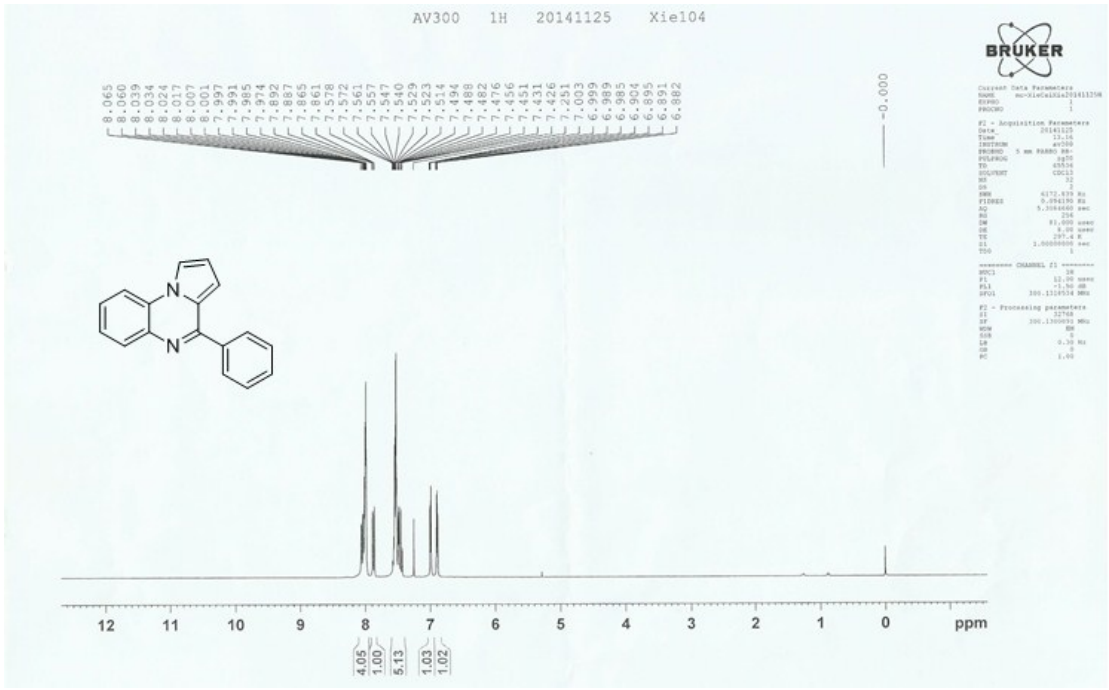
2-(1*H*-imidazol-1-yl)aniline (1k)

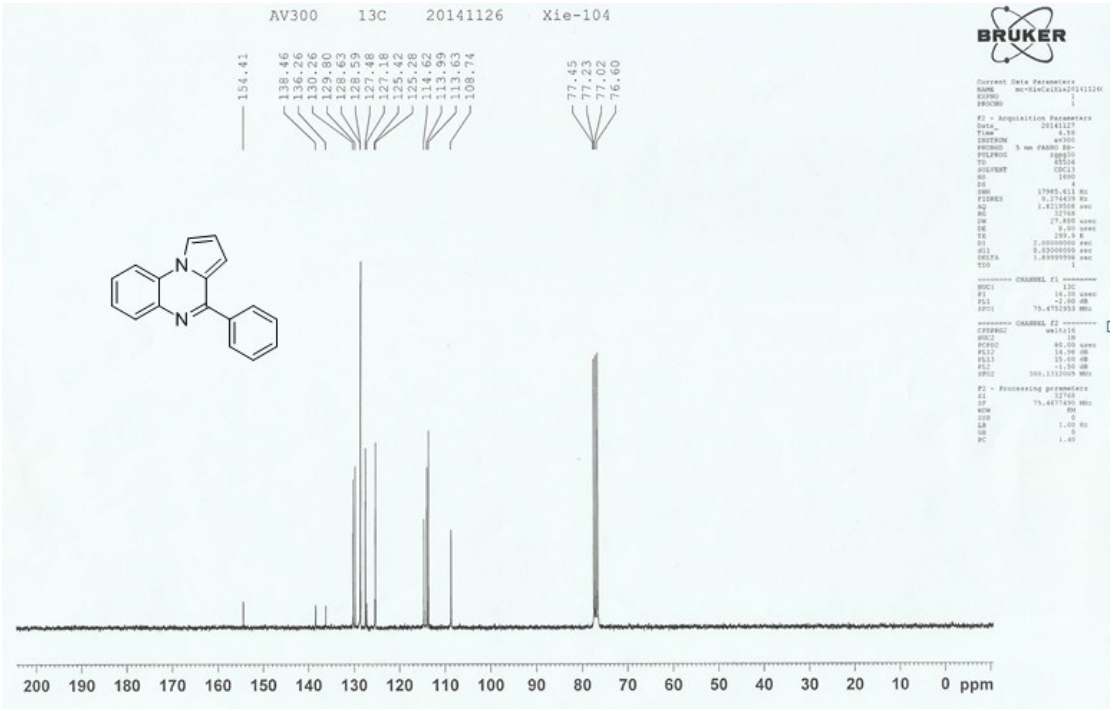




3.2 The sprctra of products

4-phenylpyrrolo[1,2-*a*]quinoxaline (4a)

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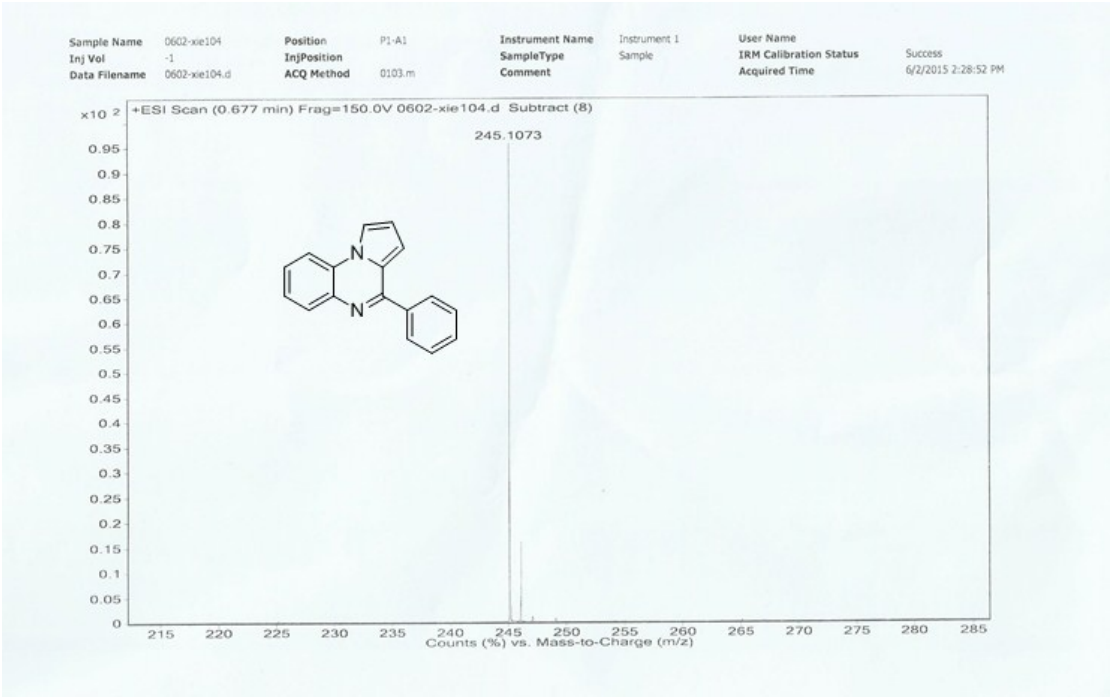
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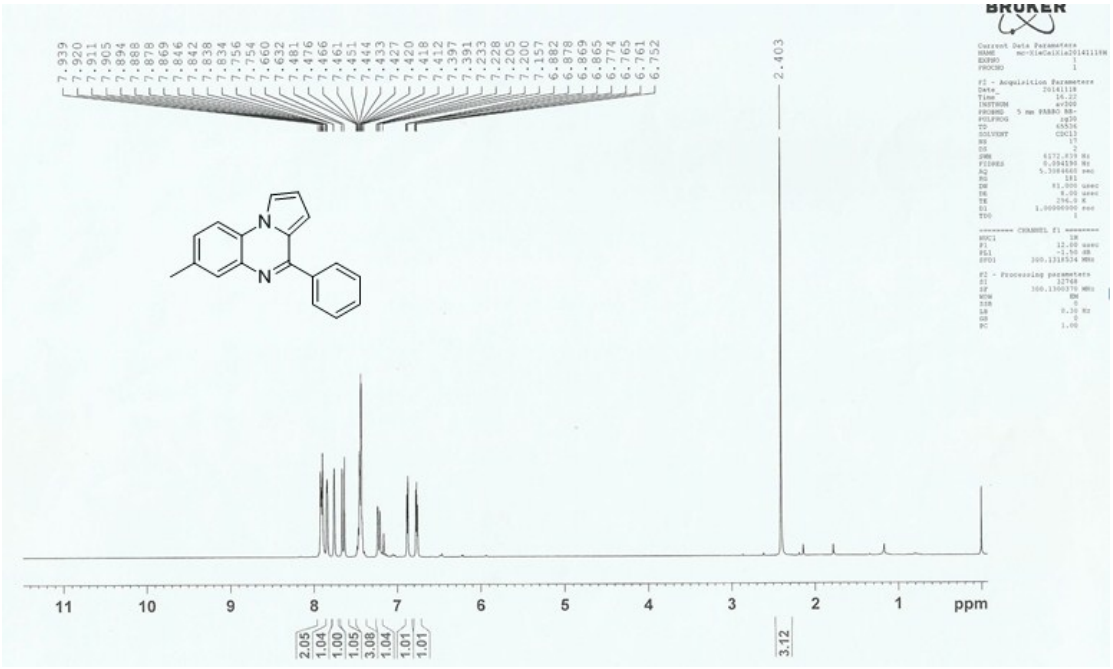
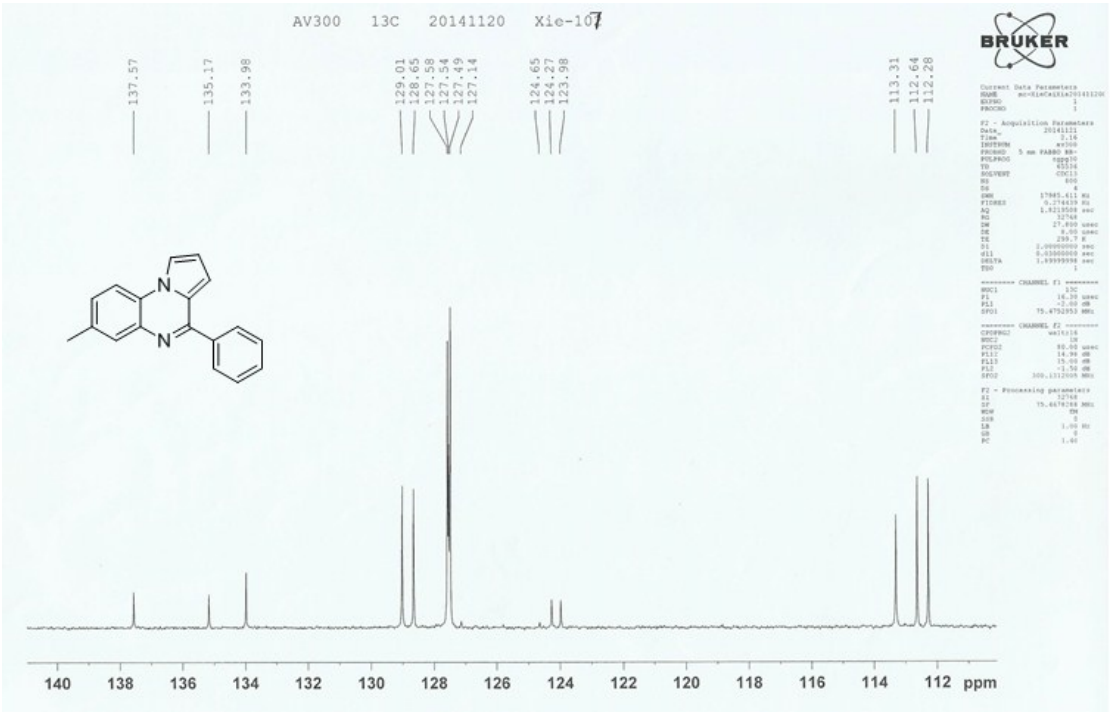
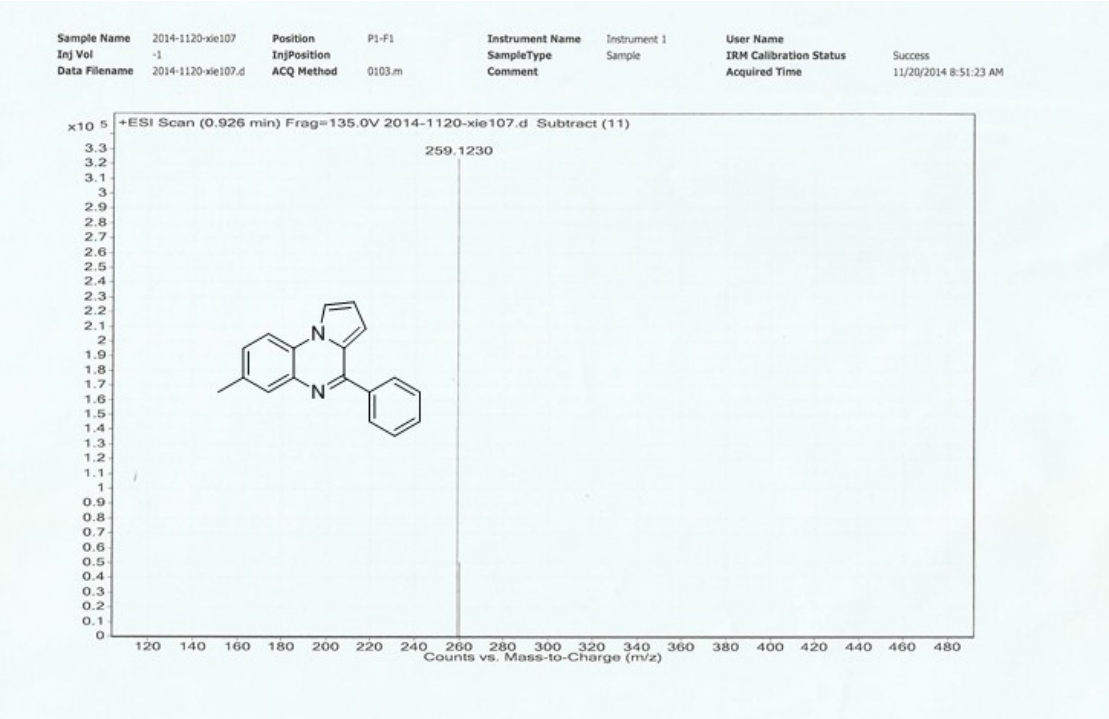
Counts (%) vs. Mass-to-Charge (m/z)

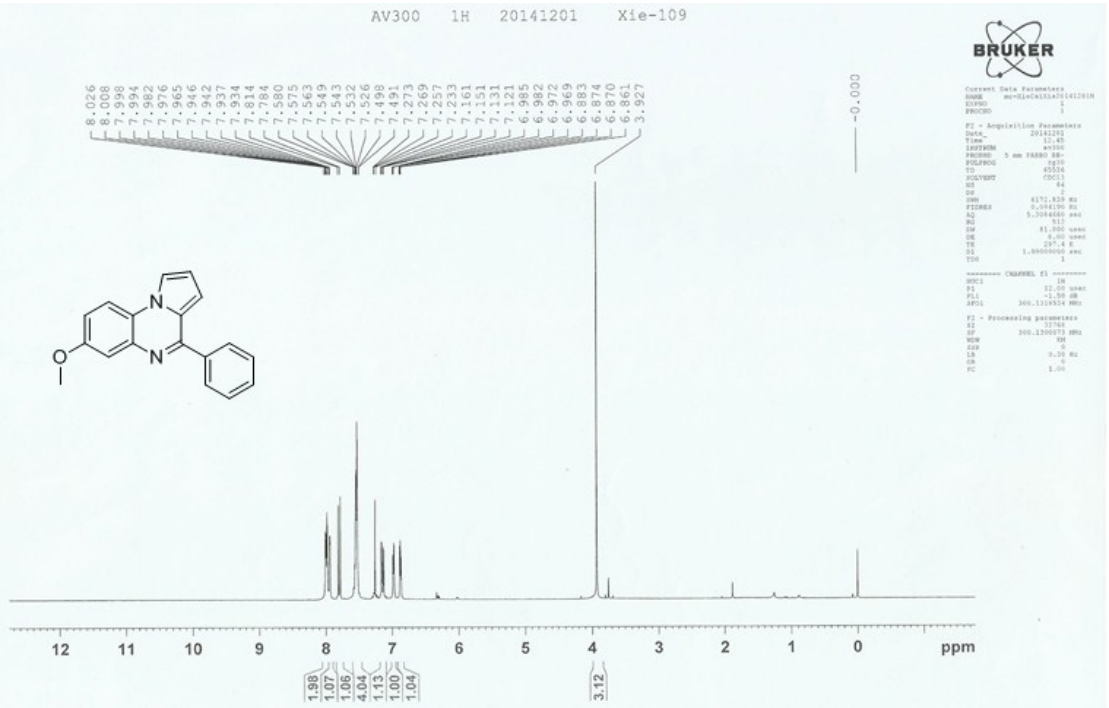
7-methyl-4-phenylpyrrolo[1,2-*a*]quinoxaline (4b)

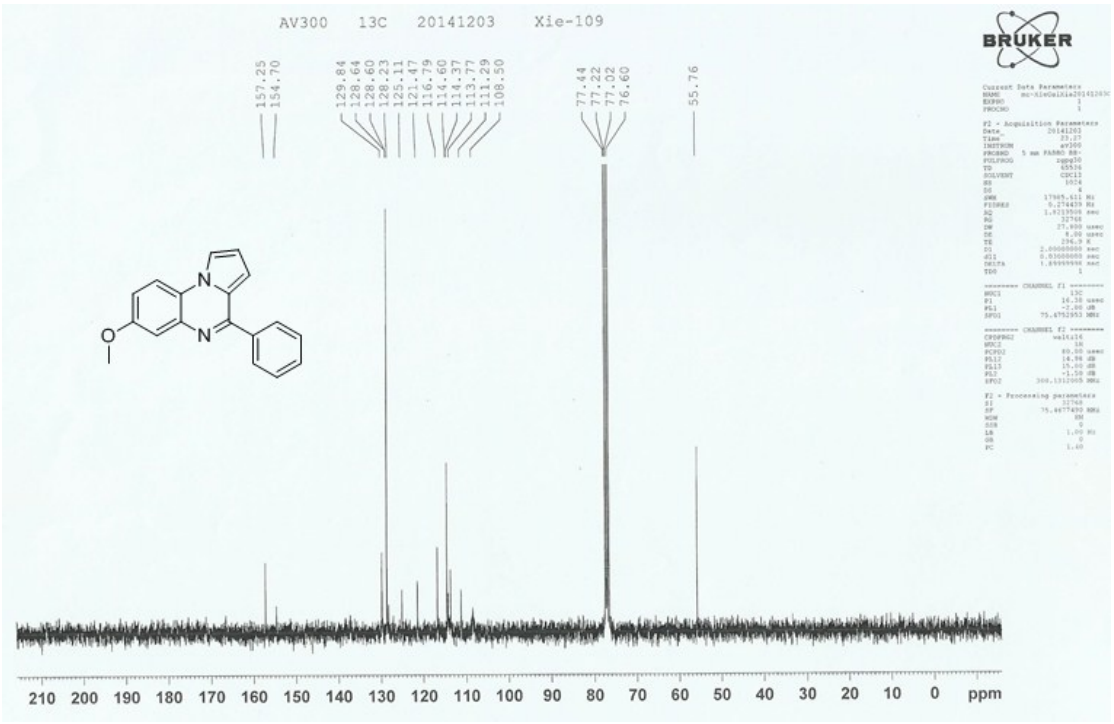






7-methoxy-4-phenylpyrrolo[1,2-*a*]quinoxaline (4c)

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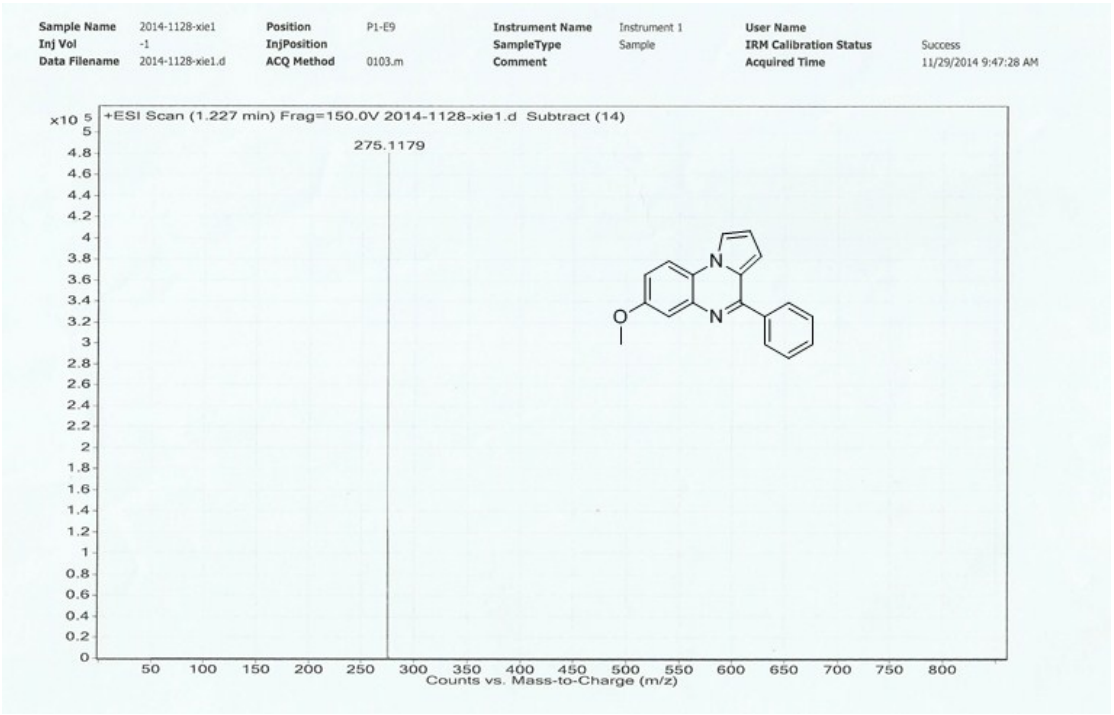
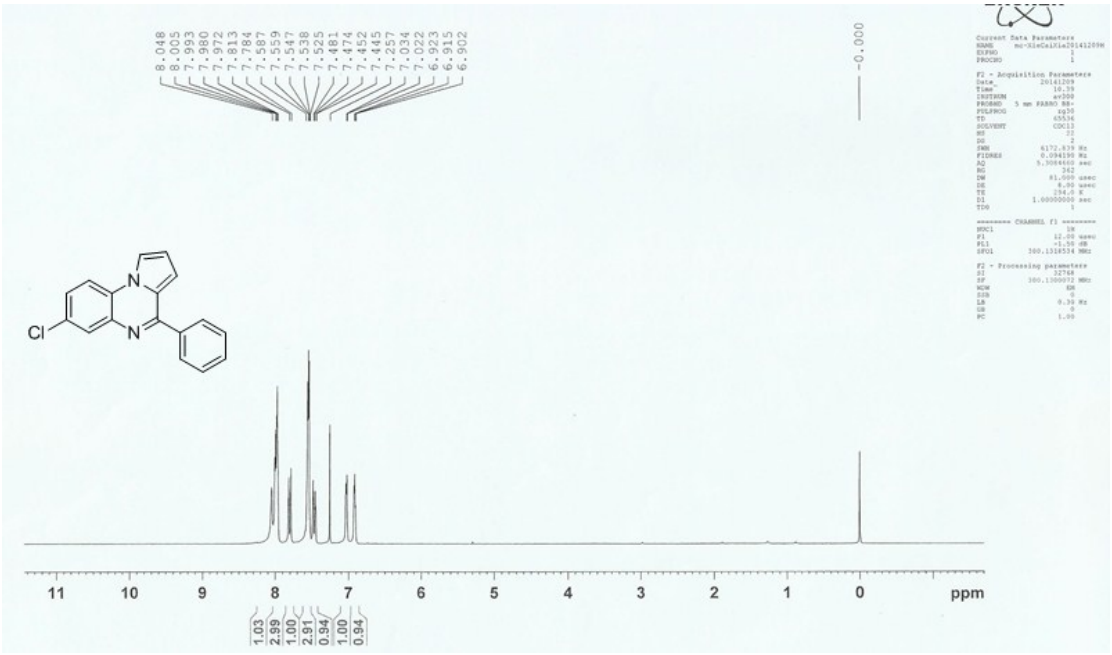
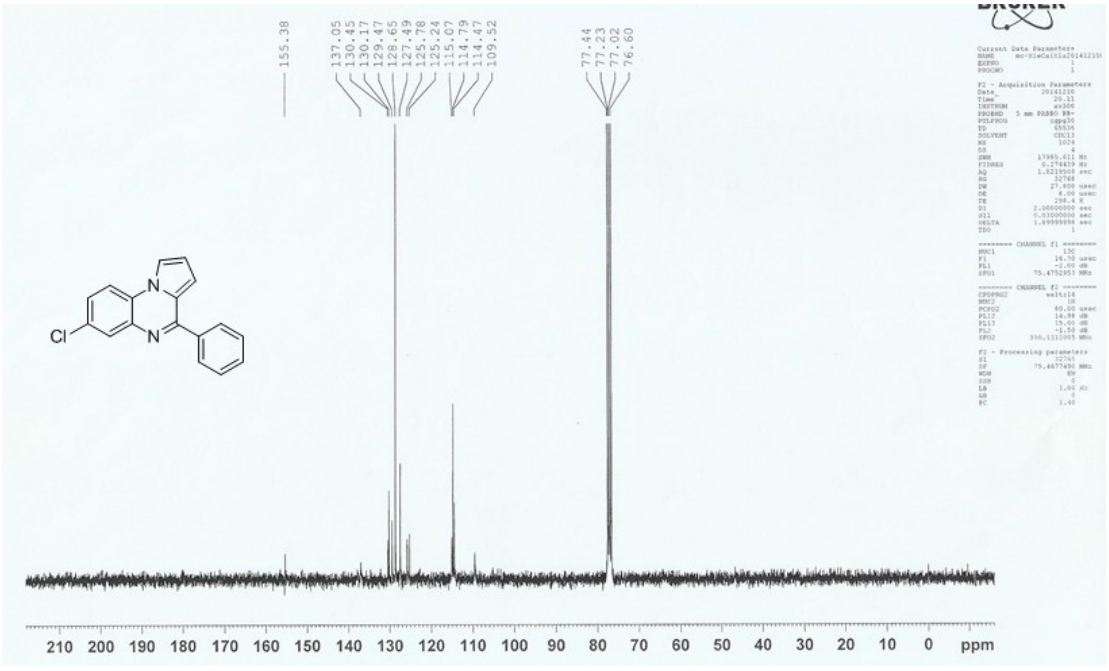
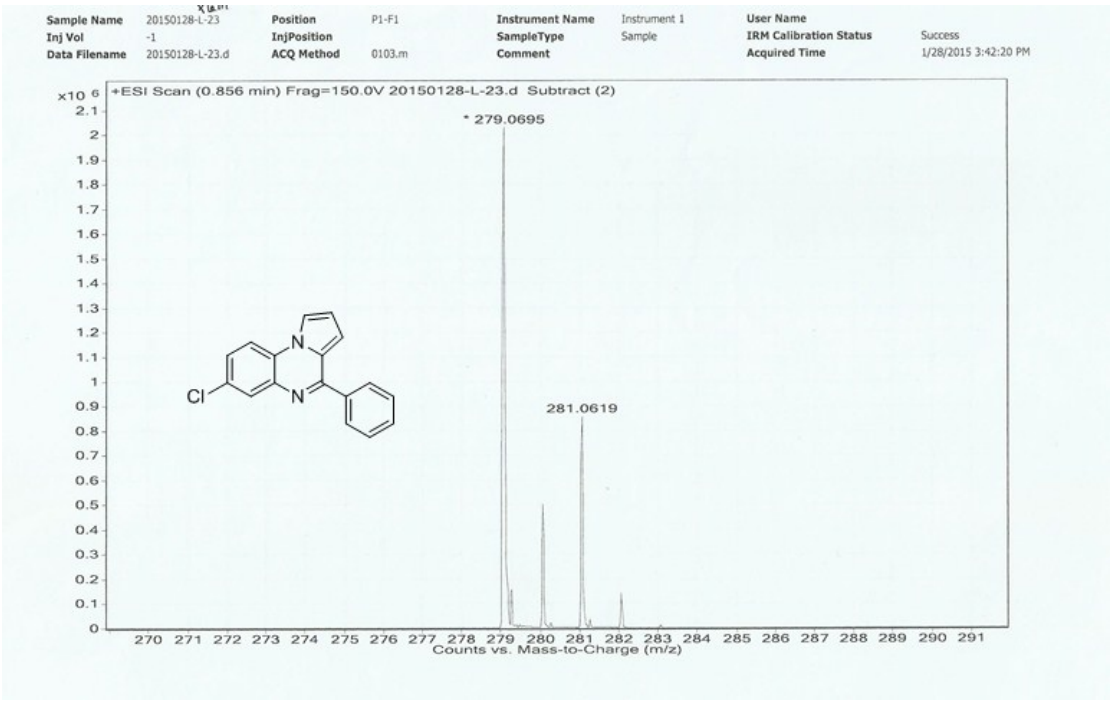
121.47
116.79
114.60
114.37
113.77
111.29
108.50
77.44
77.22
77.02
76.60
55.76
O
N
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Sample Name 2014-1128-xie1
Position P1-E9
Instrument Name Instrument 1
User Name
Inj Vol -1
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename 2014-1128-xie1.d
ACQ Method 0103.m
Comment
Acquired Time 11/29/2014 9:47:28 AM
x10 5
+ESI Scan (1.227 min) Frag=150.0V 2014-1128-xie1.d Subtract (14)
275.1179
O
N
N
Counts vs. Mass-to-Charge (m/z)

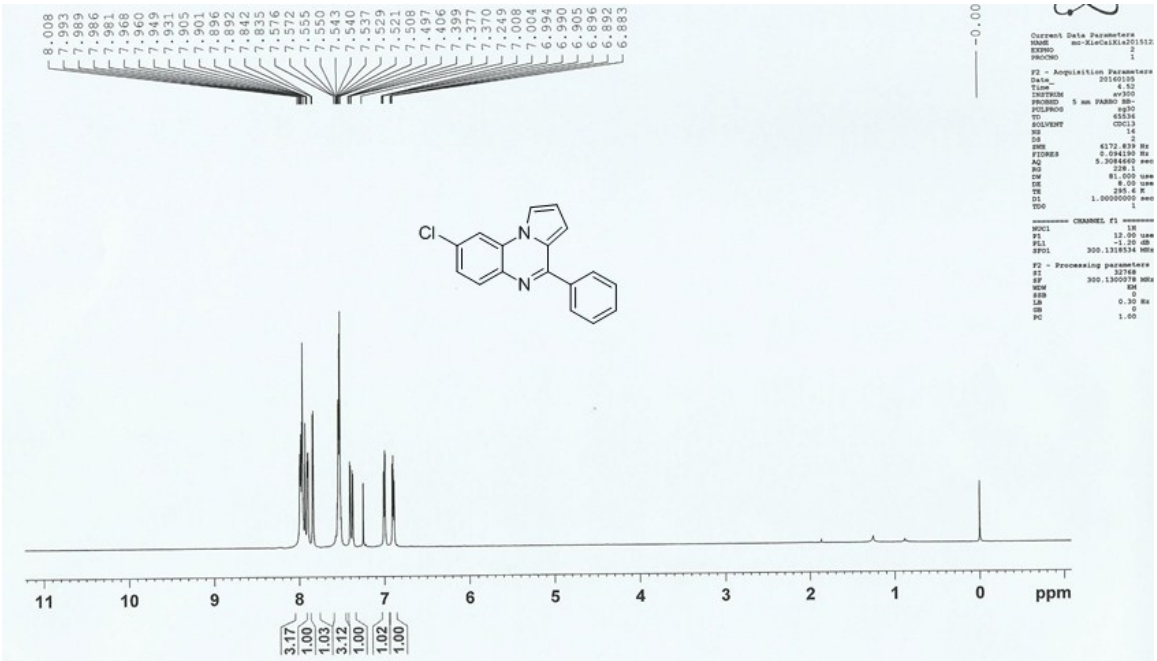
7-chloro-4-phenylpyrrolo[1,2-*a*]quinoxaline (4d)

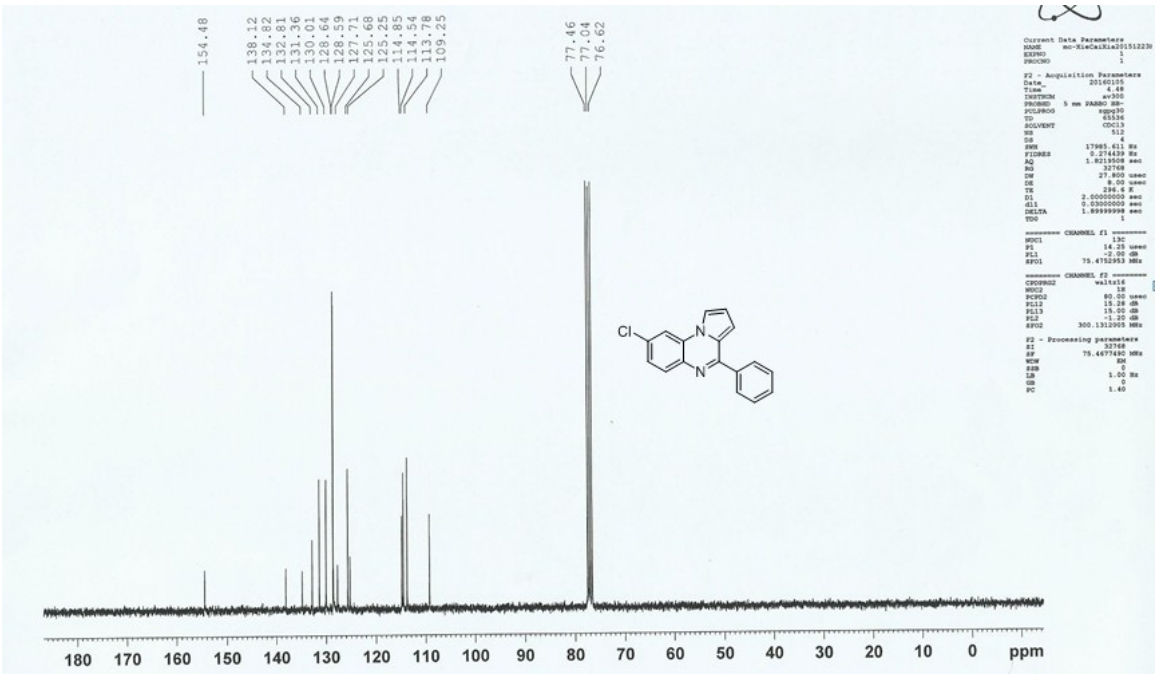


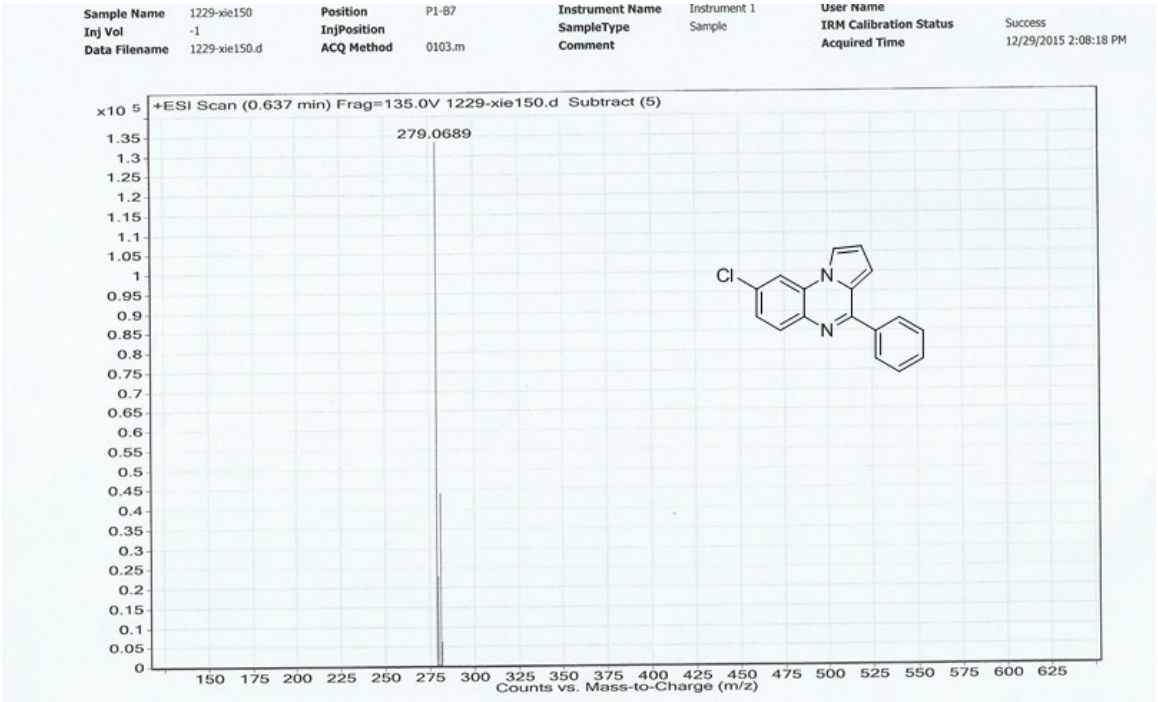




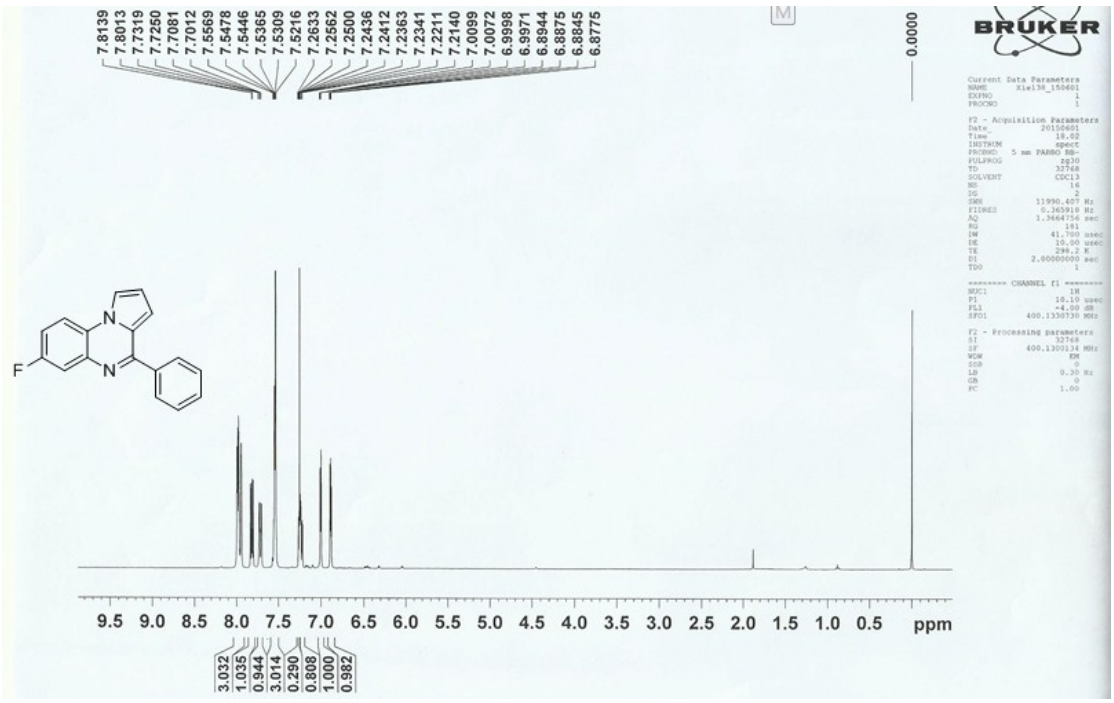
8-chloro-4-phenylpyrrolo[1,2-a]quinoxaline (4e)

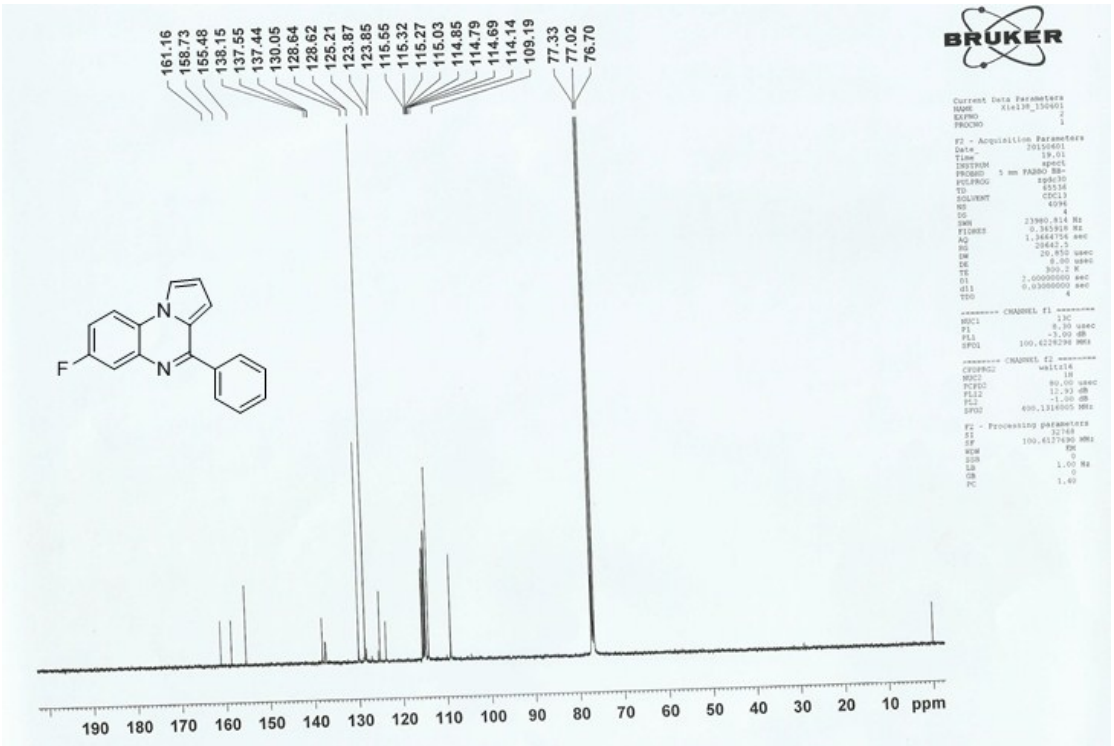


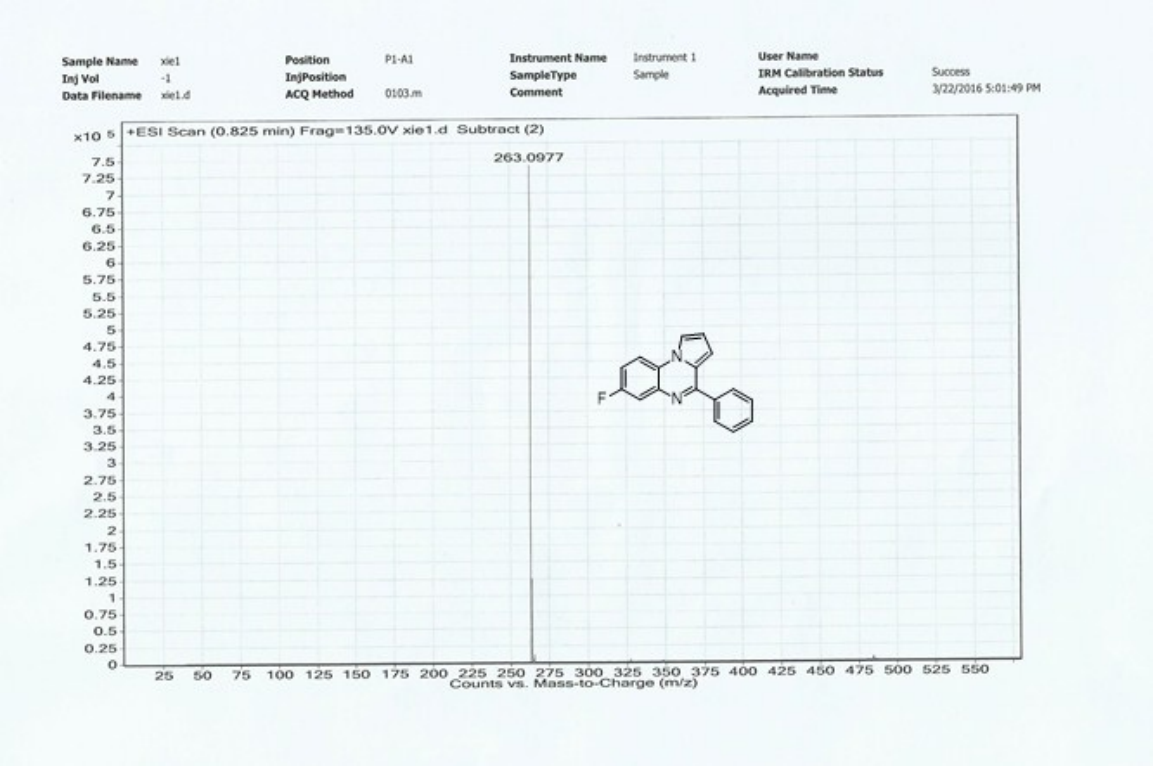

154.48
138.12
134.82
132.81
131.36
130.01
128.64
128.59
127.71
126.68
125.85
114.85
114.54
113.78
109.25
77.46
77.04
76.62
Cl
N
N
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Sample Name 1229-xie150
Position P1-B7
Instrument Name Instrument 1
User Name
Inj Vol -1
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename 1229-xie150.d
ACQ Method 0103.m
Comment
Acquired Time 12/29/2015 2:08:18 PM
+ESI Scan (0.637 min) Frag=135.0V 1229-xie150.d Subtract (5)
x10 5
279.0689
Cl
N
N
Counts vs. Mass-to-Charge (m/z)

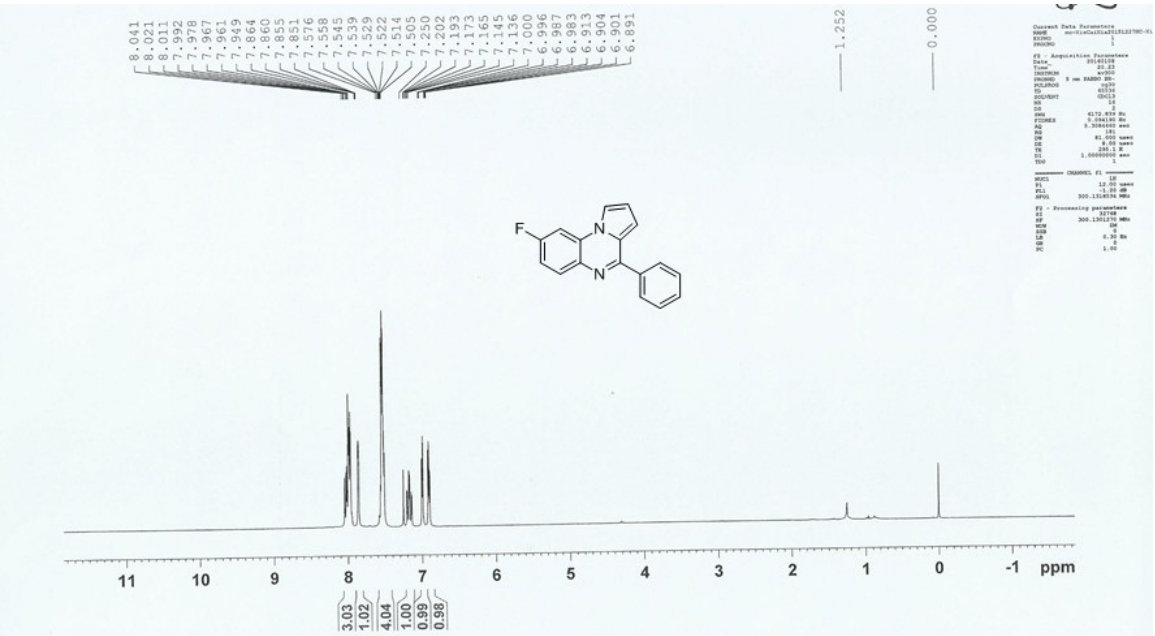
7-fluoro-4-phenylpyrrolo[1,2-*a*]quinoxaline (4f)

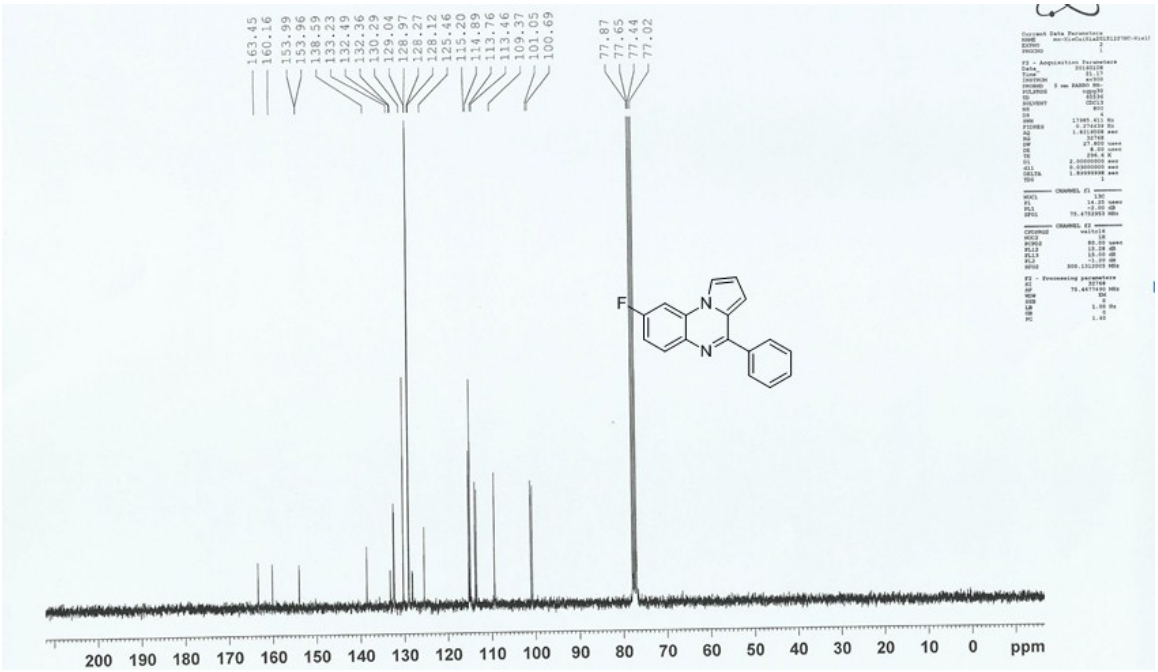


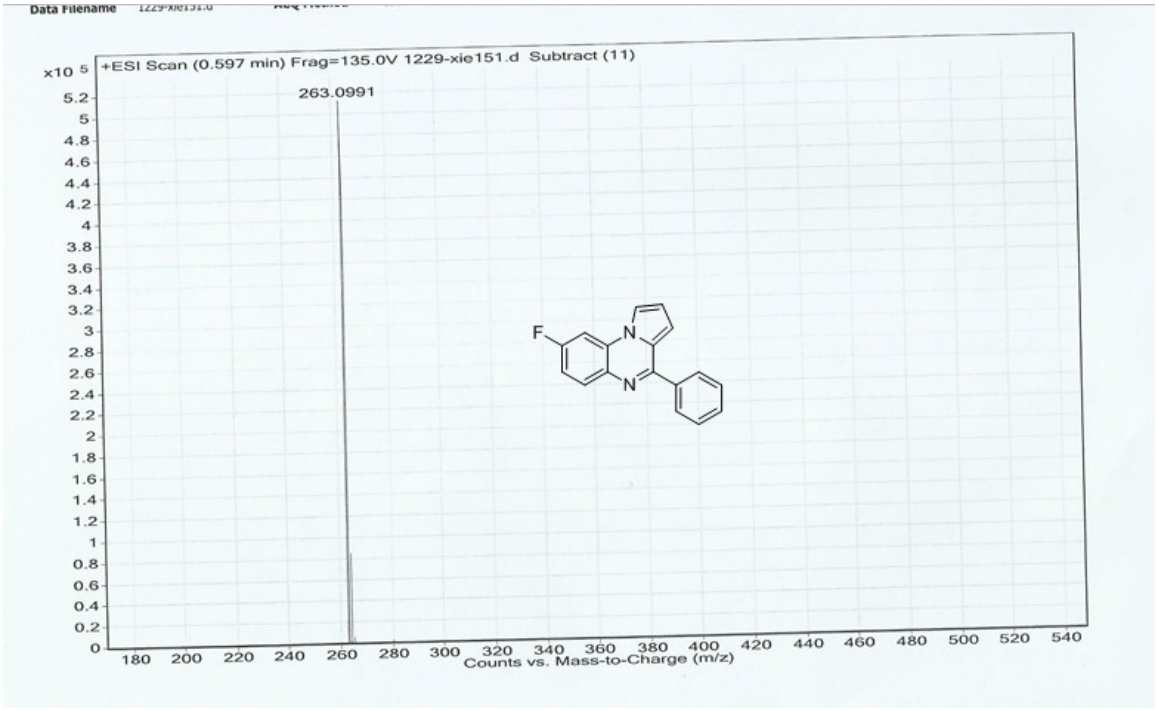




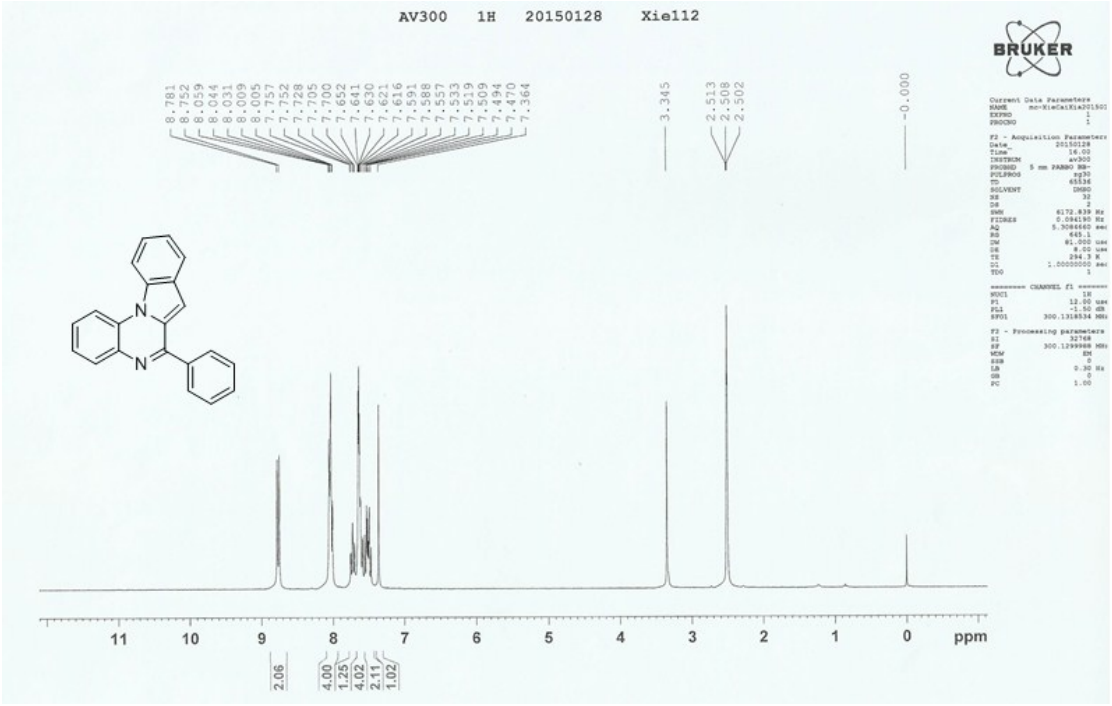
8-fluoro-4-phenylpyrrolo[1,2-a]quinoxaline (4g)

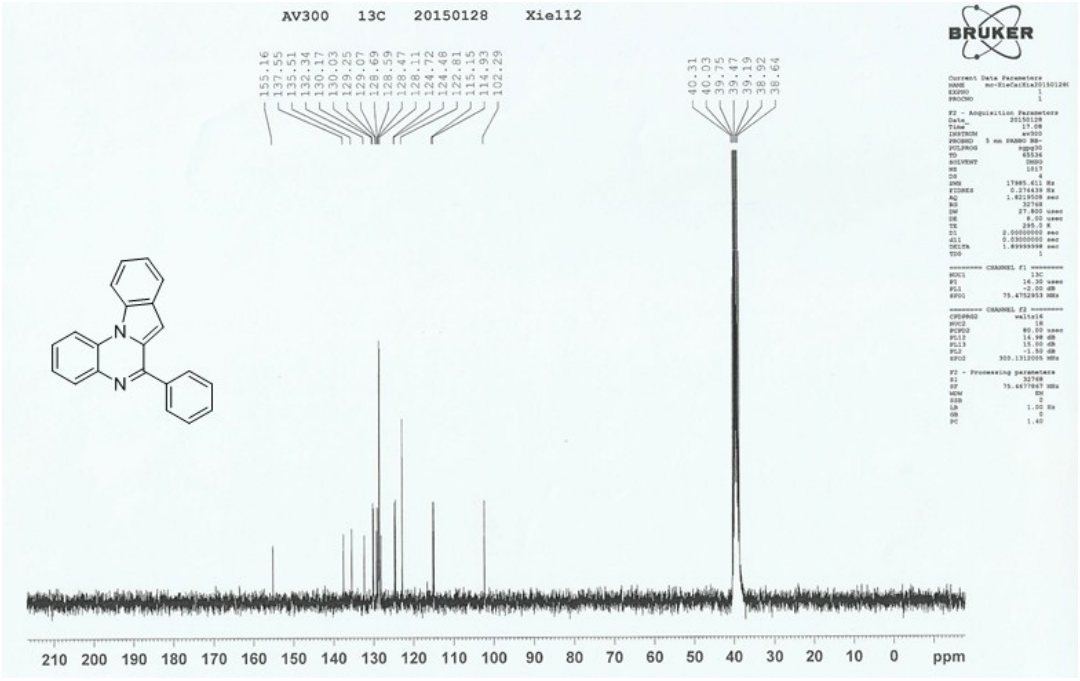


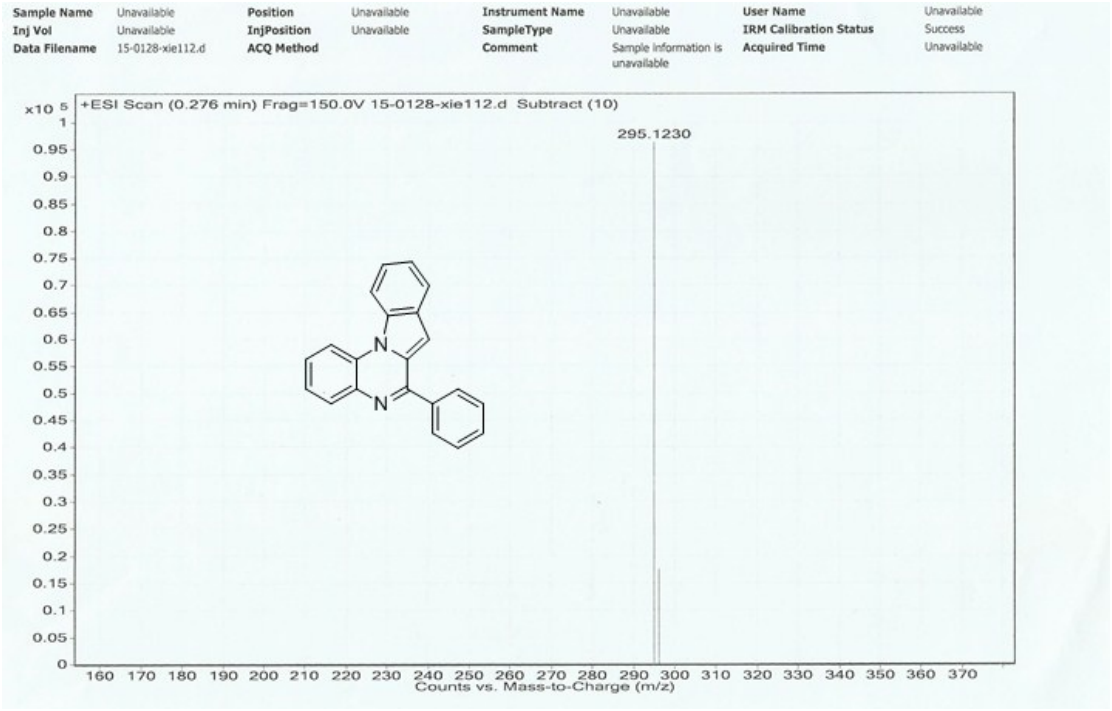

163.45
160.16
153.99
153.96
138.59
133.23
132.49
132.36
130.29
129.04
128.97
128.27
128.12
125.46
115.20
114.89
113.76
113.46
109.37
101.05
100.69
77.87
77.65
77.44
77.02
F
N
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Data Filename
+ESI Scan (0.597 min) Frag=135.0V 1229-xie151.d Subtract (11)
x10 5
263.0991
F
N
N
Counts vs. Mass-to-Charge (m/z)

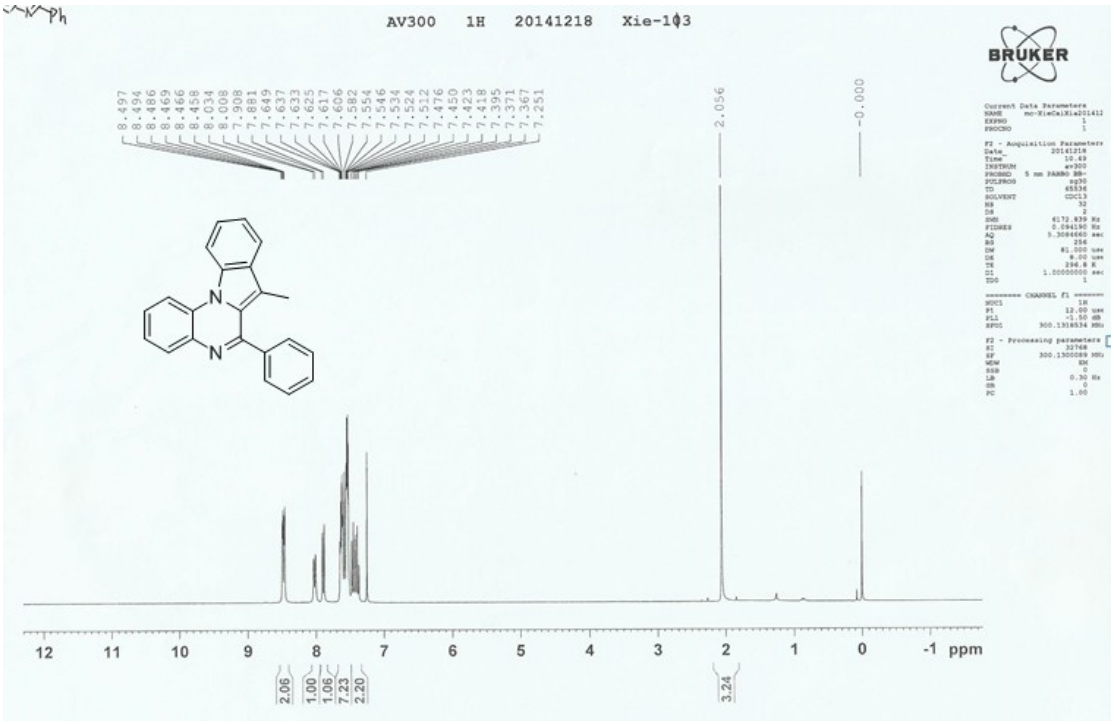
6-phenylindolo[1,2-*a*]quinoxaline (4i)

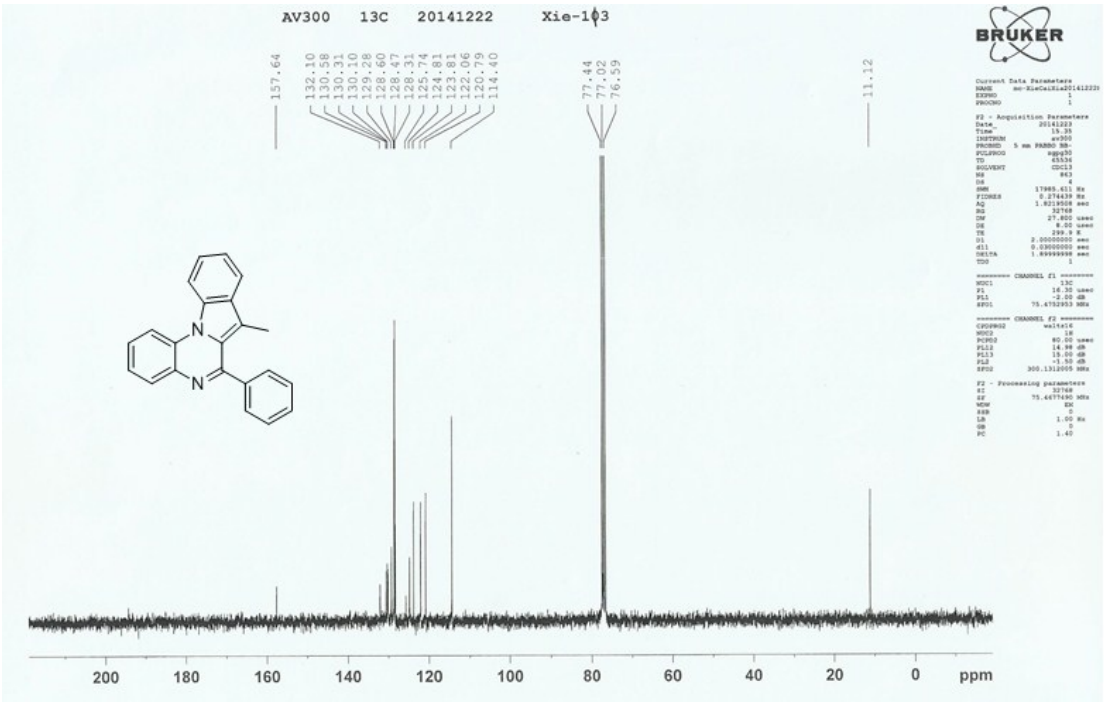


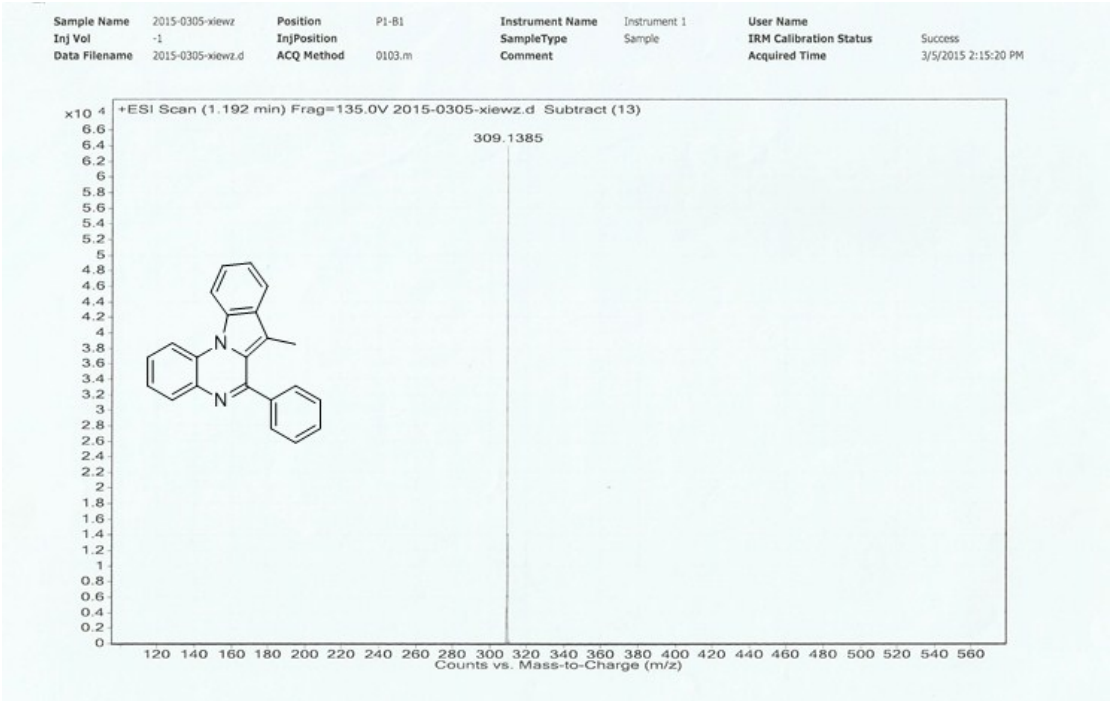




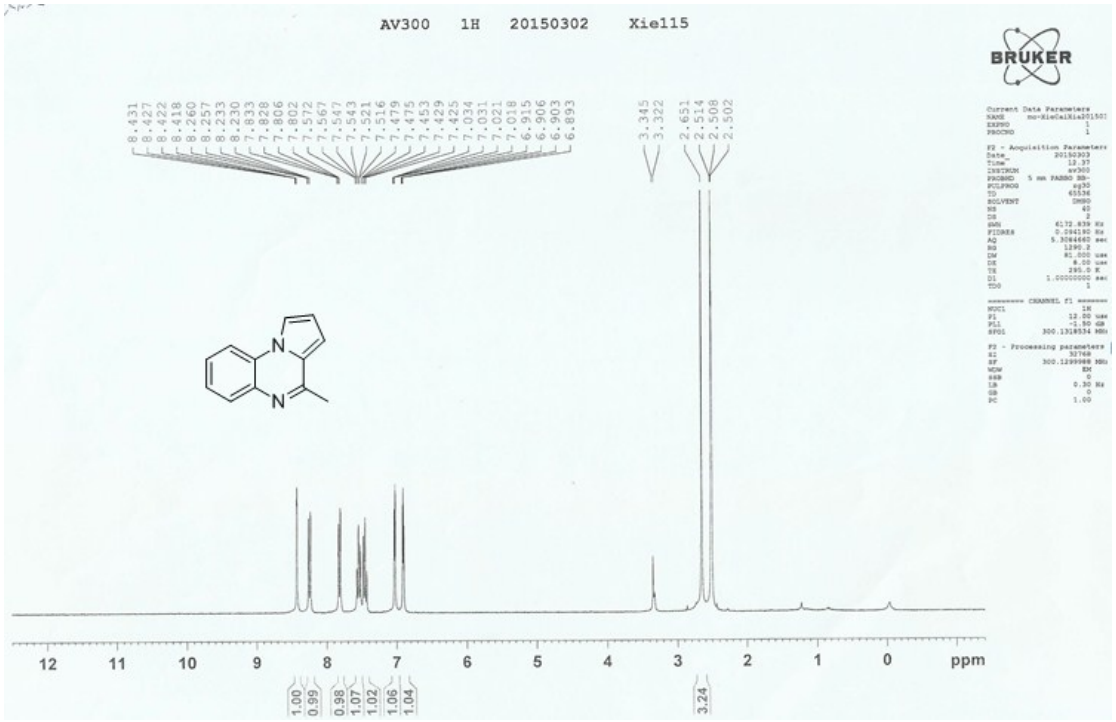
7-methyl-6-phenylindolo[1,2-*a*]quinoxaline (4j)

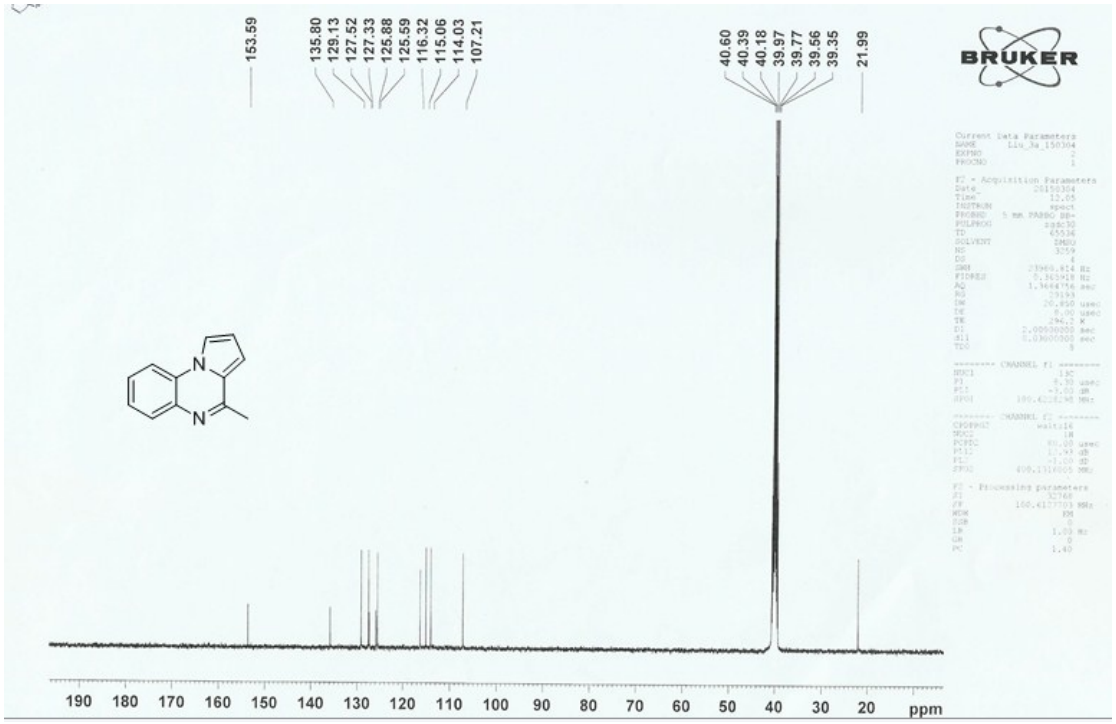


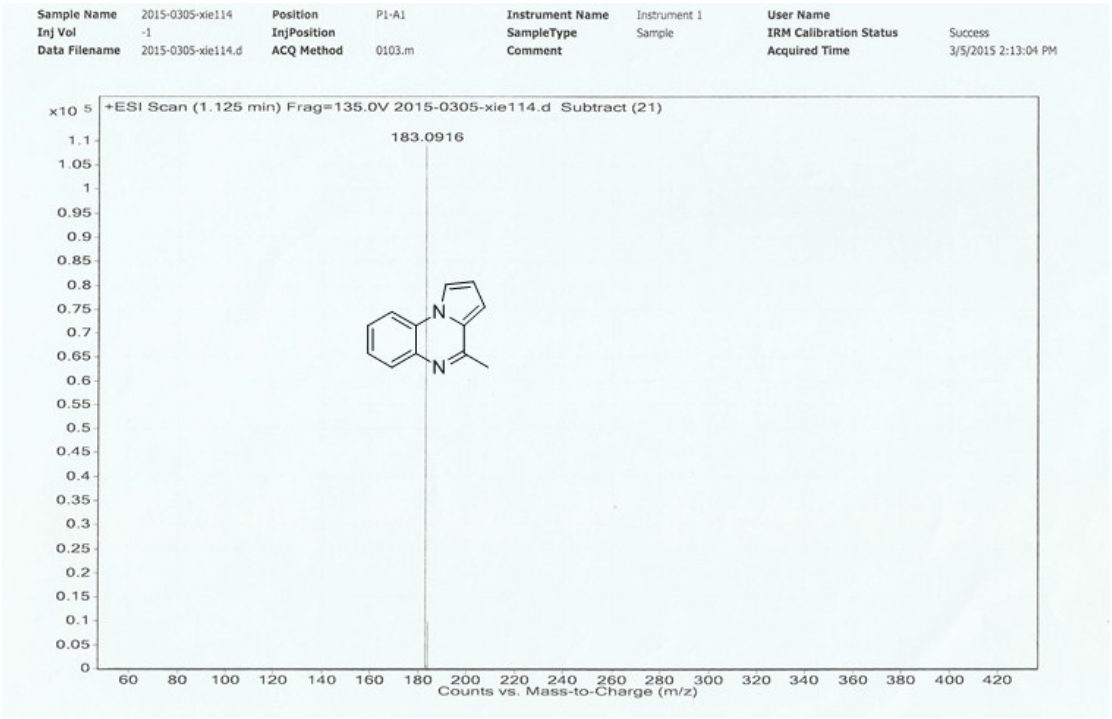

AV300 13C 20141222 Xie-103
157.64
132.10
130.58
130.31
130.10
129.28
128.60
128.47
128.31
125.74
124.81
123.81
122.06
120.79
114.40
77.44
77.02
76.59
11.12
BRUKER
200 180 160 140 120 100 80 60 40 20 0 ppm


Sample Name 2015-0305-xiewz
Position P1-B1
Instrument Name Instrument 1
User Name
Inj Vol -1
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename 2015-0305-xiewz.d
ACQ Method 0103.m
Comment
Acquired Time 3/5/2015 2:15:20 PM
+ESI Scan (1.192 min) Frag=135.0V 2015-0305-xiewz.d Subtract (13)
309.1385
x10 4
Counts vs. Mass-to-Charge (m/z)

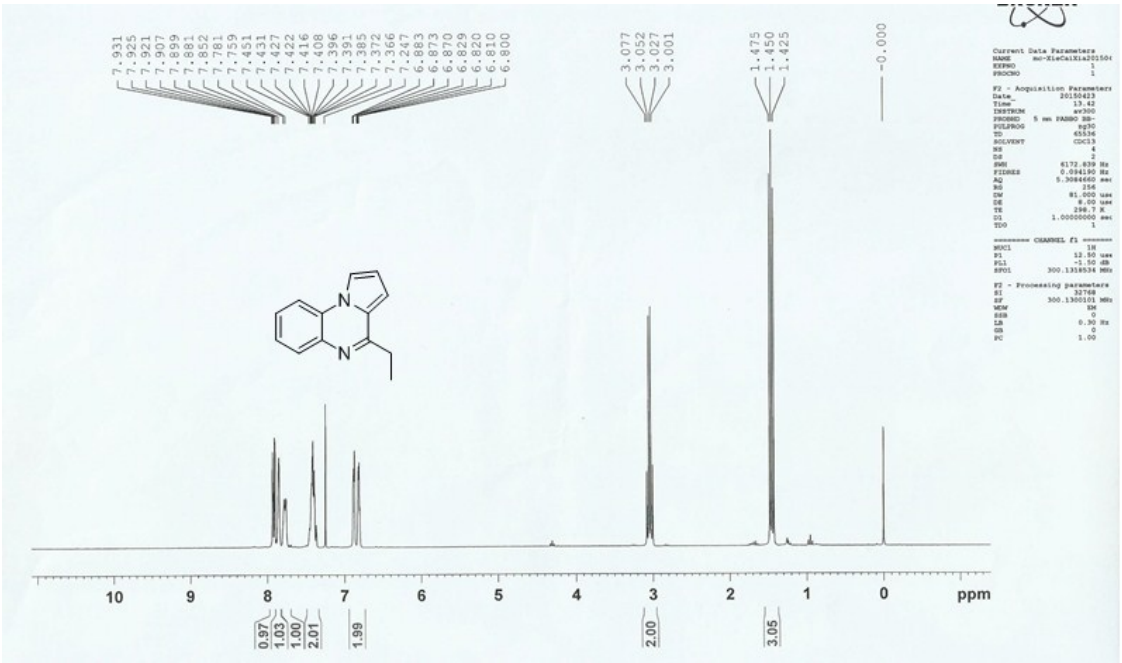
4-methylpyrrolo[1,2-*a*]quinoxaline (4k)

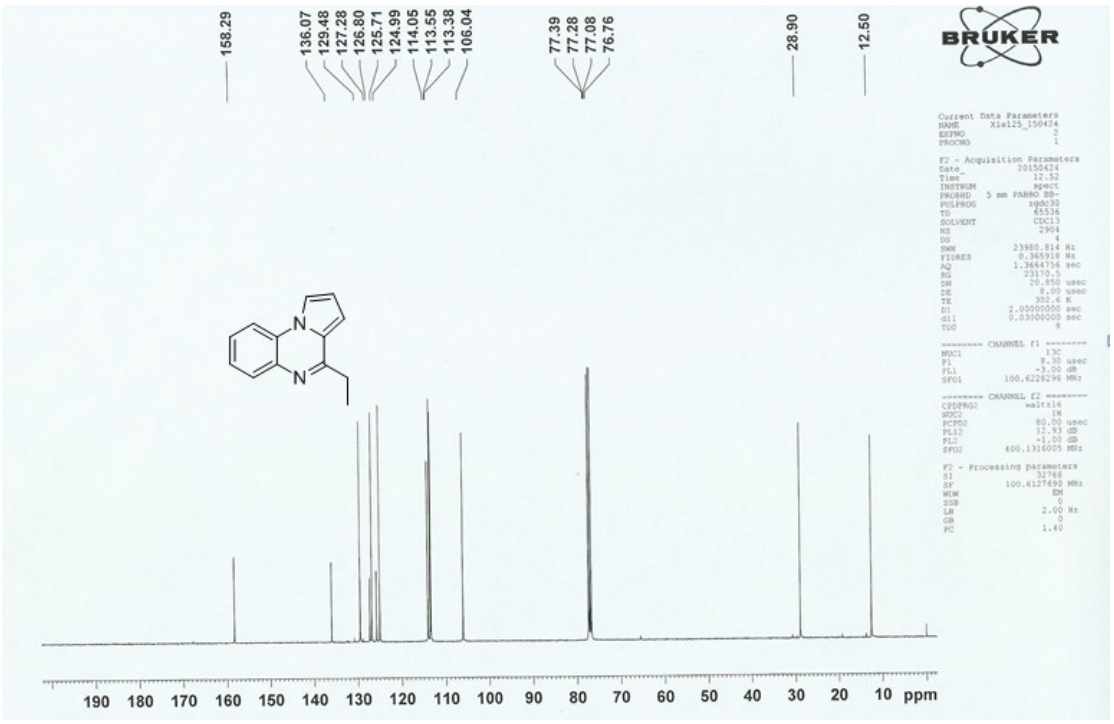


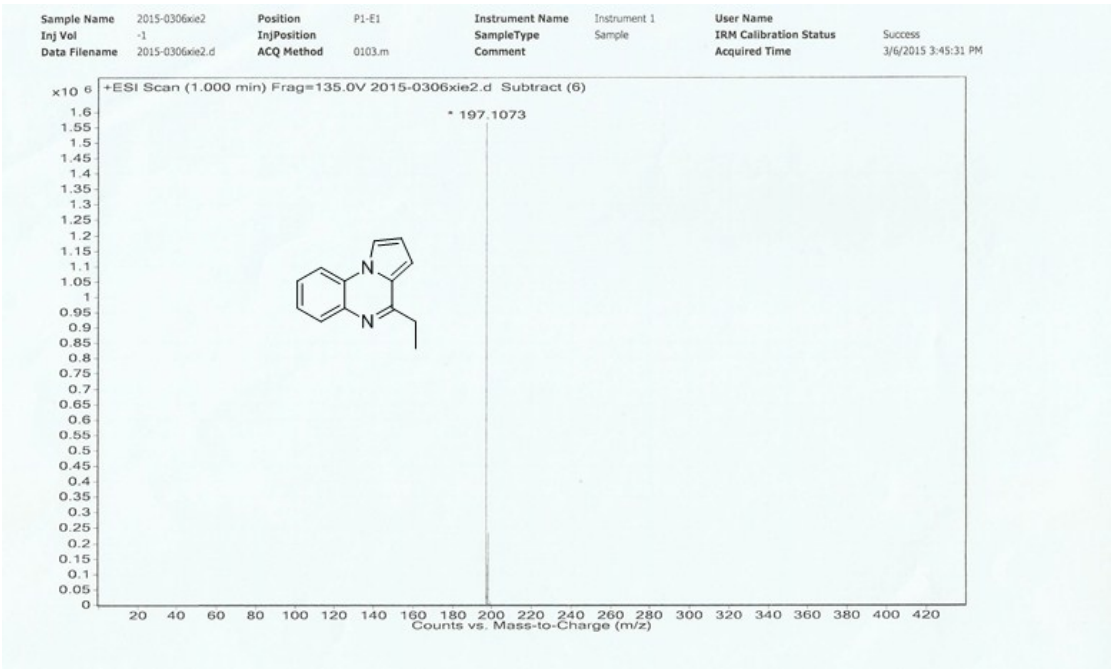




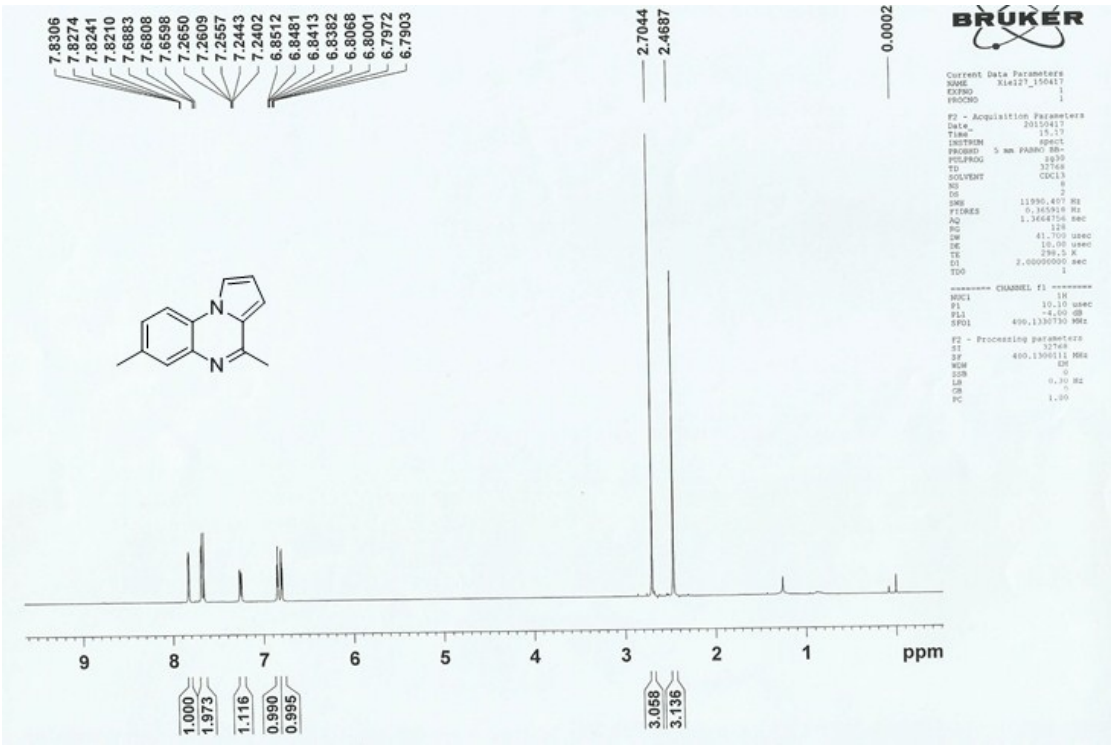
4-ethylpyrrolo[1,2-*a*]quinoxaline (4l)

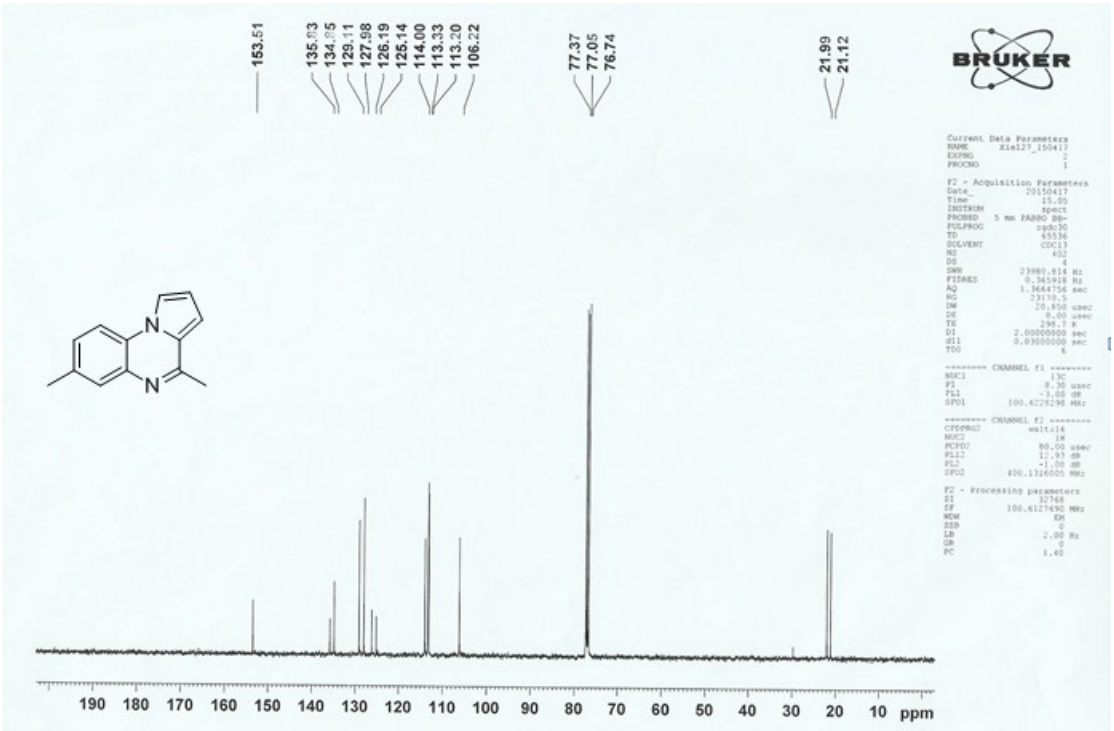


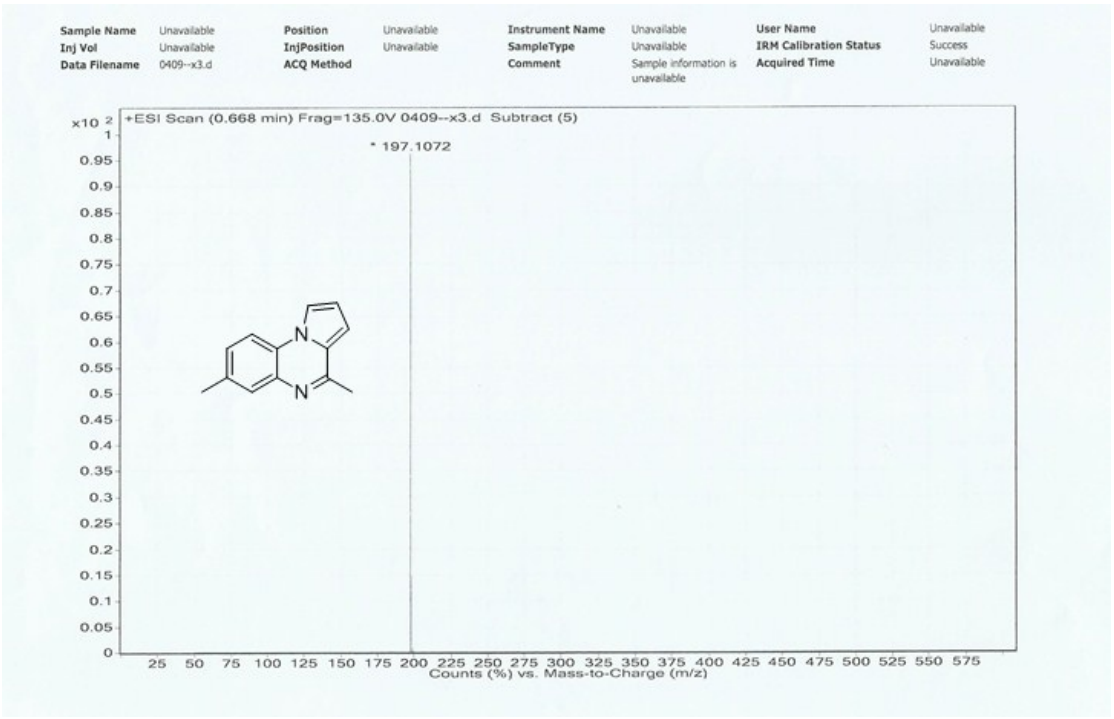

158.29
136.07
129.48
127.28
126.80
125.71
124.99
114.05
113.55
113.38
106.04
77.39
77.28
77.08
76.76
28.90
12.50
BRUKER
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Sample Name 2015-0306xie2
Position P1-E1
Instrument Name Instrument 1
User Name
Inj Vol -1
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename 2015-0306xie2.d
ACQ Method 0103.m
Comment
Acquired Time 3/6/2015 3:45:31 PM
+ESI Scan (1.000 min) Frag=135.0V 2015-0306xie2.d Subtract (6)
* 197.1073
Counts vs. Mass-to-Charge (m/z)

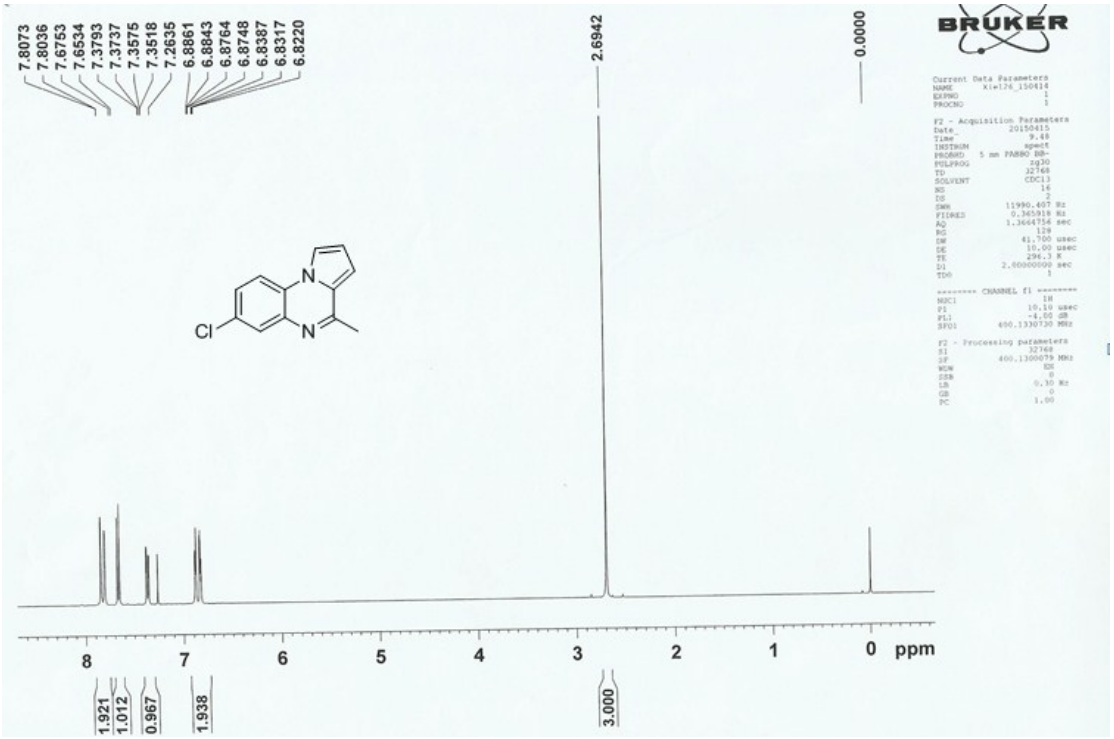
4,7-dimethylpyrrolo[1,2-*a*]quinoxaline (4n)

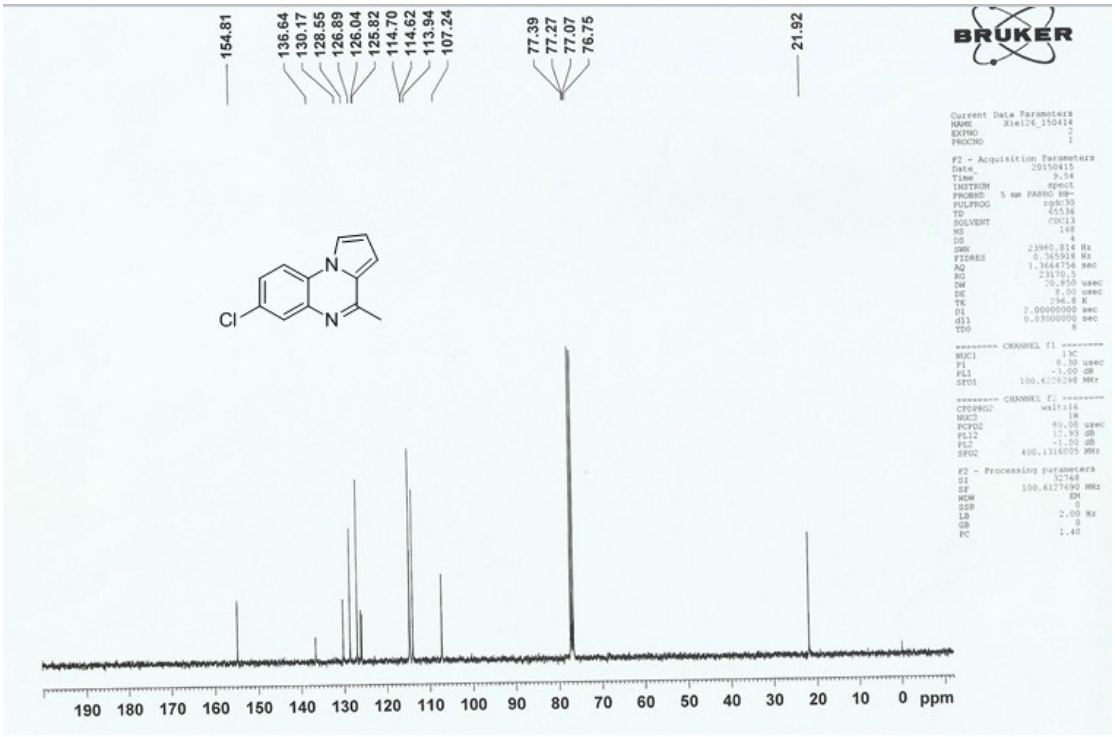


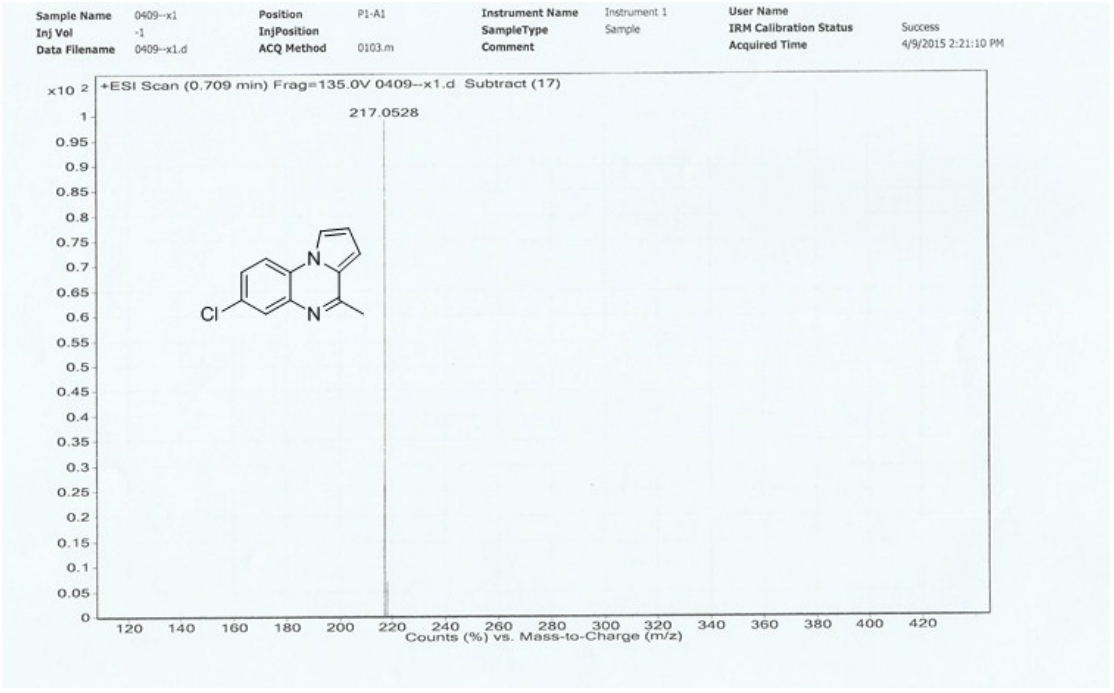




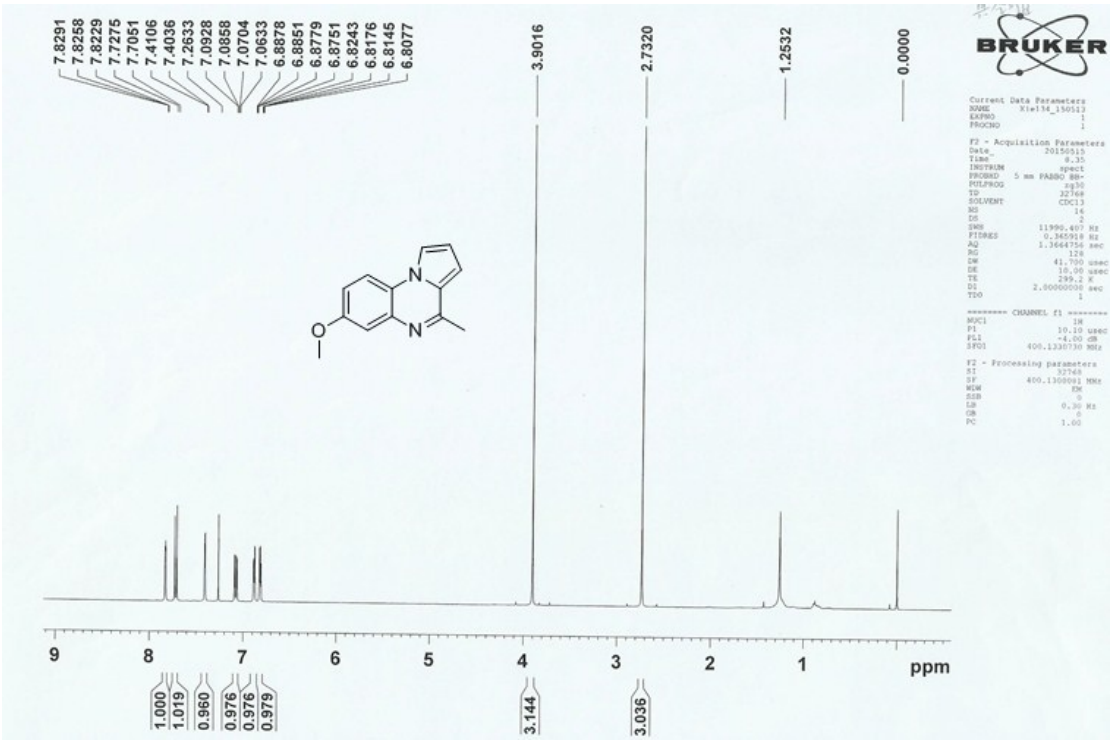
7-chloro-4-methylpyrrolo[1,2-*a*]quinoxaline (4o)

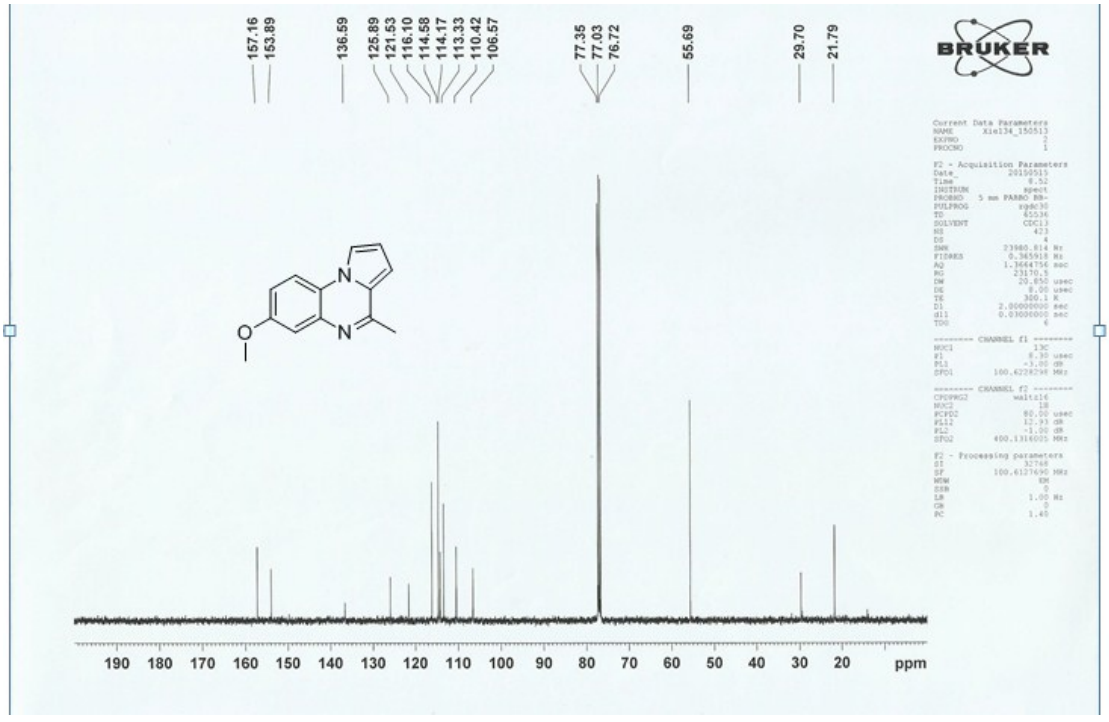


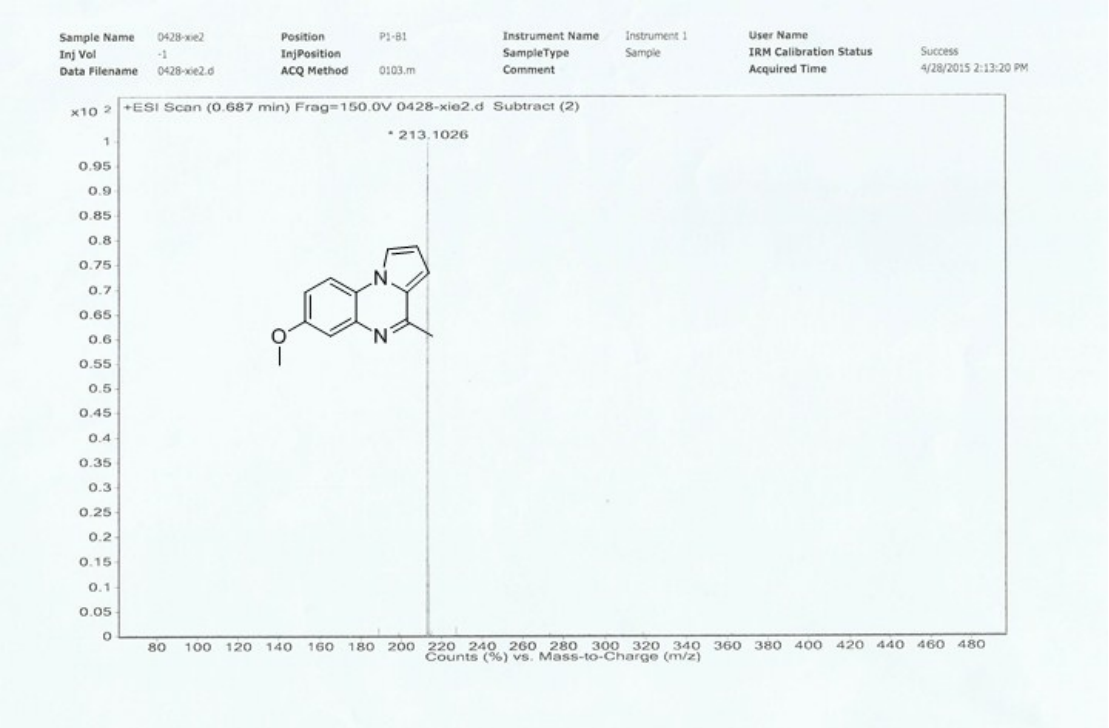

154.81
136.64
130.17
128.55
126.89
126.04
125.82
114.70
114.62
113.94
107.24
77.39
77.27
77.07
76.75
21.92
BRUKER
Cl
N
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Sample Name 0409--x1
Position P1-A1
Instrument Name Instrument 1
User Name
Inj Vol -1
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename 0409--x1.d
ACQ Method 0103.m
Comment
Acquired Time 4/9/2015 2:21:10 PM
+ESI Scan (0.709 min) Frag=135.0V 0409--x1.d Subtract (17)
x10 2
217.0528
Cl
N
N
Counts (%) vs. Mass-to-Charge (m/z)

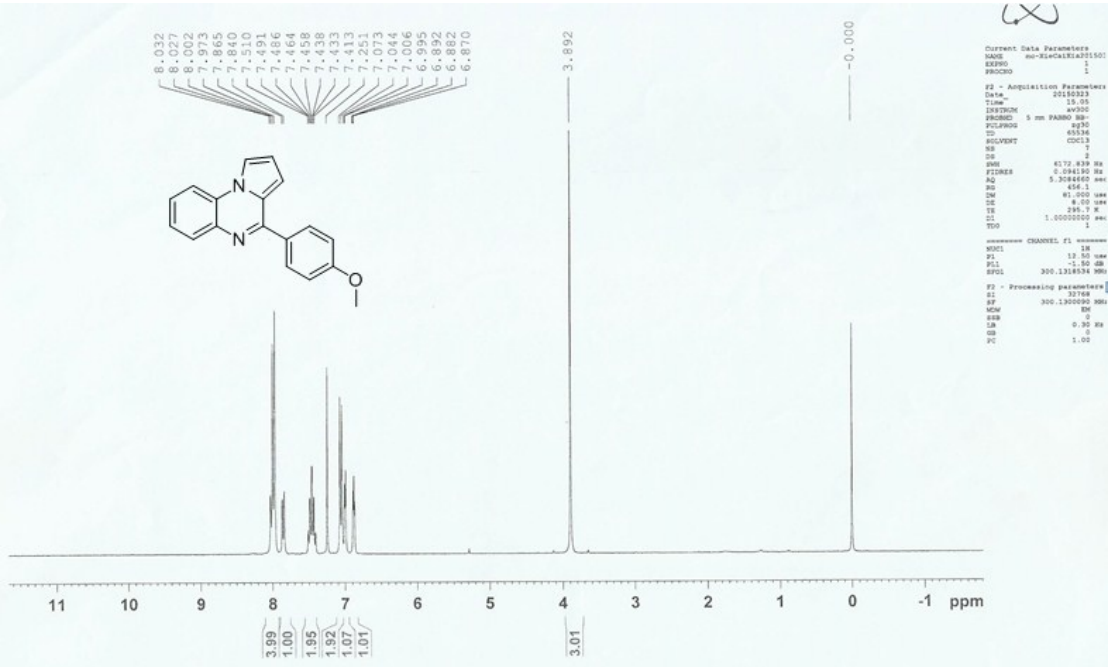
7-methoxy-4-methylpyrrolo[1,2-*a*]quinoxaline (4p)

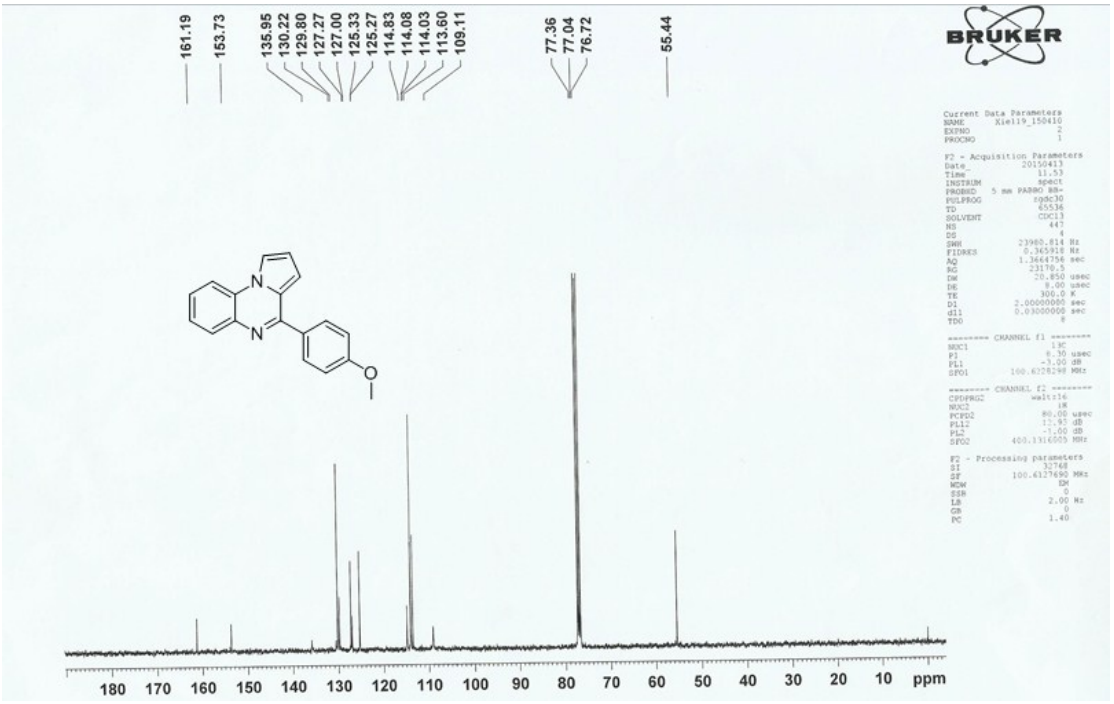


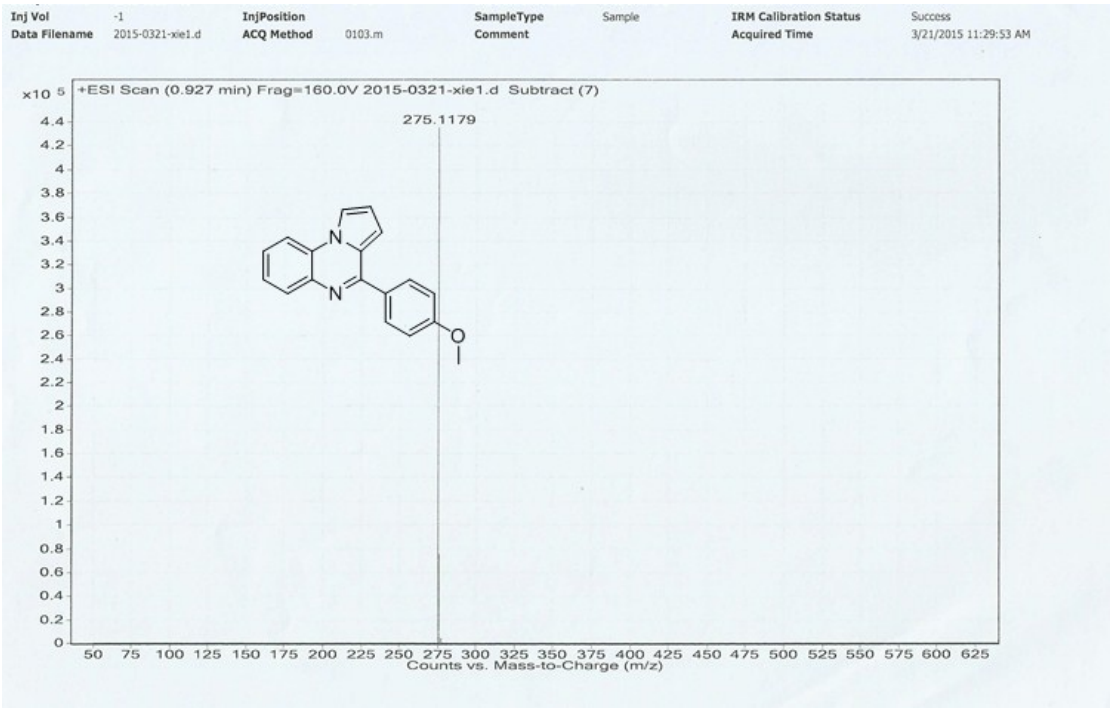




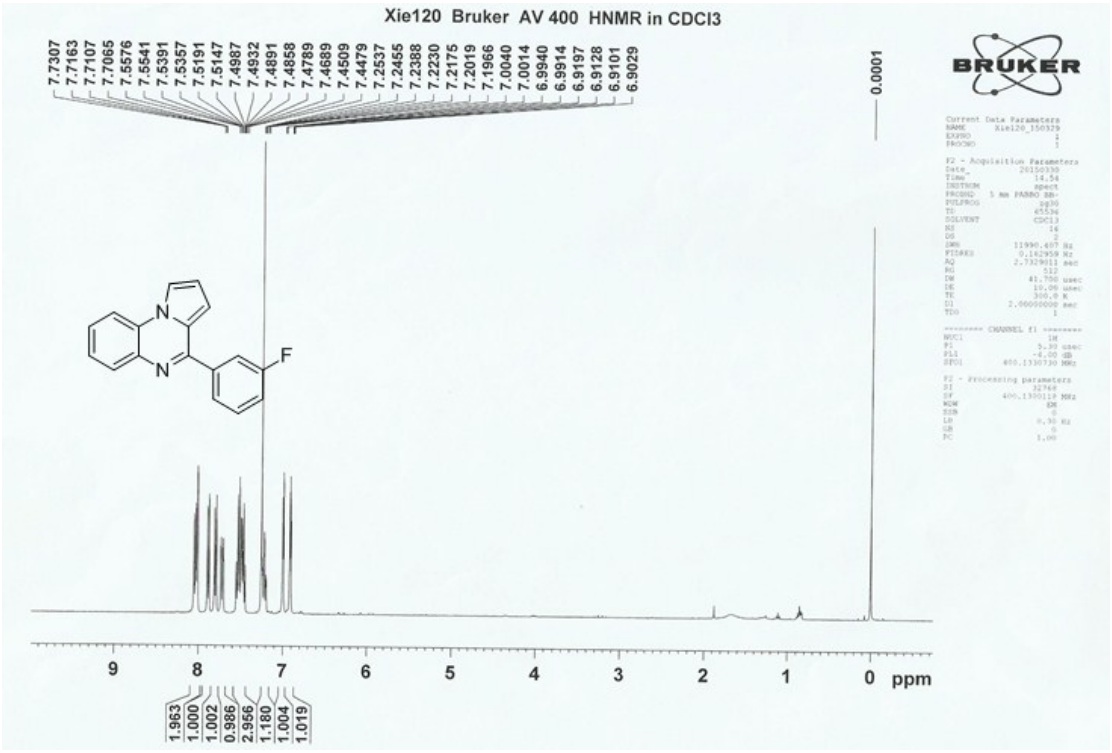
4-(4-methoxyphenyl)pyrrolo[1,2-*a*]quinoxaline (4q)

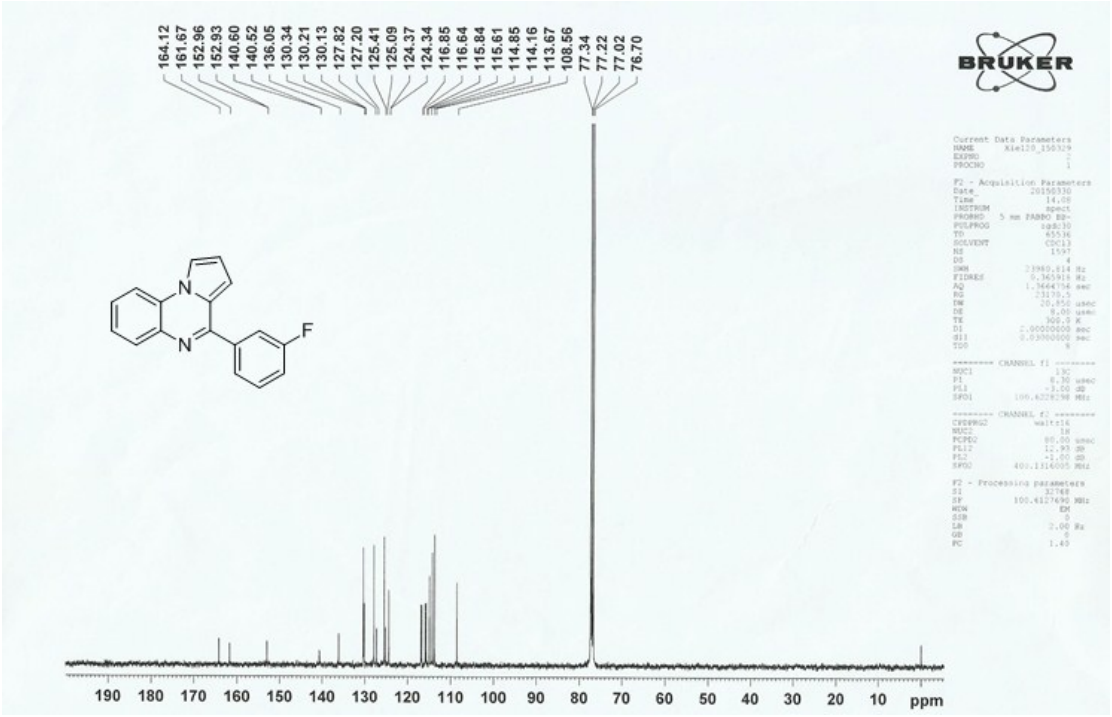


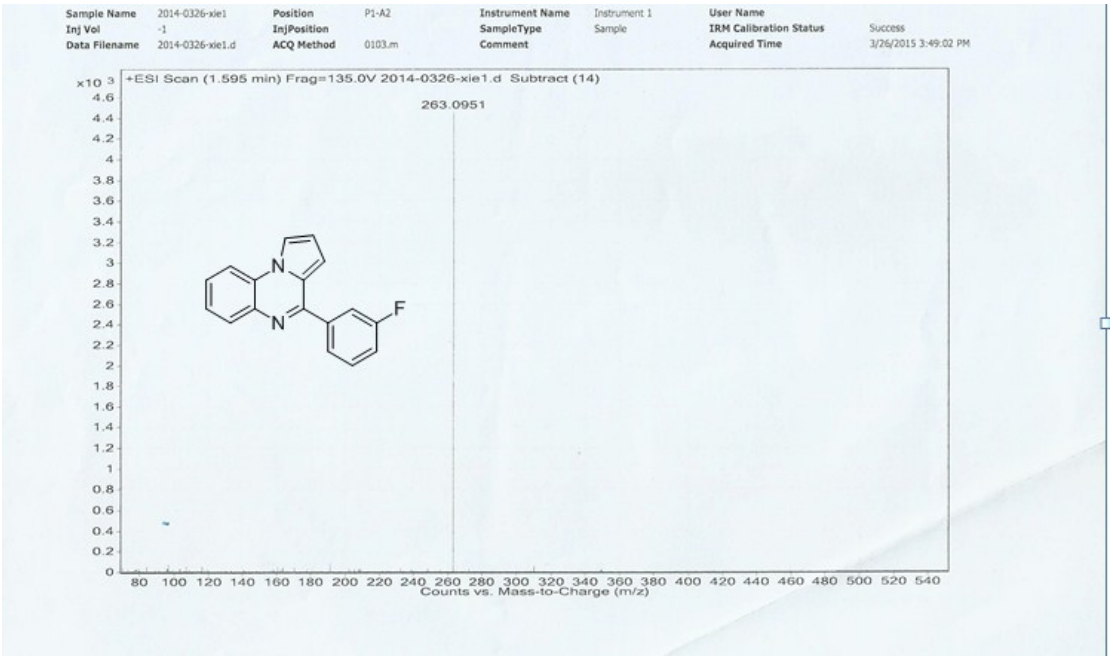

161.19
153.73
135.95
130.22
129.80
127.27
127.00
125.33
125.27
114.83
114.08
114.03
113.60
109.11
77.36
77.04
76.72
55.44
BRUKER
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Inj Vol -1
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename 2015-0321-xie1.d
ACQ Method 0103.m
Comment
Acquired Time 3/21/2015 11:29:53 AM
+ESI Scan (0.927 min) Frag=160.0V 2015-0321-xie1.d Subtract (7)
275.1179
x10 5
Counts vs. Mass-to-Charge (m/z)

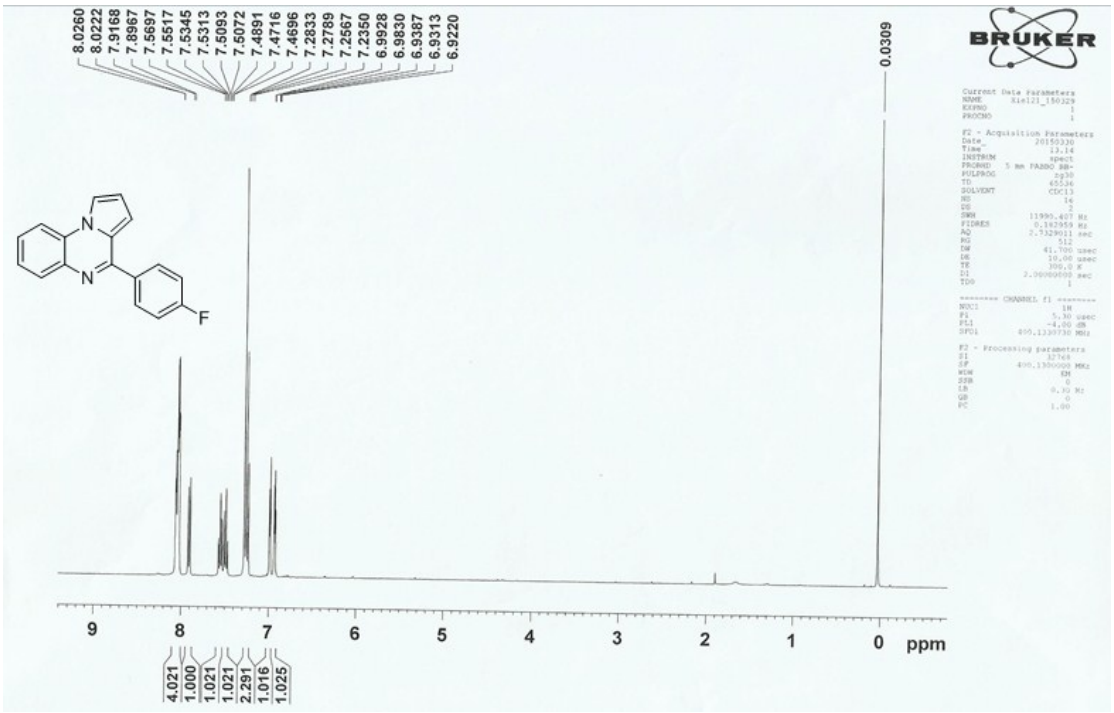
4-(3-fluorophenyl)pyrrolo[1,2-*a*]quinoxaline (4r)

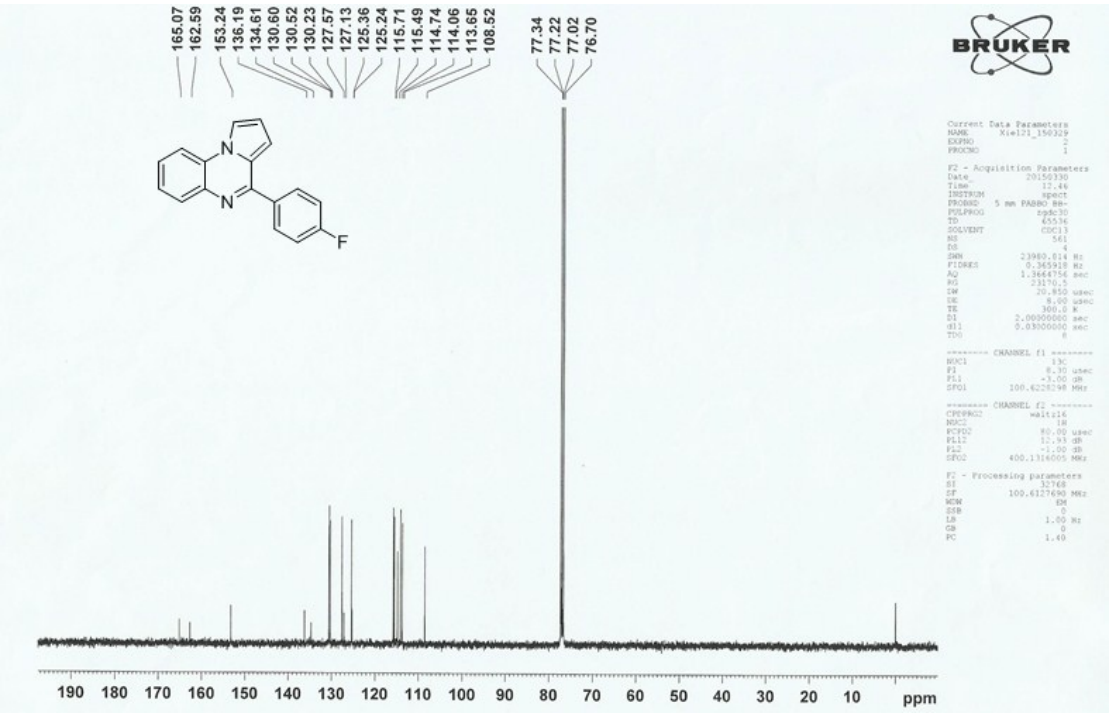


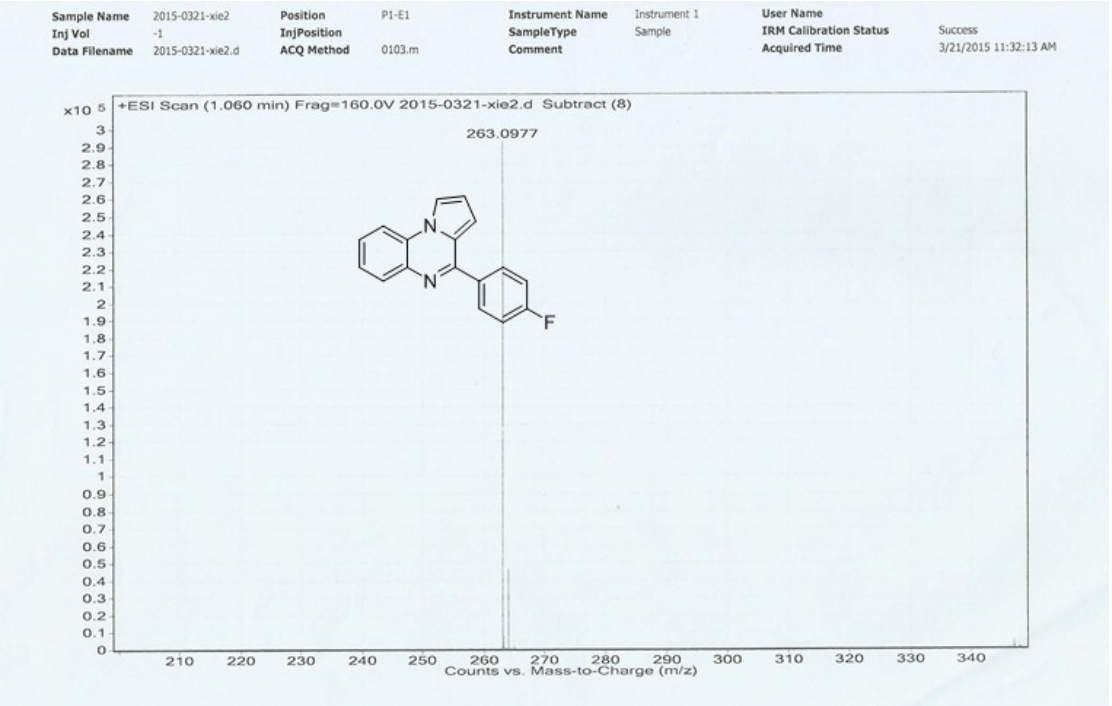




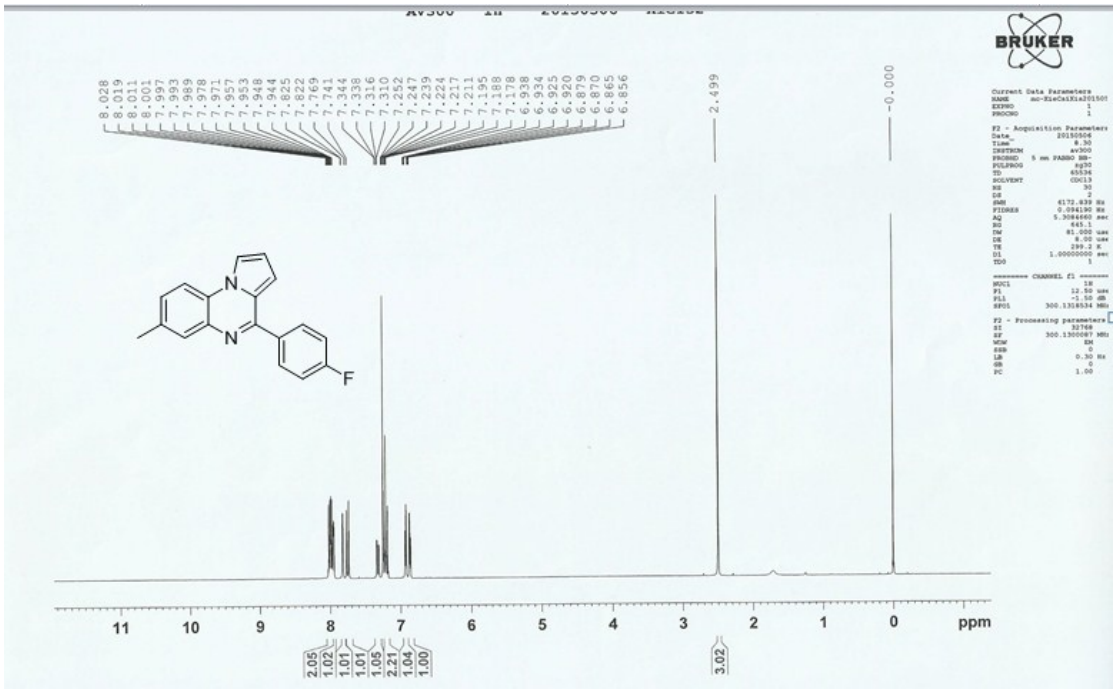
4-(4-fluorophenyl)pyrrolo[1,2-*a*]quinoxaline (4s)

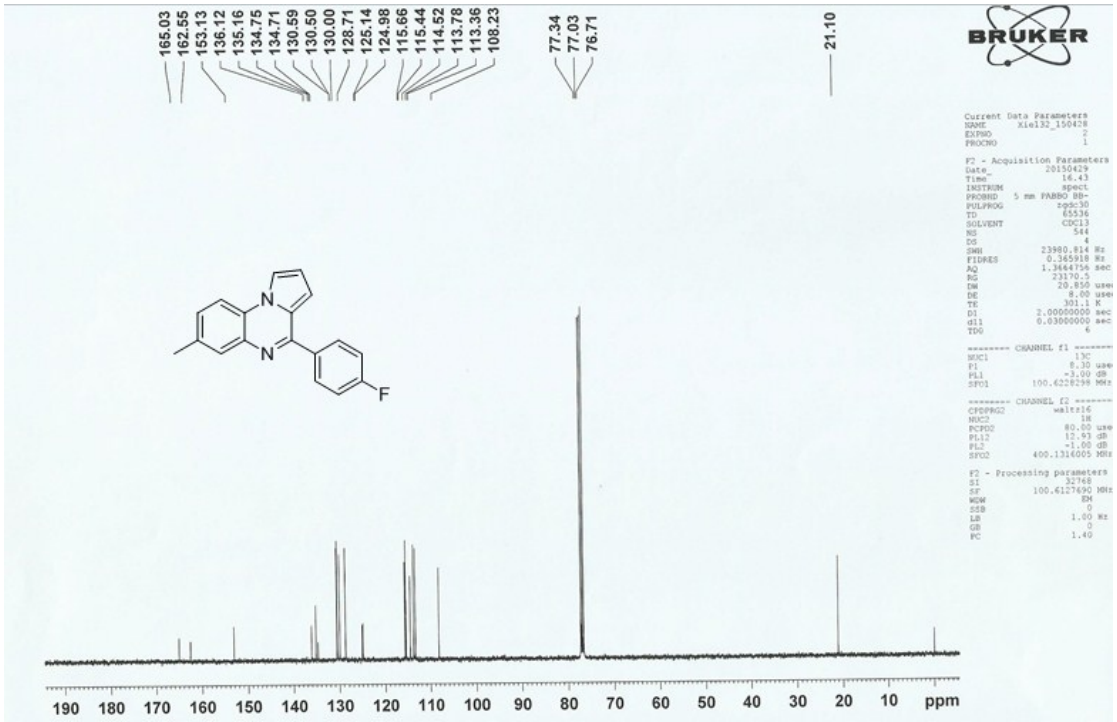


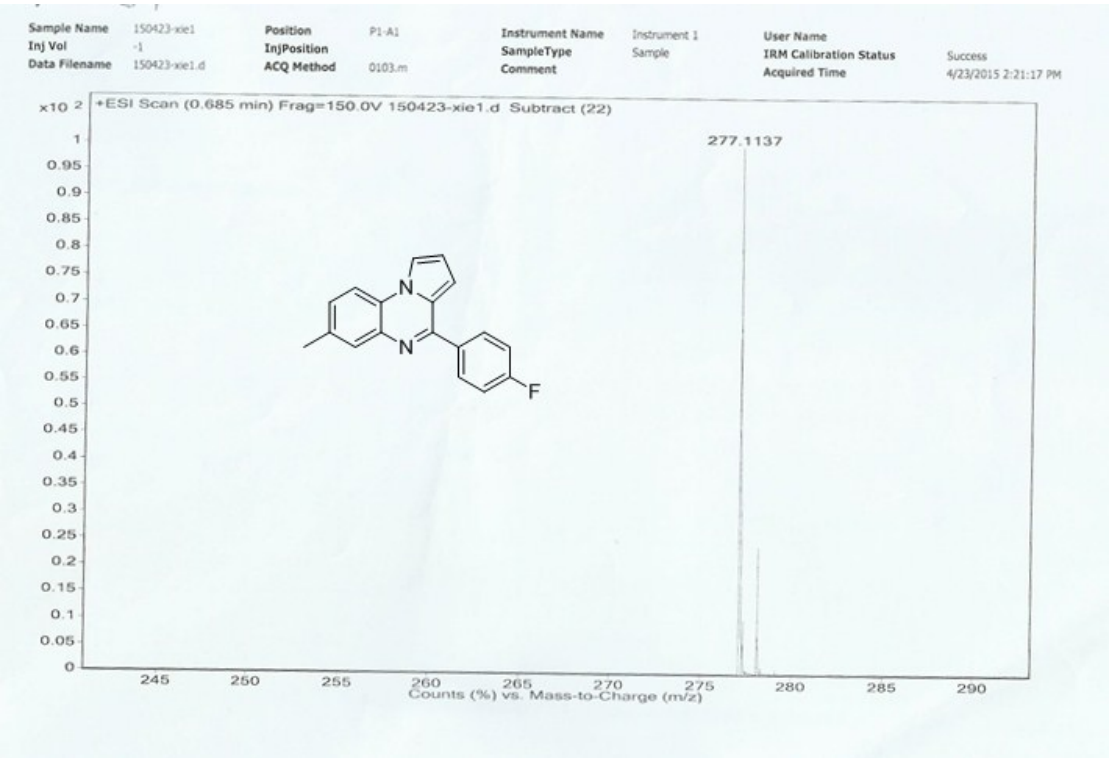

165.07
162.59
153.24
136.19
134.61
130.60
130.52
130.23
127.57
127.13
125.36
125.24
115.71
115.49
114.74
114.06
113.65
108.52
77.34
77.22
77.02
76.70
BRUKER
N
N
F
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Sample Name 2015-0321-xie2
Position P1-E1
Instrument Name Instrument 1
User Name
Inj Vol -1
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename 2015-0321-xie2.d
ACQ Method 0103.m
Comment
Acquired Time 3/21/2015 11:32:13 AM
+ESI Scan (1.060 min) Frag=160.0V 2015-0321-xie2.d Subtract (8)
x10 5
263.0977
N
N
F
Counts vs. Mass-to-Charge (m/z)

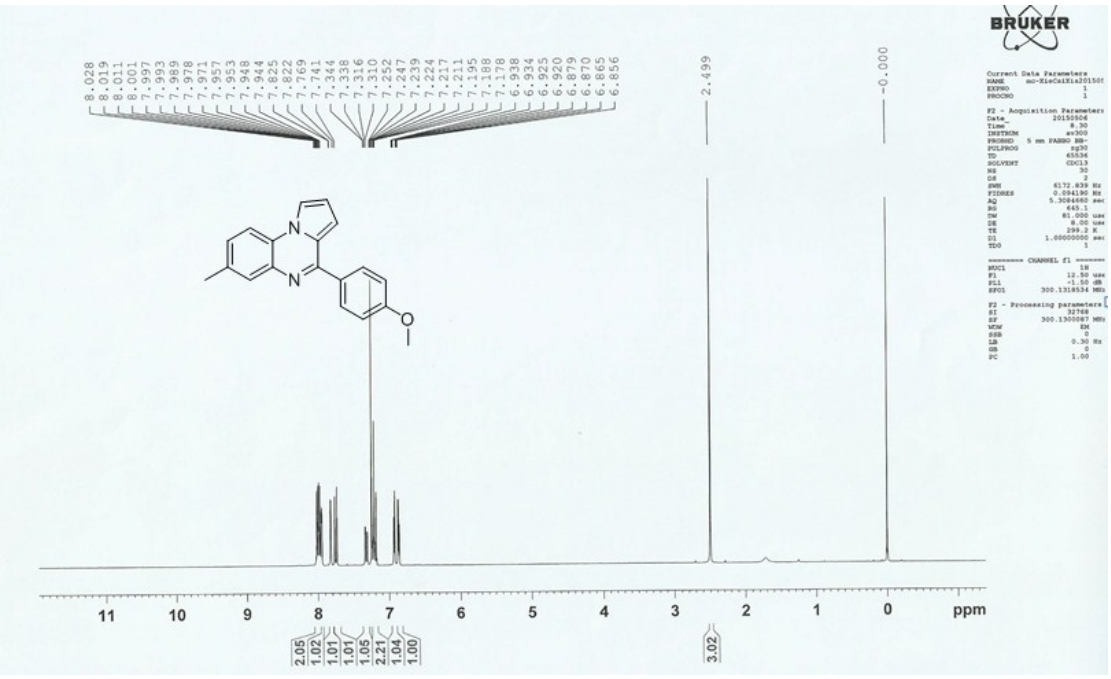
4-(4-fluorophenyl)-7-methylpyrrolo[1,2-*a*]quinoxaline (4t)

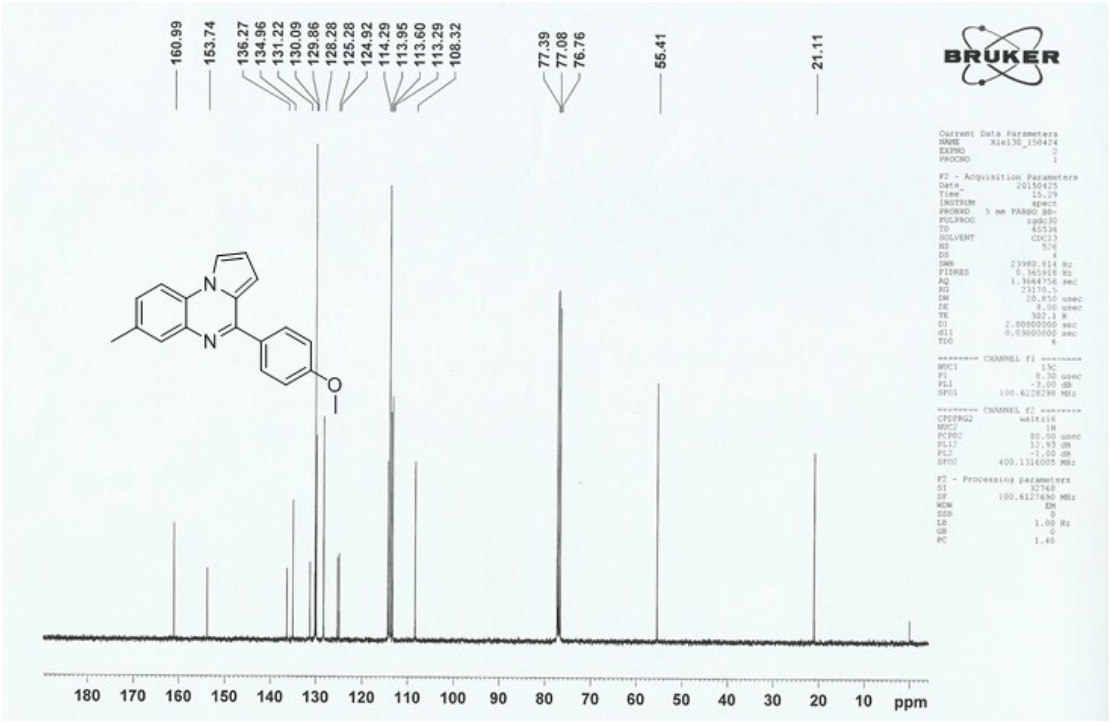


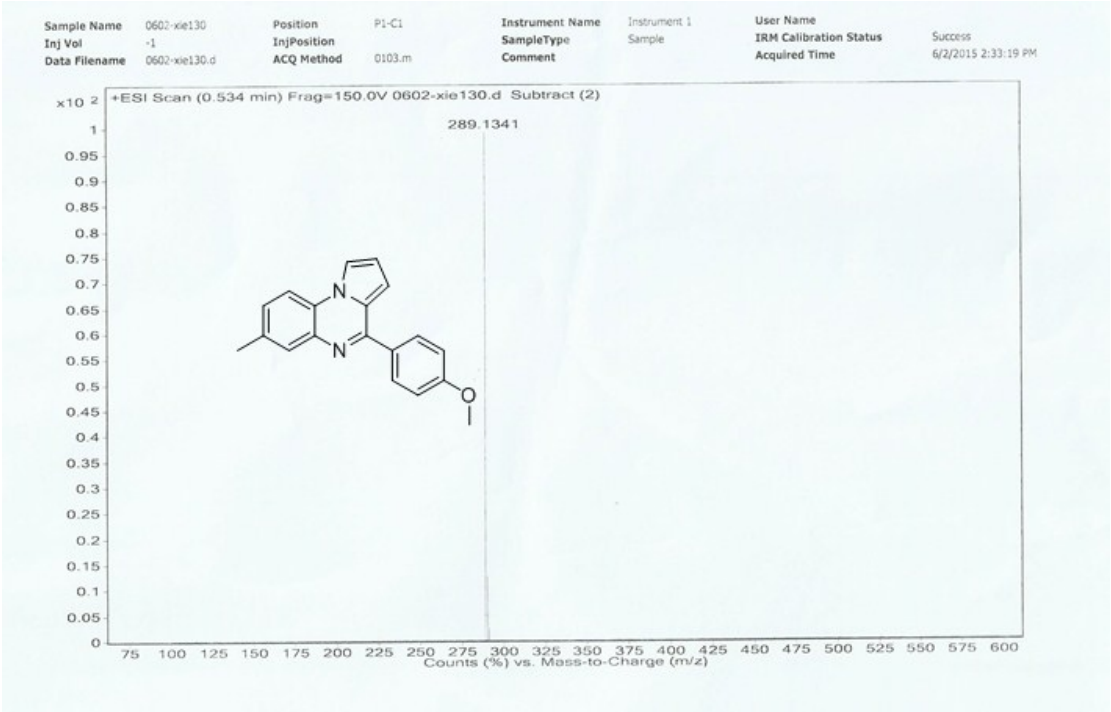




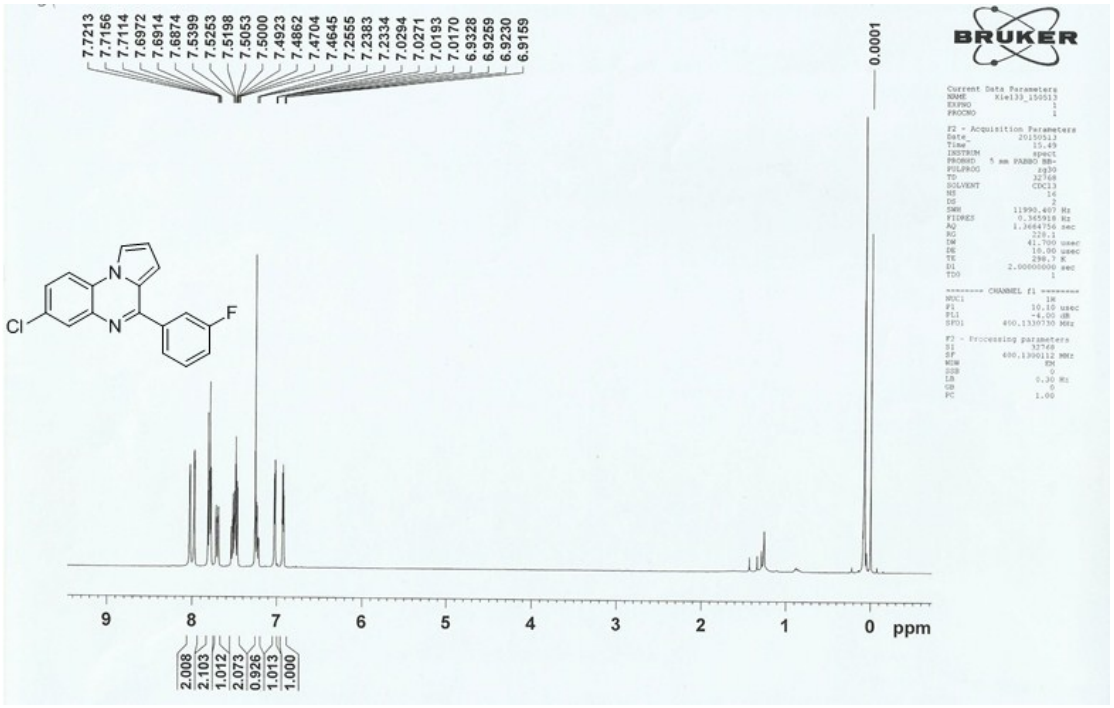
4-(4-methoxyphenyl)-7-methylpyrrolo[1,2-*a*]quinoxaline (4u)

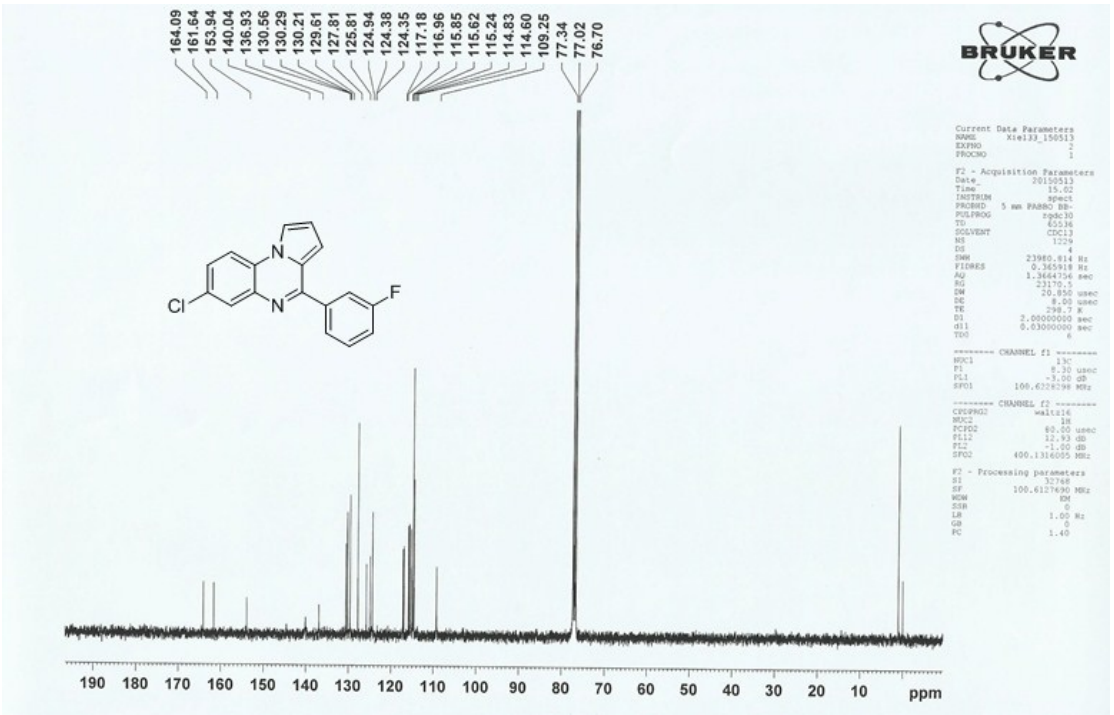


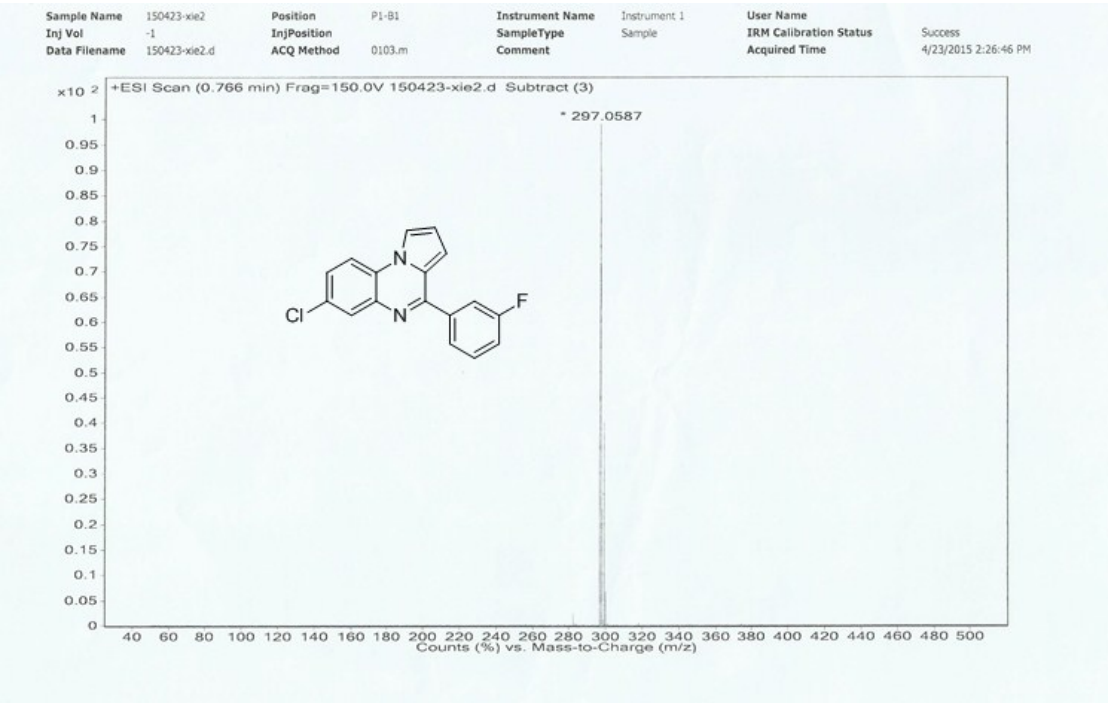

160.99
153.74
136.27
134.96
131.22
130.09
129.86
128.28
125.28
124.92
114.29
113.95
113.60
113.29
108.32
77.39
77.08
76.76
55.41
21.11
BRUKER
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Sample Name 0602-xie130
Inj Vol -1
Data Filename 0602-xie130.d
Position P1-C1
InjPosition
ACQ Method 0103.m
Instrument Name Instrument 1
SampleType Sample
Comment
User Name
IRM Calibration Status Success
Acquired Time 6/2/2015 2:33:19 PM
+ESI Scan (0.534 min) Frag=150.0V 0602-xie130.d Subtract (2)
289.1341
x10 2
Counts (%) vs. Mass-to-Charge (m/z)

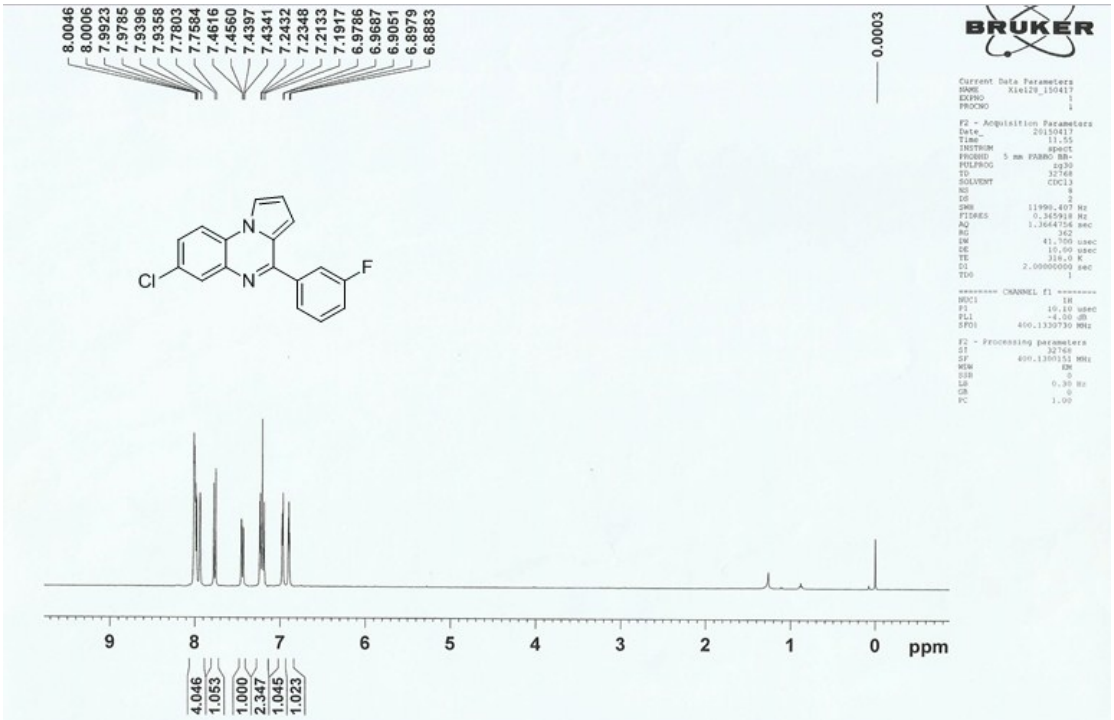
7-chloro-4-(3-fluorophenyl)pyrrolo[1,2-*a*]quinoxaline (4v)

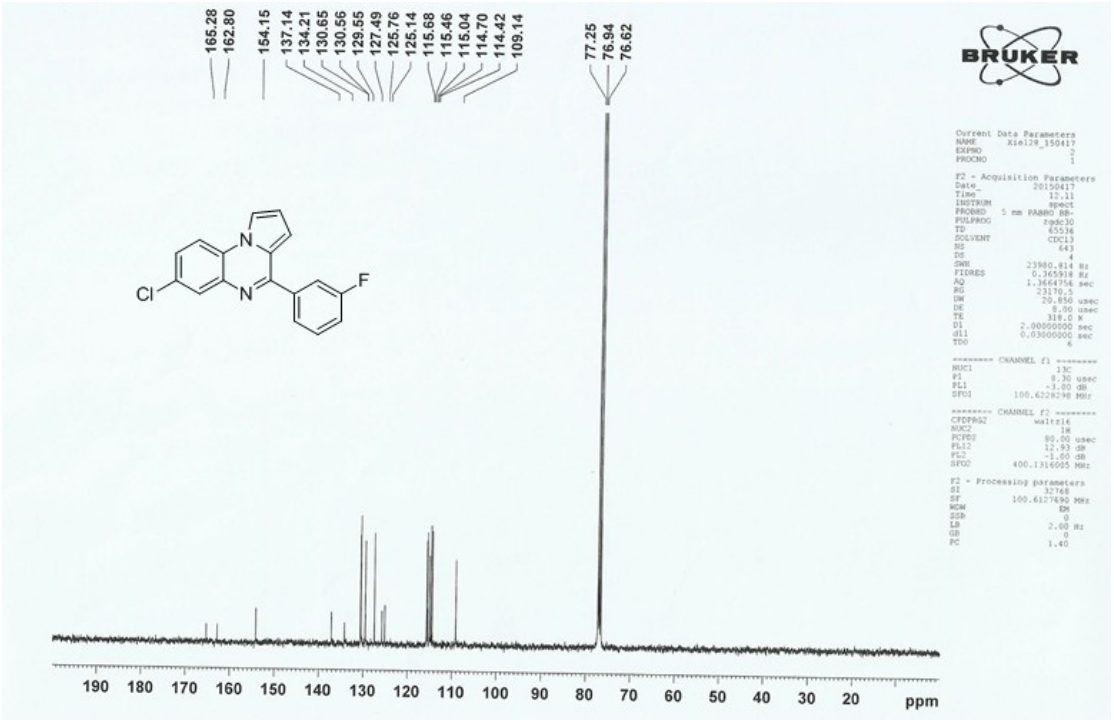


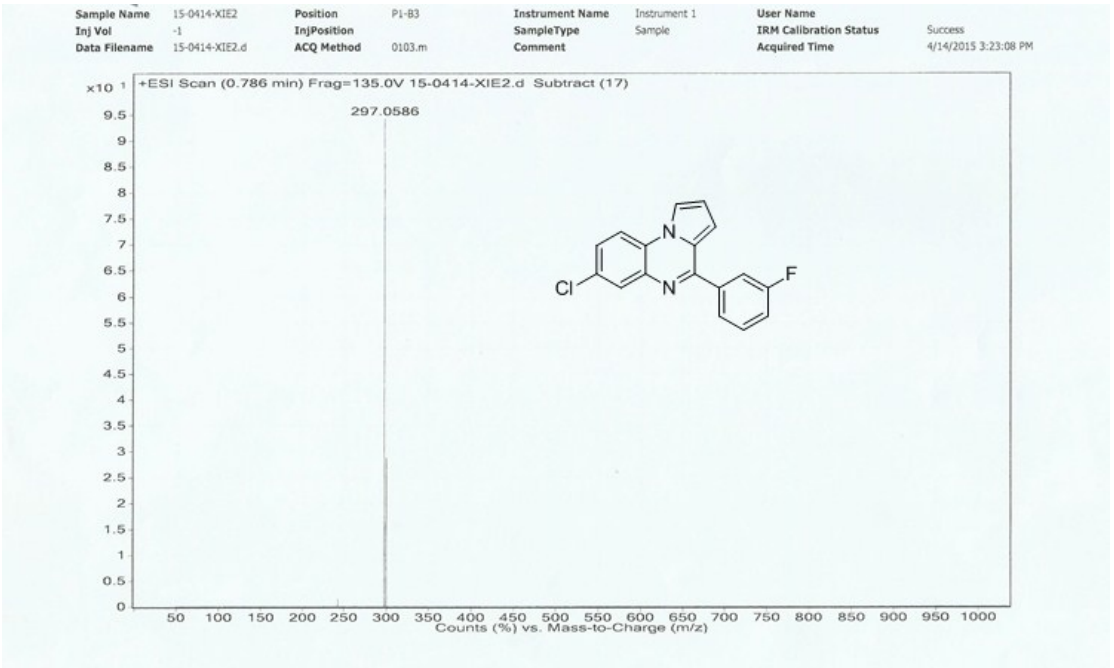




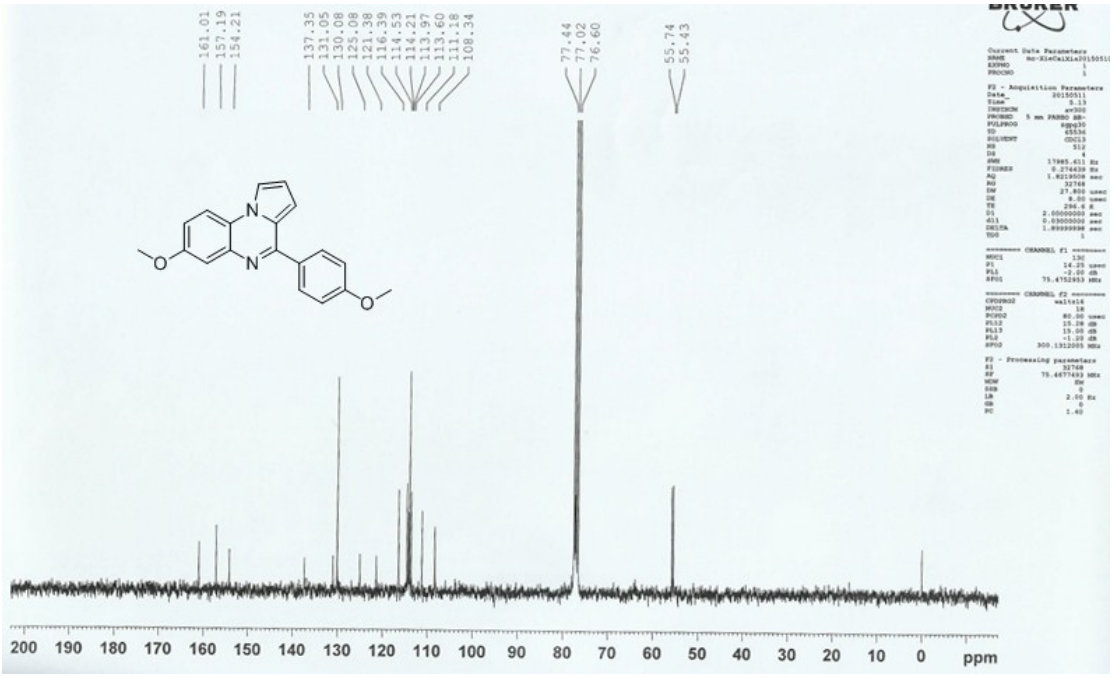
7-chloro-4-(4-fluorophenyl)pyrrolo[1,2-*a*]quinoxaline (4w)

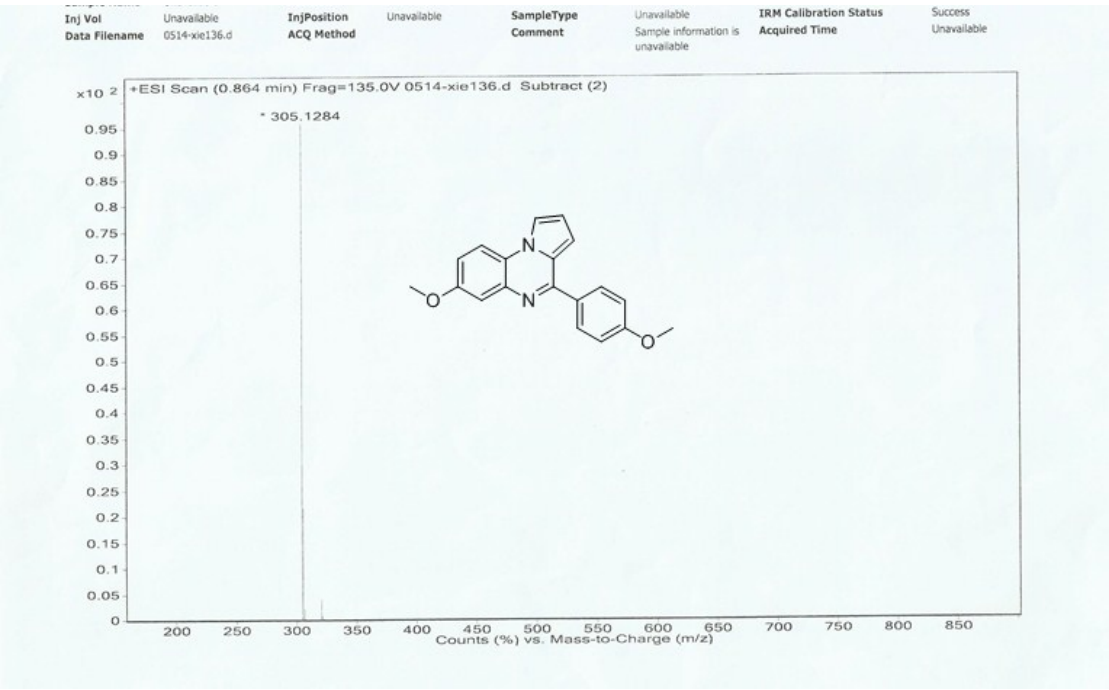



165.28
162.80
154.15
137.14
134.21
130.65
130.56
129.55
127.49
125.76
125.14
115.68
115.46
115.04
114.70
114.42
109.14
77.25
76.94
76.62
BRUKER
Cl
N
N
F
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

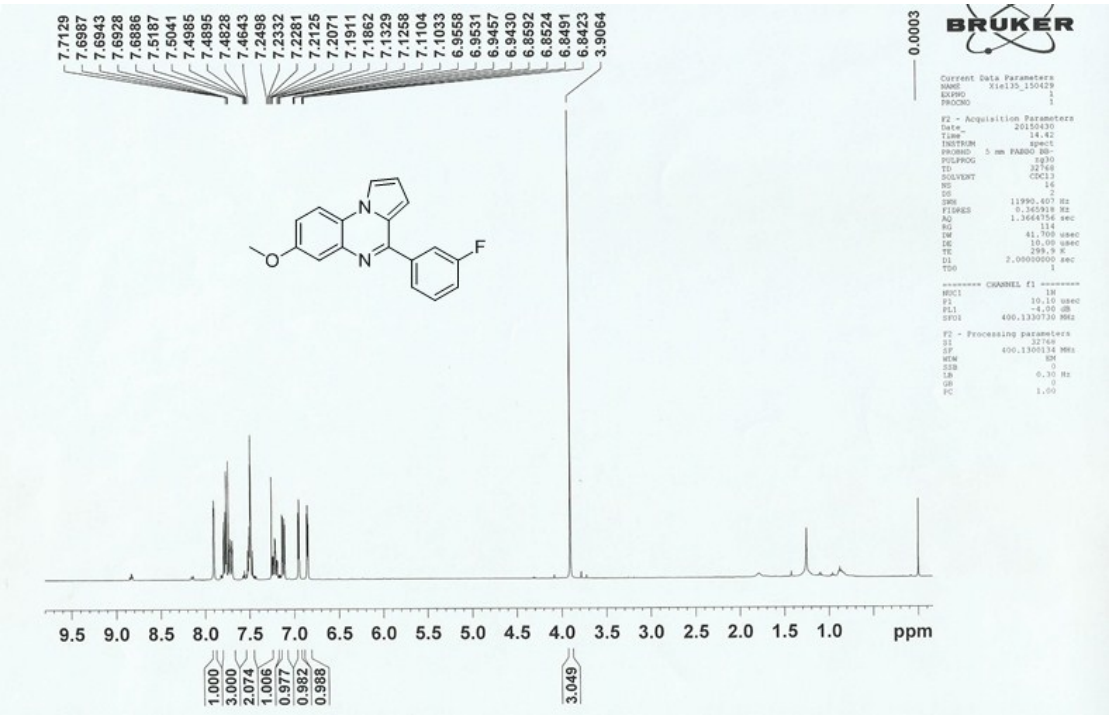

Sample Name 15-0414-XIE2
Position P1-B3
Instrument Name Instrument 1
User Name
Inj Vol -1
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename 15-0414-XIE2.d
ACQ Method 0103.m
Comment
Acquired Time 4/14/2015 3:23:08 PM
+ESI Scan (0.786 min) Frag=135.0V 15-0414-XIE2.d Subtract (17)
297.0586
x10 1
Cl
N
N
F
Counts (%) vs. Mass-to-Charge (m/z)

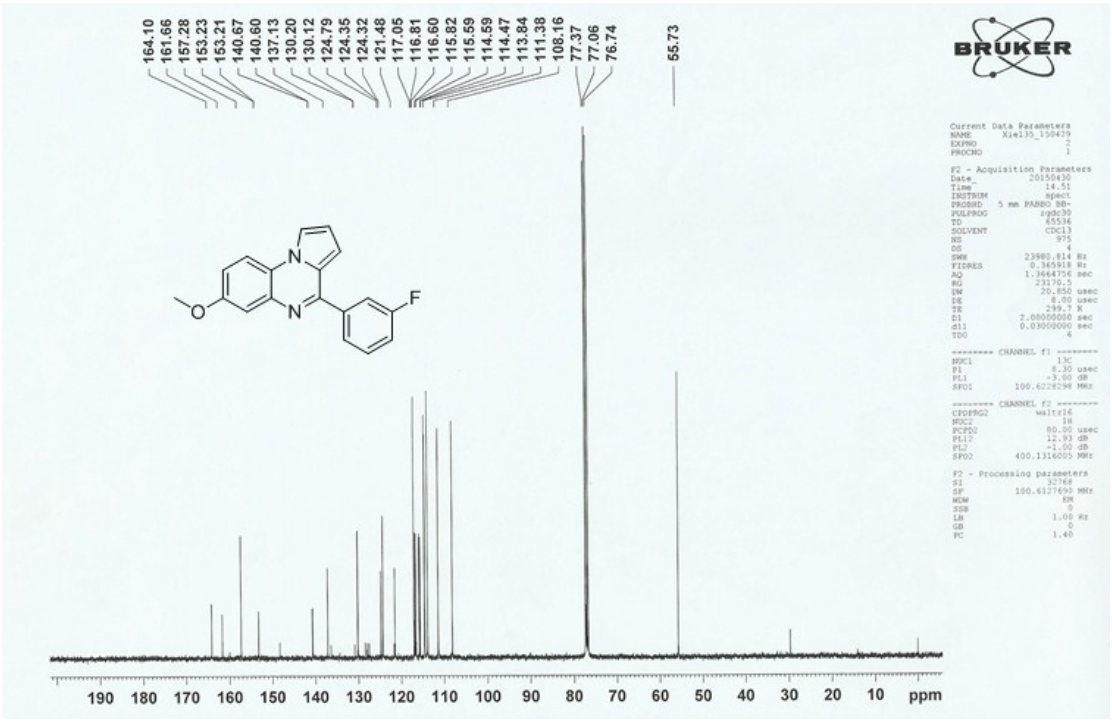
7-methoxy-4-(4-methoxyphenyl)pyrrolo[1,2-*a*]quinoxaline (4x)

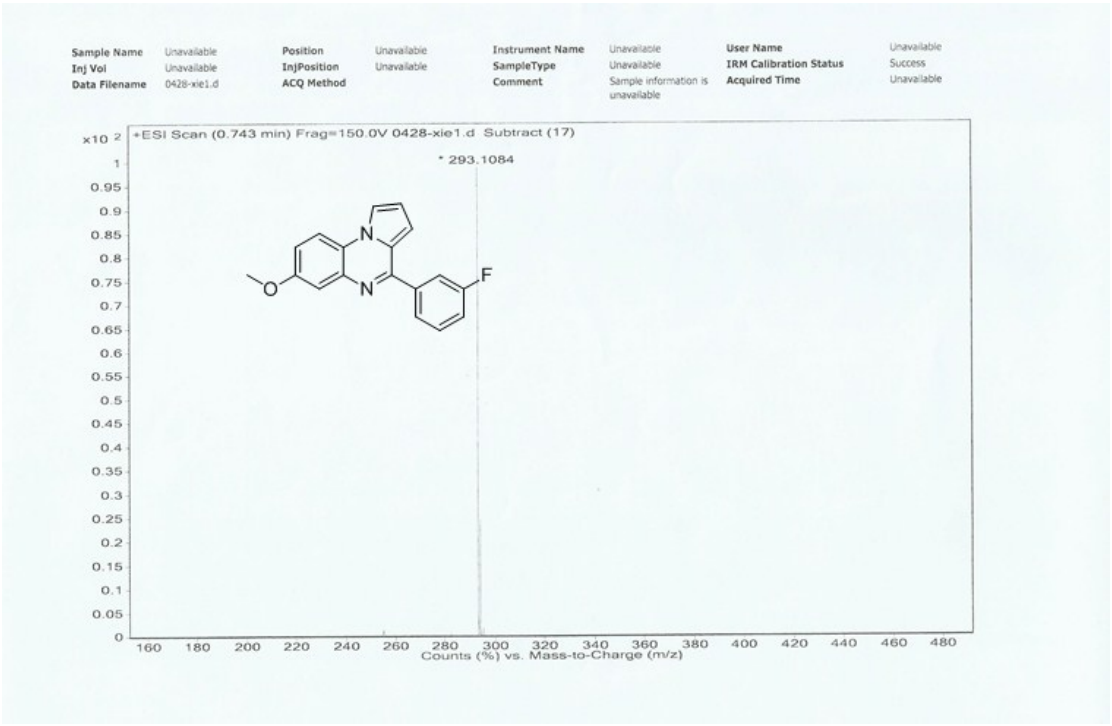




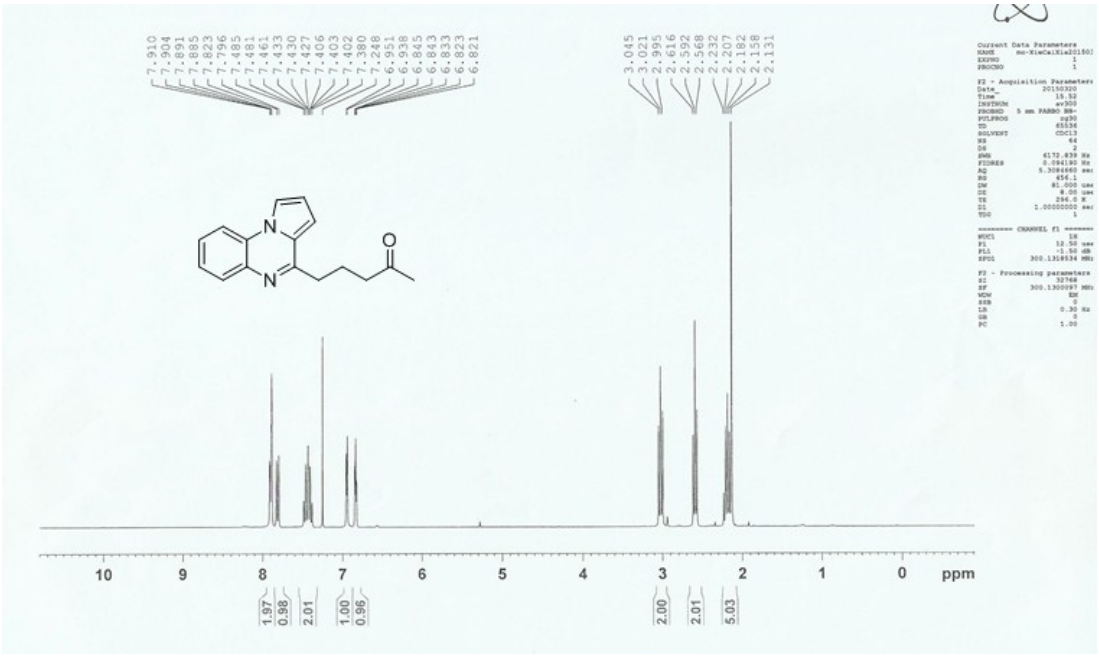
4-(3-fluorophenyl)-7-methoxypyrrolo[1,2-*a*]quinoxaline (4y)

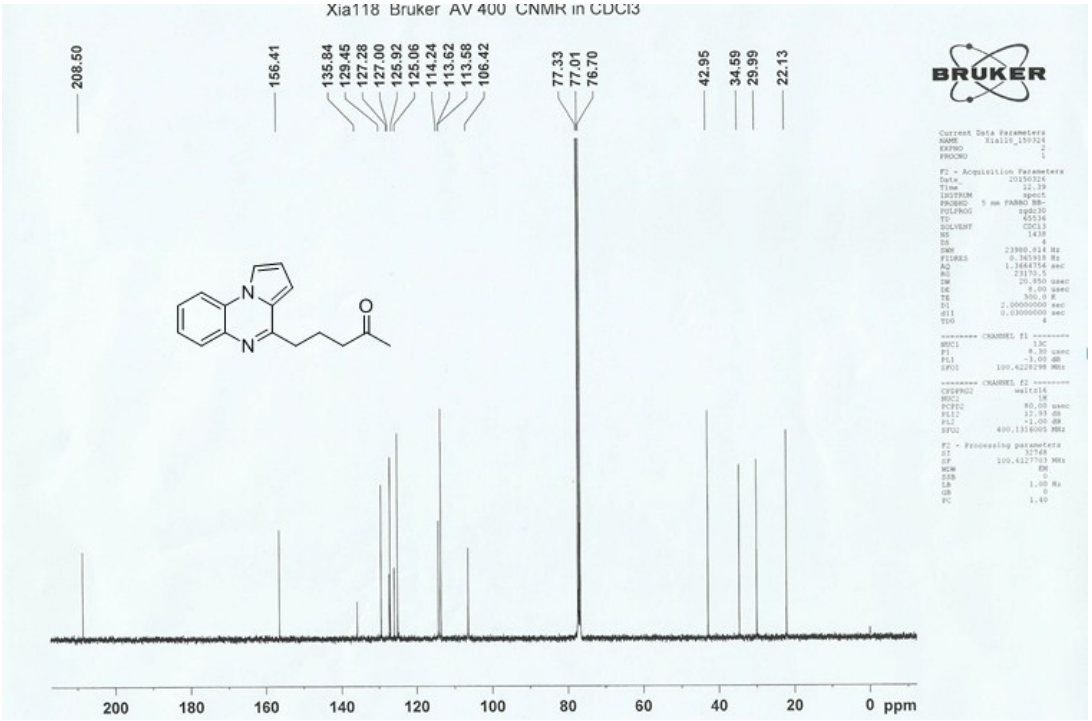


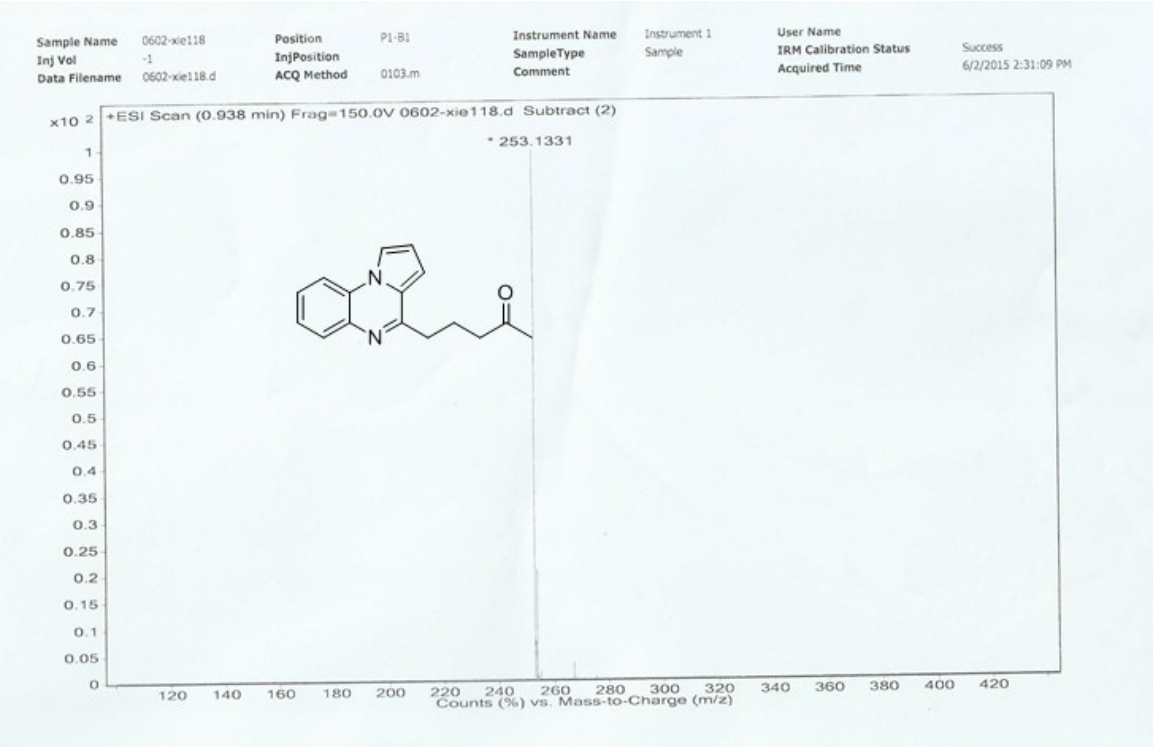

164.10
161.66
157.28
153.23
153.21
140.67
140.60
137.13
130.20
130.12
124.79
124.35
124.32
121.48
117.05
116.81
116.60
115.82
115.59
114.59
114.47
113.84
111.38
108.16
77.37
77.06
76.74
55.73
BRUKER
Current Data Parameters
NAME Xie135_150429
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150430
Time 14.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 975
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 23170.5
DW 20.850 usec
DE 8.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 6
======== CHANNEL f1 ========
NUC1 13C
P1 8.30 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 12.93 dB
PL2 -1.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
F
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Sample Name Unavailable
Position Unavailable
Instrument Name Unavailable
User Name Unavailable
Inj Vol Unavailable
InjPosition Unavailable
SampleType Unavailable
IRM Calibration Status Success
Data Filename 0428-xie1.d
ACQ Method
Comment Sample information is unavailable
Acquired Time Unavailable
+ESI Scan (0.743 min) Frag=150.0V 0428-xie1.d Subtract (17)
* 293.1084
x10 2
1
0.95
0.9
0.85
0.8
0.75
0.7
0.65
0.6
0.55
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480
Counts (%) vs. Mass-to-Charge (m/z)
N
O
F

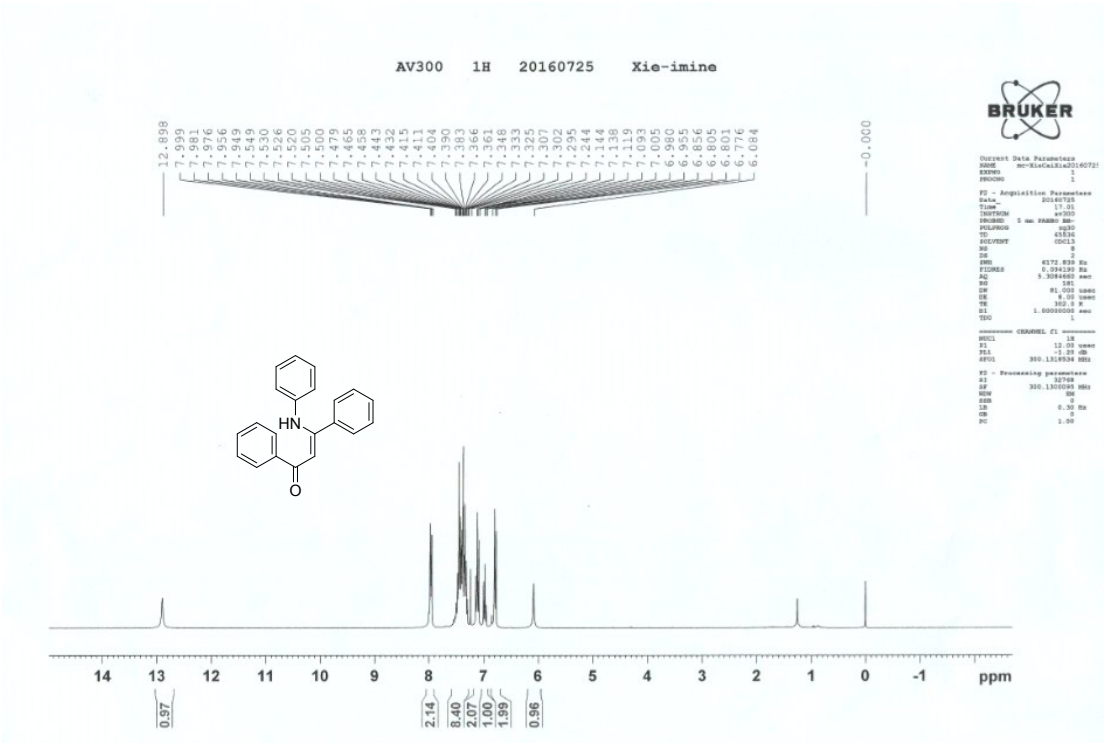
5-(pyrrolo[1,2-*a*]quinoxalin-4-yl)pentan-2-one (4z)

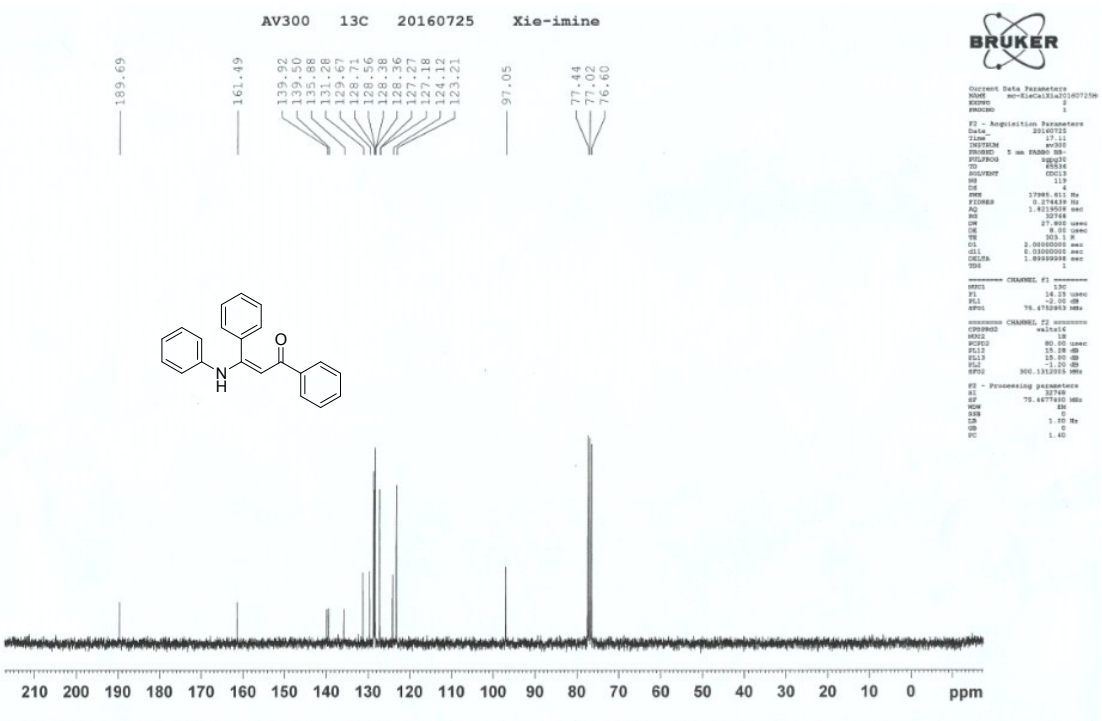


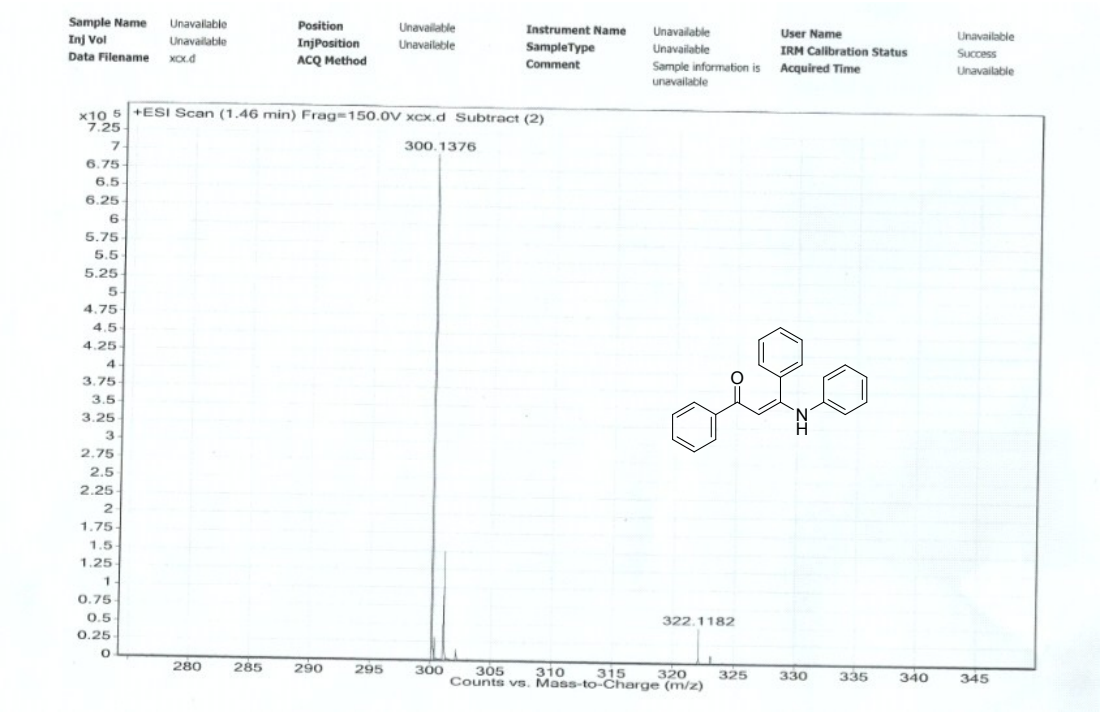




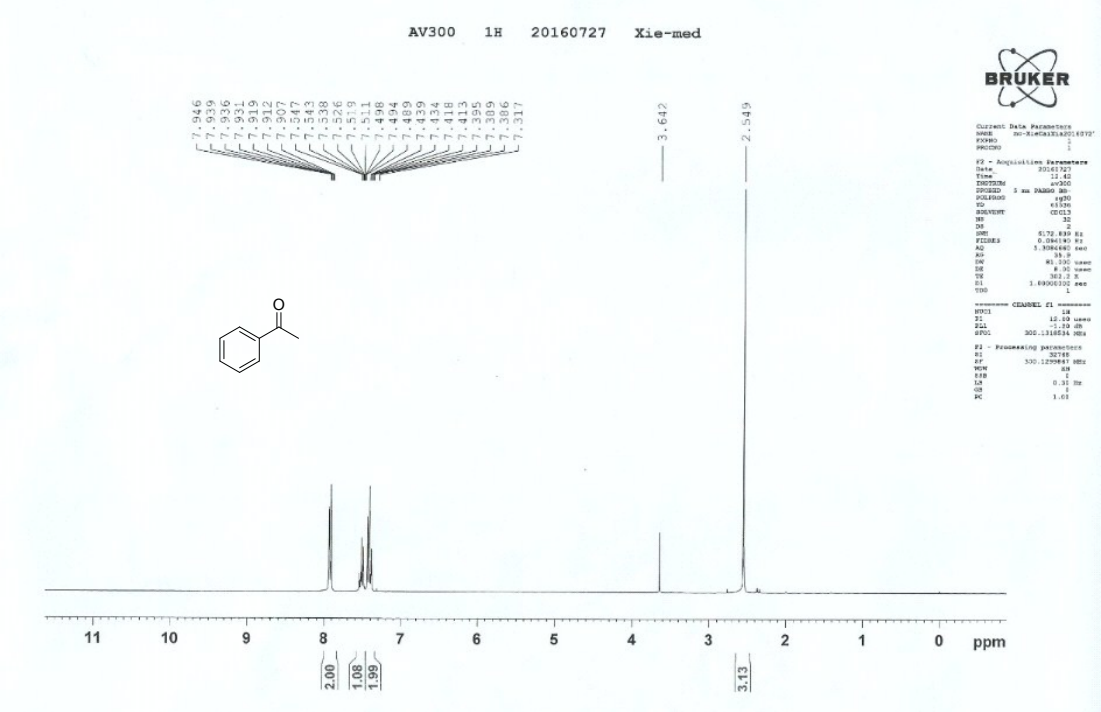
(Z)-1,3-diphenyl-3-(phenylamino)prop-2-en-1-one (5e)



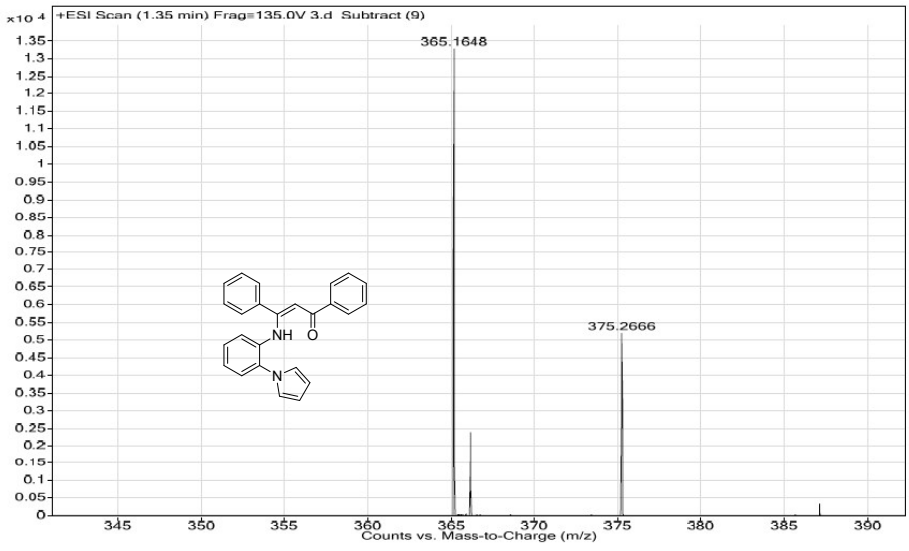

AV300 13C 20160725 Xie-imine
189.69
161.49
139.92
139.50
135.88
131.28
129.67
128.71
128.56
128.38
128.36
127.27
127.18
124.12
123.21
97.05
77.44
77.02
76.60
BRUKER
O
N
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Sample Name Unavailable
Inj Vol Unavailable
Data Filename xcx.d
Position Unavailable
InjPosition Unavailable
ACQ Method
Instrument Name Unavailable
SampleType Unavailable
Comment Sample information is unavailable
User Name Unavailable
IRM Calibration Status Success
Acquired Time Unavailable
x10 5
+ESI Scan (1.46 min) Frag=150.0V xcx.d Subtract (2)
300.1376
322.1182
O
N
H
Counts vs. Mass-to-Charge (m/z)

Acetophenone (5f)



Sample Name	Unavailable	**Position**	Unavailable	**Instrument Name**	Unavailable	**User Name**	Unavailable
Inj Vol	Unavailable	**InjPosition**	Unavailable	**SampleType**	Unavailable	**IRM Calibration Status**	Success
Data Filename	3.d	**ACQ Method**		**Comment**	Sample information is unavailable	**Acquired Time**	Unavailable



4. Reference

1. T. N. P atil, D. R. Kavthe, S. V. Shinde, B. Sridhar, *J. Org. Chem.* 2010, **75**, 3371.

2. Z. Y. Zhang, C. X. Xie, G. L. Song, L. Wen, H. Gao, C. Ma, *Org. Chem. Front.* 2015, **2**, 942.

3. M. Wasa, Y. R. Liu, P. S. Roche, N. E. Jacobsen, *J. Am. Chem. Soc.* 2014, **136**, 12872.