Supporting Information

Metal-Free Synthesis of Aminomethylated Imidazoheterocycles: Dual Role of *tert*-Butyl Hydroperoxide both as an Oxidant and Methylene Source

Om P. S. Patel, Nitesh Kumar Nandwana, Ajay Kumar Sah and Anil Kumar*

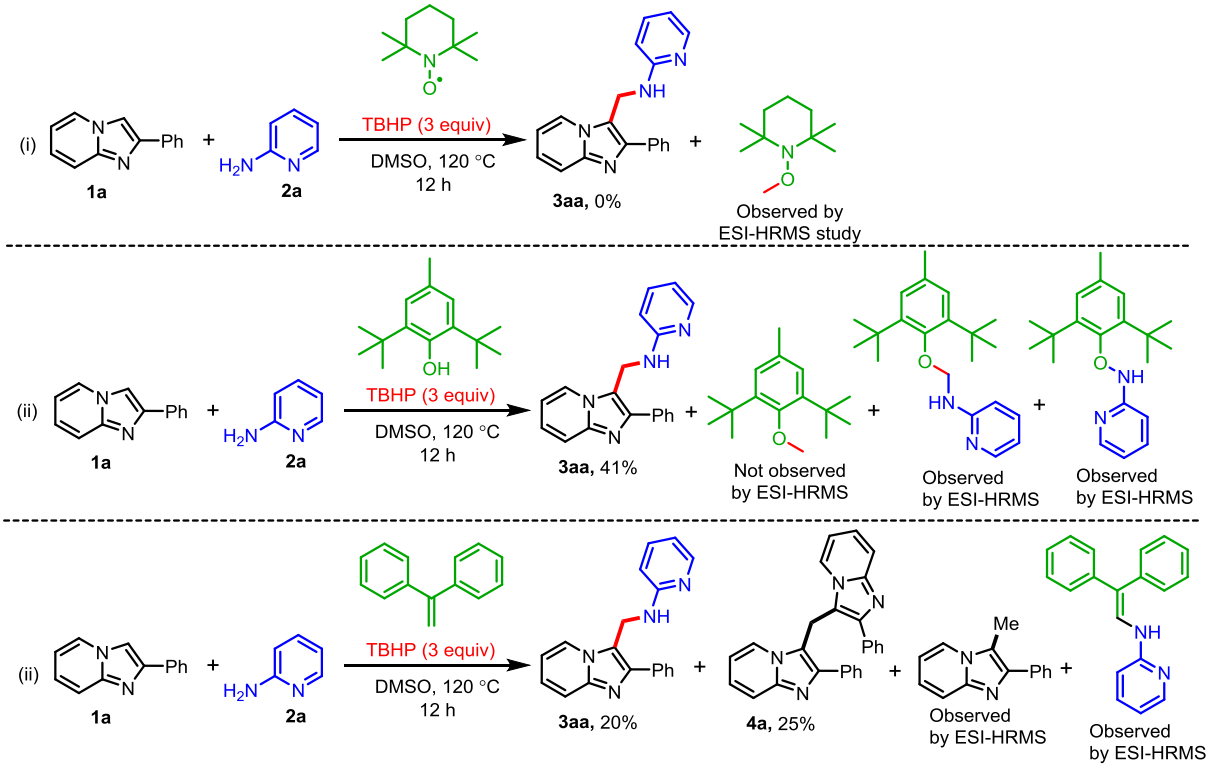
Department of Chemistry, BITS Pilani, Pilani Campus 333031, Rajasthan, India

Corresponding Author

*anilkumar@pilani.bits-pilani.ac.in

Table of Contents

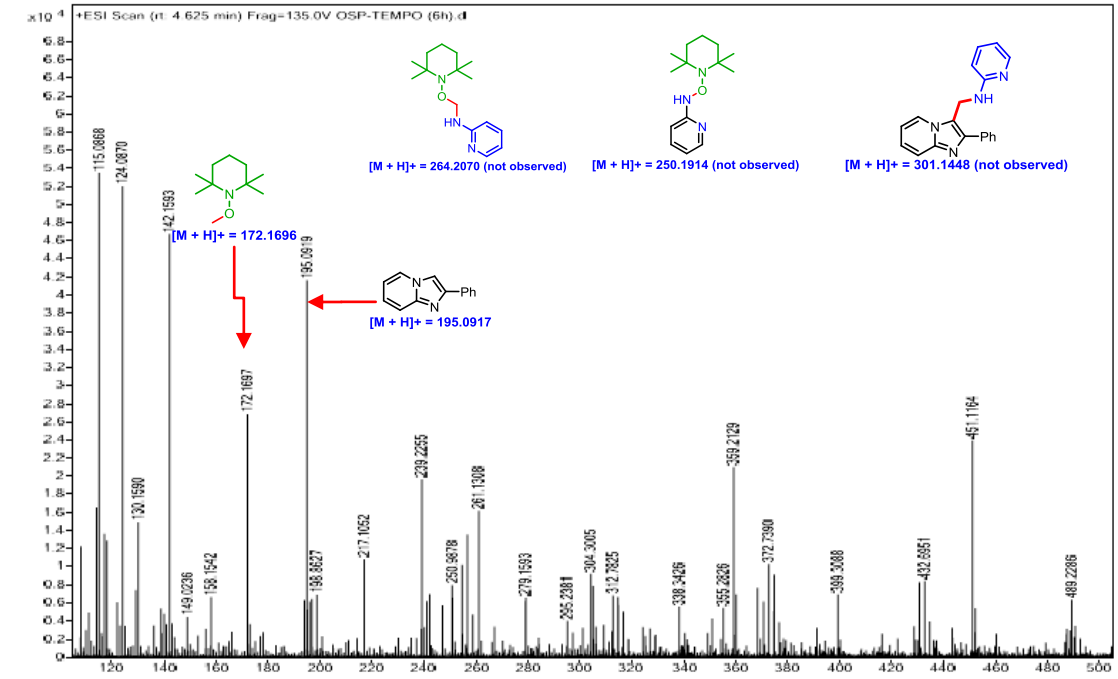
1. Radical trapping experiments



To a stirred solution of 2-phenylimidazo[1,2-*a*]pyridine **1a** (0.8 mmol) in DMSO (5 mL) were added 2-aminopyridine **2a** (1 mmol), *t*-BuOOH (70% aqueous solution) (2.4 mmol) and TEMPO/BHT/DPE (5 equiv.) at room temperature and the reaction mixture was stirred at 120 °C for 12 h. The reaction mixture was allowed to attain room temperature and poured into 20 mL of water and extracted with EtOAc (3 × 15 mL). The combined organic layer was dried over Na_2SO_4 and evaporated under vacuum. The resulting crude solid was purified over silica gel (60-120 mesh) column chromatography by using EtOAc-*n*-hexane (4.0:6.0, *v/v*) to afford **3aa** in 0, 41% and 20% yields in case of TEMPO, BHT and DPE, respectively. The same reaction mixtures were directly analysed by ESI-HRMS analysis to observe the corresponding intermediates and adducts of radical scavengers such as TEMPO, BHT and 1,1-DPE.

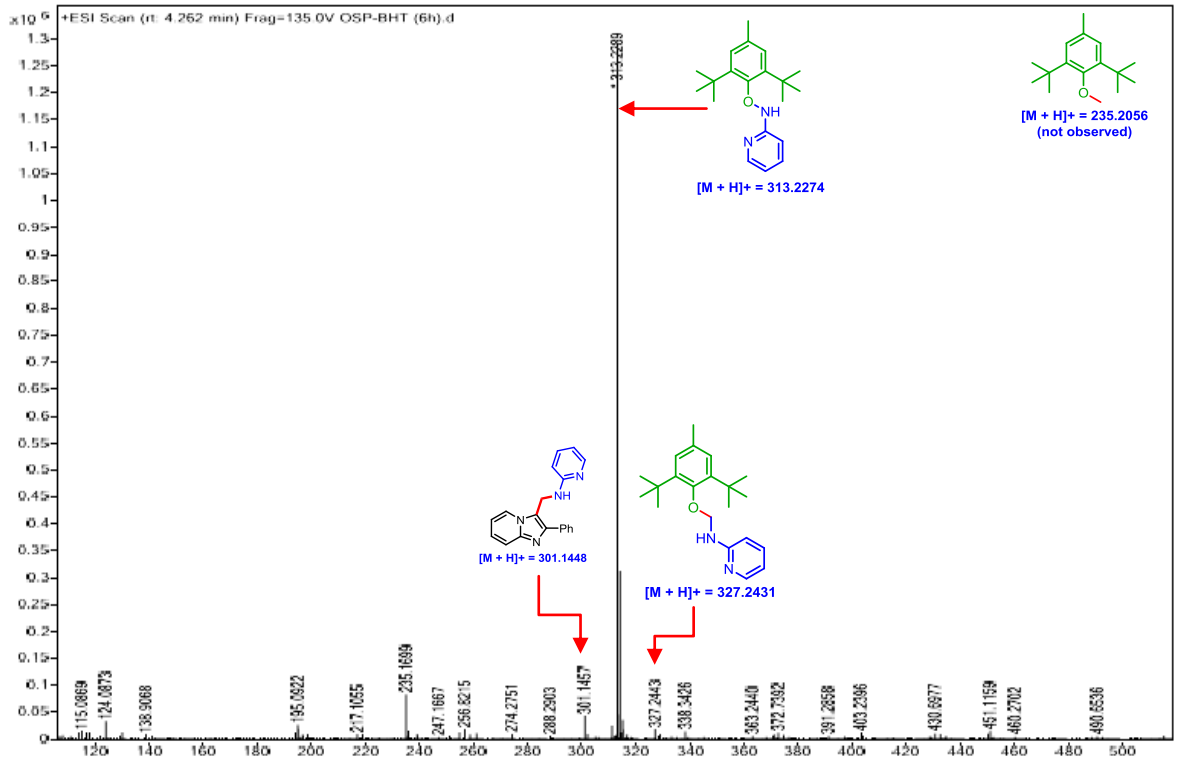
ESI-HRMS of crude reaction mixture of TEMPO adduct:

Sample Name	OSP-TEMPO (6h)	**Position**	P2-C4	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-TEMPO (6h).d	**ACQ Method**	water_acn_grad_6min.	**Comment**		**Acquired Time**	3/16/2018 6:35:10 PM

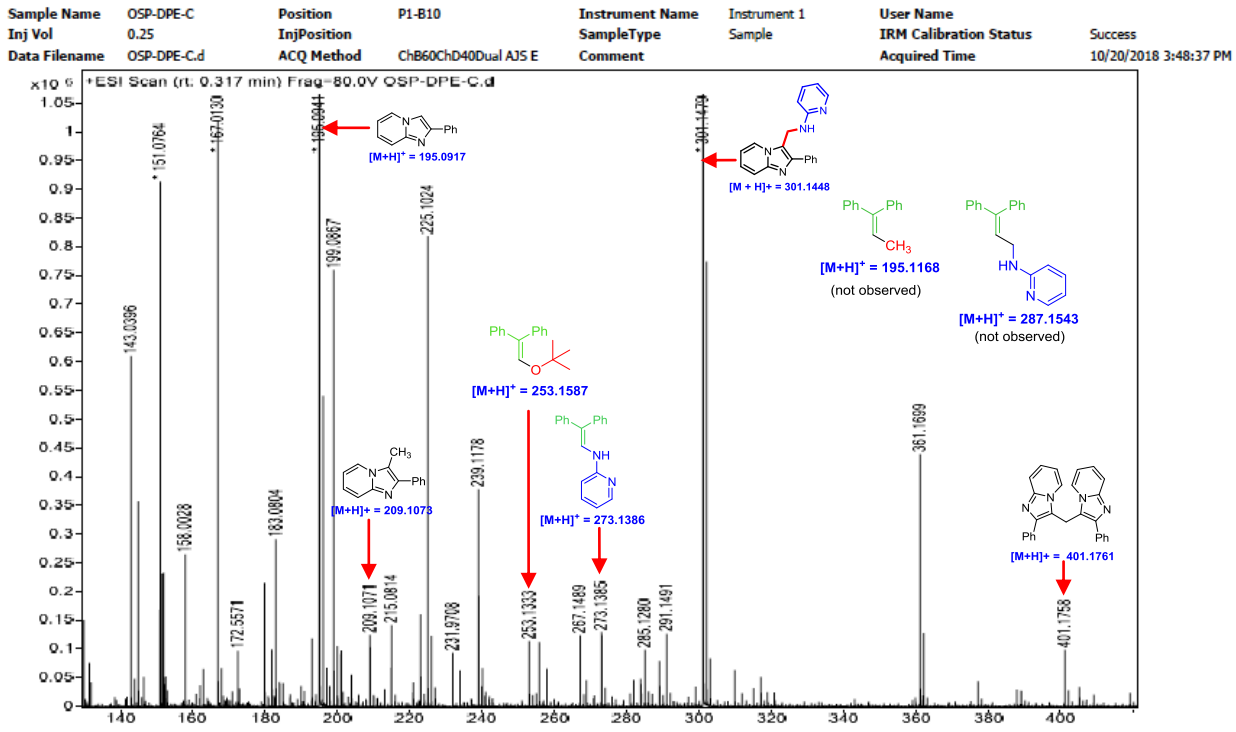


ESI-HRMS of crude reaction mixture of BHT adduct:

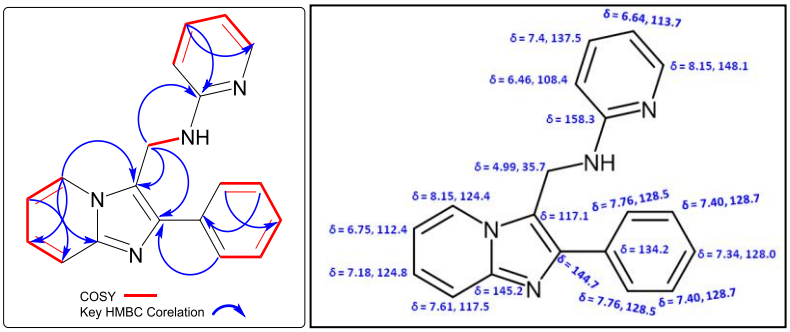
Sample Name	OSP-BHT (6h)	**Position**	P2-C6	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-BHT (6h).d	**ACQ Method**	water_acn_grad_6min.	**Comment**		**Acquired Time**	3/16/2018 6:49:19 PM



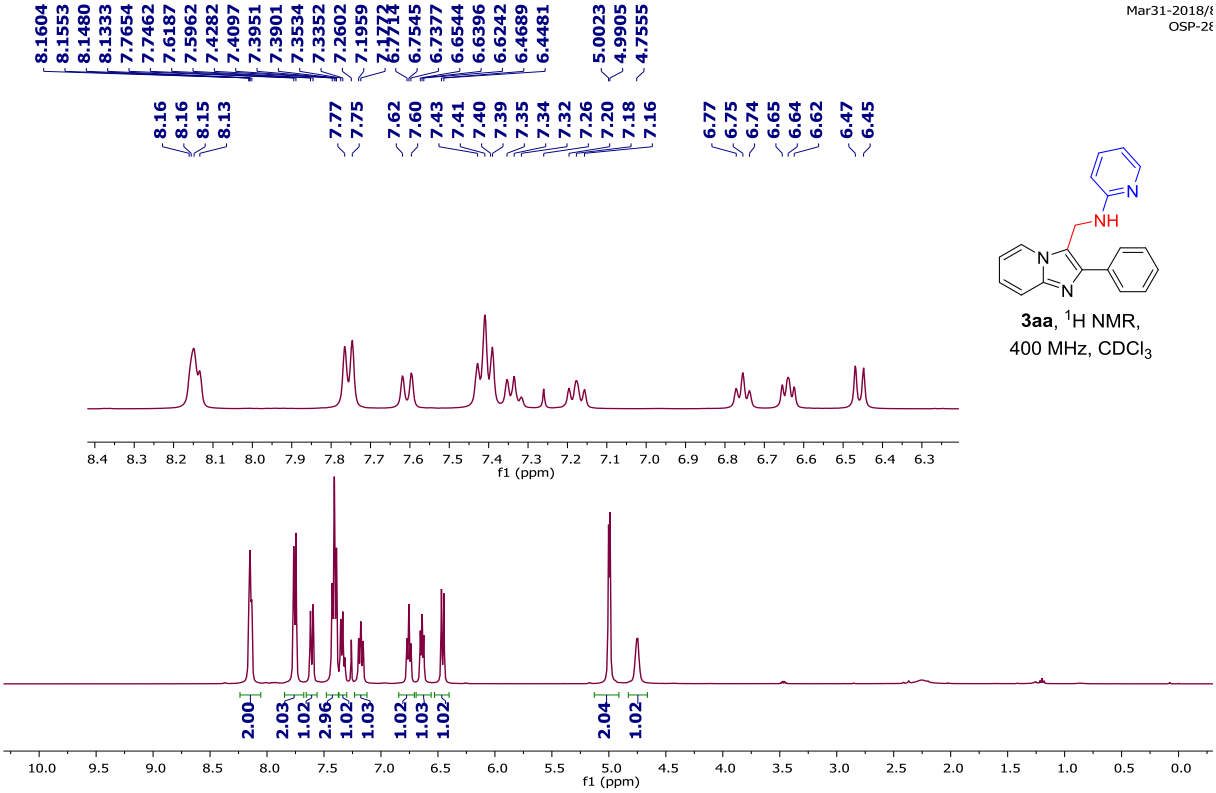
ESI-HRMS of crude reaction mixture of 1,1-DPE adduct:



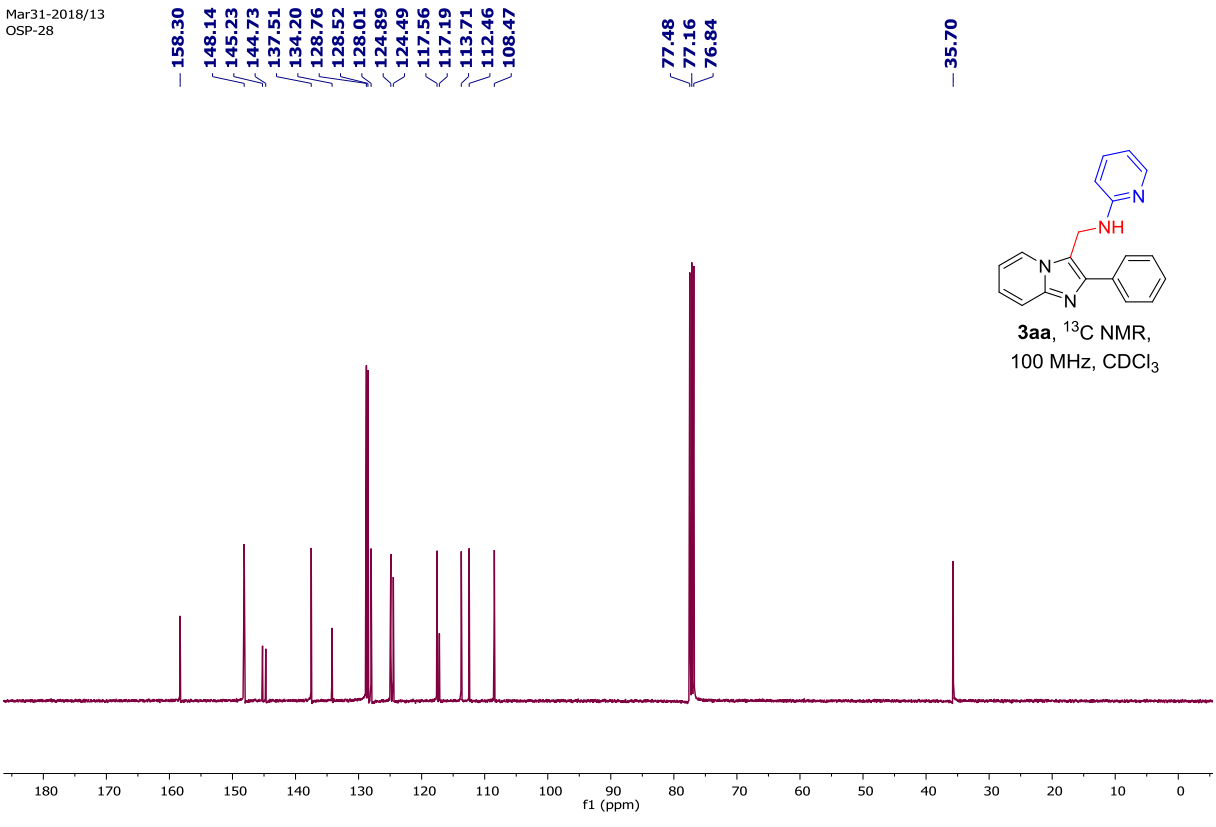
2. Unambiguous molecular structure determination of 3aa *via* 1D and 2D NMR spectroscopy



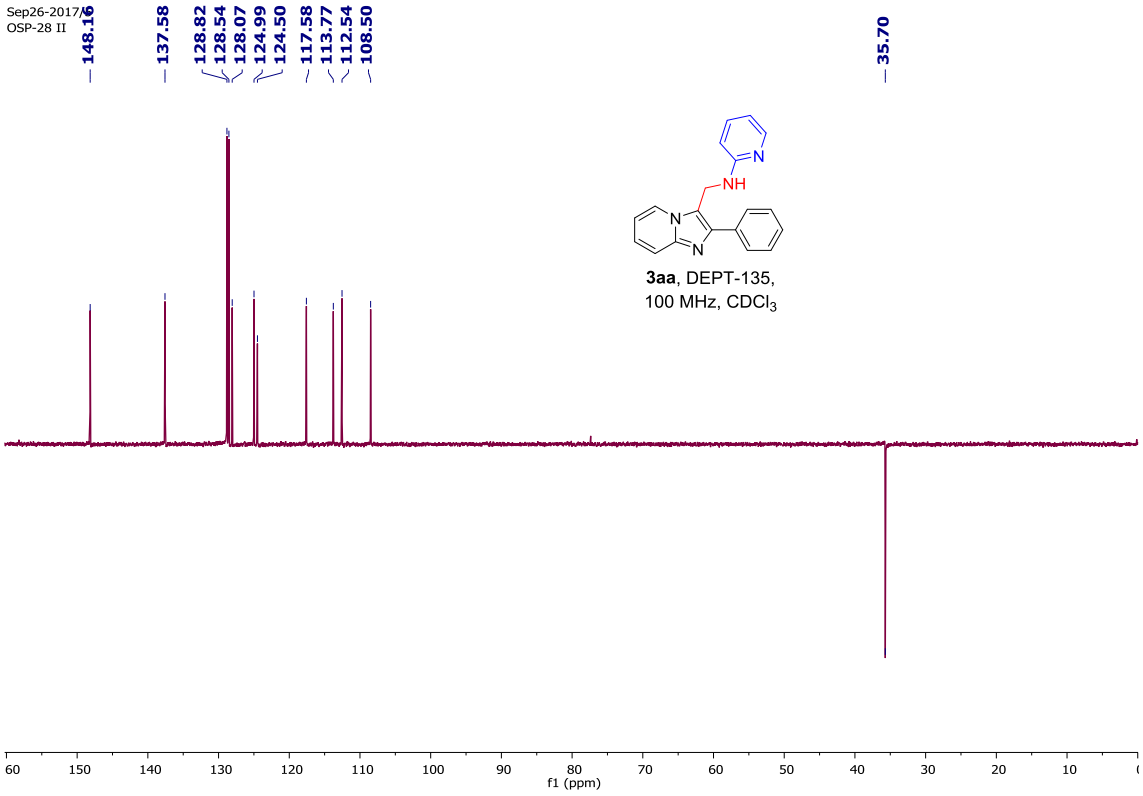
3. Copies of NMR and ESI-HRMS data

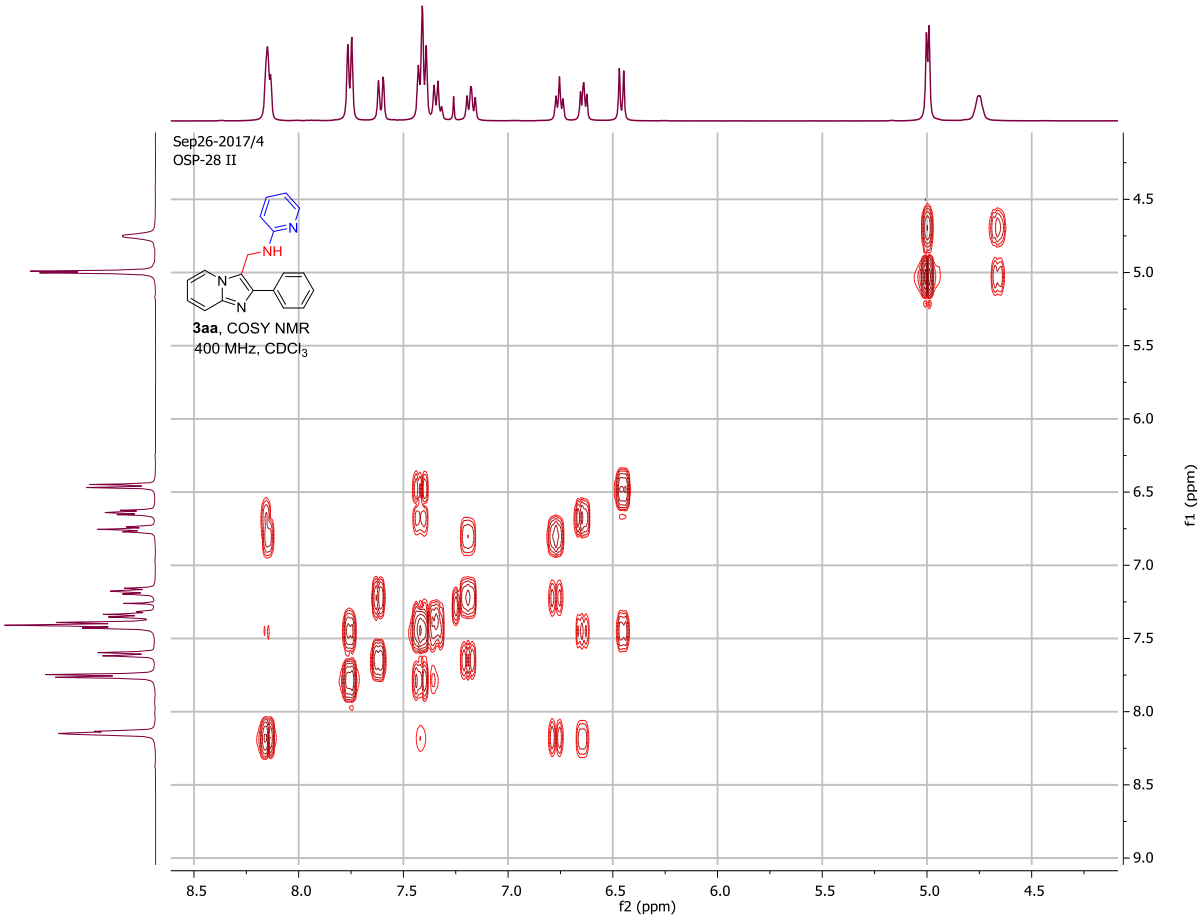


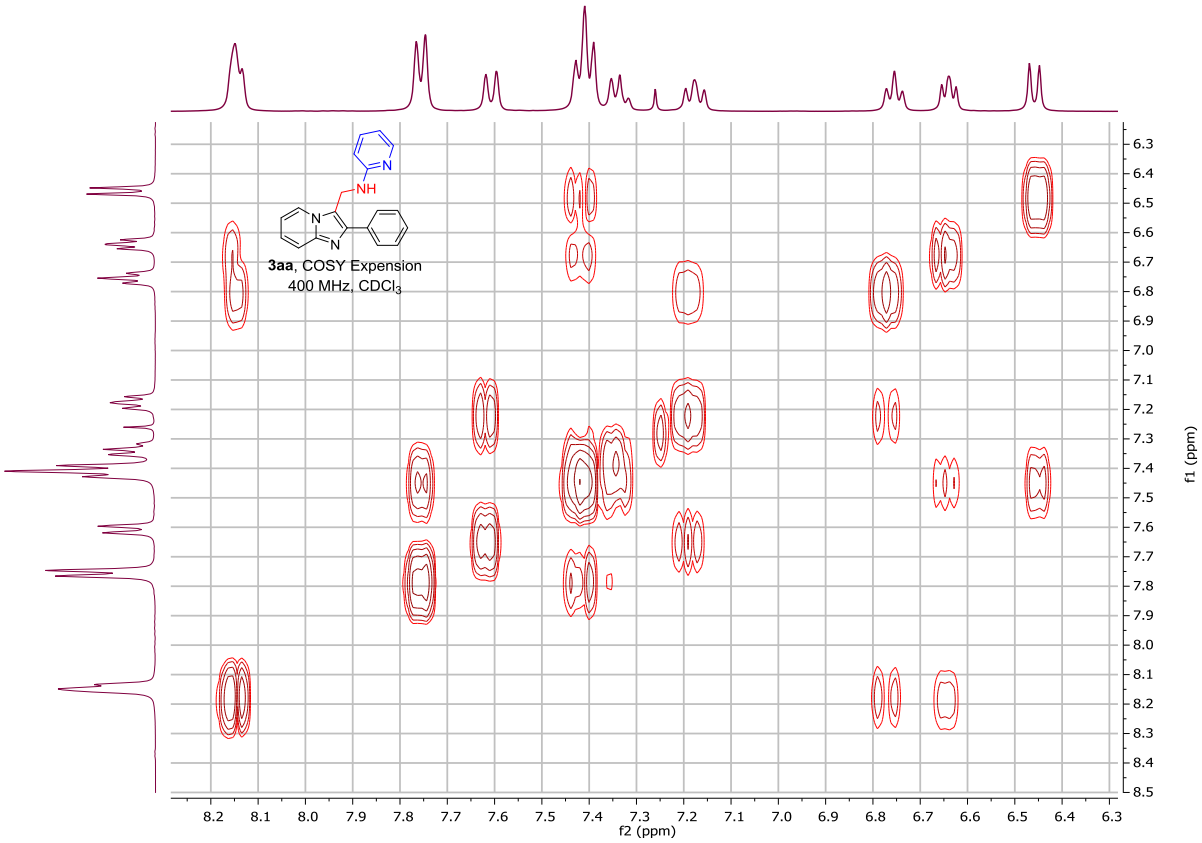
3aa, ^{1}H NMR,
400 MHz, $CDCl_3$

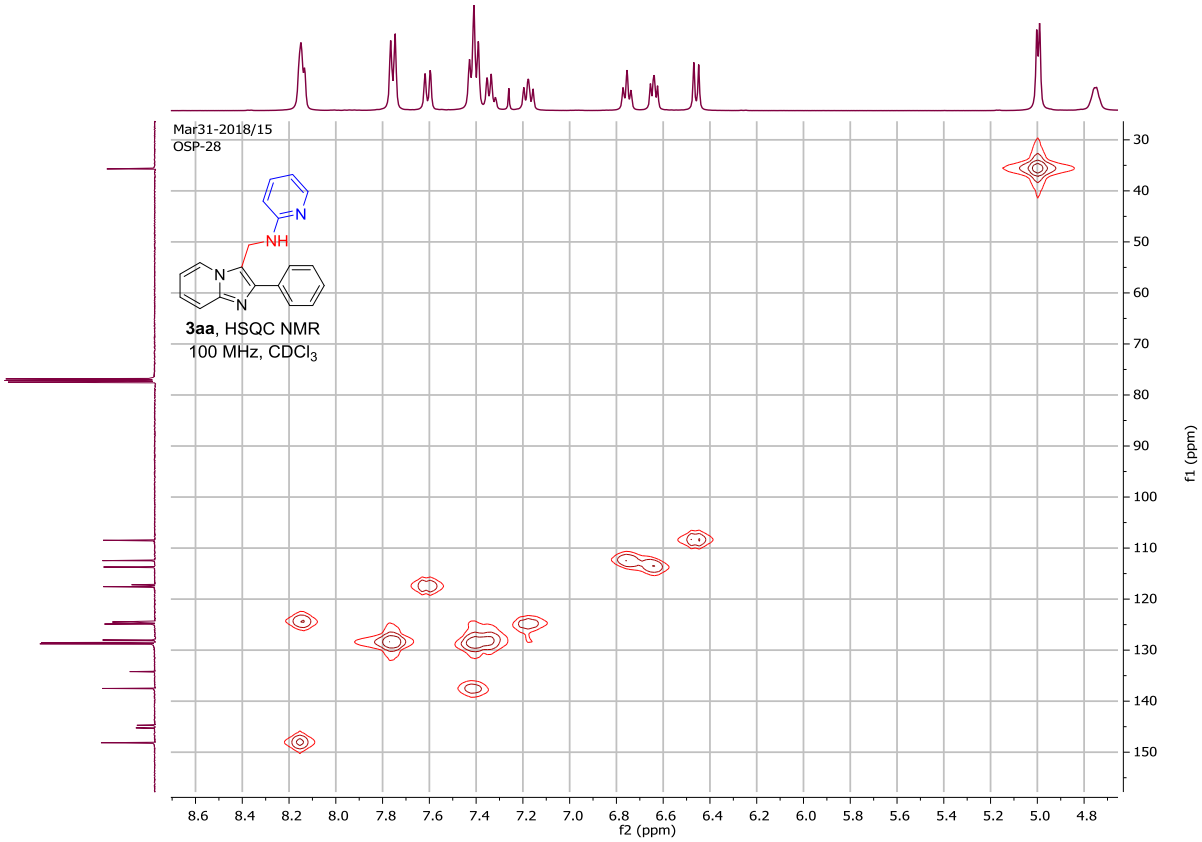


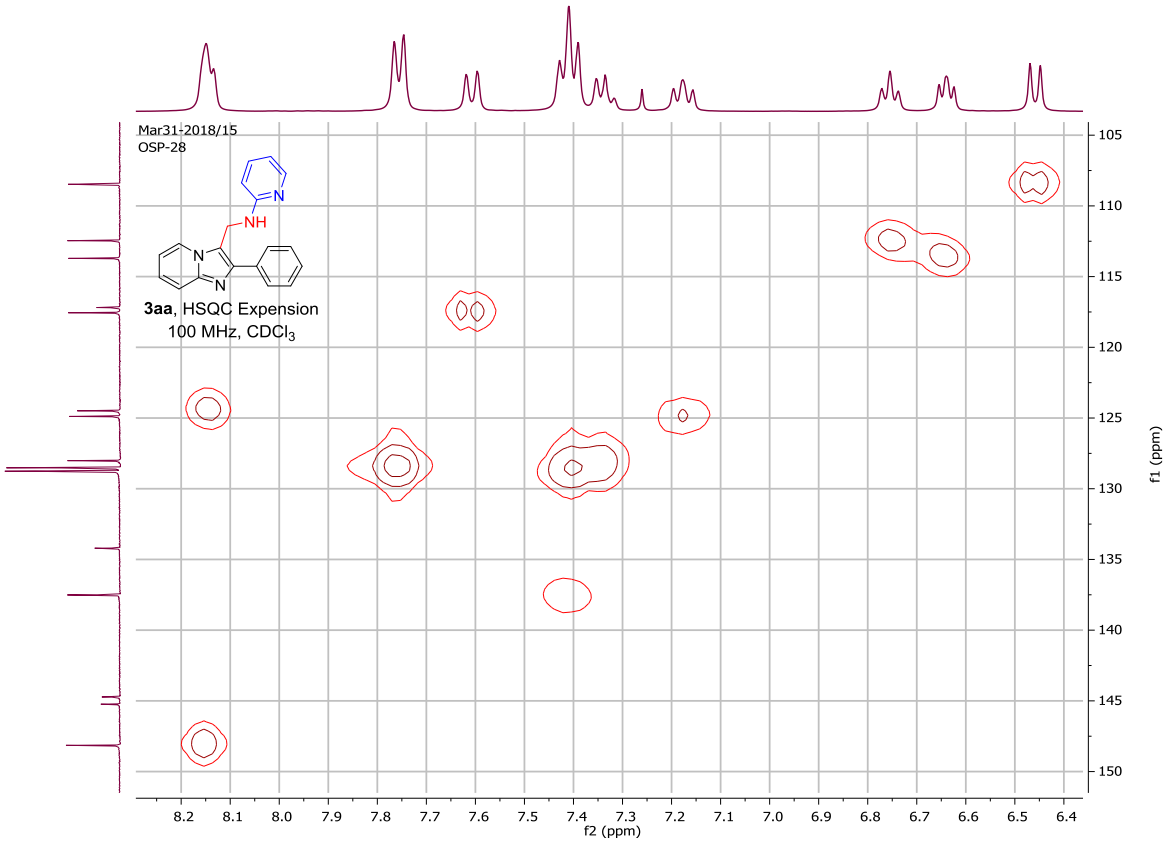
3aa, ^{13}C NMR,
100 MHz, $CDCl_3$

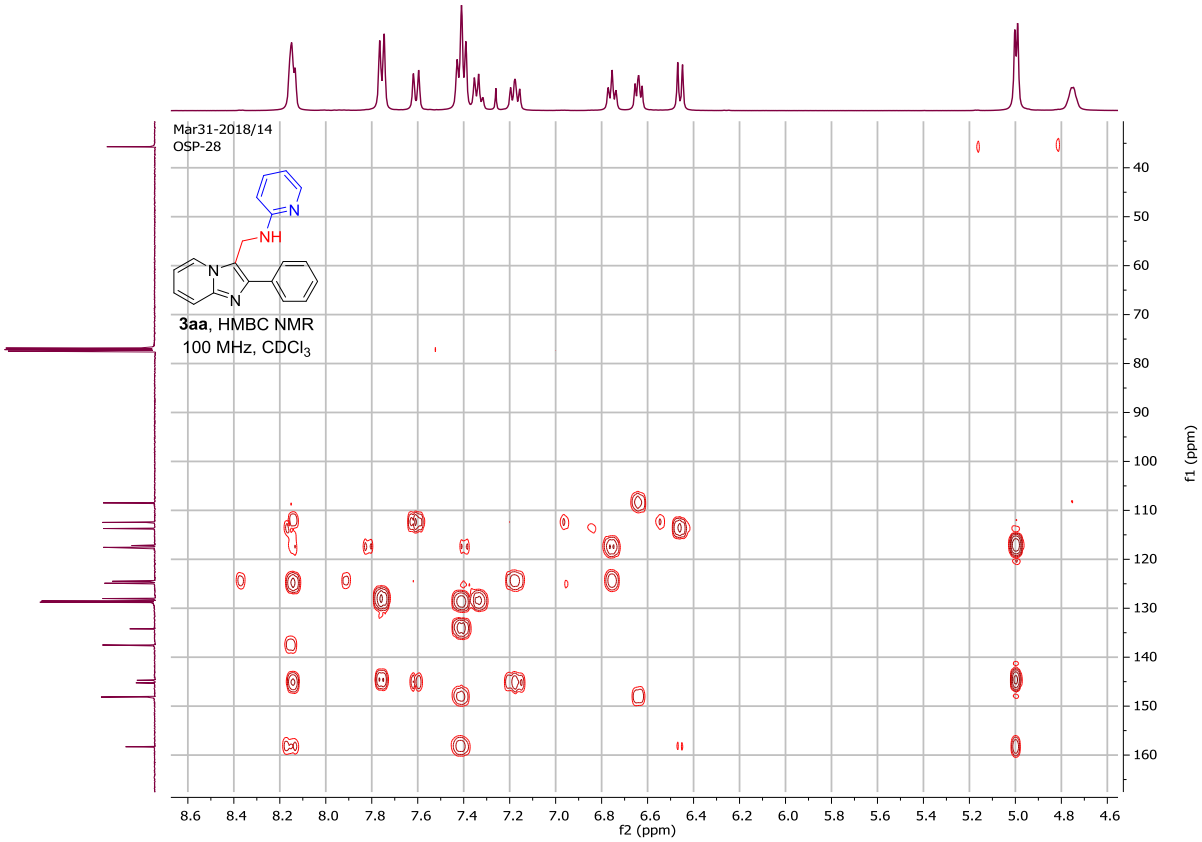

Sep26-2017/6
OSP-28 II
148.16
137.58
128.82
128.54
128.07
124.99
124.50
117.58
113.77
112.54
108.50
35.70
NH
3aa, DEPT-135,
100 MHz, CDCl3
f1 (ppm)

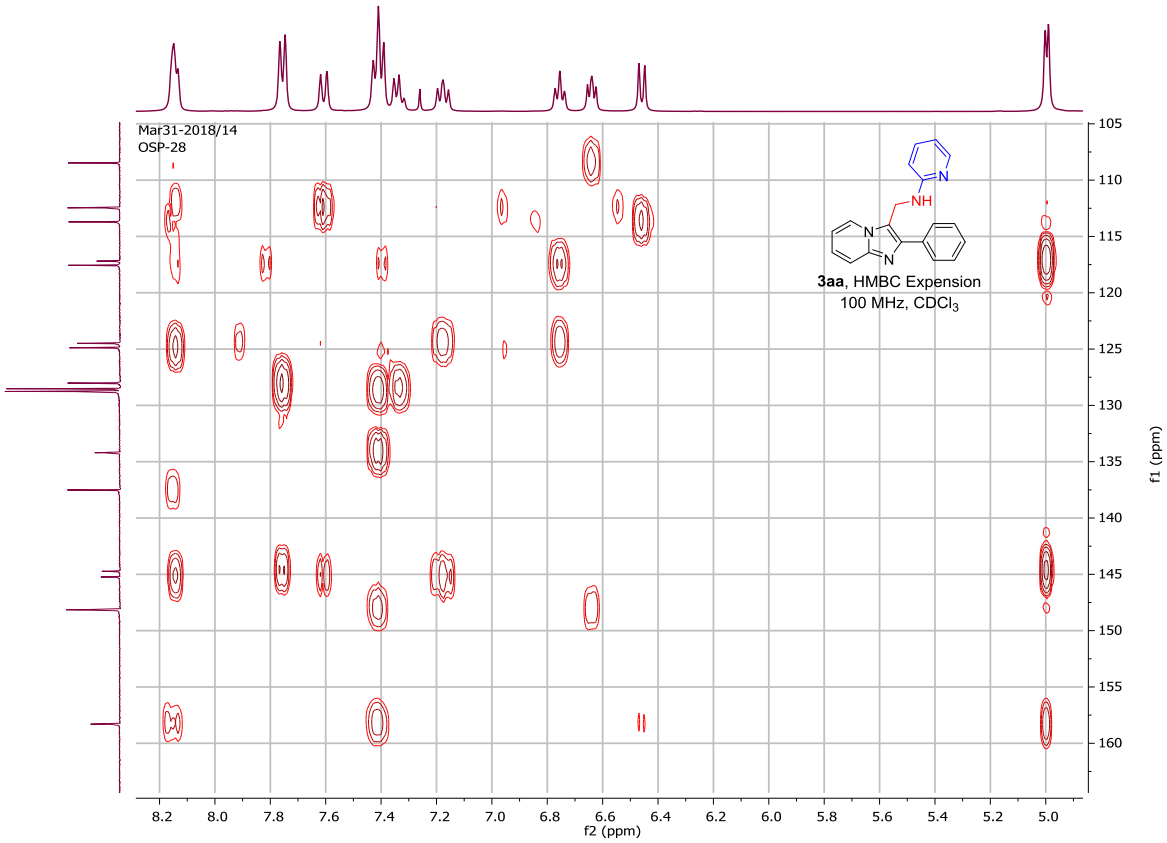

Sep26-2017/4
OSP-28 II
NH
3aa, COSY NMR
400 MHz, CDCl3
f2 (ppm)
f1 (ppm)


3aa, COSY Expension
400 MHz, CDCl3
f2 (ppm)
f1 (ppm)

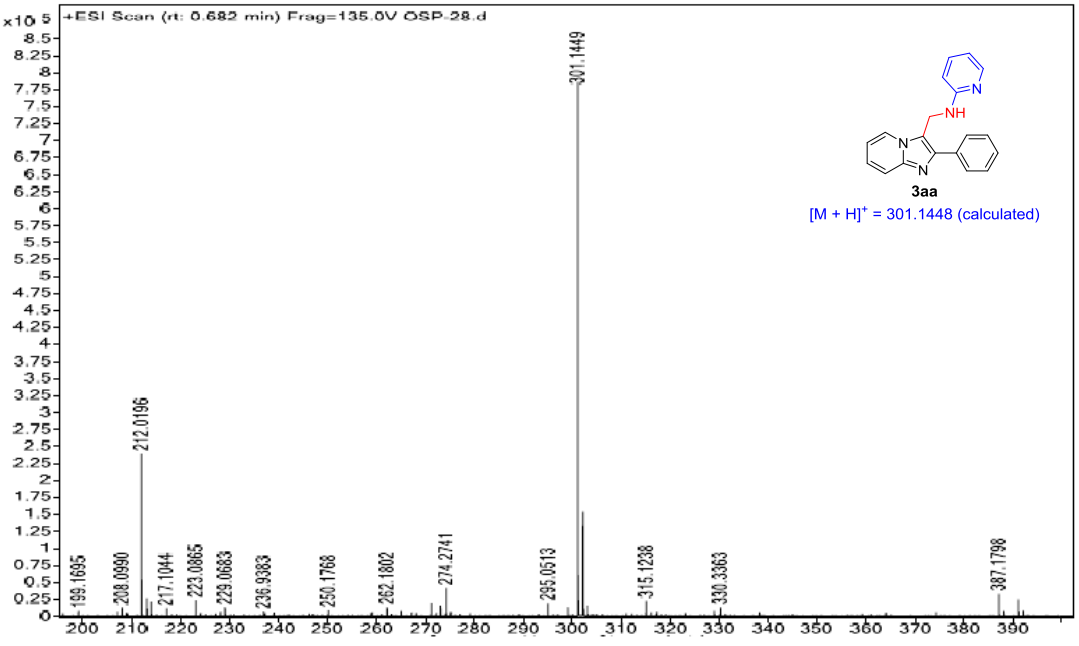

Mar31-2018/15
OSP-28
3aa, HSQC NMR
100 MHz, CDCl3
f2 (ppm)
f1 (ppm)

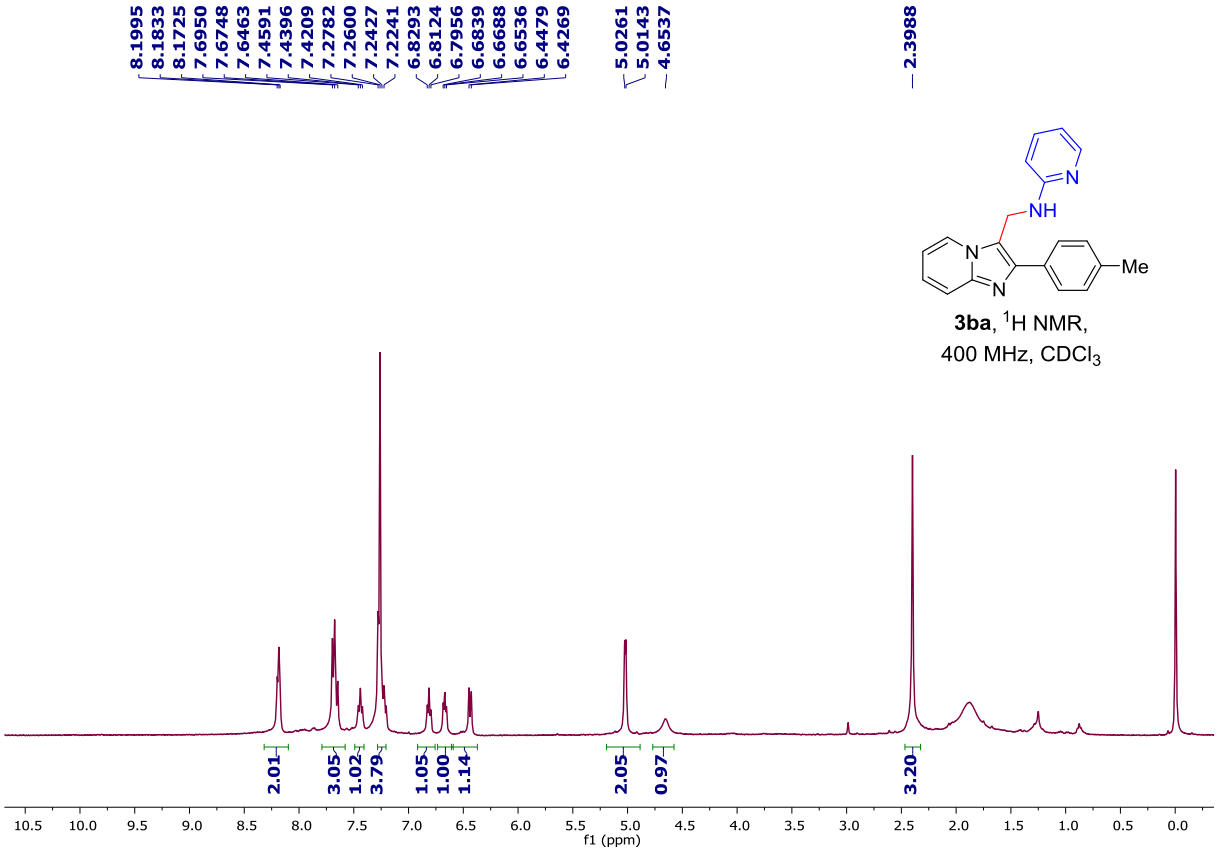

Mar31-2018/15
OSP-28
3aa, HSQC Expension
100 MHz, CDCl3
f2 (ppm)
f1 (ppm)


Mar31-2018/14
OSP-28
3aa, HMBC NMR
100 MHz, CDCl3
f2 (ppm)
f1 (ppm)

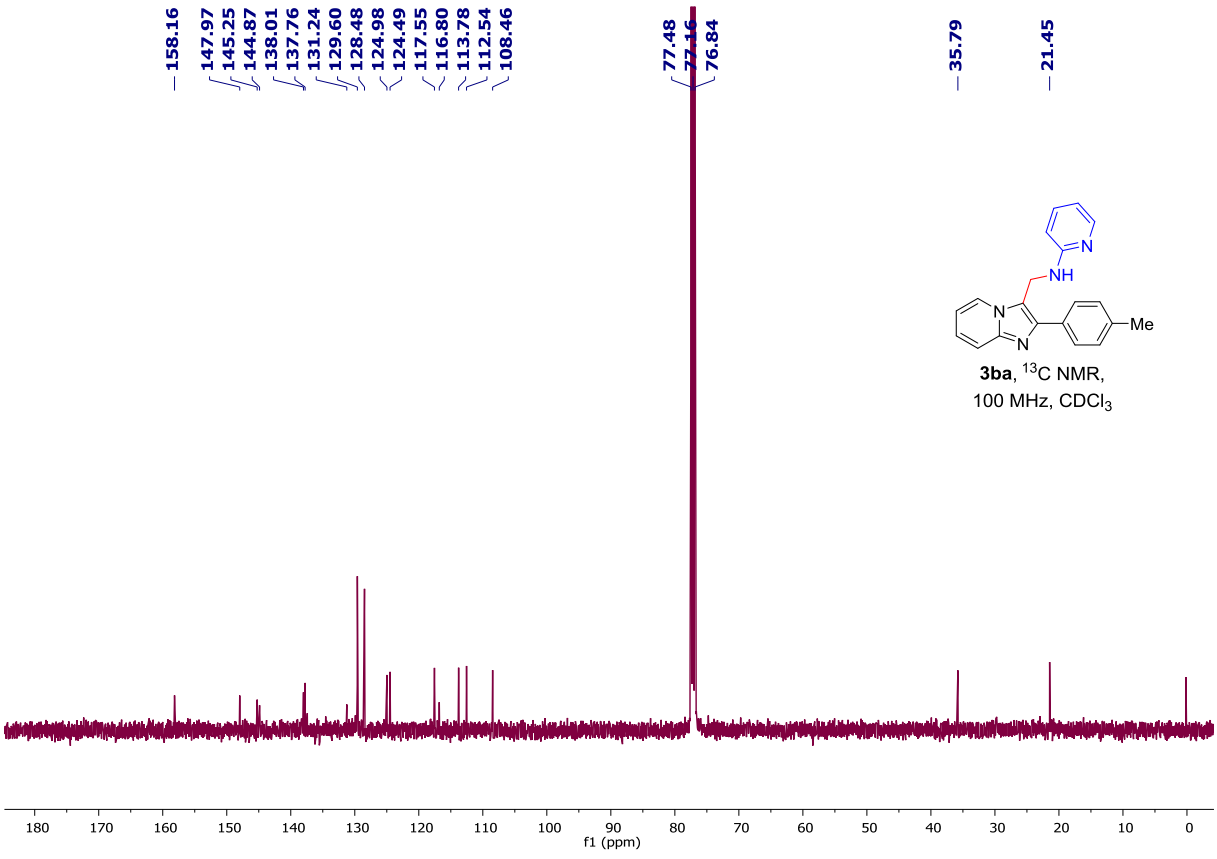


Sample Name	OSP-28	**Position**	P1-D4	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-28.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	9/22/2017 10:35:47 PM



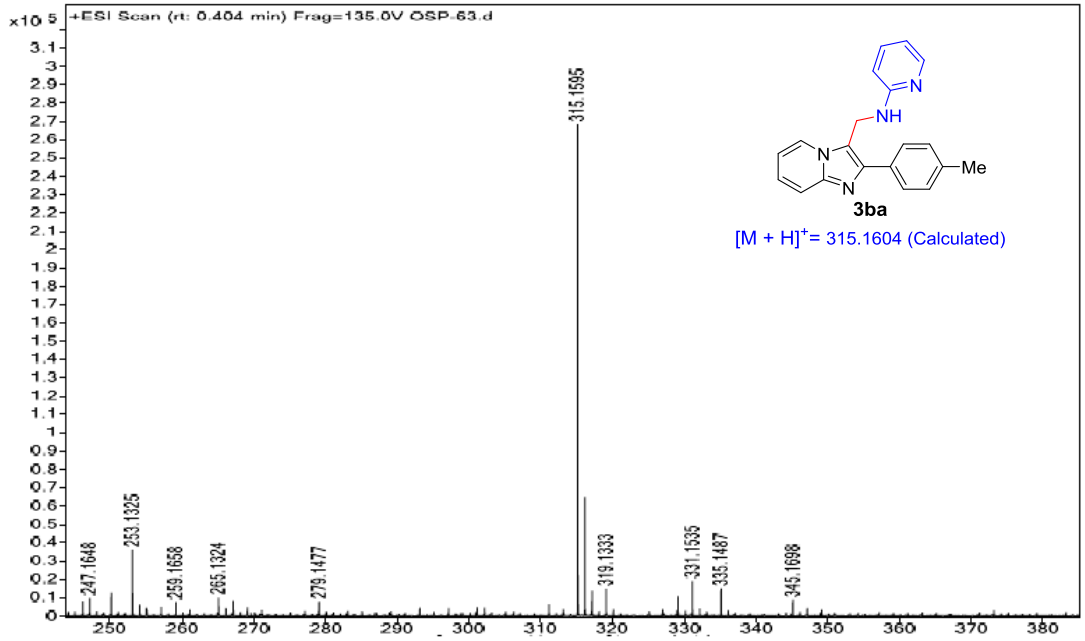


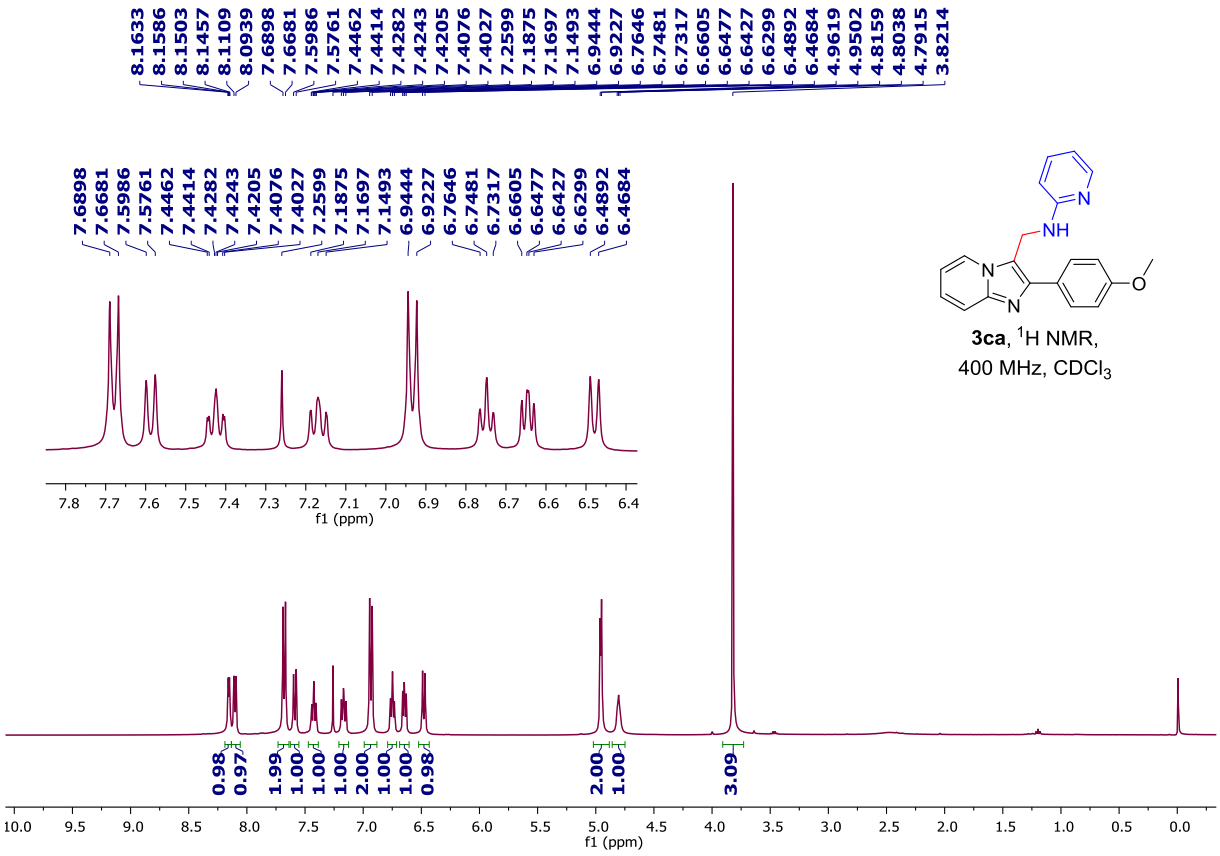
3ba, ^{1}H NMR,
400 MHz, $CDCl_3$

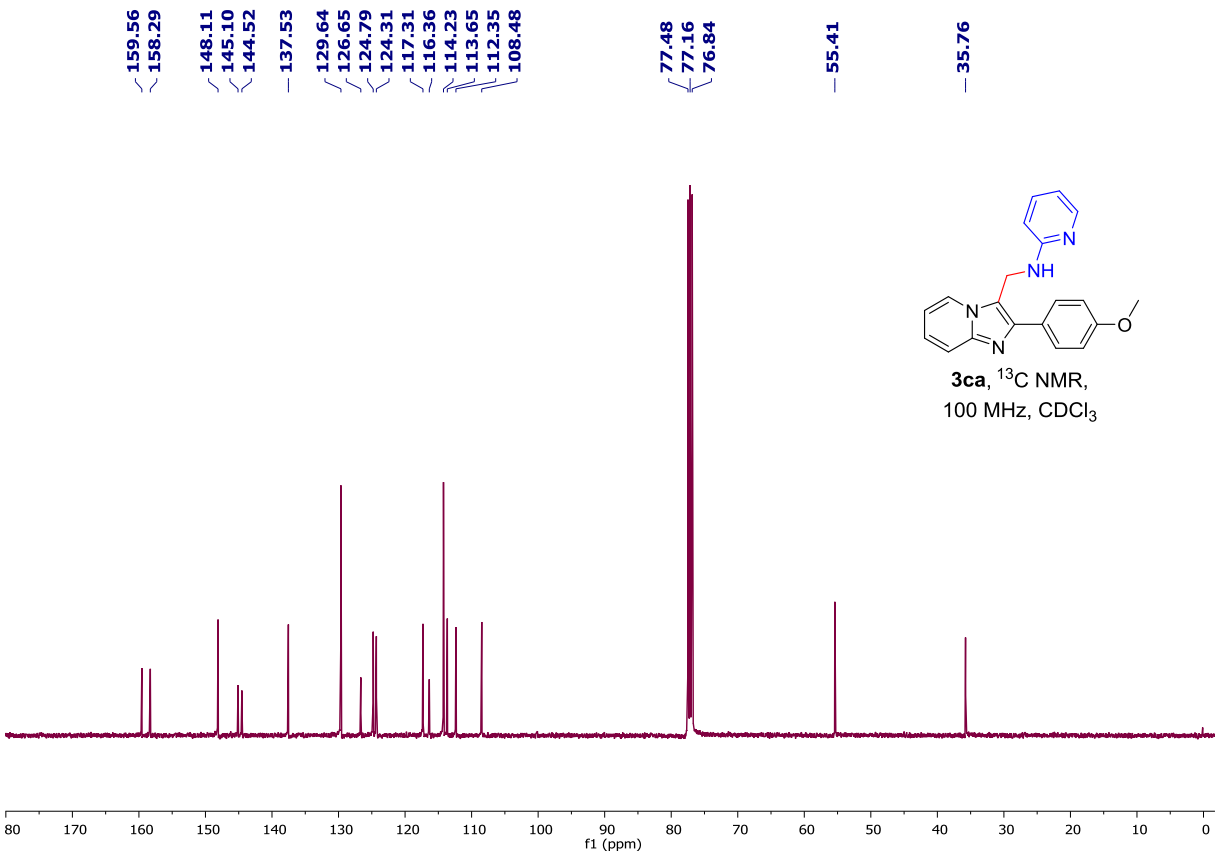


3ba, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-63	**Position**	P2-E7	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-63.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	12/8/2017 1:40:21 PM

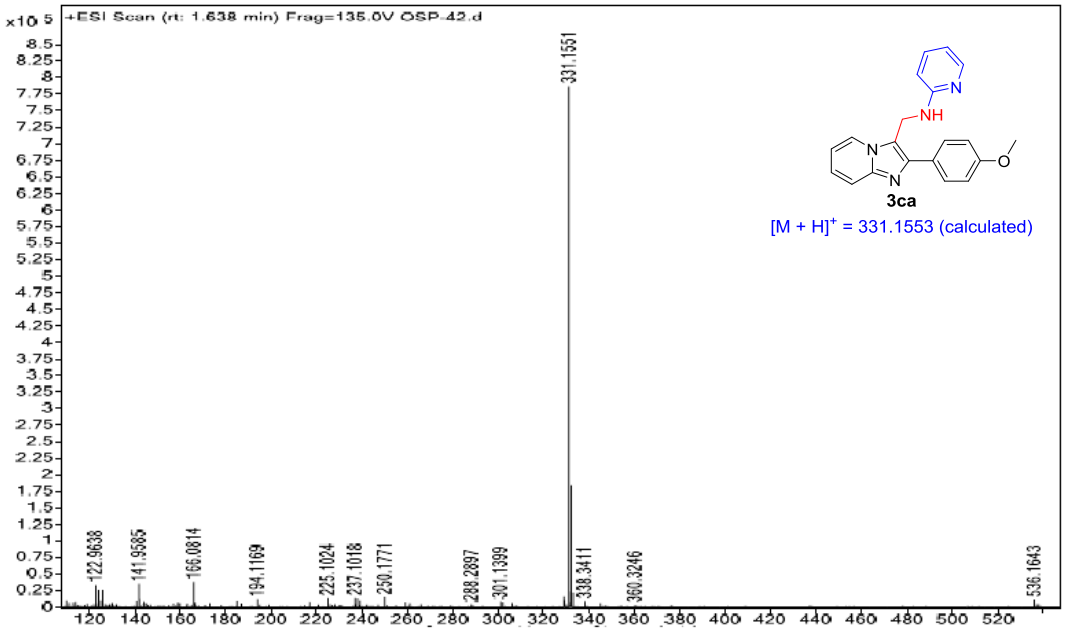




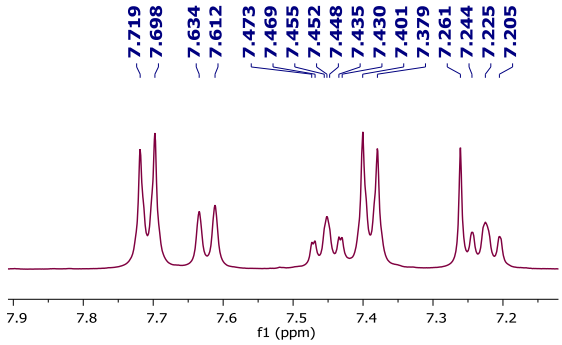


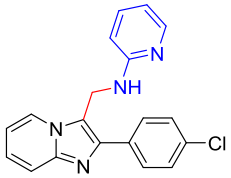
3ca, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-42	**Position**	P2-F3	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-42.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	10/12/2017 5:30:15 PM

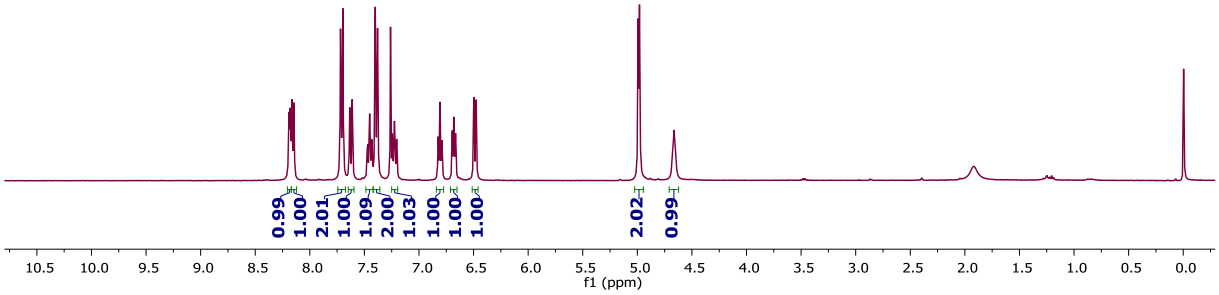


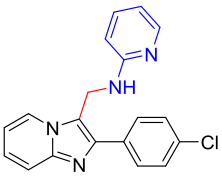
$[M + H]^+$ = 331.1553 (calculated)



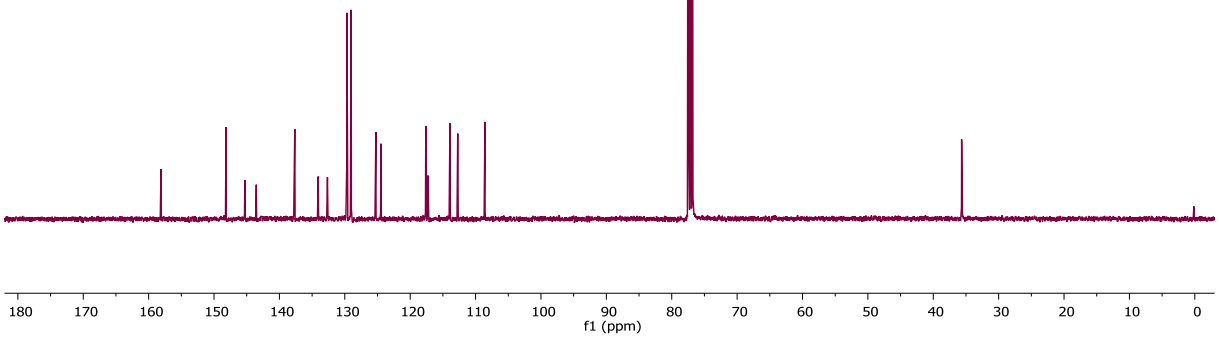


3da, ^{1}H NMR,
400 MHz, $CDCl_3$

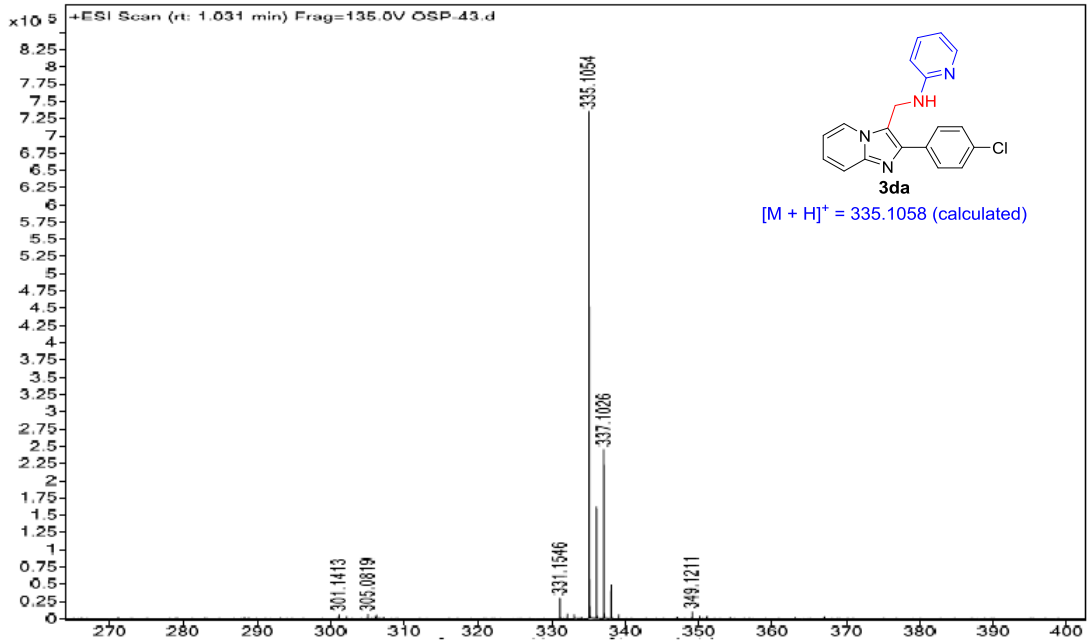


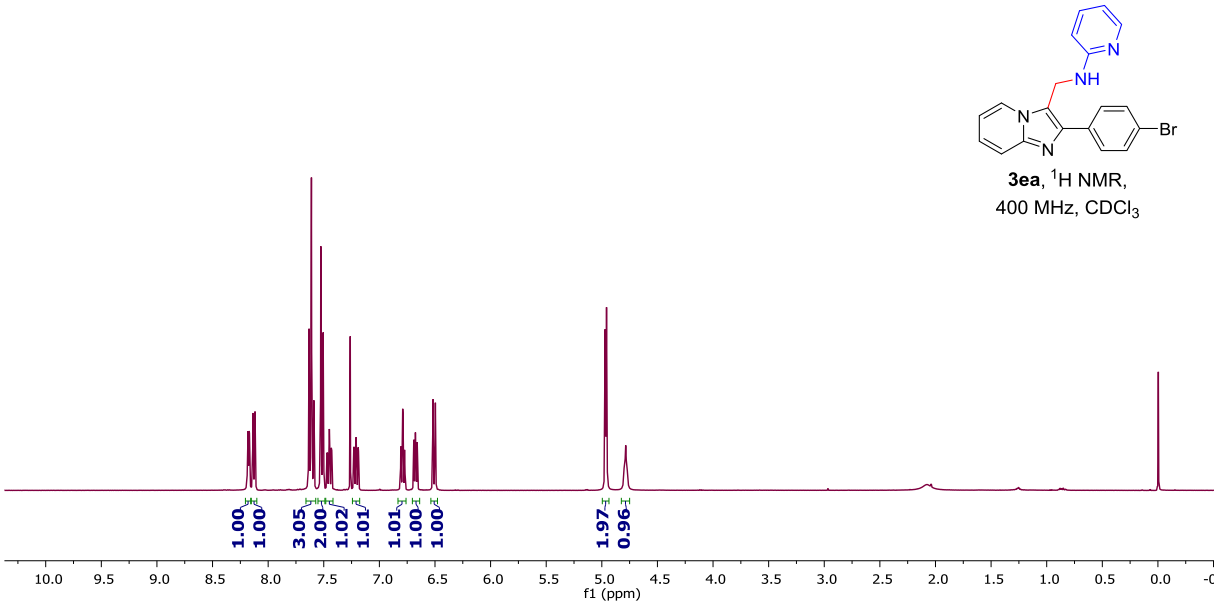


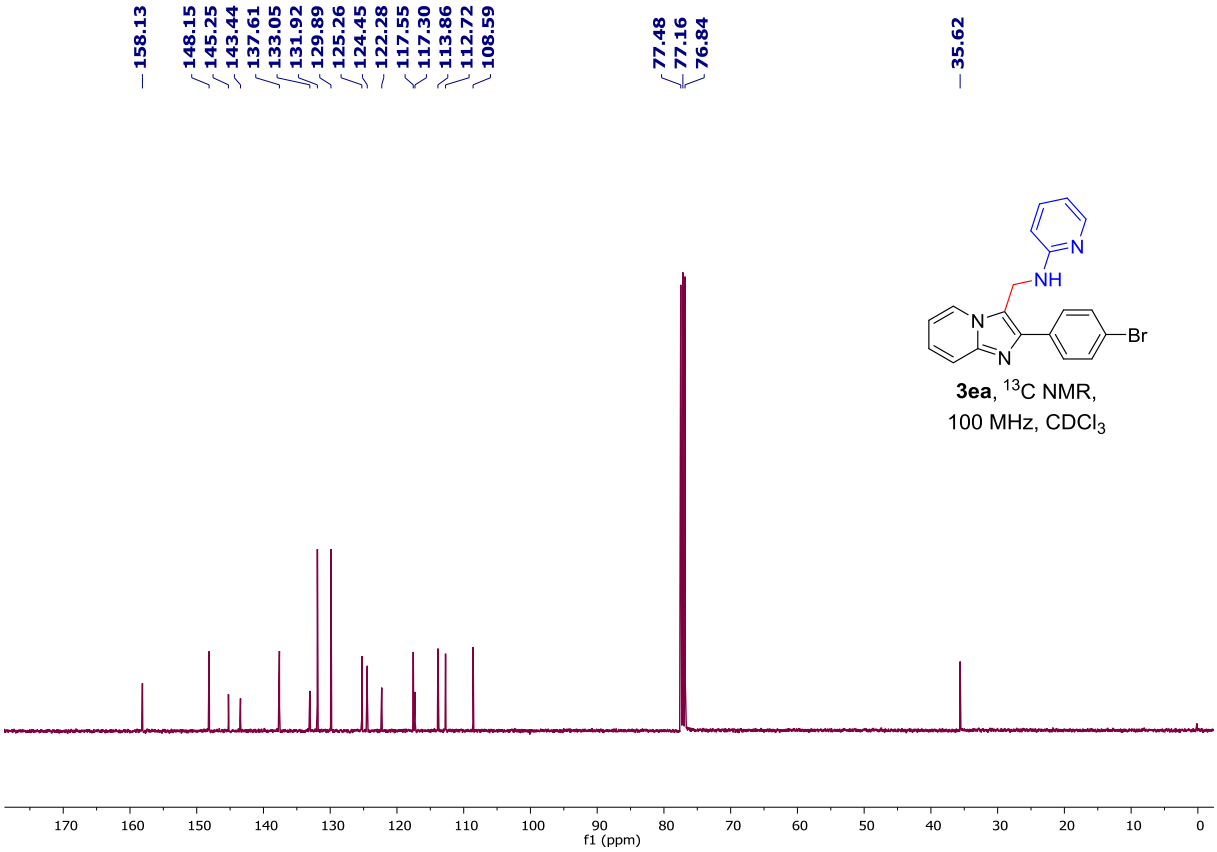
3da, ^{13}C NMR,
100 MHz, $CDCl_3$



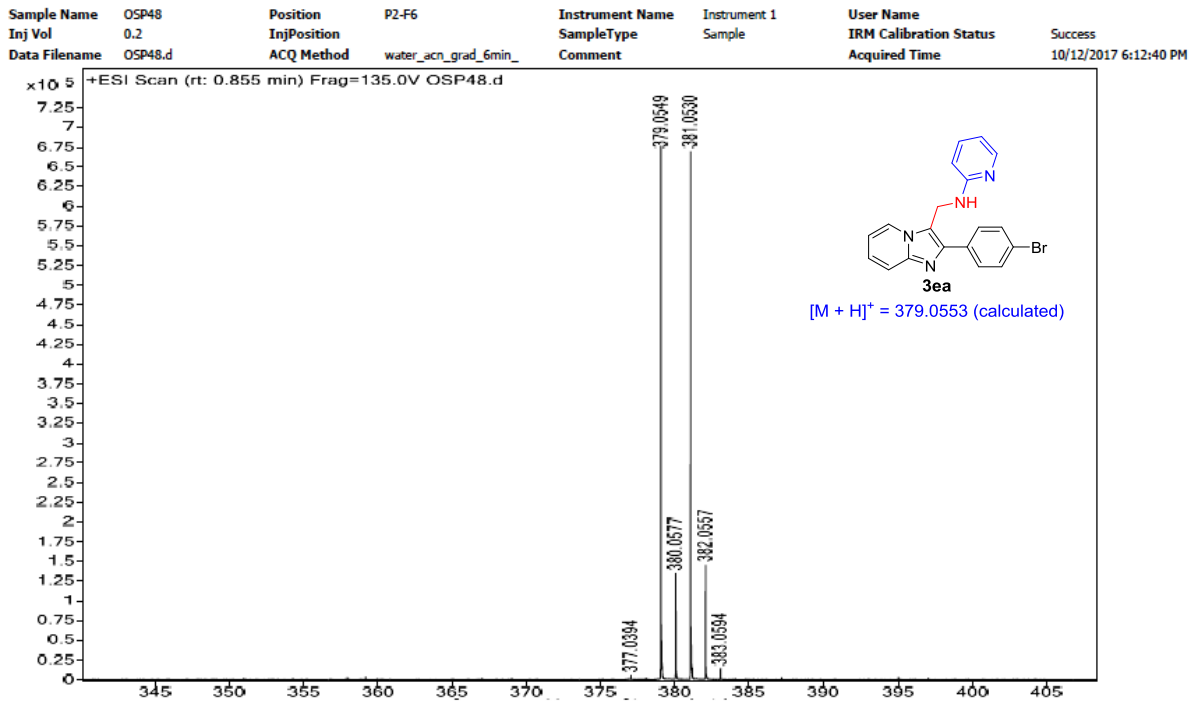
Sample Name	OSP-43	**Position**	P2-F4	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-43.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	10/12/2017 5:44:24 PM



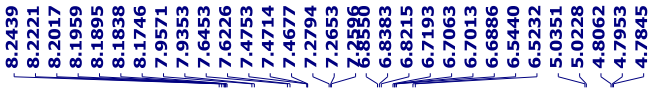


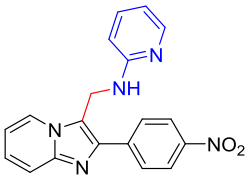


3ea, ^{13}C NMR,
100 MHz, $CDCl_3$

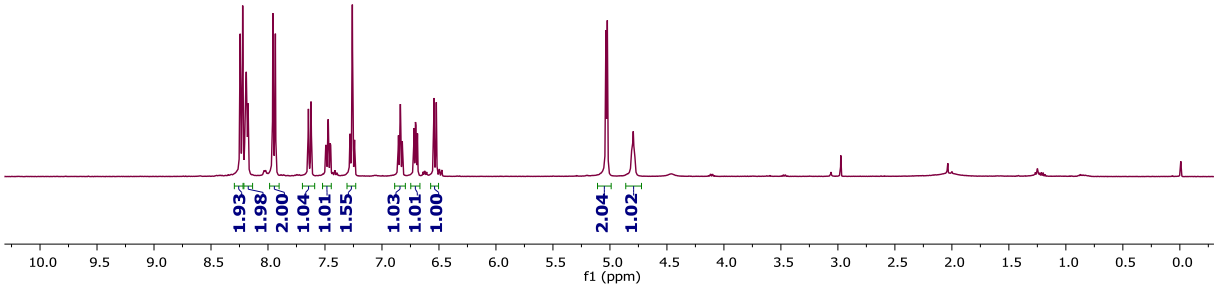


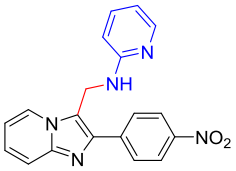
$[M + H]^+$ = 379.0553 (calculated)



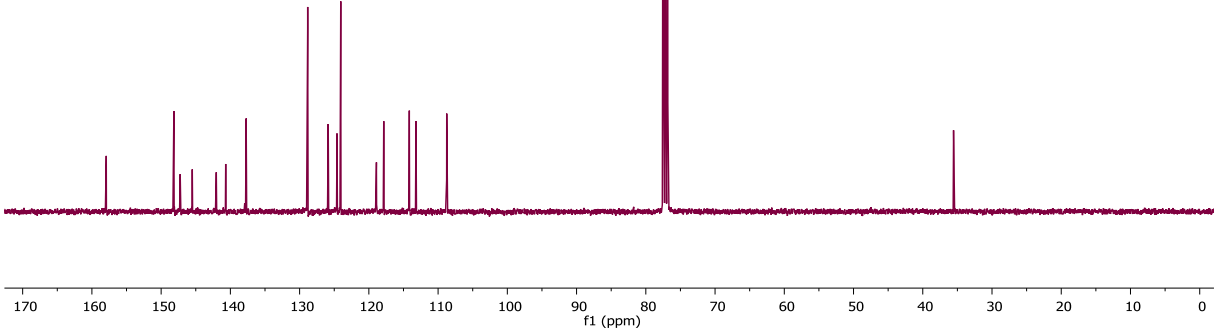


3fa, ^{1}H NMR,
400 MHz, $CDCl_3$

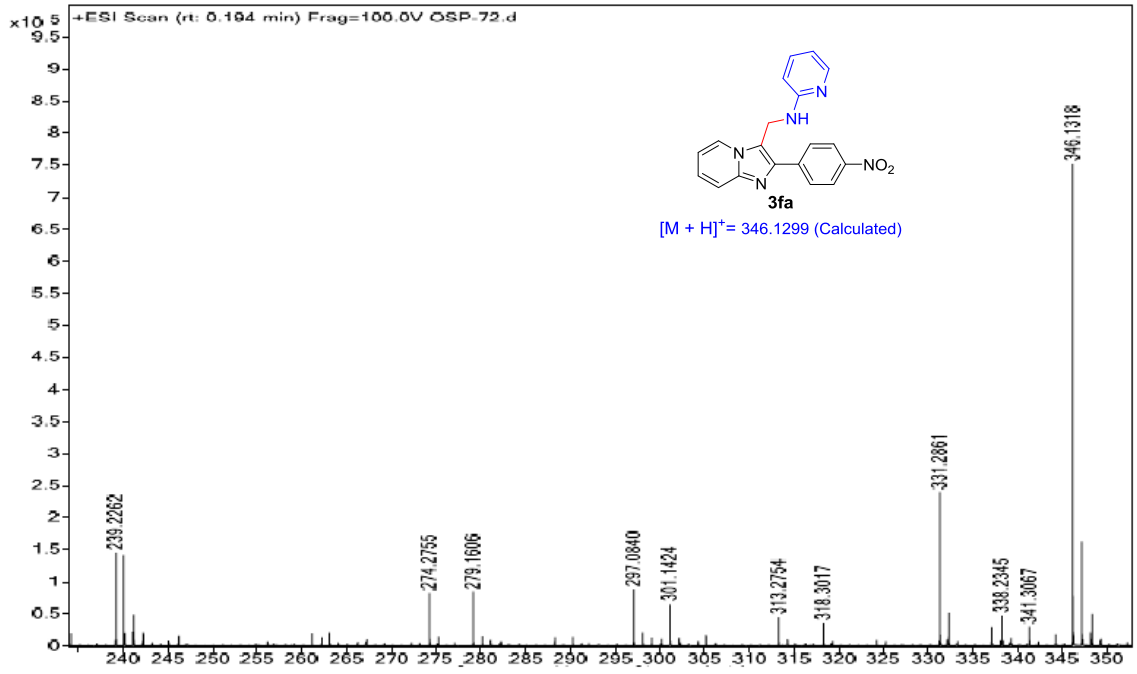


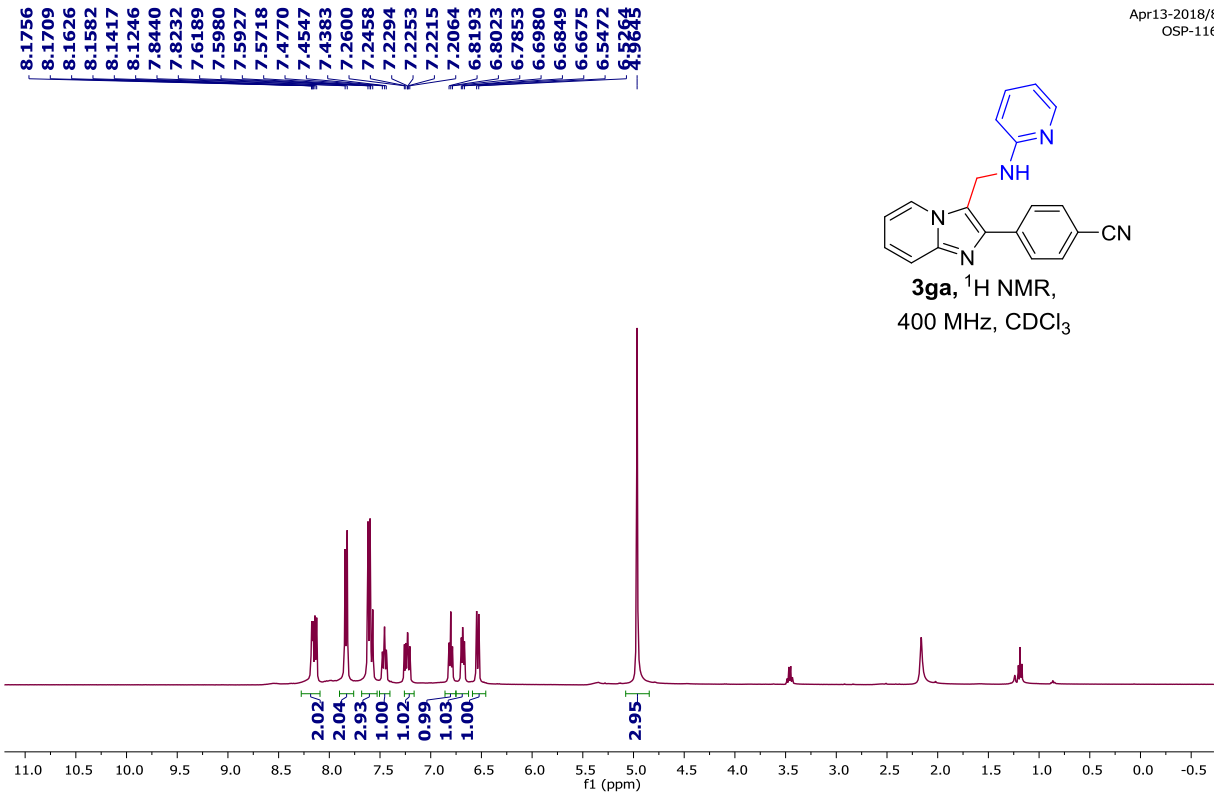


3fa, ^{13}C NMR,
100 MHz, $CDCl_3$



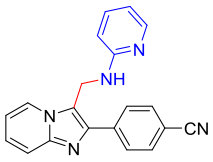
Sample Name	OSP-72	**Position**	P2-C8	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-72.d	**ACQ Method**	Methanol_isocratic.m	**Comment**		**Acquired Time**	2/2/2018 6:06:49 PM



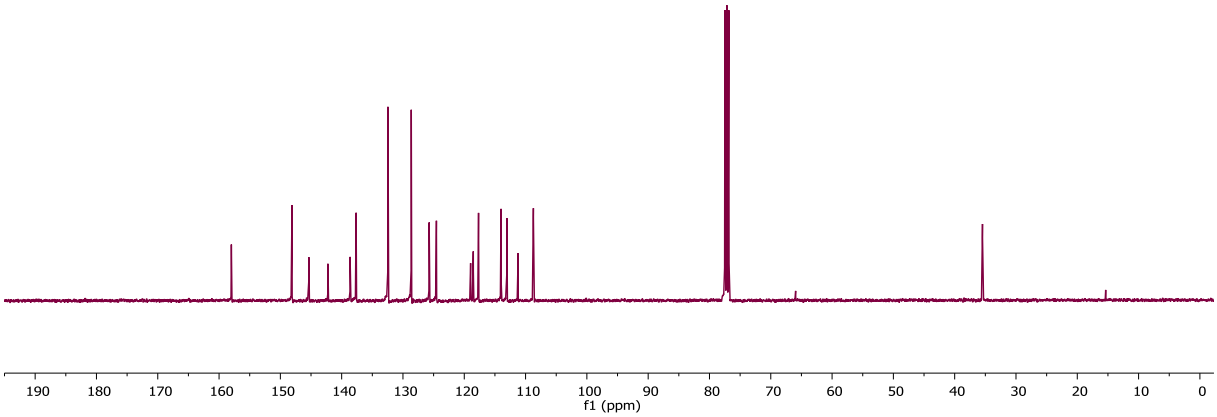


Apr13-2018/6
OSP-116

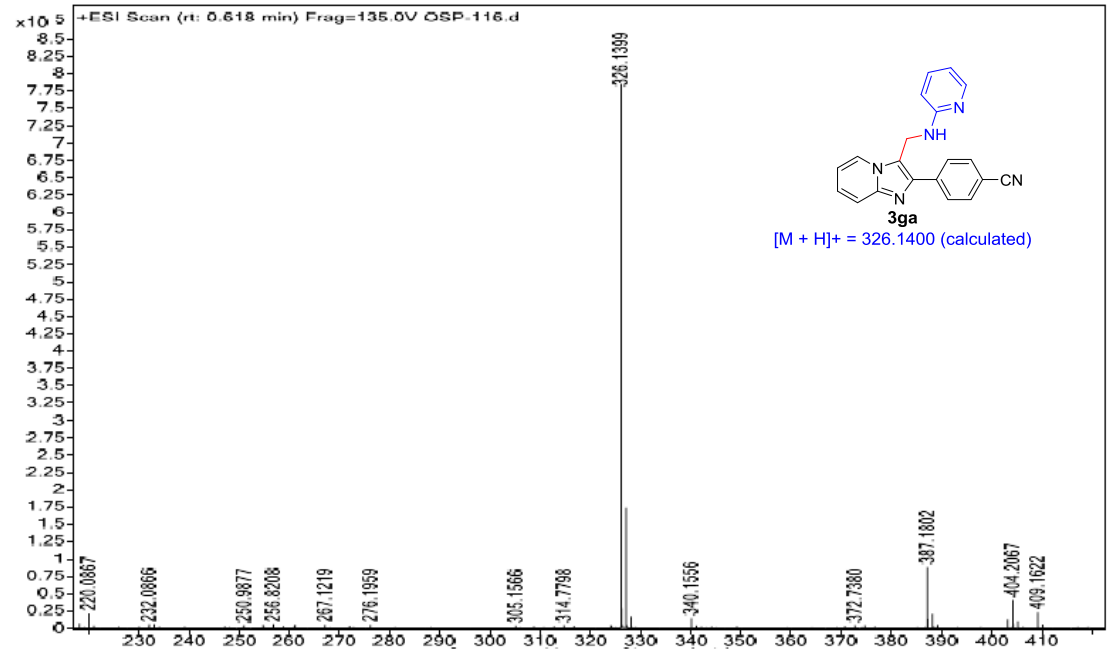
157.99
148.10
145.35
142.25
138.62
137.64
132.45
128.64
125.72
124.54
118.97
118.52
117.67
114.00
113.06
111.26
108.72
77.48
77.16
76.84
35.46

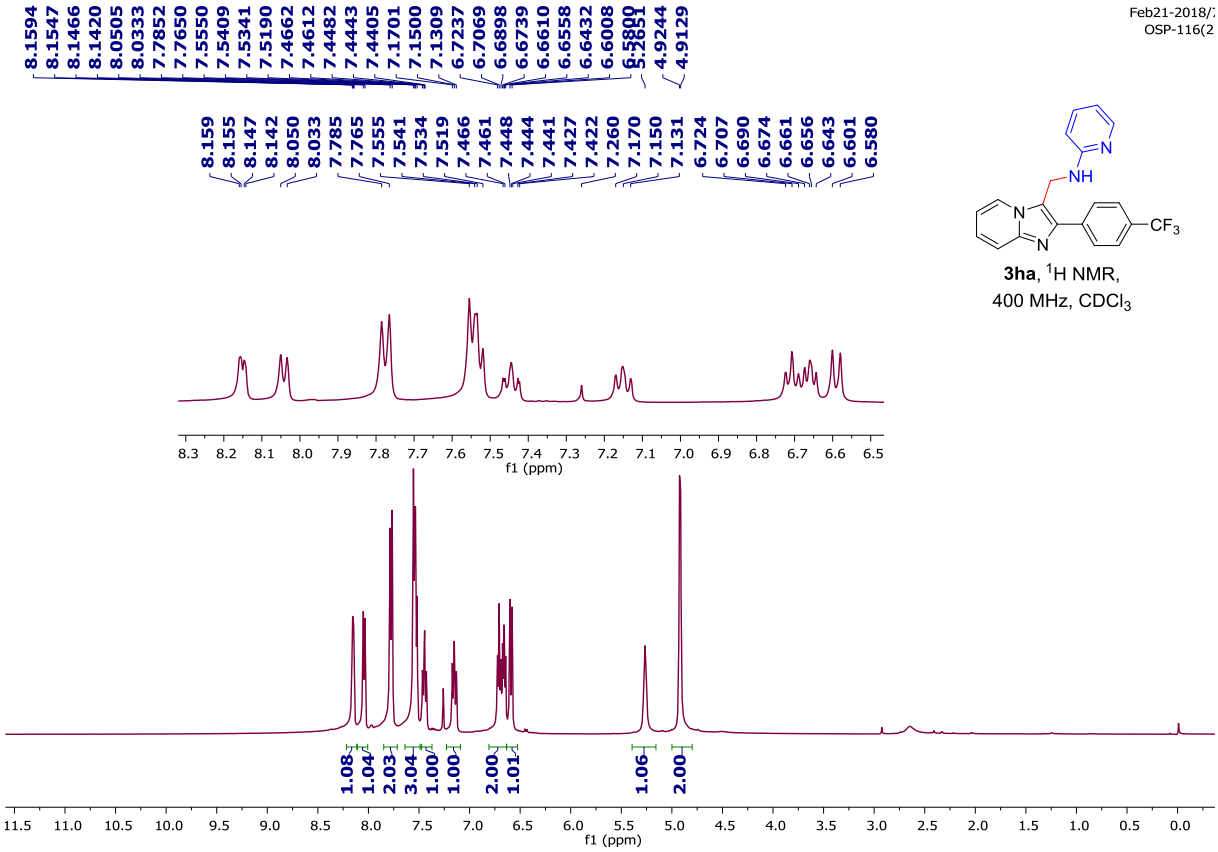


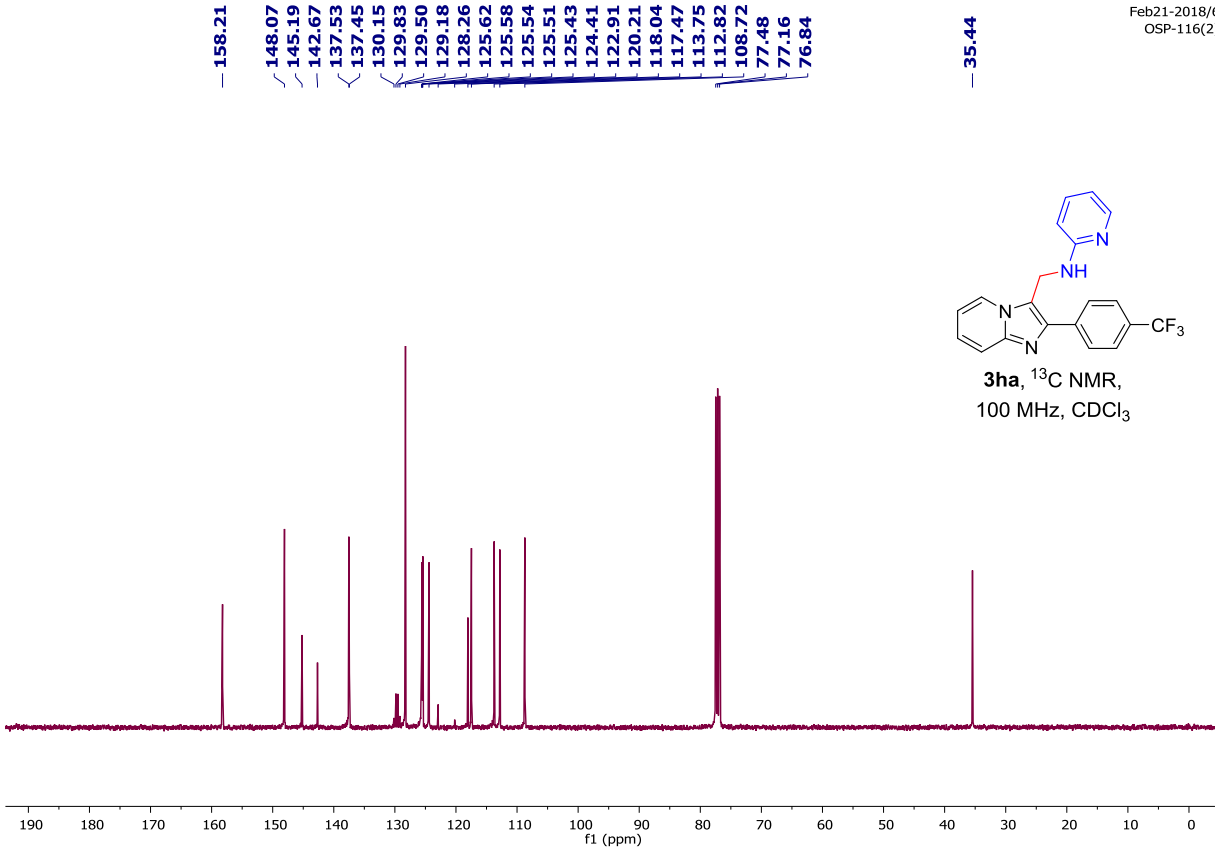
3ga, ^{13}C NMR,
100 MHz, $CDCl_3$



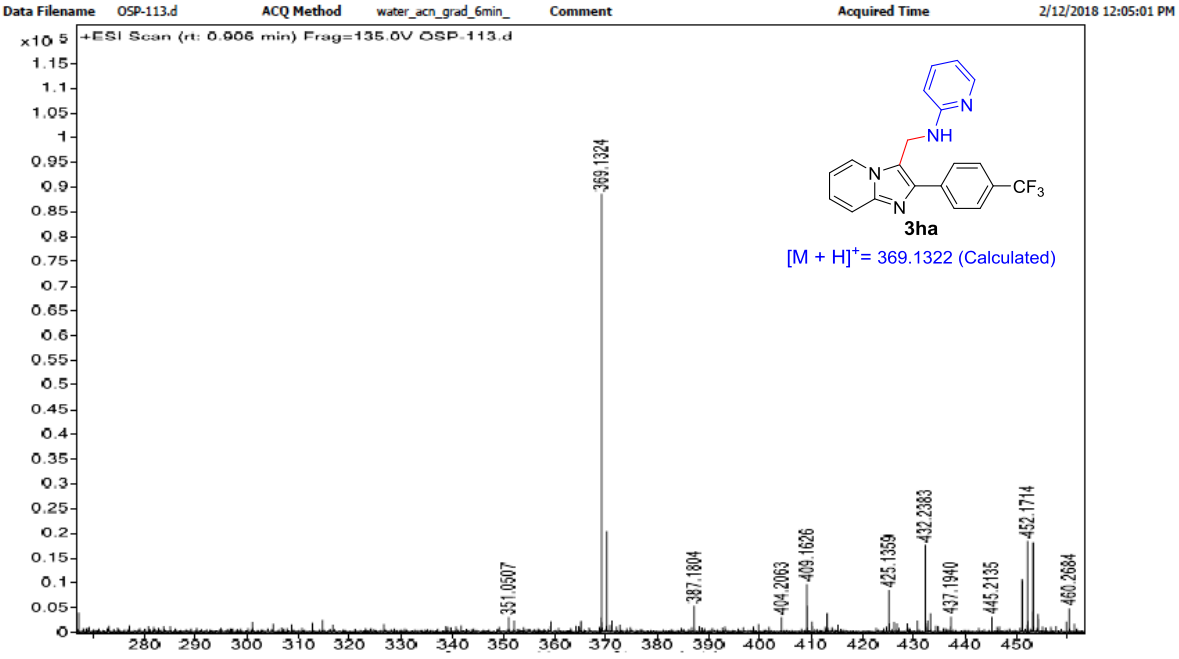
Sample Name	OSP-116	**Position**	P2-D9	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-116.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	4/12/2018 4:24:15 PM

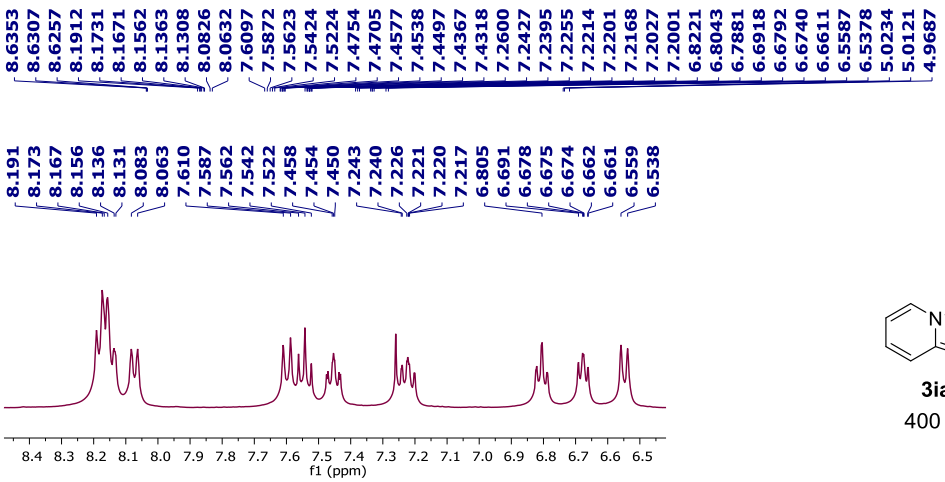


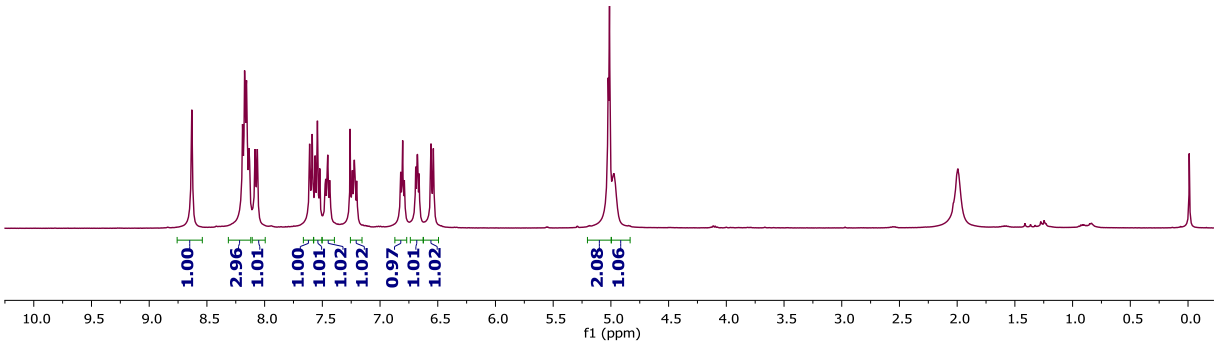

Feb21-2018/
OSP-116(2
8.1594
8.1547
8.1466
8.1420
8.0505
8.0333
7.7852
7.7650
7.5550
7.5409
7.5341
7.5190
7.4662
7.4612
7.4482
7.4443
7.4405
7.1701
7.1500
7.1309
6.7237
6.7069
6.6898
6.6739
6.6610
6.6558
6.6432
6.6008
5.2851
4.9244
4.9129
8.159
8.155
8.147
8.142
8.050
8.033
7.785
7.765
7.555
7.541
7.534
7.519
7.466
7.461
7.448
7.444
7.441
7.427
7.422
7.260
7.170
7.150
7.131
6.724
6.707
6.690
6.674
6.661
6.656
6.643
6.601
6.580
f1 (ppm)
1.08
1.04
2.03
3.04
1.00
1.00
2.00
1.01
1.06
2.00
f1 (ppm)
N
NH
N
N
CF3
3ha, 1H NMR,
400 MHz, CDCl3

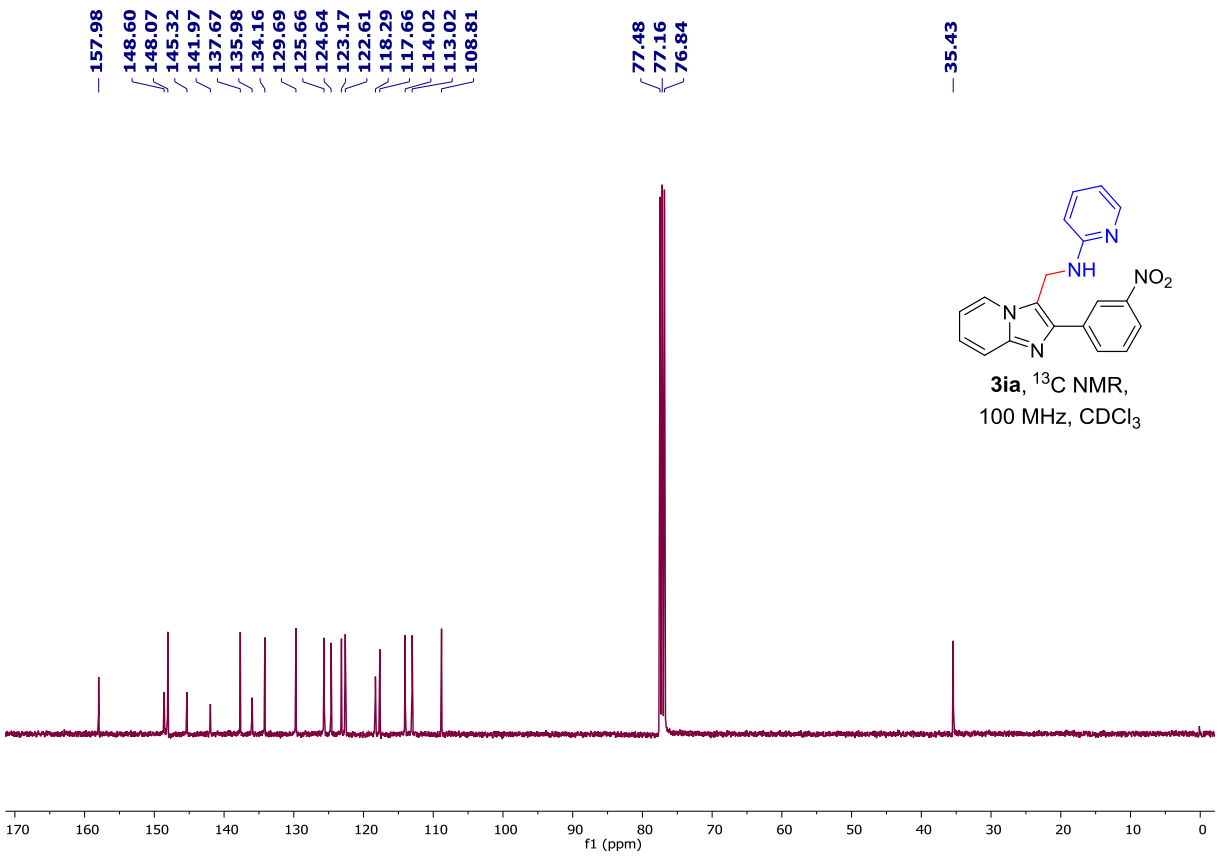

Feb21-2018/
OSP-116(2
158.21
148.07
145.19
142.67
137.53
137.45
130.15
129.83
129.50
129.18
128.26
125.62
125.58
125.54
125.51
125.43
124.41
122.91
120.21
118.04
117.47
113.75
112.82
108.72
77.48
77.16
76.84
35.44
f1 (ppm)
N
NH
N
N
CF3
3ha, 13C NMR,
100 MHz, CDCl3

Sample Name	OSP-113	**Position**	P1-E7	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-113.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	2/12/2018 12:05:01 PM



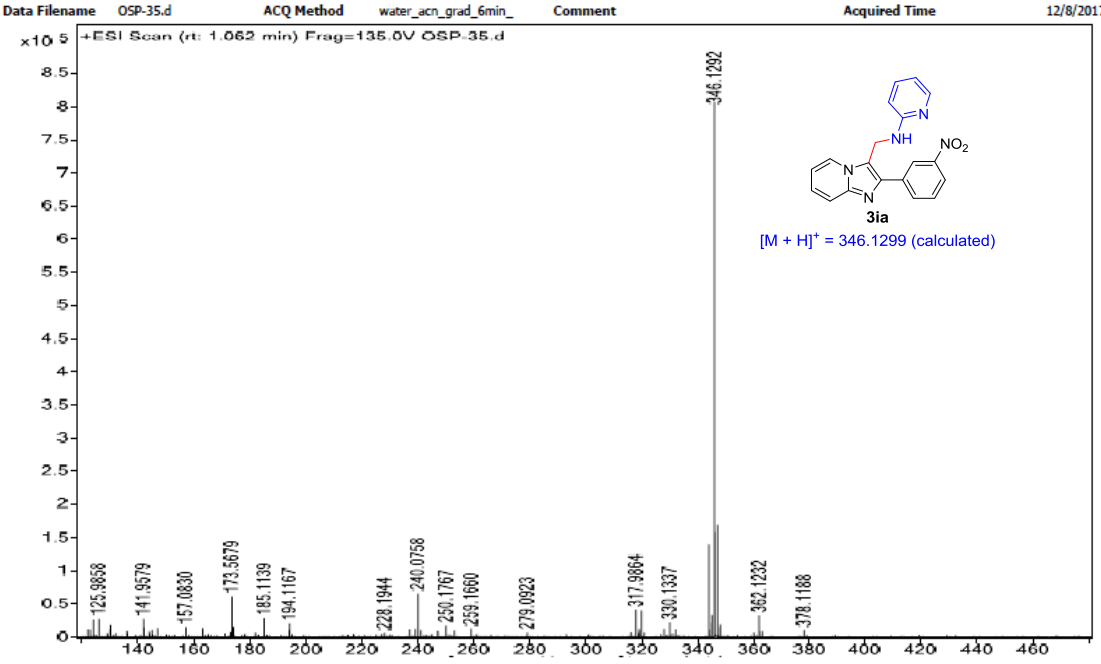






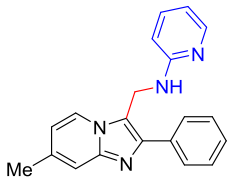
3ia, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-35	**Position**	P2-E5	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-35.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	12/8/2017 1:12:08 PM

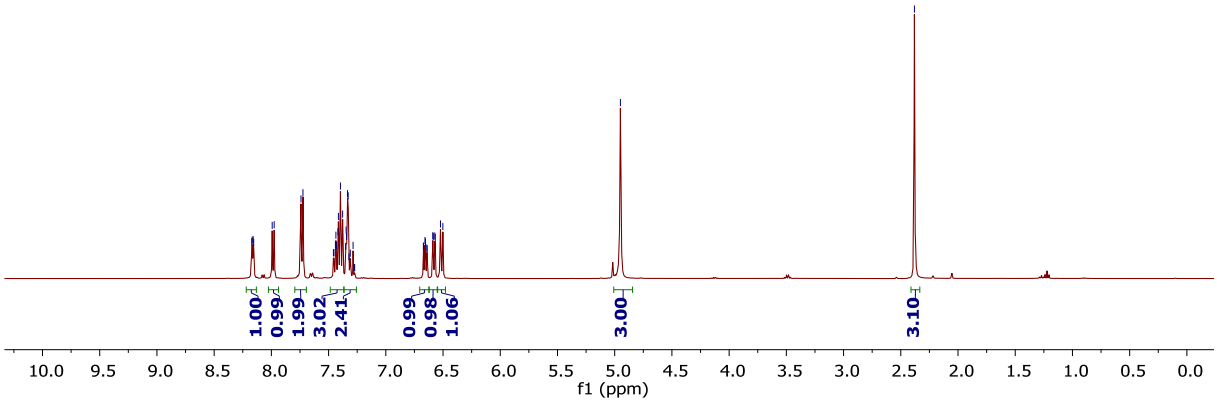


$[M + H]^+$ = 346.1299 (calculated)

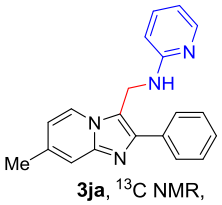
8.1717
8.1670
8.1589
8.1544
7.9929
7.9755
7.7412
7.7240
7.4401
7.4362
7.4186
7.4143
7.3965
7.3774
7.3467
7.3431
7.3346
7.3290
7.3220
6.6699
6.6580
6.6531
6.6415
6.6392
6.5895
6.5851
6.5720
6.5678
6.5224
6.5015
4.9500
2.3809



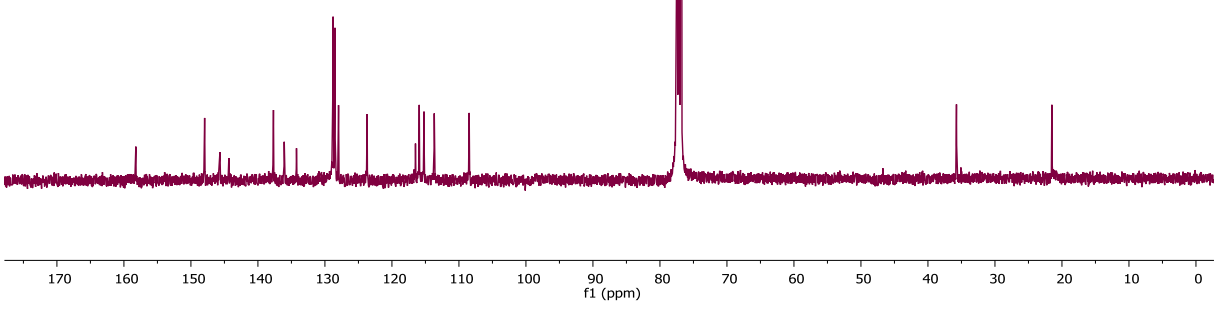
3ja, ^{1}H NMR,
400 MHz, $CDCl_3$



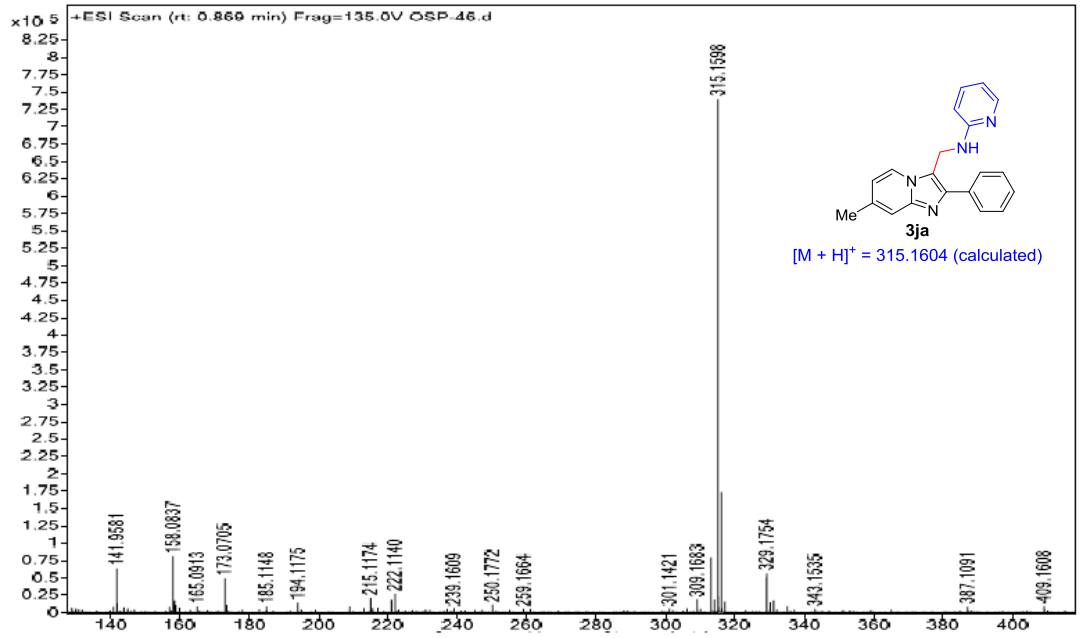
158.22
147.95
145.67
144.35
137.68
136.07
134.24
128.79
128.50
127.97
123.70
116.49
115.92
115.21
113.68
108.47
77.48
77.16
76.84
35.74
21.50



3ja, ^{13}C NMR,
100 MHz, $CDCl_3$

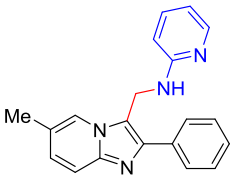


Sample Name	OSP-46	**Position**	P2-F5	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-46.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	10/12/2017 5:58:32 PM

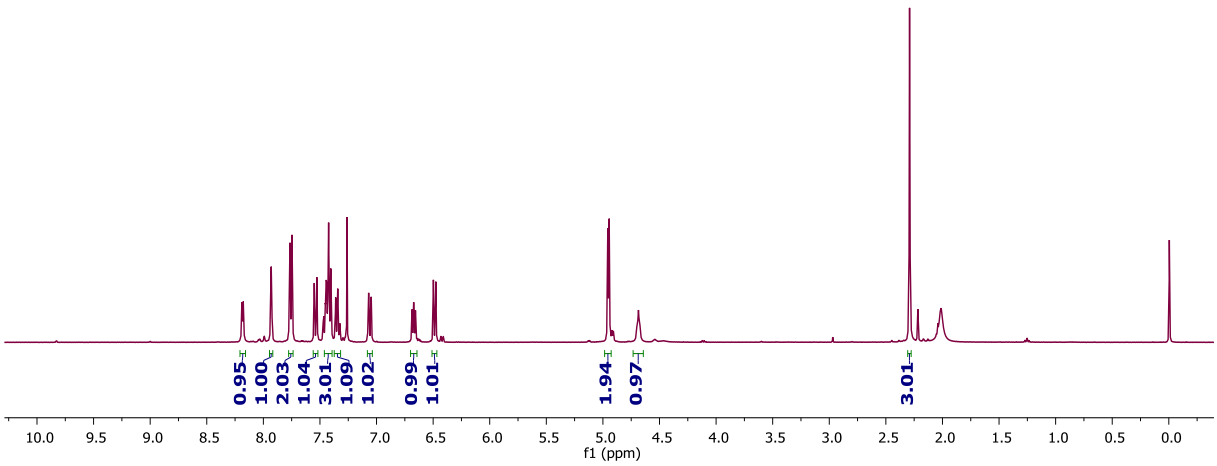


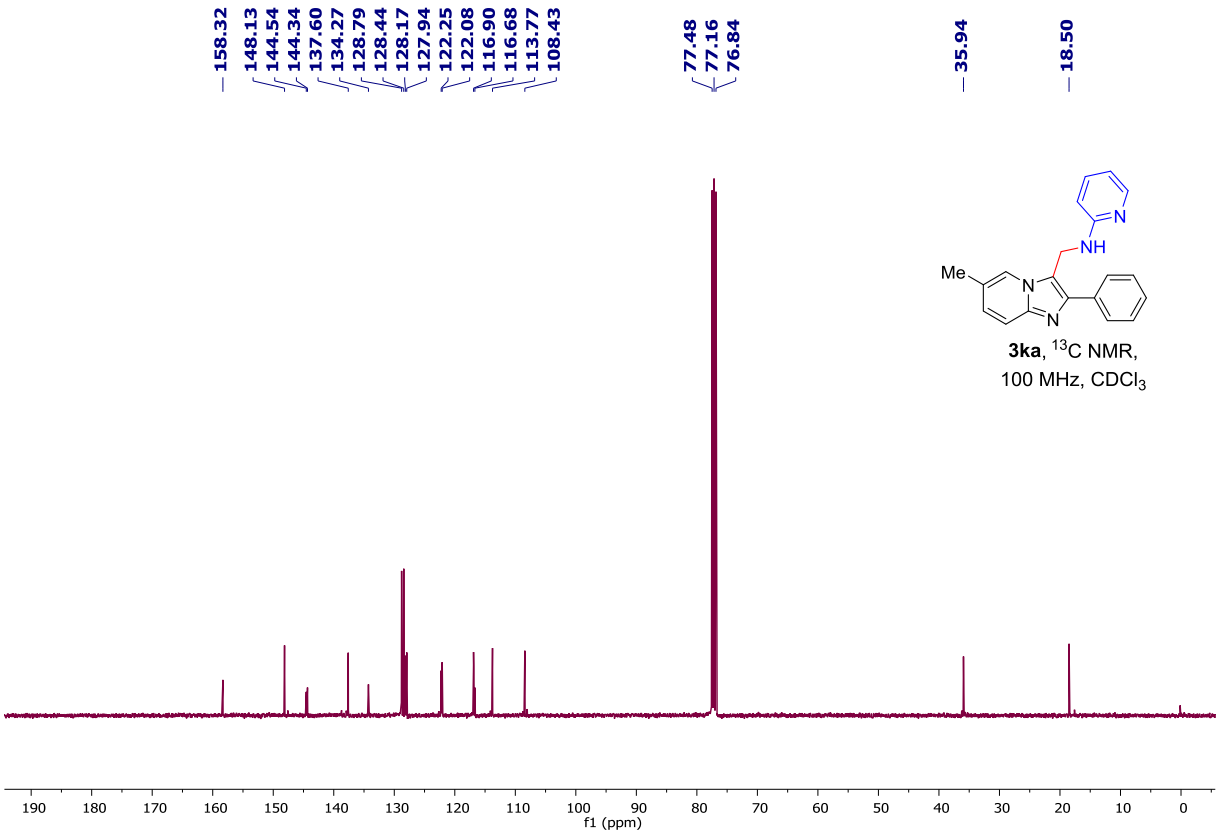
3ja

$[M + H]^+$ = 315.1604 (calculated)



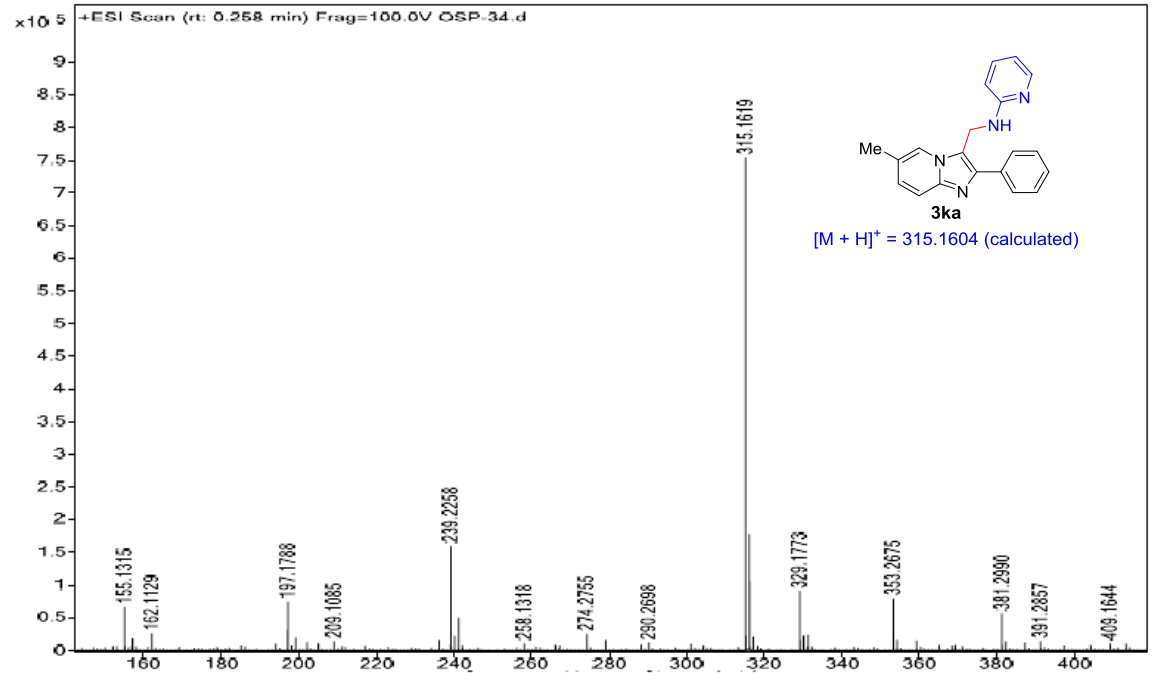
3ka, ^{1}H NMR,
400 MHz, $CDCl_3$



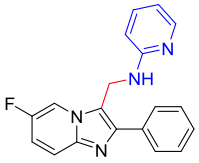


3ka, ^{13}C NMR,
100 MHz, $CDCl_3$

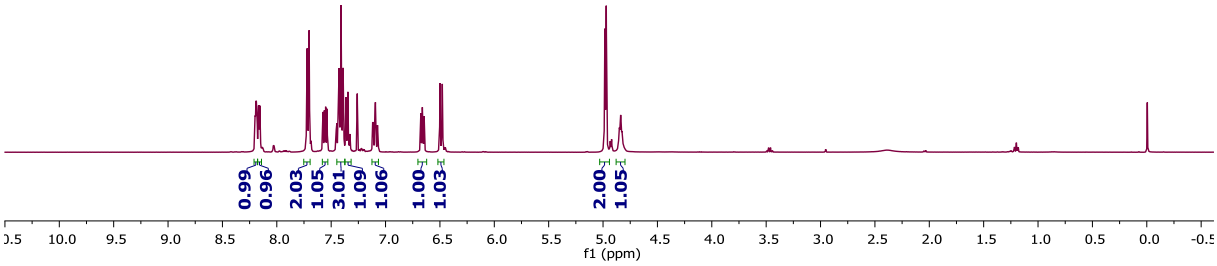
Sample Name	OSP-34	**Position**	P2-C9	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-34.d	**ACQ Method**	Methanol_isocratic.m	**Comment**		**Acquired Time**	2/2/2018 6:14:56 PM

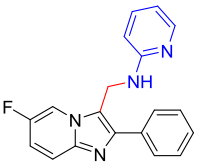


$[M + H]^+$ = 315.1604 (calculated)

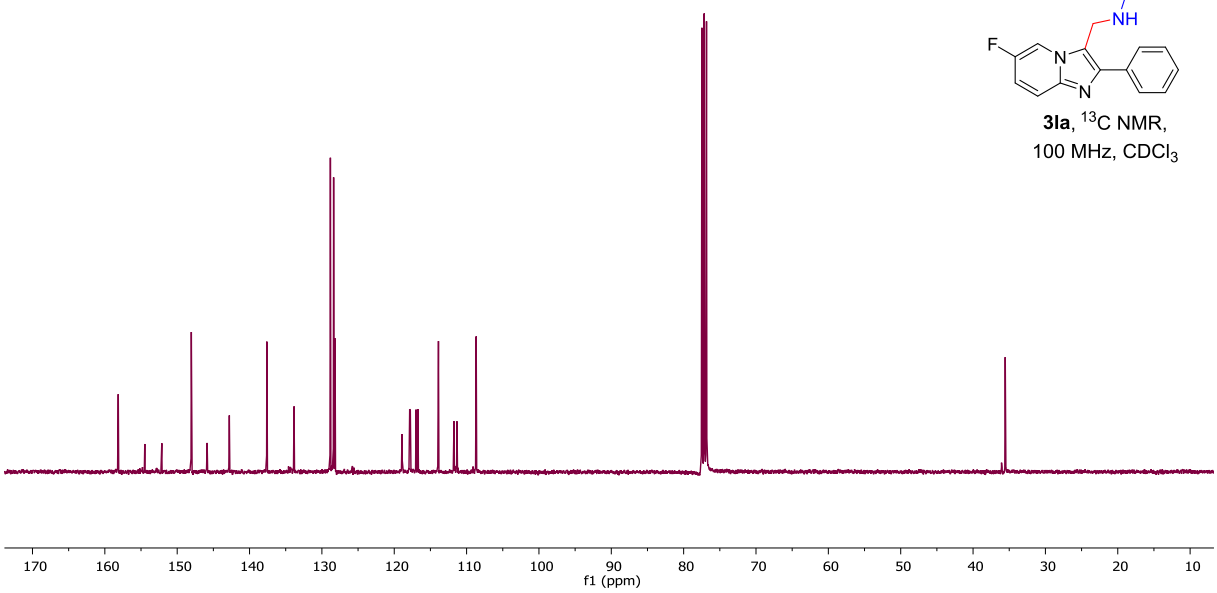


3la, ^{1}H NMR,
400 MHz, $CDCl_3$

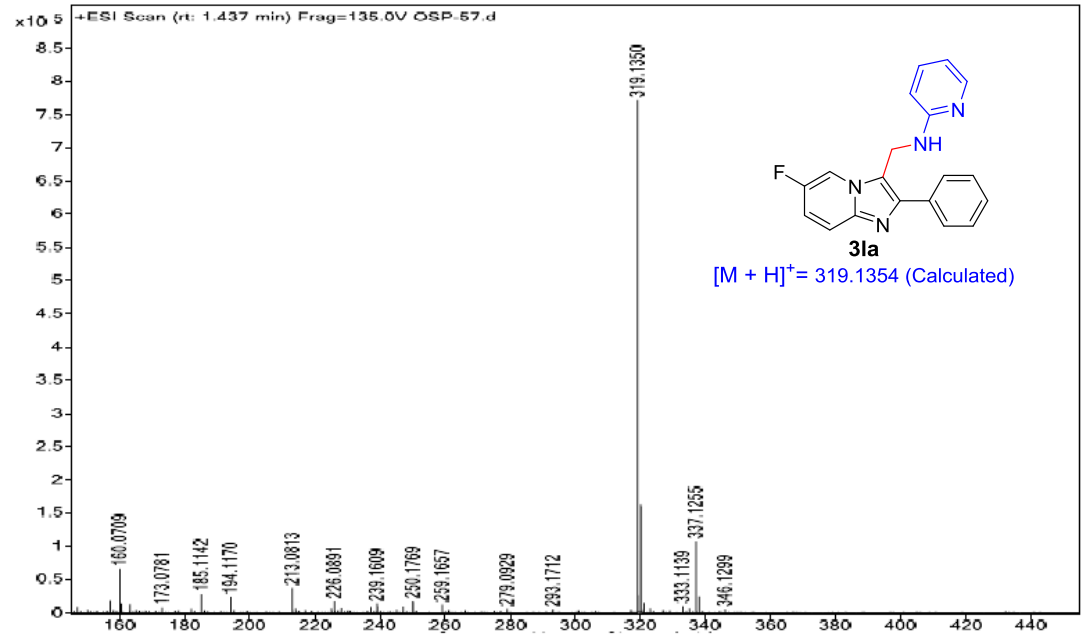




3la, ^{13}C NMR,
100 MHz, $CDCl_3$

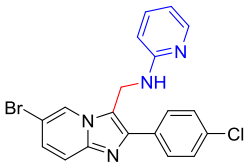


Sample Name	OPS-57	**Position**	P2-E6	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-57.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	12/8/2017 1:26:14 PM

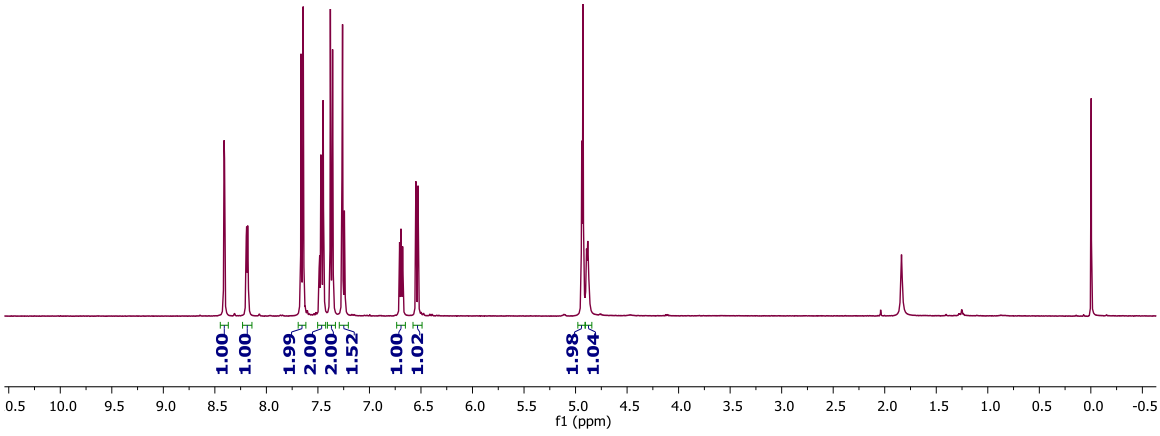


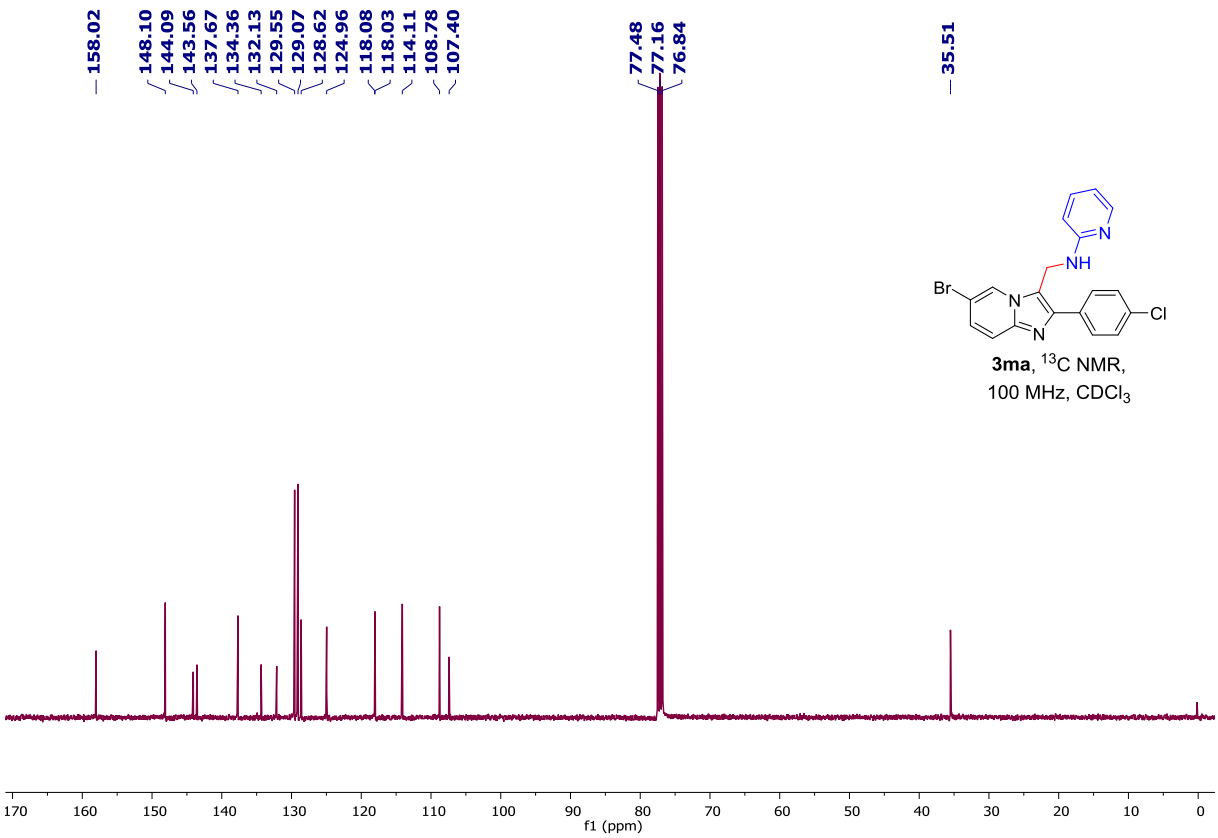
3la

$[M + H]^+$= 319.1354 (Calculated)



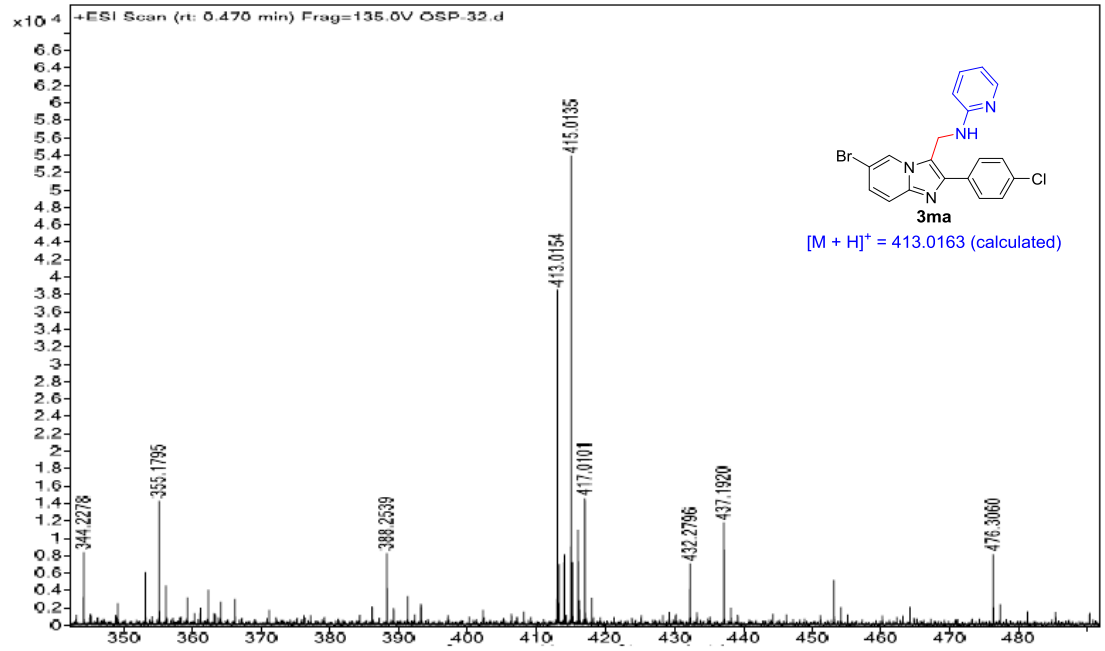
3ma, ^{1}H NMR,
400 MHz, $CDCl_3$





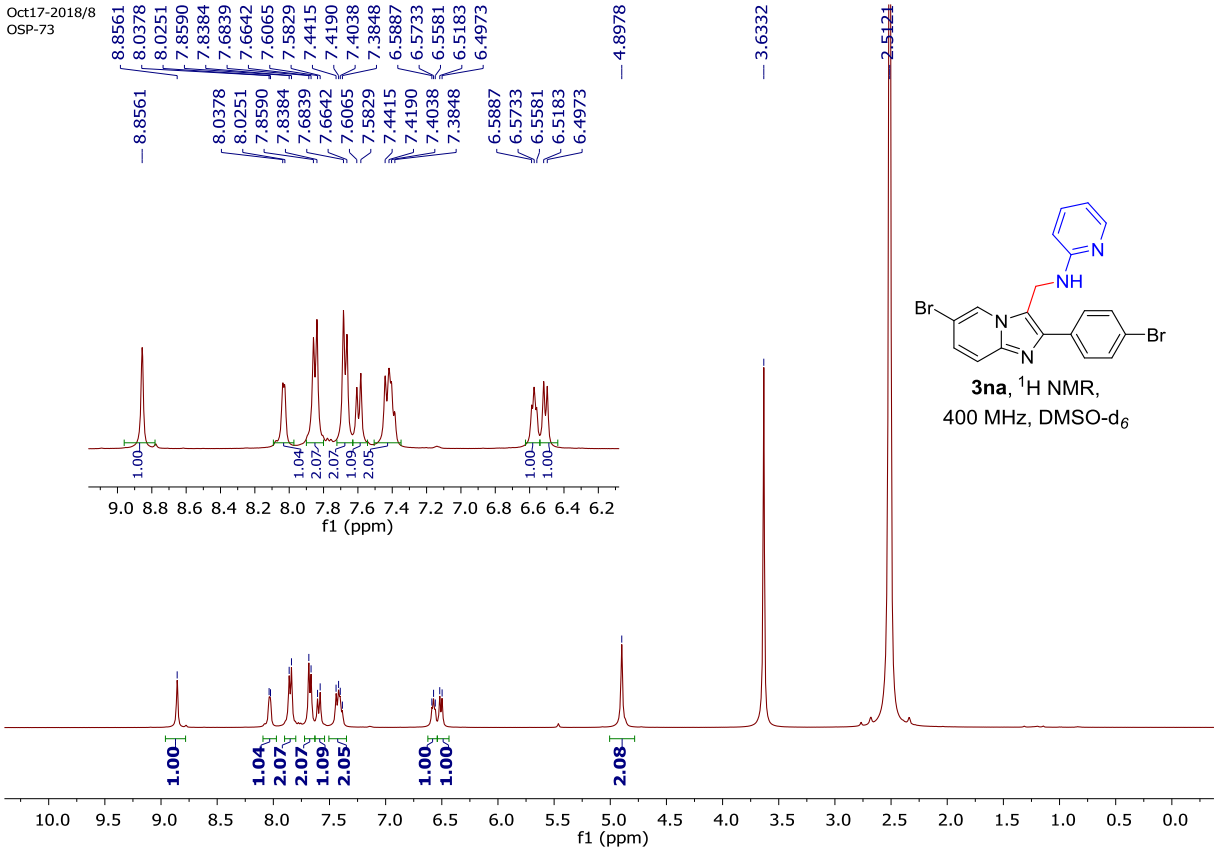
3ma, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-32	**Position**	P1-D3	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-32.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	9/22/2017 10:21:39 PM

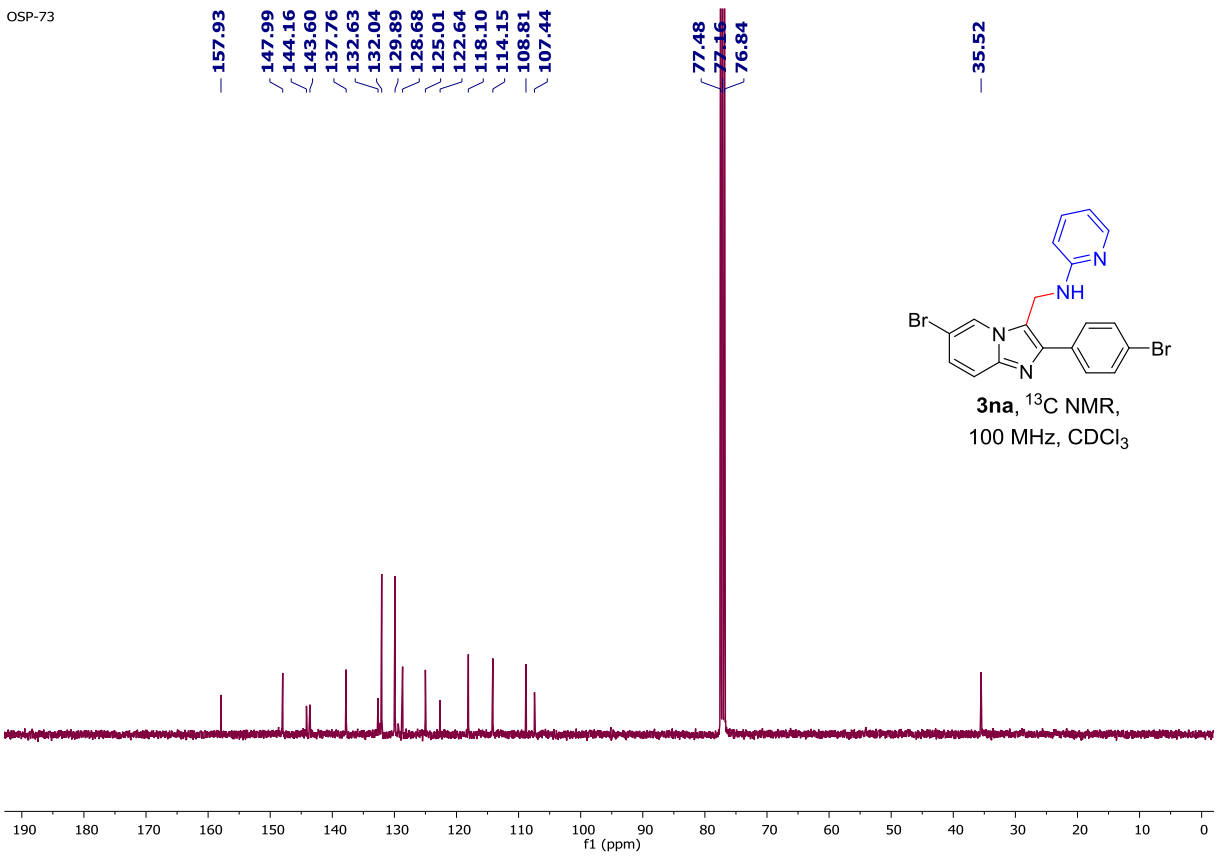


3ma

$[M + H]^+$ = 413.0163 (calculated)

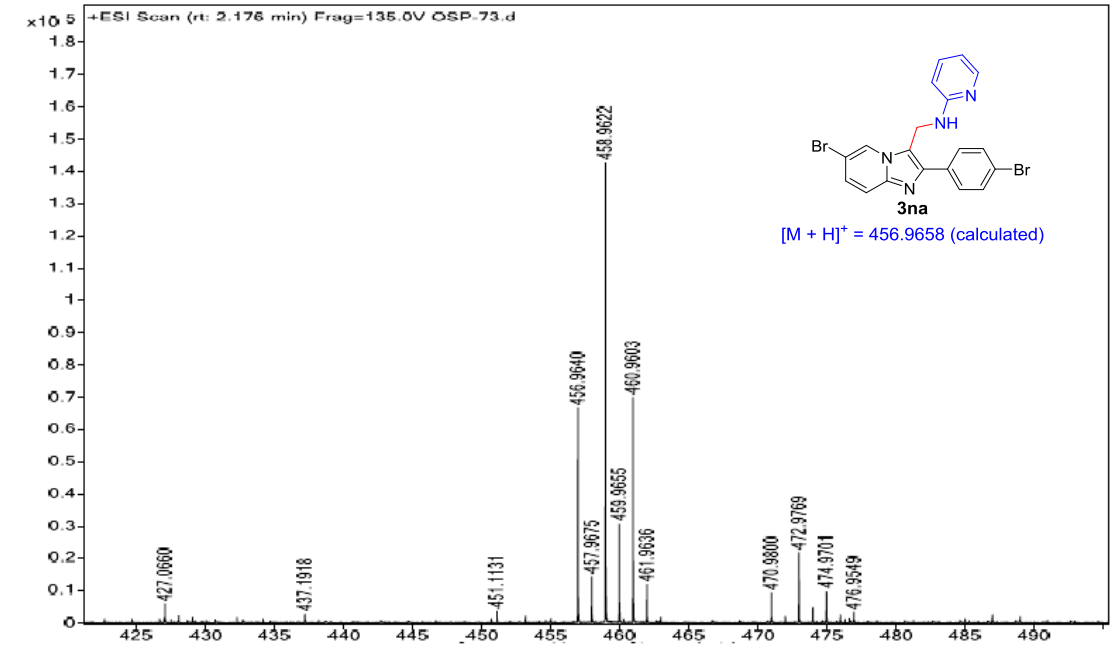


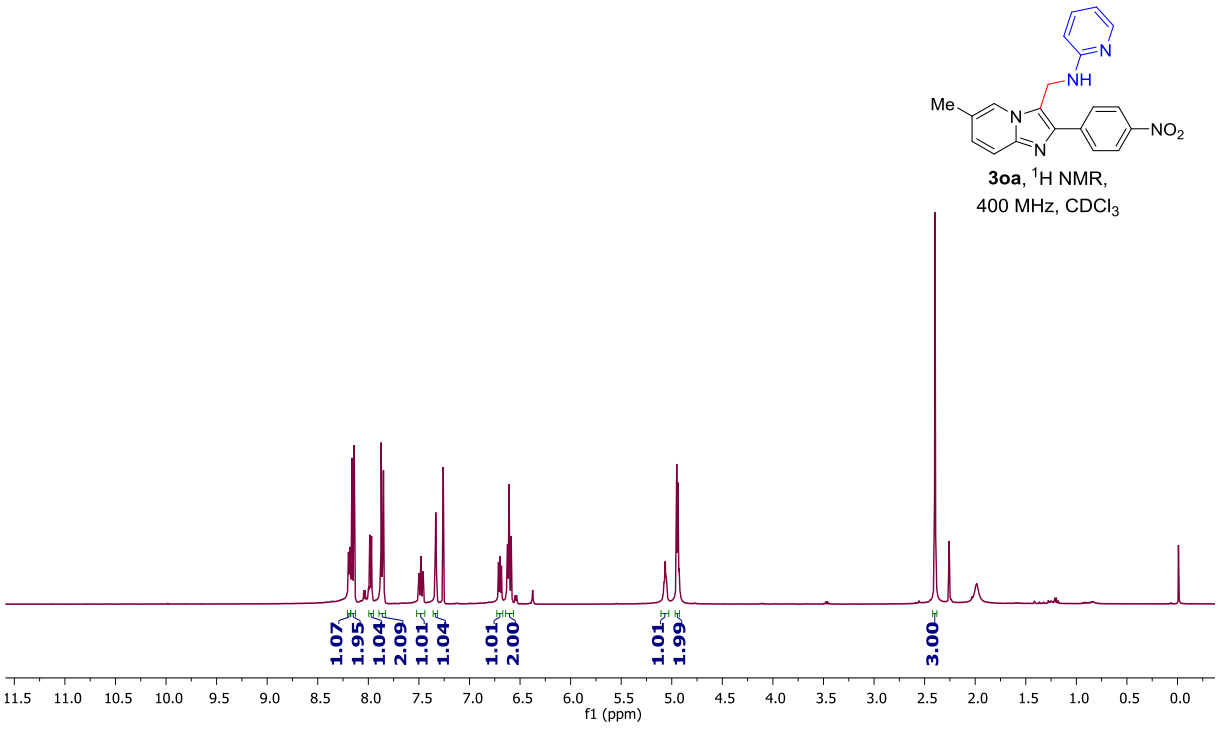
3na, ^{1}H NMR,
400 MHz, DMSO-d_6

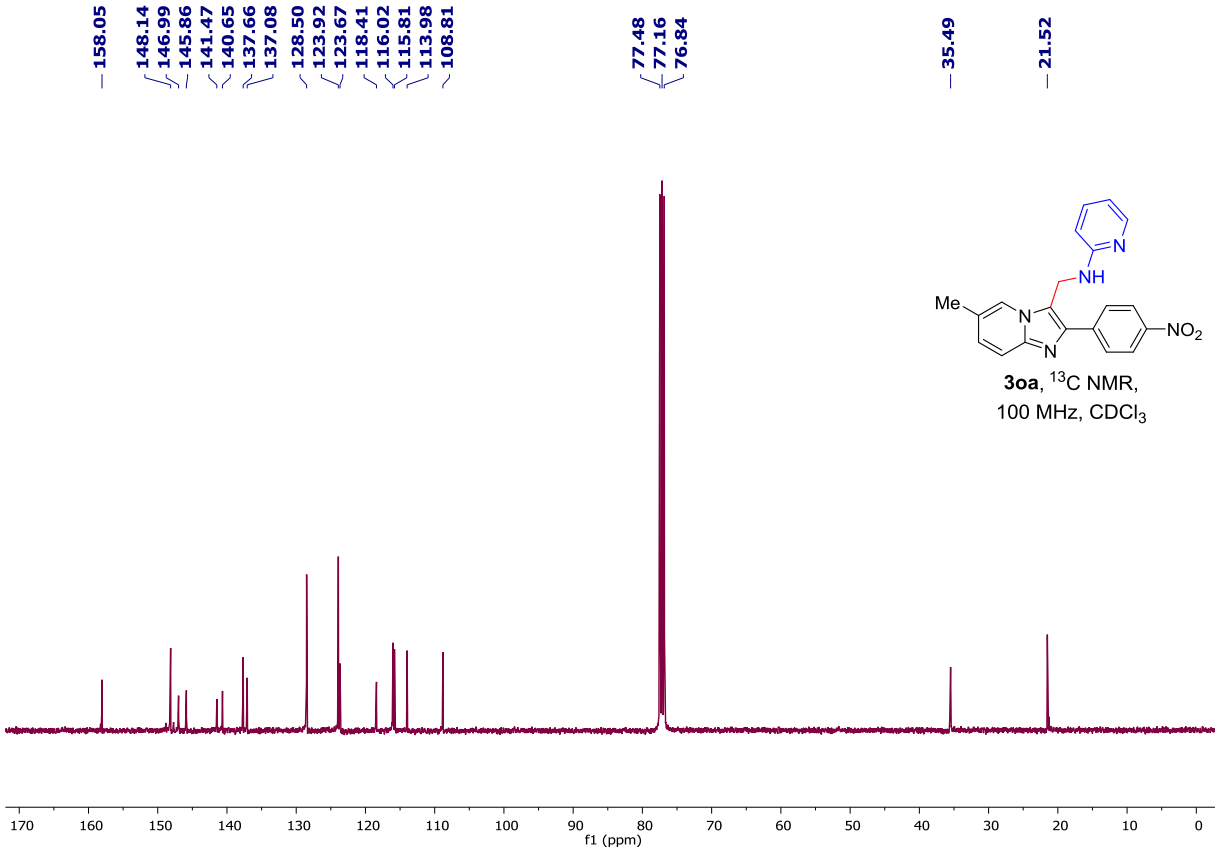


3na, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-73	**Position**	P2-D7	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-73.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	1/17/2018 5:19:39 PM

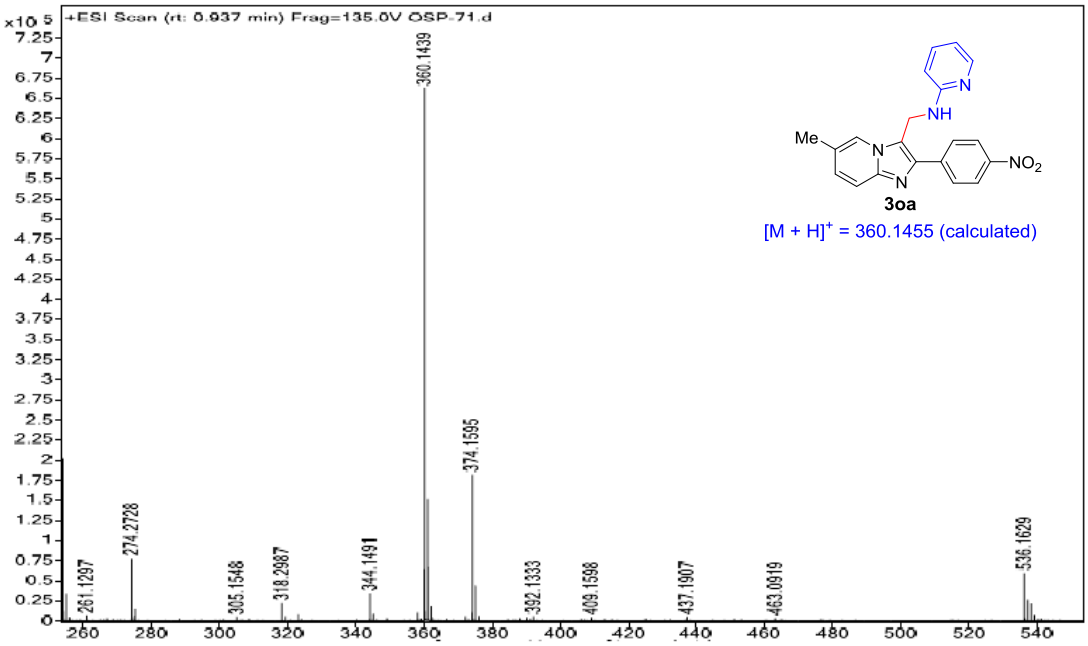






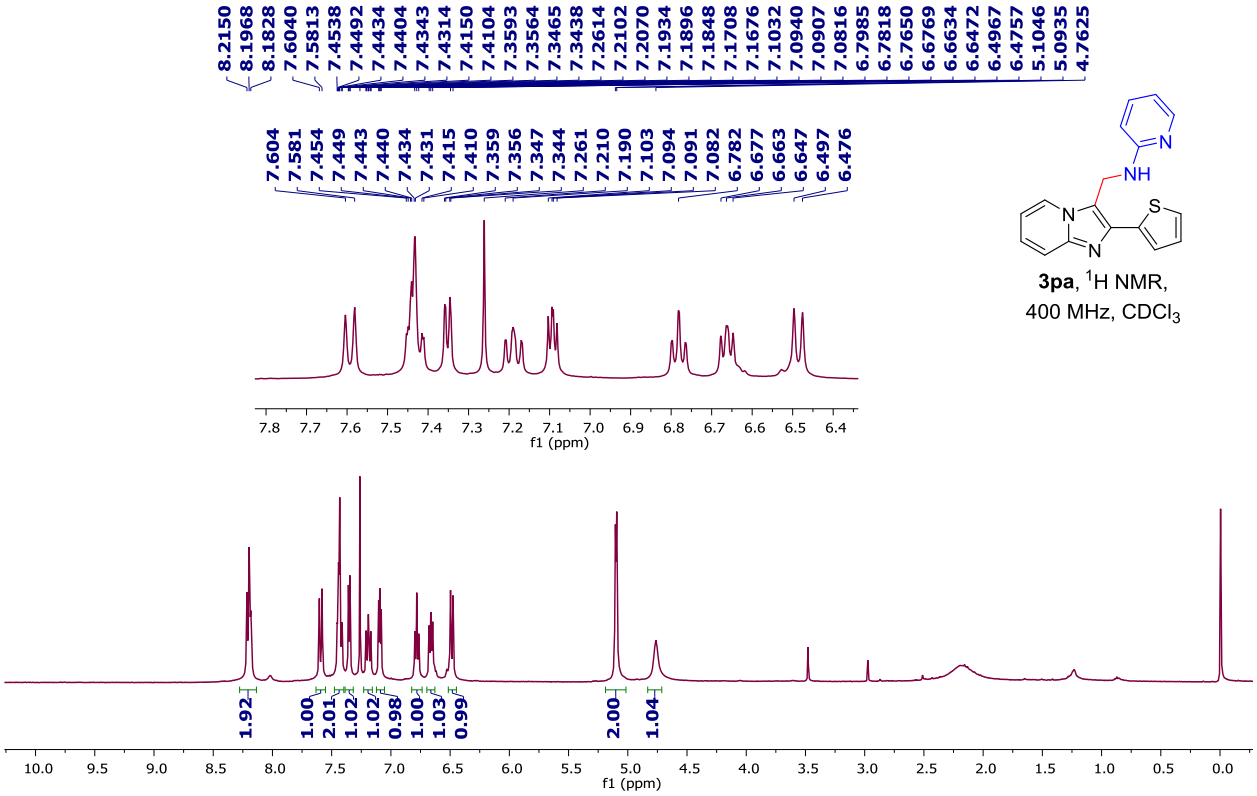
3oa, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-71	**Position**	P2-D4	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-71.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	1/17/2018 4:37:15 PM

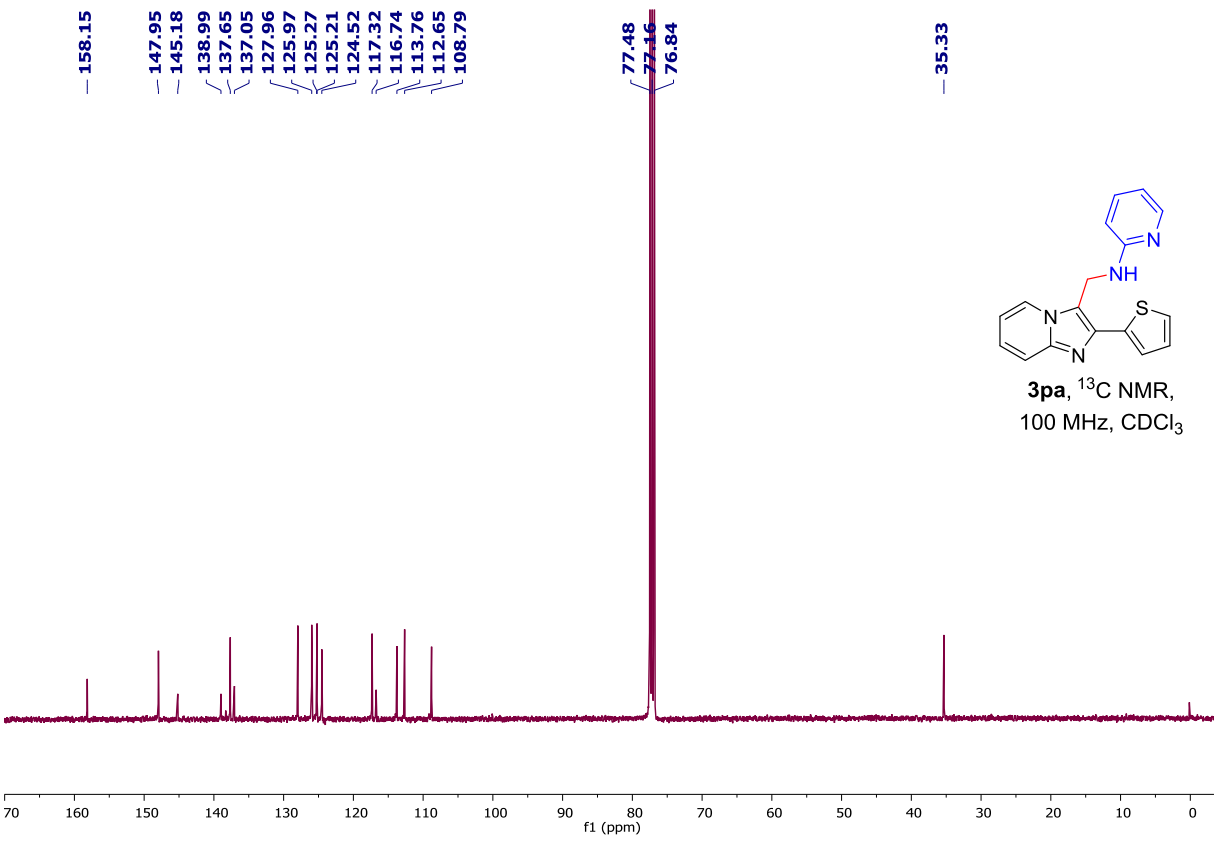


3oa

$[M + H]^+$ = 360.1455 (calculated)

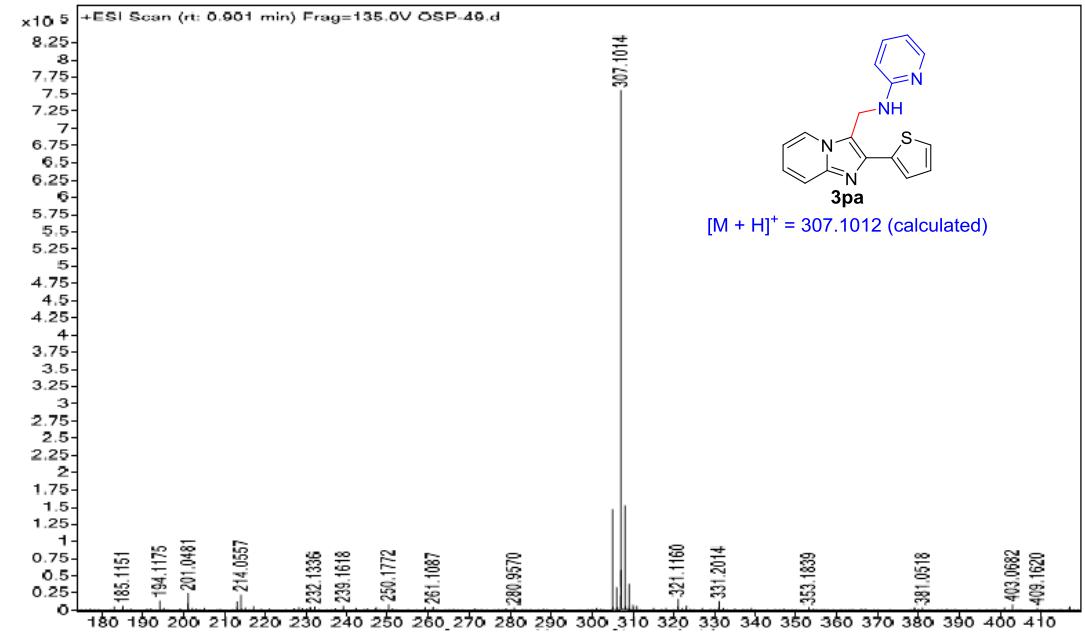


3pa, ^{1}H NMR,
400 MHz, $CDCl_3$



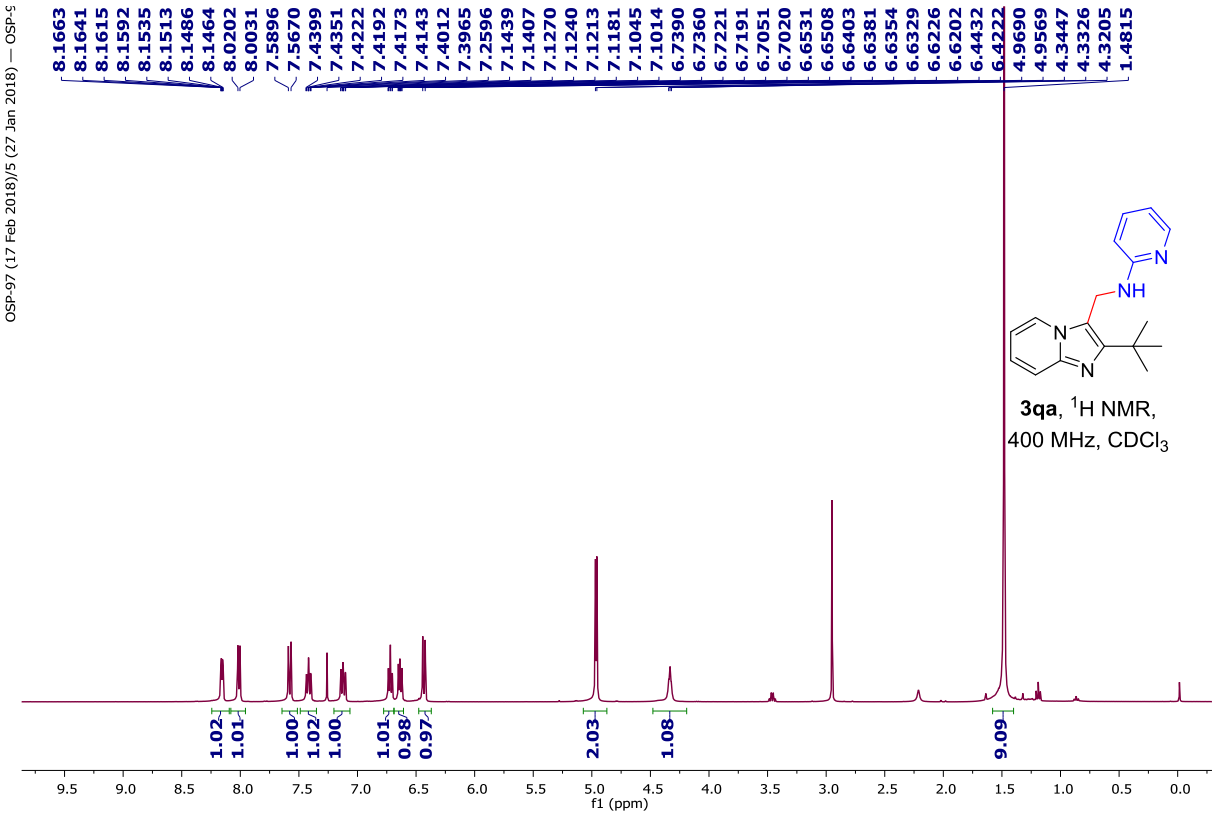
3pa, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-49	**Position**	P2-F7	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-49.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	10/12/2017 6:26:48 PM



3pa

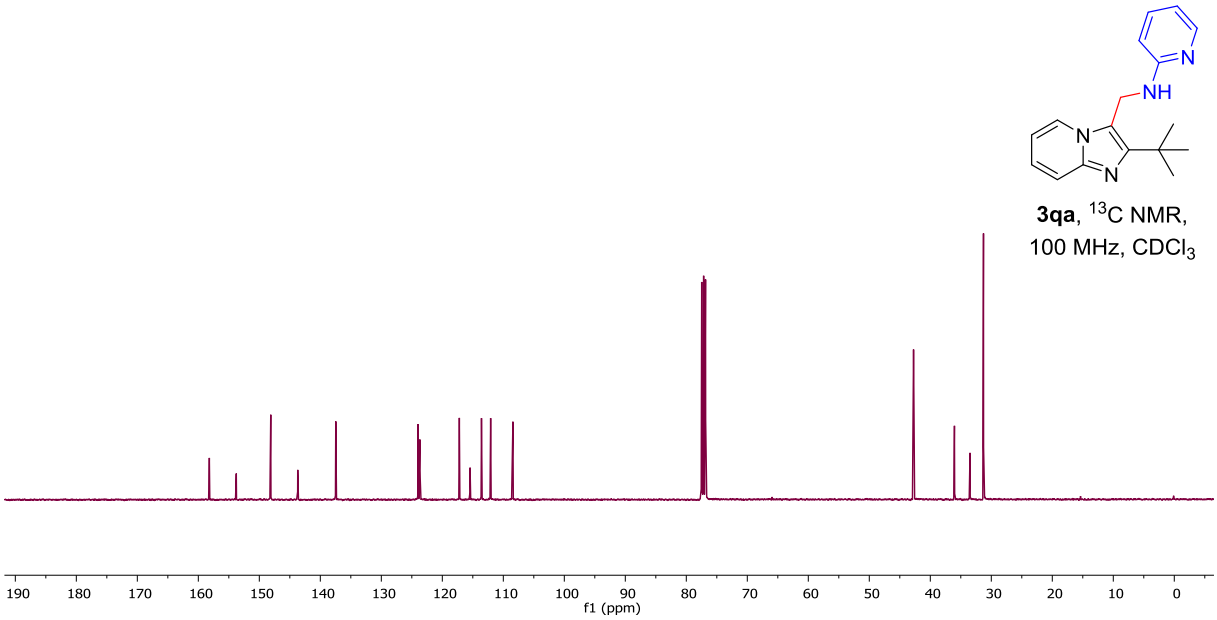
$[M + H]^+$ = 307.1012 (calculated)



3qa, ^{1}H NMR, 400 MHz, $CDCl_3$

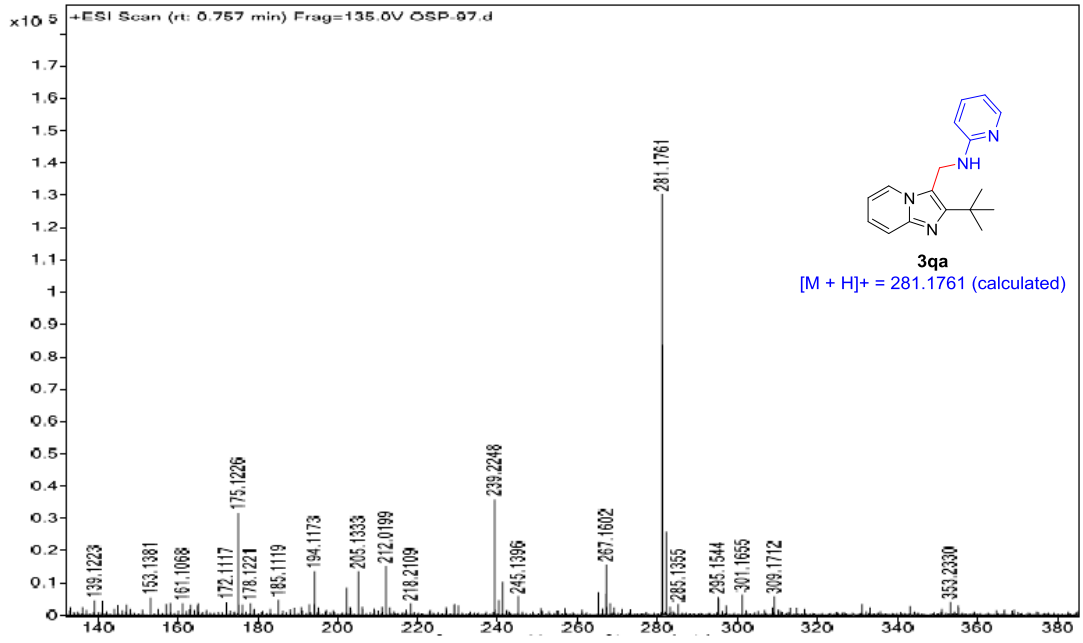
Feb19-2018/2
OSP-97

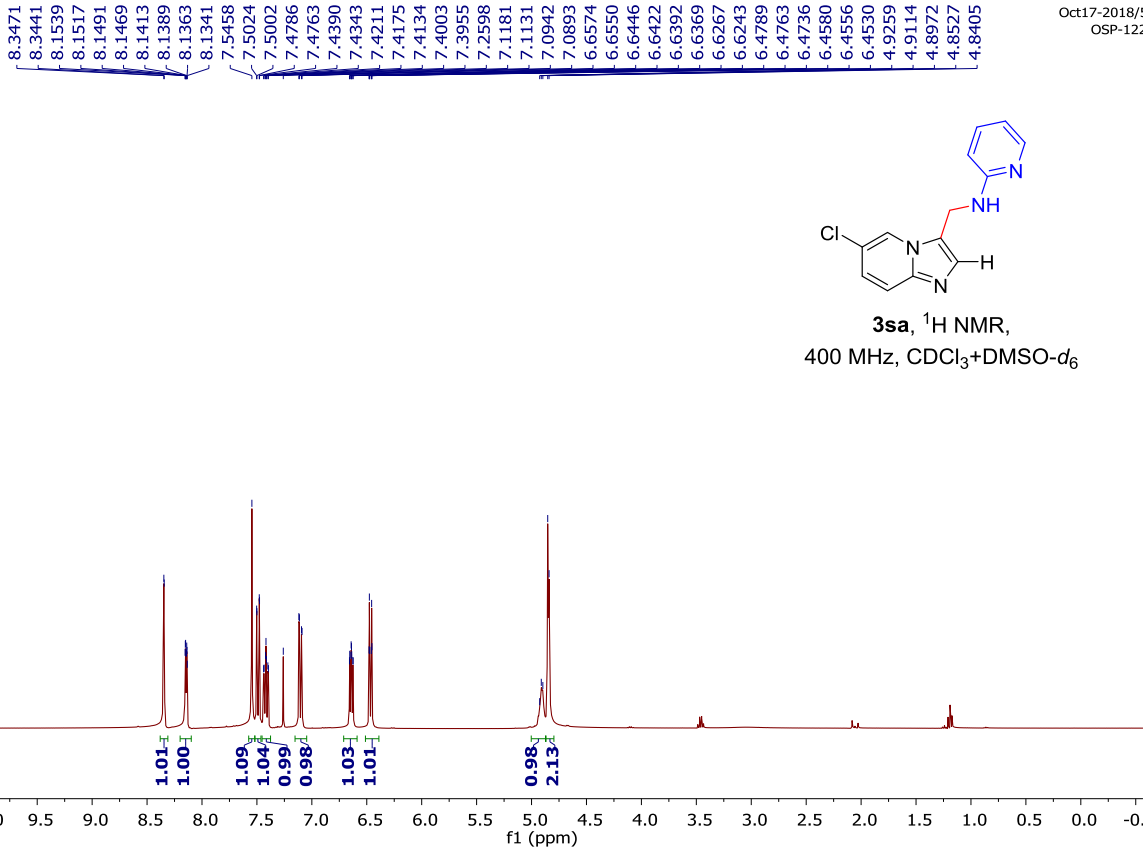
158.19
153.77
148.13
143.70
137.47
123.98
123.68
117.20
115.45
113.58
112.08
108.43
77.48
77.16
76.84
36.04
33.49
31.28



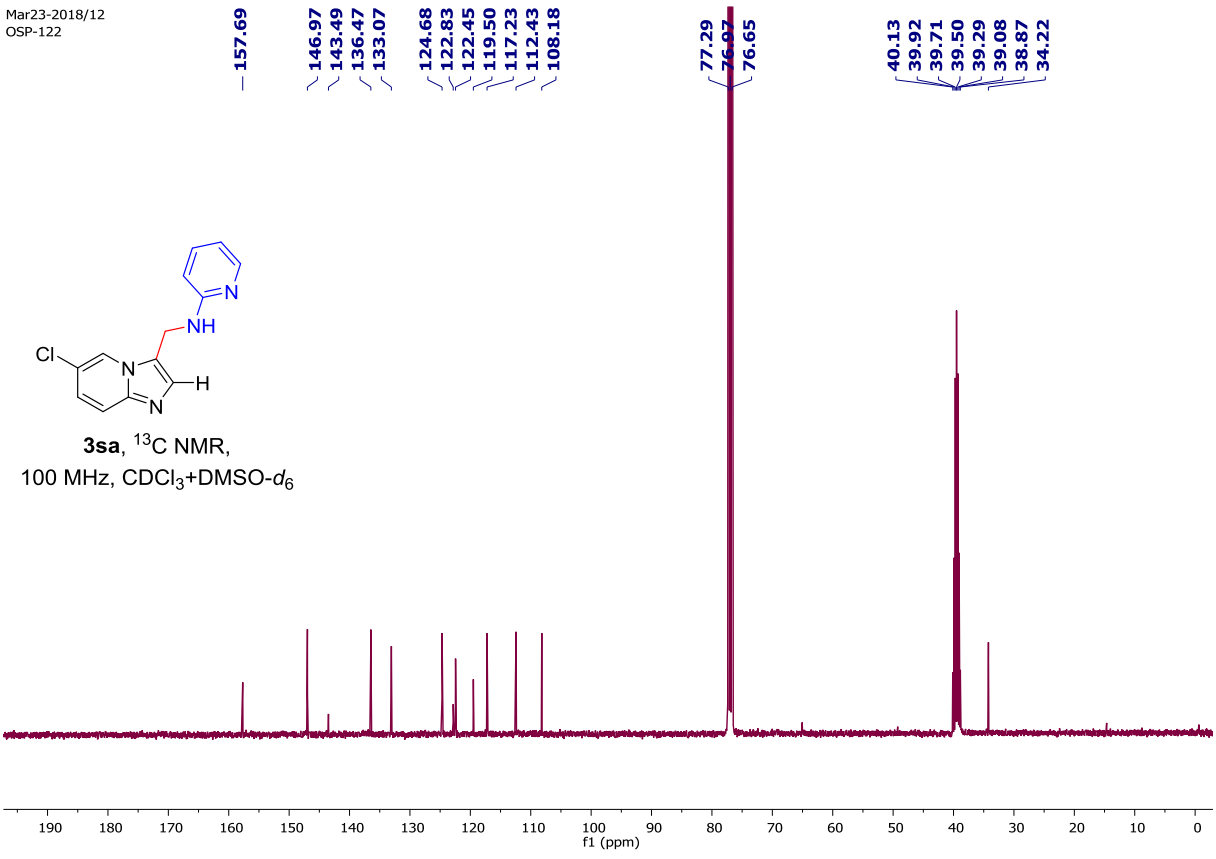
3qa, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-97	**Position**	P1-B8	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-97.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	1/24/2018 5:23:07 PM



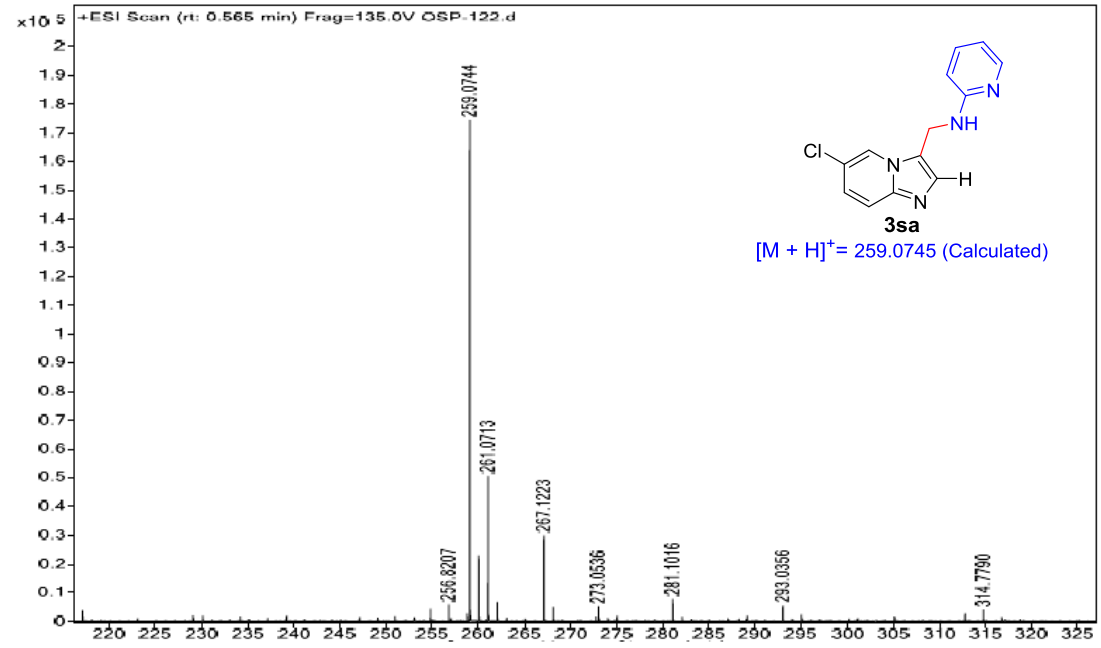


3sa, ^{1}H NMR,
400 MHz, $CDCl_3$+DMSO-d_6



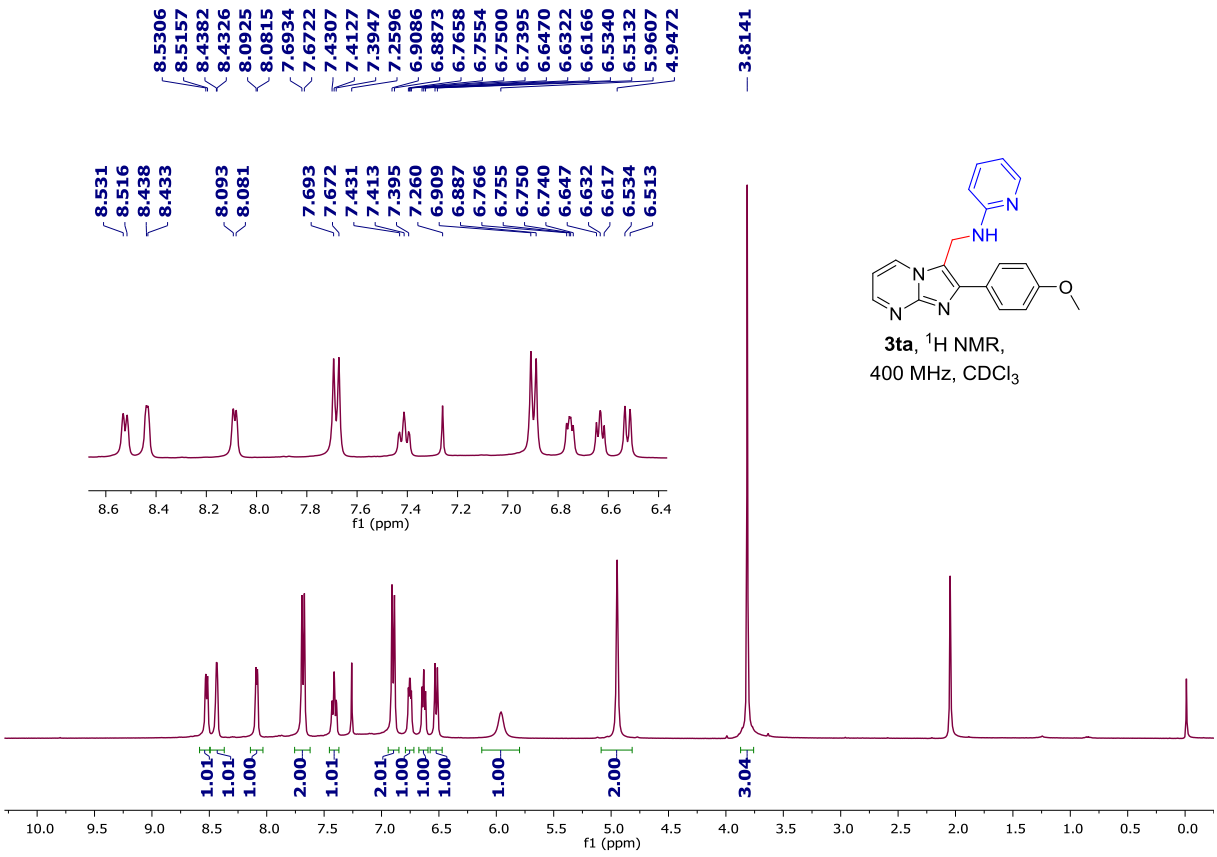
3sa, ^{13}C NMR,
100 MHz, $CDCl_3$+DMSO-d_6

Sample Name	OSP-122	**Position**	P1-B5	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-122.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	4/3/2018 12:23:21 PM

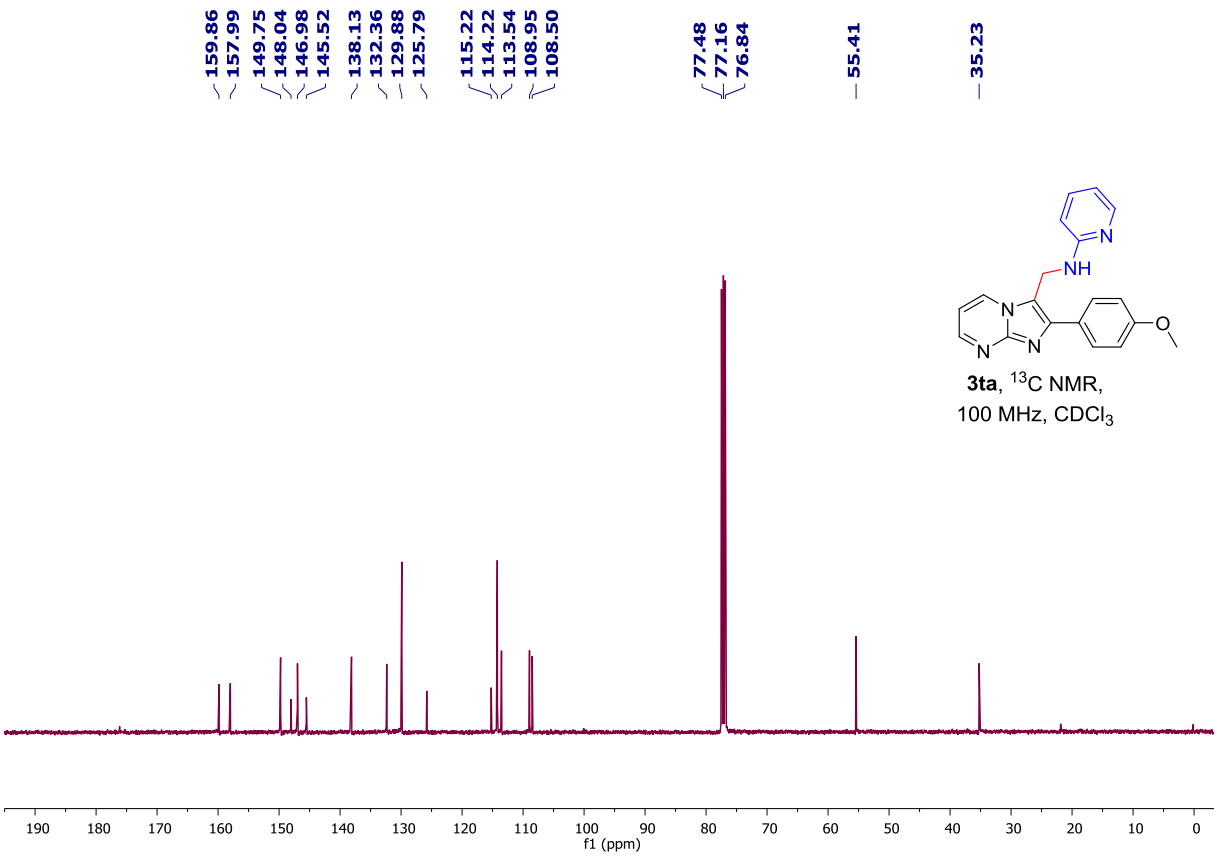


3sa

$[M + H]^+$= 259.0745 (Calculated)

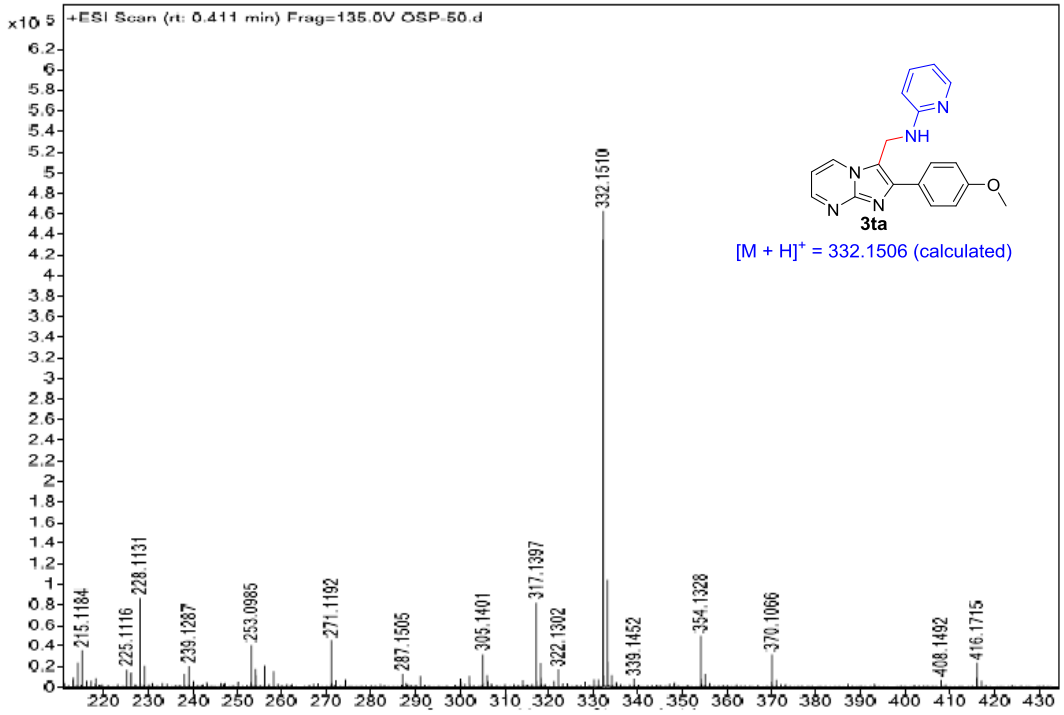


3ta, ^{1}H NMR,
400 MHz, $CDCl_3$



3ta, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-50	**Position**	P2-F8	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-50.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	10/12/2017 6:40:56 PM

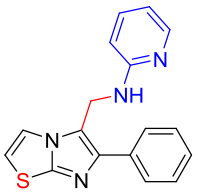


3ta

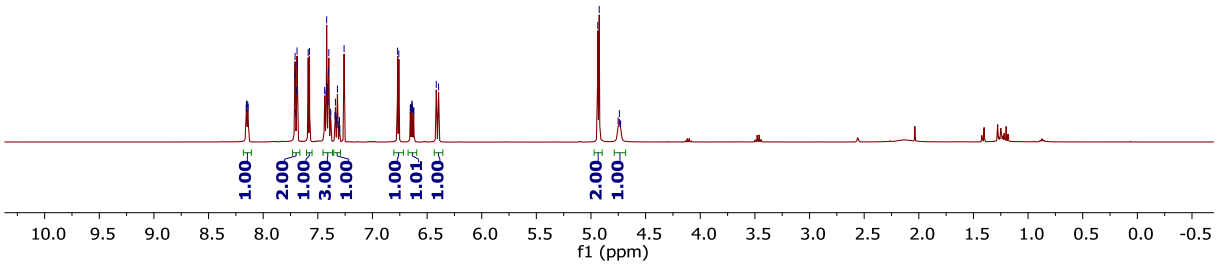
$[M + H]^+$ = 332.1506 (calculated)

Oct17-2018/7
OSP-70

8.1554 8.1533 8.1510 8.1489 8.1417 8.1372 7.7117 7.7079 7.7031 7.6939 7.6903 7.6877 7.5881 7.5769 7.4376 7.4245 7.4193 7.4154 7.4070 7.4033 7.3995 7.3860 7.3812 7.3424 7.3390 7.3356 7.3256 7.3206 7.3153 7.3054 7.3020 7.2987 7.2597 6.7701 6.7588 6.6541 6.6519 6.6414 6.6392 6.6361 6.6339 6.6235 6.6212 6.4158 6.3949 4.9382 4.9248 4.7520 4.7402 4.7290

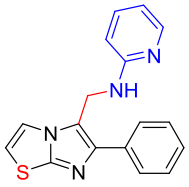


3ua, ^{1}H NMR,
400 MHz, $CDCl_3$

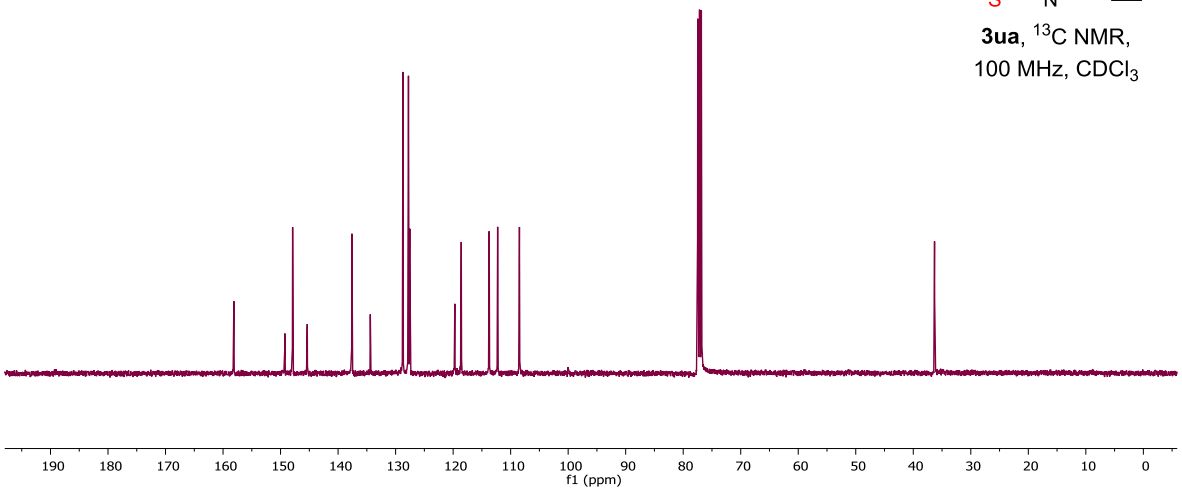


OSP-70

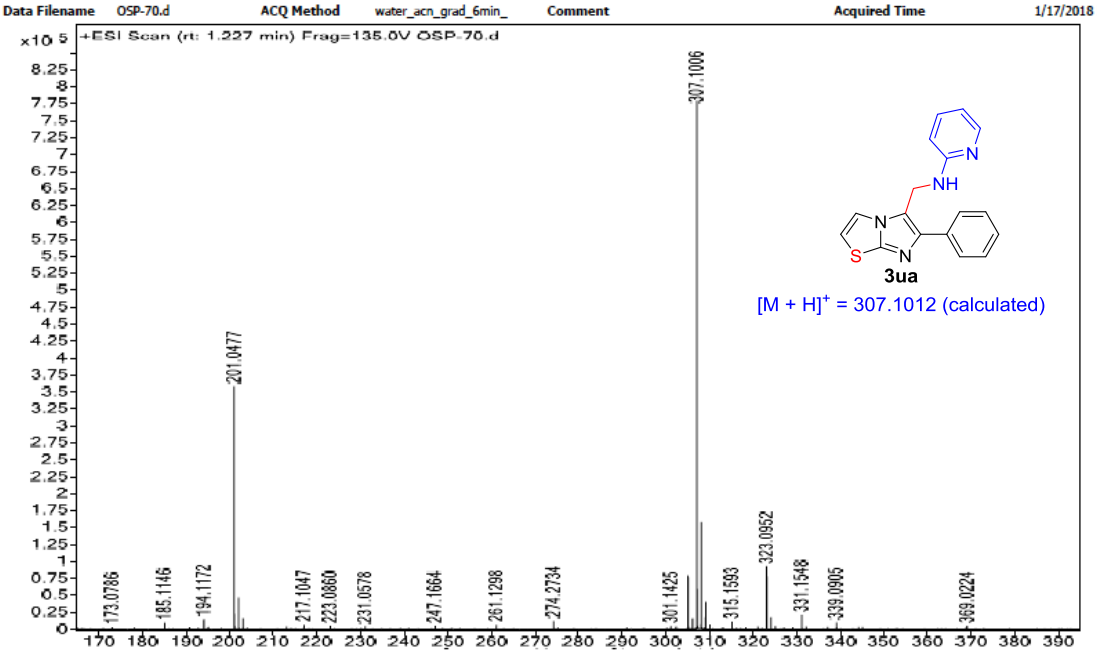
158.12 149.25 147.90 145.35 137.60 134.39 128.72 127.76 127.53 119.71 118.58 113.73 112.26 108.47 77.48 77.16 76.84 36.30



3ua, ^{13}C NMR,
100 MHz, $CDCl_3$

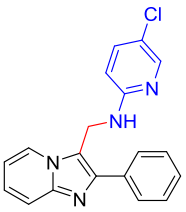


Sample Name	OSP-70	**Position**	P2-D8	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-70.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	1/17/2018 5:33:47 PM

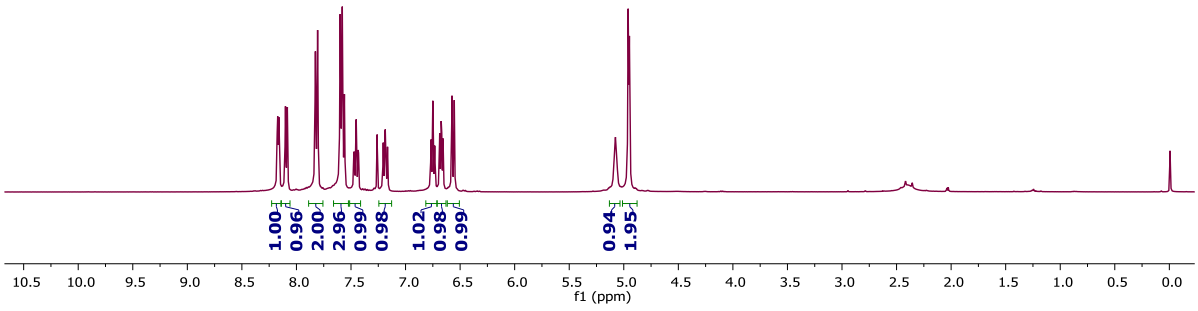


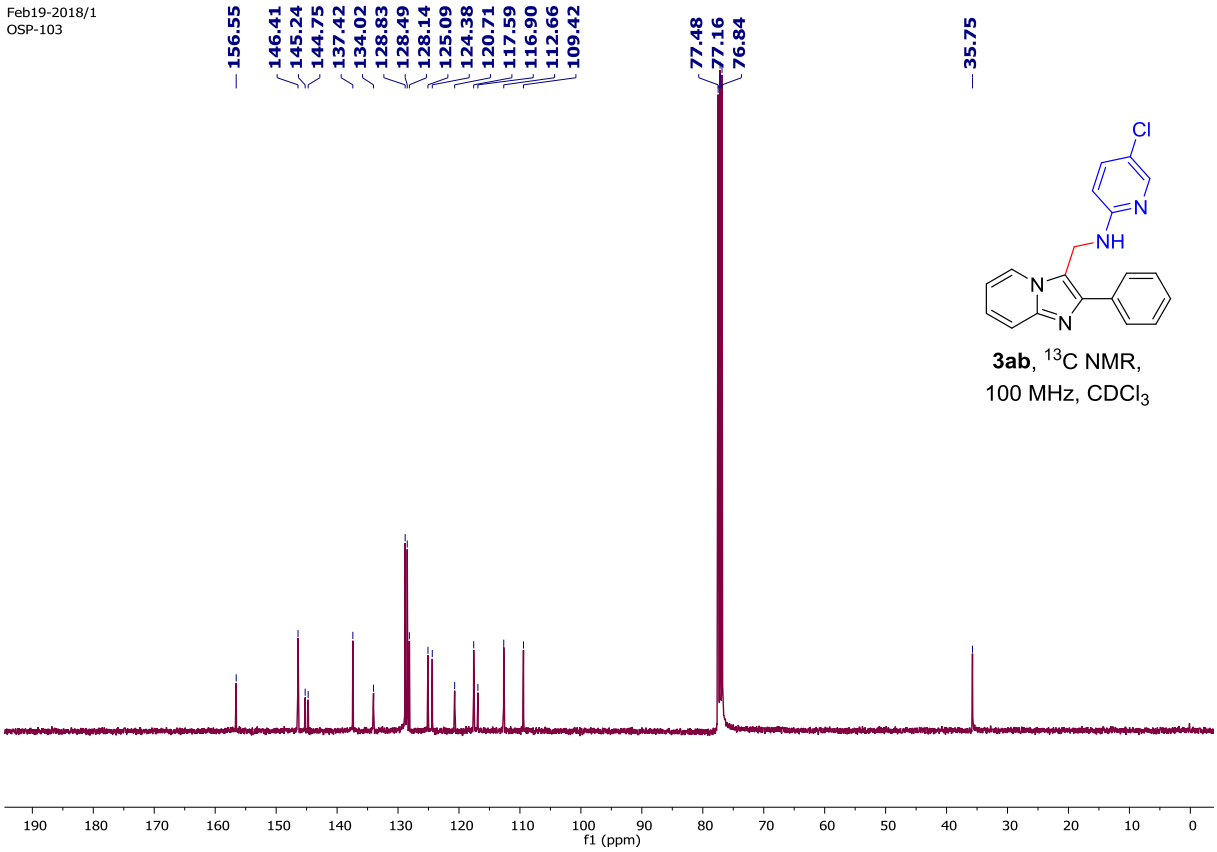
3ua

$[M + H]^+$ = 307.1012 (calculated)



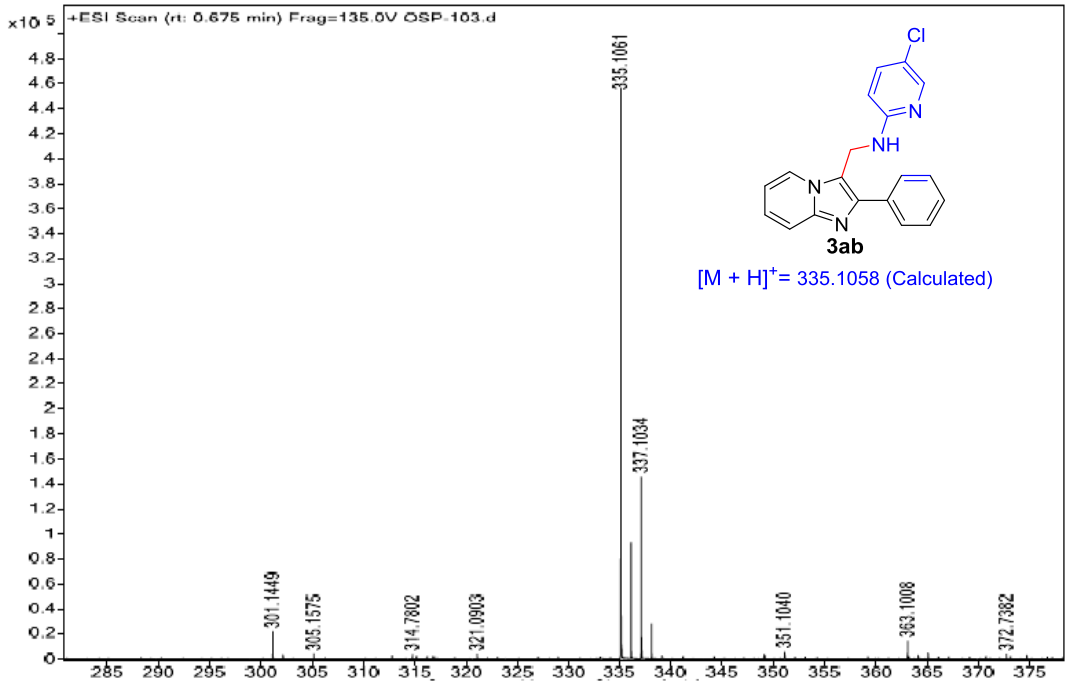
3ab, ^{1}H NMR,
400 MHz, $CDCl_3$





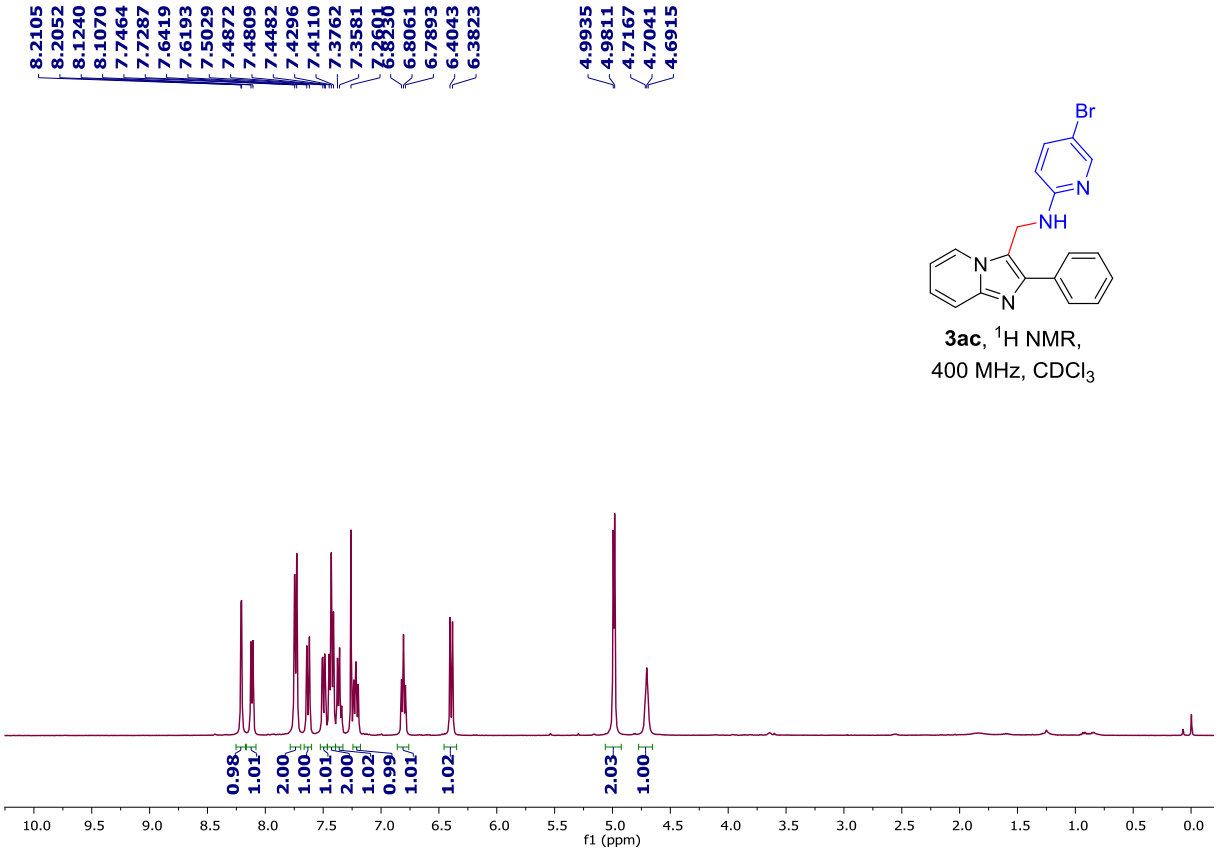
3ab, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-103	**Position**	P2-D8	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-103.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	4/12/2018 1:27:06 PM

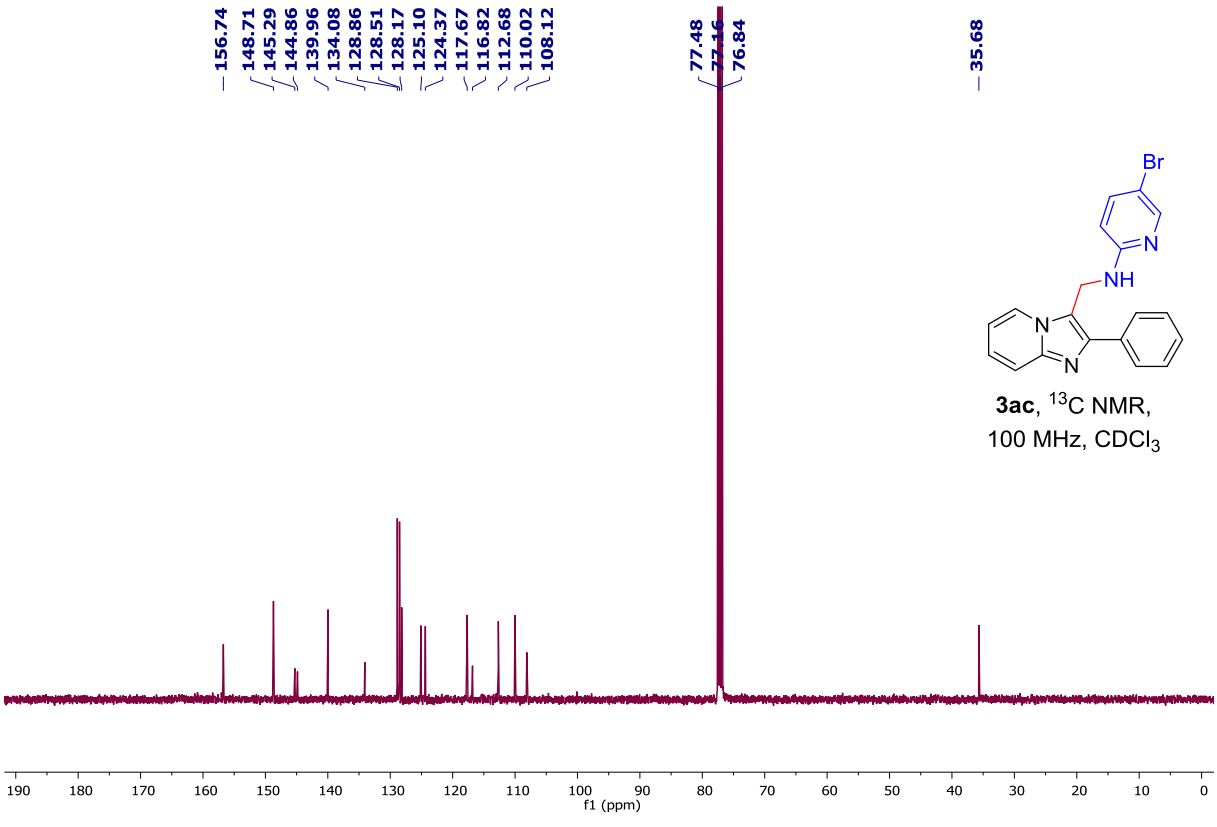


3ab

$[M + H]^+$= 335.1058 (Calculated)

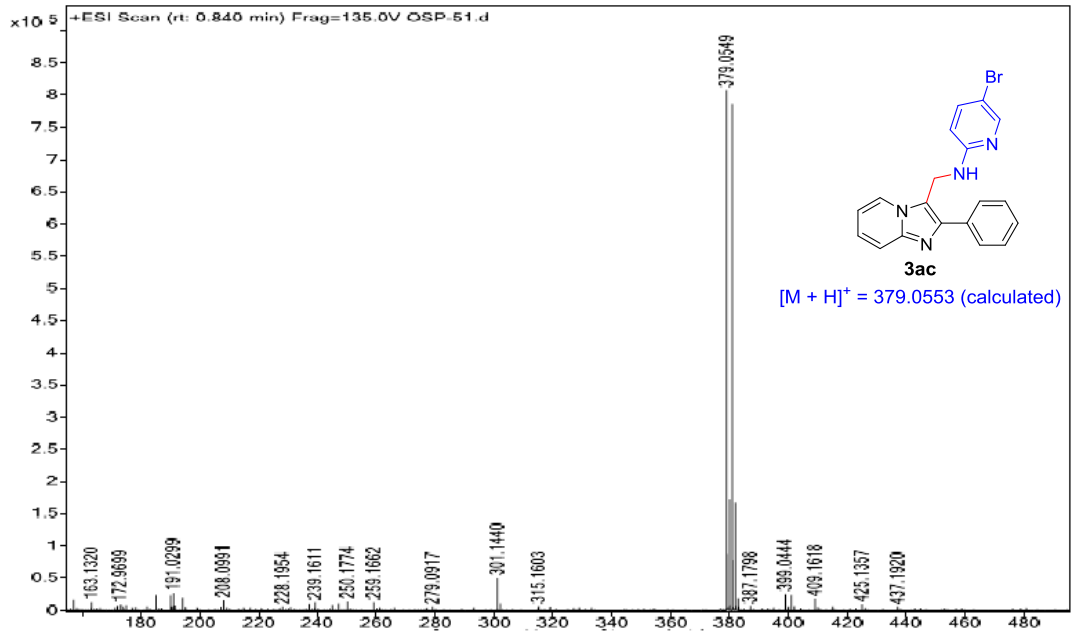


3ac, ^{1}H NMR,
400 MHz, $CDCl_3$



3ac, ^{13}C NMR,
100 MHz, $CDCl_3$

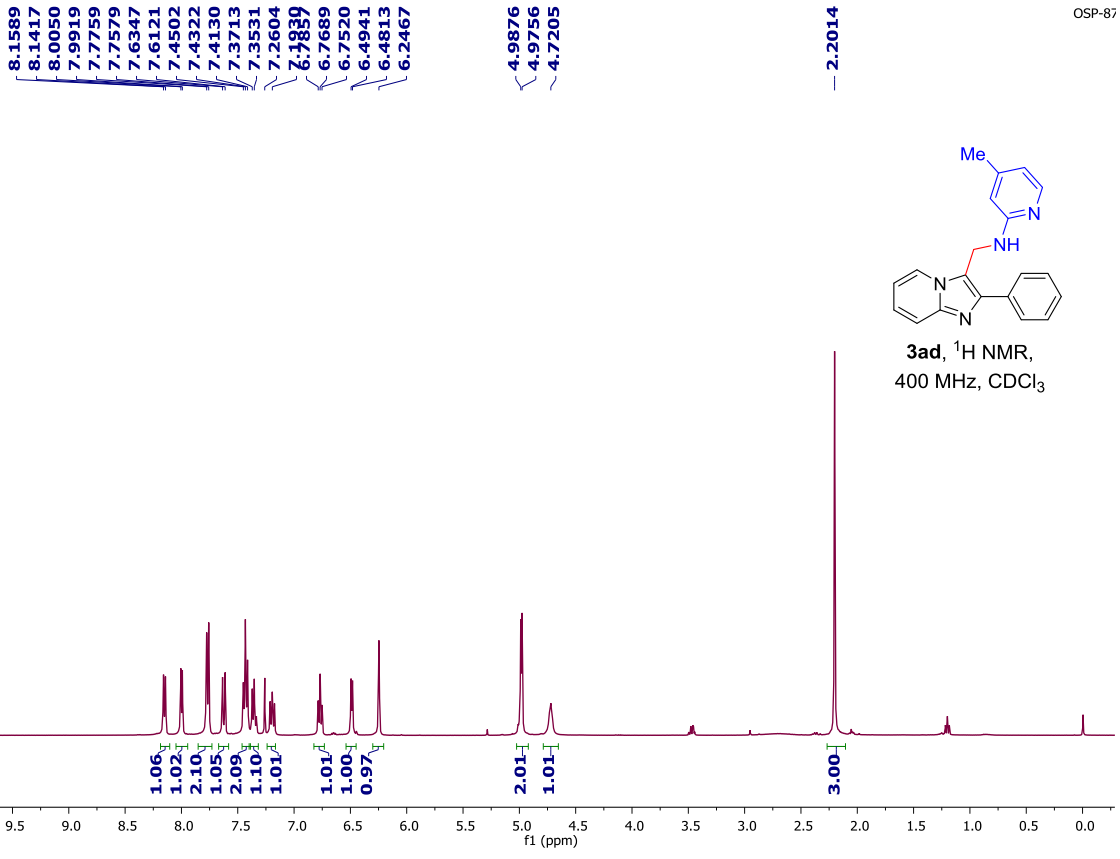
Sample Name	OSP-51	**Position**	P2-E8	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-51.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	12/8/2017 1:54:29 PM



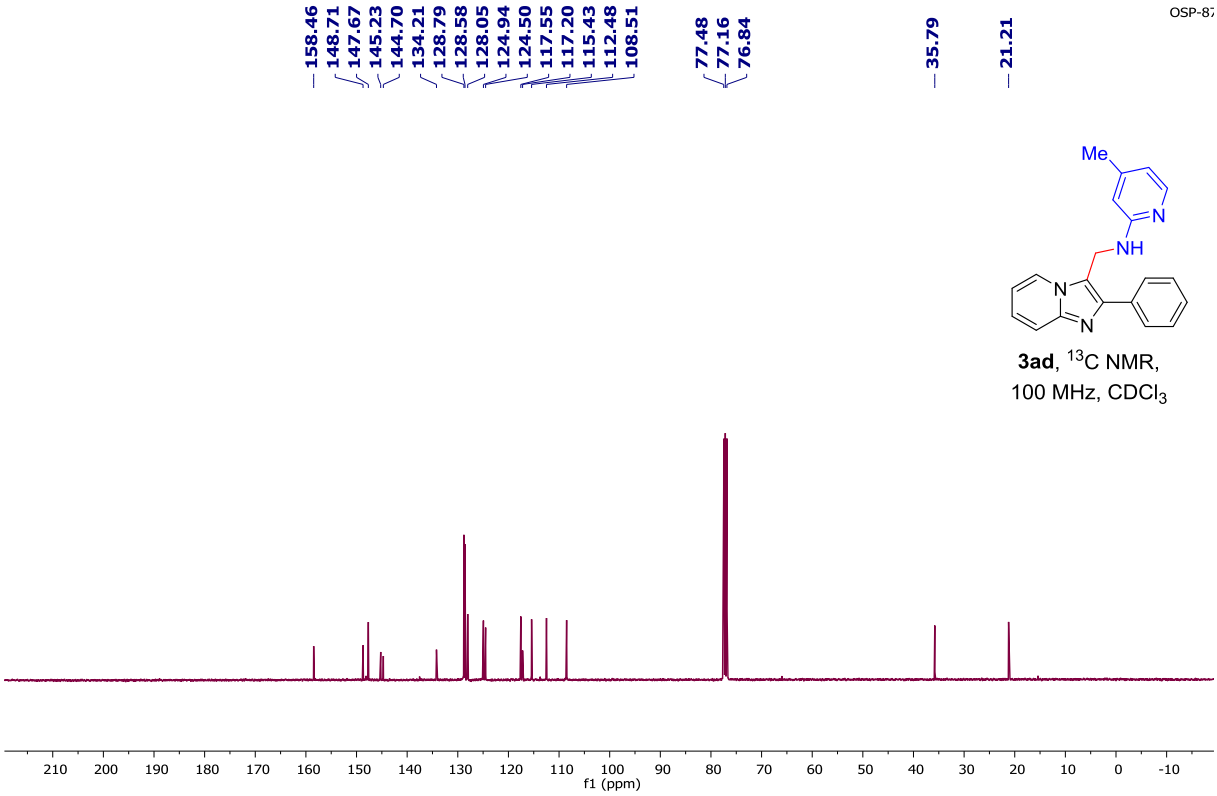
3ac

$[M + H]^+$ = 379.0553 (calculated)

OSP-87

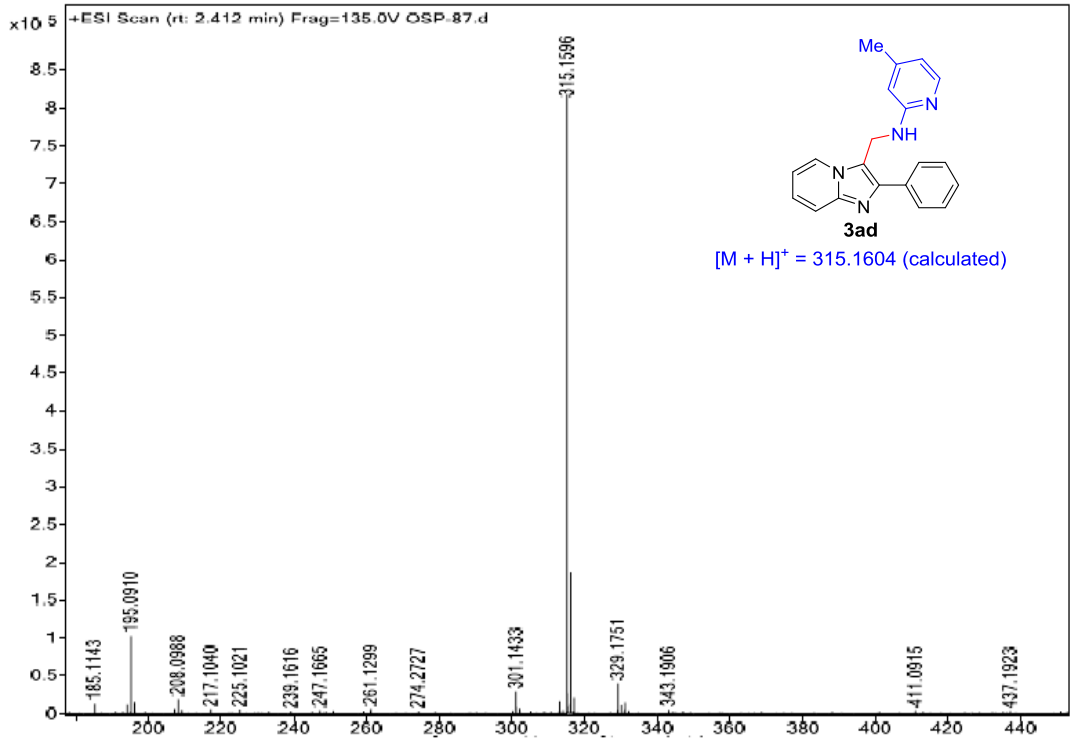


3ad, ^{1}H NMR, 400 MHz, $CDCl_3$



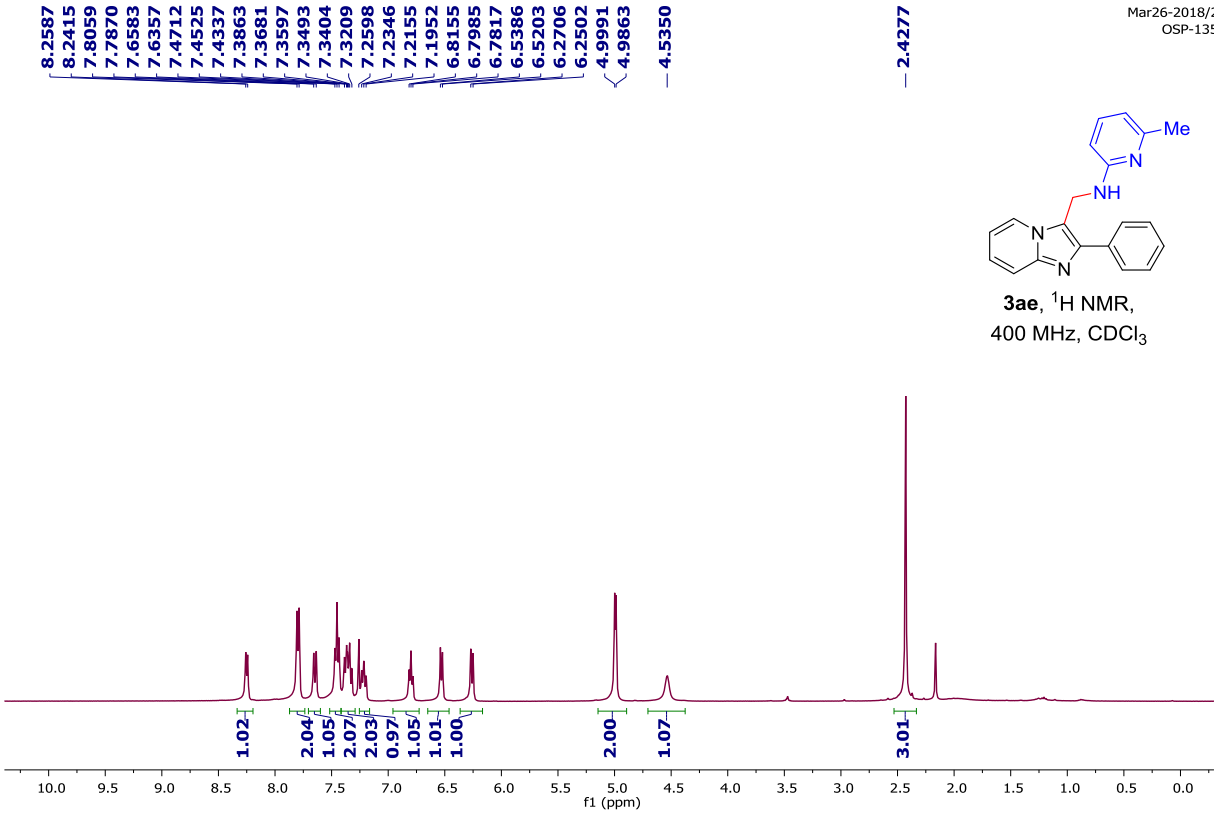
3ad, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-87	**Position**	P2-D6	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-87.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	1/17/2018 5:05:31 PM

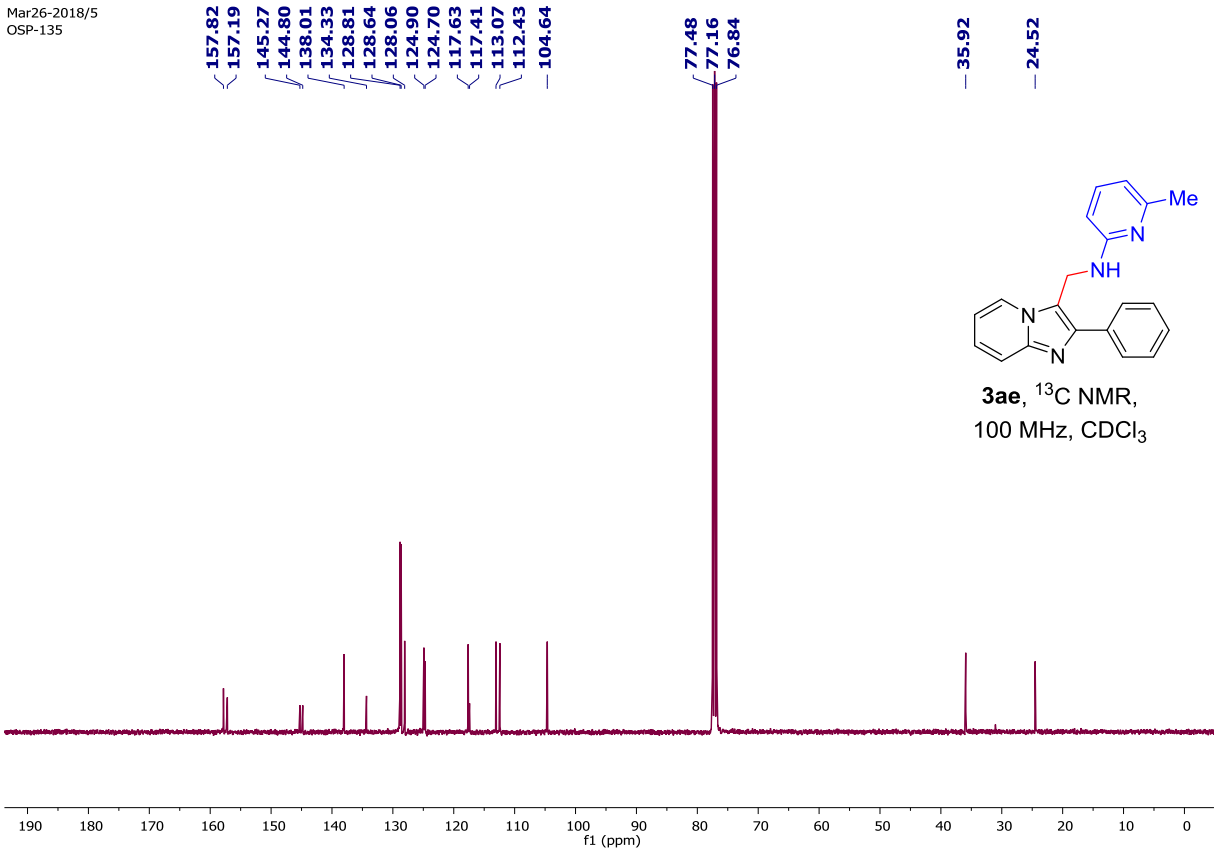


3ad

$[M + H]^+$ = 315.1604 (calculated)

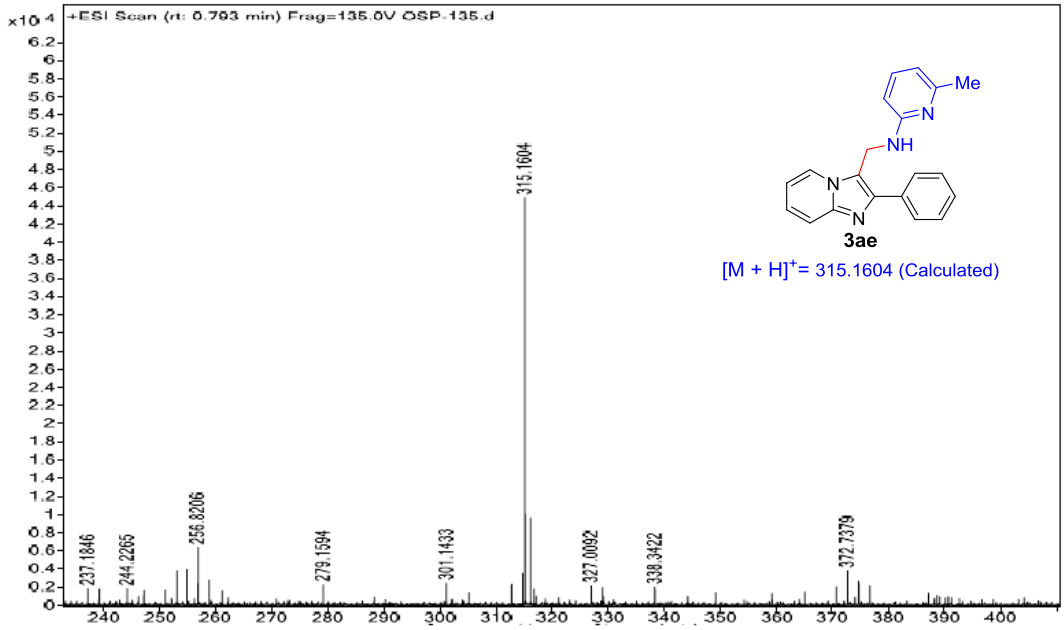


3ae, ^{1}H NMR,
400 MHz, $CDCl_3$



3ae, ^{13}C NMR,
100 MHz, $CDCl_3$

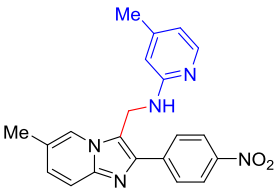
Sample Name	OSP-135	**Position**	P1-B4	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-135.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	4/3/2018 12:09:06 PM



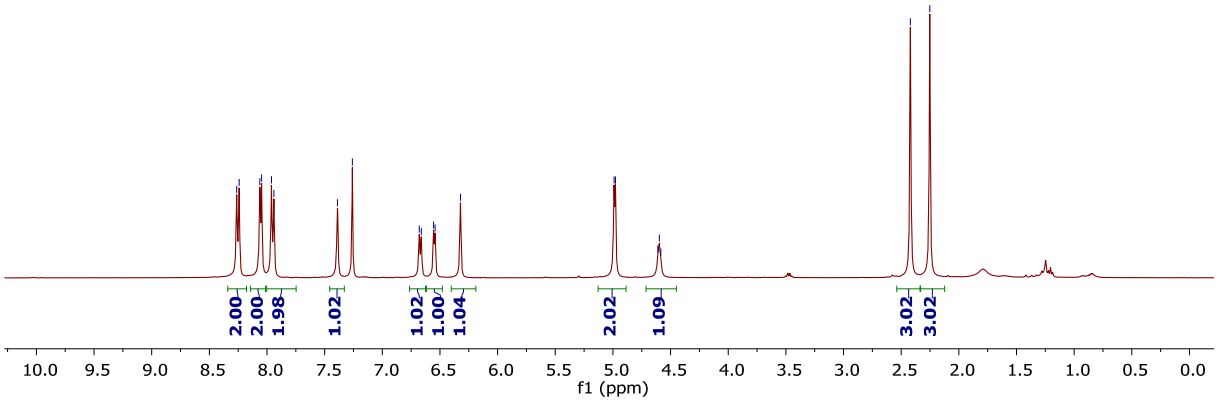
3ae

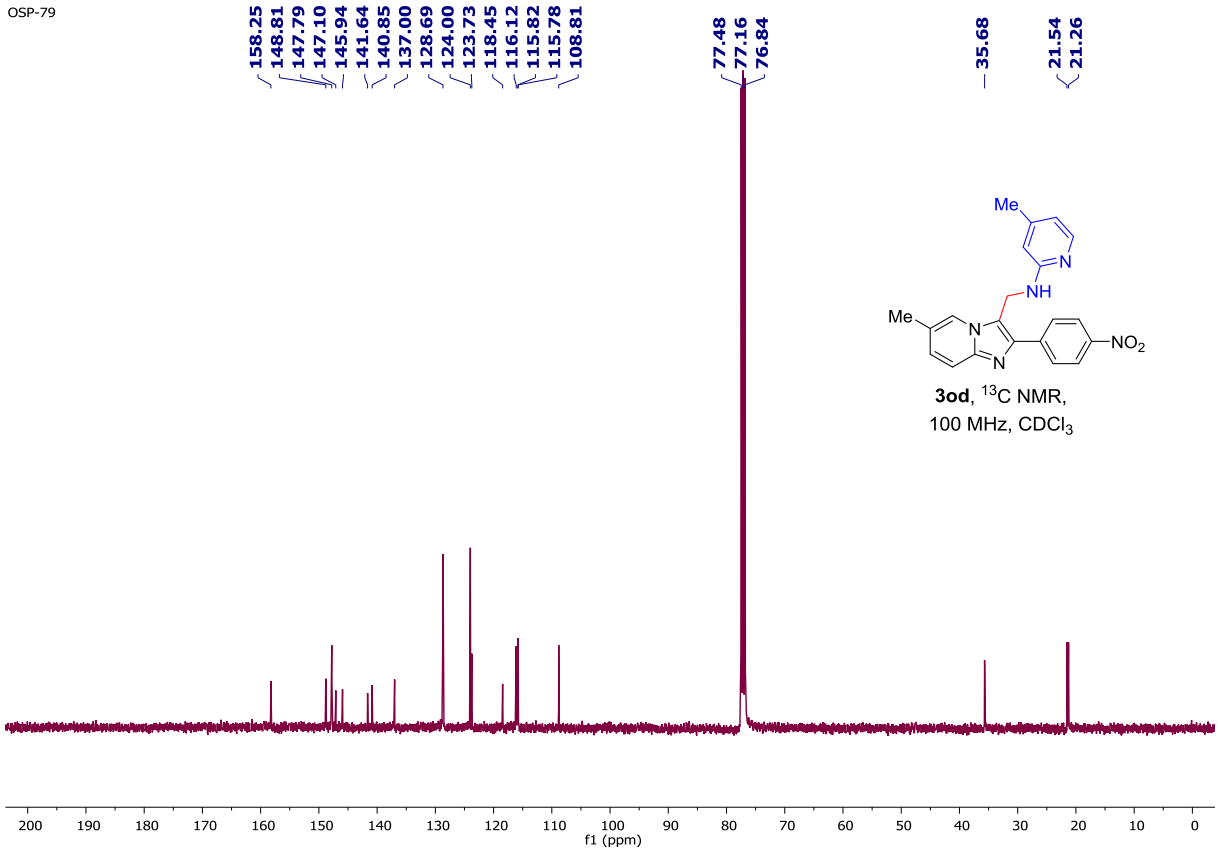
$[M + H]^+$= 315.1604 (Calculated)

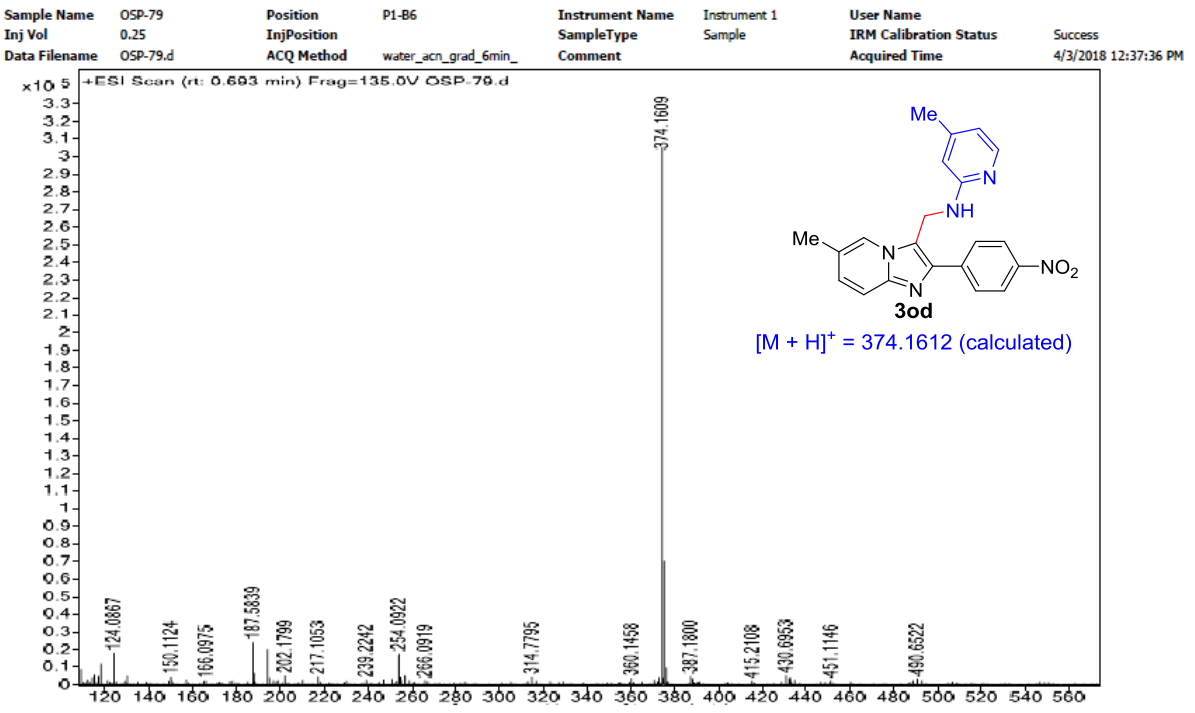
Oct17-2018/3
OSP-79



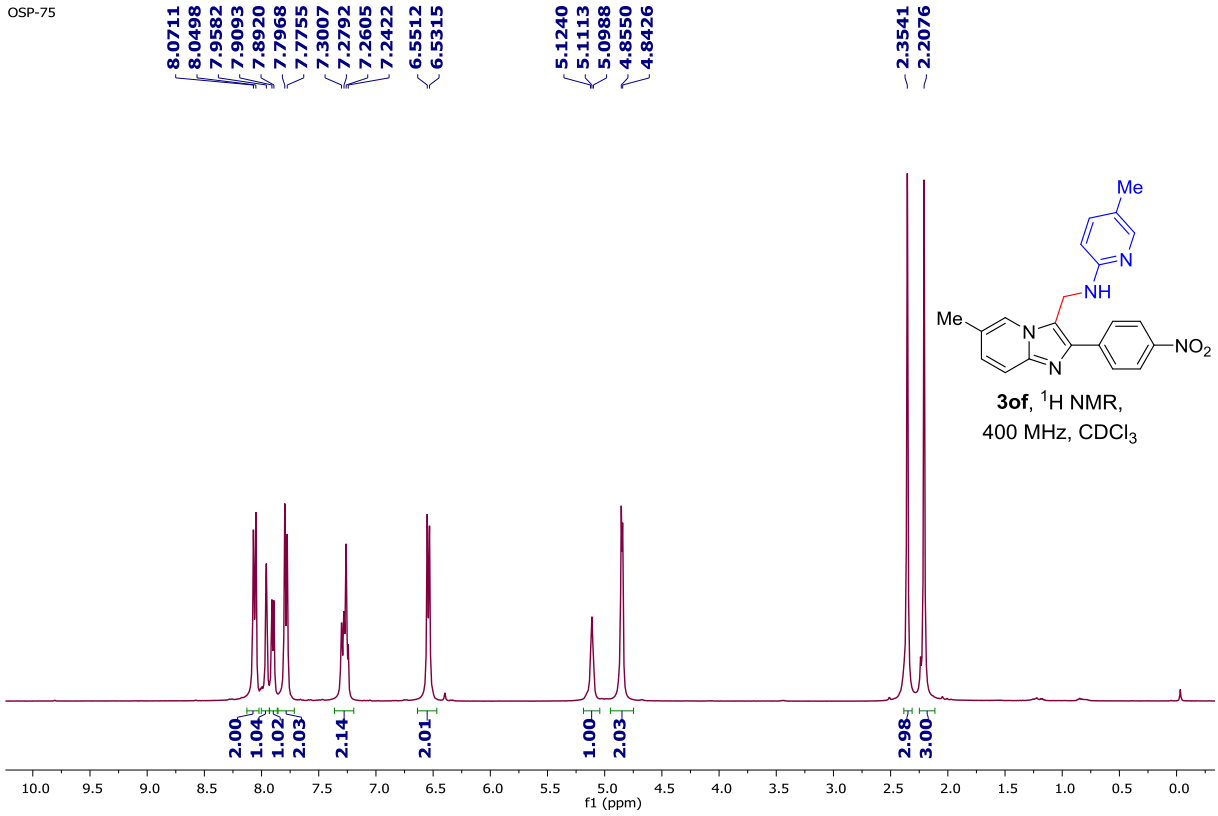
3od, ^{1}H NMR,
400 MHz, $CDCl_3$




OSP-79
158.25
148.81
147.79
147.10
145.94
141.64
140.85
137.00
128.69
124.00
123.73
118.45
116.12
115.82
115.78
108.81
77.48
77.16
76.84
35.68
21.54
21.26
Me
N
NH
Me
N
N
NO2
3od, 13C NMR,
100 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

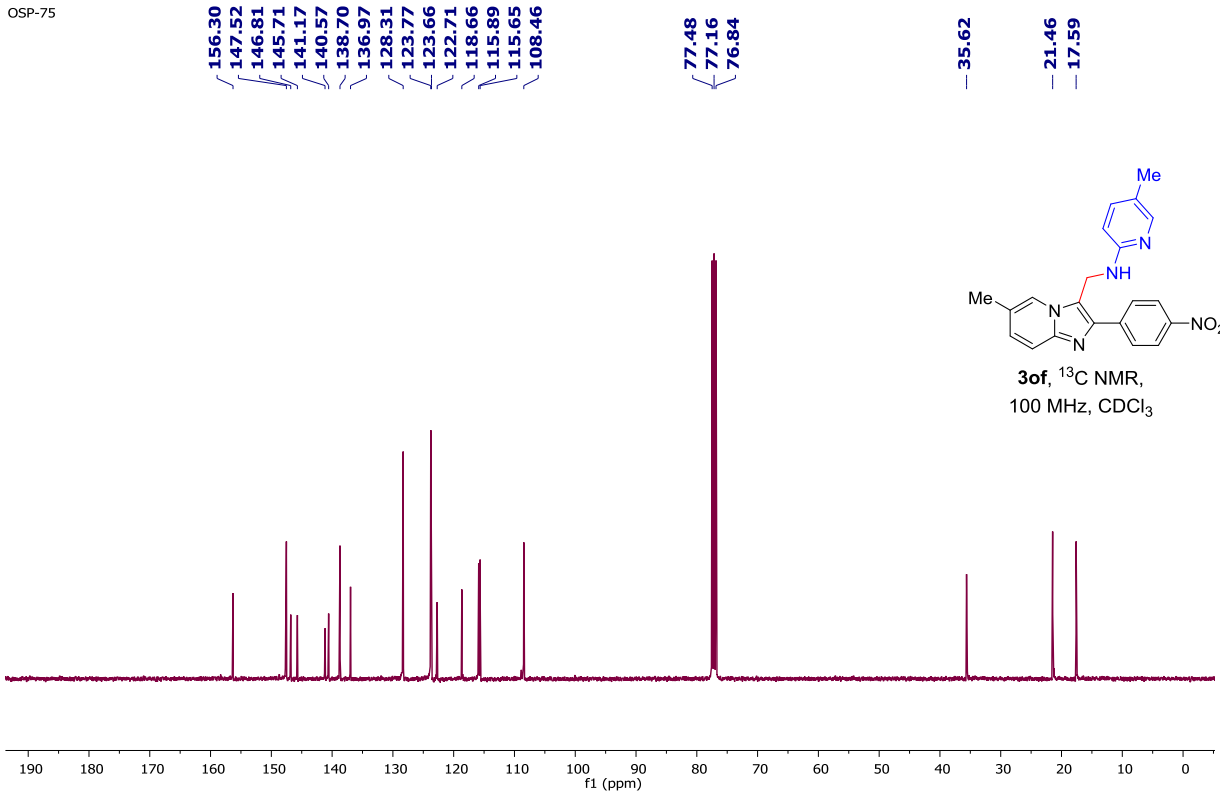

Sample Name OSP-79
Position P1-B6
Instrument Name Instrument 1
User Name
Inj Vol 0.25
InjPosition
SampleType Sample
IRM Calibration Status Success
Data Filename OSP-79.d
ACQ Method water_acn_grad_6min_
Comment
Acquired Time 4/3/2018 12:37:36 PM
x10 5
+ESI Scan (rt: 0.693 min) Frag=135.0V OSP-79.d
374.1609
124.0867
150.1124
166.0975
187.5839
202.1799
217.1053
239.2242
254.0922
266.0919
314.7795
360.1458
387.1800
415.2108
430.6953
451.1146
490.6322
120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540 560
Me
N
NH
Me
N
N
NO2
3od
[M + H]+ = 374.1612 (calculated)

OSP-75



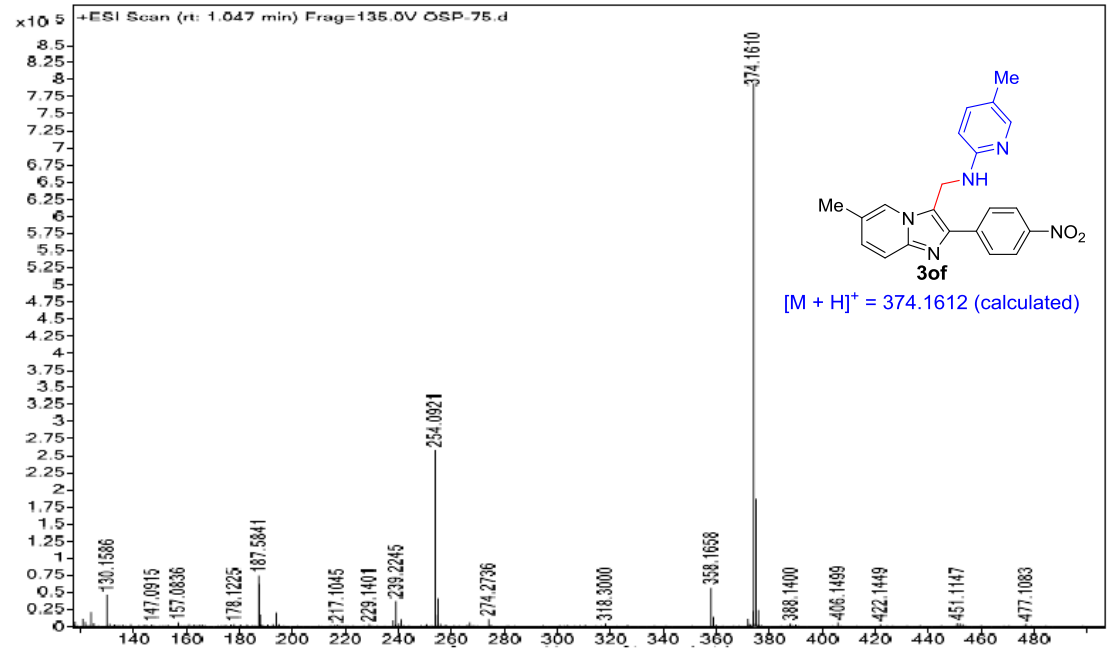
3of, ^{1}H NMR,
400 MHz, $CDCl_3$

OSP-75

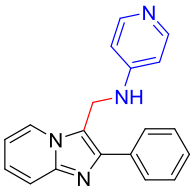


3of, ^{13}C NMR,
100 MHz, $CDCl_3$

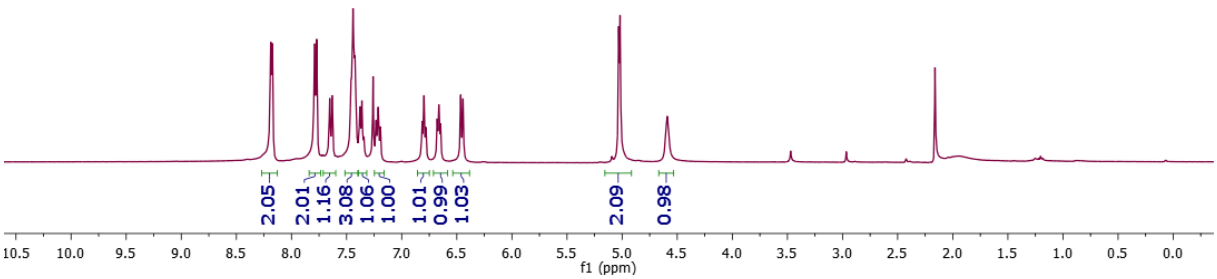
Sample Name	OSP-75	**Position**	P1-B7	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-75.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	1/24/2018 5:08:58 PM

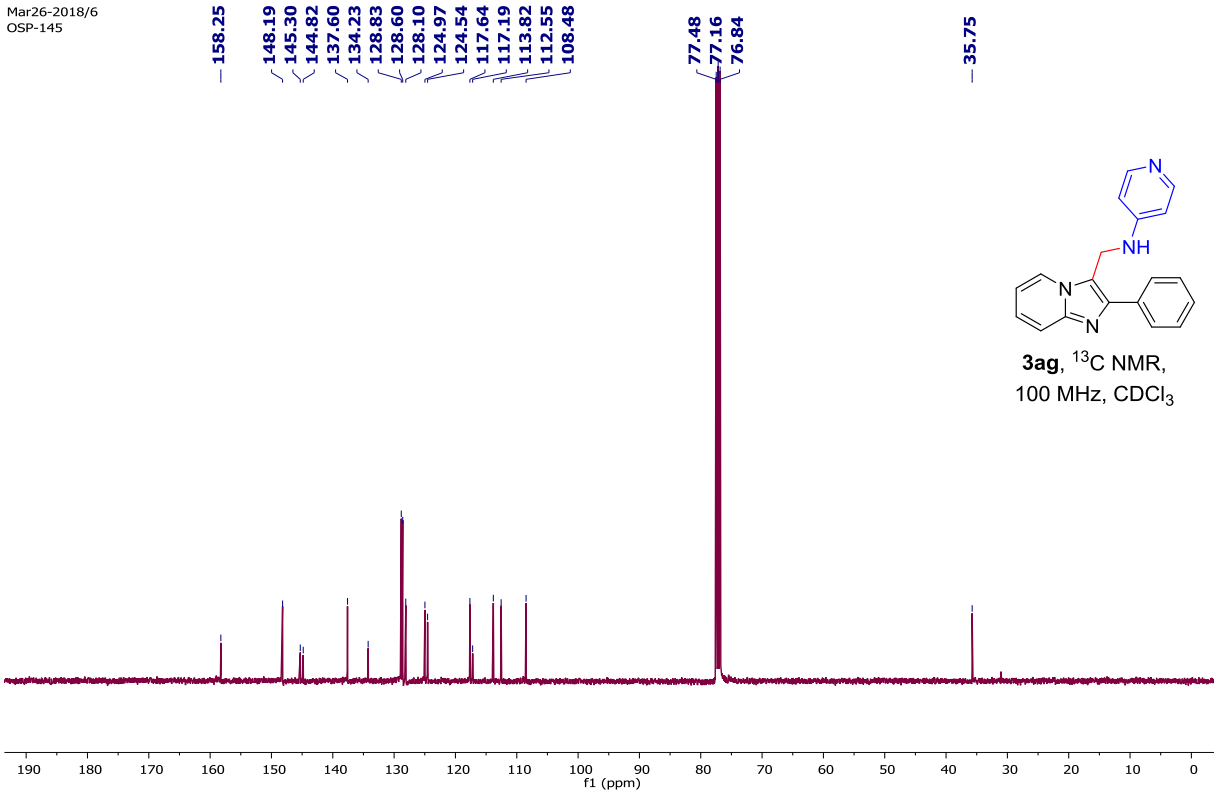


Mar26-2018/4
OSP-145



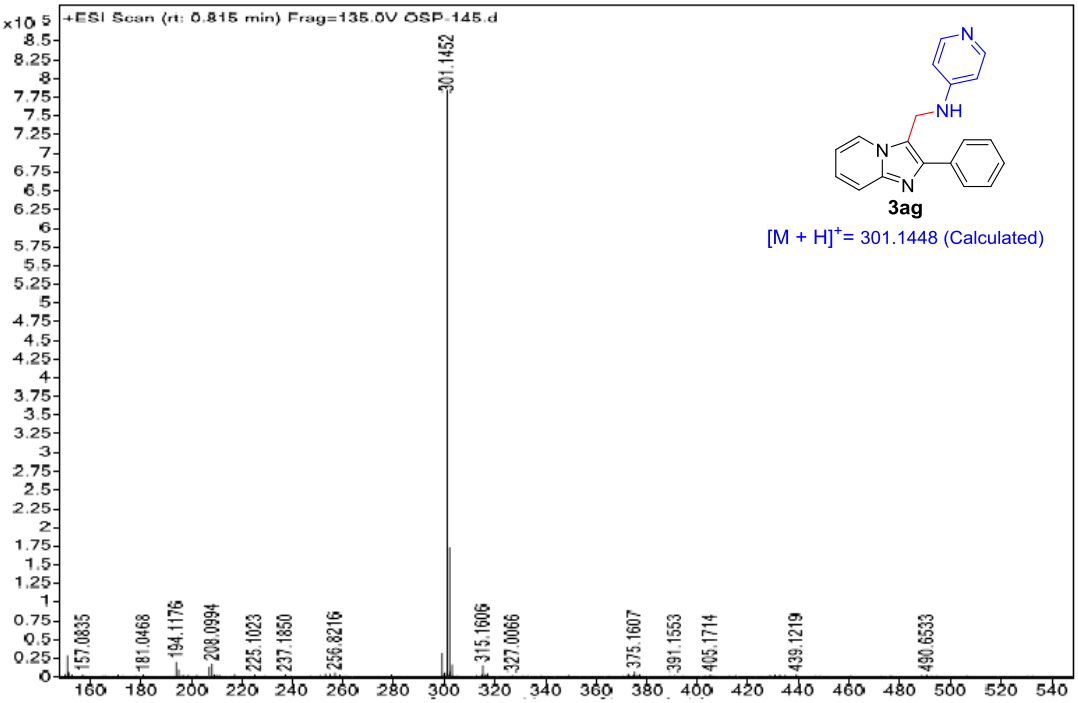
3ag, ^{1}H NMR,
400 MHz, $CDCl_3$





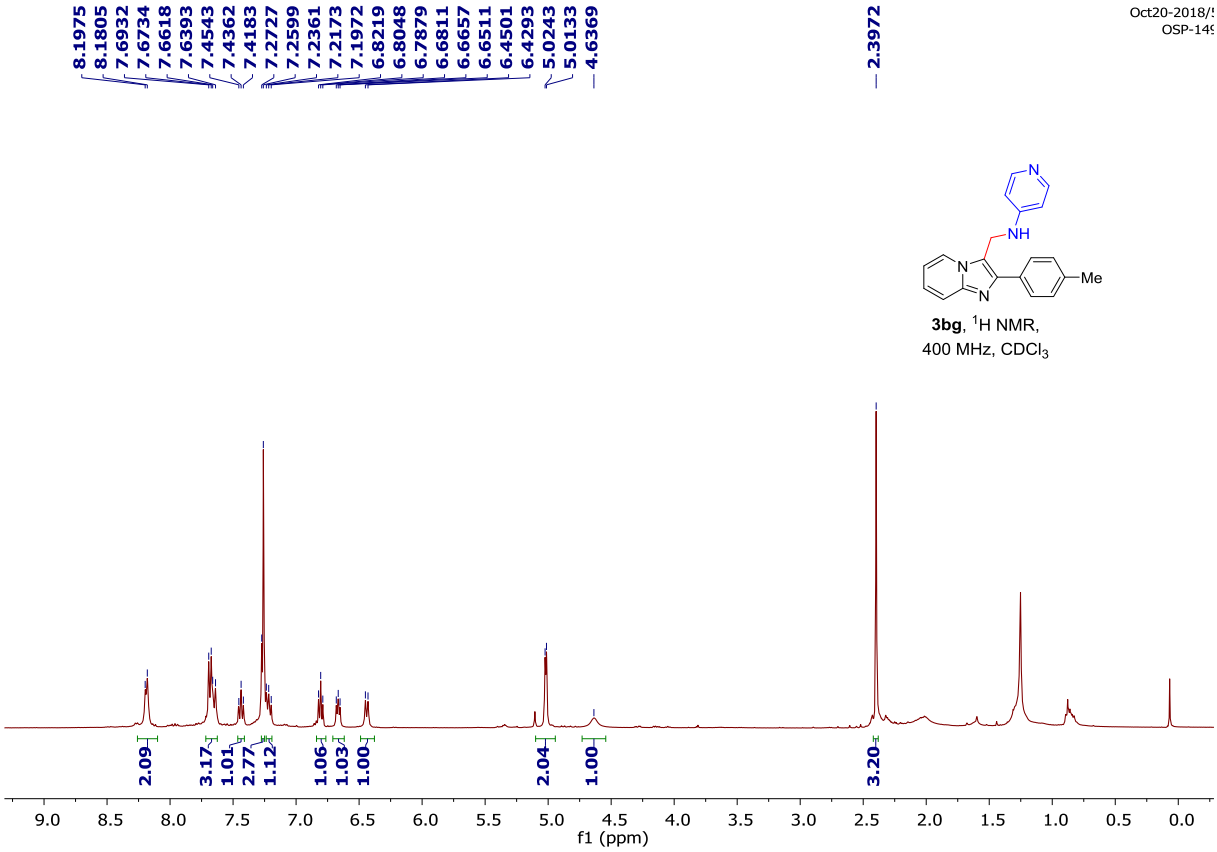
3ag, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-145	**Position**	P1-B3	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-145.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	4/3/2018 11:54:56 AM

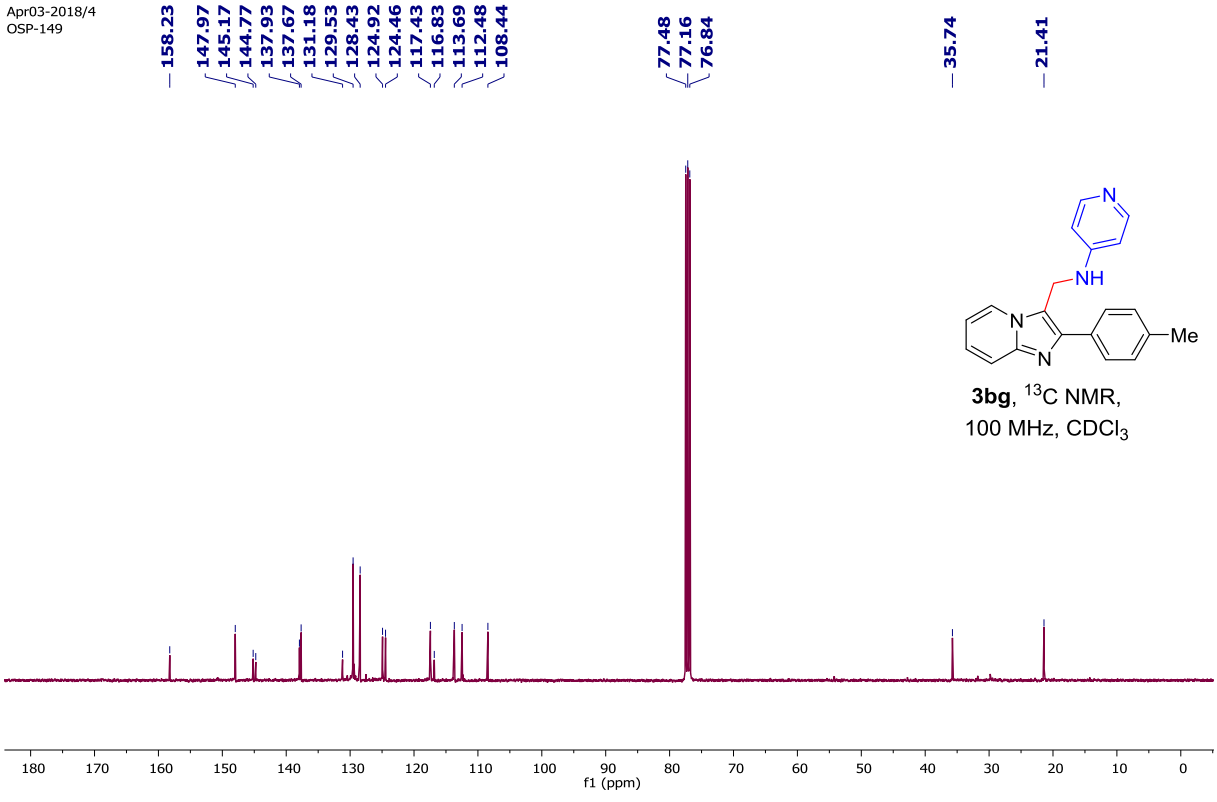


3ag

$[M + H]^+$= 301.1448 (Calculated)

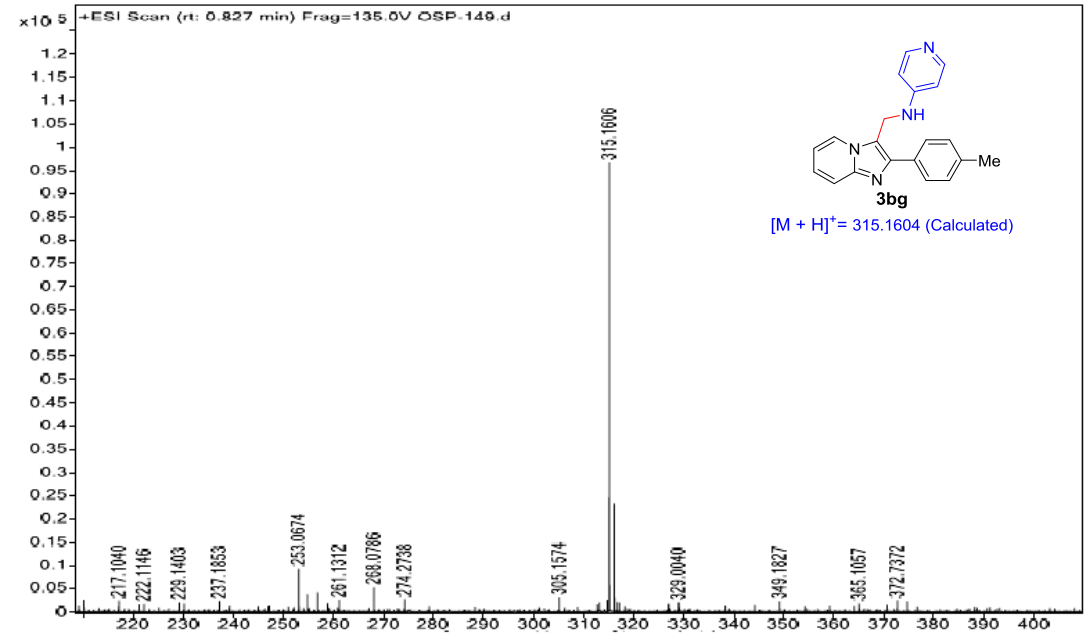


3bg, ^{1}H NMR,
400 MHz, $CDCl_3$



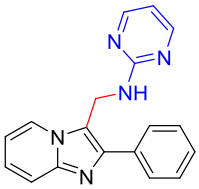
3bg, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-149	Position	P1-B2	Instrument Name	Instrument 1	User Name	
Inj Vol	0.25	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	OSP-149.d	ACQ Method	water_acn_grad_6min_	Comment		Acquired Time	4/3/2018 11:40:42 AM

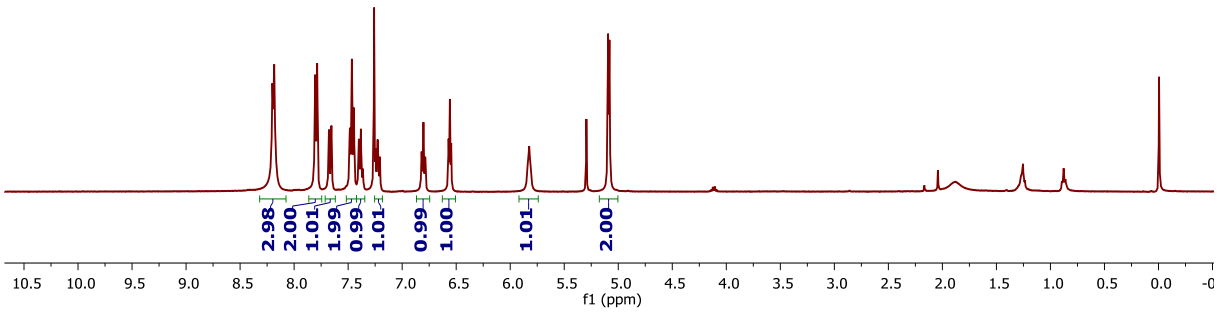


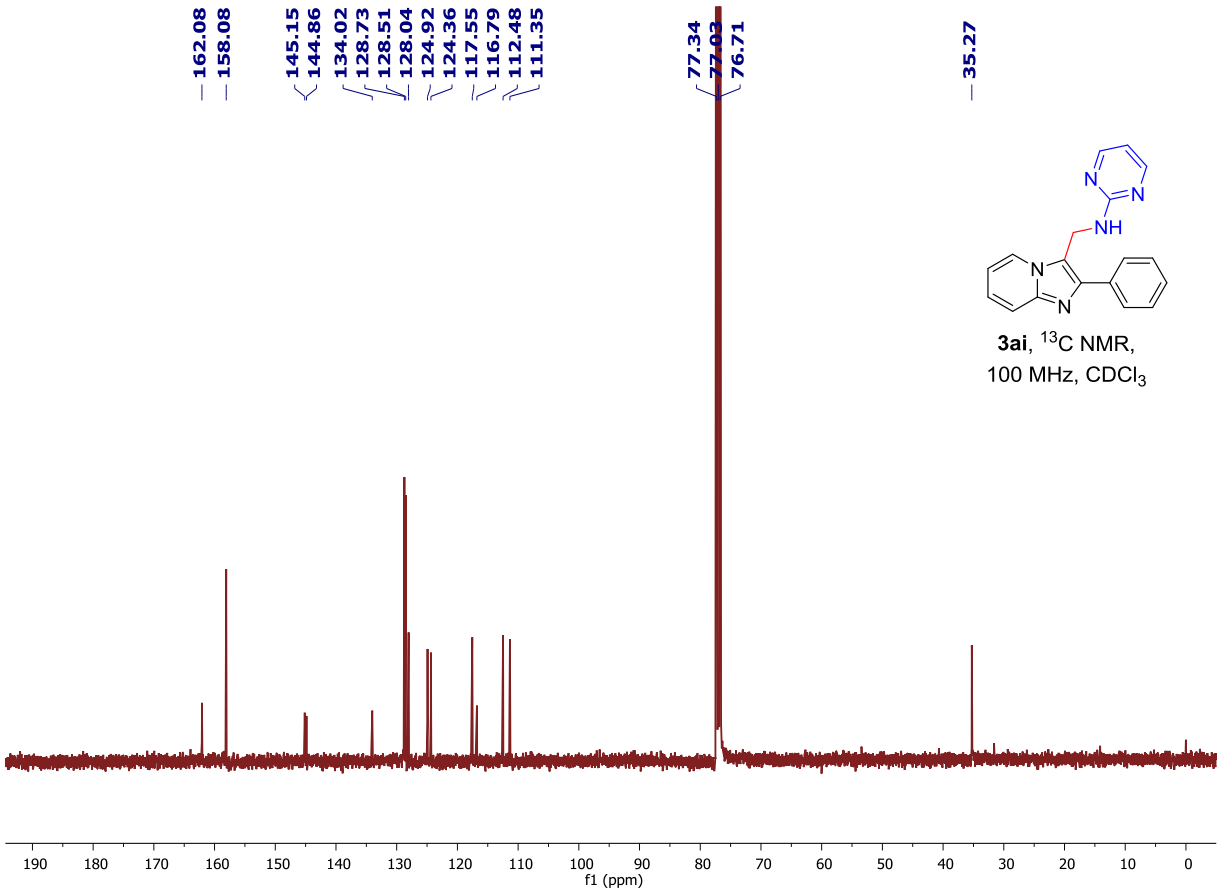
3bg

$[M + H]^+$= 315.1604 (Calculated)



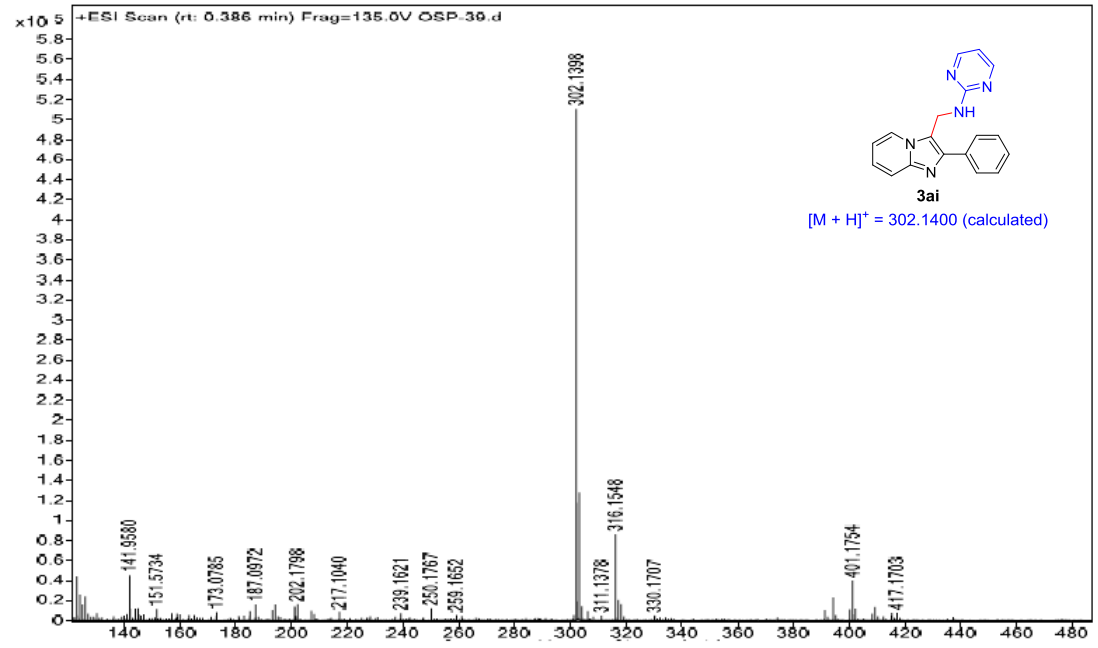
3ai, ^{1}H NMR,
400 MHz, $CDCl_3$





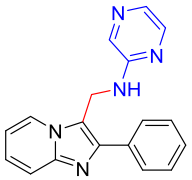
3ai, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-39	**Position**	P2-F1	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-39.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	10/12/2017 5:01:51 PM

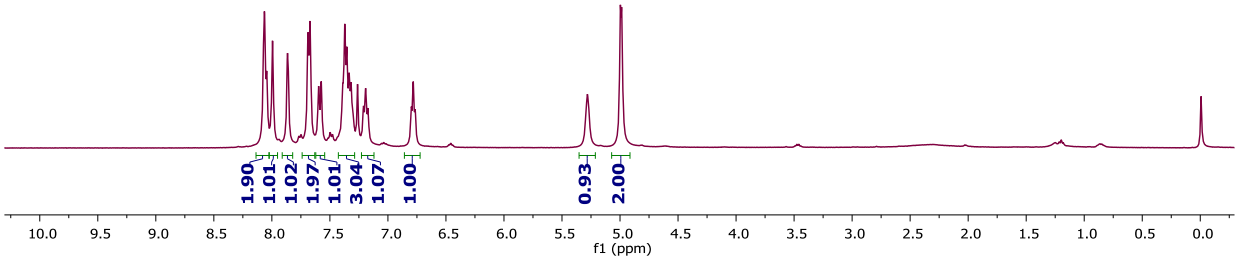


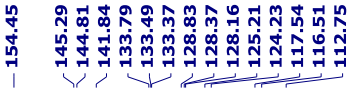
3ai

$[M + H]^+$ = 302.1400 (calculated)

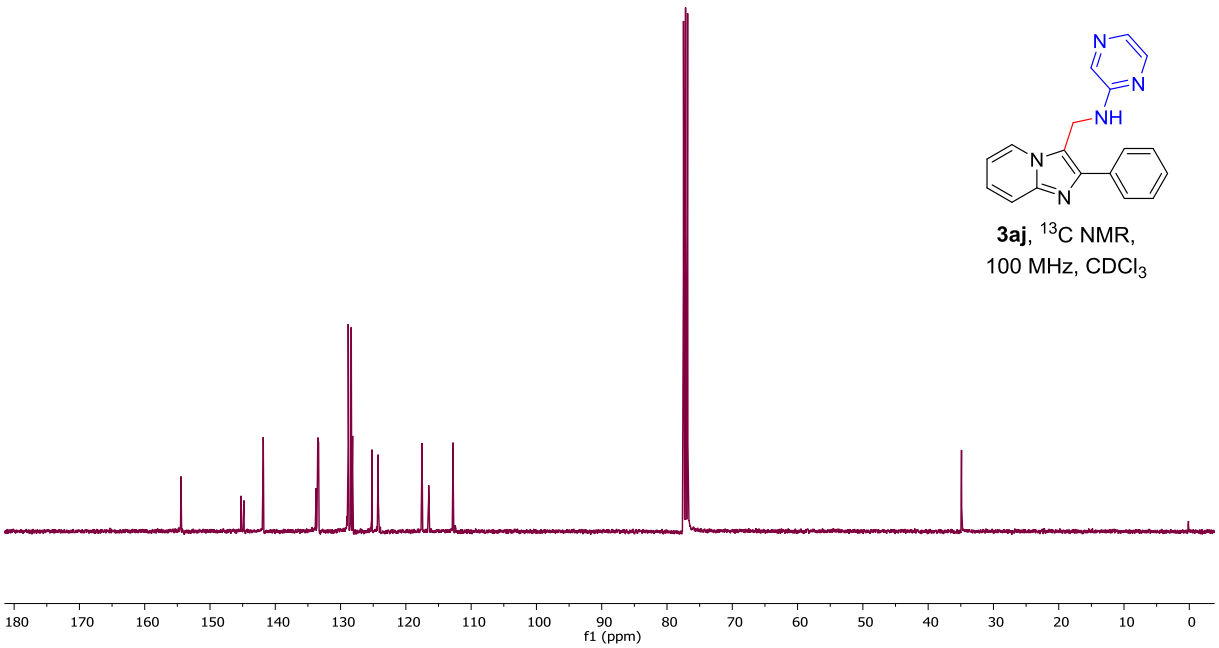


3aj, ^{1}H NMR,
400 MHz, $CDCl_3$

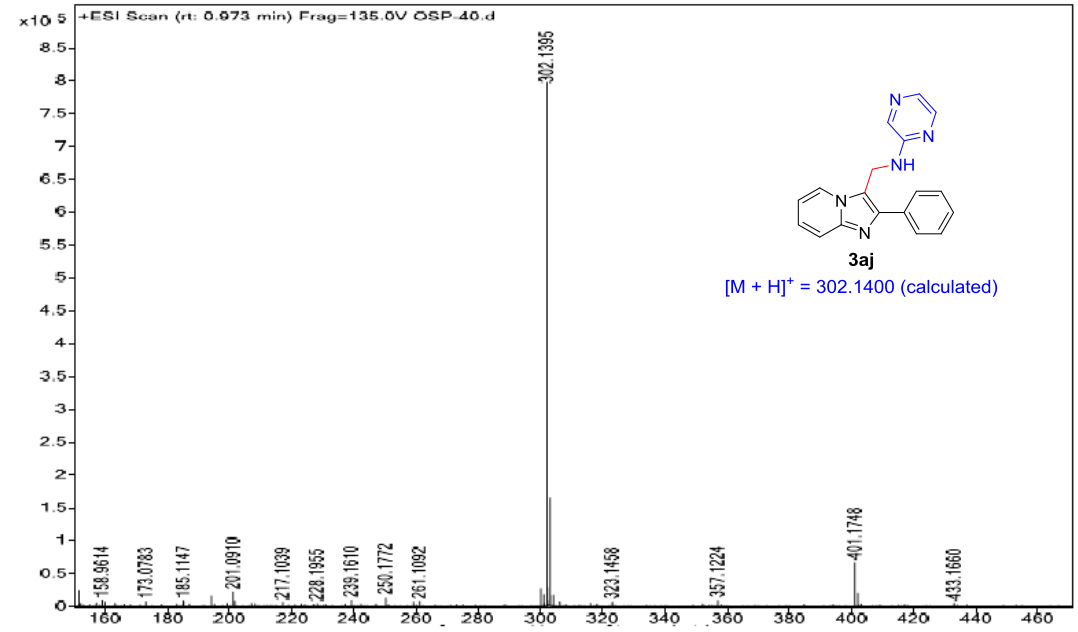




3aj, ^{13}C NMR,
100 MHz, $CDCl_3$



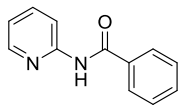
Sample Name	OSP-40	**Position**	P2-F2	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-40.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	10/12/2017 5:16:07 PM



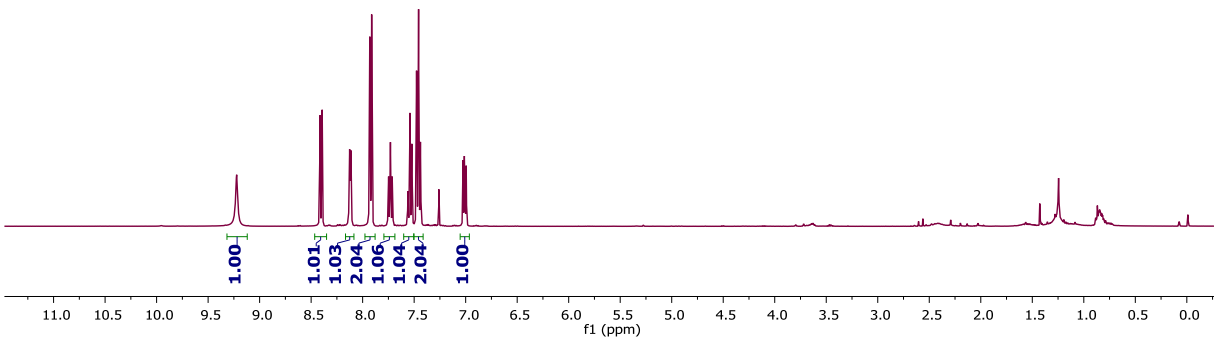
3aj

$[M + H]^+$ = 302.1400 (calculated)

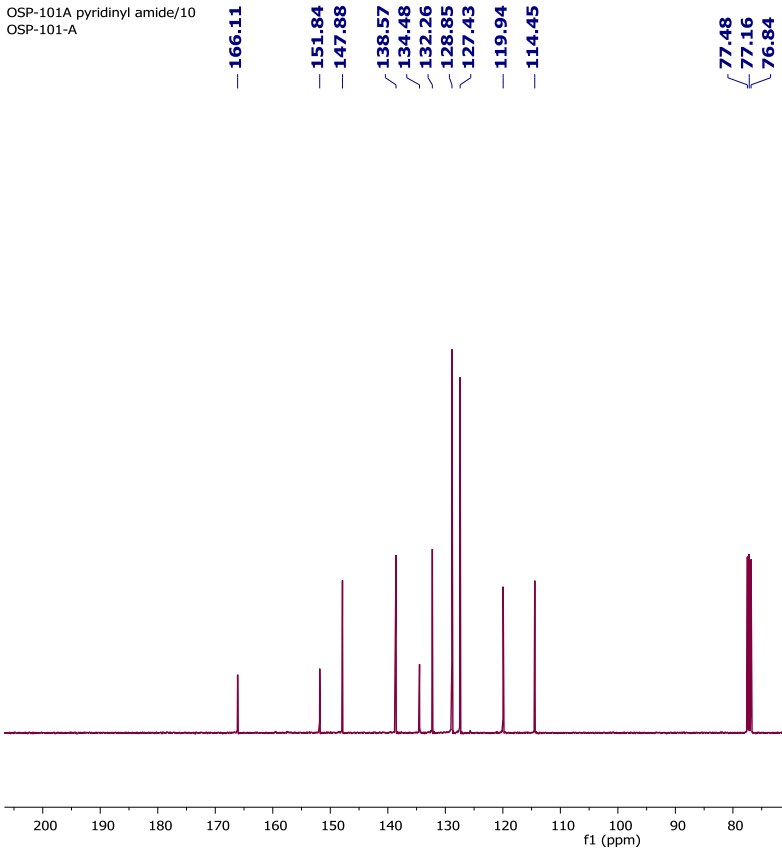
OSP-101A pyridinyl amide/2
OSP-101-A

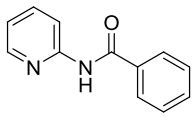


5a, ^{1}H NMR,
400 MHz, $CDCl_3$



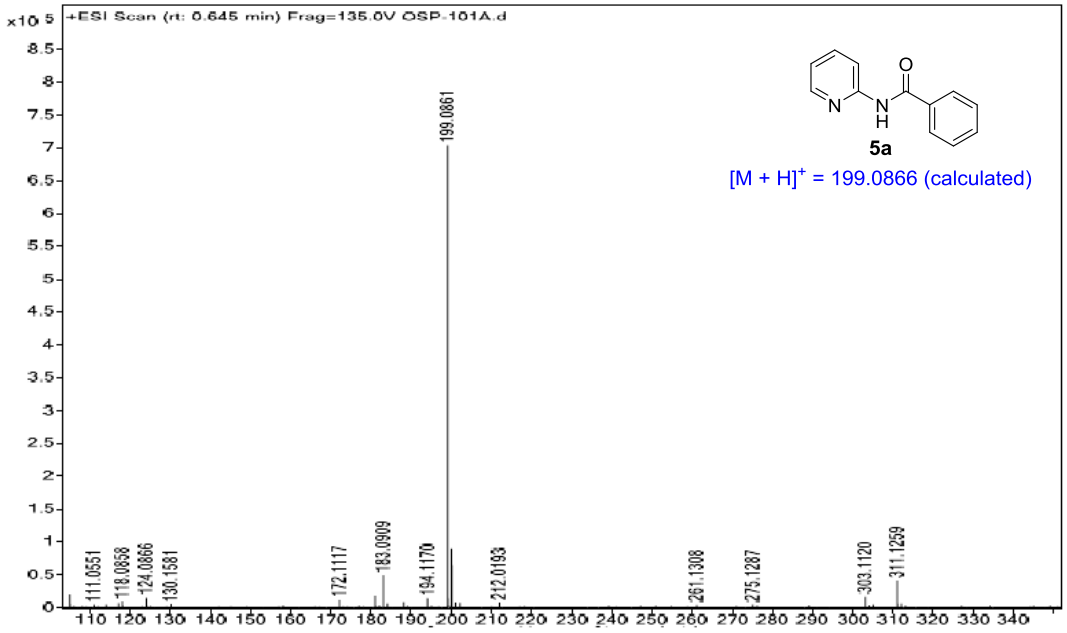
OSP-101A pyridinyl amide/10
OSP-101-A





5a, ^{13}C NMR,
100 MHz, $CDCl_3$

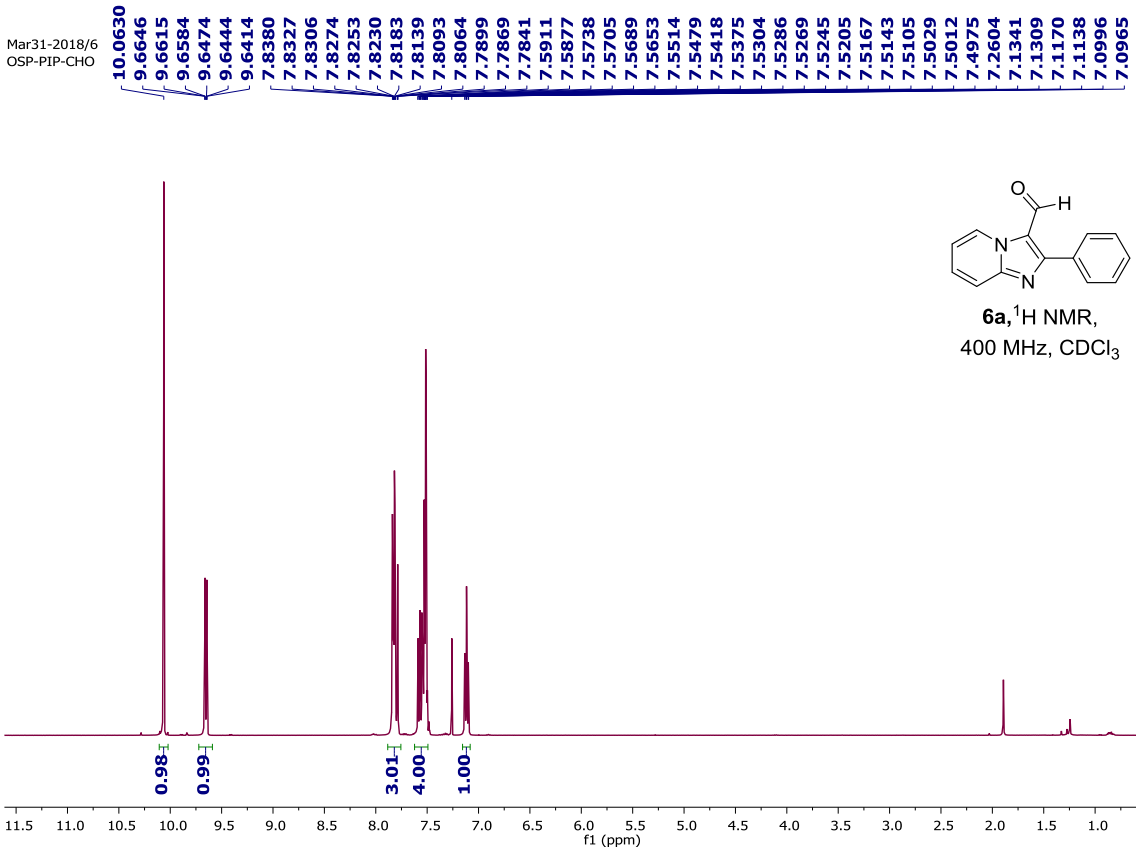
Sample Name	OSP-101A	**Position**	P2-D7	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-101A.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	4/12/2018 1:12:57 PM



5a

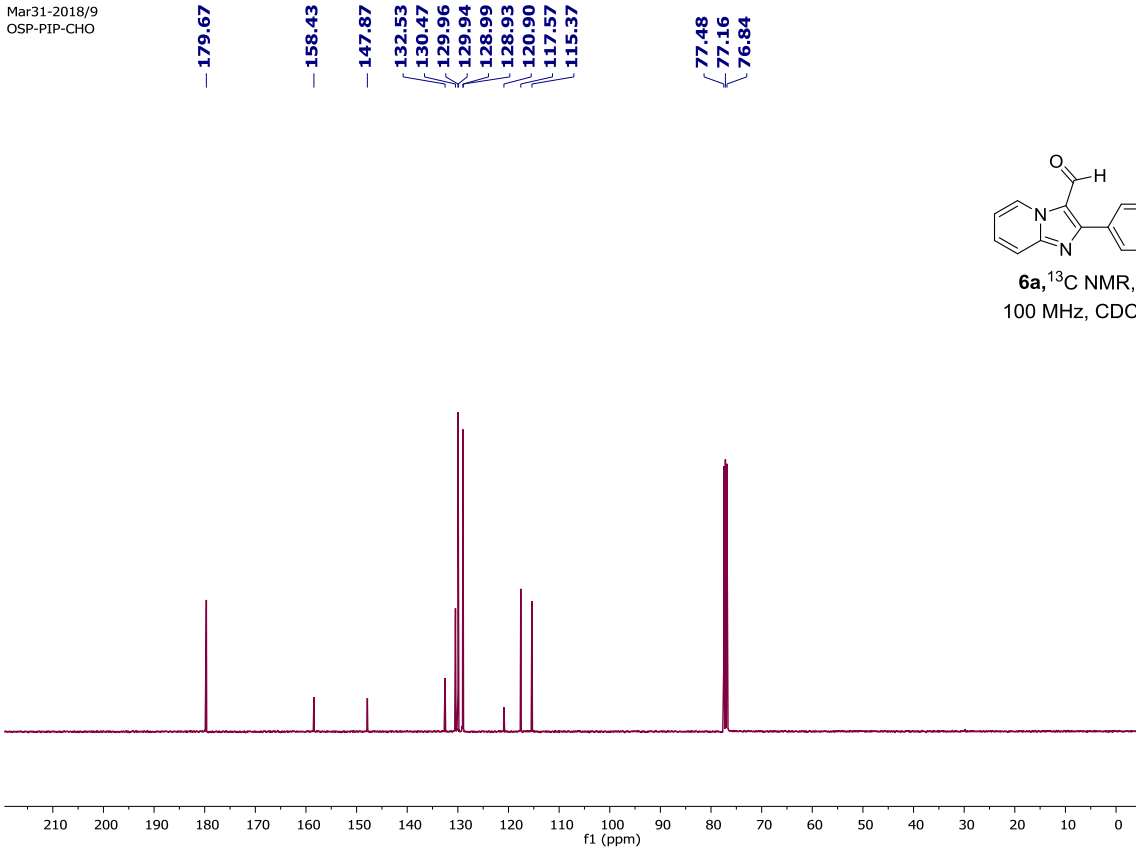
$[M + H]^+$ = 199.0866 (calculated)

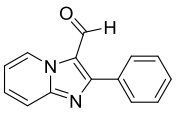
Mar31-2018/6
OSP-PIP-CHO



6a, ^{1}H NMR,
400 MHz, $CDCl_3$

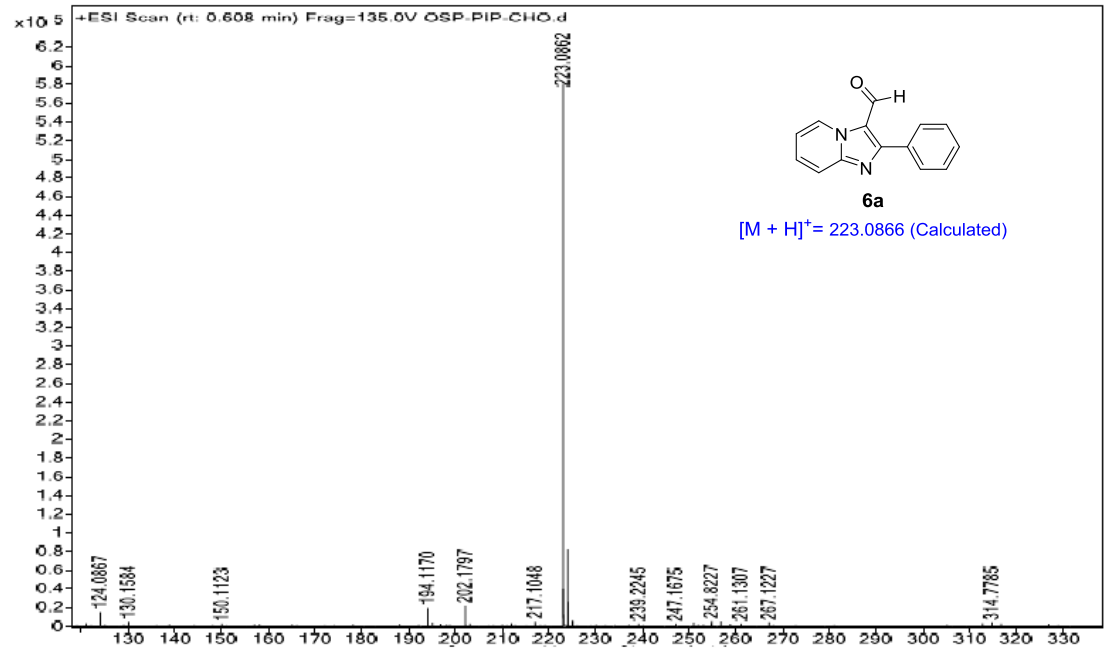
Mar31-2018/9
OSP-PIP-CHO





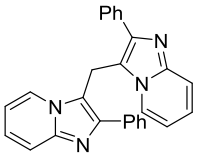
6a, ^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-PIP-CHO	**Position**	P1-B9	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-PIP-CHO.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	4/3/2018 1:20:08 PM

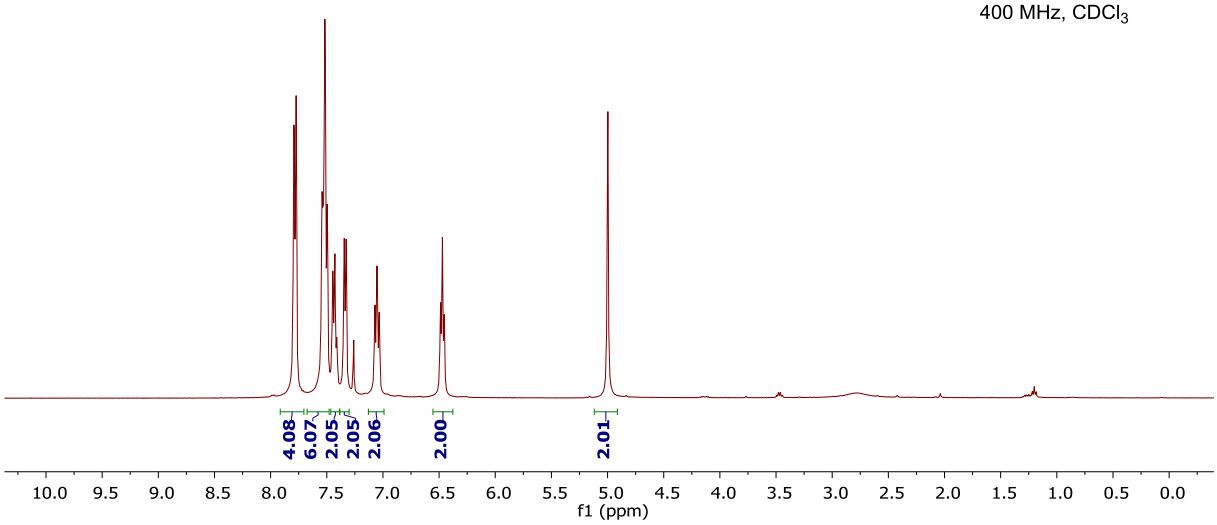


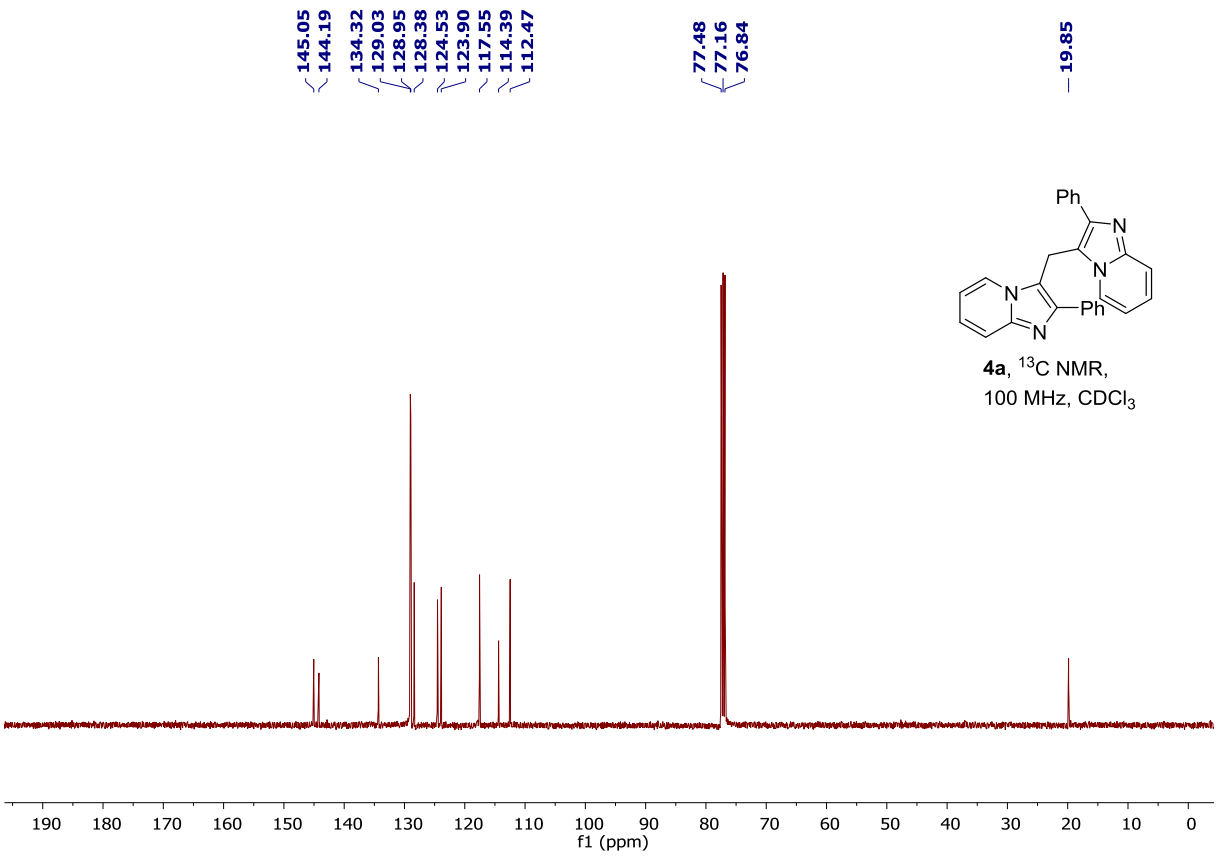
6a

$[M + H]^+$= 223.0866 (Calculated)

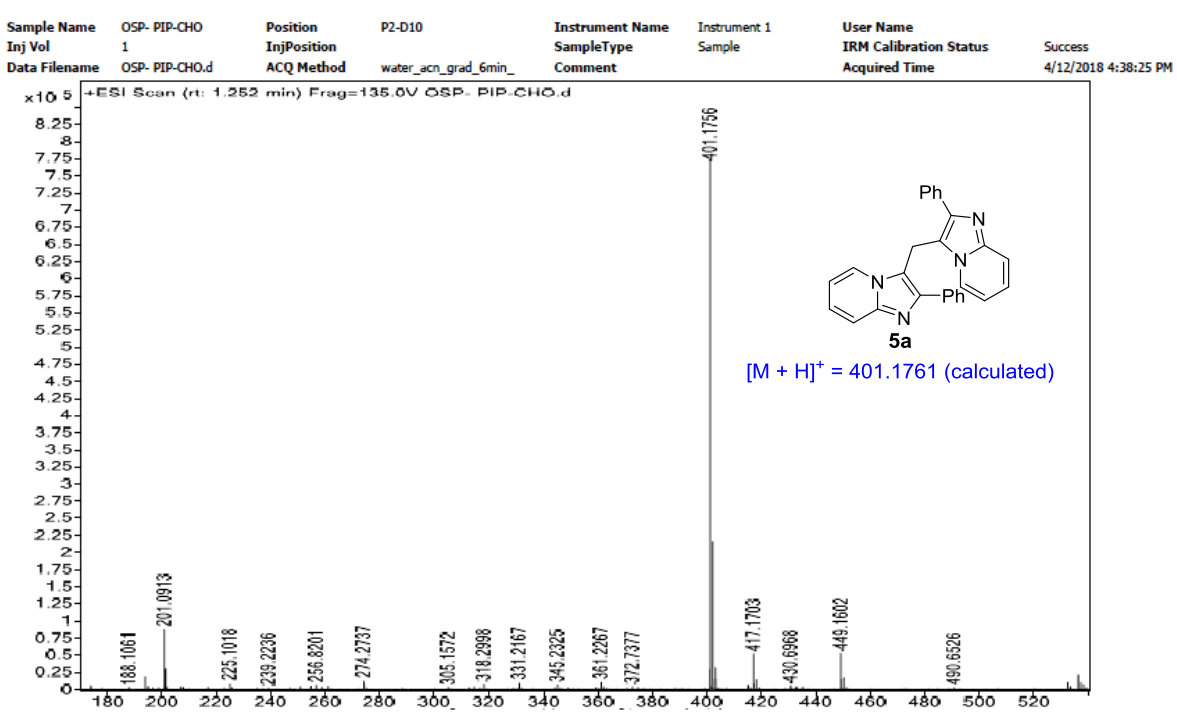


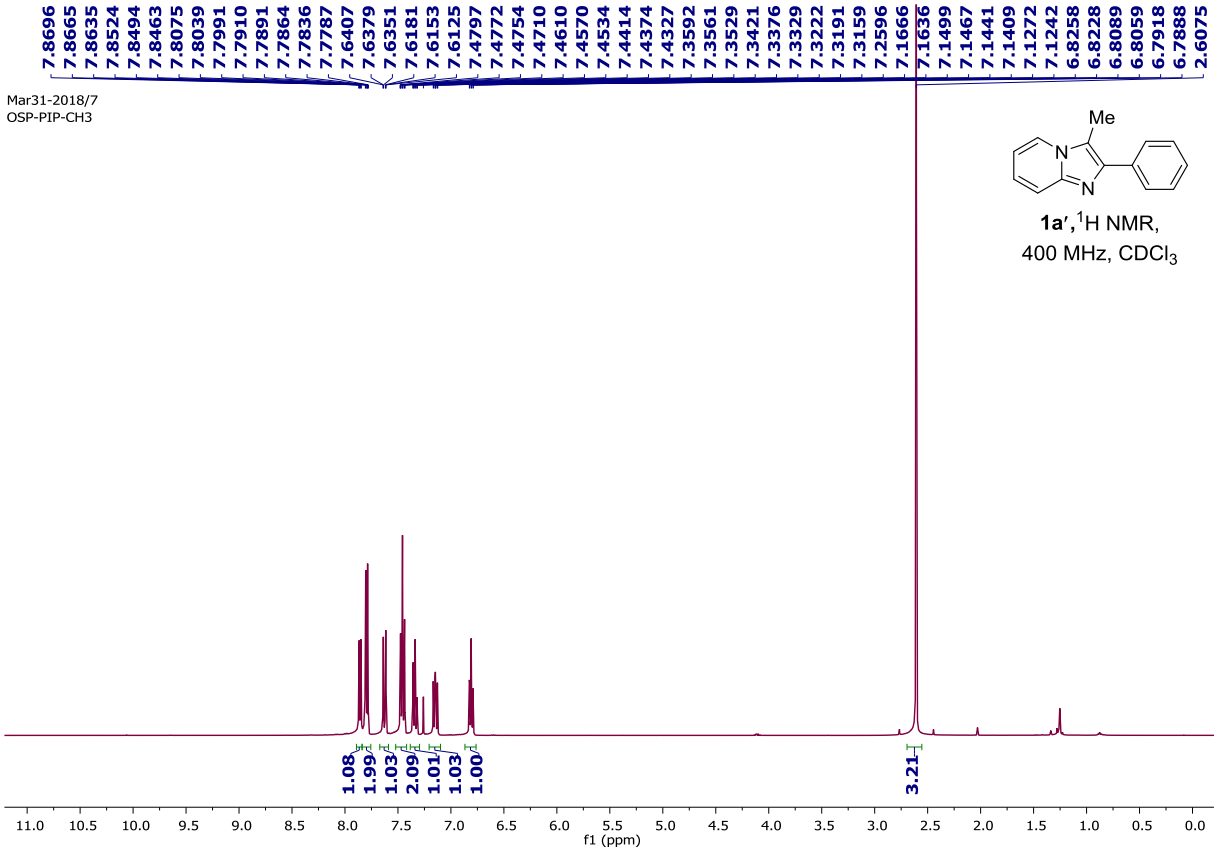
4a, ^{1}H NMR,
400 MHz, $CDCl_3$



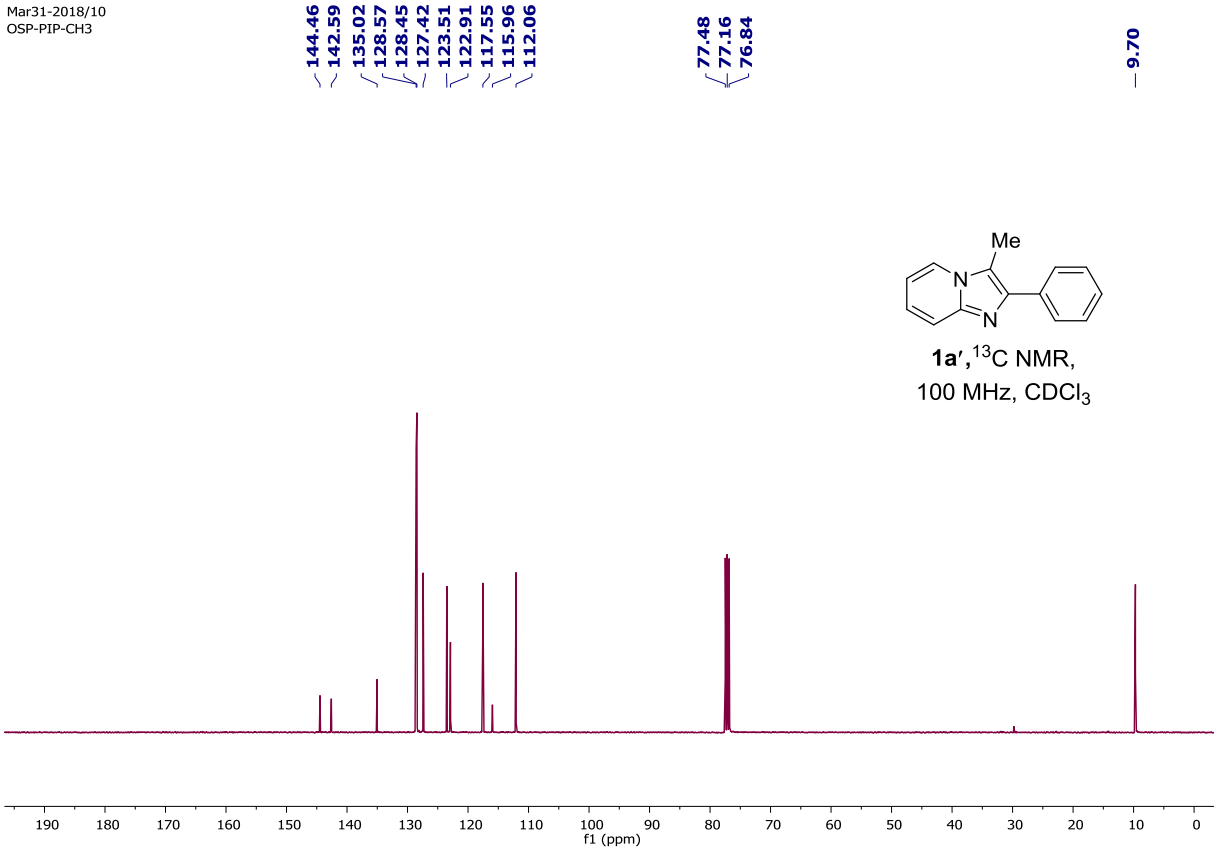


4a, ^{13}C NMR,
100 MHz, $CDCl_3$





1a′,^{1}H NMR,
400 MHz, $CDCl_3$



1a′,^{13}C NMR,
100 MHz, $CDCl_3$

Sample Name	OSP-PIP-CH3	**Position**	P1-B8	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	0.25	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	OSP-PIP-CH3.d	**ACQ Method**	water_acn_grad_6min_	**Comment**		**Acquired Time**	4/3/2018 1:06:00 PM