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

Synthesis of Multisubstituted Arylsulfones via One-Pot, Three-Component [3+3] Benzannulation Reaction

Xiang-zheng Tang[a], Jie Liang[a], Xue-jing Zhang[a], Raymond Wai-Yin Sun[b], Ming Yan[a,*], and Albert S. C. Chan[a]

[a] *School of Pharmaceutical Sciences, Sun Yat-sen University, Guangzhou 510006, China. E-mail: yanming@mail.sysu.edu.cn*

[b] *Guangzhou Lee & Man Technology Company Limited, Guangzhou 511458, China.*

Supporting Information

I. Optimization of reaction conditions

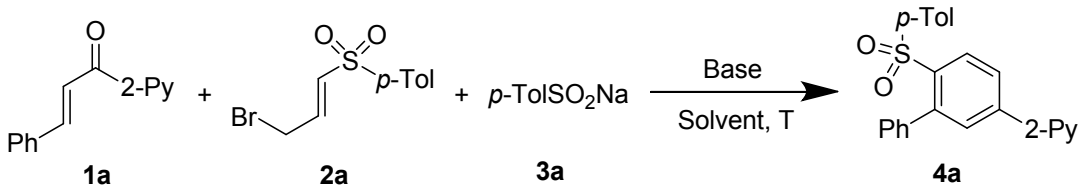


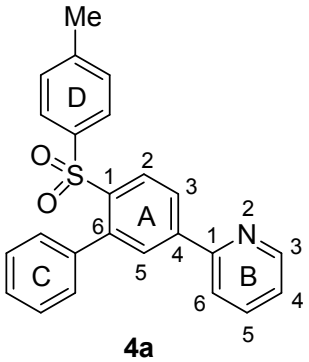
Table (SI-1). Effect of the ratio of reactants.

Entry	Solvent	Base	**1a**/**2a**/**3a**/base	T (°C)	Yield[b] (%)
1	DMF	DBU	1 : 1 : 1 : 1	60	66
2	DMF	DBU	1 : 1 : 1 : 1.2	60	70
3	DMF	DBU	1 : 1 : 1.2 : 1.2	60	74
4	DMF	DBU	1 : 1.1 : 1.2 : 1.2	60	78
5	DMF	DBU	1 : 1.1 : 1.2 : 2	60	78
6	DMF	DBU	1 : 2 : 2 : 2	60	80

Table (SI-2). Effect of the reaction time.

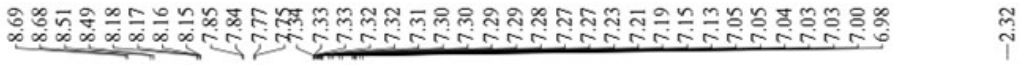
Entry	Solvent	Base	T (°C)	Time	Yield[b] (%)
1	DMF	DBU	40	24	34
2	DMF	DBU	60	24	56
3	DMF	DBU	80	24	53
4	DMF	DBU	100	24	45

II. Assignment of chemical structure of product 4a

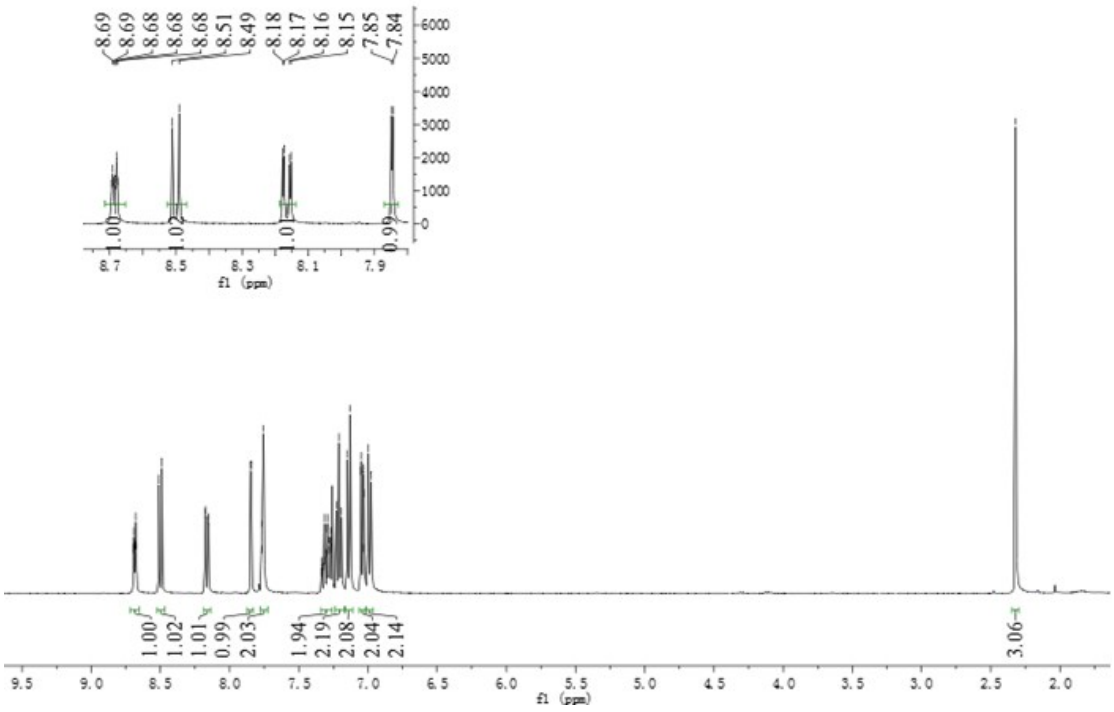


4a

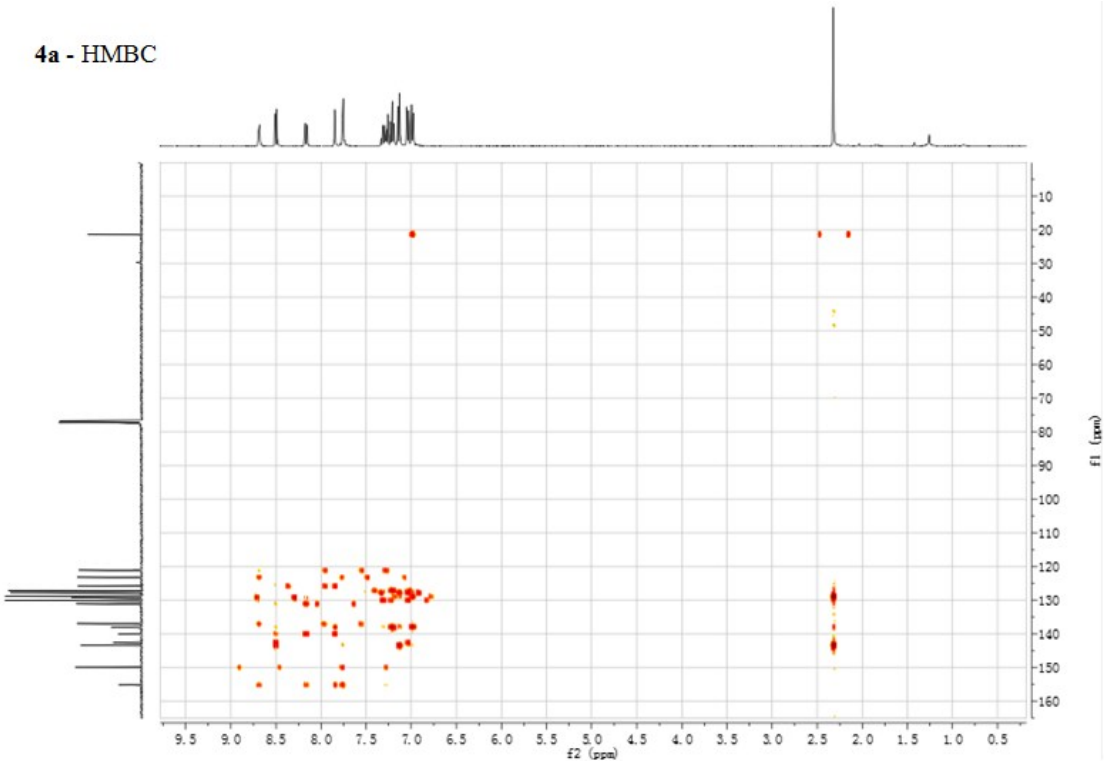
The ^{1}H NMR spectrum of **4a** showed two adjacent aromatic H(A2) and H(A3) (δ 8.50, 8.16 ppm) with a coupling constant of 8.4 Hz. In addition, the dd peak of H(A3) and second small coupling constant (1.8 Hz) indicated that it also couples with the more distant H(A5) (δ 7.85). In the ^{13}C NMR spectrum of **4a**, the signal of δ 155.27 ppm was assigned to C(B1) of the pyridine ring. The HMBC spectrum of **4a** showed that C(B1) has strong correlations with H(A3), H(A5) and H(B3). The result indicated that the pyridine ring is at A4 position. The structure of **4a** was further confirmed by HRMS.



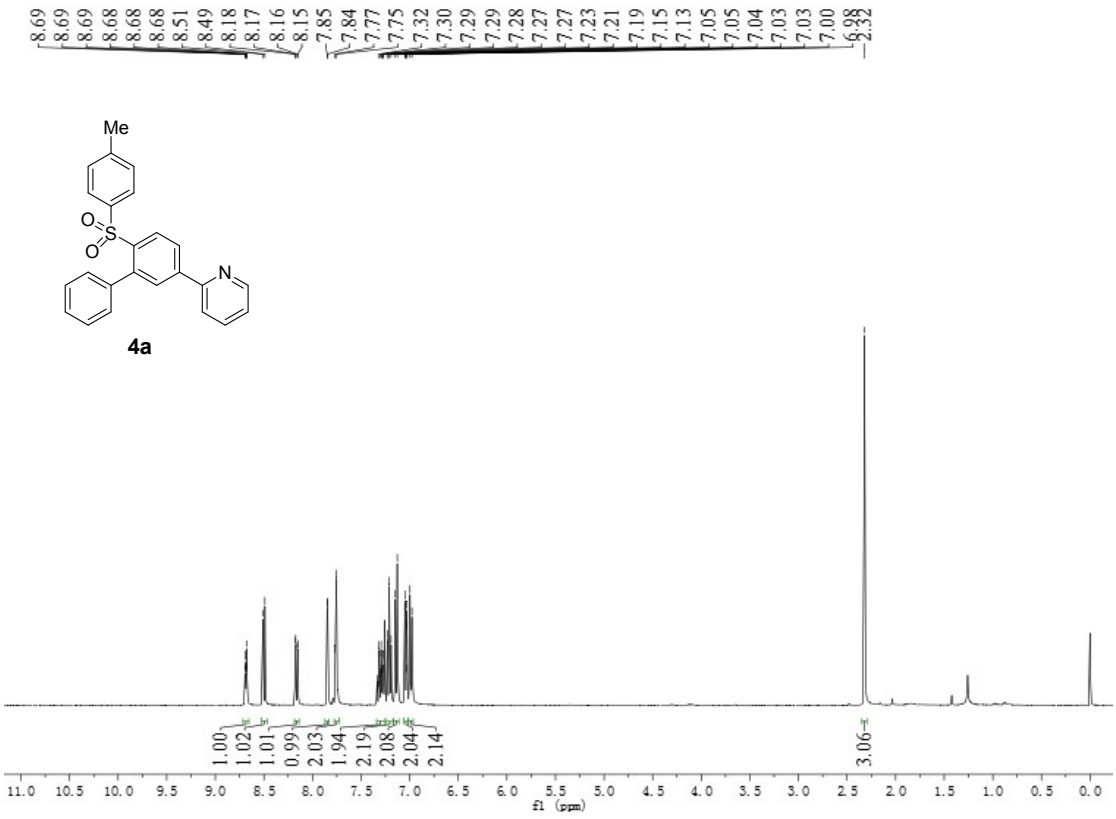
4a - ^{1}H NMR

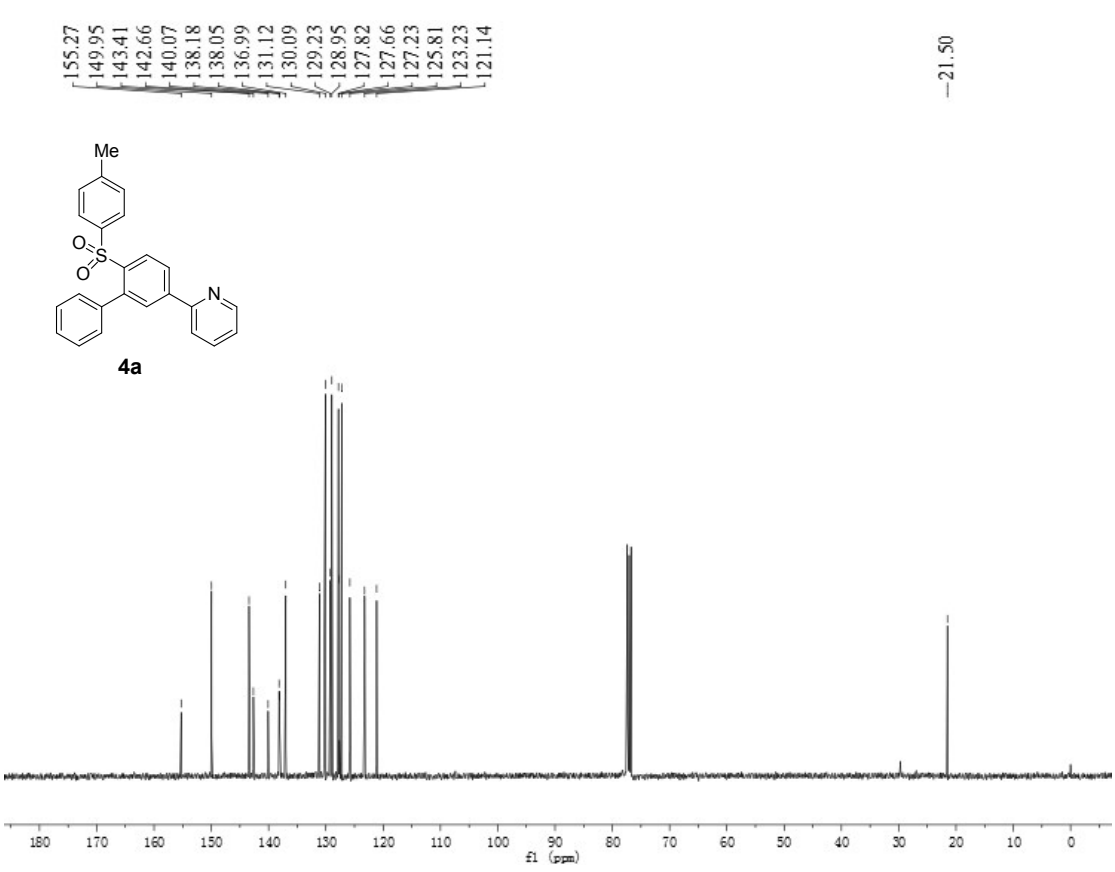


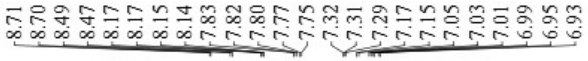
4a - HMBC



III. Copies of NMR spectra



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8.17
8.17
8.15
8.14
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7.82
7.80
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7.75
7.32
7.31
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7.15
7.05
7.03
7.01
6.99
6.95
6.93
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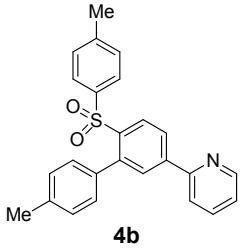
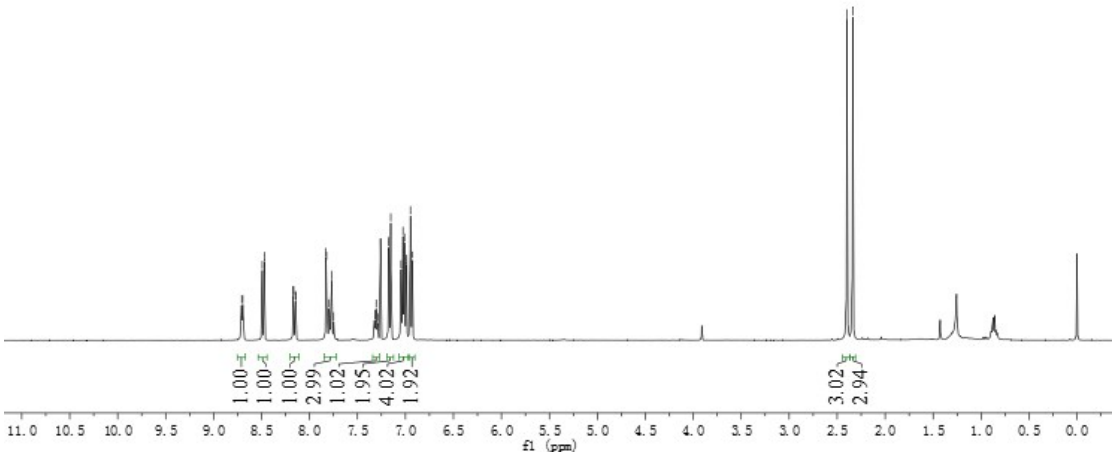
2.34


Me
O
S
O
N
Me

4b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1.00
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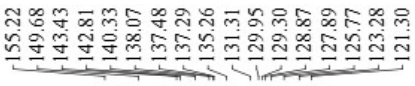
1.95
4.02
1.92
3.02
2.94
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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143.43
142.81
140.33
138.07
137.48
137.29
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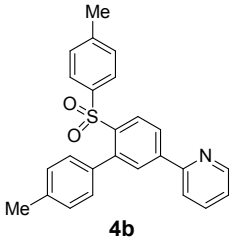
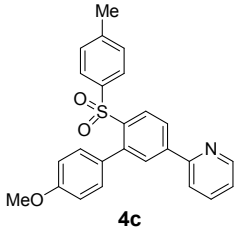
127.89
125.77
123.28
121.30
21.54
21.29


Me
O
S
O
N
Me

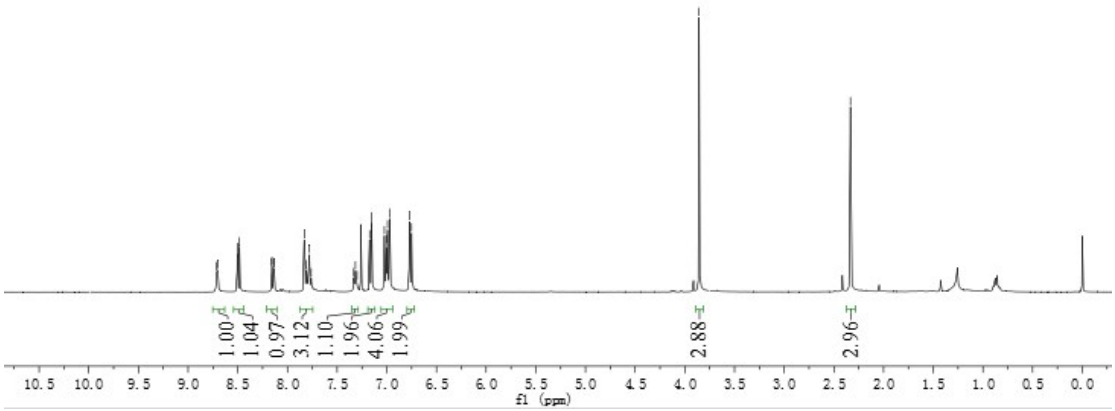
4b

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


8.71
8.70
8.50
8.48
8.16
8.16
8.14
8.13
7.83
7.81
7.78
7.76
7.33
7.32
7.30
7.17
7.15
7.02
7.00
6.99
6.97
6.77
6.75
3.86
2.33


Me
O
S
O
N
MeO

4c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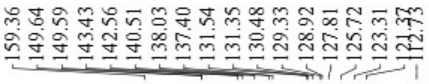
3.12
1.10
1.96
4.06
1.99
2.88
2.96
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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149.59
143.43
142.56
140.51
138.03
137.40
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129.33
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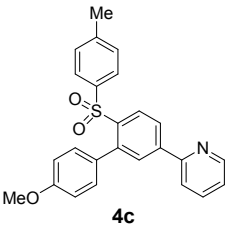
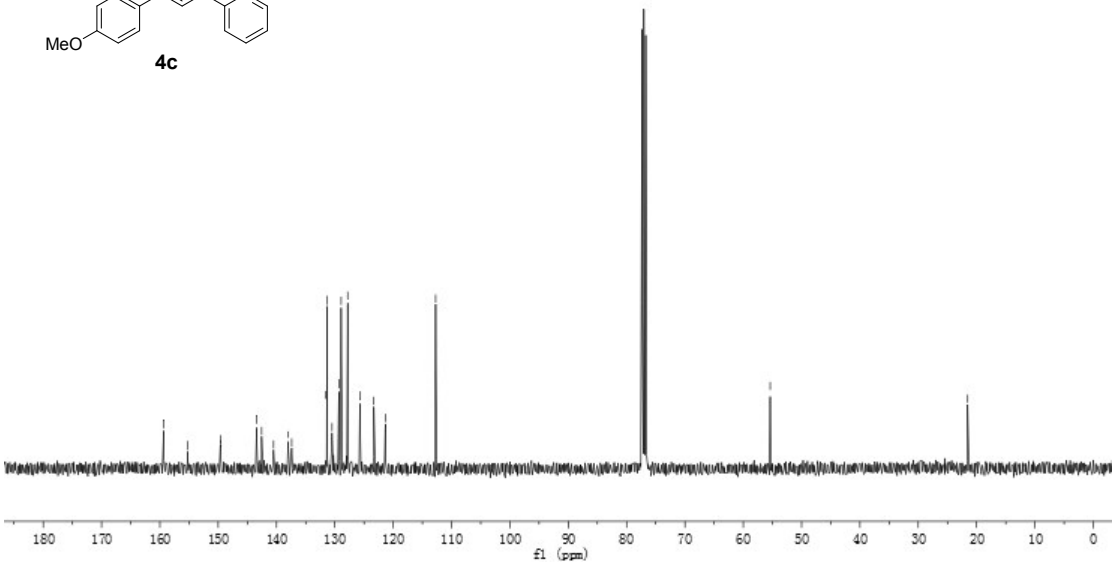
125.72
123.31
121.37
112.73
55.38
21.53


Me
O
S
O
N
MeO

4c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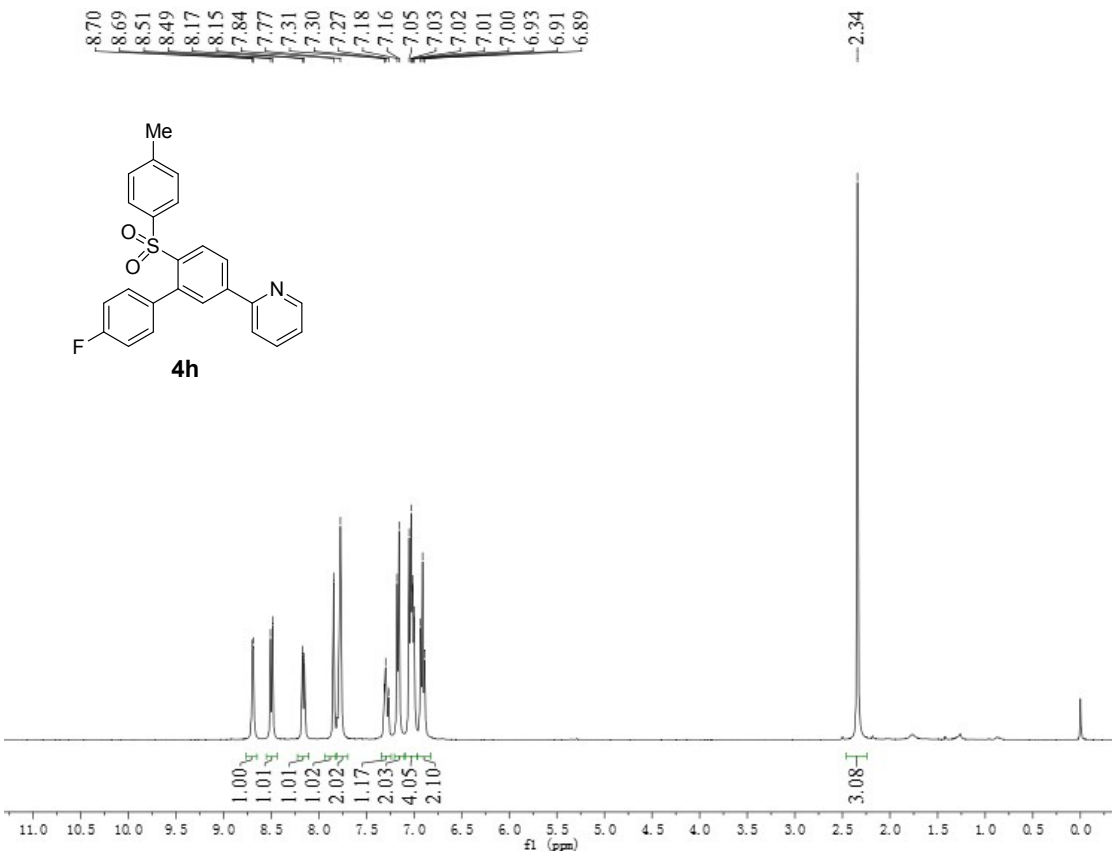
f1 (ppm)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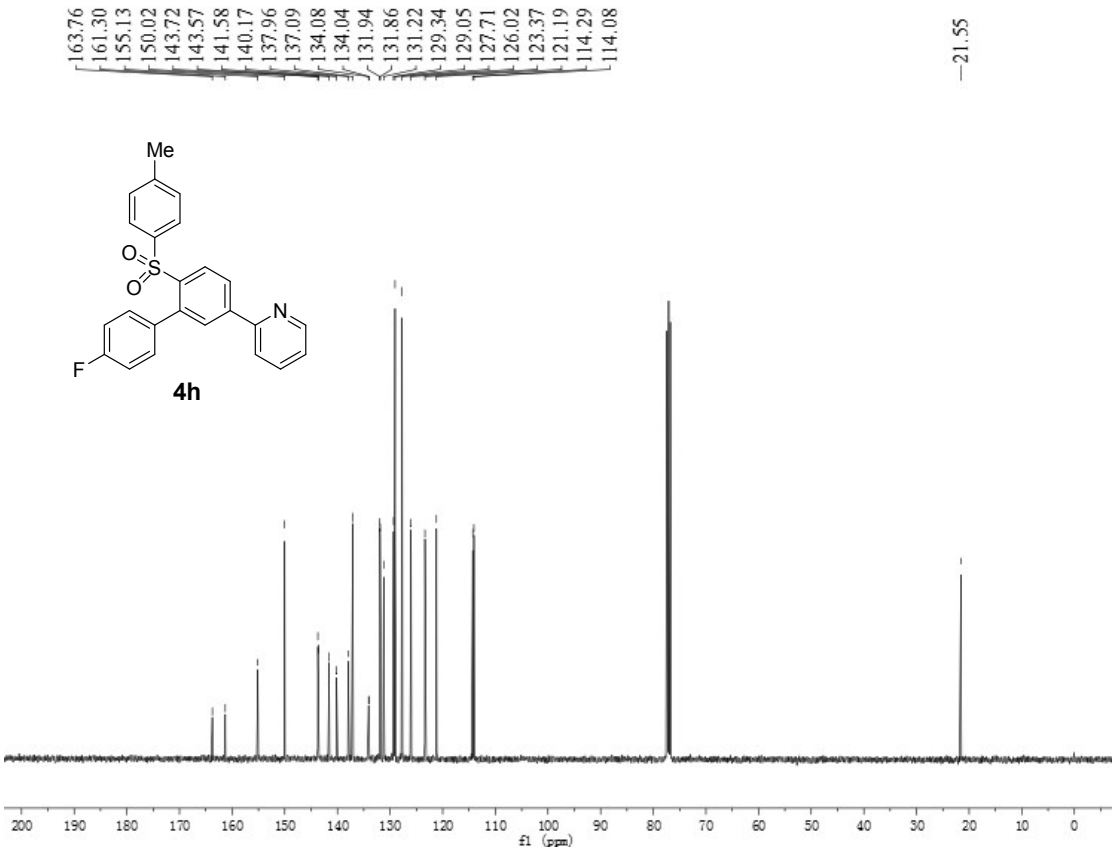
7.30
7.27
7.18
7.16
7.05
7.03
7.02
7.01
7.00
6.93
6.91
6.89
2.34
Me
O
S
O
N
F
4h
1.00
1.01
1.01
1.02
2.02
1.17
2.03
4.05
2.10
3.08
f1 (ppm)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131.86
131.22
129.34
129.05
127.71
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114.29
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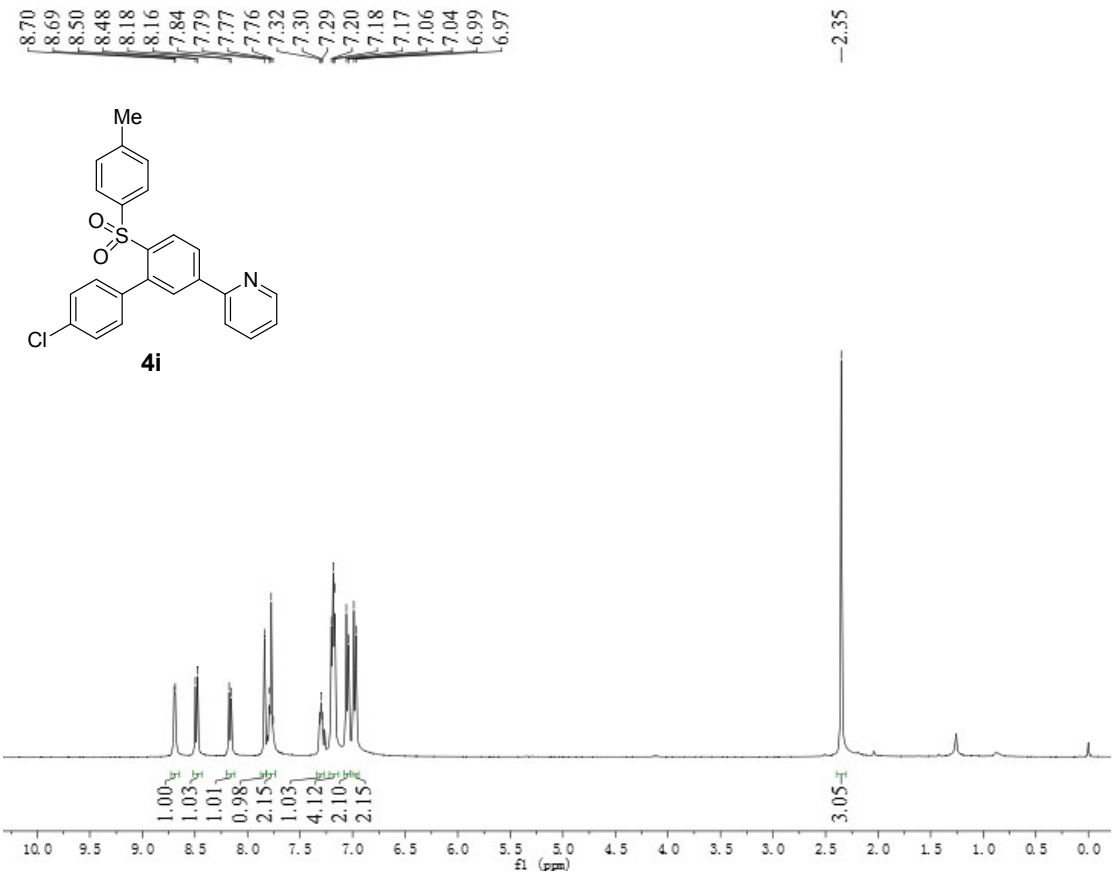
O
S
O
N
F
4h
f1 (ppm)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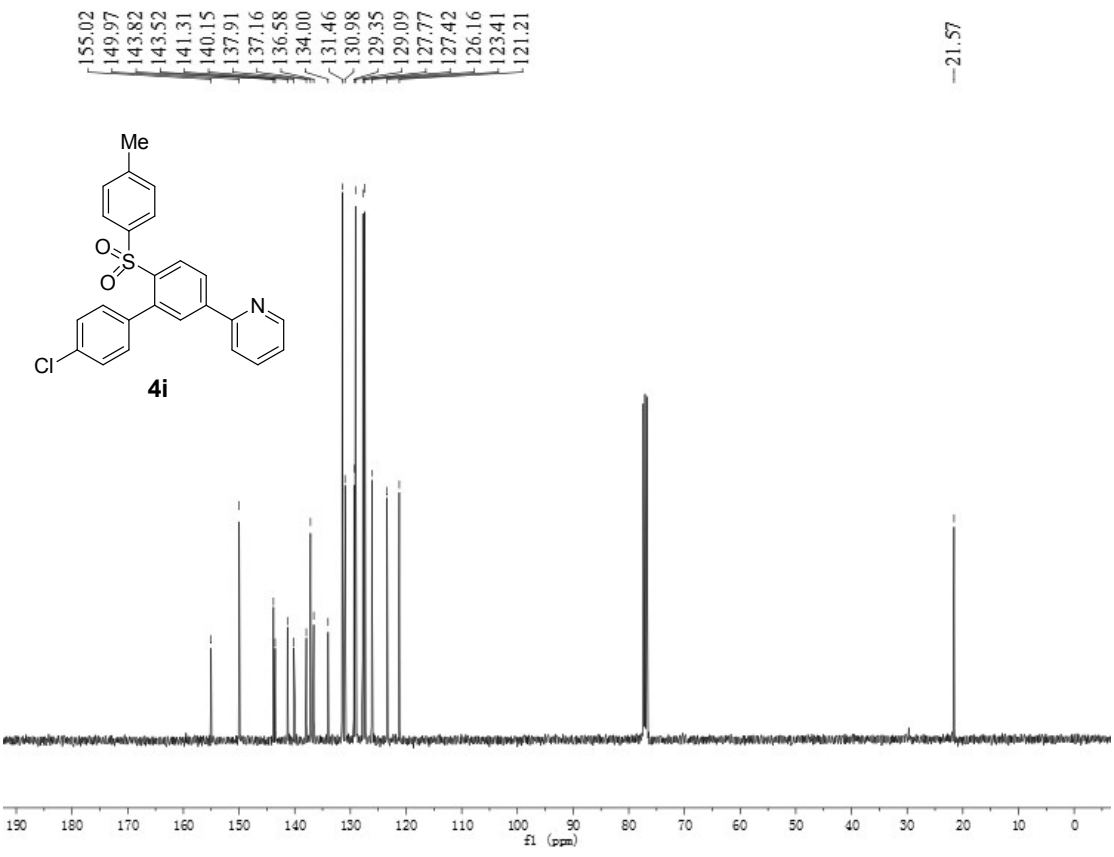
8.69
8.50
8.48
8.18
8.16
7.84
7.79
7.77
7.76
7.32
7.30
7.29
7.20
7.18
7.17
7.06
7.04
6.99
6.97
2.35
Me
O
S
O
N
Cl
4i
1.00
1.03
1.01
0.98
2.15
1.03
4.12
2.10
2.15
3.05
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.35
129.09
127.77
127.42
126.16
123.41
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O
S
O
N
Cl
4i
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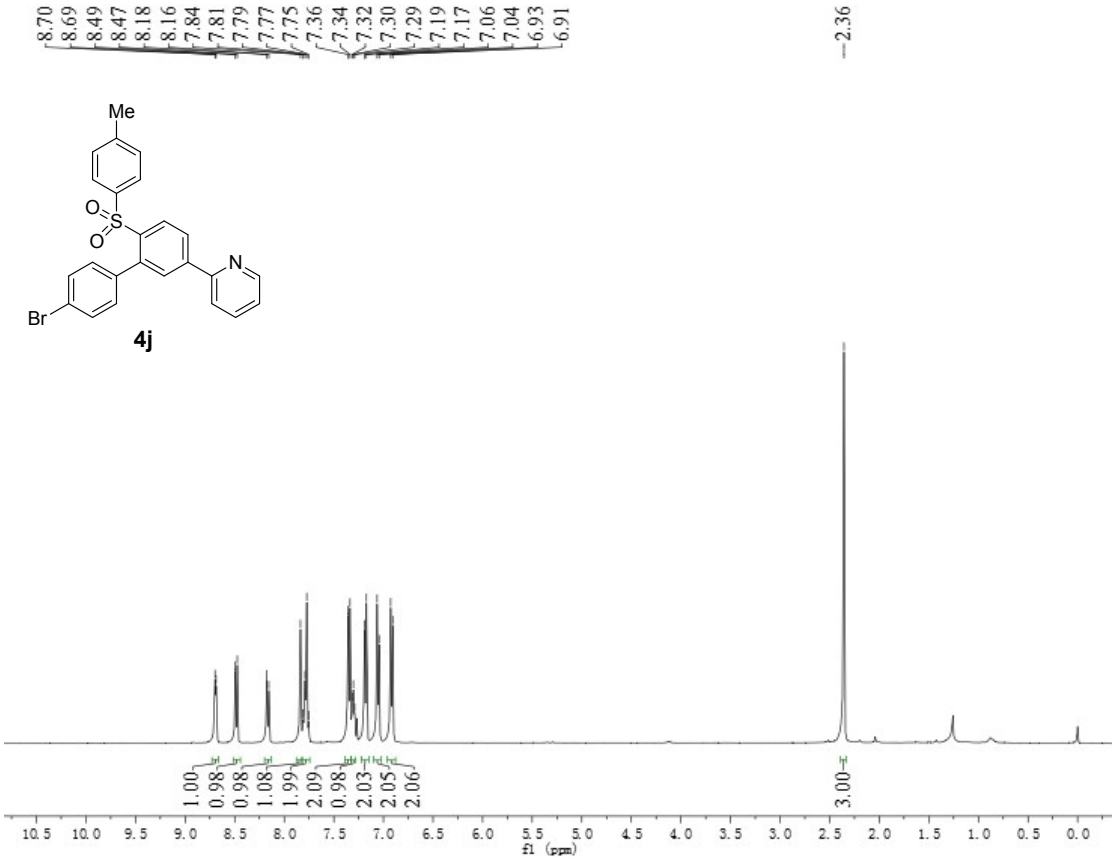
f1 (ppm)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7.34
7.32
7.30
7.29
7.19
7.17
7.06
7.04
6.93
6.91
2.36
Me
O
S
O
N
Br
4j
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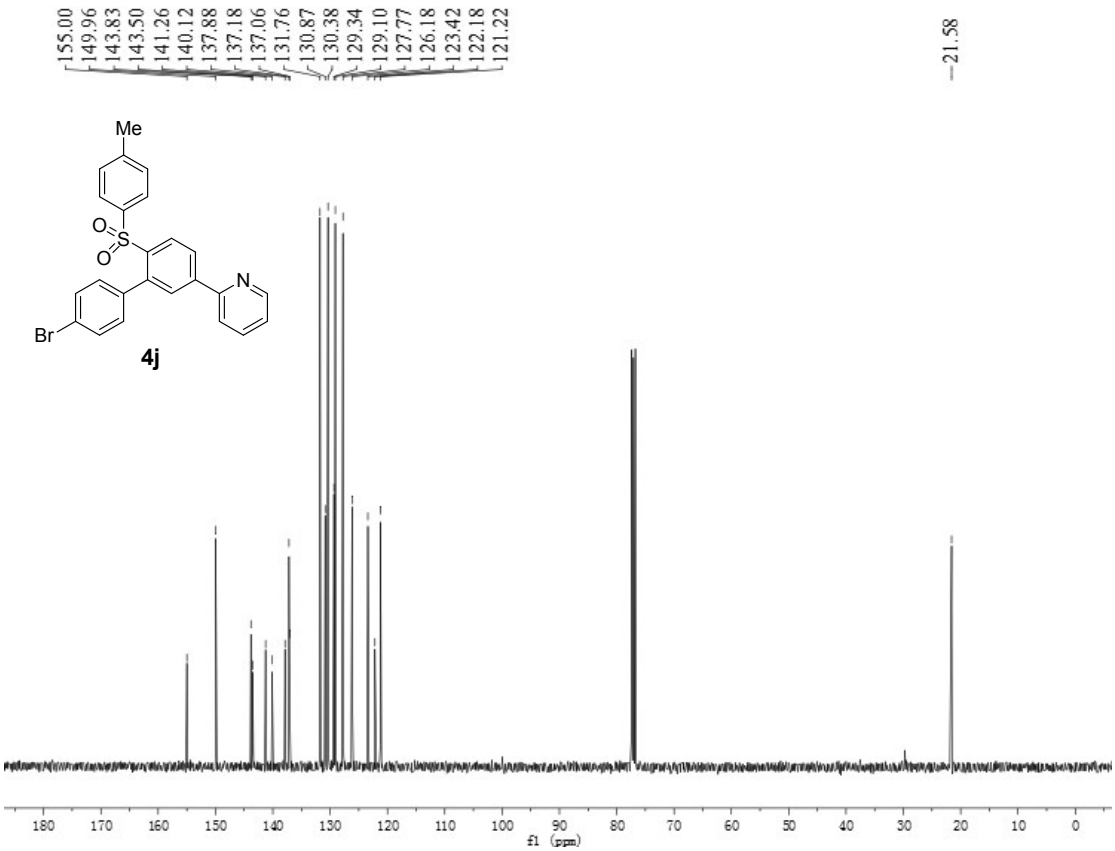
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129.34
129.10
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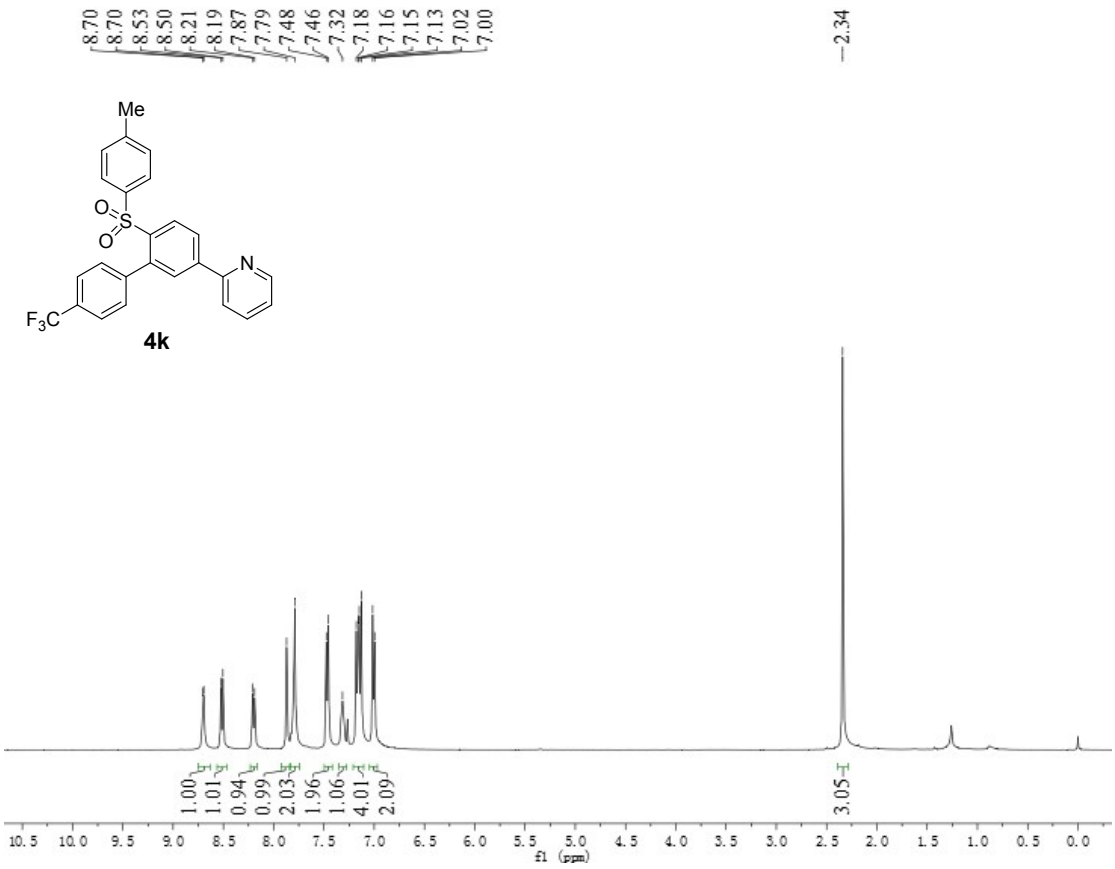
Me
O
S
O
N
Br
4j
f1 (ppm)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F3C
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0.94
0.99
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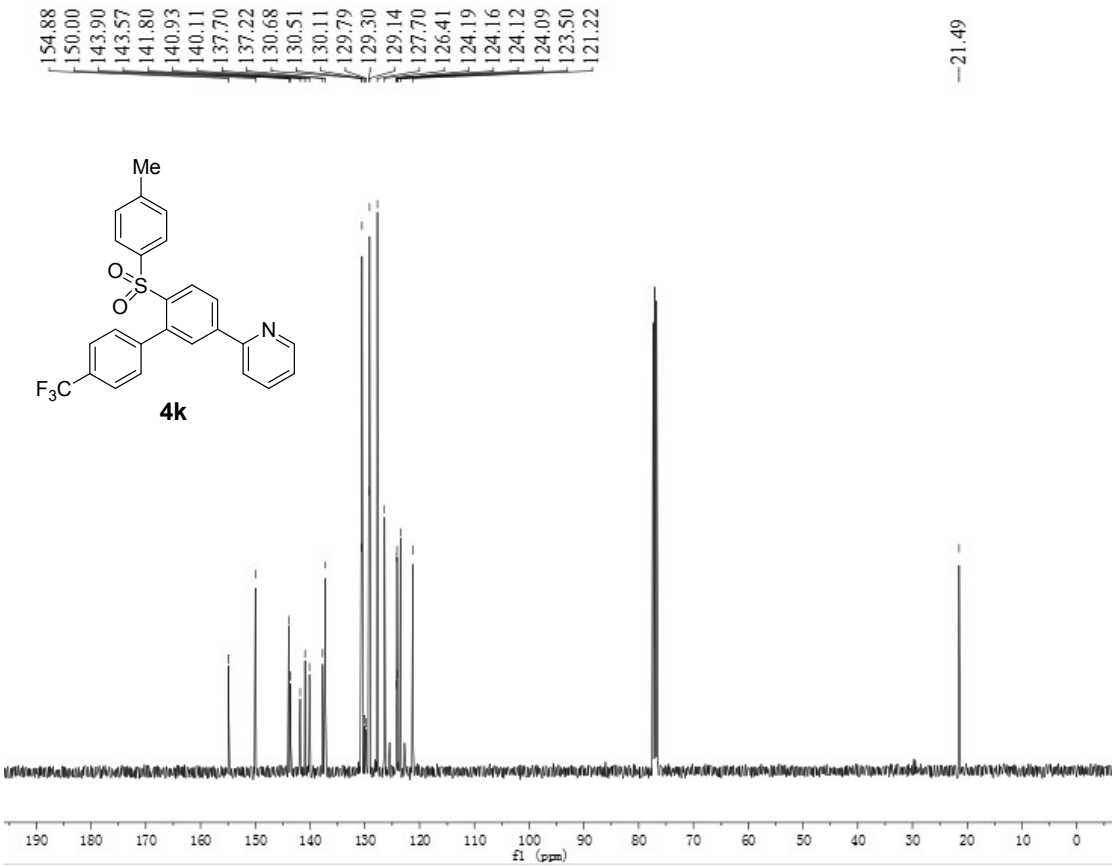
1.96
1.06
4.01
2.09
3.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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124.16
124.12
124.09
123.50
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S
O
N
F3C
4k
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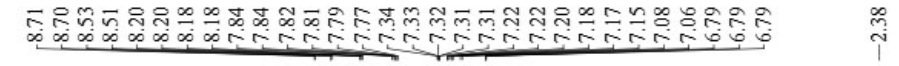
f1 (ppm)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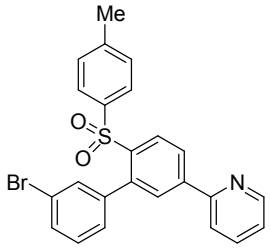
Me
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S
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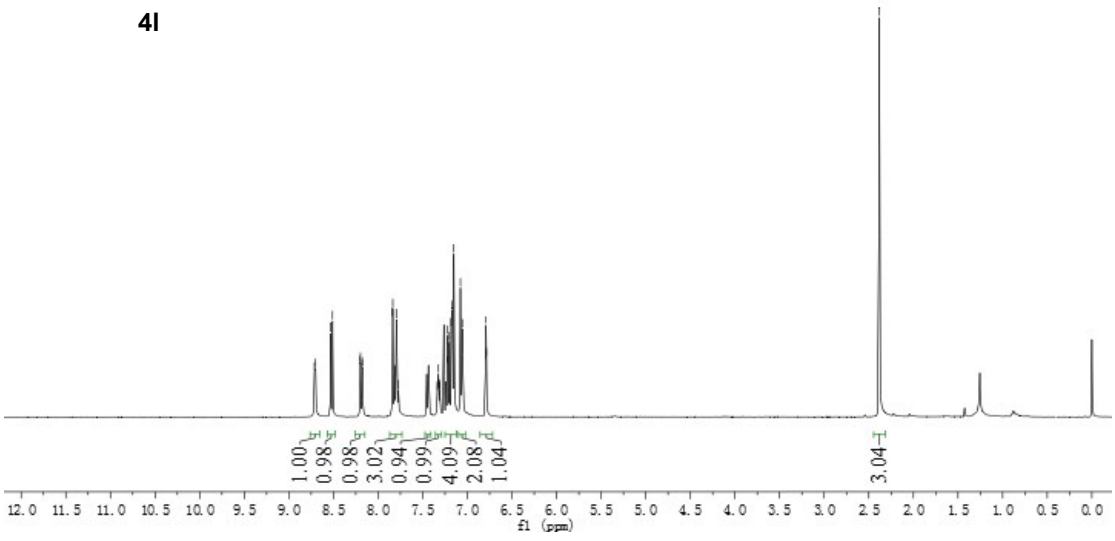
Br
N

4l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0.98
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0.94
0.99
4.09
2.08
1.04
3.04
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


154.89
149.79
144.01
143.25
140.88
140.26
139.93
137.68
137.37
132.28
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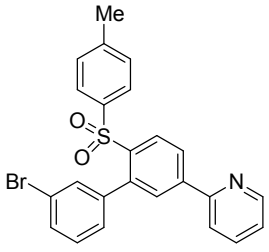
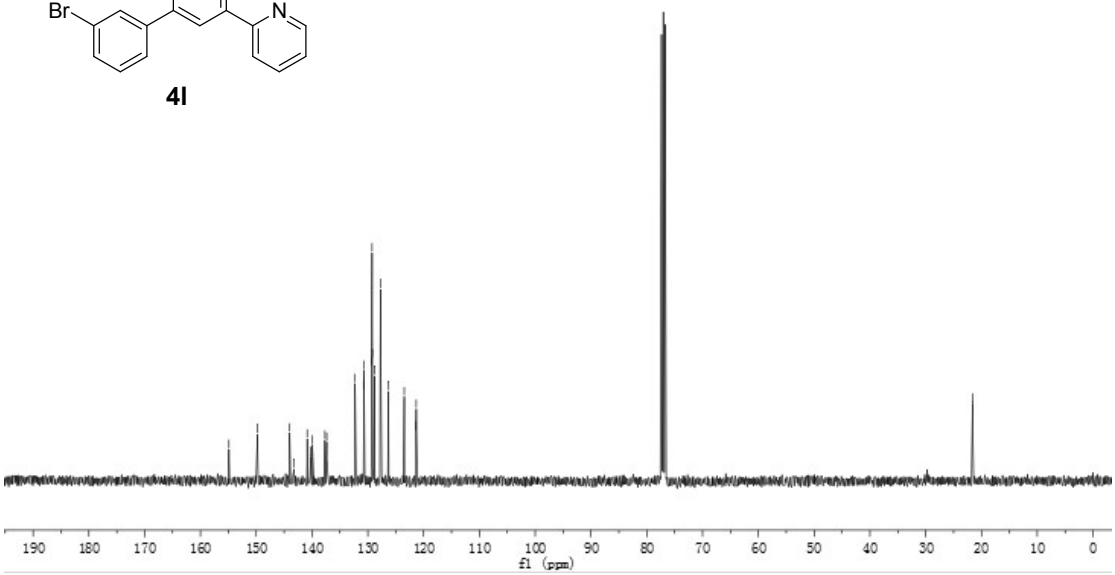
121.32
21.61


Me
O
S
O
Br
N

4l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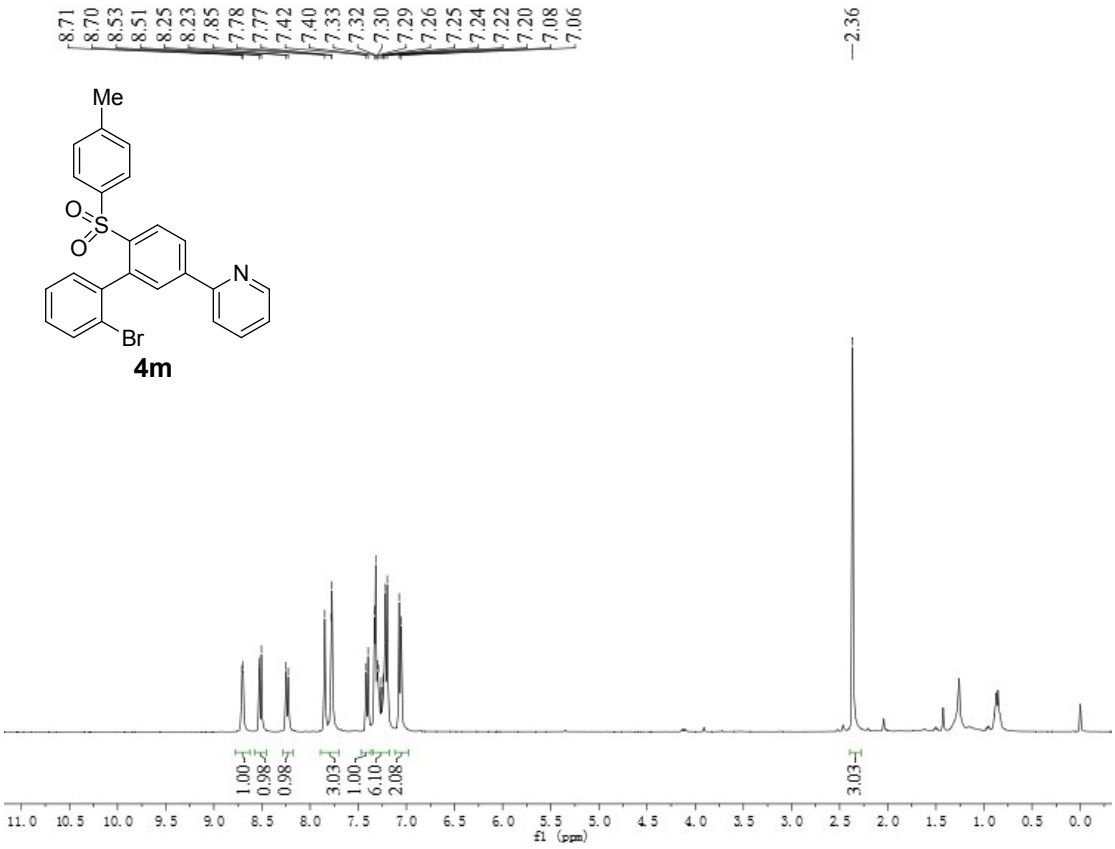
f1 (ppm)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7.30
7.29
7.26
7.25
7.24
7.22
7.20
7.08
7.06
2.36
Me
O
S
O
N
Br
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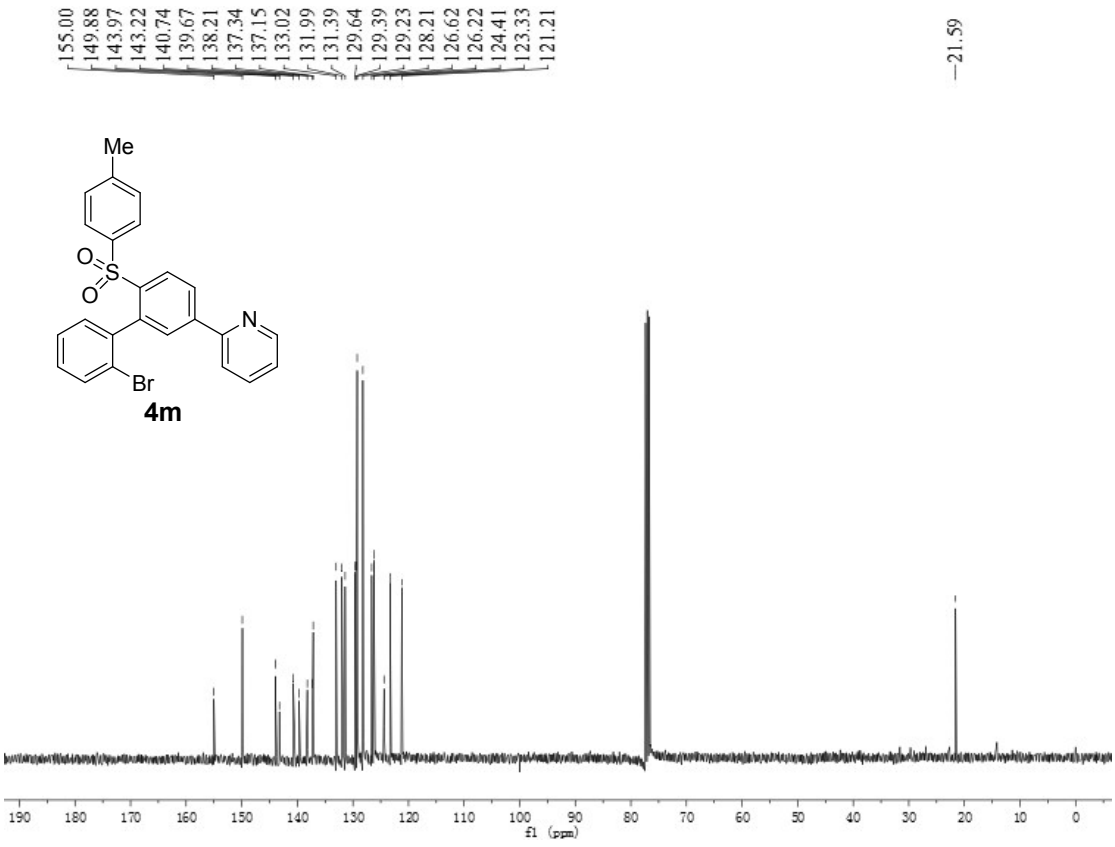
0.98
3.03
1.00
6.10
2.08
3.03
f1 (ppm)

4m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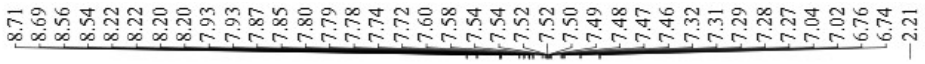
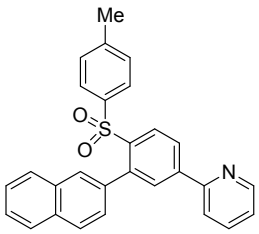
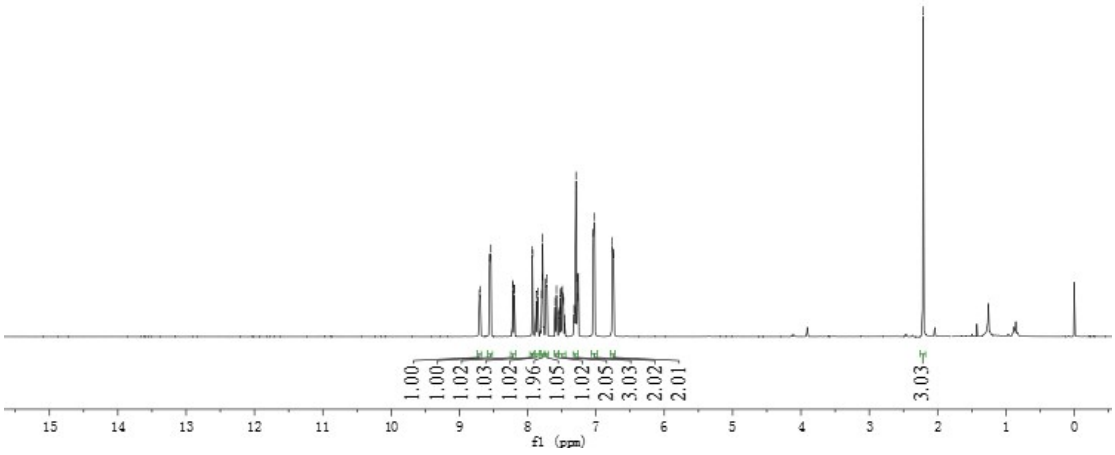
143.22
140.74
139.67
138.21
137.34
137.15
133.02
131.99
131.39
129.64
129.39
129.23
128.21
126.62
126.22
124.41
123.33
121.21
21.59
Me
O
S
O
N
Br
f1 (ppm)

4m


8.71
8.69
8.56
8.54
8.22
8.22
8.20
8.20
7.93
7.93
7.87
7.85
7.80
7.79
7.78
7.74
7.72
7.60
7.58
7.54
7.54
7.52
7.52
7.50
7.49
7.48
7.47
7.46
7.32
7.31
7.29
7.28
7.27
7.04
7.02
6.76
6.74
2.21

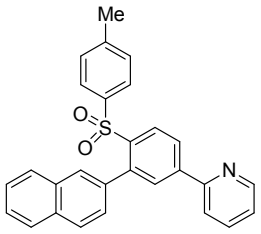

Me
O
S
O
N

4n


1.00
1.00
1.02
1.03
1.02
1.96
1.05
1.02
2.05
3.03
2.02
2.01
3.03
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

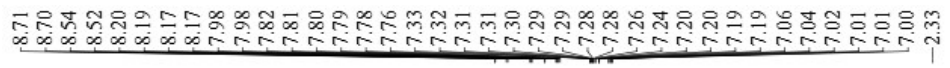

155.18
149.80
143.48
143.20
142.58
140.49
137.92
137.30
135.64
132.57
132.41
131.20
129.22
129.02
128.80
128.07
128.04
127.78
127.64
126.69
126.34
126.10
126.02
123.35
121.32

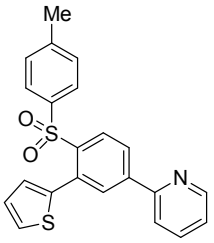

21.39


Me
O
S
O
N

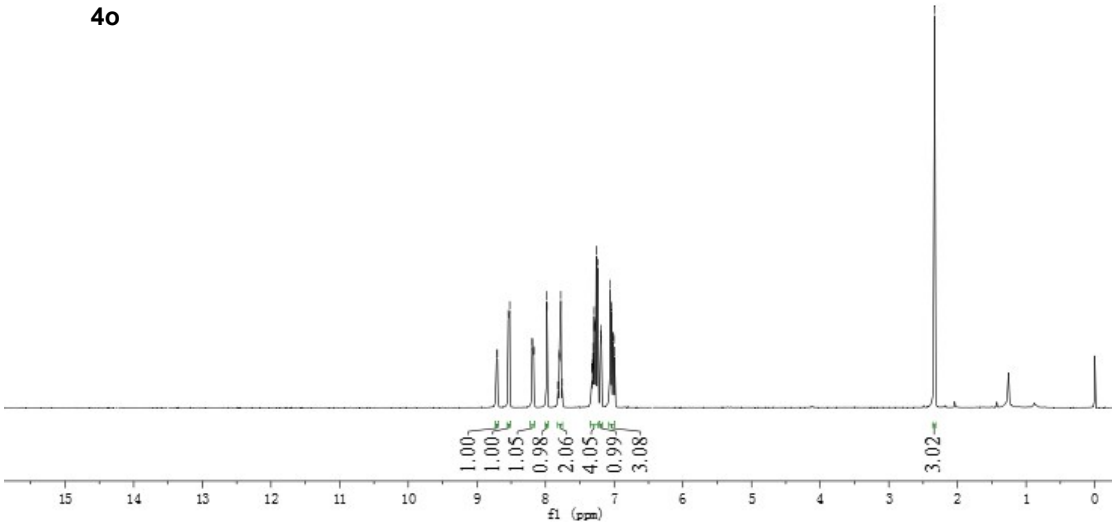
4n

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.71
8.70
8.54
8.52
8.20
8.19
8.17
8.17
7.98
7.98
7.82
7.81
7.80
7.79
7.78
7.76
7.33
7.32
7.31
7.31
7.30
7.29
7.29
7.28
7.28
7.26
7.24
7.20
7.20
7.19
7.19
7.06
7.04
7.02
7.01
7.01
7.00
2.33

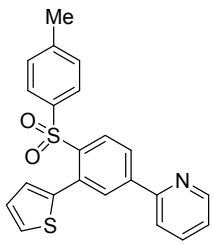

Me
O
S
O
N
S

4o


1.00
1.00
1.05
0.98
2.06
4.05
0.99
3.08
3.02
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

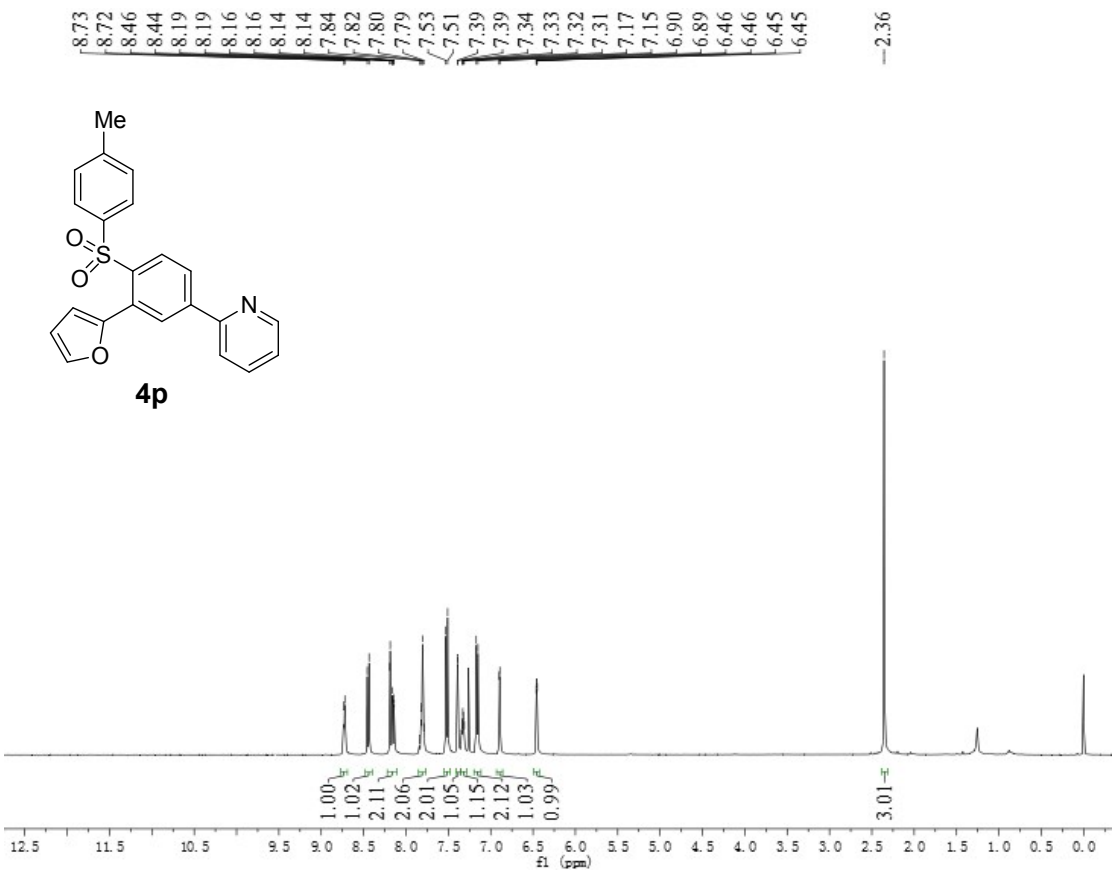

154.90
149.87
143.55
141.08
137.56
137.46
137.21
134.93
132.45
130.93
129.70
129.02
127.75
127.06
126.58
126.46
123.39
121.24

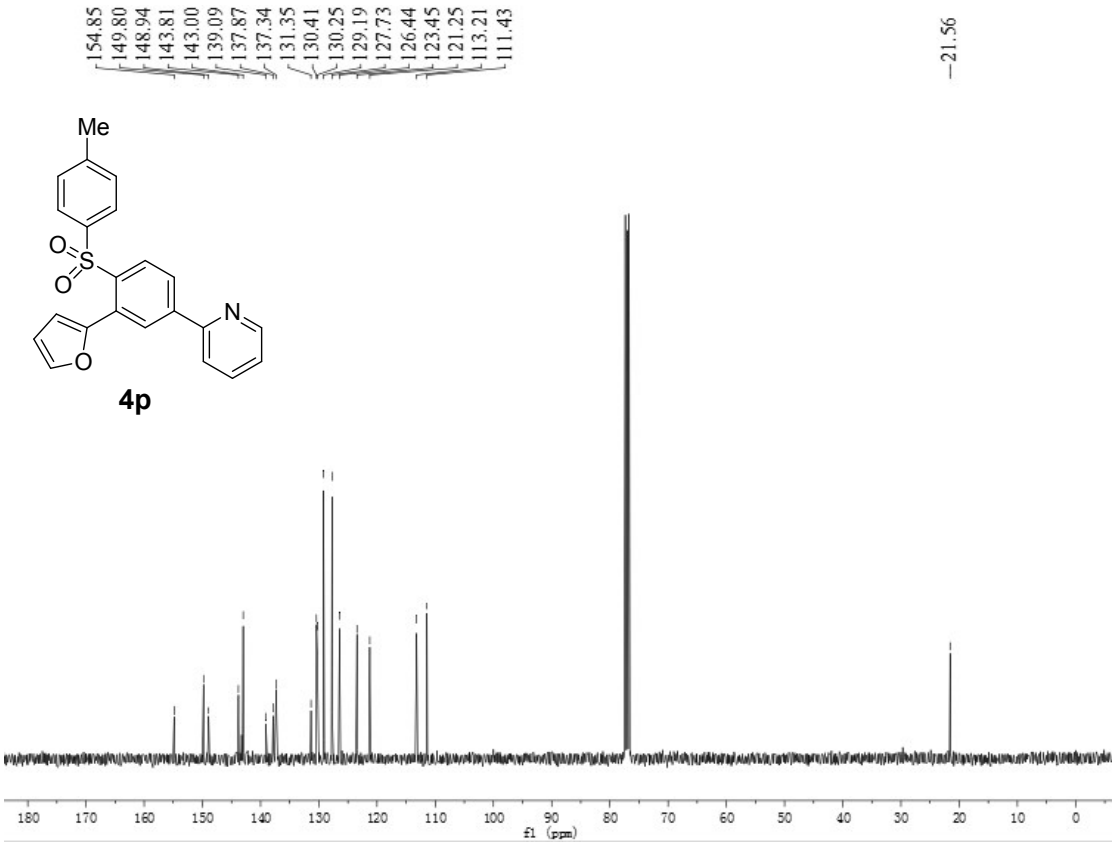

21.57

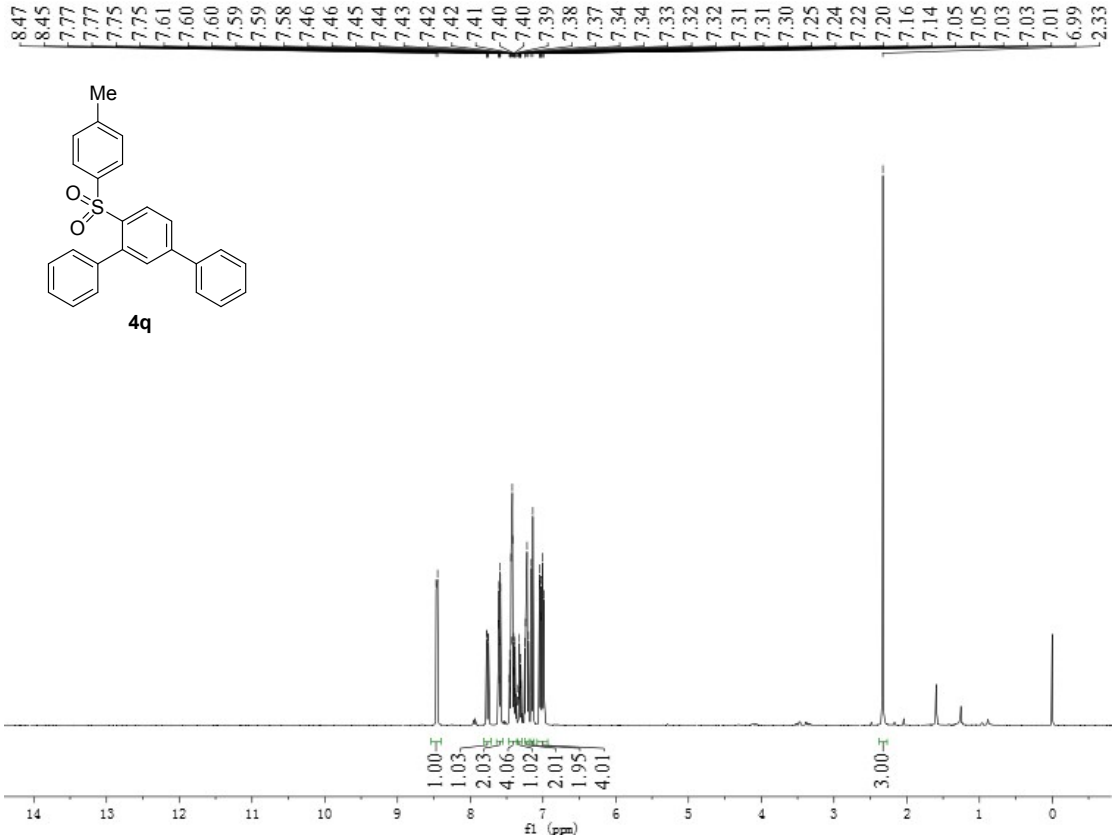

Me
O
S
O
N
S

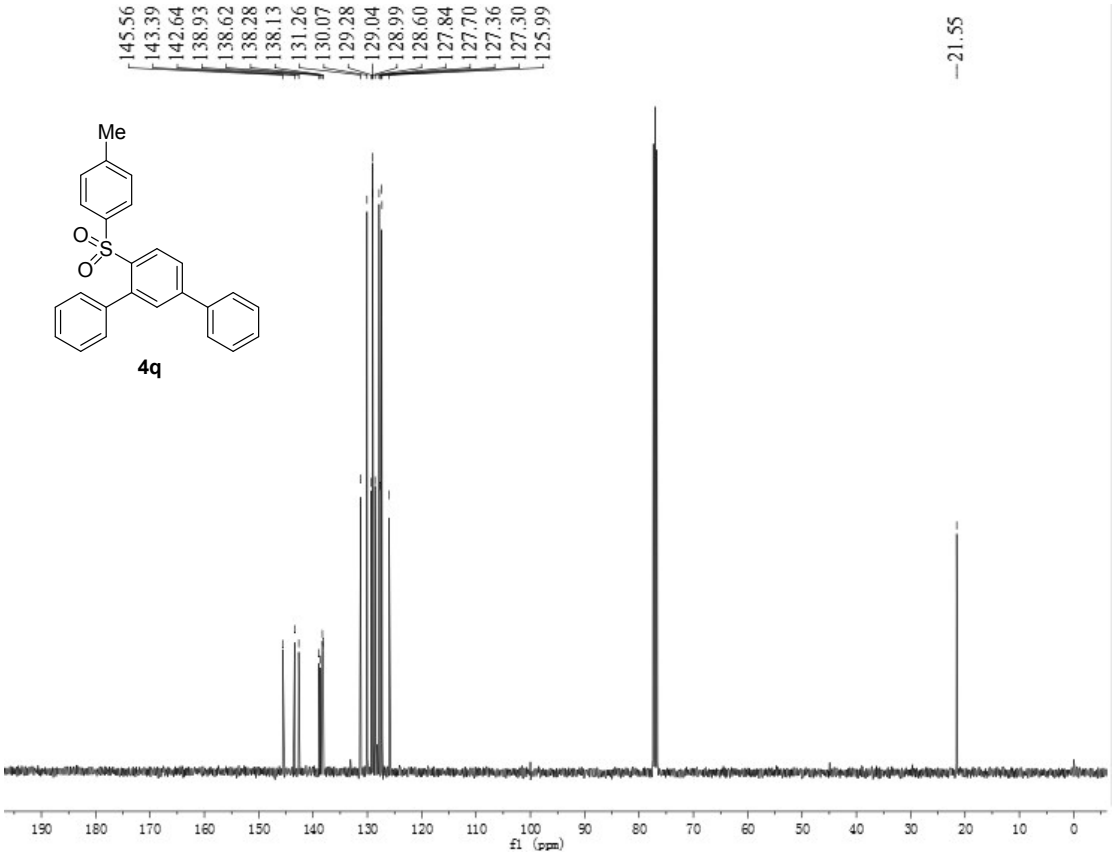
4o

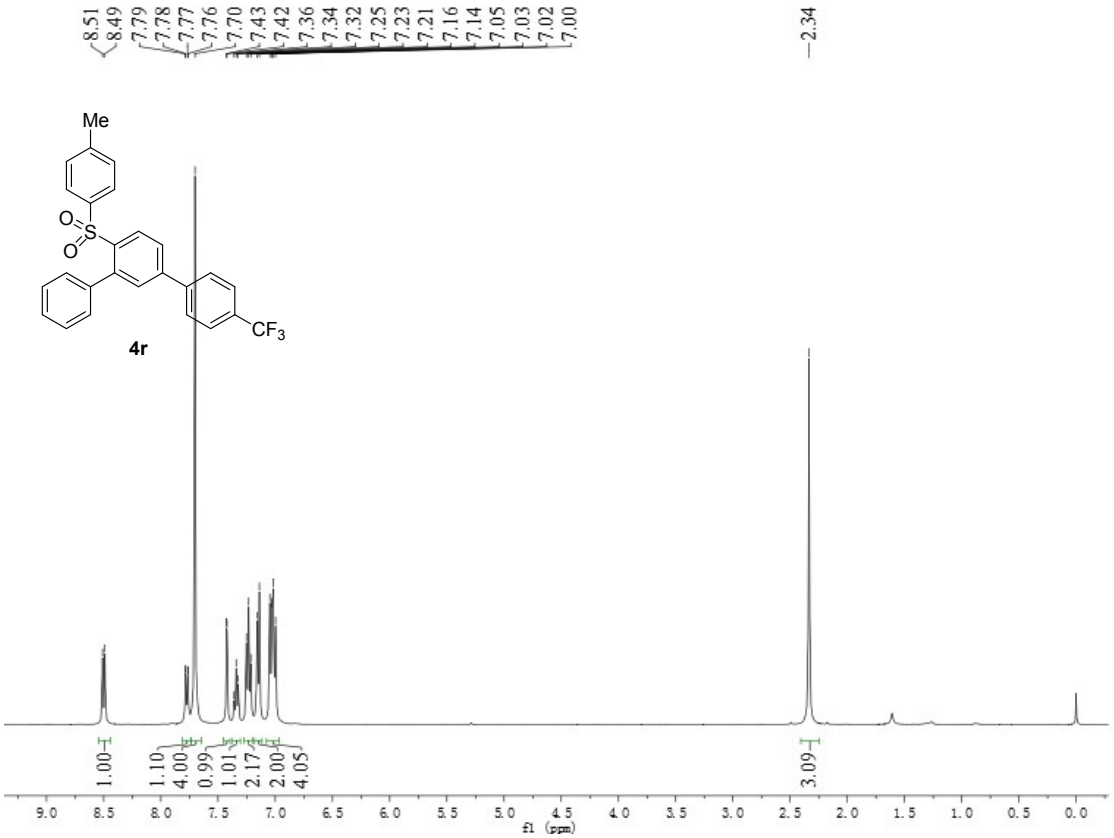
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

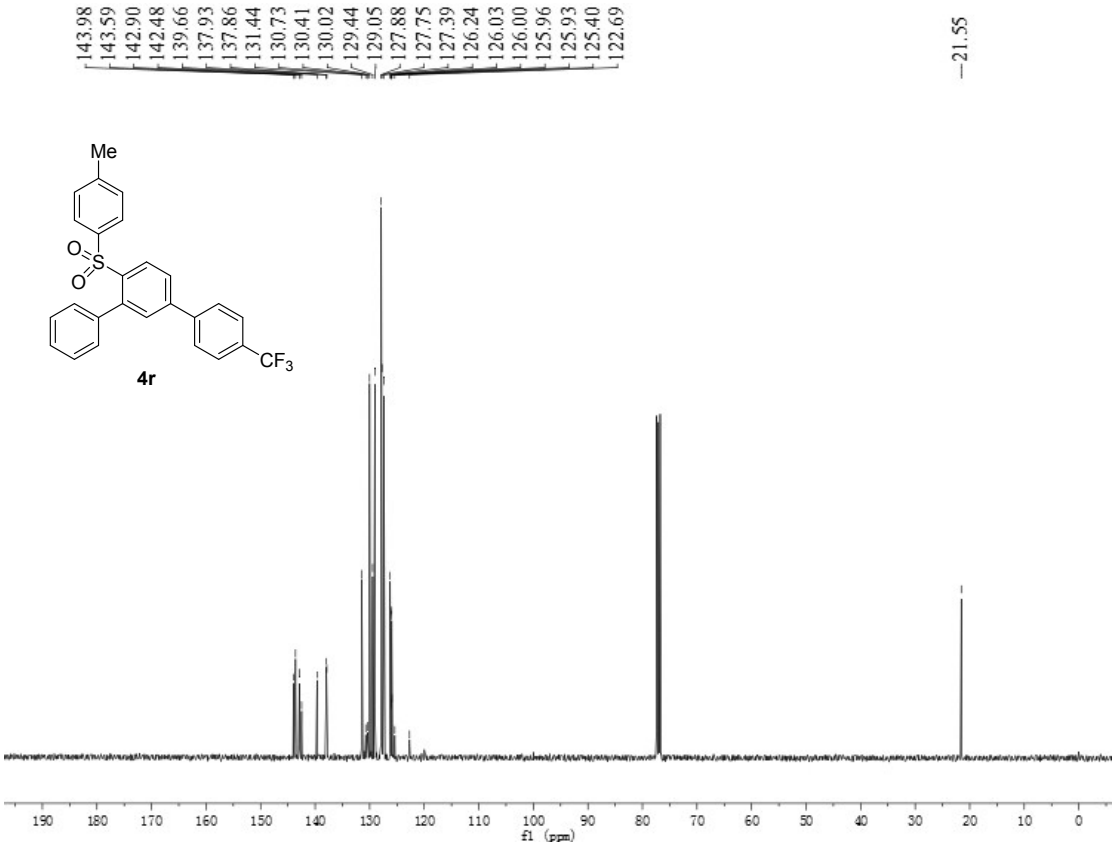

8.73
8.72
8.46
8.44
8.19
8.19
8.16
8.16
8.14
8.14
7.84
7.82
7.80
7.79
7.53
7.51
7.39
7.39
7.34
7.33
7.32
7.31
7.17
7.15
6.90
6.89
6.46
6.46
6.45
6.45
2.36
Me
O
S
O
N
O
4p
1.00
1.02
2.11
2.06
2.01
1.05
1.15
2.12
1.03
0.99
3.01
f1 (ppm)

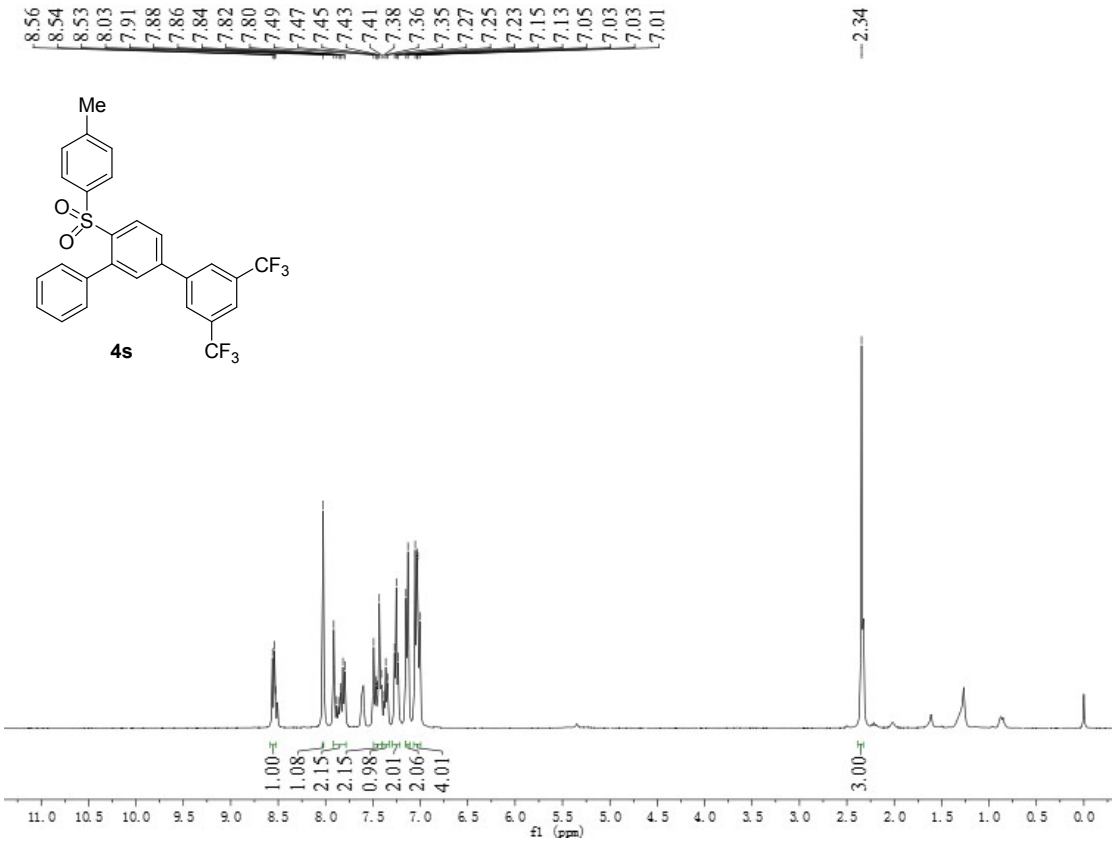

154.85
149.80
148.94
143.81
143.00
139.09
137.87
137.34
131.35
130.41
130.25
129.19
127.73
126.44
123.45
121.25
113.21
111.43
21.56
Me
O
S
O
N
O
4p
f1 (ppm)

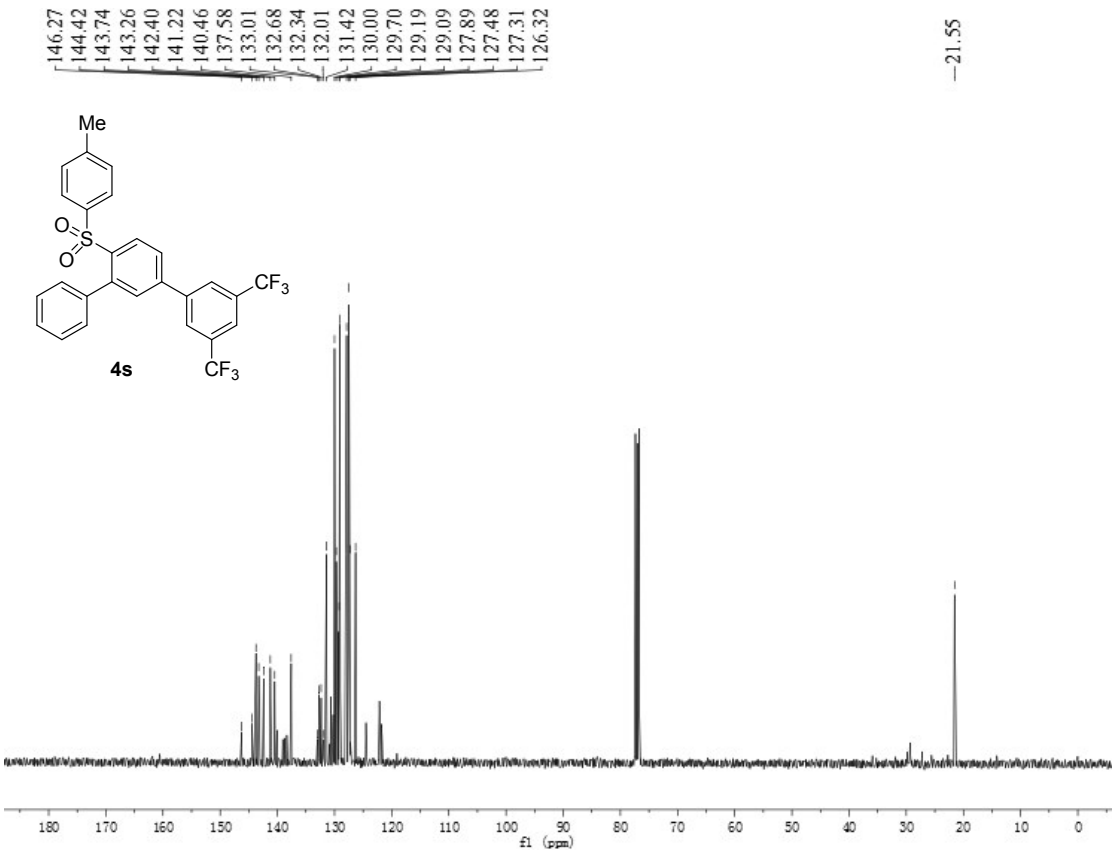

8.47
8.45
7.77
7.77
7.75
7.75
7.61
7.60
7.60
7.59
7.59
7.58
7.46
7.45
7.44
7.43
7.42
7.42
7.41
7.40
7.40
7.39
7.38
7.37
7.34
7.34
7.33
7.32
7.32
7.31
7.31
7.30
7.25
7.24
7.22
7.20
7.16
7.14
7.05
7.05
7.03
7.03
7.01
6.99
2.33
Me
O
S
O
4q
1.00
1.03
2.03
4.06
1.02
2.01
1.95
4.01
3.00
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

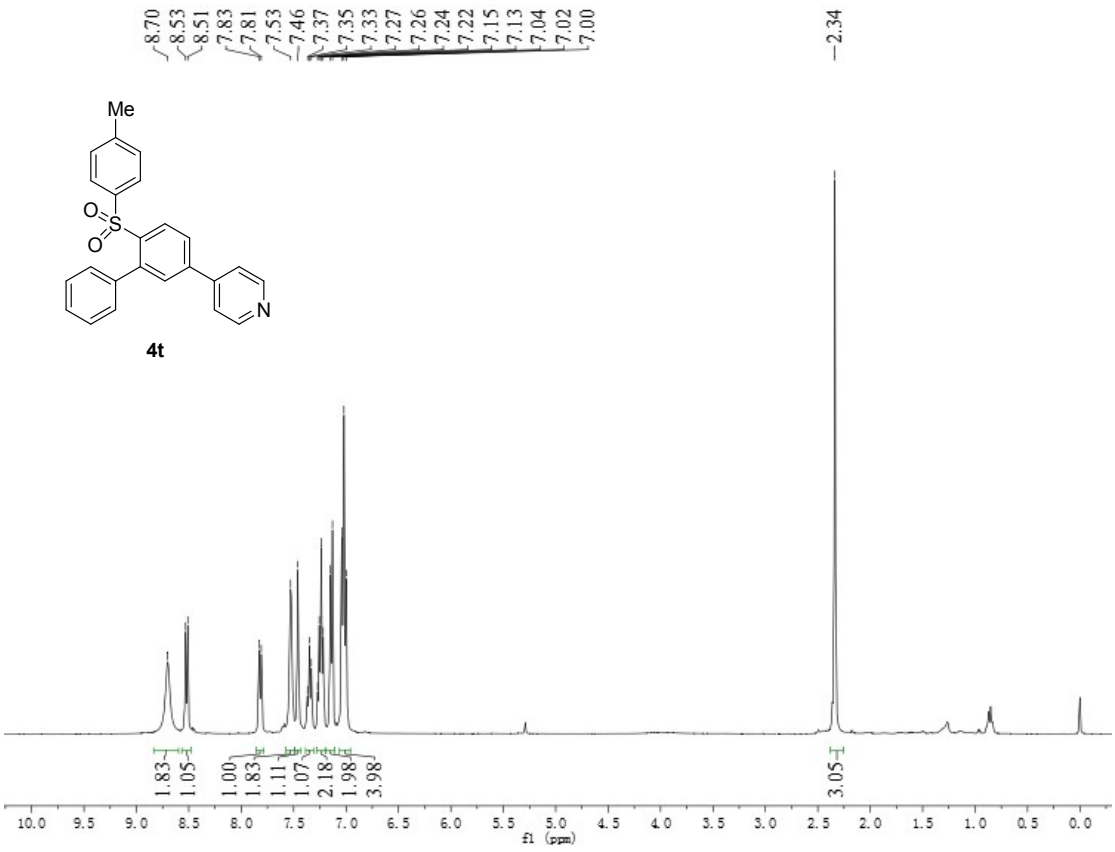

145.56
143.39
142.64
138.93
138.62
138.28
138.13
131.26
130.07
129.28
129.04
128.99
128.60
127.84
127.70
127.36
127.30
125.99
21.55
Me
O
S
O
4q
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.51
8.49
7.79
7.78
7.77
7.76
7.70
7.43
7.42
7.36
7.34
7.32
7.25
7.23
7.21
7.16
7.14
7.05
7.03
7.02
7.00
2.34
Me
O
S
O
CF3
4r
1.00
1.10
4.00
0.99
1.01
2.17
2.00
4.05
3.09
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

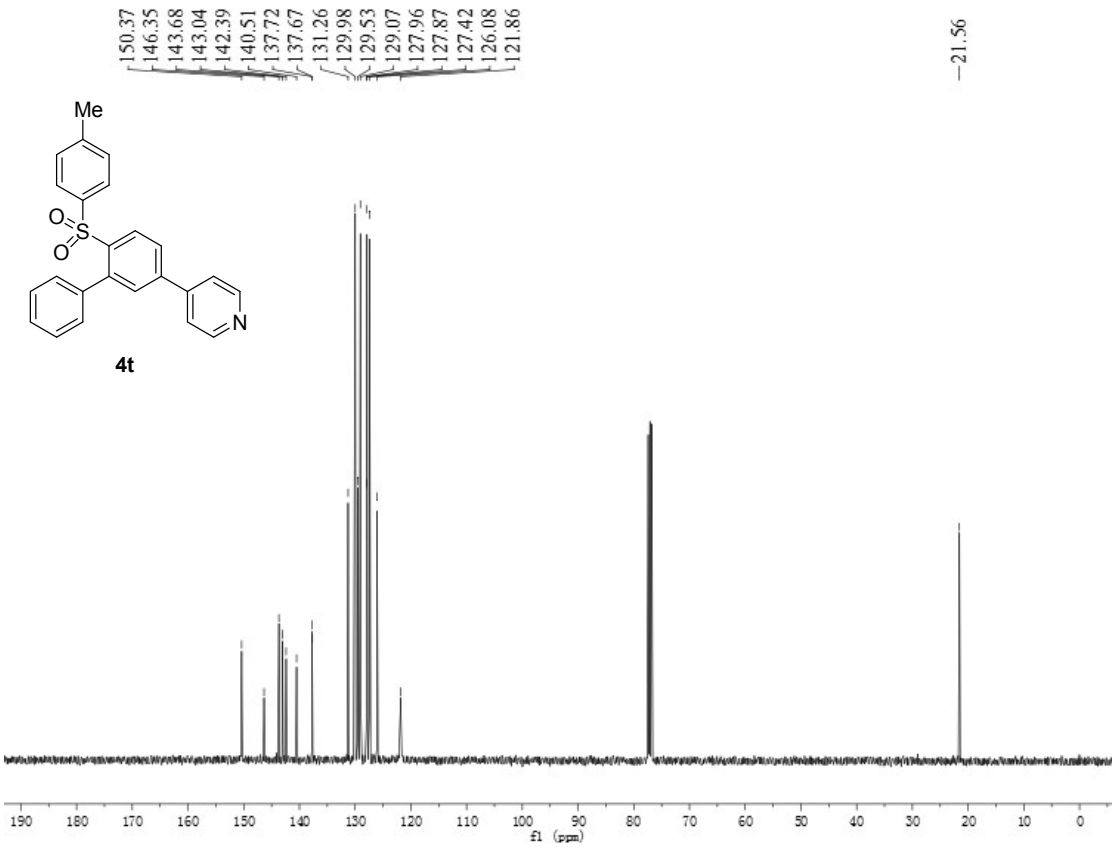

143.98
143.59
142.90
142.48
139.66
137.93
137.86
131.44
130.73
130.41
130.02
129.44
129.05
127.88
127.75
127.39
126.24
126.03
126.00
125.96
125.93
125.40
122.69
21.55
Me
O
S
O
CF3
4r
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.56
8.54
8.53
8.03
7.91
7.88
7.86
7.84
7.82
7.80
7.49
7.47
7.45
7.43
7.41
7.38
7.36
7.35
7.27
7.25
7.23
7.15
7.13
7.05
7.03
7.03
7.01
2.34
Me
O
S
O
CF3
CF3
4s
1.00
1.08
2.15
2.15
0.98
2.01
2.06
4.01
3.00
f1 (ppm)

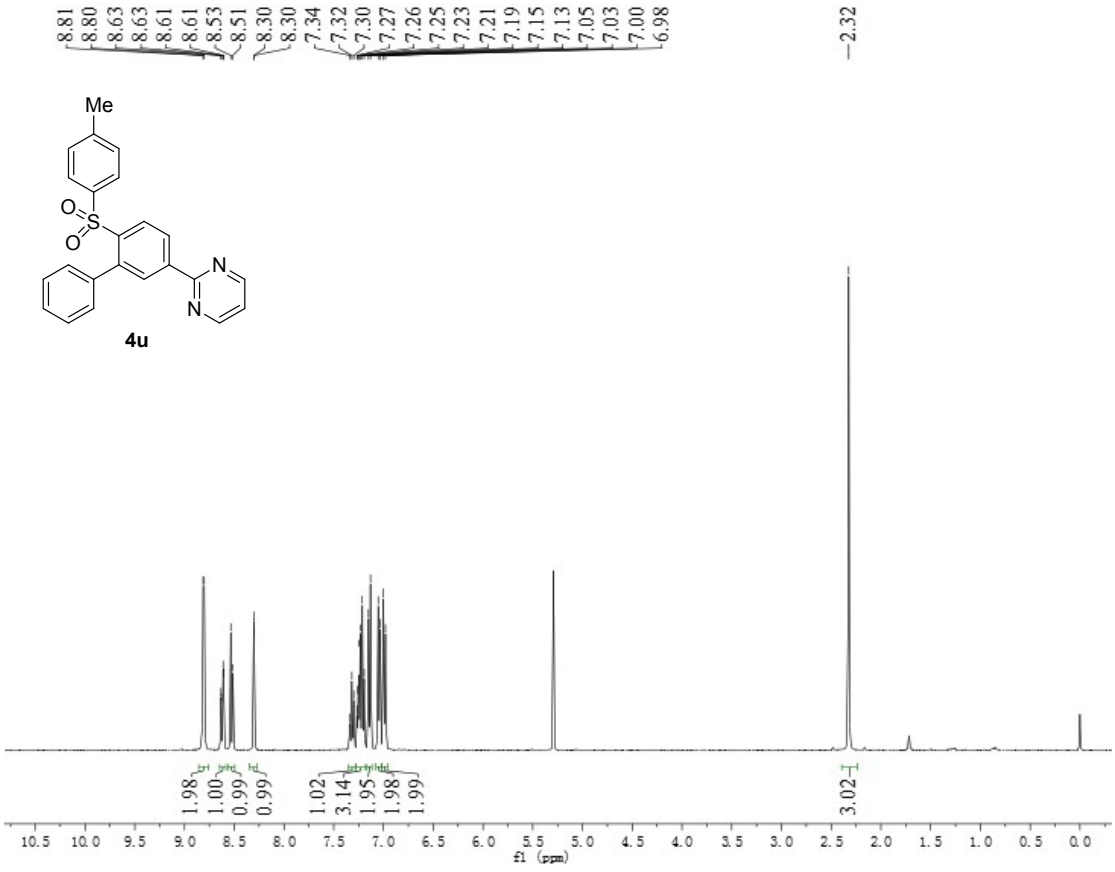

146.27
144.42
143.74
143.26
142.40
141.22
140.46
137.58
133.01
132.68
132.34
132.01
131.42
130.00
129.70
129.19
129.09
127.89
127.48
127.31
126.32
21.55
Me
O
S
O
CF3
CF3
4s
f1 (ppm)

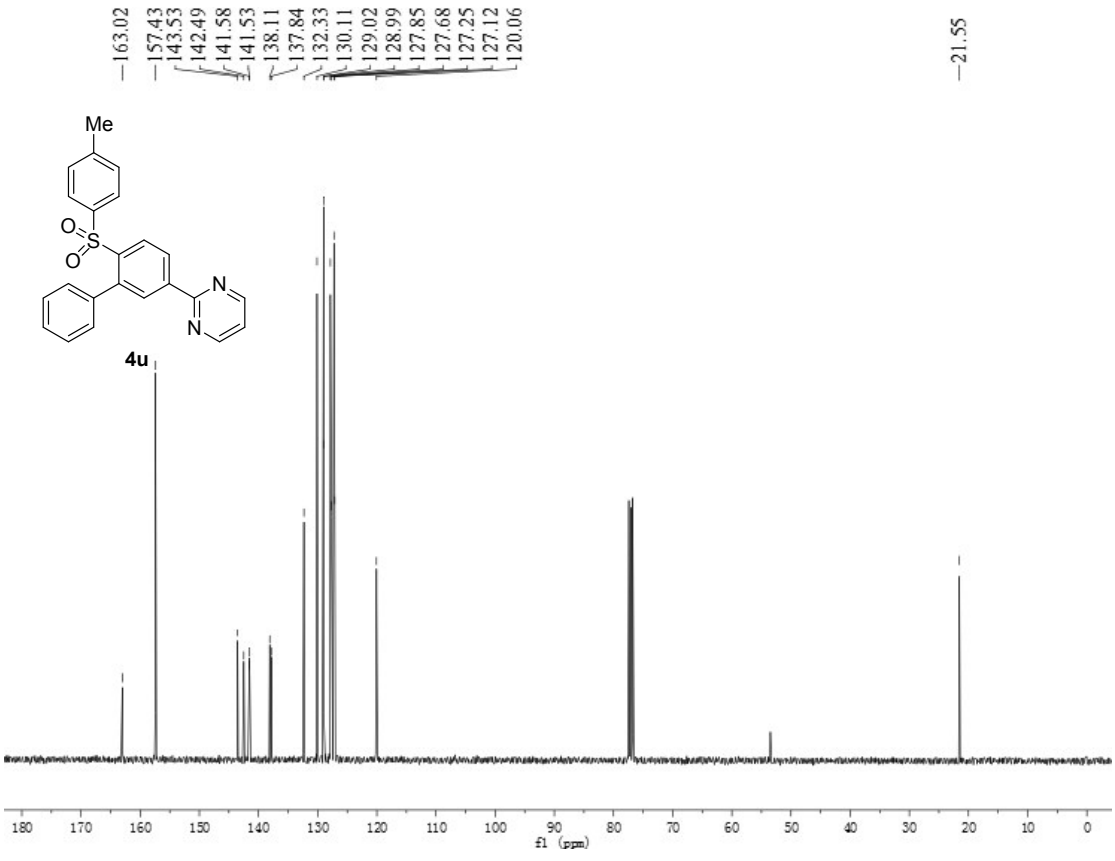

8.70
8.53
8.51
7.83
7.81
7.53
7.46
7.37
7.35
7.33
7.27
7.26
7.24
7.22
7.15
7.13
7.04
7.02
7.00
2.34
Me
O
S
O
N
1.83
1.05
1.00
1.83
1.11
1.07
2.18
1.98
3.98
3.05
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

4t

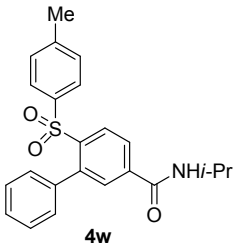

150.37
146.35
143.68
143.04
142.39
140.51
137.72
137.67
131.26
129.98
129.53
129.07
127.96
127.87
127.42
126.08
121.86
21.56
Me
O
S
O
N
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4t

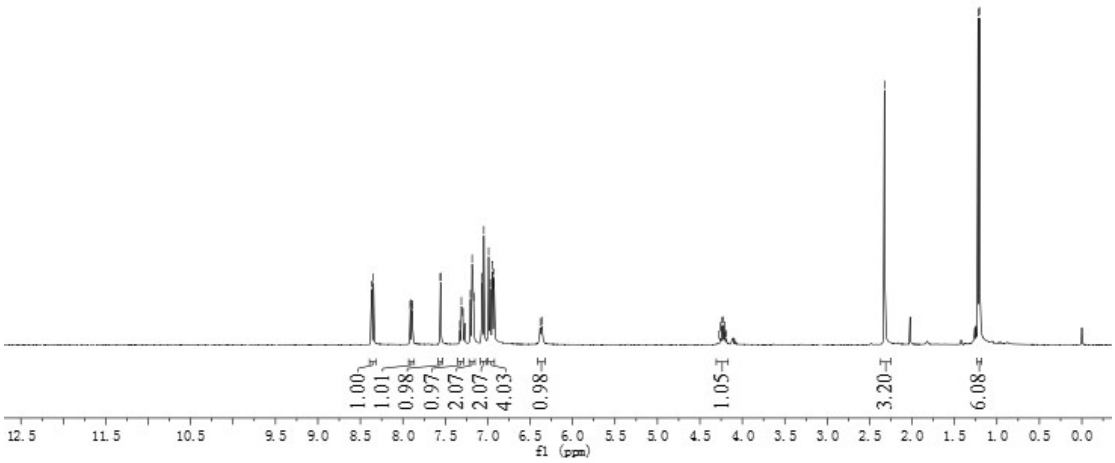

8.81
8.80
8.63
8.63
8.61
8.61
8.53
8.51
8.30
8.30
7.34
7.32
7.30
7.27
7.26
7.25
7.23
7.21
7.19
7.15
7.13
7.05
7.03
7.00
6.98
2.32
Me
O
S
O
N
N
4u
1.98
1.00
0.99
0.99
1.02
3.14
1.95
1.98
1.99
3.02
f1 (ppm)

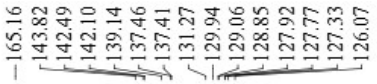

163.02
157.43
143.53
142.49
141.58
141.53
138.11
137.84
132.33
130.11
129.02
128.99
127.85
127.68
127.25
127.12
120.06
21.55
Me
O
S
O
N
N
4u
f1 (ppm)

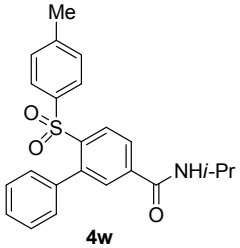

8.37
8.35
7.91
7.91
7.89
7.89
7.56
7.55
7.33
7.31
7.29
7.20
7.18
7.16
7.07
7.05
6.99
6.97
6.94
6.93
6.92
6.38
6.36
4.27
4.26
4.24
4.22
4.21
4.19
2.32
1.22
1.21


Me
O
S
O
NH*i*-Pr
O

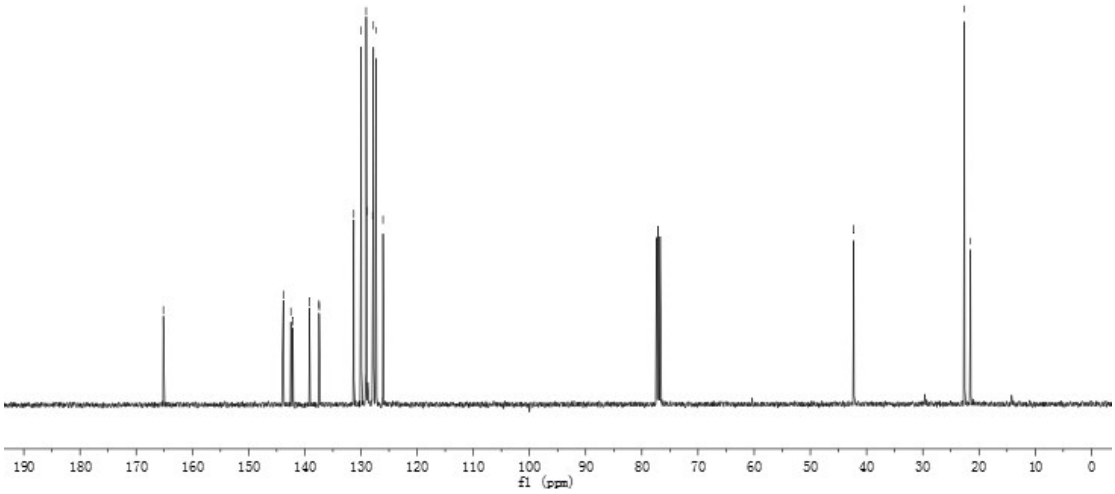
4w

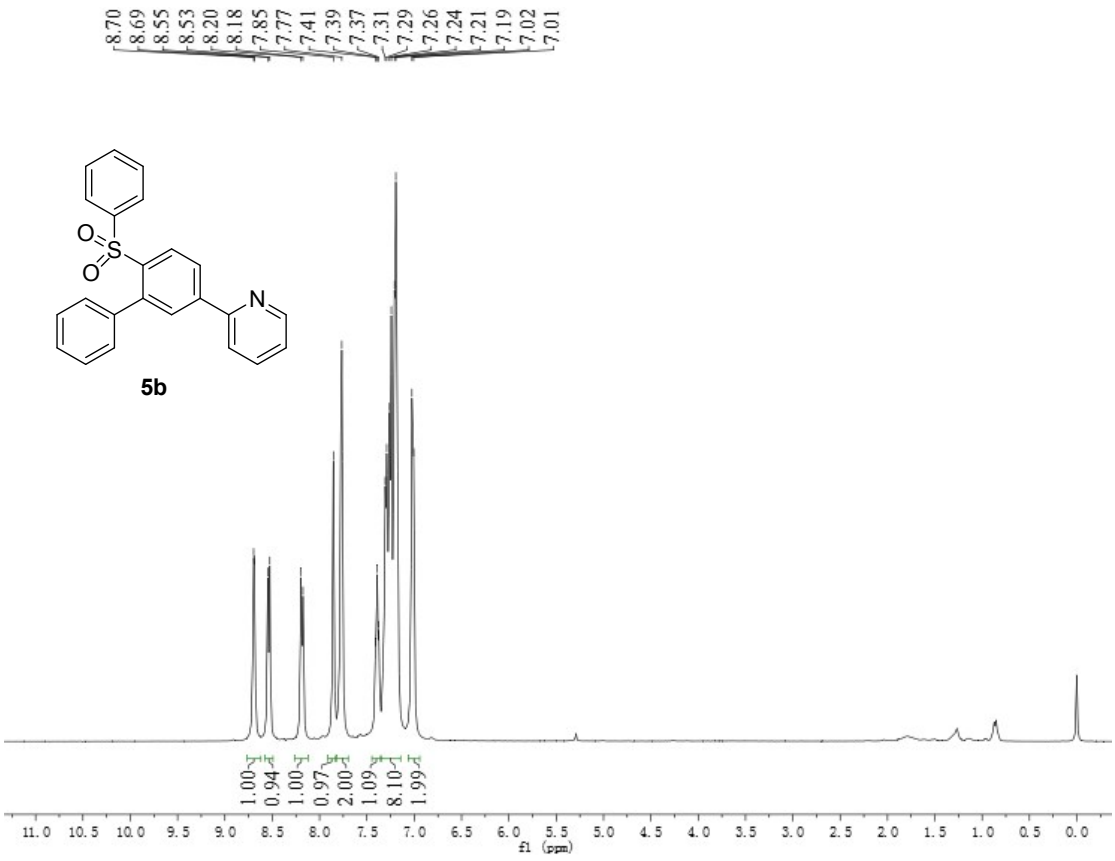

1.00
1.01
0.98
0.97
2.07
2.07
4.03
0.98
1.05
3.20
6.08
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

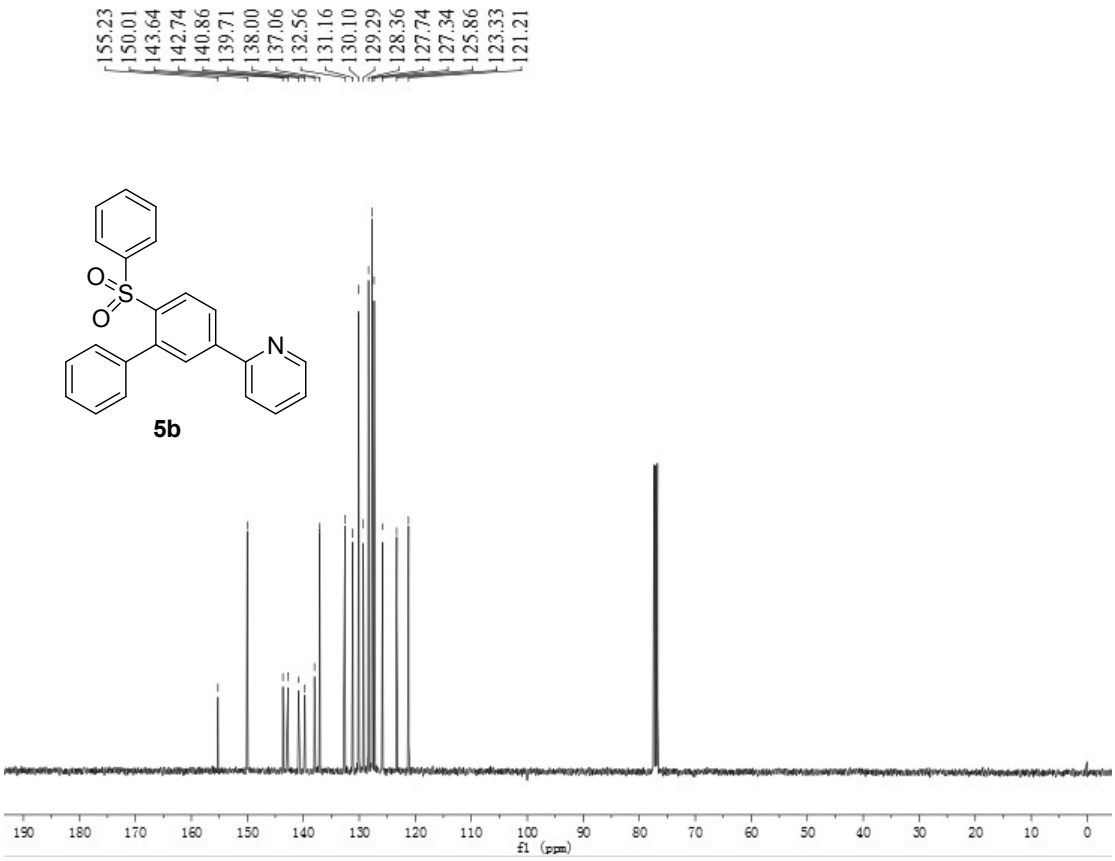

165.16
143.82
142.49
142.10
139.14
137.46
137.41
131.27
129.94
129.06
128.85
127.92
127.77
127.33
126.07
42.29
22.60
21.50

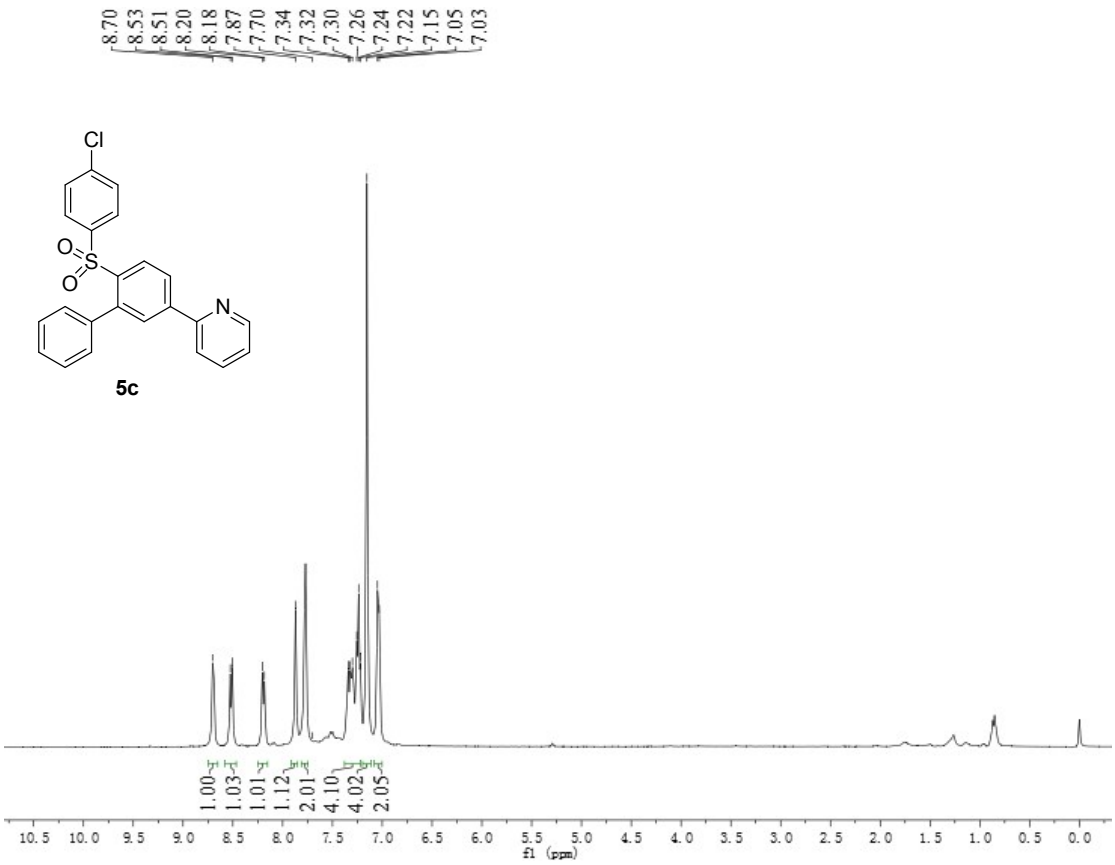

Me
O
S
O
NH*i*-Pr
O

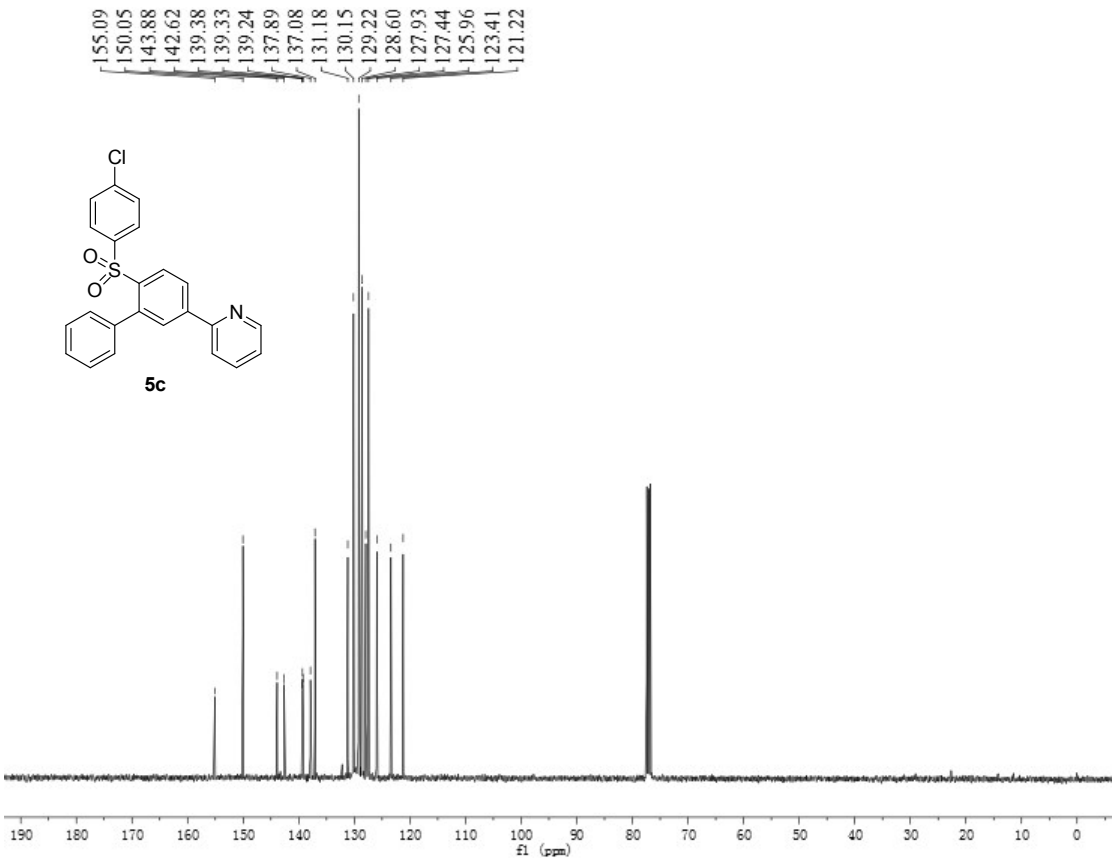
4w

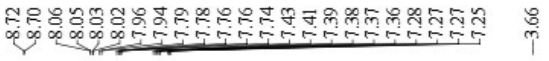

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

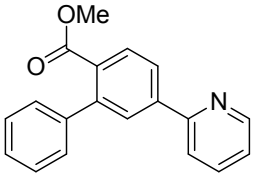

8.70
8.69
8.55
8.53
8.20
8.18
7.85
7.77
7.41
7.39
7.37
7.31
7.29
7.26
7.24
7.21
7.19
7.02
7.01
O
S
O
N
5b
1.00
0.94
1.00
0.97
2.00
1.09
8.10
1.99
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


155.23
150.01
143.64
142.74
140.86
139.71
138.00
137.06
132.56
131.16
130.10
129.29
128.36
127.74
127.34
125.86
123.33
121.21
O
S
O
N
5b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

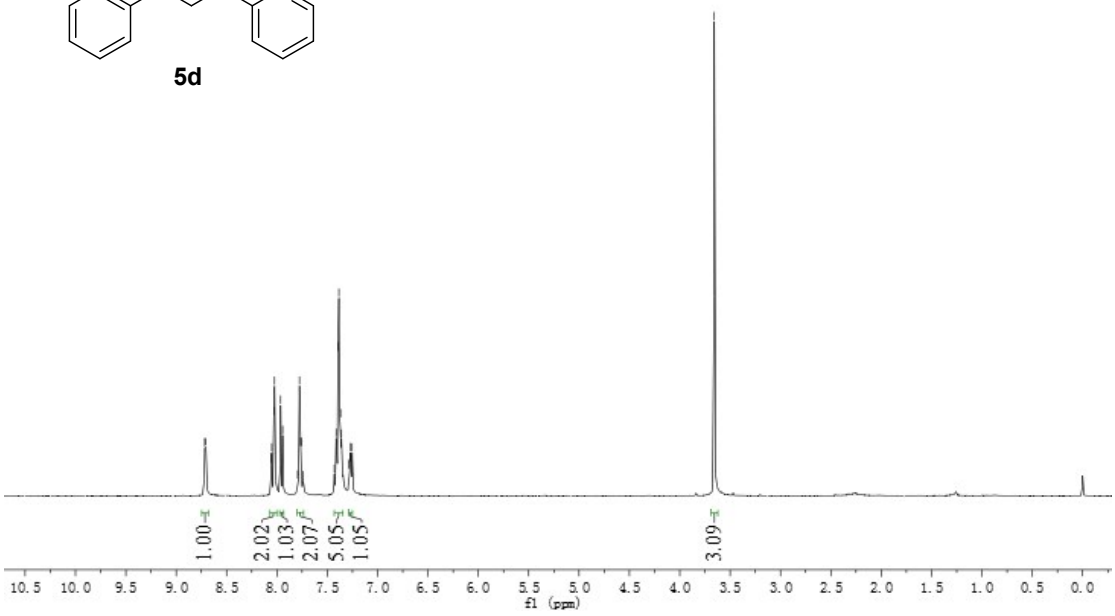

8.70
8.53
8.51
8.20
8.18
7.87
7.70
7.34
7.32
7.30
7.26
7.24
7.22
7.15
7.05
7.03
Cl
O
S
O
N
5c
1.00
1.03
1.01
1.12
2.01
4.10
4.02
2.05
f1 (ppm)

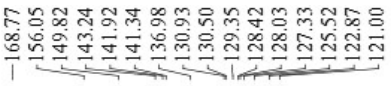

155.09
150.05
143.88
142.62
139.38
139.33
139.24
137.89
137.08
131.18
130.15
129.22
128.60
127.93
127.44
125.96
123.41
121.22
Cl
O
S
O
N
5c
f1 (ppm)


8.72
8.70
8.06
8.05
8.03
8.02
7.96
7.94
7.79
7.78
7.76
7.76
7.74
7.43
7.41
7.39
7.38
7.37
7.36
7.28
7.27
7.27
7.25
3.66

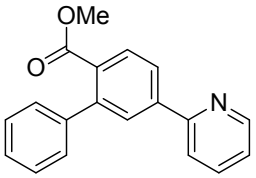

OMe
O
N

5d

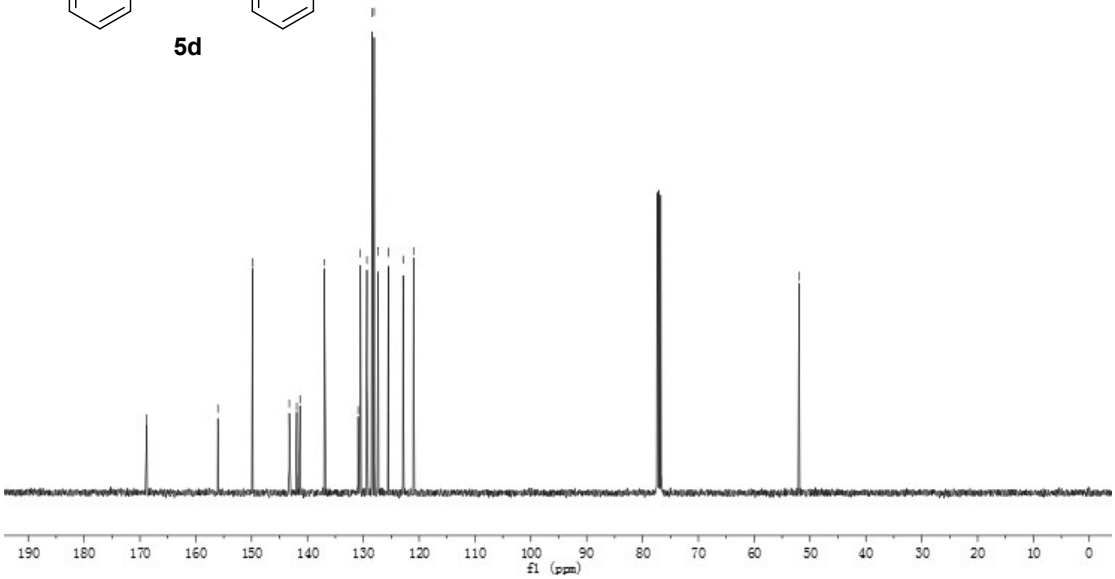

1.00
2.02
1.03
2.07
5.05
1.05
3.09
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


168.77
156.05
149.82
143.24
141.92
141.34
136.98
130.93
130.50
129.35
128.42
128.03
127.33
125.52
122.87
121.00

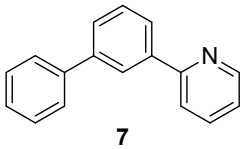

51.97


OMe
O
N

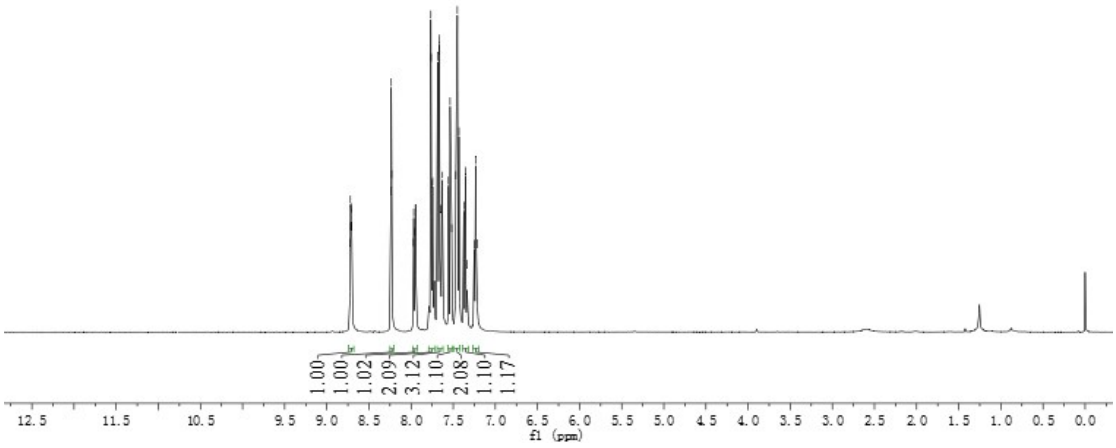
5d


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

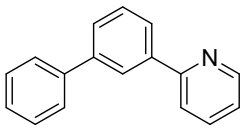

8.72
8.72
8.72
8.71
8.71
8.70
8.24
8.23
8.23
7.97
7.97
7.96
7.77
7.76
7.75
7.74
7.68
7.67
7.66
7.65
7.63
7.56
7.54
7.52
7.47
7.45
7.43
7.37
7.36
7.34
7.25
7.23
7.22


N

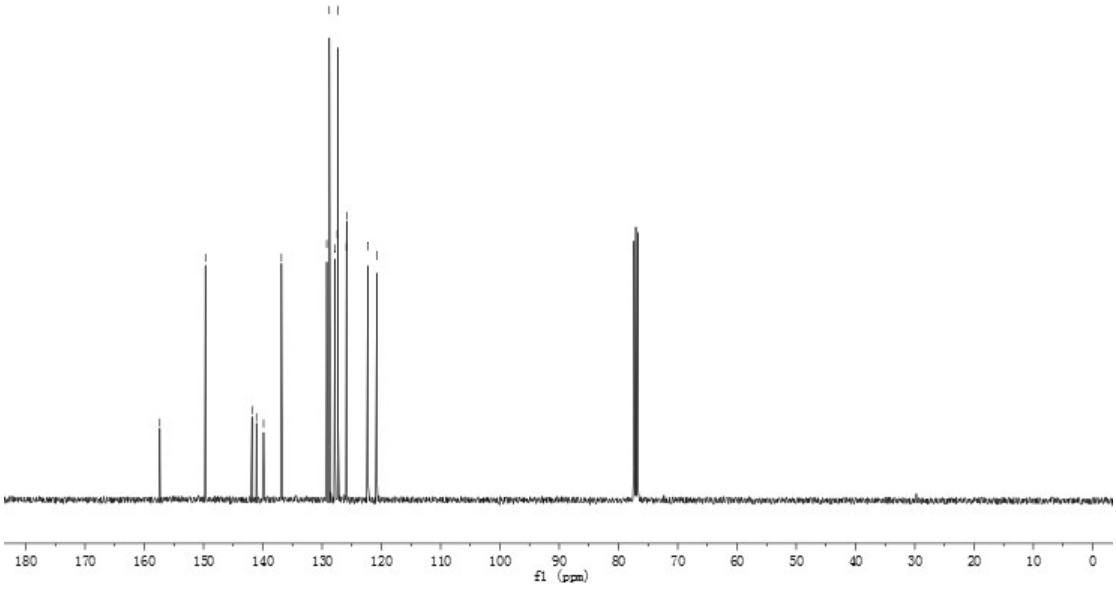
7

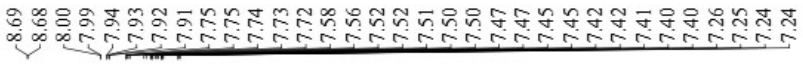

1.00
1.00
1.02
2.09
3.12
1.10
2.08
1.10
1.17
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

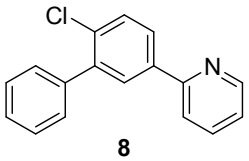

157.39
149.69
141.83
141.08
139.88
136.87
129.22
128.79
127.82
127.45
127.32
125.92
125.87
122.27
120.74


N

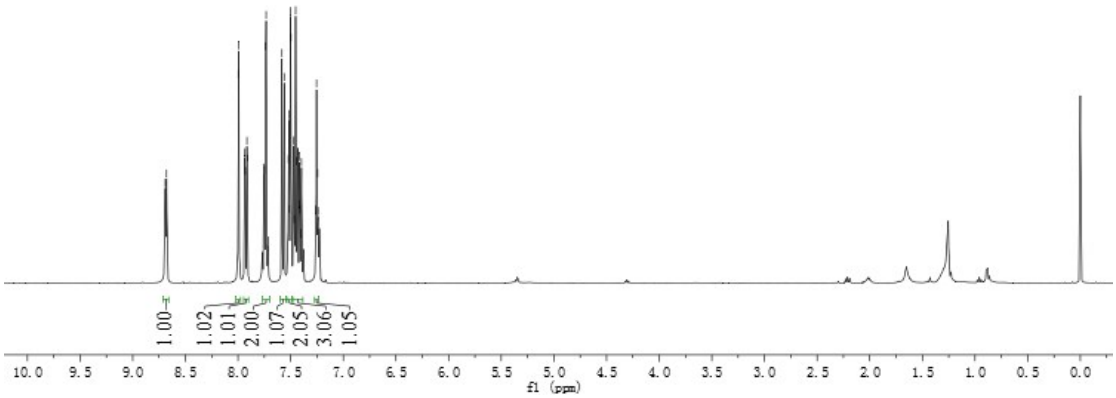
7


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

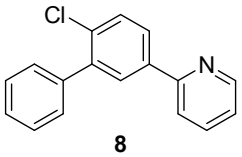

8.69
8.68
8.00
7.99
7.94
7.93
7.92
7.91
7.75
7.75
7.74
7.73
7.72
7.58
7.56
7.52
7.52
7.51
7.50
7.50
7.47
7.47
7.45
7.45
7.42
7.42
7.41
7.40
7.40
7.26
7.25
7.24
7.24


Cl
N

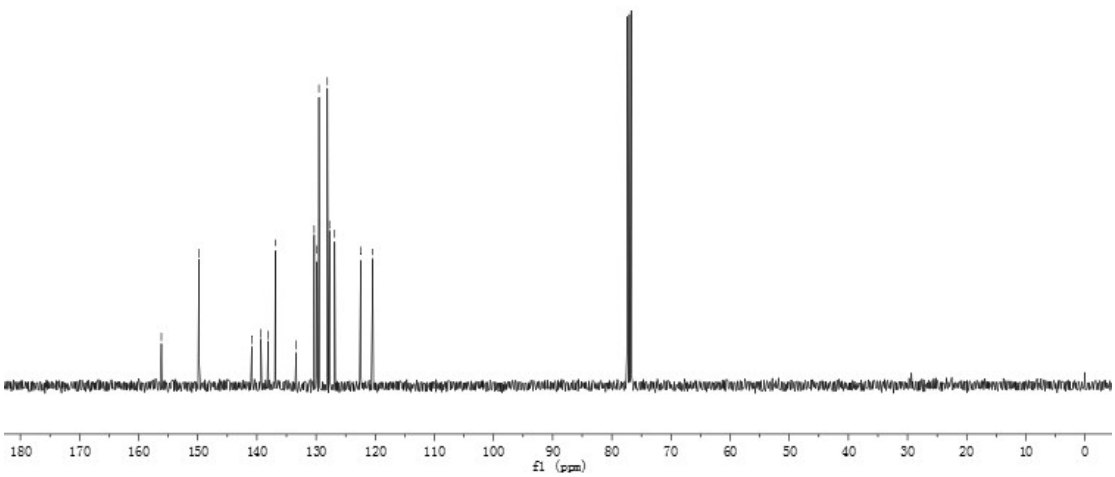
8

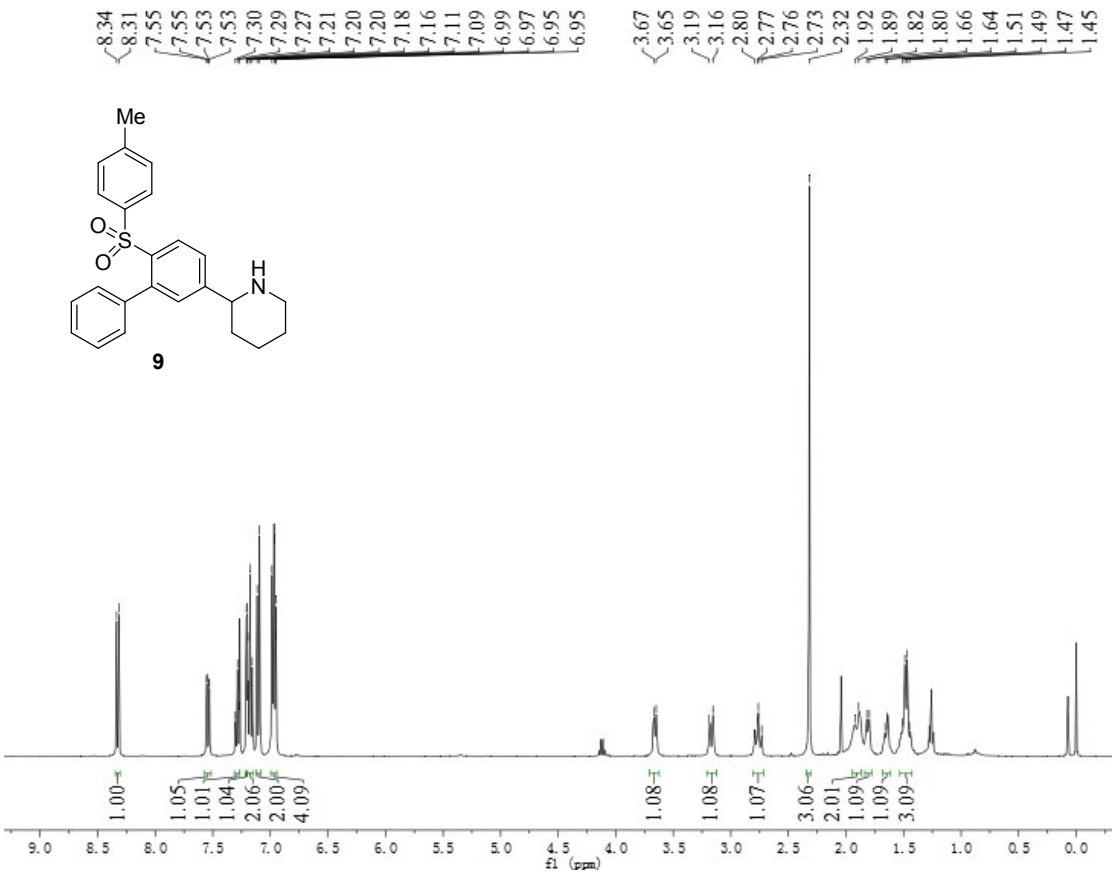

1.00
1.02
1.01
2.00
1.07
2.05
3.06
1.05
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

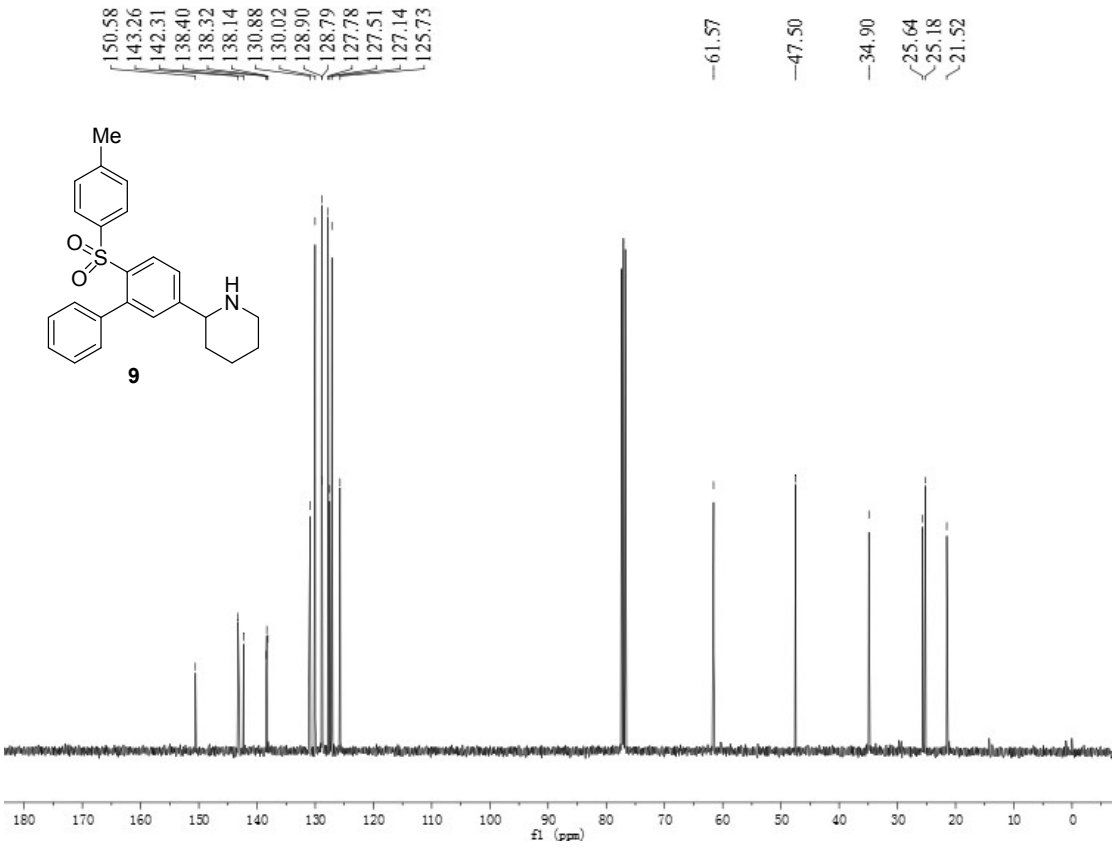

156.15
149.81
140.88
139.32
138.13
136.89
133.38
130.31
129.85
129.52
128.09
127.75
126.86
122.46
120.44


Cl
N

8


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.34
8.31
7.55
7.55
7.53
7.53
7.30
7.29
7.27
7.21
7.20
7.20
7.18
7.16
7.11
7.09
6.99
6.97
6.95
6.95
3.67
3.65
3.19
3.16
2.80
2.77
2.76
2.73
2.32
1.92
1.89
1.82
1.80
1.66
1.64
1.51
1.49
1.47
1.45
Me
O
S
O
H
N
9
1.00
1.05
1.01
1.04
2.06
2.00
4.09
1.08
1.08
1.07
3.06
2.01
1.09
1.09
3.09
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


150.58
143.26
142.31
138.40
138.32
138.14
130.88
130.02
128.90
128.79
127.78
127.51
127.14
125.73
61.57
47.50
34.90
25.64
25.18
21.52
Me
O
S
O
H
N
9
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)