Supporting Information

Nickel-Catalyzed Regioselective Arylation of Aromatic amides with Aryl Iodides Enabled by *N*,*O*-Bidendate Directing Group

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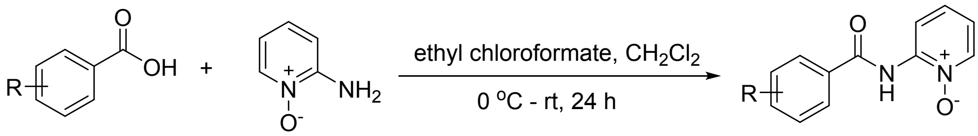
1. Experimental Section

All of 2-aminopyridine 1-oxide was synthesized according to the known method [R. Adams, S. Miyano, *J. Am. Chem. Soc.* **1954**, *76*, 2785]. The aromatic amides were synthetized from the corresponding aromatic acids and 2-aminopyridine 1-oxide (see the following experimental procedure for detail).

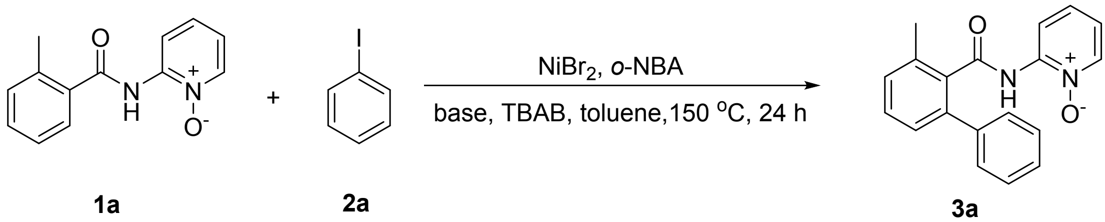
1.1 General Procedure for the Preparation of Amide Substrates



To a 100 mL reaction flask in ice-water bath were added CH_2Cl_2 (30 mL), aromatic acid (17.5 mmol), NEt_3 (3.14 g, 35.0 mmol), Ethyl chloroformate (1.90 g, 17.5 mmol), 2-aminopyridine 1-oxide (1.00 g, 10.0 mmol). The reaction mixture was gradually warmed to room temperature, and stirred for 24 hours. The reaction was quenched with water (50 mL) and extracted with CH_2Cl_2 (3 × 20 mL). The mixture was dried by $MgSO_4$ and filtered and concentrated under reduced pressure to yield a crude product, which was purified by flash chromatography (silica gel, ethyl acetate / ethanol = 6:1 to 1:1), affording the desired product as a white to yellow solid.

1.2 Optimization of reaction conditions

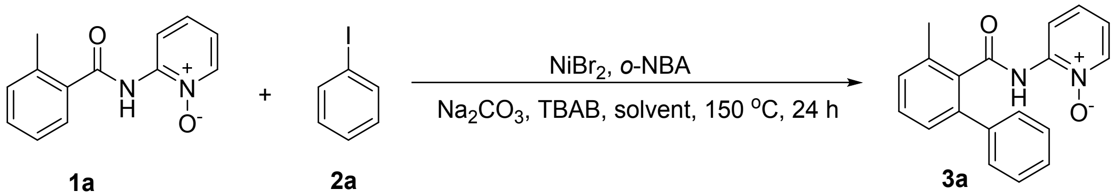
1.2.1 Optimization of the base on the model reaction[a]



entry	base	yield [%]b
1	Na_2CO_3	71
2	K_2CO_3	45
3	Cs_2CO_3	60
4	$KHCO_3$	43
5	$NaHCO_3$	40
6	CH_3COONa	45
7	K_3PO_4	35
8	^{t}BuOLi	n.r.
9	KF	n.r.
10	pyridine	n.r.

[a]Conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), $NiBr_2$ (0.04 mmol, 20 mol%), *o*-NBA (0.08 mmol, 40 mol%), base (0.4 mmol, 2.0 equiv), TBAB (0.8 mmol, 4.0 equiv), toluene (1.5 mL), 150 °C, N_2 atmosphere, 24 h. [b]Isolated yields. *o*-NBA = *o*-nitrobenzoic acid. n.r. = no reaction.

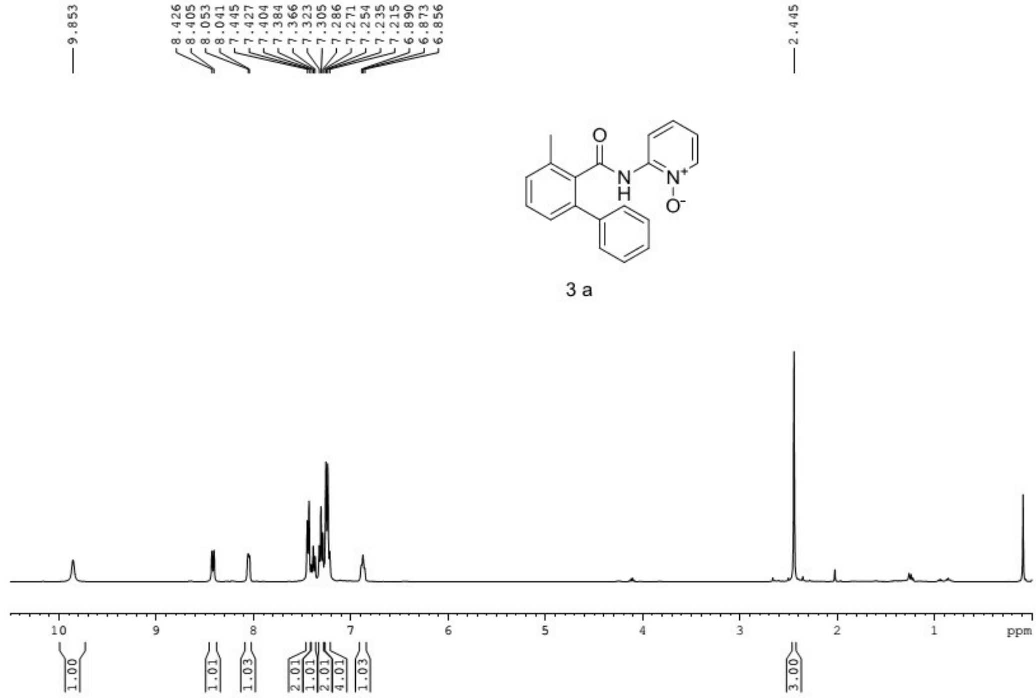
1.2.2 Optimization of the solvent on the model reaction[a]

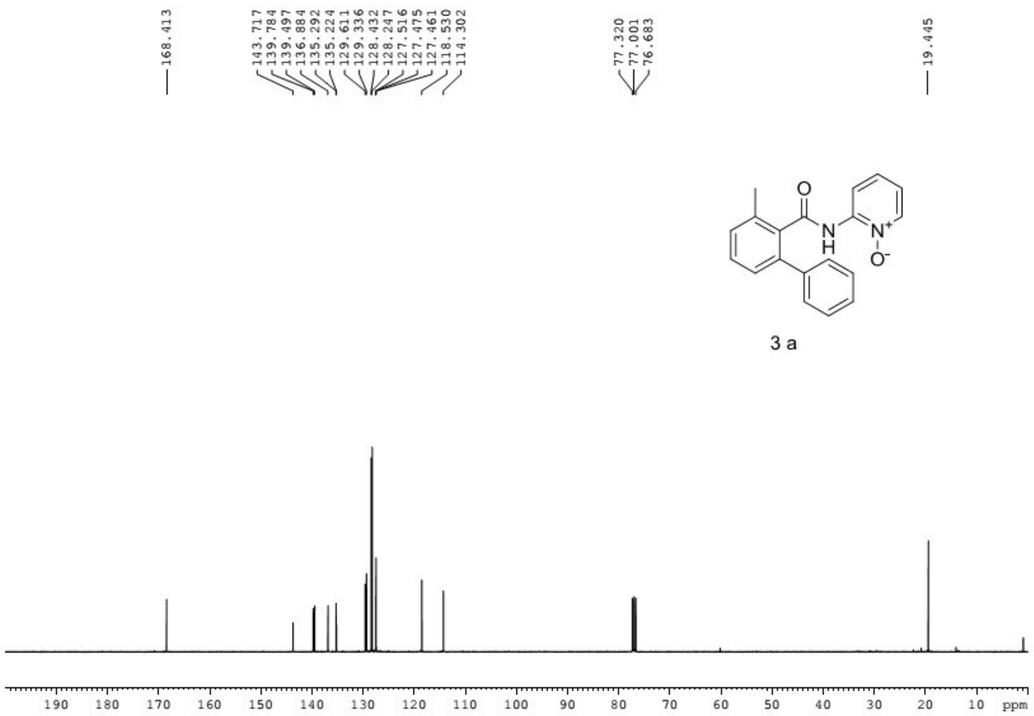


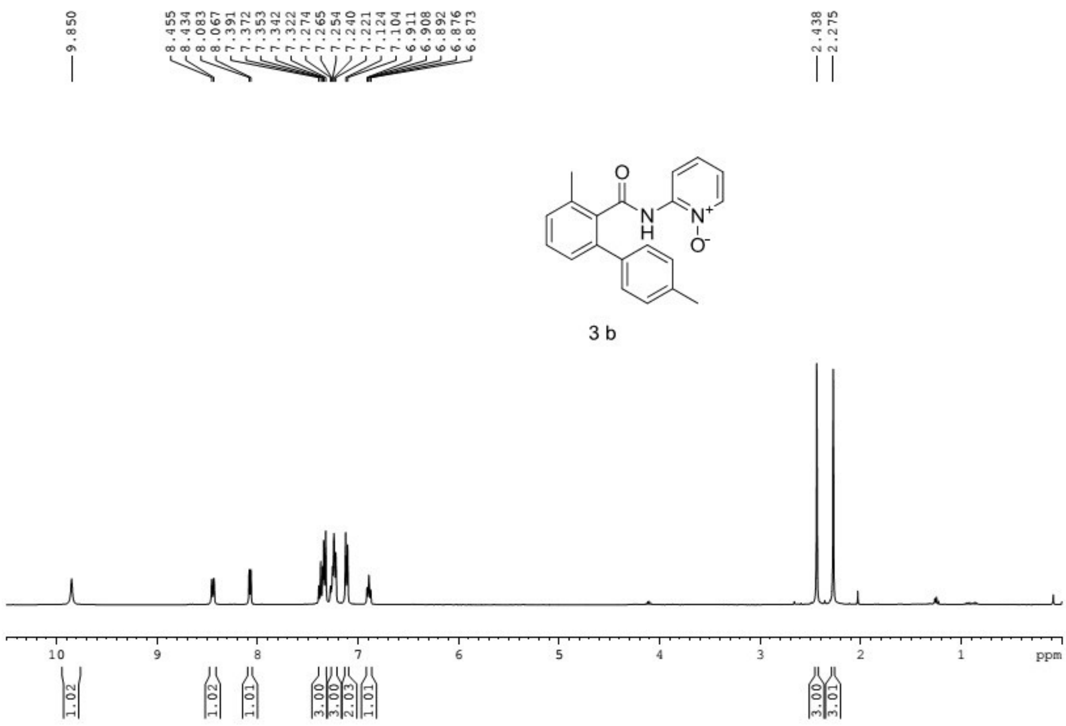
entry	solvent	yield [%][b]
1	toluene	71
2	dioxane	59
3	THF	44
4	CH_3CH_2OH	36
5	$PhCF_3$	55
6	DCE	40
7	DMF	42
8	DMA	37
9	DMSO	34
10	NMP	trace

[a]Conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), $NiBr_2$ (0.04 mmol, 20 mol%), *o*-NBA (0.08 mmol, 40 mol%), Na_2CO_3 (0.4 mmol, 2.0 equiv), TBAB (0.8 mmol, 4.0 equiv), solvent (1.5 mL), 150 °C, N_2 atmosphere, 24 h. [b]Isolated yields. *o*-NBA = *o*-nitrobenzoic acid. DCE = 1,2-dichloroethane. DMA = Dimethylacetamide. NMP = 1-Methyl-2-pyrrolidinone.

2. ^{1}H and ^{13}C NMR spectra



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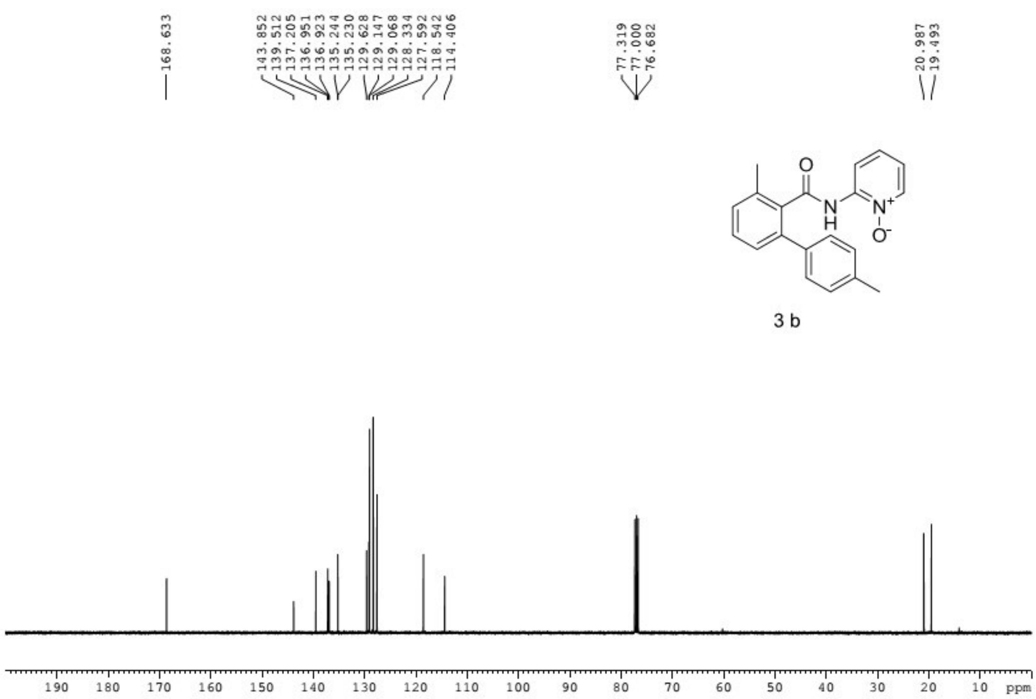
7.254
7.240
7.221
7.124
7.104
6.911
6.908
6.892
6.876
6.873
2.438
2.275
3 b
ppm
1.02
1.02
1.01
3.00
3.00
2.03
1.01
3.00
3.01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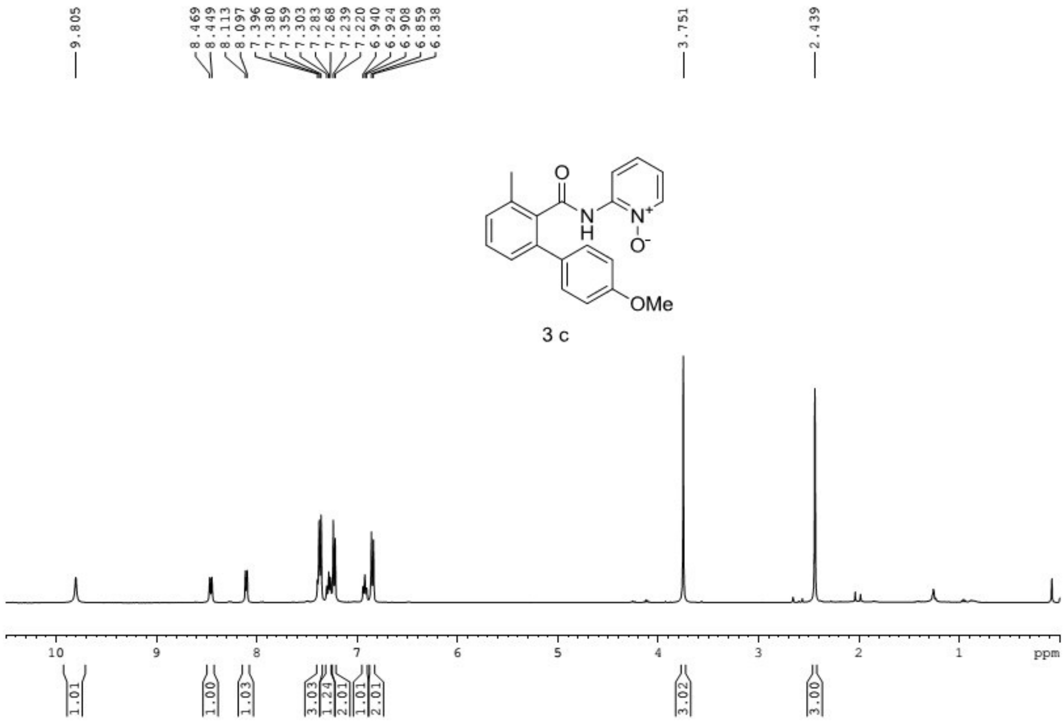
77.000
76.682
20.987
19.493
3 b
ppm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2.439
O
N
H
N+
O-
OMe
3 c
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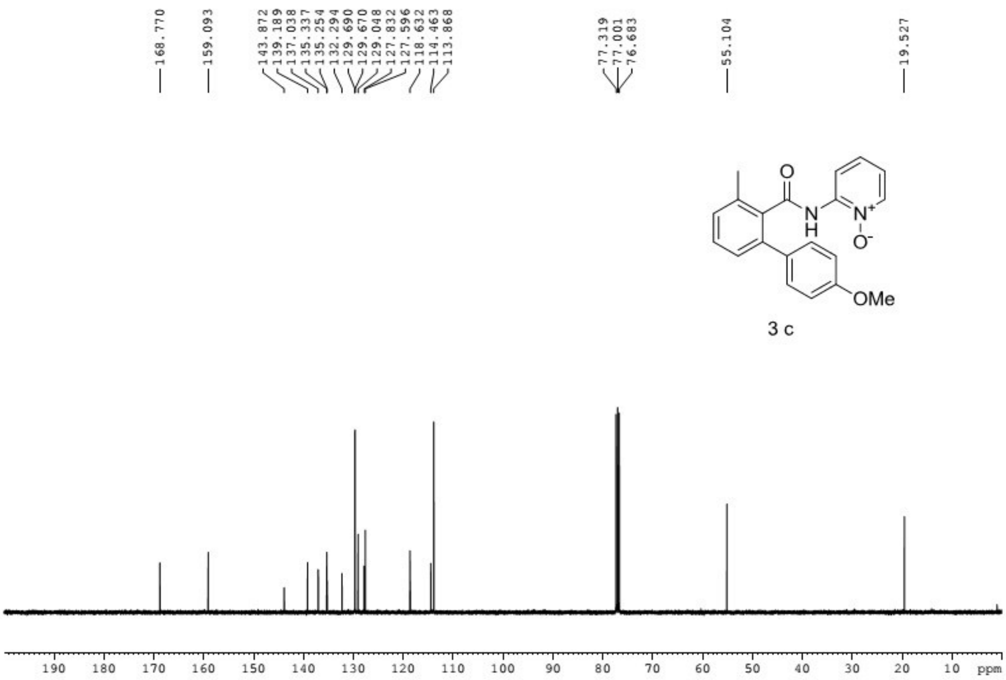
1.01
1.00
1.03
3.03 1.24 2.01 1.01 2.01
3.02
3.00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77.319 77.001 76.683
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N
H
N+
O-
OMe
3 c
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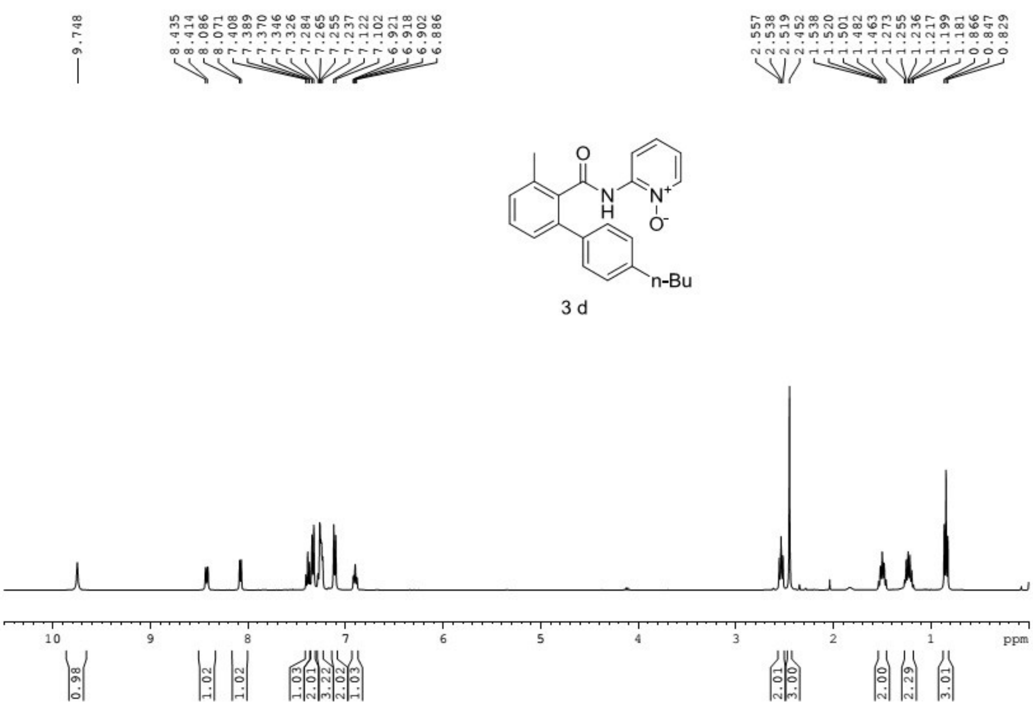
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7.237
7.122
7.102
6.921
6.918
6.902
6.886
2.557
2.538
2.519
2.452
1.538
1.520
1.501
1.482
1.463
1.273
1.255
1.236
1.217
1.199
1.181
0.866
0.847
0.829
O
N
H
N+
O-
n-Bu
3 d
10
9
8
7
6
5
4
3
2
1
ppm
0.98
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2.01
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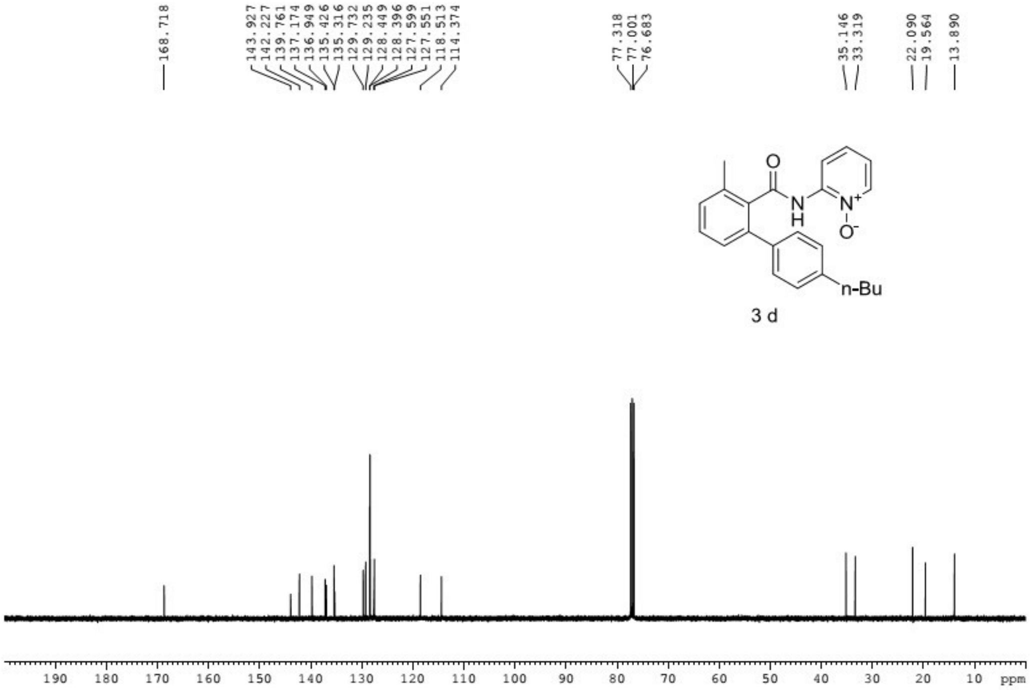
3.01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129.235
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128.396
127.599
127.551
118.513
114.374
77.318
77.001
76.683
35.146
33.319
22.090
19.564
13.890
O
N
H
N+
O-
n-Bu
3 d
190
180
170
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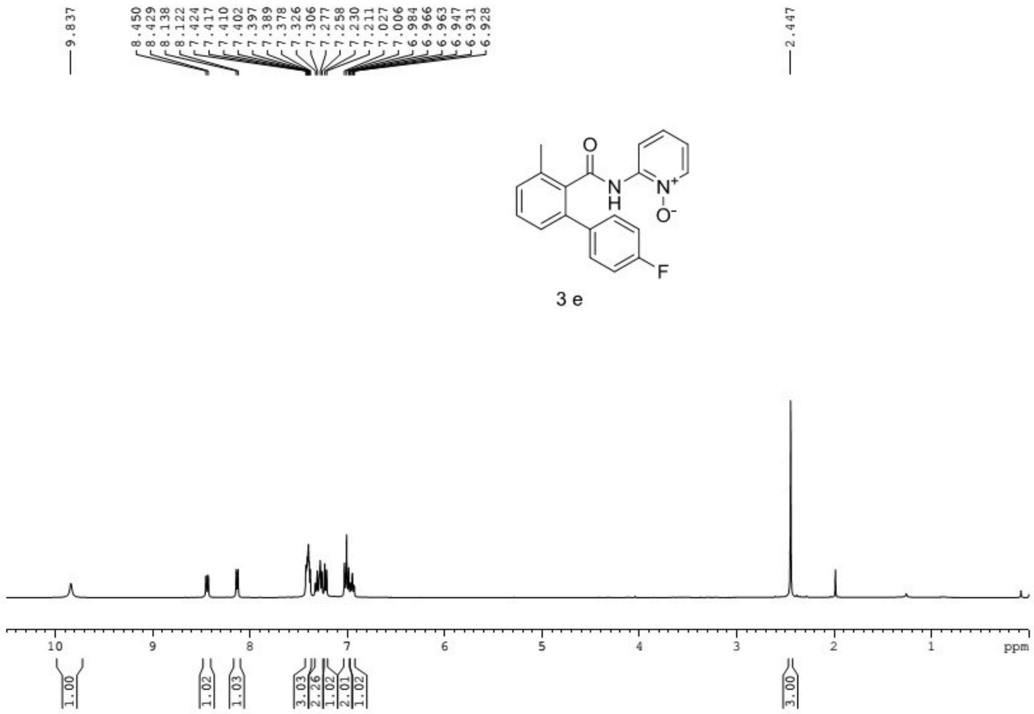
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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2.447
O
N
H
N+
O-
F
3 e
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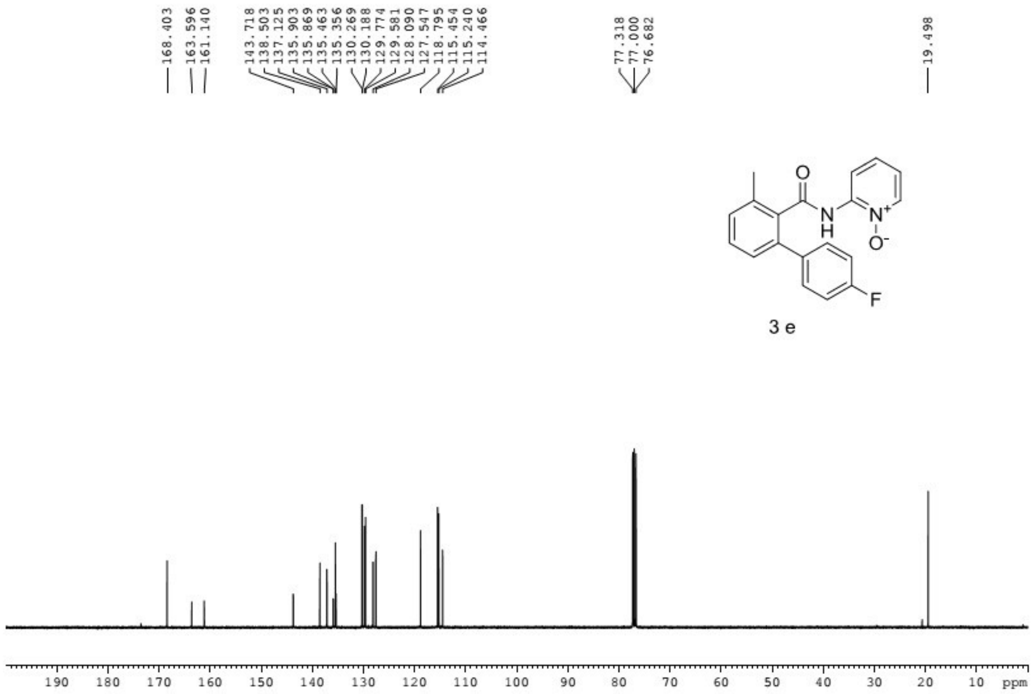
1.00 1.02 1.03 3.03 2.26 1.02 2.01 1.02 3.00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77.318 77.000 76.682
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N
H
N+
O-
F
3 e
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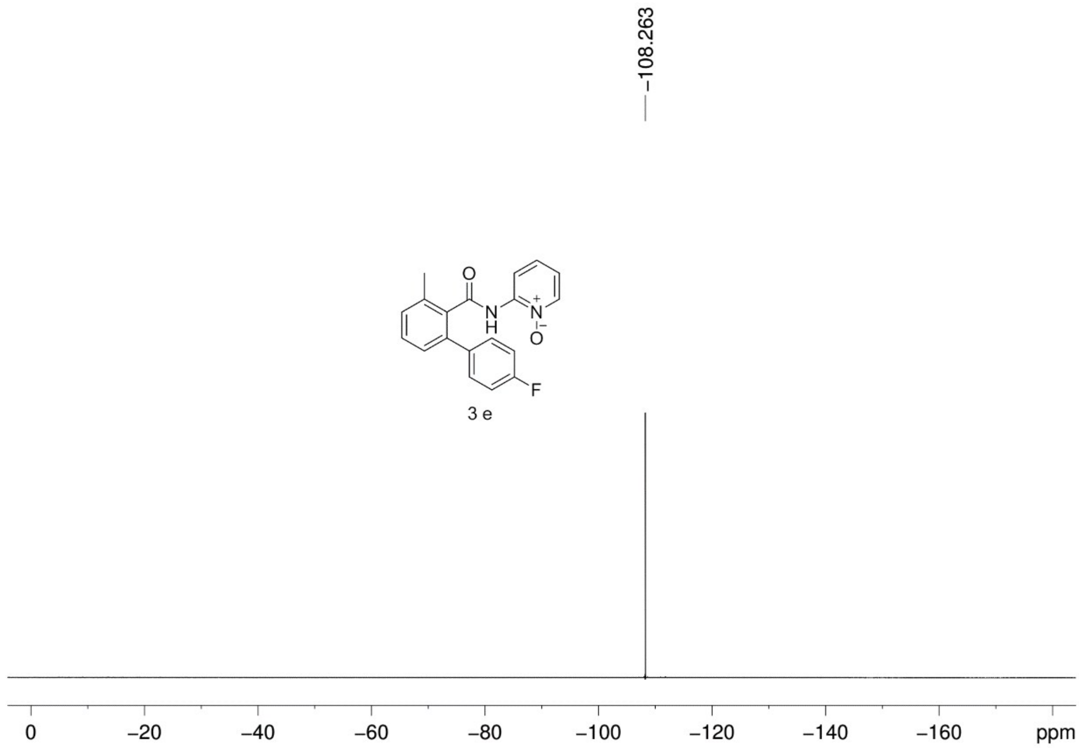

−108.263
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N
H
N
+
O
−
F
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−100
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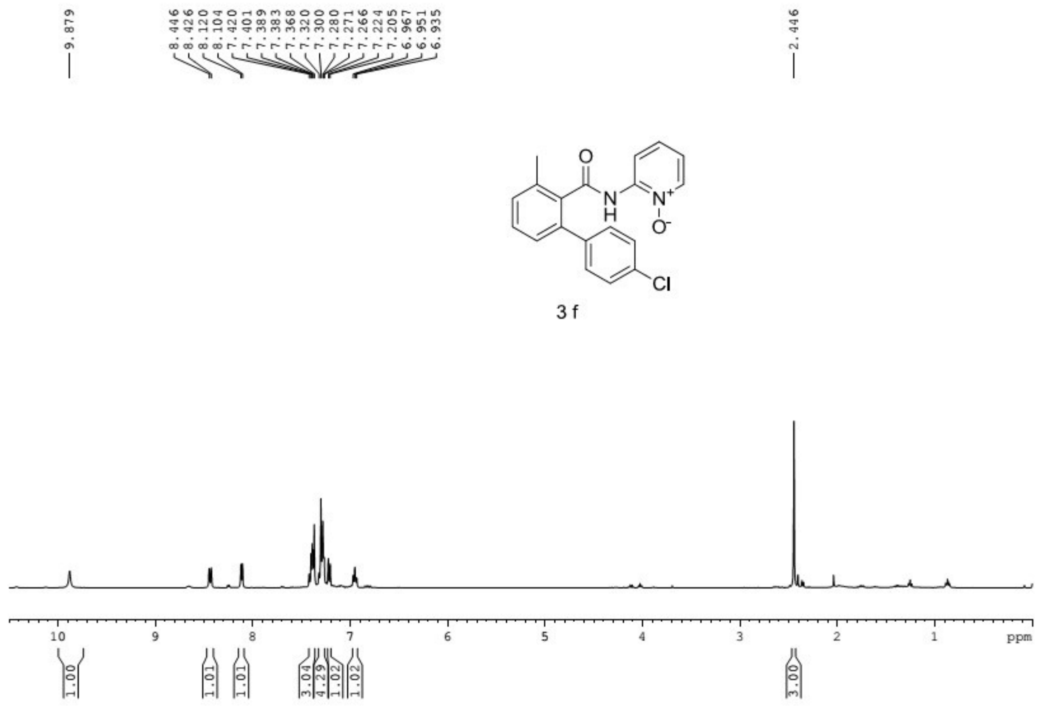
−140
−160
ppm

3 e



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7.383
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7.271
7.266
7.224
7.205
6.967
6.951
6.935
2.446
O
N
H
N
+
O
−
Cl
10
9
8
7
6
5
4
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ppm
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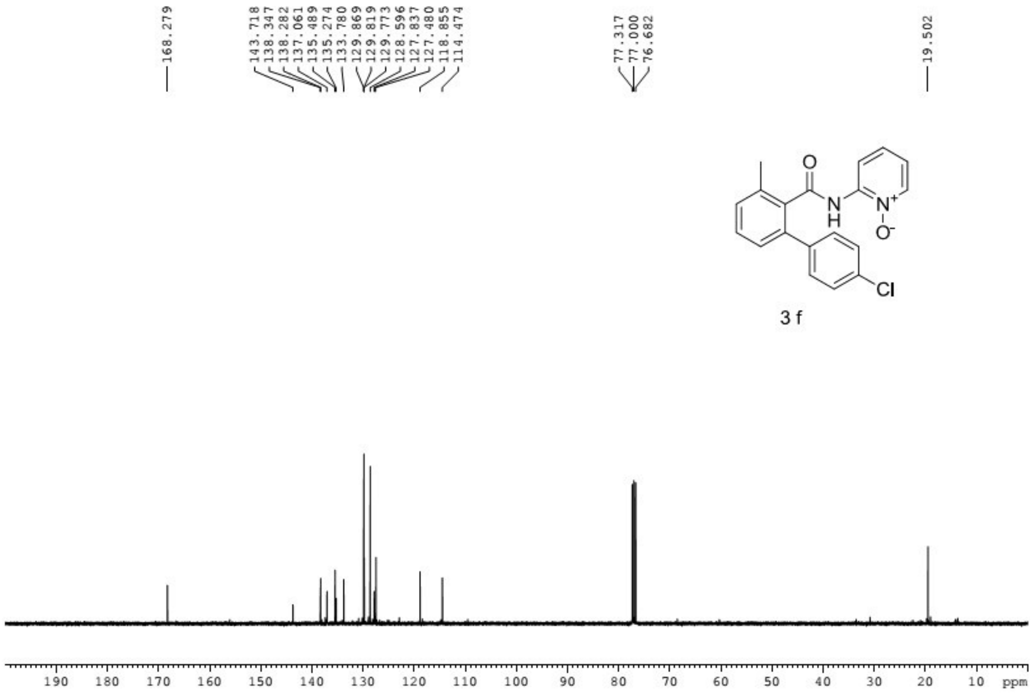
1.02
3.00

3 f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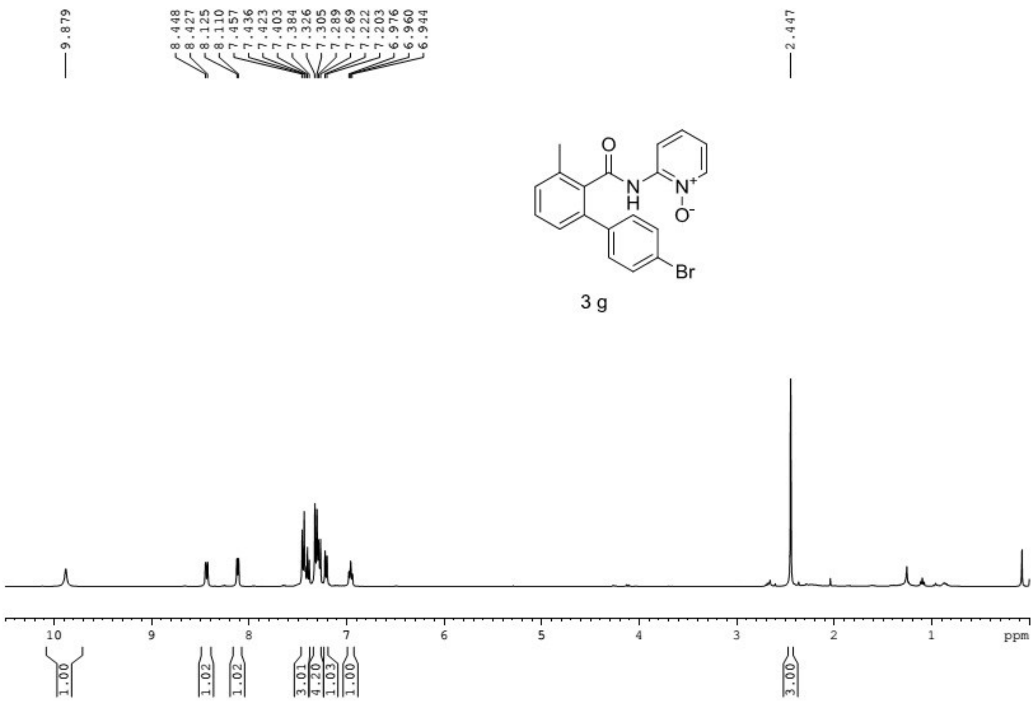
127.837
127.480
118.855
114.474
77.317
77.000
76.682
19.502
O
N
H
N+
O-
Cl
3 f
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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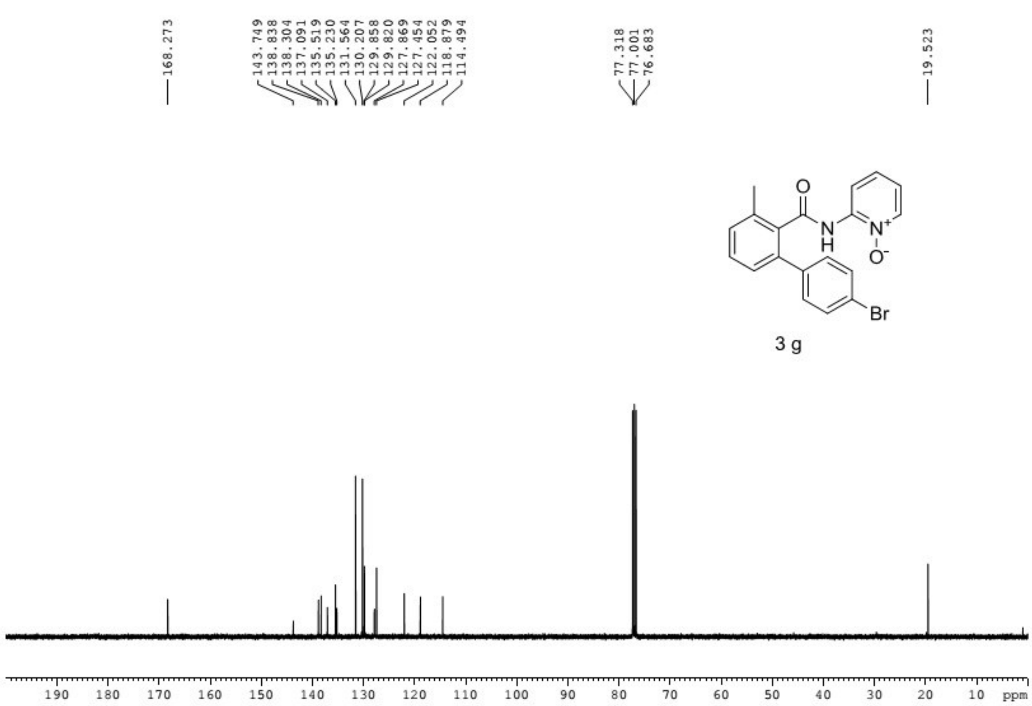
7.436
7.423
7.403
7.384
7.326
7.305
7.289
7.269
7.222
7.203
6.976
6.960
6.944
2.447
O
N
H
N+
O-
Br
3 g
10 9 8 7 6 5 4 3 2 1 ppm
1.00
1.02
1.02
3.01
4.20
1.03
1.00
3.00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127.454
122.052
118.879
114.494
77.318
77.001
76.683
19.523
O
N
H
N+
O-
Br
3 g
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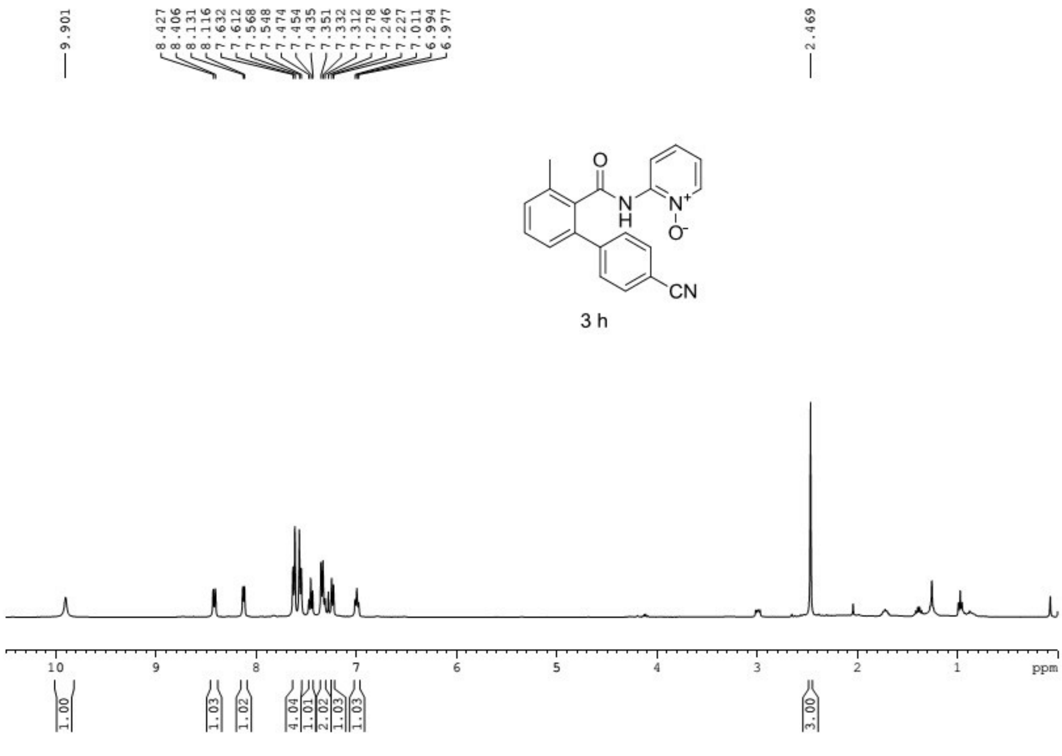
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7.332
7.312
7.278
7.246
7.227
7.011
6.994
6.977
2.469
O
N
H
N+
O-
CN
3 h
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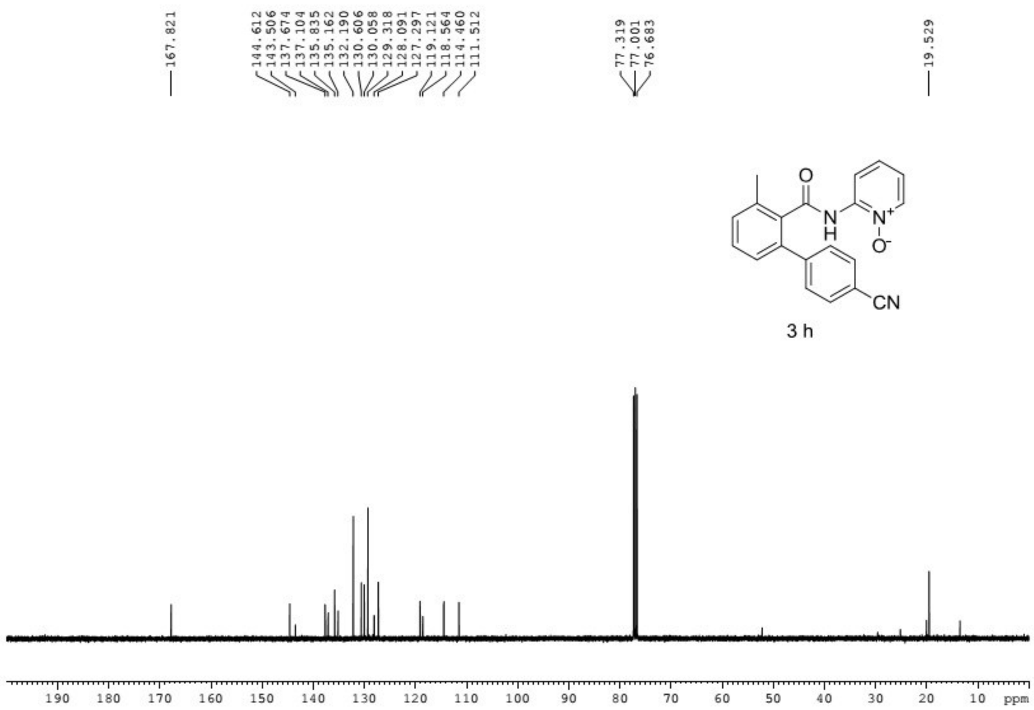
4.04
1.01
2.02
1.03
1.03
3.00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127.297
119.121
118.564
114.460
111.512
77.319
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76.683
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O
N
H
N+
O-
CN
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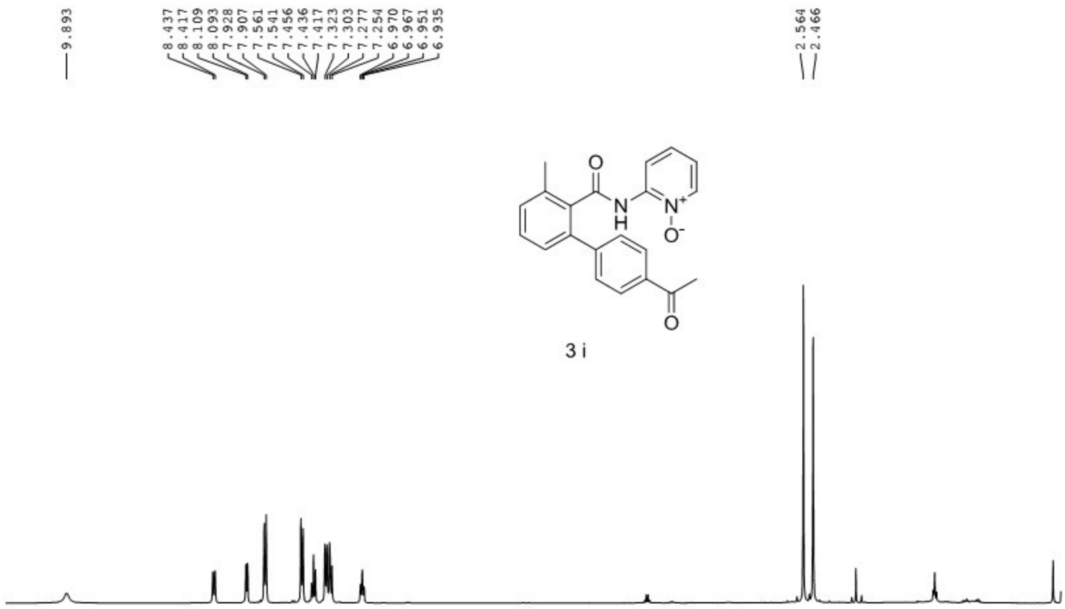
3 h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7.323
7.303
7.277
7.254
6.970
6.967
6.951
6.935
2.564
2.466
O
N
H
N+
O-
O
10 9 8 7 6 5 4 3 2 1 ppm
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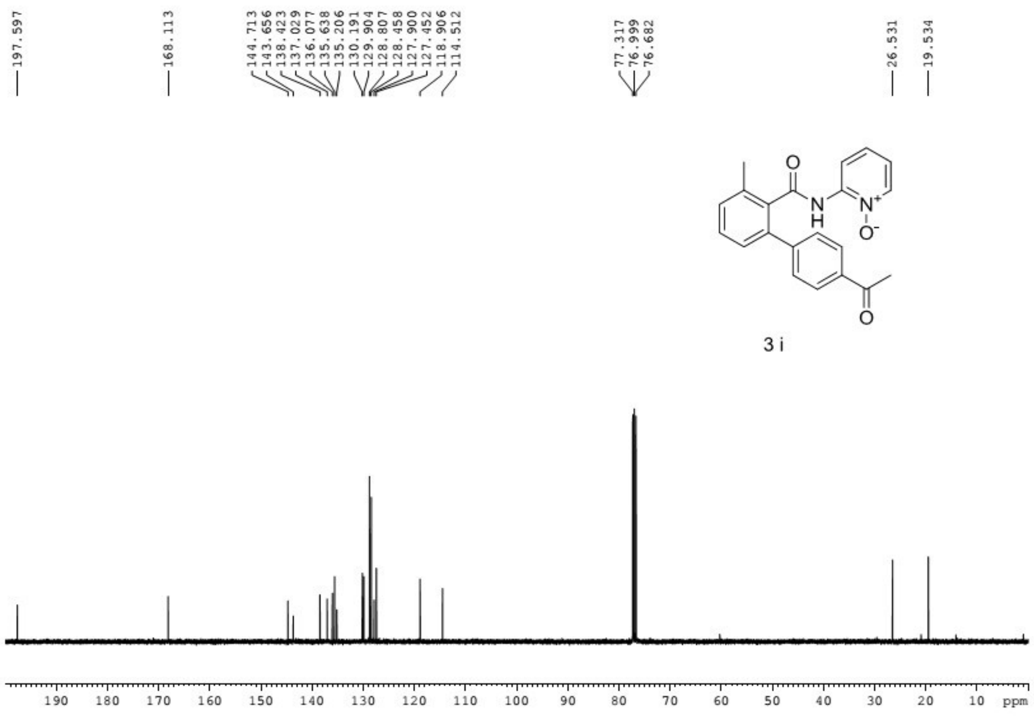
1.03
1.02
2.03
2.02
1.02
3.28
1.01
3.00
3.02

3 i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77.317 76.999 76.682
26.531
19.534
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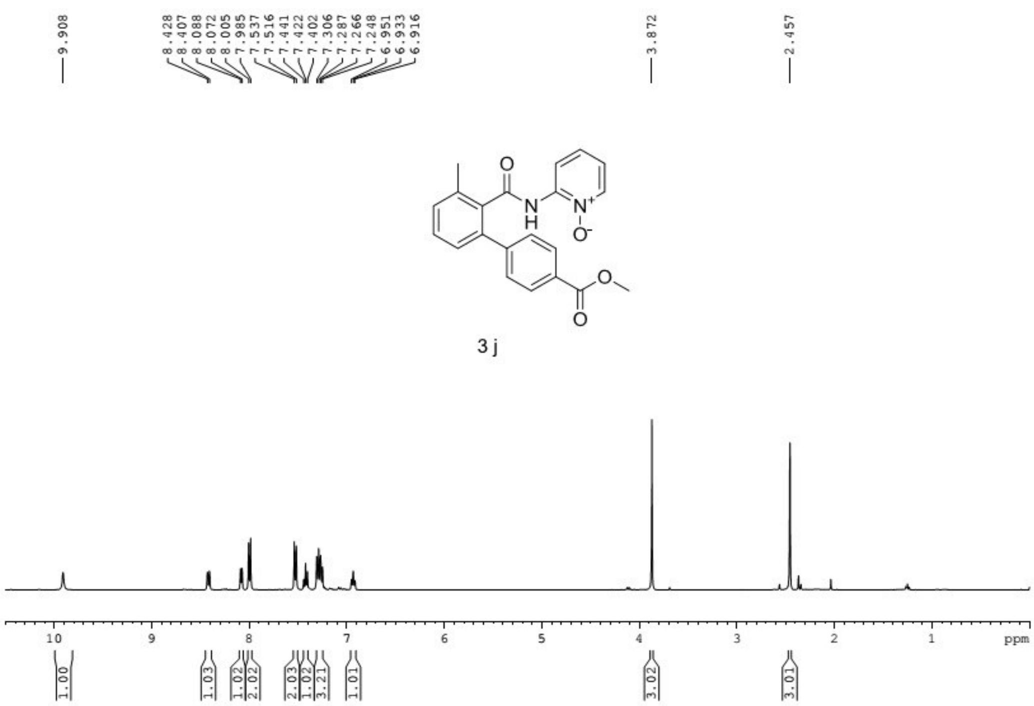
3 i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2.457
10 9 8 7 6 5 4 3 2 1 ppm
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2.03
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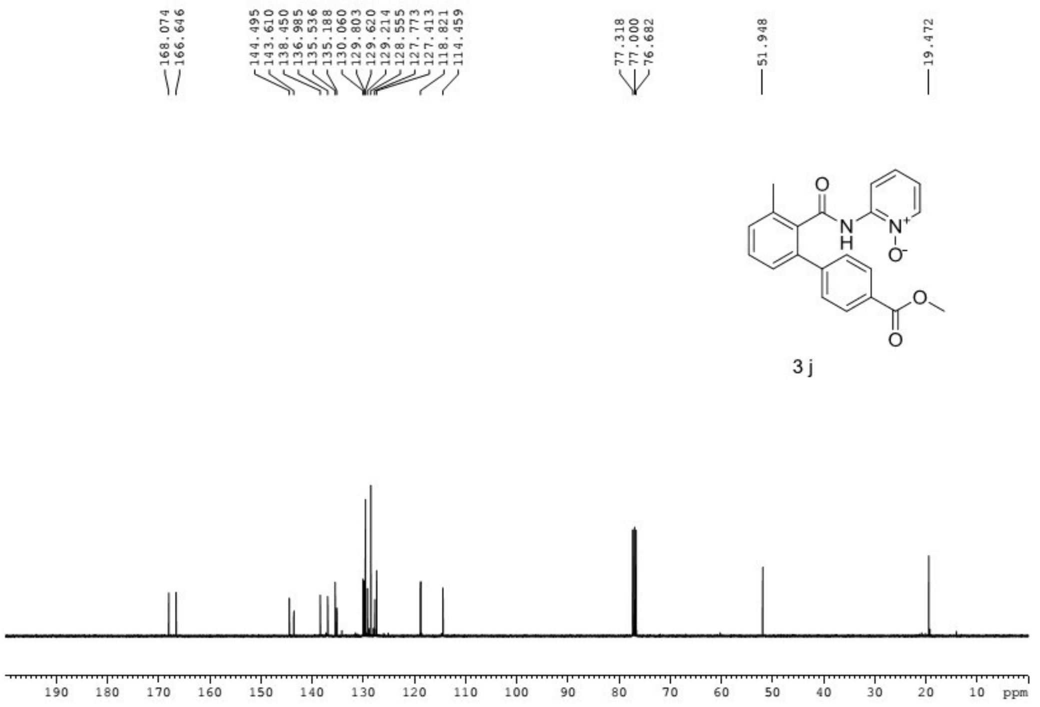
3.21
1.01
3.02
3.01

3 j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127.773
127.413
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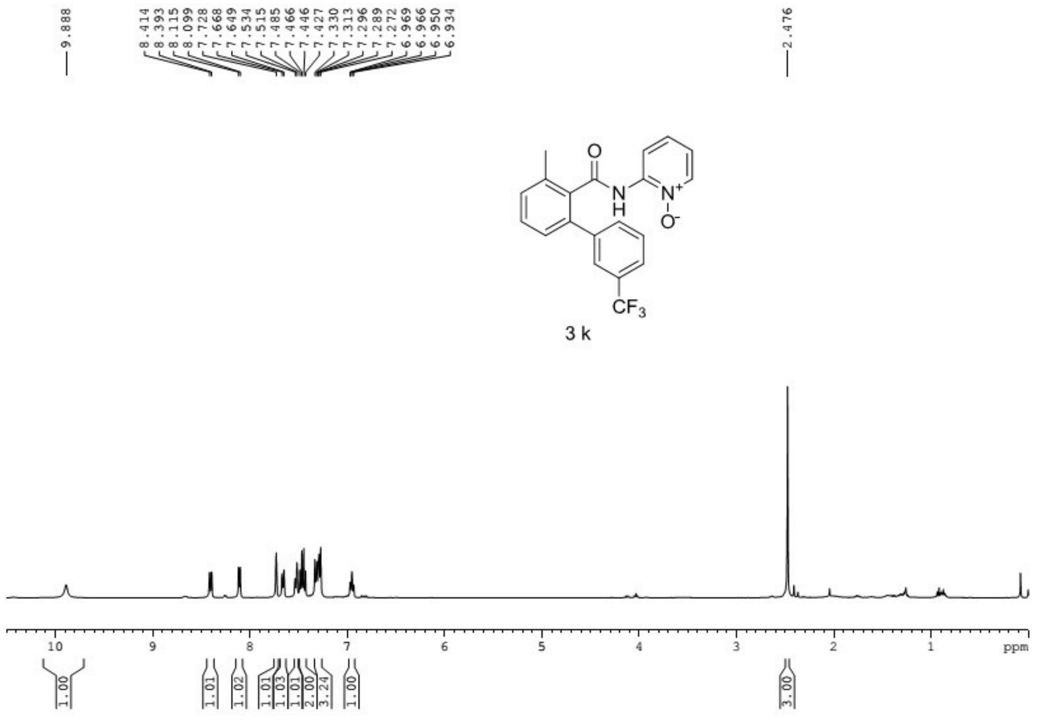
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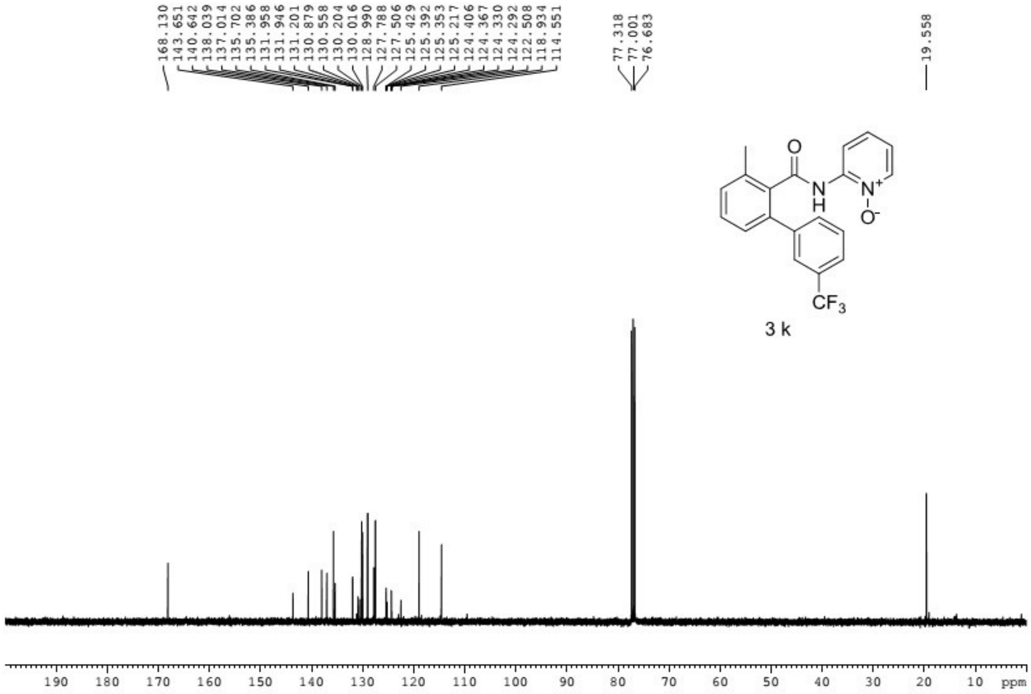
7.427
7.330
7.313
7.296
7.289
7.272
6.969
6.966
6.950
6.934
2.476
CF3
3 k
10 9 8 7 6 5 4 3 2 1 ppm
1.00
1.01
1.02
1.01
1.03
1.01
2.00
3.24
1.00
3.00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130.016
128.990
127.788
127.506
125.429
125.392
125.353
125.217
124.406
124.367
124.330
124.292
122.508
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77.318
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76.683
19.558
3 k
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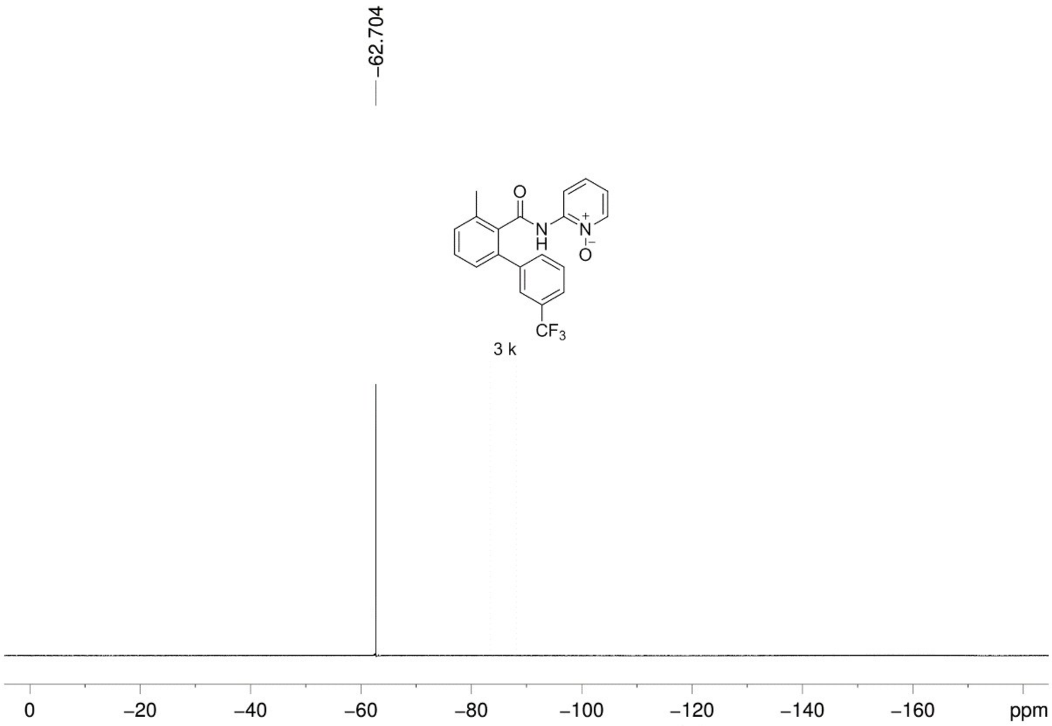
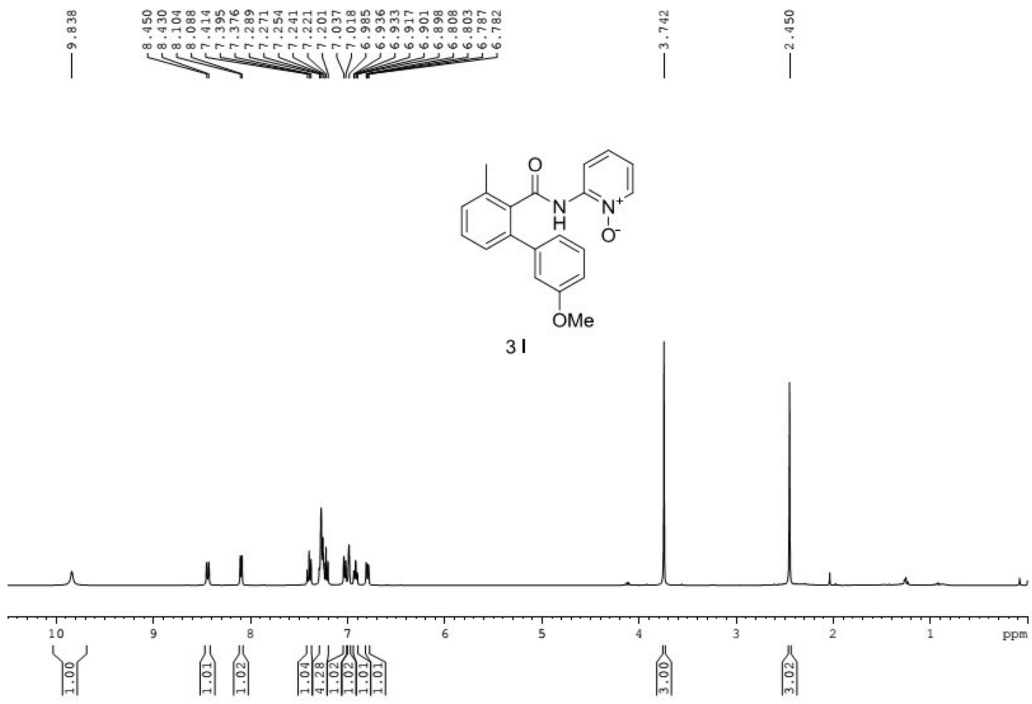

−62.704
3 k
0 −20 −40 −60 −80 −100 −120 −140 −160 ppm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7.271
7.254
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7.221
7.201
7.037
7.018
6.985
6.936
6.933
6.917
6.901
6.898
6.808
6.803
6.787
6.782
3.742
2.450
O
N
H
N+
O-
OMe
3l
10
9
8
7
6
5
4
3
2
1
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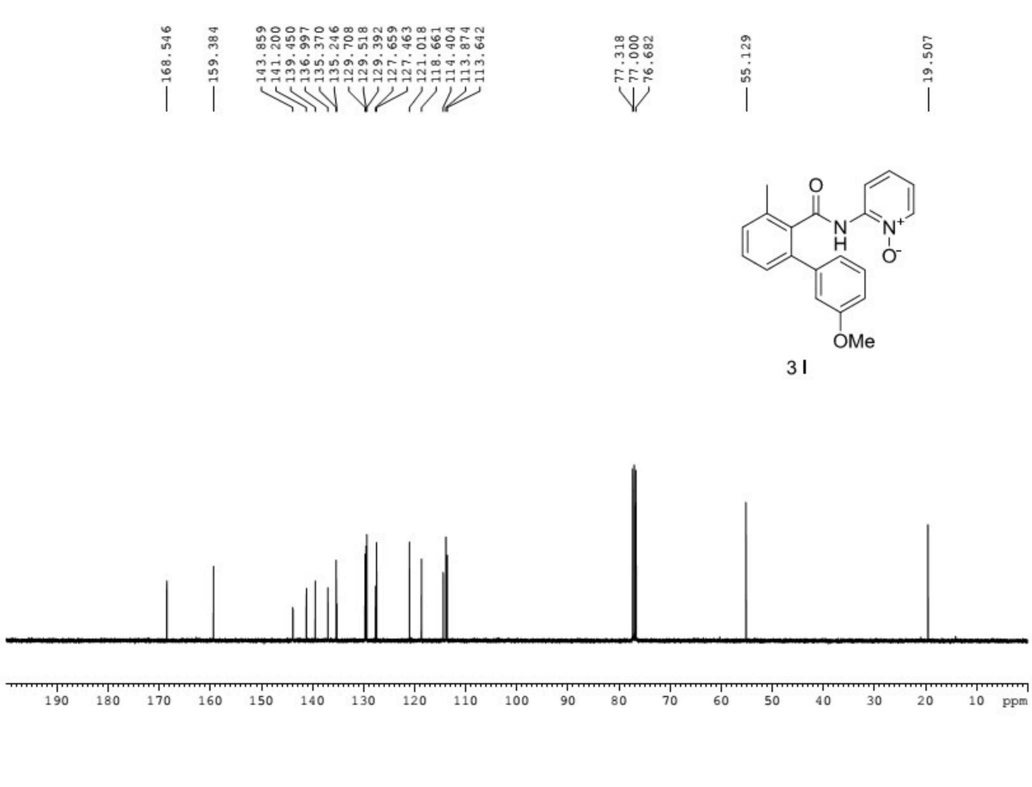
1.01
3.00
3.02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143.859
141.200
139.450
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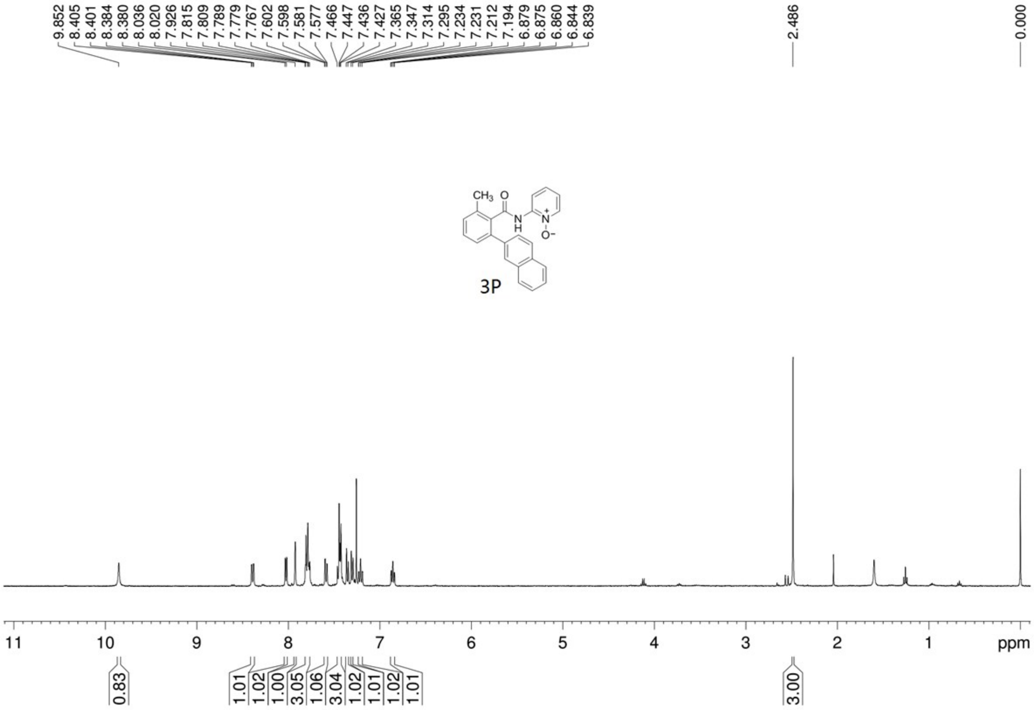
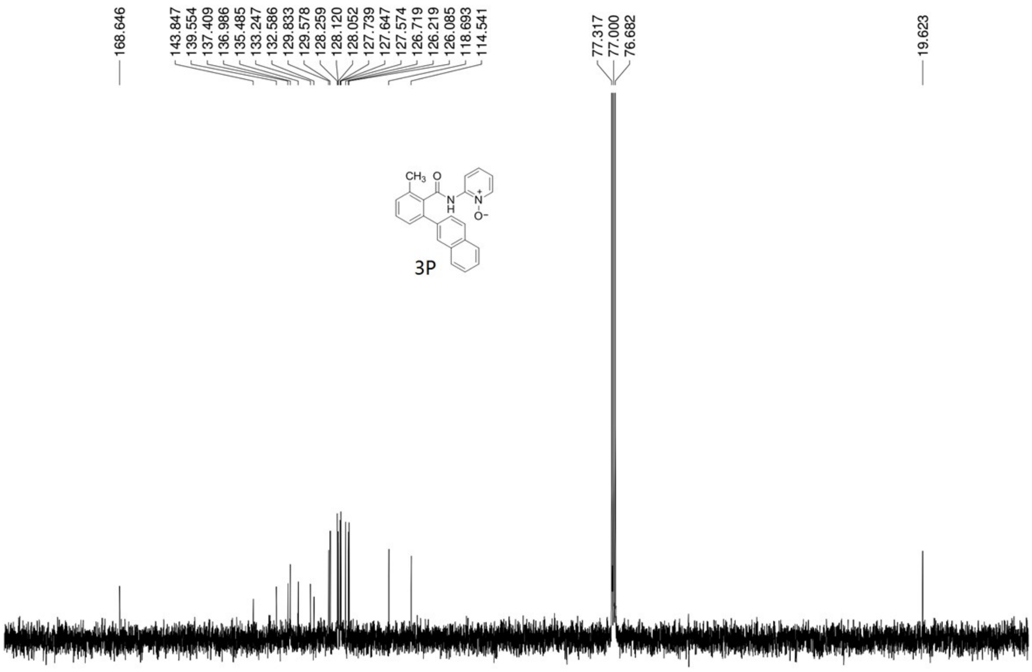
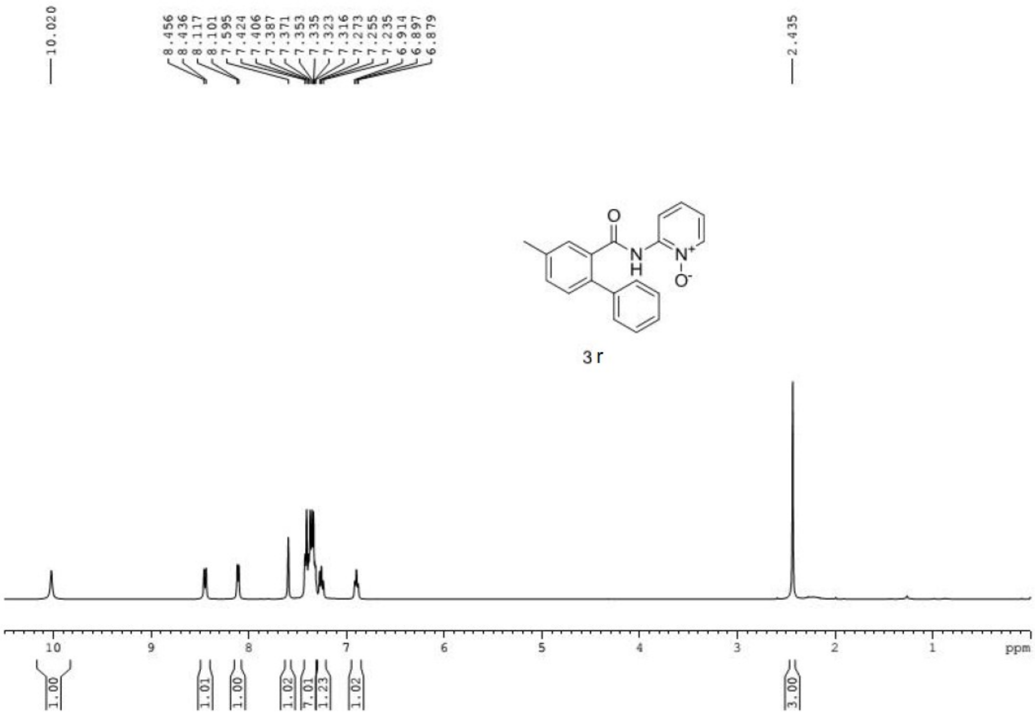
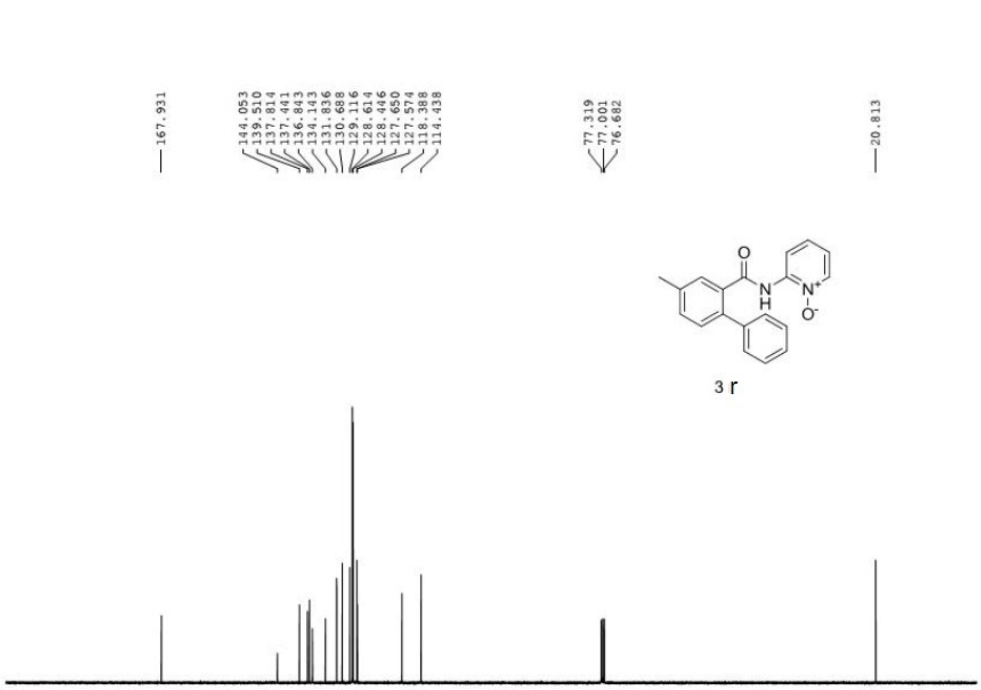
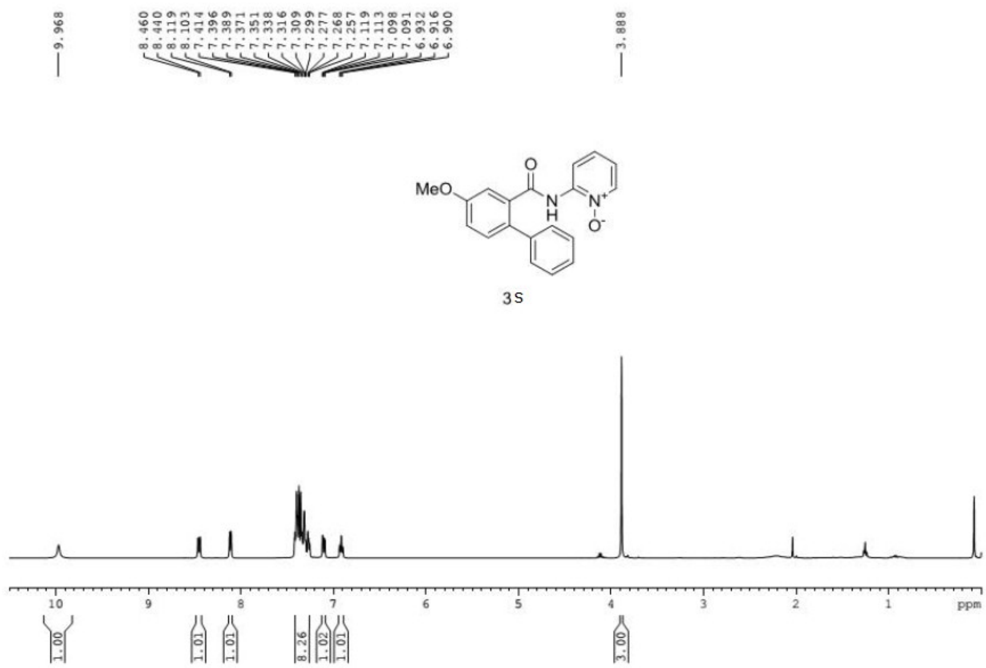
127.463
121.018
118.661
114.404
113.874
113.642
77.318
77.000
76.682
55.129
19.507
O
N
H
N+
O-
OMe
3l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

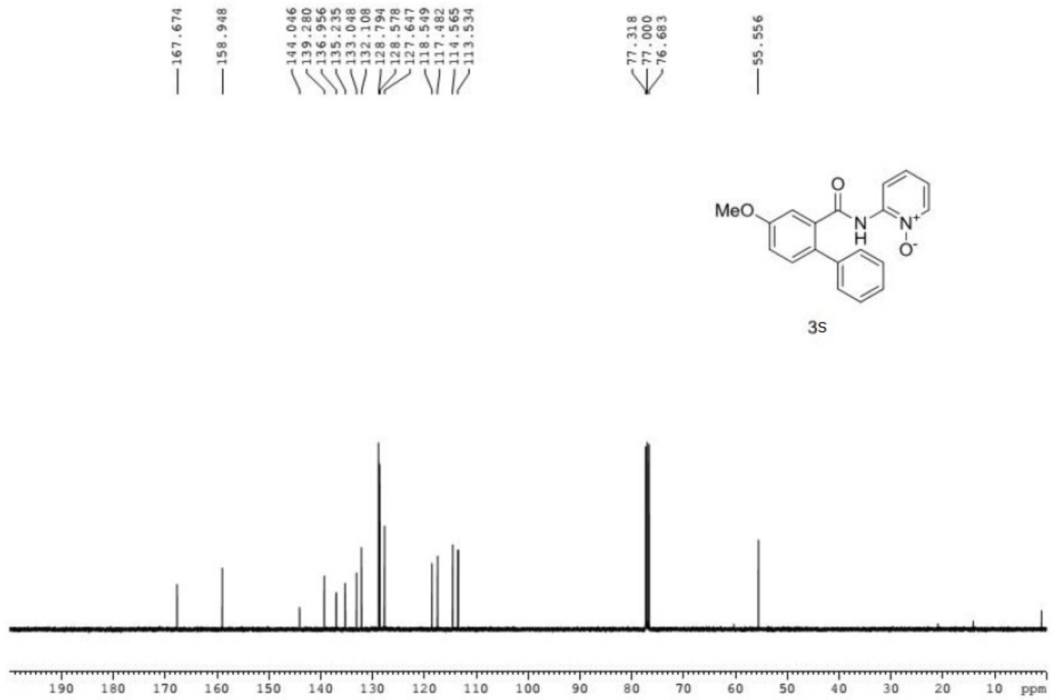

9.852 8.405 8.401 8.384 8.380 8.036 8.020 7.926 7.815 7.809 7.789 7.779 7.767 7.602 7.598 7.581 7.577 7.466 7.447 7.436 7.427 7.365 7.347 7.314 7.295 7.234 7.231 7.212 7.194 6.879 6.875 6.860 6.844 6.839
2.486
0.000
3P
11 10 9 8 7 6 5 4 3 2 1 ppm
0.83 1.01 1.02 1.00 3.05 1.06 3.04 1.02 1.01 1.02 1.01 3.00

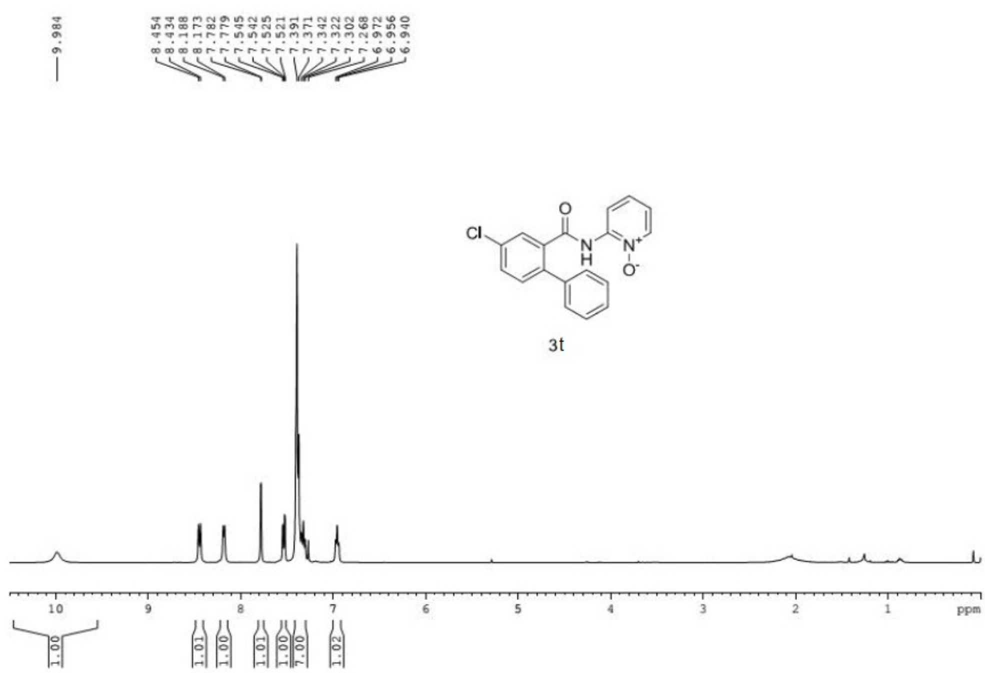

168.646
143.847 139.554 137.409 136.986 135.485 133.247 132.586 129.833 129.578 128.259 128.120 128.052 127.739 127.647 127.574 126.719 126.219 126.085 118.693 114.541
77.317 77.000 76.682
19.623
3P
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

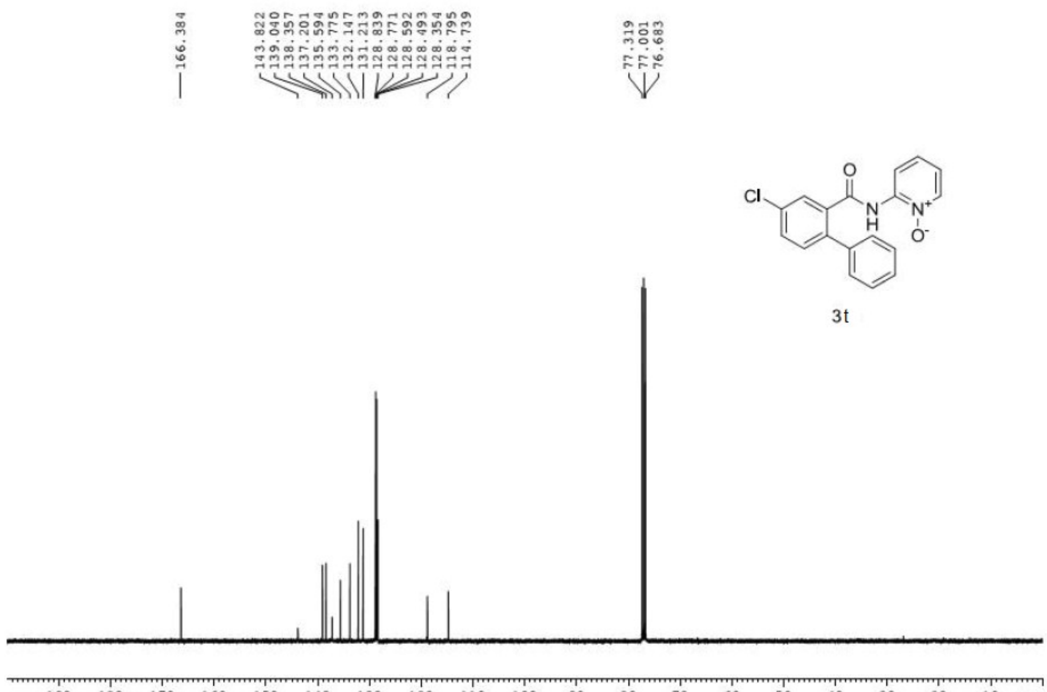

10.020
8.456
8.436
8.117
8.101
7.595
7.424
7.406
7.387
7.371
7.353
7.335
7.323
7.316
7.273
7.255
7.235
6.914
6.897
6.879
2.435
O
N
H
N+
O-
3 r
ppm
1.00
1.01
1.00
1.02
7.01
1.23
1.02
3.00

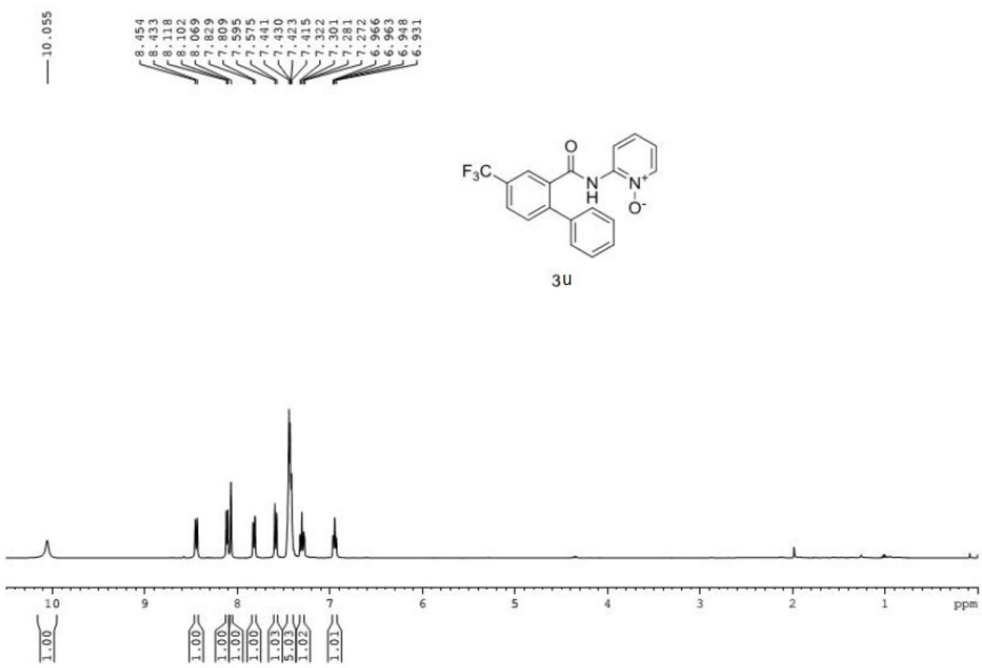

167.931
144.053
139.510
137.814
137.441
136.843
134.143
131.836
130.688
129.116
128.614
128.446
127.650
127.574
118.388
114.438
77.319
77.001
76.682
20.813
O
N
H
N+
O-
3 r
ppm

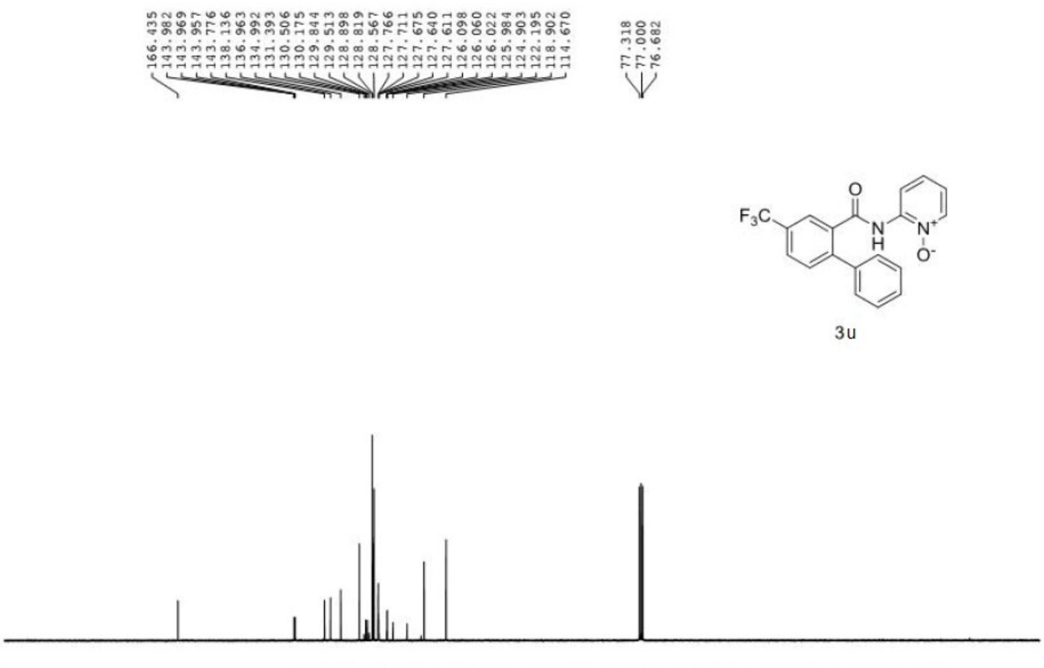

9.968
8.460
8.440
8.119
8.103
7.414
7.396
7.389
7.371
7.351
7.338
7.316
7.309
7.299
7.277
7.268
7.257
7.119
7.113
7.098
7.091
6.932
6.916
6.900
3.888
MeO
O
N
H
N+
O-
3s
1.00
1.01
1.01
8.26
1.02
1.01
3.00
ppm

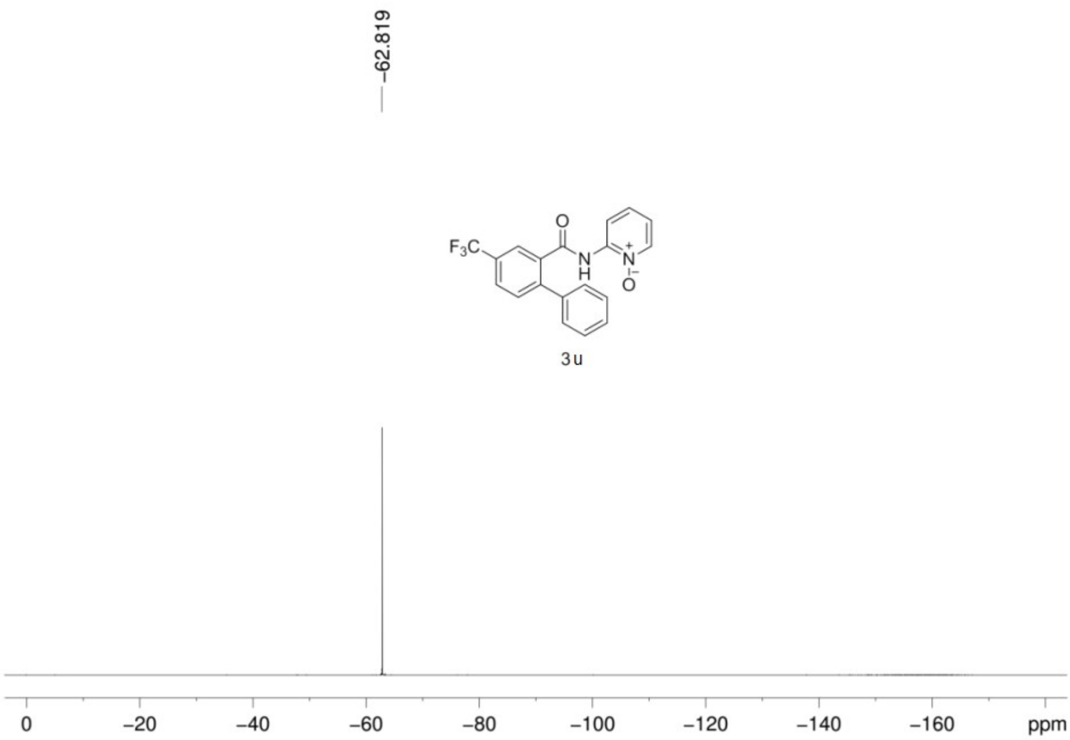

167.674
158.948
144.046
139.280
136.956
135.235
133.048
132.108
128.794
128.578
127.647
118.549
117.482
114.565
113.534
77.318
77.000
76.683
55.556
MeO
O
N
H
N+
O-
3s
ppm


9.984
8.454
8.434
8.188
8.173
7.782
7.779
7.545
7.542
7.525
7.521
7.391
7.371
7.342
7.322
7.302
7.268
6.972
6.956
6.940
Cl
O
N
H
N+
O-
3t
ppm
1.00
1.01
1.00
1.01
1.00
7.00
1.02


166.384
143.822
139.040
138.357
137.201
135.594
133.775
132.147
131.213
128.839
128.771
128.592
128.493
128.354
118.795
114.739
77.319
77.001
76.683
Cl
O
N
H
N+
O-
3t
ppm

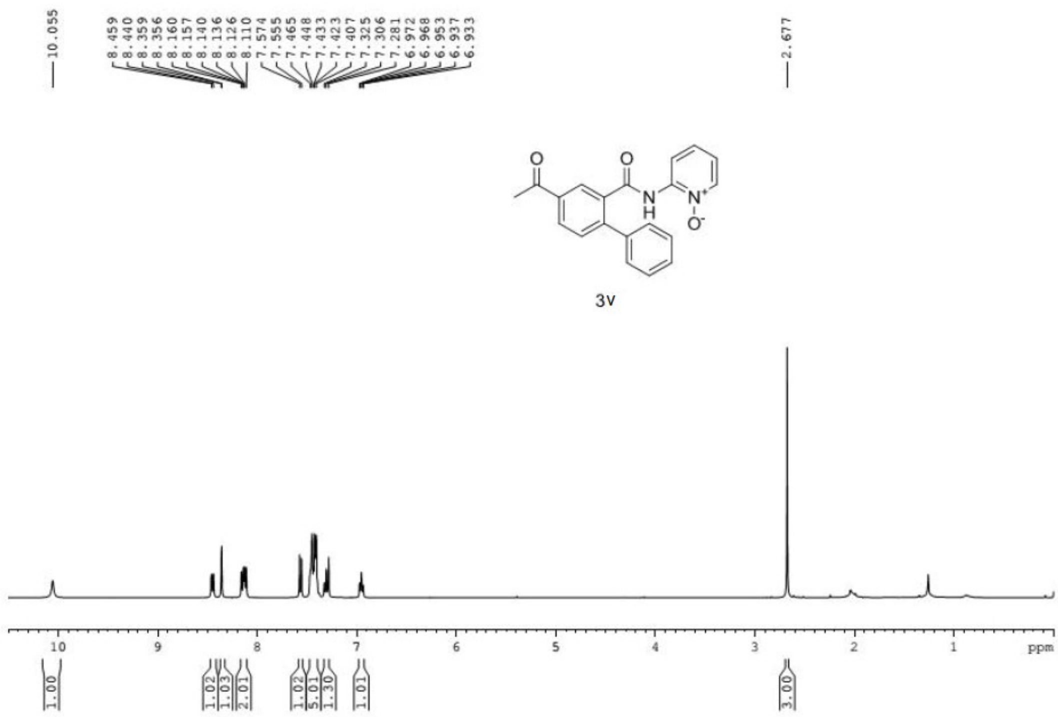

10.055
8.454
8.433
8.118
8.102
8.069
7.829
7.809
7.595
7.575
7.441
7.430
7.423
7.415
7.322
7.301
7.281
7.272
6.966
6.963
6.948
6.931
F_3C
O
N
H
N^+
O^-
3u
10 9 8 7 6 5 4 3 2 1 ppm
1.00
1.00
1.00
1.00
1.00
1.03
5.03
1.02
1.01


166.435
143.982
143.969
143.957
143.776
138.136
136.963
134.992
131.393
130.506
130.175
129.844
129.513
128.898
128.819
128.567
127.766
127.711
127.675
127.640
127.611
126.098
126.060
126.022
125.984
124.903
122.195
118.902
114.670
77.318
77.000
76.682
F_3C
O
N
H
N^+
O^-
3u
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



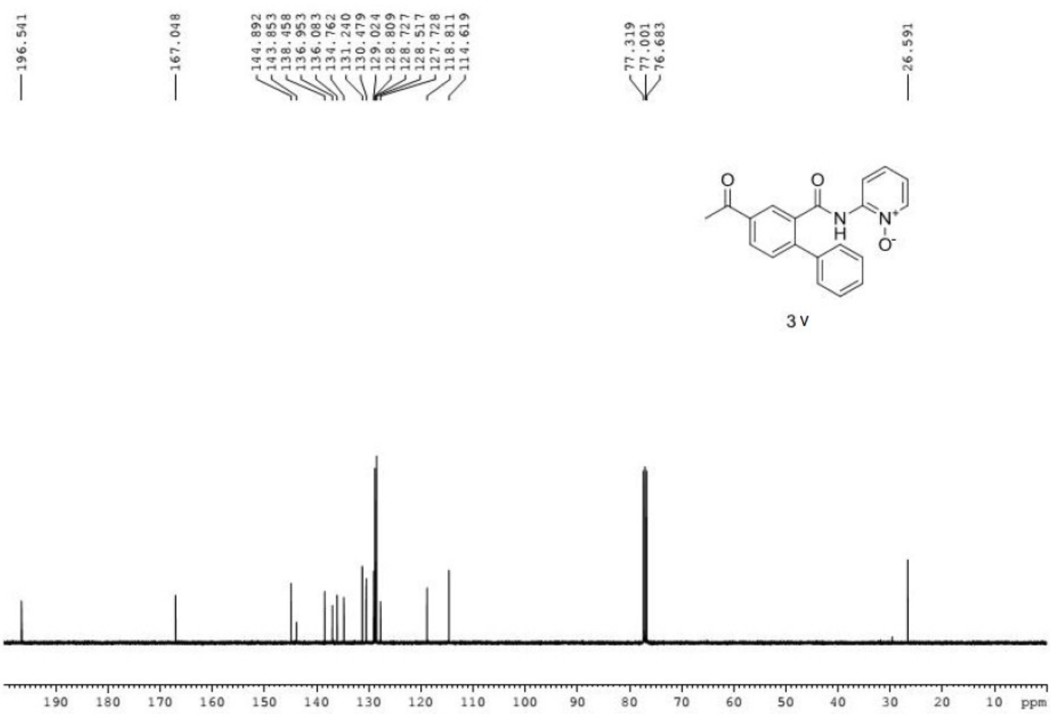
-62.819
F3C
O
N
H
N+
O-
0
-20
-40
-60
-80
-100
-120
-140
-160
ppm

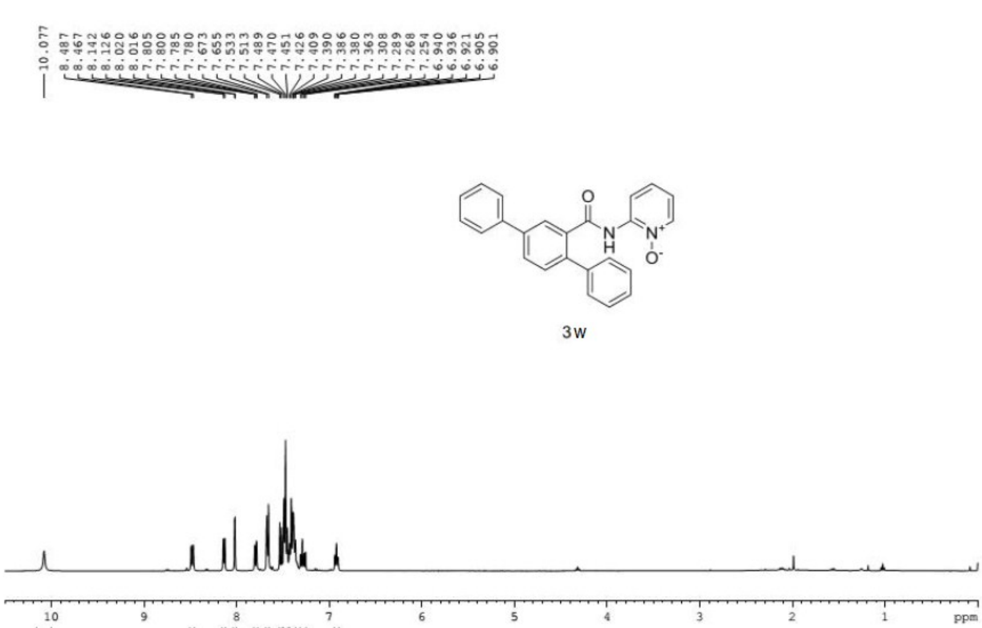
3u

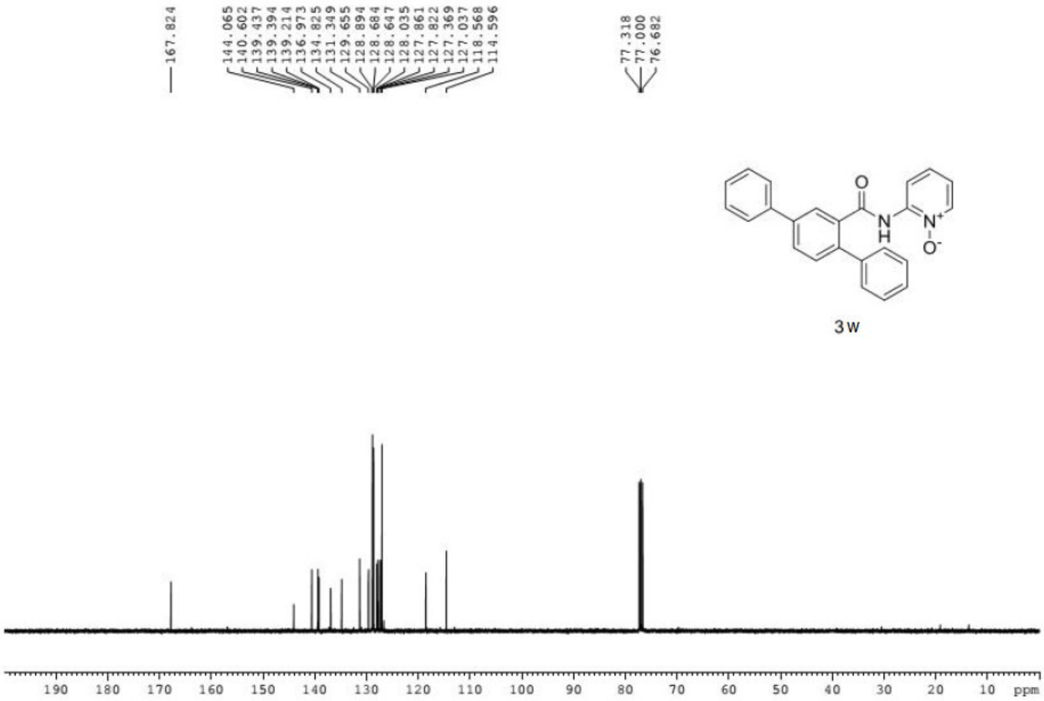


10.055
8.459
8.440
8.359
8.356
8.160
8.157
8.140
8.136
8.126
8.110
7.574
7.555
7.465
7.448
7.433
7.423
7.407
7.325
7.306
7.281
6.972
6.968
6.953
6.937
6.933
2.677
O
O
N
H
N+
O-
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.02
1.03
2.01
1.02
5.01
1.30
1.01
3.00

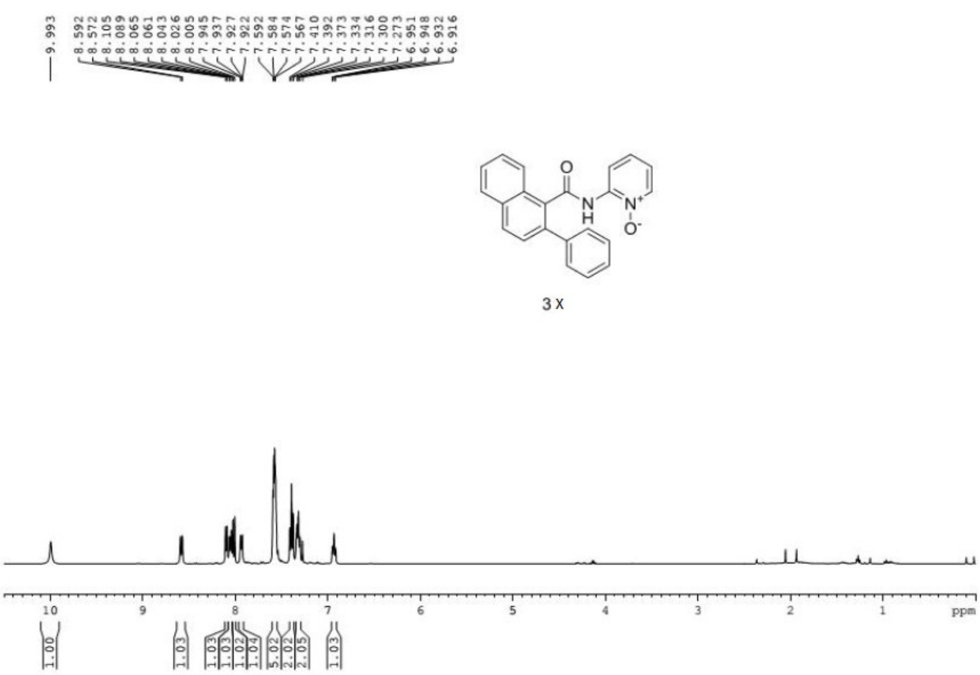
3v


196.541
167.048
144.892
143.853
138.458
136.953
136.083
134.762
131.240
130.479
129.024
128.809
128.727
128.517
127.728
118.811
114.619
77.319
77.001
76.683
26.591
3v
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

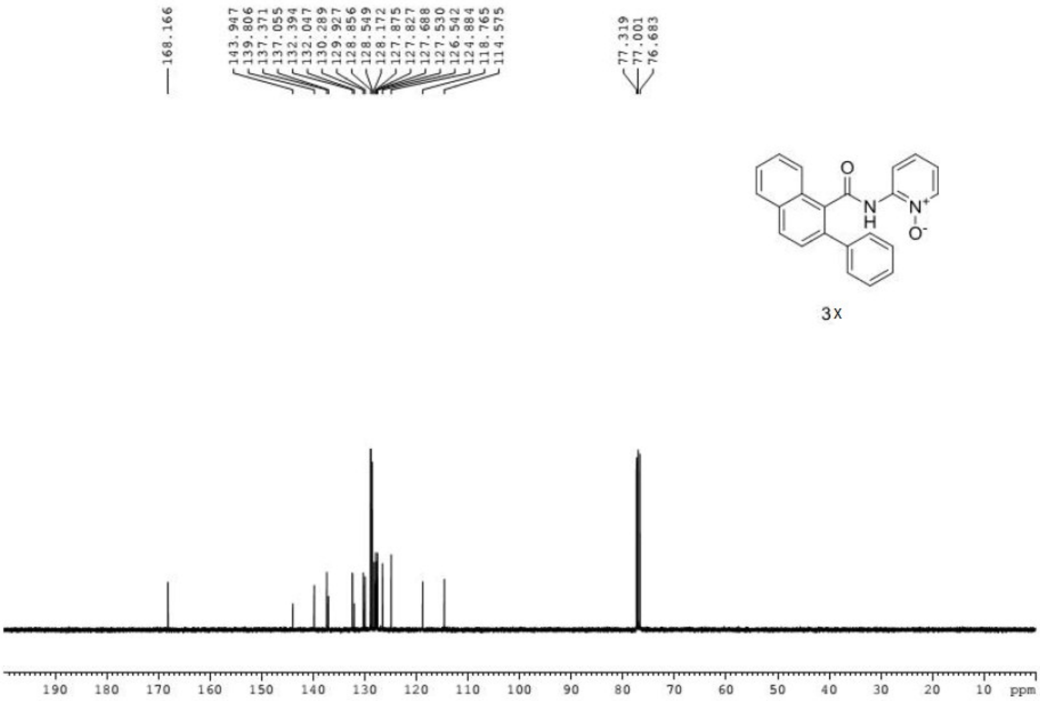

10.077
8.487
8.467
8.142
8.126
8.020
8.016
7.805
7.800
7.785
7.780
7.673
7.655
7.533
7.513
7.489
7.470
7.451
7.426
7.409
7.390
7.386
7.380
7.363
7.308
7.289
7.268
7.254
6.940
6.936
6.921
6.905
6.901
3w
10 9 8 7 6 5 4 3 2 1 ppm
1.00
1.01
1.01
1.02
1.02
2.01
1.01
4.00
4.01
1.27
1.02

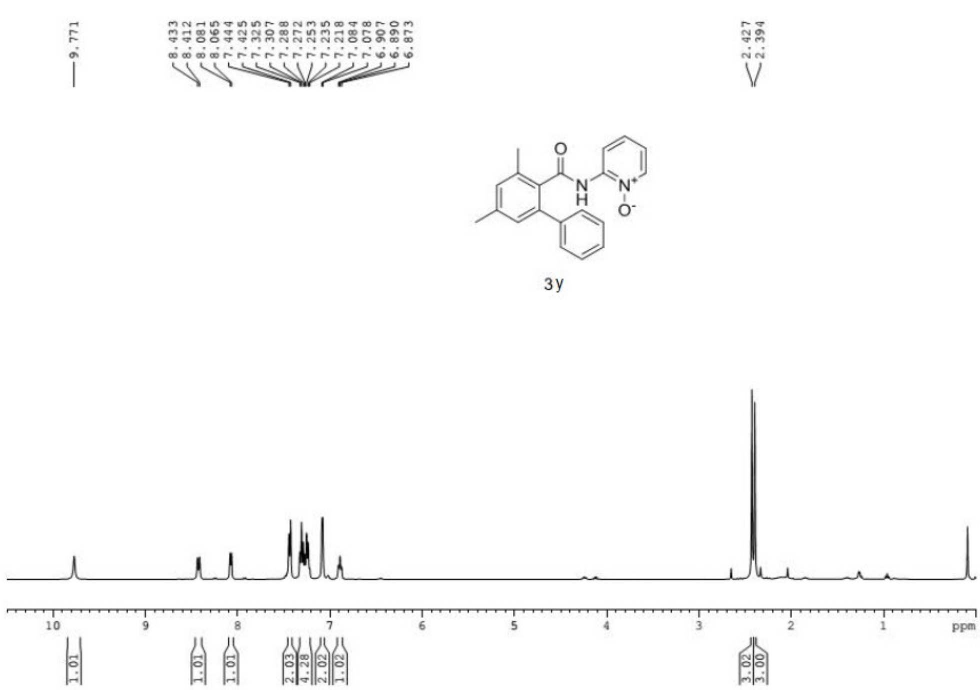

167.824
144.065
140.602
139.437
139.394
139.214
136.973
134.825
131.349
129.655
128.894
128.684
128.647
128.035
127.861
127.822
127.369
127.037
118.568
114.596
77.318
77.000
76.682
O
N
H
N+
O-
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

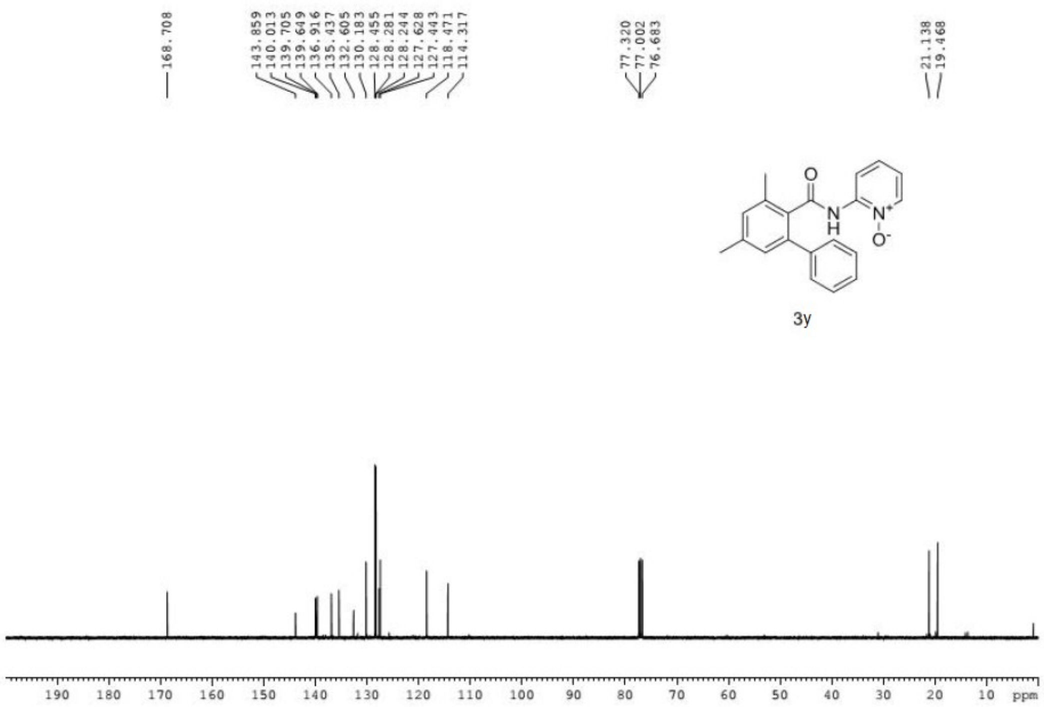
3w


9.993
8.592
8.572
8.105
8.089
8.065
8.061
8.043
8.026
8.005
7.945
7.937
7.927
7.922
7.592
7.584
7.574
7.567
7.410
7.392
7.373
7.334
7.316
7.300
7.273
6.951
6.948
6.932
6.916
O
N
H
N+
O-
10 9 8 7 6 5 4 3 2 1 ppm
1.00
1.03
1.03
1.03
1.02
1.04
5.02
2.02
2.05
1.03

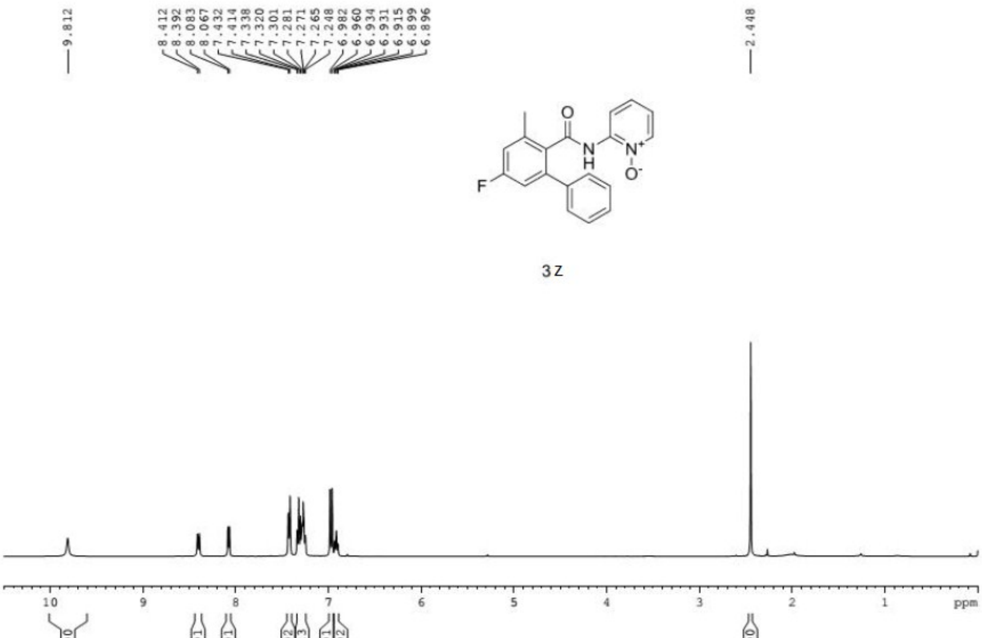
3x


168.166
143.947
139.806
137.371
137.055
132.394
132.047
130.289
129.927
128.856
128.549
128.172
127.875
127.827
127.688
127.530
126.542
124.884
118.765
114.575
77.319
77.001
76.683
3x
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

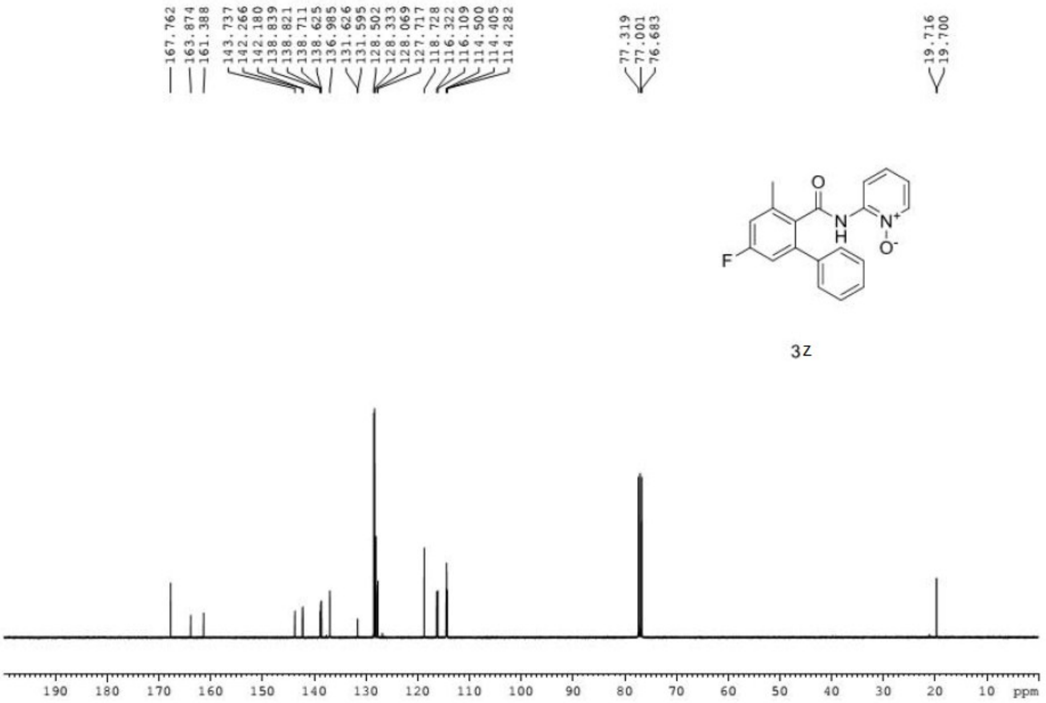

9.771
8.433
8.412
8.081
8.065
7.444
7.425
7.325
7.307
7.288
7.272
7.253
7.235
7.218
7.084
7.078
6.907
6.890
6.873
2.427
2.394
3y
10 9 8 7 6 5 4 3 2 1 ppm
1.01
1.01
1.01
2.03
4.28
2.02
1.02
3.02
3.00


168.708
143.859
140.013
139.705
139.649
136.916
135.437
132.605
130.183
128.455
128.281
128.244
127.628
127.443
118.471
114.317
77.320
77.002
76.683
21.138
19.468
O
N
H
N+
O-
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

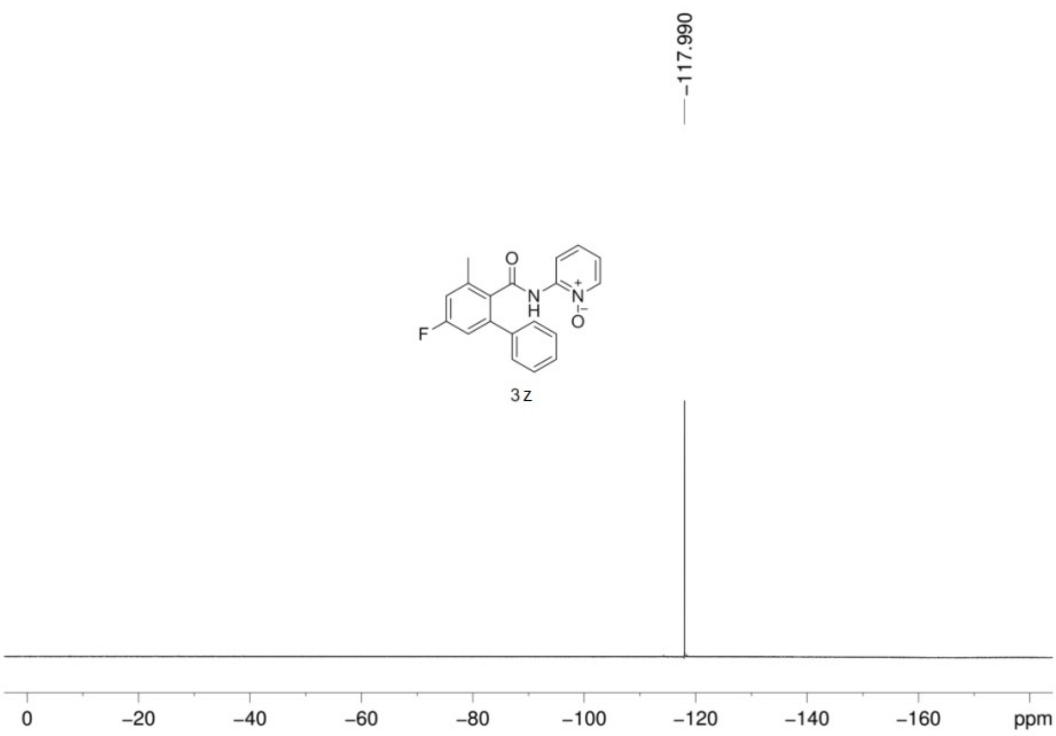
3y


9.812
8.412
8.392
8.083
8.067
7.432
7.414
7.338
7.320
7.301
7.281
7.271
7.265
7.248
6.982
6.960
6.934
6.931
6.915
6.899
6.896
2.448
O
N
H
N+
O-
F
10 9 8 7 6 5 4 3 2 1 ppm
1.00
1.01
1.01
2.02
4.23
2.01
1.02
3.00

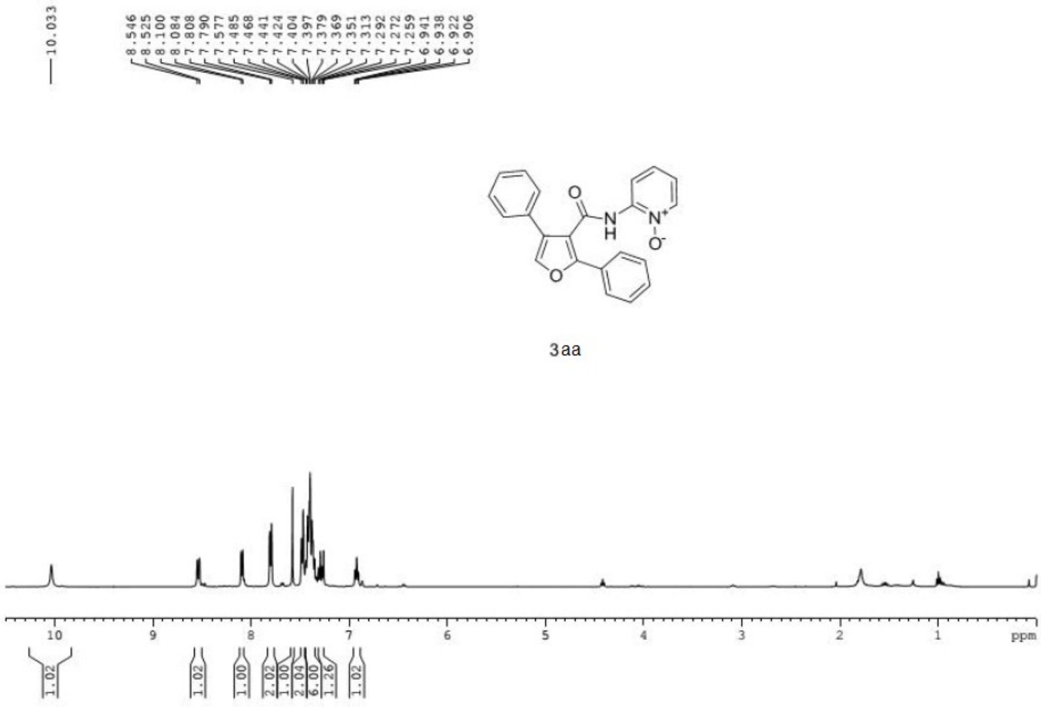
3z



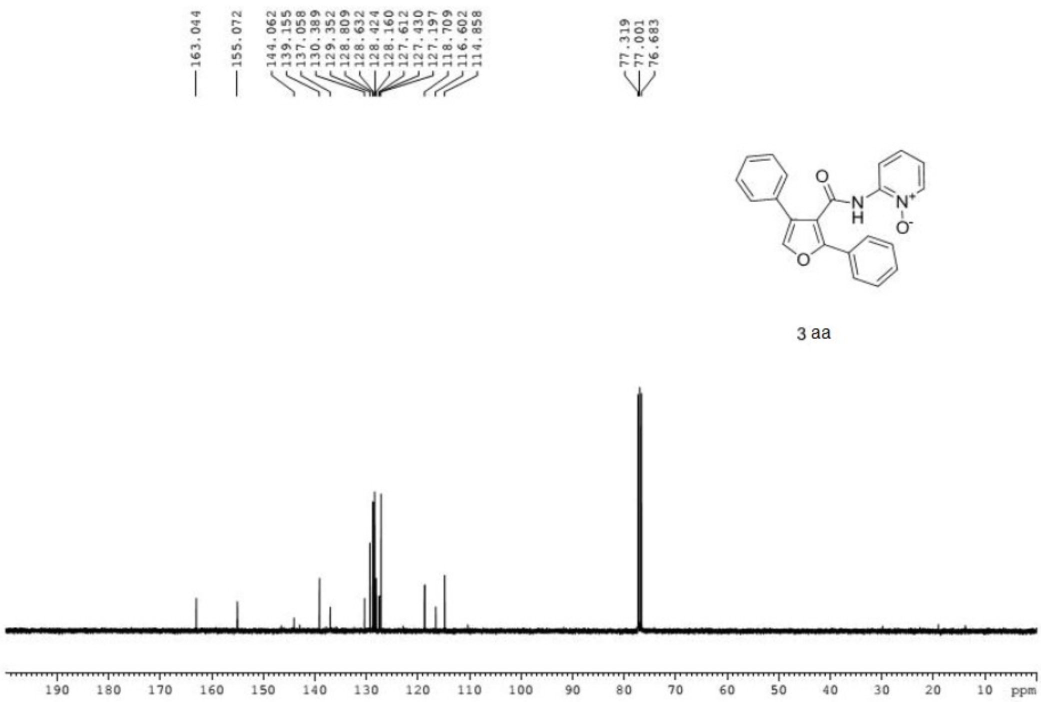
167.762
163.874
161.388
143.737
142.266
142.180
138.839
138.821
138.711
138.625
136.985
131.626
131.595
128.502
128.333
128.069
127.717
118.728
116.322
116.109
114.500
114.405
114.282
77.319
77.001
76.683
19.716
19.700
3z
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



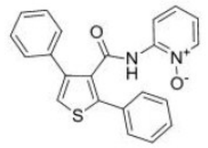
-117.990
3z
0 −20 −40 −60 −80 −100 −120 −140 −160 ppm


10.033
8.546
8.525
8.100
8.084
7.808
7.790
7.577
7.485
7.468
7.441
7.424
7.404
7.397
7.379
7.369
7.351
7.313
7.292
7.272
7.259
6.941
6.938
6.922
6.906
1.02
1.02
1.00
2.02
1.00
2.04
6.00
1.26
1.02
10 9 8 7 6 5 4 3 2 1 ppm

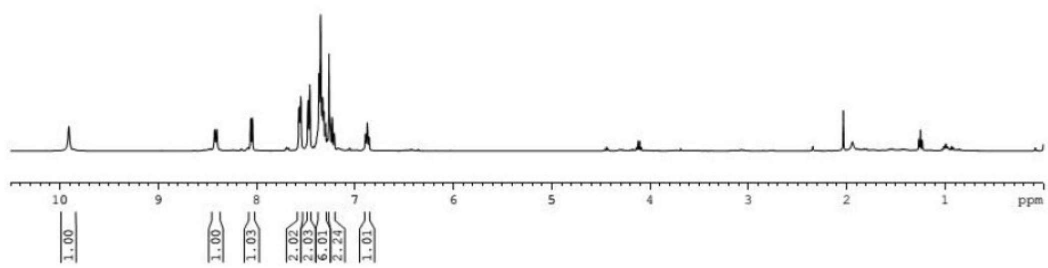
3aa


163.044
155.072
144.062
139.155
137.058
130.389
129.352
128.809
128.632
128.424
128.160
127.612
127.430
127.197
118.709
116.602
114.858
77.319
77.001
76.683
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3 aa



3 ab


9.908
8.425
8.404
8.057
8.041
7.569
7.564
7.549
7.474
7.457
7.365
7.347
7.327
7.315
7.297
7.261
7.229
7.209
6.894
6.891
6.875
6.859
1.00
1.00
1.03
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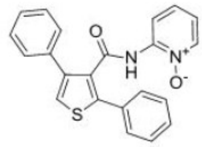
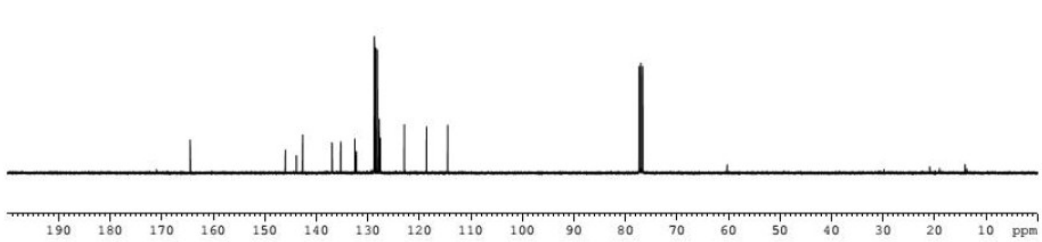
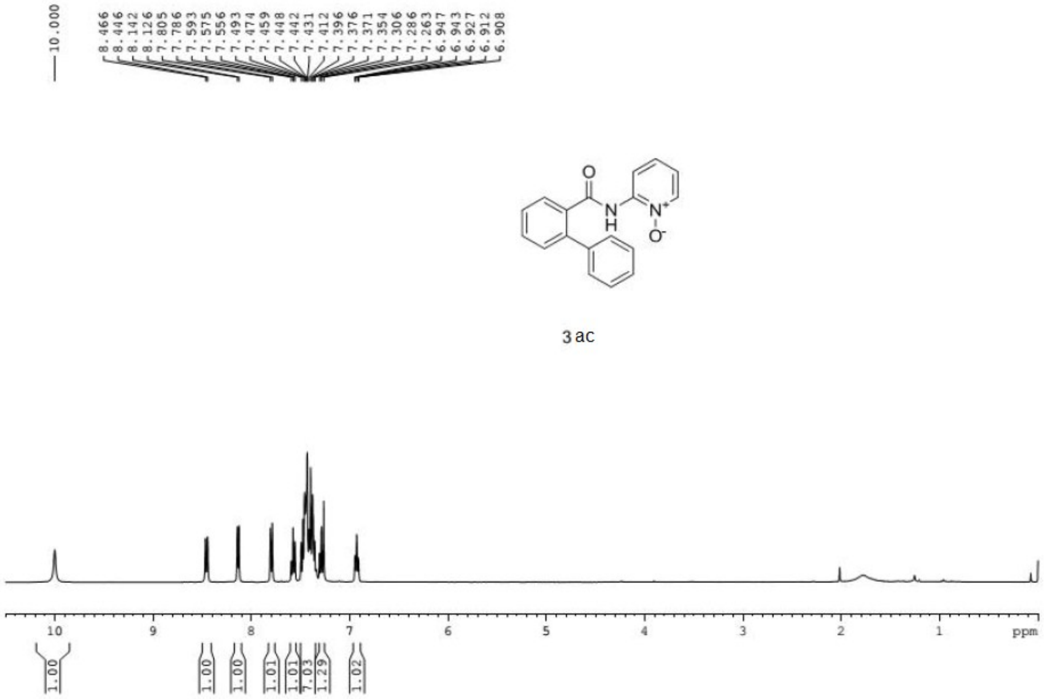
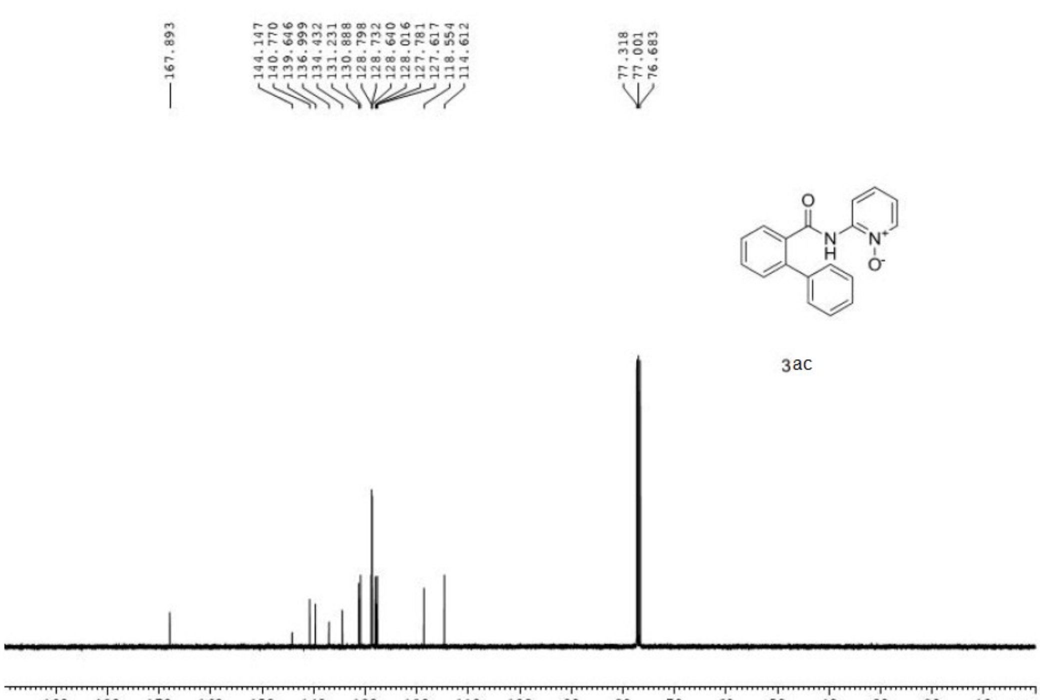
2.03
6.01
2.24
1.01
ppm

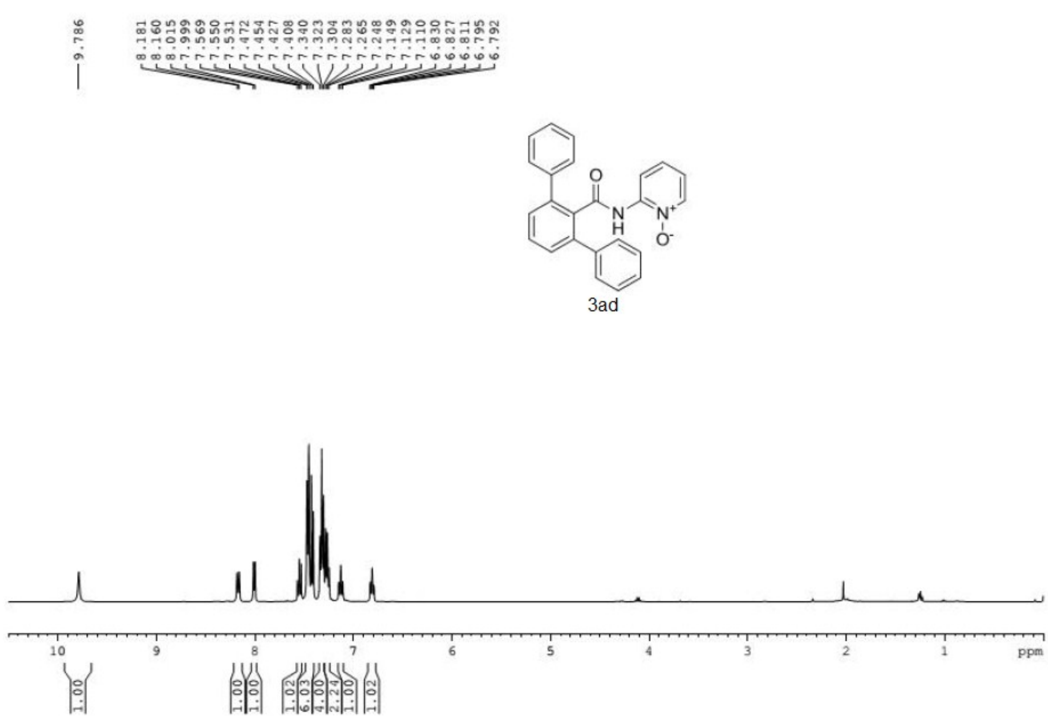


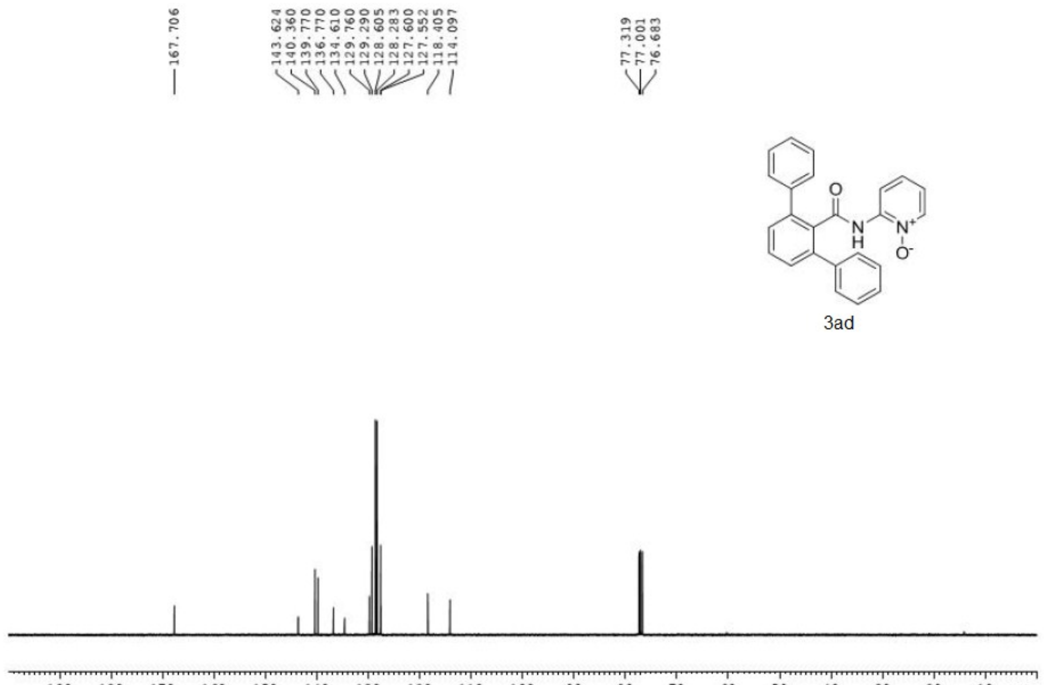
3 ab


164.431
145.961
143.827
142.625
136.917
135.224
132.546
132.250
128.802
128.757
128.729
128.482
128.162
127.793
127.575
122.925
118.624
114.501
77.319
77.001
76.683
ppm


10.000
8.466
8.446
8.142
8.126
7.805
7.786
7.593
7.575
7.556
7.493
7.474
7.459
7.448
7.442
7.431
7.412
7.396
7.376
7.371
7.354
7.306
7.286
7.263
6.947
6.943
6.927
6.912
6.908
O
N
H
N+
O-
3ac
10 9 8 7 6 5 4 3 2 1 ppm
1.00
1.00
1.00
1.01
1.01
7.03
1.29
1.02


167.893
144.147
140.770
139.646
136.999
134.432
131.231
130.888
128.798
128.732
128.640
128.016
127.781
127.617
118.554
114.612
77.318
77.001
76.683
O
N
H
N+
O-
3ac
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


9.786
8.181 8.160 8.015 7.999 7.569 7.550 7.531 7.472 7.454 7.427 7.408 7.340 7.323 7.304 7.283 7.265 7.248 7.149 7.129 7.110 6.830 6.827 6.811 6.795 6.792
3ad
1.00 1.00 1.00 1.02 6.03 4.00 2.24 1.00 1.02
ppm


167.706
143.624 140.360 139.770 136.770 134.610 129.760 129.290 128.605 128.283 127.600 127.552 118.405 114.097
77.319 77.001 76.683
3ad
ppm