Supporting Information

Room temperature iron(II)-catalyzed radical cyclization of unsaturated oximes with hypervalent iodine reagents

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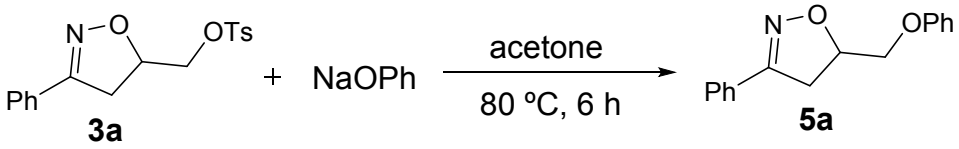
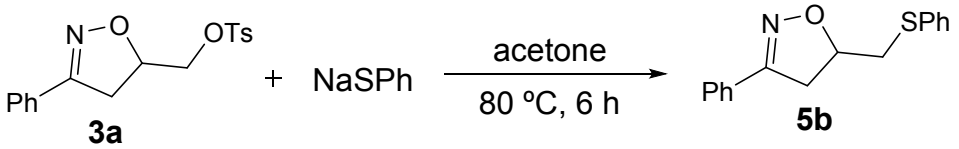
1. General Considerations

The starting materials, such as oximes and hydroxysulfonyl iodobenzene reagents, are prepared according to the reported methods, respectively [(a)Y. He, L. Li, Y. Yang, Y. Wang, J. Luo, X. Liu and Y. Liang, *Chem. Commun.*, 2013, **49**, 5687.; (b) E. A. Merritt, V. M. T. Carneiro, L. F. Silva Jr. and B. Olofsson, *J. Org. Chem*. 2010, **75**, 7416]. All the solvents were dried and freshly distilled prior to use. Products were purified by flash chromatography on silica gels, eluting with petroleum ether/ethyl acetate (3:1 to 9:1).

2. Typical procedure for the diverse transformations of 3a

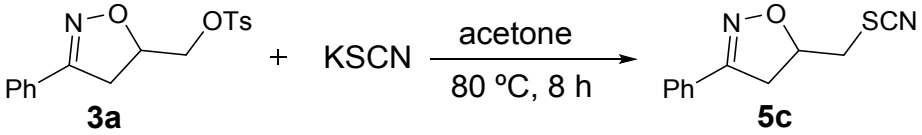


(1) A 10 mL reaction tube was charged with **3a** (66.3 mg, 0.20 mmol), NaOPh (46.4 mg, 0.40 mmol), Then acetone (1.0 mL) was added to the resulted mixture. The reaction tube was placed at 80 °C for 6 h. After that, the mixture in reaction tube was detected by TLC. After the reaction was completed, distilled water (10 mL) was added into the mixture, and extracted with dichloromethane (DCM, 3×10 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to yield the crude product, which was purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 3:1 to 9:1), affording the desired product **5a** as a white solid (38.5 mg, 76% yield).

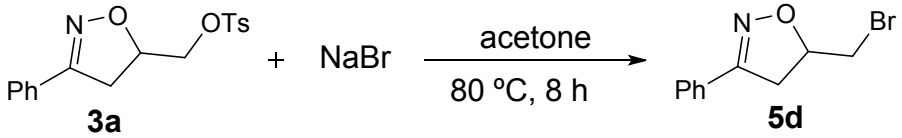


(2) A 10 mL reaction tube was charged with **3a** (66.3 mg, 0.20 mmol), NaSPh (52.9 mg, 0.40 mmol), Then acetone (1.0 mL) was added to the resulted mixture. The reaction tube was placed at 80 °C for 6 h. Then the mixture in reaction tube was

detected by TLC. After the reaction was completed, distilled water (10 mL) was added into the mixture, and extracted with dichloromethane (DCM, 3×10 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to yield the crude product, which was purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 3:1 to 9:1), affording the desired product **5b** as a white solid (47.9 mg, 89 % yield).

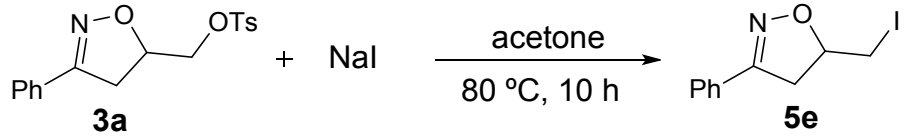


(3) A 10 mL reaction tube was charged with **3a** (66.3 mg, 0.20 mmol), KSCN (38.9 mg, 0.40 mmol), Then acetone (1.0 mL) was added to the resulted mixture. The reaction tube was placed at 80 °C for 8 h. Then, the mixture in reaction tube was detected by TLC. After the reaction was completed, distilled water (10 mL) was added into the mixture, and extracted with dichloromethane (DCM, 3×10 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to yield the crude product, which was purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 3:1 to 9:1), affording the desired product **5c** as a white solid (37.5 mg, 86 % yield).

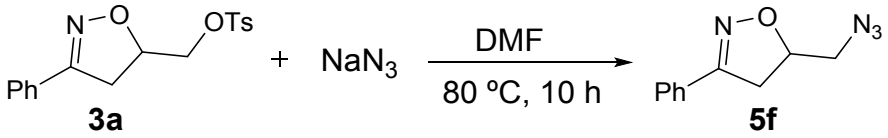


(4) A 10 mL reaction tube was charged with **3a** (66.3 mg, 0.20 mmol), NaBr (41.2 mg, 0.40 mmol), Then acetone (1.0 mL) was added to the resulted mixture. The reaction tube was placed at 80 °C for 8 h. Then, the mixture in reaction tube was detected by TLC. After the reaction was completed, distilled water (10 mL) was added into the mixture, and extracted with dichloromethane (DCM, 3×10 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to yield the crude product, which was purified by flash chromatography

(silica gel, petroleum ether/ethyl acetate = 3:1 to 9:1), affording the desired product **5d** as a white solid (38.4 mg, 80 % yield).



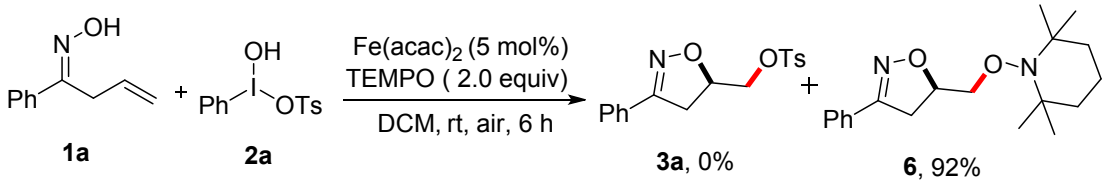
(5) A 10 mL reaction tube was charged with **3a** (66.3 mg, 0.20 mmol), NaI (60.0 mg, 0.40 mmol), Then acetone (1.0 mL) was added to the resulted mixture. The reaction tube was placed at 80 °C for 10 h. Then, the mixture in reaction tube was detected by TLC. After the reaction was completed, distilled water (10 mL) was added into the mixture, and extracted with dichloromethane (DCM, 3×10 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to yield the crude product, which was purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 3:1 to 9:1), affording the desired product **5e** as a white solid (54.5mg, 95 % yield).



(6) A 10 mL reaction tube was charged with **3a** (66.3 mg, 0.20 mmol), NaN_3 (26.0 mg, 0.30 mmol), Then DMF (1.0 mL) was added to the resulted mixture. The reaction tube was placed at 80 °C for 10 h. Then, the mixture in reaction tube was detected by TLC. After the reaction was completed, distilled water (10 mL) was added into the mixture, and extracted with dichloromethane (DCM, 3×10 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to yield the crude product, which was purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 3:1 to 9:1), affording the desired product **5f** as a white solid (36.0 mg, 89 % yield).

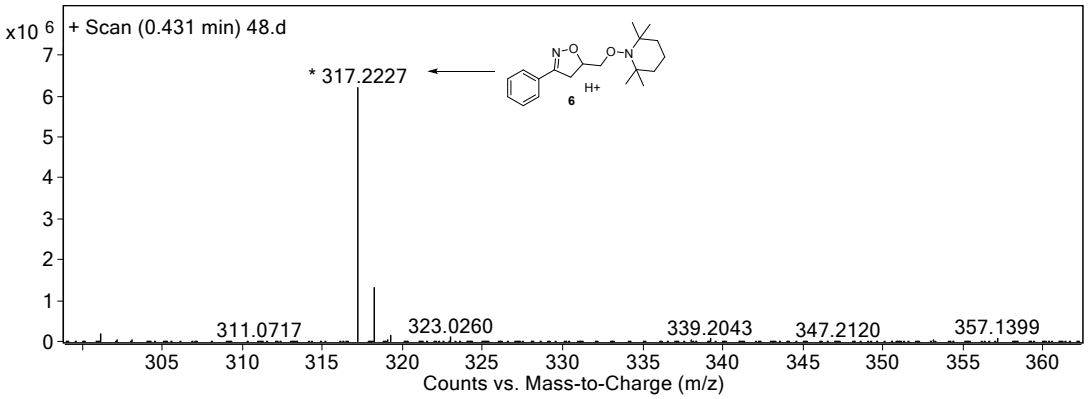
3. Mechanistic studies

(1) TEMPO experiment

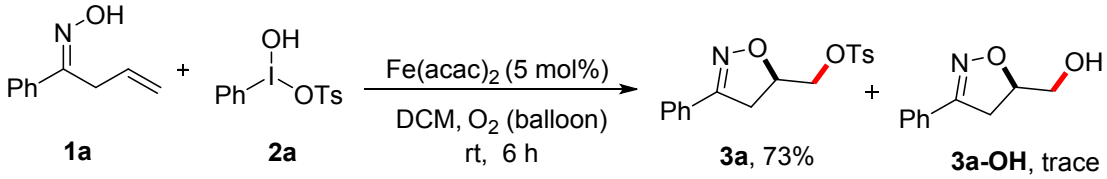


1a **2a** **3a**, 0% **6**, 92%

The reaction was carried out according to typical procedure for the synthesis of **3a**, except TEMPO (62.5 mg, 2.0 equiv) was used. The mixture was stirred at room temperature for 6 h. Then, the mixture in reaction tube was detected by TLC. After the reaction was completed, distilled water (10 mL) was added into the mixture, and extracted with dichloromethane (DCM, 3×10 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to yield the crude product, which was purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 3:1 to 9:1), affording the product **6** as a white solid (58.2 mg, 92% yield).

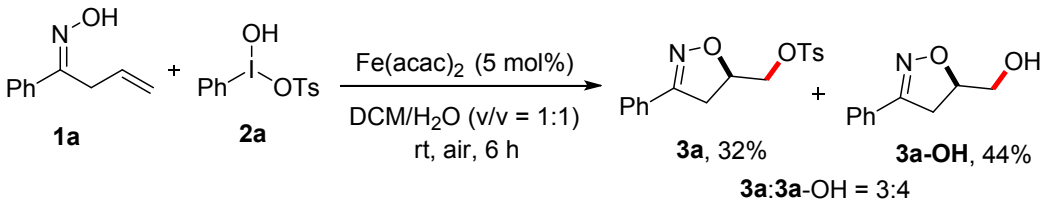


(2) The control experiments



1a **2a** **3a**, 73% **3a-OH**, trace

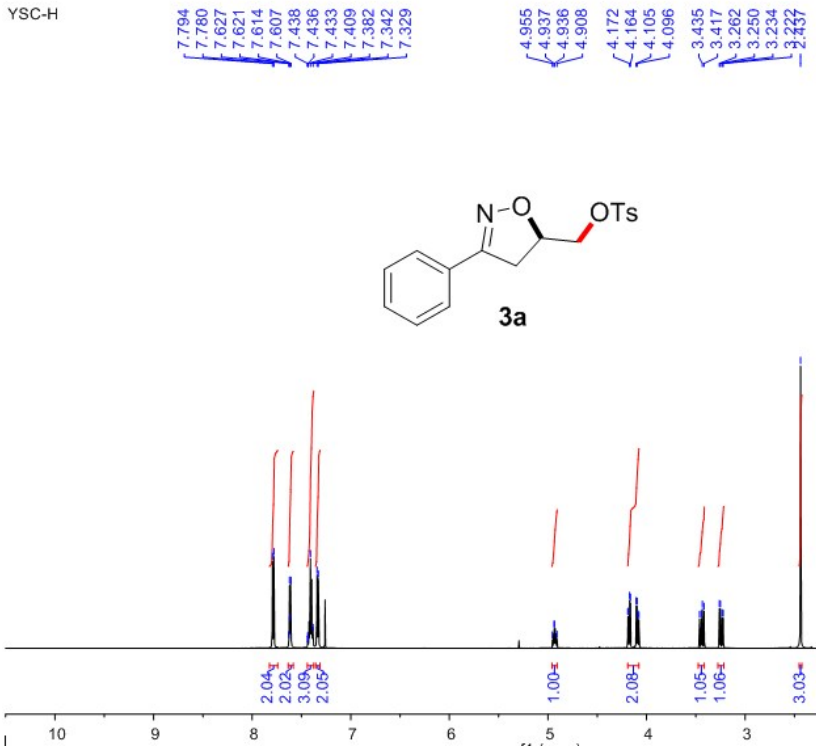
The reaction was carried out according to typical procedure for the synthesis of **3a**, except O_2 (balloon) was used. The mixture was stirred at room temperature for 6 h. Then, the mixture in reaction tube was detected by TLC. After the reaction was completed, distilled water (10 mL) was added into the mixture, and extracted with dichloromethane (DCM, 3×10 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to yield the crude product, which was purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 3:1 to 9:1), affording the product **3a** as a white solid (48.4 mg, 73% yield).



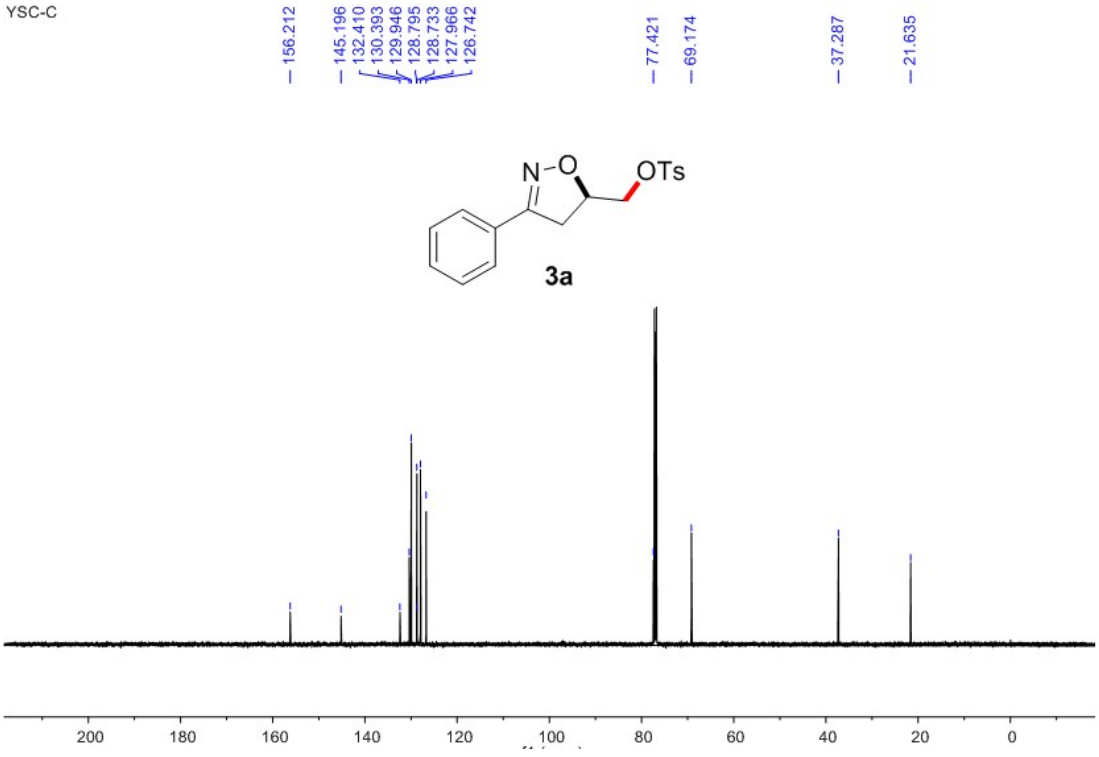
A 10 mL Reaction tube was charged with (*E*)-1-phenylbut-3-en-1-one oxime (**1a**, 32.2 mg, 0.2 mmol), $Fe(acac)_2$ (2.5 mg, 5 mmol %), DCM/H_2O (v/v =1:1,1.0 mL) and [hydroxy(tosyloxy)iodo]benzene (**2a**, 98.1 mg, 0.25 mmol) was added to the resulted mixture. The reaction tube was placed at room temperature for 6 h. Then, the mixture in reaction tube was detected by TLC. After the reaction was completed, distilled water (10 mL) was added into the mixture, and extracted with dichloromethane (DCM, 3×10 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to yield the crude product, which was purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 3:1 to 9:1), affording the desired product **3a** as a white solid (21.2 mg, 32% yield), as well as **3a-**OH as white solid (15.6 mg, 44% yield).

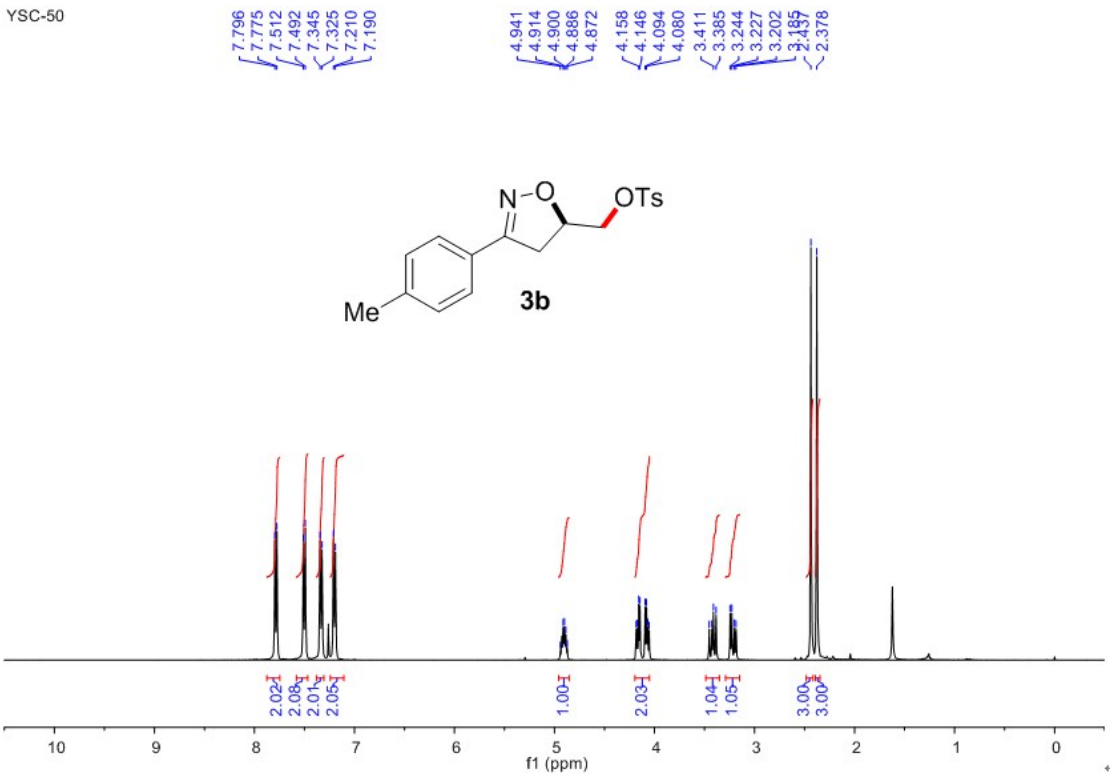
4. ^{1}H and ^{13}C NMR spectra of the products

YSC-H



YSC-C




YSC-50
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7.775
7.512
7.492
7.345
7.325
7.210
7.190
4.941
4.914
4.900
4.886
4.872
4.158
4.146
4.094
4.080
3.411
3.385
3.244
3.227
3.202
3.185
3.137
2.378
N
O
OTs
Me
3b
2.02
2.08
2.01
2.05
1.00
2.03
1.04
1.05
3.00
3.00
10
9
8
7
6
5
4
3
2
1
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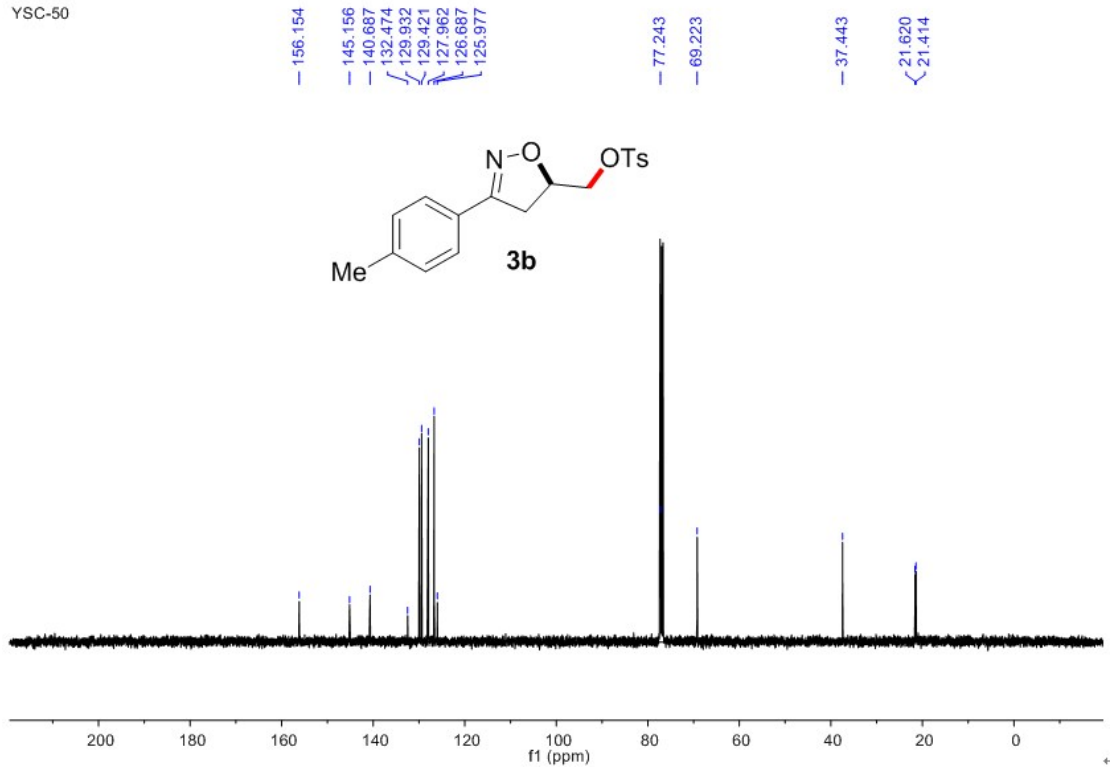
f1 (ppm)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140.687
132.474
129.932
129.421
127.962
126.687
125.977
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69.223
37.443
21.620
21.414
N
O
OTs
Me
3b
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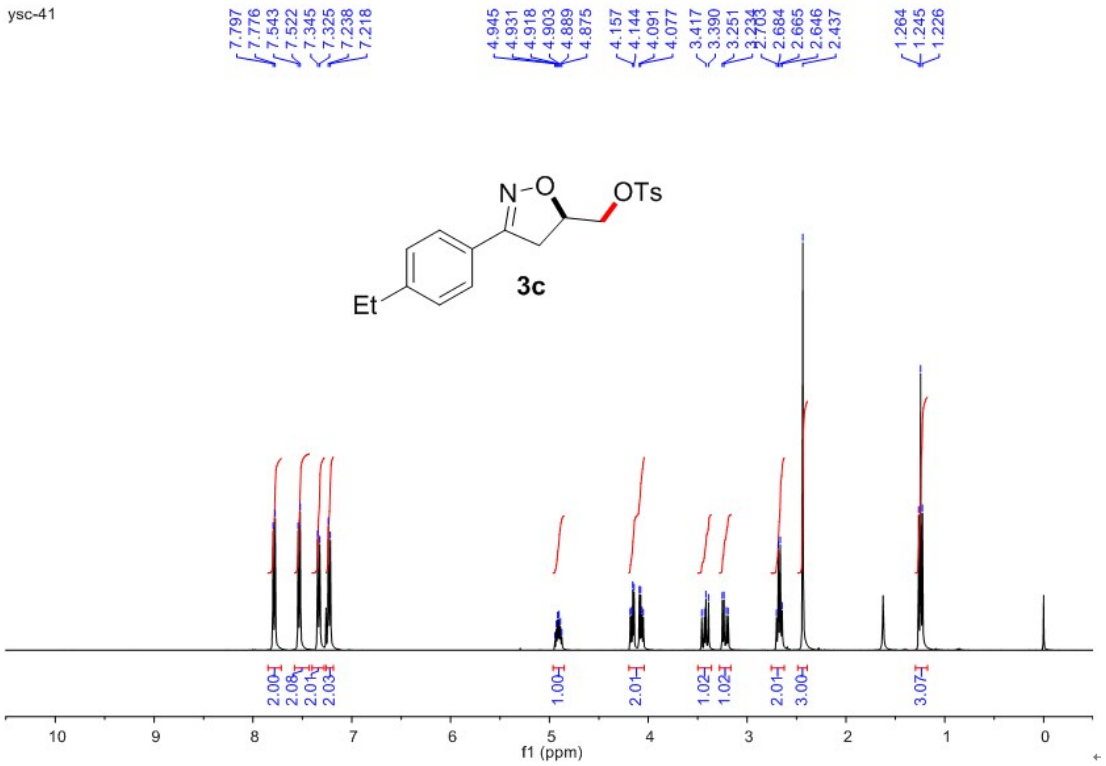
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


ysc-41
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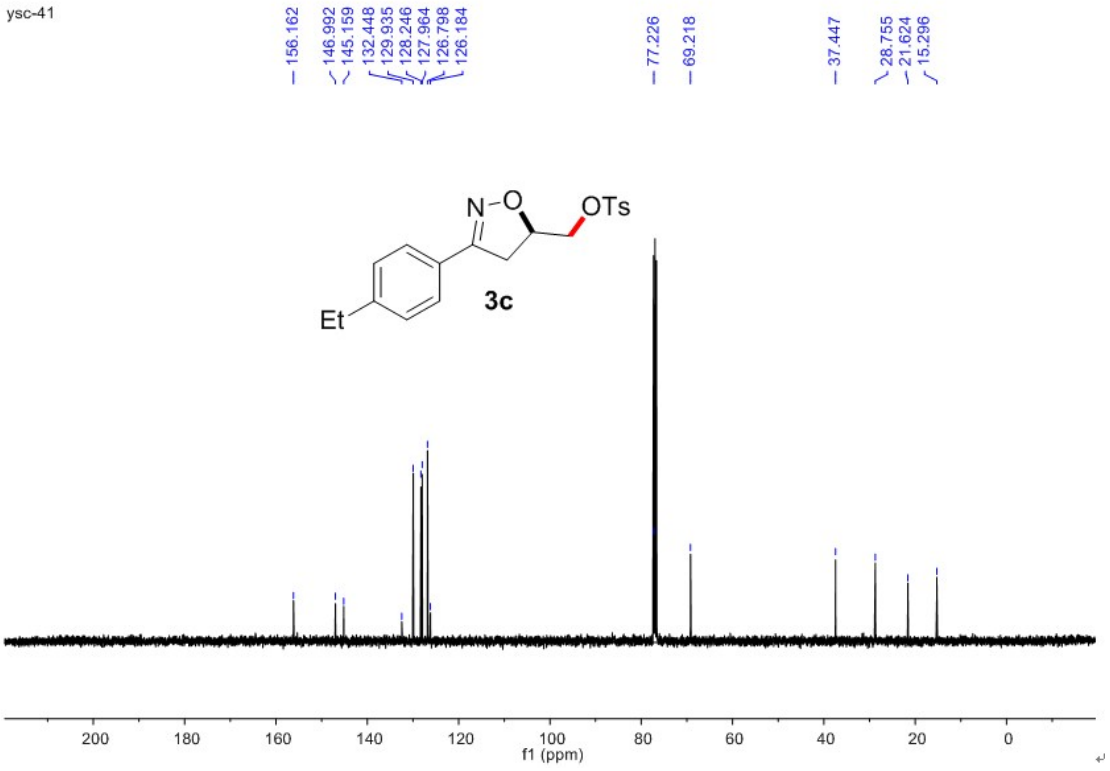
7.776
7.543
7.522
7.345
7.325
7.238
7.218
4.945
4.931
4.918
4.903
4.889
4.875
4.157
4.144
4.091
4.077
3.417
3.390
3.251
2.684
2.665
2.646
2.437
1.264
1.245
1.226
N
O
OTs
Et
3c
2.00
2.08
2.01
2.03
1.00
2.01
1.02
1.02
2.01
3.00
3.07
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


ysc-41
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146.992
145.159
132.448
129.935
128.246
127.964
126.798
126.184
77.226
69.218
37.447
28.755
21.624
15.296
N
O
OTs
Et
3c
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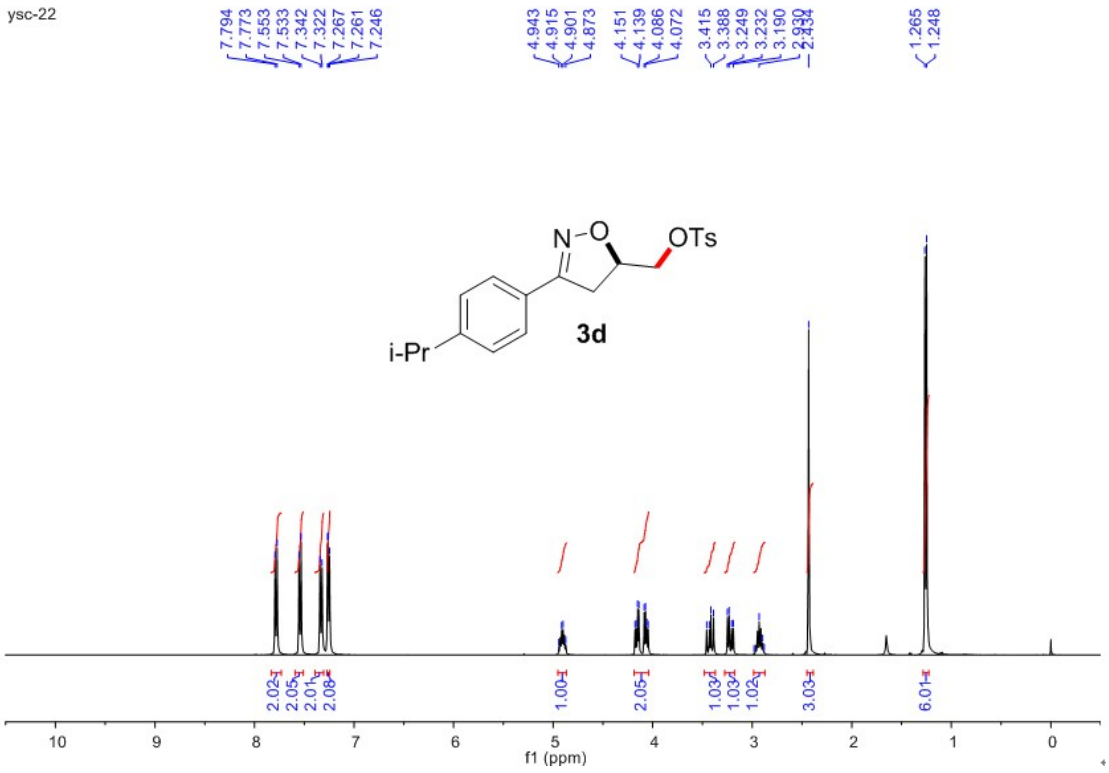
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

ysc-22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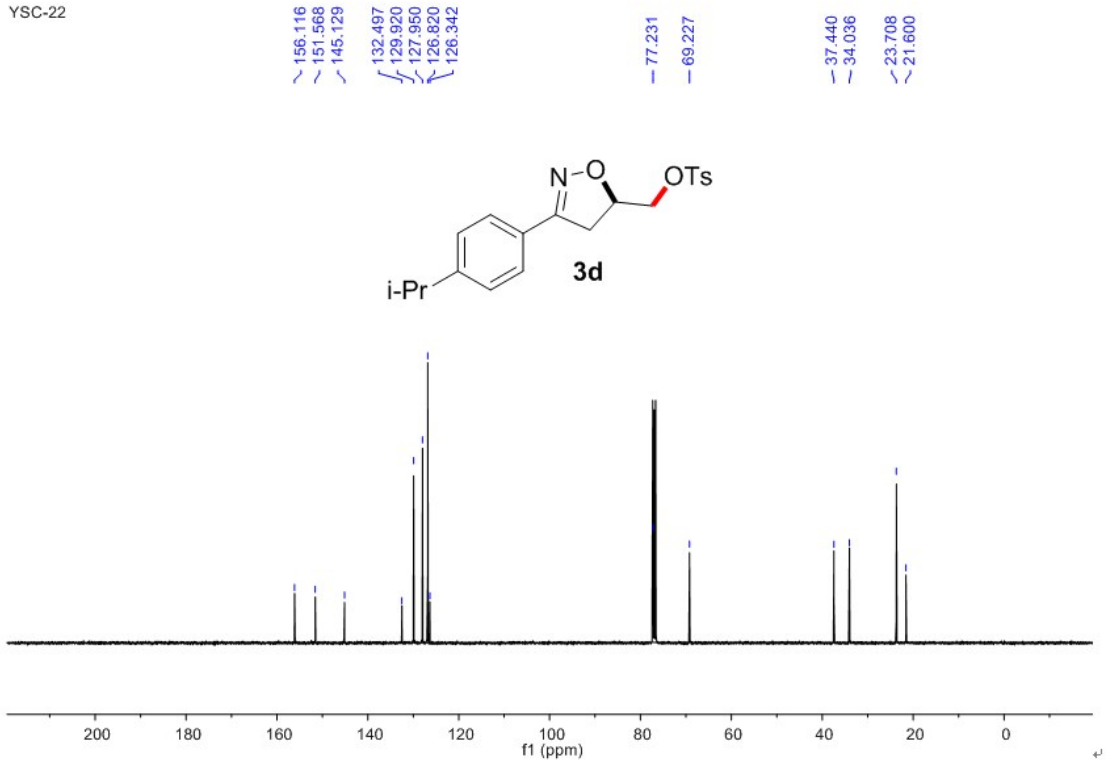
7.773
7.553
7.533
7.342
7.322
7.267
7.261
7.246
4.943
4.915
4.901
4.873
4.151
4.139
4.086
4.072
3.415
3.388
3.249
3.232
3.190
2.934
1.265
1.248
N
O
OTs
i-Pr
3d
2.02
2.05
2.01
2.08
1.00
2.05
1.03
1.03
1.02
3.03
6.01
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

YSC-22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151.568
145.129
132.497
129.920
127.950
126.820
126.342
77.231
69.227
37.440
34.036
23.708
21.600
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O
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i-Pr
3d
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140
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100
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20
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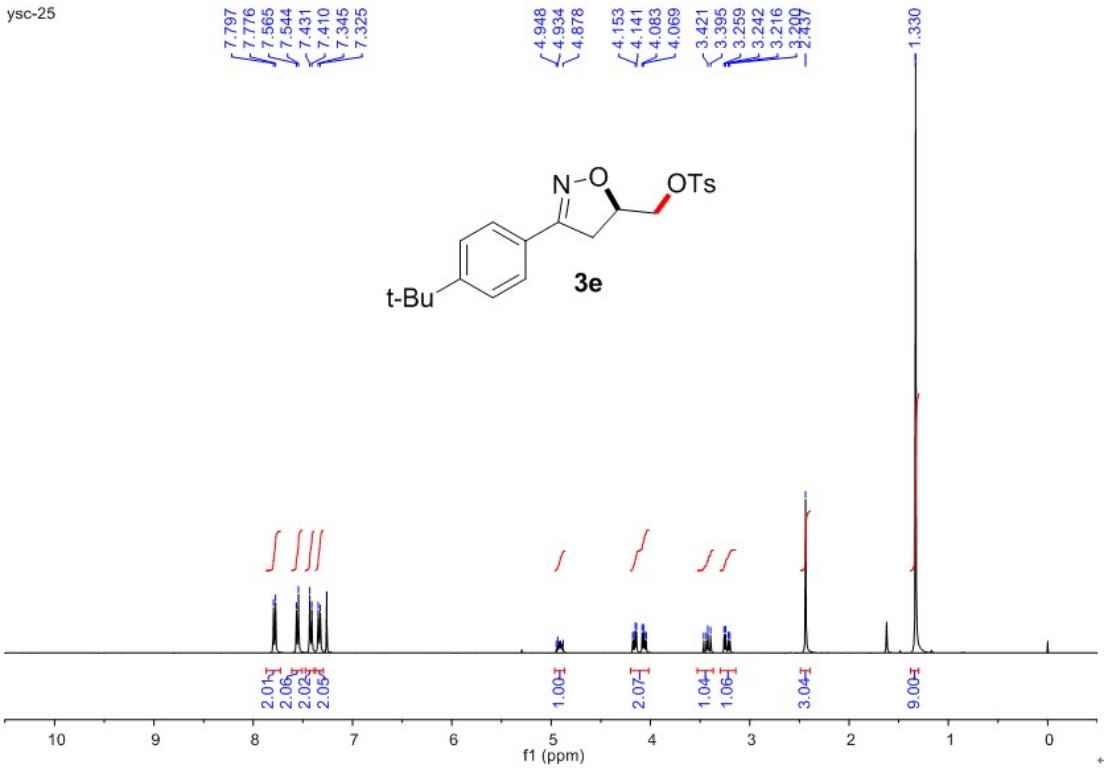
f1 (ppm)


ysc-25
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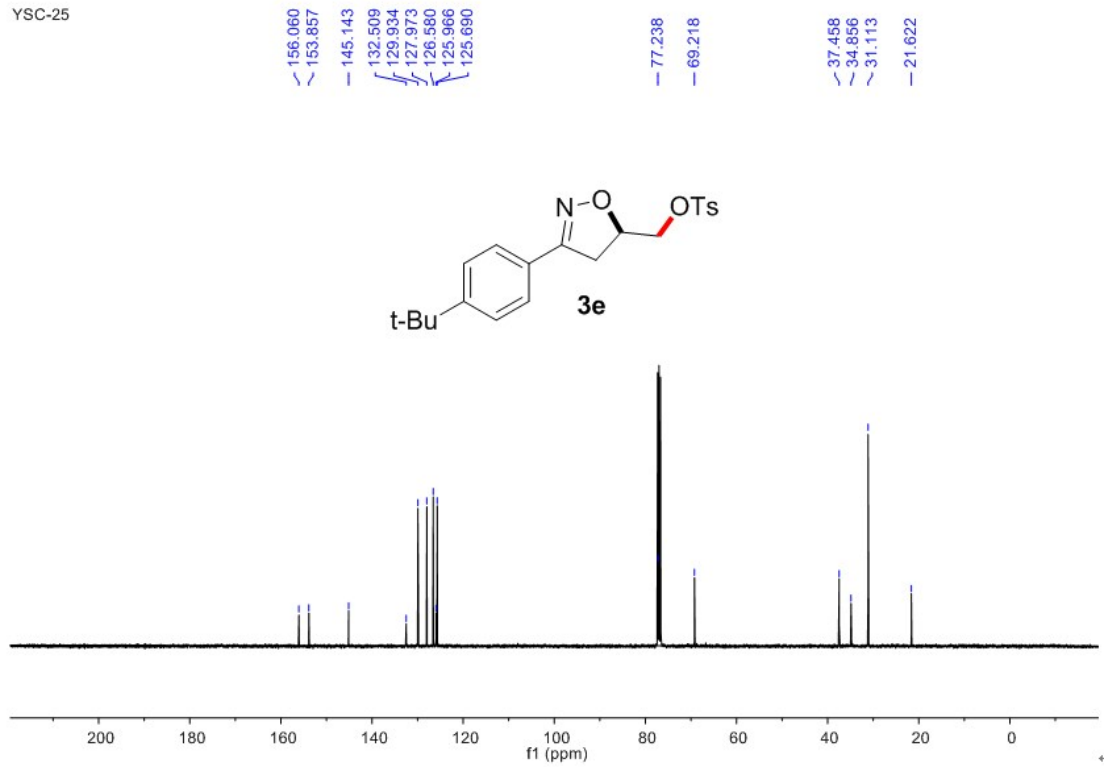
7.776
7.565
7.544
7.431
7.410
7.345
7.325
4.948
4.934
4.878
4.153
4.141
4.083
4.069
3.421
3.395
3.259
3.242
3.216
2.437
1.330
N
O
OTs
t-Bu
3e
2.01
2.06
2.02
2.05
1.00
2.07
1.04
1.06
3.04
9.00
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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132.509
129.934
127.973
126.580
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125.690
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69.218
37.458
34.856
31.113
21.622
N
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OTs
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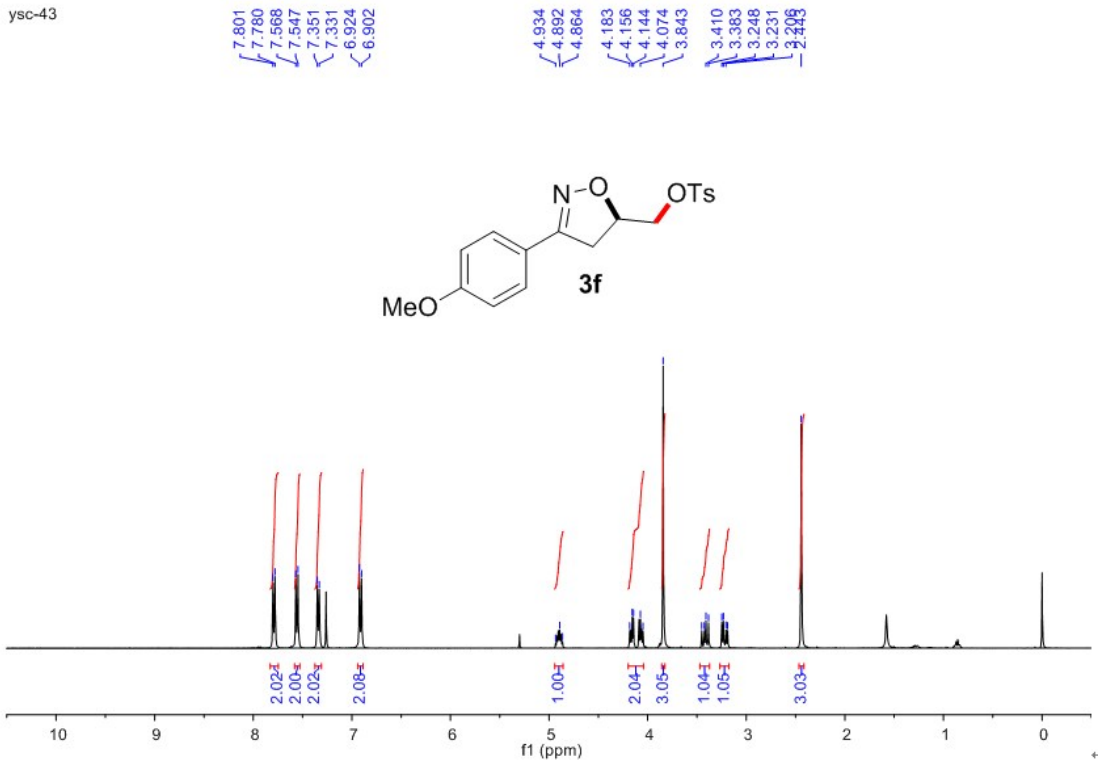
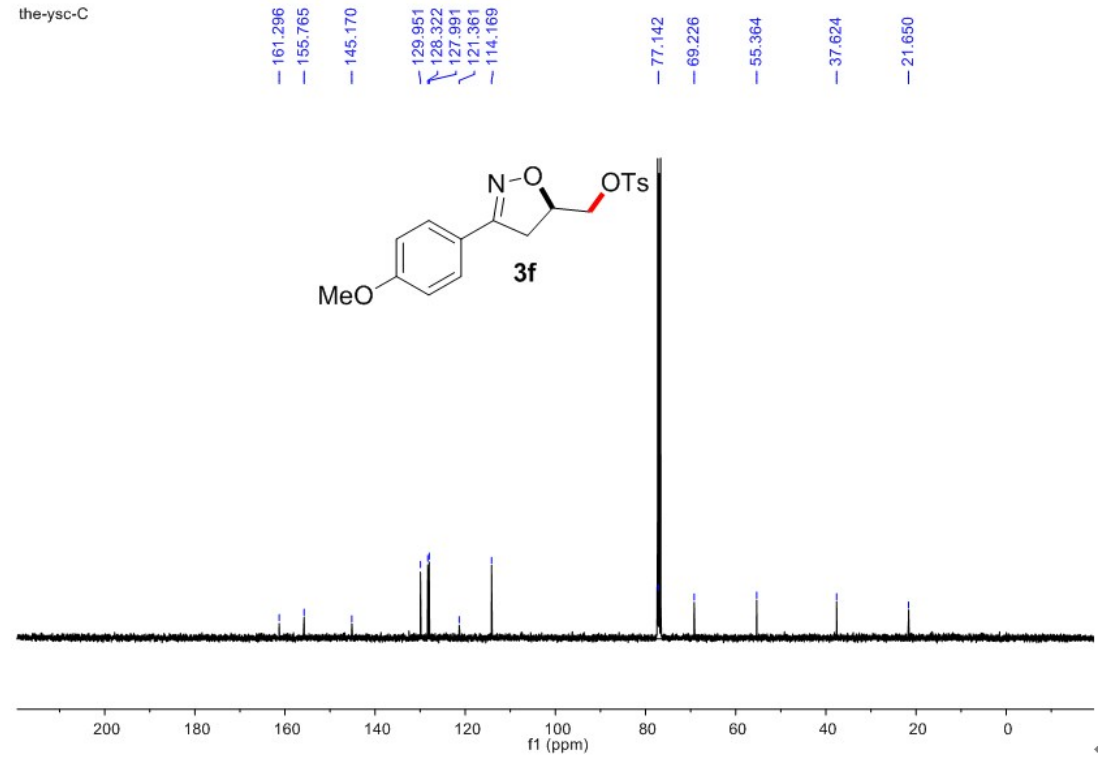
140
120
100
80
60
40
20
0
f1 (ppm)

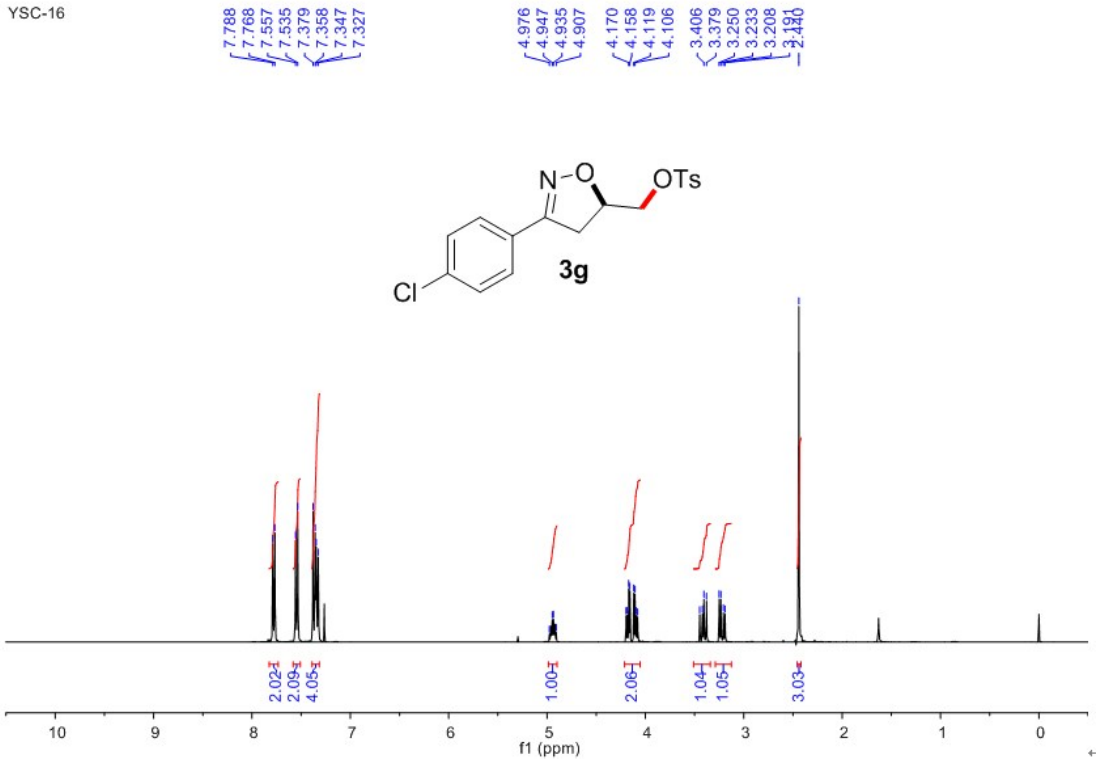
ysc-43



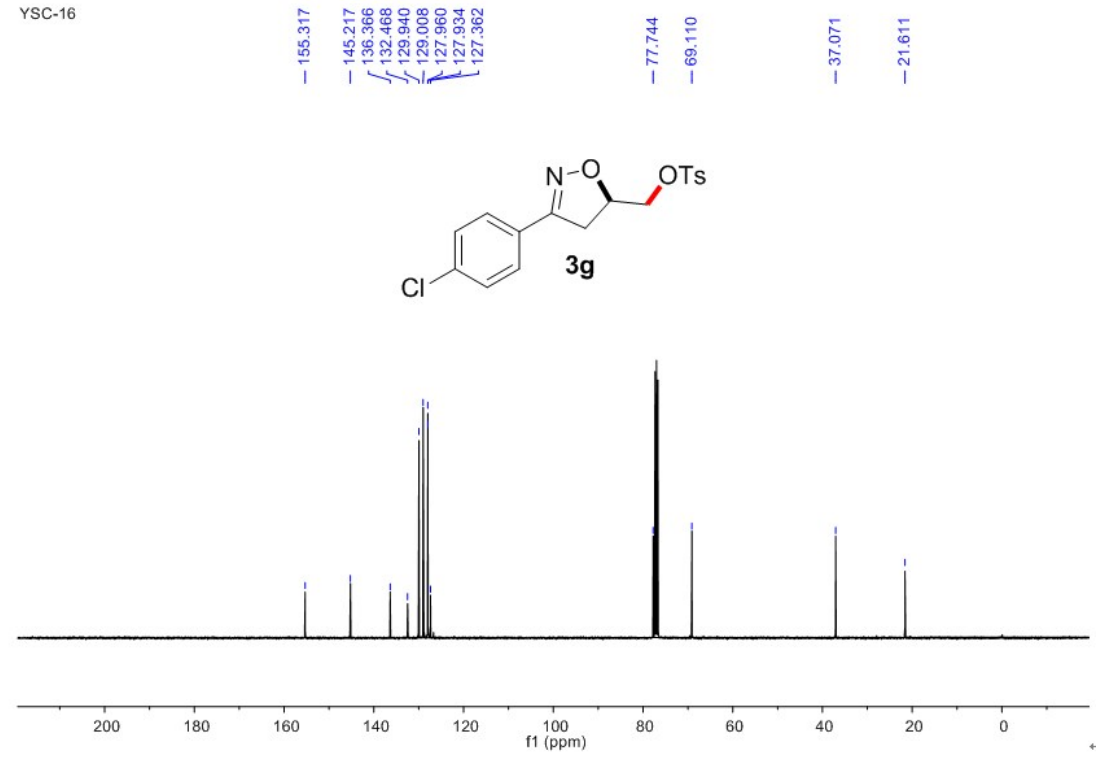
the-ysc-C



YSC-16


7.788
7.768
7.557
7.535
7.379
7.358
7.347
7.327
4.976
4.947
4.935
4.907
4.170
4.158
4.119
4.106
3.406
3.379
3.250
3.233
3.208
2.440
N
O
OTs
Cl
3g
2.02
2.09
4.05
1.00
2.06
1.04
1.05
3.03
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

YSC-16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132.468
129.940
129.008
127.960
127.934
127.362
77.744
69.110
37.071
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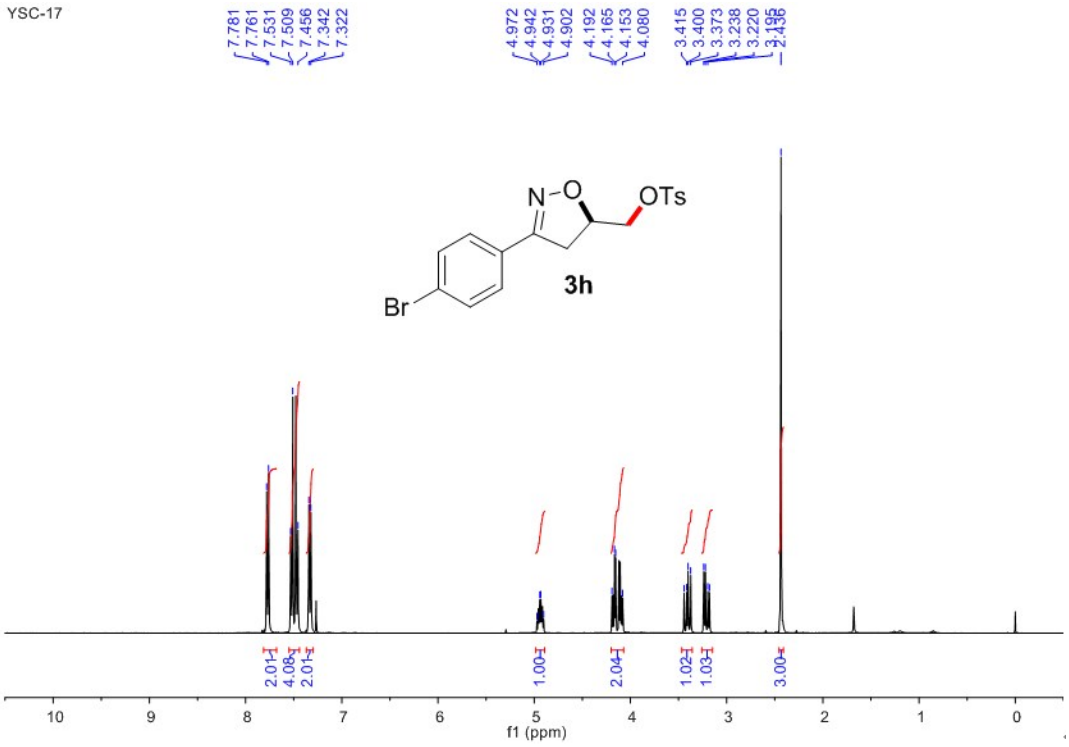
O
OTs
Cl
3g
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)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7.509
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7.342
7.322
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4.942
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4.902
4.192
4.165
4.153
4.080
3.415
3.400
3.373
3.238
3.220
2.438
N
O
OTs
Br
3h
2.01
4.08
2.01
1.00
2.04
1.02
1.03
3.00
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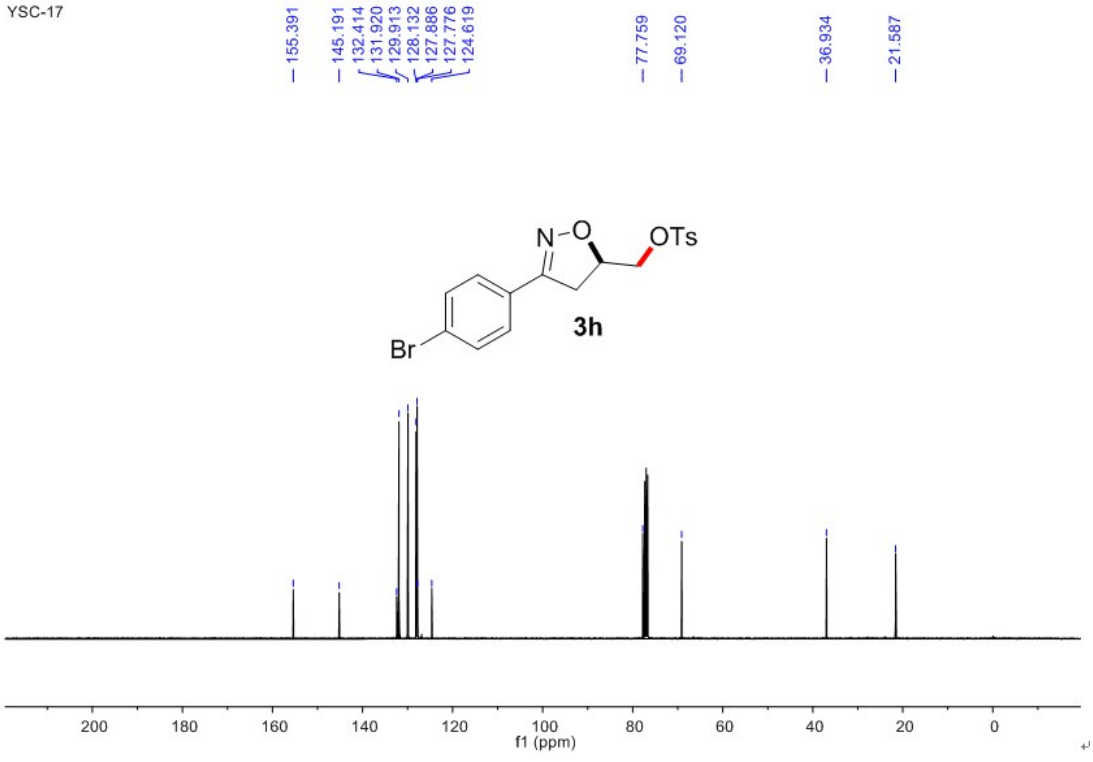
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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129.913
128.132
127.886
127.776
124.619
77.759
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36.934
21.587
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O
OTs
Br
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200
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140
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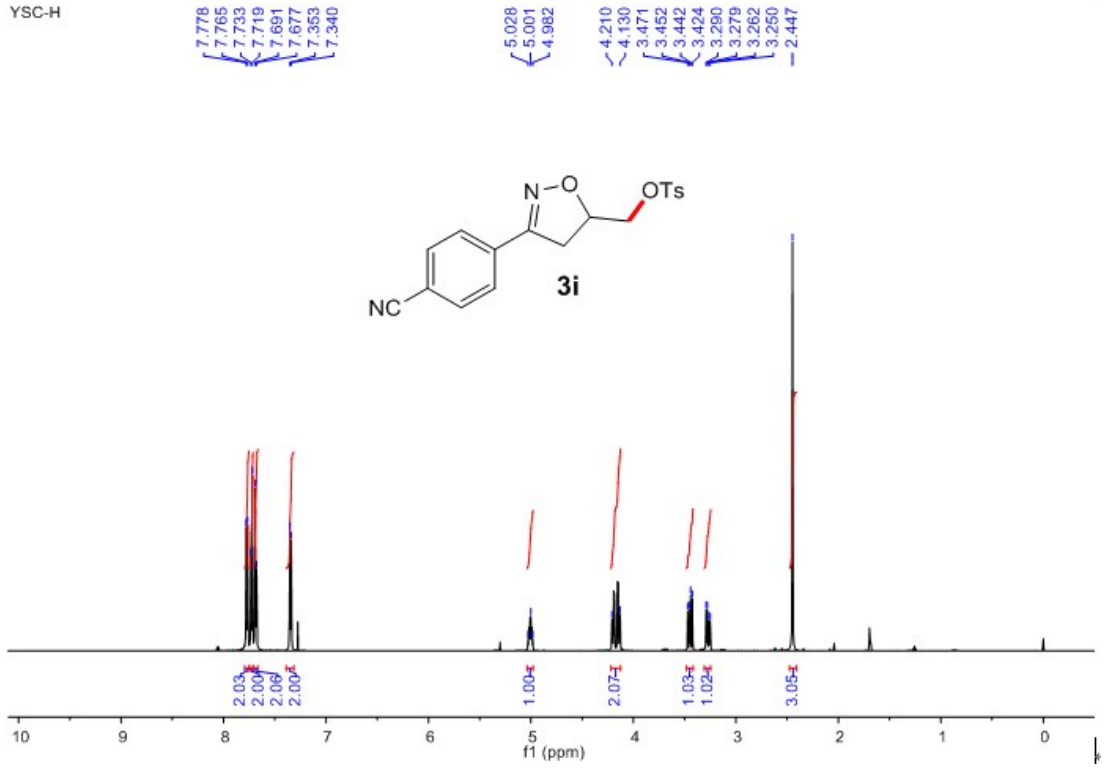
80
60
40
20
0
f1 (ppm)

YSC-H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7.719
7.691
7.677
7.353
7.340
5.028
5.001
4.982
4.210
4.130
3.471
3.452
3.442
3.424
3.290
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3.262
3.250
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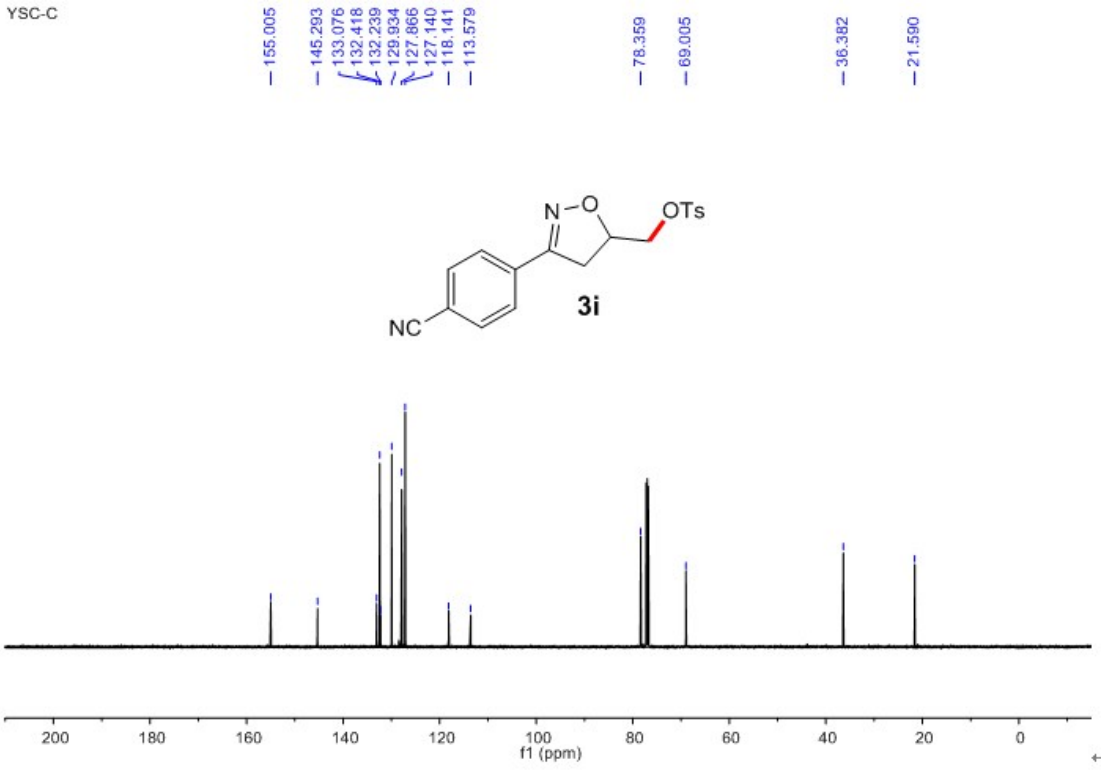
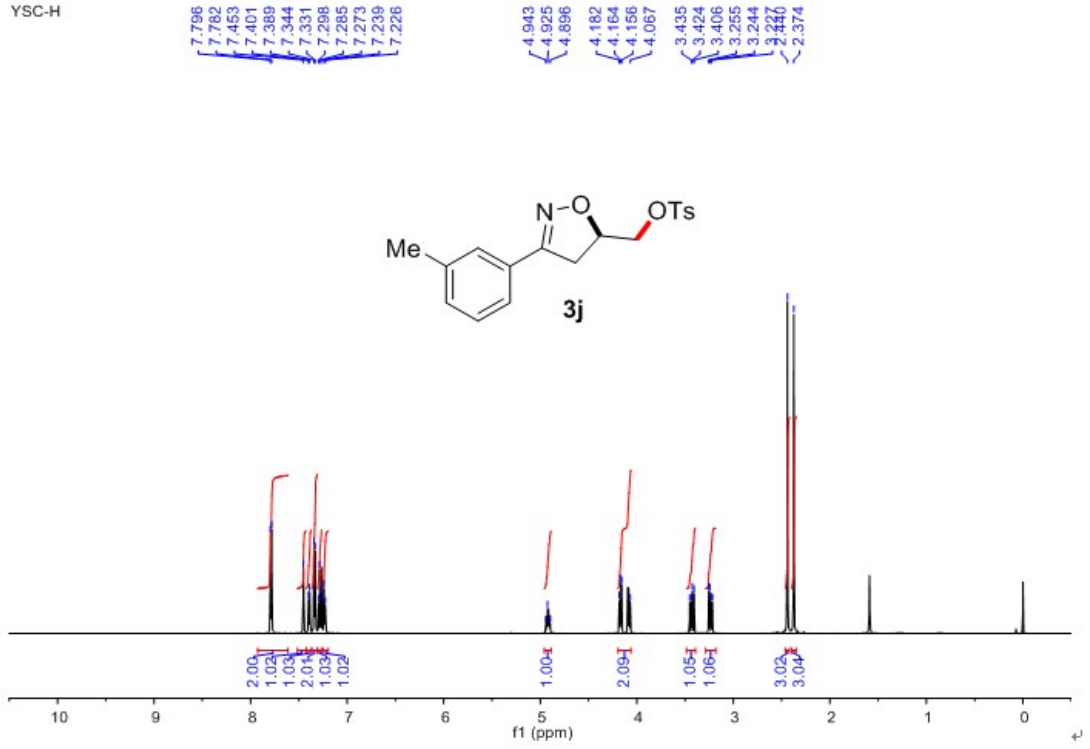
2.03
2.00
2.06
2.00
1.00
2.07
1.03
1.02
3.05
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

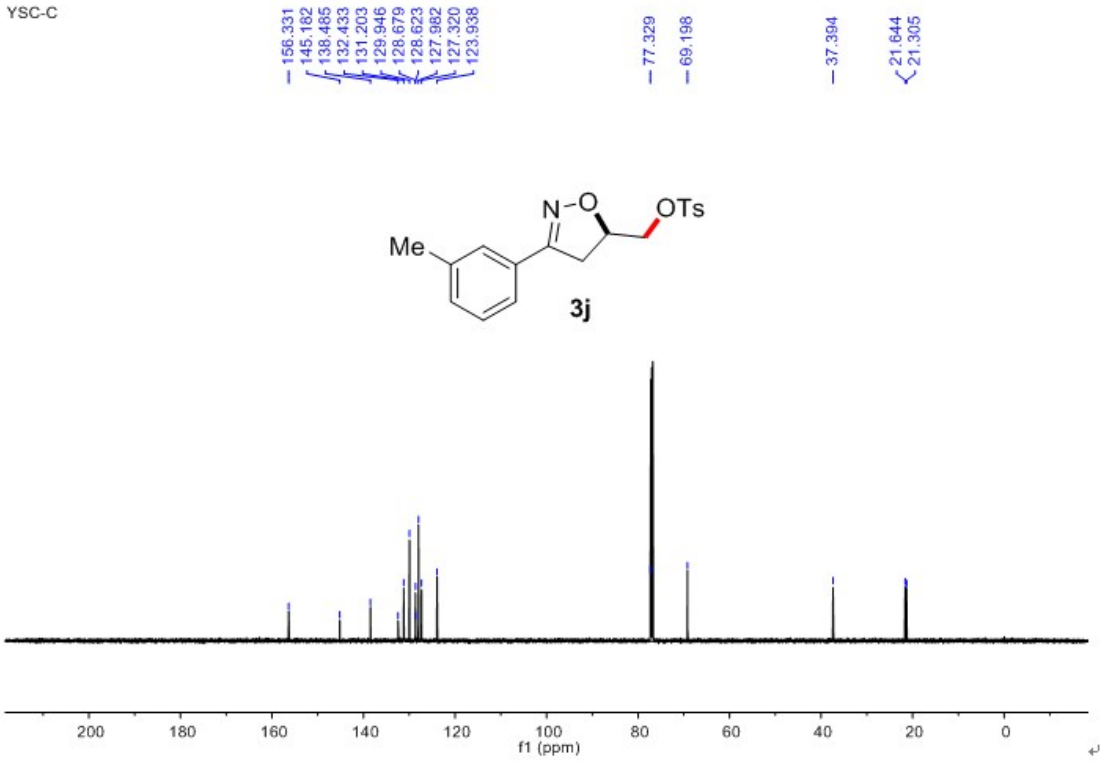
YSC-C


155.005
145.293
133.076
132.418
132.239
129.934
127.866
127.140
118.141
113.579
78.359
69.005
36.382
21.590
N
O
OTs
NC
3i
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

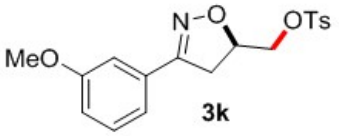
YSC-H


7.796
7.782
7.453
7.401
7.389
7.344
7.331
7.298
7.285
7.273
7.239
7.226
4.943
4.925
4.896
4.182
4.164
4.156
4.067
3.435
3.424
3.406
3.255
3.244
2.440
2.374
N
O
OTs
Me
3j
2.00
1.02
1.03
2.01
1.03
1.02
1.00
2.09
1.05
1.06
3.02
3.04
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

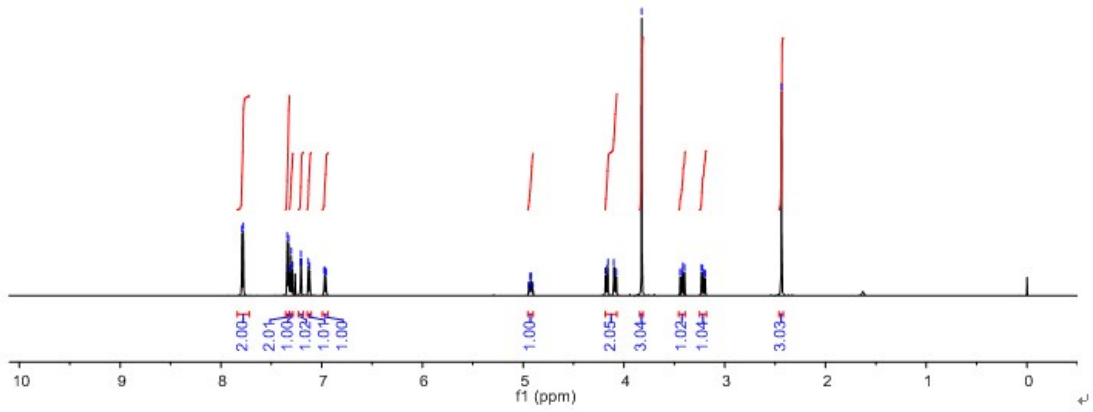
YSC-C


156.331
145.182
138.485
132.433
131.203
129.946
128.679
128.623
127.982
127.320
123.938
77.329
69.198
37.394
21.644
21.305
N
O
OTs
Me
3j
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

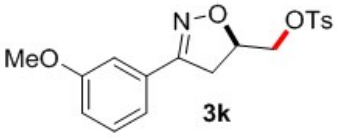
YSC-H



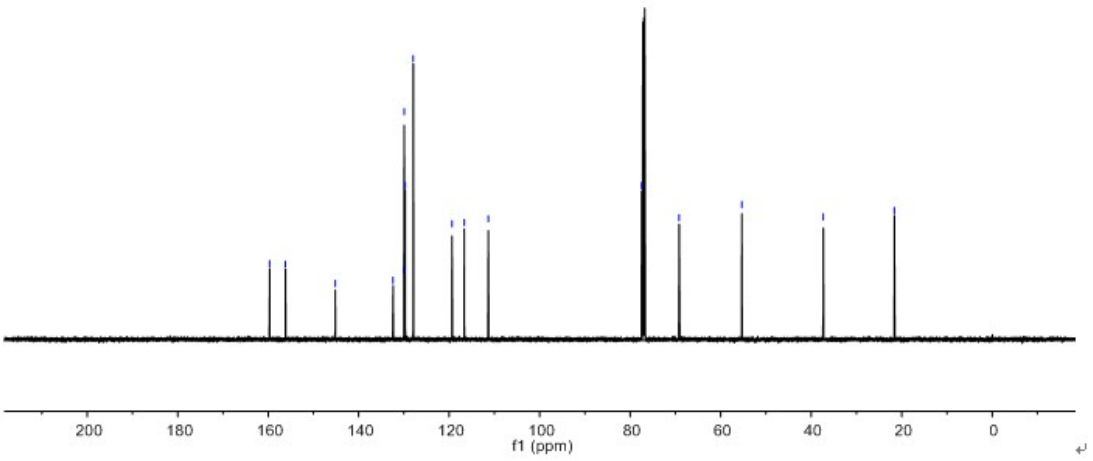
3k



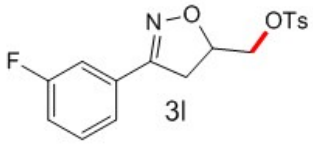
YSC-C



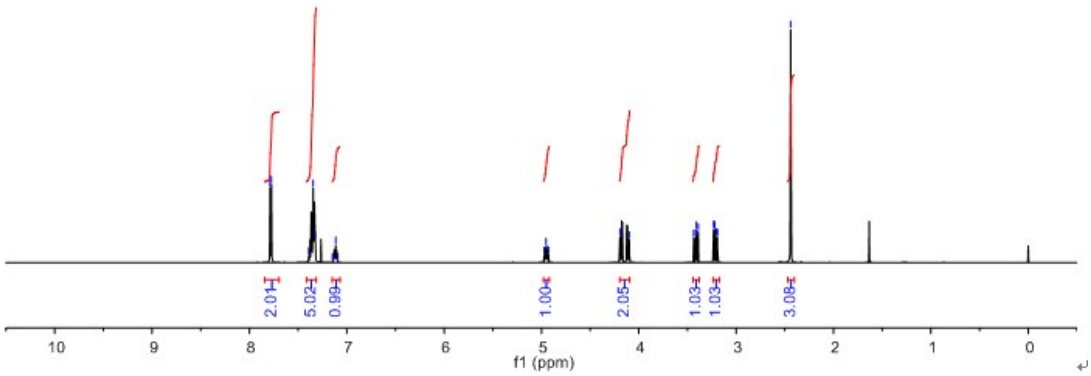
3k



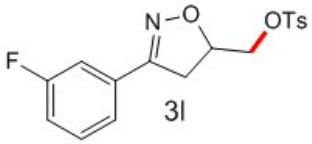
YSC-H



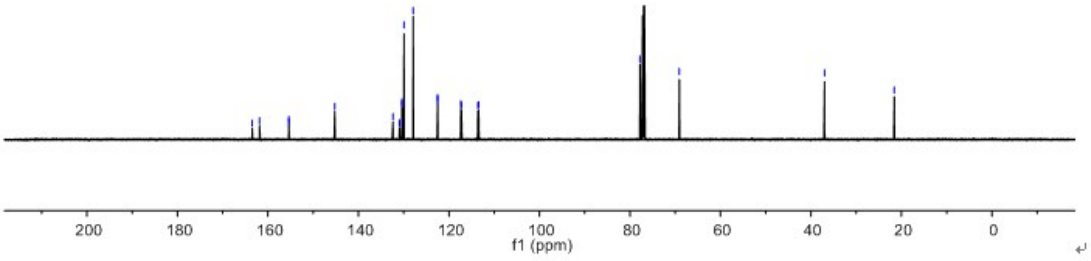
3l

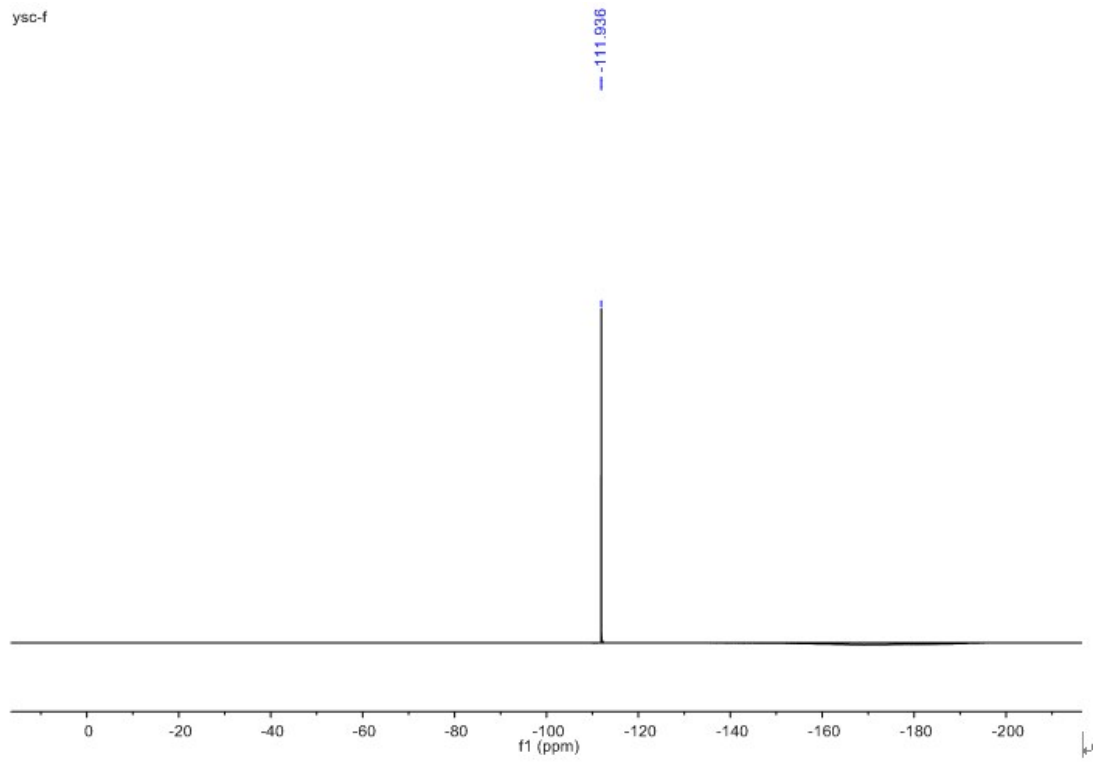


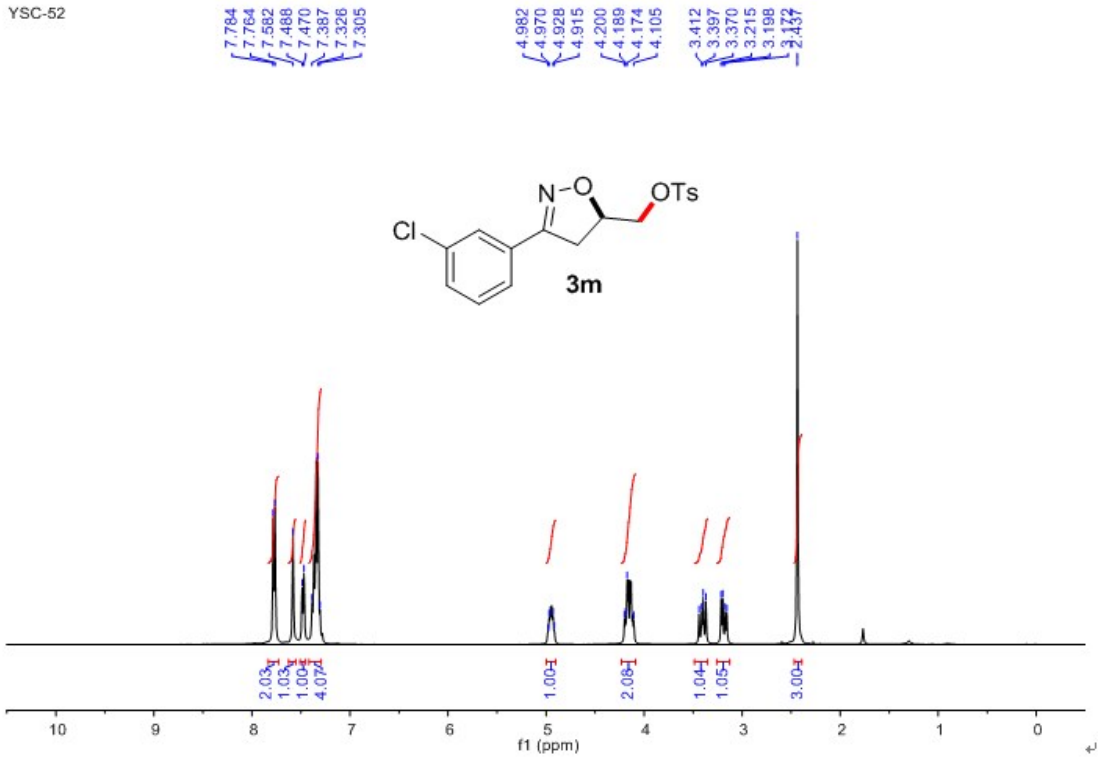
YSC-C



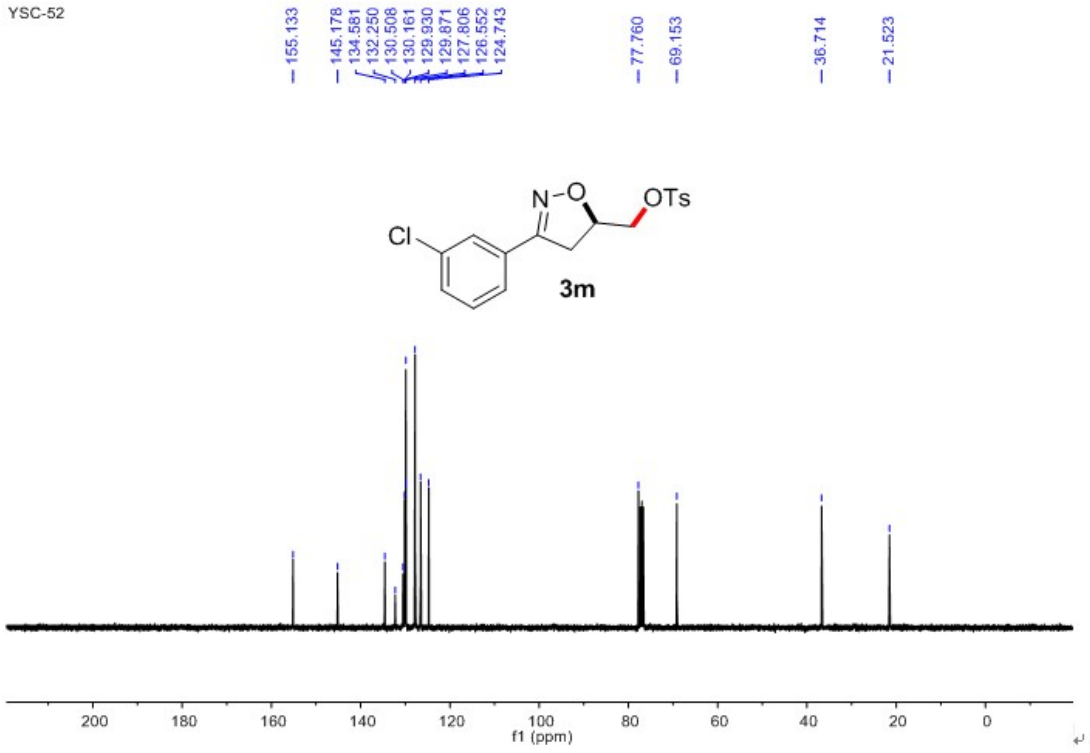
3l



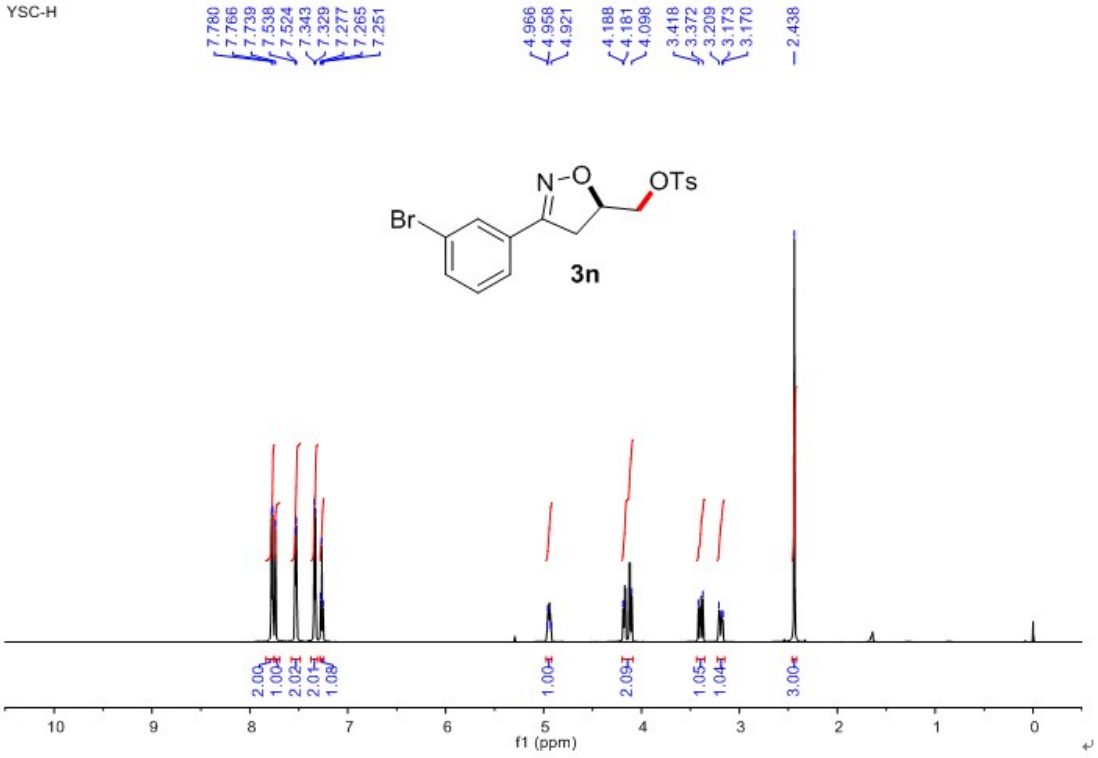

ysc-f
-111.936
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
f1 (ppm)


YSC-52
7.784
7.764
7.582
7.488
7.470
7.387
7.326
7.305
4.982
4.970
4.928
4.915
4.200
4.189
4.174
4.105
3.412
3.397
3.370
3.215
3.198
3.172
2.437
N
O
OTs
Cl
3m
2.03
1.03
1.00
4.07
1.00
2.08
1.04
1.05
3.00
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

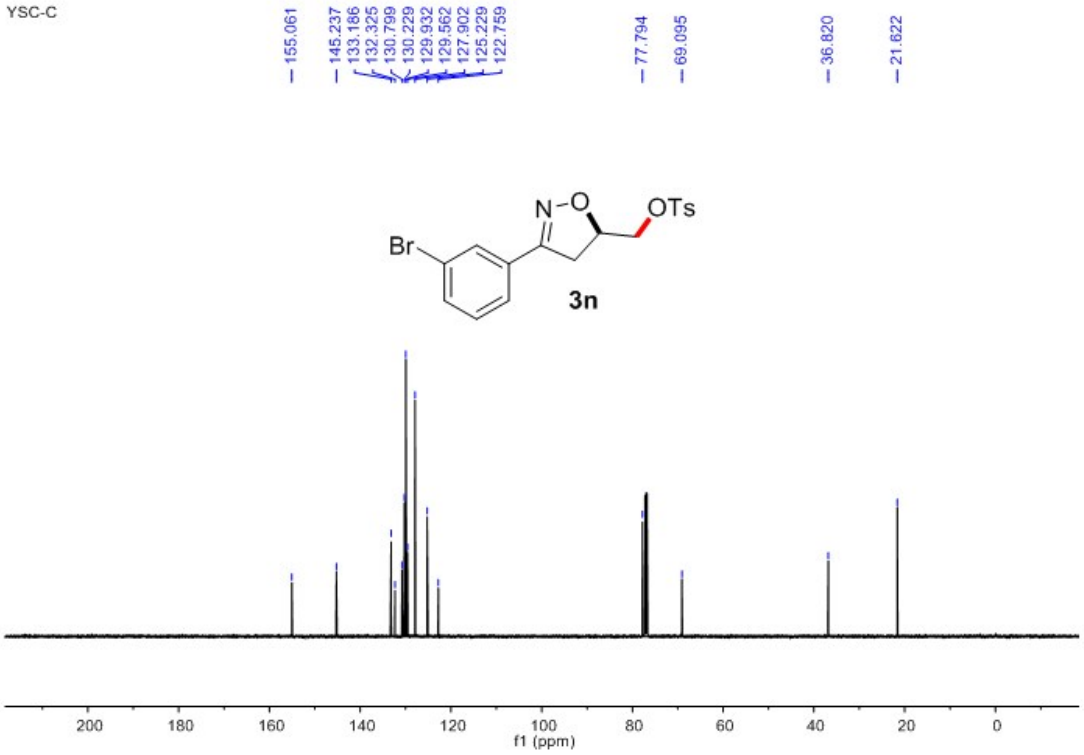
YSC-52


155.133
145.178
134.581
132.250
130.508
130.161
129.930
129.871
127.806
126.552
124.743
77.760
69.153
36.714
21.523
N
O
OTs
Cl
3m
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

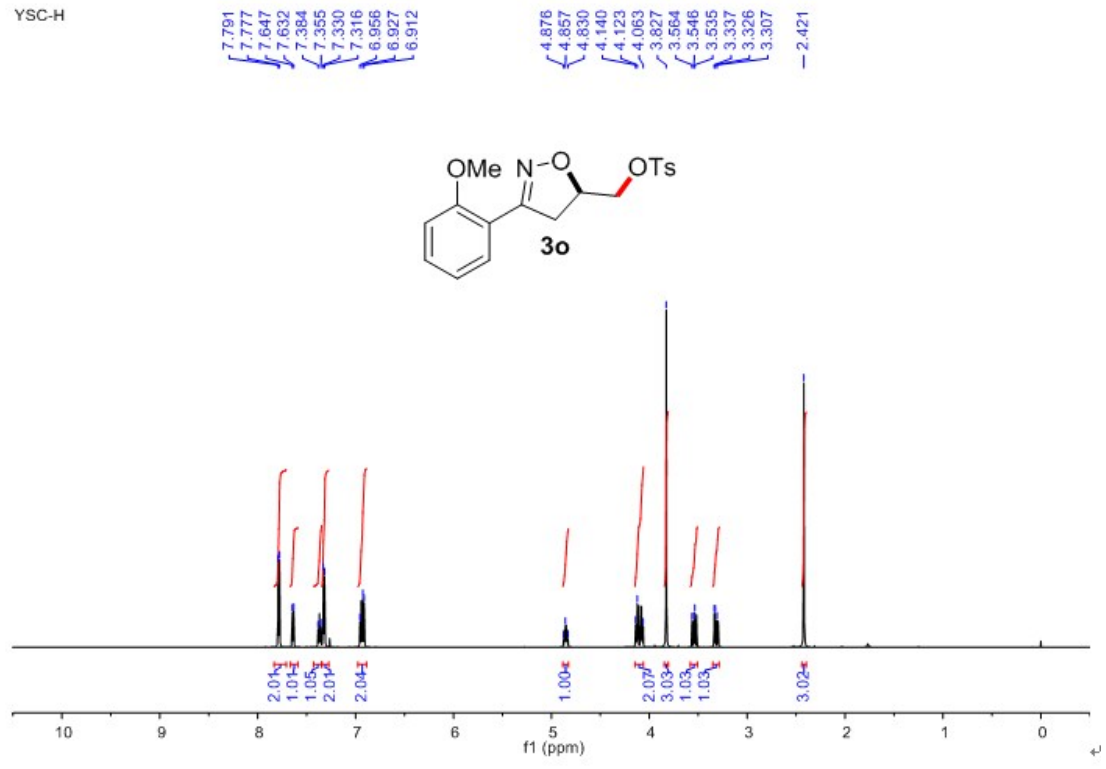
YSC-H


7.780
7.766
7.739
7.538
7.524
7.343
7.329
7.277
7.265
7.251
4.966
4.958
4.921
4.188
4.181
4.098
3.418
3.372
3.209
3.173
3.170
2.438
N
O
OTs
Br
3n
2.00
1.00
2.02
2.01
1.08
1.00
2.09
1.05
1.04
3.00
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

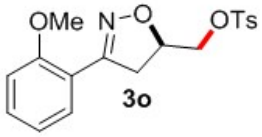
YSC-C


155.061
145.237
133.186
132.325
130.799
130.229
129.932
129.562
127.902
125.229
122.759
77.794
69.095
36.820
21.622
N
O
OTs
Br
3n
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

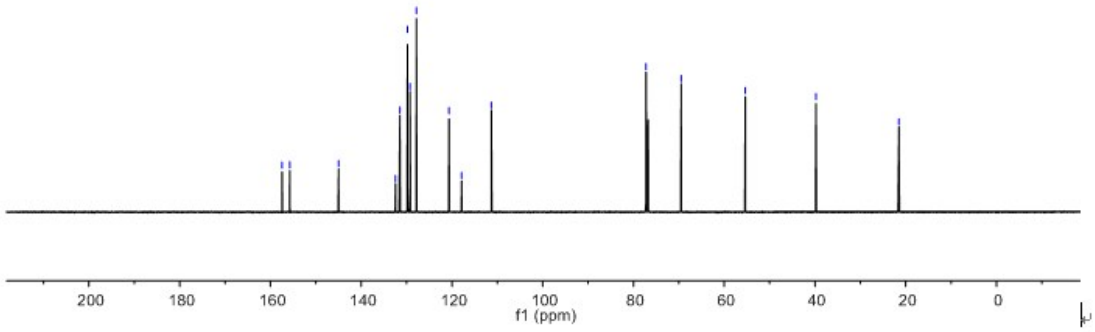
YSC-H


7.791
7.777
7.647
7.632
7.384
7.355
7.330
7.316
6.956
6.927
6.912
4.876
4.857
4.830
4.140
4.123
4.063
3.827
3.564
3.546
3.535
3.337
3.326
3.307
2.421
OMe
N
O
OTs
3o
2.01
1.01
1.05
2.01
2.04
1.00
2.07
3.03
1.03
1.03
3.02
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

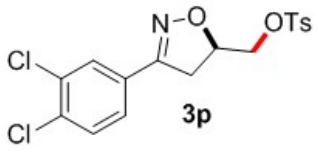
YSC-C



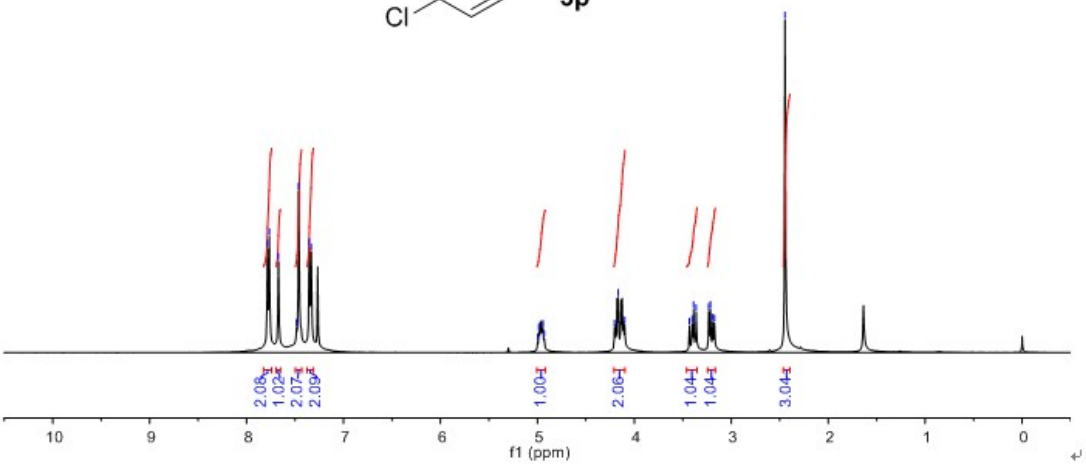
3o



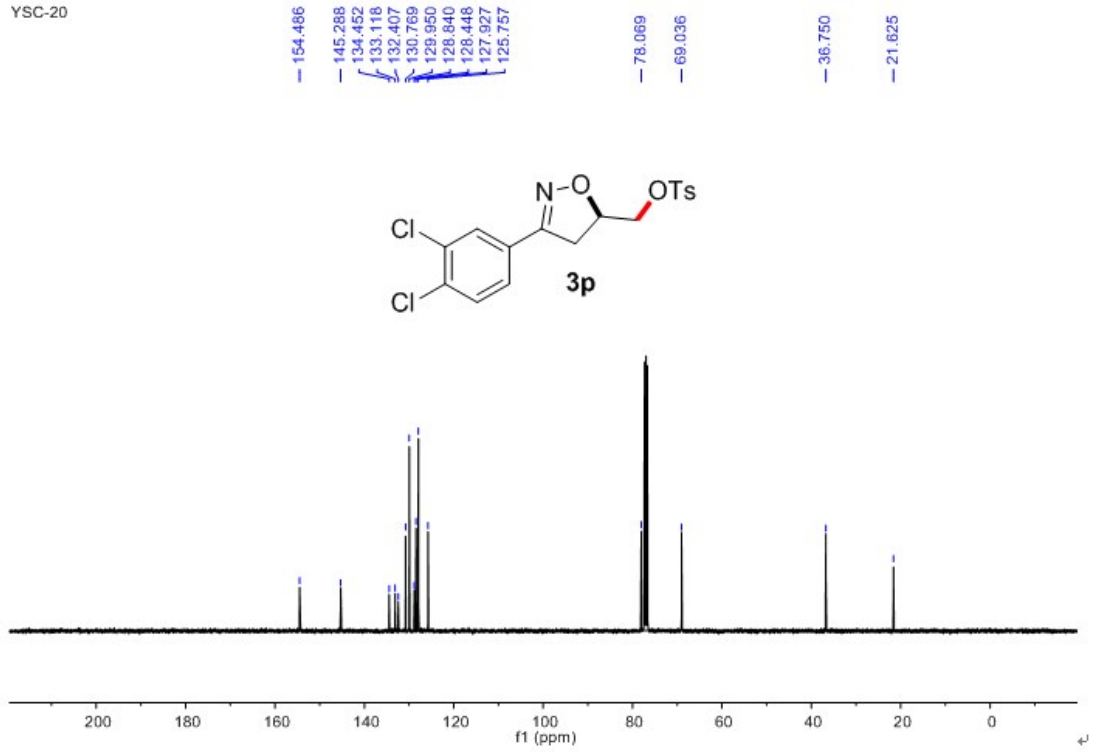
ysc-20



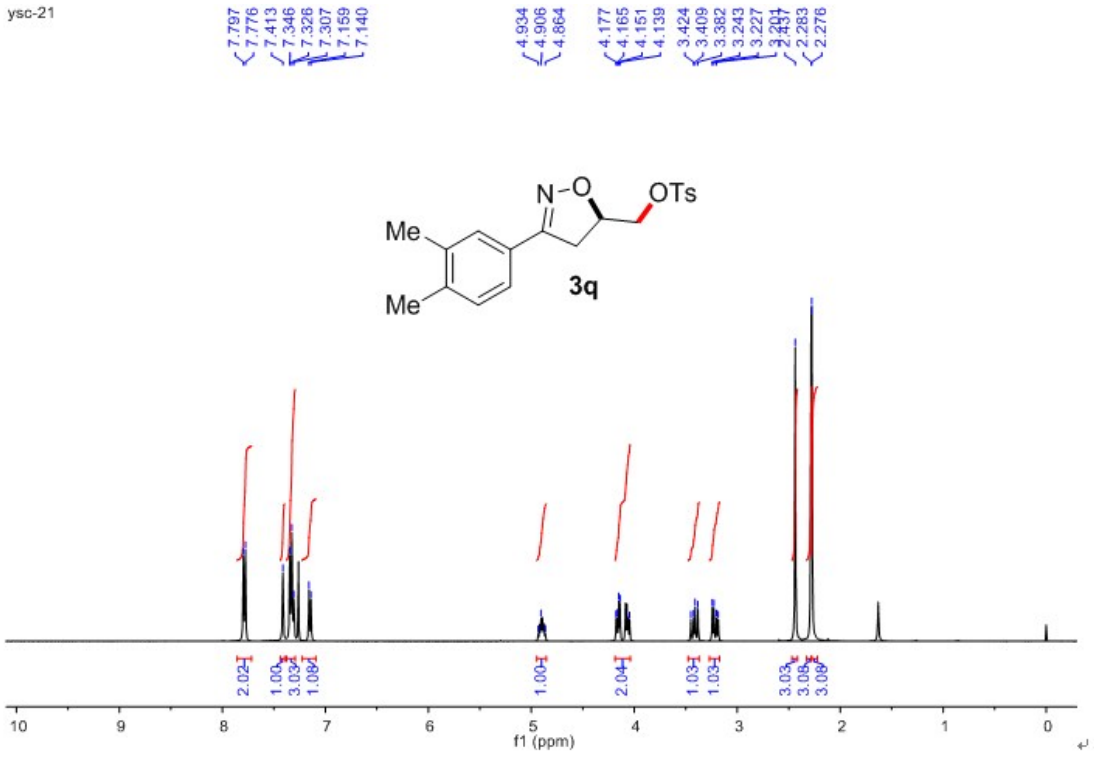
3p



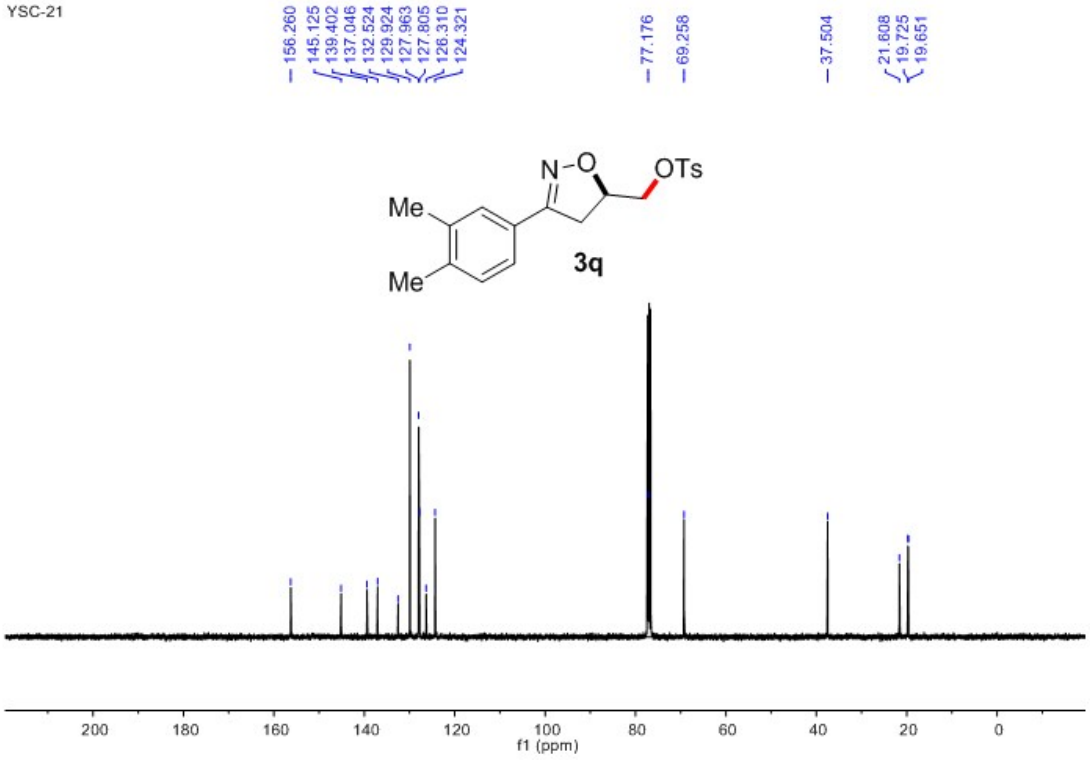
YSC-20


154.486
145.288
134.452
133.118
132.407
130.769
129.950
128.840
128.448
127.927
125.757
78.069
69.036
36.750
21.625
N
O
OTs
Cl
Cl
3p
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

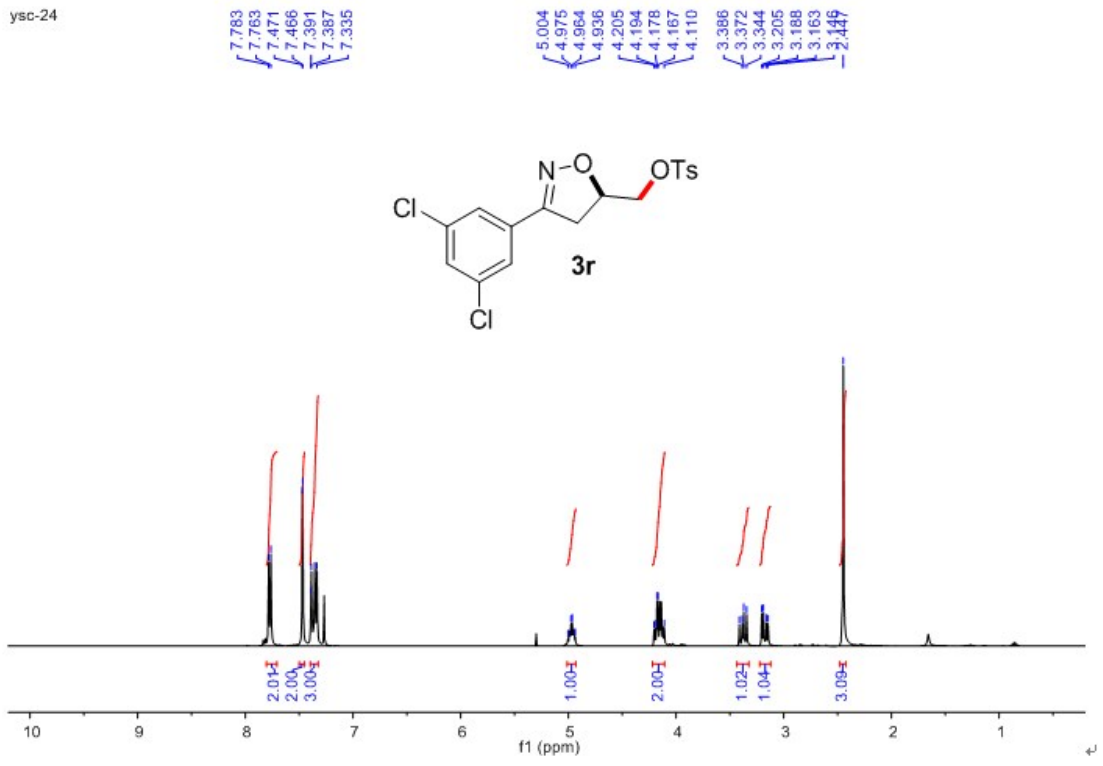
ysc-21


7.797
7.776
7.413
7.346
7.326
7.307
7.159
7.140
4.934
4.906
4.864
4.177
4.165
4.151
4.139
3.424
3.409
3.382
3.243
3.227
2.283
2.276
N
O
OTs
Me
Me
3q
2.02
1.00
3.03
1.08
1.00
2.04
1.03
1.03
3.03
3.08
3.08
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

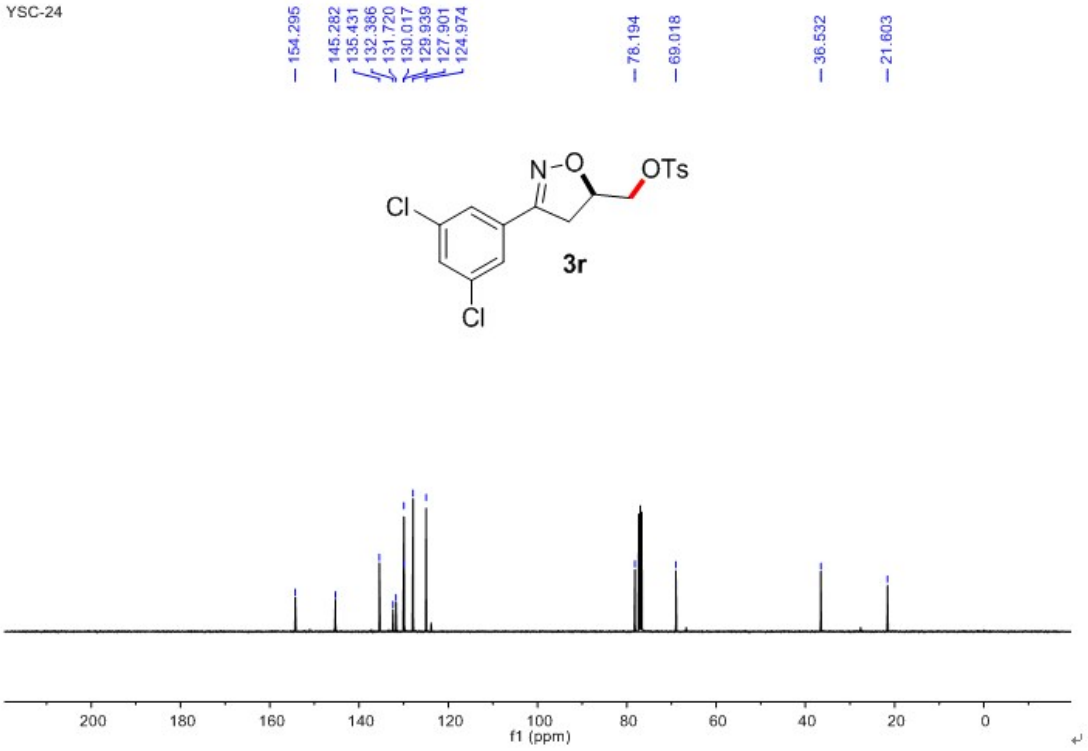
YSC-21


156.260
145.125
139.402
137.046
132.524
129.924
127.963
127.805
126.310
124.321
77.176
69.258
37.504
21.608
19.725
19.651
N
O
OTs
Me
Me
3q
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

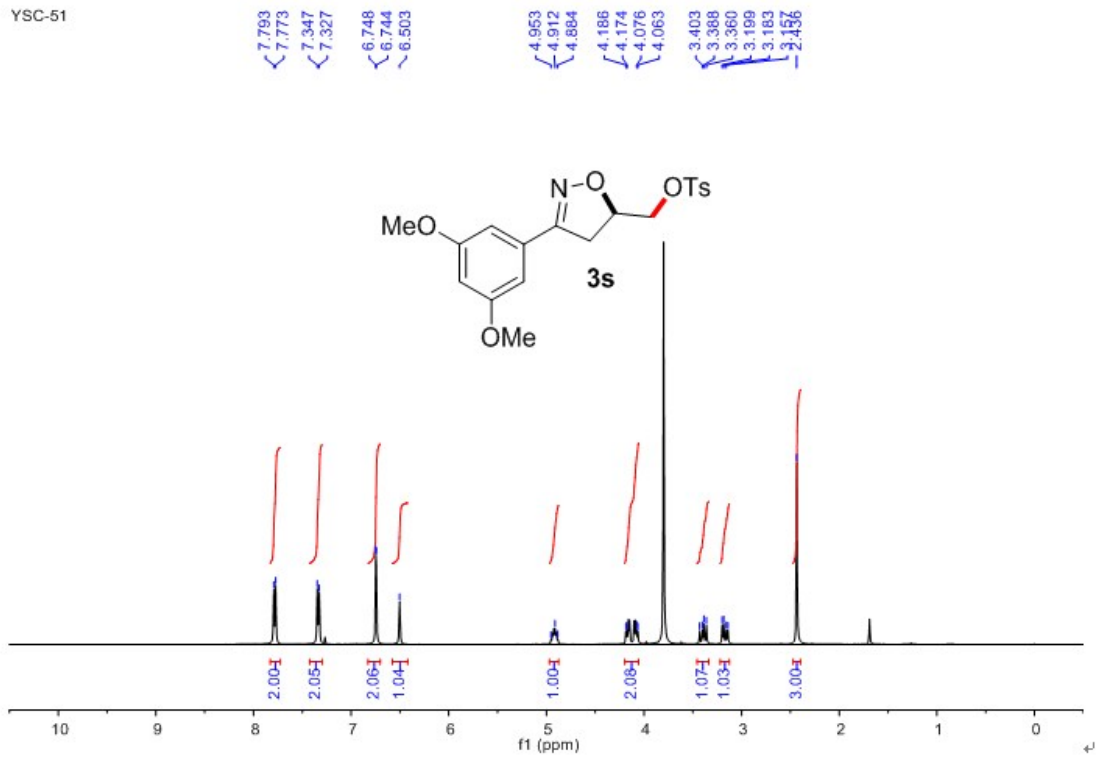
ysc-24


7.783
7.763
7.471
7.466
7.391
7.387
7.335
5.004
4.975
4.964
4.936
4.205
4.194
4.178
4.167
4.110
3.386
3.372
3.344
3.205
3.188
3.163
2.447
N
O
OTs
Cl
Cl
3r
2.01
2.00
3.00
1.00
2.00
1.02
1.04
3.09
10
9
8
7
6
5
4
3
2
1
f1 (ppm)

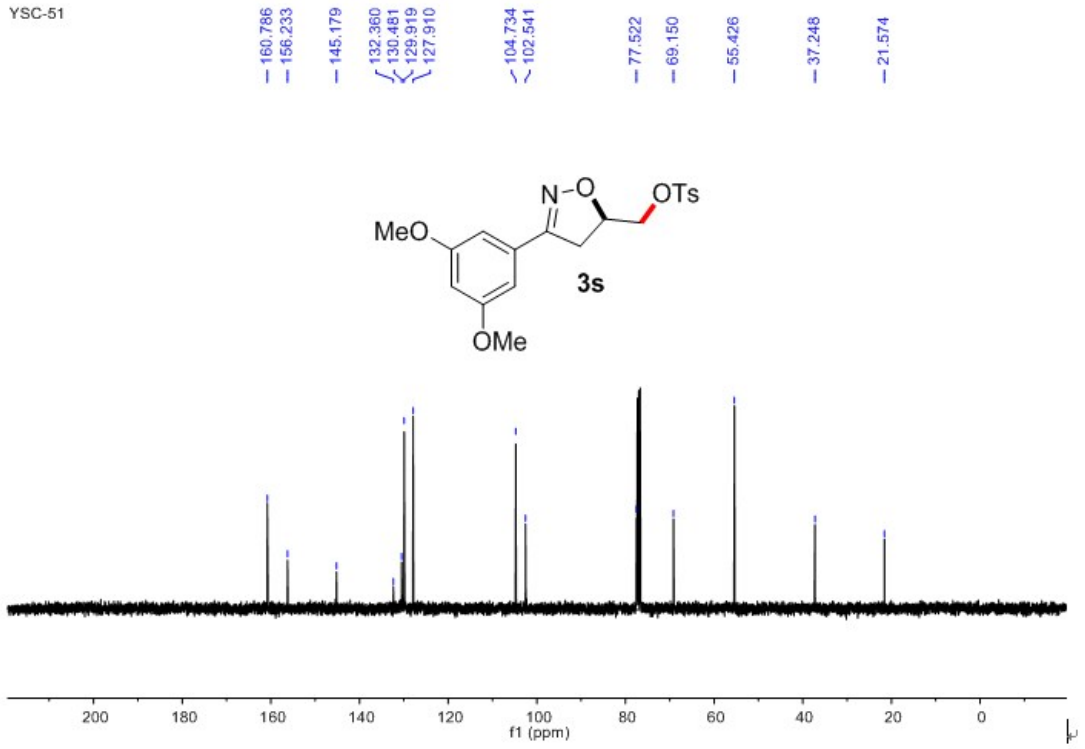
YSC-24


154.295
145.282
135.431
132.386
131.720
130.017
129.939
127.901
124.974
78.194
69.018
36.532
21.603
N
O
OTs
Cl
Cl
3r
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

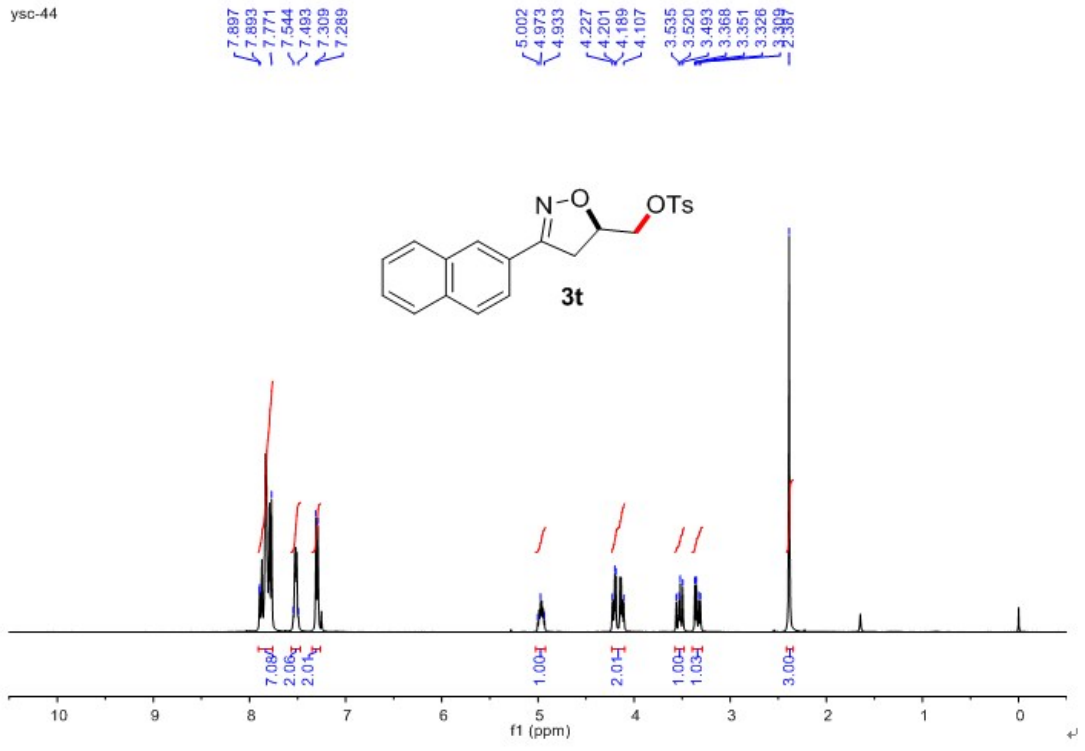
YSC-51

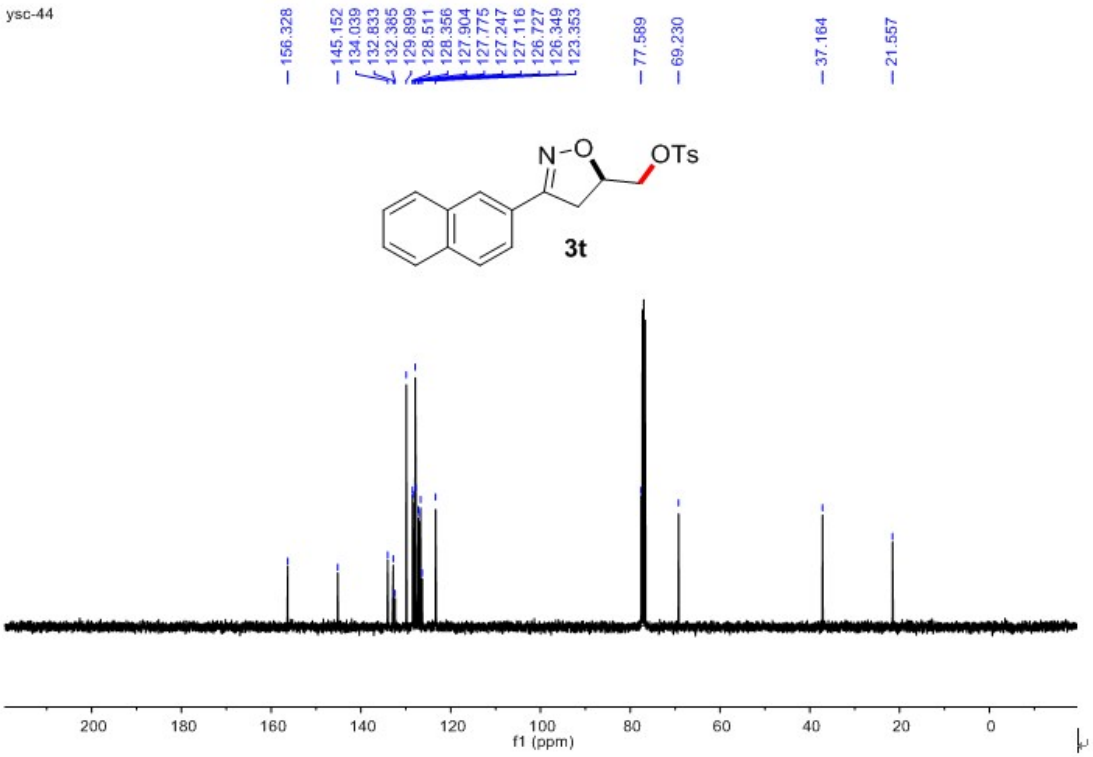

7.793
7.773
7.347
7.327
6.748
6.744
6.503
4.953
4.912
4.884
4.186
4.174
4.076
4.063
3.403
3.388
3.360
3.199
3.183
2.456
N
O
OTs
MeO
OMe
3s
2.00
2.05
2.06
1.04
1.00
2.08
1.07
1.03
3.00
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

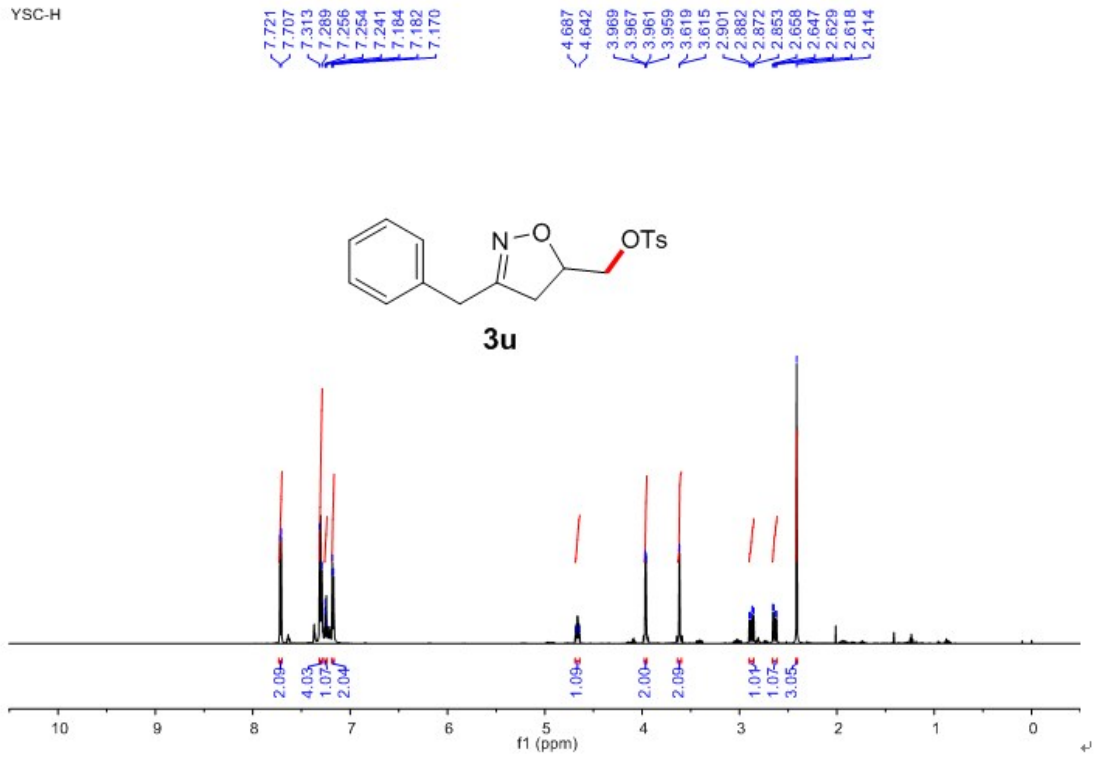
YSC-51


160.786
156.233
145.179
132.360
130.481
129.919
127.910
104.734
102.541
77.522
69.150
55.426
37.248
21.574
N
O
OTs
MeO
OMe
3s
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

ysc-44

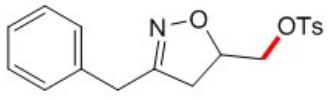

7.897
7.893
7.771
7.544
7.493
7.309
7.289
5.002
4.973
4.933
4.227
4.201
4.189
4.107
3.535
3.520
3.493
3.368
3.351
3.326
3.309
2.387
N
O
OTs
3t
7.08
2.06
2.01
1.00
2.01
1.00
1.03
3.00
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


ysc-44
156.328
145.152
134.039
132.833
132.385
129.899
128.511
128.356
127.904
127.775
127.247
127.116
126.727
126.349
123.353
77.589
69.230
37.164
21.557
N
O
OTs
3t
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

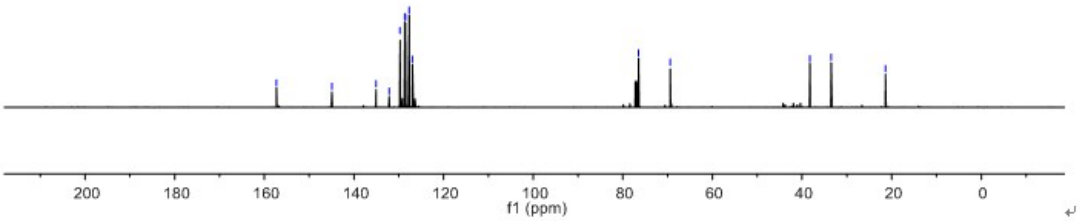

YSC-H
7.721
7.707
7.313
7.289
7.256
7.254
7.241
7.184
7.182
7.170
4.687
4.642
3.969
3.967
3.961
3.959
3.619
3.615
2.901
2.882
2.872
2.853
2.658
2.647
2.629
2.618
2.414
N
O
OTs
3u
2.09
4.03
1.07
2.04
1.09
2.00
2.09
1.01
1.07
3.05
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

YSC-C

157.295
144.925
135.075
132.155
129.722
128.650
128.513
127.682
126.937
76.480
69.427
38.252
33.498
21.392

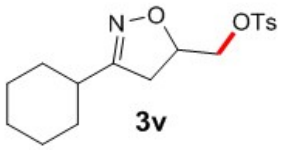


3u

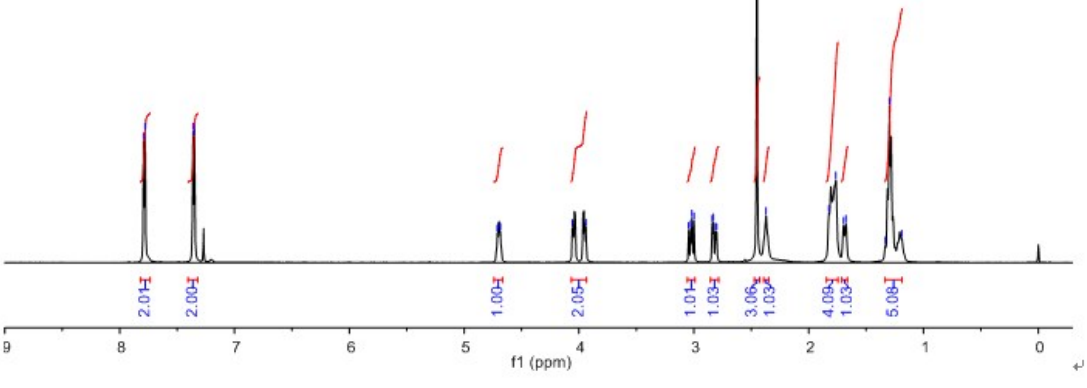


YSC-H

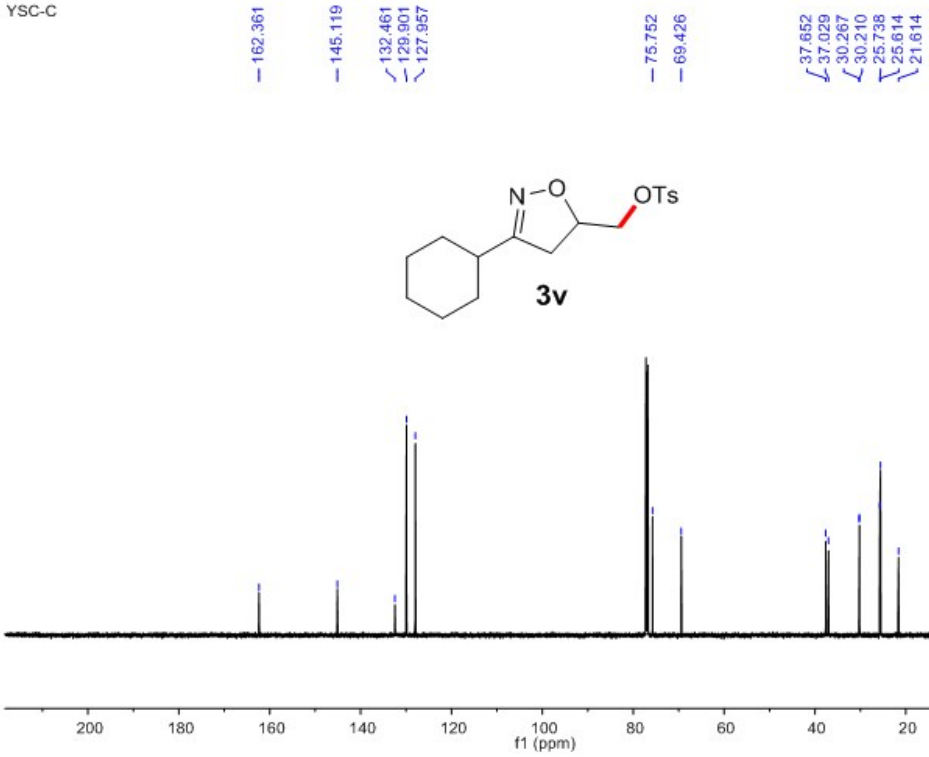
7.792
7.779
7.362
7.349
4.711
4.685
4.058
3.940
3.045
3.027
3.016
2.999
2.840
2.830
2.811
2.802
2.451
2.372
1.824
1.763
1.697
1.676
1.332
1.297
1.189



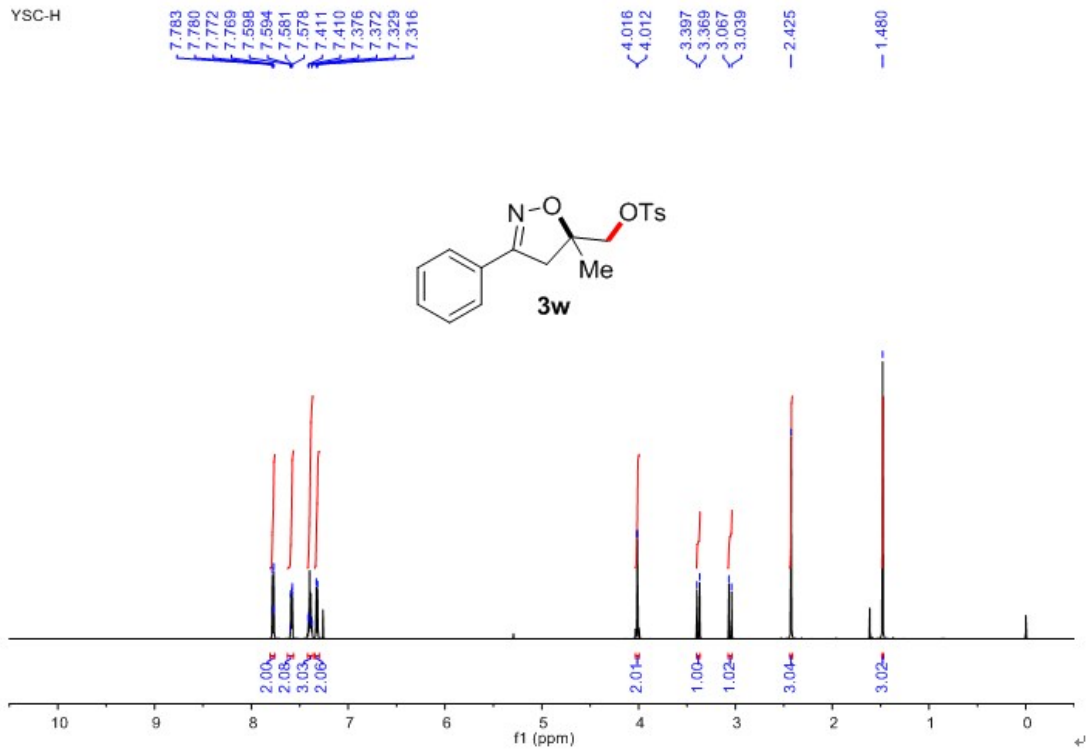
3v



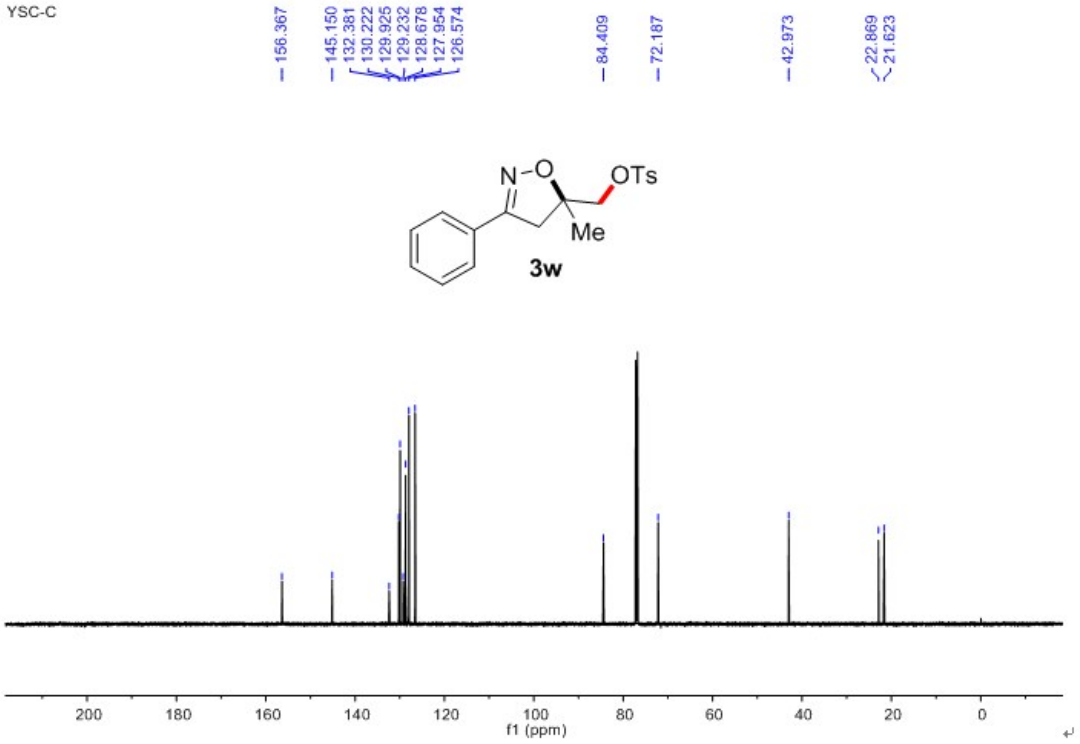
YSC-C


162.361
145.119
132.461
129.901
127.957
75.752
69.426
37.652
37.029
30.267
30.210
25.738
25.614
21.614
N
O
OTs
3v
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

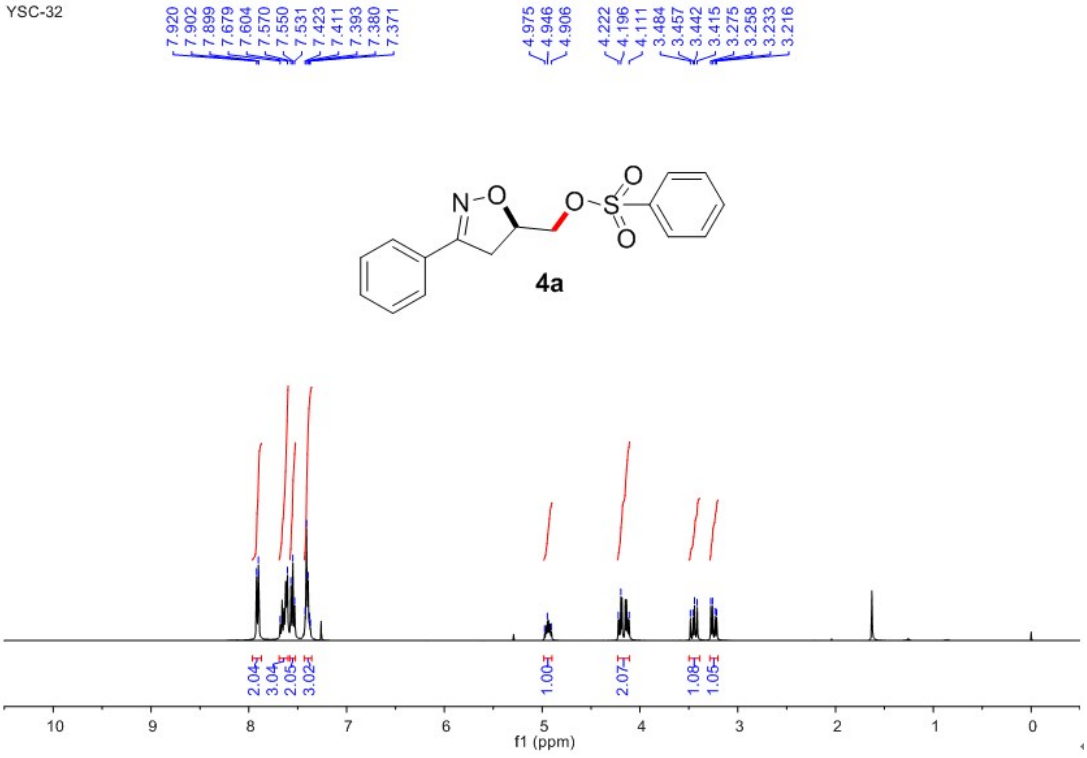
YSC-H

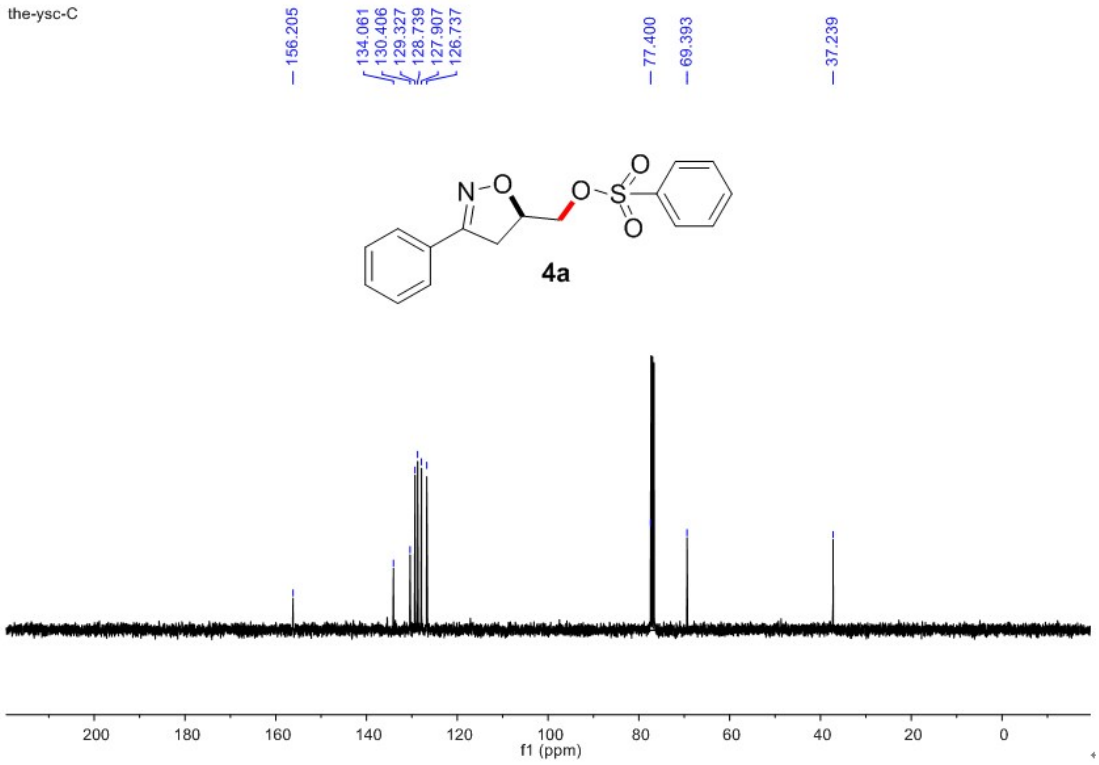

7.783
7.780
7.772
7.769
7.598
7.594
7.581
7.578
7.411
7.410
7.376
7.372
7.329
7.316
4.016
4.012
3.397
3.369
3.067
3.039
2.425
1.480
N
O
OTs
Me
3w
2.00
2.08
3.03
2.06
2.01
1.00
1.02
3.04
3.02
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

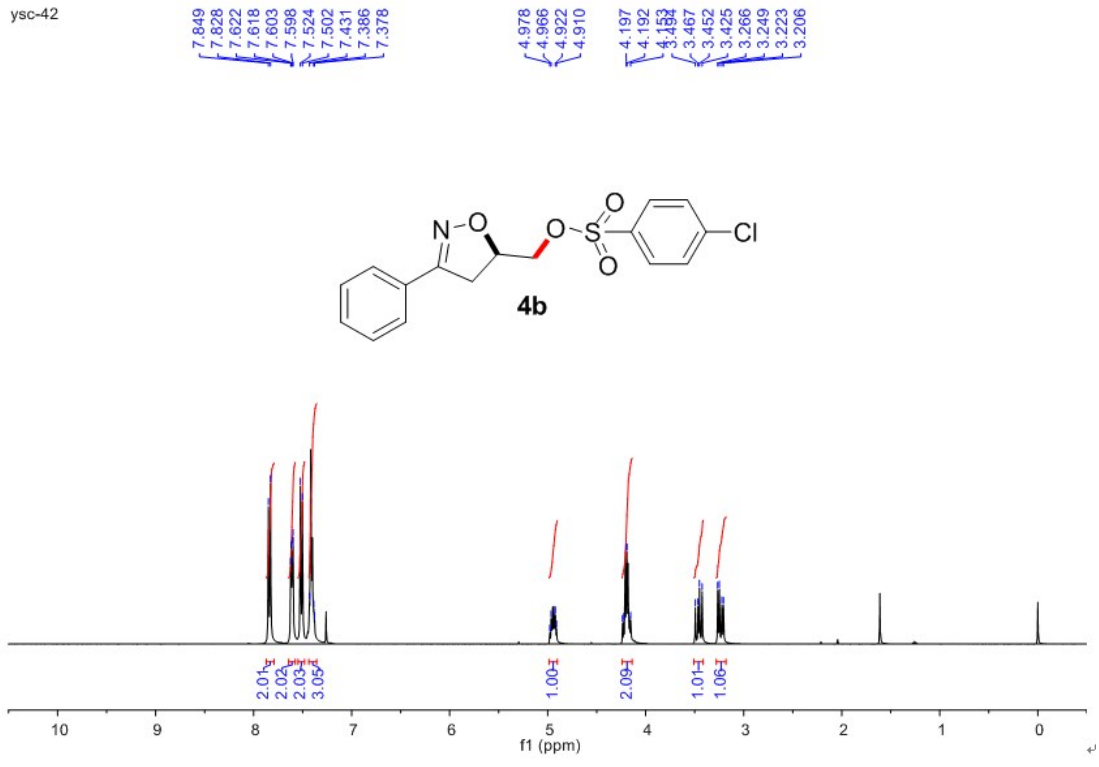
YSC-C

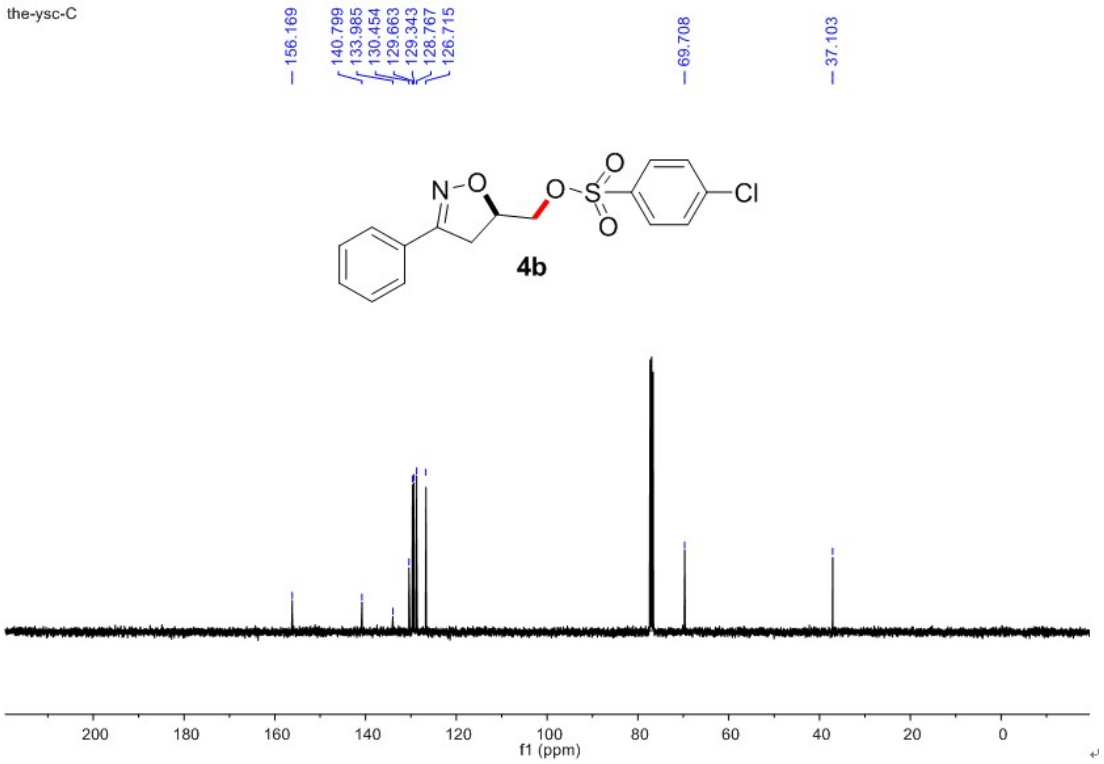

156.367
145.150
132.381
130.222
129.925
129.232
128.678
127.954
126.574
84.409
72.187
42.973
22.869
21.623
N
O
OTs
Me
3w
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

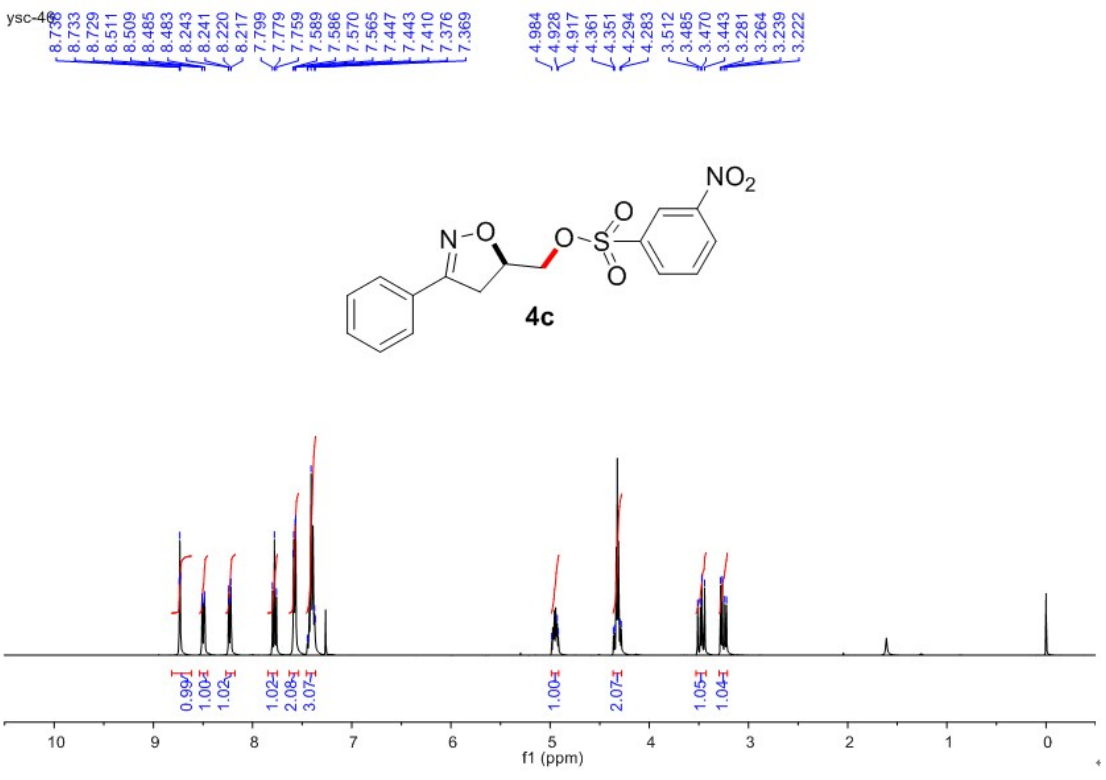
YSC-32

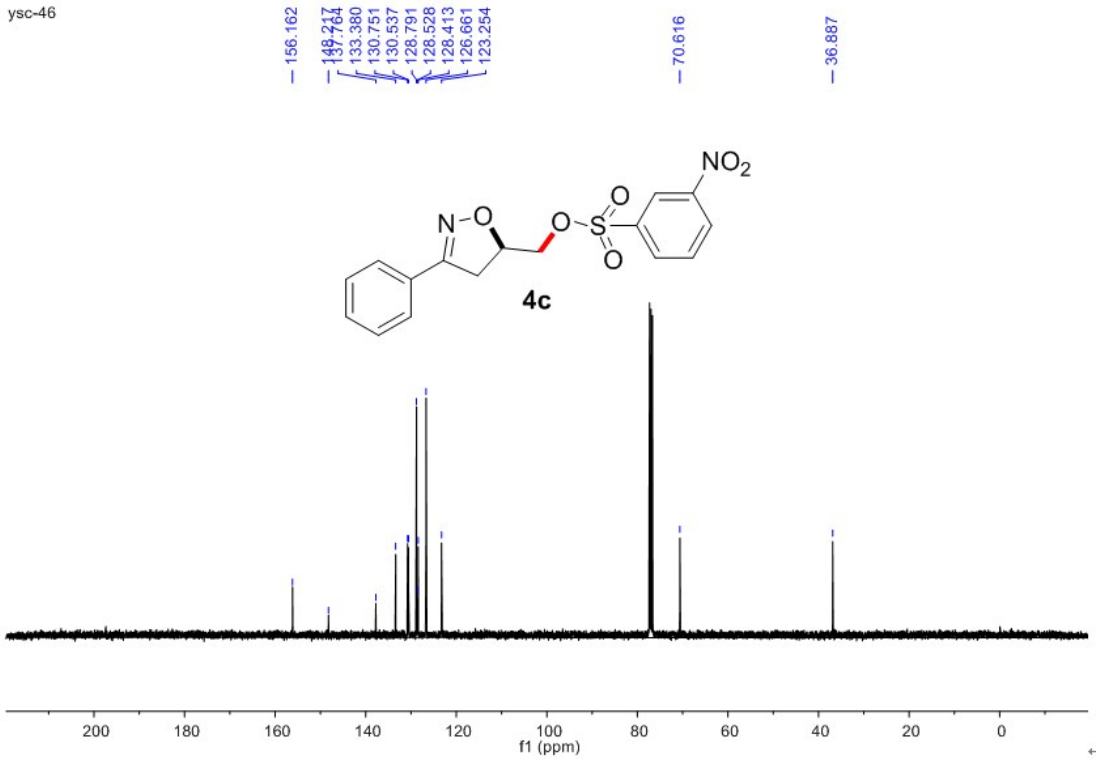

7.920
7.902
7.899
7.679
7.604
7.570
7.550
7.531
7.423
7.411
7.393
7.380
7.371
4.975
4.946
4.906
4.222
4.196
4.111
3.484
3.457
3.442
3.415
3.275
3.258
3.233
3.216
N
O
O
S
O
O
4a
2.04
3.04
2.05
3.02
1.00
2.07
1.08
1.05
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

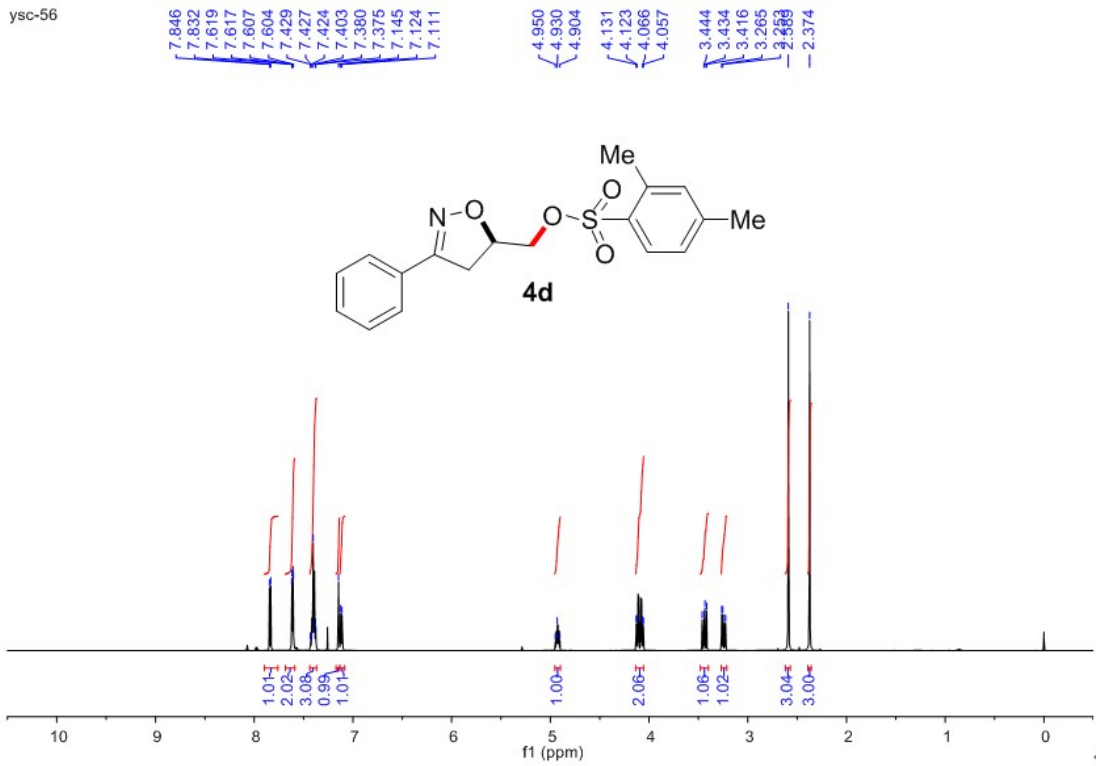

the-ysc-C
156.205
134.061
130.406
129.327
128.739
127.907
126.737
77.400
69.393
37.239
4a
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


ysc-42
7.849
7.828
7.622
7.618
7.603
7.598
7.524
7.502
7.431
7.386
7.378
4.978
4.966
4.922
4.910
4.197
4.192
3.467
3.452
3.425
3.266
3.249
3.223
3.206
Cl
4b
2.01
2.02
2.03
3.05
1.00
2.09
1.01
1.06
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

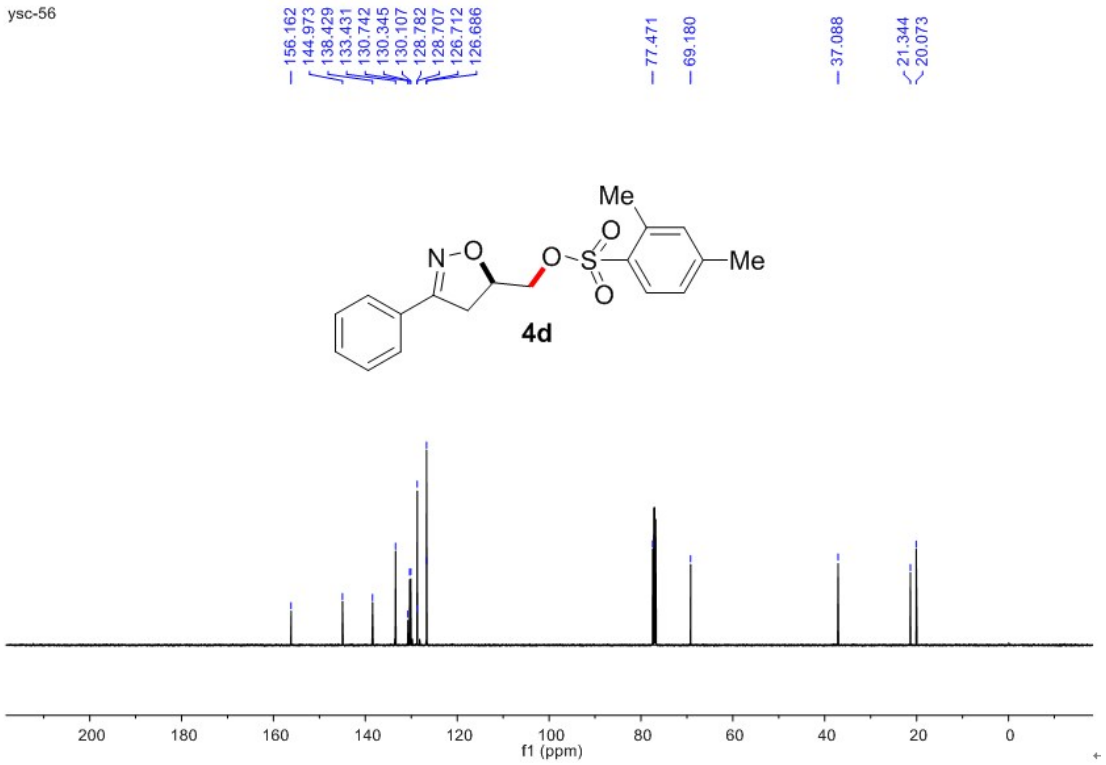

the-ysc-C
156.169
140.799
133.985
130.454
129.663
129.343
128.767
126.715
69.708
37.103
Cl
4b
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


ysc-4c
8.733
8.729
8.511
8.509
8.485
8.483
8.243
8.241
8.220
8.217
7.799
7.779
7.759
7.589
7.586
7.570
7.565
7.447
7.443
7.410
7.376
7.369
4.984
4.928
4.917
4.361
4.351
4.294
4.283
3.512
3.485
3.470
3.443
3.281
3.264
3.239
3.222
NO2
4c
0.99
1.00
1.02
1.02
2.08
3.07
1.00
2.07
1.05
1.04
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

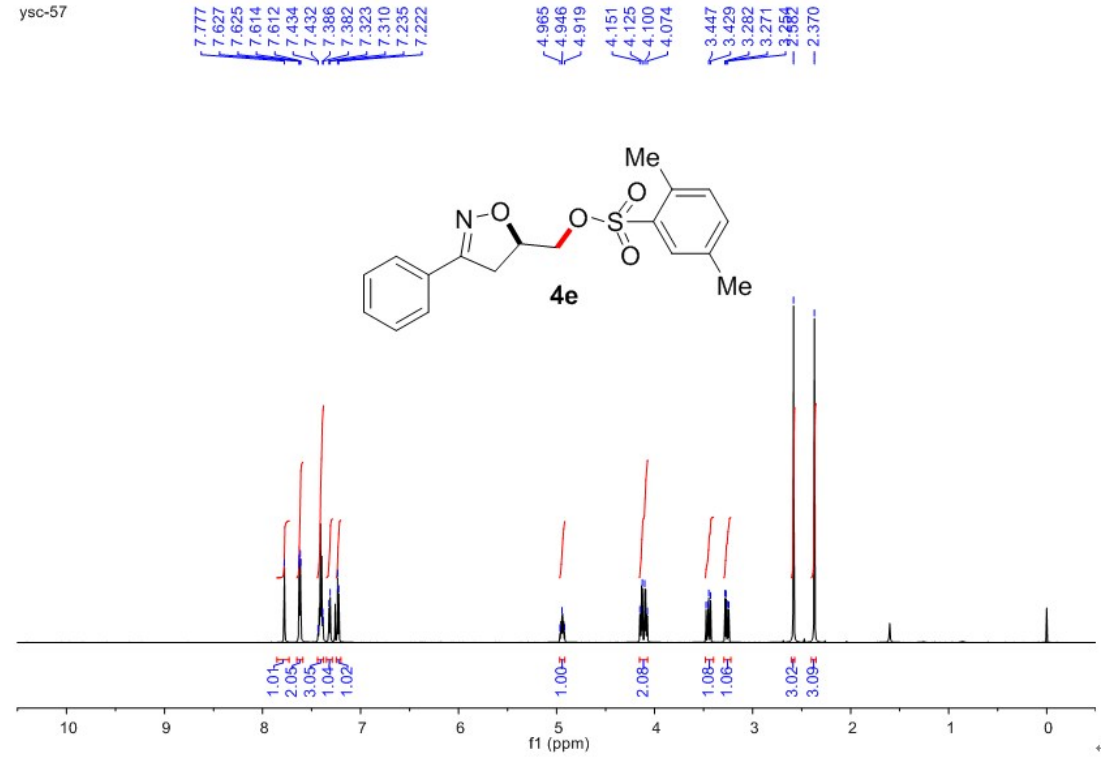

ysc-46
156.162
148.217
137.764
133.380
130.751
130.537
128.791
128.528
128.413
126.661
123.254
70.616
36.887
NO2
O
N
O
O
S
O
4c
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


ysc-56
7.846
7.832
7.619
7.617
7.607
7.604
7.429
7.427
7.424
7.403
7.380
7.375
7.145
7.124
7.111
4.950
4.930
4.904
4.131
4.123
4.066
4.057
3.444
3.434
3.416
3.265
2.374
Me
O
N
O
O
S
O
Me
4d
1.01
2.02
3.08
0.99
1.01
1.00
2.06
1.06
1.02
3.04
3.00
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

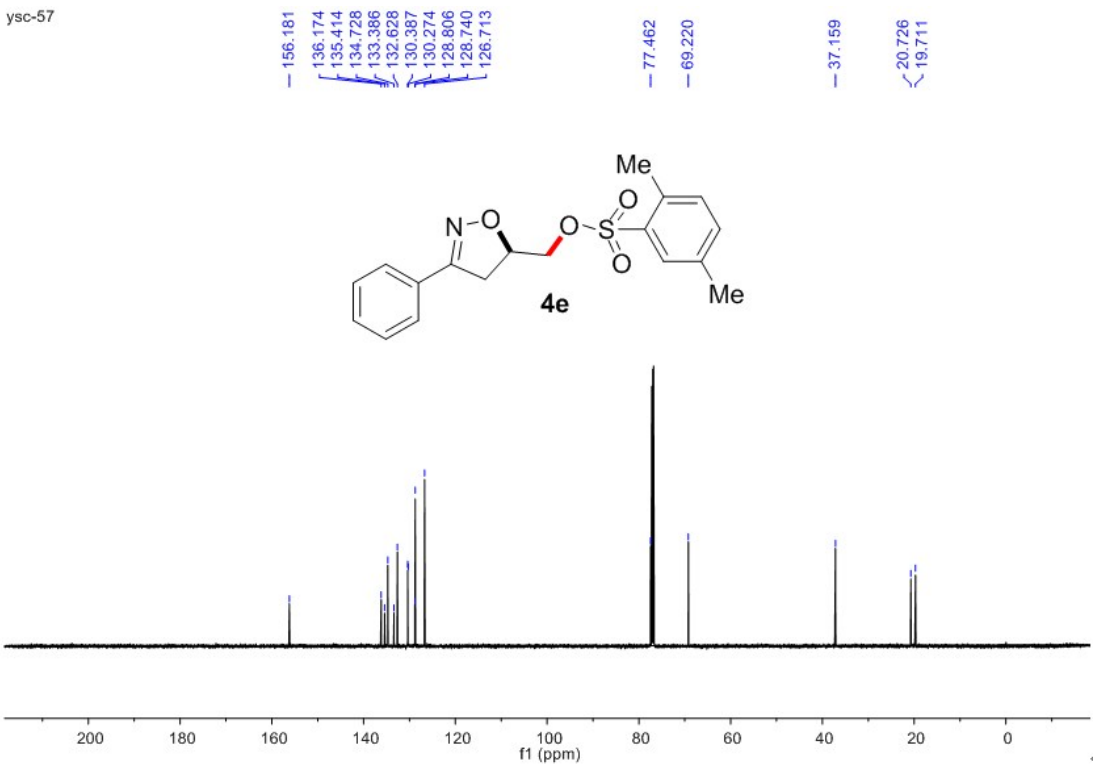
ysc-56


156.162
144.973
138.429
133.431
130.742
130.345
130.107
128.782
128.707
126.712
126.686
77.471
69.180
37.088
21.344
20.073
Me
O
N
O
O
S
O
Me
4d
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

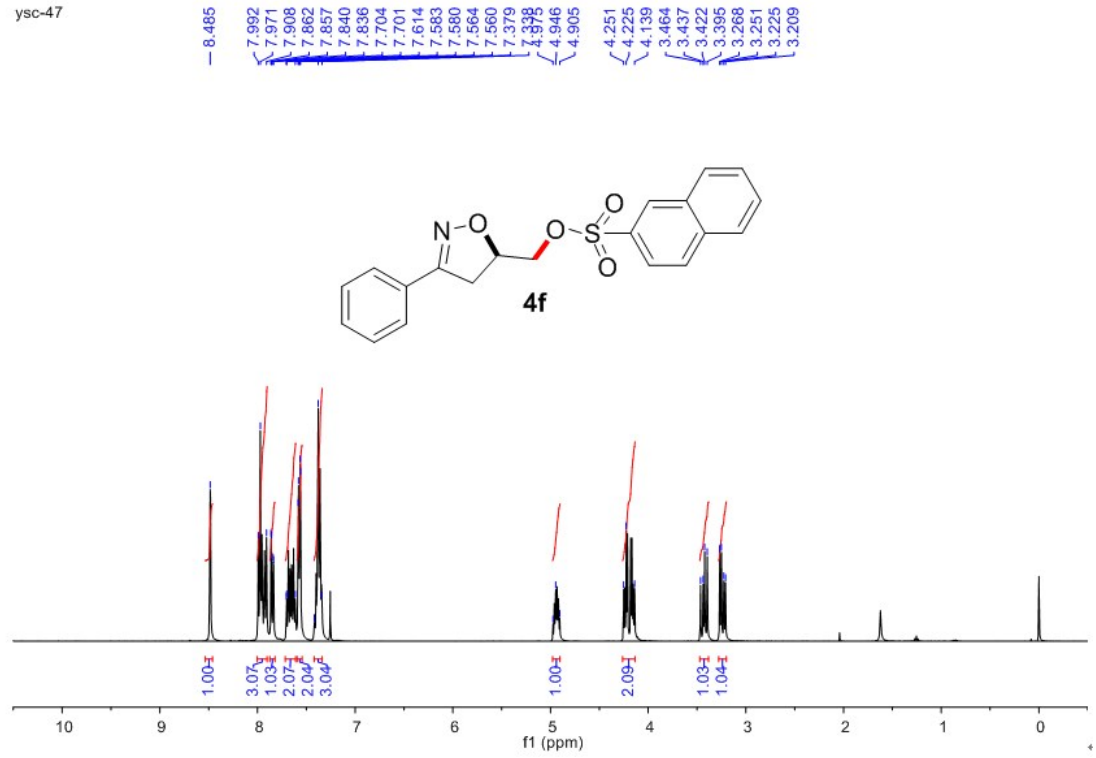
ysc-57

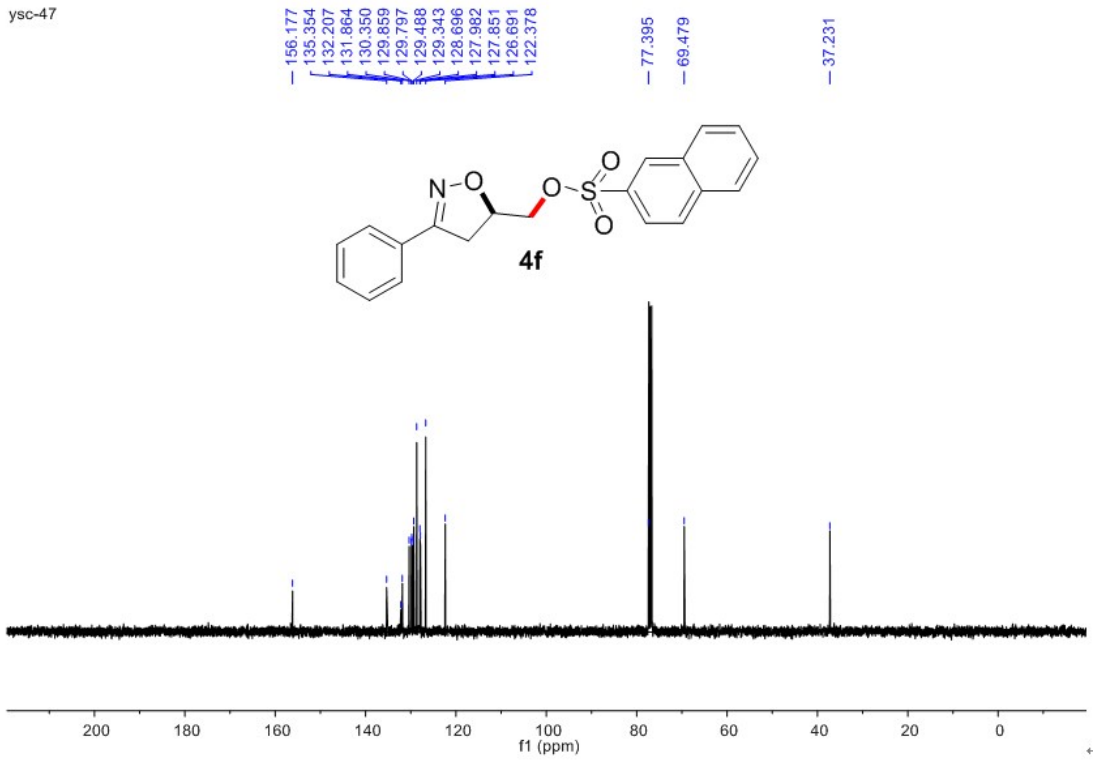

7.777
7.627
7.625
7.614
7.612
7.434
7.432
7.386
7.382
7.323
7.310
7.235
7.222
4.965
4.946
4.919
4.151
4.125
4.100
4.074
3.447
3.429
3.282
3.271
2.370
Me
O
N
O
O
S
O
Me
4e
1.01
2.05
3.05
1.04
1.02
1.00
2.08
1.08
1.06
3.02
3.09
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

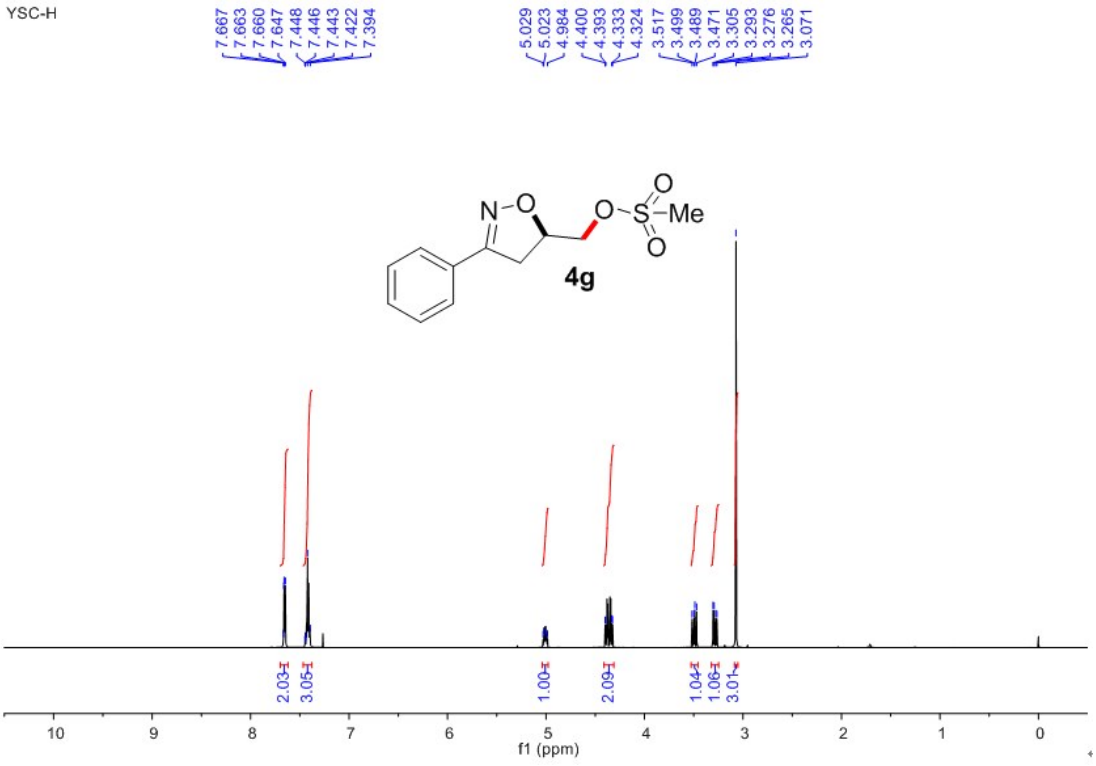
ysc-57


156.181
136.174
135.414
134.728
133.386
132.628
130.387
130.274
128.806
128.740
126.713
77.462
69.220
37.159
20.726
19.711
Me
Me
4e
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

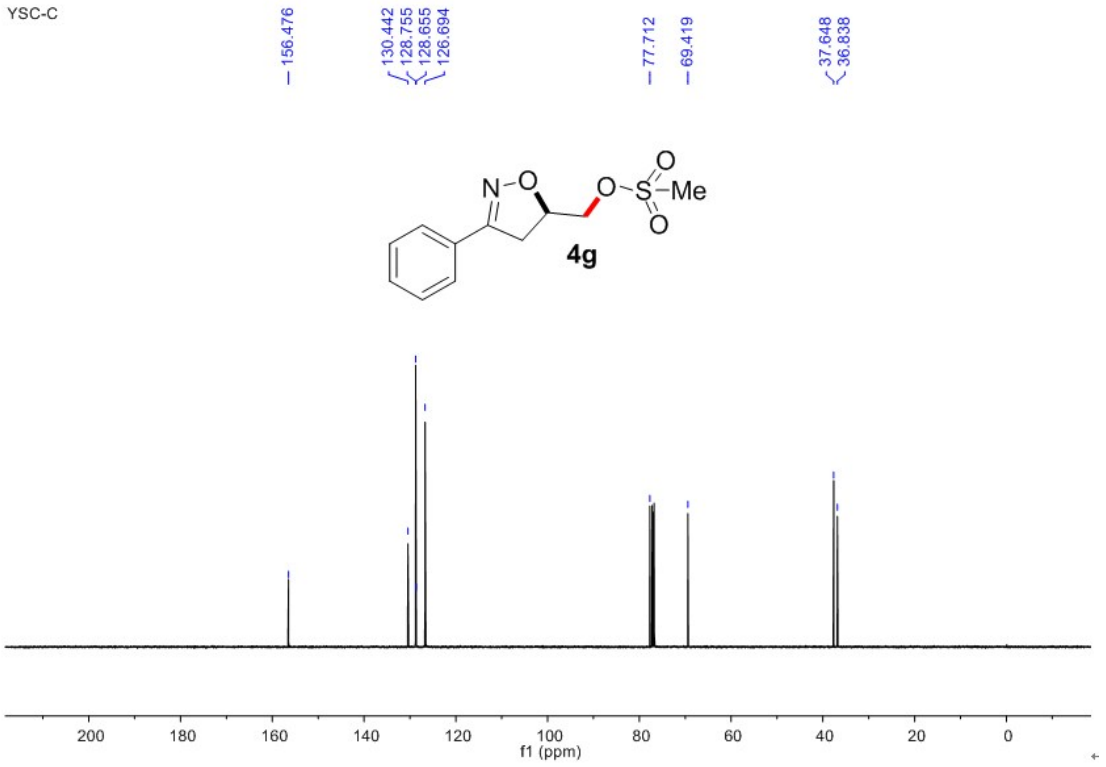
ysc-47


8.485
7.992
7.971
7.908
7.862
7.857
7.840
7.836
7.704
7.701
7.614
7.583
7.580
7.564
7.560
7.379
4.946
4.905
4.251
4.225
4.139
3.464
3.437
3.422
3.395
3.268
3.251
3.225
3.209
4f
1.00
3.07
1.03
2.07
2.04
3.04
1.00
2.09
1.03
1.04
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

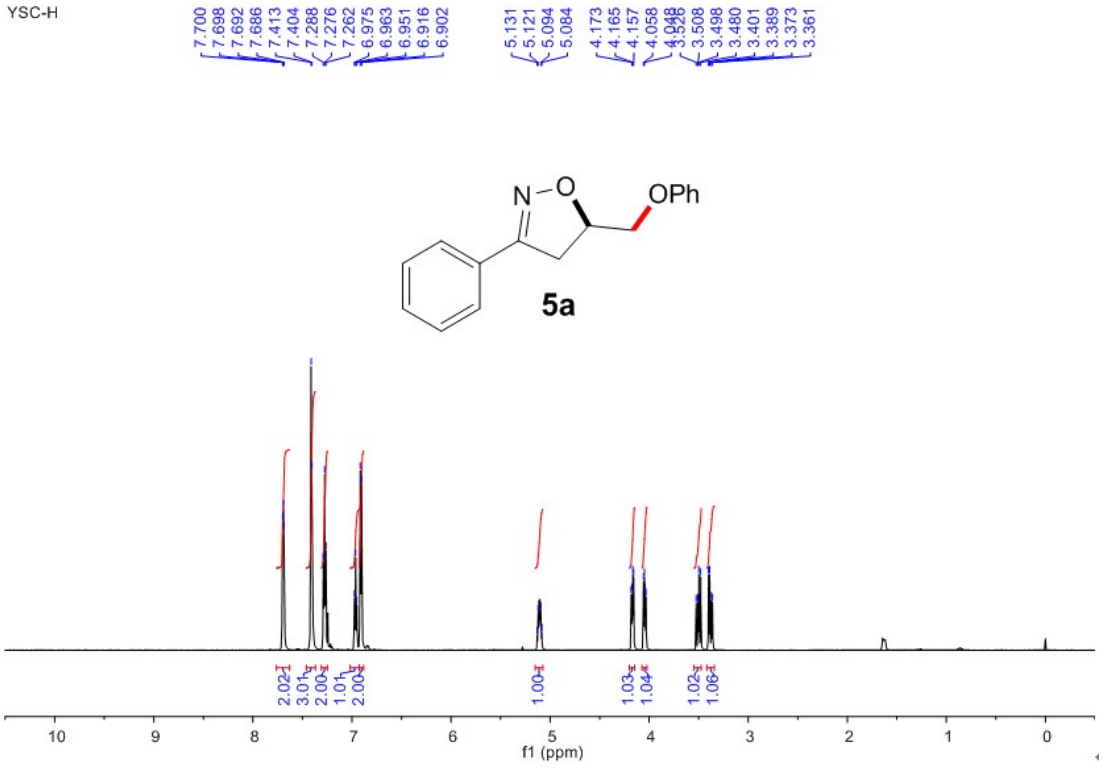

ysc-47
156.177
135.354
132.207
131.864
130.350
129.859
129.797
129.488
129.343
128.696
127.982
127.851
126.691
122.378
77.395
69.479
37.231
4f
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


YSC-H
7.667
7.663
7.660
7.647
7.448
7.446
7.443
7.422
7.394
5.029
5.023
4.984
4.400
4.393
4.333
4.324
3.517
3.499
3.489
3.471
3.305
3.293
3.276
3.265
3.071
4g
2.03
3.05
1.00
2.09
1.04
1.06
3.01
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

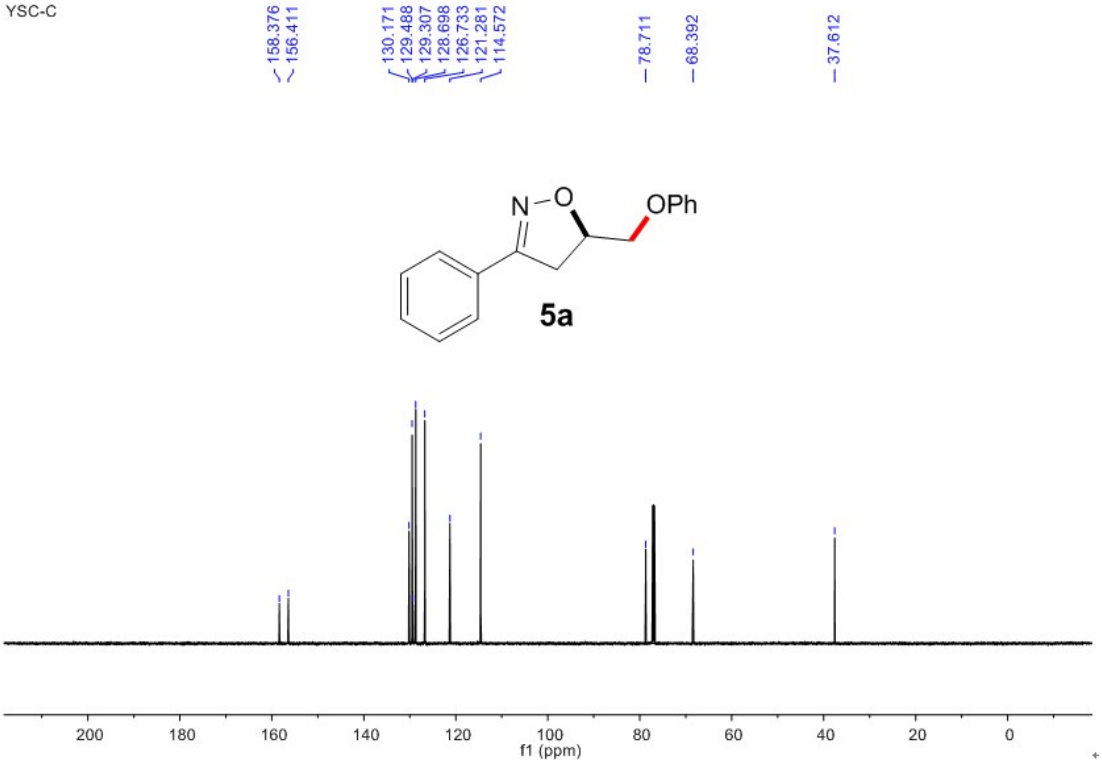
YSC-C


156.476
130.442
128.755
128.655
126.694
77.712
69.419
37.648
36.838
N
O
O
S
Me
O
O
4g
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

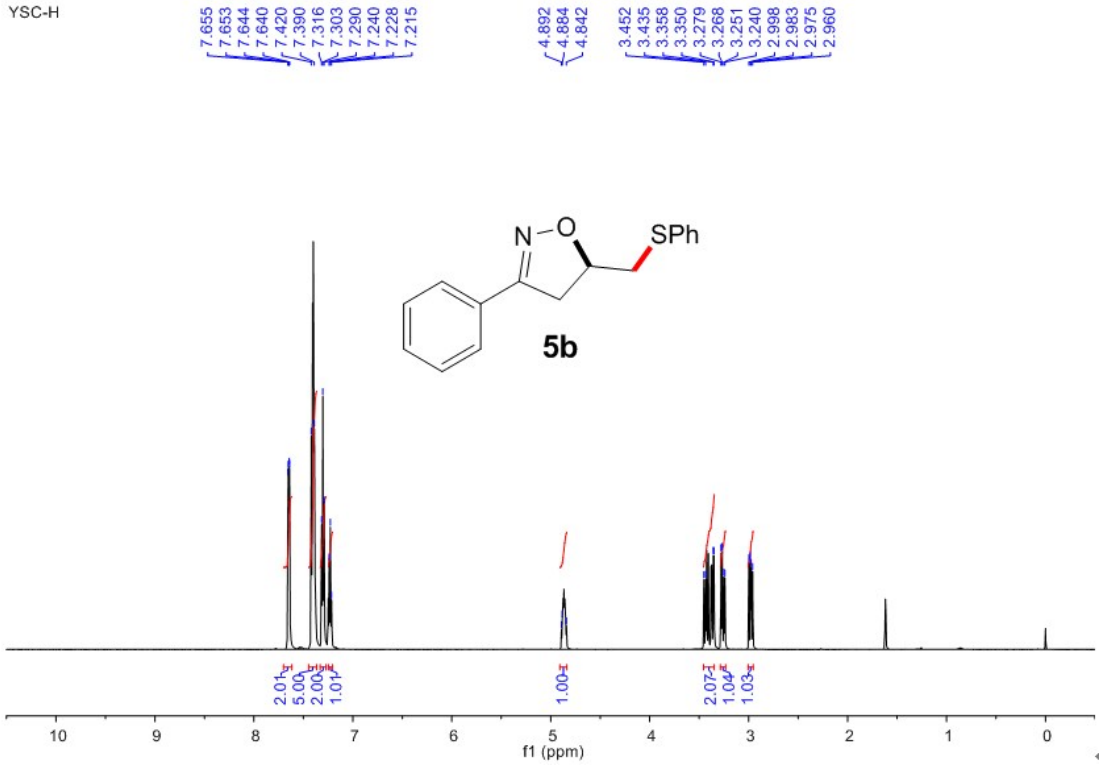
YSC-H


7.700
7.698
7.692
7.686
7.413
7.404
7.288
7.276
7.262
6.975
6.963
6.951
6.916
6.902
5.131
5.121
5.094
5.084
4.173
4.165
4.157
4.058
3.526
3.508
3.498
3.480
3.401
3.389
3.373
3.361
N
O
OPh
5a
2.02
3.01
2.00
1.01
2.00
1.00
1.03
1.04
1.02
1.06
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

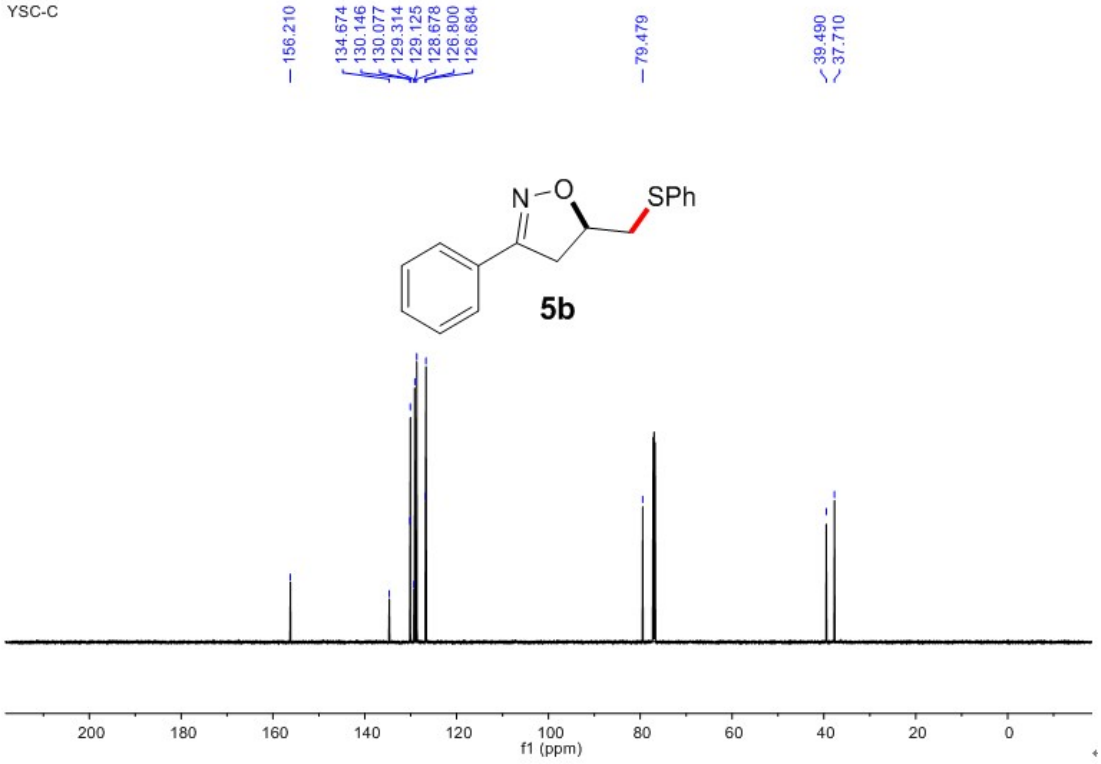
YSC-C


158.376
156.411
130.171
129.488
129.307
128.698
126.733
121.281
114.572
78.711
68.392
37.612
N
O
OPh
5a
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

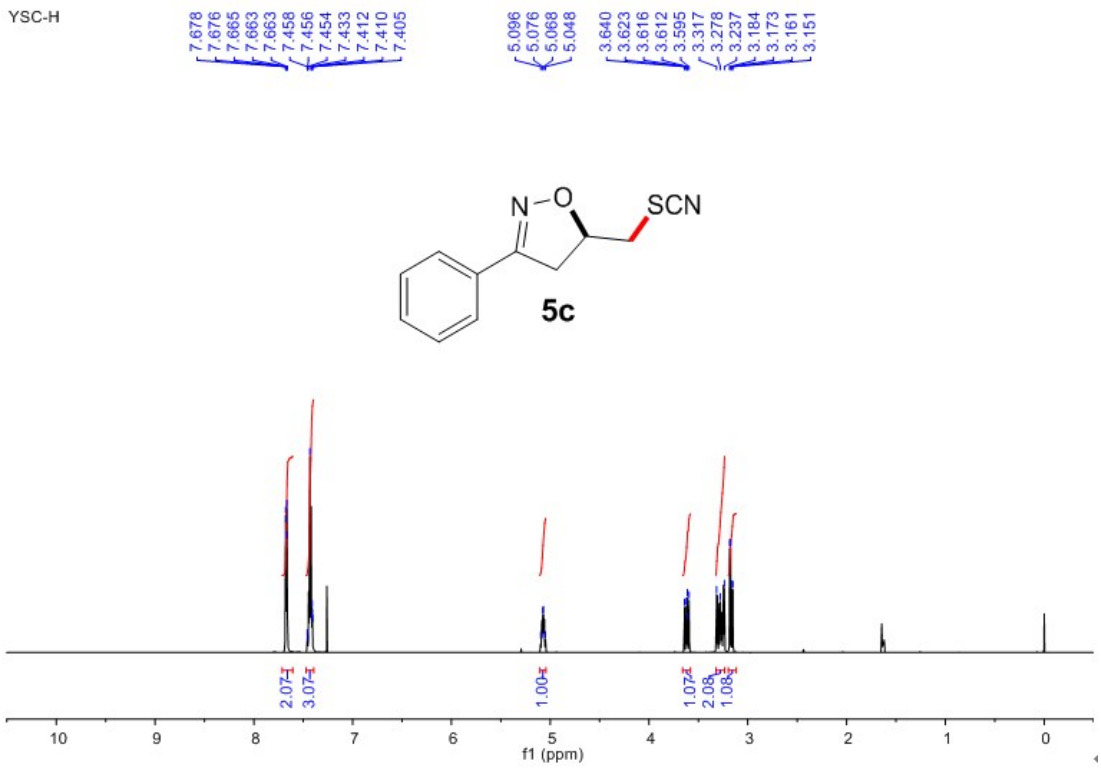
YSC-H


7.655
7.653
7.644
7.640
7.420
7.390
7.316
7.303
7.290
7.240
7.228
7.215
4.892
4.884
4.842
3.452
3.435
3.358
3.350
3.279
3.268
3.251
3.240
2.998
2.983
2.975
2.960
N
O
SPh
5b
2.01
5.00
2.00
1.01
1.00
2.07
1.04
1.03
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

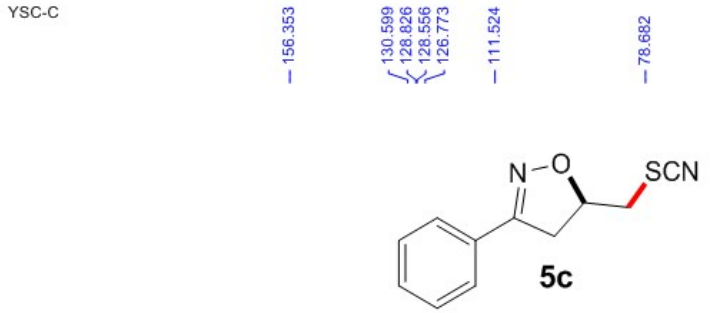
YSC-C


156.210
134.674
130.146
130.077
129.314
129.125
128.678
126.800
126.684
79.479
39.490
37.710
N
O
SPh
5b
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

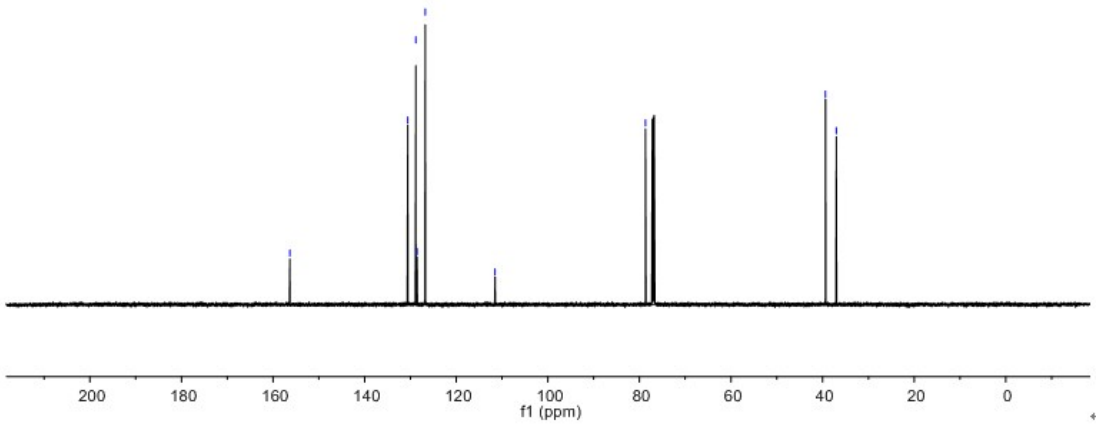
YSC-H


7.678
7.676
7.665
7.663
7.663
7.458
7.456
7.454
7.433
7.412
7.410
7.405
5.096
5.076
5.068
5.048
3.640
3.623
3.616
3.612
3.595
3.317
3.278
3.237
3.184
3.173
3.161
3.151
N
O
SCN
5c
2.07
3.07
1.00
1.07
2.08
1.08
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

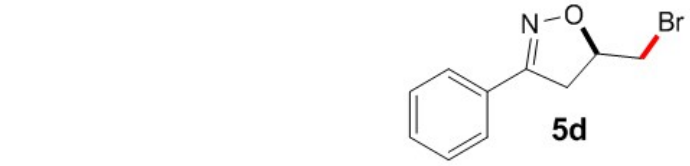
YSC-C



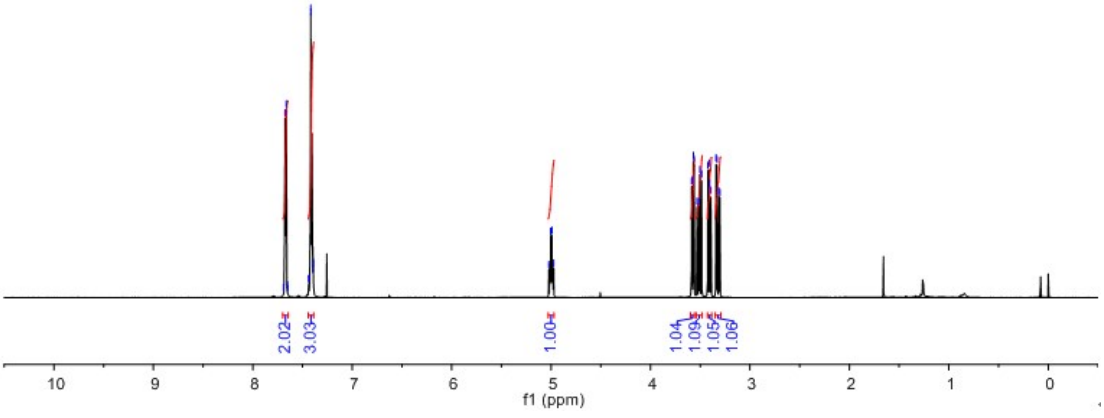
5c



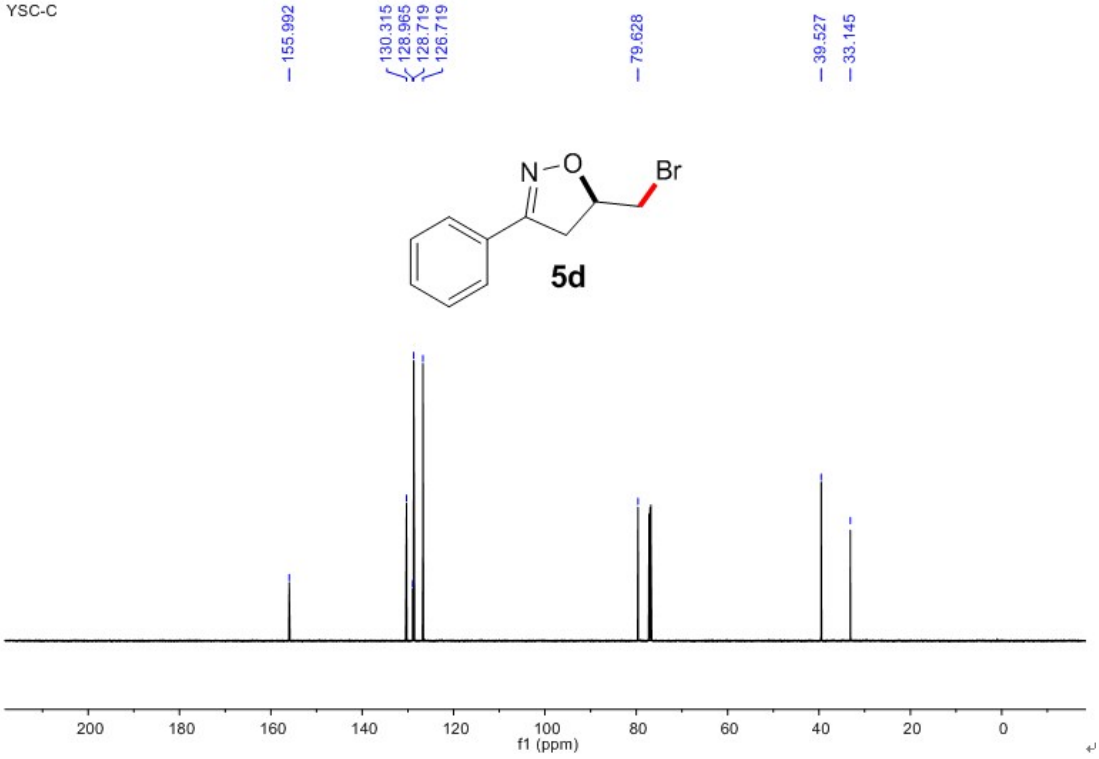
YSC-H



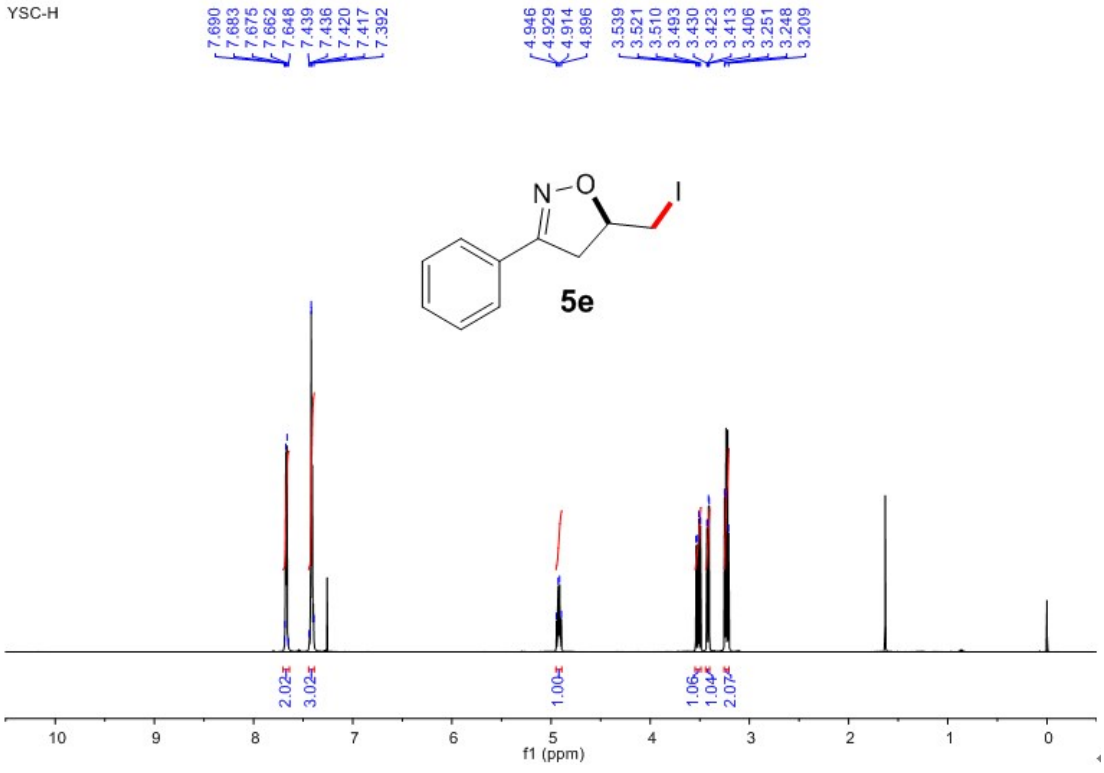
5d



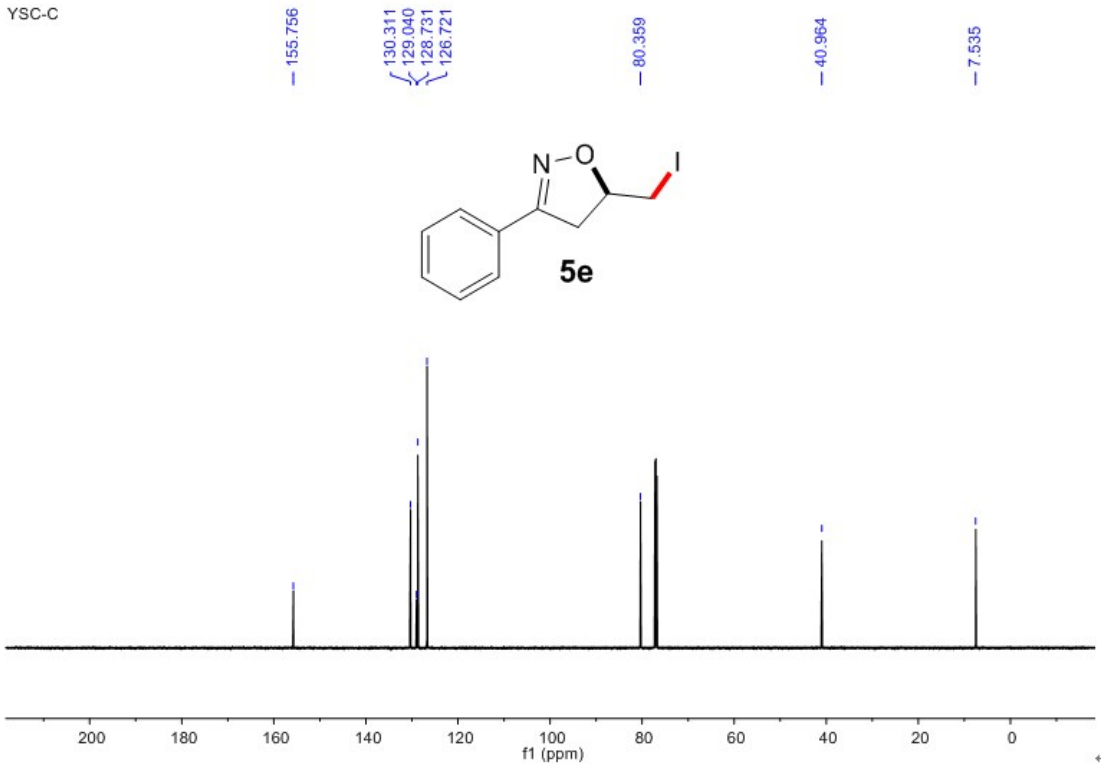
YSC-C


155.992
130.315
128.965
128.719
126.719
79.628
39.527
33.145
N
O
Br
5d
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

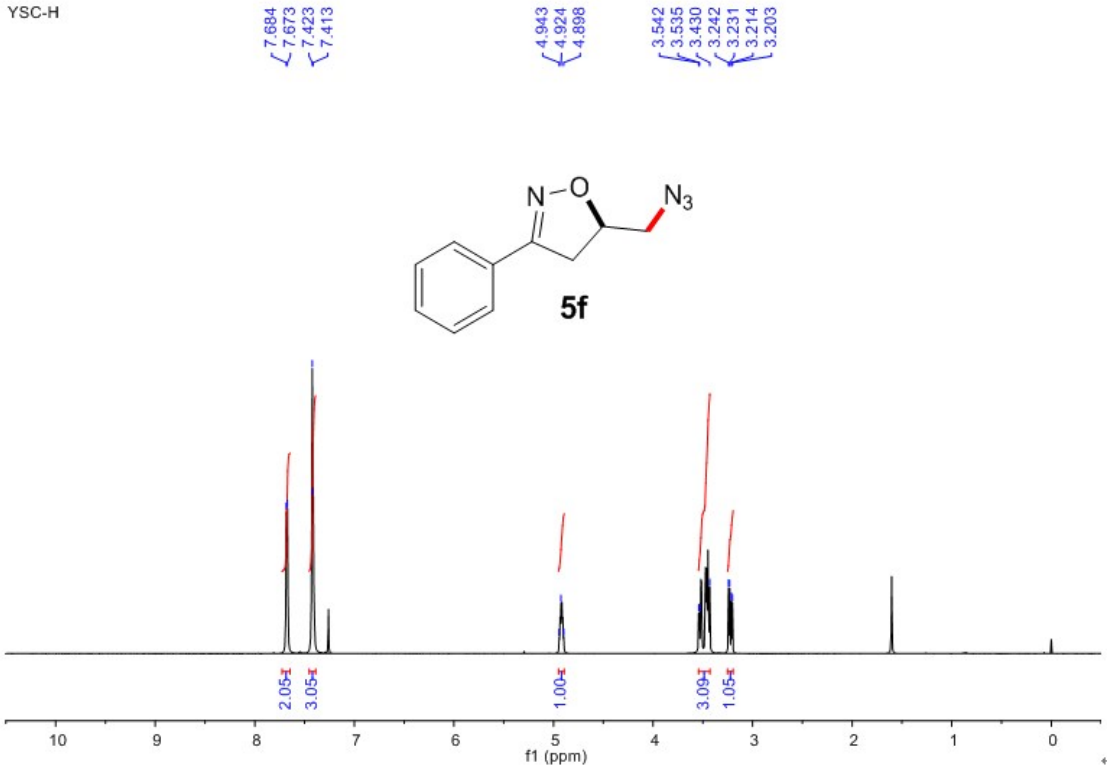
YSC-H


7.690
7.683
7.675
7.662
7.648
7.439
7.436
7.420
7.417
7.392
4.946
4.929
4.914
4.896
3.539
3.521
3.510
3.493
3.430
3.423
3.413
3.406
3.251
3.248
3.209
N
O
I
5e
2.02
3.02
1.00
1.06
1.04
2.07
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

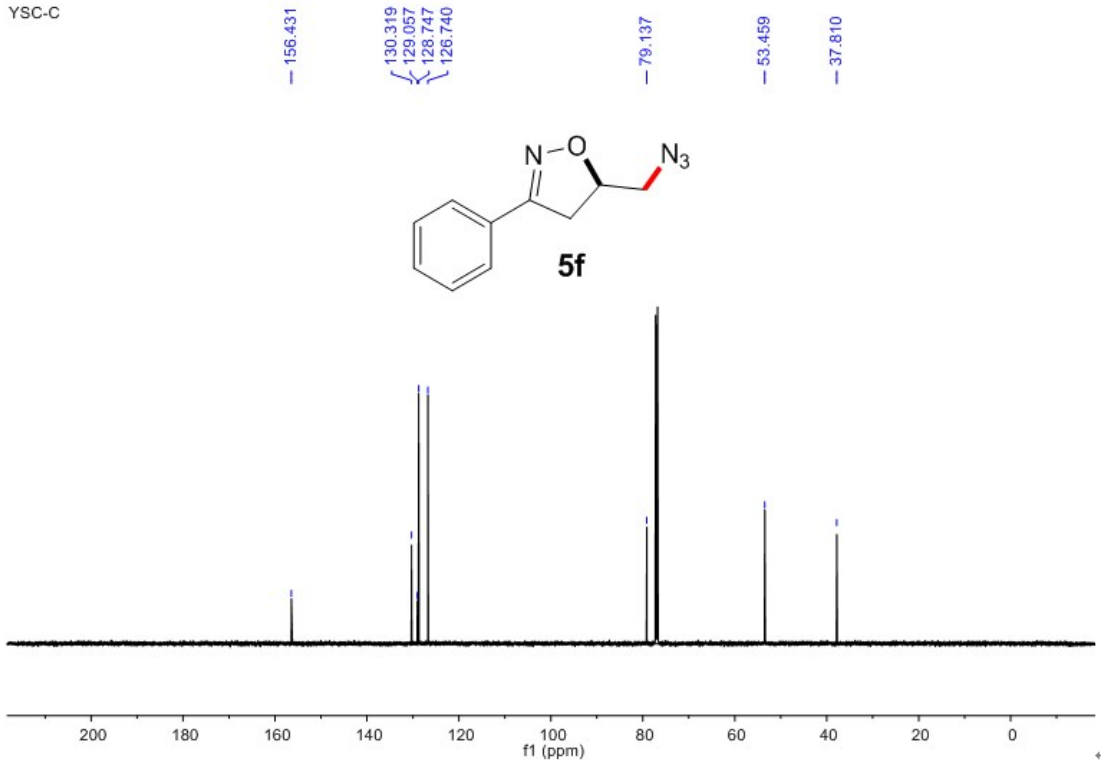
YSC-C


155.756
130.311
129.040
128.731
126.721
80.359
40.964
7.535
N
O
I
5e
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

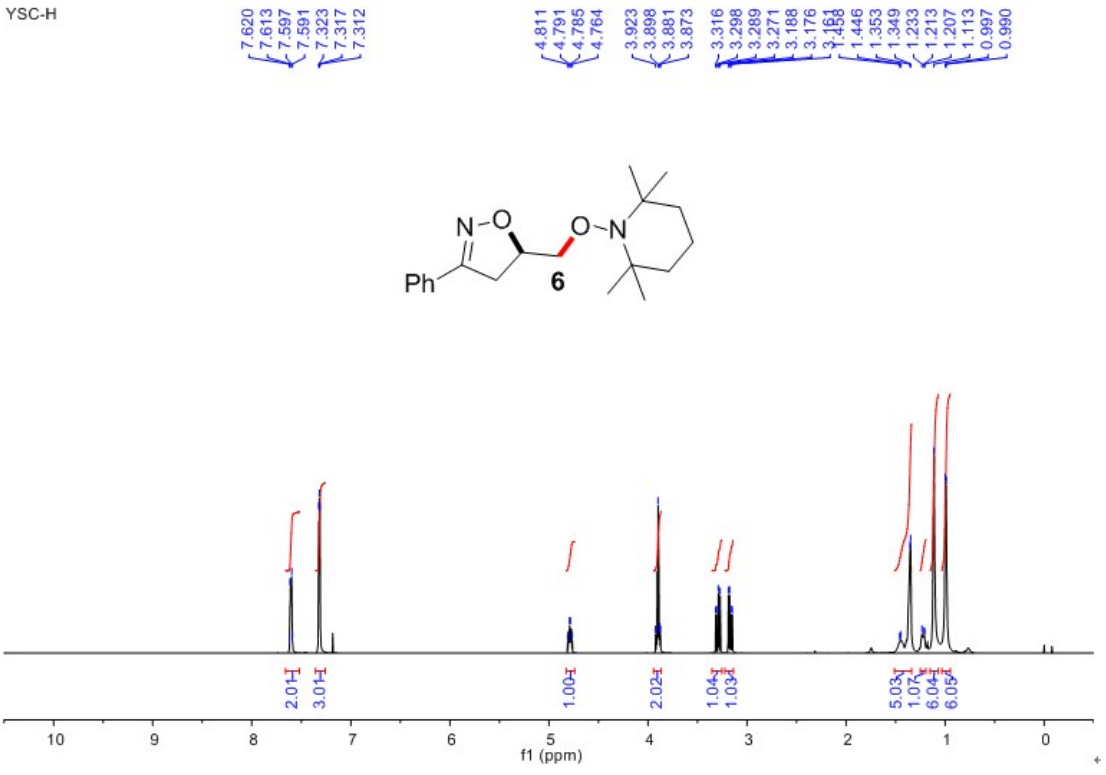
YSC-H


7.684
7.673
7.423
7.413
4.943
4.924
4.898
3.542
3.535
3.430
3.242
3.231
3.214
3.203
N
O
N3
5f
2.05
3.05
1.00
3.09
1.05
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

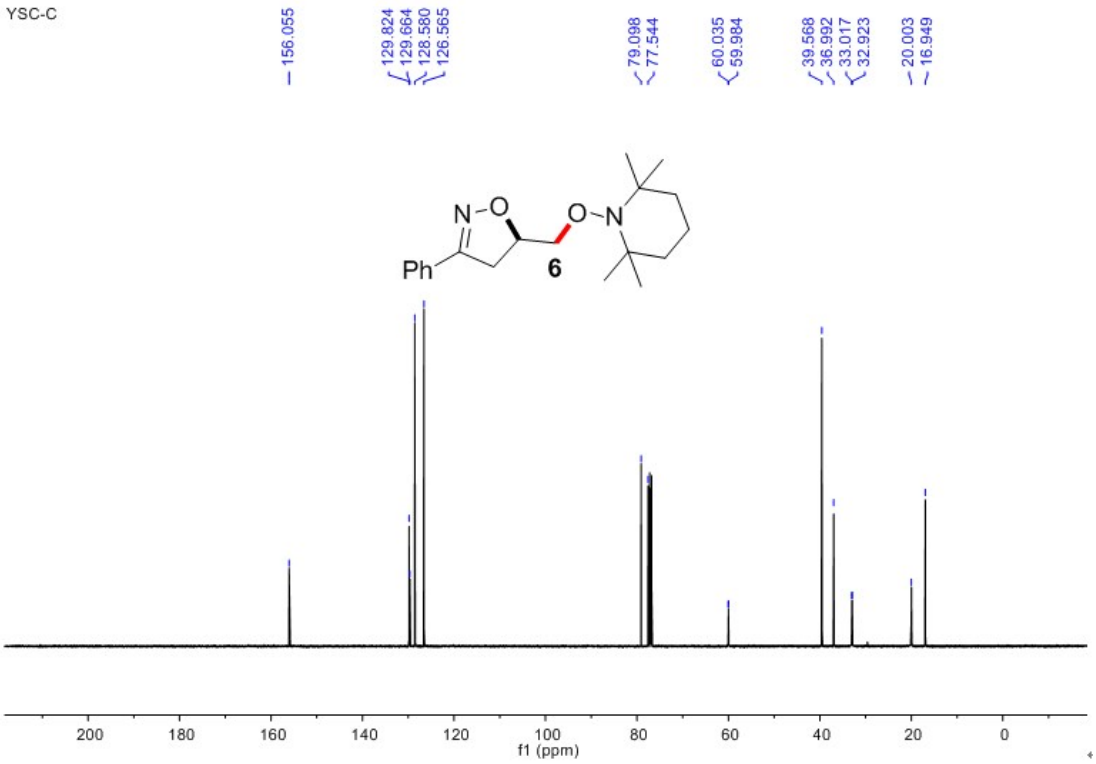
YSC-C


156.431
130.319
129.057
128.747
126.740
79.137
53.459
37.810
N
O
N3
5f
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

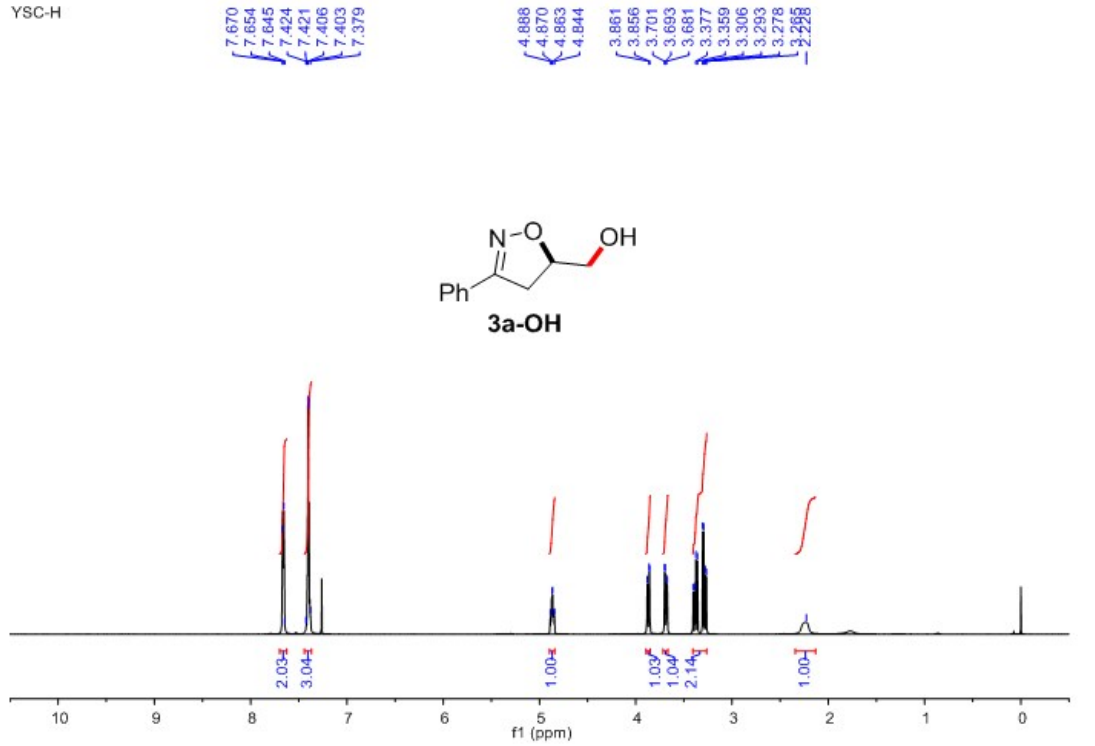
YSC-H

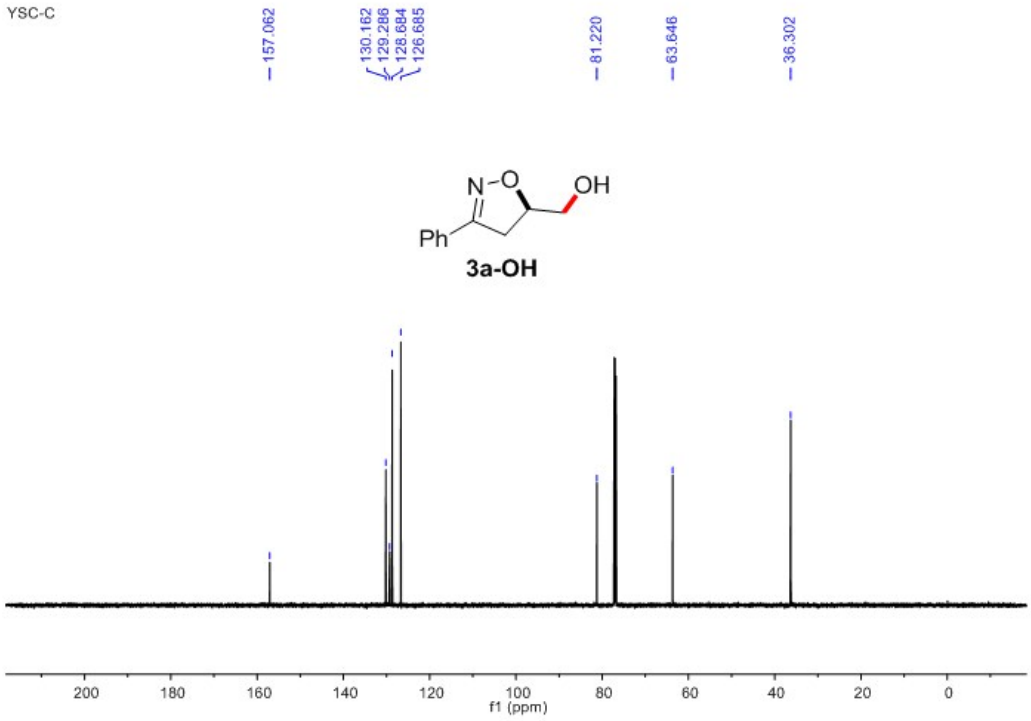

7.620
7.613
7.597
7.591
7.323
7.317
7.312
4.811
4.791
4.785
4.764
3.923
3.898
3.881
3.873
3.316
3.298
3.289
3.271
3.188
3.176
1.446
1.353
1.349
1.233
1.213
1.207
1.113
0.997
0.990
N
O
O
N
Ph
6
2.01
3.01
1.00
2.02
1.04
1.03
5.03
1.07
6.04
6.05
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

YSC-C


156.055
129.824
129.664
128.580
126.565
79.098
77.544
60.035
59.984
39.568
36.992
33.017
32.923
20.003
16.949
N
O
O
N
Ph
6
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

YSC-H


7.670
7.654
7.645
7.424
7.421
7.406
7.403
7.379
4.888
4.870
4.863
4.844
3.861
3.856
3.701
3.693
3.681
3.377
3.359
3.306
3.293
3.278
2.228
N
O
OH
Ph
3a-OH
2.03
3.04
1.00
1.03
1.04
2.14
1.00
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


YSC-C
157.062
130.162
129.286
128.684
126.685
81.220
63.646
36.302
N
O
OH
Ph
3a-OH
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

5. The crystallographic data for compounds 3a (CCDC number: 1921843)

(1) Check CIF report

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) t

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: t

Bond precision: C-C = 0.0039 A Wavelength=0.71073

Cell: a=5.7496(11) b=11.342(2) c=12.287(3)
alpha=92.62(3) beta=95.69(3) gamma=94.25(3)
Temperature: 295 K

	Calculated	Reported
Volume	794.0(3)	794.0(3)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C17 H17 N O4 S	C17 H17 N O4 S
Sum formula	C17 H17 N O4 S	C17 H17 N O4 S
Mr	331.38	331.38
Dx,g cm-3	1.386	1.386
Z	2	2
Mu (mm-1)	0.224	0.224
F000	348.0	348.0
F000'	348.41	
h,k,lmax	6,13,14	6,13,14
Nref	2823	2808
Tmin,Tmax	0.976,0.989	0.955,0.994
Tmin'	0.971	

Correction method= # Reported T Limits: Tmin=0.955 Tmax=0.994
AbsCorr = MULTI-SCAN

Data completeness= 0.995 Theta(max)= 25.050

R(reflections)= 0.0466(2154) wR2(reflections)= 0.1222(2808)

S = 1.034 Npar= 209

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

(2) The crystal structure of 3a