Supporting Information

for

Efficient gold(I)/silver(I)-cocatalyzed cascade intermolecular N-Michael addition/intramolecular hydroalkylation of unactivated alkenes with α-ketones

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Experimental section and spectra of compounds

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Experimental section

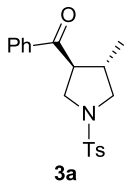
General methods. Reagents were obtained commercially and used without further purification unless indicated otherwise. All anhydrous solvents used in the reactions were dried and freshly distilled. All manipulations with air-sensitive reagents were carried out under a dry argon atmosphere. The catalysts $Au(PPh_3)Cl$ [1], $(Cy)_2$(2',4',6'-triisopropyl-*o*-biphenyl)PAuCl [1,2], (t-Bu)$_2$(o-diphenyl)PAuCl [1,2] and IPrAuCl [3] were prepared following literature procedures. 2-Methylene-3,4-dihydronaphthalen-1(*2H*)-one was prepared according to the literature procedure [4]. α,β-Unsaturated ketones were prepared following the literature procedure [5]. Substituted allylic amines were prepared following the literature procedure [6]. NMR spectra were recorded on Bruker AM300/400 spectrometers at 300/400 MHz for ^{1}H NMR and 75/100 MHz for ^{13}C NMR in $CDCl_3$ with TMS as an internal standard. The chemical shifts are expressed in ppm and coupling constants are given in Hz. Data for ^{1}H NMR are recorded as follows: Chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet), coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm). Mass spectra were obtained on a HP5989A spectrometer (EI), an IonSpec 4.7 Tesla FTMS spectrometer (MALDI), or a Bruker

Daltonics FTMS-7 spectrometer (ESI). IR spectra were recorded as KBr discs, on a Bio-Rad FTS-185 spectrometer; frequencies are given in reciprocal centimeters (cm^{-1}) and only selected absorbance is reported.

General procedure for gold/silver-cocatalyzed one-pot tandem intermolecular N-Michael addition/intramolecular hydroalkylation

A mixture of (*t*-Bu)$_2$(*o*-diphenyl)PAuCl (6.7 mg, 0.0125 mmol), $AgClO_4$ (7.8 mg, 0.0375 mmol) (*Warning! The perchlorate salt is potentially explosive and should be handled with great caution.*), α,β-unsaturated ketone (0.25 mmol) and substituted allylic amine (0.375 mmol, 1.5 equiv) in toluene (0.5 mL) was stirred at 90 °C under Ar atmosphere for 20 h. Upon completion, the solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography (eluent: EtOAc/petroleum ether = 1:12-1:6) to give the desired products.

(*trans*-4-Methyl-1-tosylpyrrolidin-3-yl)(phenyl)methanone (3a)



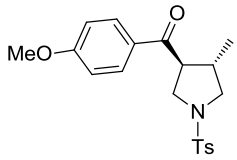
3a

trans/*cis*: 4.1:1. Major diastereomer could be separated on a silica gel column, and the relative configuration of **3a** was determined with reference to 1-(*trans*-4-methyl-1-tosylpyrrolidin-3-yl)ethanone [7].

White solid. ^{1}H NMR(300 MHz, $CDCl_3$): δ 7.86 (d, *J* = 7.3 Hz, 2H), 7.72 (d, *J* = 8.2 Hz, 2H), 7.60 (t, *J* = 7.3 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.34 (d, *J* = 7.3 Hz, 2H), 3.79 (t, *J* = 8.2 Hz, 1H), 3.55 (q, *J* = 8.0 Hz, 1H), 3.47 (dd, *J* = 7.2, 9.0 Hz, 1H), 3.29 (t, *J* = 9.0 Hz, 1H), 3.05 (t, *J* = 7.6 Hz, 1H), 2.56-2.46 (m, 1H), 2.44 (s, 3H), 1.04 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 198.1, 143,6, 136.2, 133.7, 133.4, 129.7,

128.8, 128.3, 127.6, 54.2, 50.8, 36.5, 21.6, 17.4. IR (FILM): ν_{max} 3286, 2956, 2924, 1712, 1679, 1597, 1448, 1341, 1223, 1161, 1093, 1041, 815 cm^{-1}. MS (ESI) *m*/*z*: 366 ($M+Na^+$), 344 ($M+H^+$). HRMS (ESI): calcd. for $C_{19}H_{22}NO_3S^+$ ($M+H^+$): 344.13149, found: 344.13189.

(4-Methoxyphenyl)(*trans*-4-methyl-1-tosylpyrrolidin-3-yl)methanone (3b)

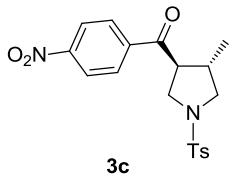


3b

trans/cis: 1.0: 1. One of the diastereomers could be separated on a silica gel column, and the relative configuration of **3b** was determined with reference to **3a**.

White solid. ^1H NMR (300 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.4Hz, 2H), 7.71 (d, *J* = 7.5Hz, 2H), 7.33 (d, *J* = 7.5Hz, 2H), 6.93 (d, *J* = 8.4Hz, 2H), 3.87 (s, 3H), 3.75 (t, *J* = 8.7 Hz, 1H), 3.54-3.43 (m, 2H), 3.27 (t, *J* = 9.0Hz, 1H), 3.04 (t, *J* = 8.7Hz, 1H), 2.54-2.44 (m, 1H), 2.44(s, 3H), 1.01 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 196.4, 163.9, 143.6, 133.4, 130.7, 129.7, 129.2, 127.5, 113.9, 55.5, 54.2, 51.6, 51.0, 36.6, 21.5, 17.3; MS (ESI) *m*/*z*: 396 ($M+Na^+$); HRMS (ESI): calcd. for $C_{20}H_{23}NNaO_4S^+$ ($M+Na^+$): 396.12400, found: 396.12381.

(*cis*-4-Methyl-1-tosylpyrrolidin-3-yl)(4-nitrophenyl)methanone (3c)



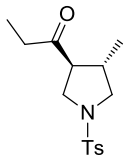
3c

trans/cis: 1.7: 1. One of the diastereomers could be separated on a silica gel column, and the relative configuration of **3c** was determined with reference to **3a**.

Pale yellow solid. ^1H NMR (300 MHz, $CDCl_3$): δ 8.32 (d, *J* = 8.4Hz, 2H), 8.04 (d, *J* = 8.4Hz, 2H), 7.77 (d, *J* = 7.8 Hz, 2H), 7.37 (d, *J* = 7.8 Hz, 2H), 4.07-3.99 (m, 1H),

3.75-3.58 (m, 3H), 3.08 (dd, *J* = 9.6, 3.6 Hz, 1H), 2.76-2.72 (m, 1H), 2.47 (s, 3H), 0.57 (d, *J* = 7.2 Hz, 3H)； ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.9, 150.9, 144.1, 141.1, 133.8, 128.0, 124.5, 55.4, 49.9, 47.9, 36.6, 21.9, 14.9; IR (FILM): ν_{max} 3108, 2967, 2925, 1688, 1602, 1526, 1493, 1407, 1346, 1221, 1164, 1093, 1032, 985 cm^{-1}; MS (ESI) *m*/*z*: 411($M+Na^+$); HRMS (ESI): calcd. for $C_{19}H_{20}N_2NaO_5S^+$ ($M+Na^+$): 411.10102, found: 411.09913.

1-(*trans*-4-Methyl-1-tosylpyrrolidin-3-yl)propan-1-one (3d)

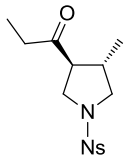


3d

trans/*cis*: 5.5:1. Major diastereomer could be separated on a silica gel column, and the relative configuration of **3d** was determined with reference to **3a**.

White solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.71 (d, *J* = 8.1Hz, 2H), 7.34 (d, *J* = 8.1Hz, 2H), 3.60 (dd, *J* = 9.6, 8.4Hz, 1H), 3.41 (dd, *J* = 9.6, 7.5Hz, 1H), 3.24 (dd, *J* = 9.9, 8.4Hz, 1H), 2.90 (dd, *J* = 9.6, 8.1Hz, 1H), 2.69 (dd, *J* = 16.5, 8.1Hz, 1H), 2.45 (s, 3H), 2.45-2.23 (m, 3H), 1.04-0.99 (m, 6H)； ^{13}C NMR (75 MHz, $CDCl_3$): δ 209.1, 143.7, 133.3, 129.7, 127.6, 56.7, 54.3, 49.9, 36.3, 36.1, 21.5, 17.5, 7.5; IR (FILM): ν_{max} 2972, 2937, 2877, 1713, 1598, 1459, 1379, 1343, 1162, 1093 cm^{-1}; MS (ESI) *m*/*z*: 318 ($M+Na^+$), 296 ($M+H^+$); HRMS (ESI): calcd. for $C_{15}H_{21}NNaO_3S^+$ ($M+Na^+$): 318.11344, found: 318.11249.

1-(*trans*-4-Methyl-1-(4-nitrophenylsulfonyl)pyrrolidin-3-yl)propan-1-one (3e)

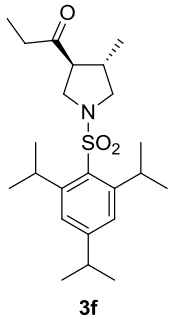


3e

trans/*cis*: 5.3:1. Major diastereomer could be separated on a silica gel column, and the relative configuration of **3e** was determined with reference to **3a**.

Pale yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.38 (d, *J* = 8.7Hz, 2H), 8.00 (d, *J* = 8.7Hz, 2H), 3.60 (dd, *J* = 9.9, 1.8Hz, 1H), 3.38-3.31 (m, 2H), 2.99 (dd, *J* = 9.6, 7.2Hz, 1H), 2.77 (dd, *J* = 15.0, 7.5Hz, 1H), 2.46-2.39 (m, 2H), 2.38-2.28 (m, 1H), 1.05 (d, *J* = 6.6Hz, 3H), 0.99 (t, *J* = 7.2Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 209.2, 150.4, 142.9, 128.9, 124.7, 56.6, 54.5, 49.8, 36.9, 36.4, 17.9, 7.8; MS (EI) *m*/*z*: 326 (M^+, 1), 140 (100), 122 (13), 113 (48), 108 (12), 85 (15), 84 (60), 82(41); HRMS (EI): calcd. for $C_{12}H_{13}N_2O_5S^+$ (M^+): 297.0545, found: 297.0542.

1-(*trans*-4-Methyl-1-(2,4,6-triisopropylphenylsulfonyl)pyrrolidin-3-yl)propan-1-one (3f)



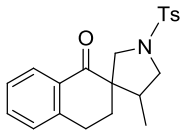
3f

trans/*cis*: 5.2:1. Major diastereomer could be separated on a silica gel column, and the relative configuration of **3f** was determined with reference to **3a**.

Pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15 (s, 2H), 4.21-4.15 (m, 2H), 3.63 (dd, *J* = 7.2, 6.3Hz, 1H), 3.41 (dd, *J* = 6.9, 5.4Hz, 1H), 3.31 (dd, *J* = 7.5, 6.3Hz, 1H), 2.97-2.78 (m, 3H), 2.51-2.44 (m, 3H), 1.27-1.22 (m, 18H), 1.09 (d, *J* = 5.1Hz,

3H), 1.04 (t, *J* = 5.4Hz, 3H); ^{13}C NMR(100 MHz, $CDCl_3$): δ 209.6, 153.1, 151.2, 131.0, 123.8, 57.0, 53.2, 48.6, 36.7, 36.1, 34.1, 29.3, 24.8, 23.5, 17.3, 7.5; MS (EI) *m*/*z*: 407 (M^+, 1), 306 (14), 268 (18), 267 (100), 251 (32), 249 (9), 218 (24), 203 (14); HRMS (EI): calcd. for $C_{23}H_{37}NO_3S^+$ (M^+): 407.2494, found: 407.2487.

4'-Methyl-1'-tosyl-3,4-dihydro-1*H*-spiro[naphthalene-2,3'-pyrrolidin]-1-one (3g)



3g

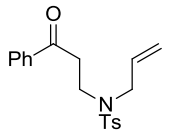
trans/*cis*: 1.8: 1. One of diastereomers could be separated on a silica gel column.

White solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.2Hz, 1H), 7.72 (d, *J* = 8.1Hz, 2H), 7.49-7.44 (m, 1H), 7.35-7.26 (m, 3H), 7.21 (d, *J* = 7.8Hz, 2H), 3.83 (d, *J* = 9.9Hz, 1H), 3.67 (dd, *J* = 9.3, 7.2Hz, 1H), 3.27 (d, *J* = 10.5Hz, 1H), 3.13-3.02 (m, 2H), 2.94-2.85 (m, 1H), 2.46 (s, 3H), 2.39-2.32 (m, 1H), 2.25-2.17 (m, 1H), 2.08-1.99 (m, 1H), 0.71 (d, *J* = 6.9Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 197.7, 143.4, 142.6, 133.6, 133.3, 132.0, 129.6, 128.7, 127.62, 127.57, 126.8, 55.6, 54.8, 54.2, 39.9, 33.2, 25.8, 21.6, 14.1; MS(ESI) *m*/*z*:: 392 ($M+Na^+$), 370 ($M+H^+$); HRMS (ESI): calcd. for $C_{21}H_{23}NNaO_3S^+$ ($M+Na^+$): 392.12909, found: 392.12958.

General procedure for control experiment

A mixture of $AgClO_4$ (20.7 mg, 0.1 mmol) (*Warning! The perchlorate salt is potentially explosive and should be handled with great caution.*), α,β-unsaturated ketone **1a** (1.0 mmol) and substituted allylic amine **2a** (1.5 mmol, 1.5 equiv) in toluene (2 mL) was stirred at 90 °C under Ar atmosphere for 3 h. Then, the solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography (eluent: EtOAc/petroleum ether = 1:10) to give the desired product **4** in 85% yield (299 mg, 0.85 mmol).

***N*-Allyl-4-methyl-N-(3-oxo-3-phenylpropyl)benzenesulfonamide (4)**



4

Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.93 (d, *J* = 8.0 Hz, 2H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.58 (t, *J* = 7.0 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 2H), 7.28 (t, *J* = 8.6 Hz, 2H), 5.75-5.62 (m, 1H), 5.22-5.13 (m, 2H), 3.84 (d, *J* = 6.6 Hz, 2H), 3.48 (t, *J* = 6.5 Hz, 2H), 3.36 (t, *J* = 7.2 Hz, 2H), 2.44 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 198.3, 143.4, 136.3, 136.2, 133.3, 133.0, 129.7, 128.6, 127.9, 127.1, 119.3, 52.1, 43.1, 38.9, 21.5. IR(FILM): ν_{max} 3064, 2922, 1682, 1644, 1598, 1581, 1494, 1449, 1417, 1382, 1342, 1306, 1287, 1211, 1157, 1092, 1018, 986, 931, 876 cm^{-1}. MS(ESI) *m*/*z*: 366 ($M+Na^+$), 344 ($M+H^+$). HRMS(ESI): calcd. for $C_{19}H_{21}NO_3SNa^+$($M+Na^+$): 366.1134, found: 366.1141. Anal. calcd. for $C_{19}H_{21}O_3NS$: C, 66.45; H, 6.16; N, 4.08, found: C, 66.45; H, 6.10; N, 4.05.

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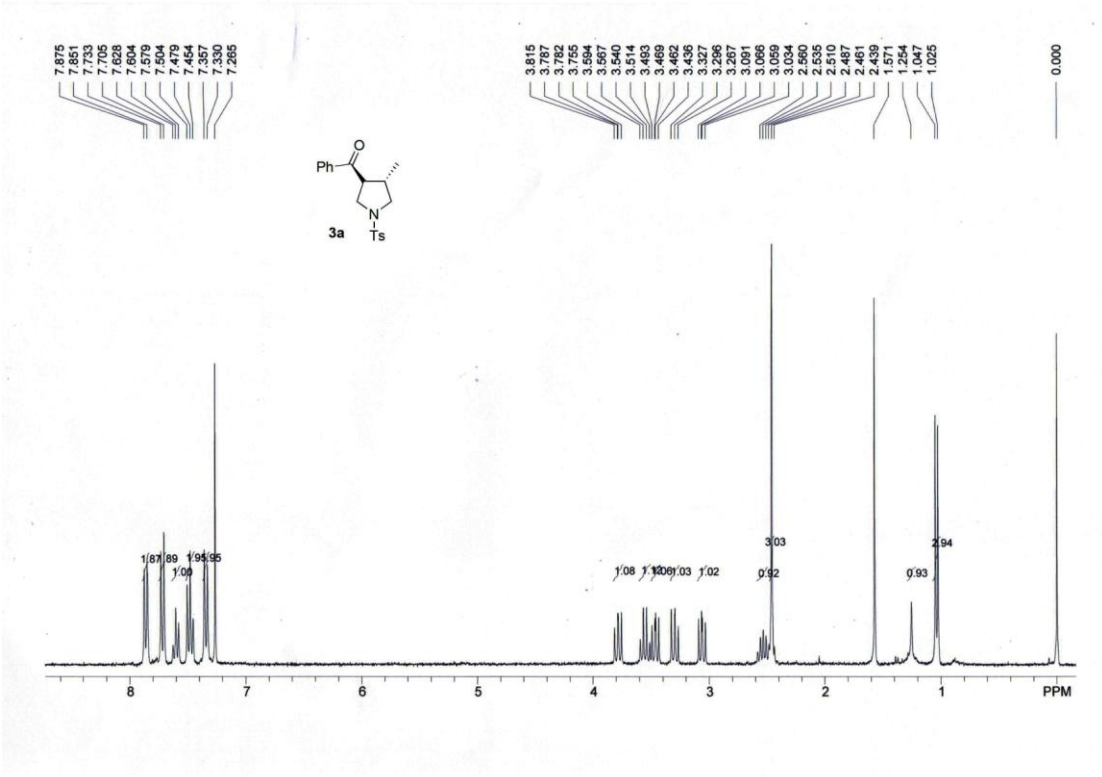
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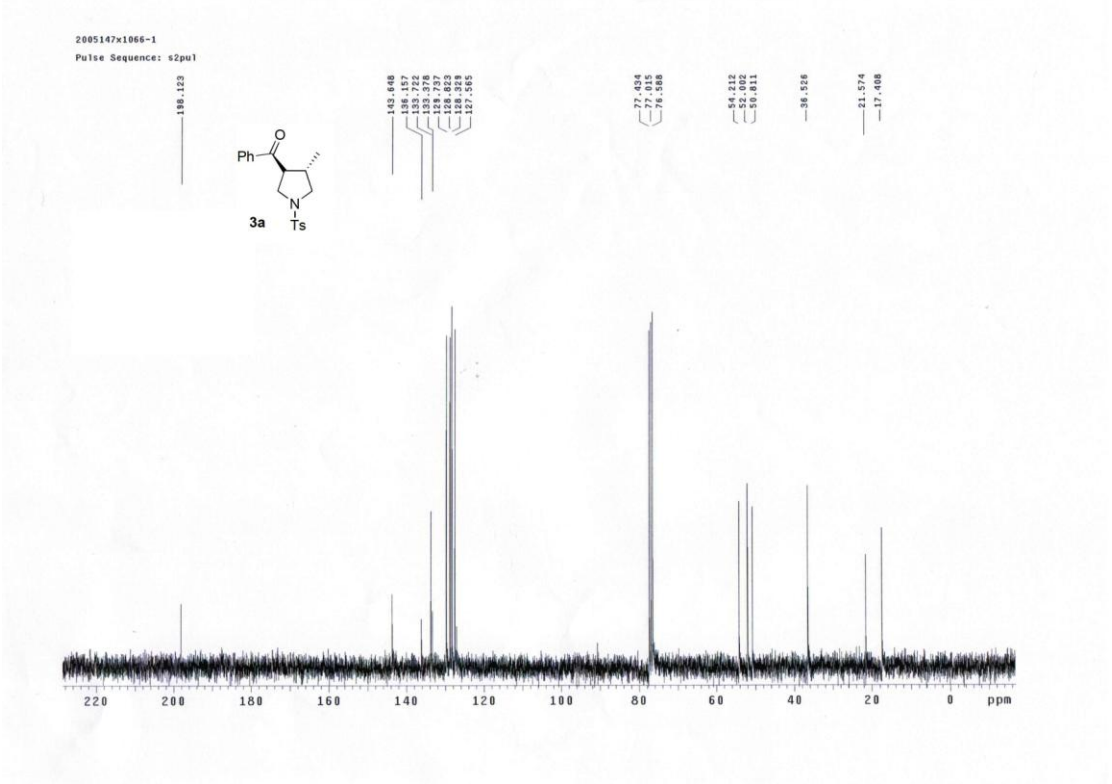
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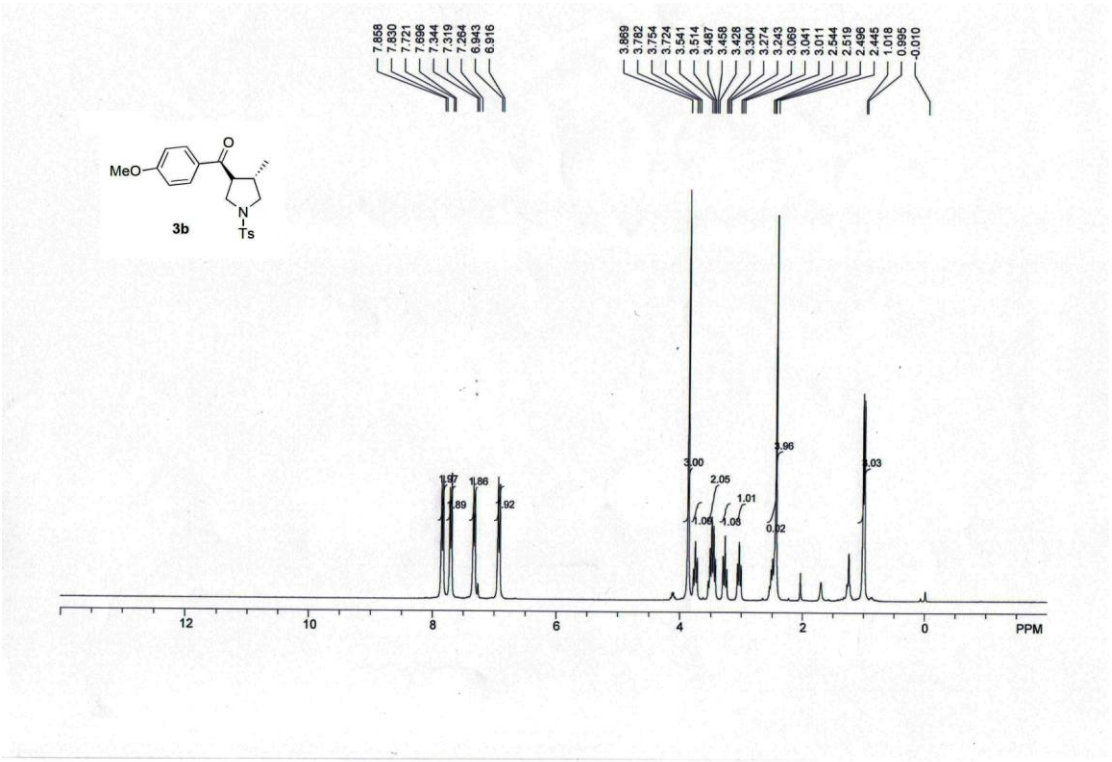
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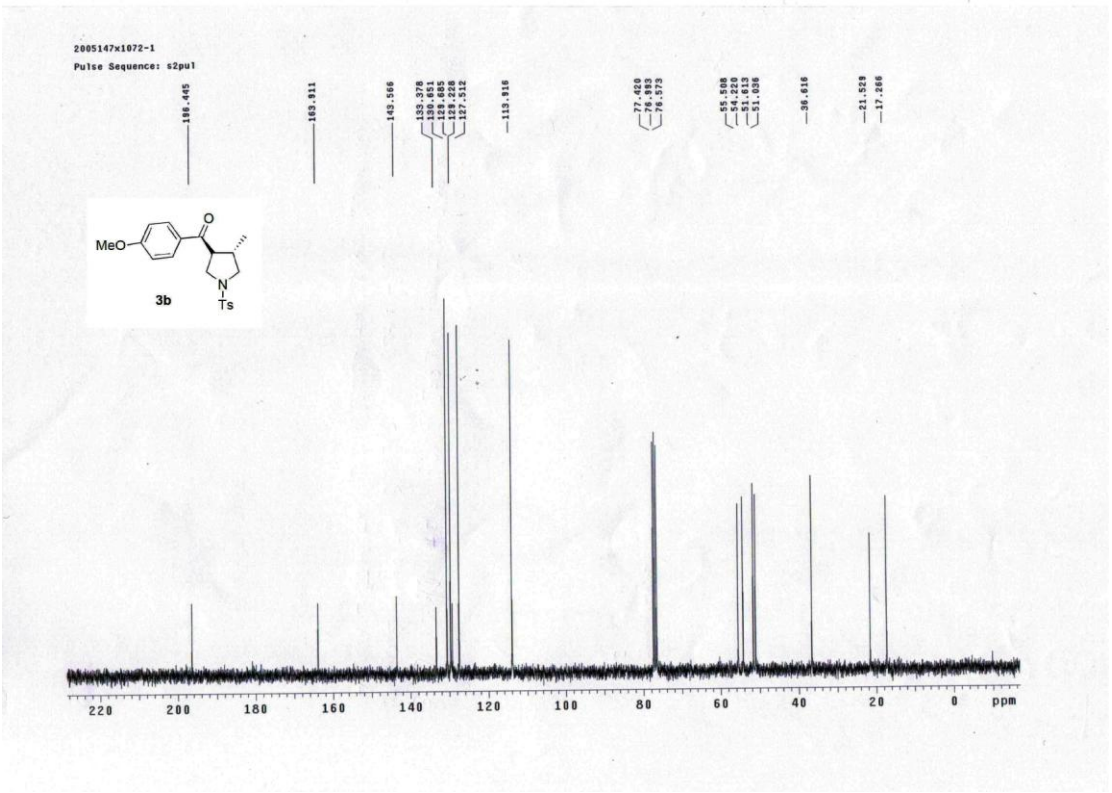
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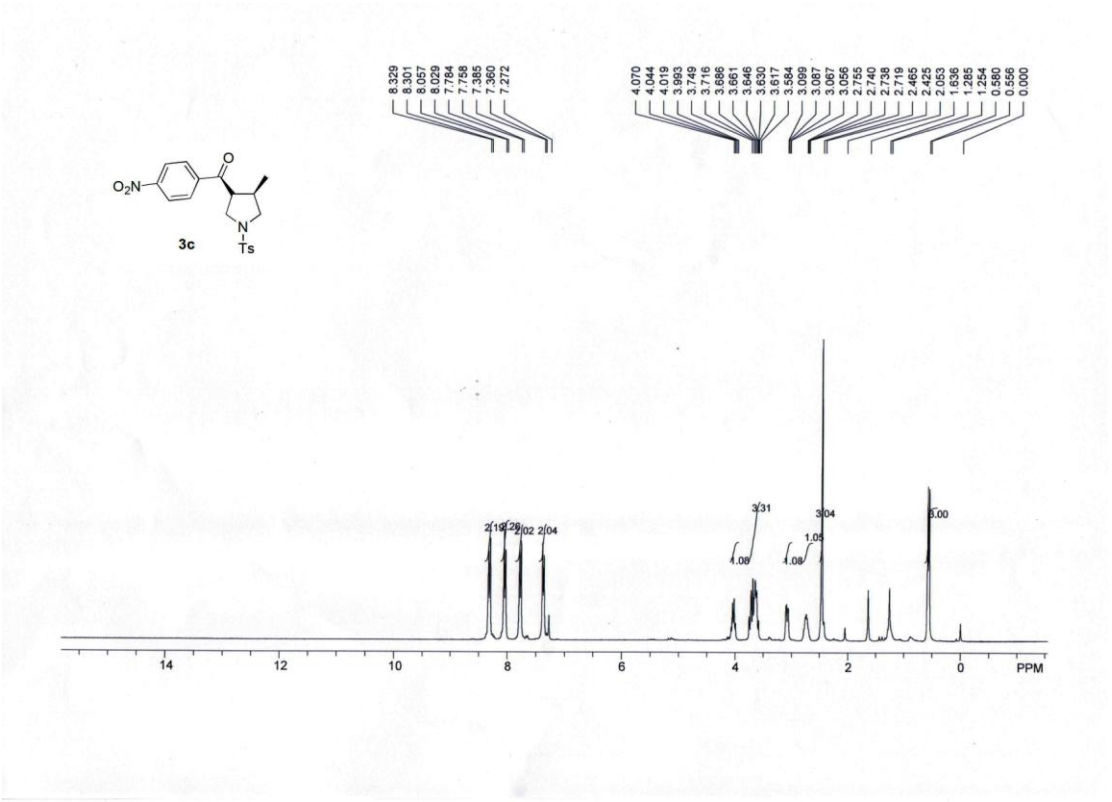
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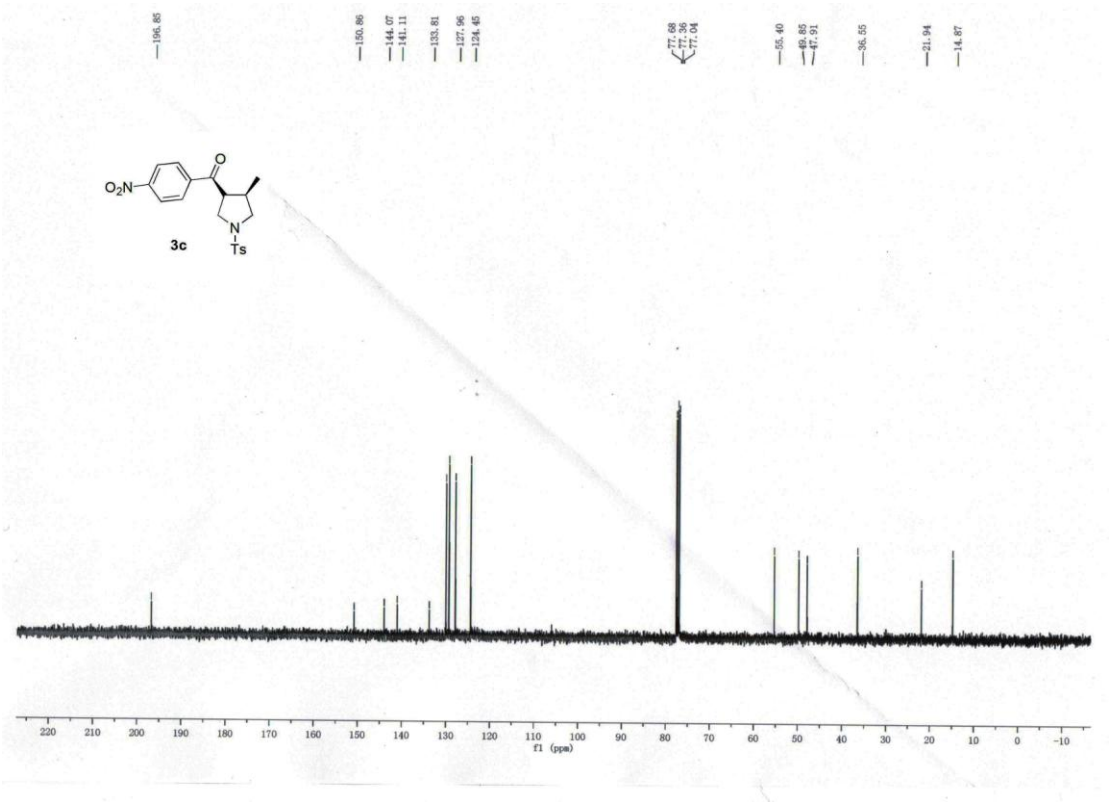
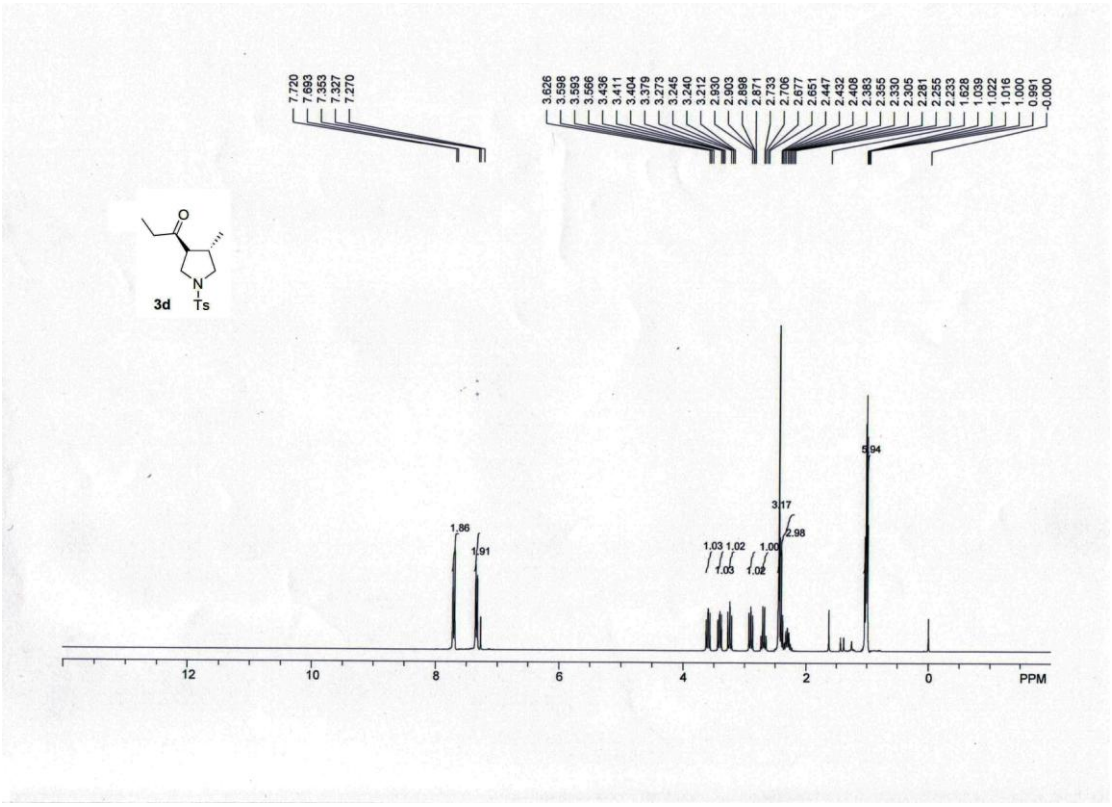
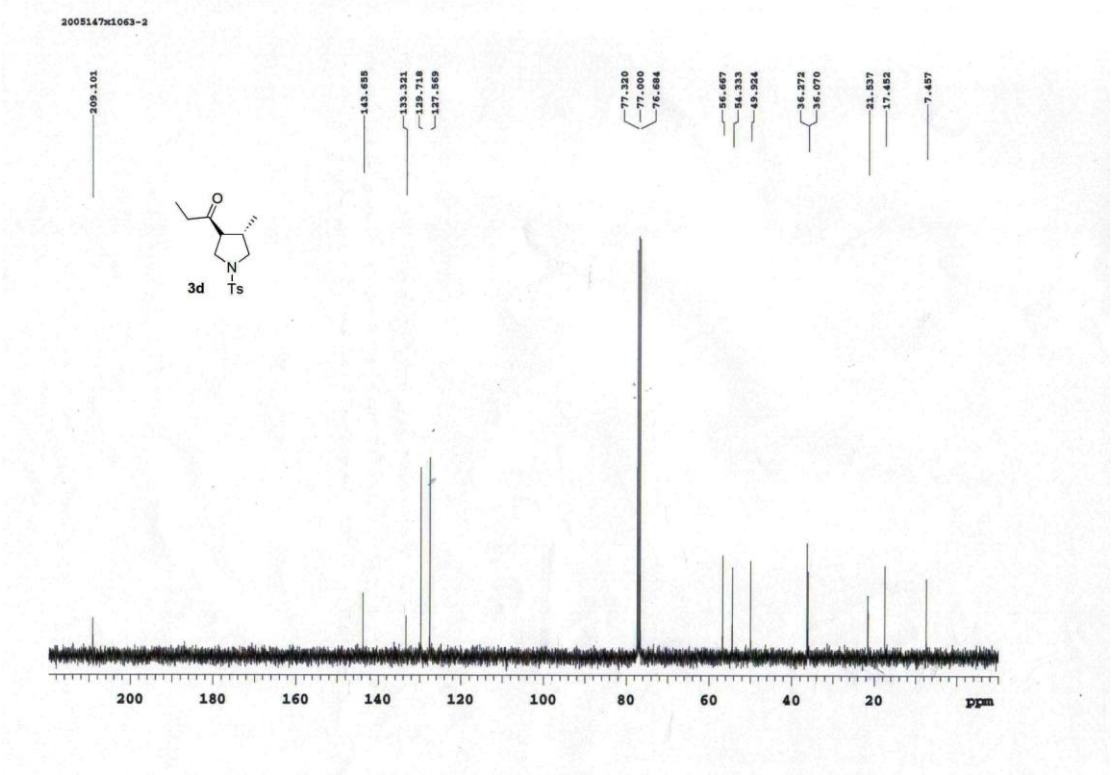
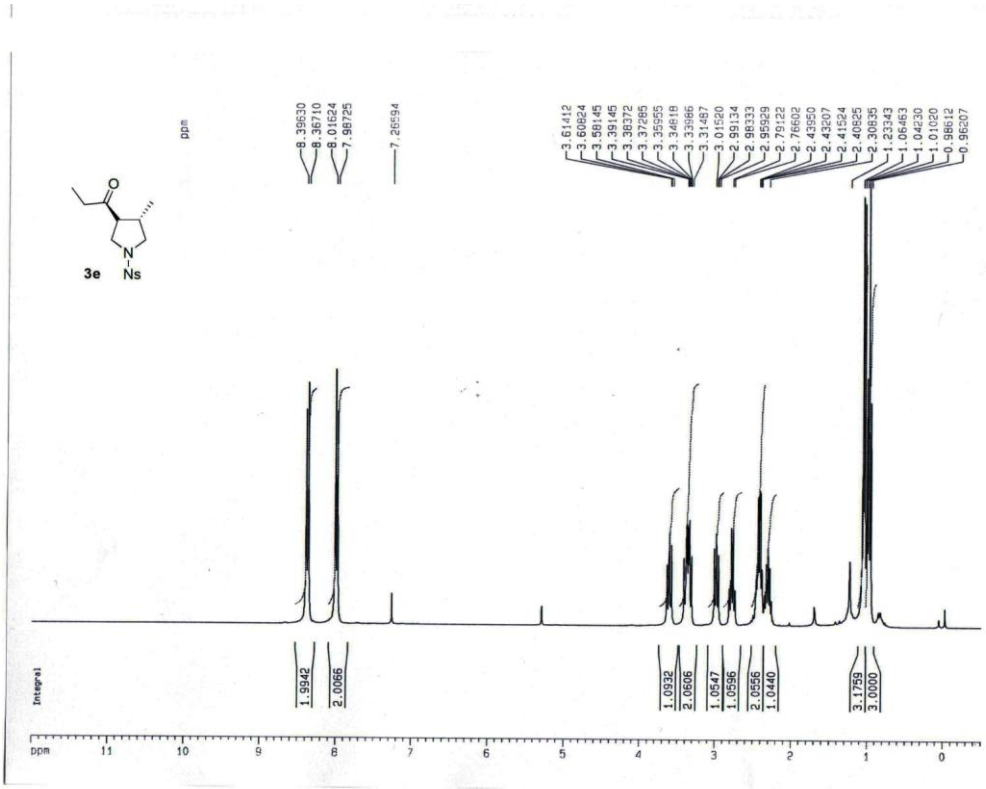
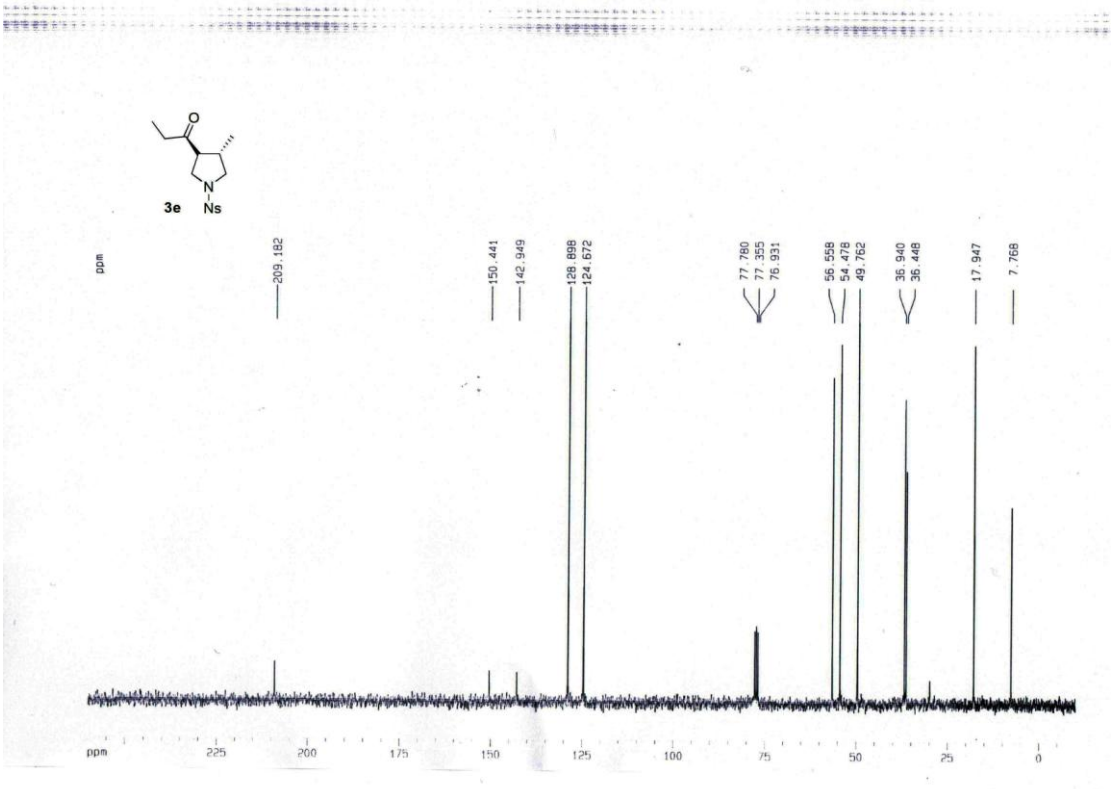
PPM

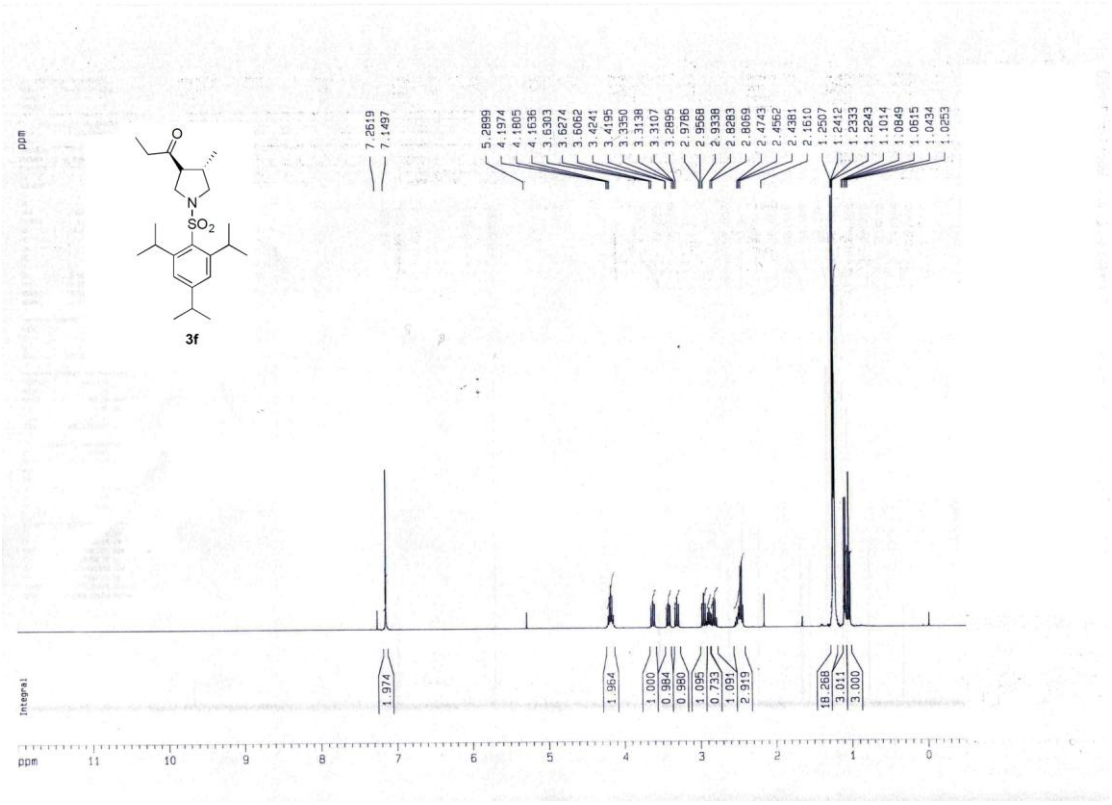

196.85
150.86
144.07
141.11
133.81
127.96
124.45
77.68
77.36
77.04
55.40
49.85
47.91
36.55
21.94
14.87
O2N
O
N
Ts
3c
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

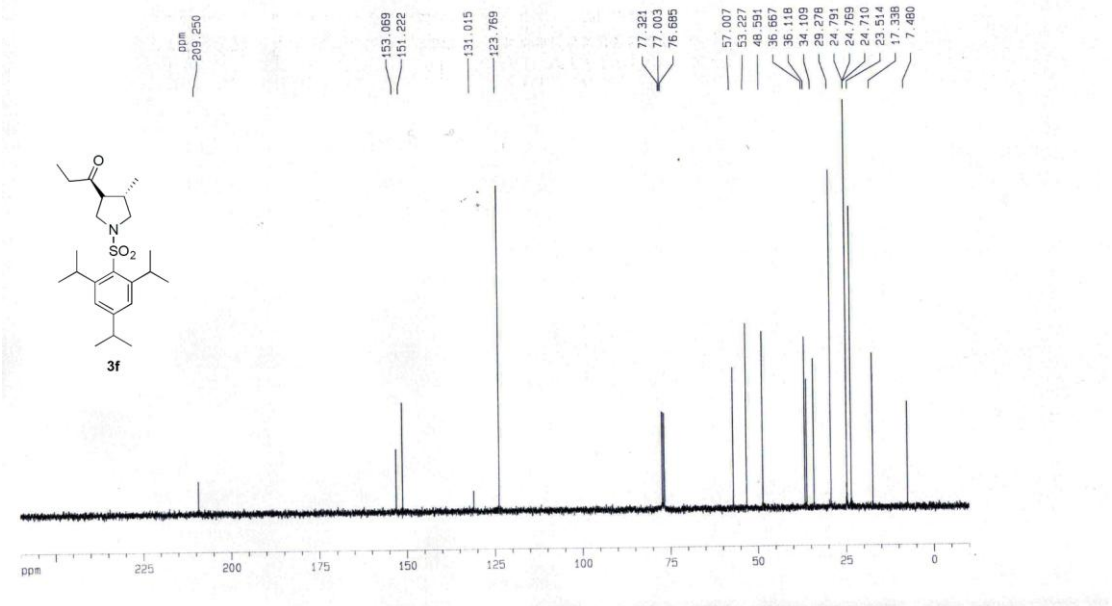

7.720
7.693
7.353
7.327
7.270
3.626
3.598
3.593
3.566
3.436
3.411
3.404
3.379
3.273
3.245
3.240
3.212
2.930
2.903
2.898
2.871
2.733
2.706
2.677
2.651
2.447
2.432
2.408
2.383
2.355
2.330
2.306
2.281
2.255
2.233
1.628
1.039
1.022
1.016
1.000
0.991
-0.000
3d
Ts
1.86
1.91
1.03
1.03
1.02
1.02
1.00
3.17
2.98
5.94
12
10
8
6
4
2
0
PPM

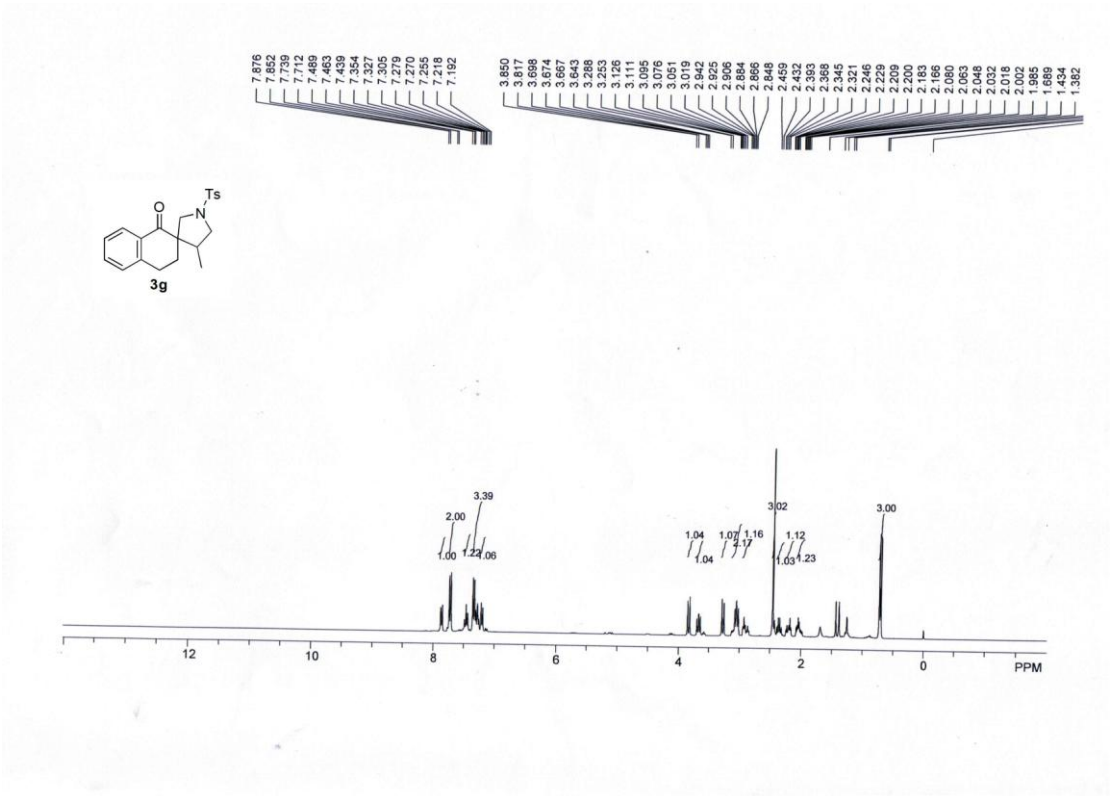

2005147m1063-2
209.101
143.685
133.321
129.718
127.569
77.320
77.000
76.684
56.667
54.333
49.324
36.272
36.070
21.537
17.452
7.457
3d
Ts
200
180
160
140
120
100
80
60
40
20
ppm

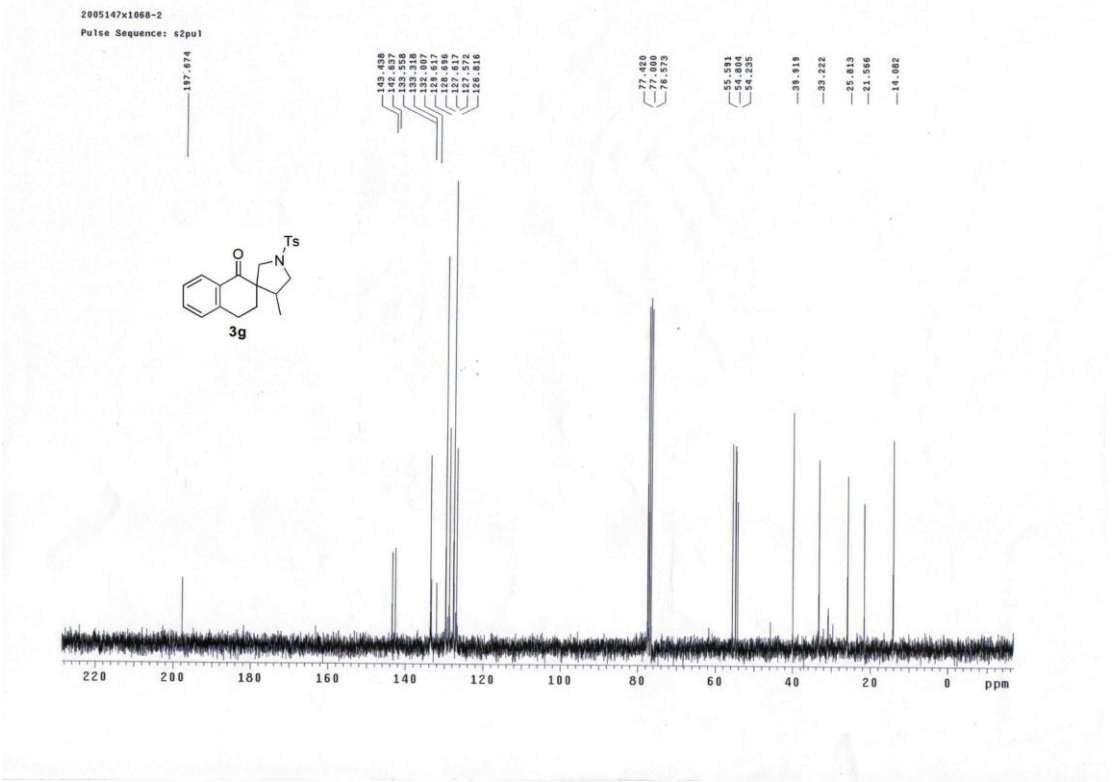

3e
Ns
ppm
8.39630
8.36710
8.01624
7.98725
7.26594
3.61412
3.60824
3.58145
3.39145
3.38372
3.37285
3.35555
3.34618
3.33986
3.31487
3.01520
2.99134
2.98333
2.95929
2.79122
2.76602
2.43207
2.43050
2.41524
2.40825
2.30835
1.23343
1.06463
1.04230
1.01020
0.98612
0.96207
Integral
1.9942
2.0066
1.0932
2.0606
1.0547
1.0596
2.0556
1.0440
3.1759
3.0000
ppm

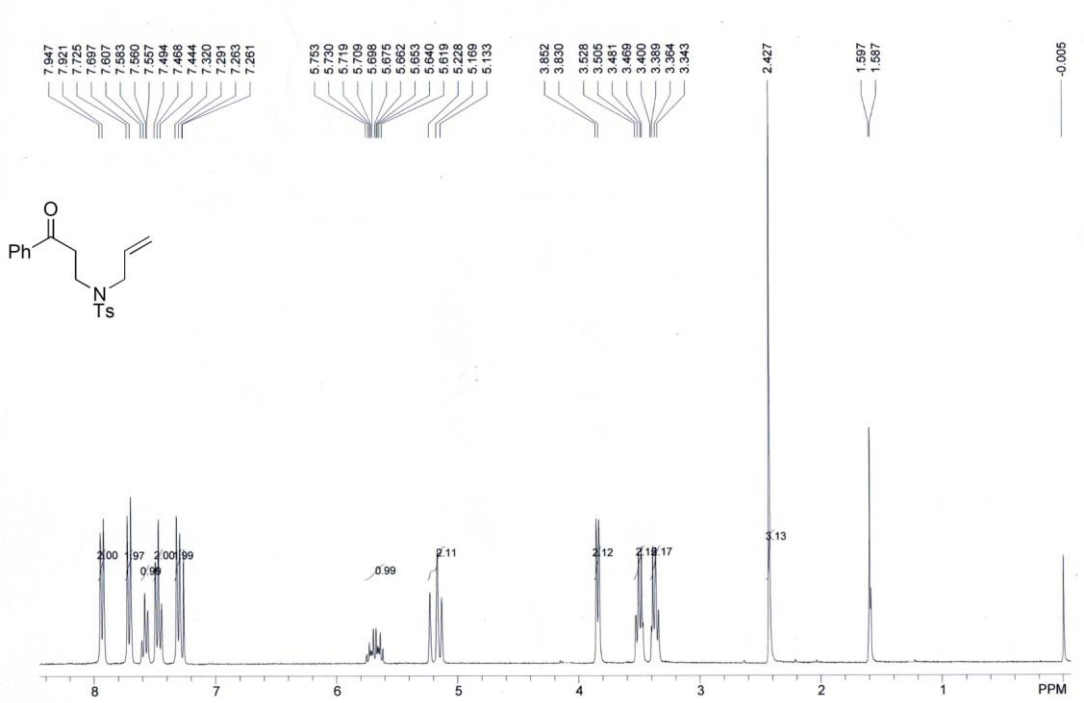

3e
Ns
ppm
209.182
150.441
142.949
128.898
124.672
77.780
77.355
76.931
56.558
54.478
49.762
36.940
36.448
17.947
7.768
ppm

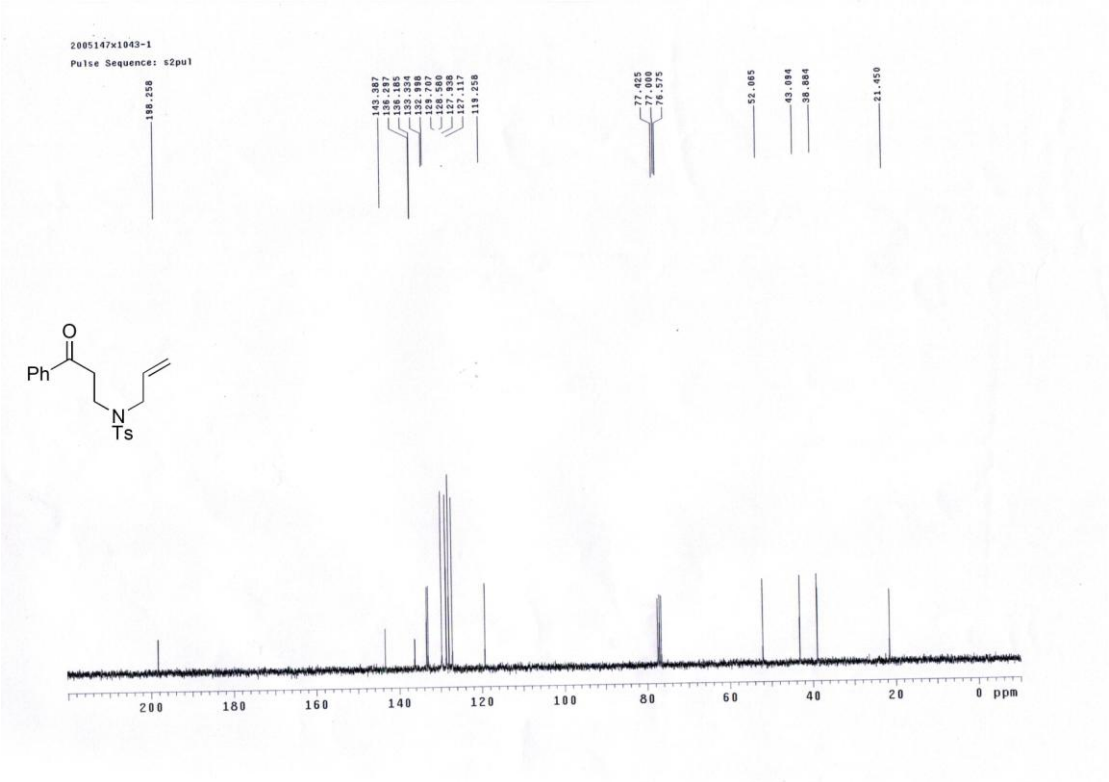

3f
7.2619
7.1497
5.2899
4.1974
4.1805
4.1636
3.6303
3.6274
3.6062
3.4241
3.4195
3.3350
3.3138
3.3107
3.2895
2.9786
2.9568
2.9338
2.8283
2.8069
2.4743
2.4562
2.4381
2.1610
1.2507
1.2412
1.2333
1.2243
1.1014
1.0849
1.0615
1.0434
1.0253
Integral
1.974
1.964
1.000
0.984
0.980
1.095
0.733
1.091
2.919
18.268
3.011
3.000
ppm


3f
ppm
209.250
153.069
151.222
131.015
123.769
77.321
77.003
76.685
57.007
53.227
48.591
36.667
36.118
34.109
29.278
24.791
24.769
24.710
23.514
17.338
7.480
ppm


7.876
7.852
7.739
7.712
7.489
7.463
7.439
7.354
7.327
7.305
7.279
7.270
7.255
7.218
7.192
3.850
3.817
3.698
3.674
3.667
3.643
3.288
3.253
3.126
3.111
3.095
3.076
3.051
3.019
2.942
2.925
2.906
2.884
2.866
2.848
2.459
2.432
2.393
2.368
2.345
2.321
2.246
2.229
2.209
2.200
2.183
2.166
2.080
2.063
2.048
2.032
2.018
2.002
1.985
1.686
1.434
1.382
Ts
O
N
3g
3.39
2.00
1.00
1.22
1.06
3.02
3.00
1.04
1.07
1.16
1.12
1.04
1.17
1.03
1.23
12
10
8
6
4
2
0
PPM


2005147x1068-2
Pulse Sequence: s2pul
197.674
143.438
143.037
133.558
133.318
132.007
129.617
128.696
127.617
127.572
126.816
77.420
77.000
76.573
55.591
54.804
54.235
39.819
33.222
25.813
21.566
14.082
Ts
O
N
3g
220
200
180
160
140
120
100
80
60
40
20
0
ppm


7.947
7.921
7.725
7.697
7.607
7.583
7.560
7.557
7.494
7.468
7.444
7.320
7.291
7.263
7.261
5.753
5.730
5.719
5.709
5.698
5.675
5.662
5.653
5.640
5.619
5.228
5.169
5.133
3.852
3.830
3.528
3.505
3.481
3.469
3.400
3.389
3.364
3.343
2.427
1.597
1.587
-0.005
O
Ph
N
Ts
2.00
1.97
2.00
0.99
0.99
2.11
2.12
2.18
2.17
3.13
8
7
6
5
4
3
2
1
PPM


2005147x1043-1
Pulse Sequence: s2pul
198.358
143.387
136.297
136.165
133.334
132.986
129.707
128.560
127.938
127.117
119.258
77.425
77.000
76.575
52.065
43.094
38.884
21.450
O
Ph
N
Ts
200
180
160
140
120
100
80
60
40
20
0 ppm