Synthesis

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Supporting Information
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Thieme

Supporting Information

Phosphite-mediated Reductive Cross-Coupling of Isatins and Nitrostyrenes

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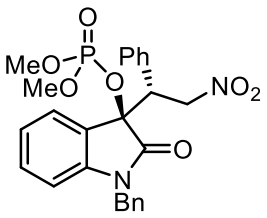
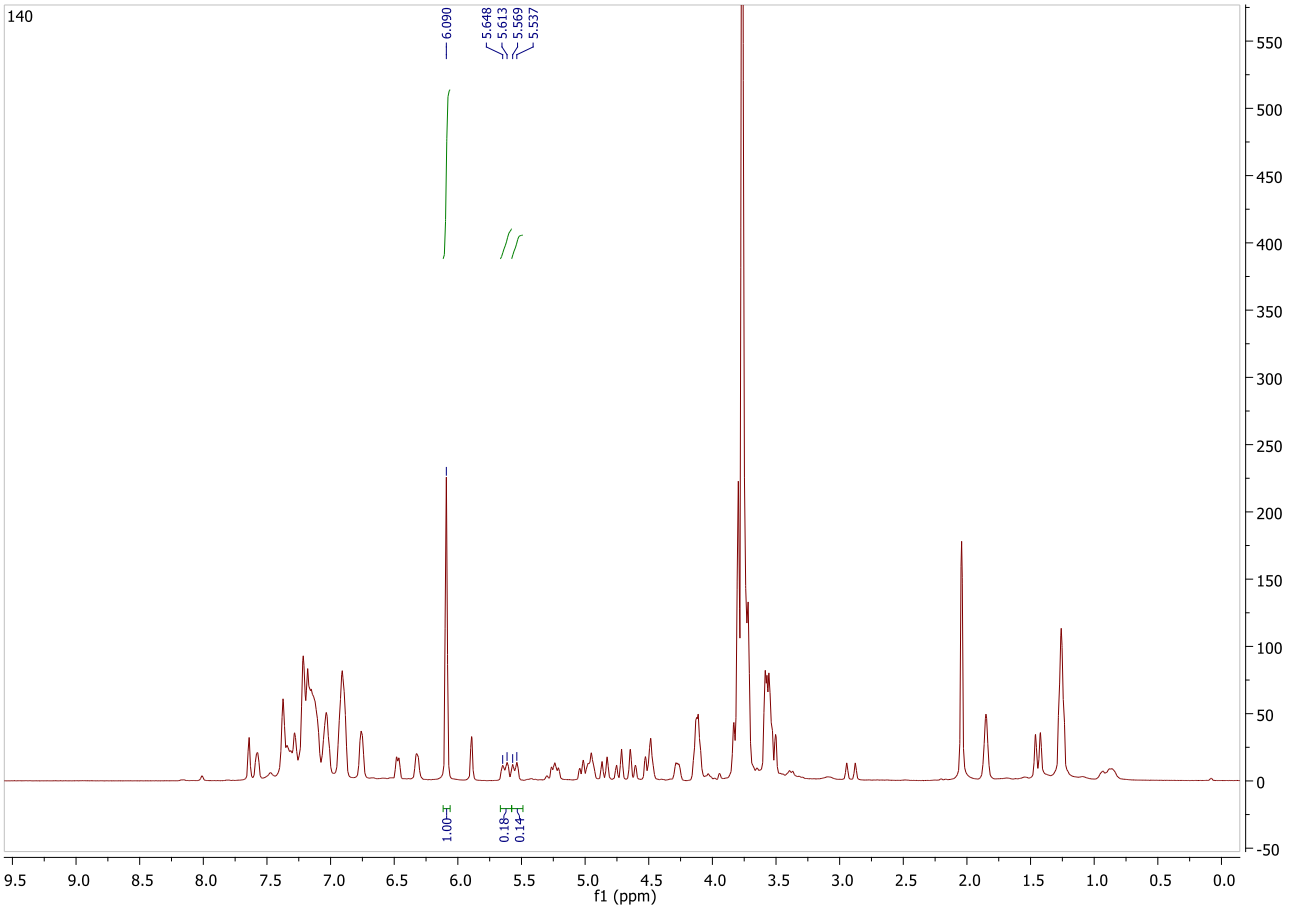
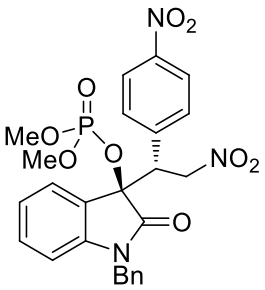
Table of Contents	**Page**
Crude ^{1}H NMR spectra	S2
^{1}H and ^{13}C NMR spectra of new compounds	S22
HPLC traces	S86

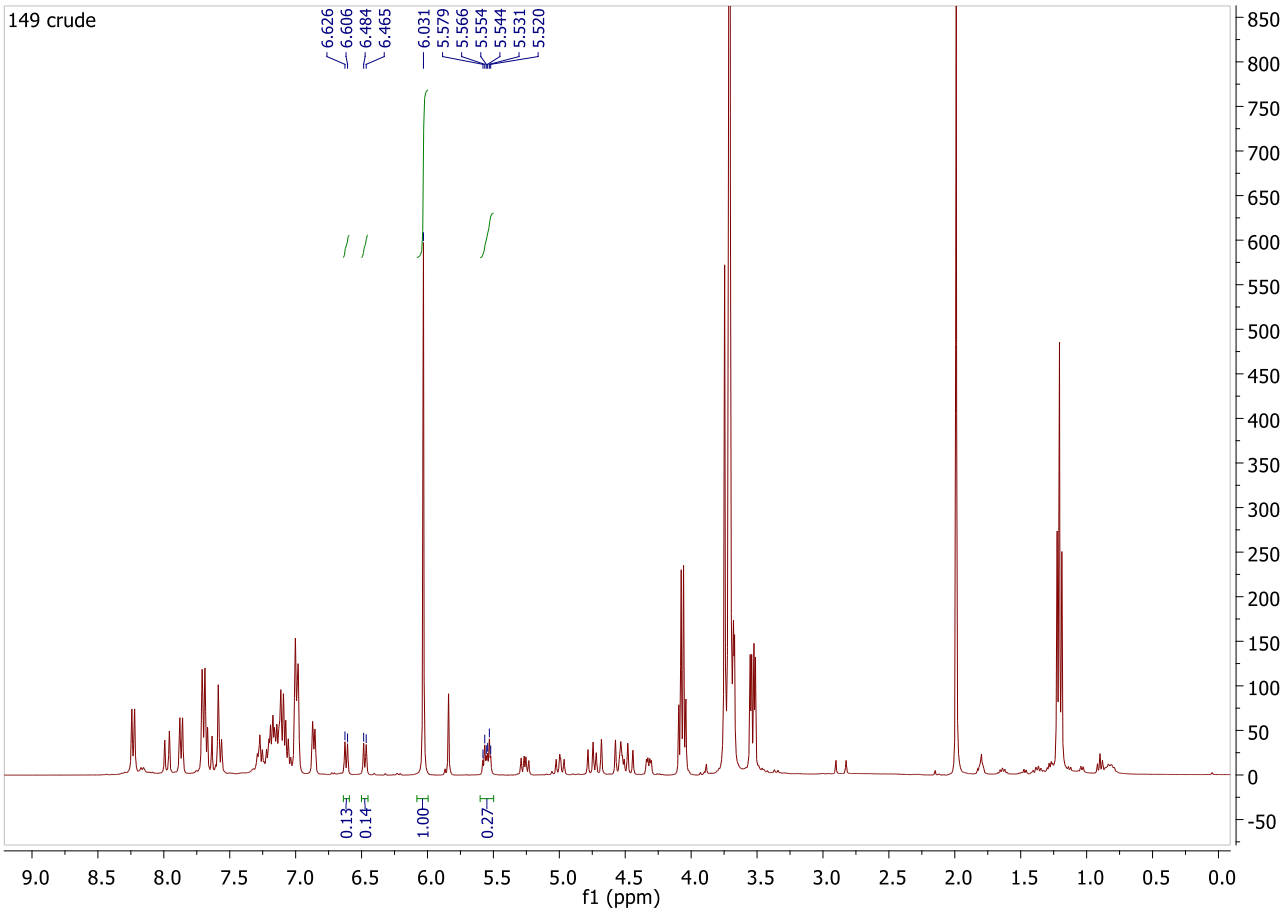
Crude ^{1}H NMR spectra for the racemic reactions



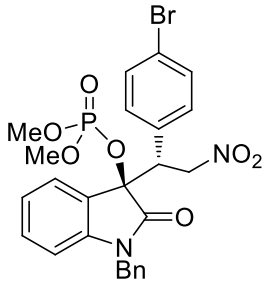


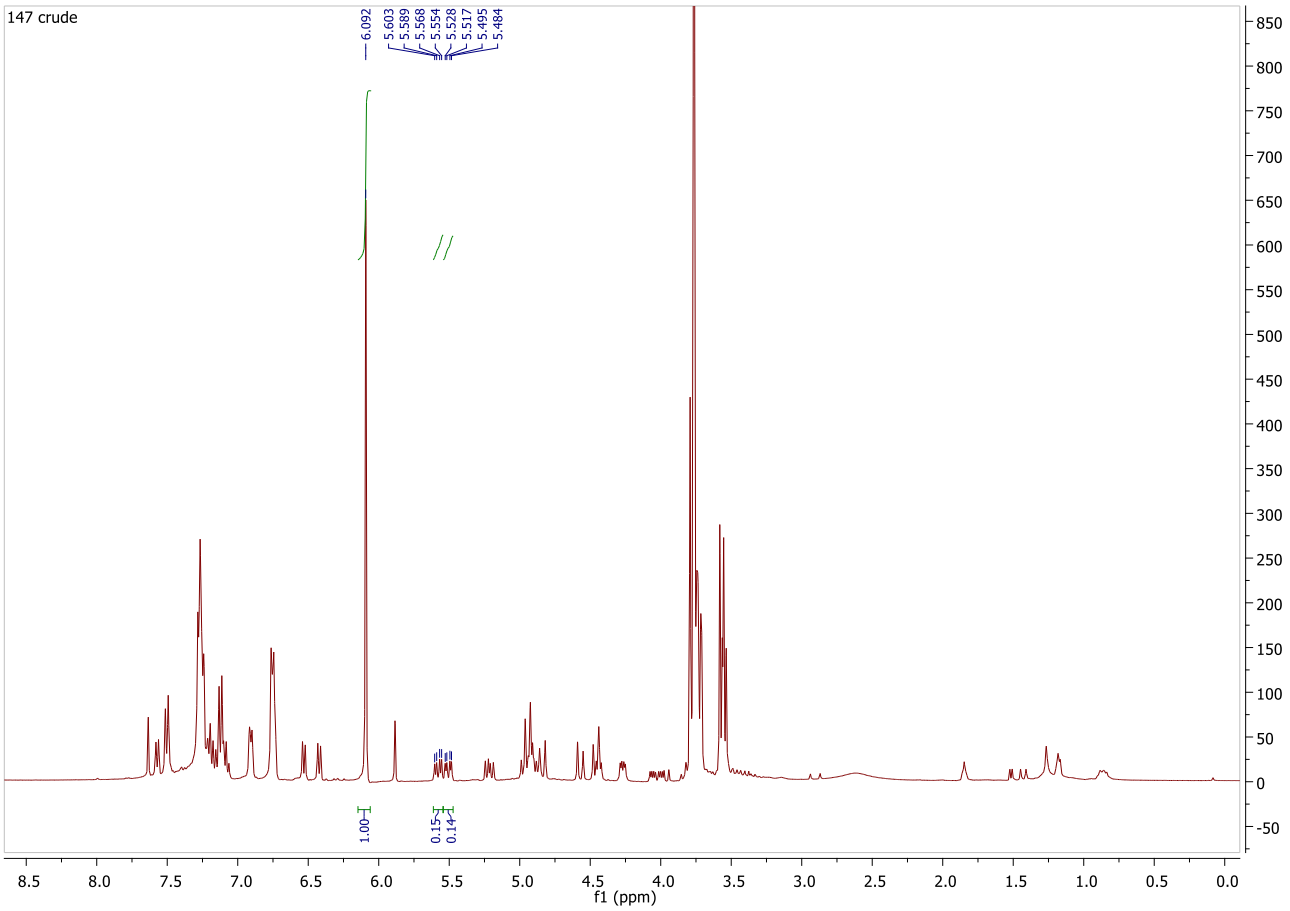
^{1}H NMR of 4aa


NO2
O
MeO
P
MeO
O
NO2
O
N
Bn

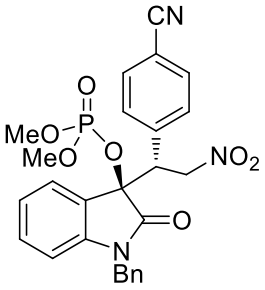

149 crude
6.626
6.606
6.484
6.465
6.031
5.579
5.566
5.554
5.544
5.531
5.520
0.13
0.14
1.00
0.27
f1 (ppm)

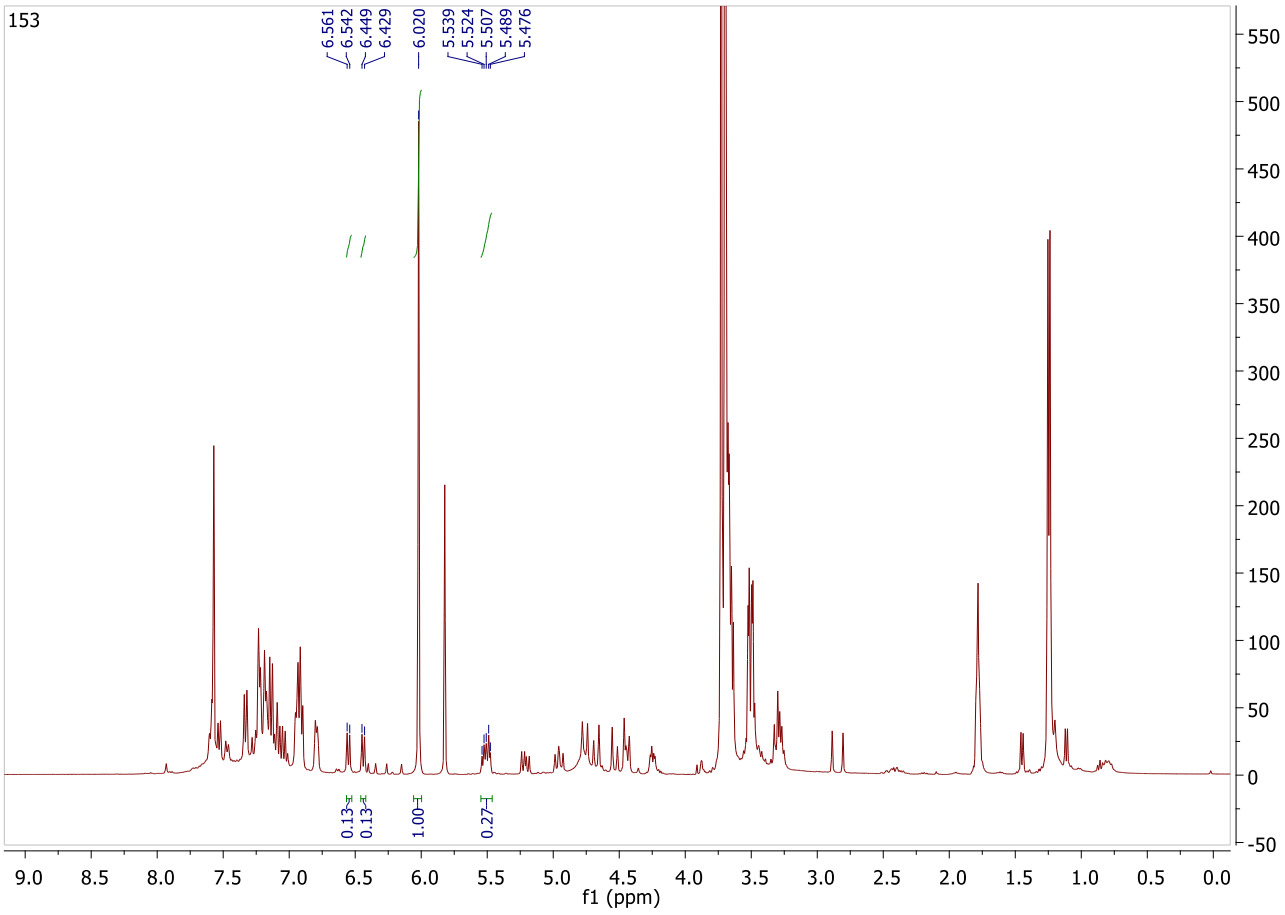
^{1}H NMR of 4ab


Br
O
MeO
P
MeO
O
NO2
O
N
Bn

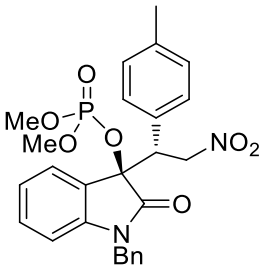

147 crude
6.092
5.603
5.589
5.568
5.554
5.528
5.517
5.495
5.484
1.00
0.15
0.14
f1 (ppm)

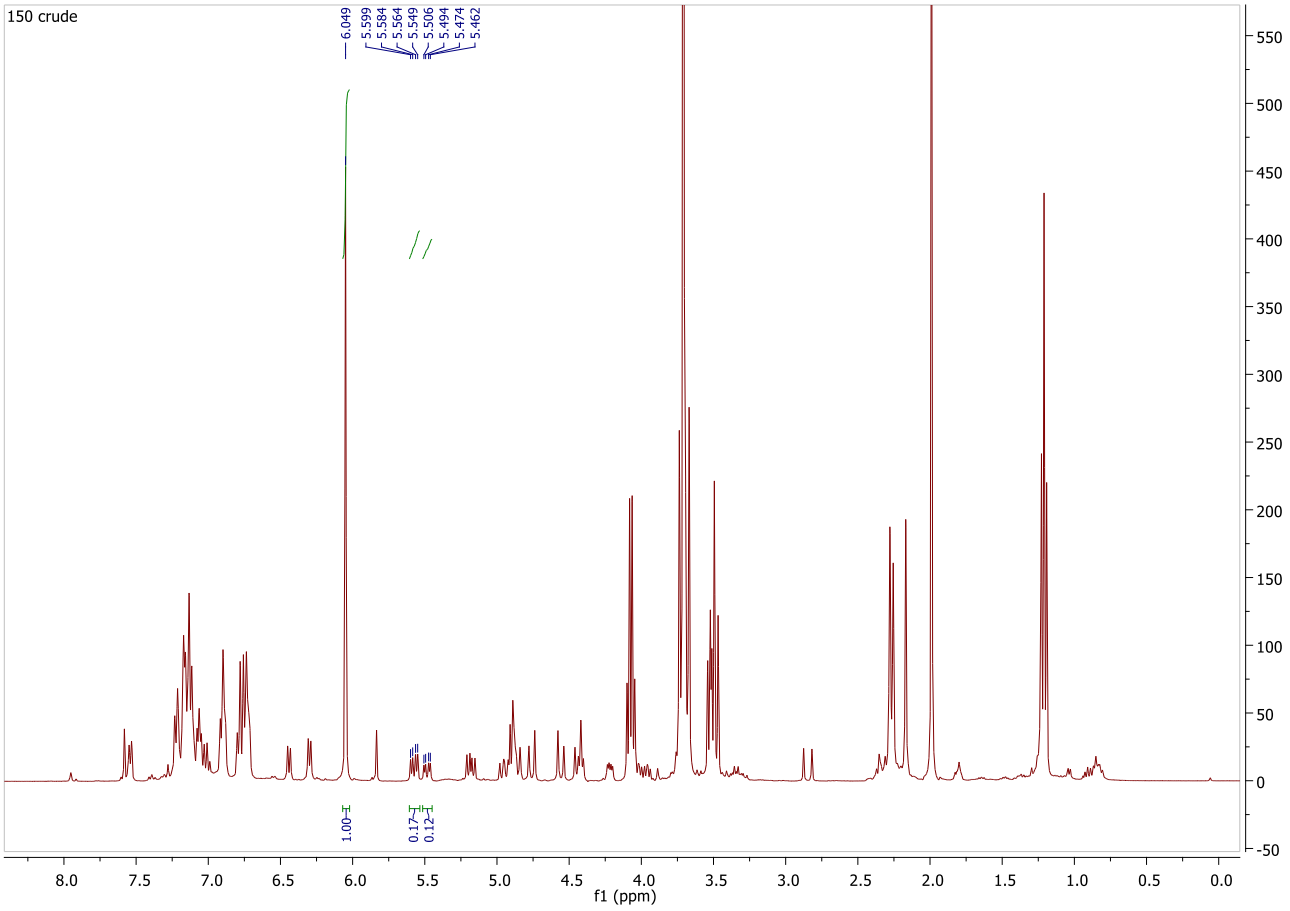
^{1}H NMR of 4ac


CN
O
P
MeO
MeO
O
NO2
O
N
Bn

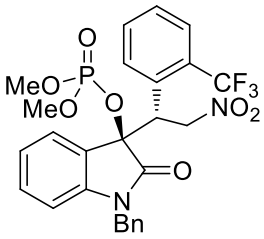

153
6.561
6.542
6.449
6.429
6.020
5.539
5.524
5.507
5.489
5.476
0.13
0.13
1.00
0.27
f1 (ppm)

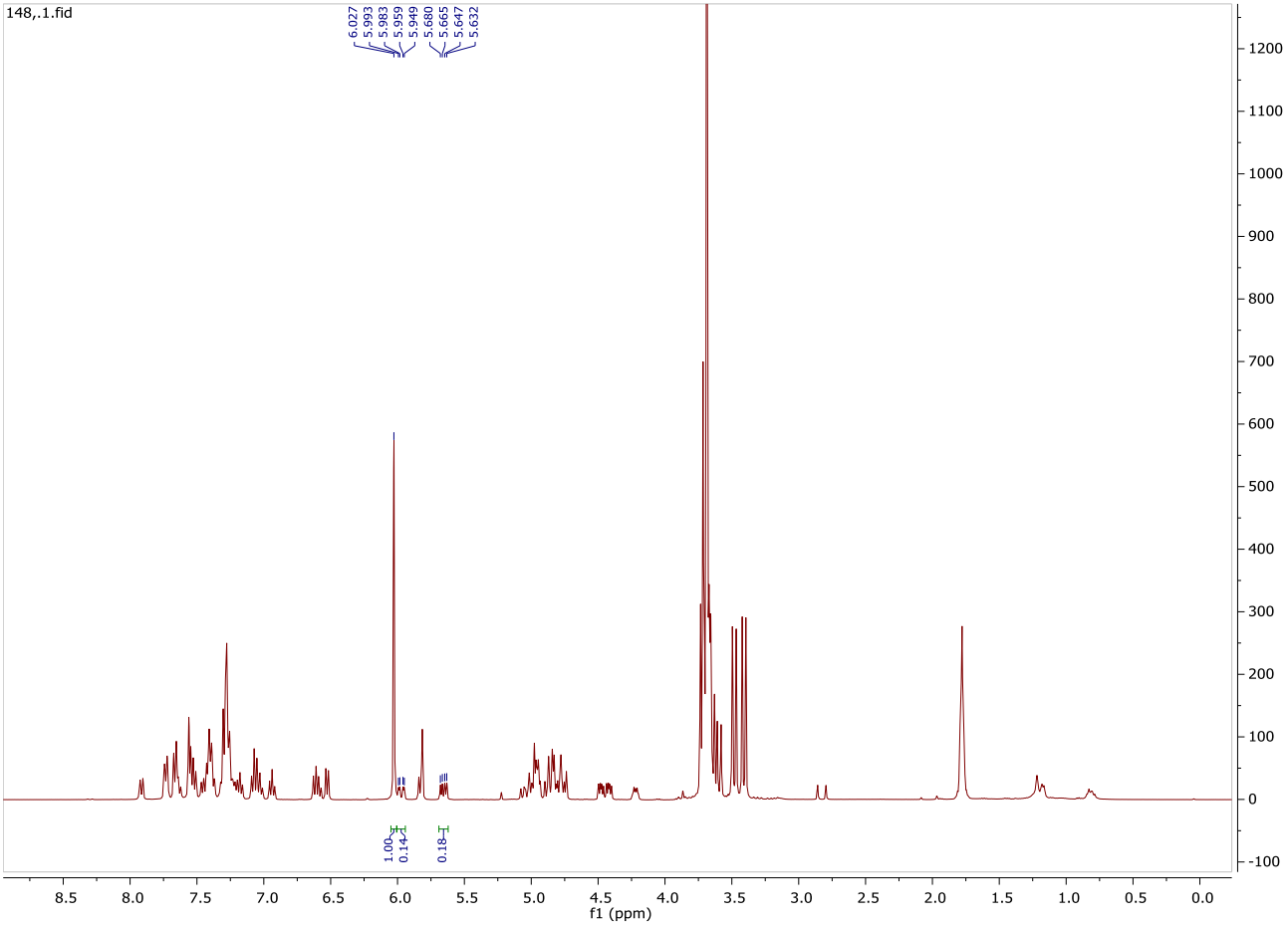
^{1}H NMR of 4ad


O
MeO
MeO
P
O
NO2
O
N
Bn

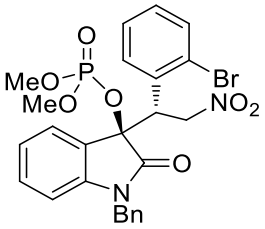

150 crude
6.049
5.599
5.584
5.564
5.549
5.506
5.494
5.474
5.462
1.00
0.17
0.12
f1 (ppm)

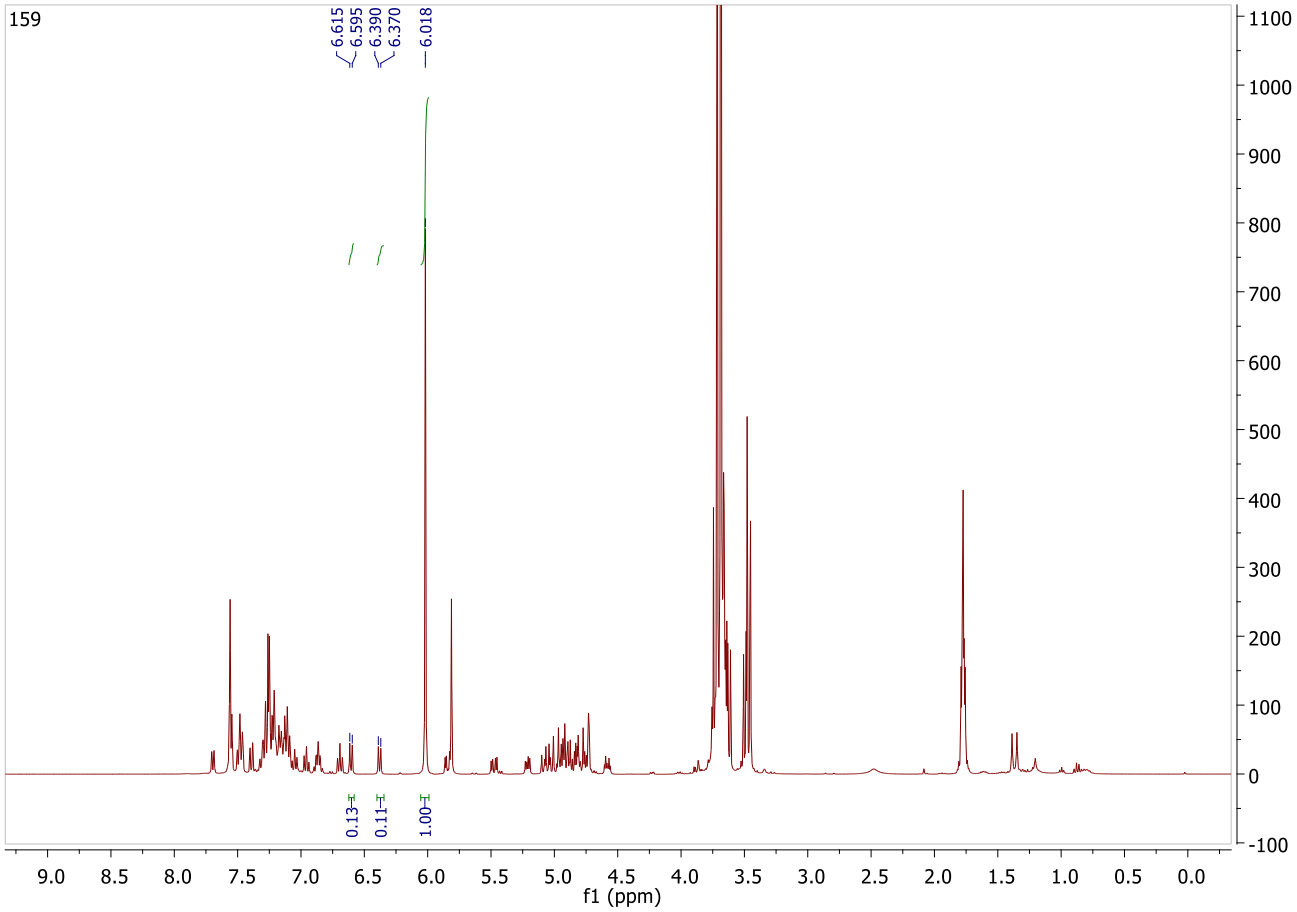
^{1}H NMR of 4ae


O
MeO
MeO
P
O
CF3
NO2
O
N
Bn

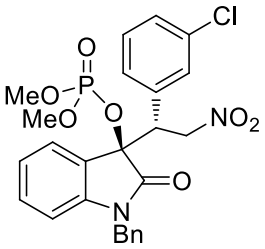

148,.1.fid
6.027
5.993
5.983
5.959
5.949
5.680
5.665
5.647
5.632
1.00
0.14
0.18
f1 (ppm)

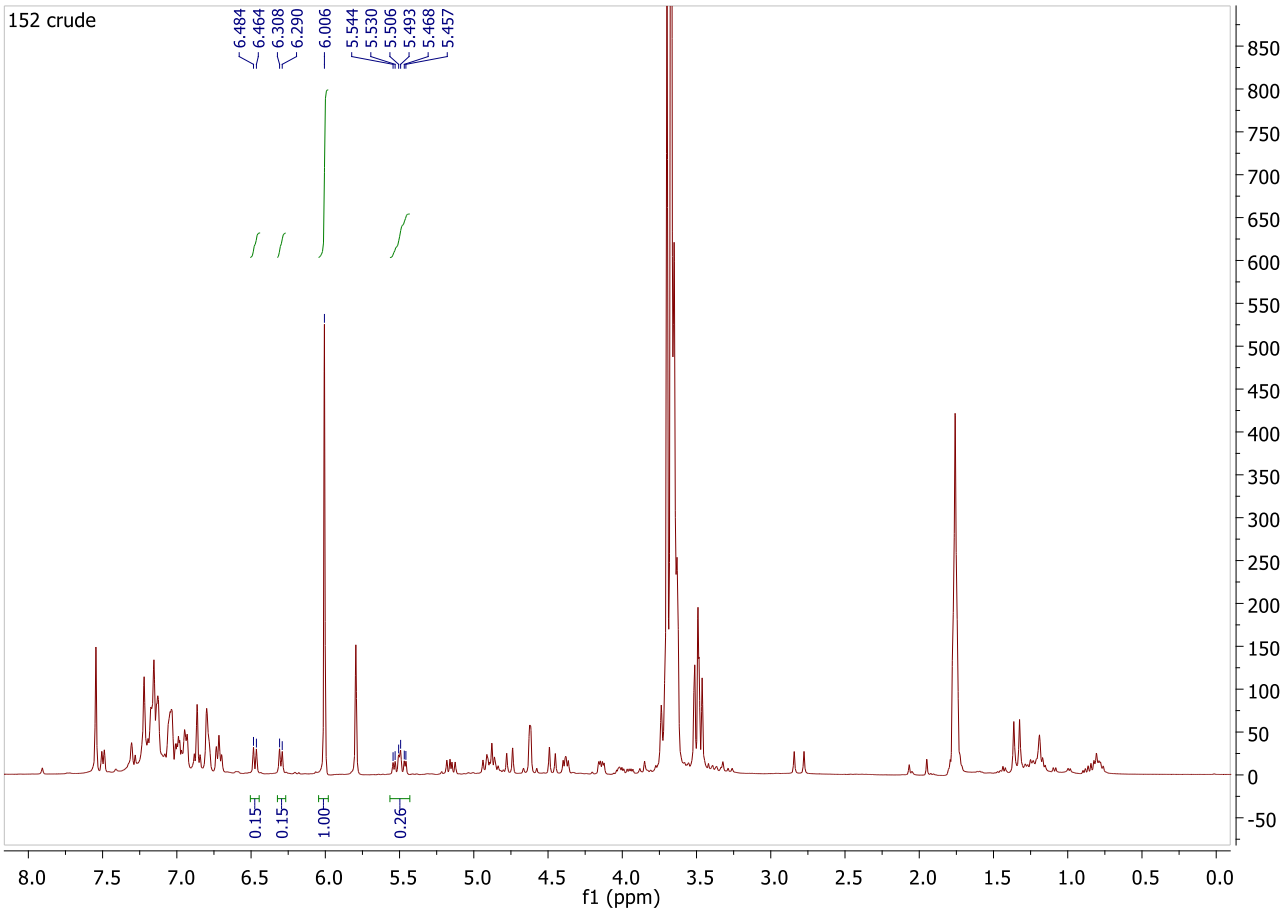
^{1}H NMR of 4af


O
MeO
P
MeO
O
Br
NO2
O
N
Bn

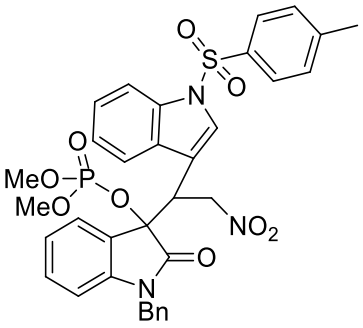

159
6.615
6.595
6.390
6.370
6.018
0.13
0.11
1.00
f1 (ppm)

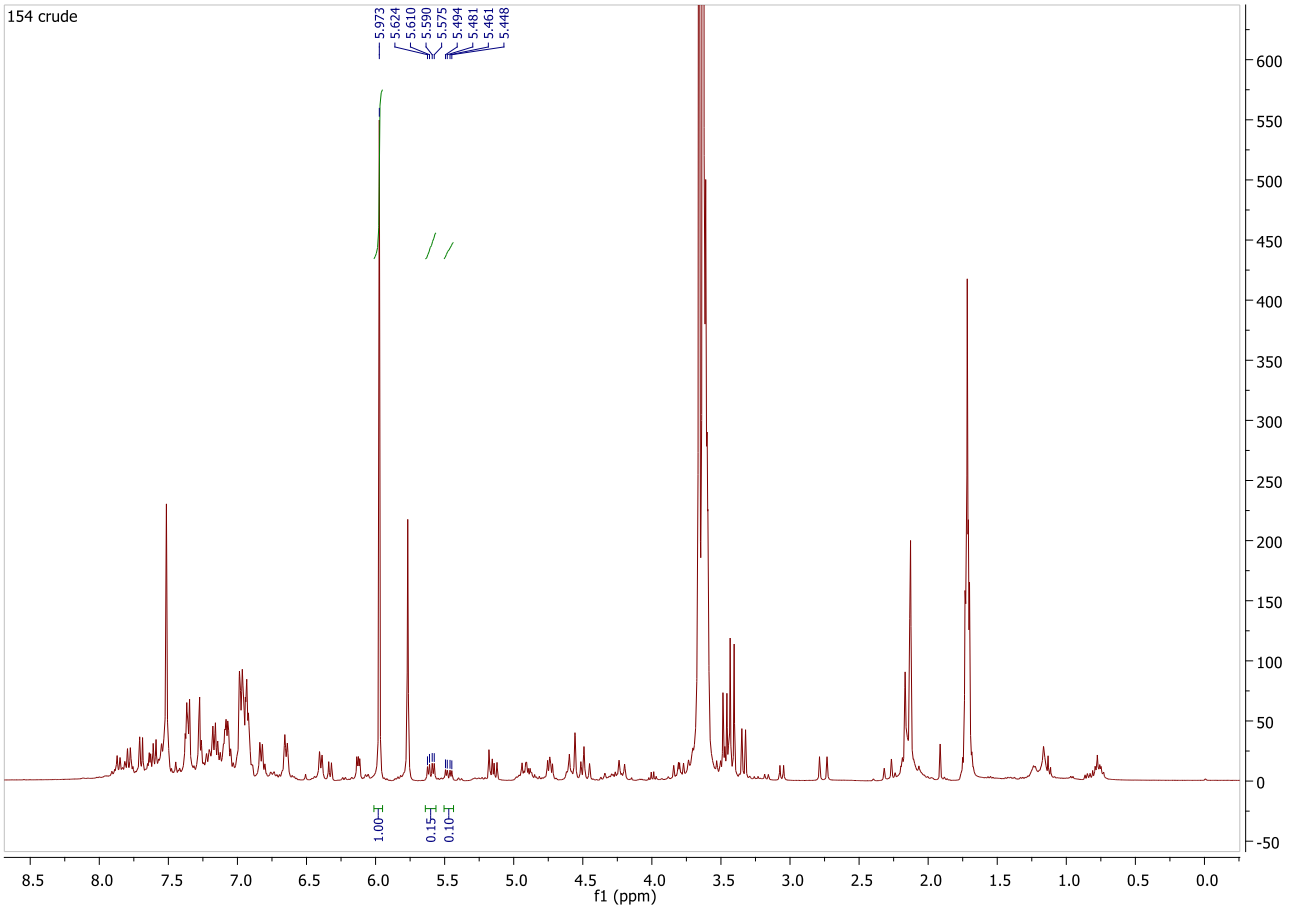
^{1}H NMR of 4ag


Cl
O
MeO
P
MeO
O
NO2
O
N
Bn

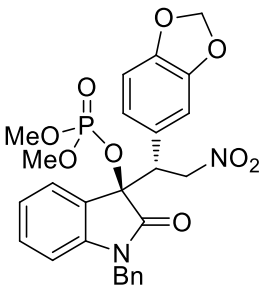

152 crude
6.484
6.464
6.308
6.290
6.006
5.544
5.530
5.506
5.493
5.468
5.457
0.15
0.15
1.00
0.26
f1 (ppm)

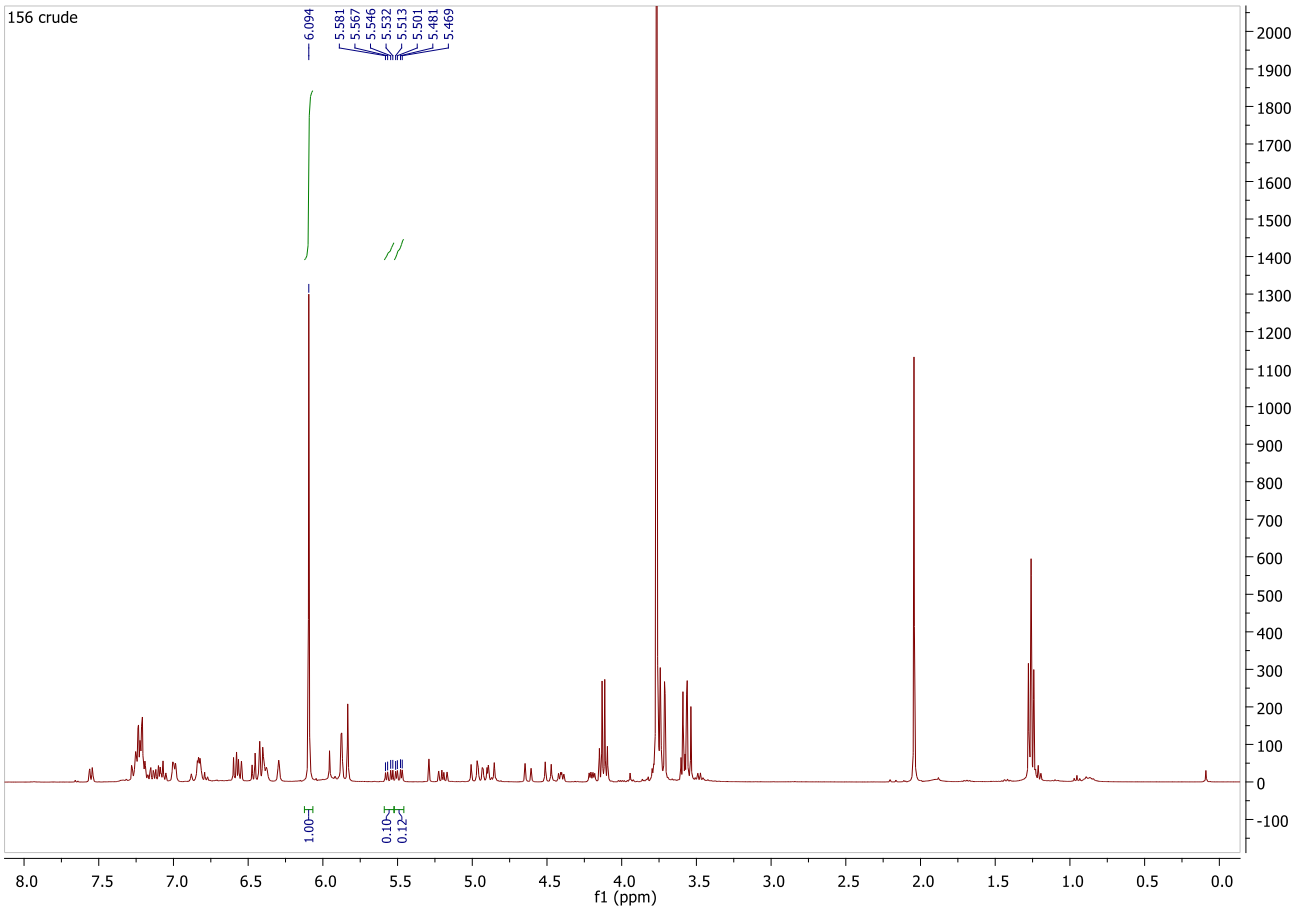
^{1}H NMR of 4ah


O
S
O
N
O
MeO
P
MeO
O
NO2
O
N
Bn

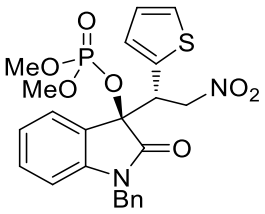

154 crude
5.973
5.624
5.610
5.590
5.575
5.494
5.481
5.461
5.448
1.00
0.15
0.10
f1 (ppm)

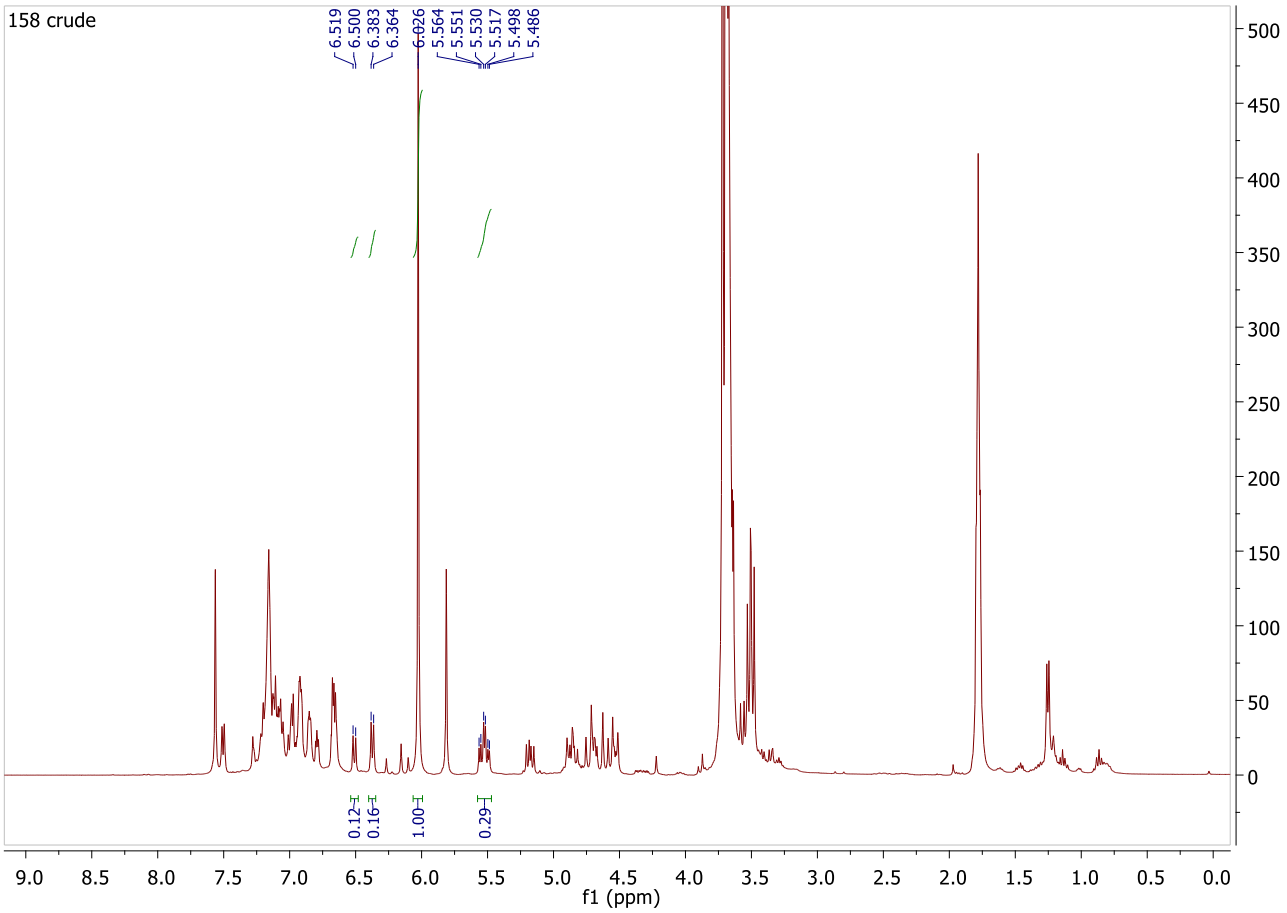
^{1}H NMR of 4ai


MeO
MeO
O
O
O
O
NO2
O
N
Bn

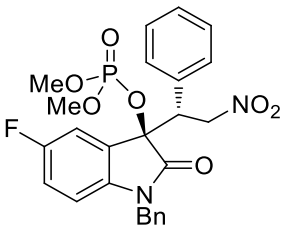

156 crude
6.094
5.581
5.567
5.546
5.532
5.513
5.501
5.481
5.469
1.00
0.10
0.12
f1 (ppm)

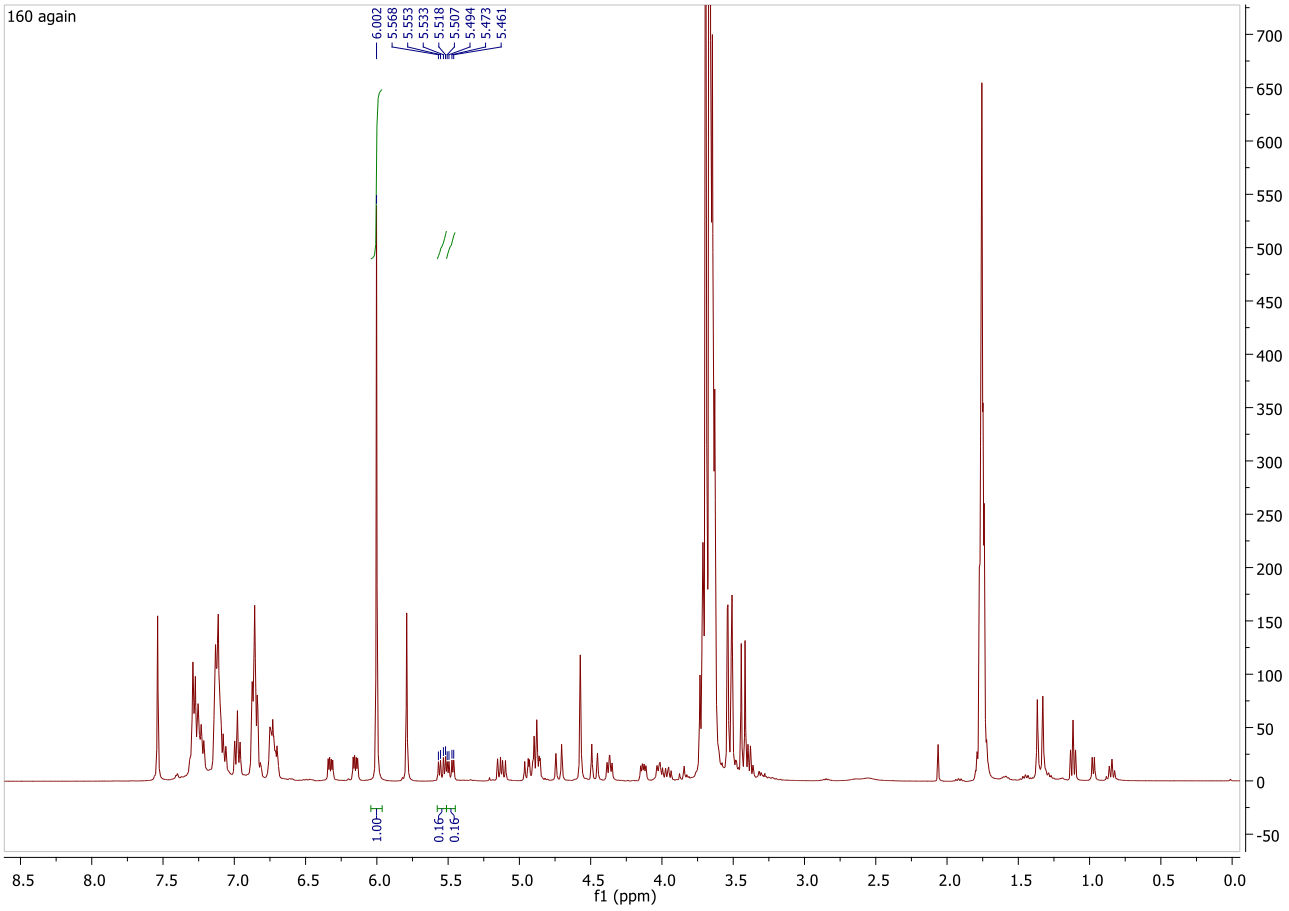
^{1}H NMR of 4aj


O
MeO
P
MeO
O
S
NO2
O
N
Bn

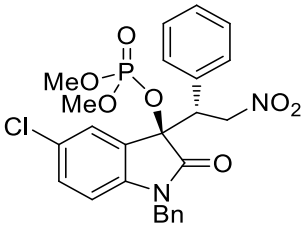

158 crude
6.519
6.500
6.383
6.364
6.026
5.564
5.551
5.530
5.517
5.498
5.486
0.12
0.16
1.00
0.29
f1 (ppm)

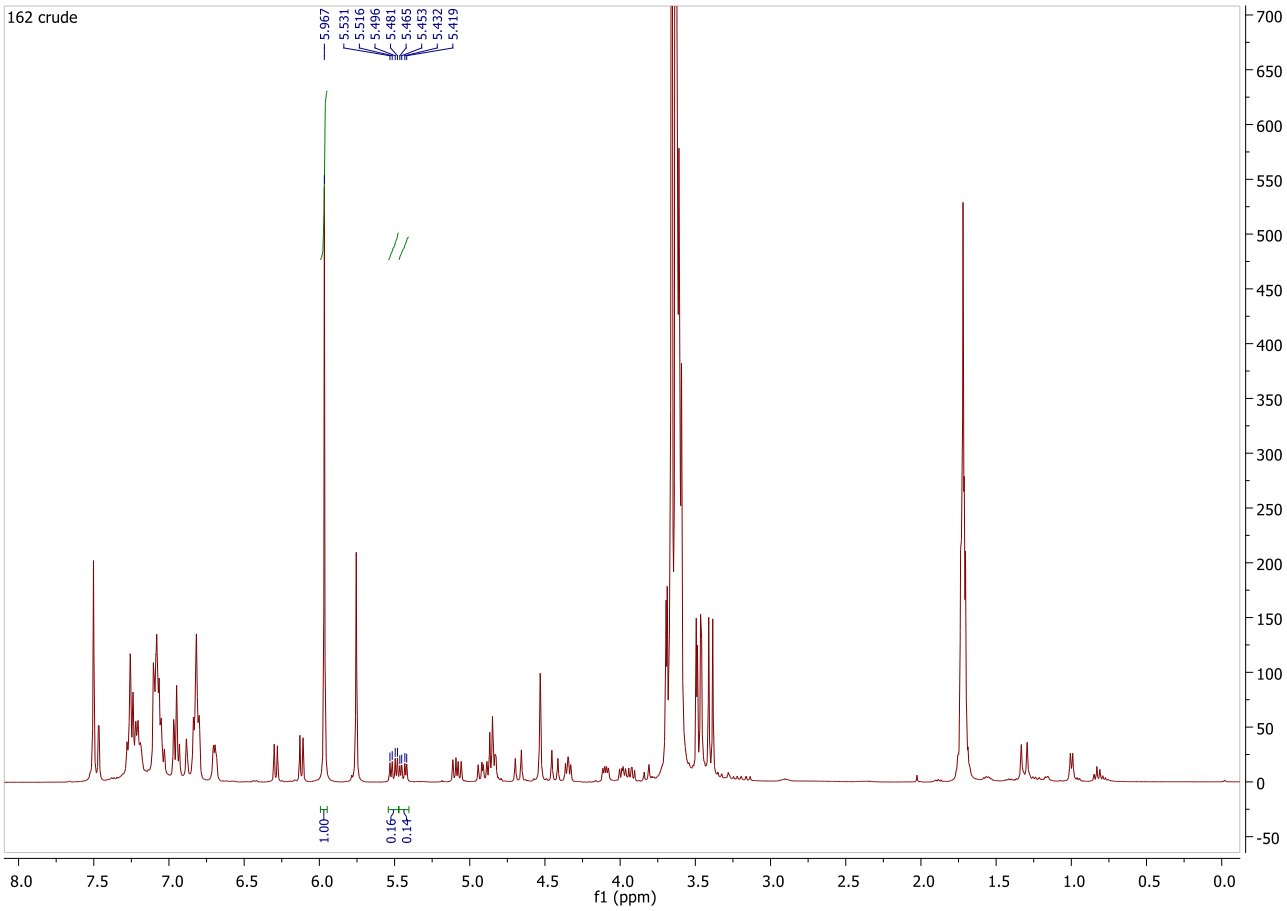
^{1}H NMR of 4ak


O
MeO
P
MeO
O
NO2
F
O
N
Bn

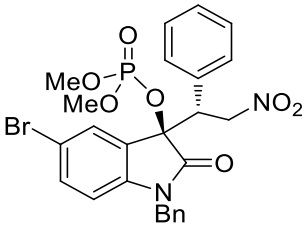

160 again
6.002
5.568
5.553
5.533
5.518
5.507
5.494
5.473
5.461
1.00
0.16
0.16
f1 (ppm)

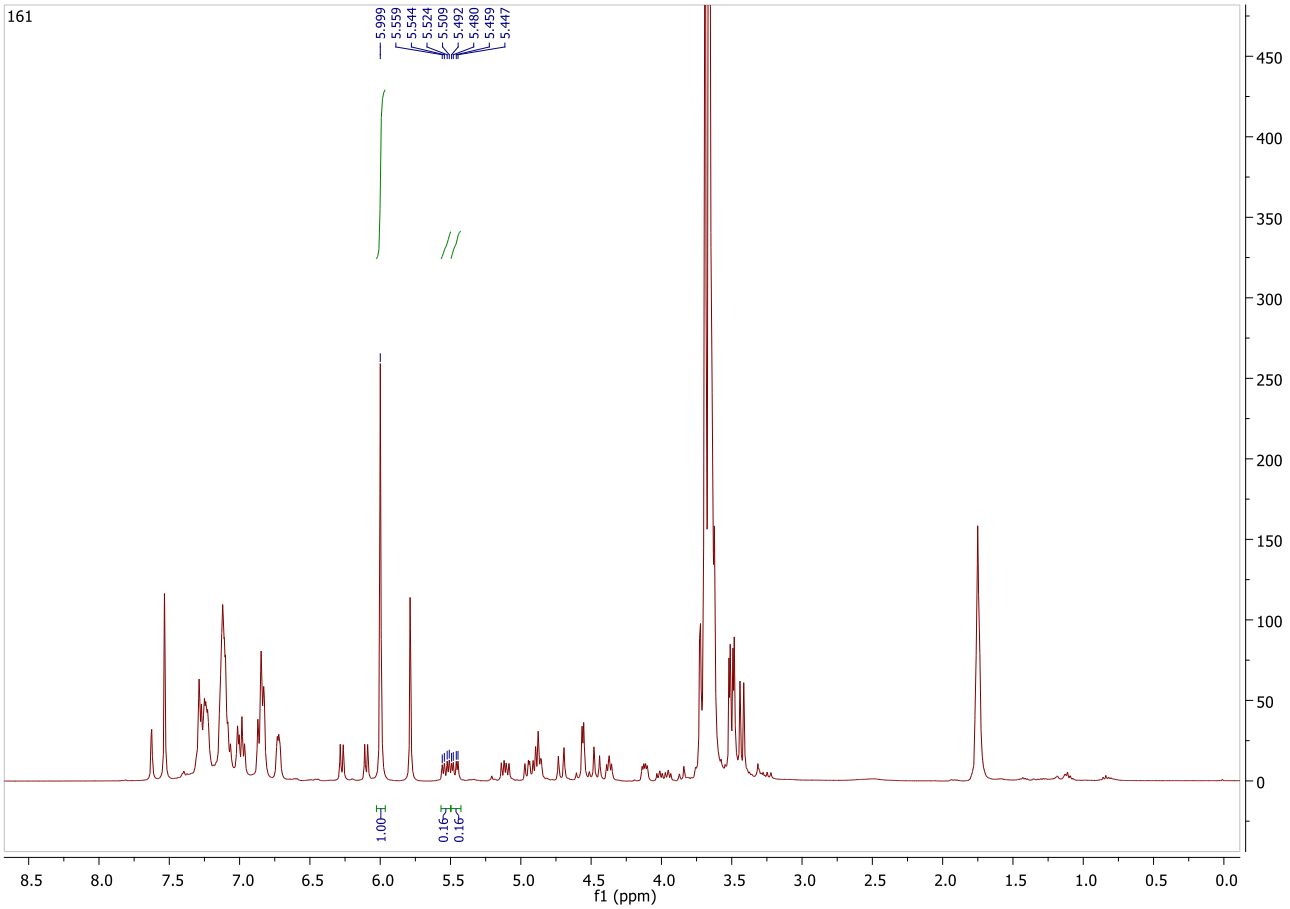
^{1}H NMR of 4ba


O
MeO
P
MeO
O
NO2
Cl
O
N
Bn

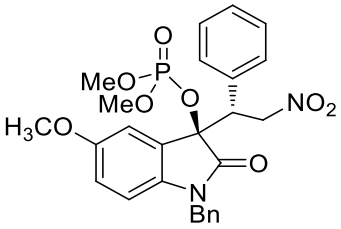

162 crude
5.967
5.531
5.516
5.496
5.481
5.465
5.453
5.432
5.419
1.00
0.16
0.14
f1 (ppm)

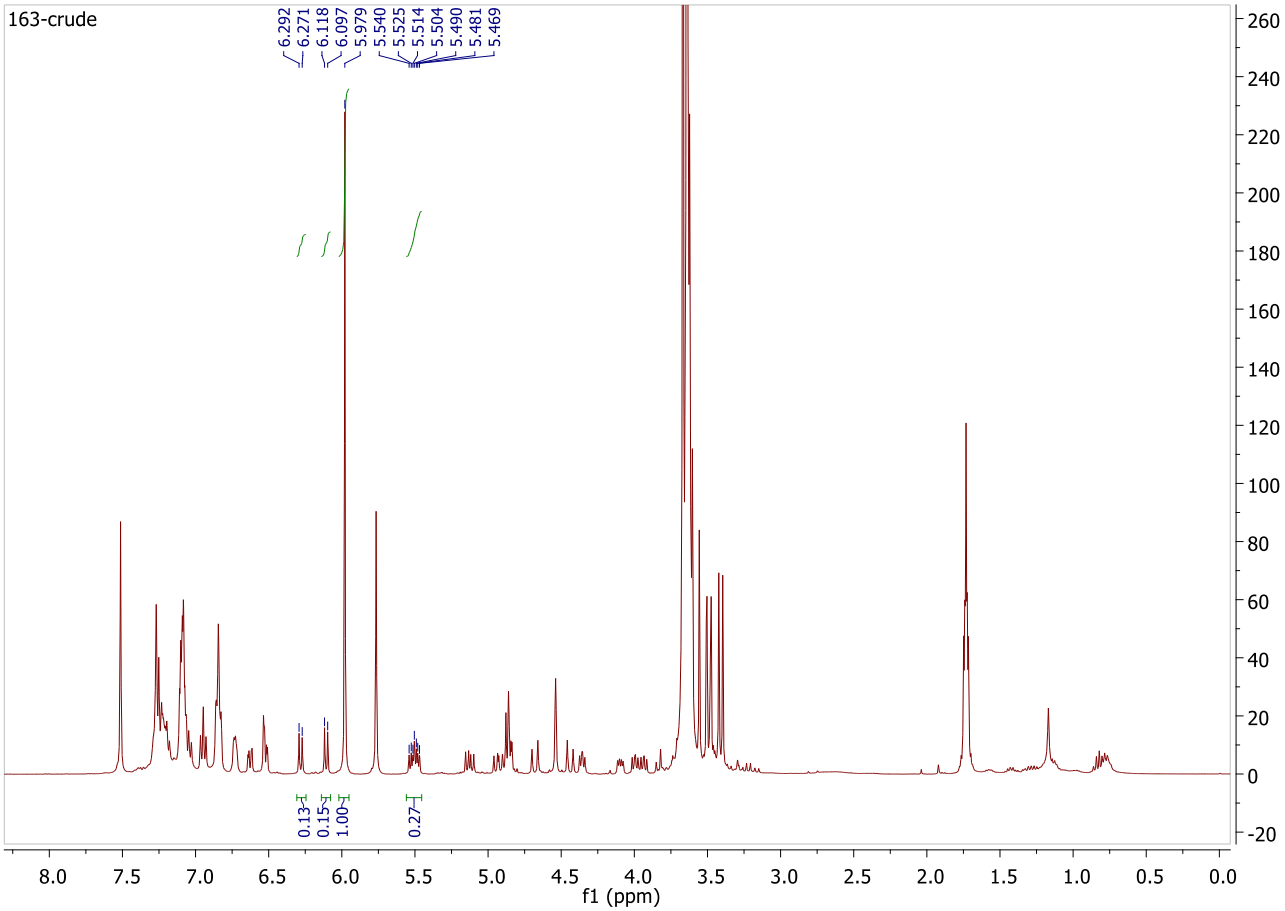
^{1}H NMR of 4ca


O
MeO
P
MeO
O
NO2
Br
O
N
Bn

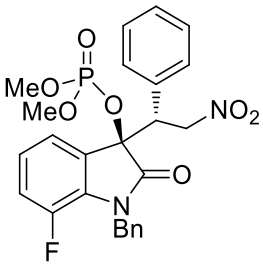

161
5.999
5.559
5.544
5.524
5.509
5.492
5.480
5.459
5.447
1.00
0.16
0.16
f1 (ppm)

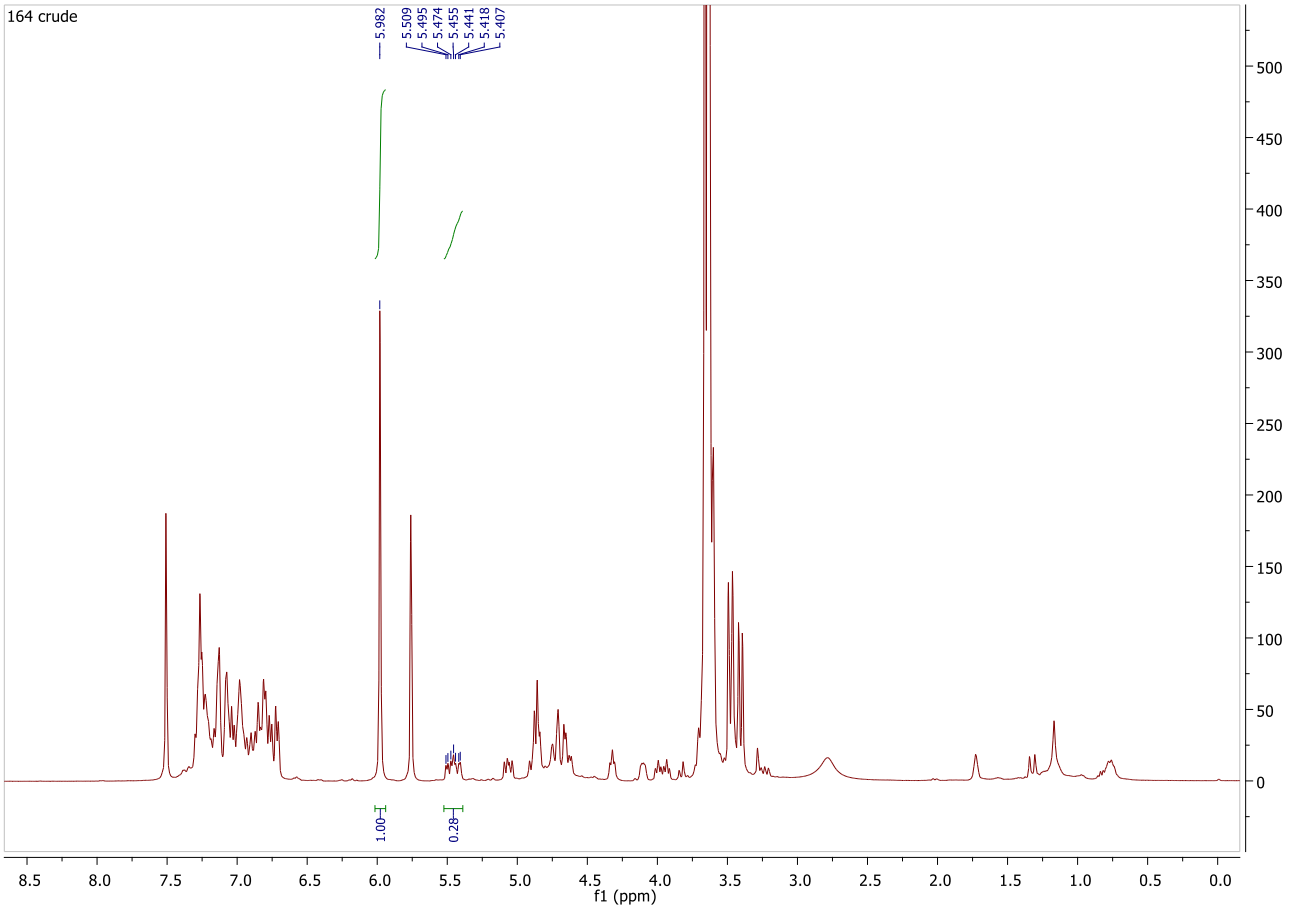
^{1}H NMR of 4da


O
MeO
P
MeO
O
NO2
H3CO
O
N
Bn

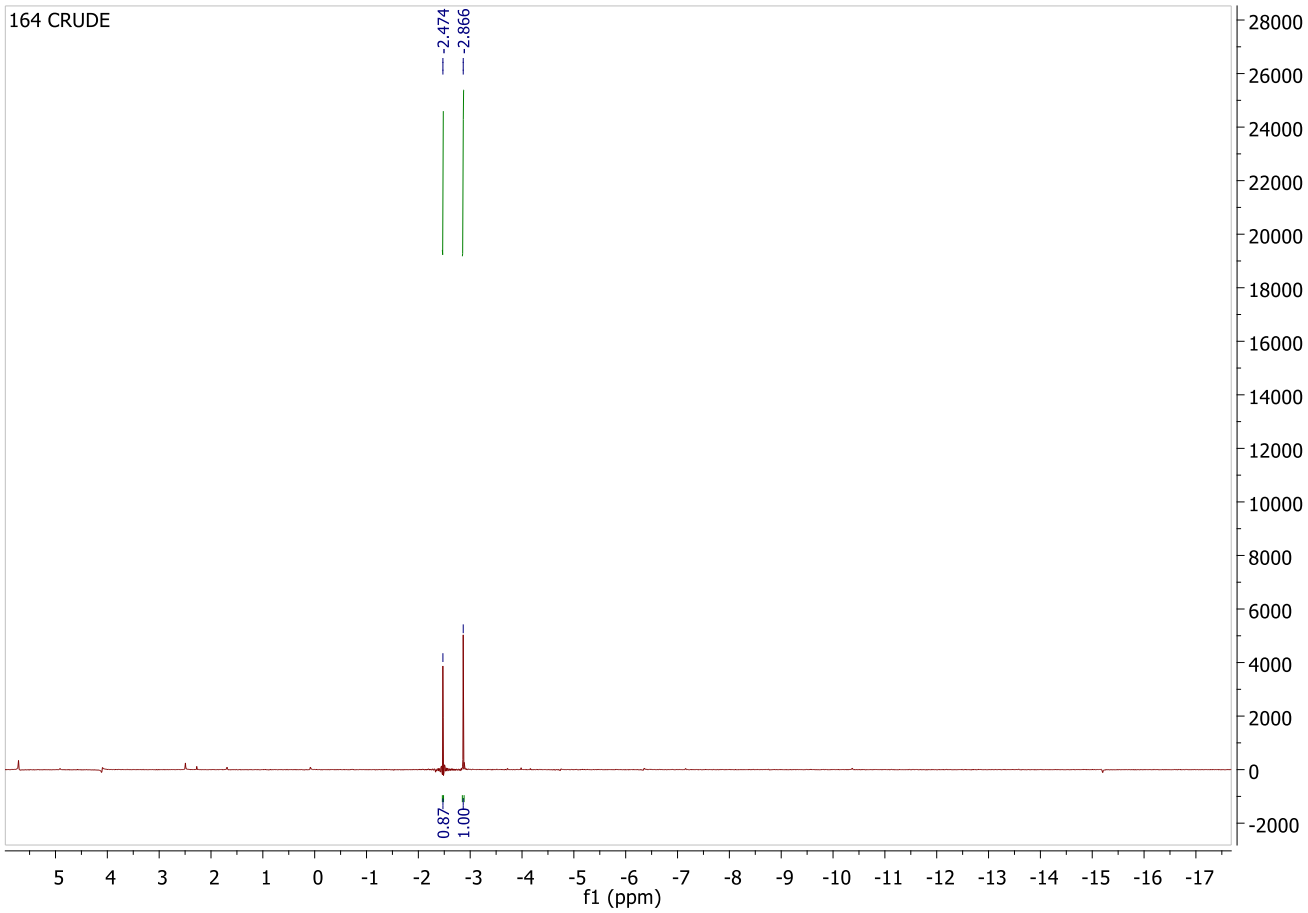

163-crude
6.292
6.271
6.118
6.097
5.979
5.540
5.525
5.514
5.504
5.490
5.481
5.469
0.13
0.15
1.00
0.27
f1 (ppm)

^{1}H NMR of 4ea

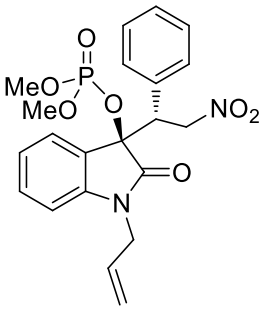

O
MeO
P
MeO
O
NO2
O
N
Bn
F

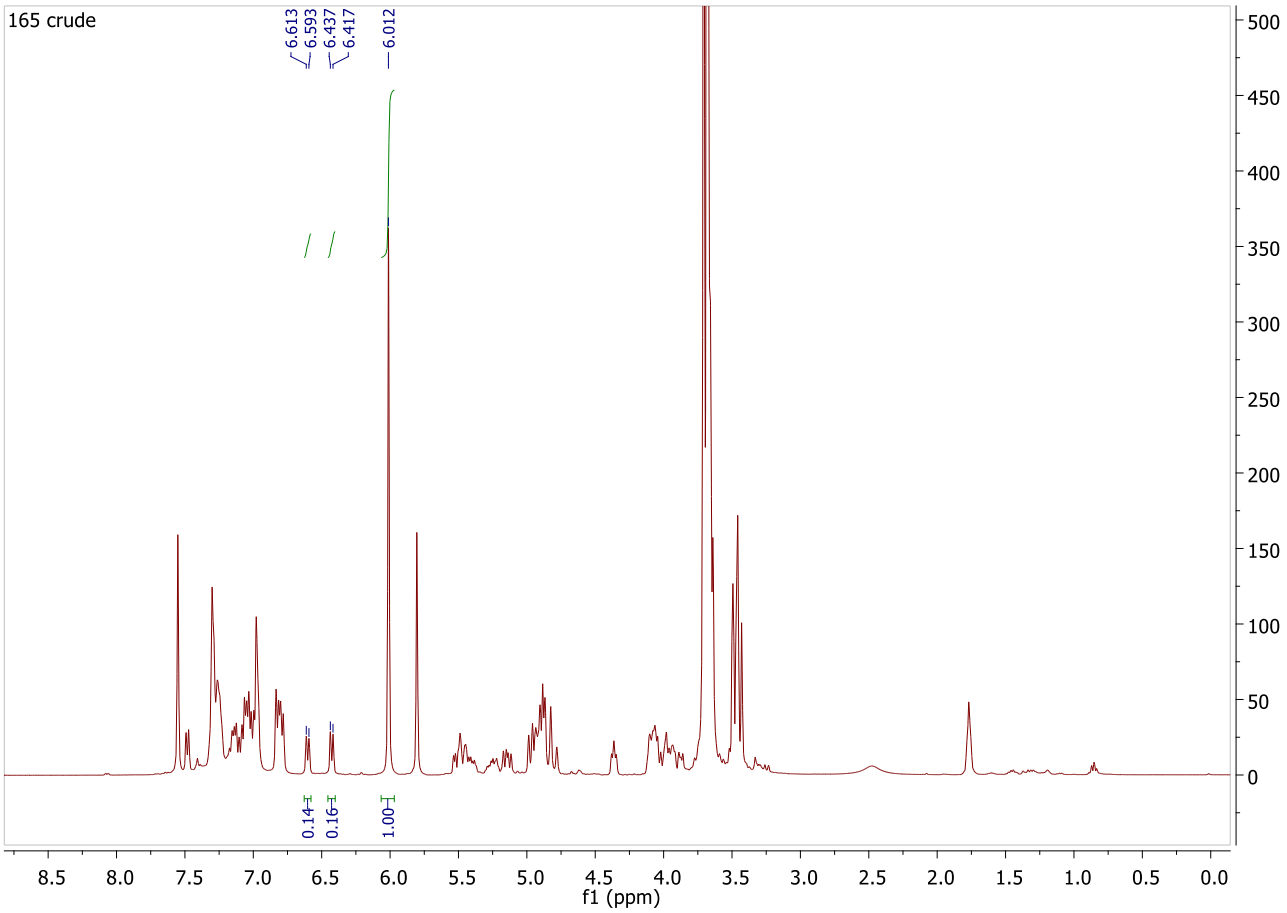

164 crude
5.982
5.509
5.495
5.474
5.455
5.441
5.418
5.407
1.00
0.28
f1 (ppm)

^{1}H NMR of 4fa

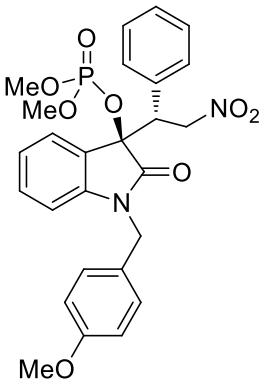


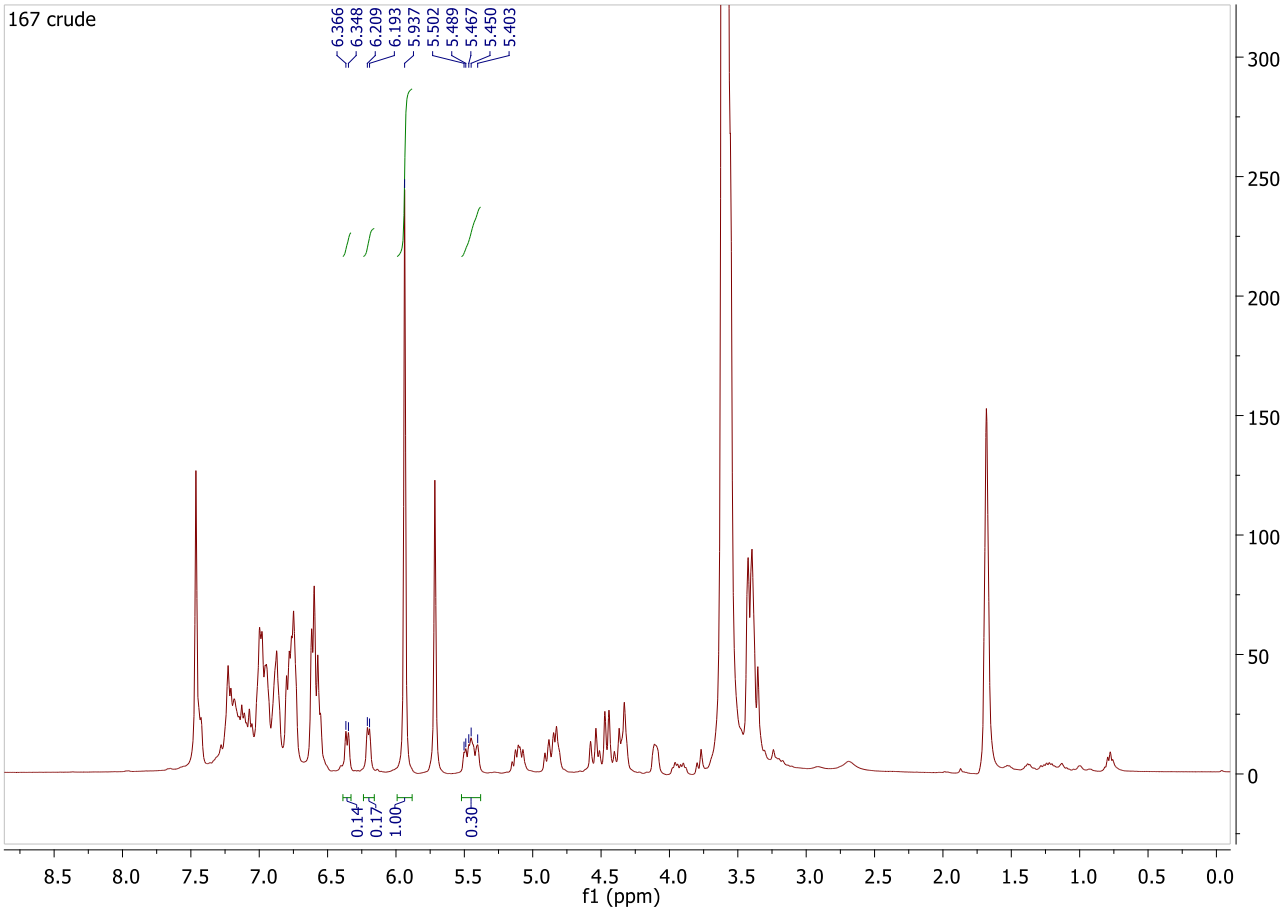
^{31}P NMR of 4fa


O
MeO
P
MeO
O
NO2
O
N

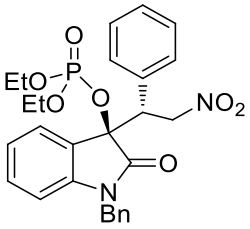

165 crude
6.613
6.593
6.437
6.417
6.012
0.14
0.16
1.00
500
450
400
350
300
250
200
150
100
50
0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

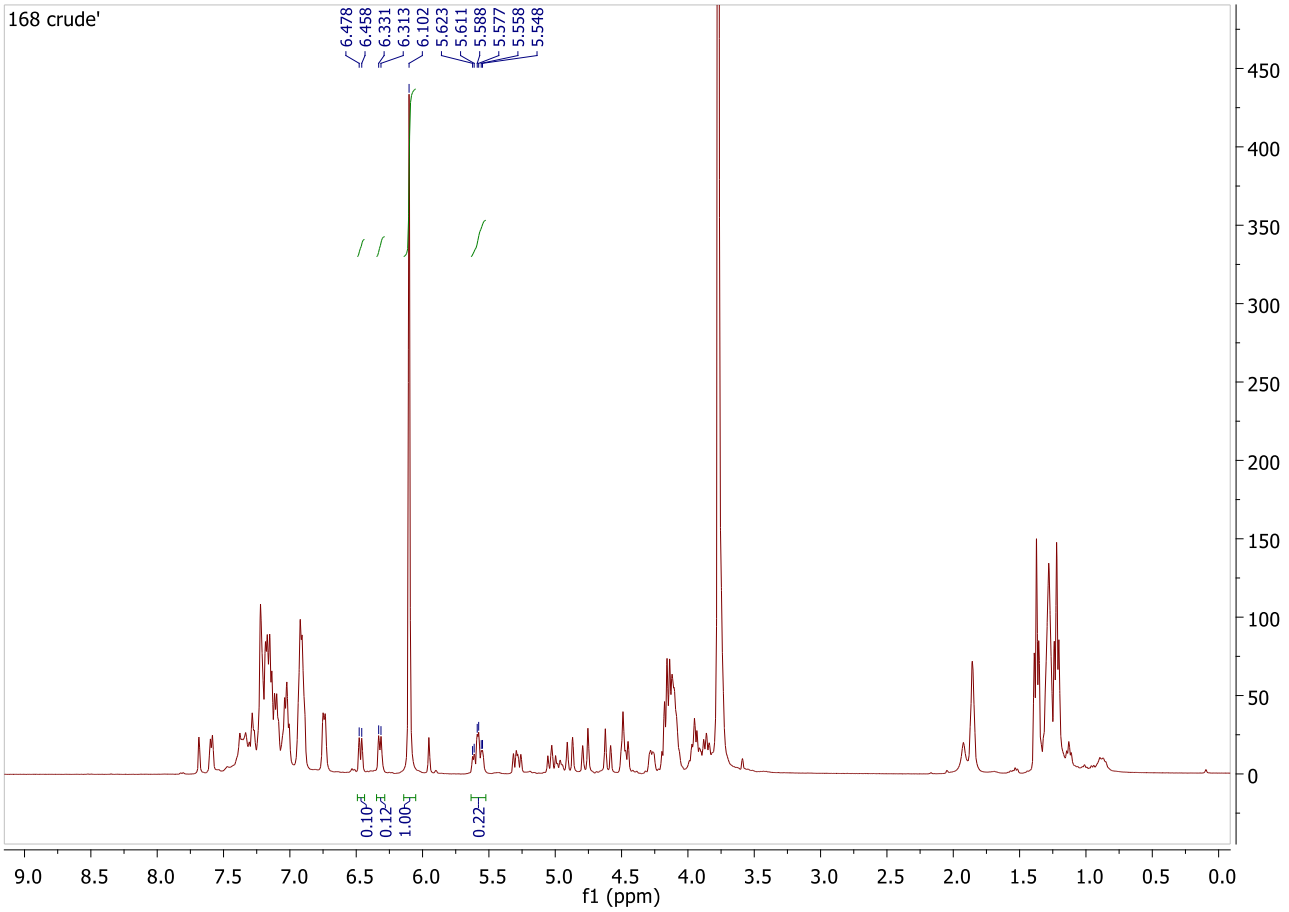
^{1}H NMR of 4ga


O
MeO
P
MeO
O
NO2
O
N
MeO

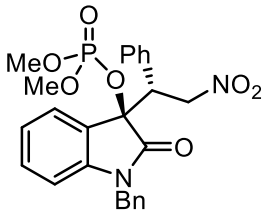

167 crude
6.366
6.348
6.209
6.193
5.937
5.502
5.489
5.467
5.450
5.403
0.14
0.17
1.00
0.30
300
250
200
150
100
50
0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

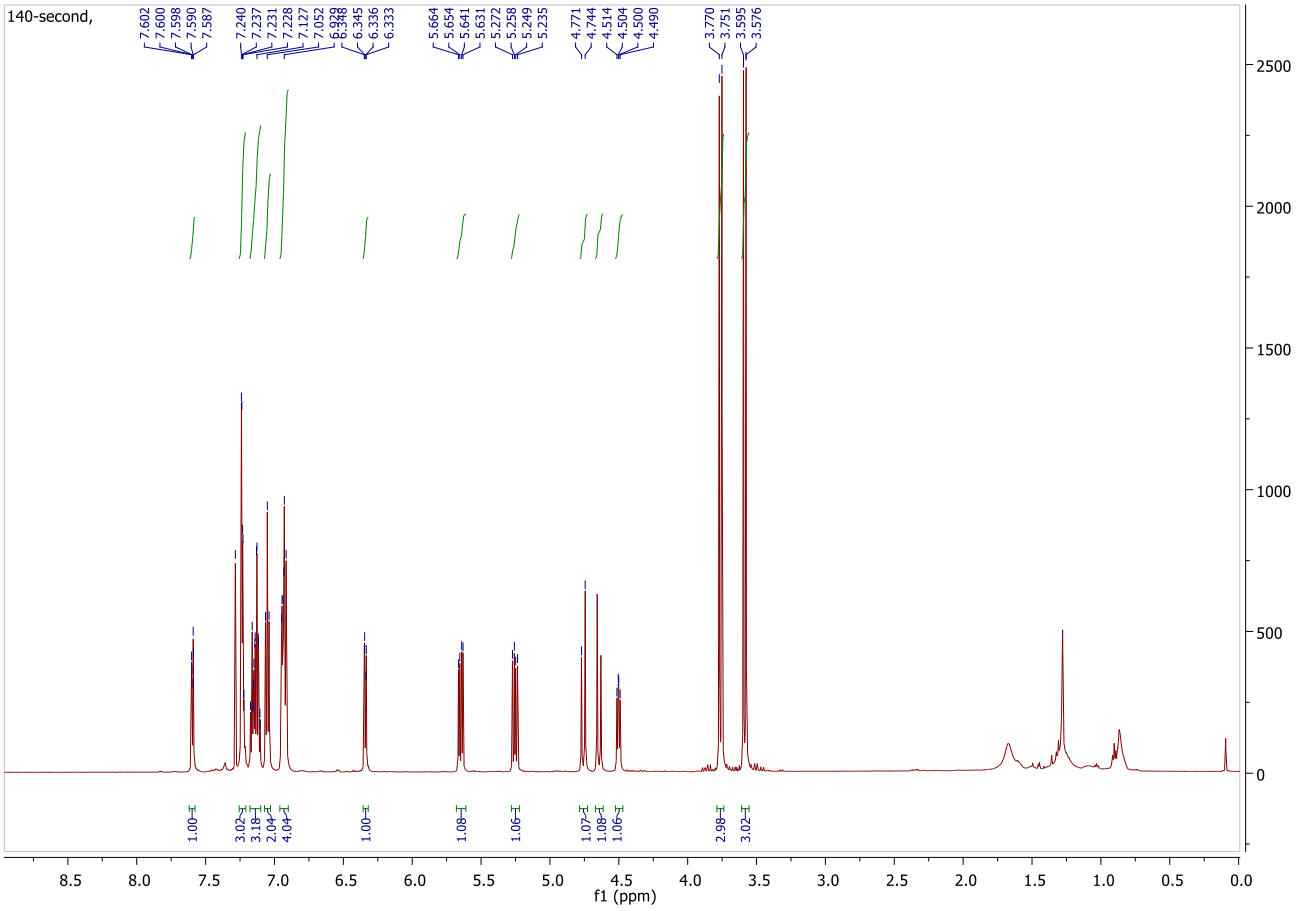
^{1}H NMR of 4ha


O
EtO
P
EtO
O
NO2
O
N
Bn

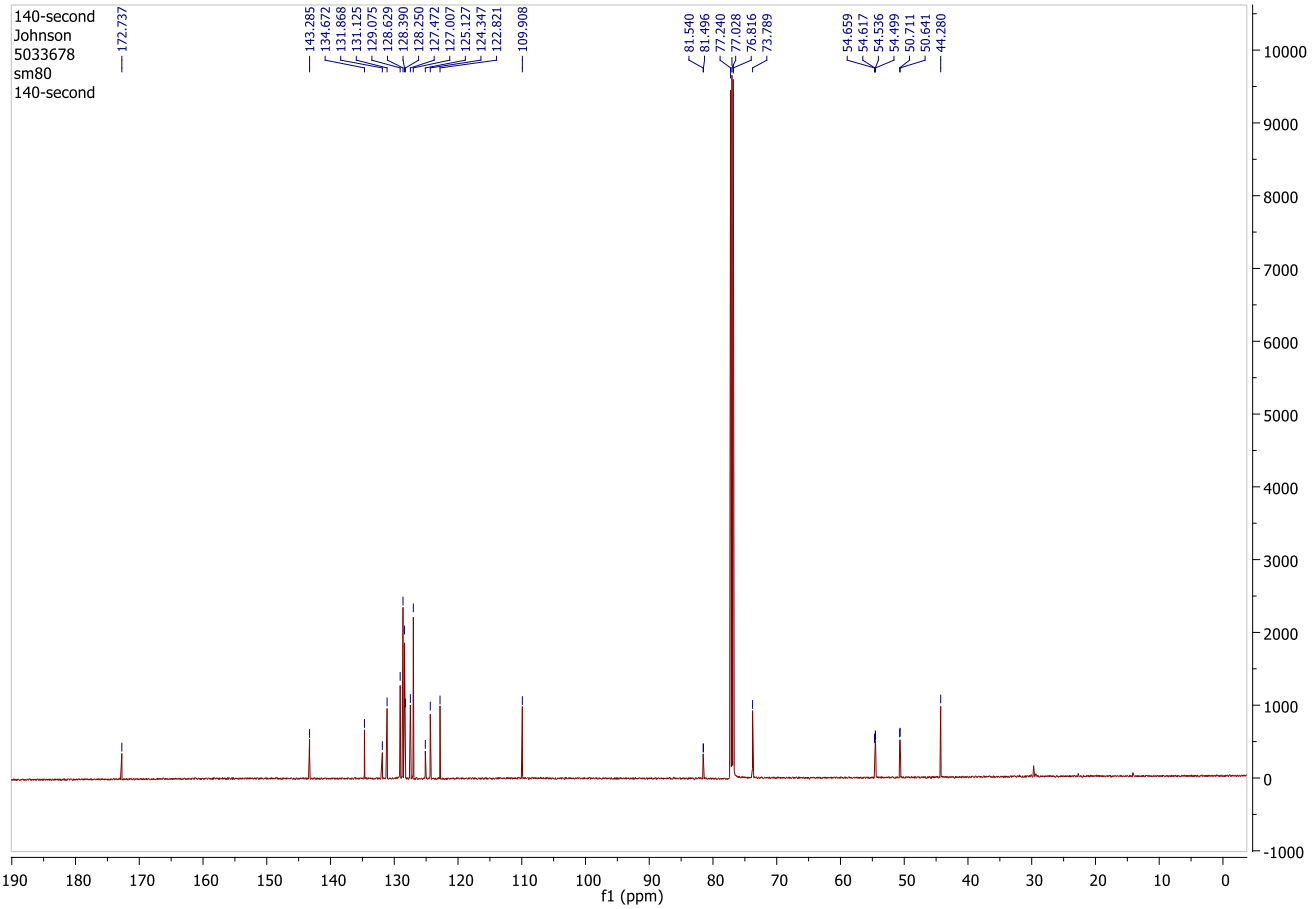

168 crude'
6.478
6.458
6.331
6.313
6.102
5.623
5.611
5.588
5.577
5.558
5.548
0.10
0.12
1.00
0.22
450
400
350
300
250
200
150
100
50
0
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

^{1}H and ^{13}C NMR spectra of new compounds

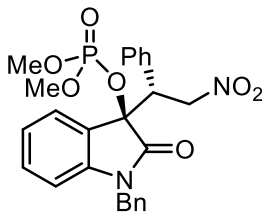


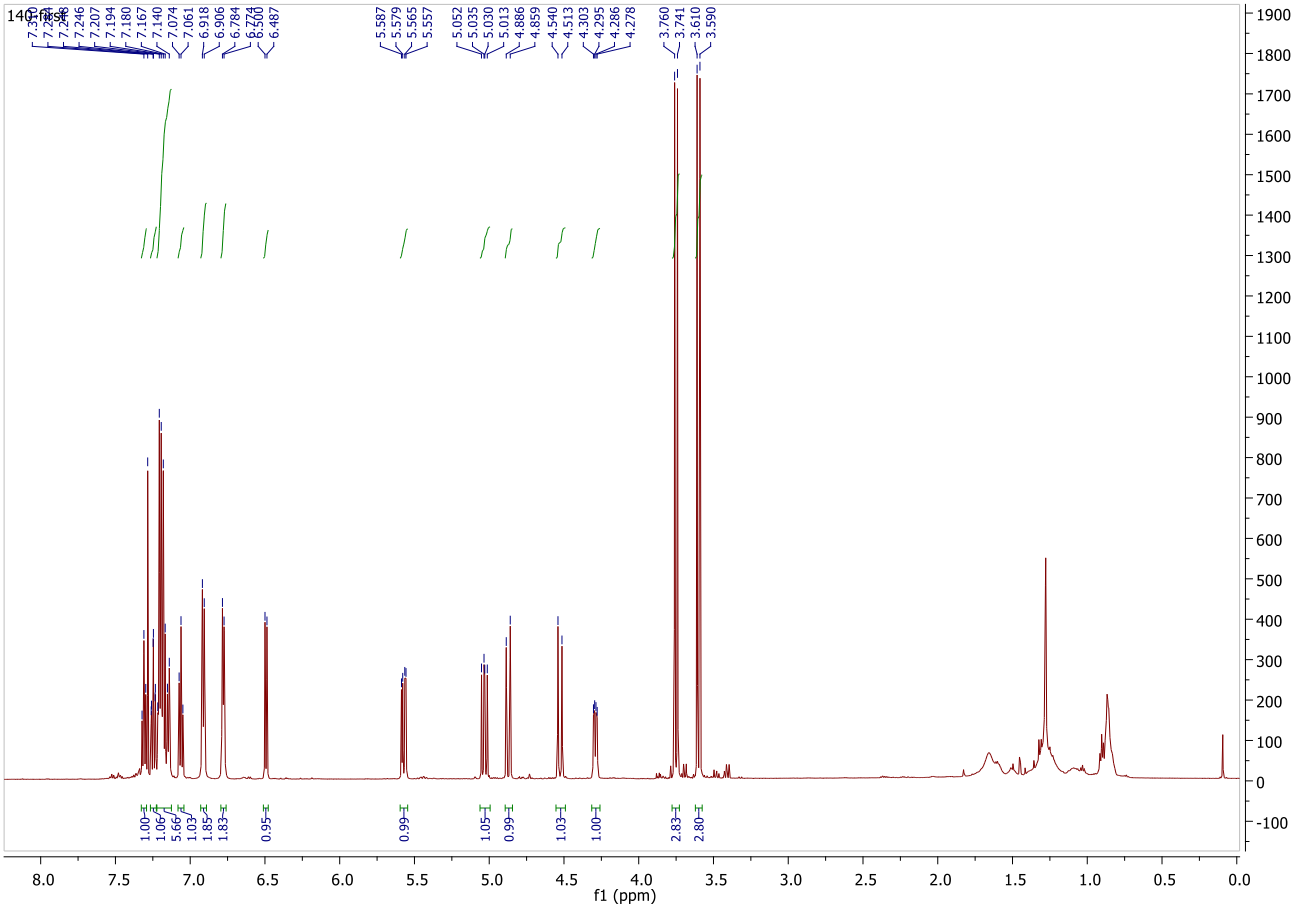


^{1}H NMR of 4aa

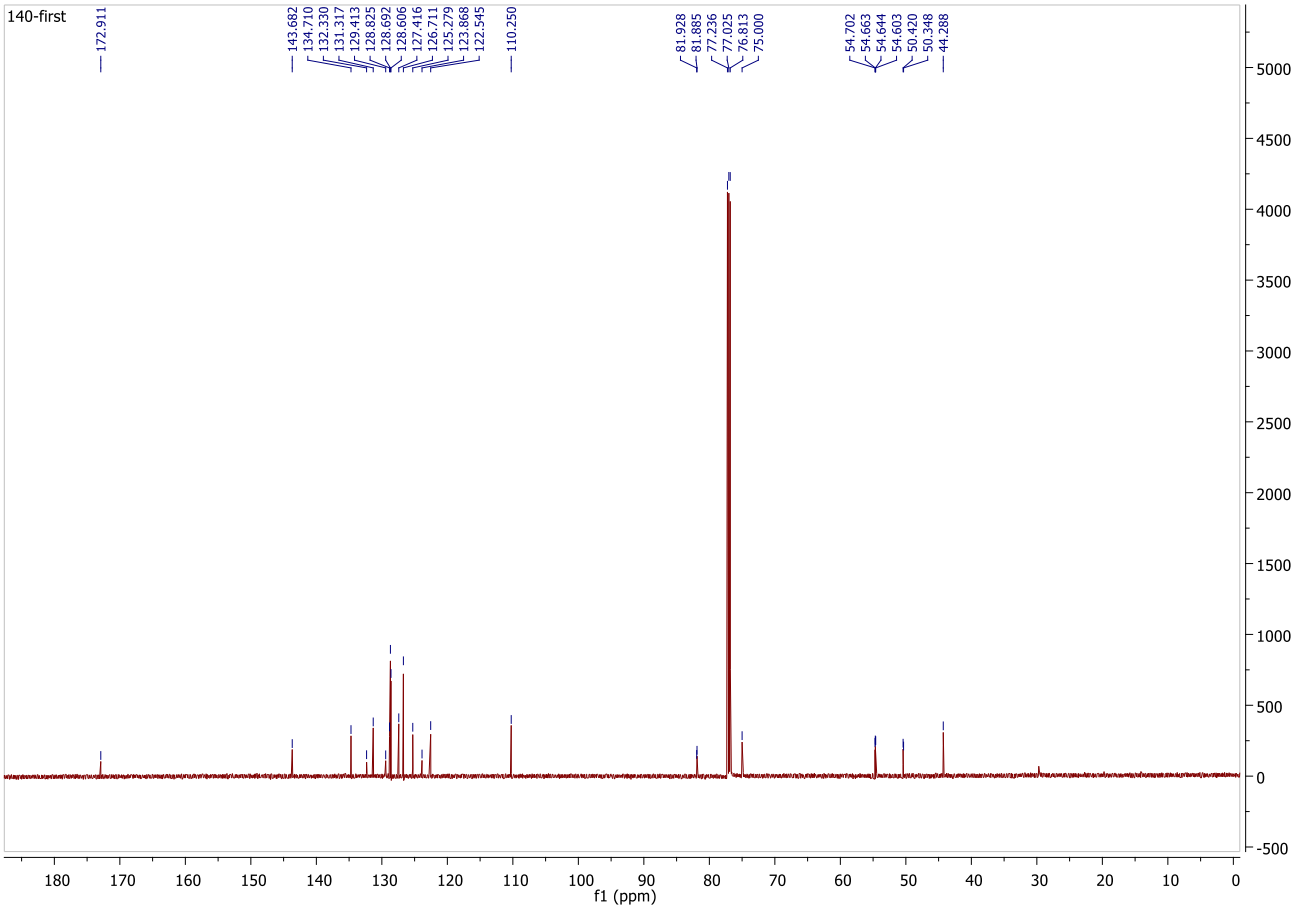


^{13}C NMR of 4aa

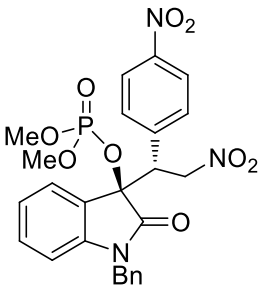


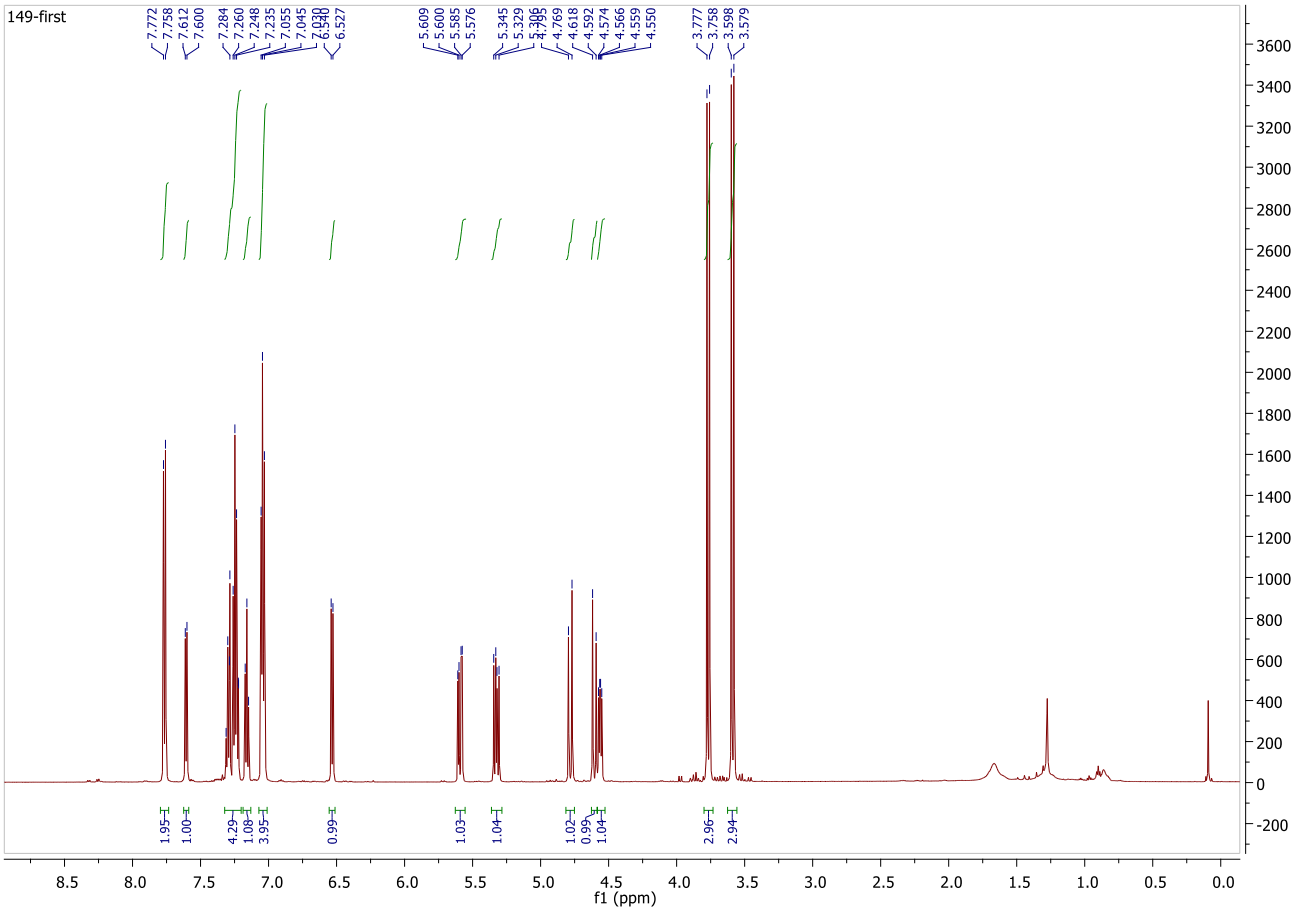


^{1}H NMR of 4aa (2nd diastereomer)

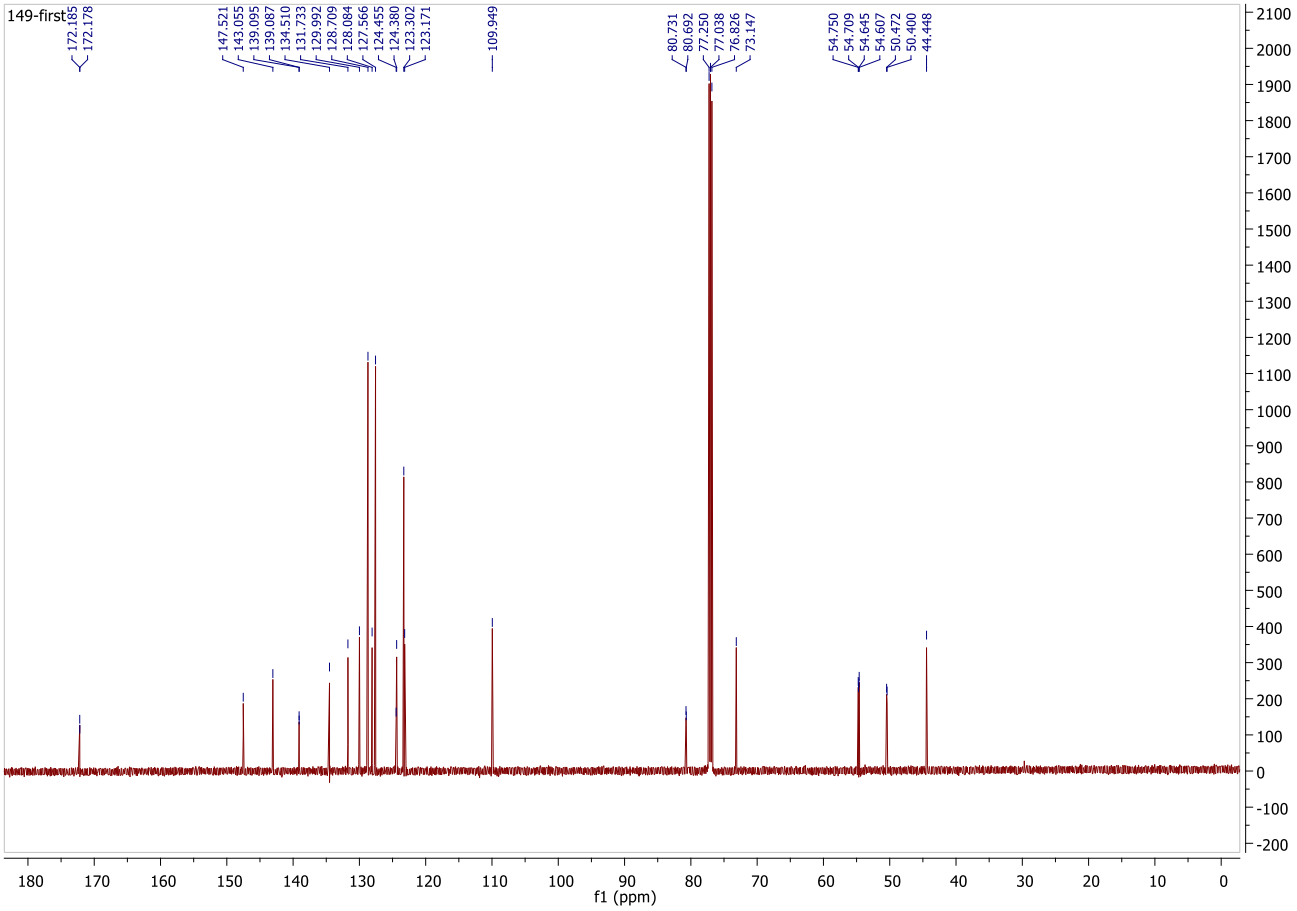


^{13}C NMR of 4aa (2nd diastereomer)

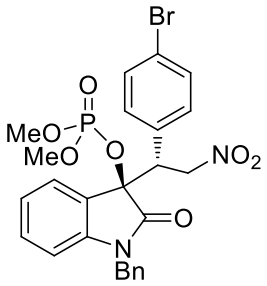

NO2
O
MeO
P
MeO
O
NO2
O
N
Bn

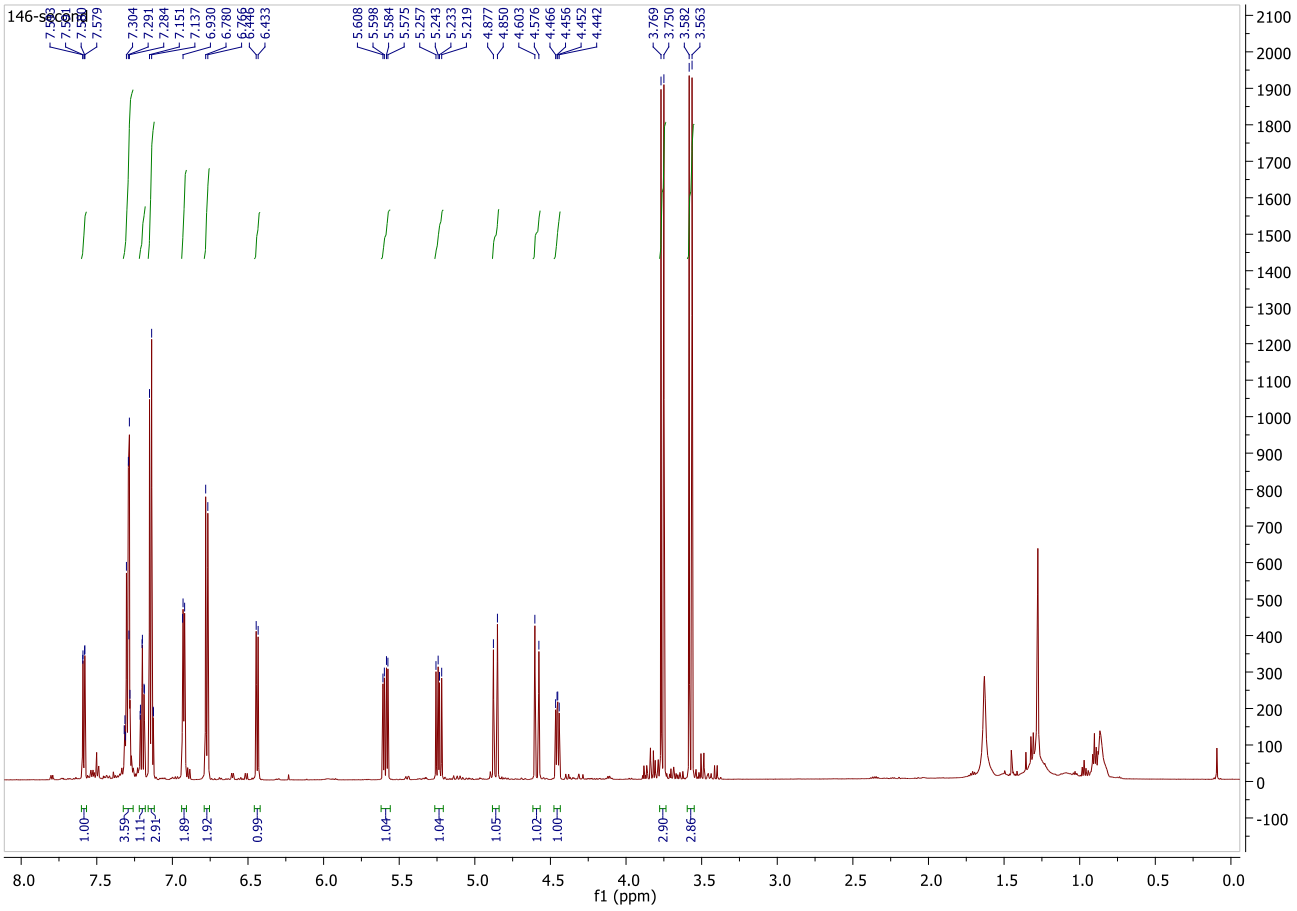

149-first
7.772
7.758
7.612
7.600
7.284
7.260
7.248
7.235
7.055
7.045
6.527
5.609
5.600
5.585
5.576
5.345
5.329
4.769
4.618
4.592
4.574
4.566
4.559
4.550
3.777
3.758
3.598
3.579
1.95
1.00
4.29
1.08
3.95
0.99
1.03
1.04
1.02
0.99
1.04
2.96
2.94
f1 (ppm)

^{1}H NMR of 4ab

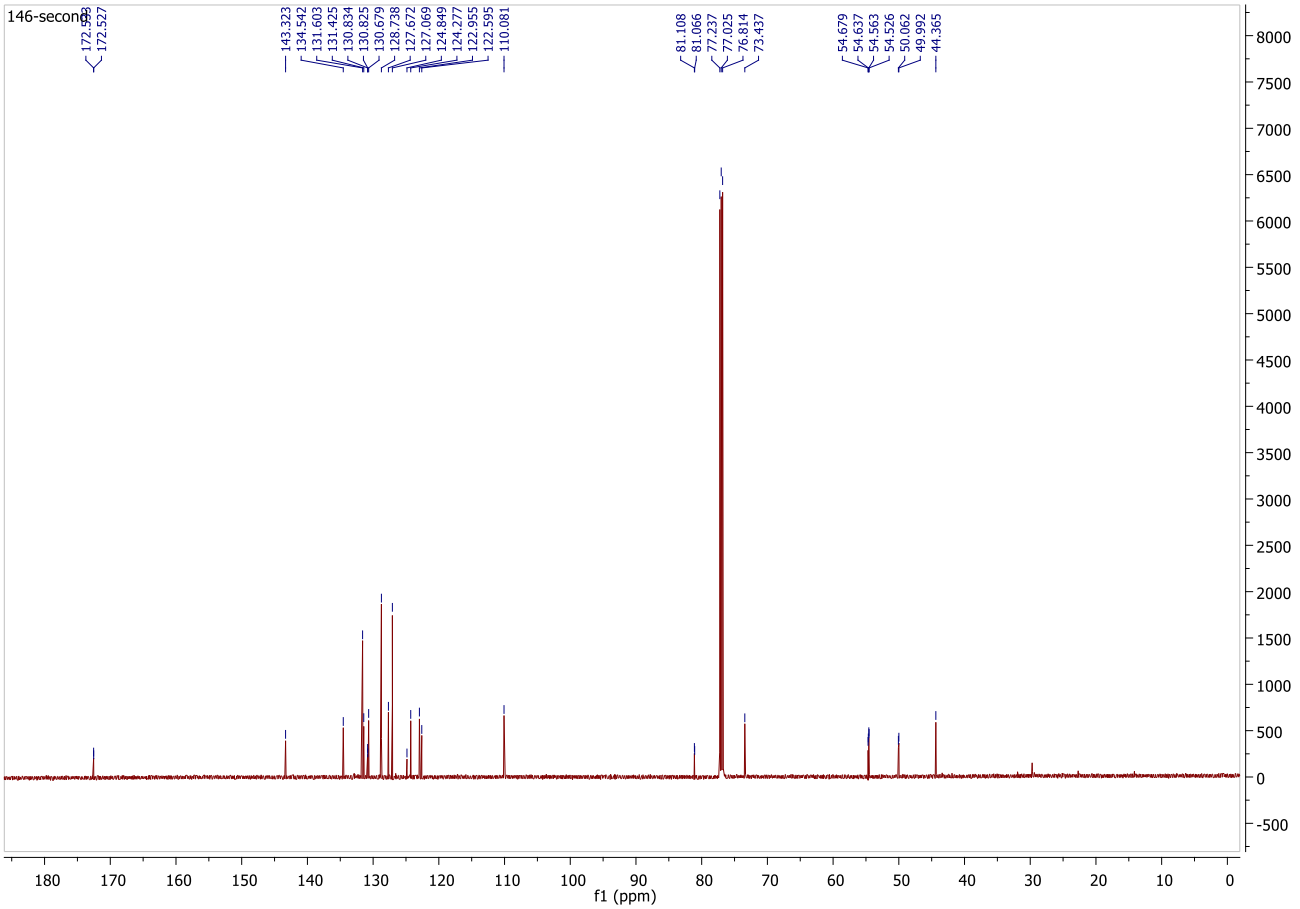


^{13}C NMR of 4ab

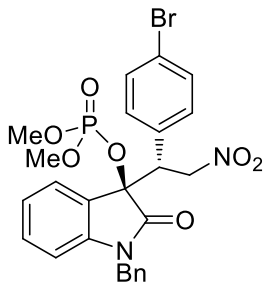

Br
O
MeO
P
MeO
O
NO2
O
N
Bn

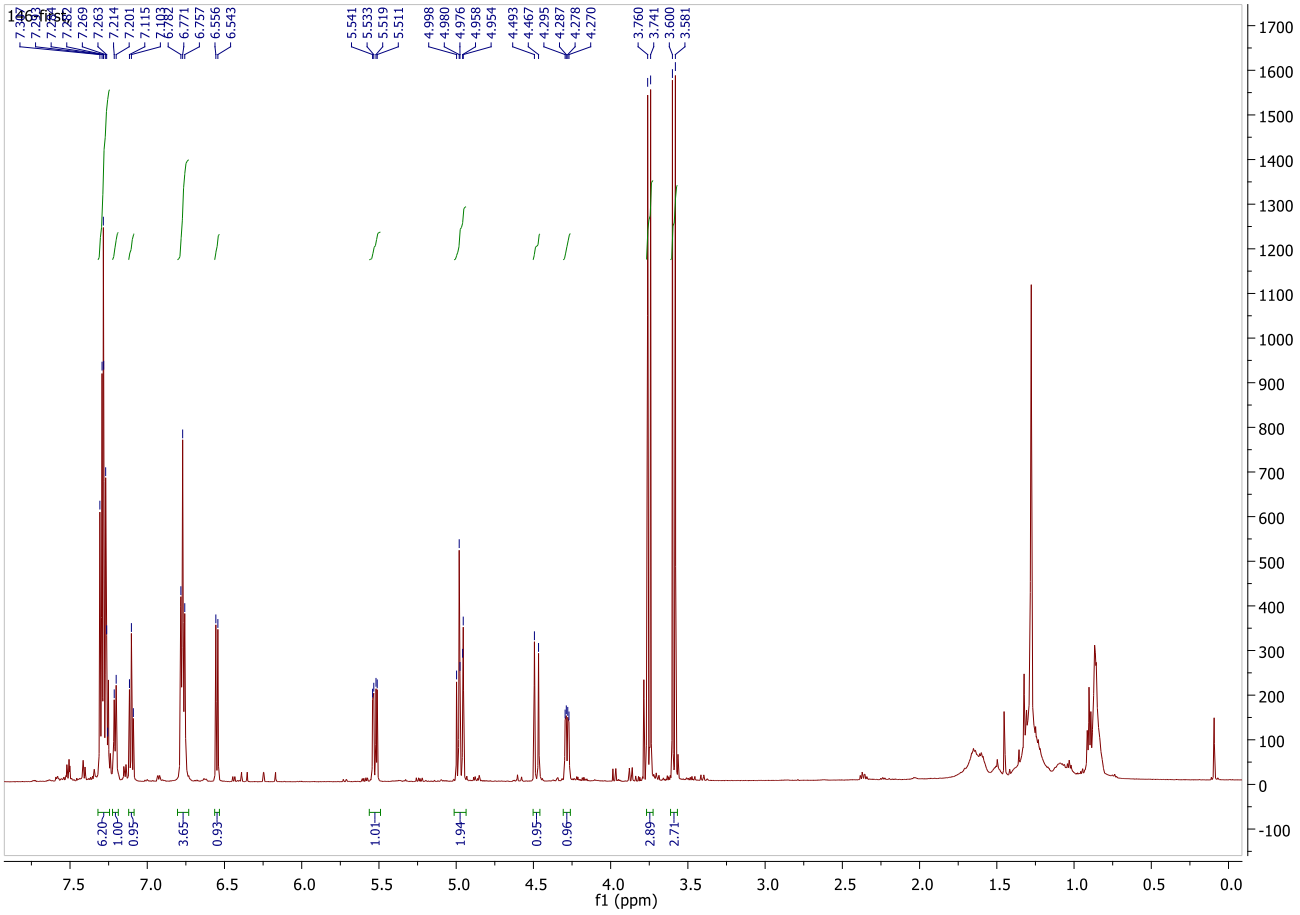

146-sc-cm1
7.579
7.304
7.291
7.284
7.151
7.137
6.930
6.780
6.766
6.448
6.433
5.608
5.598
5.584
5.575
5.257
5.243
5.233
5.219
4.877
4.850
4.603
4.576
4.466
4.456
4.452
4.442
3.769
3.750
3.582
3.563
1.00
3.59
1.11
2.91
1.89
1.92
0.99
1.04
1.04
1.05
1.02
1.00
2.90
2.86
f1 (ppm)

^{1}H NMR of 4ac

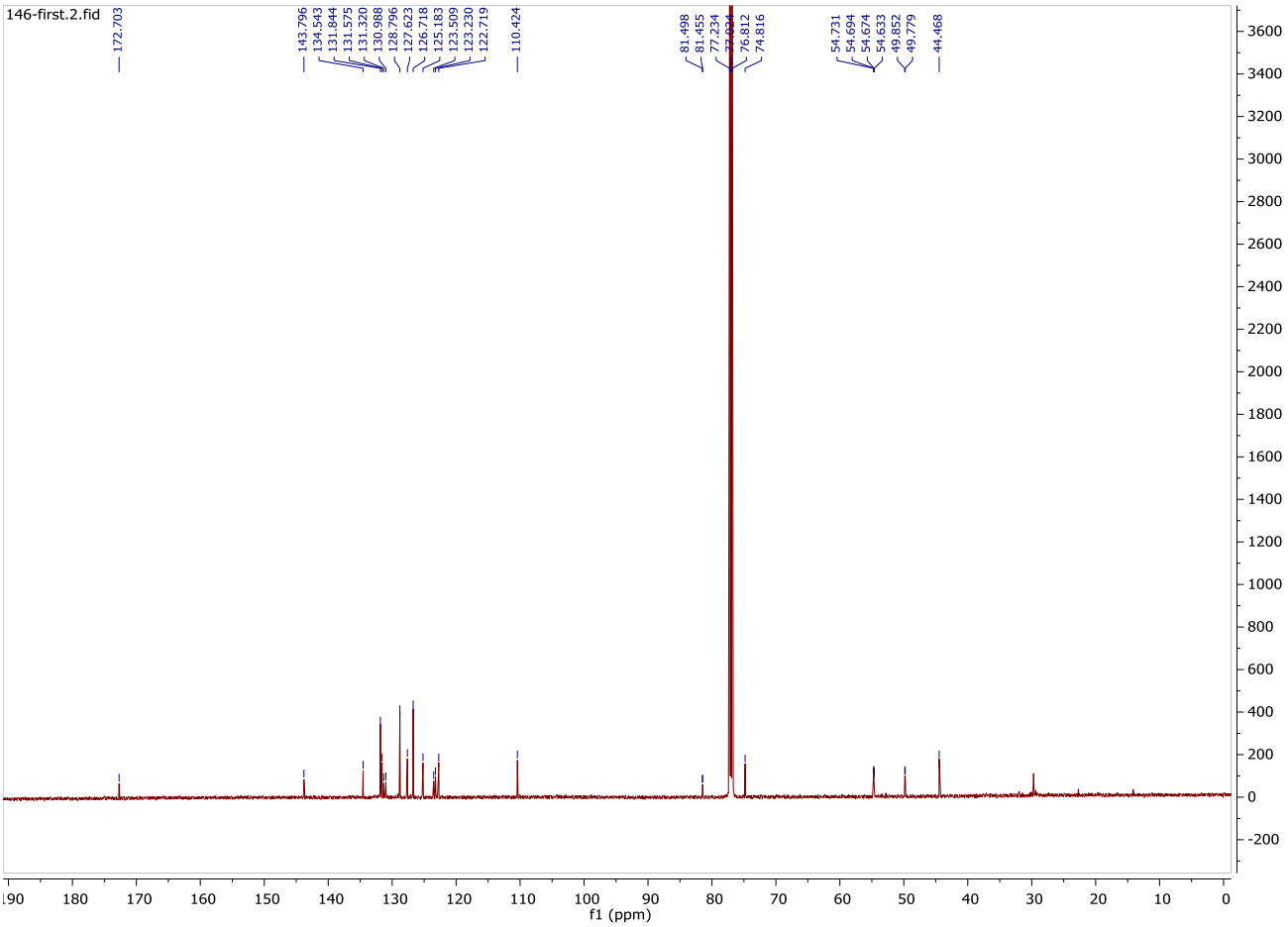


^{13}C NMR of 4ac

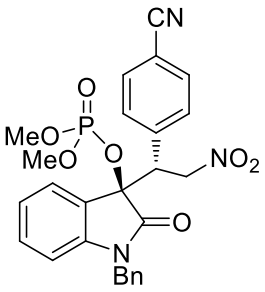


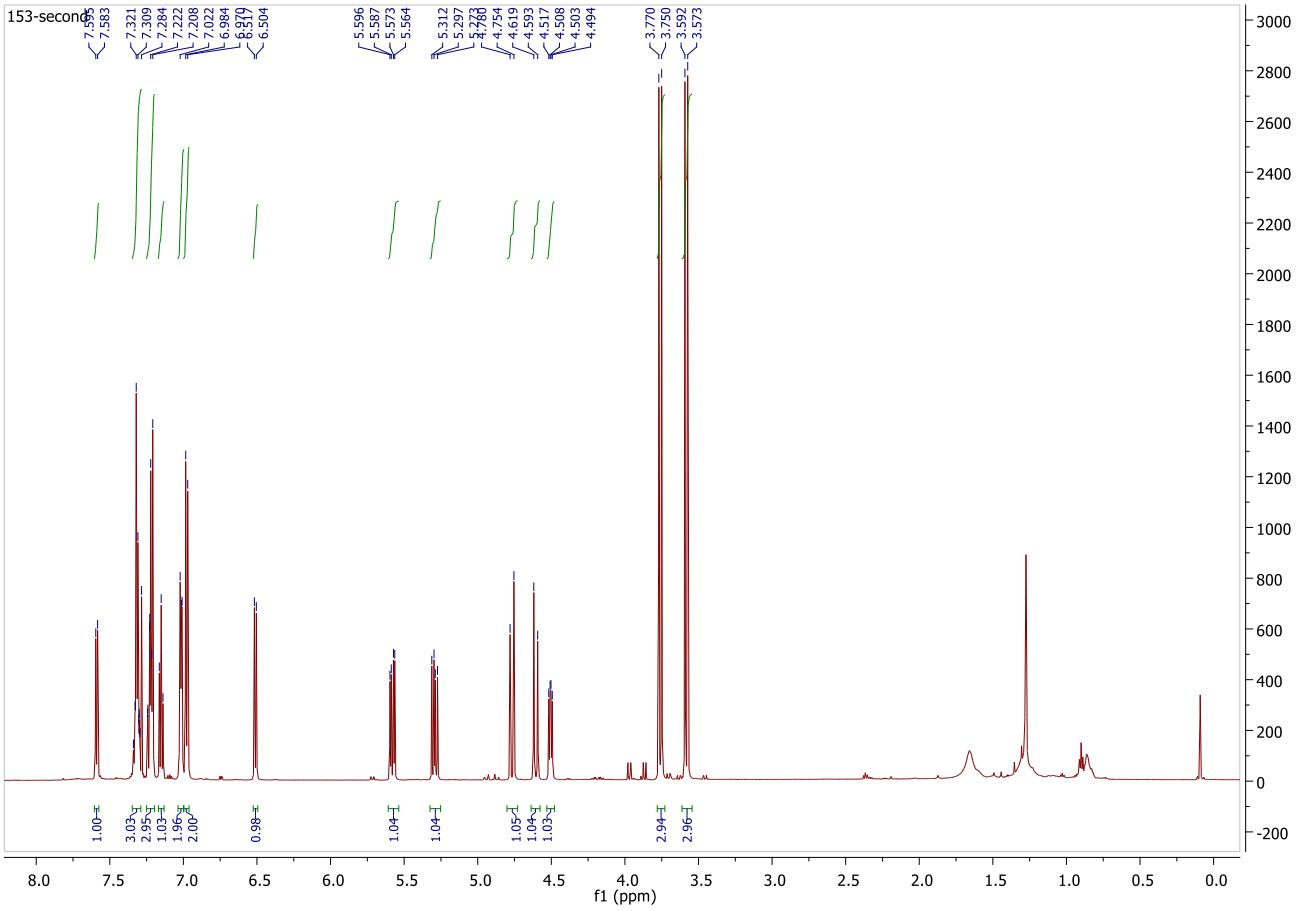


^{1}H NMR of 4ac (2nd diastereomer)

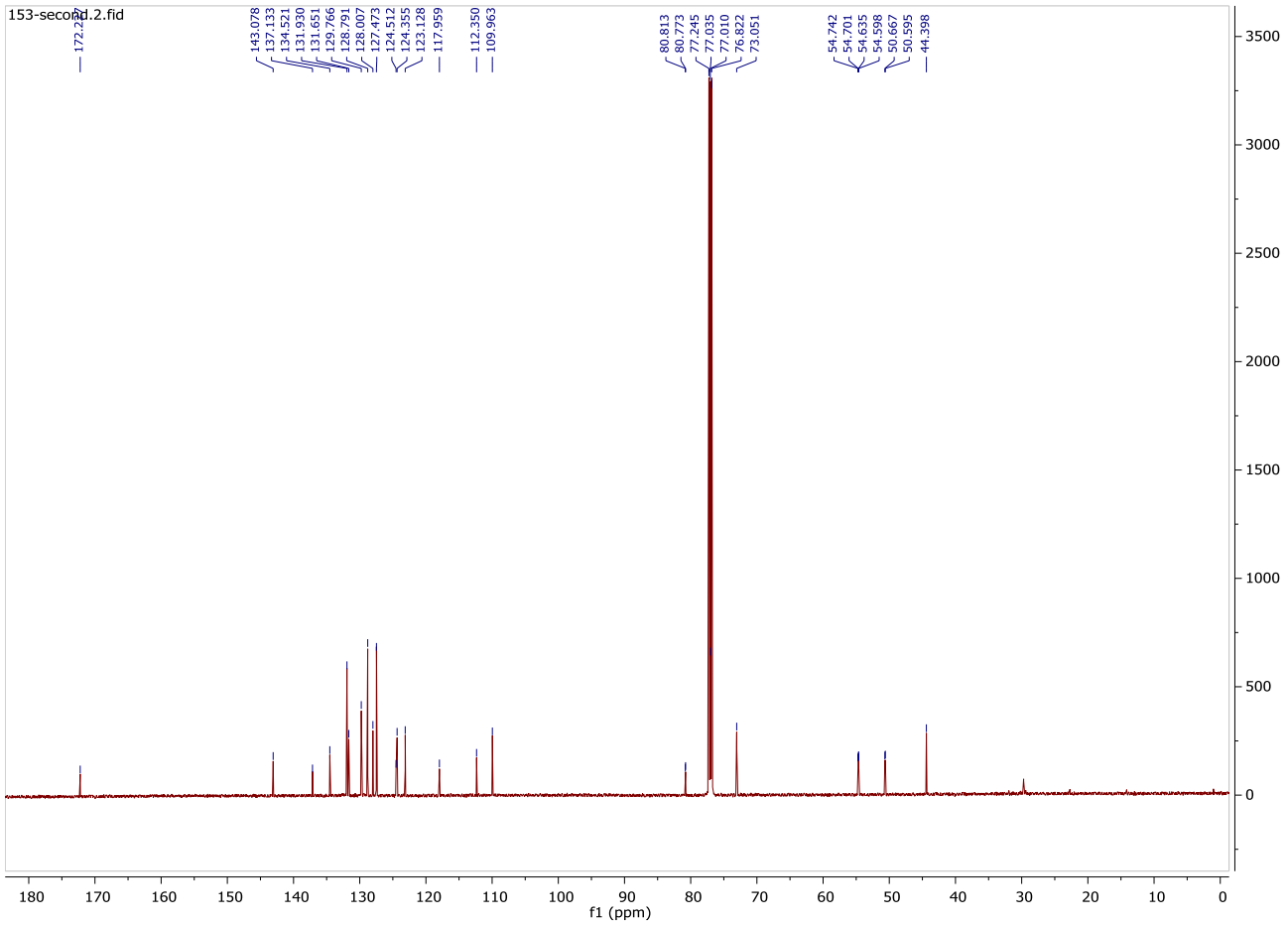


^{13}C NMR of 4ac (2nd diastereomer)

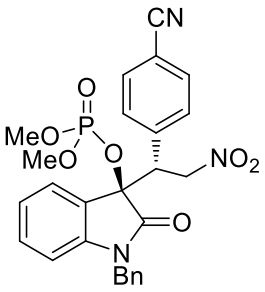

CN
O
MeO
P
MeO
O
NO2
O
N
Bn

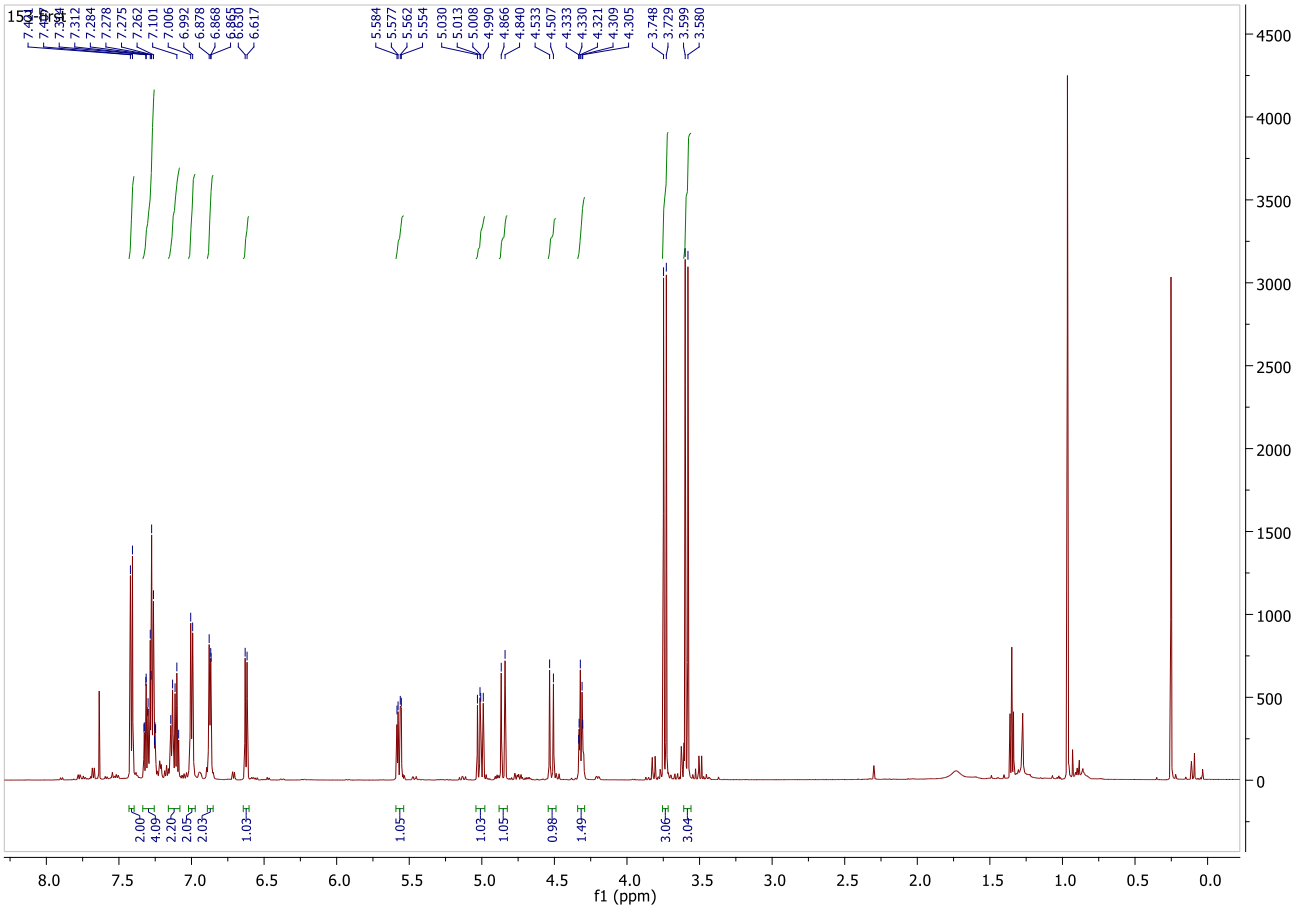

153-second
7.595
7.583
7.321
7.309
7.284
7.222
7.208
7.022
6.984
6.970
6.517
6.504
5.596
5.587
5.573
5.564
5.312
5.297
4.754
4.619
4.593
4.517
4.508
4.503
4.494
3.770
3.750
3.592
3.573
1.00
3.03
2.95
1.03
1.96
2.00
0.98
1.04
1.04
1.05
1.04
1.03
2.94
2.96
f1 (ppm)

[1]H NMR of 4ad

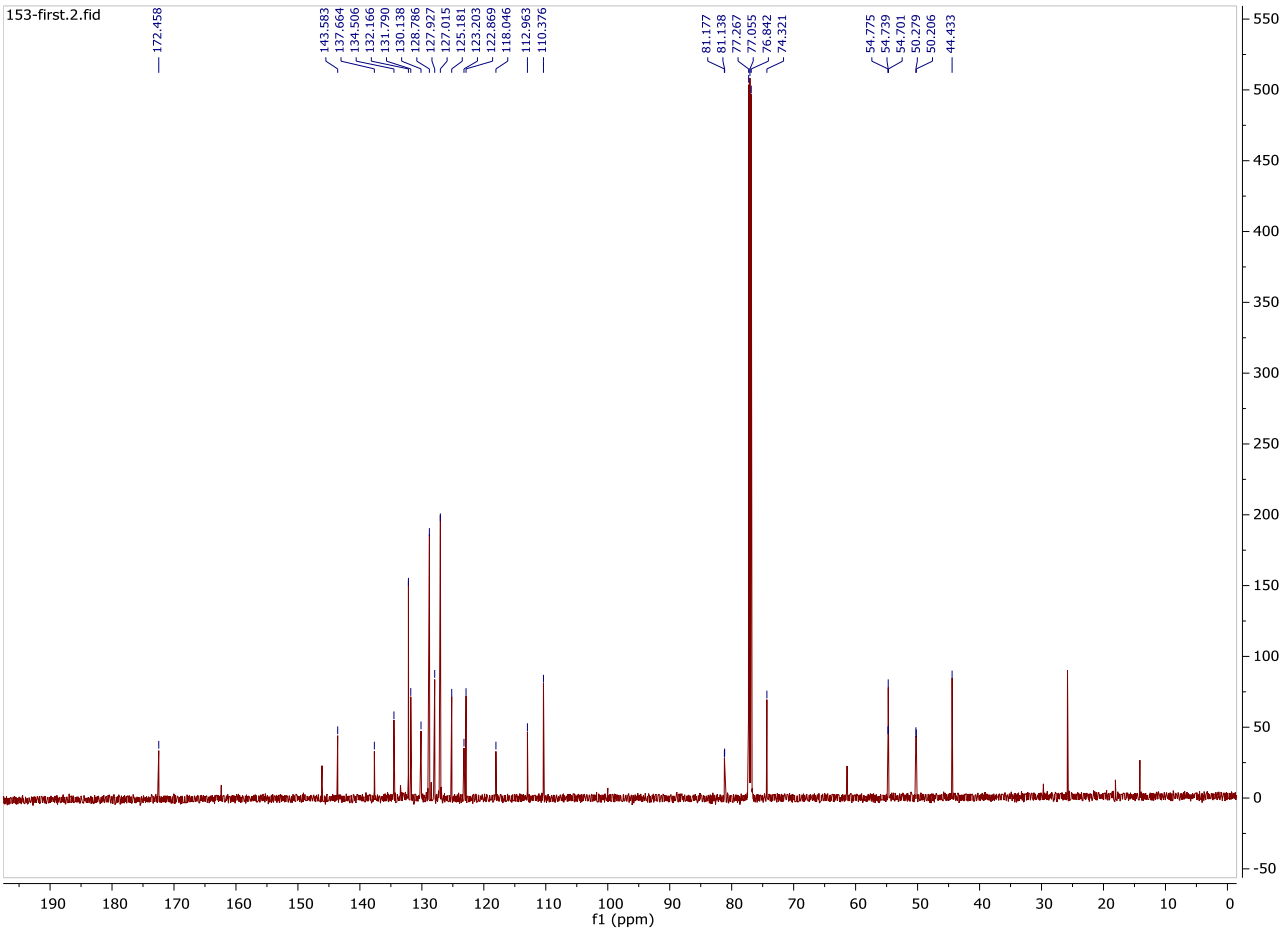


^{13}C NMR of 4ad

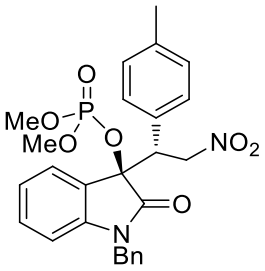


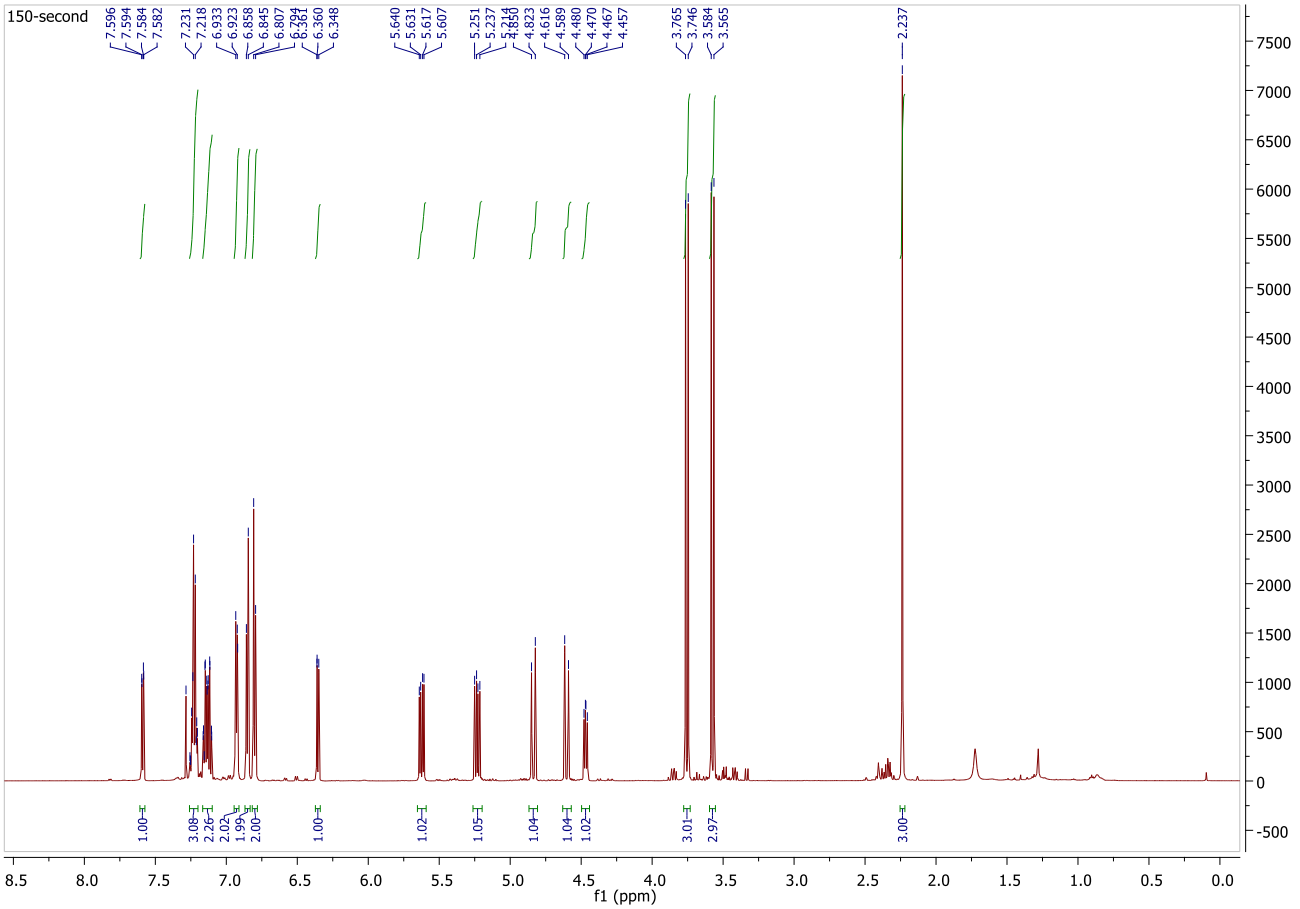


^{1}H NMR of 4ad (2nd diastereomer)

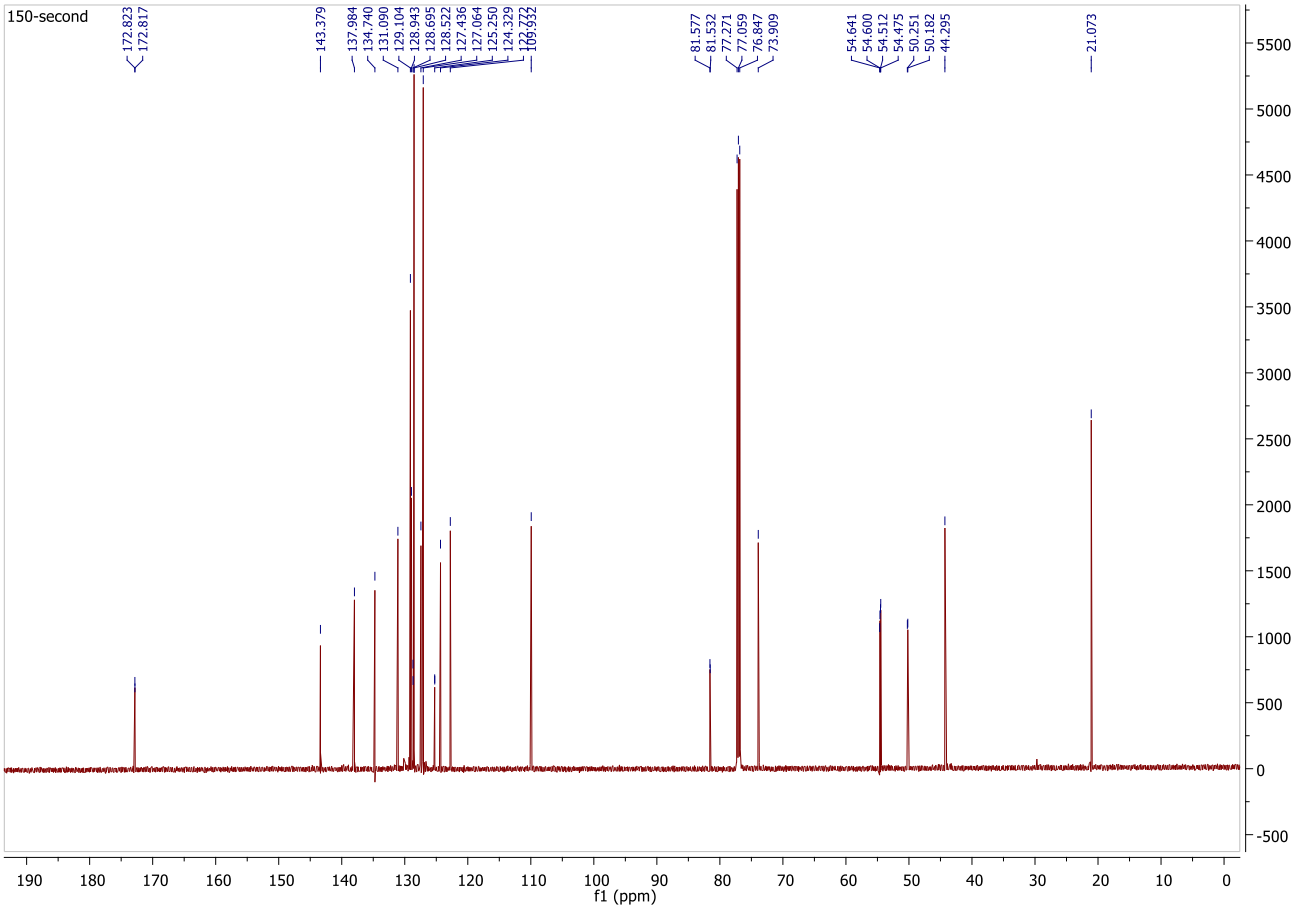


^{13}C NMR of 4ad (2nd diastereomer)

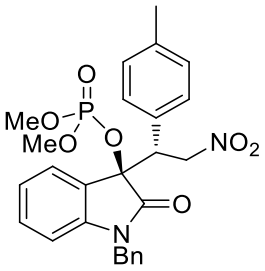


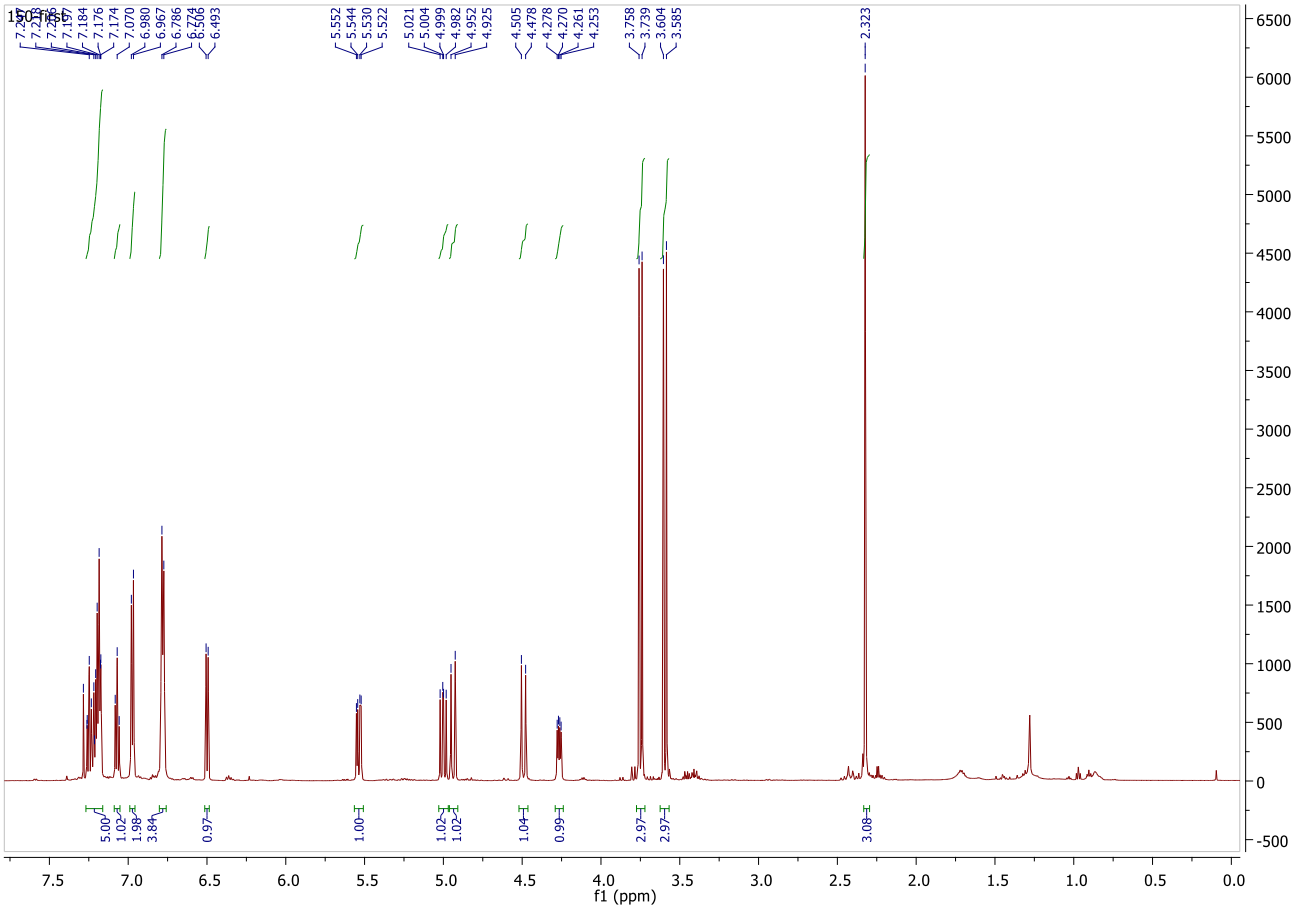


^{1}H NMR of 4ae

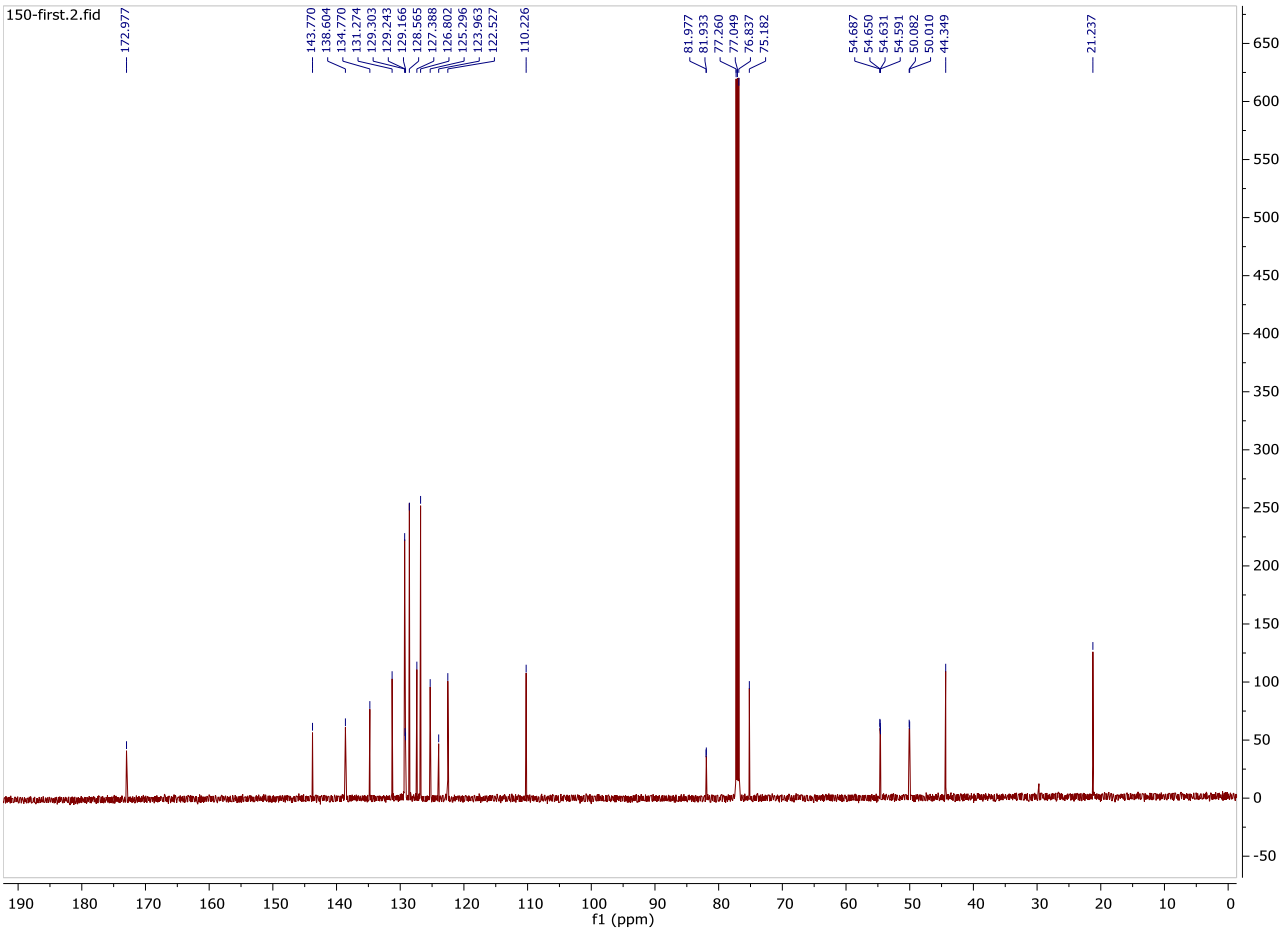


^{13}C NMR of 4ae

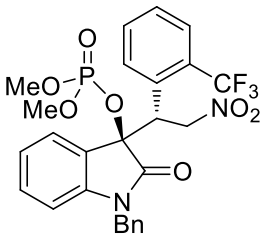


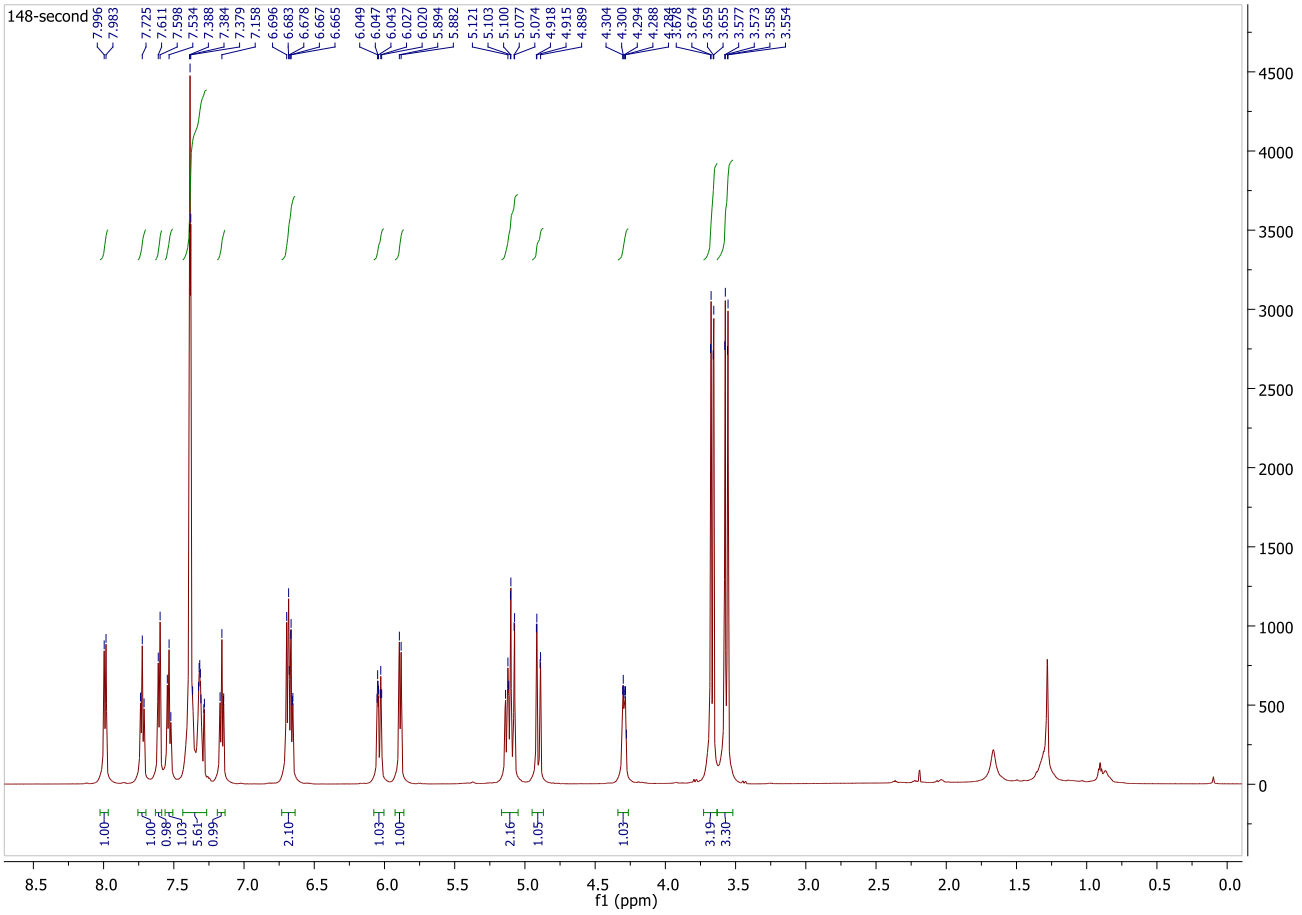


^{1}H NMR of 4ae (2nd diastereomer)

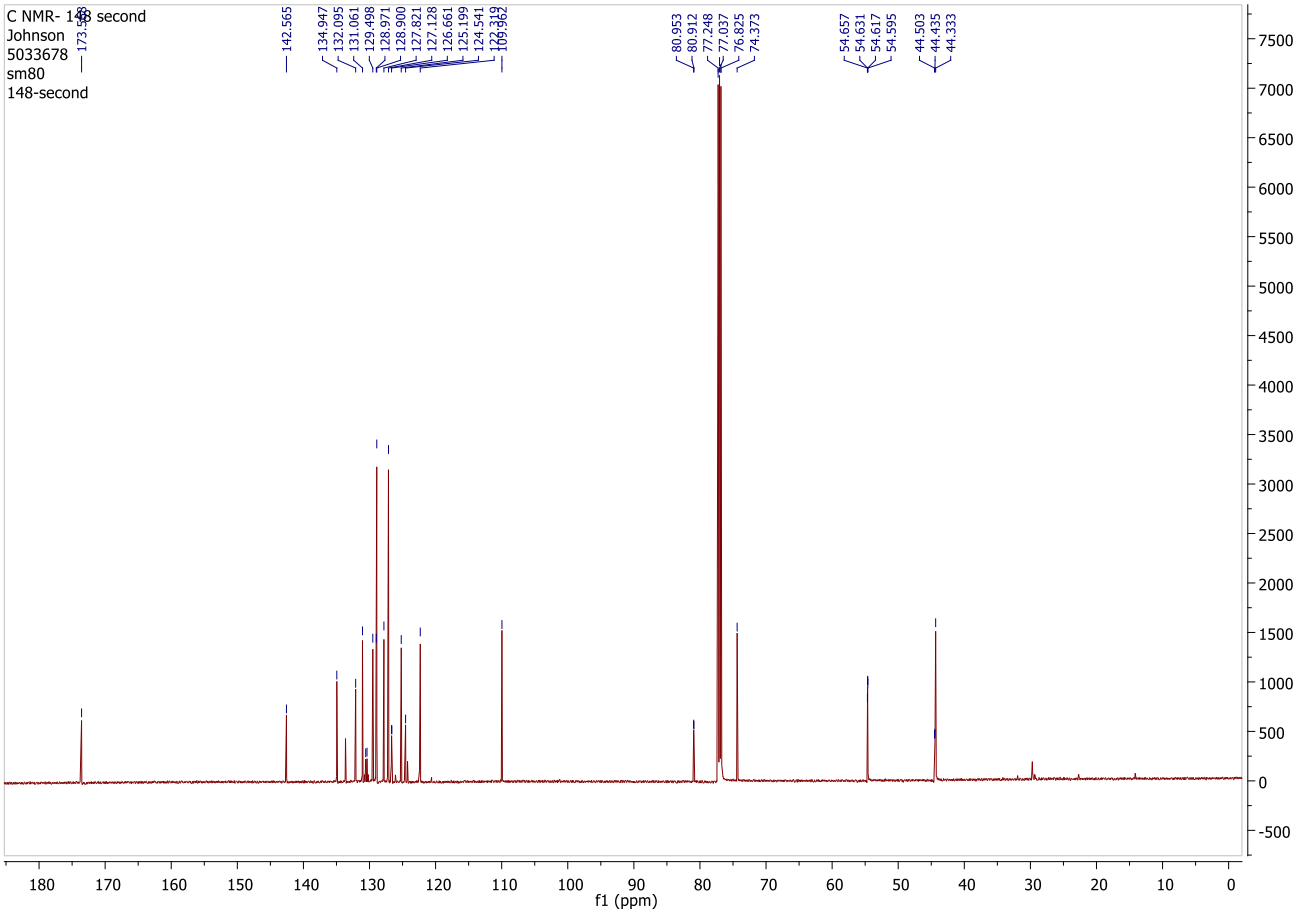


^{13}C NMR of 4ae (2nd diastereomer)

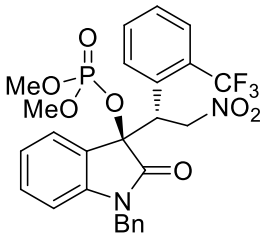

O
MeO
P
MeO
O
CF3
NO2
O
N
Bn

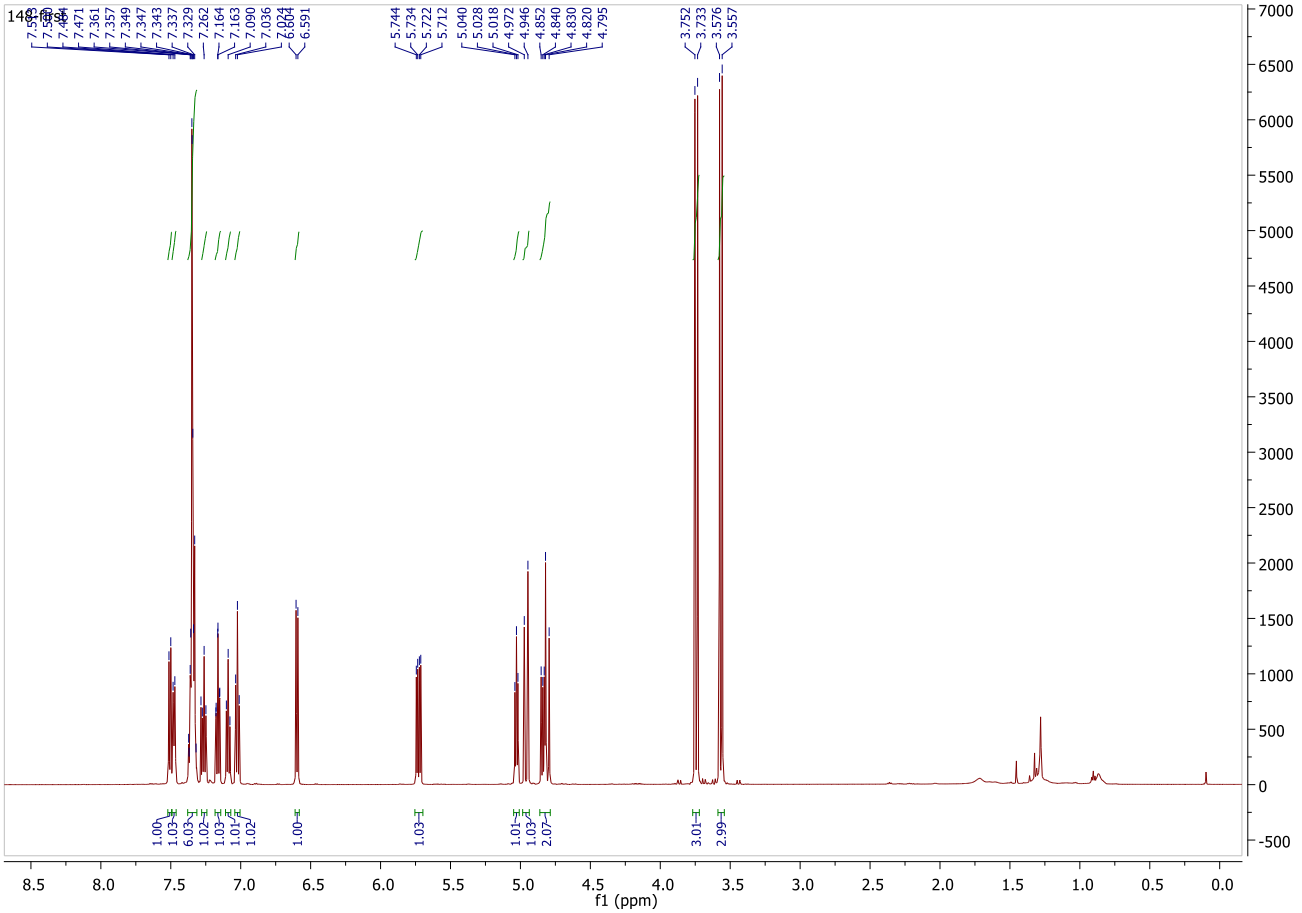

148-second
7.996
7.983
7.725
7.611
7.598
7.534
7.388
7.384
7.379
7.158
6.696
6.683
6.678
6.667
6.665
6.049
6.047
6.043
6.027
6.020
5.894
5.882
5.121
5.103
5.100
5.077
5.074
4.918
4.915
4.889
4.304
4.300
4.294
4.288
3.674
3.659
3.655
3.577
3.573
3.558
3.554
1.00
1.00
0.98
1.03
5.61
0.99
2.10
1.03
1.00
2.16
1.05
1.03
3.19
3.30
4500
4000
3500
3000
2500
2000
1500
1000
500
0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

^{1}H NMR of 4af

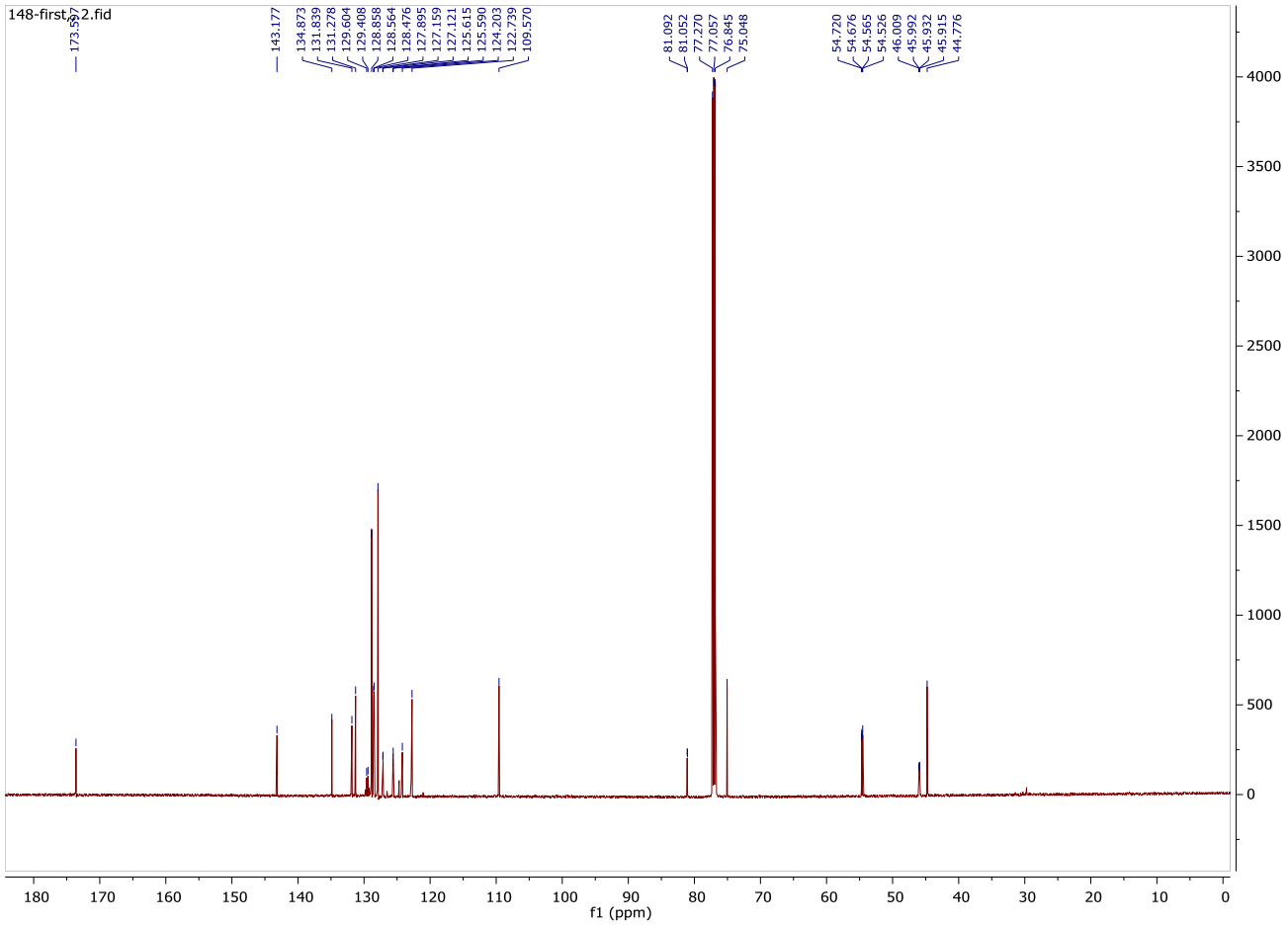


^{13}C NMR of 4af

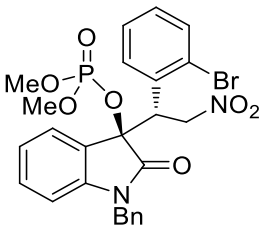


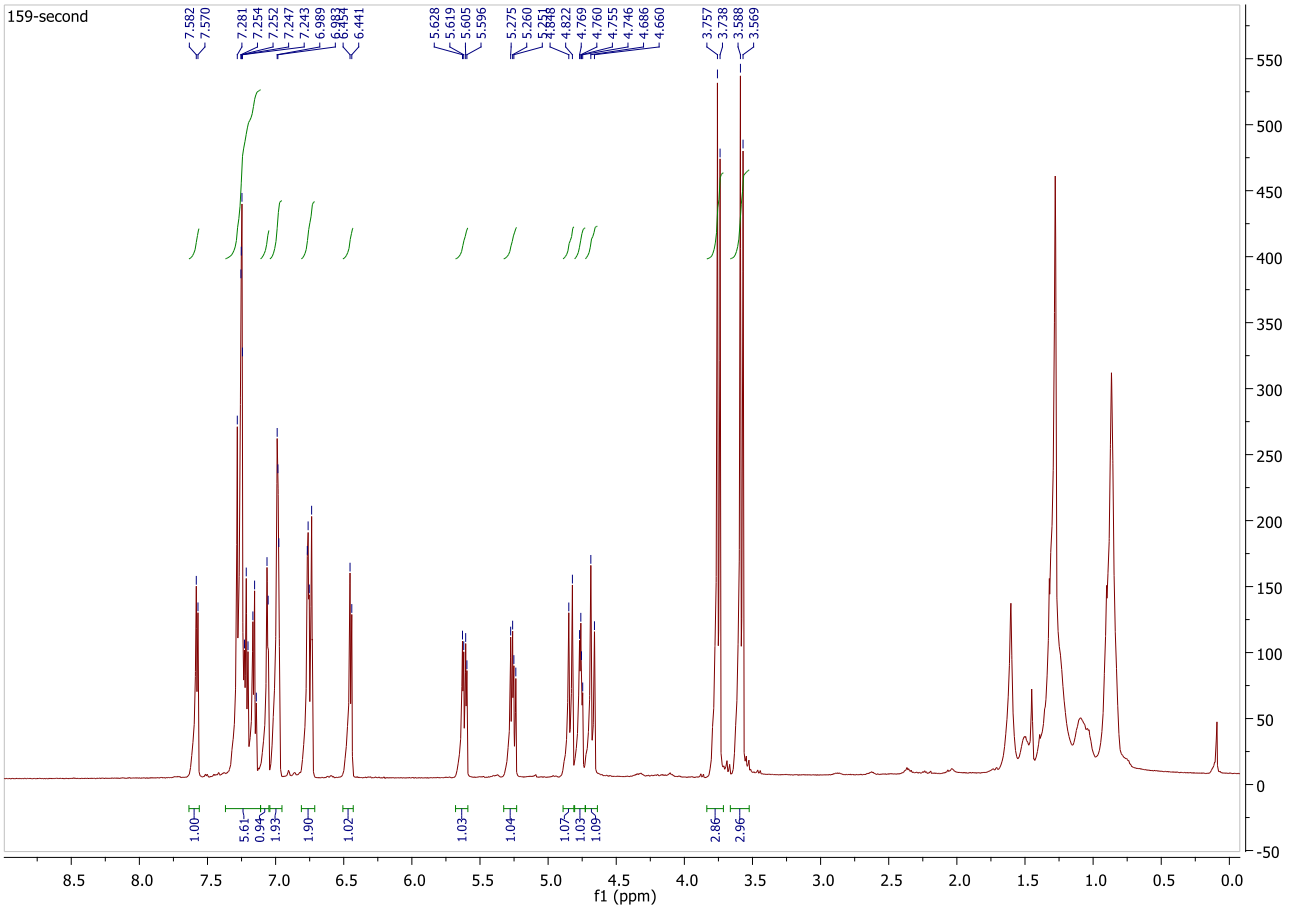


^{1}H NMR of 4af (2nd diastereomer)

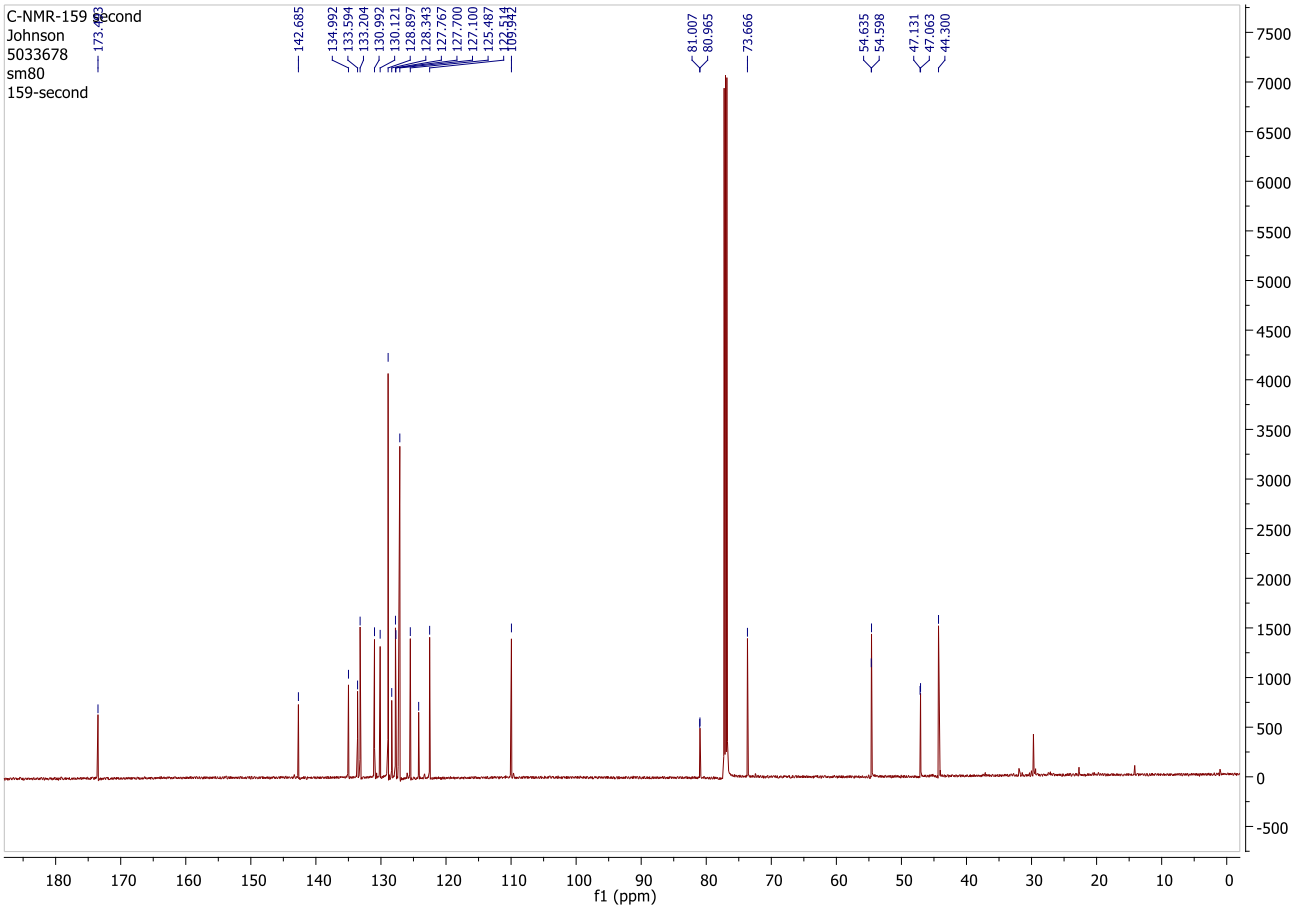


^{13}C NMR of 4af (2nd diastereomer)

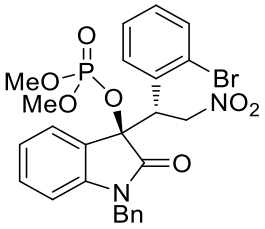


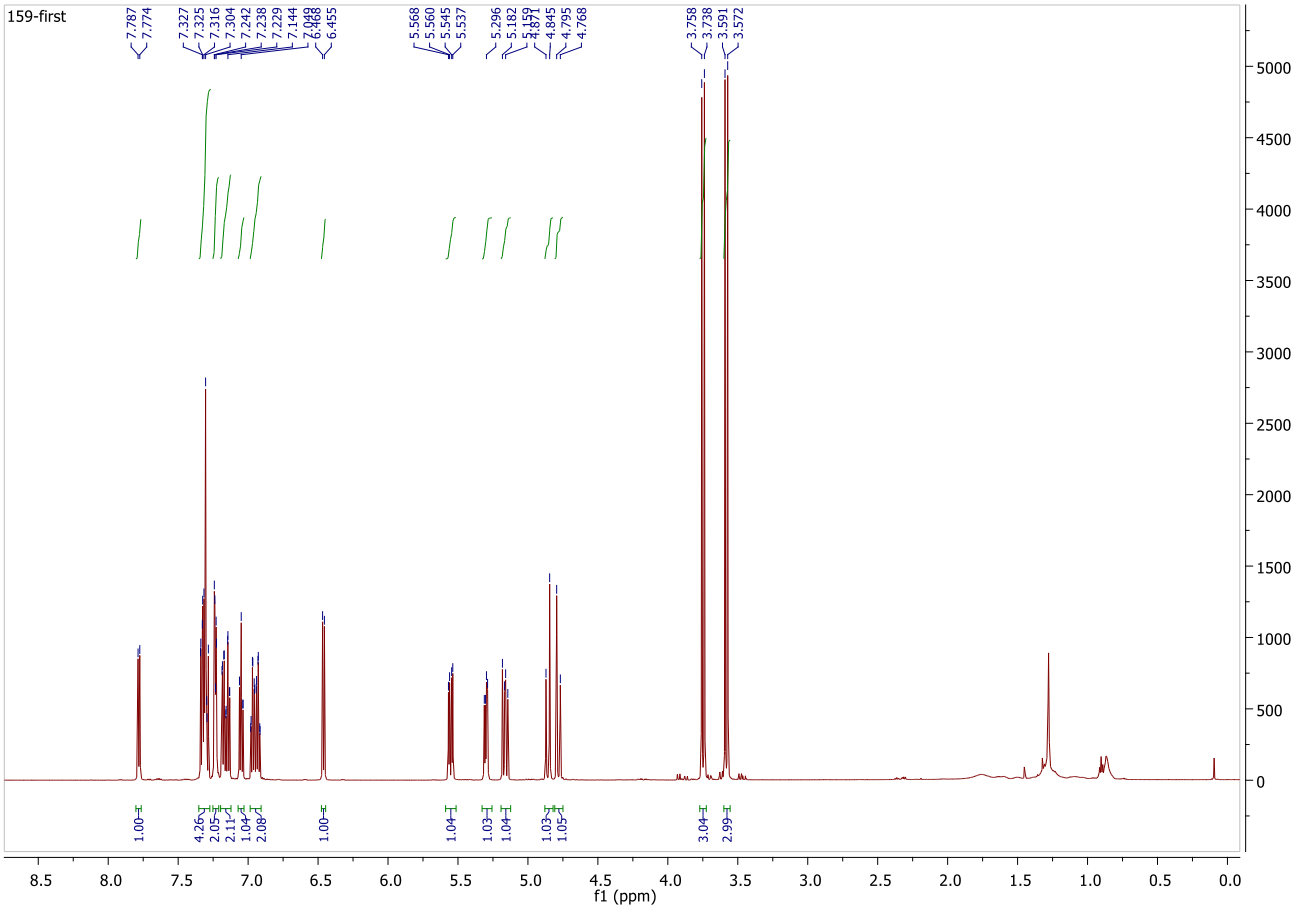


^{1}H NMR of 4ag

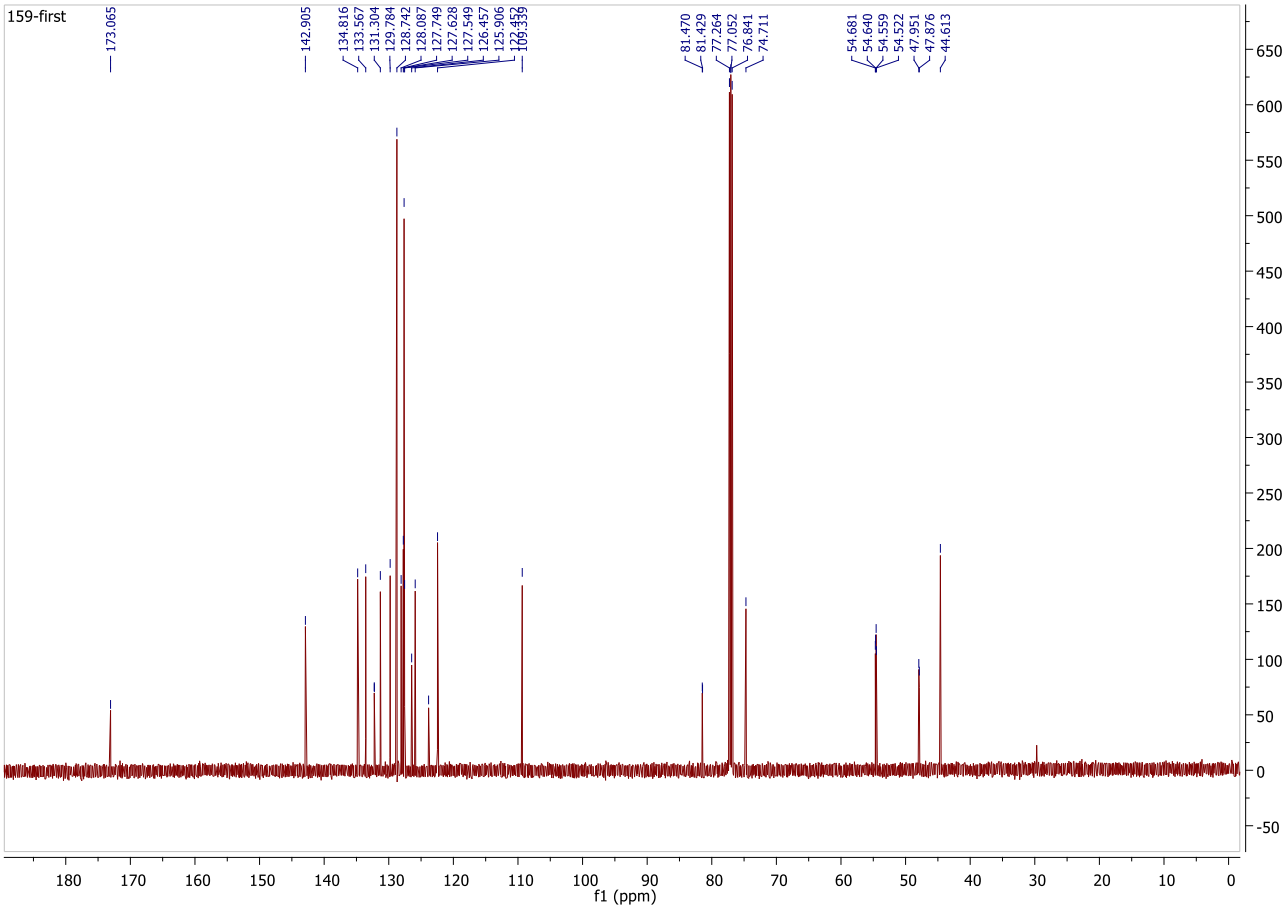


^{13}C NMR of 4ag

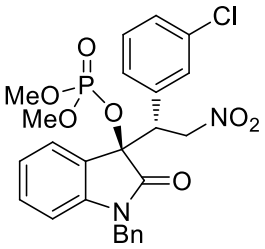


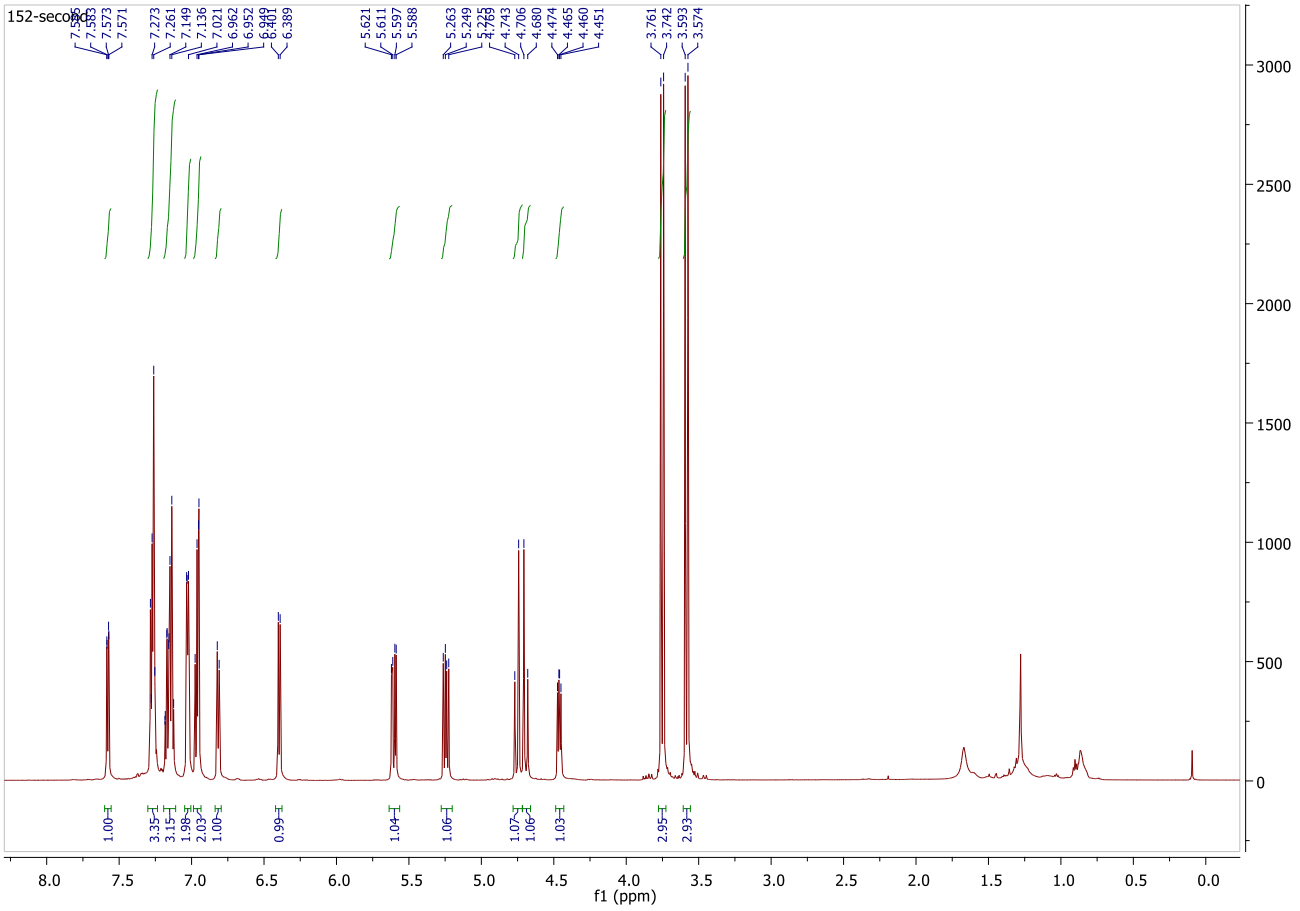


^{1}H NMR of 4ag (2nd diastereomer)

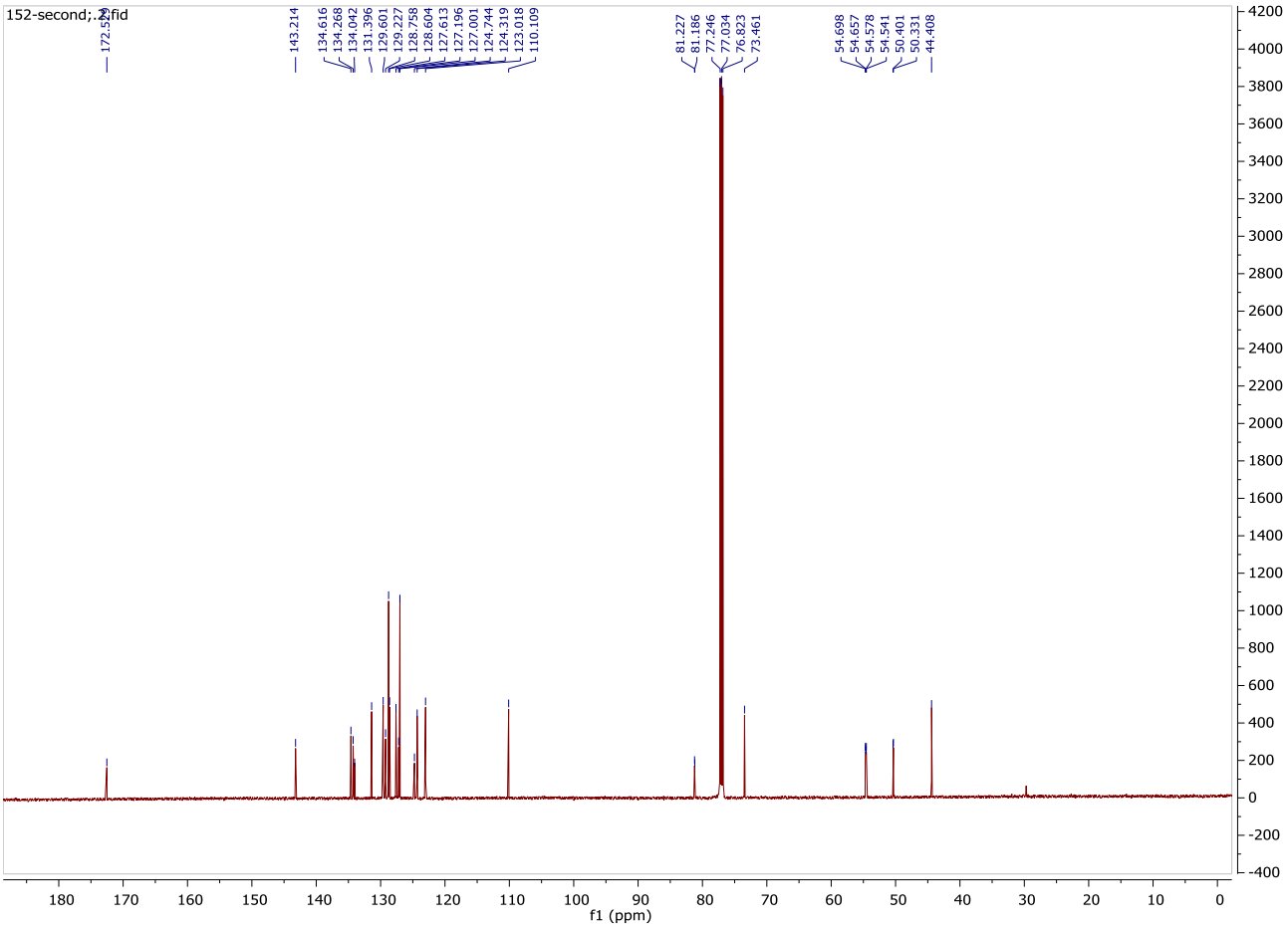


^{13}C NMR of 4ag (2nd diastereomer)

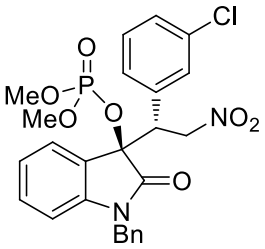

Cl
O
MeO
P
MeO
O
NO2
O
N
Bn

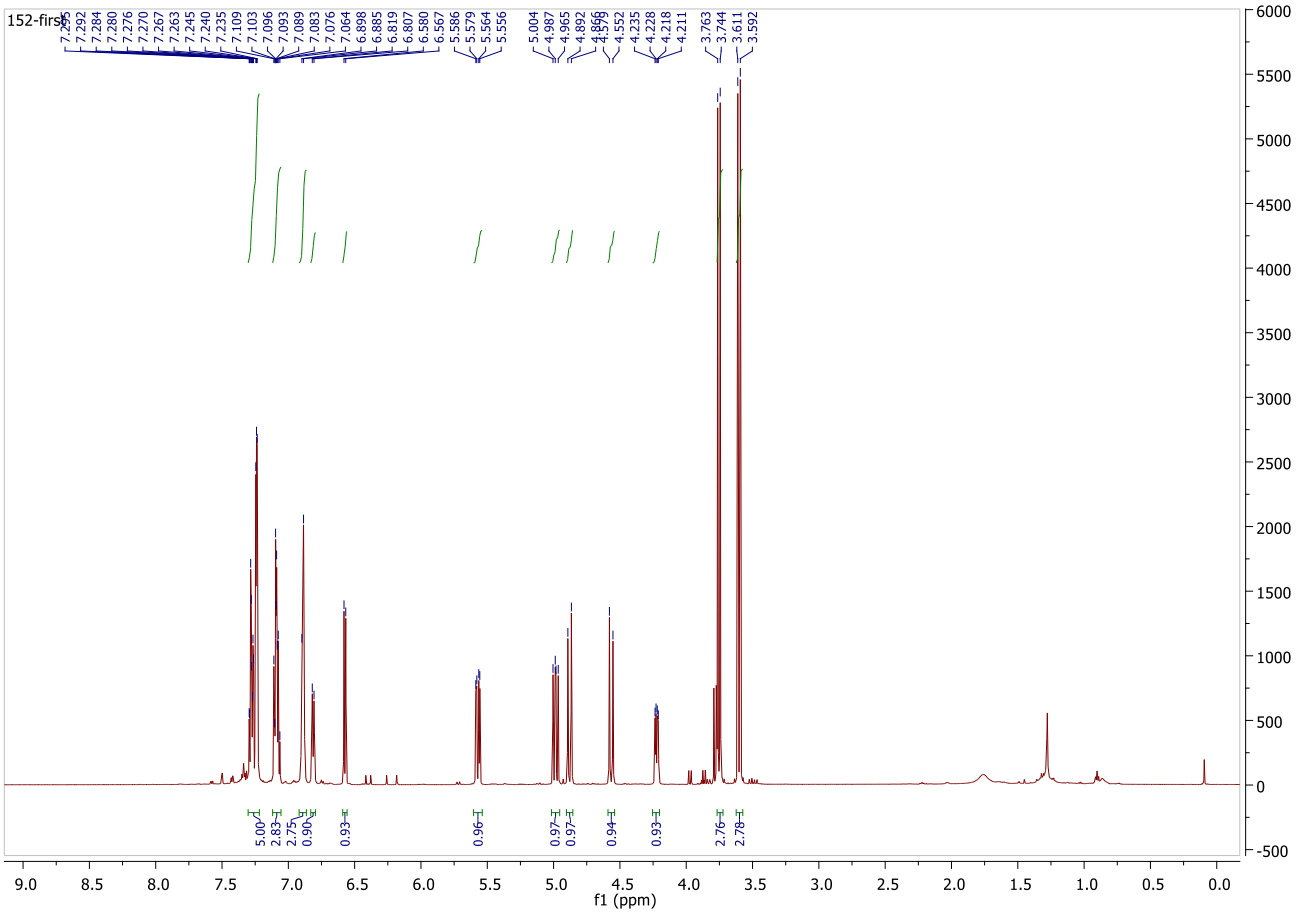

152-second
7.583
7.573
7.571
7.273
7.261
7.149
7.136
7.021
6.962
6.952
6.940
6.389
5.621
5.611
5.597
5.588
5.263
5.249
5.225
4.769
4.743
4.706
4.680
4.474
4.465
4.460
4.451
3.761
3.742
3.593
3.574
3000
2500
2000
1500
1000
500
0
1.00
3.35
3.15
1.98
2.03
1.00
0.99
1.04
1.06
1.07
1.06
1.03
2.95
2.93
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

^{1}H NMR of 4ah

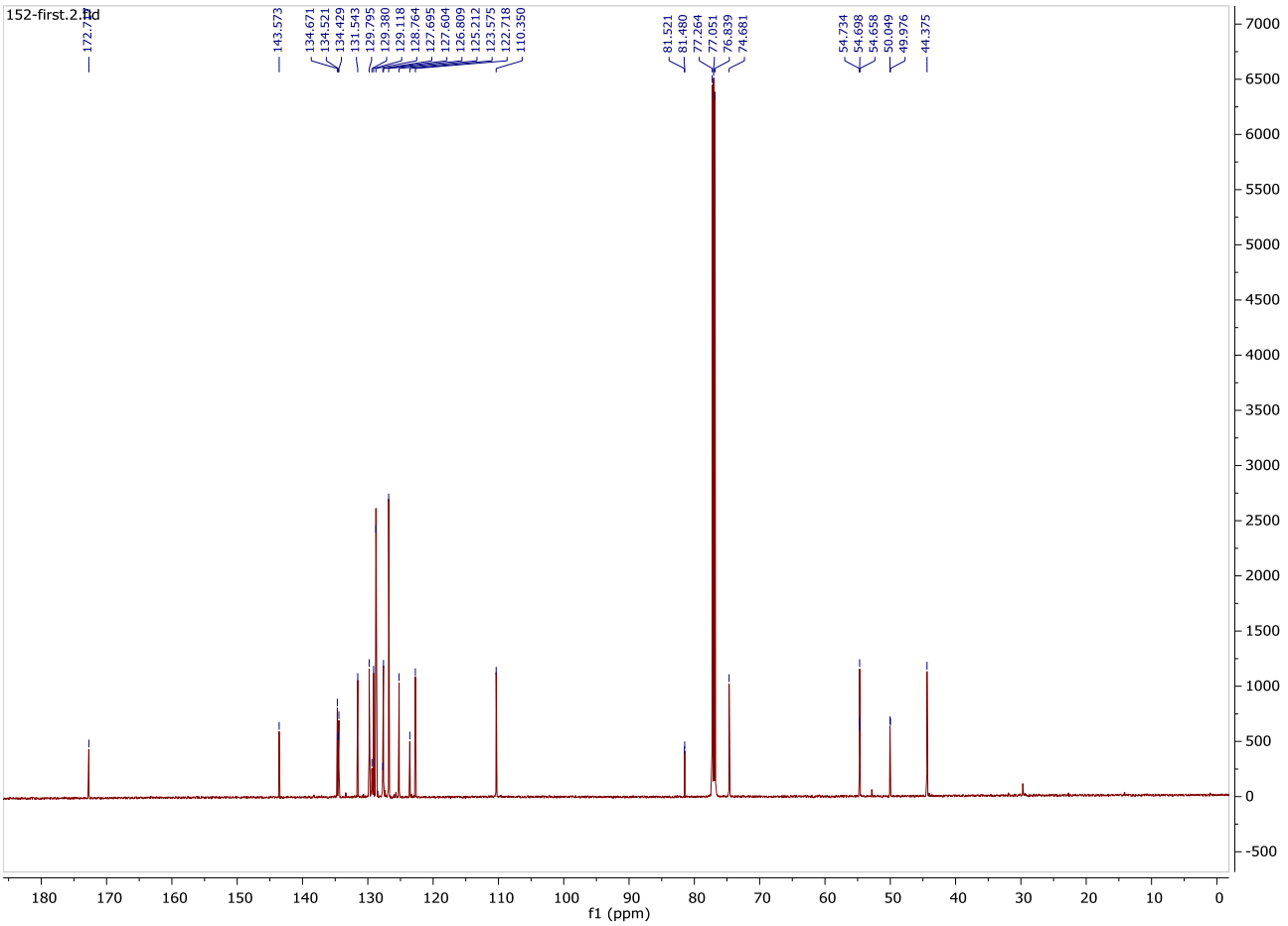


^{13}C NMR of 4ah

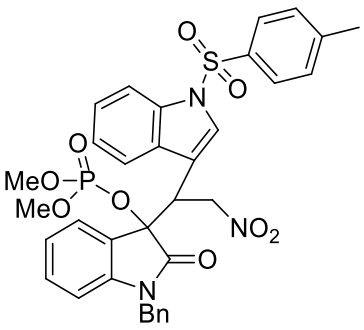


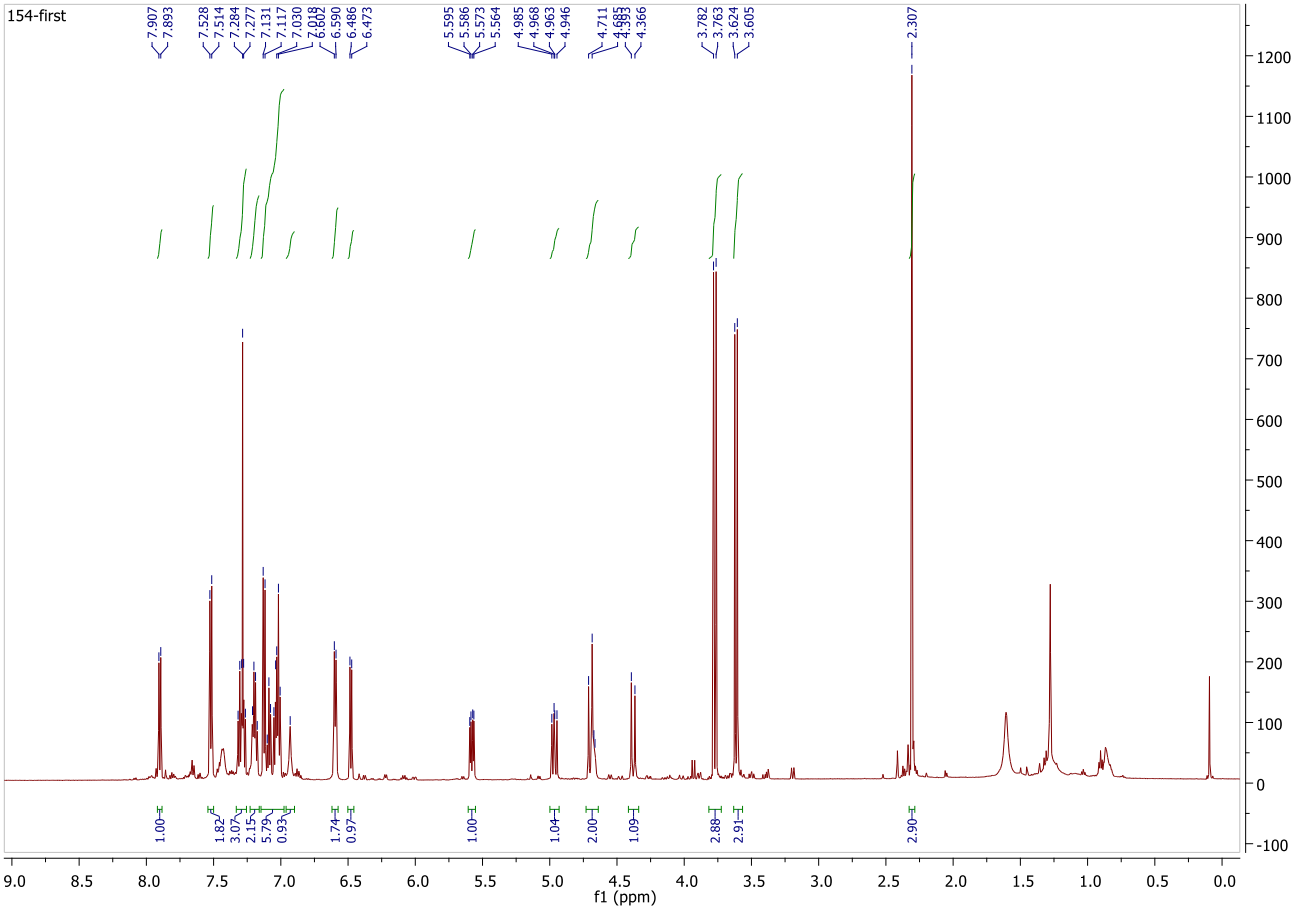


^{1}H NMR of 4ah (2nd diastereomer)

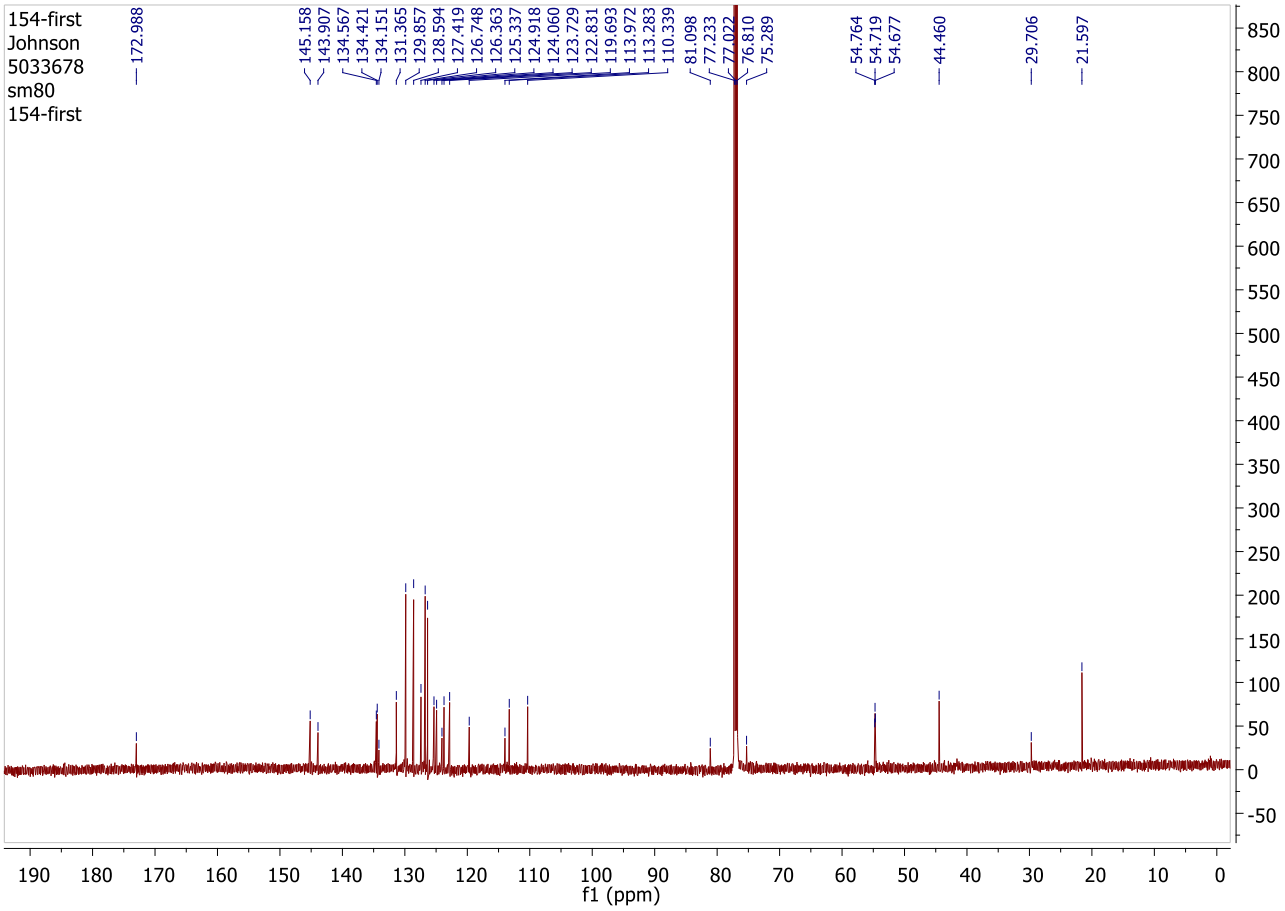


^{13}C NMR of 4ah (2nd diastereomer)

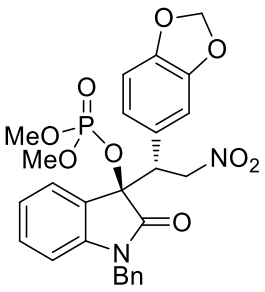

O
S
O
N
O
MeO
P
MeO
O
NO2
O
N
Bn

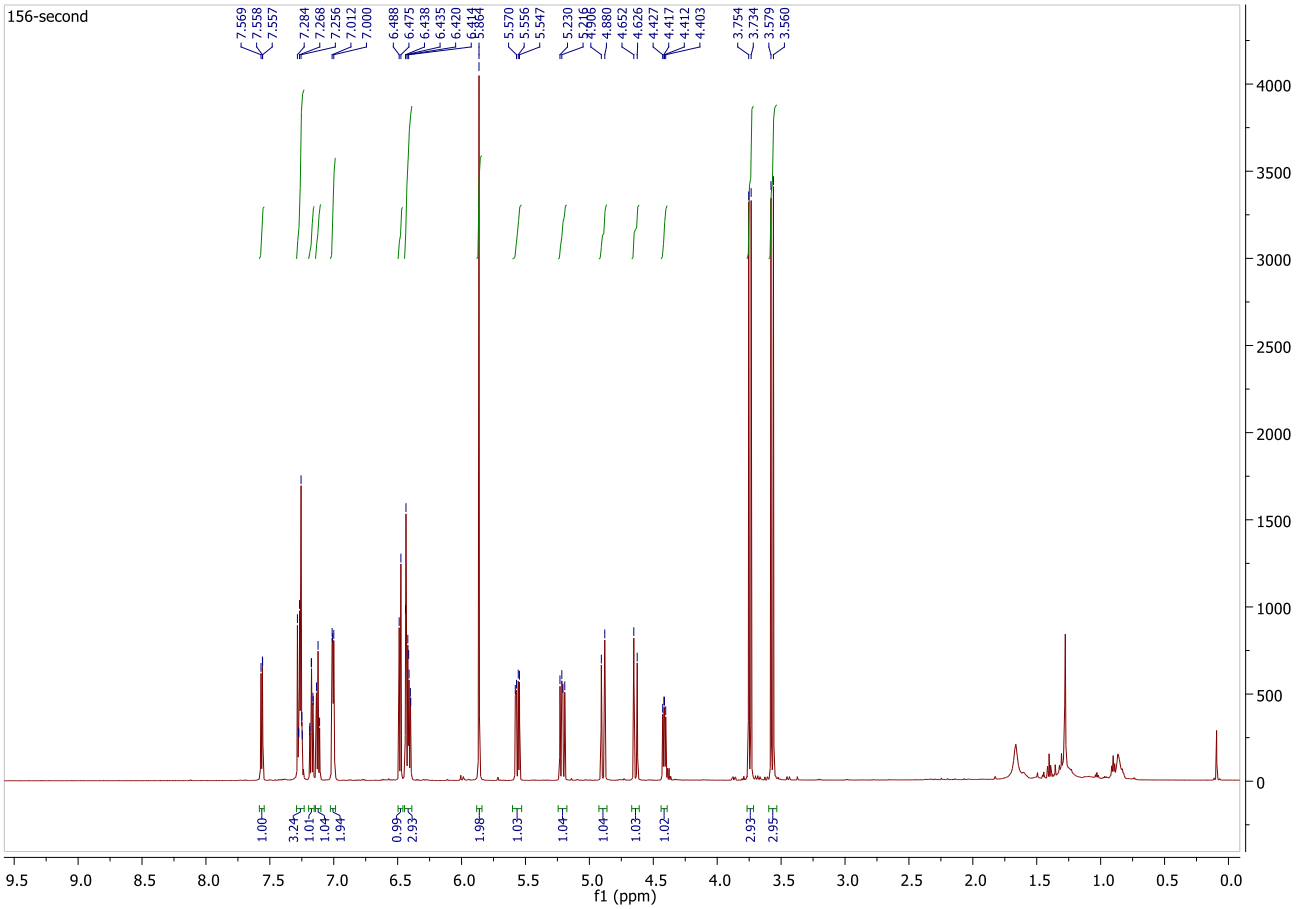

154-first
7.907
7.893
7.528
7.514
7.284
7.277
7.131
7.117
7.030
7.018
6.602
6.590
6.486
6.473
5.595
5.586
5.573
5.564
4.985
4.968
4.963
4.946
4.711
4.685
4.393
4.366
3.782
3.763
3.624
3.605
2.307
1.00
1.82
3.07
2.15
5.79
0.93
1.74
0.97
1.00
1.04
2.00
1.09
2.88
2.91
2.90
f1 (ppm)

[1]H NMR of 4ai

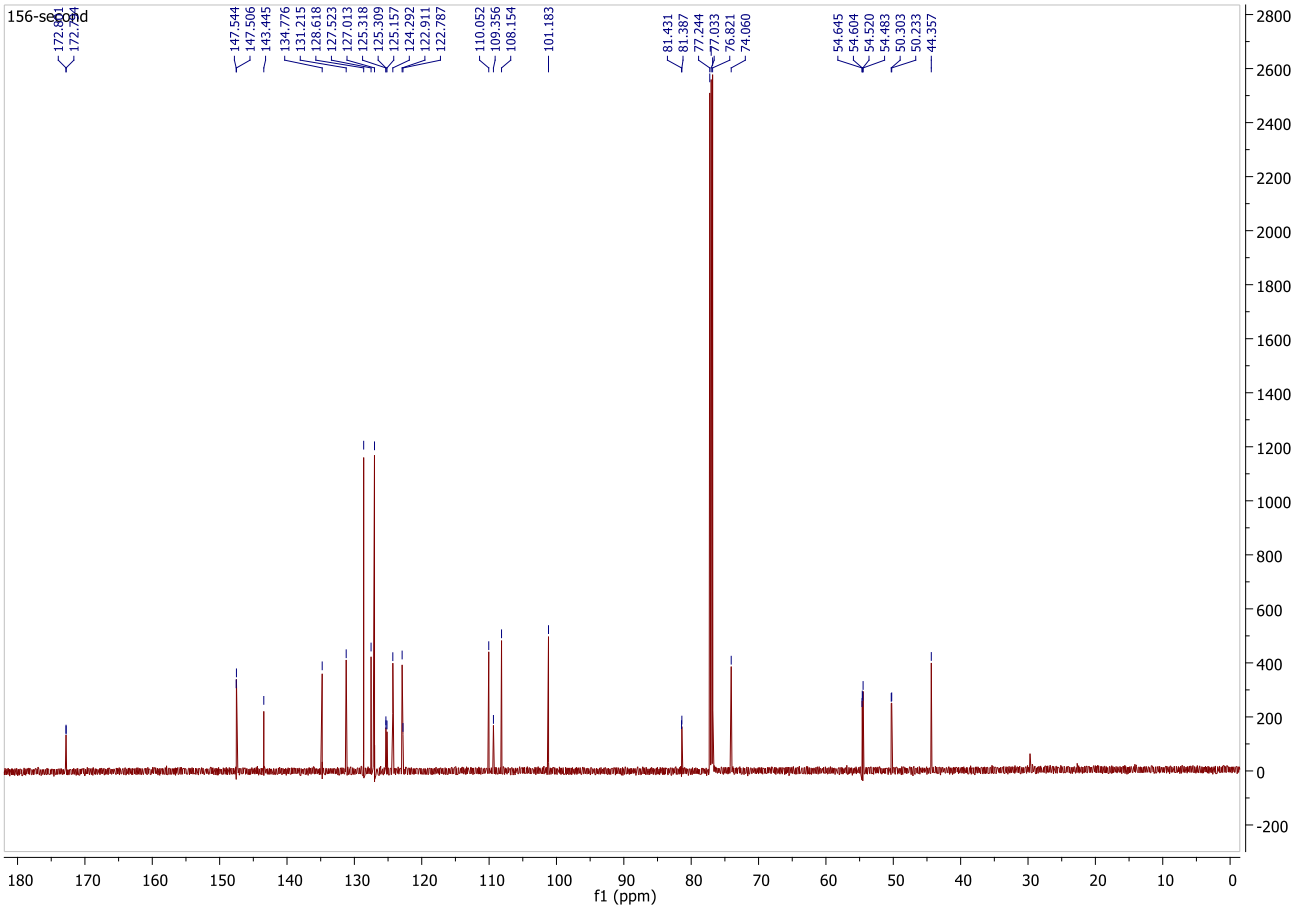


^{13}C NMR of 4ai

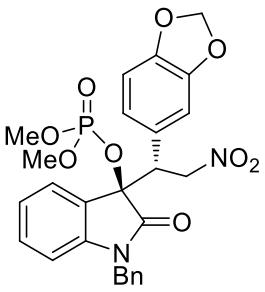

O
O
O
MeO
MeO
P
O
NO2
O
N
Bn

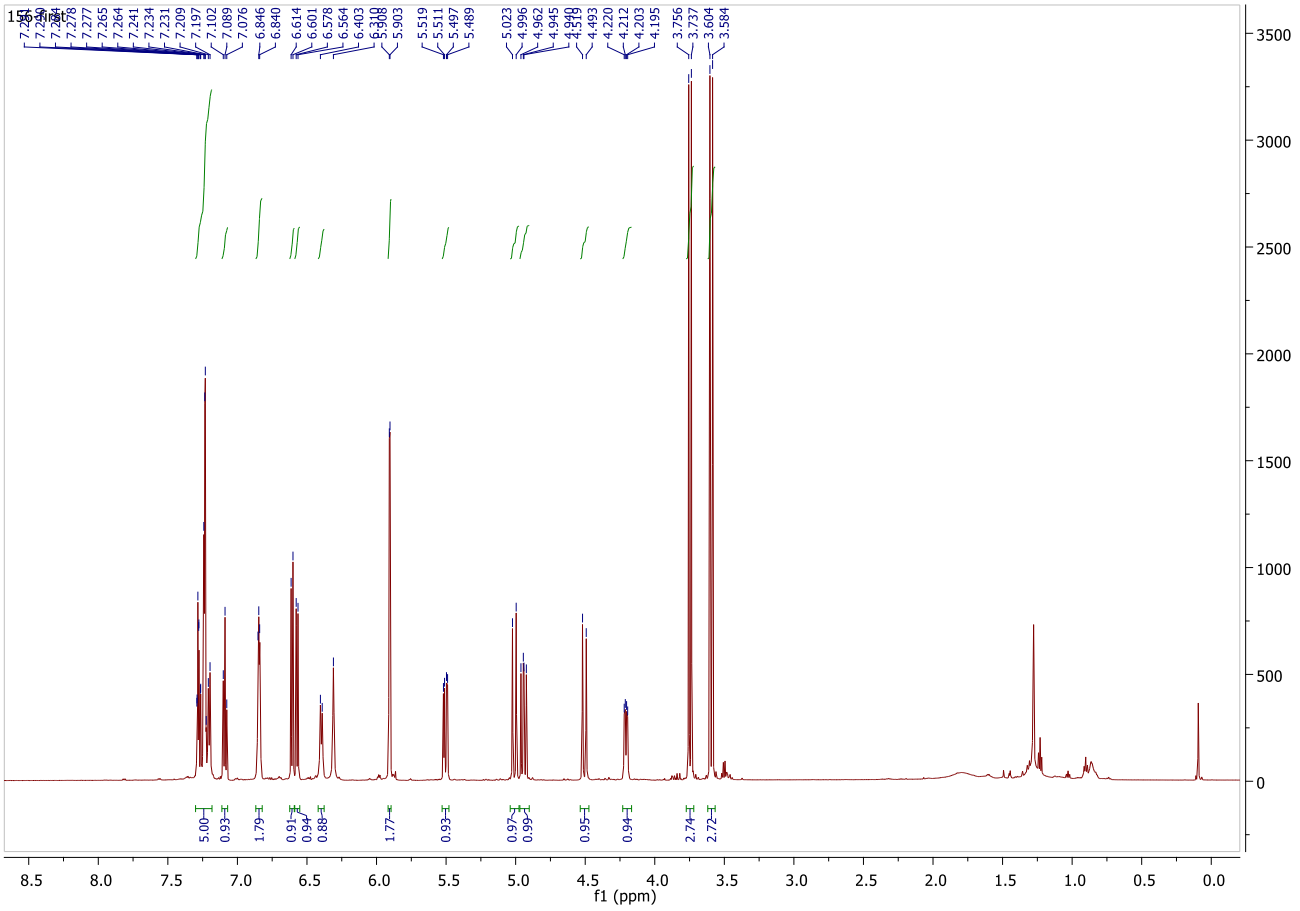

156-second
7.569
7.558
7.557
7.284
7.268
7.256
7.012
7.000
6.488
6.475
6.438
6.435
6.420
6.414
5.864
5.570
5.556
5.547
5.230
5.216
5.206
4.880
4.652
4.626
4.427
4.417
4.412
4.403
3.754
3.734
3.579
3.560
1.00
3.24
1.01
1.04
1.94
0.99
2.93
1.98
1.03
1.04
1.04
1.03
1.02
2.93
2.95
f1 (ppm)

^{1}H NMR of 4aj

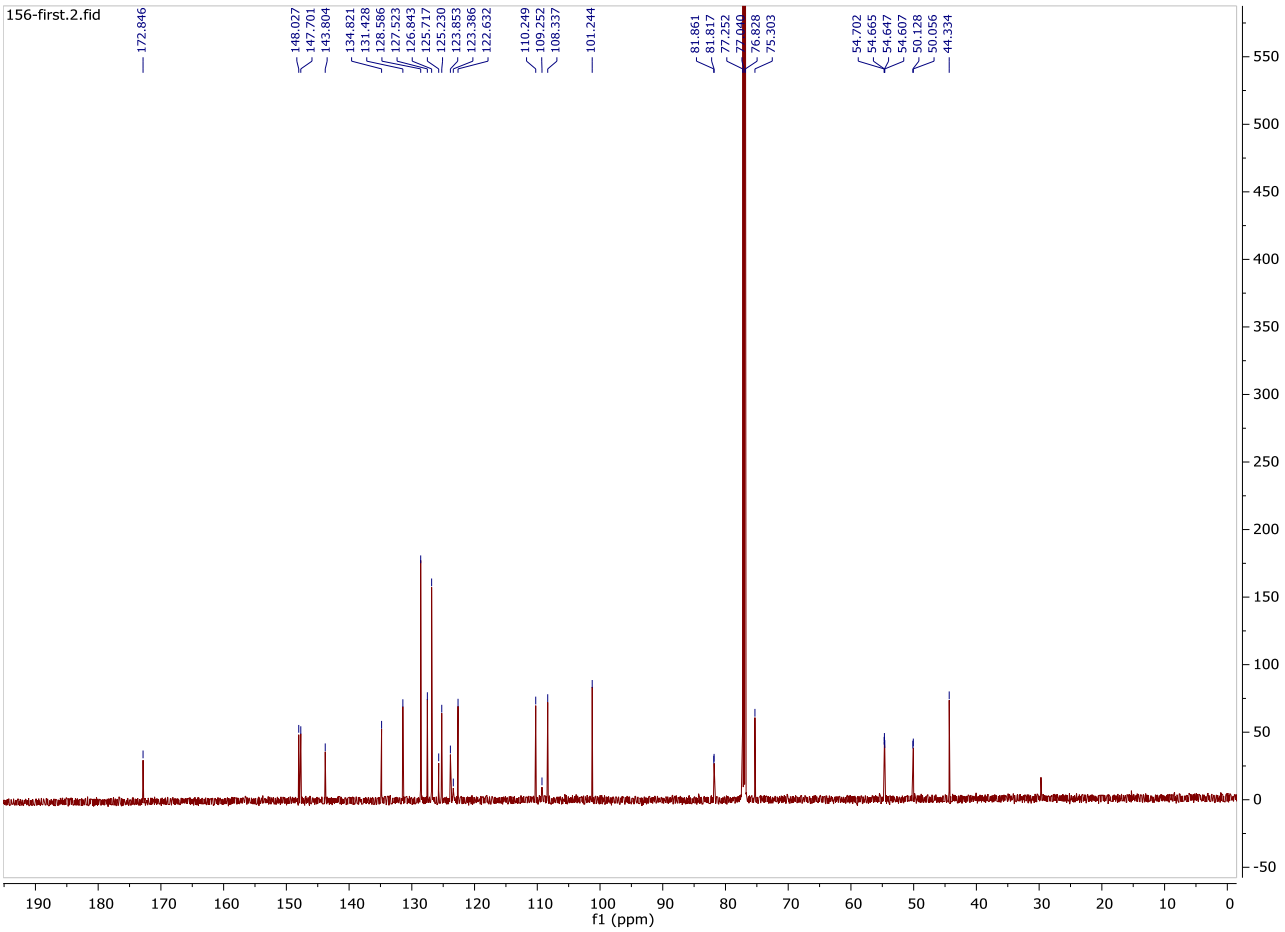


^{13}C NMR of 4aj

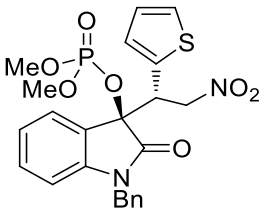


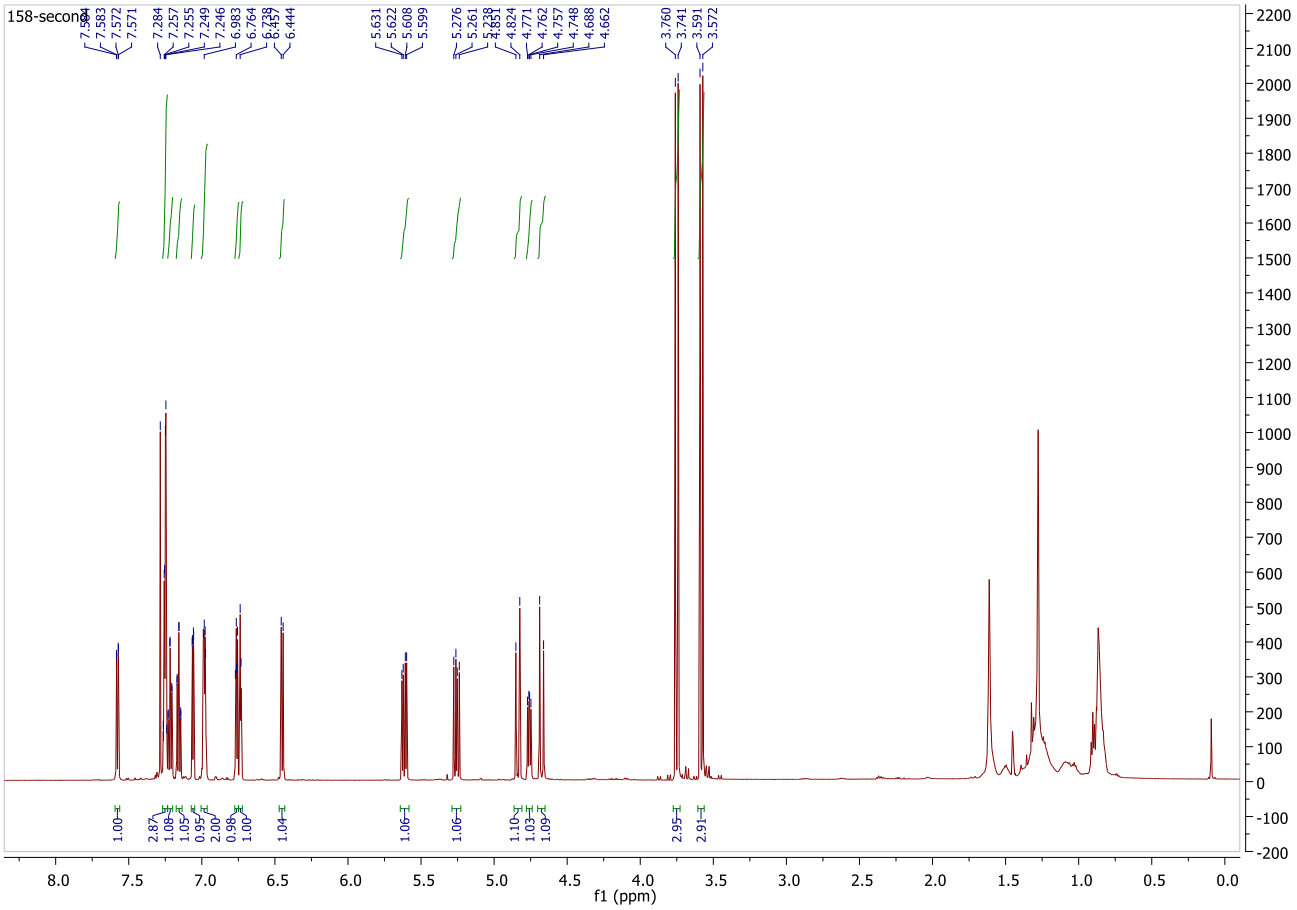


^{1}H NMR of 4aj (2nd diastereomer)

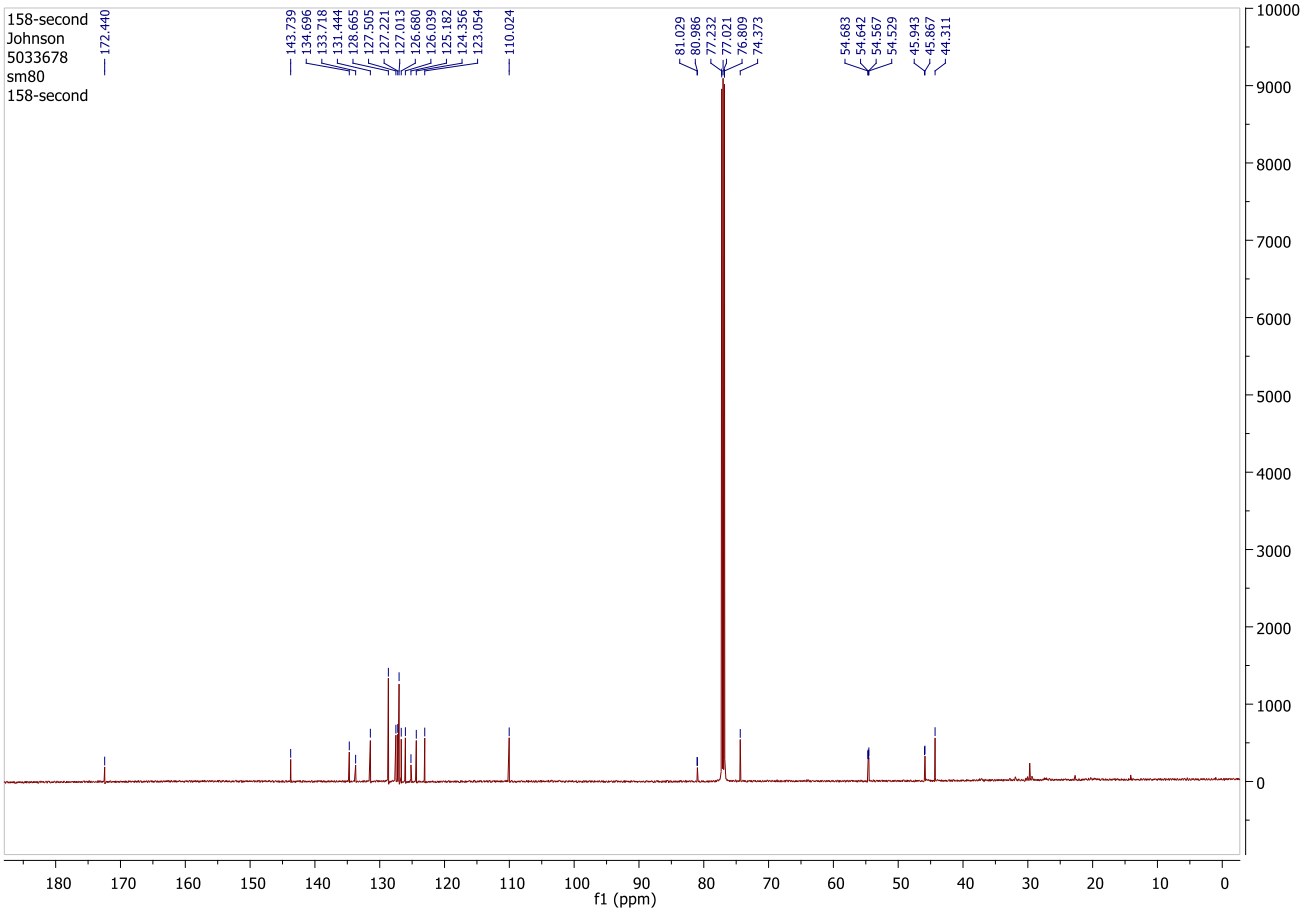


^{13}C NMR of 4aj (2nd diastereomer)

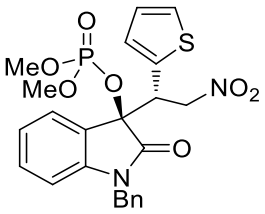

O
MeO
P
MeO
O
S
NO2
O
N
Bn

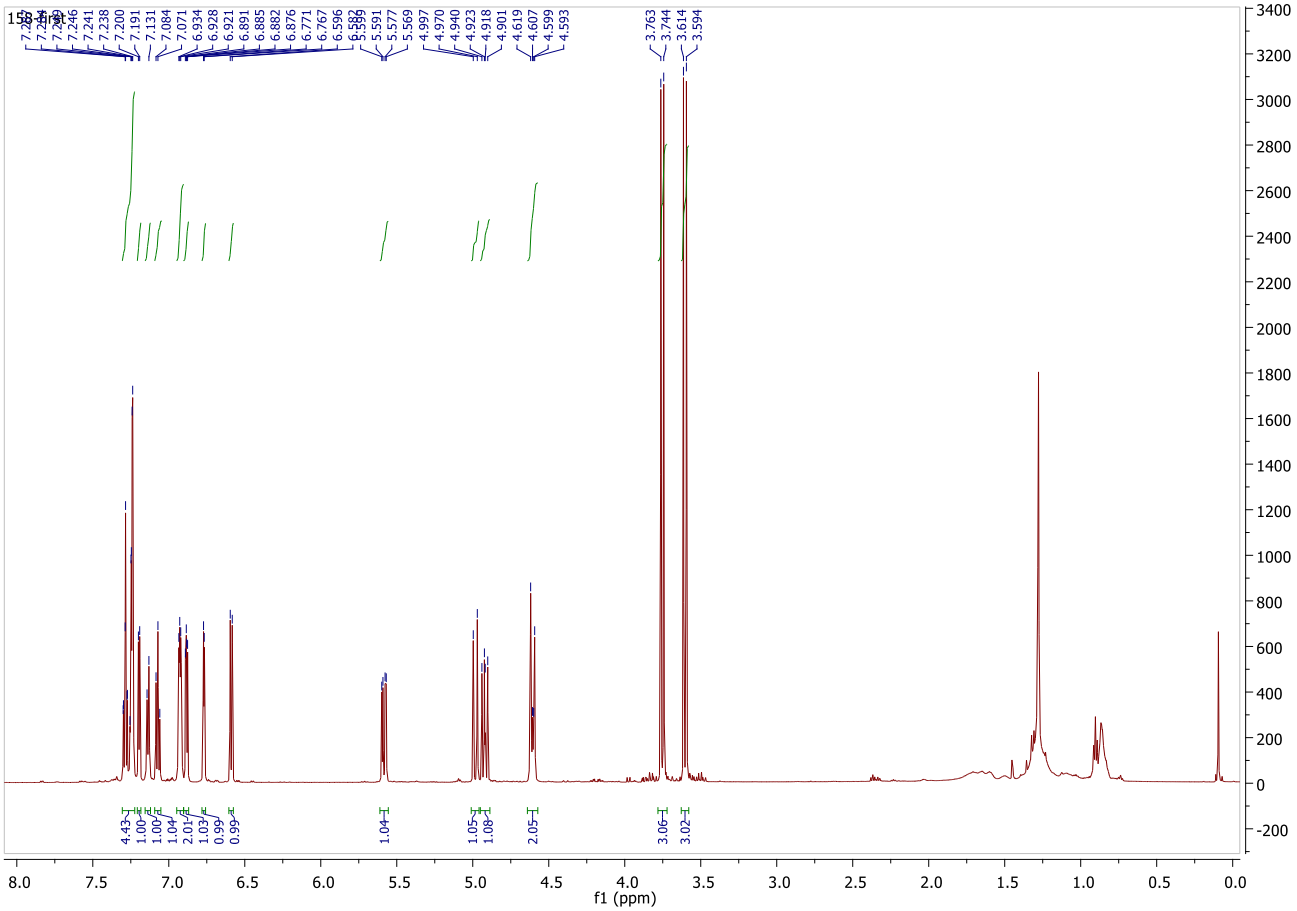

158-second
7.594
7.583
7.572
7.571
7.284
7.257
7.255
7.249
7.246
6.983
6.764
6.438
6.437
6.444
5.631
5.622
5.608
5.599
5.276
5.261
4.824
4.771
4.762
4.757
4.748
4.688
4.662
3.760
3.741
3.591
3.572
1.00
2.87
1.08
1.05
0.95
2.00
0.98
1.00
1.04
1.06
1.06
1.10
1.03
1.09
2.95
2.91
f1 (ppm)

^{1}H NMR of 4ak

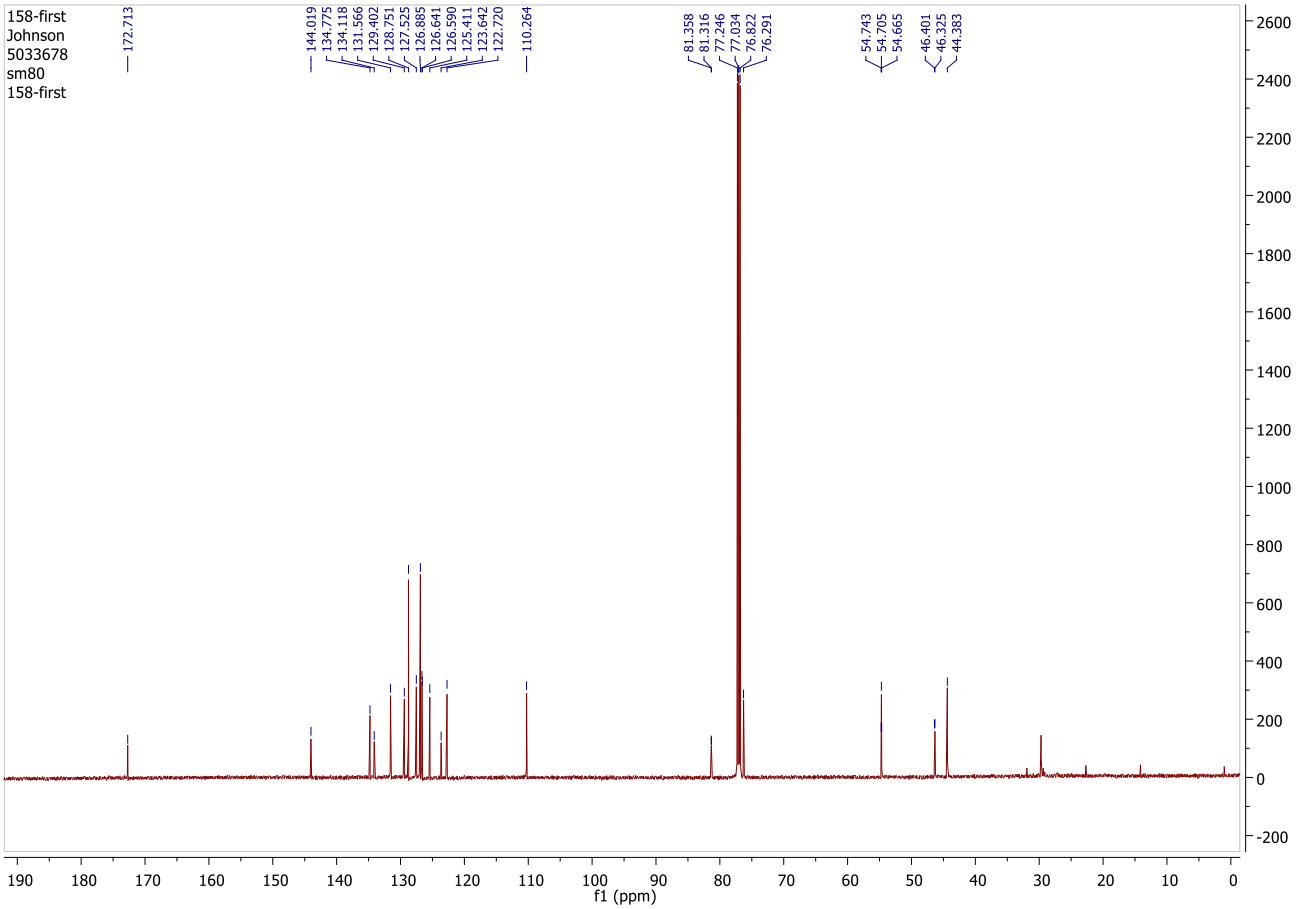


^{13}C NMR of 4ak

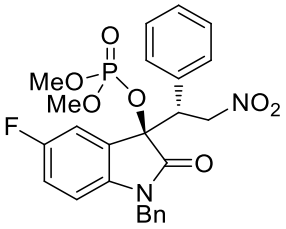


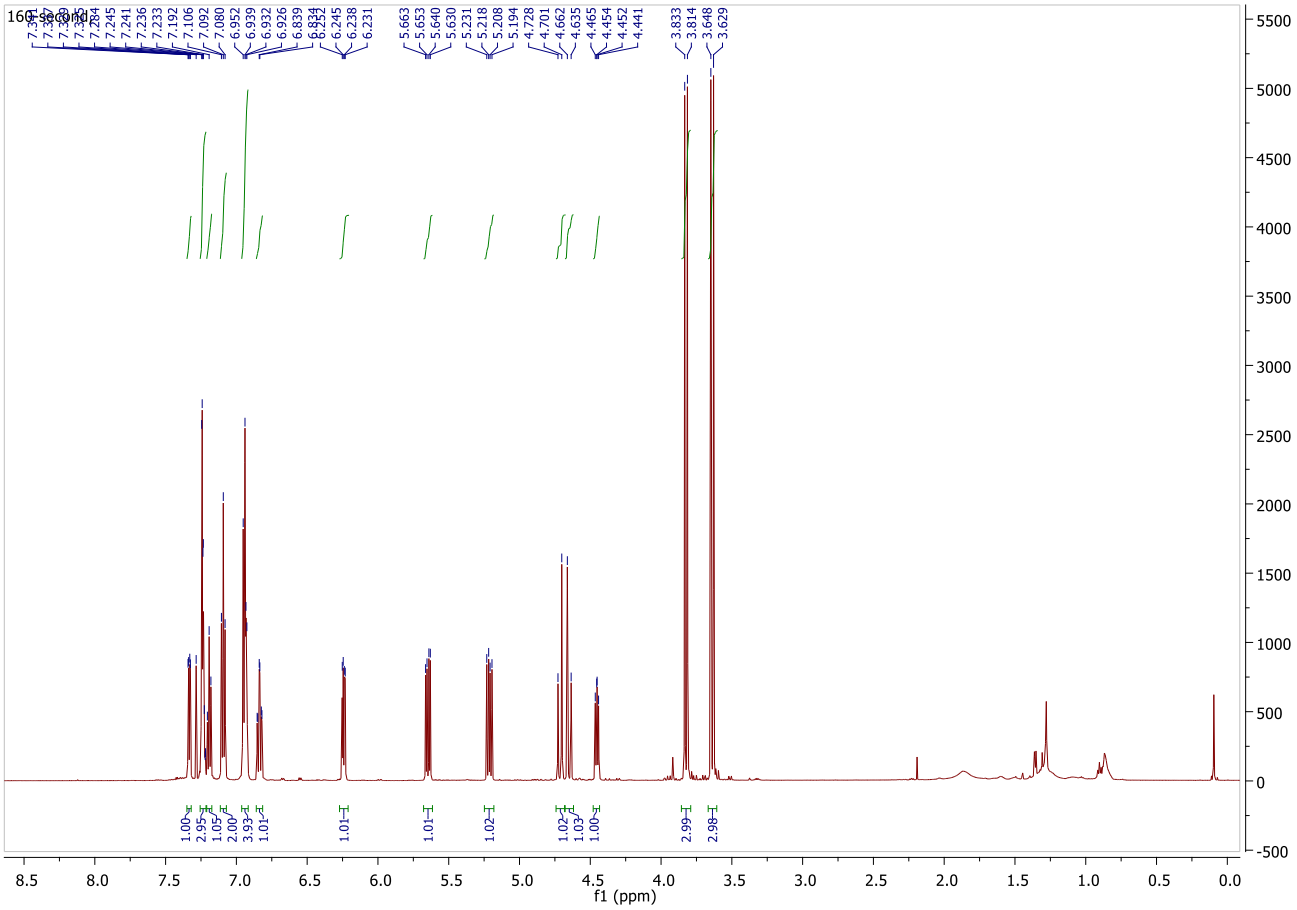


^{1}H NMR of 4ak (2nd diastereomer)

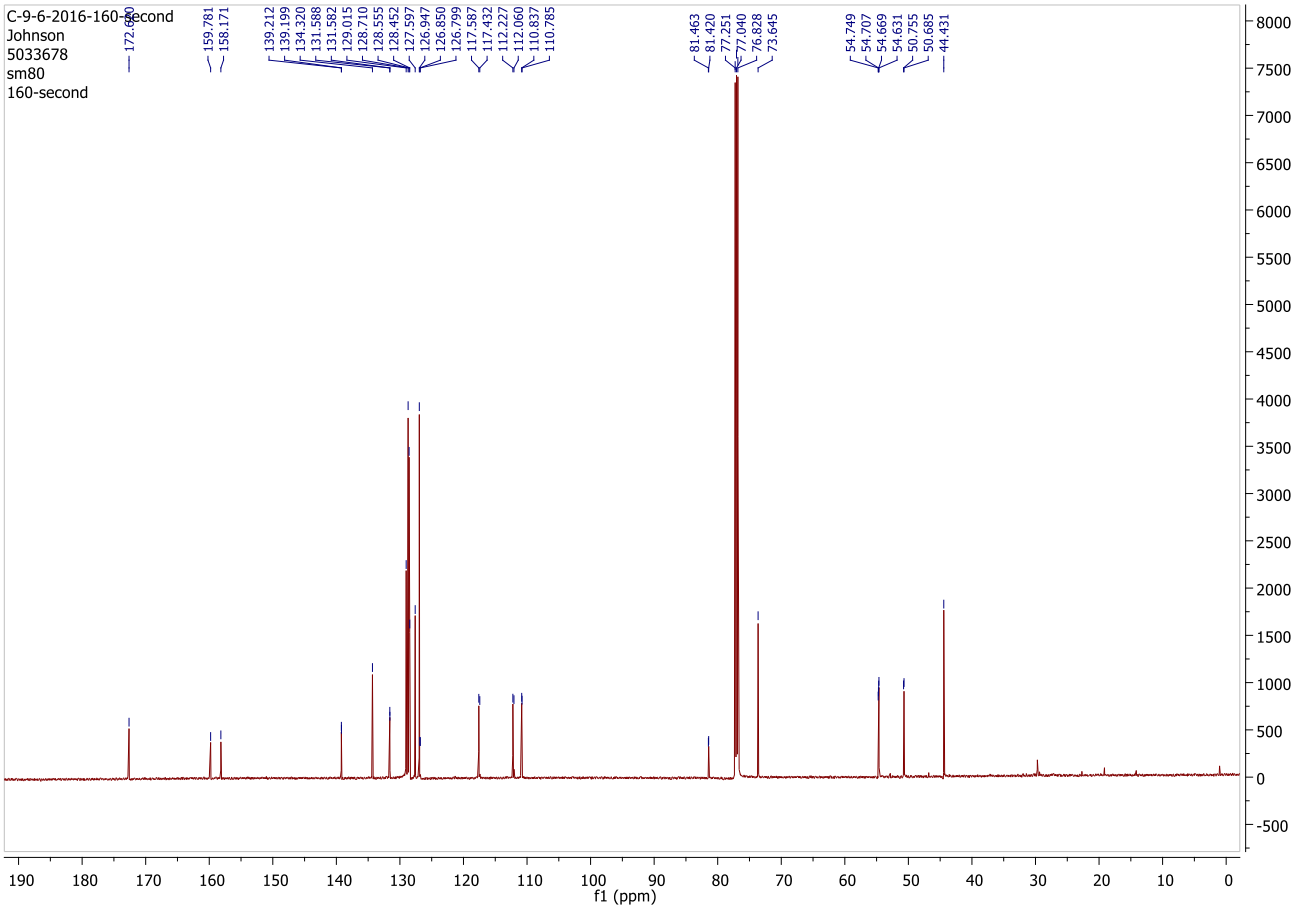


^{13}C NMR of 4ak (2nd diastereomer)

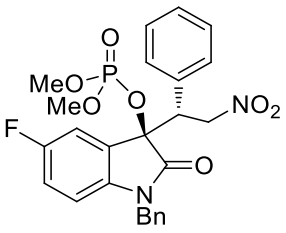

O
MeO
MeO
P
O
NO2
F
O
N
Bn

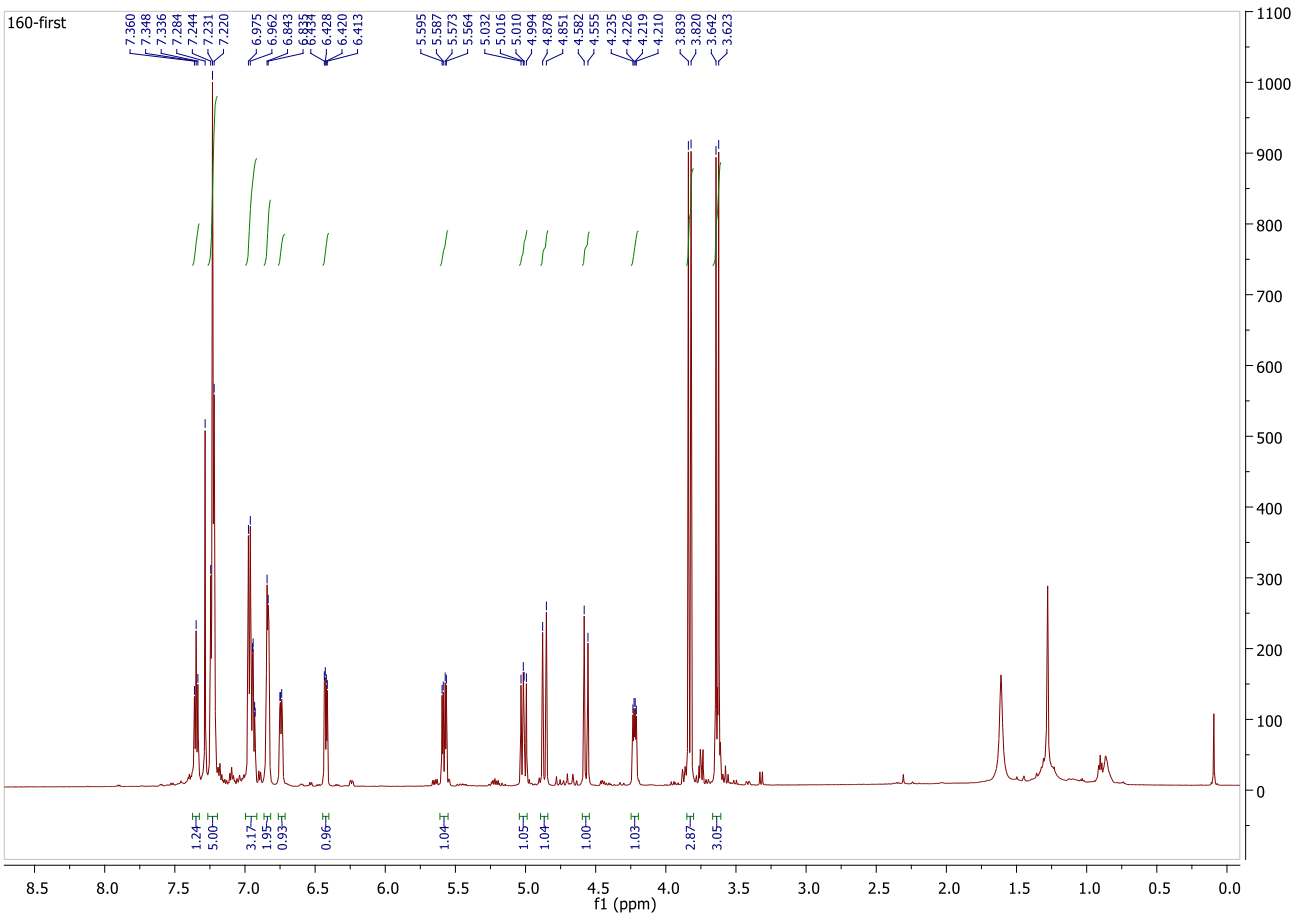

160 second
7.347
7.339
7.284
7.245
7.241
7.236
7.233
7.192
7.106
7.092
7.080
6.952
6.939
6.932
6.926
6.839
6.834
6.252
6.245
6.238
6.231
5.663
5.653
5.640
5.630
5.231
5.218
5.208
5.194
4.728
4.701
4.662
4.635
4.465
4.454
4.452
4.441
3.833
3.814
3.648
3.629
1.00
2.95
1.05
2.00
3.93
1.01
1.01
1.01
1.02
1.02
1.03
1.00
2.99
2.98
f1 (ppm)

^{1}H NMR of 4ba

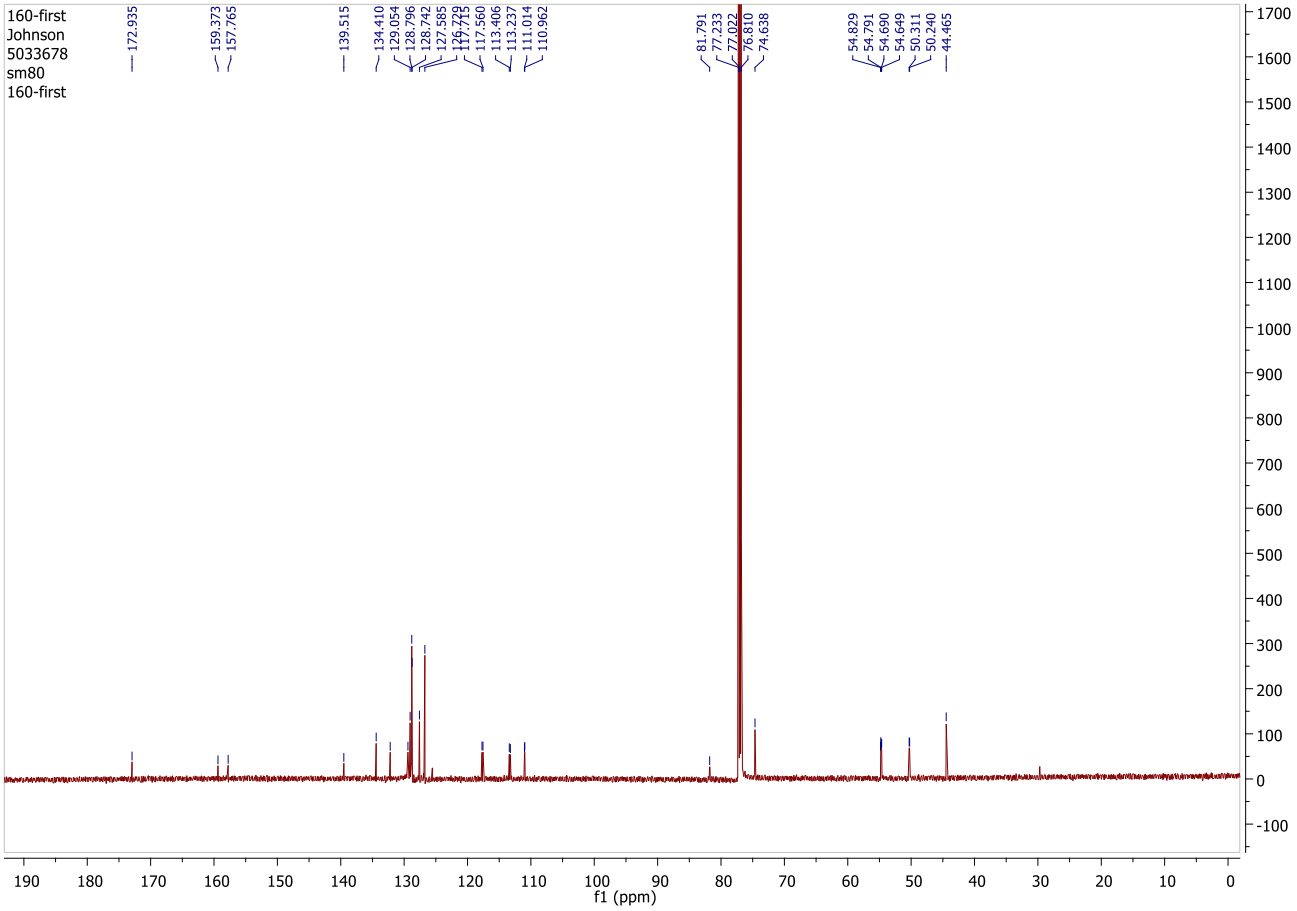


^{13}C NMR of 4ba

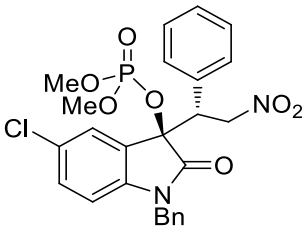


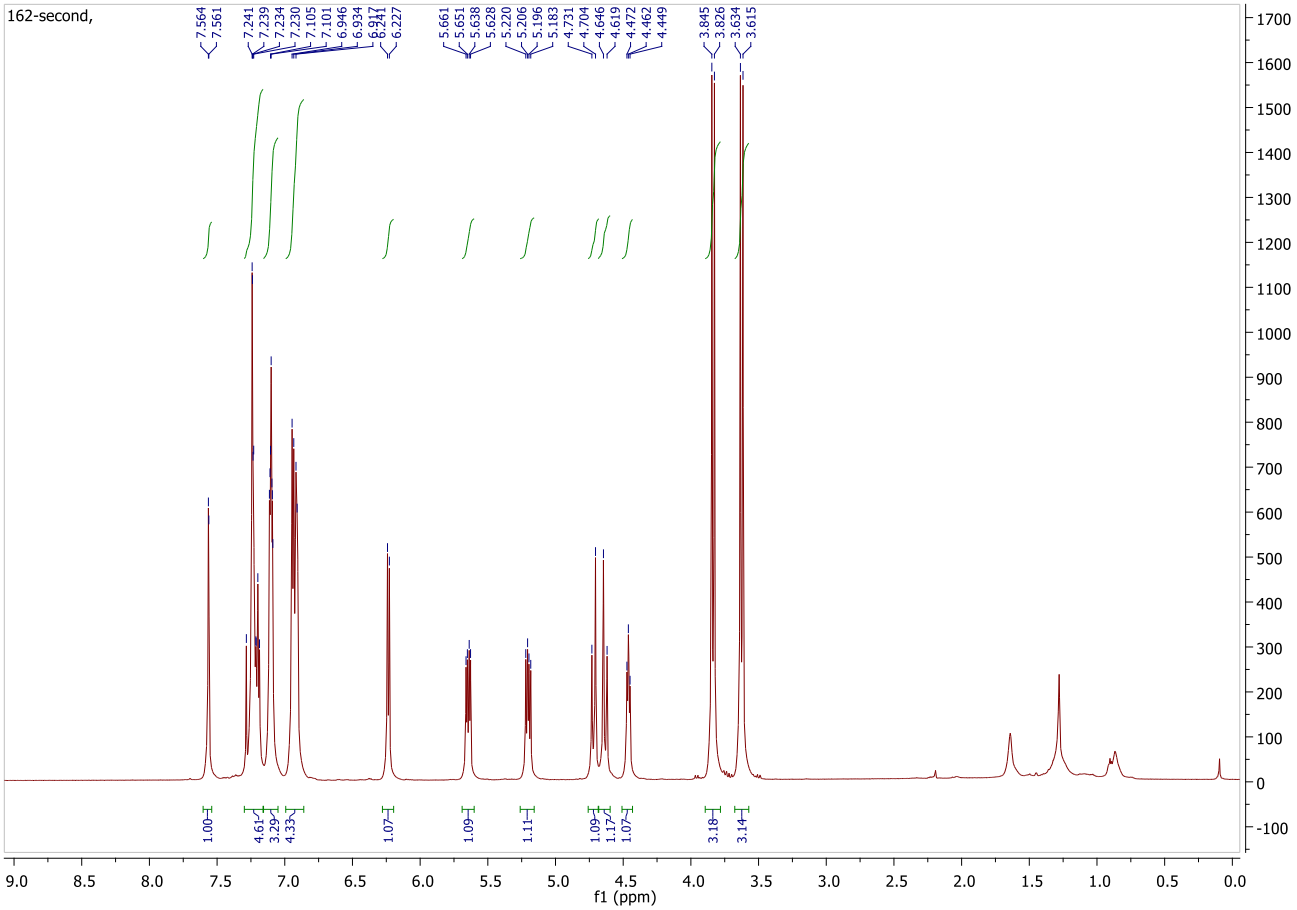


^{1}H NMR of 4ba (2nd diastereomer)

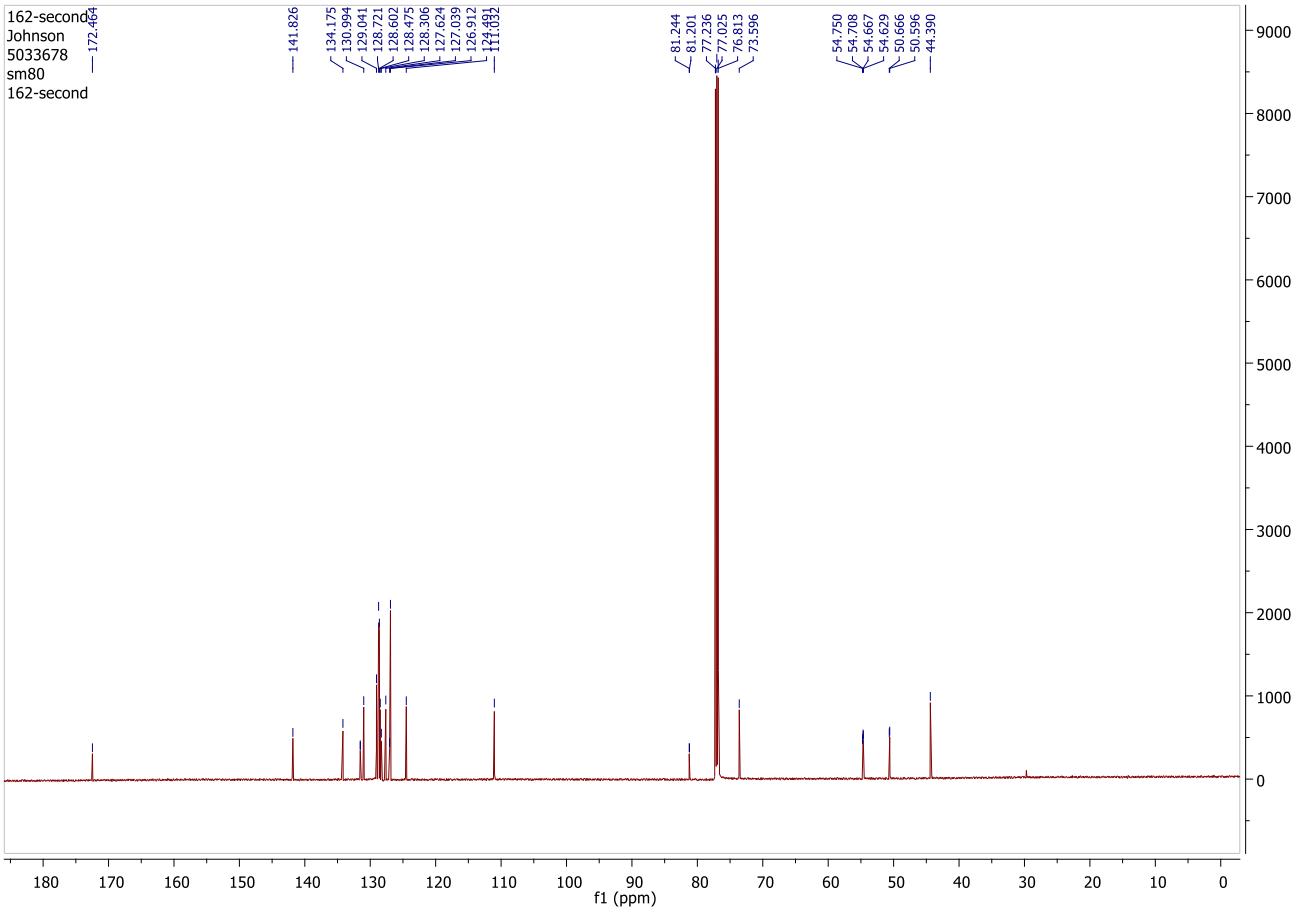


^{13}C NMR of 4ba (2nd diastereomer)

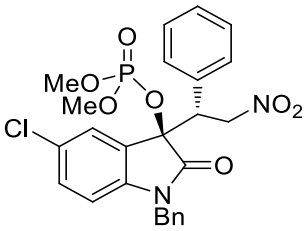

O
MeO
P
MeO
O
NO2
Cl
O
N
Bn

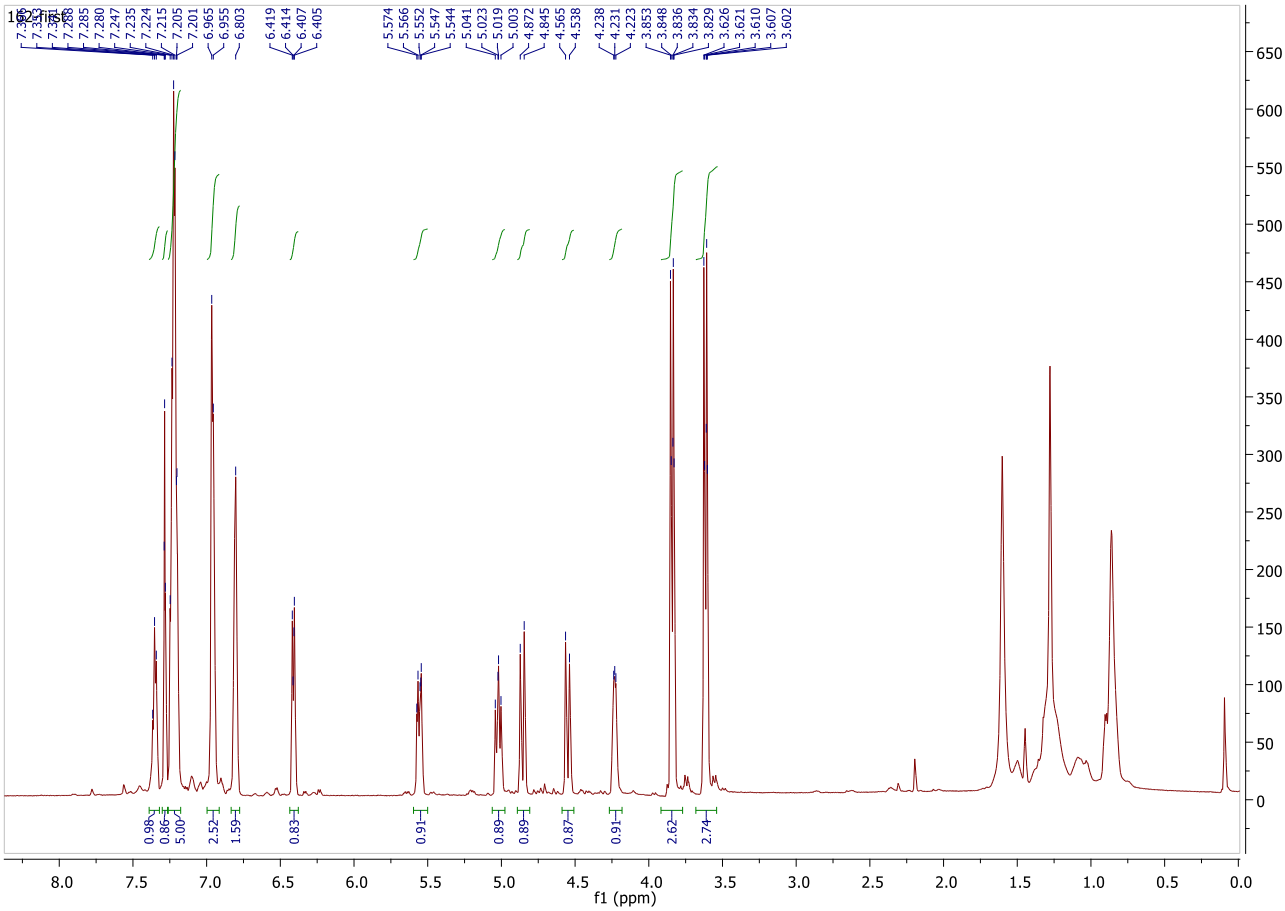

162-second,
7.564
7.561
7.241
7.239
7.234
7.230
7.105
7.101
6.946
6.934
6.241
6.227
5.661
5.651
5.638
5.628
5.220
5.206
5.196
5.183
4.731
4.704
4.646
4.619
4.472
4.462
4.449
3.845
3.826
3.634
3.615
1.00
4.61
3.29
4.33
1.07
1.09
1.11
1.09
1.17
1.07
3.18
3.14
f1 (ppm)

^{1}H NMR of 4ca

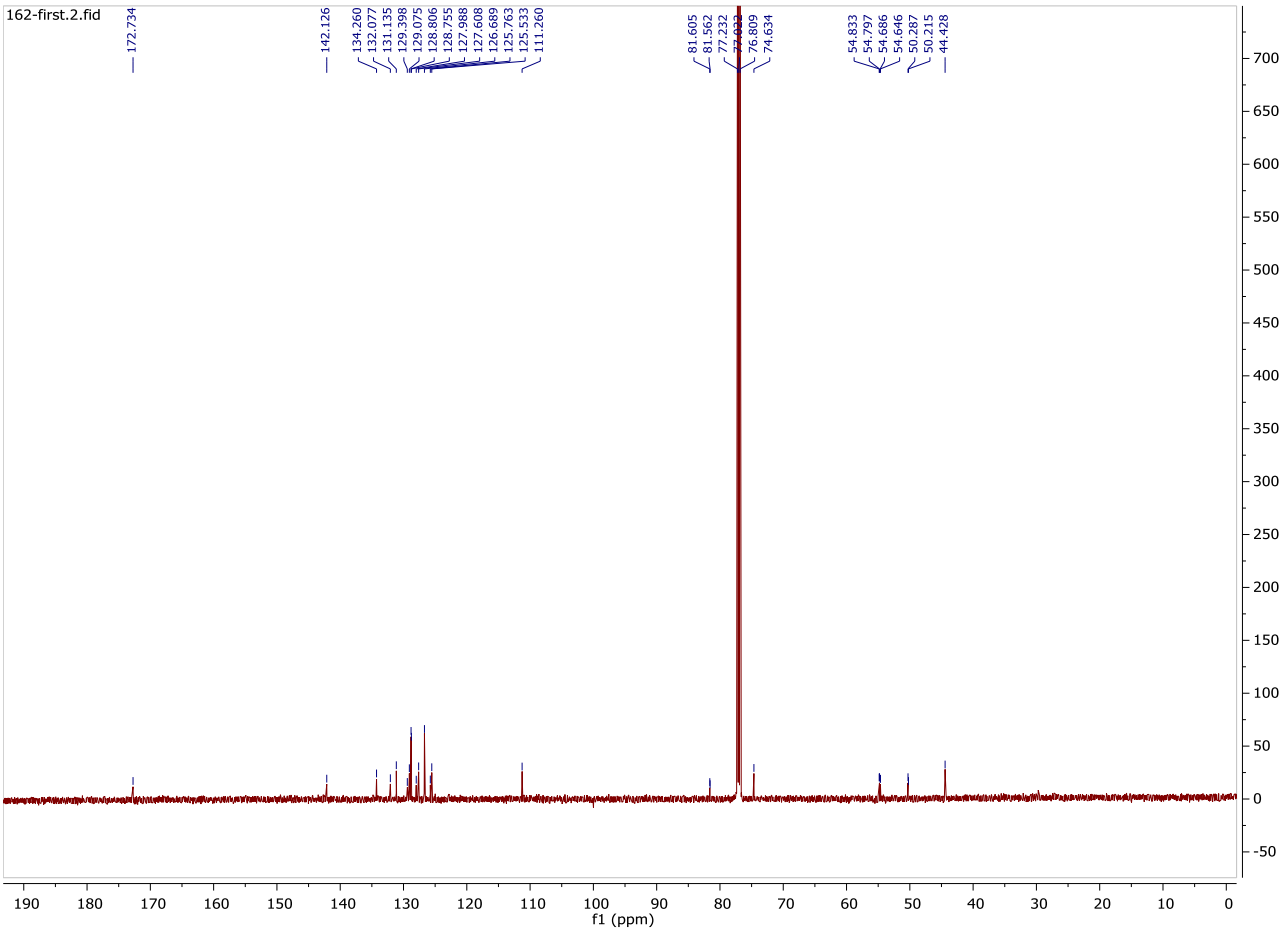


^{13}C NMR of 4ca

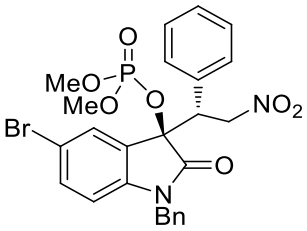


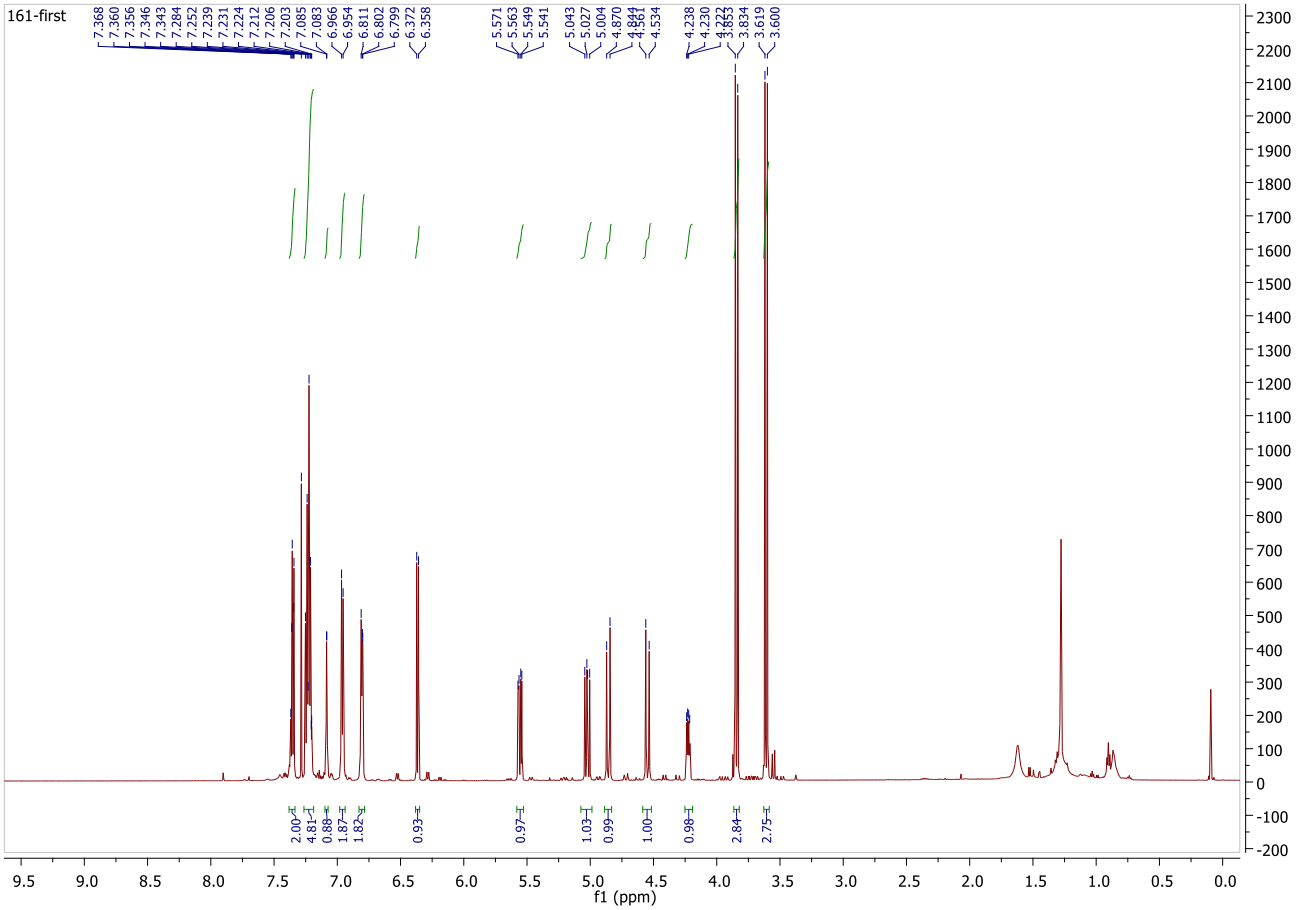


^{1}H NMR of 4ca (2nd diastereomer)

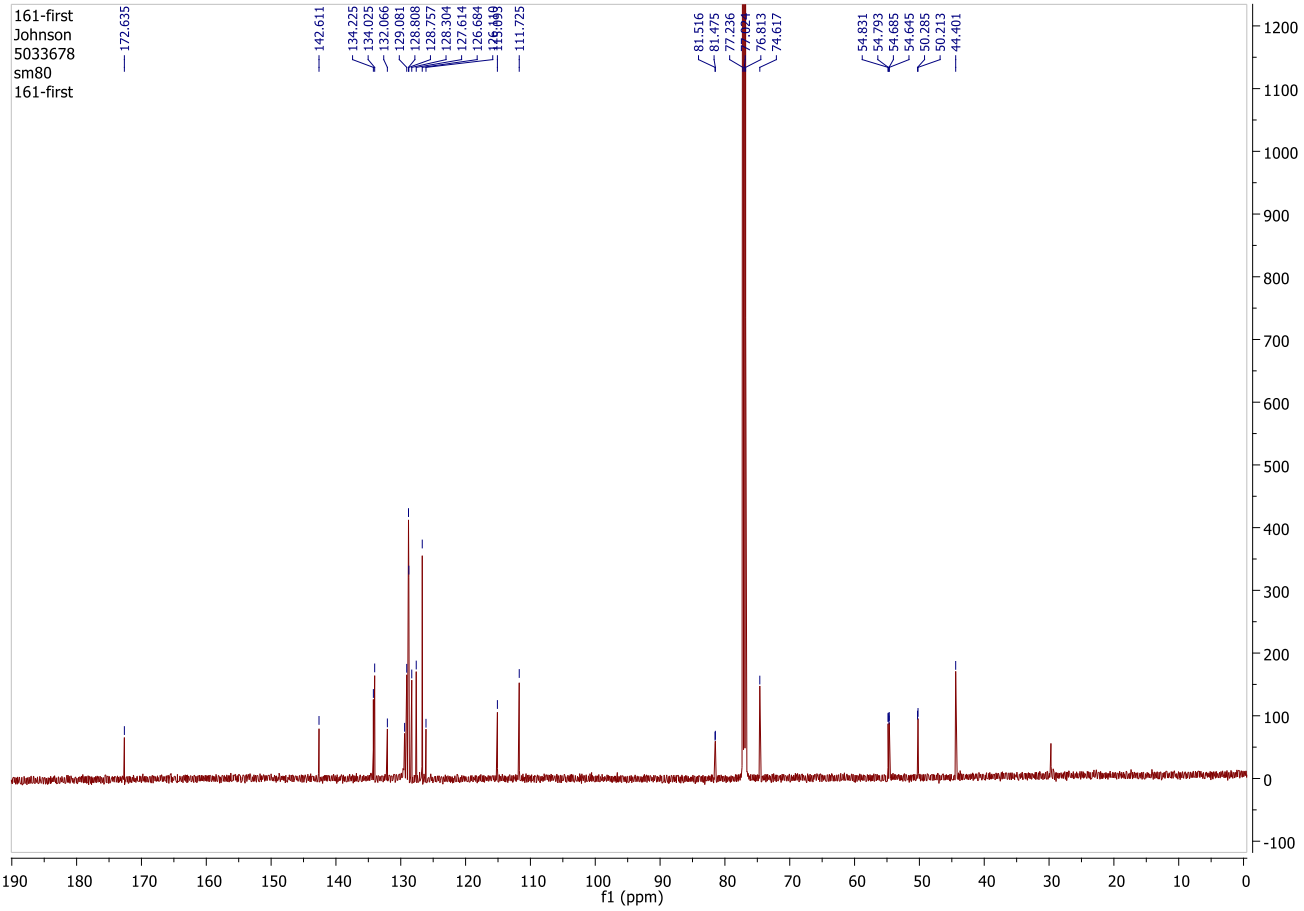


^{13}C NMR of 4ca (2nd diastereomer)

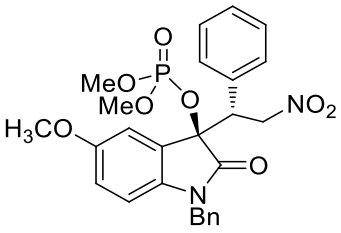

O
MeO
P
MeO
O
NO2
Br
O
N
Bn

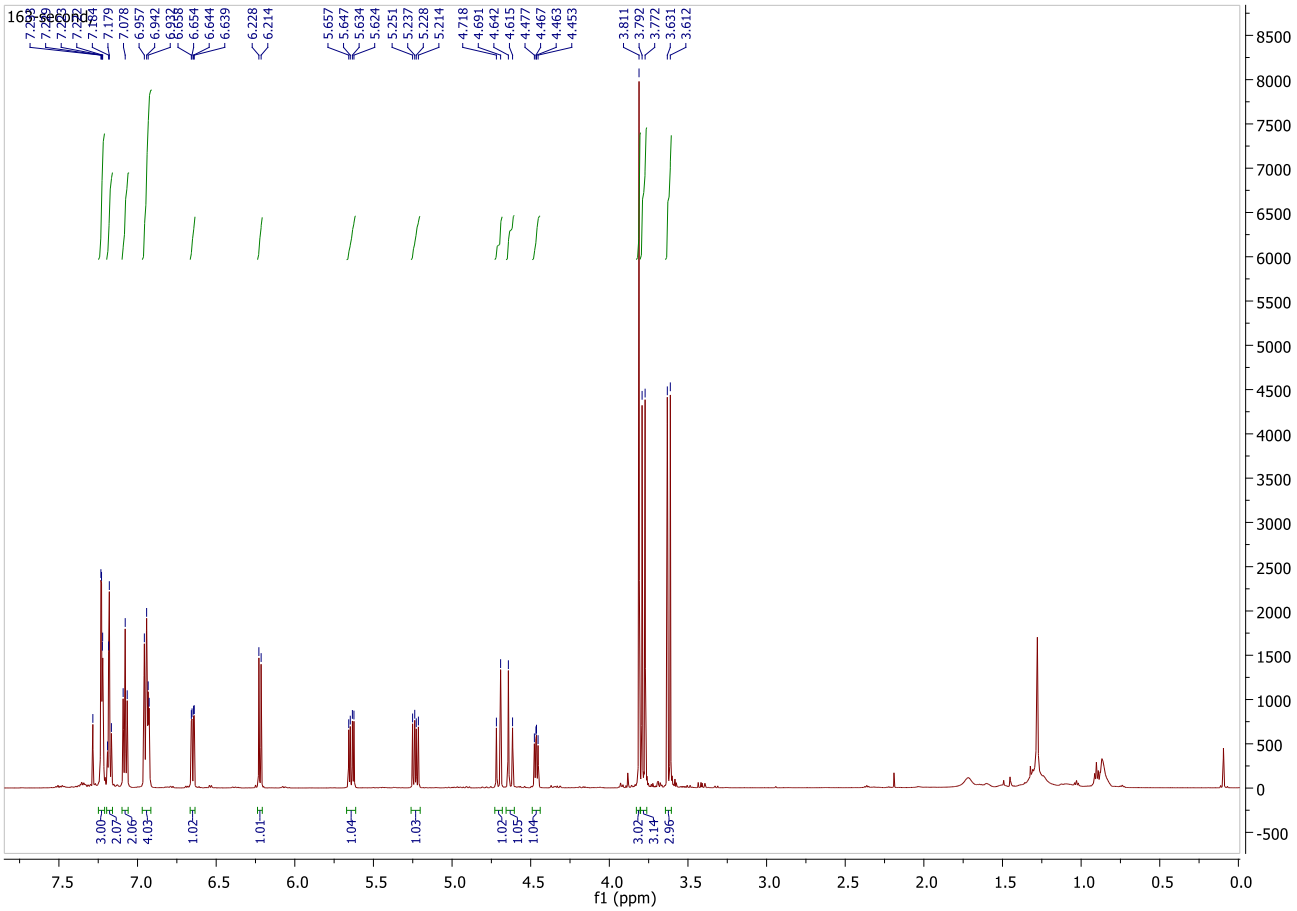

161-first
7.368
7.360
7.356
7.346
7.343
7.284
7.252
7.239
7.231
7.224
7.212
7.206
7.203
7.085
7.083
6.966
6.954
6.811
6.802
6.799
6.372
6.358
5.571
5.563
5.549
5.541
5.043
5.027
5.004
4.870
4.844
4.561
4.534
4.238
4.230
4.222
3.853
3.834
3.619
3.600
2.00
4.81
0.88
1.87
1.82
0.93
0.97
1.03
0.99
1.00
0.98
2.84
2.75
f1 (ppm)

^{1}H NMR of 4da

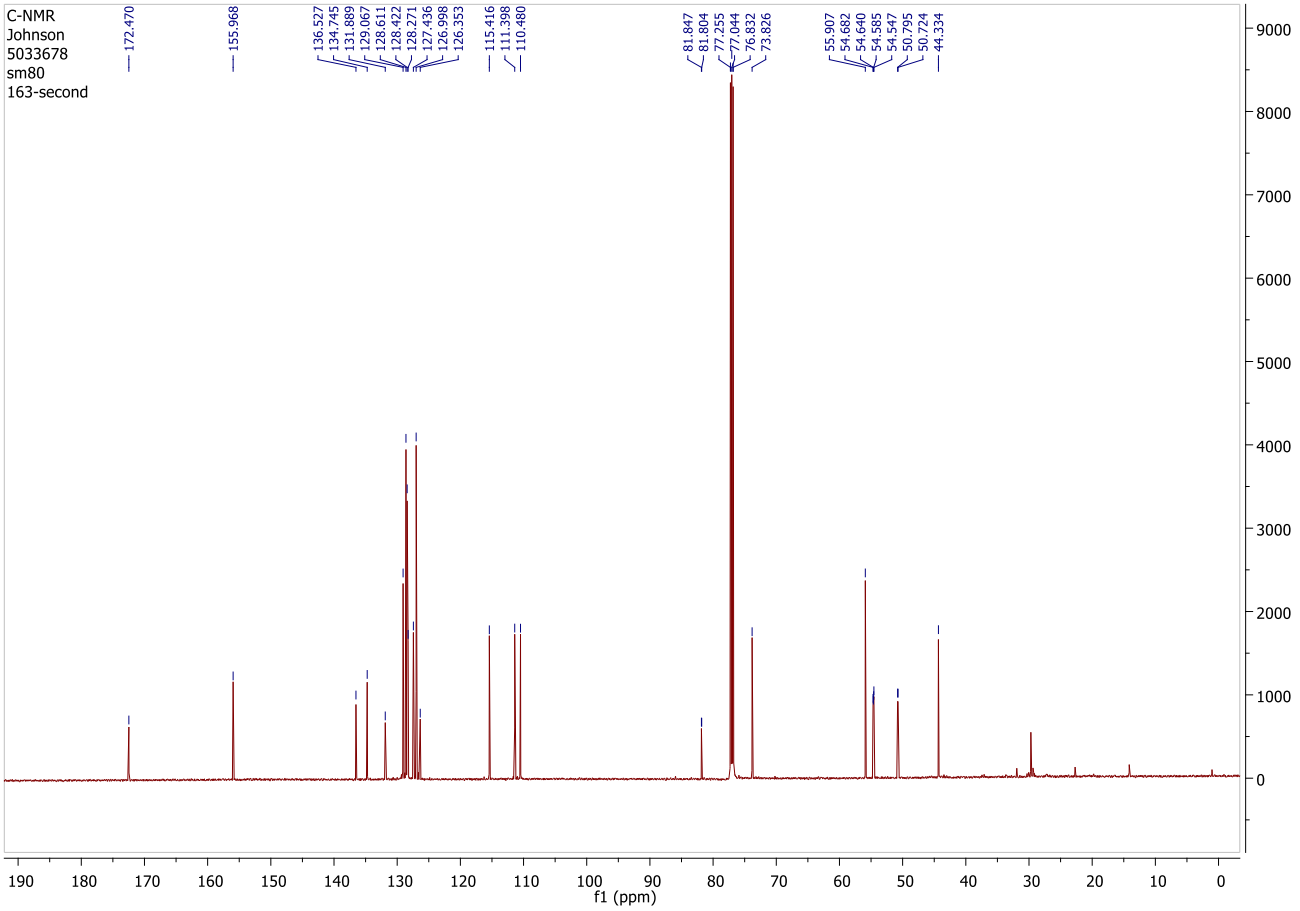


^{13}C NMR of 4da

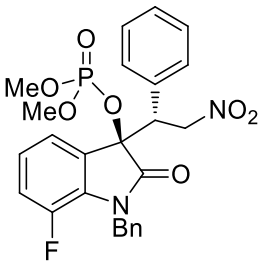

O
MeO
P
MeO
O
NO2
H3CO
O
N
Bn

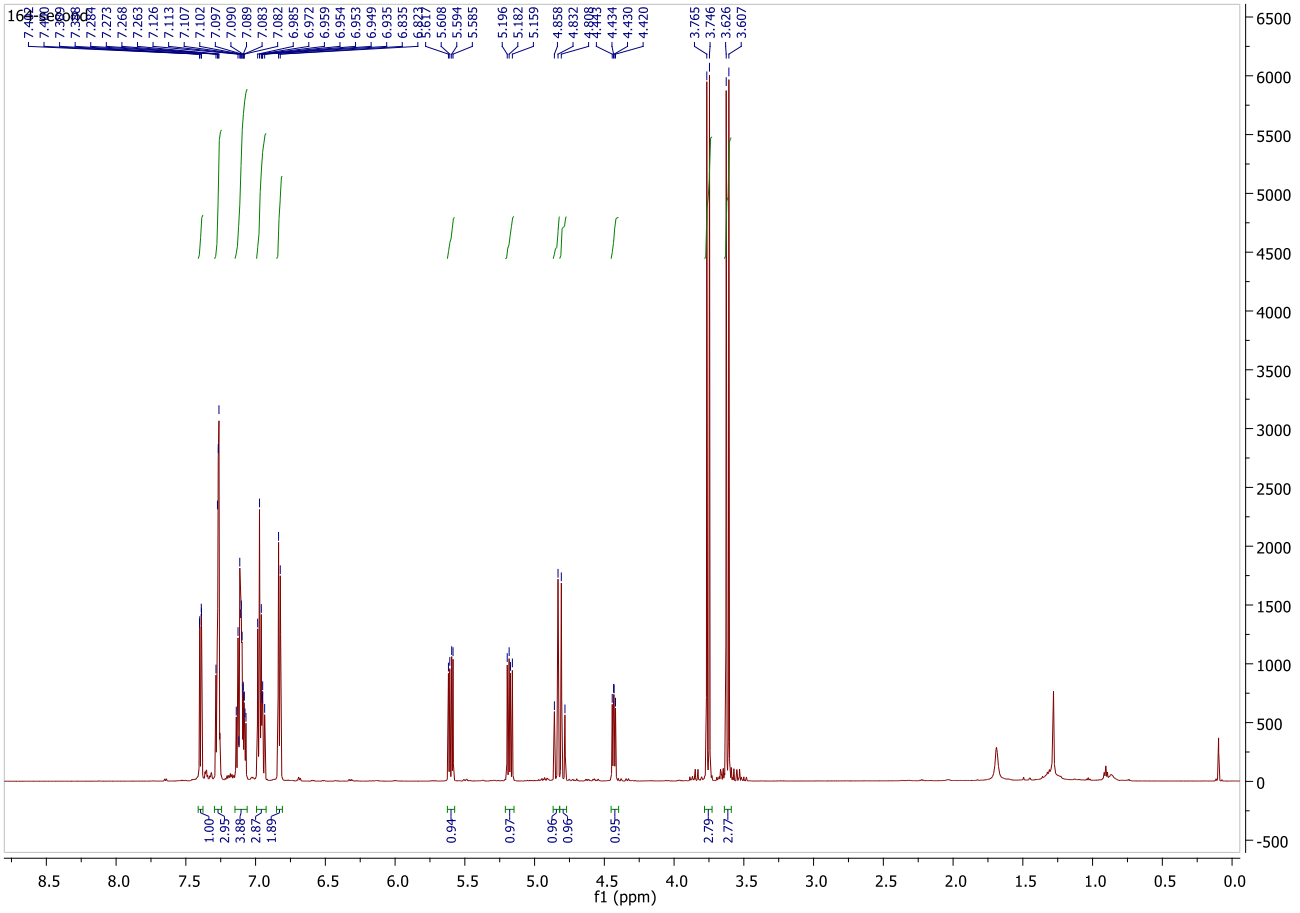

160 seconds
7.264
7.260
7.256
7.252
7.248
7.244
7.179
7.078
6.957
6.942
6.932
6.858
6.654
6.644
6.639
6.228
6.214
5.657
5.647
5.634
5.624
5.251
5.237
5.228
5.214
4.718
4.691
4.642
4.615
4.477
4.467
4.463
4.453
3.811
3.792
3.772
3.631
3.612
3.00
2.07
2.06
4.03
1.02
1.01
1.04
1.03
1.02
1.05
1.04
3.02
3.14
2.96
f1 (ppm)

^{1}H NMR of 4ea

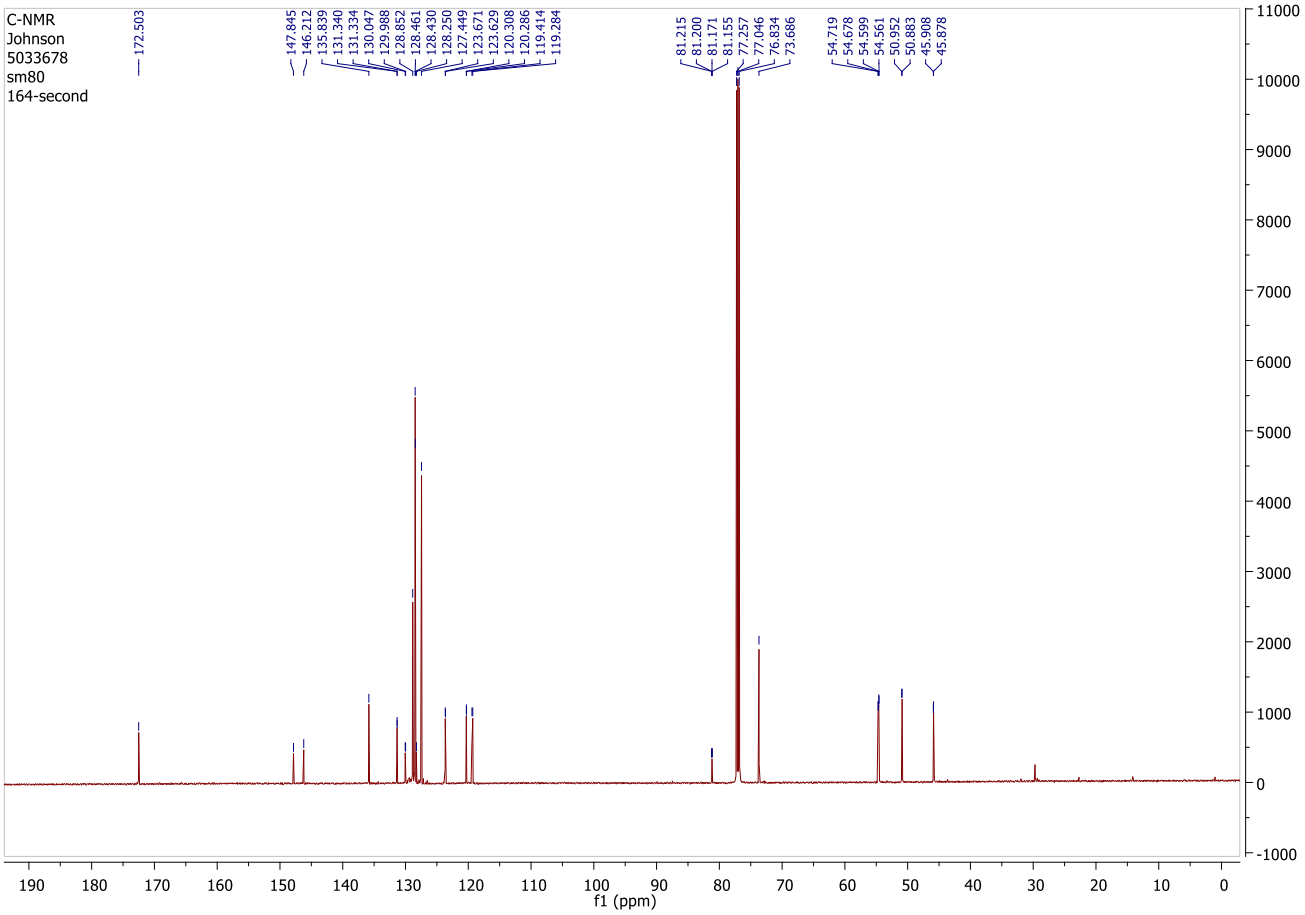


^{13}C NMR of 4ea

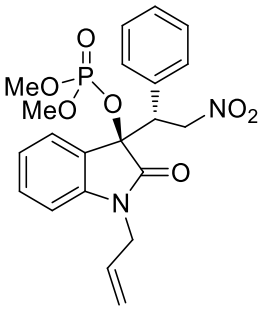

O
MeO
P
MeO
O
NO2
O
N
Bn
F

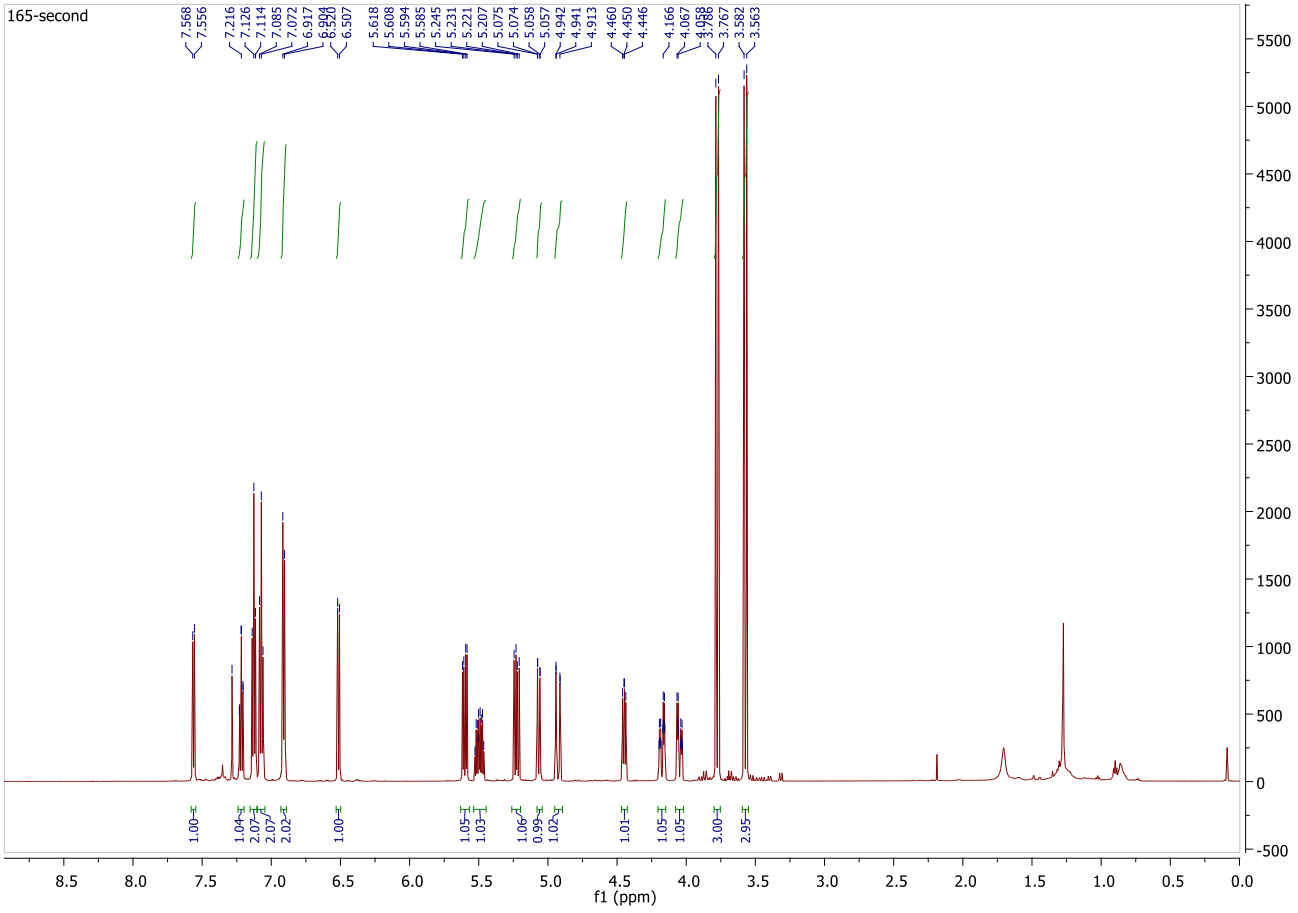

164 scans
7.460
7.273
7.268
7.263
7.126
7.113
7.107
7.102
7.097
7.090
7.089
7.083
7.082
6.985
6.972
6.959
6.954
6.953
6.949
6.935
6.835
5.608
5.594
5.585
5.196
5.182
5.159
4.858
4.832
4.434
4.430
4.420
3.765
3.746
3.626
3.607
1.00
2.95
3.88
2.87
1.89
0.94
0.97
0.96
0.96
0.95
2.79
2.77
f1 (ppm)

^{1}H NMR of 4fa

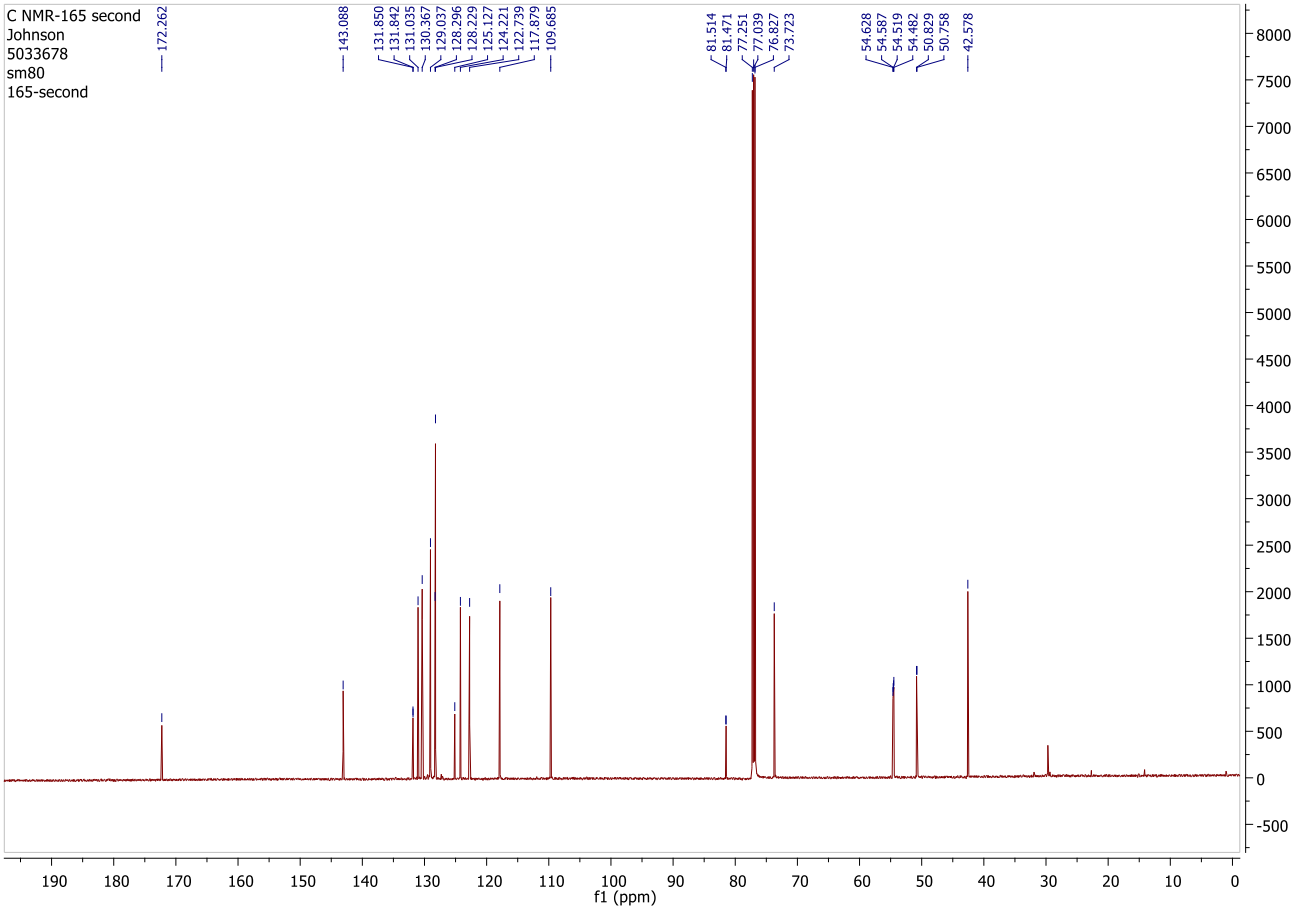


^{13}C NMR of 4fa

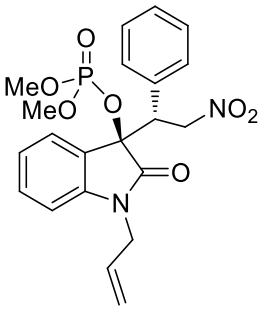

O
MeO
P
MeO
O
NO2
O
N

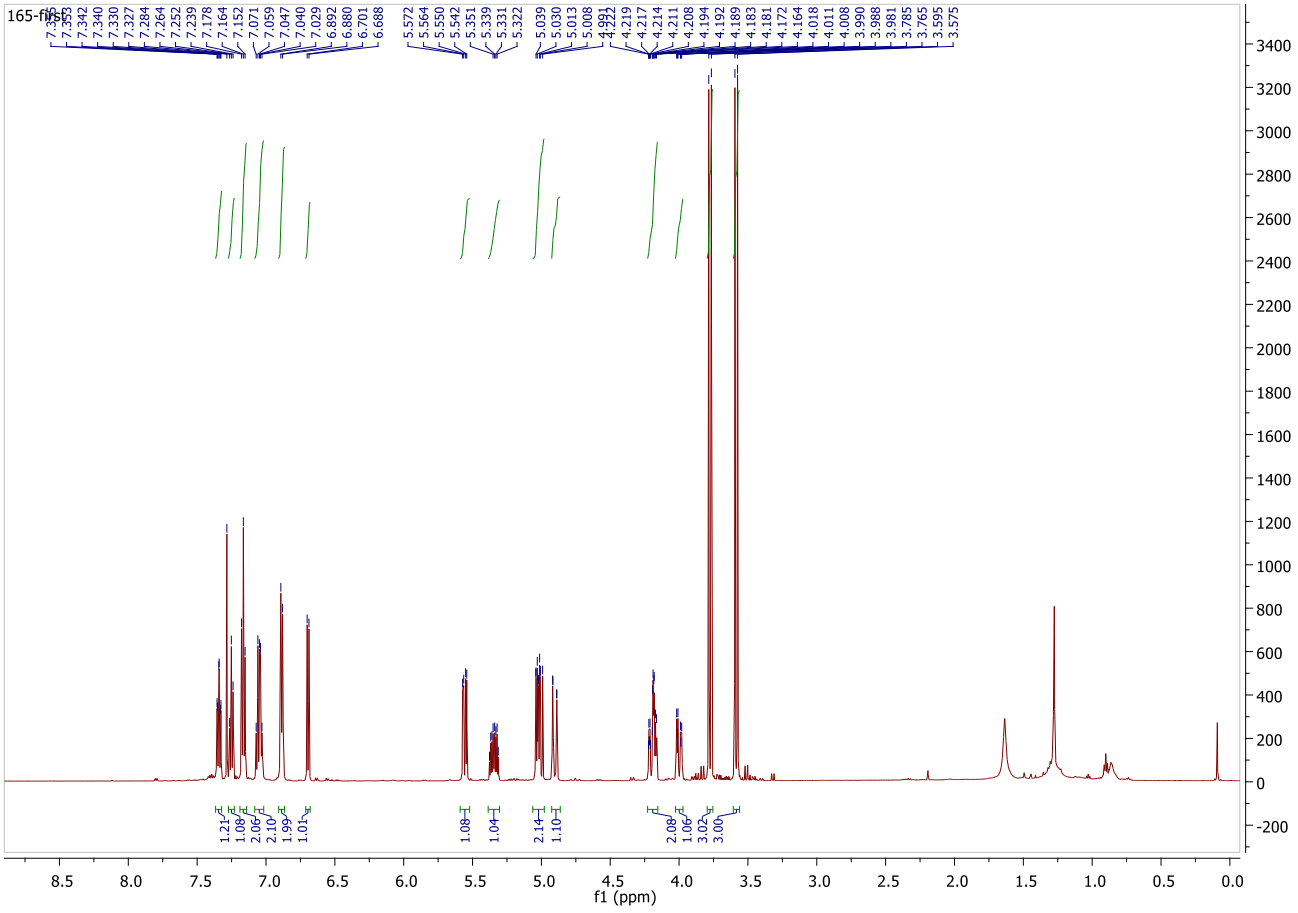

165-second
7.568
7.556
7.216
7.126
7.114
7.085
7.072
6.917
6.904
6.520
6.507
5.618
5.608
5.594
5.585
5.245
5.231
5.221
5.207
5.075
5.074
5.058
5.057
4.942
4.941
4.913
4.460
4.450
4.446
4.166
4.067
4.058
3.988
3.767
3.582
3.563
1.00
1.04
2.07
2.07
2.02
1.00
1.05
1.03
1.06
0.99
1.02
1.01
1.05
1.05
3.00
2.95
f1 (ppm)

[1]H NMR of 4ga

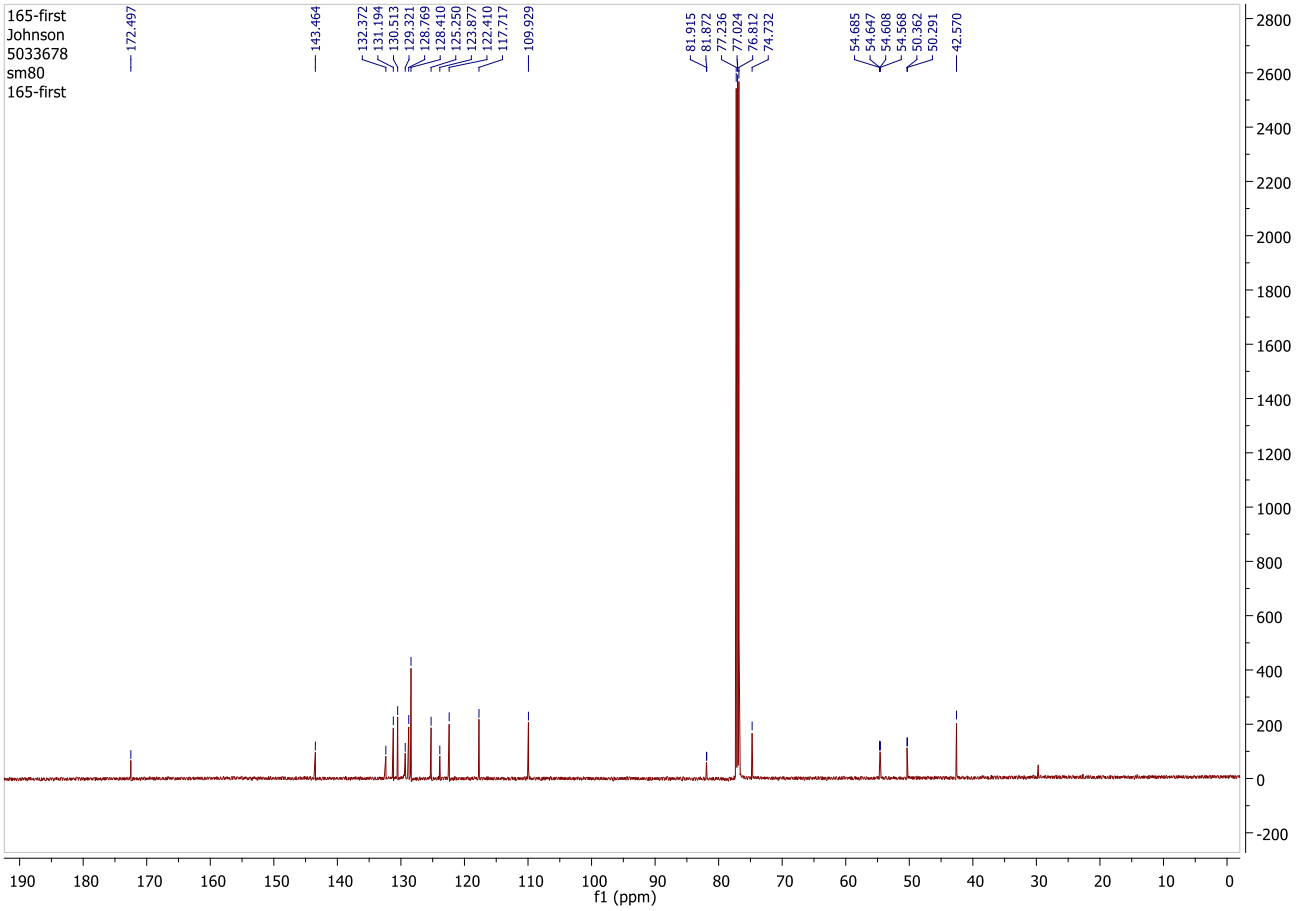


^{13}C NMR of 4ga

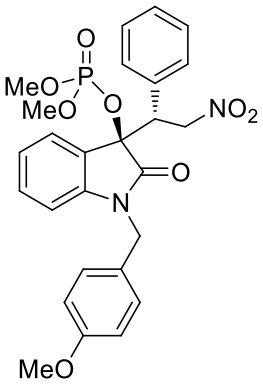


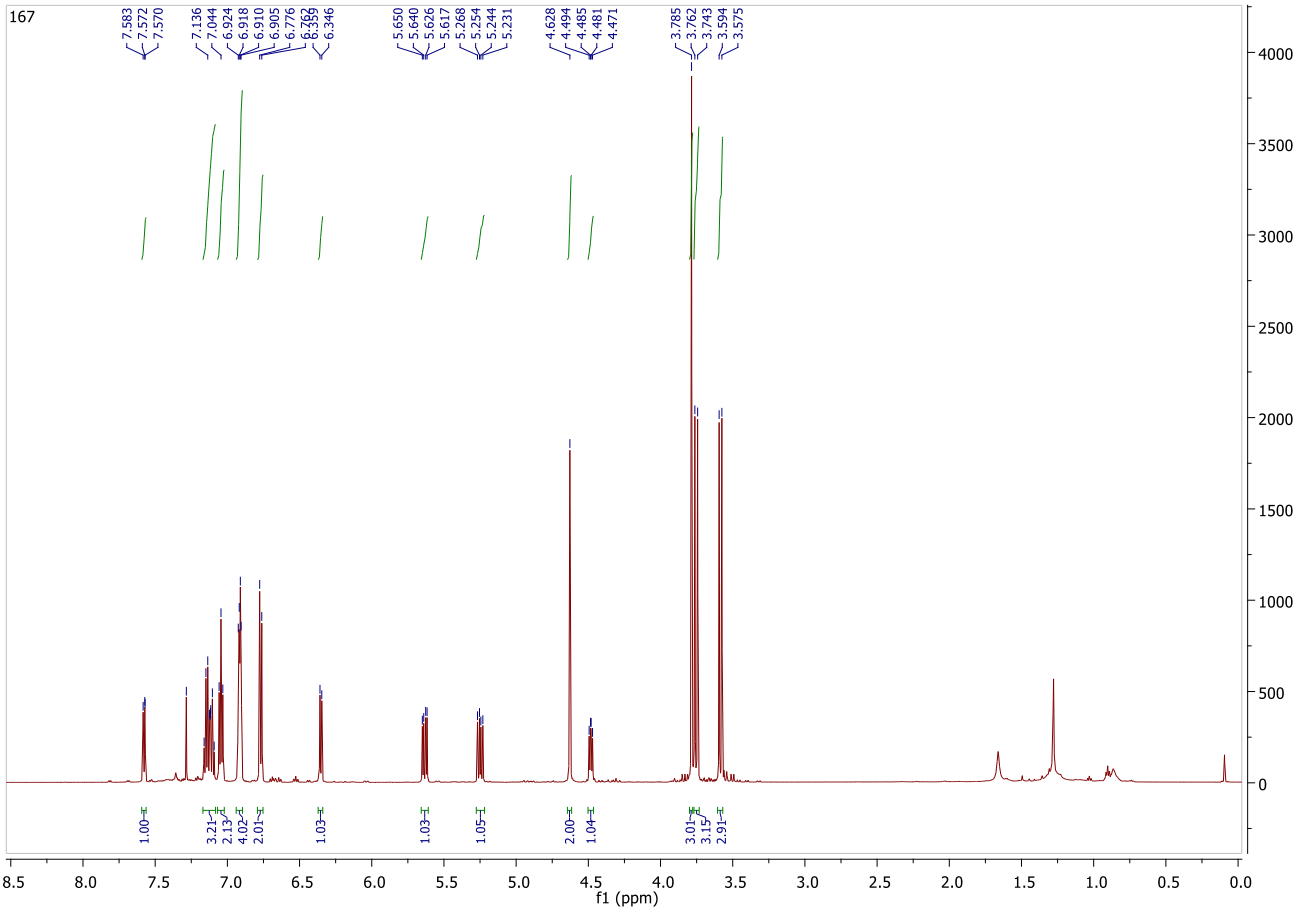


^{1}H NMR of 4ga (2nd diastereomer)

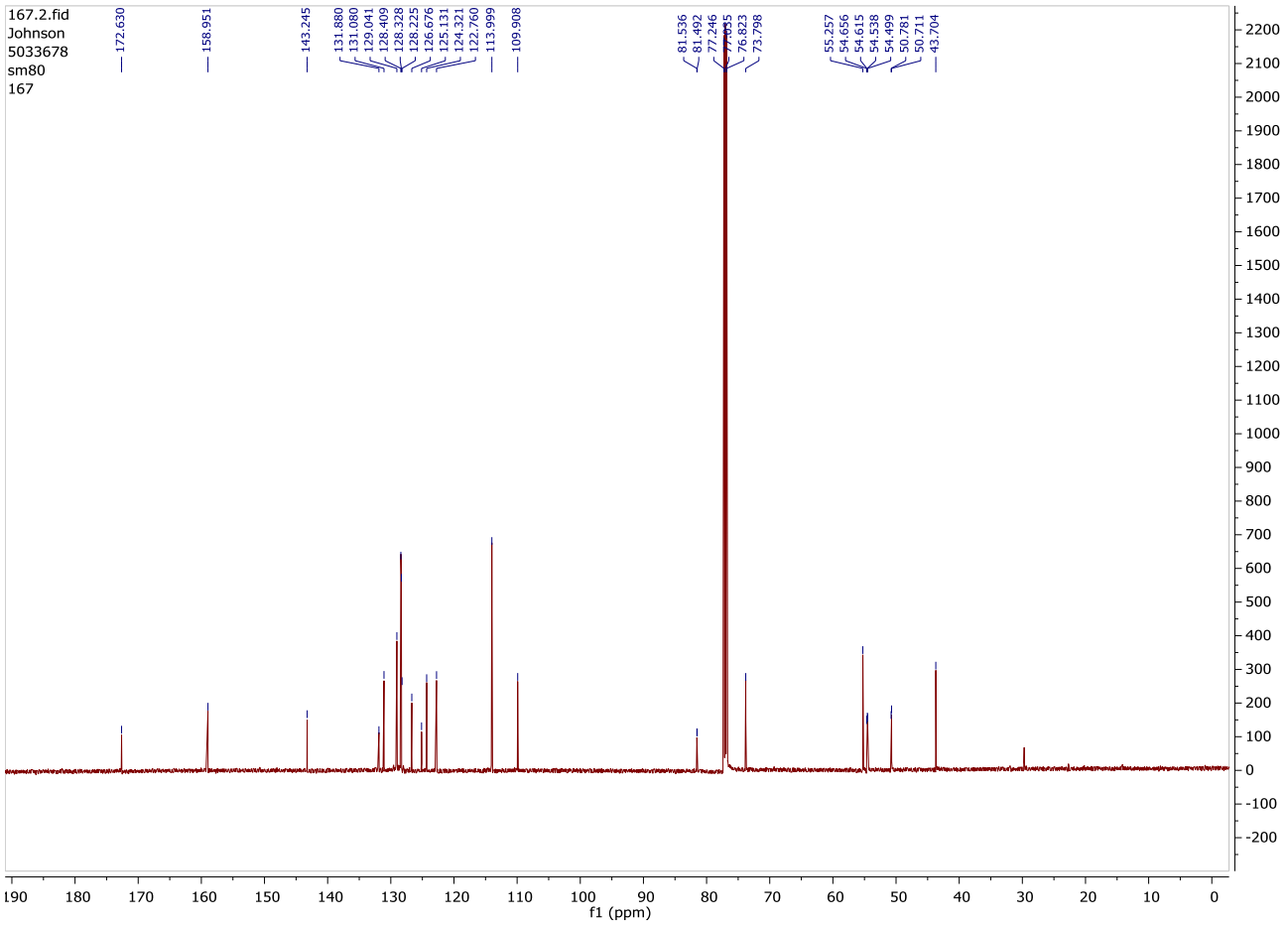


^{13}C NMR of 4ga (2nd diastereomer)

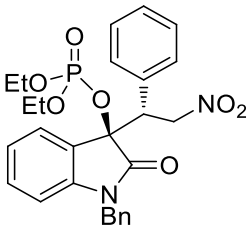

O
MeO
P
MeO
O
NO2
O
N
MeO

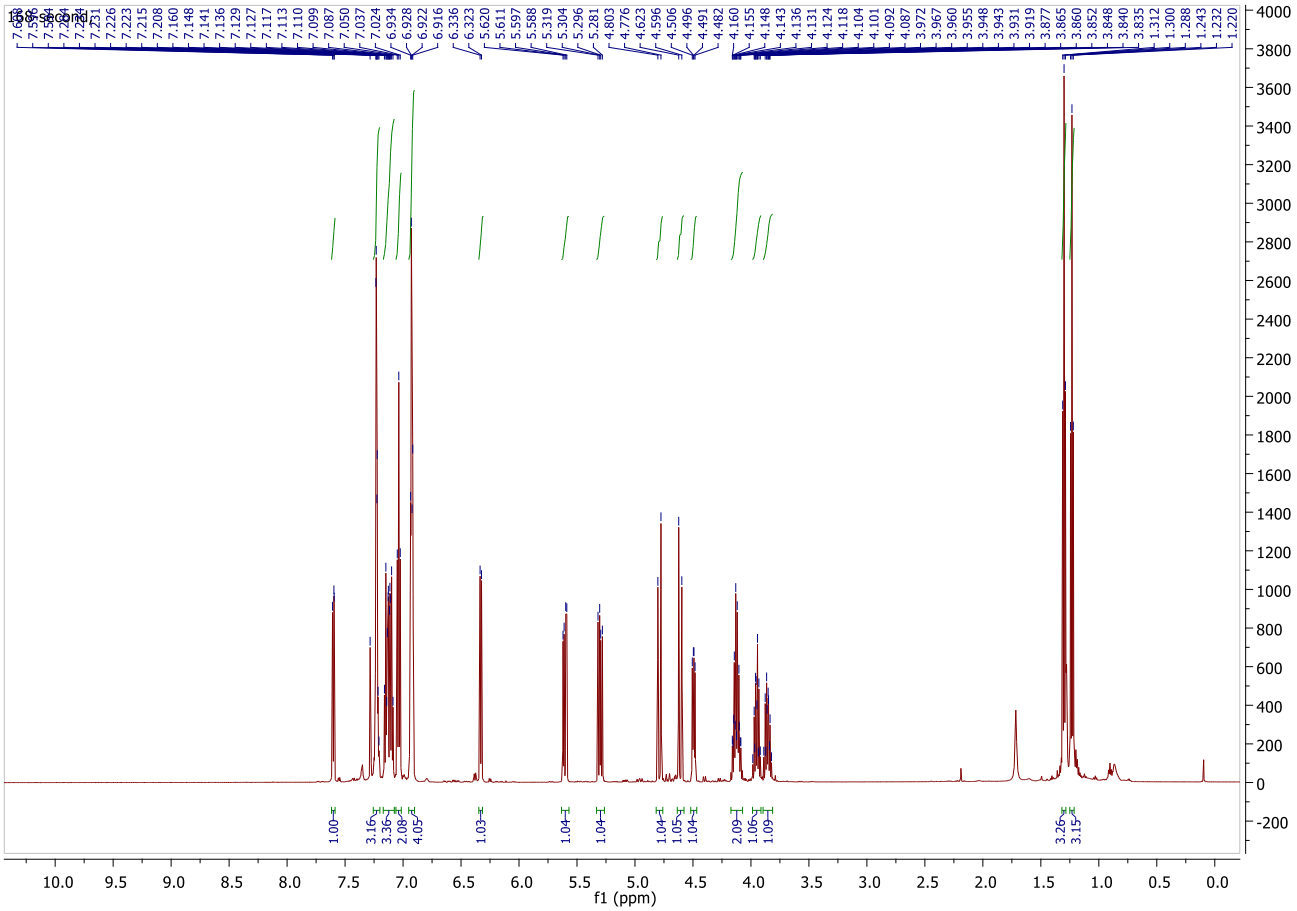

167
7.583
7.572
7.570
7.136
7.044
6.924
6.918
6.910
6.905
6.776
6.346
5.650
5.640
5.626
5.617
5.268
5.254
5.244
5.231
4.628
4.494
4.485
4.481
4.471
3.785
3.762
3.743
3.594
3.575
1.00
3.21
2.13
4.02
2.01
1.03
1.03
1.05
2.00
1.04
3.01
3.15
2.91
f1 (ppm)

^{1}H NMR of 4ha

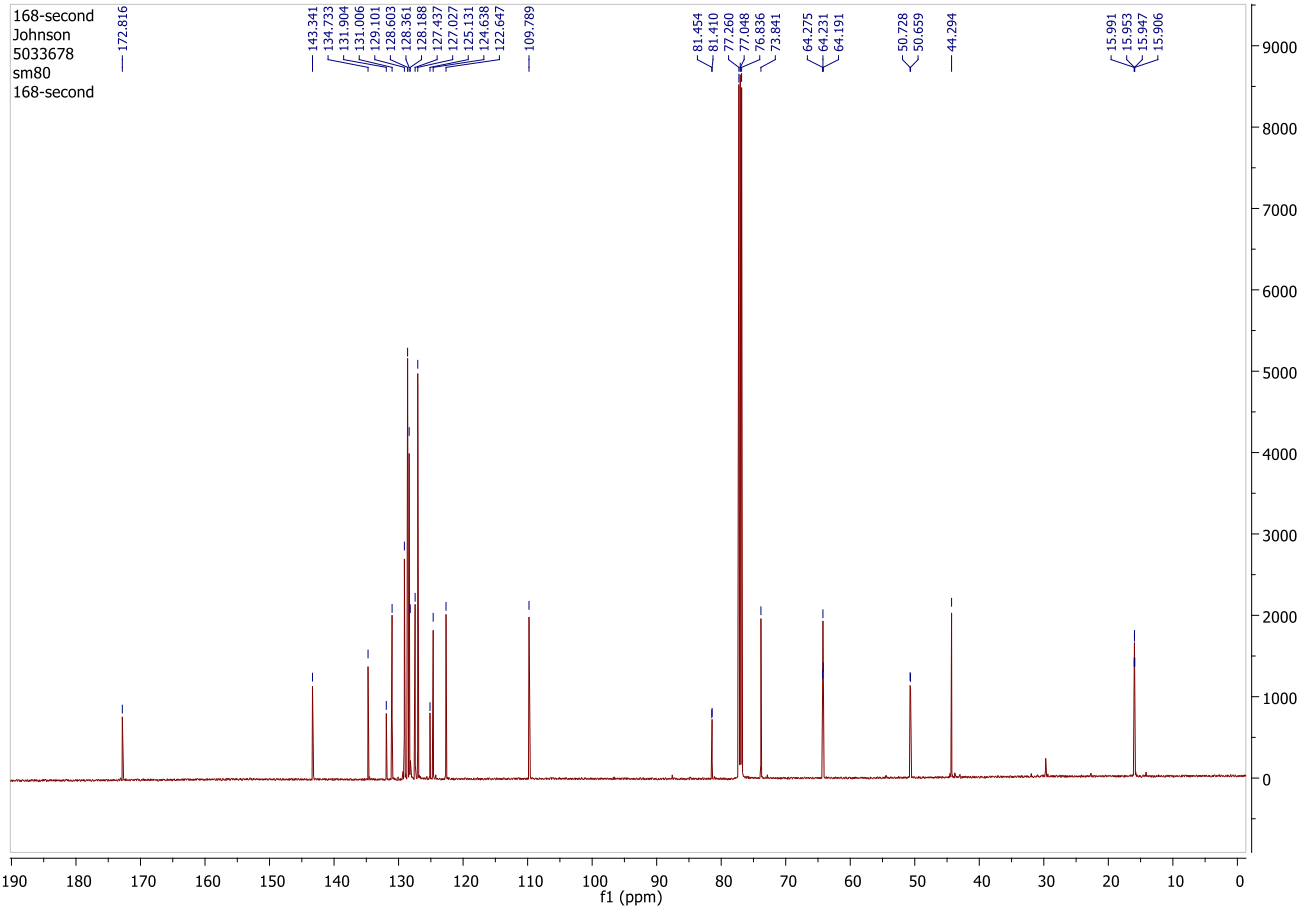


^{13}C NMR of 4ha

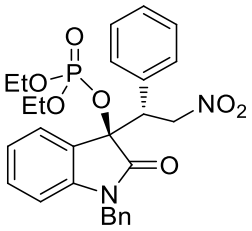


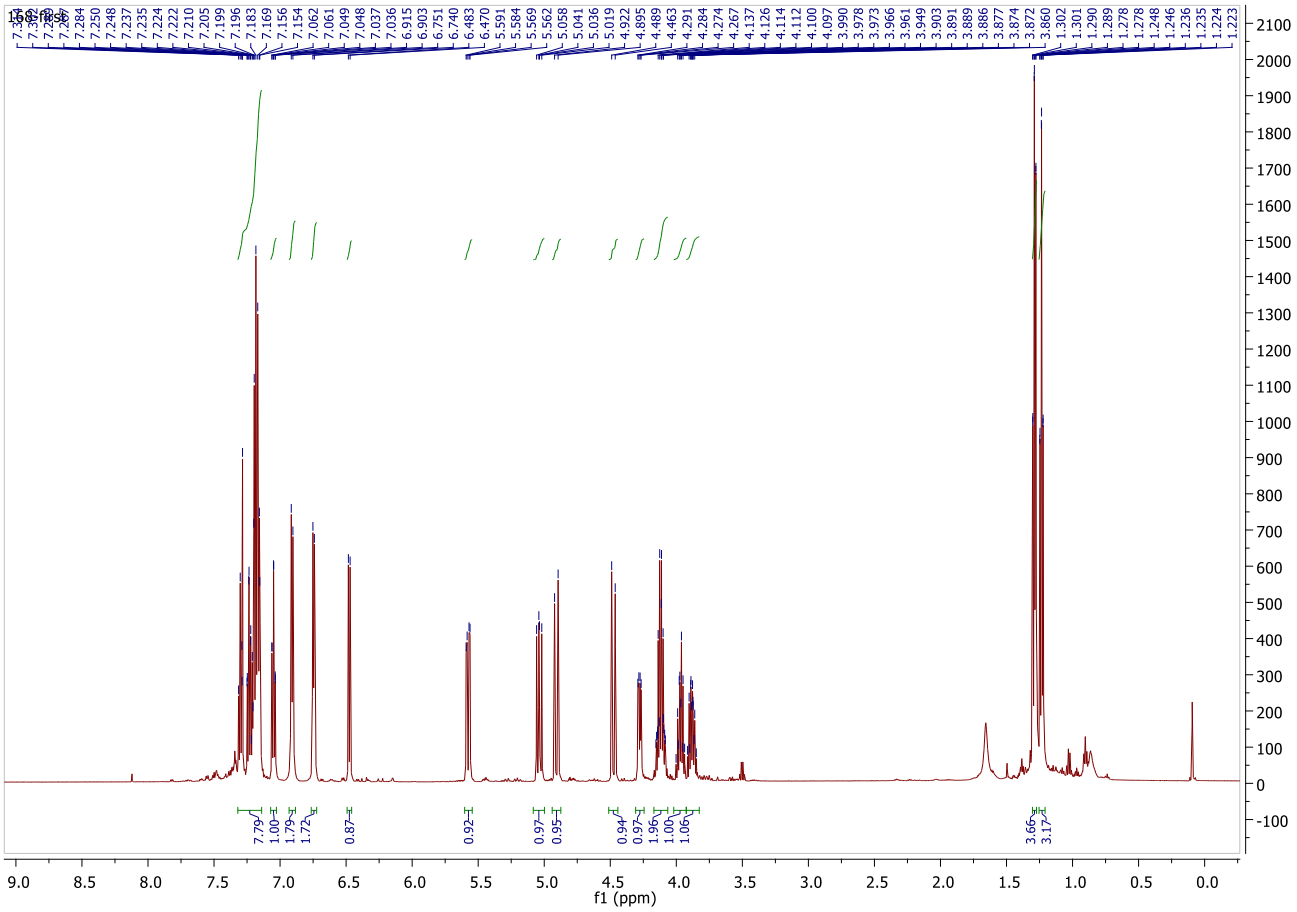


^{1}H NMR

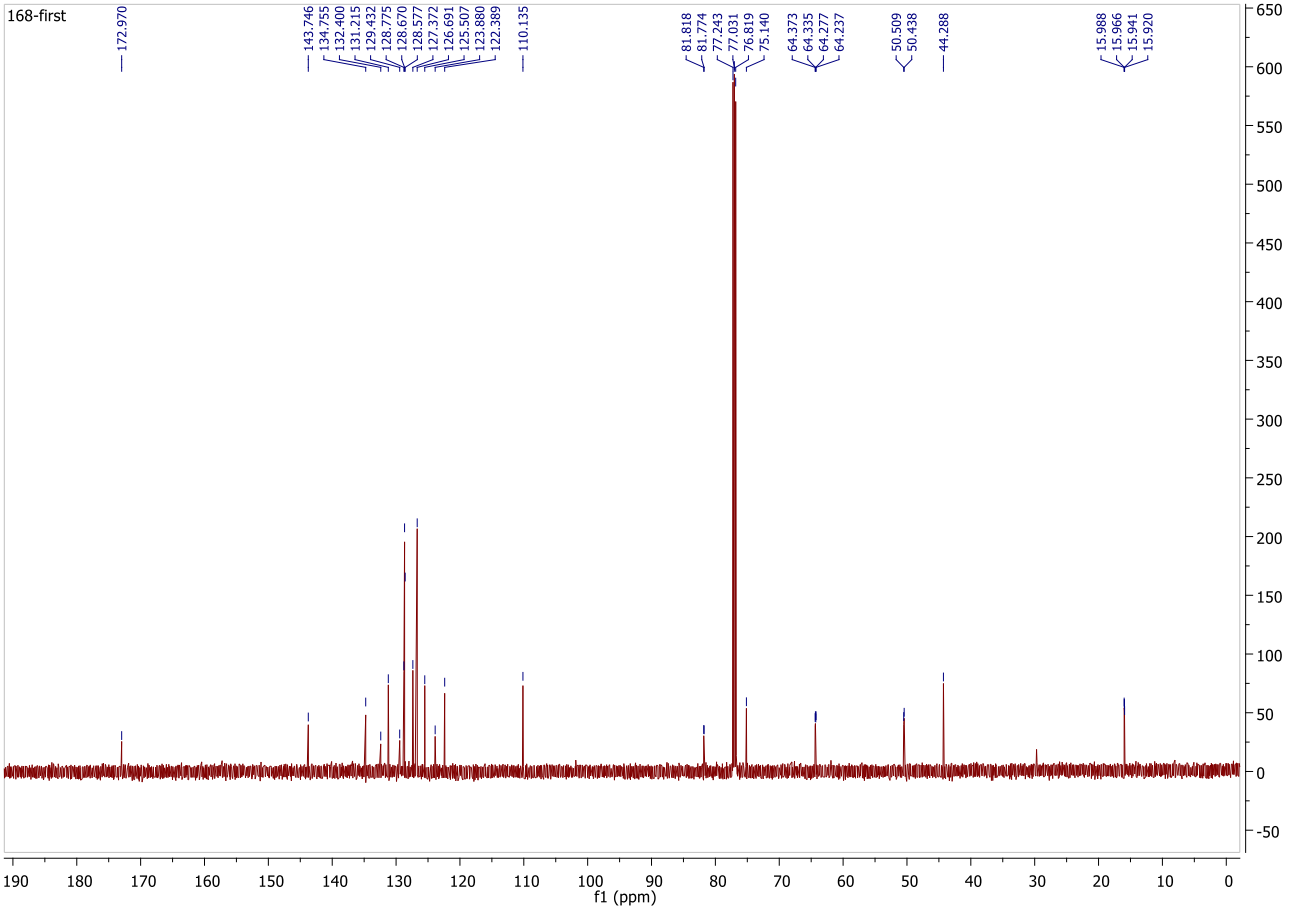

168-second
Johnson
5033678
sm80
168-second
172.816
143.341
134.733
131.904
131.006
129.101
128.603
128.361
128.188
127.437
127.027
125.131
124.638
122.647
109.789
81.454
81.410
77.260
77.048
76.836
73.841
64.275
64.231
64.191
50.728
50.659
44.294
15.991
15.953
15.947
15.906
f1 (ppm)

^{13}C NMR



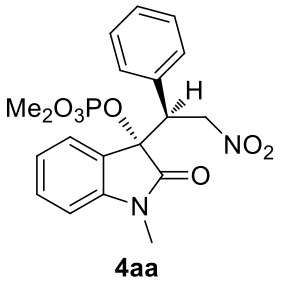


^{1}H NMR (2nd diastereomer)



^{13}C NMR (2nd diastereomer)

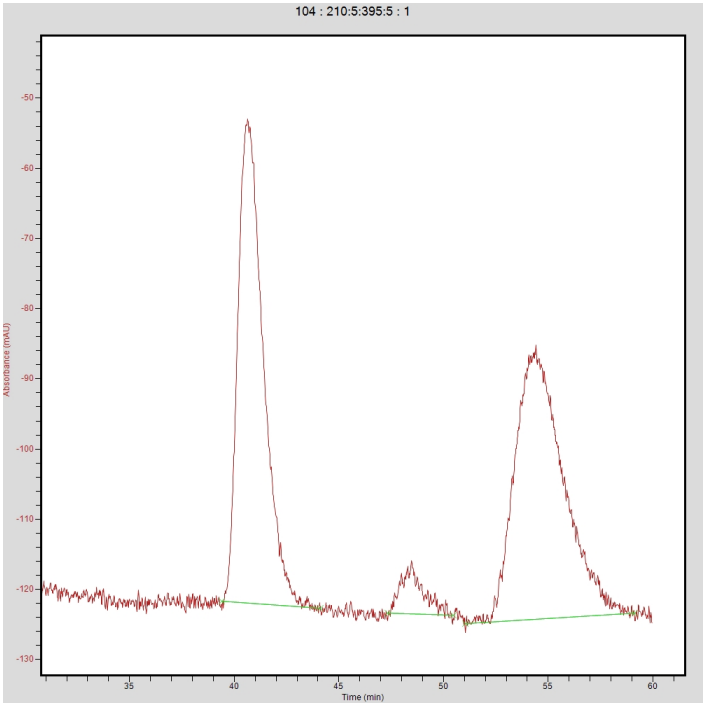
HPLC trace

(±) 

4aa

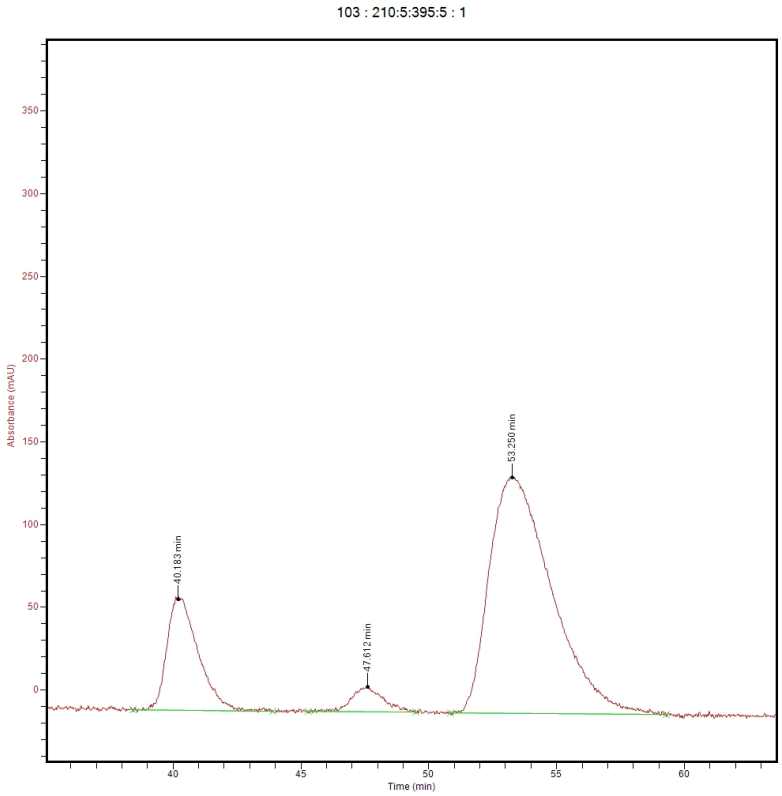
Chiralpak ID column, Hex/*i*PrOH = 85:5, flow rate = 1.0 mL/min, λ = 210 nm.

Racemic



Ret. Time (min)	Area	% Total Area
40.647	5789817.98	48.2
54.417	6210805.99	51.7

Asymmetric



Ret. Time (min)	Peak Area	% Total Area
40.183	5797356.31	19.2
53.250	24301729.12	80.7