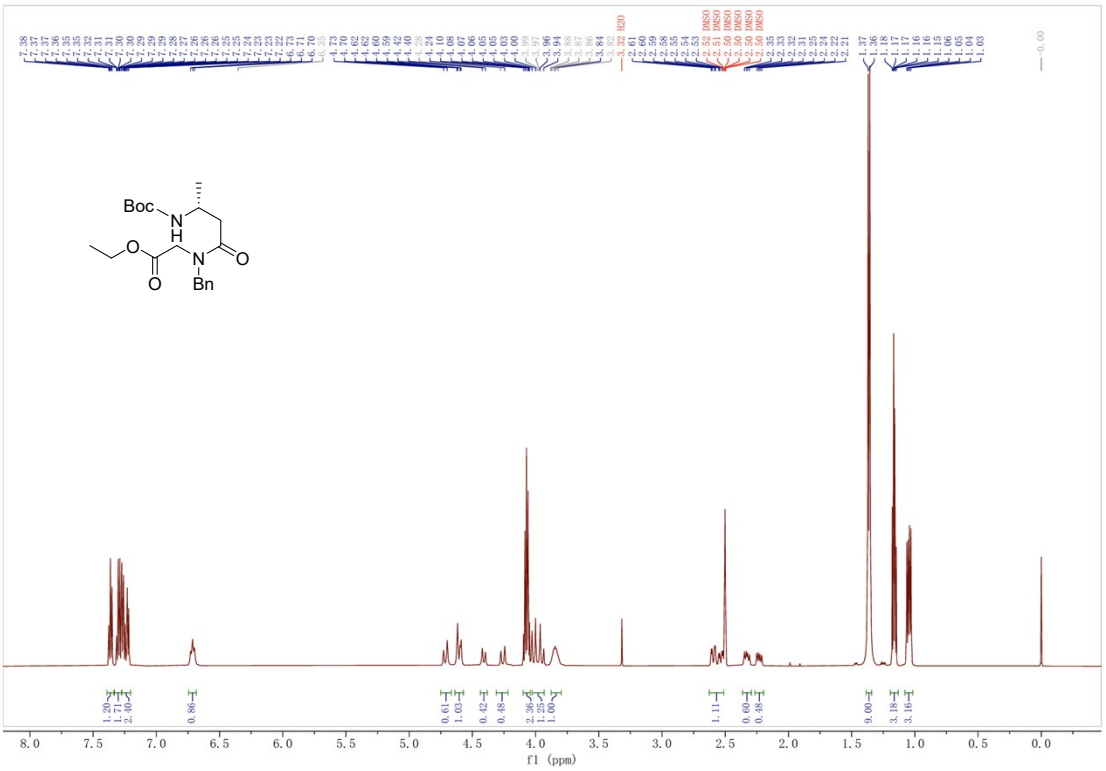
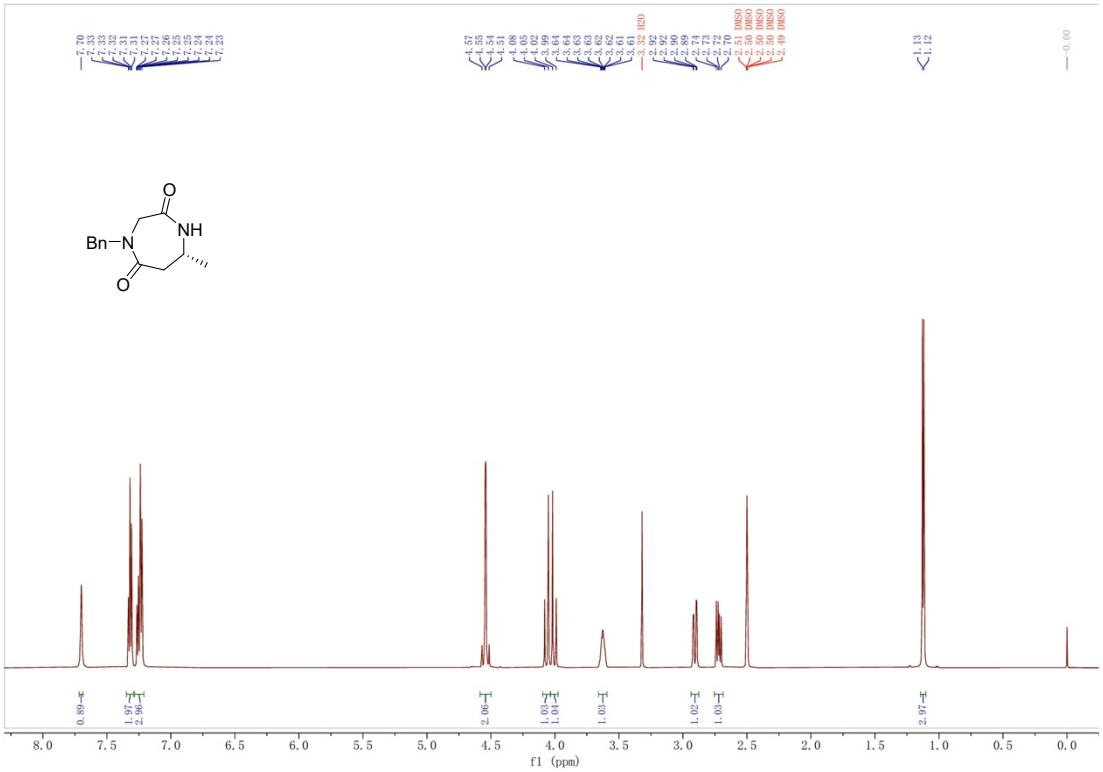
Supplementary Materials for:

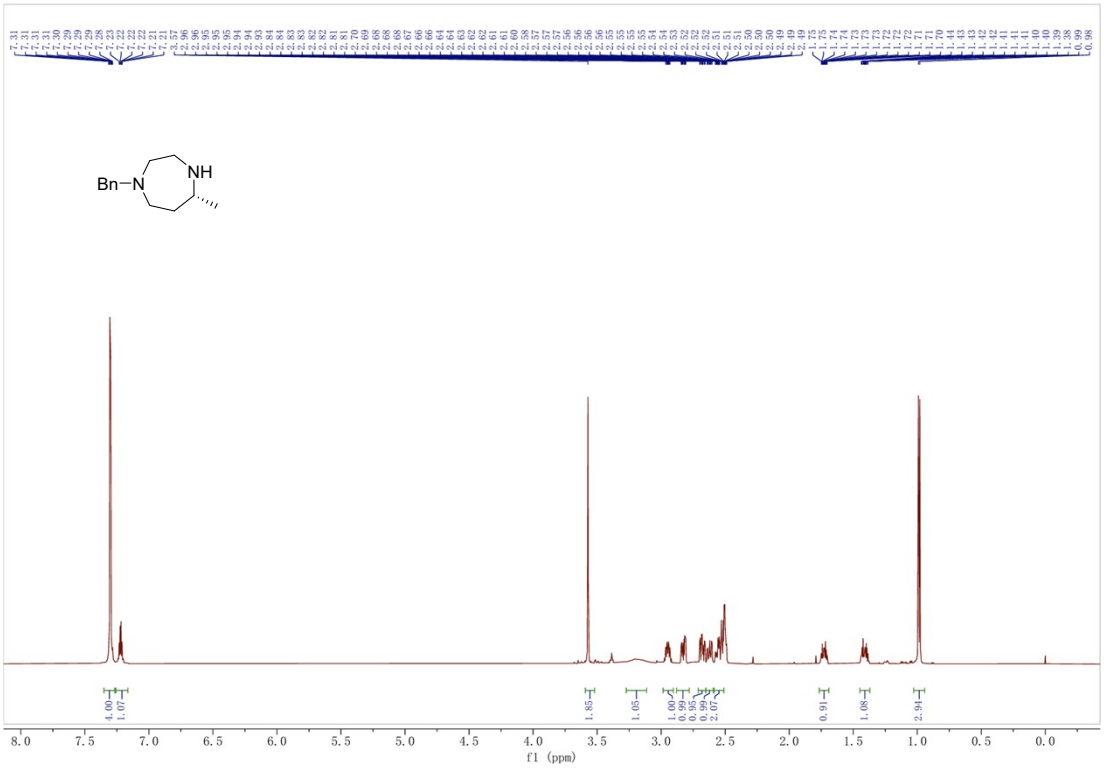
Optimized Synthesis of Suvorexant and Determination of Eight Residual Solvents by Headspace Gas Chromatography

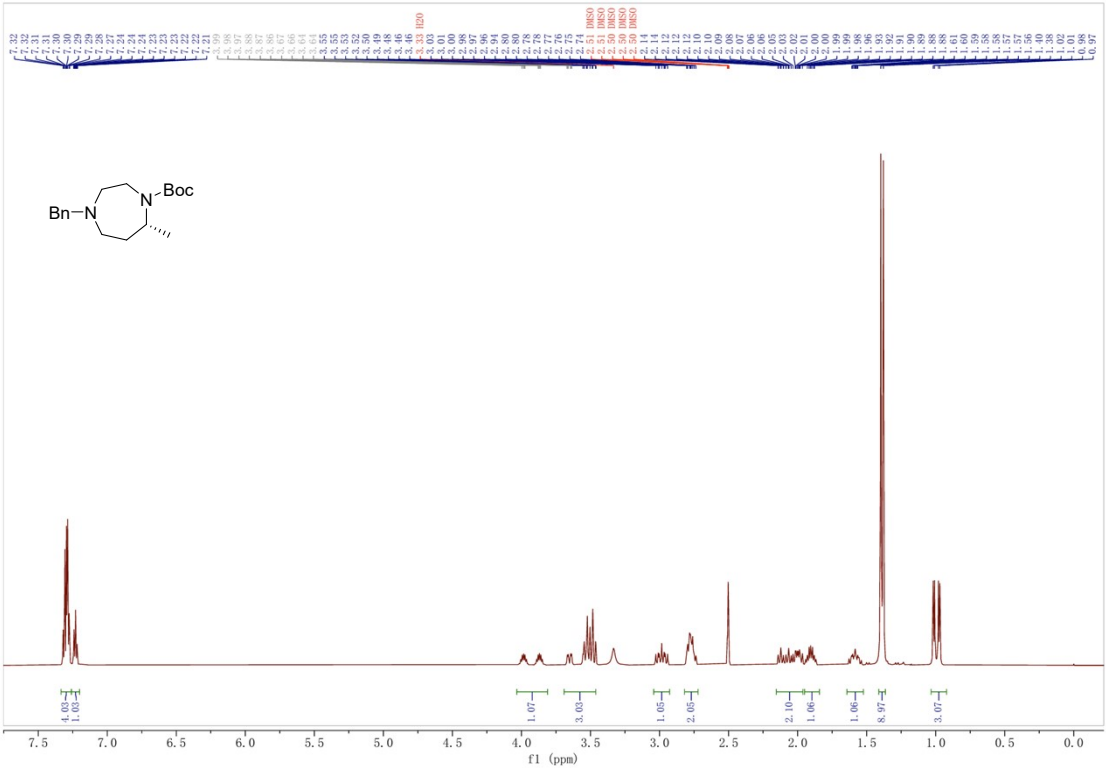
Supplementary materials for NMR spectra of the compounds:

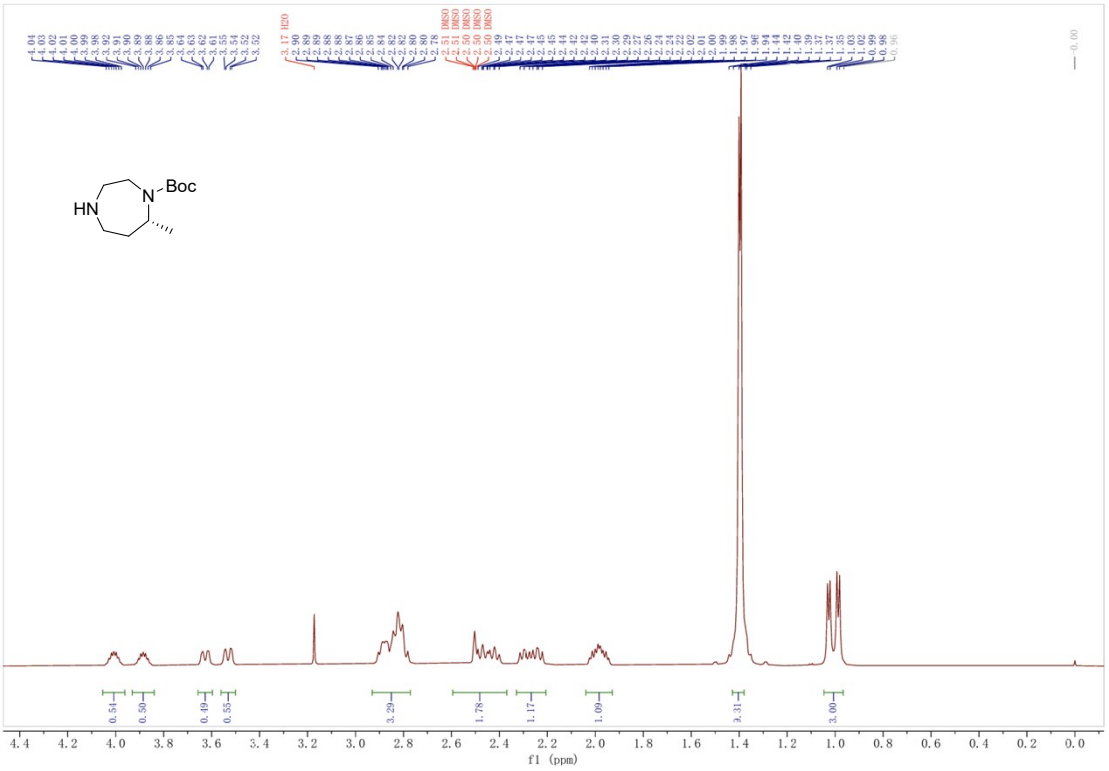
^{1}H NMR

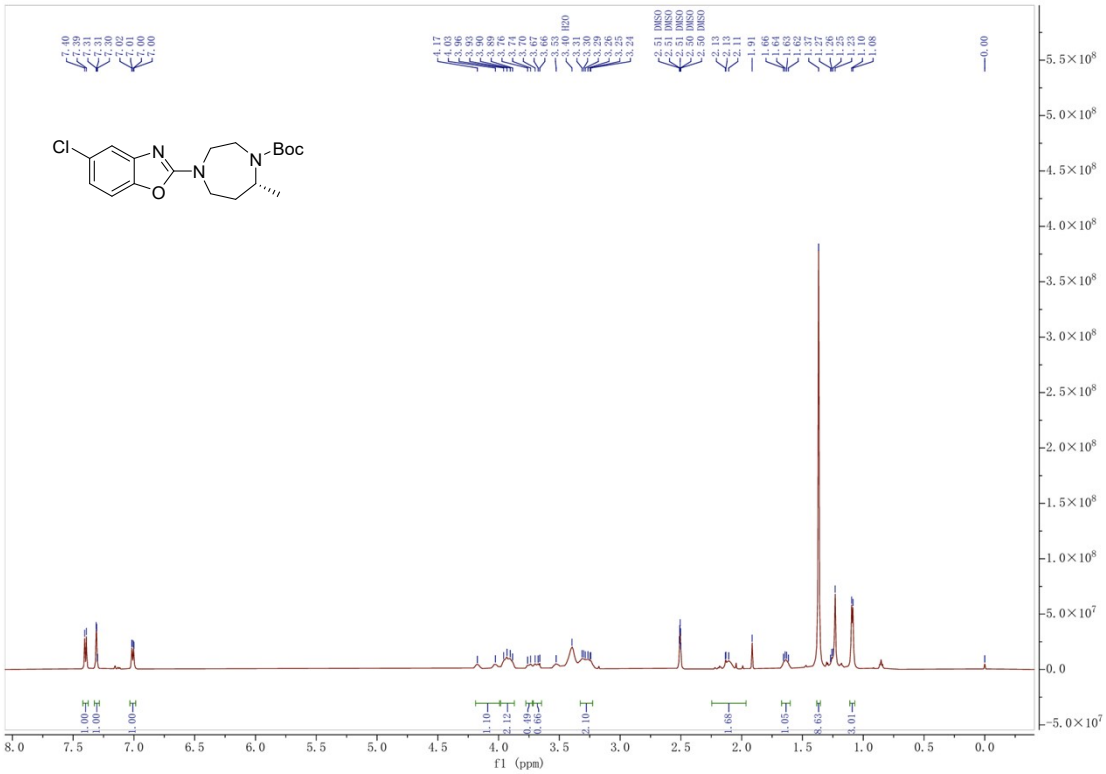


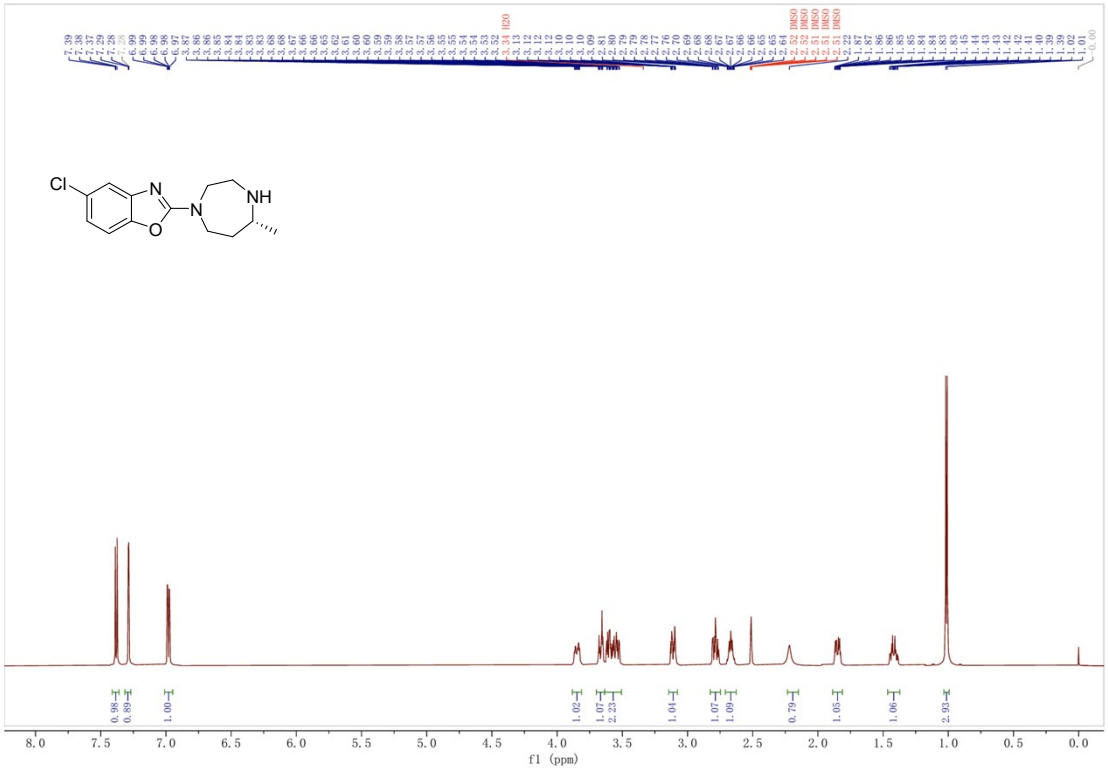


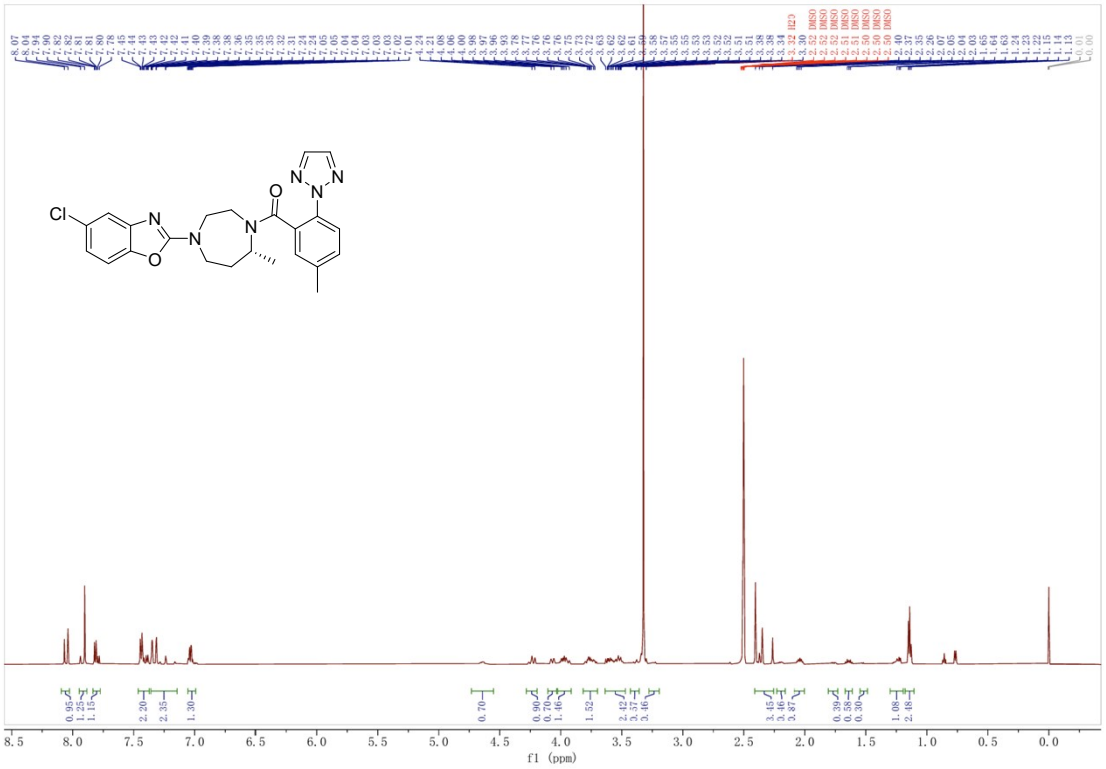

Bn–N
NH
f1 (ppm)


Bn–N
N
Boc
f1 (ppm)

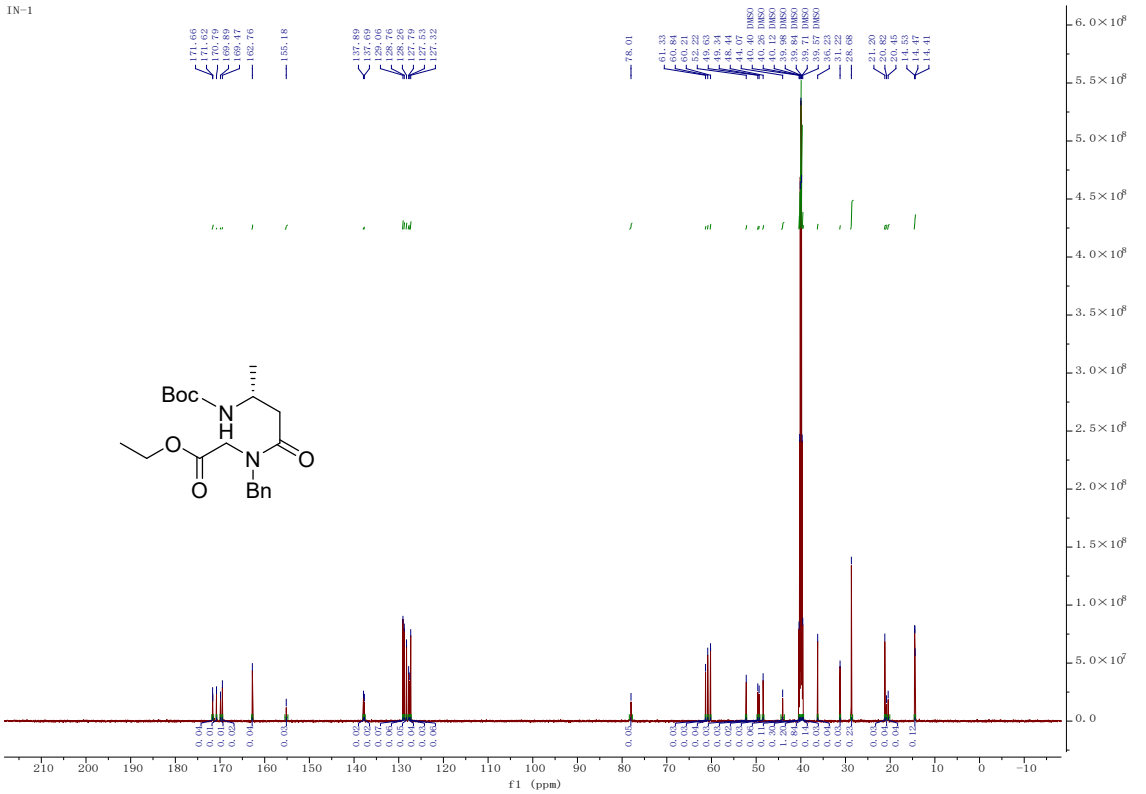

HN
N
Boc
f1 (ppm)

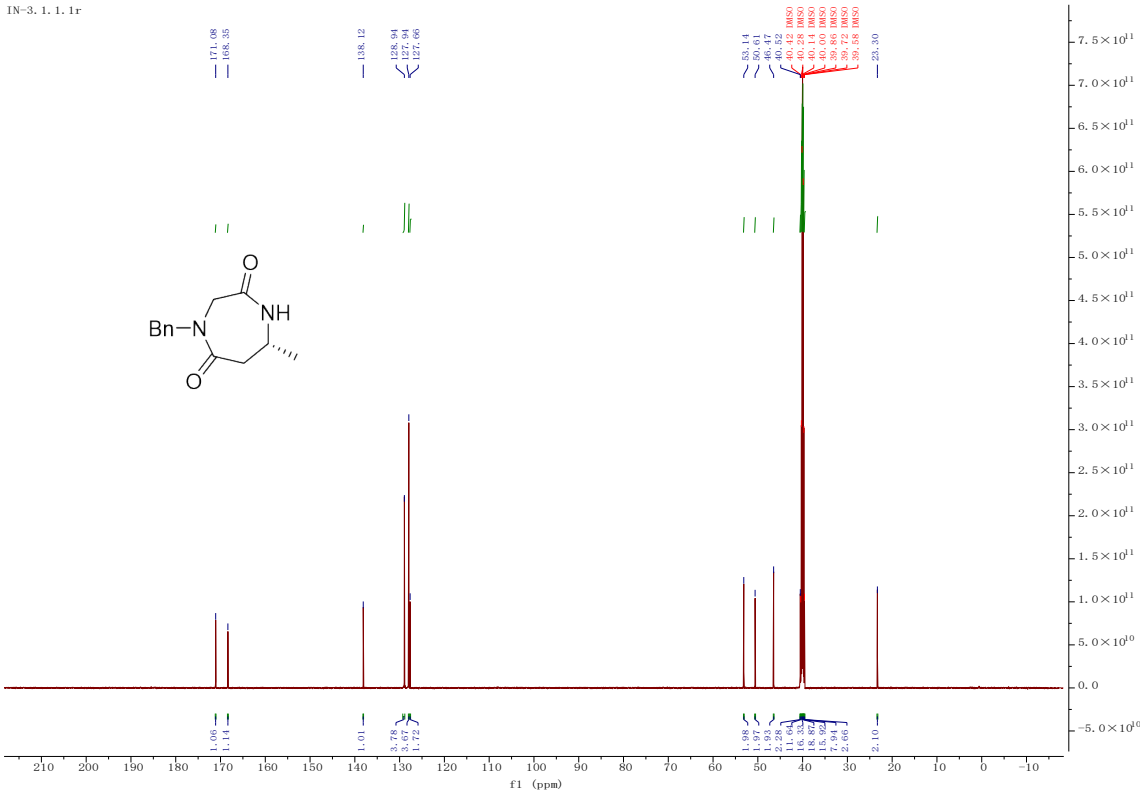

Cl
N
O
N
N
Boc
f1 (ppm)

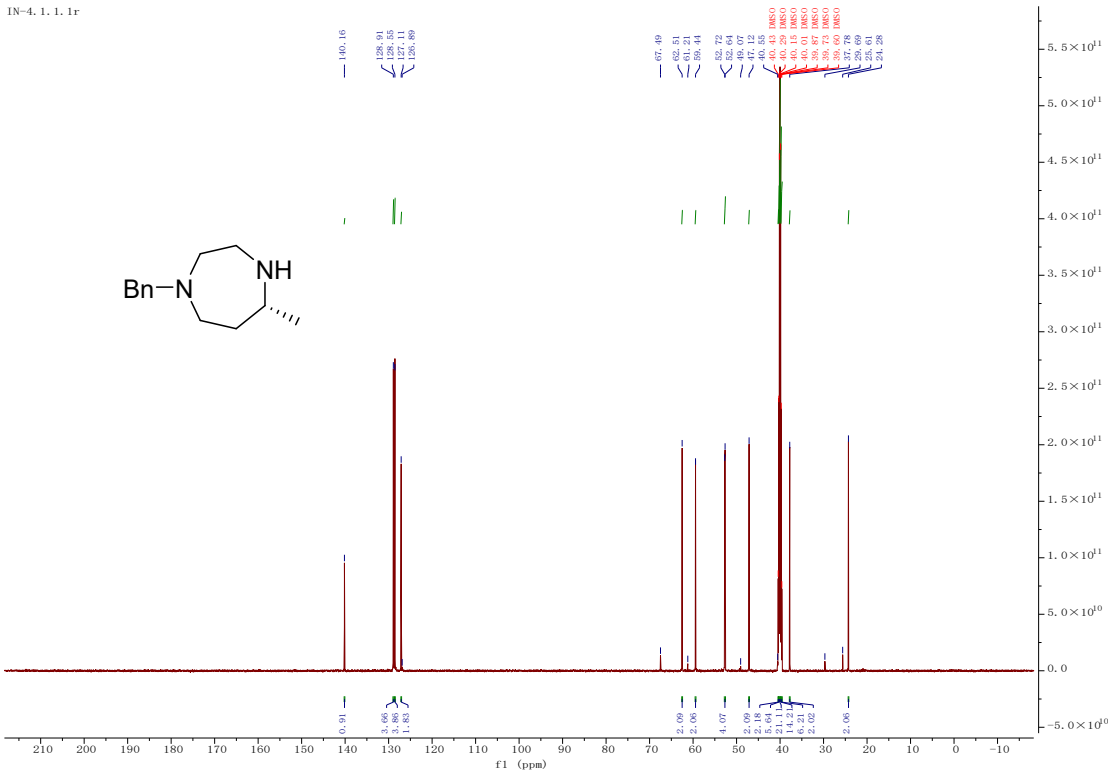

Cl
N
O
N
NH
f1 (ppm)

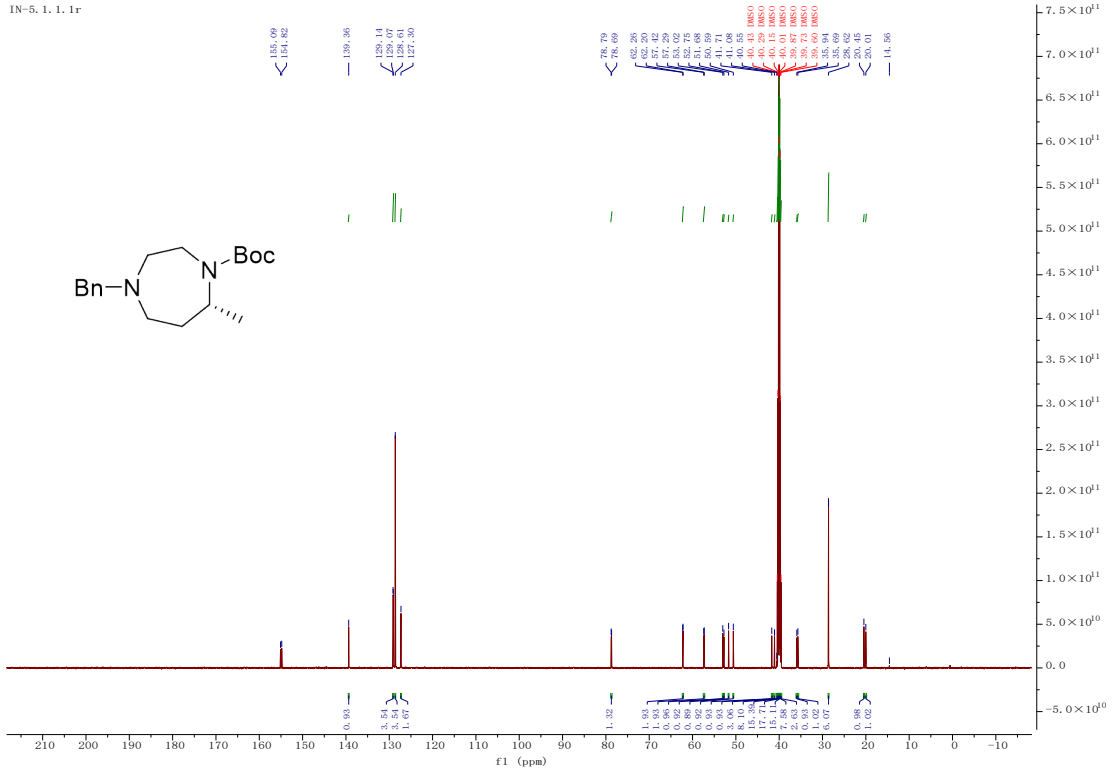

Cl
N
O
N
N
N
N
O
f1 (ppm)

^{13}C NMR

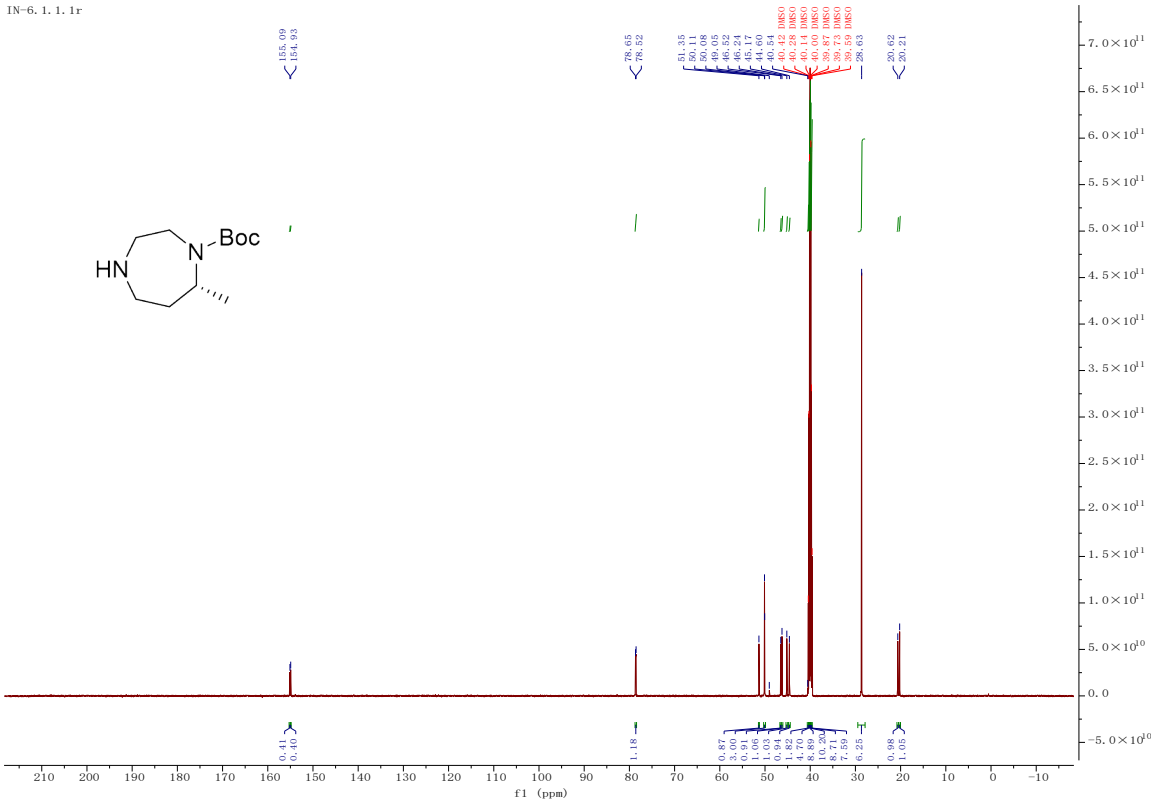




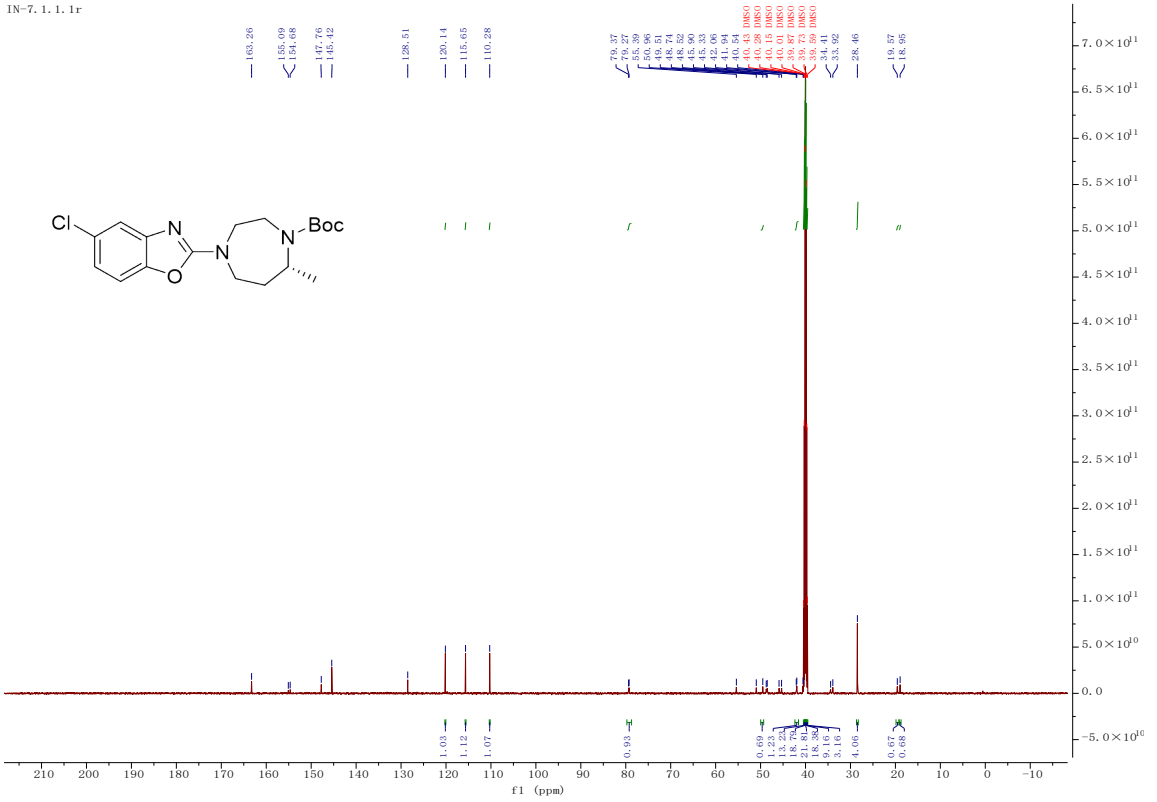

IN-4. 1. 1. 1r
Bn—N
NH
f1 (ppm)

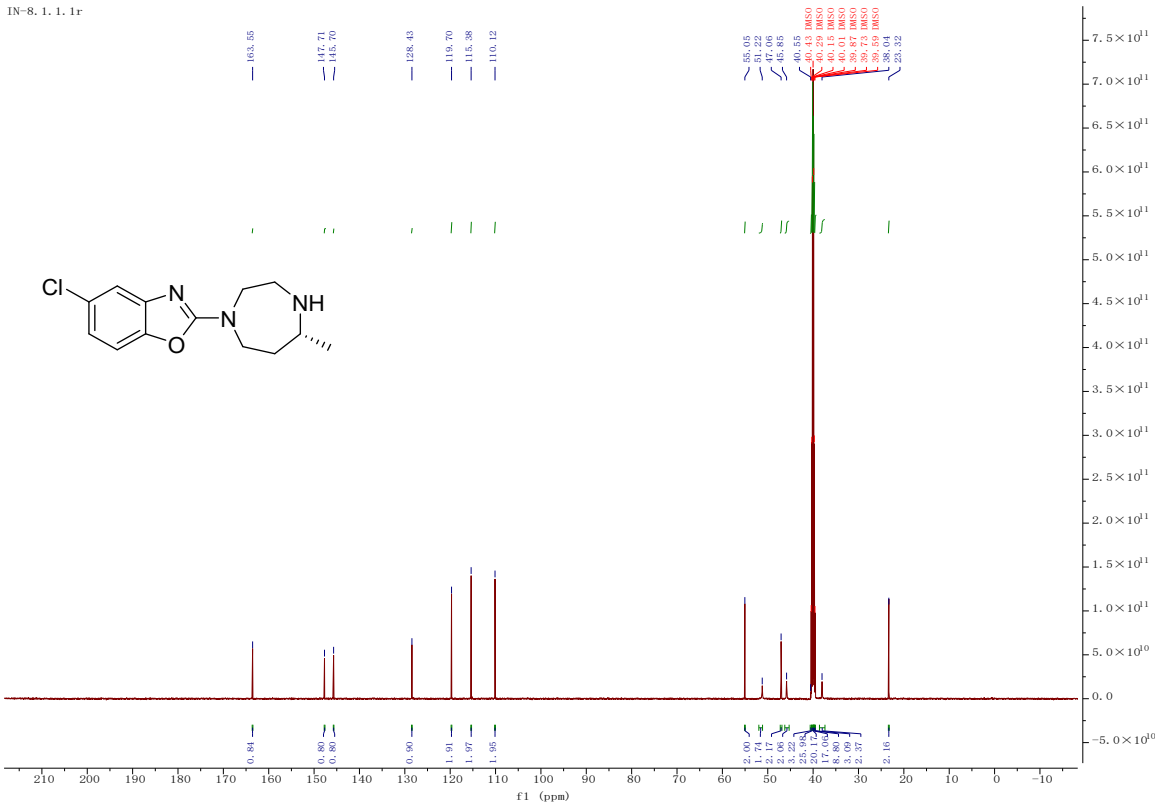

IN-5. 1. 1. 1r
Bn—N
N
Boc
f1 (ppm)

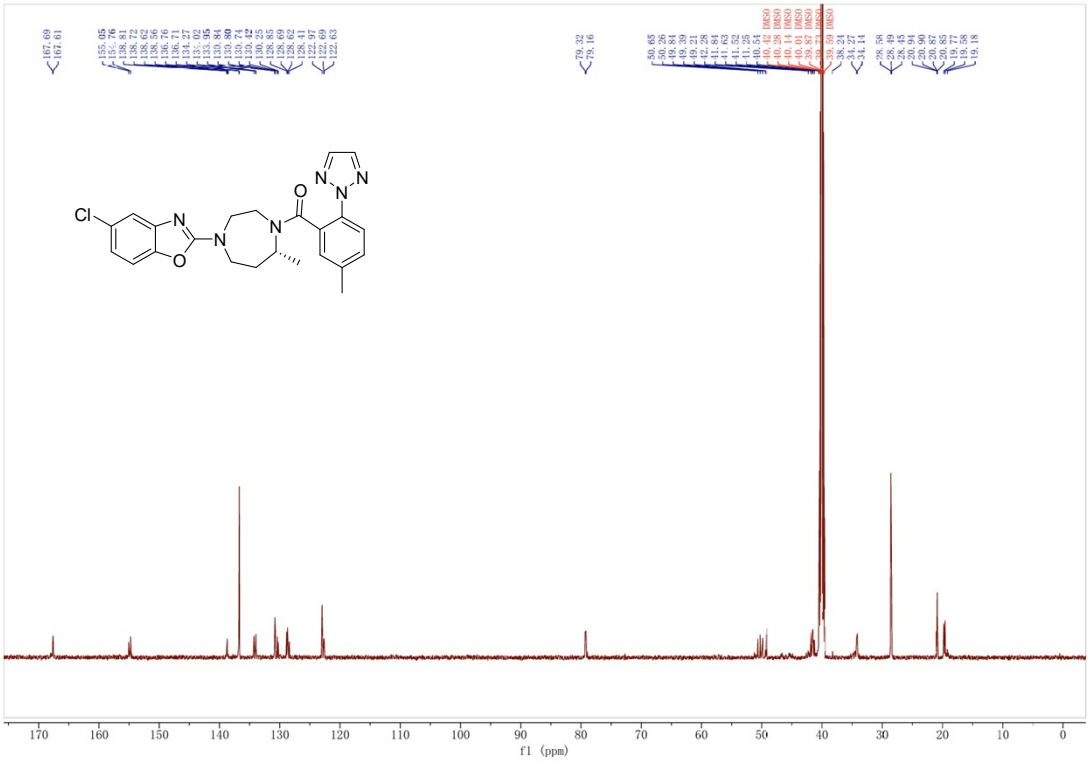
IN-6. 1. 1. 1r



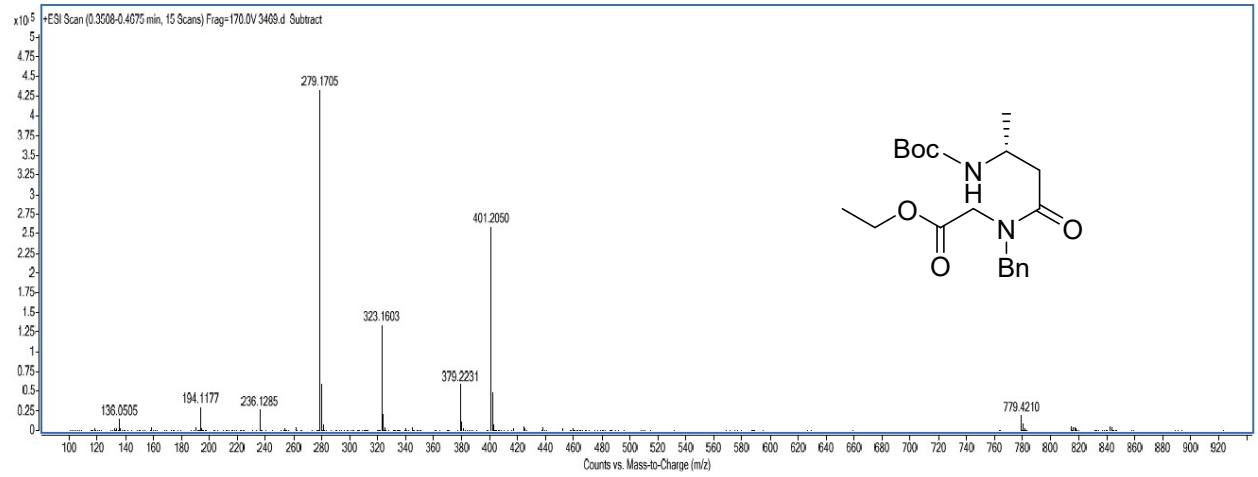
IN-7. 1. 1. 1r

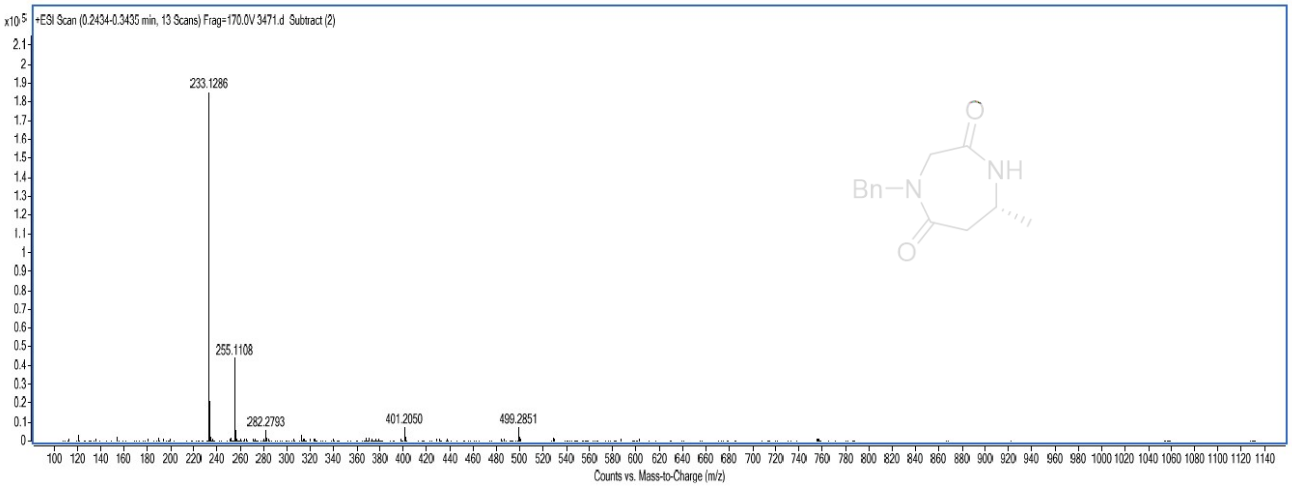


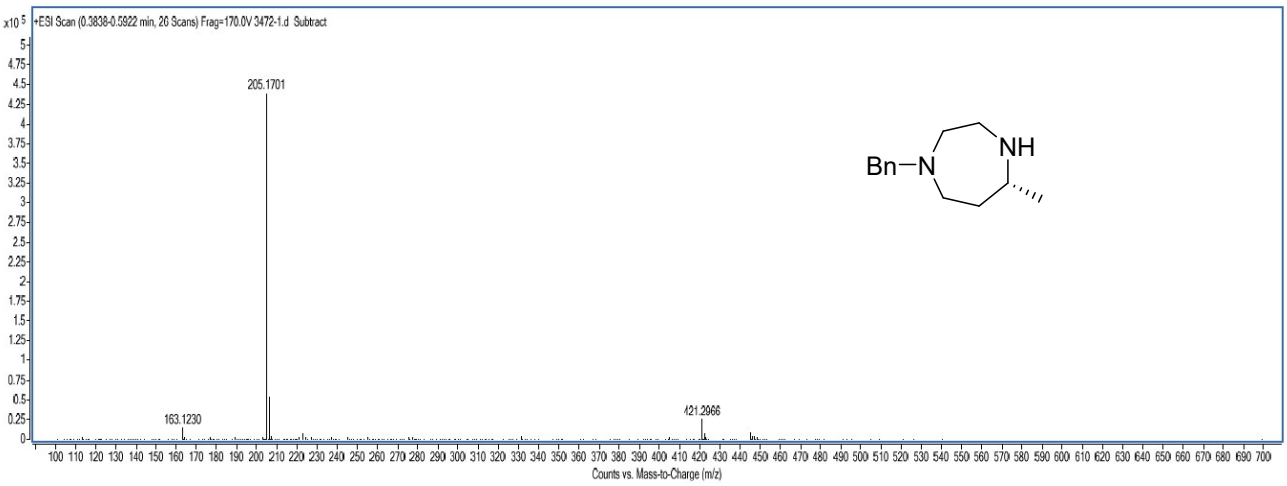

IN-8.1.1.1r
163.55
147.71
145.70
128.43
119.70
115.38
110.12
55.05
53.22
47.06
45.85
40.55
40.43 DMSO
40.29 DMSO
40.15 DMSO
40.01 DMSO
39.87 DMSO
39.73 DMSO
39.59 DMSO
38.04
23.32
Cl
N
O
N
NH
0.84
0.80
0.80
0.90
1.91
1.97
1.95
2.00
1.17
1.74
2.06
25.98
20.72
17.06
8.80
3.09
2.57
2.16
f1 (ppm)

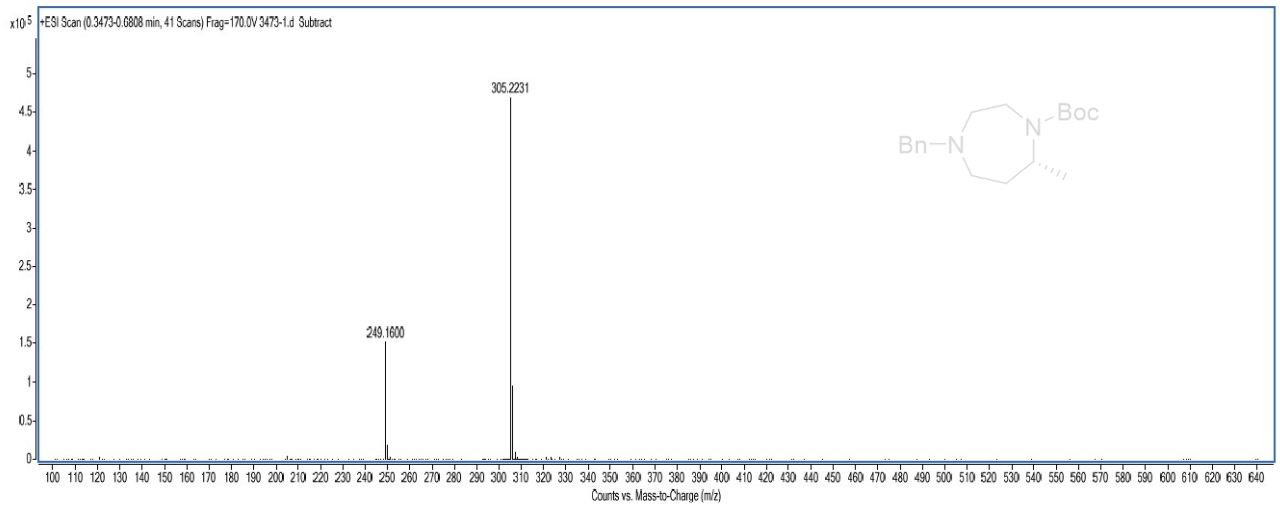

167.69
167.61
155.05
145.76
138.81
138.72
138.62
138.56
136.76
136.71
131.47
131.02
130.99
130.84
130.74
130.42
130.25
128.85
128.69
128.62
128.41
122.97
122.69
122.63
79.32
79.16
50.65
50.26
49.84
49.39
49.21
42.28
41.84
41.63
41.52
41.25
41.04
40.42 DMSO
40.28 DMSO
40.14 DMSO
40.00 DMSO
39.87 DMSO
39.73 DMSO
39.59 DMSO
34.27
34.14
28.58
28.49
28.45
28.04
20.90
20.87
20.85
19.77
19.58
19.18
Cl
N
O
N
N
N
O
f1 (ppm)

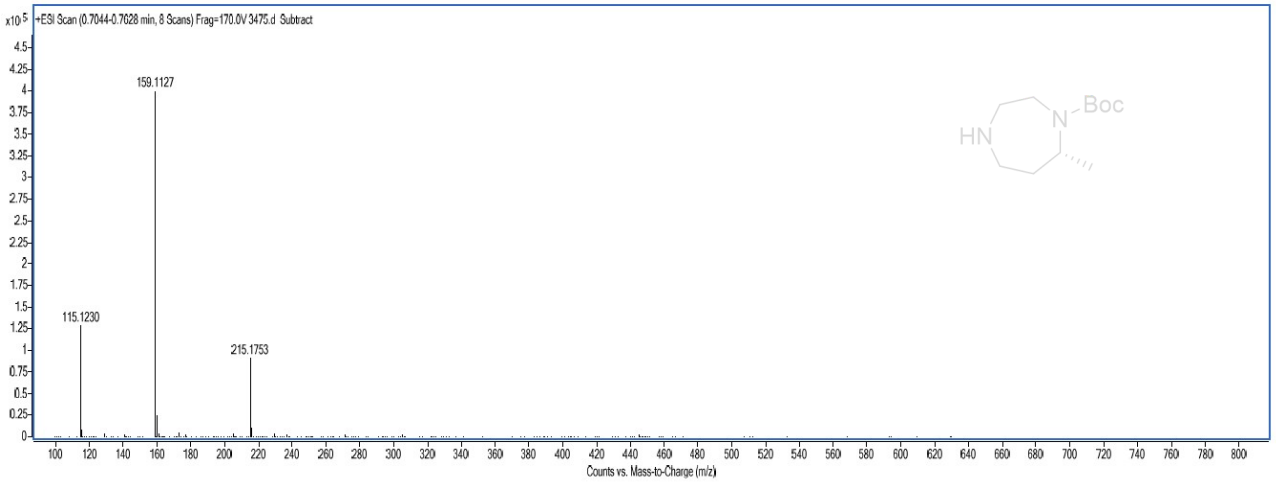
Supplementary materials for mass spectra of the compounds:

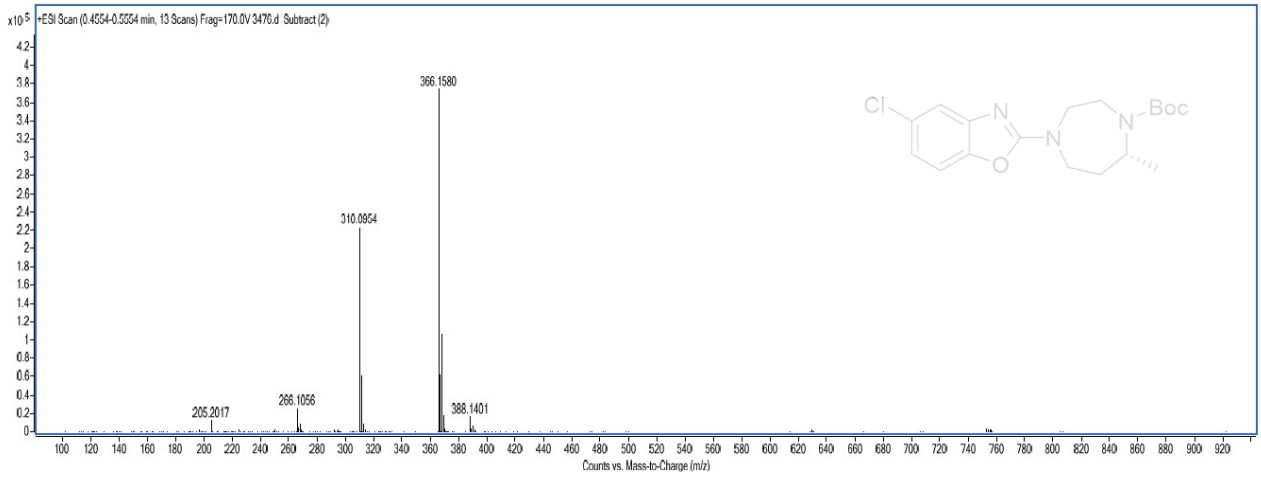


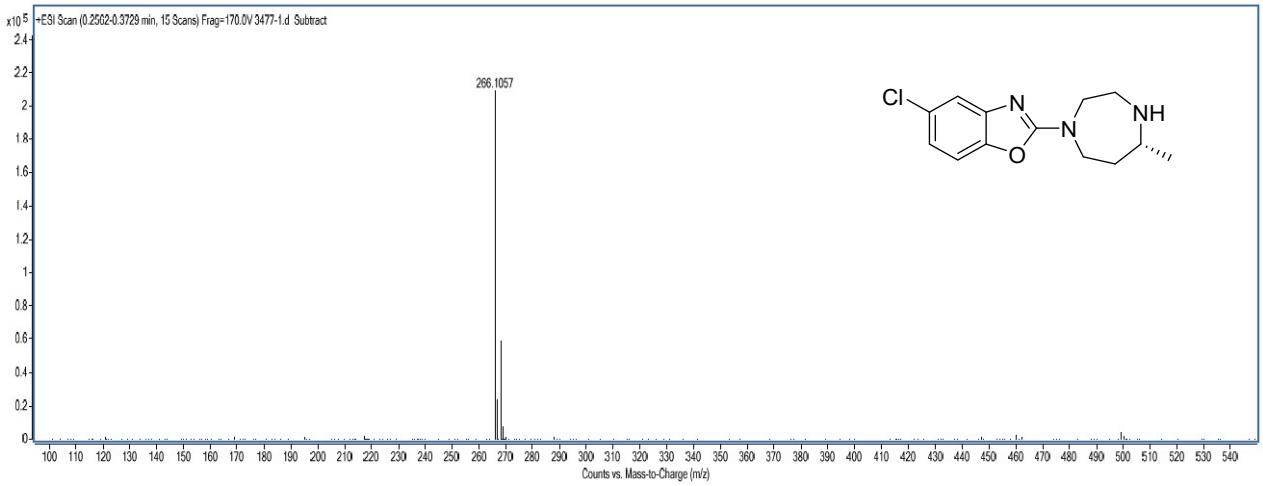


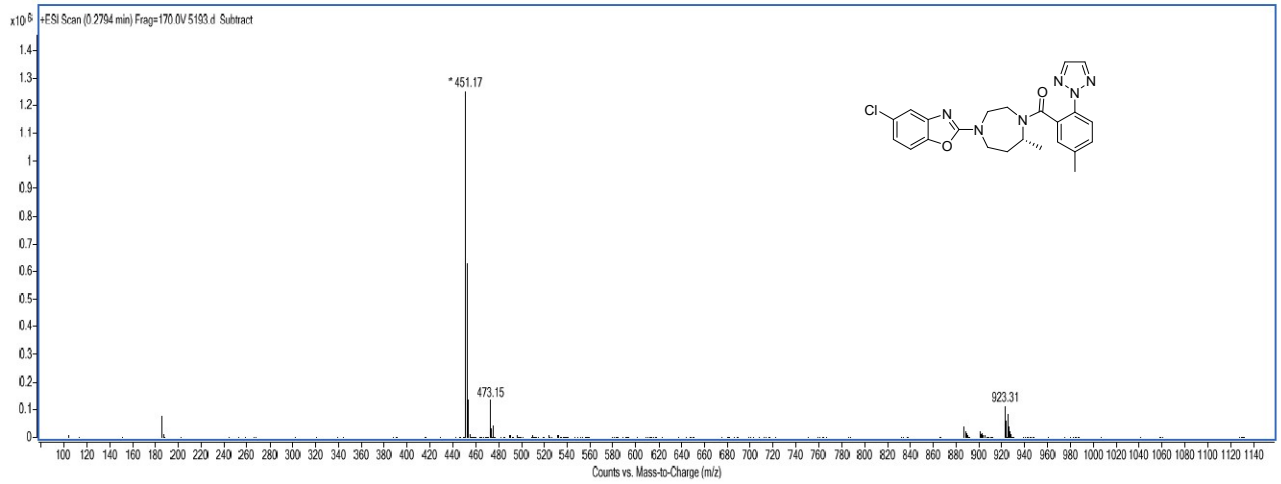



x10^5
+ESI Scan (0.3473-0.6808 min, 41 Scans) Frag=170.0V 3473-1.d Subtract
305.2231
249.1600
Boc
N
Bn
N
Counts vs. Mass-to-Charge (m/z)


x10^5
+ESI Scan (0.7044-0.7628 min, 8 Scans) Frag=170.0V 3475.d Subtract
159.1127
115.1230
215.1753
Boc
N
HN
Counts vs. Mass-to-Charge (m/z)


x10^5
+ESI Scan (0.4554-0.5554 min, 13 Scans) Frag=170.0V 3476.d Subtract (2)
366.1580
310.0954
205.2017
266.1056
388.1401
Cl
N
O
N
N
Boc
Counts vs. Mass-to-Charge (m/z)


x10 5
+ESI Scan (0.2502-0.3729 min, 15 Scans) Frag=170.0V 3477-1.d Subtract
266.1057
Cl
N
O
N
NH
Counts vs. Mass-to-Charge (m/z)


x10 6
+ESI Scan (0.2794 min) Frag=170.0V 5193.d Subtract
* 451.17
473.15
923.31
Cl
N
O
N
N
N
N
O
Counts vs. Mass-to-Charge (m/z)