Supporting Information

Studies toward the synthesis of chartelline C

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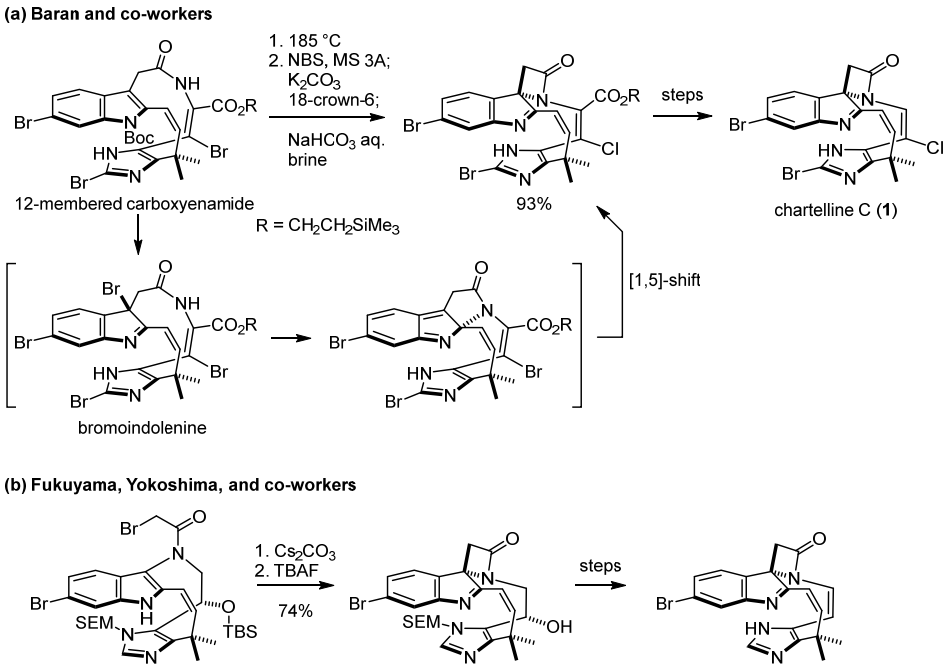


Scheme S1. Previously reported key transformations for the construction of core indolenine *β*-lactam.

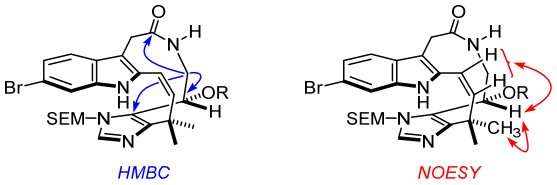
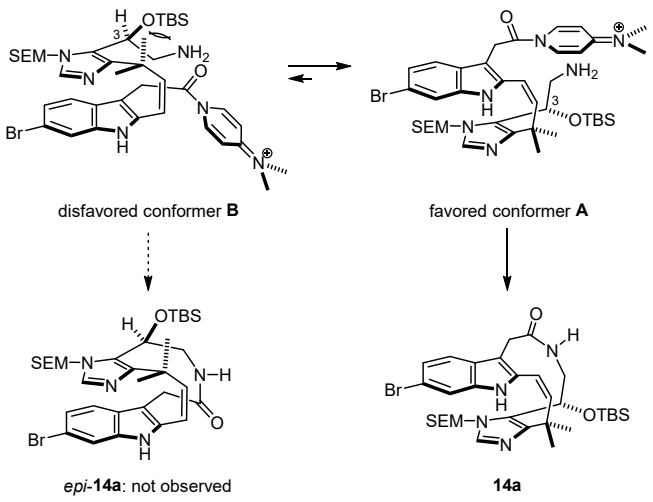


Figure S1. Diagnostic HMBC and NOESY correlations for **14a** (R = TBS).



Scheme S2. A plausible mechanism for high diastereocontrol of macrolactamization of **13**. The high diastereoselectivity with which **14a** was formed is presumably due to the sterically demanding nature of the siloxy group at C3, which causes conformer **A** to be favored and conformer **B** disfavored.

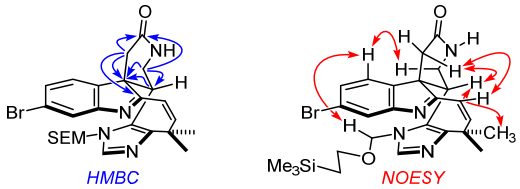
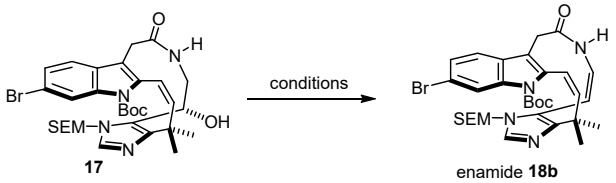
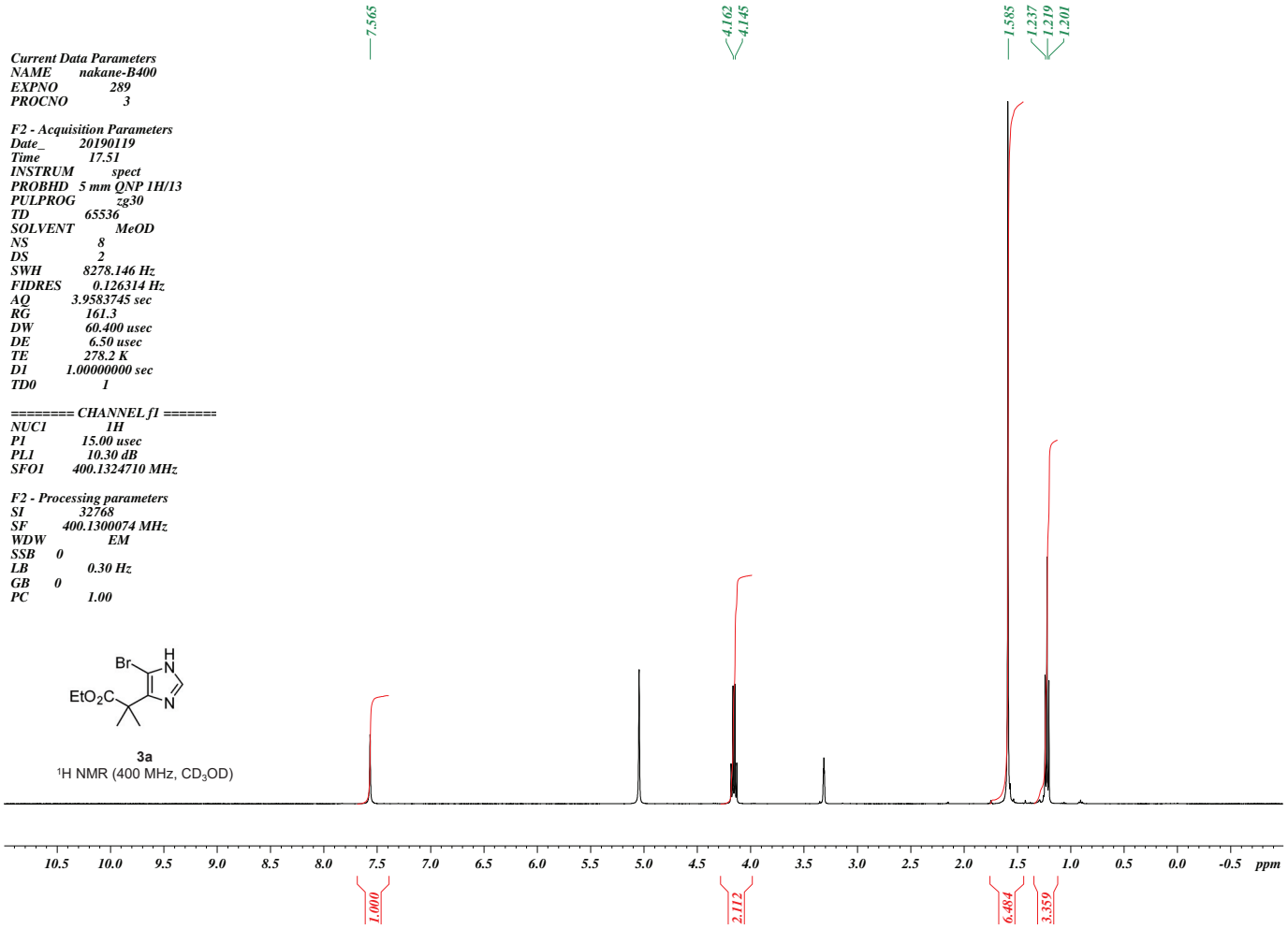


Figure S2. Diagnostic HMBC and NOESY correlations for **15**.

Table S1. Attempts of dehydration of **17**.



entry	conditions	results
1	TFAA, pyridine, $(CH_2Cl)_2$, 70 °C	**18b**: 21%
2	$(CF_3CF_2CO)_2O$, *i*-Pr_2NEt, $(CH_2Cl)_2$, reflux	**18b**: 51%
3	Ts_2O, pyridine, $(CH_2Cl)_2$, 70 °C	no reaction
4	Tf_2O, Et_3N, $(CH_2Cl)_2$, 70 °C	complex mixture
5	Ms_2O, Et_3N, $(CH_2Cl)_2$, 70 °C	**18b**: 0%
6	trichloroacetic anhydride, pyridine, $(CH_2Cl)_2$, 70 °C	complex mixture
7	DIAD, *n*-Bu_3P, THF, reflux	no reaction
8	$BF_3{\cdot}OEt_2$, THF, reflux	complex mixture
9	TFA, $(CH_2Cl)_2$, 70 °C	no reaction
10	Burgess reagent, toluene, 80 °C	**18b**: 0%
11	Martin sulfurane, $(CH_2Cl)_2$, 70 °C	no reaction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4.162
4.145
1.585
1.237
1.219
1.201
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PROCNO 3
F2 - Acquisition Parameters
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Time 17.51
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT MeOD
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 161.3
DW 60.400 usec
DE 6.50 usec
TE 278.2 K
D1 1.00000000 sec
TD0 1
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NUC1 1H
P1 15.00 usec
PL1 10.30 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300074 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
H
N
N
EtO2C
3a
1H NMR (400 MHz, CD3OD)
10.5 10.0 9.5 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 -0.5 ppm
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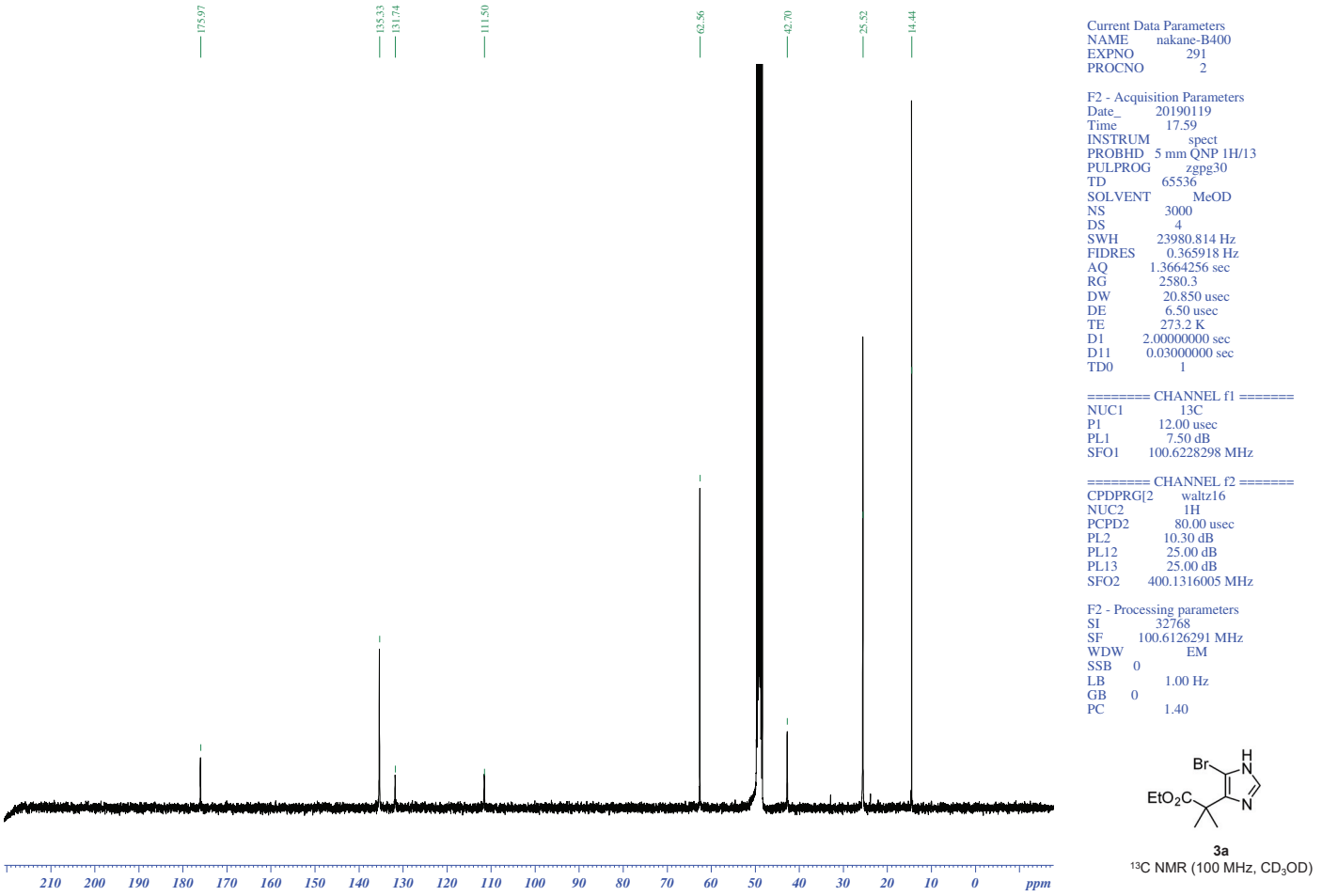
2.112
6.484
3.359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135.33
131.74
111.50
62.56
42.70
25.52
14.44
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PROCNO 2
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Time 17.59
INSTRUM spect
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PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 3000
DS 4
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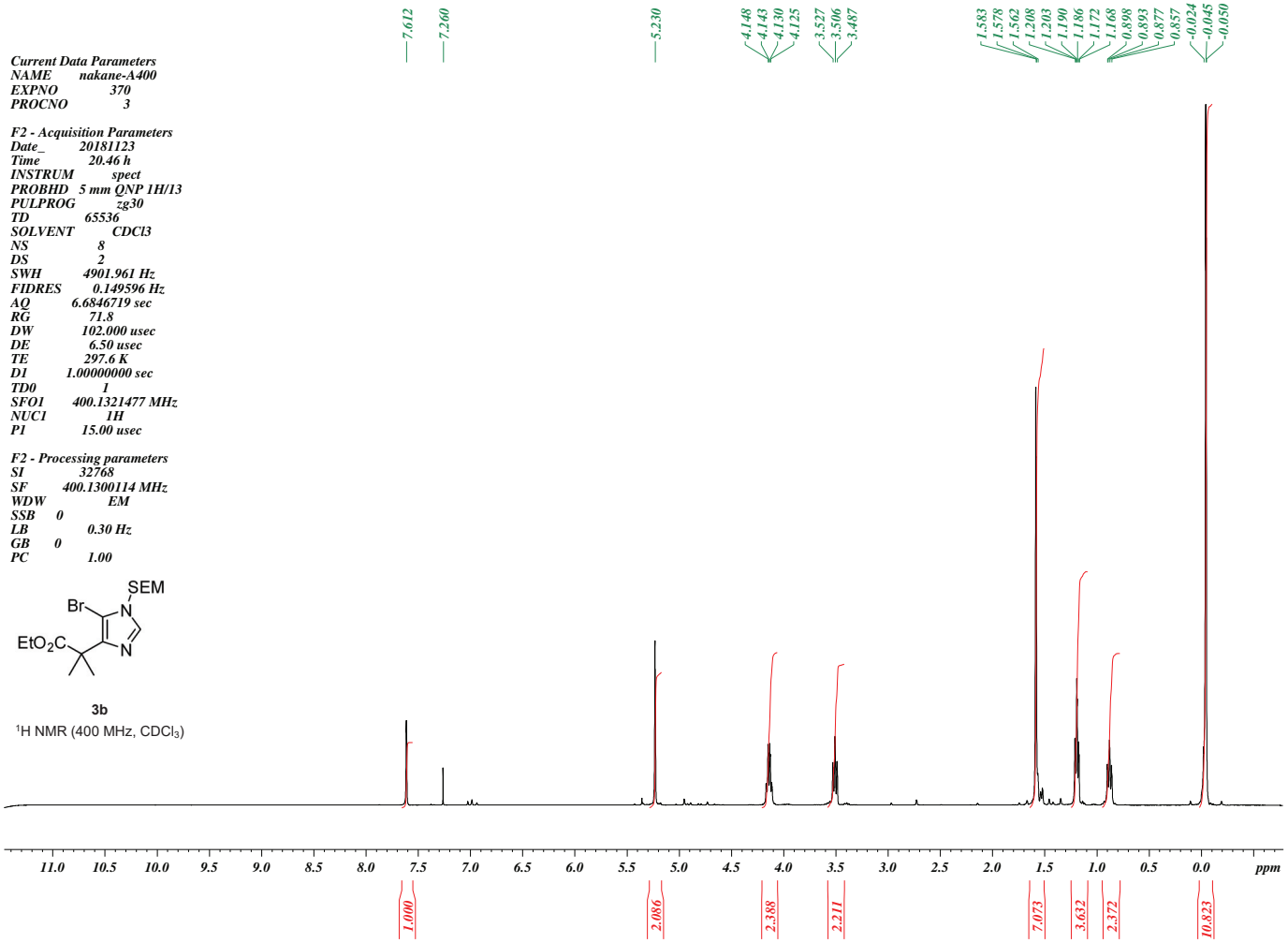
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 273.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 12.00 usec
PL1 7.50 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 =======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 10.30 dB
PL12 25.00 dB
PL13 25.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6126291 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Br
H
N
N
EtO2C
3a
13C NMR (100 MHz, CD3OD)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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PULPROG zg30
TD 65536
SOLVENT CDCl3
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DS 2
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FIDRES 0.149596 Hz
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RG 71.8
DW 102.000 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321477 MHz
NUC1 1H
P1 15.00 usec
F2 - Processing parameters
SI 32768
SF 400.1300114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
SEM
Br
N
N
EtO2C
3b
1H NMR (400 MHz, CDCl3)
7.612
7.260
5.230
4.148
4.143
4.130
4.125
3.527
3.506
3.487
1.583
1.578
1.562
1.208
1.203
1.190
1.186
1.172
1.168
0.898
0.893
0.877
0.857
-0.024
-0.045
-0.050
11.0 10.5 10.0 9.5 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 ppm
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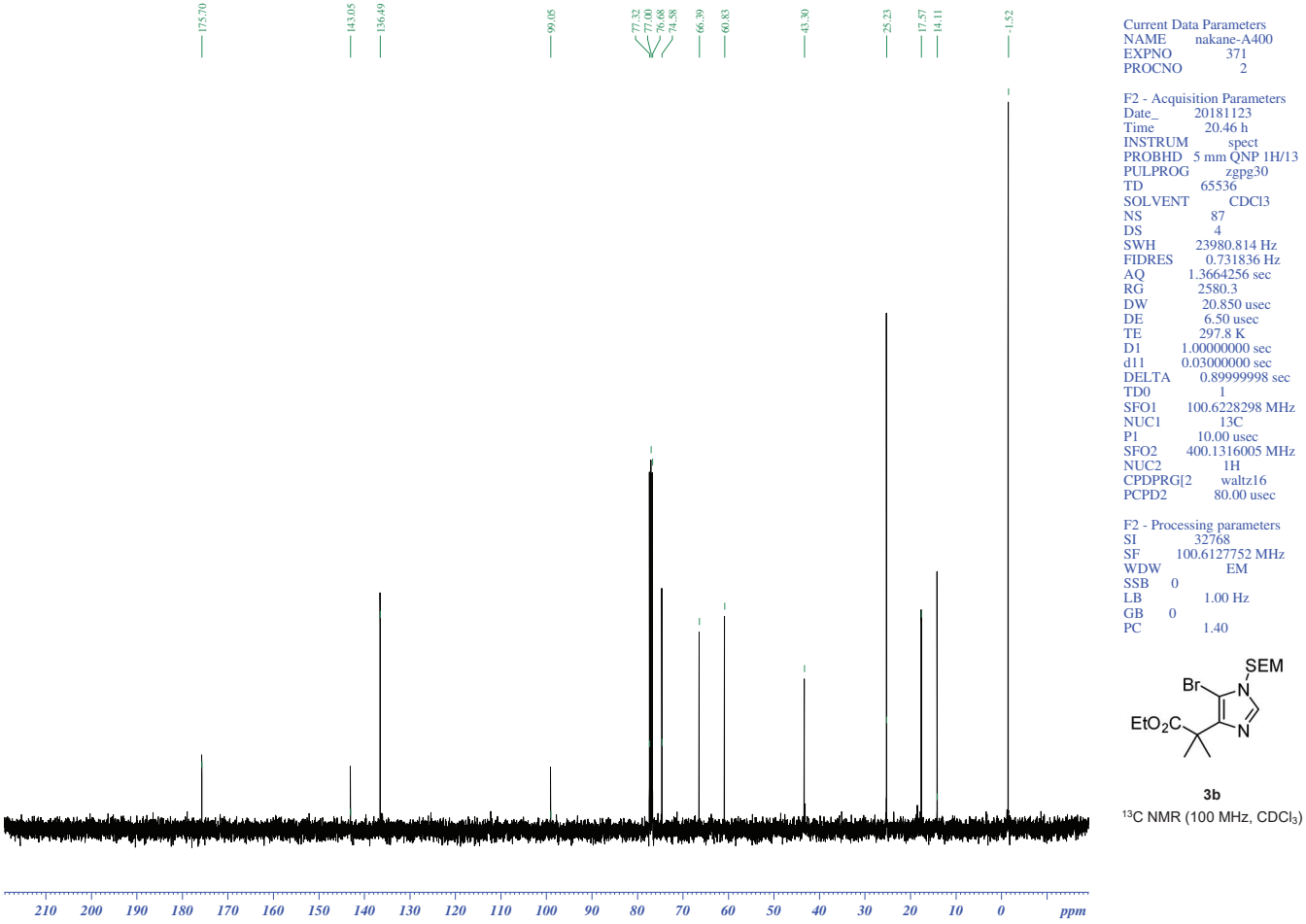
2.388
2.211
7.073
3.632
2.372
10.823


175.70
143.05
136.49
99.05
77.32
77.00
76.68
74.58
66.39
60.83
43.30
25.23
17.57
14.11
-1.52
Current Data Parameters
NAME nakane-A400
EXPNO 371
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181123
Time 20.46 h
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 87
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SWH 23980.814 Hz
FIDRES 0.731836 Hz
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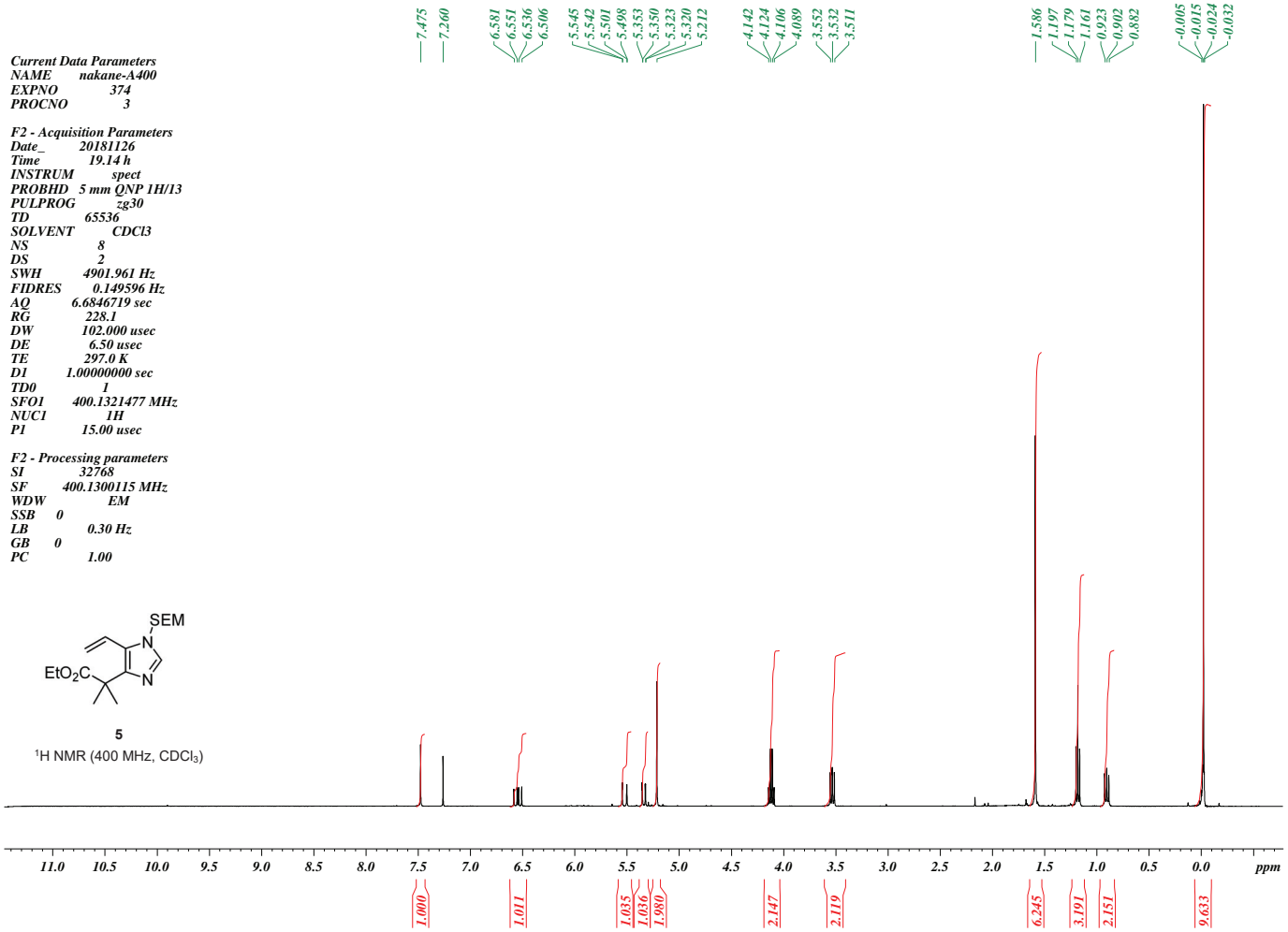
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
F2 - Processing parameters
SI 32768
SF 100.6127752 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
SEM
Br
N
N
EtO2C
3b
13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 4901.961 Hz
FIDRES 0.149596 Hz
AQ 6.6846719 sec
RG 228.1
DW 102.000 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321477 MHz
NUC1 1H
P1 15.00 usec
F2 - Processing parameters
SI 32768
SF 400.1300115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
SEM
N
N
EtO2C
5
1H NMR (400 MHz, CDCl3)
7.475
7.260
6.581
6.551
6.536
6.506
5.545
5.542
5.501
5.498
5.353
5.350
5.323
5.320
5.212
4.142
4.124
4.106
4.089
3.552
3.532
3.511
1.586
1.197
1.179
1.161
0.923
0.902
0.882
-0.005
-0.015
-0.024
-0.032
11.0 10.5 10.0 9.5 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 ppm
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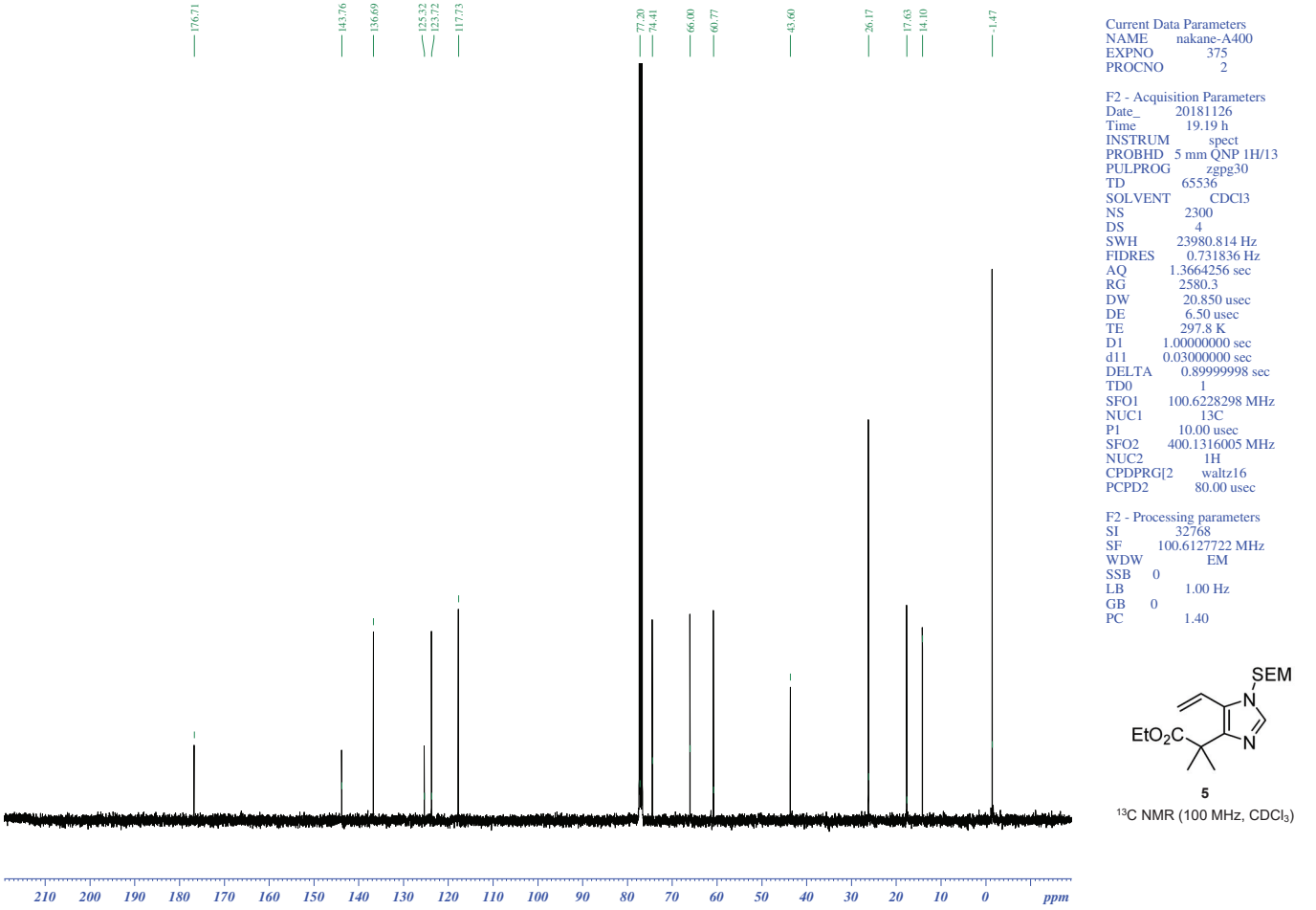
1.035
1.036
1.980
2.147
2.119
6.245
3.191
2.151
9.633


176.71
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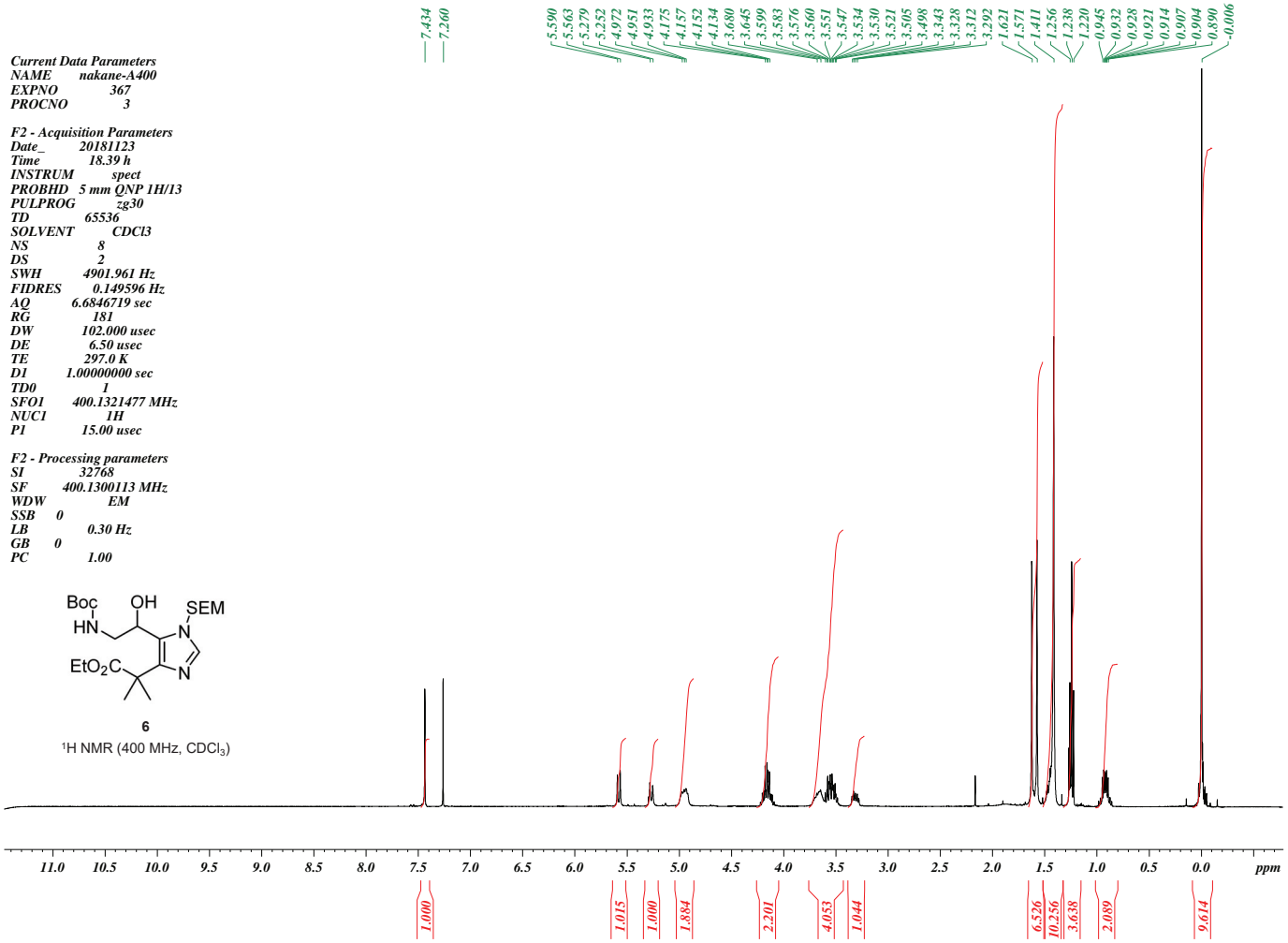
136.69
125.32
123.72
117.73
77.20
74.41
66.00
60.77
43.60
26.17
17.63
14.10
-1.47
Current Data Parameters
NAME nakane-A400
EXPNO 375
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181126
Time 19.19 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2300
DS 4
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
F2 - Processing parameters
SI 32768
SF 100.6127722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
SEM
N
N
EtO2C
5
13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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EXPNO 367
PROCNO 3
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 4901.961 Hz
FIDRES 0.149596 Hz
AQ 6.6846719 sec
RG 181
DW 102.000 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321477 MHz
NUC1 1H
P1 15.00 usec
F2 - Processing parameters
SI 32768
SF 400.1300113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Boc
OH
SEM
HN
N
EtO2C
N
6
1H NMR (400 MHz, CDCl3)
7.434
7.260
5.590
5.563
5.279
5.252
4.972
4.951
4.933
4.175
4.157
4.152
4.134
3.680
3.645
3.599
3.583
3.576
3.560
3.551
3.547
3.534
3.530
3.521
3.505
3.498
3.343
3.328
3.312
3.292
1.621
1.571
1.411
1.256
1.238
1.220
0.945
0.932
0.928
0.921
0.914
0.907
0.904
0.890
-0.006
11.0 10.5 10.0 9.5 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 ppm
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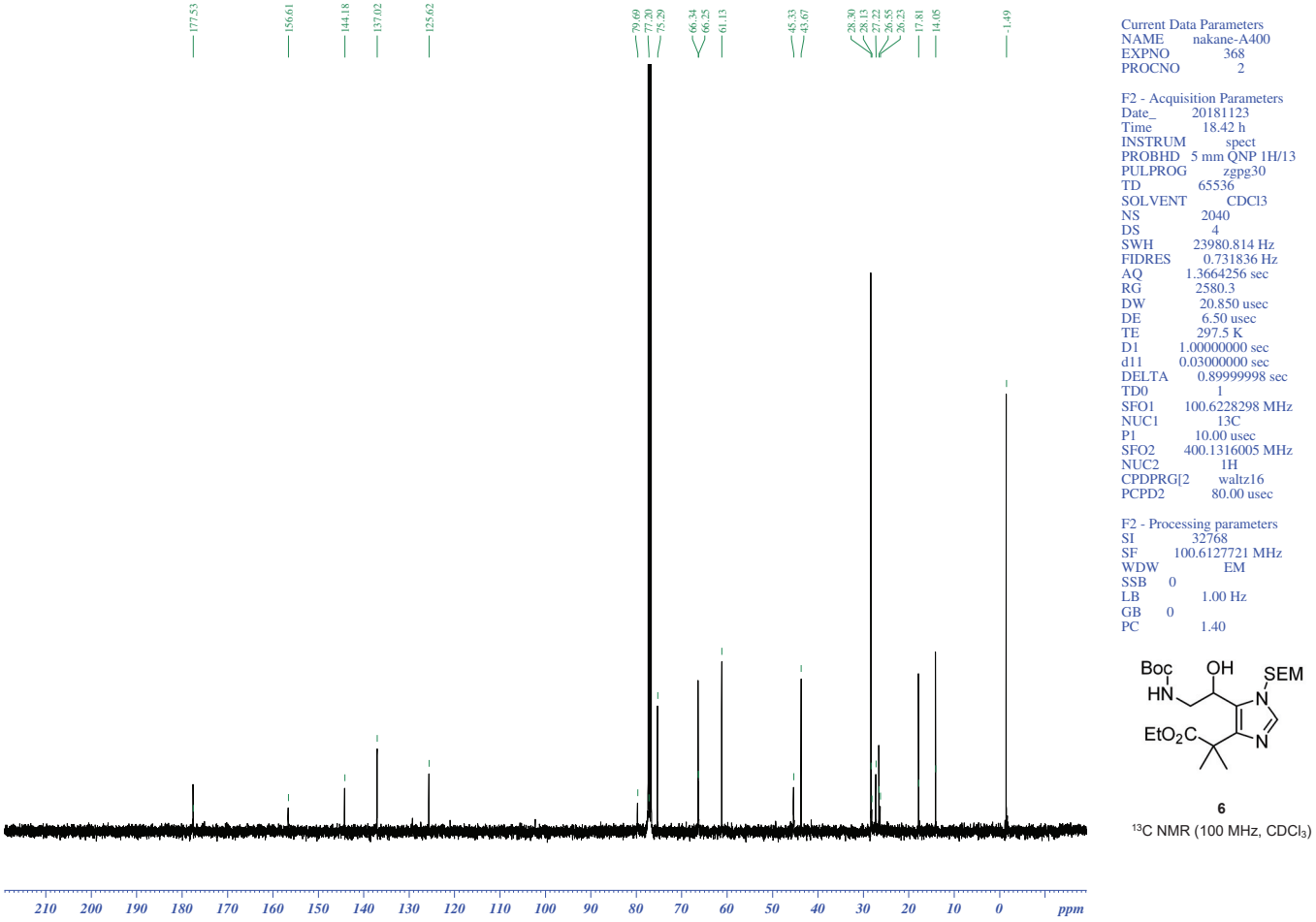
1.015
1.000
1.884
2.201
4.053
1.044
6.526
10.256
3.638
2.089
9.614


177.53
156.61
144.18
137.02
125.62
79.69
77.20
75.29
66.34
66.25
61.13
45.33
43.67
28.30
28.13
27.22
26.35
26.25
17.81
14.05
-1.49
Current Data Parameters
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EXPNO 368
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181123
Time 18.42 h
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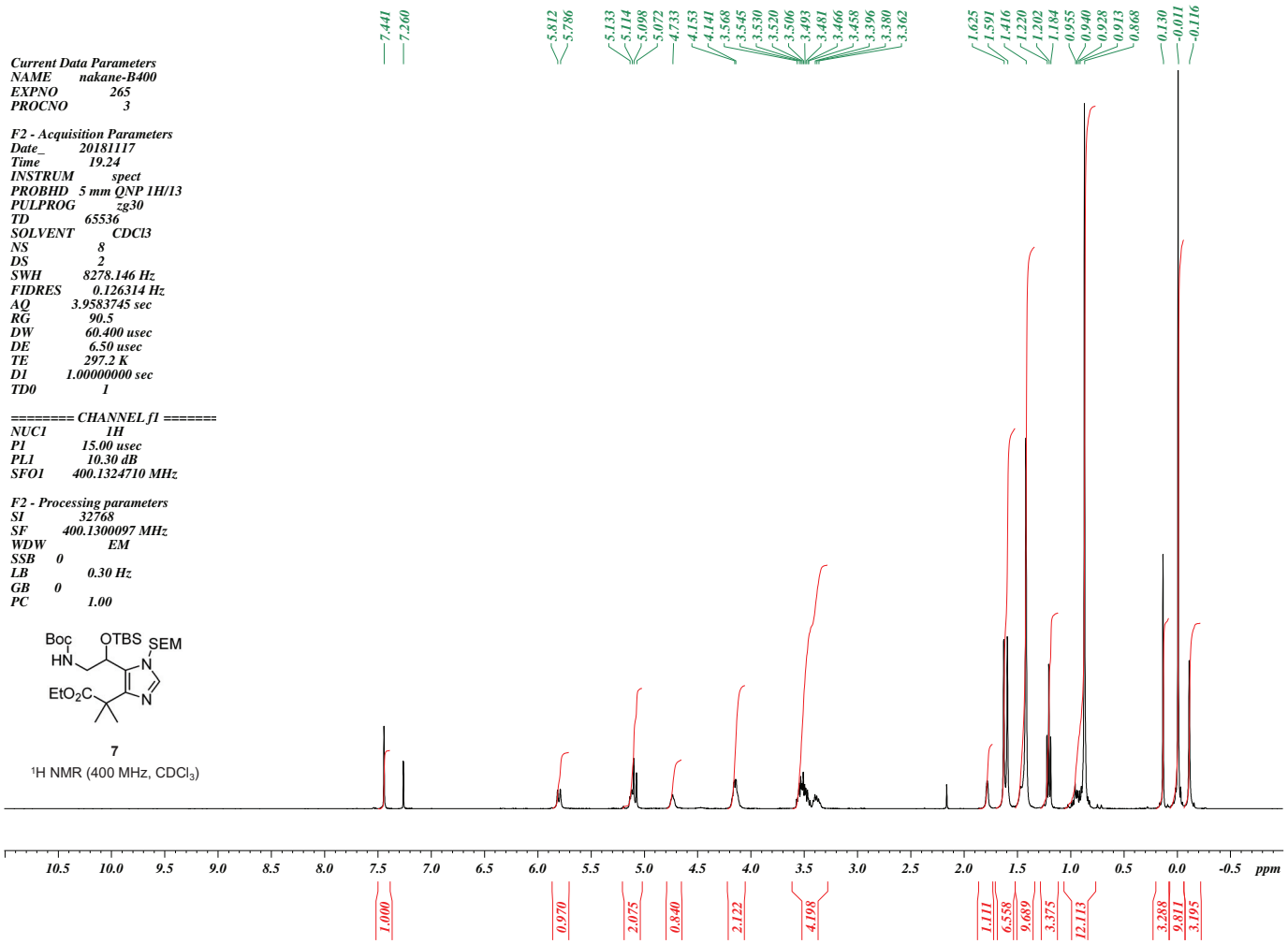
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2040
DS 4
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
F2 - Processing parameters
SI 32768
SF 100.6127721 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
OH
SEM
HN
N
EtO2C
N
6
13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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EXPNO 265
PROCNO 3
F2 - Acquisition Parameters
Date_ 20181117
Time 19.24
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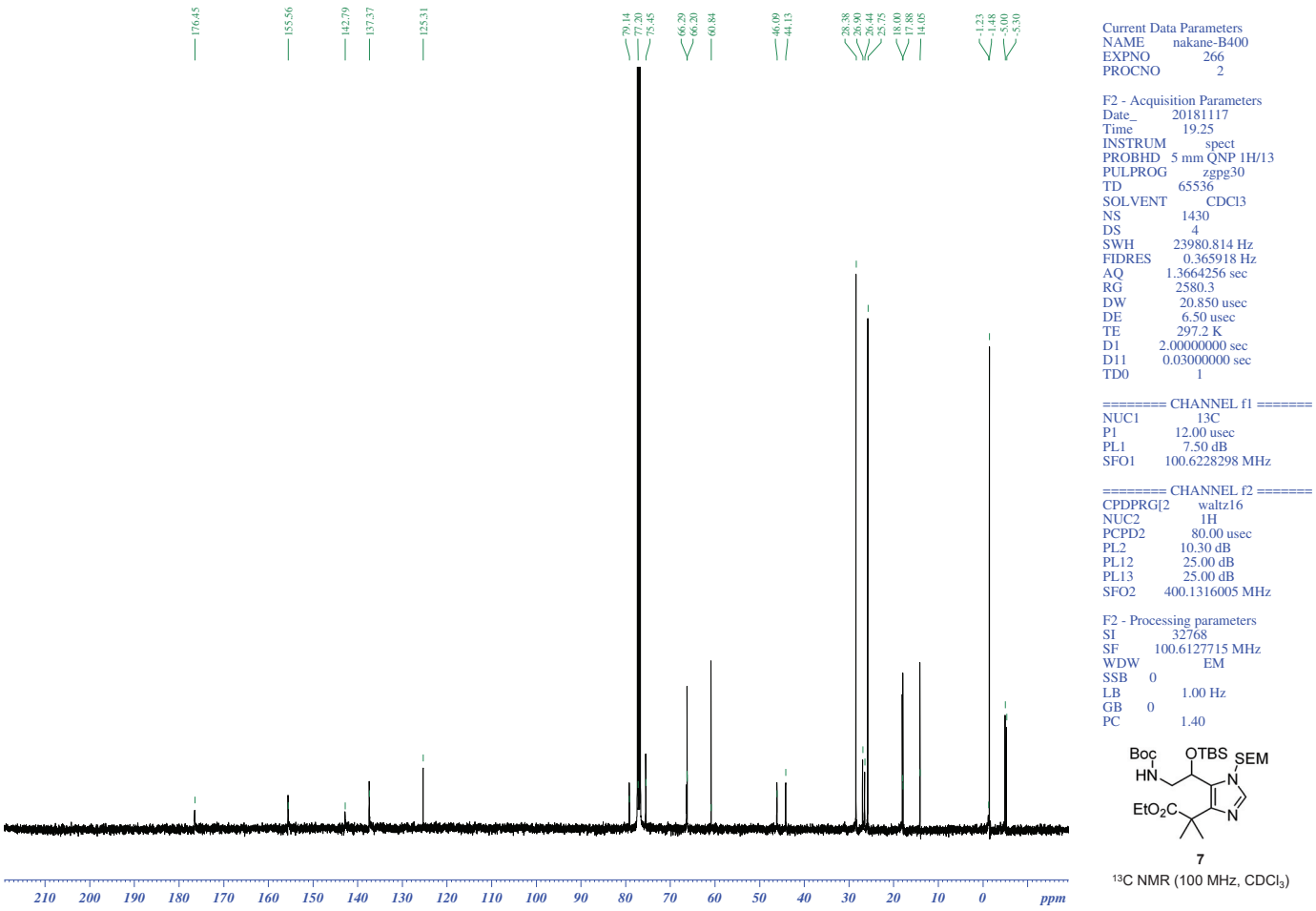
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 90.5
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 1H
P1 15.00 usec
PL1 10.30 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Boc
OTBS
SEM
HN
N
N
EtO2C
7
1H NMR (400 MHz, CDCl3)
7.441
7.260
5.812
5.786
5.133
5.114
5.098
5.072
4.733
4.153
4.141
3.568
3.545
3.530
3.520
3.506
3.493
3.481
3.466
3.458
3.396
3.380
3.362
1.625
1.591
1.416
1.220
1.202
1.184
0.955
0.940
0.928
0.913
0.868
0.130
-0.011
-0.116
10.5 10.0 9.5 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 -0.5 ppm
1.000
0.970
2.075
0.840
2.122
4.198
1.111
6.558
9.689
3.375
12.113
3.288
9.811
3.195


176.45
155.56
142.79
137.37
125.31
79.14
77.20
75.45
66.29
66.20
60.84
46.09
44.13
28.38
26.90
26.44
25.75
18.00
17.88
14.05
-1.23
-1.48
-5.00
-5.30
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EXPNO 266
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181117
Time 19.25
INSTRUM spect
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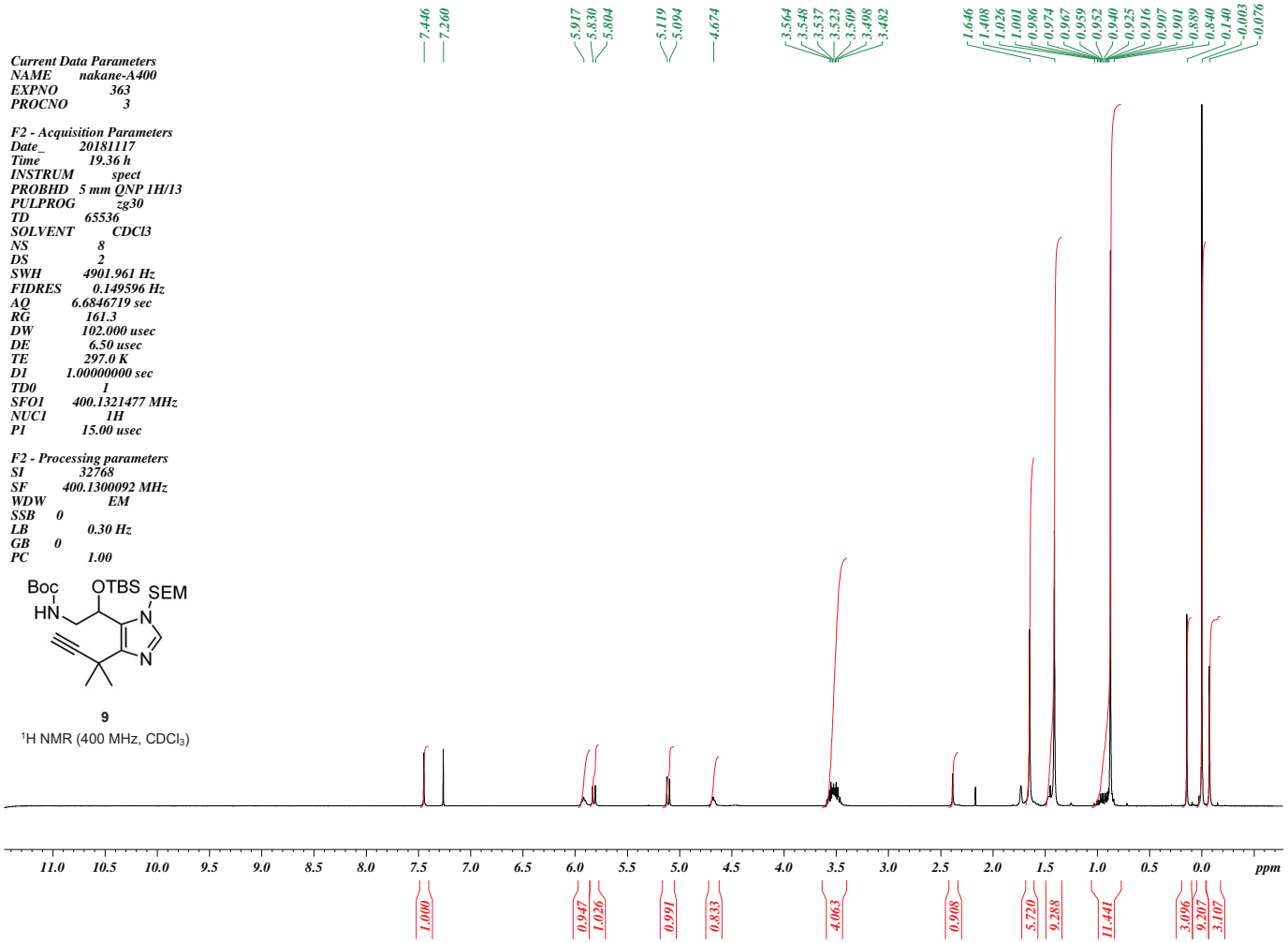
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1430
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 12.00 usec
PL1 7.50 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 =======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 10.30 dB
PL12 25.00 dB
PL13 25.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
OTBS
SEM
HN
N
N
EtO2C
7
13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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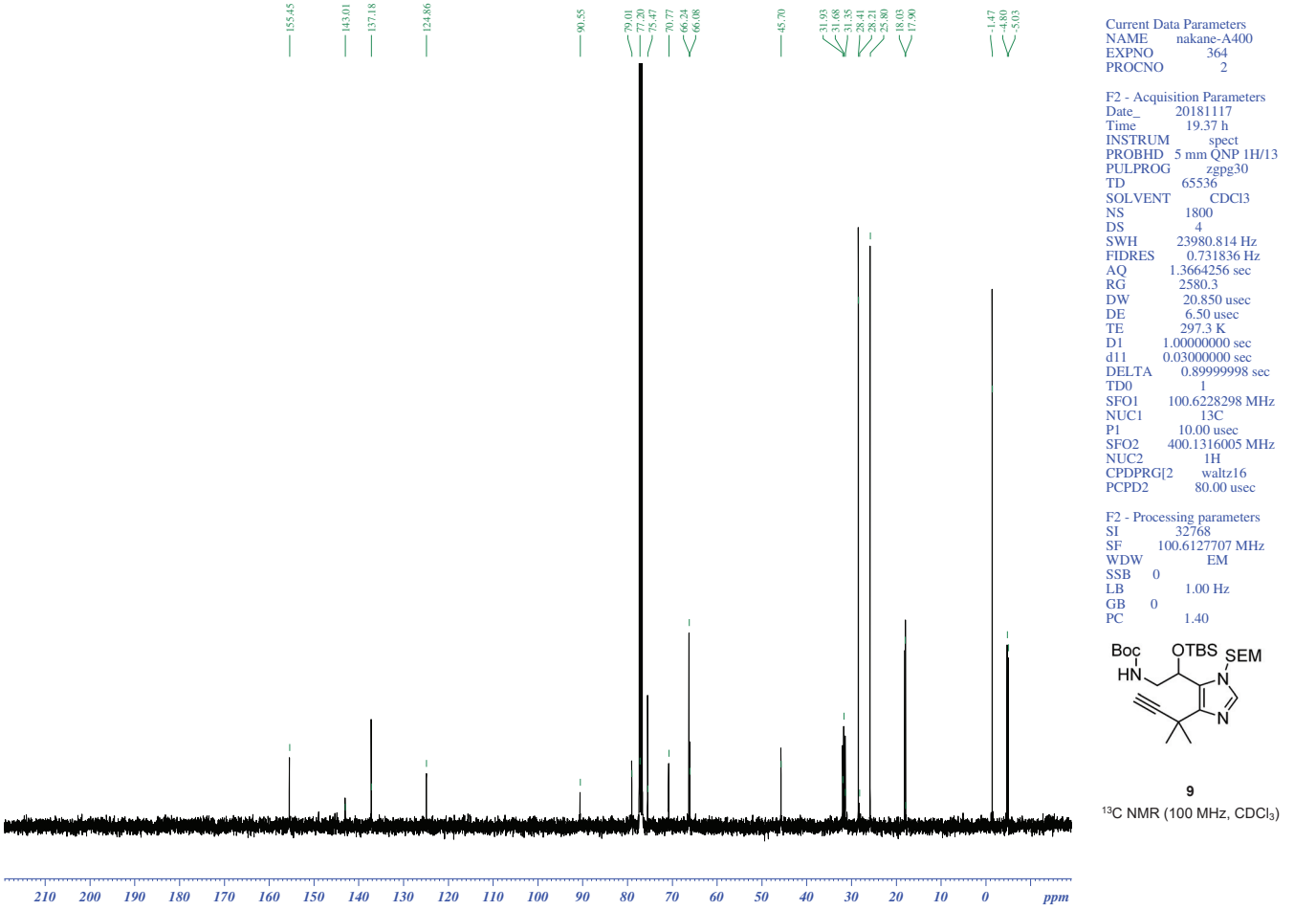
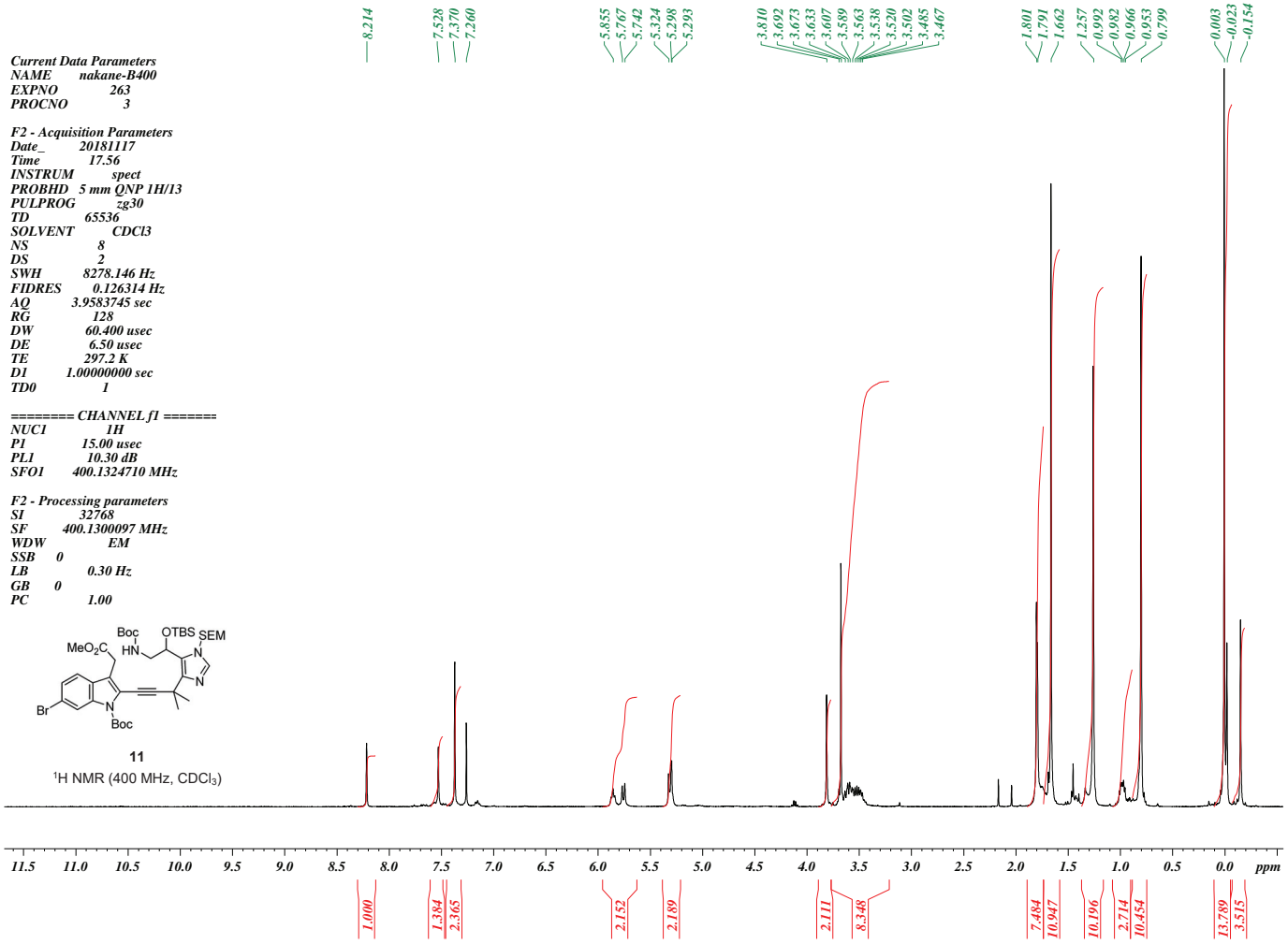
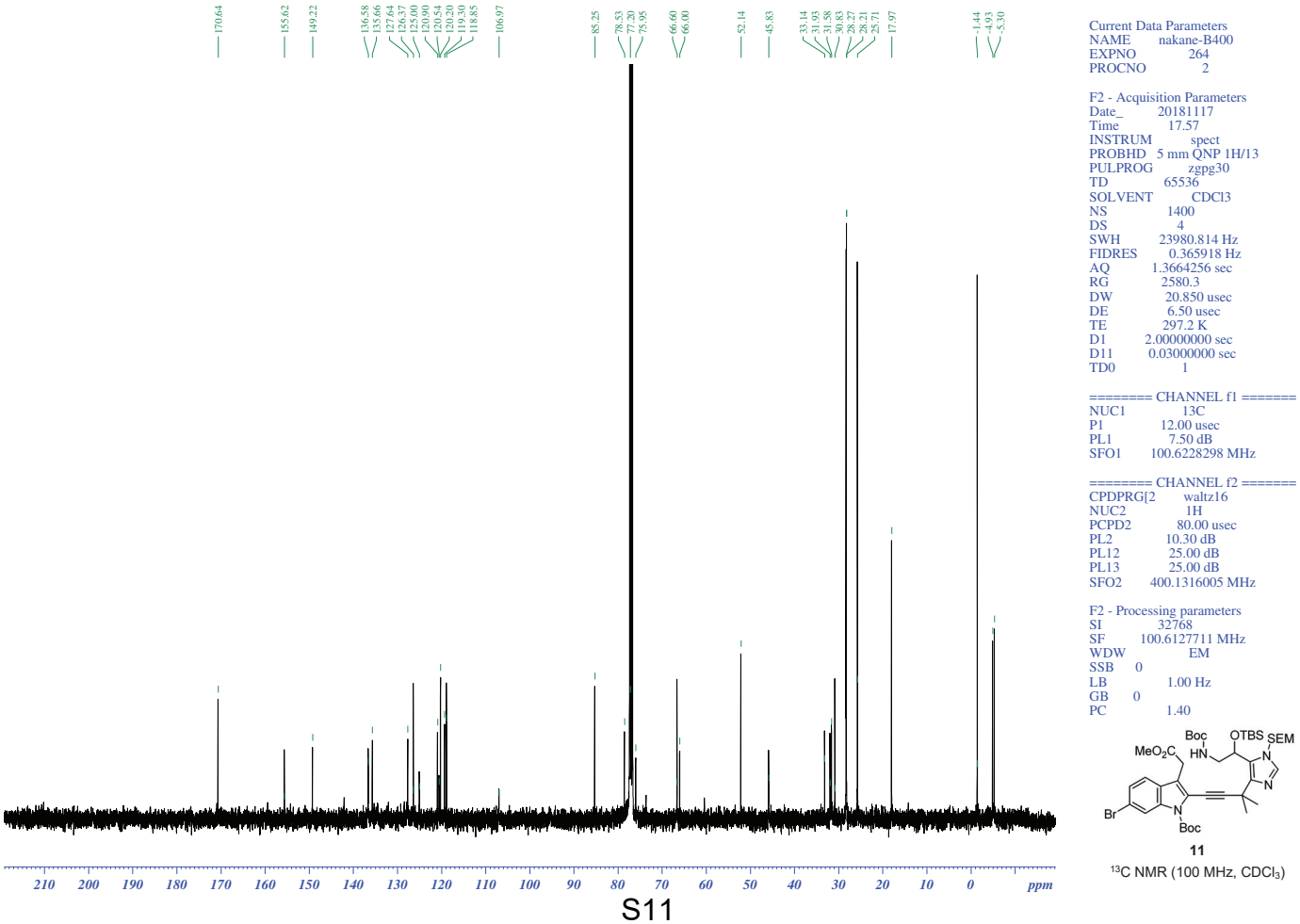
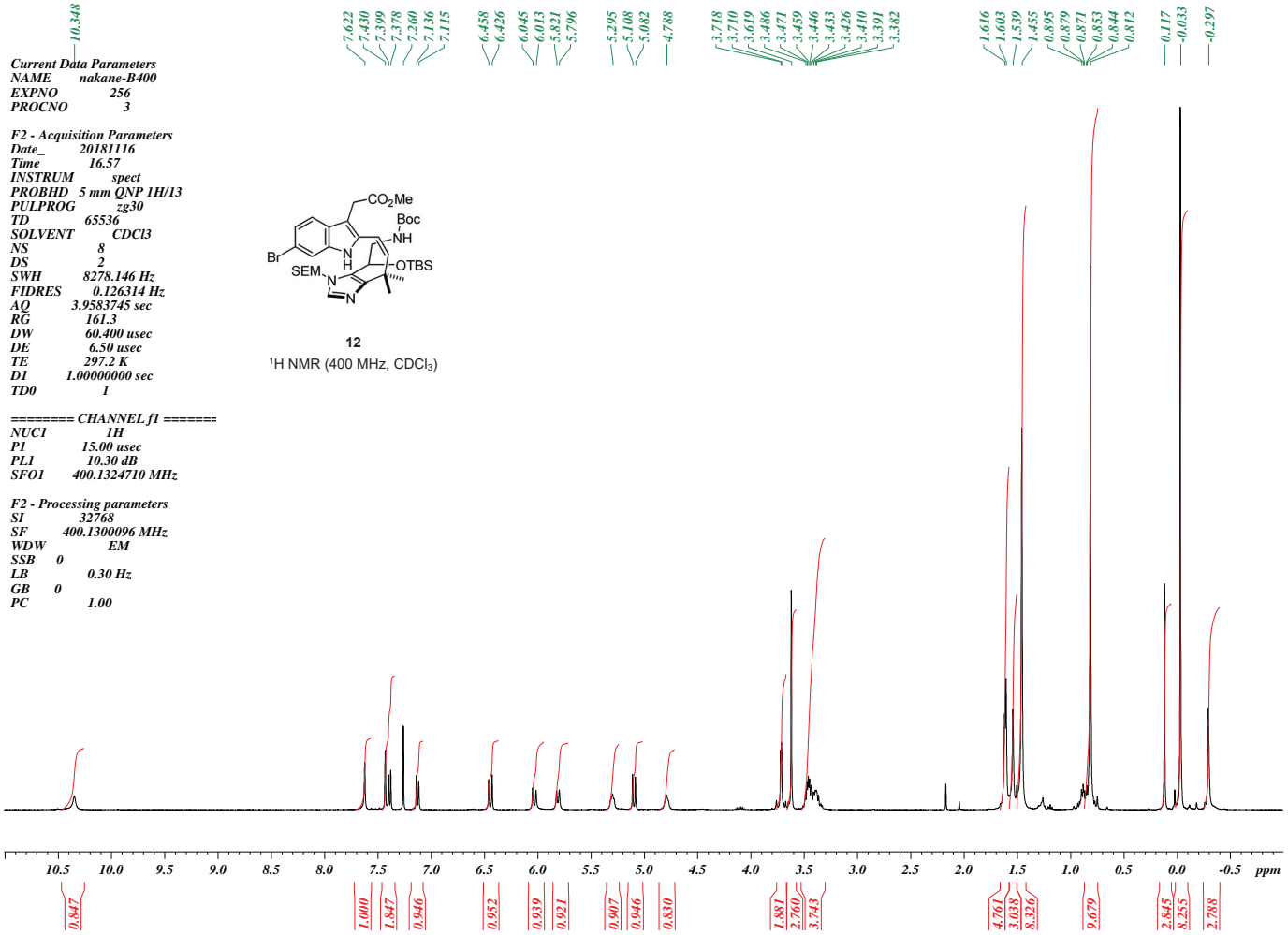
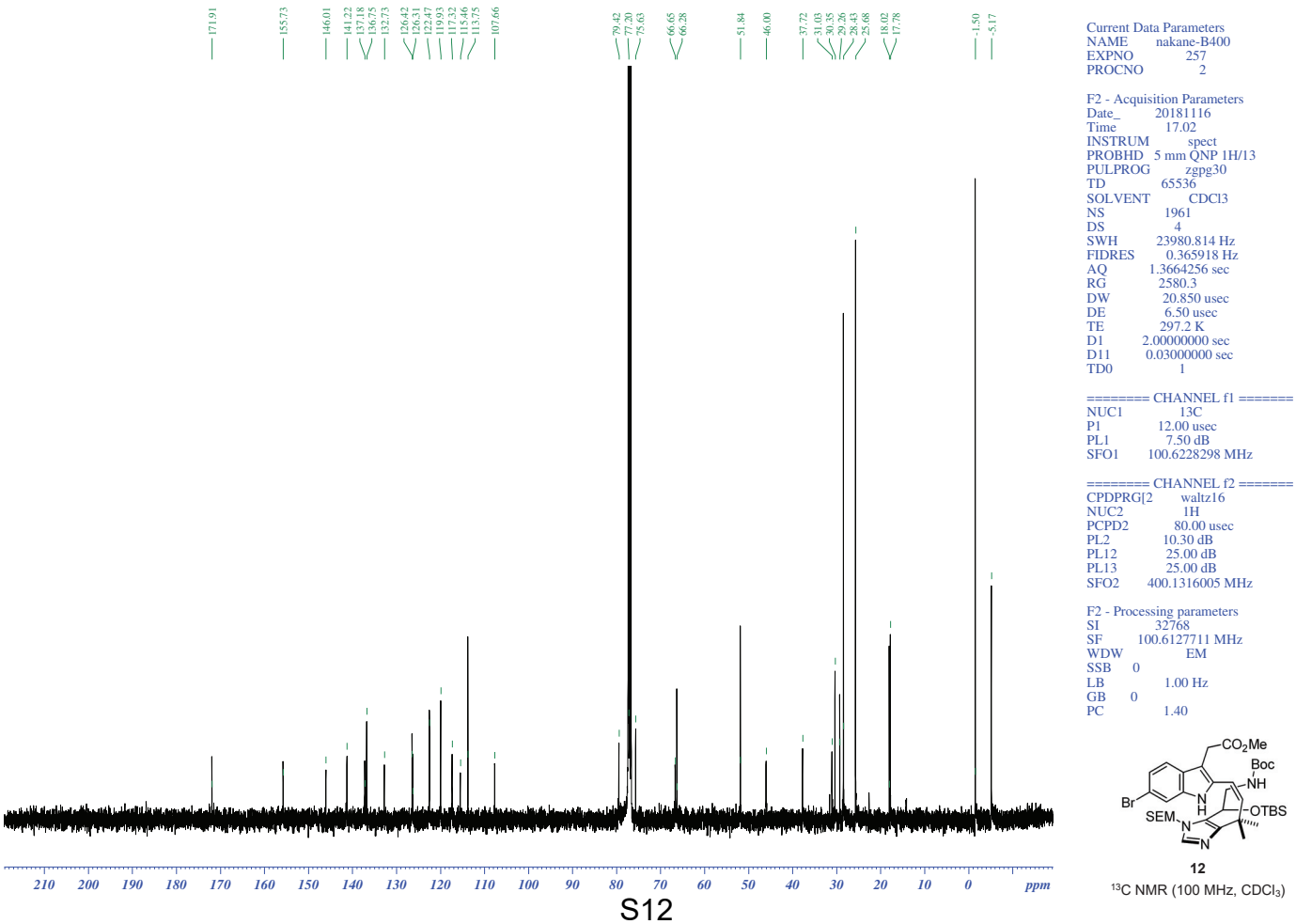
EXPNO 363
PROCNO 3
F2 - Acquisition Parameters
Date_ 20181117
Time 19.36 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 4901.961 Hz
FIDRES 0.149596 Hz
AQ 6.6846719 sec
RG 161.3
DW 102.000 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321477 MHz
NUC1 1H
P1 15.00 usec
F2 - Processing parameters
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Boc
OTBS
SEM
HN
N
N
9
1H NMR (400 MHz, CDCl3)
7.446
7.260
5.917
5.830
5.804
5.119
5.094
4.674
3.564
3.548
3.537
3.523
3.509
3.498
3.482
1.646
1.408
1.026
1.001
0.986
0.974
0.967
0.959
0.952
0.940
0.925
0.916
0.907
0.901
0.889
0.840
0.140
-0.003
-0.076
11.0 10.5 10.0 9.5 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 ppm
1.000
0.947
1.026
0.991
0.833
4.063
0.908
5.720
9.288
11.441
3.096
9.207
3.107

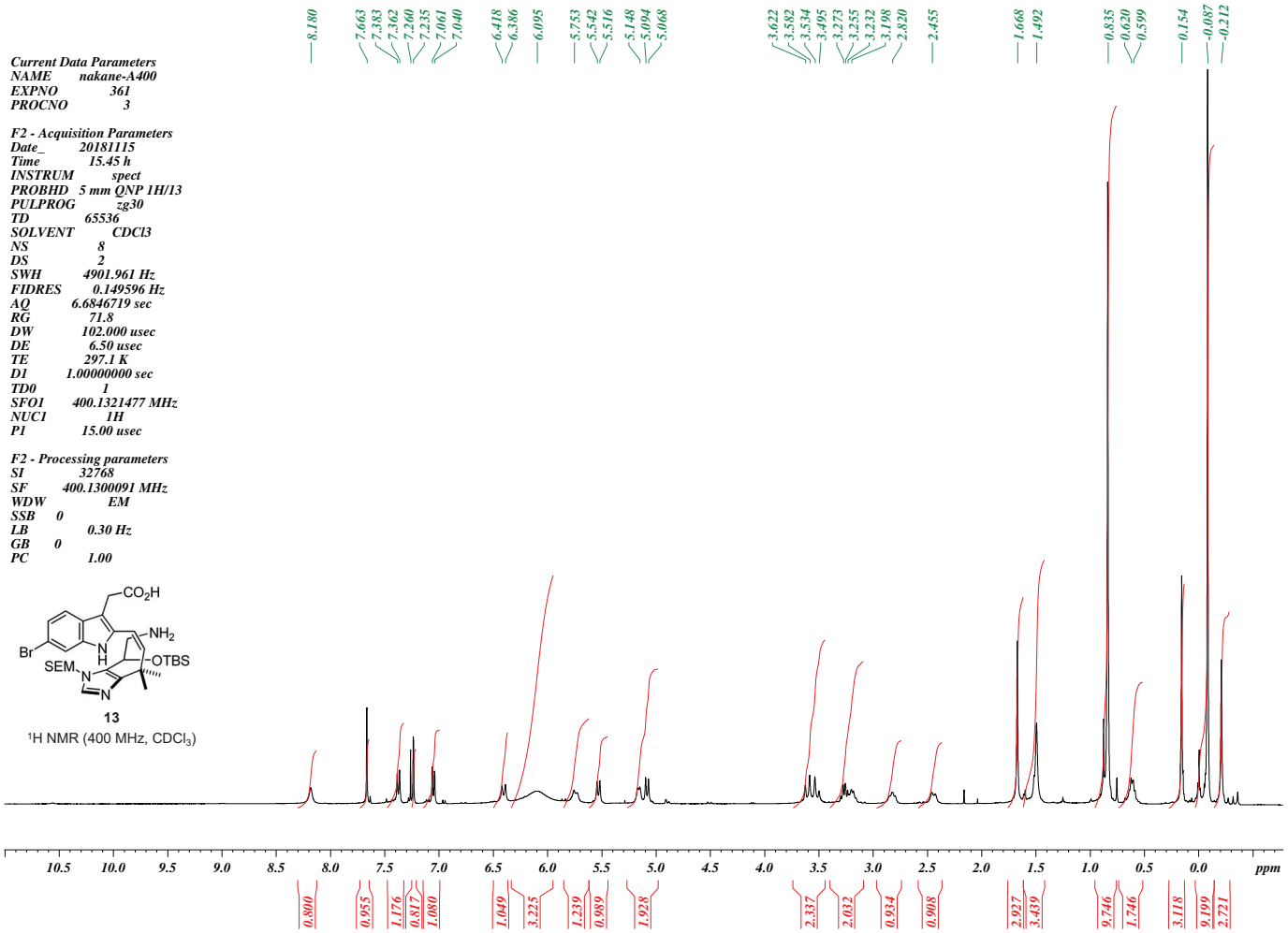

155.45
143.01
137.18
124.86
90.55
79.01
77.20
75.47
70.77
66.24
66.08
45.70
31.93
31.68
31.35
28.41
28.21
25.80
18.03
17.90
-1.47
-4.80
-5.03
Current Data Parameters
NAME nakane-A400
EXPNO 364
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181117
Time 19.37 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1800
DS 4
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
F2 - Processing parameters
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
OTBS
SEM
HN
N
N
9
13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Current Data Parameters
NAME nakane-B400
EXPNO 263
PROCNO 3
F2 - Acquisition Parameters
Date_ 20181117
Time 17.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 128
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 1H
P1 15.00 usec
PL1 10.30 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Boc
OTBS
SEM
MeO2C
HN
N
N
Br
N
Boc
11
1H NMR (400 MHz, CDCl3)
8.214
7.528
7.370
7.260
5.855
5.767
5.742
5.324
5.298
5.293
3.810
3.692
3.673
3.633
3.607
3.589
3.563
3.538
3.520
3.502
3.485
3.467
1.801
1.791
1.662
1.257
0.992
0.982
0.966
0.953
0.799
0.003
-0.023
-0.154
11.5 11.0 10.5 10.0 9.5 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 ppm
1.000
1.384
2.365
2.152
2.189
2.111
8.348
7.484
10.947
10.196
2.714
10.454
13.789
3.515


170.64
155.62
149.22
136.58
135.66
127.64
126.37
125.00
120.90
120.54
120.20
119.30
118.85
106.97
85.25
78.53
77.20
75.95
66.60
66.00
52.14
45.83
33.14
31.93
31.58
30.83
28.27
28.21
25.71
17.97
-1.44
-4.93
-5.30
Current Data Parameters
NAME nakane-B400
EXPNO 264
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181117
Time 17.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1400
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 12.00 usec
PL1 7.50 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 =======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 10.30 dB
PL12 25.00 dB
PL13 25.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127711 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Boc
OTBS
SEM
MeO2C
HN
N
N
Br
N
Boc
11
13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Current Data Parameters
NAME nakane-B400
EXPNO 256
PROCNO 3
F2 - Acquisition Parameters
Date_ 20181116
Time 16.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 161.3
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 1H
P1 15.00 usec
PL1 10.30 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
CO2Me
Boc
NH
Br
SEM
OTBS
12
1H NMR (400 MHz, CDCl3)
ppm


Current Data Parameters
NAME nakane-B400
EXPNO 257
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181116
Time 17.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1961
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 12.00 usec
PL1 7.50 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 =======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 10.30 dB
PL12 25.00 dB
PL13 25.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127711 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
CO2Me
Boc
NH
Br
SEM
OTBS
12
13C NMR (100 MHz, CDCl3)
ppm



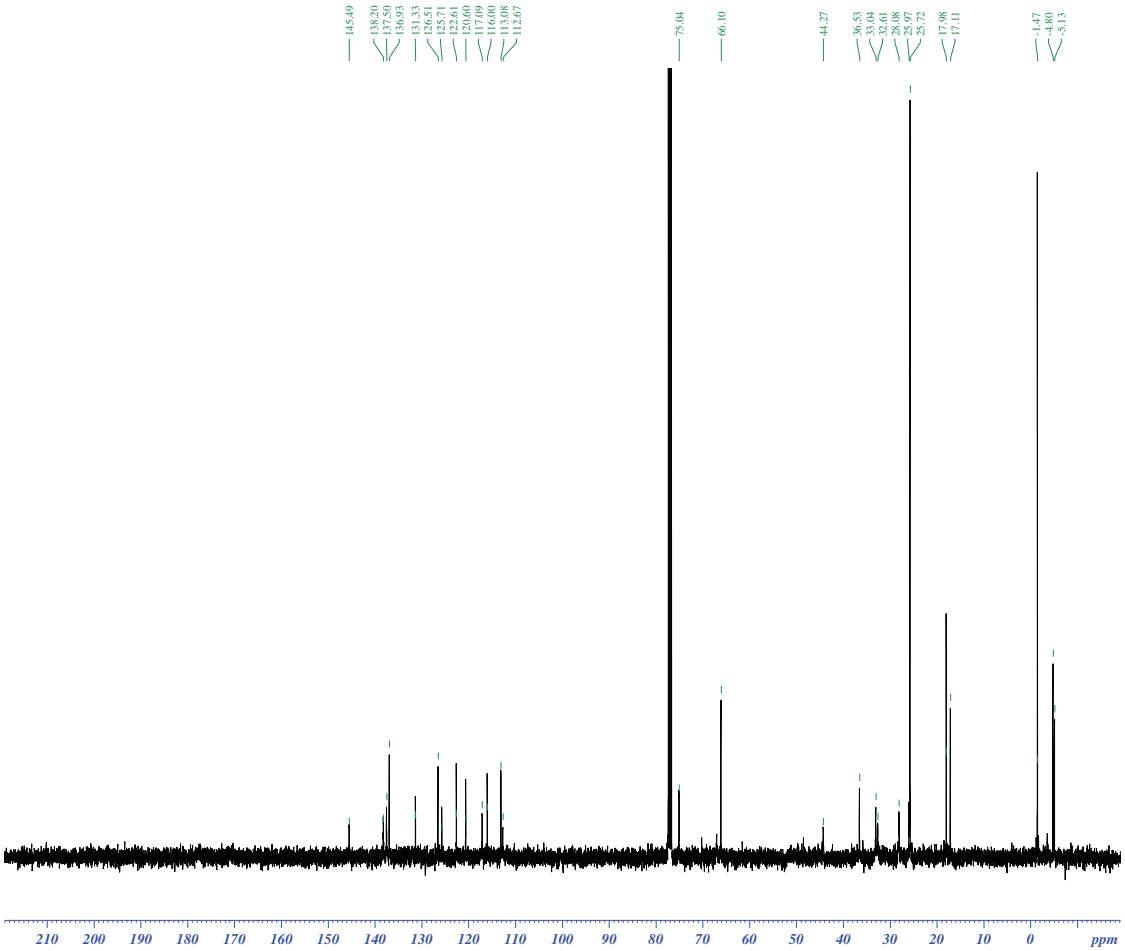
Current Data Parameters
NAME nakane-A400
EXPNO 361
PROCNO 3

F2 - Acquisition Parameters
Date_ 20181115
Time 15.45 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 4901.961 Hz
FIDRES 0.149596 Hz
AQ 6.6846719 sec
RG 71.8
DW 102.000 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321477 MHz
NUC1 1H
P1 15.00 usec

F2 - Processing parameters
SI 32768
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13

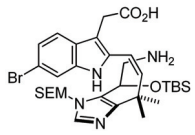
^{1}H NMR (400 MHz, $CDCl_3$)



Current Data Parameters
NAME nakane-A400
EXPNO 362
PROCNO 3

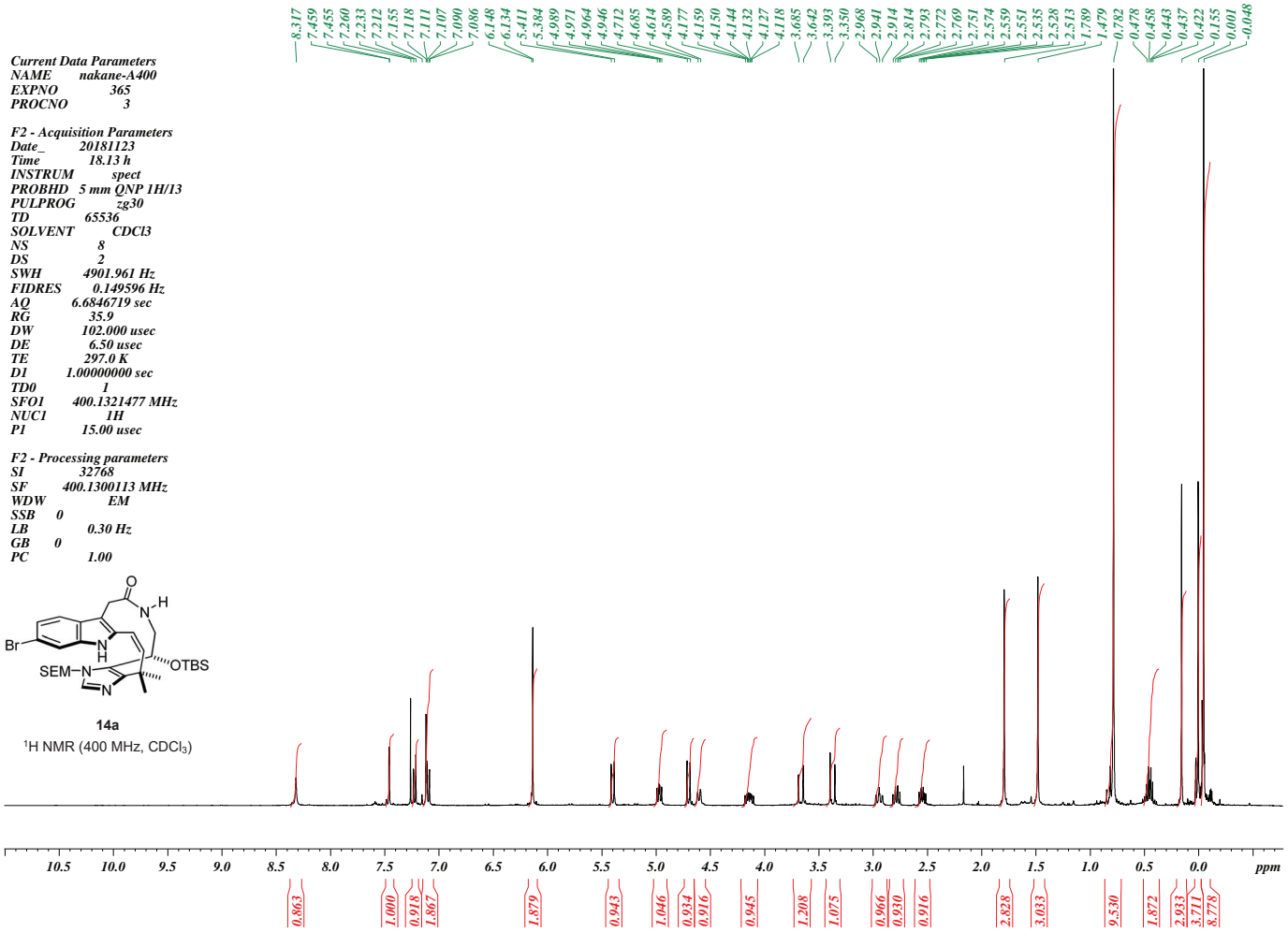
F2 - Acquisition Parameters
Date_ 20181115
Time 15.49 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec

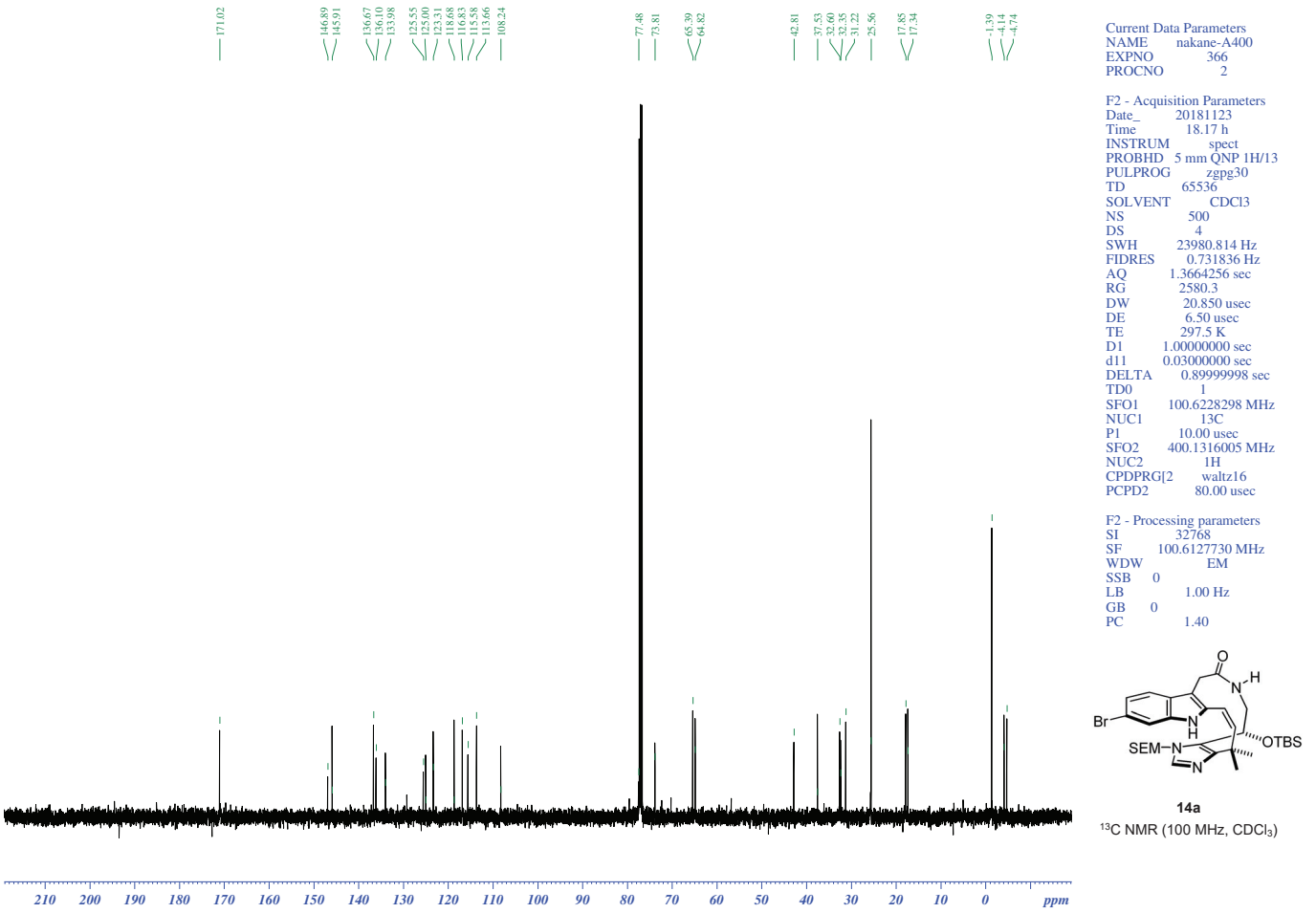
F2 - Processing parameters
SI 32768
SF 100.6127737 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



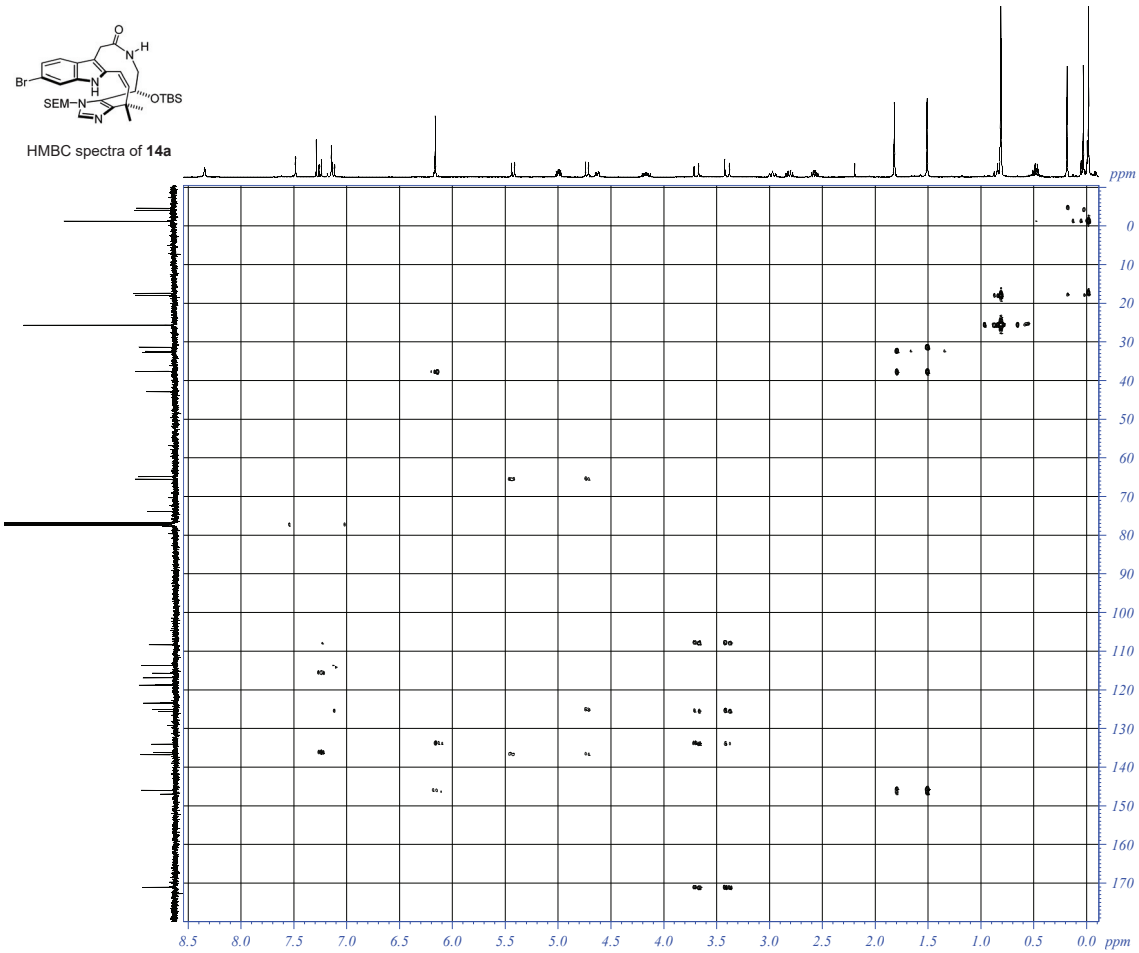
13

^{13}C NMR (100 MHz, $CDCl_3$)


Current Data Parameters
NAME nakane-A400
EXPNO 365
PROCNO 3
F2 - Acquisition Parameters
Date_ 20181123
Time 18.13 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 4901.961 Hz
FIDRES 0.149596 Hz
AQ 6.6846719 sec
RG 35.9
DW 102.000 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321477 MHz
NUC1 1H
P1 15.00 usec
F2 - Processing parameters
SI 32768
SF 400.1300113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
14a
1H NMR (400 MHz, CDCl3)
ppm


Current Data Parameters
NAME nakane-A400
EXPNO 366
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181123
Time 18.17 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 4
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
F2 - Processing parameters
SI 32768
SF 100.6127730 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
14a
13C NMR (100 MHz, CDCl3)
ppm

HMBC spectra of **14a**



Current Data Parameters
NAME nakane-A400
EXPNO 155
PROCNO 2

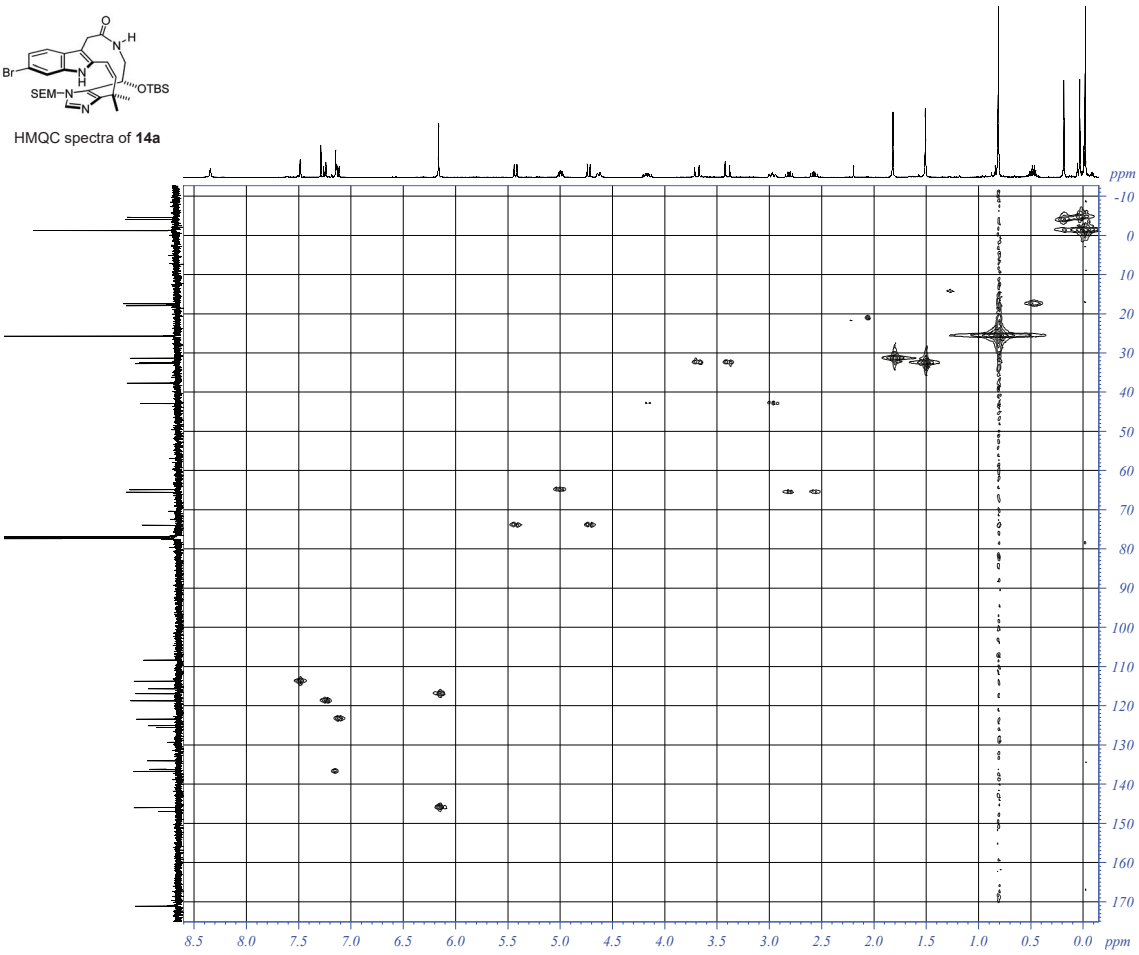
F2 - Acquisition Parameters
Date_ 20161028
Time 14.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG hmbcgplpndqf
TD 2048
SOLVENT CDCl3
NS 18
DS 16
SWH 3633.721 Hz
FIDRES 1.774278 Hz
AQ 0.2818048 sec
RG 16384
DW 137.600 usec
DE 6.50 usec
TE 297.0 K
CNST2 145.0000000
CNST13 10.0000000
d0 0.00000300 sec
D1 1.07711995 sec
d2 0.00344828 sec
d6 0.05000000 sec
D16 0.00010000 sec
in0 0 sec
ST1CNT 0
d0orig 0.00000300 sec
ph1loop 0
t1loop 0
SFO1 400.1317406 MHz
NUC1 1H
P1 15.00 usec
p2 30.00 usec
PLW1 -1.00000000 W
SFO2 100.6228298 MHz
NUC2 13C
P3 10.00 usec
PLW2 -1.00000000 W
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 100.6228 MHz
FIDRES 187.350113 Hz
SW 238.324 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 400.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 100.6127690 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0

HMQC spectra of **14a**



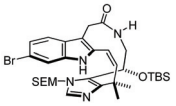
Current Data Parameters
NAME nakane-A400
EXPNO 157
PROCNO 2

F2 - Acquisition Parameters
Date_ 20161031
Time 17.22 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG hmqcgpqf
TD 2048
SOLVENT CDCl3
NS 14
DS 16
SWH 3633.721 Hz
FIDRES 3.548556 Hz
AQ 0.2818048 sec
RG 26008
DW 137.600 usec
DE 6.50 usec
TE 297.1 K
CNST2 145.0000000
d0 0.00000300 sec
D1 1.50000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
d13 0.00000400 sec
D16 0.00010000 sec
DELTA1 0.00232428 sec
in0 0 sec
SFO1 400.1317406 MHz
NUC1 1H
P1 15.00 usec
p2 30.00 usec
SFO2 100.6228298 MHz
NUC2 13C
CPDPRG[2 garp
P3 10.00 usec
PCPD2 70.00 usec
GPNAM[1] SINE.100
GPZ1 50.00 %
GPNAM[2] SINE.100
GPZ2 30.00 %
GPNAM[3] SINE.100
GPZ3 40.10 %
P16 1000.00 usec

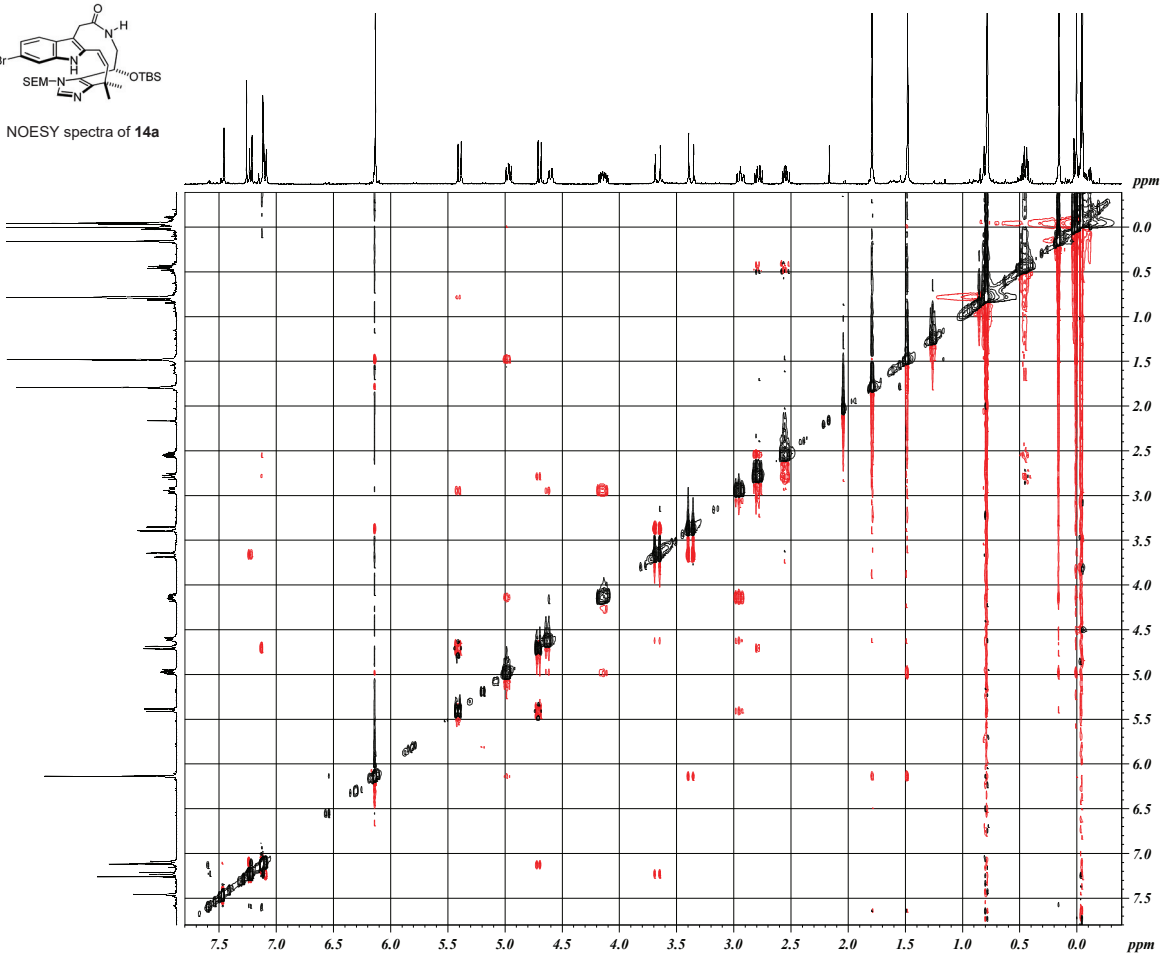
F1 - Acquisition parameters
TD 256
SFO1 100.6228 MHz
FIDRES 187.350113 Hz
SW 238.324 ppm
FnMODE QF

F2 - Processing parameters
SI 1024
SF 400.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 100.6127690 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0



NOESY spectra of **14a**




```
Current Data Parameters
NAME          nakane-A400
EXPNO                 180
PROCNO                  4

F2 - Acquisition Parameters
Date_             20170112
Time                 15.31 h
INSTRUM              spect
PROBHD   5 mm QNP 1H/13
PULPROG           noesyph
TD                     2048
SOLVENT              CDCl3
NS                      16
DS                      16
SWH             3633.721 Hz
FIDRES          3.548556 Hz
AQ             0.2818048 sec
RG                   35.9
DW                137.600 usec
DE                  6.50 usec
TE                 297.1 K
d0            -0.00001910 sec
D1             1.50000000 sec
D8             0.60000002 sec
in0       0 sec
ST1CNT                   0
SFO1         400.1316618 MHz
NUC1                    1H
P1                  15.00 usec

F1 - Acquisition parameters
TD                     256
SFO1            400.1317 MHz
FIDRES         28.388403 Hz
SW                  9.081 ppm
FnMODE        States-TPPI

F2 - Processing parameters
SI                    1024
SF           400.1300000 MHz
WDW                  QSINE
SSB                      2
LB        0 Hz
GB        0
PC                    1.00

F1 - Processing parameters
SI                    1024
MC2           States-TPPI
SF           400.1300000 MHz
WDW                  QSINE
SSB                      2
LB        0 Hz
GB        0
```

Current Data Parameters
NAME nakazaki-B400
EXPNO 5
PROCNO 2

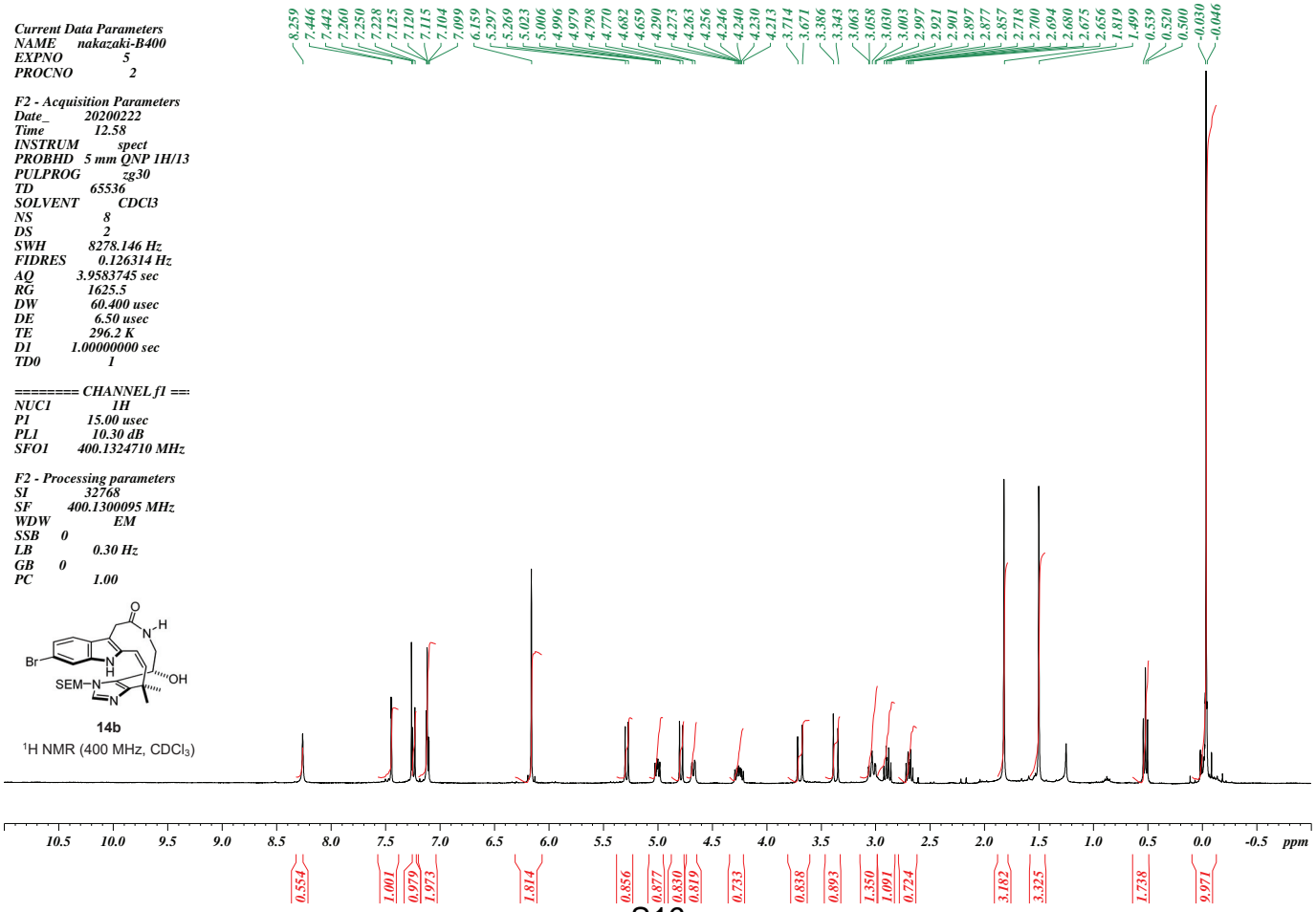
F2 - Acquisition Parameters
Date_ 20200222
Time 12.58
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 1625.5
DW 60.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

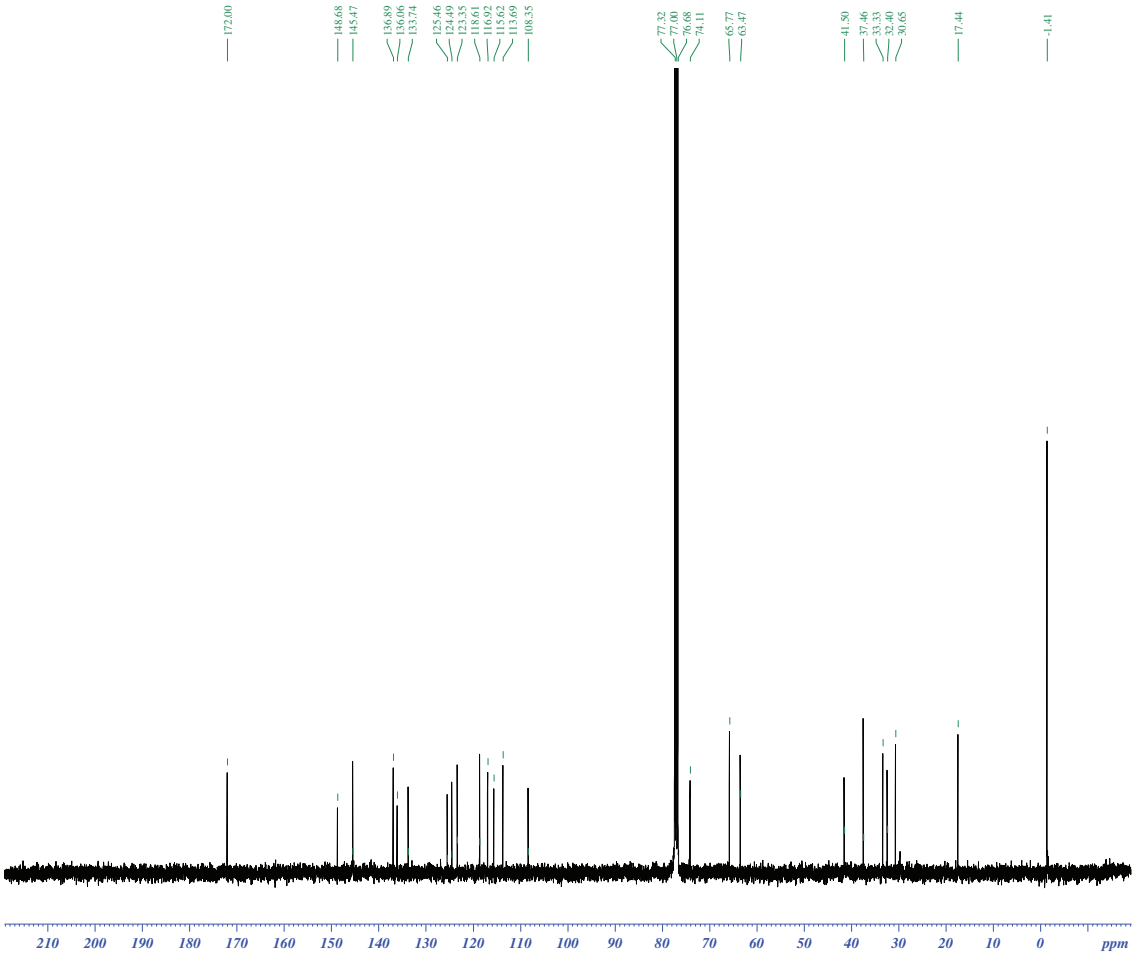
======== CHANNEL f1 ===
NUC1 1H
P1 15.00 usec
PL1 10.30 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

14b

^{1}H NMR (400 MHz, $CDCl_3$)





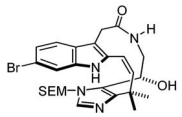
Current Data Parameters
NAME nakazaki-B400
EXPNO 6
PROCNO 2

F2 - Acquisition Parameters
Date_ 20200222
Time 13.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 1024
DW 20.850 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ======
NUC1 13C
P1 12.00 usec
PL1 7.50 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 =======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 10.30 dB
PL12 25.00 dB
PL13 25.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127718 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



14b

^{13}C NMR (100 MHz, $CDCl_3$)

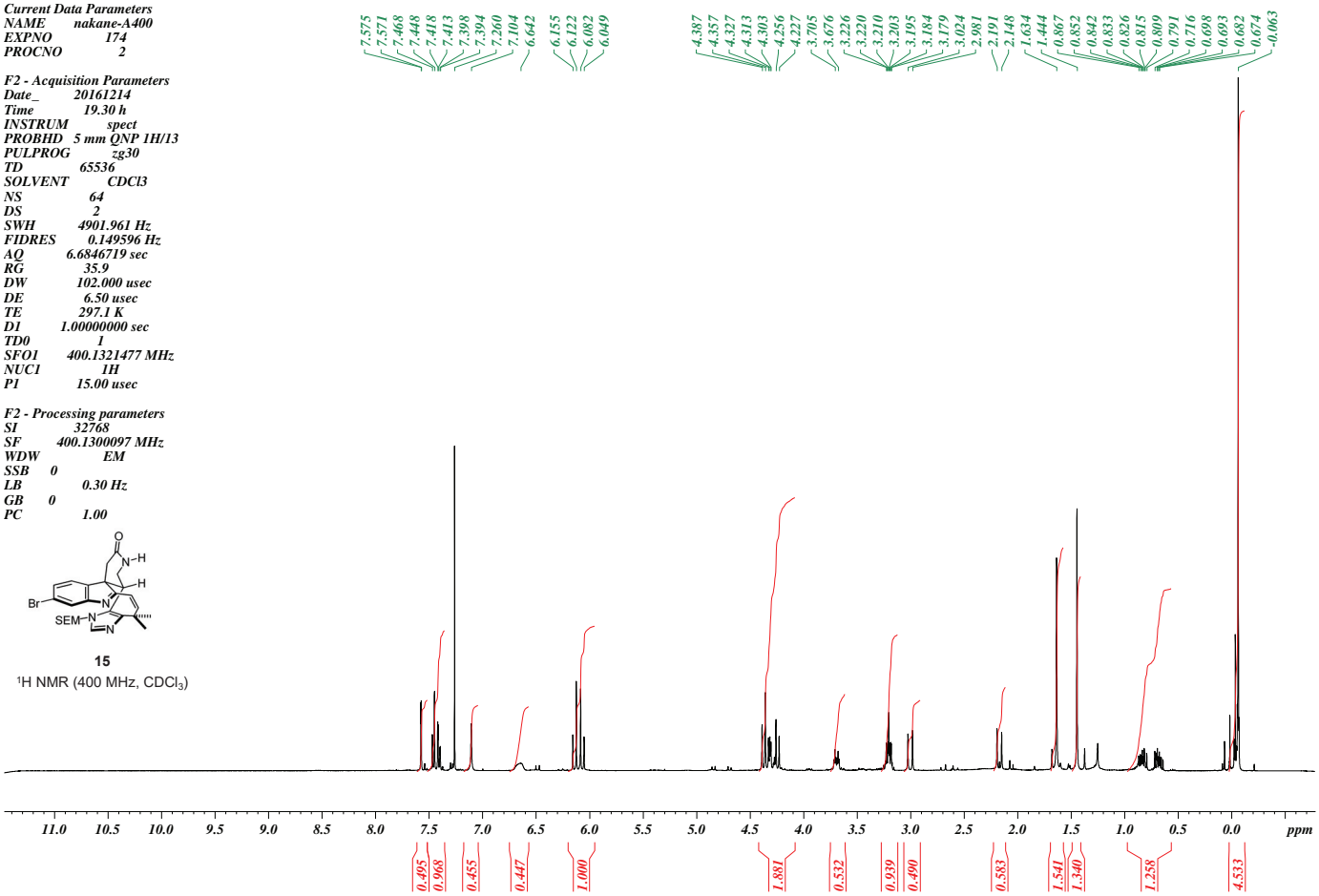
Current Data Parameters
NAME nakane-A400
EXPNO 174
PROCNO 2

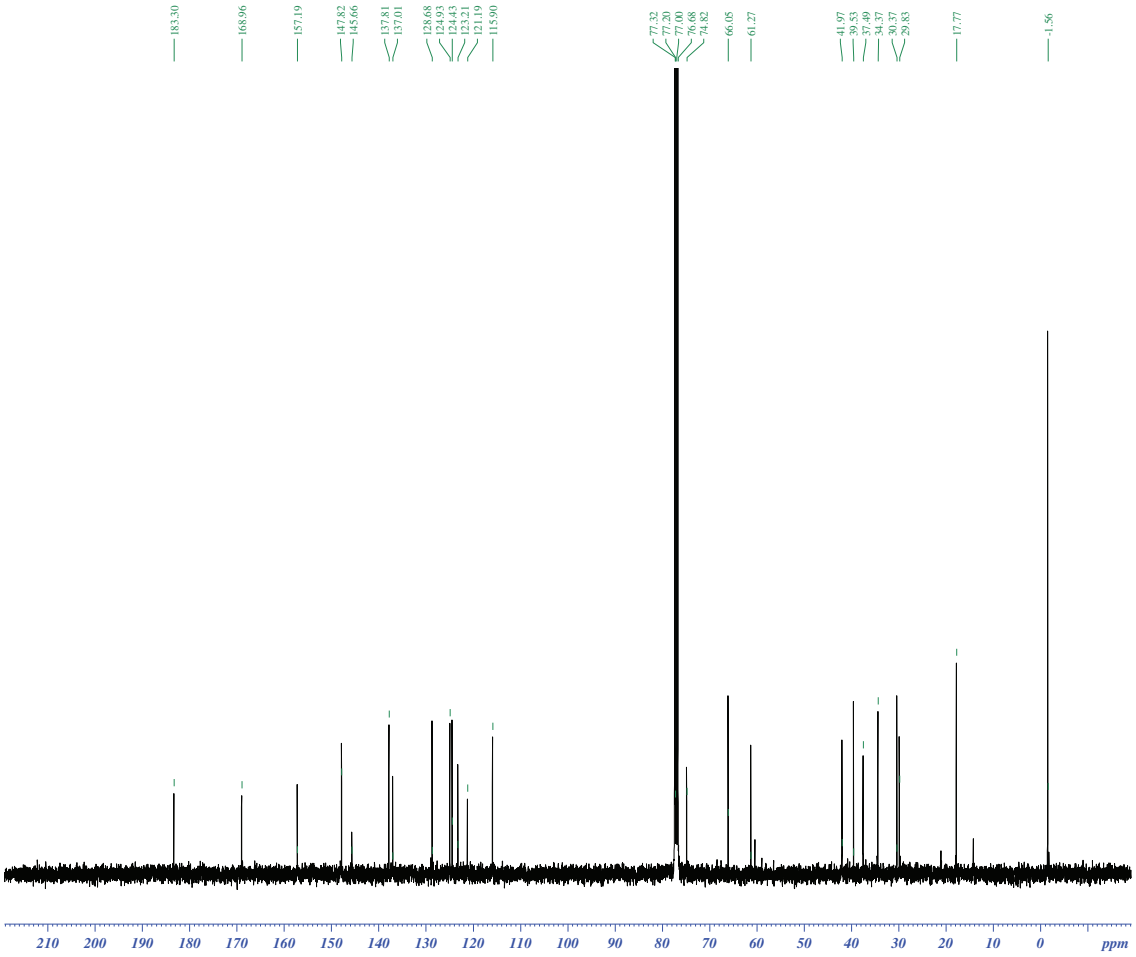
F2 - Acquisition Parameters
Date_ 20161214
Time 19.30 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 4901.961 Hz
FIDRES 0.149596 Hz
AQ 6.6846719 sec
RG 35.9
DW 102.000 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321477 MHz
NUC1 1H
P1 15.00 usec

F2 - Processing parameters
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

15

^{1}H NMR (400 MHz, $CDCl_3$)

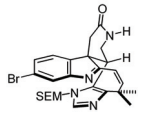




Current Data Parameters
NAME nakane-A400
EXPNO 173
PROCNO 2

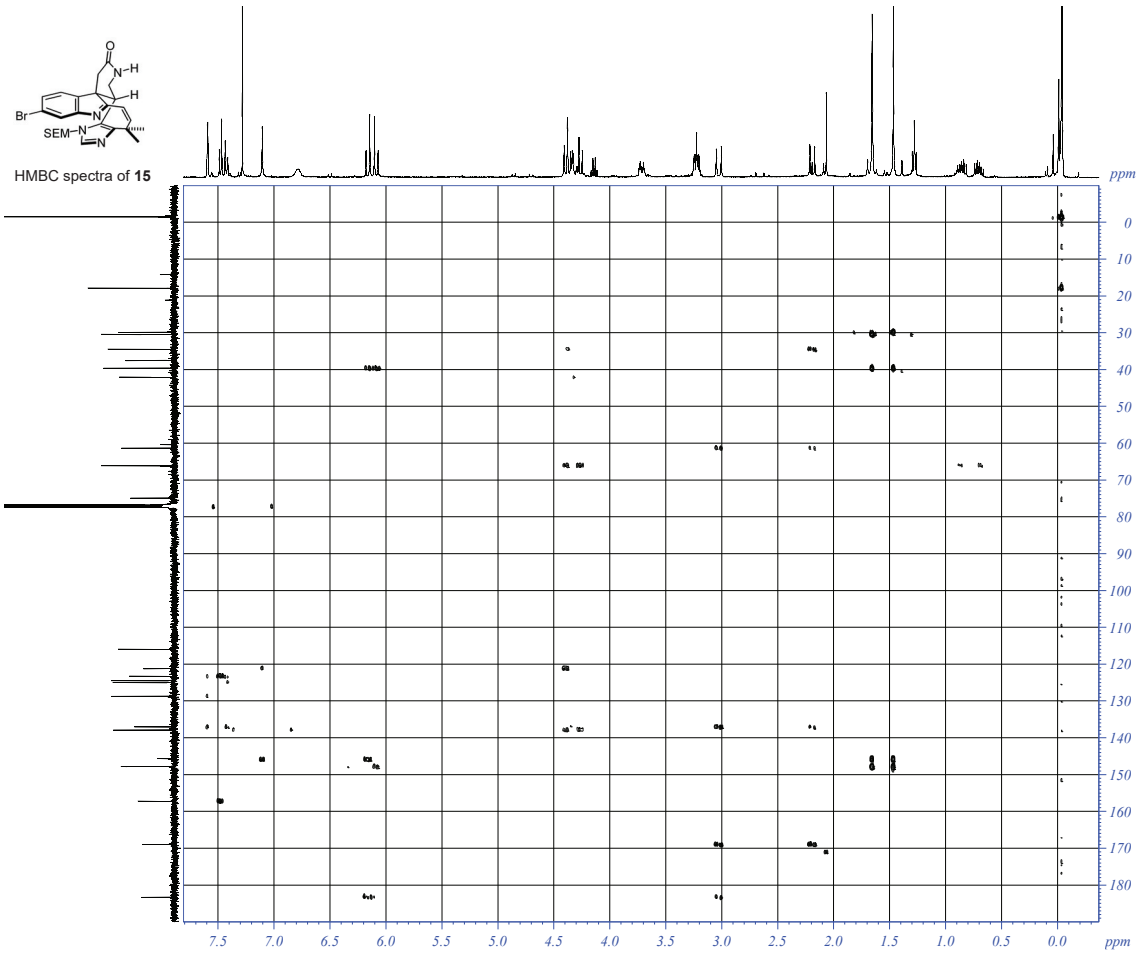
F2 - Acquisition Parameters
Date_ 20161212
Time 16.39 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4000
DS 4
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec

F2 - Processing parameters
SI 32768
SF 100.6127715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



15

^{13}C NMR (100 MHz, $CDCl_3$)



HMBC spectra of **15**

Current Data Parameters
NAME nakane-A400
EXPNO 170
PROCNO 2

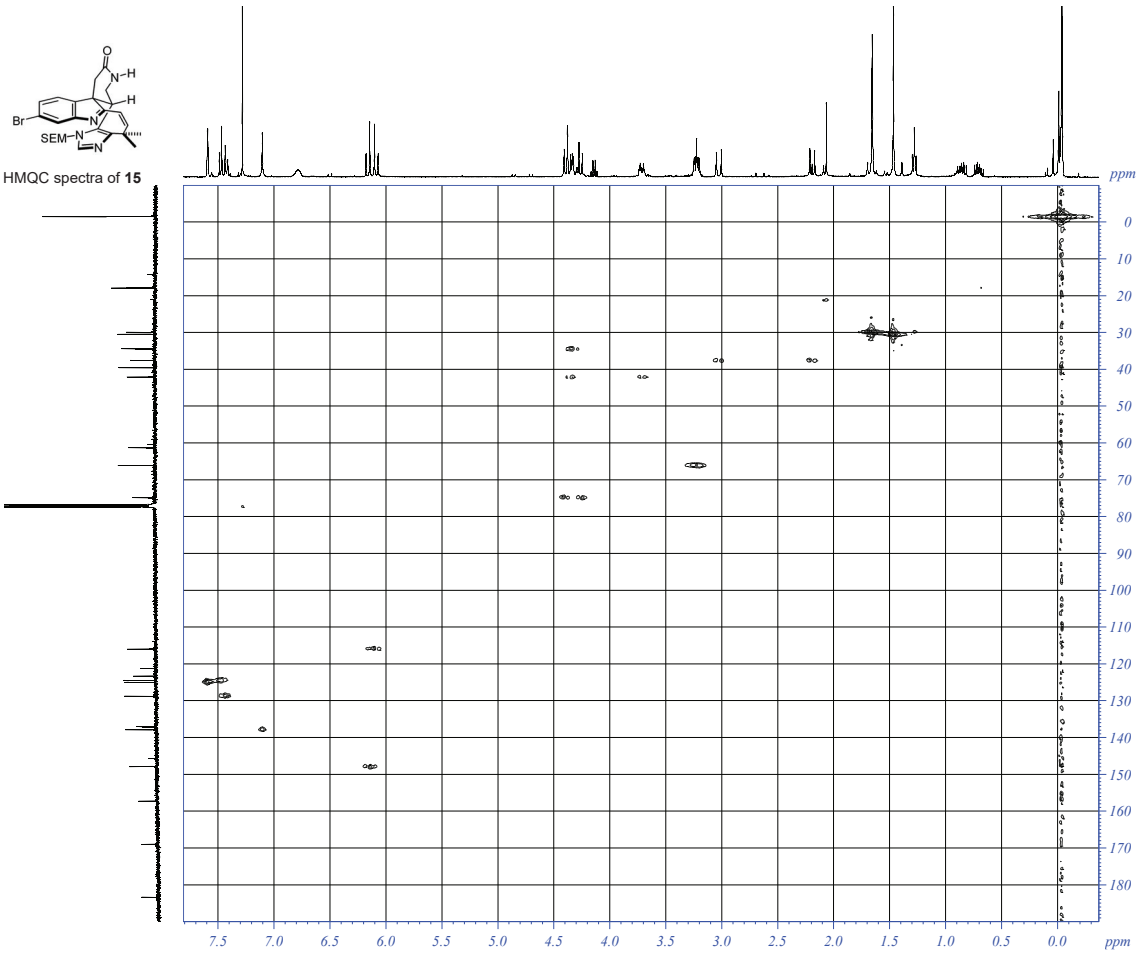
F2 - Acquisition Parameters
Date_ 20161209
Time 18.58 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG hmbcgplpndqf
TD 2048
SOLVENT CDCl3
NS 12
DS 16
SWH 3272.251 Hz
FIDRES 3.195558 Hz
AQ 0.3129344 sec
RG 16384
DW 152.800 usec
DE 6.50 usec
TE 296.7 K
CNST2 145.0000000
CNST13 10.0000000
d0 0.00000300 sec
D1 1.07711995 sec
d2 0.00344828 sec
d6 0.05000000 sec
D16 0.00010000 sec
in0 0 sec
SFO1 400.1314890 MHz
NUC1 1H
P1 15.00 usec
p2 30.00 usec
SFO2 100.6228298 MHz
NUC2 13C
P3 10.00 usec
GPNAM[1] SINE.100
GPZ1 50.00 %
GPNAM[2] SINE.100
GPZ2 30.00 %
GPNAM[3] SINE.100
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 100.6228 MHz
FIDRES 187.350113 Hz
SW 238.324 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 400.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

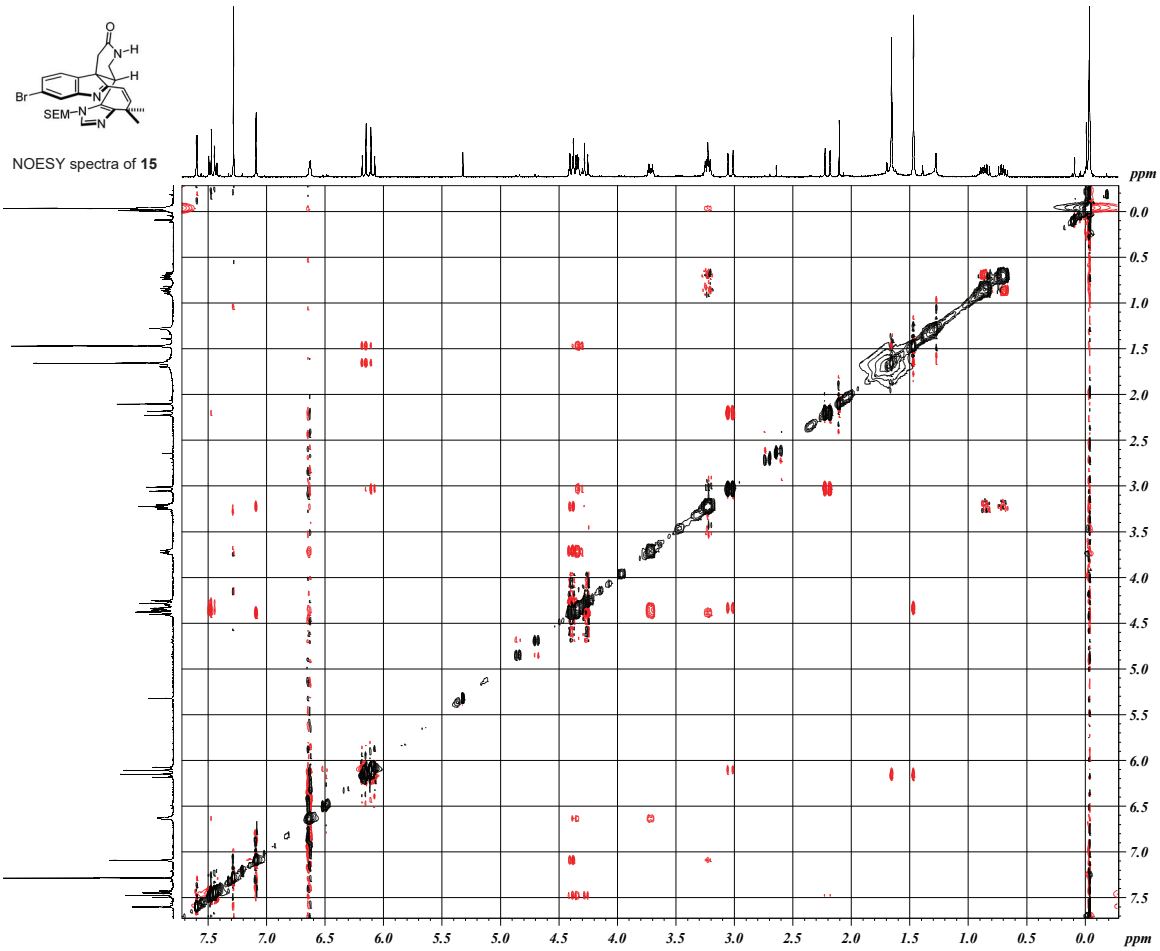
F1 - Processing parameters
SI 1024
MC2 QF
SF 100.6127690 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0

HMQC spectra of **15**

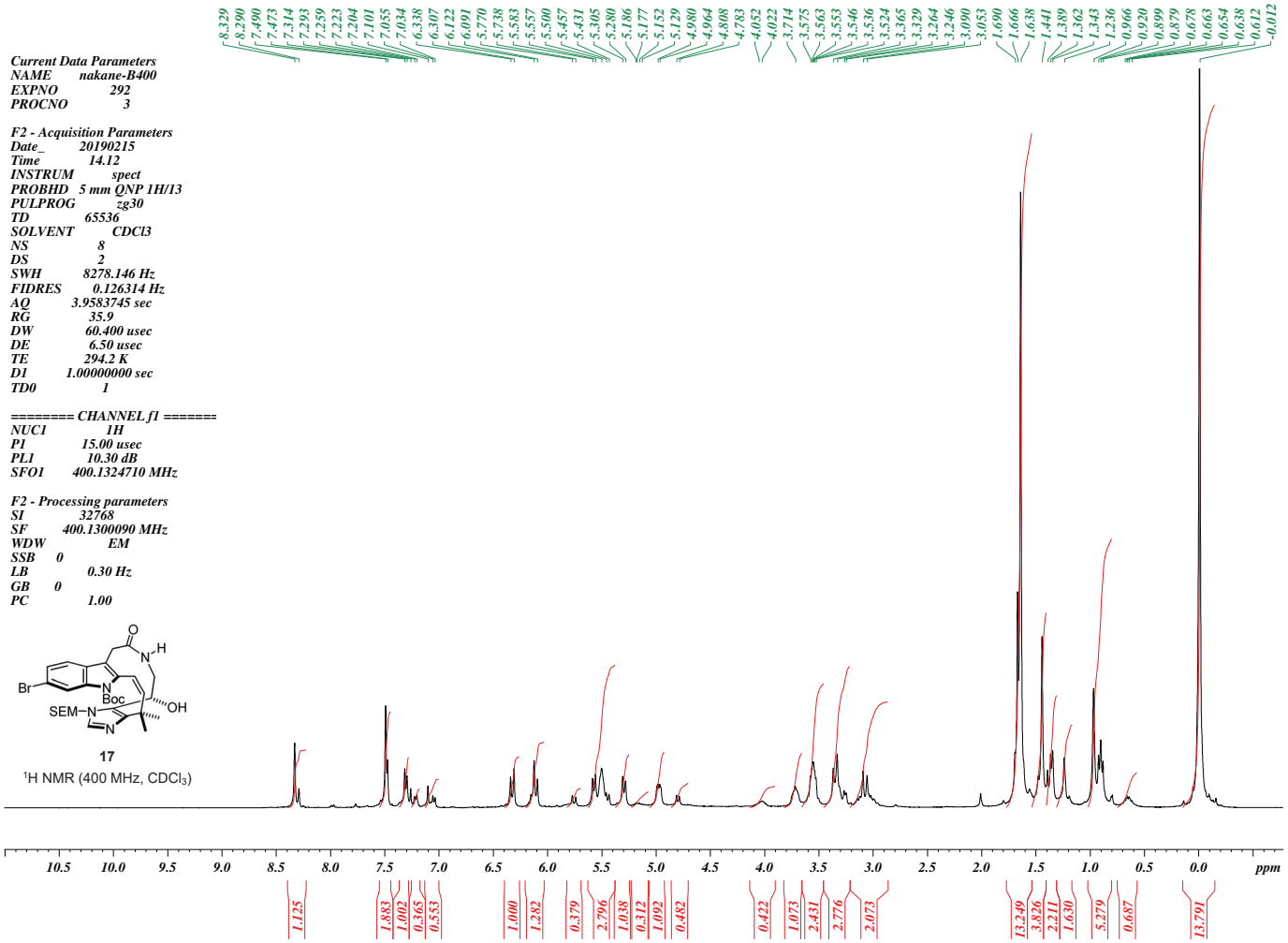


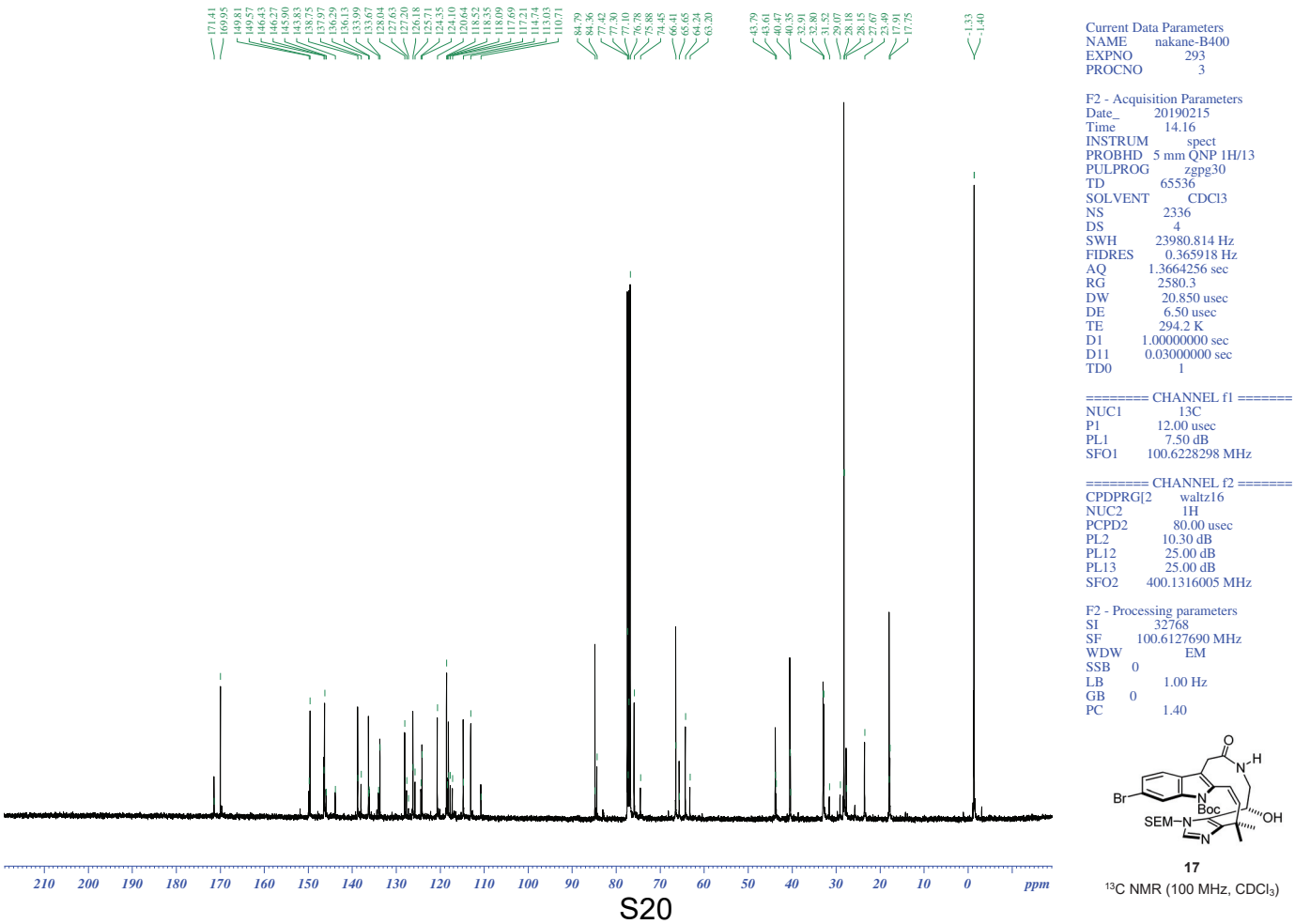
```
Current Data Parameters
NAME        nakane-A400
EXPNO           171
PROCNO            2

F2 - Acquisition Parameters
Date_        20161209
Time           20.13 h
INSTRUM          spect
PROBHD   5 mm QNP 1H/13
PULPROG       hmqcgpqf
TD              2048
SOLVENT         CDCl3
NS                 8
DS                16
SWH          3272.251 Hz
FIDRES       3.195558 Hz
AQ         0.3129344 sec
RG             26008
DW           152.800 usec
DE             6.50 usec
TE            297.9 K
CNST2       145.0000000
d0         0.00000300 sec
D1         1.50000000 sec
d2         0.00344828 sec
d12        0.00002000 sec
d13        0.00000400 sec
D16        0.00010000 sec
DELTA1     0.00232428 sec
in0     0 sec
SFO1     400.1314890 MHz
NUC1           1H
P1           15.00 usec
p2           30.00 usec
SFO2     100.6228298 MHz
NUC2           13C
CPDPRG[2        garp
P3           10.00 usec
PCPD2        70.00 usec
GPNAM[1]     SINE.100
GPZ1         50.00 %
GPNAM[2]     SINE.100
GPZ2         30.00 %
GPNAM[3]     SINE.100
GPZ3         40.10 %
P16        1000.00 usec

F1 - Acquisition parameters
TD             256
SFO1       100.6228 MHz
FIDRES     187.350113 Hz
SW         238.324 ppm
FnMODE           QF

F2 - Processing parameters
SI            1024
SF       400.1300000 MHz
WDW           QSINE
SSB               2
LB      0 Hz
GB      0
PC            1.40

F1 - Processing parameters
SI            1024
MC2             QF
SF       100.6127690 MHz
WDW           QSINE
SSB               2
LB      0 Hz
GB      0
```

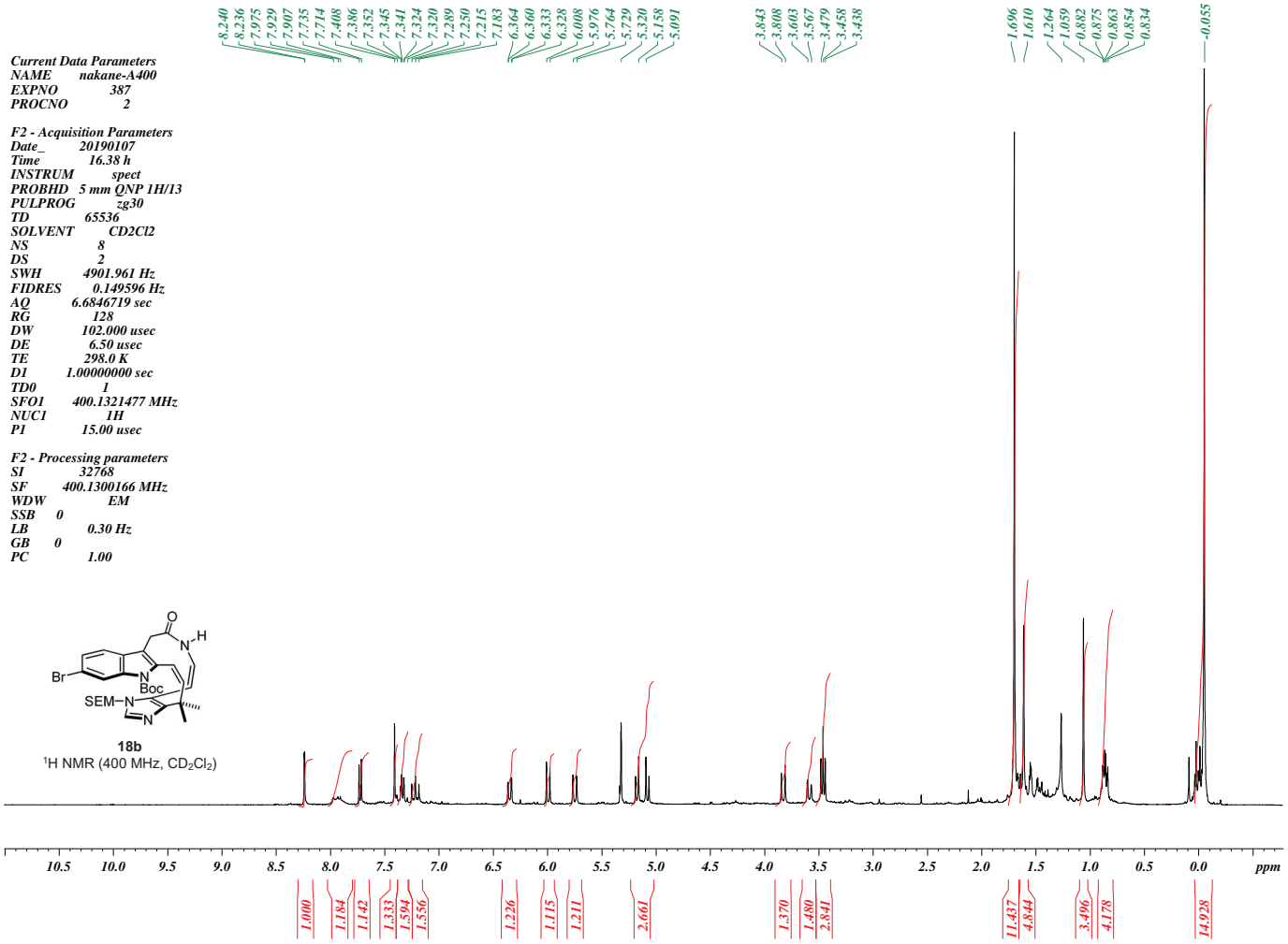

NOESY spectra of **15**

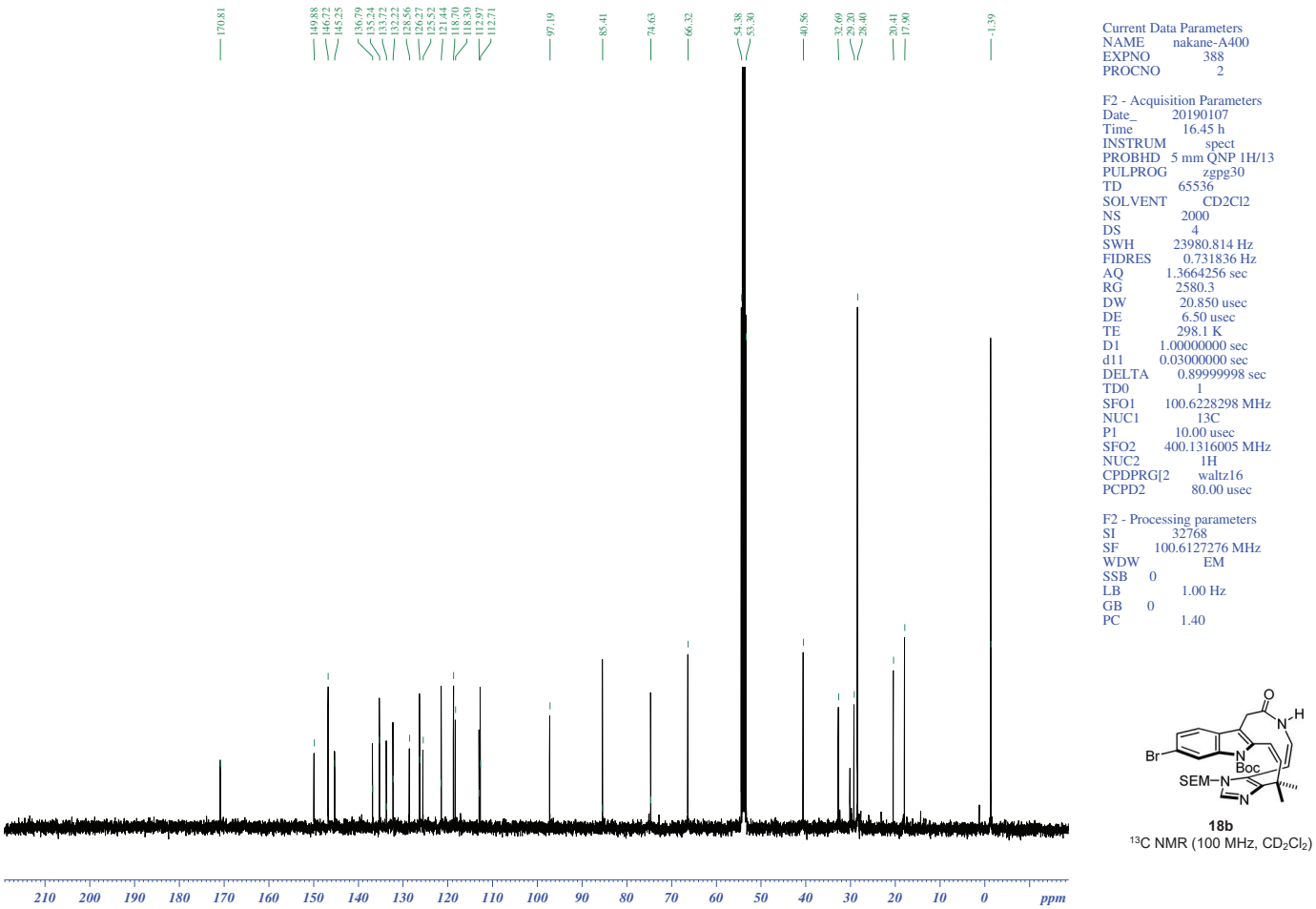


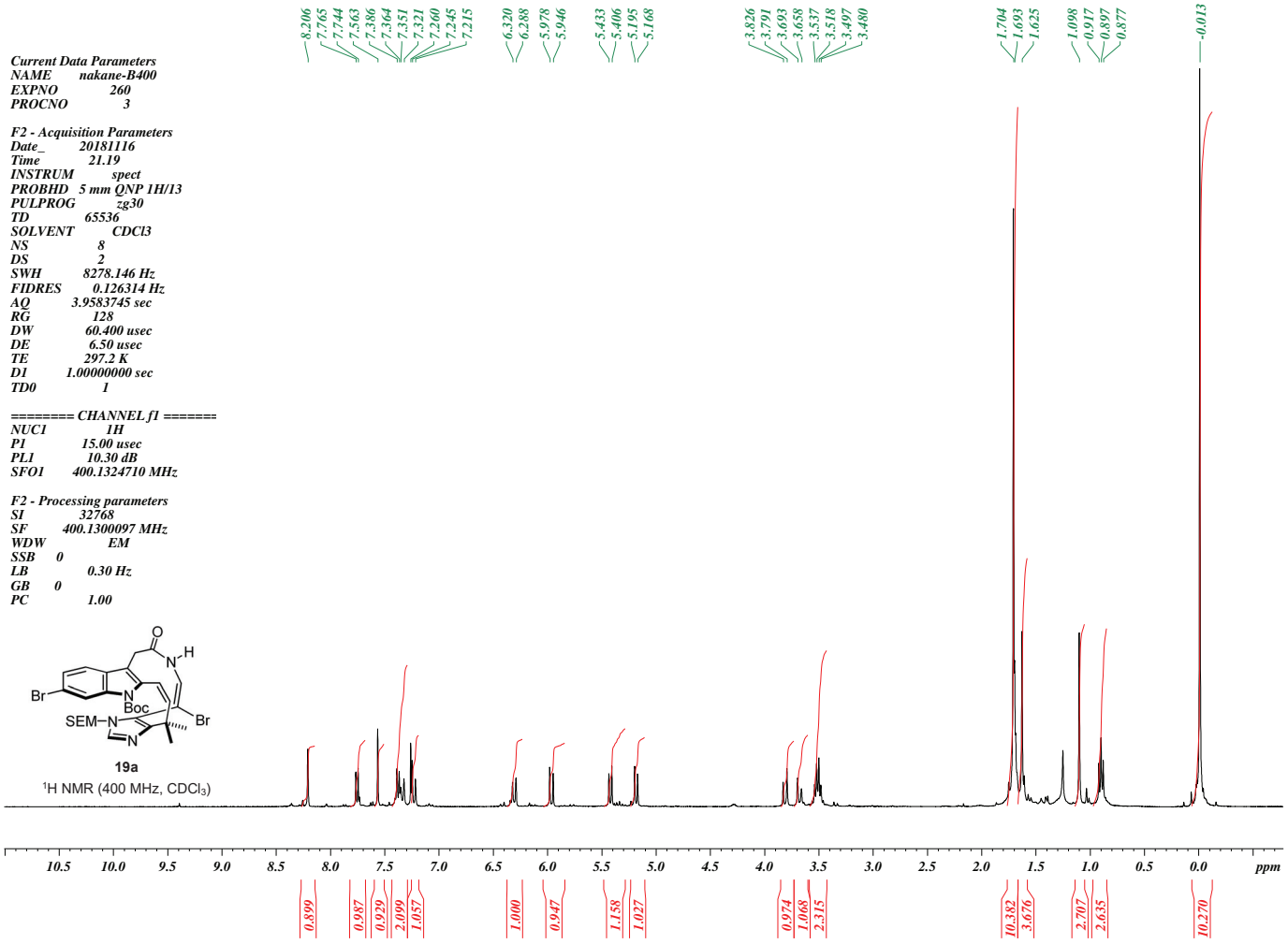
```
Current Data Parameters
NAME        nakazaki-A400
EXPNO                 4
PROCNO                2

F2 - Acquisition Parameters
Date_          20200203
Time             21.45 h
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG        noesyph
TD                2048
SOLVENT          CDCl3
NS                  64
DS                  16
SWH            3205.128 Hz
FIDRES         3.130008 Hz
AQ           0.3194880 sec
RG               645.1
DW             156.000 usec
DE               6.50 usec
TE              296.9 K
d0          -0.00001910 sec
D1           1.50000000 sec
D8           0.60000002 sec
in0       0 sec
ST1CNT                0
SFO1       400.1314886 MHz
NUC1               1H
P1             15.00 usec

F1 - Acquisition parameters
TD                  256
SFO1          400.1315 MHz
FIDRES       25.040020 Hz
SW             8.010 ppm
FnMODE      States-TPPI

F2 - Processing parameters
SI                 1024
SF         400.1300000 MHz
WDW              QSINE
SSB                   2
LB        0 Hz
GB        0
PC                 1.00

F1 - Processing parameters
SI                 1024
MC2         States-TPPI
SF         400.1300000 MHz
WDW              QSINE
SSB                   2
LB        0 Hz
GB        0
```

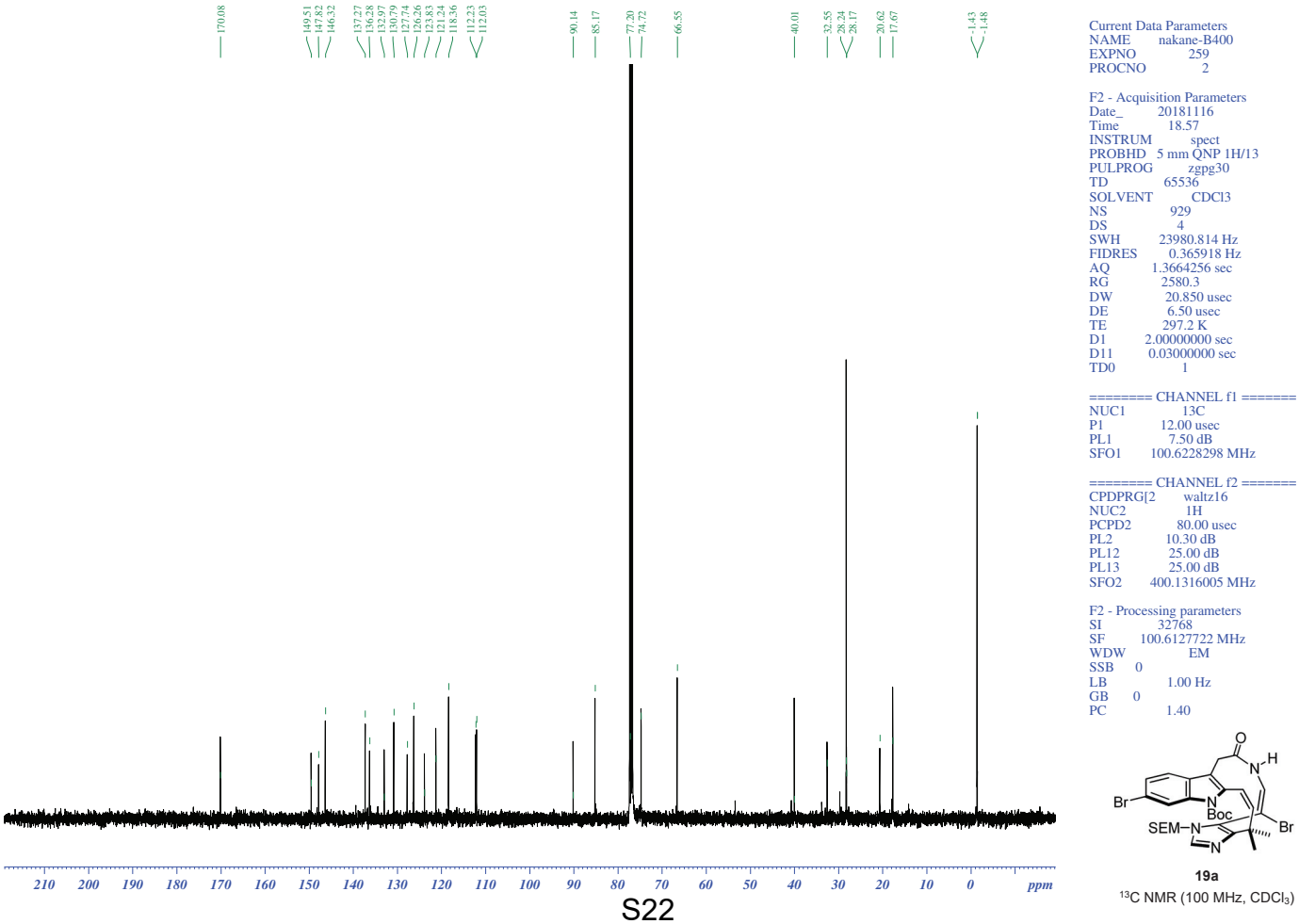

Current Data Parameters
NAME nakane-B400
EXPNO 292
PROCNO 3
F2 - Acquisition Parameters
Date_ 20190215
Time 14.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 35.9
DW 60.400 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 1H
P1 15.00 usec
PL1 10.30 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
17
1H NMR (400 MHz, CDCl3)
ppm

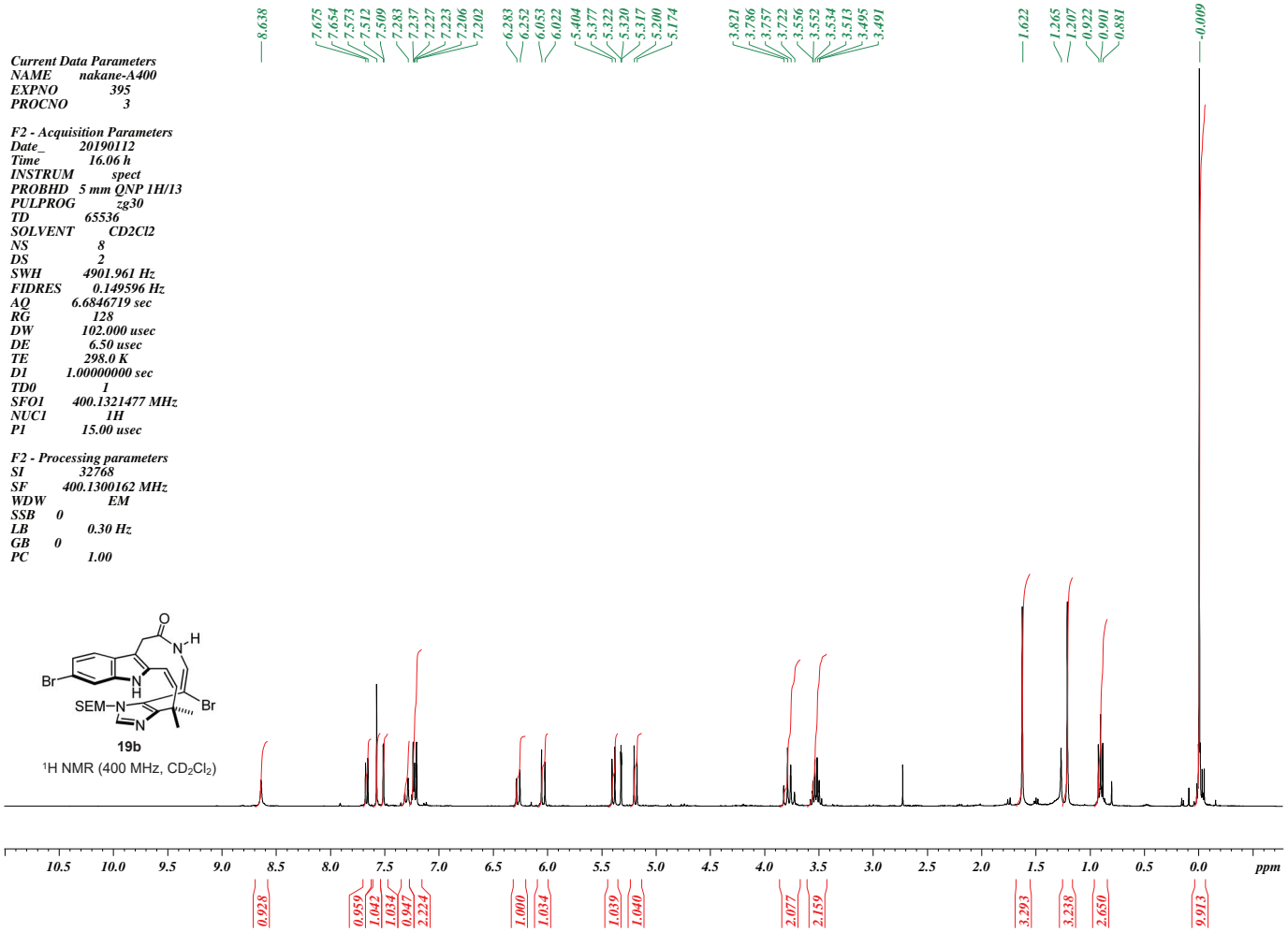

Current Data Parameters
NAME nakane-B400
EXPNO 293
PROCNO 3
F2 - Acquisition Parameters
Date_ 20190215
Time 14.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2336
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 12.00 usec
PL1 7.50 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 =======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 10.30 dB
PL12 25.00 dB
PL13 25.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
17
13C NMR (100 MHz, CDCl3)
ppm

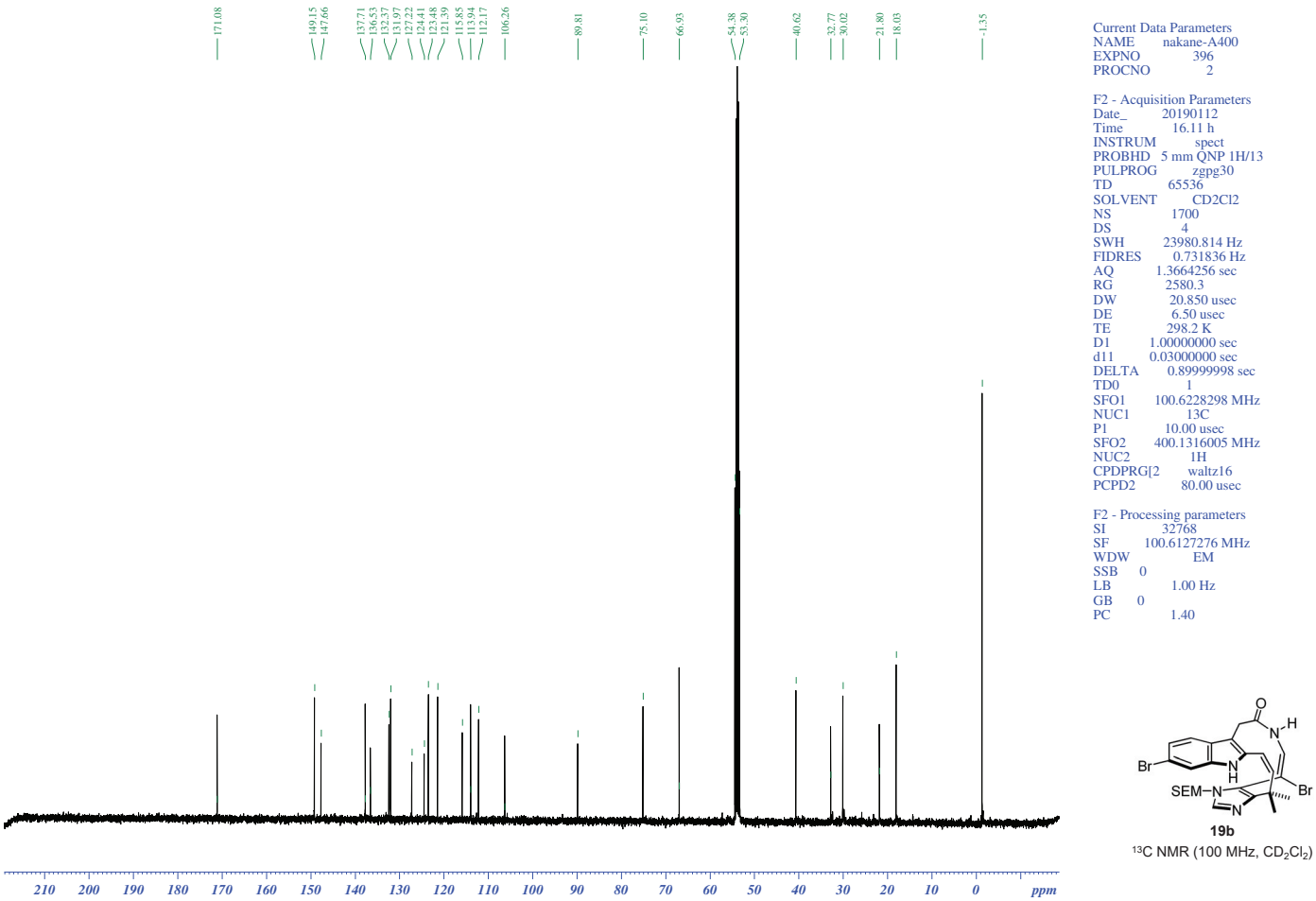

Current Data Parameters
NAME nakane-A400
EXPNO 387
PROCNO 2
F2 - Acquisition Parameters
Date_ 20190107
Time 16.38 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 8
DS 2
SWH 4901.961 Hz
FIDRES 0.149596 Hz
AQ 6.6846719 sec
RG 128
DW 102.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321477 MHz
NUC1 1H
P1 15.00 usec
F2 - Processing parameters
SI 32768
SF 400.1300166 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
18b
1H NMR (400 MHz, CD2Cl2)
ppm

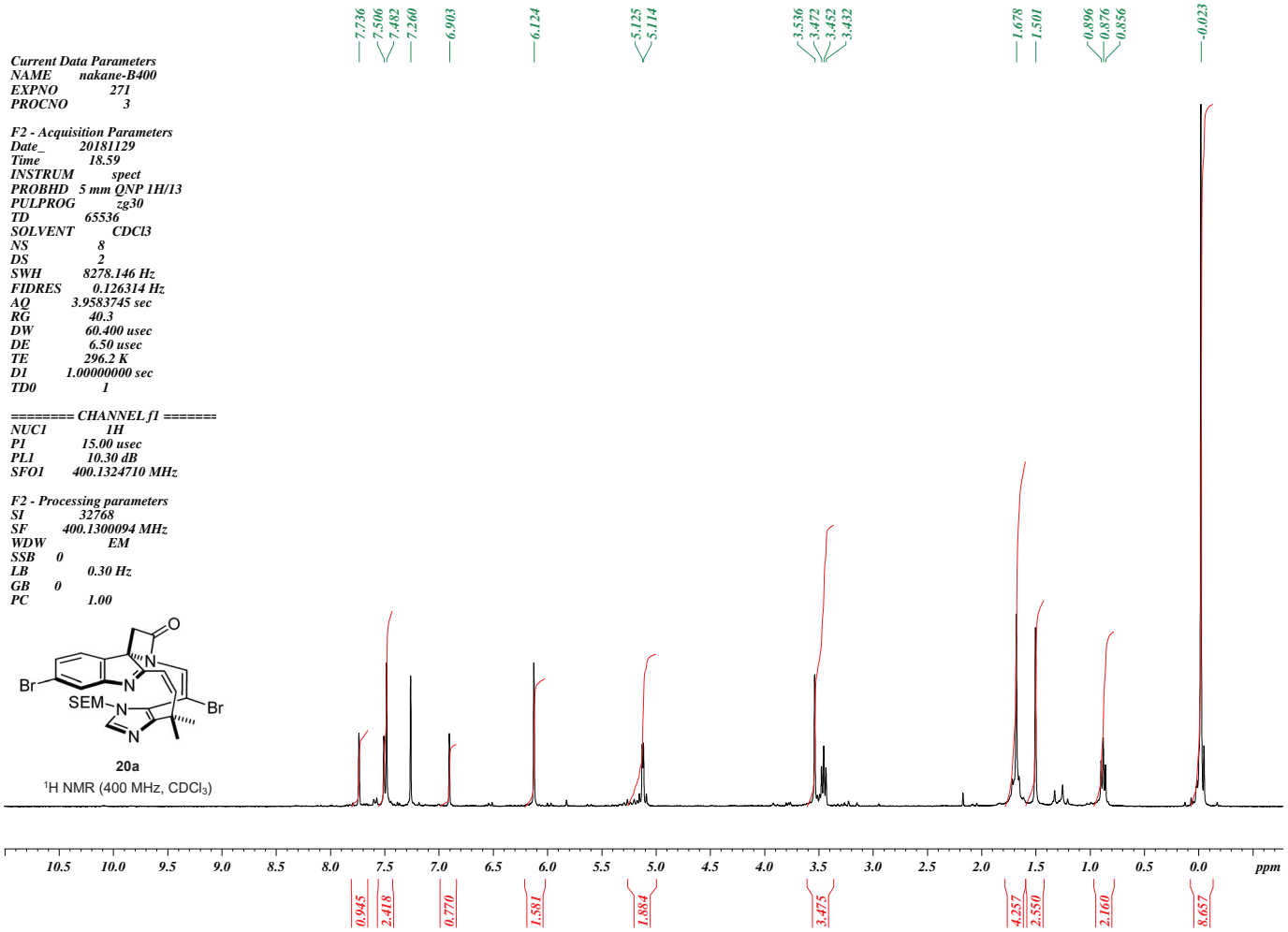

Current Data Parameters
NAME nakane-A400
EXPNO 388
PROCNO 2
F2 - Acquisition Parameters
Date_ 20190107
Time 16.45 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 2000
DS 4
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
F2 - Processing parameters
SI 32768
SF 100.6127276 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
18b
13C NMR (100 MHz, CD2Cl2)
ppm

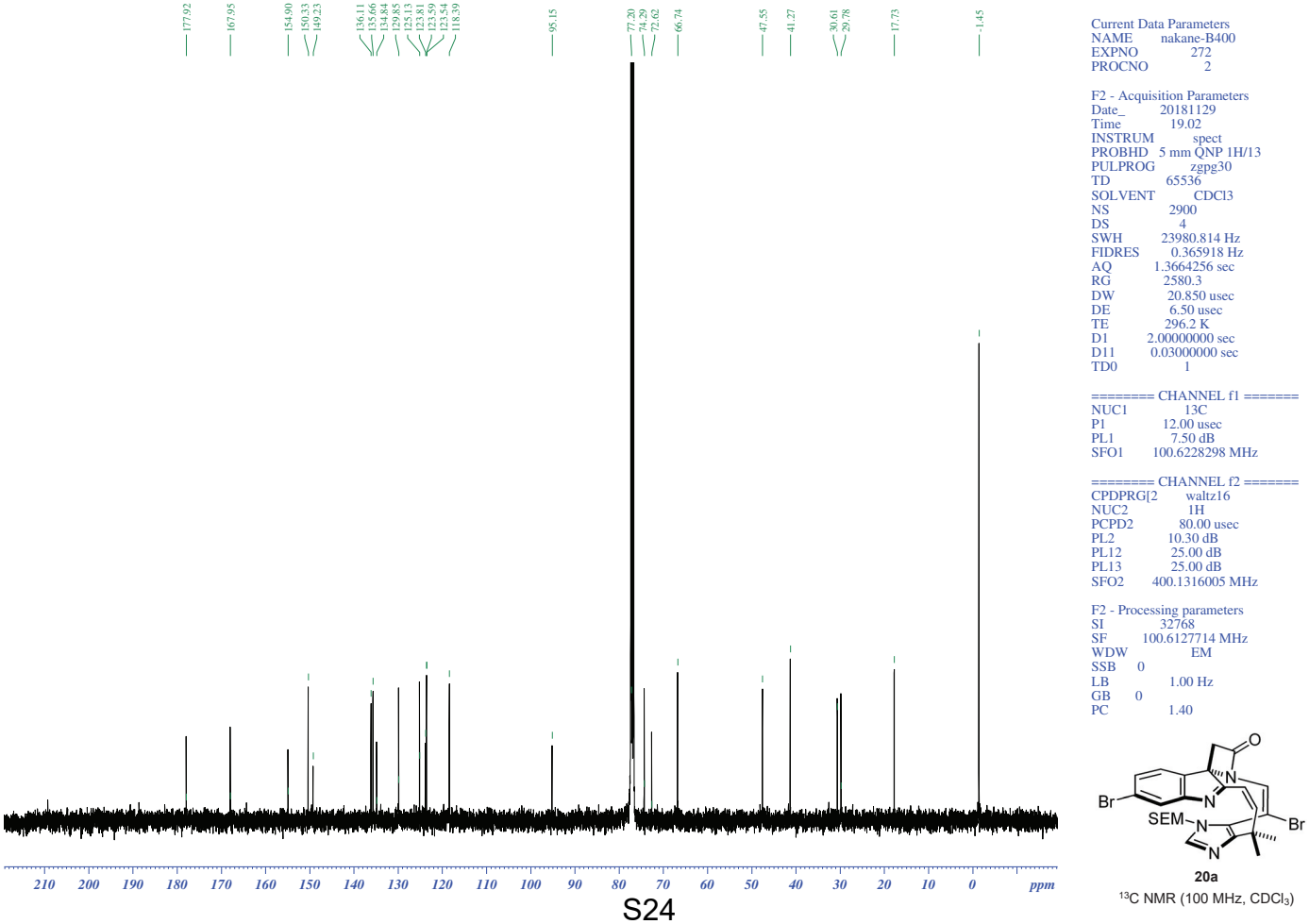

Current Data Parameters
NAME nakane-B400
EXPNO 260
PROCNO 3
F2 - Acquisition Parameters
Date_ 20181116
Time 21.19
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 128
DW 60.400 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 1H
P1 15.00 usec
PL1 10.30 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
19a
1H NMR (400 MHz, CDCl3)
ppm


Current Data Parameters
NAME nakane-B400
EXPNO 259
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181116
Time 18.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 929
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 12.00 usec
PL1 7.50 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 =======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 10.30 dB
PL12 25.00 dB
PL13 25.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19a
13C NMR (100 MHz, CDCl3)
ppm

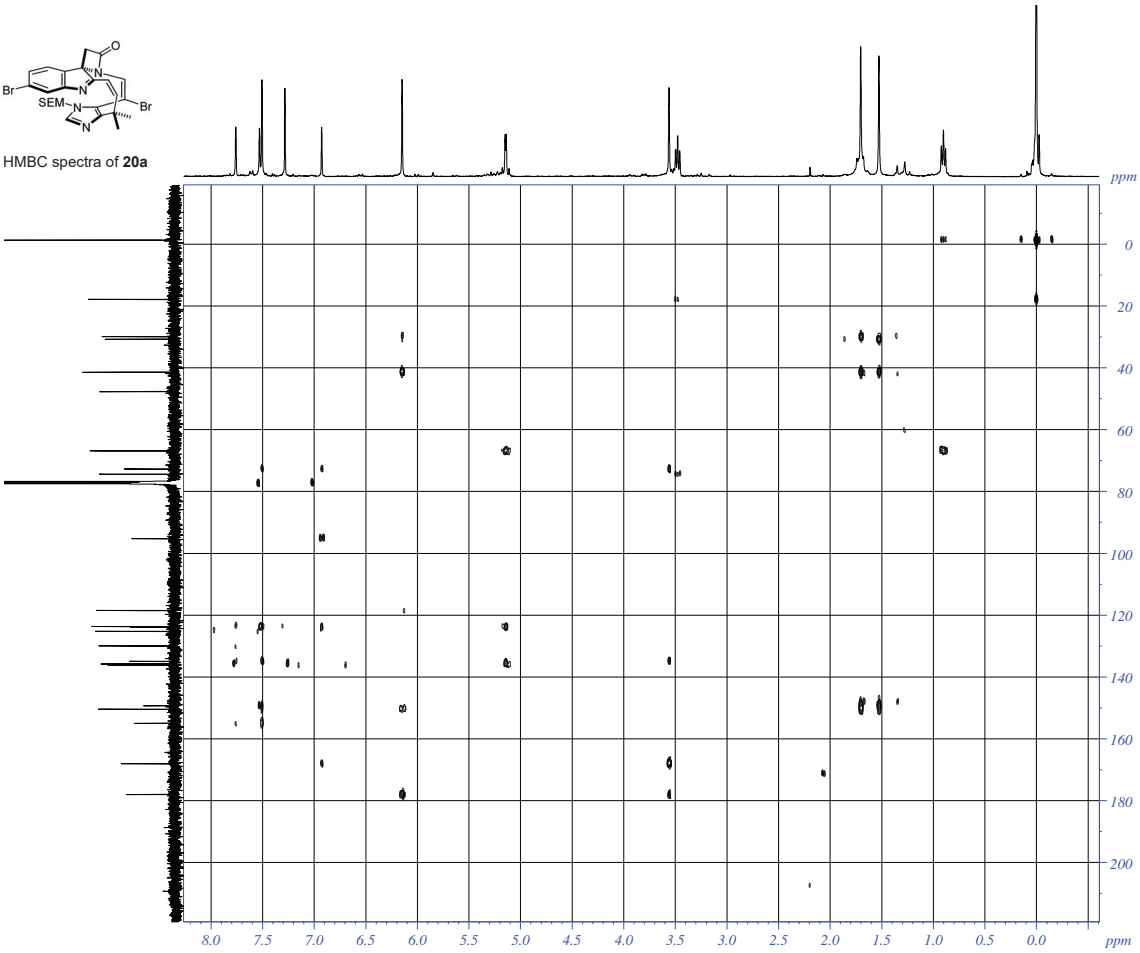

Current Data Parameters
NAME nakane-A400
EXPNO 395
PROCNO 3
F2 - Acquisition Parameters
Date_ 20190112
Time 16.06 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 8
DS 2
SWH 4901.961 Hz
FIDRES 0.149596 Hz
AQ 6.6846719 sec
RG 128
DW 102.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321477 MHz
NUC1 1H
P1 15.00 usec
F2 - Processing parameters
SI 32768
SF 400.1300162 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
19b
1H NMR (400 MHz, CD2Cl2)
ppm


Current Data Parameters
NAME nakane-A400
EXPNO 396
PROCNO 2
F2 - Acquisition Parameters
Date_ 20190112
Time 16.11 h
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 1700
DS 4
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
F2 - Processing parameters
SI 32768
SF 100.6127276 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
19b
13C NMR (100 MHz, CD2Cl2)
ppm


7.736
7.506
7.482
7.260
6.903
6.124
5.125
5.114
3.536
3.472
3.452
3.432
1.678
1.501
0.896
0.876
0.856
-0.023
Current Data Parameters
NAME nakane-B400
EXPNO 271
PROCNO 3
F2 - Acquisition Parameters
Date_ 20181129
Time 18.59
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 40.3
DW 60.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 1H
P1 15.00 usec
PL1 10.30 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
SEM
N
O
Br
20a
1H NMR (400 MHz, CDCl3)
10.5 10.0 9.5 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 ppm
0.945
2.418
0.770
1.581
1.884
3.475
4.257
2.550
2.160
8.657


177.92
167.95
154.90
150.33
149.23
136.11
135.66
134.84
129.85
125.13
123.81
123.59
123.54
118.39
95.15
77.20
74.29
72.62
66.74
47.55
41.27
30.61
29.78
17.73
-1.45
Current Data Parameters
NAME nakane-B400
EXPNO 272
PROCNO 2
F2 - Acquisition Parameters
Date_ 20181129
Time 19.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2900
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 12.00 usec
PL1 7.50 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 =======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 10.30 dB
PL12 25.00 dB
PL13 25.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Br
SEM
N
O
Br
20a
13C NMR (100 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

HMBC spectra of **20a**



Current Data Parameters
NAME nakane-B400
EXPNO 250
PROCNO 2

F2 - Acquisition Parameters
Date_ 20180804
Time 16.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG hmbcgplpndqf
TD 2048
SOLVENT CDCl3
NS 32
DS 16
SWH 3551.136 Hz
FIDRES 1.733953 Hz
AQ 0.2883584 sec
RG 16384
DW 140.800 usec
DE 6.50 usec
TE 299.2 K
CNST2 145.0000000
CNST13 10.0000000
D0 0.00000300 sec
D1 1.07711995 sec
D2 0.00344828 sec
D6 0.05000000 sec
D16 0.00020000 sec
IN0 0.00002085 sec

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
P2 30.00 usec
PL1 10.30 dB
SFO1 400.1315322 MHz

======== CHANNEL f2 ========
NUC2 13C
P3 12.00 usec
PL2 7.50 dB
SFO2 100.6228298 MHz

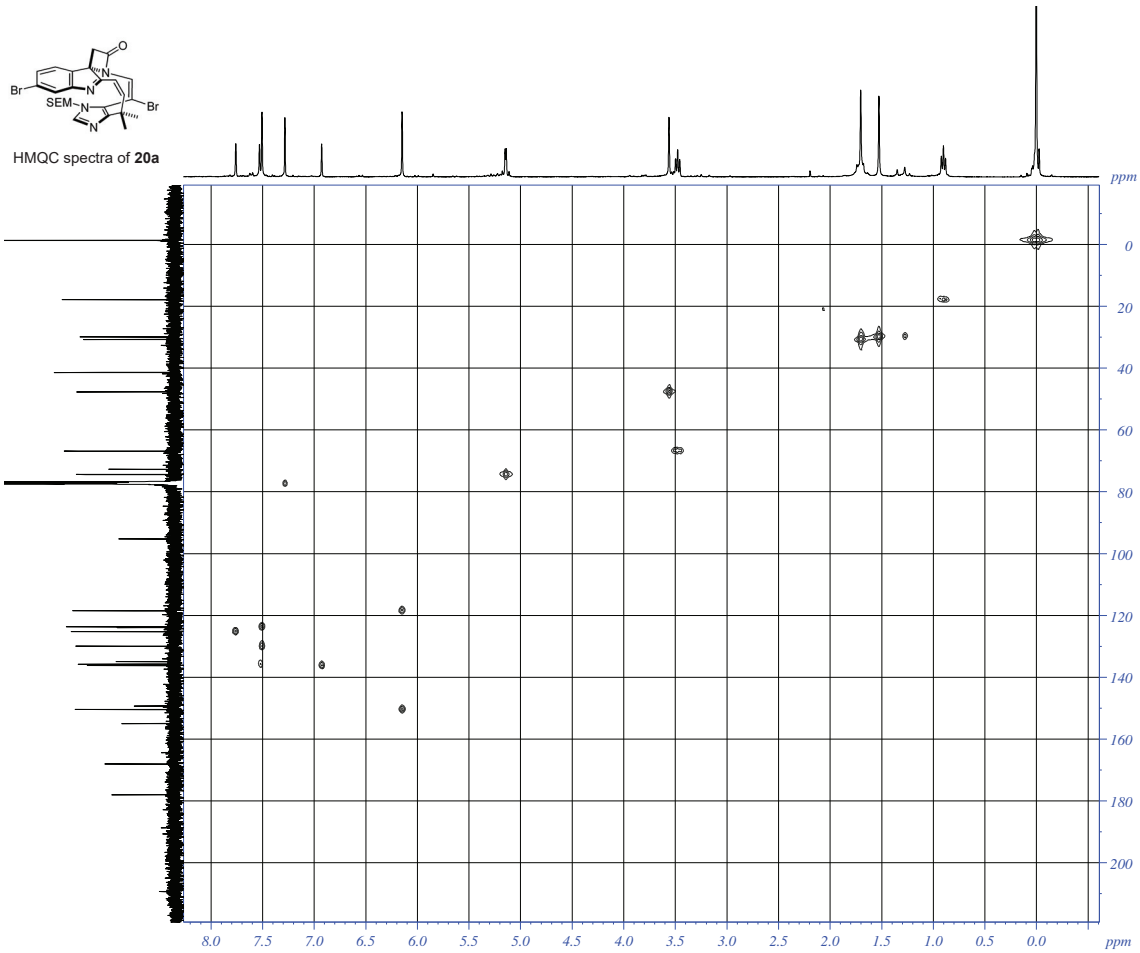
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 100.6228 MHz
FIDRES 374.700226 Hz
SW 238.324 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 400.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 100.6127690 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0

HMQC spectra of **20a**



Current Data Parameters
NAME nakane-B400
EXPNO 251
PROCNO 2

F2 - Acquisition Parameters
Date_ 20180804
Time 17.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG hmqcgpqf
TD 1024
SOLVENT CDCl3
NS 16
DS 16
SWH 3551.136 Hz
FIDRES 3.467907 Hz
AQ 0.1441792 sec
RG 26008
DW 140.800 usec
DE 6.50 usec
TE 298.2 K
CNST2 145.0000000
D0 0.00000300 sec
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00002085 sec

======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
P2 30.00 usec
PL1 10.30 dB
SFO1 400.1315322 MHz

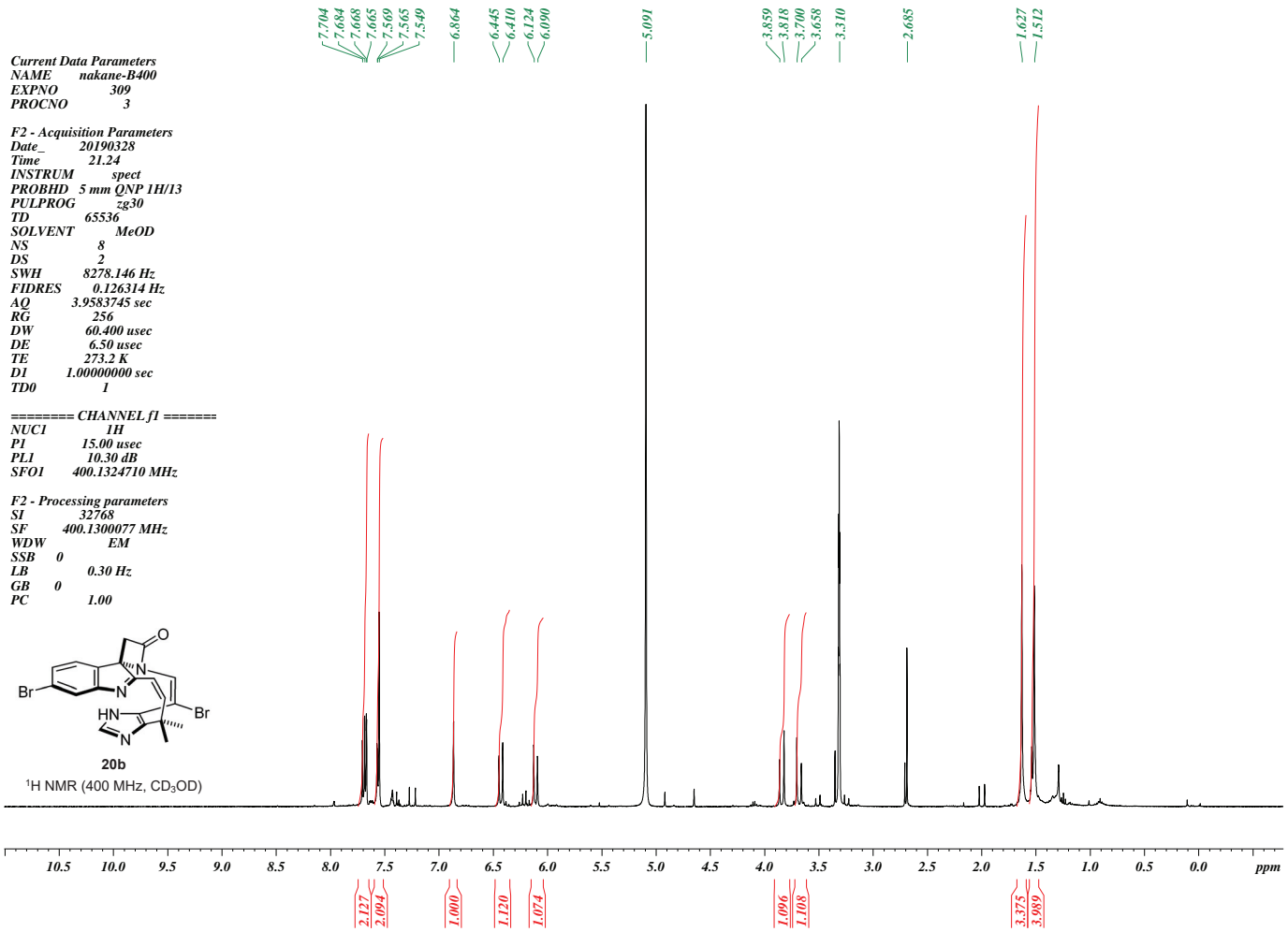
======== CHANNEL f2 ========
CPDPRG[2 garp
NUC2 13C
P3 12.00 usec
PCPD2 65.00 usec
PL2 7.50 dB
PL12 22.17 dB
SFO2 100.6228298 MHz

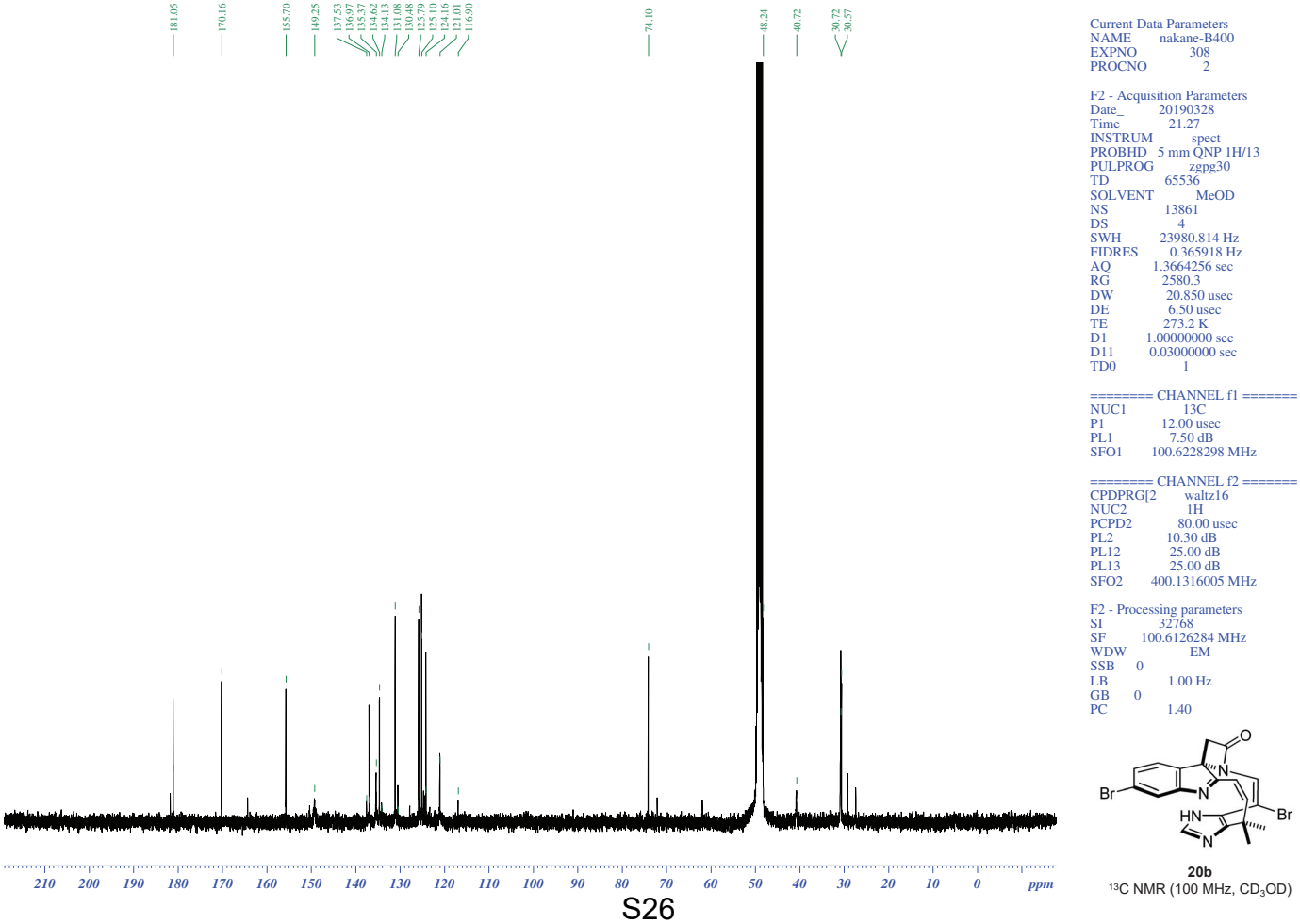
====== GRADIENT CHANNEL =====
GPNAM[1] SINE.100
GPNAM[2] SINE.100
GPNAM[3] SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

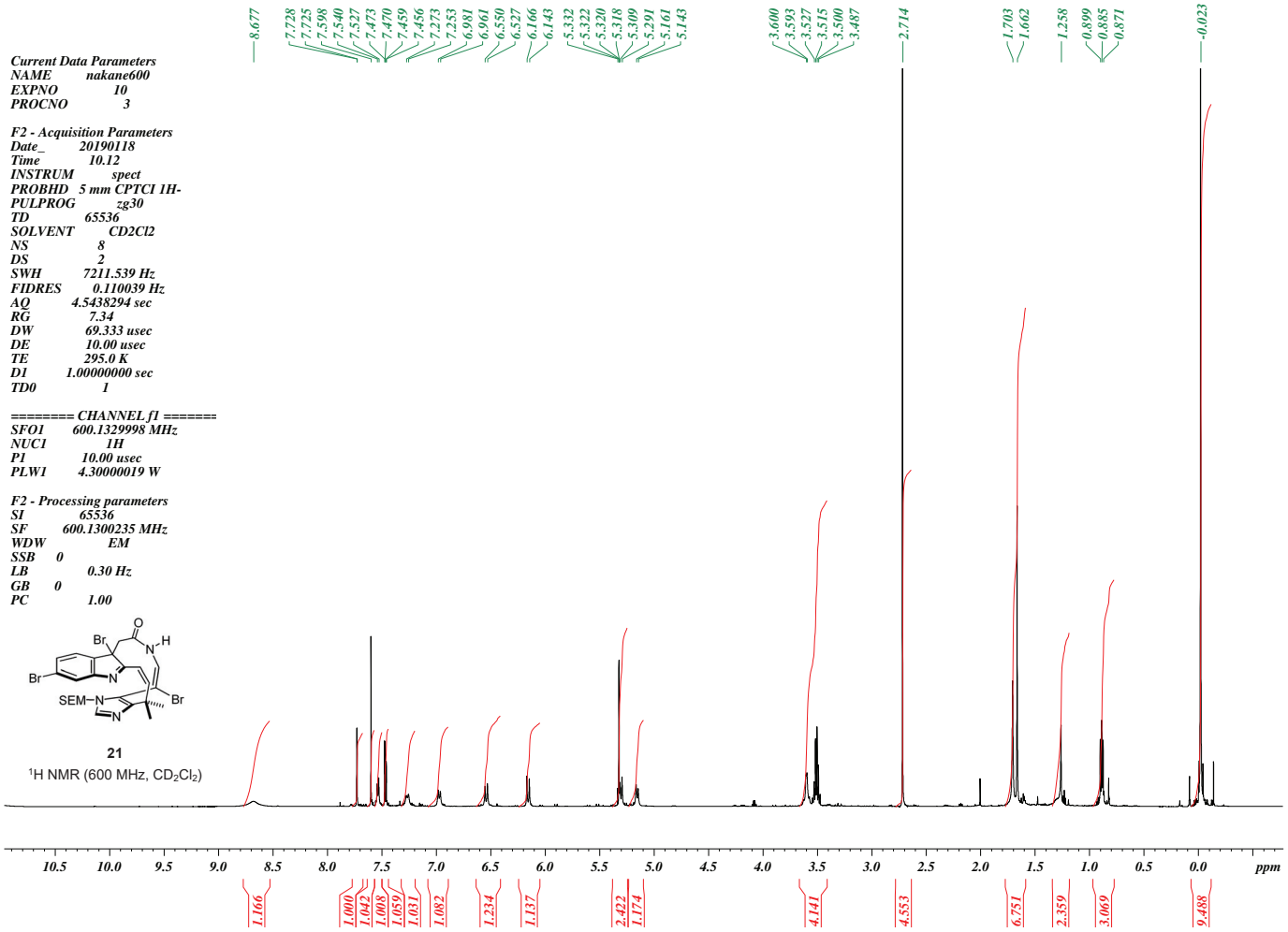
F1 - Acquisition parameters
TD 128
SFO1 100.6228 MHz
FIDRES 374.700226 Hz
SW 238.324 ppm
FnMODE QF

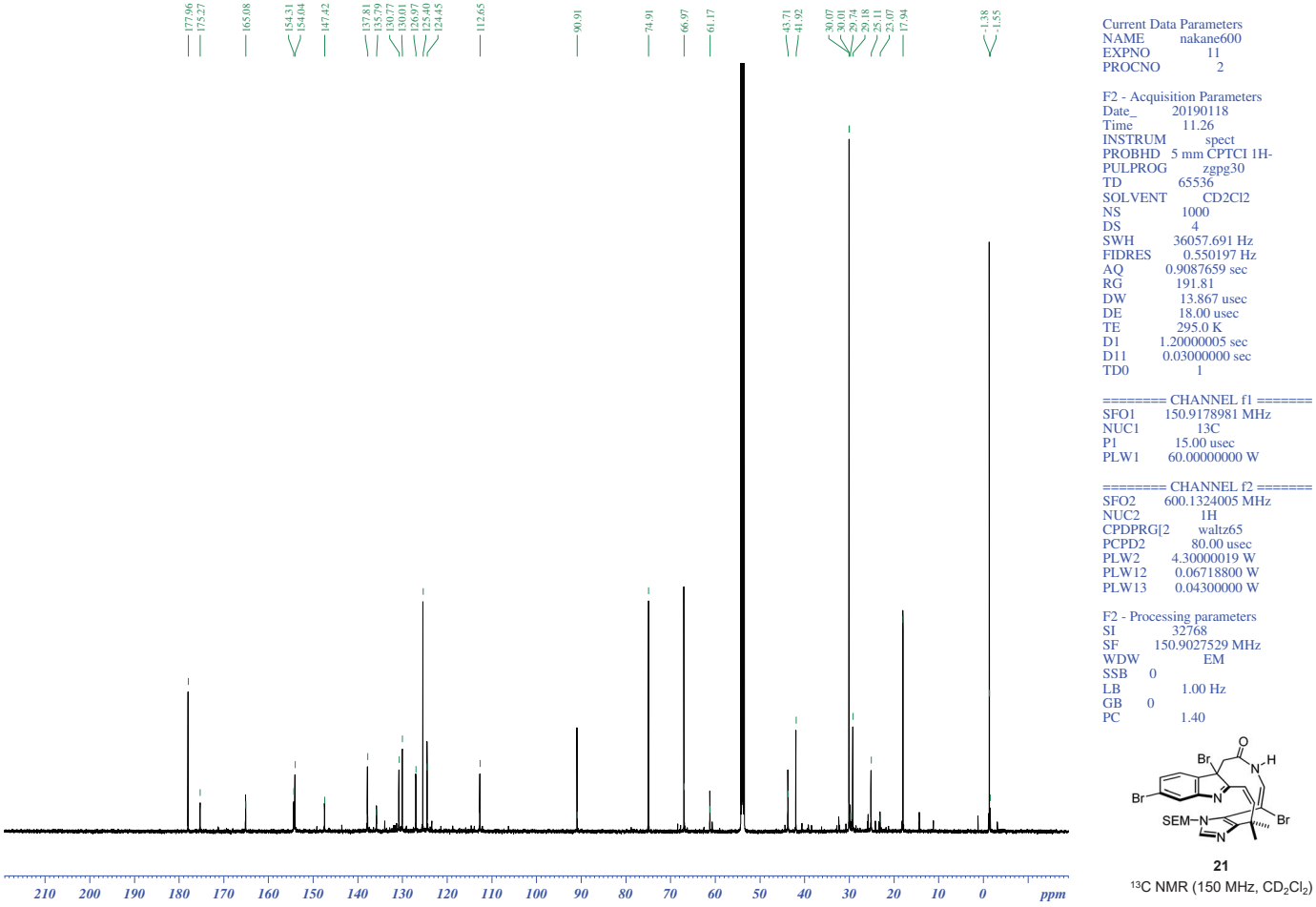
F2 - Processing parameters
SI 1024
SF 400.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 100.6127690 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

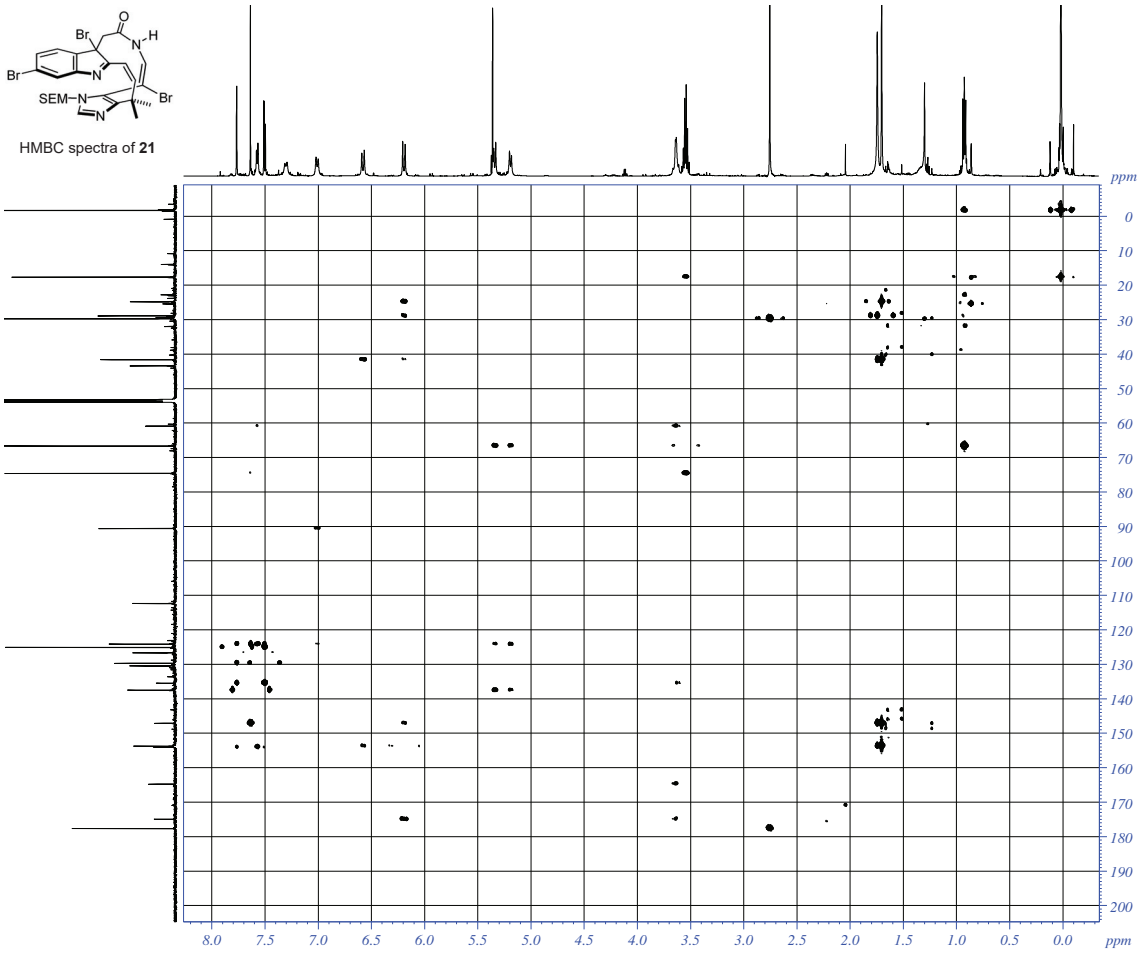

7.704
7.684
7.668
7.665
7.569
7.565
7.549
6.864
6.445
6.410
6.124
6.090
5.091
3.859
3.818
3.700
3.658
3.310
2.685
1.627
1.512
Current Data Parameters
NAME nakane-B400
EXPNO 309
PROCNO 3
F2 - Acquisition Parameters
Date_ 20190328
Time 21.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 273.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 1H
P1 15.00 usec
PL1 10.30 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
Br
HN
N
O
20b
1H NMR (400 MHz, CD3OD)
10.5 10.0 9.5 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 ppm
2.127
2.094
1.000
1.120
1.074
1.096
1.108
3.375
3.989


181.05
170.16
155.70
149.25
137.53
136.97
135.37
134.62
134.13
131.08
130.48
125.79
125.10
124.16
121.01
116.90
74.10
48.24
40.72
30.72
30.57
Current Data Parameters
NAME nakane-B400
EXPNO 308
PROCNO 2
F2 - Acquisition Parameters
Date_ 20190328
Time 21.27
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 13861
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 2580.3
DW 20.850 usec
DE 6.50 usec
TE 273.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 12.00 usec
PL1 7.50 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 =======
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 10.30 dB
PL12 25.00 dB
PL13 25.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6126284 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Br
Br
HN
N
O
20b
13C NMR (100 MHz, CD3OD)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Current Data Parameters
NAME nakane600
EXPNO 10
PROCNO 3
F2 - Acquisition Parameters
Date_ 20190118
Time 10.12
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 8
DS 2
SWH 7211.539 Hz
FIDRES 0.110039 Hz
AQ 4.5438294 sec
RG 7.34
DW 69.333 usec
DE 10.00 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
SFO1 600.1329998 MHz
NUC1 1H
P1 10.00 usec
PLW1 4.30000019 W
F2 - Processing parameters
SI 65536
SF 600.1300235 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
21
1H NMR (600 MHz, CD2Cl2)
ppm


Current Data Parameters
NAME nakane600
EXPNO 11
PROCNO 2
F2 - Acquisition Parameters
Date_ 20190118
Time 11.26
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 1000
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 191.81
DW 13.867 usec
DE 18.00 usec
TE 295.0 K
D1 1.20000005 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
SFO1 150.9178981 MHz
NUC1 13C
P1 15.00 usec
PLW1 60.00000000 W
======== CHANNEL f2 =======
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 80.00 usec
PLW2 4.30000019 W
PLW12 0.06718800 W
PLW13 0.04300000 W
F2 - Processing parameters
SI 32768
SF 150.9027529 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21
13C NMR (150 MHz, CD2Cl2)
ppm

HMBC spectra of **21**



Current Data Parameters
NAME nakane600
EXPNO 13
PROCNO 2

F2 - Acquisition Parameters
Date_ 20190118
Time 10.20
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hmbcgplpndqf
TD 2048
SOLVENT CD2Cl2
NS 16
DS 16
SWH 5720.824 Hz
FIDRES 2.793371 Hz
AQ 0.1789952 sec
RG 191.81
DW 87.400 usec
DE 15.00 usec
TE 295.0 K
CNST2 145.0000000
CNST13 10.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D2 0.00344828 sec
D6 0.05000000 sec
D16 0.00010000 sec
IN0 0.00001550 sec

======== CHANNEL f1 ========
SFO1 600.1326553 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
PLW1 4.30000019 W

======== CHANNEL f2 ========
SFO2 150.9175677 MHz
NUC2 13C
P3 15.00 usec
PLW2 60.00000000 W

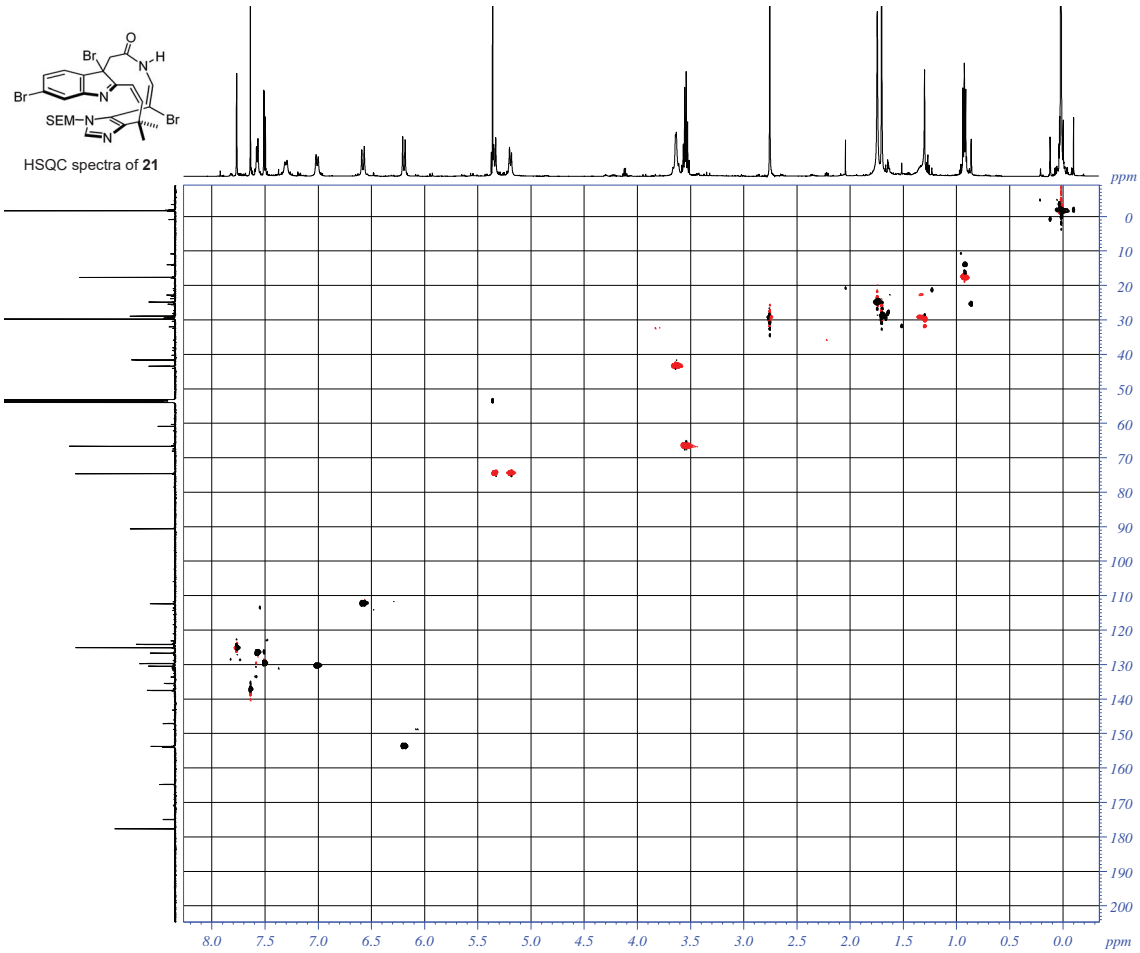
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPNAM[3] SMSQ10.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 64
SFO1 150.9176 MHz
FIDRES 1008.064514 Hz
SW 213.746 ppm
FnMODE QF

F2 - Processing parameters
SI 1024
SF 600.1300000 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 150.9028090 MHz
WDW SINE
SSB 0
LB 0 Hz
GB 0

HSQC spectra of **21**



Current Data Parameters
NAME nakane600
EXPNO 12
PROCNO 2

F2 - Acquisition Parameters
Date_ 20190118
Time 11.28
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hsqcedetgpsp.3
TD 2048
SOLVENT CD2Cl2
NS 16
DS 32
SWH 5720.824 Hz
FIDRES 2.793371 Hz
AQ 0.1789952 sec
RG 191.81
DW 87.400 usec
DE 15.00 usec
TE 295.0 K
CNST2 145.0000000
D0 0.00000300 sec
D1 1.50000000 sec
D4 0.00172414 sec
D11 0.03000000 sec
D16 0.00010000 sec
D21 0.00345000 sec
IN0 0.00001550 sec

======== CHANNEL f1 ========
SFO1 600.1326553 MHz
NUC1 1H
P1 10.00 usec
P2 20.00 usec
P28 0 usec
PLW1 4.30000019 W

======== CHANNEL f2 ========
SFO2 150.9175677 MHz
NUC2 13C
CPDPRG[2 bi_p5m4sp_4sp.2
P3 15.00 usec
P14 500.00 usec
P31 1730.00 usec
P63 1500.00 usec
PLW0 0 W
PLW2 60.00000000 W
PLW12 2.40000010 W
SPNAM[3] Crp60,0.5,20.1
SPOAL3 0.500
SPOFFS3 0 Hz
SPW3 20.62599945 W
SPNAM[14 Crp42,1.5,20.2
SPOAL14 0.500
SPOFFS14 0 Hz
SPW14 11.55099964 W
SPNAM[18 Crp60_xfil.2
SPOAL18 0.500
SPOFFS18 0 Hz
SPW18 5.96140003 W
SPNAM[31 Crp42,1.5,20.2
SPOAL31 0.500
SPOFFS31 0 Hz
SPW31 2.88770008 W

====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 64
SFO1 150.9176 MHz
FIDRES 1008.064514 Hz
SW 213.746 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 600.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 150.9028090 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0