Supplementary Information

Design and synthesis of iso-*allo*-DNJ and L-isoDALDP derivatives: pursuit of potent and selective inhibitors of α-glucosidase

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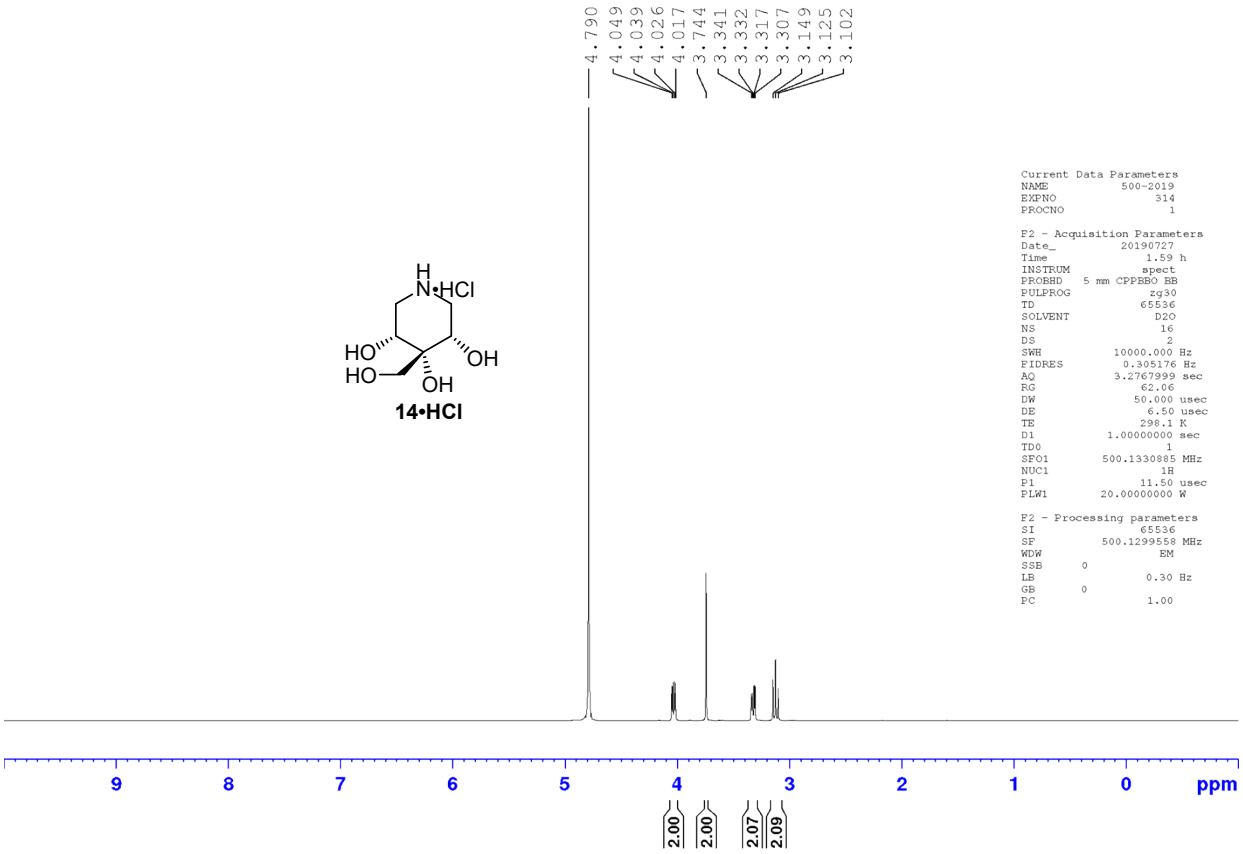
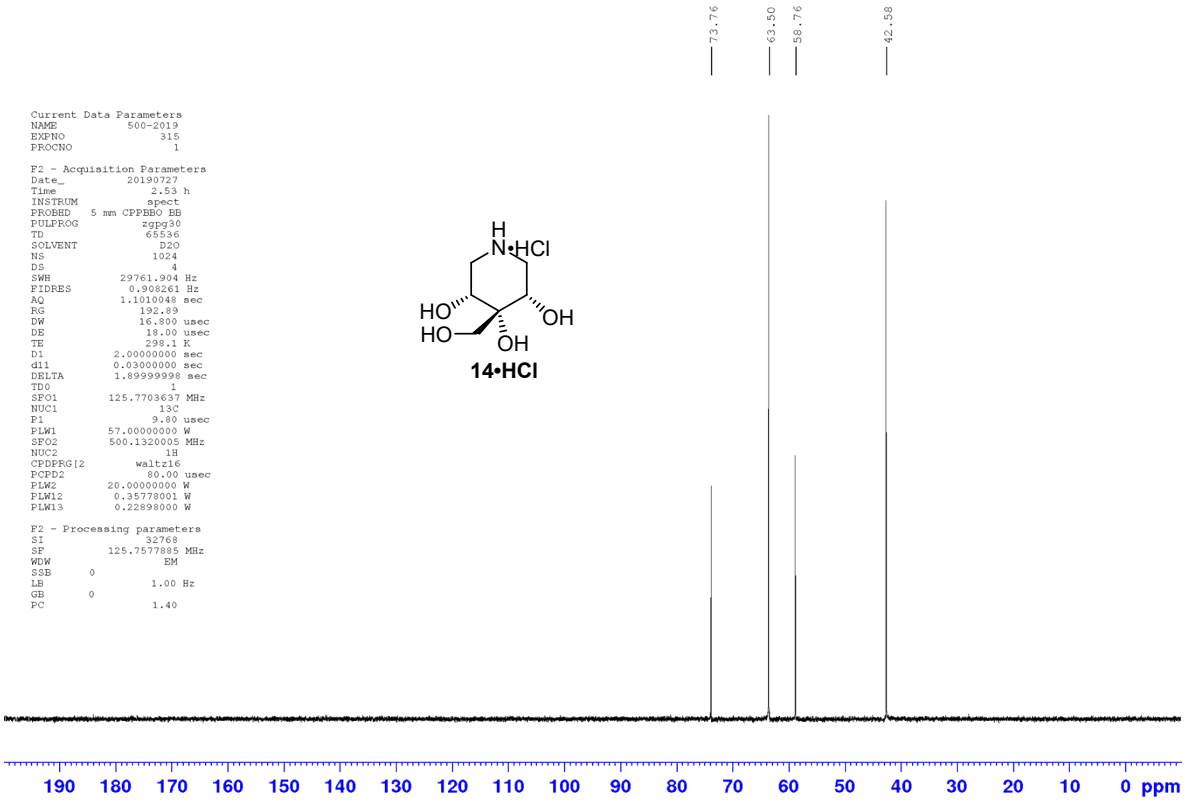
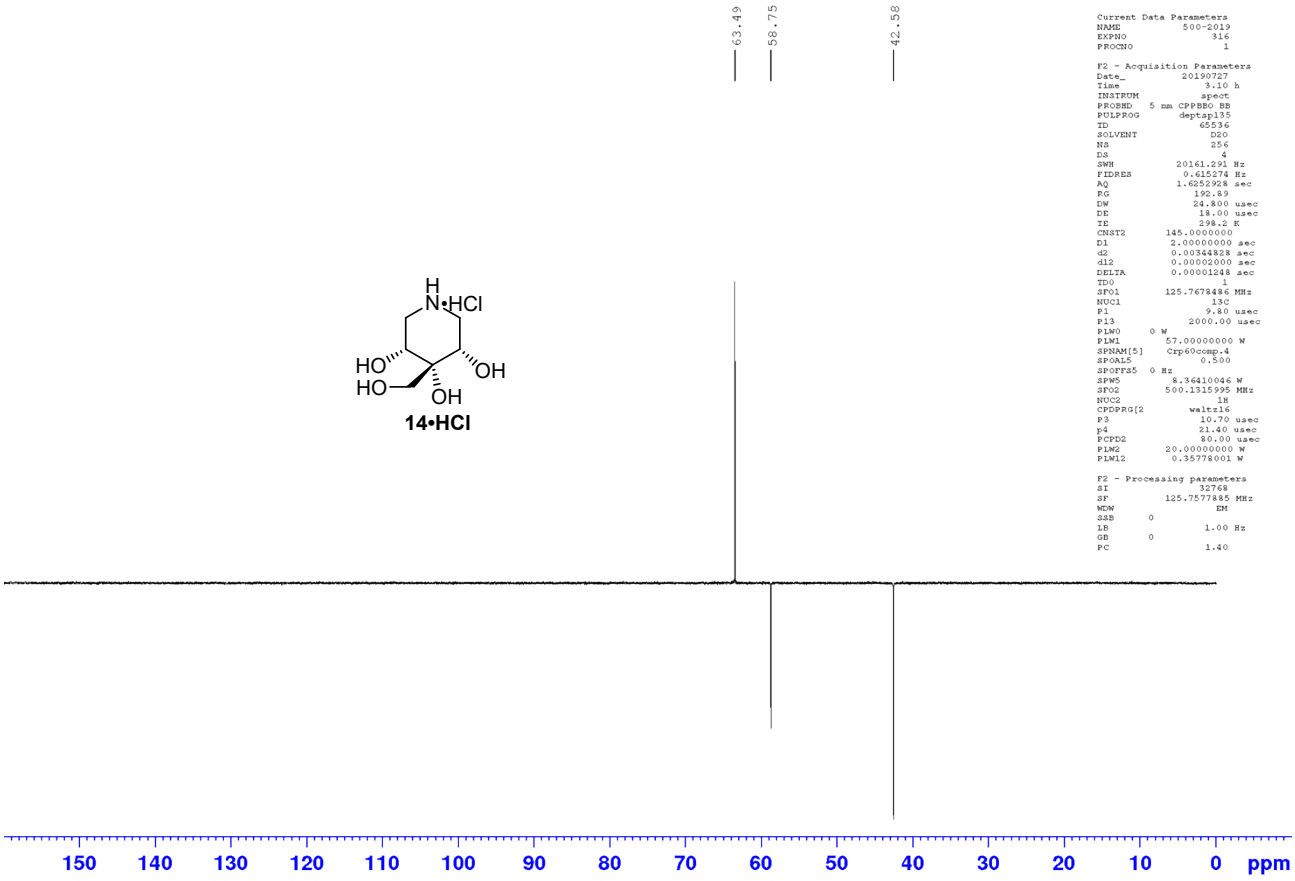
*Section I: NMR spectra and Infrared spectra for compound **14-40***

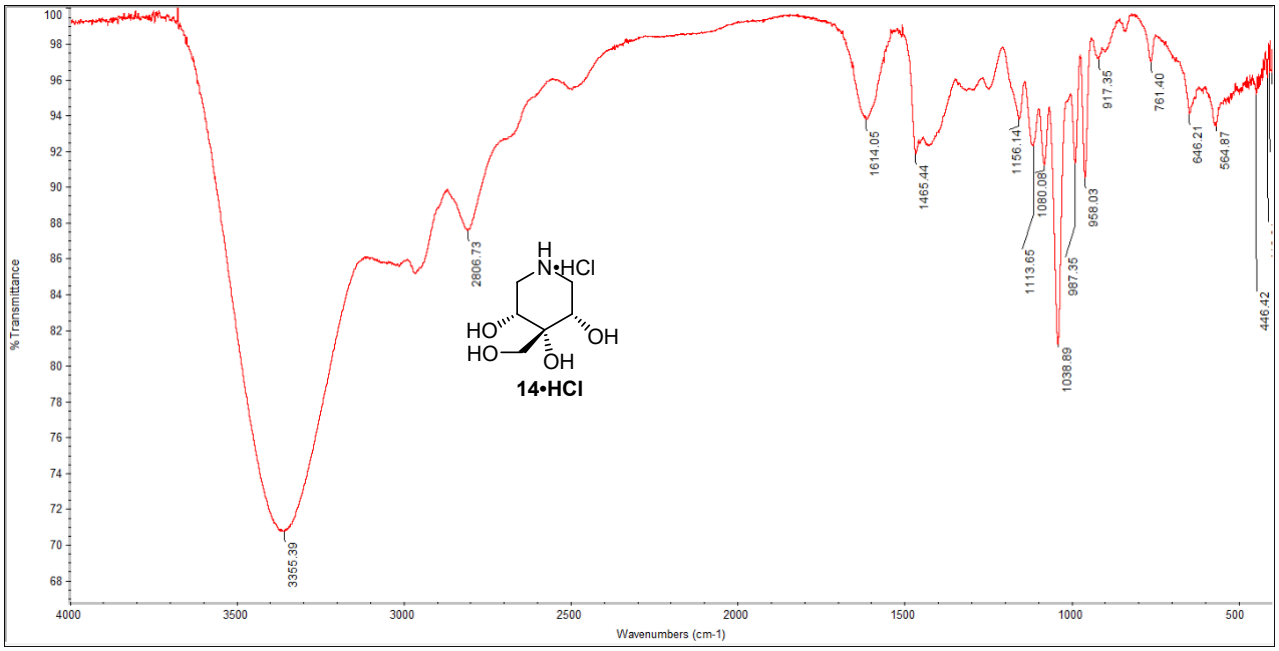
*Section I: NMR spectra and Infrared spectra for compound **14-40***

Compound 14**:**

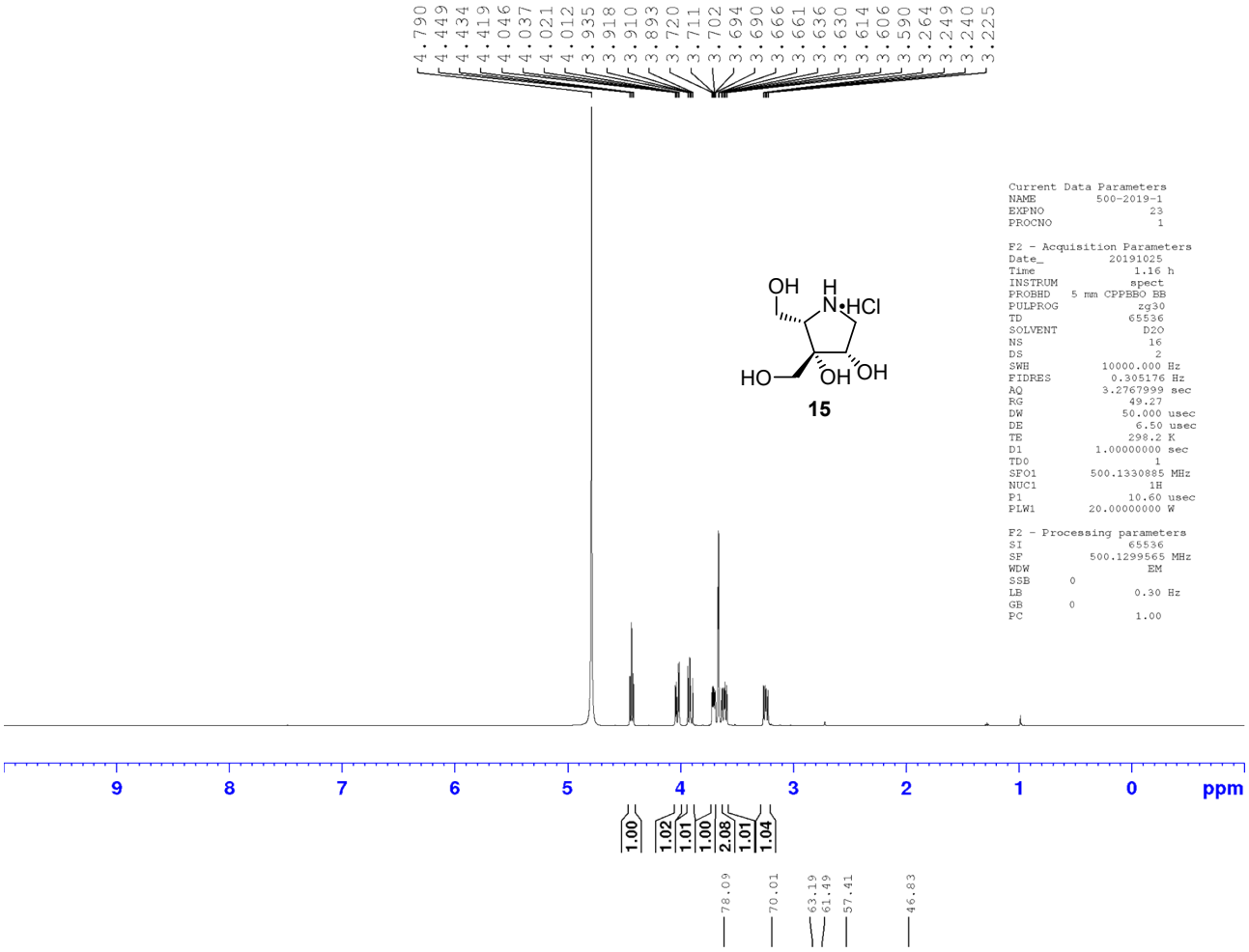


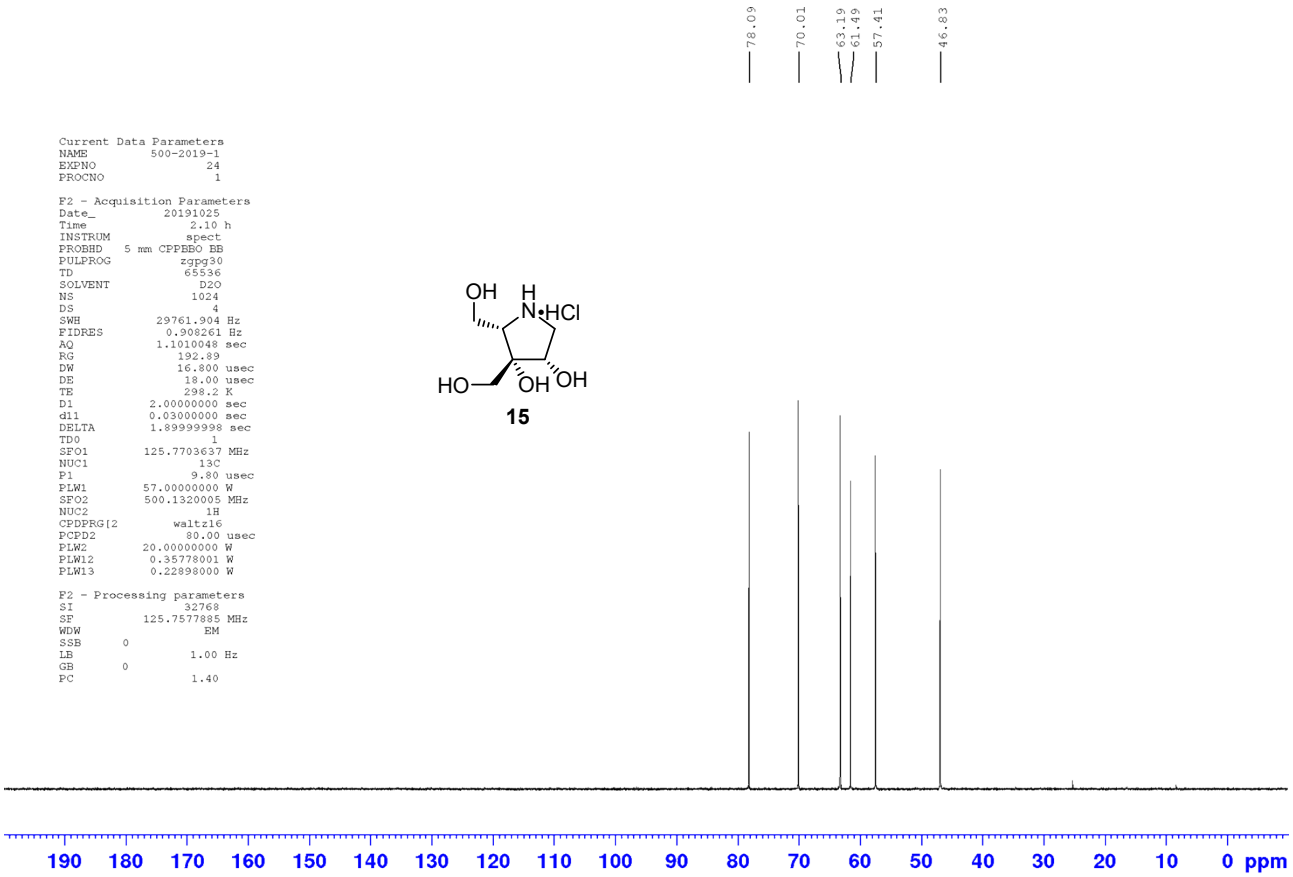


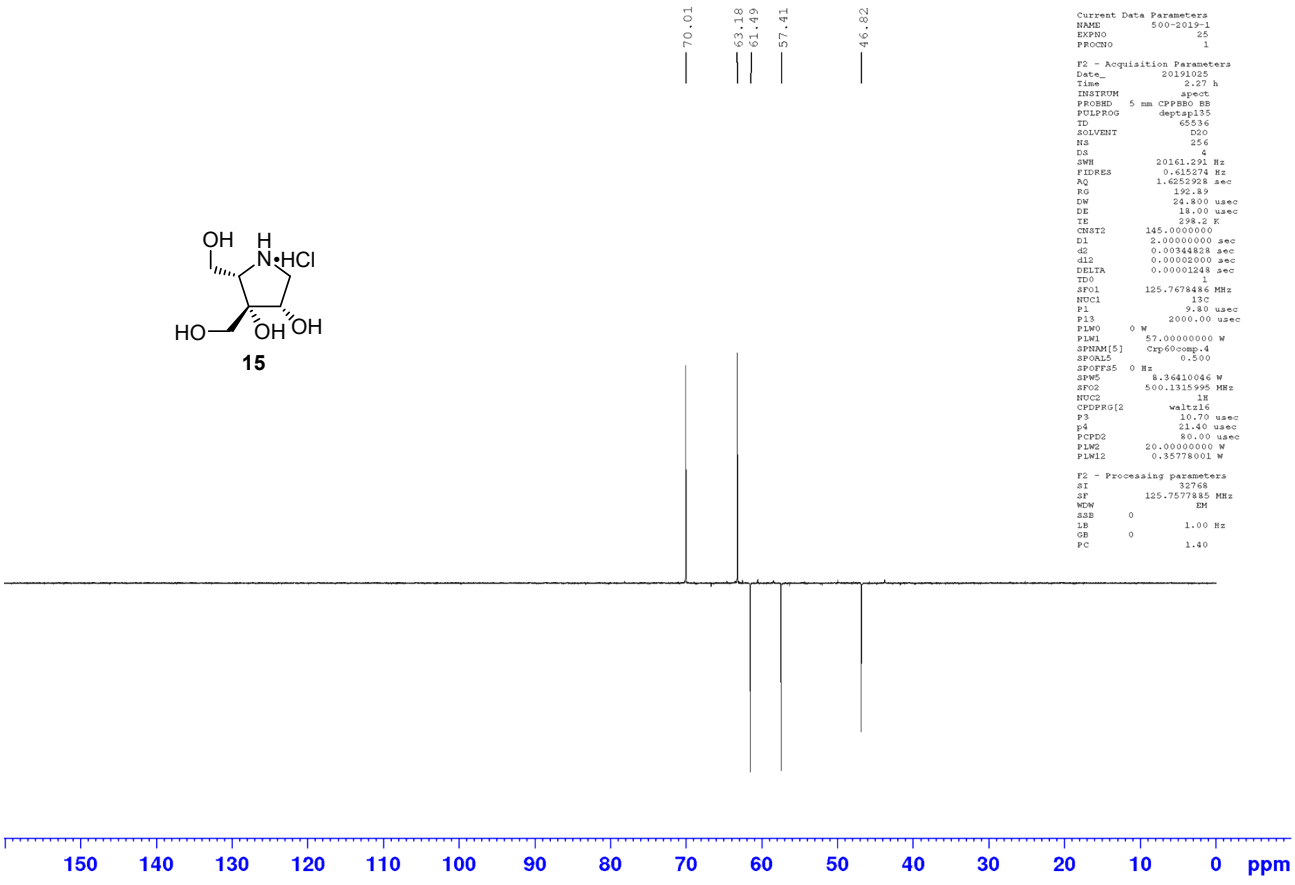

63.49
58.75
42.58
Current Data Parameters
NAME 500-2019
EXPNO 316
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190727
Time 3.10 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptsp135
TD 65536
SOLVENT D2O
NS 256
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H
N•HCl
HO
HO
OH
OH
14•HCl
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

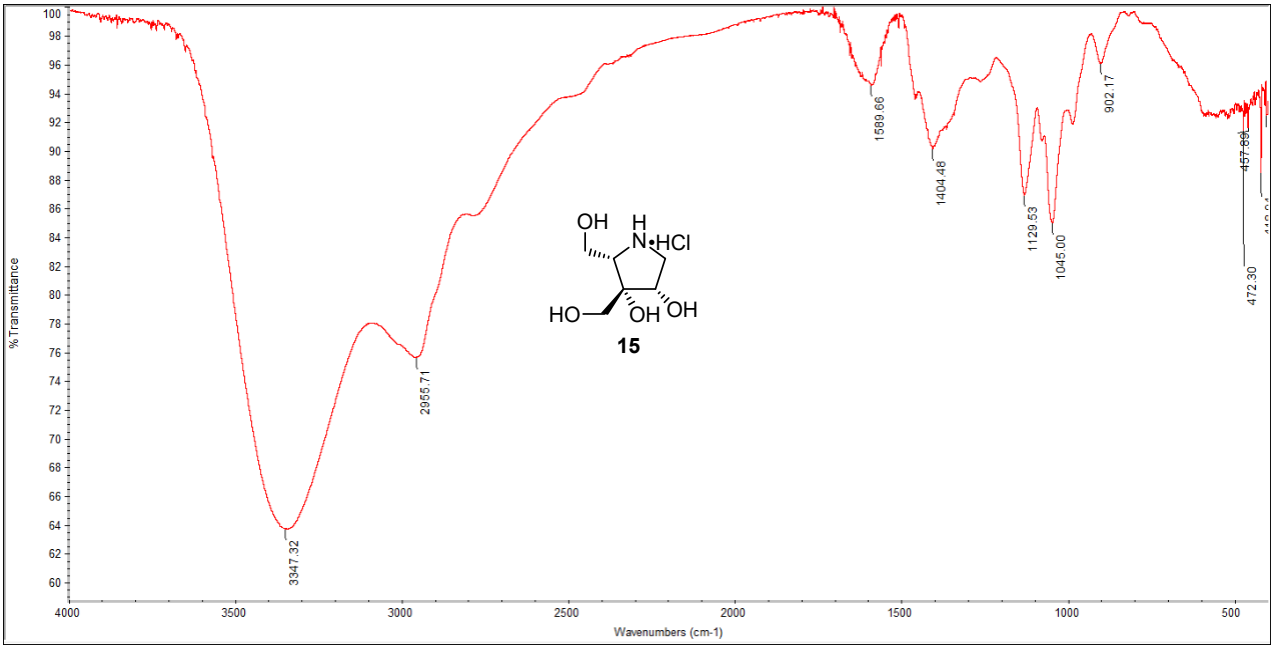

%Transmittance
Wavenumbers (cm-1)
3355.39
2806.73
1614.05
1465.44
1156.14
1113.65
1080.08
1038.89
997.35
958.03
917.35
761.40
646.21
564.87
446.42
H
N•HCl
HO
HO
OH
OH
14•HCl

Compound 15**:**

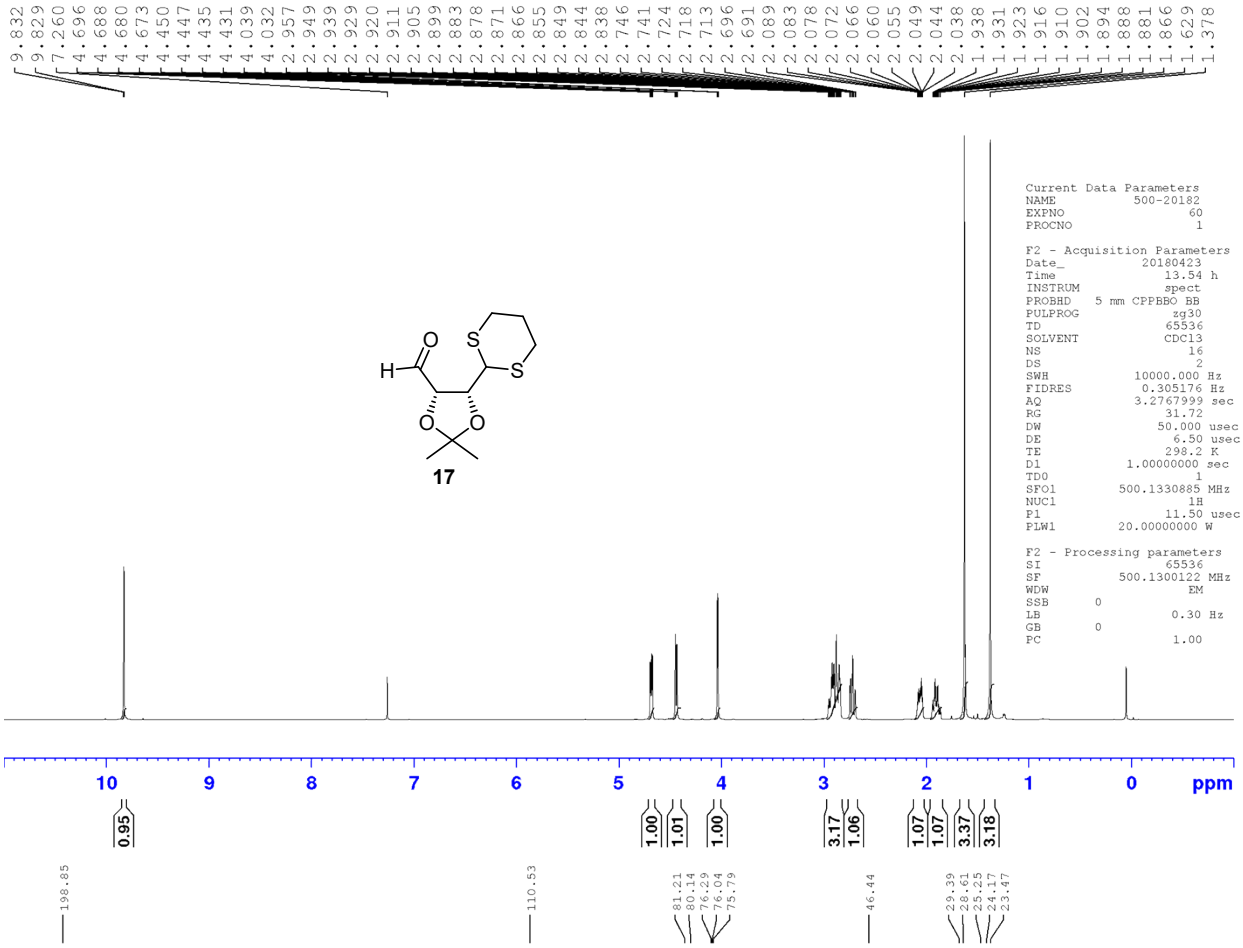

4.790
4.449
4.434
4.419
4.046
4.037
4.021
4.012
3.935
3.918
3.910
3.893
3.720
3.711
3.702
3.694
3.690
3.666
3.661
3.636
3.630
3.614
3.606
3.590
3.264
3.249
3.240
3.225
OH
H
N•HCl
HO
OH
OH
15
Current Data Parameters
NAME 500-2019-1
EXPNO 23
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191025
Time 1.16 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT D2O
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 49.27
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1299565 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
1.01
1.00
2.08
1.01
1.04

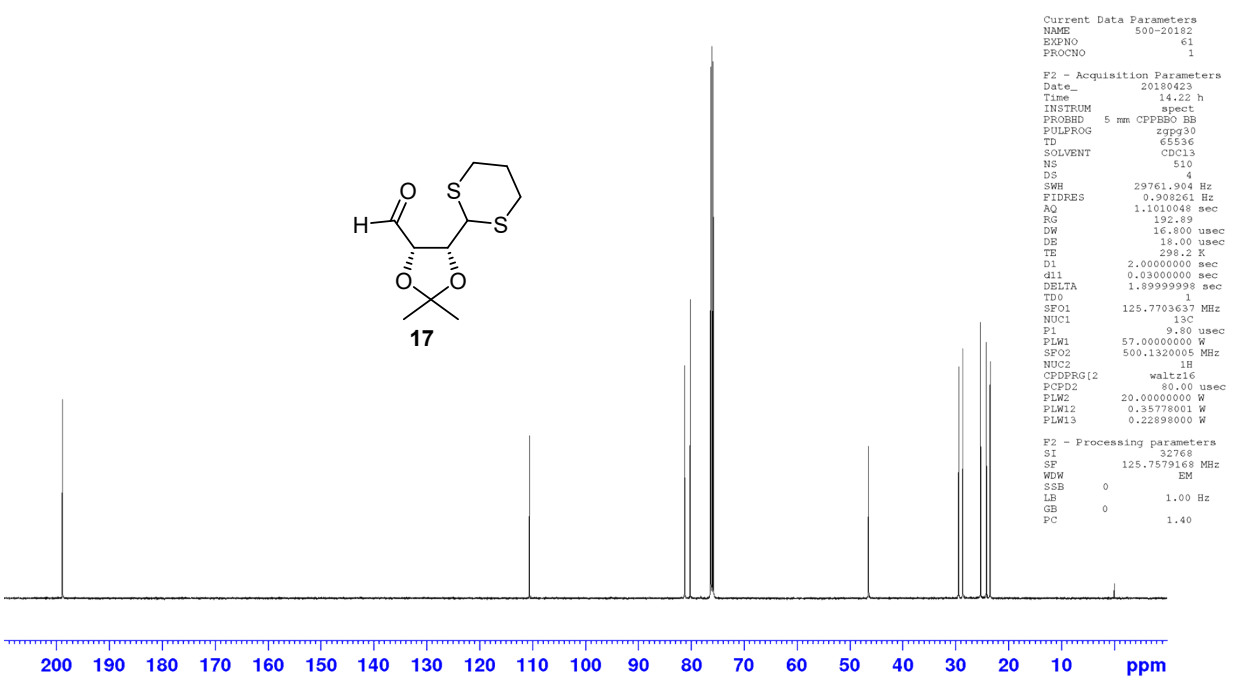

78.09
70.01
63.19
61.49
57.41
46.83
Current Data Parameters
NAME 500-2019-1
EXPNO 24
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191025
Time 2.10 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
H
N•HCl
HO
OH
OH
15
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

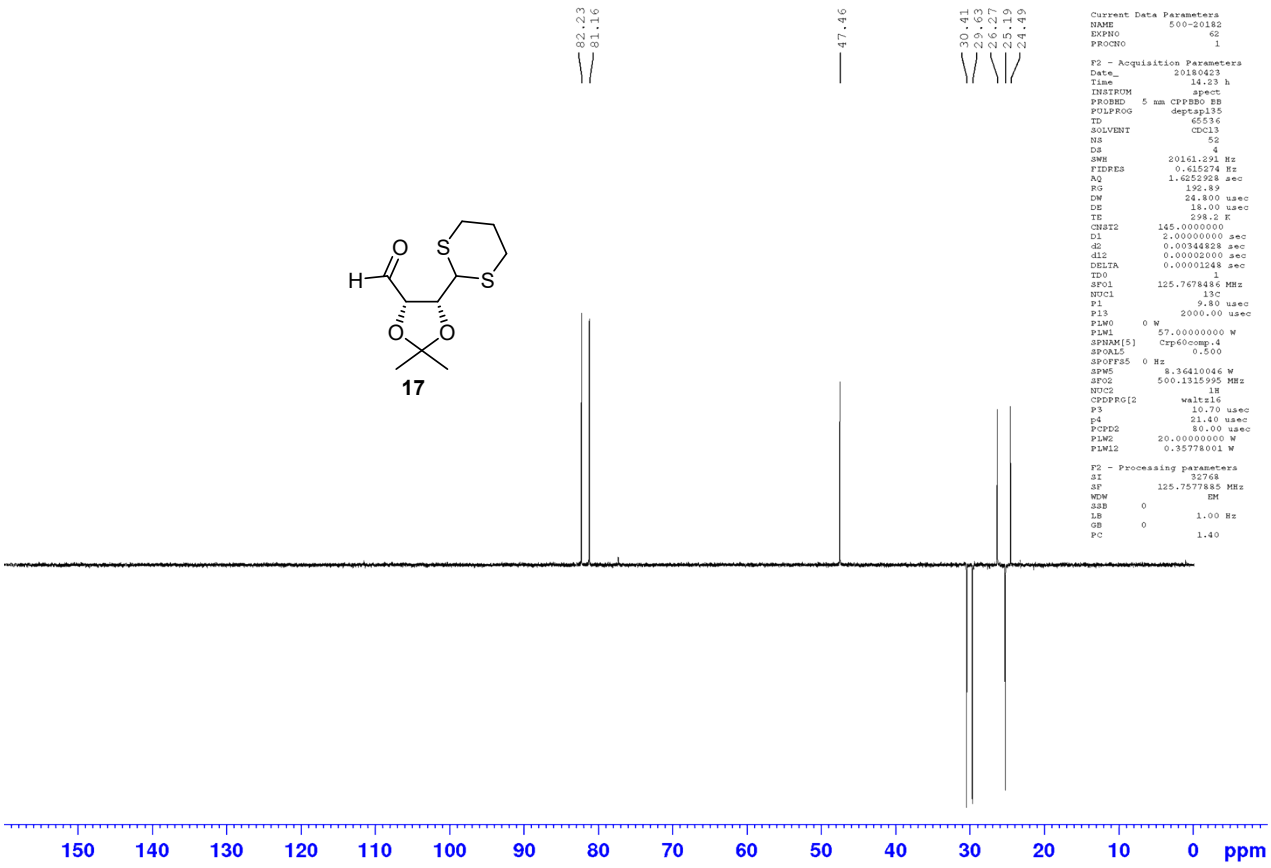

70.01
63.18
61.49
57.41
46.82
OH
H
N•HCl
HO
OH
OH
15
Current Data Parameters
NAME 500-2019-1
EXPNO 25
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191025
Time 2.27 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptsp135
TD 65536
SOLVENT D2O
NS 256
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

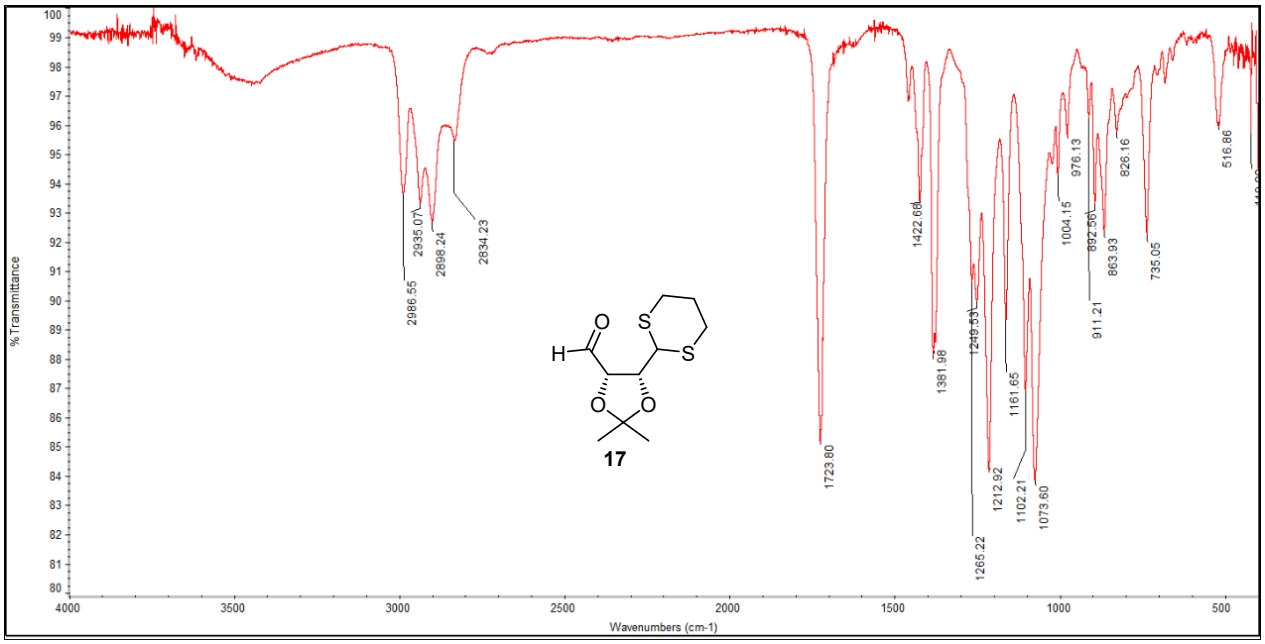

%Transmittance
Wavenumbers (cm-1)
3347.32
2955.71
1589.66
1404.48
1129.53
1045.00
902.17
472.30
OH
H
N•HCl
HO
OH
OH
15

Compound 17**:**

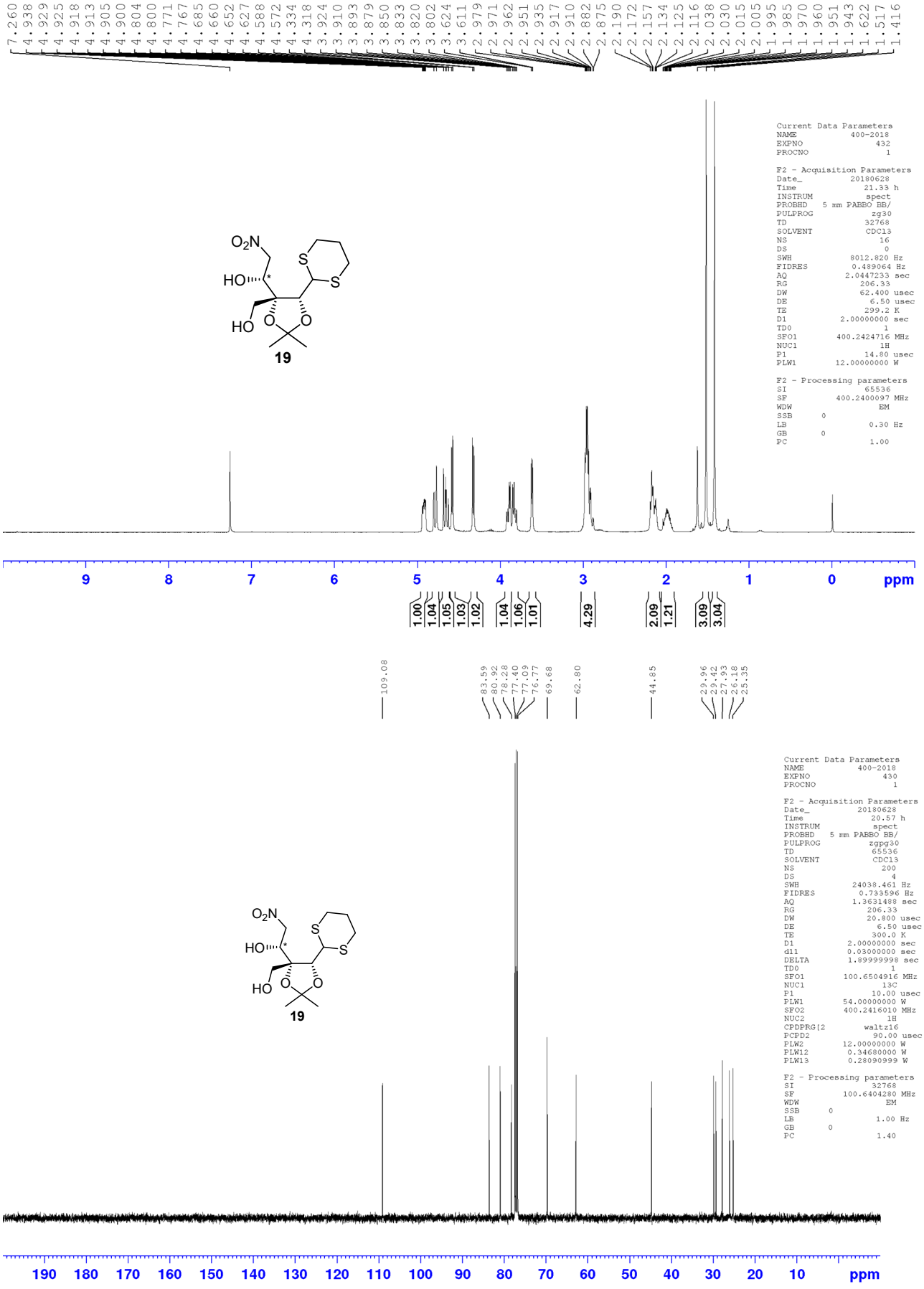

9.832
9.829
7.260
4.696
4.688
4.680
4.673
4.450
4.447
4.435
4.431
4.039
4.032
2.957
2.949
2.939
2.929
2.920
2.911
2.905
2.899
2.883
2.878
2.871
2.866
2.855
2.849
2.844
2.838
2.746
2.741
2.724
2.718
2.713
2.696
2.691
2.089
2.083
2.078
2.072
2.066
2.060
2.055
2.049
2.044
2.038
1.938
1.931
1.923
1.916
1.910
1.902
1.894
1.888
1.881
1.866
1.629
1.378
17
Current Data Parameters
NAME 500-20182
EXPNO 60
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180423
Time 13.54 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.95
1.00
1.01
1.00
3.17
1.06
1.07
1.07
3.37
3.18
198.85
110.53
81.21
80.14
76.29
76.04
75.79
46.44
29.39
28.61
25.25
24.17
23.47

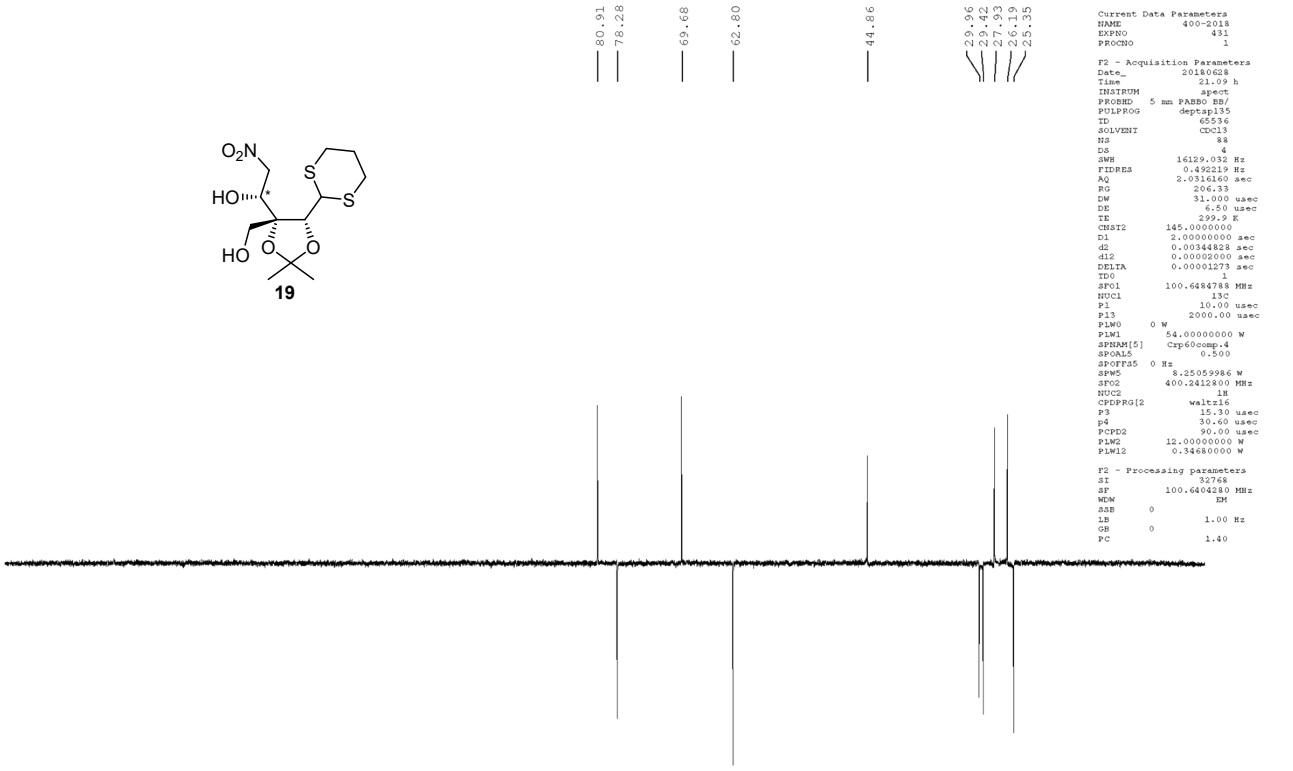

17
Current Data Parameters
NAME 500-20182
EXPNO 61
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180423
Time 14.22 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 510
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7579168 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

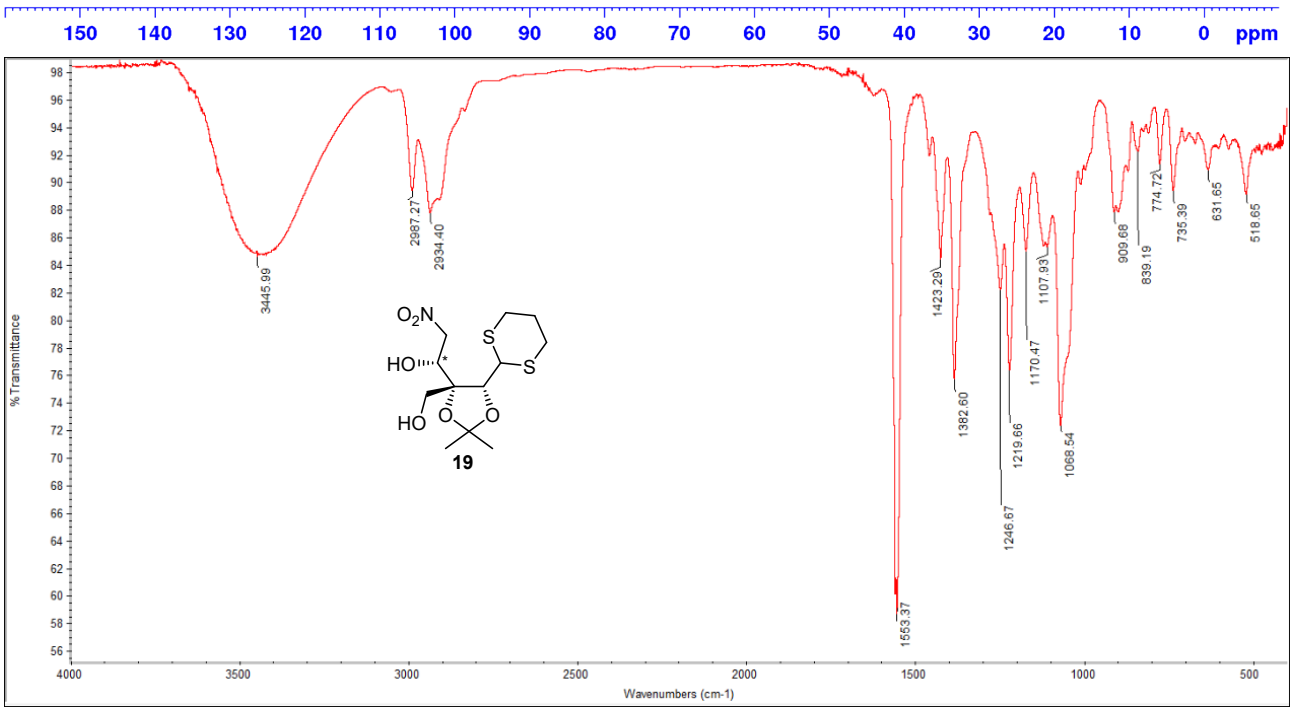

82.23
81.16
47.46
30.41
29.63
26.27
25.19
24.49
17
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


% Transmittance
Wavenumbers (cm-1)
2986.55
2935.07
2898.24
2834.23
1723.80
1422.68
1381.98
1265.22
1249.93
1212.92
1161.65
1102.21
1073.60
1004.15
976.13
911.21
892.86
863.93
826.16
735.05
516.86
17

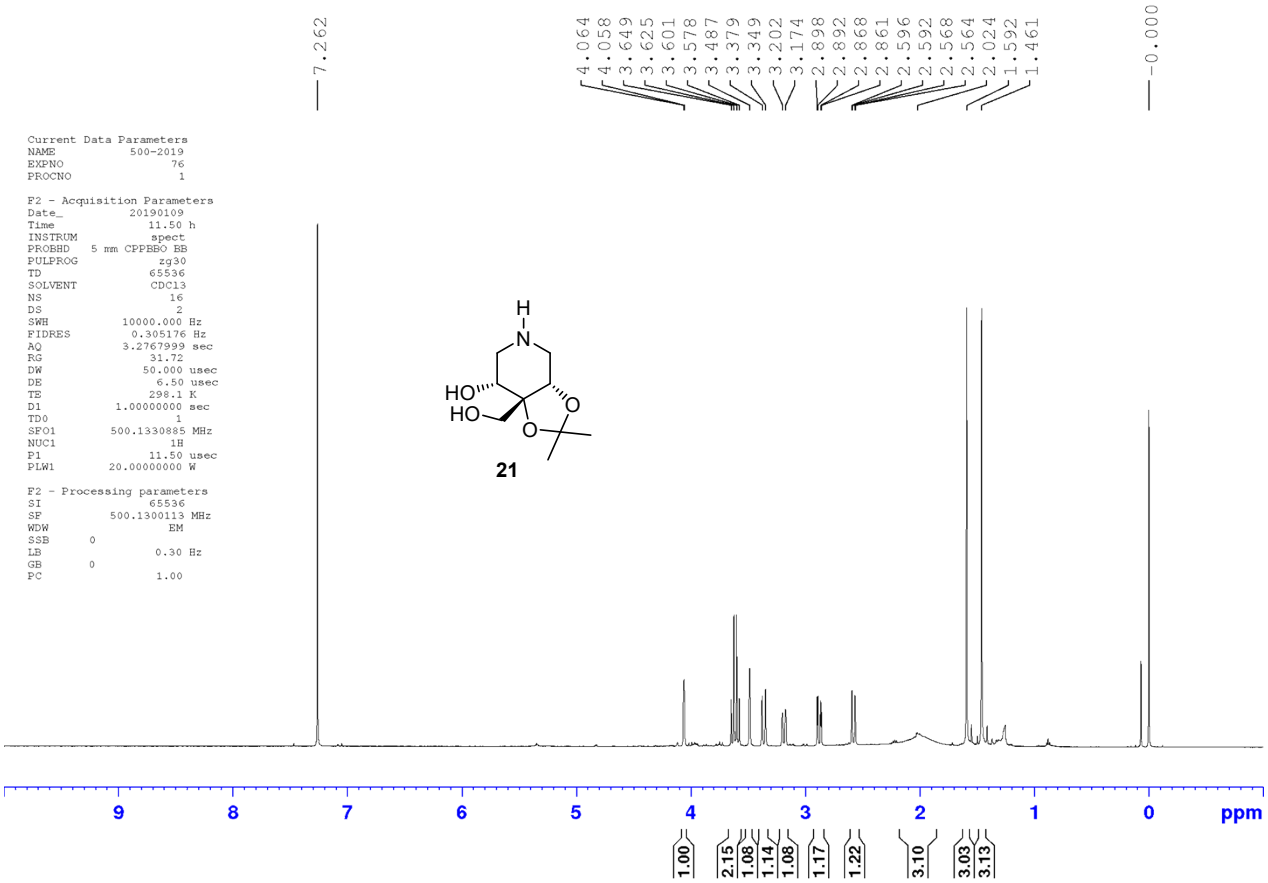
Compound 19**:**

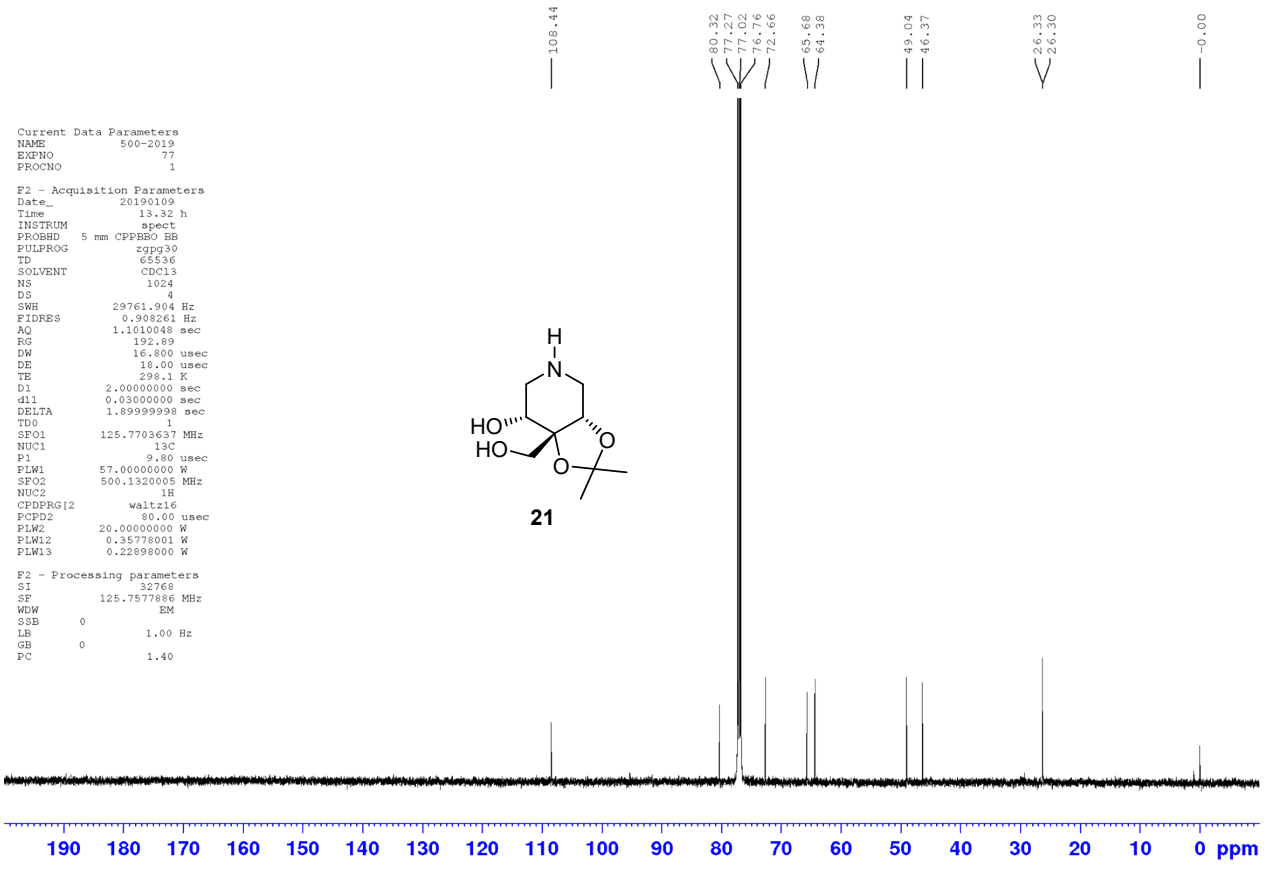

7.260 4.938 4.929 4.925 4.918 4.913 4.905 4.900 4.804 4.800 4.771 4.767 4.685 4.660 4.652 4.627 4.588 4.572 4.334 4.318 3.924 3.910 3.893 3.879 3.850 3.833 3.820 3.802 3.624 3.611 2.979 2.971 2.962 2.951 2.935 2.917 2.910 2.882 2.875 2.190 2.172 2.157 2.134 2.125 2.116 2.038 2.030 2.015 2.005 1.995 1.985 1.970 1.960 1.951 1.943 1.622 1.517 1.416
O2N
HO
HO
S
S
O
O
19
Current Data Parameters
NAME 400-2018
EXPNO 432
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180628
Time 21.33 h
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 206.33
DW 62.400 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
TD0 1
SFO1 400.2424716 MHz
NUC1 1H
P1 14.80 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.2400097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.00 1.04 1.05 1.03 1.02 1.04 1.06 1.01 4.29 2.09 1.21 3.09 3.04
109.08 83.59 80.92 78.28 77.40 77.09 76.77 69.68 62.80 44.85 29.96 29.42 27.93 26.18 25.35
O2N
HO
HO
S
S
O
O
19
Current Data Parameters
NAME 400-2018
EXPNO 430
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180628
Time 20.57 h
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 206.33
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 100.6504916 MHz
NUC1 13C
P1 10.00 usec
PLW1 54.00000000 W
SFO2 400.2416010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.34680000 W
PLW13 0.28090999 W
F2 - Processing parameters
SI 32768
SF 100.6404280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

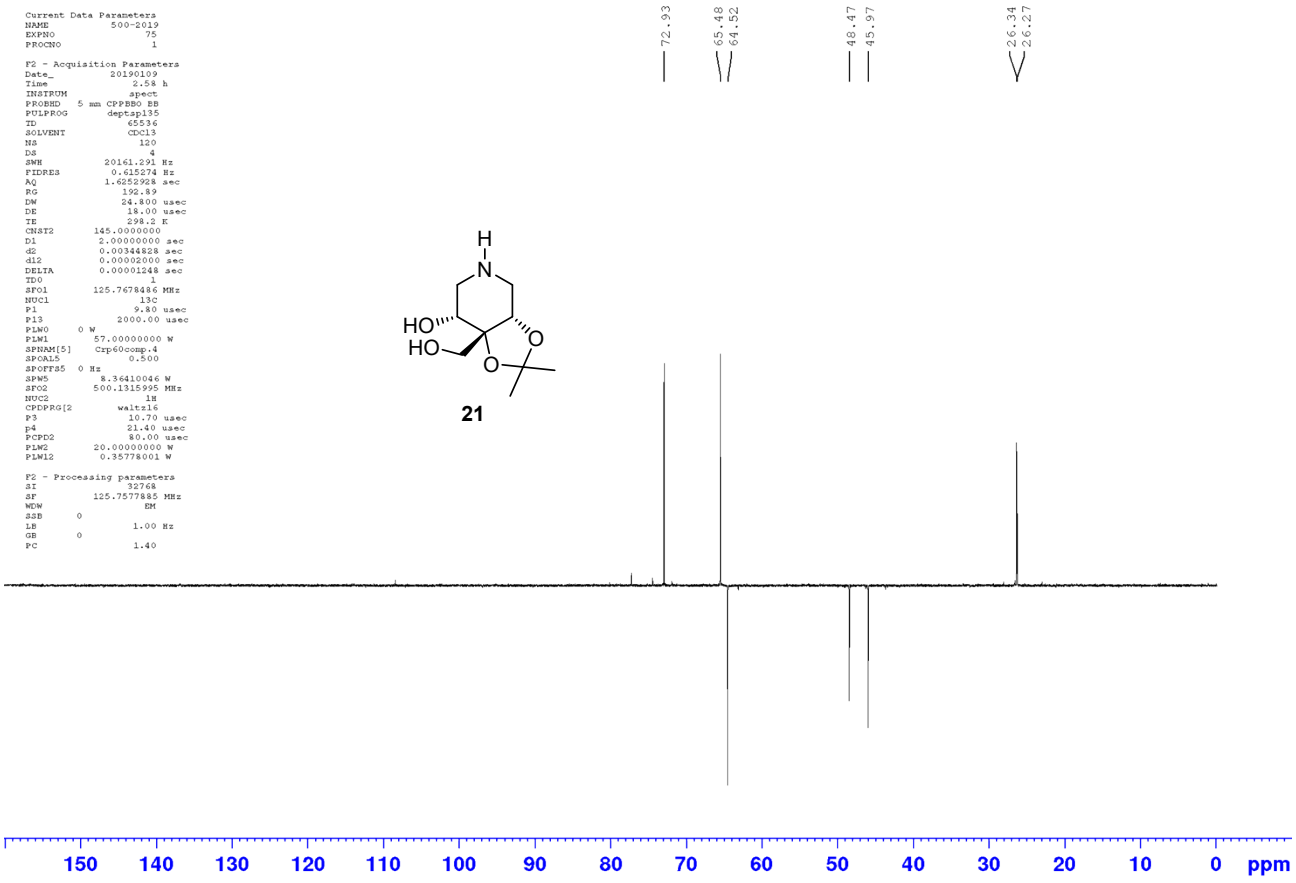

80.91
78.28
69.68
62.80
44.86
29.96
29.42
27.93
26.19
25.35
19

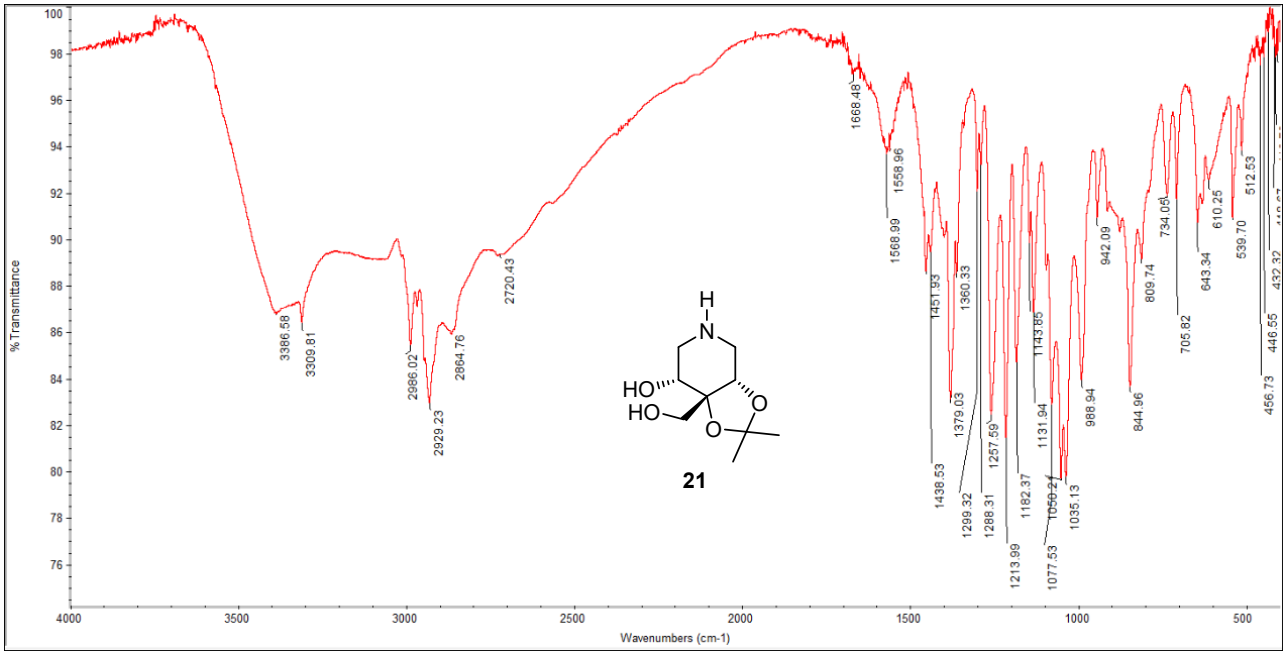

150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
% Transmittance
Wavenumbers (cm-1)
3445.99
2987.27
2934.40
1553.37
1423.29
1382.60
1246.67
1219.66
1170.47
1107.93
1068.54
909.68
839.19
774.72
735.39
631.65
518.65
19

Compound 21**:**

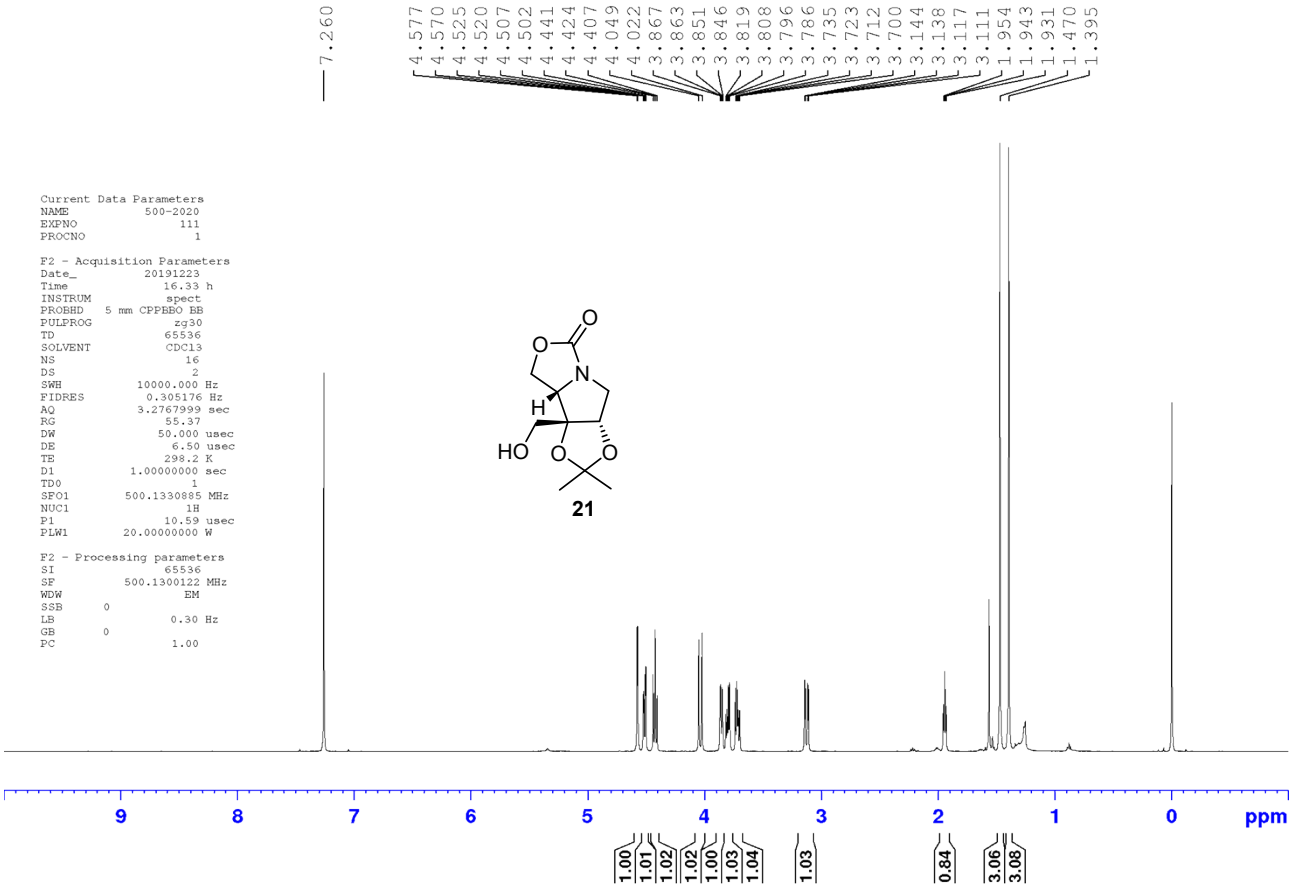

7.262
4.064
4.058
3.649
3.625
3.601
3.578
3.487
3.379
3.349
3.202
3.174
2.898
2.892
2.868
2.861
2.596
2.592
2.568
2.564
2.024
1.592
1.461
0.000
Current Data Parameters
NAME 500-2019
EXPNO 76
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190109
Time 11.50 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HO
HO
O
O
H
N
21
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.15
1.08
1.14
1.08
1.17
1.22
3.10
3.03
3.13

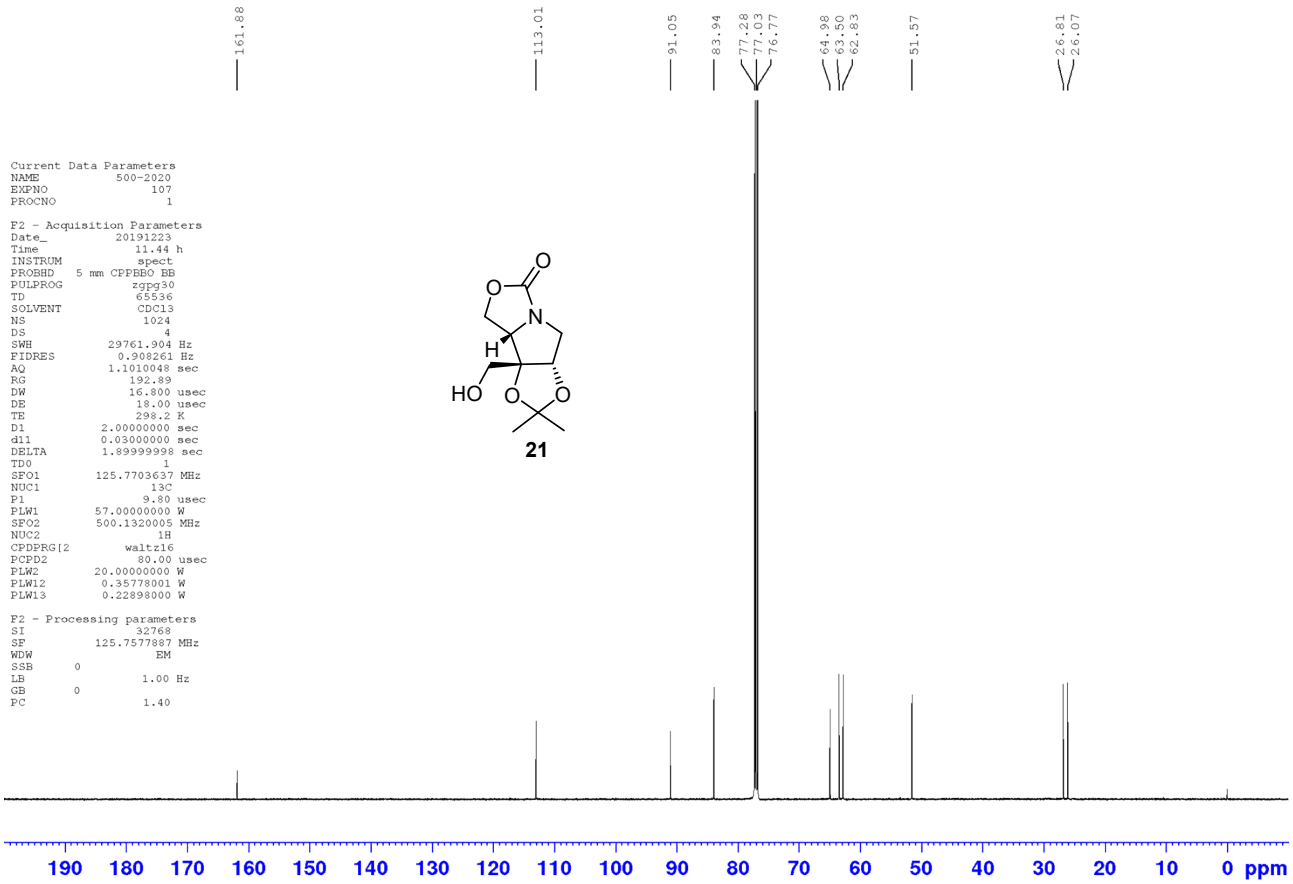

108.44
80.32
77.27
77.02
76.76
72.66
65.68
64.38
49.04
46.37
26.33
26.30
0.00
Current Data Parameters
NAME 500-2019
EXPNO 77
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190109
Time 13.32 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577886 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HO
HO
O
O
H
N
21
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

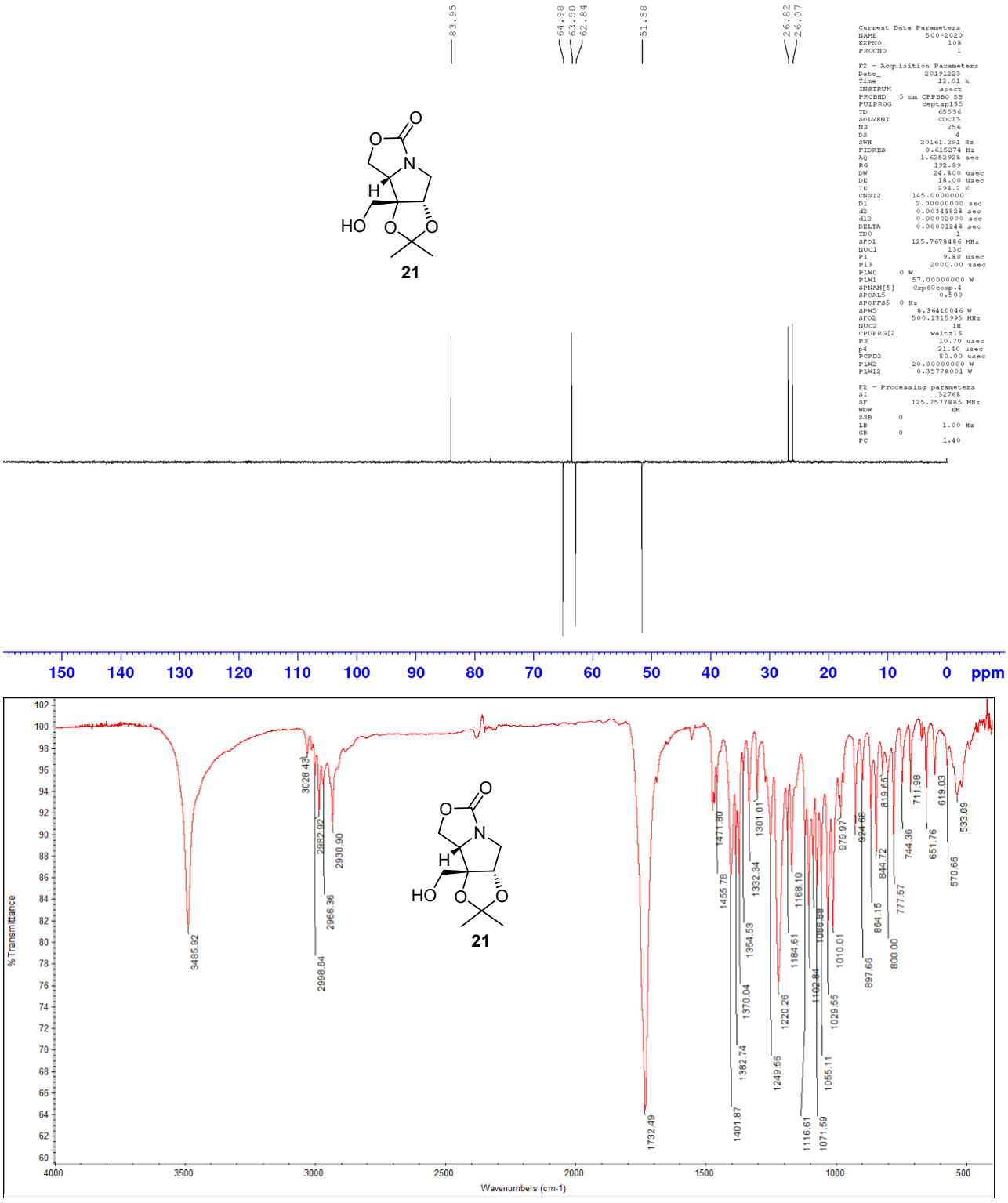

Current Data Parameters
NAME 500-2019
EXPNO 75
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190109
Time 2.58 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 120
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
72.93
65.48
64.52
48.47
45.97
26.34
26.27
HO
HO
O
O
H
N
21
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


% Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
500
100
98
96
94
92
90
88
86
84
82
80
78
76
3386.58
3309.81
2986.02
2929.23
2864.76
2720.43
1668.48
1568.99
1558.96
1451.93
1438.53
1379.03
1360.33
1299.32
1288.31
1257.59
1213.99
1182.37
1143.85
1131.94
1077.53
1050.21
1035.13
988.94
942.09
844.96
809.74
734.05
705.82
643.34
610.25
539.70
512.53
456.73
446.55
432.32
HO
HO
O
O
H
N
21

Compound 21**:**

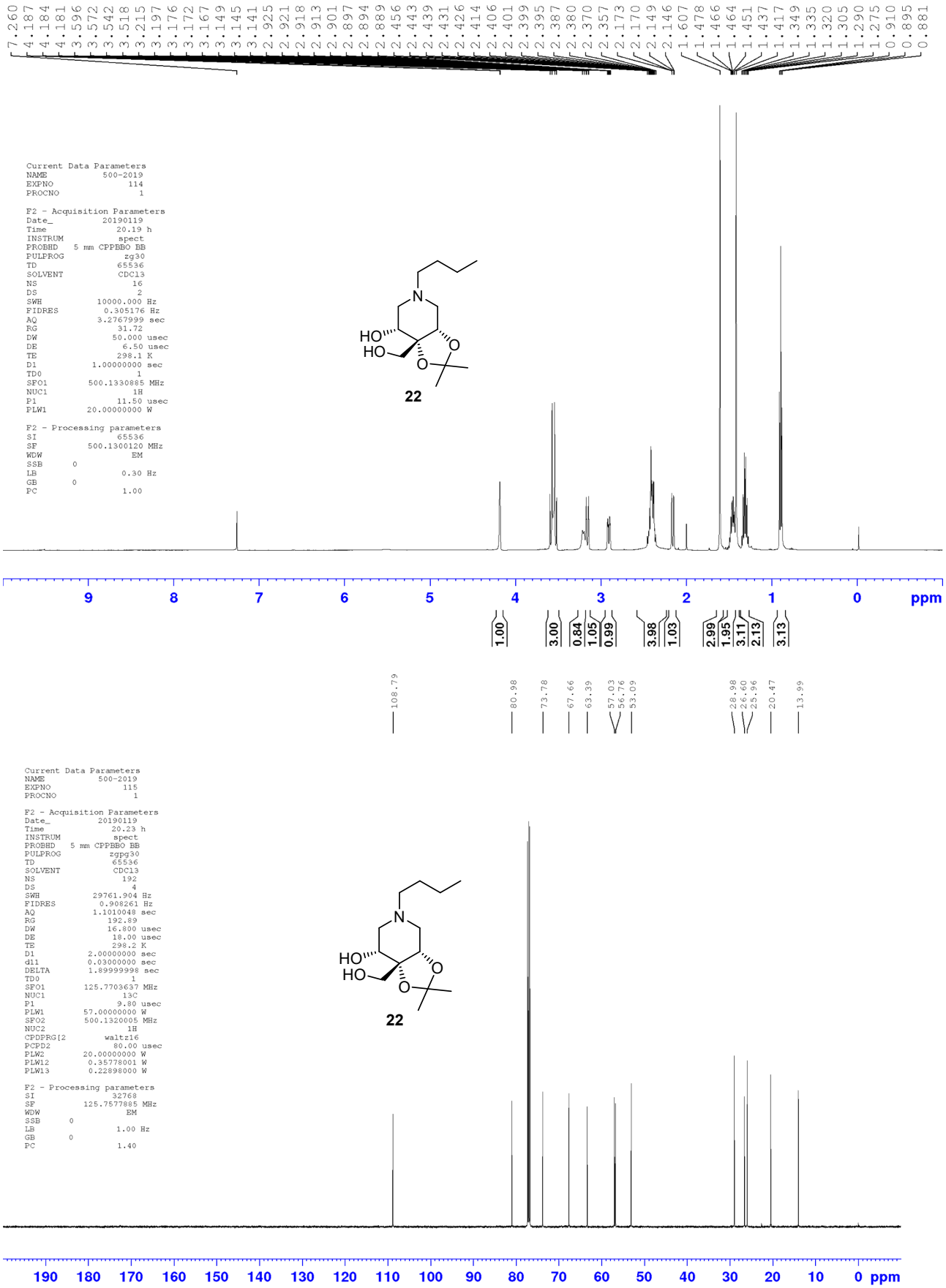

7.260
4.577
4.570
4.525
4.520
4.507
4.502
4.441
4.424
4.407
4.049
4.022
3.867
3.863
3.851
3.846
3.819
3.808
3.796
3.786
3.735
3.723
3.712
3.700
3.144
3.138
3.117
3.111
1.954
1.943
1.931
1.470
1.395
Current Data Parameters
NAME 500-2020
EXPNO 111
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191223
Time 16.33 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 55.37
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.59 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
21
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
1.02
1.02
1.00
1.03
1.04
1.03
0.84
3.06
3.08

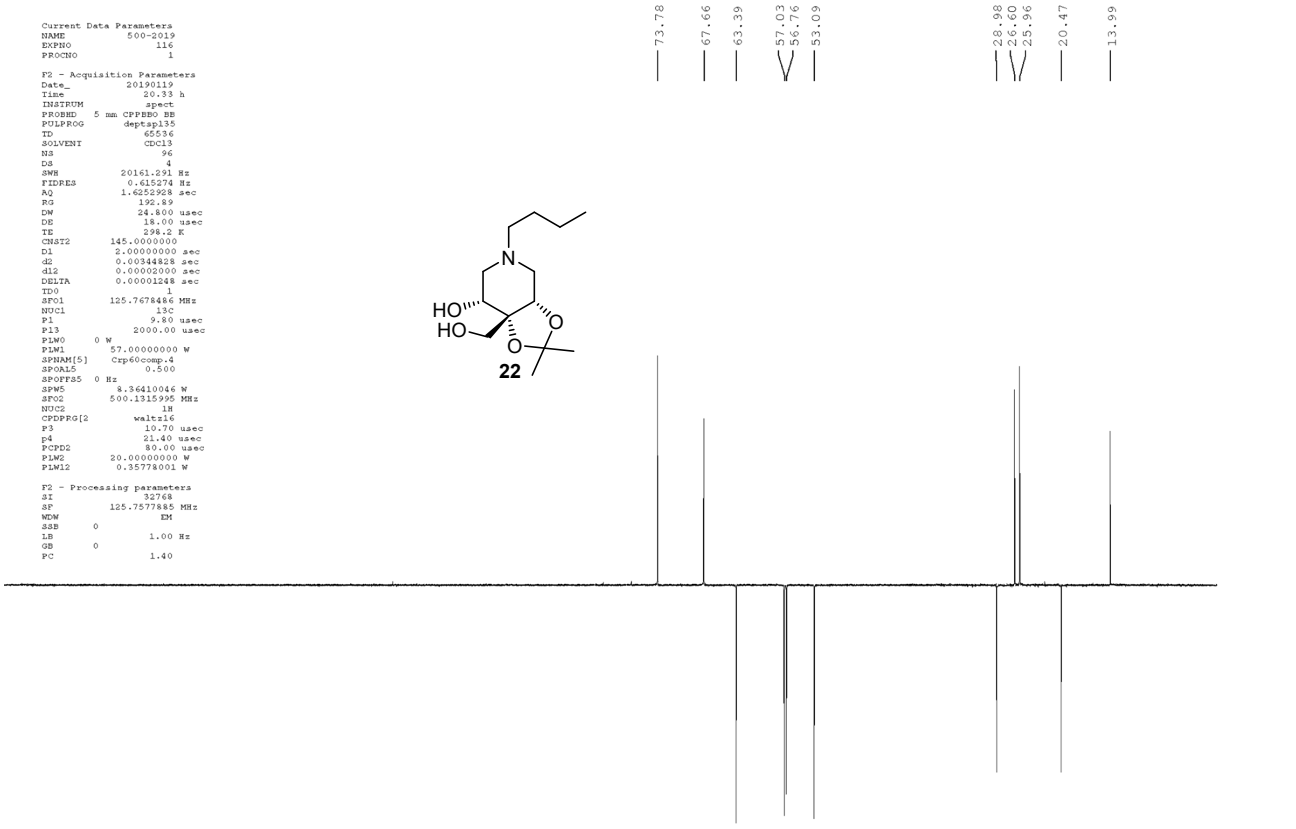

161.88
113.01
91.05
83.94
77.28
77.03
76.77
64.98
63.50
62.83
51.57
26.81
26.07
Current Data Parameters
NAME 500-2020
EXPNO 107
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191223
Time 11.44 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577887 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

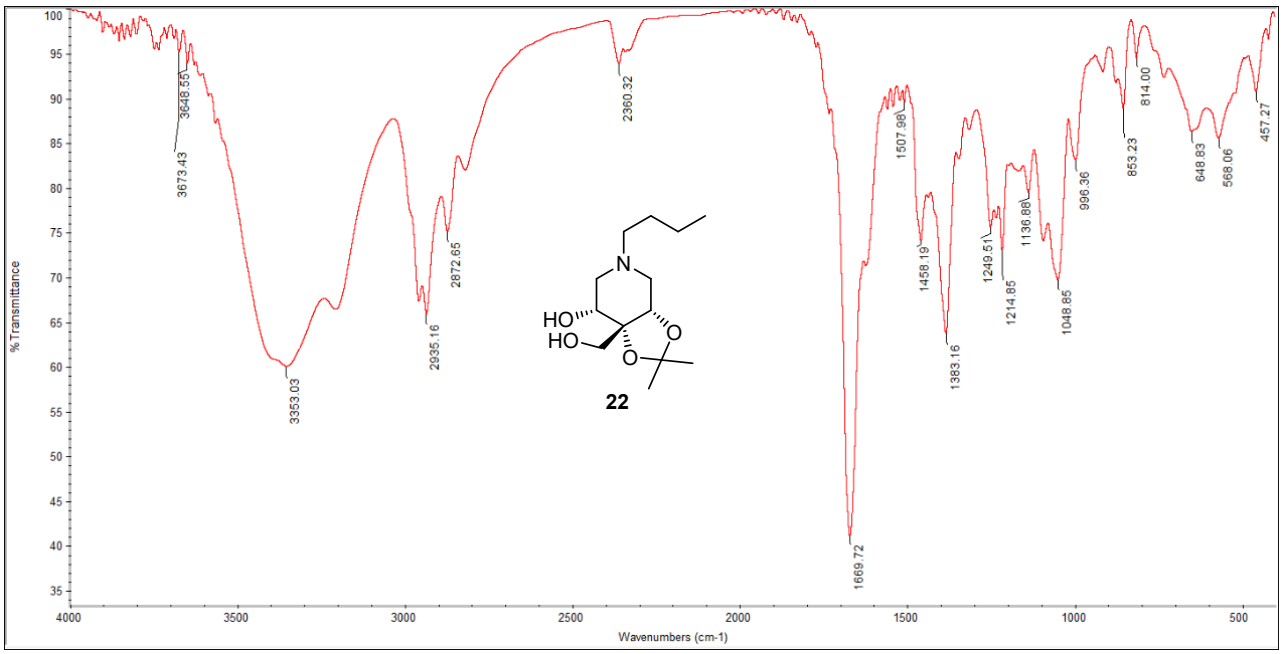

83.95
64.98
63.50
62.84
51.58
26.82
26.07
21
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

% Transmittance
Wavenumbers (cm-1)
3485.92
3028.43
2982.92
2966.36
2930.90
2898.64
1732.49
1471.80
1455.78
1401.87
1382.74
1370.04
1354.53
1332.34
1301.01
1249.56
1220.26
1184.61
1168.10
1116.61
1102.84
1071.59
1055.11
1029.55
1010.01
979.97
924.68
897.66
864.15
844.72
819.65
800.00
777.57
744.36
711.98
651.76
619.03
570.66
533.09
21

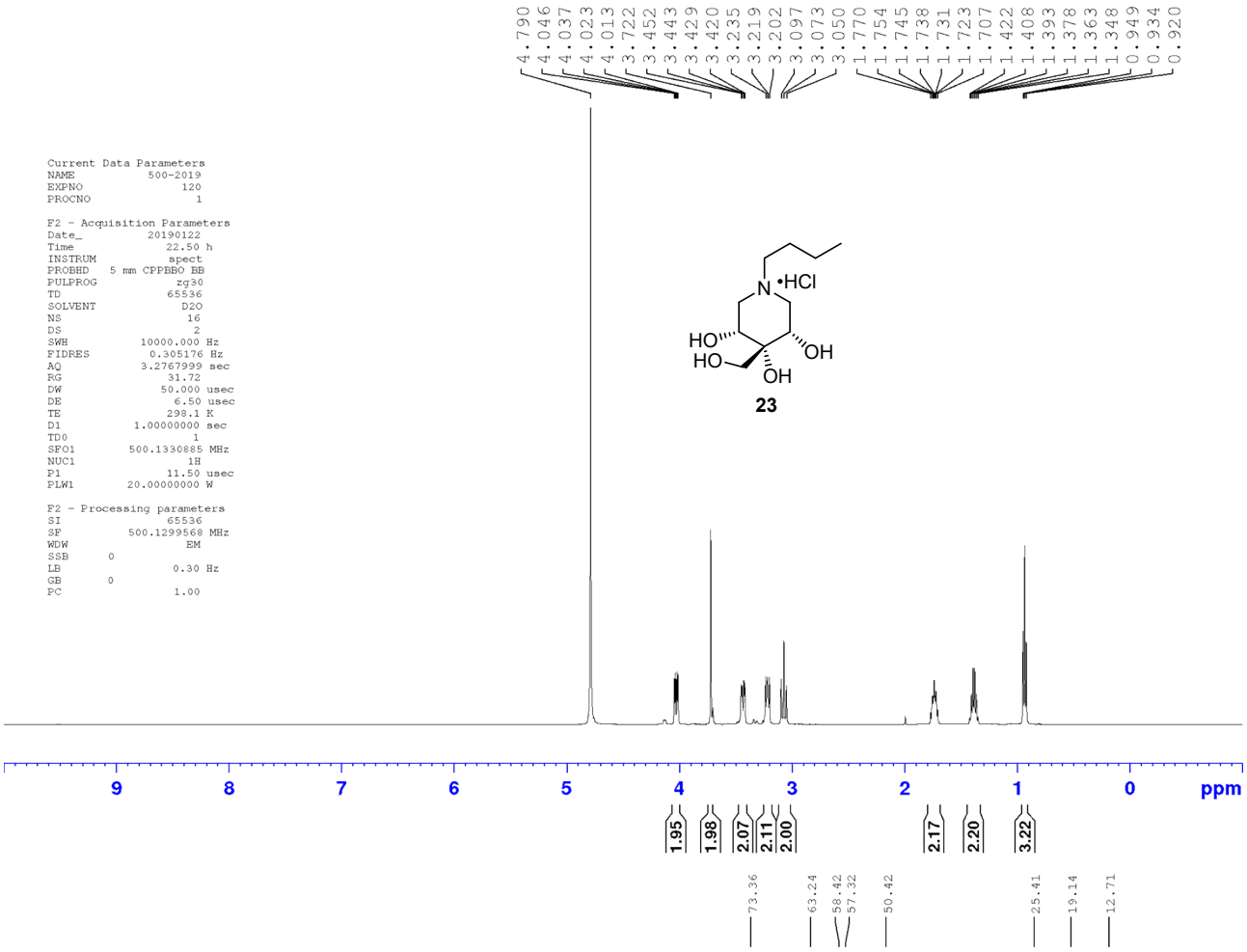
Compound 22:

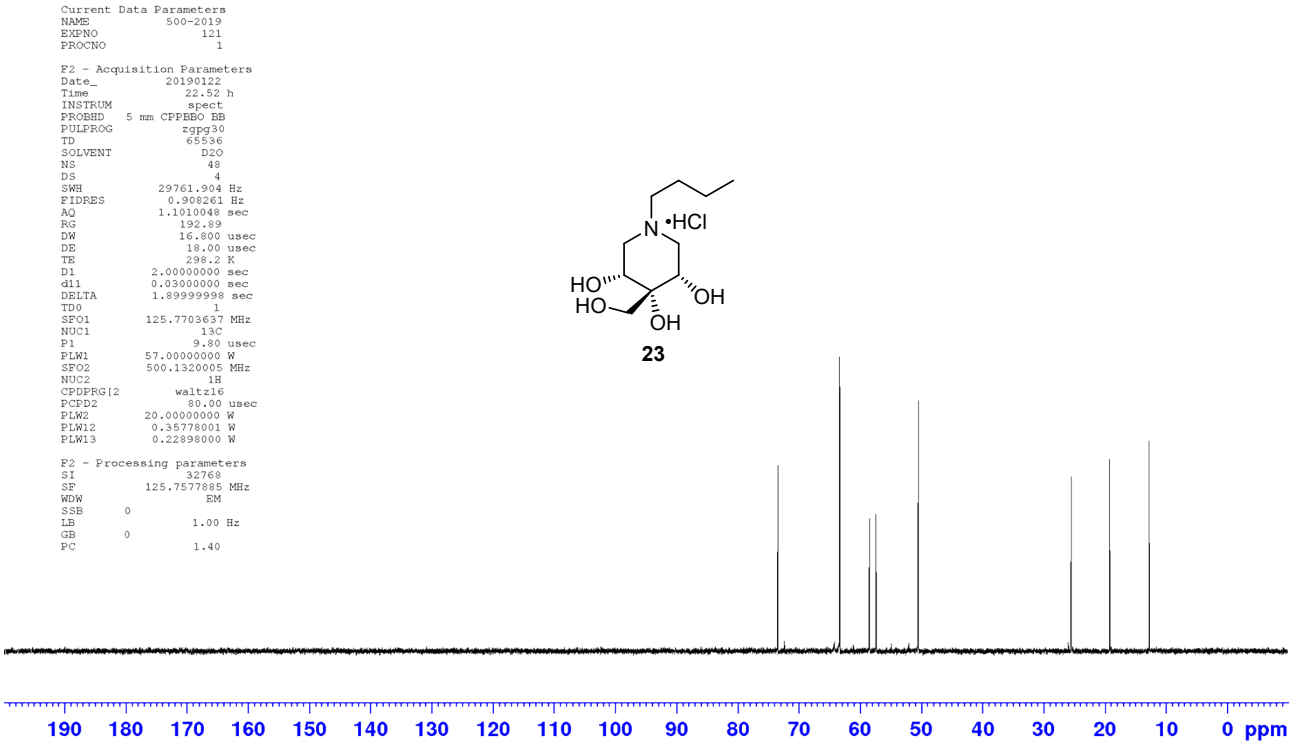

7.260 4.187 4.184 4.181 3.596 3.572 3.542 3.518 3.215 3.197 3.176 3.172 3.167 3.149 3.145 3.141 2.925 2.921 2.918 2.913 2.901 2.897 2.894 2.889 2.456 2.443 2.439 2.431 2.426 2.414 2.406 2.401 2.399 2.395 2.387 2.380 2.370 2.357 2.173 2.170 2.149 2.146 1.607 1.478 1.466 1.464 1.451 1.437 1.417 1.349 1.335 1.320 1.305 1.290 1.275 0.910 0.895 0.881
Current Data Parameters
NAME 500-2019
EXPNO 114
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190119
Time 20.19 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HO
HO
N
O
O
22
9 8 7 6 5 4 3 2 1 0 ppm
1.00 3.00 0.84 1.05 0.99 3.98 1.03 2.99 1.95 3.11 2.13 3.13
108.79 80.98 73.78 67.66 63.39 57.03 56.76 53.09 28.98 26.60 25.96 20.47 13.99
Current Data Parameters
NAME 500-2019
EXPNO 115
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190119
Time 20.23 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 192
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HO
HO
N
O
O
22
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

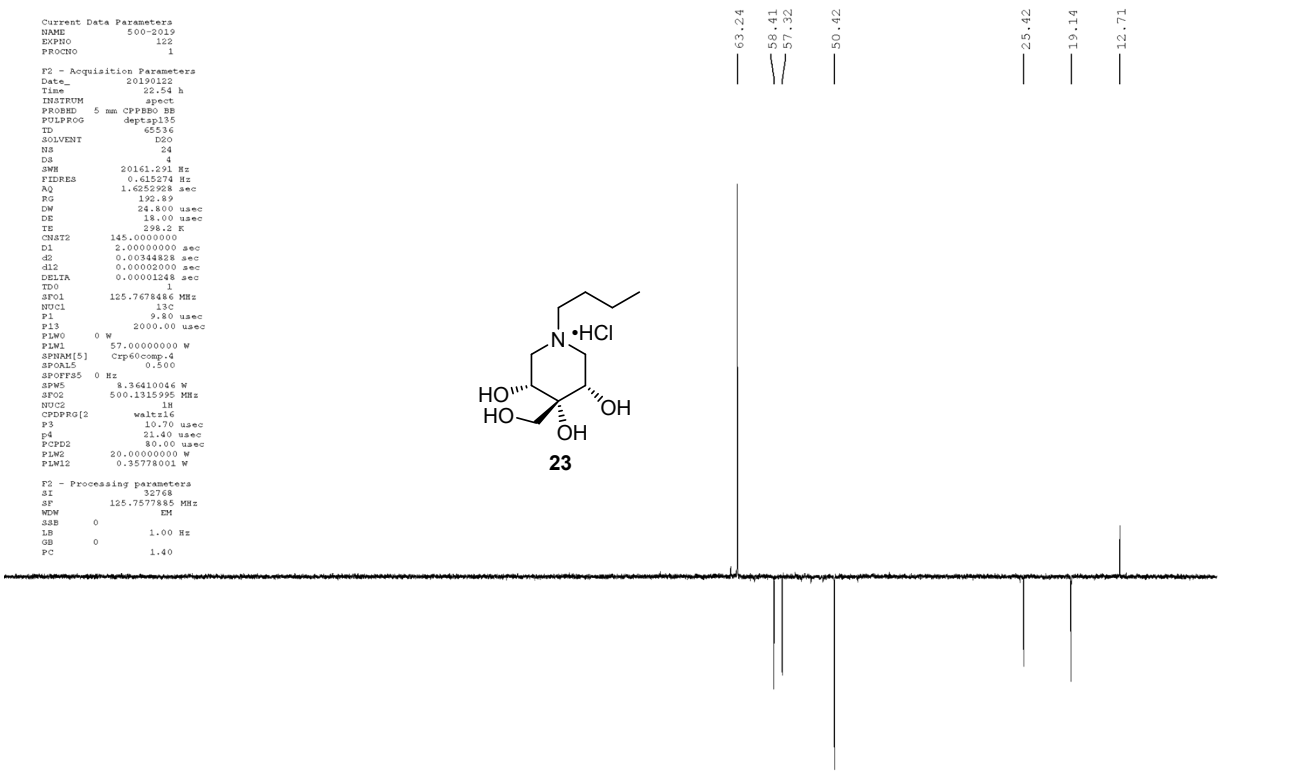

Current Data Parameters
NAME 500-2019
EXPNO 116
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190119
Time 20.33 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 96
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
73.78
67.66
63.39
57.03
56.76
53.09
28.98
26.60
25.96
20.47
13.99
HO
HO
N
O
O
22
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

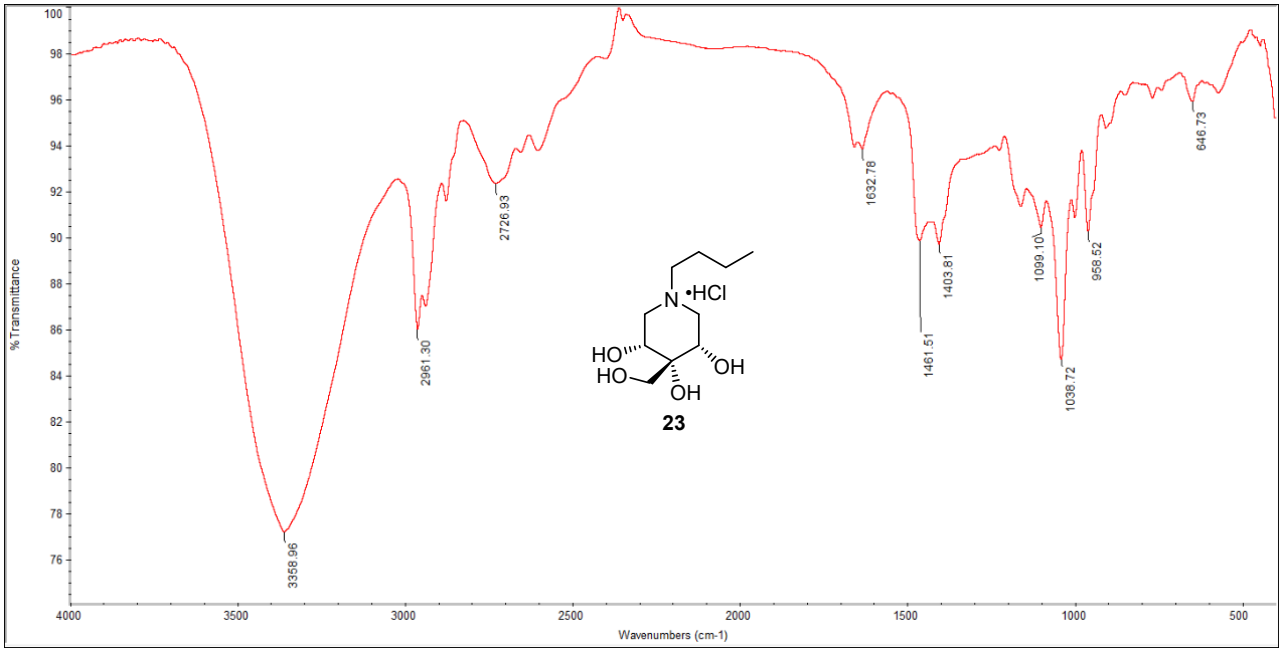

% Transmittance
Wavenumbers (cm-1)
3673.43
3648.55
3353.03
2935.16
2872.65
2360.32
1669.72
1507.98
1458.19
1383.16
1249.51
1214.85
1136.88
1048.85
996.36
853.23
814.00
648.83
568.06
457.27
HO
HO
N
O
O
22

Compound 23:

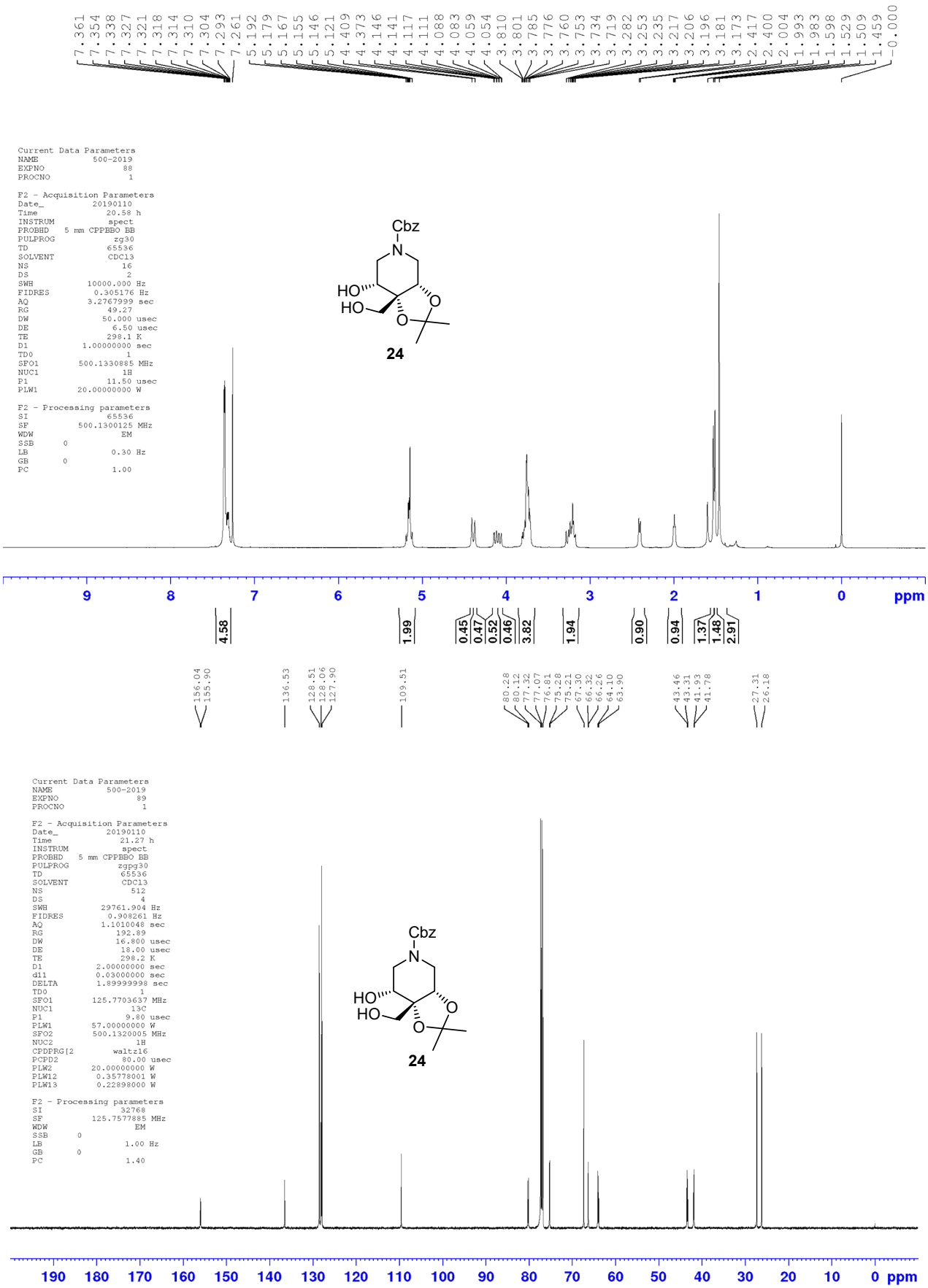

4.790
4.046
4.037
4.023
4.013
3.722
3.452
3.443
3.429
3.420
3.235
3.219
3.202
3.097
3.073
3.050
1.770
1.754
1.745
1.738
1.731
1.723
1.707
1.422
1.408
1.393
1.378
1.363
1.348
0.949
0.934
0.920
Current Data Parameters
NAME 500-2019
EXPNO 120
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190122
Time 22.50 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT D2O
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1299568 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
•HCl
HO
HO
OH
OH
23
9
8
7
6
5
4
3
2
1
0
ppm
1.95
1.98
2.07
2.11
2.00
2.17
2.20
3.22

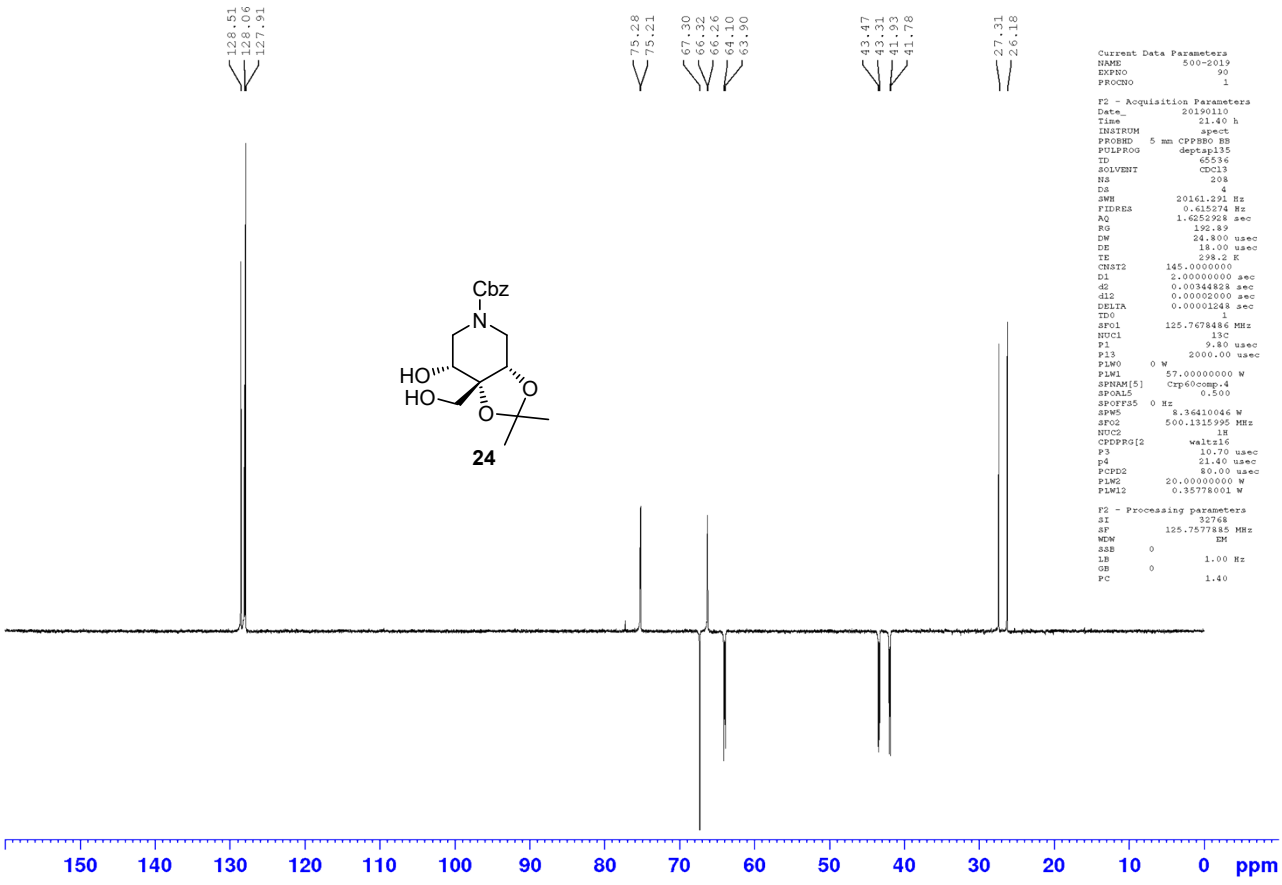

73.36
63.24
58.42
57.32
50.42
25.41
19.14
12.71
Current Data Parameters
NAME 500-2019
EXPNO 121
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190122
Time 22.52 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 48
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778801 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
•HCl
HO
HO
OH
OH
23
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

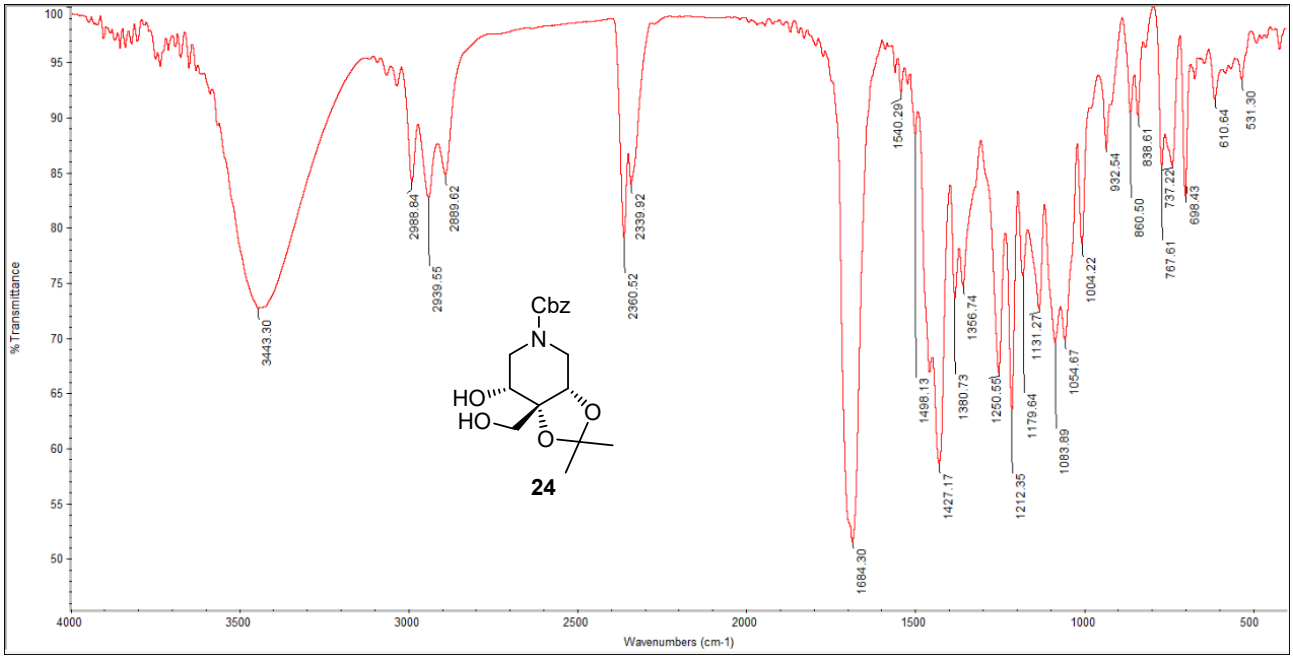

Current Data Parameters
NAME 500-2019
EXPNO 122
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190122
Time 22.54 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptap135
TD 65536
SOLVENT D2O
NS 24
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
63.24
58.41
57.32
50.42
25.42
19.14
12.71
N •HCl
HO
HO
OH
OH
23
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


% Transmittance
Wavenumbers (cm-1)
3358.96
2961.30
2726.93
1632.78
1461.51
1403.81
1099.10
1038.72
958.52
646.73
N •HCl
HO
HO
OH
OH
23

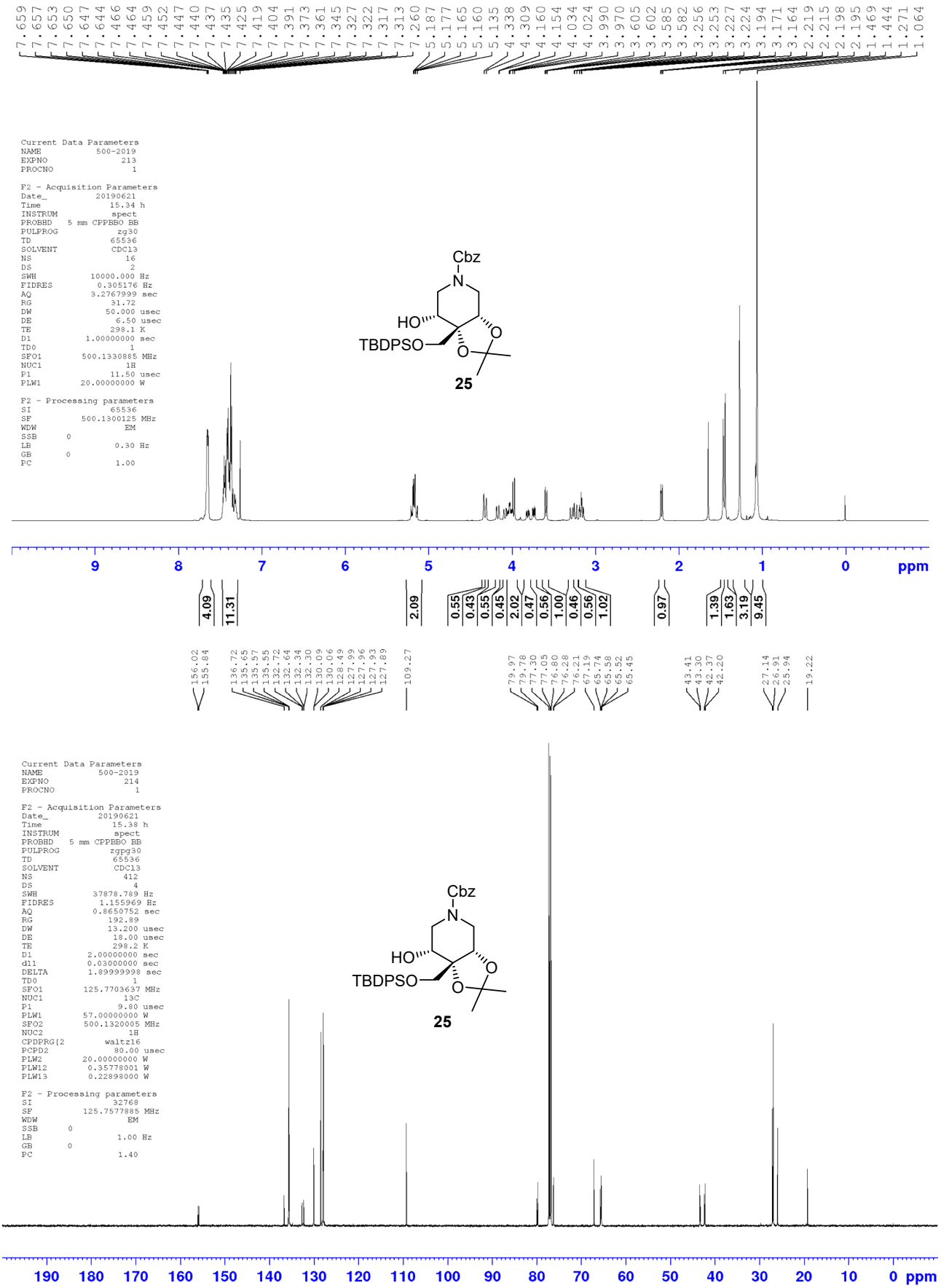
Compound 24**:**

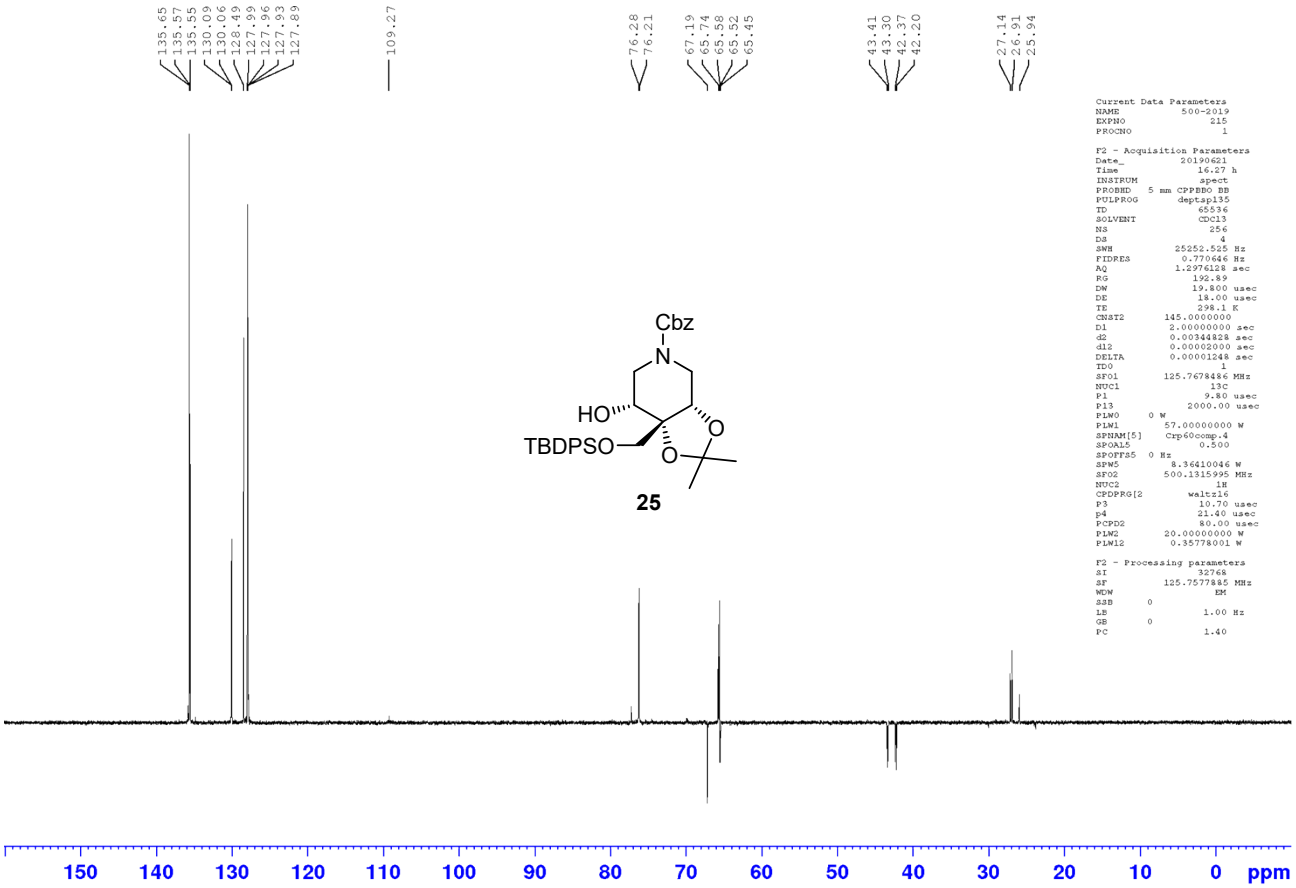

7.361
7.354
7.338
7.327
7.321
7.318
7.314
7.310
7.304
7.293
7.261
5.192
5.179
5.167
5.155
5.146
5.121
4.409
4.373
4.146
4.141
4.117
4.111
4.088
4.083
4.059
4.054
3.810
3.801
3.785
3.776
3.760
3.753
3.734
3.719
3.282
3.253
3.235
3.217
3.206
3.196
3.181
3.173
2.417
2.400
2.004
1.993
1.983
1.598
1.529
1.509
1.459
0.000
Current Data Parameters
NAME 500-2019
EXPNO 88
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190110
Time 20.58 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 49.27
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
N
HO
HO
O
O
24
9
8
7
6
5
4
3
2
1
0
ppm
4.58
1.99
0.45
0.47
0.52
0.46
3.82
1.94
0.90
0.94
1.37
1.48
2.91
156.04
155.90
136.53
128.51
128.06
127.90
109.51
80.28
80.12
77.32
77.07
76.81
75.28
75.21
67.30
66.32
66.26
64.10
63.90
43.46
43.31
41.93
41.78
27.31
26.18
Current Data Parameters
NAME 500-2019
EXPNO 89
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190110
Time 21.27 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cbz
N
HO
HO
O
O
24
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

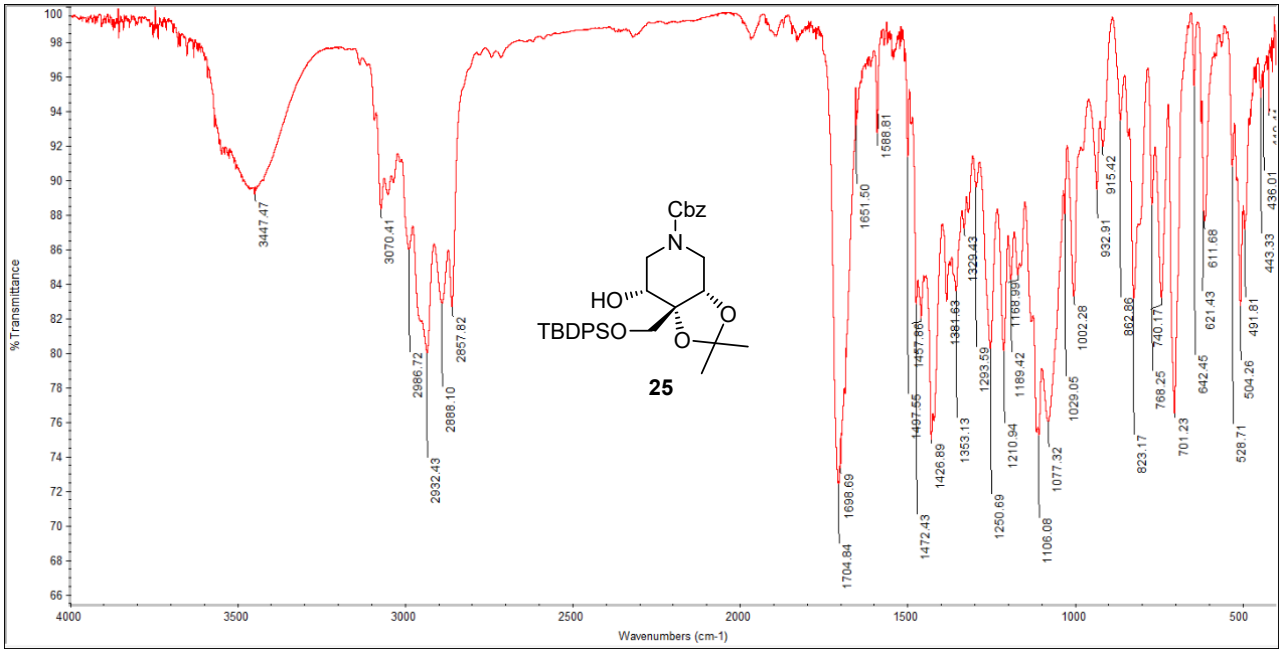

128.51
128.06
127.91
75.28
75.21
67.30
66.32
66.26
64.10
63.90
43.47
43.31
41.93
41.78
27.31
26.18
Cbz
N
HO
HO
O
O
24
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


% Transmittance
3443.30
2988.84
2939.55
2889.62
2360.52
2339.92
1684.30
1540.29
1498.13
1427.17
1380.73
1356.74
1250.56
1212.35
1179.64
1131.27
1083.89
1054.67
1004.22
932.54
860.50
838.61
767.61
737.22
698.43
610.64
531.30
Cbz
N
HO
HO
O
O
24
Wavenumbers (cm-1)

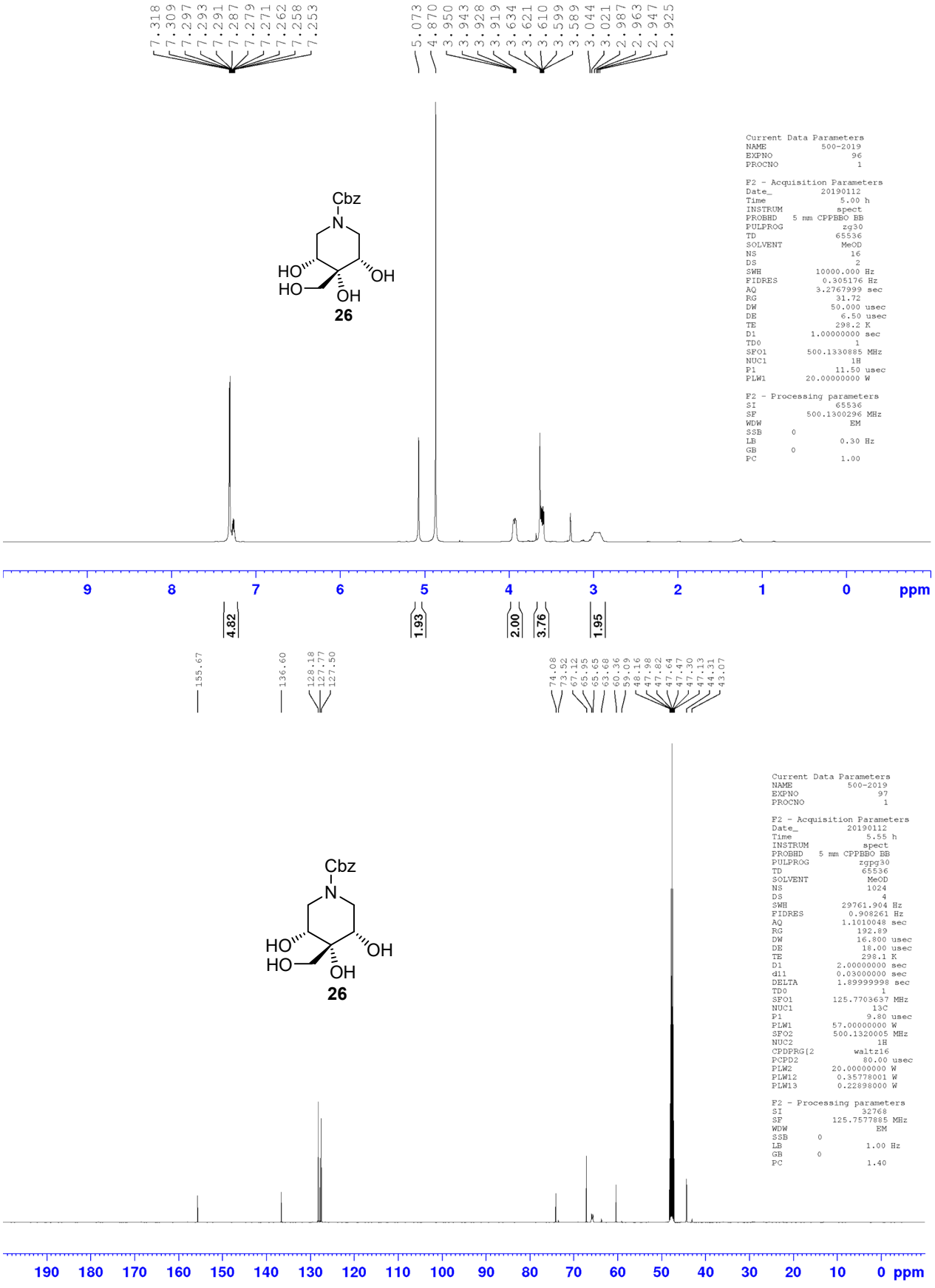
Compound 25:

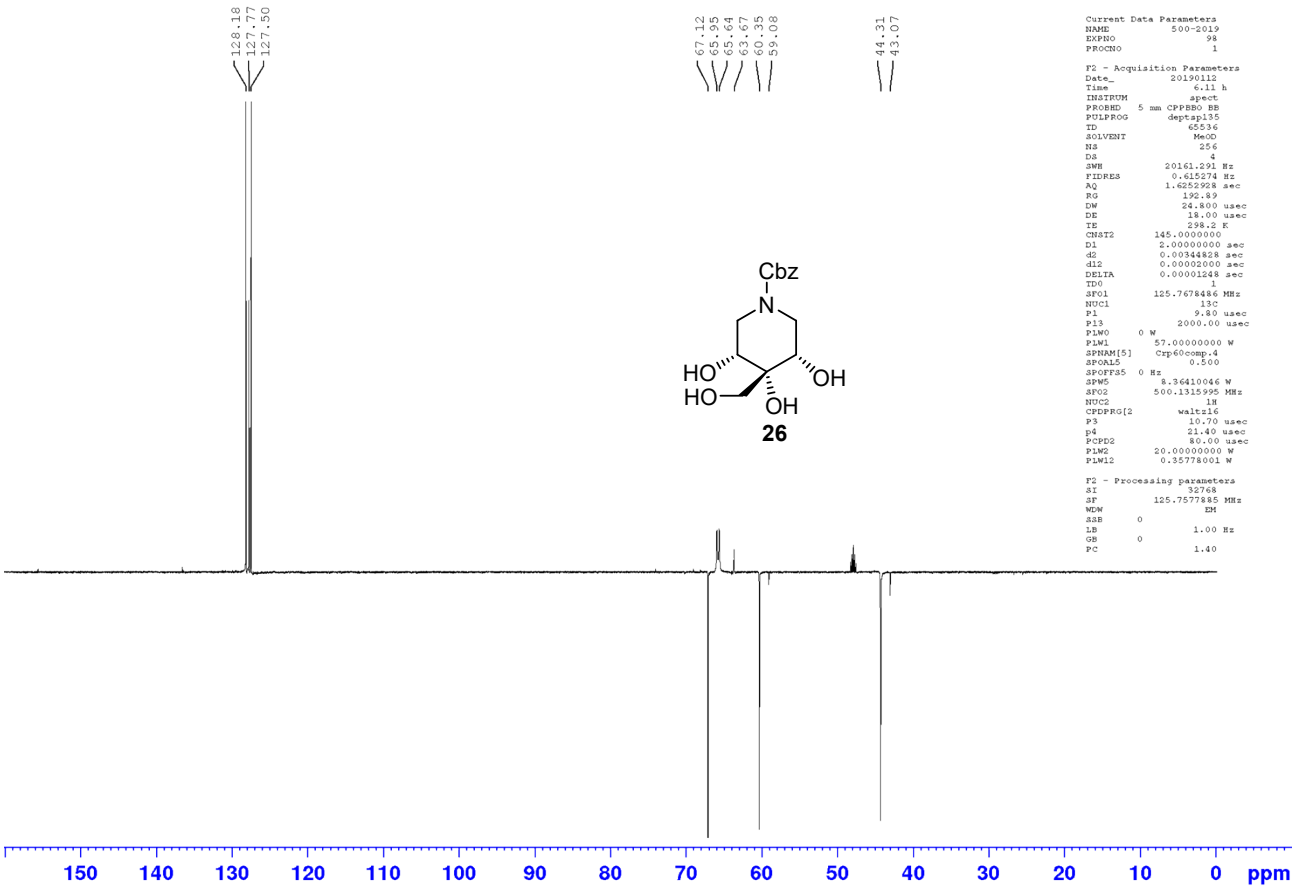

7.659 7.657 7.653 7.650 7.647 7.644 7.466 7.464 7.459 7.452 7.447 7.440 7.437 7.435 7.425 7.419 7.404 7.391 7.373 7.361 7.345 7.327 7.322 7.317 7.313 7.260 5.187 5.177 5.165 5.160 5.135 4.338 4.309 4.160 4.154 4.034 4.024 3.990 3.970 3.605 3.602 3.585 3.582 3.256 3.253 3.227 3.224 3.194 3.171 3.164 2.219 2.215 2.198 2.195 1.469 1.444 1.271 1.064
Current Data Parameters
NAME 500-2019
EXPNO 213
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190621
Time 15.34 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cbz
N
HO
TBDPSO
O
O
25
9 8 7 6 5 4 3 2 1 0 ppm
4.09 11.31 2.09 0.55 0.43 0.55 0.45 2.02 0.47 0.56 1.00 0.46 0.56 1.02 0.97 1.39 1.63 3.19 9.45
156.02 155.84 136.72 135.65 135.57 135.55 132.72 132.64 132.34 132.30 130.09 130.06 128.49 127.99 127.96 127.93 127.89 109.27 79.97 79.78 77.30 77.05 76.80 76.28 76.21 67.19 65.74 65.58 65.52 65.45 43.41 43.30 42.37 42.20 27.14 26.91 25.94 19.22
Current Data Parameters
NAME 500-2019
EXPNO 214
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190621
Time 15.38 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 412
DS 4
SWH 37878.789 Hz
FIDRES 1.155969 Hz
AQ 0.8650752 sec
RG 192.89
DW 13.200 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cbz
N
HO
TBDPSO
O
O
25
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

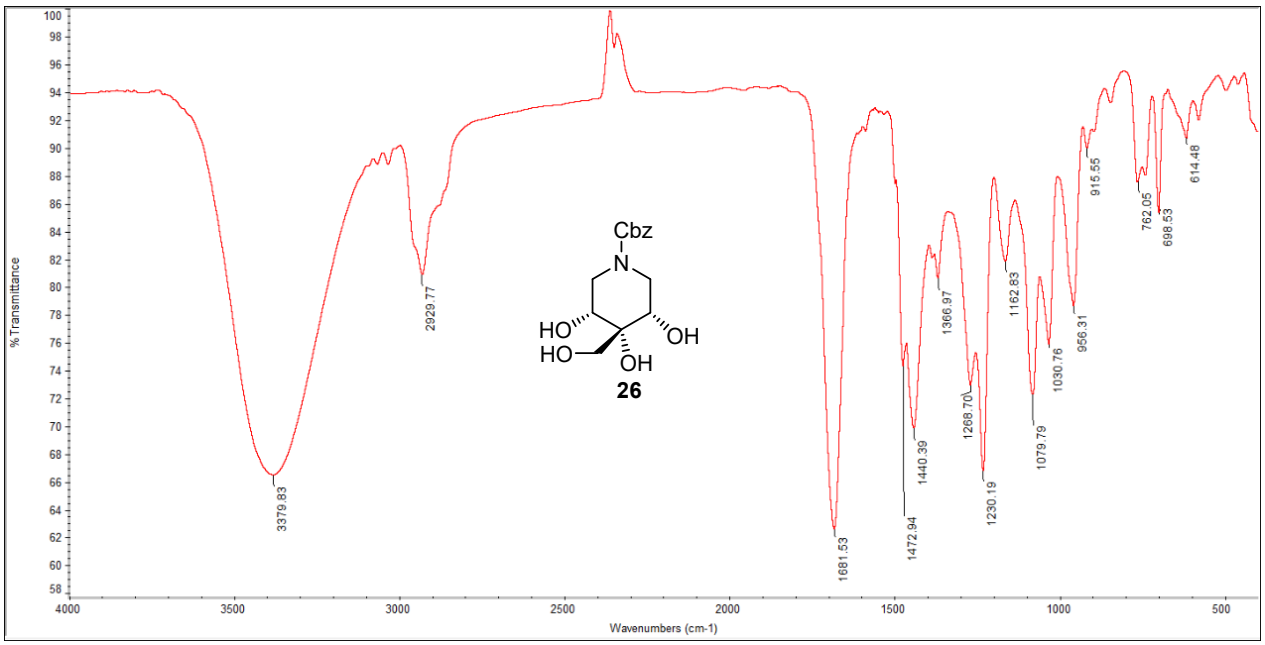

135.65
135.57
135.55
130.09
130.06
128.49
127.99
127.96
127.93
127.89
109.27
76.28
76.21
67.19
65.74
65.58
65.52
65.45
43.41
43.30
42.37
42.20
27.14
26.91
25.94
Cbz
N
HO
TBDPSO
O
O
25
Current Data Parameters
NAME 500-2019
EXPNO 215
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190621
Time 16.27 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptap135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 1.2976128 sec
RG 192.89
DW 19.800 usec
DE 18.00 usec
TE 298.1 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


% Transmittance
Wavenumbers (cm-1)
3447.47
3070.41
2986.72
2932.43
2888.10
2857.82
1704.84
1698.69
1651.50
1588.81
1497.55
1472.43
1457.86
1426.89
1381.63
1353.13
1329.43
1293.59
1250.69
1210.94
1189.42
1168.99
1106.08
1077.32
1029.05
1002.28
932.91
915.42
862.86
823.17
768.25
740.17
701.23
642.45
621.43
611.68
528.71
504.26
491.81
443.33
436.01
Cbz
N
HO
TBDPSO
O
O
25

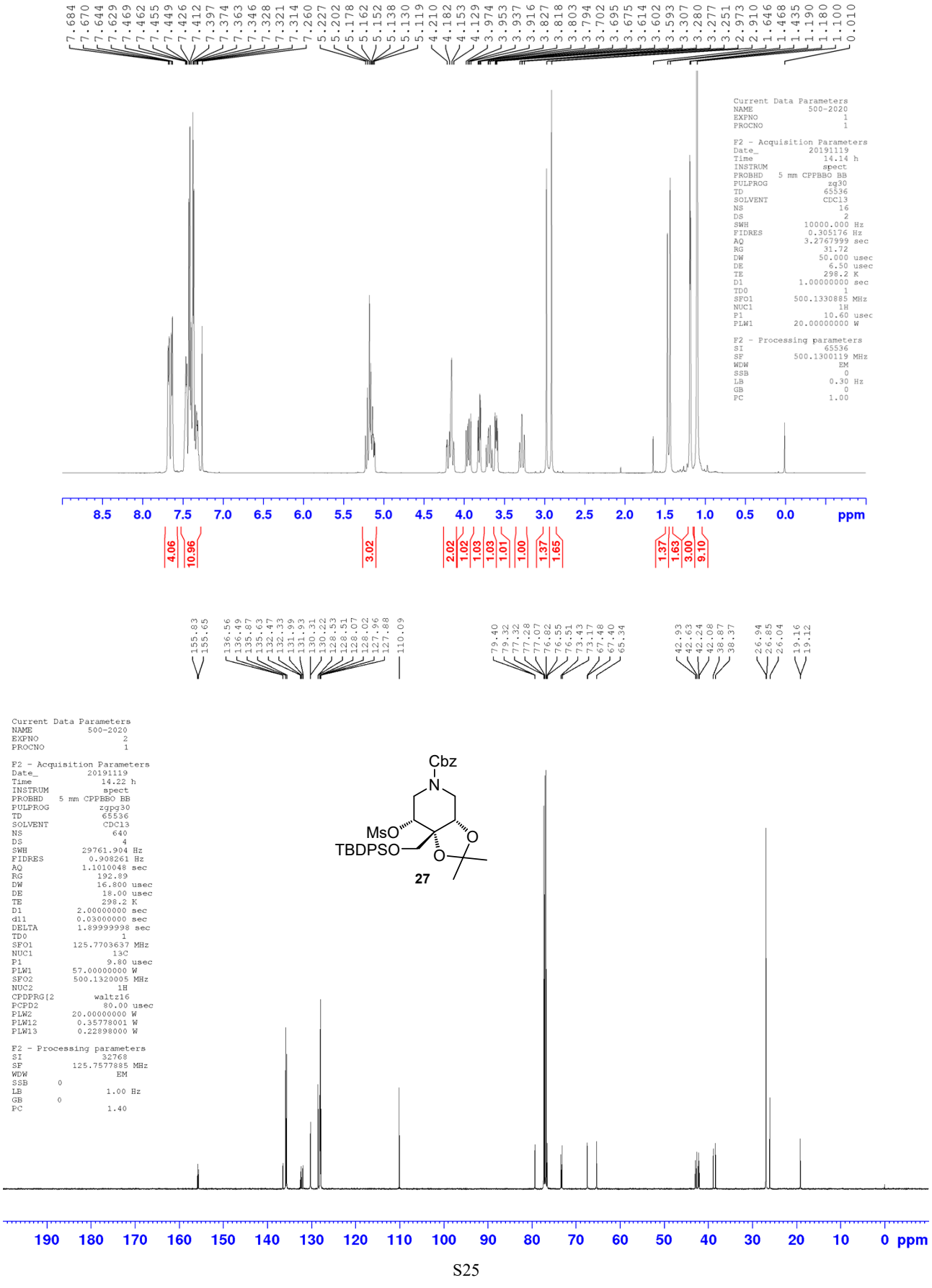
Compound 26**:**

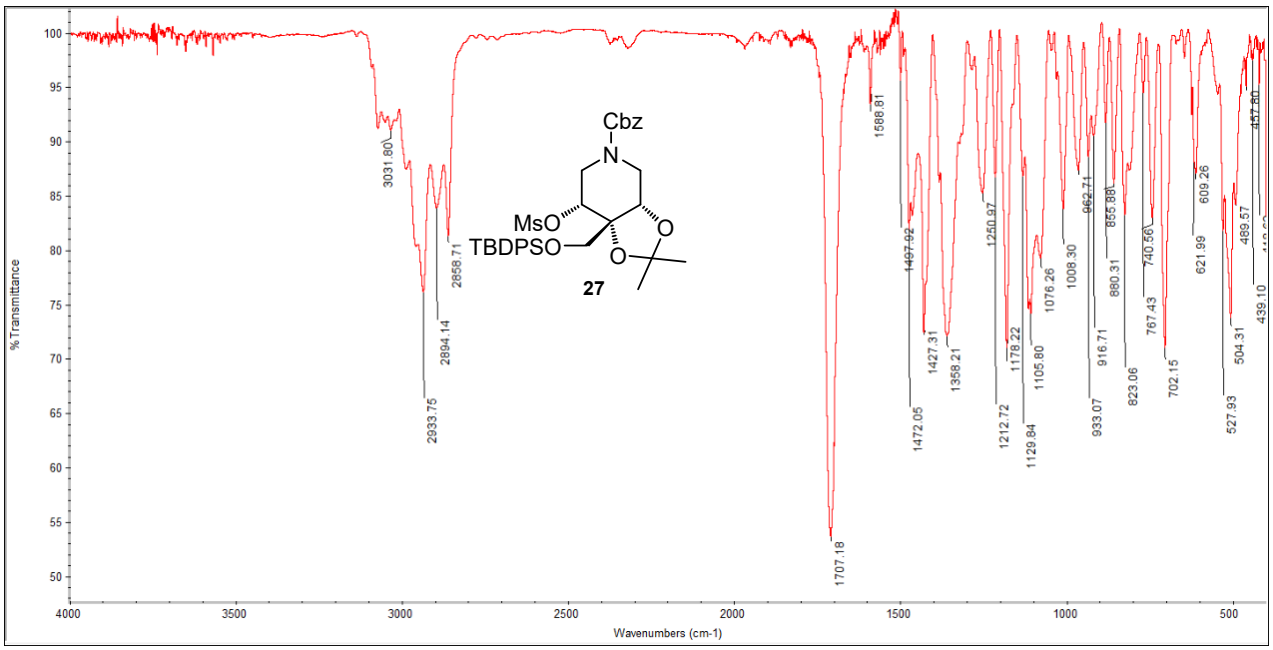

7.318
7.309
7.297
7.293
7.291
7.287
7.279
7.271
7.262
7.258
7.253
5.073
4.870
3.950
3.943
3.928
3.919
3.634
3.621
3.610
3.599
3.589
3.044
3.021
2.987
2.963
2.947
2.925
Cbz
N
HO
HO
OH
OH
26
Current Data Parameters
NAME 500-2019
EXPNO 96
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190112
Time 5.00 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300296 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
4.82
1.93
2.00
3.76
1.95
155.67
136.60
128.18
127.77
127.50
74.08
73.52
67.12
65.95
65.65
63.68
60.36
59.09
48.16
47.98
47.82
47.64
47.47
47.30
47.13
44.31
43.07
Cbz
N
HO
HO
OH
OH
26
Current Data Parameters
NAME 500-2019
EXPNO 97
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190112
Time 5.55 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


128.18
127.77
127.50
67.12
65.95
65.64
63.67
60.35
59.08
44.31
43.07
Cbz
N
HO
HO
OH
OH
26
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

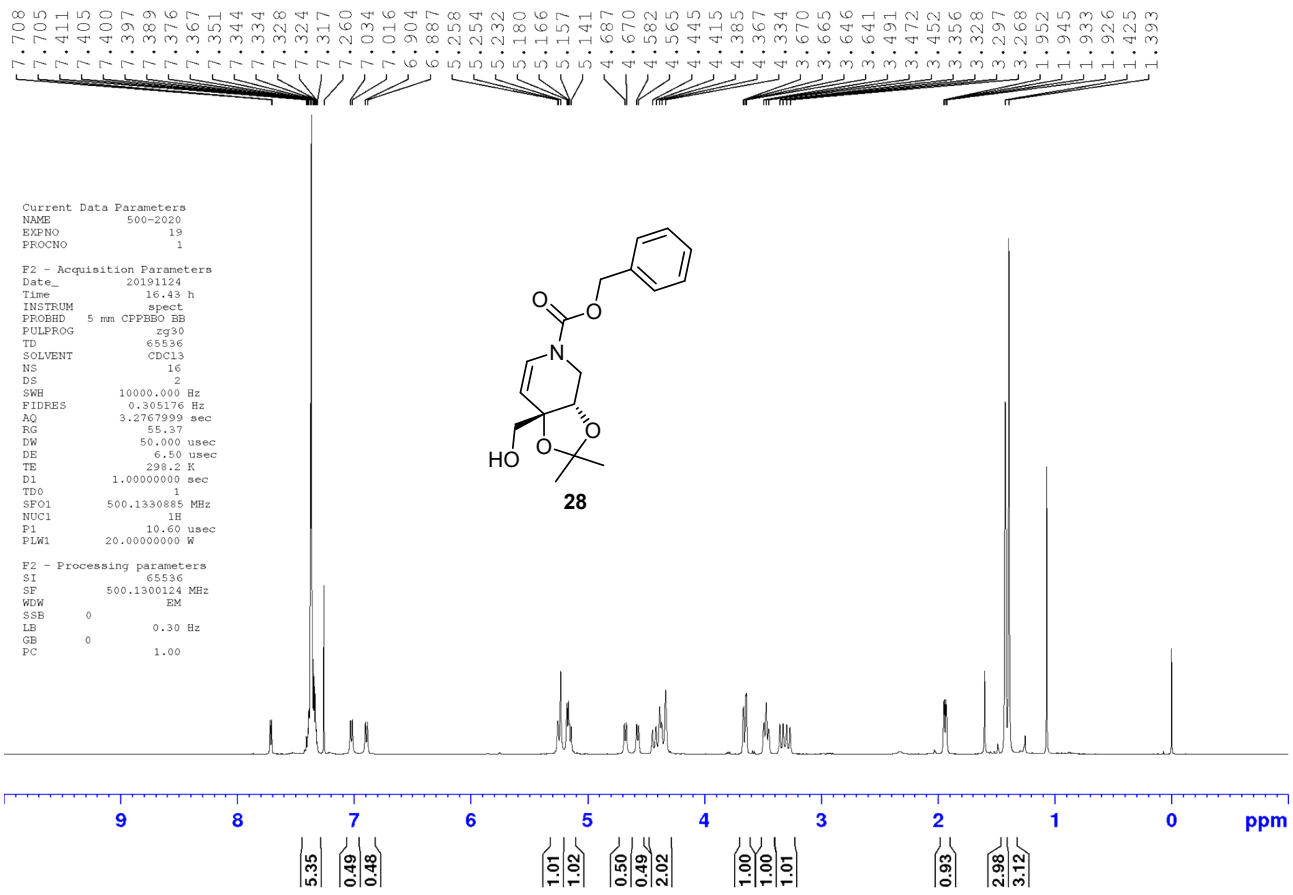

% Transmittance
3379.83
2929.77
1681.53
1472.94
1440.39
1366.97
1268.70
1230.19
1162.83
1079.79
1030.76
956.31
915.55
762.05
698.53
614.48
Cbz
N
HO
HO
OH
OH
26
Wavenumbers (cm-1)

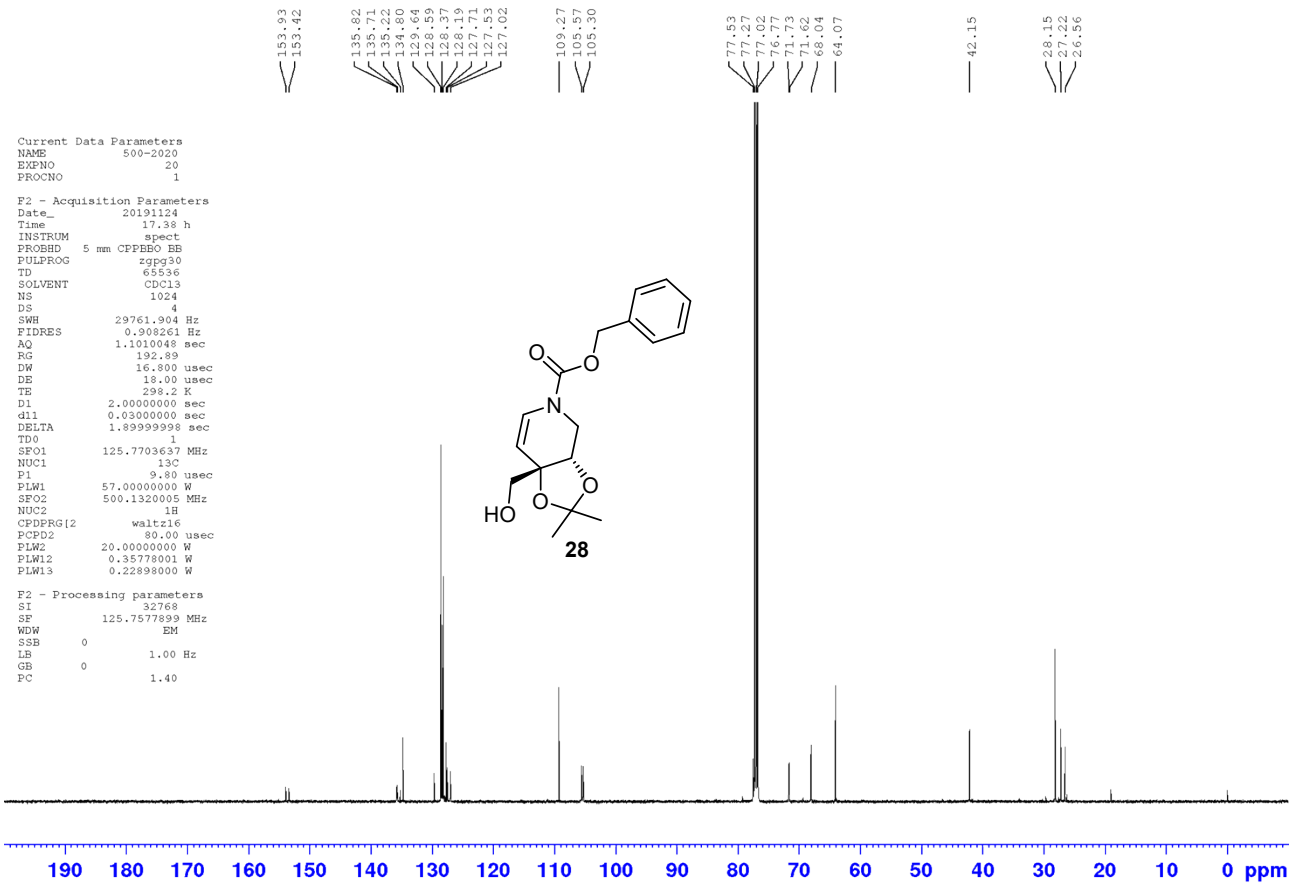
Compound 27:

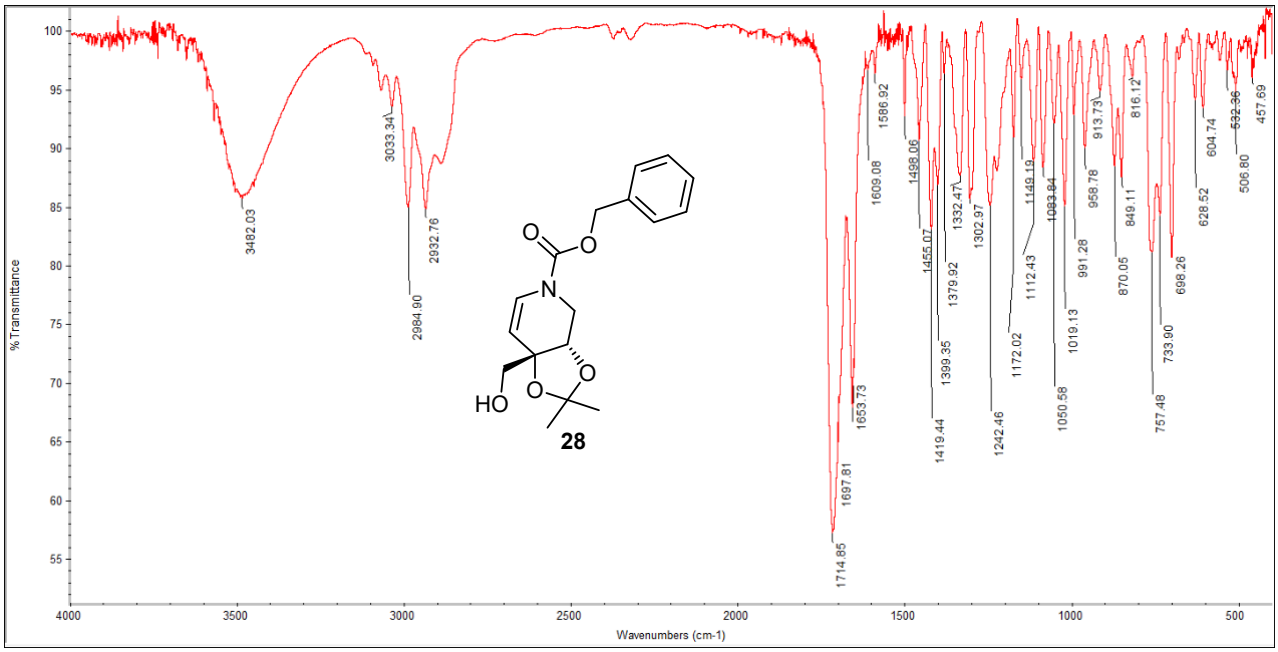

Current Data Parameters
NAME 500-2020
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191119
Time 14.14 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
Current Data Parameters
NAME 500-2020
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191119
Time 14.22 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 640
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cbz
N
MsO
TBDPSO
O
O
27
ppm


%Transmittance
Wavenumbers (cm-1)
3031.80
2933.75
2884.14
2858.71
1707.18
1588.81
1472.05
1427.31
1358.21
1250.97
1212.72
1178.22
1129.84
1105.80
1076.26
1008.30
962.71
933.07
916.71
880.31
855.88
823.06
767.43
740.56
702.15
621.99
609.26
527.93
504.31
489.57
439.10
Cbz
N
MsO
TBDPSO
O
O
27

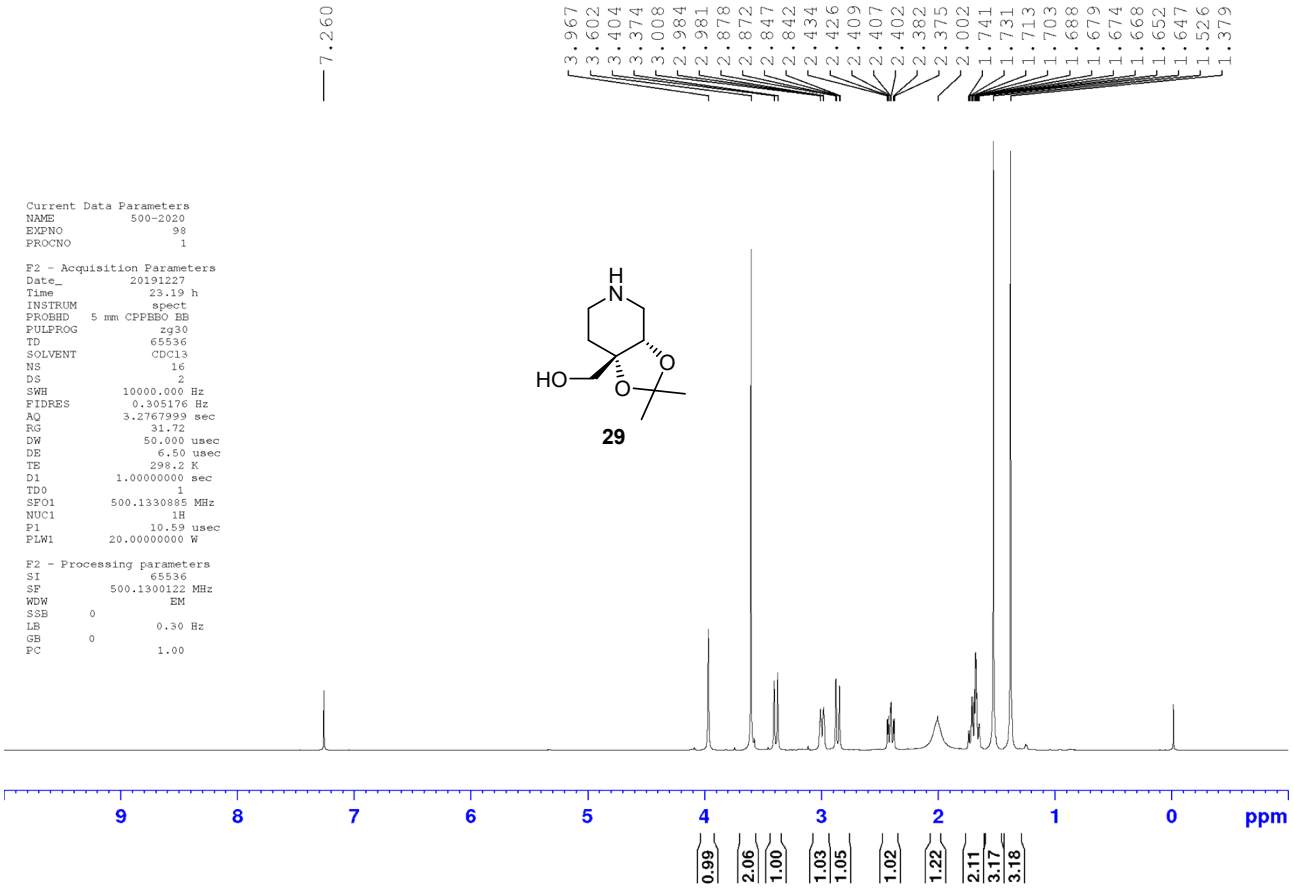
Compound 28**:**

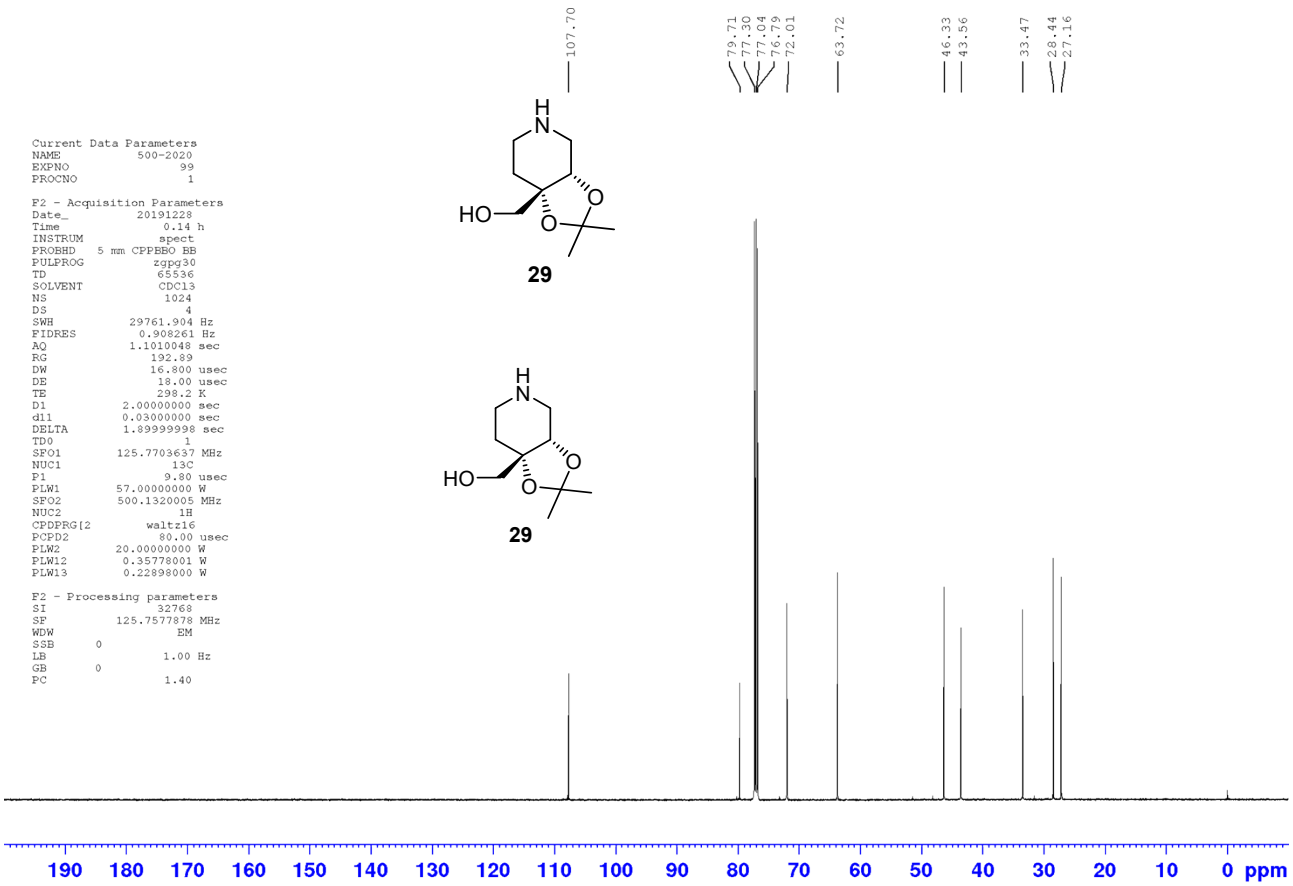

7.708
7.705
7.411
7.405
7.400
7.397
7.389
7.376
7.367
7.351
7.344
7.334
7.328
7.324
7.317
7.260
7.034
7.016
6.904
6.887
5.258
5.254
5.232
5.180
5.166
5.157
5.141
4.687
4.670
4.582
4.565
4.445
4.415
4.385
4.367
4.334
3.670
3.665
3.646
3.641
3.491
3.472
3.452
3.356
3.328
3.297
3.268
1.952
1.945
1.933
1.926
1.425
1.393
Current Data Parameters
NAME 500-2020
EXPNO 19
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191124
Time 16.43 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 55.37
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
N
O
O
HO
28
9
8
7
6
5
4
3
2
1
0
ppm
5.35
0.49
0.48
1.01
1.02
0.50
0.49
2.02
1.00
1.00
1.01
0.93
2.98
3.12

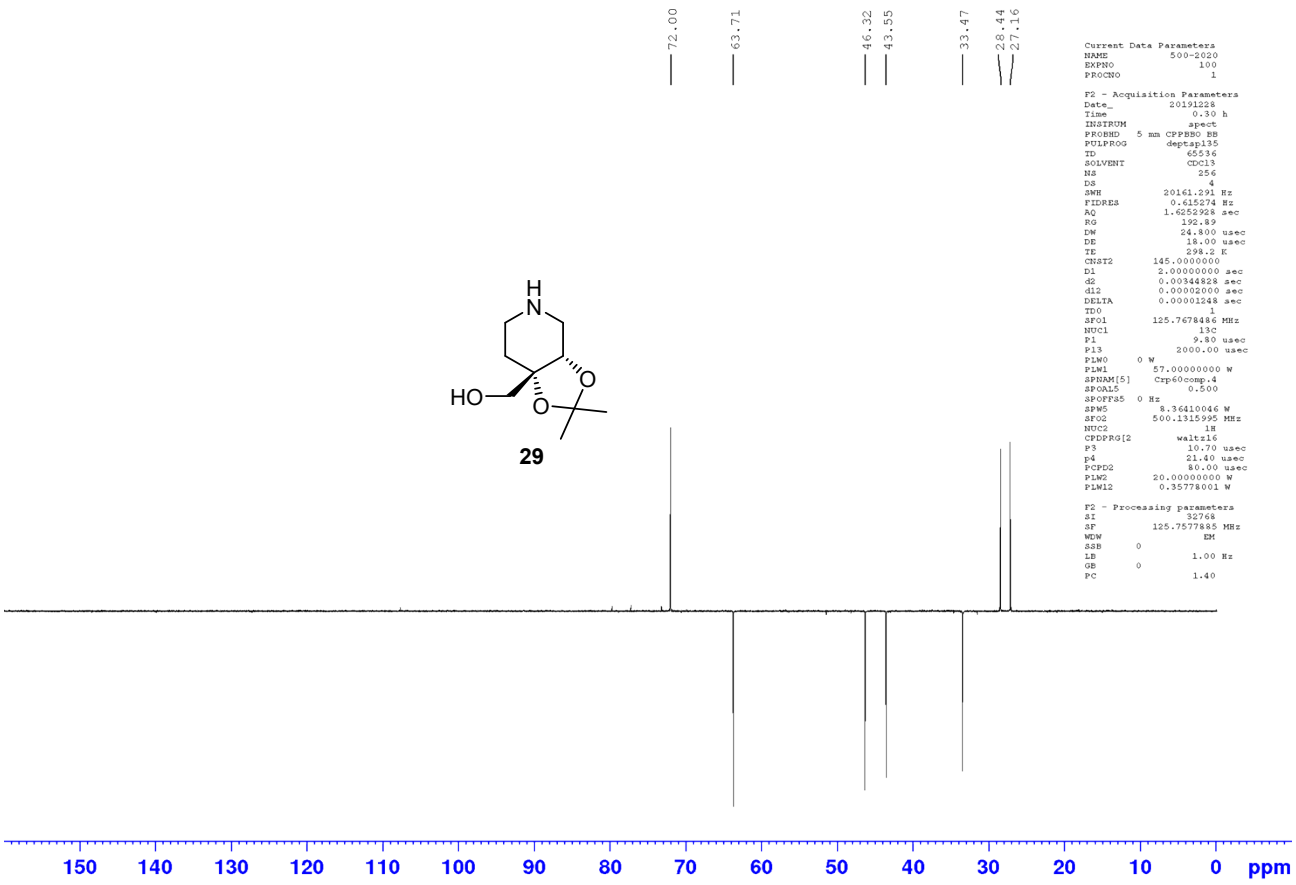

153.93
153.42
135.82
135.71
135.22
134.80
129.64
128.59
128.37
128.19
127.71
127.53
127.02
109.27
105.57
105.30
77.53
77.27
77.02
76.77
71.73
71.62
68.04
64.07
42.15
28.15
27.22
26.56
Current Data Parameters
NAME 500-2020
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191124
Time 17.38 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577899 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
N
O
O
HO
28
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

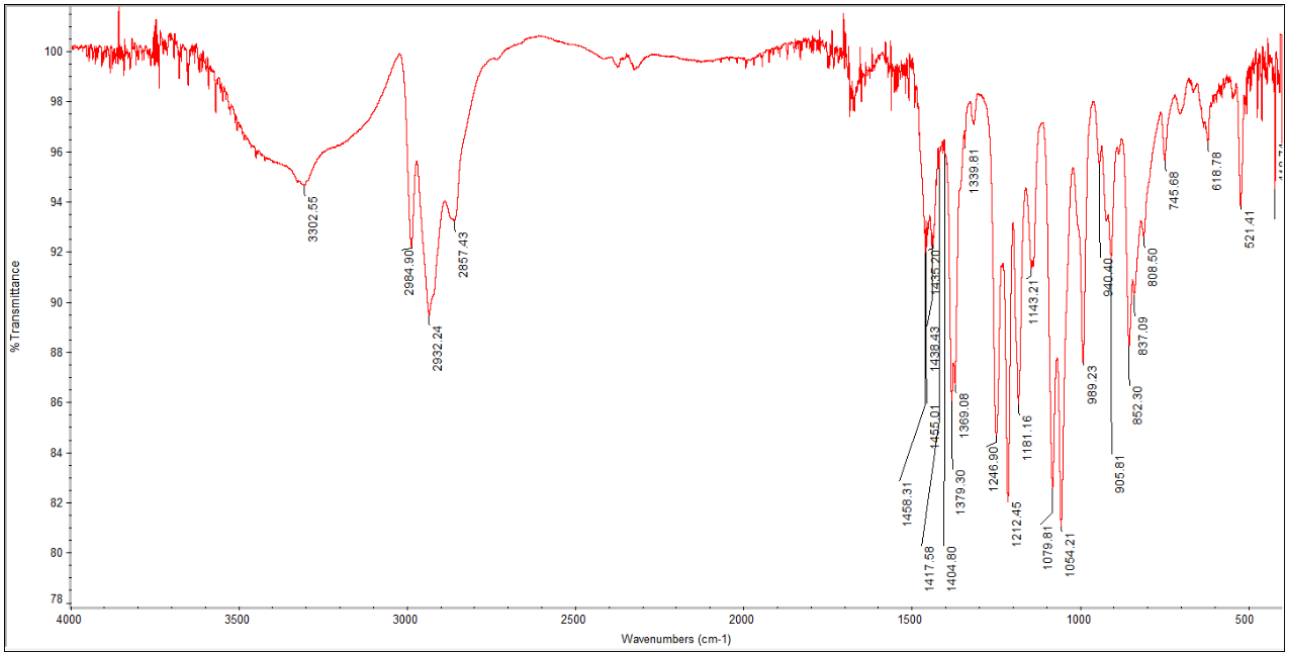

% Transmittance
Wavenumbers (cm-1)
3482.03
3033.34
2984.90
2932.76
1714.85
1697.81
1653.73
1609.08
1586.92
1498.06
1455.07
1419.44
1399.35
1379.92
1332.47
1302.97
1242.46
1172.02
1149.19
1112.43
1083.84
1050.58
1019.13
991.28
958.78
913.79
870.05
849.11
816.12
757.48
733.90
698.26
628.52
604.74
532.36
506.80
457.69
O
O
N
O
O
HO
28

Compound 29:

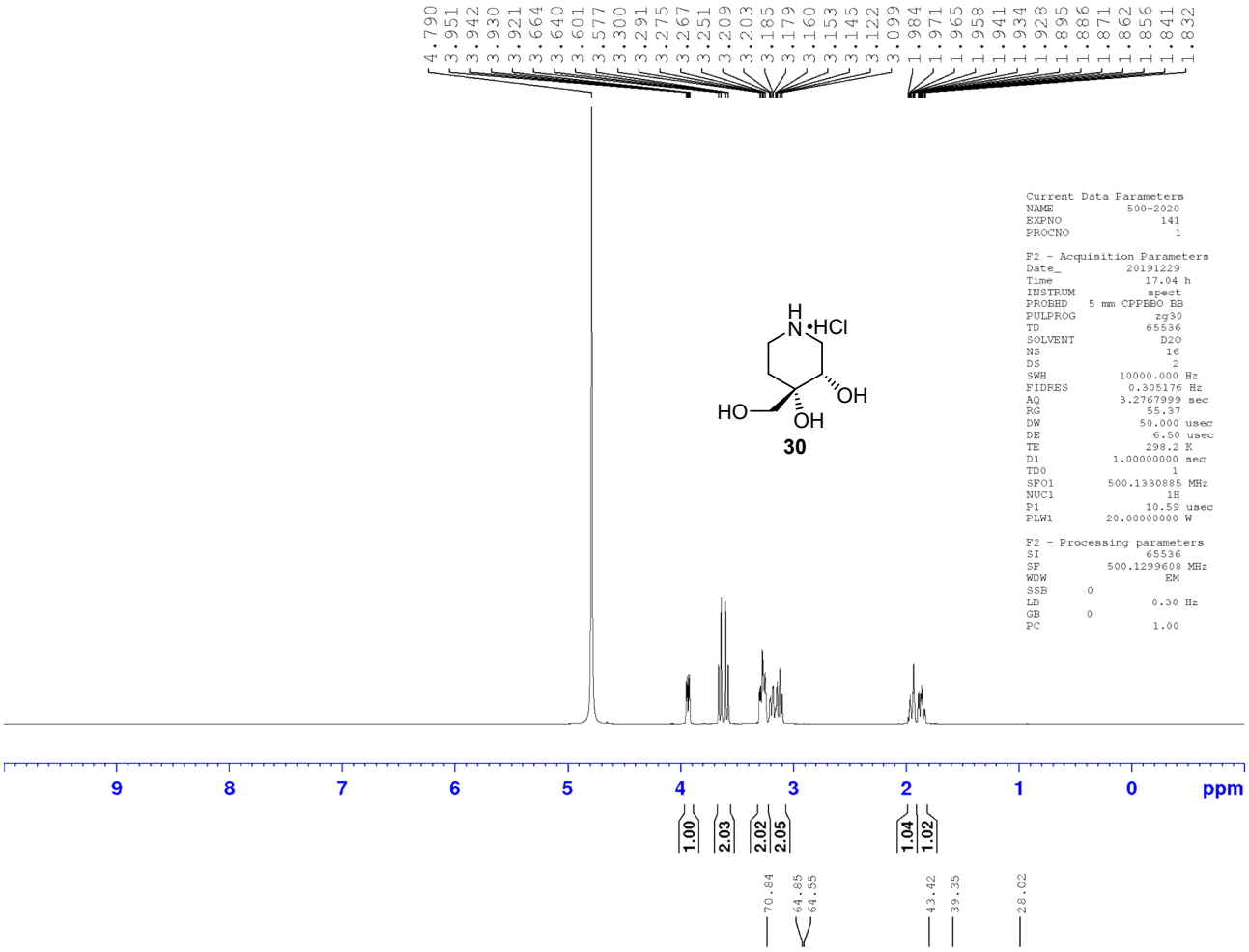

7.260
3.967
3.602
3.404
3.374
3.008
2.984
2.981
2.878
2.872
2.847
2.842
2.434
2.426
2.409
2.407
2.402
2.382
2.375
2.002
1.741
1.731
1.713
1.703
1.688
1.679
1.674
1.668
1.652
1.647
1.526
1.379
Current Data Parameters
NAME 500-2020
EXPNO 98
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191227
Time 23.19 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.59 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
N
HO
O
O
29
9
8
7
6
5
4
3
2
1
0
ppm
0.99
2.06
1.00
1.03
1.05
1.02
1.22
2.11
3.17
3.18

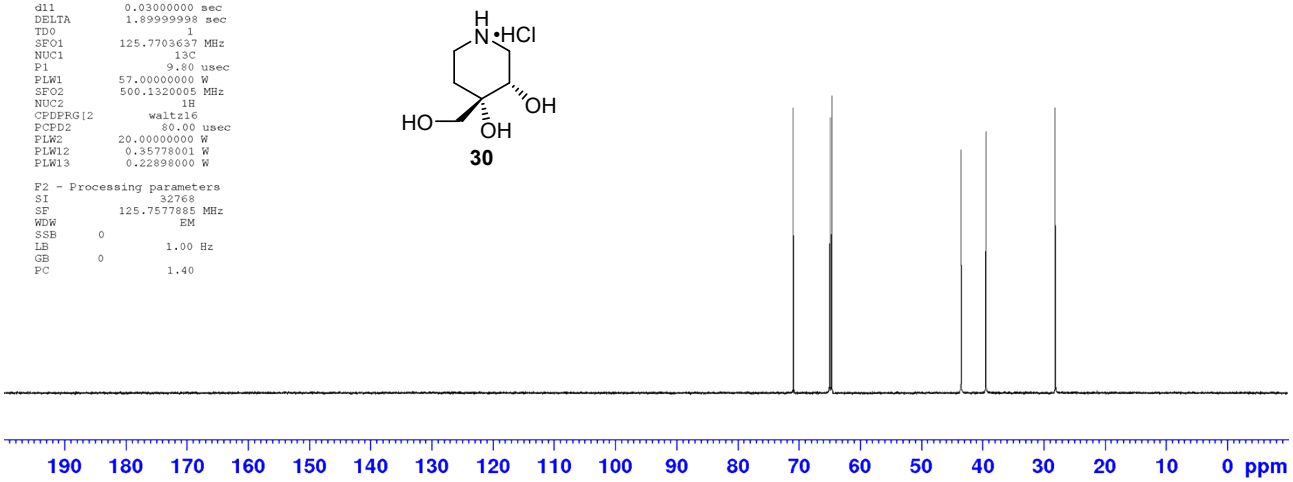

107.70
79.71
77.30
77.04
76.79
72.01
63.72
46.33
43.56
33.47
28.44
27.16
Current Data Parameters
NAME 500-2020
EXPNO 99
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191228
Time 0.14 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577878 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H
N
HO
O
O
29
H
N
HO
O
O
29
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

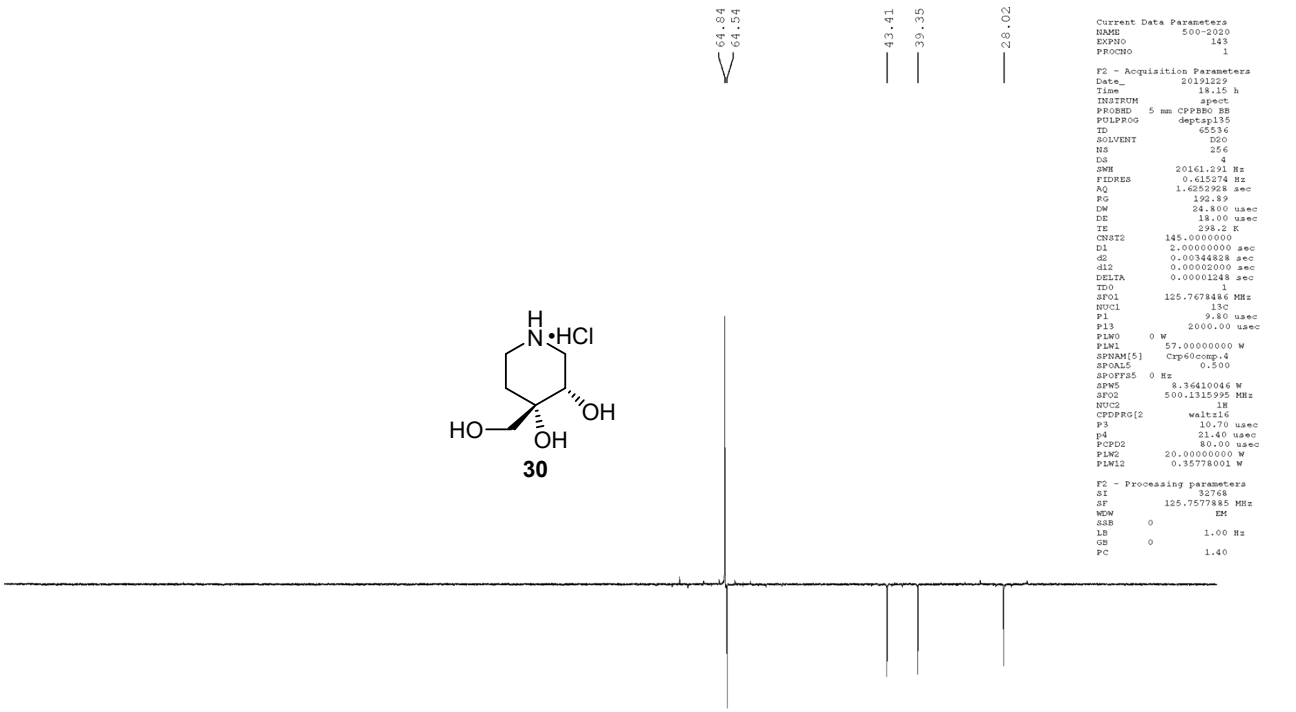

72.00
63.71
46.32
43.55
33.47
28.44
27.16
HO
O
O
H
N
29
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

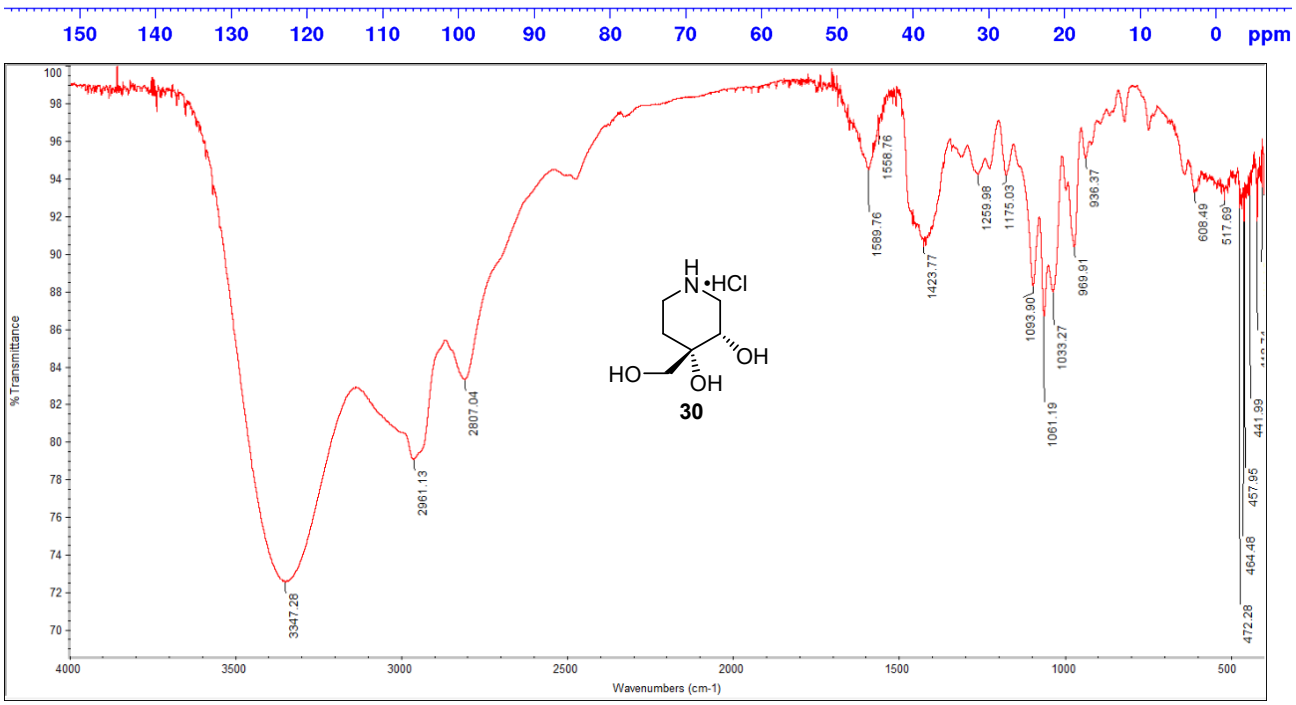

% Transmittance
Wavenumbers (cm-1)
3302.55
2984.90
2932.24
2857.43
1458.31
1455.01
1438.43
1435.20
1417.58
1404.80
1379.30
1369.08
1339.81
1246.90
1212.45
1181.16
1143.21
1079.81
1054.21
989.23
940.40
905.81
852.30
837.09
808.50
745.68
618.78
521.41

Compound 30**:**


4.790
3.951
3.942
3.930
3.921
3.664
3.640
3.601
3.577
3.300
3.291
3.275
3.267
3.251
3.209
3.203
3.185
3.179
3.160
3.153
3.145
3.122
3.099
1.984
1.971
1.965
1.958
1.941
1.934
1.928
1.895
1.886
1.871
1.862
1.856
1.841
1.832
Current Data Parameters
NAME 500-2020
EXPNO 141
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191229
Time 17.04 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT D2O
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 55.37
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.59 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1299608 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
N•HCl
HO
OH
OH
30
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.03
2.02
2.05
1.04
1.02
70.84
64.85
64.55
43.42
39.35
28.02

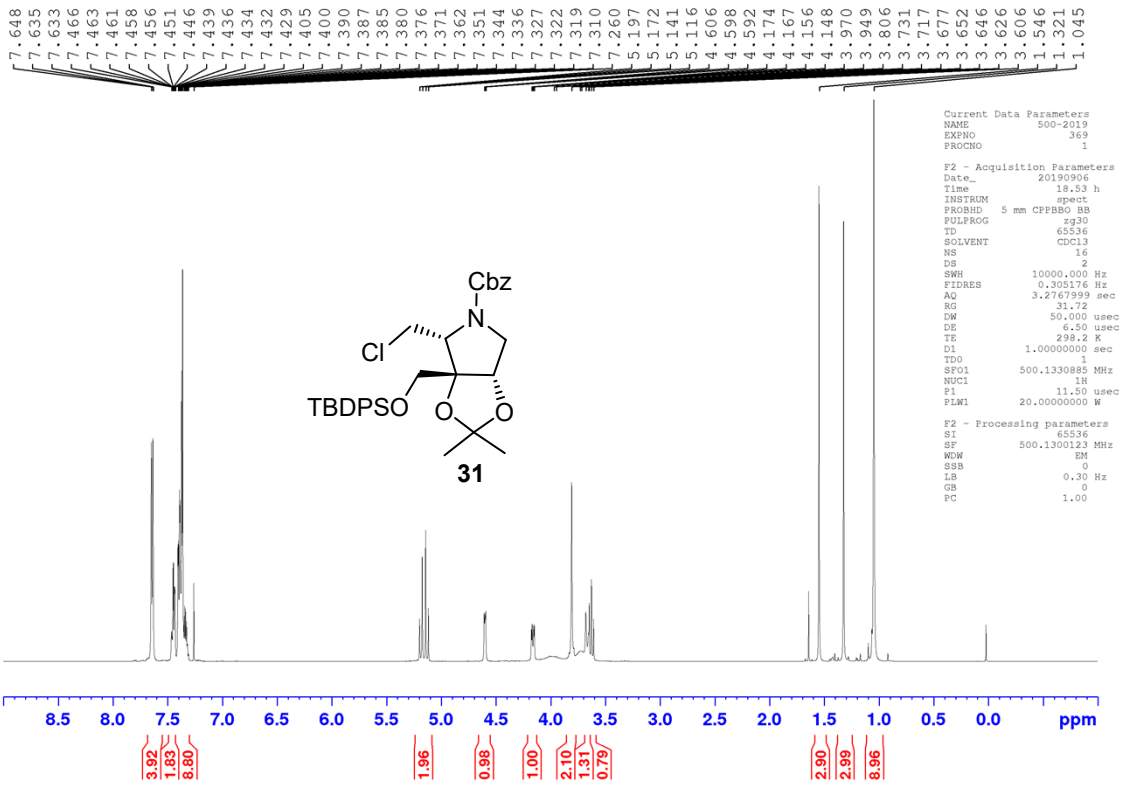

Current Data Parameters
NAME 500-2020
EXPNO 142
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191229
Time 17.59 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
H
N•HCl
HO
OH
OH
30
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

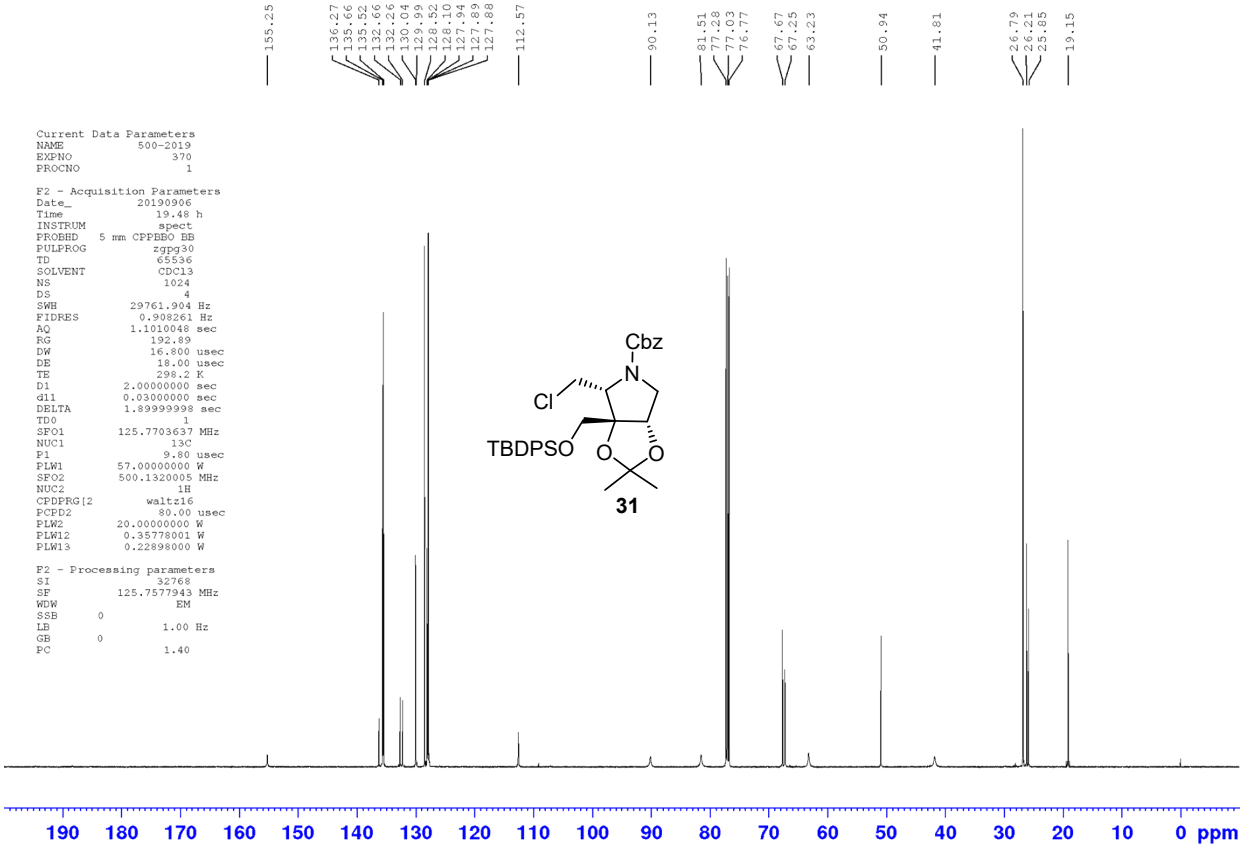

64.84
64.54
43.41
39.35
28.02
30

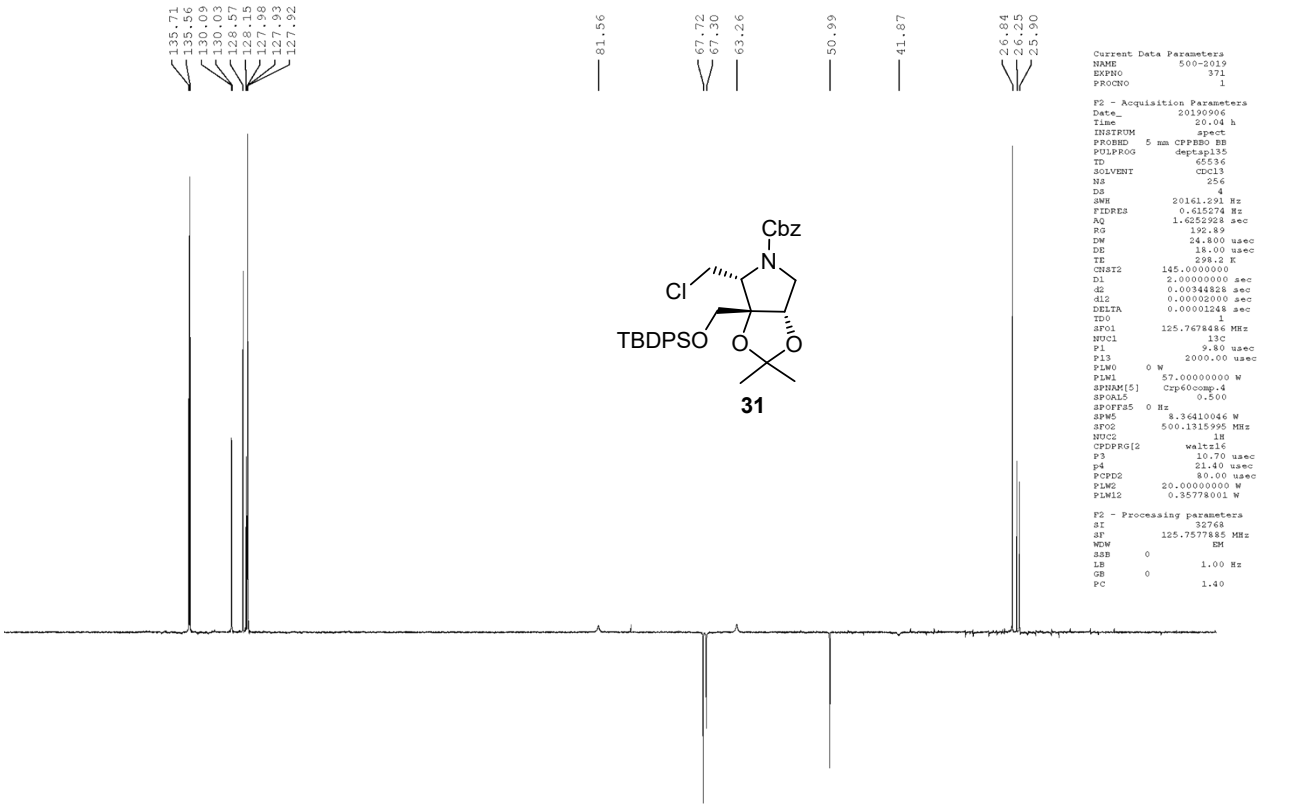

150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

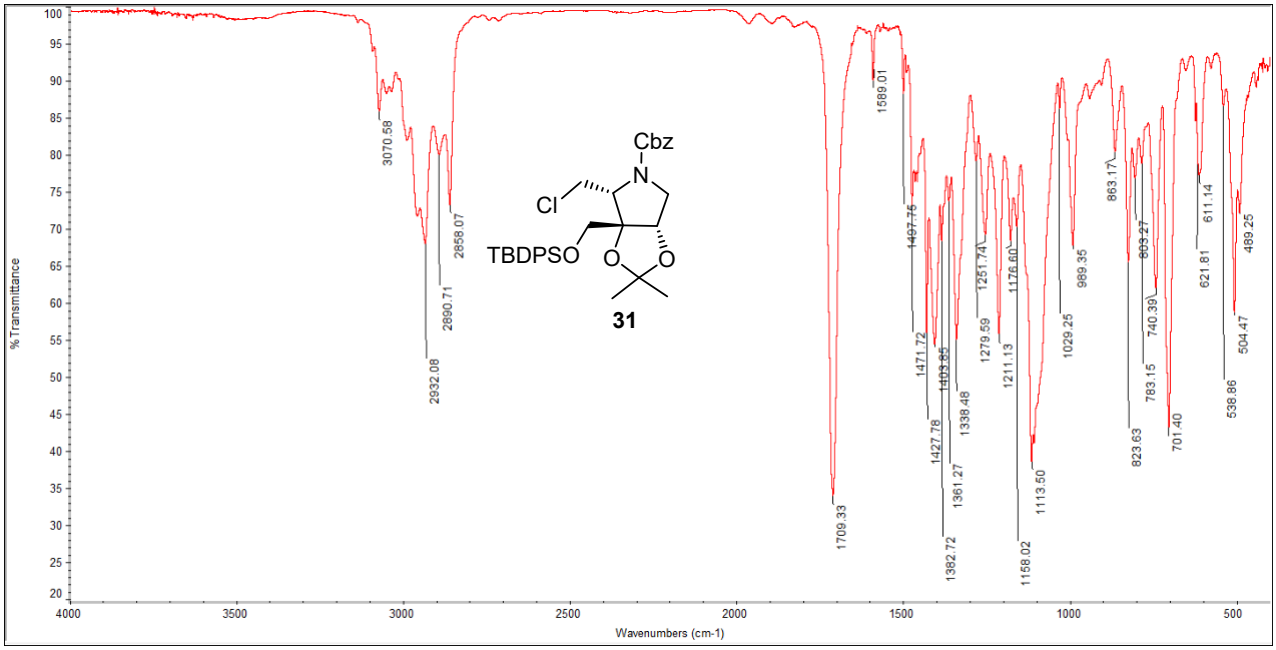
% Transmittance
Wavenumbers (cm-1)
3347.28
2961.13
2807.04
1589.76
1558.76
1423.77
1259.98
1175.03
1093.90
1061.19
1033.27
969.91
936.37
608.49
517.69
472.28
464.48
457.95
441.99
30

Compound 31**:**

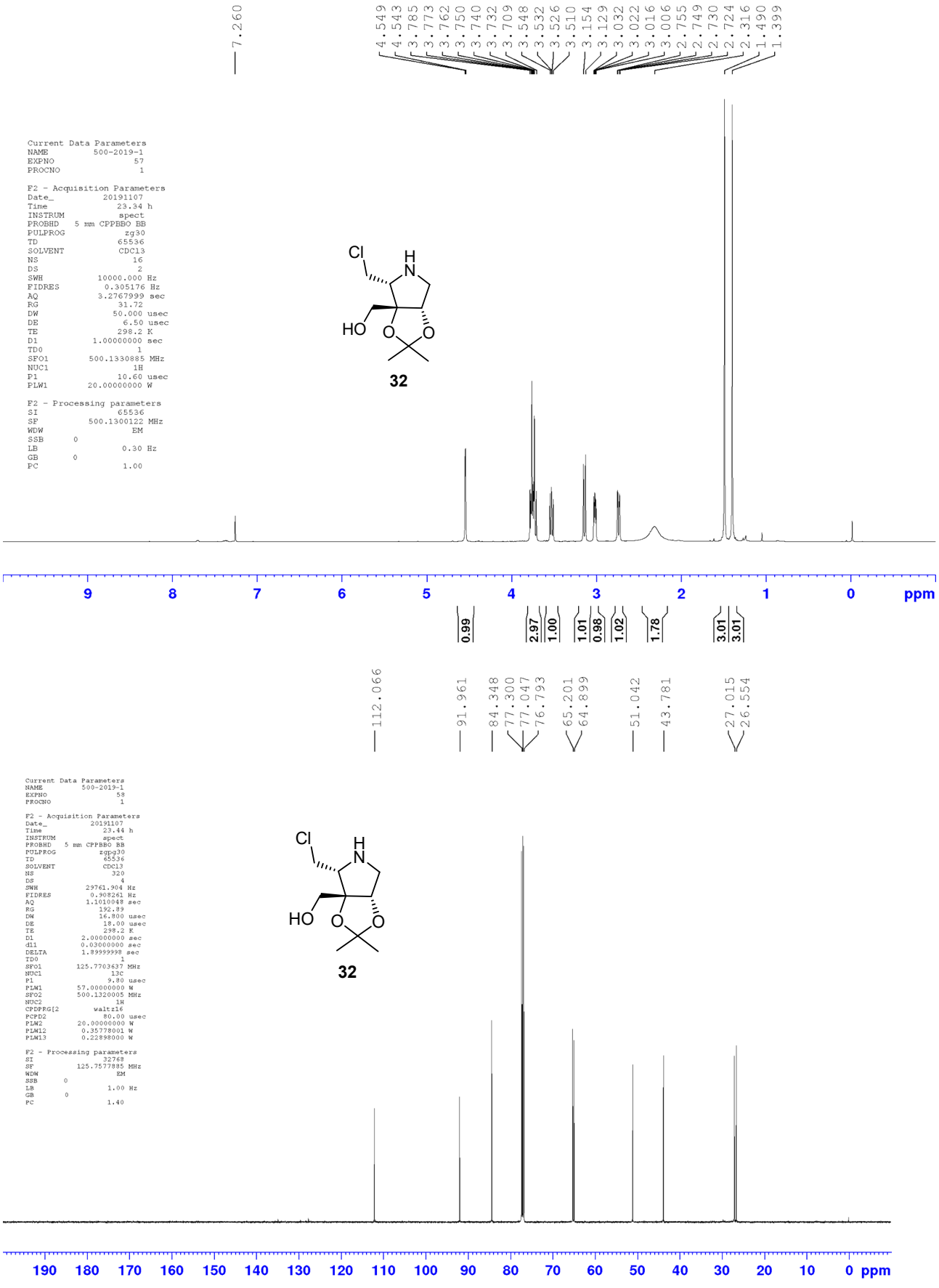

7.648
7.635
7.633
7.466
7.463
7.461
7.458
7.456
7.451
7.446
7.439
7.436
7.434
7.432
7.429
7.405
7.400
7.390
7.387
7.385
7.380
7.376
7.371
7.362
7.351
7.344
7.336
7.327
7.322
7.319
7.310
7.260
5.197
5.172
5.141
5.116
4.606
4.598
4.592
4.174
4.167
4.156
4.148
3.970
3.949
3.806
3.731
3.717
3.677
3.652
3.646
3.626
3.606
1.546
1.321
1.045
Cbz
N
Cl
TBDPSO
O
O
31
Current Data Parameters
NAME 500-2019
EXPNO 369
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190906
Time 18.53 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
3.92
1.83
8.80
1.96
0.98
1.00
2.10
1.31
0.79
2.90
2.99
8.96

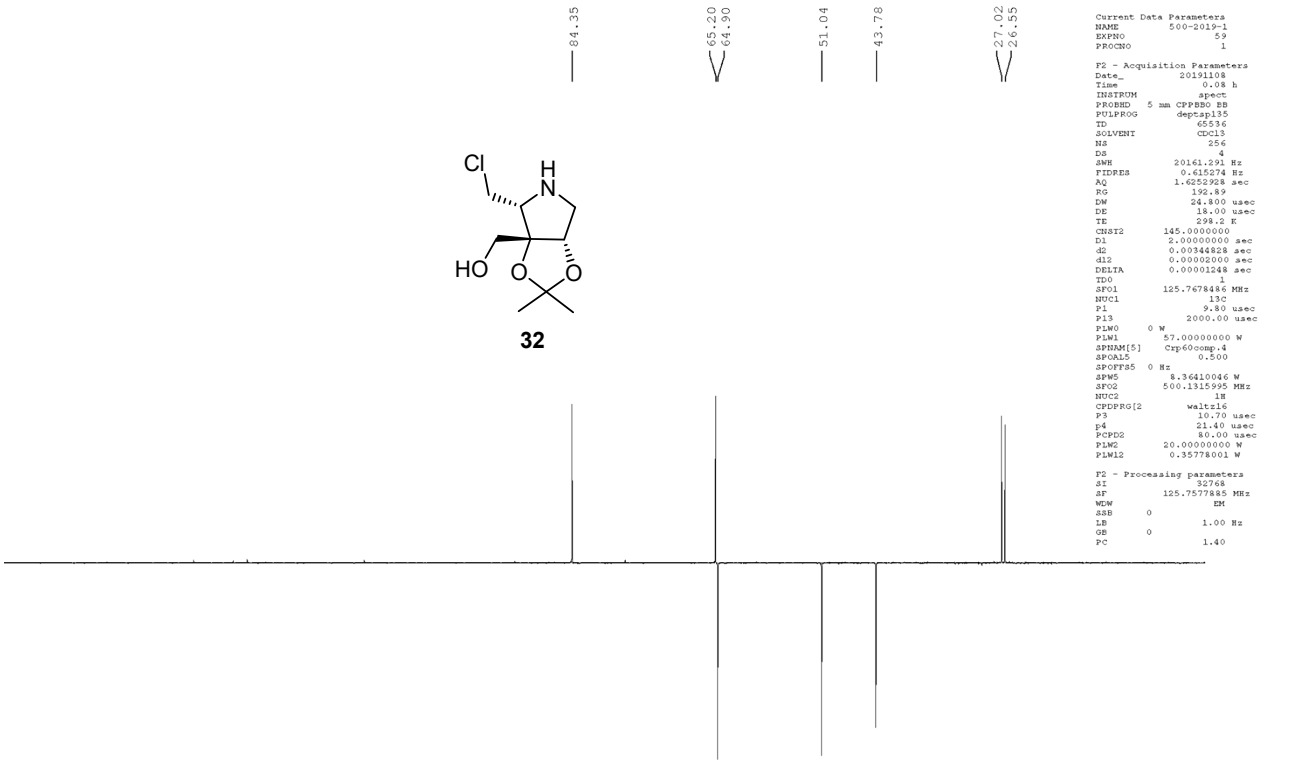

155.25
136.27
135.66
135.52
132.66
132.26
130.04
129.99
128.52
128.10
127.94
127.89
127.88
112.57
90.13
81.51
77.28
77.03
76.77
67.67
67.25
63.23
50.94
41.81
26.79
26.21
25.85
19.15
Current Data Parameters
NAME 500-2019
EXPNO 370
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190906
Time 19.48 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577943 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cbz
N
Cl
TBDPSO
O
O
31
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

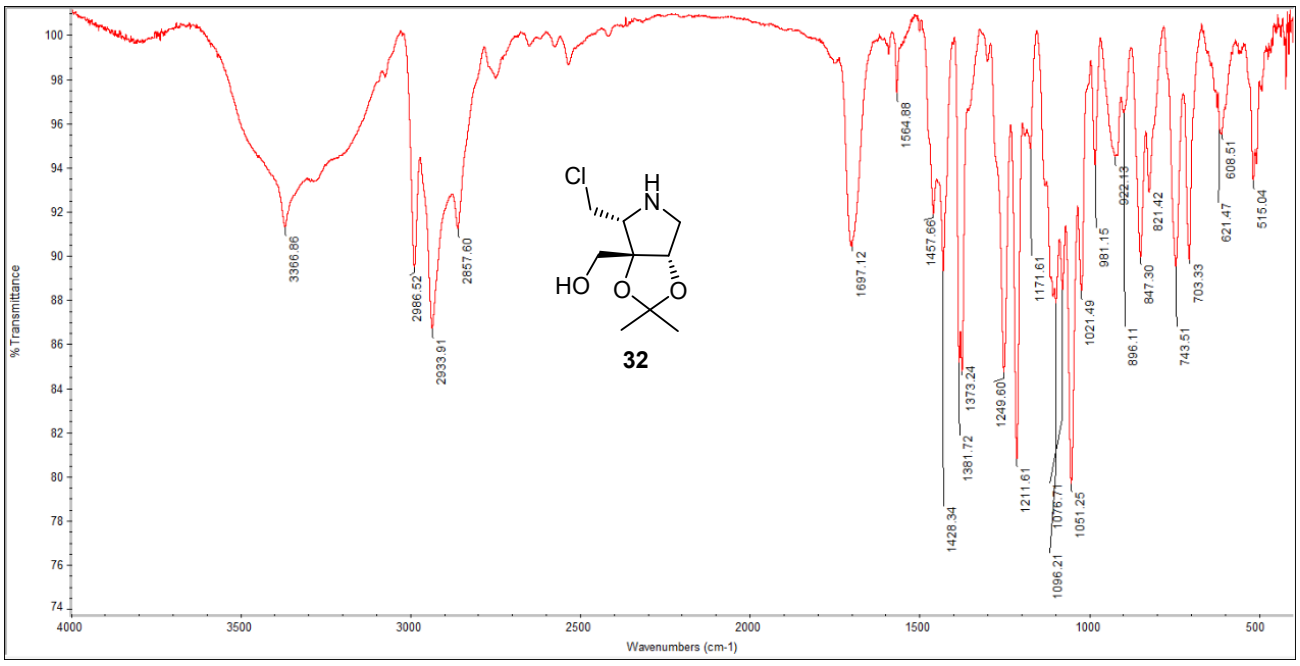

135.71
135.56
130.09
130.03
128.57
128.15
127.98
127.93
127.92
81.56
67.72
67.30
63.26
50.99
41.87
26.84
26.25
25.90
Cbz
N
Cl
TBDPSO
O
O
31
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


% Transmittance
Wavenumbers (cm-1)
3070.58
2932.08
2890.71
2858.07
1709.33
1589.01
1497.75
1471.72
1427.78
1403.85
1382.72
1361.27
1338.48
1279.59
1251.74
1211.13
1176.60
1158.02
1113.50
1029.25
989.35
863.17
823.63
809.27
783.15
740.39
701.40
621.81
611.14
538.86
504.47
489.25
Cbz
N
Cl
TBDPSO
O
O
31

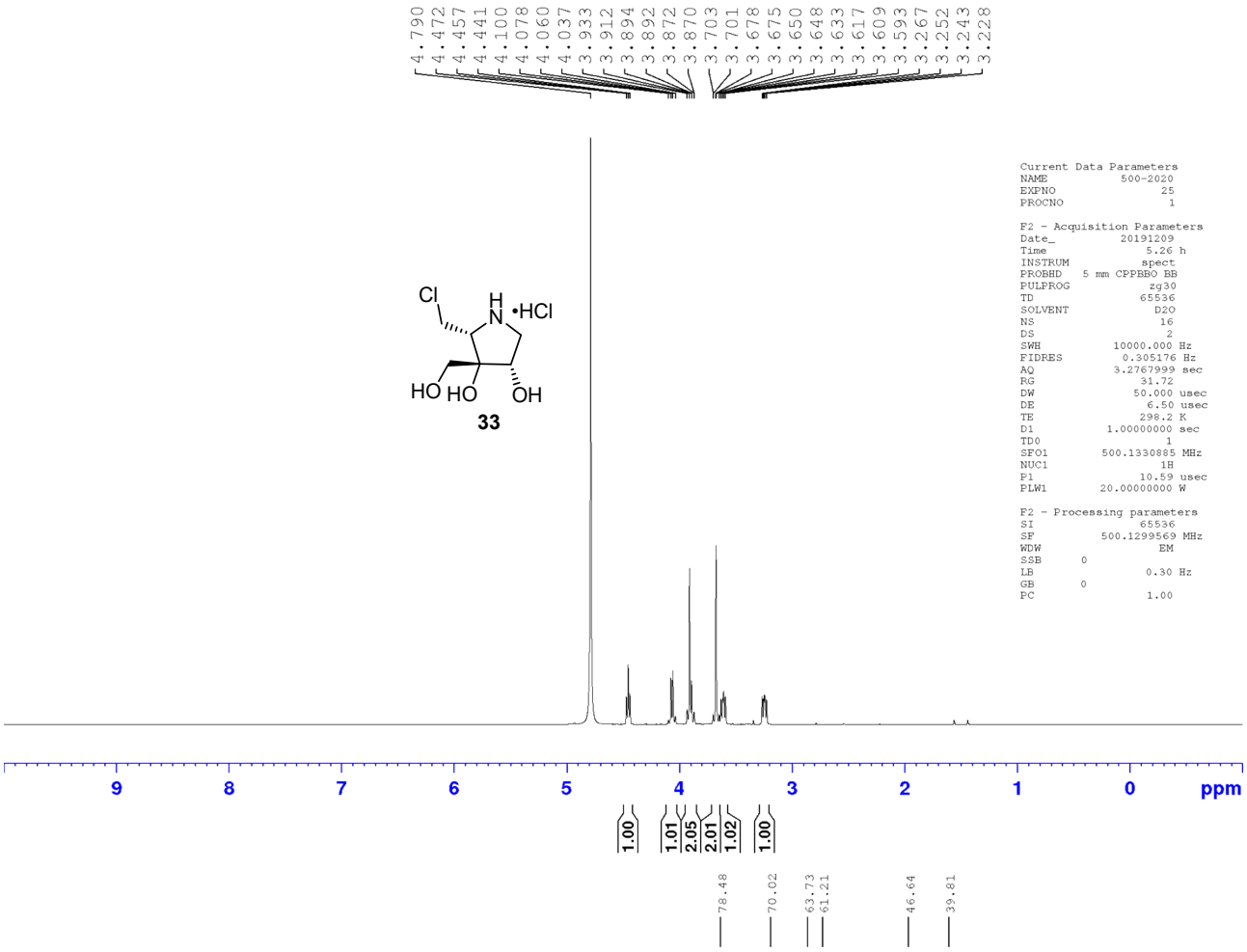
Compound 32:

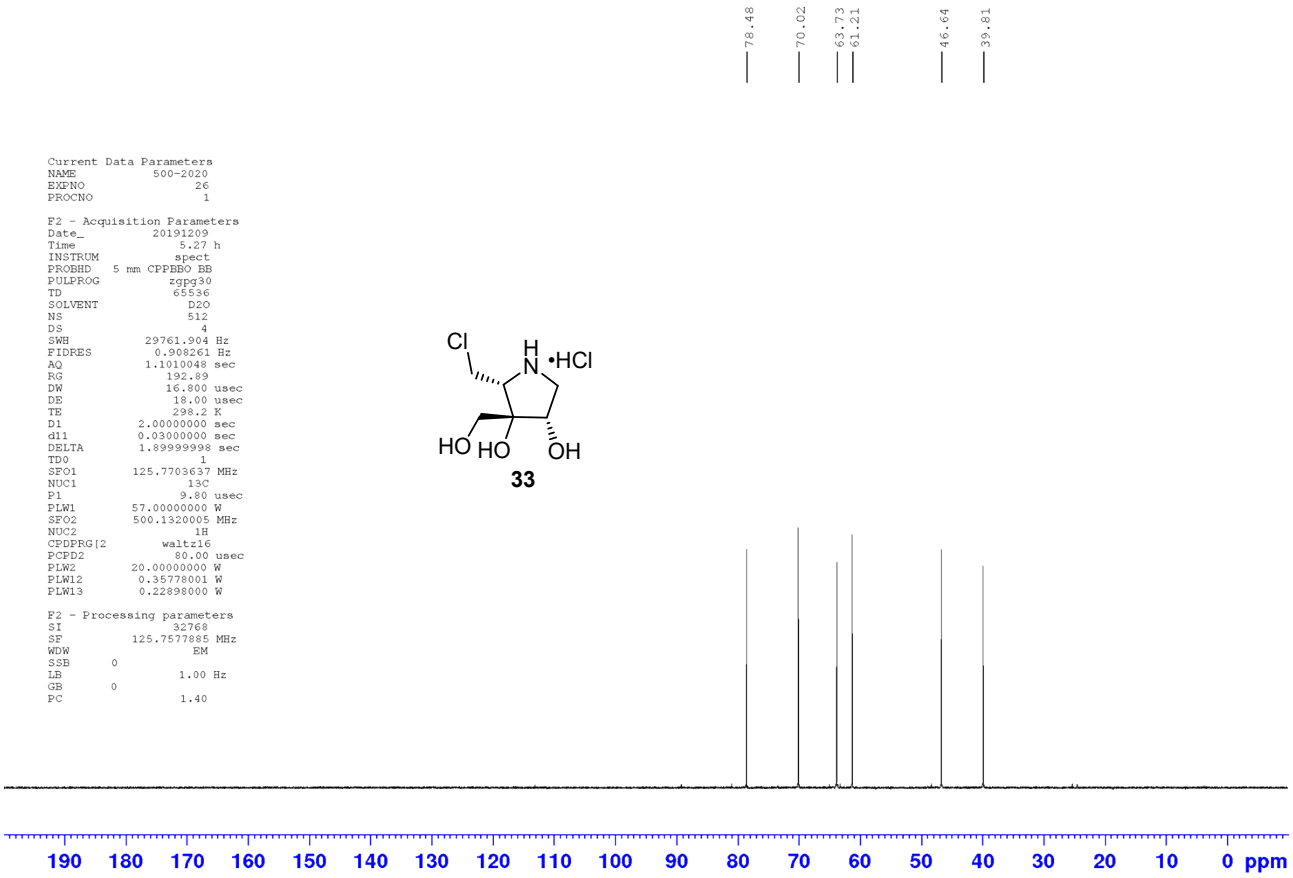

7.260
4.549
4.543
3.785
3.773
3.762
3.750
3.740
3.732
3.709
3.548
3.532
3.526
3.510
3.154
3.129
3.032
3.022
3.016
3.006
2.755
2.749
2.730
2.724
2.316
1.490
1.399
Current Data Parameters
NAME 500-2019-1
EXPNO 57
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191107
Time 23.34 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cl
H
N
HO
O
O
32
9
8
7
6
5
4
3
2
1
0
ppm
0.99
2.97
1.00
1.01
0.98
1.02
1.78
3.01
3.01
112.066
91.961
84.348
77.300
77.047
76.793
65.201
64.899
51.042
43.781
27.015
26.554
Current Data Parameters
NAME 500-2019-1
EXPNO 59
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191107
Time 23.44 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 320
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cl
H
N
HO
O
O
32
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

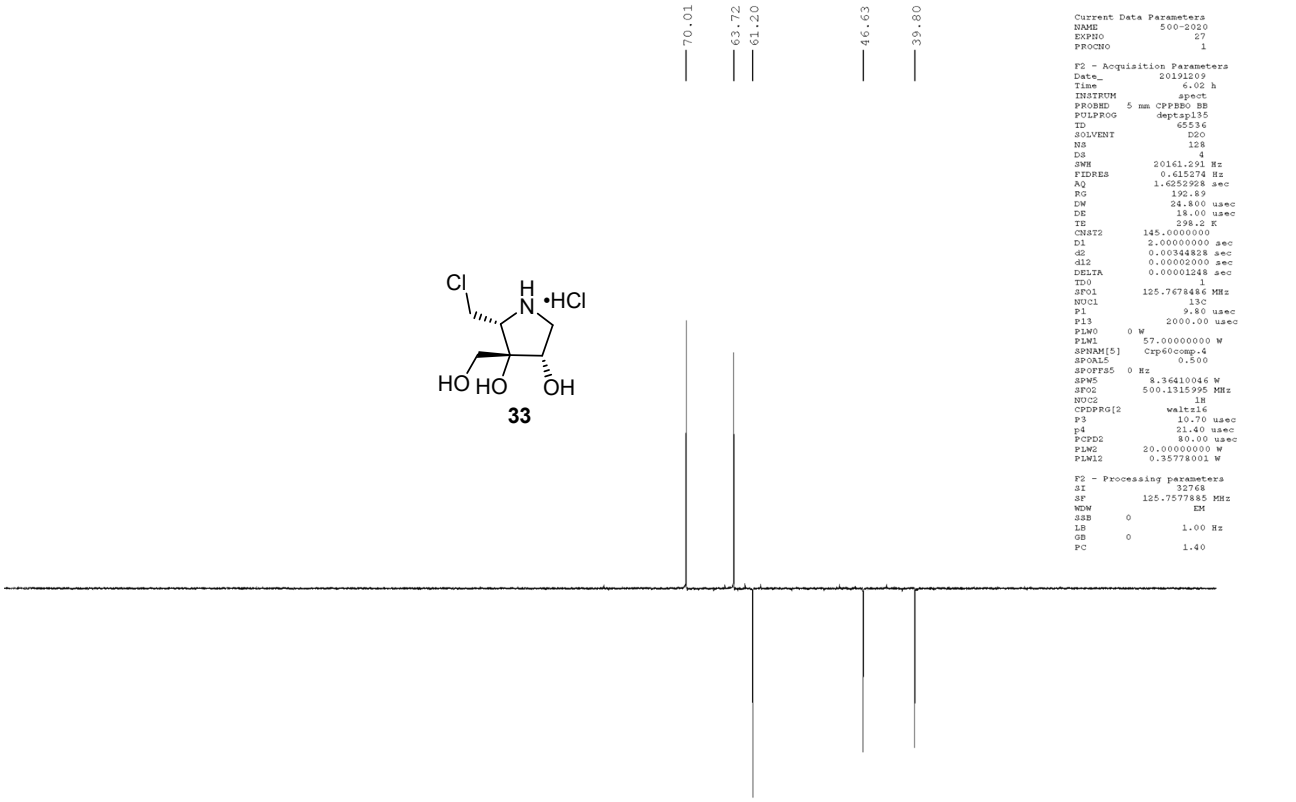

84.35
65.20
64.90
51.04
43.78
27.02
26.55
Cl
H
N
HO
O
O
32
Current Data Parameters
NAME 500-2019-1
EXPNO 59
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191108
Time 0.08 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

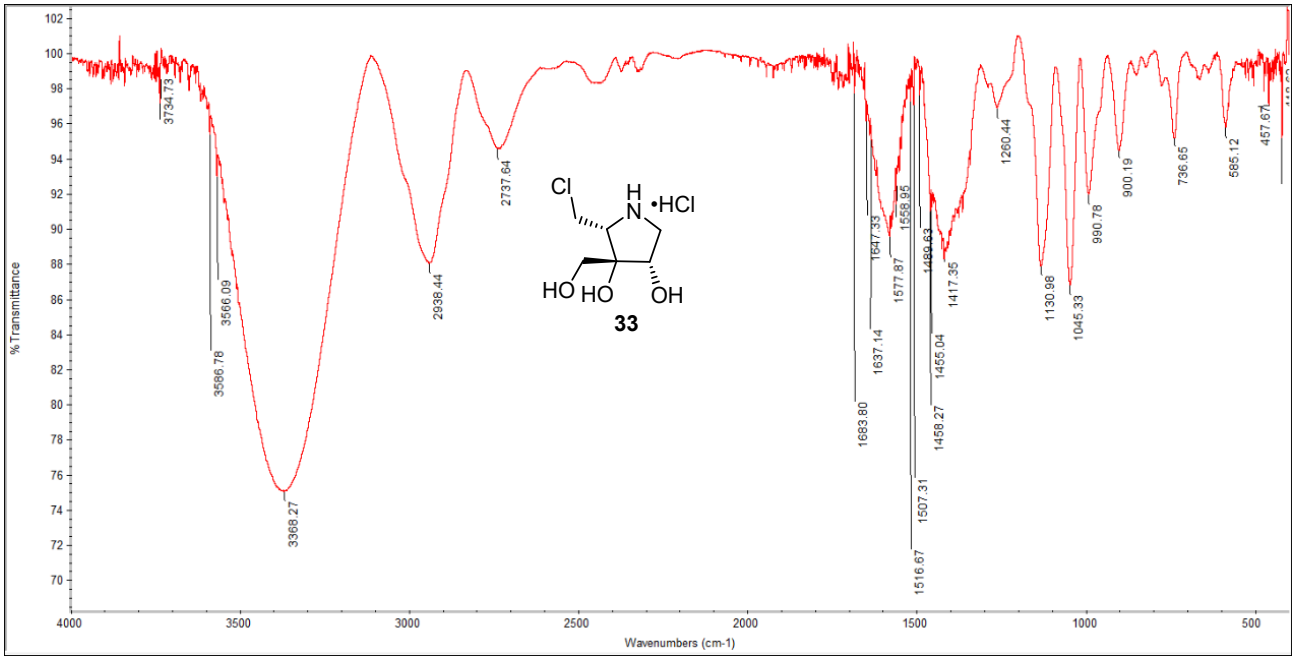

% Transmittance
Wavenumbers (cm-1)
3366.86
2986.52
2933.91
2857.60
1697.12
1564.88
1457.66
1428.34
1381.72
1373.24
1249.60
1211.61
1171.61
1096.21
1076.71
1051.25
1021.49
981.15
922.13
896.11
847.30
821.42
743.51
703.33
621.47
608.51
515.04
Cl
H
N
HO
O
O
32

Compound 33:


4.790
4.472
4.457
4.441
4.100
4.078
4.060
4.037
3.933
3.912
3.894
3.892
3.872
3.870
3.703
3.701
3.678
3.675
3.650
3.648
3.633
3.617
3.609
3.593
3.267
3.252
3.243
3.228
Cl
H
N
•HCl
HO
HO
OH
33
Current Data Parameters
NAME 500-2020
EXPNO 25
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191209
Time 5.26 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT D2O
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.59 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1299569 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
2.05
2.01
1.02
1.00

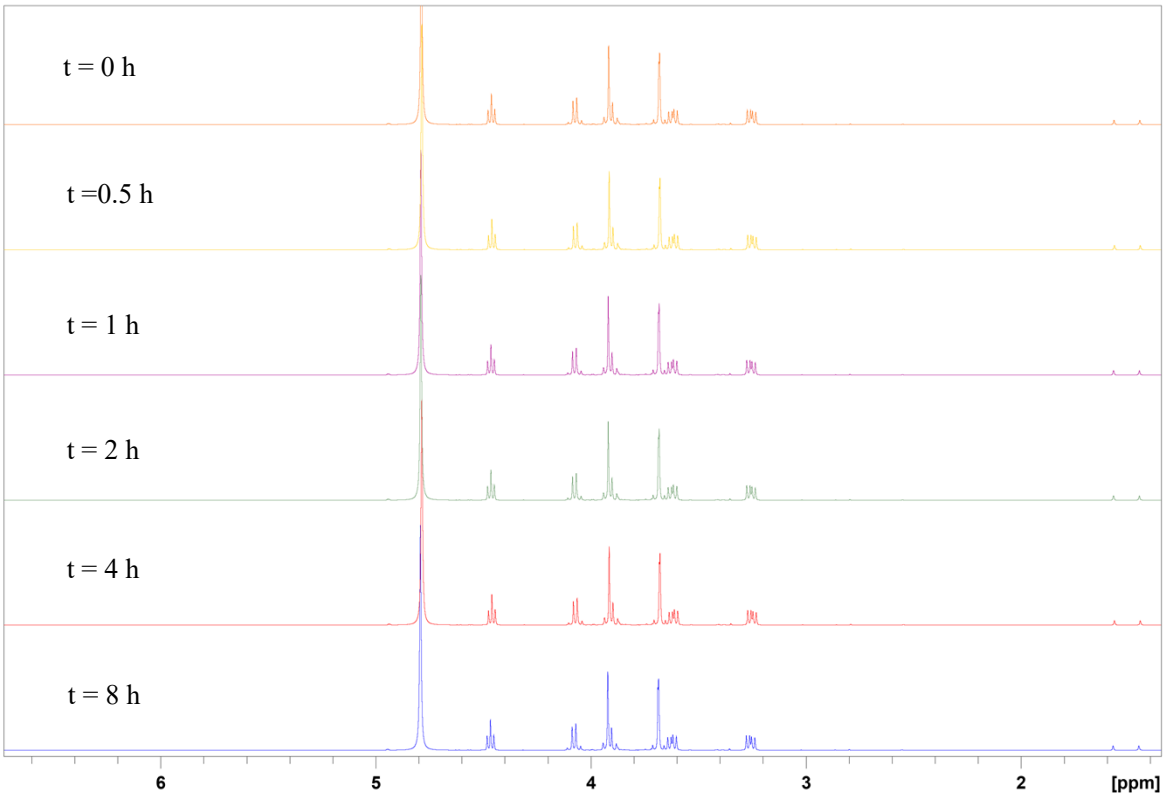

78.48
70.02
63.73
61.21
46.64
39.81
Current Data Parameters
NAME 500-2020
EXPNO 26
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191209
Time 5.27 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cl
H
N
•HCl
HO
HO
OH
33
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


70.01
63.72
61.20
46.63
39.80
Cl
H
N
•HCl
HO
HO
OH
33
Current Data Parameters
NAME 500-2020
EXPNO 27
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191209
Time 6.02 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptap135
TD 65536
SOLVENT D2O
NS 128
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

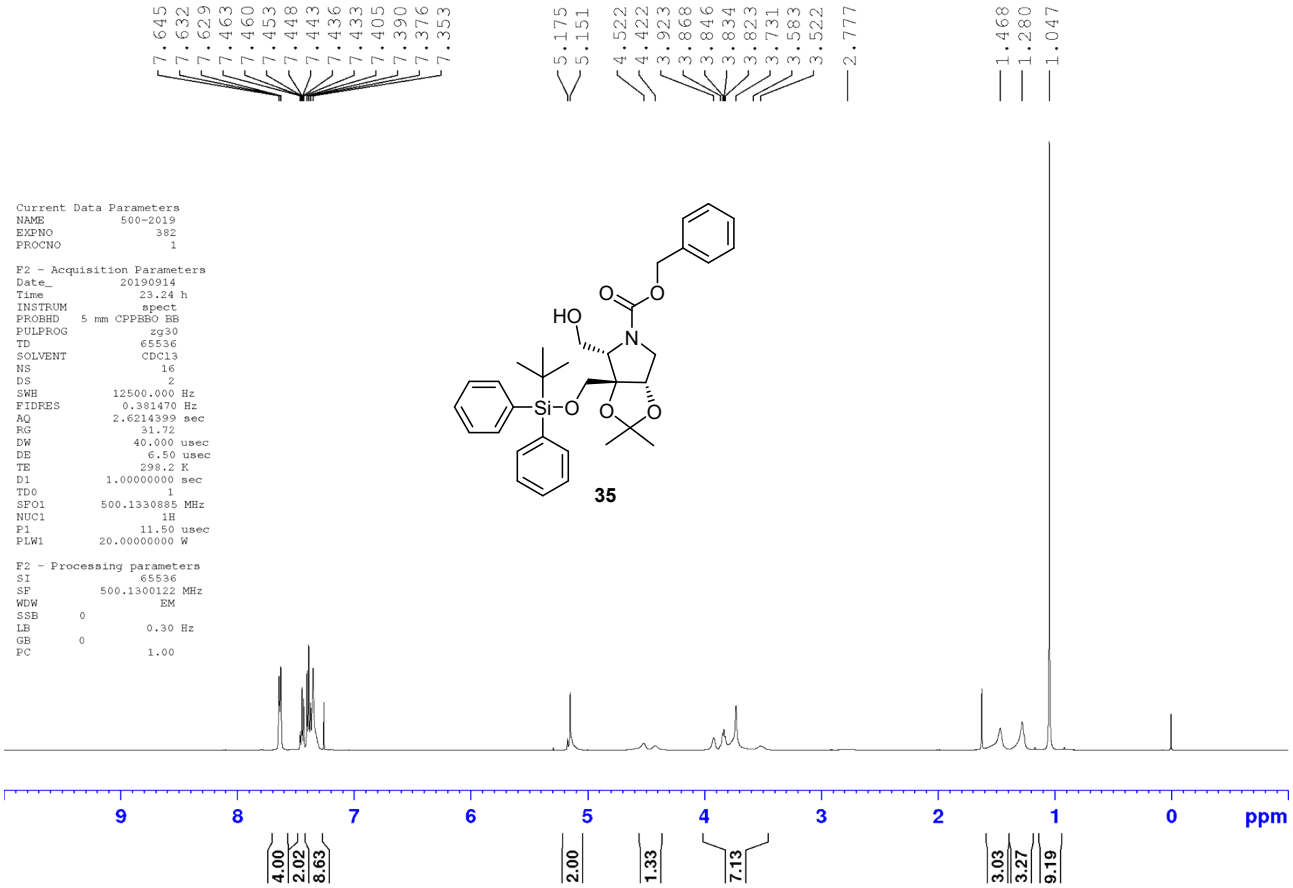

% Transmittance
Wavenumbers (cm-1)
3734.73
3566.09
3586.78
3368.27
2938.44
2737.64
1683.80
1637.14
1647.33
1577.87
1558.96
1516.67
1507.31
1489.63
1459.27
1455.04
1417.35
1260.44
1130.98
1045.33
990.78
900.19
736.65
585.12
457.67
Cl
H
N
•HCl
HO
HO
OH
33

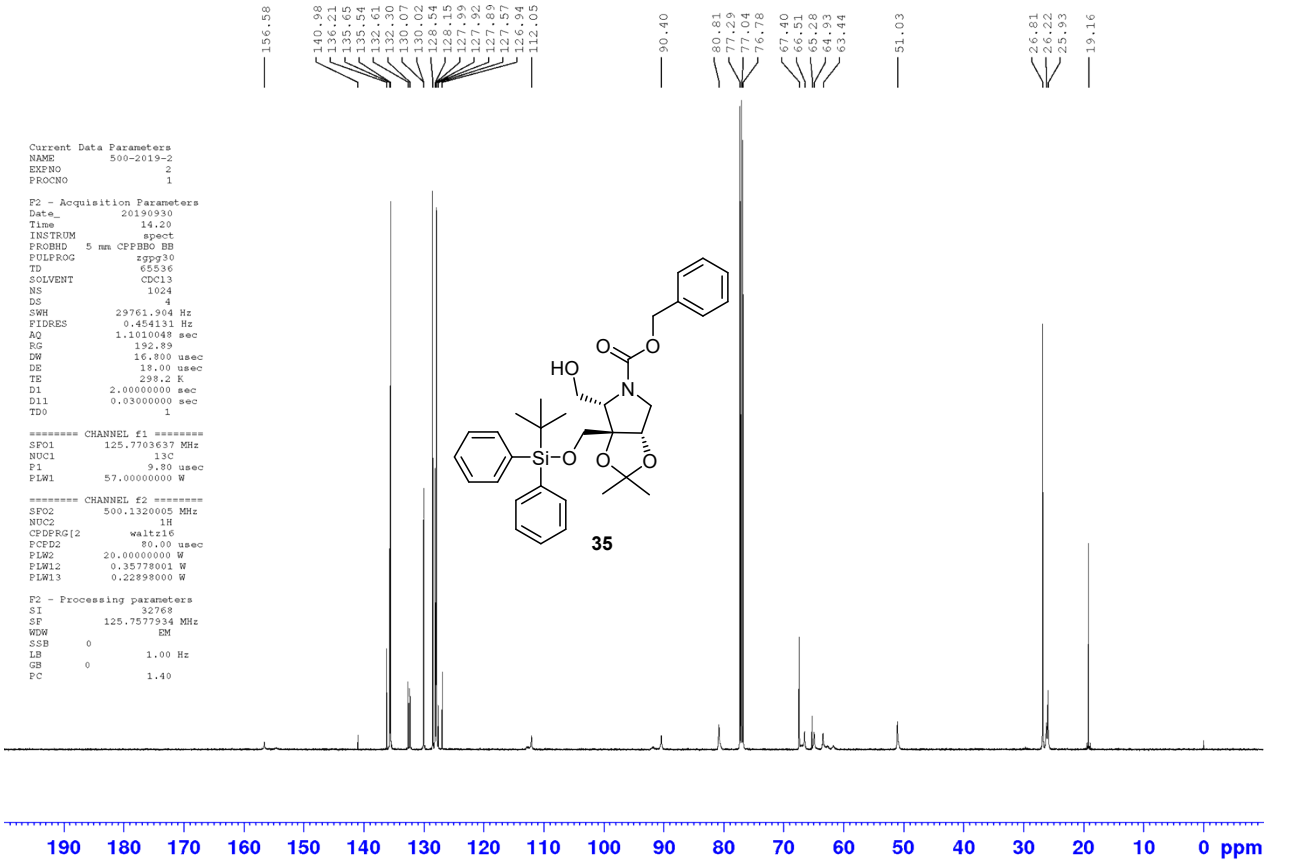
Stability experiment of compound **33** (pH = 7.0)

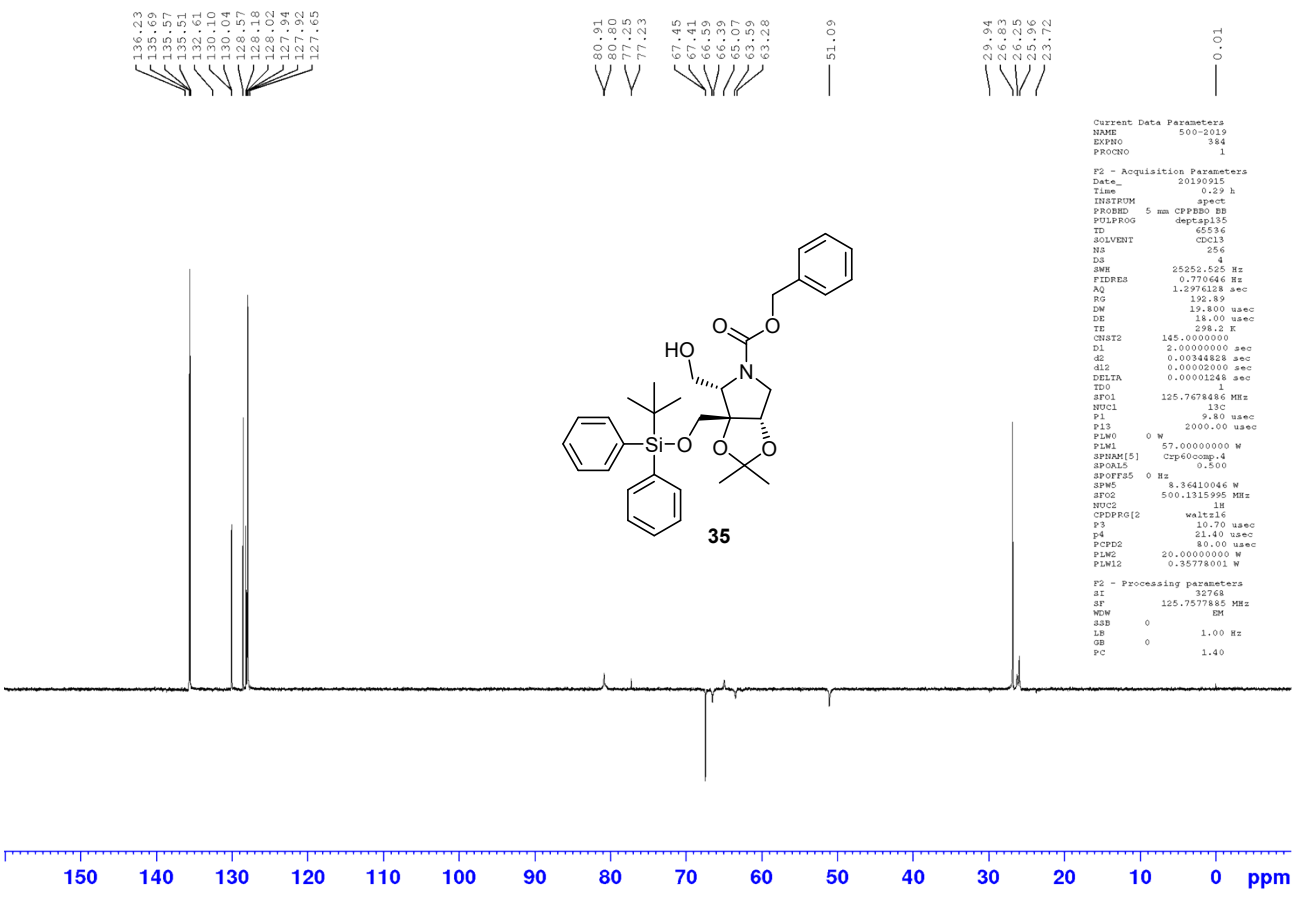
The ^{1}H NMR spectra of compound **33** (pH = 7.0) were monitored at 0.5 h, 1 h, 2 h, 4 h and 8 h, respectively. Since no change was observed even after maintaining in the solution for 8 h, the stablity of compound **33** can be ensured.

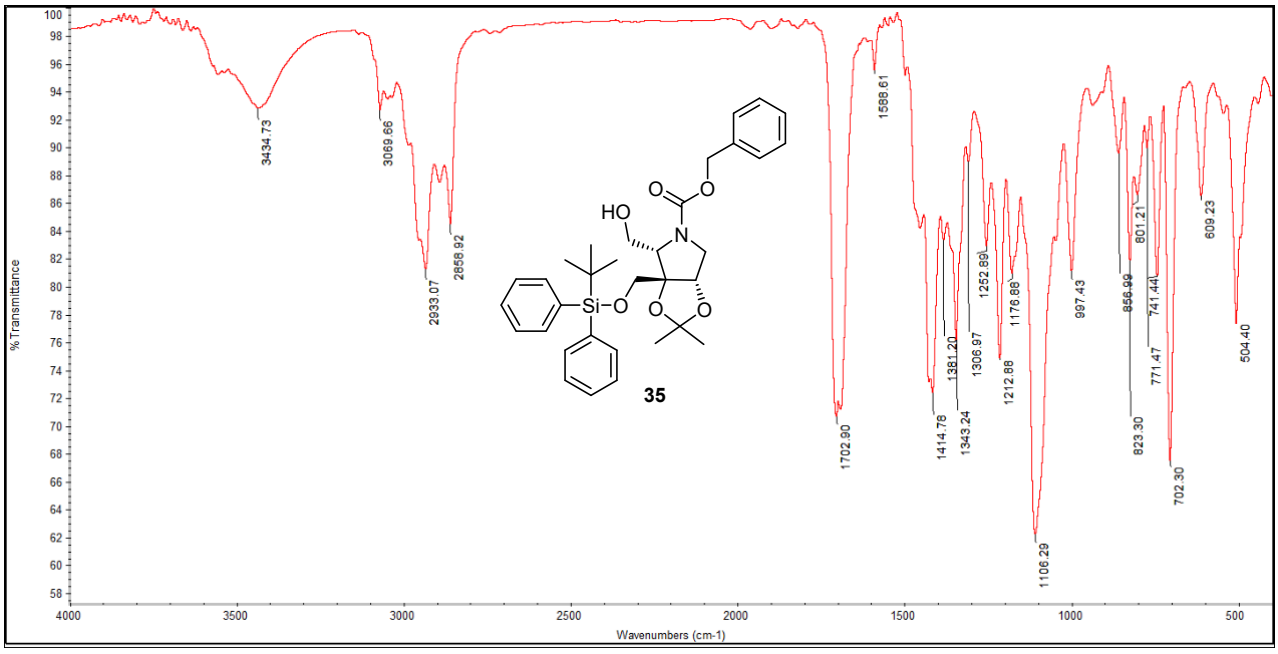


Compound 35:

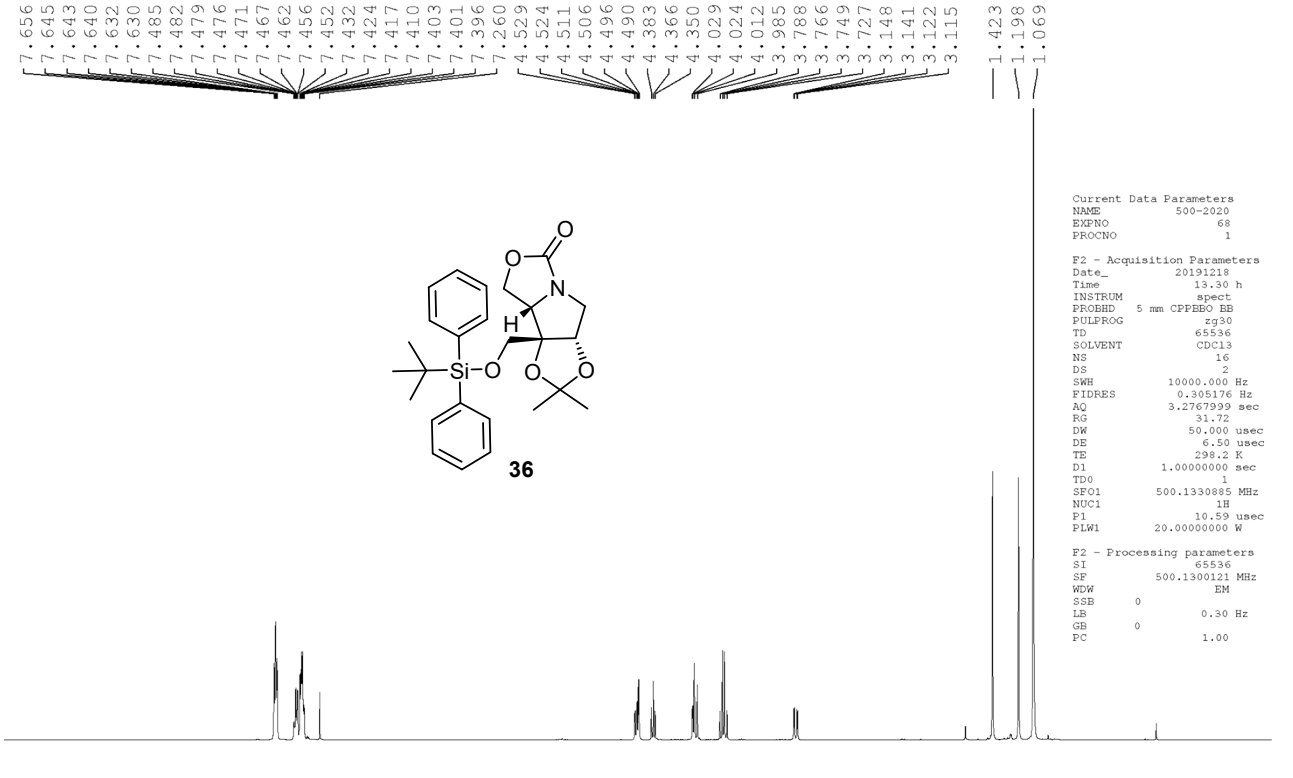

7.645
7.632
7.629
7.463
7.460
7.453
7.448
7.443
7.436
7.433
7.405
7.390
7.376
7.353
5.175
5.151
4.522
4.422
3.923
3.868
3.846
3.834
3.823
3.731
3.583
3.522
2.777
1.468
1.280
1.047
Current Data Parameters
NAME 500-2019
EXPNO 382
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190914
Time 23.24 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12500.000 Hz
FIDRES 0.381470 Hz
AQ 2.6214399 sec
RG 31.72
DW 40.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HO
N
O
Si
35
9
8
7
6
5
4
3
2
1
0
ppm
4.00
2.02
8.63
2.00
1.33
7.13
3.03
3.27
9.19

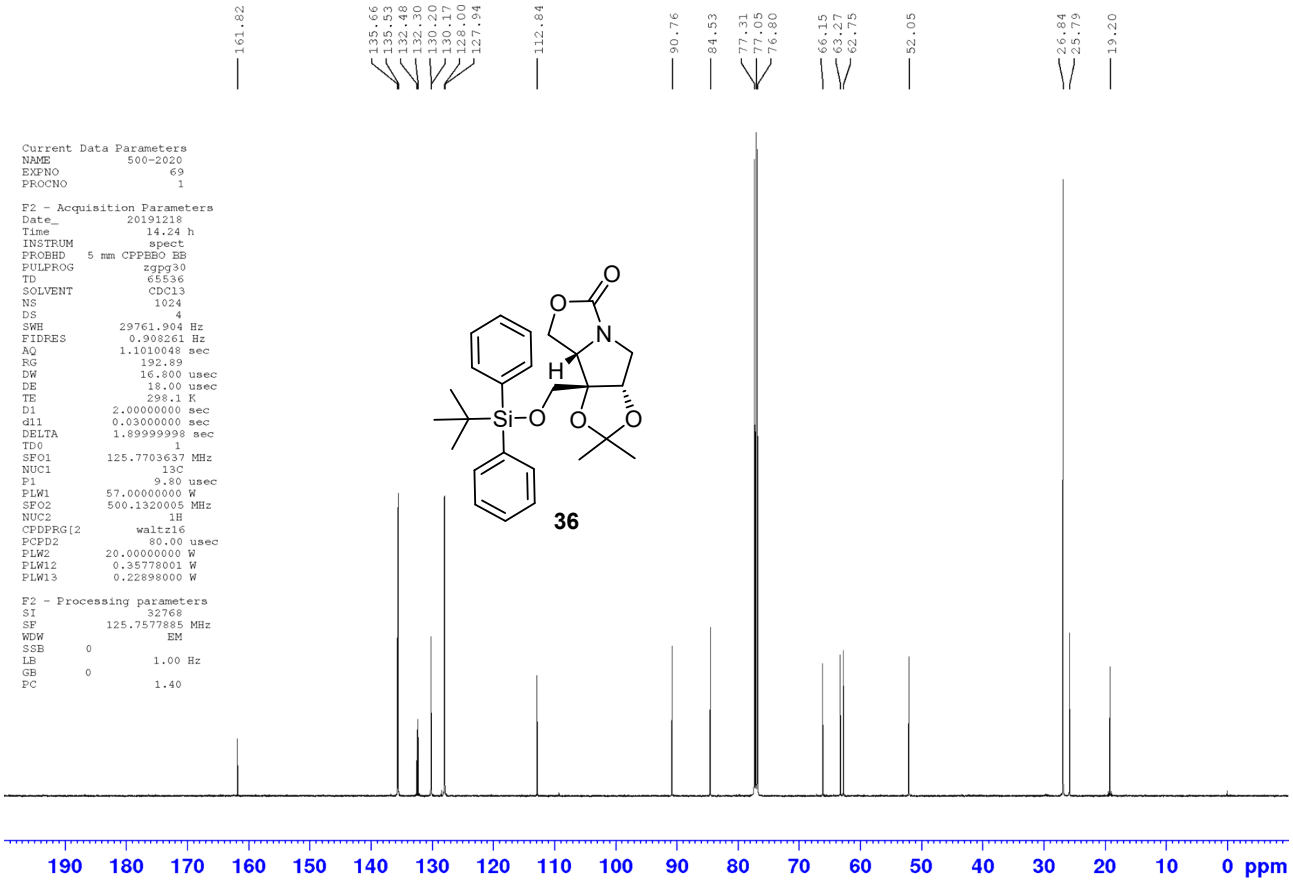

156.58
140.98
136.21
135.65
135.54
132.61
132.30
130.07
130.02
128.54
128.15
127.99
127.92
127.89
127.57
126.94
112.05
90.40
80.81
77.29
77.04
76.78
67.40
66.51
65.28
64.93
63.44
51.03
26.81
26.22
25.93
19.16
Current Data Parameters
NAME 500-2019-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190930
Time 14.20
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577934 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HO
N
O
Si
35
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

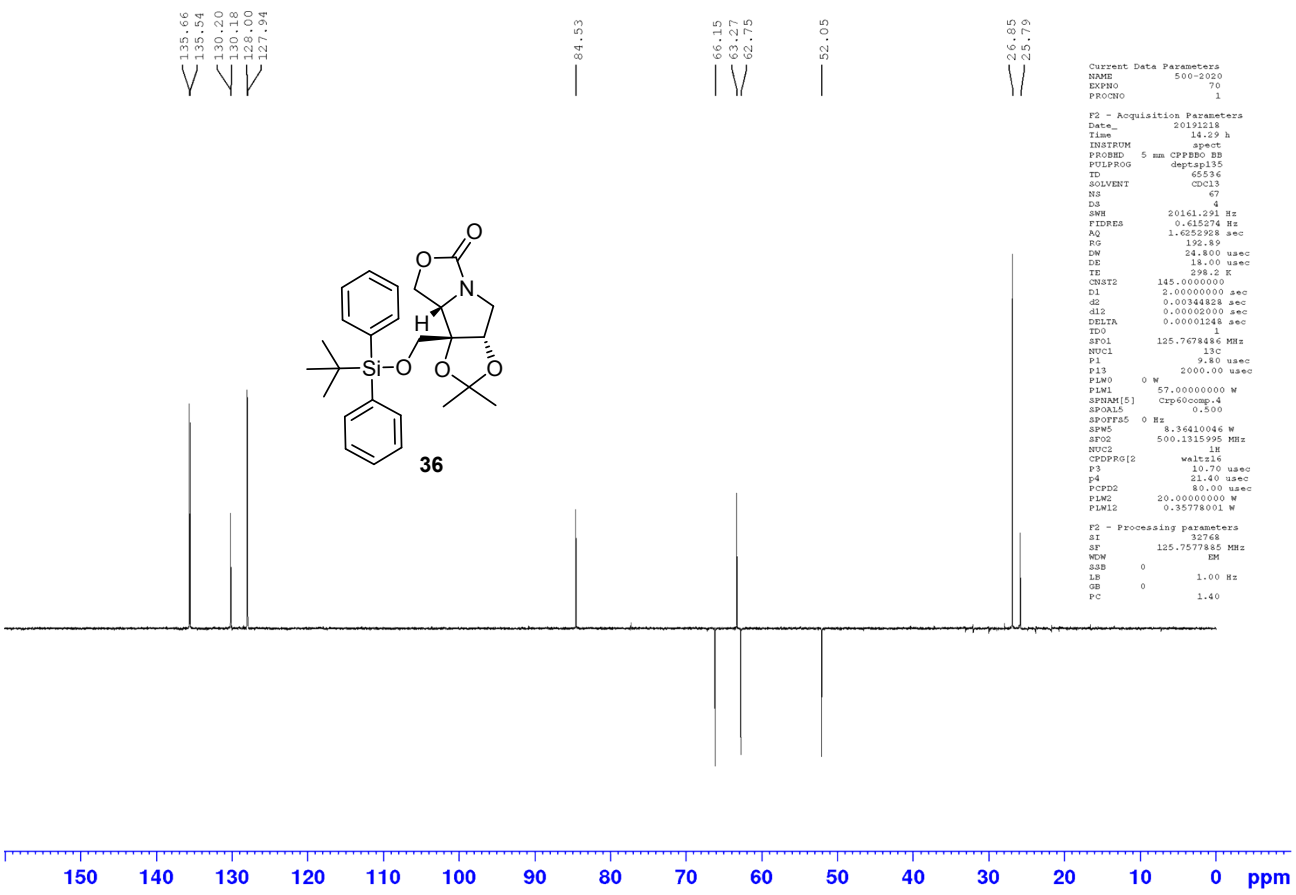

136.23
135.69
135.57
135.51
132.61
130.10
130.04
128.57
128.18
128.02
127.94
127.92
127.65
80.91
80.80
77.25
77.23
67.45
67.41
66.59
66.39
65.07
63.59
63.28
51.09
29.94
26.83
26.25
25.96
23.72
0.01
HO
N
O
O
Si
O
O
O
35
Current Data Parameters
NAME 500-2019
EXPNO 384
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190915
Time 0.29 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 1.2976128 sec
RG 192.89
DW 19.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

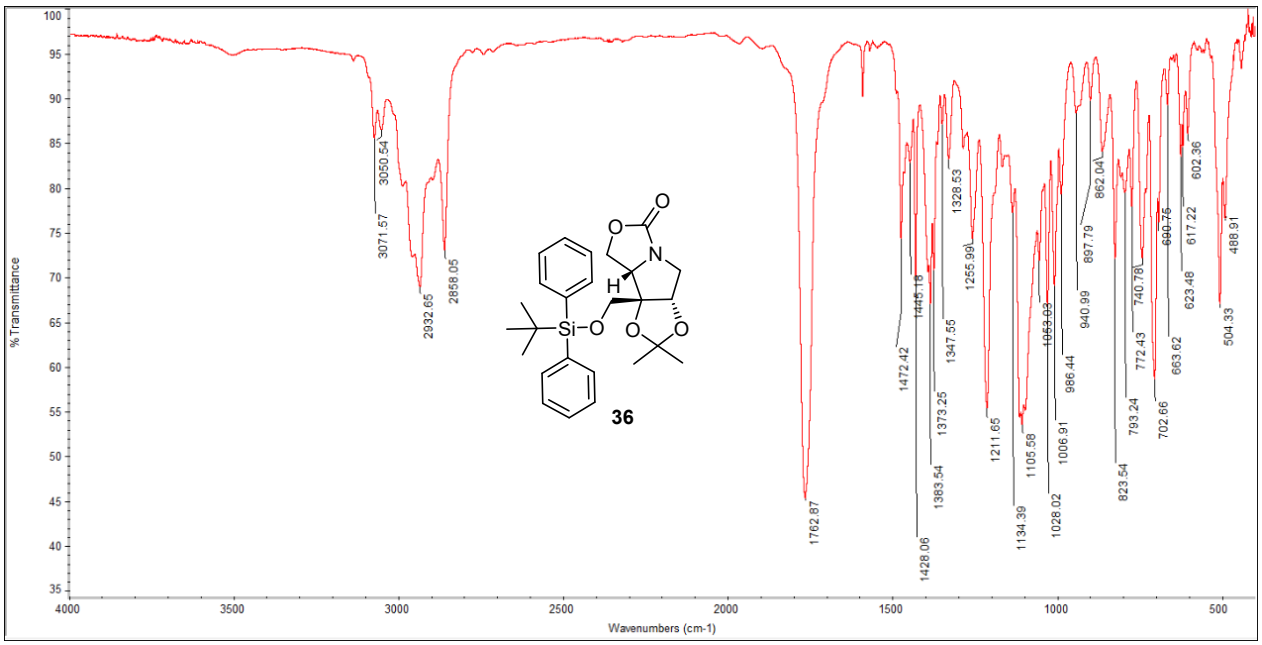

% Transmittance
Wavenumbers (cm-1)
3434.73
3069.86
2933.07
2858.92
1702.90
1588.61
1414.78
1381.20
1343.24
1306.97
1252.89
1212.88
1176.86
1106.29
997.43
856.99
823.30
801.21
771.47
741.44
702.30
609.23
504.40
HO
N
O
O
Si
O
O
O
35

Compound 36**:**

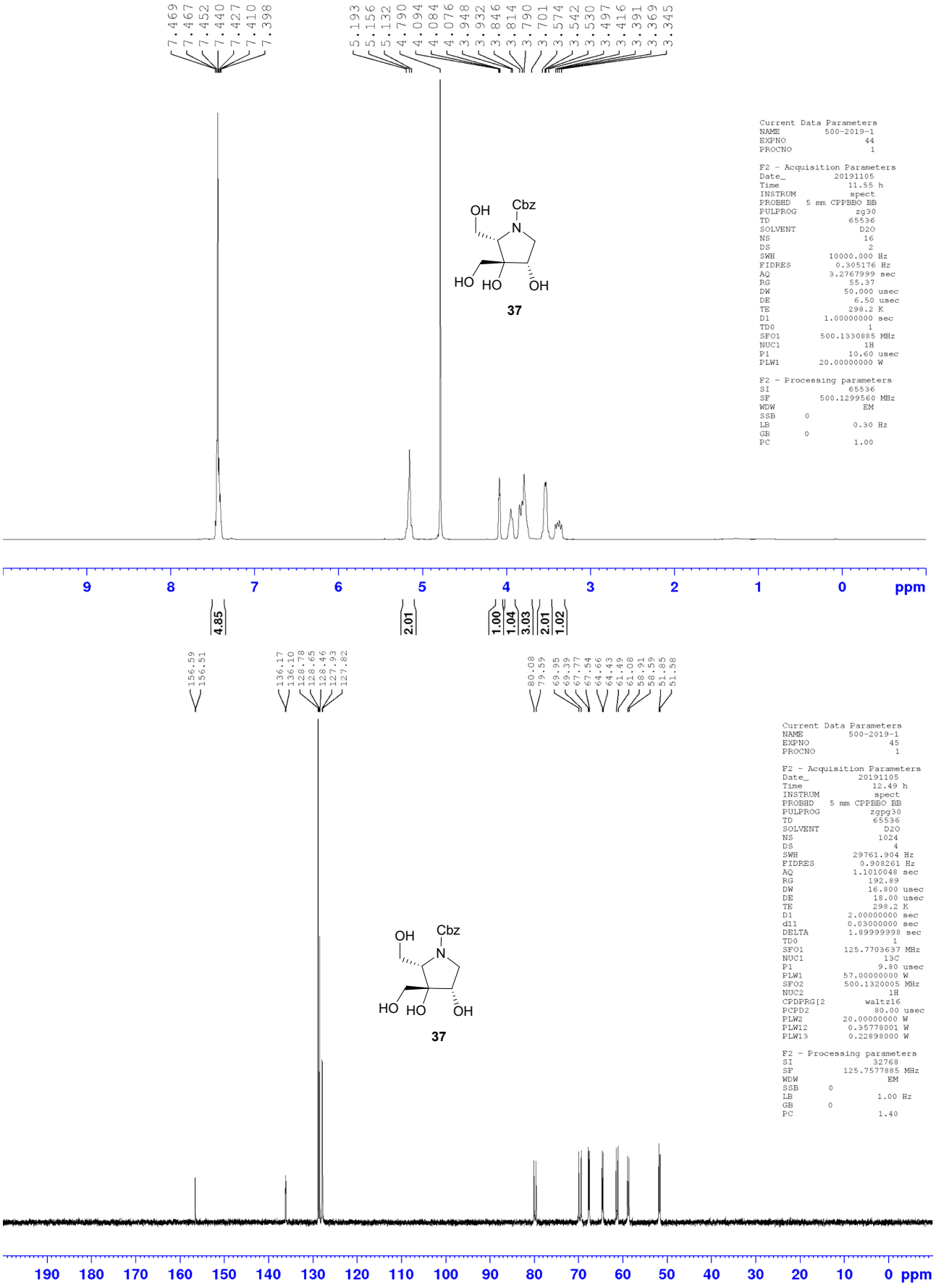

7.656
7.645
7.643
7.640
7.632
7.630
7.485
7.482
7.479
7.476
7.471
7.467
7.462
7.456
7.452
7.432
7.424
7.417
7.410
7.403
7.401
7.396
7.260
4.529
4.524
4.511
4.506
4.496
4.490
4.383
4.366
4.350
4.029
4.024
4.012
3.985
3.788
3.766
3.749
3.727
3.148
3.141
3.122
3.115
1.423
1.198
1.069
Si–O
H
N
O
36
Current Data Parameters
NAME 500-2020
EXPNO 68
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191218
Time 13.30 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.59 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
4.00 1.96 3.99 0.96 0.99 0.98 2.02 2.00 1.00 3.00 3.01 9.12

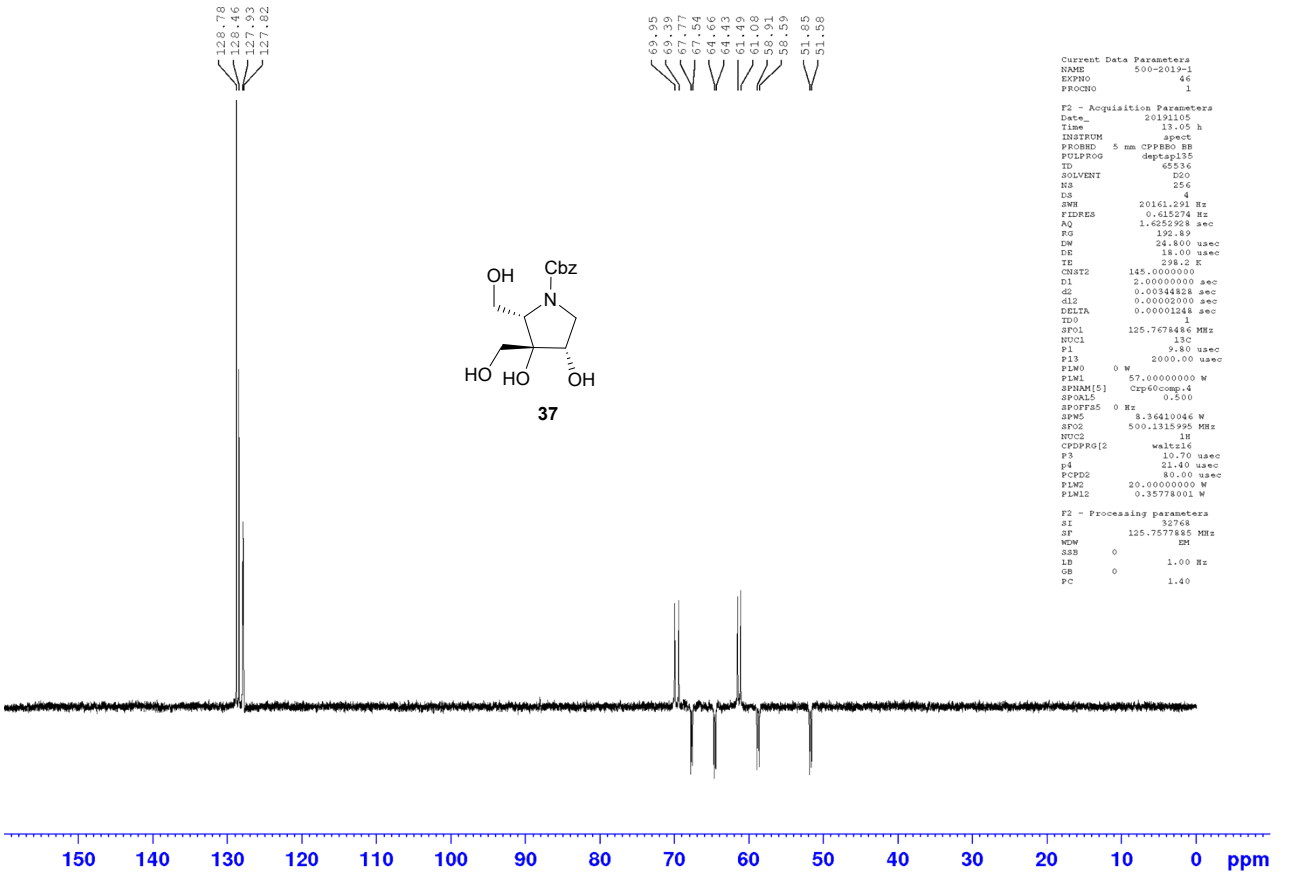

161.82
135.66
135.53
132.48
132.30
130.20
130.17
128.00
127.94
112.84
90.76
84.53
77.31
77.05
76.80
66.15
63.27
62.75
52.05
26.84
25.79
19.20
Current Data Parameters
NAME 500-2020
EXPNO 69
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191218
Time 14.24 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Si–O
H
N
O
36
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

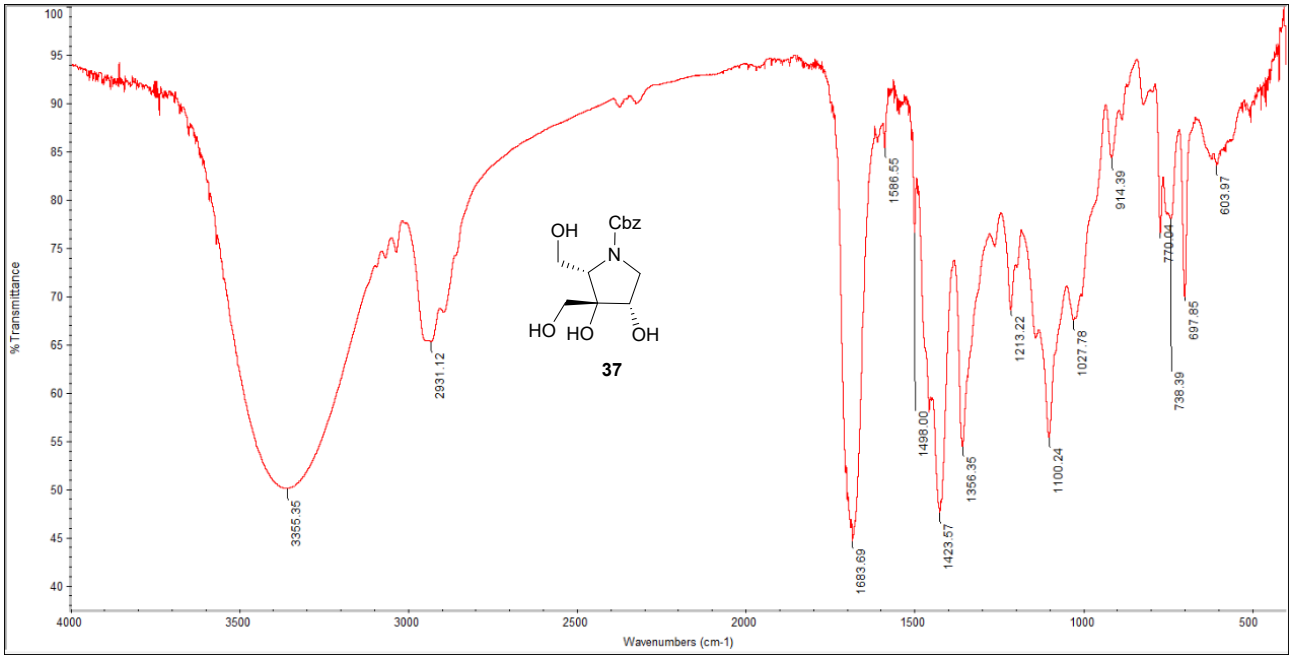

135.66
135.54
130.20
130.18
128.00
127.94
84.53
66.15
63.27
62.75
52.05
26.85
25.79
36
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


% Transmittance
3071.57
3050.54
2932.65
2858.05
1762.87
1472.42
1445.18
1428.06
1383.54
1373.25
1347.55
1328.53
1255.99
1211.65
1134.39
1105.58
1063.03
1028.02
1006.91
986.44
940.99
897.79
862.04
823.54
793.24
772.43
740.78
702.66
690.76
663.62
623.48
617.22
602.36
504.33
488.91
36
Wavenumbers (cm-1)

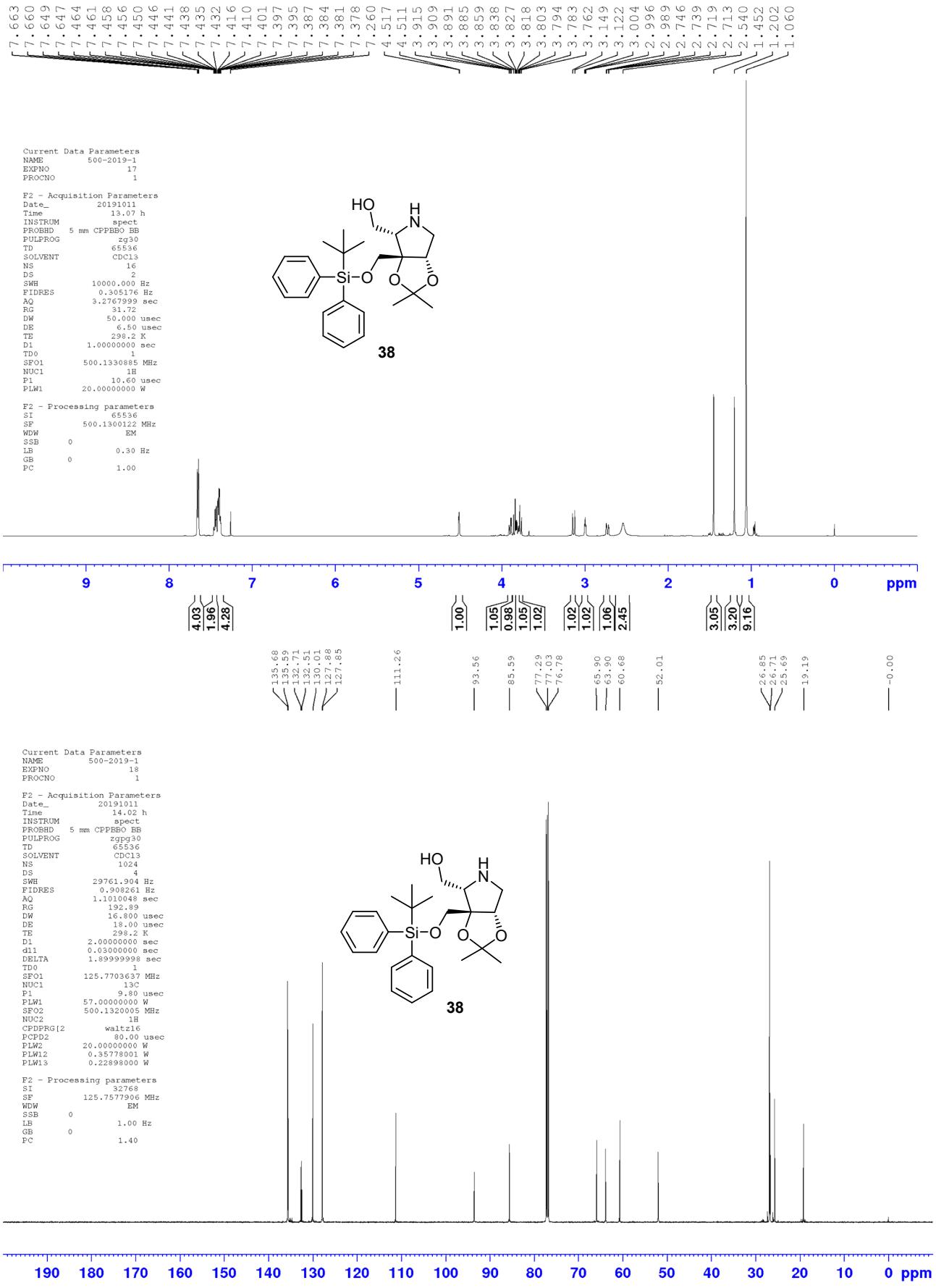
Compound 37:

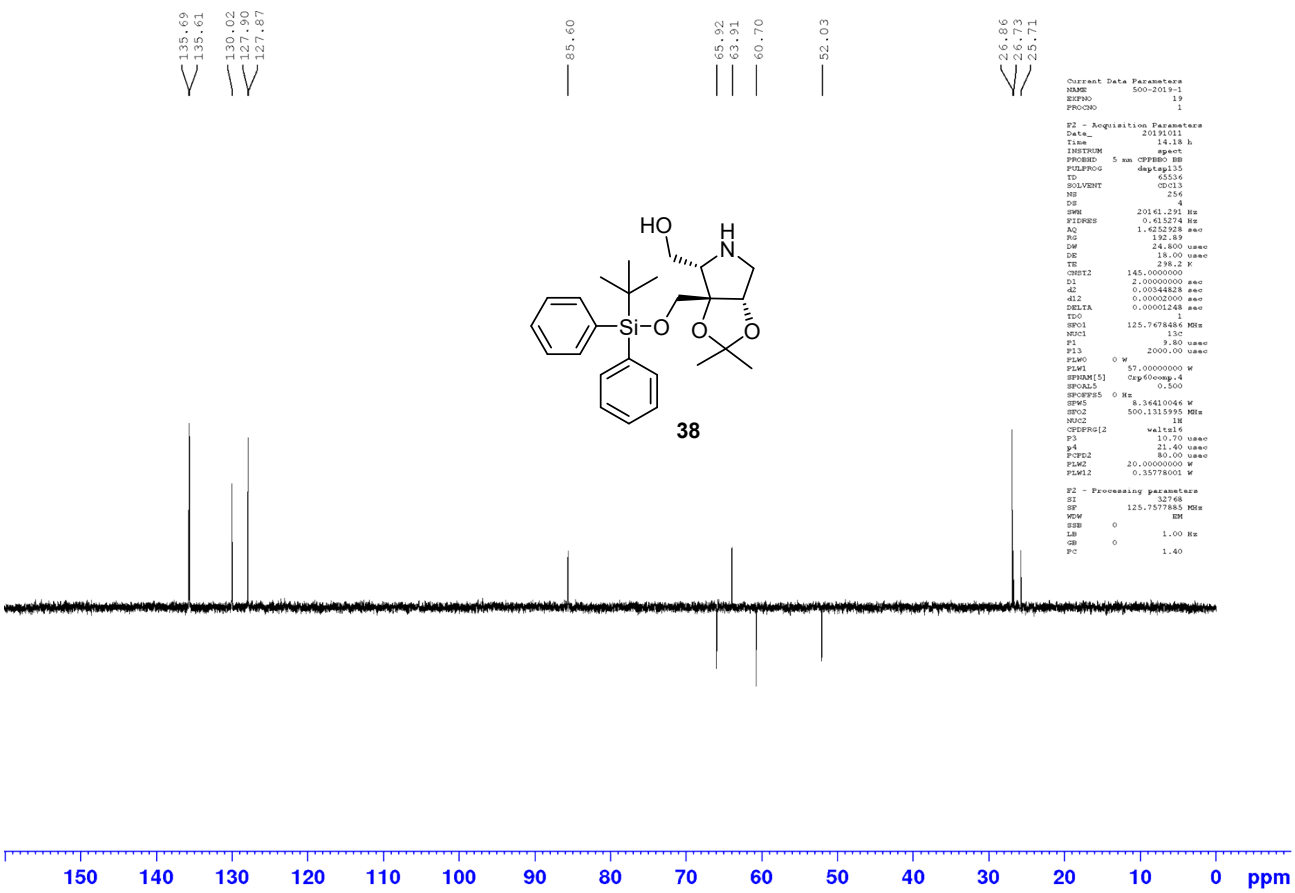

7.469
7.467
7.452
7.440
7.427
7.410
7.398
5.193
5.156
5.132
4.790
4.094
4.084
4.076
3.948
3.932
3.846
3.814
3.790
3.701
3.574
3.542
3.530
3.497
3.416
3.391
3.369
3.345
OH
Cbz
N
HO
HO
OH
37
Current Data Parameters
NAME 500-2019-1
EXPNO 44
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191105
Time 11.55 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT D2O
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 55.37
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1299560 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
4.85
2.01
1.00
1.04
3.03
2.01
1.02
156.59
156.51
136.17
136.10
128.78
128.65
128.46
127.93
127.82
80.08
79.59
69.95
69.39
67.77
67.54
64.66
64.43
61.49
61.08
58.91
58.59
51.85
51.58
OH
Cbz
N
HO
HO
OH
37
Current Data Parameters
NAME 500-2019-1
EXPNO 45
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191105
Time 12.49 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

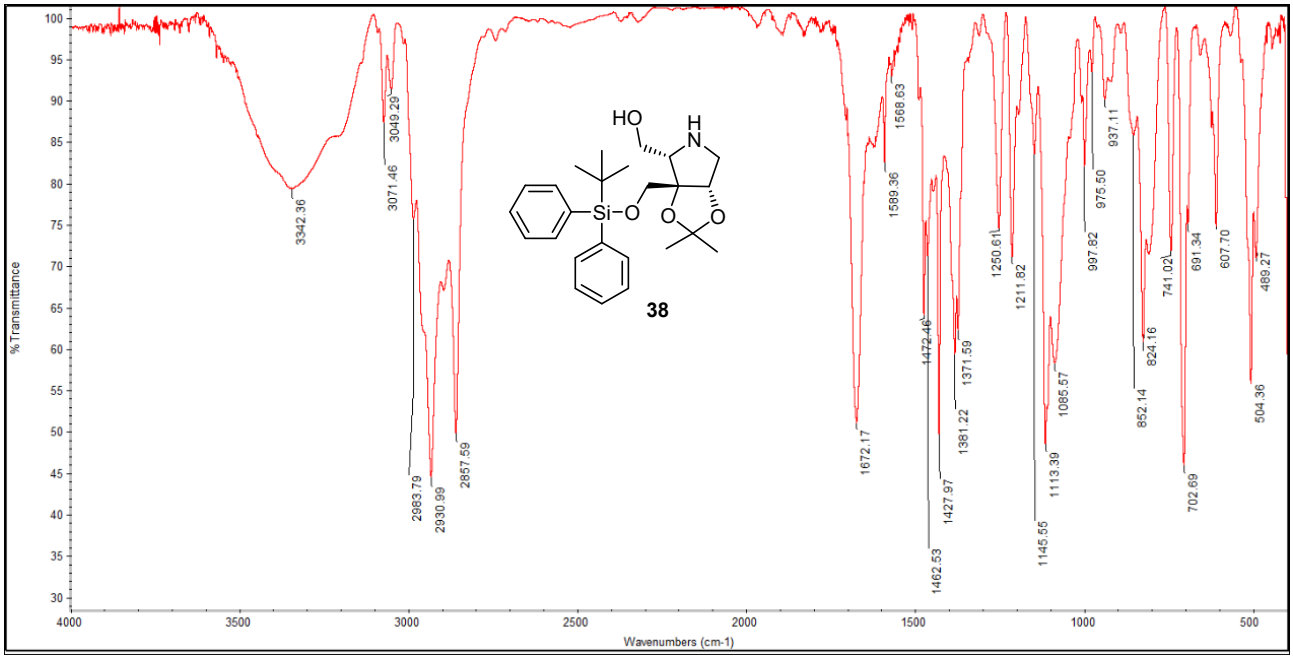

128.78
128.46
127.93
127.82
69.95
69.39
67.77
67.54
64.66
64.43
61.49
61.08
58.91
58.59
51.85
51.58
OH
Cbz
N
HO
HO
OH
37
Current Data Parameters
NAME 500-2019-1
EXPNO 46
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191105
Time 13.05 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptsp135
TD 65536
SOLVENT D2O
NS 256
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


% Transmittance
Wavenumbers (cm-1)
3355.35
2931.12
1683.69
1586.55
1498.00
1423.57
1356.35
1213.22
1100.24
1027.78
914.39
770.04
738.39
697.85
603.97
OH
Cbz
N
HO
HO
OH
37

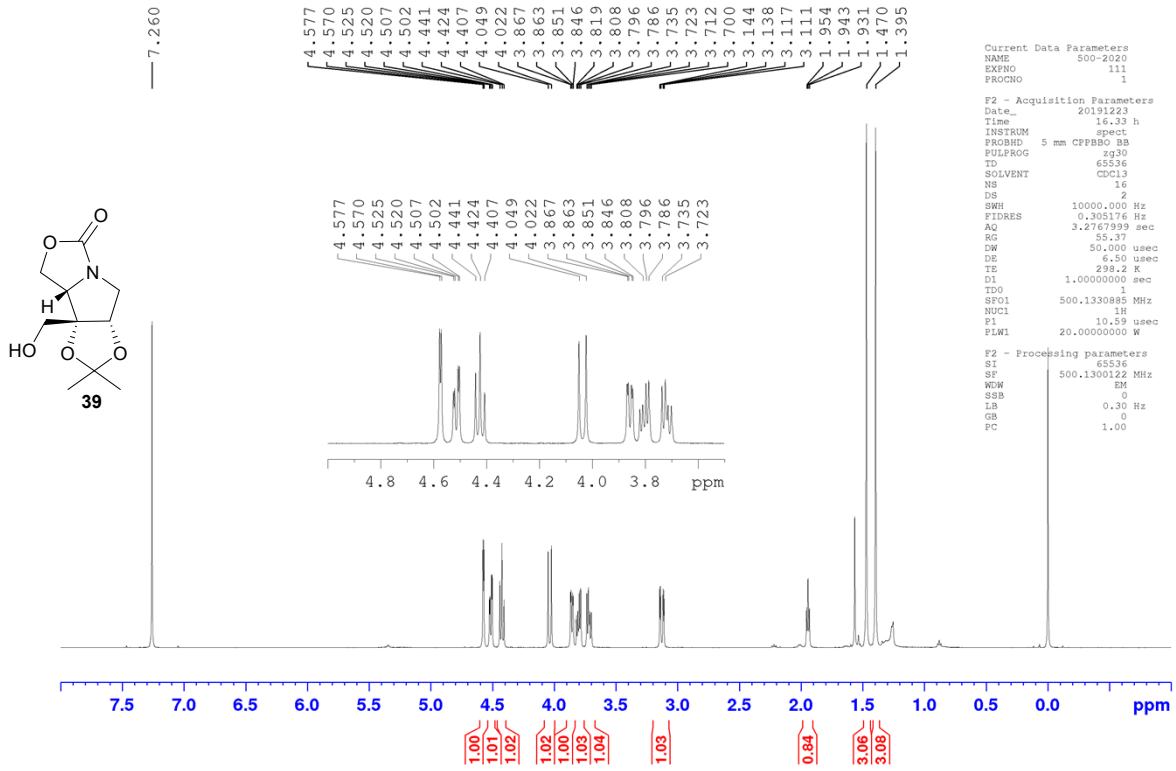
Compound 38:

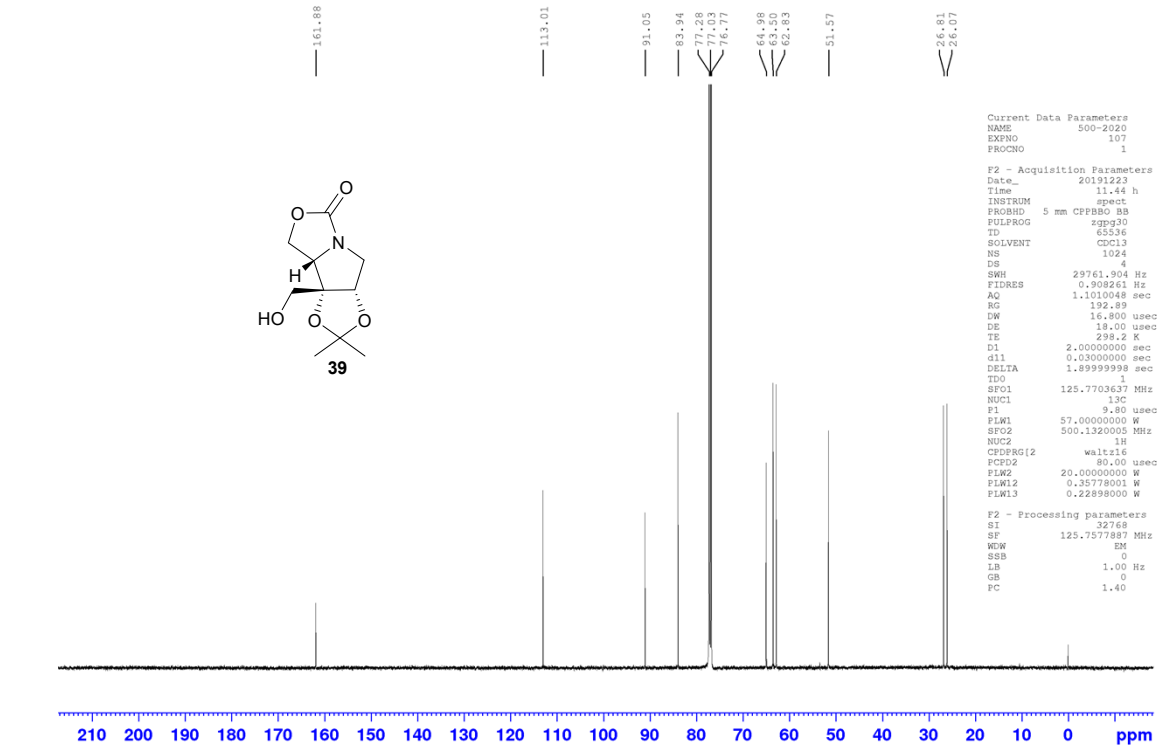

7.663 7.660 7.649 7.647 7.464 7.461 7.458 7.456 7.450 7.446 7.441 7.438 7.435 7.432 7.416 7.410 7.401 7.397 7.395 7.387 7.384 7.381 7.378 7.260 4.517 4.511 3.915 3.909 3.891 3.885 3.859 3.838 3.827 3.818 3.803 3.794 3.783 3.762 3.149 3.122 3.004 2.996 2.989 2.746 2.739 2.719 2.713 2.540 1.452 1.202 1.060
Current Data Parameters
NAME 500-2019-1
EXPNO 17
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191011
Time 13.07 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
HO
H
N
Si
O
O
O
38
9 8 7 6 5 4 3 2 1 0 ppm
4.03 1.96 4.28 1.00 1.05 0.98 1.05 1.02 1.02 1.02 1.06 2.45 3.05 3.20 9.16
135.68 135.59 132.71 132.51 130.01 127.88 127.85 111.26 93.56 85.59 77.29 77.03 76.78 65.90 63.90 60.68 52.01 26.85 26.71 25.69 19.19 -0.00
Current Data Parameters
NAME 500-2019-1
EXPNO 18
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191011
Time 14.02 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577906 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HO
H
N
Si
O
O
O
38
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

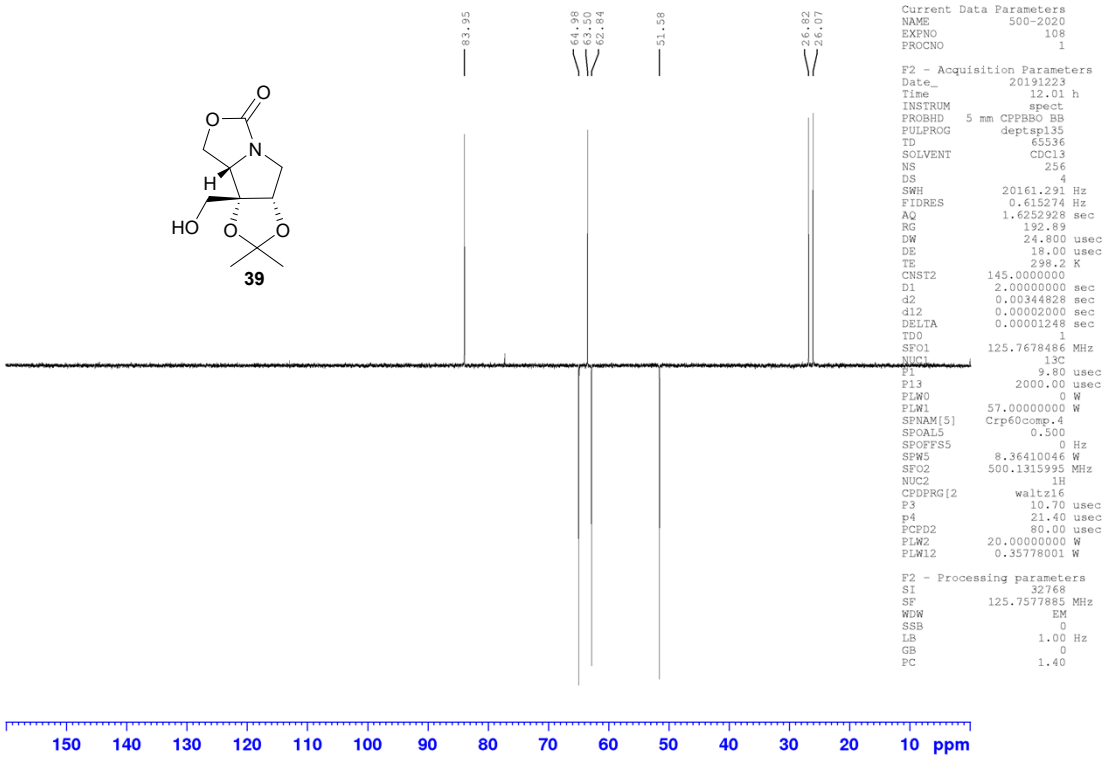

135.69
135.61
130.02
127.90
127.87
85.60
65.92
63.91
60.70
52.03
26.86
26.73
25.71
HO
H
N
Si
O
O
O
38
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


% Transmittance
Wavenumbers (cm-1)
3342.36
3071.46
3049.29
2983.79
2930.99
2857.59
1672.17
1589.36
1568.63
1472.46
1462.53
1427.97
1381.22
1371.59
1250.61
1211.82
1145.55
1113.39
1085.57
997.82
975.50
937.11
852.14
824.16
741.02
702.69
691.34
607.70
504.36
489.27
HO
H
N
Si
O
O
O
38

Compound 39:


7.260
4.577
4.570
4.525
4.520
4.507
4.502
4.441
4.424
4.407
4.049
4.022
3.867
3.863
3.851
3.846
3.819
3.808
3.796
3.786
3.735
3.723
3.712
3.700
3.144
3.138
3.117
3.111
1.954
1.943
1.931
1.470
1.395
Current Data Parameters
NAME 500-2020
EXPNO 111
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191223
Time 16.33 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 55.37
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.59 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.8
4.6
4.4
4.2
4.0
3.8
ppm
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
1.00
1.01
1.02
1.02
1.00
1.03
1.04
1.03
0.84
3.06
3.08
39


161.88
113.01
91.05
83.94
77.28
77.03
76.77
64.98
63.50
62.83
51.57
26.81
26.07
Current Data Parameters
NAME 500-2020
EXPNO 107
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191223
Time 11.44 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577887 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm
39



Current Data Parameters
NAME 500-2020
EXPNO 108
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191223
Time 12.01 h
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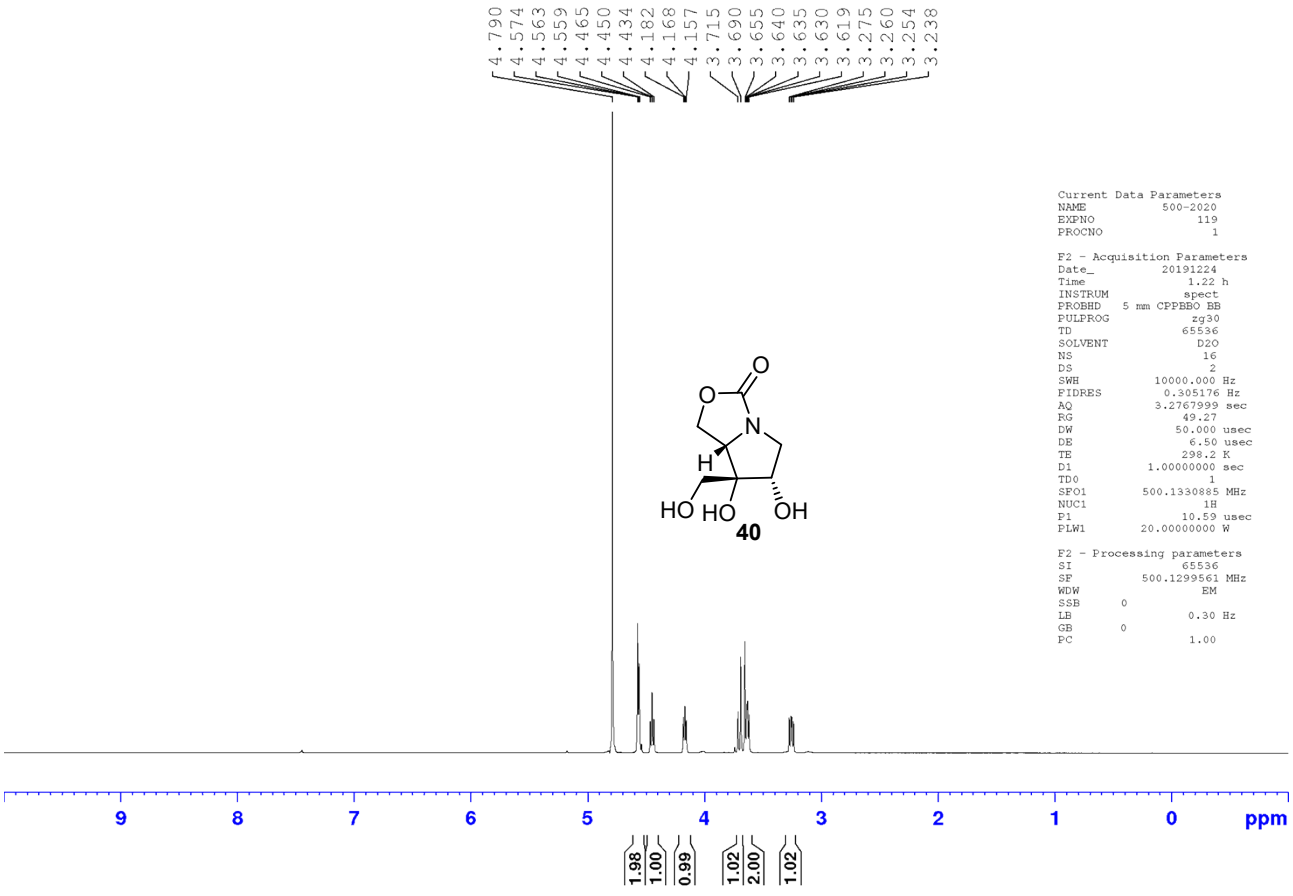
PROBHD 5 mm CPPBBO BB
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
p4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

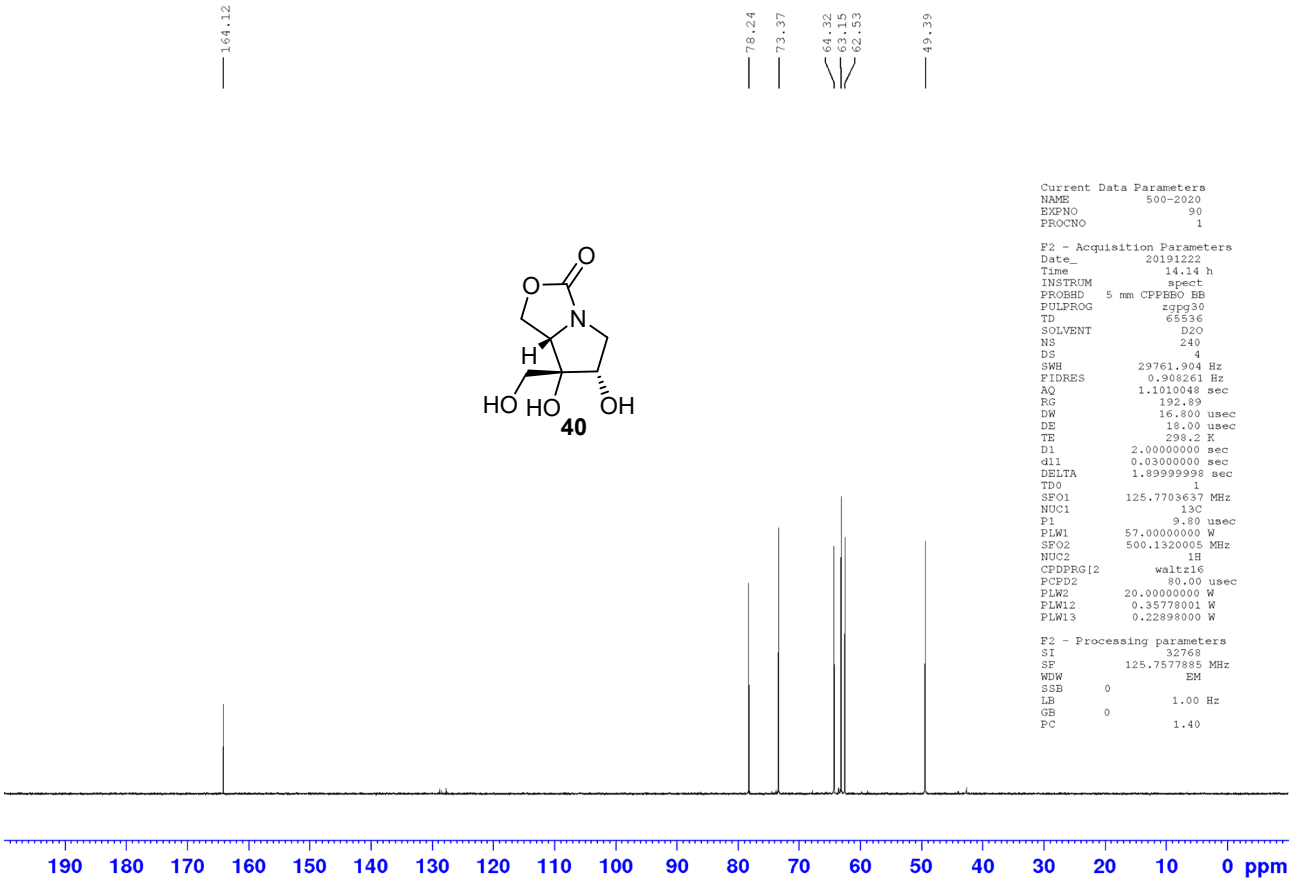
Compound 40**:**

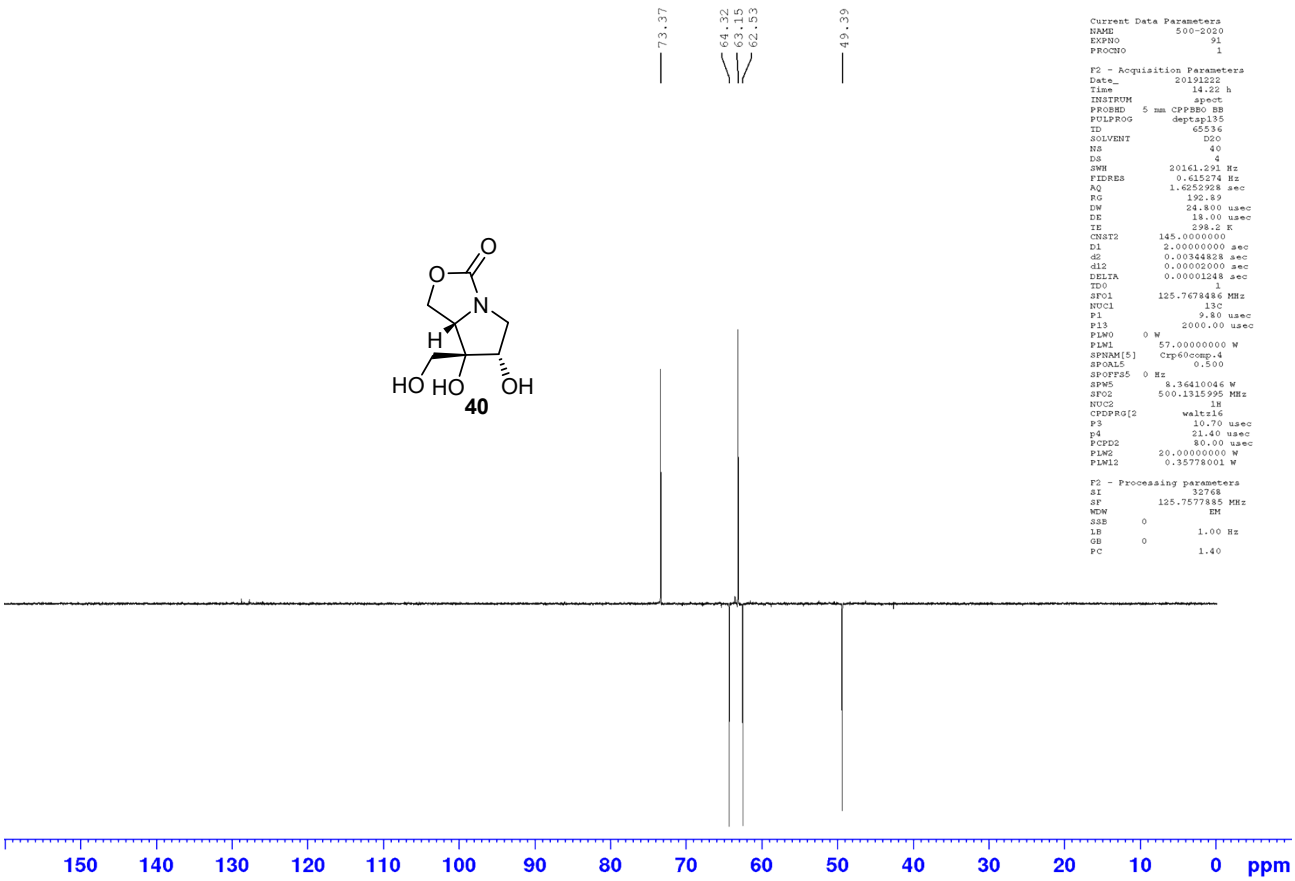


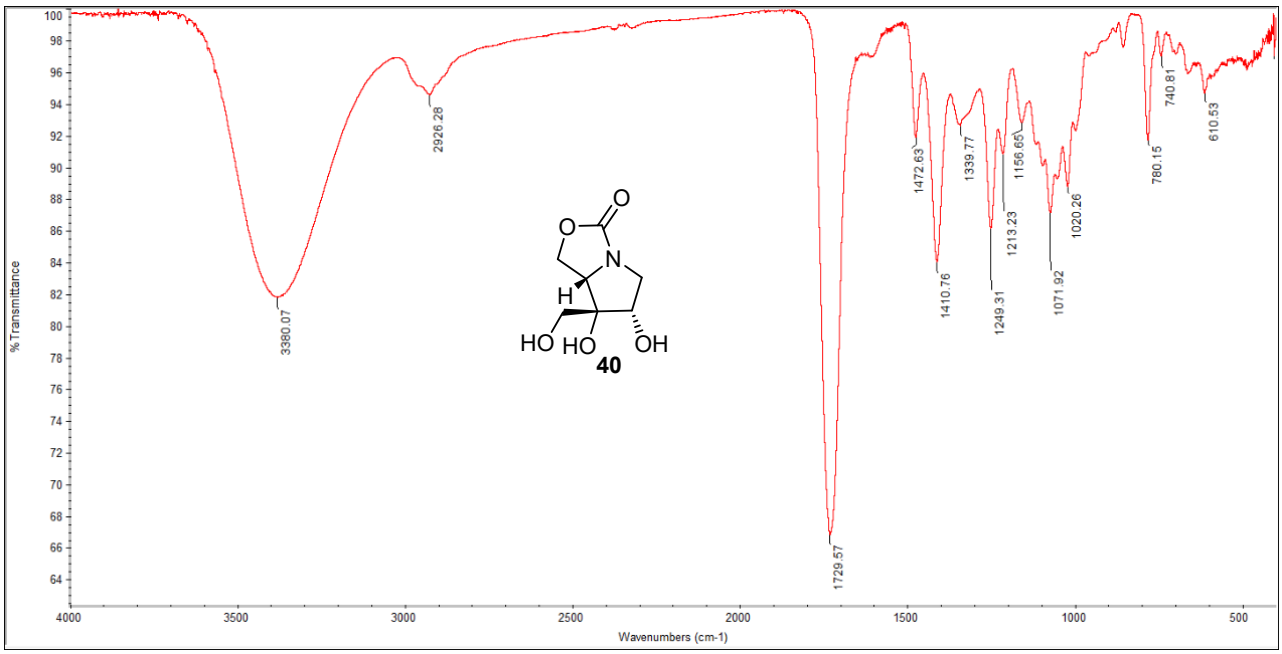
Current Data Parameters
NAME 500-2020
EXPNO 119
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191224
Time 1.22 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT D2O
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 49.27
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.59 usec
PLW1 20.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1299561 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


164.12
78.24
73.37
64.32
63.15
62.53
49.39
O
O
N
H
HO
HO
OH
40
Current Data Parameters
NAME 500-2020
EXPNO 90
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191222
Time 14.14 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 240
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


73.37
64.32
63.15
62.53
49.39
O
O
N
H
HO
HO
OH
40
Current Data Parameters
NAME 500-2020
EXPNO 91
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191222
Time 14.22 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG deptsp135
TD 65536
SOLVENT D2O
NS 40
DS 4
SWH 20161.291 Hz
FIDRES 0.615274 Hz
AQ 1.6252928 sec
RG 192.89
DW 24.800 usec
DE 18.00 usec
TE 298.2 K
CNST2 145.0000000
D1 2.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
DELTA 0.00001248 sec
TD0 1
SFO1 125.7678486 MHz
NUC1 13C
P1 9.80 usec
P13 2000.00 usec
PLW0 0 W
PLW1 57.00000000 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 8.36410046 W
SFO2 500.1315995 MHz
NUC2 1H
CPDPRG[2 waltz16
P3 10.70 usec
P4 21.40 usec
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


%Transmittance
Wavenumbers (cm-1)
3380.07
2926.26
1729.57
1472.63
1410.76
1339.77
1249.31
1213.23
1156.65
1071.92
1020.26
780.15
740.81
610.53
HO
HO
OH
40

Section II: X-Ray Crystallographic Data

Compound 15

Structure deposited at the Cambridge Crystallographic Data Centre (**CCDC 2162560**)

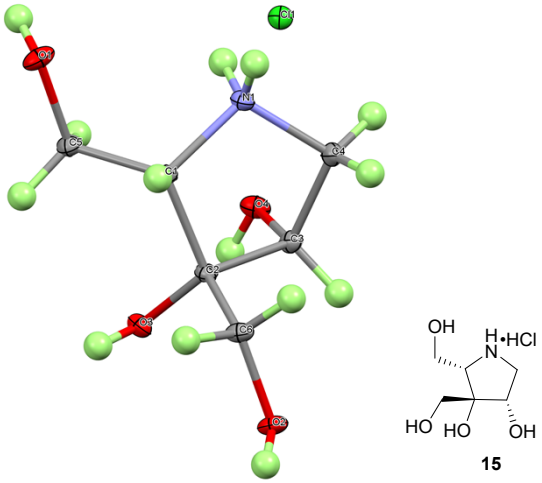


Fig 1. X-Ray ellipsoid plots of **15·HCl**

Table 1-1 Crystal data and structure refinement for compound 15·HCl.

Identification code	mx8226
Empirical formula	$C_6H_{14}ClNO_4$
Formula weight	199.63
Temperature/K	100.00(10)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	6.03620(10)
b/Å	11.5263(3)
c/Å	12.3169(3)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	856.95(3)
Z	4
ρ_{calc}g/cm^3	1.547
μ/mm^{-1}	0.423
F(000)	424.0
Crystal size/mm^3	0.35 × 0.3 × 0.15
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.84 to 61.218
Index ranges	-8 ≤ h ≤ 8, -15 ≤ k ≤ 15, -16 ≤ l ≤ 17
Reflections collected	20072
Independent reflections	2374 [R_{int} = 0.0251, R_{sigma} = 0.0160]
Data/restraints/parameters	2374/0/111

Goodness-of-fit on F^2	1.080
Final R indexes [I>=2σ (I)]	R_1 = 0.0199, wR_2 = 0.0514
Final R indexes [all data]	R_1 = 0.0204, wR_2 = 0.0515
Largest diff. peak/hole / e $Å^{-3}$	0.30/-0.17
Flack parameter	-0.149(14)

Table 1-2 Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for compound 15·HCl. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Cl1	830.0(5)	9561.7(3)	4275.8(2)	10.37(8)
O1	1485.8(18)	8858.3(9)	948.3(9)	14.2(2)
O2	2819.5(16)	4466.7(9)	3500.7(8)	12.4(2)
O3	3814.3(16)	5507.3(9)	1520.6(8)	11.37(19)
O4	7403.9(17)	6733.5(9)	2030.0(8)	12.4(2)
N1	4201(2)	8267.3(10)	2804.1(9)	9.8(2)
C1	2657(2)	7443.3(12)	2223.0(11)	9.4(2)
C2	3593(2)	6205.9(11)	2459.9(11)	8.7(2)
C3	5942(2)	6451.6(12)	2895.1(11)	10.2(2)
C4	5649(2)	7558.7(12)	3536.8(11)	11.4(2)
C5	2511(2)	7742.8(12)	1031.6(11)	11.6(3)
C6	2100(2)	5623.7(12)	3299.7(11)	10.9(3)

Table 1-3 Anisotropic Displacement Parameters (Å²×10³) for 15·HCl. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1	9.38(13)	10.53(14)	11.20(13)	-0.30(11)	1.07(11)	0.44(11)
O1	15.2(5)	10.6(5)	16.7(5)	4.7(4)	3.7(4)	4.3(4)
O2	11.5(4)	7.0(5)	18.6(5)	2.8(4)	2.0(4)	0.1(4)
O3	9.9(4)	10.5(4)	13.7(4)	-3.5(4)	-0.7(3)	-0.2(4)
O4	9.3(4)	10.4(5)	17.4(5)	-1.4(4)	3.1(4)	-0.9(4)
N1	10.0(5)	7.5(5)	11.9(5)	-1.2(4)	0.2(5)	0.0(5)
C1	7.5(5)	7.5(6)	13.1(6)	-0.6(5)	0.3(5)	-0.3(5)
C2	7.6(6)	6.7(6)	11.8(6)	-1.3(5)	-0.6(5)	0.8(5)
C3	7.6(6)	9.7(6)	13.4(6)	0.7(4)	-1.0(5)	0.5(5)
C4	10.9(6)	10.7(6)	12.6(6)	0.1(5)	-2.5(5)	-0.2(5)
C5	12.7(6)	9.1(6)	12.9(6)	0.4(5)	-0.4(5)	2.2(5)
C6	9.3(6)	7.4(6)	16.0(6)	1.0(5)	2.0(5)	0.5(5)

Table 1-4 Bond Lengths for 15·HCl.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C5	1.4305(17)	C1	C2	1.5616(18)
O2	C6	1.4242(16)	C1	C5	1.5100(19)
O3	C2	1.4159(16)	C2	C3	1.5418(19)
O4	C3	1.4213(16)	C2	C6	1.5273(18)
N1	C1	1.5110(18)	C3	C4	1.5113(19)
N1	C4	1.4984(18)			

Table 1-5 Bond Angles for 15·HCl.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C4	N1	C1	107.60(10)	C6	C2	C1	108.32(11)
N1	C1	C2	105.21(10)	C6	C2	C3	112.83(11)
C5	C1	N1	110.65(11)	O4	C3	C2	110.63(11)
C5	C1	C2	114.31(11)	O4	C3	C4	105.77(11)
O3	C2	C1	113.63(10)	C4	C3	C2	103.27(11)
O3	C2	C3	107.57(10)	N1	C4	C3	102.32(10)
O3	C2	C6	111.05(11)	O1	C5	C1	107.49(11)
C3	C2	C1	103.29(10)	O2	C6	C2	110.44(11)

Table 1-6 Hydrogen Bonds for 15·HCl.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
O1	H1	Cl1[1]	0.82	2.37	3.1916(11)	177.4
O2	H2	O1[2]	0.82	2.02	2.7760(14)	152.2
O3	H3	Cl1[2]	0.82	2.35	3.1637(10)	171.1
O4	H4	Cl1[3]	0.82	2.37	3.1606(11)	162.7
N1	H1A	O2[4]	0.89	1.90	2.7798(15)	169.8
N1	H1B	Cl1	0.89	2.25	3.1069(12)	161.7

[1]1/2-X,2-Y,-1/2+Z; [2]-X,-1/2+Y,1/2-Z; [3]1-X,-1/2+Y,1/2-Z; [4]1-X,1/2+Y,1/2-Z

Table 1-7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 15·HCl.

Atom	*x*	*y*	*z*	U(eq)
H1	2136.51	9274.46	512.42	21

H2	1762.86	4072.3	3697.86	19
H3	2551.7	5334	1336	17
H4	7575.88	6163.09	1641.09	19
H1A	5025.25	8653.05	2325.75	12
H1B	3426.54	8778.32	3190.46	12
H1C	1177.74	7507.42	2545.11	11
H3A	6506.11	5814.79	3343.84	12
H4A	4931.12	7411.43	4227.78	14
H4B	7059.11	7938.55	3664.46	14
H5A	3979.42	7762.11	712.5	14
H5B	1635.77	7166.24	650.5	14
H6A	2134.17	6063.6	3970.83	13
H6B	585.45	5614.09	3036.5	13

Compound 19

Structure deposited at the Cambridge Crystallographic Data Centre (**CCDC** 2162538)

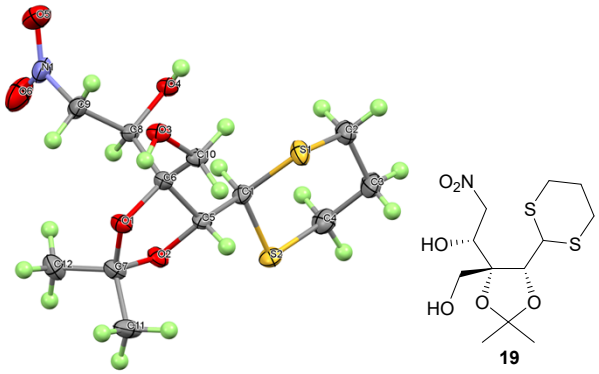


Fig 2. X-Ray ellipsoid plots of **19**

Table 2-1 Crystal data and structure refinement for compound 19.

Identification code	sa5357	
Empirical formula	C12 H21 N O6 S2	
Formula weight	339.42	
Temperature	173.15 K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 6.0310(12) Å	α= 90°.
	b = 9.2468(18) Å	β= 90°.
	c = 28.590(6) Å	γ= 90°.
Volume	1594.4(5) $Å^3$	
Z	4	
Density (calculated)	1.414 Mg/m^3	

Absorption coefficient	0.359 mm^{-1}
F(000)	720
Crystal size	0.8 x 0.254 x 0.197 mm^3
Theta range for data collection	3.452 to 27.480°.
Index ranges	-7<=h<=7, -12<=k<=11, -36<=l<=37
Reflections collected	10459
Independent reflections	3621 [R(int) = 0.0363]
Completeness to theta = 25.242°	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.43003
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3621 / 0 / 193
Goodness-of-fit on F^2	1.088
Final R indices [I>2sigma(I)]	R1 = 0.0371, wR2 = 0.0939
R indices (all data)	R1 = 0.0381, wR2 = 0.0950
Absolute structure parameter	0.02(5)
Extinction coefficient	n/a
Largest diff. peak and hole	0.397 and -0.230 e.$Å^{-3}$

Table 2-2 Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for compound 19. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S1	1276(1)	9449(1)	3826(1)	30(1)
S2	711(1)	7990(1)	2883(1)	29(1)
O1	5160(3)	5154(2)	3954(1)	24(1)
O2	2880(3)	5544(2)	3328(1)	24(1)
O3	5858(3)	6717(2)	4779(1)	23(1)
O4	49(3)	6575(2)	4490(1)	21(1)
O5	-260(5)	3868(3)	5290(1)	53(1)
O6	-541(5)	2911(3)	4603(1)	52(1)
N1	354(4)	3712(3)	4885(1)	33(1)
C1	935(4)	7749(3)	3513(1)	22(1)
C2	-1193(5)	10394(3)	3640(1)	32(1)
C3	-1321(5)	10635(3)	3115(1)	33(1)
C4	-1596(5)	9239(3)	2840(1)	34(1)
C5	3027(4)	6856(3)	3594(1)	20(1)
C6	3618(4)	6266(3)	4084(1)	19(1)
C7	4626(4)	4628(3)	3493(1)	25(1)
C8	1624(4)	5553(3)	4331(1)	19(1)
C9	2338(4)	4545(3)	4726(1)	27(1)
C10	4872(4)	7365(3)	4379(1)	21(1)
C11	6674(5)	4771(4)	3188(1)	34(1)
C12	3764(5)	3093(3)	3516(1)	33(1)

Table 2-3 Bond lengths [Å] and angles [°] for compound 19.

S1-C1	1.820(3)
S1-C2	1.807(3)
S2-C1	1.820(2)
S2-C4	1.813(3)
O1-C6	1.435(3)
O1-C7	1.441(3)
O2-C5	1.434(3)
O2-C7	1.432(3)

O3-H3	0.8400
O3-C10	1.422(3)
O4-H4	0.8400
O4-C8	1.414(3)
O5-N1	1.224(3)
O6-N1	1.221(4)
N1-C9	1.493(3)
C1-H1	1.0000
C1-C5	1.525(3)
C2-H2A	0.9900
C2-H2B	0.9900
C2-C3	1.518(4)
C3-H3A	0.9900
C3-H3B	0.9900
C3-C4	1.520(4)
C4-H4A	0.9900
C4-H4B	0.9900
C5-H5	1.0000
C5-C6	1.546(3)
C6-C8	1.543(3)
C6-C10	1.522(3)
C7-C11	1.517(4)
C7-C12	1.513(4)
C8-H8	1.0000
C8-C9	1.527(3)
C9-H9A	0.9900
C9-H9B	0.9900
C10-H10A	0.9900
C10-H10B	0.9900
C11-H11A	0.9800
C11-H11B	0.9800
C11-H11C	0.9800
C12-H12A	0.9800
C12-H12B	0.9800
C12-H12C	0.9800
C2-S1-C1	100.35(13)
C4-S2-C1	101.68(13)
C6-O1-C7	109.48(18)

C7-O2-C5	106.30(18)
C10-O3-H3	108.7
C8-O4-H4	109.5
O5-N1-C9	118.0(3)
O6-N1-O5	124.2(3)
O6-N1-C9	117.8(2)
S1-C1-S2	112.86(13)
S1-C1-H1	110.1
S2-C1-H1	110.1
C5-C1-S1	107.46(16)
C5-C1-S2	106.03(17)
C5-C1-H1	110.1
S1-C2-H2A	108.8
S1-C2-H2B	108.8
H2A-C2-H2B	107.7
C3-C2-S1	113.8(2)
C3-C2-H2A	108.8
C3-C2-H2B	108.8
C2-C3-H3A	109.0
C2-C3-H3B	109.0
C2-C3-C4	113.1(2)
H3A-C3-H3B	107.8
C4-C3-H3A	109.0
C4-C3-H3B	109.0
S2-C4-H4A	108.5
S2-C4-H4B	108.5
C3-C4-S2	115.0(2)
C3-C4-H4A	108.5
C3-C4-H4B	108.5
H4A-C4-H4B	107.5
O2-C5-C1	109.10(19)
O2-C5-H5	108.2
O2-C5-C6	101.26(18)
C1-C5-H5	108.2
C1-C5-C6	121.2(2)
C6-C5-H5	108.2
O1-C6-C5	99.69(18)
O1-C6-C8	108.50(19)

O1-C6-C10	107.45(18)
C8-C6-C5	112.74(18)
C10-C6-C5	112.4(2)
C10-C6-C8	114.8(2)
O1-C7-C11	108.3(2)
O1-C7-C12	110.7(2)
O2-C7-O1	105.44(19)
O2-C7-C11	110.9(2)
O2-C7-C12	108.4(2)
C12-C7-C11	112.8(2)
O4-C8-C6	112.66(19)
O4-C8-H8	106.8
O4-C8-C9	111.09(19)
C6-C8-H8	106.8
C9-C8-C6	112.35(19)
C9-C8-H8	106.8
N1-C9-C8	108.3(2)
N1-C9-H9A	110.0
N1-C9-H9B	110.0
C8-C9-H9A	110.0
C8-C9-H9B	110.0
H9A-C9-H9B	108.4
O3-C10-C6	111.8(2)
O3-C10-H10A	109.2
O3-C10-H10B	109.3
C6-C10-H10A	109.3
C6-C10-H10B	109.2
H10A-C10-H10B	107.9
C7-C11-H11A	109.5
C7-C11-H11B	109.5
C7-C11-H11C	109.5
H11A-C11-H11B	109.5
H11A-C11-H11C	109.5
H11B-C11-H11C	109.5
C7-C12-H12A	109.5
C7-C12-H12B	109.5
C7-C12-H12C	109.5
H12A-C12-H12B	109.5

H12A-C12-H12C	109.5
H12B-C12-H12C	109.5

Table 2-4 Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound 19. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S1	45(1)	20(1)	24(1)	-3(1)	-9(1)	4(1)
S2	41(1)	31(1)	15(1)	2(1)	-2(1)	3(1)
O1	23(1)	25(1)	22(1)	-4(1)	0(1)	5(1)
O2	29(1)	22(1)	20(1)	-6(1)	-2(1)	3(1)
O3	20(1)	32(1)	19(1)	-3(1)	-2(1)	3(1)
O4	18(1)	25(1)	21(1)	-2(1)	0(1)	2(1)
O5	64(2)	46(1)	49(2)	3(1)	29(1)	-7(1)
O6	60(2)	45(1)	51(2)	11(1)	-3(1)	-26(1)
N1	37(1)	26(1)	37(1)	11(1)	6(1)	-2(1)
C1	29(1)	21(1)	16(1)	1(1)	-2(1)	0(1)
C2	43(2)	25(1)	26(1)	2(1)	-2(1)	8(1)
C3	46(2)	28(1)	26(1)	7(1)	-4(1)	7(1)
C4	39(1)	38(2)	25(1)	7(1)	-5(1)	6(1)
C5	26(1)	20(1)	15(1)	-4(1)	1(1)	-1(1)
C6	20(1)	18(1)	19(1)	-1(1)	1(1)	1(1)
C7	25(1)	24(1)	26(1)	-7(1)	2(1)	2(1)
C8	20(1)	20(1)	17(1)	0(1)	2(1)	1(1)
C9	27(1)	25(1)	29(1)	10(1)	2(1)	2(1)
C10	20(1)	25(1)	18(1)	-2(1)	-1(1)	-2(1)
C11	33(1)	40(2)	29(1)	-12(1)	10(1)	-2(1)
C12	34(1)	23(1)	42(2)	-6(1)	0(1)	2(1)

Table 2-5 Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for compound 19.

	x	y	z	U(eq)

H3	7077	6351	4701	35
H4	616	7086	4701	32
H1	-391	7219	3635	26
H2A	-2509	9833	3740	38
H2B	-1251	11344	3799	38
H3A	48	11128	3010	40
H3B	-2588	11281	3046	40
H4A	-1821	9486	2506	41
H4B	-2953	8744	2951	41
H5	4323	7423	3475	24
H8	861	4940	4092	23
H9A	3501	3874	4614	32
H9B	2950	5115	4990	32
H10A	6043	7820	4186	25
H10B	3838	8135	4481	25
H11A	6385	4337	2882	51
H11B	7921	4273	3338	51
H11C	7038	5797	3149	51
H12A	2372	3075	3693	49
H12B	4860	2477	3672	49
H12C	3501	2734	3199	49

Table 2-6 Torsion angles [°] for compound 19.

S1-C1-C5-O2	-176.35(15)
S1-C1-C5-C6	66.8(2)
S1-C2-C3-C4	67.2(3)
S2-C1-C5-O2	-55.4(2)
S2-C1-C5-C6	-172.25(18)
O1-C6-C8-O4	177.59(17)
O1-C6-C8-C9	51.2(3)
O1-C6-C10-O3	-57.5(2)
O2-C5-C6-O1	40.4(2)
O2-C5-C6-C8	-74.4(2)
O2-C5-C6-C10	153.96(18)
O4-C8-C9-N1	62.9(3)

O5-N1-C9-C8	-118.2(3)
O6-N1-C9-C8	62.4(3)
C1-S1-C2-C3	-61.0(2)
C1-S2-C4-C3	55.6(2)
C1-C5-C6-O1	161.1(2)
C1-C5-C6-C8	46.3(3)
C1-C5-C6-C10	-85.3(3)
C2-S1-C1-S2	57.92(17)
C2-S1-C1-C5	174.45(17)
C2-C3-C4-S2	-64.0(3)
C4-S2-C1-S1	-55.68(17)
C4-S2-C1-C5	-173.06(18)
C5-O2-C7-O1	22.0(2)
C5-O2-C7-C11	-95.1(2)
C5-O2-C7-C12	140.6(2)
C5-C6-C8-O4	-73.0(2)
C5-C6-C8-C9	160.6(2)
C5-C6-C10-O3	-166.13(18)
C6-O1-C7-O2	5.8(2)
C6-O1-C7-C11	124.6(2)
C6-O1-C7-C12	-111.3(2)
C6-C8-C9-N1	-169.9(2)
C7-O1-C6-C5	-28.4(2)
C7-O1-C6-C8	89.7(2)
C7-O1-C6-C10	-145.7(2)
C7-O2-C5-C1	-167.82(19)
C7-O2-C5-C6	-38.9(2)
C8-C6-C10-O3	63.3(3)
C10-C6-C8-O4	57.4(3)
C10-C6-C8-C9	-69.0(3)

Table 2-7 Hydrogen bonds for compound 19 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O3-H3...O4#1	0.84	1.90	2.663(2)	150.0

O4-H4...O3#2	0.84	1.86	2.665(3)	160.5

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z #2 x-1/2,-y+3/2,-z+1

Compound 25

Structure deposited at the Cambridge Crystallographic Data Centre (**CCDC** 2162555)

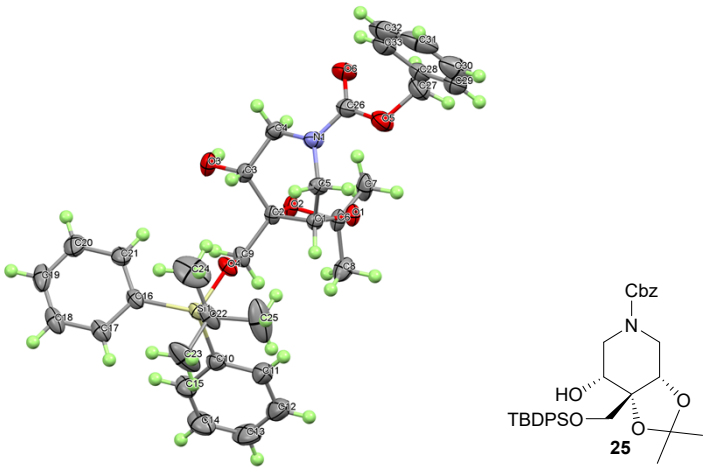


Fig 3. X-Ray ellipsoid plots of **25**

Table 3-1 Crystal data and structure refinement for compound 25.

Identification code	tx1201
Empirical formula	$C_{33}H_{41}NO_6Si$
Formula weight	575.76
Temperature/K	169.99(13)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	11.55970(10)
b/Å	10.58310(10)
c/Å	13.69520(10)
α/°	90
β/°	108.3990(10)
γ/°	90
Volume/Å^3	1589.79(3)
Z	2
ρ_{calc}g/cm^3	1.203
μ/mm^{-1}	1.001
F(000)	616.0

Crystal size/mm^3	0.28 × 0.25 × 0.2
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	6.802 to 150.504
Index ranges	-14 ≤ h ≤ 13, -12 ≤ k ≤ 12, -16 ≤ l ≤ 16
Reflections collected	30069
Independent reflections	6247 [R_{int} = 0.0237, R_{sigma} = 0.0166]
Data/restraints/parameters	6247/1/376
Goodness-of-fit on F^2	1.044
Final R indexes [I>=2σ (I)]	R_1 = 0.0296, wR_2 = 0.0748
Final R indexes [all data]	R_1 = 0.0323, wR_2 = 0.0786
Largest diff. peak/hole / e Å^{-3}	0.21/-0.20
Flack parameter	-0.003(7)

Table 3-2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for compound 25. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Si1	5196.6(4)	5004.3(6)	2667.9(4)	28.41(12)
O1	10225.7(11)	4873.1(13)	4755.0(11)	29.6(3)
O2	9518.8(11)	6788.1(12)	4114.8(11)	29.0(3)
O3	7811.8(13)	8202.6(14)	4785.5(14)	37.7(4)
O4	6496.4(12)	5416.0(14)	3535.3(10)	31.6(3)
O5	10894.7(17)	4338.2(17)	7528.2(14)	51.1(5)
O6	11132.2(16)	6461.3(19)	7795.8(13)	51.1(4)
N1	9558.5(16)	5686.5(17)	6496.3(13)	33.1(4)
C1	8985.4(15)	4870.3(18)	4737.7(14)	24.4(3)
C2	8500.9(16)	6189.1(18)	4315.5(14)	24.7(4)
C3	8158.2(17)	6961.2(19)	5127.0(16)	29.2(4)
C4	9178.6(19)	6970(2)	6152.7(17)	34.0(4)
C5	8979.6(18)	4636.2(18)	5827.4(15)	28.7(4)
C6	10401.3(17)	5851.1(18)	4100.0(16)	29.4(4)
C7	11662.2(19)	6386(2)	4585(2)	45.2(6)
C8	10204(2)	5353(2)	3019.2(19)	42.7(5)
C9	7432.6(17)	6130(2)	3323.1(15)	29.4(4)
C10	5511.3(19)	4617(2)	1441.7(16)	35.2(5)
C11	6417(2)	3730(3)	1447(2)	48.1(6)
C12	6700(3)	3440(3)	561(2)	62.2(8)
C13	6076(3)	4038(4)	-346(2)	70.6(10)
C14	5201(3)	4931(4)	-372.2(19)	65.6(8)
C15	4924(2)	5222(3)	519.9(17)	47.1(6)

C16	4092.5(18)	6351(2)	2473.7(15)	33.2(4)
C17	2901.9(19)	6276(3)	1781.4(17)	40.5(5)
C18	2068(2)	7243(3)	1687.2(19)	46.8(6)
C19	2400(2)	8320(3)	2276(2)	51.1(7)
C20	3570(3)	8433(3)	2959.9(19)	50.5(6)
C21	4400(2)	7449(3)	3056.0(17)	40.1(5)
C22	4662(2)	3627(3)	3287(2)	45.3(6)
C23	3459(3)	3099(4)	2593(3)	72.8(10)
C24	4466(4)	4143(5)	4282(3)	87.6(13)
C25	5611(3)	2593(4)	3576(4)	89.9(14)
C26	10584(2)	5562(2)	7312.6(17)	38.4(5)
C27	11956(2)	4089(3)	8421(2)	52.9(6)
C28	11558(2)	3457(2)	9238.5(17)	42.6(5)
C29	11861(3)	2206(3)	9508(2)	54.1(7)
C30	11477(3)	1630(4)	10266(2)	71.6(10)
C31	10785(3)	2300(5)	10738(2)	77.3(12)
C32	10487(3)	3532(4)	10477(2)	70.1(10)
C33	10867(2)	4101(3)	9742(2)	55.8(7)

Table 3-3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound 25. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Si1	19.2(2)	39.1(3)	25.2(2)	2.1(2)	4.53(17)	-2.5(2)
O1	22.5(6)	24.6(7)	43.8(7)	6.0(6)	13.5(5)	2.5(6)
O2	20.5(6)	21.3(7)	48.8(8)	3.3(6)	16.0(6)	-0.5(5)
O3	26.4(7)	23.5(7)	64.4(10)	-0.4(7)	16.0(7)	2.3(6)
O4	19.5(6)	45.0(9)	28.5(6)	5.2(6)	4.9(5)	-6.1(6)
O5	54.3(11)	44.6(10)	39.9(9)	3.3(7)	-5.6(8)	3.0(8)
O6	45.7(9)	53.4(11)	44.5(9)	-14.0(8)	0.5(7)	-6.4(8)
N1	34.9(9)	31.4(9)	30.5(8)	-4.1(7)	6.7(7)	-4.5(7)
C1	20.0(7)	21.5(9)	31.4(9)	-1.8(7)	7.6(6)	-3.0(7)
C2	17.6(8)	22.2(9)	35.2(9)	2.0(7)	9.4(7)	-3.0(7)
C3	23.0(9)	23.8(9)	42.5(11)	-2.6(8)	12.8(8)	-2.3(7)
C4	34.1(10)	28.1(11)	40.4(11)	-7.6(8)	12.6(9)	-3.4(8)
C5	29.3(9)	24.6(10)	31.9(10)	-0.2(7)	9.3(8)	-6.0(7)
C6	24.3(9)	20.9(9)	46.5(11)	4.3(8)	16.3(8)	2.1(7)
C7	23.2(10)	30.1(11)	83.1(18)	2.8(11)	17.8(11)	0.6(9)
C8	48.5(13)	40.4(13)	49.3(13)	4.0(10)	30.0(11)	5.5(10)
C9	23.3(9)	32.3(11)	32.5(9)	6.8(8)	8.7(8)	-1.0(8)

C10	29.0(10)	43.1(12)	32.7(10)	-6.8(8)	8.7(8)	-4.0(8)
C11	42.7(13)	51.4(15)	49.2(14)	-9.2(11)	13.1(11)	4.9(11)
C12	51.9(15)	74(2)	64.0(18)	-25.0(16)	23.0(14)	3.4(15)
C13	60.2(17)	110(3)	50.0(16)	-30.5(18)	29.2(14)	-6.7(18)
C14	54.7(15)	109(3)	34.0(12)	2.2(16)	15.9(11)	3.0(19)
C15	38.7(11)	69.4(18)	34.0(10)	2.2(11)	12.6(9)	6.1(11)
C16	24.4(9)	50.6(13)	25.3(9)	4.4(9)	8.7(7)	3.0(9)
C17	27.9(10)	57.1(15)	34.4(10)	8.4(10)	6.9(8)	2.0(10)
C18	27.8(11)	72.1(18)	41.0(12)	19.8(12)	12.0(9)	9.4(11)
C19	46.7(14)	69.7(18)	44.2(13)	17.5(13)	24.8(11)	26.9(13)
C20	58.2(15)	60.5(17)	36.2(12)	-1.1(11)	19.9(11)	15.8(13)
C21	35.7(11)	56.0(15)	27.7(10)	-3.1(9)	9.0(8)	8.2(10)
C22	24.9(10)	58.1(15)	47.0(13)	17.2(11)	3.0(9)	-7.9(10)
C23	46.7(16)	80(2)	74(2)	21.6(17)	-6.0(14)	-33.8(16)
C24	88(2)	132(4)	51.5(17)	24(2)	34.8(17)	-27(3)
C25	50.0(17)	70(2)	146(4)	63(2)	26(2)	4.0(15)
C26	37.1(11)	45.3(13)	31.4(10)	-4.2(9)	8.7(9)	-2.9(10)
C27	42.7(13)	63.4(17)	44.5(13)	5.4(12)	2.2(10)	7.2(12)
C28	36.0(11)	49.1(14)	33.4(11)	-4.4(10)	-2.2(9)	-0.1(10)
C29	54.3(15)	53.3(16)	46.1(14)	-6.3(12)	3.8(12)	3.2(12)
C30	81(2)	62(2)	52.8(16)	8.9(15)	-4.8(16)	-23.8(17)
C31	66(2)	119(3)	39.7(15)	-7.5(18)	6.0(14)	-47(2)
C32	48.7(16)	112(3)	45.7(15)	-26.3(18)	8.9(12)	-18.8(18)
C33	42.7(13)	68.5(18)	46.9(14)	-17.1(13)	0.8(11)	0.1(13)

Table 3-4 Bond Lengths for compound 25.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Si1	O4	1.6529(14)	C10	C11	1.405(3)
Si1	C10	1.872(2)	C10	C15	1.387(3)
Si1	C16	1.876(2)	C11	C12	1.387(4)
Si1	C22	1.886(2)	C12	C13	1.378(5)
O1	C1	1.426(2)	C13	C14	1.376(5)
O1	C6	1.426(2)	C14	C15	1.392(3)
O2	C2	1.437(2)	C16	C17	1.406(3)
O2	C6	1.427(2)	C16	C21	1.390(3)
O3	C3	1.410(2)	C17	C18	1.384(4)
O4	C9	1.423(2)	C18	C19	1.378(4)
O5	C26	1.351(3)	C19	C20	1.386(4)
O5	C27	1.457(3)	C20	C21	1.394(4)

O6	C26	1.217(3)	C22	C23	1.522(3)
N1	C4	1.459(3)	C22	C24	1.550(5)
N1	C5	1.461(3)	C22	C25	1.511(4)
N1	C26	1.355(3)	C27	C28	1.495(4)
C1	C2	1.546(3)	C28	C29	1.389(4)
C1	C5	1.515(3)	C28	C33	1.388(4)
C2	C3	1.529(3)	C29	C30	1.392(5)
C2	C9	1.522(3)	C30	C31	1.373(6)
C3	C4	1.523(3)	C31	C32	1.367(6)
C6	C7	1.508(3)	C32	C33	1.359(5)
C6	C8	1.519(3)			

Table 3-5 Bond Angles for compound 25.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O4	Si1	C10	108.20(8)	C11	C10	Si1	119.81(18)
O4	Si1	C16	108.97(9)	C15	C10	Si1	122.50(18)
O4	Si1	C22	103.49(9)	C15	C10	C11	117.6(2)
C10	Si1	C16	110.81(9)	C12	C11	C10	121.7(3)
C10	Si1	C22	114.73(12)	C13	C12	C11	119.0(3)
C16	Si1	C22	110.25(10)	C14	C13	C12	120.7(3)
C6	O1	C1	109.46(14)	C13	C14	C15	120.0(3)
C6	O2	C2	109.16(14)	C10	C15	C14	120.9(3)
C9	O4	Si1	124.80(12)	C17	C16	Si1	122.01(19)
C26	O5	C27	116.9(2)	C21	C16	Si1	120.97(16)
C4	N1	C5	118.31(16)	C21	C16	C17	116.9(2)
C26	N1	C4	116.92(18)	C18	C17	C16	121.7(2)
C26	N1	C5	123.47(19)	C19	C18	C17	120.0(2)
O1	C1	C2	104.35(14)	C18	C19	C20	119.9(2)
O1	C1	C5	107.52(14)	C19	C20	C21	119.6(3)
C5	C1	C2	113.72(15)	C16	C21	C20	121.8(2)
O2	C2	C1	104.35(13)	C23	C22	Si1	111.57(18)
O2	C2	C3	108.59(15)	C23	C22	C24	108.7(3)
O2	C2	C9	109.26(15)	C24	C22	Si1	106.4(2)
C3	C2	C1	110.91(15)	C25	C22	Si1	111.27(18)
C9	C2	C1	113.14(15)	C25	C22	C23	110.0(3)
C9	C2	C3	110.34(15)	C25	C22	C24	108.7(3)
O3	C3	C2	111.63(17)	O5	C26	N1	112.13(19)
O3	C3	C4	110.89(17)	O6	C26	O5	125.0(2)
C4	C3	C2	111.77(16)	O6	C26	N1	122.9(2)

N1	C4	C3	111.01(16)	O5	C27	C28	109.5(2)
N1	C5	C1	110.47(15)	C29	C28	C27	120.9(2)
O1	C6	O2	104.54(14)	C33	C28	C27	120.9(3)
O1	C6	C7	107.78(18)	C33	C28	C29	118.2(3)
O1	C6	C8	110.72(17)	C28	C29	C30	120.2(3)
O2	C6	C7	109.19(17)	C31	C30	C29	119.7(3)
O2	C6	C8	111.52(17)	C32	C31	C30	120.4(3)
C7	C6	C8	112.72(19)	C33	C32	C31	120.2(3)
O4	C9	C2	107.13(14)	C32	C33	C28	121.4(3)

Table 3-6 Hydrogen Bonds for compound 25.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
O3	H3	O1[1]	0.82	2.02	2.7869(19)	156.4

[1]2-X,1/2+Y,1-Z

Table 3-7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound 25.

Atom	*x*	*y*	*z*	U(eq)
H3	8413.88	8600.68	4768.88	57
H1	8527.76	4206.8	4276.98	29
H3A	7450.25	6552.65	5239.74	35
H4A	9869.73	7436.4	6081.08	41
H4B	8899.94	7394.6	6665.08	41
H5A	8146.25	4541.29	5830.67	34
H5B	9413.6	3859.14	6085.27	34
H7A	11828.41	7002.38	4132.34	68
H7B	12251.6	5717.39	4702.68	68
H7C	11709.08	6778.75	5228.08	68
H8A	9461.55	4873.64	2799.33	64
H8B	10876.03	4820.9	3017.38	64
H8C	10150.05	6049.51	2557.83	64
H9A	7143.93	6974.94	3098.02	35
H9B	7679.36	5726.8	2784.31	35
H11	6837.2	3327.03	2059.74	58
H12	7302.97	2850.08	579.17	75
H13	6247.73	3836.42	-946.19	85
H14	4794.88	5338.97	-986.13	79

H15	4335.96	5831.39	497.5	57
H17	2668.29	5557	1376.17	49
H18	1284.32	7167.29	1226.69	56
H19	1839.72	8969.49	2214.11	61
H20	3800.8	9162.52	3352.48	61
H21	5179.69	7528.14	3522.57	48
H23A	2842.01	3740.01	2466.02	109
H23B	3222.82	2388.26	2922.64	109
H23C	3553.74	2834.1	1951.86	109
H24A	5207.53	4521.63	4709.87	131
H24B	4240.13	3461.1	4647.96	131
H24C	3829.02	4764.72	4105.91	131
H25A	5717.94	2244.08	2962.77	135
H25B	5348.33	1941.37	3945.03	135
H25C	6370.86	2938.14	4002.28	135
H27A	12366.88	4876.15	8682.45	63
H27B	12523.65	3548.5	8225.28	63
H29	12321.03	1752.11	9180.79	65
H30	11686.92	795.3	10451.98	86
H31	10518.97	1913.99	11238.71	93
H32	10022.37	3981.25	10802.51	84
H33	10659.14	4940.56	9573.46	67

Compound 32

Structure deposited at the Cambridge Crystallographic Data Centre (CCDC 2162552)

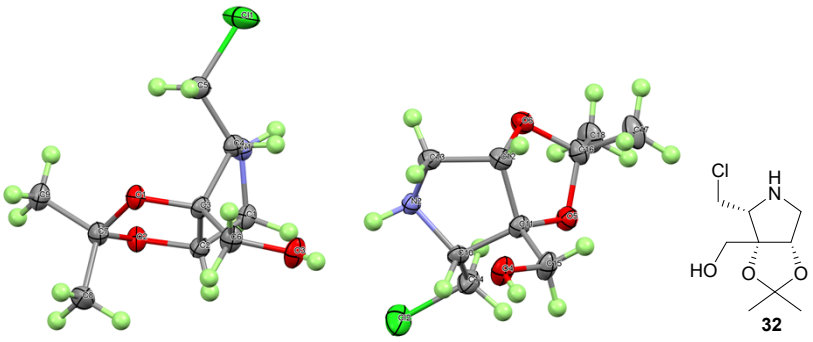


Fig 4. X-Ray ellipsoid plots of **32**

Table 4-1 Crystal data and structure refinement for compound 32.

Identification code	TX1796
Empirical formula	$C_9H_{16}ClNO_3$

Formula weight	221.68
Temperature/K	169.99(10)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	6.36560(4)
b/Å	17.75841(12)
c/Å	9.71022(7)
α/°	90
β/°	90.3845(6)
γ/°	90
Volume/Å^3	1097.647(13)
Z	4
ρ_{calc}g/cm^3	1.341
μ/mm^{-1}	2.968
F(000)	472.0
Crystal size/mm^3	0.33 × 0.31 × 0.25
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.108 to 150.302
Index ranges	$-7 \leq h \leq 5$, $-22 \leq k \leq 22$, $-11 \leq l \leq 12$
Reflections collected	12093
Independent reflections	4261 [R_{int} = 0.0211, R_{sigma} = 0.0170]
Data/restraints/parameters	4261/1/270
Goodness-of-fit on F^2	1.059
Final R indexes [I>=2σ (I)]	R_1 = 0.0273, wR_2 = 0.0710
Final R indexes [all data]	R_1 = 0.0293, wR_2 = 0.0722
Largest diff. peak/hole / e Å^{-3}	0.18/-0.34
Flack parameter	0.007(5)

Table 4-2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for compound 32. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Cl1	-421.9(12)	3136.5(4)	7163.1(7)	48.50(19)
O1	1834(2)	3434.2(8)	2830.6(16)	22.7(3)
O2	36(2)	4477.9(9)	2210.0(16)	23.5(3)
O3	5695(2)	4580.5(10)	4633.4(19)	30.2(4)
N1	-59(3)	4556.9(10)	5228(2)	21.3(4)
C1	718(3)	5163.0(12)	4328(2)	22.5(4)
C2	1653(3)	4748.4(13)	3126(2)	21.3(4)
C3	2555(3)	4015.0(12)	3738(2)	20.0(4)

C4	1539(3)	3953.5(12)	5179(2)	20.3(4)
C5	582(4)	3188.8(14)	5435(2)	29.9(5)
C6	4948(3)	3982.5(14)	3796(3)	26.2(5)
C7	787(3)	3781.4(13)	1676(2)	23.5(4)
C8	2306(4)	3905.6(16)	501(3)	34.0(5)
C9	-1049(3)	3293.8(14)	1249(3)	29.3(5)
Cl2	4336.7(12)	6864.5(3)	5306.1(7)	43.16(18)
O4	10725(2)	5447.1(10)	7790.6(18)	29.7(4)
O5	6999(2)	6642.1(9)	9564.0(17)	24.8(3)
O6	5152(3)	5619.4(9)	10231.3(16)	25.5(3)
N2	5001(3)	5443.0(10)	7236.4(19)	19.8(4)
C10	6522(3)	6074.6(12)	7255(2)	21.0(4)
C11	7639(3)	6040.4(12)	8681(2)	20.7(4)
C12	6763(3)	5324.2(12)	9363(2)	22.3(4)
C13	5832(3)	4869.7(12)	8193(2)	22.4(4)
C14	5434(4)	6821.1(14)	7022(2)	28.6(5)
C15	10019(3)	6067.2(15)	8573(3)	28.3(5)
C16	5923(4)	6321.4(13)	10727(2)	25.1(5)
C17	7430(4)	6212.1(16)	11927(3)	36.7(6)
C18	4103(4)	6824.3(16)	11102(3)	32.9(5)

Table 4-3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound 32. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1	64.9(4)	40.9(3)	40.1(4)	11.0(3)	22.6(3)	-1.2(3)
O1	23.2(7)	20.4(7)	24.4(7)	-4.5(6)	-2.1(6)	1.0(6)
O2	25.2(7)	21.9(7)	23.5(8)	-3.9(6)	-5.6(6)	1.6(6)
O3	13.9(7)	36.6(9)	40.0(9)	-13.7(7)	-1.4(6)	-0.2(6)
N1	15.9(8)	26.2(9)	21.8(9)	-3.9(7)	1.1(7)	2.4(7)
C1	20.6(10)	20.4(10)	26.5(10)	-3.3(8)	-4.2(8)	1.2(8)
C2	20.1(9)	21.3(9)	22.5(10)	0.1(8)	-1.4(8)	-2.6(8)
C3	14.9(9)	21.9(10)	23.2(10)	-3.6(8)	0.6(7)	-0.6(8)
C4	15.7(9)	23.3(10)	22.0(10)	-1.1(8)	-0.7(7)	1.2(8)
C5	33.4(11)	26.8(11)	29.5(11)	1.8(10)	7.6(9)	-1.3(10)
C6	16.1(10)	30.8(12)	31.8(12)	-9.4(9)	1.7(8)	1.6(8)
C7	23.6(10)	24.9(11)	22.1(10)	-3.7(9)	-0.8(8)	-0.3(8)
C8	36.7(13)	39.6(14)	25.7(12)	-6.3(10)	8.2(10)	-8.7(11)
C9	25.8(11)	29.8(12)	32.1(12)	-8.3(9)	-3.2(9)	-3.3(9)
Cl2	55.5(4)	32.5(3)	41.2(3)	7.2(3)	-20.7(3)	-2.6(3)

O4	13.8(7)	36.4(9)	38.8(9)	-12.8(7)	2.0(6)	0.5(6)
O5	24.5(7)	22.3(8)	27.6(8)	-4.9(6)	3.0(6)	0.5(6)
O6	24.3(7)	27.5(8)	24.7(8)	-4.0(6)	5.5(6)	0.1(6)
N2	16.6(8)	20.9(9)	22.0(9)	-2.6(7)	0.0(6)	-1.7(6)
C10	16.2(9)	24.6(10)	22.3(10)	-1.6(8)	3.3(7)	-2.7(8)
C11	14.2(9)	22.8(10)	25.2(10)	-3.6(9)	1.0(8)	0.1(8)
C12	22.1(10)	22.2(10)	22.7(10)	-0.5(8)	2.3(8)	4.2(8)
C13	20.3(10)	20.5(10)	26.4(10)	-2.1(8)	4.6(8)	0.1(8)
C14	31.0(11)	23.4(11)	31.4(11)	1.6(10)	-5.7(9)	-3.0(9)
C15	15.9(10)	34.0(12)	35.0(12)	-11.6(10)	0.2(9)	-2.1(9)
C16	25.9(11)	27.9(11)	21.7(11)	-4.4(8)	0.3(8)	3.2(9)
C17	36.2(13)	44.6(15)	29.1(12)	-6.2(11)	-9.9(10)	10.9(11)
C18	27.8(11)	36.6(12)	34.2(12)	-9.6(11)	2.1(9)	8.3(10)

Table 4-4 Bond Lengths for compound 32.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Cl1	C5	1.802(2)	Cl2	C14	1.804(2)
O1	C3	1.430(3)	O4	C15	1.413(3)
O1	C7	1.439(3)	O5	C11	1.431(3)
O2	C2	1.438(3)	O5	C16	1.442(3)
O2	C7	1.425(3)	O6	C12	1.431(3)
O3	C6	1.418(3)	O6	C16	1.422(3)
N1	C1	1.474(3)	N2	C10	1.482(3)
N1	C4	1.479(3)	N2	C13	1.474(3)
C1	C2	1.506(3)	C10	C11	1.553(3)
C2	C3	1.541(3)	C10	C14	1.512(3)
C3	C4	1.549(3)	C11	C12	1.540(3)
C3	C6	1.525(3)	C11	C15	1.520(3)
C4	C5	1.510(3)	C12	C13	1.511(3)
C7	C8	1.517(3)	C16	C17	1.517(3)
C7	C9	1.510(3)	C16	C18	1.509(3)

Table 4-5 Bond Angles for compound 32.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C3	O1	C7	108.42(16)	C11	O5	C16	108.23(16)
C7	O2	C2	105.92(16)	C16	O6	C12	105.85(16)
C1	N1	C4	106.04(16)	C13	N2	C10	106.45(16)

N1	C1	C2	103.81(16)	N2	C10	C11	106.04(17)
O2	C2	C1	110.94(17)	N2	C10	C14	111.29(17)
O2	C2	C3	102.68(16)	C14	C10	C11	111.96(18)
C1	C2	C3	105.20(17)	O5	C11	C10	111.99(17)
O1	C3	C2	104.76(17)	O5	C11	C12	104.69(16)
O1	C3	C4	111.81(17)	O5	C11	C15	107.81(17)
O1	C3	C6	108.16(17)	C12	C11	C10	104.54(16)
C2	C3	C4	104.56(16)	C15	C11	C10	112.80(18)
C6	C3	C2	114.56(18)	C15	C11	C12	114.80(19)
C6	C3	C4	112.76(18)	O6	C12	C11	102.35(16)
N1	C4	C3	105.63(16)	O6	C12	C13	111.07(17)
N1	C4	C5	111.60(17)	C13	C12	C11	105.00(17)
C5	C4	C3	112.54(18)	N2	C13	C12	104.01(17)
C4	C5	Cl1	110.22(17)	C10	C14	Cl2	110.46(16)
O3	C6	C3	108.90(17)	O4	C15	C11	109.48(17)
O1	C7	C8	110.73(18)	O5	C16	C17	110.5(2)
O1	C7	C9	108.78(18)	O5	C16	C18	108.94(19)
O2	C7	O1	104.08(16)	O6	C16	O5	104.23(16)
O2	C7	C8	111.38(19)	O6	C16	C17	111.3(2)
O2	C7	C9	109.68(18)	O6	C16	C18	109.66(19)
C9	C7	C8	111.88(19)	C18	C16	C17	111.89(19)

Table 4-6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound 32.

Atom	*x*	*y*	*z*	U(eq)
H3	7060(80)	4490(30)	4870(50)	69(12)
H1	-130(50)	4721(18)	6070(30)	31(7)
H1A	1772.64	5464.58	4797.21	27
H1B	-421.6	5488.08	4029.66	27
H2A	2719.05	5046.37	2647.07	26
H4A	2613.2	4051.61	5884.55	24
H5A	1636.91	2801.55	5303.96	36
H5B	-548.88	3100.83	4780.18	36
H6A	5399.6	3503.72	4176.71	31
H6B	5515.02	4028.46	2874.85	31
H8A	1568.91	4121.91	-268.72	51
H8B	2907.01	3432.35	233.8	51
H8C	3403	4241.33	794.43	51
H9A	-1887.54	3180.83	2040.32	44

H9B	-537.02	2833.83	854.65	44
H9C	-1888.81	3556.93	579.71	44
H4	12003.66	5471.73	7703.71	44
H2	4940(40)	5269(17)	6360(30)	25(7)
H10	7562.16	5996.16	6529.41	25
H12	7837.76	5045.45	9880.09	27
H13A	6898.56	4564.52	7753.16	27
H13B	4718.61	4543.57	8518.66	27
H14A	6433.57	7228	7148.99	34
H14B	4323.44	6883.22	7691.94	34
H15A	10444.62	6533.21	8134.77	34
H15B	10643.54	6050.7	9486.43	34
H17A	8550.72	5883.89	11654.76	55
H17B	6693.02	5992.71	12687.65	55
H17C	7998.64	6690.65	12198.75	55
H18A	4627.23	7299.64	11425.73	49
H18B	3292.64	6589.56	11813.13	49
H18C	3230.09	6903.65	10304.6	49

Compound 39

Structure deposited at the Cambridge Crystallographic Data Centre (**CCDC** 2162553)

tx1795:

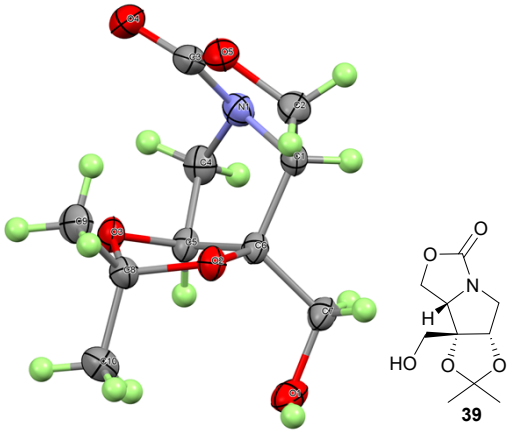


Fig 5. X-Ray ellipsoid plots of **39**

Table 5-1 Crystal data and structure refinement for compound 39.

Identification code	TX1795
Empirical formula	$C_{10}H_{15}NO_5$
Formula weight	229.23
Temperature/K	170.00(10)

Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	8.50310(10)
b/Å	11.03120(10)
c/Å	11.53190(10)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1081.685(19)
Z	4
ρ_{calc}g/cm^3	1.408
μ/mm^{-1}	0.962
F(000)	488.0
Crystal size/mm^3	0.33 × 0.12 × 0.11
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	11.1 to 150.286
Index ranges	-10 ≤ h ≤ 10, -13 ≤ k ≤ 13, -10 ≤ l ≤ 14
Reflections collected	12265
Independent reflections	2179 [R_{int} = 0.0237, R_{sigma} = 0.0129]
Data/restraints/parameters	2179/0/148
Goodness-of-fit on F^2	1.083
Final R indexes [I>=2σ (I)]	R_1 = 0.0280, wR_2 = 0.0754
Final R indexes [all data]	R_1 = 0.0292, wR_2 = 0.0766
Largest diff. peak/hole / e Å^{-3}	0.33/-0.15
Flack parameter	0.05(5)

Table 5-2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for compound 39. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O1	7356.8(16)	3077.2(12)	9162.4(12)	34.8(3)
O2	5413.6(15)	3299.8(11)	7131.2(10)	26.6(3)
O3	5389.6(16)	5353.0(11)	7086.4(10)	29.2(3)
O4	1445.6(17)	5844.7(14)	6680.9(15)	46.2(4)
O5	1592.0(16)	3817.7(13)	6650.3(13)	35.8(3)
N1	2437.8(19)	4761.9(13)	8241.2(13)	29.8(3)
C1	3056(2)	3531.2(16)	8360.2(15)	27.7(4)
C2	2169(2)	2865.3(18)	7401.6(18)	35.0(4)
C3	1816(2)	4910.4(19)	7159.8(17)	32.7(4)
C4	3616(2)	5614.0(17)	8677.6(16)	32.3(4)

C5	5159(2)	5050.9(15)	8278.9(14)	26.0(3)
C6	4874(2)	3664.1(14)	8250.3(14)	23.8(3)
C7	5702(2)	2958.6(16)	9208.0(15)	29.0(4)
C8	6043(2)	4325.7(17)	6518.1(15)	27.4(4)
C9	5443(3)	4299(2)	5282.7(15)	37.7(4)
C10	7820(2)	4334(2)	6589.0(19)	39.6(4)

Table 5-3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound 39. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	34.6(7)	34.5(7)	35.5(7)	2.2(6)	-11.9(6)	1.4(6)
O2	32.0(6)	25.9(6)	22.0(6)	1.0(4)	1.4(5)	-1.0(5)
O3	35.1(7)	26.3(6)	26.2(6)	7.0(4)	5.2(5)	1.9(5)
O4	34.8(7)	43.6(8)	60.1(9)	19.7(8)	-10.1(7)	0.2(6)
O5	31.6(6)	42.0(7)	33.9(7)	5.4(6)	-6.9(5)	-4.0(6)
N1	29.3(7)	32.5(7)	27.4(7)	1.1(6)	5.5(6)	1.1(6)
C1	29.7(9)	29.2(8)	24.1(8)	6.7(7)	1.5(7)	-3.7(7)
C2	32.3(9)	33.9(9)	38.7(10)	5.4(8)	-6.8(8)	-6.2(8)
C3	21.9(8)	40.7(10)	35.7(9)	8.4(8)	0.6(7)	0.0(8)
C4	39.7(10)	30.3(8)	27.1(8)	-2.4(7)	4.1(8)	-0.3(8)
C5	32.5(8)	24.1(7)	21.5(7)	3.2(6)	-1.9(7)	-2.2(7)
C6	28.6(8)	23.8(7)	19.1(7)	2.6(6)	-2.2(7)	-2.7(6)
C7	34.4(9)	27.4(8)	25.1(8)	5.0(7)	-6.2(7)	-2.8(7)
C8	27.1(8)	29.3(8)	25.9(8)	3.9(7)	2.8(6)	0.3(7)
C9	40.0(11)	48.8(11)	24.3(9)	5.1(8)	3.8(8)	2.2(9)
C10	28.8(9)	46.0(10)	44.1(11)	5.4(9)	4.4(8)	-0.8(9)

Table 5-4 Bond Lengths for compound 39.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C7	1.414(2)	N1	C3	1.365(2)
O2	C6	1.4274(19)	N1	C4	1.463(2)
O2	C8	1.438(2)	C1	C2	1.527(3)
O3	C5	1.4285(19)	C1	C6	1.558(2)
O3	C8	1.422(2)	C4	C5	1.523(2)
O4	C3	1.211(2)	C5	C6	1.549(2)
O5	C2	1.448(2)	C6	C7	1.524(2)
O5	C3	1.354(3)	C8	C9	1.514(2)

N1	C1	1.462(2)	C8	C10	1.513(3)

Table 5-5 Bond Angles for compound 39.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C6	O2	C8	110.04(12)	O3	C5	C6	103.37(13)
C8	O3	C5	108.14(12)	C4	C5	C6	105.91(14)
C3	O5	C2	109.79(14)	O2	C6	C1	111.48(13)
C1	N1	C4	108.53(14)	O2	C6	C5	104.30(13)
C3	N1	C1	109.67(15)	O2	C6	C7	111.28(14)
C3	N1	C4	120.17(15)	C5	C6	C1	104.28(14)
N1	C1	C2	101.60(14)	C7	C6	C1	110.58(14)
N1	C1	C6	105.18(13)	C7	C6	C5	114.61(14)
C2	C1	C6	118.46(15)	O1	C7	C6	112.69(15)
O5	C2	C1	104.58(15)	O2	C8	C9	108.78(15)
O4	C3	O5	121.55(17)	O2	C8	C10	110.48(15)
O4	C3	N1	128.3(2)	O3	C8	O2	104.78(12)
O5	C3	N1	110.12(16)	O3	C8	C9	108.51(15)
N1	C4	C5	102.96(14)	O3	C8	C10	111.14(16)
O3	C5	C4	108.28(14)	C10	C8	C9	112.81(16)

Table 5-6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound 39.

Atom	*x*	*y*	*z*	U(eq)
H1	7727.97	2518.37	8781.69	52
H1A	2778.85	3196.34	9120.24	33
H2A	2865.68	2323.81	6982.48	42
H2B	1305.36	2397.85	7720.34	42
H4A	3573.3	5674.11	9516.22	39
H4B	3470.92	6413.81	8344.94	39
H5	6052.87	5280.23	8768.89	31
H7A	5330.93	3245.43	9954.55	35
H7B	5427	2107.98	9142.68	35
H9A	5778.84	3563.04	4913.71	57
H9B	5854.49	4982.08	4865.62	57
H9C	4314.95	4334.77	5285.12	57
H10A	8137.47	4393.24	7385.74	59

H10B	8222.37	5015.21	6165.03	59
H10C	8226.03	3597.65	6261.21	59