Supporting Information for:

A Synthesis of a Rationally Designed Inhibitor of Cytochrome P450 8B1, a Therapeutic Target to Treat Obesity

Eunhee Chung,[2] Samuel D. Offei,[1] U-Ter Aondo Jia,[2] Juan Estevez,[2] Yessenia Perez,[2] Hadi D. Arman,[1] and Francis K. Yoshimoto[1]

[1]Department of Chemistry and [2]Department of Kinesiology, The University of Texas at San Antonio (UTSA), One UTSA Circle, San Antonio, TX 78249-0698

Steroids, 2021, xxxxxx

Table of Contents

1. Part 1. Figure S1: Summary of mice used for this study

Figure S1 below summarizes the results for treating mice with a high-fat and high-sucrose diet (HFHS) for 8 weeks. (CON: control, HFHS/UT: high-fat and high-sucrose diet, HFHS/OB: high-fat and high-sucrose diet with vehicle treatment that developed insulin resistance and obesity, HFHS/IR: high-fat and high-sucrose diet with vehicle treatment that developed insulin resistance but no obesity.

Panel (A) shows the body weight vs. the different groups, panel (B) shows the results for the glucose tolerance test through measurement of the area under the curve (AUC), panel (C) shows the blood glucose levels.

1. Part 1. Figure S1: Summary of mice used for this study

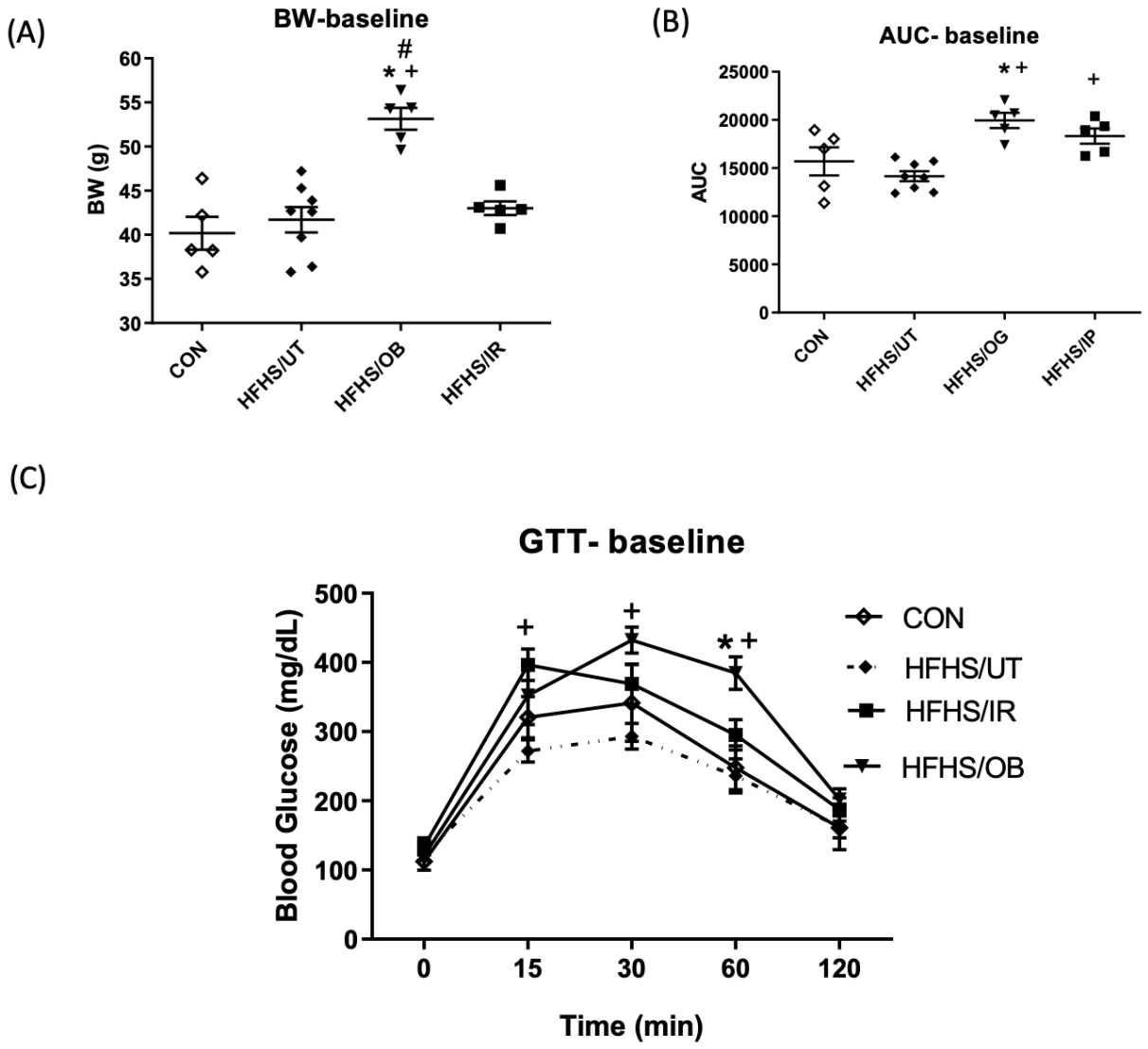
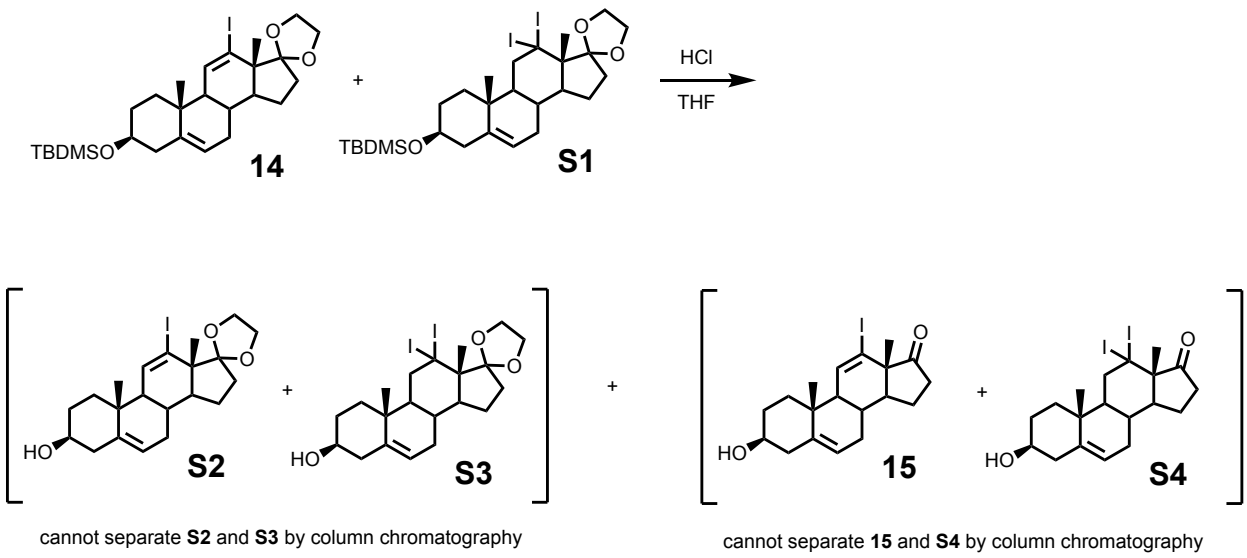


Figure S1. Effects of diet on BW and glucose tolerance tests. (A) Five mice out of 18 mice developed obesity after 8 weeks of HFHS feeding. (B) The area under the curve (AUC) of GTT and (C) glucose tolerance test (GTT). Blood glucose was measured right before glucose injection and 15, 30, 60, and 120 minutes after glucose injection. The HFHS/OB significantly increased AUC and impaired glucose clearance compared to the control (CON) group as well as HFHS/UT. The HFHS/IR mice developed insulin resistance compared to HFHS. * $P < 0.05$, vs.

CON, + P < 0.05, vs. HFHS/UT, # p < 0.05 vs. HFHS/IR. CON, control diet indicated by a closed reversed triangle (▼); HFHS/UT, mice fed with a high-fat and a high-sucrose diet (HFHS) with no alterations of body weight or glucose tolerance test, indicated by closed diamond (♦); HFHS/OB, mice fed HFHS developed obesity and insulin resistance, indicated by a closed triangle (▲); HFHS/IR, mice fed HFHS developed insulin resistance without inducing obesity, indicated by a closed circle (●). The sample size for CON, HFHS/UT, HFHS/OB, and HFHS/IR are 5, 8, 5, and 5, respectively. Group differences were compared using one-way analysis of variance (ANOVA) or two-way ANOVA followed by Tukey's multiple comparisons tests. A p-value of < 0.05 was considered a significant difference. CON: control, HFHS: high fat high sucrose diet, UT: untreated, OB: mice that developed insulin resistance with obesity, IR: mice that developed insulin resistance without obesity.

2. Part 2: C12-Diiodide Conversion to the Vinyl Iodide Using AgOTf



Scheme S1. Deprotection of the C3-OTBDMS of the vinyl iodide/diiodide (14/S1) mixture.

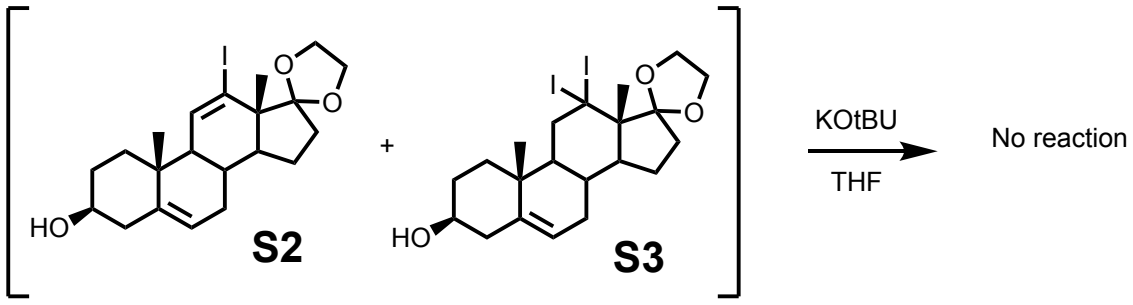
In a 500 ml round bottom flask containing the C3-OTBDMS C17-ketal for vinyl iodide-diiodide mixture (19.7 g, 0.0345 mol, 1 eq) in methanol: dichloromethane solution (300 ml, 1:2, v/v) was added para-toluenesulfonic acid (0.3 g, 1.7 mmol, 0.049 eq). The solution was stirred for 3 h. The reaction solution was diluted with dichloromethane (200 ml) and then quenched with the addition of saturated sodium bicarbonate (200 ml). The organic layer was separated from the aqueous layer and concentrated under reduced pressure to form a crude orange oil. The orange oil was purified by column chromatography (25% ethyl acetate in hexanes → 40% ethyl acetate in hexanes) to afford a mixture of the deprotected TBDMS group for both the ketal S2 co-eluting with the diiodide S3 and the ketone 15 also co-eluting with the diiodide S4 (total mass was about 12.5 g).

The conversion of the hydrazone (Scheme 1, **13**) to the vinyl iodide (**14**) gave a mixture of the vinyl iodide and diiodide (Scheme S1, **14** and **S1**). The vinyl iodide was not separable from the diiodide through silica gel column chromatography even after deprotection of the C3-TBDMS group. The following troubleshooting process resulted in the use of AgOTf to successfully convert the diiodide to vinyl iodide.

2. Part 2: C12-Diiodide Conversion to the Vinyl Iodide Using AgOTf

Elimination of the diiodide to the vinyl iodide

Potassium *tert*-butoxide treatment of the vinyl iodide-diiodide compound

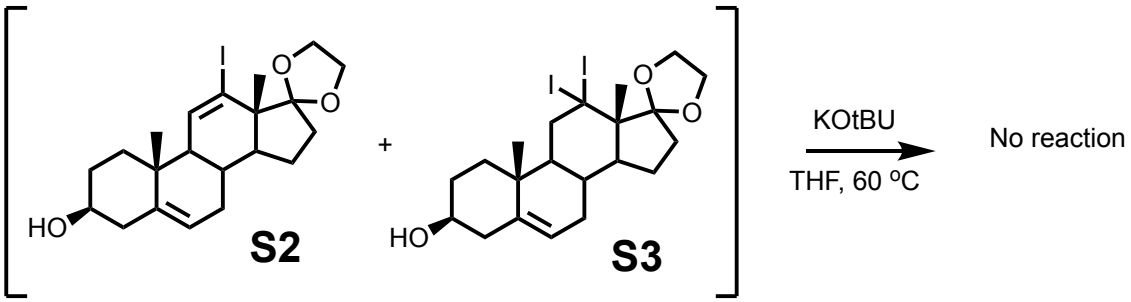


Scheme S2. Failed attempt to eliminate the diiodide (S3) to the vinyl iodide (S2).

In a screw cap vial containing the vinyl iodide-diiodide mixture (100 mg, 0.17 mmol, 1 eq) in THF (5 ml) was added potassium *tert*-butoxide (32 mg, 0.285 mmol, 1.6 eq). The mixture was stirred and aliquot NMRs were checked at 10 minutes and 45 minutes. The diiodide which is characterized by the dd at around 4.2 ppm did not disappear after 3 hr. The conversion of the diiodide to the vinyl iodide did not work with this reaction condition.

2. Part 2: C12-Diiodide Conversion to the Vinyl Iodide Using AgOTf

Reflux conditions



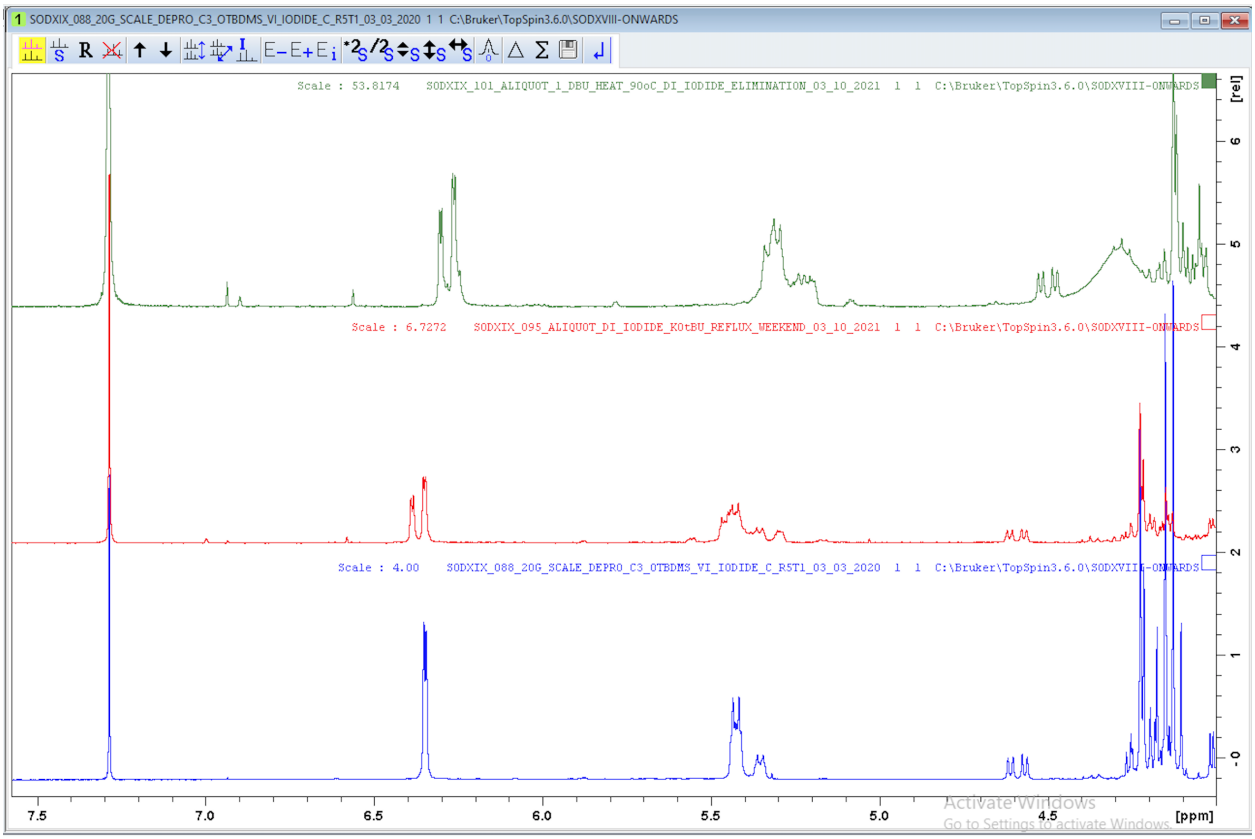
cannot separate **S2** and **S3** by column chromatography

Scheme S3. Failed attempt to eliminate the diiodide (S3) to the vinyl iodide (S2) with potassium tert-butoxide in the presence of heat.

To a 250 ml RBF containing the vinyl iodide-diiodide mixture (2.0 g, 3.43 mmol, 1 eq) and potassium *tert*-butoxide (0.32 g, 2.85 mmol, 0.83 eq) was added THF (100 ml). The reaction solution was refluxed for 12 h. The NMR of the reaction monitored subsequently and there was no conversion in the diiodide product to the vinyl-iodide.

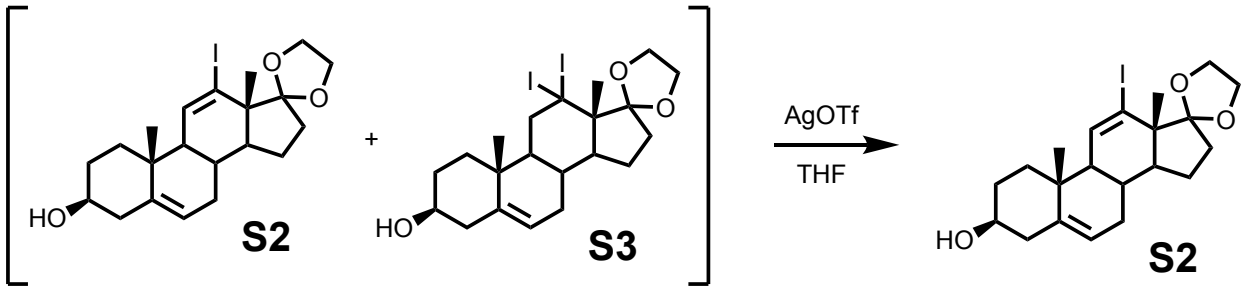
2. Part 2: C12-Diiodide Conversion to the Vinyl Iodide Using AgOTf

Bottom row (blue) mixture of vinyl iodide and diiodide (at C12) used for reaction with potassium tert-butoxide and with DBU to possibly eliminate one of the iodines in the C12-diiodide to form the vinyl iodide (NMR spectra to show that the reactions failed even after heating). Middle row (red) aliquot of the reaction mixture with potassium tert-butoxide and heating. Top row (green) aliquot of the reaction mixture with DBU and heating. The doublet of doublet at δ 4.59 is gone after purification.



2. Part 2: C12-Diiodide Conversion to the Vinyl Iodide Using AgOTf

Silver triflate treatment of the vinyl iodide-diiodide material



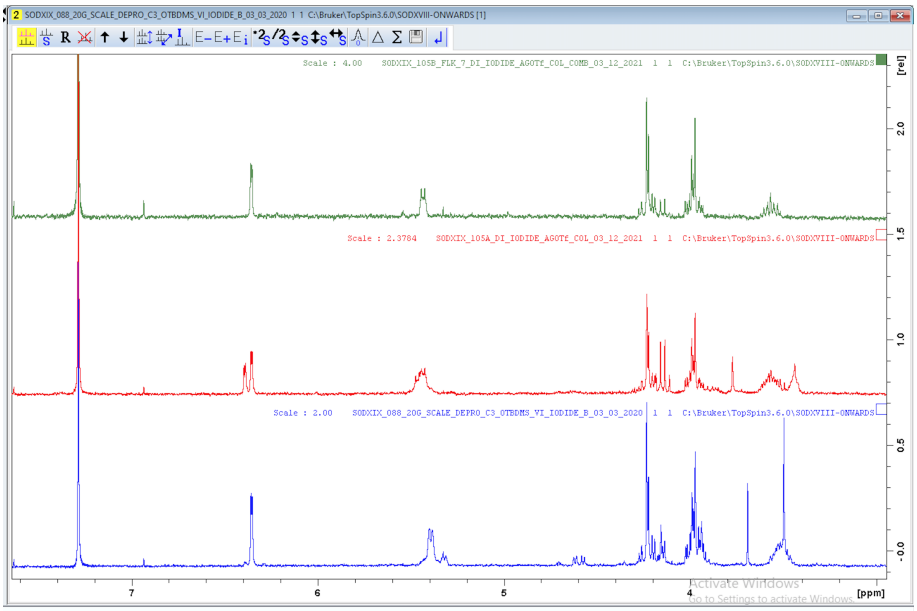
cannot separate **S2** and **S3** by column chromatography

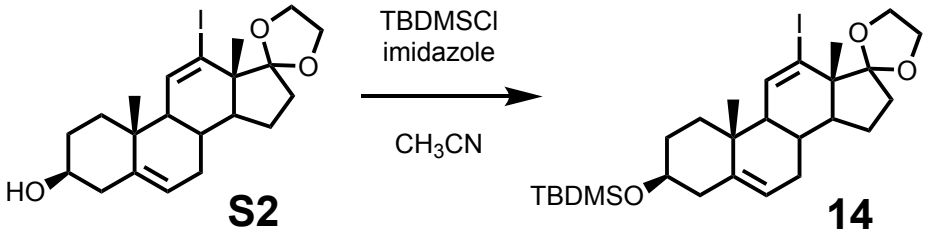
Scheme S4. Successful elimination of the diiodide (S3) to the vinyl iodide (S2).

To a solution of vinyl-iodide-diiodide (10.5 g, 0.018, 1 eq) in THF was added AgOTf (1 g, 3.89 mmol, 0.21 eq). The solution was stirred for 45 minutes. The conversion of the diiodide to the vinyl iodide was observed which was characterized by the disappearance of the "dd" at around 4.2 ppm. The reaction solution was filtered on a short column pad (100 % ethyl acetate) and concentrated under reduced pressure to afford the vinyl iodide product (10.4 g).

2. Part 2: C12-Diiodide Conversion to the Vinyl Iodide Using AgOTf

Bottom row (blue) mixture of vinyl iodide and diiodide (at C12) used for reaction with silver triflate. Middle row (red) crude reaction mixture. Top row (green) purified vinyl iodide product. The doublet of doublet at δ 4.59 is gone after purification.



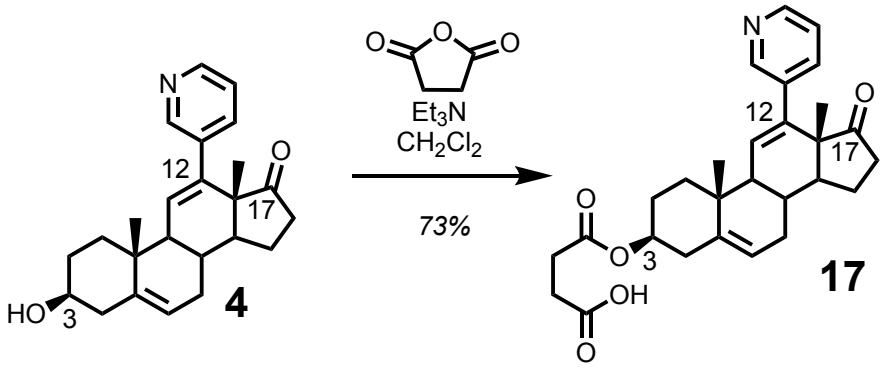


S2 14

Scheme S5. Conversion of the pure vinyl iodide (S2) to compound 15.

3. Part 3: Solubilizing Compound 17 in NaOH in saline buffer (1%, w/v)

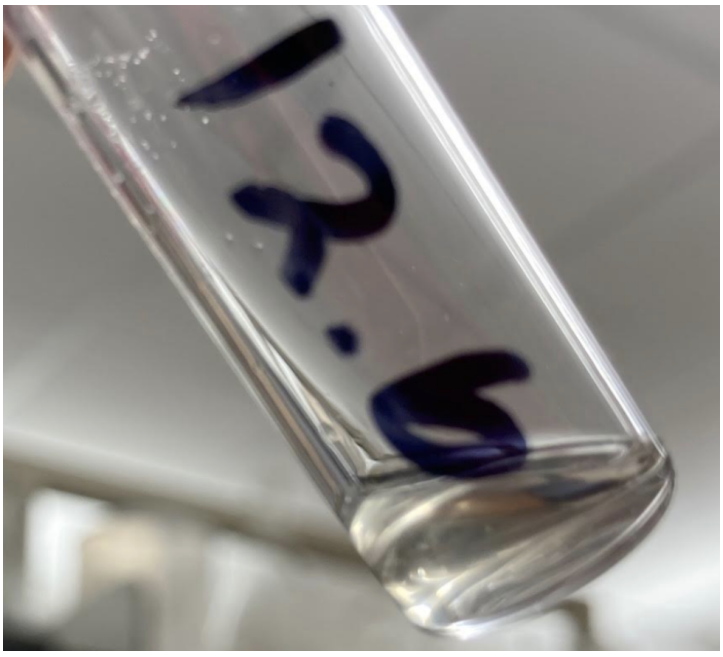
For future studies in enhancing the solubility of the C12-pyridine containing inhibitor in aqueous media, the C3-hydroxy group was protected as the succinate.



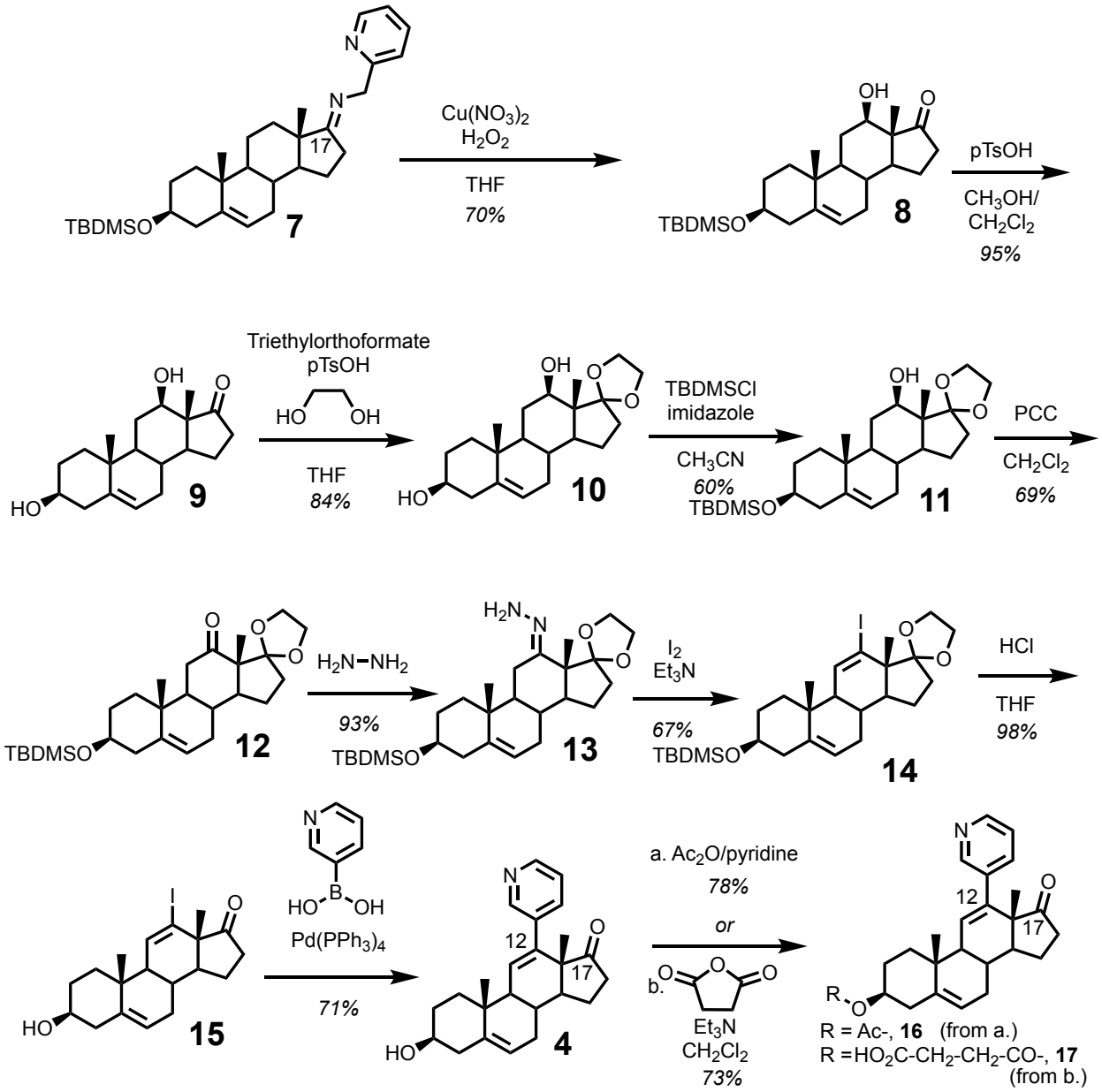
A saline stock solution was made separately (solution A: 0.25 g of NaCl and 0.25 g of $NaHCO_3$ in 24 ml of deionized water) from NaOH 10%, w/v, solution (solution B: 1 g NaOH in 10 ml of deionized water). The 1% NaOH (w/v) solution in saline buffer was made by mixing solution A and solution B (9 to 1, v/v) to make solution C.

Solution C (200 μL) was added to the succinate (12.6 mg) and left to sit on the benchtop after the solid dissolved in the solution over a period of 5 minutes.

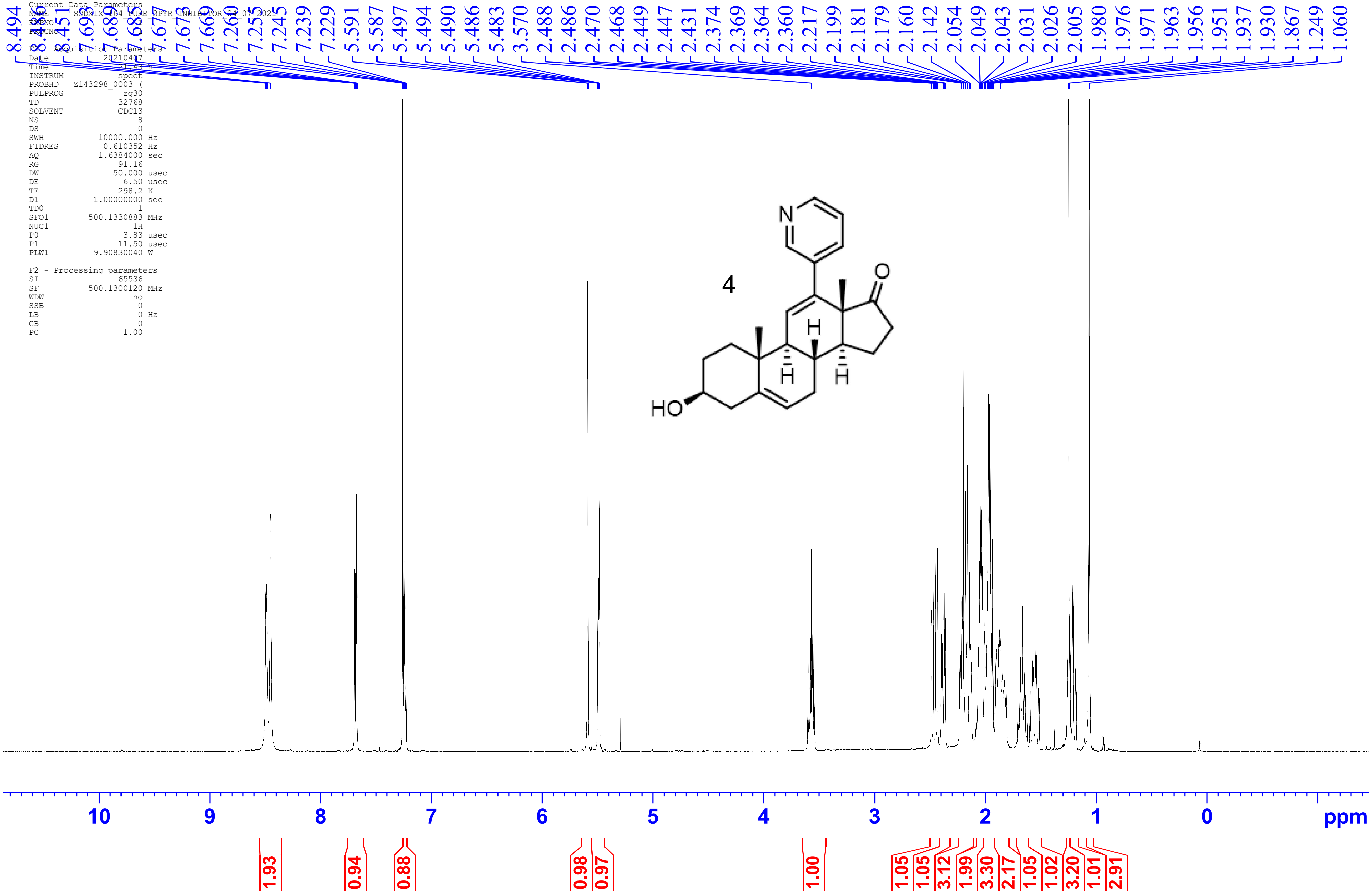
The dissolved succinate remained on the benchtop for 1 hour in solution (63 mg/ml concentration):

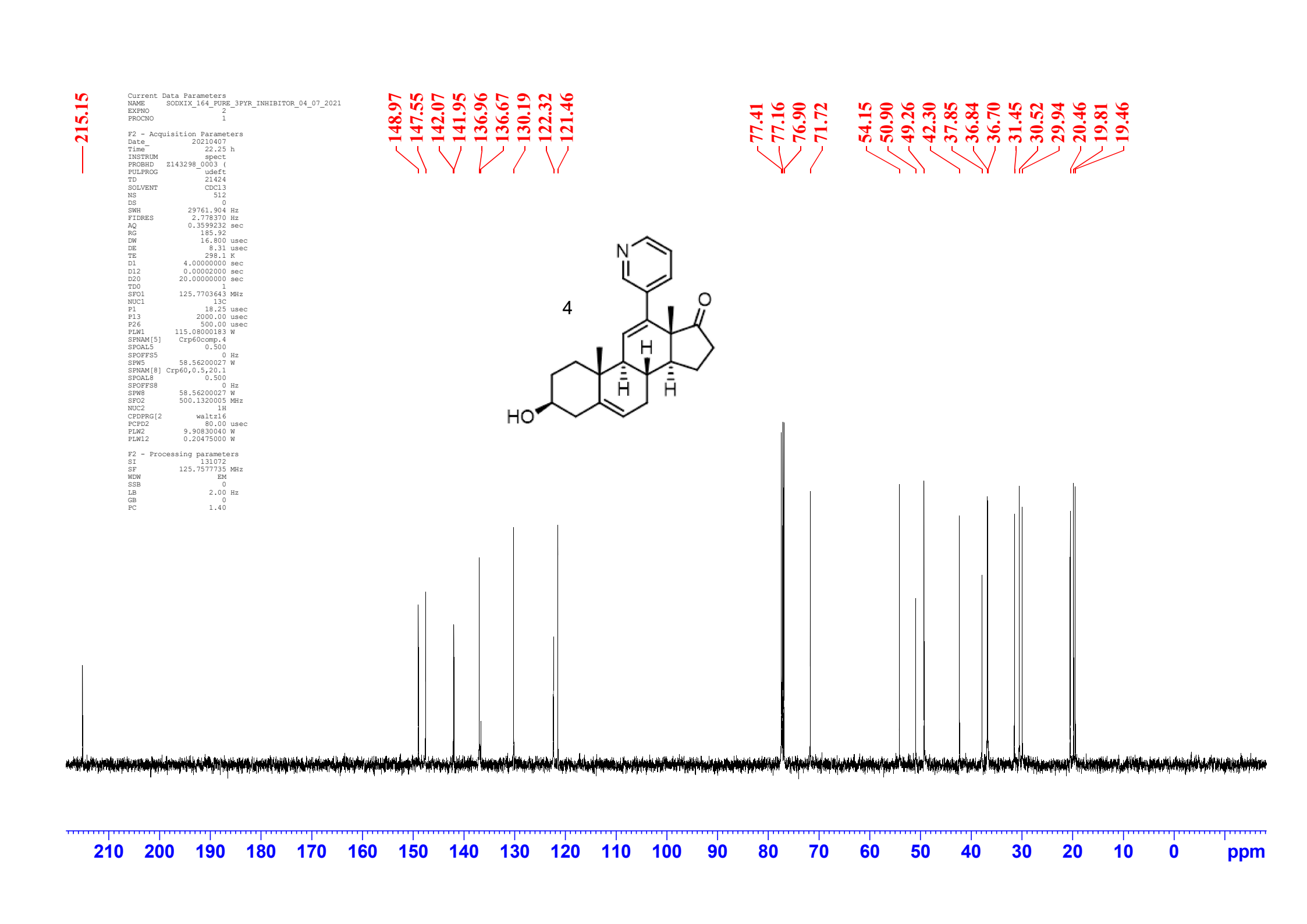


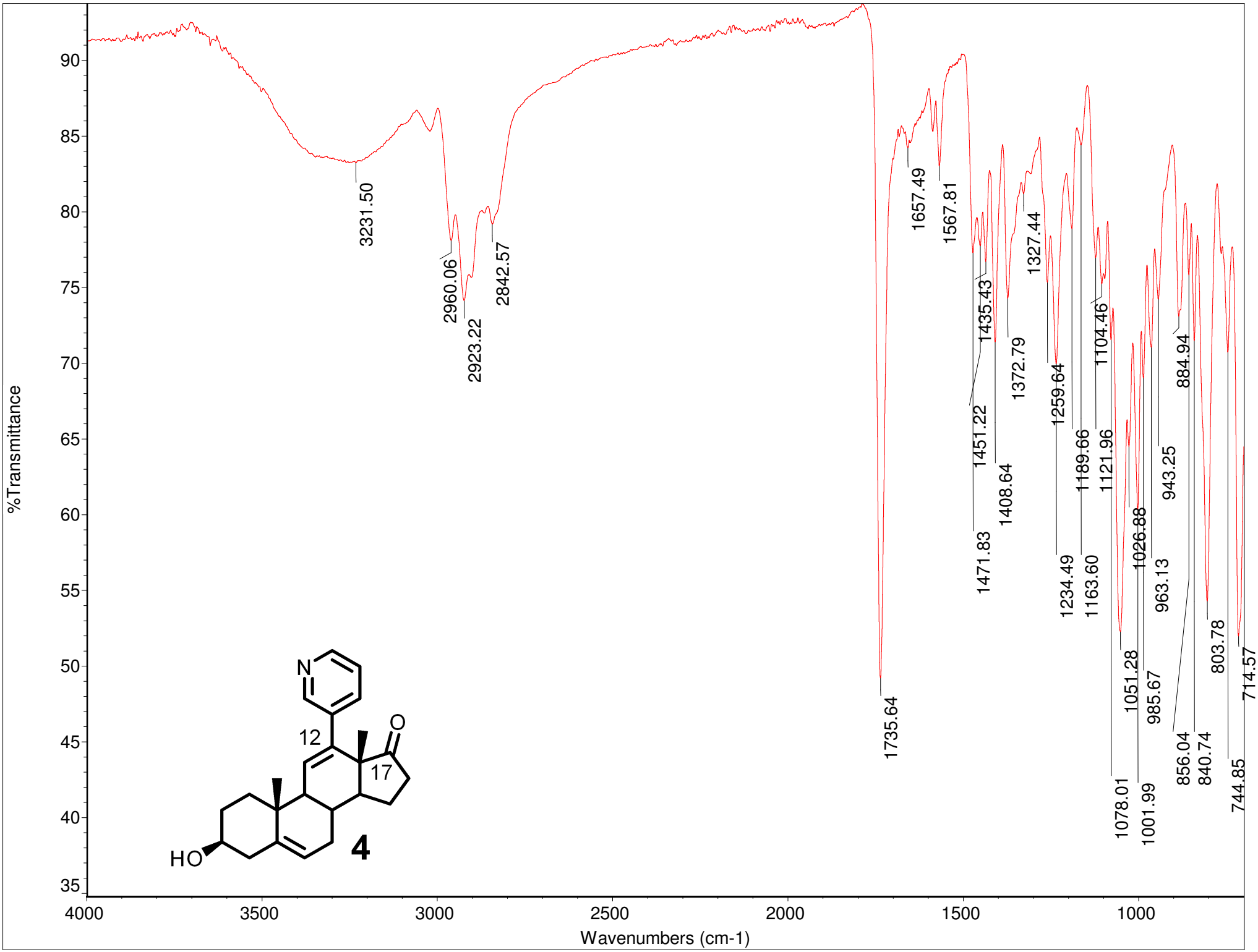
4. Part 4: ^{1}H NMR, ^{13}C NMR, IR, HRMS, and optical rotation data of synthesized compounds

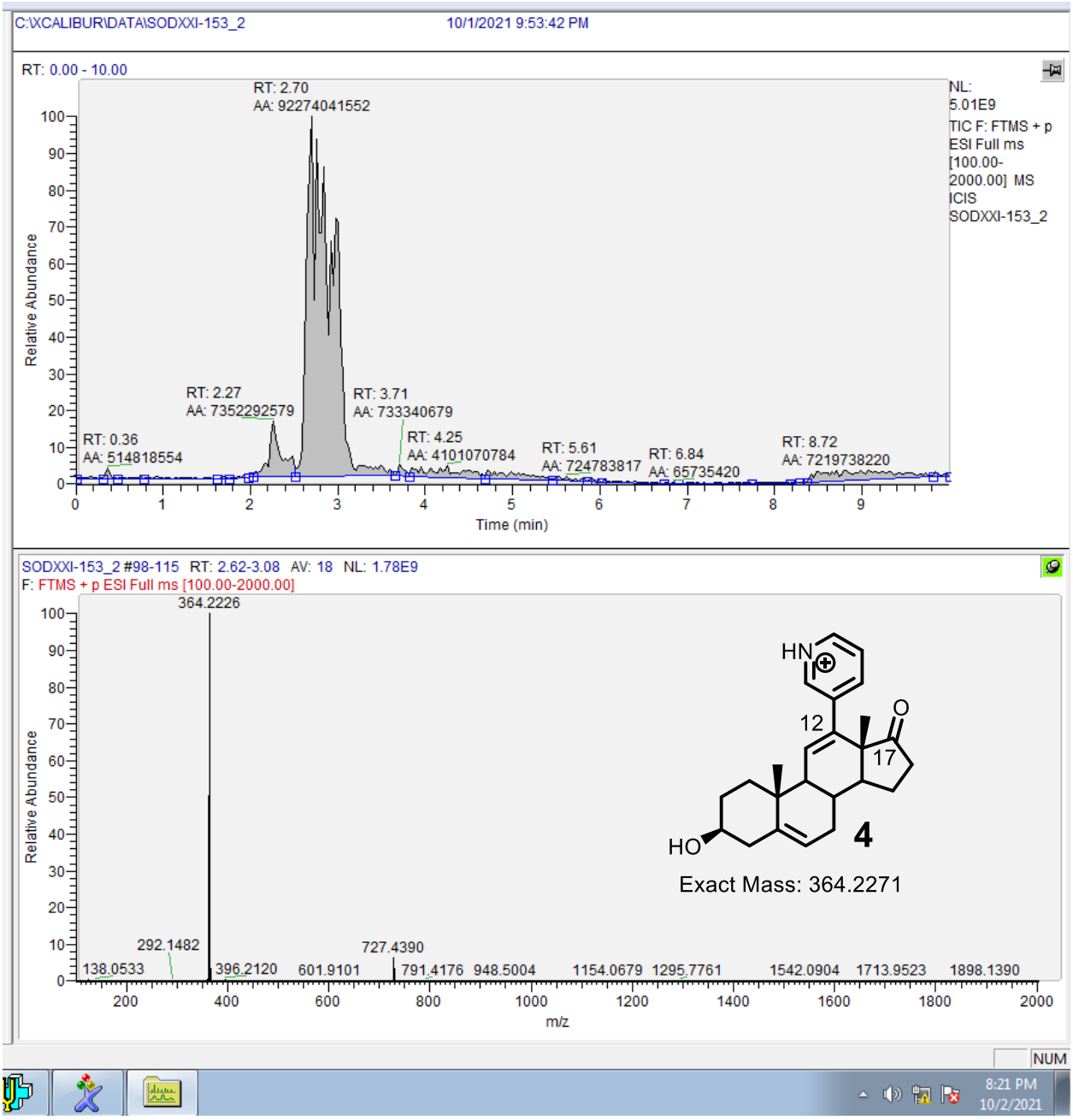


Scheme S6. Synthesized compounds presented in the main text.


Current Data Parameters
F2 - Acquisition Parameters
Date 20210407
INSTRUM spect
PROBHD Z143298_0003 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 91.16
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 3.83 usec
P1 11.50 usec
PLW1 9.90830040 W
F2 - Processing parameters
SI 65536
SF 500.1300120 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
8.494
8.451
7.693
7.677
7.673
7.669
7.260
7.255
7.245
7.239
7.229
5.591
5.587
5.497
5.494
5.490
5.486
5.483
3.570
2.488
2.486
2.470
2.468
2.449
2.447
2.431
2.374
2.369
2.364
2.360
2.217
2.199
2.181
2.179
2.160
2.142
2.054
2.049
2.043
2.031
2.026
2.005
1.980
1.976
1.971
1.963
1.956
1.951
1.937
1.930
1.867
1.249
1.060
4
N
O
H
H
H
HO
10
9
8
7
6
5
4
3
2
1
0
ppm
1.93
0.94
0.88
0.98
0.97
1.00
1.05
1.05
3.12
1.99
3.30
2.17
1.05
1.02
3.20
1.01
2.91


215.15
148.97
147.55
142.07
141.95
136.96
136.67
130.19
122.32
121.46
77.41
77.16
76.90
71.72
54.15
50.90
49.26
42.30
37.85
36.84
36.70
31.45
30.52
29.94
20.46
19.81
19.46
Current Data Parameters
NAME SODXIX_164_PURE_3PYR_INHIBITOR_04_07_2021
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210407
Time 22.25 h
INSTRUM spect
PROBHD Z143298_0003 (
PULPROG udeft
TD 21424
SOLVENT CDCl3
NS 512
DS 0
SWH 29761.904 Hz
FIDRES 2.778370 Hz
AQ 0.3599232 sec
RG 185.92
DW 16.800 usec
DE 8.31 usec
TE 298.1 K
D1 4.00000000 sec
D12 0.00002000 sec
D20 20.00000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 18.25 usec
P13 2000.00 usec
P26 500.00 usec
PLW1 115.08000183 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 58.56200027 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 58.56200027 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 9.90830040 W
PLW12 0.20475000 W
F2 - Processing parameters
SI 131072
SF 125.7577735 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
4
N
O
HO
H
H
H
210
200
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)
4000
3500
3000
2500
2000
1500
1000
90
85
80
75
70
65
60
55
50
45
40
35
3231.50
2960.06
2923.22
2842.57
1735.64
1657.49
1567.81
1471.83
1451.22
1435.43
1408.64
1372.79
1327.44
1259.64
1234.49
1189.66
1163.60
1121.96
1104.46
1078.01
1051.28
1026.88
1001.99
985.67
963.13
943.25
884.94
856.04
840.74
803.78
744.85
714.57
N
O
12
17
HO
4


C:\XCALIBUR\DATA\SODXXI-153_2
10/1/2021 9:53:42 PM
RT: 0.00 - 10.00
RT: 2.70
AA: 92274041552
NL:
5.01E9
TIC F: FTMS + p
ESI Full ms
[100.00-
2000.00] MS
ICIS
SODXXI-153_2
RT: 2.27
AA: 7352292579
RT: 3.71
AA: 733340679
RT: 0.36
AA: 514818554
RT: 4.25
AA: 4101070784
RT: 5.61
AA: 724783817
RT: 6.84
AA: 65735420
RT: 8.72
AA: 7219738220
Relative Abundance
Time (min)
SODXXI-153_2 #98-115 RT: 2.62-3.08 AV: 18 NL: 1.78E9
F: FTMS + p ESI Full ms [100.00-2000.00]
364.2226
292.1482
727.4390
138.0533
396.2120
601.9101
791.4176
948.5004
1154.0679
1295.7761
1542.0904
1713.9523
1898.1390
Relative Abundance
m/z
HN
O
12
17
HO
4
Exact Mass: 364.2271
NUM
8:21 PM
10/2/2021

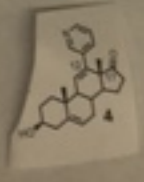
LAXCO Optical Rotation

Original O.R. : -0.082° O.R. °

-0.082

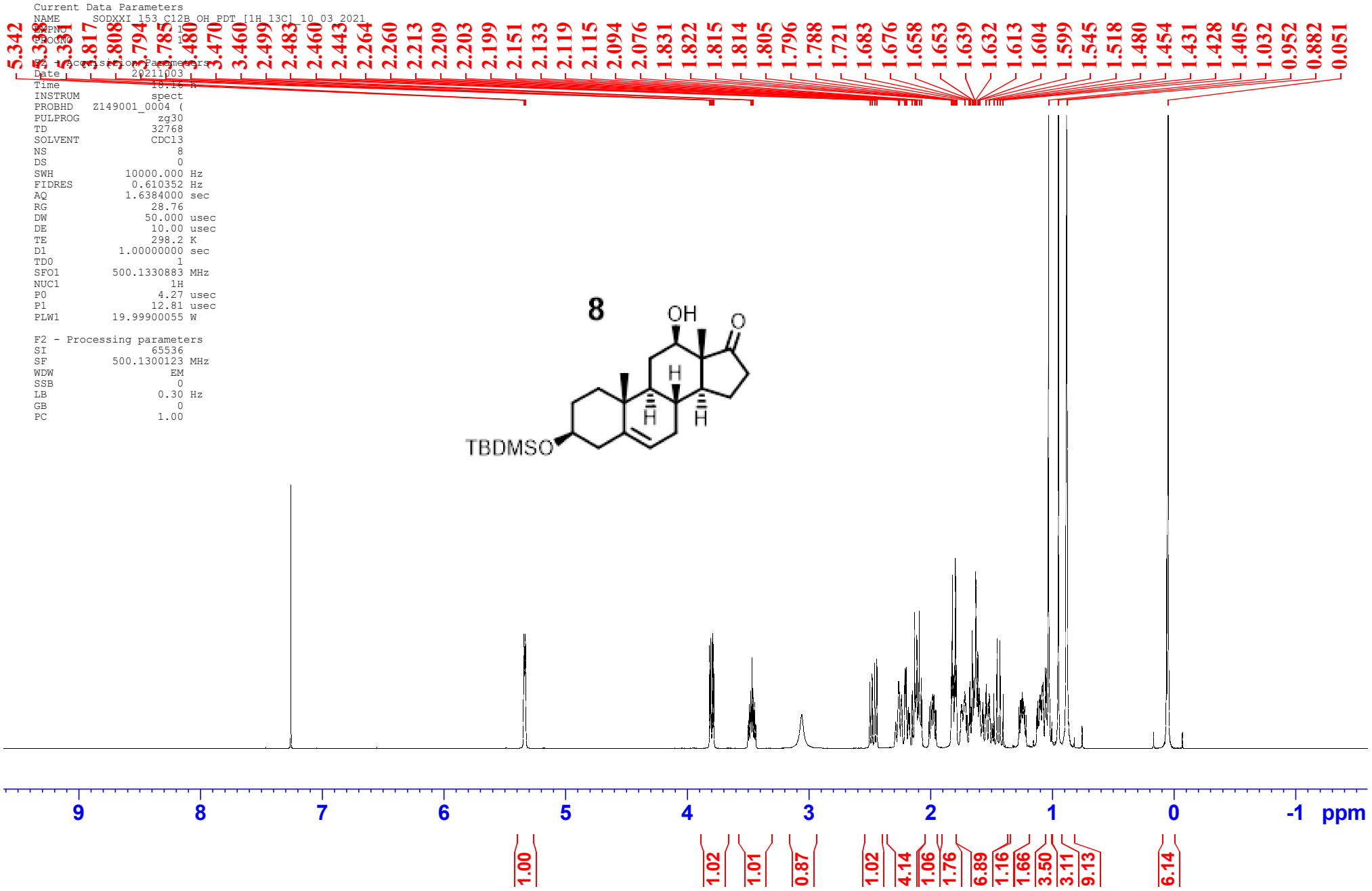
Temp. : 21.5℃ 21.5℃

WLG	Cell			ATC
589.44	1.0000dm			20.0
			GLP	
			Off	

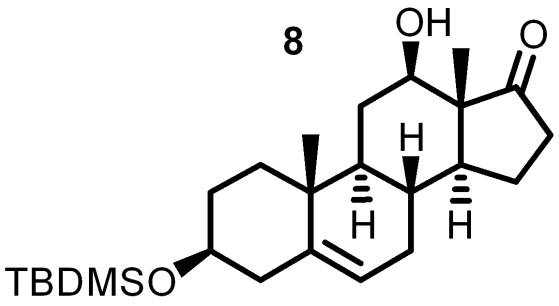


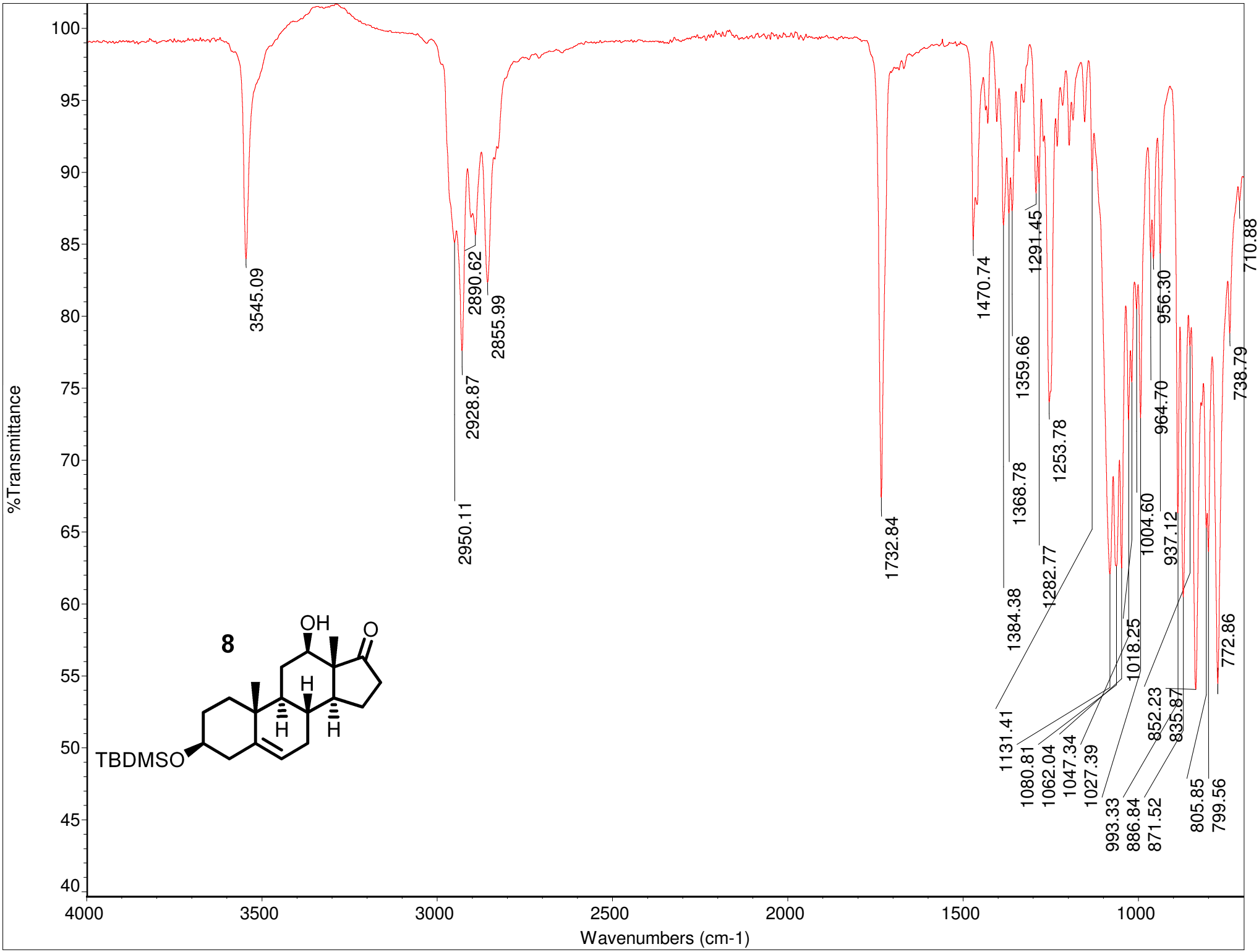
$\frac{15.2\text{mg}}{10\text{mL}}$

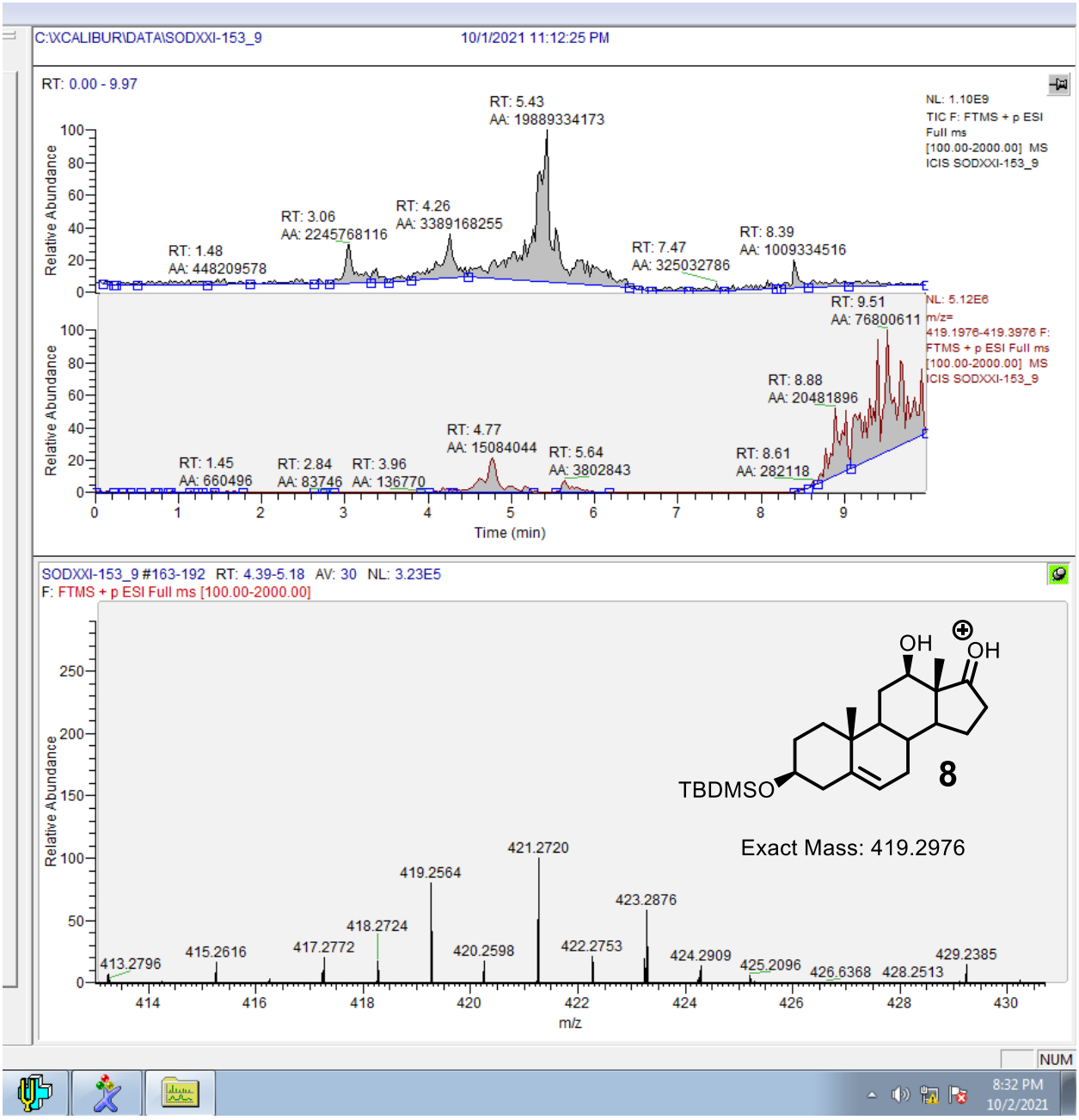
-0.082


Current Data Parameters
NAME SODXXI_153_C12B_OH_PDT_[1H_13C]_10_03_2021
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211003
Time 18.16 h
INSTRUM spect
PROBHD Z149001_0004 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 28.76
DW 50.000 usec
DE 10.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 4.27 usec
P1 12.81 usec
PLW1 19.99900055 W
F2 - Processing parameters
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
OH
O
H
H
H
TBDMSO
5.342
5.301
3.817
3.808
3.794
3.785
3.480
3.470
3.460
2.499
2.483
2.460
2.443
2.264
2.260
2.213
2.209
2.203
2.199
2.151
2.133
2.119
2.115
2.094
2.076
1.831
1.822
1.815
1.814
1.805
1.796
1.788
1.721
1.683
1.676
1.658
1.653
1.639
1.632
1.613
1.604
1.599
1.545
1.518
1.480
1.454
1.431
1.428
1.405
1.032
0.952
0.882
0.051
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
1.02
1.01
0.87
1.02
4.14
1.06
1.76
6.89
1.16
1.66
3.50
3.11
9.13
6.14

SOBXXI_153_C12B_OH_PDT_[1H_13C]_10_03_2021




%Transmittance
Wavenumbers (cm-1)
100
95
90
85
80
75
70
65
60
55
50
45
40
4000
3500
3000
2500
2000
1500
1000
3545.09
2950.11
2928.87
2890.62
2855.99
1732.84
1470.74
1384.38
1368.78
1359.66
1291.45
1282.77
1253.78
1131.41
1080.81
1062.04
1047.34
1027.39
1018.25
1004.60
993.33
964.70
956.30
937.12
886.84
871.52
852.23
835.87
805.85
799.56
772.86
738.79
710.88
8
OH
O
H
H
H
TBDMSO


C:\XCALIBUR\DATA\SODXXI-153_9
10/1/2021 11:12:25 PM
RT: 0.00 - 9.97
NL: 1.10E9
TIC F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_9
RT: 5.43
AA: 19889334173
RT: 1.48
AA: 448209578
RT: 3.06
AA: 2245768116
RT: 4.26
AA: 3389168255
RT: 7.47
AA: 325032786
RT: 8.39
AA: 1009334516
NL: 5.12E6
m/z= 419.1976-419.3976 F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_9
RT: 9.51
AA: 76800611
RT: 8.88
AA: 20481896
RT: 4.77
AA: 15084044
RT: 5.64
AA: 3802843
RT: 1.45
AA: 660496
RT: 2.84
AA: 83746
RT: 3.96
AA: 136770
RT: 8.61
AA: 282118
Relative Abundance
Time (min)
SODXXI-153_9 #163-192 RT: 4.39-5.18 AV: 30 NL: 3.23E5
F: FTMS + p ESI Full ms [100.00-2000.00]
OH
OH
TBDMSO
8
Exact Mass: 419.2976
421.2720
419.2564
423.2876
418.2724
415.2616
417.2772
420.2598
422.2753
424.2909
425.2096
426.6368
428.2513
429.2385
413.2796
m/z
NUM
8:32 PM
10/2/2021

LAXCO Optical Rotation

Original O.R.: 0.038°

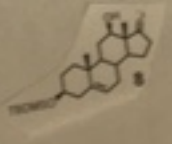
O.R. °

0.038

Temp.: 21.7℃ 21.6℃

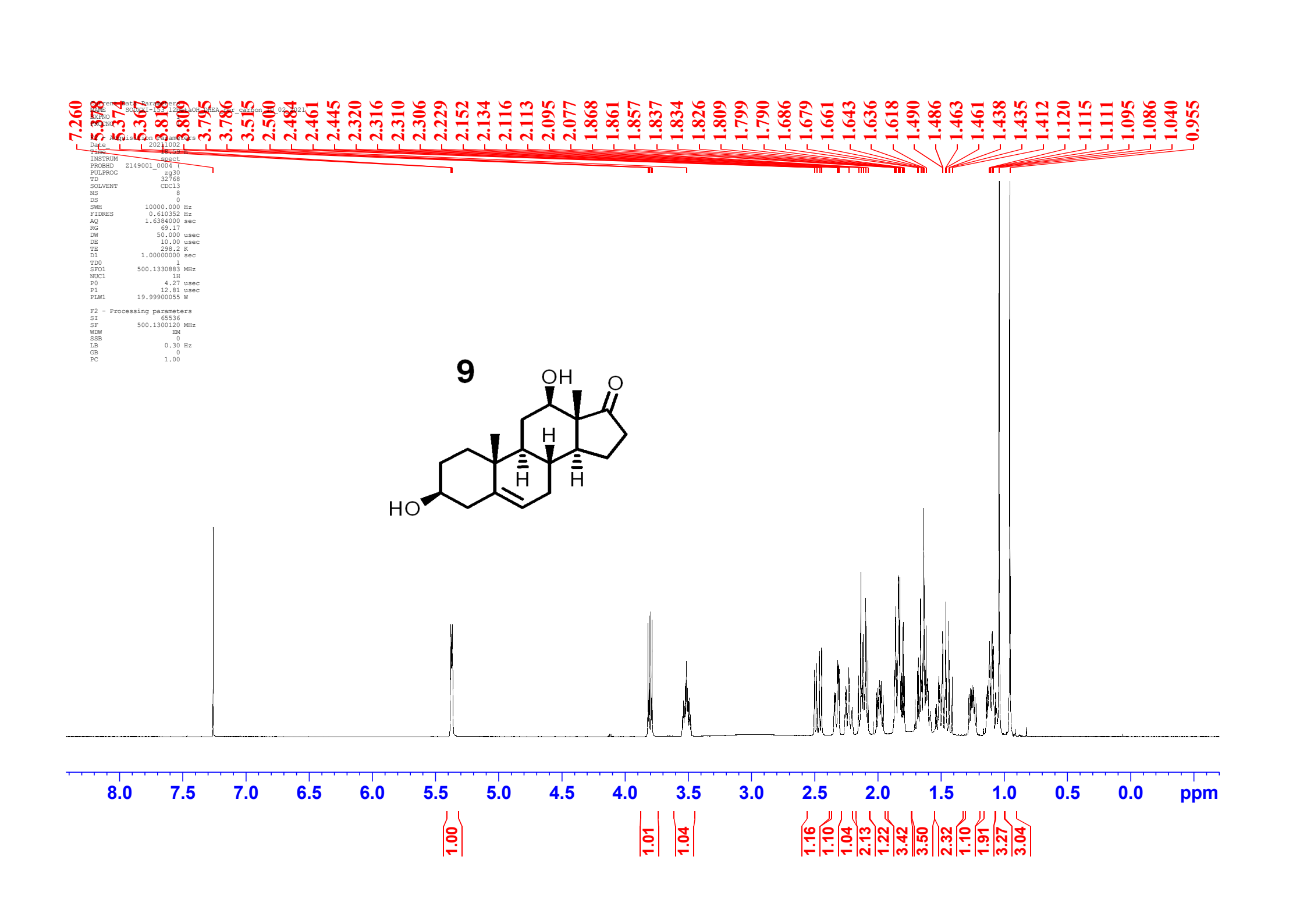
WLG	Cell			ATC
589.44	1.0000dm			20.0
Mode	Times		GLP	
single	1		Off	

database save

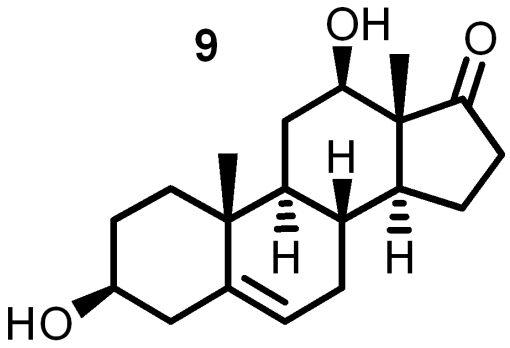


21.8 mg
10 mL

0.038

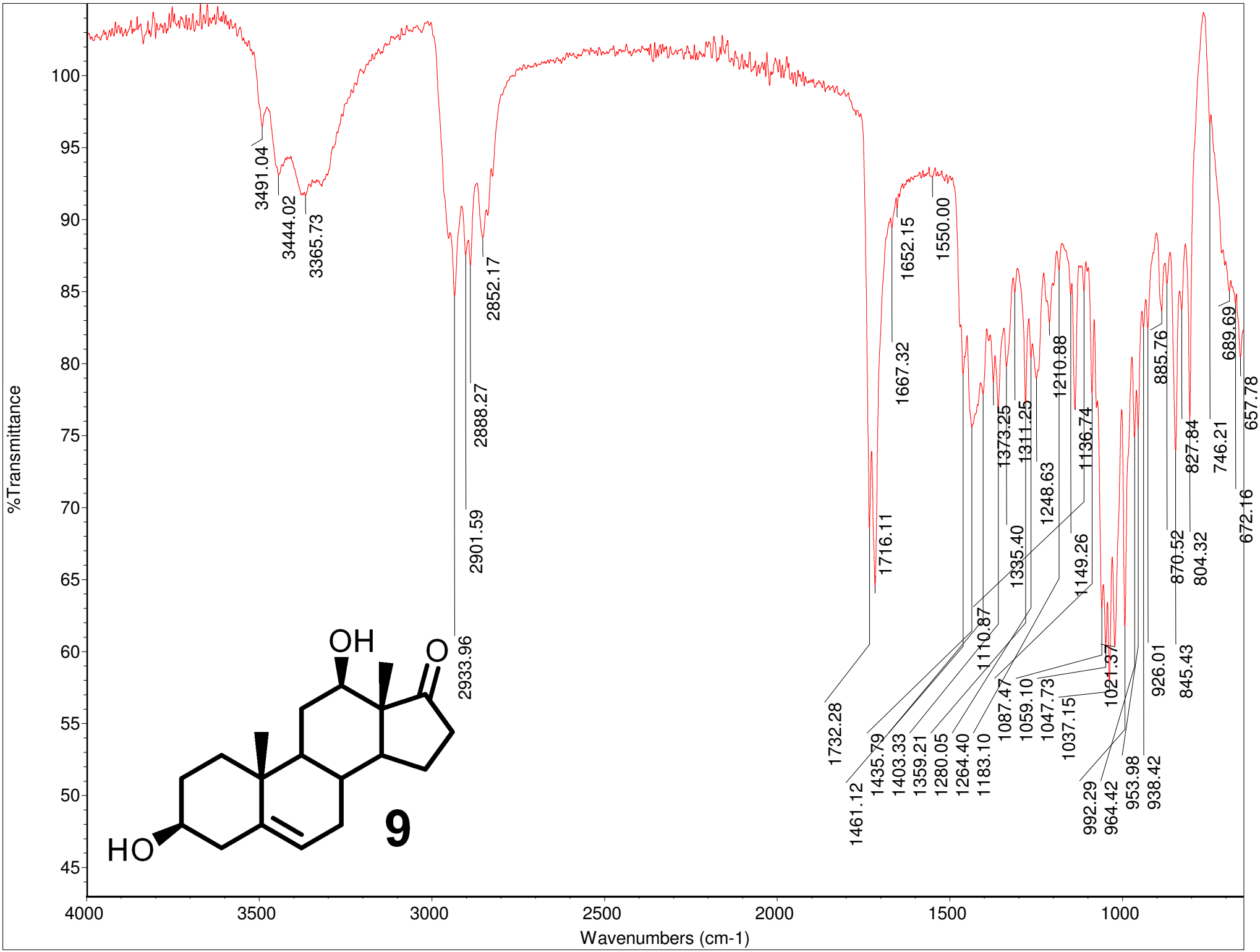

7.260
5.374
5.361
3.809
3.795
3.786
3.515
2.500
2.484
2.461
2.445
2.320
2.316
2.310
2.306
2.229
2.152
2.134
2.116
2.113
2.095
2.077
1.868
1.861
1.857
1.837
1.834
1.826
1.809
1.799
1.790
1.686
1.679
1.661
1.643
1.636
1.618
1.490
1.486
1.463
1.461
1.438
1.435
1.412
1.120
1.115
1.111
1.095
1.086
1.040
0.955
Current Data Parameters
NAME SODYXT-133_12DeTaOH_DHEA_for carbon_NL_02_2021
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211002
Time 18.59 h
INSTRUM spect
PROBHD Z149001_0004 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 69.17
DW 50.000 usec
DE 10.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 4.27 usec
P1 12.81 usec
PLW1 19.99900055 W
F2 - Processing parameters
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
OH
O
H
H
H
HO
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.01
1.04
1.16
1.10
1.04
2.13
1.22
3.42
3.50
2.32
1.10
1.91
3.27
3.04

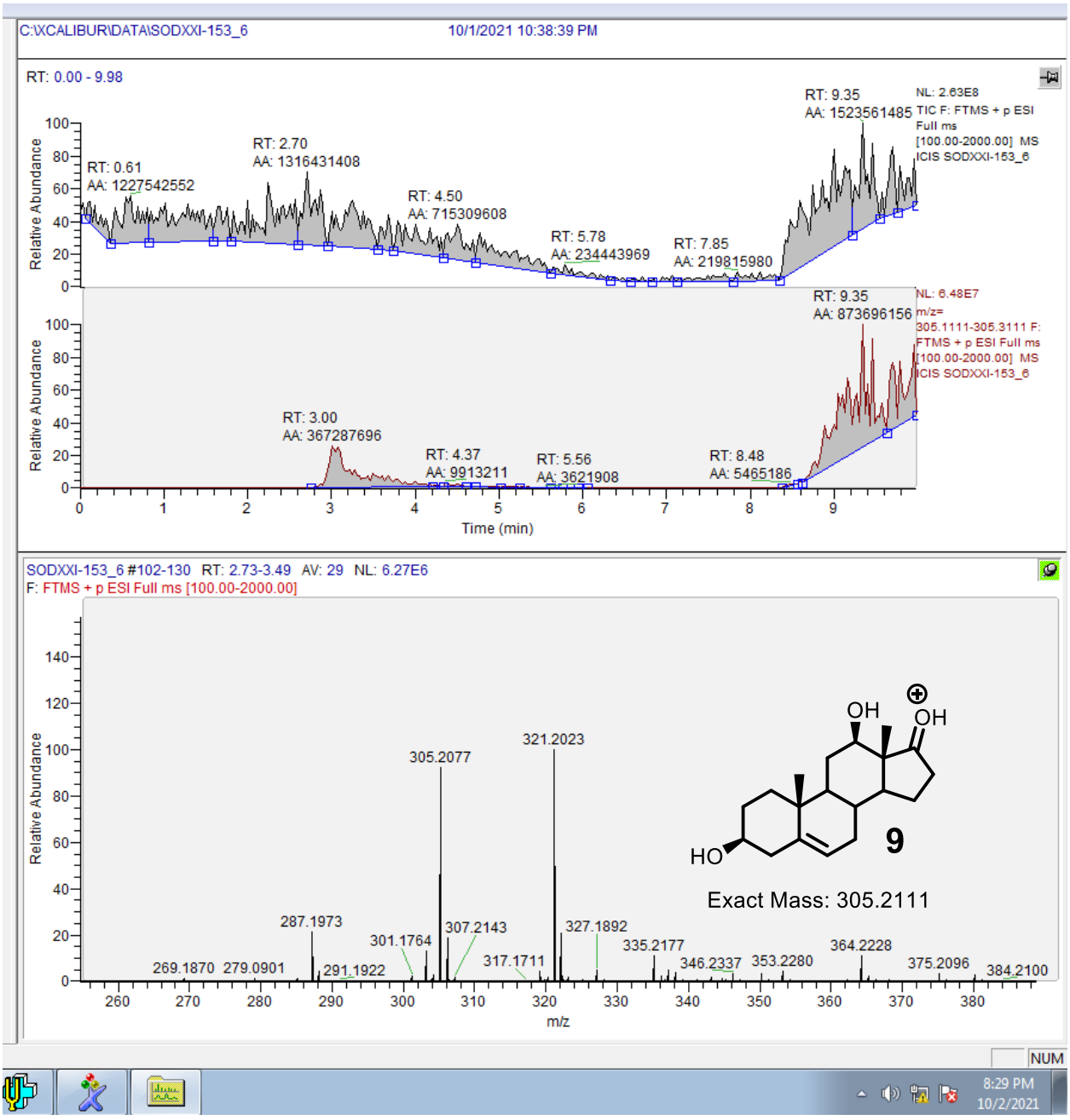
SODXXI-153_12betaOH_DHEA_for_carbon_10_02_2021



222.98
141.17
120.88
77.41
77.16
76.91
72.81
71.66
51.54
49.68
49.26
42.24
37.28
36.91
35.89
31.70
30.72
30.51
28.34
21.83
19.48
8.20

f1 (ppm)


%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
100
95
90
85
80
75
70
65
60
55
50
45
3491.04
3444.02
3365.73
2933.96
2901.59
2888.27
2852.17
1732.28
1716.11
1667.32
1652.15
1550.00
1461.12
1435.79
1403.33
1373.25
1359.21
1335.40
1311.25
1280.05
1264.40
1248.63
1210.88
1183.10
1149.26
1136.74
1110.87
1087.47
1059.10
1047.73
1037.15
1021.37
992.29
964.42
953.98
938.42
926.01
885.76
870.52
845.43
827.84
804.32
746.21
689.69
672.16
657.78
OH
O
HO
9


C:\XCALIBUR\DATA\SODXXI-153_6
10/1/2021 10:38:39 PM
RT: 0.00 - 9.98
RT: 0.61
AA: 1227542552
RT: 2.70
AA: 1316431408
RT: 4.50
AA: 715309608
RT: 5.78
AA: 234443969
RT: 7.85
AA: 219815980
RT: 9.35
AA: 1523561485
NL: 2.63E8
TIC F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_6
RT: 3.00
AA: 367287696
RT: 4.37
AA: 9913211
RT: 5.56
AA: 3621908
RT: 8.48
AA: 5465186
RT: 9.35
AA: 873696156
NL: 6.48E7
m/z= 305.1111-305.3111 F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_6
Relative Abundance
Time (min)
SODXXI-153_6 #102-130 RT: 2.73-3.49 AV: 29 NL: 6.27E6
F: FTMS + p ESI Full ms [100.00-2000.00]
305.2077
321.2023
287.1973
301.1764
307.2143
327.1892
335.2177
364.2228
269.1870
279.0901
291.1922
317.1711
346.2337
353.2280
375.2096
384.2100
m/z
OH
OH
HO
9
Exact Mass: 305.2111
NUM
8:29 PM
10/2/2021

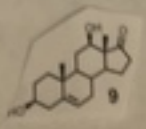
LAXCO Optical Rotation

Original O.R.: -0.025° O.R. °

-0.025

Temp.: 21.4℃ 21.4℃

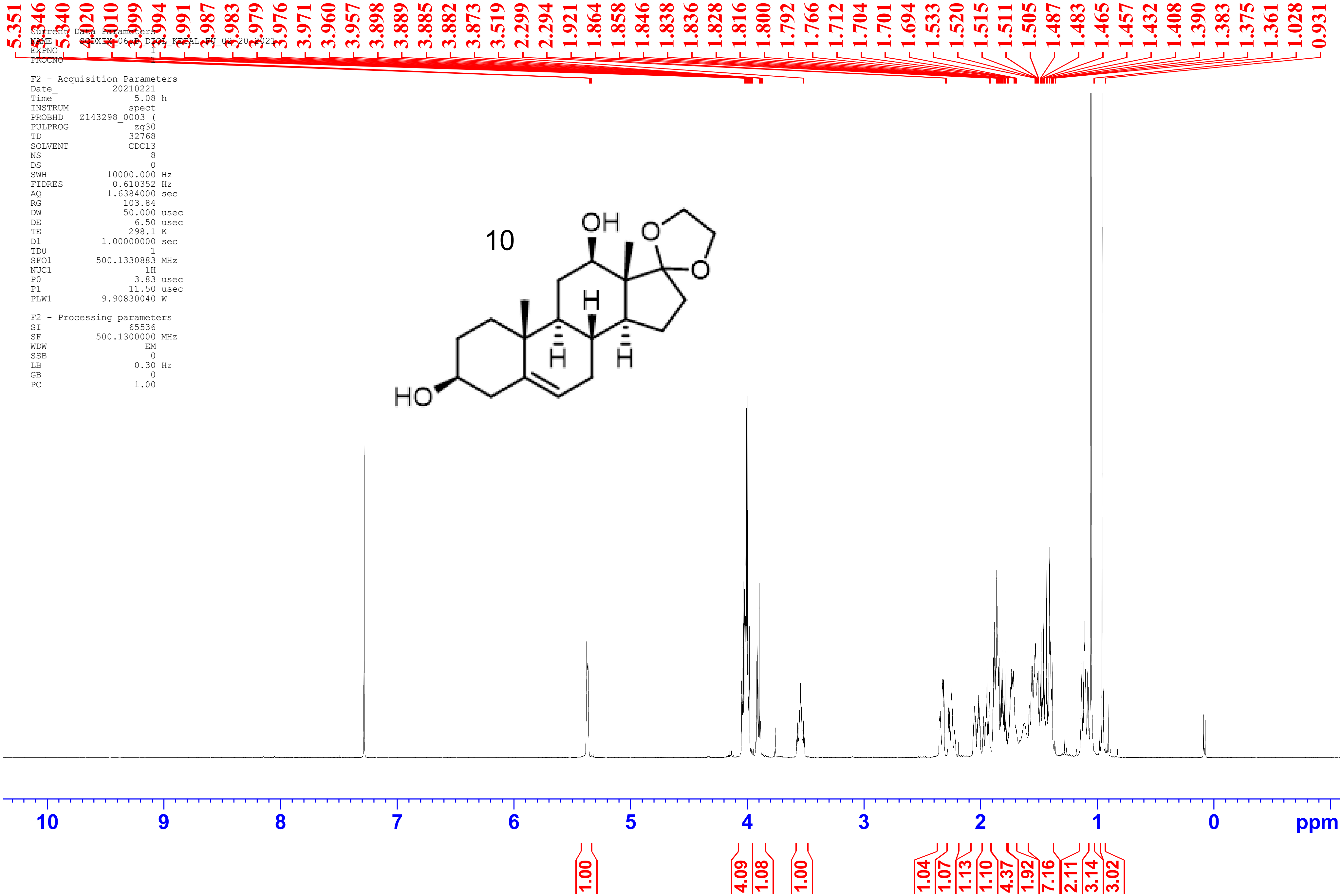
WLG	Cell			ATC
589.44	1.0000dm			20.0
Mode	Times		GLP	
single	1		off	

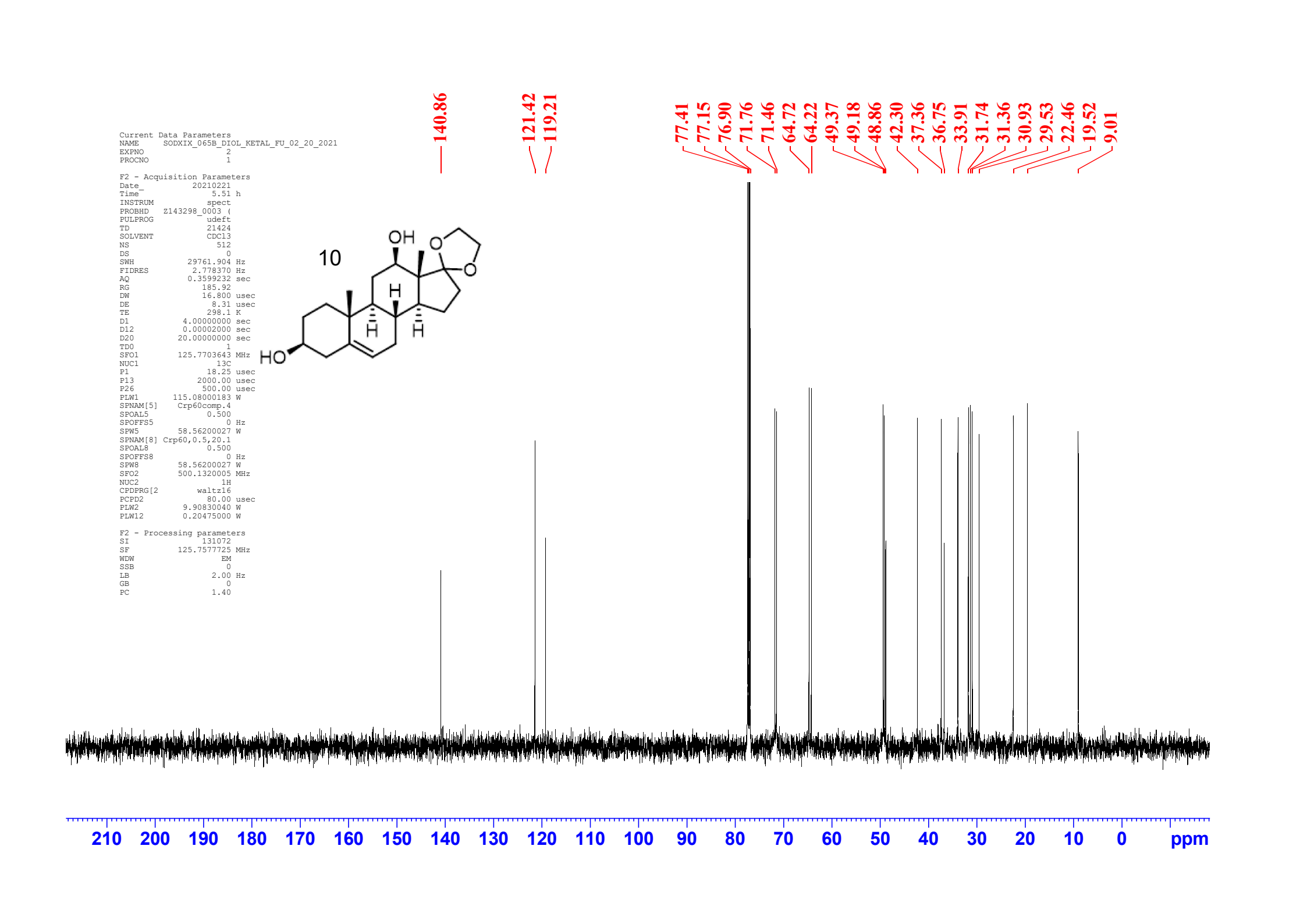


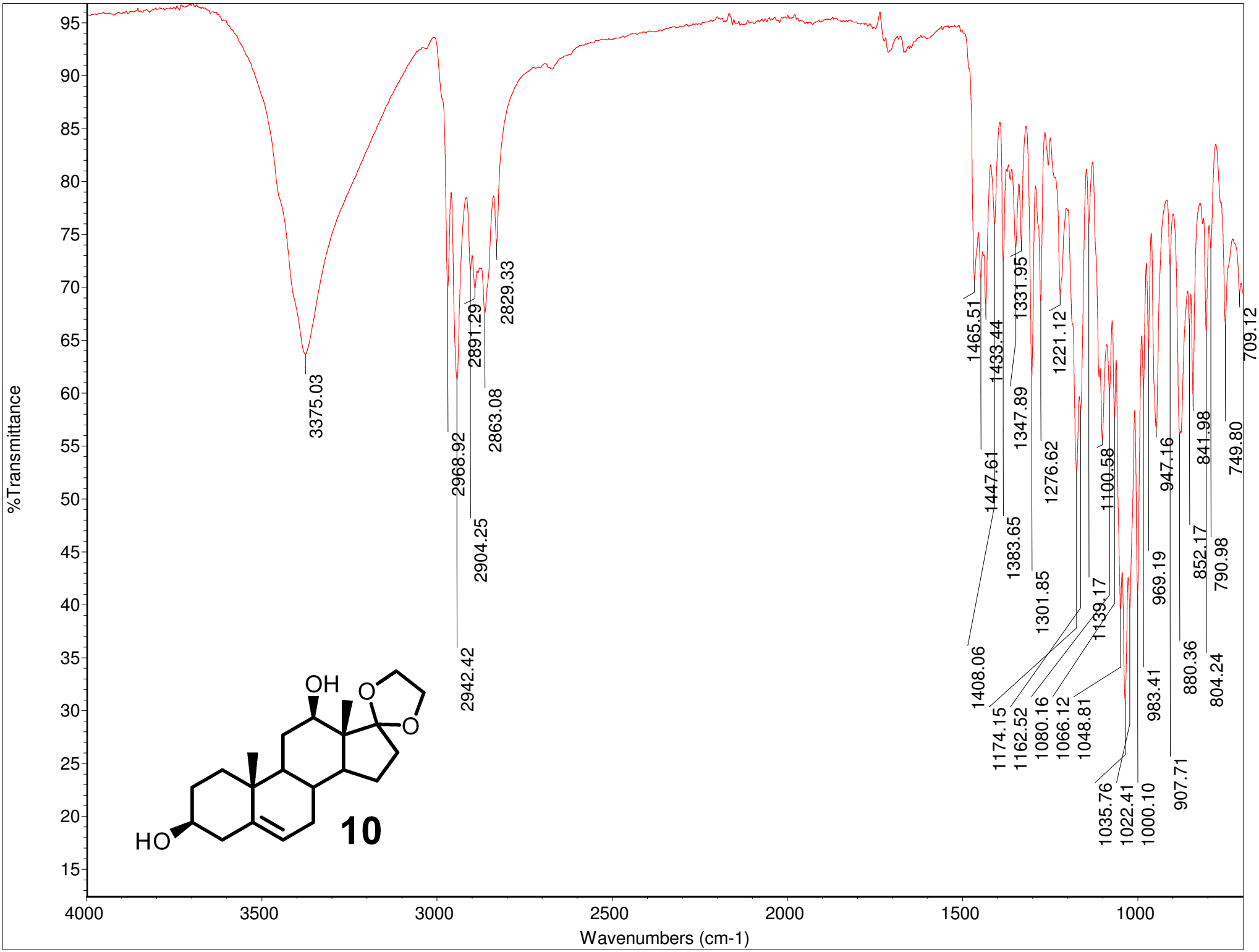
$\frac{12.8\,mg}{10\,mL}$ = 1 [illegible]

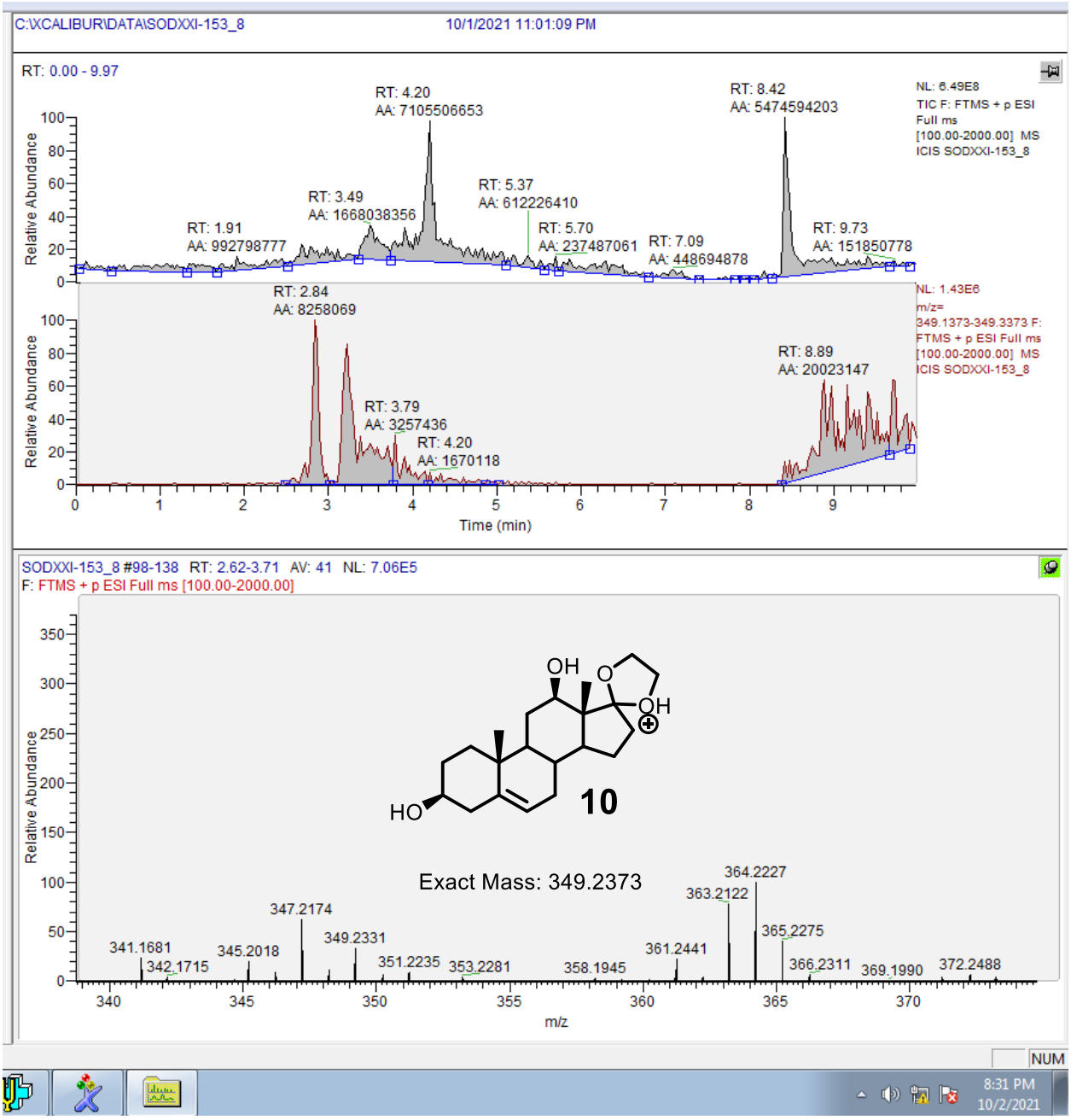
$CHCl_3$

-0.025


5.351 5.346 5.340 4.020 4.010 3.999 3.994 3.991 3.987 3.983 3.979 3.976 3.971 3.960 3.957 3.898 3.889 3.885 3.882 3.873 3.519 2.299 2.294 1.921 1.864 1.858 1.846 1.838 1.836 1.828 1.816 1.800 1.792 1.766 1.712 1.704 1.701 1.694 1.533 1.520 1.515 1.511 1.505 1.487 1.483 1.465 1.457 1.432 1.408 1.390 1.383 1.375 1.361 1.028 0.931
Current Data Parameters
NAME SODXIX_065B_DIOL_KETAL_PU_02_20_2021
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210221
Time 5.08 h
INSTRUM spect
PROBHD Z143298_0003 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 103.84
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 3.83 usec
P1 11.50 usec
PLW1 9.90830040 W
F2 - Processing parameters
SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
OH
O
O
H
H
H
HO
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00 4.09 1.08 1.00 1.04 1.07 1.13 1.10 4.37 1.92 7.16 2.11 3.14 3.02


Current Data Parameters
NAME SODXIX_065B_DIOL_KETAL_FU_02_20_2021
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210221
Time 5.51 h
INSTRUM spect
PROBHD Z143298_0003 (
PULPROG udeft
TD 21424
SOLVENT CDCl3
NS 512
DS 0
SWH 29761.904 Hz
FIDRES 2.778370 Hz
AQ 0.3599232 sec
RG 185.92
DW 16.800 usec
DE 8.31 usec
TE 298.1 K
D1 4.00000000 sec
D12 0.00002000 sec
D20 20.00000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 18.25 usec
P13 2000.00 usec
P26 500.00 usec
PLW1 115.08000183 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 58.56200027 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 58.56200027 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 9.90830040 W
PLW12 0.20475000 W
F2 - Processing parameters
SI 131072
SF 125.7577725 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
10
OH
O
O
H
H
H
HO
140.86
121.42
119.21
77.41
77.15
76.90
71.76
71.46
64.72
64.22
49.37
49.18
48.86
42.30
37.36
36.75
33.91
31.74
31.36
30.93
29.53
22.46
19.52
9.01
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


%Transmittance
Wavenumbers (cm-1)
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
4000
3500
3000
2500
2000
1500
1000
3375.03
2968.92
2942.42
2904.25
2891.29
2863.08
2829.33
1465.51
1447.61
1433.44
1408.06
1383.65
1347.89
1331.95
1301.85
1276.62
1221.12
1174.15
1162.52
1139.17
1100.58
1080.16
1066.12
1048.81
1035.76
1022.41
1000.10
983.41
969.19
947.16
907.71
880.36
852.17
841.98
804.24
790.98
749.80
709.12
OH
O
O
HO
10


C:\XCALIBUR\DATA\SODXXI-153_8
10/1/2021 11:01:09 PM
RT: 0.00 - 9.97
RT: 4.20
AA: 7105506653
RT: 8.42
AA: 5474594203
NL: 6.49E8
TIC F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_8
RT: 3.49
AA: 1668038356
RT: 5.37
AA: 612226410
RT: 1.91
AA: 992798777
RT: 5.70
AA: 237487061
RT: 7.09
AA: 448694878
RT: 9.73
AA: 151850778
Relative Abundance
RT: 2.84
AA: 8258069
NL: 1.43E6
m/z= 349.1373-349.3373 F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_8
RT: 8.89
AA: 20023147
RT: 3.79
AA: 3257436
RT: 4.20
AA: 1670118
Time (min)
SODXXI-153_8 #98-138 RT: 2.62-3.71 AV: 41 NL: 7.06E5
F: FTMS + p ESI Full ms [100.00-2000.00]
OH
O
OH
HO
10
Exact Mass: 349.2373
364.2227
363.2122
347.2174
365.2275
349.2331
341.1681
345.2018
361.2441
342.1715
351.2235
353.2281
358.1945
366.2311
369.1990
372.2488
m/z
NUM
8:31 PM
10/2/2021

LAXCO Optical Rotation

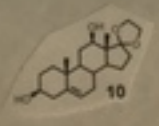
Original O.R. : -0.126° O.R. °

-0.126

Temp. : 21.2℃ 21.2℃

WLG	Cell			ATC
589.44	1.0000dm			20.0
Mode	Times		GLP	
single	1		Off	

database save

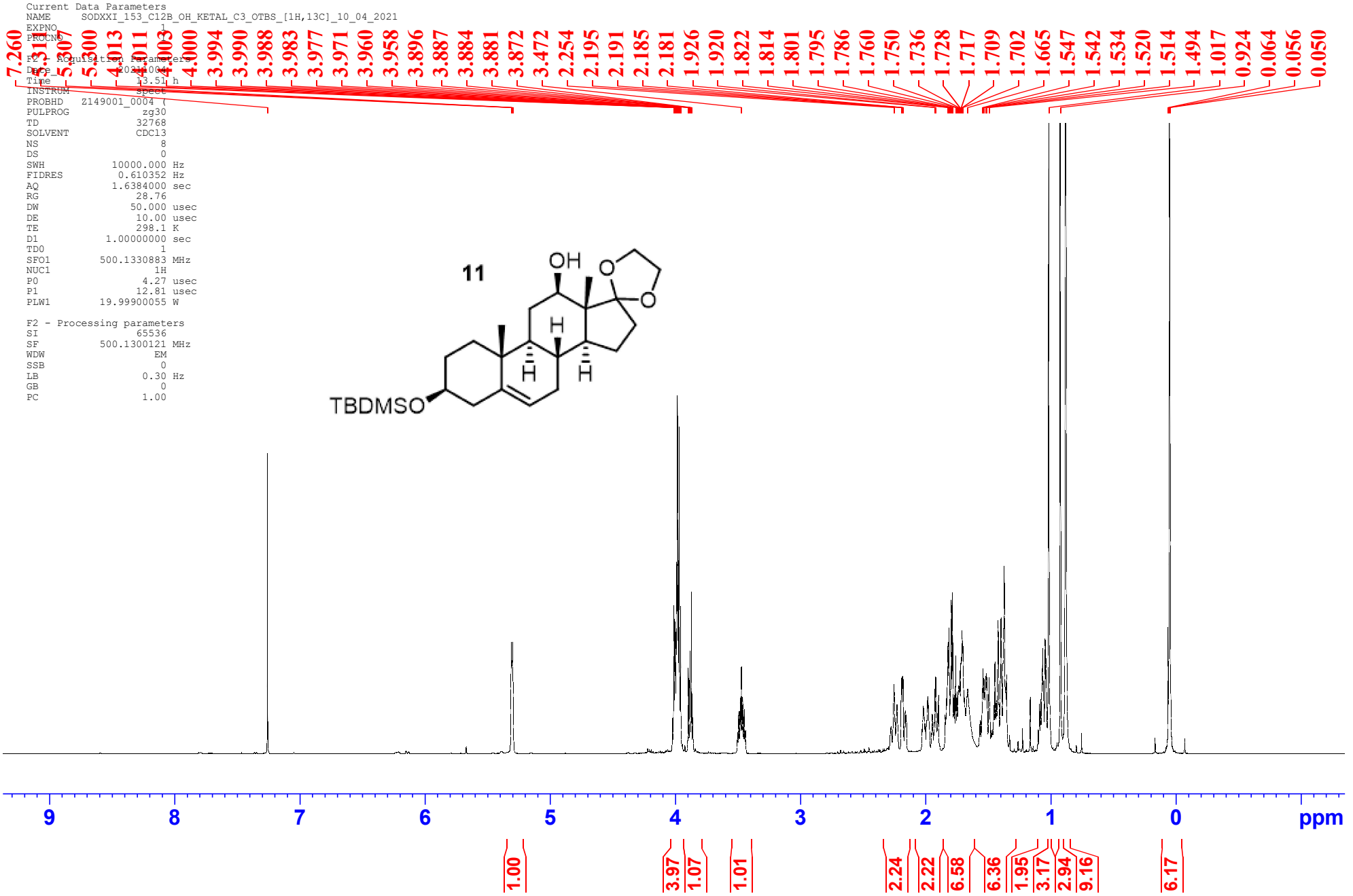


Diol- ketal

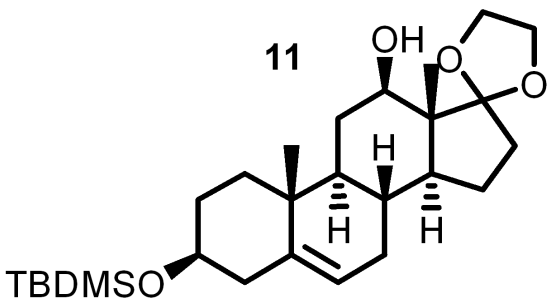
10~~3~~ mg

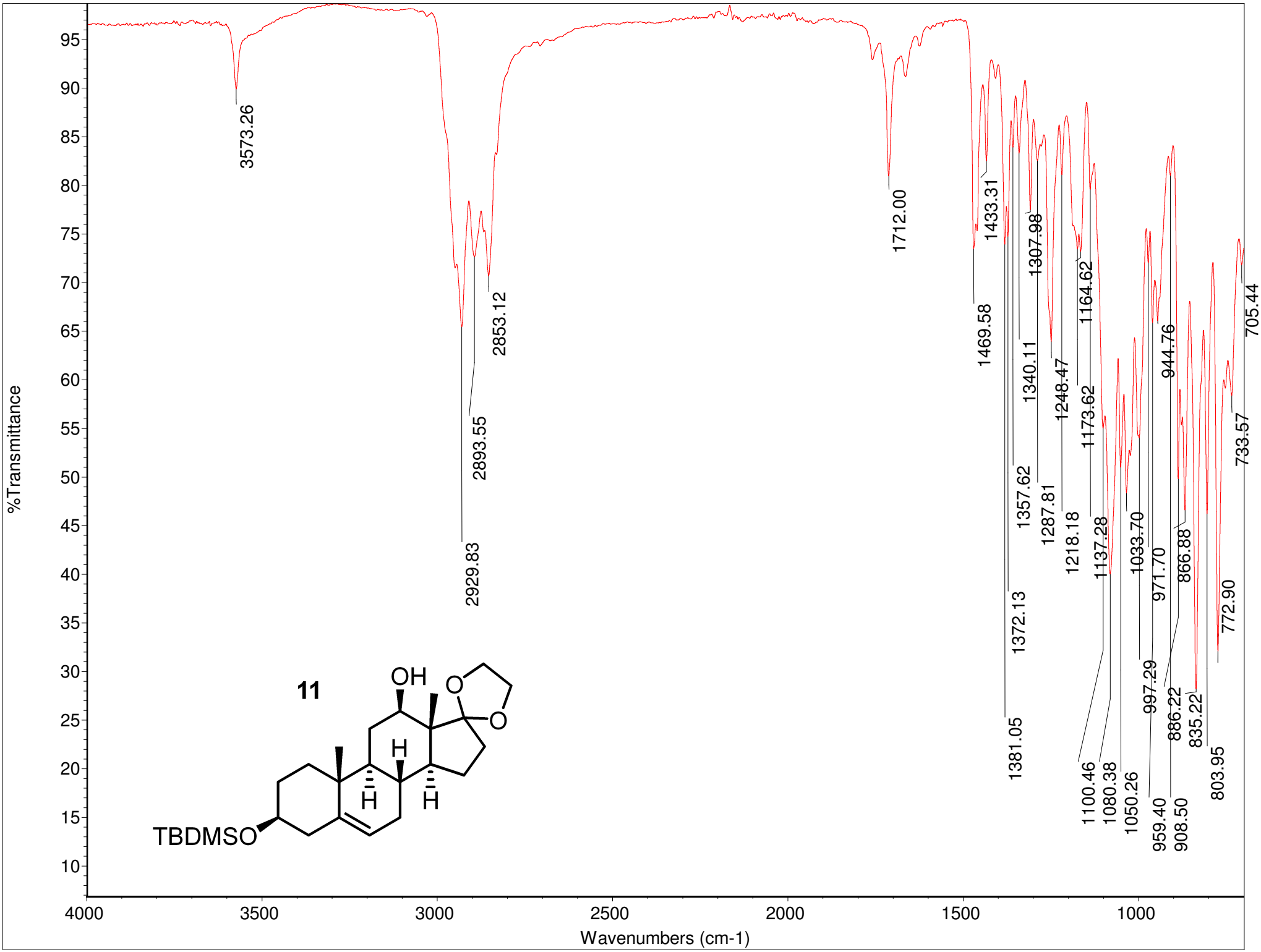
10 ml of $CHCl_3$

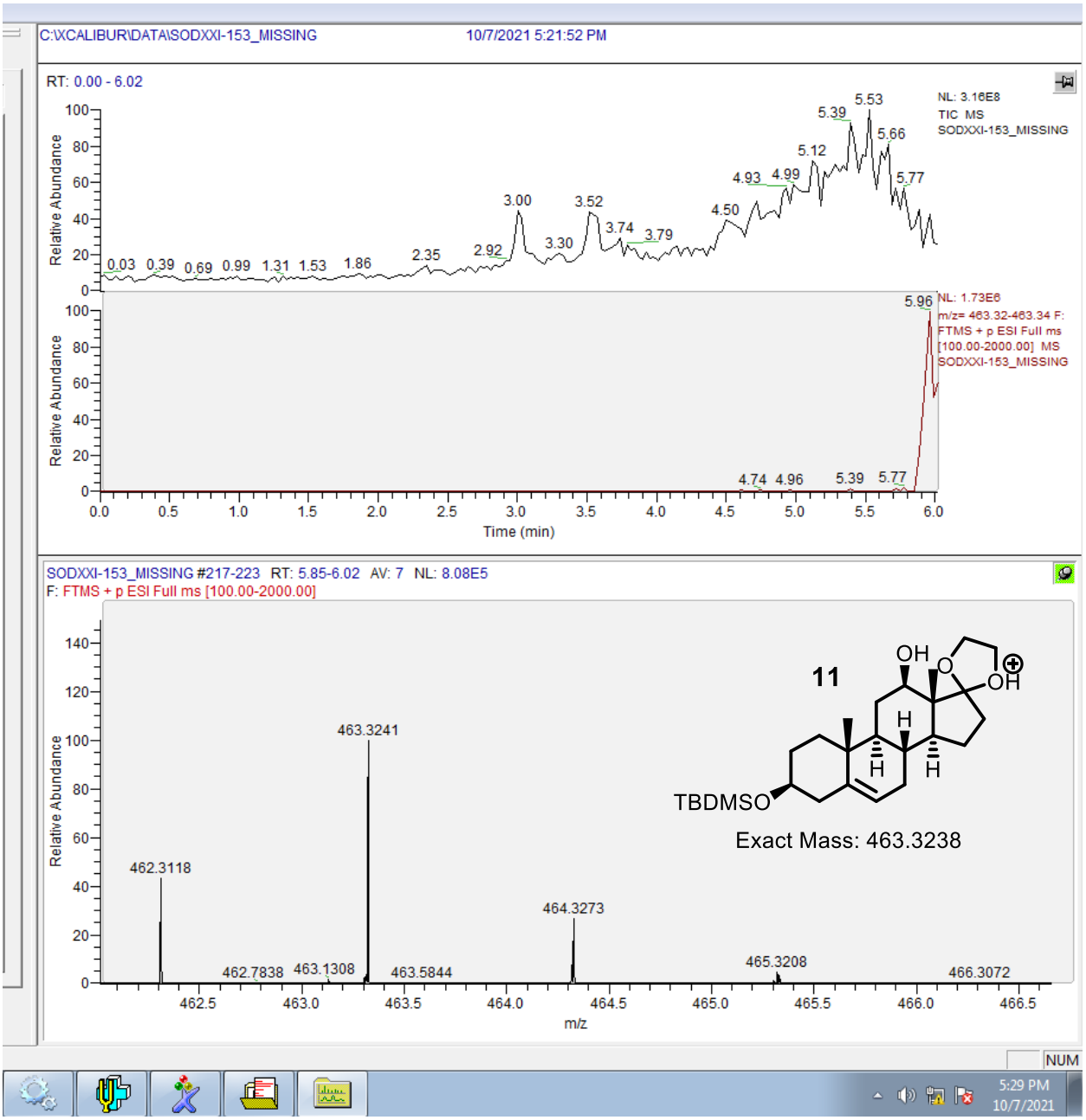
=> -0.126


Current Data Parameters
NAME SODXXI_153_C12B_OH_KETAL_C3_OTBS_[1H,13C]_10_04_2021
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210406
Time 13.51 h
INSTRUM spect
PROBHD Z149001_0004 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 28.76
DW 50.000 usec
DE 10.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 4.27 usec
P1 12.81 usec
PLW1 19.99900055 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
11
OH
O
O
H
H
H
TBDMSO
7.260
5.307
5.300
4.013
4.011
4.003
4.000
3.994
3.990
3.988
3.983
3.977
3.971
3.960
3.958
3.896
3.887
3.884
3.881
3.872
3.472
2.254
2.195
2.191
2.185
2.181
1.926
1.920
1.822
1.814
1.801
1.795
1.786
1.760
1.750
1.736
1.728
1.717
1.709
1.702
1.665
1.547
1.542
1.534
1.520
1.514
1.494
1.017
0.924
0.064
0.056
0.050
9
8
7
6
5
4
3
2
1
0
ppm
1.00
3.97
1.07
1.01
2.24
2.22
6.58
6.36
1.95
3.17
2.94
9.16
6.17

SODXXI_153_C12B_OH_KETAL_C3_OTBS_[1H,13C]_10_04_2021




%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
3573.26
2929.83
2893.55
2853.12
1712.00
1469.58
1433.31
1381.05
1372.13
1357.62
1340.11
1307.98
1287.81
1248.47
1218.18
1173.62
1164.62
1137.28
1100.46
1080.38
1050.26
1033.70
997.29
971.70
959.40
944.76
908.50
886.22
866.88
835.22
803.95
772.90
733.57
705.44
11
OH
O
O
H
H
H
TBDMSO


C:\XCALIBUR\DATA\SODXXI-153_MISSING 10/7/2021 5:21:52 PM
RT: 0.00 - 6.02
NL: 3.16E8
TIC MS
SODXXI-153_MISSING
Relative Abundance
Time (min)
NL: 1.73E6
m/z= 463.32-463.34 F:
FTMS + p ESI Full ms
[100.00-2000.00] MS
SODXXI-153_MISSING
SODXXI-153_MISSING #217-223 RT: 5.85-6.02 AV: 7 NL: 8.08E5
F: FTMS + p ESI Full ms [100.00-2000.00]
11
TBDMSO
Exact Mass: 463.3238
m/z
5:29 PM
10/7/2021

LAXCO Optical Rotation

Original O.R. : 0.009° O.R. °

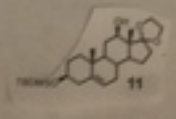
0. 009

Temp. : 21.4℃ 21.4℃

WLG | Cell | ATC

Mode | Times | GLP

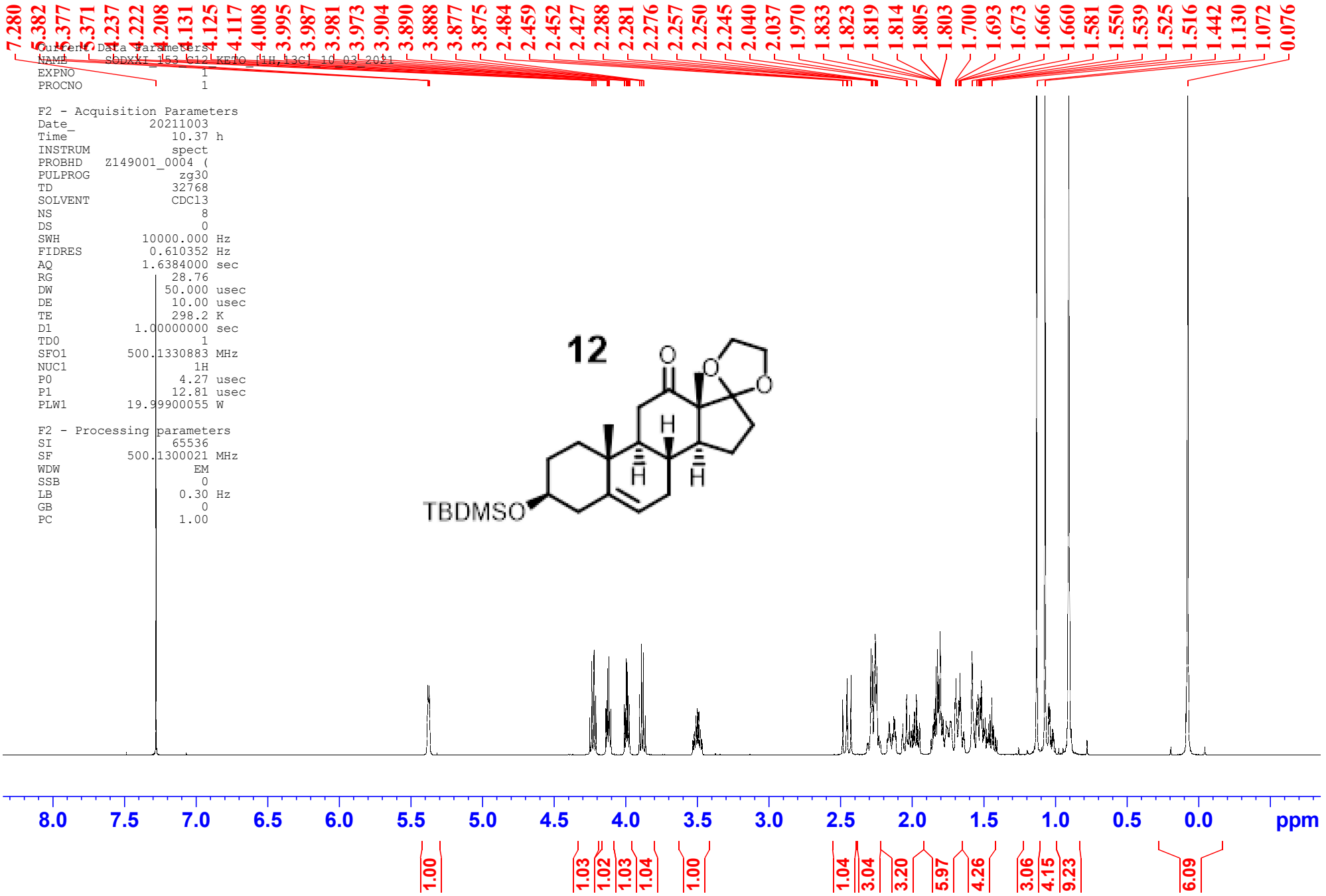
Off



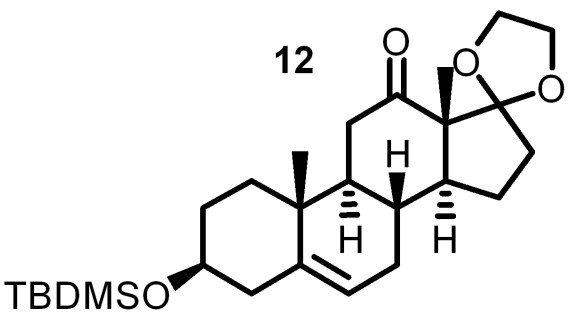
192 mg

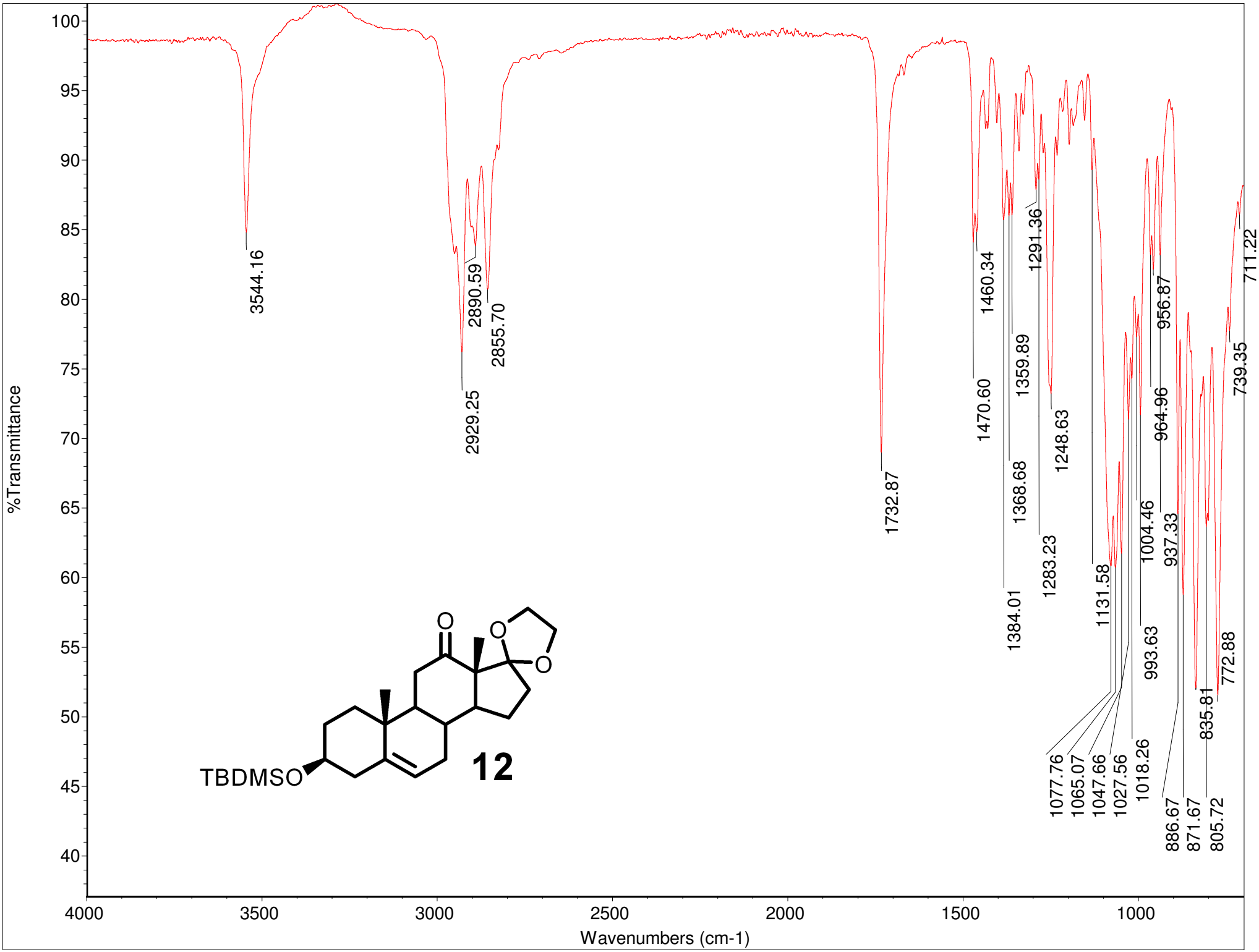
10 mL $CHCl_3$

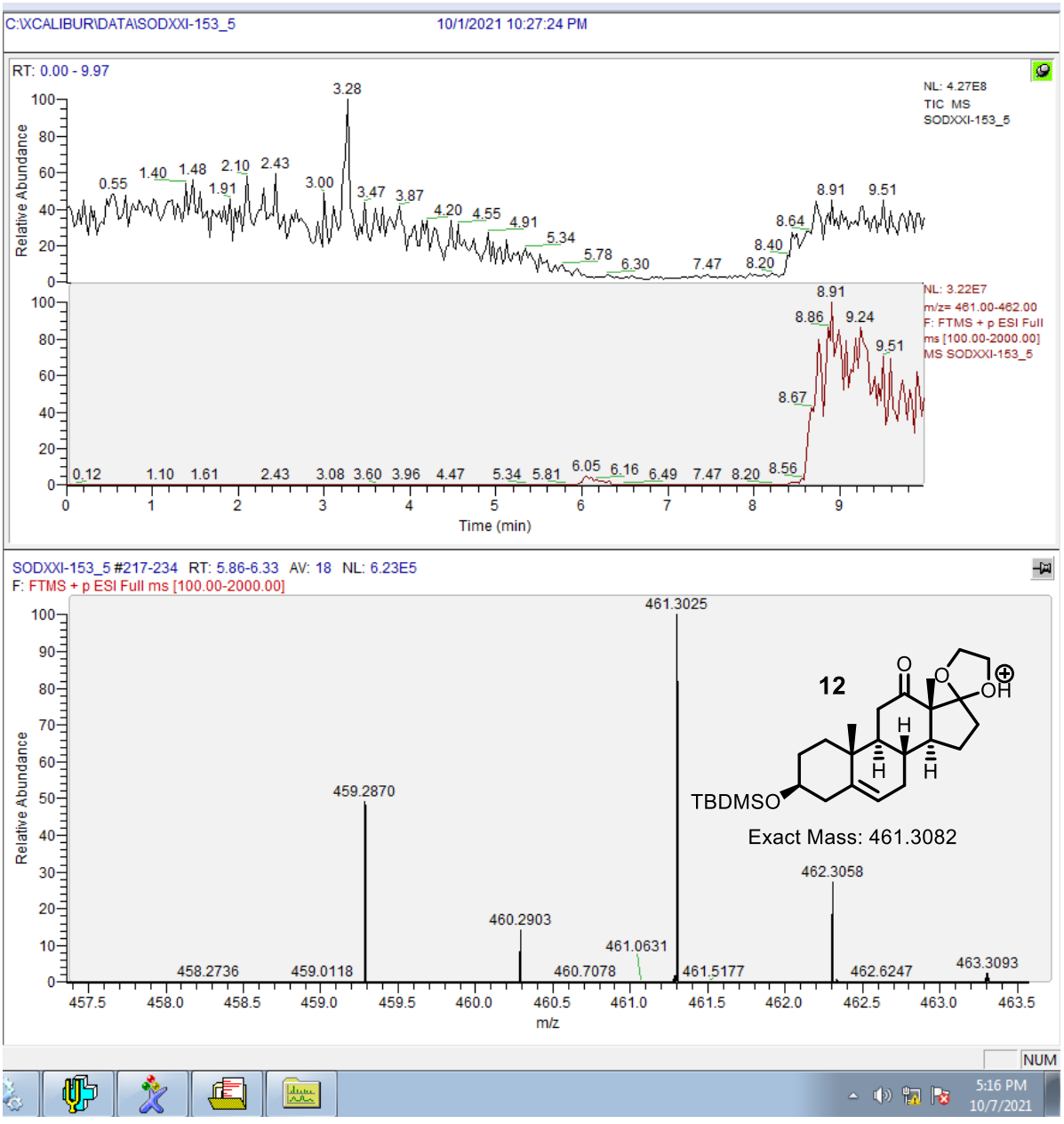
0.009

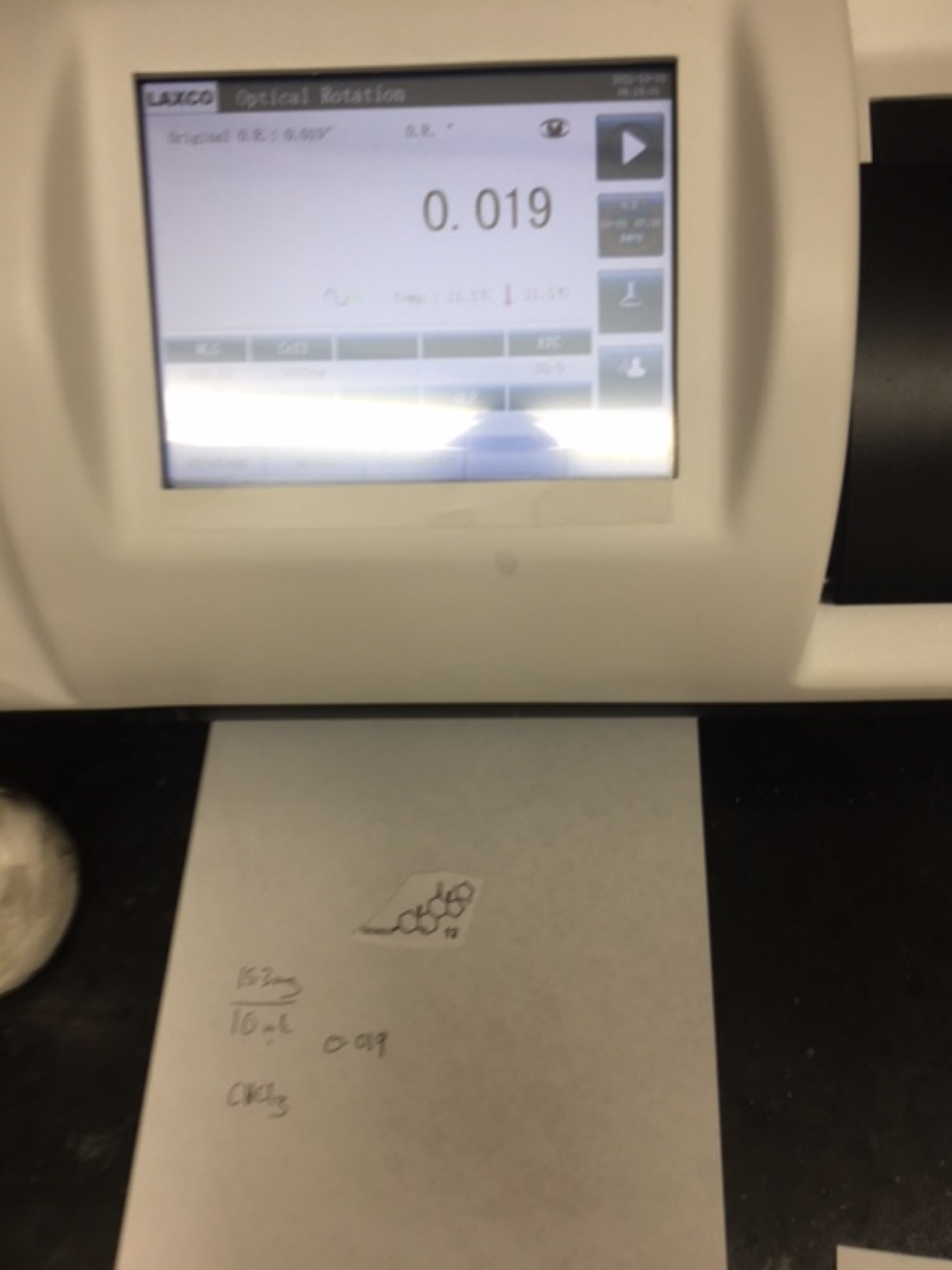

7.280
5.382
5.377
5.371
5.237
4.222
4.208
4.131
4.125
4.117
4.008
3.995
3.987
3.981
3.973
3.904
3.890
3.888
3.877
3.875
2.484
2.459
2.452
2.427
2.288
2.281
2.276
2.257
2.250
2.245
2.040
2.037
1.970
1.833
1.823
1.819
1.814
1.805
1.803
1.700
1.693
1.673
1.666
1.660
1.581
1.550
1.539
1.525
1.516
1.442
1.130
1.072
0.076
Current Data Parameters
NAME sbdxxi_153_c12_KETO_[1H,13C]_10_03_2021
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211003
Time 10.37 h
INSTRUM spect
PROBHD Z149001_0004 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 28.76
DW 50.000 usec
DE 10.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 4.27 usec
P1 12.81 usec
PLW1 19.99900055 W
F2 - Processing parameters
SI 65536
SF 500.1300021 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12
TBDMSO
O
O
O
H
H
H
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.03
1.02
1.03
1.04
1.00
1.04
3.04
3.20
5.97
4.26
3.06
4.15
9.23
6.09

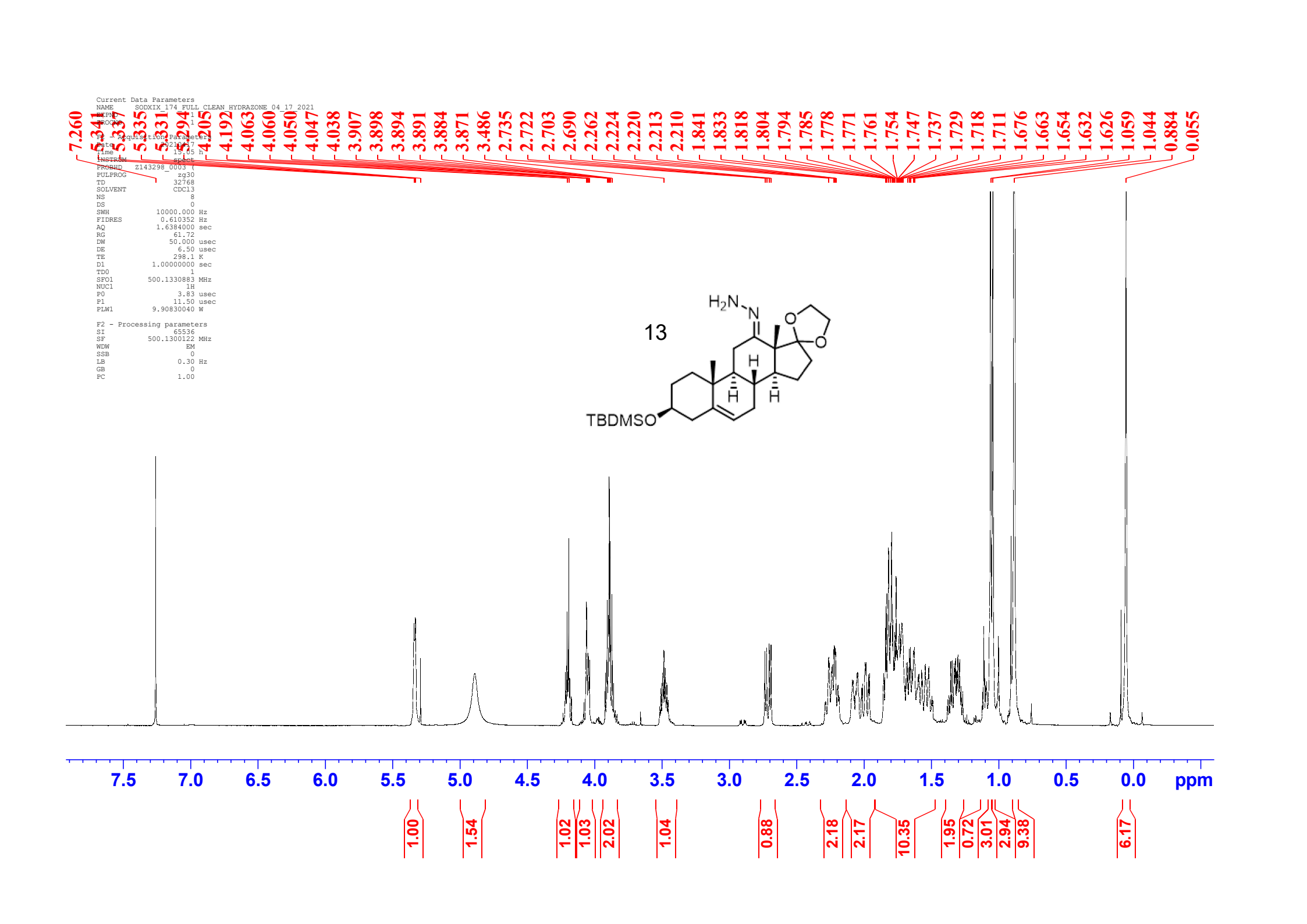
SODXXI_153_C12_KETO_[1H,13C]_10_03_2021

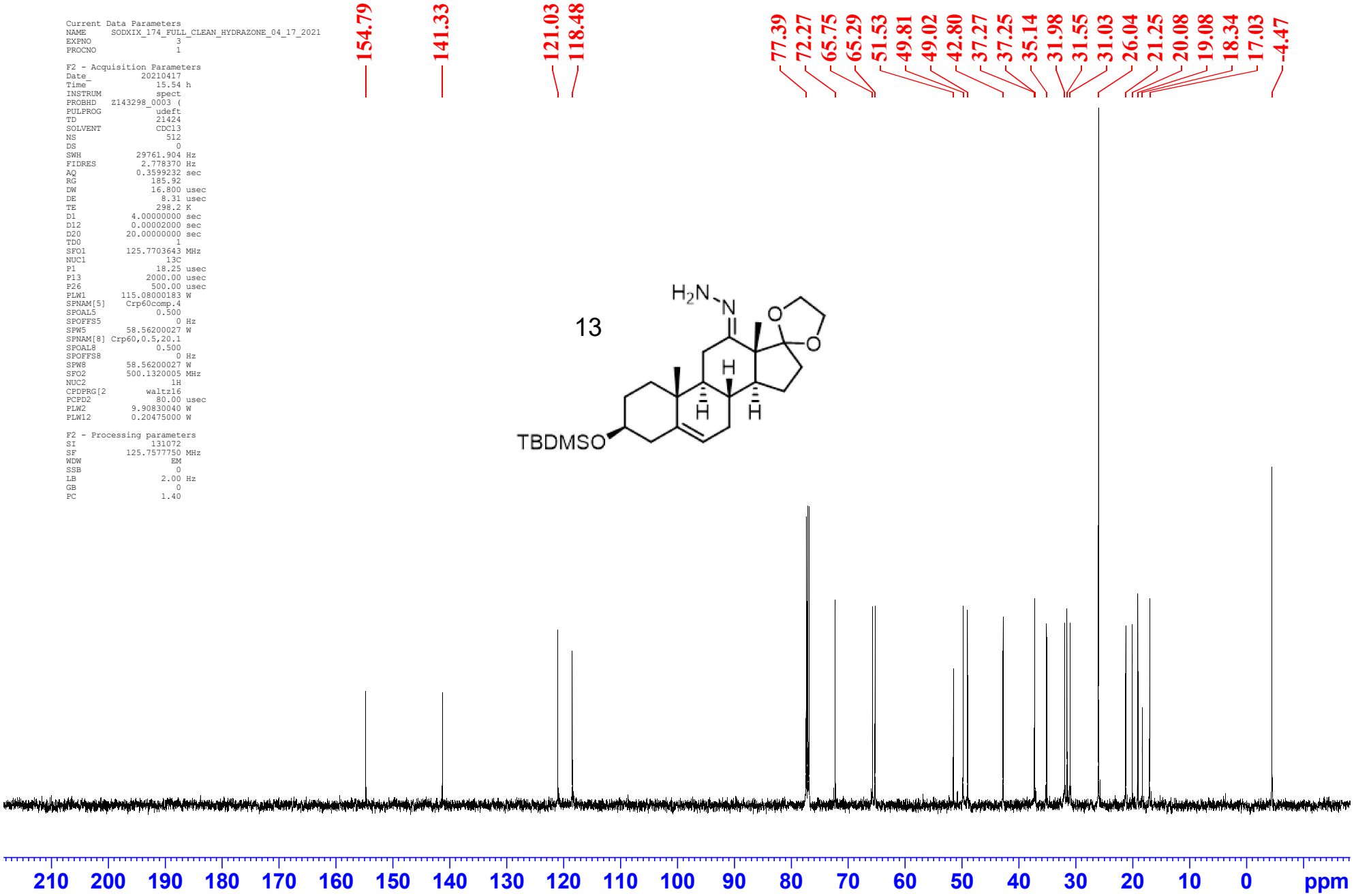


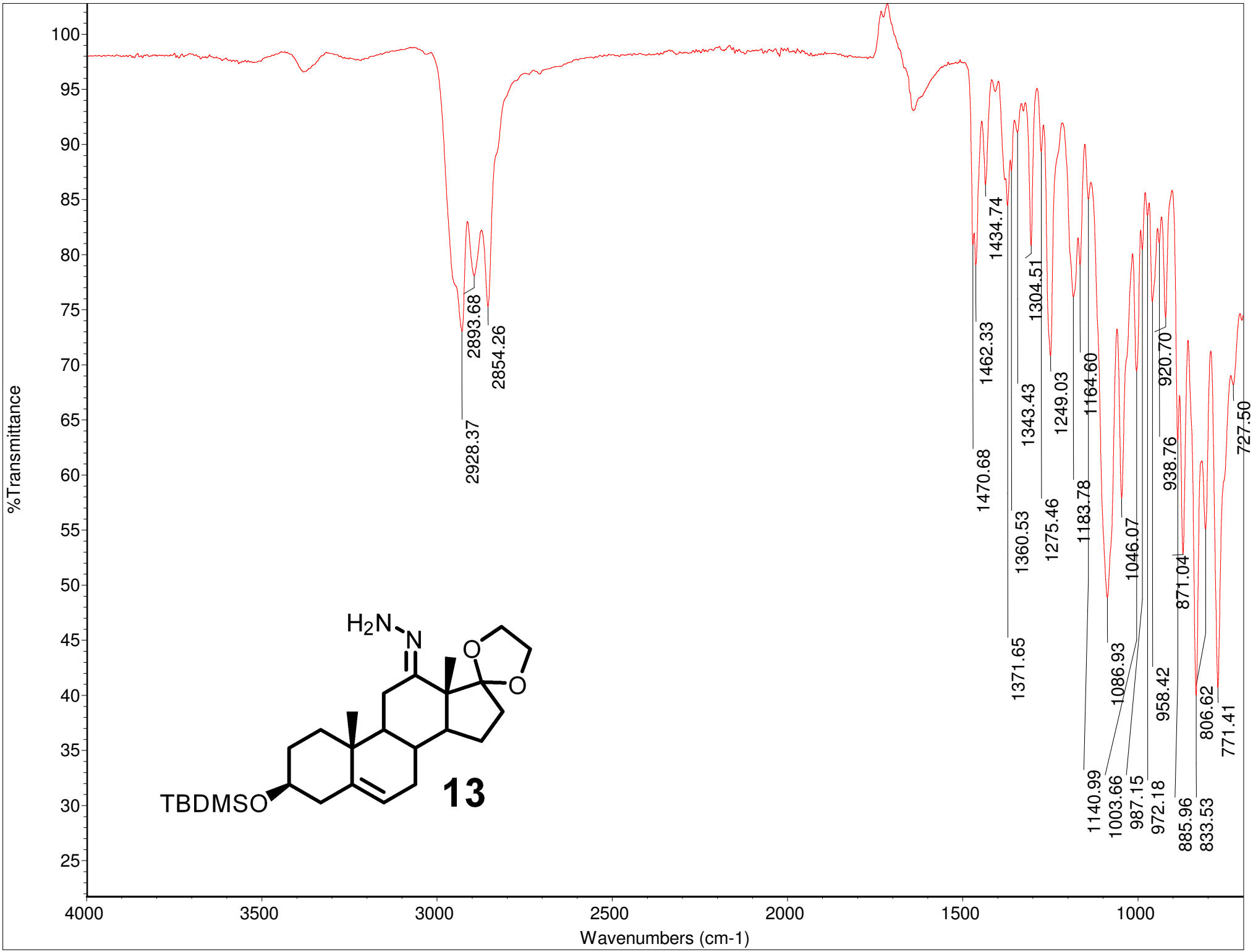

%Transmittance
Wavenumbers (cm-1)
100
95
90
85
80
75
70
65
60
55
50
45
40
4000
3500
3000
2500
2000
1500
1000
3544.16
2929.25
2890.59
2855.70
1732.87
1470.60
1460.34
1384.01
1368.68
1359.89
1291.36
1283.23
1248.63
1131.58
1077.76
1065.07
1047.66
1027.56
1018.26
1004.46
993.63
964.96
956.87
937.33
886.67
871.67
835.81
805.72
772.88
739.35
711.22
TBDMSO
O
O
O
12

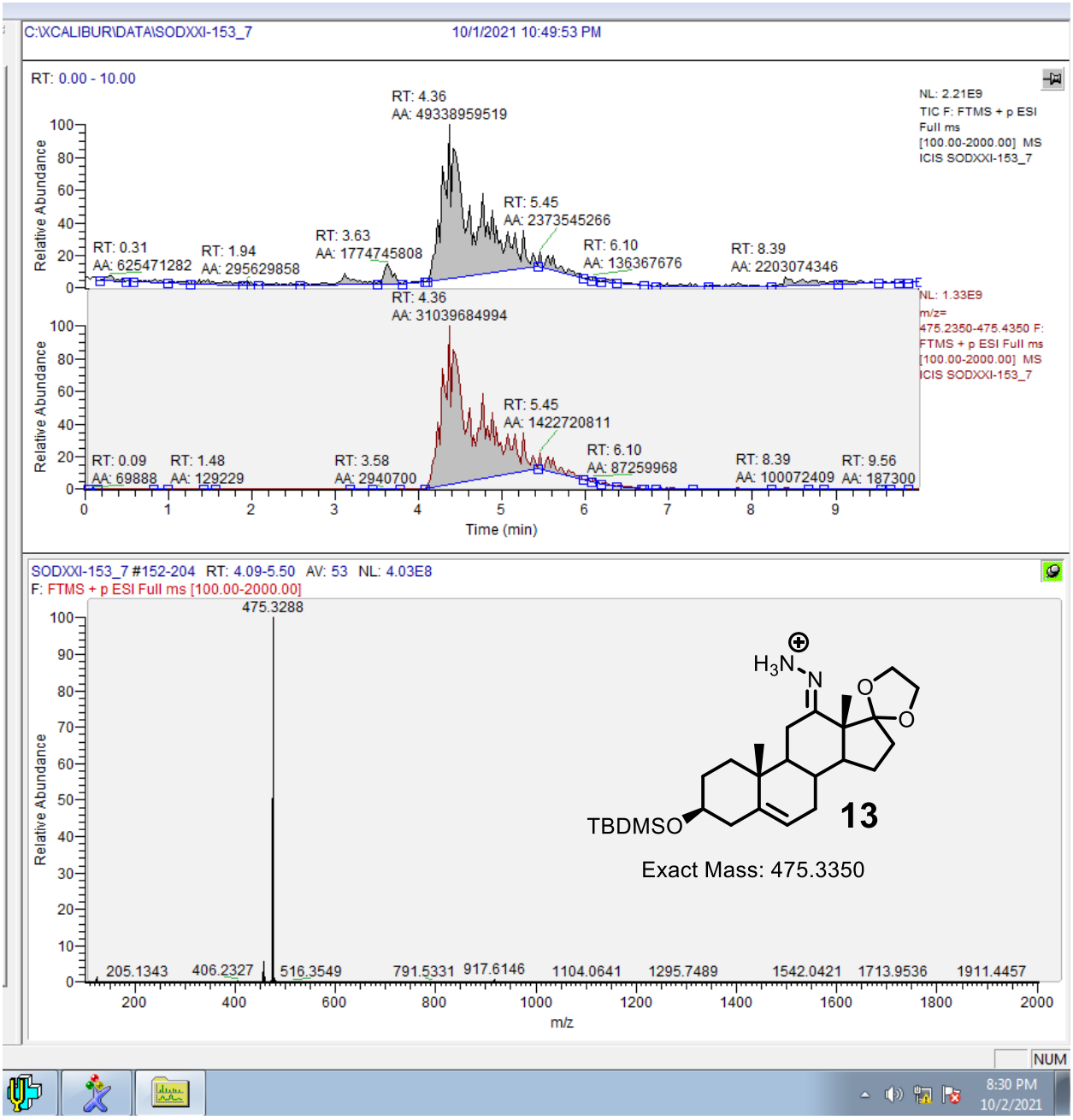

C:\XCALIBUR\DATA\SODXXI-153_5
10/1/2021 10:27:24 PM
RT: 0.00 - 9.97
NL: 4.27E8
TIC MS
SODXXI-153_5
Relative Abundance
Time (min)
NL: 3.22E7
m/z= 461.00-462.00
F: FTMS + p ESI Full
ms [100.00-2000.00]
MS SODXXI-153_5
SODXXI-153_5 #217-234 RT: 5.86-6.33 AV: 18 NL: 6.23E5
F: FTMS + p ESI Full ms [100.00-2000.00]
461.3025
459.2870
460.2903
461.0631
458.2736
459.0118
460.7078
461.5177
462.3058
462.6247
463.3093
12
TBDMSO
Exact Mass: 461.3082
m/z
NUM
5:16 PM
10/7/2021


LAXCO
Optical Rotation
0.019
ATC


Current Data Parameters
NAME SODXIX_174_FULL_CLEAN_HYDRAZONE_04_17_2021
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210417
Time 15.05 h
INSTRM spect
PROBHD Z143298_0003 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 61.72
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 3.83 usec
P1 11.50 usec
PLW1 9.90830040 W
F2 - Processing parameters
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.260
5.341
5.337
5.335
5.331
5.294
4.205
4.192
4.063
4.060
4.050
4.047
4.038
3.907
3.898
3.894
3.891
3.884
3.871
3.486
2.735
2.722
2.703
2.690
2.262
2.224
2.220
2.213
2.210
1.841
1.833
1.818
1.804
1.794
1.785
1.778
1.771
1.761
1.754
1.747
1.737
1.729
1.718
1.711
1.676
1.663
1.654
1.632
1.626
1.059
1.044
0.884
0.055
13
H2N
N
O
O
H
H
H
TBDMSO
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
1.54
1.02
1.03
2.02
1.04
0.88
2.18
2.17
10.35
1.95
0.72
3.01
2.94
9.38
6.17


Current Data Parameters
NAME SODXIX_174_FULL_CLEAN_HYDRAZONE_04_17_2021
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210417
Time 15.54 h
INSTRUM spect
PROBHD Z143298_0003 (
PULPROG udeft
TD 21424
SOLVENT CDCl3
NS 512
DS 0
SWH 29761.904 Hz
FIDRES 2.778370 Hz
AQ 0.3599232 sec
RG 185.92
DW 16.800 usec
DE 8.31 usec
TE 298.2 K
D1 4.00000000 sec
D12 0.00002000 sec
D20 20.00000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 18.25 usec
P13 2000.00 usec
P26 500.00 usec
PLW1 115.08000183 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 58.56200027 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 58.56200027 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 9.90830040 W
PLW12 0.20475000 W
F2 - Processing parameters
SI 131072
SF 125.7577750 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
154.79
141.33
121.03
118.48
77.39
72.27
65.75
65.29
51.53
49.81
49.02
42.80
37.27
37.25
35.14
31.98
31.55
31.03
26.04
21.25
20.08
19.08
18.34
17.03
-4.47
13
H2N
N
O
O
H
H
H
TBDMSO
210
200
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)
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
4000
3500
3000
2500
2000
1500
1000
2928.37
2893.68
2854.26
1470.68
1462.33
1434.74
1371.65
1360.53
1343.43
1304.51
1275.46
1249.03
1183.78
1164.60
1140.99
1086.93
1046.07
1003.66
987.15
972.18
958.42
938.76
920.70
885.96
871.04
833.53
806.62
771.41
727.50
H2N
N
O
O
TBDMSO
13


C:\XCALIBUR\DATA\SODXXI-153_7
10/1/2021 10:49:53 PM
RT: 0.00 - 10.00
NL: 2.21E9
TIC F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_7
RT: 4.36
AA: 49338959519
RT: 5.45
AA: 2373545266
RT: 3.63
AA: 1774745808
RT: 6.10
AA: 136367676
RT: 8.39
AA: 2203074346
RT: 0.31
AA: 625471282
RT: 1.94
AA: 295629858
NL: 1.33E9
m/z= 475.2350-475.4350 F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_7
RT: 4.36
AA: 31039684994
RT: 5.45
AA: 1422720811
RT: 6.10
AA: 87259968
RT: 3.58
AA: 2940700
RT: 8.39
AA: 100072409
RT: 9.56
AA: 187300
RT: 0.09
AA: 69888
RT: 1.48
AA: 129229
Relative Abundance
Time (min)
SODXXI-153_7 #152-204 RT: 4.09-5.50 AV: 53 NL: 4.03E8
F: FTMS + p ESI Full ms [100.00-2000.00]
475.3288
205.1343
406.2327
516.3549
791.5331
917.6146
1104.0641
1295.7489
1542.0421
1713.9536
1911.4457
m/z
TBDMSO
13
Exact Mass: 475.3350
NUM
8:30 PM
10/2/2021

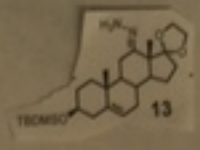
Original O.R. : -0.120° O.R. °

-0.120

Temp. : 21.6℃ 21.5℃

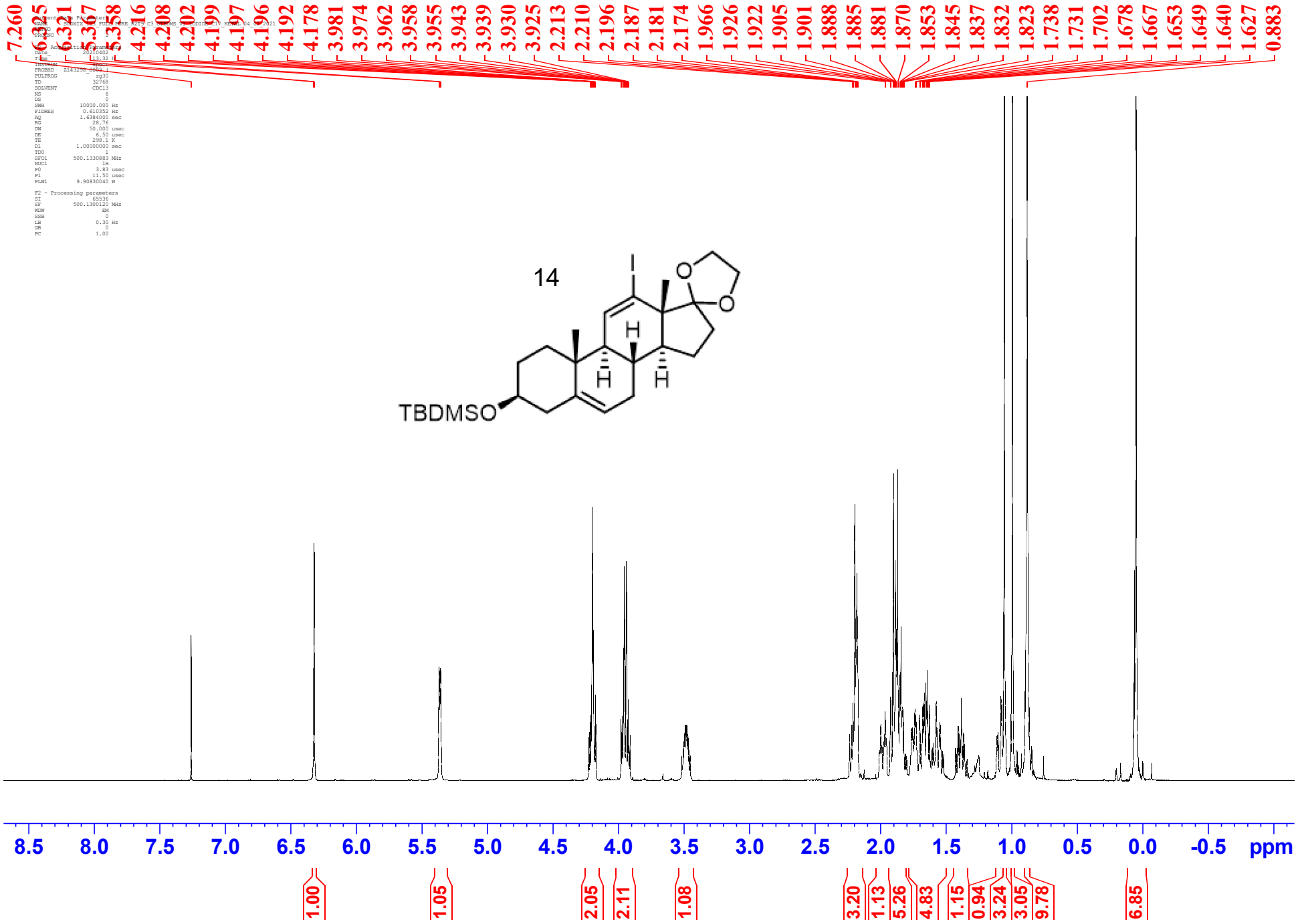
Mode	Times		GLP	
single	1		Off	

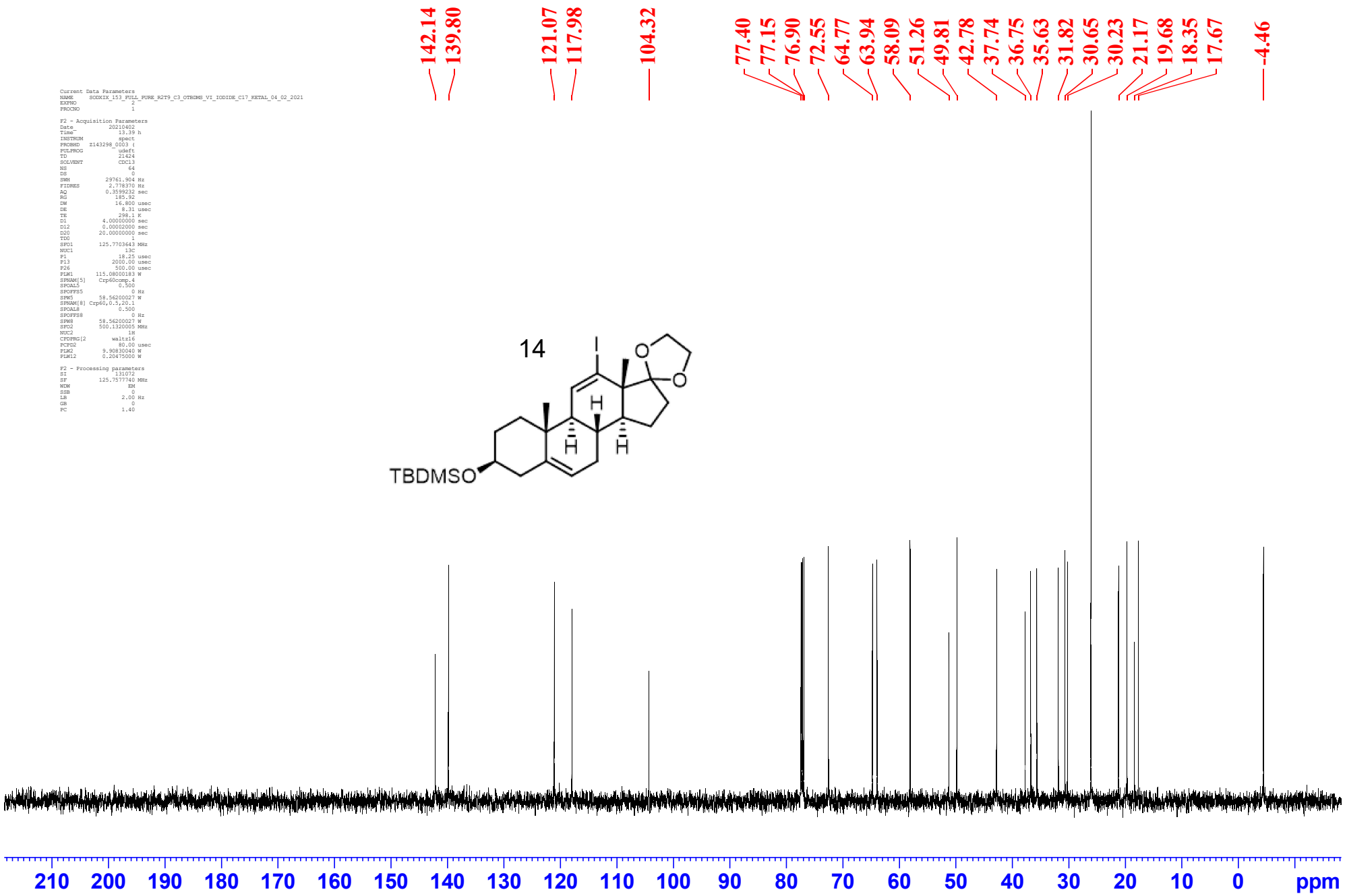
system menu database save

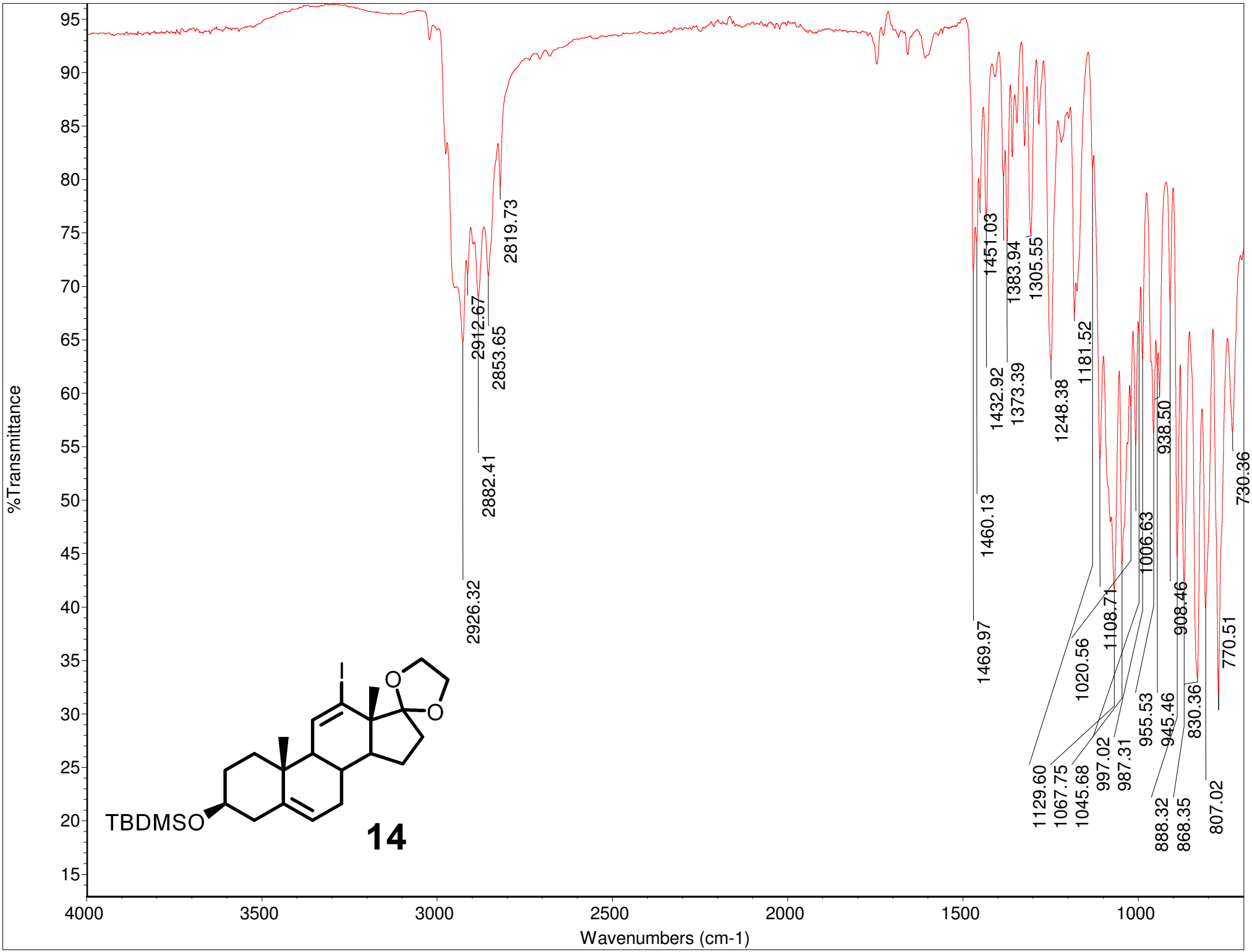


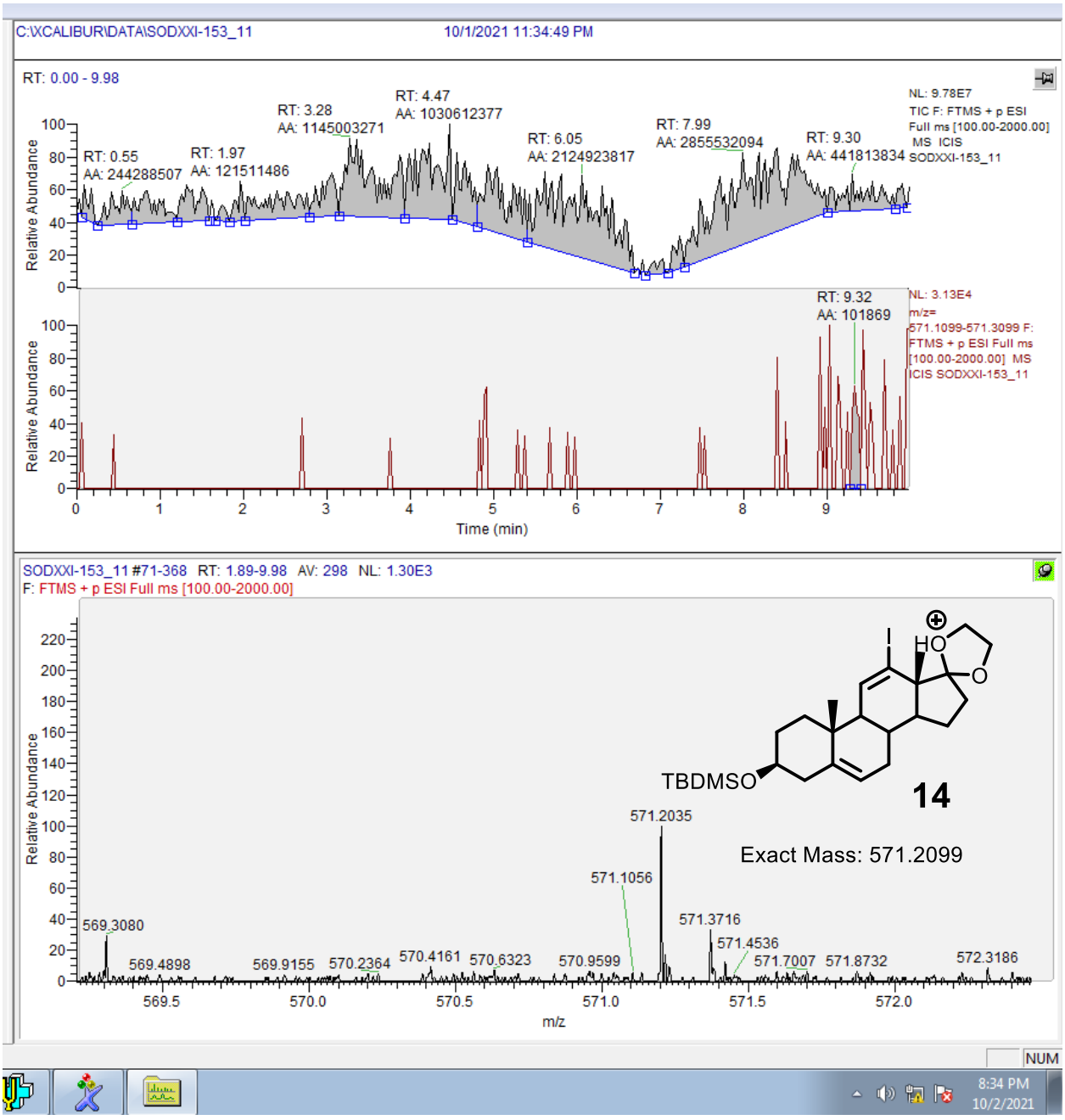
$\frac{14.4\ mg}{10\ ml}$ -0.120

$CHCl_3$


7.260
6.325
6.311
5.377
5.358
4.216
4.208
4.202
4.199
4.197
4.196
4.192
4.178
3.981
3.974
3.962
3.958
3.955
3.943
3.939
3.930
3.925
2.213
2.210
2.196
2.187
2.181
2.174
1.966
1.926
1.922
1.905
1.901
1.888
1.885
1.881
1.870
1.853
1.845
1.837
1.832
1.823
1.738
1.731
1.702
1.678
1.667
1.653
1.649
1.640
1.627
0.883
14
I
O
O
H
H
H
TBDMSO
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
ppm
1.00
1.05
2.05
2.11
1.08
3.20
1.13
5.26
4.83
1.15
0.94
3.24
3.05
9.78
6.85


142.14
139.80
121.07
117.98
104.32
77.40
77.15
76.90
72.55
64.77
63.94
58.09
51.26
49.81
42.78
37.74
36.75
35.63
31.82
30.65
30.23
21.17
19.68
18.35
17.67
-4.46
14
I
O
O
H
H
H
TBDMSO
Current Data Parameters
NAME SODXIX_153_FULL_PURE_R2T9_C3_OTBDMS_VI_IODIDE_C17_KETAL_04_02_2021
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210402
Time 13.39 h
INSTRUM spect
PROBHD Z143298_0003 (
PULPROG udeft
TD 21424
SOLVENT CDCl3
NS 64
DS 0
SWH 29761.904 Hz
FIDRES 2.778370 Hz
AQ 0.3599232 sec
RG 185.92
DW 16.800 usec
DE 8.31 usec
TE 298.1 K
D1 4.00000000 sec
D12 0.00002000 sec
D20 20.00000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 18.25 usec
P13 2000.00 usec
P26 500.00 usec
PLW1 115.08000183 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 58.56200027 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 58.56200027 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 9.90830040 W
PLW12 0.20475000 W
F2 - Processing parameters
SI 131072
SF 125.7577740 MHz
WDW EM
SSB 0
LB 2.00 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


%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
2926.32
2912.67
2882.41
2853.65
2819.73
1469.97
1460.13
1451.03
1432.92
1383.94
1373.39
1305.55
1248.38
1181.52
1129.60
1108.71
1067.75
1045.68
1020.56
1006.63
997.02
987.31
955.53
945.46
938.50
908.46
888.32
868.35
830.36
807.02
770.51
730.36
TBDMSO
14


C:\XCALIBUR\DATA\SODXXI-153_11
10/1/2021 11:34:49 PM
RT: 0.00 - 9.98
RT: 0.55
AA: 244288507
RT: 1.97
AA: 121511486
RT: 3.28
AA: 1145003271
RT: 4.47
AA: 1030612377
RT: 6.05
AA: 2124923817
RT: 7.99
AA: 2855532094
RT: 9.30
AA: 441813834
NL: 9.78E7
TIC F: FTMS + p ESI
Full ms [100.00-2000.00]
MS ICIS
SODXXI-153_11
Relative Abundance
RT: 9.32
AA: 101869
NL: 3.13E4
m/z=
571.1099-571.3099 F:
FTMS + p ESI Full ms
[100.00-2000.00] MS
ICIS SODXXI-153_11
Time (min)
SODXXI-153_11 #71-368 RT: 1.89-9.98 AV: 298 NL: 1.30E3
F: FTMS + p ESI Full ms [100.00-2000.00]
TBDMSO
HO
O
I
14
Exact Mass: 571.2099
571.2035
571.1056
571.3716
571.4536
569.3080
569.4898
569.9155
570.2364
570.4161
570.6323
570.9599
571.7007
571.8732
572.3186
m/z
NUM
8:34 PM
10/2/2021

LAXCO Optical Rotation

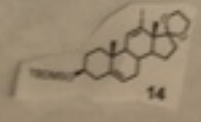
Original O.R. : -0.024° O.R. °

-0.024

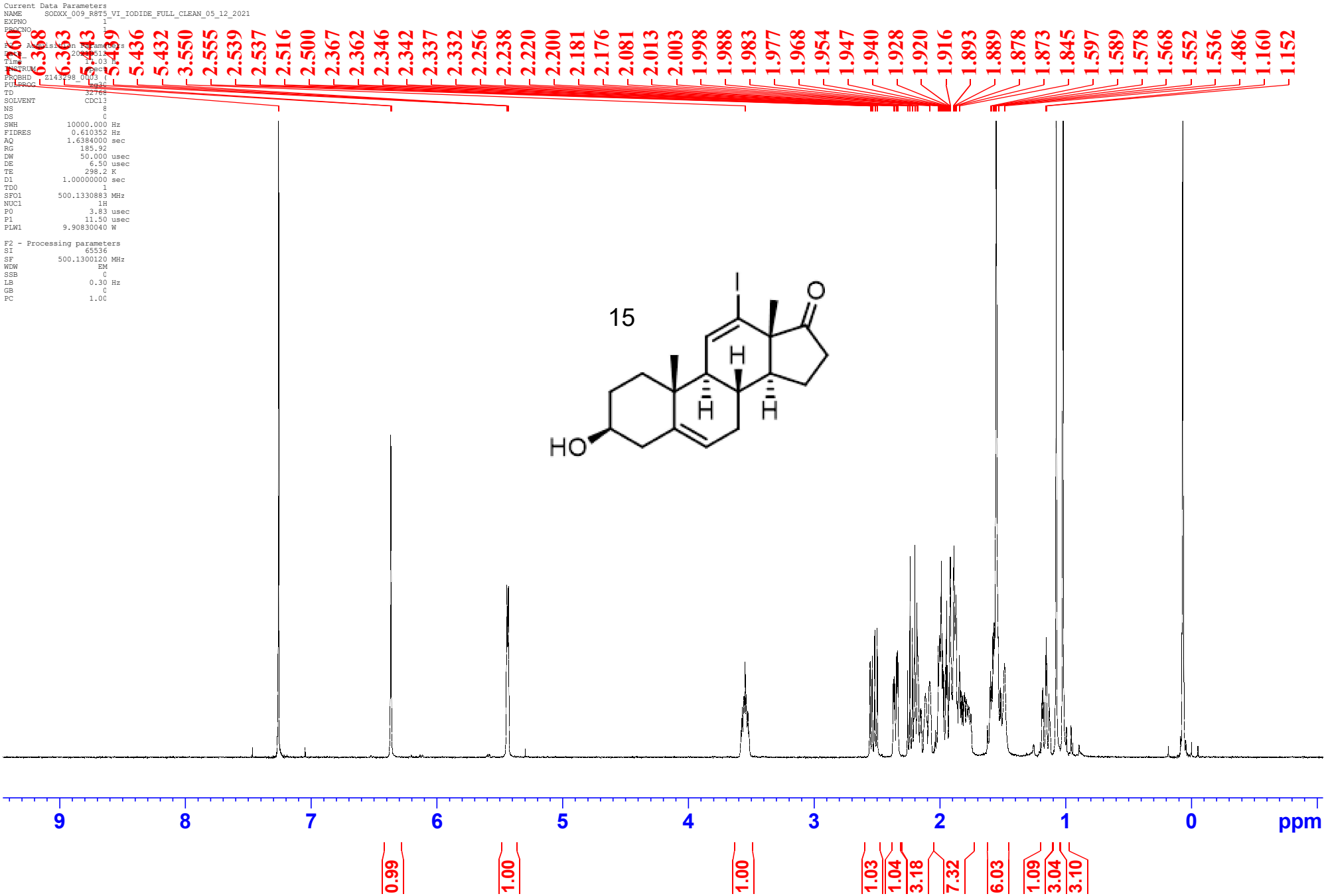
Temp. : 21.6℃ 21.6℃

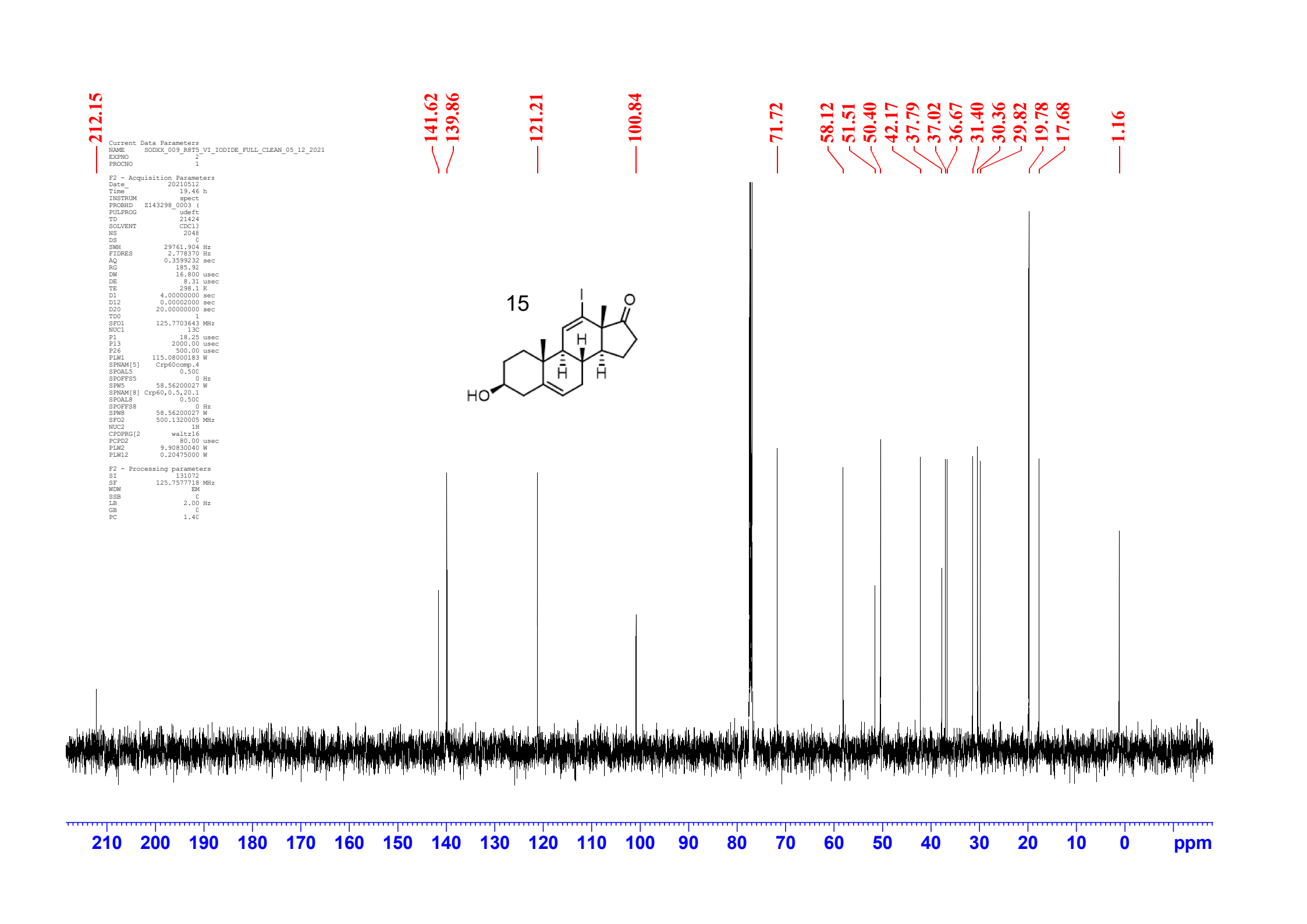
WLG	Cell			ATC
589.44	1.0000dm			20.0
Mode	Times		GLP	
Single	1		Off	

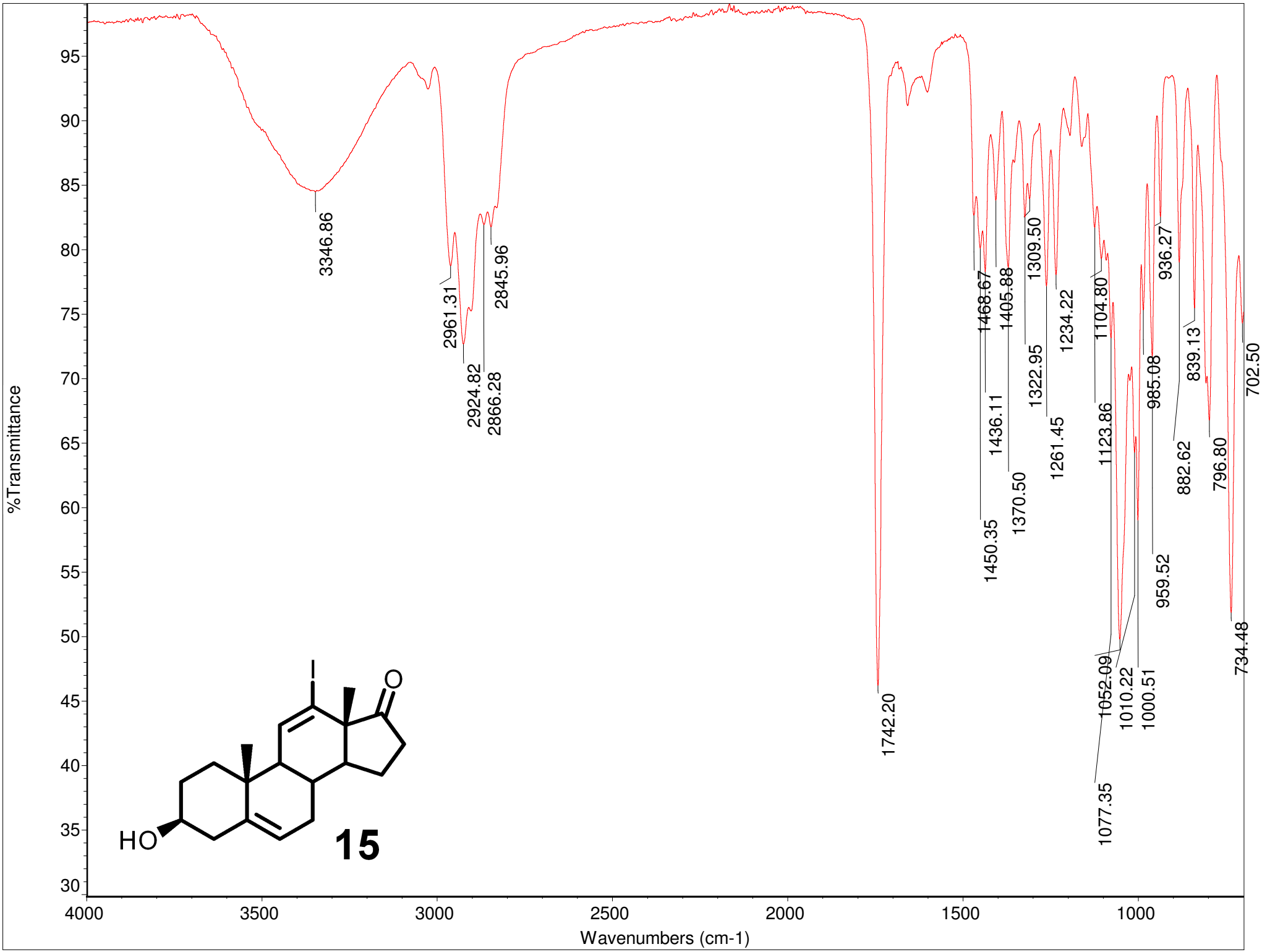
Save

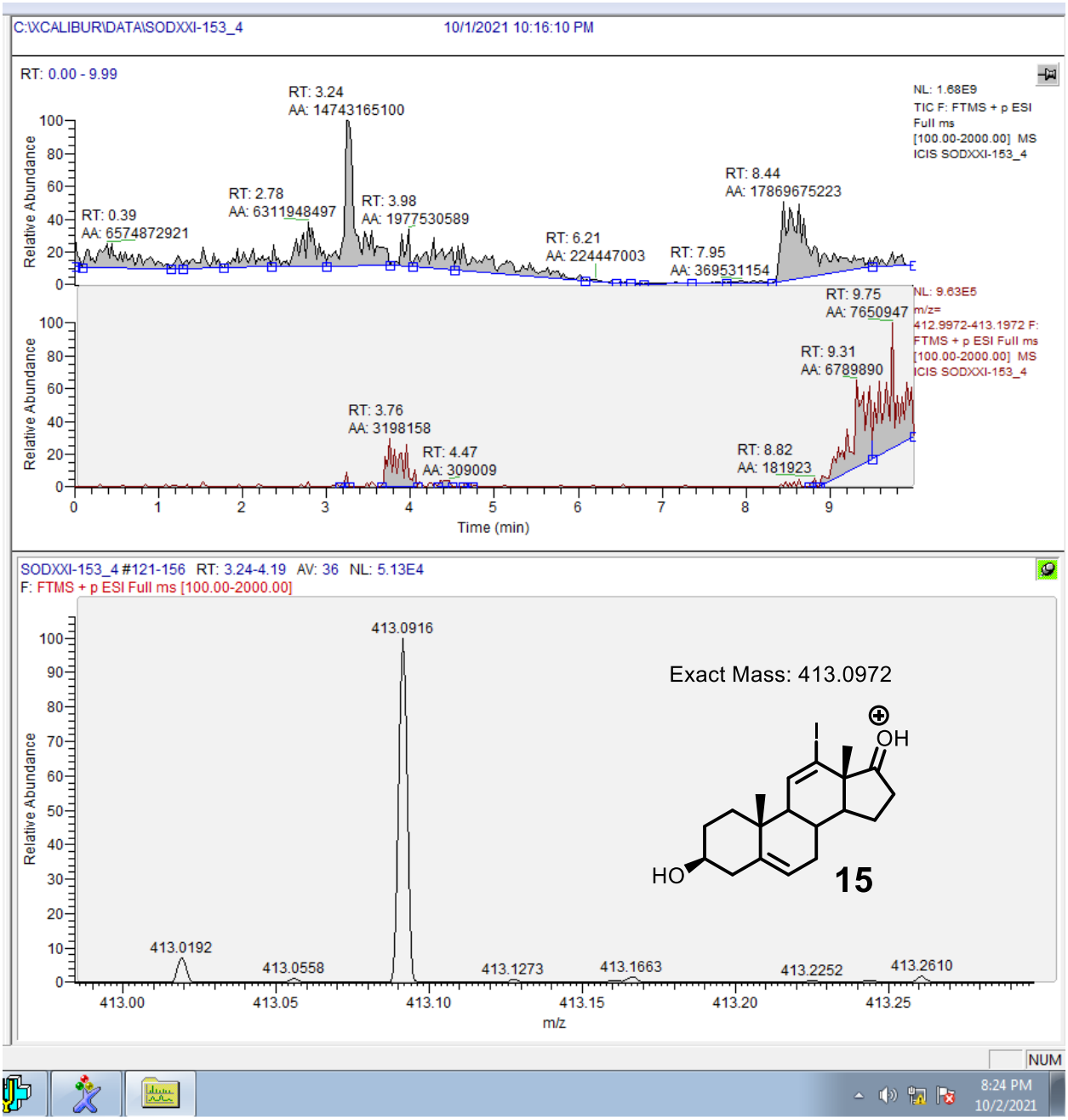


$\frac{4.1\,mg}{10\,ml}$ $CHCl_3$ -0.024


Current Data Parameters
NAME SODXX_009_RBT5_VI_IODIDE_FULL_CLEAN_05_12_2021
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210512
Time 14.03 h
INSTRUM spect
PROBHD Z143298_0003 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 185.92
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 3.83 usec
P1 11.50 usec
PLW1 9.90830040 W
F2 - Processing parameters
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
6.368
6.333
5.433
5.419
5.436
5.432
3.550
2.555
2.539
2.537
2.516
2.500
2.367
2.362
2.346
2.342
2.337
2.332
2.256
2.238
2.220
2.200
2.181
2.176
2.081
2.013
2.003
1.998
1.988
1.983
1.977
1.968
1.954
1.947
1.940
1.928
1.920
1.916
1.893
1.889
1.878
1.873
1.845
1.597
1.589
1.578
1.568
1.552
1.536
1.486
1.160
1.152
15
I
O
H
H
H
HO
9
8
7
6
5
4
3
2
1
0
ppm
0.99
1.00
1.00
1.03
1.04
3.18
7.32
6.03
1.09
3.04
3.10


212.15
141.62
139.86
121.21
100.84
71.72
58.12
51.51
50.40
42.17
37.79
37.02
36.67
31.40
30.36
29.82
19.78
17.68
1.16
Current Data Parameters
NAME SODXX_009_R8T5_VI_IODIDE_FULL_CLEAN_05_12_2021
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210512
Time 19.46 h
INSTRUM spect
PROBHD Z143298_0003 (
PULPROG udeft
TD 21424
SOLVENT CDCl3
NS 2048
DS 0
SWH 29761.904 Hz
FIDRES 2.778370 Hz
AQ 0.3599232 sec
RG 185.92
DW 16.800 usec
DE 8.31 usec
TE 298.1 K
D1 4.00000000 sec
D12 0.00002000 sec
D20 20.00000000 sec
TD0 1
SFO1 125.7703643 MHz
NUC1 13C
P1 18.25 usec
P13 2000.00 usec
P26 500.00 usec
PLW1 115.08000183 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 58.56200027 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 58.56200027 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 9.90830040 W
PLW12 0.20475000 W
F2 - Processing parameters
SI 131072
SF 125.7577718 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
15
I
O
H
H
H
HO
210
200
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)
4000
3500
3000
2500
2000
1500
1000
95
90
85
80
75
70
65
60
55
50
45
40
35
30
3346.86
2961.31
2924.82
2866.28
2845.96
1742.20
1468.67
1450.35
1436.11
1405.88
1370.50
1322.95
1309.50
1261.45
1234.22
1123.86
1104.80
1077.35
1052.09
1010.22
1000.51
985.08
959.52
936.27
882.62
839.13
796.80
734.48
702.50
HO
O
15


C:\XCALIBUR\DATA\SODXXI-153_4
10/1/2021 10:16:10 PM
RT: 0.00 - 9.99
NL: 1.68E9
TIC F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_4
RT: 0.39
AA: 6574872921
RT: 2.78
AA: 6311948497
RT: 3.24
AA: 14743165100
RT: 3.98
AA: 1977530589
RT: 6.21
AA: 224447003
RT: 7.95
AA: 369531154
RT: 8.44
AA: 17869675223
NL: 9.63E5
m/z= 412.9972-413.1972 F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_4
RT: 3.76
AA: 3198158
RT: 4.47
AA: 309009
RT: 8.82
AA: 181923
RT: 9.31
AA: 6789890
RT: 9.75
AA: 7650947
Relative Abundance
Time (min)
SODXXI-153_4 #121-156 RT: 3.24-4.19 AV: 36 NL: 5.13E4
F: FTMS + p ESI Full ms [100.00-2000.00]
413.0916
413.0192
413.0558
413.1273
413.1663
413.2252
413.2610
m/z
Exact Mass: 413.0972
OH
HO
15
NUM
8:24 PM
10/2/2021

LAXCO Optical Rotation

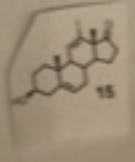
Original O.R. : -0.081°

O.R. °

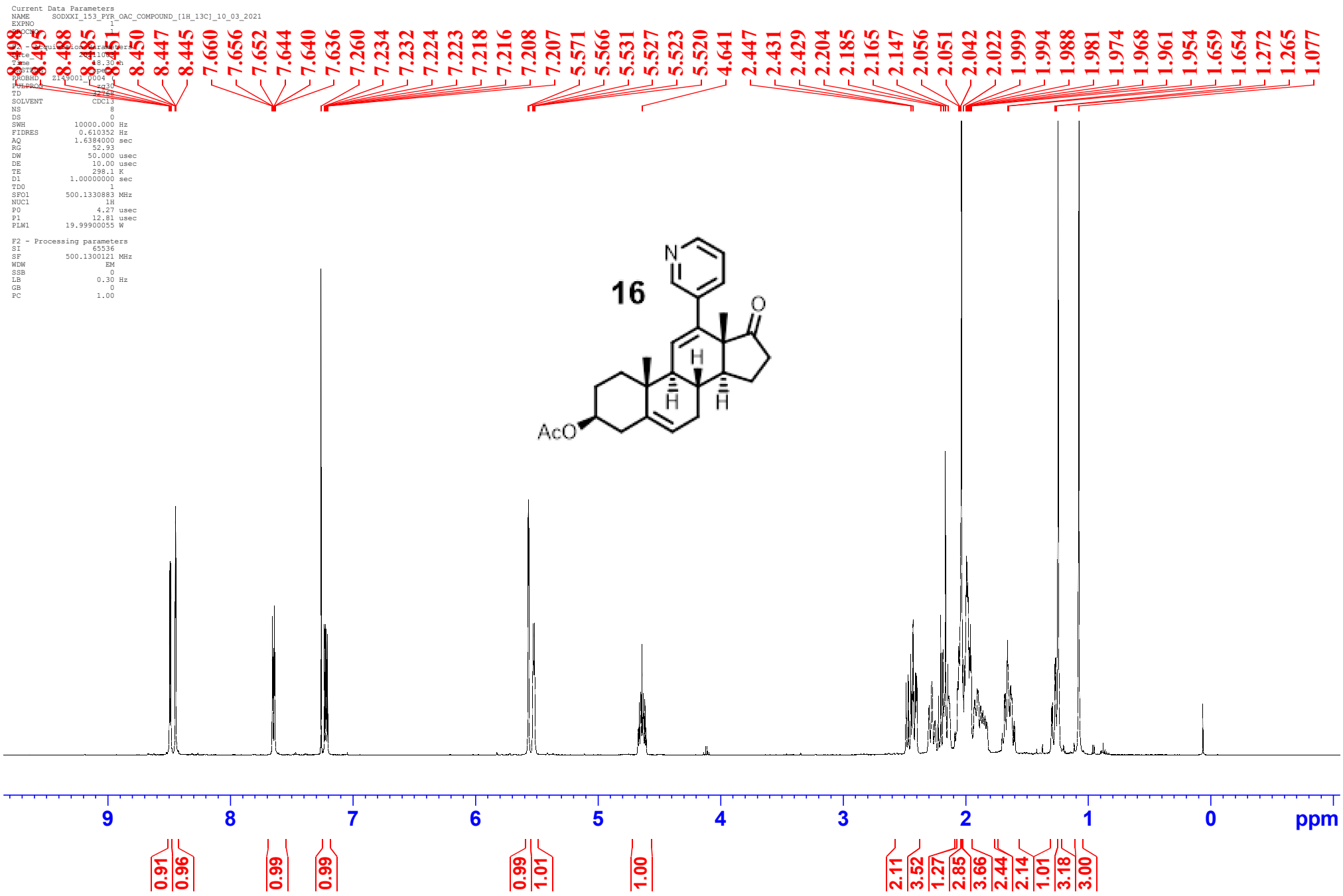
-0.081

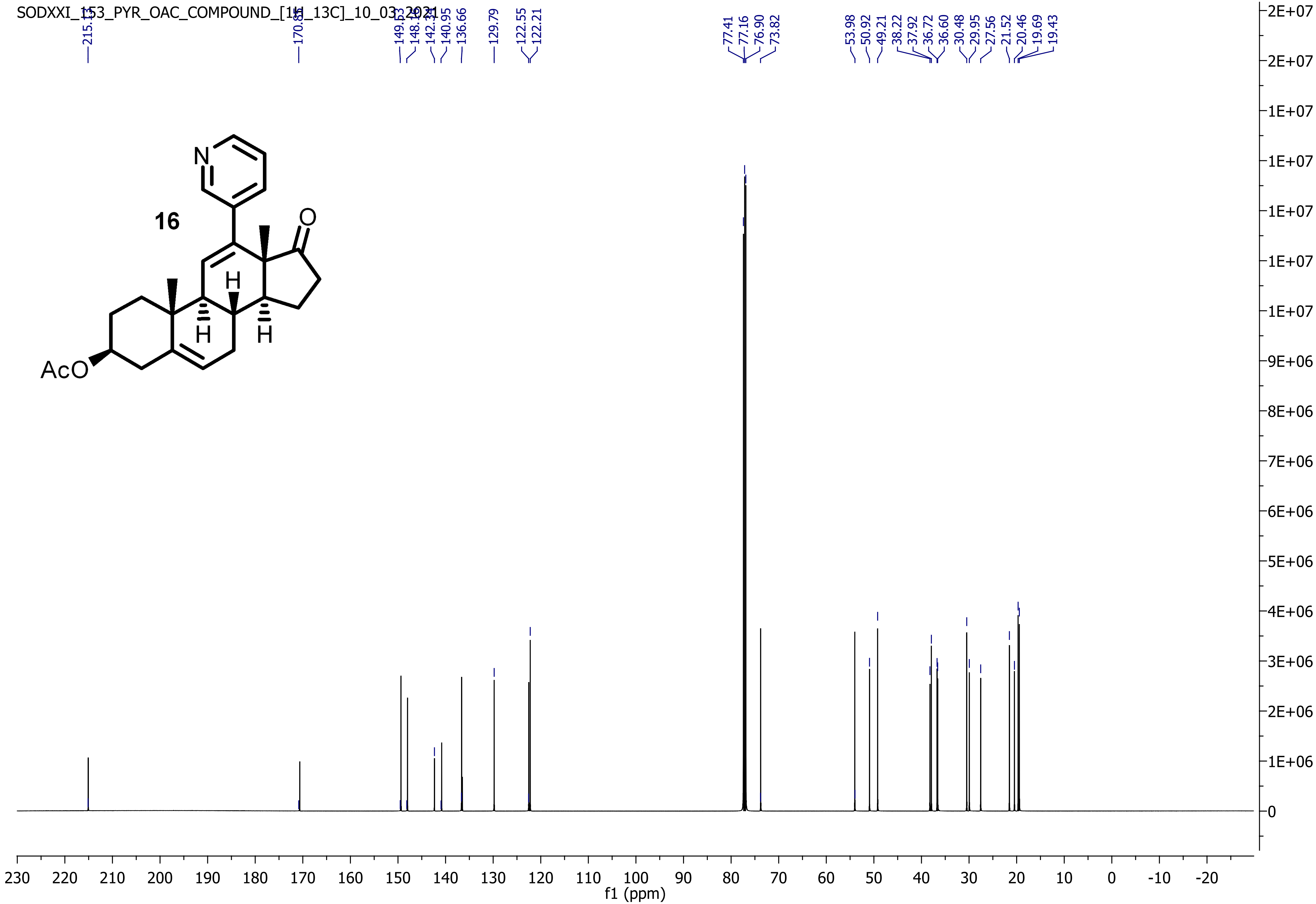
Temp. : 21.6℃ 21.5℃

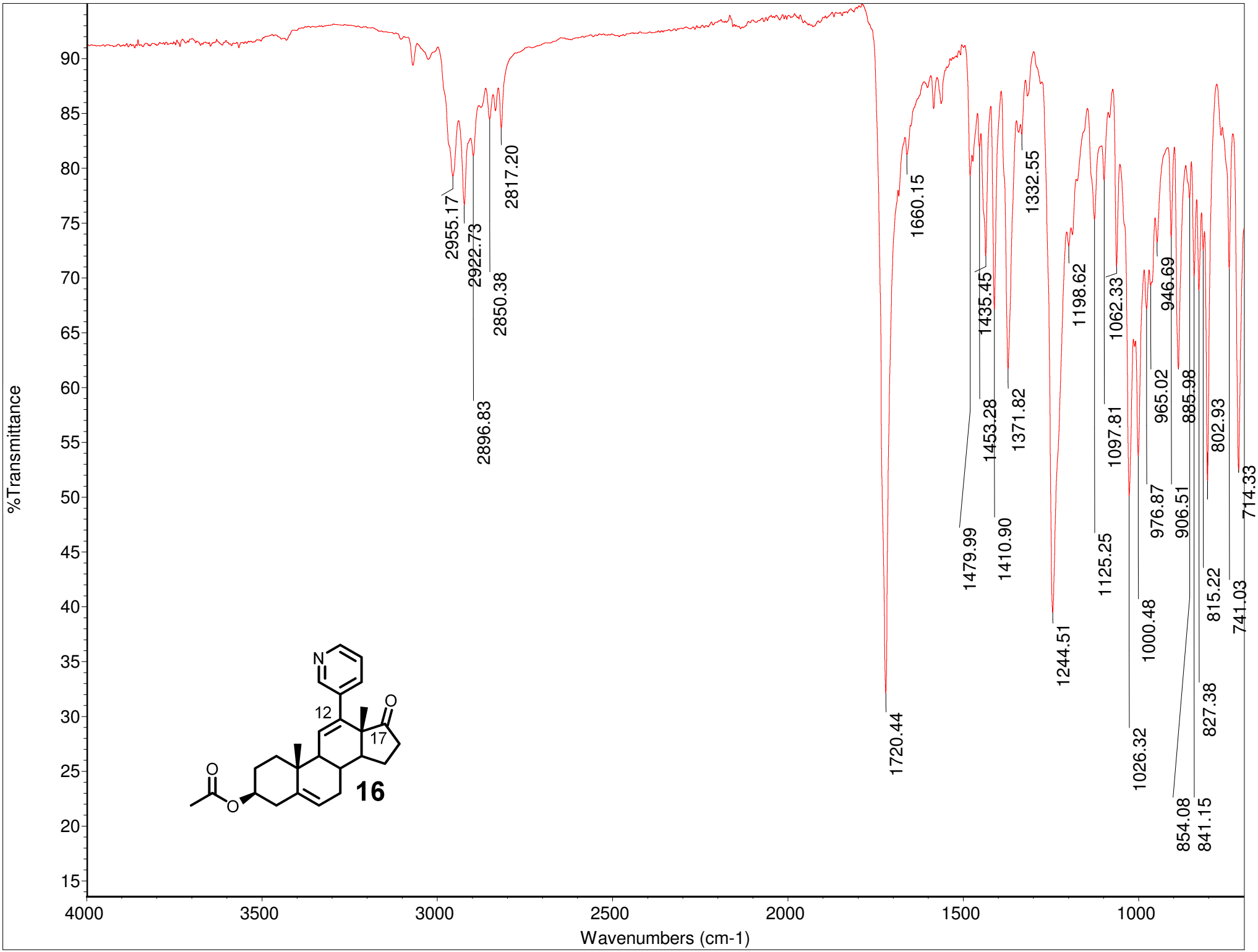
W.C	Cell			ATC
589.44	1.0000dm			20.0
Mode	Times		GLP	
Single	1		OFF	

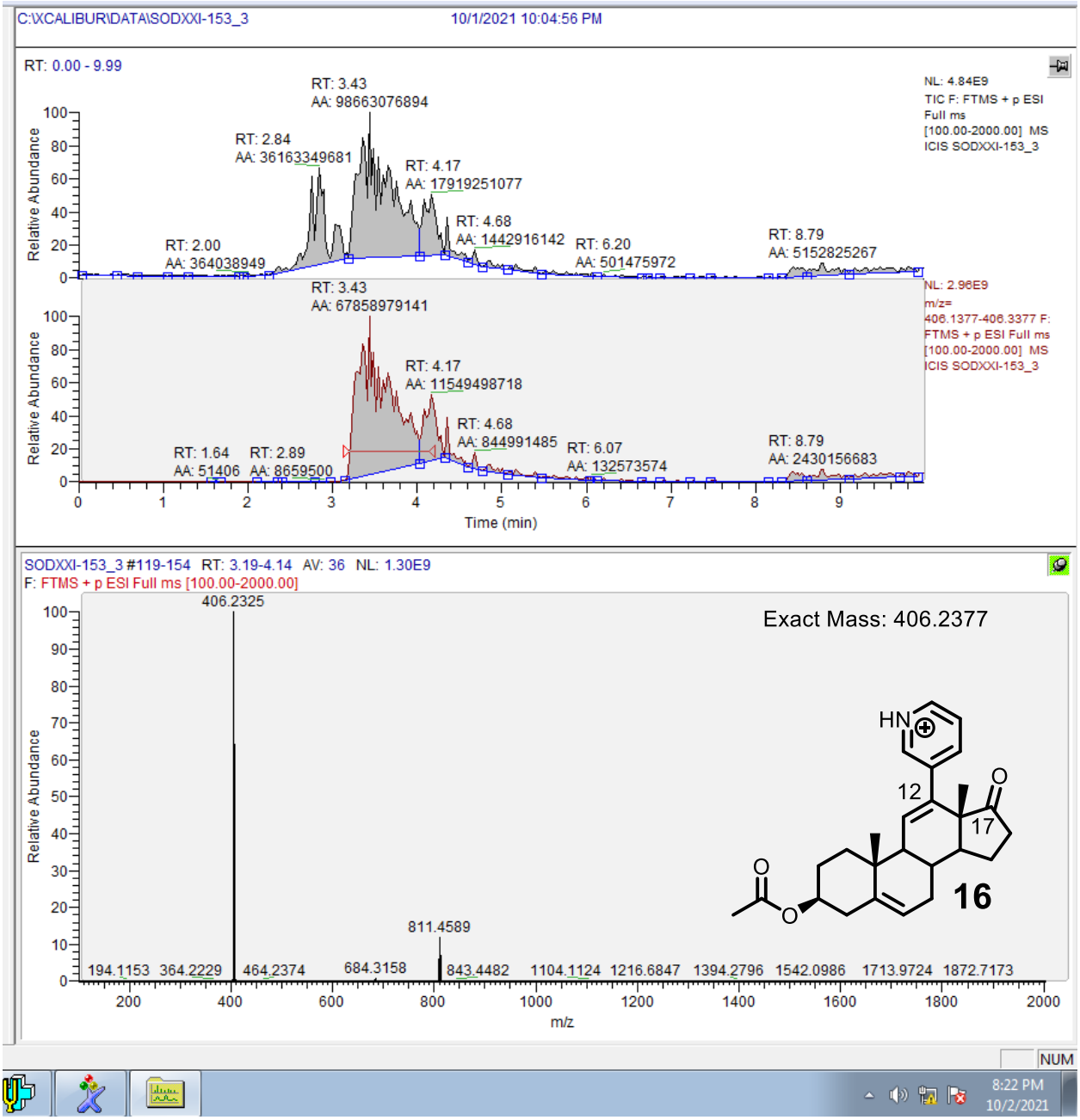


3.3mg / 10mL CHCl3 → -0.081


Current Data Parameters
NAME SODXXI_153_PYR_OAC_COMPOUND_[1H_13C]_10_03_2021
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211013
Time 18.30 h
INSTRUM spect
PROBHD ZIA9001_0004 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 52.93
DW 50.000 usec
DE 10.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 4.27 usec
P1 12.81 usec
PLW1 19.99900055 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.498
8.495
8.488
8.485
8.451
8.450
8.447
8.445
7.660
7.656
7.652
7.644
7.640
7.636
7.260
7.234
7.232
7.224
7.223
7.218
7.216
7.208
7.207
5.571
5.566
5.531
5.527
5.523
5.520
4.641
2.447
2.431
2.429
2.204
2.185
2.165
2.147
2.056
2.051
2.042
2.022
1.999
1.994
1.988
1.981
1.974
1.968
1.961
1.954
1.659
1.654
1.272
1.265
1.077
16
N
O
H
H
H
AcO
9
8
7
6
5
4
3
2
1
0
ppm
0.91
0.96
0.99
0.99
0.99
1.01
1.00
2.11
3.52
1.27
2.85
3.66
2.44
2.14
1.01
3.18
3.00


SODXXI_153_PYR_OAC_COMPOUND_[1H_13C]_10_03_2021
215.13
170.85
149.53
148.16
142.31
140.95
136.66
129.79
122.55
122.21
77.41
77.16
76.90
73.82
53.98
50.92
49.21
38.22
37.92
36.72
36.60
30.48
29.95
27.56
21.52
20.46
19.69
19.43
16
N
O
H
H
H
AcO
2E+07
2E+07
1E+07
1E+07
1E+07
1E+07
1E+07
9E+06
8E+06
7E+06
6E+06
5E+06
4E+06
3E+06
2E+06
1E+06
0
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)


%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
2955.17
2922.73
2896.83
2850.38
2817.20
1720.44
1660.15
1479.99
1435.45
1453.28
1410.90
1371.82
1332.55
1244.51
1198.62
1125.25
1097.81
1062.33
1026.32
1000.48
976.87
965.02
946.69
906.51
885.98
854.08
841.15
827.38
815.22
802.93
741.03
714.33
N
O
O
12
17
O
16


C:\XCALIBUR\DATA\SODXXI-153_3
10/1/2021 10:04:56 PM
RT: 0.00 - 9.99
NL: 4.84E9
TIC F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_3
RT: 3.43
AA: 98663076894
RT: 2.84
AA: 36163349681
RT: 4.17
AA: 17919251077
RT: 4.68
AA: 1442916142
RT: 2.00
AA: 364038949
RT: 6.20
AA: 501475972
RT: 8.79
AA: 5152825267
Relative Abundance
NL: 2.96E9
m/z= 406.1377-406.3377 F: FTMS + p ESI Full ms [100.00-2000.00] MS ICIS SODXXI-153_3
RT: 3.43
AA: 67858979141
RT: 4.17
AA: 11549498718
RT: 4.68
AA: 844991485
RT: 1.64
AA: 51406
RT: 2.89
AA: 8659500
RT: 6.07
AA: 132573574
RT: 8.79
AA: 2430156683
Time (min)
SODXXI-153_3 #119-154 RT: 3.19-4.14 AV: 36 NL: 1.30E9
F: FTMS + p ESI Full ms [100.00-2000.00]
406.2325
Exact Mass: 406.2377
811.4589
194.1153 364.2229 464.2374 684.3158 843.4482 1104.1124 1216.6847 1394.2796 1542.0986 1713.9724 1872.7173
m/z
HN
O
12
17
O
O
16
NUM
8:22 PM
10/2/2021

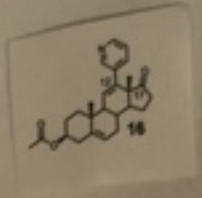
Original O.R.: -0.094° O.R. °

-0.094

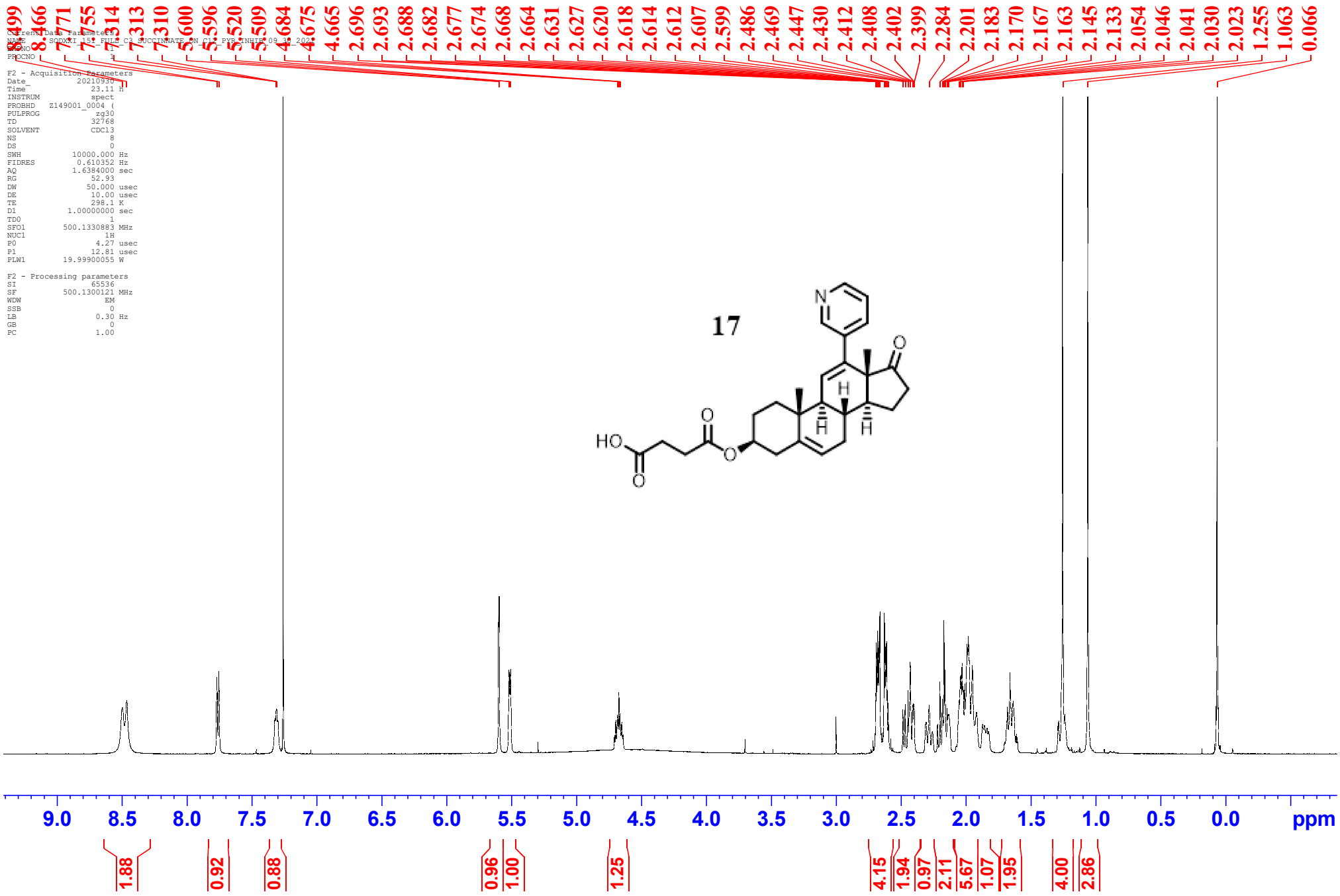
Temp.: 21.6℃ 21.5℃

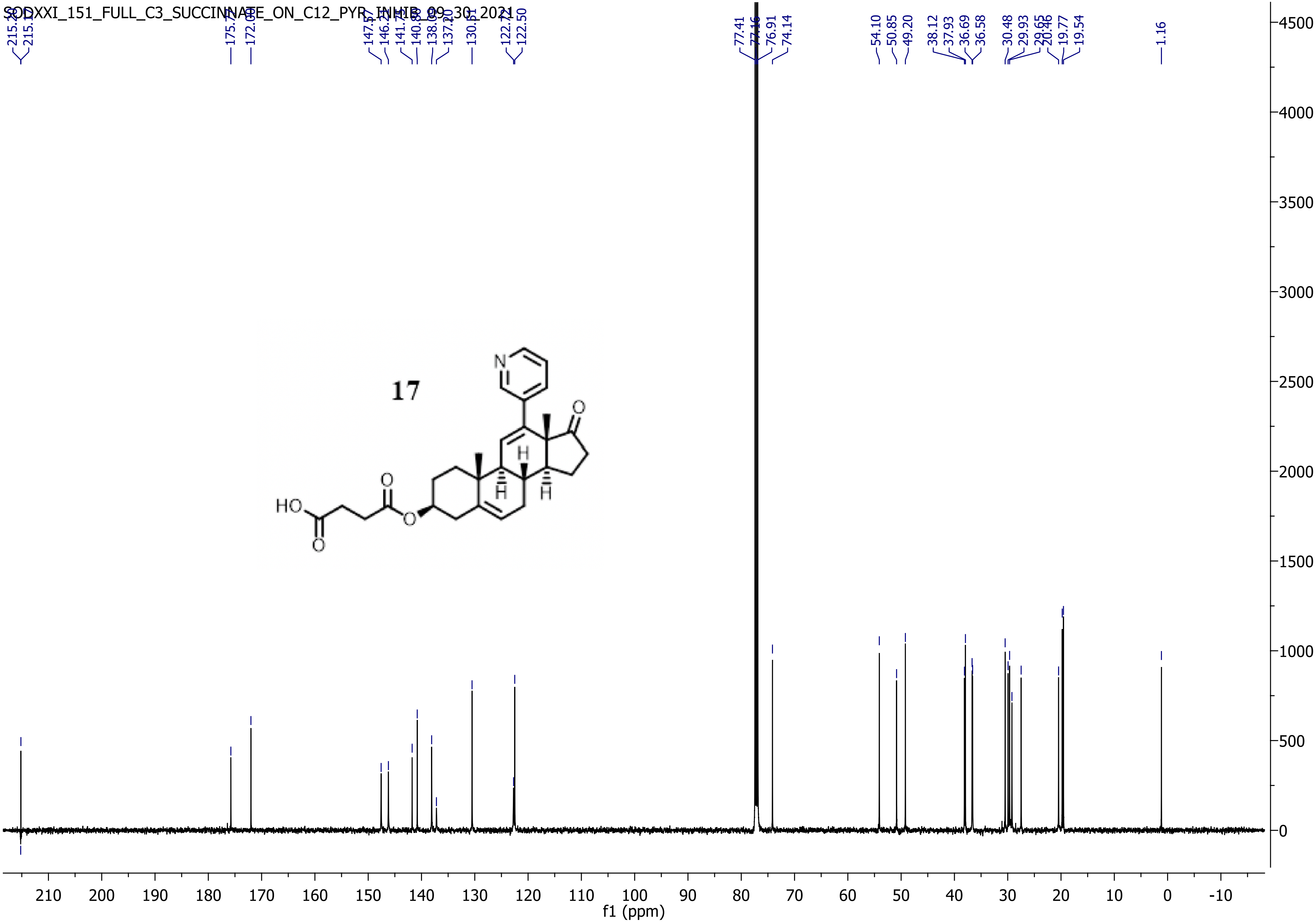
WLG	Cell			ATC
589.44	1.0000dm			20.0
Mode	Times		GLP	
single	1		Off	

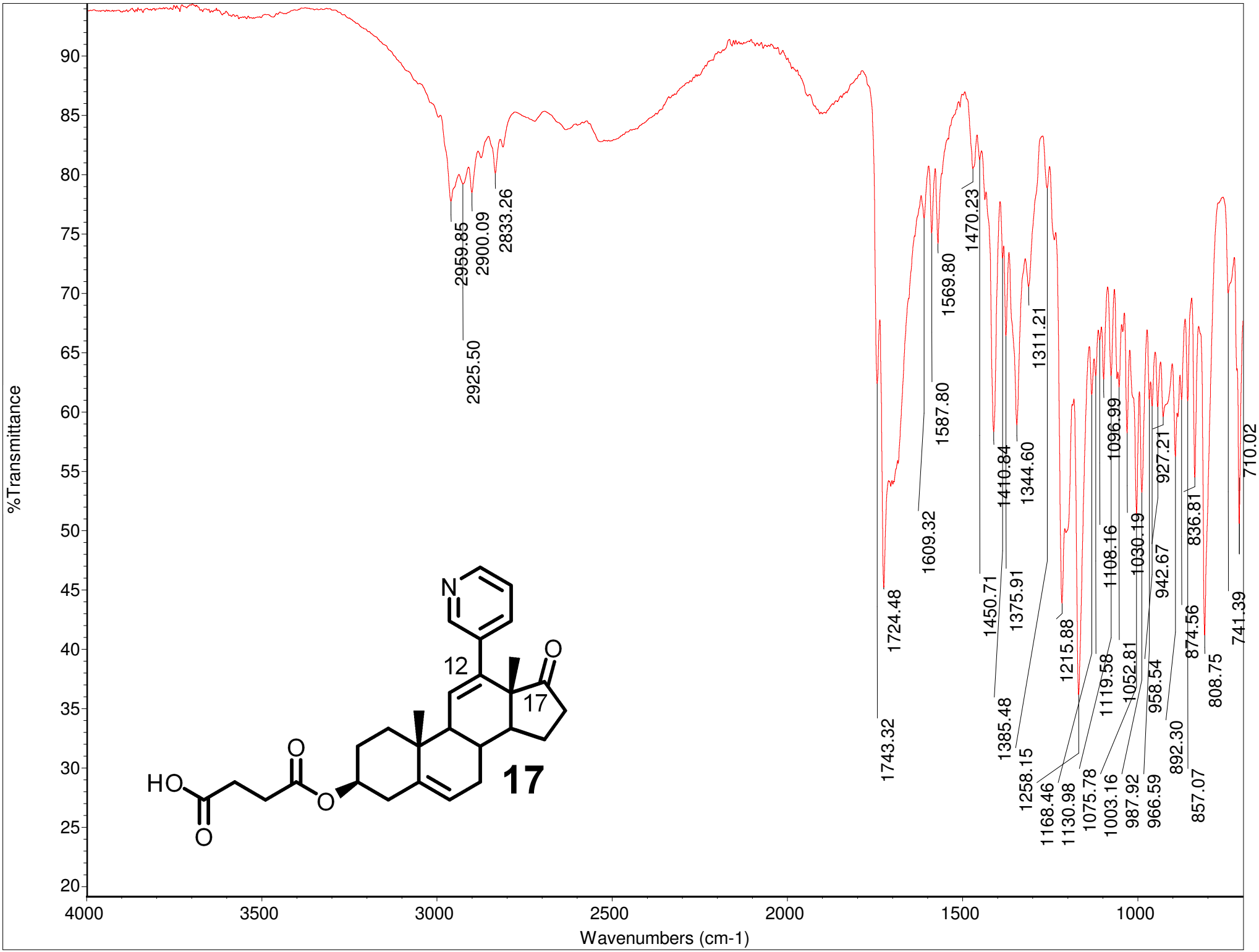
menu

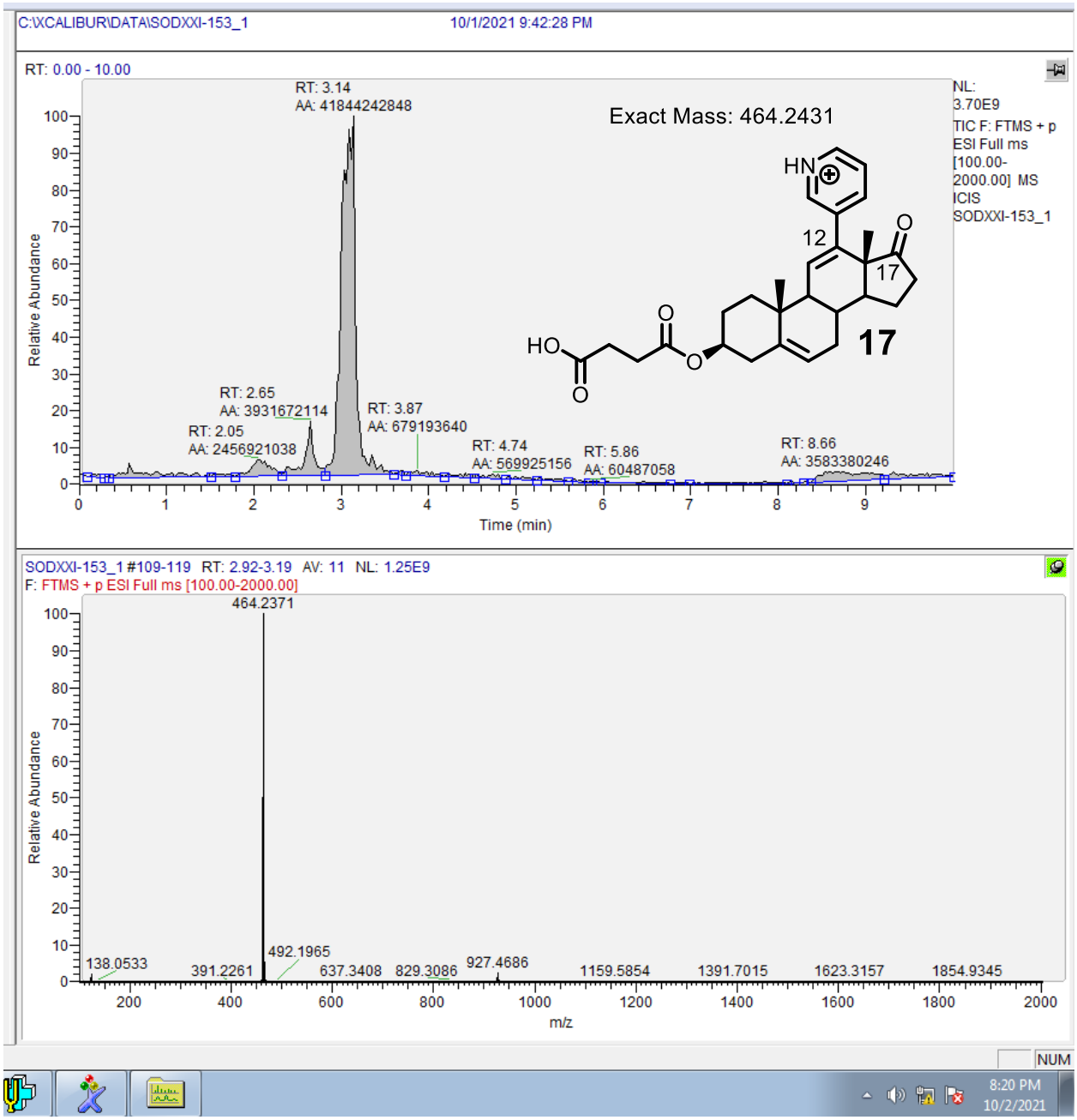


18.5mg / 10ml -0.094


8.499
8.466
7.771
7.755
7.314
7.313
7.310
6.600
5.596
5.520
5.509
4.684
4.675
4.665
2.696
2.693
2.688
2.682
2.677
2.674
2.668
2.664
2.631
2.627
2.620
2.618
2.614
2.612
2.607
2.599
2.486
2.469
2.447
2.430
2.412
2.408
2.402
2.399
2.284
2.201
2.183
2.170
2.167
2.163
2.145
2.133
2.054
2.046
2.041
2.030
2.023
1.255
1.063
0.066
Current Data Parameters
NAME SQDXRI_15_FULL_C3_SUCCINATE_ON_C3_PYR_INHIB_09_10_202
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210930
Time 23.11 H
INSTRUM spect
PROBHD Z149001_0004 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.610352 Hz
AQ 1.6384000 sec
RG 52.93
DW 50.000 usec
DE 10.00 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330883 MHz
NUC1 1H
P0 4.27 usec
P1 12.81 usec
PLW1 19.99900055 W
F2 - Processing parameters
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
17
N
O
H
H
H
HO
O
O
O
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.88
0.92
0.88
0.96
1.00
1.25
4.15
1.94
0.97
2.11
5.67
1.07
1.95
4.00
2.86


SODXXI_151_FULL_C3_SUCCINNATE_ON_C12_PYR_INHIB_09_30_2021
122.50
77.41
77.16
76.91
74.14
54.10
50.85
49.20
38.12
37.93
36.69
36.58
30.48
29.93
29.65
20.46
19.77
19.54
1.16
17
N
O
HO
H
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
4500 4000 3500 3000 2500 2000 1500 1000 500 0


%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
2959.85
2925.50
2900.09
2833.26
1743.32
1724.48
1609.32
1587.80
1569.80
1470.23
1450.71
1410.84
1385.48
1375.91
1344.60
1311.21
1258.15
1215.88
1168.46
1130.98
1119.58
1108.16
1096.99
1075.78
1052.81
1030.19
1003.16
987.92
966.59
958.54
942.67
927.21
892.30
874.56
857.07
836.81
808.75
741.39
710.02
HO
O
O
O
N
12
17
O
17


C:\XCALIBUR\DATA\SODXXI-153_1
10/1/2021 9:42:28 PM
RT: 0.00 - 10.00
RT: 3.14
AA: 41844242848
Exact Mass: 464.2431
NL:
3.70E9
TIC F: FTMS + p
ESI Full ms
[100.00-
2000.00] MS
ICIS
SODXXI-153_1
RT: 2.65
AA: 3931672114
RT: 2.05
AA: 2456921038
RT: 3.87
AA: 679193640
RT: 4.74
AA: 569925156
RT: 5.86
AA: 60487058
RT: 8.66
AA: 3583380246
Relative Abundance
Time (min)
HN
12
17
O
HO
17
SODXXI-153_1 #109-119 RT: 2.92-3.19 AV: 11 NL: 1.25E9
F: FTMS + p ESI Full ms [100.00-2000.00]
464.2371
492.1965
138.0533
391.2261
637.3408
829.3086
927.4686
1159.5854
1391.7015
1623.3157
1854.9345
m/z
NUM
8:20 PM
10/2/2021

LAXCO Optical Rotation

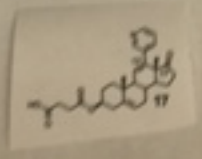
Original O.R. : 0.105°

O.R. °

0.105

Temp. : 21.5℃ 21.5℃

WLG	Cell			ATC
589.44	1 100mm			20.0
Mode	Times		GLP	
single	1		OFF	



$$\frac{14.0\,mg}{10\,mg}$$

0.105

Table S1: Crystallographic data and structure refinement for **compound 16**

Identification code	Compound 16
Empirical formula	$C_{26}H_{31}NO_3$
Formula weight	405.52
Crystal system	Monoclinic
Space group	*P2₁*
a (Å)	8.3667(2)
b (Å)	12.9356(2)
c (Å)	10.5610(2)
α (°)	90
β (°)	113.026(2)
γ (°)	90
Volume (Å^3)	1051.93(5)
Z	2
ρ (calc.)	1.280
λ	1.54184
Temp. (K)	100.0(1)
F(000)	436
μ (mm^{-1})	0.655
T_{min}, T_{max}	0.378, 1.000
$2\theta_{range}$ (°)	9.10 to 152.8
Reflections collected	6891
Independent reflections	3400 [R(int) = 0.0267]
Completeness	97.3%
Data / restraints / parameters	3400 / 1 / 275
Observed data [I > 2σ(I)]	3293
wR(F^2 all data)	0.0800
R(*F* obsd data)	0.0306
Goodness-of-fit on F^2	1.06
largest diff. peak and hole (e Å^{-3})	0.19 / -0.13

$$wR_2 = \{ \Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2] \}^{1/2}$$

$$R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$$

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) hpd326

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: hpd326

```
Bond precision:   C-C = 0.0032 A          Wavelength=1.54184

Cell:             a=8.36666(17)     b=12.93557(19)      c=10.5610(2)
                  alpha=90          beta=113.026(2)     gamma=90
Temperature:      100 K

                 Calculated                Reported
Volume           1051.93(4)                1051.93(4)
Space group      P 21                      P 1 21 1
Hall group       P 2yb                     P 2yb
Moiety formula   C26 H31 N O3              C26 H31 N O3
Sum formula      C26 H31 N O3              C26 H31 N O3
Mr               405.52                    405.52
Dx,g cm-3        1.280                     1.280
Z                2                         2
Mu (mm-1)        0.655                     0.655
F000             436.0                     436.0
F000'            437.24
h,k,lmax         10,16,13                  10,16,12
Nref             4430[ 2317]               3400
Tmin,Tmax        0.885,0.940               0.378,1.000
Tmin'            0.877

Correction method= # Reported T Limits: Tmin=0.378 Tmax=1.000
AbsCorr = GAUSSIAN

Data completeness= 1.47/0.77        Theta(max)= 76.376

R(reflections)= 0.0306( 3293)                wR2(reflections)=
                                             0.0800( 3400)
S = 1.062                 Npar= 275
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT029_ALERT_3_C _diffrn_measured_fraction_theta_full value Low . 0.973 Why?
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 53 Report
PLAT915_ALERT_3_C No Flack x Check Done: Low Friedel Pair Coverage 61 %

Alert level G

PLAT791_ALERT_4_G Model has Chirality at C3 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C8 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C9 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C10 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C13 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C14 (Sohnke SpGr) S Verify
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 136 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 3.3 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 11 Info

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
9 **ALERT level G** = General information/check it is not something unexpected

0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
7 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 13/07/2021; check.def file version of 13/07/2021

Datablock hpd326 - ellipsoid plot