

Supporting Information

Formal Synthesis of (±)-Pentalenolactone A Methyl Ester.

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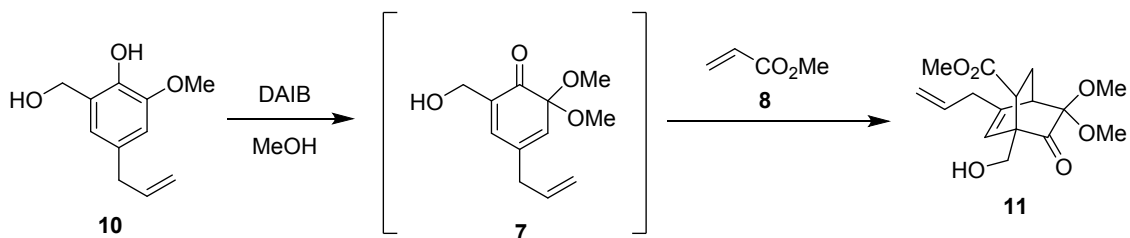
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Experimental Data

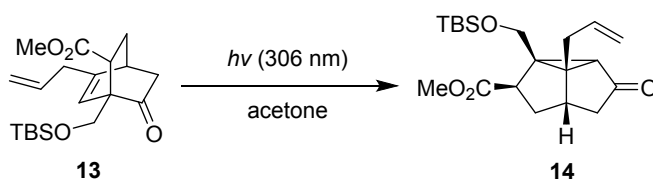
Optimization of Reactions

Table S1. Studies for Diel-Alder Reaction of MOB 7 with methyl acrylate 8



Entry	Conditions	Yield of 11
1	methyl acrylate (5 equiv.), MeOH/CH ₂ Cl ₂	63%
2	methyl acrylate (20 equiv.), MeOH/CH ₂ Cl ₂	70%
3	methyl acrylate (20 equiv.), MeOH	80%
4	methyl acrylate (20 equiv.), MeOH, BHT (10 wt%)	88%

Table S2. Studies for ODPM Rearrangement of 13

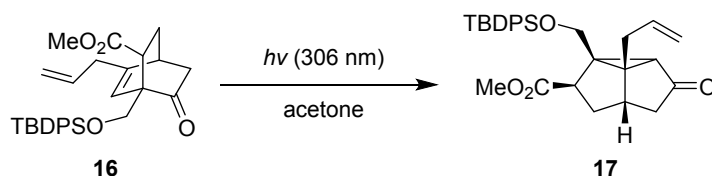


Entry	Lamps ^a	Material ^b	Concentration	Time	Yield ^c
1	16	quartz	80 mg/80 mL	240 min	59%
2	16	quartz	80 mg/80 mL	180 min	50%
3	16	quartz	80 mg/80 mL	60 min	53%
4	16	quartz	160 mg/160 mL	300 min	25%
5	16	quartz	40 mg/80 mL	60 min	35%
6	16	quartz	97 mg/10 mL	315 min	21%
7 ^d	16	quartz	97 mg/10 mL	315 min	S.M. recovered

8	8	quartz	80 mg/80 mL	60 min	48% (57% brsm)
9	8	Pyrex	80 mg/80 mL	60 min	35% (51% brsm)
10	4	quartz	80 mg/80 mL	150 min	35% (53% brsm)
11 ^e	8	quartz	80 mg/80 mL	75 min	43% (62% brsm)
12	8	quartz	80 mg/80 mL	50 min	39% (73% brsm)
13 ^f	8	quartz	80 mg/80 mL	75 min	30% (42% brsm)

^aThe number of lamps in the photoreactor. ^bThe material of the photoreaction tube. ^cIsolated yield. ^dThe addition of 10 equiv. acetophenone as a sensitizer. ^eTwo tubes a time during the irradiation. ^fThree tubes a time during the irradiation.

Table S3. Studies for ODPM Rearrangement of 16



Entry	Lamps ^a	Concentration	Pump speed	Yield ^b
1	16	0.002 M	1 mL/min	41%
2	16	0.002 M	2 mL/min	43%
3	16	0.002 M	4 mL/min	52%
4	16	0.002 M	6 mL/min	45%
5	16	0.001 M	1 mL/min	19%
6 ^c	16	0.002 M	4 mL/min	44%
7	16	0.004 M	4 mL/min	47%
8 ^d	8	0.004 M	4 mL/min	26%
9 ^e	8	0.004 M	4 mL/min	41%
10 ^f	16	0.004 M	4 mL/min	54%
11	16	0.004 M	5 mL/min	51%

^aThe number of lamps in the photoreactor. ^bIsolated yield. ^cGram-scale. ^dNew lamps was installed on the photoreactor. ^eOld lamps was used for the photoreaction. ^fThe photoreaction was carried out after the pump and tubing were washed with solvents.

Continuous-Flow Conditions:

Pump: HPLC pump (JASCO PU-2080 PLUS)

Tubing: PTFE (ID: 1.34 mm, OD: 1.64 mm, 54 mL)

Figure S1. Continuous-Flow Photoreaction

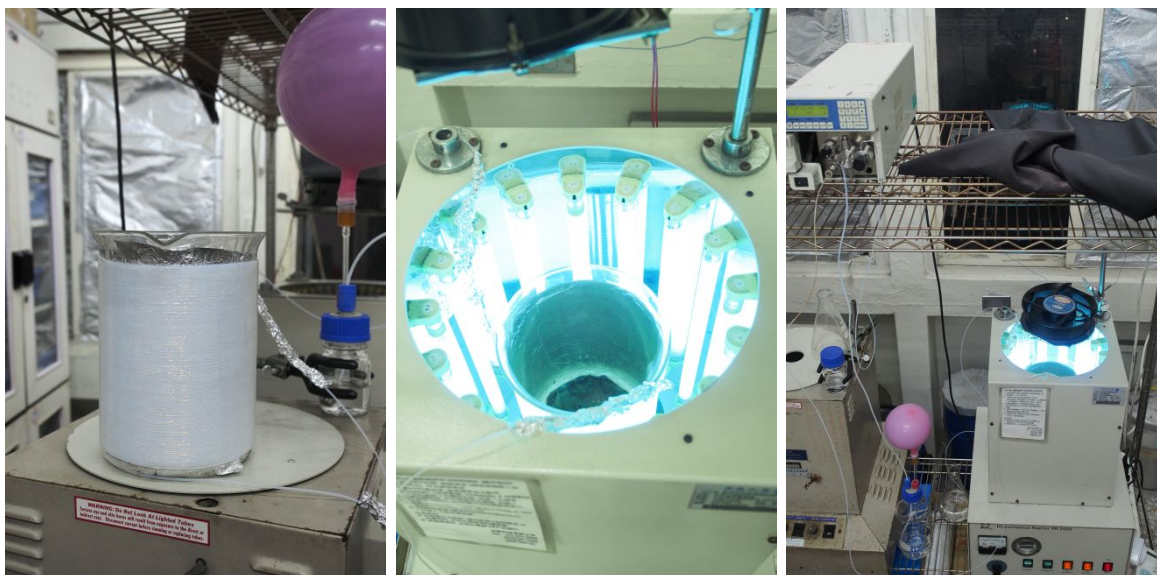
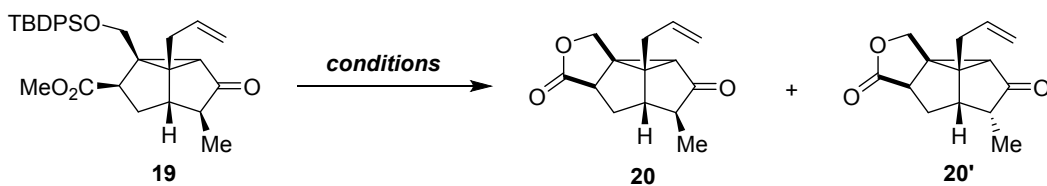
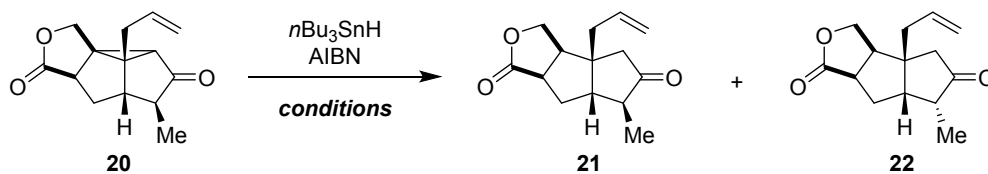


Table S4. Studies for Desilylation/Lactonization of 19



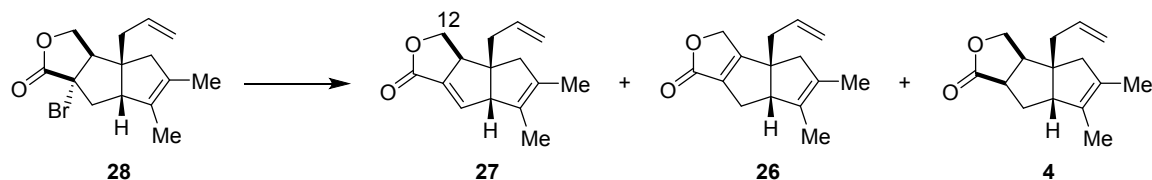
Entry	Conditions	Yield ^a (Ratio 20 : 20') ^b
1	TBAF (1 M/THF), rt, 15 h	90% (5 : 1)
2	TBAF (1 M/THF), rt, 25 h	76% (1 : 1)
3	TFA, CH ₂ Cl ₂ , rt, 48 h	quant. (1 : 0)
4	TFA, DCE, reflux, 5 h	79% (5 : 1)

^aIsolated yield. ^bThe ratio of **20** and **20'** were determined by ¹H NMR from the isolated products.

Table S5. Studies for $n\text{Bu}_3\text{SnH}$ Reduction of **20**

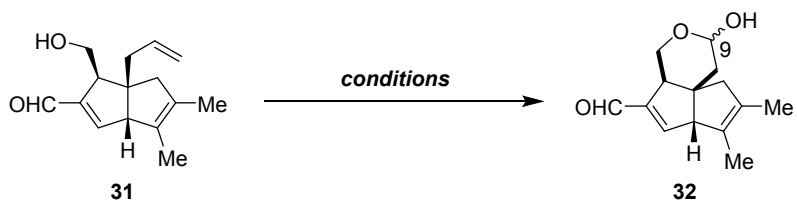
Entry	Conditions	Yield ^a
1	Benzene (0.1 M), reflux, 8 h	21 , 49% (70% brsm)+ 22 , 8%
2	Benzene (0.1 M), reflux, 30 h	21 , 36% (67% brsm)+ 22 , 24%
3	Benzene (0.1 M), 160 °C(μw), 30 min	21 , 65% (71% brsm)
4 ^b	Benzene (0.1 M), 160 °C(μw), 1 h	21 , 60% (70% brsm)
5	toluene (0.2 M), 160 °C(μw), 30 min	21 , 74% (87% brsm)
6	toluene (0.2 M), 140 °C(μw), 30 min	21 , 59% (69% brsm)
7	toluene (0.4 M), 160 °C(μw), 30 min	21 , 78% (88% brsm)

^aIsolated yield. ^b(TMS)₃SiH was used as the hydride source with AIBN as the radical initiator.

Table S6. Studies for dehydrobromination of 28

Entry	Conditions	Yield ^a (27/26/4)
1	DBU(2 equiv.), 0.2 M, reflux, 22 h	38%(54% brsm)/10%/9%
2 ^b	DBU(2 equiv.), 0.2 M, reflux, 22 h	13%(28% brsm)/3%/19%
3	DBU(2 equiv.), 0.2 M, reflux, 14 h	23%(43% brsm)/5%/trace
4	DBU(2 equiv.), 0.2 M, reflux, 36 h	29%(38% brsm)/10%/trace
5	DBU(4 equiv.), 0.2 M, reflux, 22 h	27%(30% brsm)/14%/10%
6	DBU(2 equiv.), 0.3 M, reflux, 22 h	38%(50% brsm)/16%/trace
7	DBU(2 equiv.), 0.5 M, reflux, 22 h	0%/10%/trace
8	DBU(2 equiv.), 0.5 M, reflux, 4 h	36%(54% brsm)/11%/trace
9	DBU(2 equiv.), 0.5 M, reflux, 2 h	16%(54% brsm)/7%/trace
10	DBU(2 equiv.), 0.5 M, reflux, 1 h	18%(66% brsm)/8%/trace
11	DBU(2 equiv.), 0.5 M, 50 °C, 22 h	7%(19% brsm)/0%/0%
12	DBU(2 equiv.), 0.5 M, 70 °C, 22 h	17%(77% brsm)/0%/0%
13	DBU(2 equiv.), 0.5 M, 90 °C, 22 h	21%(28% brsm)/13%/0%
14	DBU(4 equiv.), 0.5 M, reflux, 1 h	31%(72% brsm)/trace/trace
15	DBU(6 equiv.), 0.5 M, reflux, 1 h	32%(55% brsm)/10%/trace

^aIsolated yield. ^bBHT as the additive.

Table S7. Studies for Oxidative Cleavage of 31

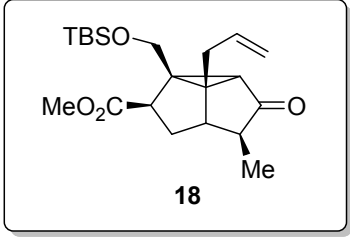
Entry	Conditions	Yield ^a of 32
1	OsO ₄ (0.02 equiv.), NaIO ₄ (4 equiv.), 2,6-lutidine (2 equiv.), dioxane/H ₂ O (3:1), rt, 20 h	20% yield (57% brsm)
2	K ₂ OsO ₄ • 2H ₂ O (0.02 equiv.), NaIO ₄ (4 equiv.), 2,6-lutidine (2 equiv.), THF/H ₂ O (1:1), rt, 20 h	26% yield (86% brsm)
3	K ₂ OsO ₄ • 2H ₂ O (0.22 equiv.), NaIO ₄ (3 equiv.), 2,6-lutidine (4 equiv.), THF/H ₂ O (1:1), rt, 8 h	13% yield (14% brsm)

^aIsolated yield.

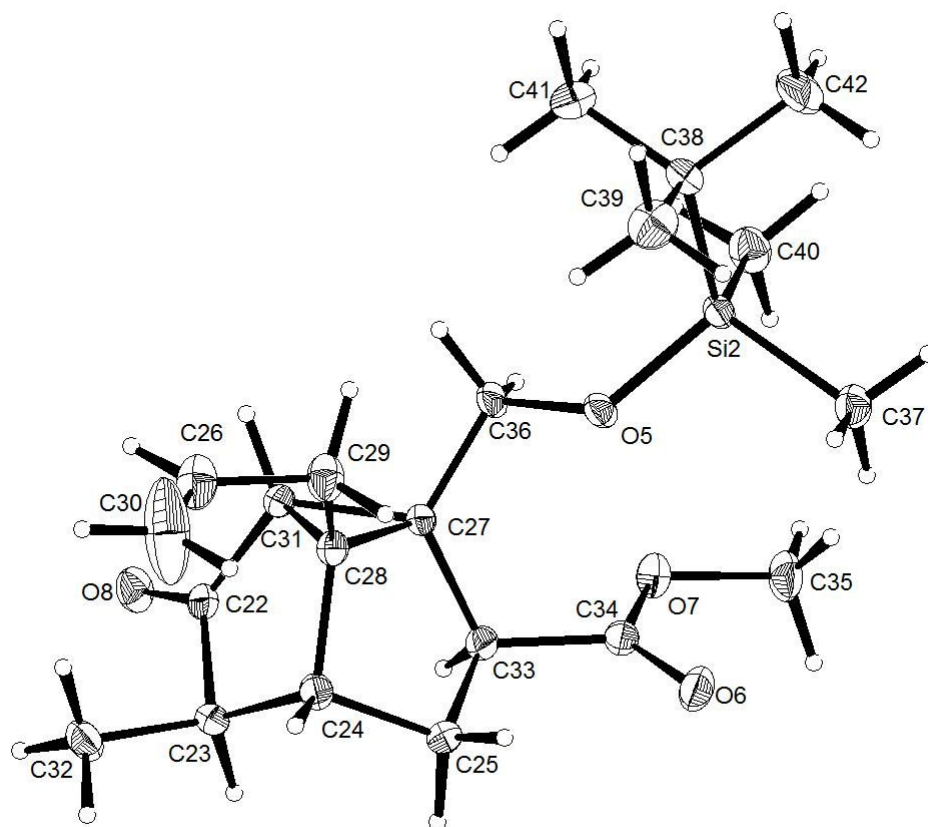
X-ray Crystallographic Analysis

X-ray crystallographic analysis of 18.

Crystal data and structure refinement

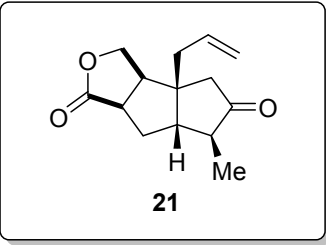
Empirical formula	C ₂₁ H ₃₄ O ₄ Si	
Formula weight	378.57	
Temperature	103(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 11.8826(2) Å	a = 67.8036(8)°.
	b = 14.0278(2) Å	b = 70.5153(8)°.
	c = 14.7882(3) Å	g = 77.3415(10)°.
Volume	2139.34(7) Å ³	
Z	4	
Density (calculated)	1.175 Mg/m ³	
Absorption coefficient	0.131 mm ⁻¹	
F(000)	824	
Crystal size	0.2 x 0.2 x 0.15 mm ³	
Theta range for data collection	1.548 to 28.323°.	
Index ranges	-15 ≤ h ≤ 15, -18 ≤ k ≤ 18, -19 ≤ l ≤ 18	
Reflections collected	40282	
Independent reflections	10616 [R(int) = 0.0491]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7457 and 0.7115	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	10616 / 0 / 469	
Goodness-of-fit on F ²	1.079	
Final R indices [I > 2σ(I)]	R1 = 0.0463, wR2 = 0.1125	
R indices (all data)	R1 = 0.0683, wR2 = 0.1246	

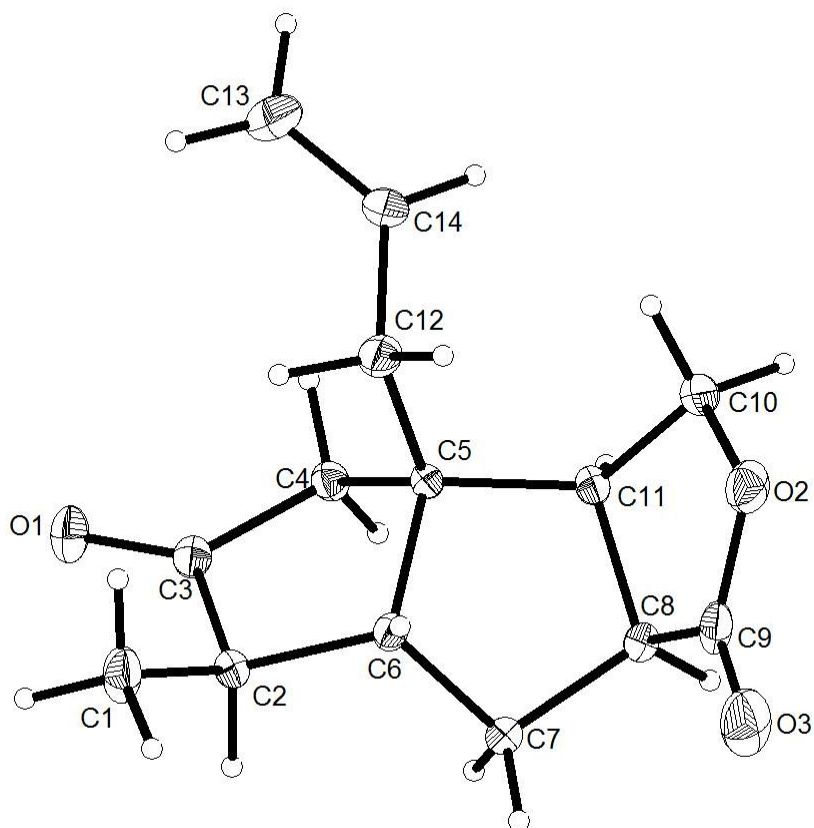
Extinction coefficient	n/a
Largest diff. peak and hole	0.391 and -0.467 e.Å ⁻³



X-ray thermal ellipsoid plot of **18** (50% probability)

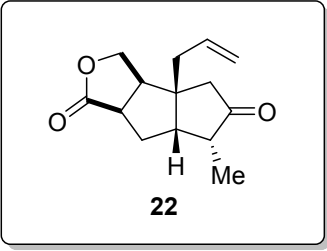
X-ray crystallographic analysis of 21.Crystal data and structure refinement

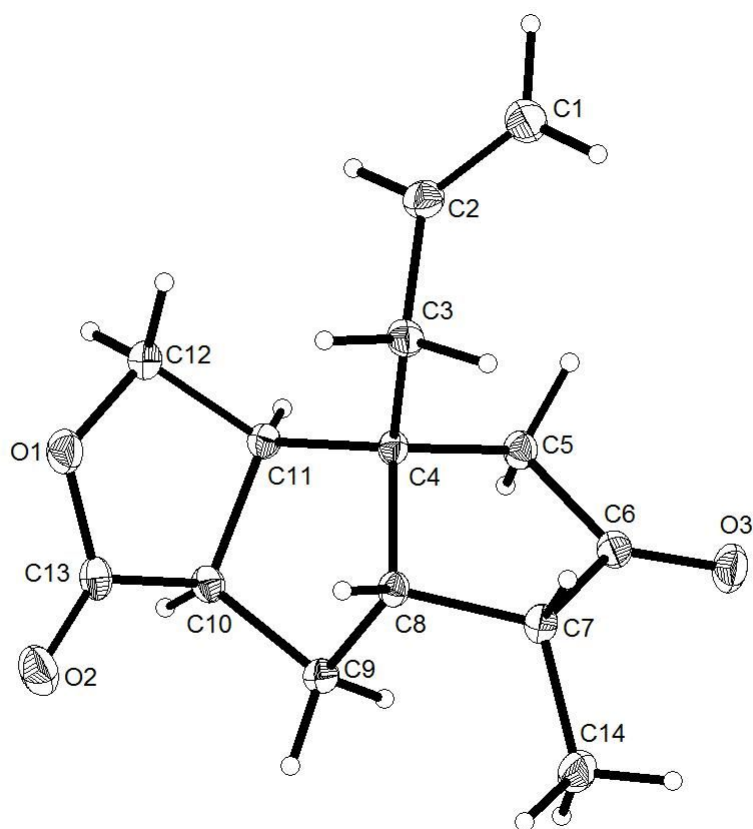
Empirical formula	C ₁₄ H ₁₈ O ₃	
Formula weight	234.28	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 6.4887(4) Å	a = 90°.
	b = 7.1740(4) Å	b = 93.117(4)°.
	c = 25.6544(15) Å	g = 90°.
Volume	1192.44(12) Å ³	
Z	4	
Density (calculated)	1.305 Mg/m ³	
Absorption coefficient	0.090 mm ⁻¹	
F(000)	504	
Crystal size	0.2 x 0.2 x 0.2 mm ³	
Theta range for data collection	2.95 to 28.31°.	
Index ranges	-8 ≤ h ≤ 8, -9 ≤ k ≤ 9, -30 ≤ l ≤ 34	
Reflections collected	11785	
Independent reflections	2955 [R(int) = 0.0322]	
Completeness to theta = 28.31°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7457 and 0.7116	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2955 / 0 / 154	
Goodness-of-fit on F ²	1.031	
Final R indices [I > 2σ(I)]	R1 = 0.0411, wR2 = 0.0977	
R indices (all data)	R1 = 0.0558, wR2 = 0.1067	
Largest diff. peak and hole	0.380 and -0.207 e.Å ⁻³	



X-ray thermal ellipsoid plot of **21** (50% probability)

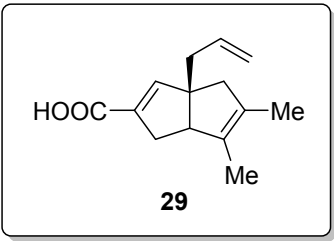
X-ray crystallographic analysis of 22.Crystal data and structure refinement

Empirical formula	C ₁₄ H ₁₈ O ₃	
Formula weight	234.28	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 6.57590(10) Å	a = 90.6670(10)°.
	b = 7.08190(10) Å	b = 99.8020(10)°.
	c = 13.0369(3) Å	g = 100.1960(10)°.
Volume	588.269(18) Å ³	
Z	2	
Density (calculated)	1.323 Mg/m ³	
Absorption coefficient	0.092 mm ⁻¹	
F(000)	252	
Crystal size	0.2 x 0.15 x 0.1 mm ³	
Theta range for data collection	1.59 to 28.28°.	
Index ranges	-8 ≤ h ≤ 8, -9 ≤ k ≤ 8, -17 ≤ l ≤ 17	
Reflections collected	10548	
Independent reflections	2909 [R(int) = 0.0149]	
Completeness to theta = 28.28°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7457 and 0.7182	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2909 / 0 / 154	
Goodness-of-fit on F ²	1.064	
Final R indices [I > 2σ(I)]	R1 = 0.0370, wR2 = 0.0980	
R indices (all data)	R1 = 0.0412, wR2 = 0.1017	
Largest diff. peak and hole	0.386 and -0.199 e.Å ⁻³	



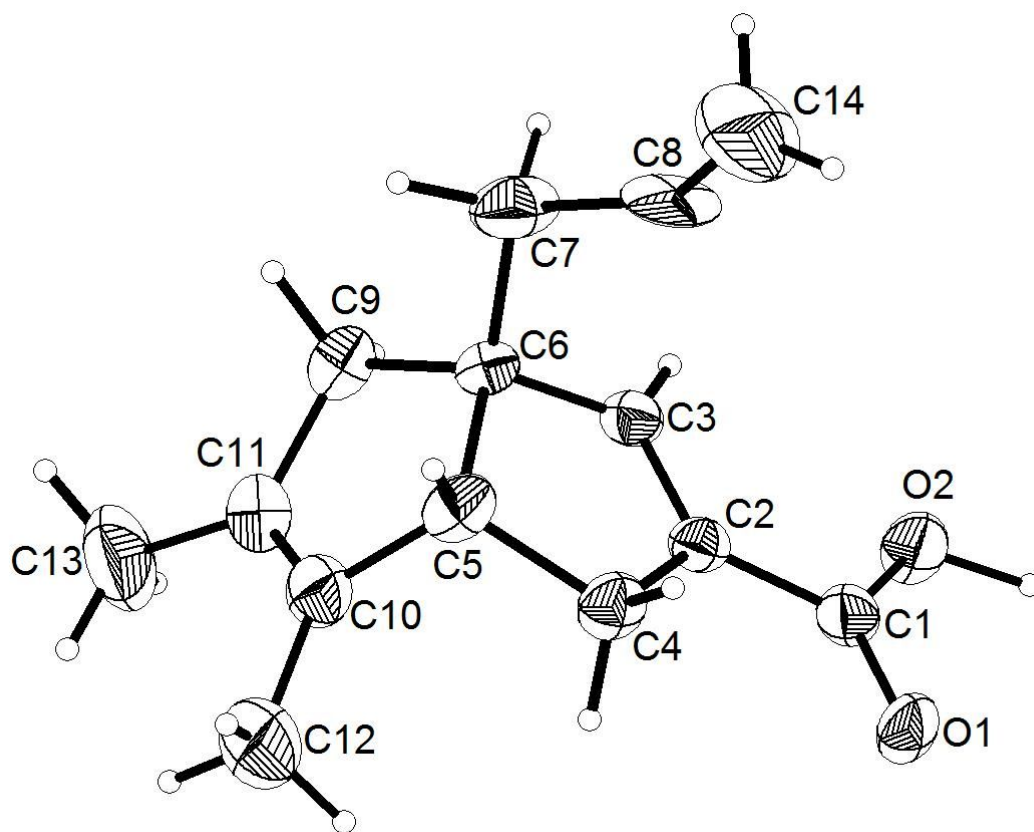
X-ray thermal ellipsoid plot of **22** (50% probability)

X-ray crystallographic analysis of 29.Crystal data and structure refinement

Empirical formula	C ₁₄ H ₁₇ O ₂	
Formula weight	217.27	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	$\alpha = 90^\circ$. $\beta = 105.433(2)^\circ$. $\gamma = 90^\circ$.
Space group	P2 ₁ /n	
Unit cell dimensions	a = 7.6989(2) Å	
	b = 14.1778(3) Å	
	c = 12.0083(3) Å	1263.48(5) Å ³
Volume		
Z	4	
Density (calculated)	1.142 Mg/m ³	
Absorption coefficient	0.075 mm ⁻¹	0.2 x 0.2 x 0.1 mm ³
F(000)	468	
Crystal size		
Theta range for data collection	2.271 to 28.282°.	
Index ranges	-10 ≤ h ≤ 10, -18 ≤ k ≤ 18, -15 ≤ l ≤ 12	11168
Reflections collected		
Independent reflections	3092 [R(int) = 0.0577]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	0.7457 and 0.6954
Max. and min. transmission		
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3092 / 6 / 166	
Goodness-of-fit on F ²	1.126	R ₁ = 0.1031, wR ₂ = 0.2106
Final R indices [I > 2σ(I)]		
R indices (all data)	R ₁ = 0.1600, wR ₂ = 0.2306	
Extinction coefficient	n/a	

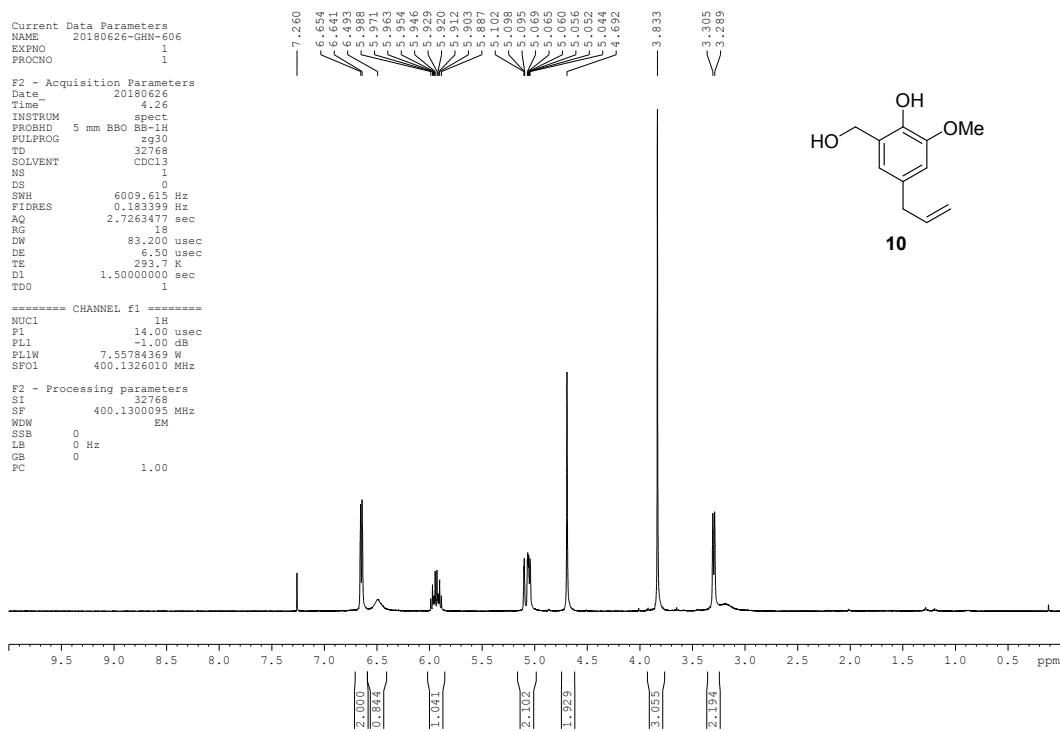
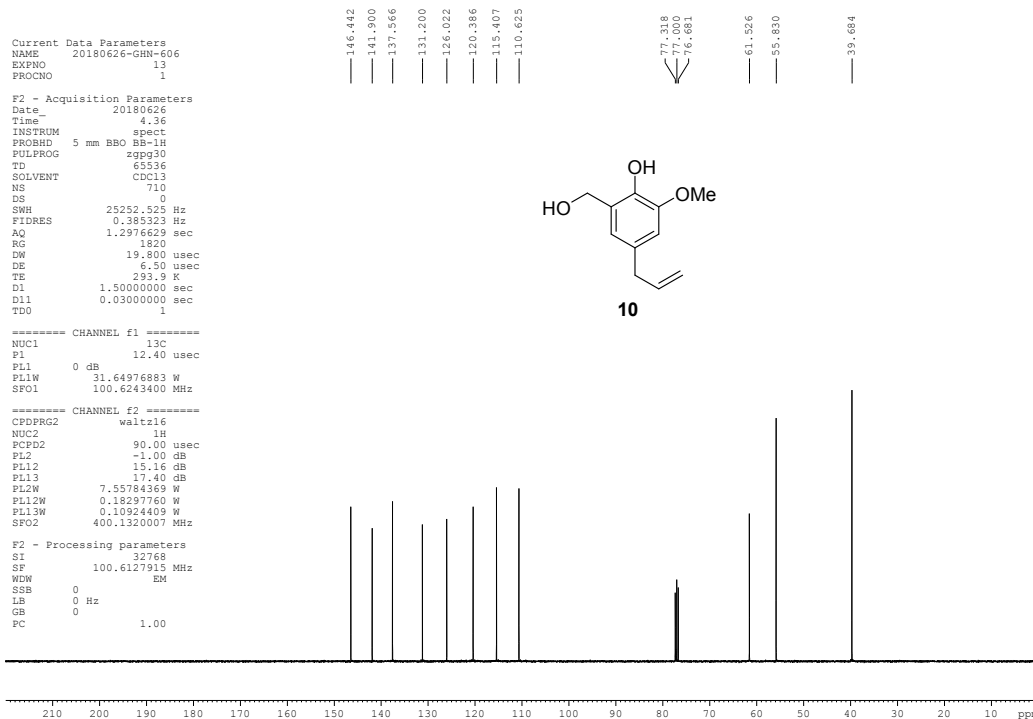
Largest diff. peak and hole

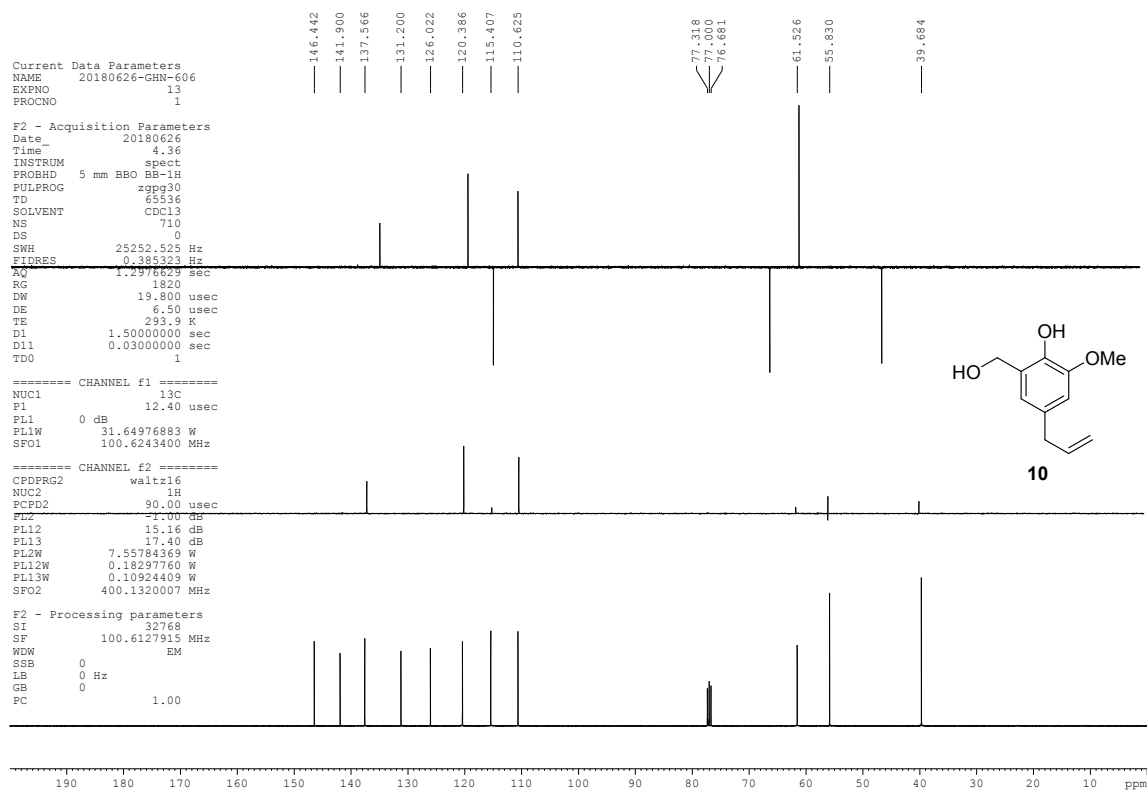
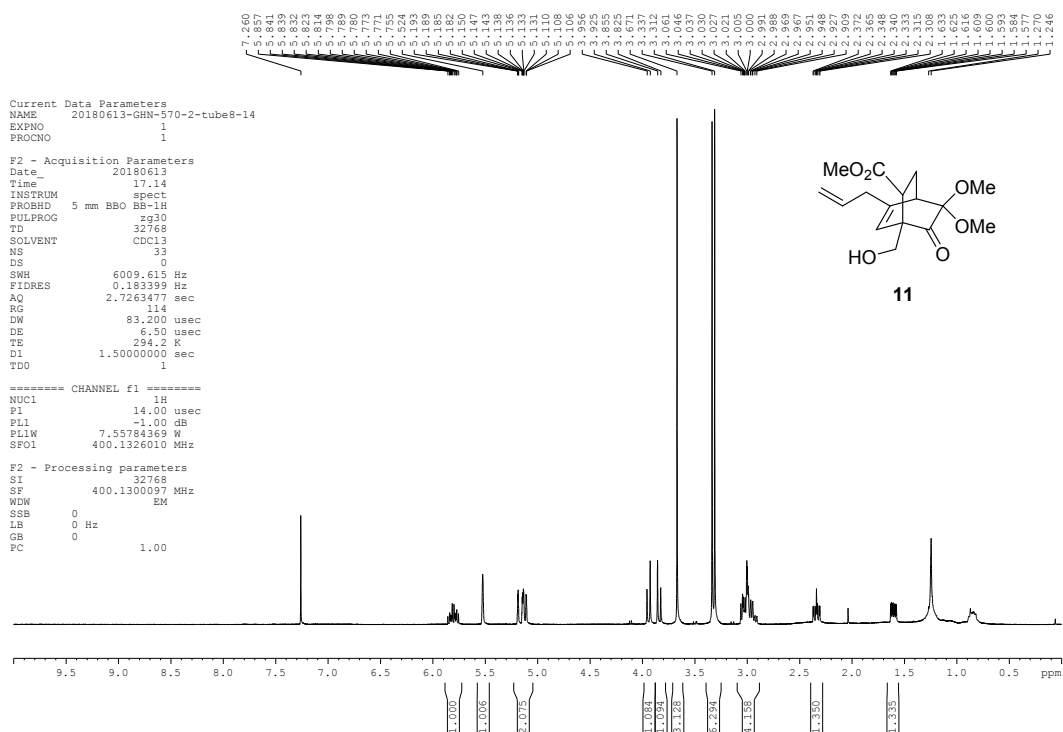
0.325 and -0.294 e.Å⁻³

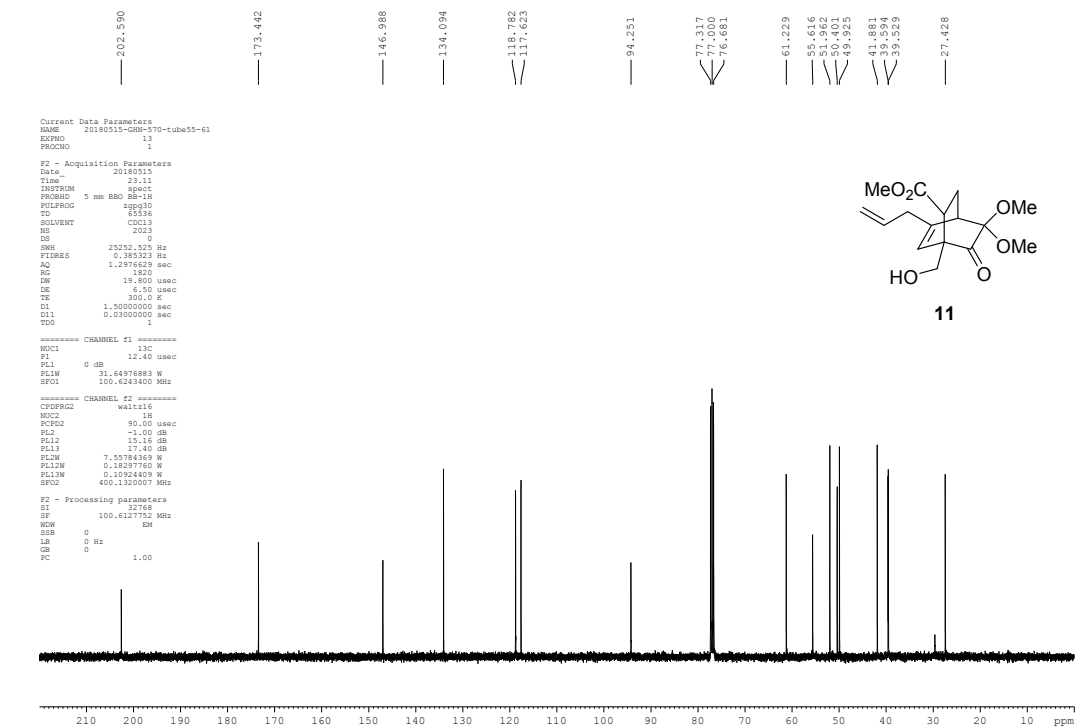
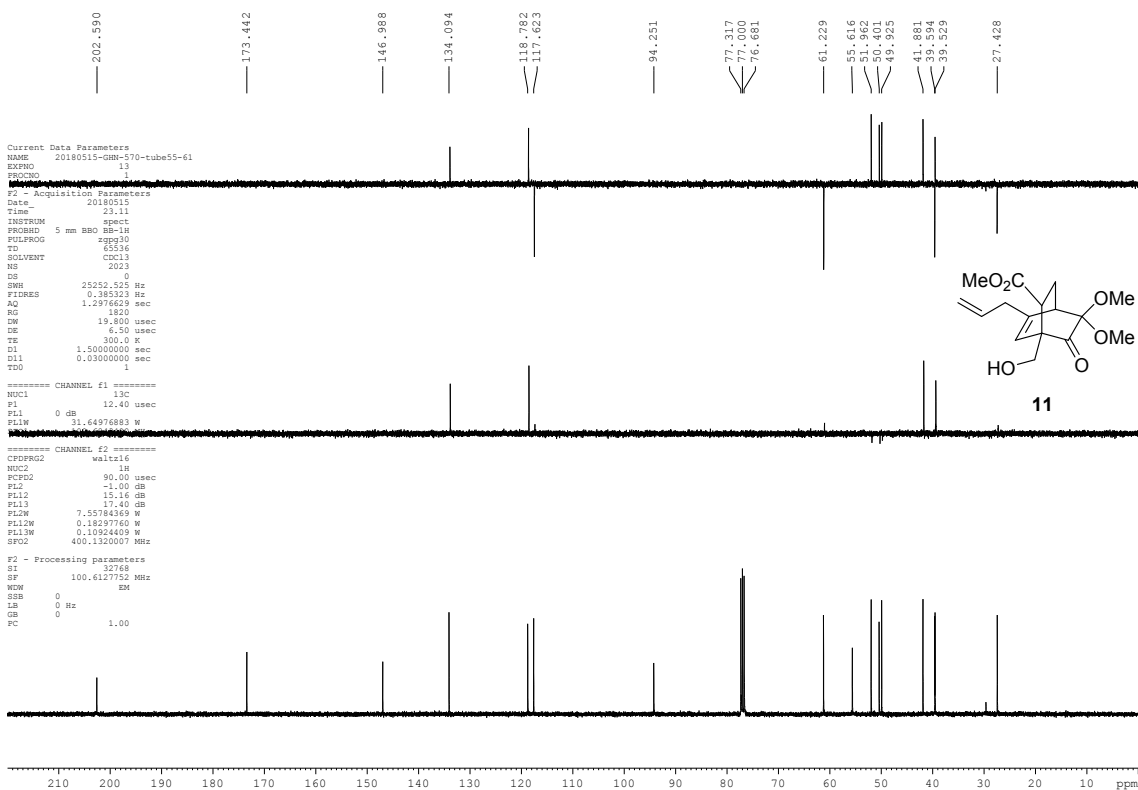


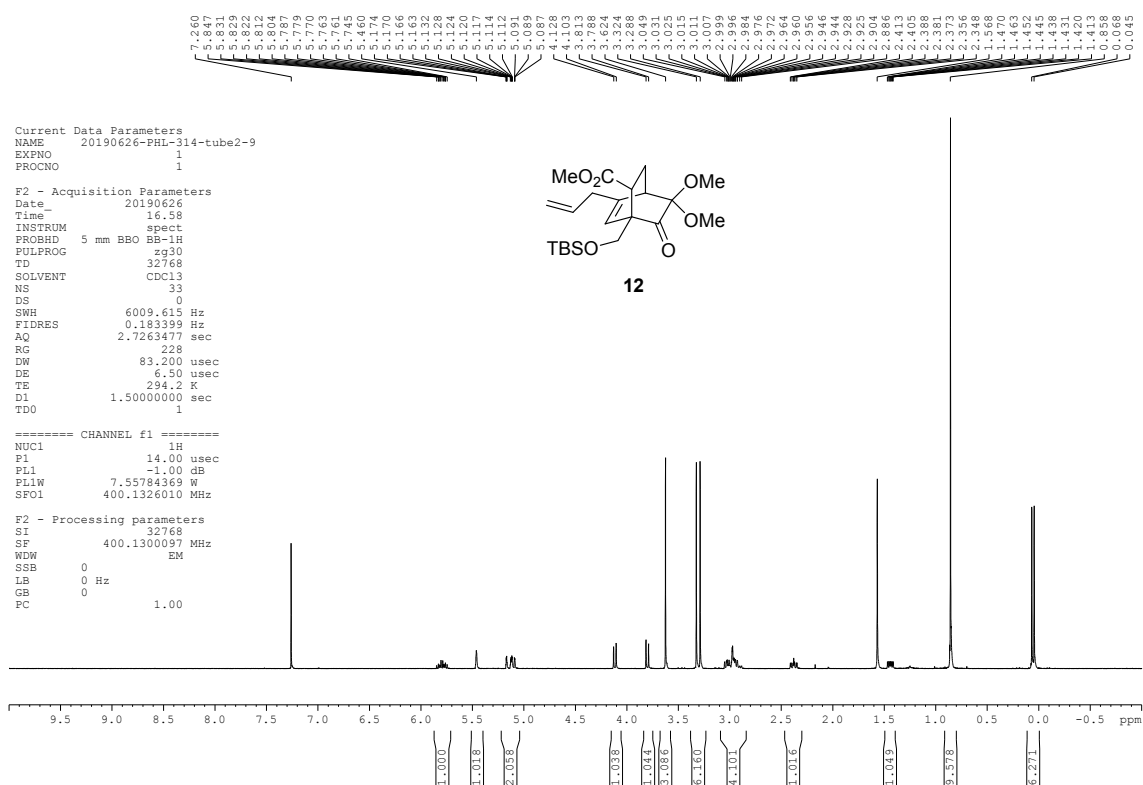
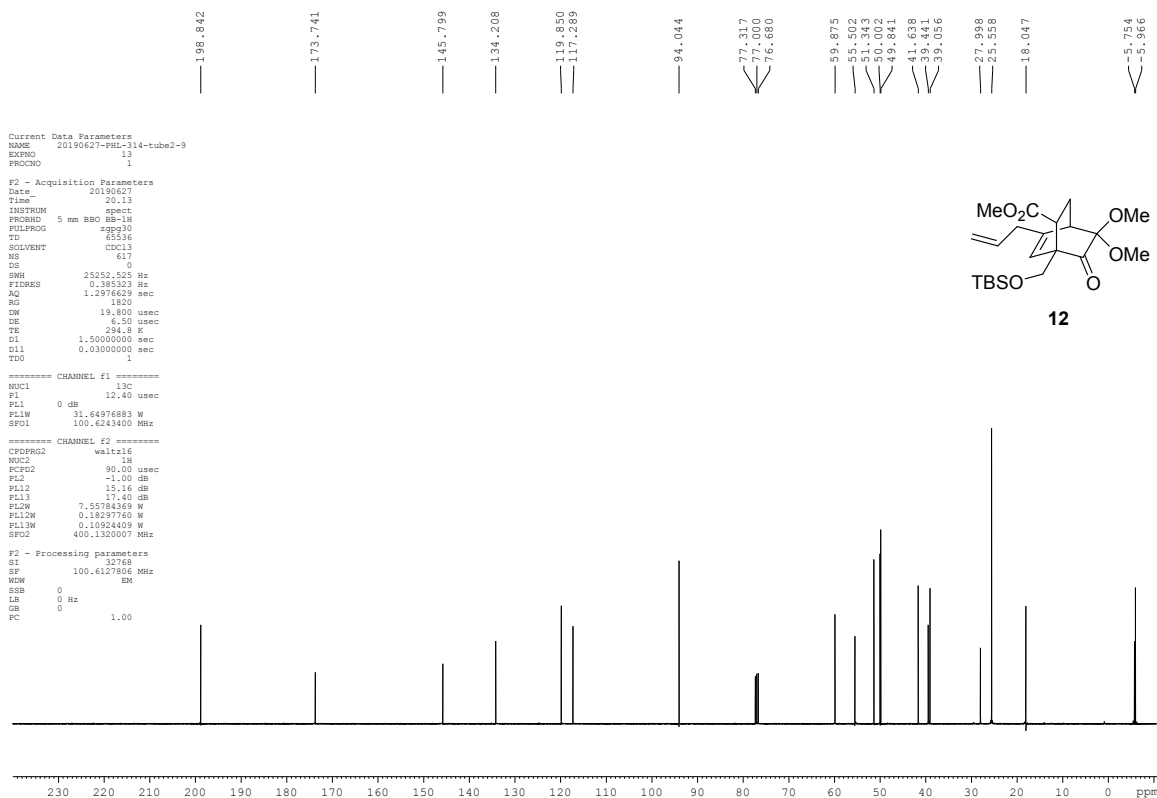
X-ray thermal ellipsoid plot of **29** (50% probability)

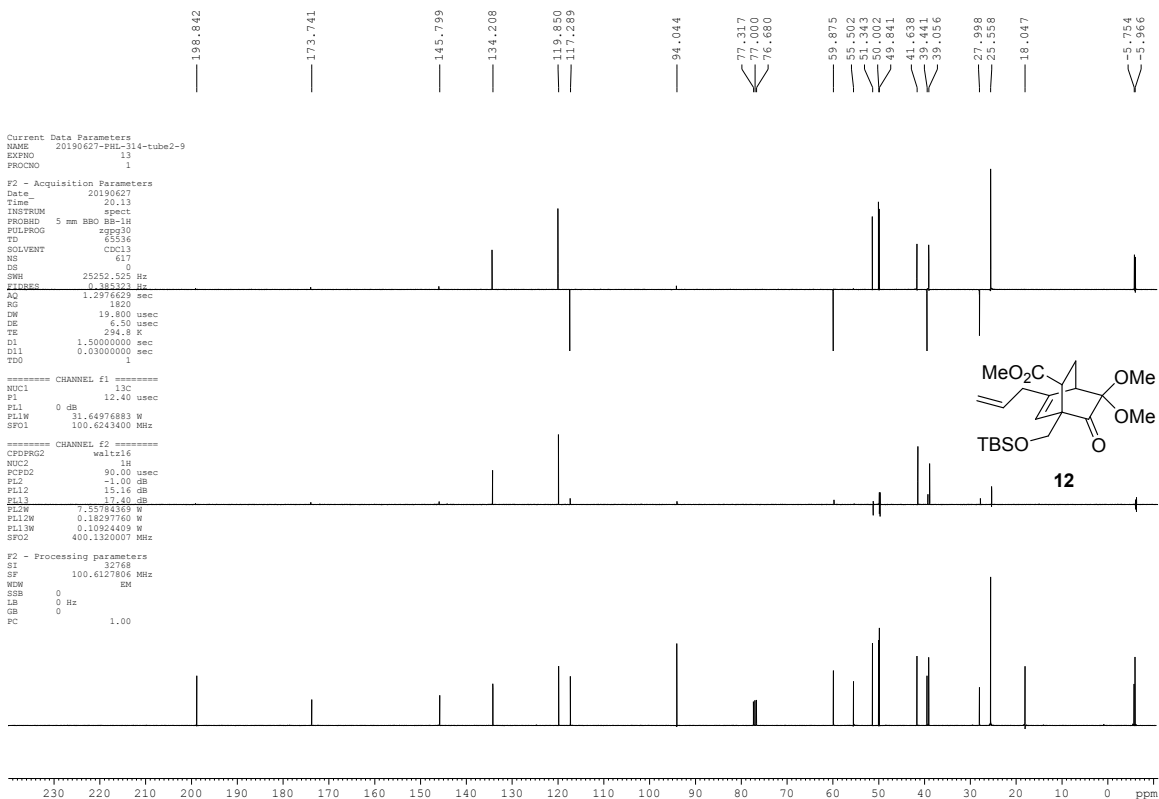
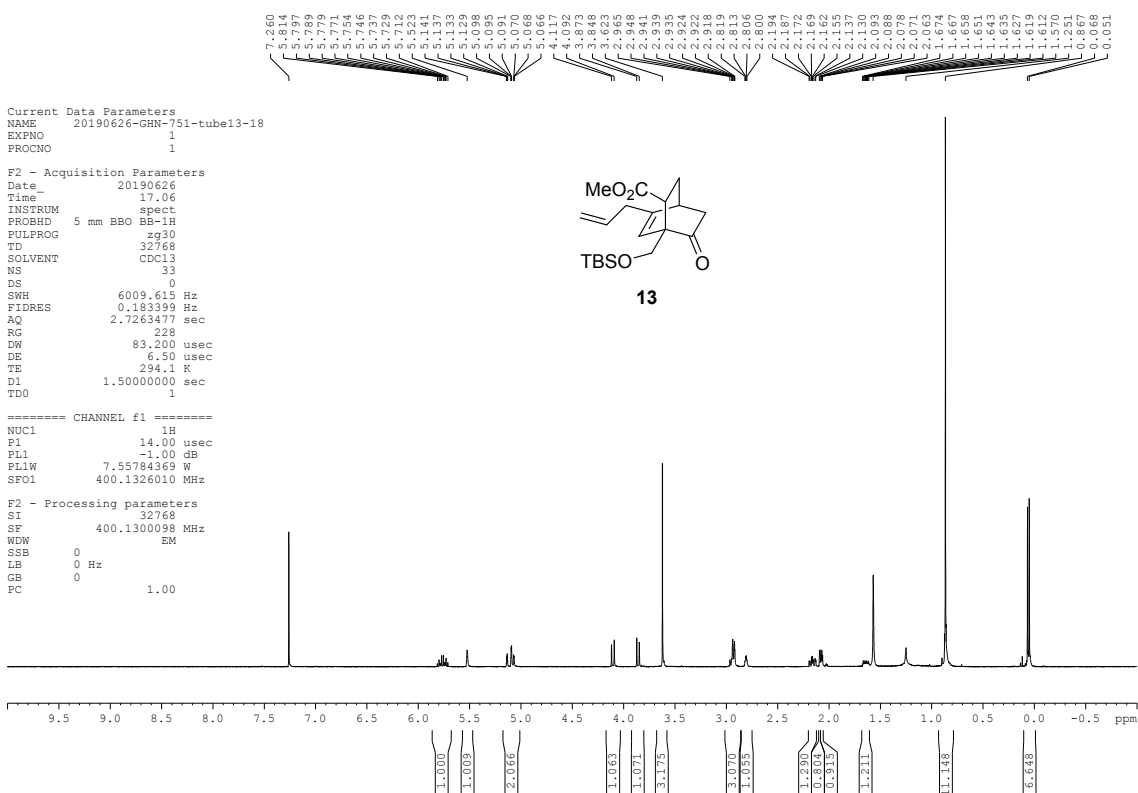
Spectroscopic Data

¹H NMR of 10, CDCl₃, 400 MHz, 24 °C¹³C NMR of 10, CDCl₃, 100 MHz, 24 °C

DEPT 135, DEPT 90 and ^{13}C NMR of 10, CDCl_3 , 100 MHz, 24 °C ^1H NMR of 11, CDCl_3 , 400 MHz, 24 °C

^{13}C NMR of 11, CDCl_3 , 100 MHz, 24 °C**DEPT 135, DEPT 90 and ^{13}C NMR of 11, CDCl_3 , 100 MHz, 24 °C**

¹H NMR of 12, CDCl₃, 400 MHz, 24 °C**¹³C NMR of 12, CDCl₃, 100 MHz, 24 °C**

DEPT 135, DEPT 90 and ^{13}C NMR of 12, CDCl_3 , 100 MHz, 24 °C ^1H NMR of 13, CDCl_3 , 400 MHz, 24 °C

Current Data Parameters
 NAME 20190627-DHM-51-tube13-18
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190627
 Time 20.47
 INSTRUM spect
 PROBRD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT cdcl3
 NS 256
 DS 8
 SWH 25252.525 Mhz
 FIDRES 0.385323 Mhz
 AQ 1.2976629 sec
 RG 1820
 DW 19.800 usec
 DE 6.50 usec
 TE 294.0 K
 D1 1.50000000 sec
 D11 0.03000000 sec
 TDO 4

***** CHANNEL f1 *****
 NUC1 13C
 P1 12.40 usec
 PL1 0 dB
 PL1W 31.64976883 W
 SFO1 100.6243400 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 15.16 dB
 PL13 17.40 dB
 PL2W 7.55784369 W
 PL12W 0.18297760 W
 PL13W 0.10924409 W
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127754 MHz
 NWDW 824
 SSB 0
 LS 0 Hz
 GB 0
 PC 1.00

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

208.406
173.83
147.439
134.316
120.031
117.167
77.319
77.000
76.683
60.355
55.997
51.486
39.907
38.099
38.916
35.569
32.252
25.718
18.221
5.633
5.800

MeO₂C
TBSO
13

Current Data Parameters
NAME 20190627-GHN-751-tubel3-18
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190627
Time_ 20.47
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CCl₃
NS 256
DS 0
SWH 25252.525 Hz

===== CHANNEL f1 =====
NUC1 ¹³C
P1 12.40 usec
PL1 0 dB
PL1W 31.64976883 W
SFO1 100.6243400 MHz

===== CHANNEL f2 =====
CFPRMG2 waltz16
NUC2 ¹H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.16 dB
PL13 17.40 dB
PL1W 0.18287765 W
PL1W 0.10924409 W
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127754 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

208.406
175.83
147.439
134.316
120.031
117.167
77.319
77.000
76.683
60.355
55.997
51.486
39.807
39.099
38.666
38.545
32.252
32.252
25.718
18.221
5.625
5.625
5.625

MeO₂C
TBSO
13

Current Data Parameters

NAME 20190508-PHL-34-50
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20190508
Time 17.01
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 36
DW 83.200 usec
DE 6.50 usec
TE 293.8 K
D1 1.5000000 sec
TD0 1

===== CHANNEL f1 =====

NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 7.55784369 W
SFO1 400.1326010 MHz

F2 - Processing parameters

SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

7.260
5.906
5.887
5.879
5.863
5.821
5.803
5.188
5.146
5.122
5.096
3.743
3.716
3.693
3.666
2.919
2.904
2.890
2.875
2.772
2.768
2.753
2.593
2.571
2.548
2.524
2.497
2.481
2.460
2.435
2.423
2.388
2.371
2.360
2.345
1.810
1.765
1.694
1.679
1.650
1.550
1.576
1.252
0.863
0.022
0.011

14

1.000
1.985
5.123
0.919
0.932
3.818
0.948
1.016
1.111
10.096
6.135

Current Data Parameters
NAME 20190506-PIL-34-50-13c
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190506
Time 23.42
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 25250.555 Hz
FIDRES 0.385323 Hz
AQ 1.29786219 sec
RG 912
DW 19.800 usec
DE 6.50 usec
TE 294.7 K
D1 1.50000000 sec
D11 0.03000000 sec
TDO 1

***** CHANNEL f1 *****
NUC1 13C
P1 12.40 usec
PL1 0 dB
PL1W 31.64976883 W
SFO1 100.6243400 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.16 dB
PL13 17.40 dB
PL2W 7.55781489 W
PL12W 0.18297760 W
PL13W 0.10924409 W
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127721 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

Chemical structure of compound 14 is shown above the spectrum. The structure is a bicyclic compound with a TBSO group, a methoxy group (MeO₂C), and a vinyl group.

Chemical shift (ppm): 230, 220, 210, 200, 190, 180, 170, 160, 150, 140, 130, 120, 110, 100, 90, 80, 70, 60, 50, 40, 30, 20, 10, 0, ppm

Current Data Parameters

NAME 20190506-PBL-34-50-13C
EXPRO 13
PROCNO 1

F2 - Acquisition Parameters

Date_ 20190506
Time_ 23.42
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1004
DS 0
SHE 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 912
DW 19.800 usec
DE 6.50 usec
TE 300.2 K

===== CHANNEL f1 =====

NUC1 13C
P1 12.40 usec
PL1 0 dB
PL1W 31.64976883 W
SFO1 100.6263400 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 15.16 dB
PL13 17.40 dB
PL2W 7.55784369 W
PL12W 0.18297760 W
PL13W 0.103254400 W
SFO2 400.1460950 MHz

F2 Acquisition Parameters

SF 100.6127721 MHz
MW EM
SBB 0
LB 0 Hz
GB 0
PC 1.00

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

214.047
173.175
135.253
117.103
77.317
77.062
76.681
61.755
52.010
51.833
48.570
46.207
46.102
42.337
40.903
33.206
25.745
18.063
5.496
5.728

TBSO
MeO₂C
H
14

COC(=O)[C@H]1[C@H](OC(C)(C)C)C2=C[C@H](C=C)C[C@@H]2[C@H]1O

Current Data Parameters

NAME 20180626-GHN-607-tube7-17
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20180626
Time 1.51
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 17
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 12.7
DW 83.200 usec
DE 6.50 usec
TE 293.7 K
D1 1.50000000 sec
TDO 1

===== CHANNEL f1 =====

NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PLW1 7.55784369 W
SFO1 400.1326010 MHz

F2 - Processing parameters

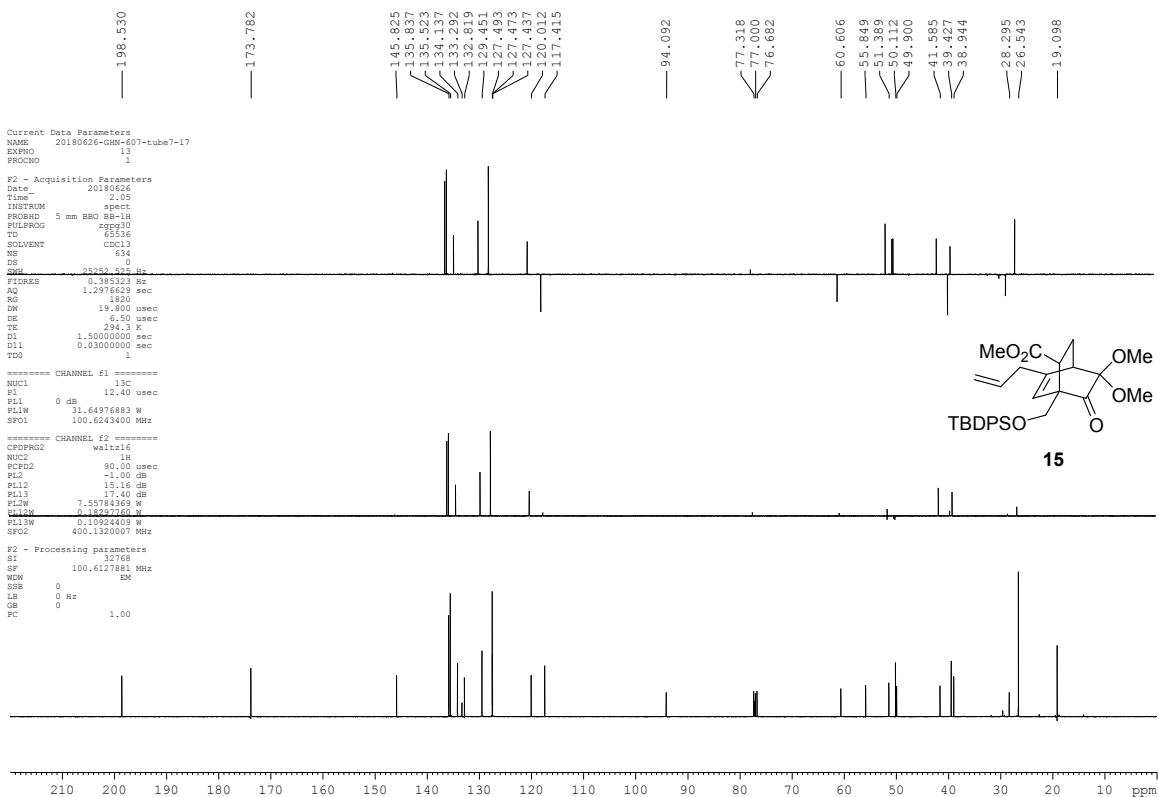
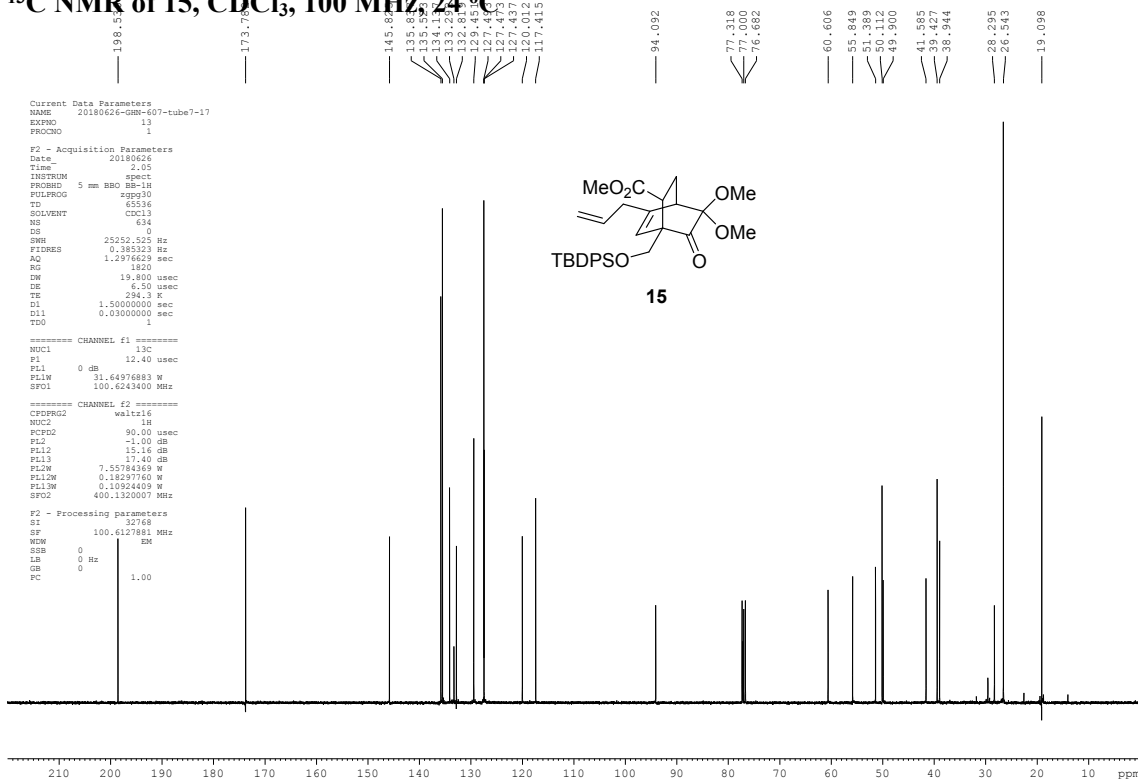
SF 400.130091 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

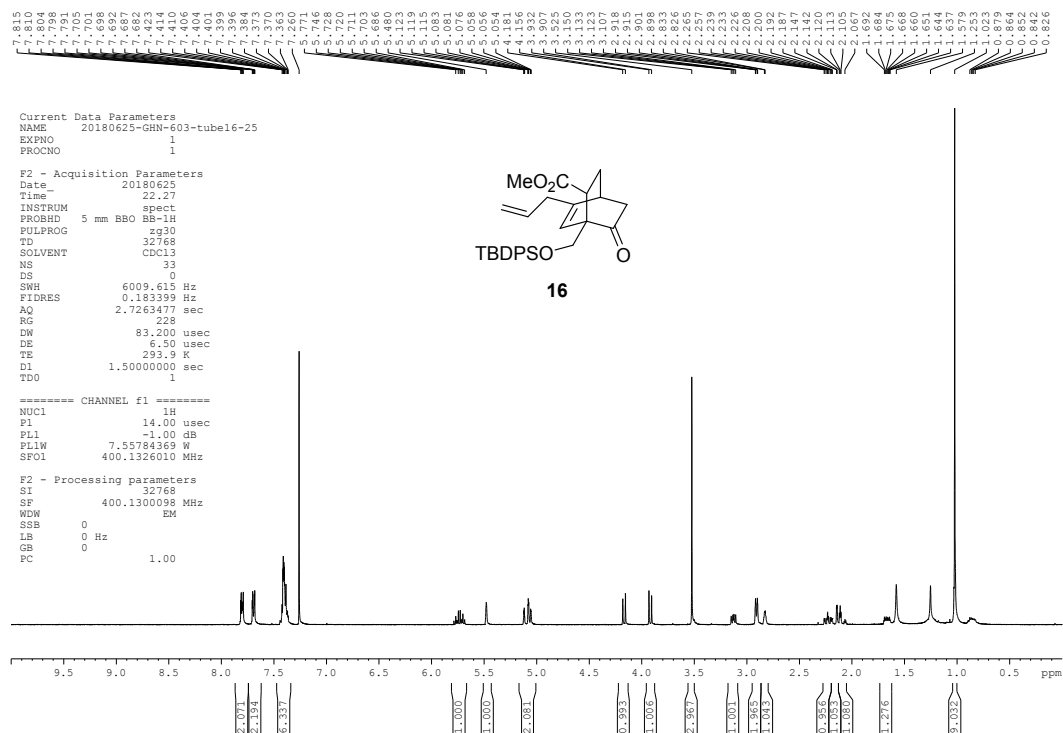
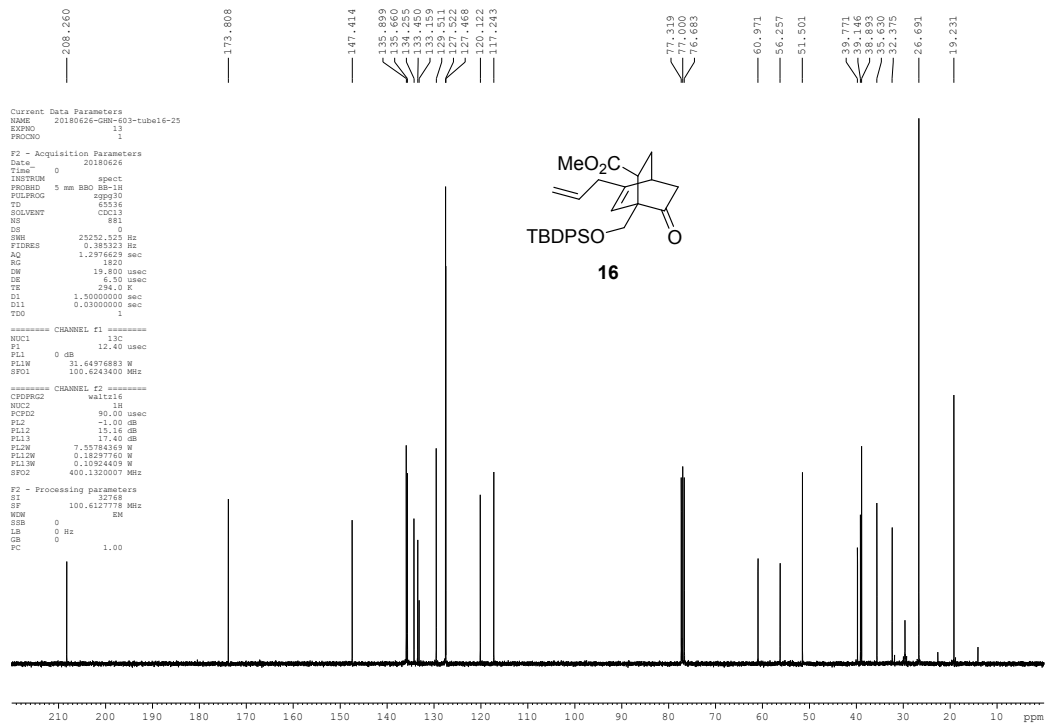
COC(=O)C1=CC(=C(C=C1)OC(=O)C)OC(=O)C

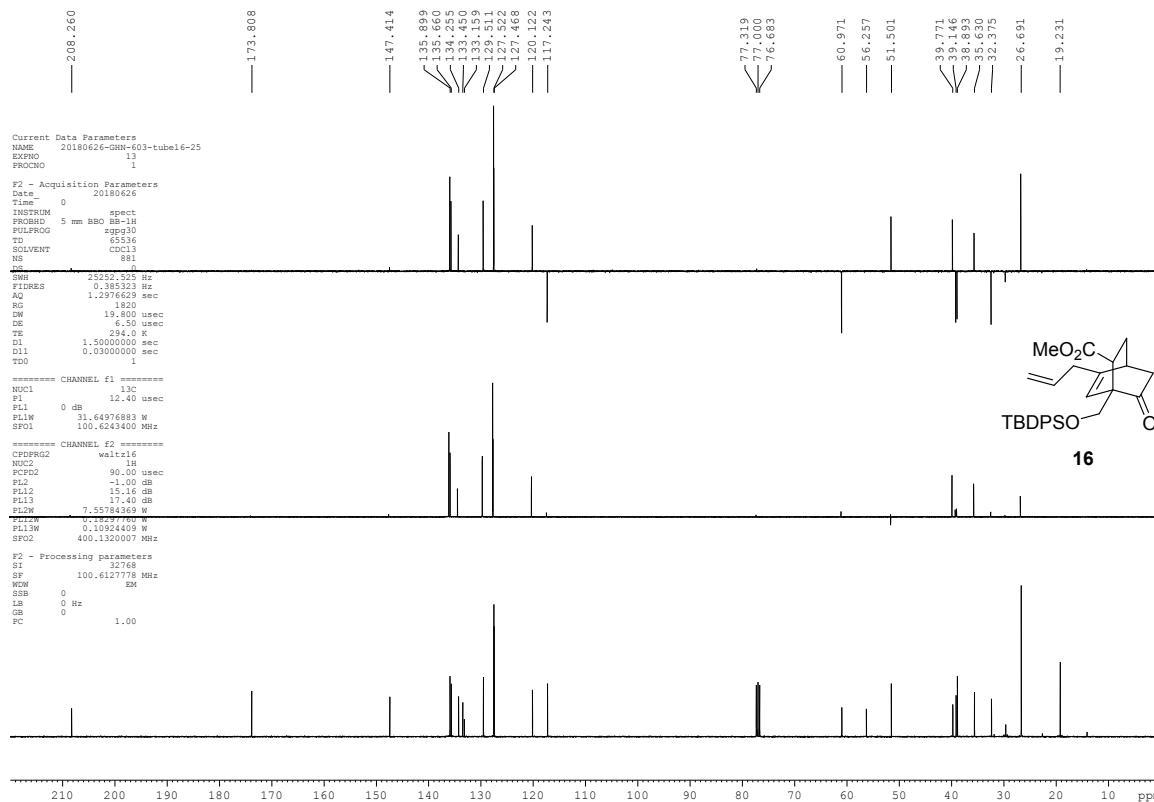
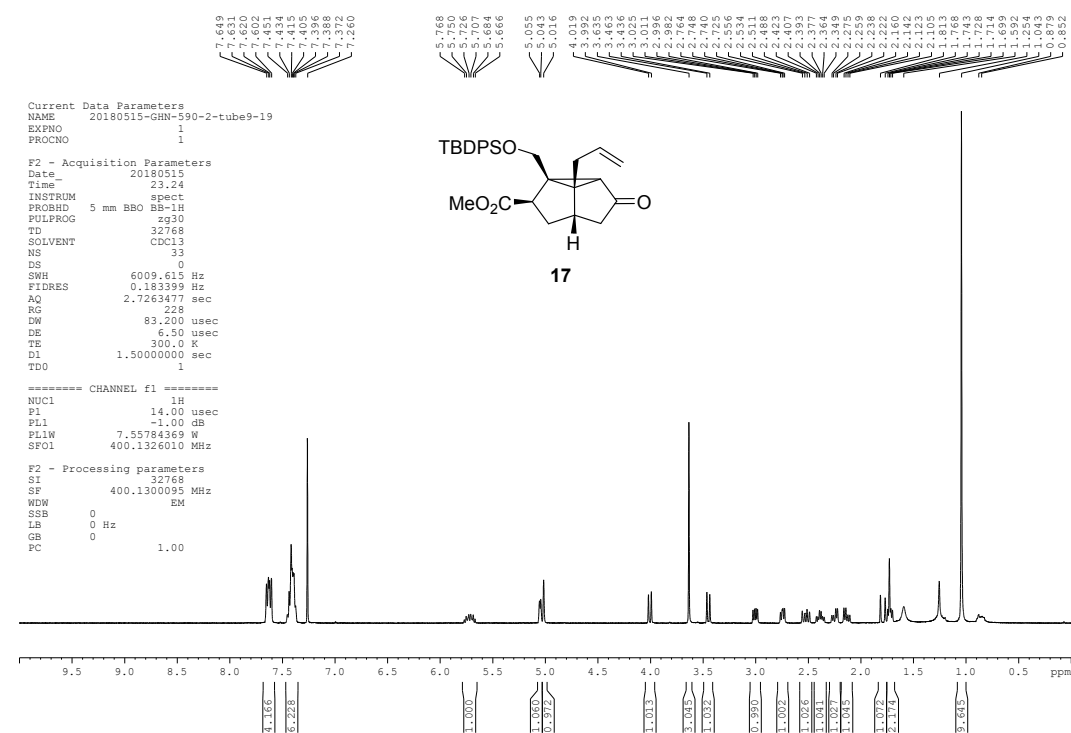
15

TBDPSO

Integration values (from left to right): 2.021, 2.060, 6.139, 1.000, 1.002, 2.042, 1.000, 1.000, 3.055, 2.033, 1.053, 2.052, 1.010, 1.213, 9.281.



¹H NMR of 16, CDCl₃, 400 MHz, 24 °C**¹³C NMR of 16, CDCl₃, 100 MHz, 24 °C**

DEPT 135, DEPT 90 and ^{13}C NMR of 16, CDCl_3 , 100 MHz, 24 °C ^1H NMR of 17, CDCl_3 , 400 MHz, 24 °C

—213.416
 —173.657
 —135.741
 —135.618
 —134.958
 —132.900
 —129.851
 —129.751
 —127.736
 —127.672
 —117.682
 —77.317
 —77.000
 —76.682
 —63.036
 —52.127
 —51.862
 —48.965
 —47.410
 —46.276
 —42.961
 —41.225
 —33.211
 —26.746
 —19.132

Current Data Parameters
 NAME 20180514-GHM-590-2-tube9-19
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180514
 Time 23.09
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1255
 DS 0
 SWH 25252.525 Hz
 FIDRES 0.383323 Hz
 AQ 1.2976629 sec
 RG 1620
 DW 19.800 usec
 DE 6.50 usec
 TE 295.0 K
 D1 1.5000000 sec
 D11 0.0300000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 12.40 usec
 PL1 0 dB
 PL1W 31.64976883 W
 SFO1 100.6243400 MHz

===== CHANNEL f2 =====
 CDPGR2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 15.16 dB
 PL13 17.40 dB
 PL2W 7.55784369 W
 PL12W 0.18229760 W
 PL13W 0.10924409 W
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127745 MHz
 WDW EM
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

Chemical structure of compound 17, a bicyclic molecule. It features a ketone group (=O) on the right, a methoxy group (MeO₂C) on the left, and a TBDPSO group (tert-butyldiphenylsilyloxy) at the top. The structure is labeled with '17' below it.

17

¹H NMR spectrum of compound 17 in CDCl₃. The x-axis represents the chemical shift in ppm, ranging from 220 to 0. The spectrum shows several sharp peaks: a multiplet around 7.3 ppm (aromatic protons), a multiplet around 4.7 ppm (protons adjacent to the methoxy group), a singlet around 3.7 ppm (methoxy protons), and a multiplet around 1.3 ppm (protons of the bicyclic system). The solvent peak for CDCl₃ is visible at 7.26 ppm.

Current Data Parameters
 NAME 20180514-GMR-590-2-tube9-19
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180514
 Time 23.09
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1253
 DS 4
 SWH 25252.525 Hz
 FIDRES 0.385323 Hz
 RG 1620
 DW 19.890 usec
 DE 6.50 usec
 TE 295.0 K
 D1 1.50000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 12.40 usec
 PL1 0 dB
 PL1W 31.64976883 W
 SFO1 100.6241400 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 15.14 dB
 PL13 17.40 dB
 PL1W 7.55784363 W
 PL12W 0.18297760 W

F2 - Processing parameters
 SI 32768
 SF 100.6127745 MHz
 WDW EM
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00

135.741
133.658
133.078
132.900
129.851
129.751
129.472
117.089

77.317
77.000
76.682

63.036
52.127
51.862
48.965
47.410
47.197
46.961
41.225
33.211
26.746
19.132

135.741
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129.472
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129.751
129.472
117.089

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129.472
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47.197
46.961
41.225
33.211
26.746
19.132

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117.089

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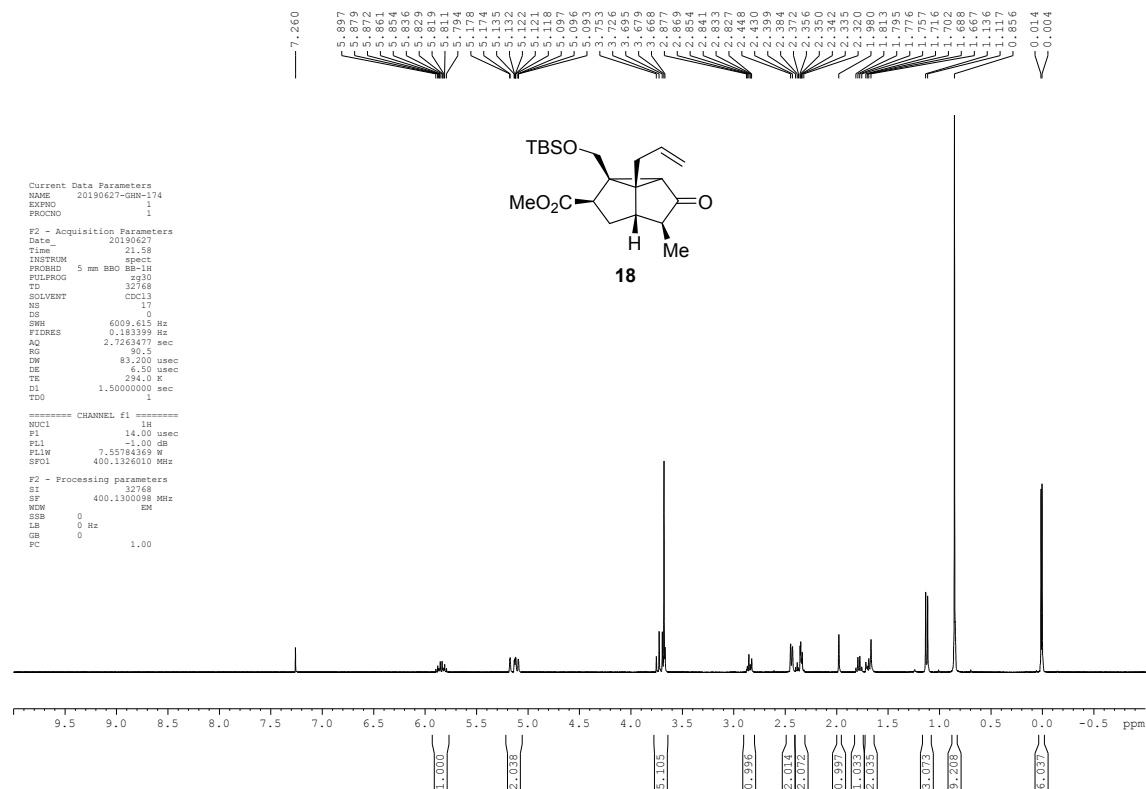
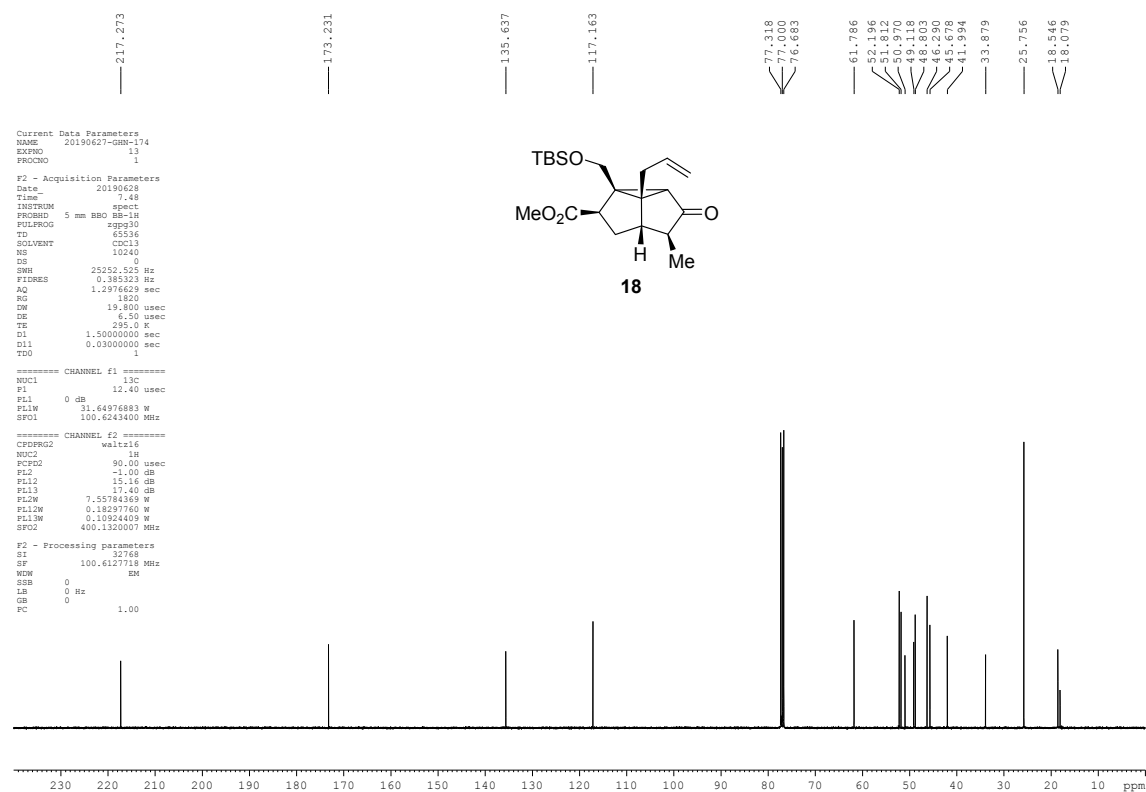
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77.000
76.682

63.036
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46.961
41.225
33.211
26.746
19.132

135.741
133.658
133.078
132.900
129.851
129.751
129.472
117.089

77.317
77.000
76.682

63.036
52.127
51.862
48.965
47.410
47.197

¹H NMR of 18, CDCl₃, 400 MHz, 24 °C**¹³C NMR of 18, CDCl₃, 100 MHz, 24 °C**

Current Data Parameters

NAME 20190627-GHN-174
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20190628
Time 7.48
INSTRUM spect
PROBHD 5 mm BBO BB-HL
P1 12.40 usec
PL1 0 dB
PL1W 31.64976883 W
SFO1 100.6243400 MHz

===== CHANNEL f1 =====

NUC1 13C
P1 12.40 usec
PL1 0 dB
PL1W 31.64976883 W
SFO1 100.6243400 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL2 19.800 usec
PL2W 7.55784369 W
PL2W 0.18297760 W
PL2W 0.1092469 W
SFO2 400.1320007 MHz

F2 - Processing parameters

SI 32768
SF 100.6127718 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
FC 1.00

Chemical Structure:

The structure shows a bicyclic system with a TBSO group, a MeO₂C group, a Me group, and a vinyl group. The carbon atom attached to the MeO₂C group is labeled **18**.

Current Data Parameters

NAME	20180625-GHN-604-2-tubel1-25
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20180625
Time	22.32
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl ₃
NS	33
DS	0
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	228
DW	83.200 usec
DE	6.50 usec
TE	293.9 K
D1	1.50000000 sec
TDO	1

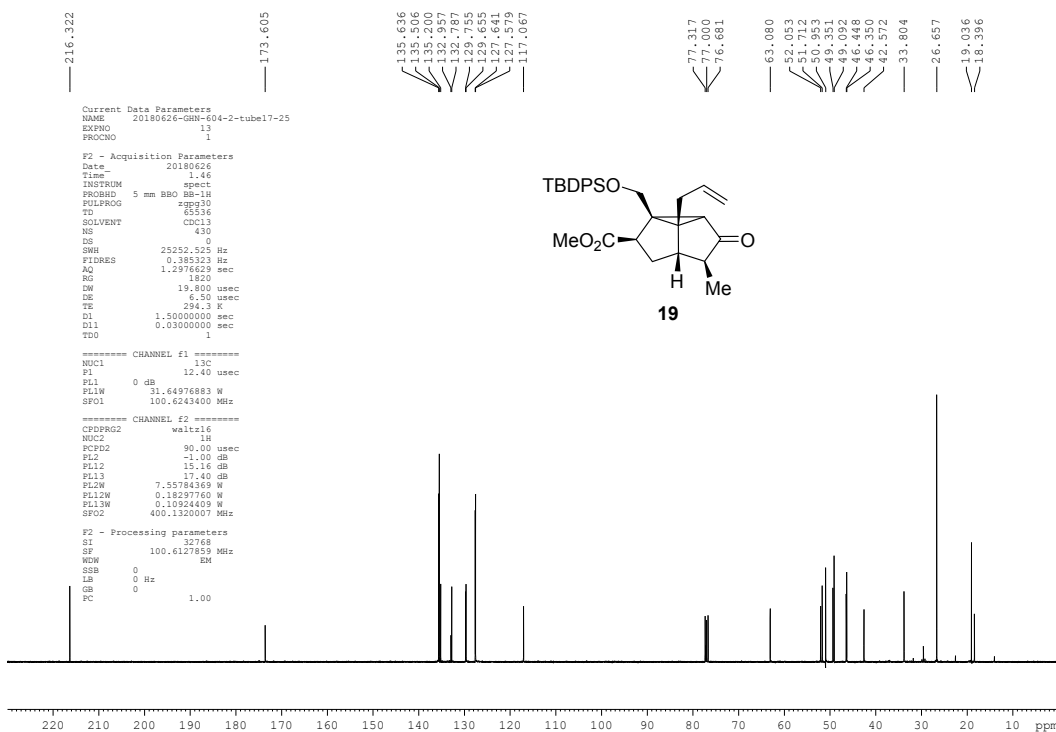
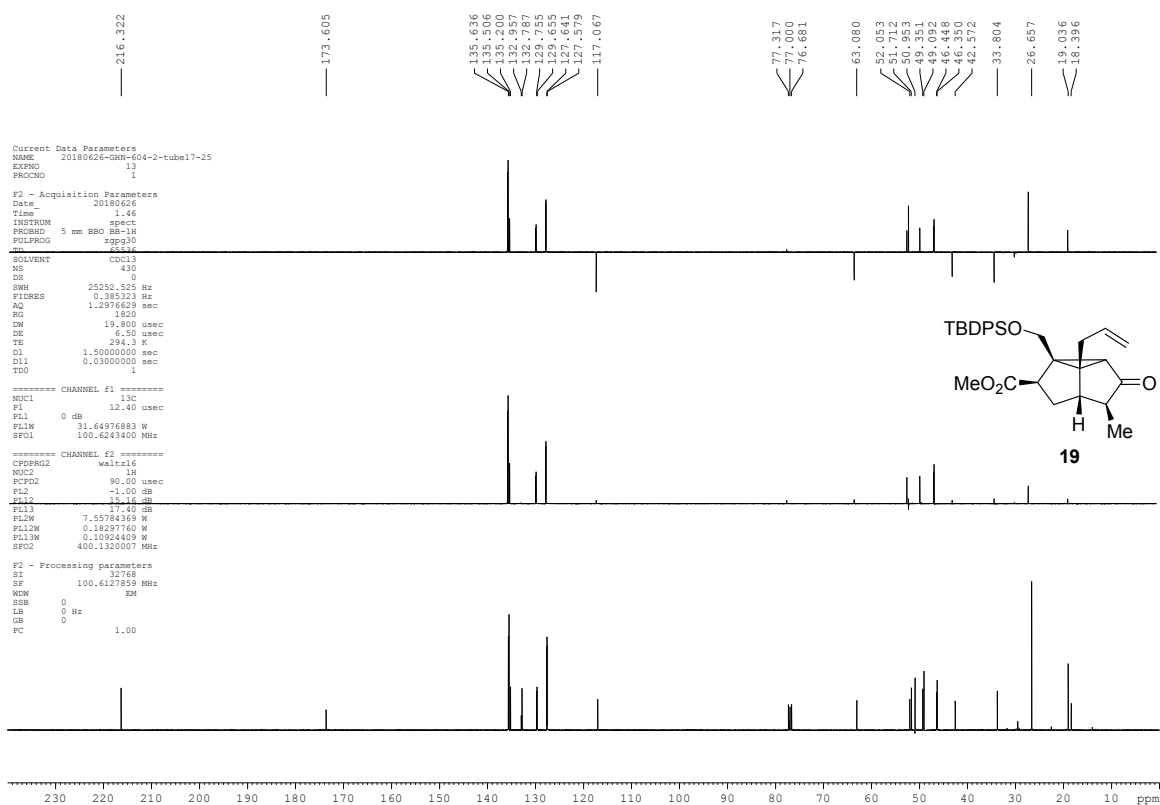
===== CHANNEL f1 =====

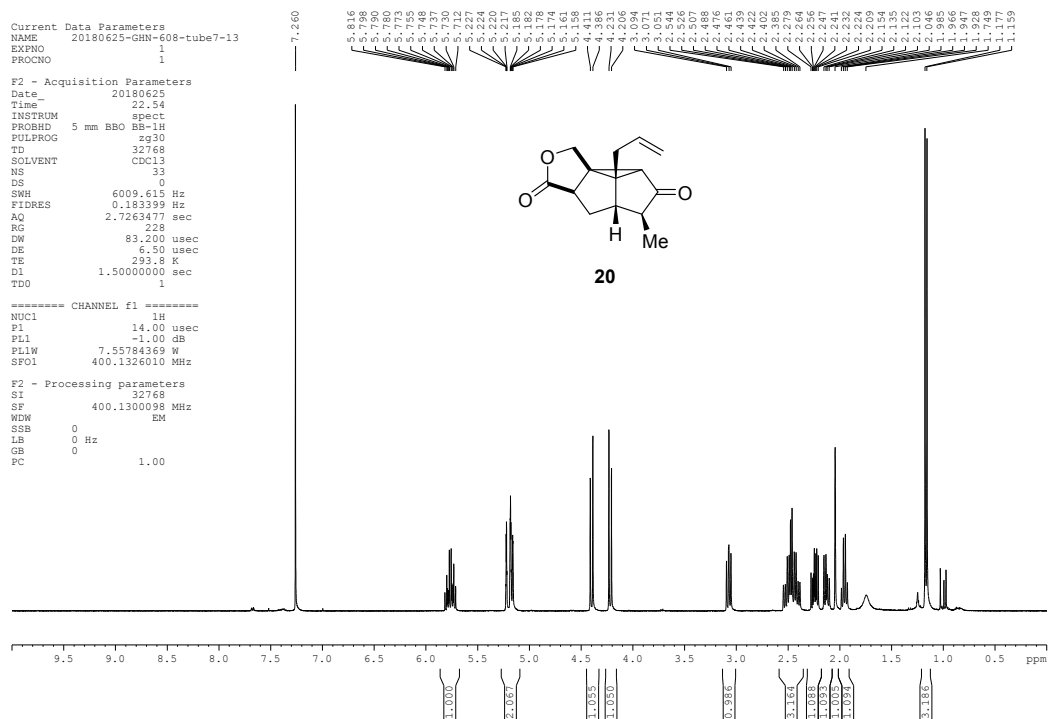
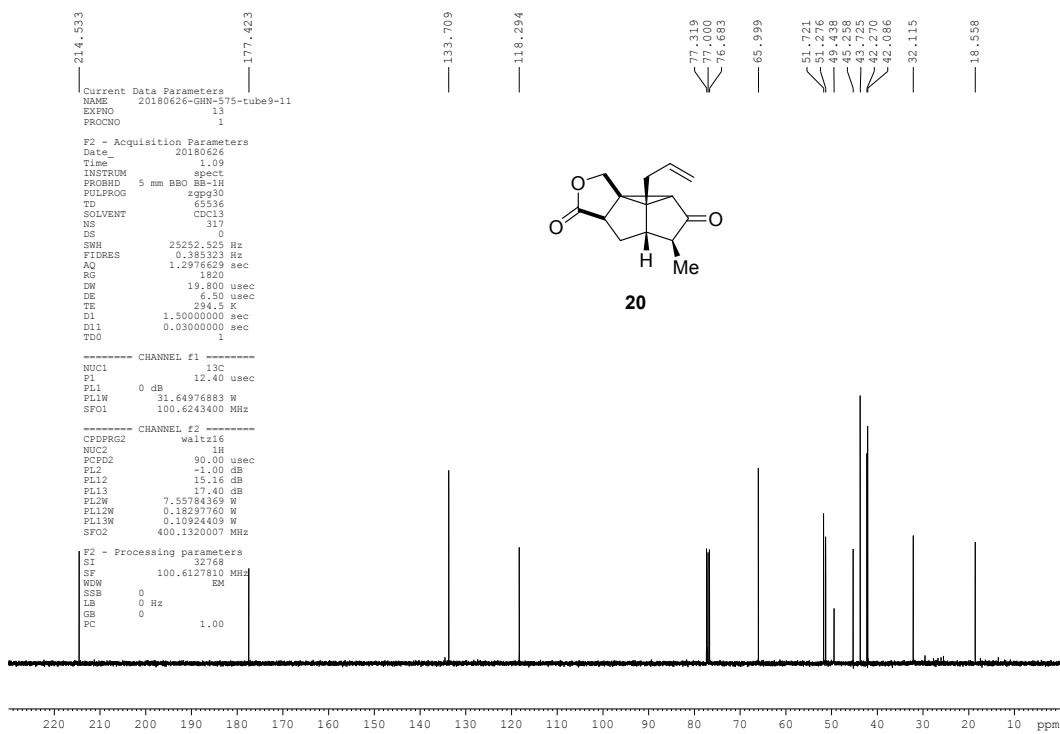
NUC1	1H
P1	14.00 usec
PL1	-1.00 dB
PL1W	7.55784369 W
SFO1	400.1326010 MHz

F2 - Processing parameters

SI	32768
SF	400.1300098 MHz
WDW	EM
SSB	0
LB	0 Hz
GB	0
PC	1.00

19

^{13}C NMR of 19, CDCl_3 , 100 MHz, 24 °C**DEPT 135, DEPT 90 and ^{13}C NMR of 19, CDCl_3 , 100 MHz, 24 °C**

¹H NMR of 20, CDCl₃, 400 MHz, 24 °C**¹³C NMR of 20, CDCl₃, 100 MHz, 24 °C**

Current Data Parameters
NAME 20180626-GHM-575-tube9-11
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180626
Time 1.09
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 317
DS 0
SWH 25252.523 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 1820
DW 19.800 usec
DE 6.50 usec
TE 294.5 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.40 usec
PL1 0 dB
PL1W 31.6497683 W
SFO1 100.6241400 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 19.00 dB
PL12 19.00 dB
PL13 17.40 dB
PL1W 7.55784363 W
PL12W 0.18297760 W
PL13W 0.18924409 W
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127810 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

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118.294
77.319
77.000
76.683
65.999
51.721
51.276
49.488
47.725
43.725
42.270
42.086
32.115
18.558

CC1=C(C(=O)OCC1=O)C=C

20

Current Data Parameters

NAME 20180706-GHN-427-tube23-34
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20180706
Time 3.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 17
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 256
DW 83.200 usec
DE 6.50 usec
TE 293.9 K
D1 1.50000000 sec
TD0 1

===== CHANNEL f1 =====

NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 7.55784369 W
SFO1 400.1326010 MHz

F2 - Processing parameters

SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

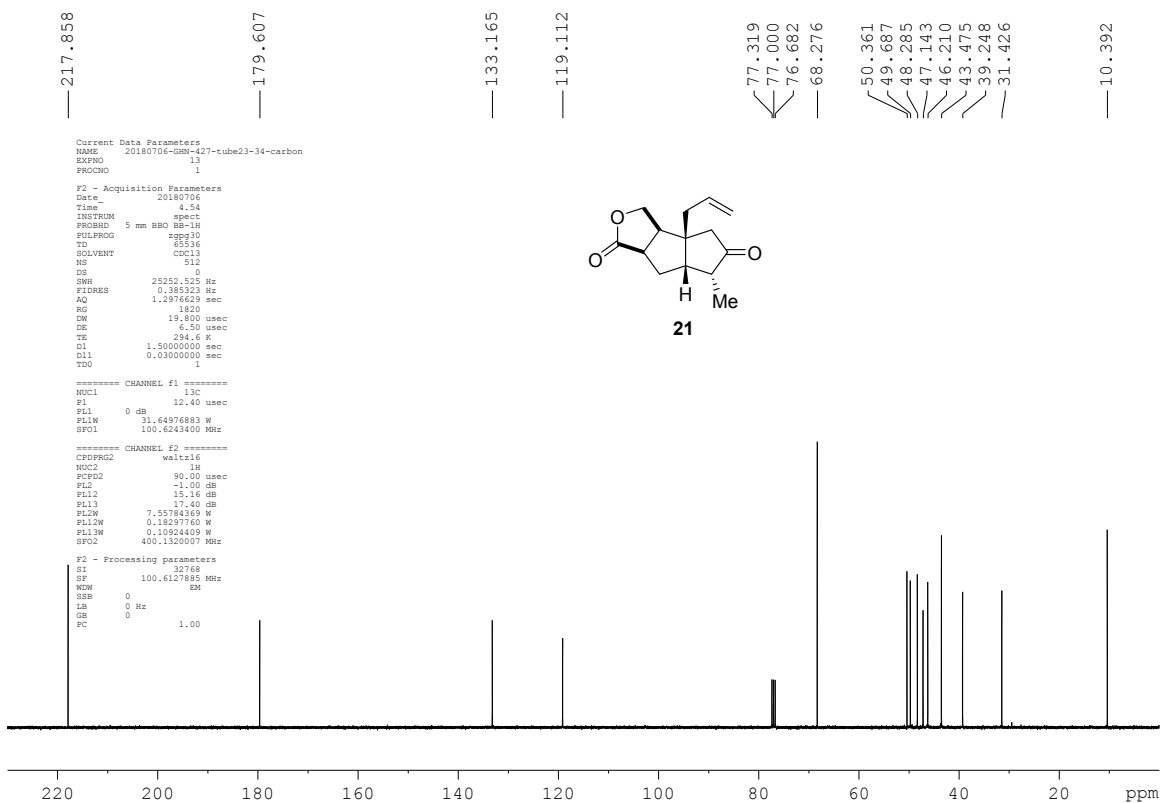
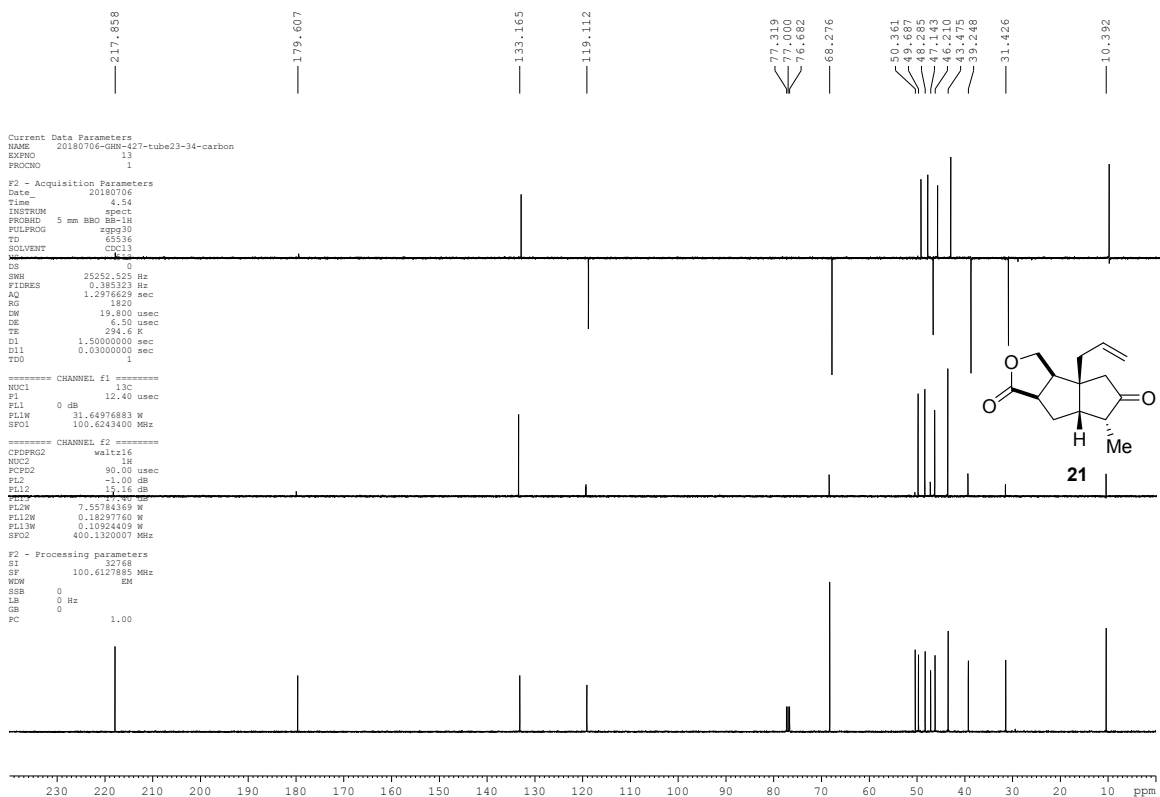
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5.159
5.156
5.152
5.147
5.113
5.110
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4.383
4.374
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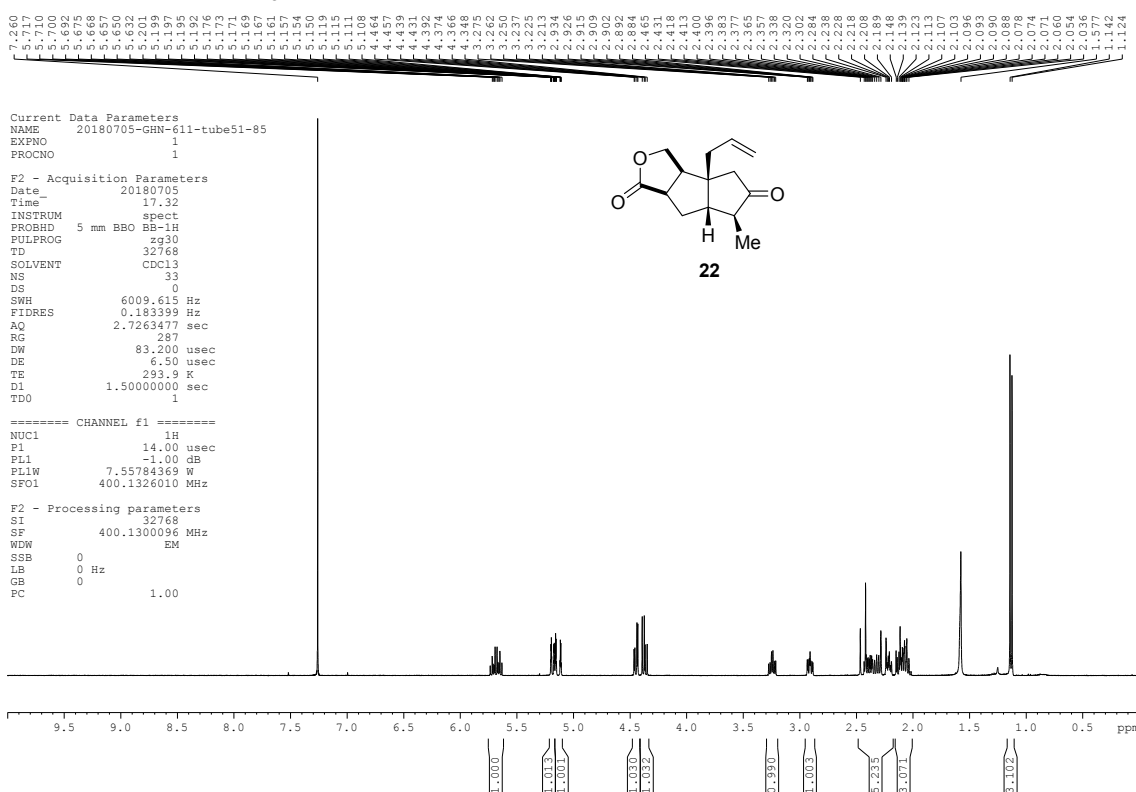
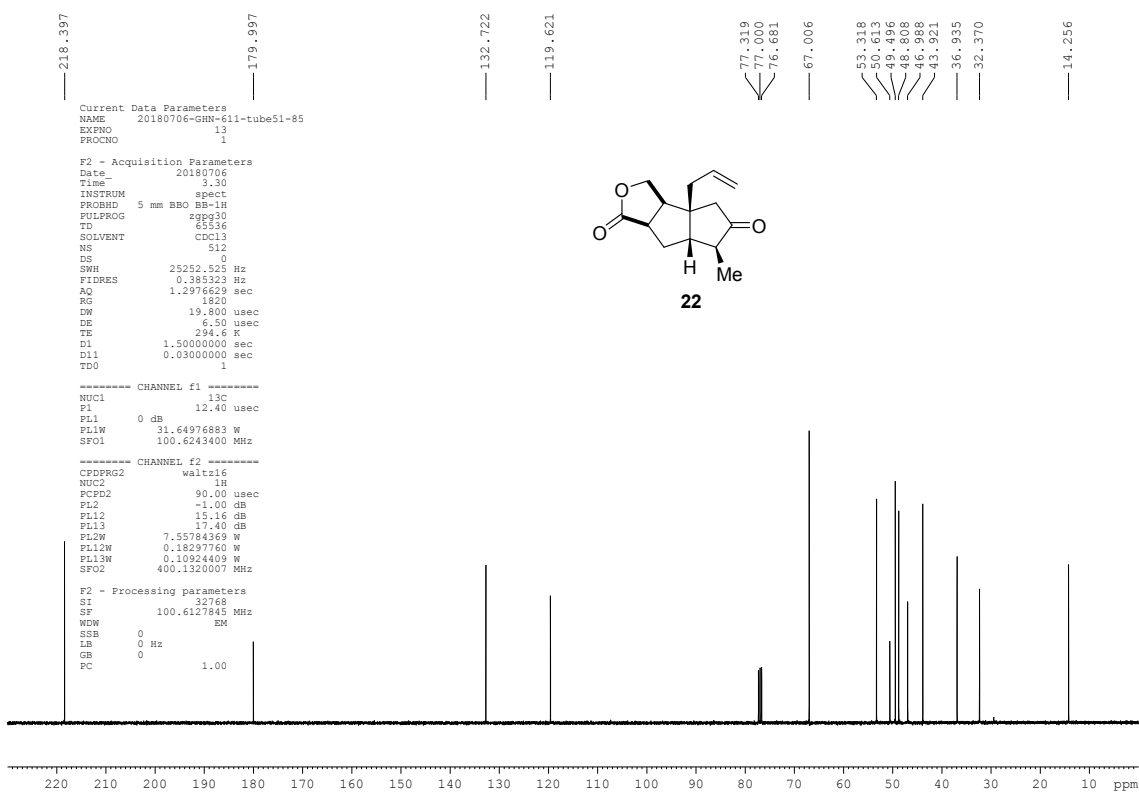
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3.122

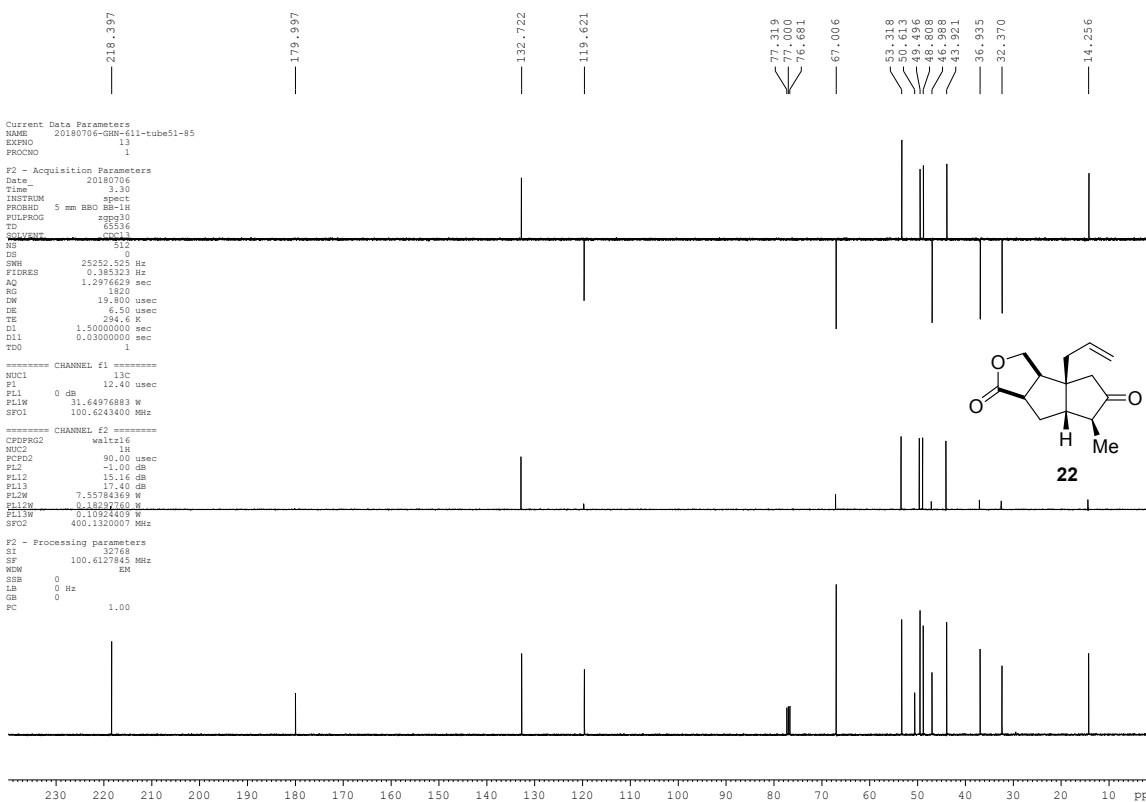
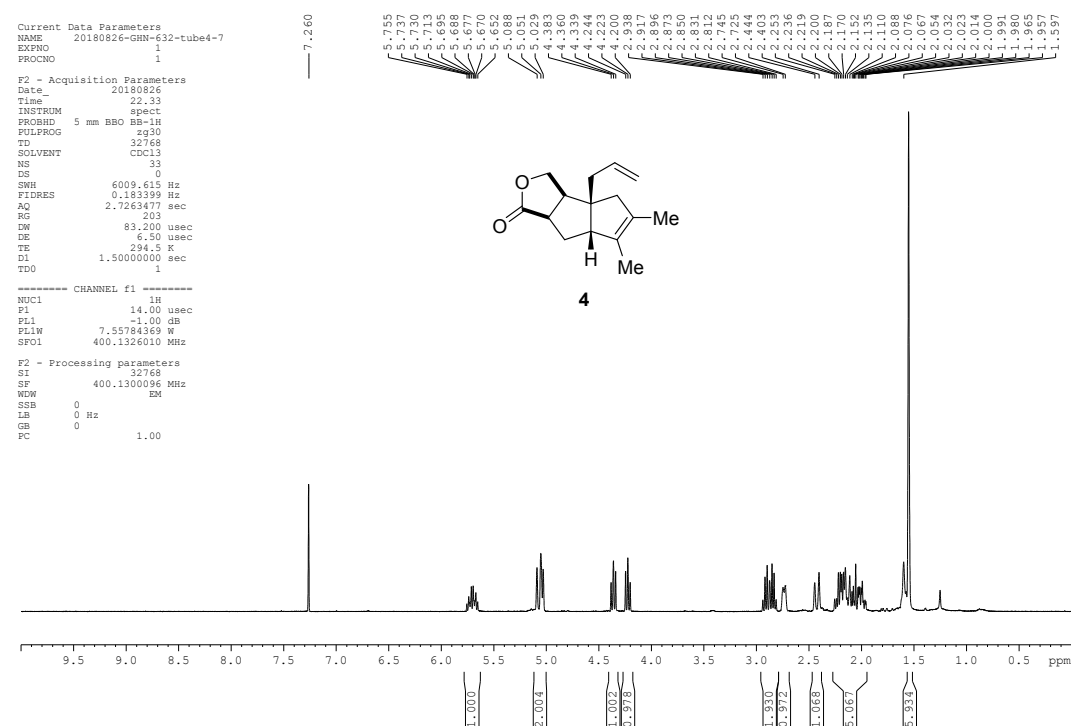
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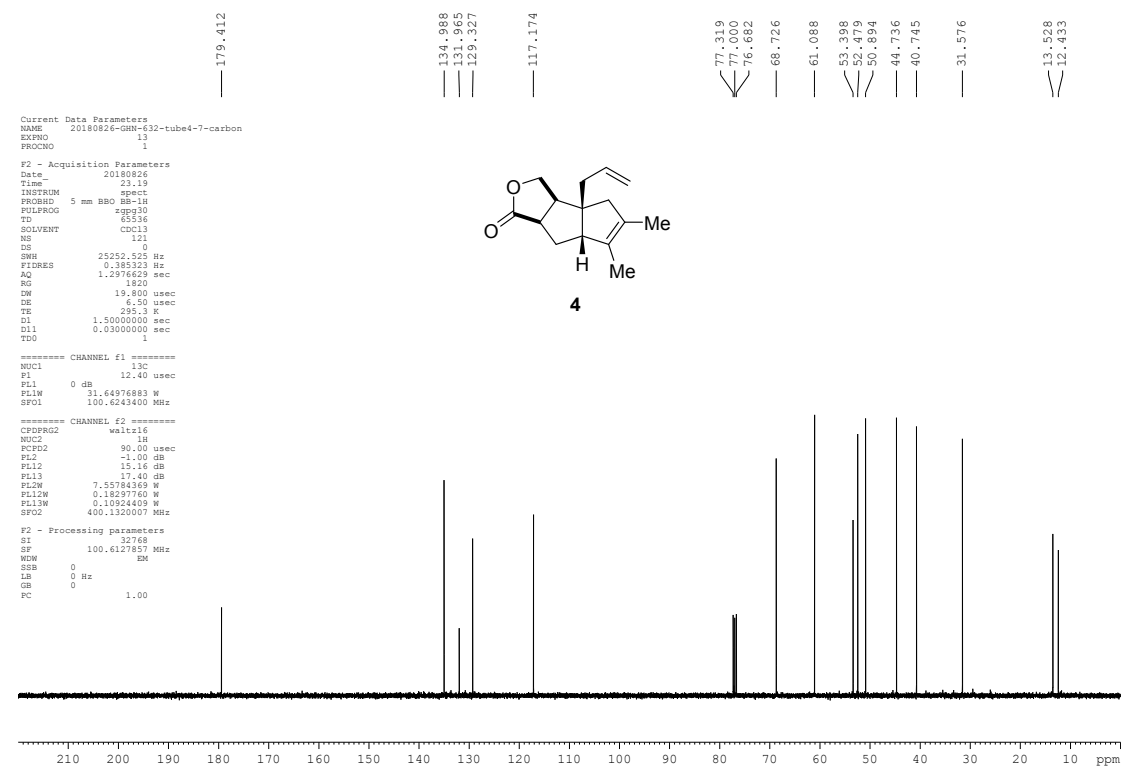
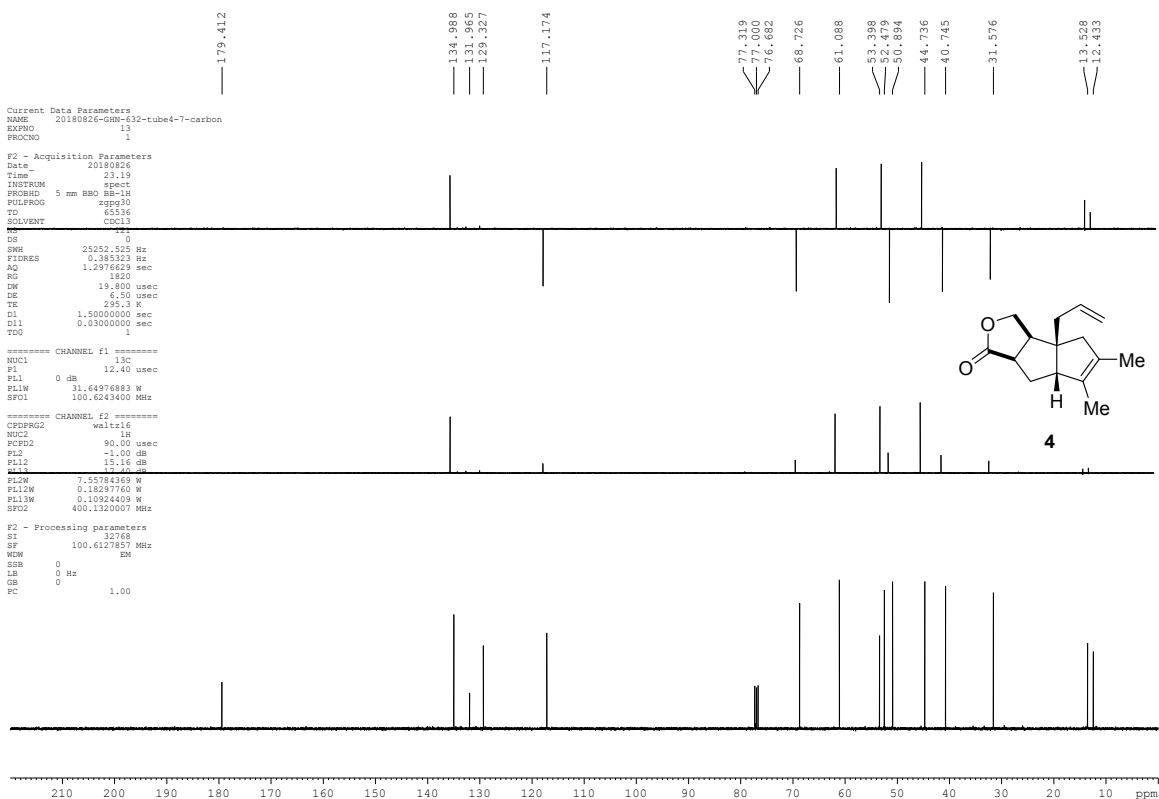
21

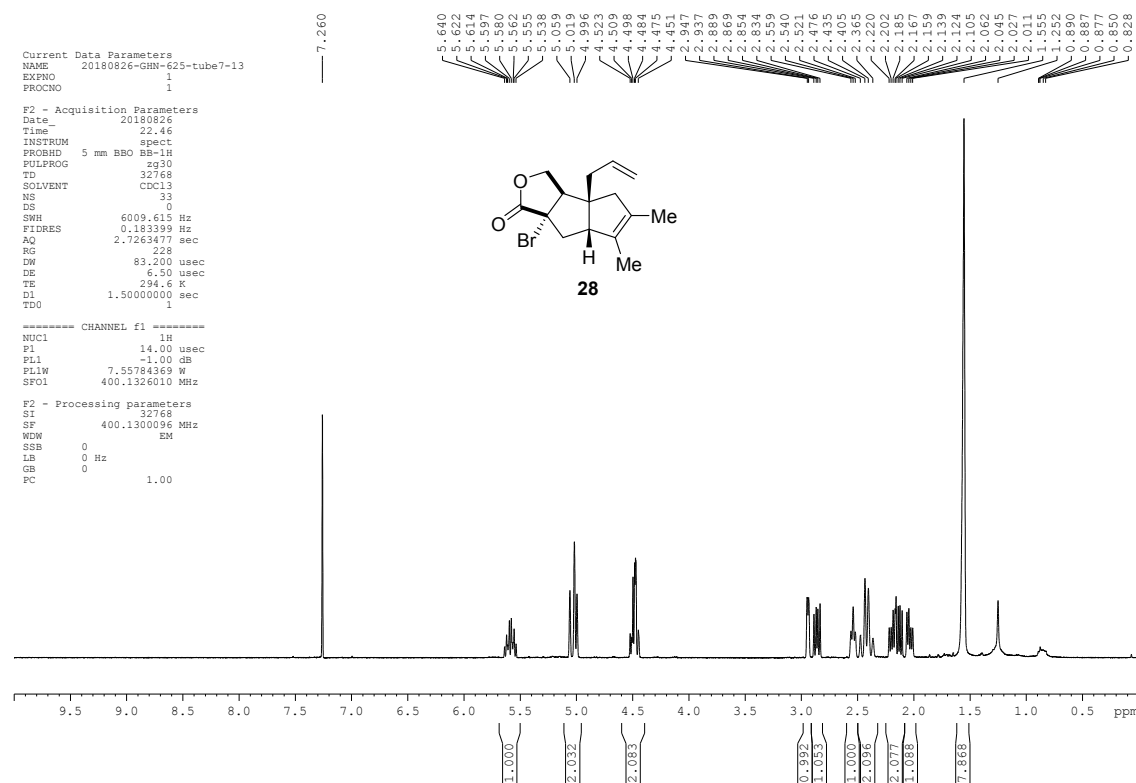
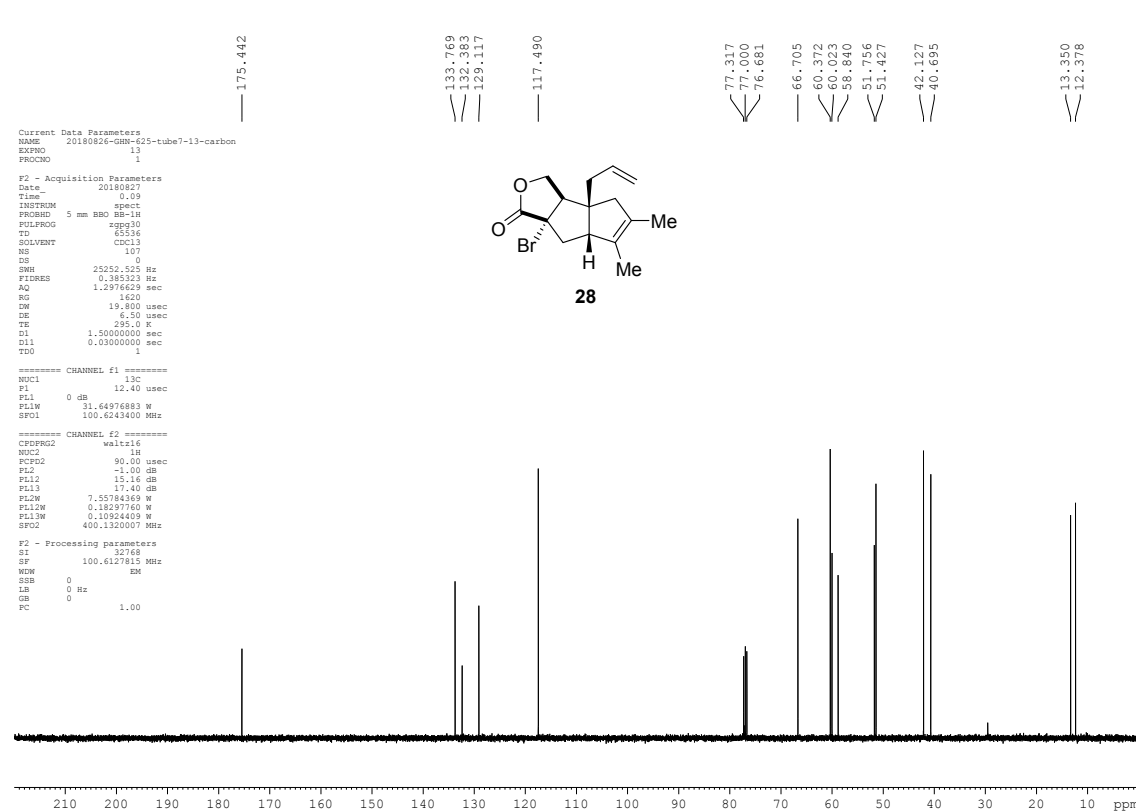
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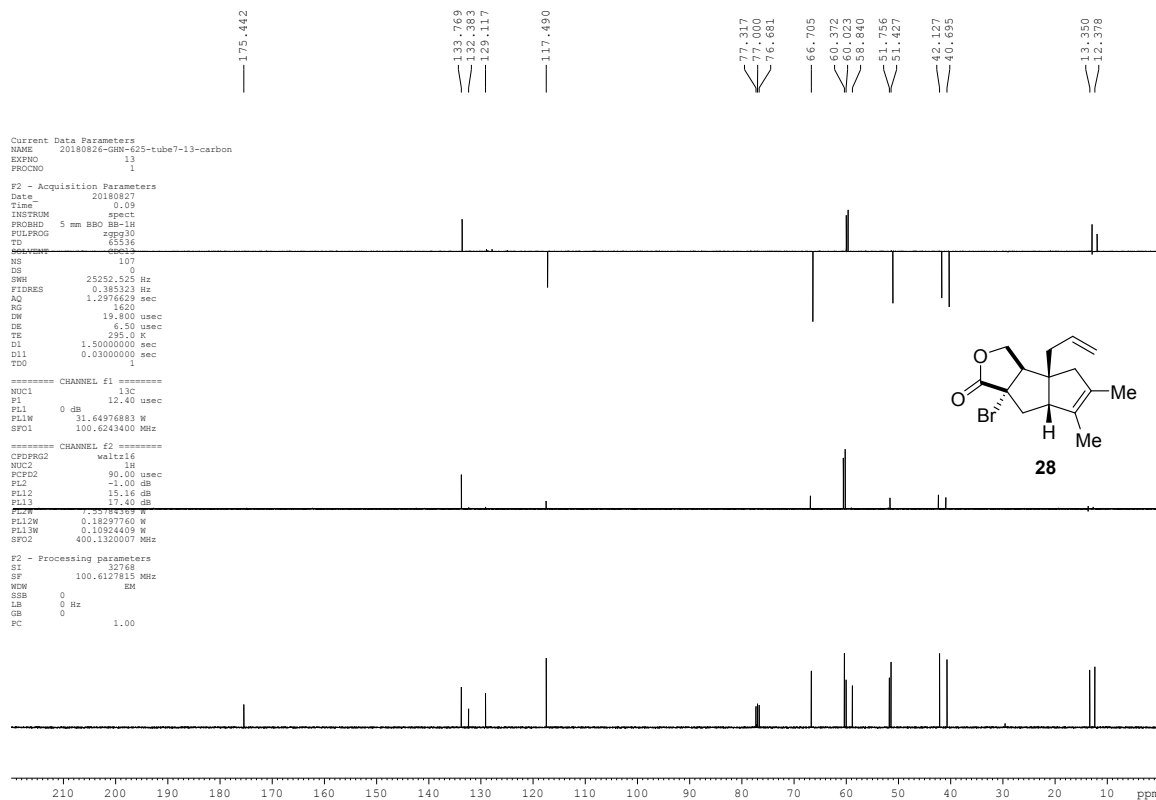
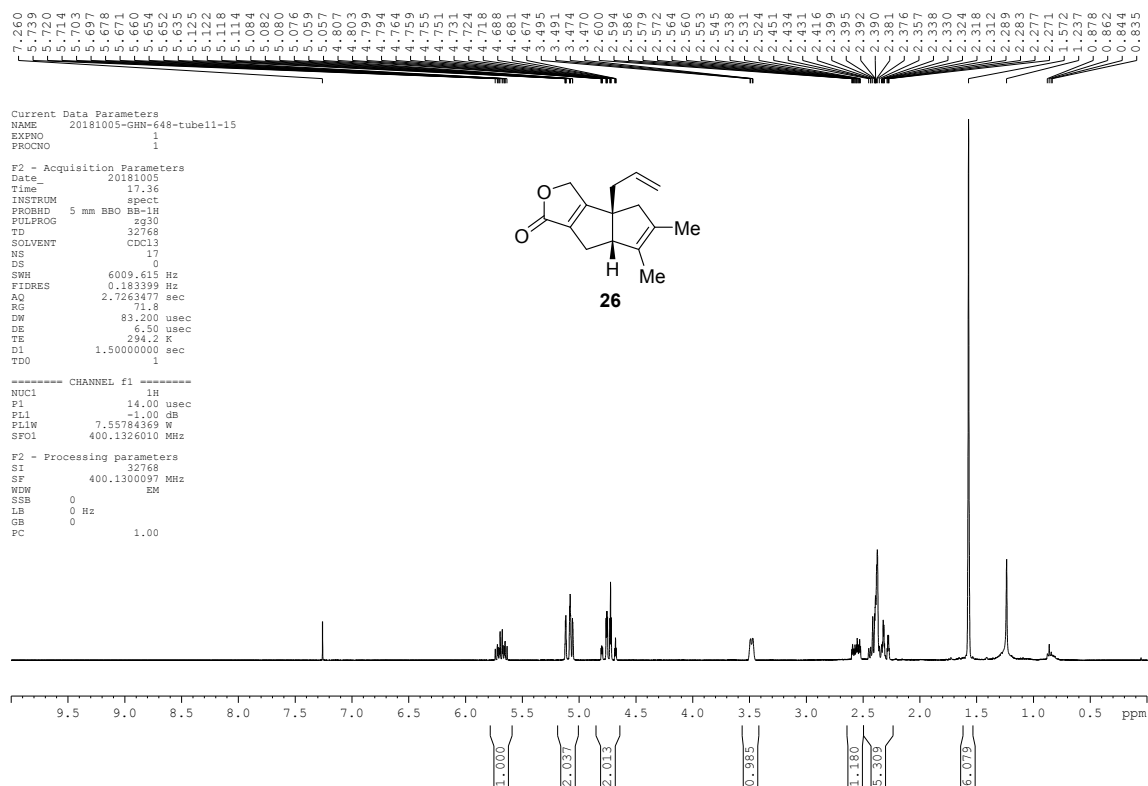
^{13}C NMR of 21, CDCl_3 , 100 MHz, 24 °C**DEPT 135, DEPT 90 and ^{13}C NMR of 21, CDCl_3 , 100 MHz, 24 °C**

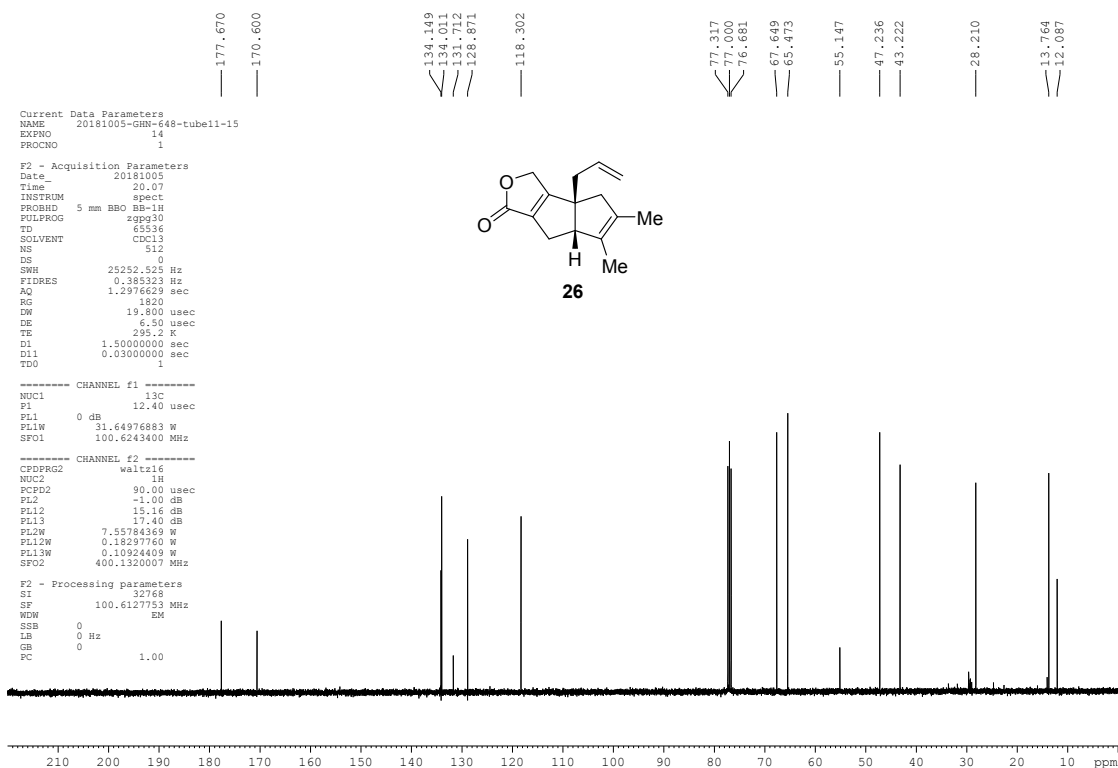
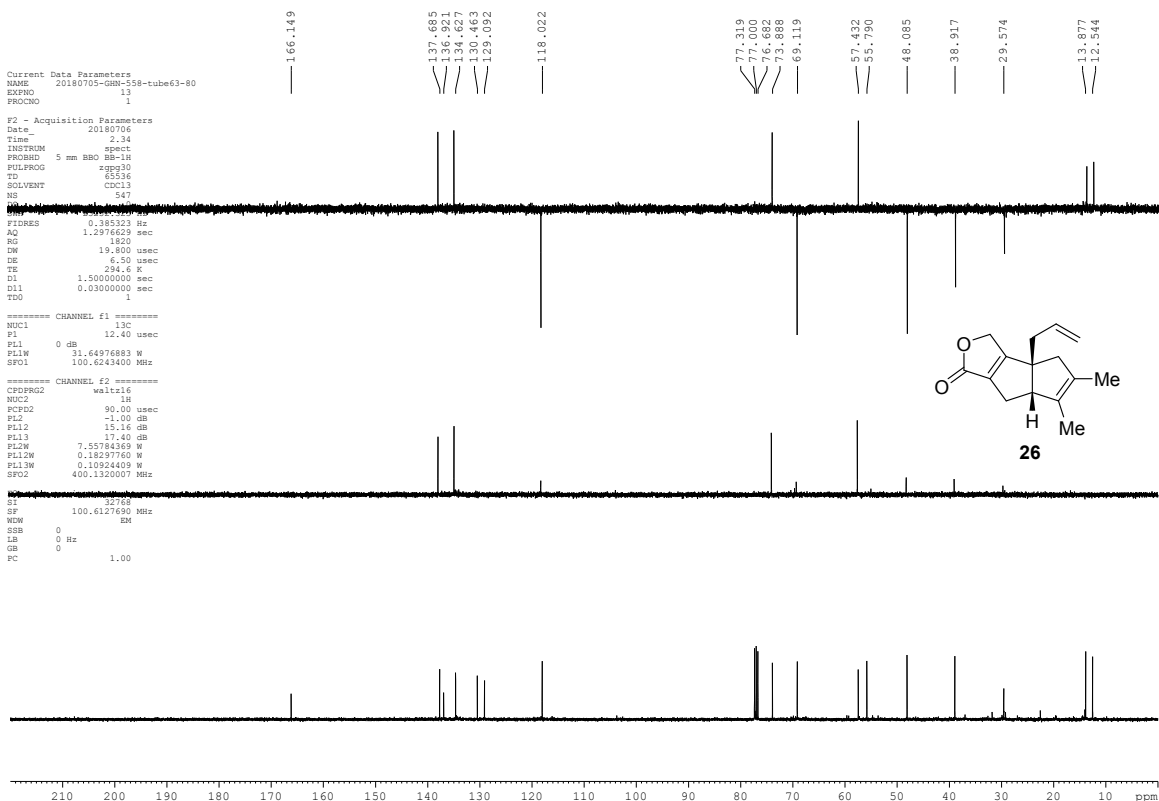
¹H NMR of 22, CDCl₃, 400 MHz, 24 °C**¹³C NMR of 22, CDCl₃, 100 MHz, 24 °C**

DEPT 135, DEPT 90 and ^{13}C NMR of 22, CDCl_3 , 100 MHz, 24 °C ^1H NMR of 4, CDCl_3 , 400 MHz, 24 °C

^{13}C NMR of 4, CDCl_3 , 100 MHz, 24 °C**DEPT 135, DEPT 90 and ^{13}C NMR of 4, CDCl_3 , 100 MHz, 24 °C**

¹H NMR of 28, CDCl₃, 400 MHz, 24 °C**¹³C NMR of 28, CDCl₃, 100 MHz, 24 °C**

DEPT 135, DEPT 90 and ^{13}C NMR of 28, CDCl_3 , 100 MHz, 24 °C ^1H NMR of 26, CDCl_3 , 400 MHz, 24 °C

^{13}C NMR of 26, CDCl_3 , 100 MHz, 24 °C**DEPT 135, DEPT 90 and ^{13}C NMR of 26, CDCl_3 , 100 MHz, 24 °C**

Current Data Parameters

NAME 20180626-GHN-558-tube63-20

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20180626

Time 17.34

INSTRUM spect

PROBHD 5 mm BBO BB-1H

PULPROG zg30

TD 32768

SOLVENT CDCl3

NS 33

DS 0

SWH 6009.615 Hz

FIDRES 0.183399 Hz

AQ 2.7263477 sec

RG 256

DW 83.200 usec

DE 6.50 usec

TE 294.0 K

TD 1.50000000 sec

TD0 1

===== CHANNEL f1 =====

NUC1 1H

P1 14.00 usec

PL1 -1.00 dB

PL12 7.5574369 W

SFO1 400.1326010 MHz

F2 - Processing parameters

SI 32768

SF 400.1300098 MHz

WDW EM

SSB 0

LB 0 Hz

GB 0

PC 1.00

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Current Data Parameters
 NAME 20180705-GHM-558-tube63-80
 EXPRO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180706
 Time_ 2.34
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 547
 DS 0
 SWH 25252.525 Hz
 FIDRES 0.385323 Hz
 AQ 1.2976629 sec
 RG 1820
 DW 19.800 usec
 DE 6.50 usec
 TE 294.6 K
 D1 1.50000000 sec
 D11 0.03000000 sec
 TDO 1

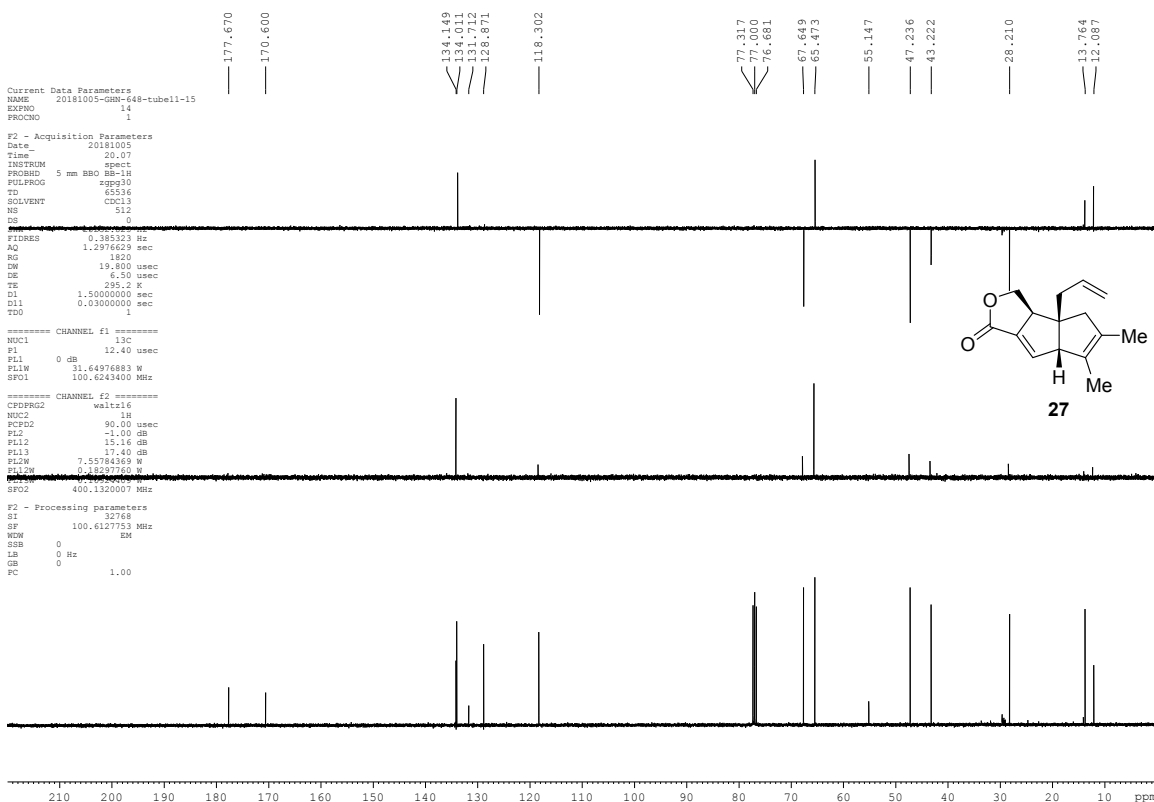
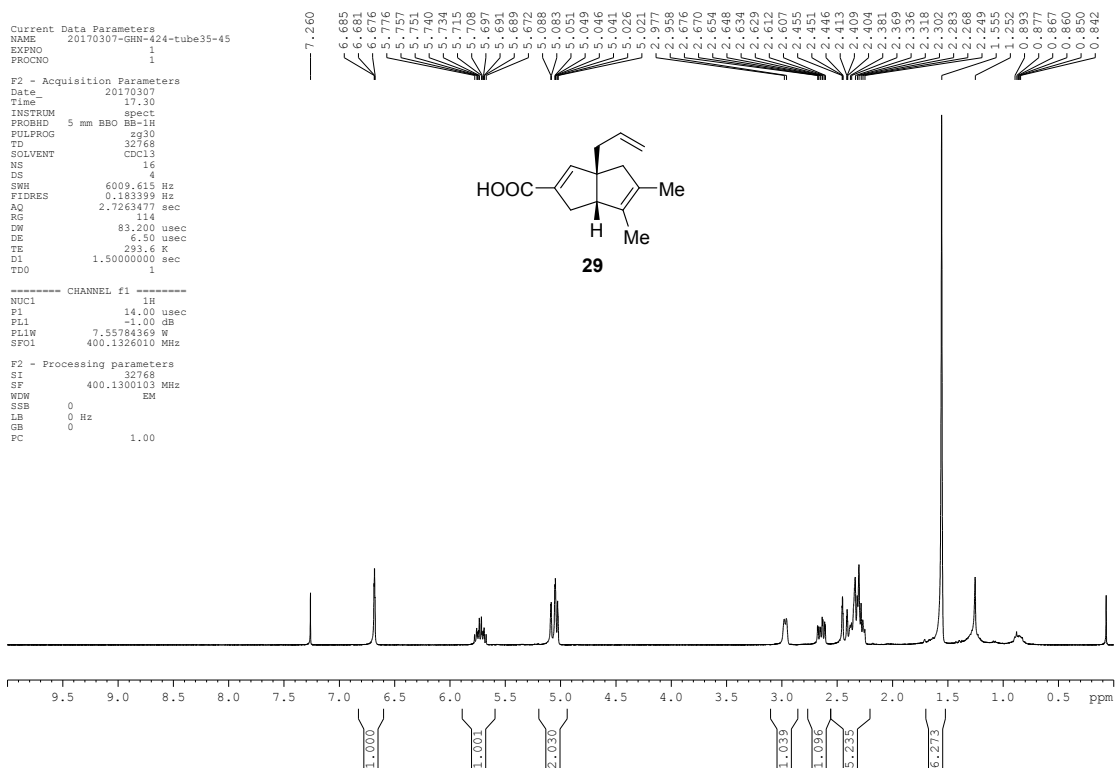
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 P1 12.40 usec
 PL1 0 dB
 PL1W 31.64976883 W
 SFO1 100.6243400 MHz

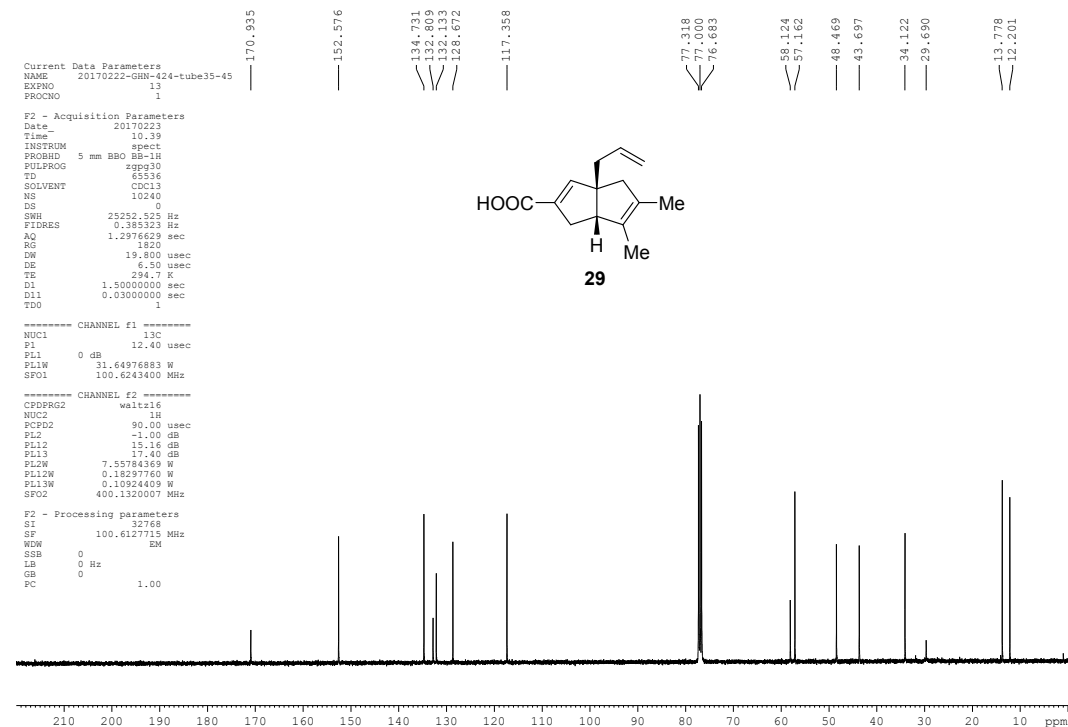
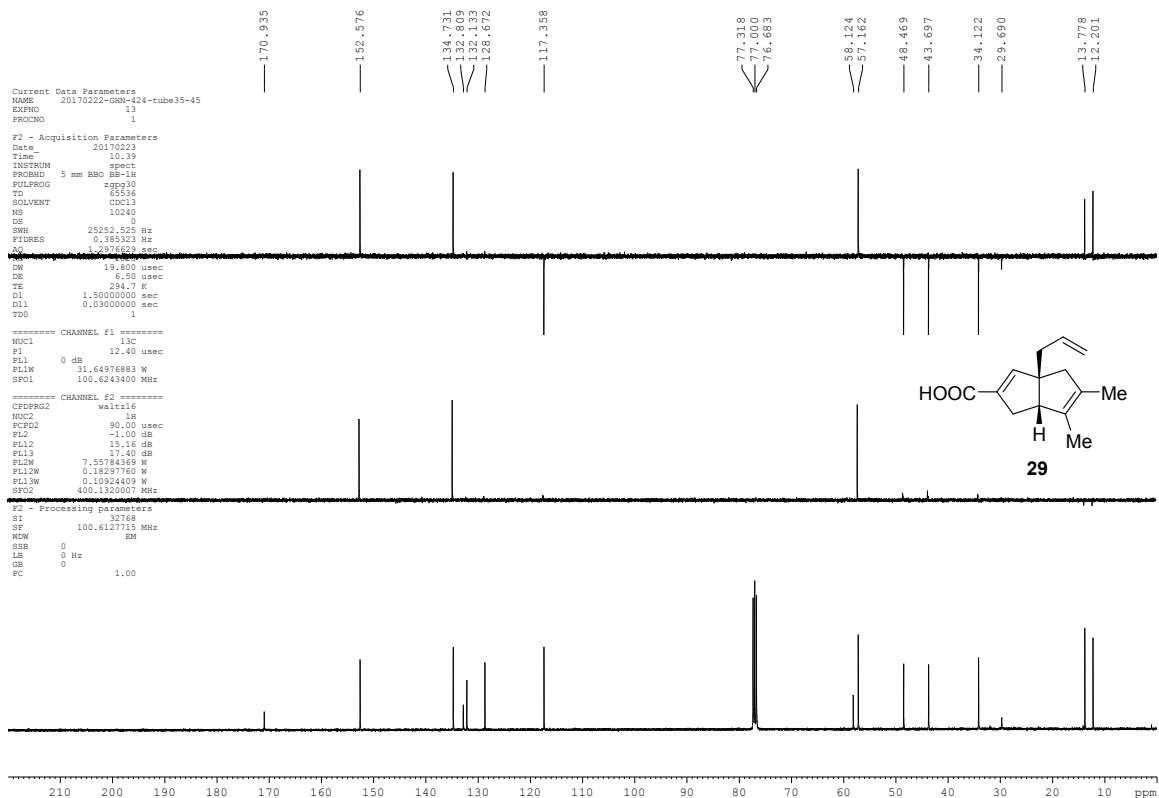
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 15.14 dB
 PL13 17.40 dB
 PL2W 7.55784369 W
 PL12W 0.18297760 W
 PL13W 0.10924409 W
 SFO2 400.1320007 MHz

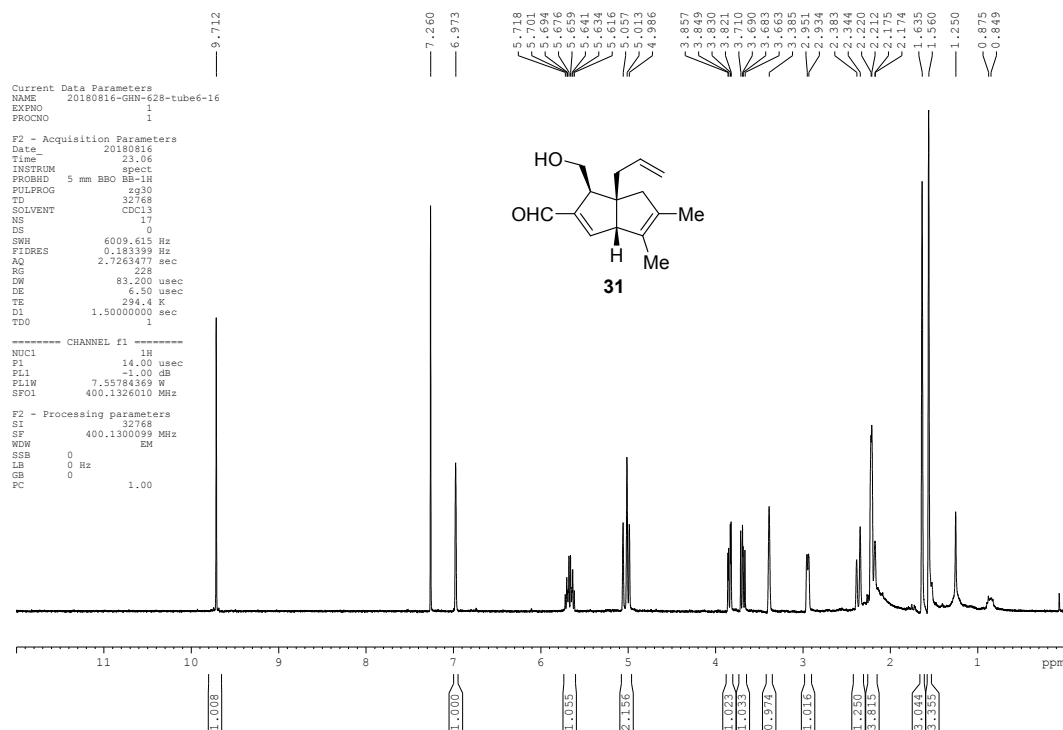
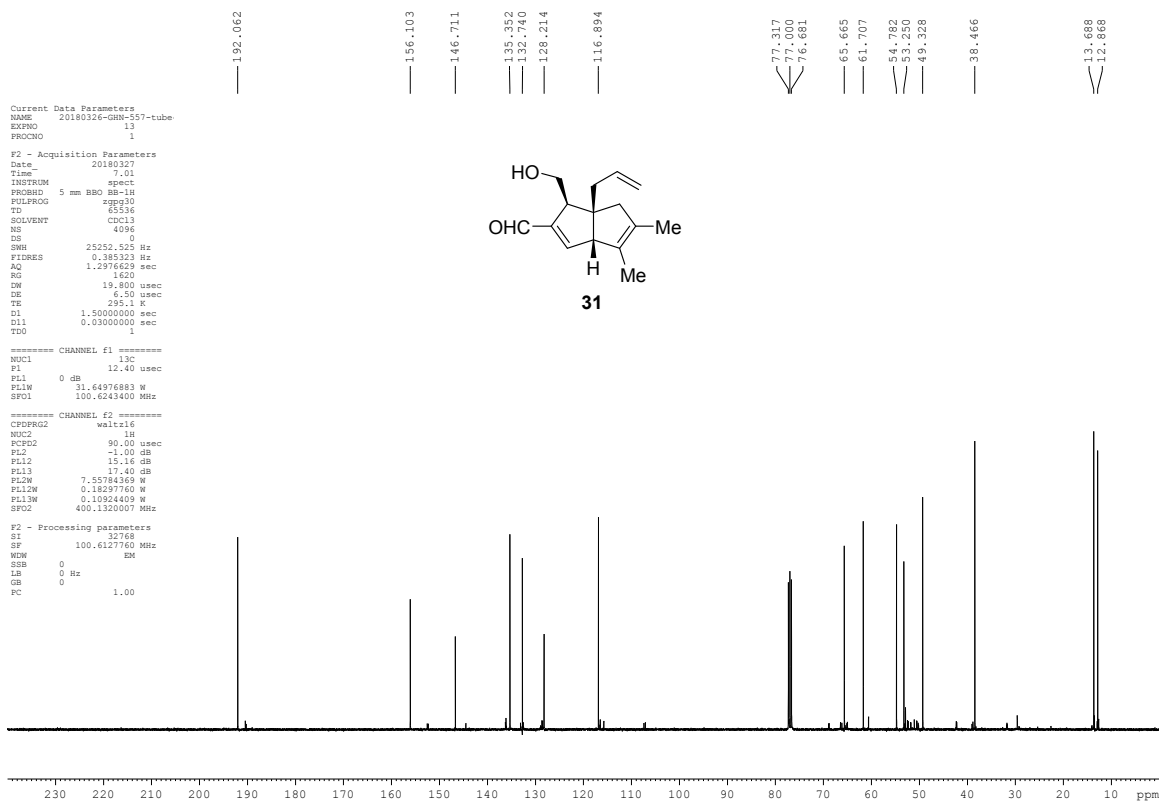
F2 - Processing parameters
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 SF 100.6127690 MHz
 NW 1
 SSB EM
 LB 0 Hz
 GB 0
 PC 1.00

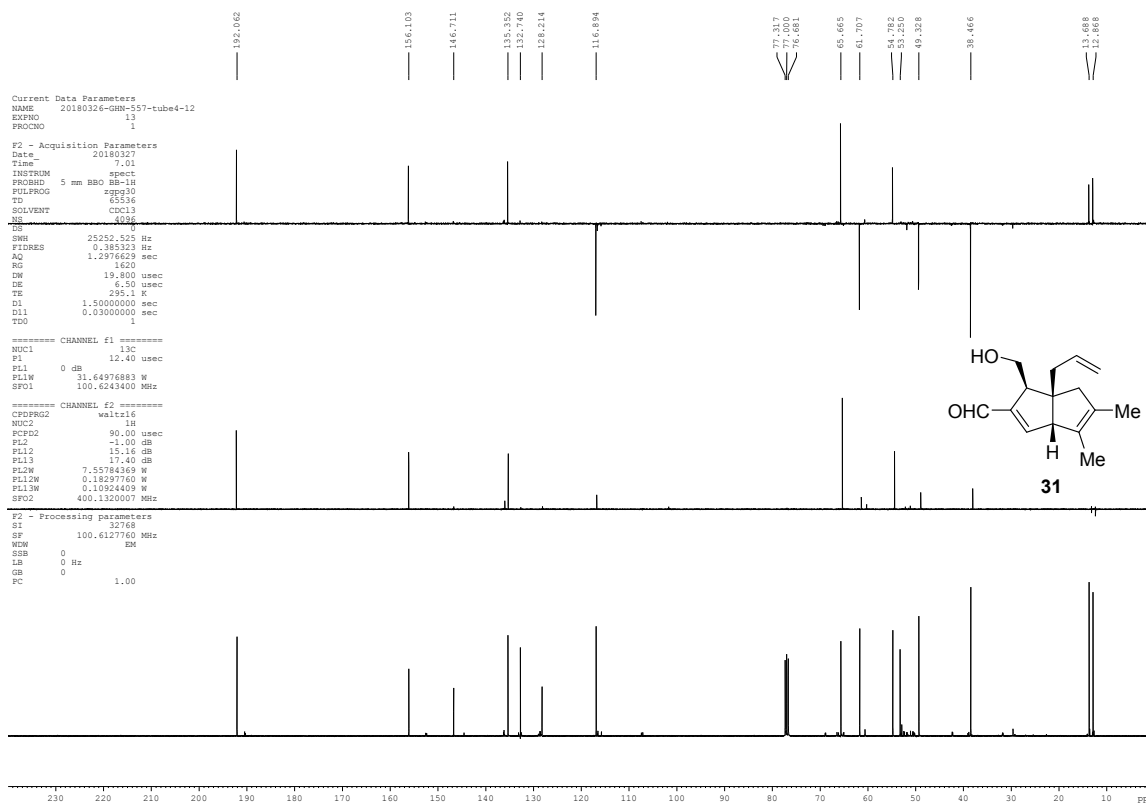
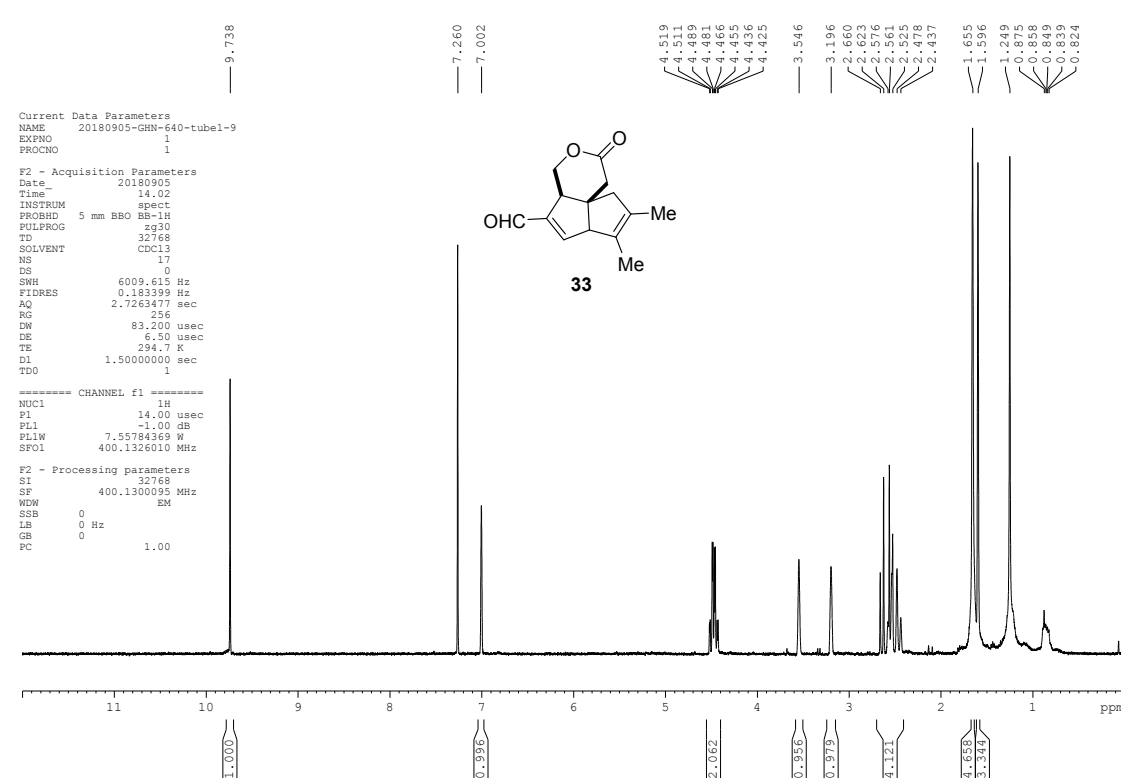
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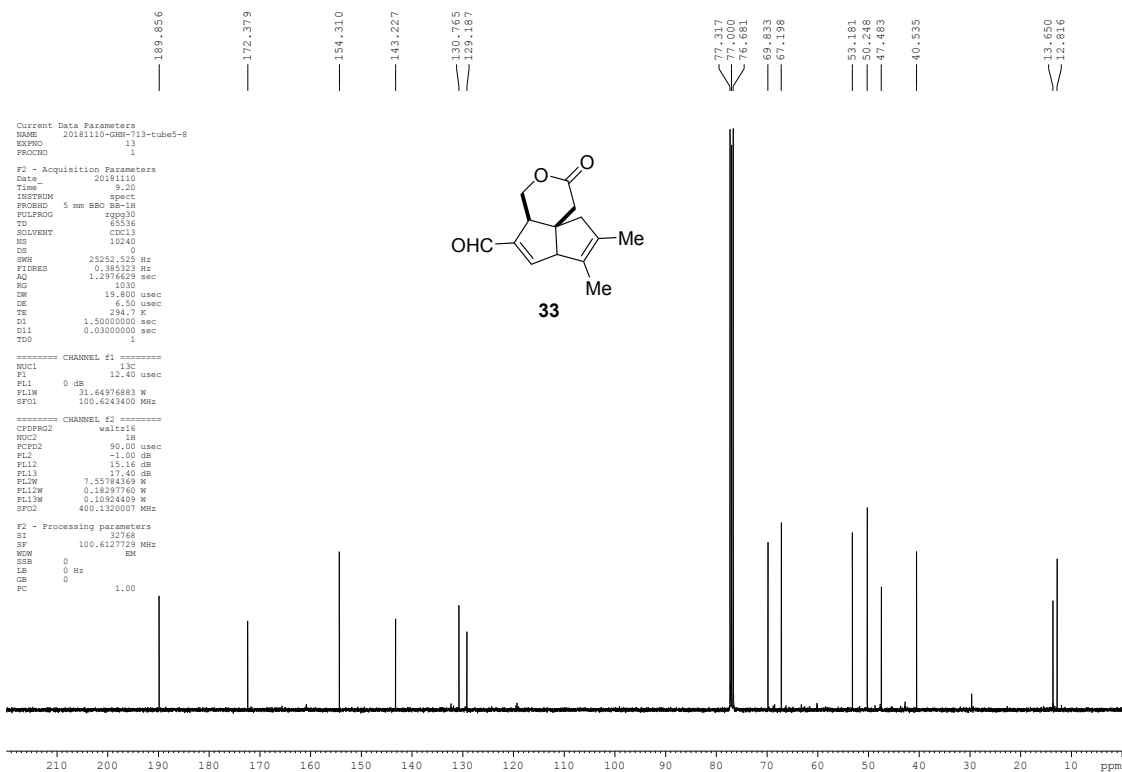
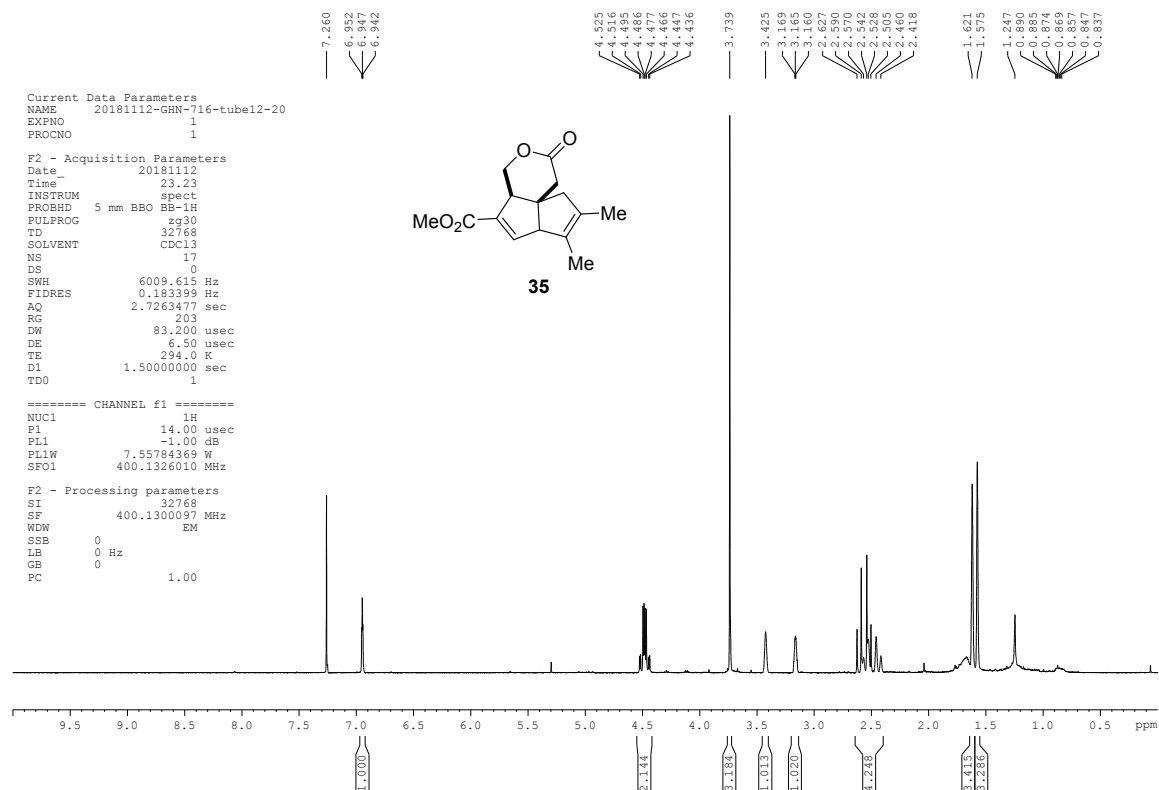
27

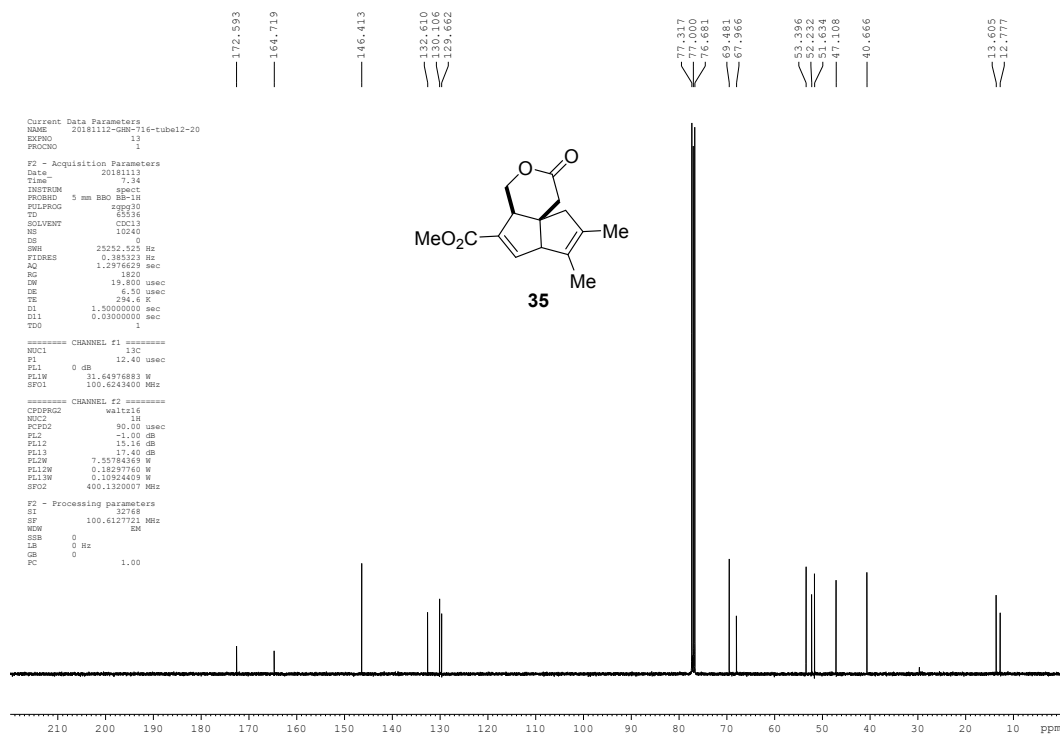
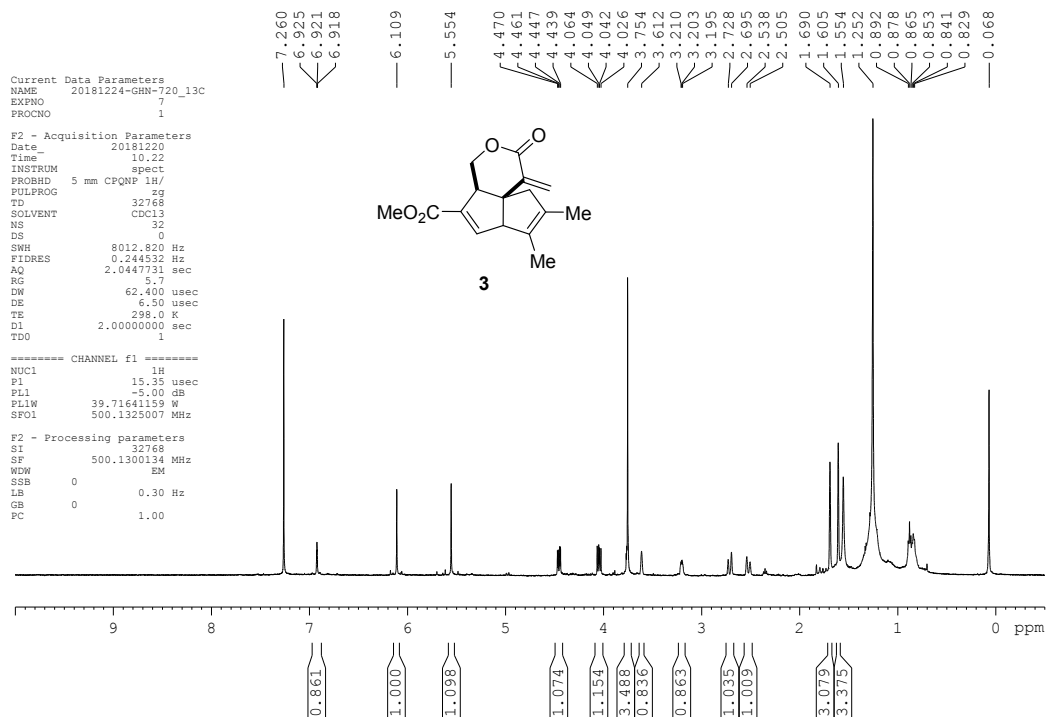
DEPT 135, DEPT 90 and ^{13}C NMR of 27, CDCl_3 , 100 MHz, 24 °C ^1H NMR of 29, CDCl_3 , 400 MHz, 24 °C

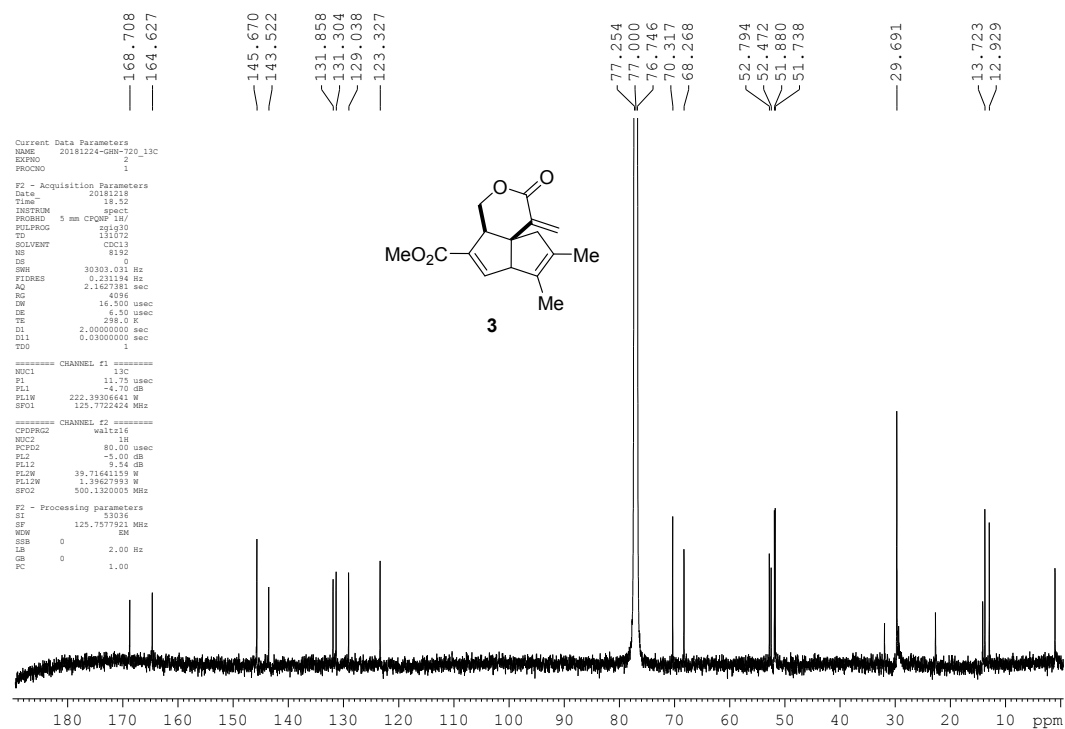
^{13}C NMR of 29, CDCl_3 , 100 MHz, 24 °C**DEPT 135, DEPT 90 and ^{13}C NMR of 29, CDCl_3 , 100 MHz, 24 °C**

¹H NMR of 31, CDCl₃, 400 MHz, 24 °C**¹³C NMR of 31, CDCl₃, 100 MHz, 24 °C**

DEPT 135, DEPT 90 and ^{13}C NMR of 31, CDCl_3 , 100 MHz, 24 °C ^1H NMR of 33, CDCl_3 , 400 MHz, 24 °C

¹³C NMR of 33, CDCl₃, 100 MHz, 24 °C**¹H NMR of 35, CDCl₃, 400 MHz, 24 °C**

¹³C NMR of 35, CDCl₃, 100 MHz, 24 °C**¹H NMR of 3, CDCl₃, 500 MHz, 24 °C**

^{13}C NMR of 3, CDCl_3 , 125 MHz, 24 °C**DEPT 135, DEPT 90 and ^{13}C NMR of 3, CDCl_3 , 100 MHz, 24 °C**