Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Synthesis of tetracyclic chromenones *via* platinum (II) chloride catalysed cascade cyclization of enediyne–enones

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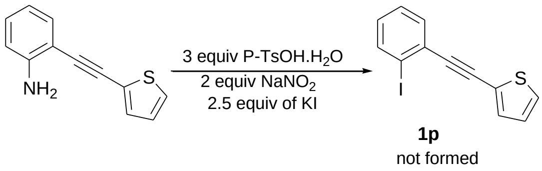
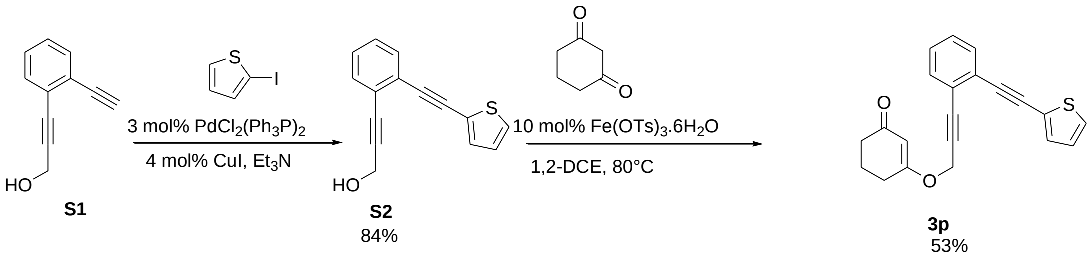
General methods: Purification of crude compounds was done by column chromatography using silica gel (100-200 mesh, Himedia) Melting points were determined in capillary tubes and are uncorrected. FTIR spectra were recorded on Perkin-Elmer spectrometer Spectrum Two and absorbencies are reported in cm^{-1}. NMR spectra were recorded at 500 MHz on JEOL spectrometer and 400 MHz on Brucker spectrometer. The chemical shifts are reported in ppm downfield to TMS (δ = 0) for ^{1}H NMR and relative to the central $CDCl_3$ resonance (δ = 77.0) for ^{13}C NMR. The coupling constants *J* are given in Hz. ESI mass spectra were recorded on LCQ Fleet, Thermo Fisher Instruments Limited, US mass spectrometer. All solvents and commercially available chemicals were used as received.

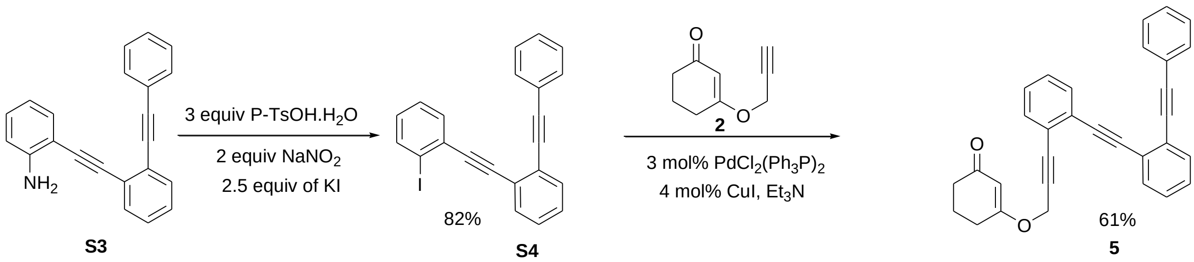




Compound 3p: To synthesis **3p** first we tried to preapare iodo coupling partner **1p**, but iodination of 2-(2-(thiophen-2-yl)ethynyl)benzenamine with P-TsOH.H_2O unfortunately failed so we tried alternative method. To synthesis 3p first we coupled **S1** with 2-iodo thiophene to give **S2**, and followed condensation of **S2** with cyclohexane-1,3-dione in the presence iron(III) tosylate hexahydrate the compound **3p** obtained in moderate yield.

A suspension of 2-iodothiophene (1.0 equiv), $PdCl_2(PPh_3)_2$ (0.03 equiv) and CuI (0.04 equiv) in triethylamine under nitrogen atmosphere. The reaction mixture was stirred at 30°C for 30 minutes. After addition of the **S1**[1] (1.0 equiv) the suspension was stirred for 3 hours at 30°C. The reaction mixture was diluted with water and extracted with EtOAc. The combined organic phases were washed with brine and dried over Na_2SO_4. Removal of the solvent under reduced pressure and purification by column chromatography (PE : EtOAc 8 : 2) furnished the product **S2** as brownish orange viscous fluid. IR (Neat) ν_{max}: 492, 544, 580, 625, 659, 703, 758, 803, 832, 853, 952, 974, 1022, 1035, 1094, 1124, 1161, 1212, 1287, 1357, 1420, 1445, 1477, 1520, 1574, 1593, 1632, 1715, 2203, 2857, 2920, 3060, 3106, 3363 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.06 (brs, 1H), 4.57 (s, 2H), 7.01 (dd, *J* = 5.1, 3.6 Hz, 1H), 7.23 – 7.33 (m, 4H), 7.43 – 7.52 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.8, 84.3, 86.8, 91.8, 92.0, 123.2, 125.0, 125.7, 127.4, 127.8, 128.2, 128.4, 131.4, 132.0, 132.3.

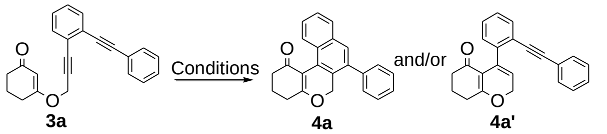
To a stirred solution of the cyclohexane-1,3-dione (1.0 equiv) in 1,2-dichloroethane, the alcohol **S2** (1.2 equiv) and $Fe(OTs)_3.6H_2O$ (0.1 equiv) were added, and the resulting mixture was heated at 80°C under nitrogen atmosphere for 24h. The reaction mixture was diluted with water and extracted with dichloromethane. The combined organic phases were washed with brine and dried over Na_2SO_4. Removal of the solvent under reduced pressure and purification by column chromatography (PE : EtOAc 6 : 4) furnished the desired product **3p** as brownish orange viscous fluid. IR (Neat) ν_{max}: 443, 498, 528, 612, 705, 759, 825, 853, 911, 954, 1000, 1034, 1058, 1094, 1135, 1179, 1213, 1241, 1255, 1327, 1357, 1385, 1421, 1446, 1478, 1520, 1606, 1651, 1718, 1959, 2204, 2871, 2948, 3072 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 1.97 (quint, *J* = 6.3 Hz, 2H), 2.30 – 2.38 (m, 2H), 2.45 (t, *J* = 6.3 Hz, 2H), 4.85 (s, 2H), 5.54 (s, 1H), 7.03 (dd, *J* = 5.1, 3.7 Hz, 1H), 7.25 – 7.36 (m, 4H), 7.46 – 7.52 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.2, 28.9, 36.8, 57.1, 86.0, 86.8, 87.0, 91.6, 103.7, 123.1, 124.2, 125.9, 127.3, 127.9, 128.2, 128.9, 131.6, 132.3, 132.4, 176.8, 199.7; MS (ESI+): *m/z* = 355 $[M+Na^+]$



Compound 5: To a solution of *p*-TsOH·H_2O (3 equiv) in MeCN was added the **S3**[2] (1 equiv). The resulting suspension of amine salt was cooled to 10–15 °C and to this was added, gradually, a solution of $NaNO_2$ (2 equiv) and KI (2.5 equiv) in H_2O. The reaction mixture was stirred for 10 min then allowed to come to 20 °C and stirred for 2h. To the reaction mixture was then added saturated $NaHCO_3$ (until pH = 9–10) and saturated $Na_2S_2O_3$ solution. Then extracted with EtOAc and dried over Na_2SO_4. The crude was purified by column chromatography (Pet.ether – EtOAc, 9:1) to afford **S4**. The products **S4** is spectrally identical to previously reported sample.[3]

A suspension of iodo compound **S4** (1.0 equiv), $PdCl_2(PPh_3)_2$ (0.03 equiv) and CuI (0.04 equiv) in triethylamine under nitrogen atmosphere. The reaction mixture was stirred at 30°C for 30 minutes. After the addition of **2**(1.5 equiv) the suspension was stirred for 3 hours at 30°C. The reaction mixture was filtered through celite pad and purification by column chromatography (PE : EtOAc 6 : 4) the desired compound was isolated as a brownish orange viscous fluid. IR (Neat) ν_{max}: 494, 585, 611, 690, 756, 823, 860, 954, 999, 1135, 1178, 1212, 1240, 1327, 1356, 1384, 1428, 1446, 1494, 1606, 1654, 2216, 2948, 3056 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 1.91 (quint, *J* = 6.1 Hz, 2H), 2.27 – 2.35 (m, 4H), 4.60 (s, 2H), 5.38 (s, 1H), 7.28 – 7.34 (m, 7H), 7.50 (d, *J* = 7.0 Hz, 1H), 7.55 – 7.61 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.2, 28.8, 36.8, 57.1, 86.1, 86.8, 88.4, 91.8, 92.4, 93.7, 103.6, 123.3, 124.4, 125.7, 125.9, 126.2, 128.1, 128.3, 128.4, 128.5, 128.6, 128.8, 131.9, 132.0, 132.2, 132.3, 132.4, 176.7, 199.6.

Table 1 Optimization of reaction conditions for **4a**

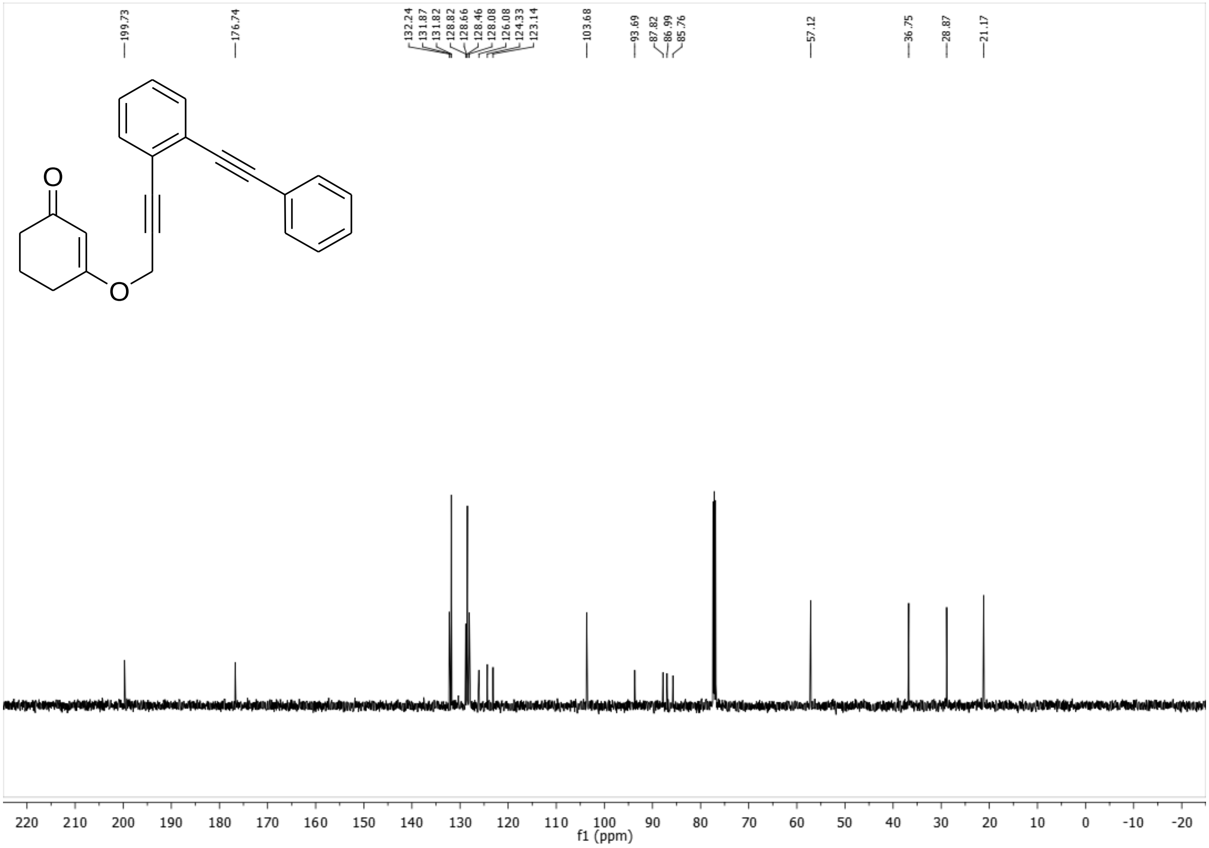


Entry	Catalyst, Additives(equiv)	Solvent	Conditions	Yield[a] of **4a/4a'**(%)
1	$PdCl_2$(0.1), $CuCl_2$(2)	THF	80°C, 1 h	-[b]
2	$PdCl_2$(0.1), $CuBr_2$(2)	THF	80°C, 1 h	-[b]
3	$CuBr_2$(2)	THF	80°C, 2 h	-[c]
4	$PdCl_2$ (0.1), LiBr(2)	CH_3CN	r.t, 5 h	-[b]
5	$Pd(OAc)_2$ (0.1), LiBr(2)	CH_3CN	r.t, 5 h	-[b]
6	$Pd(OAc)_2$ (0.1), PhI(OAc)(1.5), NaOAc(1.5)	CH_3CN	r.t, 5 h	-[b]
7	$FeCl_3$(10)	CH_3CN	80°C, 6 h	-[c]
8	$In(OTf)_3$(0.1)	Toluene	110°C, 2 h	-[c]
9	$PtCl_2$(0.05)	CH_3CN	80°C, 8 h	52(**4a'/4a** = 1:2)
10	$PtCl_2$(0.05)	CH_3CN	80°C, 24 h	43(**4a**)
11	$PtCl_2$(0.05)	Toluene	110°C, 3 h	86(**4a**)
12	$PtCl_2$(0.02)	Toluene	110°C, 3 h	88**(4a)**
13	$PtCl_4$(0.02)	Toluene	110°C, 3 h	72(**4a**)
14	$Pt(Ph_3P)_4$(0.05)	Toluene	110°C, 24 h	-
15	$(Ph_3P)AuCl$(0.05)	1,2-DCE	80°C, 24 h	-
16	$(Ph_3P)AuCl$(0.05)	Toluene	110°C, 24 h	-
17	$AuCl_3$(0.05)	1,2-DCE	80°C, 24 h	30(**4a'**)[d]
18	$AuCl_3$(0.05)	Toluene	110°C, 24 h	30(**4a'**)[d]
19	$(Ph_3P)AuCl$(0.05), $AgBF_4$(0.1)	1,2-DCE	80°C, 4 h	27(**4a**)
20	$(Ph_3P)AuCl$(0.05), $AgBF_4$(0.1)	Toluene	110°C, 4 h	32(**4a**)

[a] Isolated yield. [b] Complex reaction mixture. [c] Ether link cleavage lead to 3-(2-(2-phenylethynyl)phenyl)prop-2-yn-1-ol. [d] 50% of 3a is recovered.

References

1) D. Aburano, F. Inagaki, S. Tomonaga and C. Mukai, *J. Org. Chem.*, 2009, **74**, 5590.
2) K. Hirano, Y. Inaba, N. Takahashi, M. Shimano, S. Oishi, N. Fujii and H. Ohno, *J. Org. Chem.*, 2011, **76**, 1212
3) A. Orita, N. Yoshioka, P. Struwe, A. Braier, A. Beckmann and J. Otera, *Chem. Eur. J.*, 1999, **5**, 1355.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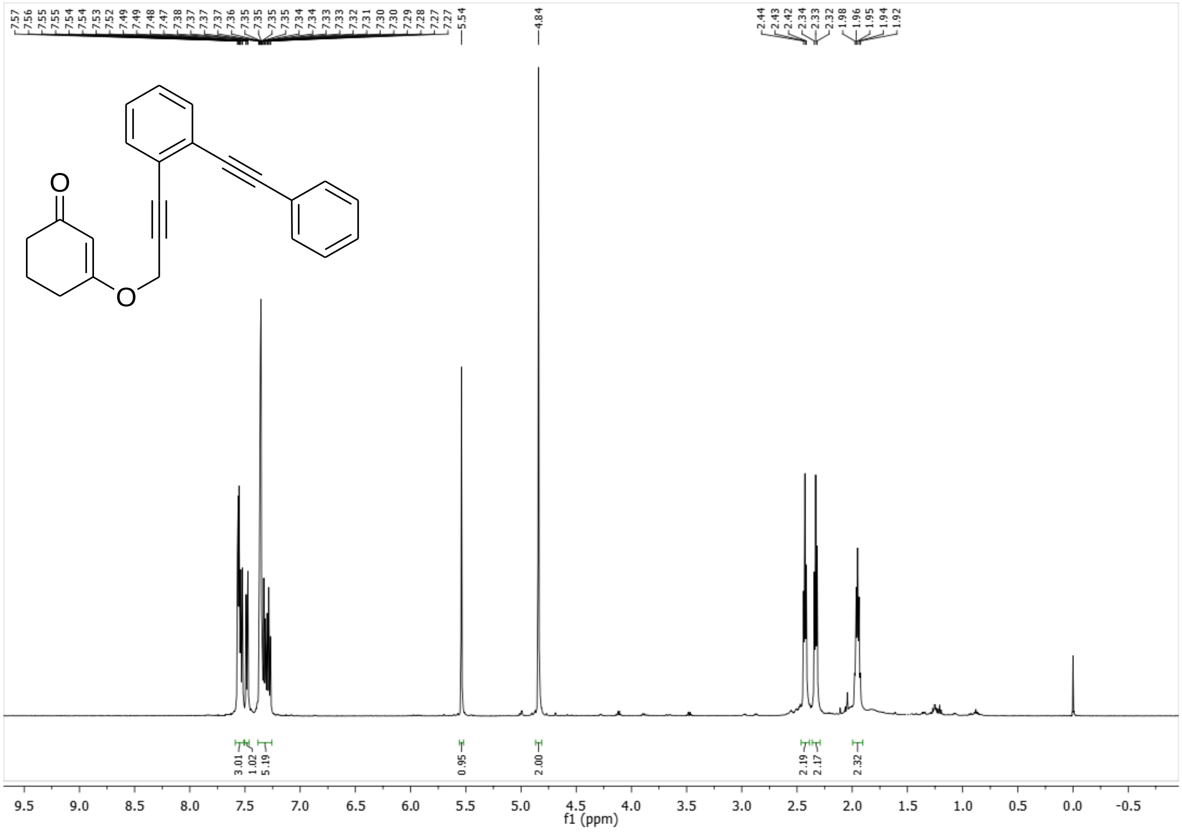
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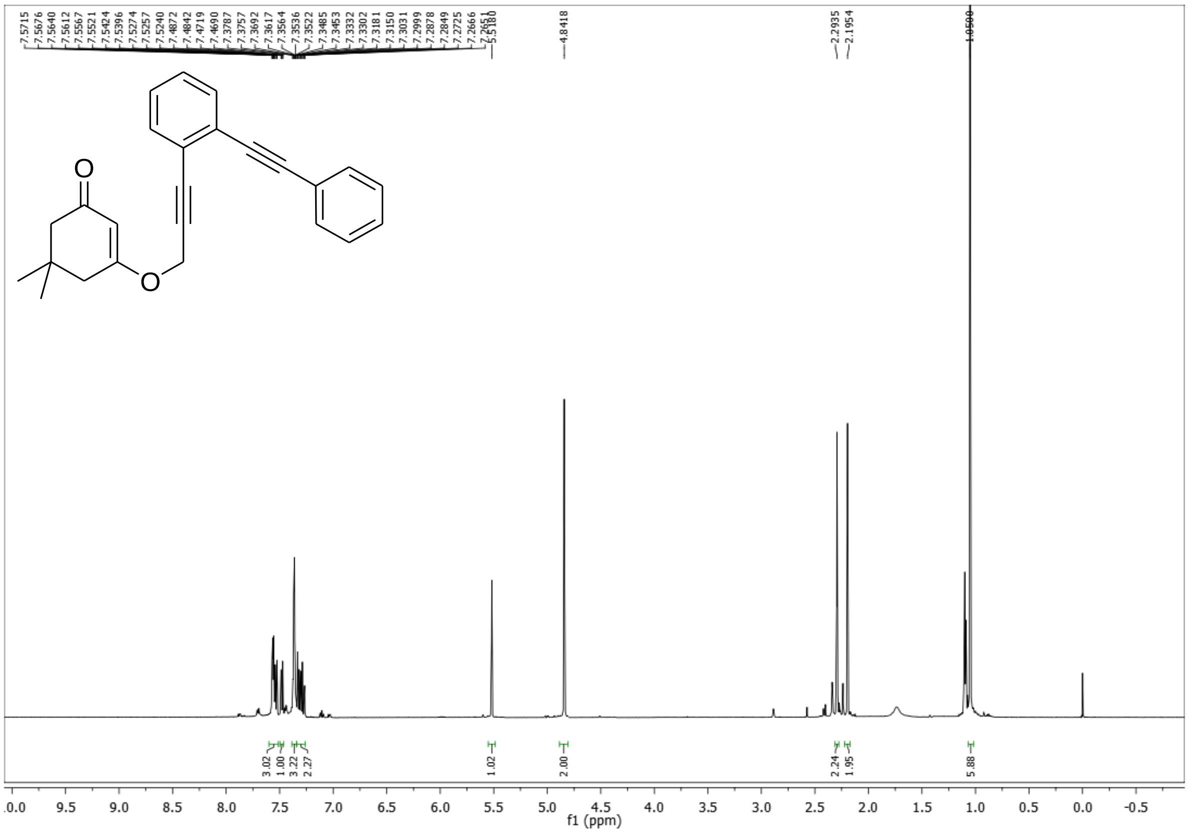

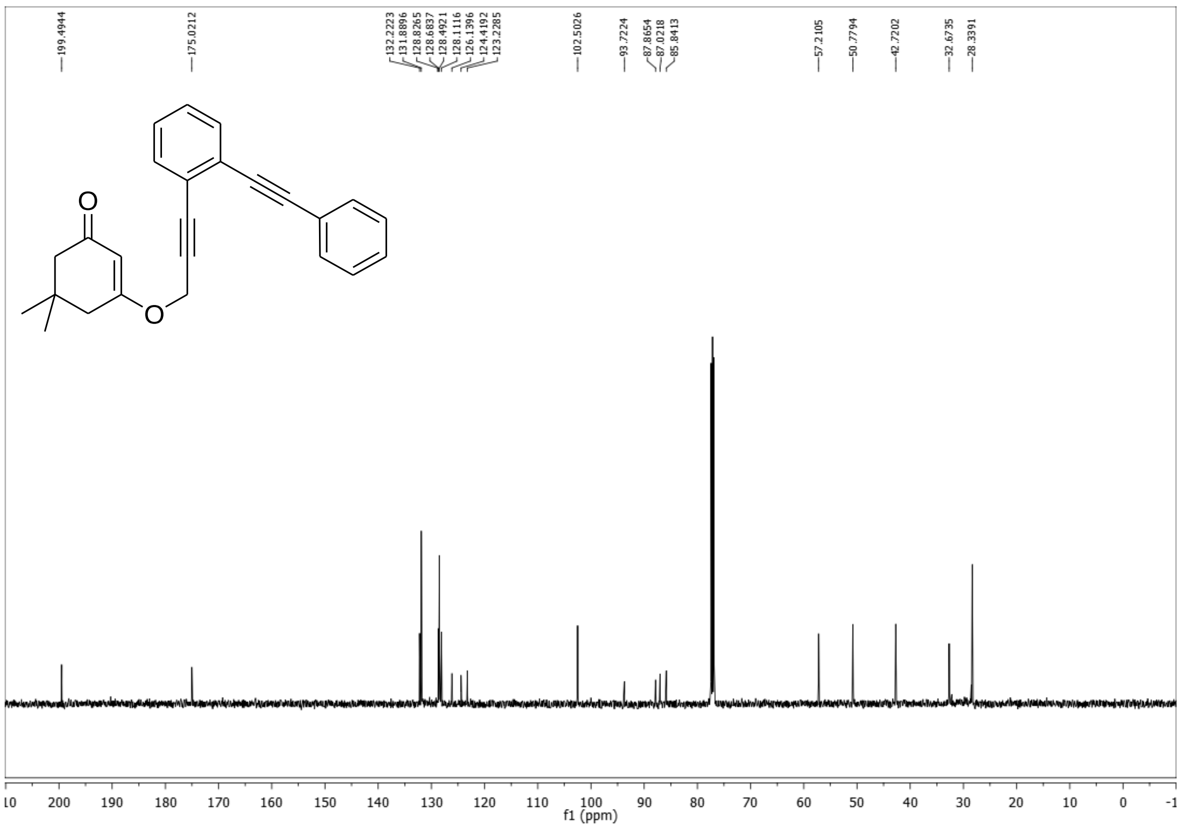

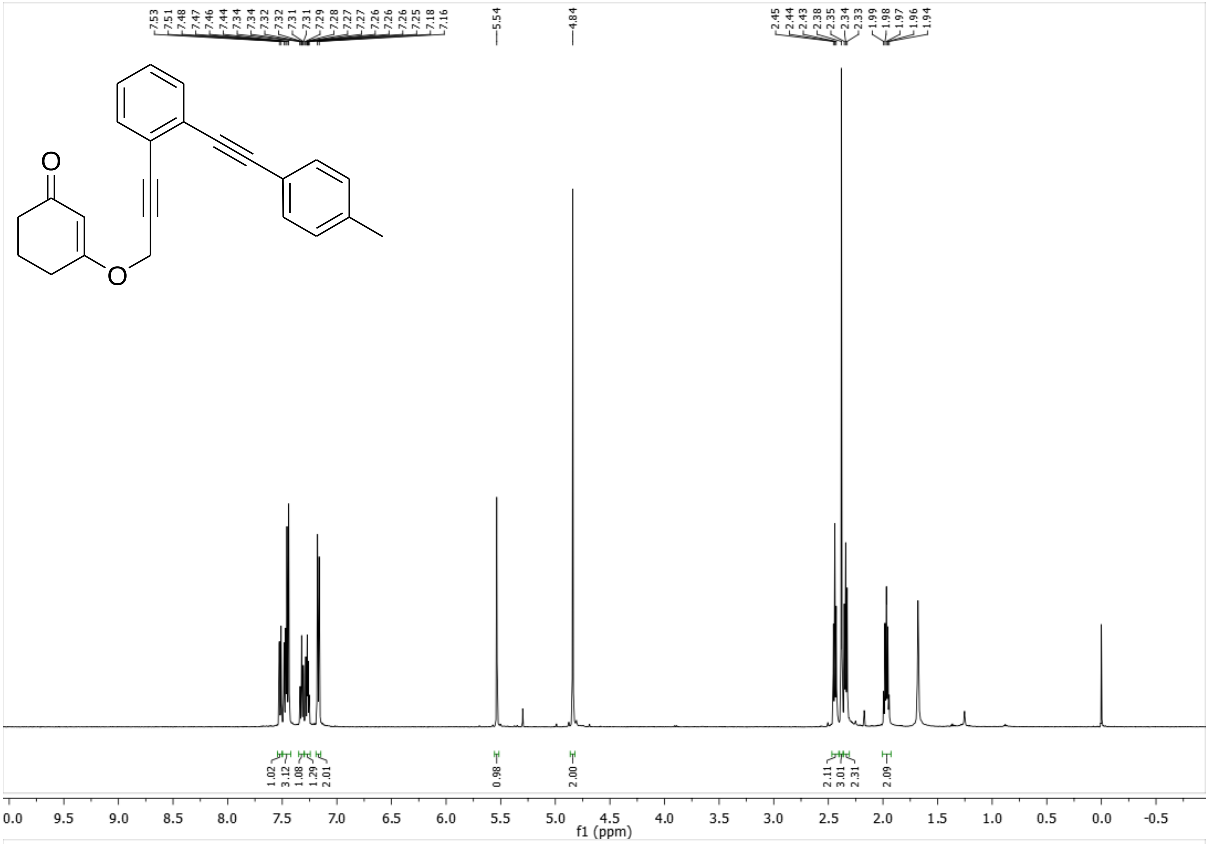
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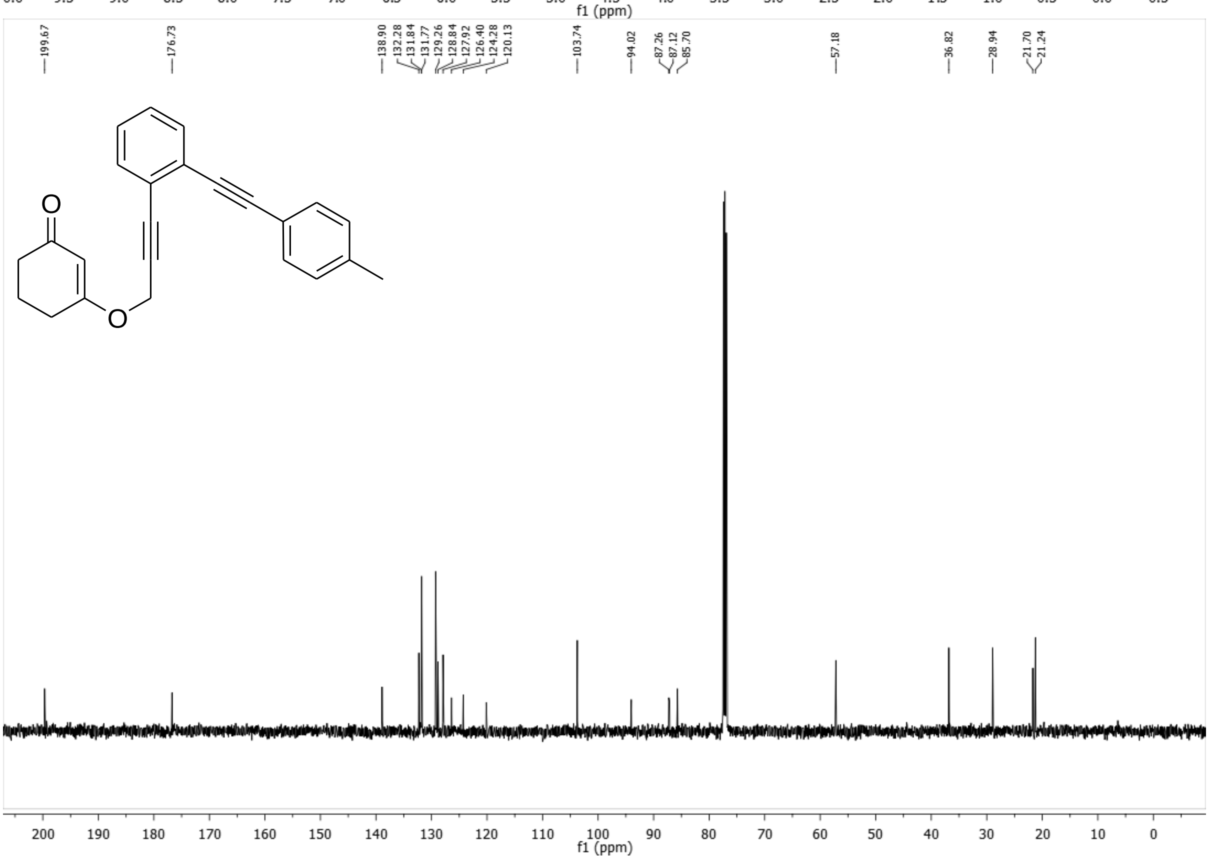
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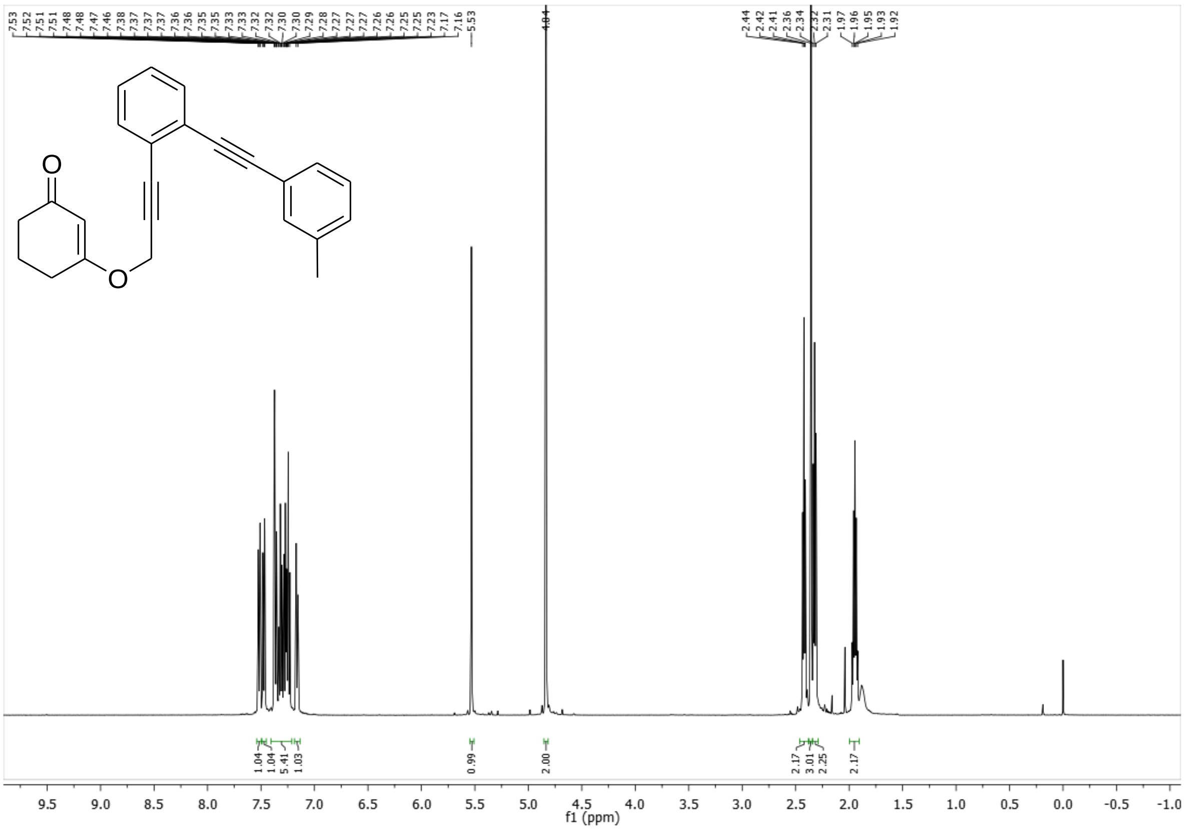
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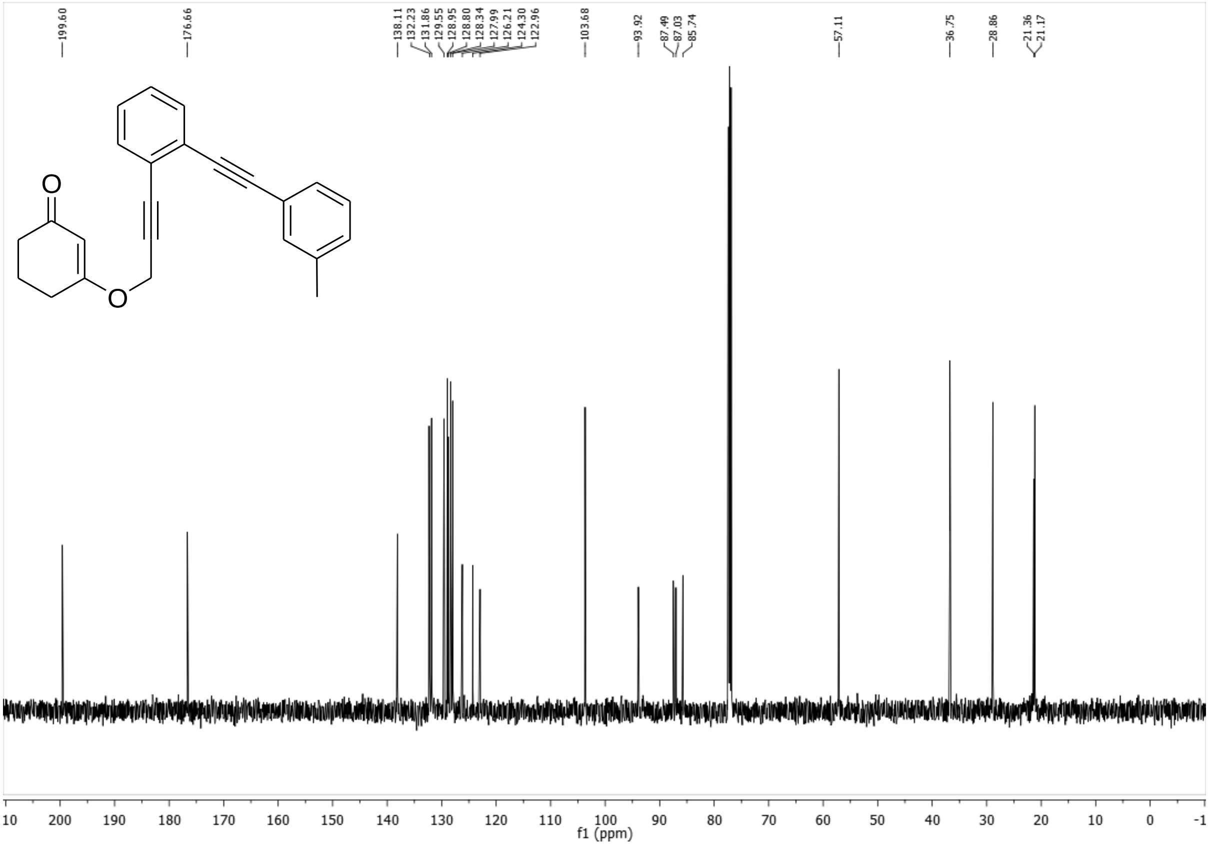
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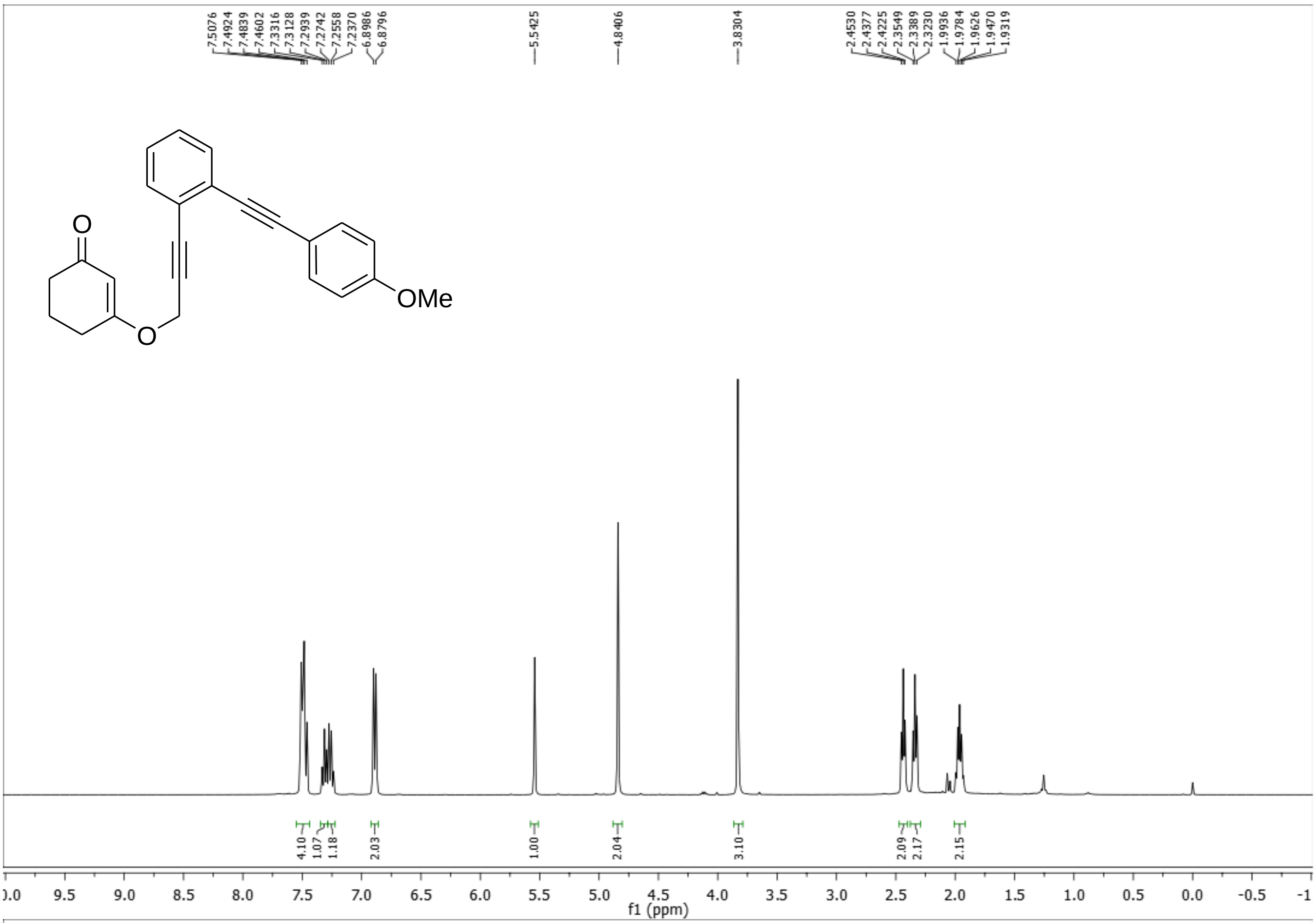
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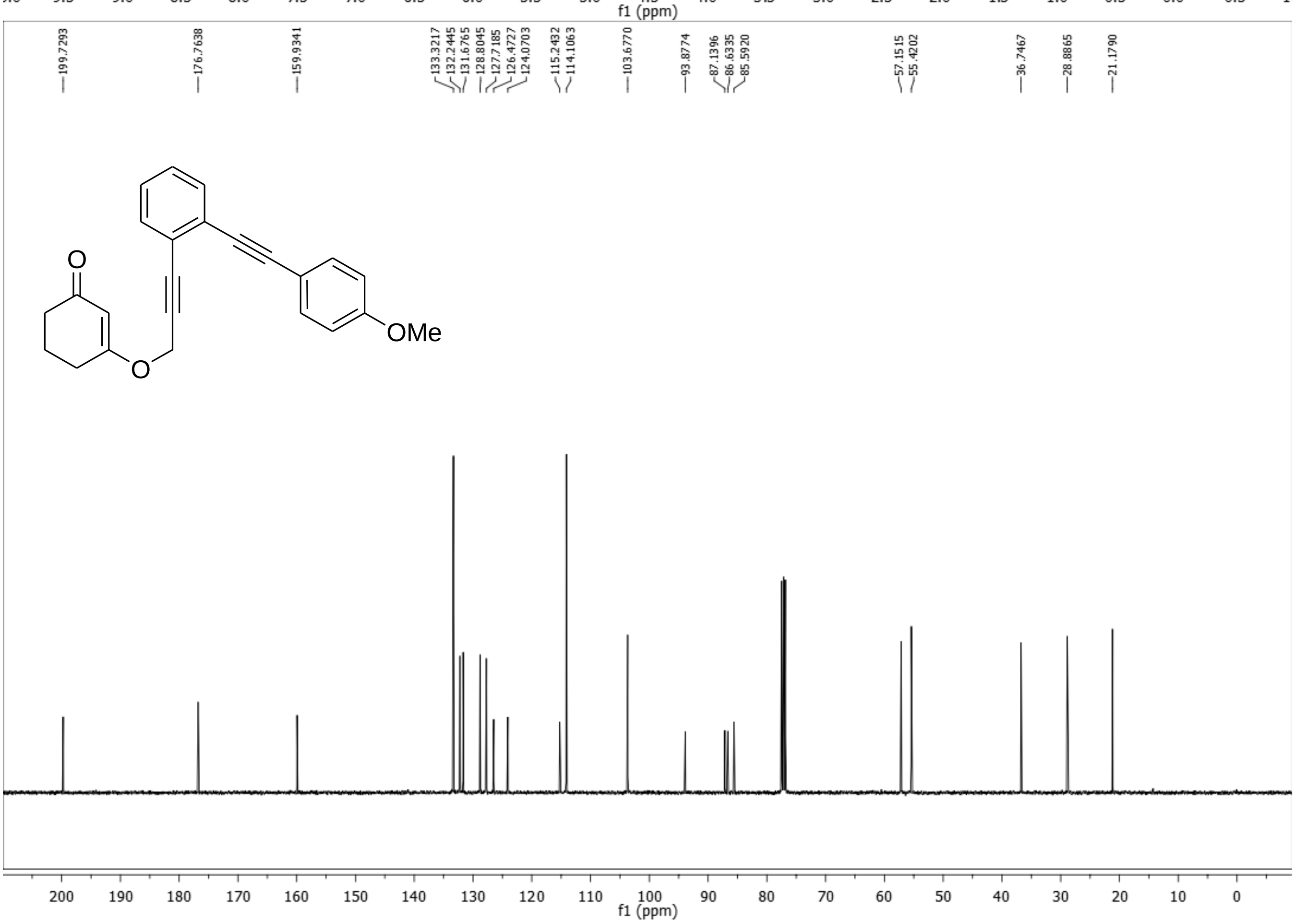
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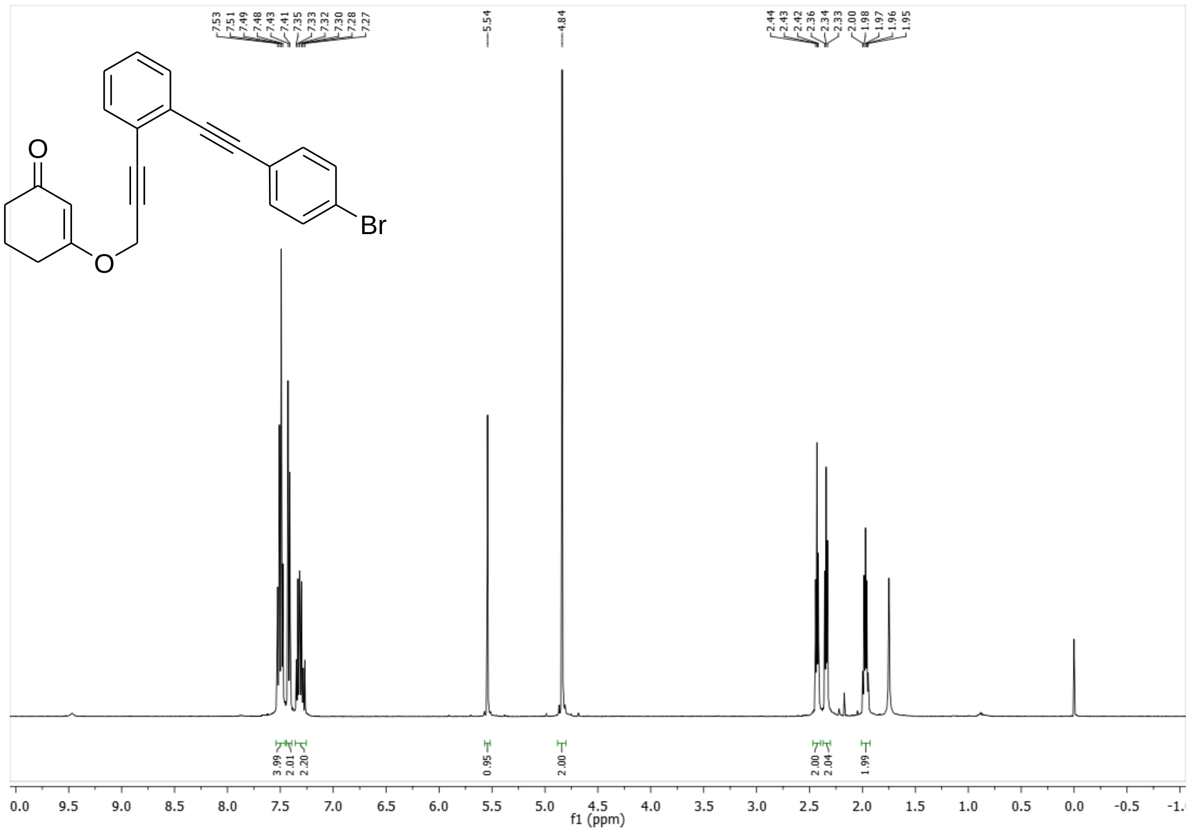

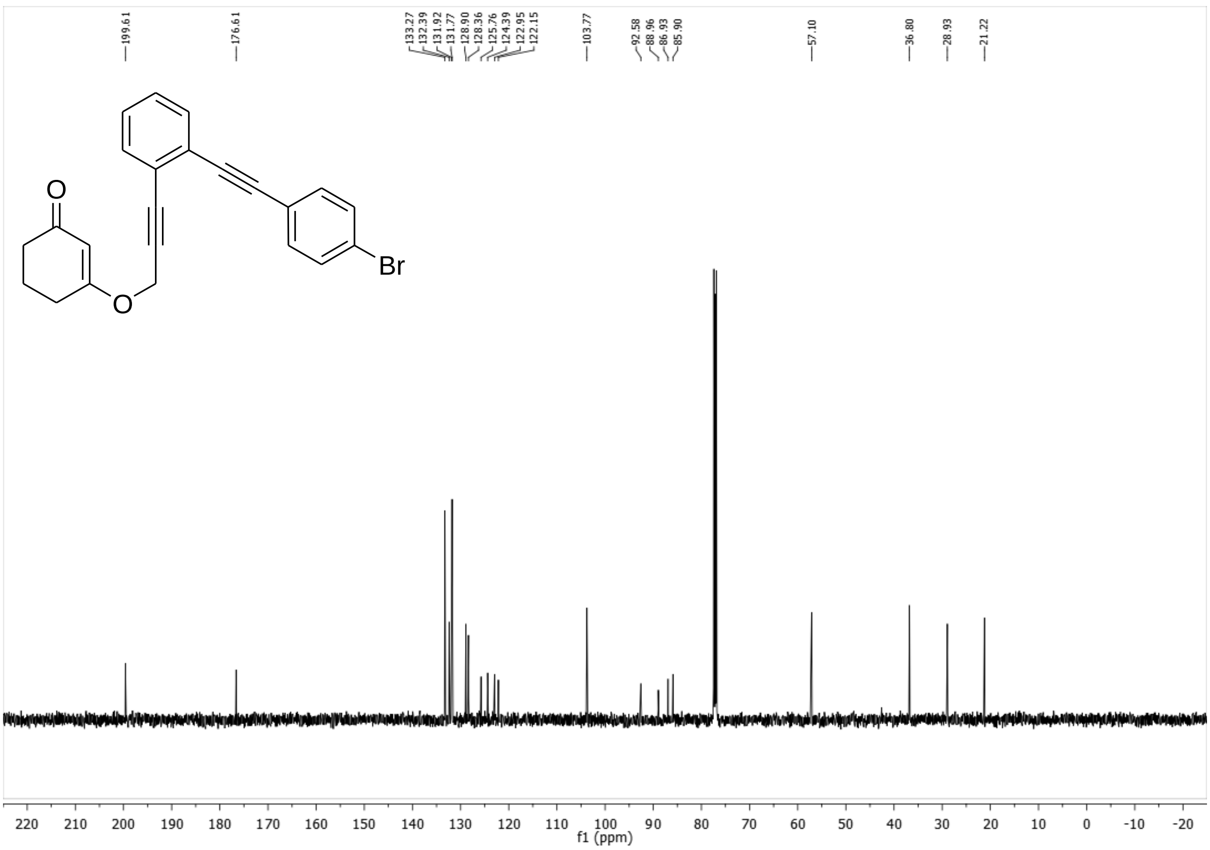

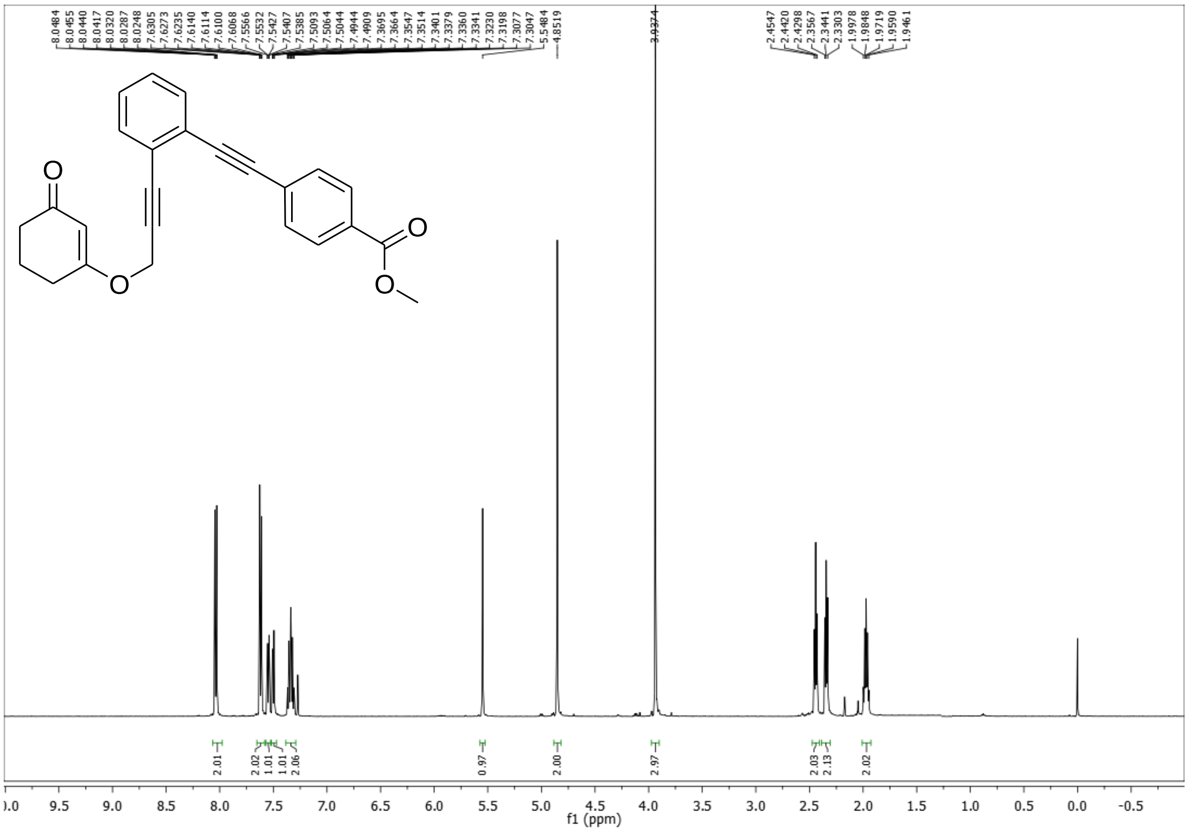

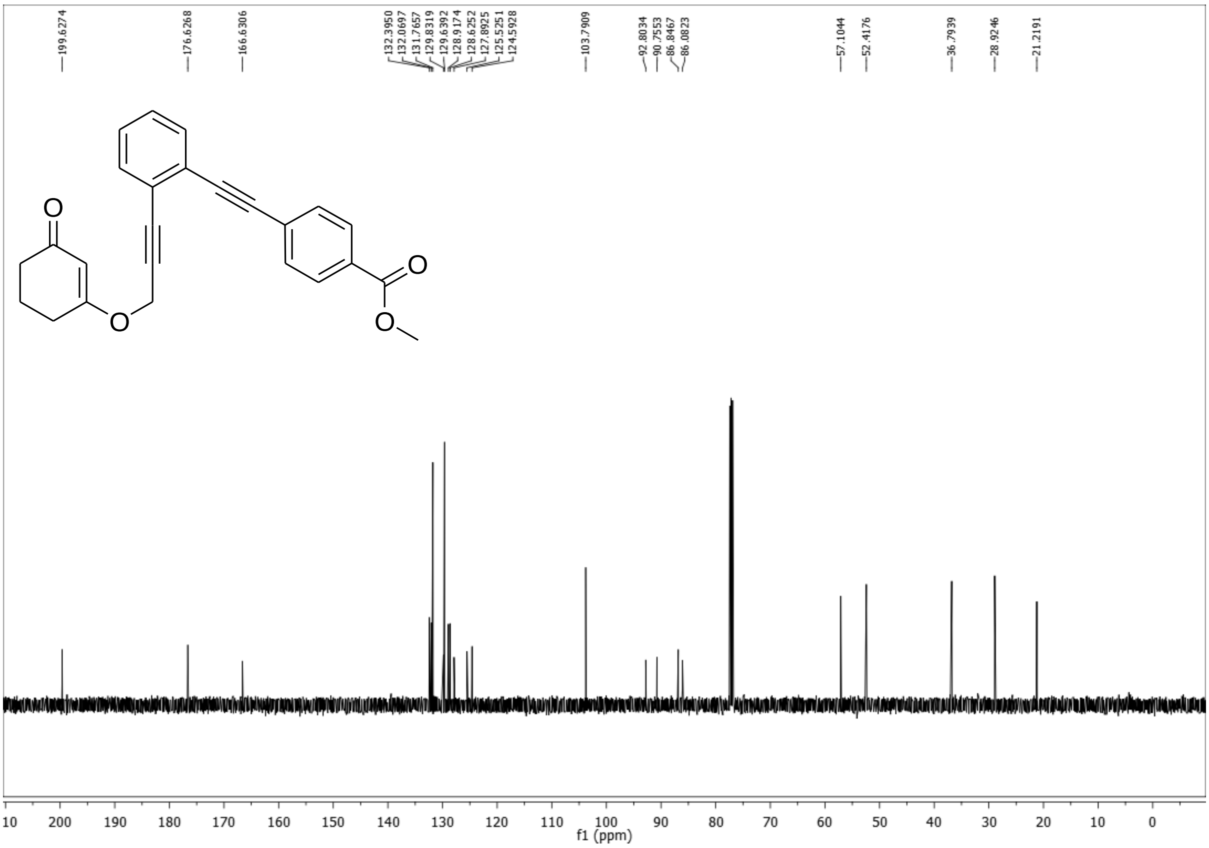

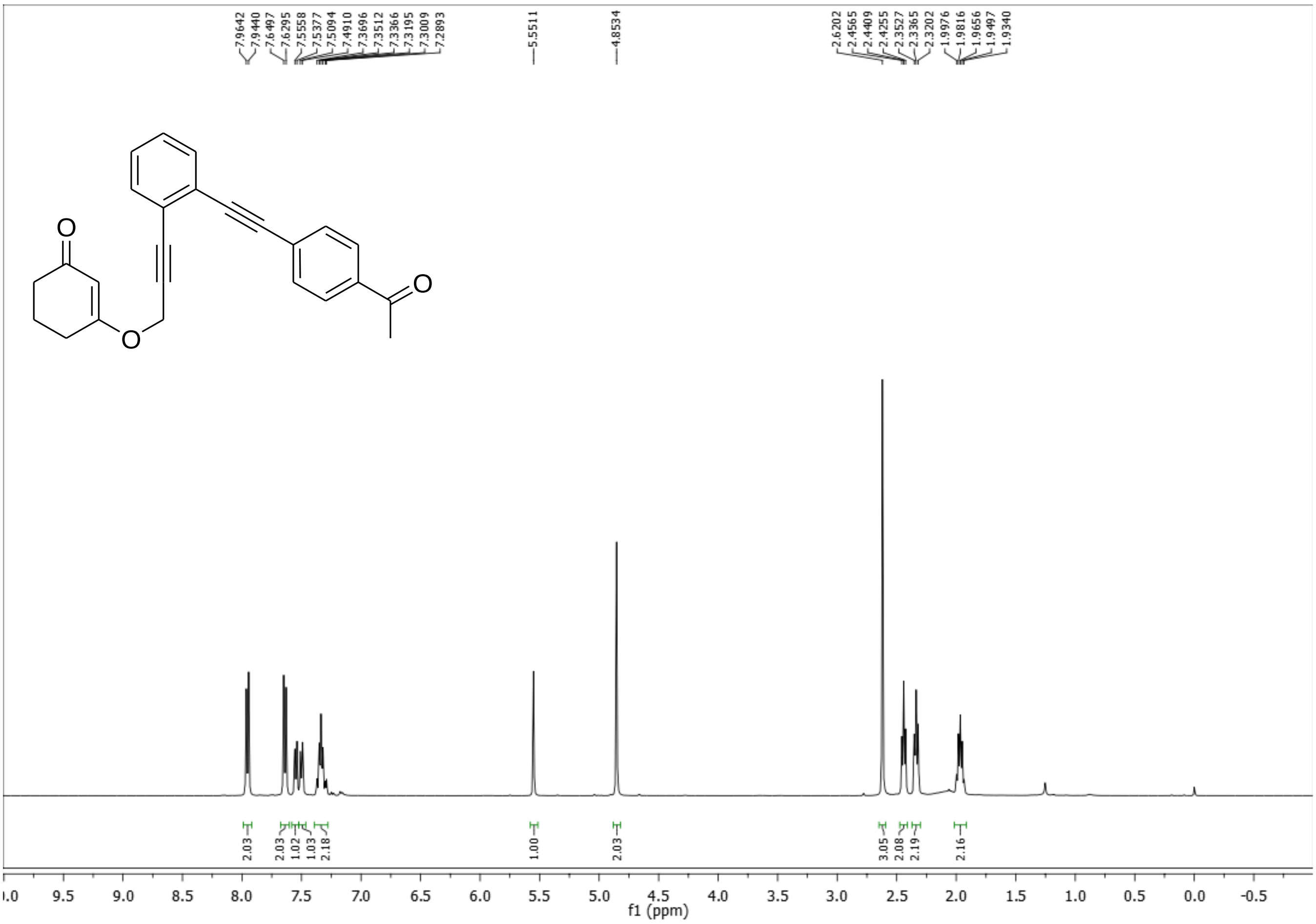

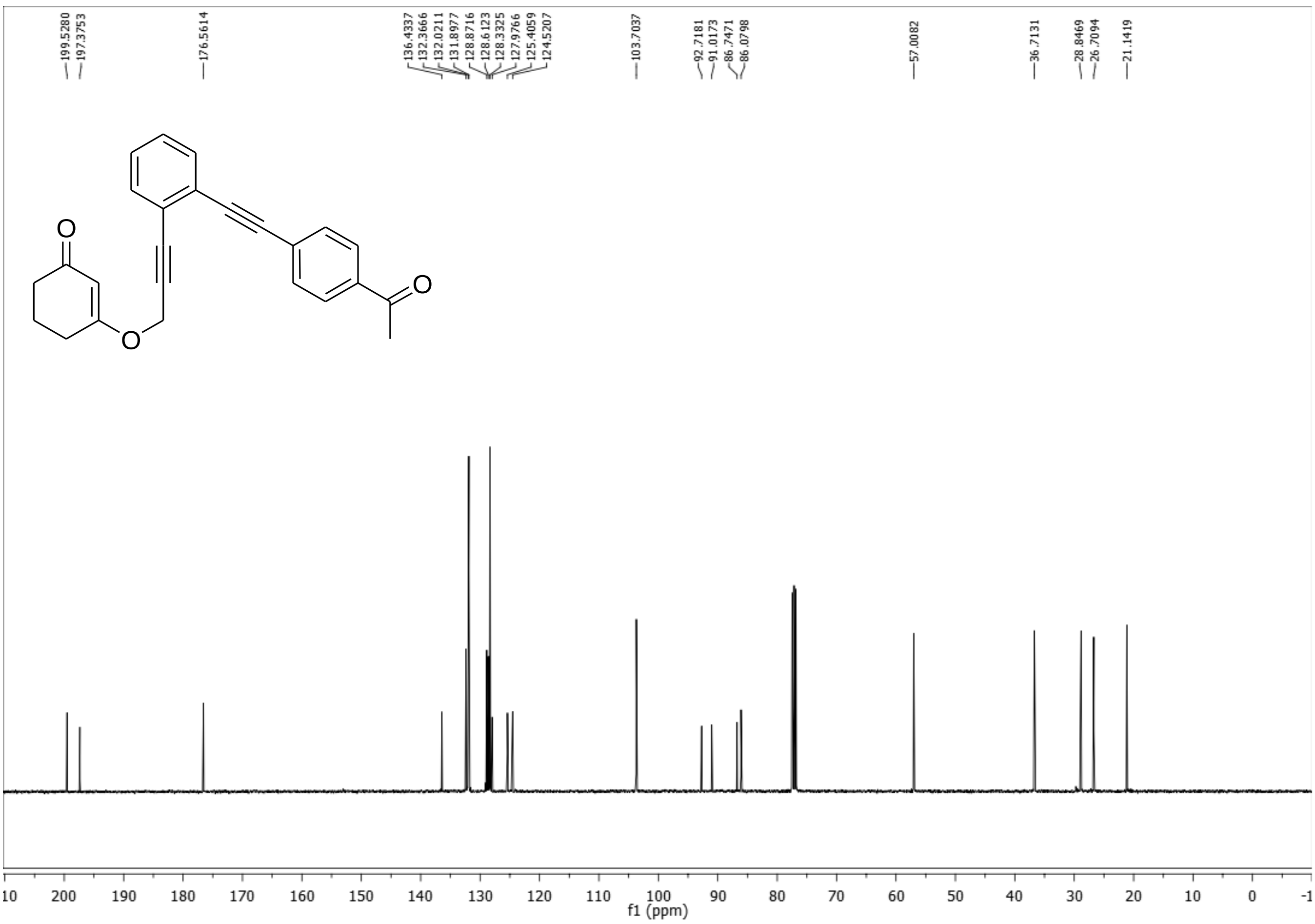

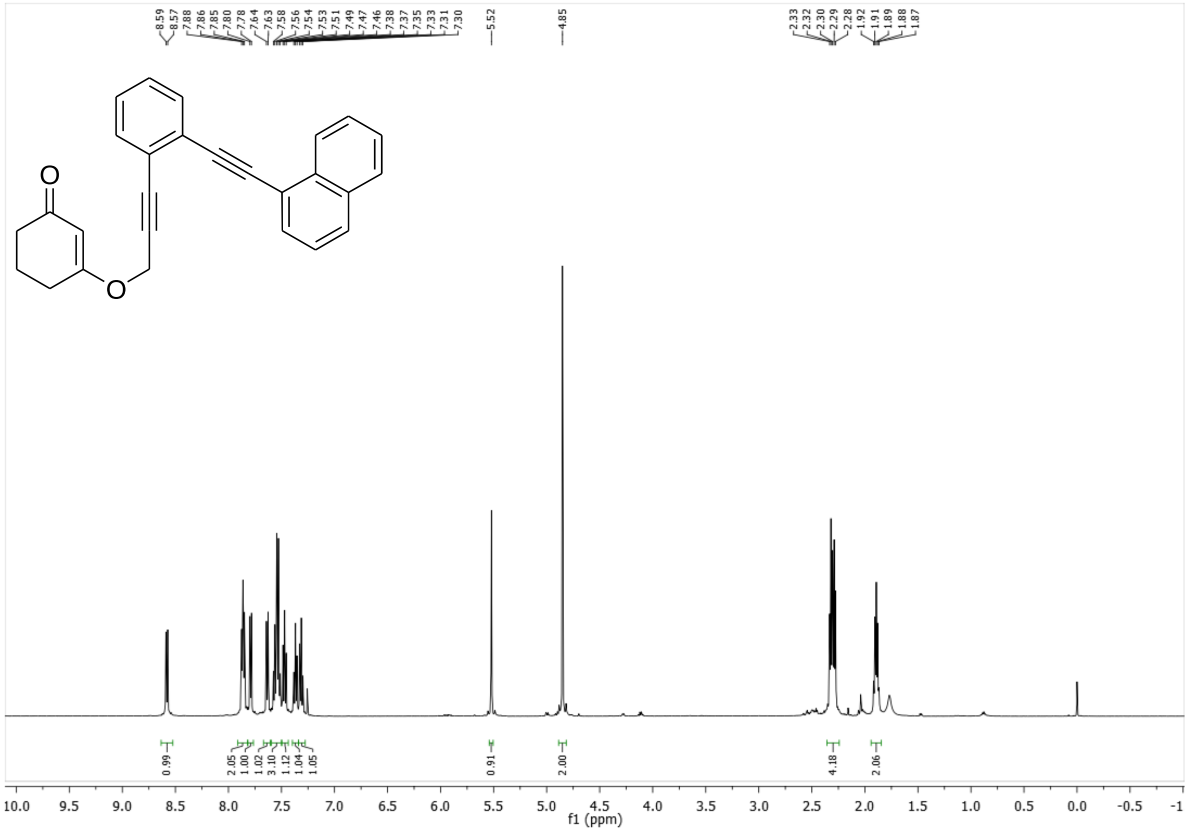

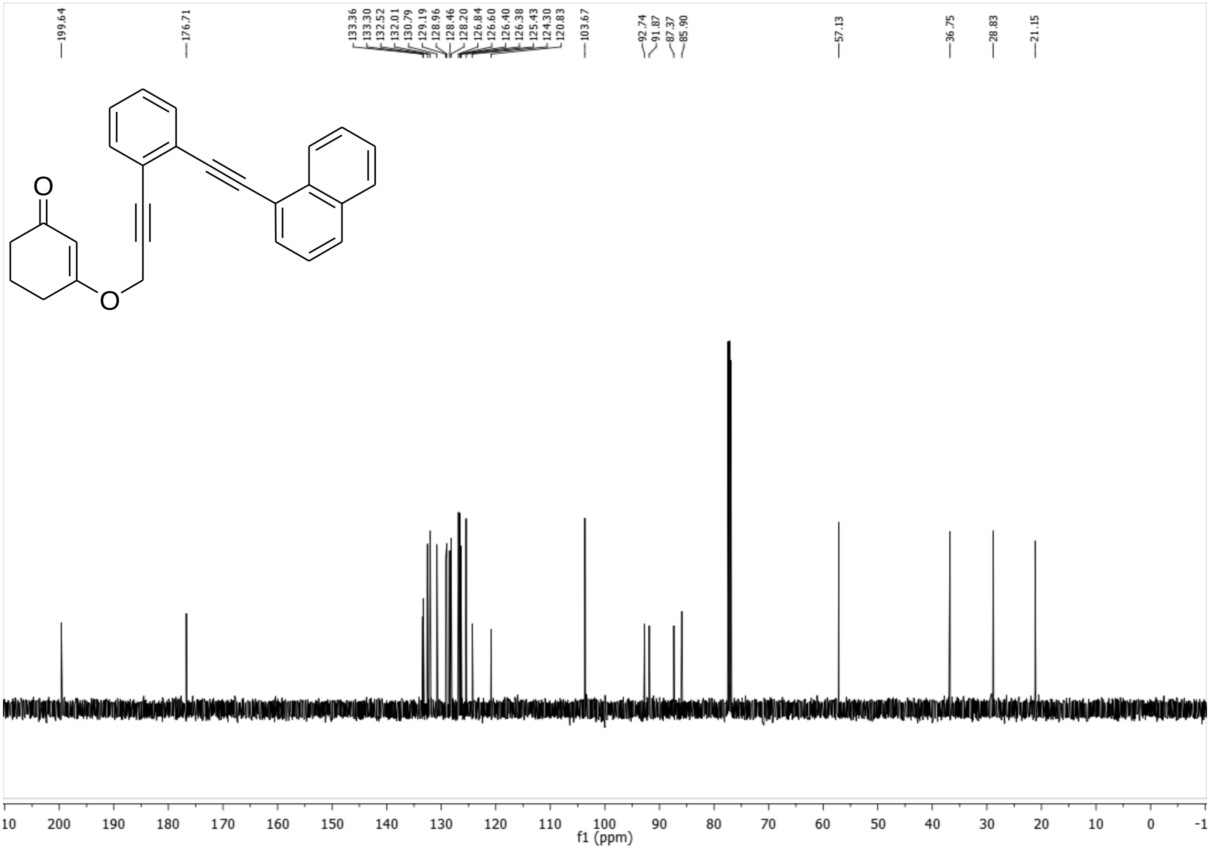

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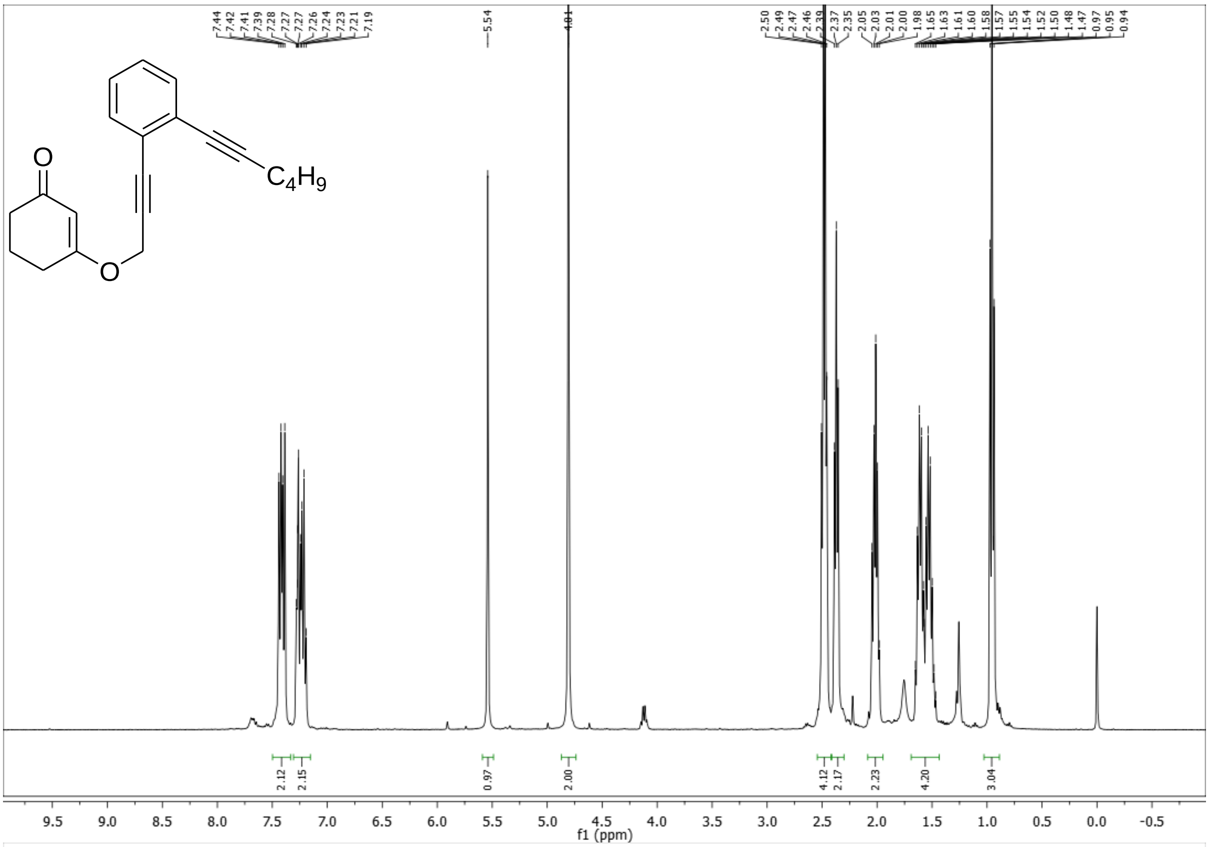

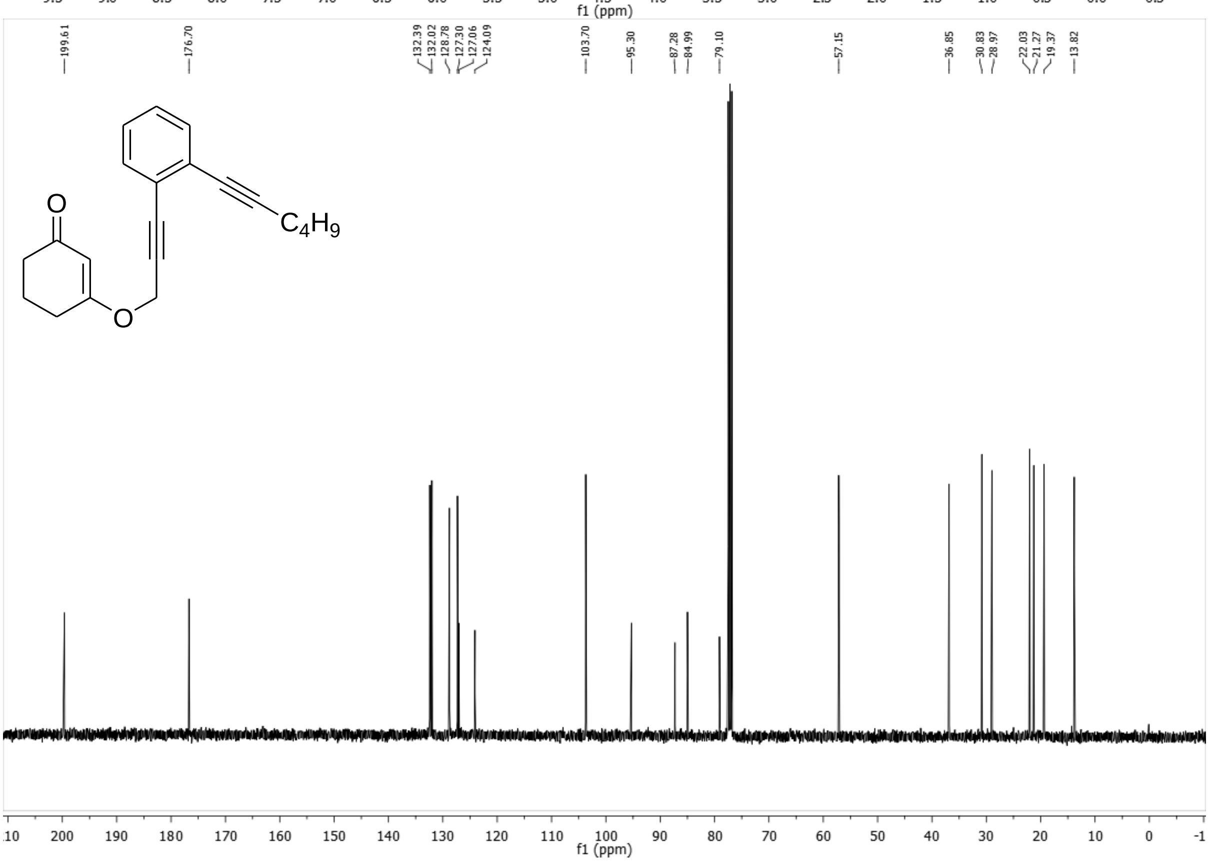

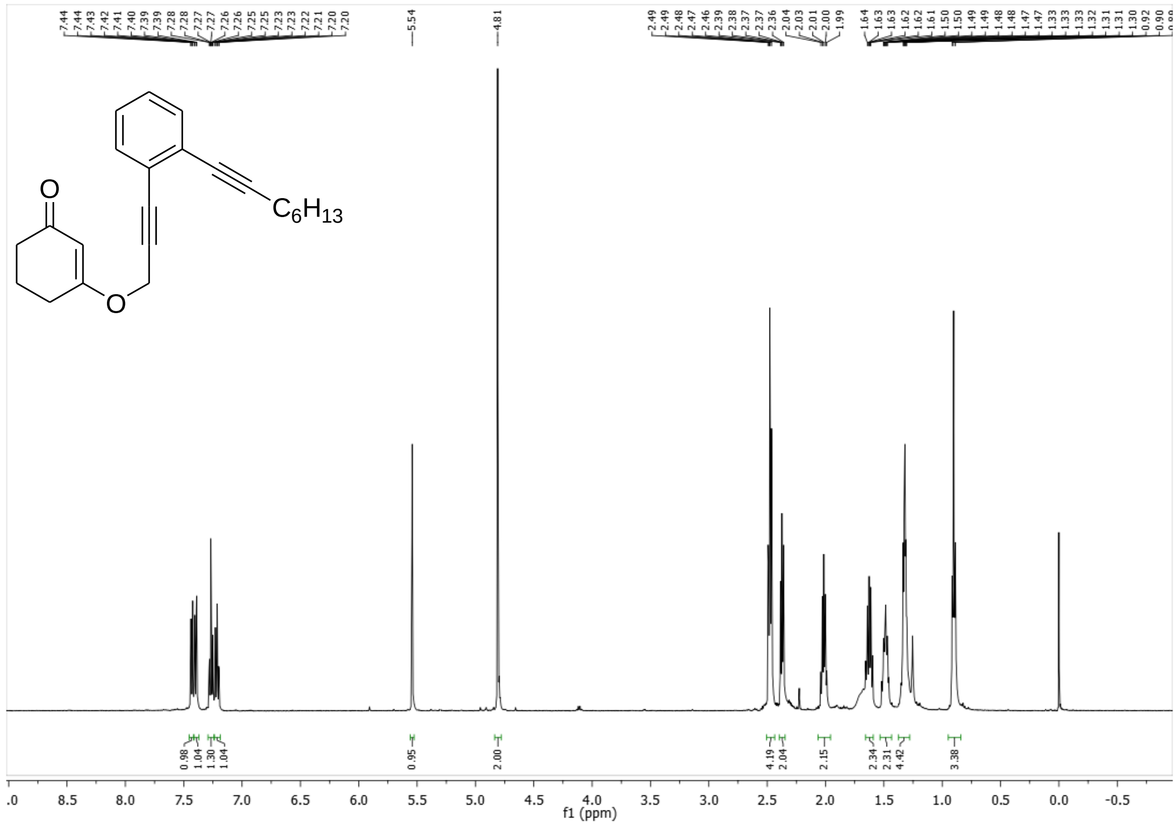
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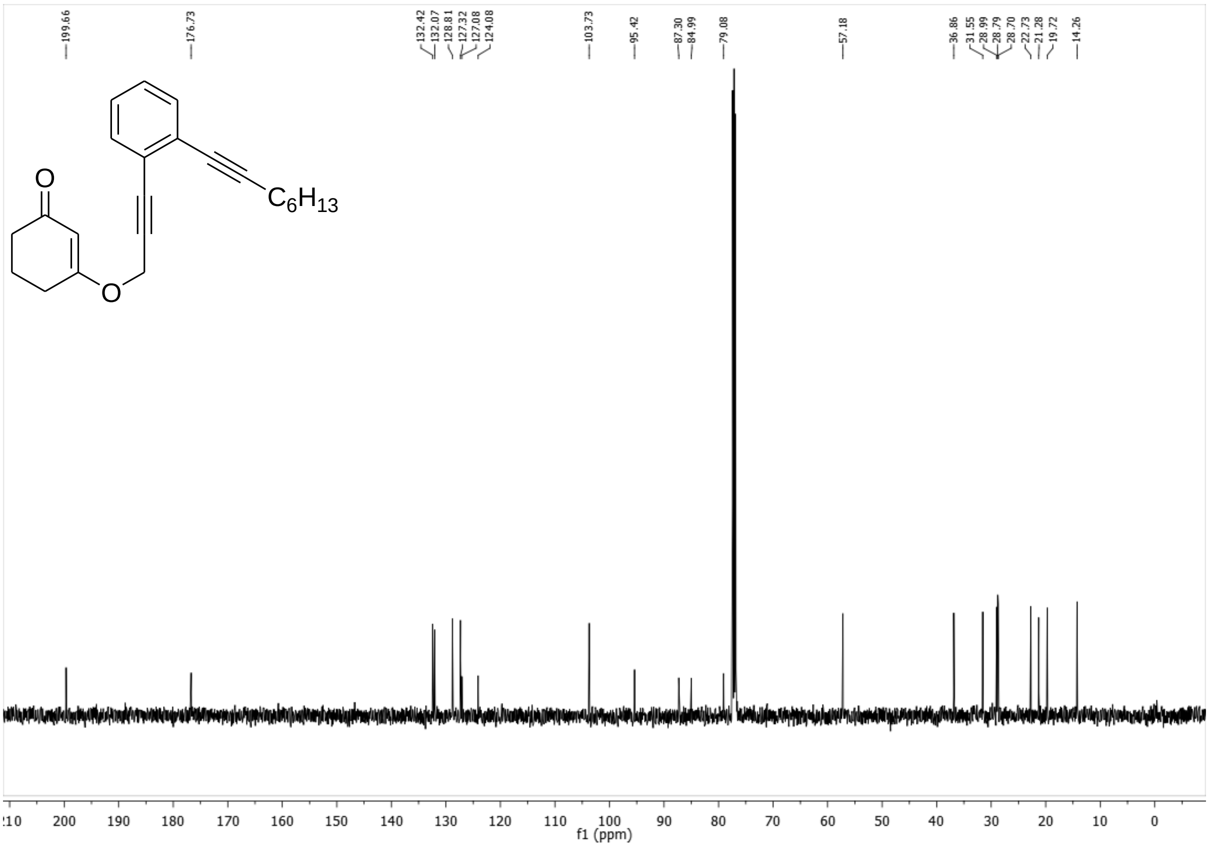
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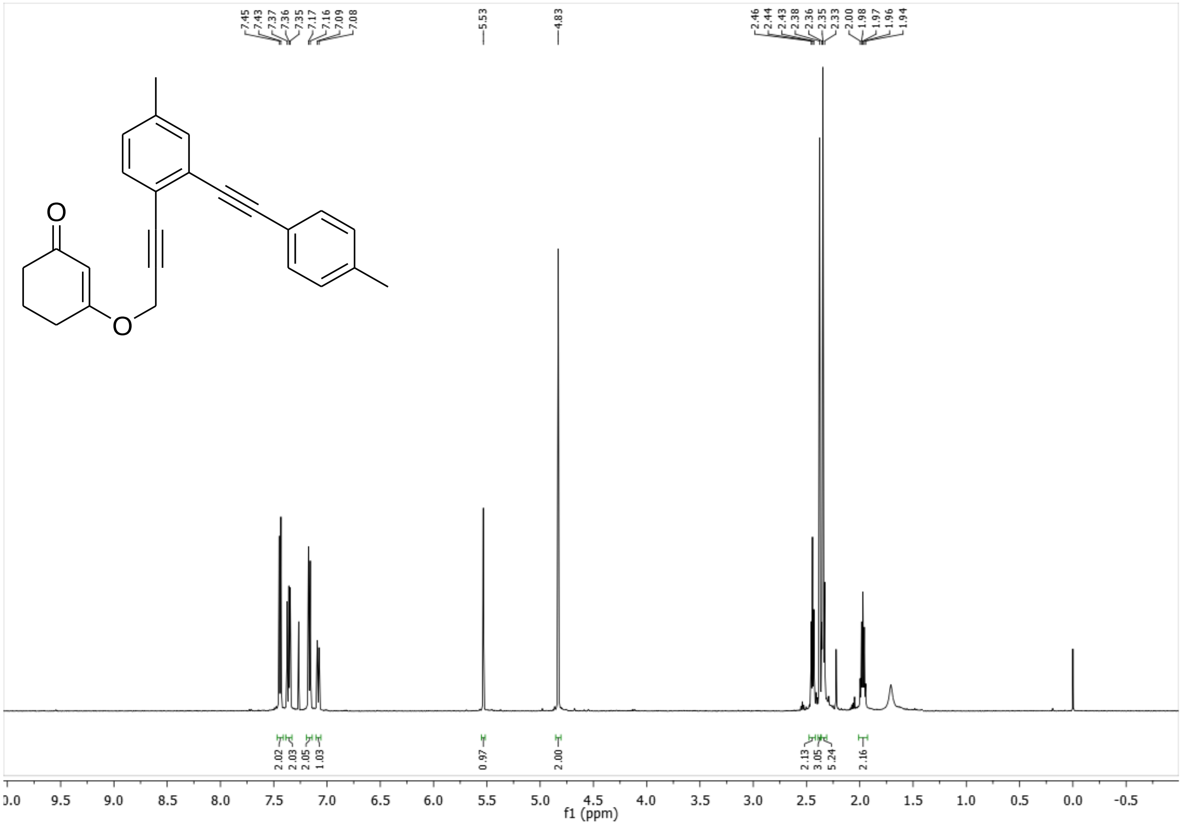

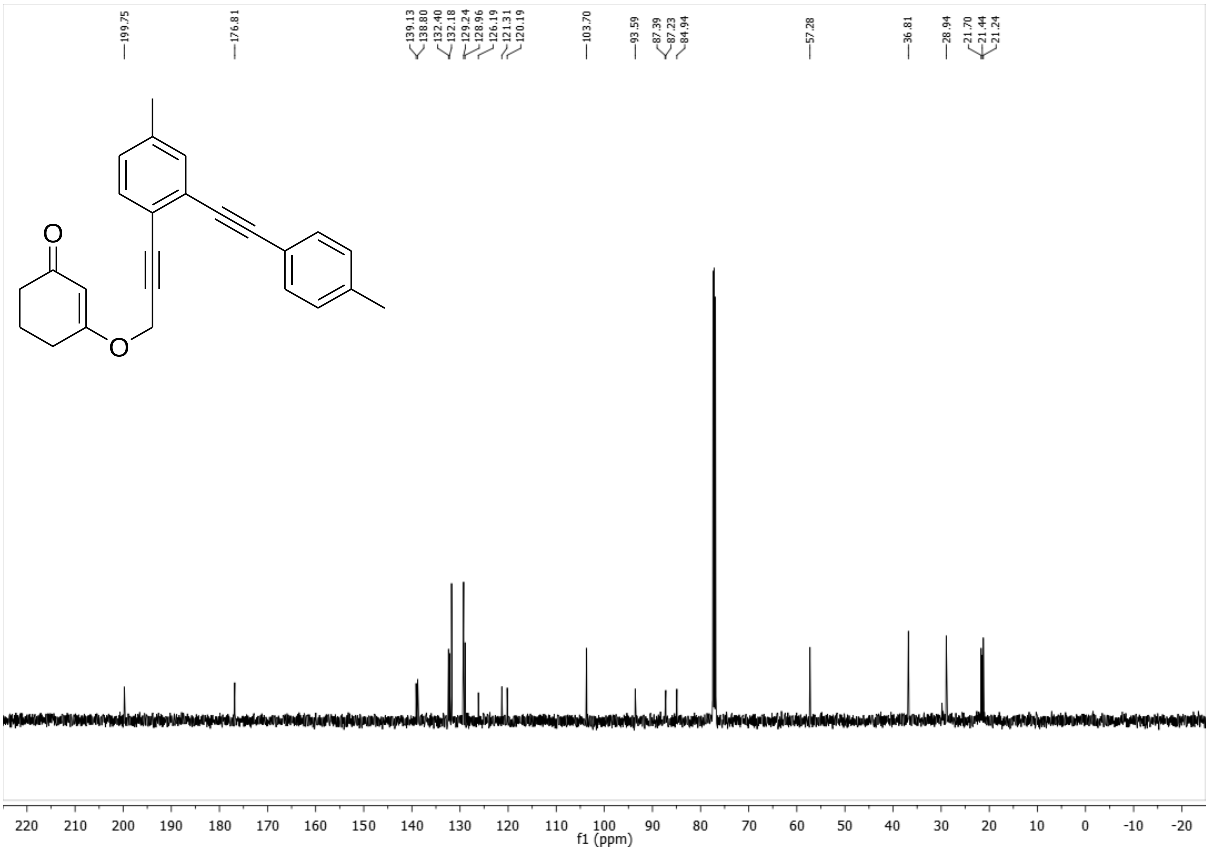

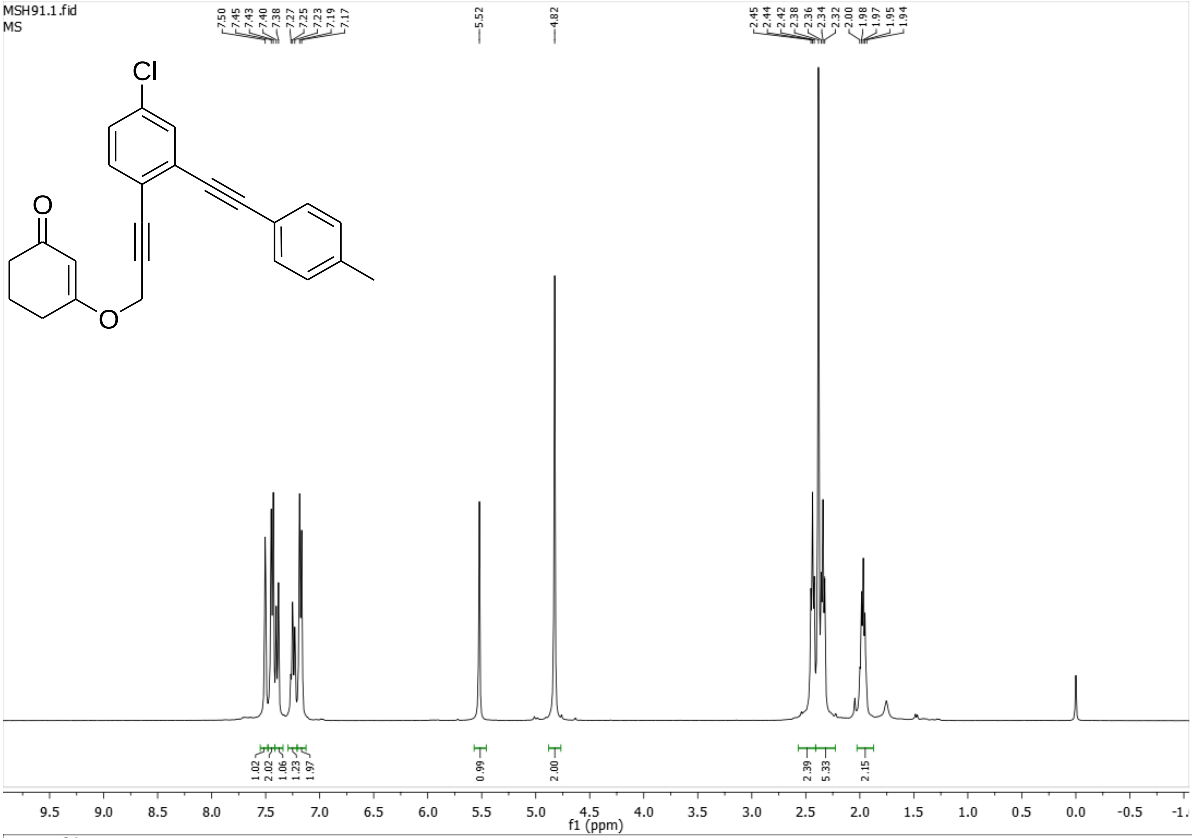
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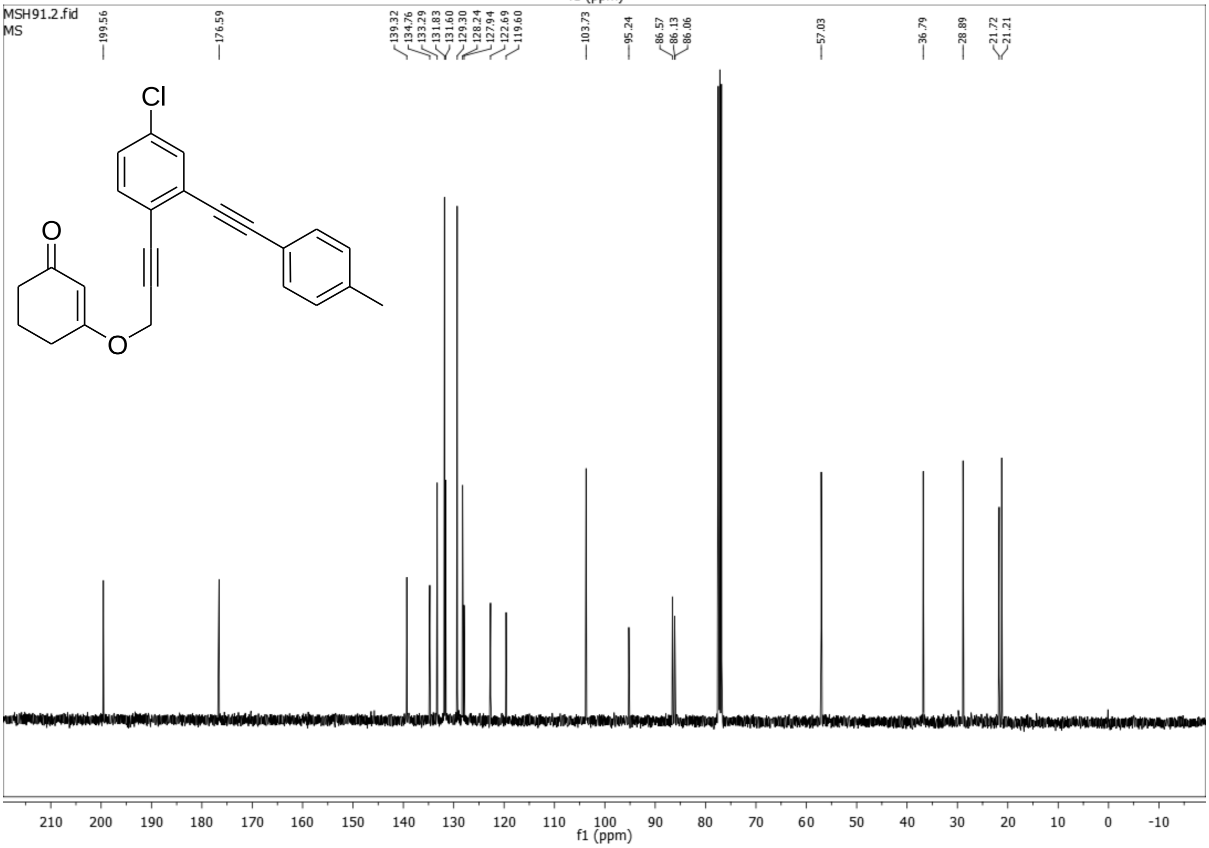
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176.59
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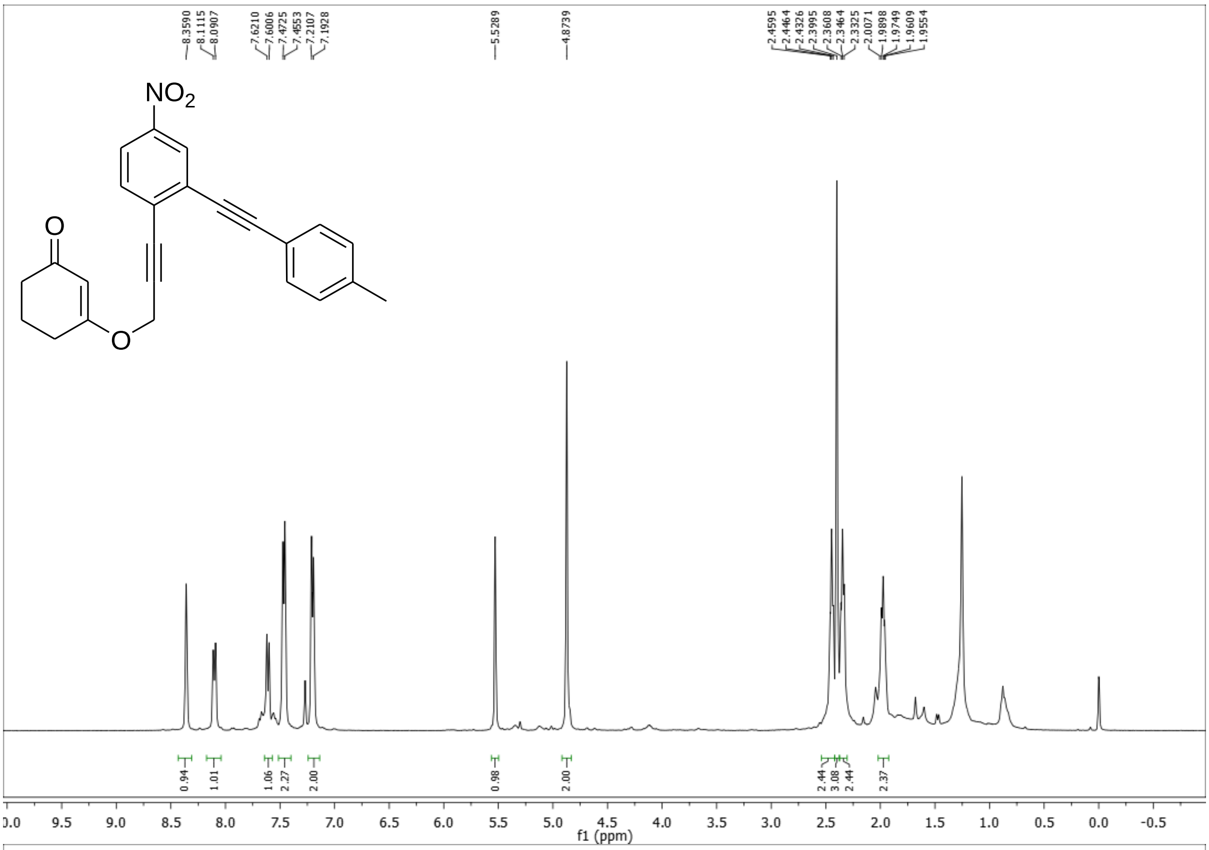
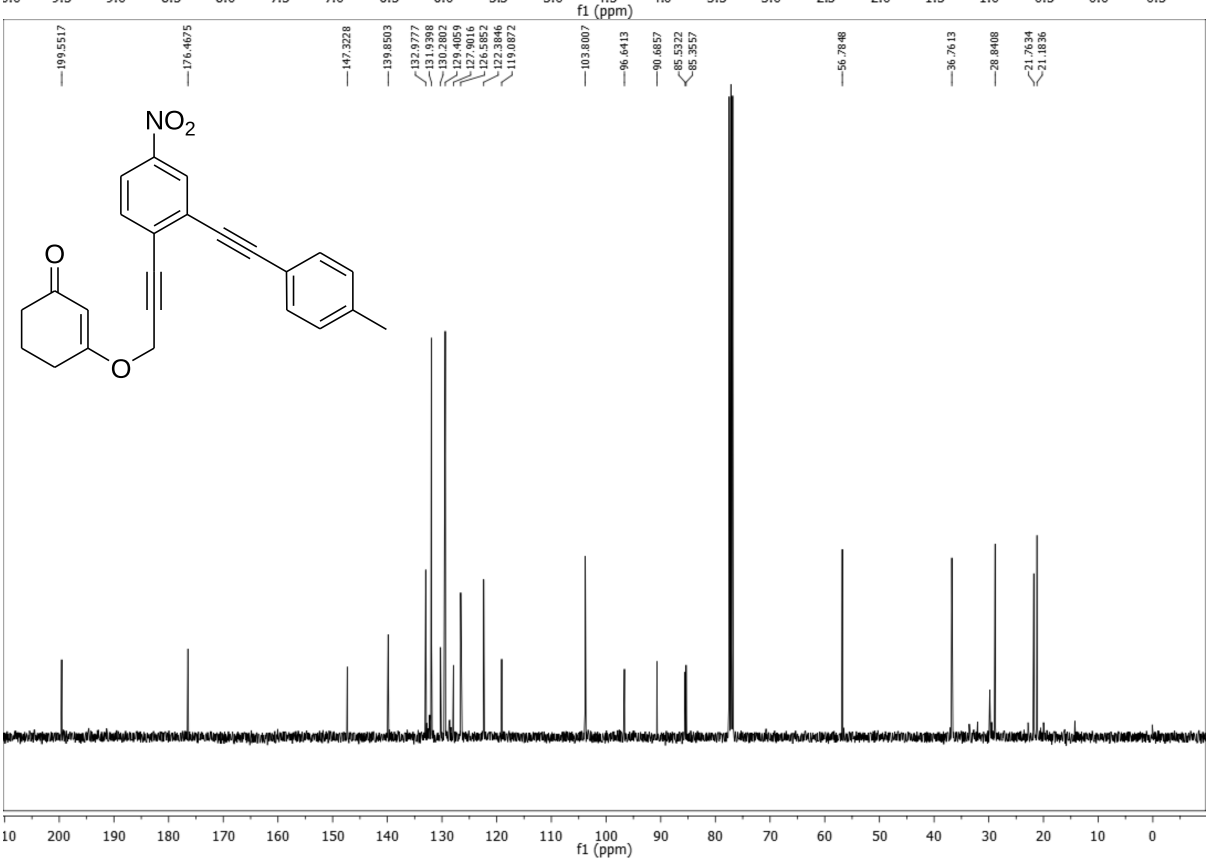
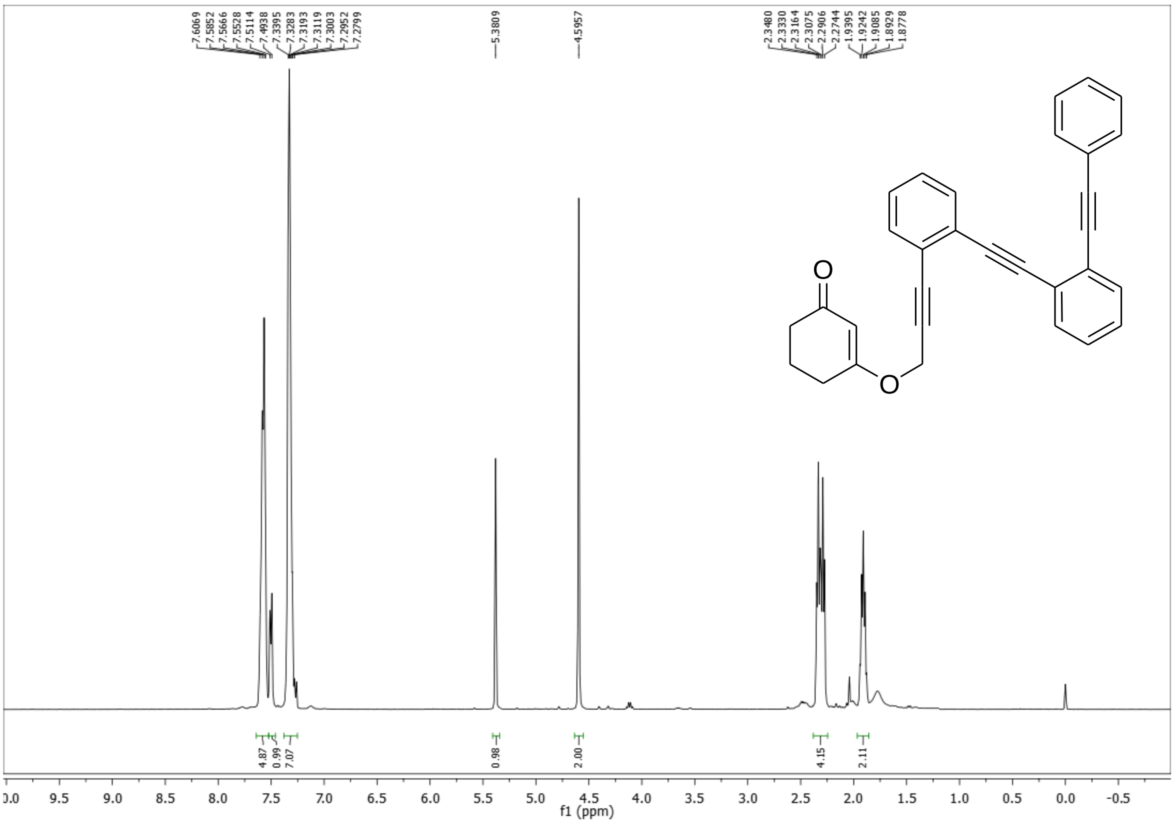
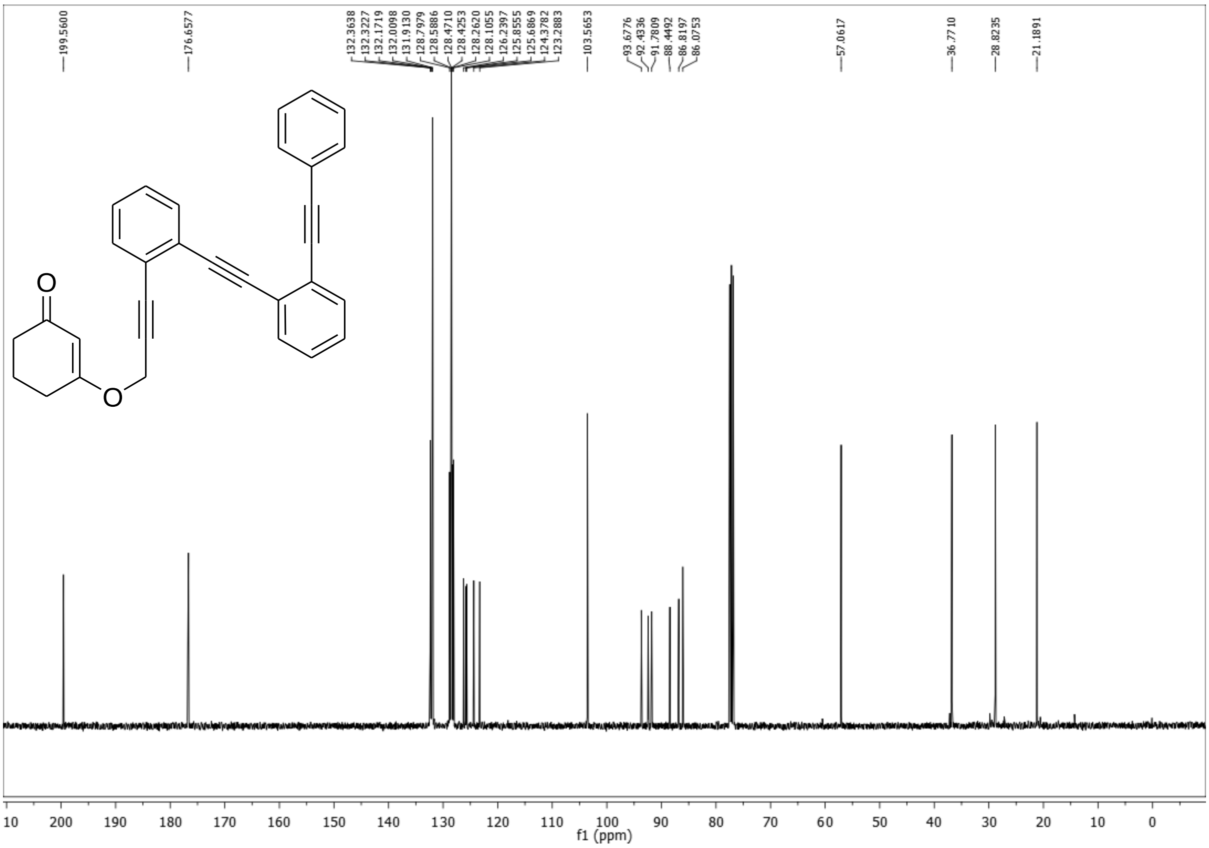
103.73
95.24
86.57 86.13 86.06
57.03
36.79
28.89
21.72 21.21
Cl
O
O
f1 (ppm)

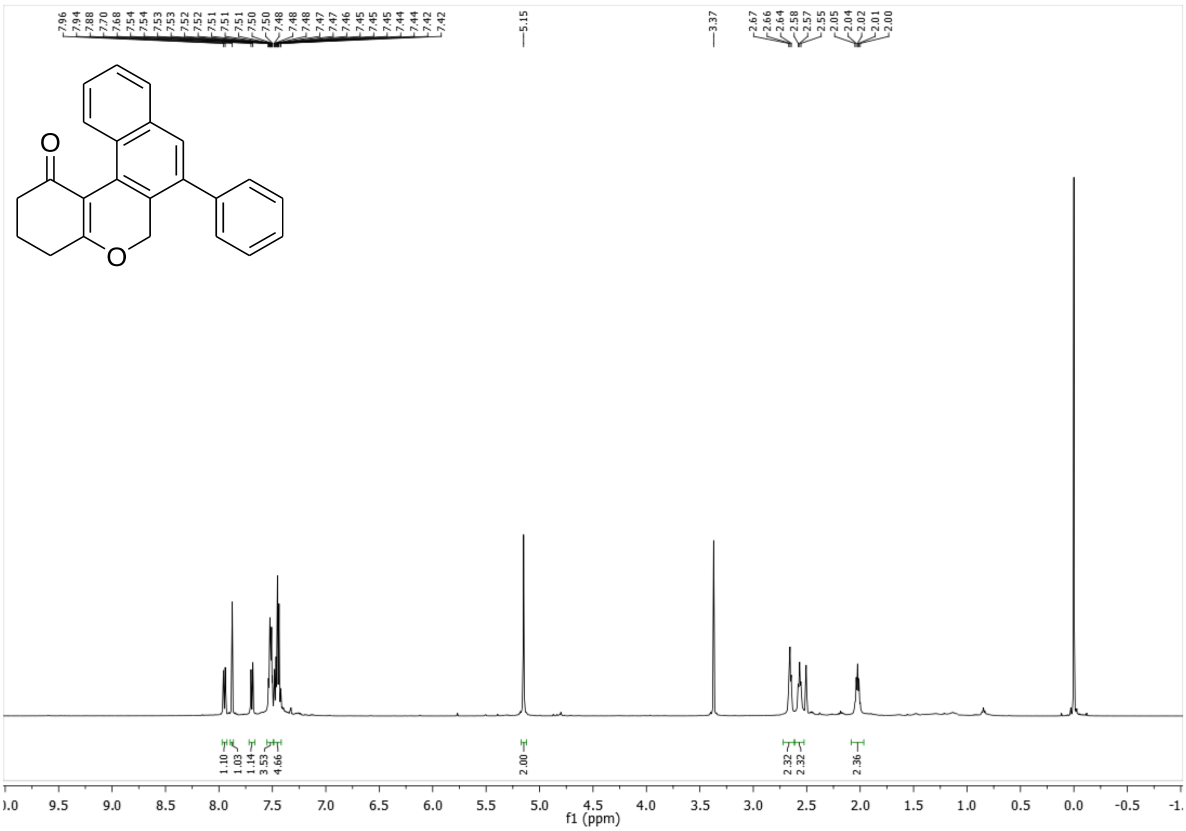
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry






O
O
f1 (ppm)


O
O
f1 (ppm)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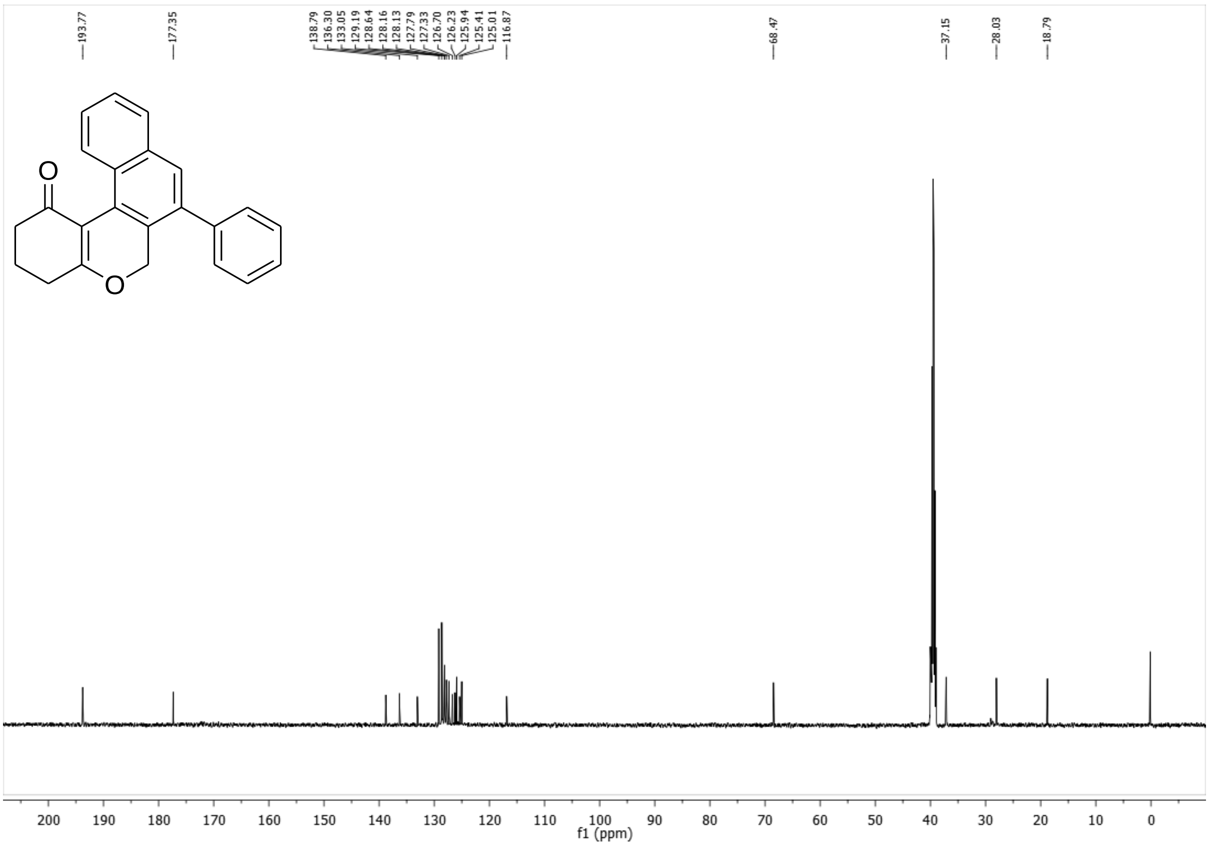
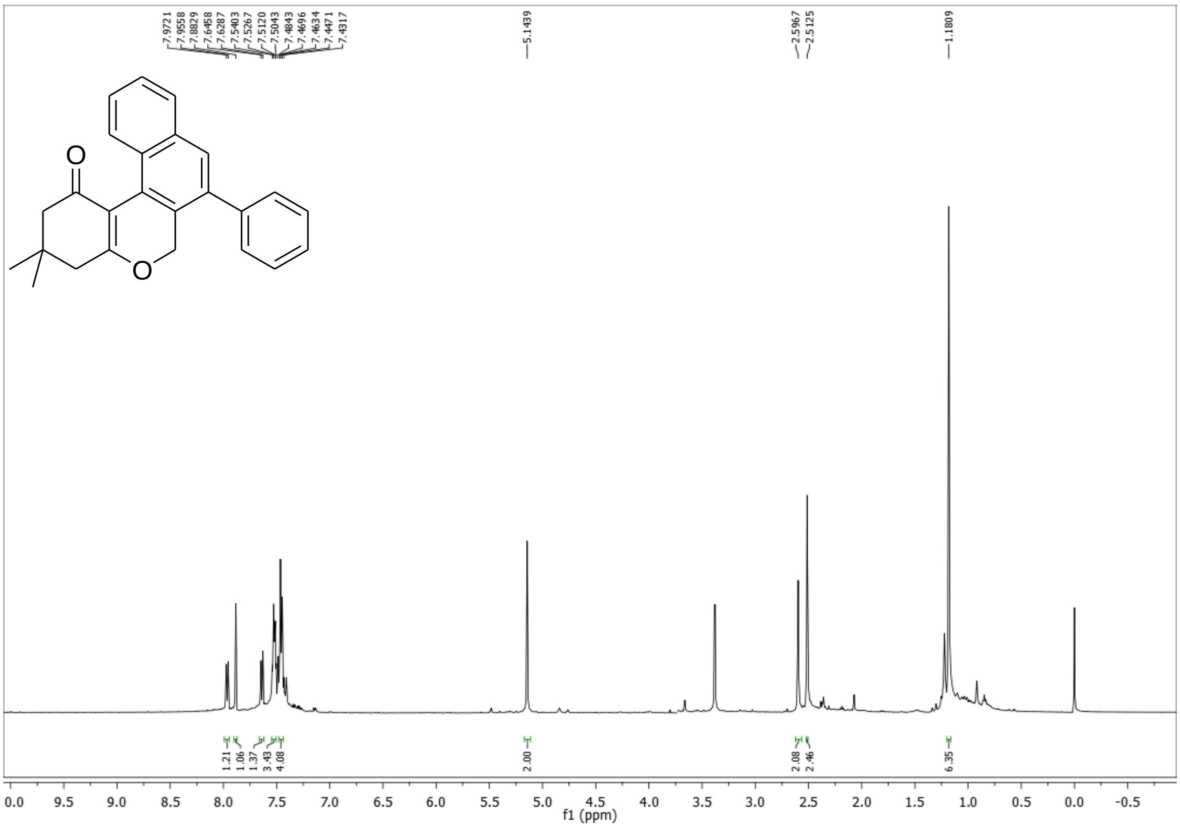
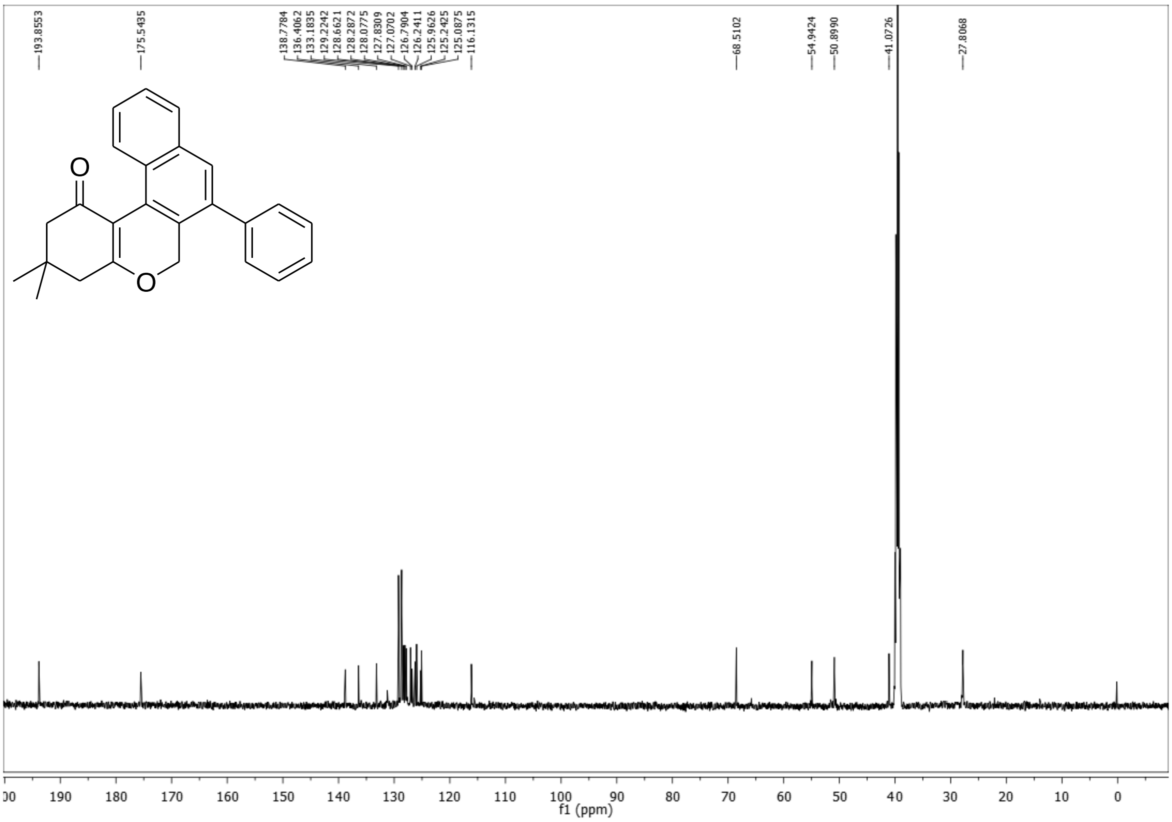
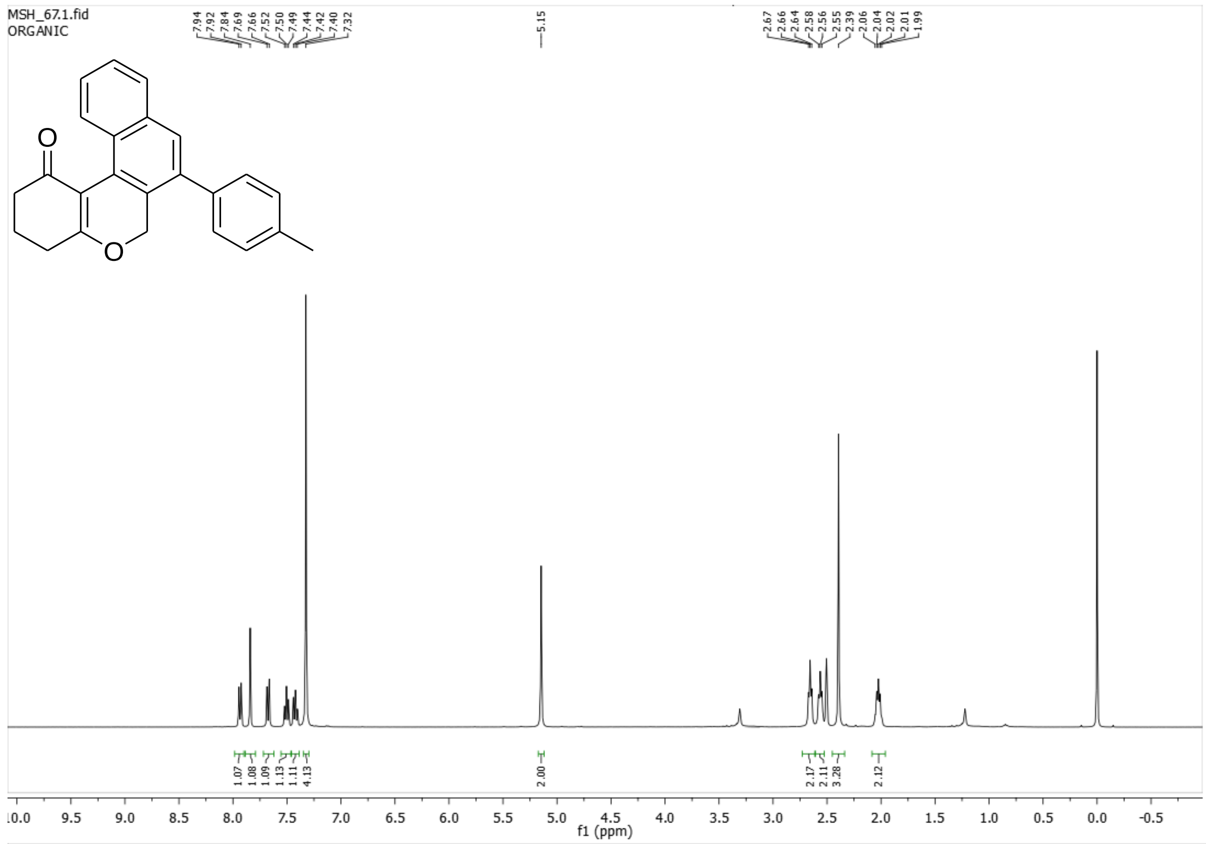
7.94
7.88
7.70
7.68
7.54
7.54
7.53
7.53
7.52
7.52
7.51
7.51
7.51
7.50
7.50
7.48
7.48
7.48
7.47
7.47
7.46
7.45
7.45
7.45
7.44
7.44
7.42
7.42
5.15
3.37
2.67
2.66
2.64
2.58
2.57
2.55
2.05
2.04
2.02
2.01
2.00
O
O
1.10
1.03
1.14
3.53
4.66
2.00
2.32
2.32
2.36
f1 (ppm)

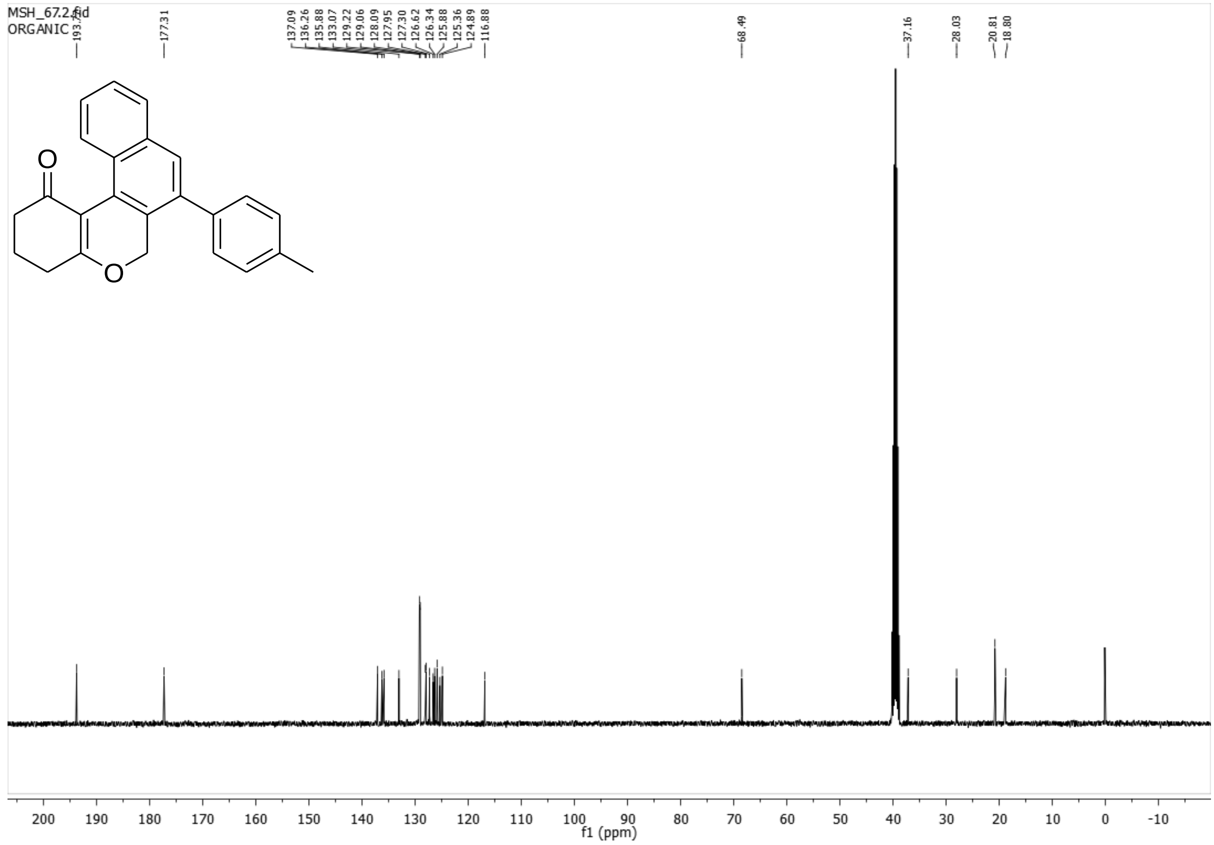

193.77
177.35
138.79
136.30
133.05
129.19
128.64
128.16
128.13
127.79
127.33
126.70
126.23
125.94
125.41
125.01
116.87
68.47
37.15
28.03
18.79
O
O
f1 (ppm)

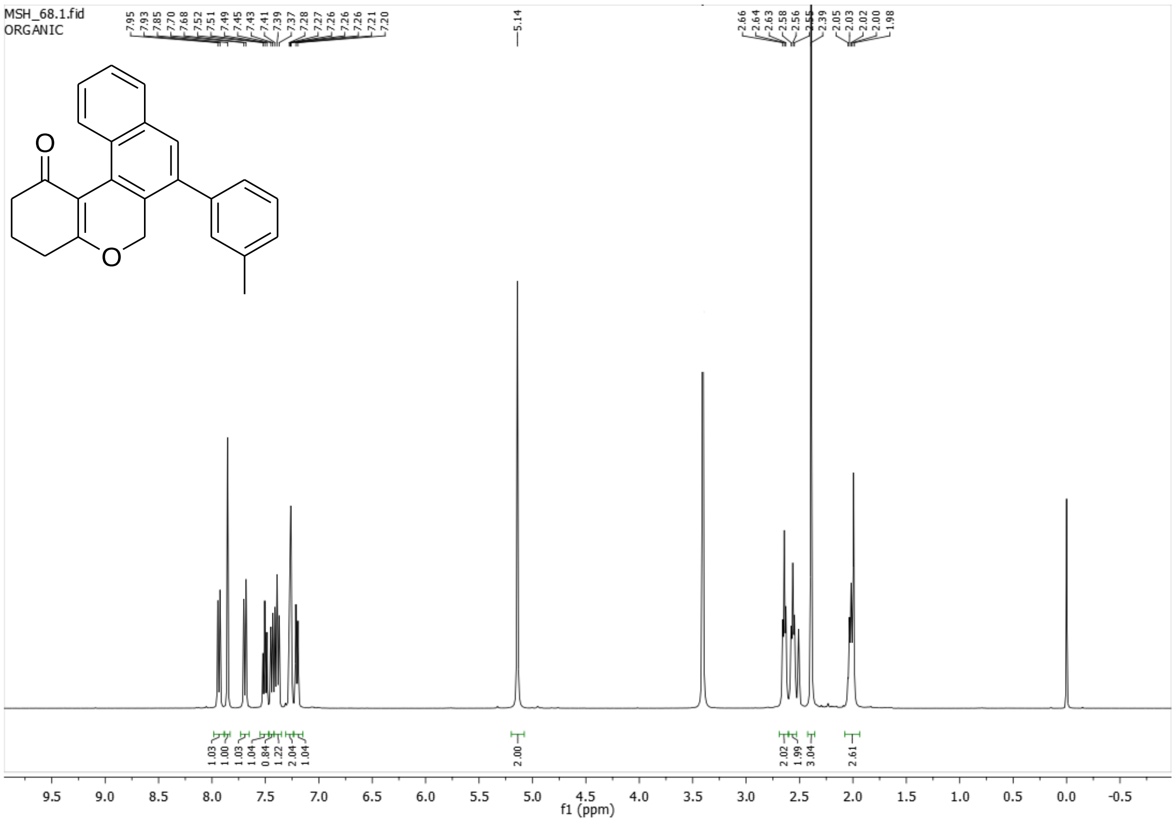
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

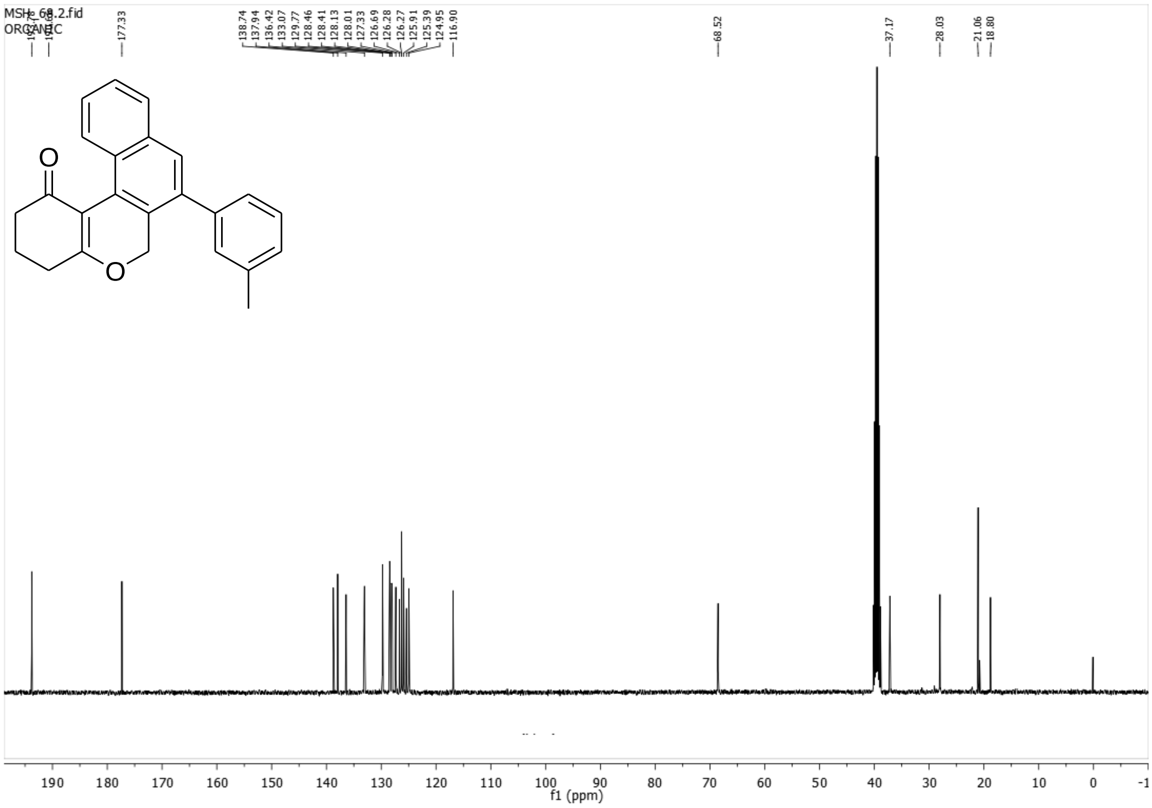


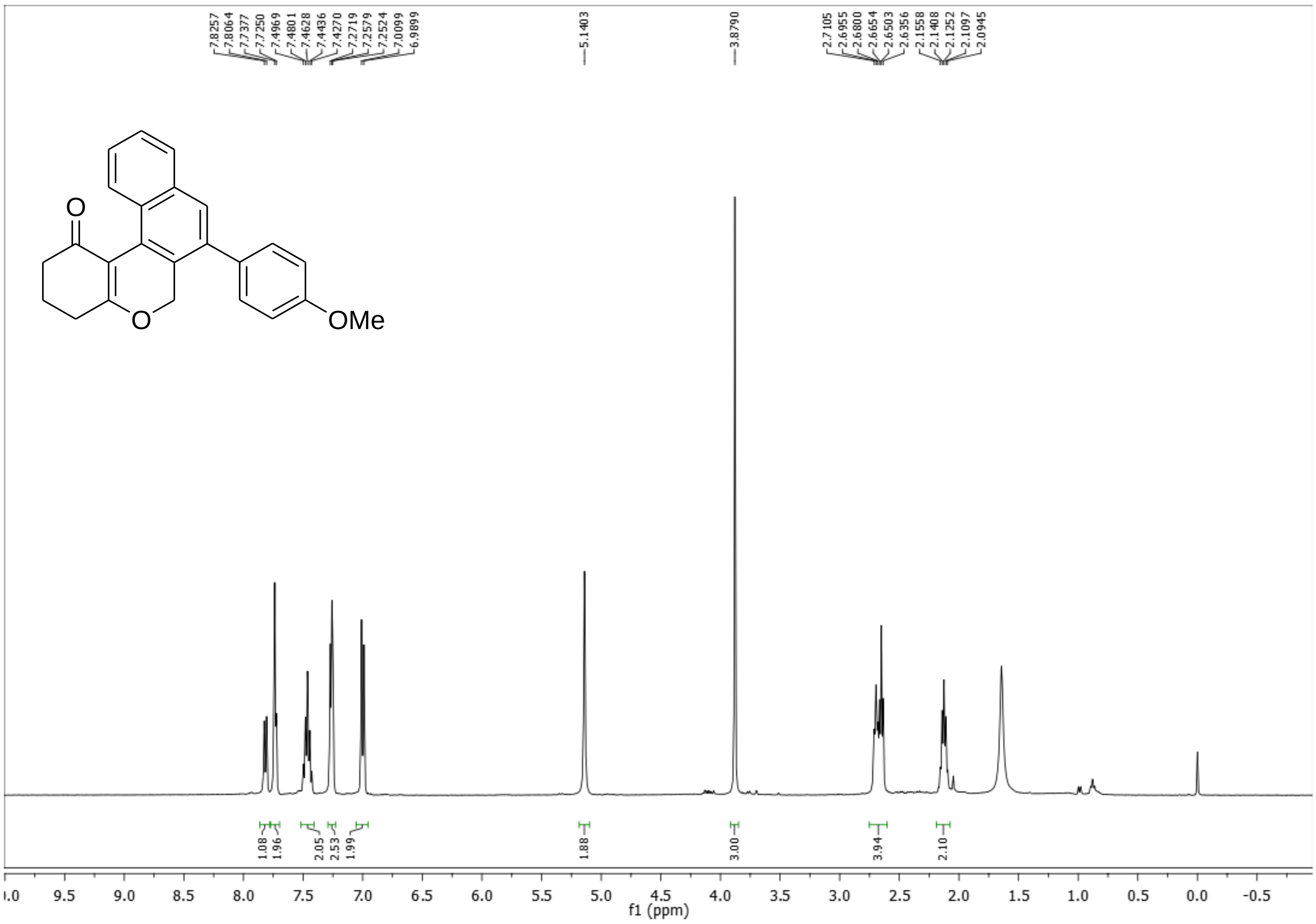



MSH_67.1.fid
ORGANIC
7.94
7.92
7.84
7.69
7.66
7.52
7.50
7.49
7.44
7.42
7.40
7.32
5.15
2.67
2.66
2.64
2.58
2.56
2.55
2.39
2.06
2.04
2.02
2.01
1.99
O
O
1.07
1.08
1.09
1.13
1.11
4.13
2.00
2.17
2.11
3.28
2.12
f1 (ppm)


MSH_67.2.fid
ORGANIC
177.31
137.09
136.26
135.88
133.07
129.22
129.06
128.09
127.95
127.30
126.62
126.34
125.88
125.36
124.89
116.88
68.49
37.16
28.03
20.81
18.80
O
O
f1 (ppm)



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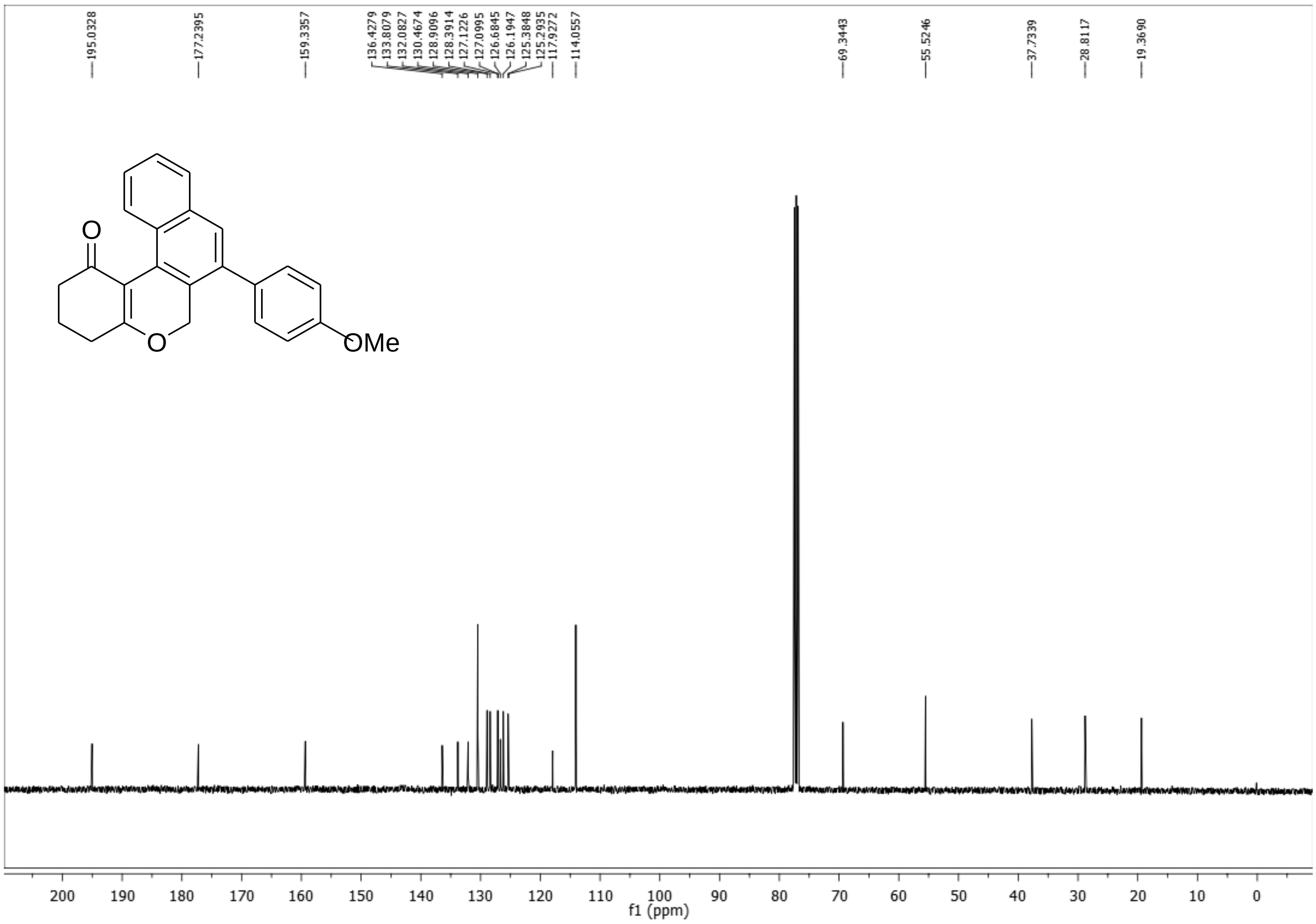
7.8064
7.7377
7.7250
7.4969
7.4801
7.4628
7.4436
7.4270
7.2719
7.2579
7.2524
7.0099
6.9899
5.1403
3.8790
2.7105
2.6955
2.6800
2.6654
2.6503
2.6356
2.1558
2.1408
2.1252
2.1097
2.0945
O
O
OMe
1.08
1.96
2.05
2.53
1.99
1.88
3.00
3.94
2.10
f1 (ppm)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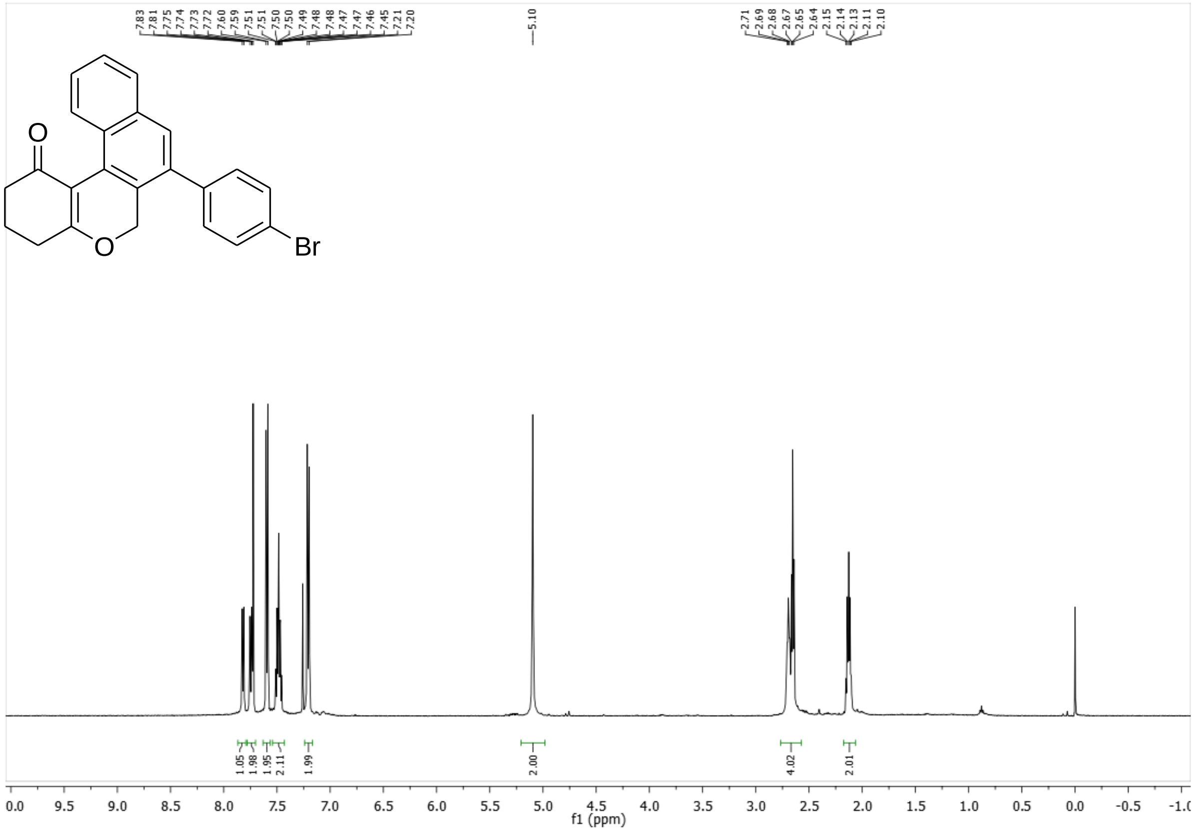
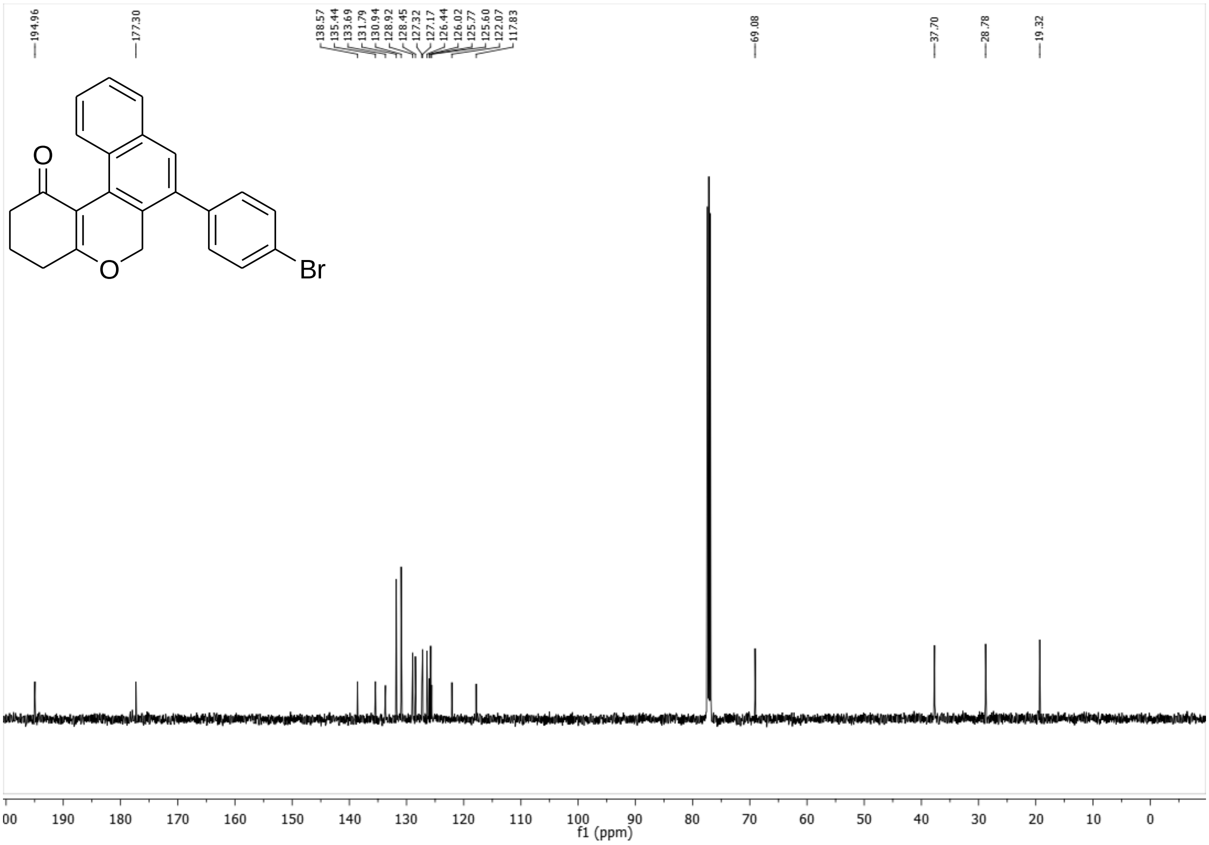
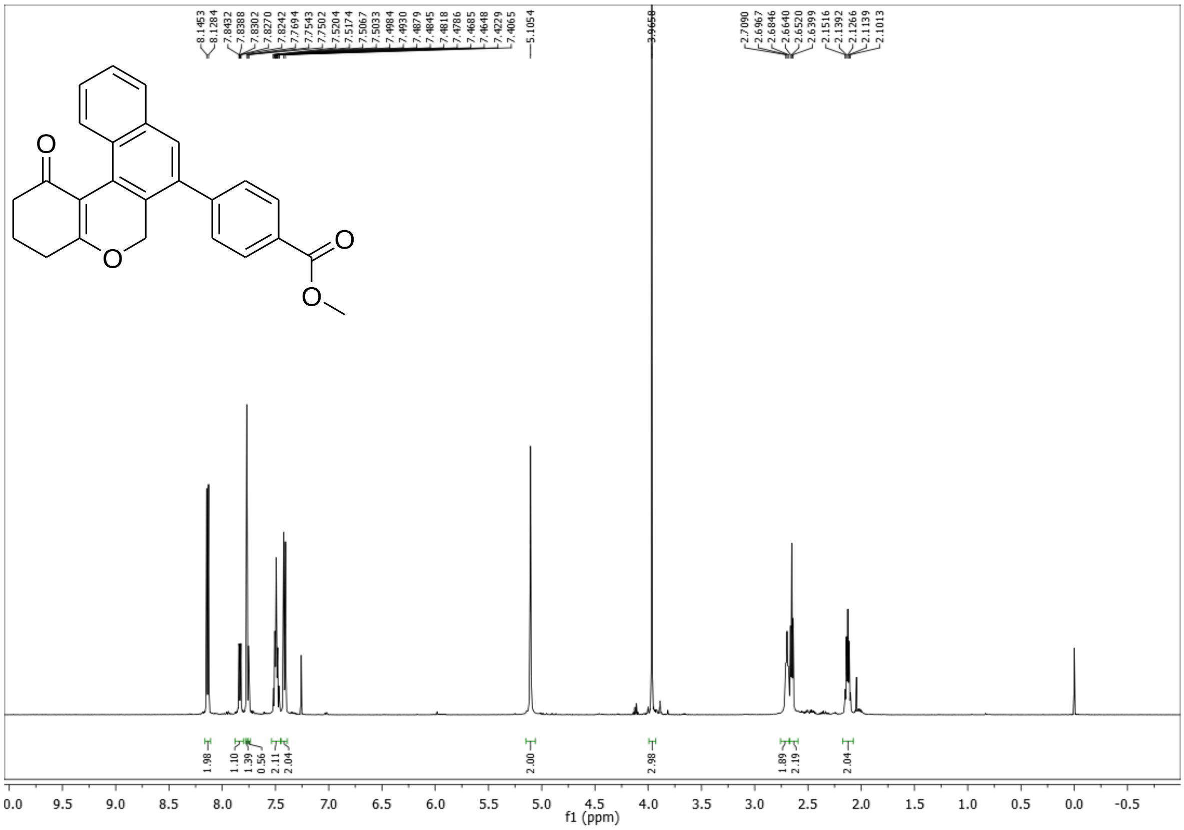
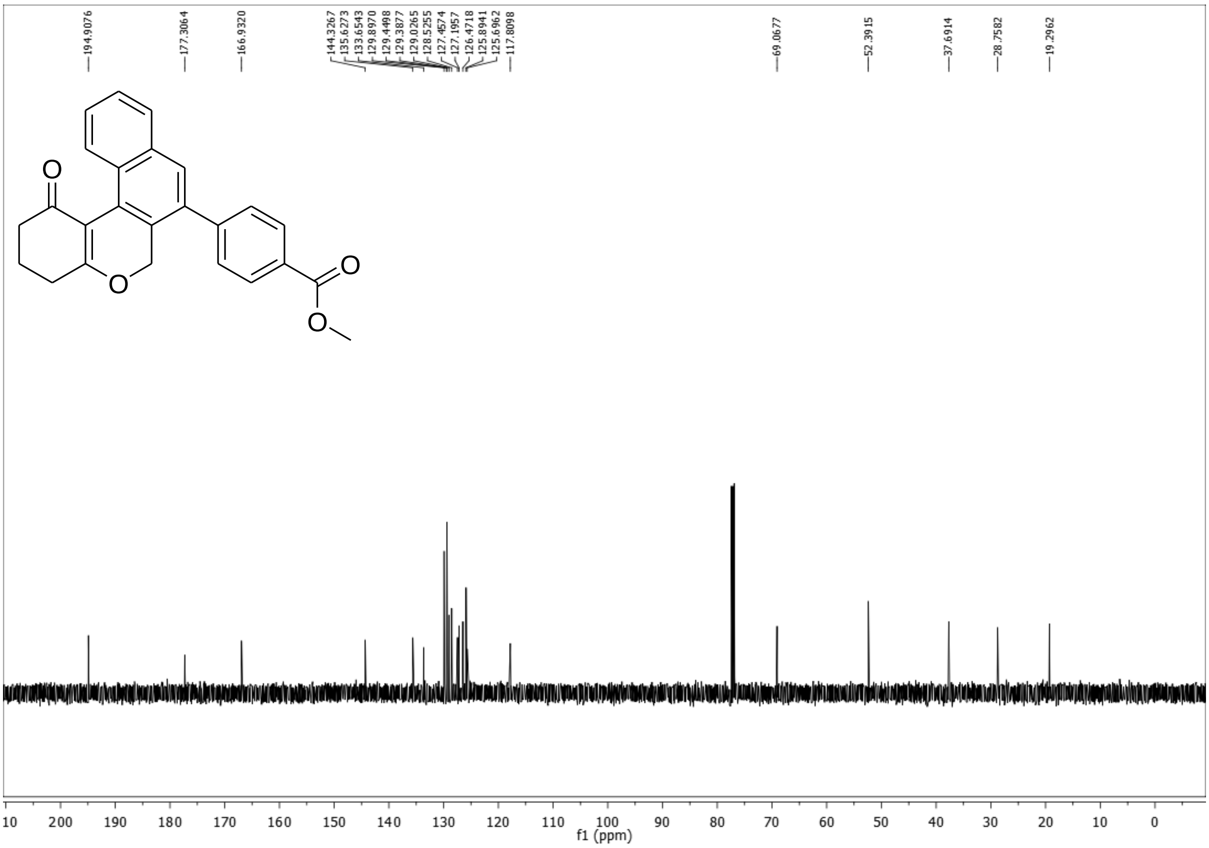
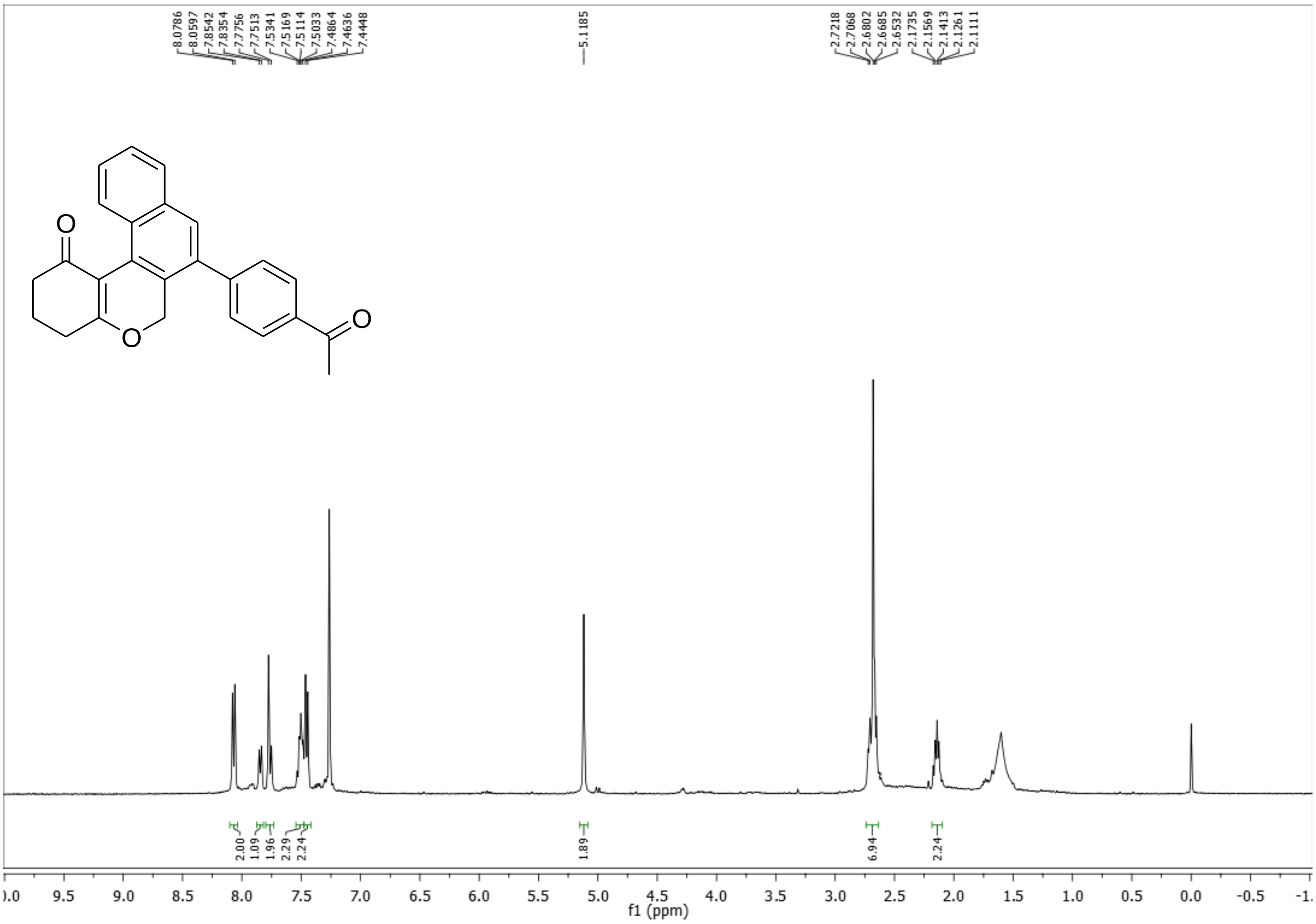
177.2395
159.3357
136.4279
133.8079
132.0827
130.4674
128.9096
128.3914
127.1226
127.0995
126.6845
126.1947
125.3848
125.2935
117.9272
114.0557
69.3443
55.5246
37.7339
28.8117
19.3690
O
O
OMe
f1 (ppm)

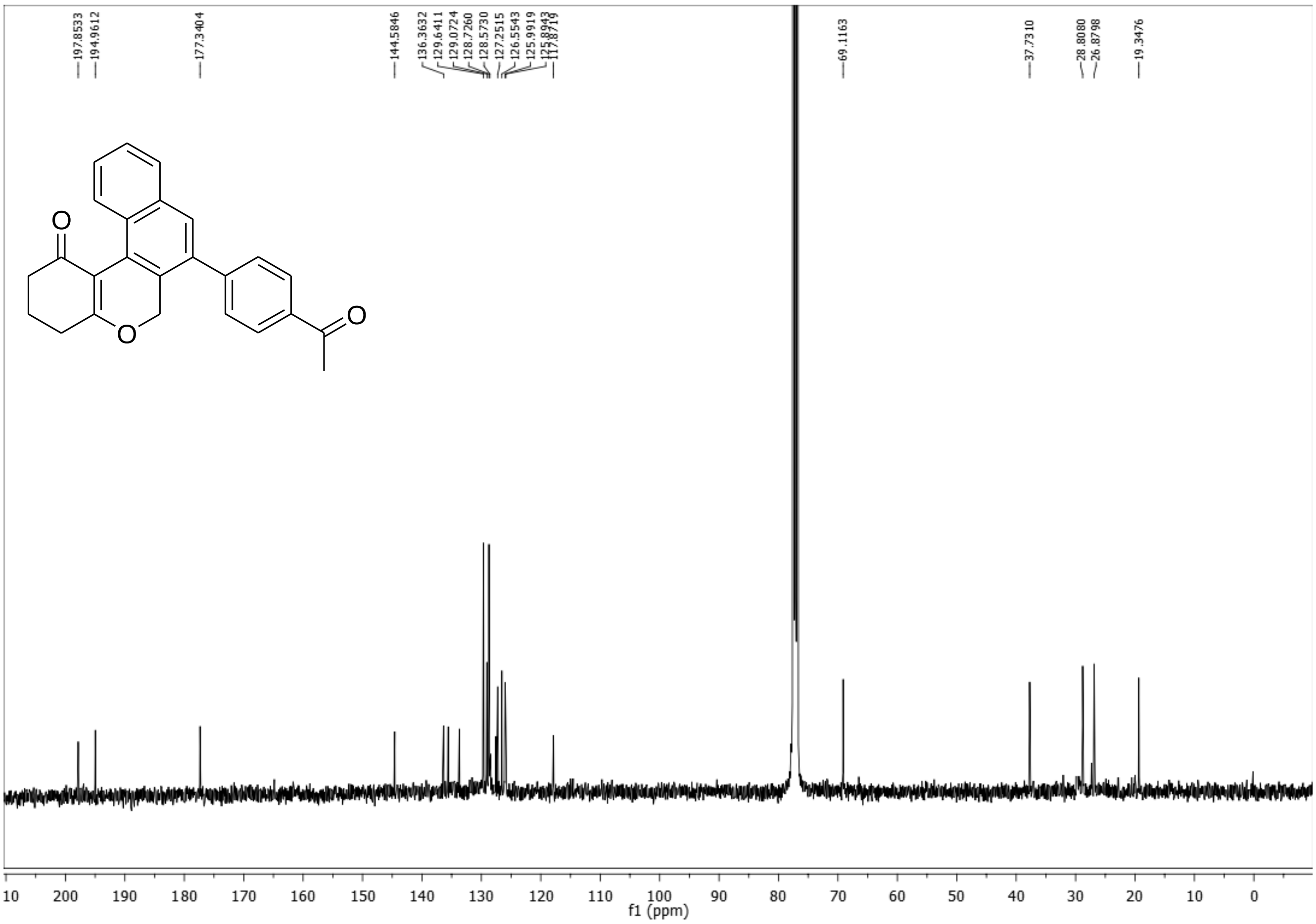

O
O
Br
f1 (ppm)

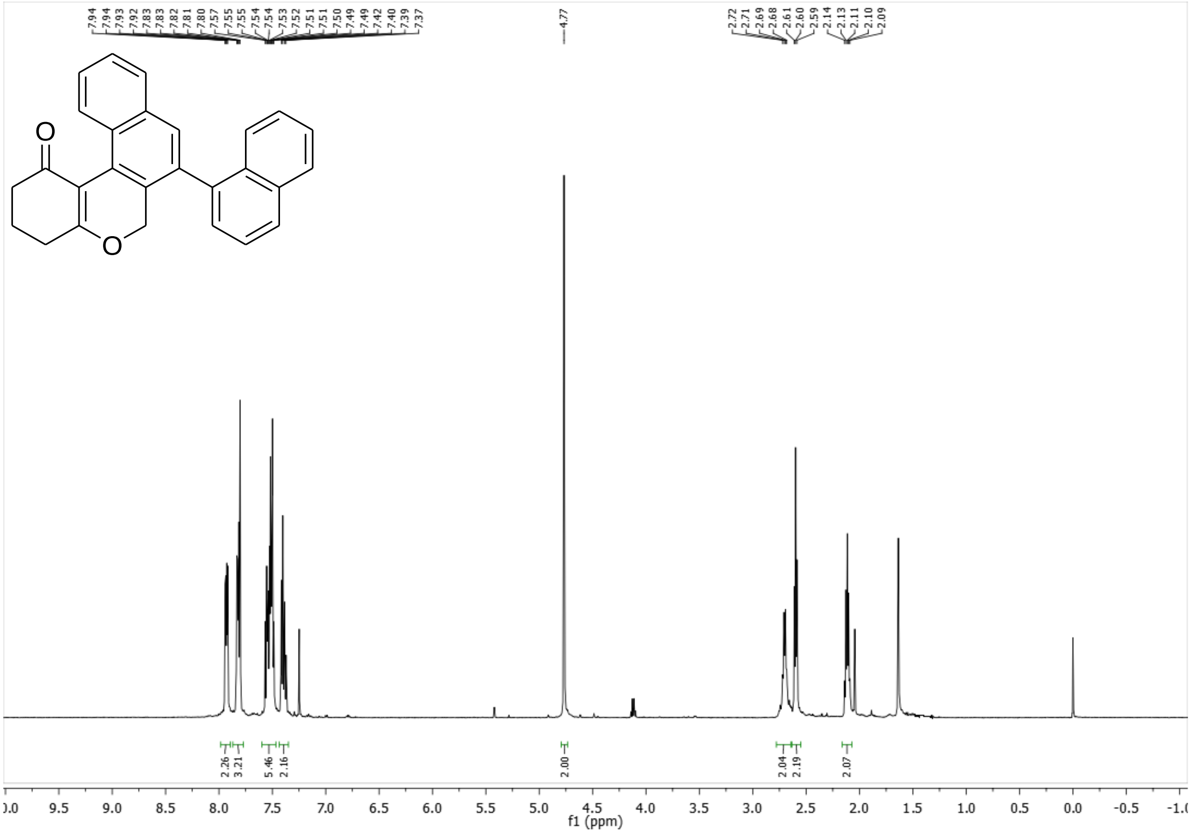

O
O
Br
f1 (ppm)

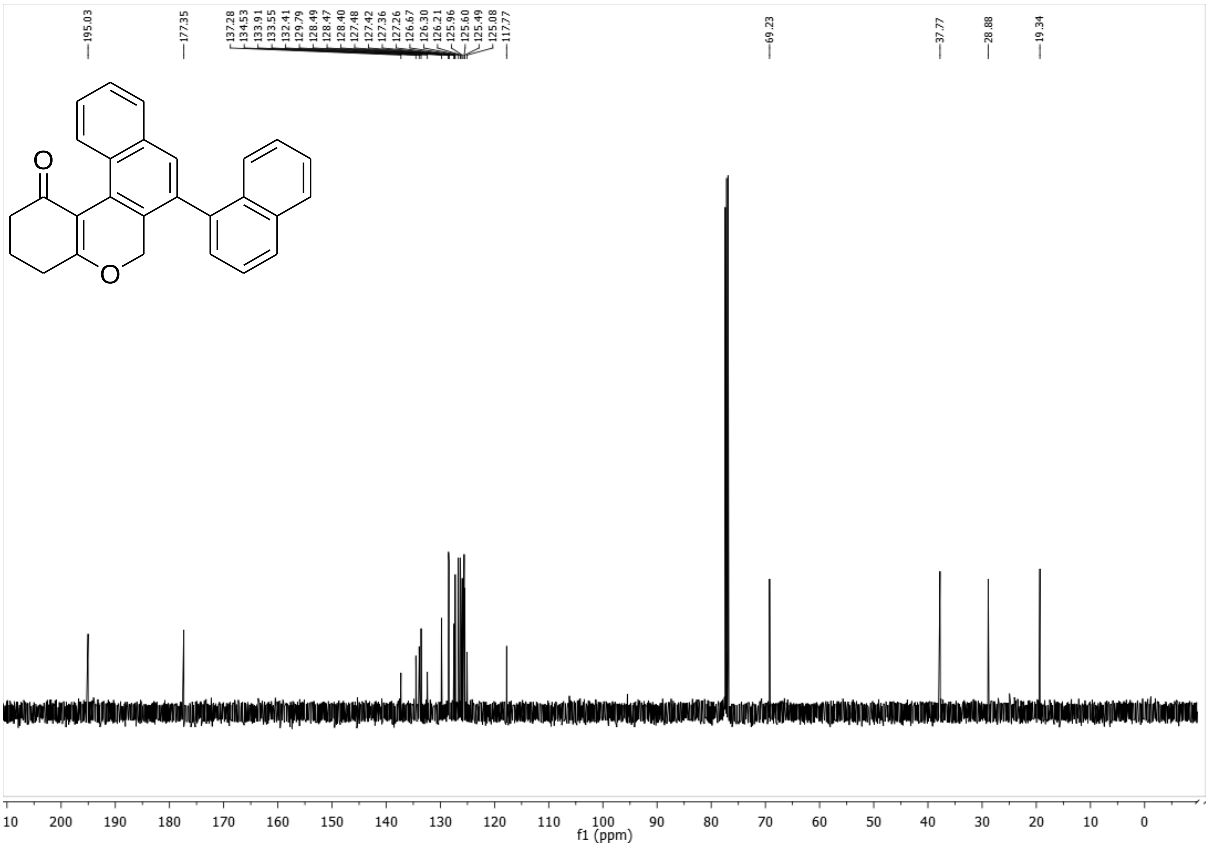

O
O
O
O
1.98
1.10
1.39
0.56
2.11
2.04
2.00
2.98
1.89
2.19
2.04
f1 (ppm)


O
O
O
O
194.9076
177.3064
166.9320
69.0677
52.3915
37.6914
28.7582
19.2962
f1 (ppm)






7.94
7.94
7.93
7.92
7.83
7.83
7.82
7.81
7.80
7.57
7.55
7.55
7.54
7.54
7.53
7.52
7.51
7.51
7.50
7.49
7.49
7.42
7.40
7.39
7.37
4.77
2.72
2.71
2.69
2.68
2.61
2.60
2.59
2.14
2.13
2.11
2.10
2.09
O
O
2.26
3.21
5.46
2.16
2.00
2.04
2.19
2.07
f1 (ppm)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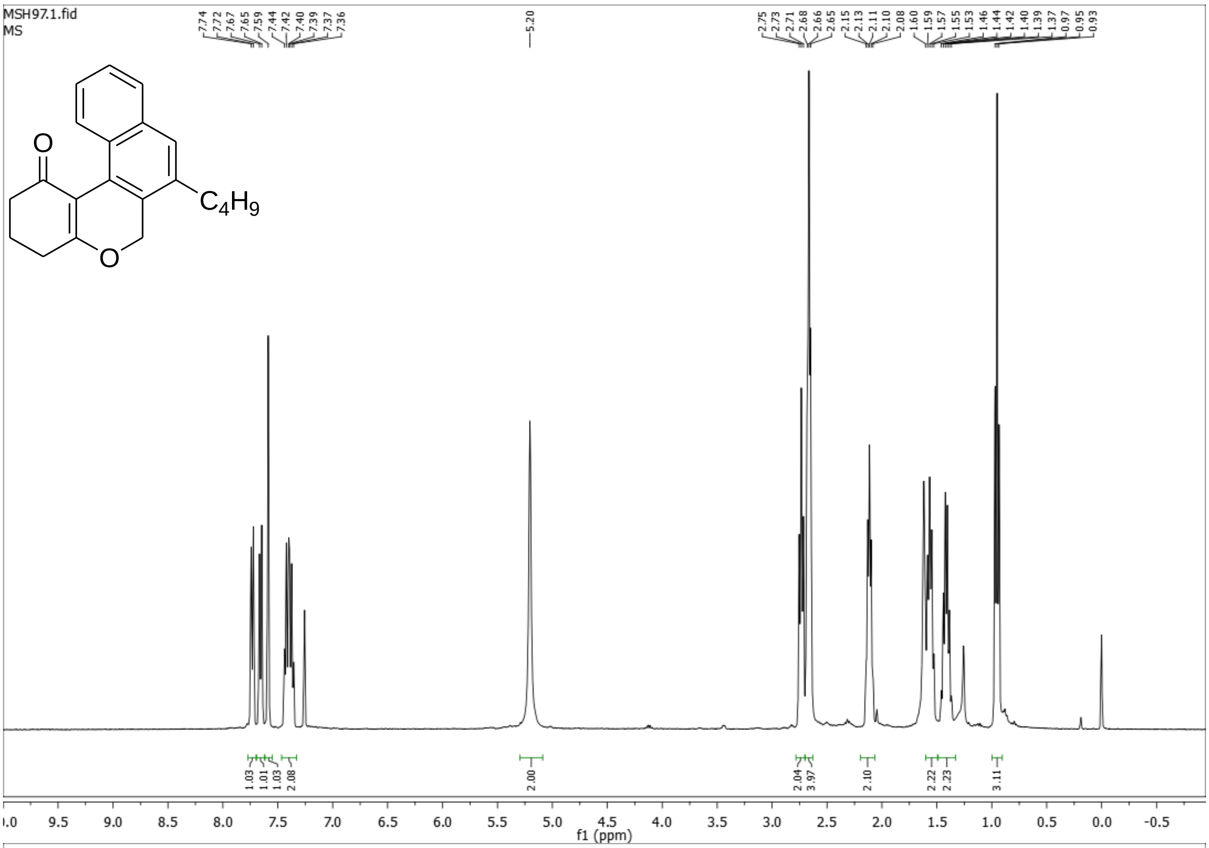
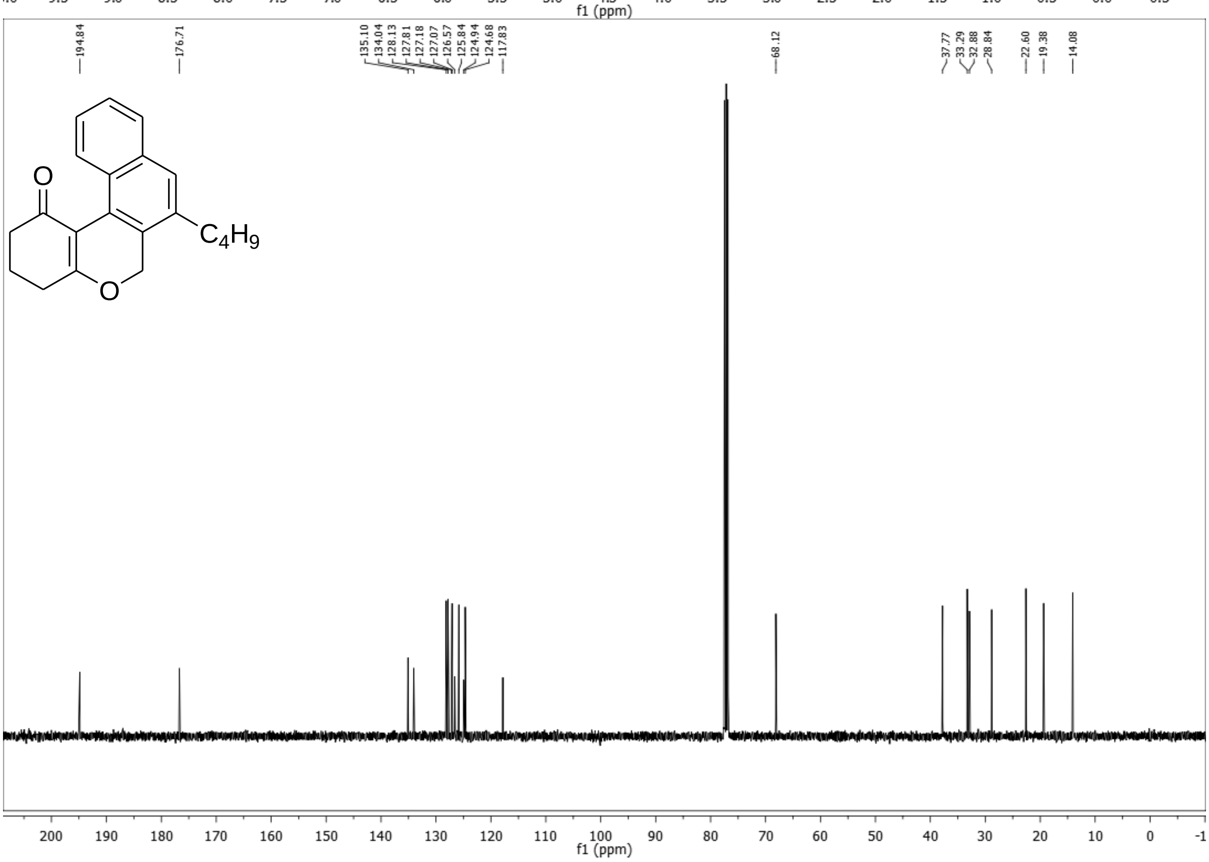
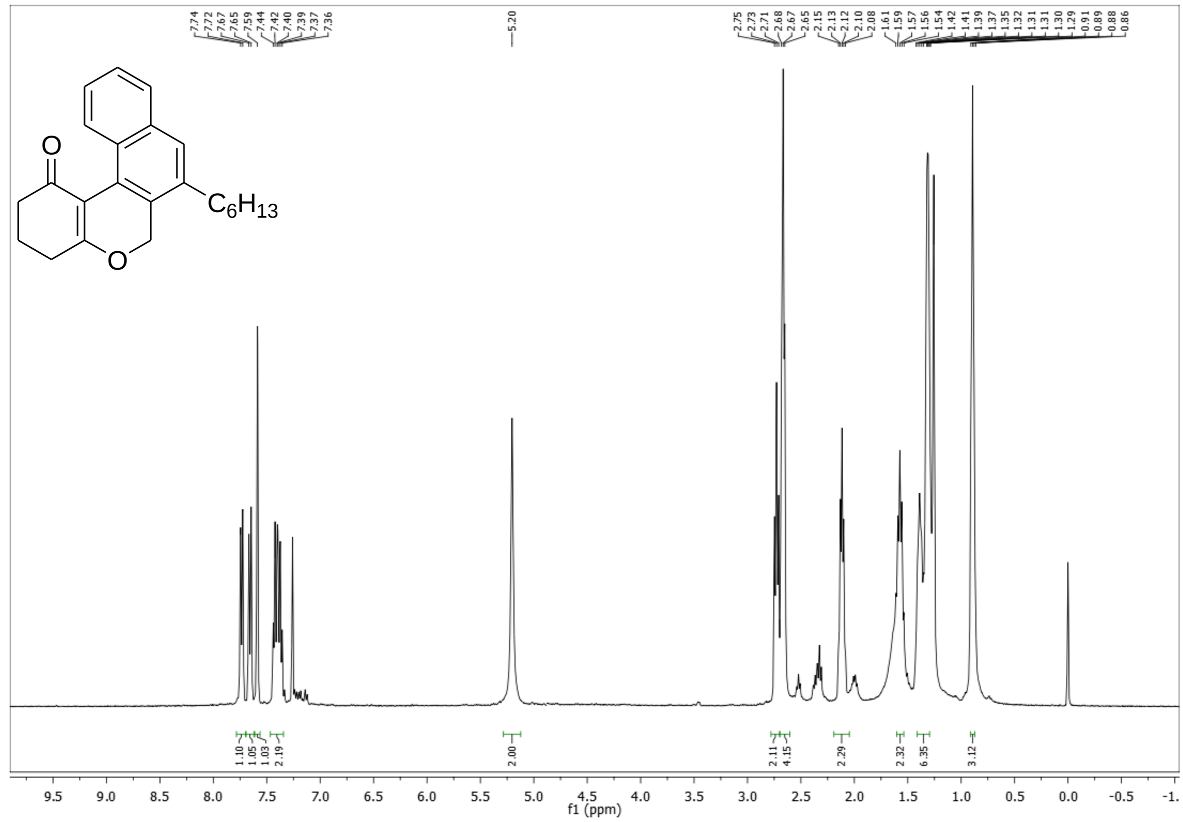
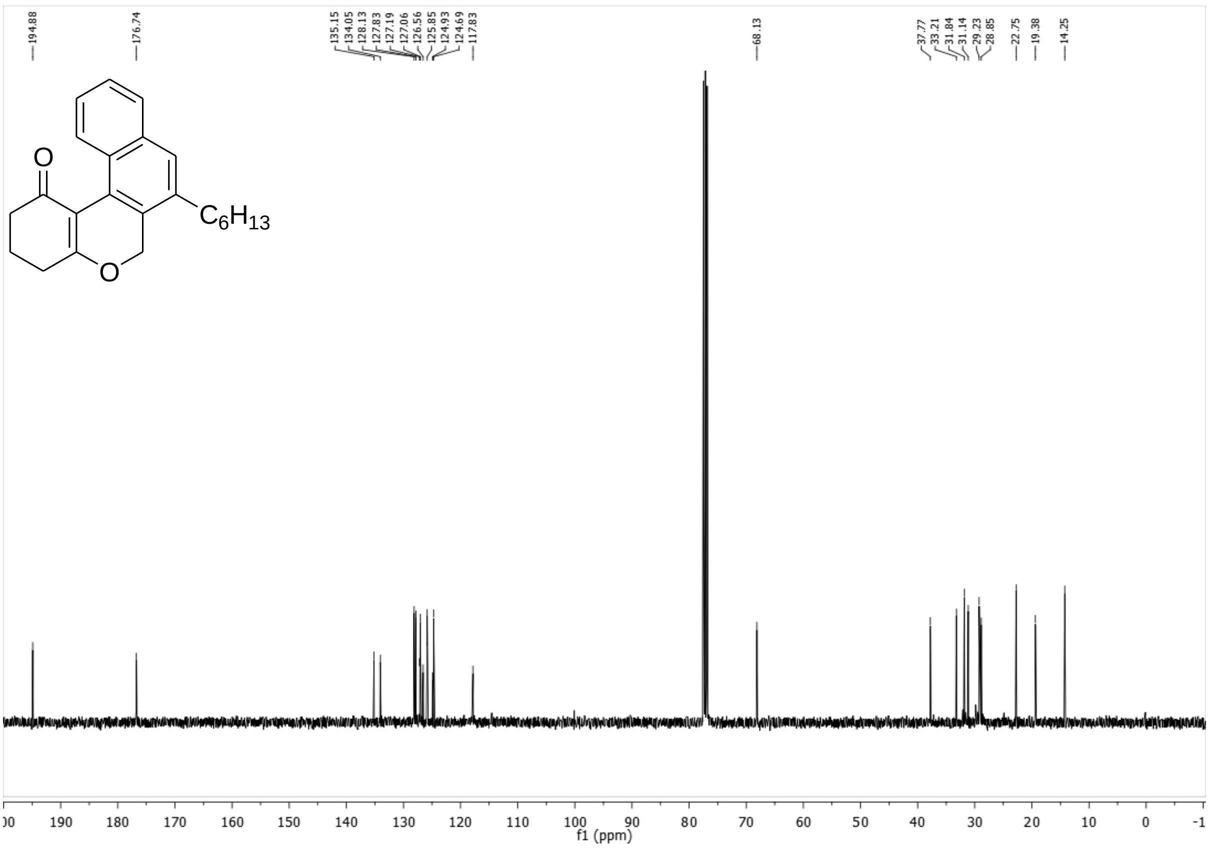
177.35
137.28
134.53
133.91
133.55
132.41
129.79
128.49
128.47
128.40
127.48
127.42
127.36
127.26
126.67
126.30
126.21
125.96
125.60
125.49
125.08
117.77
69.23
37.77
28.88
19.34
O
O
f1 (ppm)

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

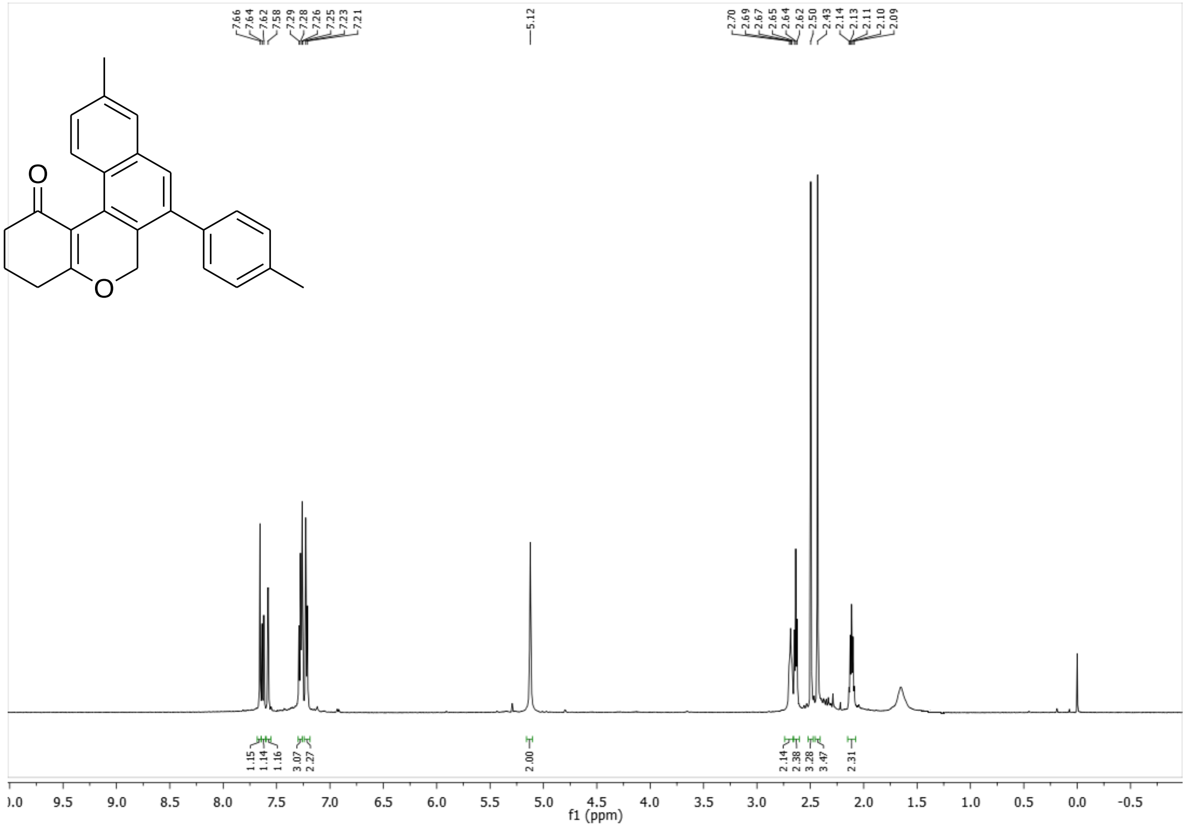


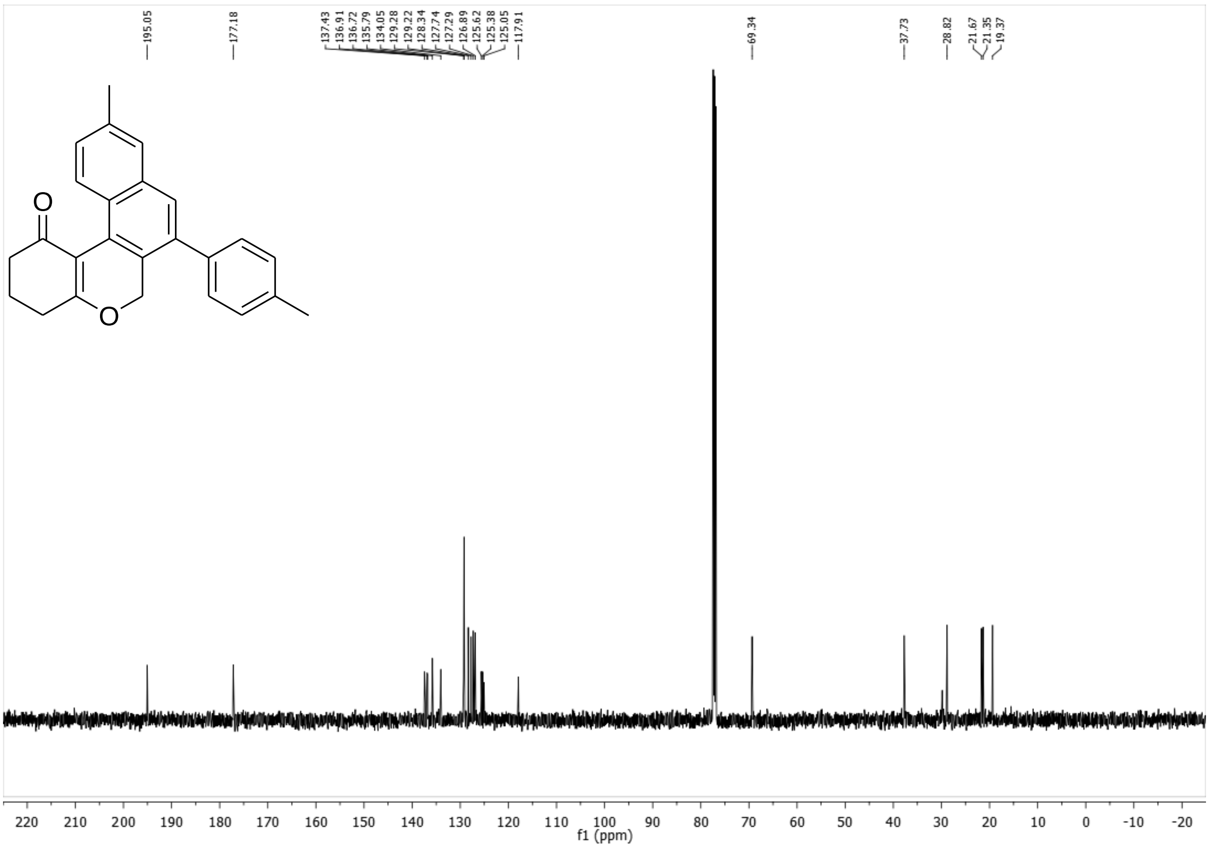


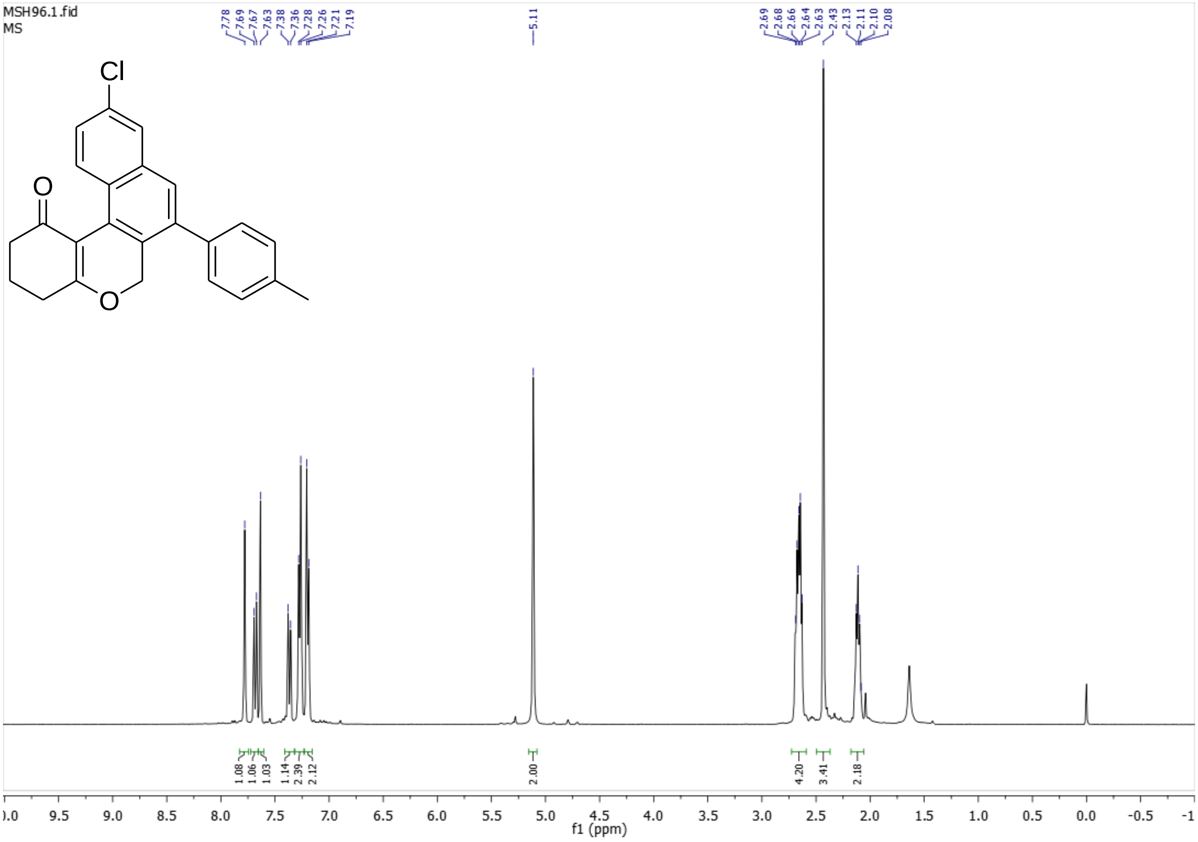


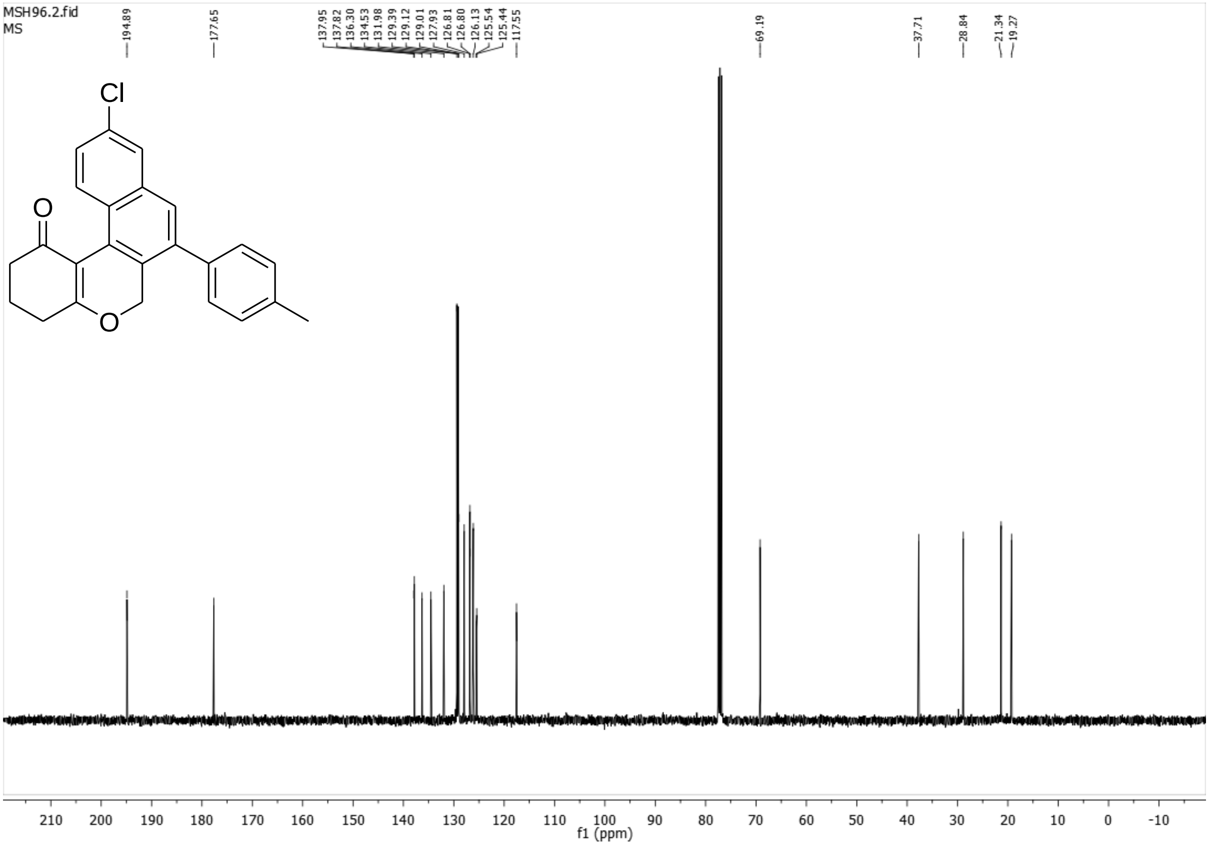


Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry






MSH96.1.fid
MS
Cl
O
O
7.78
7.69
7.67
7.63
7.38
7.36
7.28
7.26
7.21
7.19
5.11
2.69
2.68
2.66
2.64
2.63
2.43
2.13
2.11
2.10
2.08
1.08
1.06
1.03
1.14
2.39
2.12
2.00
4.20
3.41
2.18
f1 (ppm)


MSH96.2.fid
MS
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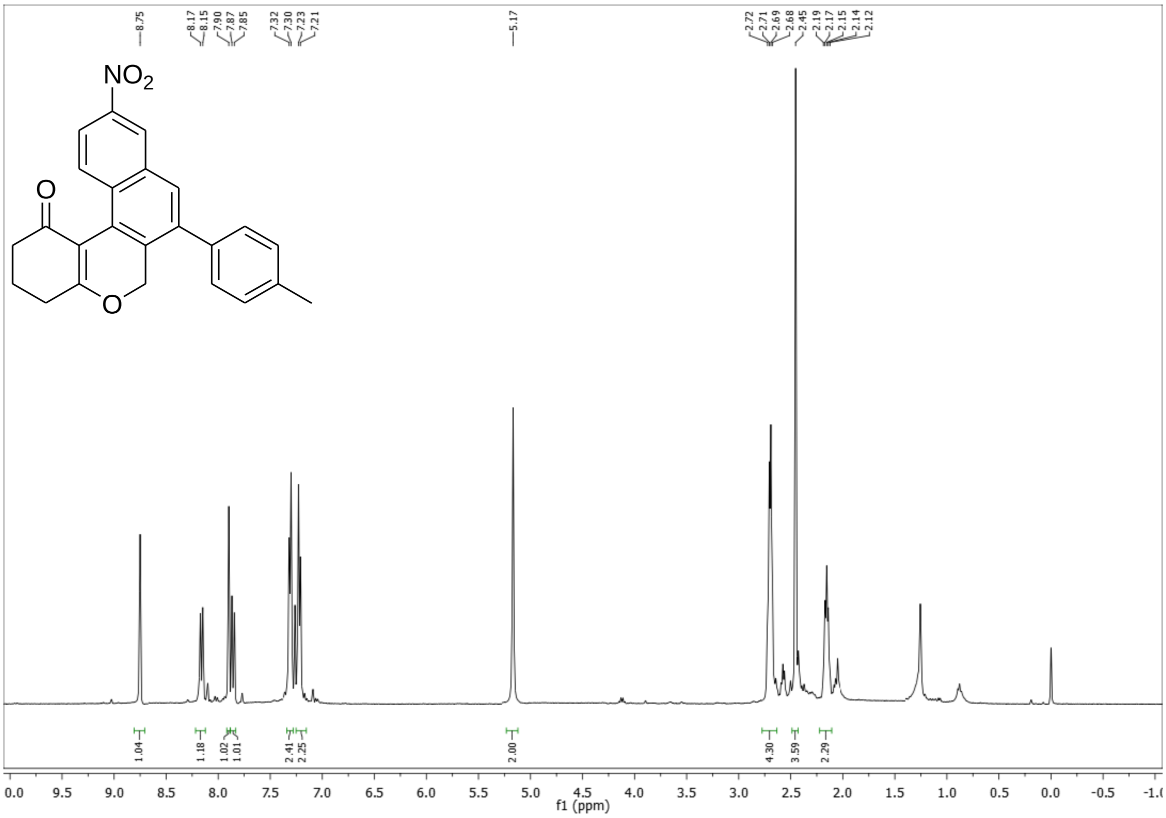
177.65
137.95
137.82
136.30
134.53
131.98
129.39
129.12
129.01
127.93
126.81
126.80
126.13
125.54
125.44
117.55
69.19
37.71
28.84
21.34
19.27
Cl
O
O
f1 (ppm)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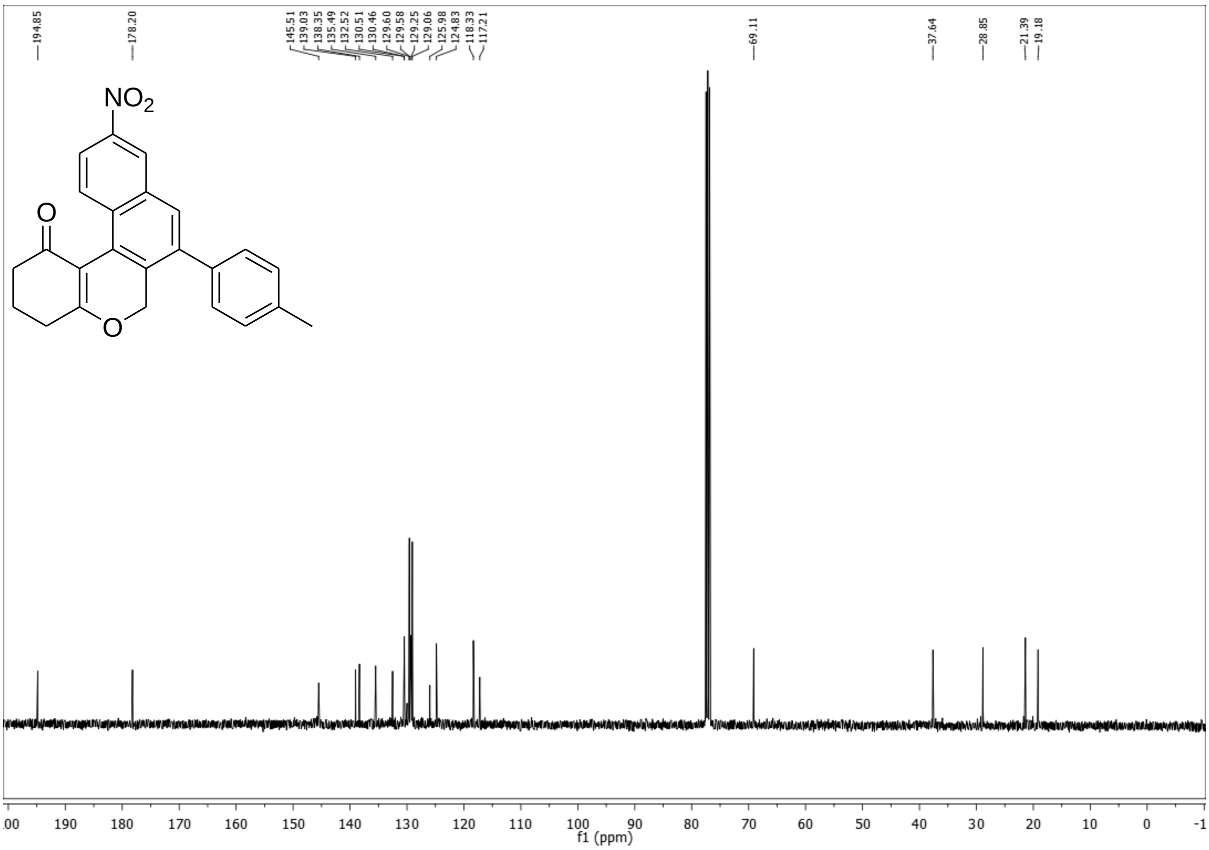
8.17
8.15
7.90
7.87
7.85
7.32
7.30
7.23
7.21
5.17
2.72
2.71
2.69
2.68
2.45
2.19
2.17
2.15
2.14
2.12
NO_2
O
O
1.04
1.18
1.02
1.01
2.41
2.25
2.00
4.30
3.59
2.29
f1 (ppm)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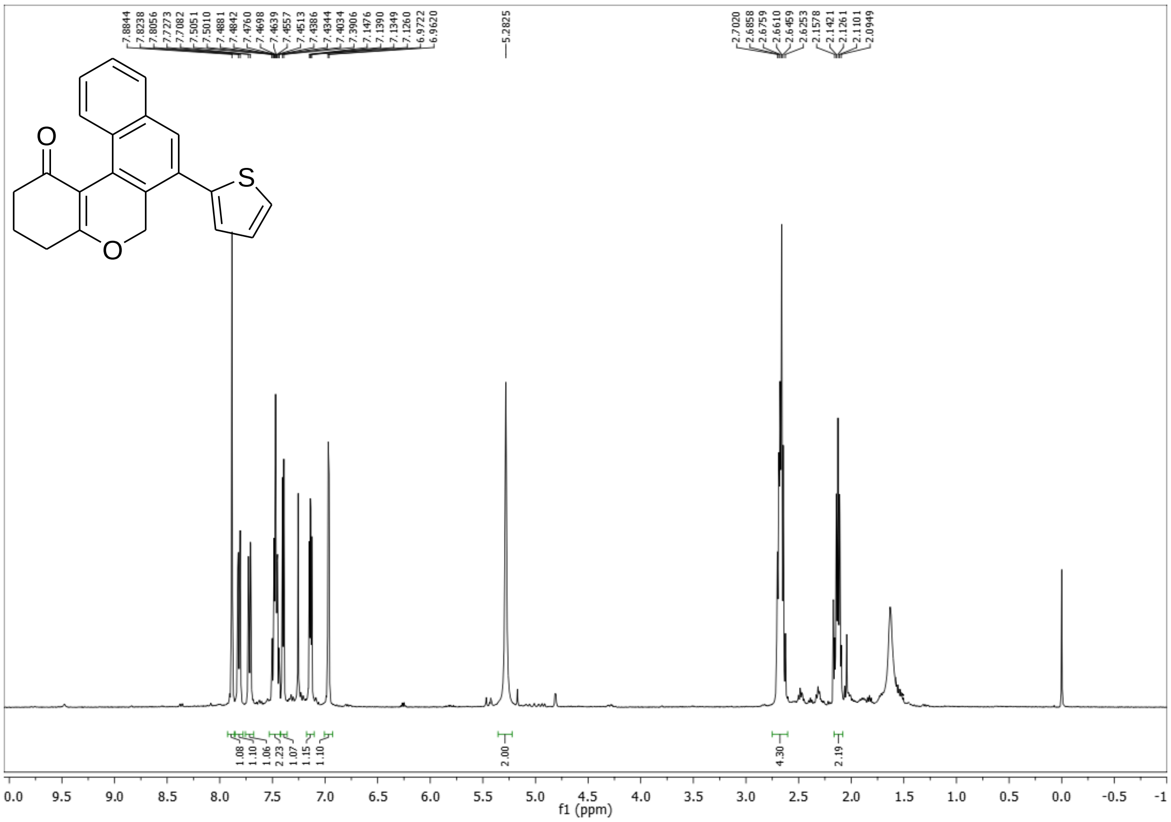
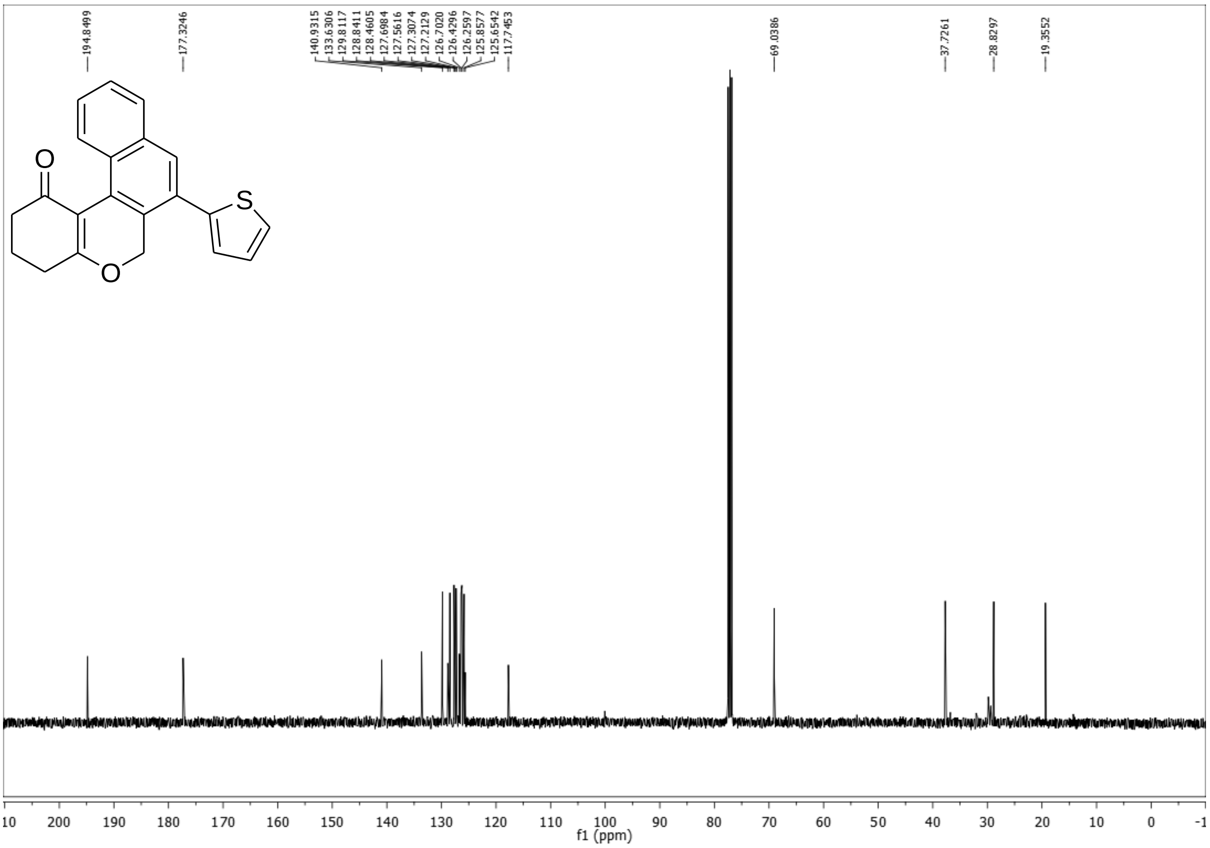
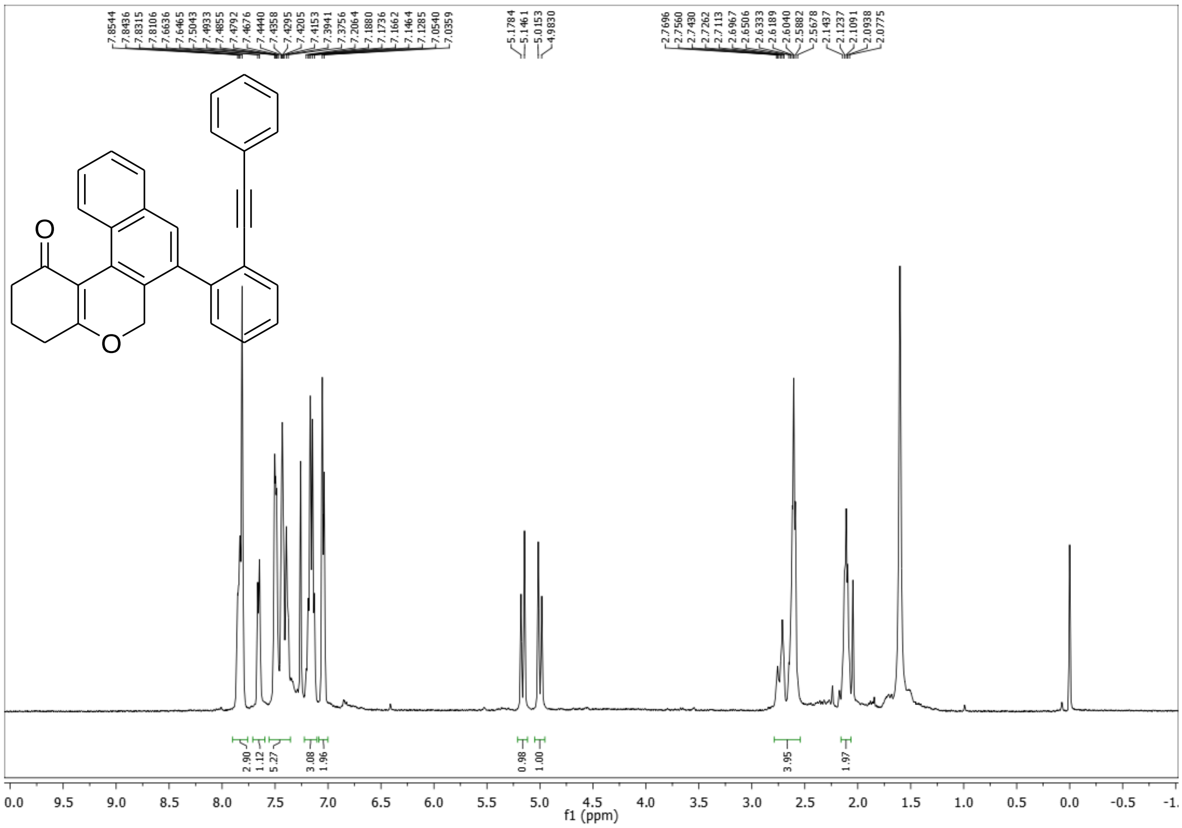
178.20
145.51
139.03
138.35
135.49
132.52
130.51
130.46
129.60
129.58
129.25
129.06
125.98
124.83
118.33
117.21
69.11
37.64
28.85
21.39
19.18
NO_2
O
O
f1 (ppm)

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry



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7.8436
7.8315
7.8106
7.6636
7.6465
7.5043
7.4933
7.4855
7.4792
7.4676
7.4440
7.4358
7.4295
7.4205
7.4153
7.3941
7.3756
7.2064
7.1880
7.1736
7.1662
7.1464
7.1285
7.0540
7.0359
5.1784
5.1461
5.0153
4.9830
2.7696
2.7560
2.7430
2.7262
2.7113
2.6967
2.6506
2.6333
2.6189
2.6040
2.5882
2.5678
2.1437
2.1237
2.1091
2.0938
2.0775
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O
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1.12
5.27
3.08
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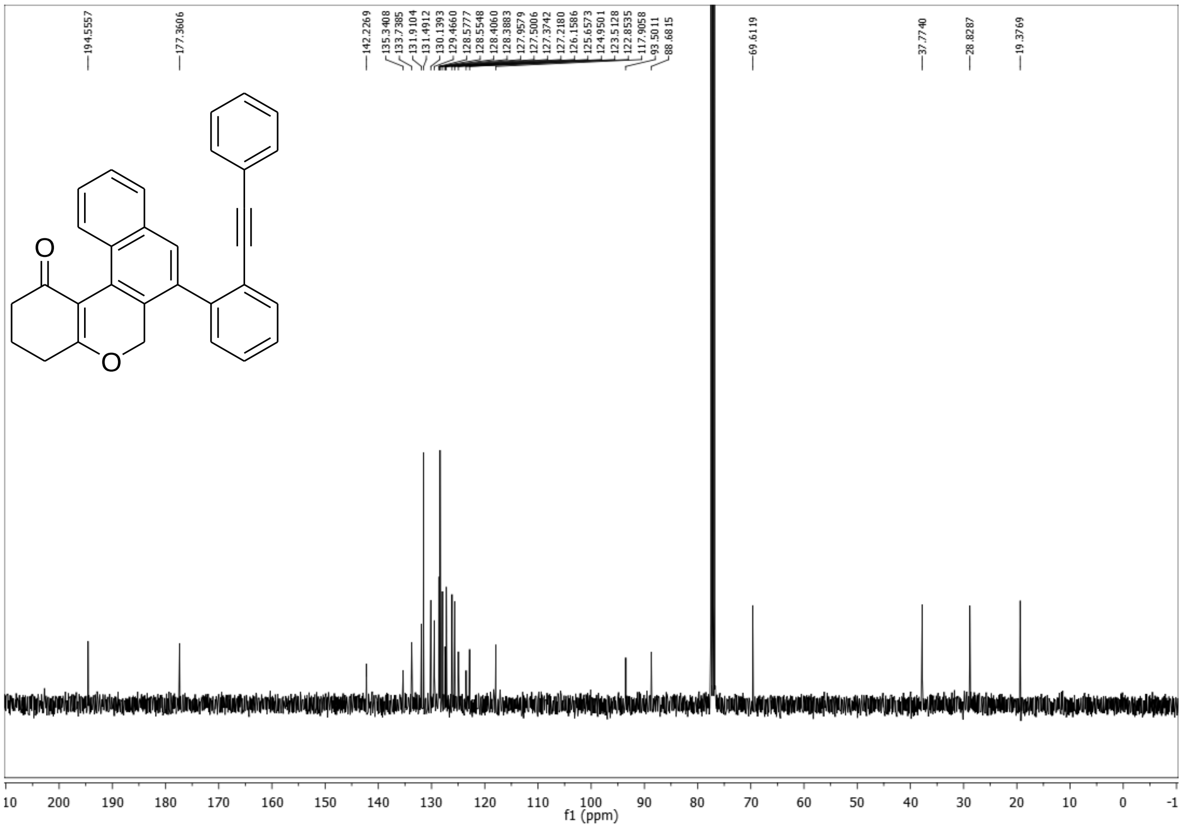
1.00
3.95
1.97
f1 (ppm)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131.9104
131.4912
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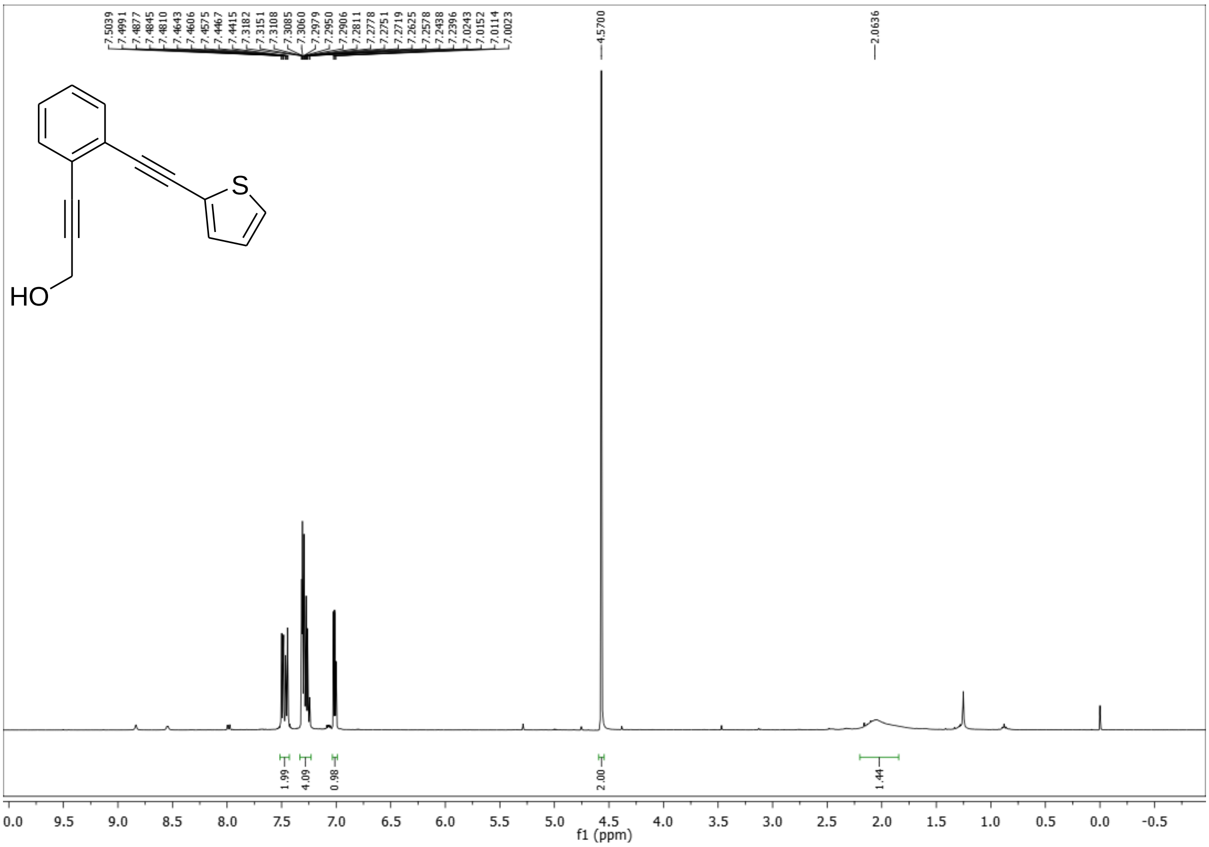
129.4660
128.5777
128.5548
128.4060
128.3883
127.9579
127.5006
127.3742
127.2180
126.1586
125.6573
124.9501
123.5128
122.8535
117.9058
93.5011
88.6815
69.6119
37.7740
28.8287
19.3769
O
O
f1 (ppm)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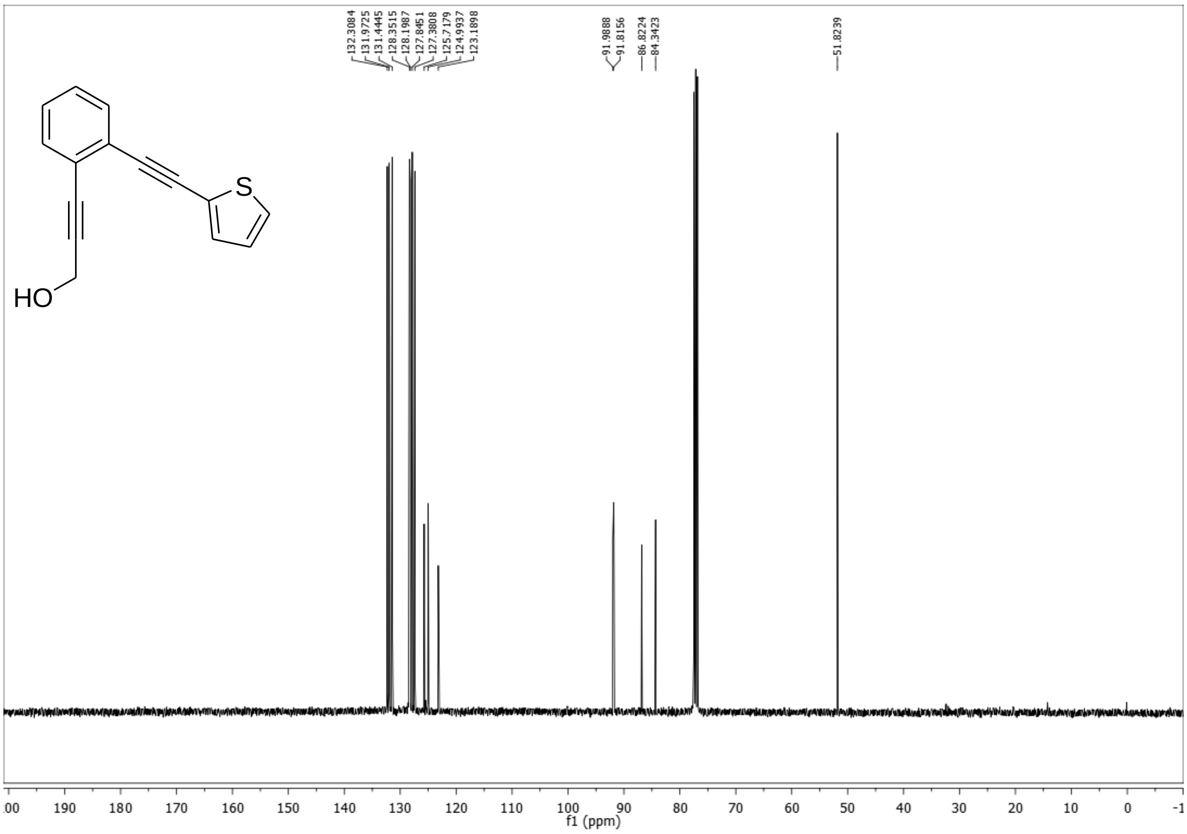
7.4877
7.4845
7.4810
7.4643
7.4606
7.4575
7.4467
7.4415
7.3182
7.3151
7.3108
7.3085
7.3060
7.2979
7.2950
7.2906
7.2811
7.2778
7.2751
7.2719
7.2625
7.2578
7.2438
7.2396
7.0243
7.0152
7.0114
7.0023
4.5700
2.0636
S
HO
1.99
4.09
0.98
2.00
1.44
f1 (ppm)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131.9725
131.4445
128.3515
128.1987
127.8451
127.3808
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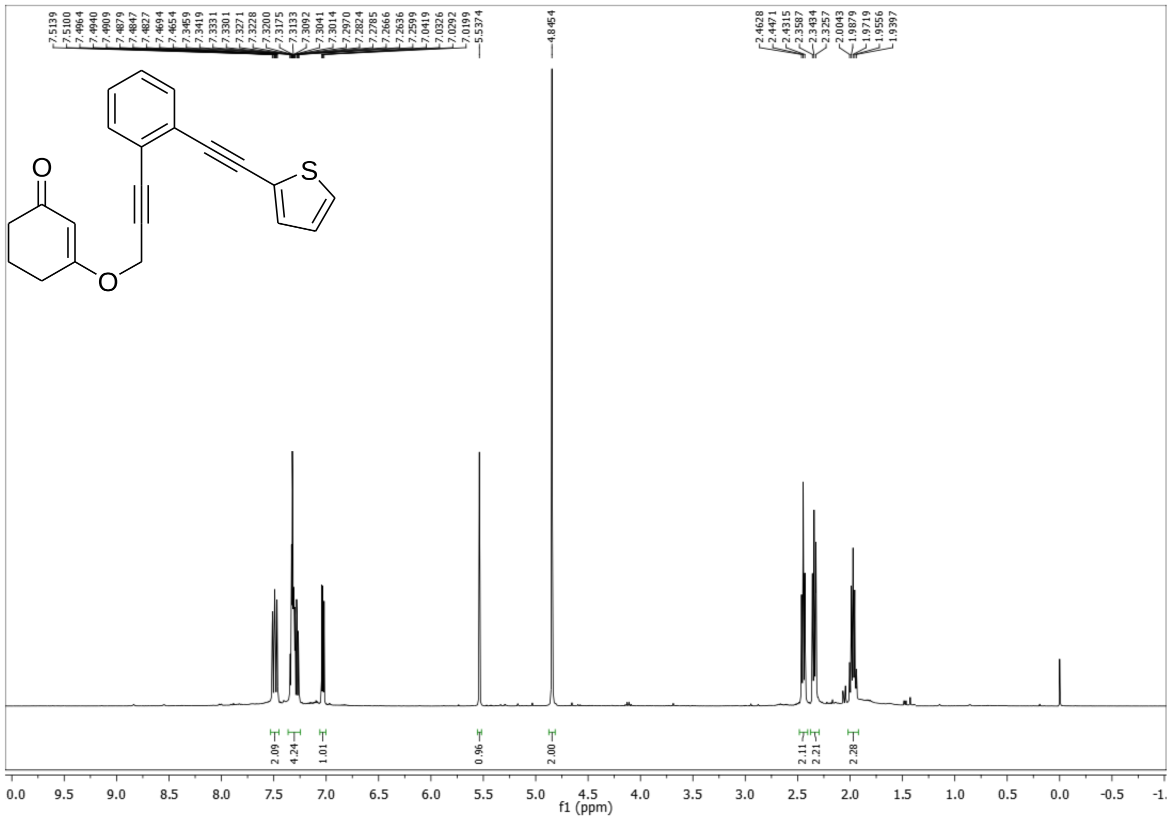
123.1898
91.9888
91.8156
86.8224
84.3423
51.8239
S
HO
f1 (ppm)


7.5139
7.5100
7.4964
7.4940
7.4909
7.4879
7.4847
7.4827
7.4694
7.4654
7.3459
7.3419
7.3331
7.3301
7.3271
7.3228
7.3200
7.3175
7.3133
7.3092
7.3041
7.3014
7.2970
7.2824
7.2785
7.2666
7.2636
7.2599
7.0419
7.0326
7.0292
7.0199
5.5374
4.8454
2.4628
2.4471
2.4315
2.3587
2.3434
2.3257
2.0043
1.9879
1.9719
1.9556
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S
O
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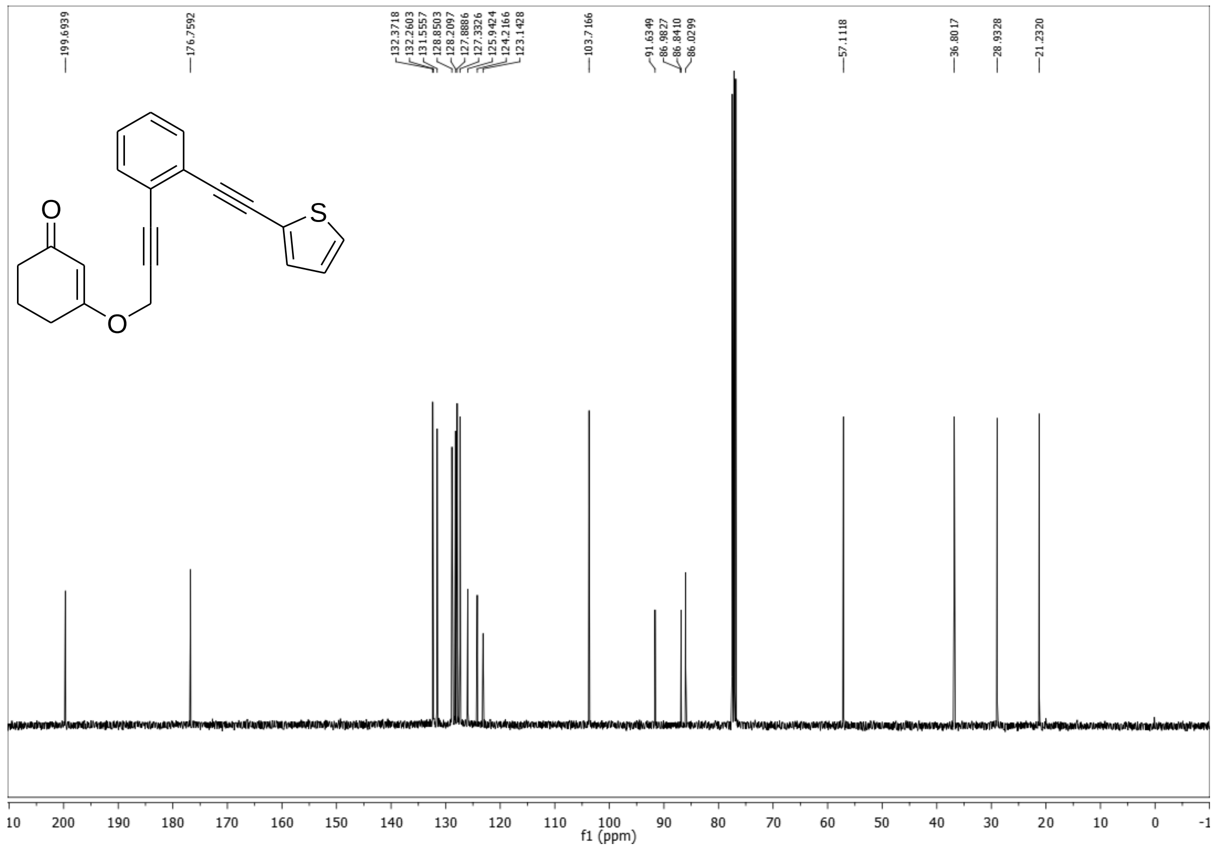
4.24
1.01
0.96
2.00
2.11
2.21
2.28
f1 (ppm)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132.2603
131.5557
128.8503
128.2097
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127.3326
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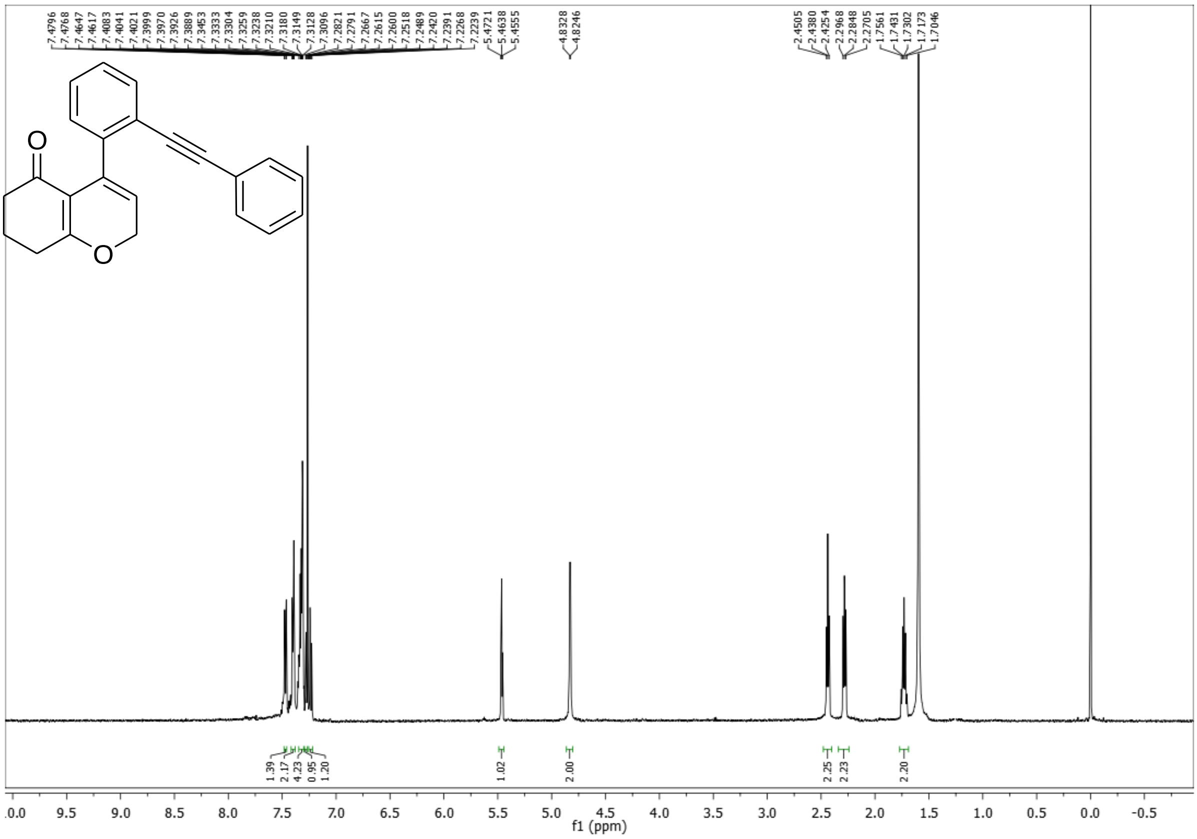
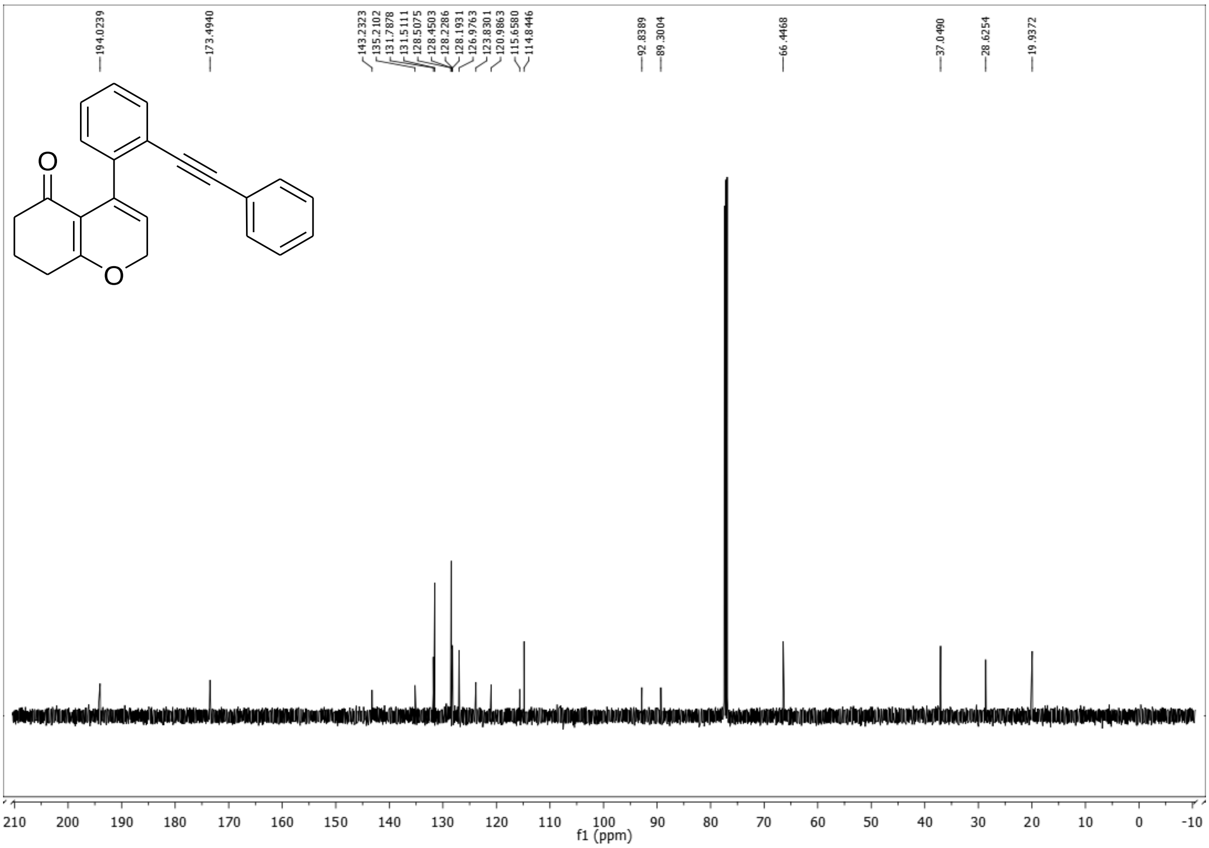
124.2166
123.1428
103.7166
91.6349
86.9827
86.8410
86.0299
57.1118
36.8017
28.9328
21.2320
O
S
O
f1 (ppm)





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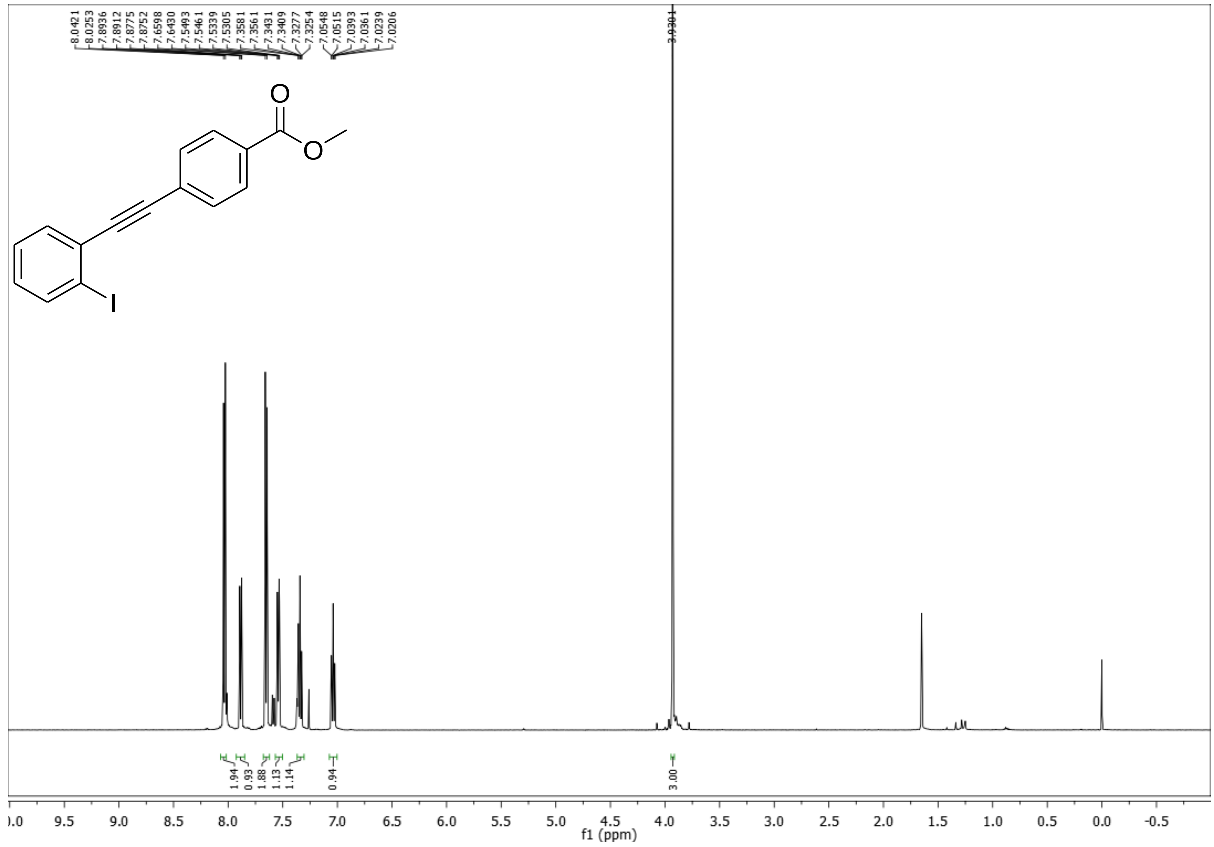

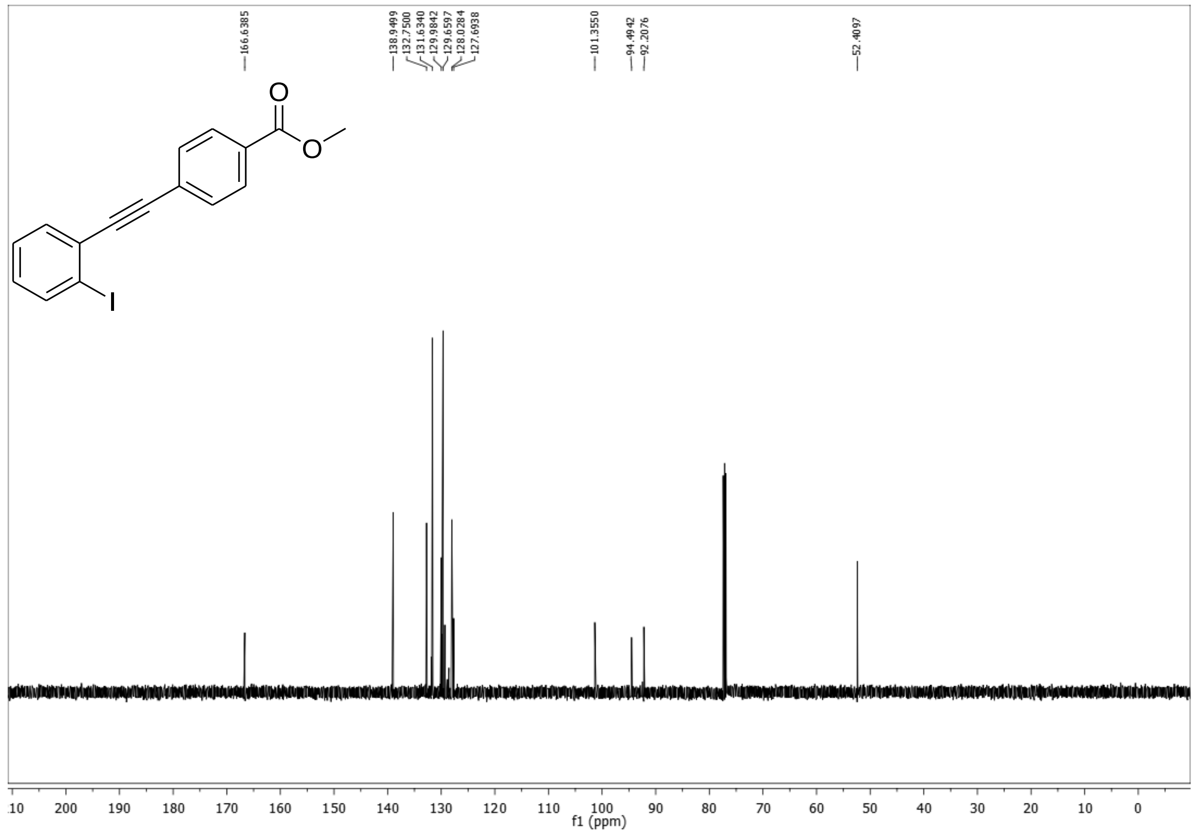

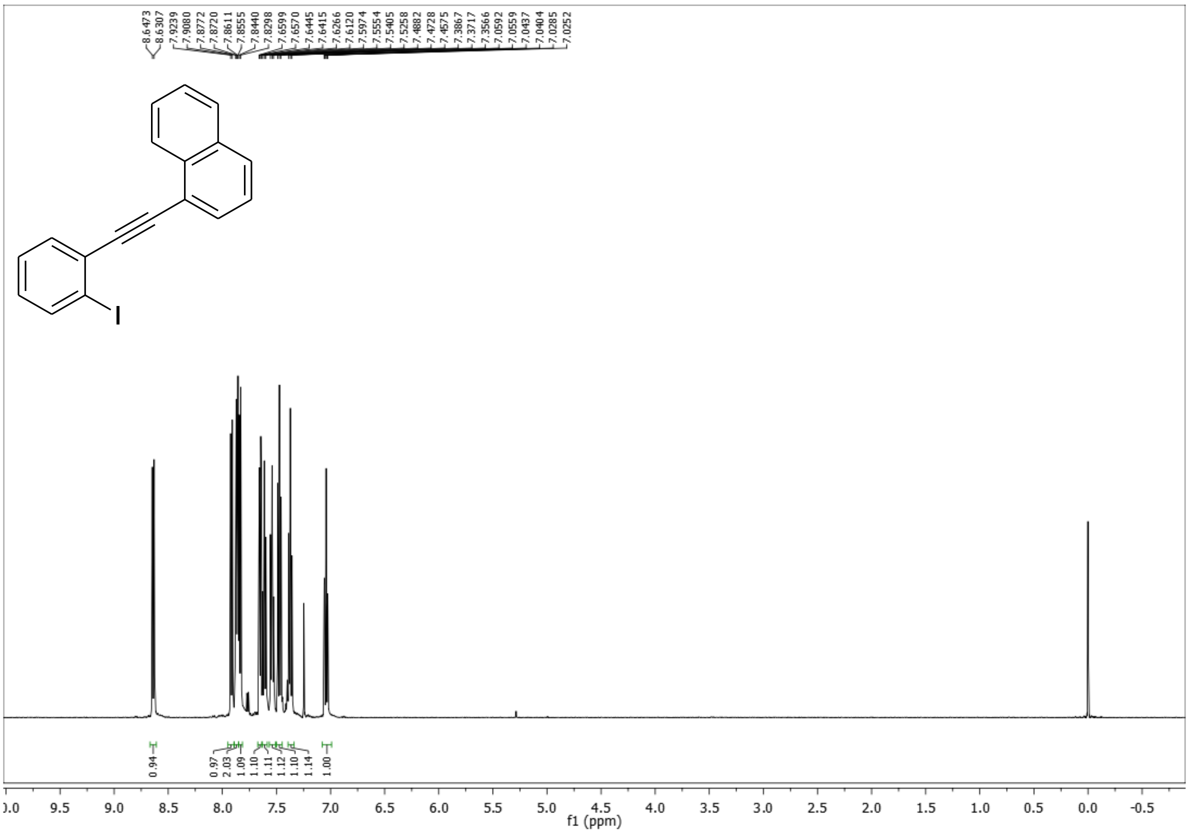

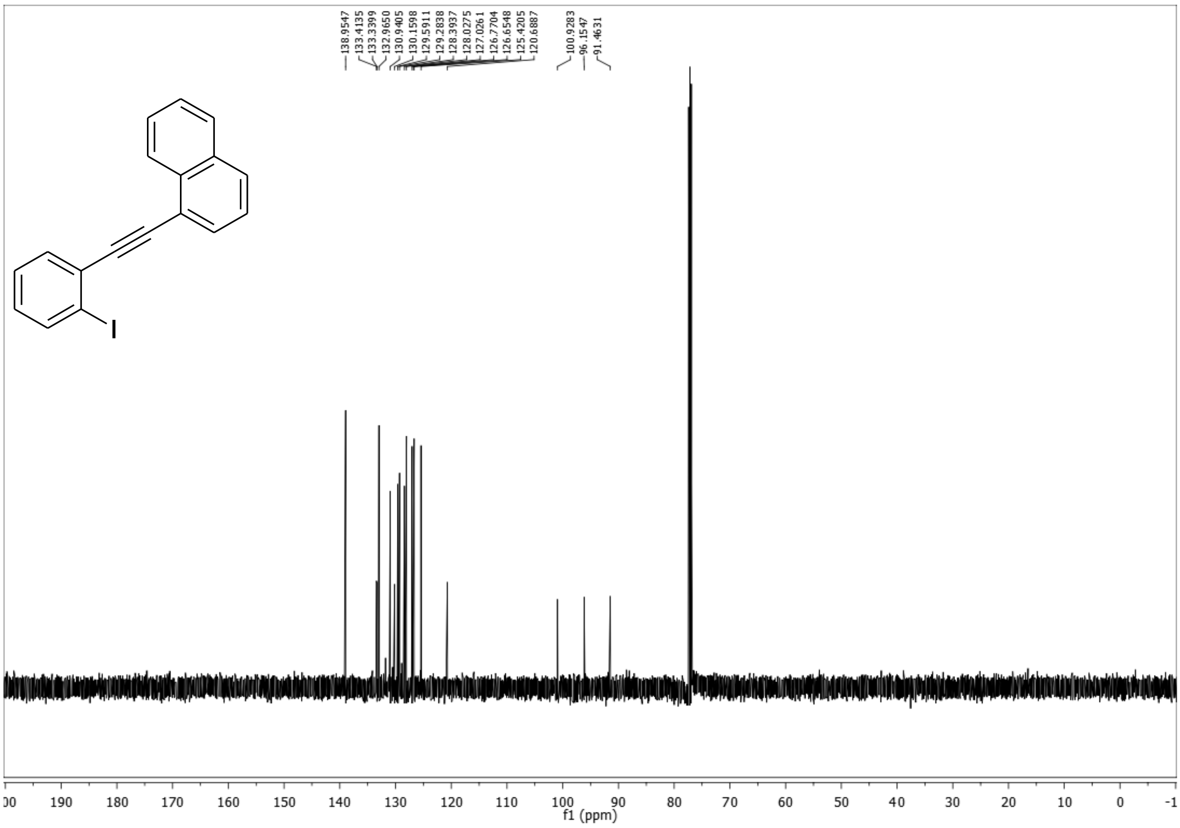

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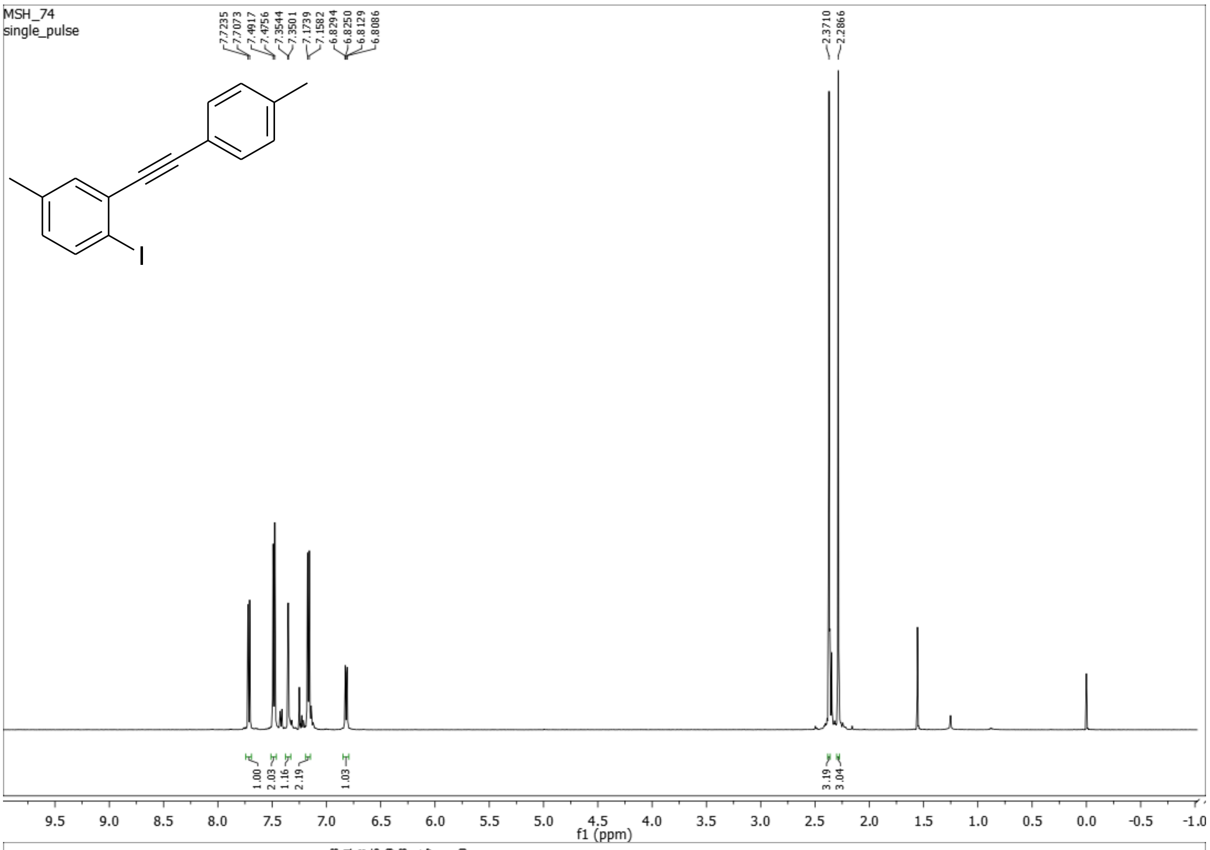

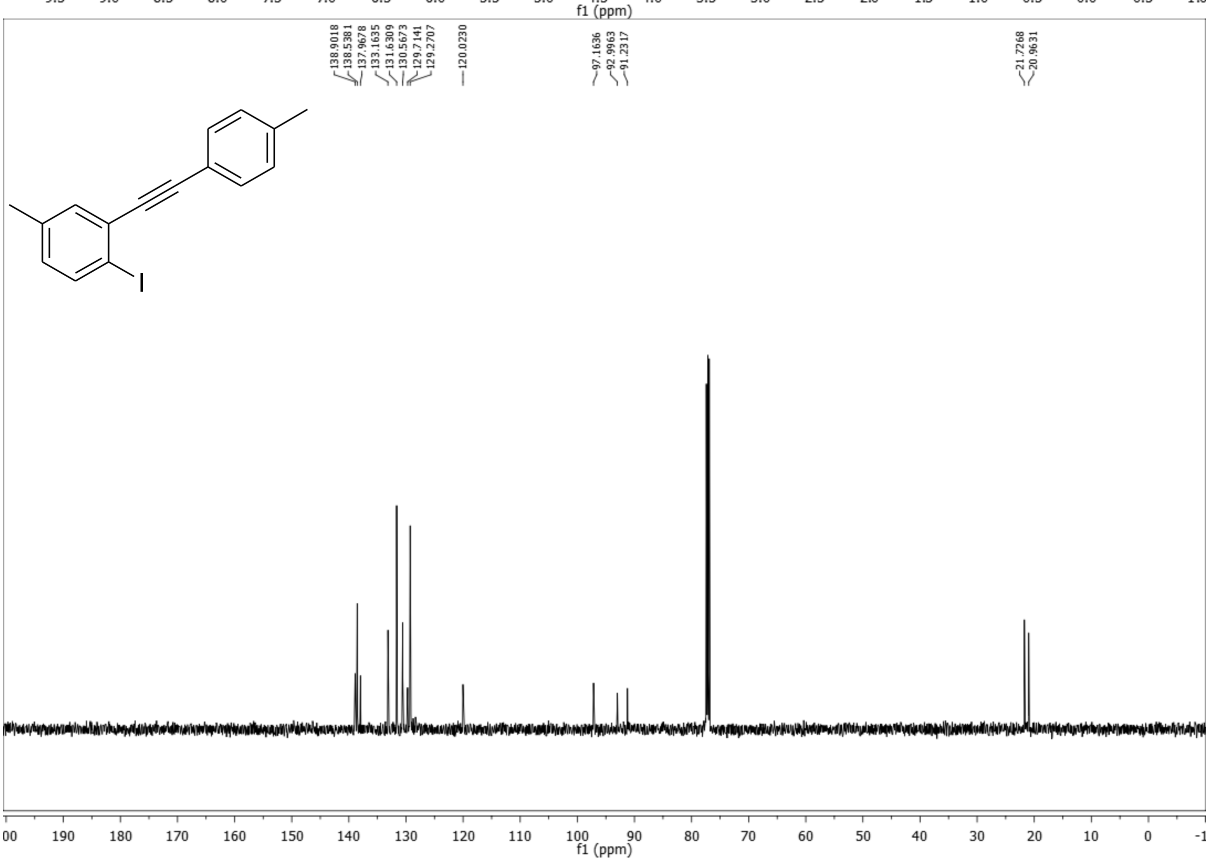

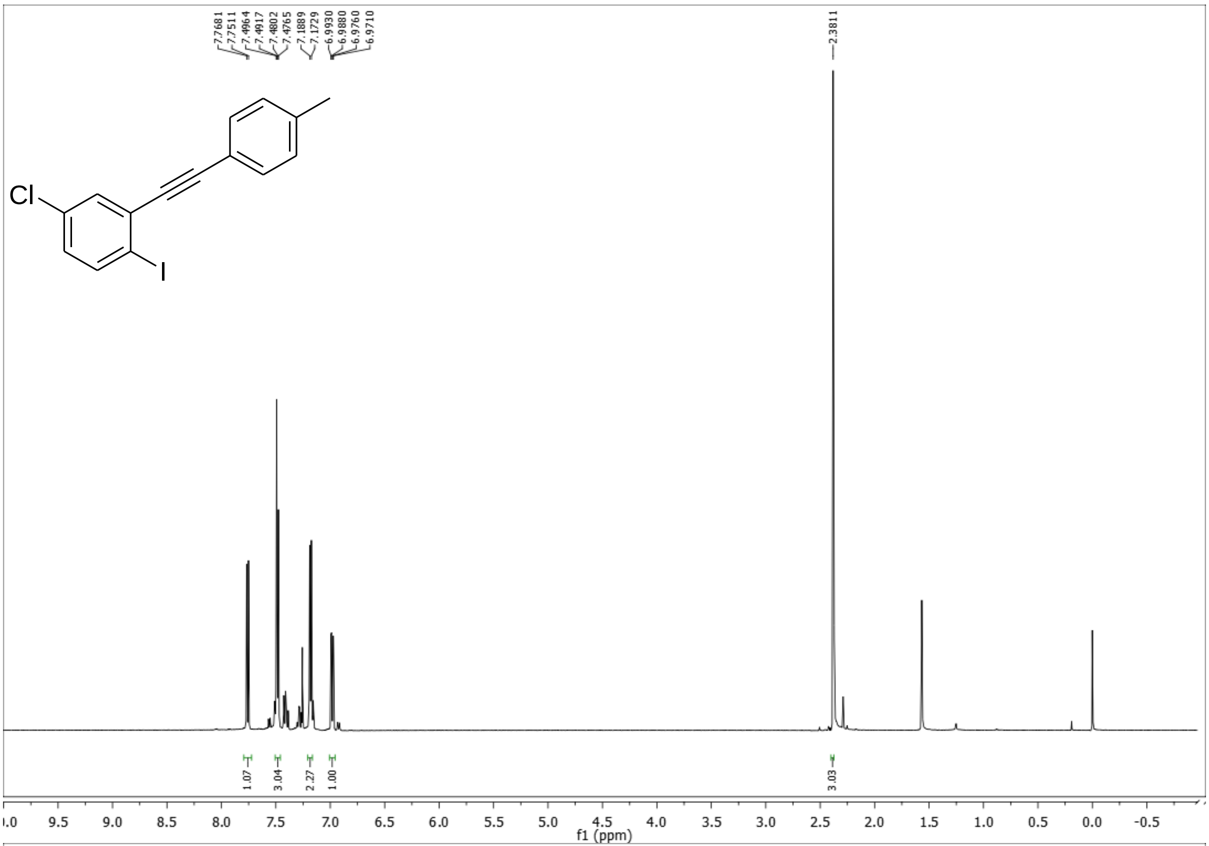
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6.9710
2.3811
Cl
I
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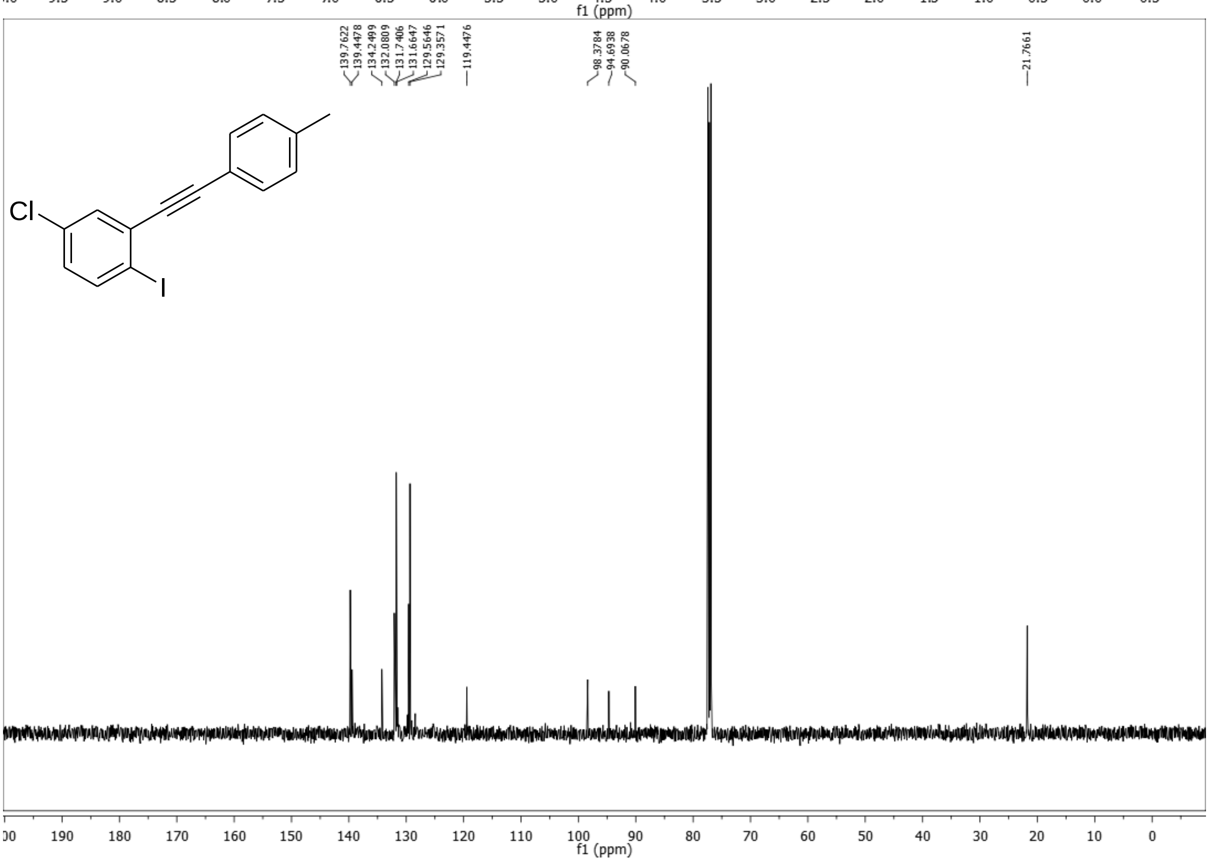
3.04
2.27
1.00
3.03
f1 (ppm)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90.0678
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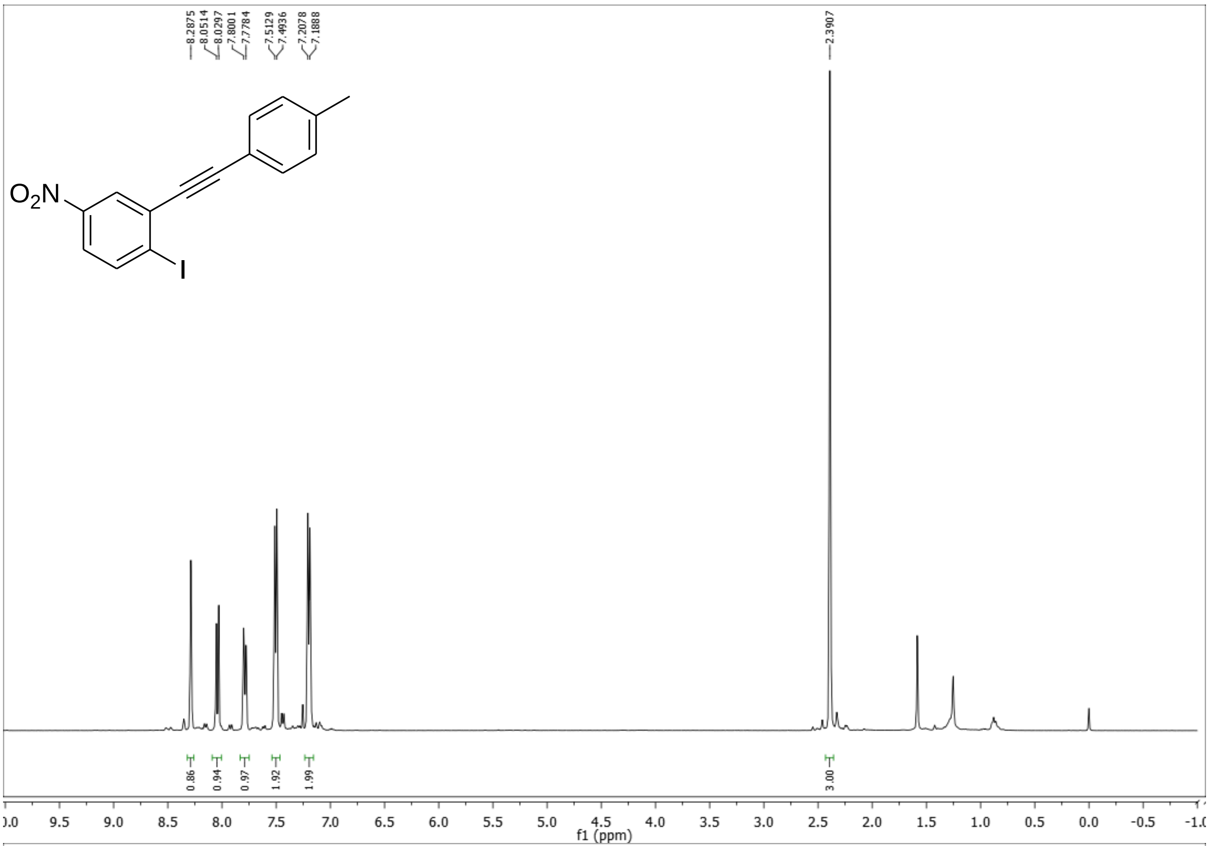
Cl
I
f1 (ppm)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7.4936
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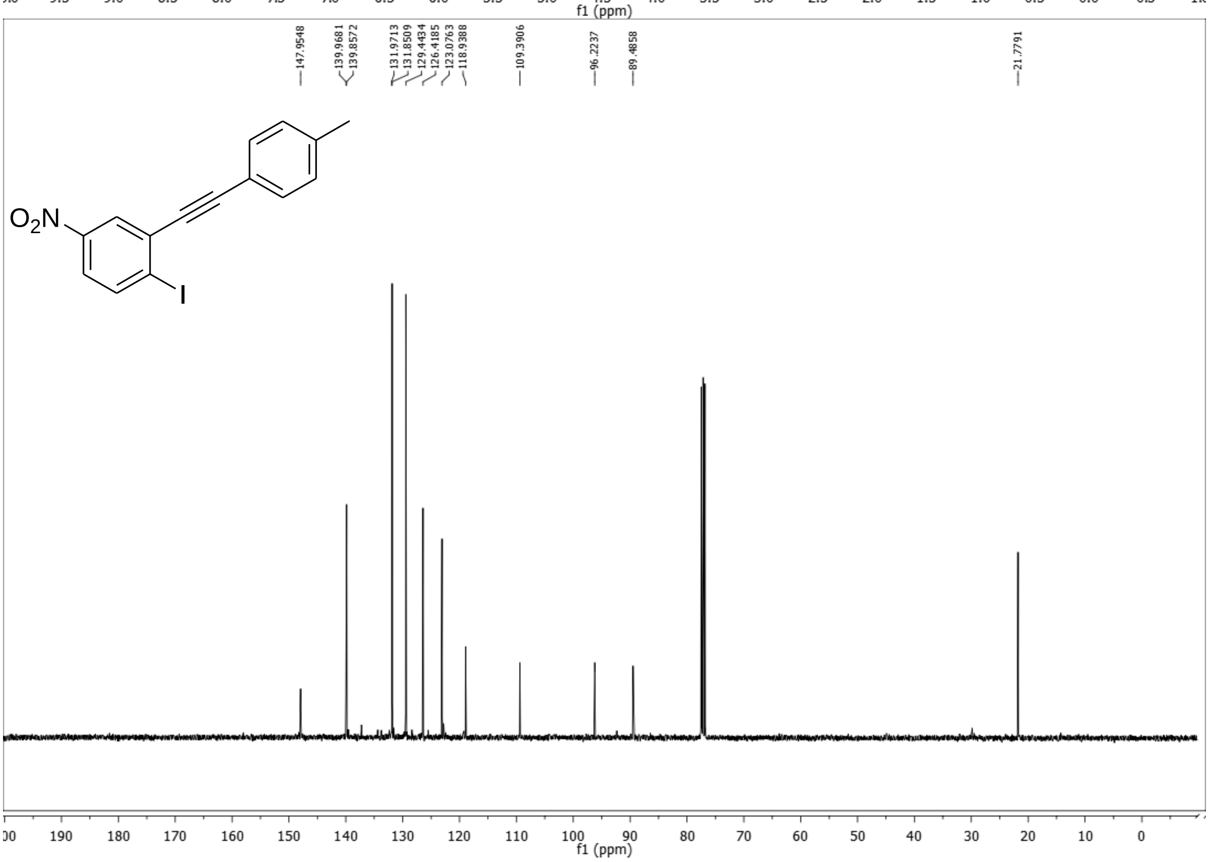
O2N
I
0.86
0.94
0.97
1.92
1.99
3.00
f1 (ppm)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89.4858
21.7791
O2N
I
f1 (ppm)

CIF file of compound 4p (CCDC 948676)

Table 1. Crystal data and structure refinement for new.

Identification code	new
Empirical formula	C H N O S
Formula weight	75.09
Temperature	273(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pbca
Unit cell dimensions	a = 13.4815(4) A alpha = 90 deg. b = 8.2127(3) A beta = 90 deg. c = 28.9529(9) A gamma = 90 deg.
Volume	3205.65(18) A^3
Z, Calculated density	42, 1.634 Mg/m^3
Absorption coefficient	0.781 mm^-1
F(000)	1596
Crystal size	0.35 x 0.25 x 0.20 mm
Theta range for data collection	1.41 to 24.99 deg.
Limiting indices	-16<=h<=15, -7<=k<=7, -34<=l<=33
Reflections collected / unique	10575 / 2401 [R(int) = 0.0222]
Completeness to theta = 24.99	85.3 %
Absorption correction Multi-scan	
Max. and min. transmission	0.8594 and 0.7716
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2401 / 0 / 237
Goodness-of-fit on F^2	1.089
Final R indices [I>2sigma(I)]	R1 = 0.0357, wR2 = 0.0818
R indices (all data)	R1 = 0.0456, wR2 = 0.0894
Largest diff. peak and hole	0.135 and -0.199 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for new.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	6890(1)	5791(2)	1234(1)	54(1)
C(2)	6824(1)	6259(3)	786(1)	59(1)
C(3)	6253(1)	5345(2)	477(1)	51(1)
C(4)	5737(1)	4018(2)	625(1)	43(1)
C(5)	5780(1)	3492(2)	1091(1)	37(1)
C(6)	5300(1)	2064(2)	1263(1)	38(1)
C(7)	4619(1)	1062(2)	980(1)	39(1)
C(8)	3842(1)	1747(3)	684(1)	43(1)
C(9)	3276(1)	599(3)	379(1)	59(1)
C(10)	3785(2)	-1010(3)	299(1)	63(1)
C(11)	4078(1)	-1767(3)	756(1)	59(1)
C(12)	4610(1)	-562(3)	1049(1)	47(1)
C(13)	5161(1)	-208(2)	1810(1)	54(1)
C(14)	5540(1)	1460(2)	1696(1)	40(1)
C(15)	6154(1)	2362(2)	2005(1)	40(1)
C(16)	6538(1)	3812(2)	1853(1)	45(1)
C(17)	6401(1)	4388(2)	1398(1)	41(1)
C(18)	6390(1)	1778(2)	2475(1)	46(1)
S(1)	7529(2)	2172(3)	2708(1)	60(1)
C(19)	5818(10)	988(19)	2779(5)	68(4)
S(1A)	5603(7)	721(12)	2826(3)	61(1)
C(19A)	7272(14)	2240(30)	2696(6)	94(10)
C(20)	6334(2)	675(3)	3231(1)	75(1)
C(21)	7240(2)	1313(3)	3198(1)	75(1)
O(1)	5101(1)	-1240(2)	1406(1)	59(1)
O(2)	3610(1)	3189(2)	700(1)	56(1)

Table 3. Bond lengths [A] and angles [deg] for new.

C(1)-C(2)	1.355(3)
C(1)-C(17)	1.410(3)
C(2)-C(3)	1.398(3)
C(3)-C(4)	1.363(3)
C(4)-C(5)	1.417(2)
C(5)-C(17)	1.424(2)
C(5)-C(6)	1.429(2)
C(6)-C(14)	1.386(2)
C(6)-C(7)	1.481(2)
C(7)-C(12)	1.349(3)
C(7)-C(8)	1.464(2)
C(8)-O(2)	1.226(2)
C(8)-C(9)	1.501(3)
C(9)-C(10)	1.507(3)
C(10)-C(11)	1.514(3)
C(11)-C(12)	1.489(3)
C(12)-O(1)	1.347(2)
C(13)-O(1)	1.446(2)
C(13)-C(14)	1.498(3)
C(14)-C(15)	1.427(2)
C(15)-C(16)	1.371(2)
C(15)-C(18)	1.477(2)
C(16)-C(17)	1.413(2)
C(18)-C(19)	1.339(10)
C(18)-C(19A)	1.403(18)
C(18)-S(1A)	1.707(8)
C(18)-S(1)	1.708(2)
S(1)-C(21)	1.632(4)
C(19)-C(20)	1.502(16)
S(1A)-C(20)	1.533(10)
C(19A)-C(21)	1.64(2)
C(20)-C(21)	1.331(3)
C(2)-C(1)-C(17)	121.52(17)
C(1)-C(2)-C(3)	119.73(18)
C(4)-C(3)-C(2)	120.60(17)
C(3)-C(4)-C(5)	121.49(16)
C(4)-C(5)-C(17)	117.38(15)
C(4)-C(5)-C(6)	124.30(15)
C(17)-C(5)-C(6)	118.17(15)
C(14)-C(6)-C(5)	120.23(14)
C(14)-C(6)-C(7)	116.51(15)
C(5)-C(6)-C(7)	122.92(14)
C(12)-C(7)-C(8)	117.42(16)
C(12)-C(7)-C(6)	118.19(15)
C(8)-C(7)-C(6)	123.60(17)
O(2)-C(8)-C(7)	122.09(16)
O(2)-C(8)-C(9)	119.97(16)
C(7)-C(8)-C(9)	117.80(18)
C(8)-C(9)-C(10)	114.18(16)
C(9)-C(10)-C(11)	110.14(15)
C(12)-C(11)-C(10)	110.53(17)
O(1)-C(12)-C(7)	121.25(16)
O(1)-C(12)-C(11)	113.52(18)
C(7)-C(12)-C(11)	125.23(17)
O(1)-C(13)-C(14)	112.17(14)
C(6)-C(14)-C(15)	121.08(16)
C(6)-C(14)-C(13)	116.51(14)
C(15)-C(14)-C(13)	122.35(15)
C(16)-C(15)-C(14)	118.03(15)
C(16)-C(15)-C(18)	119.72(15)

C(14)-C(15)-C(18)	122.25(17)
C(15)-C(16)-C(17)	122.66(15)
C(1)-C(17)-C(16)	121.72(15)
C(1)-C(17)-C(5)	119.15(15)
C(16)-C(17)-C(5)	119.09(16)
C(19)-C(18)-C(19A)	108.5(11)
C(19)-C(18)-C(15)	129.8(7)
C(19A)-C(18)-C(15)	121.0(9)
C(19)-C(18)-S(1A)	4.7(10)
C(19A)-C(18)-S(1A)	113.1(9)
C(15)-C(18)-S(1A)	125.4(3)
C(19)-C(18)-S(1)	110.4(7)
C(19A)-C(18)-S(1)	6.5(9)
C(15)-C(18)-S(1)	119.71(17)
S(1A)-C(18)-S(1)	114.8(3)
C(21)-S(1)-C(18)	92.69(18)
C(18)-C(19)-C(20)	112.9(10)
C(20)-S(1A)-C(18)	93.9(5)
C(18)-C(19A)-C(21)	104.9(13)
C(21)-C(20)-C(19)	107.2(4)
C(21)-C(20)-S(1A)	121.7(3)
C(19)-C(20)-S(1A)	14.7(6)
C(20)-C(21)-S(1)	116.80(19)
C(20)-C(21)-C(19A)	105.7(7)
S(1)-C(21)-C(19A)	12.3(7)
C(12)-O(1)-C(13)	113.84(14)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for new.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	51(1)	44(1)	67(1)	-5(1)	-6(1)	-12(1)
C(2)	55(1)	50(1)	72(1)	10(1)	2(1)	-15(1)
C(3)	47(1)	52(1)	54(1)	5(1)	5(1)	-2(1)
C(4)	37(1)	43(1)	49(1)	-7(1)	2(1)	0(1)
C(5)	31(1)	33(1)	48(1)	-7(1)	3(1)	3(1)
C(6)	31(1)	33(1)	50(1)	-7(1)	1(1)	2(1)
C(7)	35(1)	35(1)	49(1)	-7(1)	3(1)	-2(1)
C(8)	36(1)	50(2)	43(1)	-3(1)	4(1)	-6(1)
C(9)	59(1)	64(2)	54(1)	-2(1)	-10(1)	-18(1)
C(10)	65(1)	68(2)	56(1)	-20(1)	10(1)	-28(1)
C(11)	55(1)	45(1)	79(1)	-17(1)	2(1)	-9(1)
C(12)	37(1)	42(2)	61(1)	-9(1)	0(1)	-1(1)
C(13)	55(1)	44(1)	62(1)	2(1)	-11(1)	-7(1)
C(14)	33(1)	33(1)	53(1)	-3(1)	1(1)	0(1)
C(15)	35(1)	39(1)	47(1)	-6(1)	2(1)	2(1)
C(16)	41(1)	44(1)	51(1)	-13(1)	-4(1)	-3(1)
C(17)	35(1)	35(1)	54(1)	-6(1)	1(1)	0(1)
C(18)	45(1)	46(1)	48(1)	-7(1)	0(1)	3(1)
S(1)	55(1)	76(1)	50(1)	-16(1)	-13(1)	4(1)
C(19)	67(7)	75(8)	61(5)	2(4)	4(4)	10(5)
S(1A)	69(3)	61(2)	54(1)	5(1)	14(1)	-9(2)
C(19A)	99(18)	118(14)	65(9)	32(8)	26(9)	13(9)
C(20)	98(2)	70(2)	57(1)	2(1)	11(1)	7(1)
C(21)	85(2)	89(2)	51(1)	-13(1)	-15(1)	17(2)
O(1)	61(1)	35(1)	81(1)	-5(1)	-14(1)	0(1)
O(2)	46(1)	56(1)	65(1)	-3(1)	-9(1)	7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for new.

	x	y	z	U(eq)
H(1)	7266	6410	1438	65
H(2)	7156	7184	684	71
H(3)	6227	5645	167	61
H(4)	5346	3445	416	51
H(9A)	3166	1120	83	71
H(9B)	2632	396	517	71
H(10A)	4372	-846	111	76
H(10B)	3343	-1739	134	76
H(11A)	3488	-2143	916	71
H(11B)	4502	-2701	701	71
H(13A)	5597	-708	2035	64
H(13B)	4508	-115	1948	64
H(16)	6904	4443	2058	54
H(19)	5170	667	2718	81
H(19A)	7767	2919	2583	113
H(20)	6071	137	3485	90
H(21)	7686	1271	3442	90