Supporting Information

Two Catalytic Protocols for Achmatowicz Rearrangement by Using Cyclic Diacyl Peroxides as Oxidants

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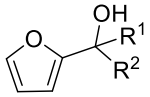
1 General Methods and Materials

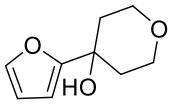
All reactions were carried out directly under open air. Commercially available reagents, starting materials and solvents were used without further purification. The reaction solvent, CH_2Cl_2 and CH_3OH was used without purification. All reactions were monitored by TLC and visualized by UV lamp (254 nm)/or by staining with a solution of 10 g phosphomolybdic acid and 100 mL EtOH followed by heating. Flash column chromatography was performed using 200-300 mesh silica gel. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz or 150 MHz) spectra were obtained on Bruker 400M or 600M nuclear resonance spectrometers. HR-ESI-MS spectra were recorded on a Bruker Esquire LC mass spectrometer using electrospray ionization. Coupling constants are reported in Hertz (Hz). Data for ^{1}H-NMR spectra were reported as follows: chemical shift *(*ppm, referenced to protium; s = singlet, br s= broad singlet, d = doublet, t = triplet, dd = doublet of doublets, td = triplet of doublets, m = multiplet, coupling constant (Hz), and integration). Data for ^{1}H-NMR were reported in (ppm) relative to residual solvent peak ($CDCl_3$: 7.26 ppm). Data for ^{13}C-NMR were reported in (ppm) relative to residual solvent peak ($CDCl_3$: 77.16 ppm).

2 Synthesis of the Furfuryl Alcohol Substrates

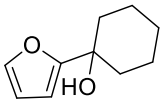
Furfuryl alcohols **1a**, **1b**, **1c**, **1d**, **1f**, **1g**, **1j**, **1l**, **1k**, **1m**, **1o**, **1q**, **1r**, **1e**, **1h**, **1p** and **1t** was prepared according to a literature procedure[1]. Alcohols **1i**[2], **1n**[3] and **1s**[4] were synthesized according to known literature methods.



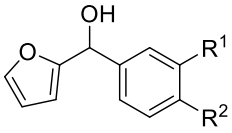
1a (R^1=n-Pr, R^2=H,)
1b (R^1=n-Pr, R^2=n-Pr)
1c (R^1=H, R^2=H)
1f (R^1=Cy, R^2=H)
1g (R^1=Me, R^2=Me)
1j (R^1=Me, R^2=n-Bu)
1l (R^1=Ph, R^2=Ph)



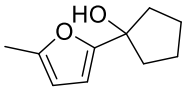
1d



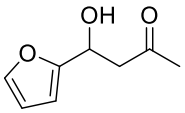
1e



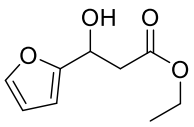
1k (R^1=H, R^2=H)
1m (R^1=H, R^2=Cl)
1o (R^1=H, R^2=Br)
1q (R^1=H, R^2=OMe)
1r (R^1=OMe, R^2=H)



1h



1i



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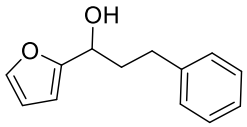
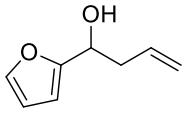
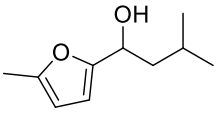


1p



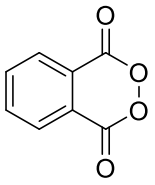
1s



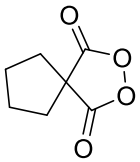
1t

3 Synthesis of the Cyclic Diacyl Peroxides

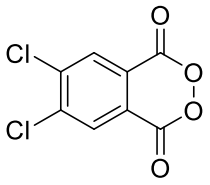
Phthaloyl peroxide (PPO)[5], 4,5-dichlorophthaloyl deroxide (Cl-PPO)[6] and cyclopentyl malonoyl peroxide (MPO) [5] were synthesized according to known literature methods.



PPO



MPO



Cl-PPO

4 Experimental procedures for Table 1 and Table 2

Experimental procedures for Table 1: To a stirred solution of the 2-butyl-6-hydroxy-2H-pyran-3(6H)-one (0.5 mmol, 1.0 equiv) in a 5 mL Corresponding solvent were added catalyst (0.015 mmol) and oxidant (0.6 mmol, 1.2 equiv) at Corresponding temperature. Upon completion (12 h).The reaction was then quenched by

addition of saturated aqueous $NaHCO_3$ (10 mL) and extract with EtOAc (3 × 10 mL). The combined organic fractions were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the pure. product.

Experimental procedures for Table 2: To a Schlenk tube was added with crude 2-butyl-6-hydroxy-2H-pyran-3(6H)-one (0.5 mmol, 1.0 equiv) and photoredox catalysis (2.5 μmol, 0.5 mol%), followed by the oxidant (0.5mmol, 1.0 equiv)in a 5 mL Corresponding solvent. The reaction mixture was kept in the dark while N_2 was bubbled through it for 15 minutes. The resulting solution was then irradiated by blue LEDs lamps and magnetically stirred until the starting material was completely consumed (about 5 h) The reaction was then quenched by addition of saturated aqueous $NaHCO_3$ (10 mL) and extract with EtOAc (3 × 10 mL). The combined organic fractions were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the pure product.

5 General experimental procedures

General Procedure for the Synthesis of 2a-2q using TBAB and PPO: To a stirred solution of the furfuryl alcohol (0.5 mmol) in a 4/1 mixture of THF and H_2O (5 mL) were added TBAB (8 mg, 0.015 mmol) and PPO (98 mg, 0.6 mmol) at 25 ℃. Upon completion (12 h).The reaction was then quenched by addition of saturated aqueous $NaHCO_3$ (10 mL) and extract with EtOAc (3 × 10 mL). The combined organic fractions were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the pure. product.

General Procedure for the Synthesis of 2a-2q using Cl-PPO and $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$: To a Schlenk tube was added with crude furfuryl alcohol substrate (0.5 mmol, 1 equiv) and $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ (2.8 mg, 2.5 μmol, 0.5 mol%), followed by the Cl-PPO(115 mg, 0.5 mmol, 1.0 equiv)in a 3/1 mixture of MeCN and H_2O (5 mL). The reaction mixture was kept in the dark while N_2 was bubbled through it for 15 minutes. The resulting solution was then irradiated by blue LEDs lamps and magnetically stirred until the starting material was completely consumed (about 5 h) The reaction was then quenched by addition of saturated aqueous $NaHCO_3$ (10 mL) and extract with EtOAc (3 × 10 mL). The combined organic fractions were dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the pure product.

6 General experimental procedures for gram-scale synthesis

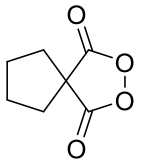
Gram-scale synthesis of 2c using TBAB and PPO: To a stirred solution of the furfuryl alcohol (2.5 mmol) in a 4/1 mixture of THF and H_2O (25 mL) were added TBAB (40 mg,

0.075 mmol) and PPO (490 mg, 3.0 mmol) at 25 ℃. Upon completion (12 h).The reaction was then quenched by addition of saturated aqueous $NaHCO_3$ (50 mL) and extract with EtOAc (3 × 50 mL). The combined organic fractions were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the pure. product.

Gram-scale synthesis of 2c using Cl-PPO and [Ir(dF(CF_3)ppy)$_2$(dtbbpy)](PF_6): To a Schlenk tube was added with crude furfuryl alcohol substrate (5 mmol, 1 equiv) and [Ir(dF(CF_3)ppy)$_2$(dtbbpy)](PF_6) (14 mg, 12.5 μmol, 0.5 mol%), followed by the Cl-PPO(575 mg, 2.5 mmol, 1.0 equiv)in a 3/1 mixture of MeCN and H_2O (25 mL). The reaction mixture was kept in the dark while N_2 was bubbled through it for 15 minutes. The resulting solution was then irradiated by blue LEDs lamps and magnetically stirred until the starting material was completely consumed (about 5 h) The reaction was then quenched by addition of saturated aqueous $NaHCO_3$ (50 mL) and extract with EtOAc (3 × 50 mL). The combined organic fractions were dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the pure product.

7 Characterization of Products in Details:

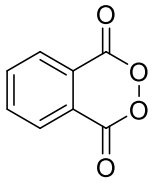
2,3-dioxaspiro[4.4]nonane-1,4-dione[5]



MPO

White solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.38 – 2.20 (m, 4H), 2.10 – 1.93 (m, 4H).**^{13}C NMR** (151 MHz, $CDCl_3$) δ 175.8, 46.9, 37.7, 26.8.

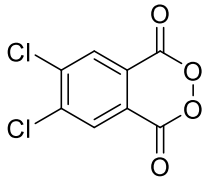
benzo[d][1,2]dioxine-1,4-dione[5]



PPO

White solid **^{1}H NMR** (400 MHz, Chloroform-d) δ 8.29 (dd, J = 5.8, 3.3 Hz, 2H), 8.03 (dd, J = 5.8, 3.3 Hz, 2H).**^{13}C NMR** (151 MHz, Chloroform-d) δ 162.11 , 136.65 , 130.30 , 123.73 .

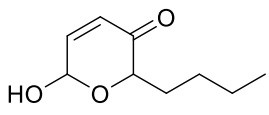
6,7-dichlorobenzo[d][1,2]dioxine-1,4-dione[6]



Cl-PPO

White solid **^{1}H NMR** (600 MHz, Chloroform-d) δ 8.34 (s, 2H). **^{13}C NMR** (151 MHz, Chloroform-d) δ 160.59 , 142.63 , 132.03 , 122.76 .

2-butyl-6-hydroxy-2H-pyran-3(6H)-one (2a)[7]

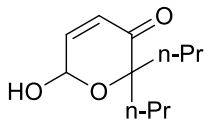


2a

White solid.R_f = 0.28(Petroleum ether:Ethyl acetate = 4:1) **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.91 (dd, *J* = 10.2, 3.4 Hz, 4H), 6.12 (d, *J* = 10.2 Hz, 4H), 5.66 (s, 4H), 4.57

(dd, *J* = 8.0, 3.9 Hz, 3H), 4.09 (ddd, *J* = 8.4, 4.0, 1.3 Hz, 1H), 3.28 (d, *J* = 4.8 Hz, 3H), 2.05 – 1.86 (m, 4H), 1.86 – 1.61 (m, 4H), 1.60 – 1.18 (m, 16H), 0.94 (s, 12H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.72, 196.35, 147.67, 144.33, 128.77, 127.66, 90.89, 87.65, 78.97, 77.34, 77.02, 76.70, 74.25, 30.35, 29.34, 27.27, 27.14, 22.49, 22.45, 13.93, 13.91.

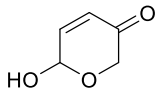
6-hydroxy-2,2-dipropyl-2H-pyran-3(6H)-one (2b)



2b

Light yellow oil. R_f = 0.22(Petroleum ether:Ethyl acetate = 10:1). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.84 (dd, *J* = 10.3, 2.2 Hz, 1H), 6.06 (dd, *J* = 10.3, 1.3 Hz, 1H), 5.73 (d, *J* = 5.6 Hz, 1H), 3.78 (d, *J* = 6.2 Hz, 1H), 1.97 – 1.13 (m, 8H), 0.88 (d, *J* = 5.0 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 199.25, 145.59, 127.19, 87.64, 84.71, 39.58, 37.62, 16.77, 16.55, 14.36. **HRMS (ESI)**: $[M+H]^+$ calcd for $C_{11}H_{19}O_3^+$: 199.1329, found: 199.1333.

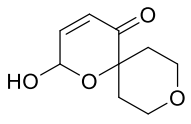
6-hydroxy-2H-pyran-3(6H)-one(2c)[2]



2c

Light yellow oil. R_f = 0.15 (Petroleum ether:Ethyl acetate = 2:1). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.98 (dd, *J* = 10.4, 3.0 Hz, 1H), 6.19 (dd, *J* = 10.4, 0.9 Hz, 1H), 5.71 – 5.59 (m, 1H), 4.59 (d, *J* = 16.9 Hz, 1H), 4.16 (d, *J* = 16.9 Hz, 1H), 3.40 (d, *J* = 5.3 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 194.57, 145.76, 127.93, 88.24, 66.62.

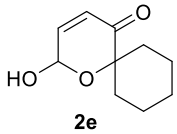
2-hydroxy-1,9-dioxaspiro[5.5]undec-3-en-5-one (2d)



2d

Light yellow oil. R_f = 0.42(Petroleum ether:Ethyl acetate = 1:1). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.89 (dd, *J* = 10.3, 2.4 Hz, 1H), 6.10 (d, *J* = 10.3 Hz, 1H), 5.73 (dd, *J* = 6.1, 2.4 Hz, 1H), 4.12 (d, *J* = 6.4 Hz, 1H), 3.86 (d, *J* = 2.7 Hz, 4H), 2.14 – 1.92 (m, 3H), 1.76 – 1.57 (m, 1H). **^{13}C NMR** (101 MHz,Chloroform-*d*) δ 197.57, 145.31, 126.34, 87.59, 77.48, 62.85, 62.81, 34.22, 31.57. **HRMS (ESI)**: $[M+H]^+$ calcd for $C_{11}H_{19}O_3^+$: 199.1329, found: 199.1333.

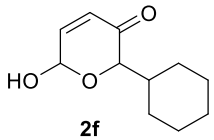
2-hydroxy-1-oxaspiro[5.5]undec-3-en-5-one(2e)[8]



2e

Light yellow oil. Rf = 0.36(Petroleum ether:Ethyl acetate = 1:1) **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 6.85 (dd, *J* = 10.8, 4.4 Hz, 1H), 6.06 (dd, *J* = 11.3, 4.6 Hz, 1H), 5.71 (d, *J* = 6.1 Hz, 1H), 3.37 (t, *J* = 5.7 Hz, 1H), 2.18 – 1.44 (m, 9H), 1.44 – 1.13 (m, 1H).**^{13}C NMR** (151 MHz, Chloroform-*d*) δ 199.25, 145.32, 126.86, 87.57, 80.68, 33.45, 31.03, 25.15, 21.00, 20.57.

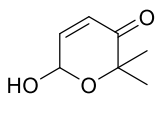
2-cyclohexyl-6-hydroxy-2H-pyran-3(6H)-one(2f)[8]



2f

Light yellow oil. R_f = 0.22(Petroleum ether:Ethyl acetate = 10:1). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.01 – 6.74 (m, 1H), 6.12 (ddd, *J* = 22.1, 10.4, 3.0 Hz, 1H), 5.65 (dd, *J* = 22.6, 4.5 Hz, 1H), 4.38 (d, *J* = 3.4 Hz, 1H), 3.40 (s, 1H), 2.11 (dh, *J* = 11.7, 3.7 Hz, 1H), 1.94 – 0.94 (m, 11H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 196.83, 196.35, 148.10, 144.44, 129.44, 128.12, 91.17, 87.59, 83.02, 78.45, 38.57, 38.43, 29.42, 29.32, 26.63, 26.53, 26.46, 26.38, 26.15, 26.14, 26.10.

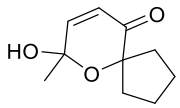
6-hydroxy-2,2-dimethyl-2H-pyran-3(6H)-one(2g)[8]



2g

Light yellow oil. R_f = 0.15(Petroleum ether:Ethyl acetate = 2:1). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 6.90 (d, *J* = 10.3 Hz, 1H), 6.09 (d, *J* = 9.9 Hz, 1H), 5.71 (s, 1H), 3.75 (s, 1H), 1.50 (s, 3H), 1.40 (s, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 198.95, 145.83, 126.45, 87.86, 79.47, 26.51, 23.75.

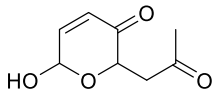
7-hydroxy-7-methyl-6-oxaspiro[4.5]dec-8-en-10-one(2h)[9]



2h

Light yellow oil. R_f = 0.15(Petroleum ether:Ethyl acetate = 10:1). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.82 (d, *J* = 10.2 Hz, 1H), 6.07 (d, *J* = 10.2 Hz, 1H), 2.79 (s, 1H), 2.46 – 2.13 (m, 2H), 1.75 (d, *J* = 15.3 Hz, 7H), 1.61 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 199.33, 147.58, 125.62, 92.84, 88.76, 40.56, 37.82, 30.22, 25.12, 24.40.

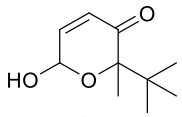
6-hydroxy-2-(2-oxopropyl)-2H-pyran-3(6H)-one(2i)[10]



2i

Light yellow oil. R_f = 0.20(Petroleum ether:Ethyl acetate = 2:1). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.07 – 6.76 (m, 1H), 6.17 (dd, *J* = 18.6, 10.3 Hz, 1H), 5.61 (t, *J* = 2.9 Hz, 1H), 5.07 (dd, *J* = 7.5, 3.8 Hz, 1H), 3.12 (t, *J* = 3.4 Hz, 1H), 2.89 (ddd, *J* = 32.3, 17.6, 7.5 Hz, 1H), 2.23 (d, *J* = 5.1 Hz, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 206.21, 206.11, 195.84, 195.28, 148.80, 144.87, 128.40, 126.90, 124.46, 98.73, 91.04, 87.65, 74.52, 70.05, 44.02, 43.71, 30.56, 30.43.

2-(tert-butyl)-6-hydroxy-2-methyl-2H-pyran-3(6H)-one (2j)

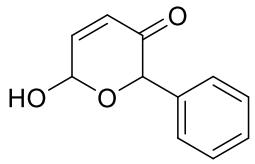


2j

Light yellow oil. R_f = 0.20(Petroleum ether:Ethyl acetate = 10:1) **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 6.84 (d, *J* = 17.7 Hz, 1H), 6.07 (dd, *J* = 47.8, 10.2 Hz, 1H), 5.72 (d, *J* = 40.6 Hz, 1H), 3.34 (s, 1H), 1.50 (s, 2H), 1.05 (s, 4H), 0.99 (s, 5H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 200.11, 200.09, 146.57, 143.30, 129.19, 128.40, 87.83, 87.62, 85.80, 85.13,

38.97, 38.35, 25.59, 25.49, 22.59, 16.42. **HRMS (ESI)**: $[M+H]^+$ calcd for $C_{10}H_{17}O_3^+$: 185.1172, found: 185.1177.

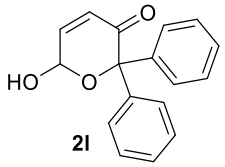
6-hydroxy-2-phenyl-2H-pyran-3(6H)-one (2k)[10]



2k

Light yellow oil. R_f = 0.22(Petroleum ether:Ethyl acetate = 2:1). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.52 – 7.24 (m, 5H), 7.04 – 6.78 (m, 1H), 6.21 (ddd, *J* = 16.1, 10.3, 1.2 Hz, 1H), 5.66 (d, *J* = 14.7 Hz, 1H), 5.57 (s, 1H), 5.05 (d, *J* = 1.4 Hz,), 4.02 (s, 1H).**^{13}C NMR** (101 MHz, Chloroform-*d*) δ 194.93, 148.69, 145.23, 135.25, 129.21, 128.73, 128.66, 128.50, 128.47, 128.00, 127.94, 127.60, 91.43, 87.93, 81.09, 76.97.

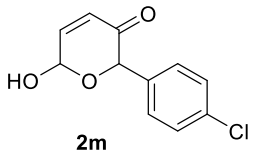
6-hydroxy-2,2-diphenyl-2H-pyran-3(6H)-one(2l)[8]



2l

Light yellow oil. R_f = 0.22(Petroleum ether:Ethyl acetate = 4:1). **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.40 – 7.24 (m, 10H), 6.87 (dd, *J* = 10.2, 2.6 Hz, 1H), 6.29 (d, *J* = 10.3 Hz, 1H), 5.48 (s, 1H), 3.33 (s, 1H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 147.48, 141.27, 137.33, 129.02, 128.67, 128.62, 127.92, 127.79, 127.62, 127.53, 88.87, 86.97.

2-(4-chlorophenyl)-6-hydroxy-2H-pyran-3 (6H)-one (2m)

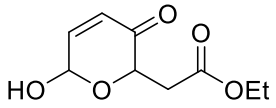


2m

Light yellow oil. R_f = 0.15(Petroleum ether:Ethyl acetate = 2:1). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.42 – 7.24 (m, 4H), 6.98 (ddd, *J* = 13.7, 10.3, 2.4 Hz, 1H), 6.23 (ddd, *J* =

22.6, 10.3, 1.2 Hz, 1H), 5.82 – 5.66 (m, 1H), 5.57 (s, 1H), 5.07 (d, *J* = 1.4 Hz, 0H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 194.23, 148.52, 145.00, 134.48, 133.69, 129.24, 129.13, 128.61, 128.59, 127.53, 91.46, 88.01, 80.21, 76.04. **HRMS (ESI)**: $[M+H]^+$ calcd for $C_{11}H_{10}ClO_3^+$: 225.0313, found: 225.0317.

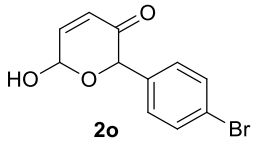
ethyl 2-(6-hydroxy-3-oxo-3,6-dihydro-2H-pyran-2-yl)acetate (2n)[10]



2n

Light yellow oil. R_f = 0.22(Petroleum ether:Ethyl acetate = 2:1). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.06 – 6.77 (m, 1H), 6.11 (dd, *J* = 18.0, 10.3 Hz, 1H), 5.78 – 5.45 (m, 1H), 4.96 (dd, *J* = 7.4, 4.0 Hz, 1H), 4.10 (q, *J* = 7.3 Hz, 2H), 3.08 – 2.56 (m, 2H), 1.21 (td, *J* = 7.2, 3.1 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 195.26, 194.70, 171.11, 171.08, 148.88, 145.03, 128.21, 126.85, 90.95, 87.58, 75.23, 70.69, 61.16, 61.07, 35.90, 35.30, 14.04.

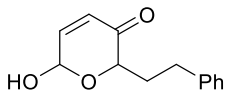
2-(4-bromophenyl)-6-hydroxy-2H-pyran-3 (6H)-one (2o)



2o

Light yellow oil. R_f = 0.42(Petroleum ether:Ethyl acetate = 2:1) ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 (d, *J* = 8.5 Hz, 2H), 7.30 – 7.15 (m, 2H), 7.00 (dd, *J* = 10.3, 3.4 Hz, 1H), 6.25 (dd, *J* = 25.8, 10.3 Hz, 1H), 5.91 – 5.67 (m, 1H), 5.58 (s, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 193.85, 148.26, 144.75, 134.22, 131.56, 129.53, 127.65, 122.69, 91.48, 88.08, 77.04, 76.10. **HRMS (ESI)**: $[M+H]^+$ calcd for $C_{11}H_{10}BrO_3^+$: 268.9808, found: 268.9805.

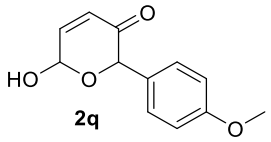
6-hydroxy-2-phenethyl-2H-pyran-3(6H)-one (2p)[10]



2p

Light yellow oil. R_f = 0.45(Petroleum ether:Ethyl acetate = 2:1) **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.46 – 7.10 (m, 5H), 7.05 – 6.79 (m, 1H), 6.15 (dd, *J* = 16.0, 10.2 Hz, 1H), 5.67 (d, *J* = 10.8 Hz, 1H), 4.59 (dd, *J* = 8.1, 3.6 Hz, 1H), 3.46 (d, *J* = 5.0 Hz, 1H), 2.80 (tq, *J* = 14.8, 8.8, 6.3 Hz, 2H), 2.48 – 1.96 (m, 2H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 196.65, 147.90, 144.54, 141.36, 128.75, 128.62, 128.58, 128.48, 128.44, 127.57, 126.09, 126.06, 90.92, 87.69, 77.76, 77.28, 73.23, 32.20, 31.36, 31.03, 31.00.

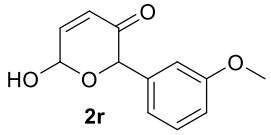
6-hydroxy-2-(4-methoxyphenyl)-2H-pyran-3(6H)-one(2q)[10]



2q

Light yellow oil. R_f = 0.15(Petroleum ether:Ethyl acetate = 2:1) **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.34 – 7.21 (m, 2H), 7.01 – 6.88 (m, 3H), 6.23 (ddd, *J* = 19.6, 10.3, 1.2 Hz, 1H), 5.67 (s, 1H), 5.53 (s, 1H), 3.82 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 195.10, 159.78, 148.44, 145.00, 129.35, 129.32, 129.28, 127.75, 127.47, 113.99, 113.96, 91.46, 88.02, 80.83, 76.69, 55.32.

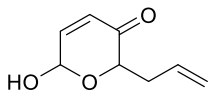
6-hydroxy-2-(3-methoxyphenyl)-2H-pyran-3 (6H)-one (2r)



2r

Light yellow oil. R_f = 0.15(Petroleum ether:Ethyl acetate = 2:1) **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.39 – 7.23 (m, 1H), 7.09 – 6.80 (m, 3H), 6.18 (d, *J* = 10.3 Hz, 1H), 5.60 (d, *J* = 50.1 Hz, 1H), 3.80 (s, 2H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 194.65, 159.60, 159.55, 148.64, 145.25, 136.70, 129.50, 129.47, 129.18, 127.61, 120.42, 120.36, 114.28, 114.25, 113.54, 113.52, 91.43, 87.95, 80.95, 77.32, 76.89, 55.31, 55.30. **HRMS (E**SI): $[M+H]^+$ calcd for $C_{12}H_{13}O_4^+$: 221.0808, found: 221.0804.

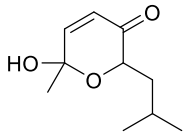
2-allyl-6-hydroxy-2H-pyran-3(6H)-one(2s)[10]



2s

Light yellow oil. R_f = 0.3(Petroleum ether:Ethyl acetate = 1:1) **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.10 – 6.76 (m, 1H), 6.15 (ddd, *J* = 26.9, 10.5, 3.2 Hz, 1H), 5.84 (tp, *J* = 16.7, 6.4 Hz, 1H), 5.67 (d, *J* = 4.0 Hz, 1H), 5.29 – 4.94 (m, 2H), 4.68 (dt, *J* = 8.0, 3.8 Hz, 1H), 3.29 (t, *J* = 4.2 Hz, 1H), 2.91 – 2.62 (m, 1H), 2.62 – 2.36 (m, 1H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 196.83, 196.35, 148.10, 144.44, 129.44, 128.12, 91.17, 87.59, 83.02, 78.45, 38.57, 38.43, 29.42, 29.32, 26.63, 26.53, 26.46, 26.38, 26.15, 26.14, 26.10.

6-hydroxy-2-isobutyl-6-methyl-2H-pyran-3(6H)-one (2t)



2t

Light yellow oil. R_f = 0.42(Petroleum ether:Ethyl acetate = 2:1) **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.04 – 6.65 (m, 1H), 6.06 (dd, *J* = 11.1, 4.5 Hz, 1H), 5.71 (d, *J* = 6.1 Hz, 1H), 3.37 (t, *J* = 5.7 Hz, 1H), 2.17 – 1.49 (m, 9H), 1.39 – 1.13 (m, 1H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 199.25, 145.32, 126.86, 87.57, 80.68, 33.45, 31.03, 25.15, 21.00, 20.57. **HRMS (ESI)**: $[M+H]^+$ calcd for $C_{10}H_{17}O_3^+$: 185.1172, found: 185.1177.

8 References

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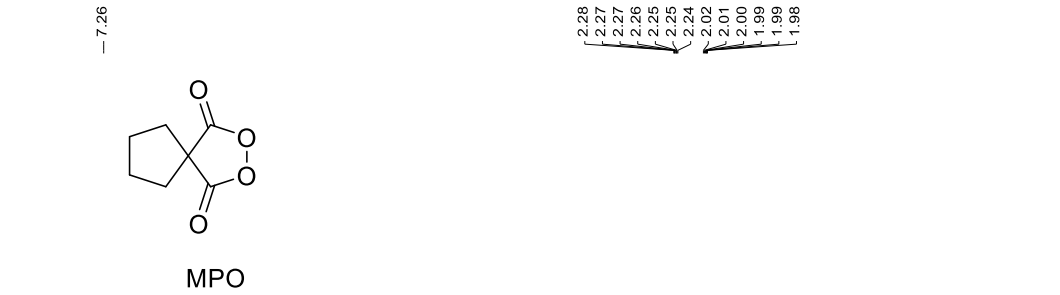
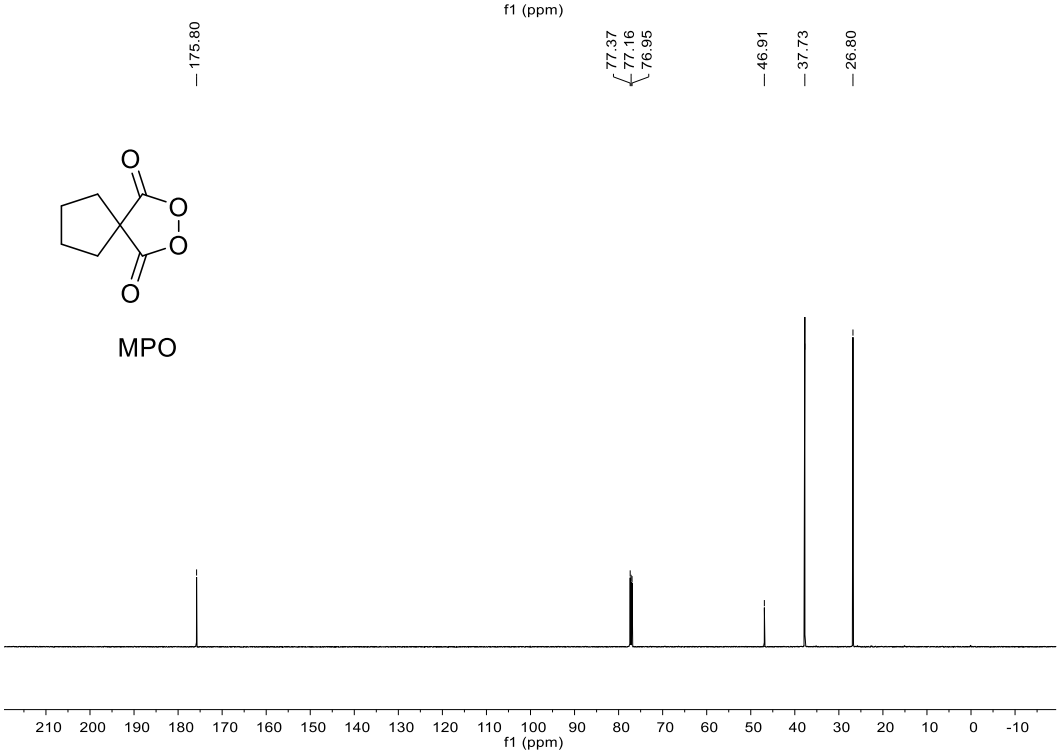
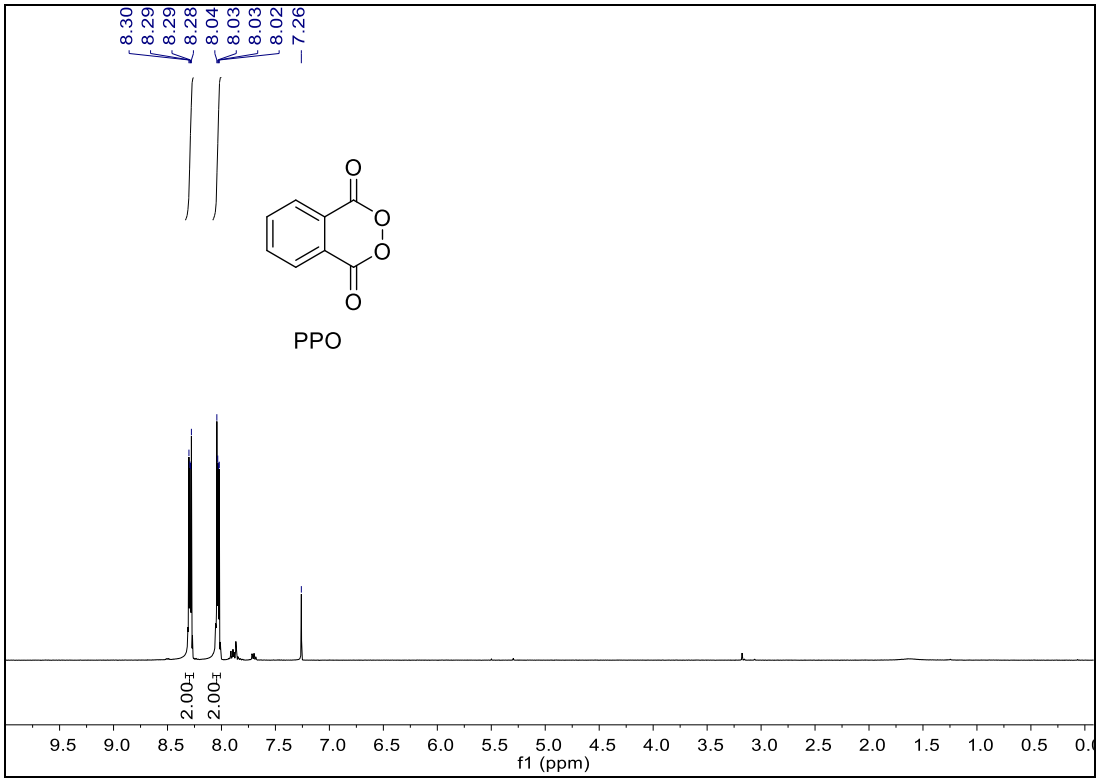
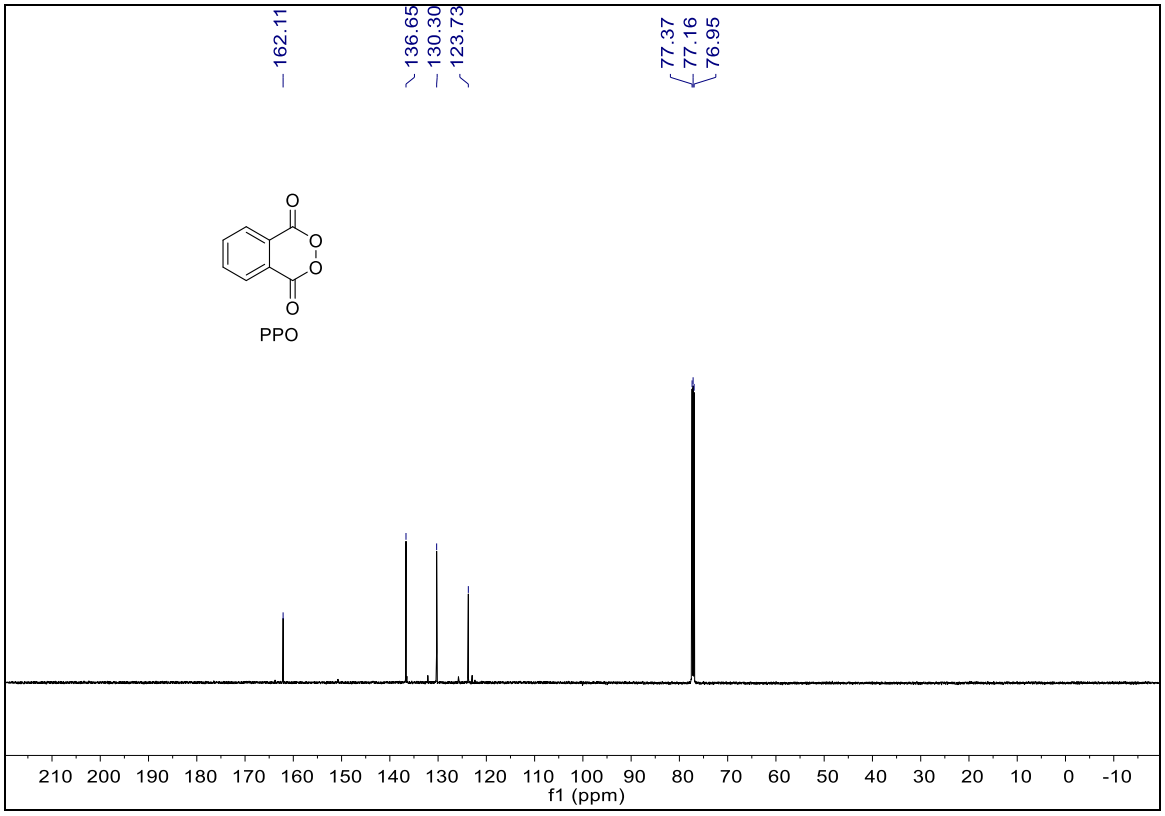
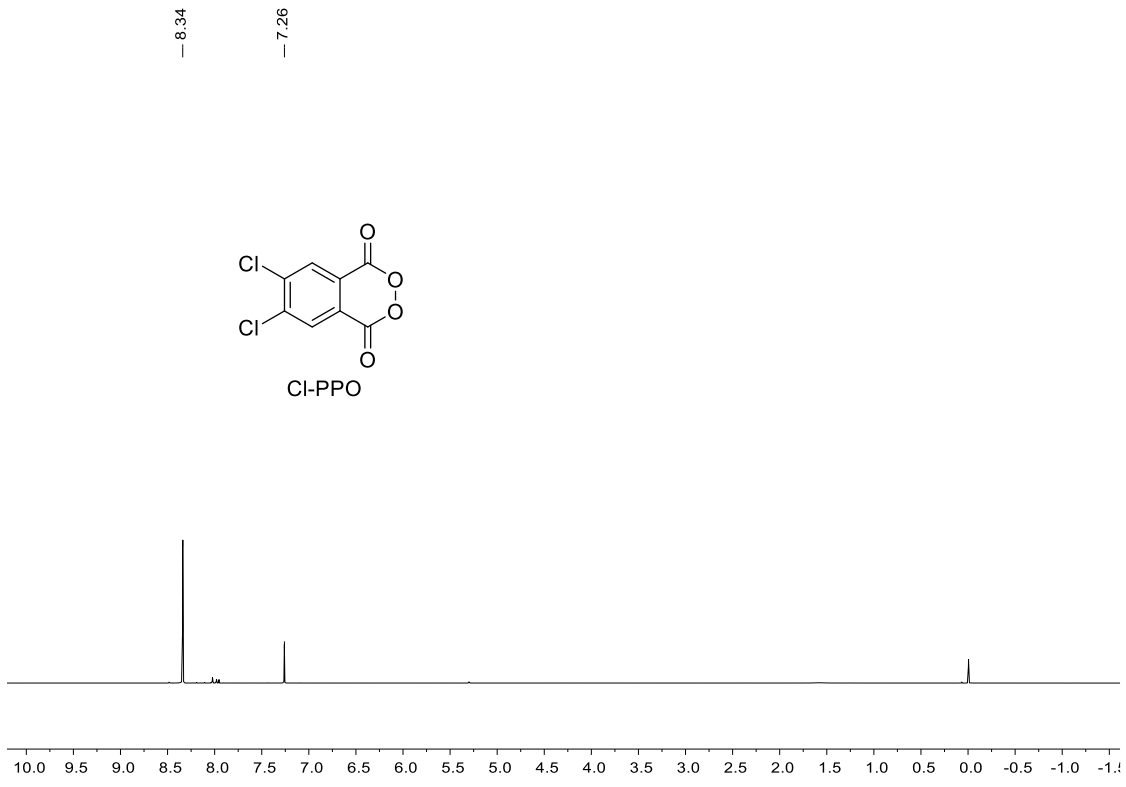
9 ^{1}H, ^{13}C NMR spectra of products

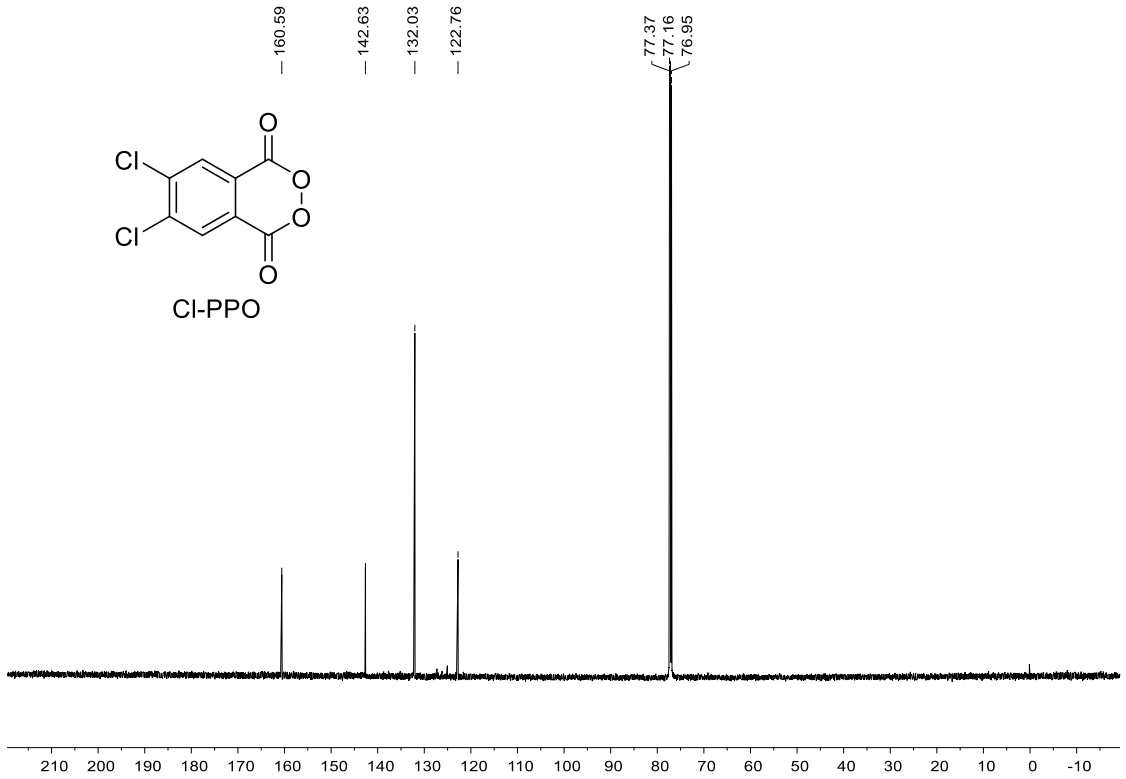


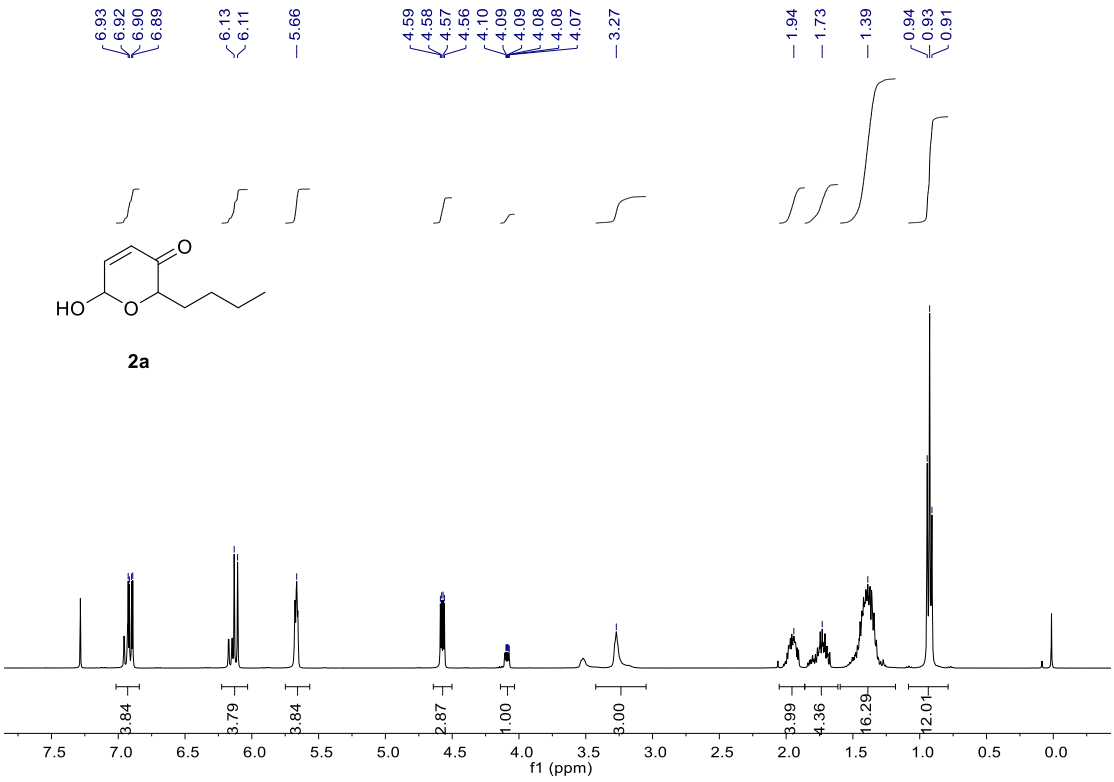



8.30
8.29
8.29
8.28
8.04
8.03
8.03
8.02
7.26
PPO
2.00
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

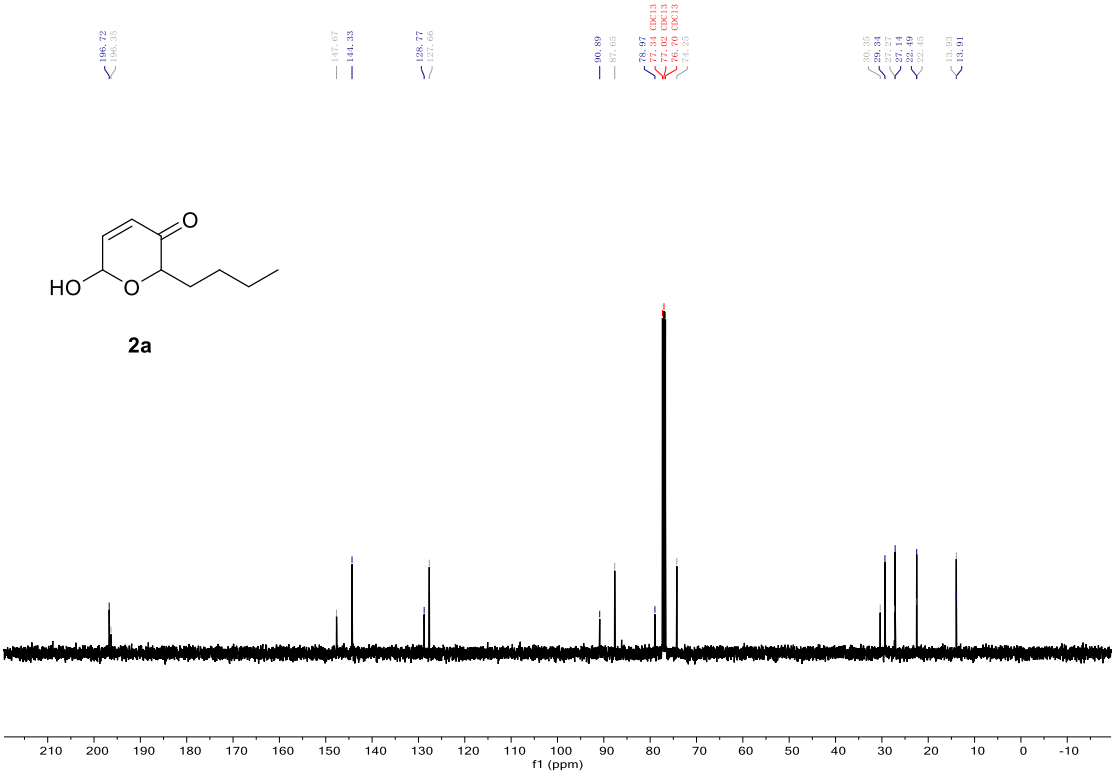

162.11
136.65
130.30
123.73
77.37
77.16
76.95
PPO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.34
7.26
Cl
Cl
O
O
O
O
Cl-PPO
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0

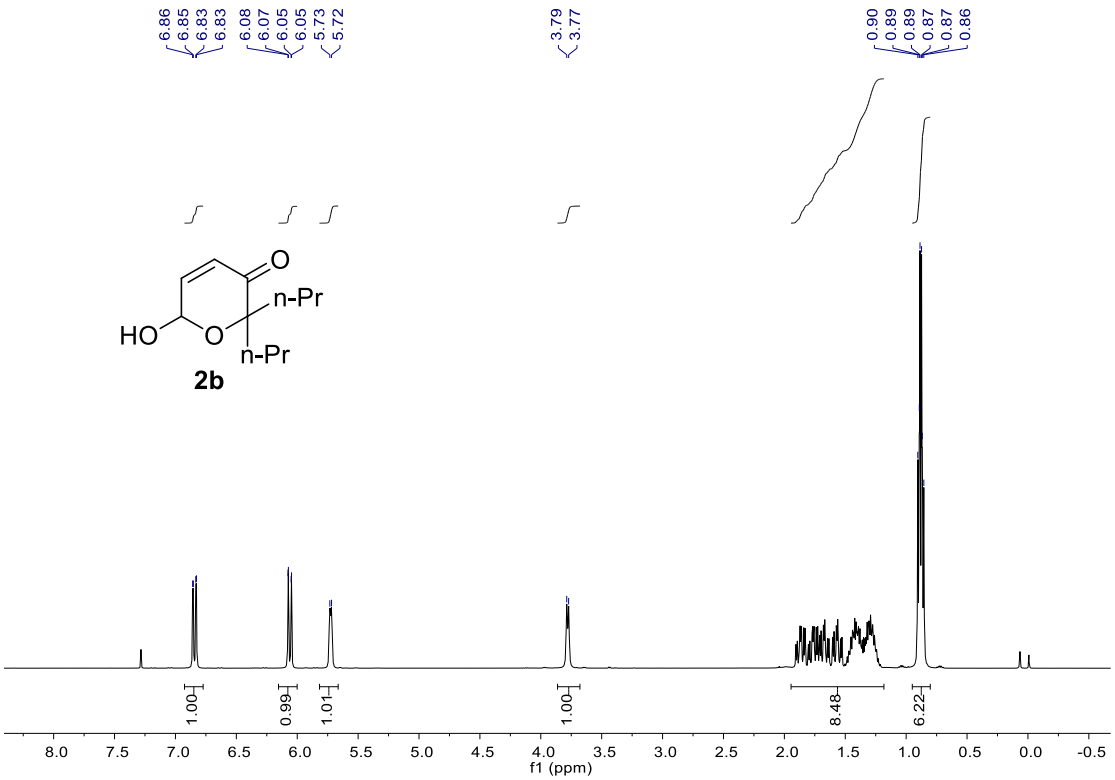

160.59
142.63
132.03
122.76
77.37
77.16
76.95
Cl
Cl
O
O
O
O
Cl-PPO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

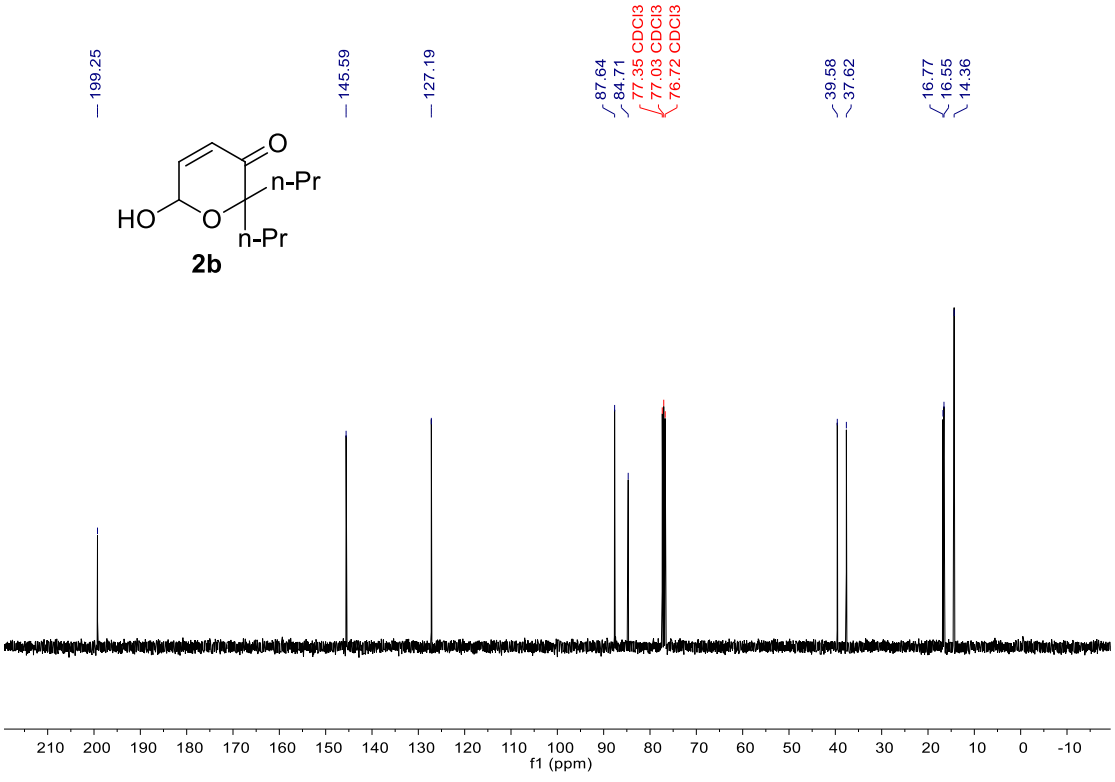

6.93
6.92
6.90
6.89
6.13
6.11
5.66
4.59
4.58
4.57
4.56
4.10
4.09
4.09
4.08
4.08
4.07
3.27
1.94
1.73
1.39
0.94
0.93
0.91
HO
O
O
3.84
3.79
3.84
2.87
1.00
3.00
3.99
4.36
16.29
12.01
f1 (ppm)

2a


196.72
166.55
147.67
144.33
128.77
127.66
90.89
87.65
78.97
77.34 CDCl3
77.02 CDCl3
76.70 CDCl3
74.55
30.35
29.34
27.27
22.14
22.00
22.45
13.93
13.91
HO
O
O
f1 (ppm)

2a


6.86
6.85
6.83
6.83
6.08
6.07
6.05
6.05
5.73
5.72
3.79
3.77
0.90
0.89
0.89
0.87
0.87
0.86
O
n-Pr
HO
O
n-Pr
2b
1.00
0.99
1.01
1.00
8.48
6.22
f1 (ppm)


199.25
145.59
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87.64
84.71
77.35 CDCl3
77.03 CDCl3
76.72 CDCl3
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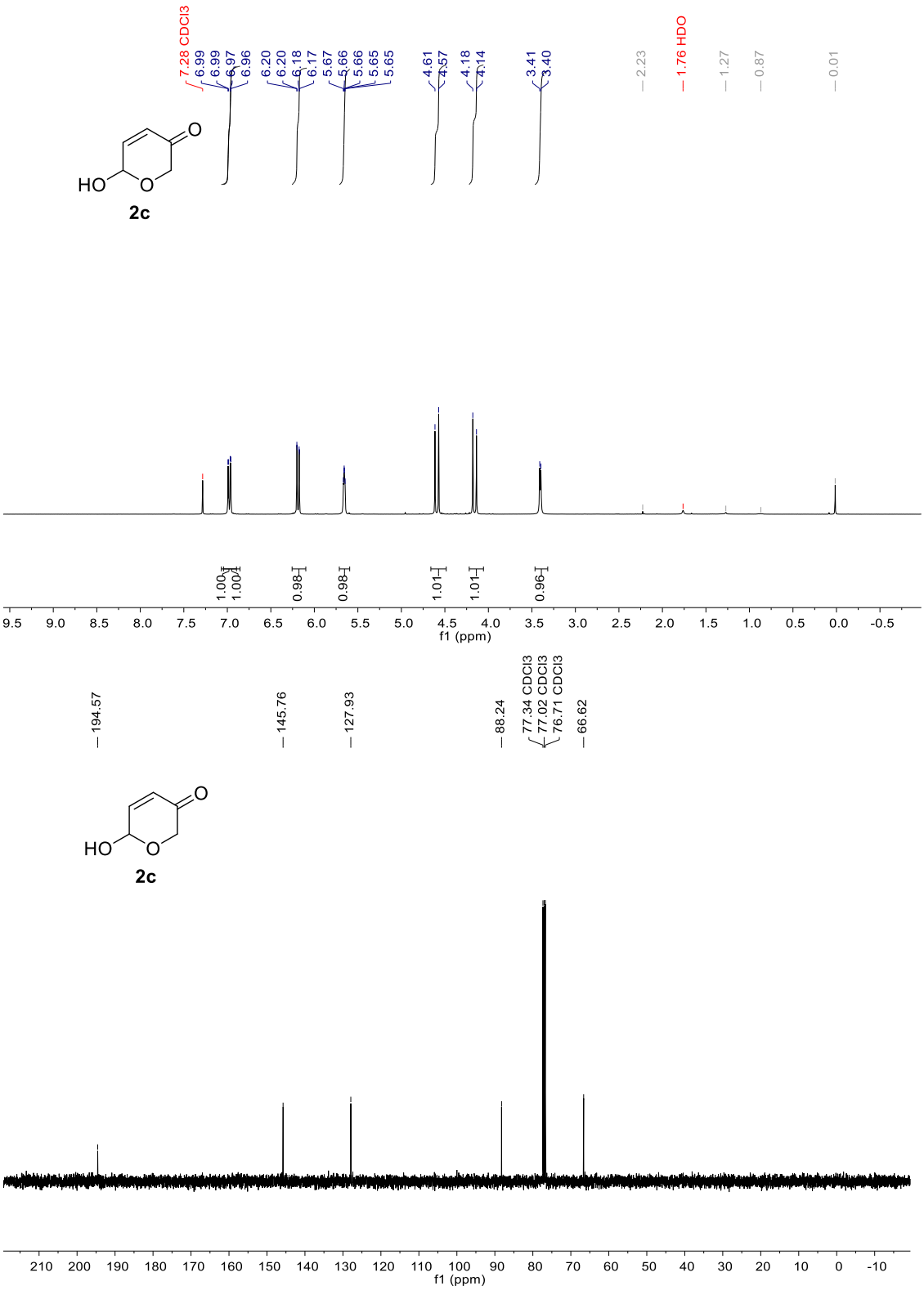
16.77
16.55
14.36
O
n-Pr
HO
O
n-Pr
2b
f1 (ppm)


7.28 CDCl3
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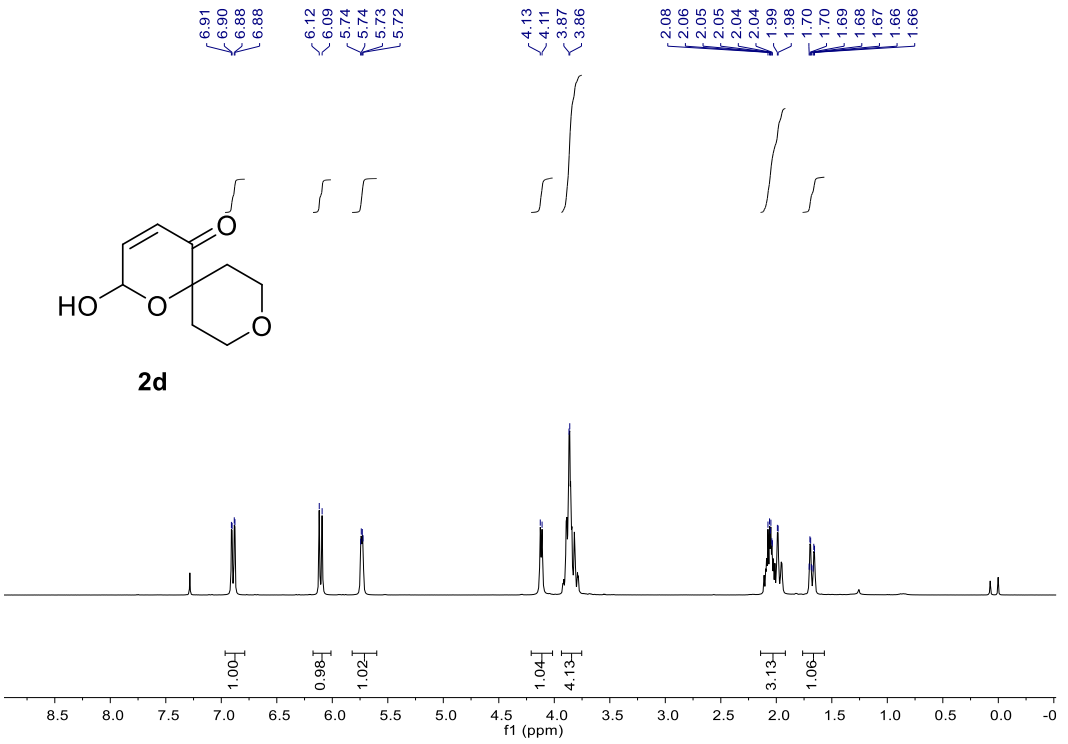
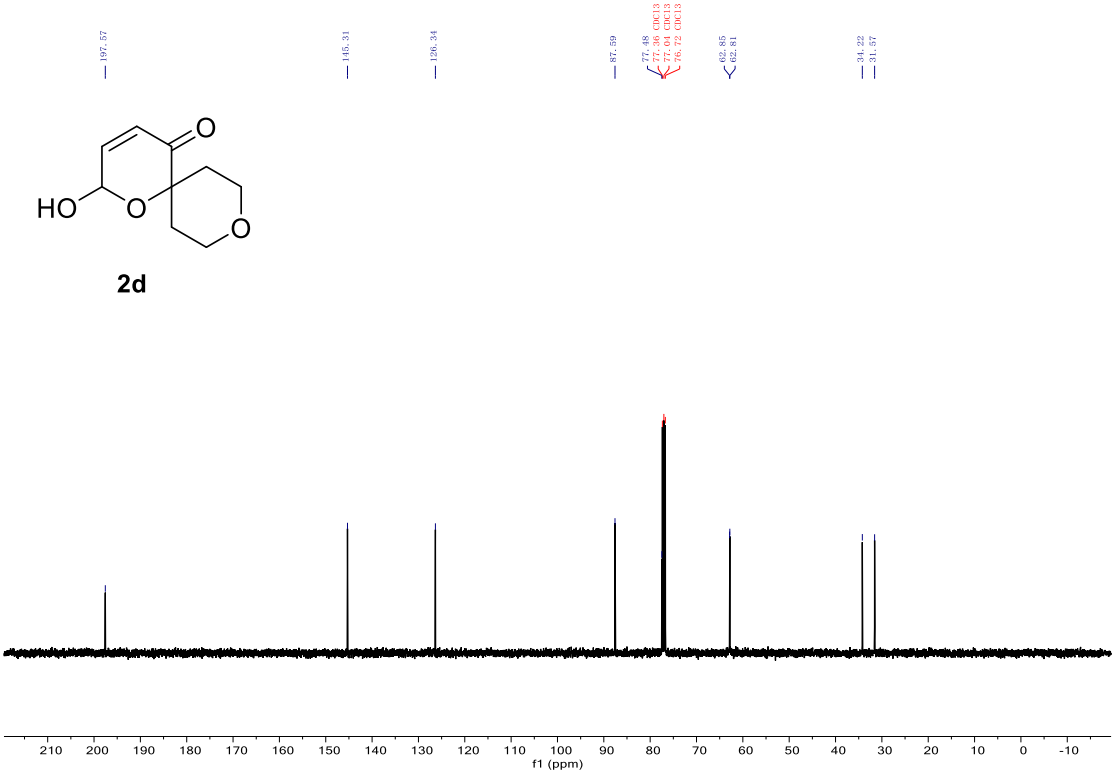
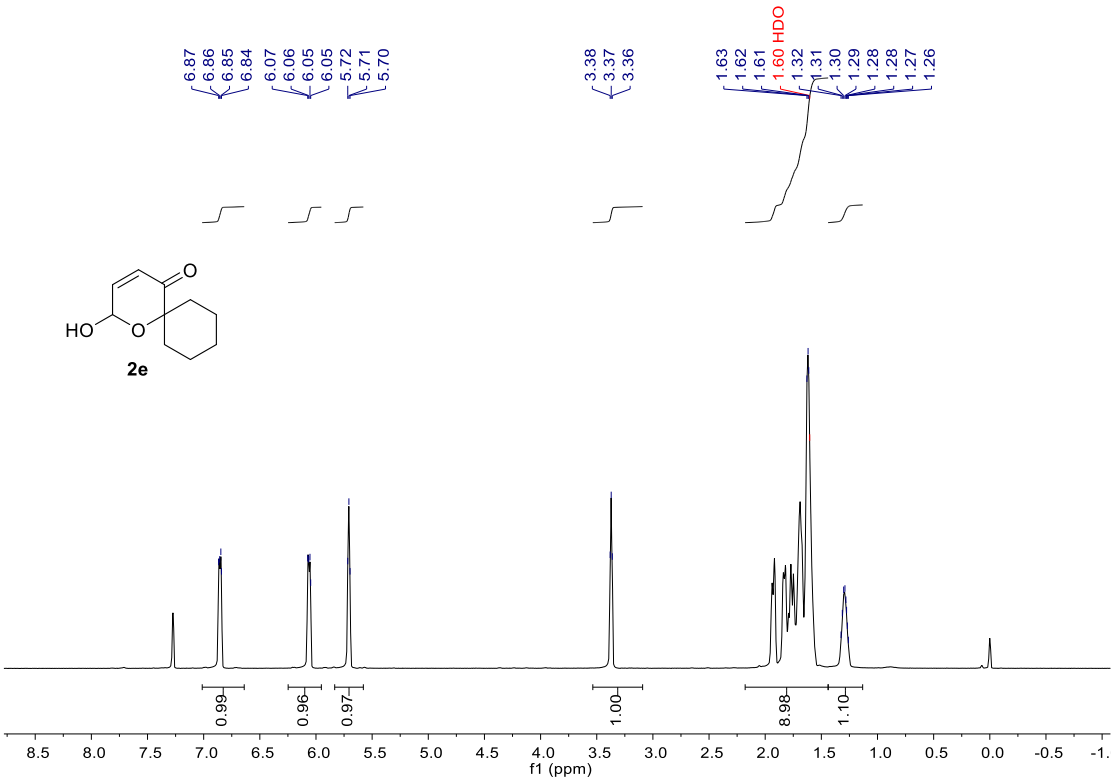
6.99
6.97
6.96
6.20
6.20
6.18
6.17
5.67
5.66
5.66
5.65
5.65
4.61
4.57
4.18
4.14
3.41
3.40
2.23
1.76 HDO
1.27
0.87
0.01
HO
O
O
2c
1.00
1.00
0.98
0.98
1.01
1.01
0.96
f1 (ppm)
194.57
145.76
127.93
88.24
77.34 CDCl3
77.02 CDCl3
76.71 CDCl3
66.62
HO
O
O
2c
f1 (ppm)

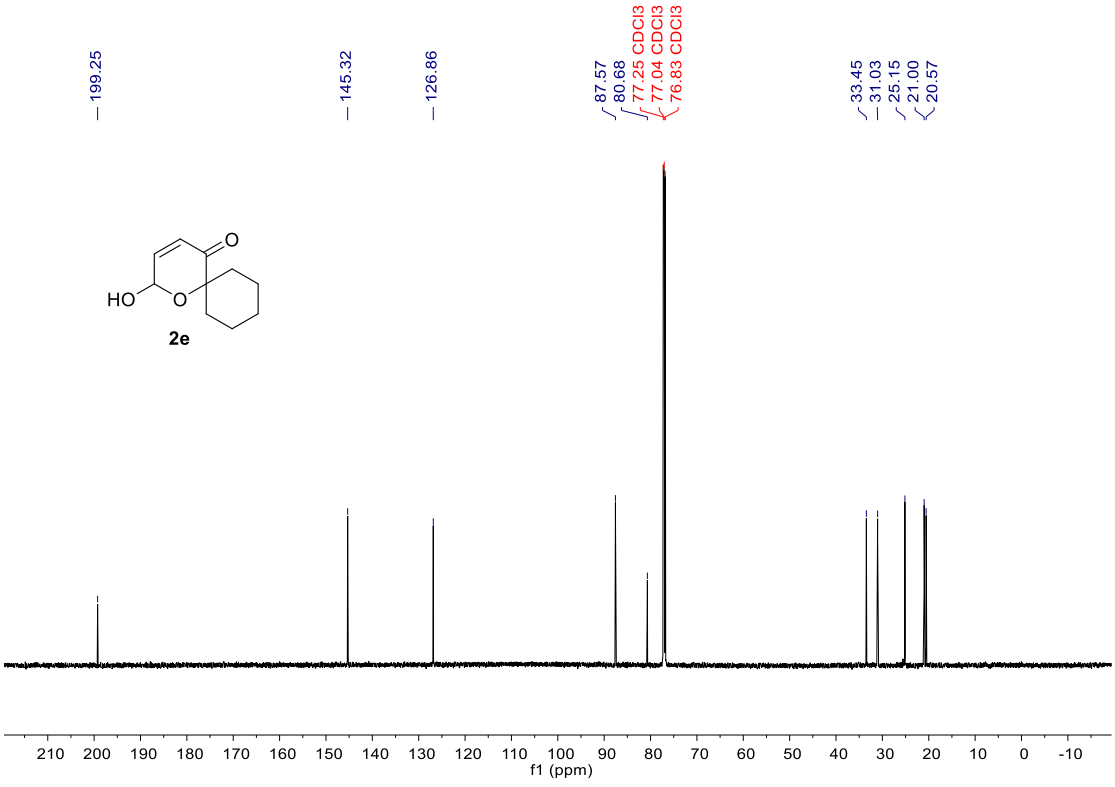

6.91
6.90
6.88
6.88
6.12
6.09
5.74
5.74
5.73
5.72
4.13
4.11
3.87
3.86
2.08
2.06
2.05
2.05
2.04
2.04
1.99
1.98
1.70
1.70
1.69
1.68
1.67
1.66
1.66
HO
O
O
O
1.00
0.98
1.02
1.04
4.13
3.13
1.06
f1 (ppm)

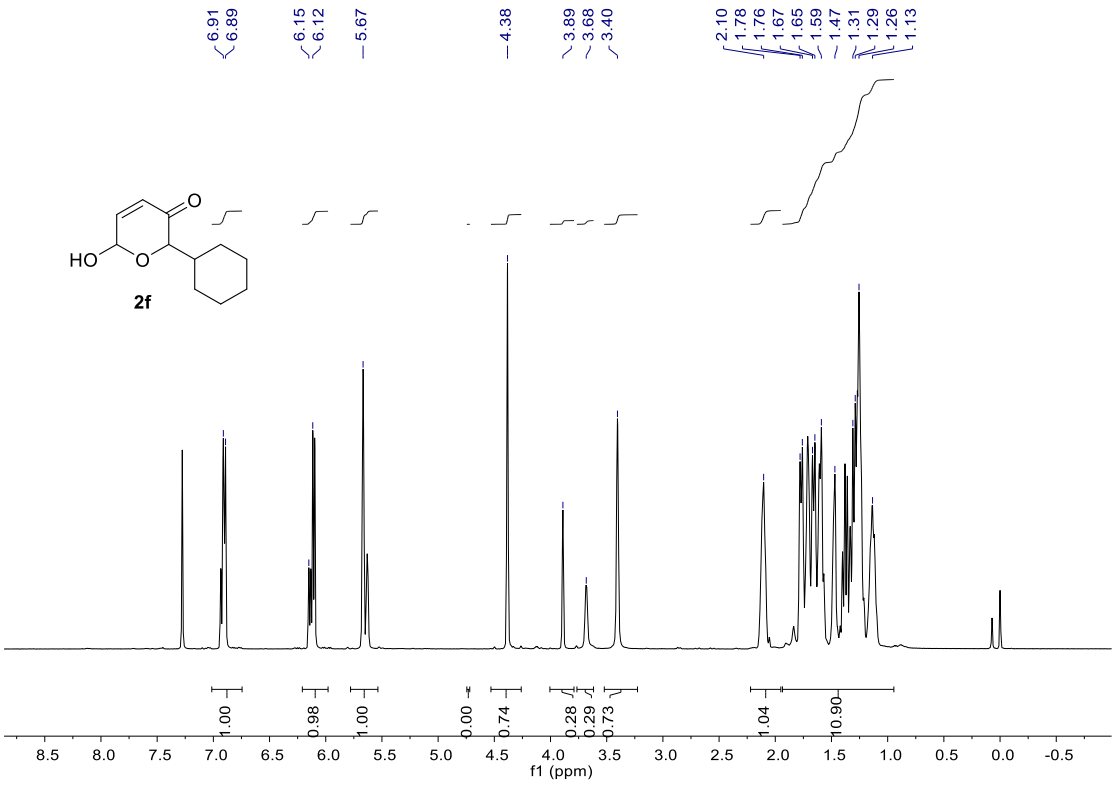
2d

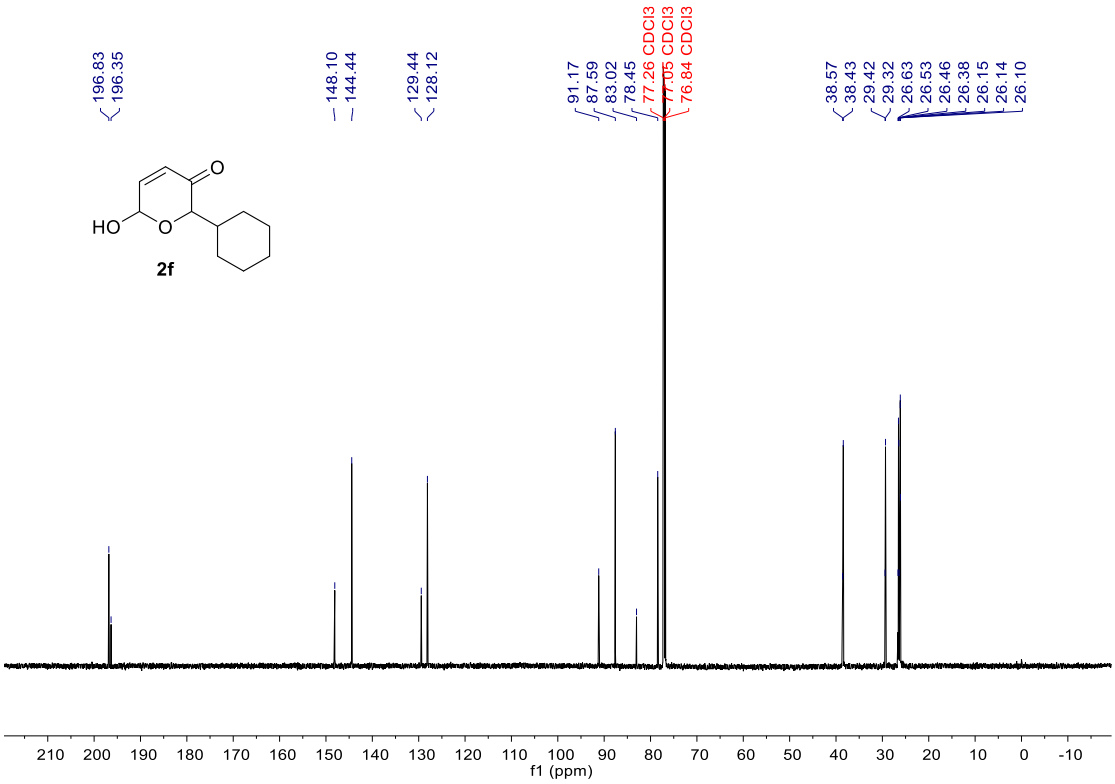

197.57
145.31
126.34
87.59
77.48
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
62.85
62.81
34.22
31.67
HO
O
O
O
f1 (ppm)

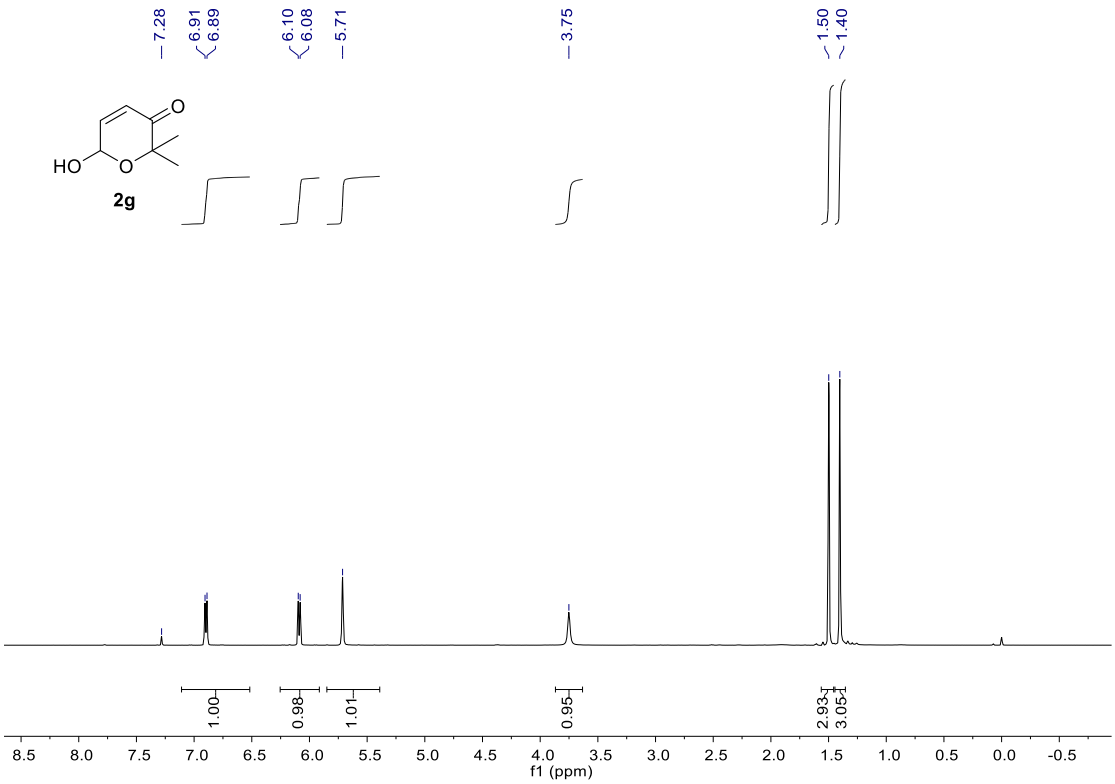
2d

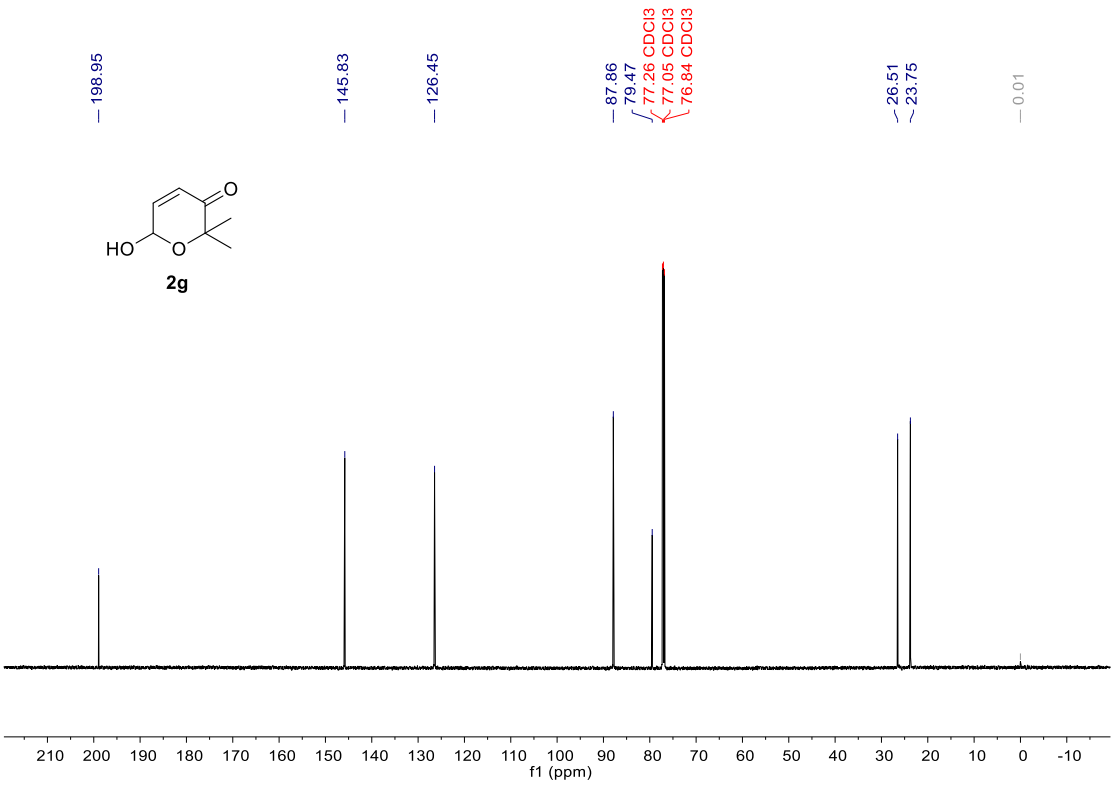

6.87
6.86
6.85
6.84
6.07
6.06
6.05
6.05
5.72
5.71
5.70
3.38
3.37
3.36
1.63
1.62
1.61
1.60 HDO
1.32
1.31
1.30
1.29
1.28
1.28
1.27
1.26
HO
O
O
2e
0.99
0.96
0.97
1.00
8.98
1.10
f1 (ppm)

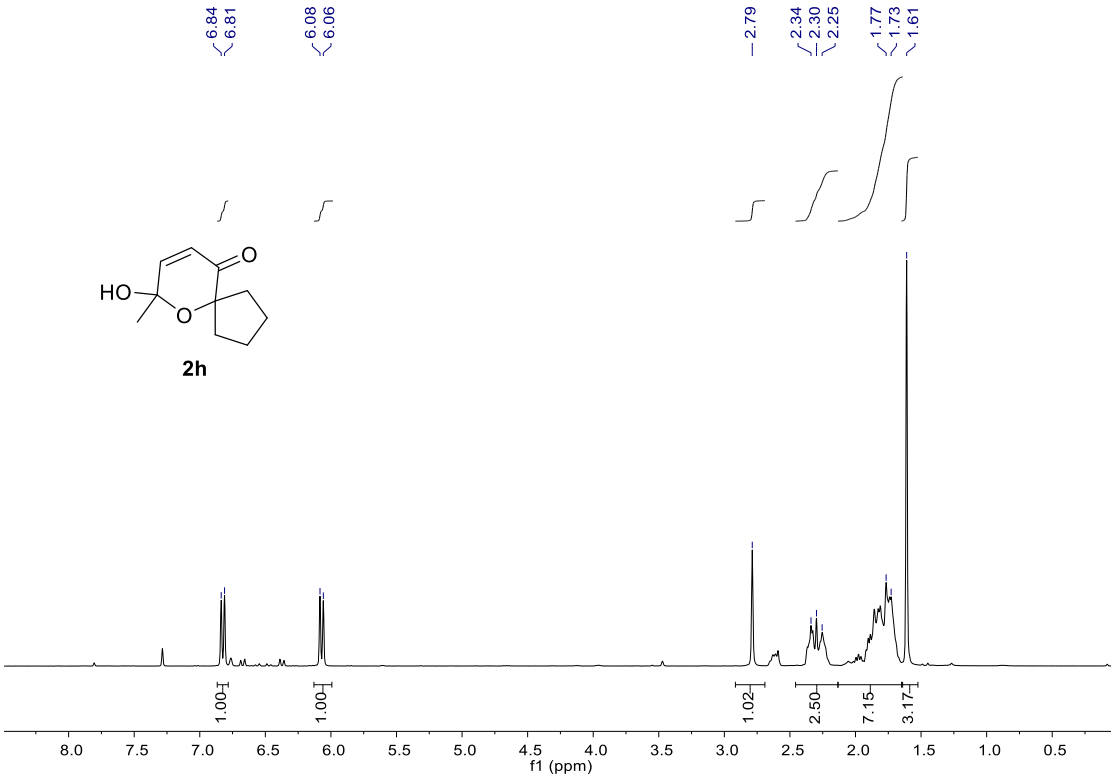

199.25
145.32
126.86
87.57
80.68
77.25 CDCl3
77.04 CDCl3
76.83 CDCl3
33.45
31.03
25.15
21.00
20.57
HO
O
O
2e
f1 (ppm)

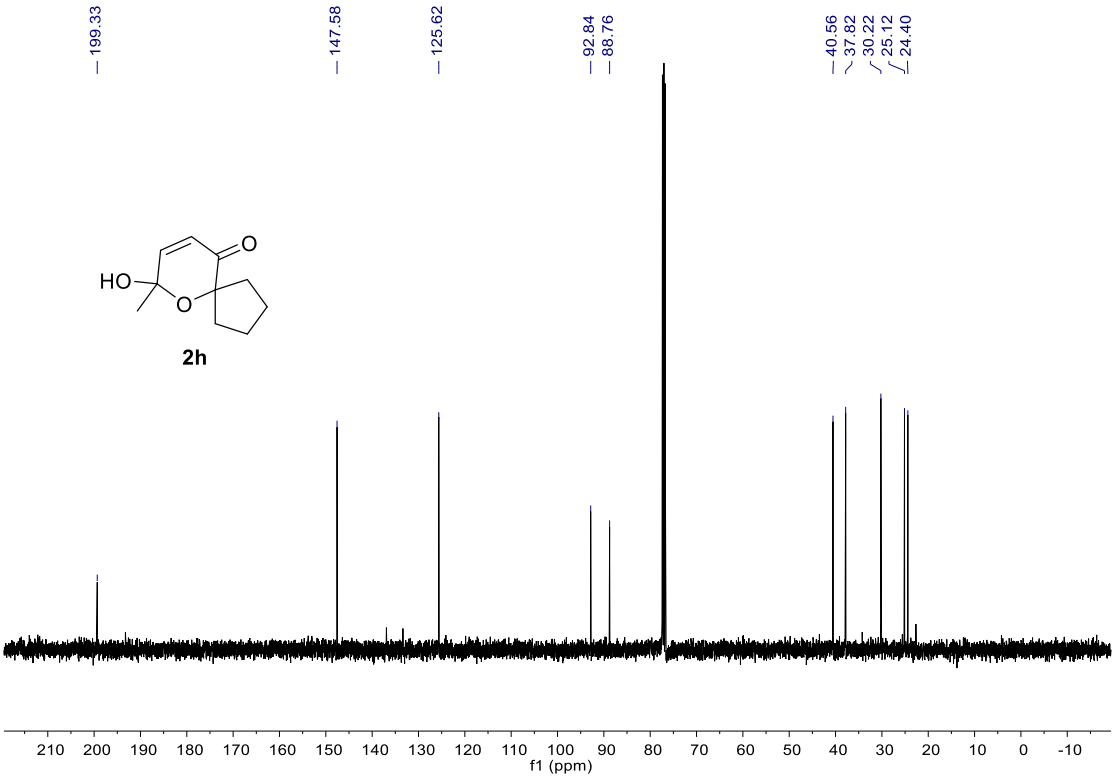

6.91
6.89
6.15
6.12
5.67
4.38
3.89
3.68
3.40
2.10
1.78
1.76
1.67
1.65
1.59
1.47
1.31
1.29
1.26
1.13
HO
O
O
2f
1.00
0.98
1.00
0.00
0.74
0.28
0.29
0.73
1.04
10.90
f1 (ppm)

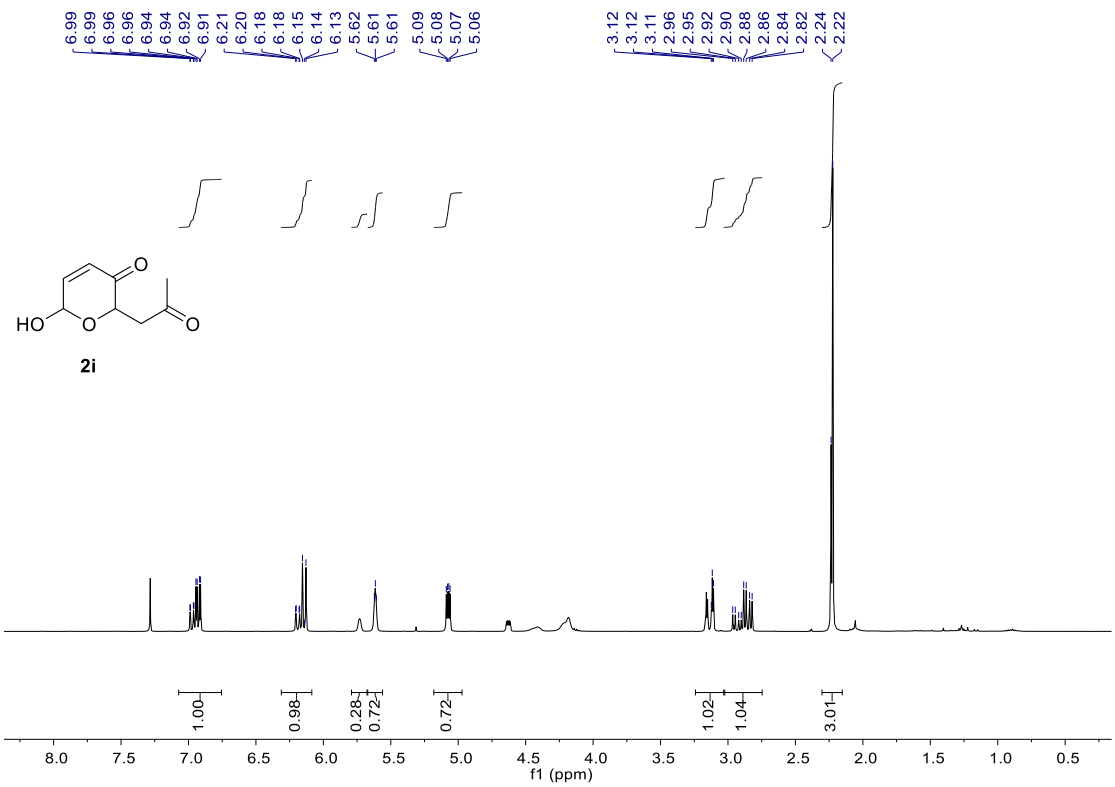

196.83
196.35
148.10
144.44
129.44
128.12
91.17
87.59
83.02
78.45
77.26 CDCl3
77.05 CDCl3
76.84 CDCl3
38.57
38.43
29.42
29.32
26.63
26.53
26.46
26.38
26.15
26.14
26.10
HO
O
O
2f
f1 (ppm)

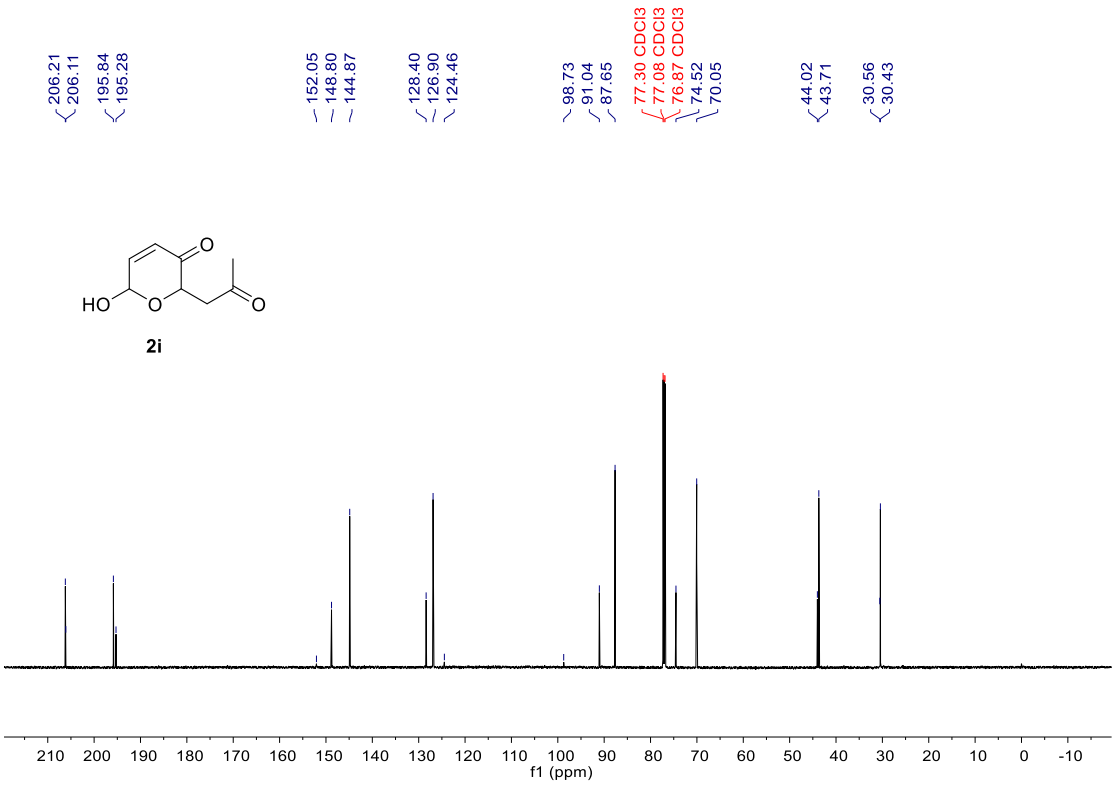

7.28
6.91
6.89
6.10
6.08
5.71
3.75
1.50
1.40
HO
O
O
2g
1.00
0.98
1.01
0.95
2.93
3.05
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

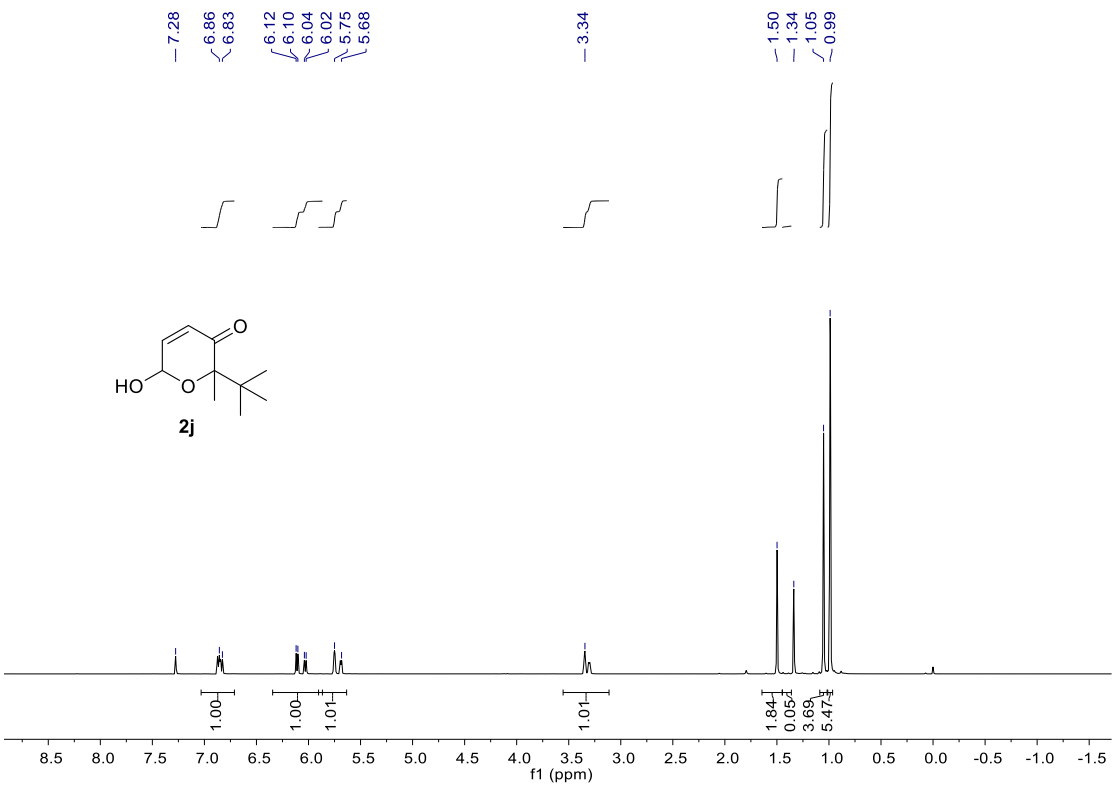

198.95
145.83
126.45
87.86
79.47
77.26 CDCl3
77.05 CDCl3
76.84 CDCl3
26.51
23.75
0.01
HO
O
O
2g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

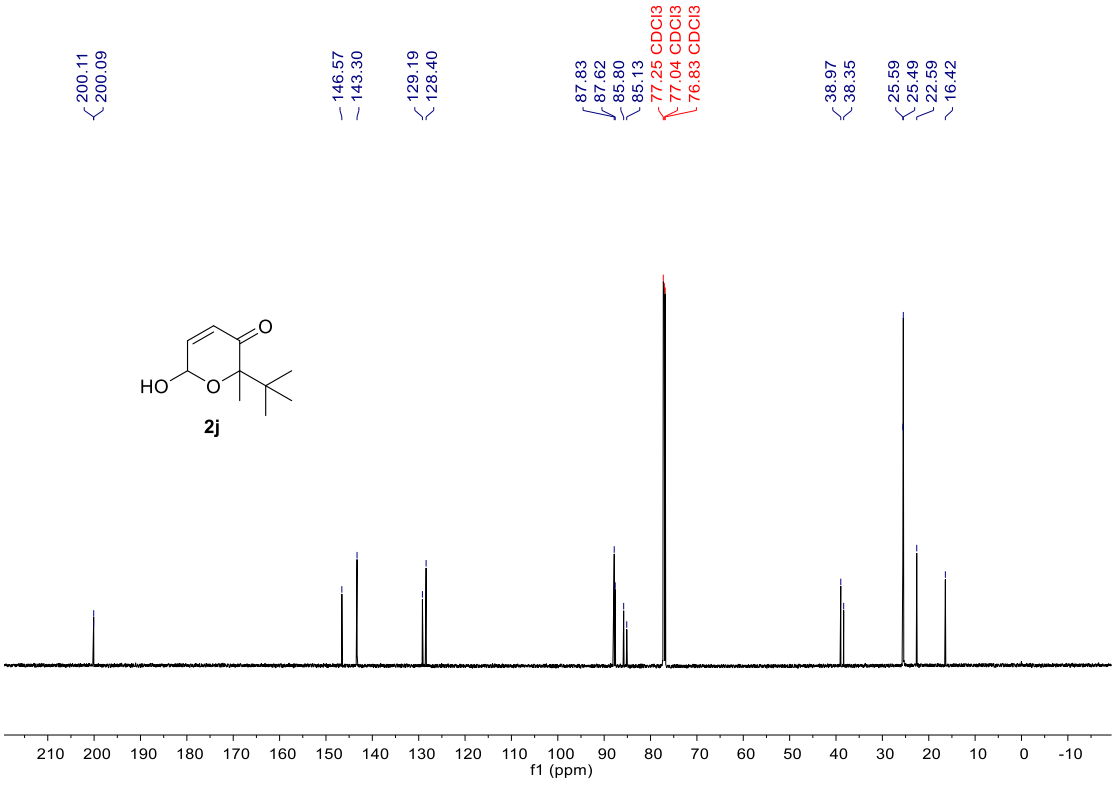

6.84
6.81
6.08
6.06
2.79
2.34
2.30
2.25
1.77
1.73
1.61
HO
O
O
2h
1.00
1.00
1.02
2.50
7.15
3.17
f1 (ppm)

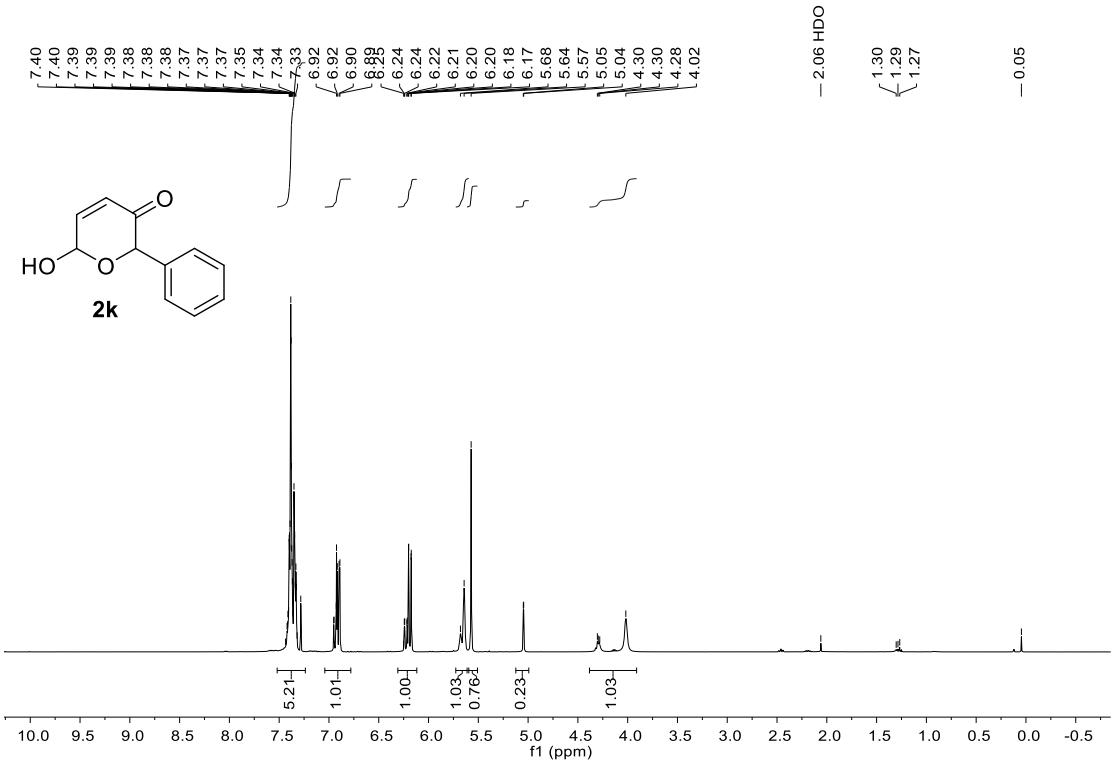

199.33
147.58
125.62
92.84
88.76
40.56
37.82
30.22
25.12
24.40
HO
O
O
2h
f1 (ppm)

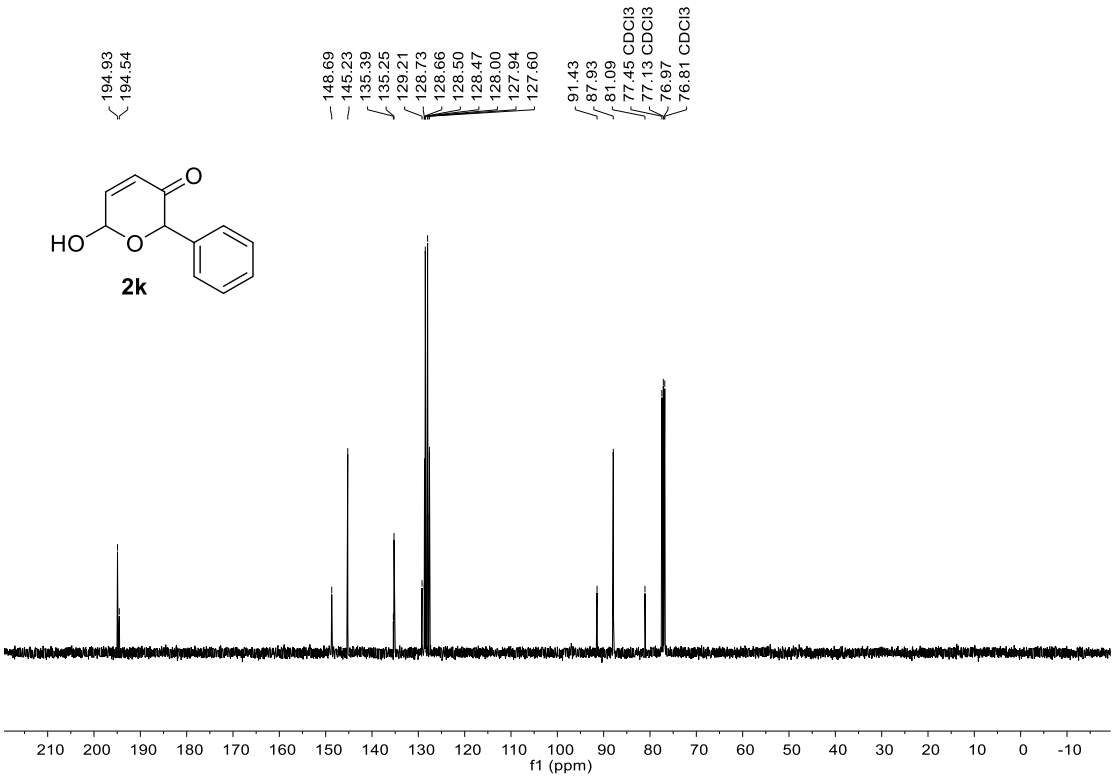

6.99
6.99
6.96
6.96
6.94
6.94
6.92
6.91
6.21
6.20
6.18
6.18
6.15
6.14
6.13
5.62
5.61
5.61
5.09
5.08
5.07
5.06
3.12
3.12
3.11
2.96
2.95
2.92
2.90
2.88
2.86
2.84
2.82
2.24
2.22
HO
O
O
O
2i
1.00
0.98
0.28
0.72
0.72
1.02
1.04
3.01
f1 (ppm)


206.21
206.11
195.84
195.28
152.05
148.80
144.87
128.40
126.90
124.46
98.73
91.04
87.65
77.30 CDCl3
77.08 CDCl3
76.87 CDCl3
74.52
70.05
44.02
43.71
30.56
30.43
HO
O
O
O
2i
f1 (ppm)


7.28
6.86
6.83
6.12
6.10
6.04
6.02
5.75
5.68
3.34
1.50
1.34
1.05
0.99
HO
O
O
2j
1.00
1.00
1.01
1.01
1.84
0.05
3.69
5.47
f1 (ppm)


200.11
200.09
146.57
143.30
129.19
128.40
87.83
87.62
85.80
85.13
77.25 CDCl3
77.04 CDCl3
76.83 CDCl3
38.97
38.35
25.59
25.49
22.59
16.42
HO
O
O
2j
f1 (ppm)


7.40
7.40
7.39
7.39
7.39
7.38
7.38
7.38
7.37
7.37
7.37
7.35
7.34
7.34
7.33
6.92
6.92
6.90
6.89
6.25
6.24
6.24
6.22
6.21
6.20
6.20
6.18
6.17
5.68
5.64
5.57
5.05
5.04
4.30
4.30
4.28
4.02
2.06 HDO
1.30
1.29
1.27
0.05
HO
O
O
2k
5.21
1.01
1.00
1.03
0.76
0.23
1.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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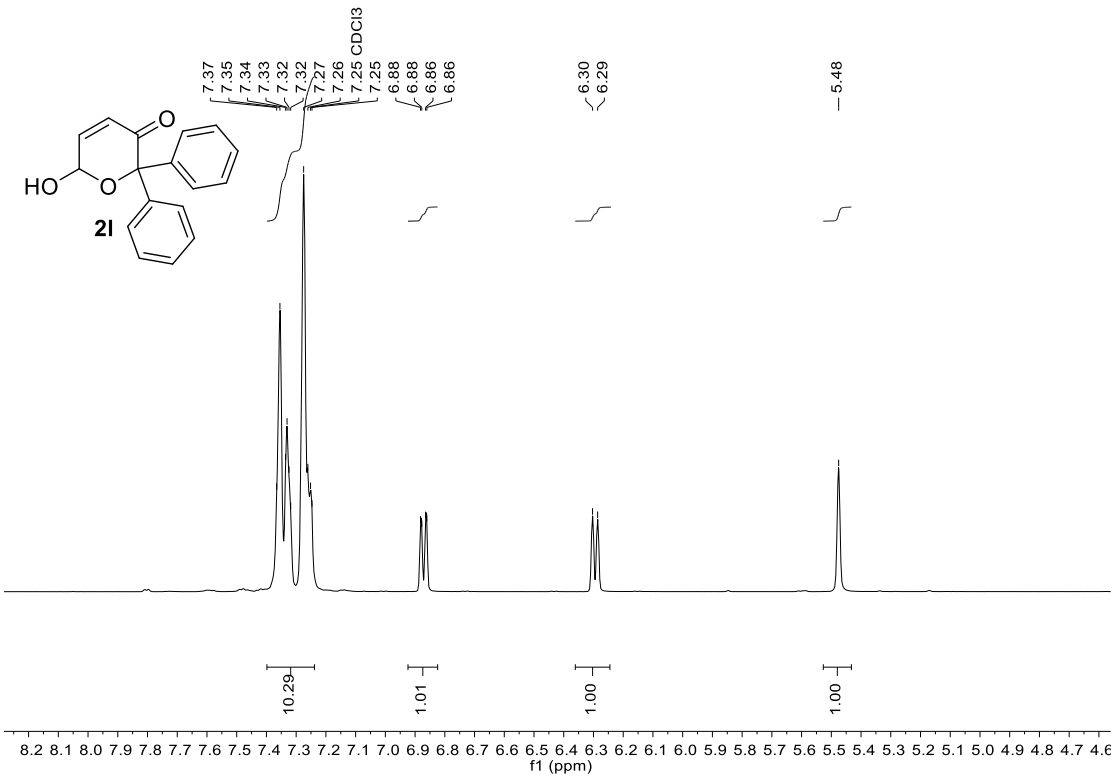
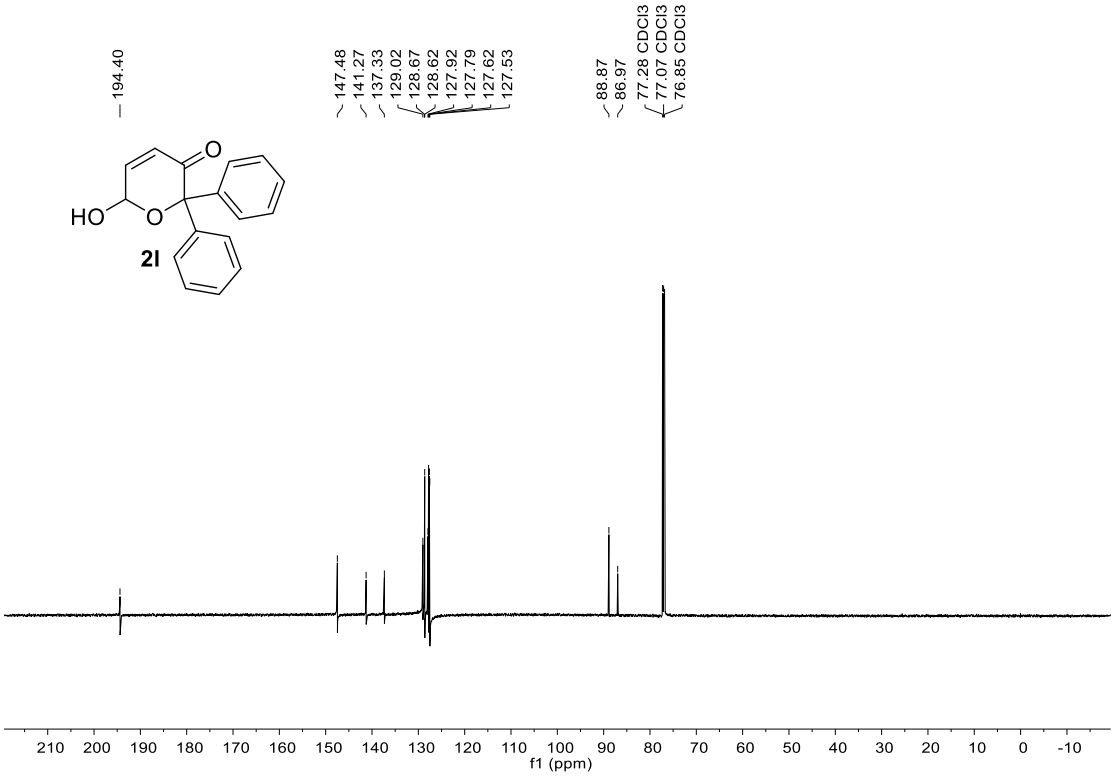
194.54
148.69
145.23
135.39
135.25
129.21
128.73
128.66
128.50
128.47
128.00
127.94
127.60
91.43
87.93
81.09
77.45 CDCl3
77.13 CDCl3
76.97
76.81 CDCl3
HO
O
O
2k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.37
7.35
7.34
7.33
7.32
7.32
7.27
7.26
7.25 CDCl3
7.25
6.88
6.88
6.86
6.86
6.30
6.29
5.48
HO
O
O
2l
10.29
1.01
1.00
1.00
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6
f1 (ppm)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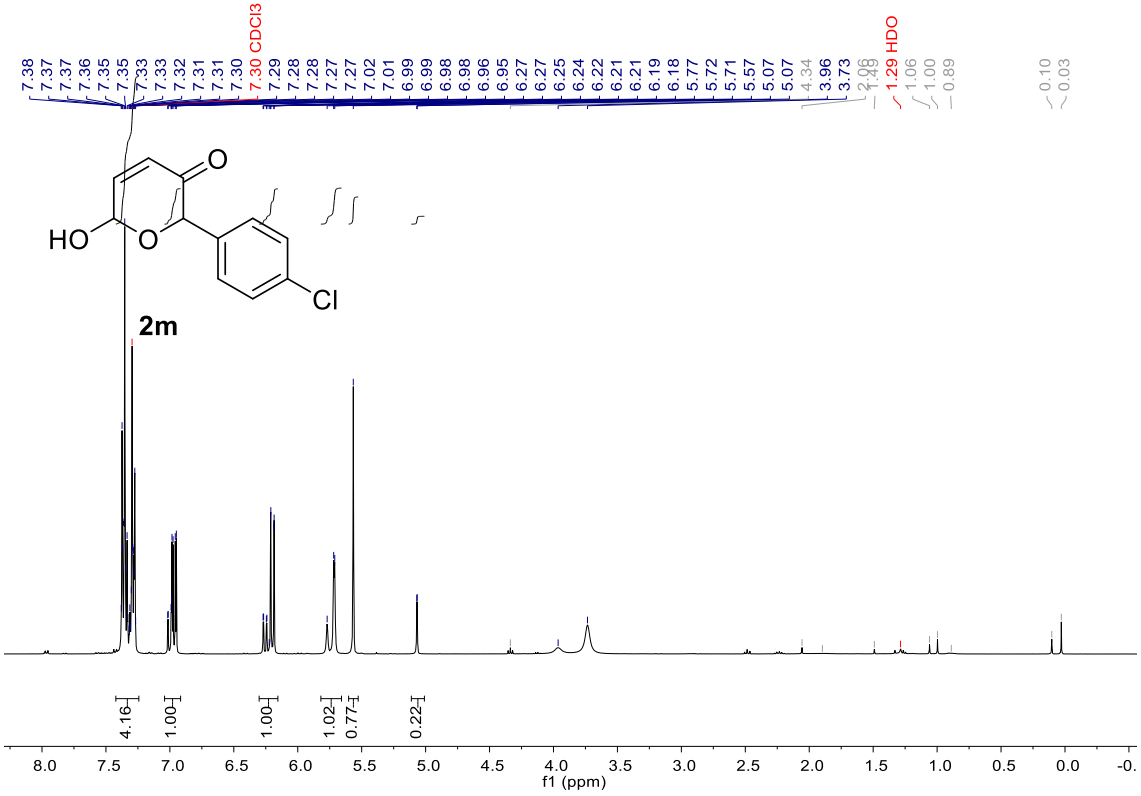
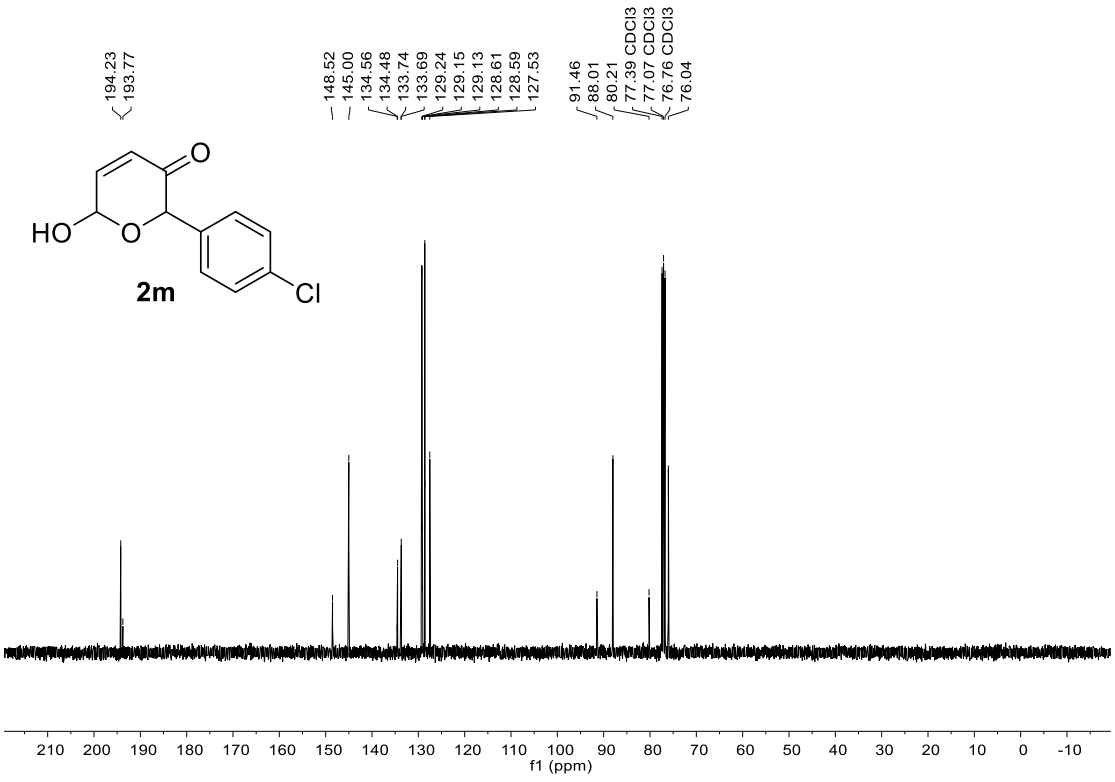
147.48
141.27
137.33
129.02
128.67
128.62
127.92
127.79
127.62
127.53
88.87
86.97
77.28 CDCl3
77.07 CDCl3
76.85 CDCl3
HO
O
O
2l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.38
7.37
7.37
7.36
7.35
7.35
7.33
7.33
7.32
7.31
7.31
7.30
7.30 CDCl3
7.29
7.28
7.28
7.27
7.27
7.02
7.01
6.99
6.99
6.98
6.98
6.96
6.95
6.27
6.27
6.25
6.24
6.22
6.21
6.21
6.19
6.18
5.77
5.72
5.71
5.57
5.07
5.07
4.34
3.96
3.73
2.06
1.49
1.29 HDO
1.06
1.00
0.89
0.10
0.03
HO
O
O
Cl
2m
4.16
1.00
1.00
1.02
0.77
0.22
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.
f1 (ppm)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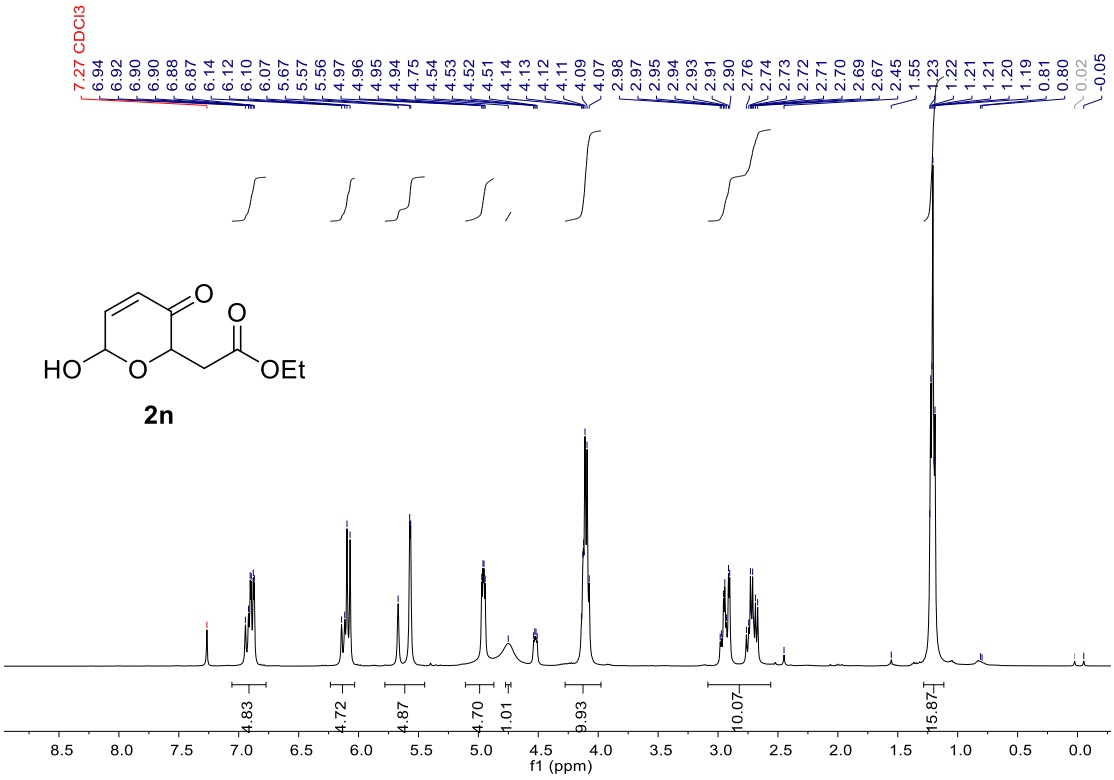
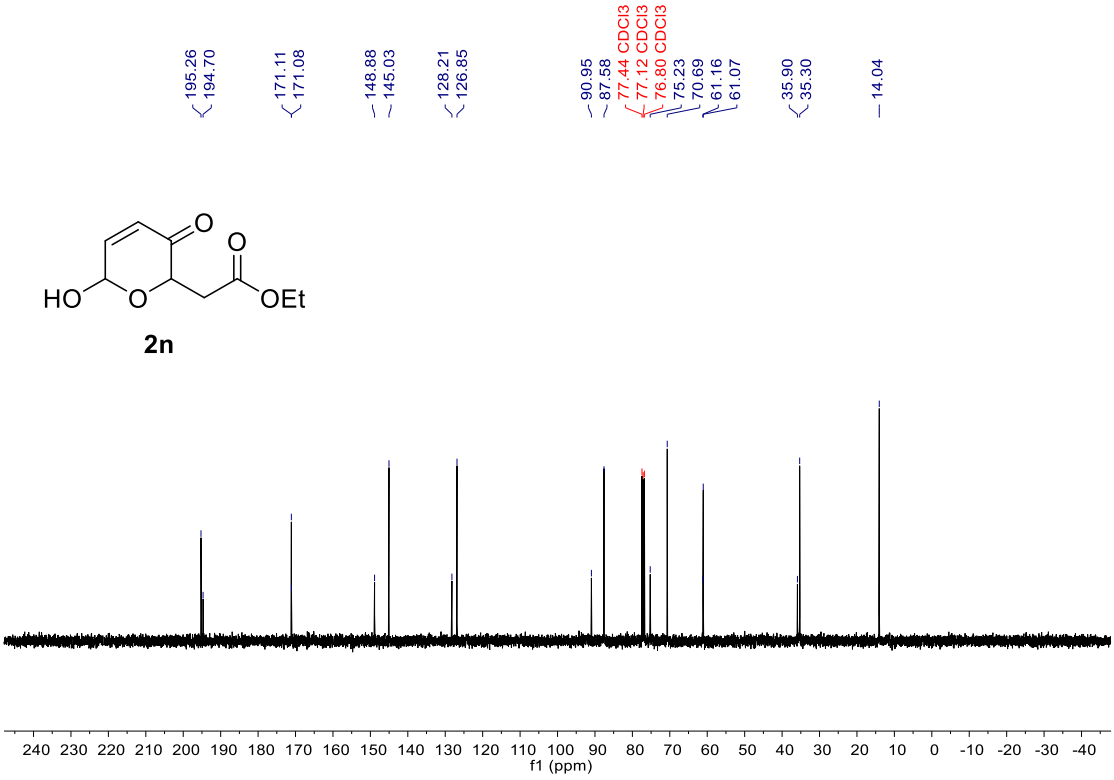
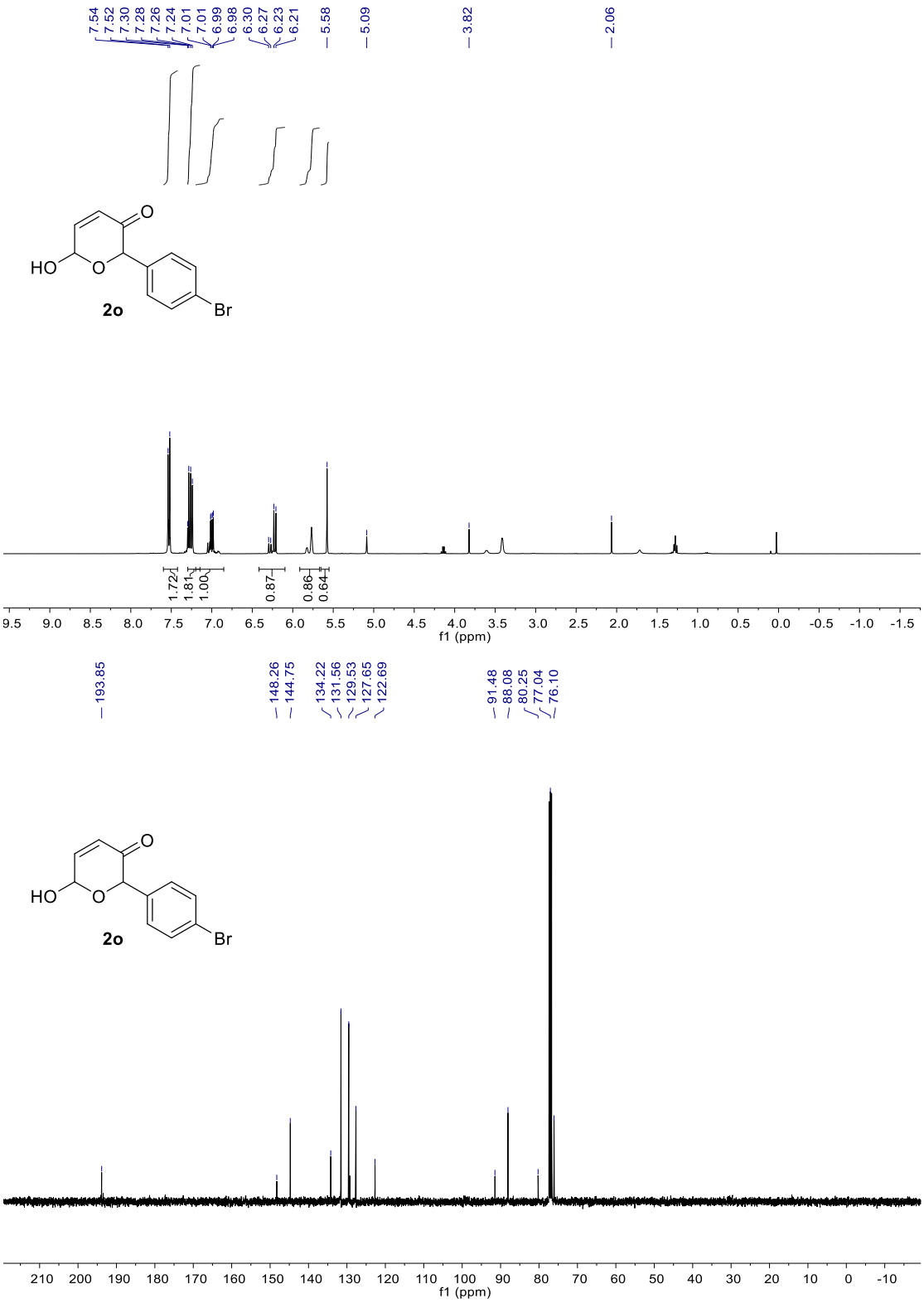
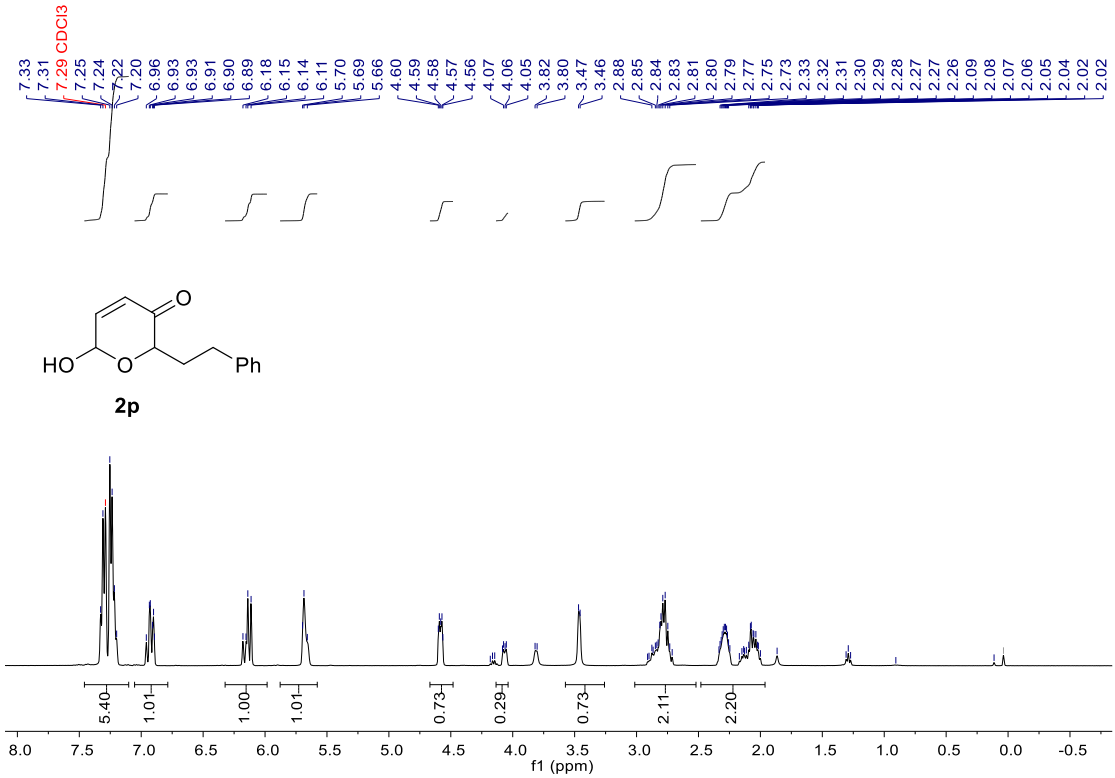
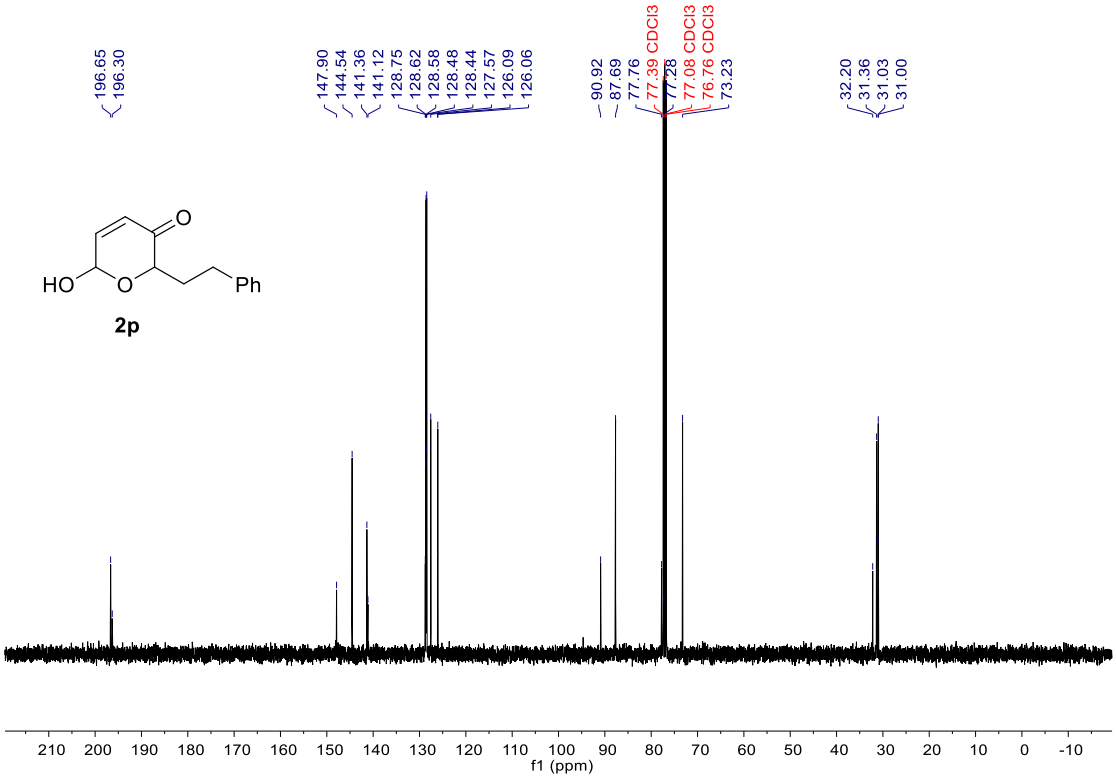
134.56
134.48
133.74
133.69
129.24
129.15
129.13
128.61
128.59
127.53
91.46
88.01
80.21
77.39 CDCl3
77.07 CDCl3
76.76 CDCl3
76.04
HO
O
O
Cl
2m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.27 CDCl3
6.94
6.92
6.90
6.90
6.88
6.87
6.14
6.12
6.10
6.07
5.67
5.57
5.56
4.97
4.96
4.95
4.94
4.75
4.54
4.53
4.52
4.51
4.14
4.13
4.12
4.11
4.09
4.07
2.98
2.97
2.95
2.94
2.93
2.91
2.90
2.76
2.74
2.73
2.72
2.71
2.70
2.69
2.67
2.45
1.55
1.23
1.22
1.21
1.21
1.20
1.19
0.81
0.80
0.02
-0.05
HO
O
O
O
OEt
2n
4.83
4.72
4.87
4.70
1.01
9.93
10.07
15.87
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
f1 (ppm)


195.26
194.70
171.11
171.08
148.88
145.03
128.21
126.85
90.95
87.58
77.44 CDCl3
77.12 CDCl3
76.80 CDCl3
75.23
70.69
61.16
61.07
35.90
35.30
14.04
HO
O
O
O
OEt
2n
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)


7.54
7.52
7.30
7.28
7.26
7.24
7.01
7.01
6.99
6.98
6.30
6.27
6.23
6.21
5.58
5.09
3.82
2.06
HO
O
O
Br
2o
1.72
1.81
1.00
0.87
0.86
0.64
f1 (ppm)
193.85
148.26
144.75
134.22
131.56
129.53
127.65
122.69
91.48
88.08
80.25
77.04
76.10
HO
O
O
Br
2o
f1 (ppm)


7.33
7.31
7.29 CDCl3
7.25
7.24
7.22
7.20
6.96
6.93
6.93
6.91
6.90
6.89
6.18
6.15
6.14
6.11
5.70
5.69
5.66
4.60
4.59
4.58
4.57
4.56
4.07
4.06
4.05
3.82
3.80
3.47
3.46
2.88
2.85
2.84
2.83
2.81
2.80
2.79
2.77
2.75
2.73
2.33
2.32
2.31
2.30
2.29
2.28
2.27
2.27
2.26
2.09
2.08
2.07
2.06
2.05
2.04
2.02
2.02
O
HO
O
Ph
2p
5.40
1.01
1.00
1.01
0.73
0.29
0.73
2.11
2.20
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
f1 (ppm)


196.65
196.30
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144.54
141.36
141.12
128.75
128.62
128.58
128.48
128.44
127.57
126.09
126.06
90.92
87.69
77.76
77.39 CDCl3
77.26
77.08 CDCl3
76.76 CDCl3
73.23
32.20
31.36
31.03
31.00
O
HO
O
Ph
2p
210
200
190
180
170
160
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140
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120
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80
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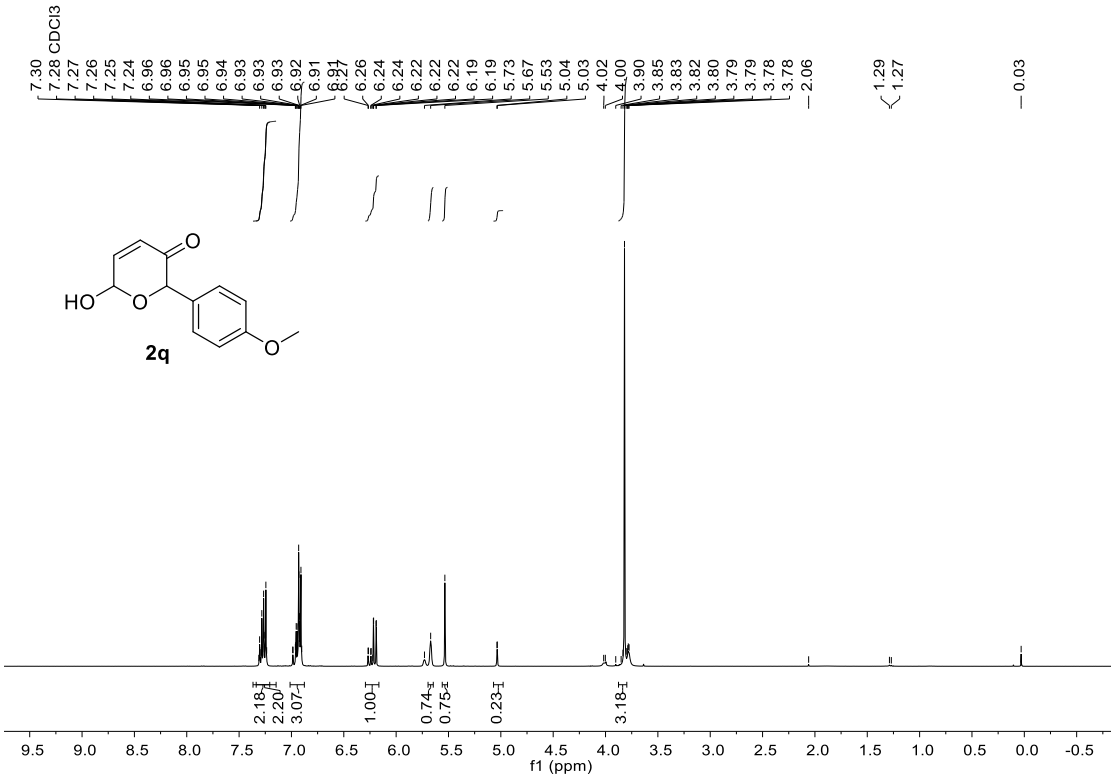
50
40
30
20
10
0
-10
f1 (ppm)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7.28 CDCl3
7.27
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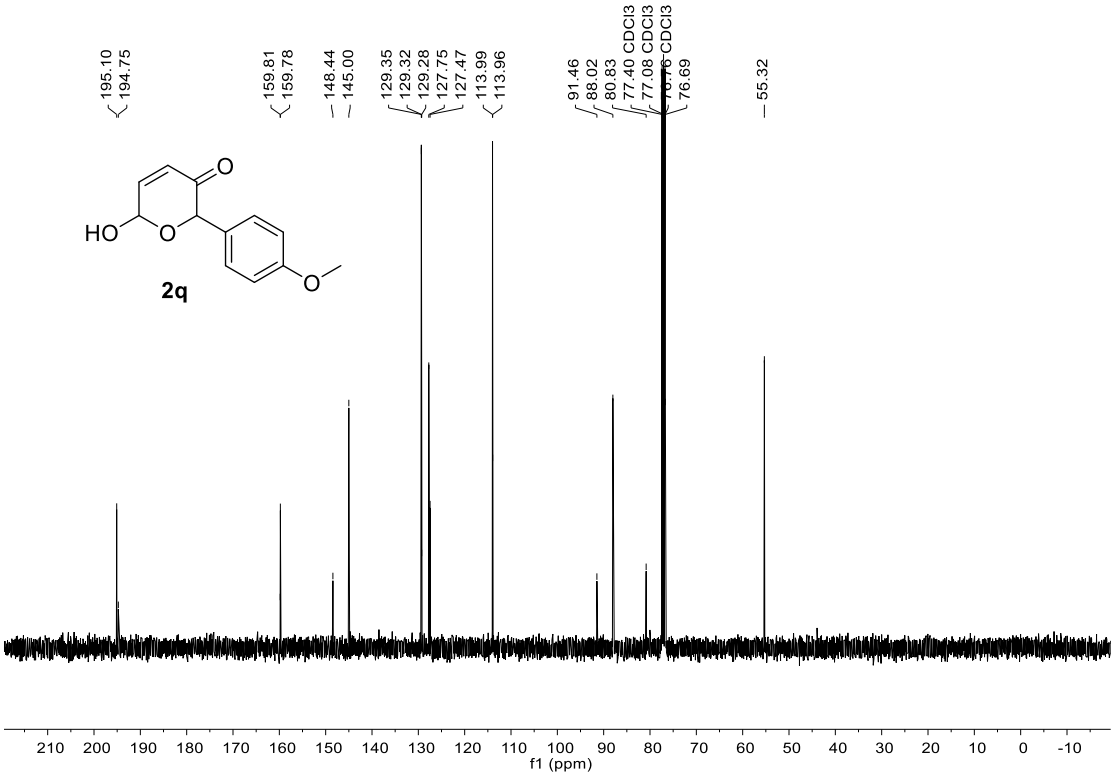
6.96
6.96
6.95
6.95
6.94
6.93
6.93
6.93
6.92
6.91
6.91
6.27
6.26
6.24
6.24
6.22
6.22
6.22
6.19
6.19
5.73
5.67
5.53
5.04
5.03
4.02
4.00
3.90
3.85
3.83
3.82
3.80
3.79
3.79
3.78
3.78
2.06
1.29
1.27
0.03
HO
O
O
O
2q
2.18
2.20
3.07
1.00
0.74
0.75
0.23
3.18
f1 (ppm)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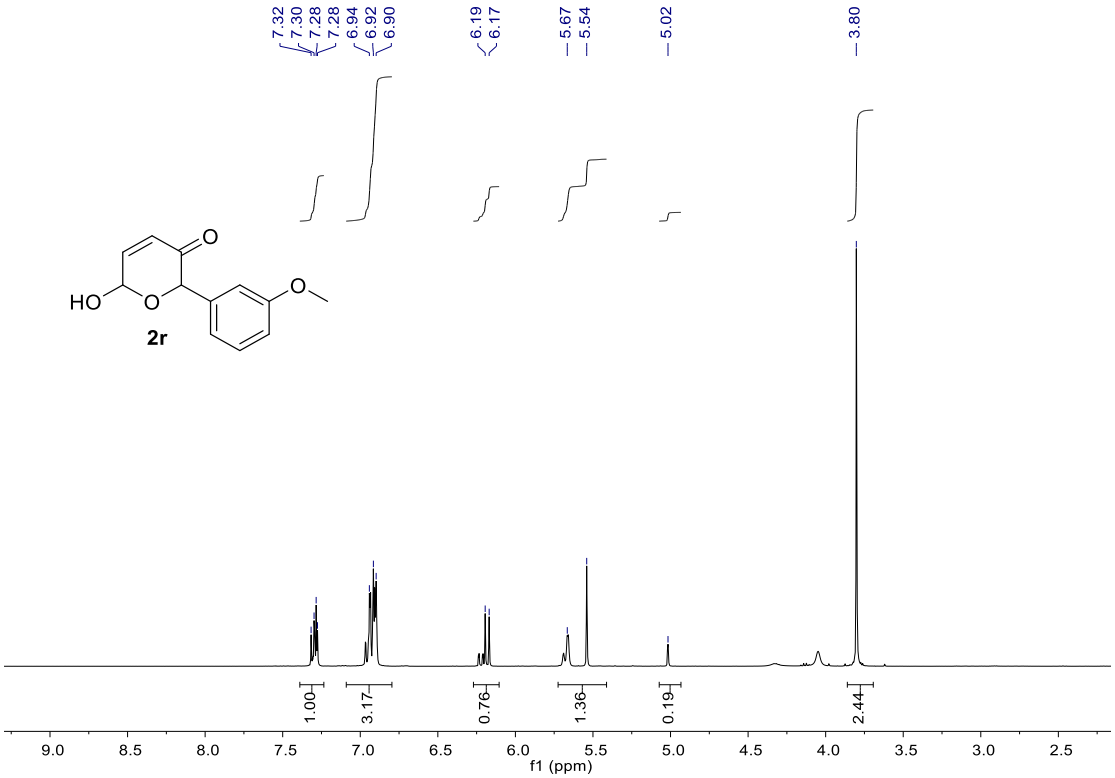
194.75
159.81
159.78
148.44
145.00
129.35
129.32
129.28
127.75
127.47
113.99
113.96
91.46
88.02
80.83
77.40 CDCl3
77.08 CDCl3
76.76 CDCl3
76.69
55.32
HO
O
O
O
2q
f1 (ppm)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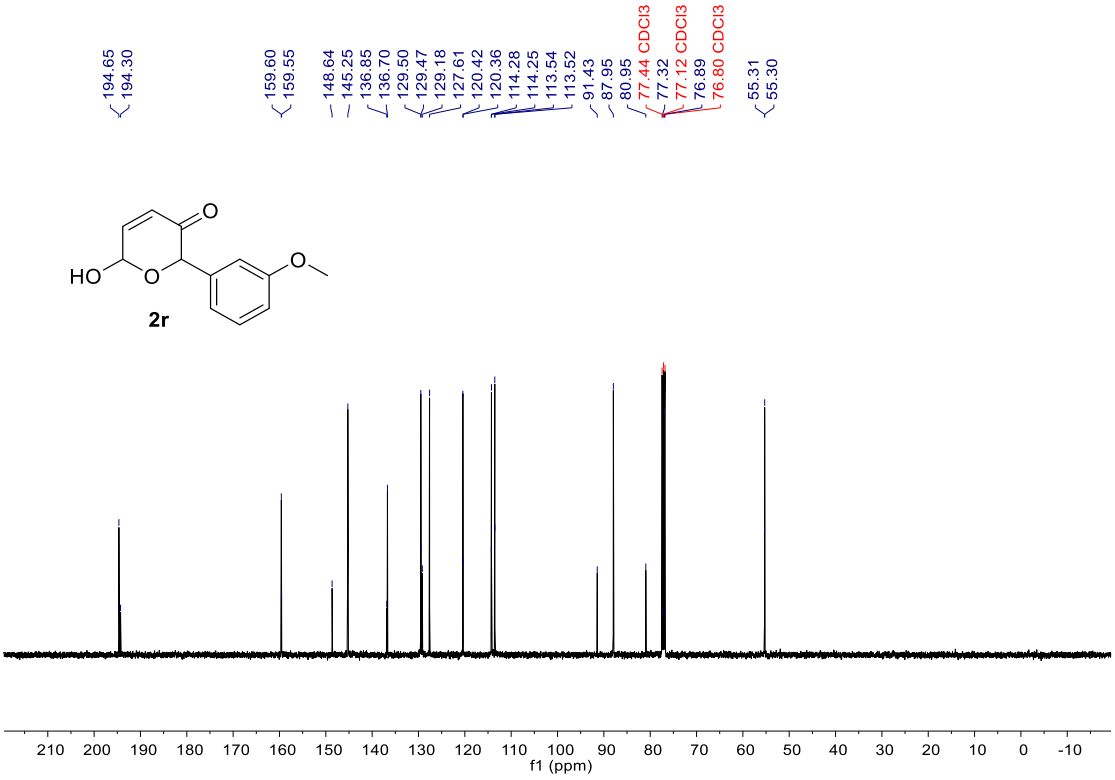
7.30
7.28
7.28
6.94
6.92
6.90
6.19
6.17
5.67
5.54
5.02
3.80
HO
O
O
O
2r
1.00
3.17
0.76
1.36
0.19
2.44
f1 (ppm)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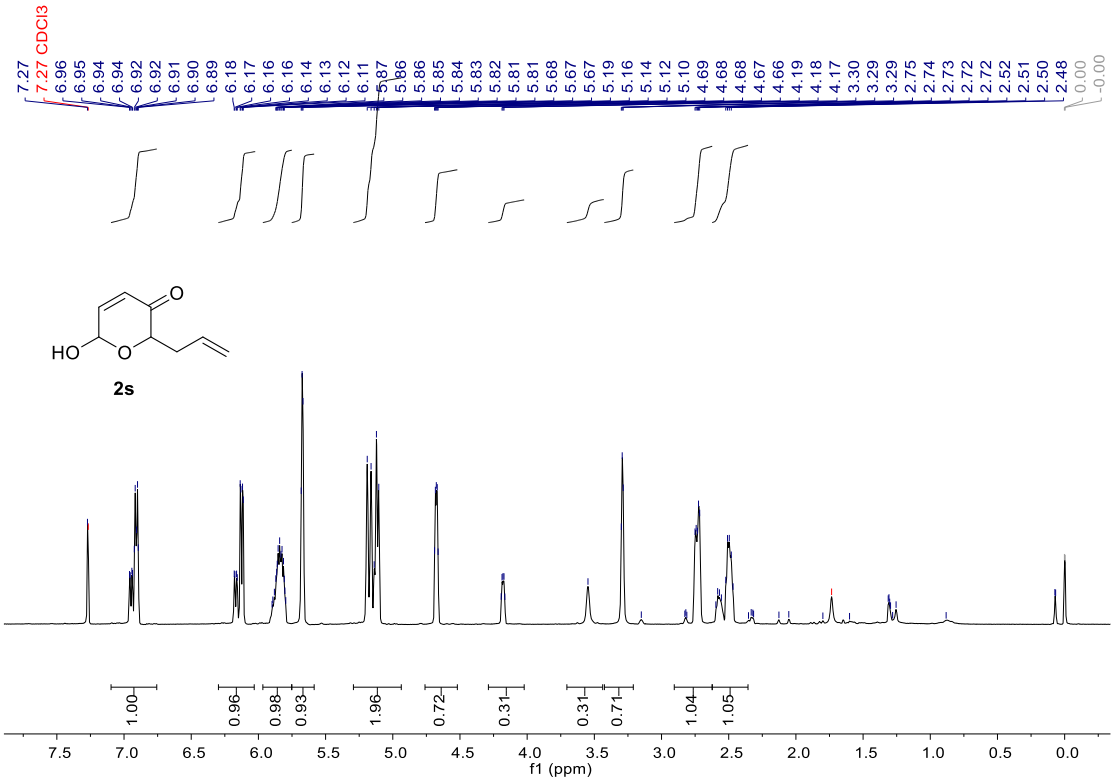
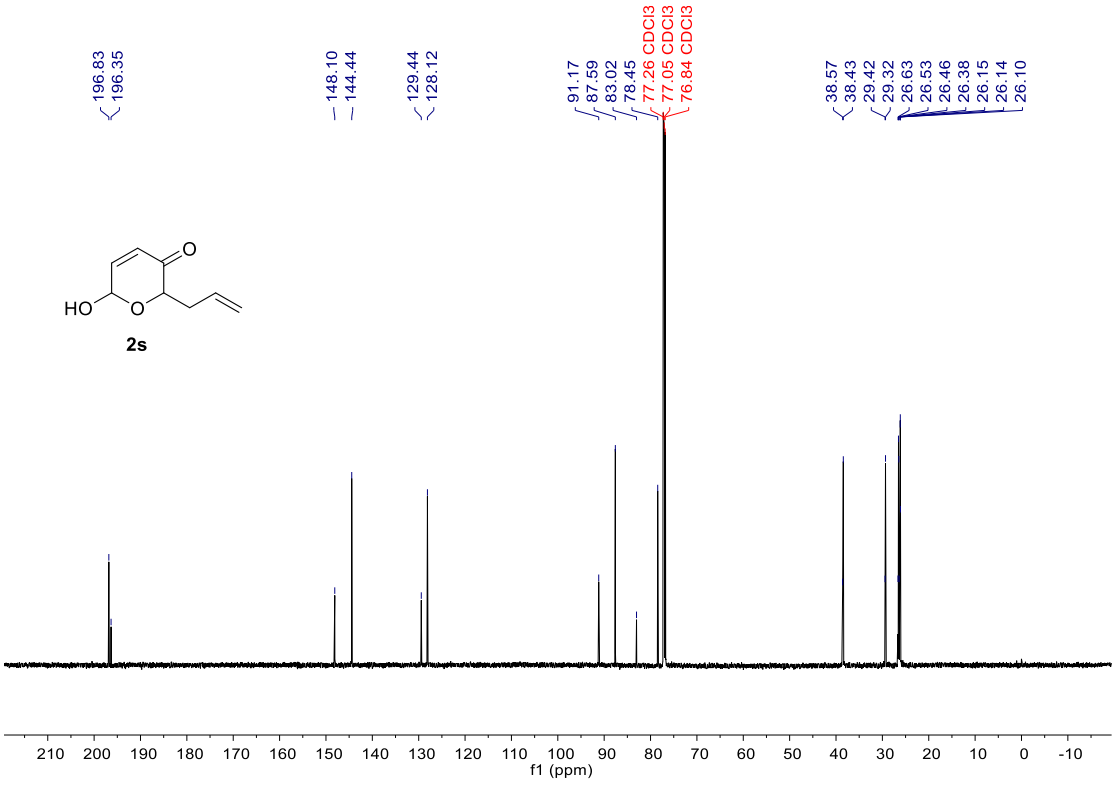
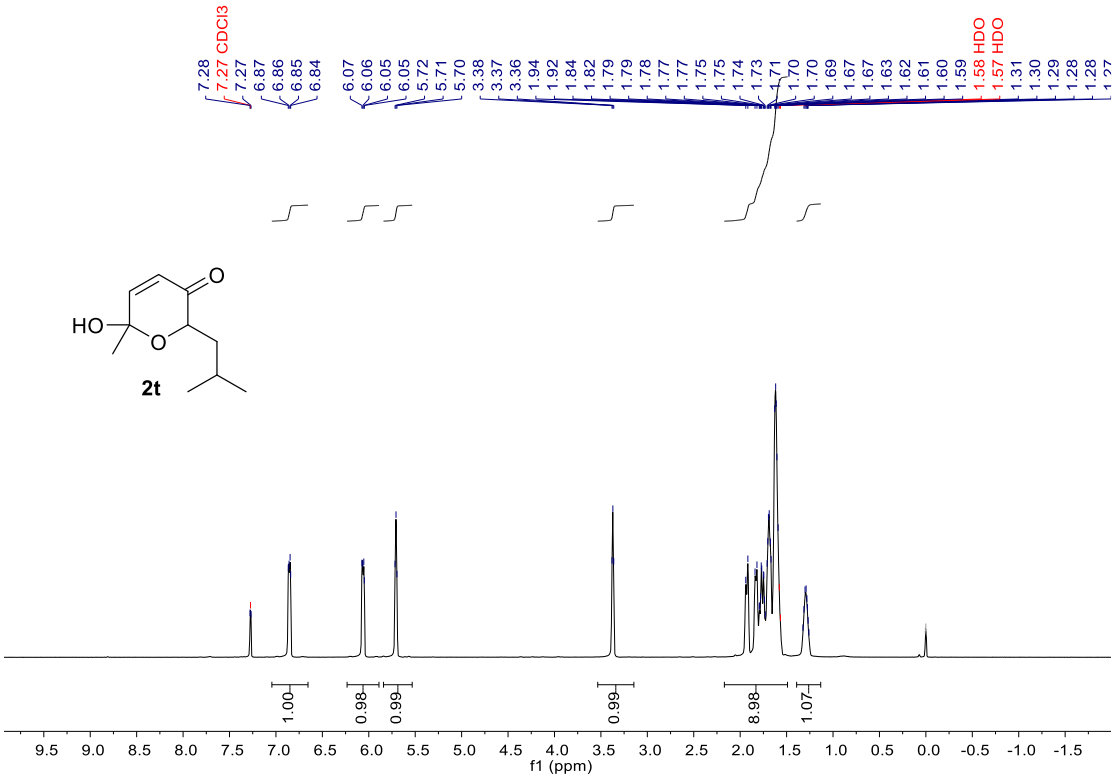
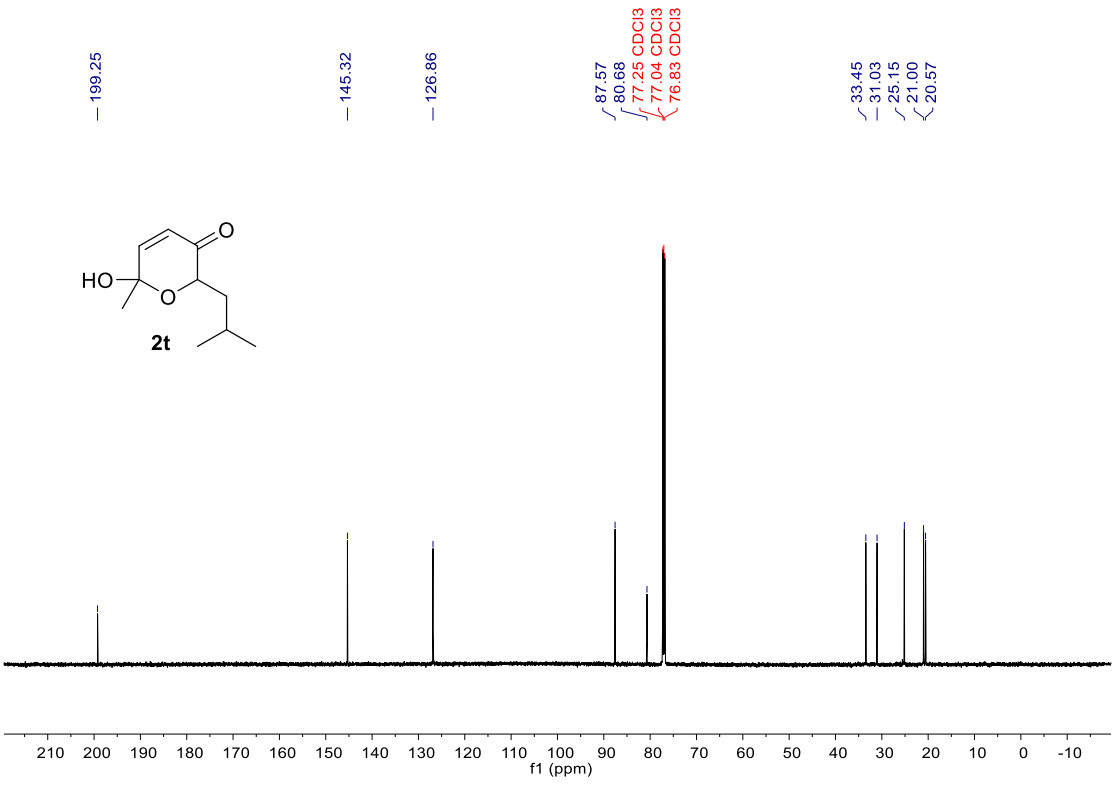
194.30
159.60
159.55
148.64
145.25
136.85
136.70
129.50
129.47
129.18
127.61
120.42
120.36
114.28
114.25
113.54
113.52
91.43
87.95
80.95
77.44 CDCl3
77.32
77.12 CDCl3
76.89
76.80 CDCl3
55.31
55.30
HO
O
O
O
2r
f1 (ppm)


7.27
7.27 CDCl3
6.96
6.95
6.94
6.94
6.92
6.92
6.91
6.90
6.89
6.18
6.17
6.16
6.16
6.14
6.13
6.12
6.11
5.87
5.86
5.86
5.85
5.84
5.83
5.82
5.81
5.81
5.68
5.67
5.67
5.19
5.16
5.14
5.12
5.10
4.69
4.68
4.68
4.67
4.66
4.19
4.18
4.17
3.30
3.29
3.29
2.75
2.74
2.73
2.72
2.72
2.52
2.51
2.50
2.48
0.00
-0.00
HO
O
O
2s
1.00
0.96
0.98
0.93
1.96
0.72
0.31
0.31
0.71
1.04
1.05
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
f1 (ppm)


196.83
196.35
148.10
144.44
129.44
128.12
91.17
87.59
83.02
78.45
77.26 CDCl3
77.05 CDCl3
76.84 CDCl3
38.57
38.43
29.42
29.32
26.63
26.53
26.46
26.38
26.15
26.14
26.10
HO
O
O
2s
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.28
7.27 CDCl3
7.27
6.87
6.86
6.85
6.84
6.07
6.06
6.05
6.05
5.72
5.71
5.70
3.38
3.37
3.36
1.94
1.92
1.84
1.82
1.79
1.79
1.78
1.77
1.77
1.75
1.75
1.74
1.73
1.71
1.70
1.70
1.69
1.67
1.67
1.63
1.62
1.61
1.60
1.59
1.58 HDO
1.57 HDO
1.31
1.30
1.29
1.28
1.28
HO
O
O
2t
1.00
0.98
0.99
0.99
8.98
1.07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)


199.25
145.32
126.86
87.57
80.68
77.25 CDCl3
77.04 CDCl3
76.83 CDCl3
33.45
31.03
25.15
21.00
20.57
HO
O
O
2t
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)