Supporting Information

CoH-Catalyzed Radical Hydroalkylation of Alkenes with 1,3 - Dicarbonyls

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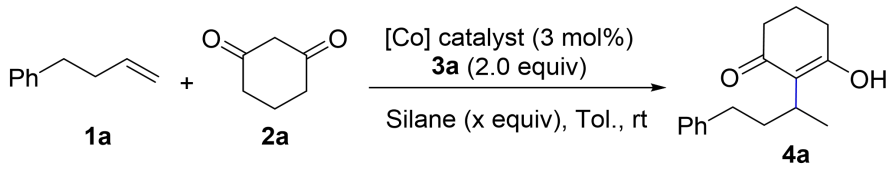
I. General Information

All reactions were carried out under an argon atmosphere with dry solvents using anhydrous conditions unless otherwise stated. Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. Reaction solvents were distilled over sodium or CaH_2 and stored under nitrogen atmosphere. All reactions were monitored by thin layer chromatography (TLC) using Macherey - Nagel 0.20 mm silica gel 60 plates. Flash column chromatography was performed on silica gel 60 (particle size 300 - 400 mesh ASTM, purchased from Taizhou, China). Compounds were visualized by irradiation with UV light, or stained with iodine/silica gel, or potassium permanganate. ^{1}H, ^{2}H, ^{13}C and ^{19}F NMR spectra were recorded at 25 ºC on a Varian 500 or on a Bruker 600 MHz spectrometer. ^{1}H NMR chemical shifts were referenced to $CDCl_3$ signal (7.26 ppm). ^{13}C NMR chemical shifts were referenced to the solvent resonance (77.00 ppm, $CDCl_3$). The following abbreviations (or combinations) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, m = multiplet, br = broad, q = quadruplet, h = heptlet. High-resolution mass spectra HRMS (ESI-TOF) were recorded on Brucker microtof. Enantiomeric excesses (ee) were determined by Agilent 1260 Series HPLC. Co(salen) catalyst **[Co]-1~[Co]-5**,[1] **Co(III)-OAc**,[2] **1b-1i**,[3] **1v-1x**,[4] **2k-2n**,[5] **2o-2p**,[6] and **2q'**[7] were prepared according to the previously reported literatures.

II. Optimization of Reaction Conditions

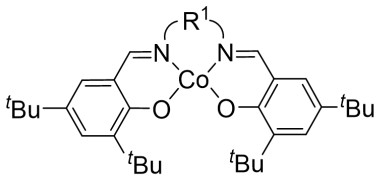
Table S1. The screening of silanes and catalysts for aliphatic alkene.[a]



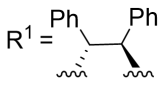
1a 2a 4a

Entry	Catalyst	Silane(x equiv)	Yield[b]
1	**[Co]-1**	TMDSO (2.0)	81%
2	**[Co]-2**	TMDSO (2.0)	50%
3	**[Co]-3**	TMDSO (2.0)	52%
4	**[Co]-4**	TMDSO (2.0)	29%
5	**[Co]-3**	$PhSiH_3$ (2.0)	37%
6	**[Co]-3**	$PhMe_2SiH$ (2.0)	34%
7	**[Co]-1**	TMDSO (4.0)	99%

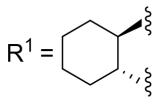
[a]Reaction conditions: **1a** (0.1 mmol), **2a** (1.5 equiv), **[Co]** catalyst (3 mol%), silane (x equiv), **3a** (2.0 equiv), toluene (0.1 M), rt, 3 h. [b]Yield determined by ^1H NMR spectroscopy using CH_2Br_2 as an internal standard.



$R^1 = CH_2CH_2$, **[Co]-1**

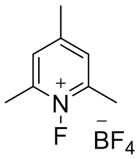


, **[Co]-2**



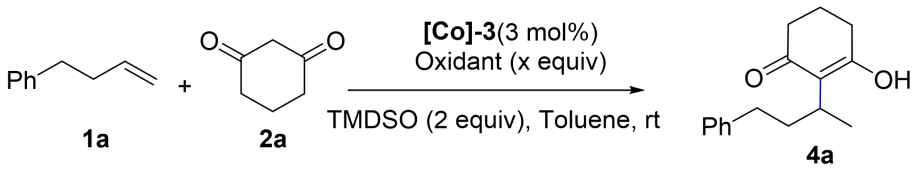
, **[Co]-3**

$R^1 = CMe_2CMe_2$, **[Co]-4**



3a

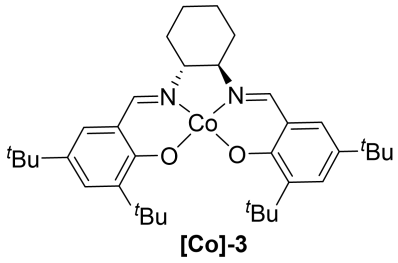
Table S2. The screening of oxidants for aliphatic alkene.[a]



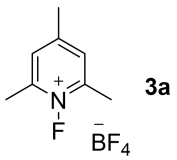
1a 2a 4a

Entry	Oxidant (x equiv)	Yield[b]
1	**3a** (1.2)	33%
2	PhIO (1.2)	＜5%
3	**3b** (1.2)	30%
4	NFSI (1.2)	＜5%
5	Selectfluor (1.2)	＜5%
6	^{t}BuOOH (1.2)	＜5%
7	**3a** (2.0)	52%
8	**3a** (0.5)	21%

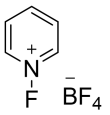
[a]Reaction conditions: **1a** (0.1 mmol), **2a** (1.5 equiv), **[Co]-3** (3 mol%), TMDSO (2.0 equiv), oxidant (x equiv), toluene (0.1 M), rt, 3 h. [b]Yield determined by ^{1}H NMR spectroscopy using CH_2Br_2 as an internal standard. NFSI = *N*-Fluorobenzenesulfonimide; Selectfluor = 1-Chloromethyl-4-fluoro-1,4-diazoniabicyclo[2.2.2]octane-bis(tetrafluorobor ate).



[Co]-3

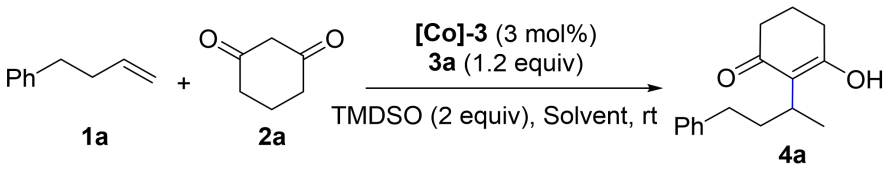


3a



3b

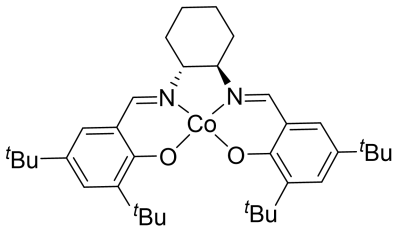
Table S3. The screening of solvents for aliphatic alkenes.[a]



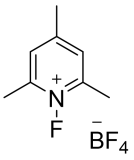
1a 2a 4a

Entry	Solvent	Yield[b]
1	Toluene	33%
2	MeCN	9%
3	DCM	14%
4	THF	26%
5	CF_3Ph	34%
6	THF/MeCN (5:1)	13%

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (1.5 equiv), **[Co]-3** (3 mol%), TMDSO (2.0 equiv), **3a** (1.2 equiv), solvent (0.1 M), rt, 3 h. [b]Yield determined by ^{1}H NMR spectroscopy using CH_2Br_2 as an internal standard. TMDSO = 1,1,3,3,-tetramethyldisiloxane.

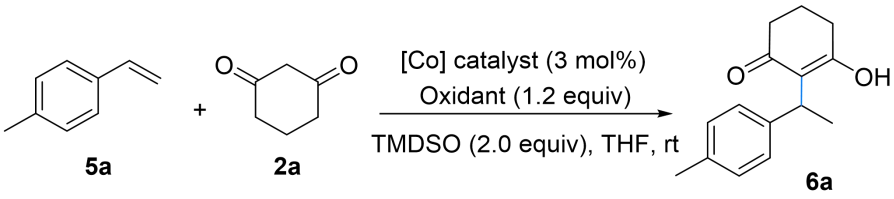


[Co]-3



3a

Table S4. Optimization of reaction conditions for activated alkenes.[a]



Entry	Catalyst	Oxidant	Yield[b]
1	**[Co]-2**	**3a**	99%
2	**[Co]-3**	**3a**	56%
3	**[Co]-4**	**3a**	66%
4	**[Co]-2**	^{t}BuOOH	trace
5	**[Co]-2**	^{t}BuOO^{t}Bu	0%
6	**[Co]-2**	$PhI(OAc)_2$	0%
7	**[Co]-2**	$K_2S_2O_8$	0%
8	**[Co]-2**	selectfluor	0%
9	**[Co]-2**	NFSI	5%

[a]Reaction conditions: **5a** (0.1 mmol), **2a** (1.5 equiv), [Co] catalyst (3 mol%), TMDSO (2.0 equiv), oxidant (1.2 equiv), THF (0.1 M), rt, 3h. [b]Yield determined by ^{1}H NMR spectroscopy using CH_2Br_2 as an internal standard.

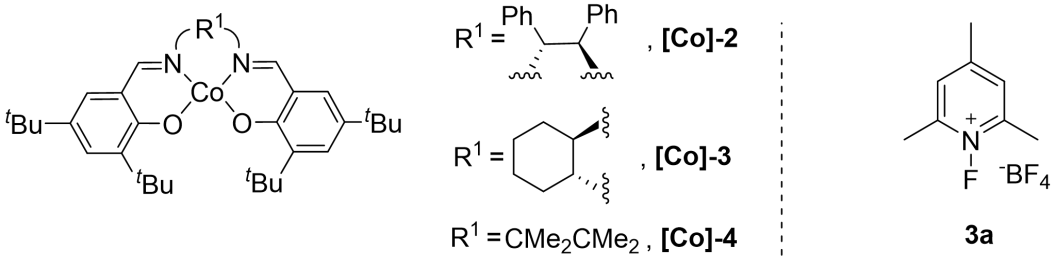
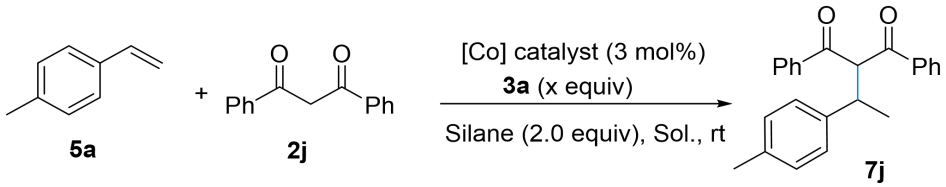
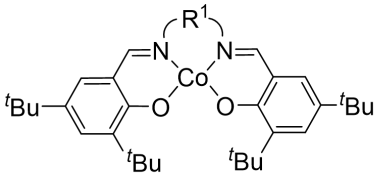


Table S5. Optimization of reaction conditions for acyclic 1,3-diketone.[a]

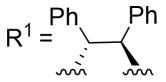


Entry	Catalyst	Silane	Solvent	Yield[b]
1	**[Co]-1**	TMDSO	MeCN	33%
2	**[Co]-2**	TMDSO	MeCN	55%
3	**[Co]-3**	TMDSO	MeCN	36%
4	**[Co]-4**	TMDSO	MeCN	26%
5	**[Co]-3**	$PhSiH_3$	MeCN	15%
6	**[Co]-3**	Et_3SiH	MeCN	39%
7	**[Co]-2**	TMDSO	DCM	23%
8	**[Co]-2**	TMDSO	THF	8%
9[c]	**[Co]-2**	TMDSO	MeCN	66%

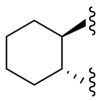
[a]Reaction conditions: **5a** (0.1 mmol), **2j** (1.5 equiv), [Co] catalyst (3 mol%), silane (2.0 equiv), **3a** (1.2 equiv), solvent (0.1 M), rt. [b]Yield determined by ^1H NMR spectroscopy using CH_2Br_2 as an internal standard. [c]**3a** (2.0 equiv).



$R^1 = CH_2CH_2$, **[Co]-1**

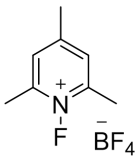


, **[Co]-2**



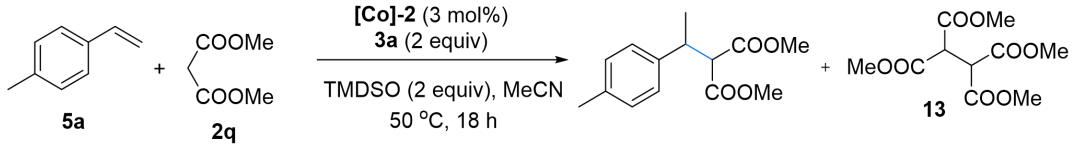
, **[Co]-3**

$R^1 = CMe_2CMe_2$, **[Co]-4**



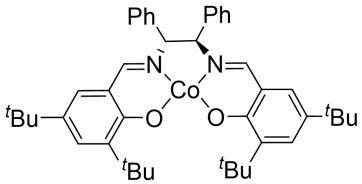
3a

Table S6. Optimization of reaction conditions for malonates.[a]

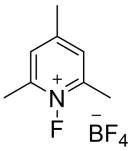


Entry	Variation from above conditions	5a[d]	13[d]	Yield[d]
1[b]	using NaH as additive	86%	33%	nr.
2[c]	using LDA as additive	100%	52%	nr.
3[c]	using LHMDS as additive	98%	46%	nr.
4	using **2q'** instead of **2q**	94%	31%	nr.

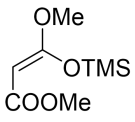
[a]Reaction conditions: **5a** (0.1 mmol), **2q** (2.0 equiv), **[Co]-2** (3 mol%), TMDSO (2.0 equiv), **3a** (2.0 equiv), MeCN (0.1 M), 50 °C, 18 h. ***Note：*** *the metal enolates derived from malonates were pre-synthesized in a seperate dry Schlenk tube.* [b]The malonate **2q** was preactivation with NaH (1.1 equiv) in THF at 0 °C under N_2 for 0.5 h. [c]The malonate **2q** was preactivation with additive (1.1 equiv) in THF at -78 °C under N_2 for 0.5 h. [d]Yield determined by ^{1}H NMR spectroscopy using CH_2Br_2 as an internal standard. LDA = Lithium diisopropylamide; LHMDS = Lithium bis(trimethylsilyl)amide.



[Co]-2



3a

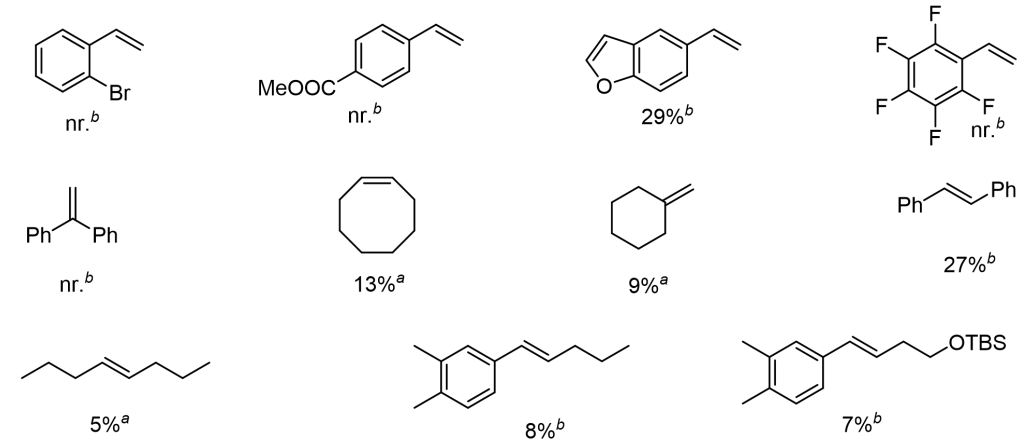


2q'

For the dimerization by-product **13** of malonates, we speculated that it may be resulted from the exchange between enol metal intermediate with cobalt(III) species in the reaction mixture to produce the enol cobalt(III) complex, which then isomerized into carbon-Co(III), and then split to produce carbon free radicals.

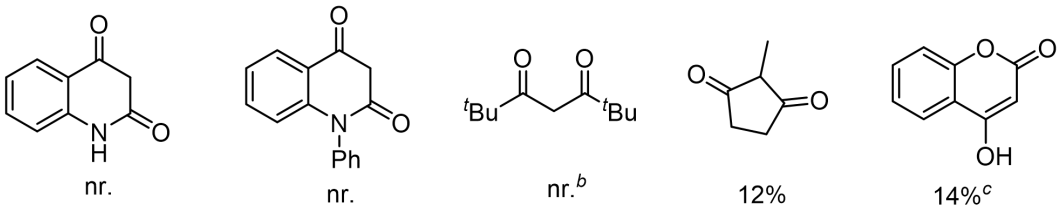
Table S7. Some inferior results during the scope of compounds.

a. Alkenes



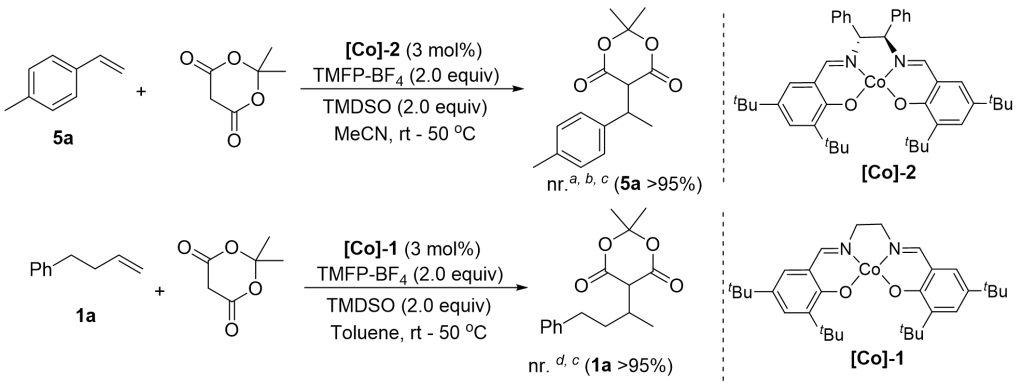
[a]alkenes (0.1 mmol), **2a** (1.5 equiv), **[Co]-2** (3 mol%), TMDSO (2.0 equiv), **3a** (2.0 equiv), toluene (0.1 M), rt. [b]Using 1.2 equivalent of **3a** in THF.

b. 1,3-Dicarbonyls[a]



[a]alkenes (0.1 mmol), 1,3-dicarbonyls (2.0 equiv), **[Co]-2** (3 mol%), TMDSO (2.0 equiv), **3a** (2.0 equiv), MeCN (0.1 M), rt. [b]using mixed solvent (MeCN: 1,3-dicarbonyls = 5:1) (0.1 M). [c]Using 50 °C instead of rt.

c. The attempts using Meldrum,s acid



[a]Reaction conditions: **5a** (0.1 mmol), Meldrum's acid (1.5 equiv), **[Co]-2** (3 mol%), TMDSO (2.0 equiv), TMFP-BF_4 (2.0 equiv), MeCN (0.1 M), rt, 24 h. [b]THF instead of MeCN. [c]50 °C instead of rt. [d]**1a** (0.1 mmol), Meldrum's acid (1.5 equiv), **[Co]-1** (3 mol%), TMDSO (4.0 equiv), TMFP-BF_4 (2.0 equiv), toluene (0.1 M) at room temperature for 24 h.

III. General Procedures of CoH-catalyzed Reaction

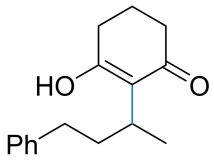
Method A : To a dry Schlenk tube containing a magnetic stir bar were added **[Co]-1** (0.006 mmol, 3 mol%), TMFP-BF_4 (0.40 mmol, 2.0 equiv), and dry toluene (2 mL) in sequence. After the reaction mixture was stirred for 5 min at room temperature, olefin (0.20 mmol, 1.0 equiv) and 1,3-diketone (0.30 mmol. 1.5 equiv) were added. Subsequently, TMDSO (0.80 mmol, 4.0 equiv) was added dropwise to the reaction. After stirring at room temperature for 3 hours, until the reaction was complete as indicated by TLC. The reaction mixture was then quenched with H_2O, extracted with DCM (3×10ml), and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product.

Method B : To a dry Schlenk tube containing a magnetic stir bar were added **[Co]-2** (0.006 mmol, 3 mol%), TMFP-BF_4 (0.24 mmol, 1.2 equiv), and dry THF (2 mL) in sequence. After the reaction mixture was stirred for 5 min at room temperature, olefin (0.20 mmol, 1.0 equiv) and 1,3-diketone (0.30 mmol. 1.5 equiv) were added. Subsequently, TMDSO (0.40 mmol, 2.0 equiv) was added dropwise to the reaction mixture. After stirring at room temperature for 3 hours, until the reaction was complete as indicated by TLC. The reaction mixture was then quenched with H_2O, extracted with DCM (3×10ml), and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product.

Method C : To a dry Schlenk tube containing a magnetic stir bar were added **[Co]-2** (0.006 mmol, 3 mol%), TMFP-BF_4 (0.40 mmol, 2.0 equiv), and dry MeCN (2 mL) in sequence. After the reaction mixture was stirred for 5 min at room temperature, olefin (0.20 mmol, 1.0 equiv) and 1,3-diketone (0.30 mmol. 1.5 equiv) were added. Subsequently, TMDSO (0.40 mmol, 2.0 equiv) was added dropwise to the reaction mixture. After stirring at room temperature for 3 hours, until the reaction was complete as indicated by TLC. The reaction mixture was then quenched with H_2O, extracted with DCM (3×10ml), and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product.

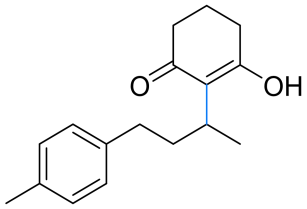
Method D : To a dry Schlenk tube containing a magnetic stir bar were added **[Co]-2** (0.006 mmol, 3 mol%), TMFP-BF_4 (0.40 mmol, 2.0 equiv), and dry MeCN (2 mL) in sequence. After the reaction mixture was stirred for 5 min at room temperature, olefin (0.20 mmol, 1.0 equiv) and TMS-enol ethers (0.40 mmol. 2.0 equiv) were added. Subsequently, TMDSO (0.40 mmol, 2.0 equiv) was added dropwise to the reaction mixture. After stirring at 50 ^oC for 18 hours, until the reaction was complete as indicated by TLC. The reaction mixture was then quenched with H_2O, extracted with DCM (3×10ml), and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product.

IV. Analytical Data of Products



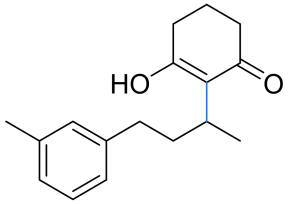
3-Hydroxy-2-(4-phenylbutan-2-yl)cyclohex-2-en-1-one (4a):

Following **Method A**, **4a** was obtained as colorless oil (48.4 mg, 99% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.28 (t, *J* = 7.2 Hz, 2H), 7.20 (t, *J* = 7.2 Hz, 1H), 7.16 (d, *J* = 7.2 Hz, 2H), 5.31 (s, 1H), 4.32 – 4.25 (m, 1H), 2.76 – 2.61 (m, 2H), 2.41 – 2.32 (m, 4H), 2.07 – 2.00 (m, 1H), 2.00 – 1.94 (m, 2H), 1.86 (m, 1H), 1.91 – 1.82 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 177.0, 141.1, 128.5, 128.3, 126.1, 103.0, 73.8, 37.6, 36.7, 31.7, 29.5, 21.2, 19.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{20}NaO_2$ ($[M + Na]^+$), 267.1262, found, 267.1263.



3-Hydroxy-2-(4-(*p*-tolyl)butan-2-yl)cyclohex-2-en-1-one (4b):

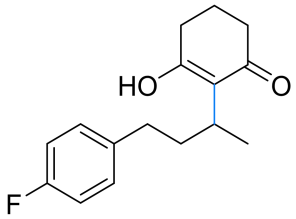
Following **Method A**, **4b** was obtained as colorless oil (30.0 mg, 58% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.09 (d, *J* = 7.8 Hz, 2H), 7.03 (d, *J* = 7.8 Hz, 2H), 5.29 (s, 1H), 4.26 (h, *J* = 6.0 Hz, 1H), 2.70 – 2.56 (m, 2H), 2.39 – 2.33 (m, 4H), 2.31 (s, 3H), 2.03 – 1.93 (m, 3H), 1.86 – 1.79 (m, 1H), 1.27 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.9, 177.0, 138.0, 135.6, 129.2, 128.2, 103.0, 73.8, 37.7, 36.7, 31.2, 29.5, 21.2, 21.0, 19.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{22}NaO_2$ ($[M + Na]^+$), 281.1644, found, 281.1646.



3-Hydroxy-2-(4-(*m*-tolyl)butan-2-yl)cyclohex-2-en-1-one (4c):

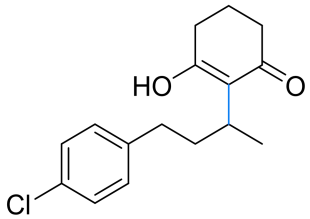
Following **Method A**, **4c** was obtained as colorless oil (27.4 mg, 53% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ

7.17 (t, *J* = 7.8 Hz, 1H), 7.01 (d, *J* = 7.8 Hz, 1H), 6.98 – 6.93 (m, 2H), 5.30 (s, 1H), 4.26 (h, *J* = 6.0 Hz, 1H), 2.70-2.54 (m, 2H), 2.40-2.32 (m, 4H), 2.32 (s, 3H), 2.05 – 1.95 (m, 3H), 1.88-1.80 (m, 1H), 1.28 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 177.0, 141.0, 138.1, 129.2, 128.4, 126.8, 125.3, 103.1, 73.8, 37.6, 36.8, 31.6, 29.5, 21.4, 21.2, 19.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{22}NaO_2$ ($[M + Na]^+$), 281.1644, found, 281.1643.



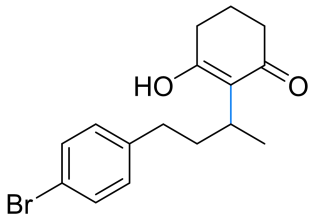
2-(4-(4-Fluorophenyl)butan-2-yl)-3-hydroxycyclohex-2-en-1-one (4d):

Following **Method A**, **4d** was obtained as colorless oil (33.0 mg, 63% yield), TLC: R_f = 0.3 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.12 – 7.08 (m, 2H), 7.00 – 6.94 (m, 2H), 5.30 (s, 1H), 4.27 (h, *J* = 6.0 Hz, 1H), 2.72 – 2.56 (m, 2H), 2.36 (dt, *J* = 10.2, 6.6 Hz, 4H), 2.04 – 1.93 (m, 3H), 1.87 – 1.79 (m, 1H), 1.29 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.9, 176.9, 161.4 (d, *J* = 244.6 Hz),136.7 (d, *J* = 3.0 Hz), 129.7 (d, *J* = 6.0 Hz), 115.3 (d, *J* = 21.1 Hz), 103.1, 73.7, 37.7, 36.7, 30.9, 29.5, 21.2, 19.1. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -117.25 – -117.32 (m, 1F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{19}FNaO_2$ ($[M + Na]^+$), 285.1382, found, 285.1384.



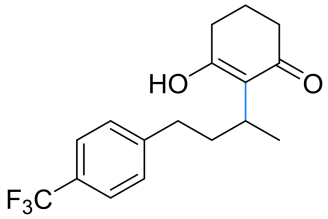
2-(4-(4-Chlorophenyl)butan-2-yl)-3-hydroxycyclohex-2-en-1-one (4e):

Following **Method A**, **4e** was obtained as colorless oil (35.6 mg, 64% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.4 Hz, 2H), 7.08 (d, *J* = 8.4 Hz, 2H), 5.30 (s, 1H), 4.30 – 4.23 (m, 1H), 2.71 – 2.57 (m, 2H), 2.35 (q, *J* = 6.6 Hz, 4H), 2.02 – 1.94 (m, 3H), 1.87 – 1.79 (m, 1H), 1.28 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.8, 139.6, 131.8, 129.7, 128.6, 103.1, 73.7, 37.5, 36.7, 31.1, 29.4, 21.2, 19.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{19}ClNaO_2$ ($[M + Na]^+$), 301.0989, found, 301.0989.



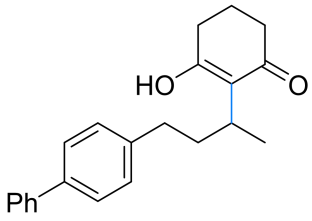
2-(4-(4-Bromophenyl)butan-2-yl)-3-hydroxycyclohex-2-en-1-one (4f):

Following **Method A**, **4f** was obtained as colorless oil (38.8 mg, 60% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 5.30 (s, 1H), 4.26 (h, *J* = 6.0 Hz, 1H), 2.71 – 2.55 (m, 2H), 2.40 – 2.30 (m, 4H), 2.02 – 1.94 (m, 3H), 1.87 – 1.79 (m, 1H), 1.28 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.8, 140.1, 131.6, 130.1, 119.8, 103.1, 73.7, 37.4, 36.7, 31.2, 29.4, 21.2, 19.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{19}BrNaO_2$ ($[M + Na]^+$), 345.0582, found, 345.0583.



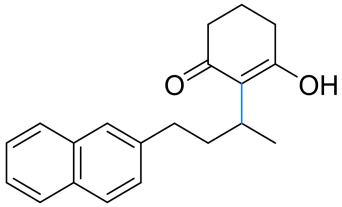
3-Hydroxy-2-(4-(4-(trifluoromethyl)phenyl)butan-2-yl)cyclohex-2-en-1-one (4g):

Following **Method A**, **4g** was obtained as colorless oil (38.0 mg, 61% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.8 Hz, 2H), 7.27 (d, *J* = 7.8 Hz, 2H), 5.31 (s, 1H), 4.29 (h, *J* = 6.0 Hz, 1H), 2.82 – 2.66 (m, 2H), 2.42 – 2.28 (m, 4H), 2.07 – 2.00 (m, 1H), 2.00 – 1.94 (m, 2H), 1.93 – 1.84 (m, 1H), 1.30 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.8, 145.4, 128.7, 128.5 (d, *J* = 31.7 Hz), 125.4 (q, *J* = 3.6 Hz), 124.3 (d, *J* = 271.8 Hz), 103.1, 73.7, 37.3, 36.7, 31.6, 29.4, 21.2, 19.1. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = -62.36. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{19}F_3NaO_2$ ($[M + Na]^+$), 335.1350, found, 335.1352.



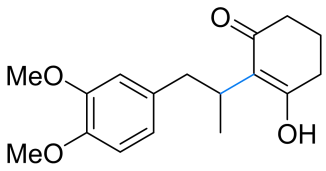
2-(4-([1,1'-Biphenyl]-4-yl)butan-2-yl)-3-hydroxycyclohex-2-en-1-one (4h):

Following **Method A**, **4h** was obtained as colorless oil (31.4 mg, 49% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.57 (d, *J* = 7.2 Hz, 2H), 7.52 (d, *J* = 7.8 Hz, 2H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.22 (d, *J* = 7.8 Hz, 2H), 5.33 (s, 1H), 4.32 (h, *J* = 6.0 Hz, 1H), 2.79 – 2.63 (m, 2H), 2.36 (dt, *J* = 11.4, 6.6 Hz, 4H), 2.10 – 2.02 (m, 1H), 1.98 (p, *J* = 6.6 Hz, 2H), 1.93 – 1.85 (m, 1H), 1.31 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 200.0, 177.0, 140.9, 140.3, 139.1, 128.8, 128.8, 128.8, 127.3, 127.1, 127.0, 103.1, 73.9, 37.6, 36.8, 31.4, 29.5, 21.3, 19.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{22}H_{24}NaO_2$ ([M + Na]$^+$), 343.1659, found, 343.1657.



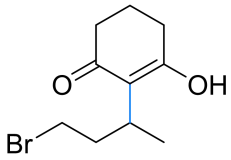
3-Hydroxy-2-(4-(naphthalen-2-yl)butan-2-yl)cyclohex-2-en-1-one (4i):

Following **Method A**, **4i** was obtained as colorless oil (34.2 mg, 58% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.78 (dt, *J* = 18.0, 8.4 Hz, 3H), 7.58 (s, 1H), 7.47 – 7.40 (m, 2H), 7.29 (dd, *J* = 8.4, 1.8 Hz, 1H), 5.32 (s, 1H), 4.32 (h, *J* = 6.0 Hz, 1H), 2.92 – 2.74 (m, 2H), 2.40 – 2.25 (m, 4H), 2.15 – 2.06 (m, 1H), 2.00 – 1.89 (m, 3H), 1.31 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.9, 138.6, 133.6, 132.1, 128.1, 127.6, 127.4, 127.1, 126.4, 126.1, 125.3, 103.1, 73.9, 37.5, 36.7, 32.0, 29.5, 21.2, 19.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{20}H_{22}NaO_2$ ([M + Na]$^+$), 317.1527, found, 317.1529.



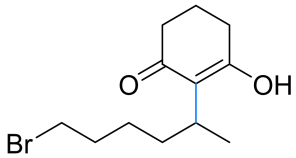
2-(1-(3,4-Dimethoxyphenyl)propan-2-yl)-3-hydroxycyclohex-2-en-1-one (4j):

Following **Method A**, **4j** was obtained as colorless oil (39.0 mg, 67% yield), TLC: R_f = 0.3 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 6.80 (d, *J* = 8.0 Hz, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.69 (s, 1H), 5.38 (s, 1H), 4.46 (h, *J* = 6.0 Hz, 1H), 3.87 (d, *J* = 3.5 Hz, 7H), 2.95 (dd, *J* = 14.0, 6.0 Hz, 1H), 2.75 (dd, *J* = 14.0, 6.0 Hz, 1H), 2.41 – 2.29 (m, 4H), 2.01 – 1.90 (m, 2H), 1.27 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.9, 176.8, 148.8, 147.8, 129.7, 121.4, 112.6, 111.2, 103.1, 75.3, 55.9, 41.6, 36.7, 29.5, 21.2, 18.8. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{22}NaO_4$ ([M + Na]$^+$), 313.1300, found, 313.1302.



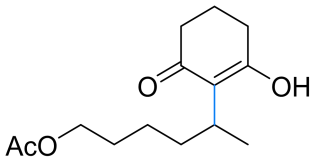
2-(4-Bromobutan-2-yl)-3-hydroxycyclohex-2-en-1-one (4k):

Following **Method A**, **4k** was obtained as colorless oil (14.8 mg, 30% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.40 (s, 1H), 4.54-4.46 (m, 1H), 3.43 (t, *J* = 6.5 Hz, 2H), 2.44 – 2.30 (m, 4H), 2.29 – 2.20 (m, 1H), 2.11 – 2.02 (m, 1H), 1.98 (p, *J* = 6.5 Hz, 2H), 1.30 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.5, 103.4, 72.3, 38.9, 36.7, 29.3, 28.7, 21.2, 18.7. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{10}H_{15}BrNaO_2$ ($[M + Na]^+$), 269.0245, found, 269.0235.



2-(6-Bromohexan-2-yl)-3-hydroxycyclohex-2-en-1-one (4l):

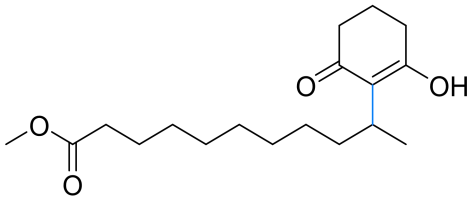
Following **Method A**, **4l** was obtained as colorless oil (41.2 mg, 75% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 5.34 (s, 1H), 4.28 (h, *J* = 6.0 Hz, 1H), 3.41 (t, *J* = 6.6 Hz, 2H), 2.38 (q, *J* = 6.0 Hz, 2H), 2.36 – 2.32 (m, 2H), 1.98 (p, *J* = 6.6 Hz, 2H), 1.87 (p, *J* = 7.2 Hz, 2H), 1.74 – 1.66 (m, 1H), 1.62 – 1.44 (m, 3H), 1.27 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 177.0, 103.0, 74.3, 36.7, 34.9, 33.4, 32.4, 29.5, 23.9, 21.2, 19.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{12}H_{19}BrNaO_2$ ($[M + Na]^+$), 297.0576, found, 297.0575.



5-(2-Hydroxy-6-oxocyclohex-1-en-1-yl)hexyl acetate (4m):

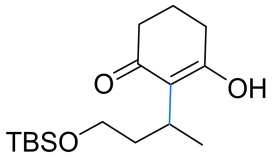
Following **Method A**, **4m** was obtained as colorless oil (34.6 mg, 68% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.37 (s, 1H), 4.30 (h, *J* = 6.0 Hz, 1H), 4.09 (t, *J* = 6.5 Hz, 2H), 2.39 (dt, *J* = 13.5, 6.0 Hz, 4H), 2.08 (s, 3H), 2.01 (p, *J* = 6.5 Hz, 2H), 1.79 – 1.70 (m, 1H), 1.66 (q, *J* = 7.0 Hz, 2H), 1.64 – 1.57 (m, 1H), 1.53 – 1.35 (m, 2H), 1.29 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 177.0, 171.1, 103.0, 74.4, 64.2, 36.7, 35.4, 29.5,

28.4, 21.9, 21.2, 21.0, 19.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{22}NaO_4$ ([M + Na]$^+$), 277.1548, found, 277.1537.



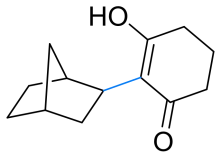
Methyl-10-(2-hydroxy-6-oxocyclohex-1-en-1-yl)undecanoate (4n):

Following **Method A**, **4n** was obtained as colorless oil (45.4 mg, 73% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 5.34 (s, 1H), 4.25 (h, *J* = 6.0 Hz, 1H), 3.67 (s, 3H), 2.37 (td, *J* = 6.0, 3.0 Hz, 2H), 2.34 (t, *J* = 6.6 Hz, 2H), 2.30 (t, *J* = 7.8 Hz, 2H), 1.98 (p, *J* = 6.6 Hz, 2H), 1.72 – 1.65 (m, 1H), 1.62 (p, *J* = 7.2 Hz, 2H), 1.56 – 1.47 (m, 1H), 1.37 – 1.27 (m, 11H), 1.25 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 200.0, 177.1, 174.3, 102.9, 74.7, 51.4, 36.7, 35.8, 34.1, 29.5, 29.4, 29.3, 29.1, 29.1, 25.3, 24.9, 21.2, 19.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{18}H_{30}NaO_4$ ([M + Na]$^+$), 333.2214, found, 333.2216.



2-(4-((*Tert*-butyldimethylsilyl)oxy)butan-2-yl)-3-hydroxycyclohex-2-en-1-one (4o):

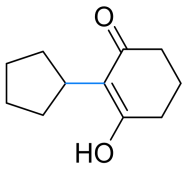
Following **Method A**, **4o** was obtained as colorless oil (25.6 mg, 43% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.36 (s, 1H), 4.46 (h, *J* = 6.0 Hz, 1H), 3.69 – 3.61 (m, 2H), 2.40 – 2.24 (m, 4H), 1.99 – 1.91 (m, 2H), 1.89 – 1.81 (m, 1H), 1.76 – 1.68 (m, 1H), 1.25 (d, *J* = 6.0 Hz, 3H), 0.85 (s, 9H), 0.00 (s, 6H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 199.8, 176.9, 103.2, 71.4, 59.0, 39.0, 36.8, 29.5, 25.9, 21.2, 19.2, 18.3, -5.5. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{30}NaO_3Si$ ([M + Na]$^+$), 321.0867, found, 321.0868.



2-((1S,4R)-Bicyclo[2.2.1]heptan-2-yl)-3-hydroxycyclohex-2-en-1-one (4p):

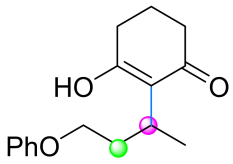
Following **Method A**, **4p** was obtained as colorless oil (18.2 mg, 44% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.31 (s, 1H), 4.03 (d, *J* = 7.0 Hz, 1H), 2.41 (d, *J* = 5.0 Hz, 1H), 2.37 – 2.29 (m, 5H),

2.00 – 1.92 (m, 2H), 1.71 (ddd, *J* = 13.5, 7.0, 2.5 Hz, 1H), 1.57 – 1.51 (m, 2H), 1.51 – 1.44 (m, 2H), 1.19 (d, *J* = 10.0 Hz, 1H), 1.14 – 1.04 (m, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.7, 103.7, 81.3, 41.0, 39.8, 36.7, 35.3, 35.3, 29.3, 28.2, 24.1, 21.3. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{13}H_{18}NaO_2$ ($[M + Na]^+$), 229.1197, found, 229.1199.



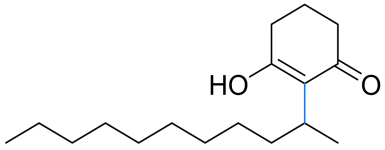
2-Cyclopentyl-3-hydroxycyclohex-2-en-1-one (4q):

Following **Method A**, **4q** was obtained as colorless oil (17.4 mg, 48% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 5.34 (s, 1H), 4.62 (tt, *J* = 6.0, 2.4 Hz, 1H), 2.39 – 2.31 (m, 4H), 1.97 (p, *J* = 6.6 Hz, 2H), 1.91 – 1.83 (m, 2H), 1.82 – 1.77 (m, 2H), 1.76 – 1.70 (m, 2H), 1.65 – 1.56 (m, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 177.1, 103.7, 80.5, 36.7, 32.7, 29.4, 24.1, 21.3. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{11}H_{16}NaO_2$ ($[M + Na]^+$), 203.1146, found, 203.1145.



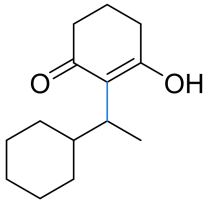
3-Hydroxy-2-(4-phenoxybutan-2-yl)cyclohex-2-en-1-one (4r):

Following **Method A**, **4r** was obtained as colorless oil (26.4 mg, 51% yield), rr = 6:1 (C2 :C3), TLC: R_f = 0.3 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.27 (t, *J* = 7.0 Hz, 2H), 6.94 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 8.0 Hz, 2H), 5.40 (s, 1H), 4.58 (h, *J* = 6.0 Hz, 1H), 4.02 (t, *J* = 5.5 Hz, 2H), 2.37 – 2.29 (m, 4H), 2.17 – 2.09 (m, 1H), 2.08 – 2.00 (m, 1H), 1.99 – 1.90 (m, 2H), 1.33 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.7, 158.7, 129.5, 120.9, 114.5, 103.3, 71.5, 63.8, 36.7, 35.7, 29.4, 23.7, 21.2, 19.2, 9.5. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{20}NaO_3$ ($[M + Na]^+$), 283.1213, found, 283.1211.



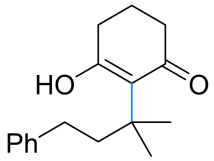
3-Hydroxy-2-(undecan-2-yl)cyclohex-2-en-1-one (4s):

Following **Method A**, **4s** was obtained as colorless oil (34.6 mg, 65% yield), TLC: R_f = 0.6 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.32 (s, 1H), 4.24 (h, *J* = 6.0 Hz, 1H), 2.43 – 2.26 (m, 4H), 1.96 (p, *J* = 6.5 Hz, 2H), 1.71 – 1.59 (m, 1H), 1.55 – 1.45 (m, 1H), 1.31 – 1.17 (m, 17H), 0.87 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.9, 177.1, 102.9, 74.7, 36.7, 35.8, 31.9, 29.5, 29.5, 29.5, 29.4, 29.3, 25.3, 22.6, 21.2, 19.1, 14.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{30}NaO_2$ ([M + Na]$^+$), 289.1950, found, 289.1952.



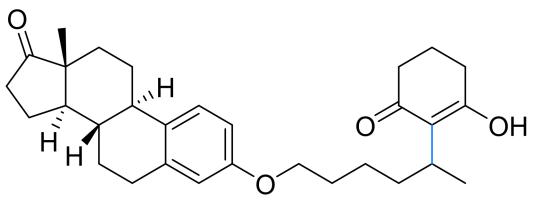
2-(1-Cyclohexylethyl)-3-hydroxycyclohex-2-en-1-one (4t):

Following **Method A**, **4t** was obtained as colorless oil (37.4 mg, 84% yield), TLC: R_f = 0.6 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 5.34 (s, 1H), 4.06 (p, *J* = 6.0 Hz, 1H), 2.40 – 2.36 (m, 2H), 2.36 – 2.33 (m, 2H), 1.97 (p, *J* = 6.6 Hz, 2H), 1.82 – 1.71 (m, 3H), 171 – 1.63 (m, 2H), 1.58 – 1.50 (m, 1H), 1.27 – 1.21 (m, 2H), 1.20 (d, *J* = 6.0 Hz, 3H), 1.15 (tt, *J* = 12.6, 3.0 Hz, 1H), 1.07 – 0.93 (m, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 200.0, 177.4, 102.8, 78.6, 42.5, 36.7, 29.5, 28.6, 28.2, 26.4, 26.0, 25.9, 21.2, 15.9. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{22}NaO_2$ ([M + Na]$^+$), 245.1408, found, 245.1408.



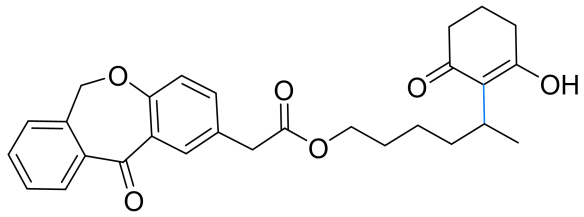
3-Hydroxy-2-(2-methyl-4-phenylbutan-2-yl)cyclohex-2-en-1-one (4u):

Following **Method A**, **4u** was obtained as colorless oil (13.4 mg, 26% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.5 Hz, 2H), 7.22 – 7.15 (m, 3H), 5.55 (s, 1H), 2.70 – 2.63 (m, 2H), 2.32 (q, *J* = 6.0 Hz, 4H), 2.08 – 2.00 (m, 2H), 1.95 (p, *J* = 6.5 Hz, 2H), 1.51 (s, 6H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 200.0, 175.3, 141.7, 128.5, 128.3, 126.0, 106.2, 82.9, 43.3, 36.5, 30.5, 30.2, 26.2, 21.3. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{22}NaO_2$ ([M + Na]$^+$), 281.1402, found, 281.1400.



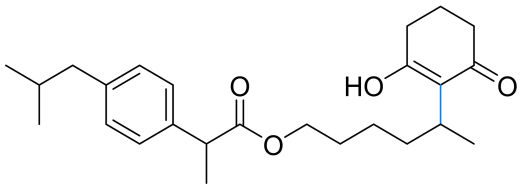
(8S,9R,13R,14R)-3-((5-(2-Hydroxy-6-oxocyclohex-1-en-1-yl)hexyl)oxy)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (4v):

Following **Method A**, **4v** was obtained as colorless oil (56.6 mg, 61% yield), dr = 1:1, TLC: R_f = 0.3 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.19 (d, *J* = 8.4 Hz, 1H), 6.70 (d, *J* = 8.4 Hz, 1H), 6.64 (s, 1H), 5.34 (s, 1H), 4.28 (p, *J* = 6.0 Hz, 1H), 3.93 (t, *J* = 6.0 Hz, 2H), 2.95 – 2.83 (m, 2H), 2.50 (dd, *J* = 19.2, 8.4 Hz, 1H), 2.42 – 2.30 (m, 5H), 2.28 – 2.21 (m, 1H), 2.18 – 2.10 (m, 1H), 2.09 – 2.02 (m, 1H), 2.02 – 1.90 (m, 4H), 1.84 – 1.70 (m, 4H), 1.66 – 1.57 (m, 3H), 1.55 – 1.45 (m, 5H), 1.46 – 1.39 (m, 1H), 1.27 (d, *J* = 6.0 Hz, 3H), 0.91 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 220.9, 199.9, 177.1, 157.0, 137.8, 132.0, 126.3, 114.5, 112.1, 103.0, 74.5, 67.5, 50.4, 48.0, 44.0, 38.4, 36.7, 35.9, 35.6, 31.6, 29.7, 29.5, 29.1, 26.6, 25.9, 22.1, 21.6, 21.2, 19.1, 13.9. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{30}H_{40}NaO_4$ ($[M + Na]^+$), 487.2614, found, 487.2616.



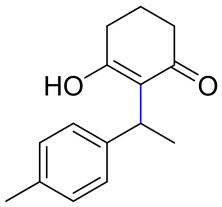
5-(2-Hydroxy-6-oxocyclohex-1-en-1-yl)hexyl-2-(11-oxo-6,11-dihydrodibenzo[b,e]oxepin-2-yl)acetate (4w):

Following **Method A**, **4w** was obtained as colorless oil (42.8 mg, 46% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.11 (d, *J* = 2.4 Hz, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.42 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.36 (d, *J* = 7.2 Hz, 1H), 7.02 (d, *J* = 8.4 Hz, 1H), 5.31 (s, 1H), 5.17 (s, 2H), 4.23 (h, *J* = 6.0 Hz, 1H), 4.09 (t, *J* = 6.6 Hz, 2H), 3.63 (s, 2H), 2.37 – 2.28 (m, 4H), 1.95 (p, *J* = 6.6 Hz, 2H), 1.71 – 1.60 (m, 3H), 1.58 – 1.49 (m, 1H), 1.45 – 1.37 (m, 1H), 1.36 – 1.30 (m, 1H), 1.22 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 190.8, 176.9, 171.4, 160.4, 140.4, 136.3, 135.5, 132.8, 132.4, 129.4, 129.2, 127.8, 125.1, 121.0, 102.9, 74.3, 73.6, 64.6, 40.2, 36.7, 35.4, 29.6, 29.4, 28.4, 21.7, 21.1, 19.0. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{28}H_{30}NaO_6$ ($[M + Na]^+$), 485.1938, found, 485.1936.



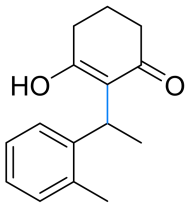
5-(2-Hydroxy-6-oxocyclohex-1-en-1-yl)hexyl-2-(4-isobutylphenyl)propanoate (4x):

Following **Method A**, **4x** was obtained as colorless oil (52.8 mg, 66% yield), dr = 1:1, TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.24 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 5.35 (s, 1H), 4.28 – 4.20 (m, 1H), 4.10 (t, *J* = 6.5 Hz, 2H), 3.72 (q, *J* = 7.0 Hz, 1H), 2.48 (d, *J* = 7.0 Hz, 2H), 2.43 – 2.33 (m, 4H), 2.01 (p, *J* = 6.5 Hz, 2H), 1.93 – 1.83 (m, 1H), 1.71 – 1.58 (m, 3H), 1.57 – 1.48 (m, 4H), 1.42 – 1.30 (m, 2H), 1.25 (dd, *J* = 6.0, 3.0 Hz, 3H), 0.93 (d, *J* = 6.5 Hz, 6H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 177.0, 174.8, 140.5, 137.8, 137.8, 129.3, 127.1, 103.0, 74.4, 74.4, 64.3, 64.3, 45.2, 45.0, 36.7, 35.3, 35.3, 30.2, 29.5, 28.4, 28.3, 22.4, 21.7, 21.2, 19.0, 18.5. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{25}H_{36}NaO_4$ ($[M + Na]^+$), 423.2322, found, 423.2332.



3-Hydroxy-2-(1-(*p*-tolyl)ethyl)cyclohex-2-en-1-one (6a):

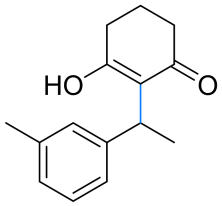
Following **Method B**, **6a** was obtained as colorless oil (42.8 mg, 93% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.18 – 7.12 (m, 4H), 5.28 (s, 1H), 5.17 (q, J = 6.6 Hz, 1H), 2.47 – 2.37 (m, 2H), 2.32 (s, 3H), 2.30 – 2.21 (m, 2H), 1.98-1.88 (m, 2H), 1.56 (d, J = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.6, 138.3, 137.7, 129.4, 125.3, 104.5, 76.6, 36.6, 29.4, 23.6, 21.1, 21.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{18}NaO_2$ ($[M + Na]^+$), 253.1199, found, 253.1199.



3-Hydroxy-2-(1-(*o*-tolyl)ethyl)cyclohex-2-en-1-one (6b):

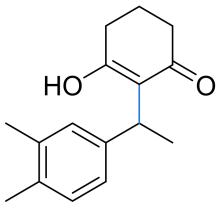
Following **Method B**, **6b** was obtained as colorless oil (42.4 mg, 92% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.28 (dd, *J* = 7.2, 1.8 Hz, 1H), 7.21 – 7.15 (m, 2H), 7.13 (dd, *J* = 7.2, 1.8 Hz, 1H), 5.37 (q, *J* = 6.6 Hz, 1H), 5.12 (s, 1H), 2.53 – 2.41 (m, 2H), 2.34 (s, 3H), 2.32 – 2.23 (m, 2H), 2.03 – 1.91 (m, 2H), 1.55 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.5, 139.4, 133.8, 130.8, 127.8, 126.7, 124.3, 104.4, 73.8, 36.7, 29.4, 22.0,

21.2, 19.0. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{18}NaO_2$ ([M + Na]$^+$), 253.1199, found, 253.1197.



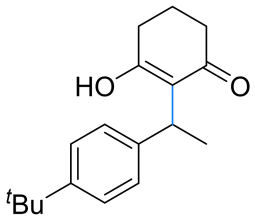
3-Hydroxy-2-(1-(*m*-tolyl)ethyl)cyclohex-2-en-1-one (6c):

Following **Method B**, **6c** was obtained as colorless oil (43.2 mg, 94% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.23 (t, *J* = 7.8 Hz, 1H), 7.11 – 7.05 (m, 3H), 5.28 (s, 1H), 5.16 (q, *J* = 6.6 Hz, 1H), 2.51 – 2.40 (m, 2H), 2.35 (s, 3H), 2.32 – 2.23 (m, 2H), 2.01 – 1.90 (m, 2H), 1.56 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.6, 141.3, 138.5, 128.8, 128.7, 126.1, 122.4, 104.6, 77.3, 77.1, 76.8, 76.7, 36.7, 29.4, 23.7, 21.5, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{18}NaO_2$ ([M + Na]$^+$), 253.1199, found, 253.1194.



2-(1-(3,4-Dimethylphenyl)ethyl)-3-hydroxycyclohex-2-en-1-one (6d):

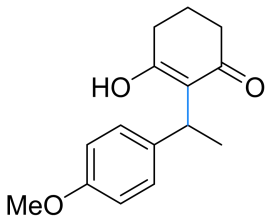
Following **Method B**, **6d** was obtained as colorless oil (48.4 mg, 99% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.10 (d, *J* = 7.8 Hz, 1H), 7.03 (s, 1H), 7.00 (d, *J* = 7.8 Hz, 1H), 5.29 (s, 1H), 5.14 (q, *J* = 6.6 Hz, 1H), 2.49 – 2.38 (m, 2H), 2.32 – 2.26 (m, 2H), 2.25 (s, 3H), 2.23 (s, 3H), 2.00 – 1.90 (m, 2H), 1.56 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.7, 138.8, 137.0, 136.5, 130.0, 126.8, 122.8, 104.5, 76.7, 36.7, 29.4, 23.7, 21.2, 19.9, 19.5. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{20}NaO_2$ ([M + Na]$^+$), 267.1453, found, 267.1433.



2-(1-(4-(*Tert*-butyl)phenyl)ethyl)-3-hydroxycyclohex-2-en-1-one (6e):

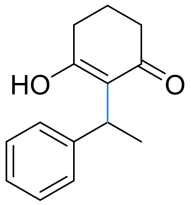
Following **Method B**, **6e** was obtained as colorless oil (45.8 mg, 84% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ

7.37 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 5.32 (s, 1H), 5.21 (q, *J* = 6.6 Hz, 1H), 2.52-2.41 (m, 2H), 2.37 – 2.24 (m, 2H), 2.03-1.92 (m, 2H), 1.59 (d, *J* = 6.6 Hz, 3H), 1.33 (s, 9H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.6, 150.9, 138.2, 125.7, 125.1, 104.5, 76.6, 36.7, 34.6, 31.3, 29.4, 23.5, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{18}H_{24}NaO_2$ ($[M + Na]^+$), 295.1548, found, 295.1549.



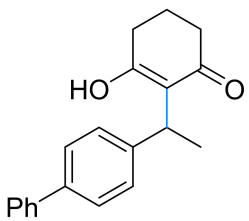
3-Hydroxy-2-(1-(4-methoxyphenyl)ethyl)cyclohex-2-en-1-one (6f):

Following **Method B**, **6f** was obtained as colorless oil (48.8 mg, 99% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.21 (d, *J* = 8.5 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 5.30 (s, 1H), 5.17 (q, *J* = 6.5 Hz, 1H), 3.80 (s, 3H), 2.49 – 2.36 (m, 2H), 2.34 – 2.23 (m, 2H), 2.01 – 1.88 (m, 2H), 1.56 (d, *J* = 6.5 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.7, 159.3, 133.4, 126.8, 114.2, 104.5, 76.4, 55.3, 36.6, 29.4, 23.5, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{18}NaO_3$ ($[M + Na]^+$), 269.1147, found, 269.1148.



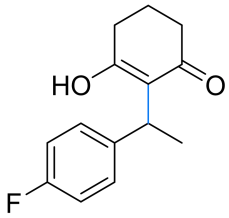
3-Hydroxy-2-(1-phenylethyl)cyclohex-2-en-1-one (6g):

Following **Method B**, **6g** was obtained as colorless oil (26.8 mg, 62% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.34 (t, *J* = 7.2 Hz, 2H), 7.27 (t, *J* = 9.0 Hz, 3H), 5.27 (s, 1H), 5.20 (q, *J* = 6.6 Hz, 1H), 2.51 – 2.40 (m, 2H), 2.34 – 2.23 (m, 2H), 2.02 – 1.91 (m, 2H), 1.58 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.5, 141.4, 128.8, 128.0, 125.4, 104.6, 76.7, 36.7, 29.4, 23.7, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{16}NaO_2$ ($[M + Na]^+$), 239.1041, found, 239.1043.



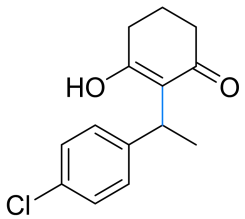
2-(1-([1,1'-Biphenyl]-4-yl)ethyl)-3-hydroxycyclohex-2-en-1-one (6h):

Following **Method B**, **6h** was obtained as colorless oil (57.8 mg, 99% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.8 Hz, 4H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.39 – 7.35 (m, 3H), 5.35 (s, 1H), 5.28 (q, *J* = 6.6 Hz, 1H), 2.55 – 2.45 (m, 2H), 2.39 – 2.26 (m, 2H), 2.06 – 1.93 (m, 2H), 1.65 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.5, 141.1, 140.6, 140.3, 128.8, 127.6, 127.4, 127.1, 125.9, 104.7, 76.4, 36.7, 29.4, 23.6, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{20}H_{20}NaO_2$ ($[M + Na]^+$), 315.1432, found, 315.1432.



2-(1-(4-Fluorophenyl)ethyl)-3-hydroxycyclohex-2-en-1-one (6i):

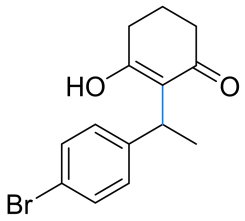
Following **Method B**, **6i** was obtained as colorless oil (45.0 mg, 96% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.30 – 7.24 (m, 2H), 7.05 (t, *J* = 8.4 Hz, 2H), 5.27 (s, 1H), 5.21 (q, *J* = 6.6 Hz, 1H), 2.52 – 2.39 (m, 2H), 2.36 – 2.26 (m, 2H), 2.04 – 1.91 (m, 2H), 1.58 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.6, 176.3, 162.3 (d, *J* = 246.9 Hz), 137.1 (d, *J* = 3.4 Hz), 127.1 (d, *J* = 8.1 Hz), 115.7 (d, *J* = 21.0 Hz), 104.6, 75.9, 36.6, 29.3, 23.6, 21.1. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -113.98 – -114.05 (m, 1F). **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{15}FNaO_2$ ($[M + Na]^+$), 257.0946, found, 257.0948.



2-(1-(4-Chlorophenyl)ethyl)-3-hydroxycyclohex-2-en-1-one (6j):

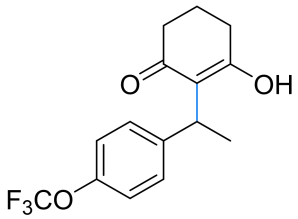
Following **Method B**, **6j** was obtained as colorless oil (34.0 mg, 68% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 5.23 (s, 1H), 5.17 (q, *J* = 6.6 Hz, 1H), 2.51 – 2.38 (m, 2H), 2.34 – 2.24 (m, 2H), 2.01 – 1.91 (m, 2H), 1.56 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.6, 176.2, 139.9, 133.8, 129.1, 126.8,

104.7, 77.2, 77.0, 76.8, 75.9, 36.6, 29.3, 23.6, 21.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{15}ClNaO_2$ ([M + Na]$^+$), 273.0651, found, 273.0652.



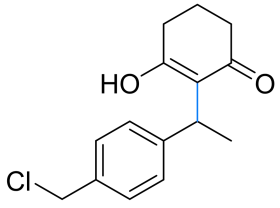
2-(1-(4-Bromophenyl)ethyl)-3-hydroxycyclohex-2-en-1-one (6k):

Following **Method B**, **6k** was obtained as colorless oil (34.8 mg, 59% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 5.24 (s, 1H), 5.18 (q, *J* = 6.6 Hz, 1H), 2.53 – 2.40 (m, 2H), 2.36 – 2.25 (m, 2H), 2.04 – 1.92 (m, 2H), 1.57 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.5, 176.1, 140.4, 132.0, 127.1, 121.9, 104.7, 75.9, 36.6, 29.3, 23.5, 21.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{15}BrNaO_2$ ([M + Na]$^+$), 317.0145, found, 317.0147.



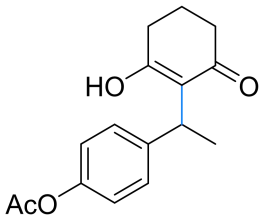
3-Hydroxy-2-(1-(4-(trifluoromethoxy)phenyl)ethyl)cyclohex-2-en-1-one (6l):

Following **Method B**, **6l** was obtained as colorless oil (34.8 mg, 58% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.4 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 2H), 5.26 – 5.19 (m, 2H), 2.53 – 2.39 (m, 2H), 2.35 – 2.25 (m, 2H), 1.97 (qt, *J* = 7.2, 6.0 Hz, 2H), 1.57 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.6, 176.2, 148.8, 140.0, 126.8, 121.3, 120.4 (q, *J* = 255.0 Hz), 104.6, 75.7, 36.6, 29.3, 23.6, 21.1. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = -57.85. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{15}F_3NaO_3$ ([M + Na]$^+$), 323.0963, found, 323.0965.



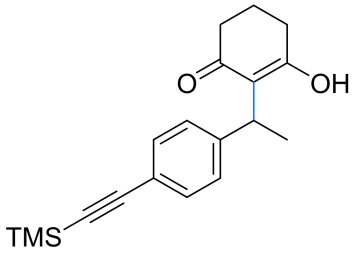
2-(1-(4-(Chloromethyl)phenyl)ethyl)-3-hydroxycyclohex-2-en-1-one (6m):

Following **Method B**, **6m** was obtained as colorless oil (36.4 mg, 69% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.8 Hz, 2H), 7.29 (d, *J* = 7.8 Hz, 2H), 5.26 (s, 1H), 5.22 (q, *J* = 6.6 Hz, 1H), 4.59 (s, 2H), 2.53 – 2.41 (m, 2H), 2.36 – 2.25 (m, 2H), 2.04 – 1.92 (m, 2H), 1.59 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.6, 176.3, 141.6, 137.3, 129.1, 125.8, 104.7, 76.2, 45.8, 36.7, 29.3, 23.6, 21.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{17}ClNaO_2$ ($[M + Na]^+$), 287.0634, found, 287.0635.



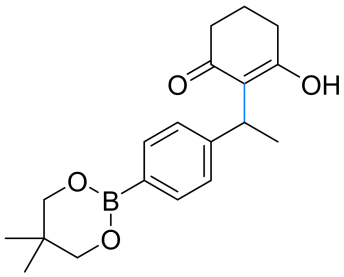
4-(1-(2-Hydroxy-6-oxocyclohex-1-en-1-yl)ethyl)phenyl acetate (6n):

Following **Method B**, **6n** was obtained as colorless oil (50.4 mg, 92% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 5.29 (s, 1H), 5.22 (q, *J* = 6.6 Hz, 1H), 2.53 – 2.39 (m, 2H), 2.36 – 2.26 (m, 5H), 2.03 – 1.91 (m, 2H), 1.58 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.6, 176.3, 169.3, 150.3, 138.8, 126.6, 121.9, 104.5, 76.0, 36.6, 29.4, 23.5, 21.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{18}NaO_4$ ($[M + Na]^+$), 297.1008, found, 297.1009.



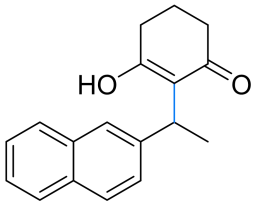
3-Hydroxy-2-(1-(4-((trimethylsilyl)ethynyl)phenyl)ethyl)cyclohex-2-en-1-one (6o):

Following **Method B**, **6o** was obtained as colorless oil (65.0 mg, 40% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 5.22 (s, 1H), 5.17 (q, *J* = 6.5 Hz, 1H), 2.51 – 2.36 (m, 2H), 2.34 – 2.20 (m, 2H), 2.04 – 1.86 (m, *J* = 6.5 Hz, 2H), 1.55 (d, *J* = 6.5 Hz, 3H), 0.24 (s, 8H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.6, 176.3, 141.7, 132.5, 125.3, 123.0, 104.8, 104.6, 94.8, 76.2, 36.8, 29.4, 23.5, 21.2, 0.00. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{19}H_{24}NaO_2Si$ ($[M + Na]^+$), 335.1386, found, 335.1388.



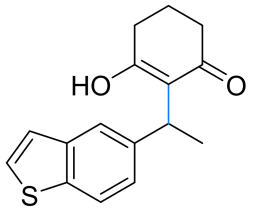
2-(1-(4-(5,5-Dimethyl-1,3,2-dioxaborinan-2-yl)phenyl)ethyl)-3-hydroxycyclohex-2-en-1-one (6p):

Following **Method B**, **6p** was obtained as colorless oil (28.8 mg, 44% yield), TLC: R_f = 0.3 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 5.24 (s, 1H), 5.19 (q, *J* = 6.5 Hz, 1H), 3.75 (s, 4H), 2.50 – 2.35 (m, 2H), 2.33 – 2.20 (m, 2H), 2.01 – 1.87 (m, 2H), 1.56 (d, *J* = 6.5 Hz, 3H), 1.00 (s, 6H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.5, 143.7, 134.4, 124.5, 104.6, 76.7, 72.3, 36.7, 31.9, 29.7, 29.4, 23.5, 21.9, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{19}H_{25}BNaO_4$ ($[M + Na]^+$), 351.1582, found, 351.1580.



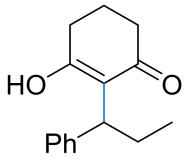
3-Hydroxy-2-(1-(naphthalen-2-yl)ethyl)cyclohex-2-en-1-one (6q):

Following **Method B**, **6q** was obtained as colorless oil (52.2 mg, 98% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.4 Hz, 1H), 7.81 (dt, *J* = 7.2, 2.4 Hz, 2H), 7.72 (s, 1H), 7.53 – 7.44 (m, 2H), 7.40 (dd, *J* = 8.4, 1.8 Hz, 1H), 5.37 (q, *J* = 6.6 Hz, 1H), 5.34 (s, 1H), 2.54 – 2.43 (m, 2H), 2.34 – 2.21 (m, 2H), 2.03 – 1.89 (m, 2H), 1.66 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.5, 138.7, 133.2, 133.1, 128.9, 128.0, 127.8, 126.4, 126.2, 124.5, 123.1, 104.7, 76.8, 36.7, 29.4, 23.7, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{18}H_{18}NaO_2$ ($[M + Na]^+$), 289.1199, found, 289.1188.



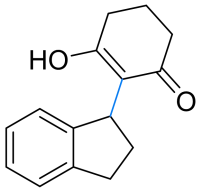
2-(1-(Benzo[b]thiophen-5-yl)ethyl)-3-hydroxycyclohex-2-en-1-one (6r):

Following **Method B**, **6r** was obtained as colorless oil (38.2 mg, 70% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.4 Hz, 1H), 7.73 (d, *J* = 1.8 Hz, 1H), 7.47 (d, *J* = 5.4 Hz, 1H), 7.31 (dd, *J* = 5.4, 0.6 Hz, 1H), 7.28 – 7.26 (m, 1H), 5.35 – 5.30 (m, 2H), 2.54 – 2.41 (m, 2H), 2.34 – 2.22 (m, 2H), 2.03 – 1.89 (m, 2H), 1.64 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.5, 139.8, 139.4, 137.7, 127.4, 123.8, 123.0, 121.7, 120.5, 104.7, 76.8, 36.7, 29.4, 24.0, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{16}NaO_2S$ ($[M + Na]^+$), 295.0955, found, 295.0966.



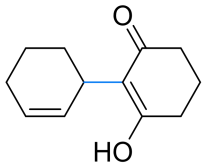
3-Hydroxy-2-(1-phenylpropyl)cyclohex-2-en-1-one (6s):

Following **Method B**, **6s** was obtained as colorless oil (31.4 mg, 68% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 7.0 Hz, 2H), 5.24 (s, 1H), 4.92 (t, *J* = 6.5 Hz, 1H), 2.53 – 2.39 (m, 2H), 2.33 – 2.21 (m, 2H), 2.01 – 1.90 (m, 3H), 1.87 – 1.77 (m, 1H), 0.92 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.8, 140.0, 128.7, 128.0, 125.9, 104.7, 82.0, 36.6, 30.7, 29.3, 21.1, 9.9. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{18}NaO_2$ ($[M + Na]^+$), 253.1308, found, 253.1306.



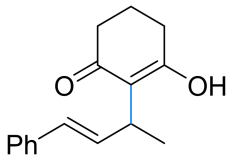
2-(2,3-Dihydro-1H-inden-1-yl)-3-hydroxycyclohex-2-en-1-one (6t):

Following **Method B**, **6t** was obtained as yellow solid (31.4 mg, 99% yield, m.p. 123°C), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.5 Hz, 1H), 7.35 – 7.28 (m, 2H), 7.25 (t, *J* = 7.0 Hz, 1H), 5.63 (dd, *J* = 7.0, 3.5 Hz, 1H), 5.59 (s, 1H), 3.12 (ddd, *J* = 15.5, 8.5, 6.0 Hz, 1H), 2.91 (ddd, *J* = 16.0, 9.0, 5.0 Hz, 1H), 2.55 – 2.46 (m, 1H), 2.45-2.34 (m, 4H), 2.18-2.10 (m, 1H), 2.05-1.96 (m, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 177.3, 144.3, 140.2, 129.4, 126.9, 125.5, 125.0, 103.8, 82.1, 36.8, 31.7, 30.2, 29.4, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{16}NaO_2$ ($[M + Na]^+$), 251.1041, found, 251.1043.



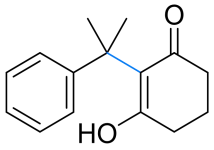
6-Hydroxy-[1,1'-bi(cyclohexane)]-2',6-dien-2-one (6u):

Following **Method B**, **6u** was obtained as colorless oil (38.4 mg, 71% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 6.00 (dt, *J* = 10.0, 3.5 Hz, 1H), 5.77 (dd, *J* = 10.0, 3.0 Hz, 1H), 5.41 (s, 1H), 4.75 – 4.62 (m, 1H), 2.40 (t, *J* = 6.5 Hz, 2H), 2.35 (t, *J* = 6.5 Hz, 2H), 2.16 – 2.00 (m, 2H), 2.01 – 1.95 (m, 2H), 1.93 – 1.80 (m, 2H), 1.80 – 1.72 (m, 1H), 1.68 – 1.58 (m, 1H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.9, 133.4, 124.5, 103.2, 71.5, 36.7, 29.6, 27.8, 24.9, 21.2, 18.7. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{12}H_{16}NaO_2$ ($[M + Na]^+$), 215.0964, found, 215.0964.



(E)-3-hydroxy-2-(4-phenylbut-3-en-2-yl)cyclohex-2-en-1-one (6v):

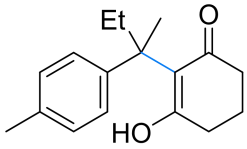
Following **Method B**, **6v** was obtained as colorless oil (37.2 mg, 77% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.8 Hz, 2H), 7.32 (t, *J* = 7.8 Hz, 2H), 7.25 (t, *J* = 7.2 Hz, 1H), 6.56 (d, *J* = 16.2 Hz, 1H), 6.15 (dd, *J* = 16.2, 6.6 Hz, 1H), 5.44 (s, 1H), 4.89 (p, *J* = 6.6 Hz, 1H), 2.42 (t, *J* = 6.6 Hz, 2H), 2.37 – 2.29 (m, 2H), 1.98 (p, *J* = 6.6 Hz, 2H), 1.48 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.8, 176.7, 135.9, 131.9, 128.6, 128.4, 128.1, 126.6, 104.0, 75.1, 36.7, 29.5, 21.2, 21.0. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{16}H_{18}NaO_2$ ($[M + Na]^+$), 265.1365, found, 265.1362.



3-Hydroxy-2-(2-phenylpropan-2-yl)cyclohex-2-en-1-one (6w):

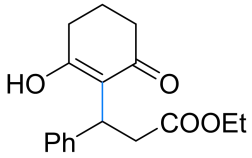
Following **Method B**, **6w** was obtained as colorless oil (39.6 mg, 86% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 – 7.28 (m, 2H), 7.23 (dd, *J* = 15.0, 8.0 Hz, 3H), 4.76 (s, 1H), 2.39 (t, *J* = 6.0 Hz, 2H), 2.24 – 2.17 (m, 2H), 1.91 (p, *J* = 6.5 Hz, 2H), 1.72 (s, 6H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.4, 174.2, 143.8, 128.8, 127.5, 124.6, 108.3, 82.7, 36.4, 30.1, 29.3, 21.2.

HRMS (ESI-TOF) (m/z): Calcd for $C_{15}H_{18}NaO_2$ ([M + Na]$^+$), 253.1308, found, 253.1306.



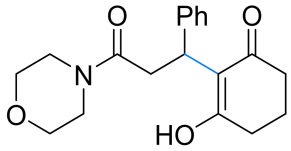
3-Hydroxy-2-(2-(*p*-tolyl)butan-2-yl)cyclohex-2-en-1-one (6x):

Following **Method B**, **6x** was obtained as colorless oil (46.4 mg, 90% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.17 – 7.06 (m, 4H), 4.81 (s, 1H), 2.47 – 2.35 (m, 2H), 2.31 (s, 3H), 2.25 – 2.21 (m, 2H), 2.07 – 1.99 (m, 1H), 1.97 – 1.91 (m, 2H), 1.91 – 1.83 (m, 1H), 1.72 (s, 3H), 0.83 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.5, 174.3, 140.0, 137.0, 129.3, 125.0, 108.4, 85.2, 37.1, 36.5, 30.1, 23.0, 21.2, 21.0, 8.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{22}NaO_2$ ([M + Na]$^+$), 281.1512, found, 281.1514.



Ethyl 3-(2-hydroxy-6-oxocyclohex-1-en-1-yl)-3-phenylpropanoate (6y):

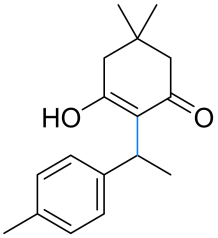
Following **Method B**, **6y** was obtained as colorless oil (34.6 mg, 60% yield), TLC: R_f = 0.3 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.37 (t, *J* = 7.8 Hz, 2H), 7.32 (t, *J* = 7.8 Hz, 3H), 5.55 (dd, *J* = 9.0, 4.8 Hz, 1H), 5.30 (s, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 3.00 (dd, *J* = 16.2, 9.0 Hz, 1H), 2.74 (dd, *J* = 16.2, 4.8 Hz, 1H), 2.49 – 2.38 (m, 2H), 2.38 – 2.19 (m, 2H), 2.03 – 1.86 (m, 2H), 1.26 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.5, 176.0, 169.6, 138.6, 129.0, 128.6, 125.8, 105.2, 76.8, 60.9, 43.1, 36.6, 29.0, 21.1, 14.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{20}NaO_4$ ([M + Na]$^+$), 311.1118, found, 311.1117.



3-Hydroxy-2-(3-morpholino-3-oxo-1-phenylpropyl)cyclohex-2-en-1-one (6z):

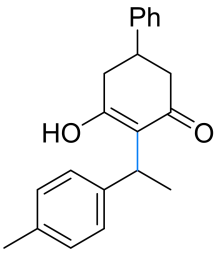
Following **Method B**, **6a** was obtained as colorless oil (31.0 mg, 47% yield), TLC: R_f = 0.3 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.35 (d, *J* = 6.5 Hz, 2H), 7.30 (d, *J* = 7.0 Hz, 3H), 5.66 (dd, *J* = 8.0, 4.5 Hz, 1H), 5.28 (s, 1H), 3.68 – 3.56 (m, 5H), 3.49 – 3.39 (m, 2H), 3.37 – 3.29 (m, 1H), 3.04 (dd, *J* = 15.5, 8.0 Hz, 1H), 2.68 (dd, *J* = 15.5, 4.5 Hz, 1H), 2.51 – 2.36 (m, 2H), 2.33 – 2.20 (m,

2H), 2.02 – 1.87 (m, 2H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 199.4, 175.7, 167.6, 139.4, 129.0, 128.5, 125.7, 105.5, 77.2, 66.8, 66.5, 46.1, 42.1, 41.2, 36.6, 29.1, 21.0. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{19}H_{23}NNaO_4$ ($[M + Na]^+$), 352.1657, found, 352.1656.



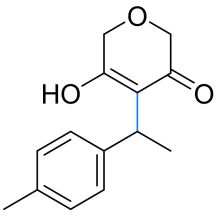
3-Hydroxy-5,5-dimethyl-2-(1-(*p*-tolyl)ethyl)cyclohex-2-en-1-one (7b):

Following **Method B**, **7b** was obtained as colorless oil (51.2 mg, 99% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.16 (s, 4H), 5.27 (s, 1H), 5.20 (q, *J* = 6.6 Hz, 1H), 2.38 – 2.27 (m, 5H), 2.21 – 2.11 (m, 2H), 1.58 (d, *J* = 6.6 Hz, 3H), 1.09 (s, 3H), 1.03 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.6, 174.9, 138.4, 137.7, 129.5, 125.3, 103.5, 76.6, 50.6, 43.3, 32.5, 28.3, 28.2, 23.7, 21.1. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{22}NaO_2$ ($[M + Na]^+$), 257.1535, found, 257.1536.



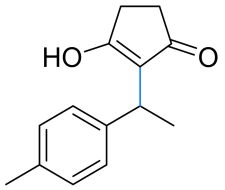
5-Hydroxy-4-(1-(*p*-tolyl)ethyl)-1,6-dihydro-[1,1'-biphenyl]-3(2H)-one (7c):

Following **Method B**, **7c** was obtained as colorless oil (62.0 mg, 99% yield), dr = 1:1, TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37-7.30 (m, 2H), 7.29 – 7.21 (m, 3H), 7.21-7.17 (m, 2H), 7.15 (s, 2H), 5.37 (s, 1H), 5.26-5.17 (m, 1H), 3.41-3.25 (m, 1H), 2.77 – 2.43 (m, 4H), 2.34 (d, *J* = 4.5 Hz, 3H), 1.58 (dd, *J* = 10.0, 6.5 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 198.8, 175.7, 175.5, 142.8, 138.3, 138.1, 137.9, 129.6, 129.5, 128.8, 127.0, 126.7, 125.4, 125.4, 104.4, 104.4, 77.2, 76.8, 43.8, 39.4, 39.2, 37.0, 36.9, 23.7, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{21}H_{22}NaO_2$ ($[M + Na]^+$), 329.1512, found, 329.1513.



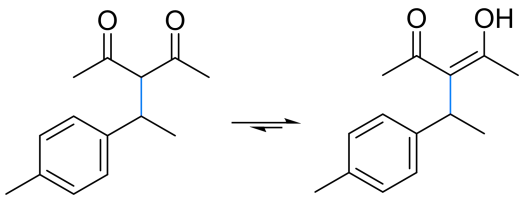
5-Hydroxy-4-(1-(*p*-tolyl)ethyl)-2H-pyran-3(6H)-one (7d):

Following **Method B**, **7d** was obtained as colorless oil (46.0 mg, 99% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.16 (s, 4H), 5.38 (s, 1H), 5.21 (q, *J* = 6.5 Hz, 1H), 4.34 – 4.21 (m, 2H), 4.02 (d, *J* = 3.5 Hz, 2H), 2.34 (s, 3H), 1.59 (d, *J* = 6.5 Hz, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 194.9, 173.9, 138.3, 137.5, 129.6, 125.4, 101.8, 77.7, 71.4, 65.8, 23.4, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{16}NaO_3$ ($[M + Na]^+$), 255.1148, found, 255.1148.



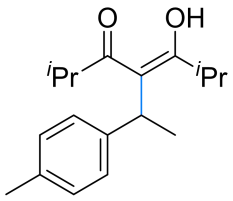
3-Hydroxy-2-(1-(*p*-tolyl)ethyl)cyclopent-2-en-1-one (7e):

Following **Method C**, **7e** was obtained as colorless oil (15.2 mg, 35% yield), TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 5:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.21 – 7.13 (m, 4H), 5.18 (s, 1H), 5.16 (q, *J* = 6.5 Hz, 1H), 2.69 – 2.54 (m, 2H), 2.41 – 2.35 (m, 2H), 2.34 (s, 3H), 1.63 (d, *J* = 6.5 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 206.1, 188.9, 138.2, 137.8, 129.5, 125.6, 106.4, 80.5, 33.8, 28.9, 23.3, 21.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{16}NaO_2$ ($[M + Na]^+$), 239.0970, found, 239.0971.



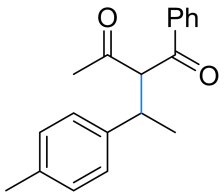
4-Hydroxy-3-(1-(*p*-tolyl)ethyl)pentan-2-one (7f):

Following **Method C**, **7f** was obtained as colorless oil (28.6 mg, 65% yield), keto : enol = 1:1.3, TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 2:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.17 (s, 4H), 7.09 (q, *J* = 8.0 Hz, 4 H), 5.39 (s, 1H), 5.15 (q, *J* = 6.5 Hz, 1H), 4.02 (d, *J* = 11.5 Hz, 1 H), 3.56 (dq, *J* = 13.5, 7.0 Hz, 1 H), 2.34 (s, 3 H), 2.31 (s, 3 H), 2.30 (s, 3 H), 2.27 (s, 3 H), 2.02 (s, 3H), 1.85 (s, 3 H), 1.54 (d, *J* = 6.5 Hz, 3H), 1.20 (d, *J* = 7.0 Hz, 3 H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 203.7, 197.0, 170.6, 140.0, 138.9, 137.5, 136.6, 129.5, 129.4, 127.1, 125.2, 101.8, 77.0, 76.0, 40.1, 32.0, 29.8, 29.7, 23.9, 21.1, 21.0, 20.0. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{14}H_{18}NaO_2$ ($[M + Na]^+$), 241.1204, found, 241.1206.



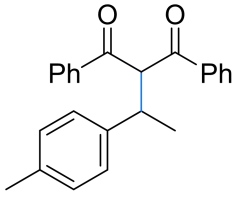
5-Hydroxy-2,6-dimethyl-4-(1-(*p*-tolyl)ethyl)hept-4-en-3-one (7g):

Following **Method C**, **7g** was obtained as colorless oil (19.8 mg, 36% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.16 (s, 4H), 5.22 (s, 1H), 5.09 (q, *J* = 6.5 Hz, 1H), 4.02 – 3.91 (m, 1H), 2.33 (s, 4H), 1.53 (d, *J* = 6.5 Hz, 3H), 1.12 (d, *J* = 7.0 Hz, 3H), 1.09 (d, *J* = 7.0 Hz, 3H), 0.97 (d, *J* = 7.0 Hz, 3H), 0.89 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 203.2, 177.8, 139.2, 137.4, 129.3, 125.1, 98.5, 75.4, 42.1, 29.6, 23.7, 21.1, 19.8, 19.7, 18.8, 18.8. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{18}H_{26}NaO_2$ ([M + Na]$^+$), 297.1694, found, 297.1696.



1-Phenyl-2-(1-(*p*-tolyl)ethyl)butane-1,3-dione (7i):

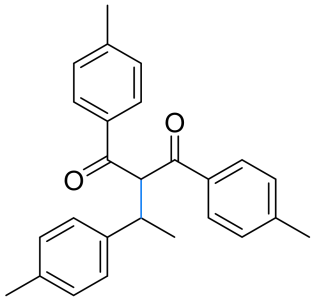
Following **Method C**, **7i** was obtained as colorless oil (29.2 mg, 52% yield), dr = 1:1.4, TLC: R_f = 0.5 (Petroleum ether : Ethyl acetate = 10:1) [UV]. The characterization data of **7i** was full agreement with the reported literature.[8] **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.09 (d, *J* = 10 Hz, 2H), 7.81 (d, *J* = 7.5 Hz, 2H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.53 - 7.49 (m, 2H), 7.47 - 7.43 (m, 1H), 7.36 (t, *J* = 7.5 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 7.5 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 8.0 Hz, 2H), 4.88 (d, *J* = 11.5 Hz, 1H), 4.80 (d, *J* = 11.0 Hz, 1H), 3.87 - 3.78 (m, 2H), 2.32 (s, 3H), 2.23 (s, 3H), 2.21(s, 3H), 1.92 (s, 3H), 1.28 (d, *J* = 7.0 Hz, 3H), 1.19 (d, *J* = 6.8 Hz, 3H).



1,3-Diphenyl-2-(1-(*p*-tolyl)ethyl)propane-1,3-dione (7j):

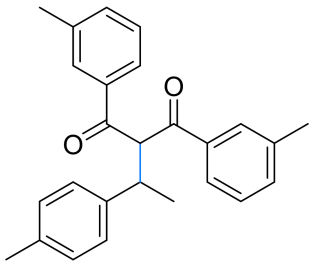
Following **Method C**, **7j** was obtained as colorless oil (45.2 mg, 66% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ

8.06 (d, J = 7.5 Hz, 2H), 7.77 (d, J = 7.5 Hz, 2H), 7.57 (t, J = 7.5 Hz, 1H), 7.45 (t, J = 7.5 Hz, 2H), 7.42 (d, J = 7.5 Hz, 1H), 7.32 – 7.25 (m, 2H), 7.17 (d, J = 8.0 Hz, 2H), 7.00 (d, J = 7.5 Hz, 2H), 5.62 (d, J = 10.0 Hz, 1H), 4.11 – 4.02 (m, 1H), 2.22 (s, 3H), 1.33 (d, J = 7.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 195.1, 194.7, 140.9, 137.2, 137.0, 136.1, 133.6, 133.0, 129.1, 128.9, 128.6, 128.5, 127.6, 65.1, 40.8, 21.0, 20.4. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{24}H_{22}NaO_2$ ([M + Na]$^+$), 365.1408, found, 365.1406.



1,3-Di-*p*-tolyl-2-(1-(*p*-tolyl)ethyl)propane-1,3-dione (7k):

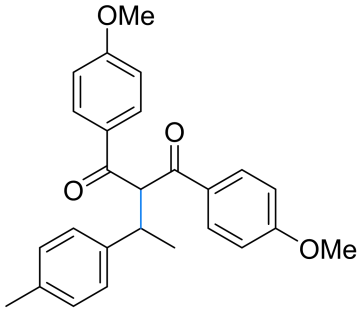
Following **Method C**, **7k** was obtained as yellow solid (59.2 mg, 80% yield, m.p. 171^{o}C), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.97 (d, J = 8.0 Hz, 2H), 7.69 (d, J = 8.0 Hz, 2H), 7.24 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 7.08 (d, J = 8.0 Hz, 2H), 7.00 (d, J = 7.5 Hz, 2H), 5.56 (d, J = 10.0 Hz, 1H), 4.10 – 4.00 (m, 1H), 2.38 (s, 3H), 2.29 (s, 3H), 2.22 (s, 3H), 1.30 (d, J = 7.0 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 194.8, 194.2, 144.4, 143.8, 141.2, 136.0, 134.8, 134.6, 129.5, 129.1, 129.1, 128.7, 127.5, 65.0, 40.7, 21.6, 21.5, 21.0, 20.5. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{26}H_{26}NaO_2$ ([M + Na]$^+$), 393.1720, found, 393.1722.



1,3-Di-*m*-tolyl-2-(1-(*p*-tolyl)ethyl)propane-1,3-dione (7l):

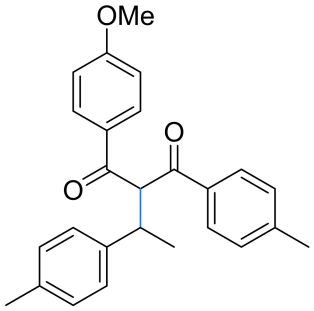
Following **Method C**, **7l** was obtained as colorless oil (39.2 mg, 53% yield), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.84 (d, J = 7.5 Hz, 1H), 7.80 (s, 1H), 7.58 – 7.50 (m, 2H), 7.39 – 7.30 (m, 2H), 7.22 (d, J = 7.5 Hz, 1H), 7.20 – 7.12 (m, 3H), 6.99 (d, J = 7.5 Hz, 2H), 5.56 (d, J = 10.0 Hz, 1H), 4.07 – 3.98 (m, 1H), 2.36 (s, 3H), 2.26 (s, 3H), 2.22 (s, 3H), 1.31 (d, J = 7.0 Hz,

3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 195.3, 194.9, 141.1, 138.7, 138.2, 137.3, 137.1, 136.0, 134.3, 133.7, 129.4, 129.2, 129.0, 128.6, 128.2, 127.6, 126.1, 125.7, 65.1, 40.7, 21.3, 21.2, 20.9, 20.2. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{26}H_{26}NaO_2$ ($[M + Na]^+$), 393.1720, found, 393.1720.



1,3-Bis(4-methoxyphenyl)-2-(1-(*p*-tolyl)ethyl)propane-1,3-dione (7m):

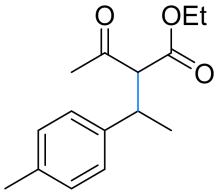
Following **Method C**, **7m** was obtained as yellow solid (79.6 mg, 99% yield, m.p. 165°C), TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.09 (d, *J* = 7.8 Hz, 2H), 7.83 (d, *J* = 7.8 Hz, 2H), 7.18 (d, *J* = 7.8 Hz, 2H), 7.02 (d, *J* = 7.8 Hz, 2H), 6.93 (d, *J* = 7.8 Hz, 2H), 6.79 (d, *J* = 7.8 Hz, 2H), 5.46 (d, *J* = 10.2 Hz, 1H), 4.11 – 4.03 (m, 1H), 3.86 (s, 3H), 3.79 (s, 3H), 2.24 (s, 3H), 1.32 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 193.8, 193.2, 163.8, 163.4, 141.3, 135.9, 131.3, 131.0, 130.3, 130.1, 129.1, 127.5, 114.0, 113.6, 65.3, 55.5, 55.4, 40.6, 21.0, 20.6. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{26}H_{26}NaO_4$ ($[M + Na]^+$), 425.1592, found, 425.1593.



1-(4-Methoxyphenyl)-3-(*p*-tolyl)-2-(1-(*p*-tolyl)ethyl)propane-1,3-dione (7n):

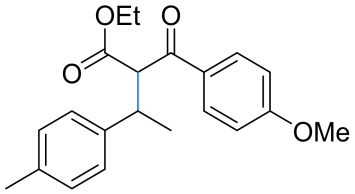
Following **Method C**, **7n** was obtained as yellow solid (37.2 mg, 48% yield, m.p. 170°C), dr = 1:1, TLC: R_f = 0.4 (Petroleum ether : Ethyl acetate = 10:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.05 (d, *J* = 9.0 Hz, 1H), 7.97 (d, *J* = 8.4 Hz, 1H), 7.78 (d, *J* = 9.0 Hz, 1H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.24 (d, *J* = 7.8 Hz, 1H), 7.16 (d, *J* = 7.8 Hz, 2H), 7.08 (d, *J* = 7.8 Hz, 1H), 6.99 (d, *J* = 7.8 Hz, 2H), 6.91 (d, *J* = 9.0 Hz, 1H), 6.76 (d, *J* = 9.0 Hz, 1H), 5.48 (d, *J* = 10.0 Hz, 1H), 4.04 (dq, *J* = 10.2, 7.2 Hz, 1H), 3.81 (d, *J* = 43.2 Hz, 3H), 2.34 (d, *J* = 52.2 Hz, 3H), 2.22 (s, 3H), 1.29 (dd, *J* = 7.2, 1.8 Hz,

3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 195.0, 194.4, 193.6, 193.0, 163.8, 163.4, 144.5, 143.8, 141.3, 141.2, 135.9, 134.8, 134.6, 131.4, 131.0, 130.2, 130.0, 129.5, 129.2, 129.1, 128.8, 127.5, 114.0, 113.6, 65.1, 55.5, 55.4, 40.7, 40.6, 21.7, 21.6, 21.0, 20.6, 20.5. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{26}H_{26}NaO_3$ ($[M + Na]^+$), 409.1669, found, 409.1667.



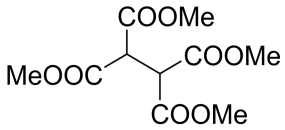
Ethyl-2-acetyl-3-(*p*-tolyl)butanoate (8a):

Following **Method D**, **8a** was obtained as colorless oil (20.9 mg, 42% yield), dr = 1:1, TLC: R_f = 0.6 (Petroleum ether : Ethyl acetate = 5:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.12 – 7.06 (m, 8H), 4.22 (q, *J* = 7.2 Hz, 2H), 3.90 (q, *J* = 7.2 Hz, 2H), 3.76 (d, *J* = 10.8 Hz, 1H), 3.71 (d, *J* = 10.8 Hz, 1H), 3.58 – 3.45 (m, 2H), 2.29 (m, 9H), 1.94 (s, 3H), 1.32 – 1.26 (m, 6H), 1.21 (d, *J* = 7.2 Hz, 3H), 0.97 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 202.6, 202.5, 168.7, 168.2, 140.2, 140.0, 136.4, 136.3, 129.4, 129.1, 127.2, 127.2, 67.7, 67.2, 61.4, 61.1, 39.7, 39.4, 29.8, 29.5, 21.0, 20.7, 20.4, 14.1, 13.7. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{15}H_{20}NaO_3$ ($[M + Na]^+$), 248.1412, found, 248.1408.



Ethyl-2-(4-methoxybenzoyl)-3-(*p*-tolyl)butanoate (8b):

Following **Method D**, **8b** was obtained as colorless oil (30.6 mg, 45% yield), dr = 1:1.4, TLC: R_f = 0.6 (Petroleum ether : Ethyl acetate = 5:1) [UV]. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.10 (d, *J* = 9.0 Hz, 2H), 7.87 (d, *J* = 9.0 Hz, 2H), 7.20 (d, *J* = 7.8 Hz, 2H), 7.11 (d, *J* = 7.8 Hz, 4H), 6.98 (d, *J* = 7.8 Hz, 2H), 6.96 (d, *J* = 9.0 Hz, 2H), 6.85 (d, *J* = 9.0 Hz, 2H), 4.62 (d, *J* = 10.8 Hz, 1H), 4.56 (d, *J* = 10.8 Hz, 1H), 4.23 – 4.10 (m, 2H), 3.88 (s, 3H), 3.85 – 3.78 (m, 7H), 2.31 (s, 3H), 2.21 (s, 3H), 1.34 (d, *J* = 6.0 Hz, 3H), 1.24 – 1.19 (m, 6H), 0.90 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 192.3, 191.9, 169.0, 168.4, 164.0, 163.7, 141.1, 140.6, 136.2, 135.9, 131.2, 130.9, 130.1, 129.8, 129.1, 127.6, 127.2, 113.9, 113.7, 77.3, 77.0, 76.8, 61.5, 61.5, 61.1, 61.1, 55.6, 55.5, 39.8, 39.2, 21.1, 21.0, 20.8, 20.5, 14.1, 13.7. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{21}H_{24}NaO_4$ ($[M + Na]^+$), 363.1567, found, 363.1568.

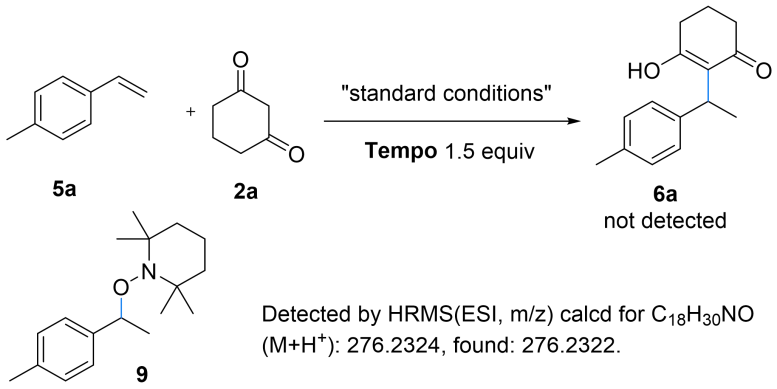


Tetramethylethane-1,1,2,2-tetracarboxylate (13):

12 was obtained as yellow oil, TLC: R_f = 0.6 (Petroleum ether : Ethyl acetate = 5:1) [UV]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.81 (s, 2H), 3.98 (s, 12H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 175.7, 67.6, 55.4. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{10}H_{14}NaO_8$ ($[M + Na]^+$), 285.0581, found, 285.0581.

V. Mechanistic Experiments

a) Radical inhibition experiment



To a dry Schlenk tube containing a magnetic stir bar were added **[Co]-2** (0.003 mmol, 3 mol%), TMFP-BF_4 (0.12 mmol, 1.2 equiv), and dry THF (1 mL) in sequence. After stirred for 5 min, olefin **5a** (0.10 mmol, 1.0 equiv), 1,3-diketone **2a** (0.15 mmol, 1.5 equiv) and radical inhibitors Tempo (0.2 mmol, 1.5 equiv) were added. Then TMDSO (0.20 mmol, 2.0 equiv) was added dropwise. After stirring for 3 hours, the reaction mixture was extracted with DCM, and the combined organic layers were concentrated in vacuo.

In the system of Tempo, the formation of **6a** was almost completely suppressed. In particular, the Tempo-trapped product **9** was detected by high-resolution mass spectrometry (HRMS) analysis, as shown signal at m/z 276.2322 in **Figure S1**. This result suggested that an alkyl radical intermediate is probably involved in this transformation, consistent with the speculated metalhydride HAT process.

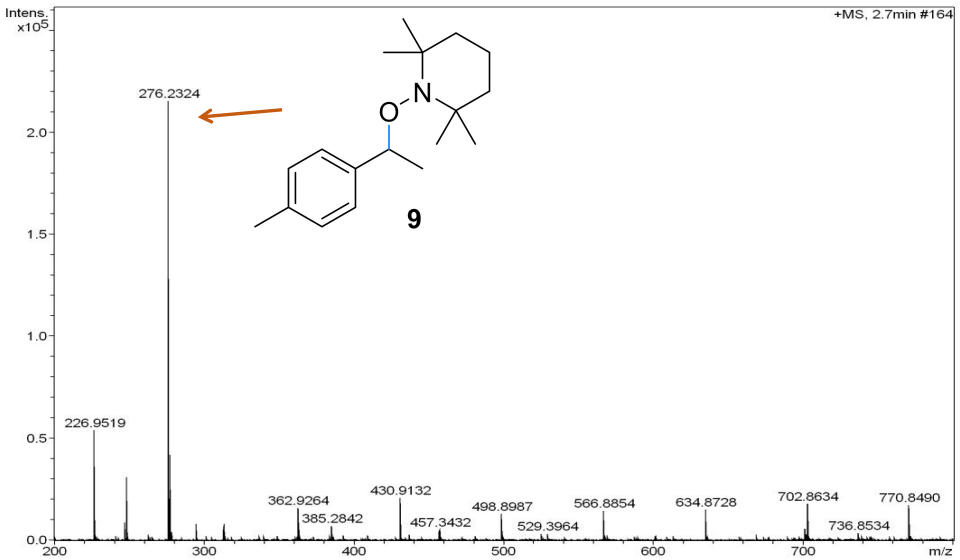


Fig. S1 Tempo trapped alkyl radical intermediate

b) Radical clock experiment

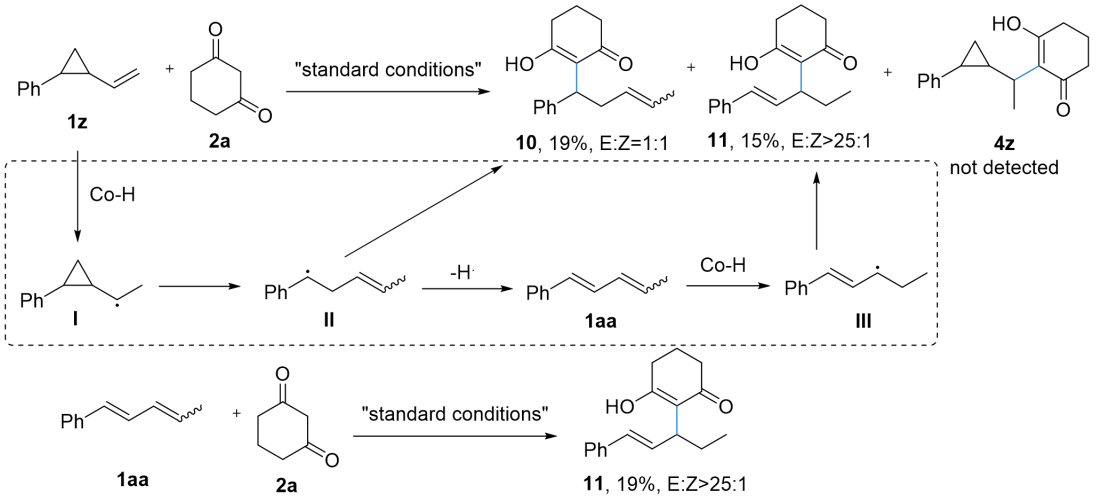


Fig. S2 Radical clock experiment

To a dry Schlenk tube containing a magnetic stir bar were added **[Co]-1** (0.003 mmol, 3 mol%), TMFP-BF_4 (0.20 mmol, 2.0 equiv), and dry toluene (1 mL). After stirred for 5 min, olefin **1z** (0.10 mmol, 1.0 equiv) and 1,3-diketone **2a** (0.15 mmol. 1.5 equiv) were added. Then TMDSO (0.40 mmol, 4.0 equiv) was added dropwise. After stirring for 3 hours,the reaction mixture was extracted with DCM, and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product.

It shows that the hydrocarbonization product **4z** has not been detected, while the ring-opening/carbonization product **10** and **11** was detected in 19% and 15%, respectively. These phenomena suggest that an alkyl radical intermediate is probably involved in this transformation, in line with the speculated CoH-mediated HAT process. The formation of compound **11** probably undergo ring-opening isomerization of olefin **1z** to 1, 3-diene **1aa**, followed by CoH-mediated hydrofunctionalization process. To test this hypothesis, we use presynthesized 1,3-dienes **1aa** to conduct the reaction under standard conditions. As we expected, the desired product **11** was obtained in 19% yield with excellent stereoselectivity.

3-Hydroxy-2-(1-phenylpent-3-en-1-yl)cyclohex-2-en-1-one (10):

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (t, *J* = 7.5 Hz, 2H), 7.29 – 7.26 (m, 1H), 7.25 – 7.20 (m, 2H), 5.61 – 5.43 (m, 1H), 5.37 – 5.27 (m, 1H), 5.23 (s, 1H), 4.99 (dt, *J* = 14.0, 6.5 Hz, 1H), 2.74 – 2.37 (m, 4H), 2.32 – 2.20 (m, 2H), 2.02 – 1.86 (m, 2H), 1.63 (dd, *J* = 6.5, 1.5 Hz, 1.5H), 1.51 (dd, *J* = 6.5, 1.5 Hz, 1.5H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.7, 176.6, 139.7, 139.7, 129.0, 128.7, 128.7, 128.1, 128.1, 127.5, 125.9,

125.9, 125.4, 124.4, 104.9, 104.8, 80.7, 80.3, 40.9, 36.7, 35.3, 29.3, 21.1, 18.0, 12.9. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{20}NaO_2$ ([M + Na]$^+$), 279.1356, found, 279.1366.

(E)-3-hydroxy-2-(1-phenylpent-1-en-3-yl)cyclohex-2-en-1-one (11):

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.0 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.27 – 7.25 (m, 1H), 6.54 (d, *J* = 16.0 Hz, 1H), 6.07 (dd, *J* = 16.0, 7.5 Hz, 1H), 5.41 (s, 1H), 4.61 (q, *J* = 6.5 Hz, 1H), 2.43 (td, *J* = 6.5, 2.0 Hz, 2H), 2.32 (t, *J* = 6.5 Hz, 2H), 1.98 (p, *J* = 6.5 Hz, 3H), 1.84 (dq, *J* = 14.5, 7.0 Hz, 1H), 1.74 (dq, *J* = 14.0, 7.0 Hz, 1H), 0.96 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.9, 177.0, 135.9, 132.9, 128.6, 128.1, 127.0, 126.6, 104.1, 80.6, 36.7, 29.4, 29.4, 28.3, 21.2, 9.5. **HRMS** (ESI-TOF) (m/z): Calcd for $C_{17}H_{20}NaO_2$ ([M + Na]$^+$), 279.1356, found, 279.1356.

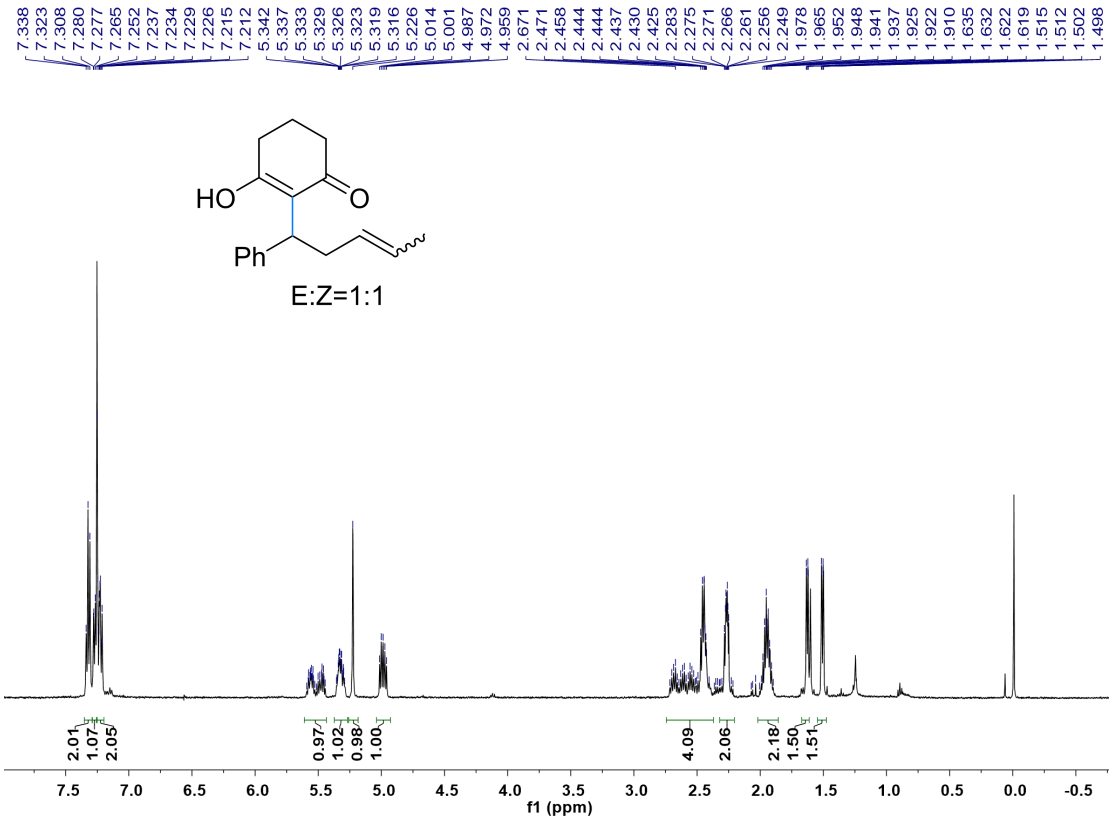


Fig. S3 ^{1}H NMR data of **10**

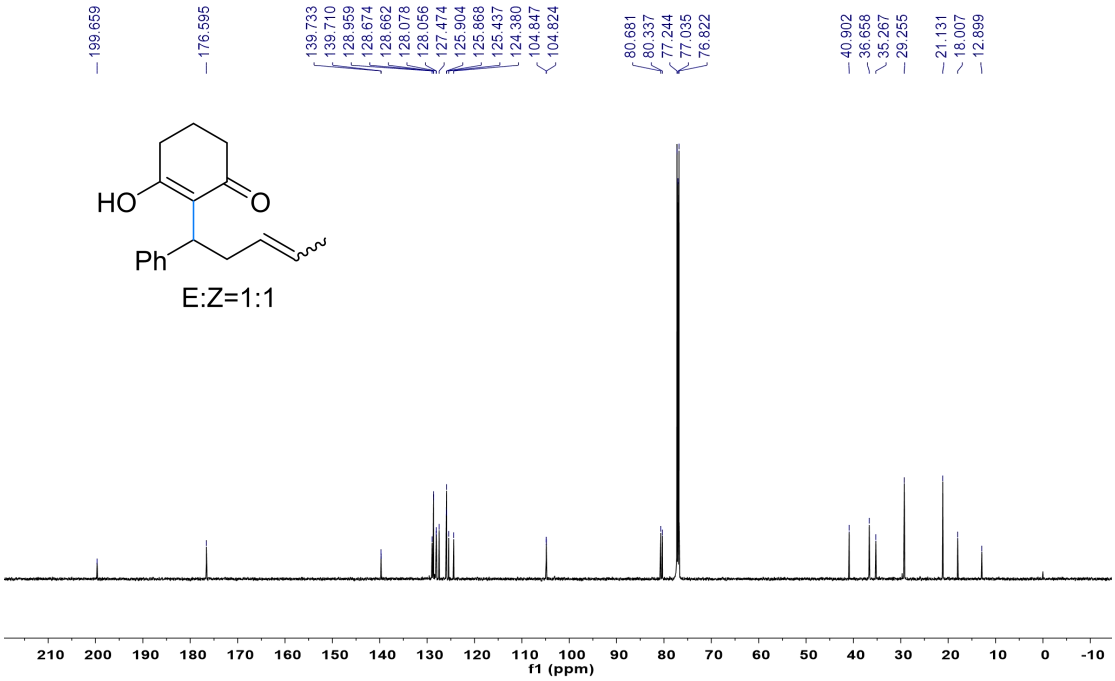


Fig. S4 ^{13}**C NMR** data of **10**

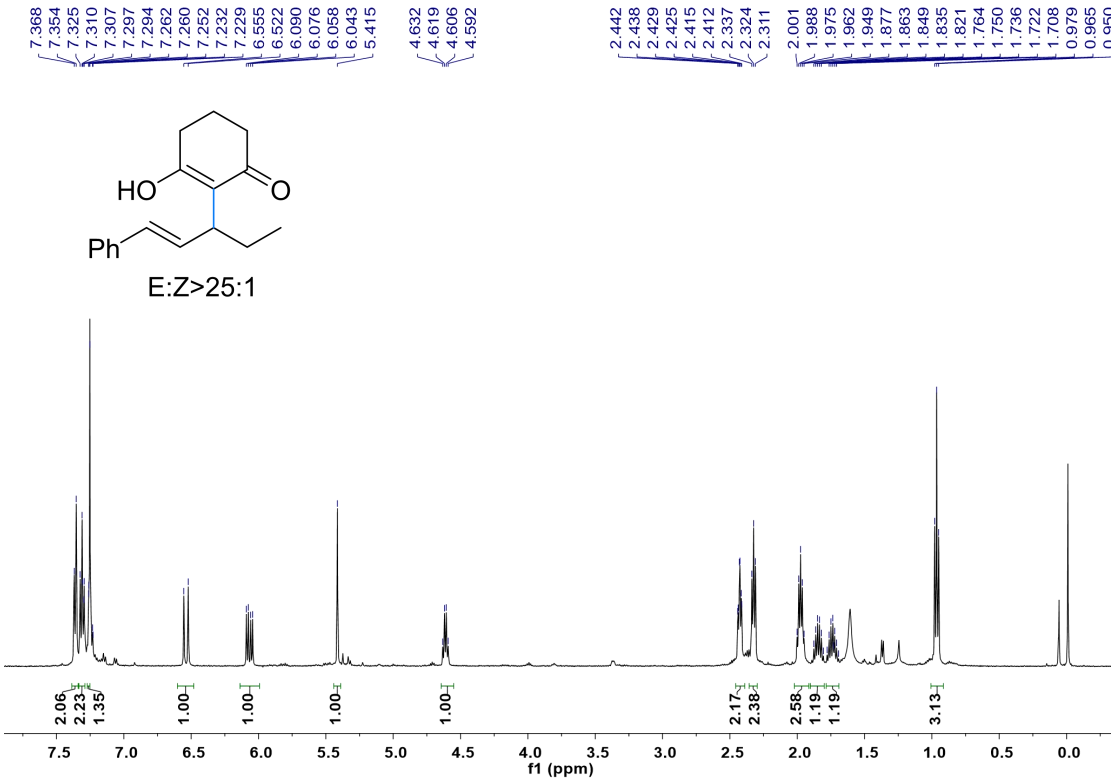


Fig. S5 ^{1}**H NMR** data of **11**

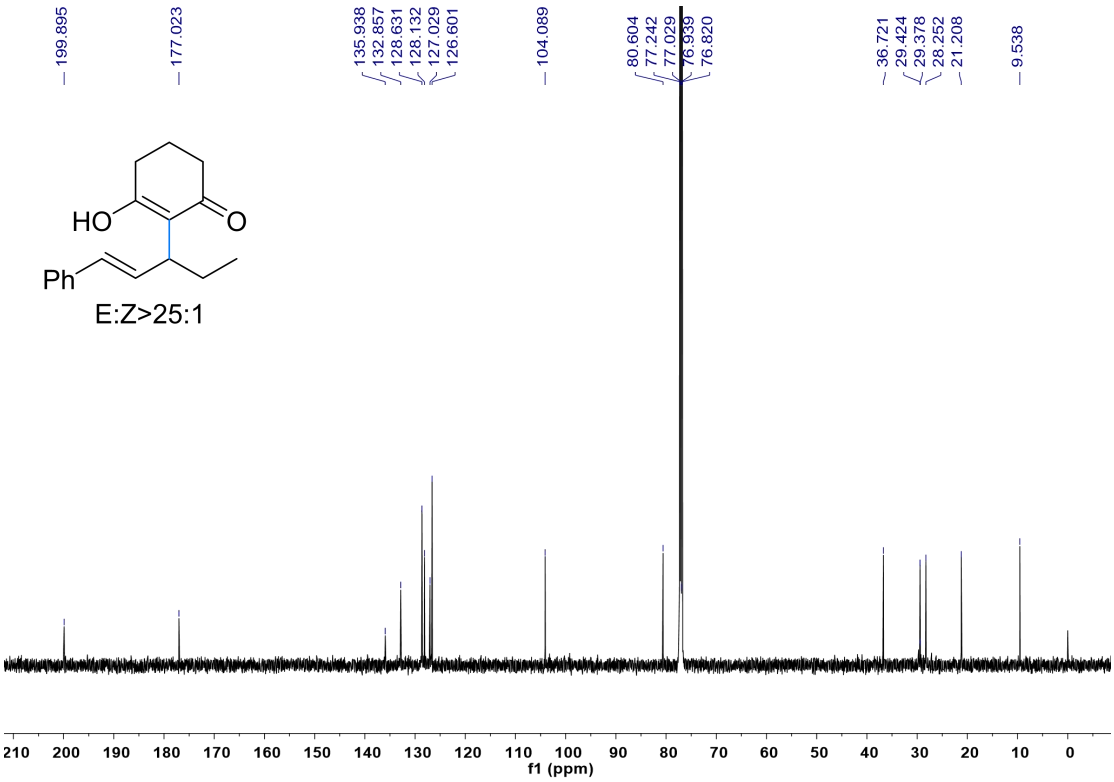
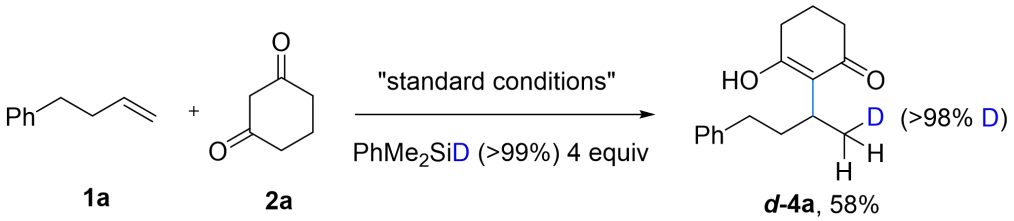


Fig. S6 ^{13}**C NMR** data of **11**

c) Deuterium experiment



1a **2a** ***d*-4a**, 58%

Preparation of Me_2PhSiD: To a stirring suspension of $LiAlD_4$ (210 mg, 5 mmol) in dry Et_2O (12 mL) was added $Me_2PhSiCl$ (2.55g, 15 mmol) dropwise at ambient temperature under Ar. The reaction mixture was refluxed at 40 °C for 12 h. The reaction was cooled to room temperature. Then, the reaction was quenched by adding aqueous solution of sodium hydroxide (15 mL, 10 wt%) into the crude reaction mixture, which was subsequently extracted by diethyl ether for three times. The combined organic layers were dried over Na_2SO_4, evaporated under reduced pressure, and purified by column chromatography on silica gel to give $PhMe_2SiD$ in 82% yield (1.70g, >99% D). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.55 – 7.53 (m, 2H), 7.37 – 7.34 (m, 3H), 4.44 – 4.42 (m, 0.00H), 0.34 (s, 6H).

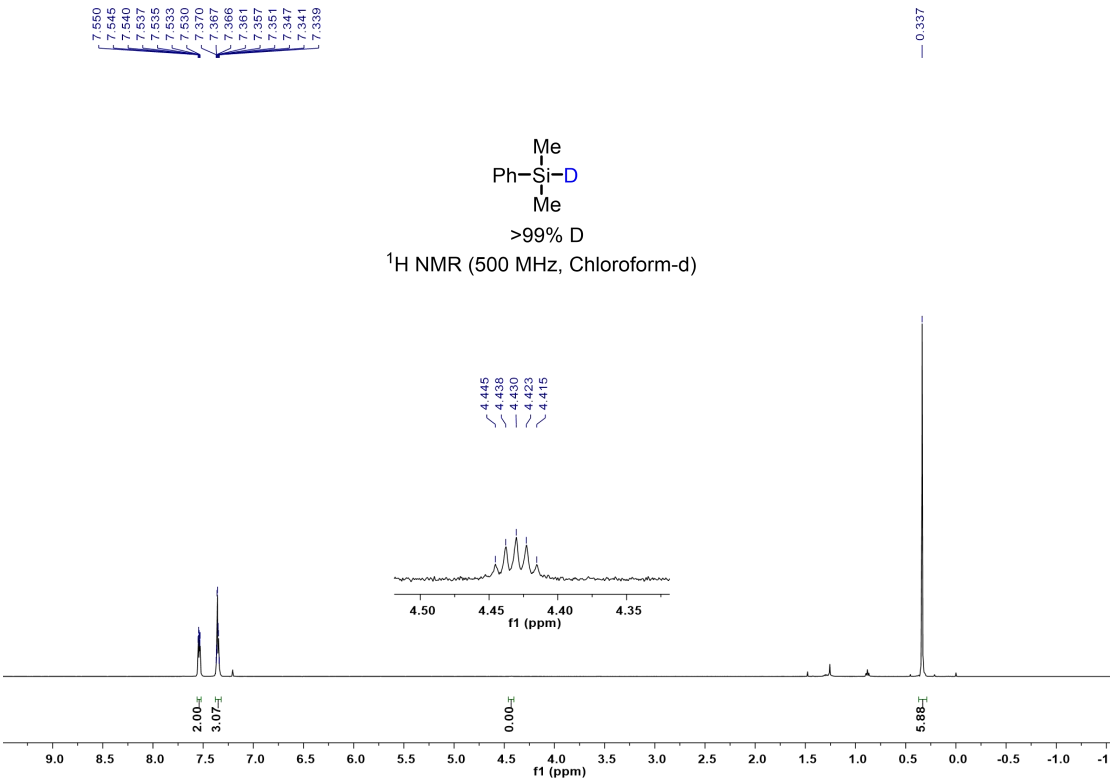


Fig. S7 ^{1}H NMR data of **Me$_2$PhSiD**

A deuterium-labeled experimental reaction of **1a** using PhMe$_2$SiD (＞99% D) was conducted to afford ***d*-4a** in 58% yield with >98% D at the terminal carbon site, which demonstrated that the hydrogen came from hydrosilane and the HAT process was irreversible.

To a dry Schlenk tube containing a magnetic stir bar were added **[Co]-1** (0.003 mmol, 3 mol%), TMFP-BF_4 (0.20 mmol, 2.0 equiv), and dry toluene (1 mL). After stirred for 5 min, olefin **1a** (0.10 mmol, 1.0 equiv) and 1,3-diketone **2a** (0.15 mmol. 1.5 equiv) were added. Then PhMe$_2$SiD (0.40 mmol, 4.0 equiv) was added dropwise. After stirring for 3 hours,the reaction mixture was extracted with DCM, and the combined organic layers were concentrated in vacuo. The resulting crude product was purified by flash column chromatography on silica gel to obtain product.

***d*-4a: ^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.35 – 7.26 (m, 2H), 7.22 – 7.12 (m, 3H), 5.30 (s, 1H), 4.27 (p, J = 6.0 Hz, 1H), 2.85 – 2.58 (m, 2H), 2.45 – 2.30 (m, 4H), 2.06 – 1.94 (m, 3H), 1.90 – 1.81 (m, 1H), 1.27 (d, J = 6.0 Hz, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 199.9, 176.9, 141.1, 128.5, 128.3, 126.1, 103.1, 77.3, 77.1, 76.9, 73.8, 37.6, 36.8, 31.7, 29.5, 29.4, 21.2, 18.8 (t, J = 18.9 Hz). ^{2}H NMR (77 MHz,) δ 1.29.

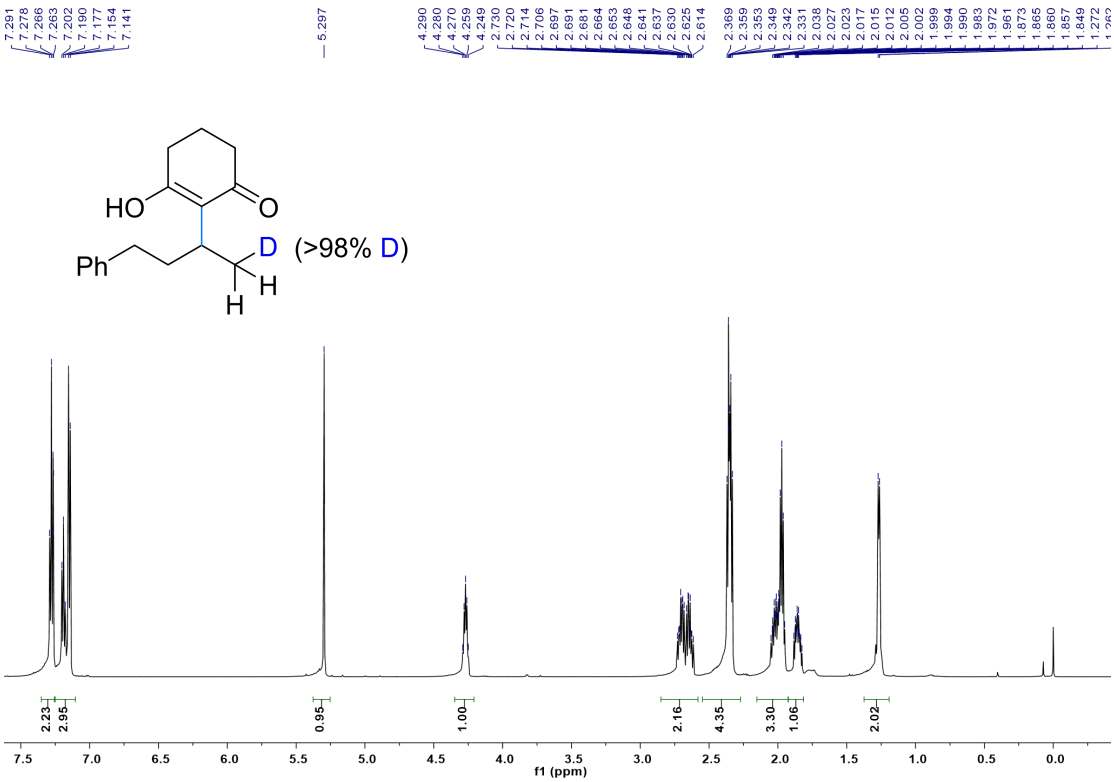


Fig. S8 ^{1}**H NMR** data of ***d*-4a**

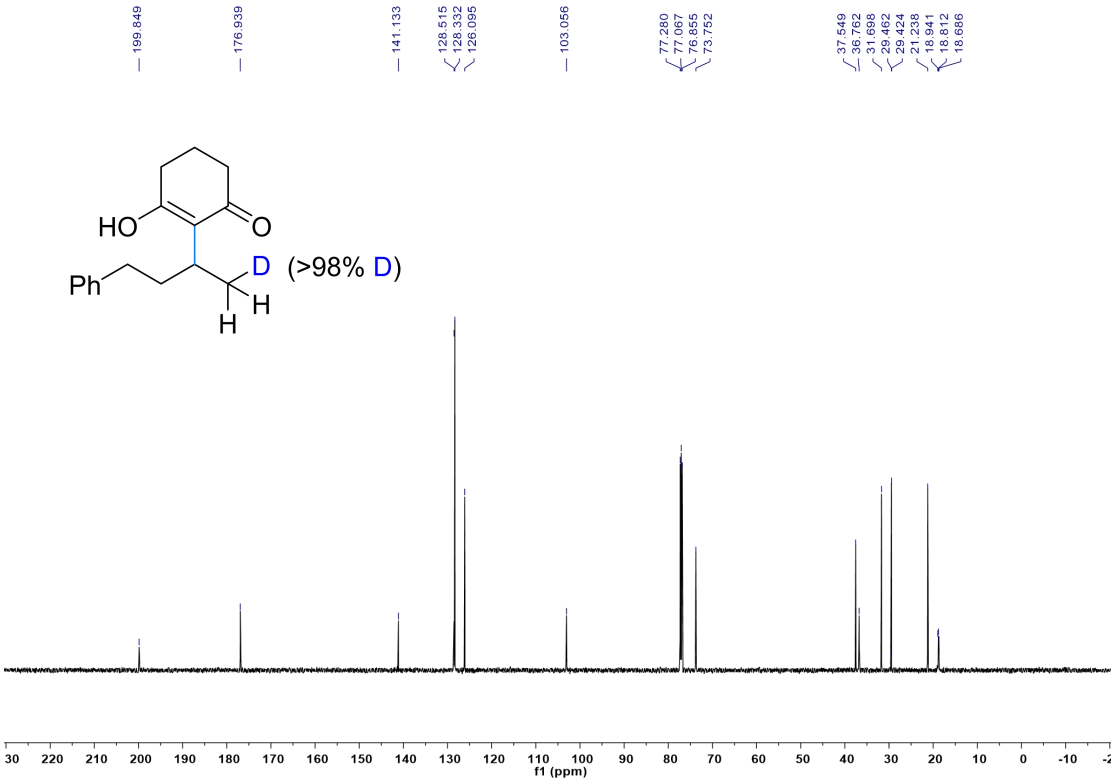


Fig. S9 ^{13}**C NMR** data of ***d*-4a**

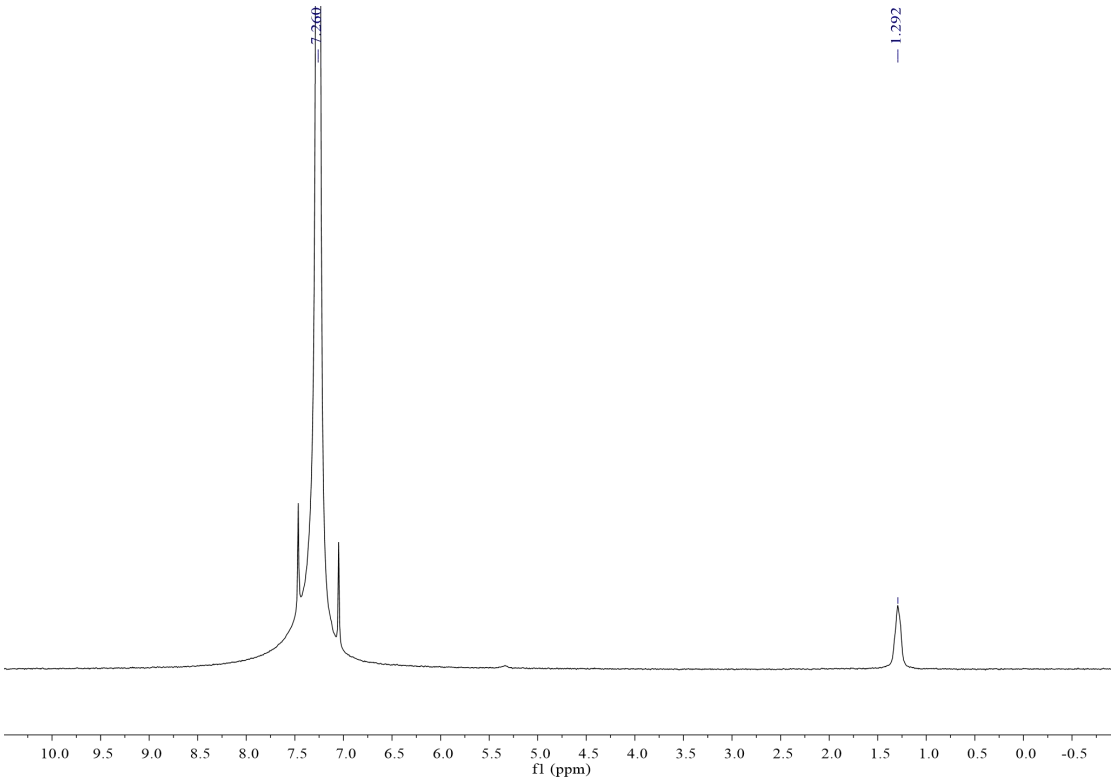
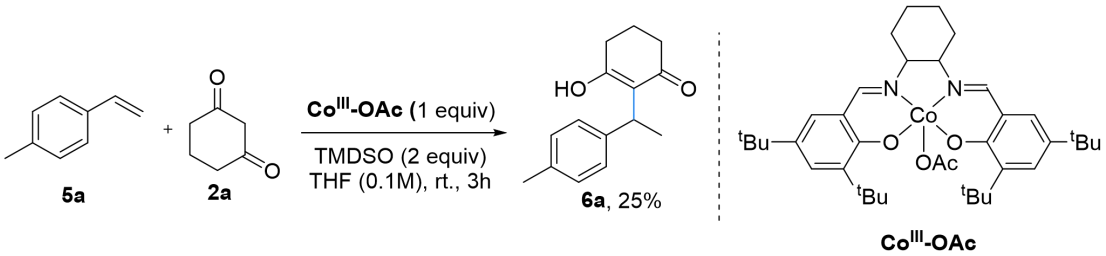


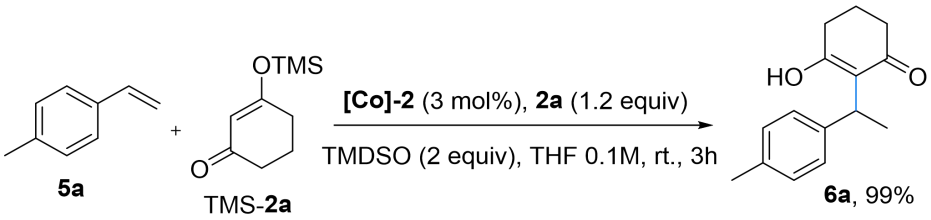
Fig. S10 ^{2}H NMR data of ***d*-4a**

d) Stoichiometric experiments



We performed the preliminary stoichiometric experiments using easily prepared cobalt(III)-salen complexes, and a small amount of desired product **6a** were obtained in the absence of oxidant. These results indicate that cobalt(III) species might be as the single electron oxidant enabling the hydrocarbonization.

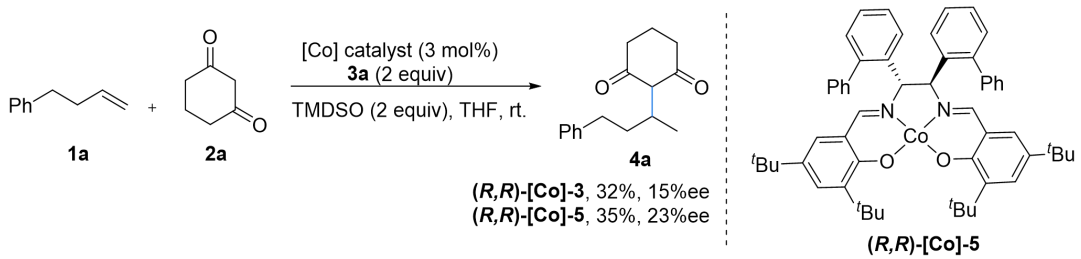
e) Control experiments with TMS-2a



The compound TMS-**2a** was prepared form **2a** using the procedure described for **reference 9**. To a dry Schlenk tube containing a magnetic stir bar were added [**Co]-2** (0.003 mmol, 3 mol%), TMFP-BF_4 (0.12 mmol, 1.2 equiv), and dry THF (1 mL). After stirred for 5 min, olefin **5a** (0.10 mmol, 1.0 equiv) and compound TMS-**2a** (0.15 mmol, 1.5 equiv) were added. Then TMDSO (0.20 mmol, 2.0 equiv) was added dropwise. After stirring for 3 hours, the reaction mixture was extracted with DCM, and the combined organic layers were concentrated in vacuo.

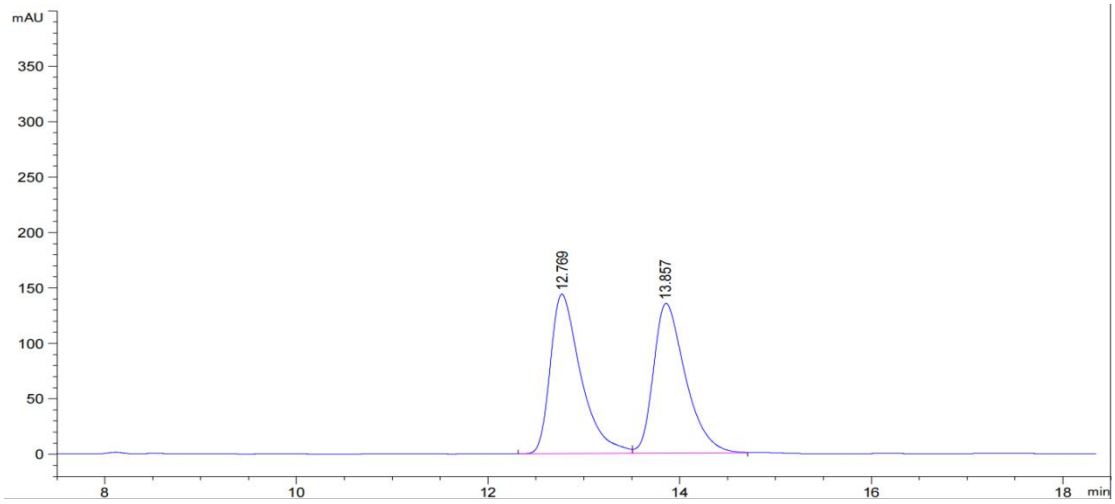
We conducted control experiments to understand the role of the enol in this reaction by using TMS-**2a**. In the case of TMS-**2a**, the yield was similar to that using **2a** (99% yield). These results suggest the enol form of **2a** might be a potential intermediate in this reaction.

f) Control experiments with non-racemic cobalt complex

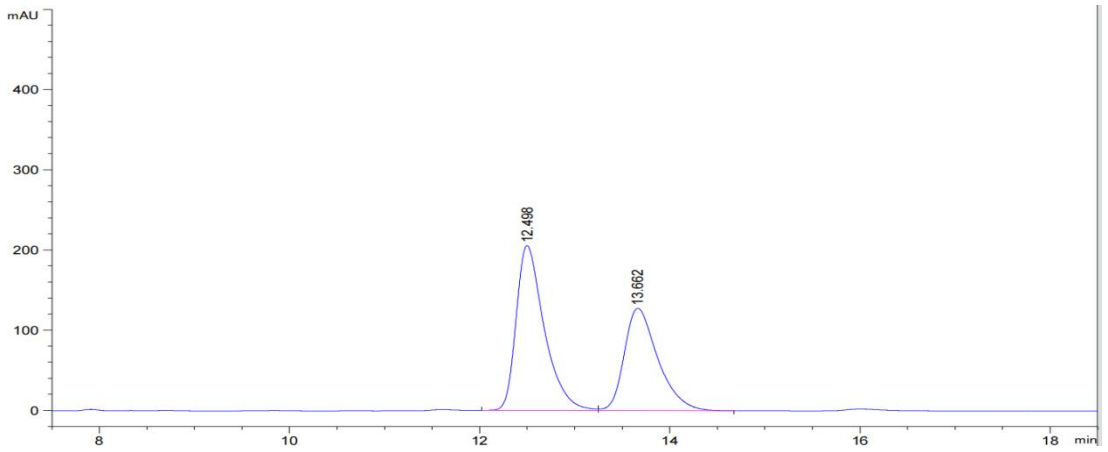


We also tested the non-racemic cobalt complexes **(*R,R*)-[Co]-3** and **(*R,R*)-[Co]-5** to explore asymmetric induction. Although the enantioselectivities with both catalysts were poor, slight asymmetric inductions were observed. These results suggest that the cobalt catalyst interacts with the substrate during the C−C bond formation and that the reaction does not proceed via free radical or achiral cation intermediates.

(*R,R*)-[Co]-3 as catalyst: **HPLC** analysis (AD, Hexane/IPA = 95/5, 0.8 mL/min, 254 nm) indicated 57:42 e.r. tR (major) = 12.50 min, tR (minor) = 13.66 min.

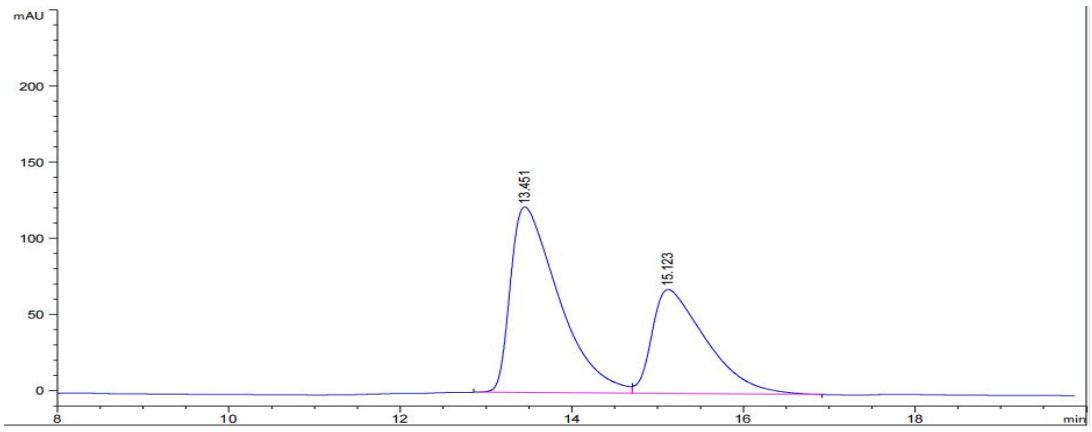


Peak	PetTime	Type	Width(min)	Area(mAU*S)	Hight(mAU)	Area%
1	12.769	MF R	0.3691	3185.87622	143.86394	50.3526
2	13.857	FM R	0.3874	3141.26196	135.13353	49.6474



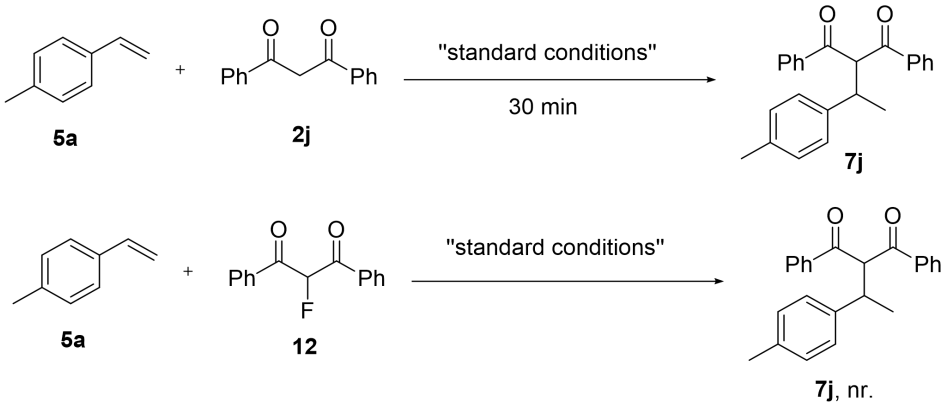
Peak	PetTime	Type	Width(min)	Area(mAU*S)	Hight(mAU)	Area%
1	12.498	MF R	0.3422	4221.75781	205.59094	57.4285
2	13.662	FM R	0.4092	3129.56812	127.47267	42.5715

(*R,R*)-[Co]-5 as catalyst: **HPLC** analysis (AD, Hexane/IPA = 95/5, 0.8 mL/min, 254 nm) indicated 57:42 e.r. tR (major) = 13.45 min, tR (minor) = 15.12 min.



Peak	PetTime	Type	Width(min)	Area(mAU*S)	Hight(mAU)	Area%
1	13.451	MF R	0.6531	4769.58496	121.71183	61.5419
2	15.123	FM R	0.7290	2980.56030	68.14246	38.4581

g) Control experiments with *α*-Fluorinated-1,3-diketone



As for the possibility of electrophilic fluorination of the *β*-diketone **2** under this Co-catalyzed transformation,[10] the reaction mixture within 30 min was studied under ^{19}F-NMR analysis, and no fluorinated intermediate was observed except for the fluorine signal of the oxidant **3a**. We also used the presynthesized α-fluorinated 1,3-diactone **12**[11] to conduct the reaction under standard conditions, notably, the desired product **7j** was not observed.

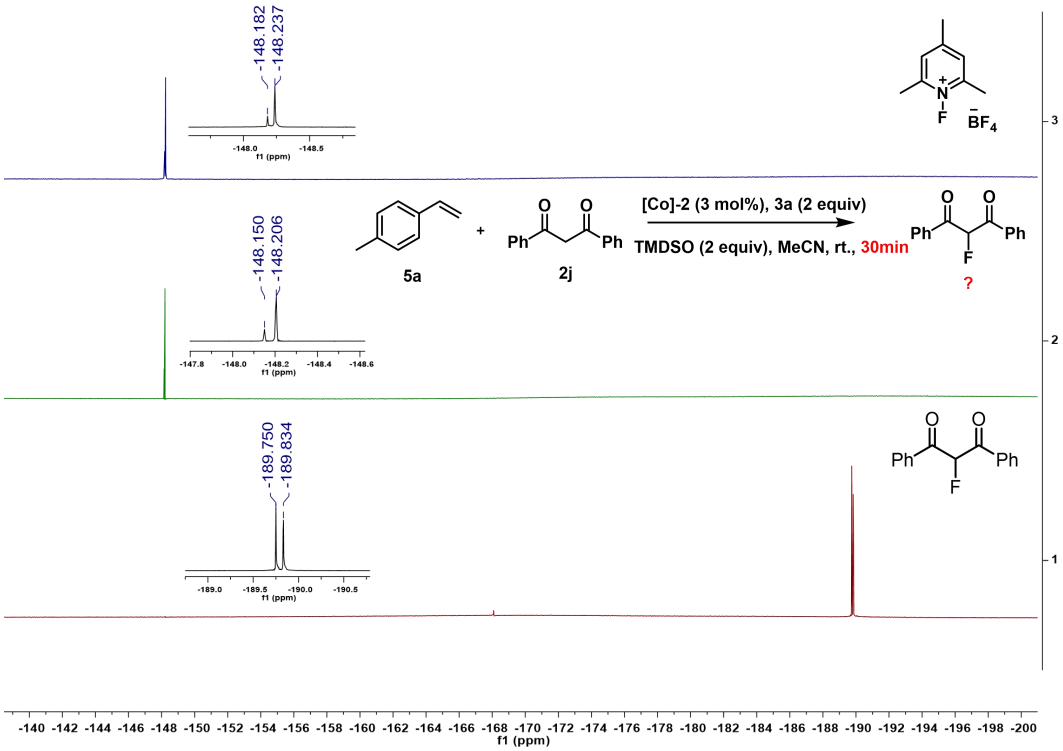
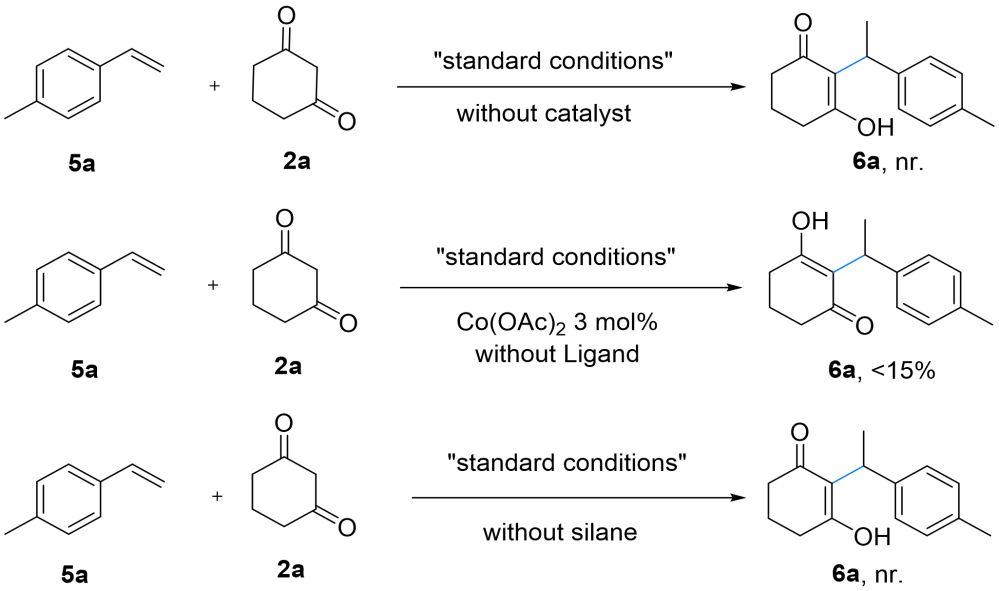


Fig. S11 **^{19}F NMR** data analysis

h) Control experiments



Control experiments showed that no product was formed in the absence of catalysts and silane. The reaction in the absence of ligand also provided the desired product in very low yields (<15% yield).

VI. Reference

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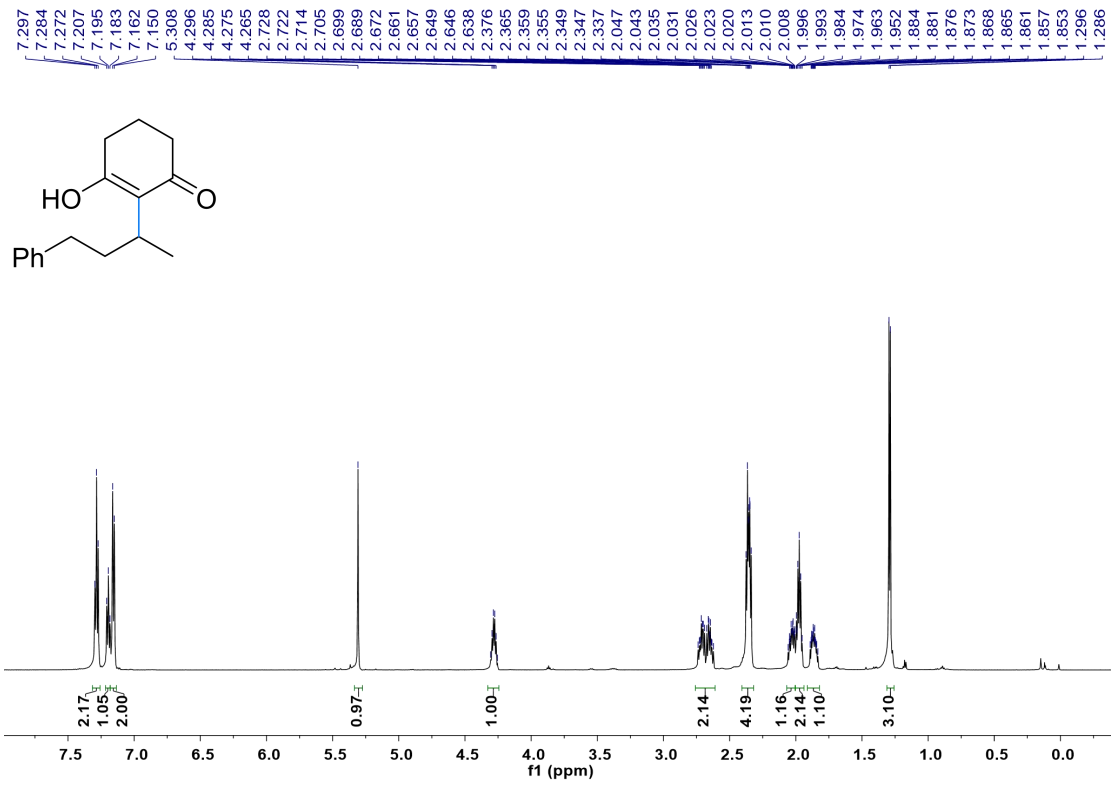
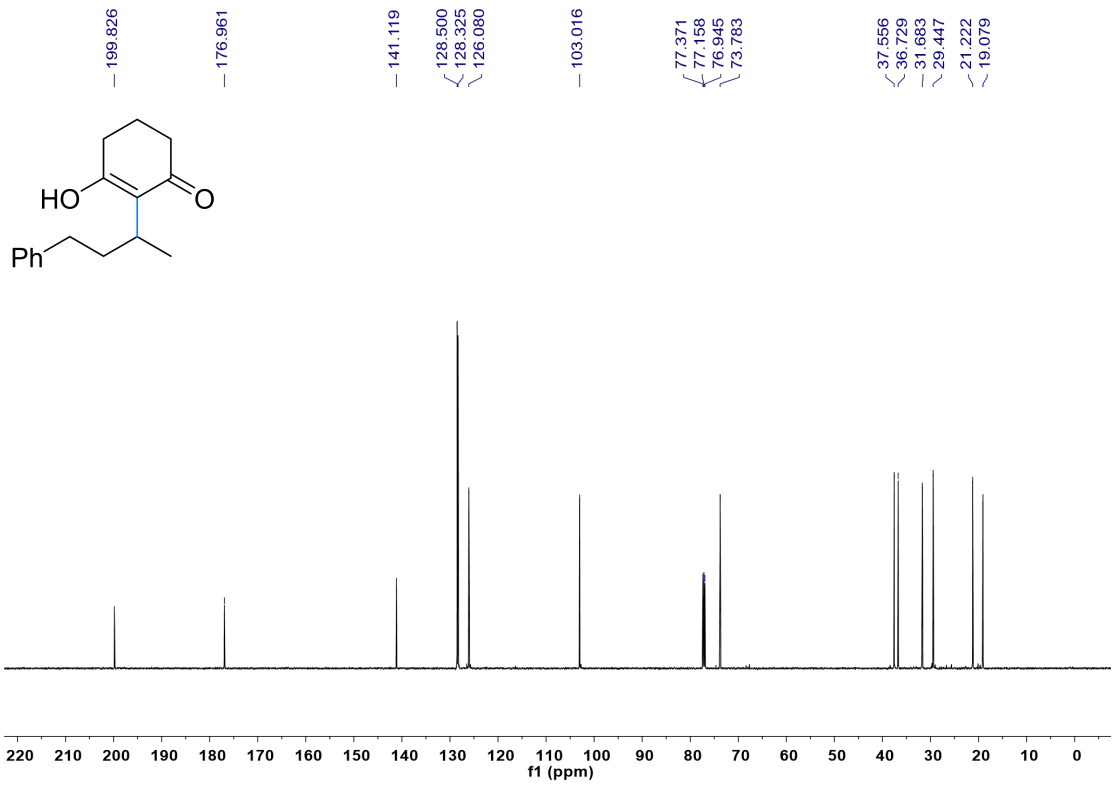
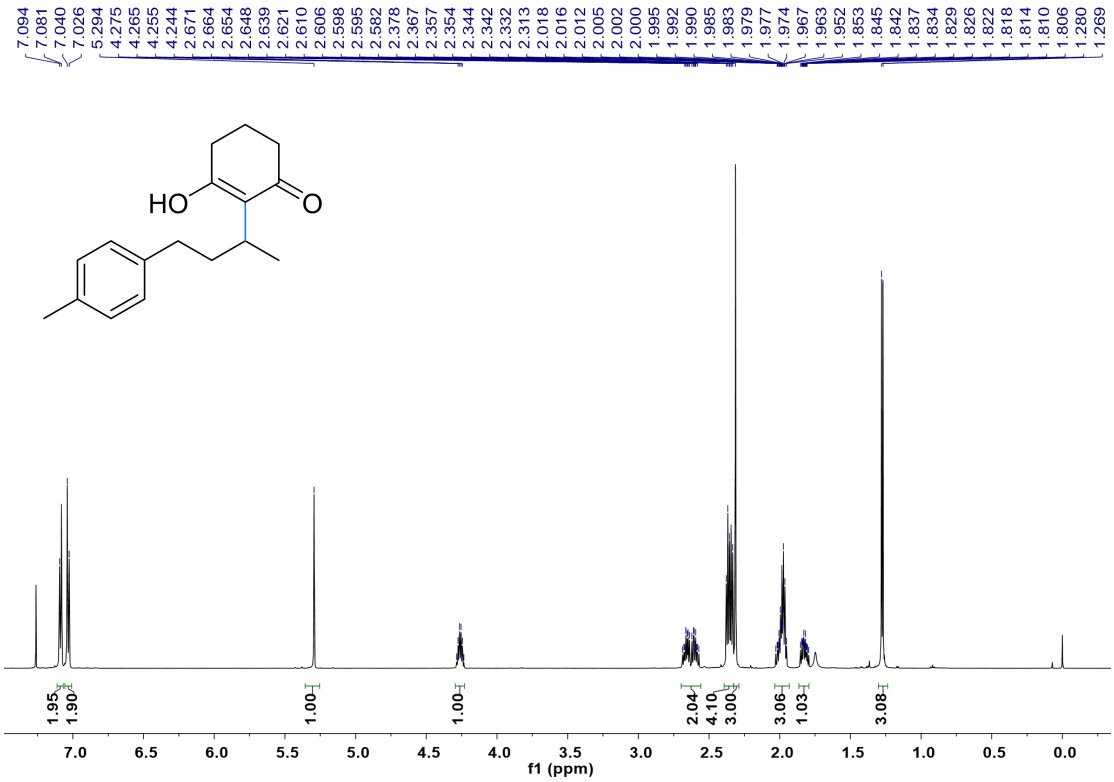
VII. ^{1}H, ^{13}C, ^{19}F Spectra of New Compounds



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7.026
5.294
4.275
4.265
4.255
4.244
2.671
2.664
2.654
2.648
2.639
2.621
2.610
2.606
2.598
2.595
2.582
2.378
2.367
2.357
2.354
2.344
2.342
2.332
2.313
2.018
2.016
2.012
2.005
2.002
2.000
1.995
1.992
1.990
1.985
1.983
1.979
1.977
1.974
1.967
1.963
1.952
1.853
1.845
1.842
1.837
1.834
1.829
1.826
1.822
1.818
1.814
1.810
1.806
1.280
1.269
HO
O
1.95
1.90
1.00
1.00
2.04
4.10
3.00
3.06
1.03
3.08
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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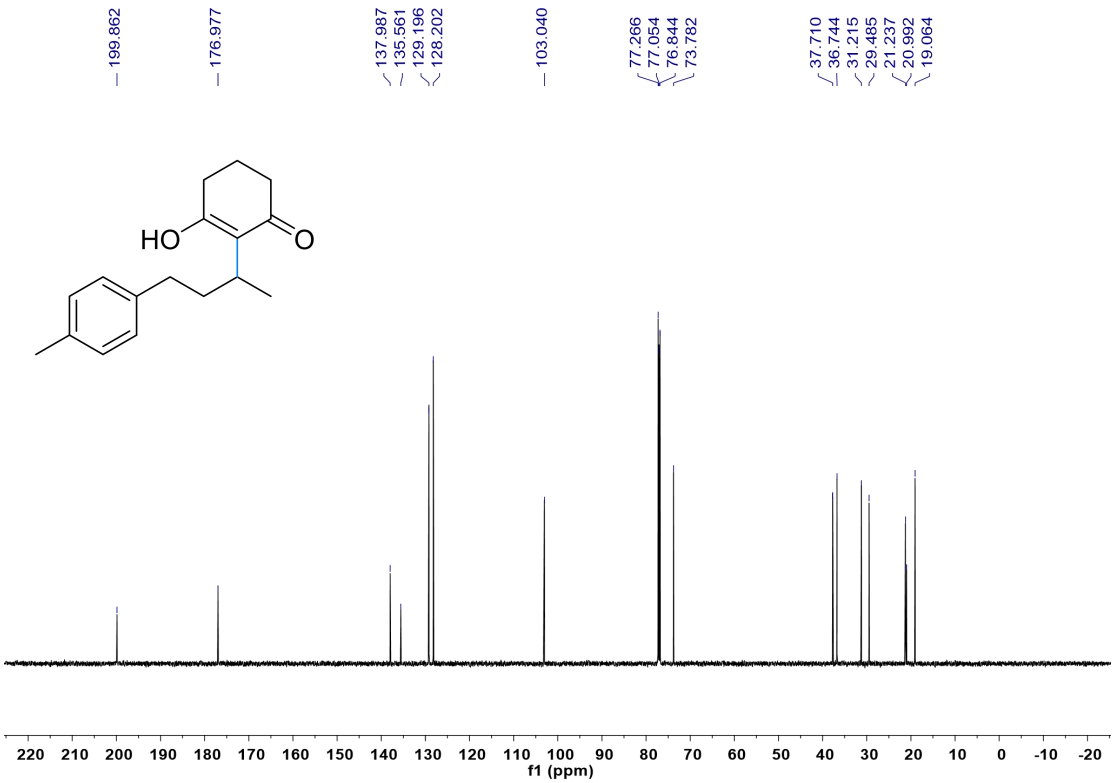
1.0
0.5
0.0
f1 (ppm)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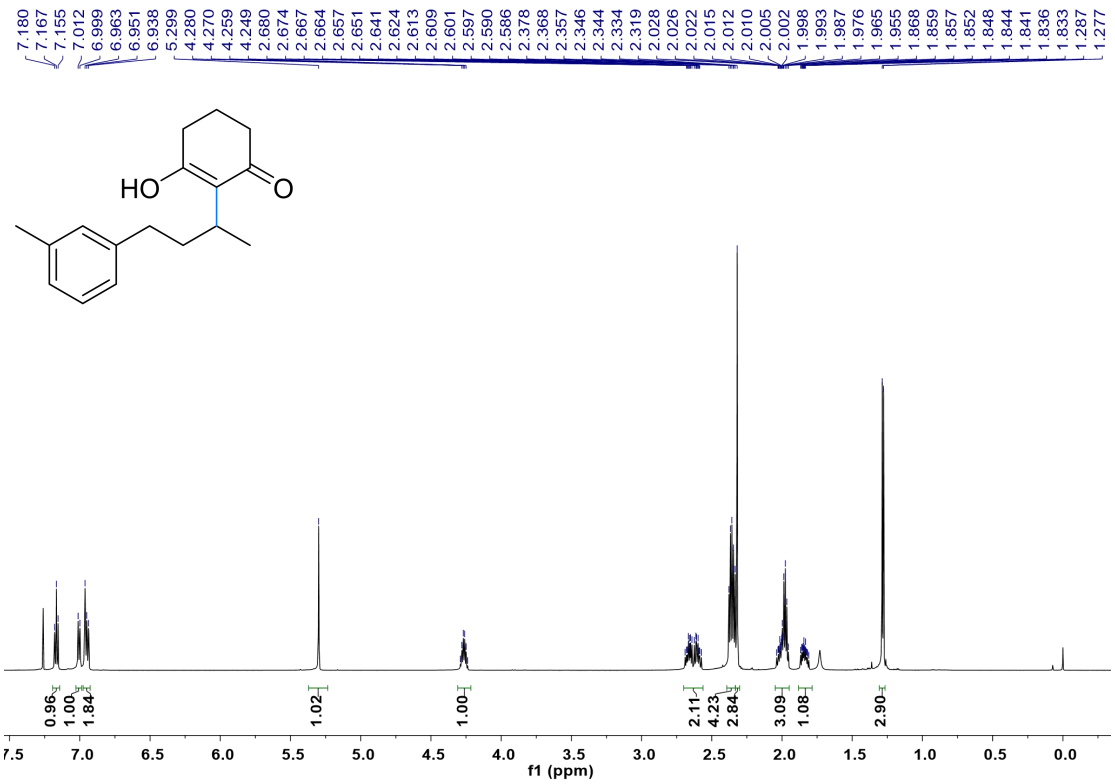
129.196
128.202
103.040
77.266
77.054
76.844
73.782
37.710
36.744
31.215
29.485
21.237
20.992
19.064
HO
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)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4.249
2.680
2.674
2.667
2.664
2.657
2.651
2.641
2.624
2.613
2.609
2.601
2.597
2.590
2.586
2.378
2.368
2.357
2.346
2.344
2.334
2.319
2.028
2.026
2.022
2.015
2.012
2.010
2.005
2.002
1.998
1.993
1.987
1.976
1.965
1.955
1.868
1.859
1.857
1.852
1.848
1.844
1.841
1.836
1.833
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1.277
HO
O
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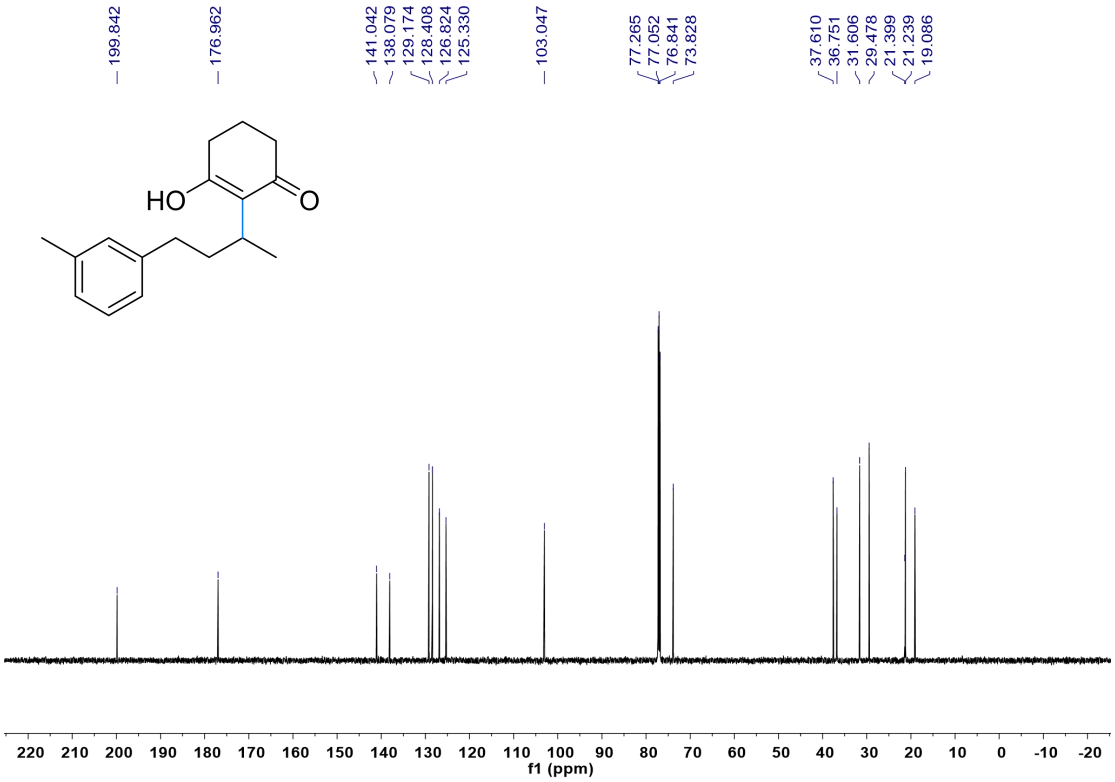
f1 (ppm)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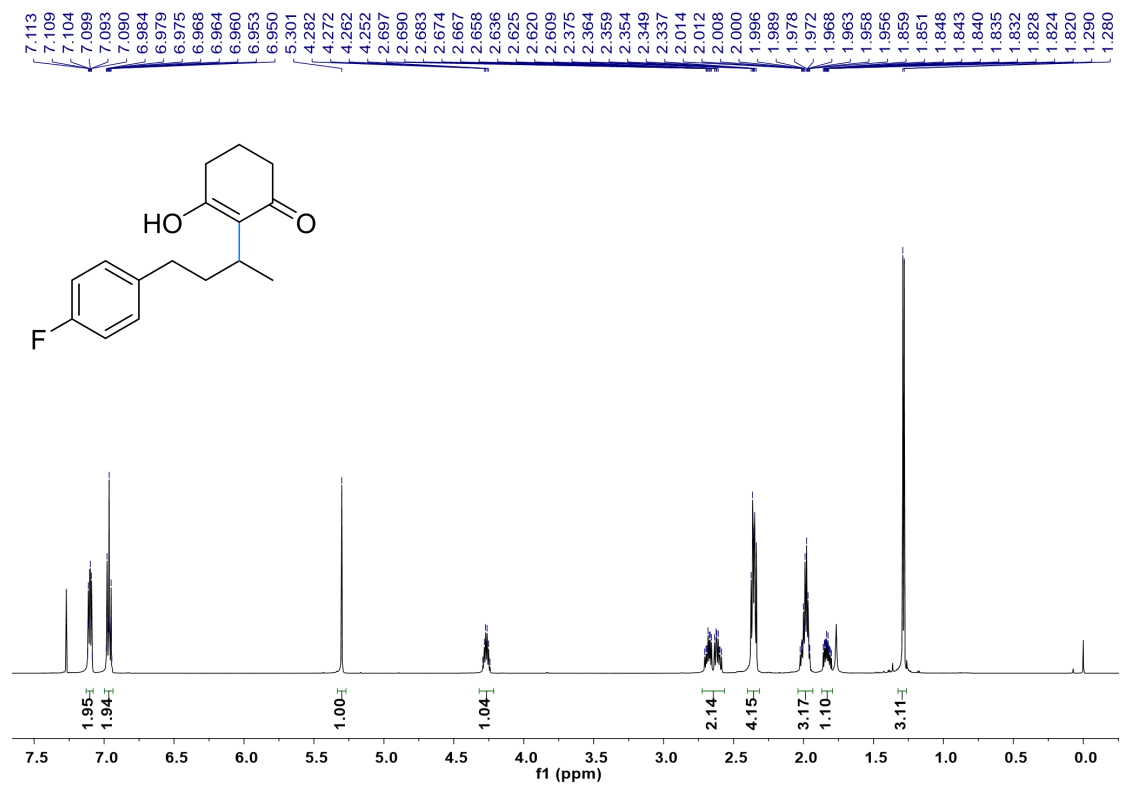
HO
O
f1 (ppm)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6.960
6.953
6.950
5.301
4.282
4.272
4.262
4.252
2.697
2.690
2.683
2.674
2.667
2.658
2.636
2.625
2.620
2.609
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1.963
1.958
1.956
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1.828
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1.280
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2.5
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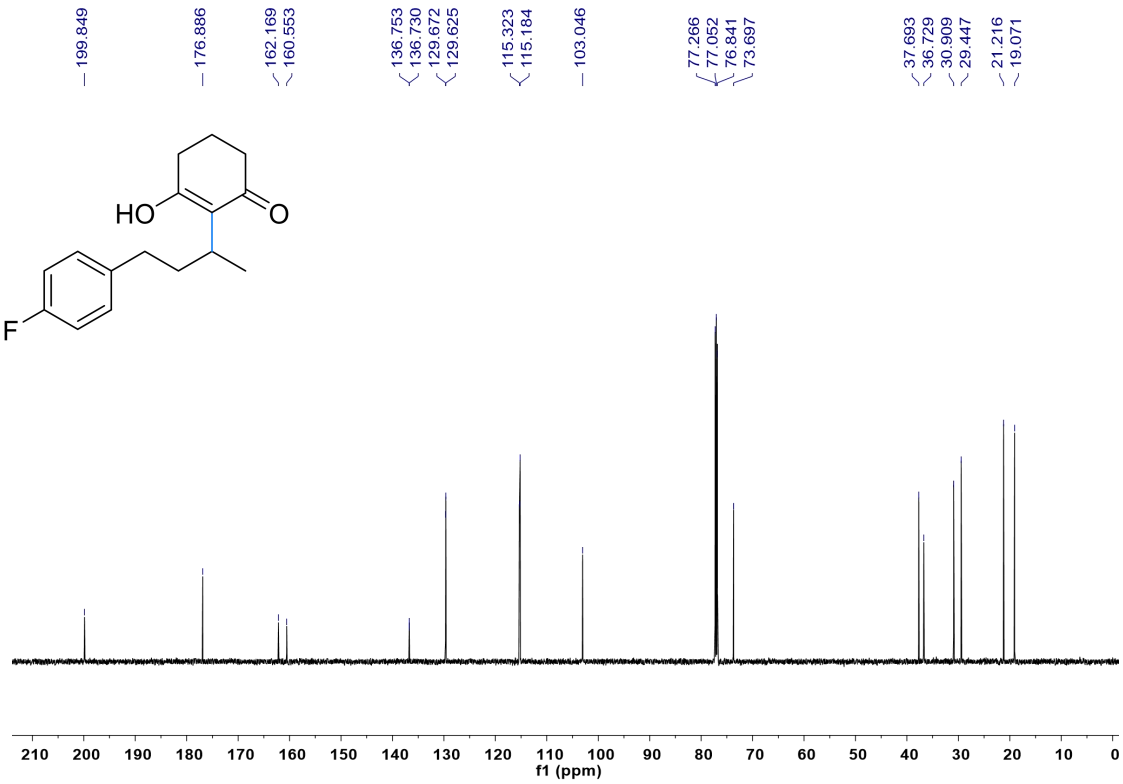
0.0
f1 (ppm)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77.052
76.841
73.697
37.693
36.729
30.909
29.447
21.216
19.071
HO
O
F
210
200
190
180
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150
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130
120
110
100
90
80
70
60
50
40
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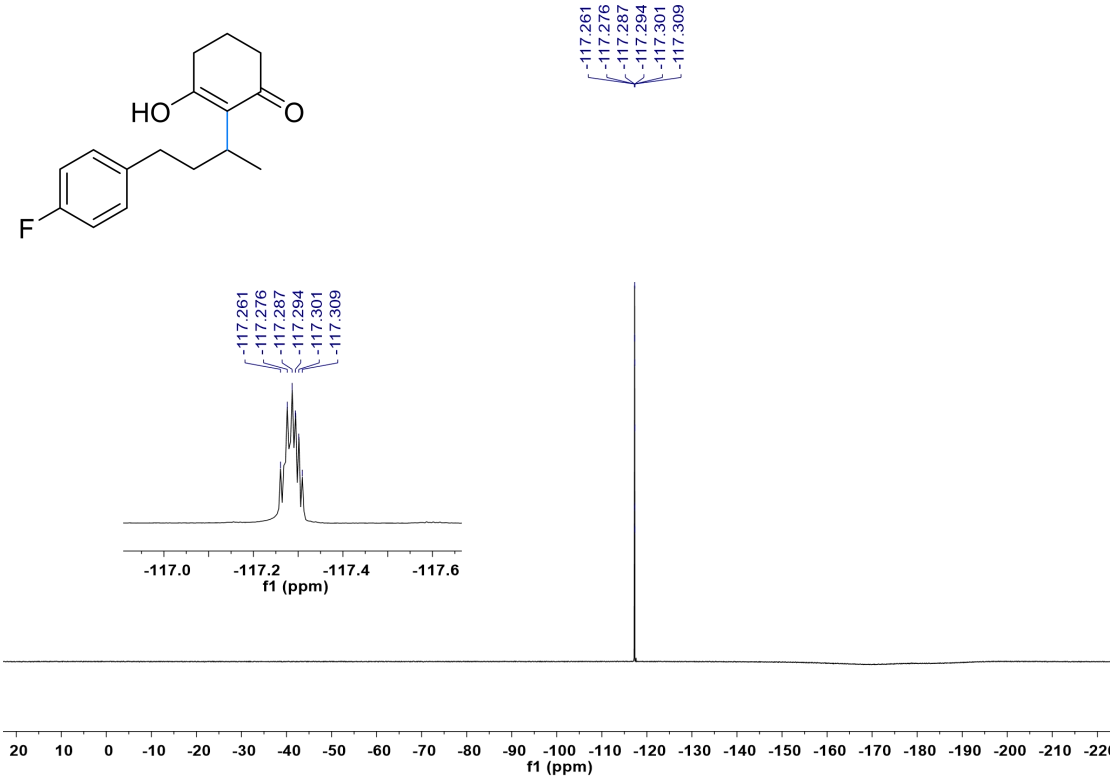
10
0
f1 (ppm)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f1 (ppm)
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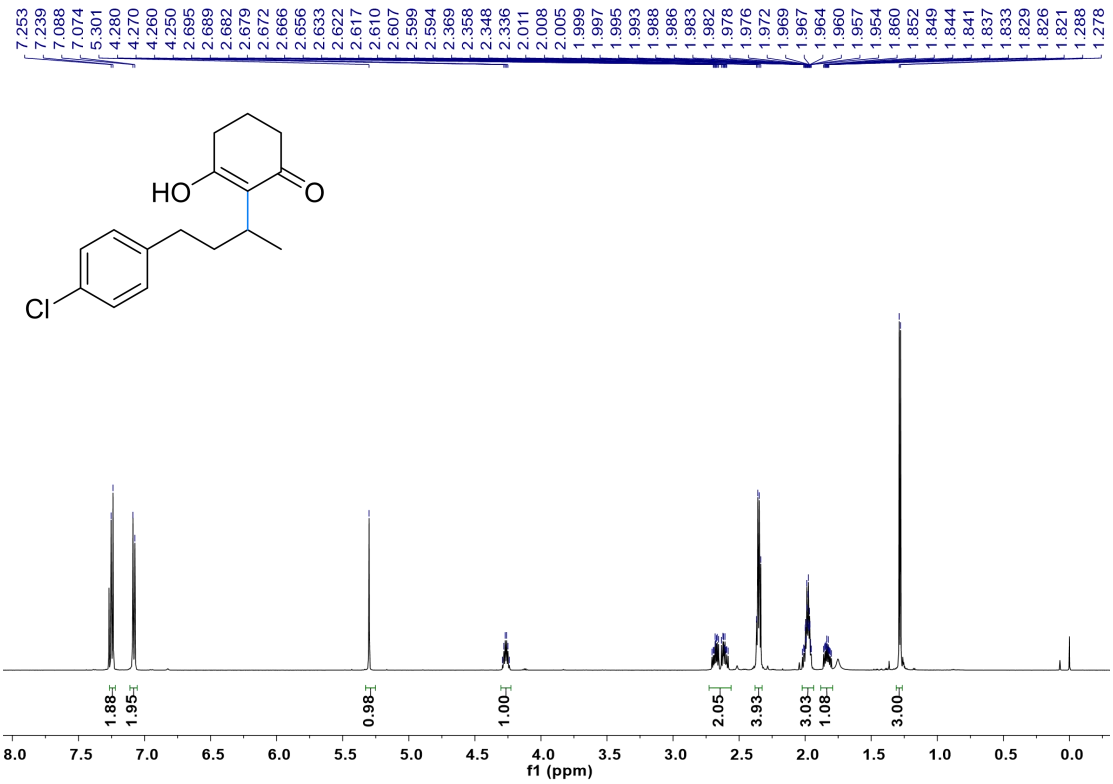
f1 (ppm)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2.666
2.656
2.633
2.622
2.617
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2.607
2.599
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HO
O
Cl
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0.98
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2.05
3.93
3.03
1.08
3.00
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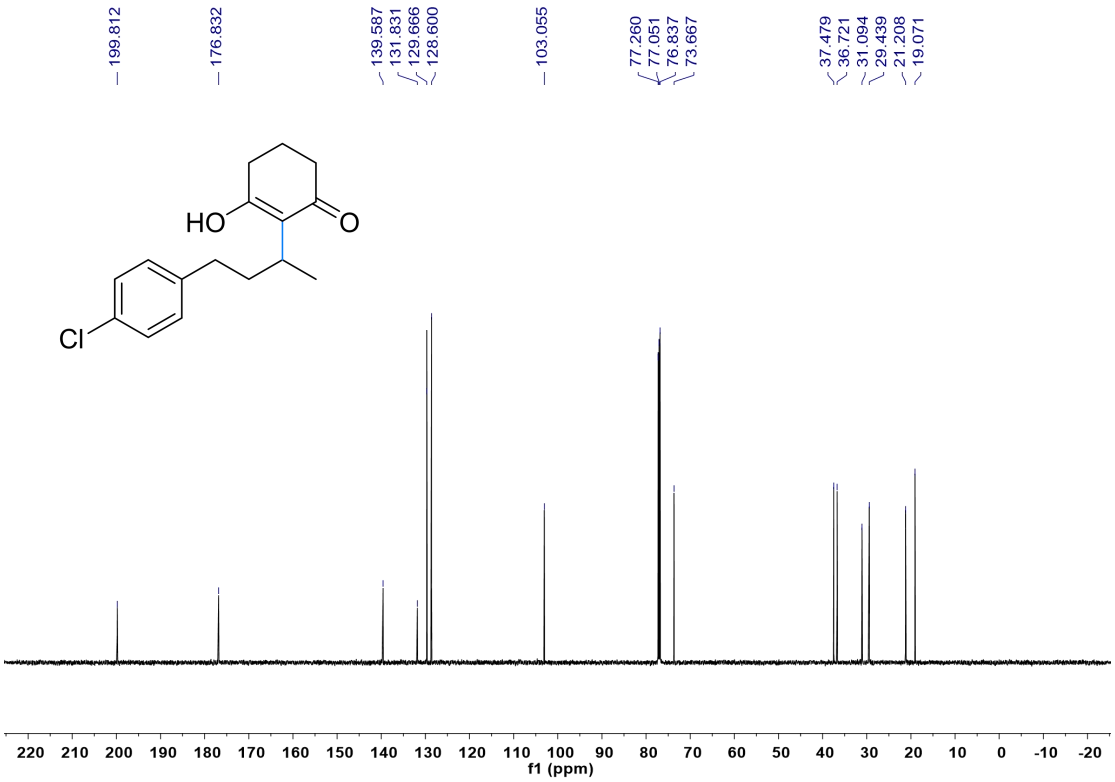
f1 (ppm)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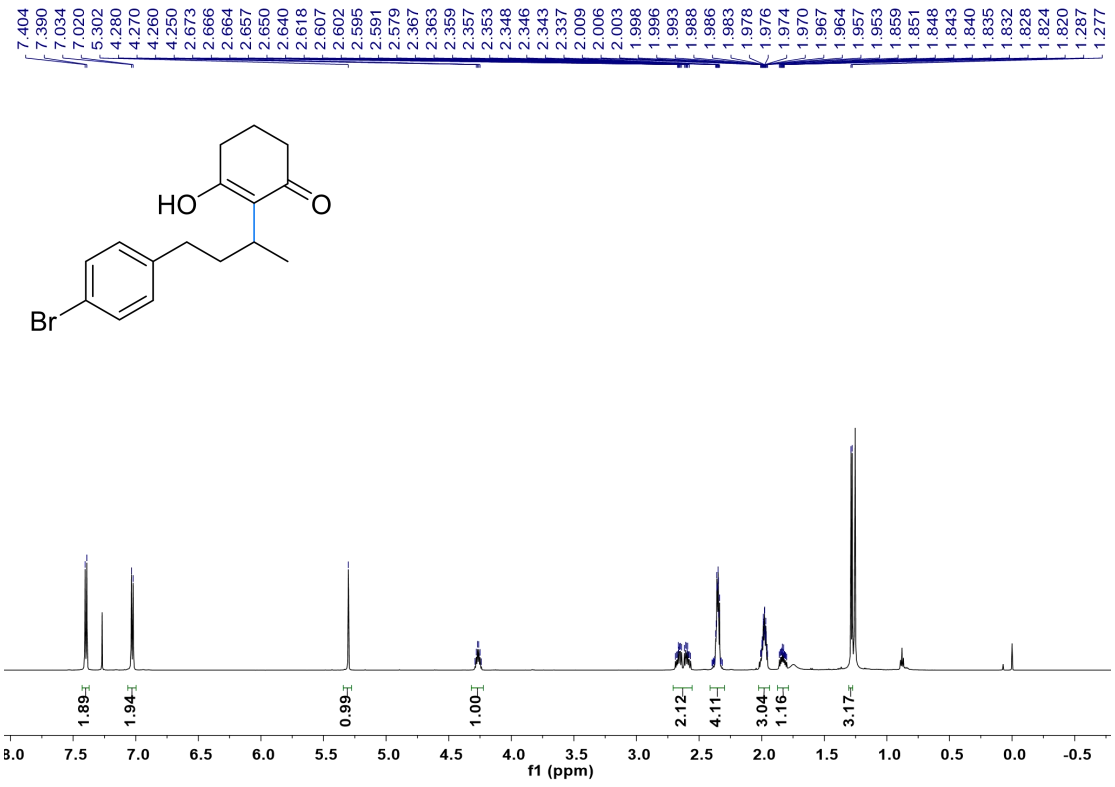
HO
O
Cl
f1 (ppm)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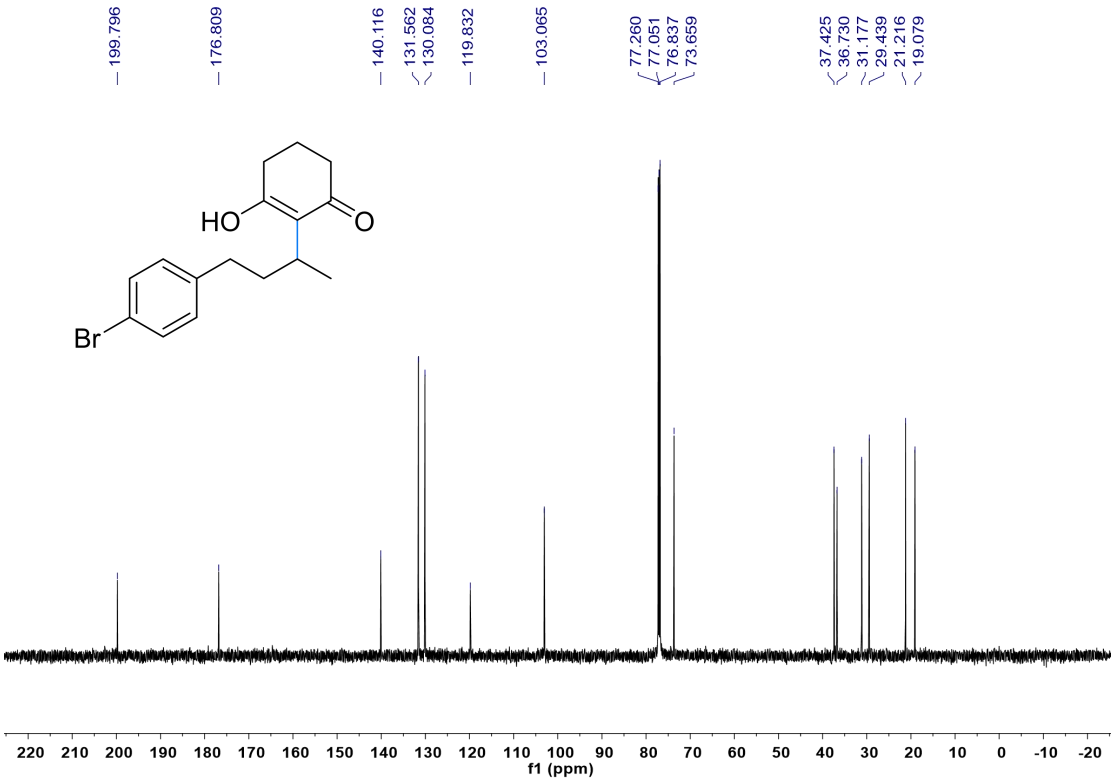
f1 (ppm)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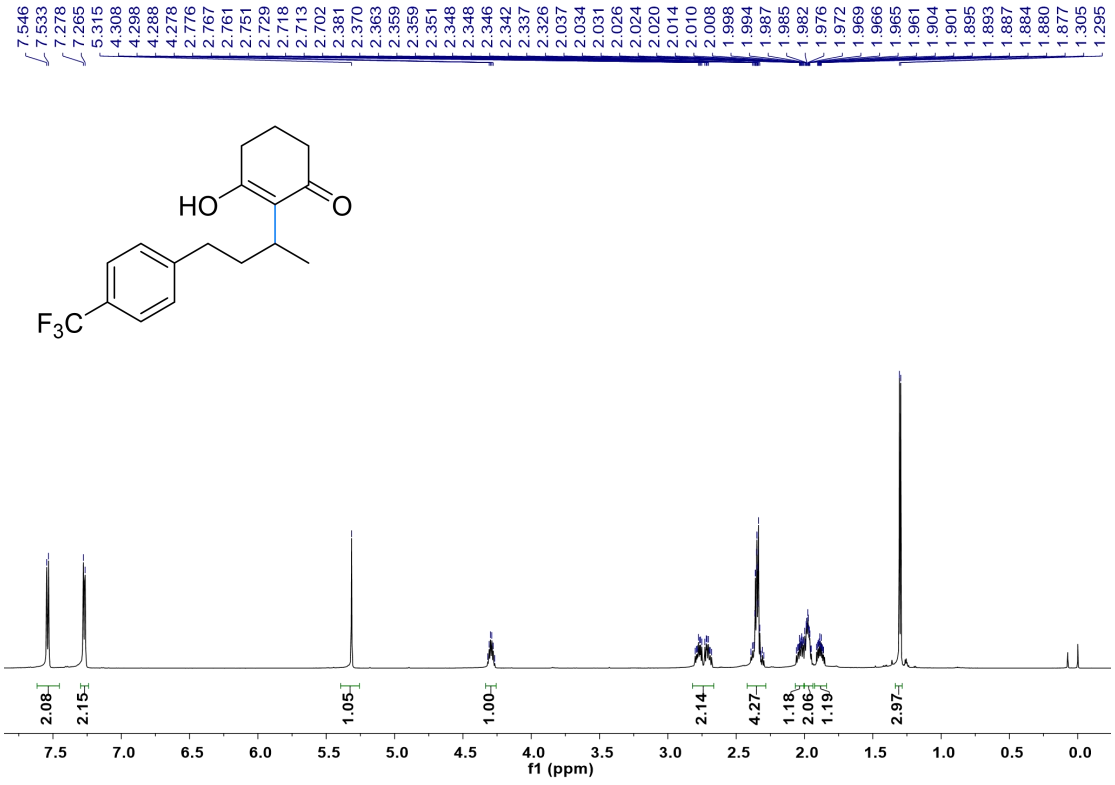
Br
f1 (ppm)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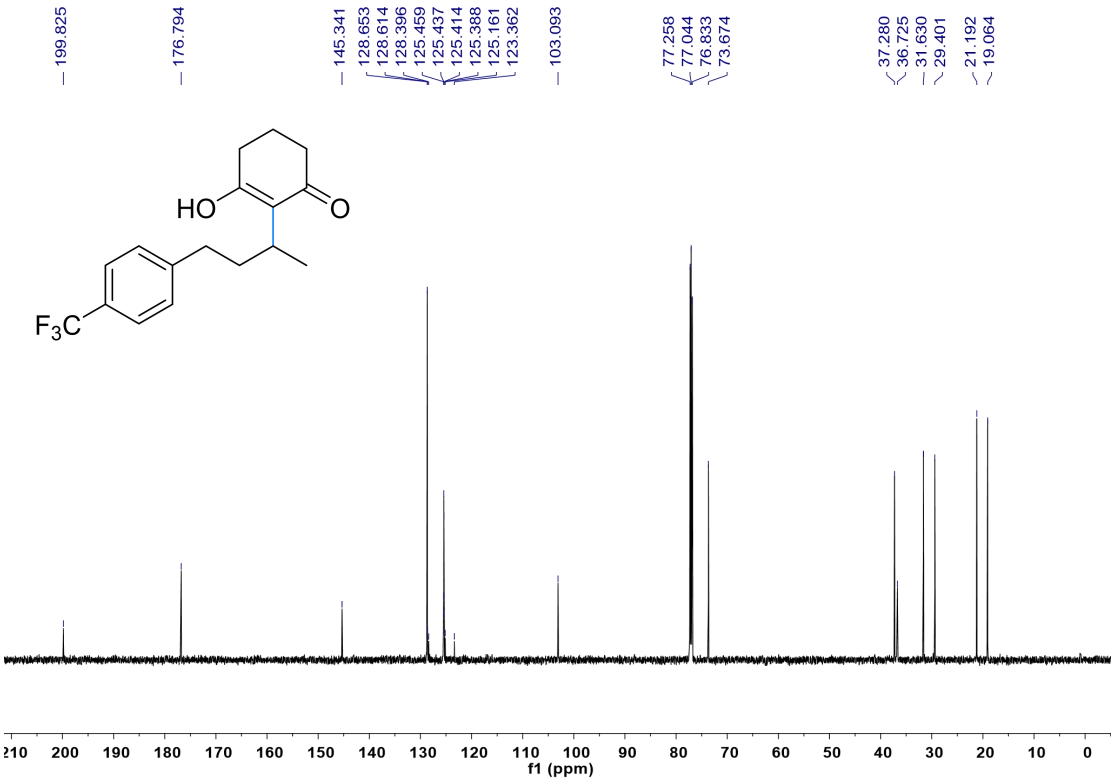
f1 (ppm)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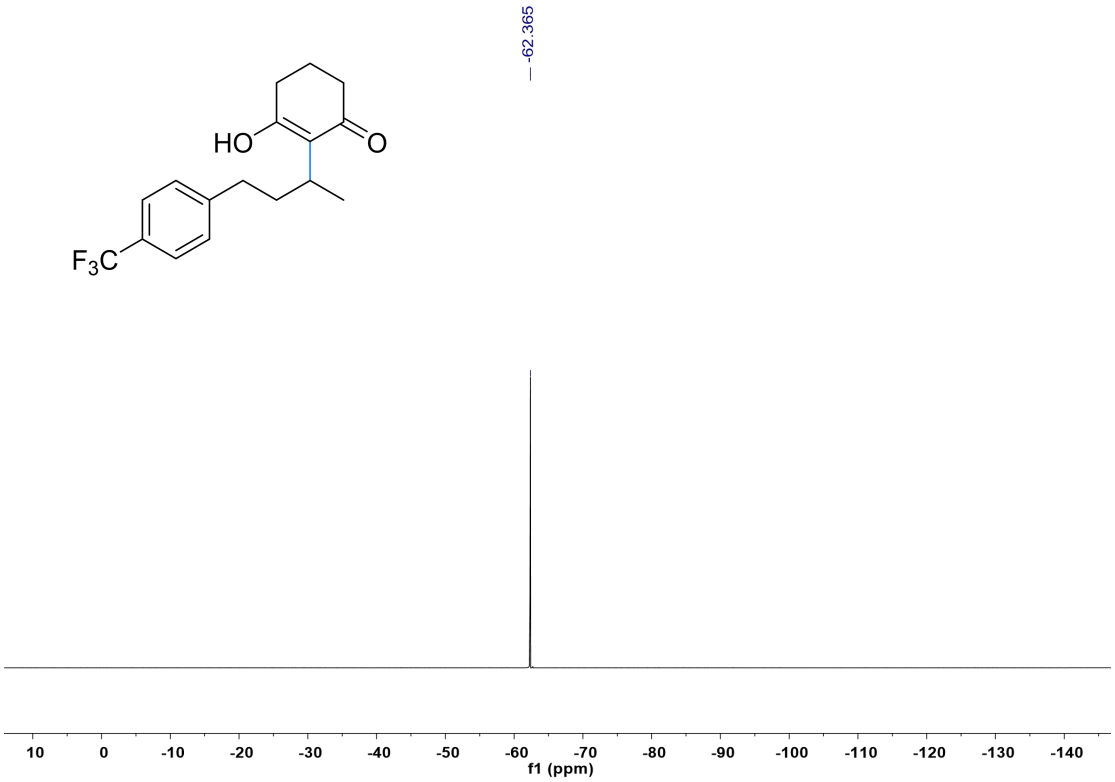
O
F3C
f1 (ppm)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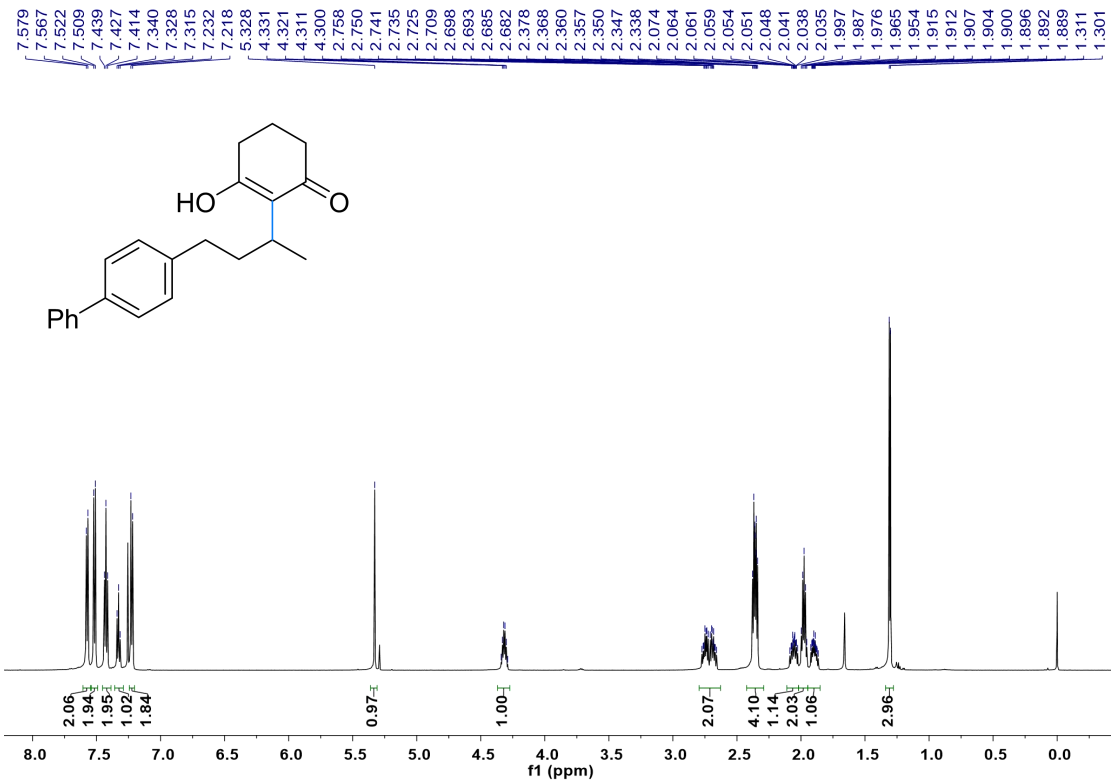
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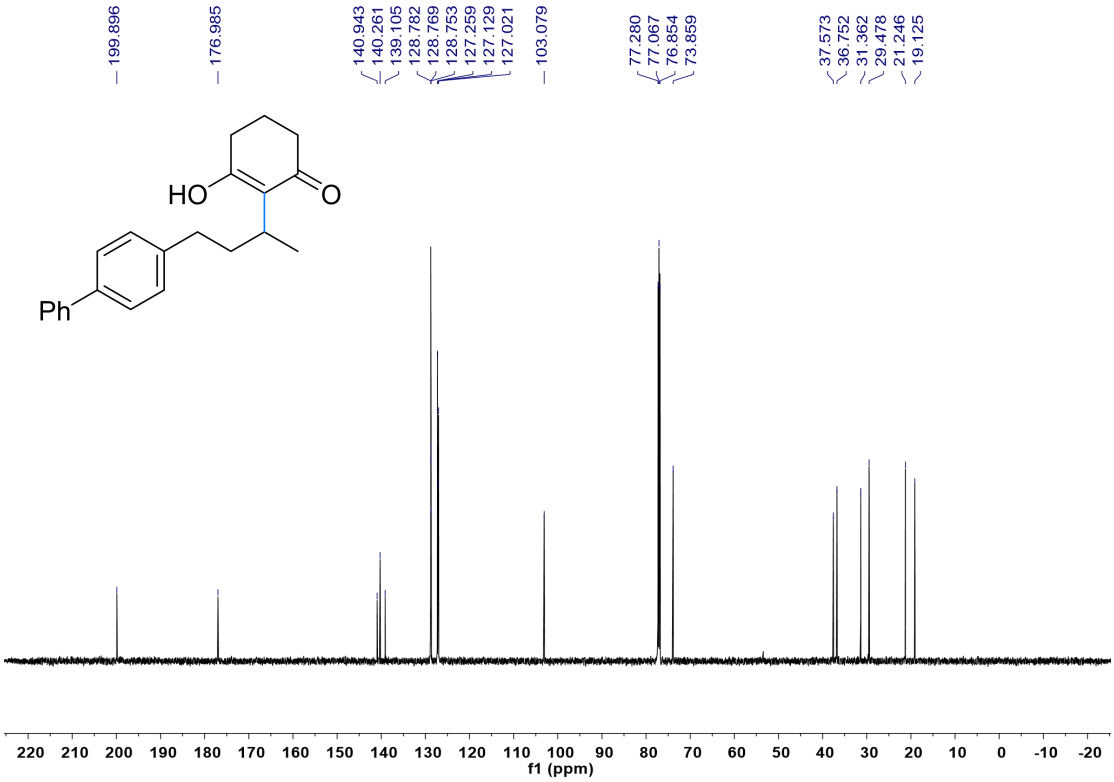
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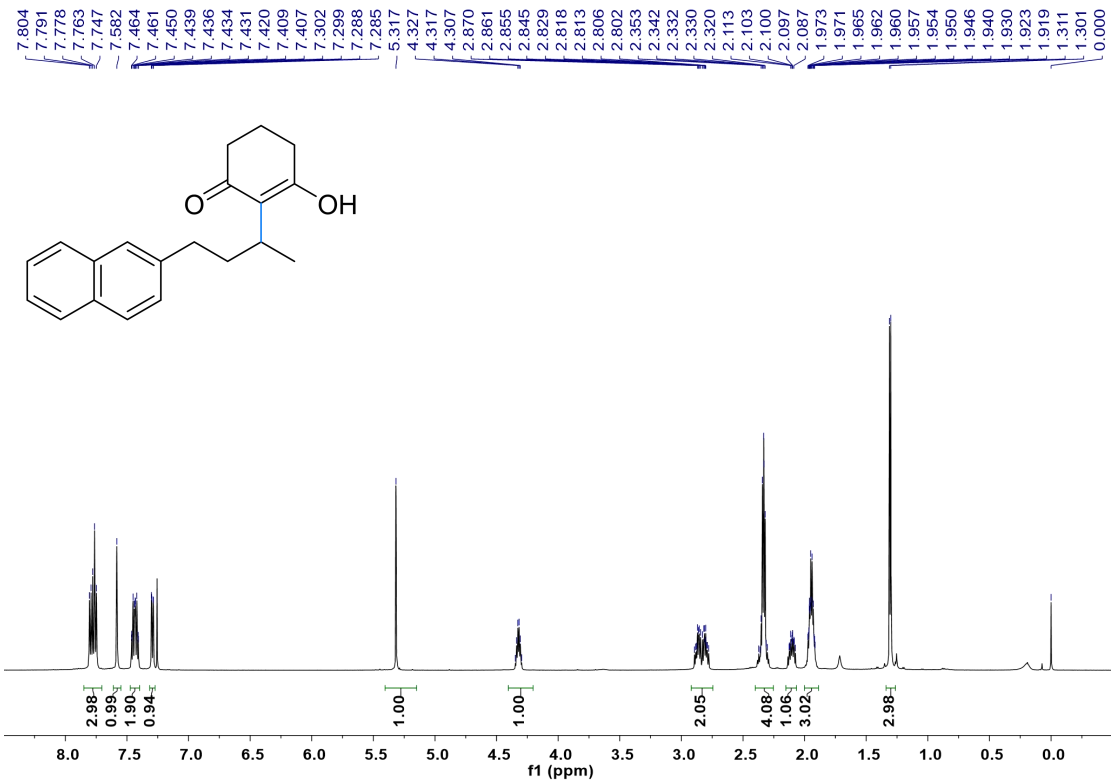
HO
O
Ph
f1 (ppm)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1.962
1.960
1.957
1.954
1.950
1.946
1.940
1.930
1.923
1.919
1.311
1.301
0.000
O
OH
2.98
0.99
1.90
0.94
1.00
1.00
2.05
4.08
1.06
3.02
2.98
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
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1.0
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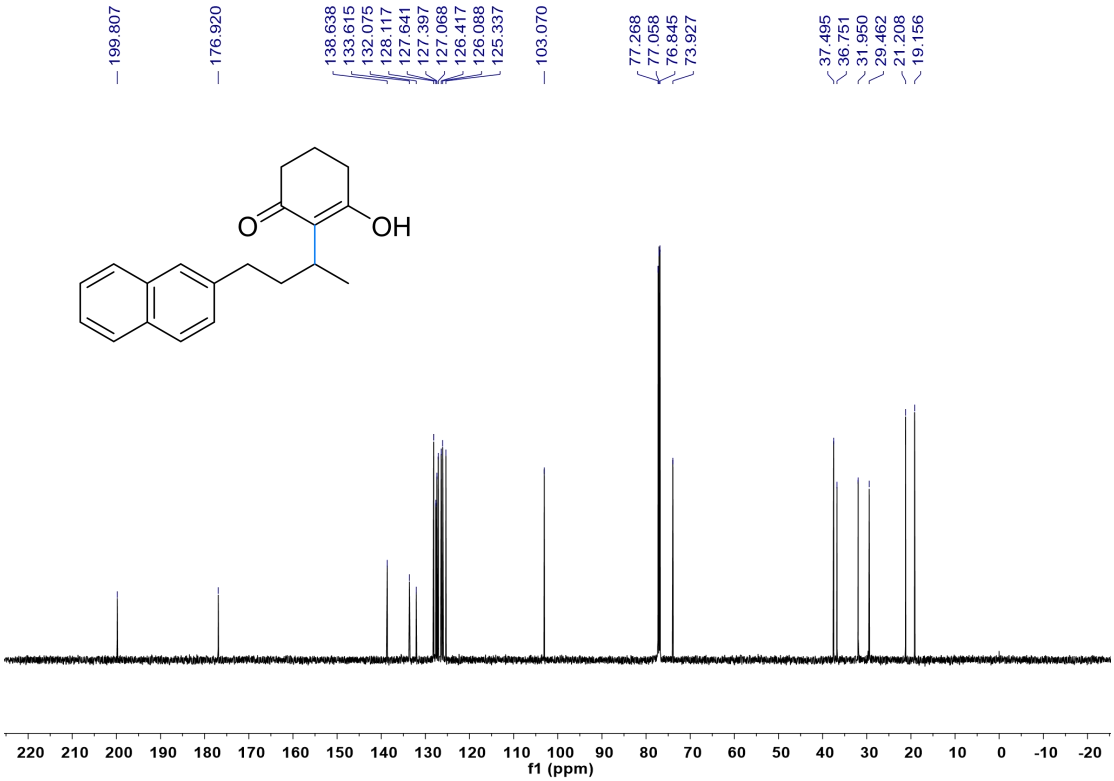
0.0
f1 (ppm)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77.268
77.058
76.845
73.927
37.495
36.751
31.950
29.462
21.208
19.156
O
OH
220
210
200
190
180
170
160
150
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130
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100
90
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60
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40
30
20
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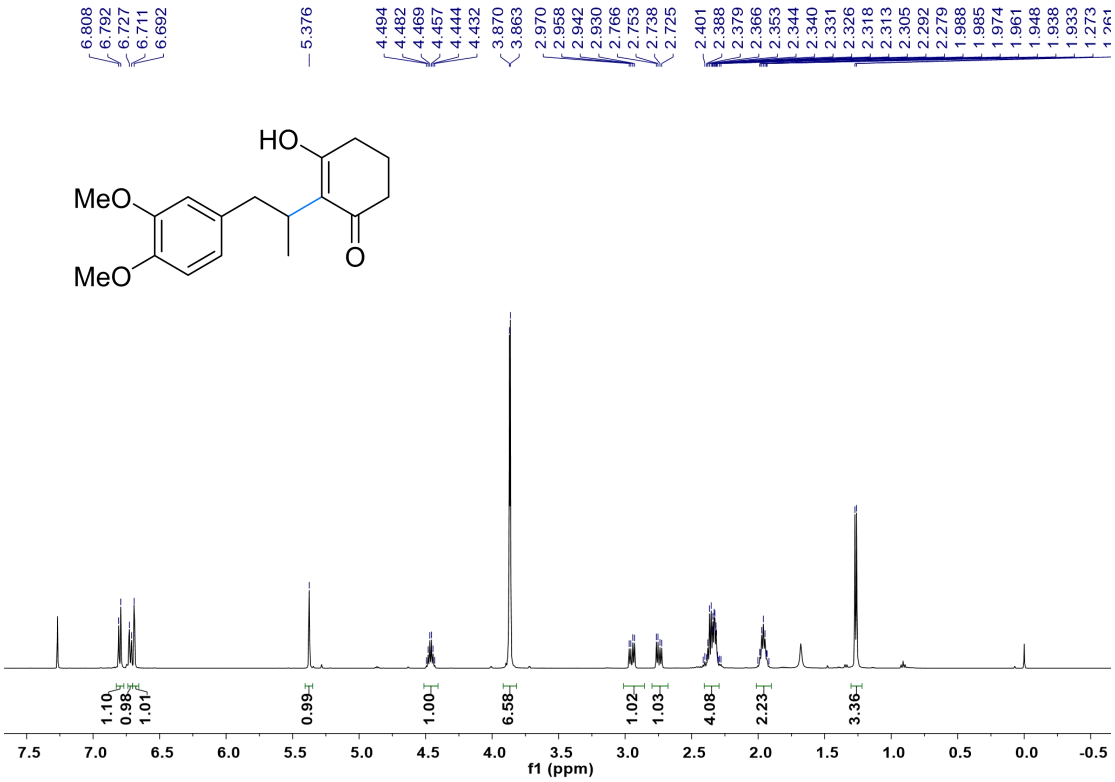
-10
-20
f1 (ppm)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3.863
2.970
2.958
2.942
2.930
2.766
2.753
2.738
2.725
2.401
2.388
2.379
2.366
2.353
2.344
2.340
2.331
2.326
2.318
2.313
2.305
2.292
2.279
1.988
1.985
1.974
1.961
1.948
1.938
1.933
1.273
HO
MeO
MeO
O
1.10
0.98
1.01
0.99
1.00
6.58
1.02
1.03
4.08
2.23
3.36
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.0
2.5
2.0
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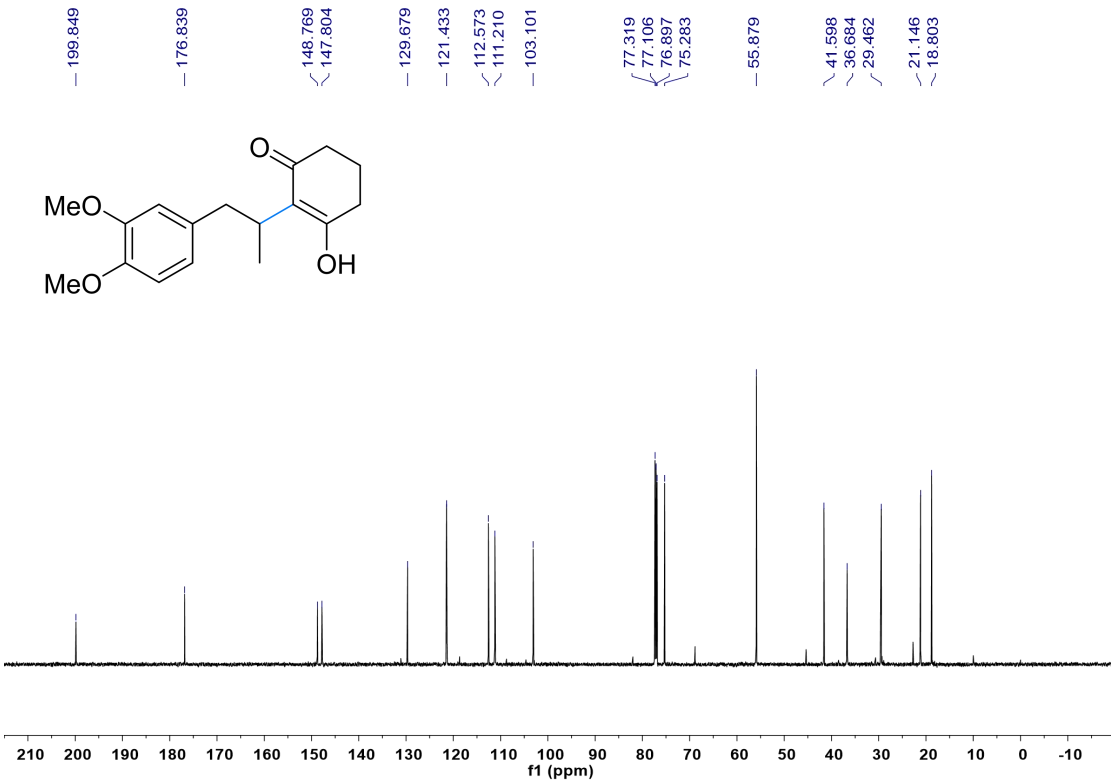
0.5
0.0
-0.5
f1 (ppm)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55.879
41.598
36.684
29.462
21.146
18.803
O
MeO
MeO
OH
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200
190
180
170
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100
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80
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40
30
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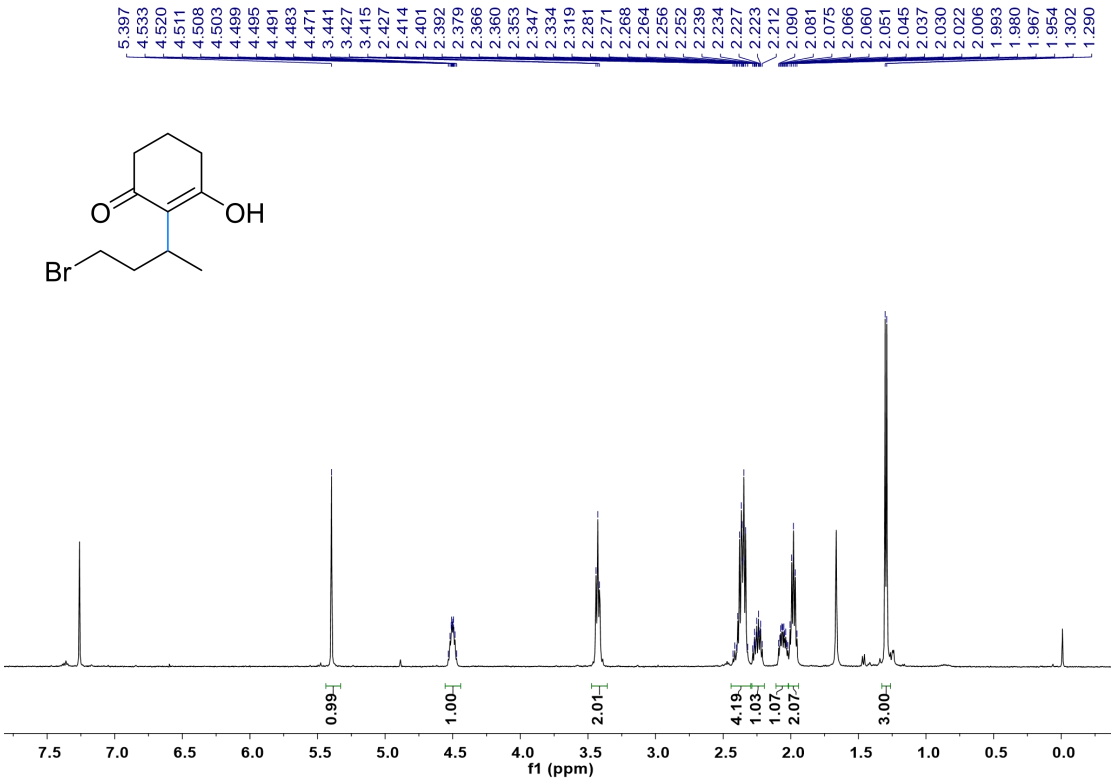
-10
f1 (ppm)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4.503
4.499
4.495
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4.483
4.471
3.441
3.427
3.415
2.427
2.414
2.401
2.392
2.379
2.366
2.360
2.353
2.347
2.334
2.319
2.281
2.271
2.268
2.264
2.256
2.252
2.239
2.234
2.227
2.223
2.212
2.090
2.081
2.075
2.066
2.060
2.051
2.045
2.037
2.030
2.022
2.006
1.993
1.980
1.967
1.954
1.302
1.290
O
OH
Br
0.99
1.00
2.01
4.19
1.03
1.07
2.07
3.00
7.5
7.0
6.5
6.0
5.5
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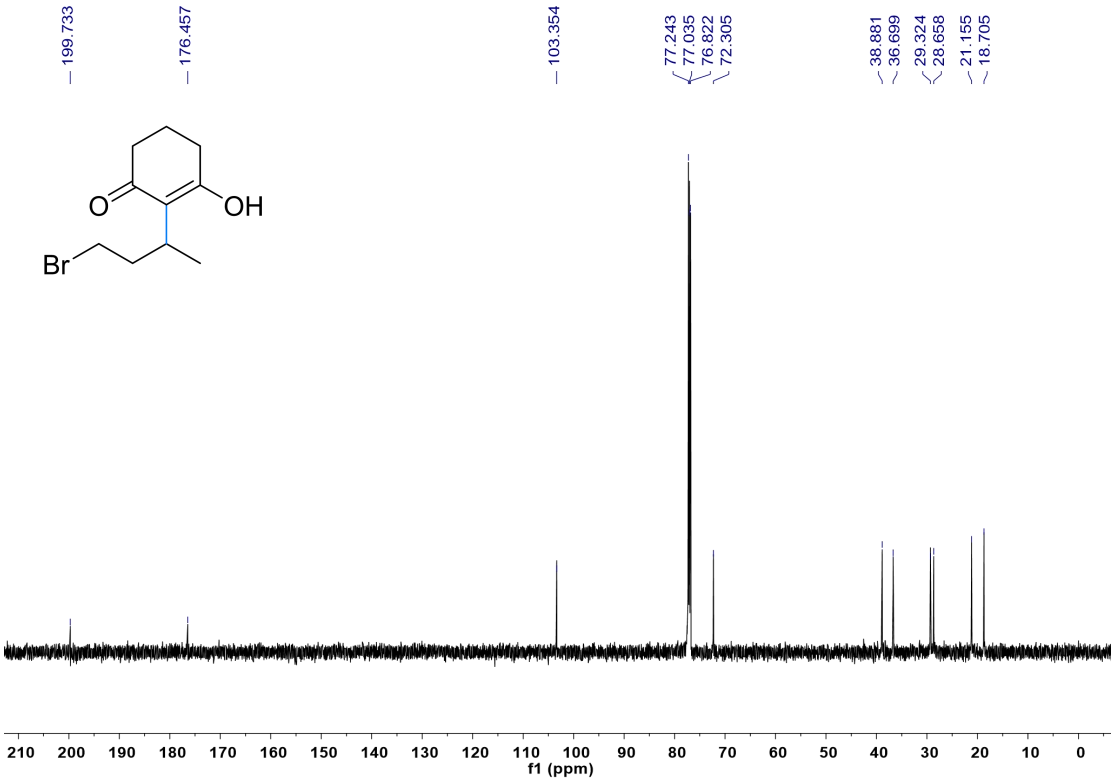
0.0
f1 (ppm)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18.705
O
OH
Br
210
200
190
180
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130
120
110
100
90
80
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60
50
40
30
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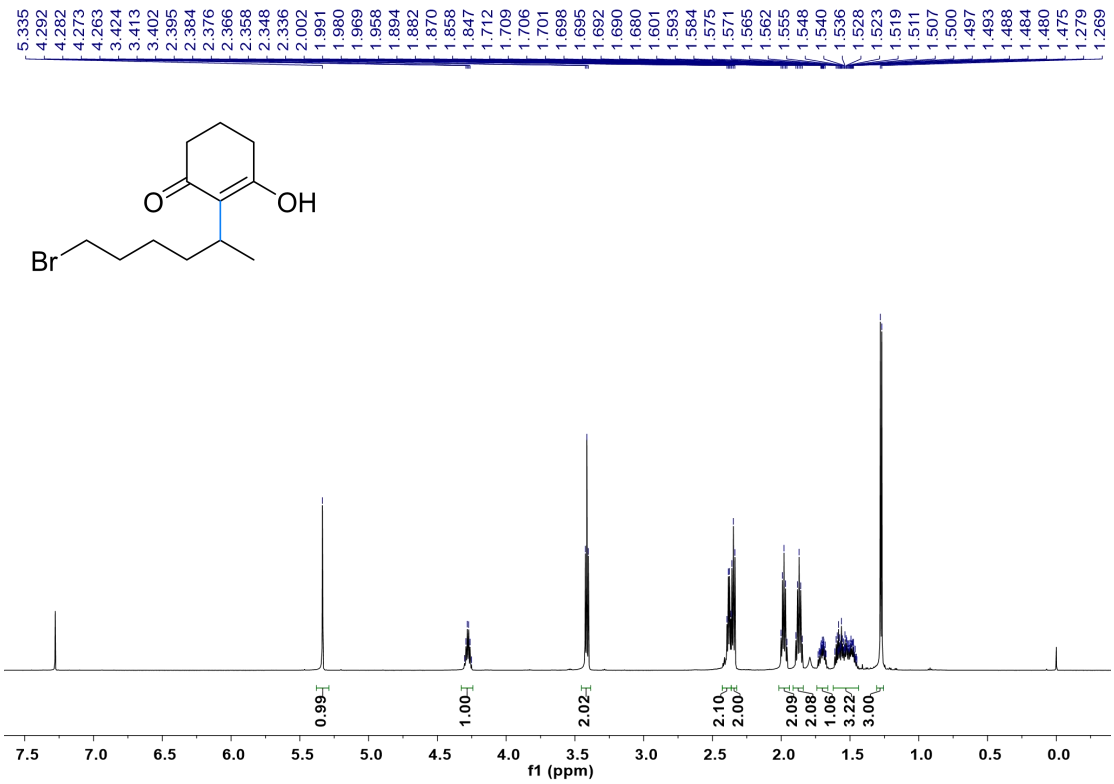
10
0
f1 (ppm)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2.366
2.358
2.348
2.336
2.002
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1.980
1.969
1.958
1.894
1.882
1.870
1.858
1.847
1.712
1.709
1.706
1.701
1.698
1.695
1.692
1.690
1.680
1.601
1.593
1.584
1.575
1.571
1.565
1.562
1.555
1.548
1.540
1.536
1.528
1.523
1.519
1.511
1.507
1.500
1.497
1.493
1.488
1.484
1.480
1.475
1.279
1.269
O
OH
Br
0.99
1.00
2.02
2.10
2.00
2.09
2.08
1.06
3.22
3.00
7.5
7.0
6.5
6.0
5.5
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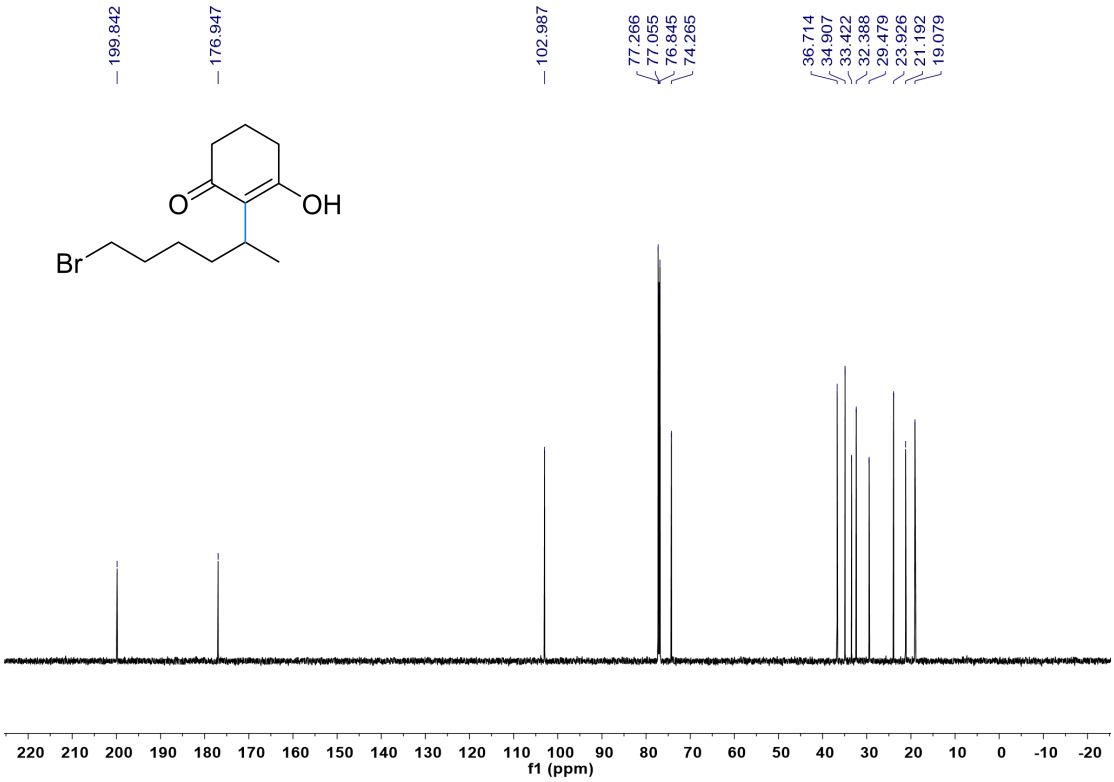
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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29.479
23.926
21.192
19.079
O
OH
Br
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
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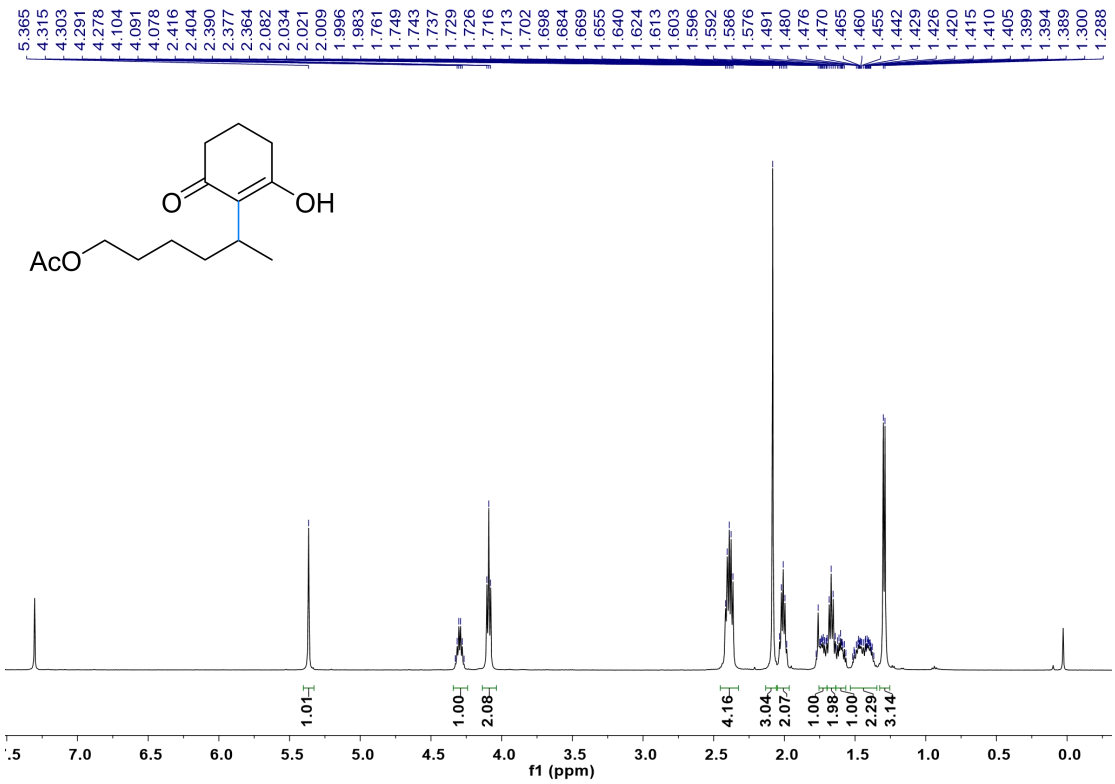
-10
-20
f1 (ppm)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2.377
2.364
2.082
2.034
2.021
2.009
1.996
1.983
1.761
1.749
1.743
1.737
1.729
1.726
1.716
1.713
1.702
1.698
1.684
1.669
1.655
1.640
1.624
1.613
1.603
1.596
1.592
1.586
1.576
1.491
1.480
1.476
1.470
1.465
1.460
1.455
1.442
1.429
1.426
1.420
1.415
1.410
1.405
1.399
1.394
1.389
1.300
1.288
O
OH
AcO
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4.16
3.04
2.07
1.00
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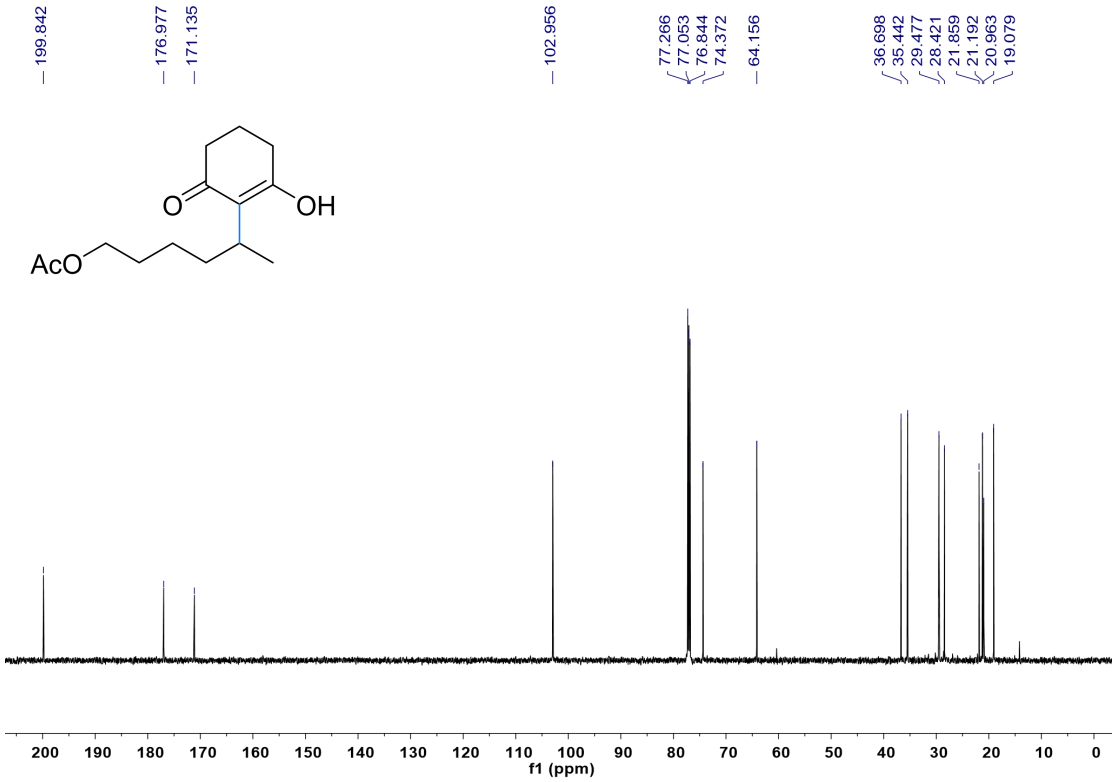
1.00
2.29
3.14
f1 (ppm)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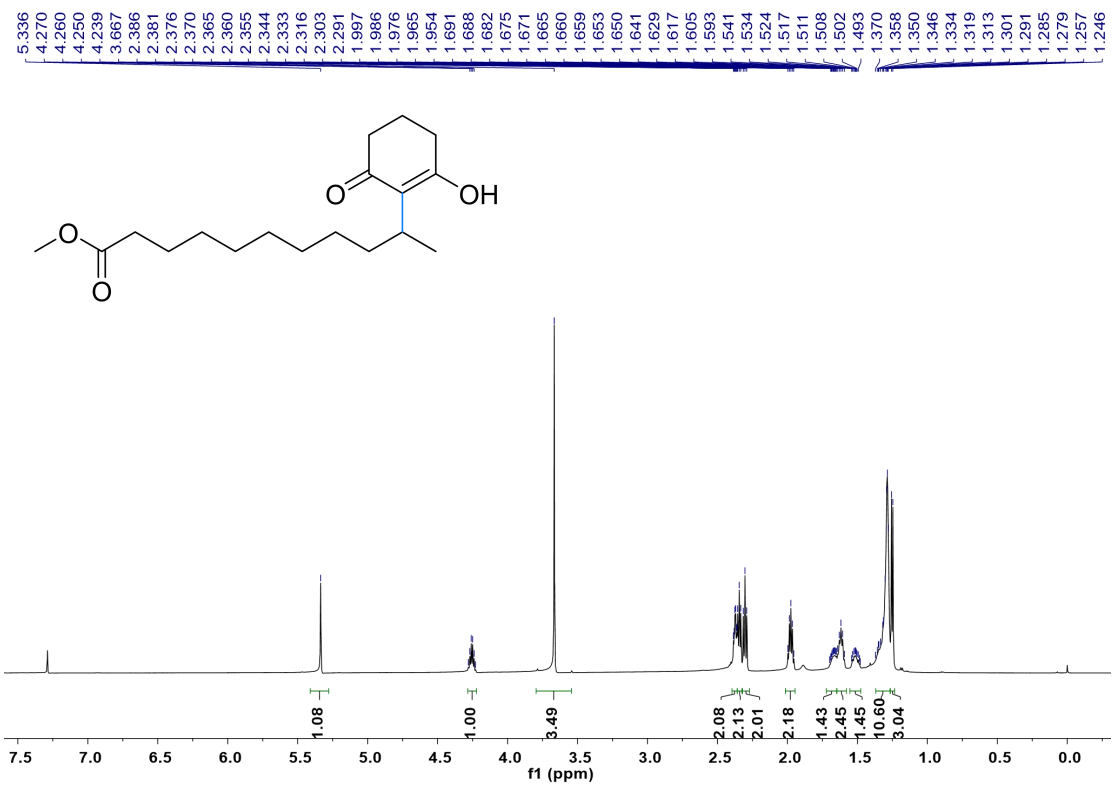
21.859
21.192
20.963
19.079
O
OH
AcO
f1 (ppm)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2.360
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2.344
2.333
2.316
2.303
2.291
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1.986
1.976
1.965
1.954
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1.688
1.682
1.675
1.671
1.665
1.660
1.659
1.653
1.650
1.641
1.629
1.617
1.605
1.593
1.541
1.534
1.524
1.517
1.511
1.508
1.502
1.493
1.370
1.358
1.350
1.346
1.334
1.319
1.313
1.301
1.291
1.285
1.279
1.257
1.246
O
OH
O
O
1.08
1.00
3.49
2.08
2.13
2.01
2.18
1.43
2.45
1.45
10.60
3.04
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
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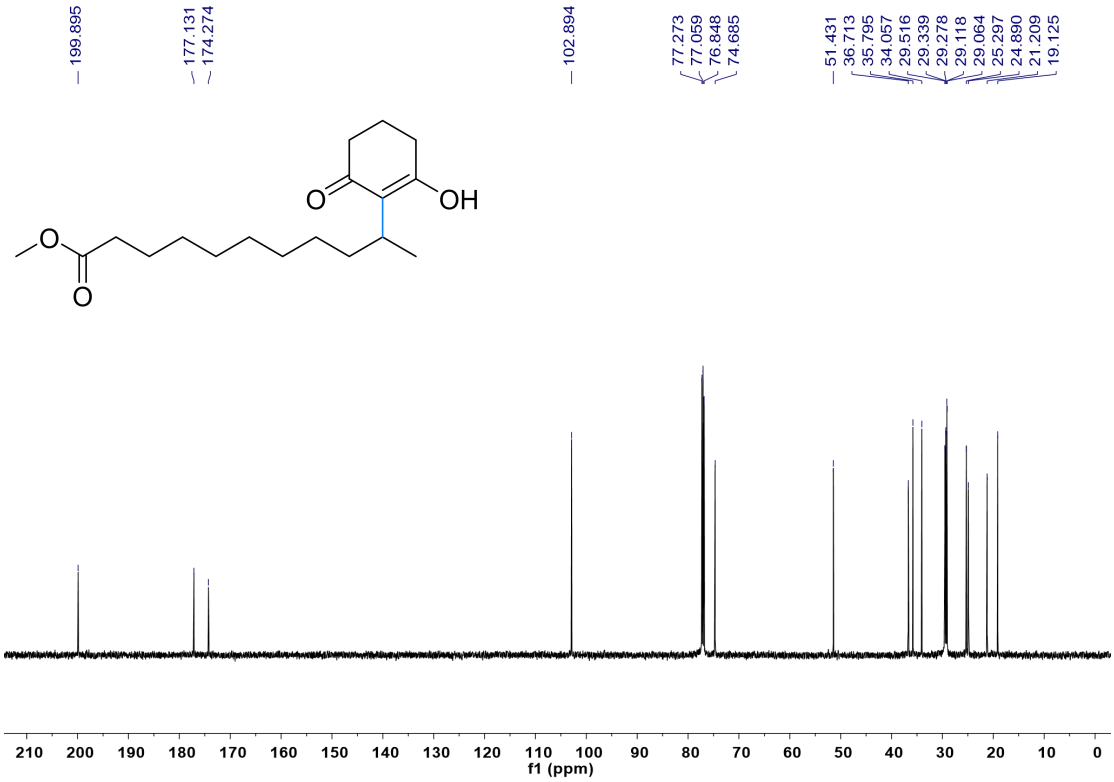
0.0
f1 (ppm)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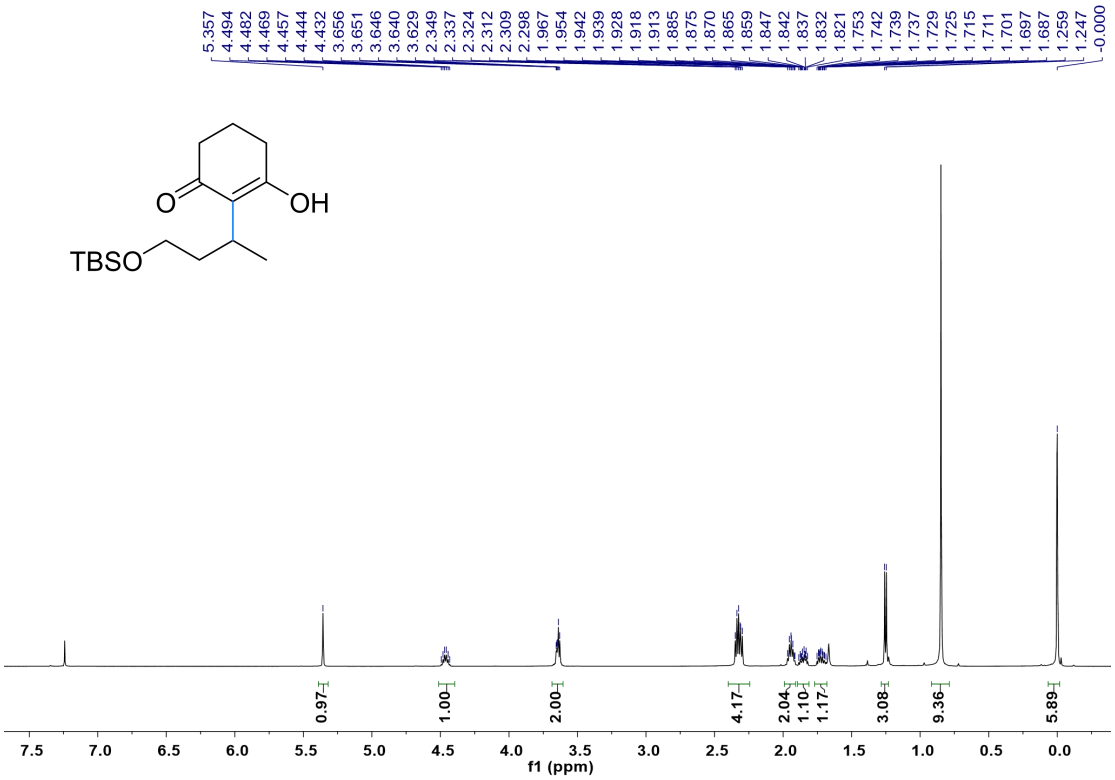
34.057
29.516
29.339
29.278
29.118
29.064
25.297
24.890
21.209
19.125
O
OH
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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3.349
2.337
2.324
2.312
2.309
2.298
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1.954
1.942
1.939
1.928
1.918
1.913
1.885
1.875
1.870
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1.739
1.737
1.729
1.725
1.715
1.711
1.701
1.697
1.687
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1.247
-0.000
O
OH
TBSO
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2.00
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2.04
1.10
1.17
3.08
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6.5
6.0
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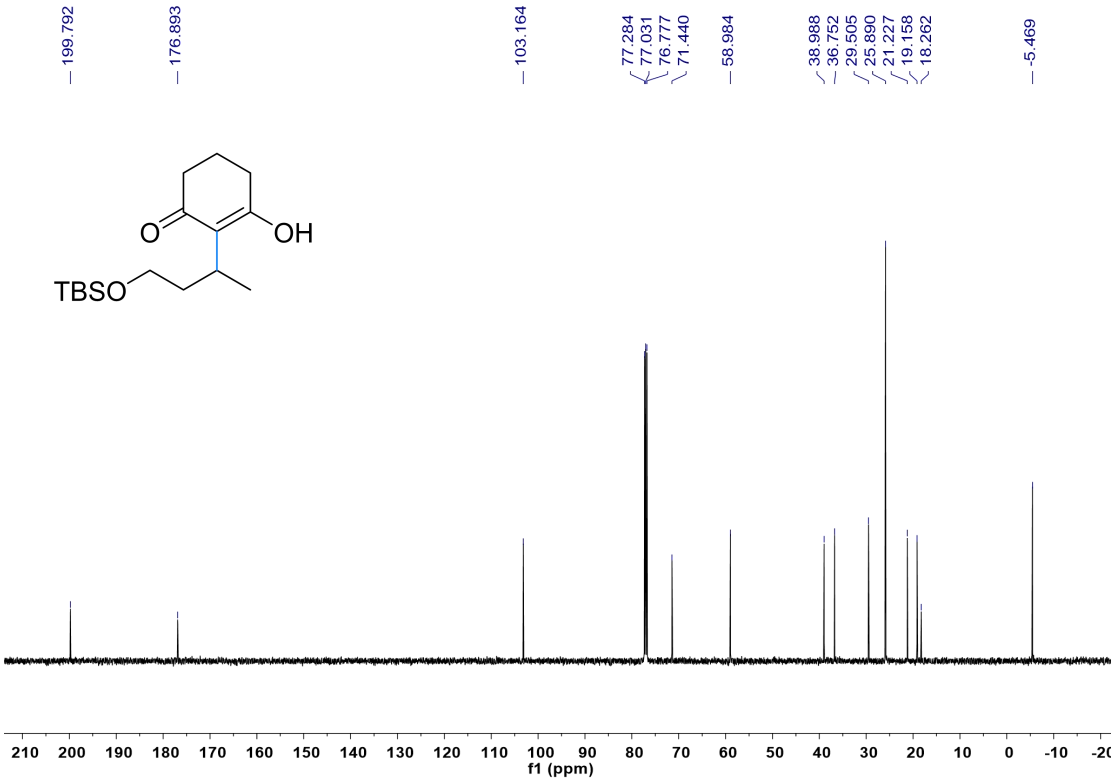
f1 (ppm)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21.227
19.158
18.262
-5.469
O
OH
TBSO
210
200
190
180
170
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120
110
100
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80
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60
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40
30
20
10
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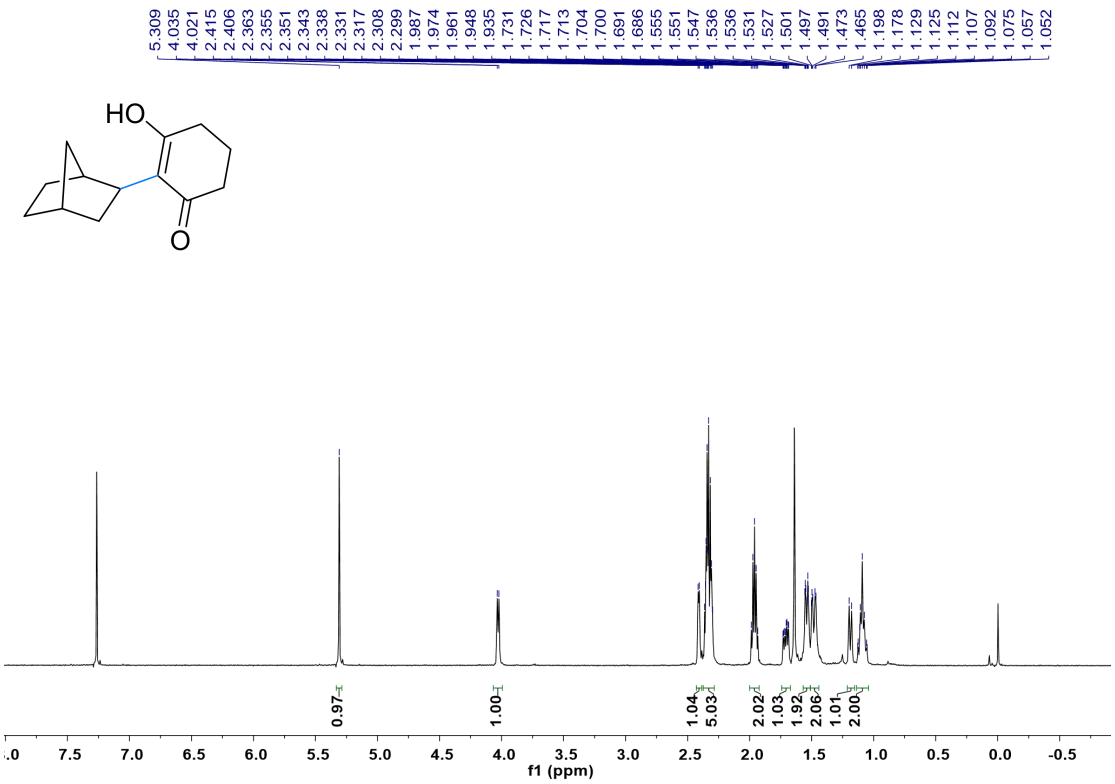
f1 (ppm)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2.299
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1.731
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1.713
1.704
1.700
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1.536
1.536
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1.075
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1.052
HO
O
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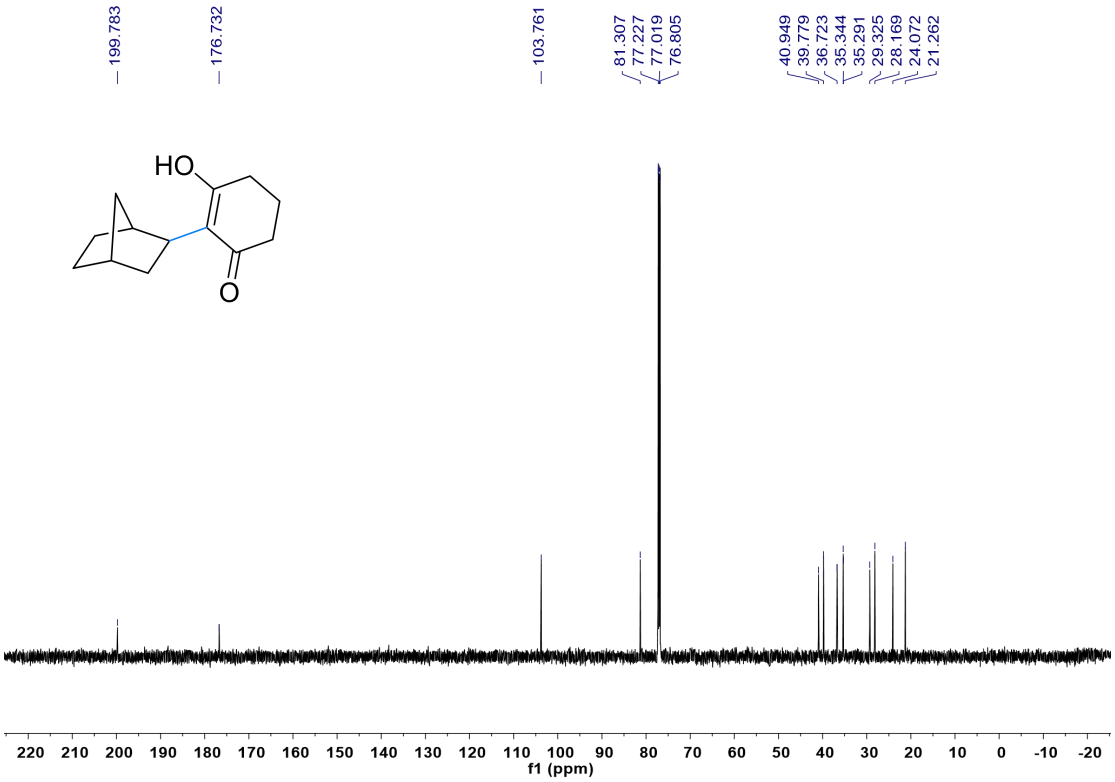
2.06
1.01
2.00
f1 (ppm)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24.072
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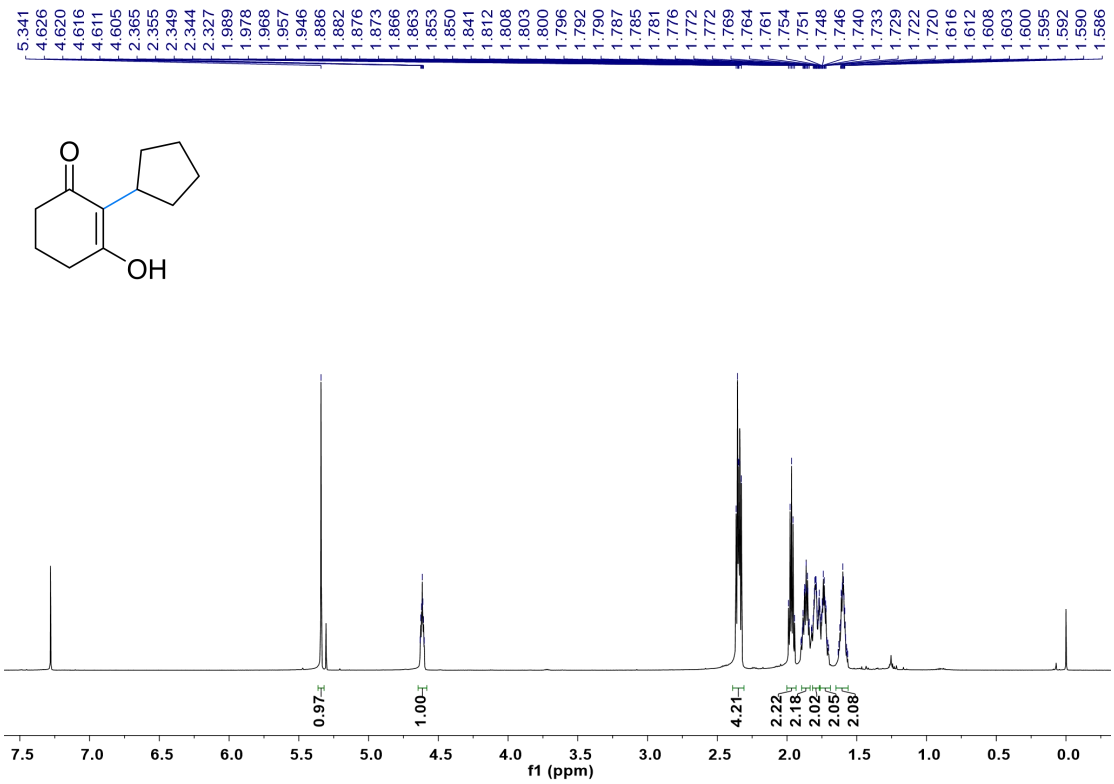
HO
O
f1 (ppm)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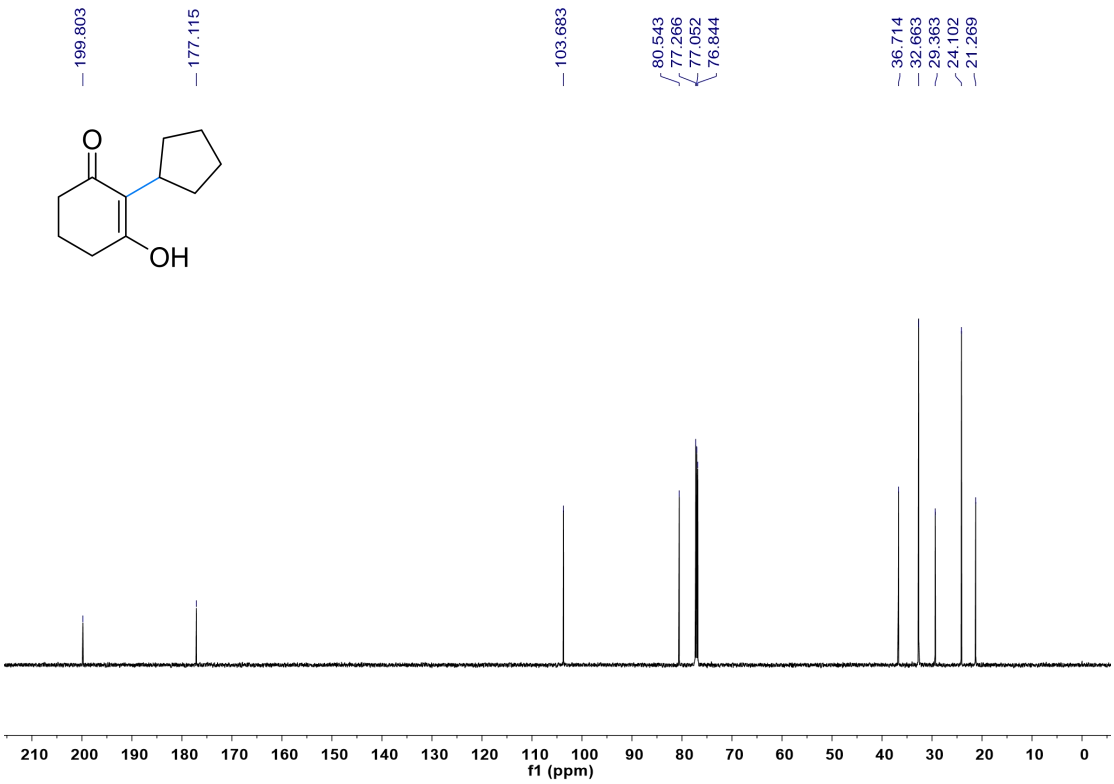
1.729
1.722
1.720
1.616
1.612
1.608
1.603
1.600
1.595
1.592
1.590
1.586
O
OH
0.97
1.00
4.21
2.22
2.18
2.02
2.05
2.08
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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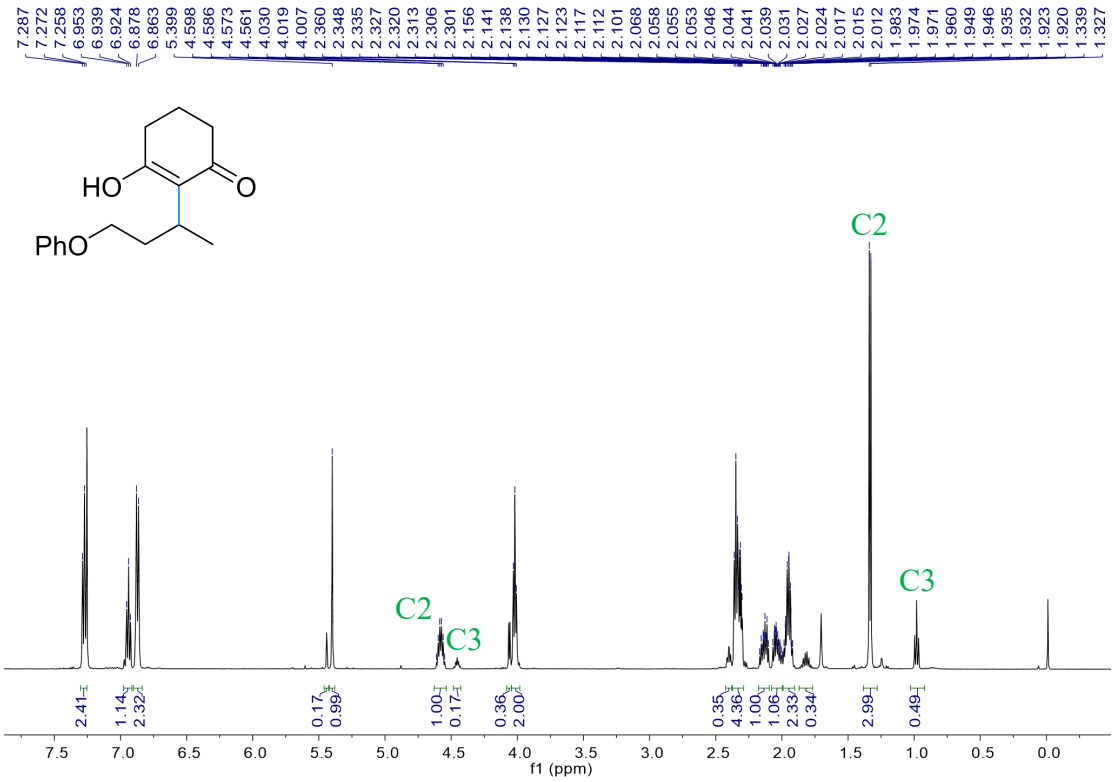
O
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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4.019
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2.015
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1.946
1.935
1.932
1.923
1.920
1.339
1.327
HO
O
PhO
C2
C3
C2
C3
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1.14
2.32
0.17
0.99
1.00
0.17
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4.36
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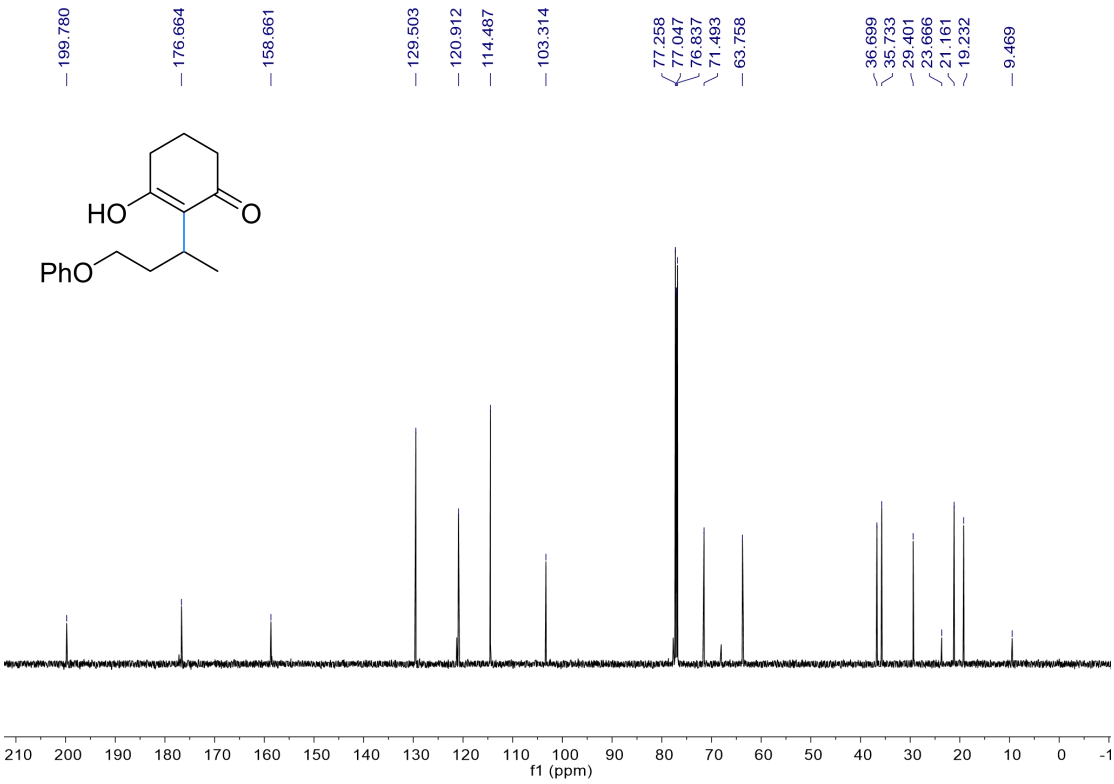
0.34
2.99
0.49
f1 (ppm)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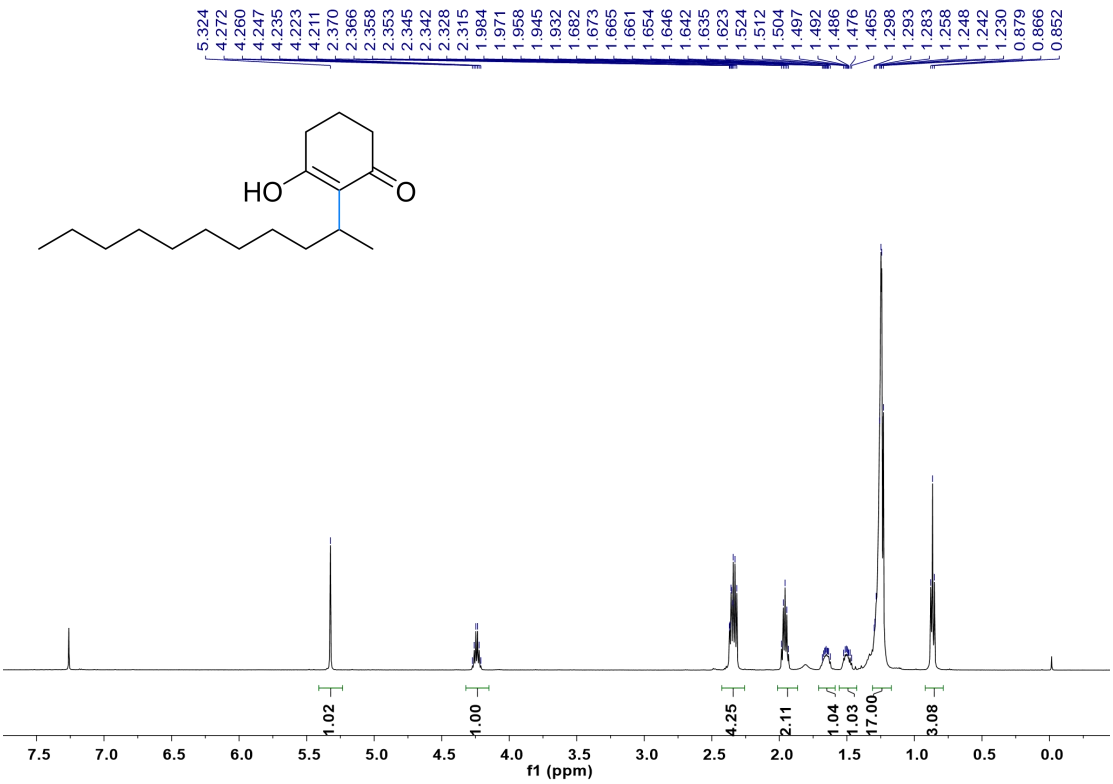
29.401
23.666
21.161
19.232
9.469
HO
O
PhO
f1 (ppm)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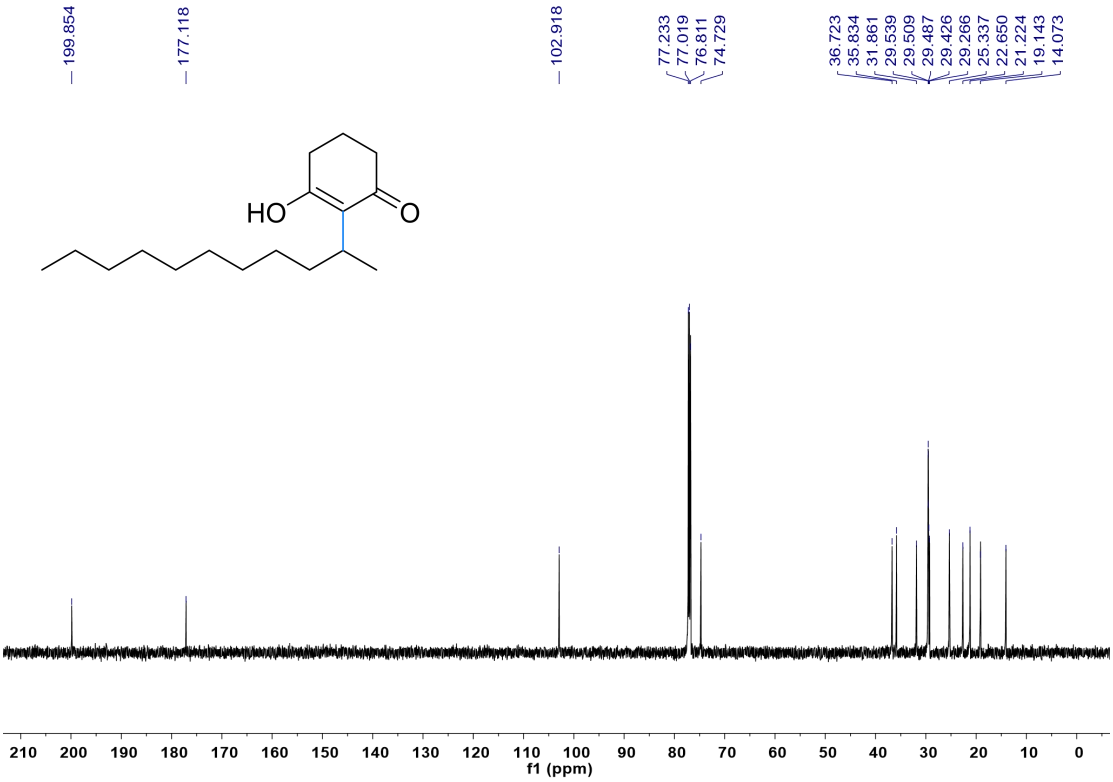
1.230
0.879
0.866
0.852
HO
O
1.02
1.00
4.25
2.11
1.04
1.03
17.00
3.08
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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36.723
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31.861
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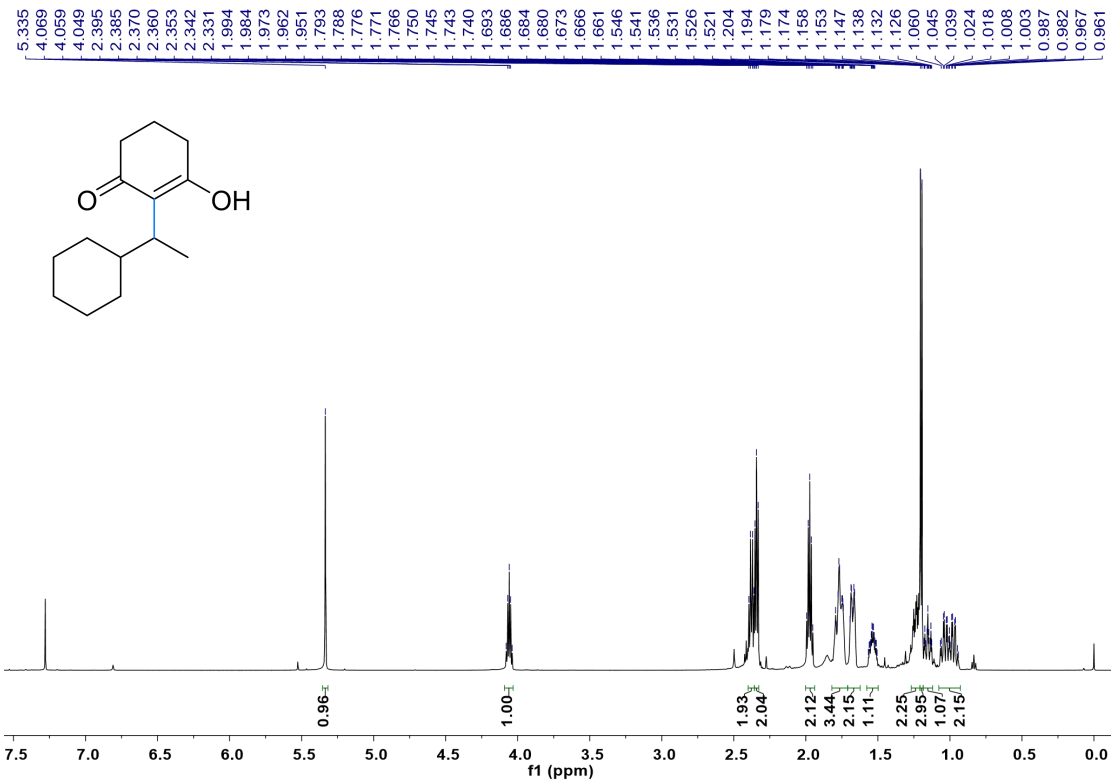
29.509
29.487
29.426
29.266
25.337
22.650
21.224
19.143
14.073
HO
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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2.360
2.353
2.342
2.331
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1.984
1.973
1.962
1.951
1.793
1.788
1.776
1.771
1.766
1.750
1.745
1.743
1.740
1.693
1.686
1.684
1.680
1.673
1.666
1.661
1.546
1.541
1.536
1.531
1.526
1.521
1.204
1.194
1.179
1.174
1.158
1.153
1.147
1.138
1.132
1.126
1.060
1.045
1.039
1.024
1.018
1.008
1.003
0.987
0.982
0.967
0.961
O
OH
0.96
1.00
1.93
2.04
2.12
3.44
2.15
1.11
2.25
2.95
1.07
2.15
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
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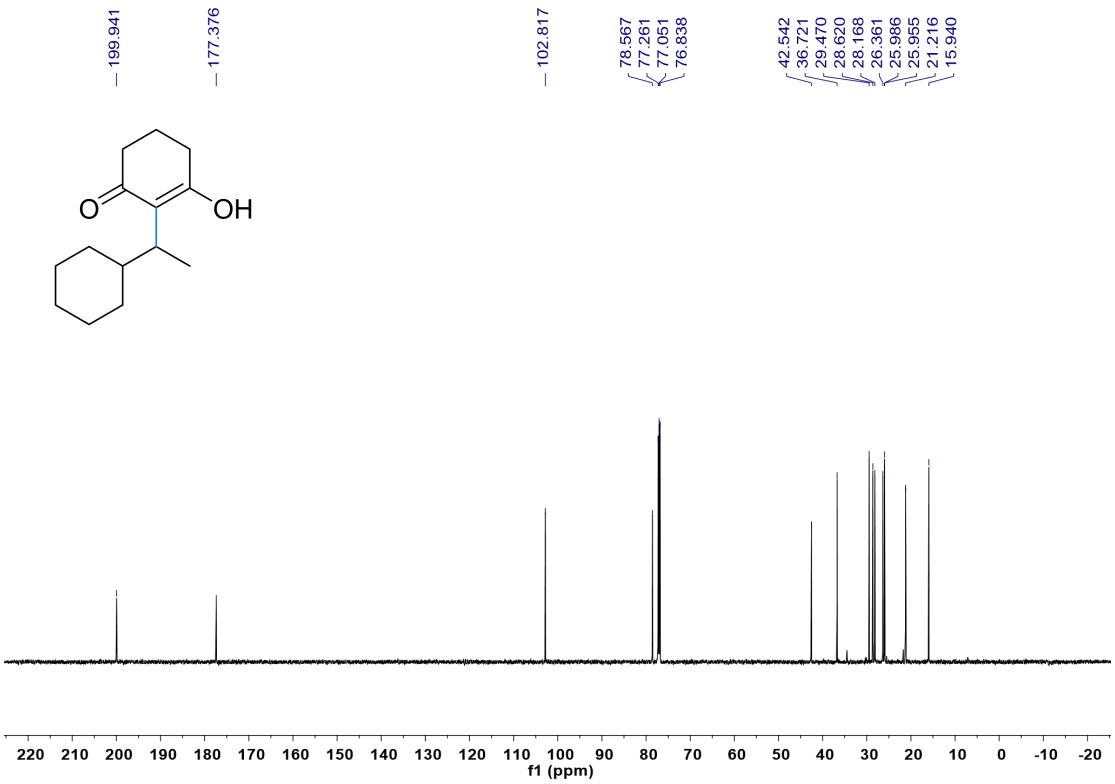
0.0
f1 (ppm)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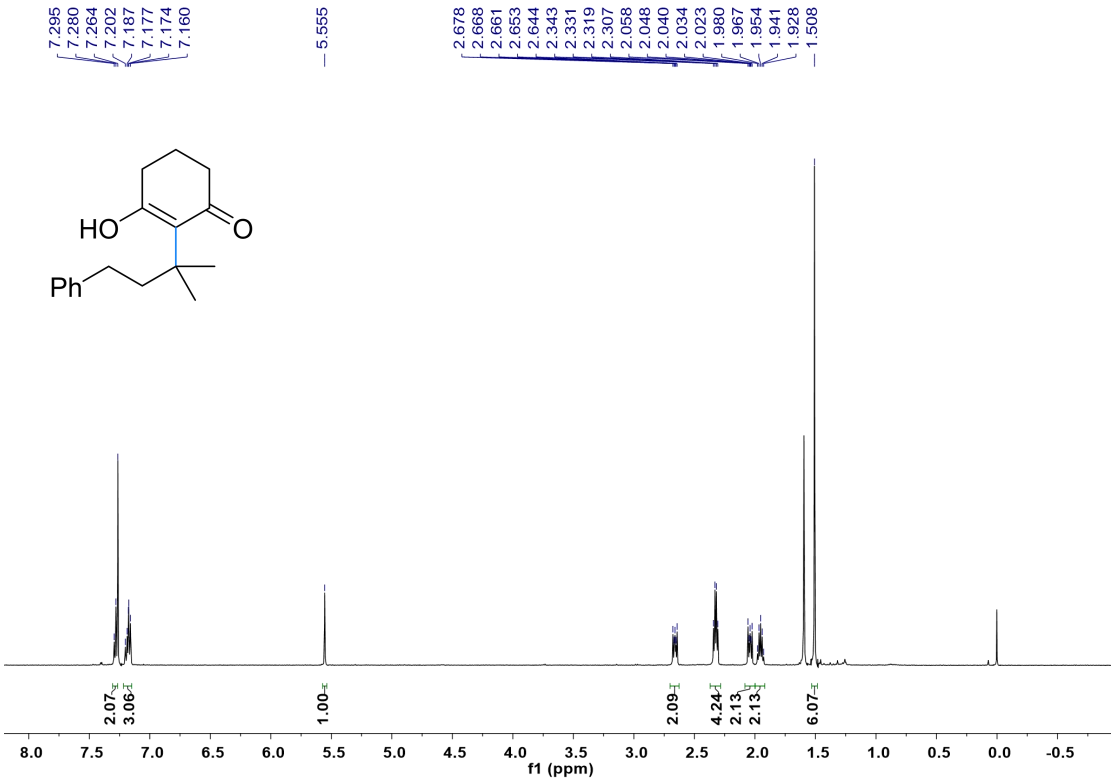
28.168
26.361
25.986
25.955
21.216
15.940
O
OH
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)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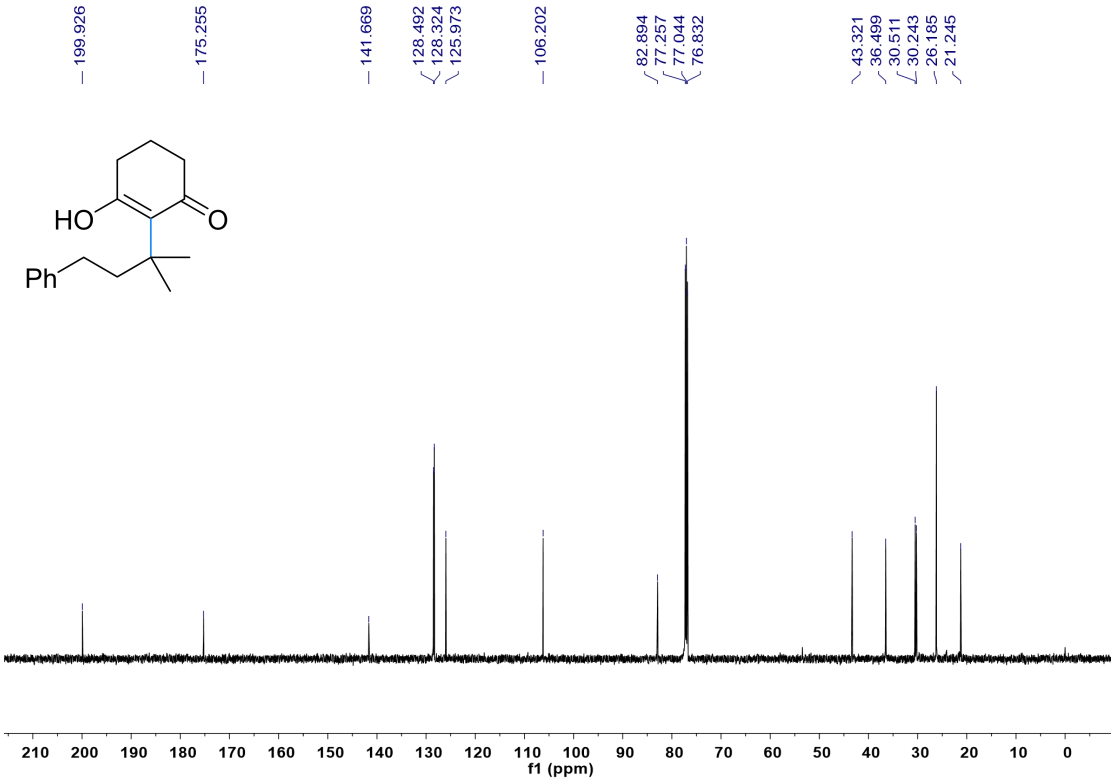
7.177
7.174
7.160
5.555
2.678
2.668
2.661
2.653
2.644
2.343
2.331
2.319
2.307
2.058
2.048
2.040
2.034
2.023
1.980
1.967
1.954
1.941
1.928
1.508
HO
O
Ph
2.07
3.06
1.00
2.09
4.24
2.13
2.13
6.07
f1 (ppm)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141.669
128.492
128.324
125.973
106.202
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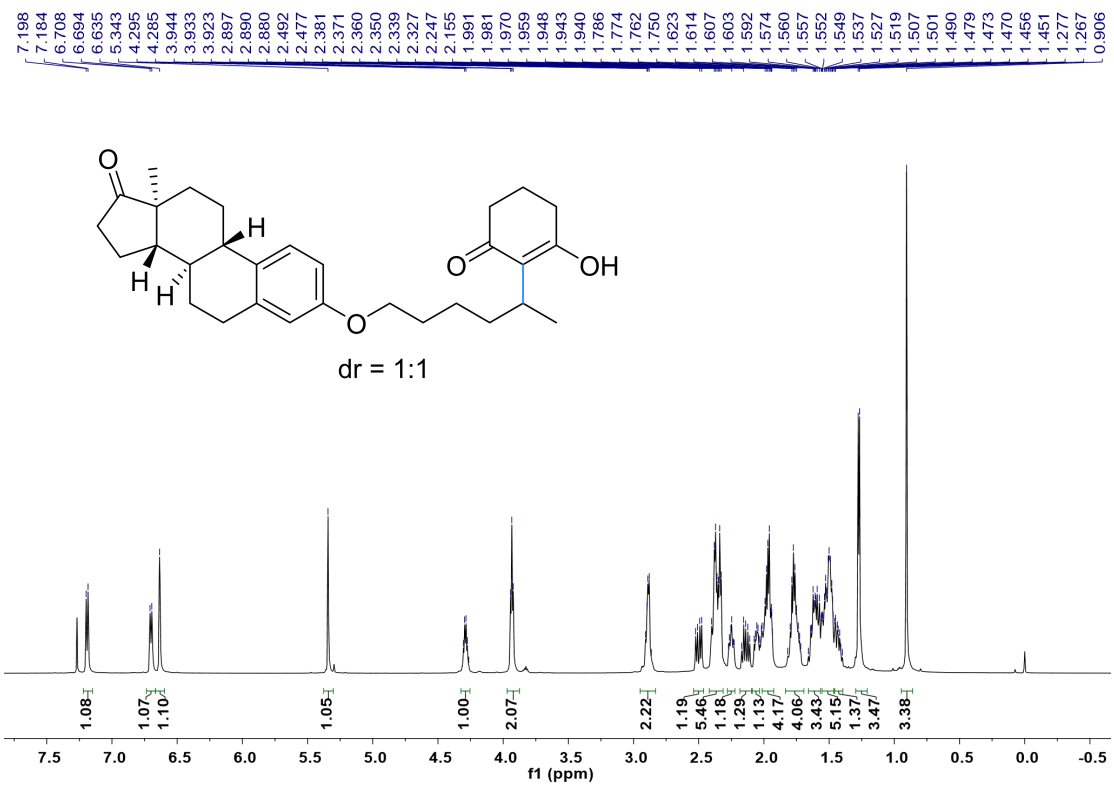
43.321
36.499
30.511
30.243
26.185
21.245
HO
O
Ph
f1 (ppm)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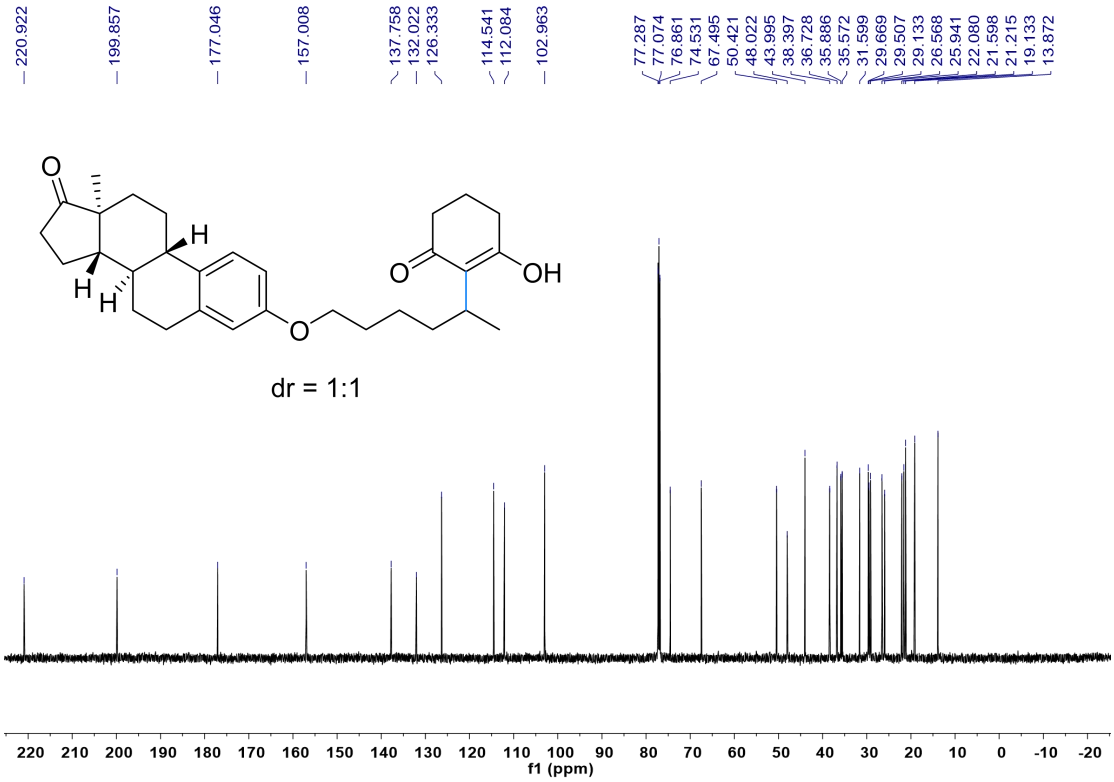
7.184
6.708
6.694
6.635
5.343
4.295
4.285
3.944
3.933
3.923
2.897
2.890
2.880
2.492
2.477
2.381
2.371
2.360
2.350
2.339
2.327
2.247
2.155
1.991
1.981
1.970
1.959
1.948
1.943
1.940
1.786
1.774
1.762
1.750
1.623
1.614
1.607
1.603
1.592
1.574
1.560
1.557
1.552
1.549
1.537
1.527
1.519
1.507
1.501
1.490
1.479
1.473
1.470
1.456
1.451
1.277
1.267
0.906
O
H
H
H
O
O
OH
dr = 1:1
1.08
1.07
1.10
1.05
1.00
2.07
2.22
1.19
5.46
1.18
1.29
1.13
4.17
4.06
3.43
5.15
1.37
3.47
3.38
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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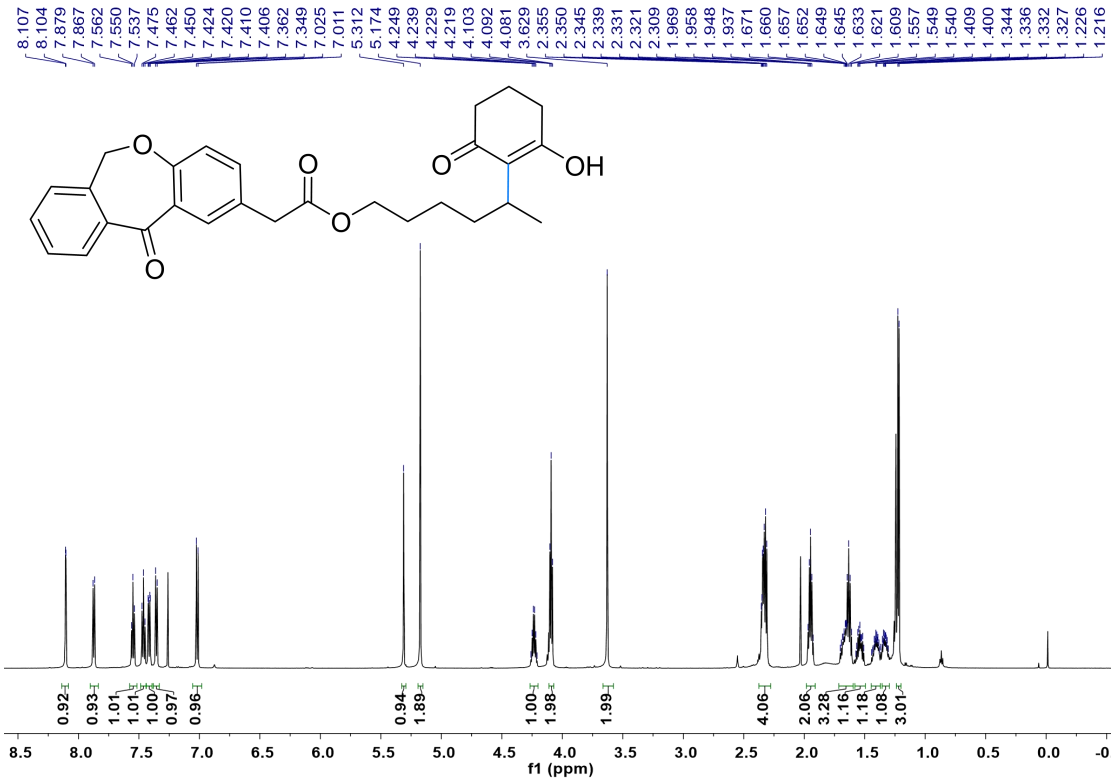
177.046
157.008
137.758
132.022
126.333
114.541
112.084
102.963
77.287
77.074
76.861
74.531
67.495
50.421
48.022
43.995
38.397
36.728
35.886
35.572
31.599
29.669
29.507
29.133
26.568
25.941
22.080
21.598
21.215
19.133
13.872
O
H
H
H
O
O
OH
dr = 1:1
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)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8.104
7.879
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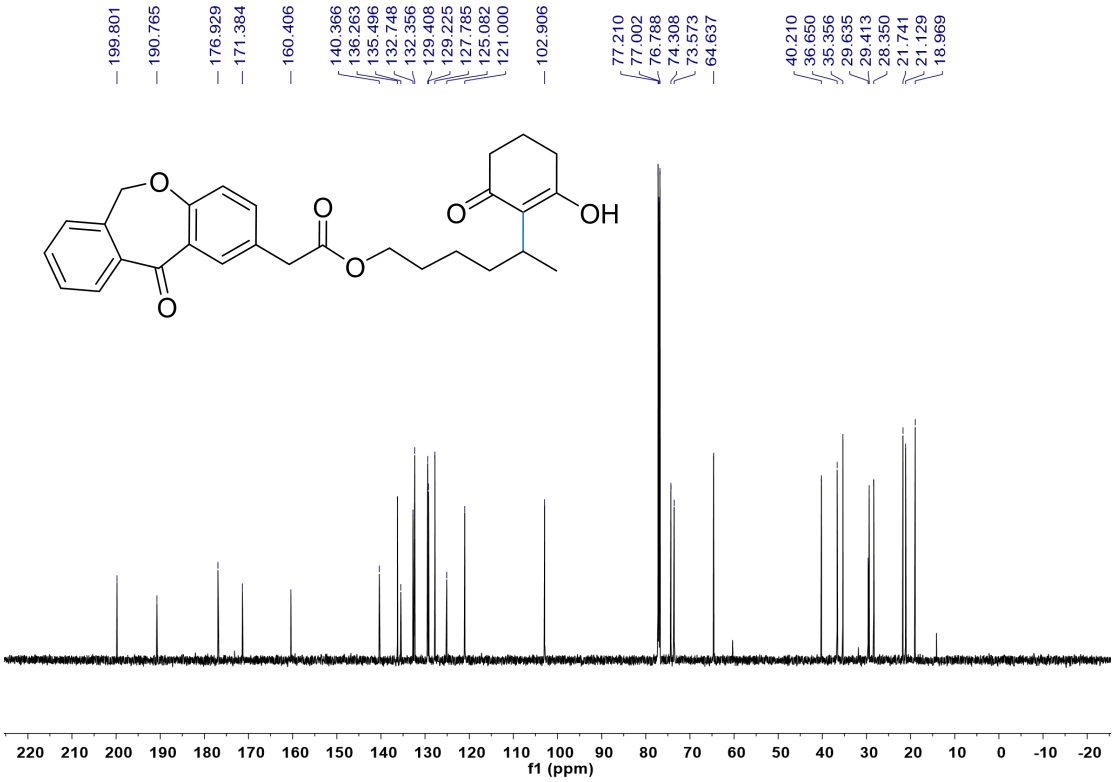
7.562
7.550
7.537
7.475
7.462
7.450
7.424
7.420
7.410
7.406
7.362
7.349
7.025
7.011
5.312
5.174
4.249
4.239
4.229
4.219
4.103
4.092
4.081
3.629
2.355
2.350
2.345
2.339
2.331
2.321
2.309
1.969
1.958
1.948
1.937
1.671
1.660
1.657
1.652
1.649
1.645
1.633
1.621
1.609
1.557
1.549
1.540
1.409
1.400
1.344
1.336
1.332
1.327
1.226
1.216
O
O
O
O
OH
0.92
0.93
1.01
1.01
1.00
0.97
0.96
0.94
1.89
1.00
1.98
1.99
4.06
2.06
3.28
1.16
1.18
1.08
3.01
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
f1 (ppm)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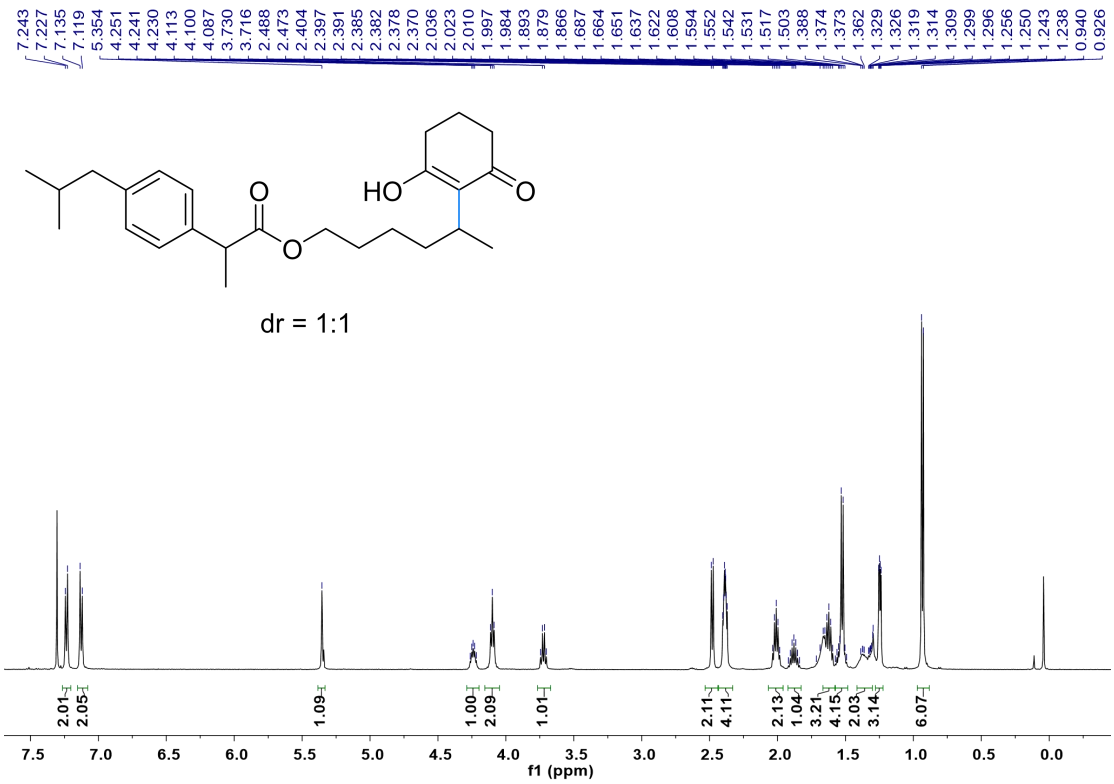
160.406
140.366
136.263
135.496
132.748
132.356
129.408
129.225
127.785
125.082
121.000
102.906
77.210
77.002
76.788
74.308
73.573
64.637
40.210
36.650
35.356
29.635
29.413
28.350
21.741
21.129
18.969
O
O
O
O
OH
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)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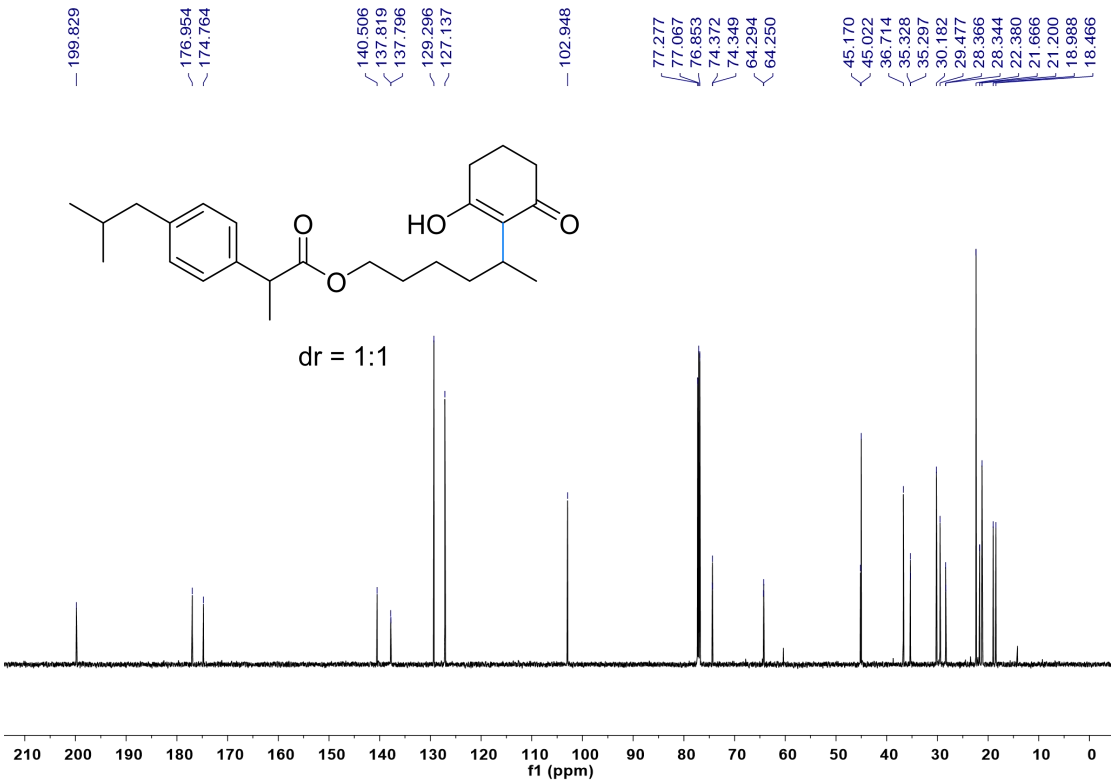
7.227
7.135
7.119
5.354
4.251
4.241
4.230
4.113
4.100
4.087
3.730
3.716
2.488
2.473
2.404
2.397
2.391
2.385
2.382
2.378
2.370
2.036
2.023
2.010
1.997
1.984
1.893
1.879
1.866
1.687
1.664
1.651
1.637
1.622
1.608
1.594
1.552
1.542
1.531
1.517
1.503
1.388
1.374
1.373
1.362
1.329
1.326
1.319
1.314
1.309
1.299
1.296
1.256
1.250
1.243
1.238
0.940
0.926
HO
O
O
O
dr = 1:1
2.01
2.05
1.09
1.00
2.09
1.01
2.11
4.11
2.13
1.04
3.21
4.15
2.03
3.14
6.07
f1 (ppm)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174.764
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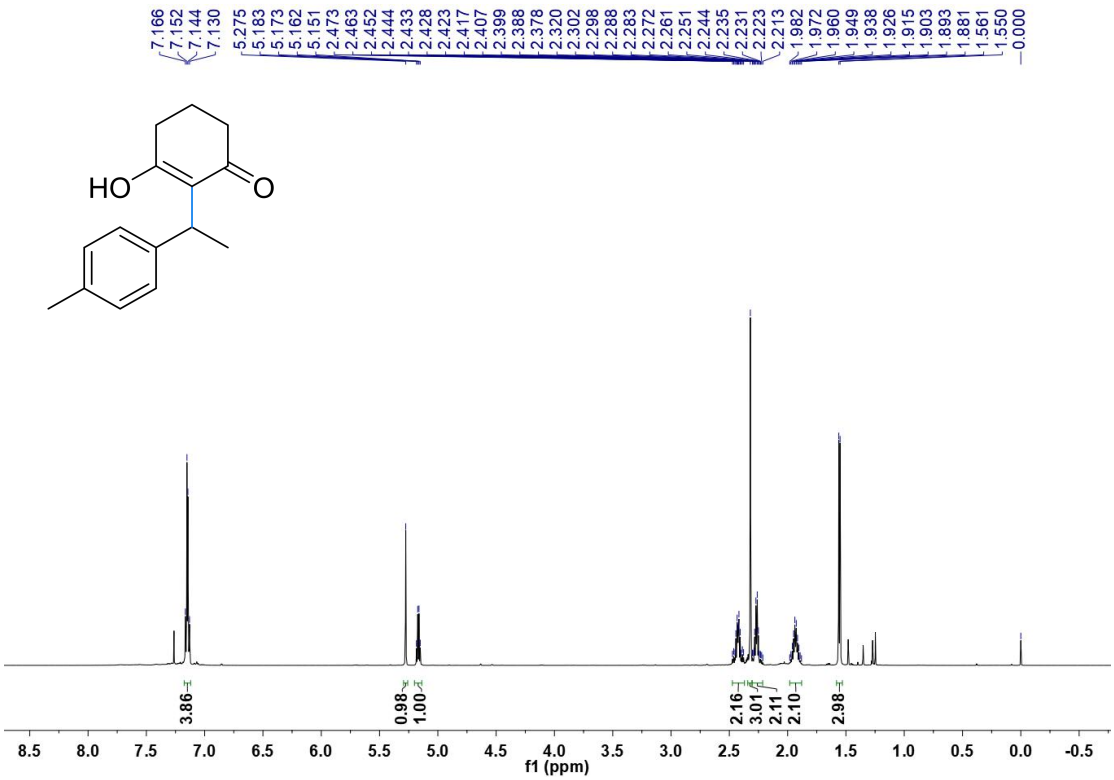
129.296
127.137
102.948
77.277
77.067
76.853
74.372
74.349
64.294
64.250
45.170
45.022
36.714
35.328
35.297
30.182
29.477
28.366
28.344
22.380
21.666
21.200
18.988
18.466
HO
O
O
O
dr = 1:1
f1 (ppm)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7.130
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5.183
5.173
5.162
5.151
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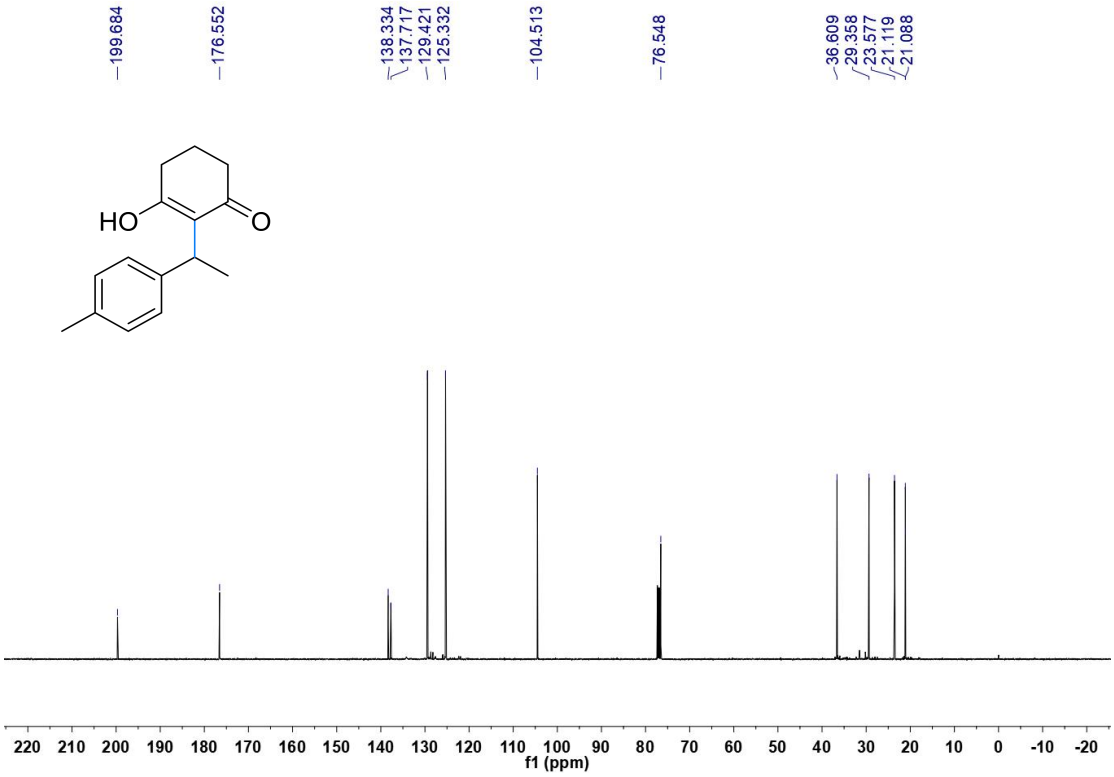
2.463
2.452
2.444
2.433
2.428
2.423
2.417
2.407
2.399
2.388
2.378
2.320
2.302
2.298
2.288
2.283
2.272
2.261
2.251
2.244
2.235
2.231
2.223
2.213
1.982
1.972
1.960
1.949
1.938
1.926
1.915
1.903
1.893
1.881
1.561
1.550
0.000
HO
O
3.86
0.98
1.00
2.16
3.01
2.11
2.10
2.98
f1 (ppm)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137.717
129.421
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104.513
76.548
36.609
29.358
23.577
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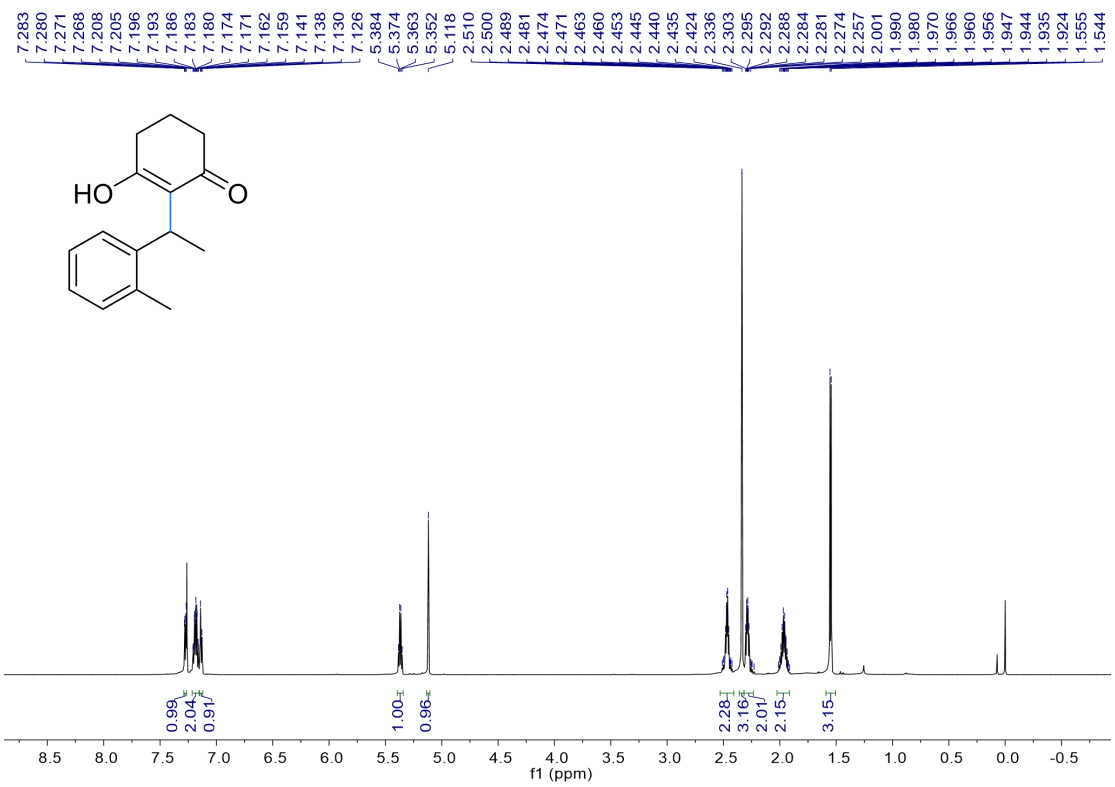
21.088
HO
O
f1 (ppm)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7.205
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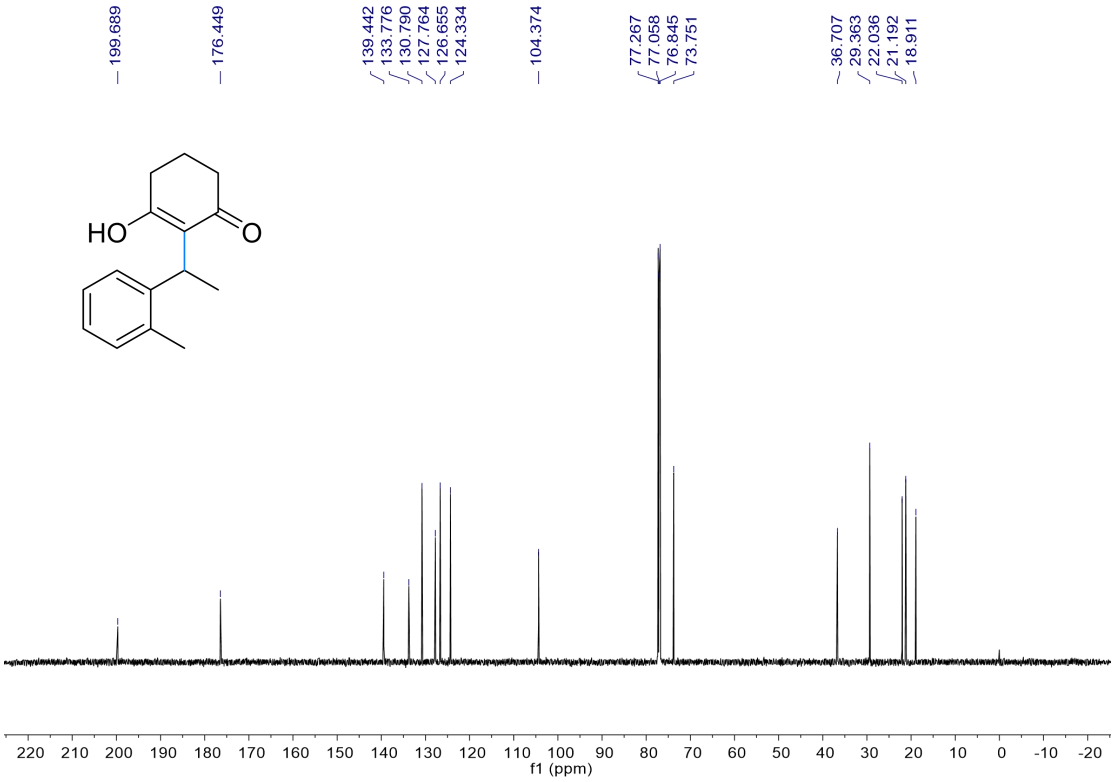
7.193
7.186
7.183
7.180
7.174
7.171
7.162
7.159
7.141
7.138
7.130
7.126
5.384
5.374
5.363
5.352
5.118
2.510
2.500
2.489
2.481
2.474
2.471
2.463
2.460
2.453
2.445
2.440
2.435
2.424
2.336
2.303
2.295
2.292
2.288
2.284
2.281
2.274
2.257
2.001
1.990
1.980
1.970
1.966
1.960
1.956
1.947
1.944
1.935
1.924
1.555
1.544
HO
O
0.99
2.04
0.91
1.00
0.96
2.28
3.16
2.01
2.15
3.15
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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124.334
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77.058
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73.751
36.707
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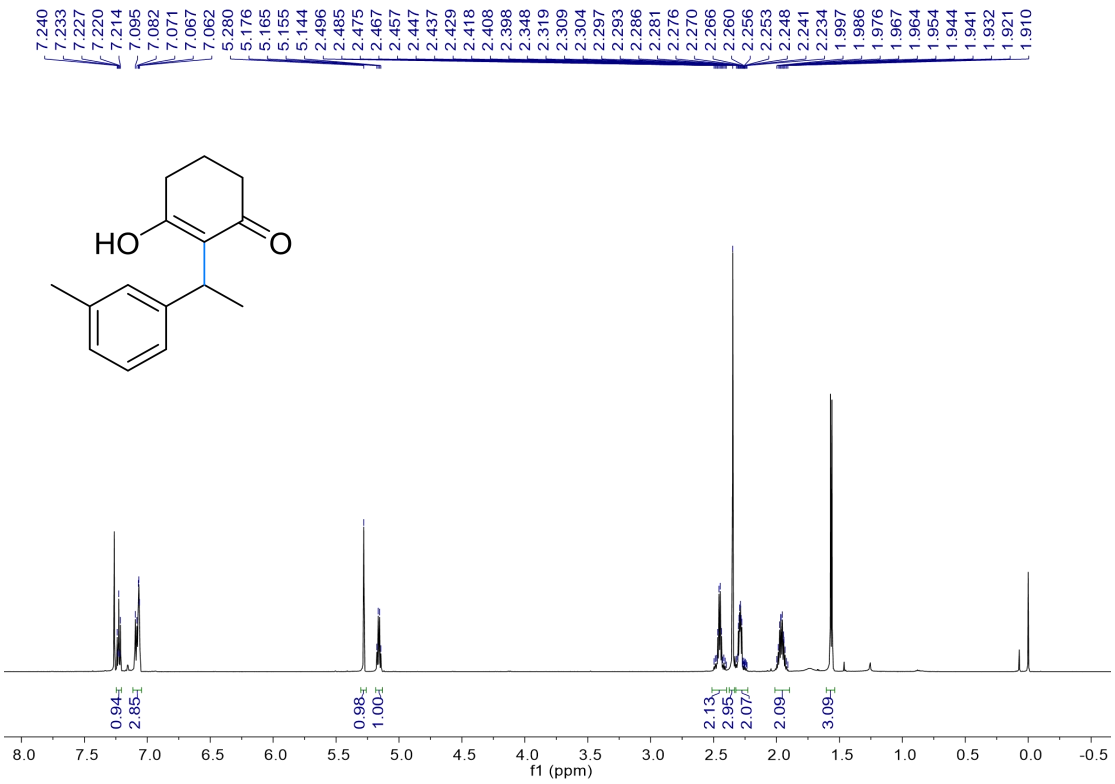
18.911
HO
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)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5.155
5.144
2.496
2.485
2.475
2.467
2.457
2.447
2.437
2.429
2.418
2.408
2.398
2.348
2.319
2.309
2.304
2.297
2.293
2.286
2.281
2.276
2.270
2.266
2.260
2.256
2.253
2.248
2.241
2.234
1.997
1.986
1.976
1.967
1.964
1.954
1.944
1.941
1.932
1.921
1.910
HO
O
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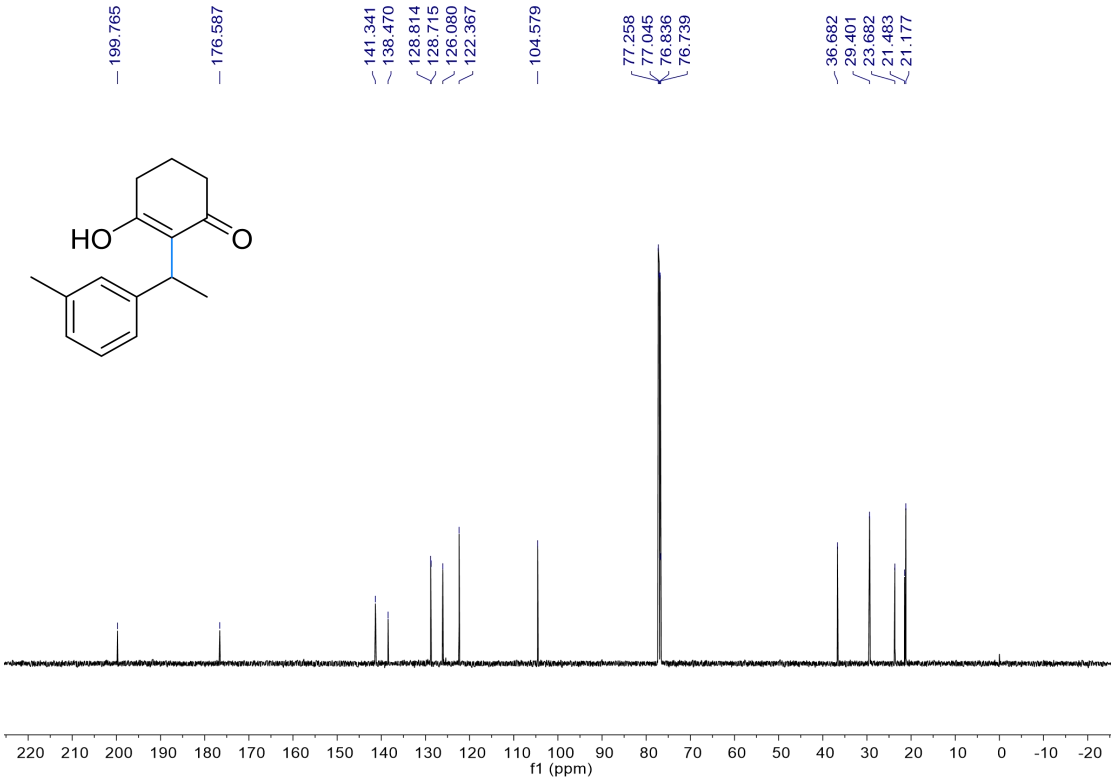
2.85
0.98
1.00
2.13
2.95
2.07
2.09
3.09
f1 (ppm)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76.739
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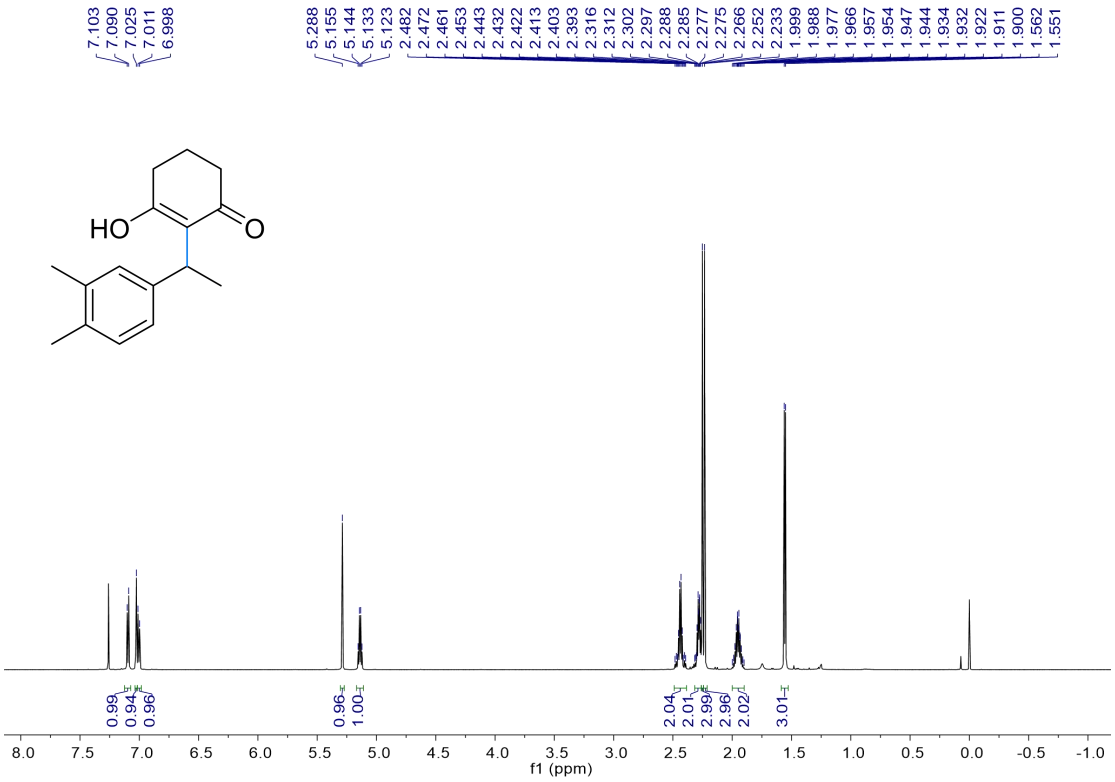
29.401
23.682
21.483
21.177
HO
O
f1 (ppm)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5.144
5.133
5.123
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2.472
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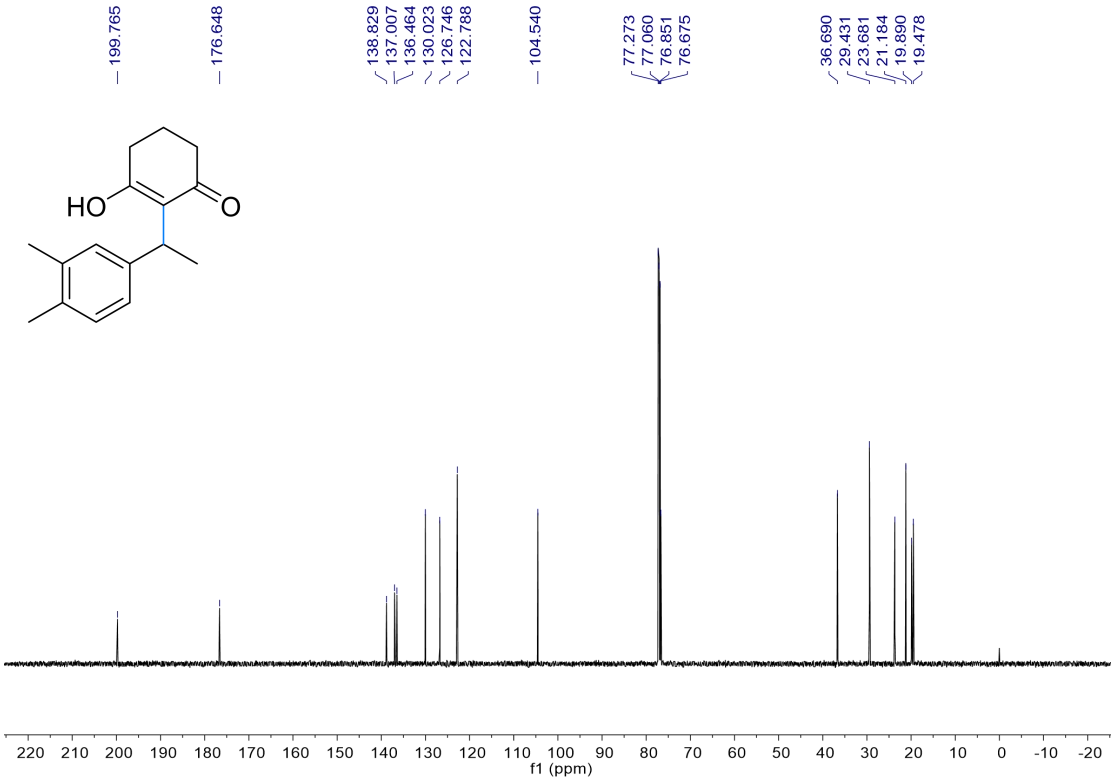
2.453
2.443
2.432
2.422
2.413
2.403
2.393
2.316
2.312
2.302
2.297
2.288
2.285
2.277
2.275
2.266
2.252
2.233
1.999
1.988
1.977
1.966
1.957
1.954
1.947
1.944
1.934
1.932
1.922
1.911
1.900
1.562
1.551
HO
O
0.99
0.94
0.96
0.96
1.00
2.04
2.01
2.99
2.96
2.02
3.01
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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76.675
36.690
29.431
23.681
21.184
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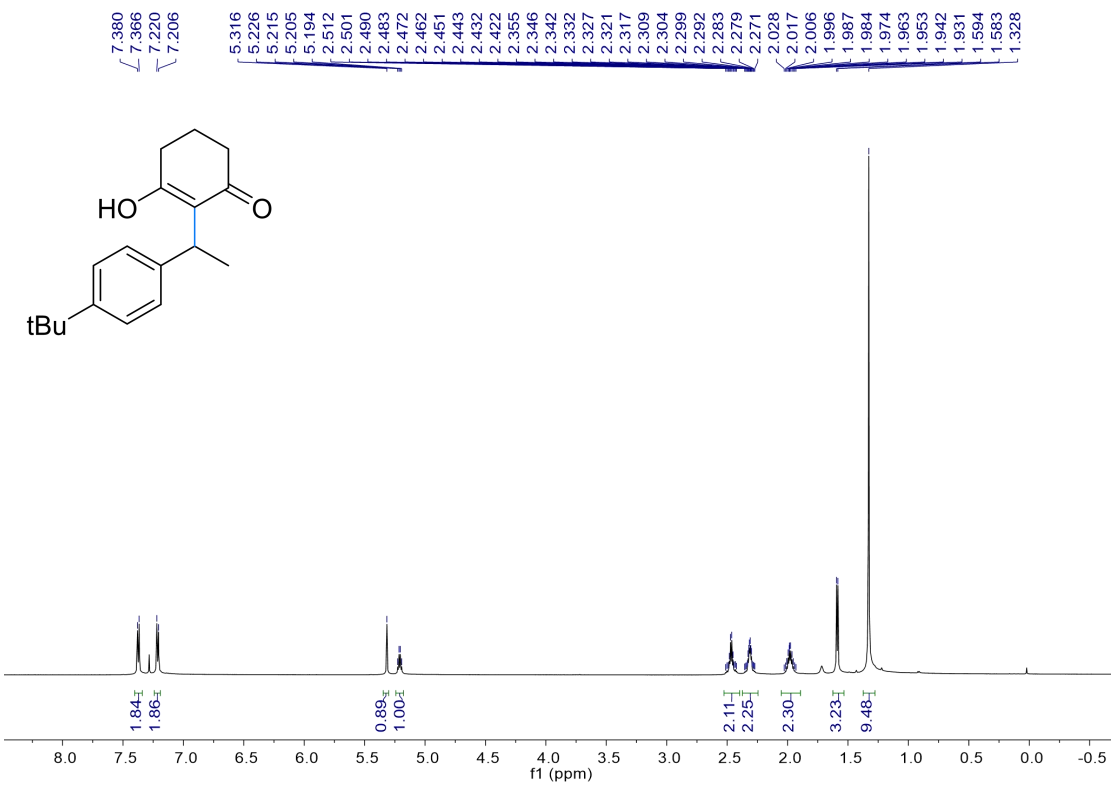
19.478
HO
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)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2.321
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2.304
2.299
2.292
2.283
2.279
2.271
2.028
2.017
2.006
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1.953
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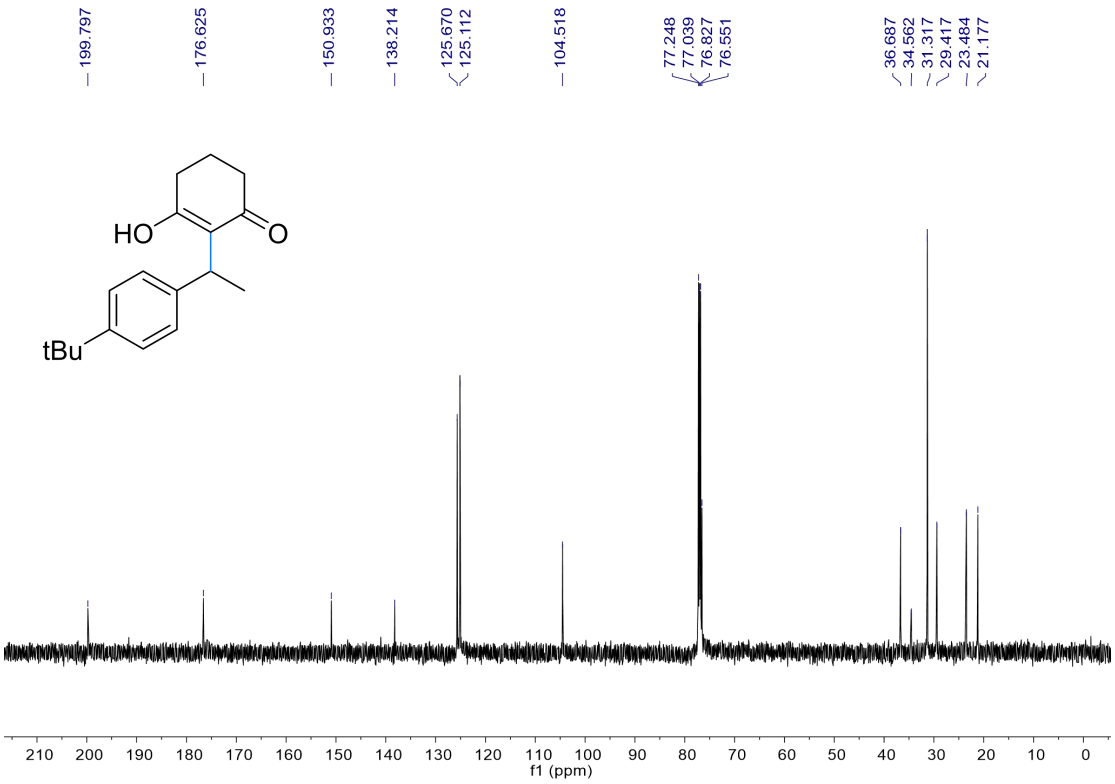
1.583
1.328
HO
O
tBu
1.84
1.86
0.89
1.00
2.11
2.25
2.30
3.23
9.48
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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150.933
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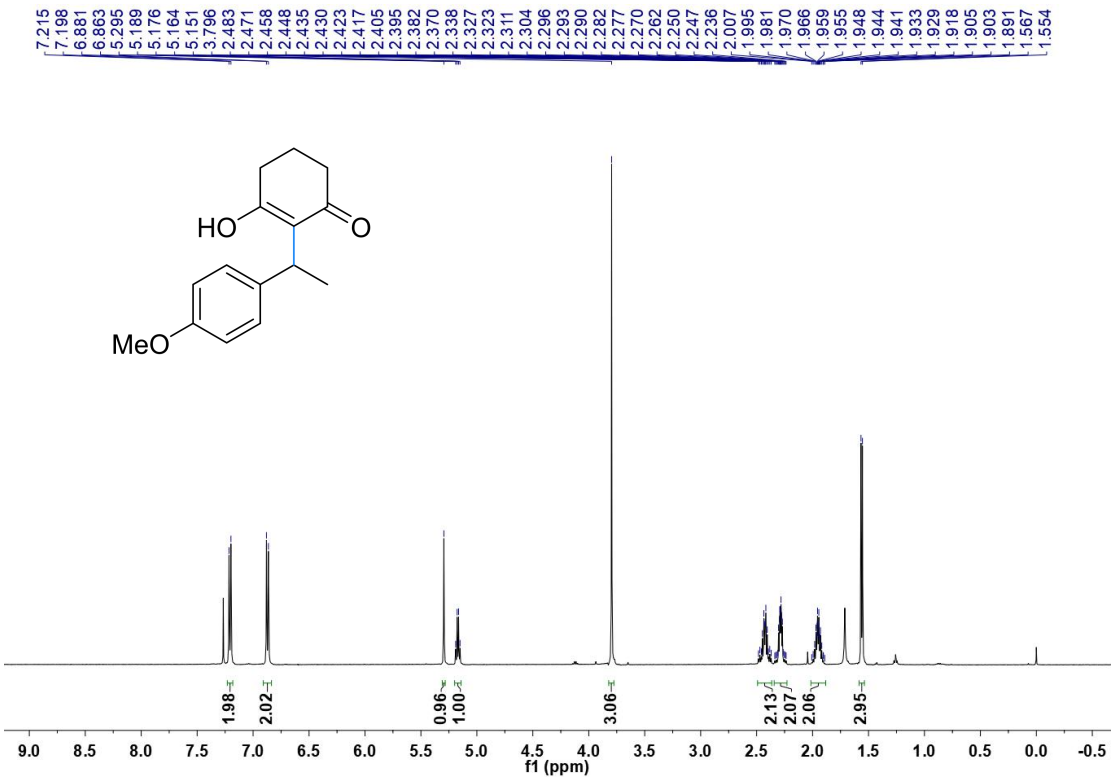
125.670
125.112
104.518
77.248
77.039
76.827
76.551
36.687
34.562
31.317
29.417
23.484
21.177
HO
O
tBu
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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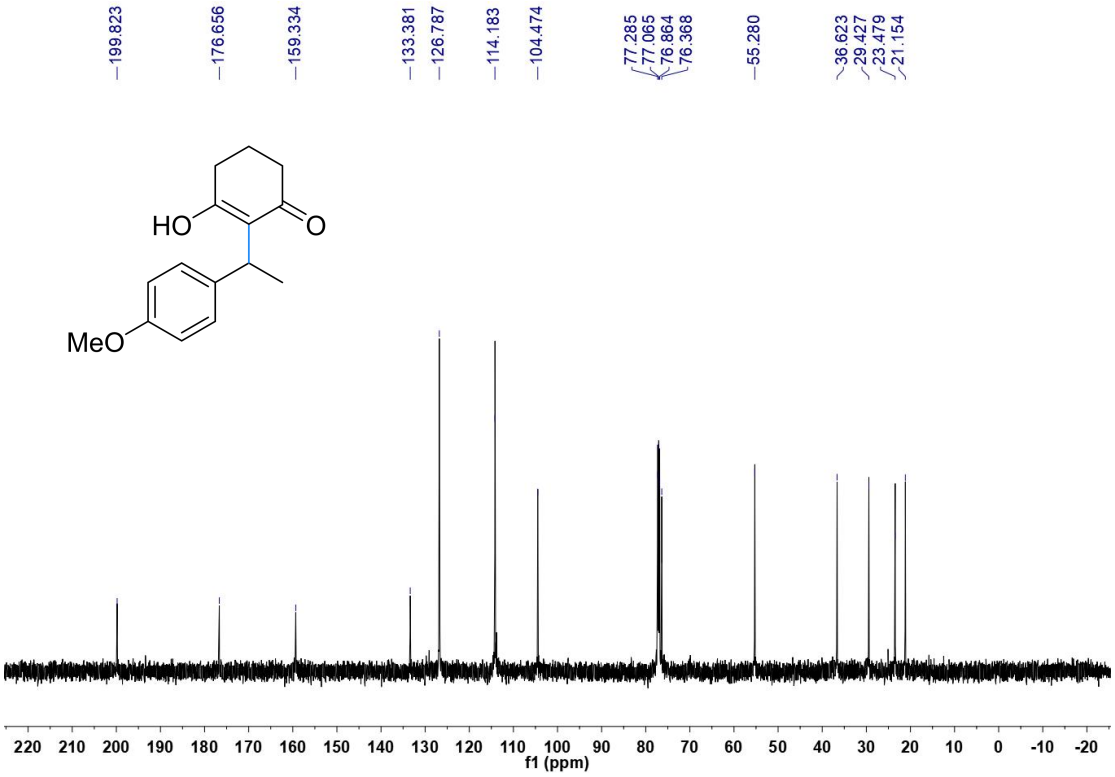
2.471
2.458
2.448
2.435
2.430
2.423
2.417
2.405
2.395
2.382
2.370
2.338
2.327
2.323
2.311
2.304
2.296
2.293
2.290
2.282
2.277
2.270
2.262
2.250
2.247
2.236
2.007
1.995
1.981
1.970
1.966
1.959
1.955
1.948
1.944
1.941
1.933
1.929
1.918
1.905
1.903
1.891
1.567
1.554
HO
O
MeO
1.98
2.02
0.96
1.00
3.06
2.13
2.07
2.06
2.95
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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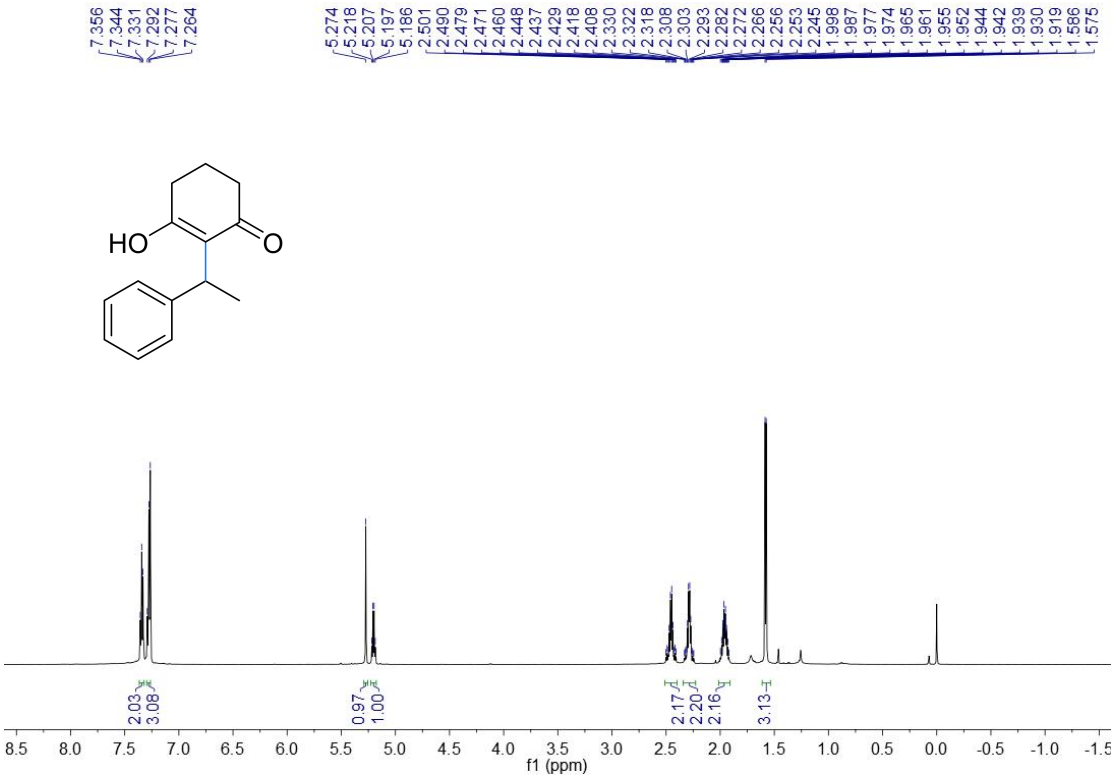
77.285
77.065
76.864
76.368
55.280
36.623
29.427
23.479
21.154
HO
O
MeO
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)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7.331
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5.274
5.218
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5.186
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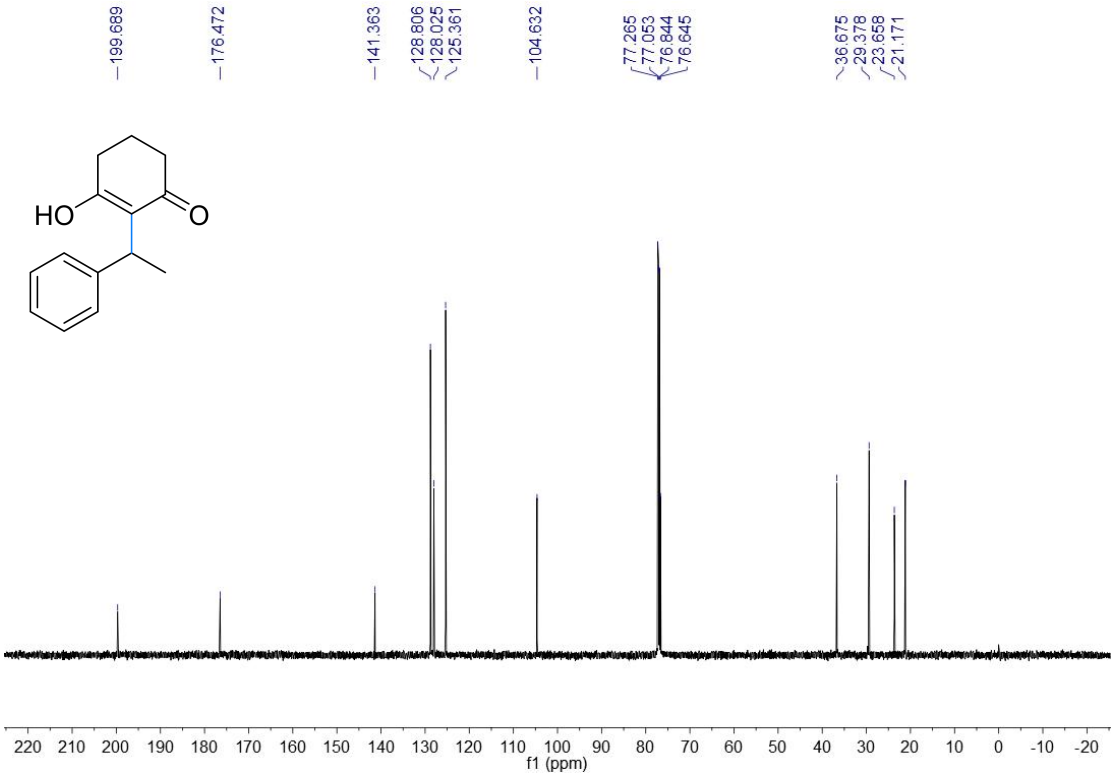
2.490
2.479
2.471
2.460
2.448
2.437
2.429
2.418
2.408
2.330
2.322
2.318
2.308
2.303
2.293
2.282
2.272
2.266
2.256
2.253
2.245
1.998
1.987
1.977
1.974
1.965
1.961
1.955
1.952
1.944
1.942
1.939
1.930
1.919
1.586
1.575
HO
O
2.03
3.08
0.97
1.00
2.17
2.20
2.16
3.13
f1 (ppm)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128.025
125.361
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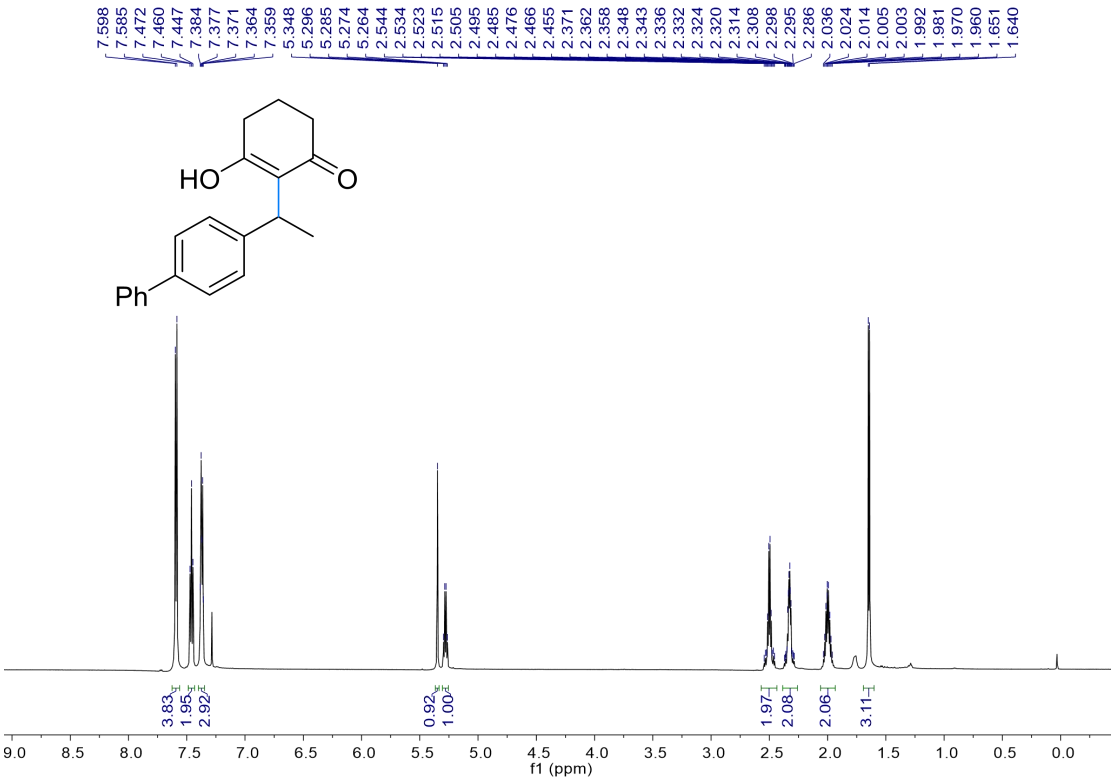
77.265
77.053
76.844
76.645
36.675
29.378
23.658
21.171
HO
O
f1 (ppm)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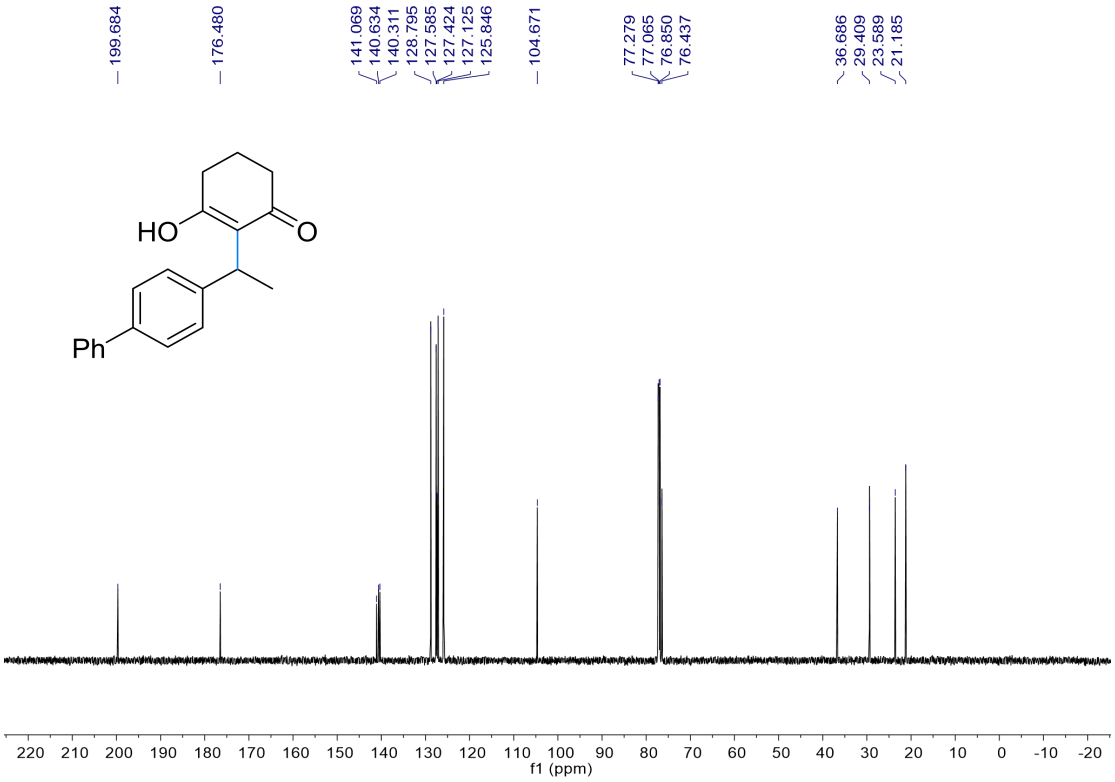
7.472
7.460
7.447
7.384
7.377
7.371
7.364
7.359
5.348
5.296
5.285
5.274
5.264
2.544
2.534
2.523
2.515
2.505
2.495
2.485
2.476
2.466
2.455
2.371
2.362
2.358
2.348
2.343
2.336
2.332
2.324
2.320
2.314
2.308
2.298
2.295
2.286
2.036
2.024
2.014
2.005
2.003
1.992
1.981
1.970
1.960
1.651
1.640
HO
O
Ph
3.83
1.95
2.92
0.92
1.00
1.97
2.08
2.06
3.11
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
f1 (ppm)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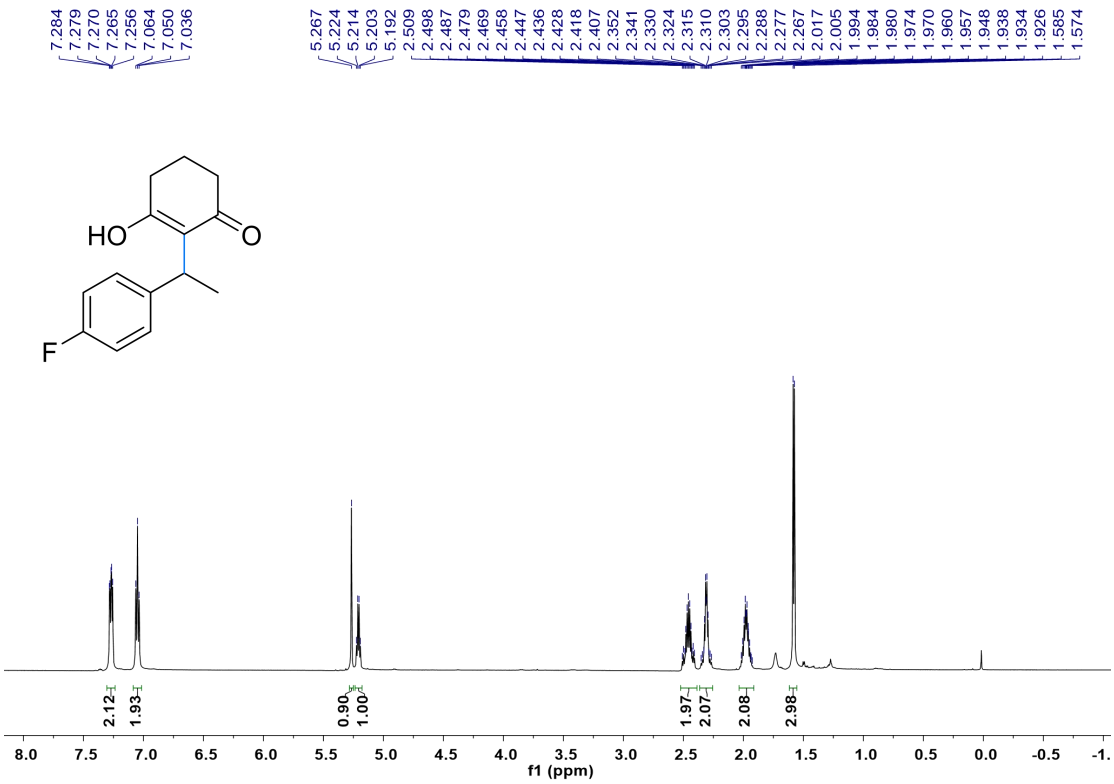
141.069
140.634
140.311
128.795
127.585
127.424
127.125
125.846
104.671
77.279
77.065
76.850
76.437
36.686
29.409
23.589
21.185
HO
O
Ph
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)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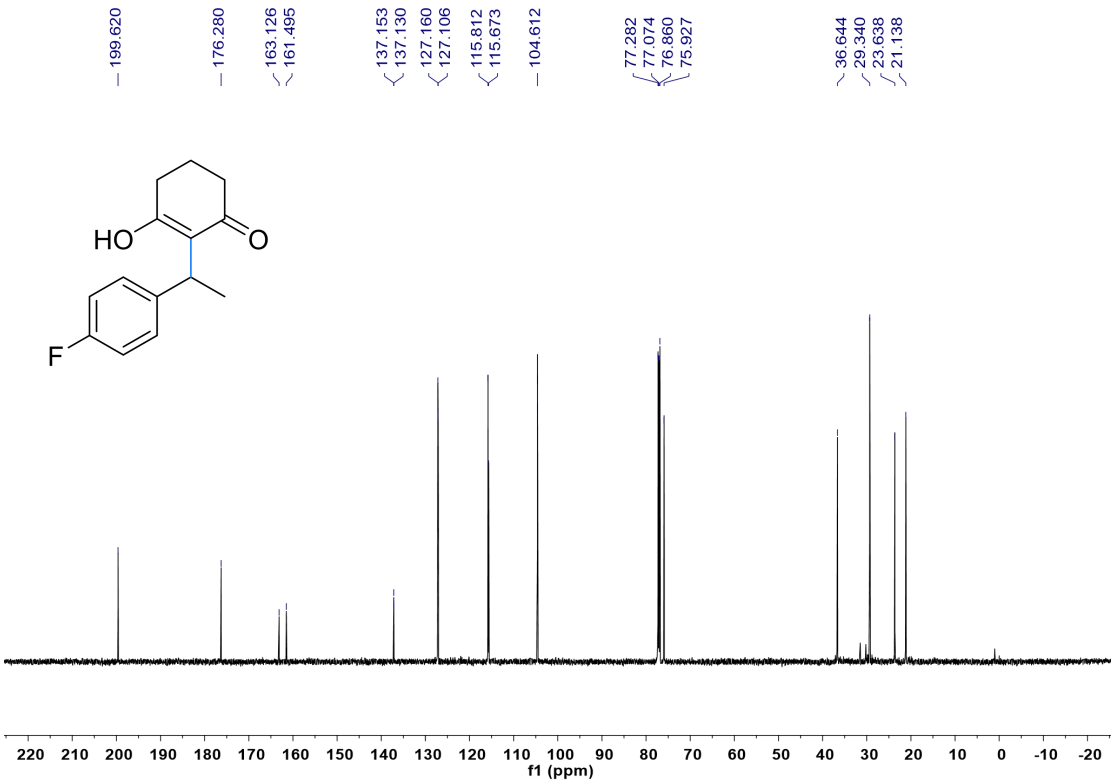
7.279
7.270
7.265
7.256
7.064
7.050
7.036
5.267
5.224
5.214
5.203
5.192
2.509
2.498
2.487
2.479
2.469
2.458
2.447
2.436
2.428
2.418
2.407
2.352
2.341
2.330
2.324
2.315
2.310
2.303
2.295
2.288
2.277
2.267
2.017
2.005
1.994
1.984
1.980
1.974
1.970
1.960
1.957
1.948
1.938
1.934
1.926
1.585
1.574
HO
O
F
2.12
1.93
0.90
1.00
1.97
2.07
2.08
2.98
f1 (ppm)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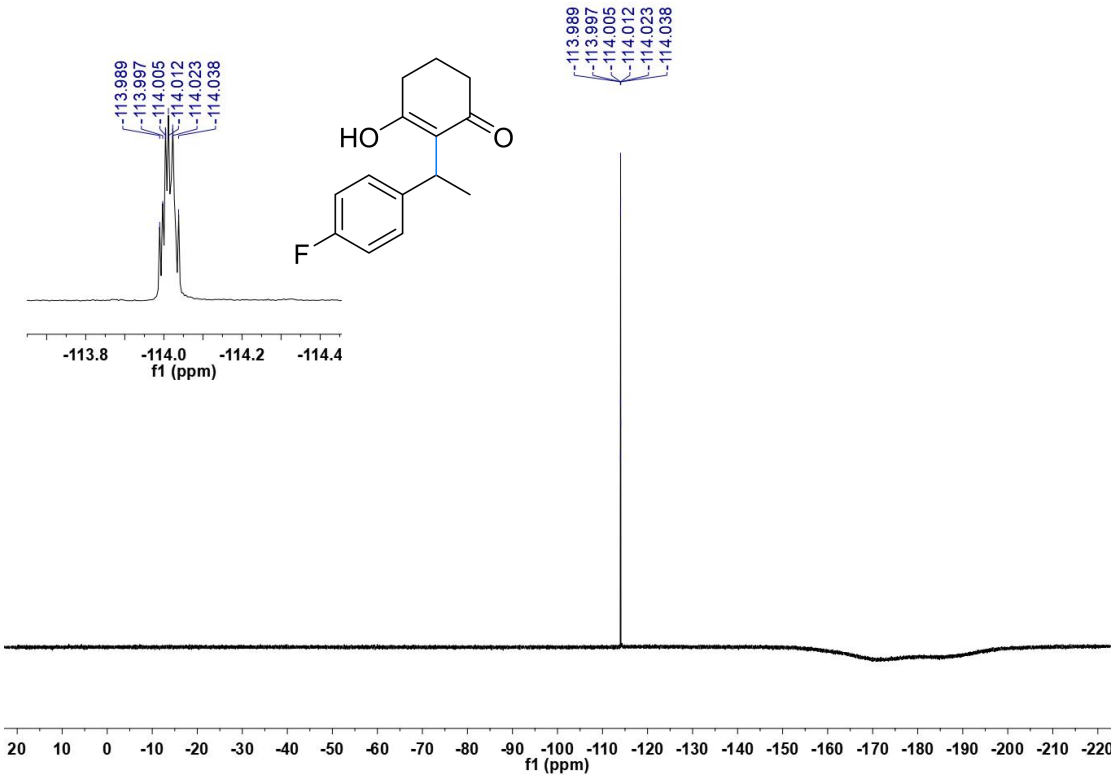
176.280
163.126
161.495
137.153
137.130
127.160
127.106
115.812
115.673
104.612
77.282
77.074
76.860
75.927
36.644
29.340
23.638
21.138
HO
O
F
f1 (ppm)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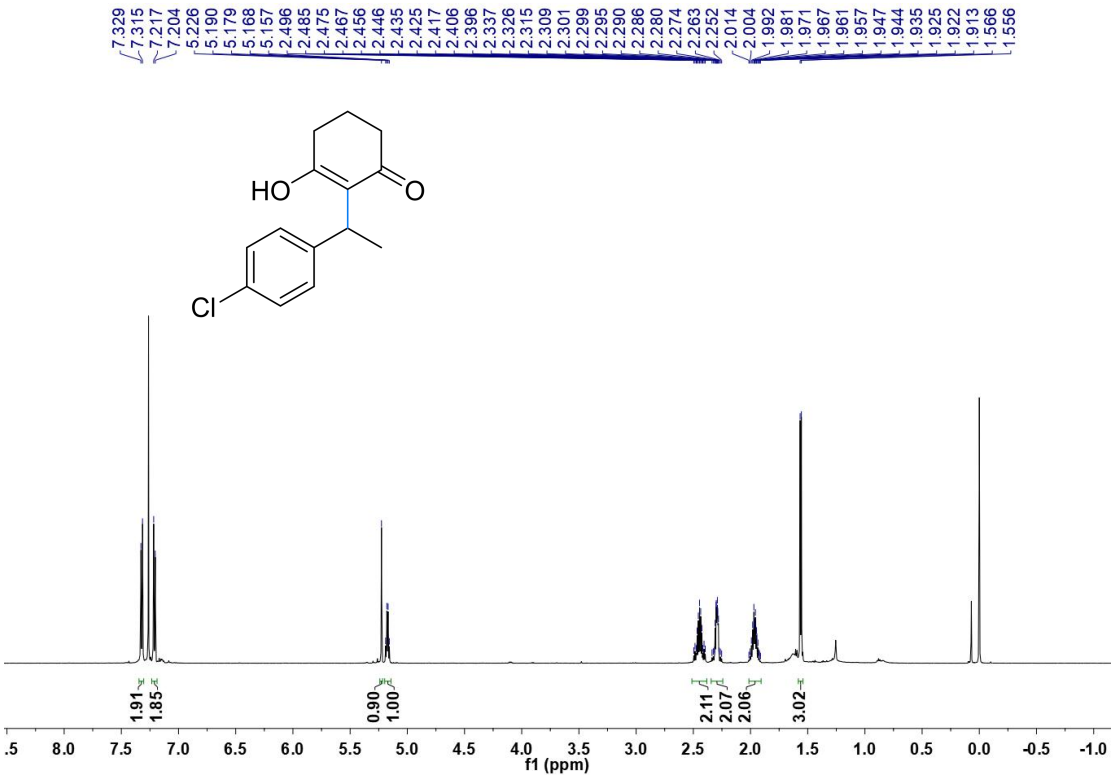
-113.997
-114.005
-114.012
-114.023
-114.038
HO
O
F
-113.8
-114.0
-114.2
-114.4
f1 (ppm)
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)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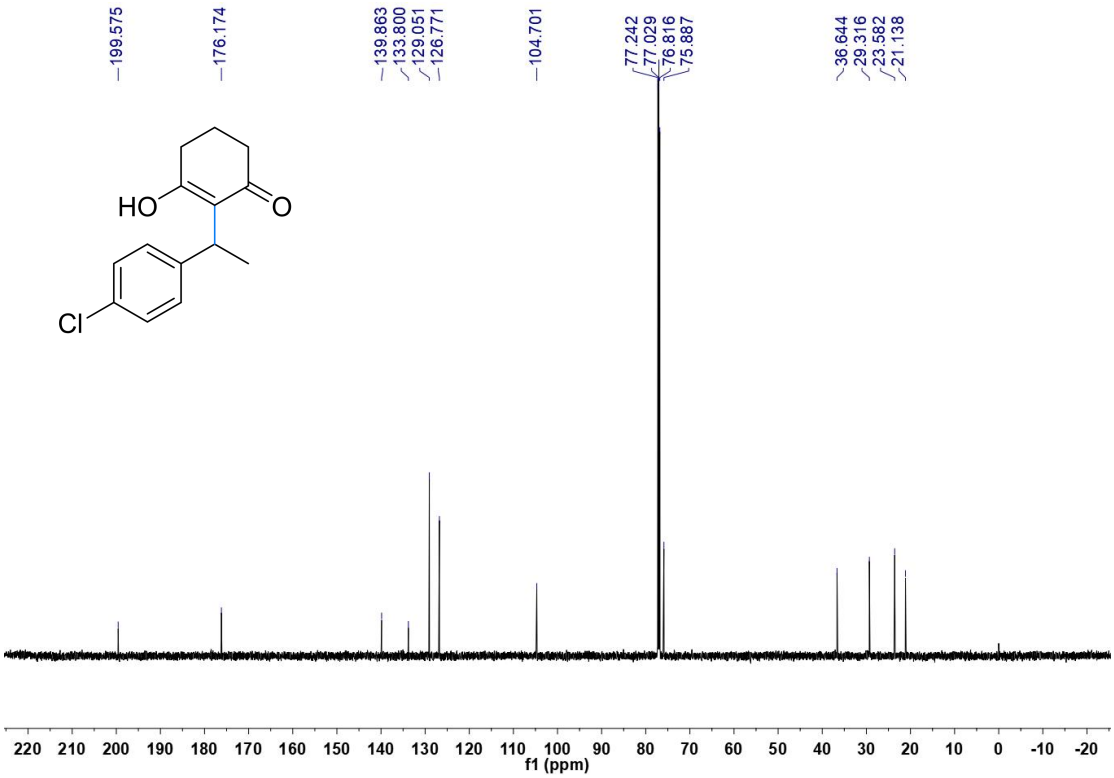
7.217
7.204
5.226
5.190
5.179
5.168
5.157
2.496
2.485
2.475
2.467
2.456
2.446
2.435
2.425
2.417
2.406
2.396
2.337
2.326
2.315
2.309
2.301
2.299
2.295
2.290
2.286
2.280
2.274
2.263
2.252
2.014
2.004
1.992
1.981
1.971
1.967
1.961
1.957
1.947
1.944
1.935
1.925
1.922
1.913
1.566
1.556
HO
O
Cl
1.91
1.85
0.90
1.00
2.11
2.07
2.06
3.02
.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
f1 (ppm)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139.863
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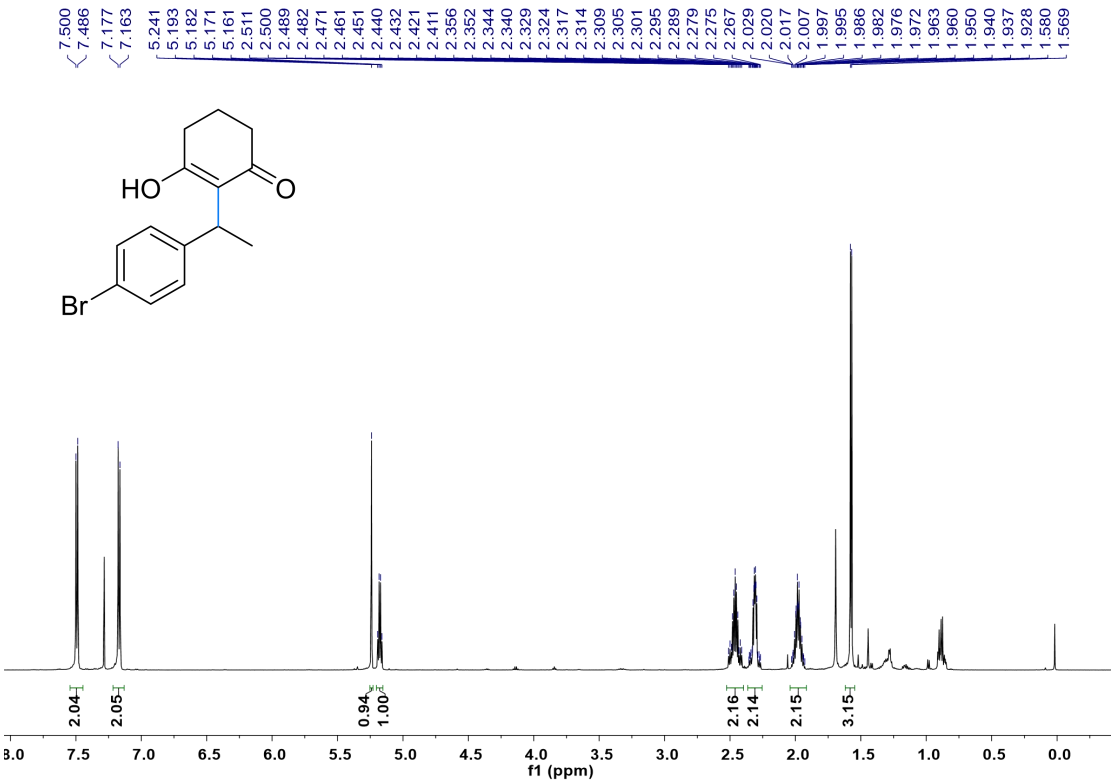
129.051
126.771
104.701
77.242
77.029
76.816
75.887
36.644
29.316
23.582
21.138
HO
O
Cl
f1 (ppm)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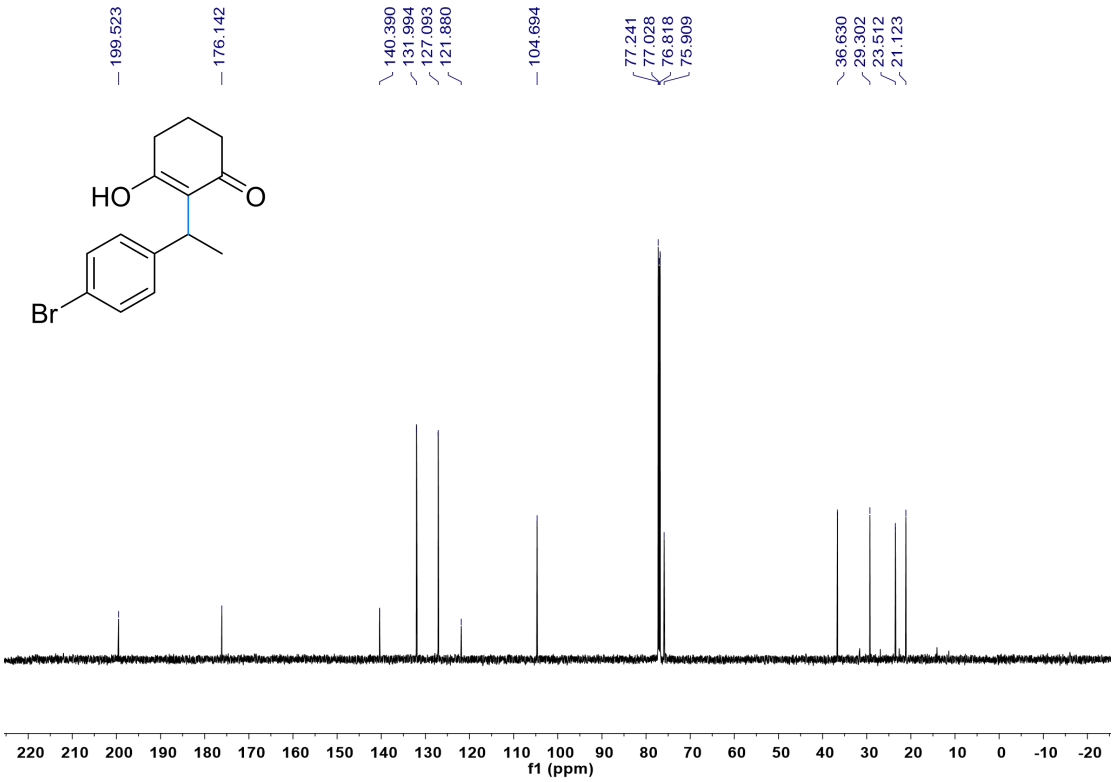
7.486
7.177
7.163
HO
O
Br
2.04
2.05
0.94
1.00
2.16
2.14
2.15
3.15
f1 (ppm)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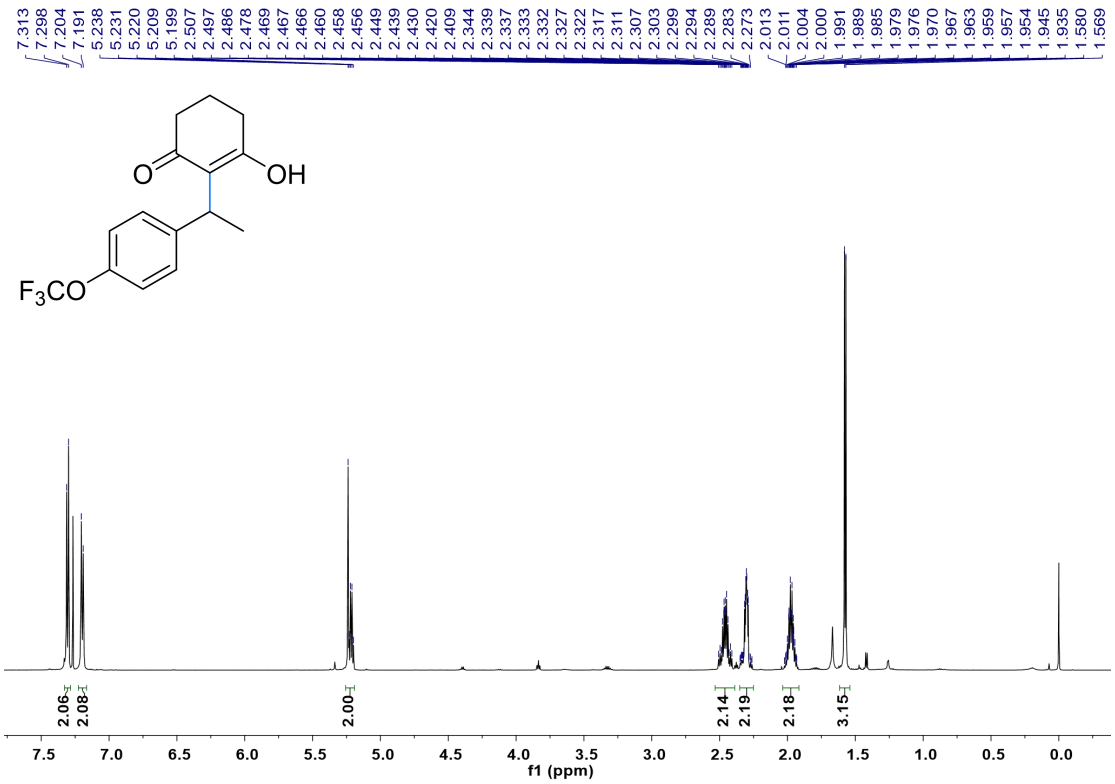
140.390
131.994
127.093
121.880
104.694
77.241
77.028
76.818
75.909
36.630
29.302
23.512
21.123
HO
O
Br
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)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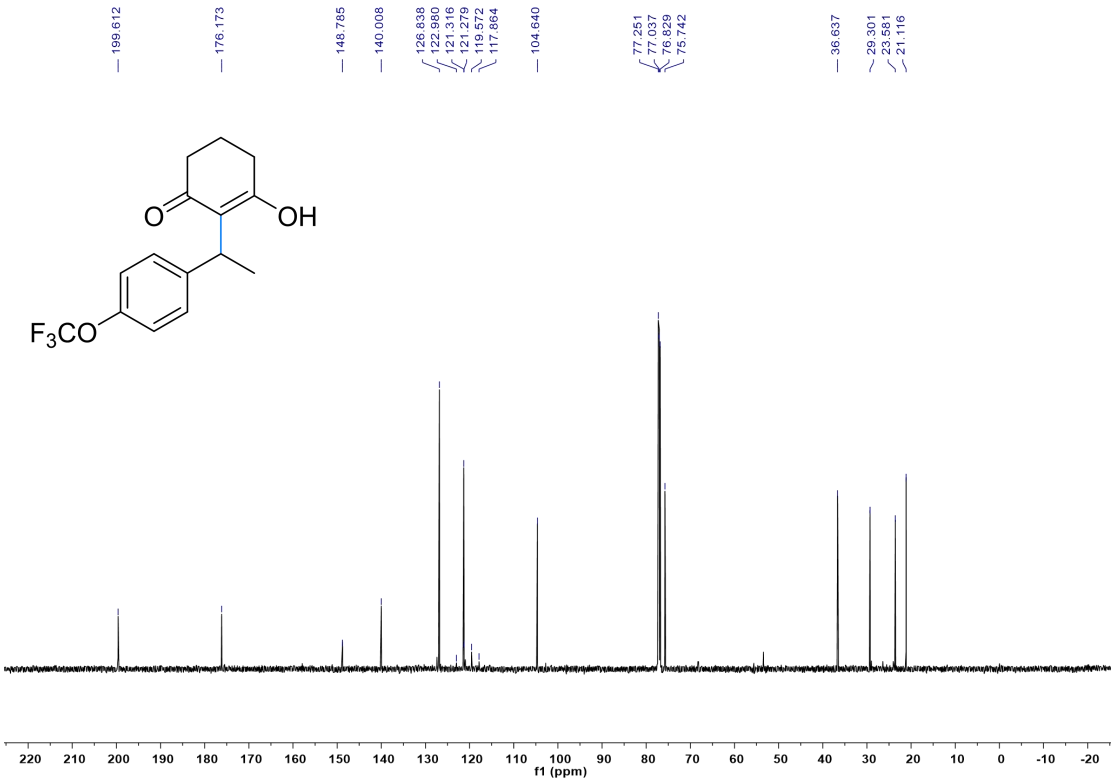
5.231
5.220
5.209
5.199
2.507
2.497
2.486
2.478
2.469
2.467
2.466
2.460
2.458
2.456
2.449
2.439
2.430
2.420
2.409
2.344
2.339
2.337
2.333
2.332
2.327
2.322
2.317
2.311
2.307
2.303
2.299
2.294
2.289
2.283
2.273
2.013
2.011
2.004
2.000
1.991
1.989
1.985
1.979
1.976
1.970
1.967
1.963
1.959
1.957
1.954
1.945
1.935
1.580
1.569
O
OH
F3CO
2.06
2.08
2.00
2.14
2.19
2.18
3.15
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)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117.864
104.640
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77.037
76.829
75.742
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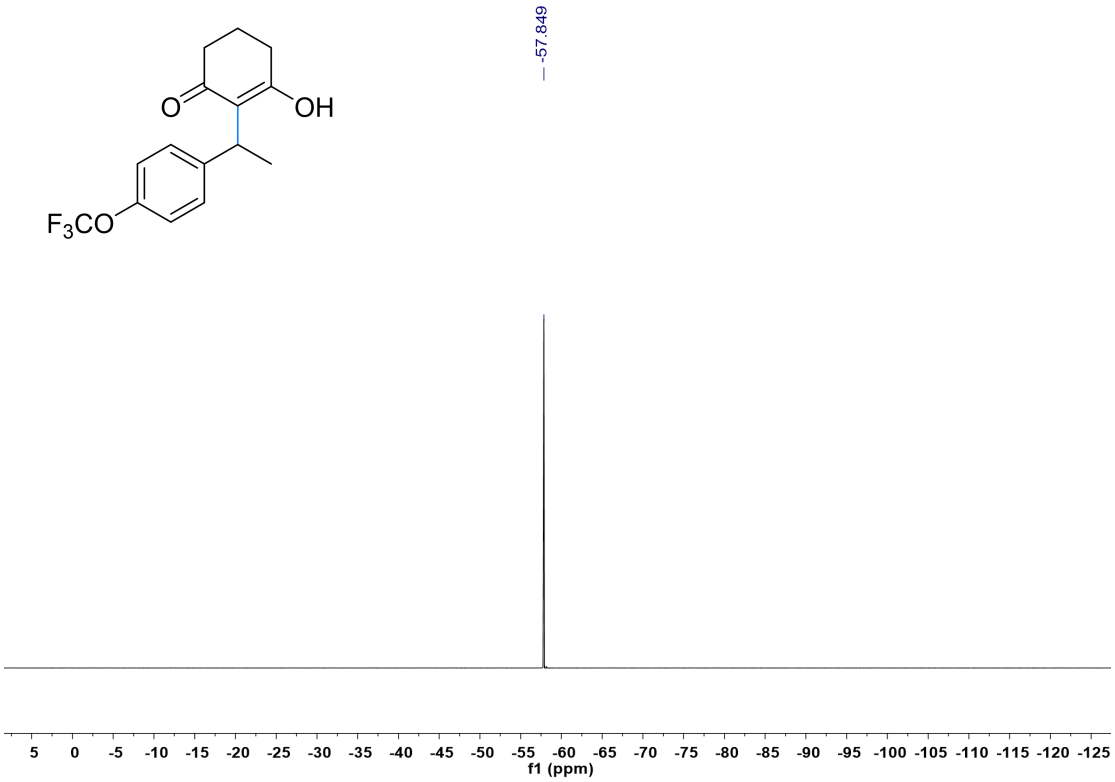
23.581
21.116
O
OH
F3CO
f1 (ppm)


-57.849
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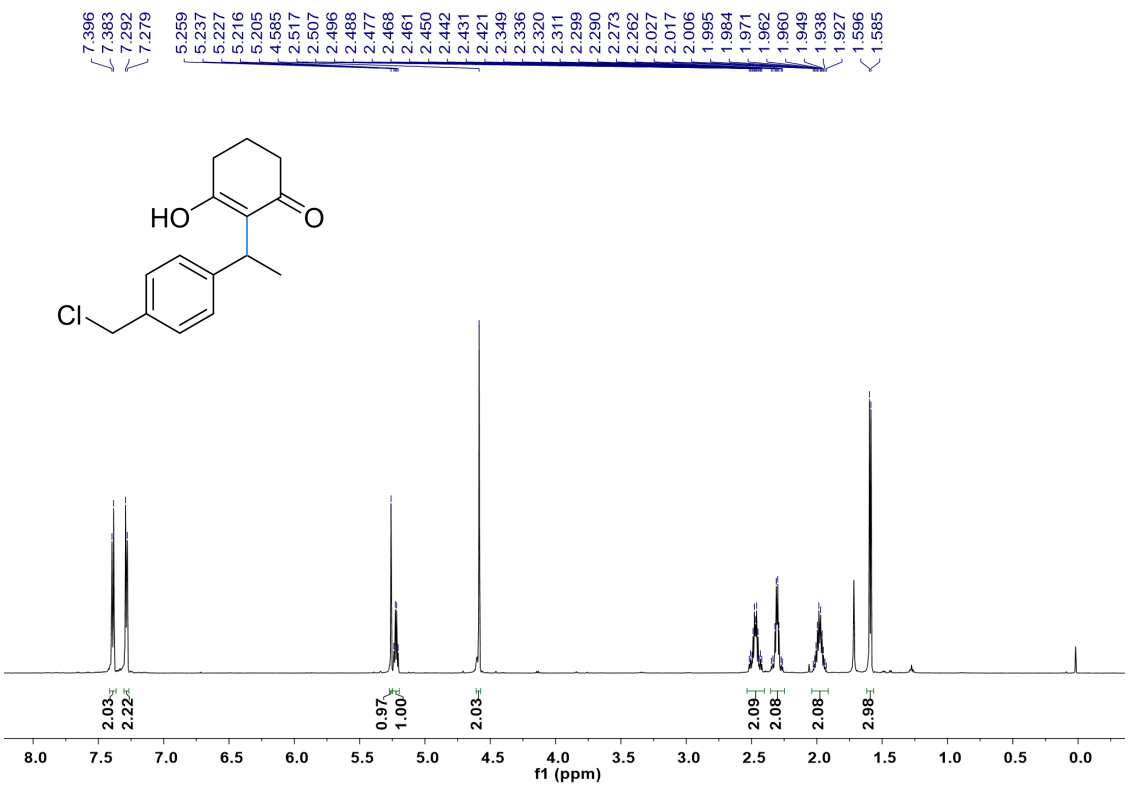
OH
F3CO
f1 (ppm)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5.227
5.216
5.205
4.585
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2.507
2.496
2.488
2.477
2.468
2.461
2.450
2.442
2.431
2.421
2.349
2.336
2.320
2.311
2.299
2.290
2.273
2.262
2.027
2.017
2.006
1.995
1.984
1.971
1.962
1.960
1.949
1.938
1.927
1.596
1.585
HO
O
Cl
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2.22
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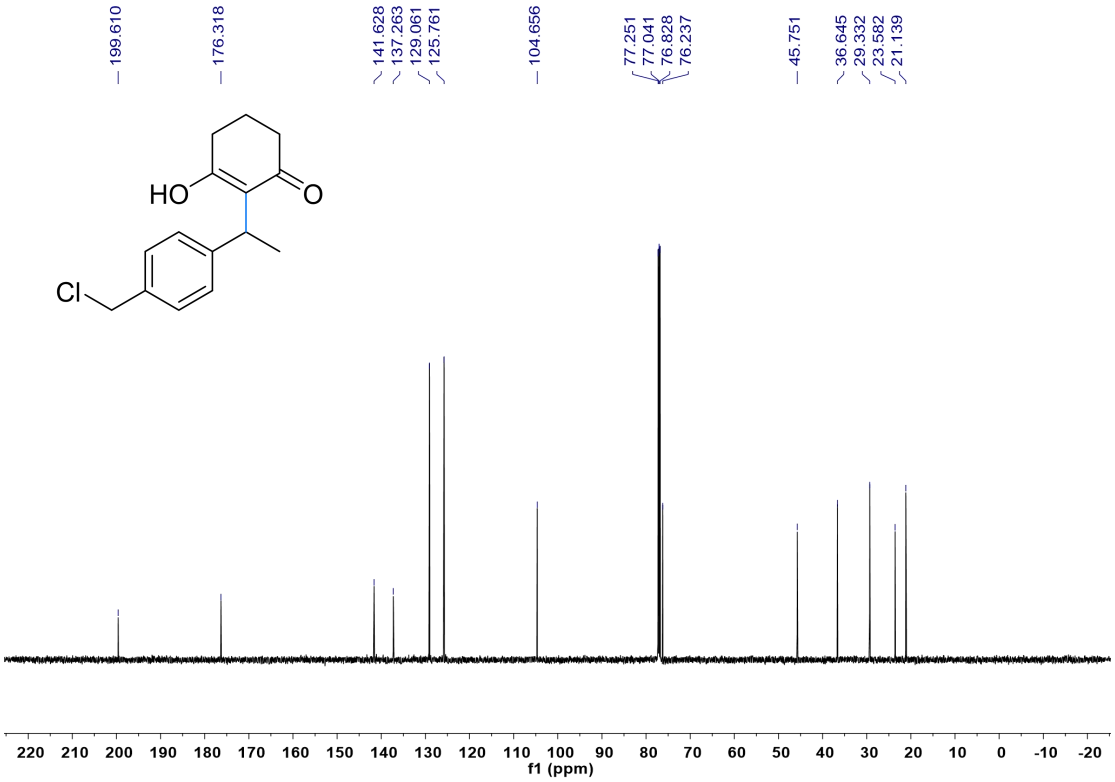
1.00
2.03
2.09
2.08
2.08
2.98
f1 (ppm)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23.582
21.139
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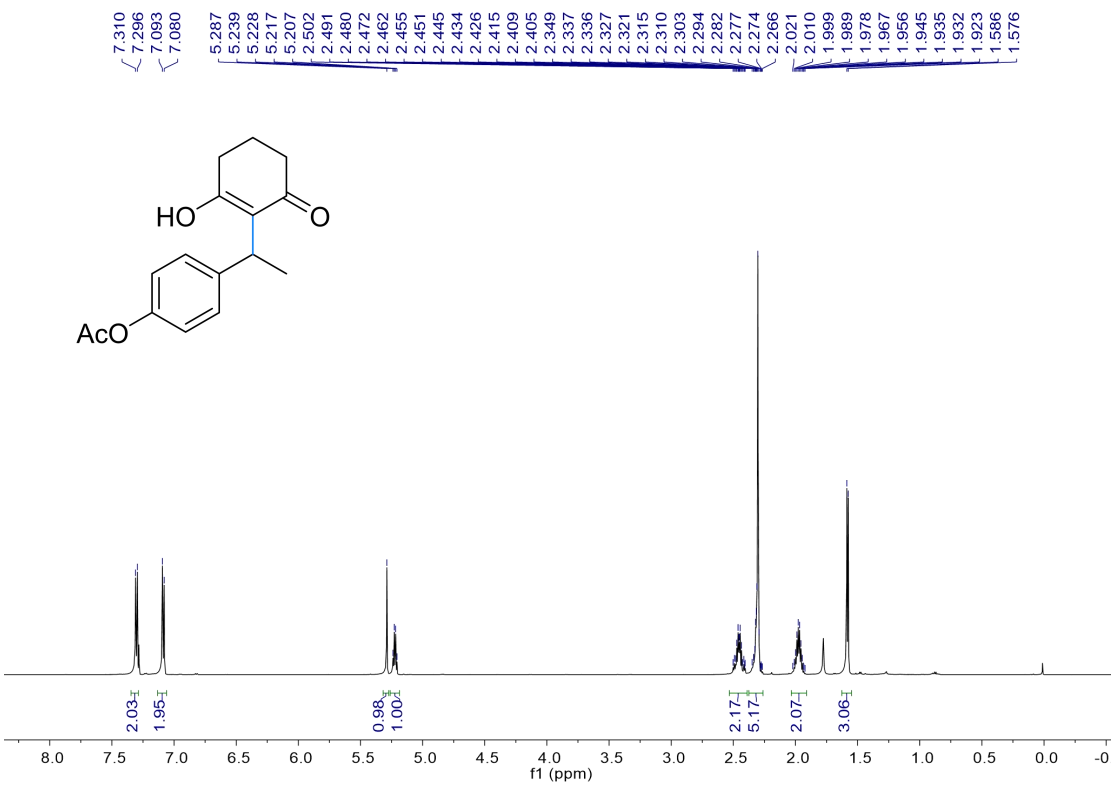
O
Cl
f1 (ppm)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5.228
5.217
5.207
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2.491
2.480
2.472
2.462
2.455
2.451
2.445
2.434
2.426
2.415
2.409
2.405
2.349
2.337
2.336
2.327
2.321
2.315
2.310
2.303
2.294
2.282
2.277
2.274
2.266
2.021
2.010
1.999
1.989
1.978
1.967
1.956
1.945
1.935
1.932
1.923
1.586
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HO
O
AcO
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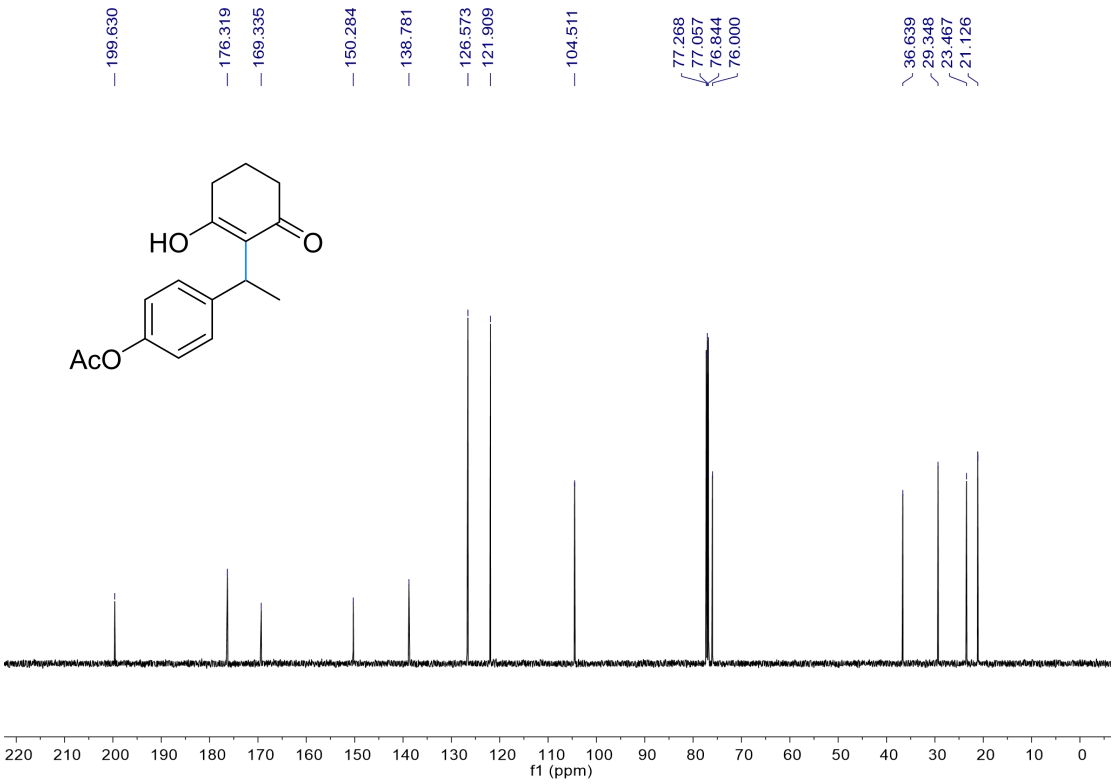
5.17
2.07
3.06
f1 (ppm)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121.909
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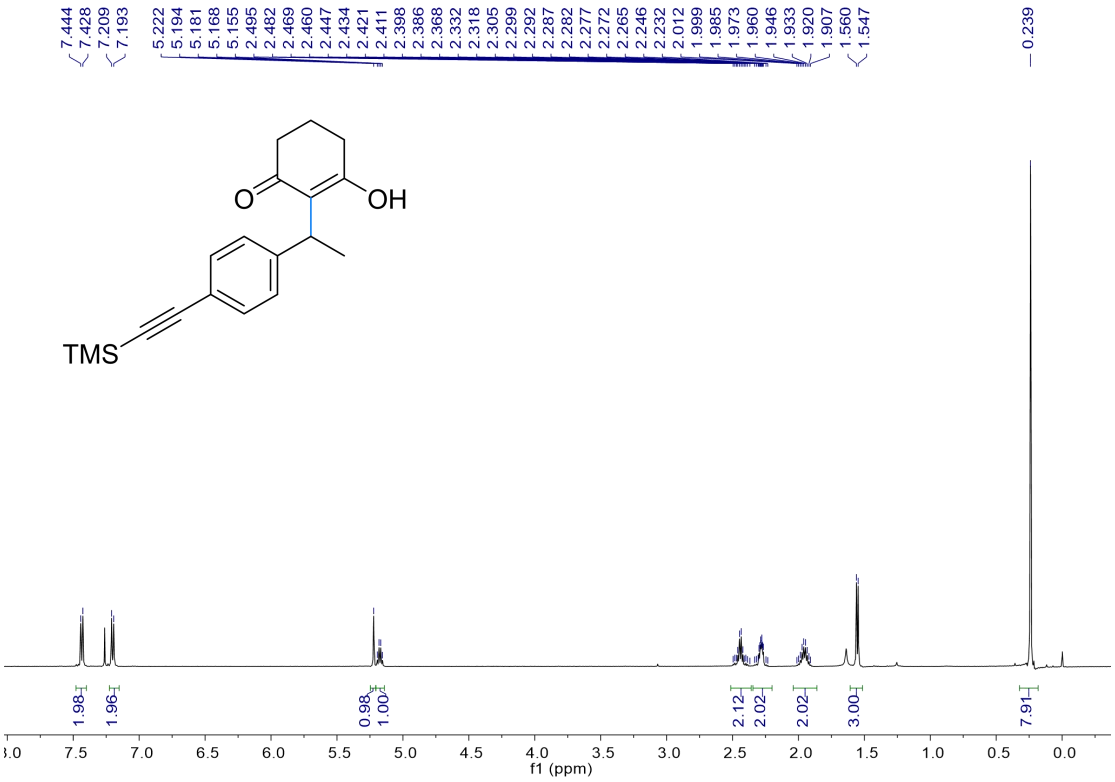
76.000
36.639
29.348
23.467
21.126
HO
O
AcO
f1 (ppm)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5.168
5.155
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2.469
2.460
2.447
2.434
2.421
2.411
2.398
2.386
2.368
2.332
2.318
2.305
2.299
2.292
2.287
2.282
2.277
2.272
2.265
2.246
2.232
2.012
1.999
1.985
1.973
1.960
1.946
1.933
1.920
1.907
1.560
1.547
0.239
O
OH
TMS
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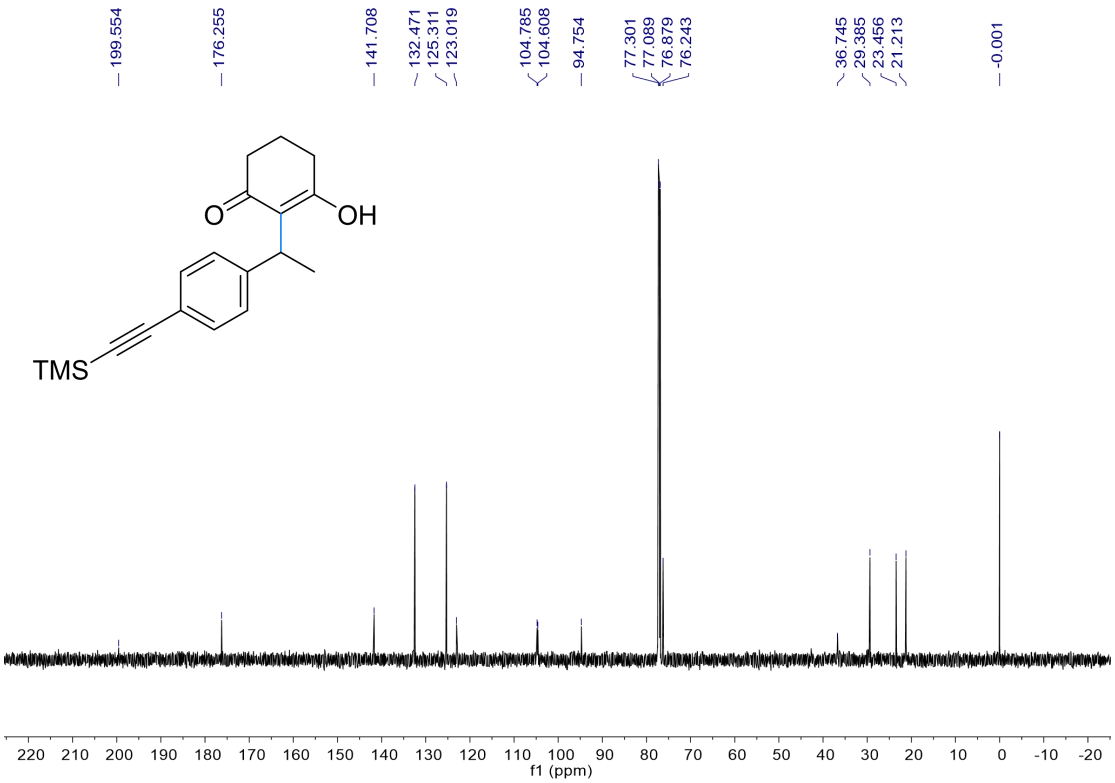
1.96
0.98
1.00
2.12
2.02
2.02
3.00
7.91
f1 (ppm)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104.608
94.754
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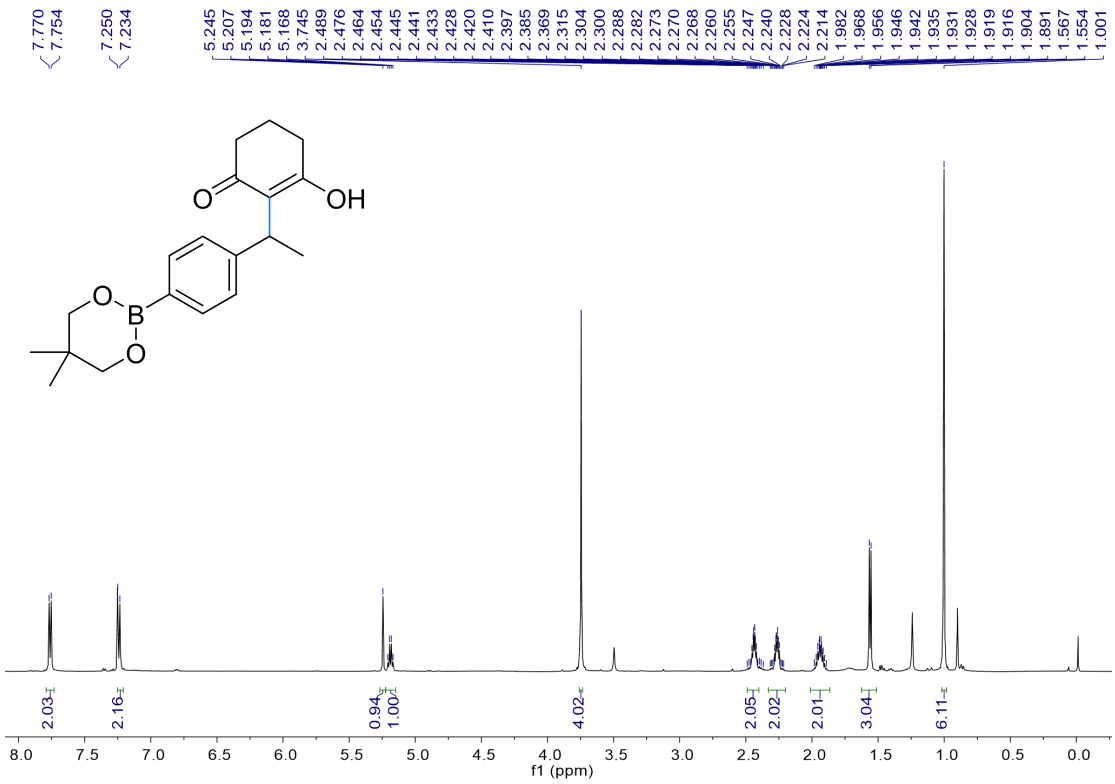
76.243
36.745
29.385
23.456
21.213
-0.001
O
OH
TMS
f1 (ppm)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5.168
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2.428
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2.315
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2.300
2.288
2.282
2.273
2.270
2.268
2.260
2.255
2.247
2.240
2.228
2.224
2.214
1.982
1.968
1.956
1.946
1.942
1.935
1.931
1.928
1.919
1.916
1.904
1.891
1.567
1.554
1.001
O
OH
O
B
O
2.03
2.16
0.94
1.00
4.02
2.05
2.02
2.01
3.04
6.11
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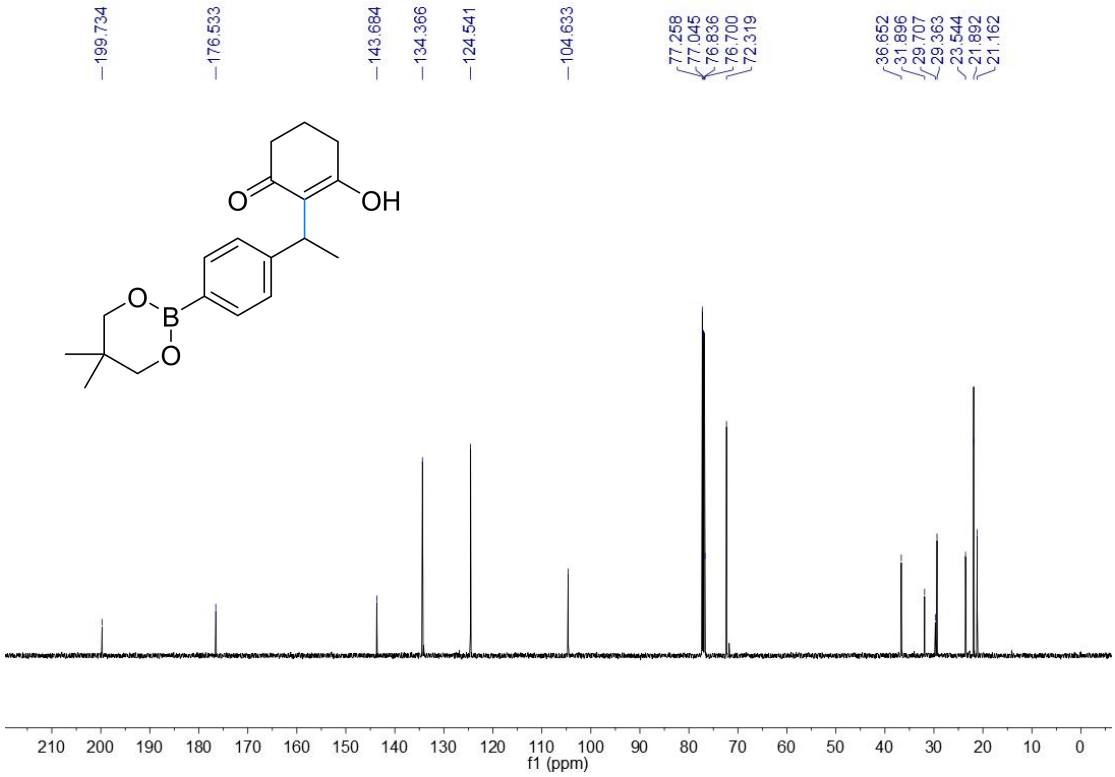
f1 (ppm)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31.896
29.707
29.363
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O
OH
O
B
O
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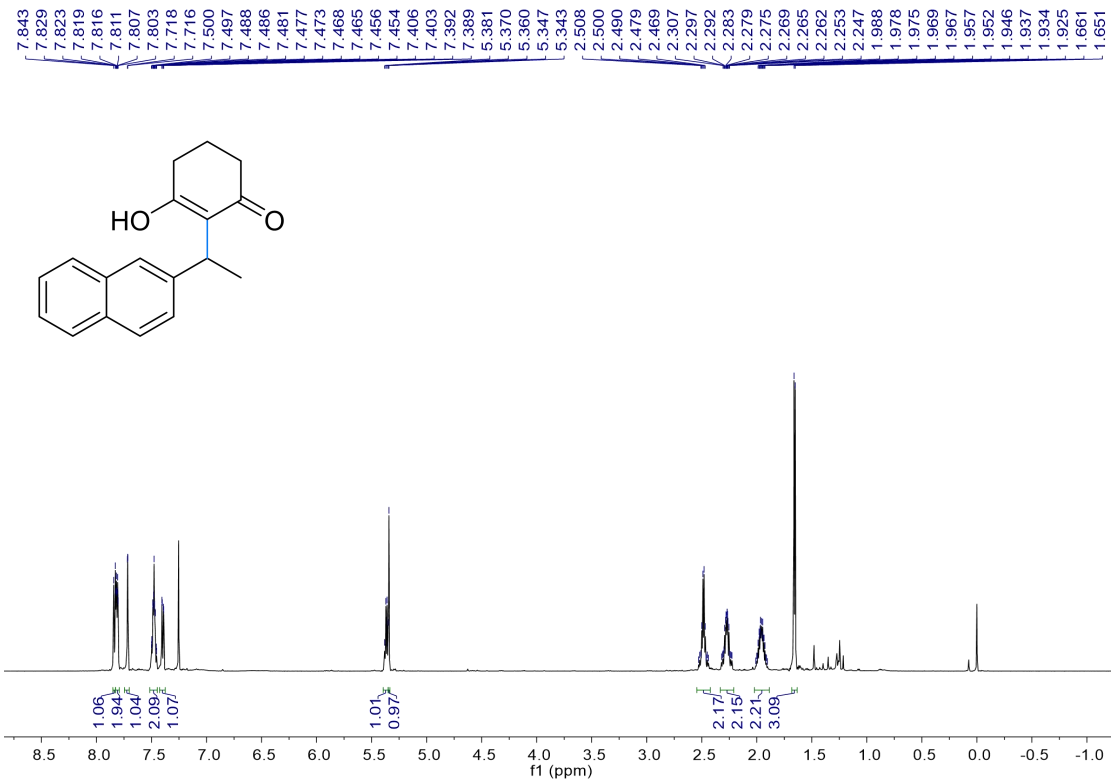
f1 (ppm)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7.481
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7.473
7.468
7.465
7.456
7.454
7.406
7.403
7.392
7.389
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5.370
5.360
5.347
5.343
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2.490
2.479
2.469
2.307
2.297
2.292
2.283
2.279
2.275
2.269
2.265
2.262
2.253
2.247
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1.978
1.975
1.969
1.967
1.957
1.952
1.946
1.937
1.934
1.925
1.661
1.651
HO
O
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1.01
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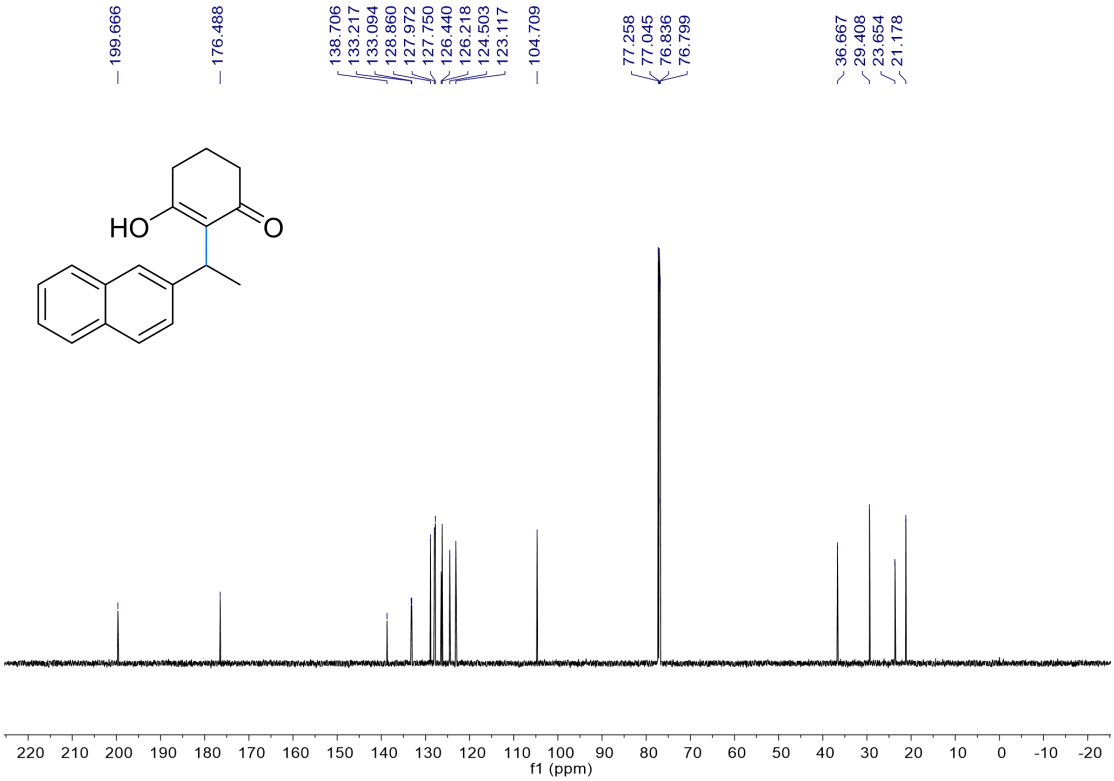
3.09
f1 (ppm)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23.654
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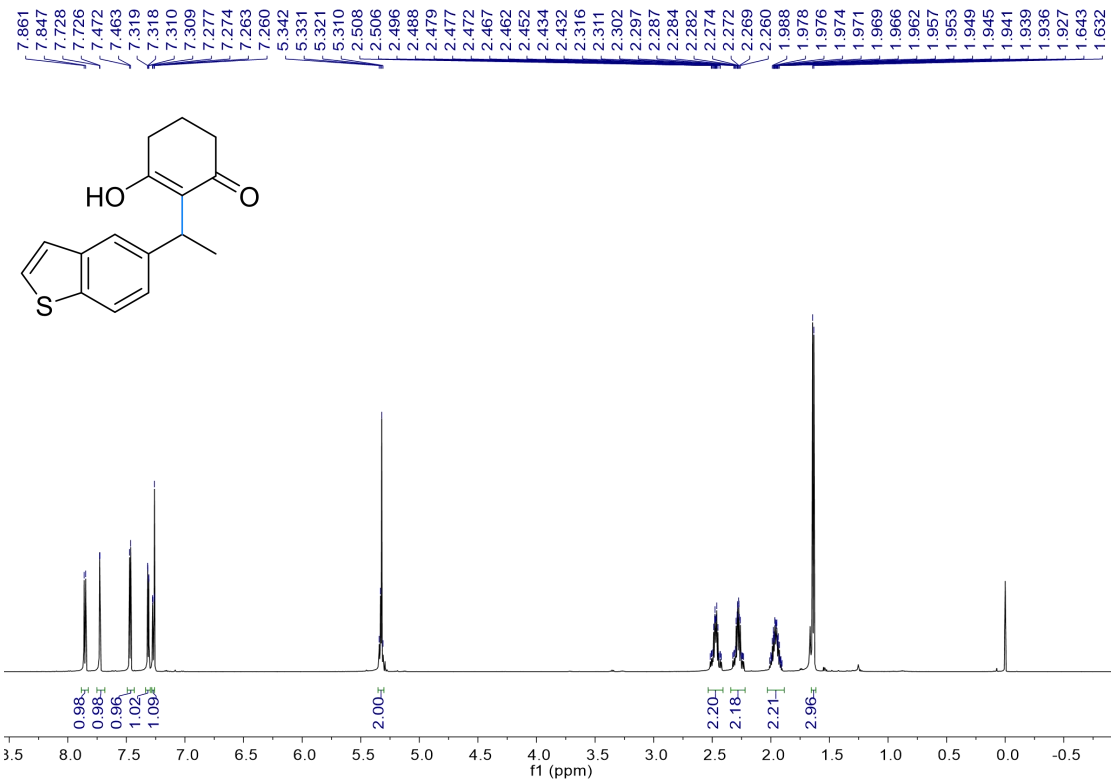
HO
O
f1 (ppm)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2.496
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2.287
2.284
2.282
2.274
2.272
2.269
2.260
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1.976
1.974
1.971
1.969
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1.962
1.957
1.953
1.949
1.945
1.941
1.939
1.936
1.927
1.643
1.632
HO
O
S
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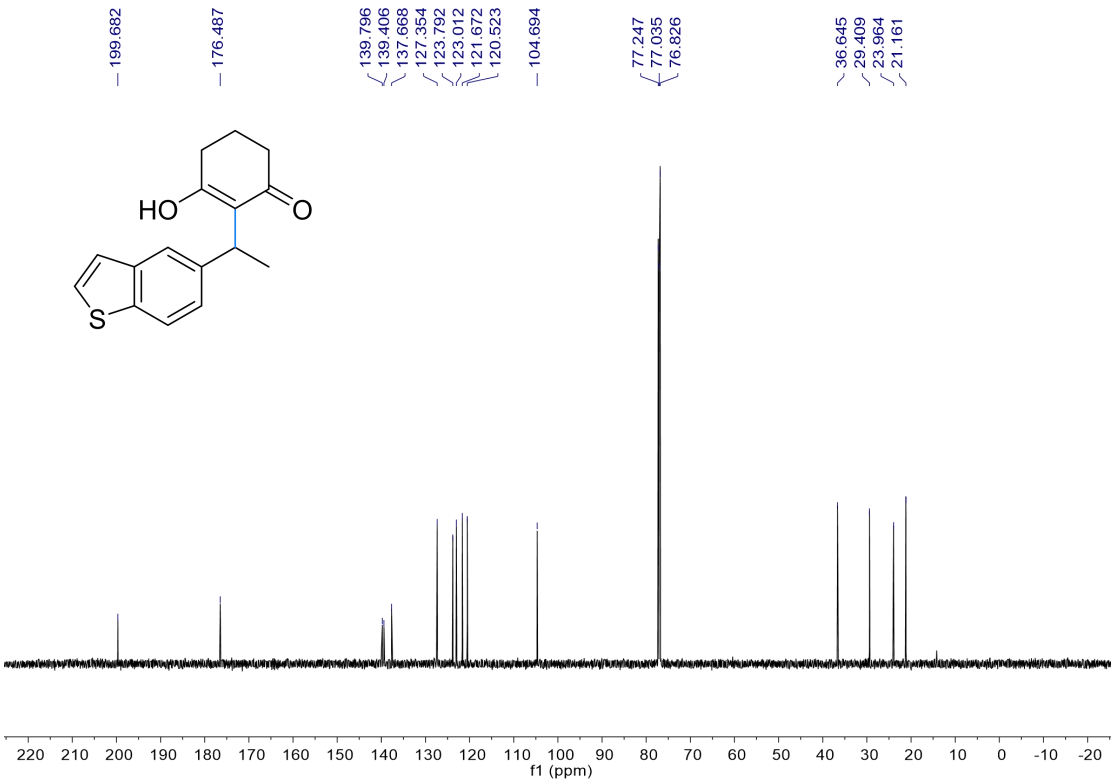
0.96
1.02
1.09
2.00
2.20
2.18
2.21
2.96
f1 (ppm)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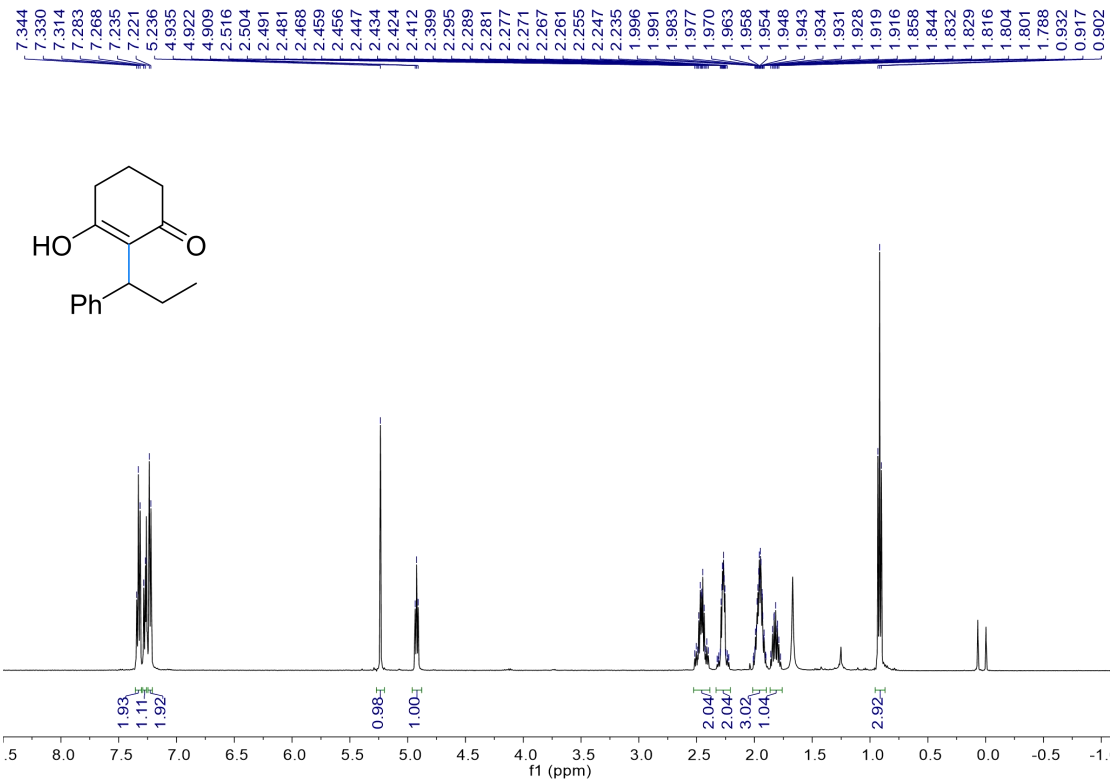
HO
O
S
f1 (ppm)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2.504
2.491
2.481
2.468
2.459
2.456
2.447
2.434
2.424
2.412
2.399
2.295
2.289
2.281
2.277
2.271
2.267
2.261
2.255
2.247
2.235
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1.991
1.983
1.977
1.970
1.963
1.958
1.954
1.948
1.943
1.934
1.931
1.928
1.919
1.916
1.858
1.844
1.832
1.829
1.816
1.804
1.801
1.788
0.932
0.917
0.902
HO
O
Ph
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1.92
0.98
1.00
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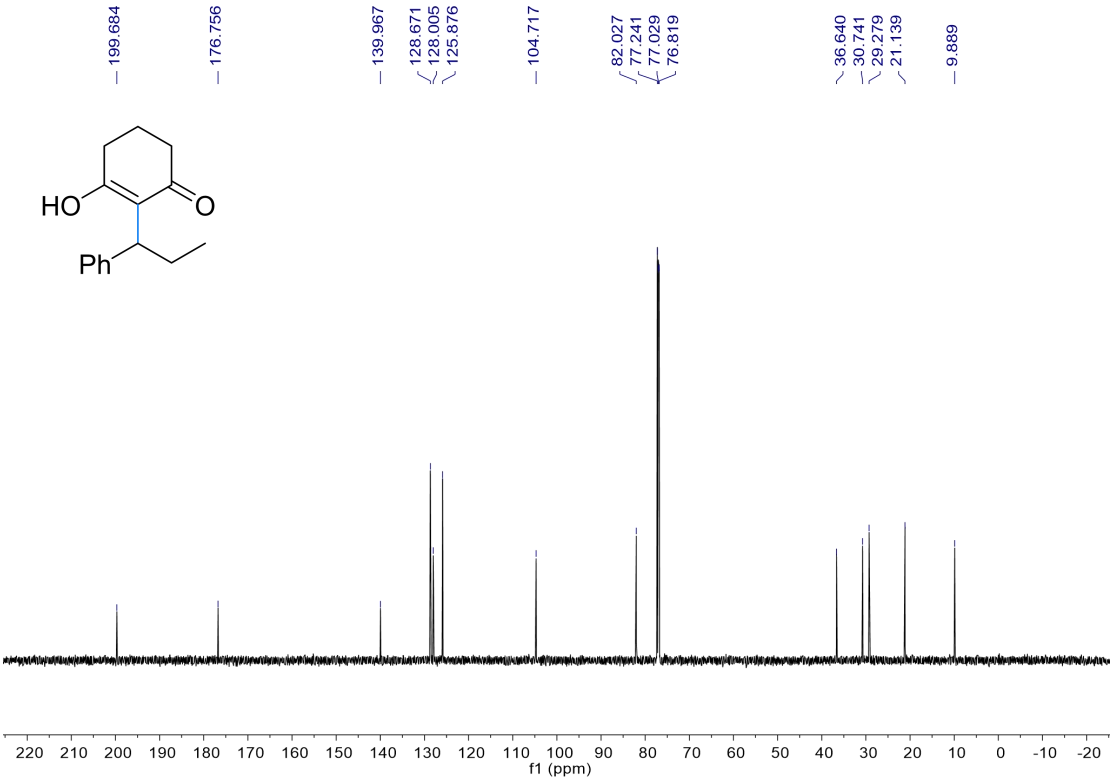
2.04
3.02
1.04
2.92
f1 (ppm)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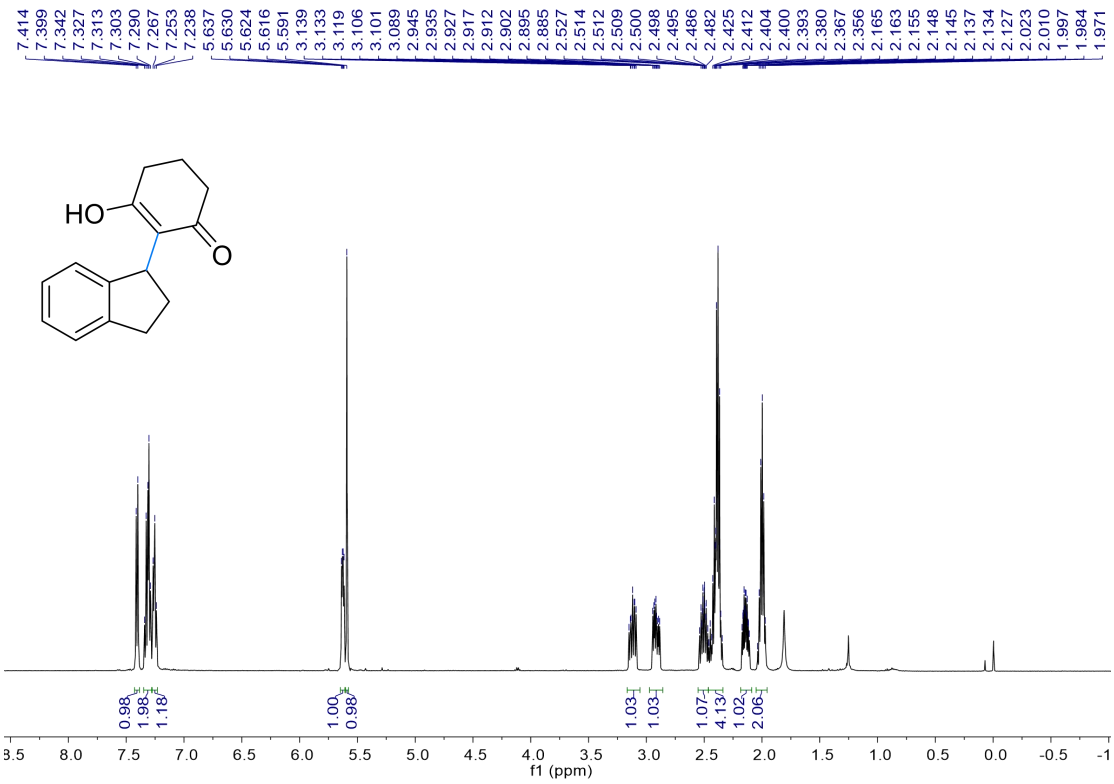
9.889
HO
O
Ph
f1 (ppm)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7.267
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7.238
5.637
5.630
5.624
5.616
5.591
3.139
3.133
3.119
3.106
3.101
3.089
2.945
2.935
2.927
2.917
2.912
2.902
2.895
2.885
2.527
2.514
2.512
2.509
2.500
2.498
2.495
2.486
2.482
2.425
2.412
2.404
2.400
2.393
2.380
2.367
2.356
2.165
2.163
2.155
2.148
2.145
2.137
2.134
2.127
2.023
2.010
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1.984
1.971
HO
O
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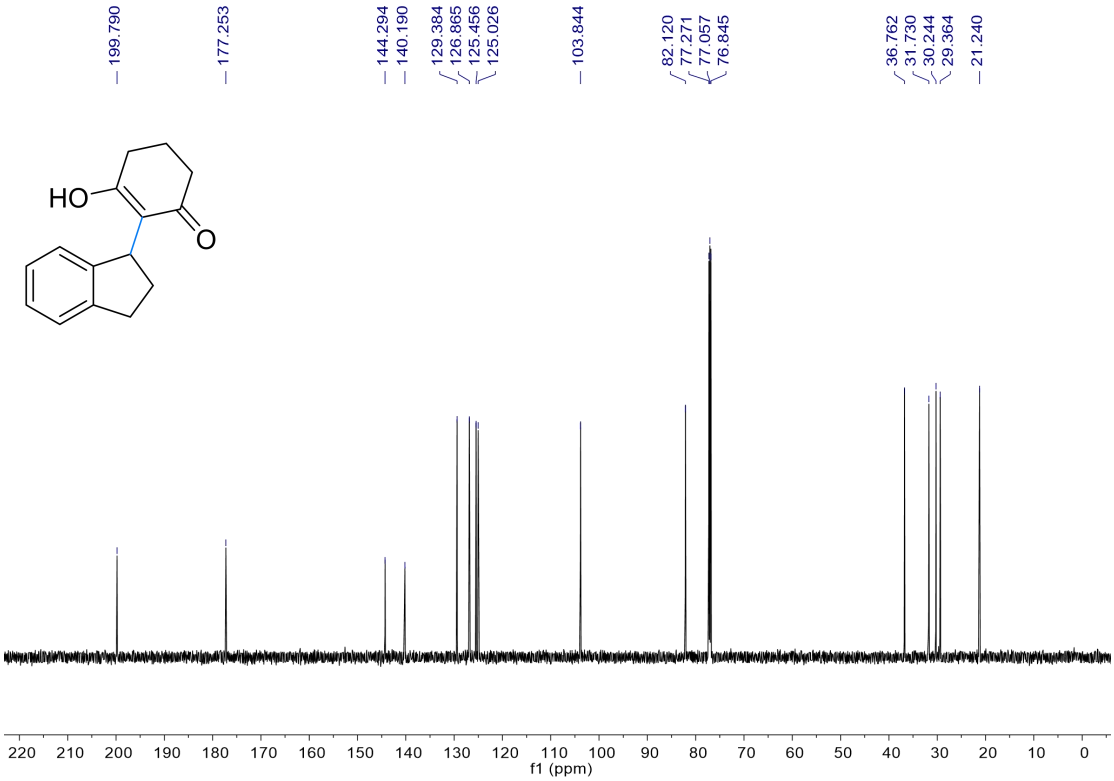
1.00
0.98
1.03
1.03
1.07
4.13
1.02
2.06
f1 (ppm)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125.026
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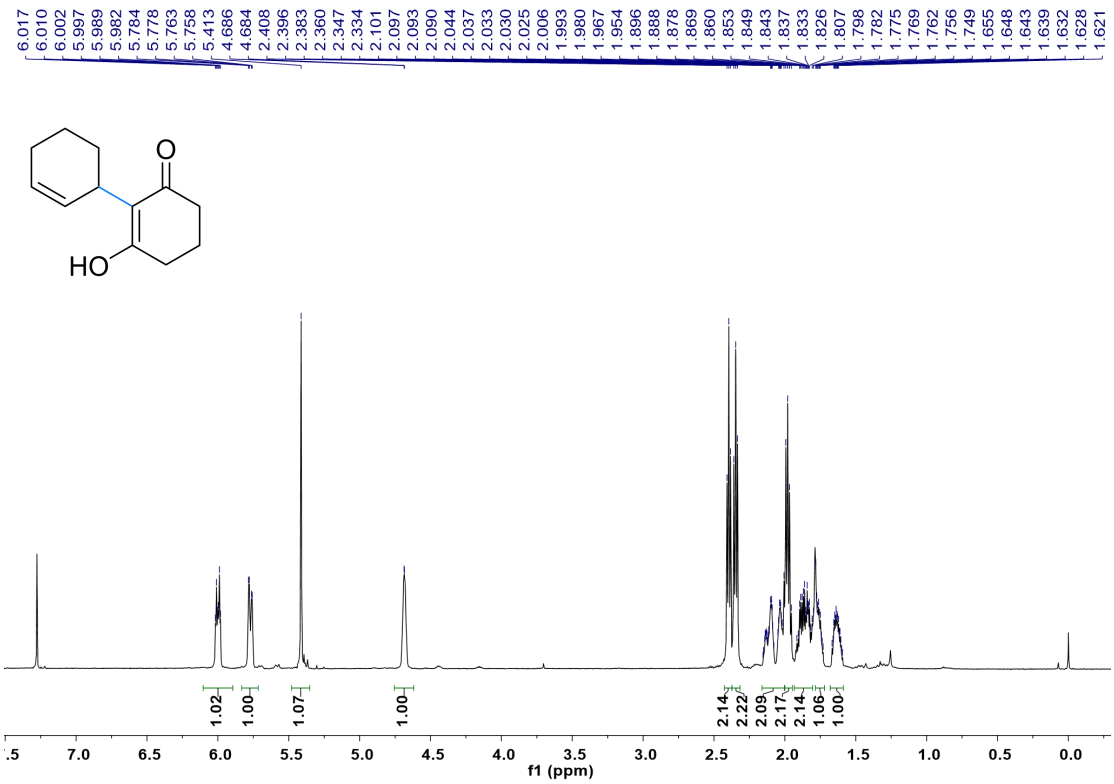
76.845
36.762
31.730
30.244
29.364
21.240
HO
O
f1 (ppm)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6.010
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4.684
2.408
2.396
2.383
2.360
2.347
2.334
2.101
2.097
2.093
2.090
2.044
2.037
2.033
2.030
2.025
2.006
1.993
1.980
1.967
1.954
1.896
1.888
1.878
1.869
1.860
1.853
1.849
1.843
1.837
1.833
1.826
1.807
1.798
1.782
1.775
1.769
1.762
1.756
1.749
1.655
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1.643
1.639
1.632
1.628
1.621
O
HO
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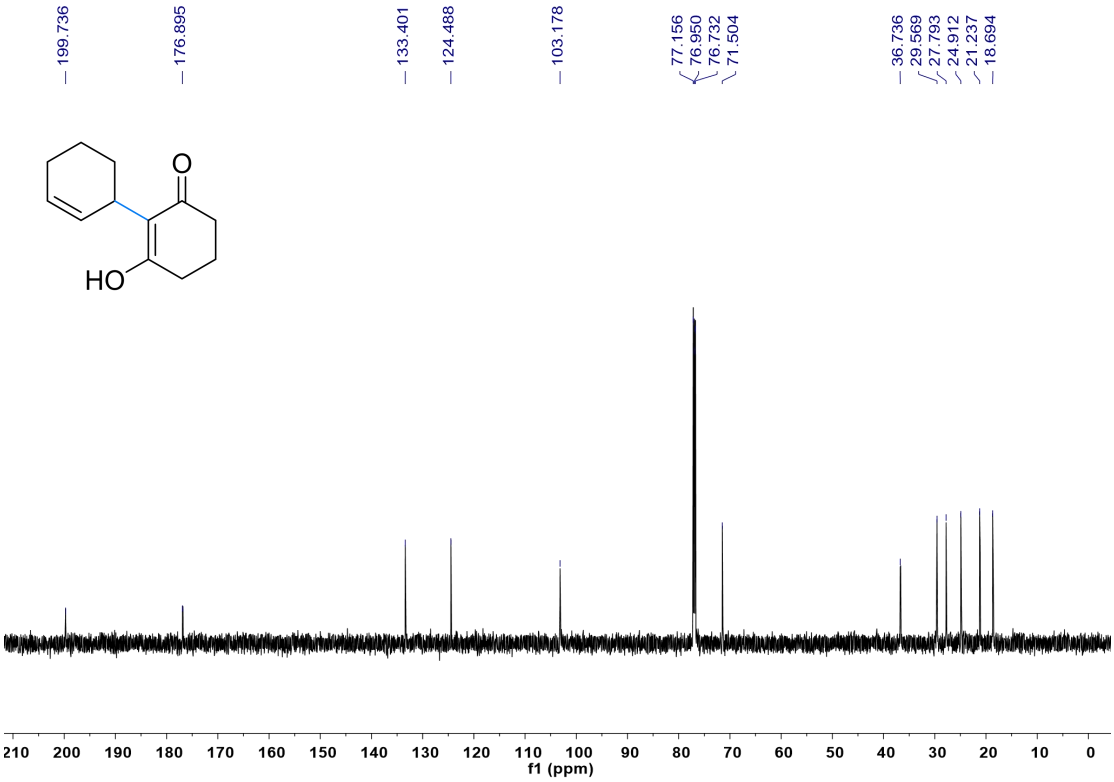
1.00
1.07
1.00
2.14
2.22
2.09
2.17
2.14
1.06
1.00
f1 (ppm)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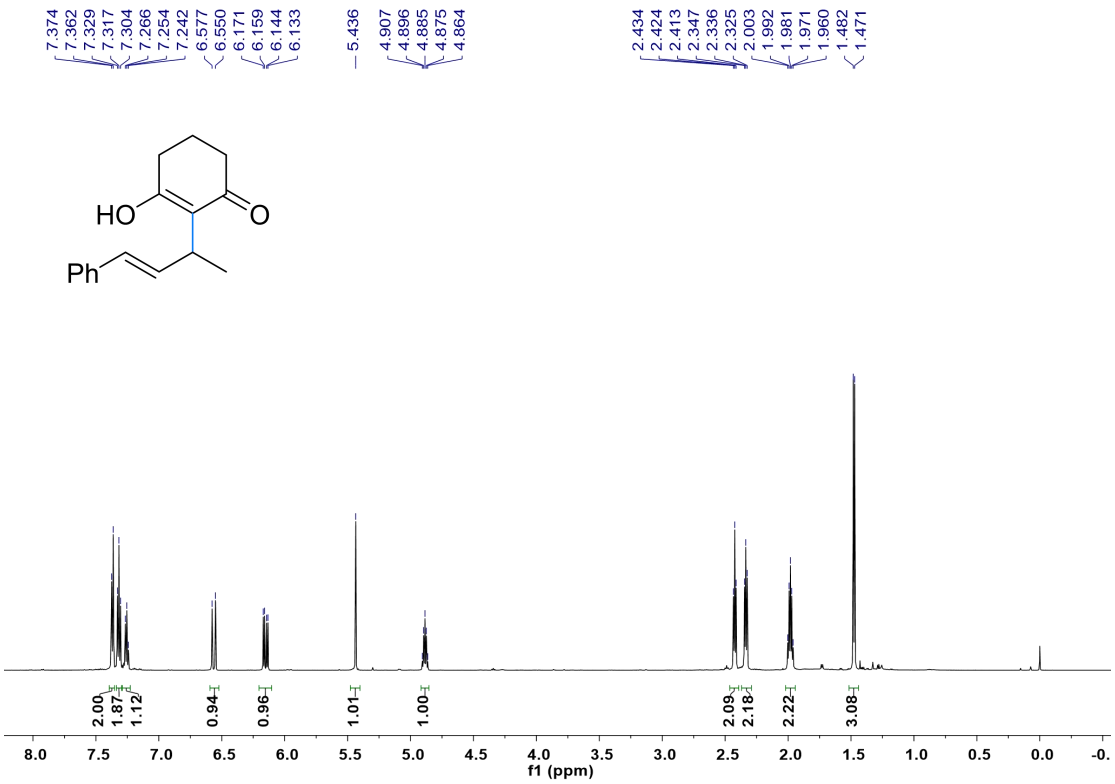
24.912
21.237
18.694
O
HO
f1 (ppm)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6.550
6.171
6.159
6.144
6.133
5.436
4.907
4.896
4.885
4.875
4.864
2.434
2.424
2.413
2.347
2.336
2.325
2.003
1.992
1.981
1.971
1.960
1.482
1.471
HO
O
Ph
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0.94
0.96
1.01
1.00
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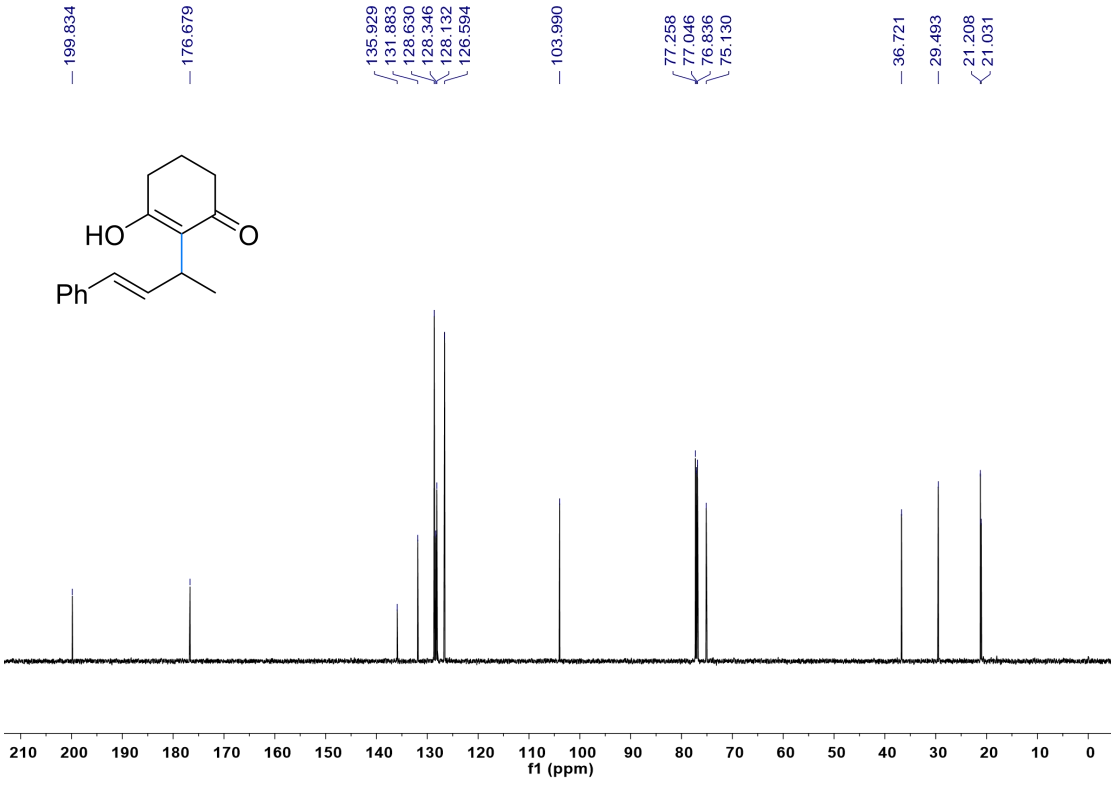
2.18
2.22
3.08
f1 (ppm)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128.132
126.594
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76.836
75.130
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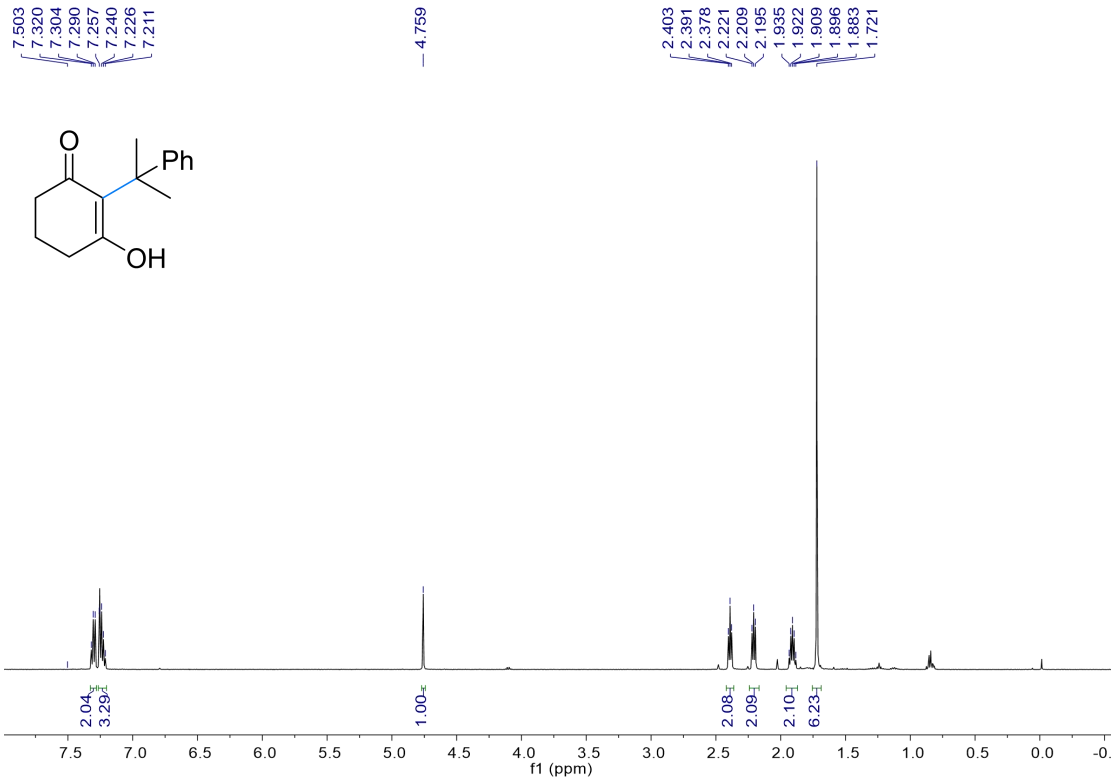
29.493
21.208
21.031
HO
O
Ph
f1 (ppm)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2.209
2.195
1.935
1.922
1.909
1.896
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1.721
O
Ph
OH
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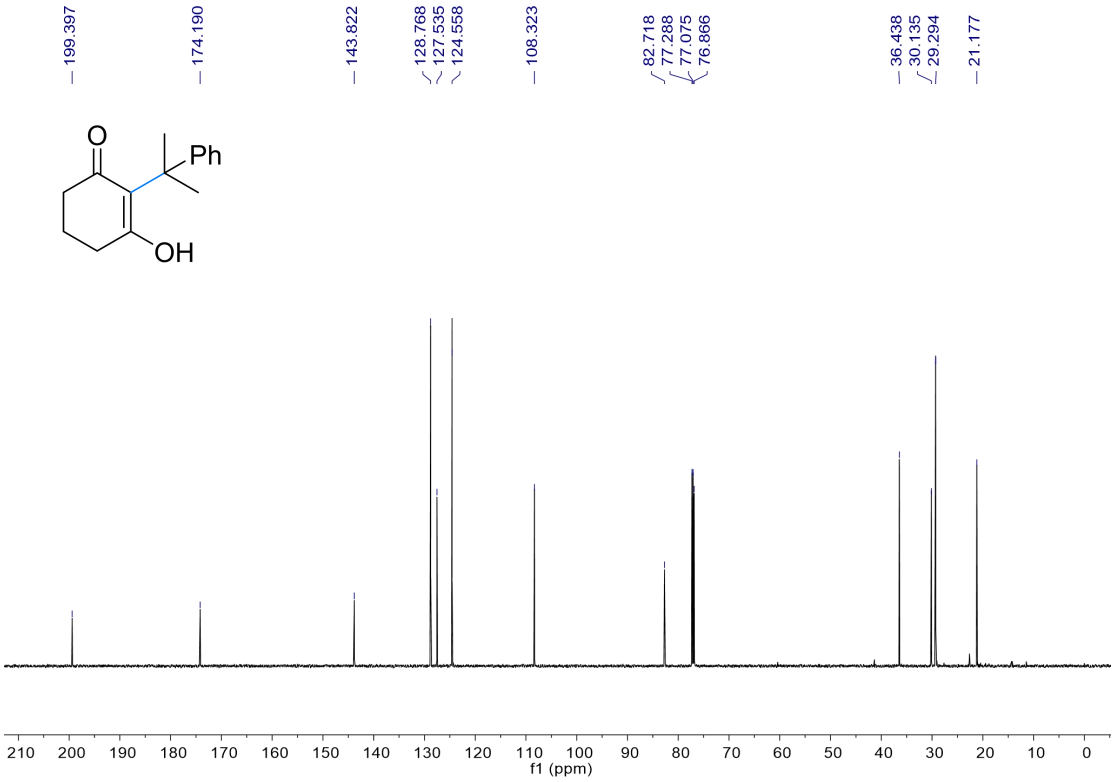
3.29
1.00
2.08
2.09
2.10
6.23
f1 (ppm)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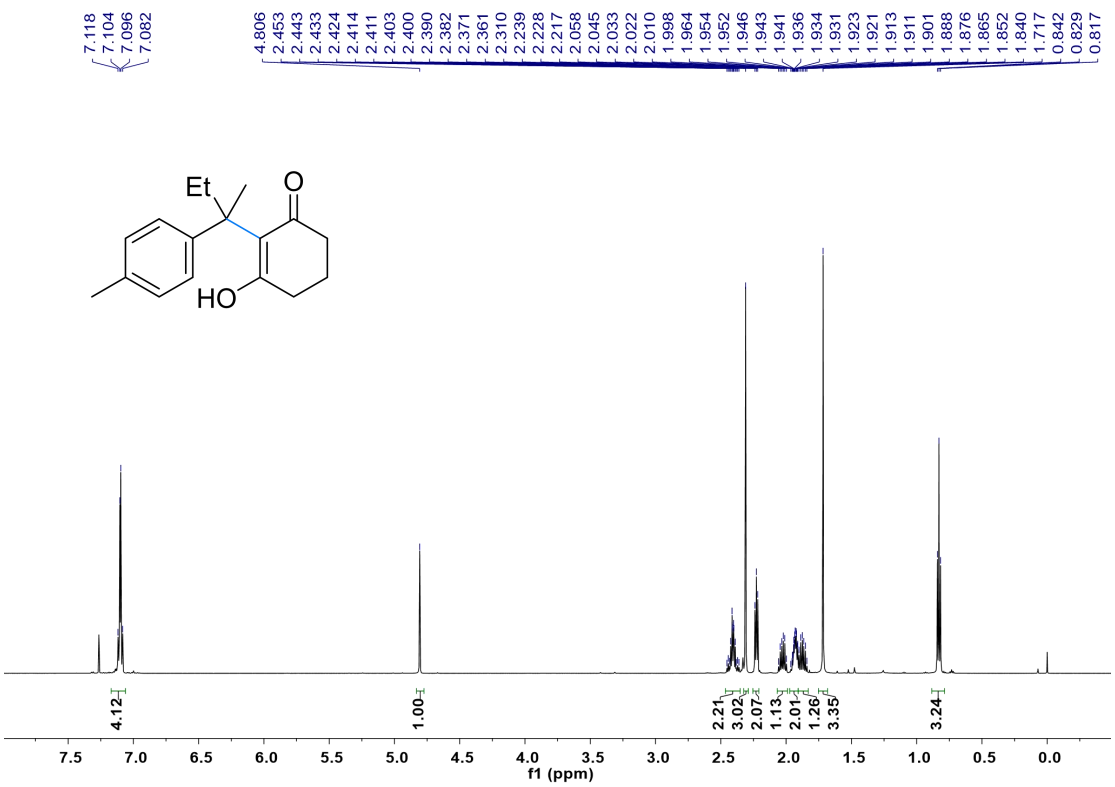
O
Ph
OH
f1 (ppm)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2.411
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2.310
2.239
2.228
2.217
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2.045
2.033
2.022
2.010
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1.954
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1.901
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1.876
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1.852
1.840
1.717
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0.829
0.817
Et
O
HO
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2.21
3.02
2.07
1.13
2.01
1.26
3.35
3.24
7.5
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6.5
6.0
5.5
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3.0
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1.0
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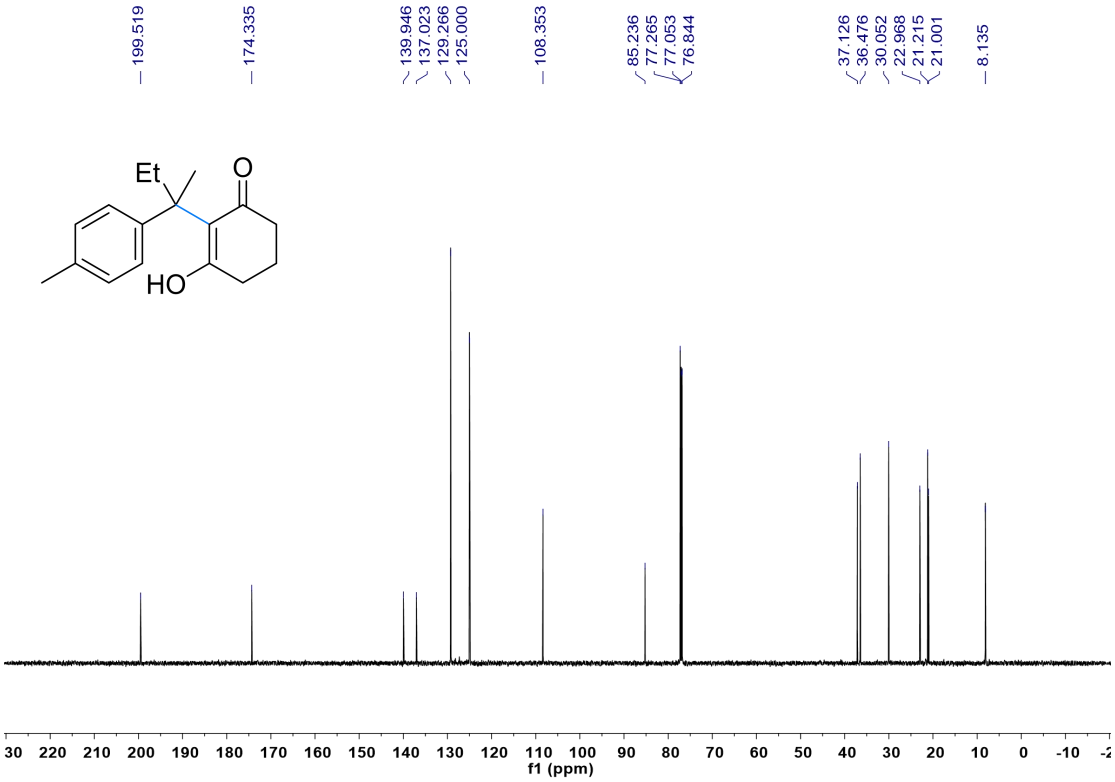
0.0
f1 (ppm)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36.476
30.052
22.968
21.215
21.001
8.135
Et
O
HO
30
220
210
200
190
180
170
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140
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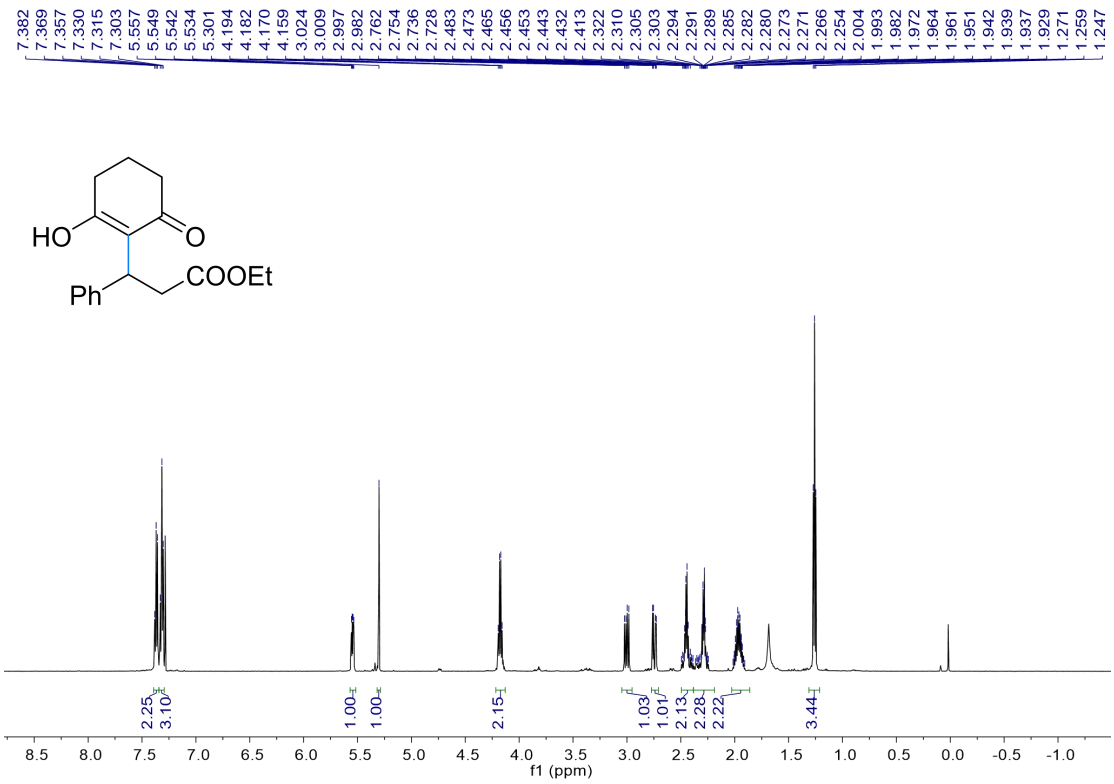
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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1.259
1.247
HO
O
COOEt
Ph
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1.00
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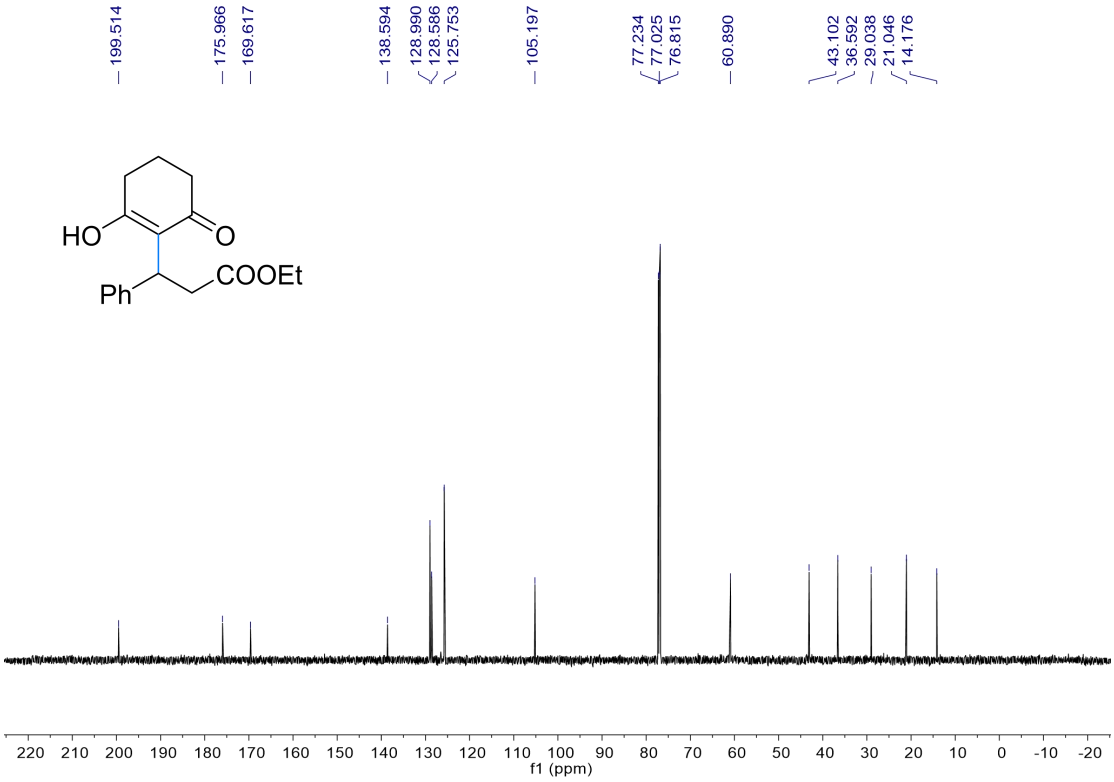
2.28
2.22
3.44
f1 (ppm)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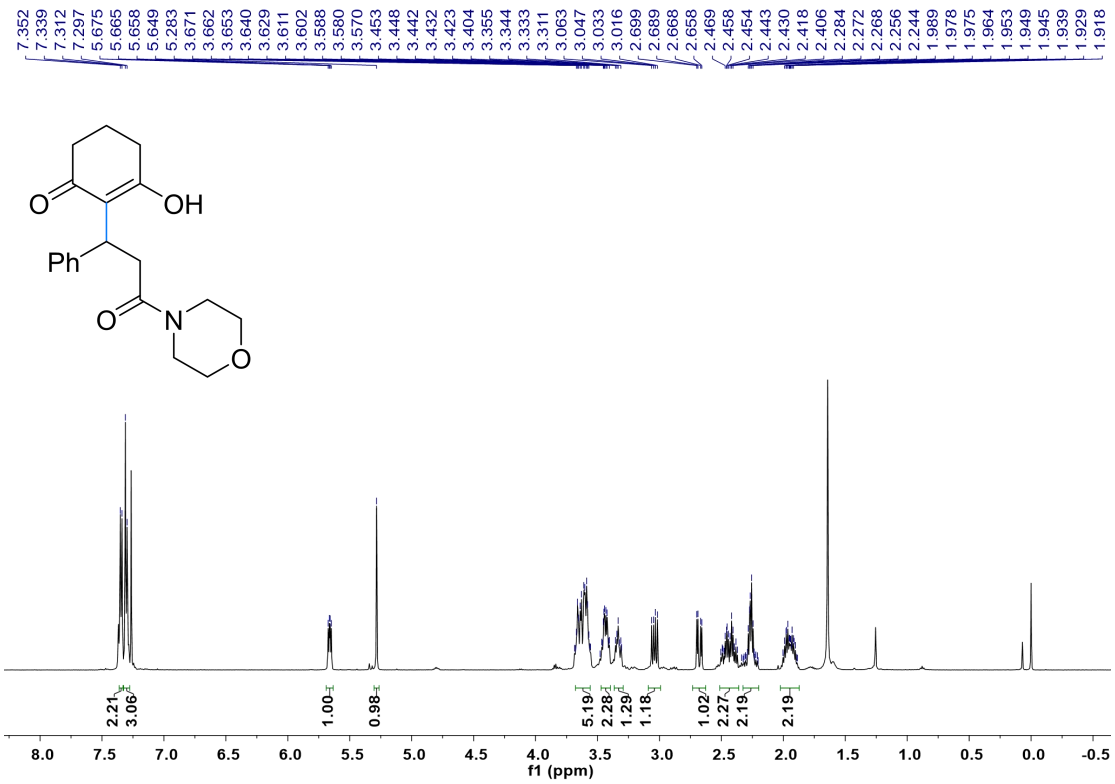
HO
O
COOEt
Ph
f1 (ppm)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3.588
3.580
3.570
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3.442
3.432
3.423
3.404
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3.344
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1.939
1.929
1.918
O
OH
Ph
O
N
O
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3.06
1.00
0.98
5.19
2.28
1.29
1.18
1.02
2.27
2.19
2.19
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
2.5
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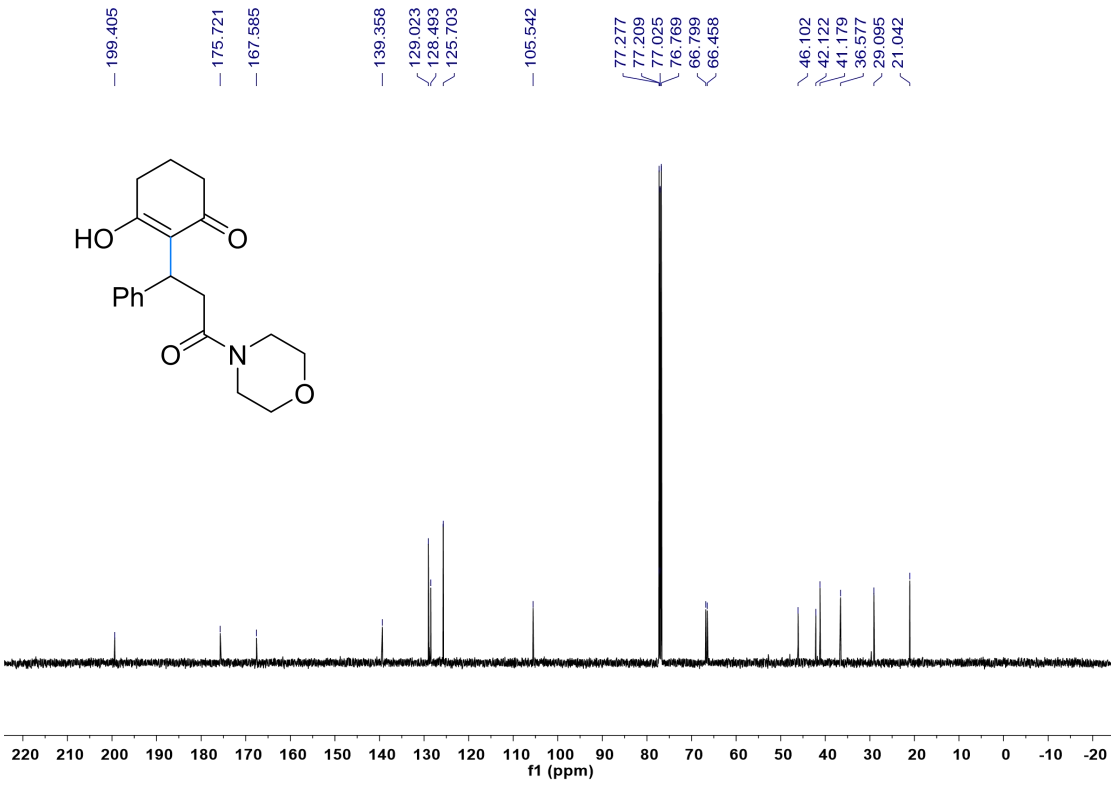
-0.5
f1 (ppm)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76.769
66.799
66.458
46.102
42.122
41.179
36.577
29.095
21.042
HO
O
Ph
O
N
O
220
210
200
190
180
170
160
150
140
130
120
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100
90
80
70
60
50
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20
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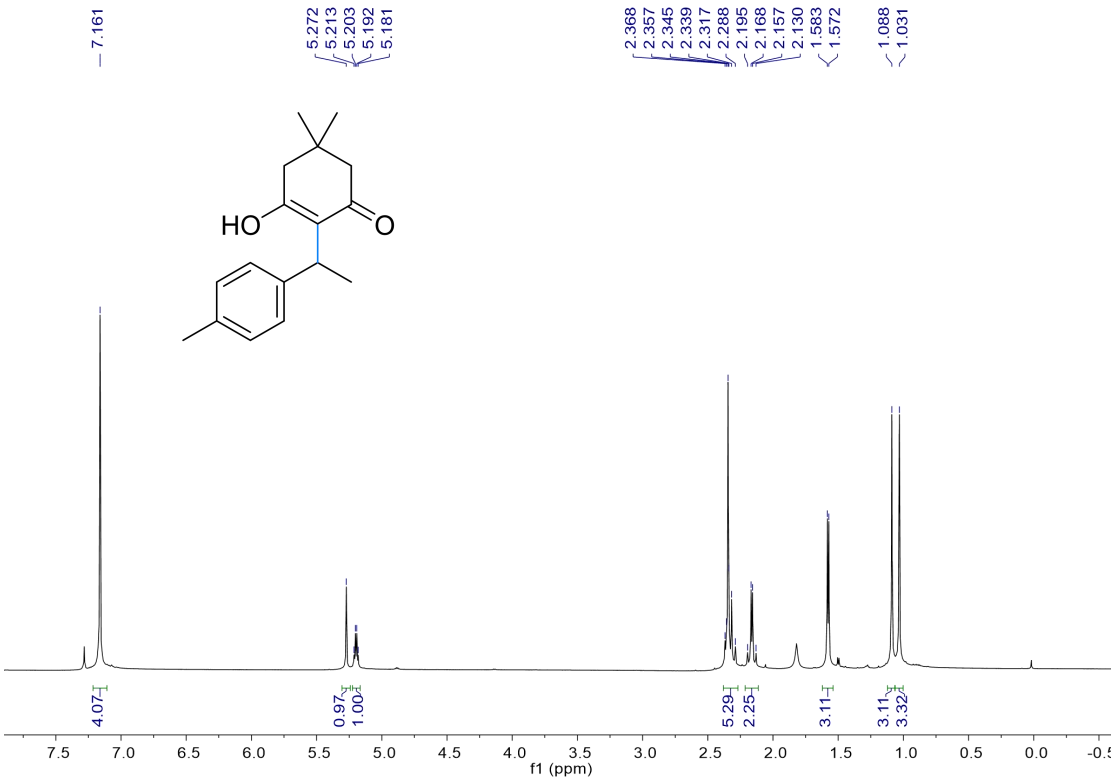
-10
-20
f1 (ppm)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2.195
2.168
2.157
2.130
1.583
1.572
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O
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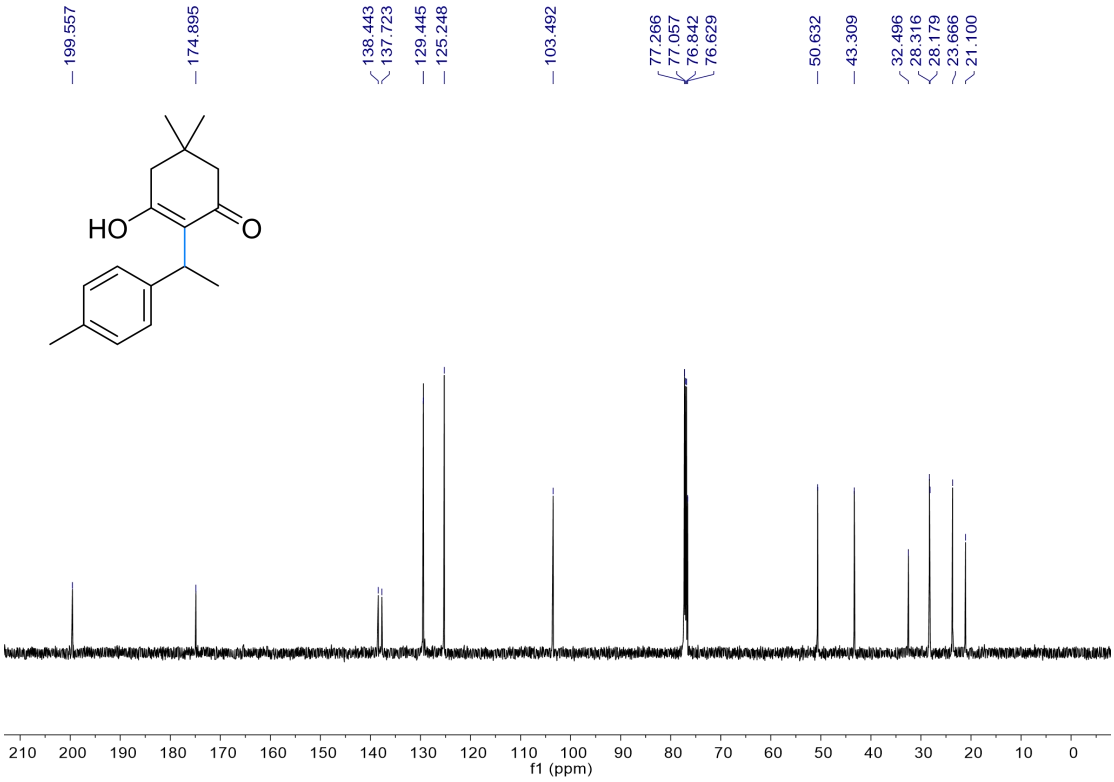
3.11
3.11
3.32
f1 (ppm)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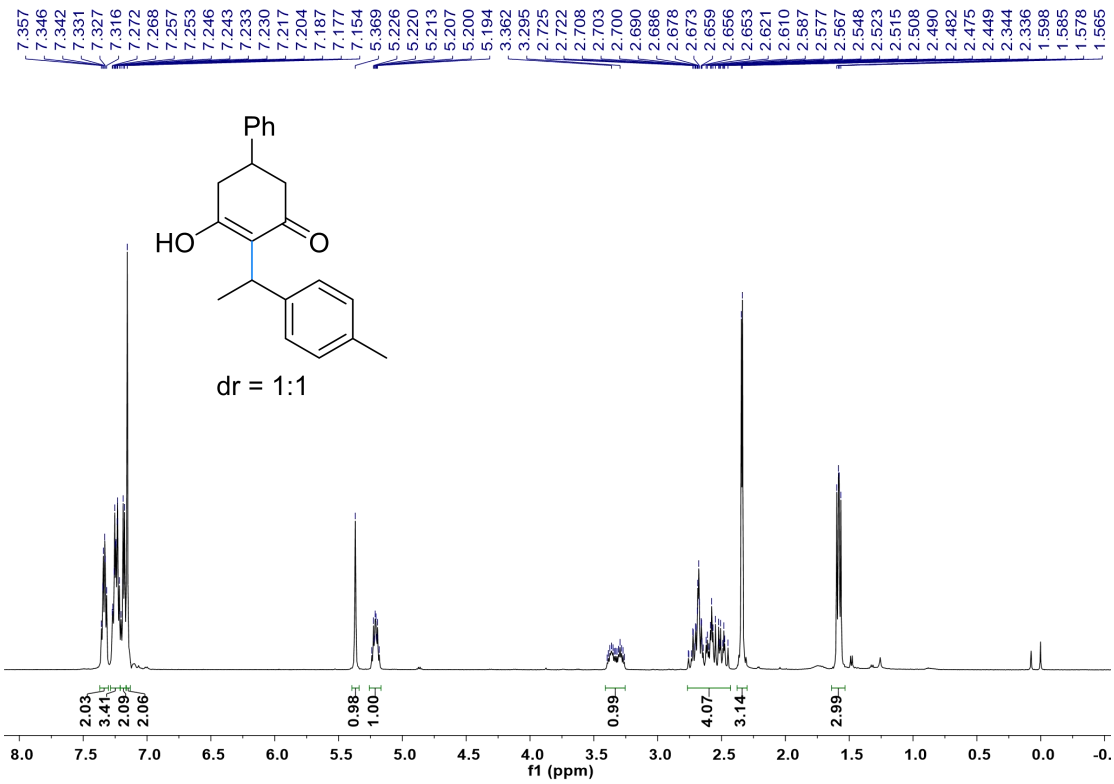
137.723
129.445
125.248
103.492
77.266
77.057
76.842
76.629
50.632
43.309
32.496
28.316
28.179
23.666
21.100
HO
O
f1 (ppm)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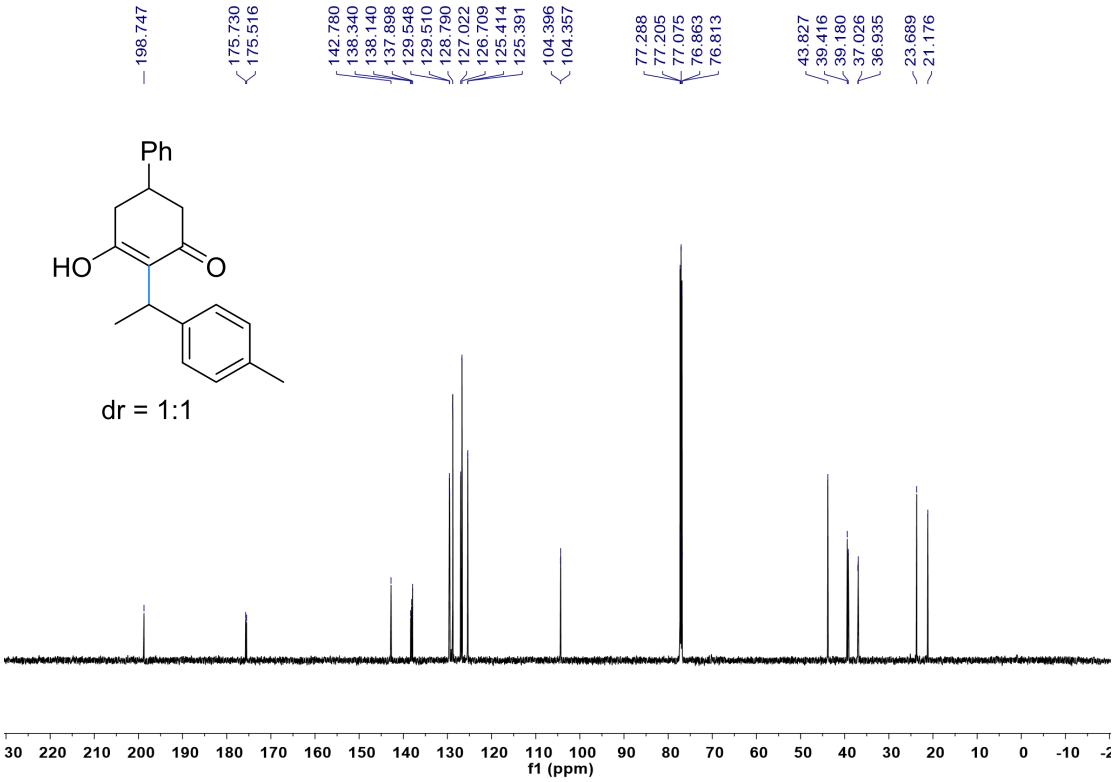
7.346
7.342
7.331
7.327
7.316
7.272
7.268
7.257
7.253
7.246
7.243
7.233
7.230
7.217
7.204
7.187
7.177
7.154
5.369
5.226
5.220
5.213
5.207
5.200
5.194
3.362
3.295
2.725
2.722
2.708
2.703
2.700
2.690
2.686
2.678
2.673
2.659
2.656
2.653
2.621
2.610
2.587
2.577
2.567
2.548
2.523
2.515
2.508
2.490
2.482
2.475
2.449
2.344
2.336
1.598
1.585
1.578
1.565
Ph
HO
O
dr = 1:1
2.03
3.41
2.09
2.06
0.98
1.00
0.99
4.07
3.14
2.99
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)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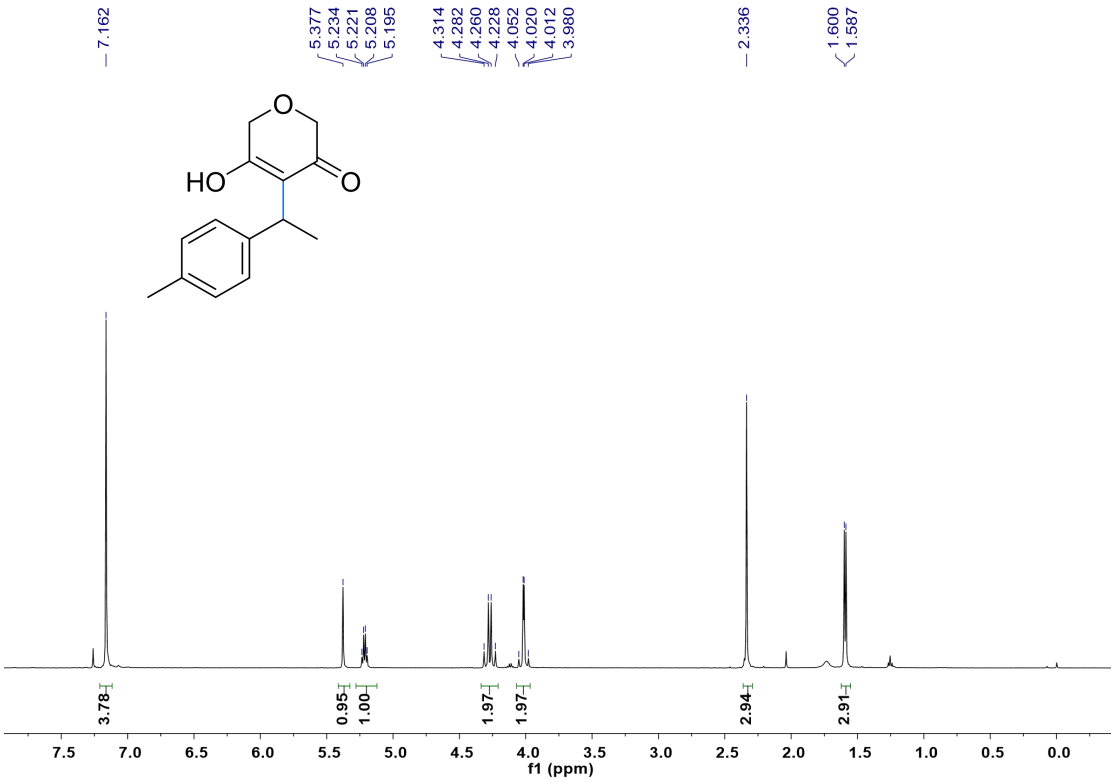
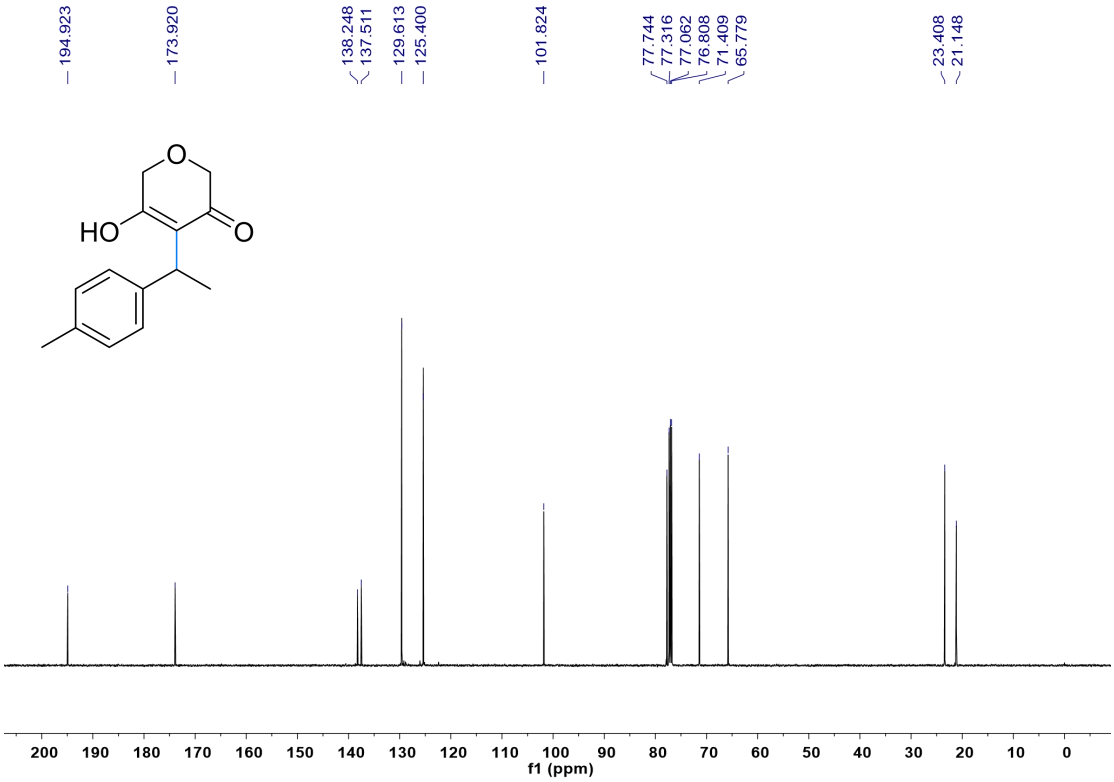
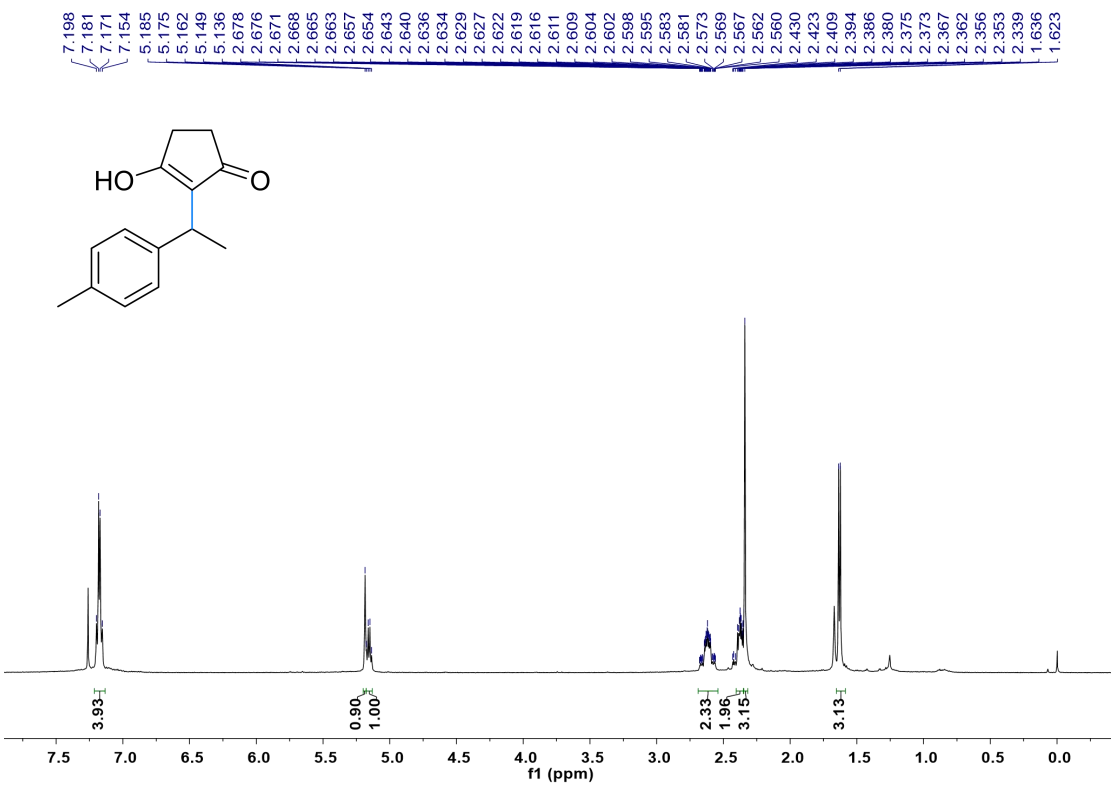
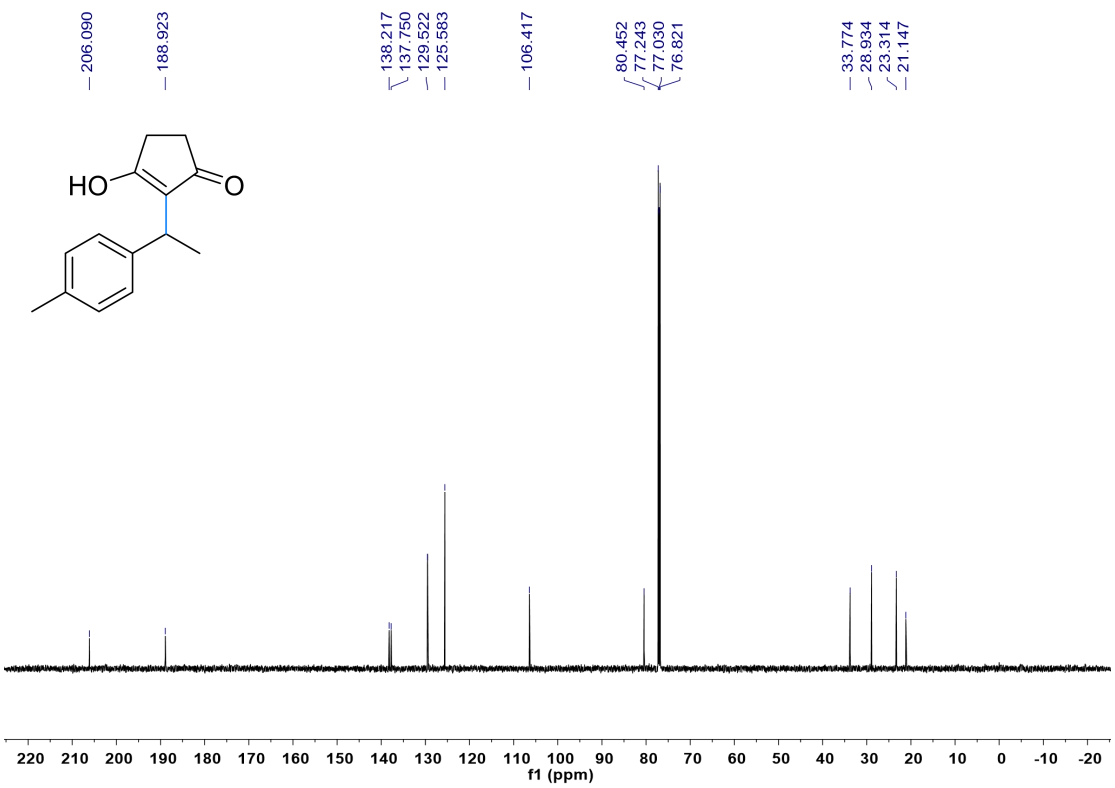
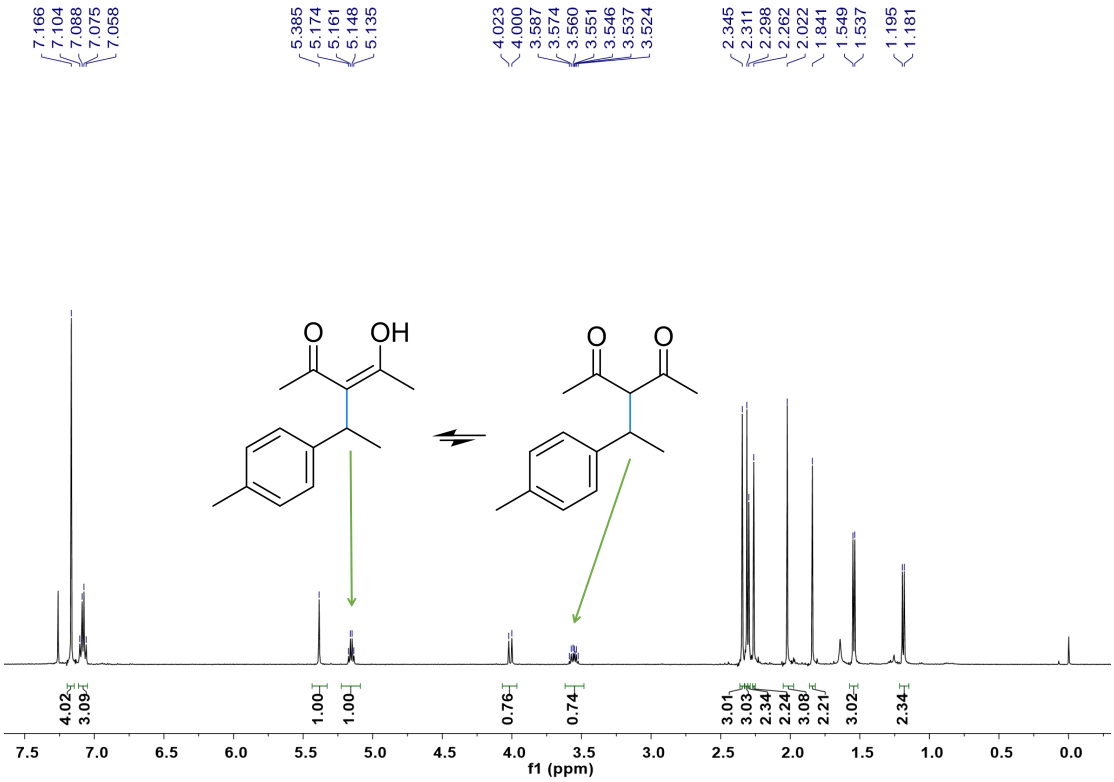
175.516
142.780
138.340
138.140
137.898
129.548
129.510
128.790
127.022
126.709
125.414
125.391
104.396
104.357
77.288
77.205
77.075
76.863
76.813
43.827
39.416
39.180
37.026
36.935
23.689
21.176
Ph
HO
O
dr = 1:1
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

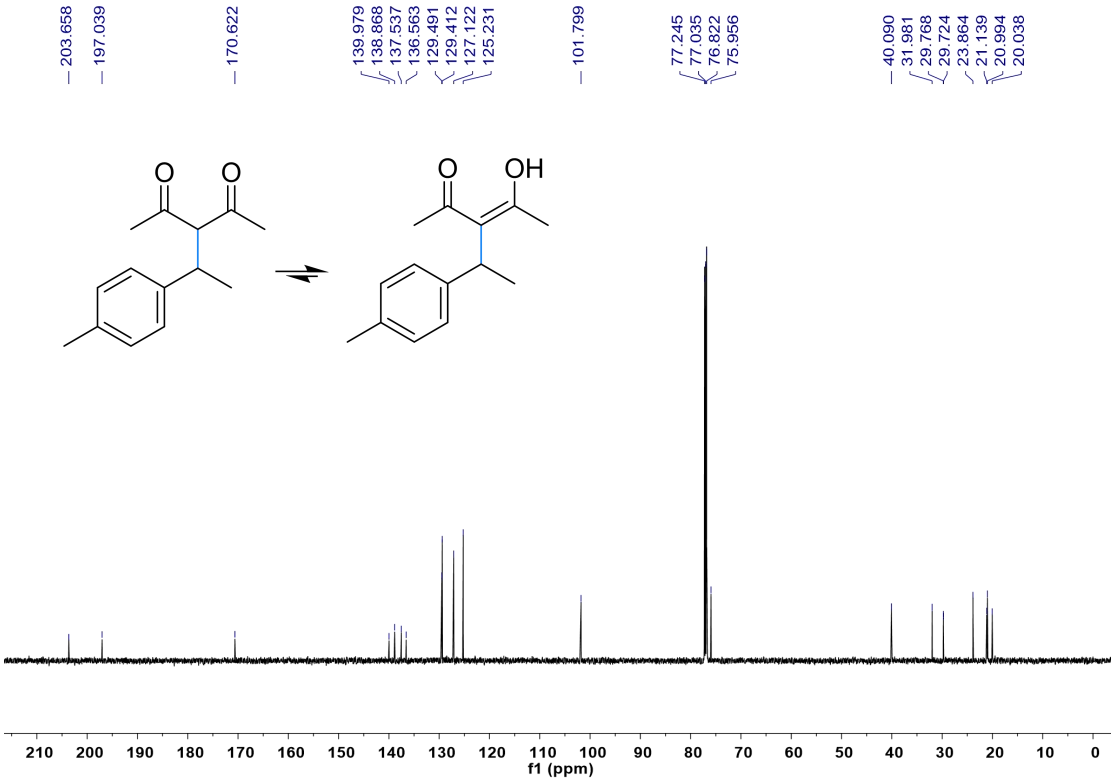

7.162
5.377
5.234
5.221
5.208
5.195
4.314
4.282
4.260
4.228
4.052
4.020
4.012
3.980
2.336
1.600
1.587
HO
O
O
3.78
0.95
1.00
1.97
1.97
2.94
2.91
f1 (ppm)

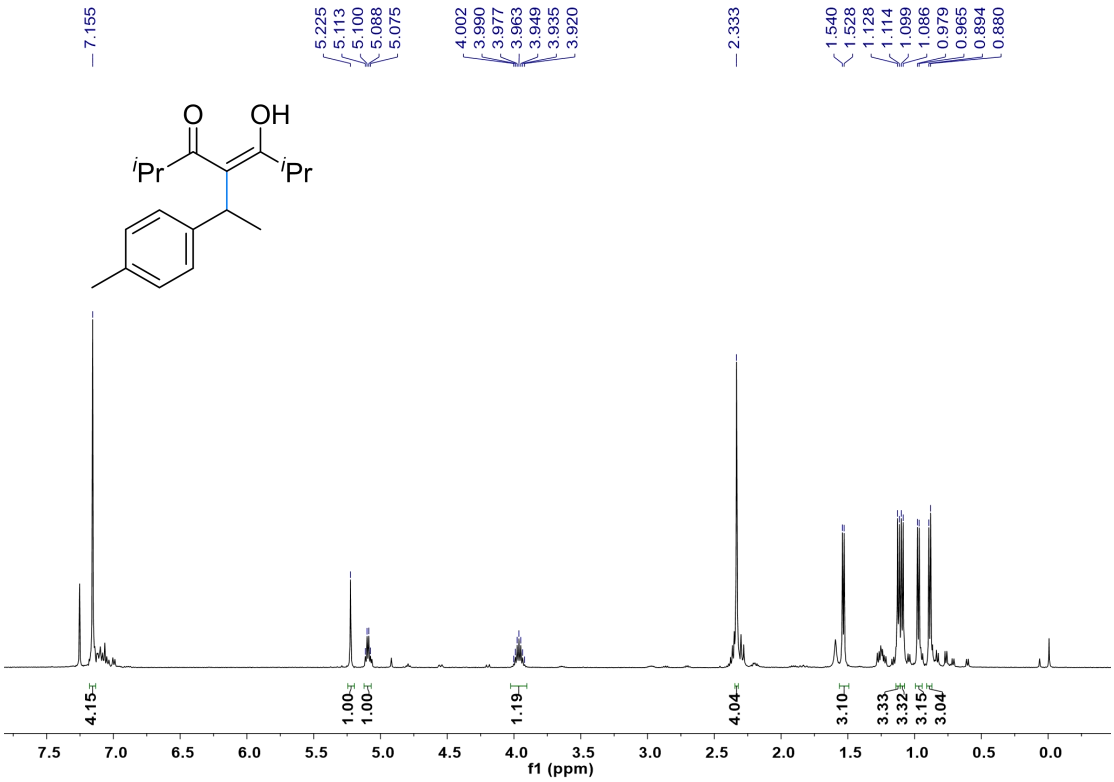

194.923
173.920
138.248
137.511
129.613
125.400
101.824
77.744
77.316
77.062
76.808
71.409
65.779
23.408
21.148
HO
O
O
f1 (ppm)

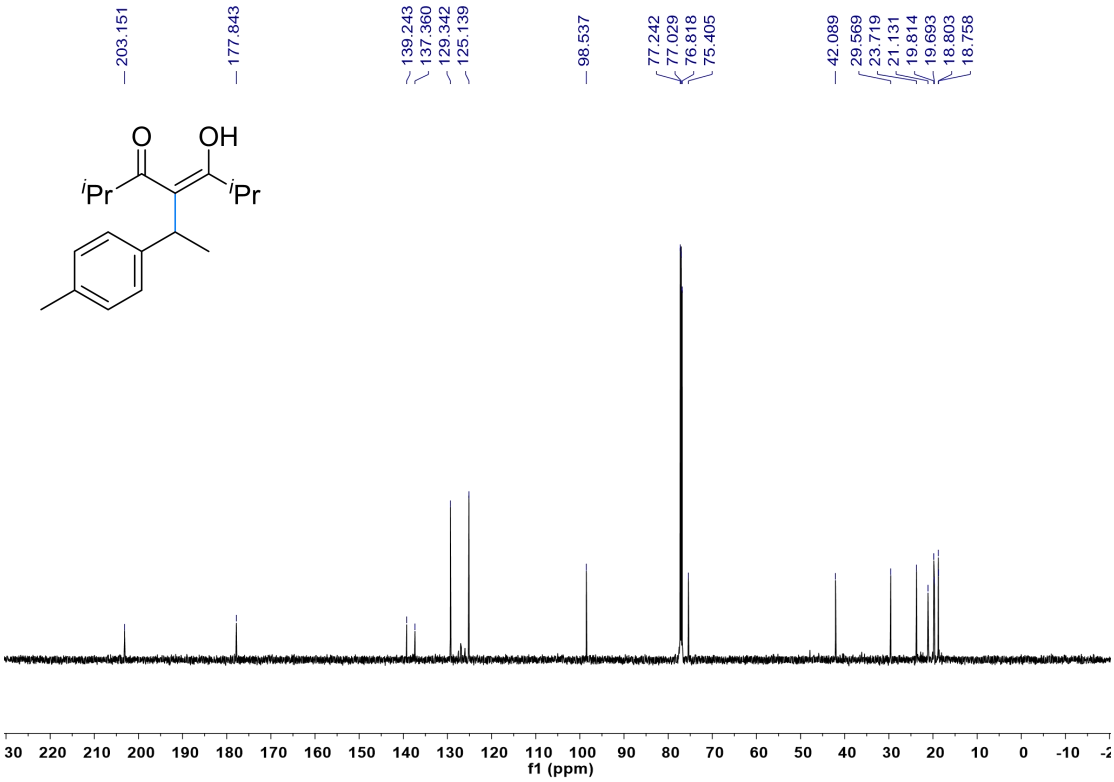

7.198
7.181
7.171
7.154
5.185
5.175
5.162
5.149
5.136
2.678
2.676
2.671
2.668
2.665
2.663
2.657
2.654
2.643
2.640
2.636
2.634
2.629
2.627
2.622
2.619
2.616
2.611
2.609
2.604
2.602
2.598
2.595
2.583
2.581
2.573
2.569
2.567
2.562
2.560
2.430
2.423
2.409
2.394
2.386
2.380
2.375
2.373
2.367
2.362
2.356
2.353
2.339
1.636
1.623
HO
O
3.93
0.90
1.00
2.33
1.96
3.15
3.13
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)

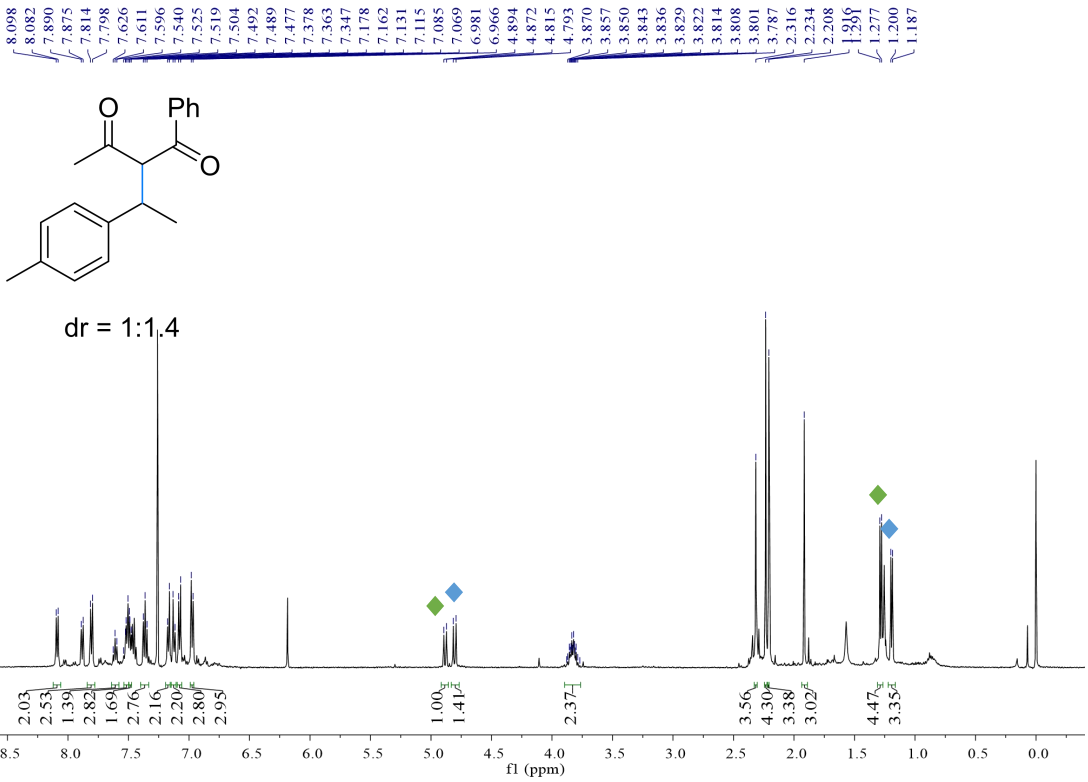

206.090
188.923
138.217
137.750
129.522
125.583
106.417
80.452
77.243
77.030
76.821
33.774
28.934
23.314
21.147
HO
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
f1 (ppm)

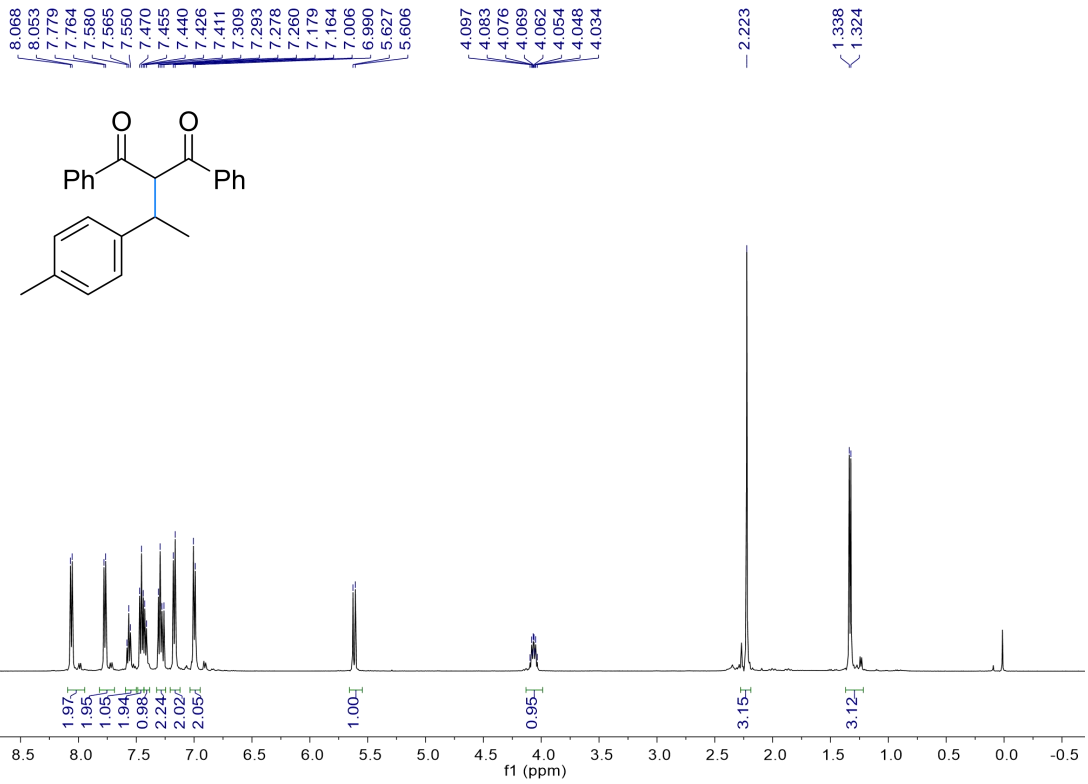

7.166
7.104
7.088
7.075
7.058
5.385
5.174
5.161
5.148
5.135
4.023
4.000
3.587
3.574
3.560
3.551
3.546
3.537
3.524
2.345
2.311
2.298
2.262
2.022
1.841
1.549
1.537
1.195
1.181
O
OH
O
O
4.02
3.09
1.00
1.00
0.76
0.74
3.01
3.03
2.34
2.24
3.08
2.21
3.02
2.34
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

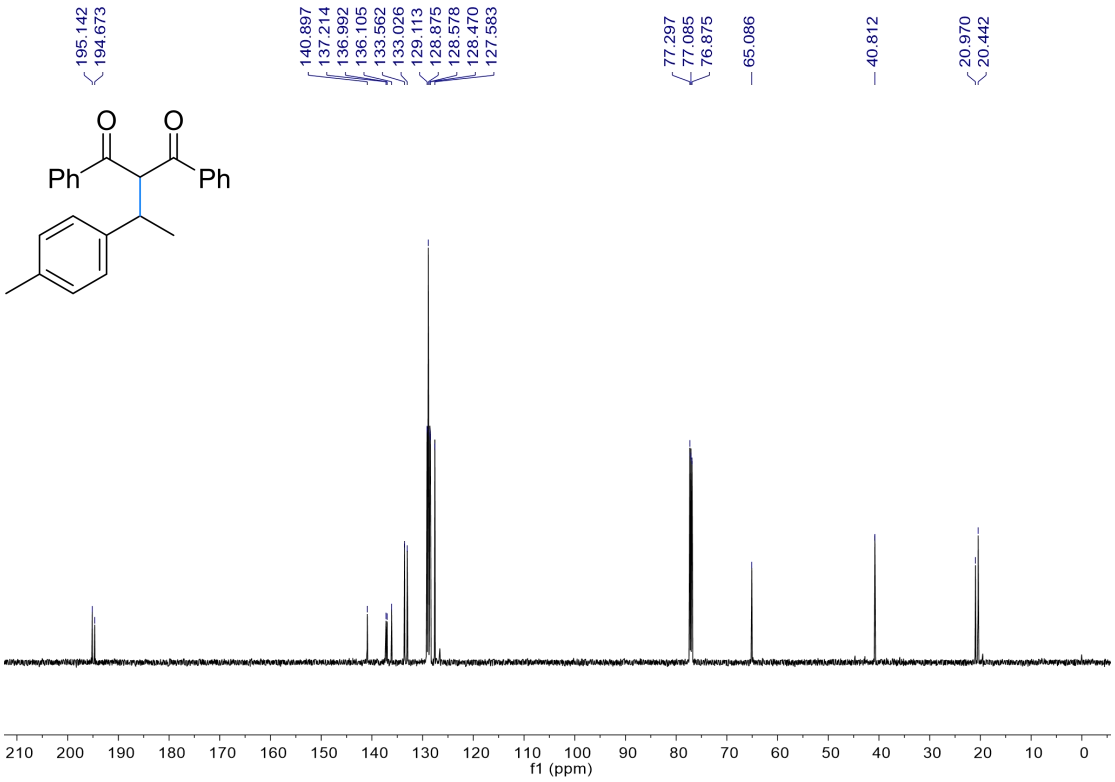

203.658
197.039
170.622
139.979
138.868
137.537
136.563
129.491
129.412
127.122
125.231
101.799
77.245
77.035
76.822
75.956
40.090
31.981
29.768
29.724
23.864
21.139
20.994
20.038
O
O
O
OH
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

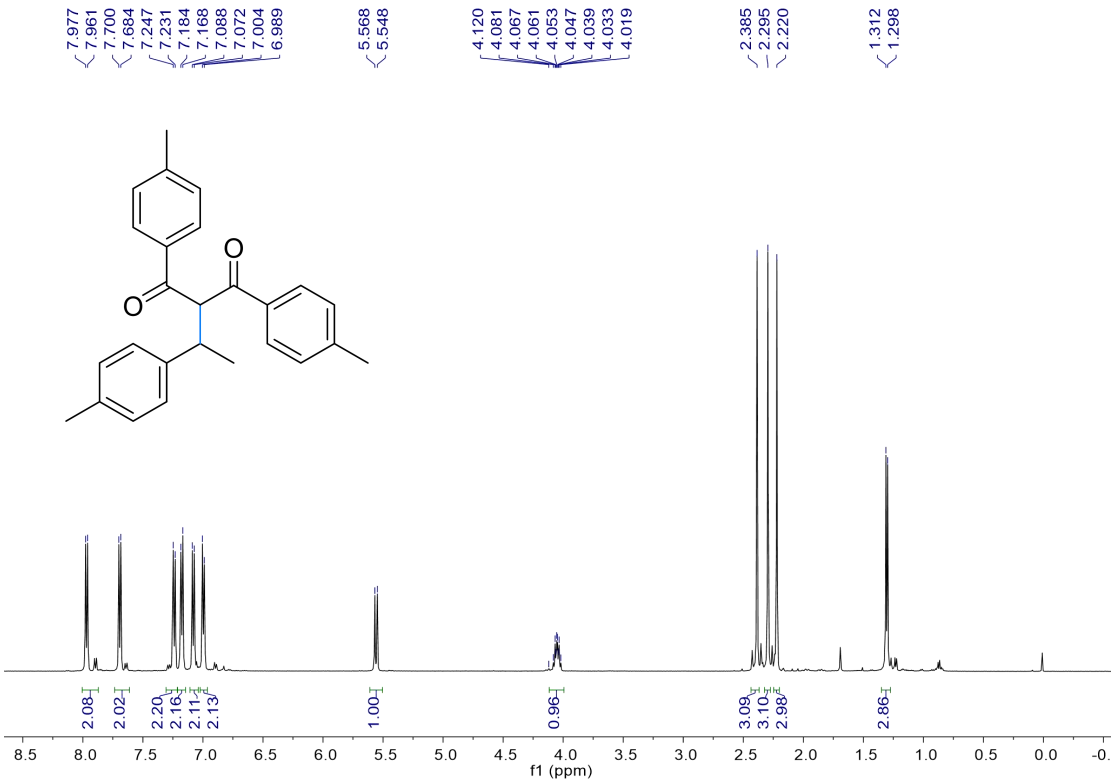

7.155
5.225
5.113
5.100
5.088
5.075
4.002
3.990
3.977
3.963
3.949
3.935
3.920
2.333
1.540
1.528
1.128
1.114
1.099
1.086
0.979
0.965
0.894
0.880
O
OH
iPr
iPr
4.15
1.00
1.00
1.19
4.04
3.10
3.33
3.32
3.15
3.04
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

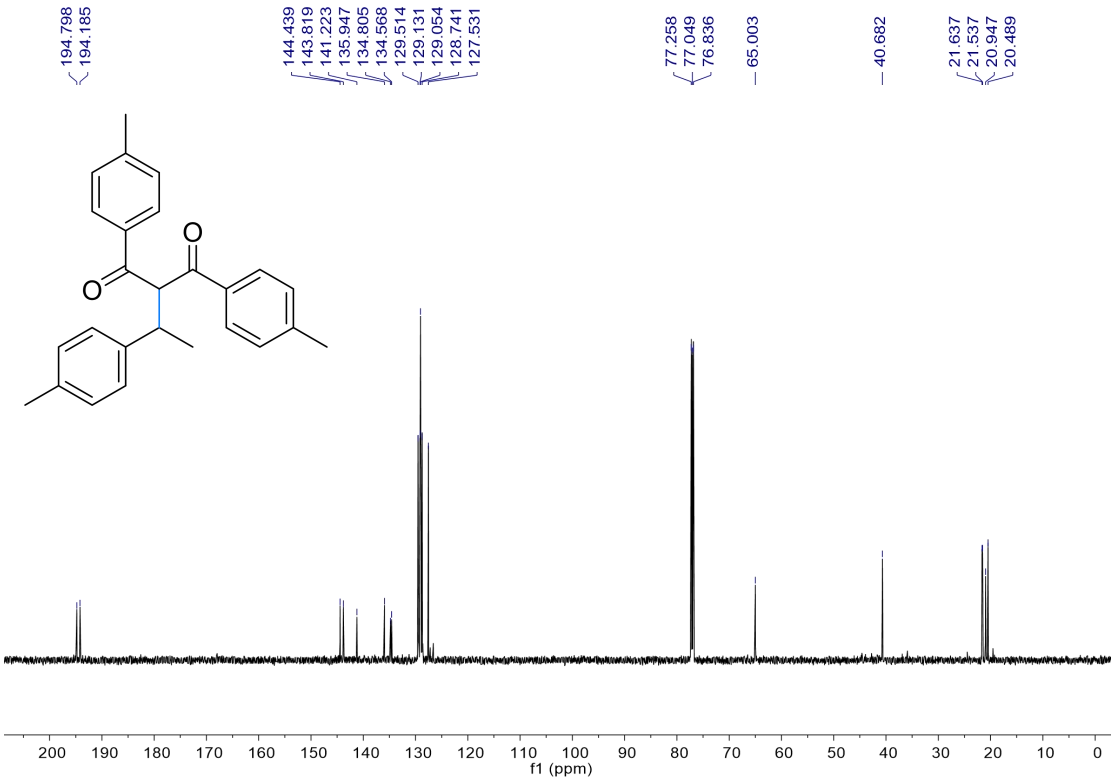

203.151
177.843
139.243
137.360
129.342
125.139
98.537
77.242
77.029
76.818
75.405
42.089
29.569
23.719
21.131
19.814
19.693
18.803
18.758
O
OH
iPr
iPr
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.098
8.082
7.890
7.875
7.814
7.798
7.626
7.611
7.596
7.540
7.525
7.519
7.504
7.492
7.489
7.477
7.378
7.363
7.347
7.178
7.162
7.131
7.115
7.085
7.069
6.981
6.966
4.894
4.872
4.815
4.793
3.870
3.857
3.850
3.843
3.836
3.829
3.822
3.814
3.808
3.801
3.787
2.316
2.234
2.208
1.291
1.277
1.200
1.187
O
Ph
O
dr = 1:1.4
2.03
2.53
1.39
2.82
1.69
2.76
2.16
2.20
2.80
2.95
1.00
1.41
2.37
3.56
4.30
3.38
3.02
4.47
3.35
f1 (ppm)


8.068
8.053
7.779
7.764
7.580
7.565
7.550
7.470
7.455
7.440
7.426
7.411
7.309
7.293
7.278
7.260
7.179
7.164
7.006
6.990
5.627
5.606
4.097
4.083
4.076
4.069
4.062
4.054
4.048
4.034
2.223
1.338
1.324
O
O
Ph
Ph
1.97
1.95
1.05
1.94
0.98
2.24
2.02
2.05
1.00
0.95
3.15
3.12
f1 (ppm)


195.142
194.673
140.897
137.214
136.992
136.105
133.562
133.026
129.113
128.875
128.578
128.470
127.583
77.297
77.085
76.875
65.086
40.812
20.970
20.442
O
O
Ph
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.977
7.961
7.700
7.684
7.247
7.231
7.184
7.168
7.088
7.072
7.004
6.989
5.568
5.548
4.120
4.081
4.067
4.061
4.053
4.047
4.039
4.033
4.019
2.385
2.295
2.220
1.312
1.298
O
O
2.08
2.02
2.20
2.16
2.11
2.13
1.00
0.96
3.09
3.10
2.98
2.86
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.
f1 (ppm)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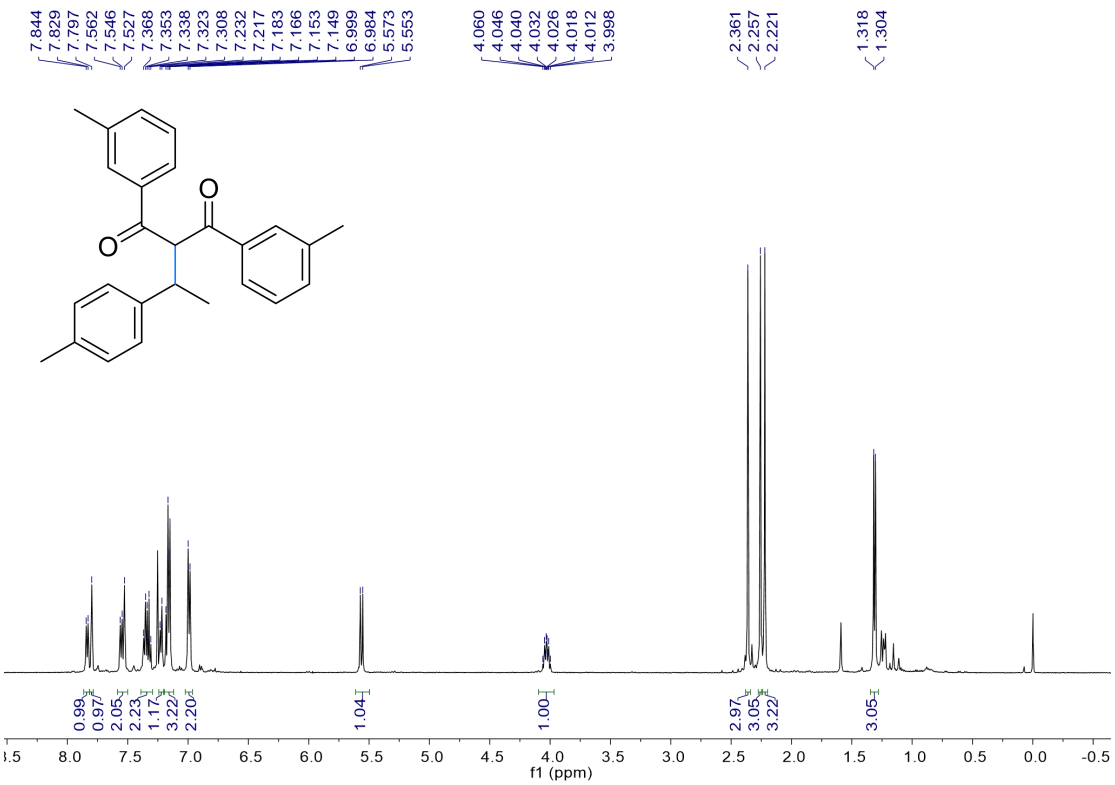
143.819
141.223
135.947
134.805
134.568
129.514
129.131
129.054
128.741
127.531
77.258
77.049
76.836
65.003
40.682
21.637
21.537
20.947
20.489
O
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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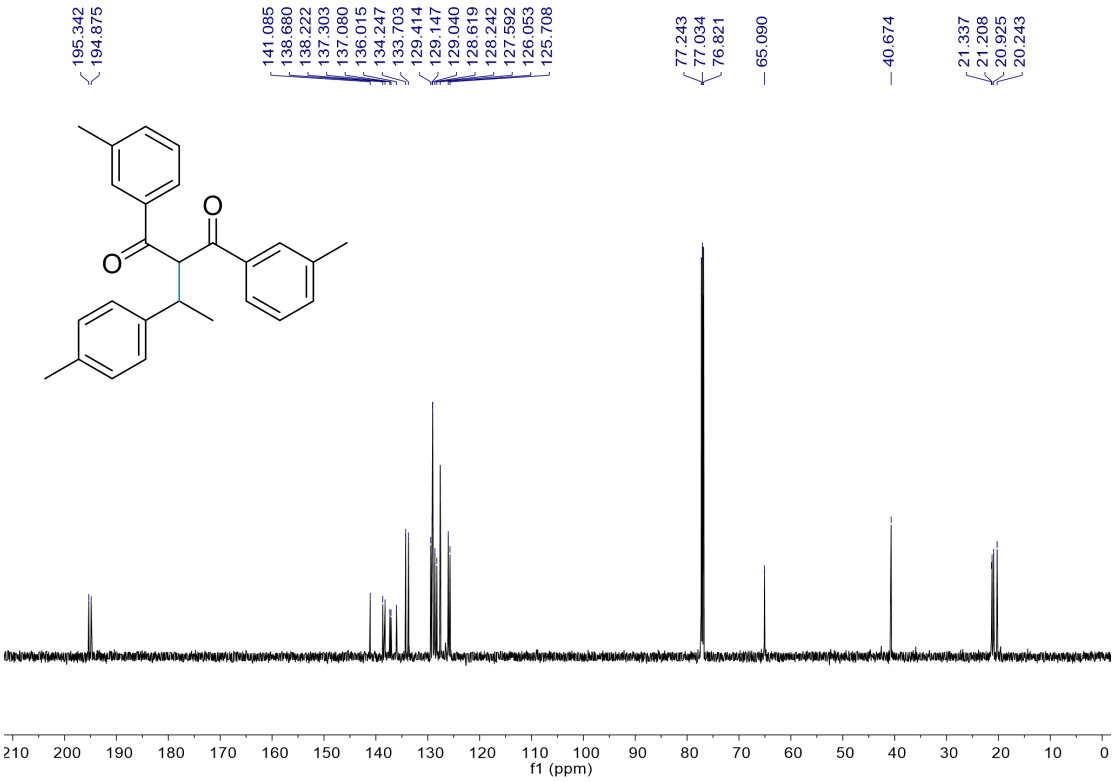
7.797
7.562
7.546
7.527
7.368
7.353
7.338
7.323
7.308
7.232
7.217
7.183
7.166
7.153
7.149
6.999
6.984
5.573
5.553
4.060
4.046
4.040
4.032
4.026
4.018
4.012
3.998
2.361
2.257
2.221
1.318
1.304
O
O
0.99
0.97
2.05
2.23
1.17
3.22
2.20
1.04
1.00
2.97
3.05
3.22
3.05
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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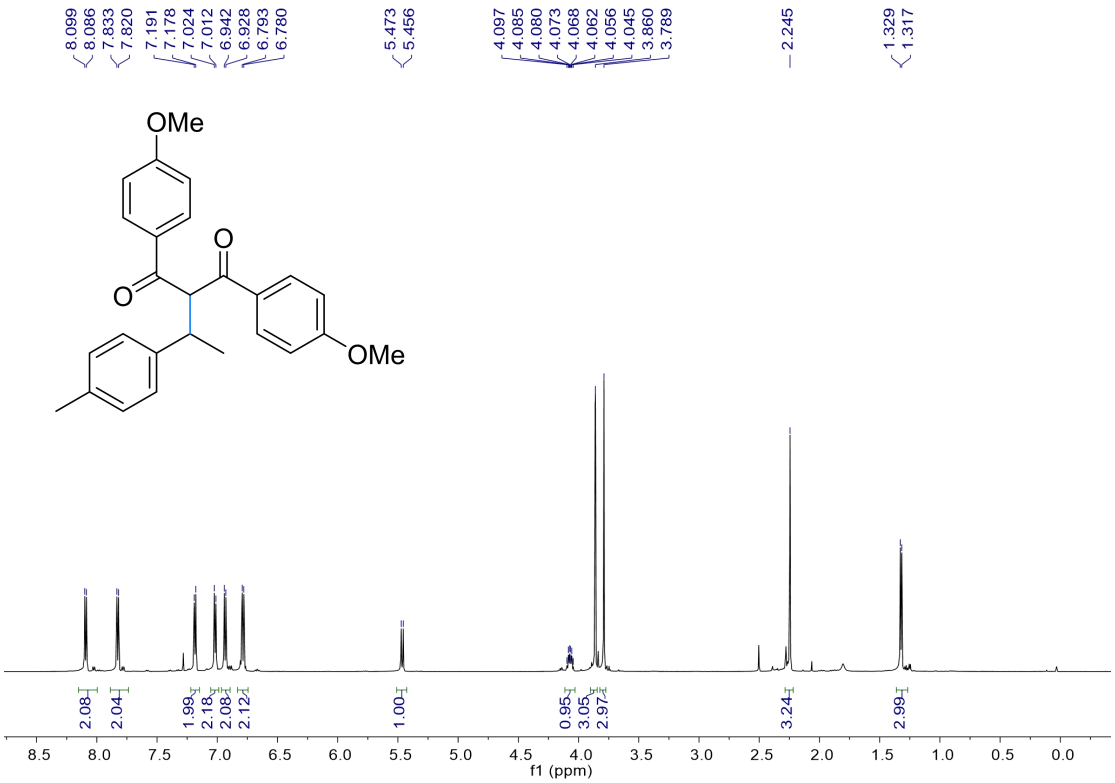
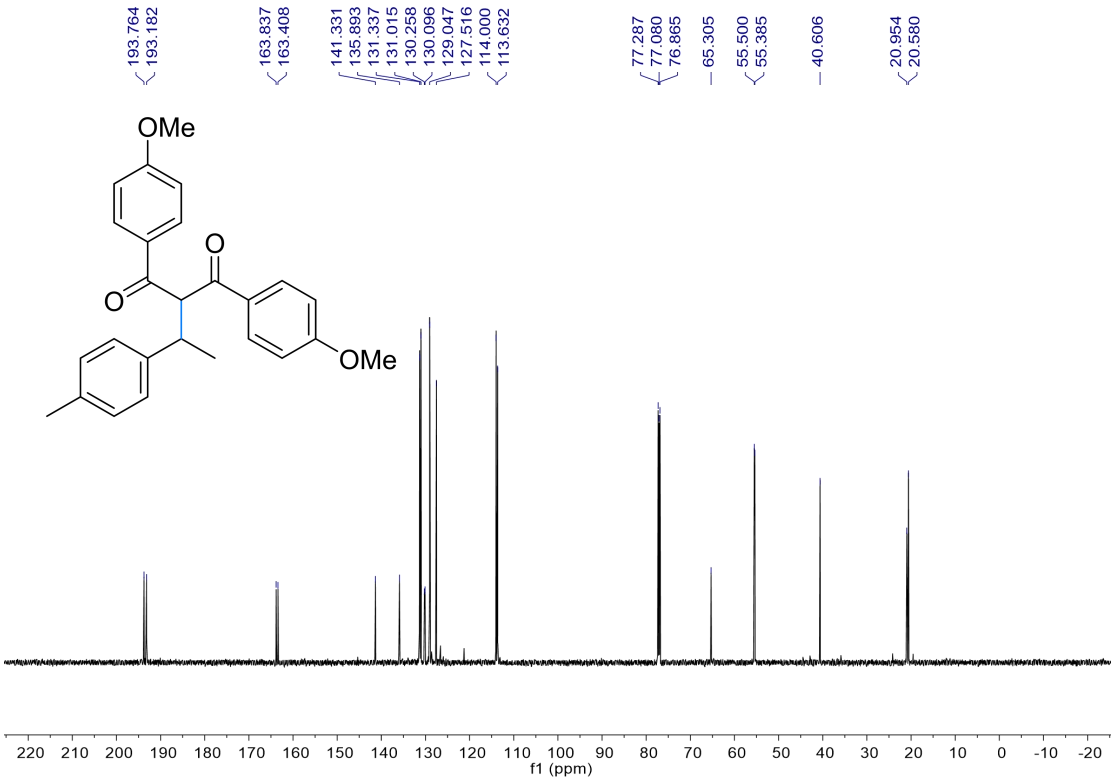
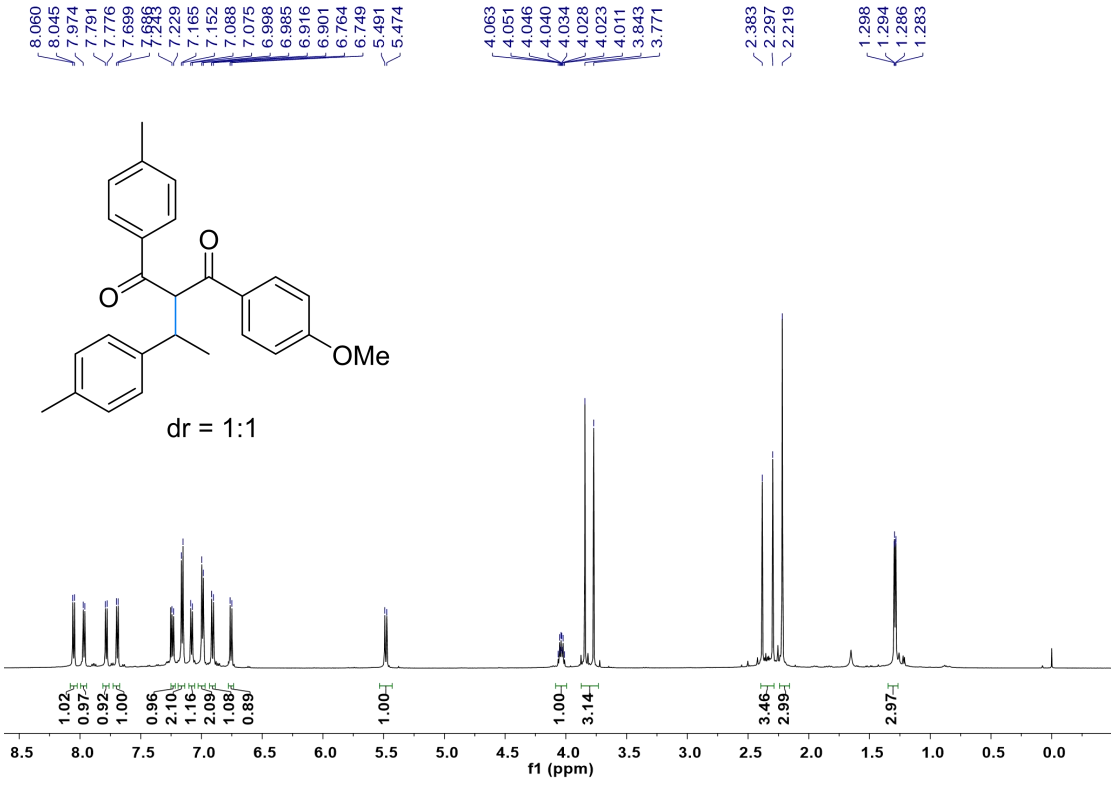
194.875
141.085
138.680
138.222
137.303
137.080
136.015
134.247
133.703
129.414
129.147
129.040
128.619
128.242
127.592
126.053
125.708
77.243
77.034
76.821
65.090
40.674
21.337
21.208
20.925
20.243
O
O
f1 (ppm)


8.099
8.086
7.833
7.820
7.191
7.178
7.024
7.012
6.942
6.928
6.793
6.780
5.473
5.456
4.097
4.085
4.080
4.073
4.068
4.062
4.056
4.045
3.860
3.789
2.245
1.329
1.317
OMe
OMe
O
O
2.08
2.04
1.99
2.18
2.08
2.12
1.00
0.95
3.05
2.97
3.24
2.99
f1 (ppm)


193.764
193.182
163.837
163.408
141.331
135.893
131.337
131.015
130.258
130.096
129.047
127.516
114.000
113.632
77.287
77.080
76.865
65.305
55.500
55.385
40.606
20.954
20.580
OMe
O
O
OMe
f1 (ppm)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8.045
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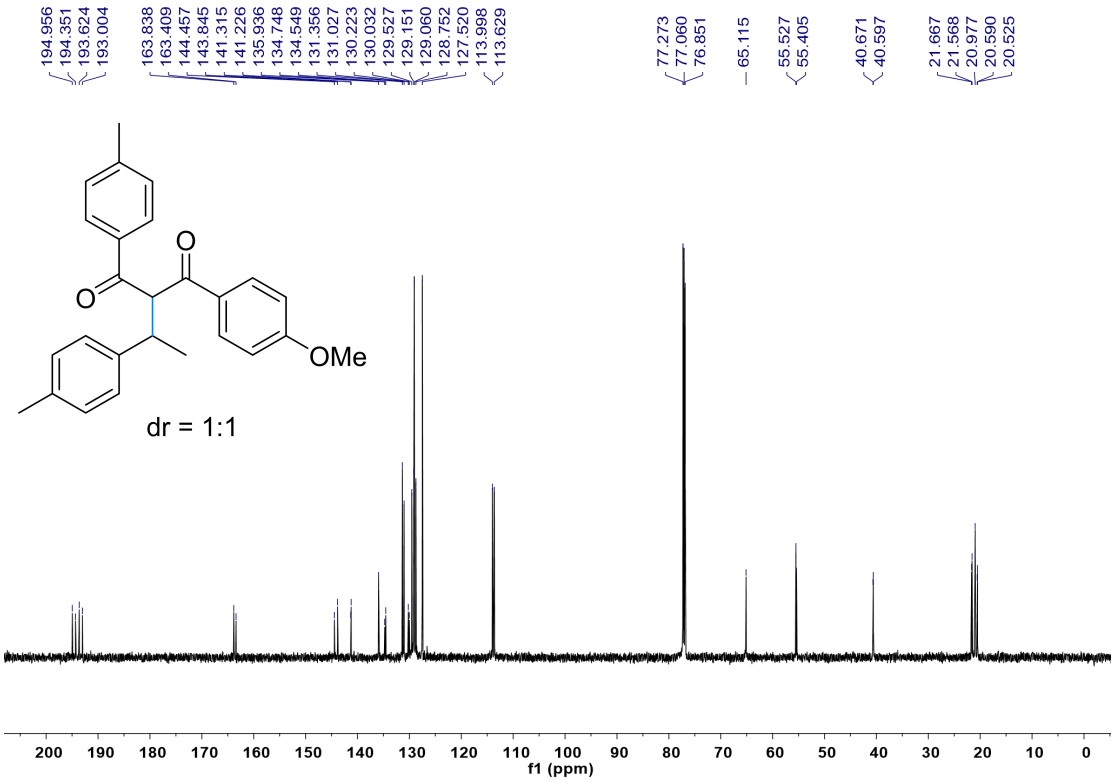
7.791
7.776
7.699
7.229
7.165
7.152
7.088
7.075
6.998
6.985
6.916
6.901
6.764
6.749
5.491
5.474
4.063
4.051
4.046
4.040
4.034
4.028
4.023
4.011
3.843
3.771
2.383
2.297
2.219
1.298
1.294
1.286
1.283
O
O
OMe
dr = 1:1
1.02
0.97
0.92
1.00
0.96
2.10
1.16
2.09
1.08
0.89
1.00
1.00
3.14
3.46
2.99
2.97
f1 (ppm)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193.624
193.004
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163.409
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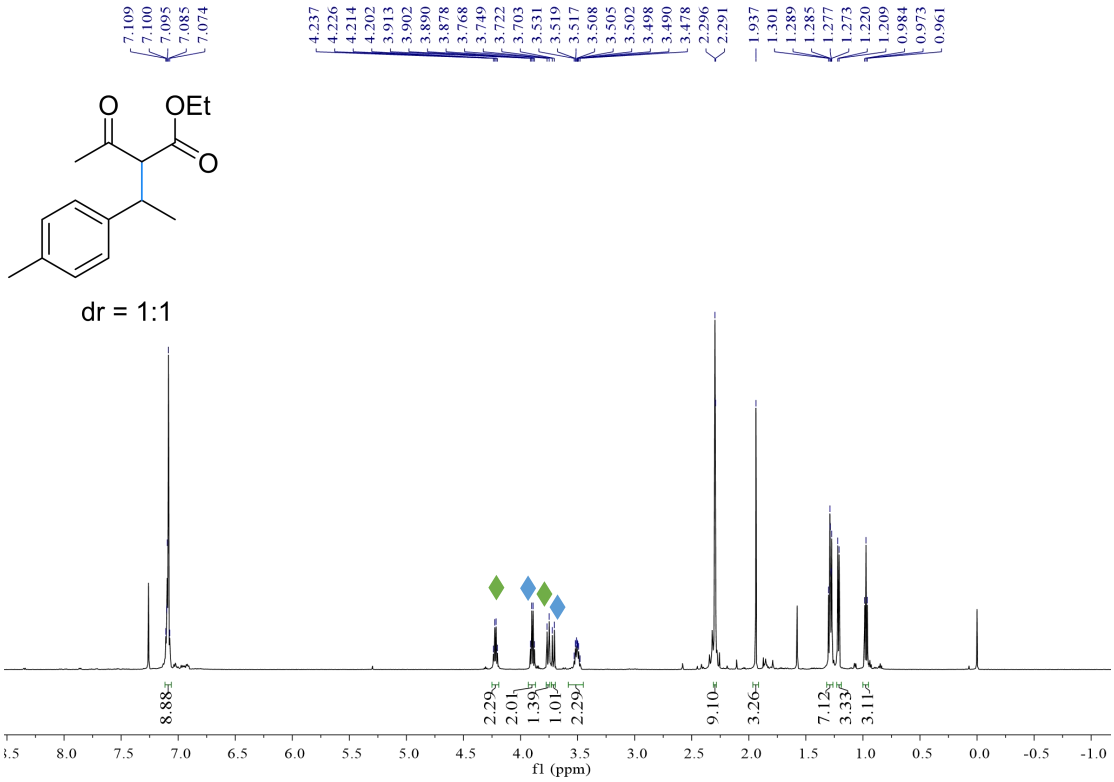
143.845
141.315
141.226
135.936
134.748
134.549
131.356
131.027
130.223
130.032
129.527
129.151
129.060
128.752
127.520
113.998
113.629
77.273
77.060
76.851
65.115
55.527
55.405
40.671
40.597
21.667
21.568
20.977
20.590
20.525
O
O
OMe
dr = 1:1
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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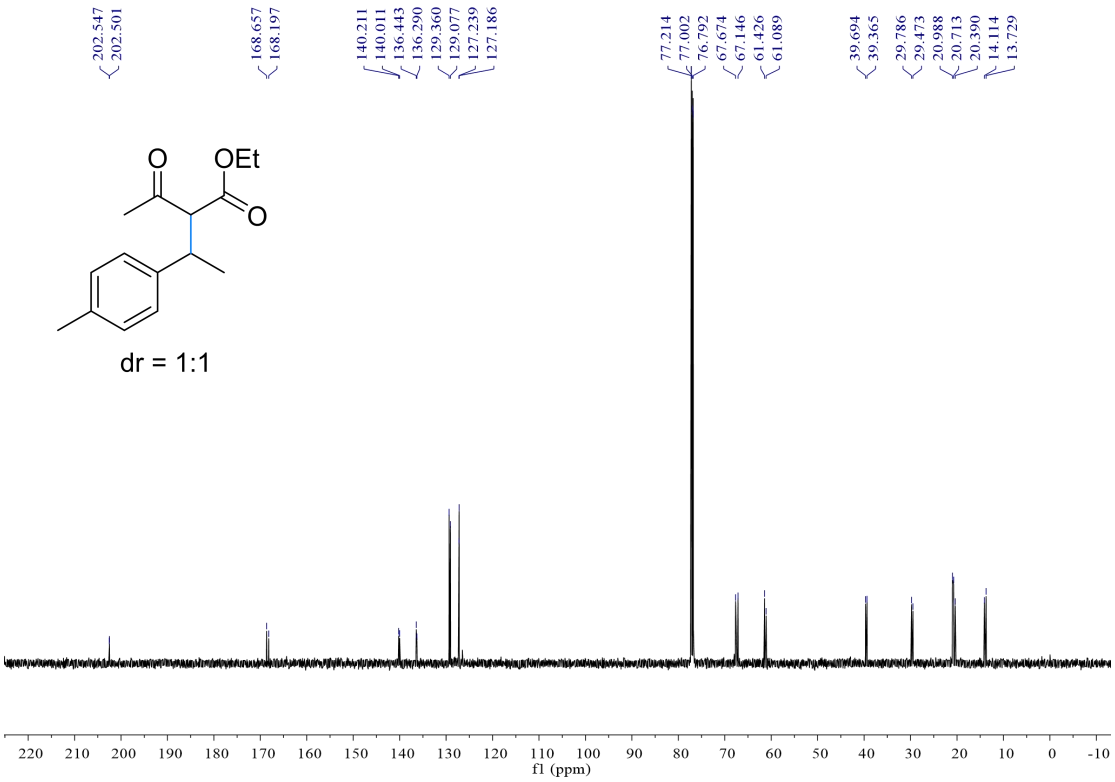
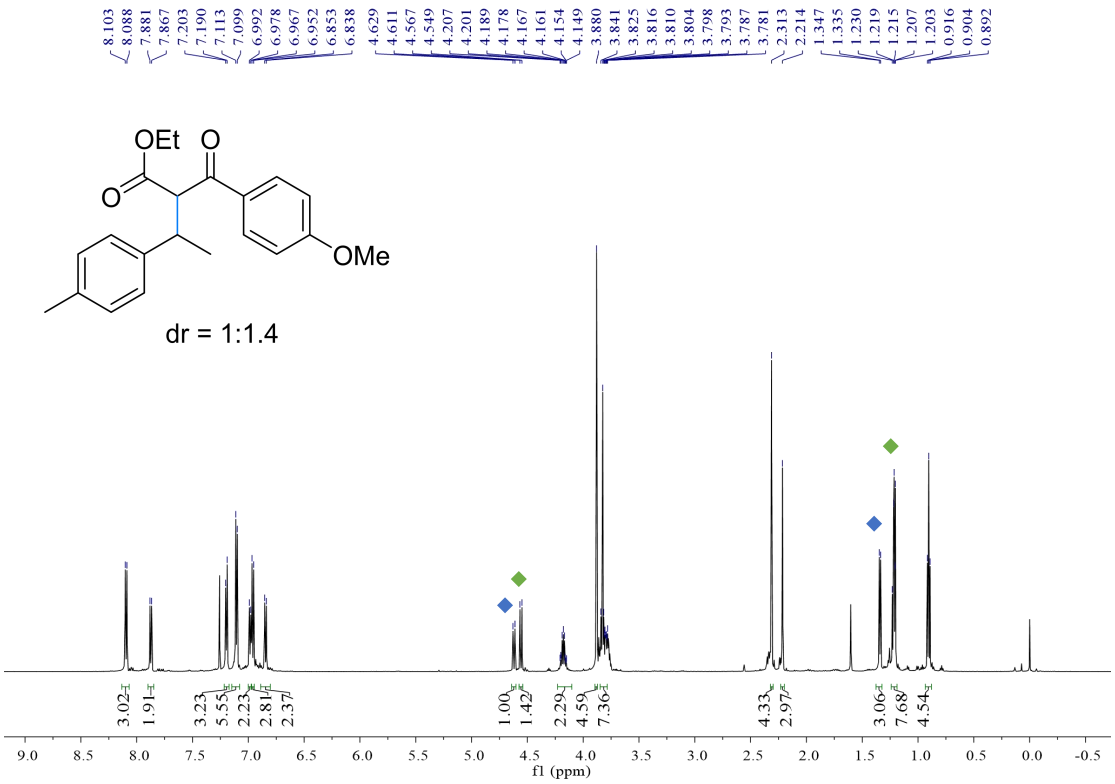
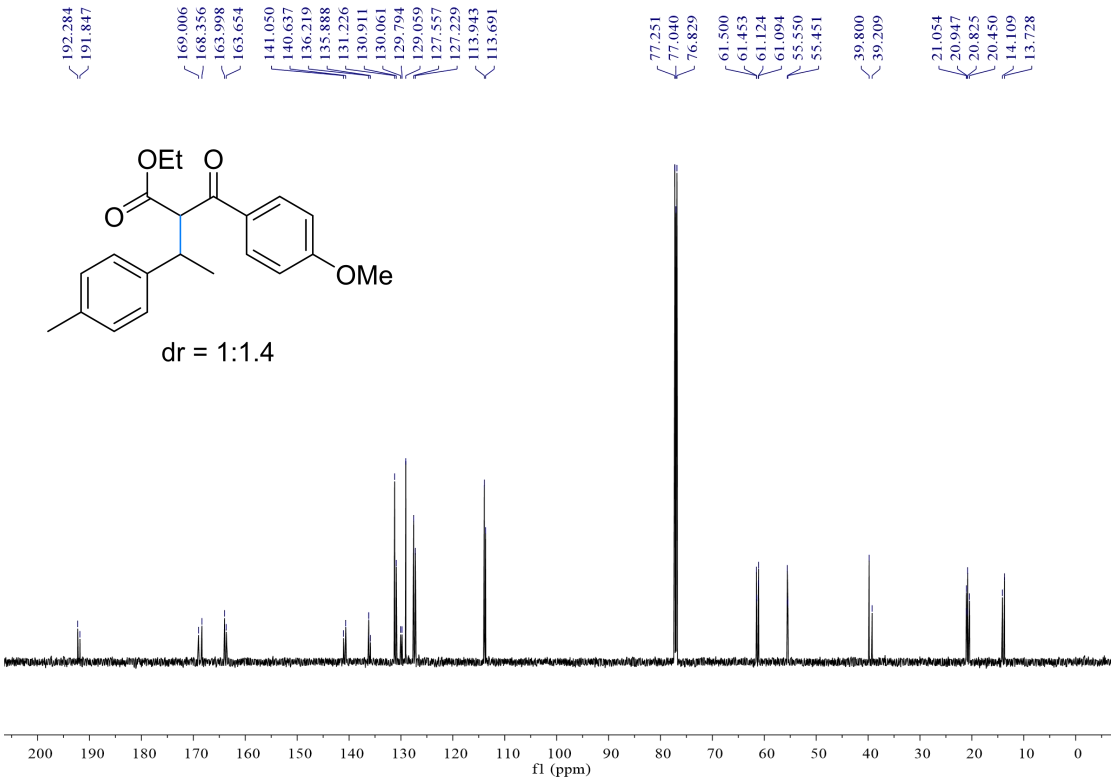
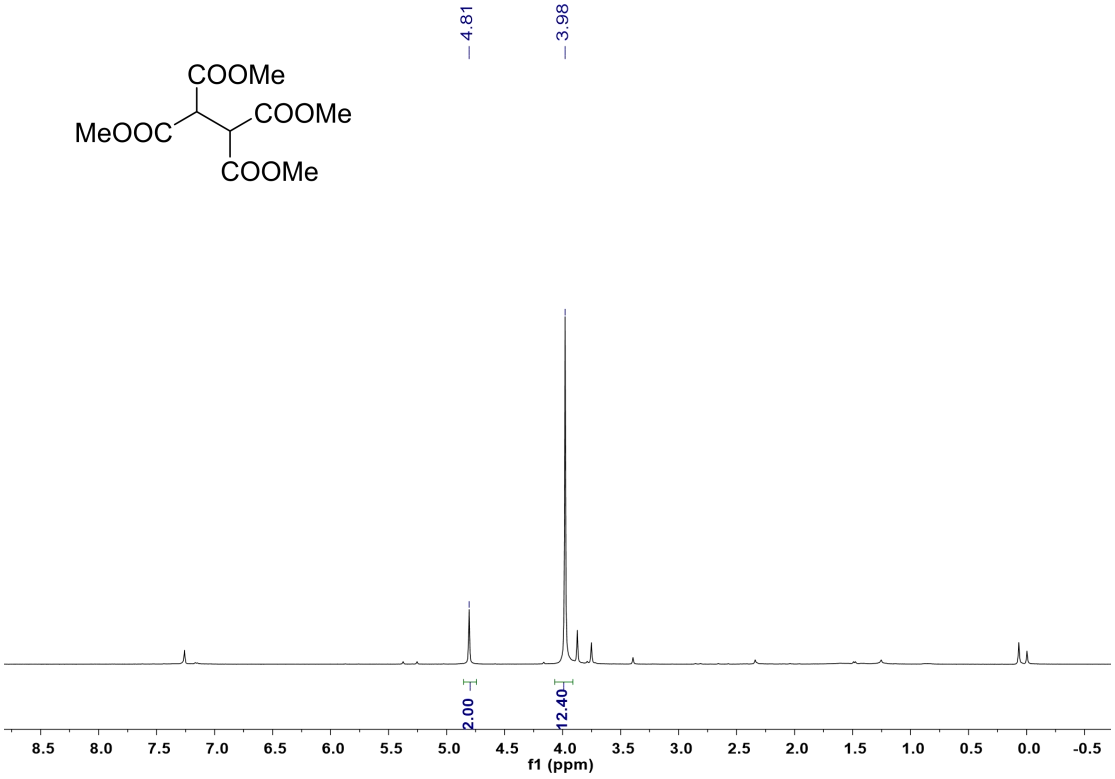
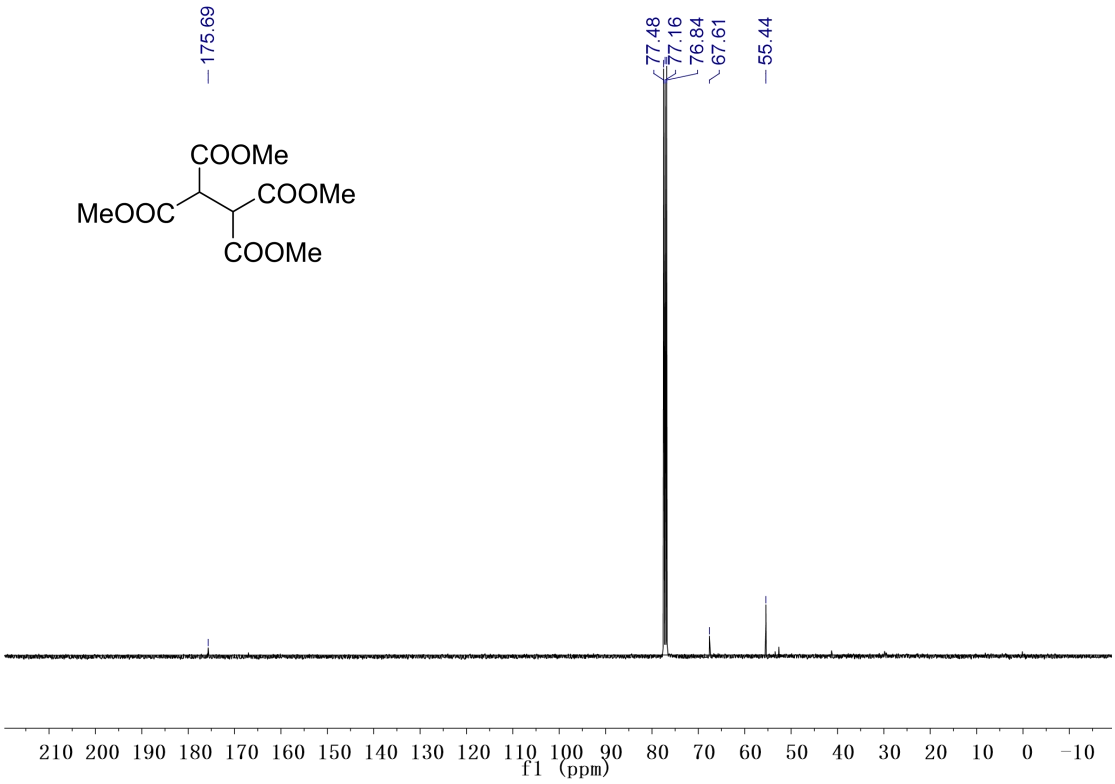
7.100
7.095
7.085
7.074
4.237
4.226
4.214
4.202
3.913
3.902
3.890
3.878
3.768
3.749
3.722
3.703
3.531
3.519
3.517
3.508
3.505
3.502
3.498
3.490
3.478
2.296
2.291
1.937
1.301
1.289
1.285
1.277
1.273
1.220
1.209
0.984
0.973
0.961
O
OEt
O
dr = 1:1
8.88
2.29
2.01
1.39
1.01
2.29
9.10
3.26
7.12
3.33
3.11
8.0 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
f1 (ppm)


202.547
202.501
168.657
168.197
140.211
140.011
136.443
136.290
129.360
129.077
127.239
127.186
77.214
77.002
76.792
67.674
67.146
61.426
61.089
39.694
39.365
29.786
29.473
20.988
20.713
20.390
14.114
13.729
O
OEt
O
dr = 1:1
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.103
8.088
7.881
7.867
7.203
7.190
7.113
7.099
6.992
6.978
6.967
6.952
6.853
6.838
4.629
4.611
4.567
4.549
4.207
4.201
4.189
4.178
4.167
4.161
4.154
4.149
3.880
3.841
3.825
3.816
3.810
3.804
3.798
3.793
3.787
3.781
2.313
2.214
1.347
1.335
1.230
1.219
1.215
1.207
1.203
0.916
0.904
0.892
OEt
O
O
OMe
dr = 1:1.4
3.02
1.91
3.23
5.55
2.23
2.81
2.37
1.00
1.42
2.29
4.59
7.36
4.33
2.97
3.06
7.68
4.54
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)


192.284
191.847
169.006
168.356
163.998
163.654
141.050
140.637
136.219
135.888
131.226
130.911
130.061
129.794
129.059
127.557
127.229
113.943
113.691
77.251
77.040
76.829
61.500
61.453
61.124
61.094
55.550
55.451
39.800
39.209
21.054
20.947
20.825
20.450
14.109
13.728
OEt
O
O
OMe
dr = 1:1.4
f1 (ppm)


4.81
3.98
COOMe
MeOOC
COOMe
COOMe
2.00
12.40
f1 (ppm)


175.69
77.48
77.16
76.84
67.61
55.44
COOMe
COOMe
MeOOC
COOMe
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)