Supporting Information

Chromium Catalyzed Sustainable C-C and C-N Bond Formation: C-Alkylation and Friedländer Quinoline Synthesis Using Alcohols

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1. General information

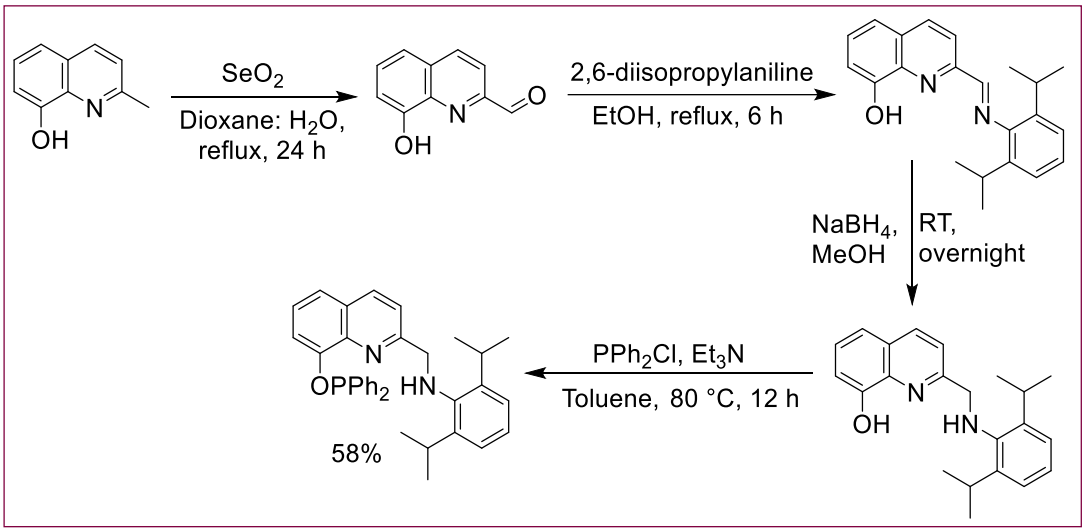
All manipulations were performed under inert nitrogen atmosphere by using standard Schlenk techniques and inside glove box, unless stated otherwise. Standard solvents were heated under reflux over suitable drying agent (sodium/benzophenone for THF, Et_2O, hexane; calcium hydride for $CHCl_3$ and kept under freshly dried molecular sieves), distilled, and stored over activated 4Å molecular sieves in the glovebox under nitrogen atmosphere. Deuterated solvents were purged with nitrogen and kept in glovebox. Unless otherwise stated, commercial reagents were used without purification, only benzyl alcohol and acetophenone were purified via vacuum distillation. Chromium chloride ($CrCl_2$) was purchased form Sigma- Aldrich, calcium hydride (CaH_2) and all other materials were purchased from Avra, BLDpharma, TCI and Sigma- Aldrich, stored under nitrogen and used as received. Ligand precursors were prepared according to literature procedure. [1, 2] Reaction temperature reported is the temperature of the oil bath. All the reaction for ligand synthesis and substrate scope was monitored by TLC and isolated by column chromatography or flash chromatography (silica gel 60-120). Then product was condensed with the help of rotary evaporator. Isolated product was characterized by using ^{1}H NMR, ^{13}C NMR from Bruker 500 MHz spectrometer using $CDCl_3$ or C_6D_6. All spectra were recorded at room temperature, unless otherwise noted. ATR-IR spectra was recorded on Perkin-Elmer FT-IR spectrometer. Solution-state UV–visible absorption spectrum was recorded on Cary 4000 UV–vis spectrophotometer using dichloromethane (DCM) solvent. Mass spectra were recorded on Agilent spectrometer. EPR analysis was performed on a JES-FA200 ESR Spectrometer in the X-band region (8.75–9.65 GHz). Elemental analysis was carried out on a Flash smart V CHNS/O.

2. Experimental Section:

a) Synthesis of ligand precursors and N-((8-((diphenylphosphaneyl)oxy)quinolin-2-yl)methyl)-2,6-diisopropylaniline (PONN^H)

(2-(((2,6-diisopropylphenyl)amino)methyl)quinolin-8-ol) was prepared according to the procedure in literature.[1] 8-hydroxy-2-quinolinecarbaldehyde (1g, 5.83 mmol) which was obtained by oxidation of 8-methyl quinolinol by SeO_2, was heated in ethanol (30 mL) to 80 ^{o}C, followed by dropwise addition of ethanol solution of phenylamine (540 mg, 5.83 mmol in 30 mL ethanol). This reaction mixture was allowed to reflux for 6 h and then cooled to room temperature. The precursor was obtained in pure form by column chromatography using 1/3 dichloromethane/ petroleum ether (1% triethylamine) in the form of yellow needles (yield = 58%).

(2-(((2,6-diisopropylphenyl)amino)methyl)quinolin-8-ol) (1004 mg, 3 mmol) was dissolved in 20 ml toluene in a 100 mL Schlenk tube under nitrogen atmosphere, and an equimolar amount of triethyl amine (418 µL, 3 mmol) was added to it dropwise, while stirring vigorously. After 5 min., 3 mmol (540 µL) of chlorodiphenylphosphine was also added dropwise, while stirring the solution vigorously at room temp. Now the Schlenk tube was closed tightly and kept for heating at 80 ^{o}C overnight. The reaction mixture was cooled to room temperature and the solvent was evaporated under vacuum. The residue was extracted with 20 mL of toluene and filtered through celite. Again the solvent was evaporated under vacuum. This product was again washed with dried hexane, and finally obtained as colourless oily liquid in 58% yield after drying in vacuum. The purity of the ligand was checked with ^{31}P NMR data. ^{31}P NMR (203 MHz, Chloroform-*d*) δ 117.3. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 8.4 Hz, 1H), 7.70 (dd, *J* = 11.5, 7.6 Hz, 1H), 7.63 (dd, *J* = 13.7, 7.5 Hz, 1H), 7.51 (t, *J* = 7.9 Hz, 6H), 7.29 (dt, *J* = 8.3, 4.1 Hz, 1H), 7.22 (d, *J* = 7.3 Hz, 1H), 7.16 (t, *J* = 7.3 Hz, 6H), 7.10 (d, *J* = 7.7 Hz, 1H), 7.07 (d, *J* = 8.2 Hz, 1H), 7.00 (d, *J* = 7.7 Hz, 1H), 6.84 (s, 1H), 4.78 (s, 1H), 3.60 (p, *J* = 6.6 Hz, 2H), 1.06 (d, *J* = 6.6 Hz, 12H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 152.78, 143.44, 135.40 (d, *J* = 7.1 Hz), 132.59 (d, *J* = 2.8 Hz), 131.85, 131.66, 131.51, 131.42 – 131.17 (m), 130.79, 130.70, 130.38, 129.61, 128.97, 128.87, 128.64 (d, *J* = 7.1 Hz), 128.45 (dd, *J* = 14.3, 9.7 Hz), 119.95, 117.53, 113.03, 45.82, 28.34, 24.49. IR frequencies (ATR): 3380 (N-H), 2962 (C=C-H), 2861 (C-C-H), 1589 (aryl C-H).



Scheme S1: Synthetic access to ligand N-((8-((diphenylphosphaneyl)oxy)quinolin-2-yl)methyl)-2,6-diisopropylaniline ($PONN^{H}$)

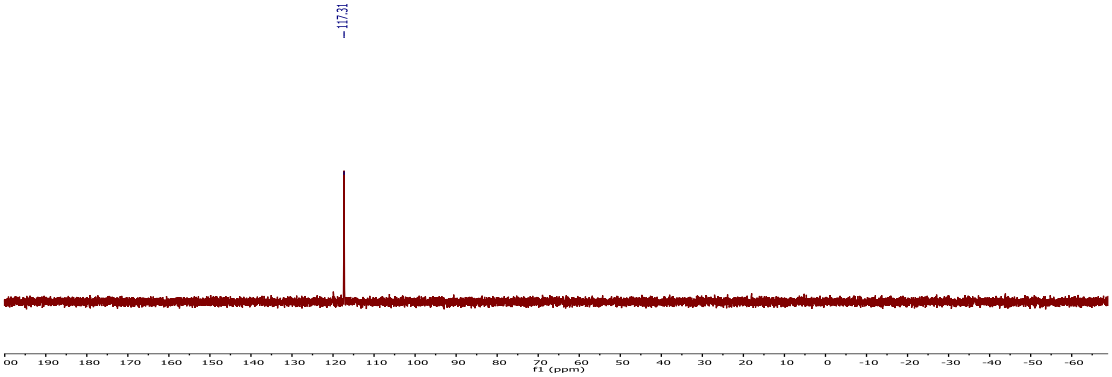


Figure S1. ^{31}P NMR spectrum of $PONN^{H}$ in $CDCl_3$ at room temperature.

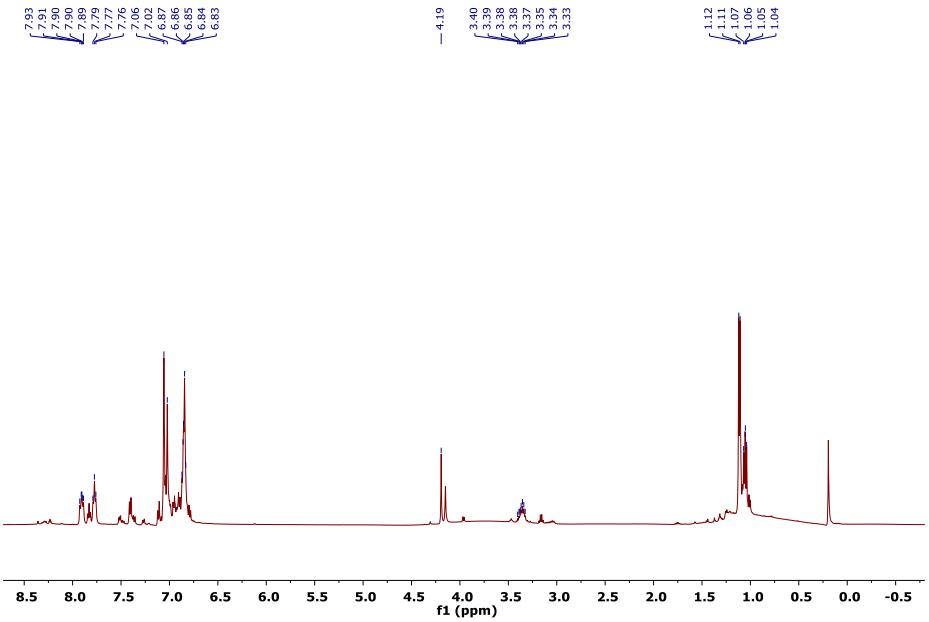


Figure S2: ^{1}H NMR spectrum of the PONN^{H} ligand in C_6D_6 at room temperature (contains precursor impurities).

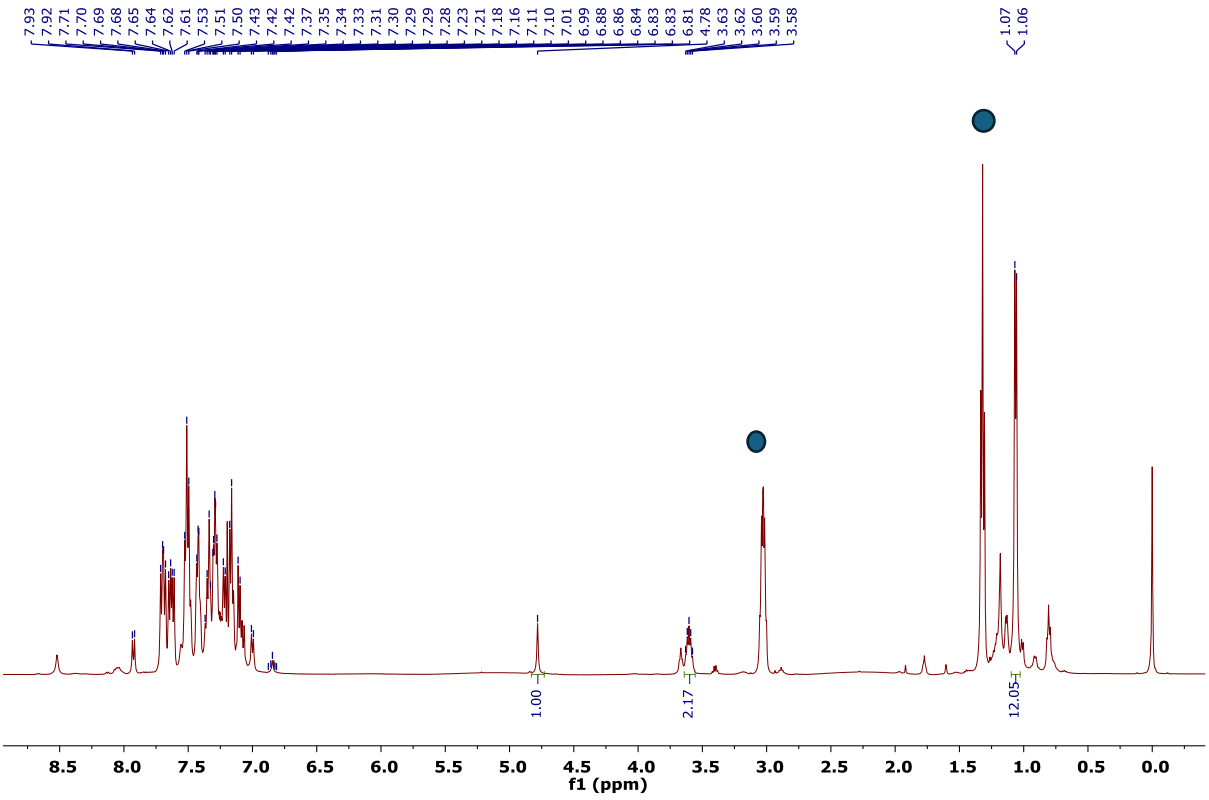


Figure S3: ^{1}H NMR spectrum of the crude reaction mixture for the synthesis of PONN^{H} ligand in C_6D_6 at room temperature containing Et_3NH^+ salt ● .

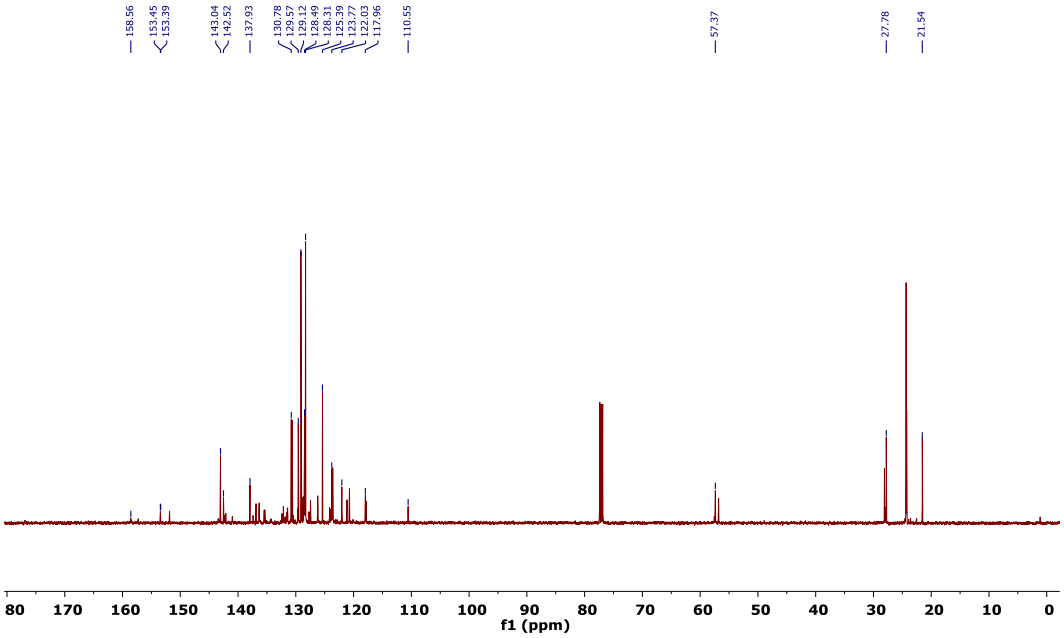


Figure S4: ^{13}C NMR spectrum of PONN^{H} in $CDCl_3$ at room temperature.

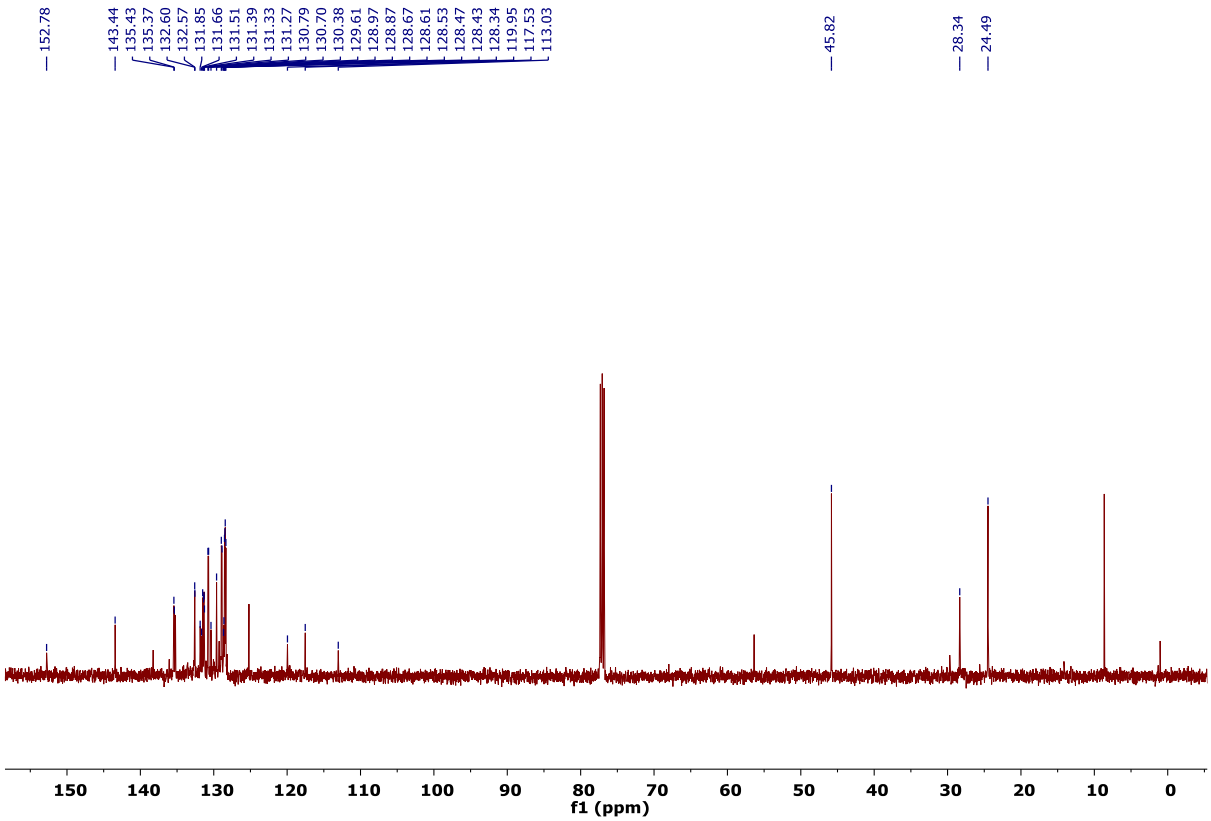


Figure S5: ^{13}C NMR spectrum of the crude reaction mixture of PONN^{H} in $CDCl_3$ at room temperature.

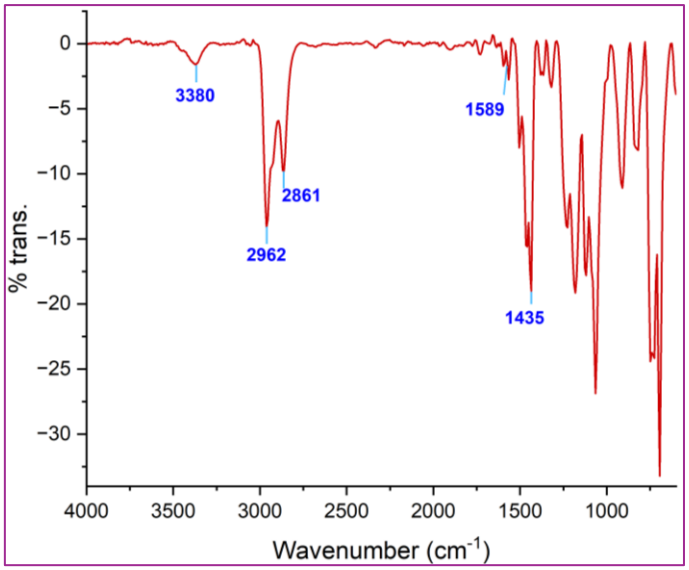
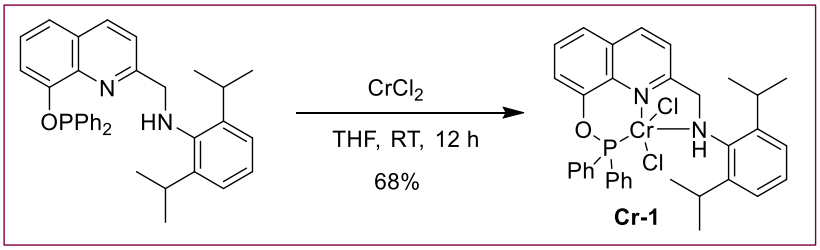


Figure S6: IR data for **PONN^{H}** ligand (ATR)

b) Synthesis of $CrCl_2$(PONN^{H}) (Cr-1)

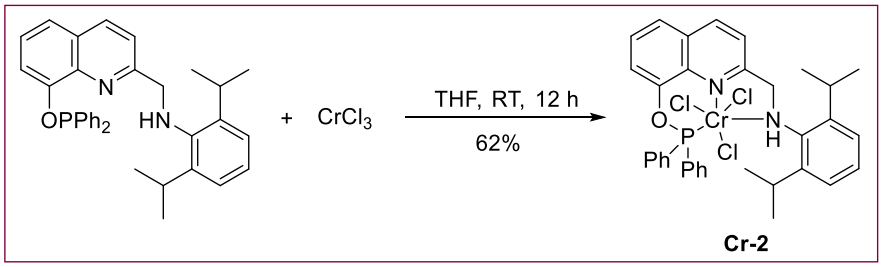
To a suspension of 5 mmol $CrCl_2$ in THF, 1 equiv. of PONN^{H} ligand was added dropwise and stirred at room temperature overnight. Dark green coloured suspension formed was washed twice with hexane and dried in vacuo to get green crystalline powder. **^{1}H NMR (500 MHz, Benzene-d_6)** δ 7.32 (s, 2H), 7.08 – 7.02 (m, 44H), 7.00 (s, 3H), 4.79 (s, 0H), 4.27 (s, 1H), 2.25 (s, 1H), 2.11 (s, 1H), 1.55 (s, 2H). **Selected IR data (ATR, cm^{-1}):** 3054 (N-H), 2959 (C-H) 1564(N-H). **UV–vis (DCM)** λ/nm: 274nm (sharp), 374 (broad). **HRMS** calcd. for $C_{34}H_{35}Cl_2CrN_2OP$ + Na^+: 663.1161, observed: 663.4545. **Room temperature X-band EPR, g value:** 2.002. Chemical Formula: $C_{34}H_{35}Cl_2CrN_2OP$. **Elemental Analysis (calculated) :** C, 63.66; H, 5.50; Cl, 11.05; Cr, 8.10; N, 4.37; O, 2.49; P, 4.83. **Observed:** C, 59.205; H, 5.888; N, 3.736.



Scheme S2: Preparation of **Cr-1**.

c) Synthesis of $CrCl_3$(PONN^{H}):

To a suspension of 80 mg (0.5 mmol) $CrCl_3$ in THF, 1 equiv. of THF solution of PONN^{H} ligand (260 mg) was added dropwise and stirred at room temperature overnight. The brown coloured suspension so formed was dried in vacuo and washed twice with hexane to get golden brown powder in 62% yield. HRMS calcd. for $C_{34}H_{35}Cl_3CrN_2OP$+2Na^+: 721.0958, observed: 721.0955, for $C_{34}H_{35}Cl_3CrN_2OP$+ 2Na^+ + 2Li^+: 735.0958, observed: 735.1824; for $C_{34}H_{35}Cl_3CrN_2OP$ - Cl^- + Na^+ 663.0998: , observed: 663.1467 .



Scheme S3: Preparation of **Cr-2.**

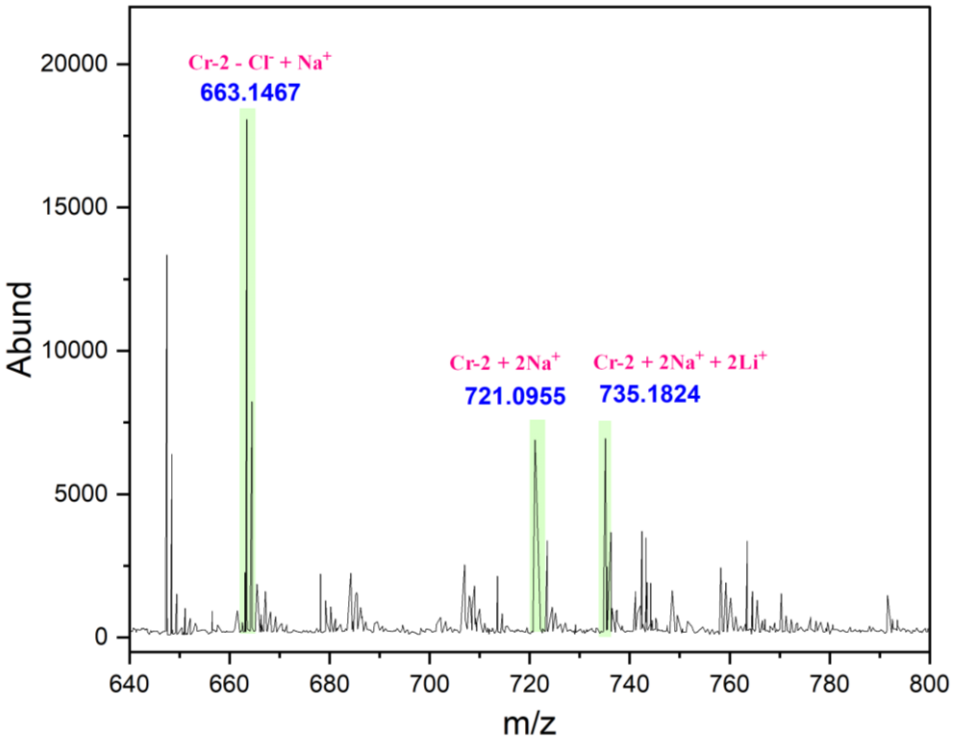


Figure S7: HRMS spectrum of **Cr-2**.

d) **Determination of Magnetic Susceptibility by Evans Method.**[3, 4]

Weight of catalyst taken for NMR measurement = 2.5 mg

Molecular mass of Cr-1 = 641.5369

Shift in ^{1}H NMR peak of CDCl3 (400 MHz) ($\Delta\nu$) = 0.1156ppm = 46.24Hz

By using the following formula for calculating χ_m:

$$\chi_m = \frac{\Delta\nu}{S_f \nu_0} \times \frac{1000}{c}$$

Where, $S_f = \frac{4\pi}{3}$, the value of χ_m was calculated to be equal to 0.00354 cm^{3}mol^{-1}.

Putting this value in following equation, (taking the value of $\chi_m = \chi^{para}$), we get:

$$\mu_{eff} = \sqrt{8 X_m^{para} T}$$

Where T = 298K, $\mu_{eff} = \sqrt{8 \cdot 225} = 2.86$.

3. Spectra of various studies of Cr-1 and catalytic reactions

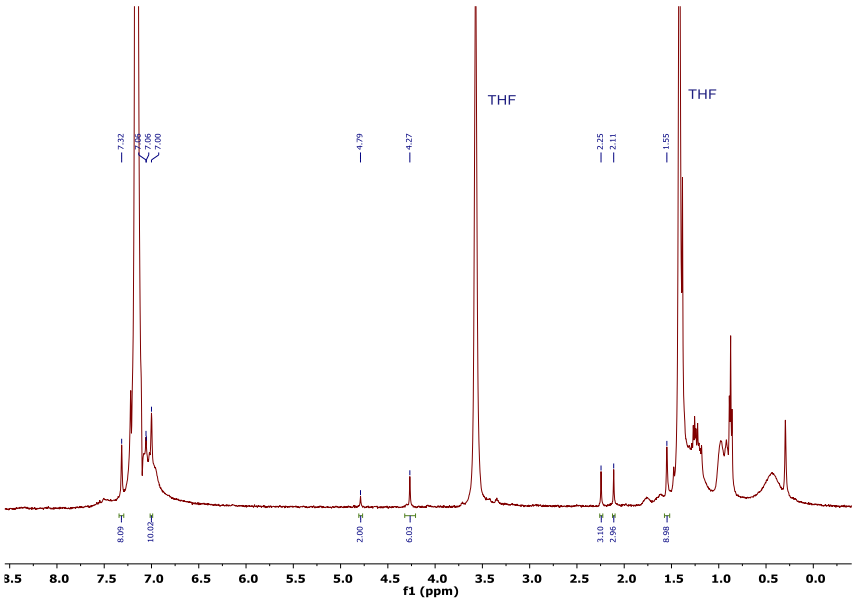


Figure S8: ^{1}H NMR for **Cr-1**.

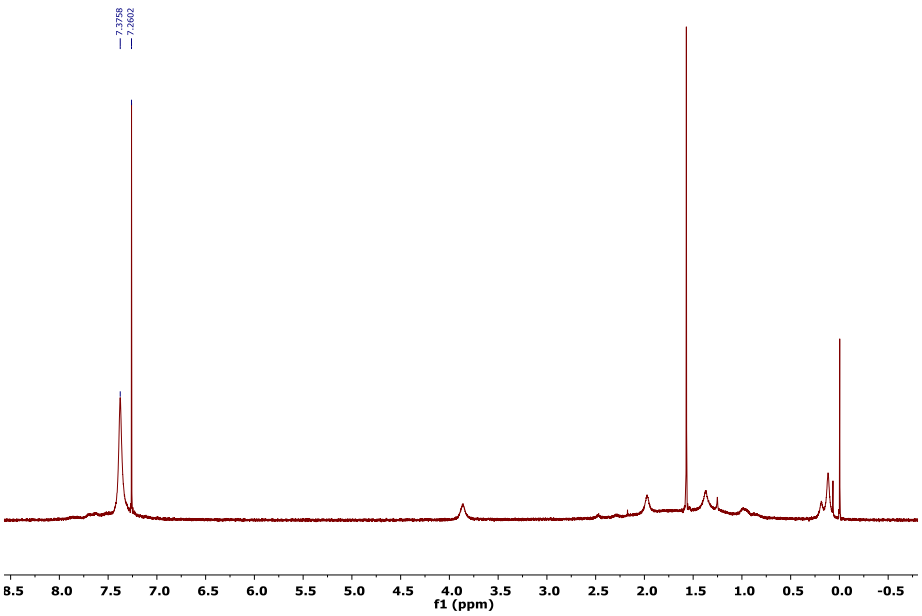


Figure S9: ^{1}H NMR data of **Cr-1** for the Evans measurement.

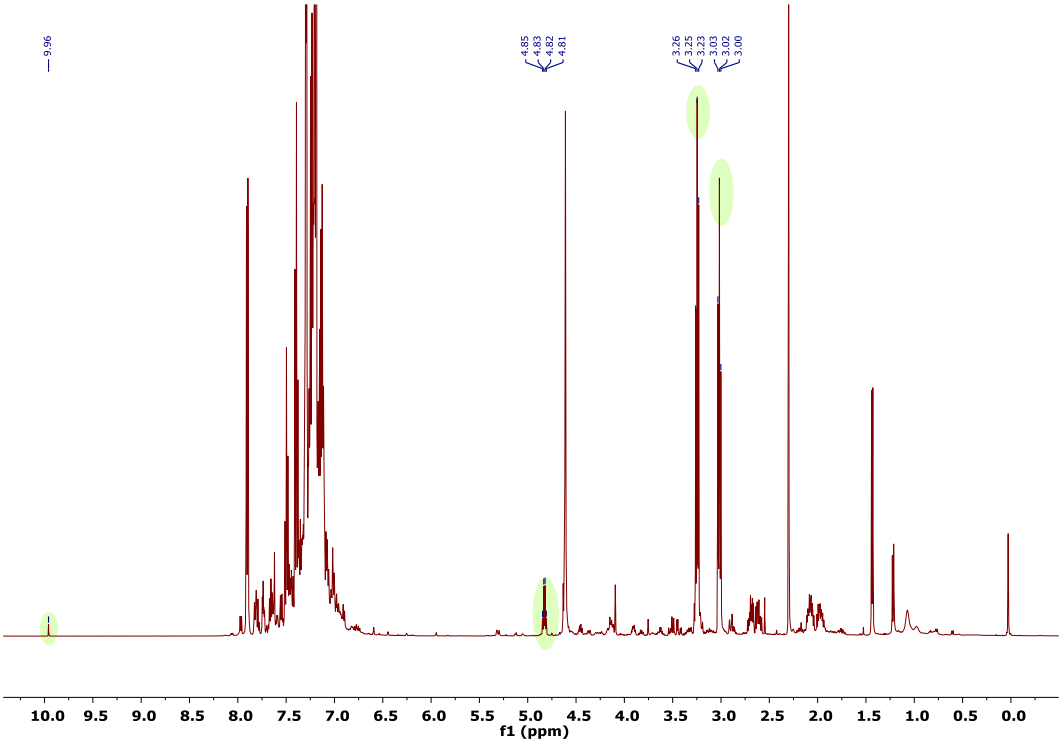


Figure S10: ^{1}H NMR of the catalytic reaction mixture of benzyl alcohol and acetophenone after 3h at 135 ^{o}C.

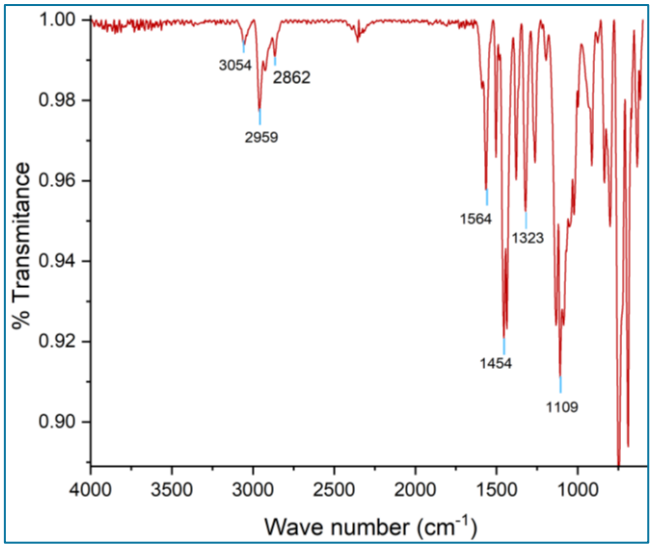


Figure S11: IR data for **Cr-1**.

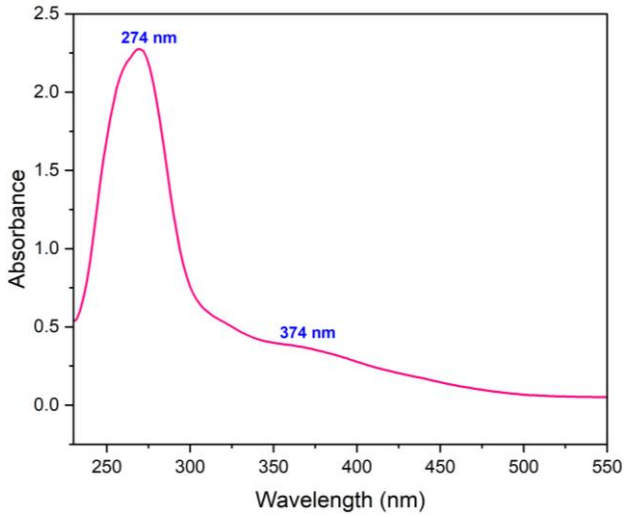


Figure S12: UV-Vis. spectrum of **Cr-1** in DCM.

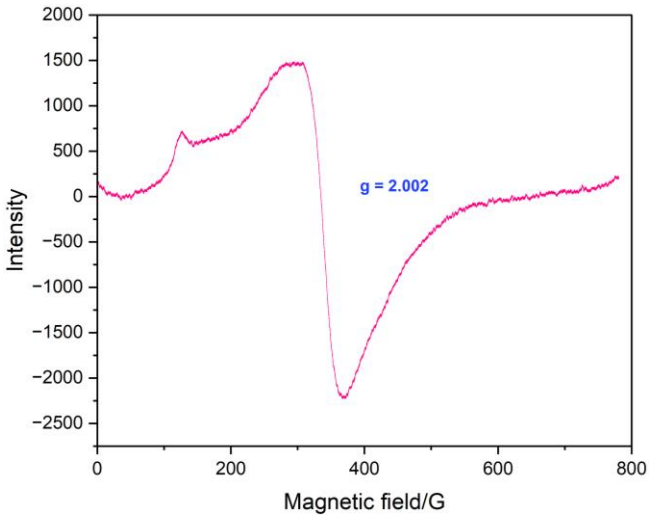


Figure S13: Room temperature X-band EPR for **Cr-1**.

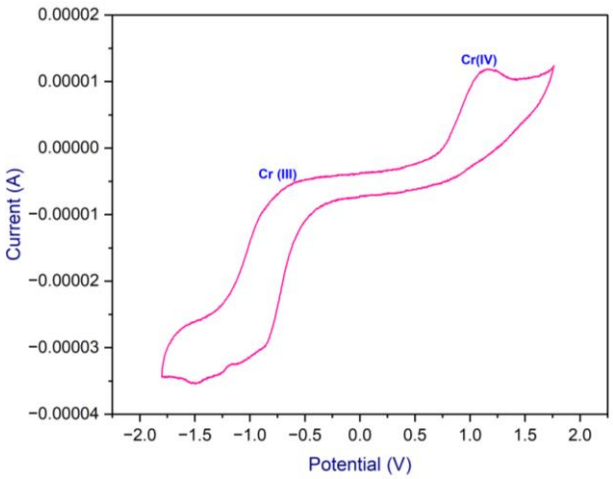


Figure S14: CV curve of **Cr-1**. Conditions: 1.0 mM analyte in 0.1 M $TBAPF_6$/MeCN, under dry argon atmosphere, glassy carbon working electrode, platinum wire counter electrode, Ag/AgCl reference electrode, 0.02 V/s scan rate.

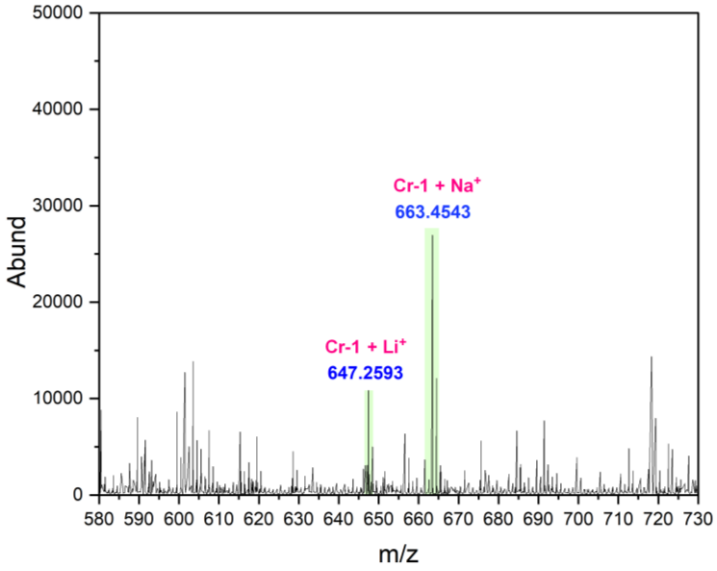


Figure S15: HRMS data for $CrCl_2(PONN^H)$ complex (m/z+Na^+ and m/z+ Li^+)

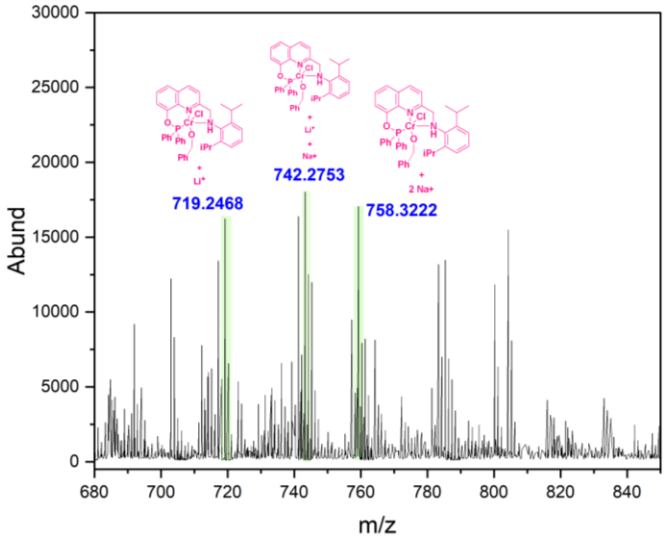


Figure S16: HRMS data for the formation of Alkoxy-complex **Cr-1b.**

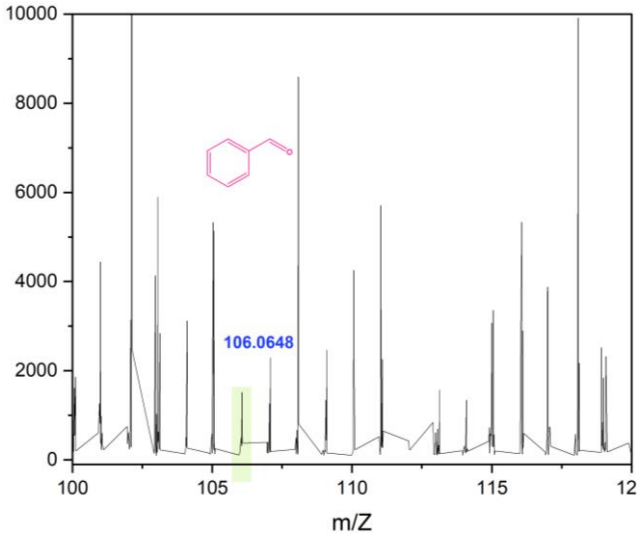


Figure S17: HRMS data for aldehyde formation during catalytic reaction via dehydrogenation of benzyl alcohol.

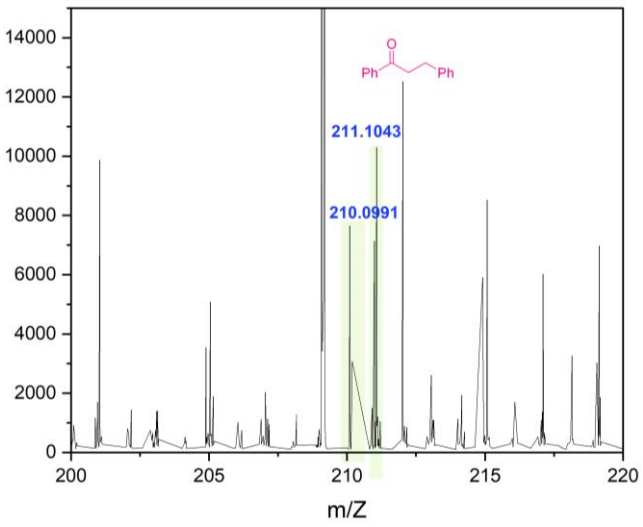


Figure S18: HRMS data for product formation 1,3-diphenylpropan-1-one (expected HRMS: m/z = 212.1201, observed = 212.0914)

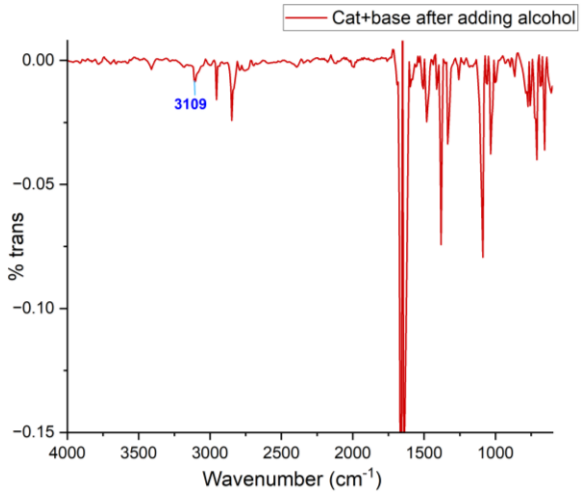


Figure S19: IR spectrum recorded after adding benzyl alcohol to the reaction mixture of complex **Cr-1** and *t*BuOK (ATR-IR).

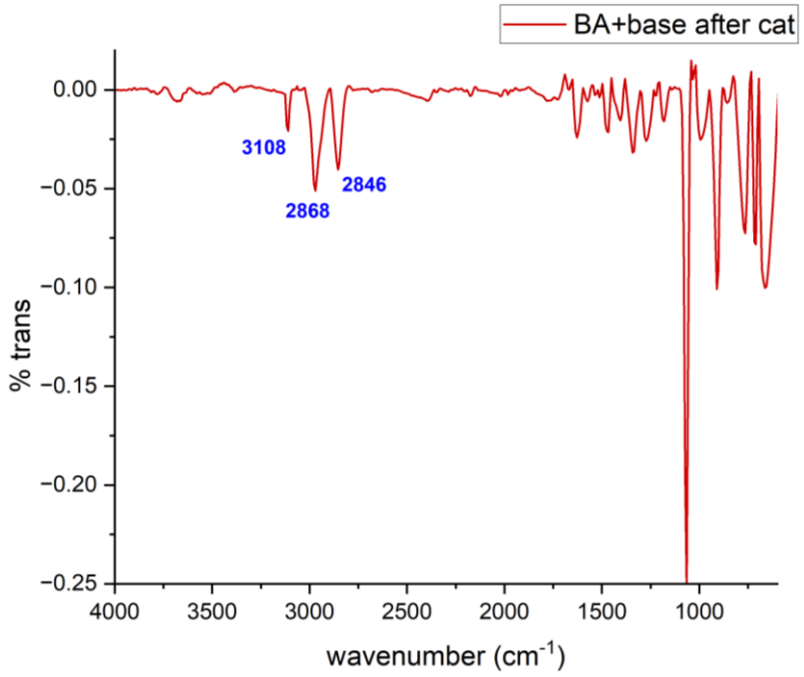
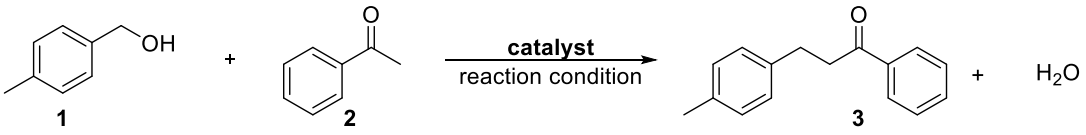


Figure S20: IR spectrum recorded after adding complex **Cr-1** to potassium benzyloxy species (formed by the reaction of benzyl alcohol with *t*BuOK) (ATR-IR).

5. General procedure for the catalytic reactions of Table S1:

0.5 mmol of *p*-methyl benzyl alcohol and 0.5 mmol of acetophenone were added to 0.015 mmol of catalyst **Cr-1** and 20 mol% of ^{t}BuOK (or other bases, as mentioned in Table 1) and the mixture was dissolved in 1.5 mL of toluene (or t-Amyl alcohol or 1,4- dioxane or THF; Table 1, entries 11, 12, 13, and 14 respectively) and placed in a 25 mL Schlenk tube under N_2. The tube was heated at 120-135 ºC with stirring for the mentioned time. The reaction mixture was then cooled down and the products formation was monitored using TLC. Products were analyzed by NMR spectroscopy using 1,1,2,2-tetrachloroethane as internal standard.

6. Table S1: Optimization table for the α-alkylation of acetophenone using *p*-methylbenzyl alcohol.[a]



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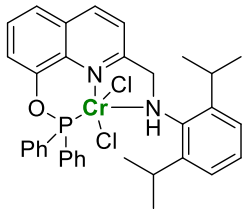
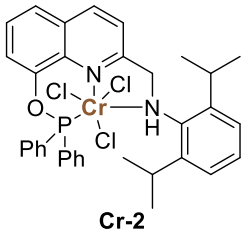


Cr-1



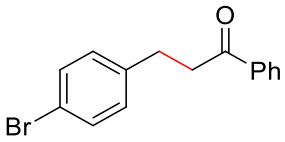
Cr-2

entry	base (mol %)	catalyst (mol %)	solvent (mL)	temperature (°C)	time (h)	yield(%)
1	KO^{t}Bu (20%)	**Cr-1** (3)	Toluene (1.5)	120	24	62
2[b]	KO^{t}Bu (20%)	**Cr-1** (3)	Toluene (1.5)	135	24	76
3	KO^{t}Bu (20%)	**Cr-1** (3)	Toluene (1.5)	135	24	88
4	KO^{t}Bu (20%)	**Cr-1** (3)	Toluene (1.5)	135	36	92
5	NaOH (20%)	**Cr-1** (3)	Toluene (1.5)	135	24	84
6	KOH (20%)	**Cr-1** (3)	Toluene (1.5)	135	24	80
7	NaO^{t}Bu (20%)	**Cr-1** (3)	Toluene (1.5)	135	24	78
8	Cs_2CO_3 (20%)	**Cr-1** (3)	Toluene (1.5)	135	24	86
9	LiO^{t}Bu (20%)	**Cr-1** (3)	Toluene (1.5)	135	24	84
10	-	**Cr-1** (3)	Toluene (1.5)	135	24	<5
11	KO^{t}Bu (20%)	**Cr-1** (3)	*tert*-amyl alcohol (1.5)	135	24	50
12	KO^{t}Bu (20%)	**Cr-1** (3)	Dioxane (1.5)	135	24	trace
13	Cs_2CO_3 (20%)	**Cr-1** (3)	Dioxane (1.5)	135	24	trace
14	KO^{t}Bu (20%)	**Cr-1** (3)	THF(1.5)	135	24	trace
15[c]	KO^{t}Bu (20%)	**Cr-1** (3)	Toluene (1.5)	135	36	98
16[c]	KO^{t}Bu (20%)	**Cr-1** (3)	chlorobenzene (1.5)	135	36	45
17[c]	KO^{t}Bu (20%)	**Cr-1** (3)	mesitylene (1.5)	135	36	71
18[c]	KO^{t}Bu (20%)	-	Toluene (1.5)	135	36	trace
19[c]	KO^{t}Bu (20%)	**$CrCl_2$** (3)	Toluene (1.5)	135	36	20
20[c]	LiOH (20%)	**Cr-1** (3)	Toluene (1.5)	135	36	87
21[b,c]	KO^{t}Bu (20%)	**Cr-2** (3)	Toluene (1.5)	135	24	59
22[d]	NaO^{t}Bu (5%)	**Cr-2** (0.005)	-	140	3	6
23[d]	NaO^{t}Bu (20%)	**Cr-2** (3)	Toluene (1)	135	24	54

[a]Reaction condition: *p*-methyl benzyl alcohol (0.5 mmol) and acetophenone (0.5 mmol). [b]Using Benzyl alcohol as substrate. [c]Yields were determined by ^{1}H NMR analysis based on substrate consumption or 1,1,2,2-tetrachloroethane as internal standard. [d]Benzyl alcohol (2 mmol) and 1-phenylethanol (2 mmol). Yields were determined by ^{1}H NMR analysis based on substrate consumption or 1,1,2,2-tetrachloroethane as internal standard.

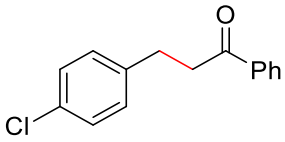
7. Analytical data of isolated products and NMR spectra

3a) 3-(4-bromophenyl)-1-phenylpropan-1-one[5]



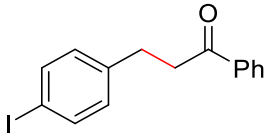
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 7.0 Hz, 2H), 7.62 – 7.56 (m, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.44 (d, *J* = 8.3 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 3.31 (t, *J* = 7.5 Hz, 2H), 3.06 (t, *J* = 7.5 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.27, 140.73, 137.20, 133.65, 132.01, 130.71, 129.11, 128.47, 120.35, 40.53, 29.89.

3b) 3-(4-chlorophenyl)-1-phenylpropan-1-one[5]



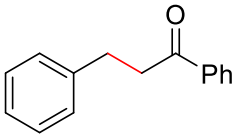
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.98 – 7.93 (m, 2H), 7.56 (d, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.26 (d, *J* = 8.3 Hz, 2H), 7.19 (d, *J* = 8.3 Hz, 2H), 3.28 (t, *J* = 7.5 Hz, 2H), 3.05 (t, *J* = 7.5 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.31, 140.19, 137.18, 133.64, 132.31, 130.29, 129.07 (d, *J* = 6.8 Hz), 128.46, 40.59, 29.82.

3c) 3-(4-iodophenyl)-1-phenylpropan-1-one[6]



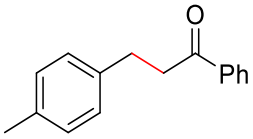
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 7.0 Hz, 2H), 7.61 (d, *J* = 8.3 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.01 (d, *J* = 8.3 Hz, 2H), 3.28 (t, *J* = 7.6 Hz, 2H), 3.02 (t, *J* = 7.5 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.25, 141.43, 138.01, 137.21, 133.66, 131.06, 129.12, 128.49, 91.67, 40.51, 30.00.

3d) 1,3-diphenylpropan-1-one[5]



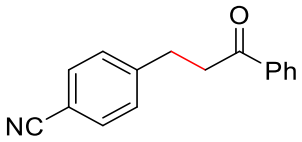
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 6.9 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.21 (d, *J* = 7.3 Hz, 2H), 7.18 (d, *J* = 6.5 Hz, 2H), 7.13 (t, *J* = 7.1 Hz, 1H), 3.23 (t, 2H), 2.99 (t, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.70, 141.76, 137.33, 133.53, 129.07, 129.00, 128.89, 128.51, 126.60, 40.92, 30.60.

3e) 1-phenyl-3-(p-tolyl)propan-1-one[5]



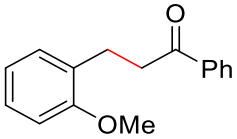
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 7.6 Hz, 2H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 7.8 Hz, 2H), 3.31 – 3.27 (m, 2H), 3.07 – 3.01 (m, 2H), 2.33 (s, 3H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.37, 138.21, 136.91, 135.65, 133.05, 129.23, 128.62, 128.32, 128.07, 40.64, 29.74, 21.03.

3f) 4-(3-Oxo-3-phenylpropyl)benzonitrile[7]



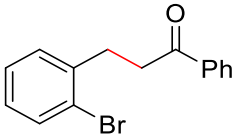
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 7.0 Hz, 2H), 7.57 (t, *J* = 7.6 Hz, 3H), 7.46 (t, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 3.33 (t, *J* = 7.4 Hz, 2H), 3.14 (t, *J* = 7.4 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 198.72, 147.48, 137.03, 133.83, 132.80, 129.82, 129.19, 128.47, 119.46, 110.56, 39.92, 30.47.

3g) 3-(2-methoxyphenyl)-1-phenylpropan-1-one[5]



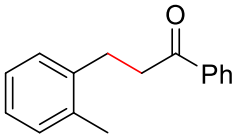
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.00 (d, *J* = 8.1 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.26 – 7.19 (m, 2H), 6.92 (d, *J* = 7.4 Hz, 1H), 6.88 (d, *J* = 8.6 Hz, 1H), 3.84 (s, 3H), 3.28 (t, *J* = 8.7, 6.8 Hz, 2H), 3.07 (t, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 200.41, 157.95, 137.41, 133.32, 130.59, 129.95, 128.95, 128.54, 127.94, 120.96, 110.68, 55.62, 39.37, 26.16.

3h) 3-(2-bromophenyl)-1-phenylpropan-1-one[8]



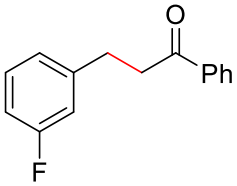
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 7.0 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.38 (t, *J* = 7.7 Hz, 2H), 7.24 (dd, *J* = 7.6, 1.8 Hz, 1H), 7.16 (dd, *J* = 7.5, 1.3 Hz, 1H), 7.00 (td, *J* = 7.7, 1.8 Hz, 1H), 3.24 (t, *J* = 7.9 Hz, 2H), 3.11 (t, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.41, 141.05, 137.23, 133.60, 133.37, 131.29, 129.09, 128.56, 128.46, 128.11, 124.84, 39.09, 31.28.

3i) 1-phenyl-3-(o-tolyl)propan-1-one[8]



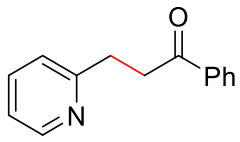
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.21 (t, *J* = 5.4 Hz, 1H), 7.19 – 7.14 (m, 3H), 3.30 – 3.25 (m, 2H), 3.11 – 3.06 (m, 2H), 2.38 (s, 3H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.41, 139.42, 136.88, 136.03, 133.27, 133.00, 130.39, 128.79, 128.55, 128.20, 126.37, 126.23, 39.15, 27.55, 19.48.

3j) 3-(3-fluorophenyl)-1-phenylpropan-1-one[8]



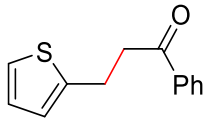
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 7.9 Hz, 2H), 7.55 (t, 1H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.23 (t, 1H), 7.02 (d, *J* = 7.6 Hz, 1H), 6.95 (d, *J* = 9.9 Hz, 1H), 6.88 (td, *J* = 8.6, 2.6 Hz, 1H), 3.29 (t, *J* = 8.2, 7.0 Hz, 2H), 3.06 (t, *J* = 7.6 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.24, 163.41 (d, *J* = 245.5 Hz), 144.32 (d, *J* = 7.1 Hz), 137.22, 133.64, 130.40 (d, *J* = 8.6 Hz), 128.49, 124.57 (d, *J* = 2.7 Hz), 115.79 (d, *J* = 20.8 Hz), 113.49 (d, *J* = 20.9 Hz), 40.44, 30.21 (d, *J* = 1.7 Hz). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -113.44.

3l) 1-phenyl-3-(pyridin-2-yl)propan-1-one[9]



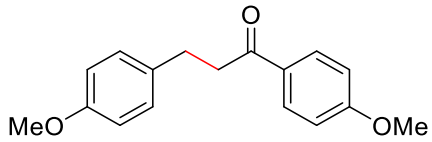
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.51 (d, *J* = 4.6 Hz, 1H), 7.98 (d, *J* = 6.9 Hz, 2H), 7.61 (td, *J* = 7.7, 1.9 Hz, 1H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.28 (d, *J* = 7.8 Hz, 1H), 7.12 (td, 1H), 3.52 (t, *J* = 7.2 Hz, 2H), 3.25 (t, *J* = 7.2 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.68, 161.01, 149.29, 137.29, 137.22, 133.49, 129.01, 128.55, 124.08, 121.83, 38.29, 32.32.

3m) 1-phenyl-3-(thiophen-2-yl)propan-1-one[9]



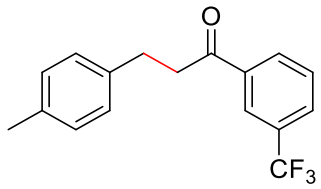
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 7.0 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 2H), 7.13 (d, *J* = 3.9 Hz, 1H), 6.93 (t, 1H), 6.87 (d, *J* = 2.3 Hz, 1H), 3.37 (t, *J* = 6.6 Hz, 2H), 3.31 (t, 2H). ^{13}C{1H} NMR (126 MHz, Chloroform-*d*) δ 199.07, 144.37, 137.23, 133.66, 129.12, 128.52, 127.34, 125.16, 123.86, 41.03, 24.70.

3p) 1,3-bis(4-methoxyphenyl)propan-1-one[8]



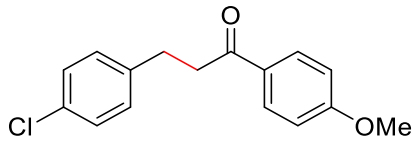
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 8.9 Hz, 2H), 7.17 (d, *J* = 8.6 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 3.86 (s, 3H), 3.79 (s, 3H), 3.22 (t, *J* = 7.7 Hz, 2H), 3.00 (dd, *J* = 8.4, 7.0 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 198.40, 163.85, 158.39, 133.92, 130.73, 130.44, 129.77, 114.35, 114.14, 55.88, 55.69, 40.79, 29.91.

3q) 3-(p-tolyl)-1-(3-(trifluoromethyl)phenyl)propan-1-one



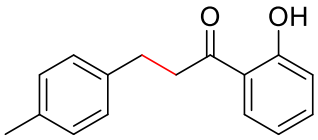
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.19 (s, 0H), 8.13 (d, *J* = 7.8 Hz, 0H), 7.81 (d, *J* = 7.8 Hz, 0H), 7.60 (t, *J* = 7.8 Hz, 1H), 7.14 (q, *J* = 8.0 Hz, 2H), 3.31 (t, *J* = 8.3, 6.9 Hz, 1H), 3.05 (t, *J* = 7.6 Hz, 1H), 2.33 (s, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 198.38, 138.26, 137.85, 136.32, 131.86, 131.62, 129.92, 129.77, 128.78, 125.39, 125.25, 41.24, 30.00, 21.47. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.80. HRMS: calculated: 292.1075; observed: 292.1298.

3s) 3-(4-chlorophenyl)-1-(4-methoxyphenyl)propan-1-one[8]



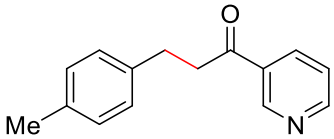
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 8.9 Hz, 1H), 7.17 (d, *J* = 8.2 Hz, 1H), 7.09 (d, *J* = 8.4 Hz, 1H), 6.84 (d, *J* = 8.9 Hz, 1H), 3.78 (s, 2H), 3.14 (t, *J* = 7.6 Hz, 1H), 2.94 (t, *J* = 7.5 Hz, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 197.87, 163.95, 140.35, 132.21, 130.73, 130.28, 129.00, 114.19, 55.92, 40.23, 29.98.

3v) 2-(4-methylbenzyl)-3,4-dihydronaphthalen-1(2H)-one[10]



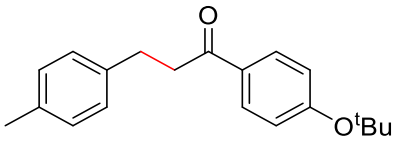
^{1}H NMR (500 MHz, Chloroform-*d*) δ 12.31 (s, 1H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.18 – 7.10 (m, 4H), 6.99 (d, *J* = 8.4 Hz, 1H), 6.91 – 6.85 (m, 1H), 3.31 (t, *J* = 7.7 Hz, 2H), 3.03 (t, *J* = 7.7 Hz, 2H), 2.33 (s, 3H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 205.91, 162.86, 138.00, 136.70, 136.24, 130.22, 129.67, 128.64, 119.68, 119.30, 118.95, 40.59, 30.02, 21.40.

3w) 1-(pyridin-3-yl)-3-(p-tolyl)propan-1-one



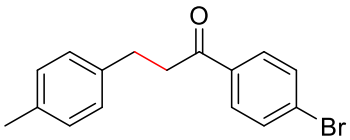
^{1}H NMR (500 MHz, Chloroform-*d*) δ 9.16 (s, 1H), 8.77 (d, *J* = 3.1 Hz, 1H), 8.23 (d, *J* = 7.9 Hz, 1H), 7.42 (dd, *J* = 8.0, 4.8 Hz, 1H), 7.13 (q, *J* = 8.1 Hz, 4H), 3.33 – 3.26 (m, 2H), 3.05 (t, *J* = 7.6 Hz, 2H), 2.32 (s, 3H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 198.28, 153.50, 149.66, 137.85, 136.08, 135.74, 132.37, 129.51, 128.50, 123.94, 41.09, 29.61, 21.23. HRMS: calculated = 225.1154. Observed m/z= 225.1627.

3x) 1-(4-(tert-butoxy)phenyl)-3-(p-tolyl)propan-1-one



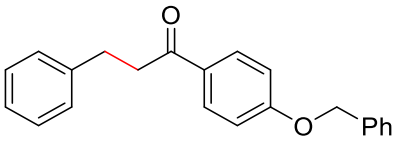
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.92 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 7.9 Hz, 2H), 7.12 (d, *J* = 7.8 Hz, 2H), 7.04 (s, 1H), 3.31 – 3.24 (m, 2H), 3.04 (t, *J* = 7.8 Hz, 2H), 2.34 (s, 3H), 1.35 (s, 9H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.05, 156.77, 138.36, 135.60, 134.35, 129.23, 128.33, 128.06, 125.56, 40.57, 35.13, 31.13, 29.84, 21.05. HRMS: calculated = 296.1776. Observed m/z = 296.2017.

3y) 1-(4-bromophenyl)-3-(p-tolyl)propan-1-one[10]



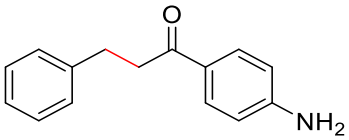
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.81 (d, *J* = 8.5 Hz, 2H), 7.59 (d, *J* = 8.6 Hz, 2H), 7.16 – 7.09 (m, 4H), 3.24 (t, *J* = 8.4, 7.0 Hz, 2H), 3.02 (t, *J* = 7.7 Hz, 2H), 2.33 (s, 3H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 198.50, 138.15, 135.96, 135.81, 132.13, 129.79, 129.46, 128.49, 128.41, 40.79, 29.84, 21.23.

3aa) 1-(4-benzylphenyl)-3-phenylpropan-1-one[11]



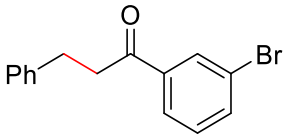
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 8.8 Hz, 1H), 7.32 (d, *J* = 1.8 Hz, 0H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.24 (d, *J* = 7.2 Hz, 0H), 7.18 (d, *J* = 7.0 Hz, 1H), 7.14 (d, *J* = 7.3 Hz, 1H), 7.10 (td, *J* = 7.8, 7.3, 2.9 Hz, 1H), 7.03 (d, *J* = 7.0 Hz, 0H), 6.89 (d, *J* = 8.8 Hz, 1H), 5.00 (s, 1H), 3.13 (t, *J* = 7.7 Hz, 1H), 2.95 (t, *J* = 7.9 Hz, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 197.86, 162.64, 141.52, 136.23, 130.39, 129.08, 128.77, 128.58, 128.51, 128.45, 128.33, 127.56, 126.16, 114.64, 70.17, 40.19, 30.36.

3ab) 1-(4-aminophenyl)-3-phenylpropan-1-one[8]



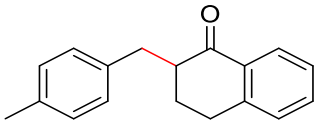
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 8.6 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 3H), 7.25 – 7.23 (m, 1H), 7.22 – 7.15 (m, 2H), 6.63 (d, *J* = 8.7 Hz, 2H), 3.20 (dd, *J* = 8.8, 6.7 Hz, 2H), 3.08 – 3.02 (m, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 148.56, 147.66, 135.72, 134.26, 129.39, 128.50, 123.85, 71.50, 40.79, 31.63, 21.20.

3ac) 1-(3-bromophenyl)-3-phenylpropan-1-one[12]



^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.99 (s, 1H), 7.78 (dt, *J* = 7.8, 1.3 Hz, 1H), 7.59 (ddd, *J* = 7.9, 2.0, 1.0 Hz, 1H), 7.25 – 7.23 (m, 2H), 7.21 (d, *J* = 7.4 Hz, 1H), 7.17 (d, *J* = 2.0 Hz, 2H), 7.15 (d, 1H), 7.12 (d, 1H), 3.19 (t, 2H), 2.98 (t, *J* = 7.6 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 198.01, 141.19, 138.80, 136.14, 131.38, 130.44, 129.20, 128.64, 126.76, 126.48, 123.22, 40.76, 30.18.

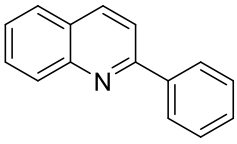
3ad) 8-hydroxy-2-(4-methylbenzyl)-3,4-dihydronaphthalen-1(2H)-one[10]



^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.07 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.22 (d, *J* = 7.8 Hz, 1H), 7.12 (s, 4H), 3.45 (dd, *J* = 13.8, 4.1 Hz, 1H), 3.00 – 2.87 (m, 3H), 2.73 (ddt, *J* = 11.6, 9.6, 4.3 Hz, 1H), 2.61 (dd, *J* = 13.8, 9.6 Hz, 1H), 2.33 (s, 1H), 2.12 (dq, *J* = 13.5, 4.5 Hz, 1H), 1.79 (dtd, *J* = 13.4, 11.6, 5.1 Hz, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 199.75, 144.28, 137.12, 135.85, 133.47, 132.73, 129.37, 129.32, 128.93, 127.77, 126.83, 49.74, 35.43, 28.82, 27.85, 21.26.

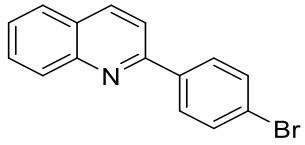
Friedländer Quinoline Synthesis

6a) 2-phenylquinoline[13]



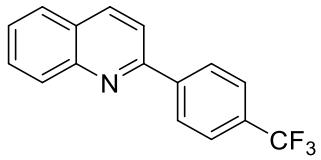
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.22 (t, *J* = 9.2 Hz, 2H), 8.18 (d, *J* = 7.1 Hz, 2H), 7.88 (d, *J* = 8.5 Hz, 1H), 7.83 (d, *J* = 8.1, 1.4 Hz, 1H), 7.74 (t, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.54 (t, 3H), 7.48 (t, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 157.38, 148.22, 139.61, 136.90, 129.75, 129.70, 129.40, 128.88, 127.64, 127.49, 127.22, 126.35, 119.06.

6b) 2-(4-bromophenyl)quinoline[13]



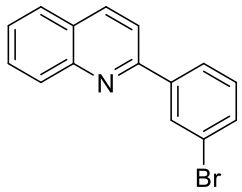
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.21 (d, *J* = 8.6 Hz, 1H), 8.17 (d, *J* = 8.5 Hz, 1H), 8.06 (d, *J* = 8.5 Hz, 2H), 7.82 (d, *J* = 8.6 Hz, 2H), 7.74 (t, *J* = 7.7 Hz, 1H), 7.65 (d, *J* = 8.5 Hz, 2H), 7.54 (t, *J* = 7.5 Hz, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 156.48, 148.67, 138.92, 137.47, 132.44, 130.34, 130.15, 129.56, 127.96, 127.71, 127.00, 124.41, 118.96.

6c) 2-(4-(trifluoromethyl)phenyl)quinoline[13]



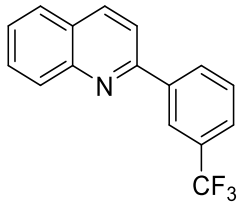
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.28 (d, *J* = 8.1 Hz, 2H), 8.24 (d, *J* = 8.6 Hz, 1H), 8.20 (d, *J* = 8.6 Hz, 1H), 7.89 – 7.82 (m, 2H), 7.80 – 7.73 (m, 3H), 7.56 (ddd, *J* = 8.1, 6.8, 1.2 Hz, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 156.09, 148.71, 143.38, 137.59, 131.54, 130.46, 130.31, 128.30, 127.99, 127.90, 127.32, 126.20, 125.77, 119.21. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.41.

6d) 2-(3-bromophenyl)quinoline[13]



^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.37 (s, 1H), 8.23 (d, *J* = 8.6 Hz, 1H), 8.19 (d, *J* = 8.5 Hz, 1H), 8.08 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 8.6 Hz, 2H), 7.77 – 7.74 (m, 1H), 7.60 (d, *J* = 6.9 Hz, 1H), 7.55 (d, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 7.9 Hz, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 155.82, 148.38, 141.84, 135.28, 132.44, 130.84, 130.53, 130.12, 129.97, 127.70, 127.55, 126.88, 126.27, 123.36, 118.89.

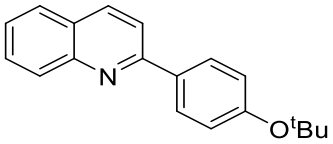
6e) 2-(3-(trifluoromethyl)phenyl)quinoline[14]



^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.48 (s, 1H), 8.36 (d, *J* = 7.8 Hz, 1H), 8.26 (d, *J* = 8.6 Hz, 1H), 8.21 (d, *J* = 8.5 Hz, 1H), 7.89 (d, *J* = 8.6 Hz, 1H), 7.85 (d, *J* = 8.2 Hz, 1H), 7.81 – 7.70 (m, 3H), 7.65 (t, *J* = 7.8 Hz, 1H), 7.57 (t, *J* = 7.5 Hz, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 155.78, 148.43, 140.54, 137.42, 131.68 –

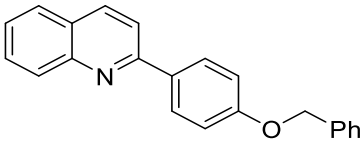
131.08 (m), 130.94, 130.23, 130.01, 129.53, 127.74, 127.02, 126.12, 124.64, 118.80. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.52.

6f) 2-(4-(tert-butoxy)phenyl)quinoline



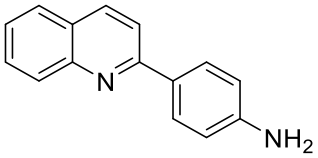
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.21 (d, *J* = 8.5 Hz, 2H), 8.11 (d, *J* = 8.5 Hz, 2H), 7.87 (d, *J* = 8.6 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.73 (ddd, *J* = 8.4, 6.8, 1.5 Hz, 1H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.52 (t, *J* = 6.9 Hz, 1H), 1.39 (s, 9H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 157.58, 152.86, 148.40, 129.87, 129.82, 127.67, 127.56, 127.32, 126.37, 126.07, 119.20, 34.98, 31.52. HRMS: calculated = 277.1467, observed = 277.1758, 278.1505.

6g) 2-(4-(benzyloxy)phenyl)quinoline[15]



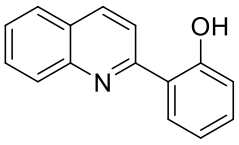
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.17 (dd, *J* = 11.2, 8.6 Hz, 4H), 7.82 (dd, *J* = 13.3, 8.4 Hz, 2H), 7.72 (t, *J* = 7.6 Hz, 1H), 7.50 (dd, *J* = 17.6, 7.1 Hz, 3H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.35 (t, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 8.8 Hz, 2H), 5.16 (s, 2H). ^{13}C {^{1}H}NMR (500 MHz, Chloroform-*d*) δ 160.24, 157.02, 136.99, 136.96, 129.86, 129.63, 129.16, 128.84, 128.25, 127.70, 127.64, 127.12, 126.19, 118.77, 115.40, 70.27, 28.20, 24.38.

6i) 4-(quinolin-2-yl)aniline[16]



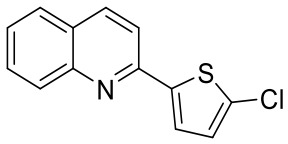
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.15 (d, *J* = 8.6 Hz, 2H), 8.04 (d, *J* = 8.4 Hz, 2H), 7.80 (dd, *J* = 12.2, 8.0 Hz, 2H), 7.69 (t, *J* = 7.7 Hz, 1H), 7.48 (t, *J* = 7.4 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 157.38, 148.16, 136.88, 129.79, 129.40, 129.13, 127.62, 127.00, 125.90, 118.62, 115.35.

6j) 2-(quinoline-2-yl)phenol[17]



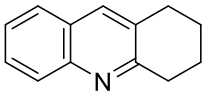
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.27 (d, *J* = 8.9 Hz, 1H), 8.04 (d, *J* = 9.0 Hz, 2H), 7.95 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.83 (d, *J* = 1.4 Hz, 1H), 7.74 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.55 (ddd, *J* = 8.1, 6.9, 1.1 Hz, 1H), 7.37 (ddd, *J* = 8.5, 7.1, 1.6 Hz, 1H), 7.11 (dd, *J* = 8.2, 1.3 Hz, 1H), 6.97 (t, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 161.46, 158.38, 145.06, 138.21, 132.59, 131.02, 128.02, 127.93, 127.45, 127.20, 127.00, 119.39, 119.25, 119.16, 117.76.

6k) 2-(5-chlorothiophen-2-yl)quinoline[18]



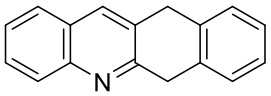
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.15 (d, *J* = 8.6 Hz, 2H), 7.77 (d, *J* = 8.1 Hz, 1H), 7.71 (d, *J* = 8.5 Hz, 2H), 7.50 (t, *J* = 7.7 Hz, 2H), 6.97 (d, *J* = 3.8 Hz, 1H).

6l) 1,2,3,4-tetrahydroacridine[19]



^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 8.5 Hz, 1H), 7.83 (s, 1H), 7.73 – 7.68 (m, 1H), 7.61 (ddd, *J* = 8.4, 6.7, 1.5 Hz, 1H), 7.44 (ddd, *J* = 8.0, 6.7, 1.2 Hz, 1H), 3.15 (t, *J* = 6.5 Hz, 2H), 2.98 (t, *J* = 6.1 Hz, 2H), 2.03 – 1.97 (m, 2H), 1.93 – 1.87 (m, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 159.47, 146.47, 135.53, 131.25, 128.91, 128.22, 127.43, 127.12, 125.89, 33.58, 29.45, 23.37, 23.08.

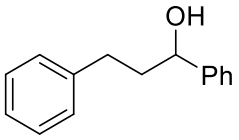
6m) 6,11-dihydrobenzo[b]acridine[20]



^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.60 (d, *J* = 7.8 Hz, 1H), 8.16 (d, *J* = 8.5 Hz, 1H), 7.92 (s, 1H), 7.75 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.66 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.51 – 7.42 (m, 2H), 7.38 (td, *J* = 7.4, 1.5 Hz, 1H), 7.29 (d, *J* = 1.4 Hz, 1H), 3.15 – 3.11 (m, 2H), 3.02 (dd, *J* = 8.4, 5.5 Hz, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 153.57, 147.76, 139.64, 134.85, 133.98, 130.80, 129.92, 129.57, 128.89, 128.16, 128.07, 127.55, 127.13, 126.29, 29.03, 28.61.

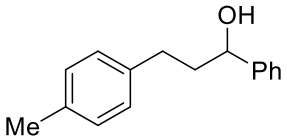
NMR data for β-alkylation of secondary alcohols:

8a) 1,3-diphenylpropan-1-ol[21]



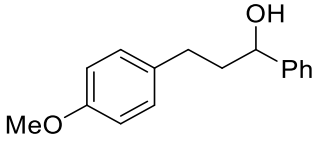
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.27 (d, *J* = 4.4 Hz, 4H), 7.19 (t, 3H), 7.11 (d, *J* = 7.6 Hz, 3H), 4.60 (dd, *J* = 7.8, 5.3 Hz, 1H), 2.75 – 2.46 (m, 2H), 2.10 – 1.92 (m, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 144.59, 141.80, 128.55, 128.47, 128.42, 127.68, 125.96, 125.89, 73.92, 40.49, 32.09.

8b) 1-phenyl-3-(p-tolyl)propan-1-ol[21]



^{1}H NMR (500 MHz, Chloroform-*d*) 7.27 (s, 4H), 7.21 (q, 1H), 7.02 (s, 4H), 4.60 (dd, *J* = 7.8, 5.4 Hz, 1H), 2.60 (dddd, *J* = 39.3, 14.1, 9.5, 6.1 Hz, 2H), 2.25 (s, 3H), 1.98 (dddd, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 145.07, 139.11, 135.73, 129.53, 128.95, 128.77, 128.05, 126.40, 74.33, 41.01, 32.04, 21.45.

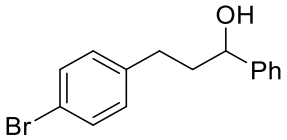
8c) 3-(4-methoxyphenyl)-1-phenylpropan-1-ol[22]



^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.25 (s, 4H), 7.19 (q, 1H), 7.02 (d, *J* = 8.7 Hz, 2H), 6.74 (d, *J* = 8.8 Hz, 2H), 4.57 (dd, *J* = 7.9, 5.3 Hz, 1H), 3.69 (s, 3H), 2.68 – 2.42 (m, 2H), 2.07 – 1.86 (m, 2H).

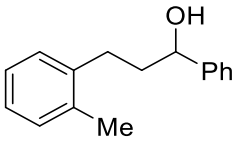
$^{13}C\{^{1}H\}$ NMR (126 MHz, Chloroform-*d*) δ 157.81, 144.68, 133.86, 129.36, 128.53, 127.63, 125.98, 113.86, 73.86, 55.29, 40.73, 31.16.

8d) 3-(4-bromophenyl)-1-phenylpropan-1-ol[22]



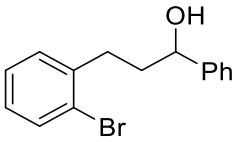
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.33 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 15.0 Hz, 1H), 7.25 – 7.21 (m, 1H), 7.00 (d, *J* = 8.0 Hz, 1H), 4.60 (dd, *J* = 7.9, 5.3 Hz, 1H), 2.60 (dddd, *J* = 30.1, 13.7, 9.5, 6.0 Hz, 2H), 2.09 – 1.81 (m, 2H). $^{13}C\{^{1}H\}$ NMR (126 MHz, Chloroform-*d*) δ 144.41, 140.77, 131.44, 130.24, 128.61, 127.78, 125.90, 119.60, 73.72, 40.26, 31.46.

8e) 1-phenyl-3-(o-tolyl)propan-1-ol[22]



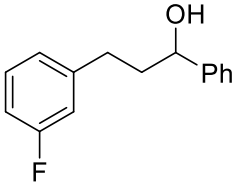
1H NMR (500 MHz, Chloroform-*d*) δ 7.43 – 7.35 (m, 4H), 7.33 (q, 1H), 7.19 – 7.10 (m, 4H), 4.76 (dd, *J* = 7.8, 5.3 Hz, 1H), 2.73 (dddd, *J* = 67.8, 14.1, 10.3, 5.7 Hz, 2H), 2.30 (s, 3H), 2.16 – 1.94 (m, 2H). $^{13}C\{^{1}H\}$ NMR (126 MHz, Chloroform-*d*) δ 144.60, 140.03, 136.00, 130.24, 128.77, 128.57, 127.70, 126.03, 126.00, 125.95, 74.25, 39.25, 29.46, 19.26.

8f) 3-(2-bromophenyl)-1-phenylpropan-1-ol[23]



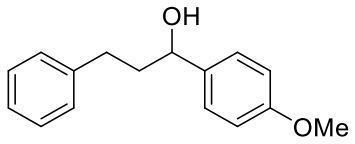
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 8.0 Hz, 1H), 7.39 (q, *J* = 7.0 Hz, 4H), 7.34 – 7.29 (m, 1H), 7.25 (d, *J* = 3.4 Hz, 1H), 7.08 (ddd, *J* = 8.8, 5.7, 3.5 Hz, 1H), 4.75 (dd, *J* = 7.9, 5.4 Hz, 1H), 2.87 (dddd, *J* = 62.9, 13.7, 9.8, 6.0 Hz, 2H), 2.15 – 2.01 (m, 3H). $^{13}C\{^{1}H\}$ NMR (126 MHz, Chloroform-*d*) δ 144.40, 141.18, 132.86, 130.44, 128.56, 127.71, 127.67, 127.49, 125.95, 124.48, 73.98, 38.92, 32.62.

8h) 3-(3-fluorophenyl)-1-phenylpropan-1-ol[21]



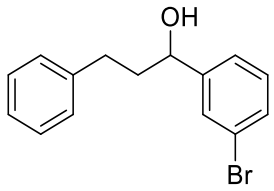
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.28 (d, *J* = 3.2 Hz, 3H), 7.22 (dq, *J* = 5.8, 3.1 Hz, 1H), 7.15 (q, *J* = 8.0, 6.2 Hz, 2H), 6.89 (d, *J* = 7.7 Hz, 1H), 6.81 (ddt, *J* = 13.3, 8.3, 4.0 Hz, 2H), 4.61 (dd, *J* = 7.9, 5.3 Hz, 1H), 2.71 – 2.56 (m, 2H), 2.11 – 1.89 (m, 2H). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -113.69.

8k) 1-(4-methoxyphenyl)-3-phenylpropan-1-ol[21]



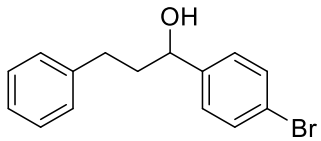
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.21 (q, 4H), 7.13 (dd, 3H), 6.83 (d, *J* = 8.7 Hz, 1H), 4.57 (dd, *J* = 7.8, 5.7 Hz, 0H), 3.74 (s, 1H), 2.72 – 2.53 (m, 1H), 2.13 – 1.92 (m, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 159.14, 141.88, 136.74, 128.47, 128.41, 127.25, 125.86, 113.92, 73.52, 55.32, 40.37, 32.16.

8l) 1-(3-bromophenyl)-3-phenylpropan-1-ol[21]



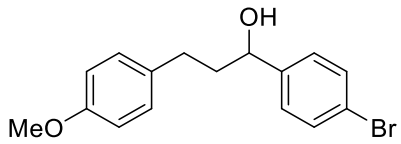
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.44 (d, *J* = 1.9 Hz, 1H), 7.34 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.21 (ddd, *J* = 13.1, 8.0, 4.1 Hz, 3H), 7.17 – 7.10 (m, 4H), 4.58 (ddd, *J* = 7.7, 5.1, 2.2 Hz, 1H), 2.74 – 2.55 (m, 2H), 2.08 – 1.88 (m, 2H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 146.99, 141.46, 130.67, 130.12, 129.07, 128.50, 128.46, 126.02, 124.55, 122.67, 73.16, 40.52, 31.94.

8m) 1-(4-bromophenyl)-3-phenylpropan-1-ol[21]



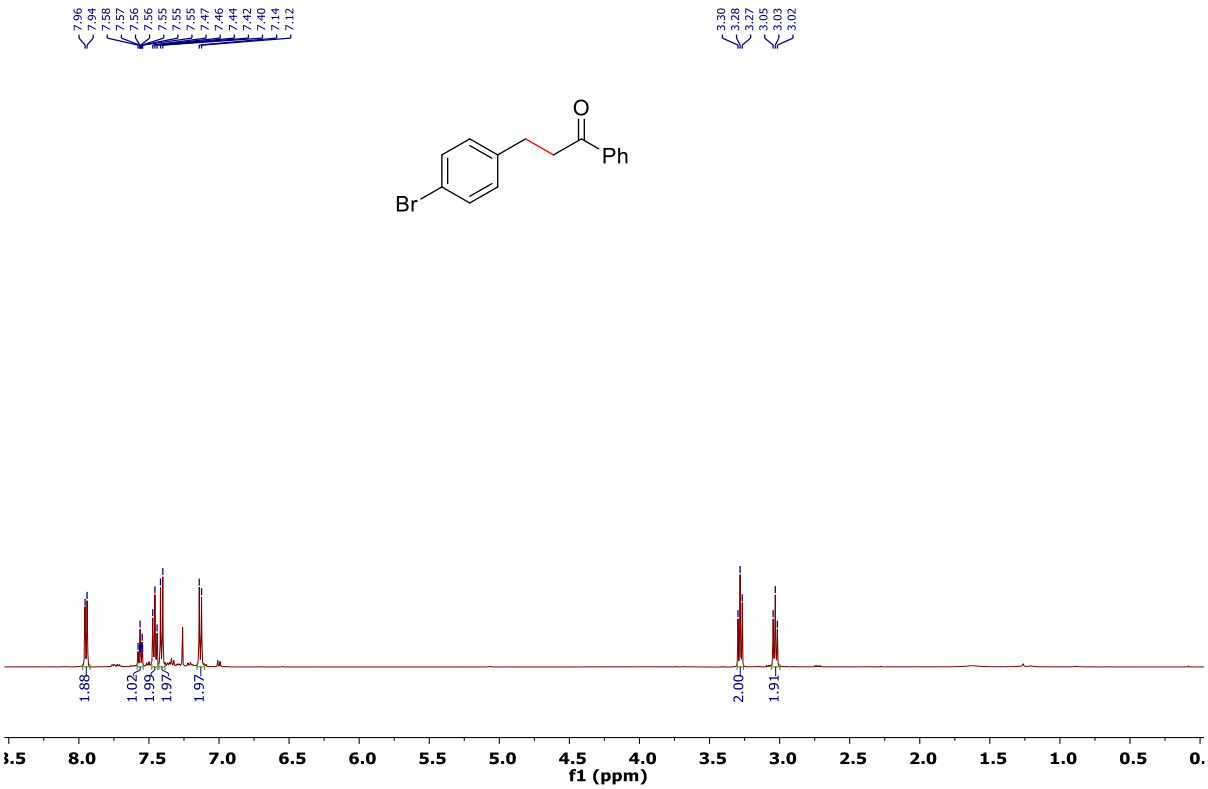
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.50 (d, *J* = 8.2 Hz, 1H), 7.38 (d, *J* = 4.3 Hz, 0H), 7.31 (q, *J* = 7.2, 6.6 Hz, 1H), 7.25 – 7.19 (m, 2H), 4.68 (dd, *J* = 7.9, 5.2 Hz, 0H), 2.72 (dddd, *J* = 29.8, 13.9, 9.4, 6.2 Hz, 1H), 2.18 – 1.96 (m, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 143.57, 141.49, 131.61, 128.48, 128.44, 127.68, 126.00, 121.38, 73.20, 40.48, 31.92.

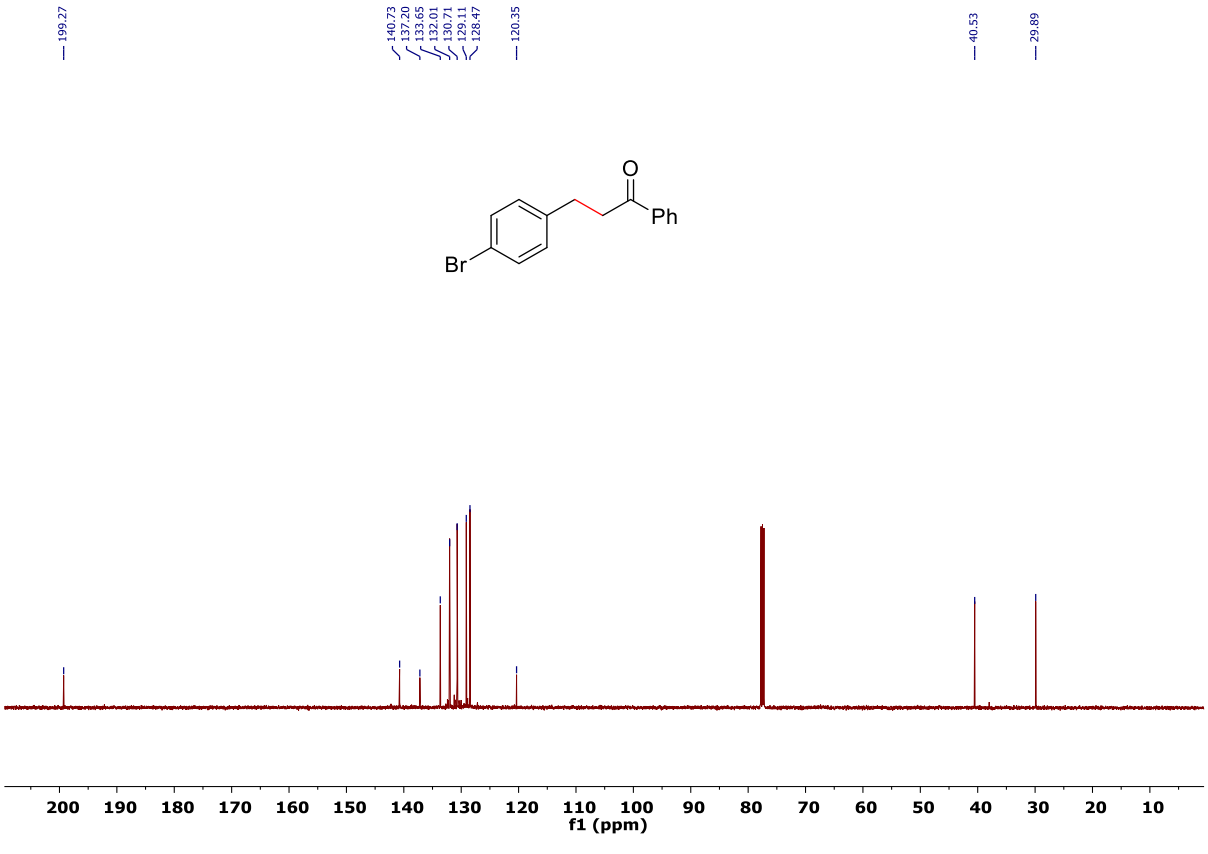
8n) 1-(4-bromophenyl)-3-(4-methoxyphenyl)propan-1-ol[24]

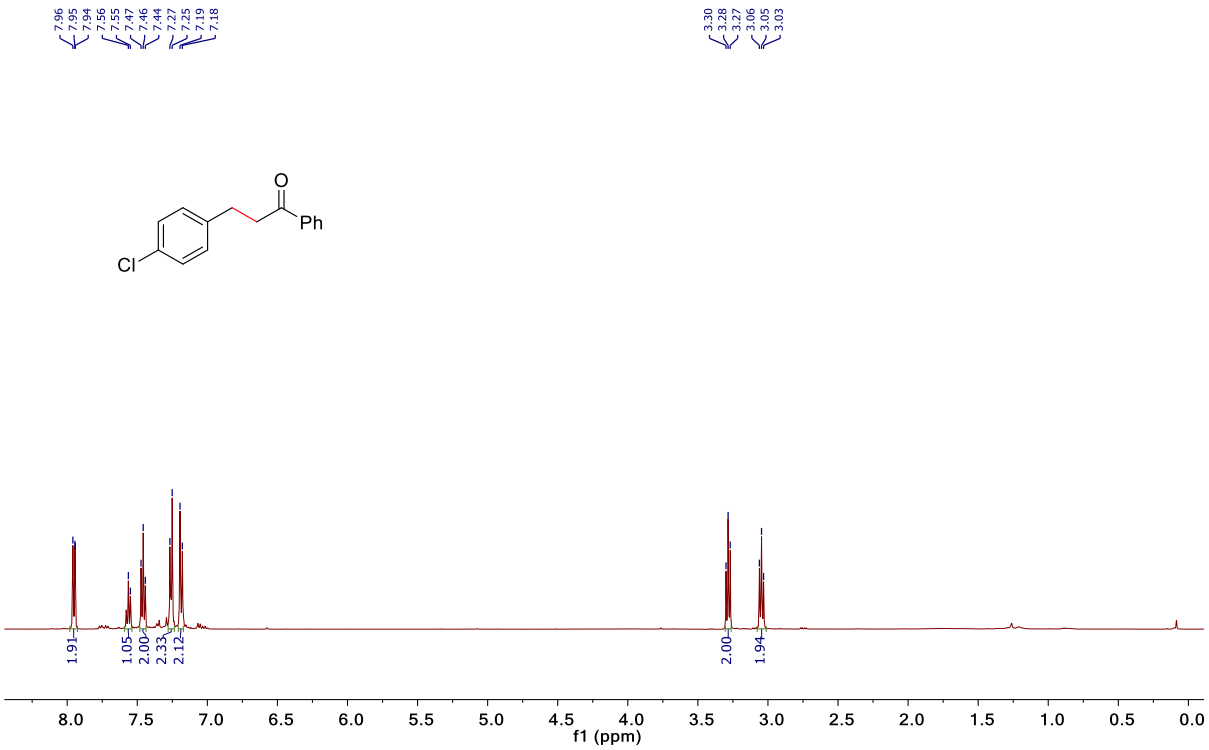


^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.49 (d, *J* = 8.3 Hz, 0H), 7.23 (d, *J* = 6.7 Hz, 1H), 7.11 (d, *J* = 8.7 Hz, 1H), 6.85 (d, *J* = 8.9 Hz, 1H), 4.65 (ddd, *J* = 7.8, 5.2, 2.1 Hz, 0H), 3.81 (s, 1H), 2.66 (qdd, *J* = 14.3, 9.0, 6.2 Hz, 1H), 2.14 – 1.89 (m, 1H). ^{13}C{^{1}H} NMR (126 MHz, Chloroform-*d*) δ 157.88, 143.64, 133.50, 131.59, 129.33, 127.69, 121.32, 113.90, 73.16, 55.29, 40.71, 31.00.

8. ^{1}H (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra for the isolated compounds:



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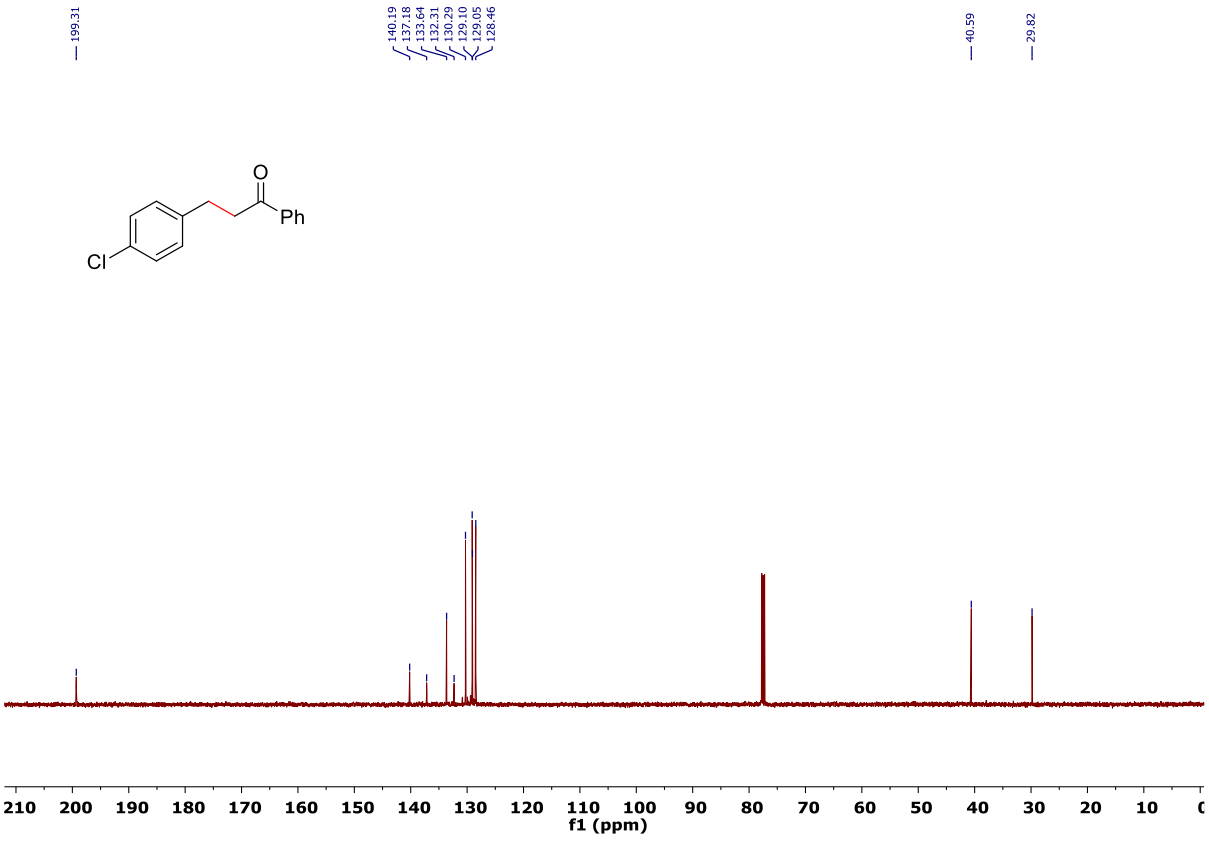
7.94
7.56
7.55
7.47
7.46
7.44
7.27
7.25
7.19
7.18
3.30
3.28
3.27
3.06
3.05
3.03
O
Ph
Cl
1.91
1.05
2.00
2.33
2.12
2.00
1.94
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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140.19
137.18
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130.29
129.10
129.05
128.46
40.59
29.82
O
Ph
Cl
210
200
190
180
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140
130
120
110
100
90
80
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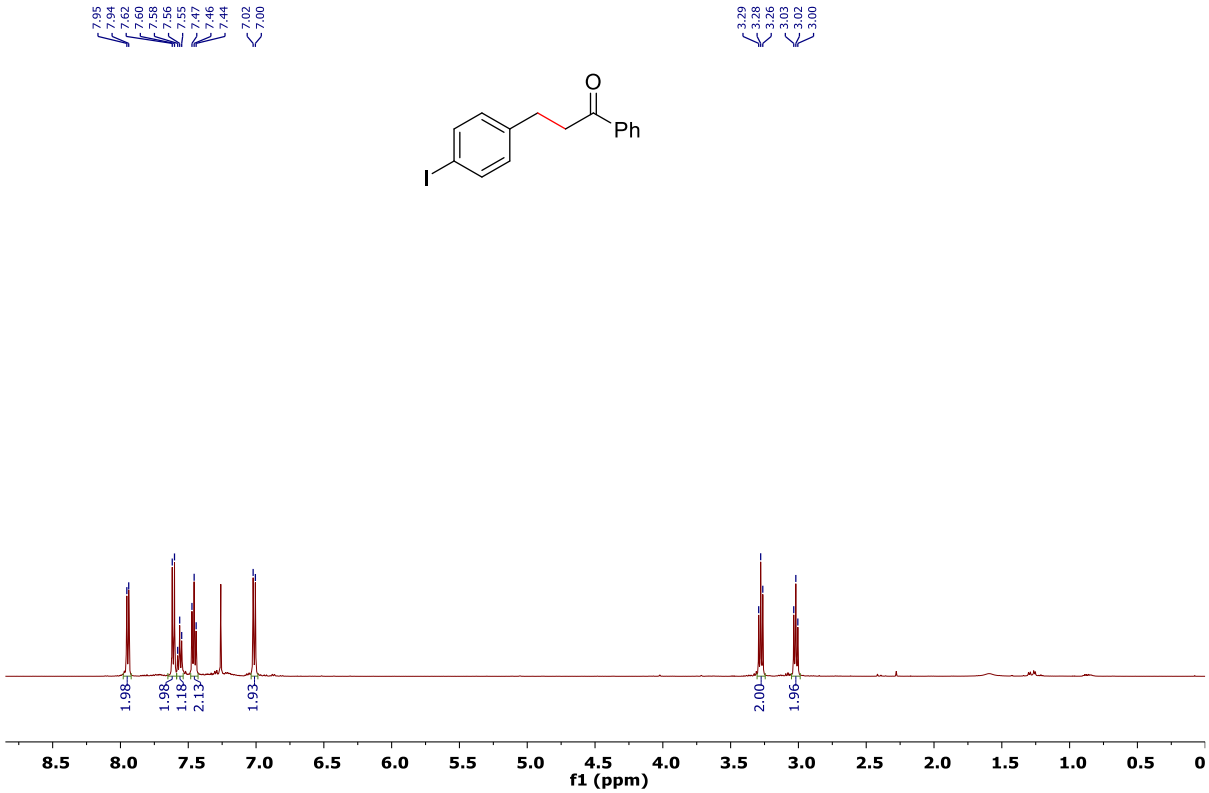
60
50
40
30
20
10
f1 (ppm)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7.56
7.55
7.47
7.46
7.44
7.02
7.00
3.29
3.28
3.26
3.03
3.02
3.00
O
Ph
I
1.98
1.98
1.18
2.13
1.93
2.00
1.96
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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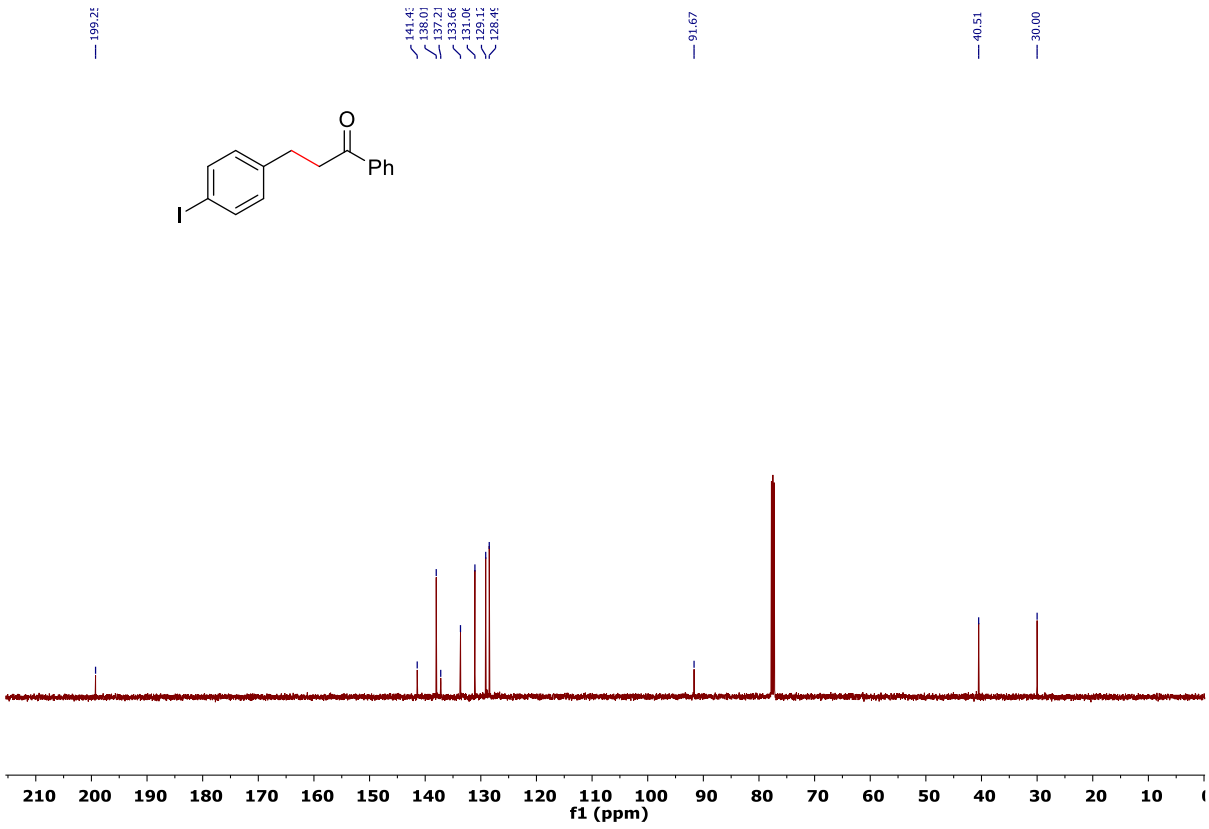
f1 (ppm)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137.2
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131.0
129.1
128.4
91.67
40.51
30.00
O
Ph
I
210
200
190
180
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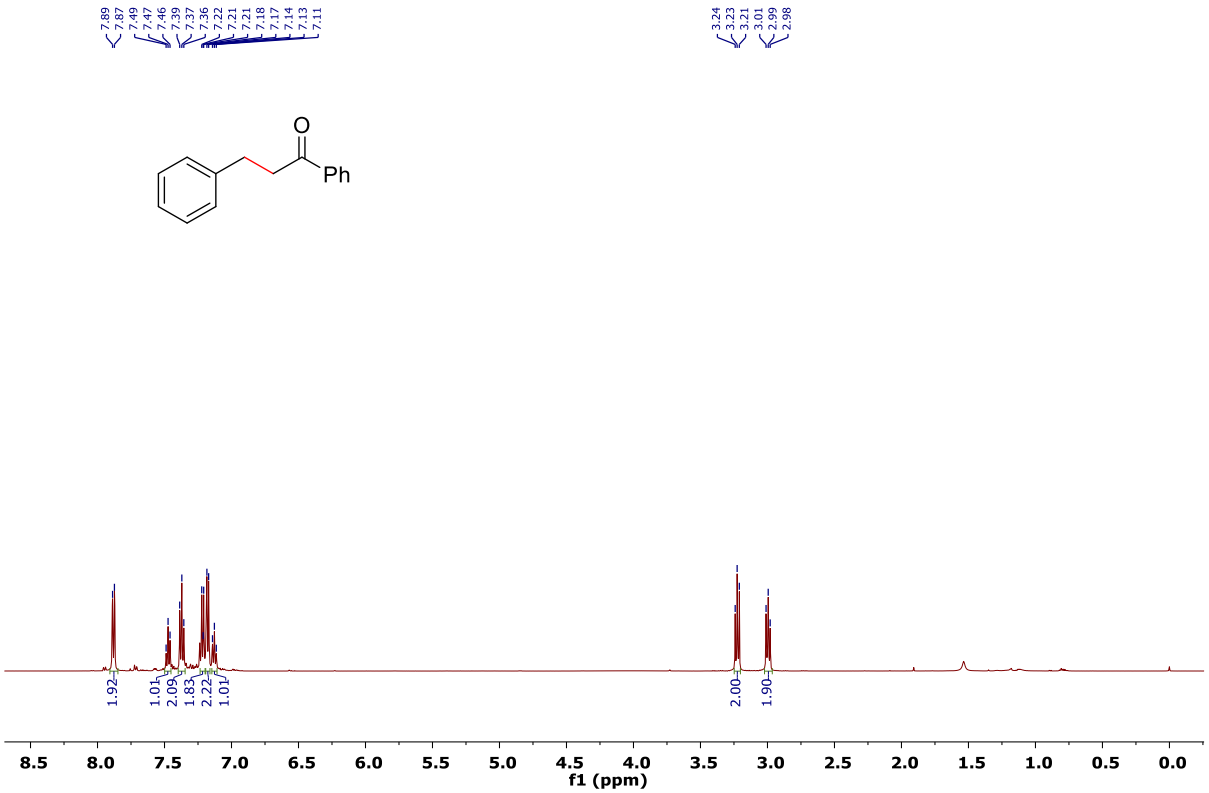
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.36
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7.21
7.21
7.18
7.17
7.14
7.13
7.11
3.24
3.23
3.21
3.01
2.99
2.98
O
Ph
1.92
1.01
2.09
1.83
2.22
1.01
2.00
1.90
8.5
8.0
7.5
7.0
6.5
6.0
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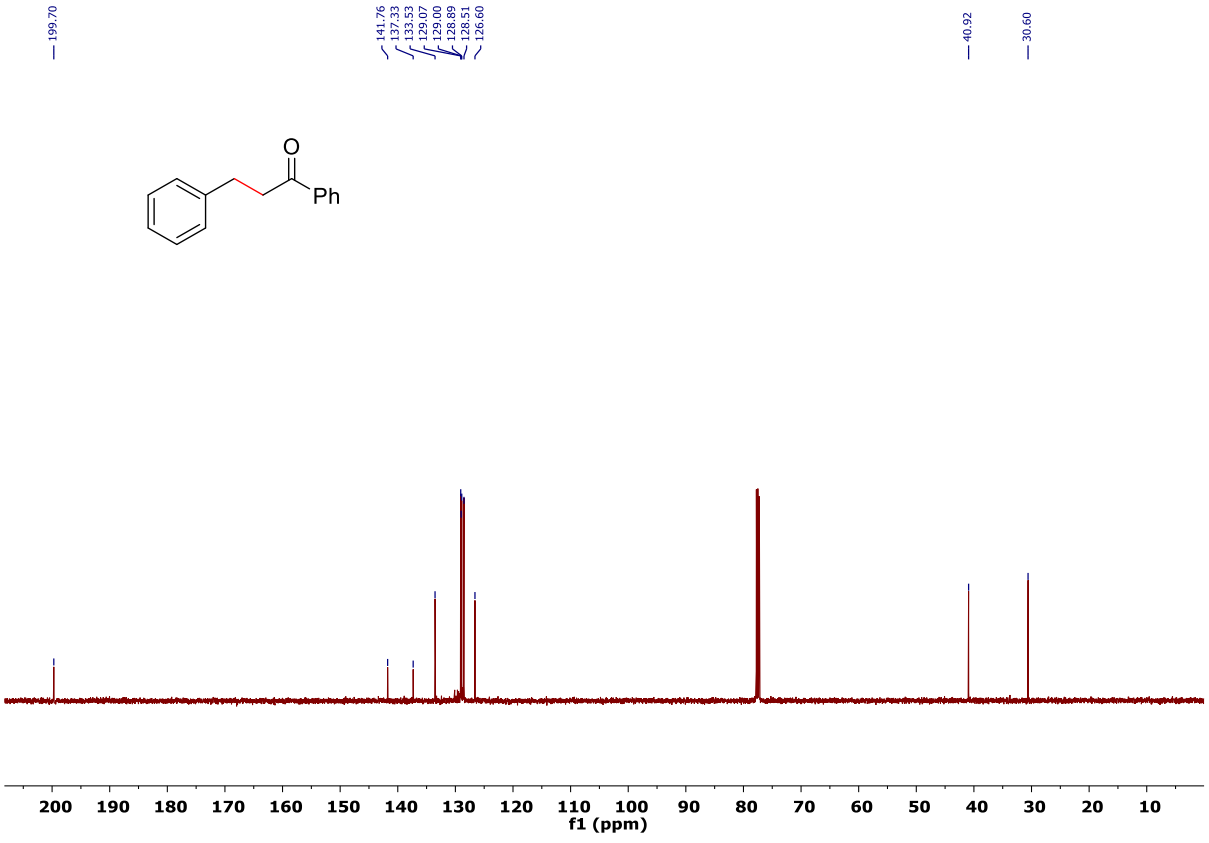
f1 (ppm)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137.33
133.53
129.07
129.00
128.89
128.51
126.60
40.92
30.60
O
Ph
200
190
180
170
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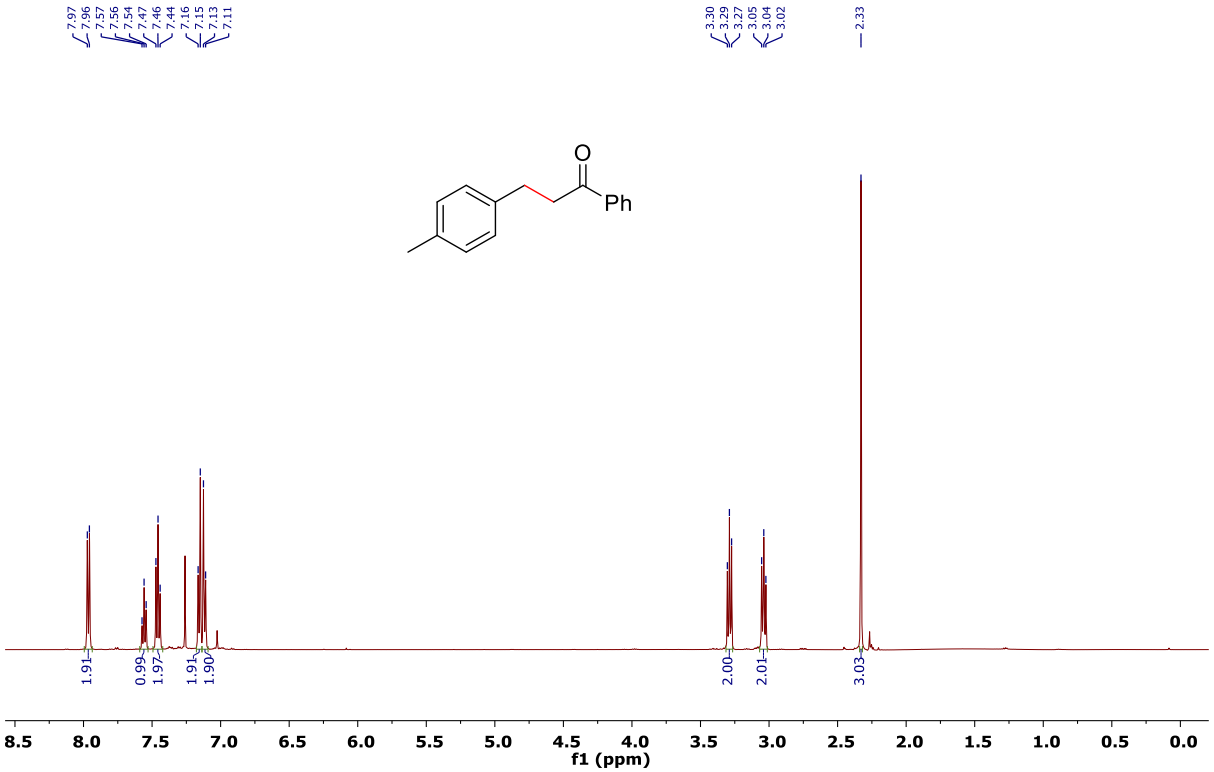
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.44
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7.13
7.11
3.30
3.29
3.27
3.05
3.04
3.02
2.33
O
Ph
1.91
0.99
1.97
1.91
1.90
2.00
2.01
3.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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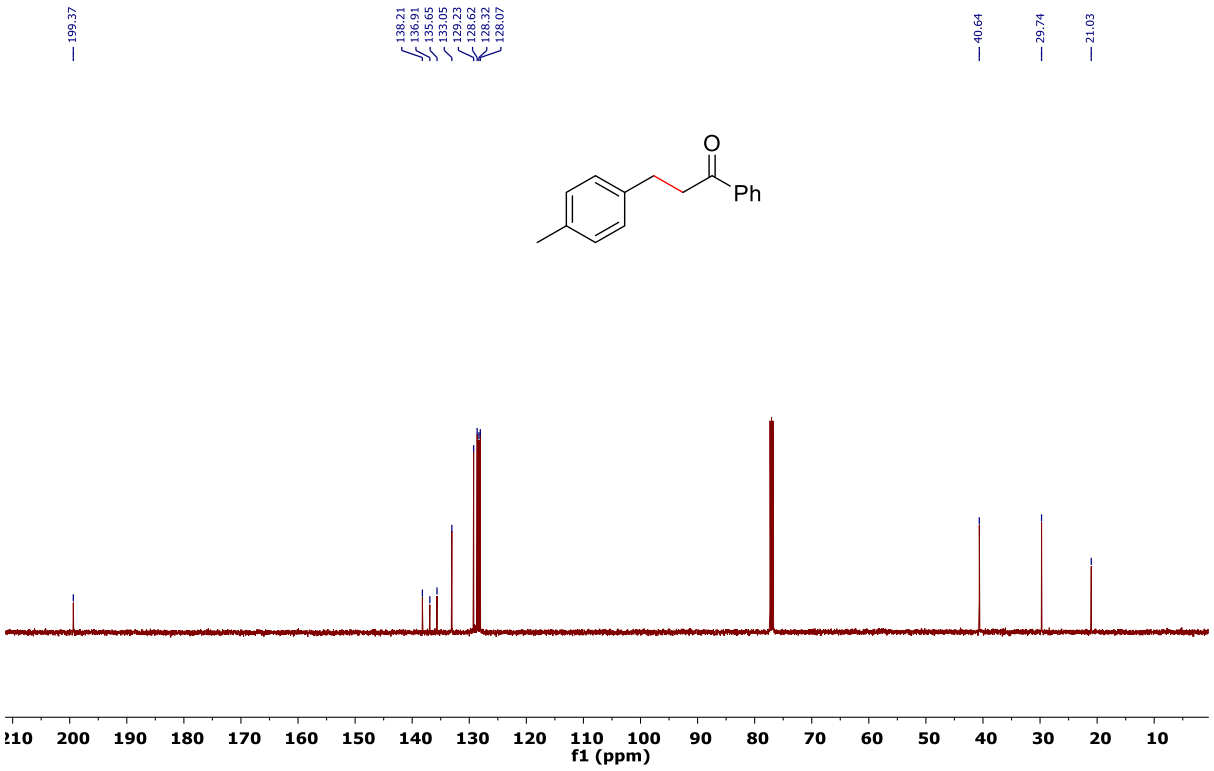
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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129.23
128.62
128.32
128.07
40.64
29.74
21.03
O
Ph
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200
190
180
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140
130
120
110
100
90
80
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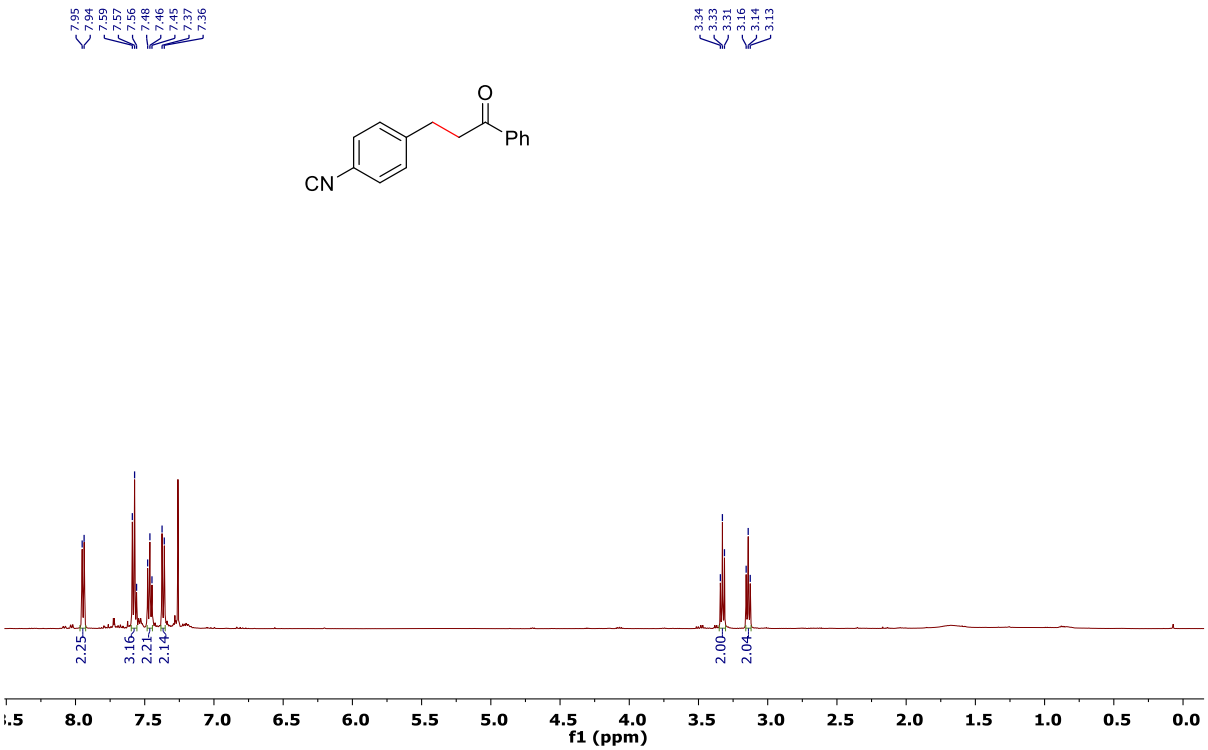
60
50
40
30
20
10
f1 (ppm)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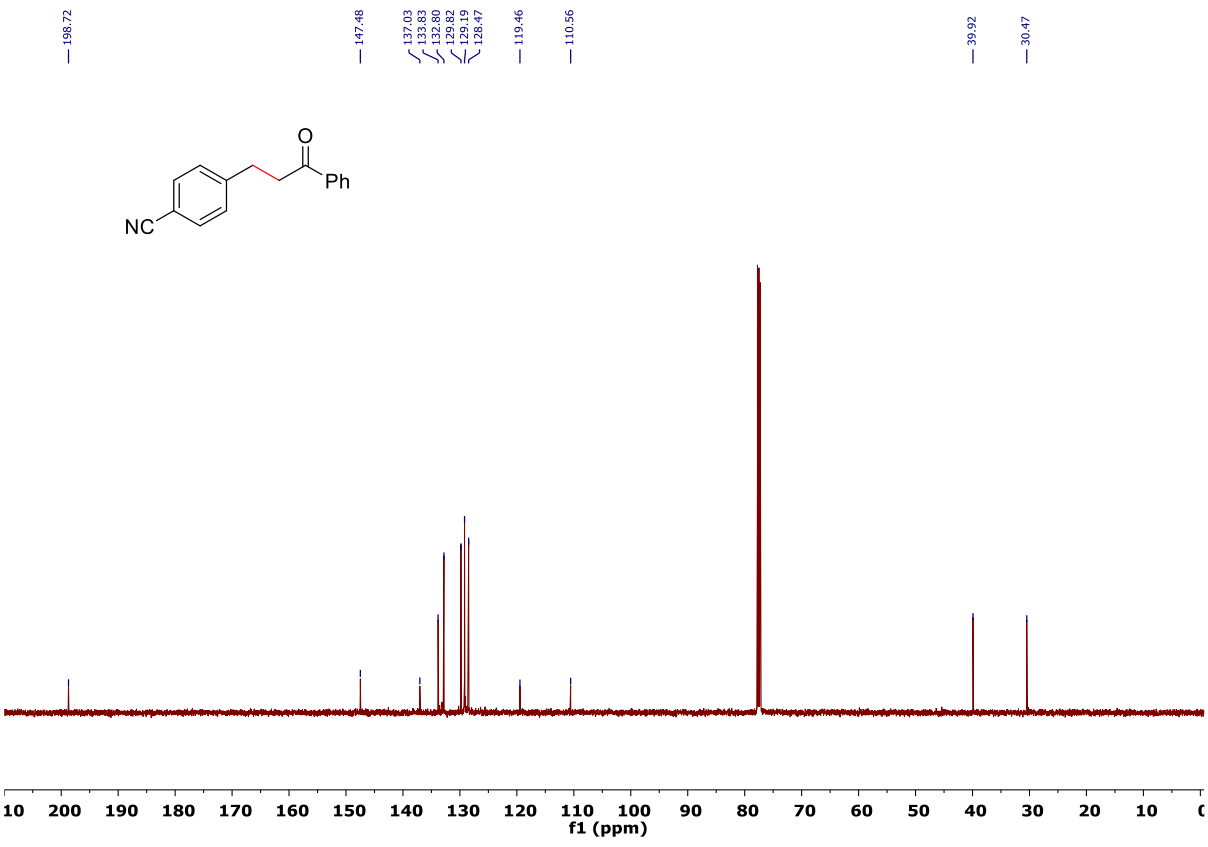
3.33
3.31
3.16
3.14
3.13
O
Ph
CN
2.25
3.16
2.21
2.14
2.00
2.04
f1 (ppm)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39.92
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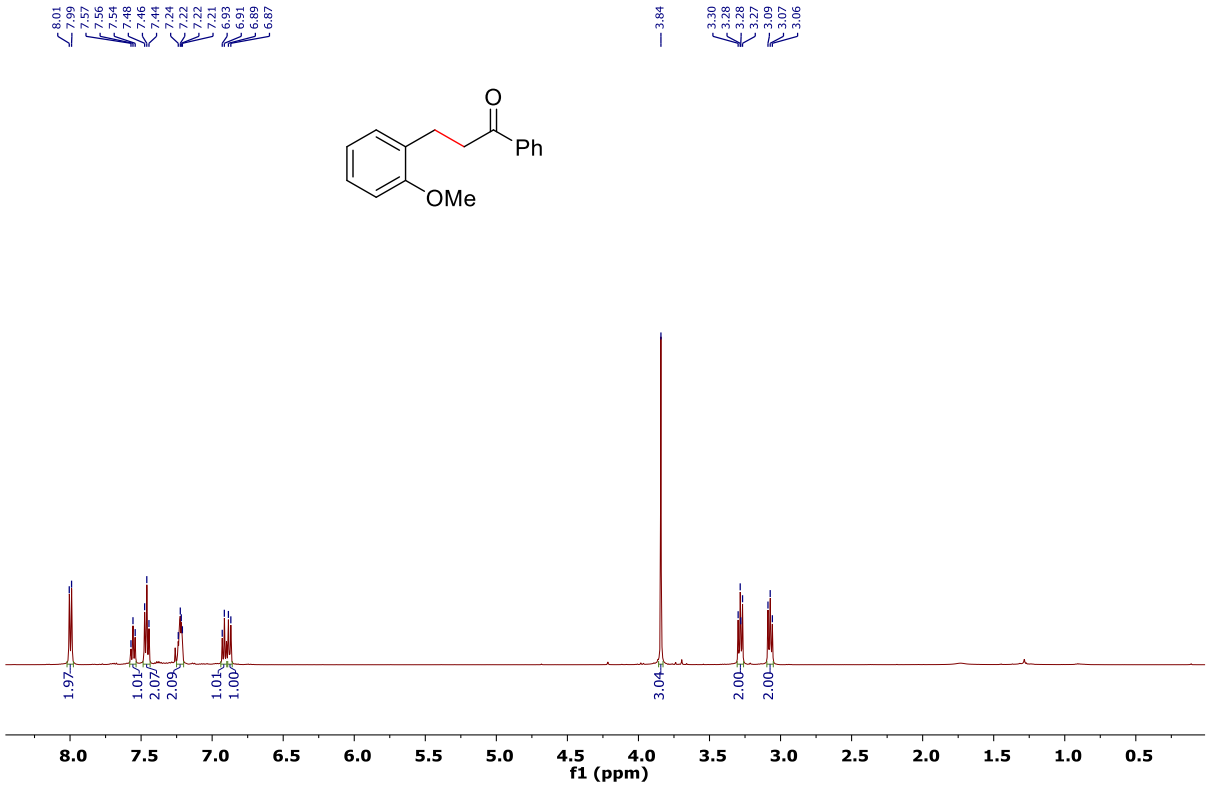
Ph
NC
f1 (ppm)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6.89
6.87
3.84
3.30
3.28
3.28
3.27
3.09
3.07
3.06
O
Ph
OMe
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1.01
2.07
2.09
1.01
1.00
3.04
2.00
2.00
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7.0
6.5
6.0
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2.5
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1.5
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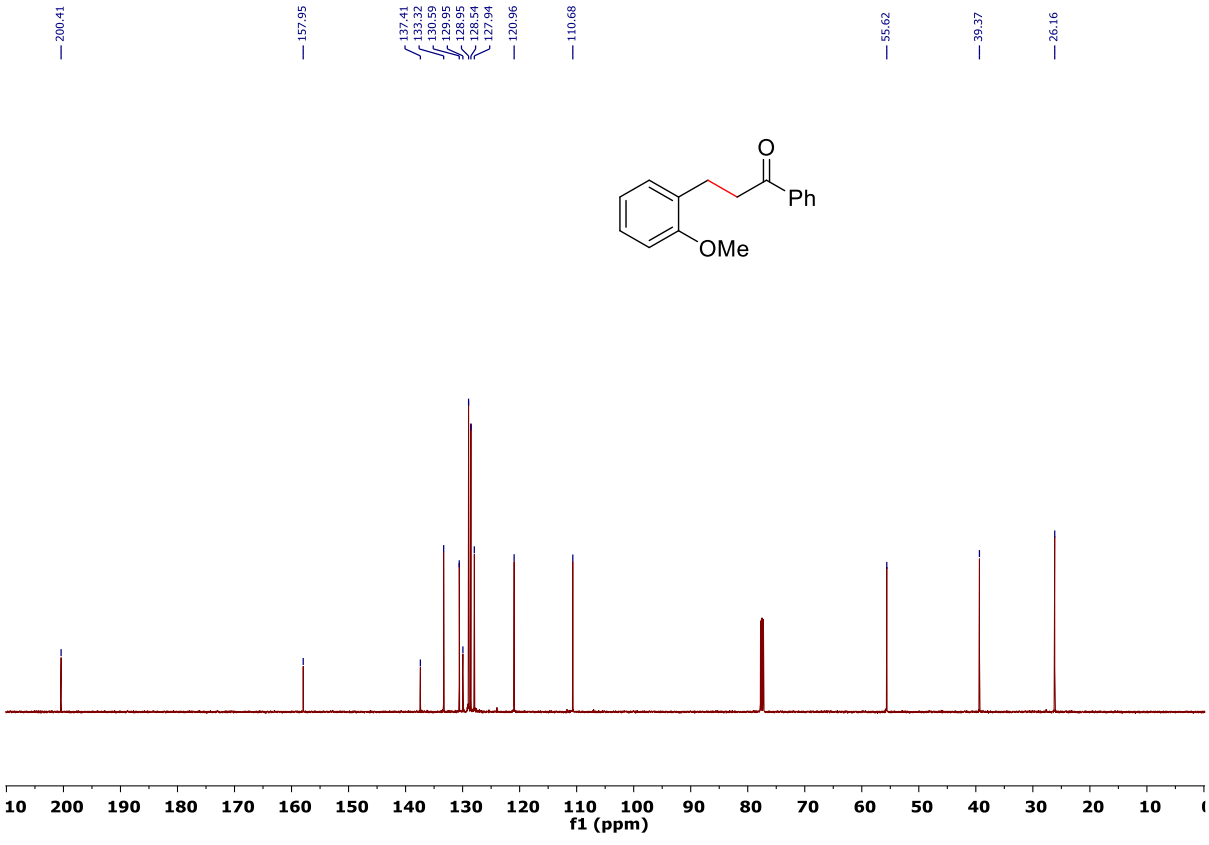
0.5
f1 (ppm)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128.54
127.94
120.96
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55.62
39.37
26.16
O
Ph
OMe
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.91
7.89
7.69
7.48
7.46
7.39
7.38
7.36
7.25
7.25
7.23
7.23
7.16
7.16
7.15
7.15
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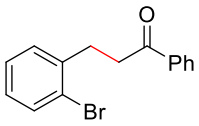
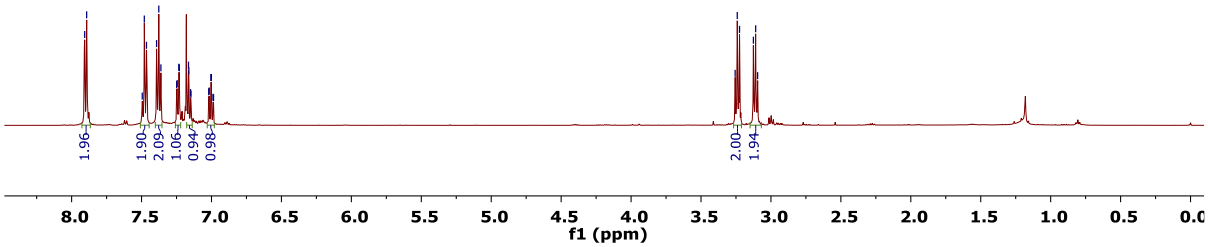
7.00
6.99
6.99


3.26
3.24
3.22
3.13
3.11
3.10


O
Ph
Br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1.06
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0.98
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1.94
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


199.41
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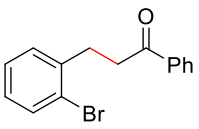
31.28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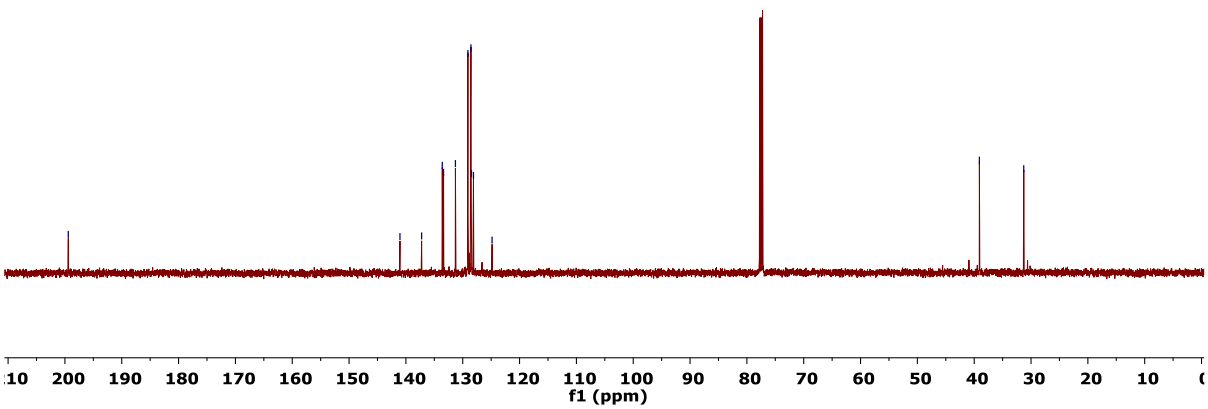
Ph
Br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120
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100
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80
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50
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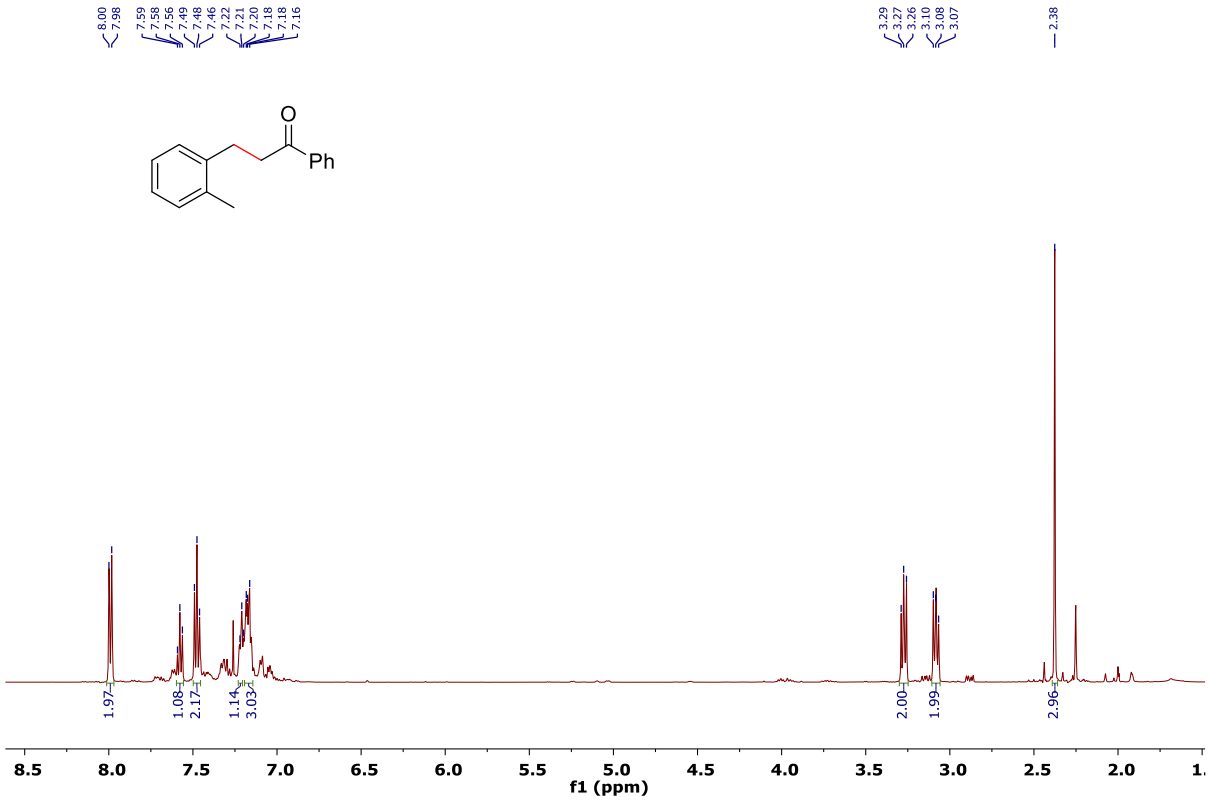
f1 (ppm)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7.18
7.16
3.29
3.27
3.26
3.10
3.08
3.07
2.38
O
Ph
1.97
1.08
2.17
1.14
3.03
2.00
1.99
2.96
8.5
8.0
7.5
7.0
6.5
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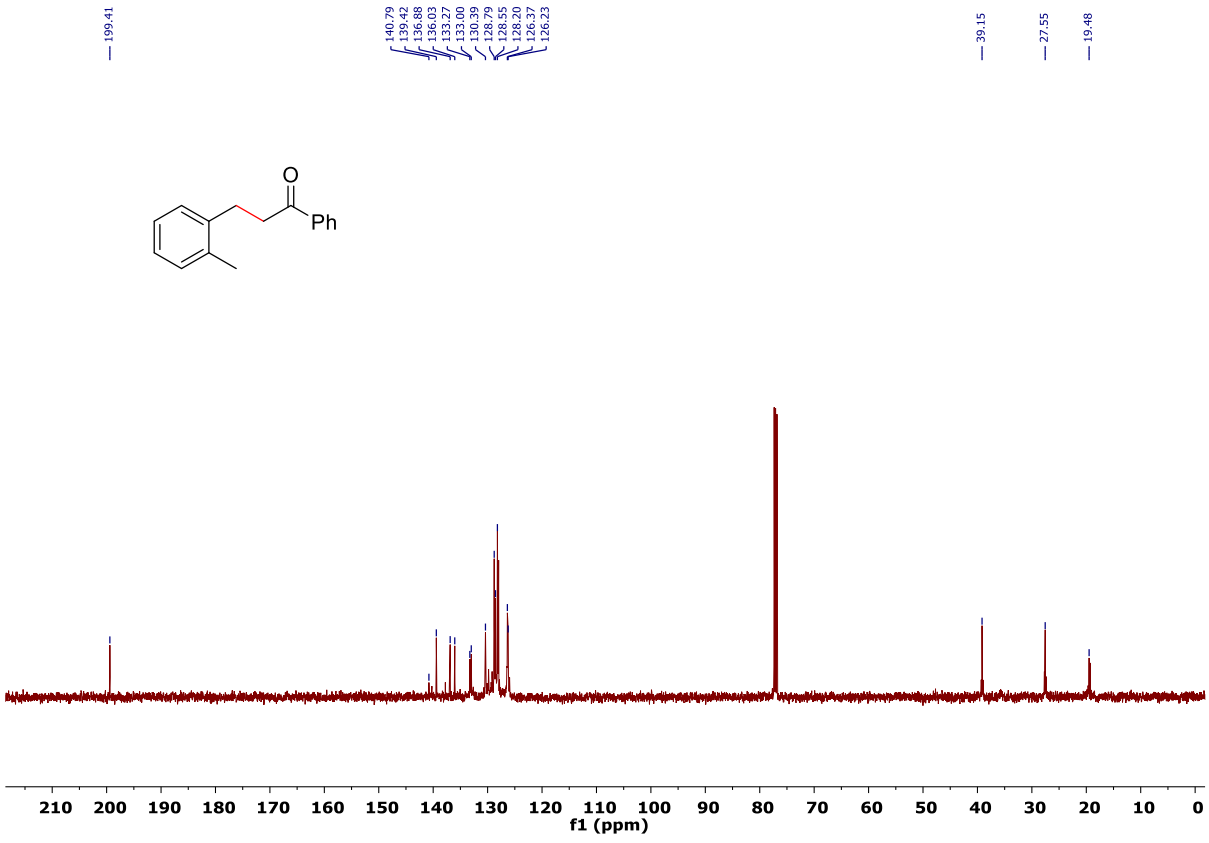
4.0
3.5
3.0
2.5
2.0
f1 (ppm)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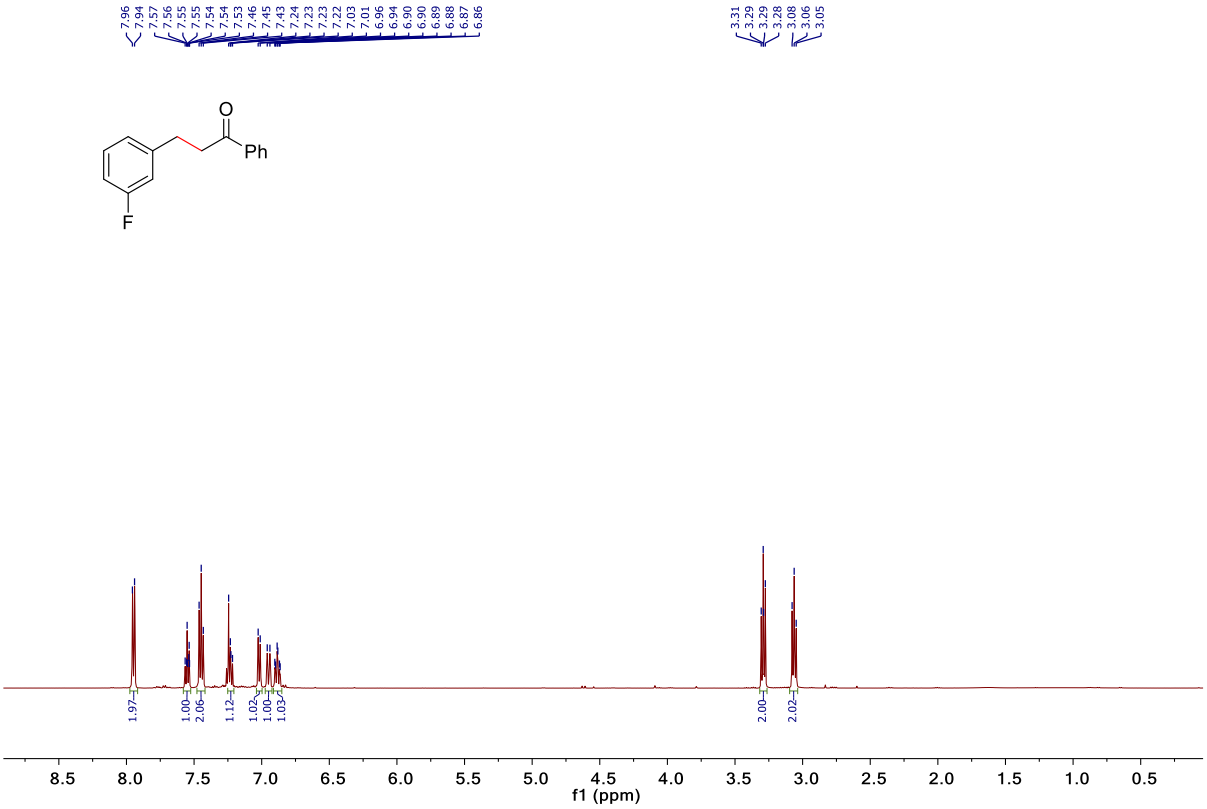
126.23
39.15
27.55
19.48
O
Ph
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.24
7.23
7.23
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7.03
7.01
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6.86
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3.08
3.06
3.05
O
Ph
F
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1.02
1.00
1.03
2.00
2.02
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3.0
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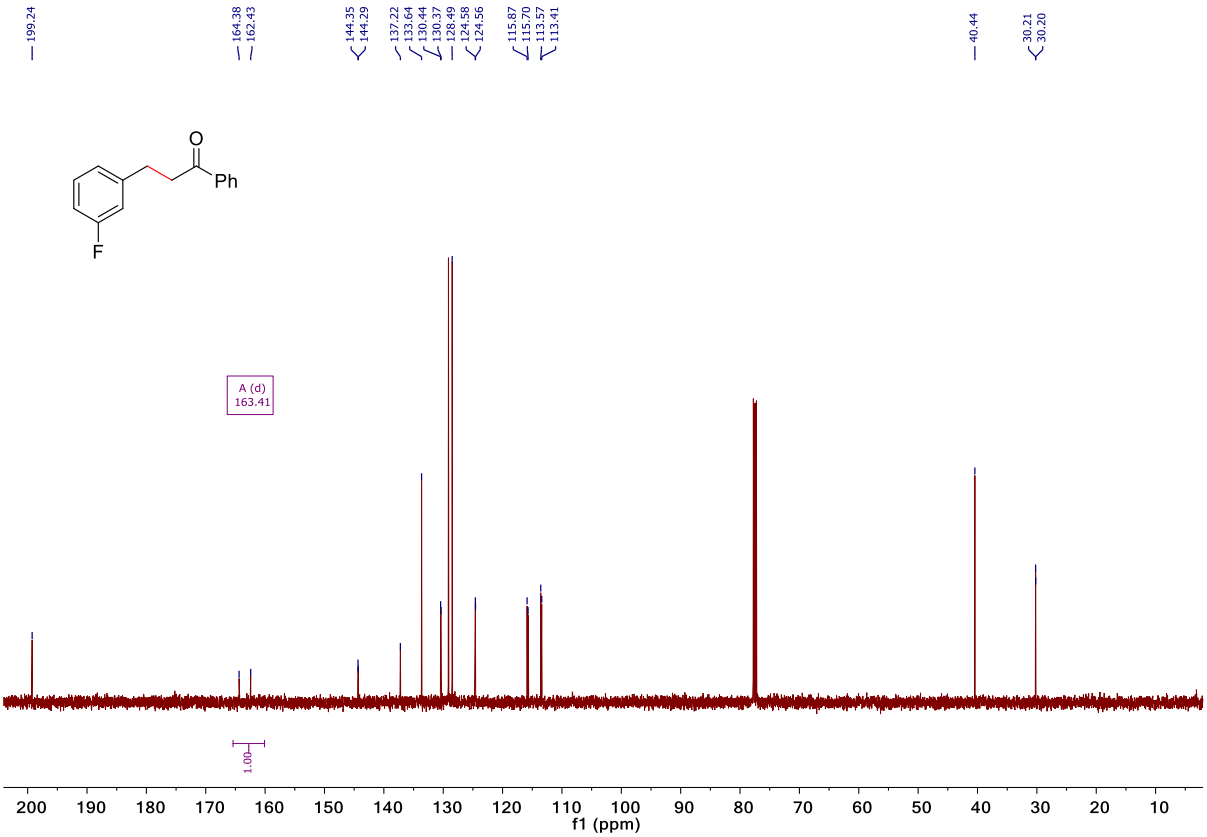
f1 (ppm)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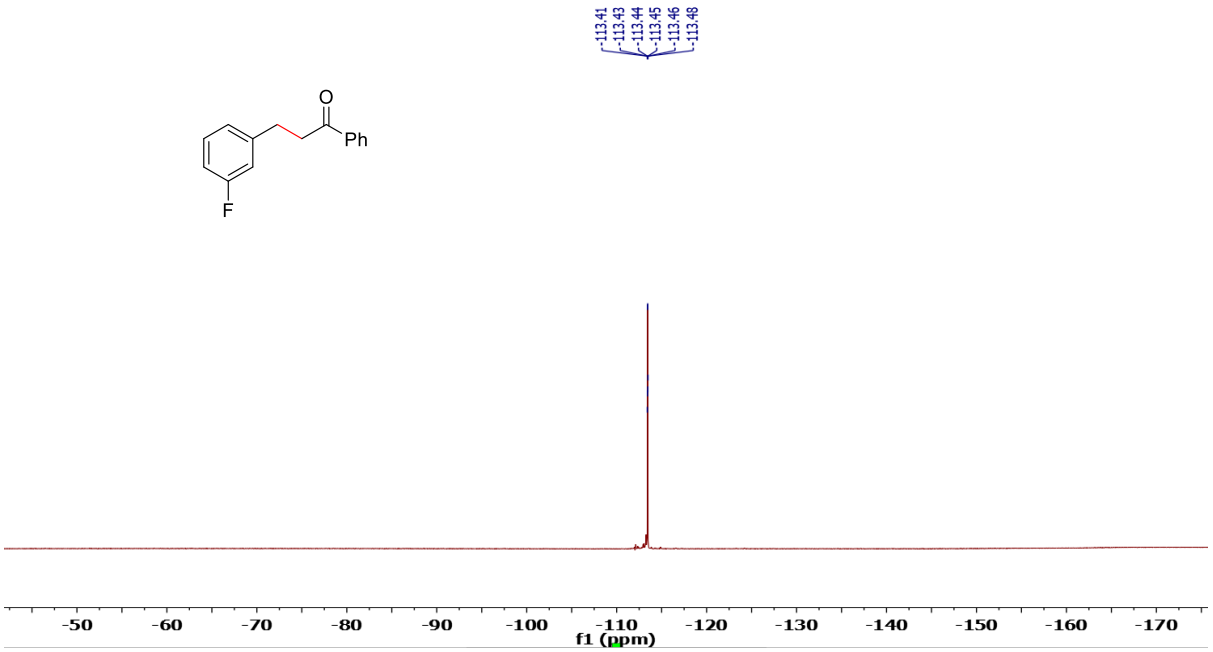
115.70
113.57
113.41
40.44
30.21
30.20
O
Ph
F
A (d)
163.41
1.00
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

^{19}F NMR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O
Ph
F
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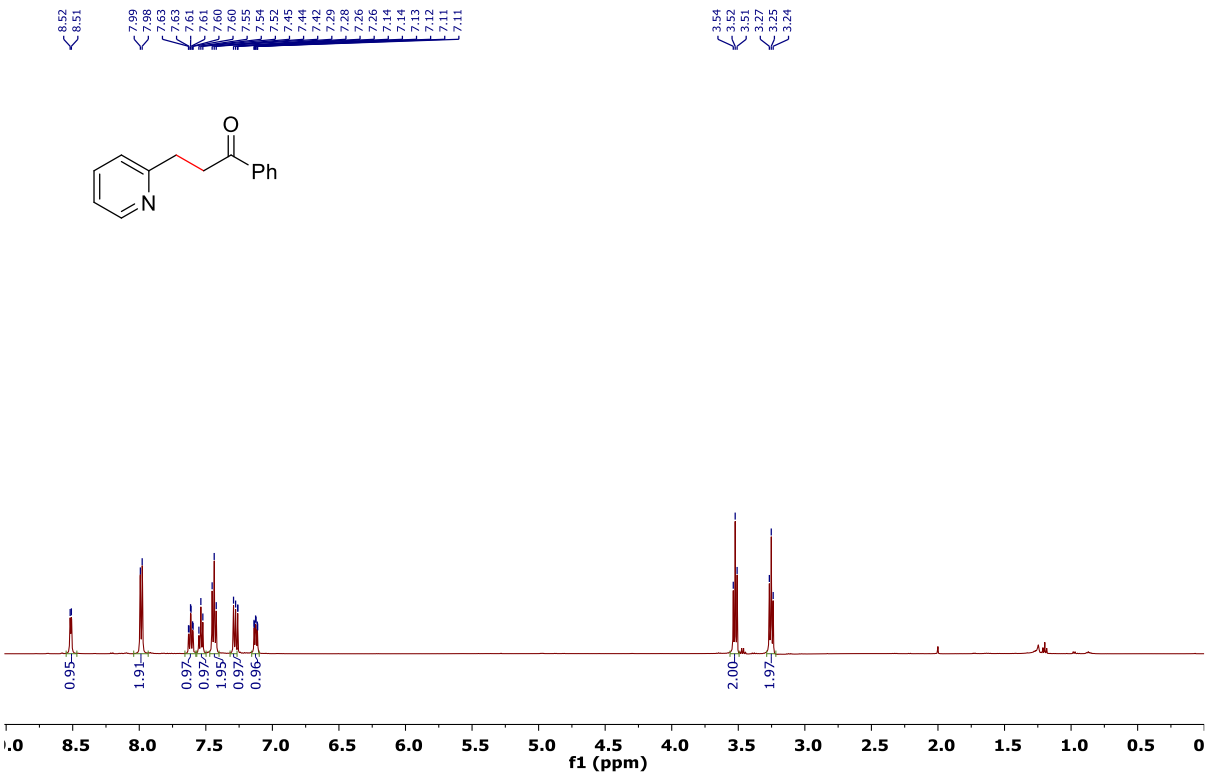
-130
-140
-150
-160
-170
f1 (ppm)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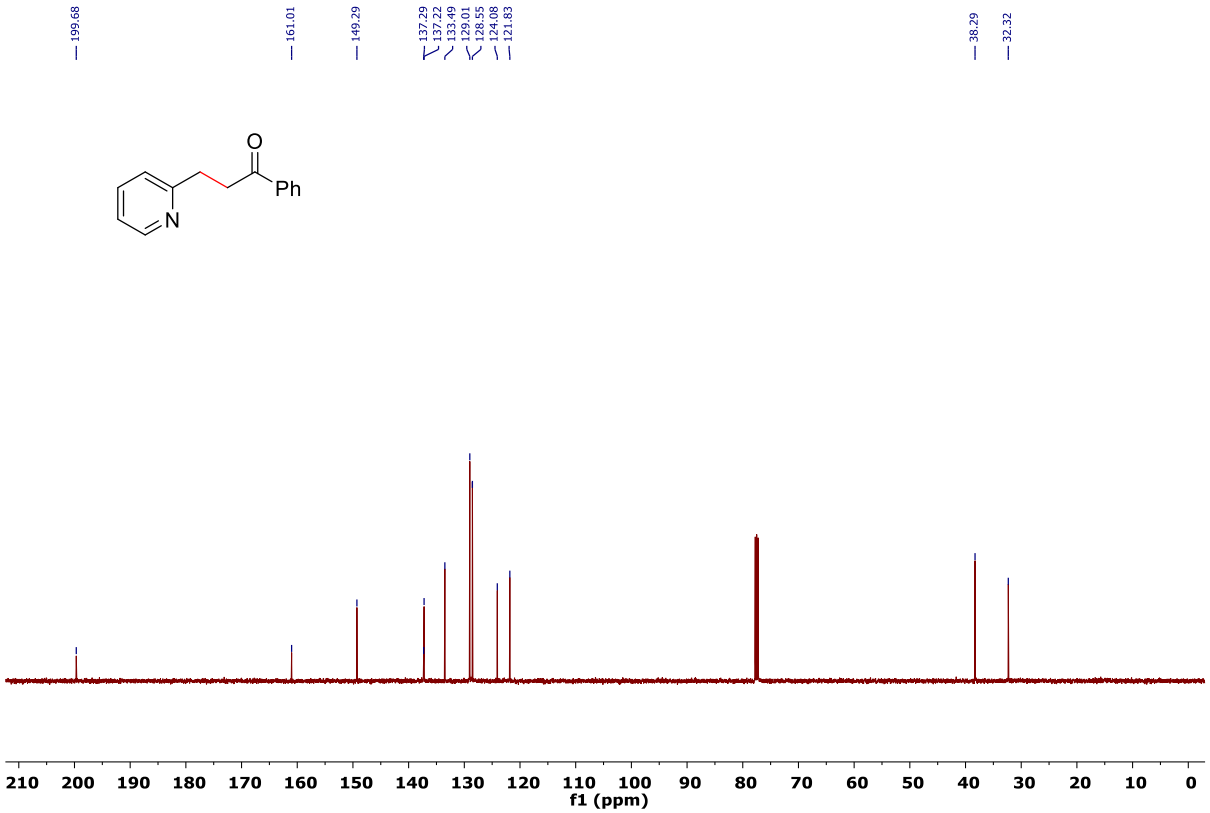
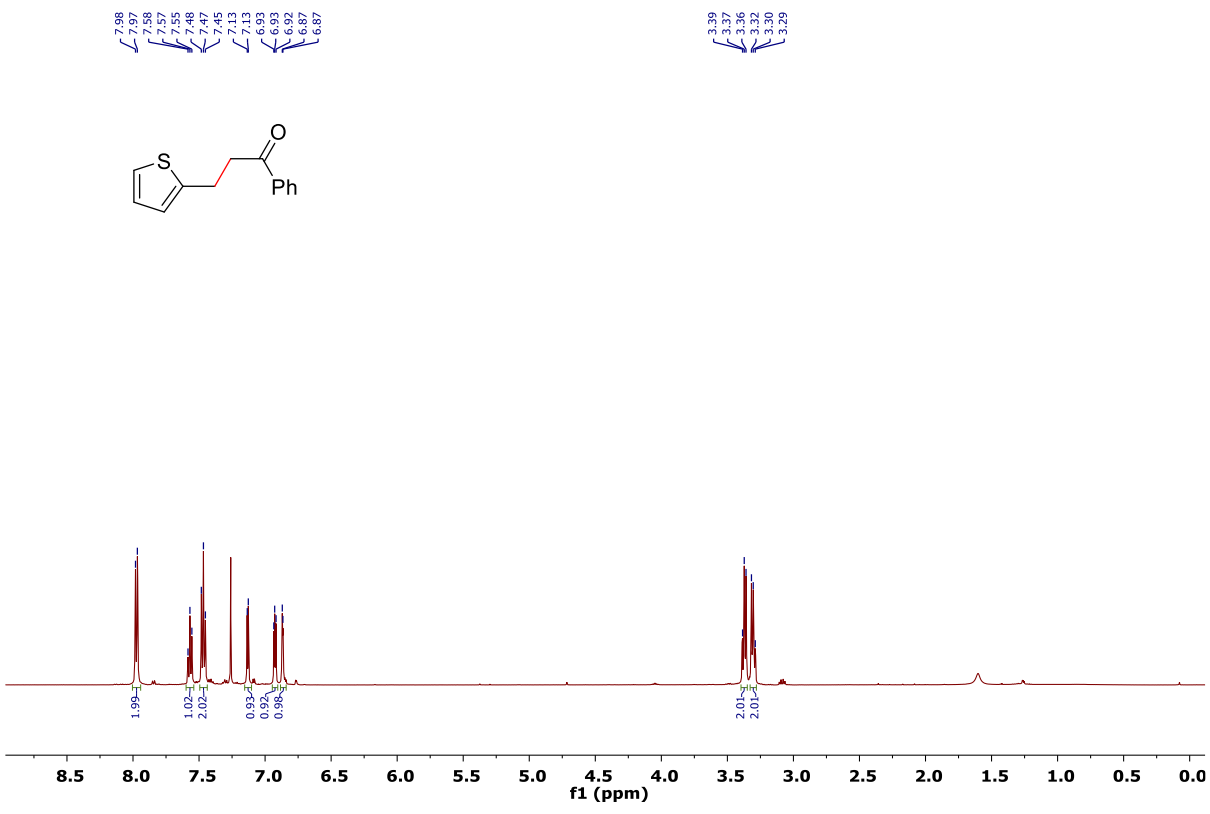
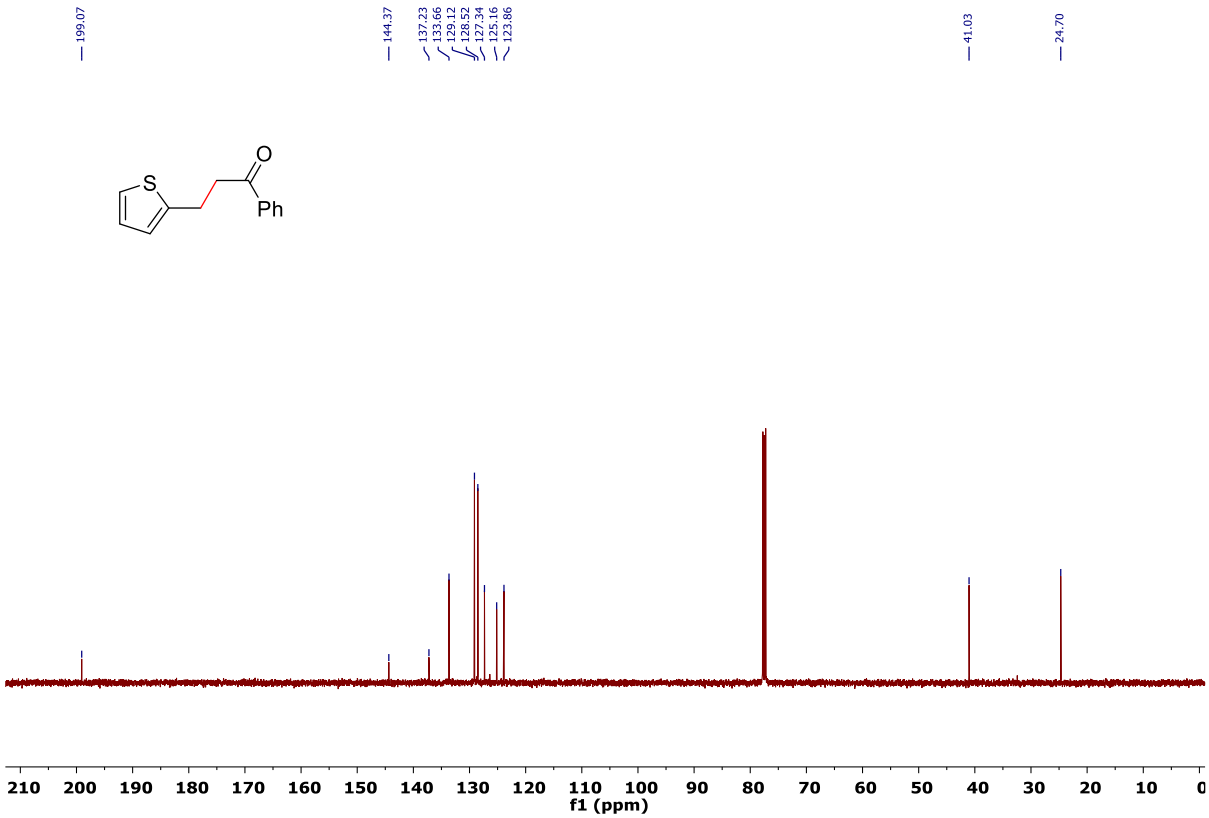
7.60
7.60
7.55
7.54
7.52
7.45
7.44
7.42
7.29
7.28
7.26
7.14
7.14
7.13
7.12
7.11
7.11
3.54
3.52
3.51
3.27
3.25
3.24
O
Ph
N
0.95
1.91
0.97
0.97
1.95
0.97
0.96
2.00
1.97
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)


199.68
161.01
149.29
137.29
137.22
133.49
129.01
128.55
124.08
121.83
38.29
32.32
O
Ph
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.98
7.97
7.58
7.57
7.55
7.48
7.47
7.45
7.13
6.93
6.93
6.92
6.87
6.87
3.39
3.37
3.36
3.32
3.30
3.29
O
S
Ph
1.99
1.02
2.02
0.93
0.92
0.98
2.01
2.01
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
f1 (ppm)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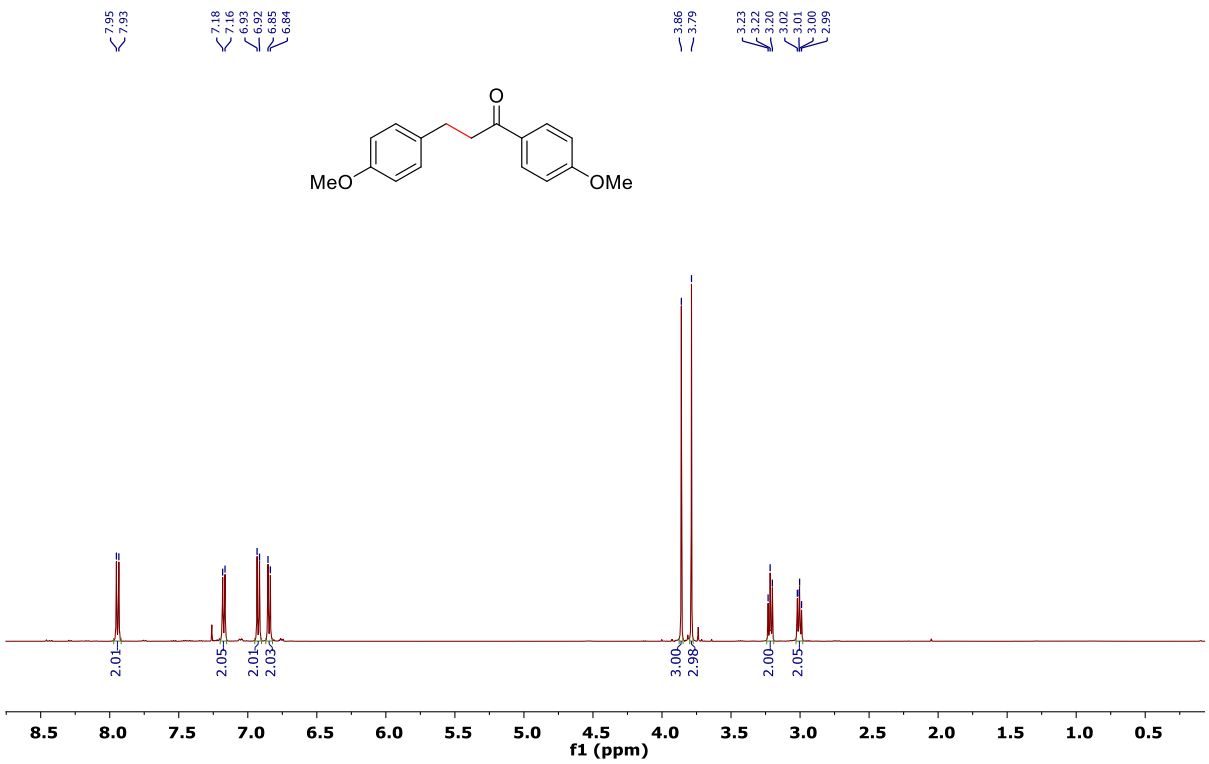
144.37
137.23
133.66
129.12
128.52
127.34
125.16
123.86
41.03
24.70
O
S
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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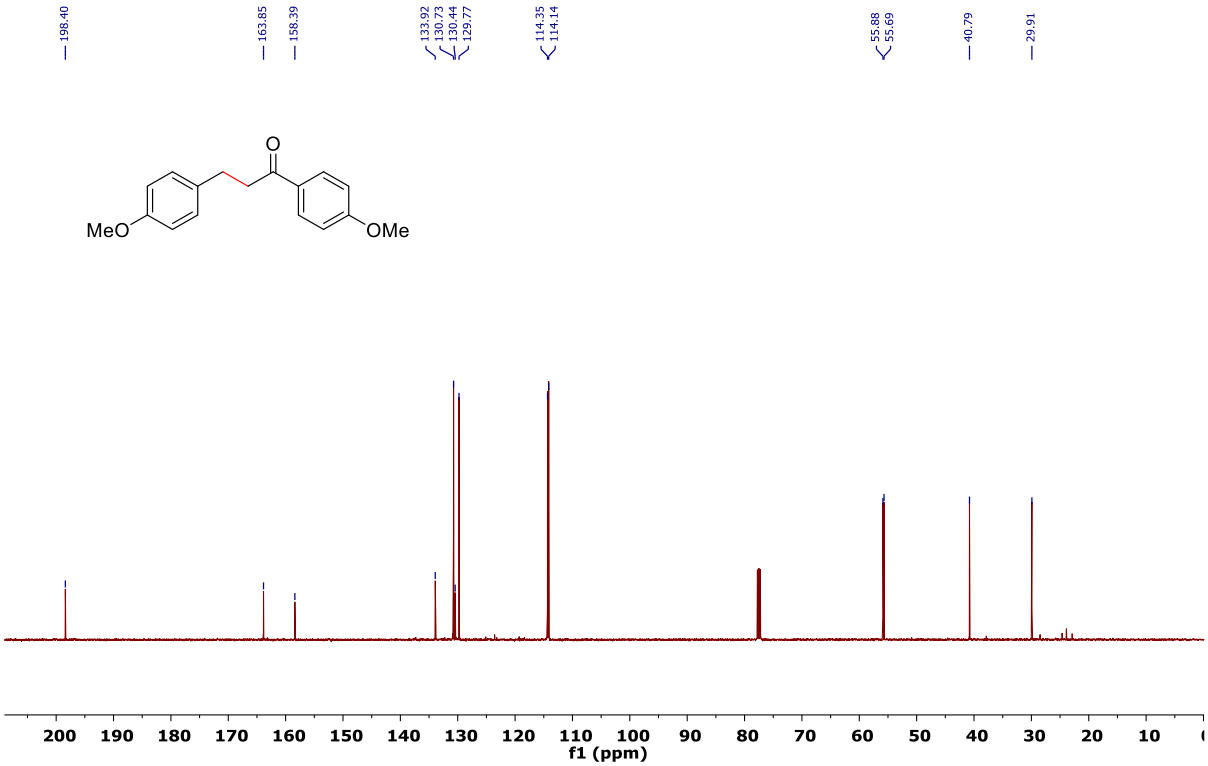
7.93
7.18
7.16
6.93
6.92
6.85
6.84
3.86
3.79
3.23
3.22
3.20
3.02
3.01
3.00
2.99
O
MeO
OMe
2.01
2.05
2.01
2.03
3.00
2.98
2.00
2.05
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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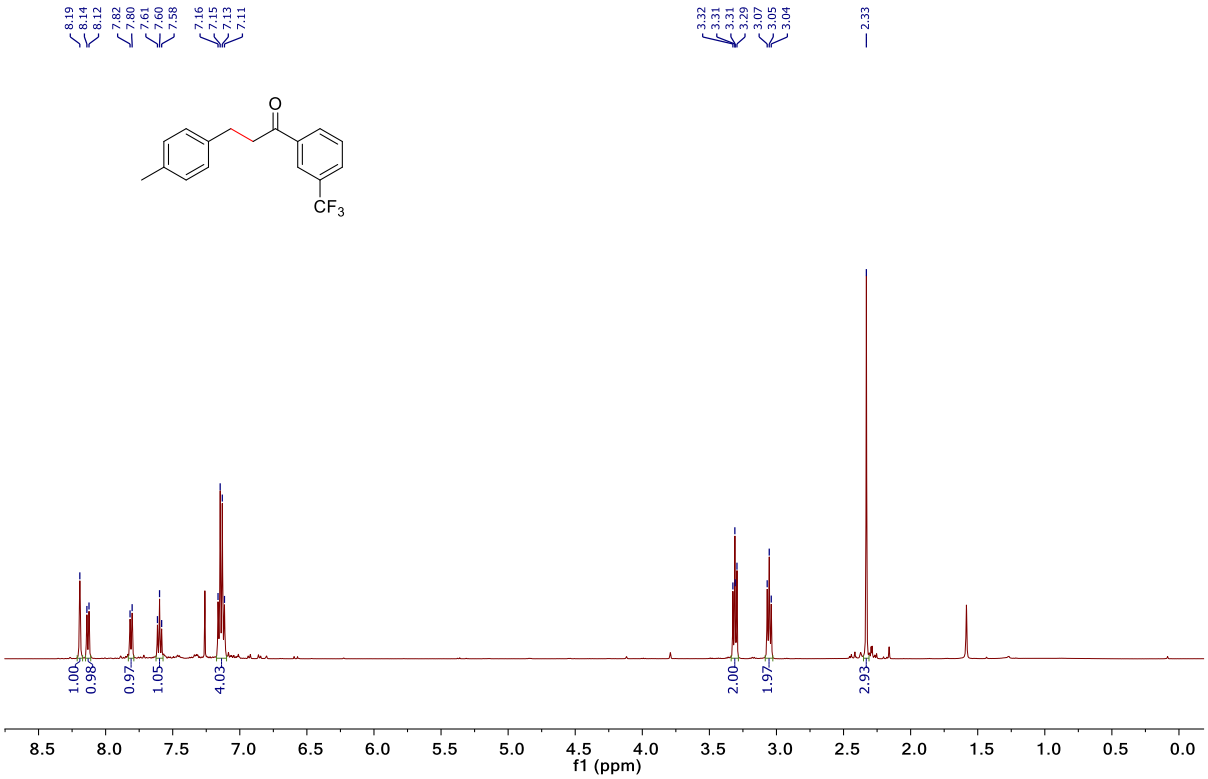
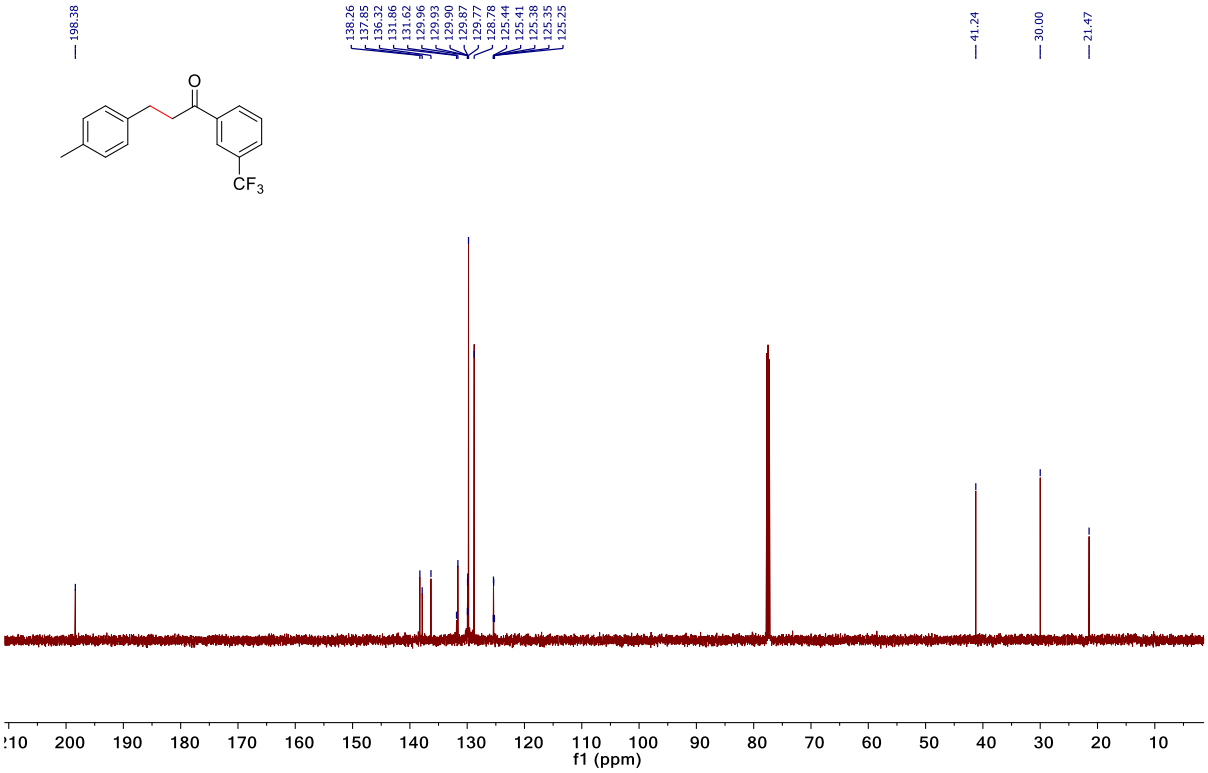
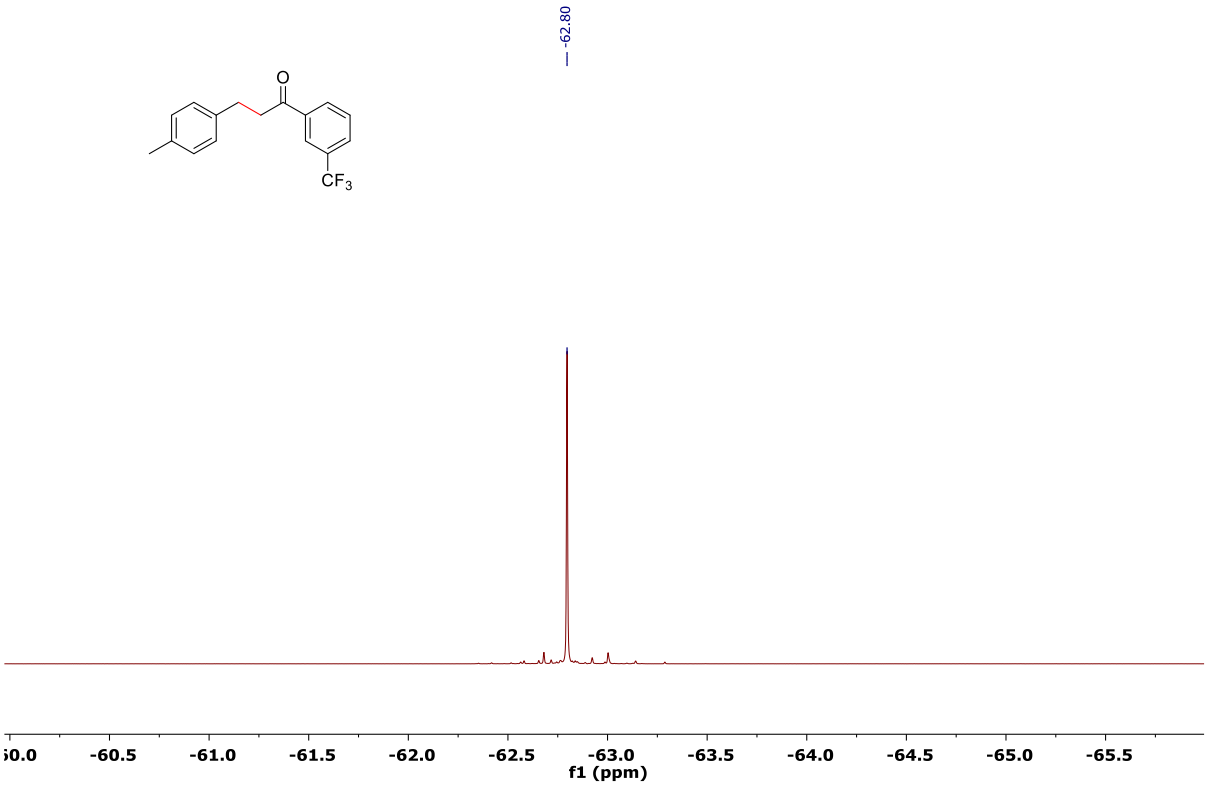
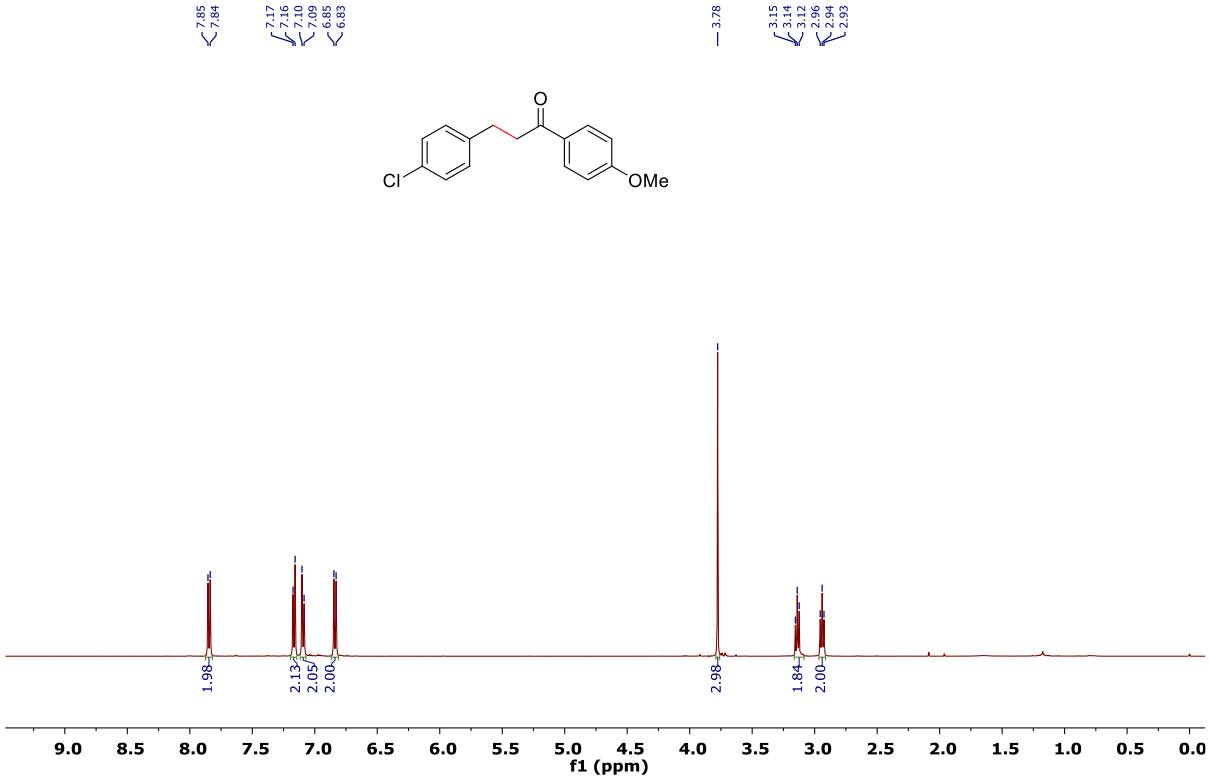
163.85
158.39
133.92
130.73
130.44
129.77
114.35
114.14
55.88
55.69
40.79
29.91
O
MeO
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

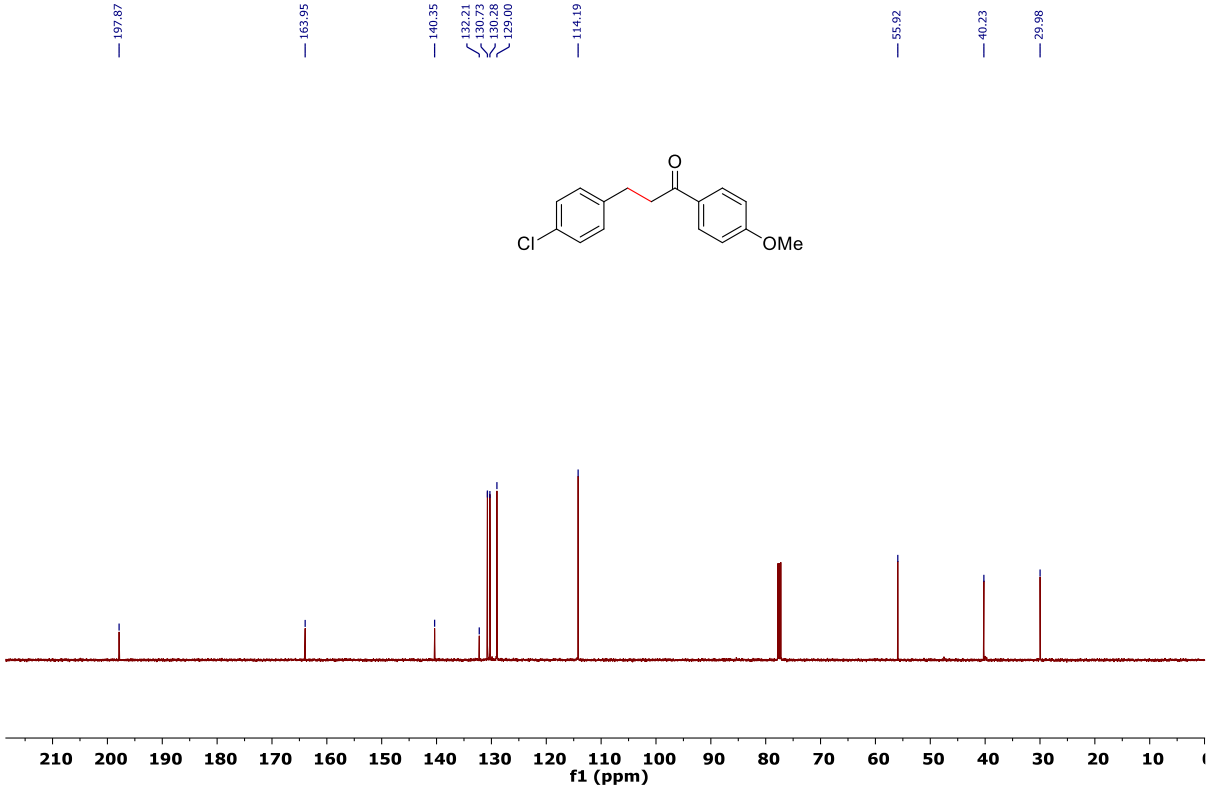

8.19
8.14
8.12
7.82
7.80
7.61
7.60
7.58
7.16
7.15
7.13
7.11
3.32
3.31
3.31
3.29
3.07
3.05
3.04
2.33
O
CF3
1.00
0.98
0.97
1.05
4.03
2.00
1.97
2.93
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

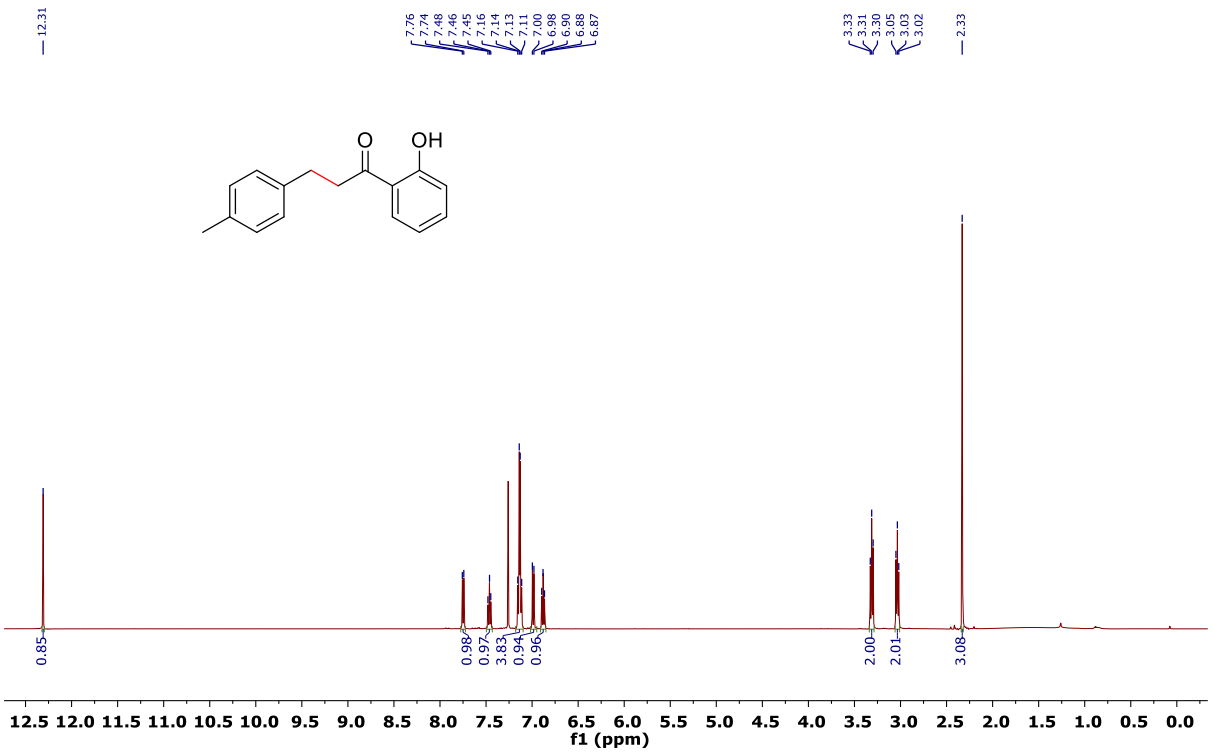

198.38
138.26
137.85
136.32
131.86
131.62
129.96
129.93
129.90
129.87
129.77
128.78
125.44
125.41
125.38
125.35
125.25
41.24
30.00
21.47
O
CF3
f1 (ppm)

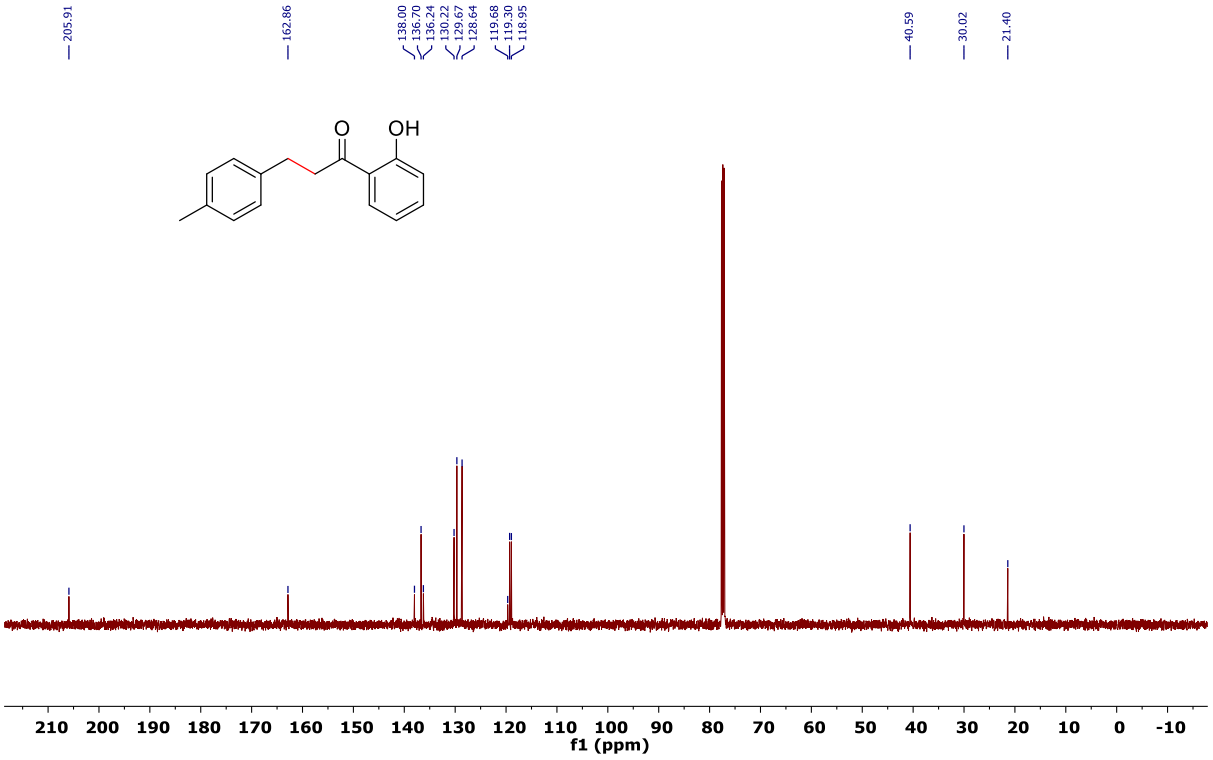
^{19}F NMR

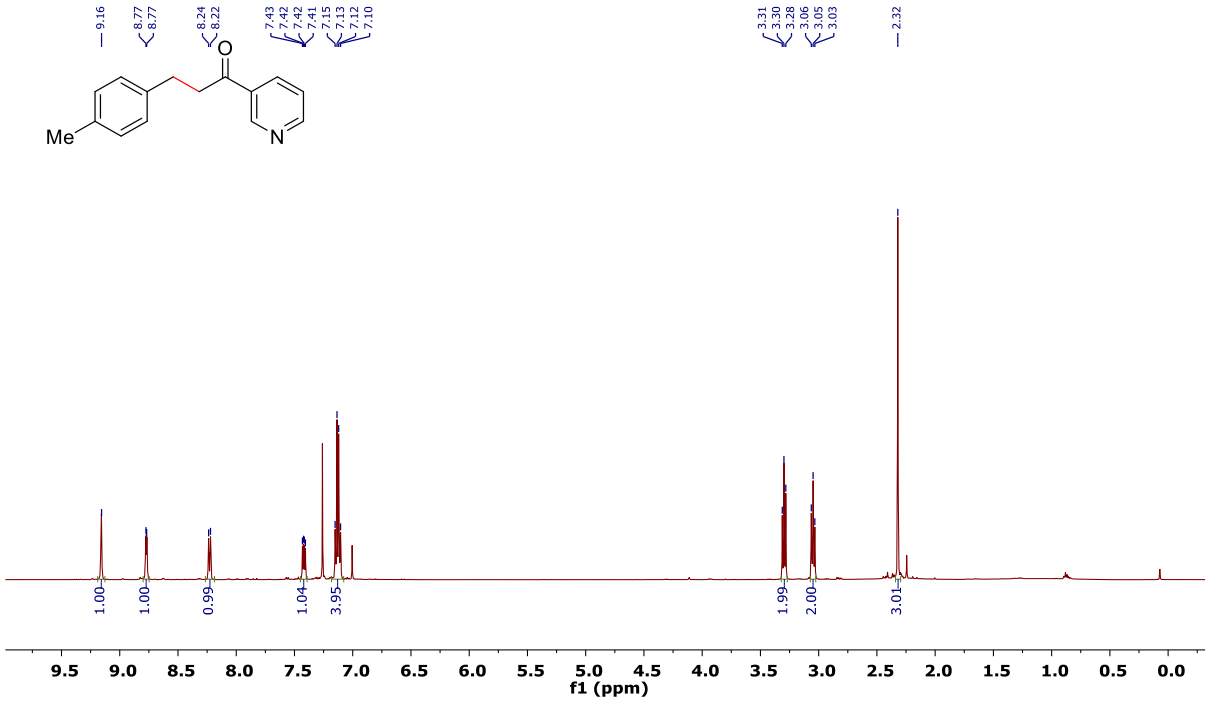

-62.80
O
CF3
f1 (ppm)

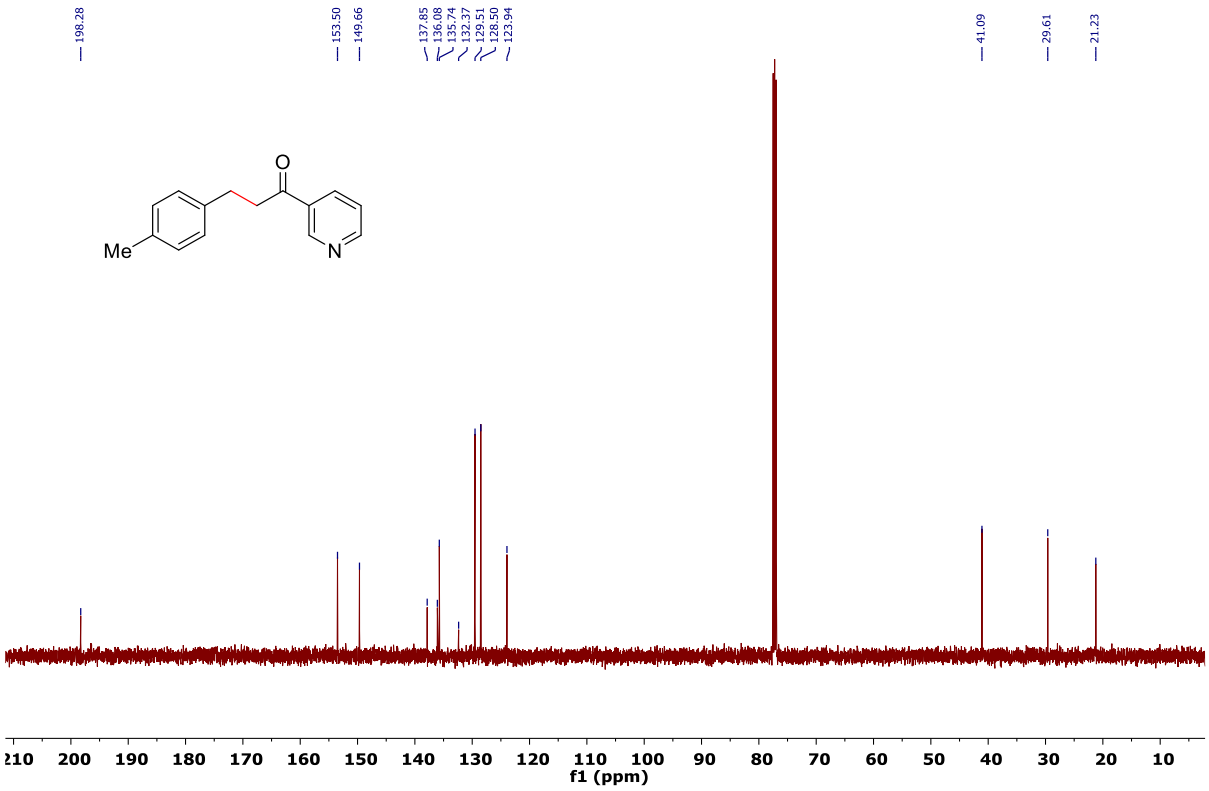

7.85
7.84
7.17
7.16
7.10
7.09
6.85
6.83
3.78
3.15
3.14
3.12
2.96
2.94
2.93
O
Cl
OMe
1.98
2.13
2.05
2.00
2.98
1.84
2.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

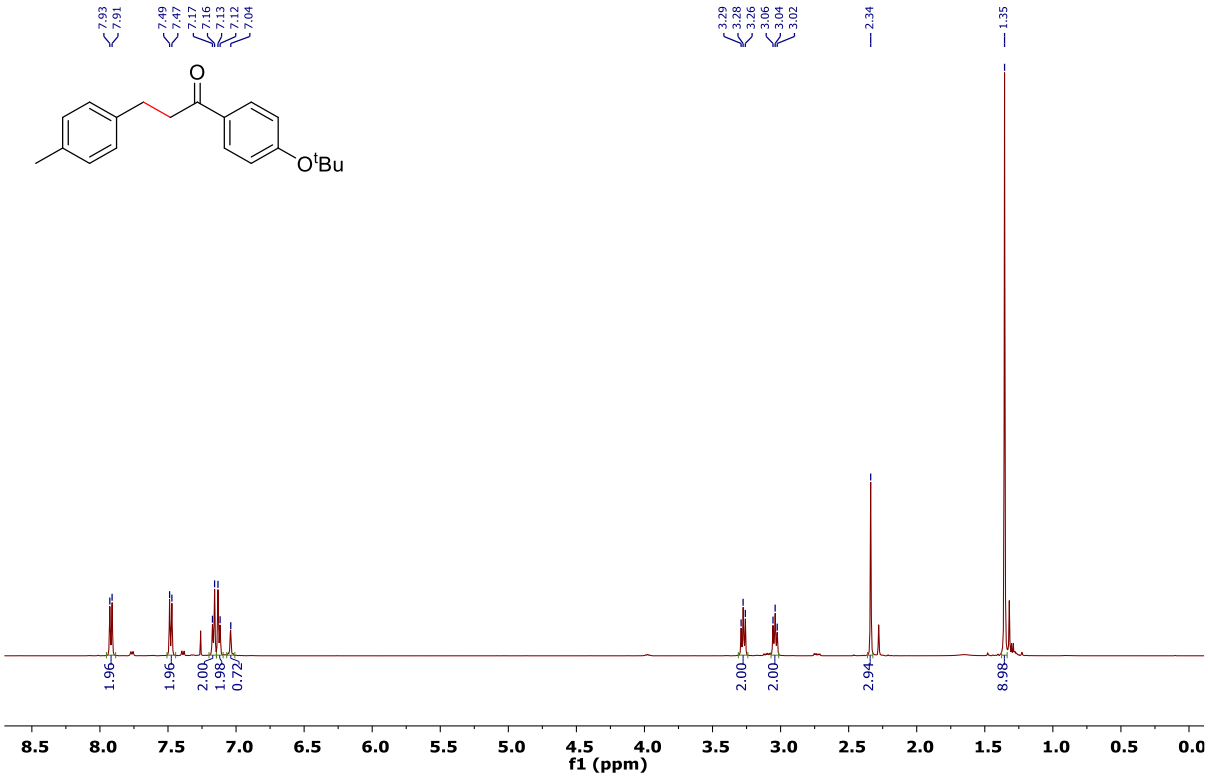

197.87
163.95
140.35
132.21
130.73
130.28
129.00
114.19
55.92
40.23
29.98
O
Cl
OMe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

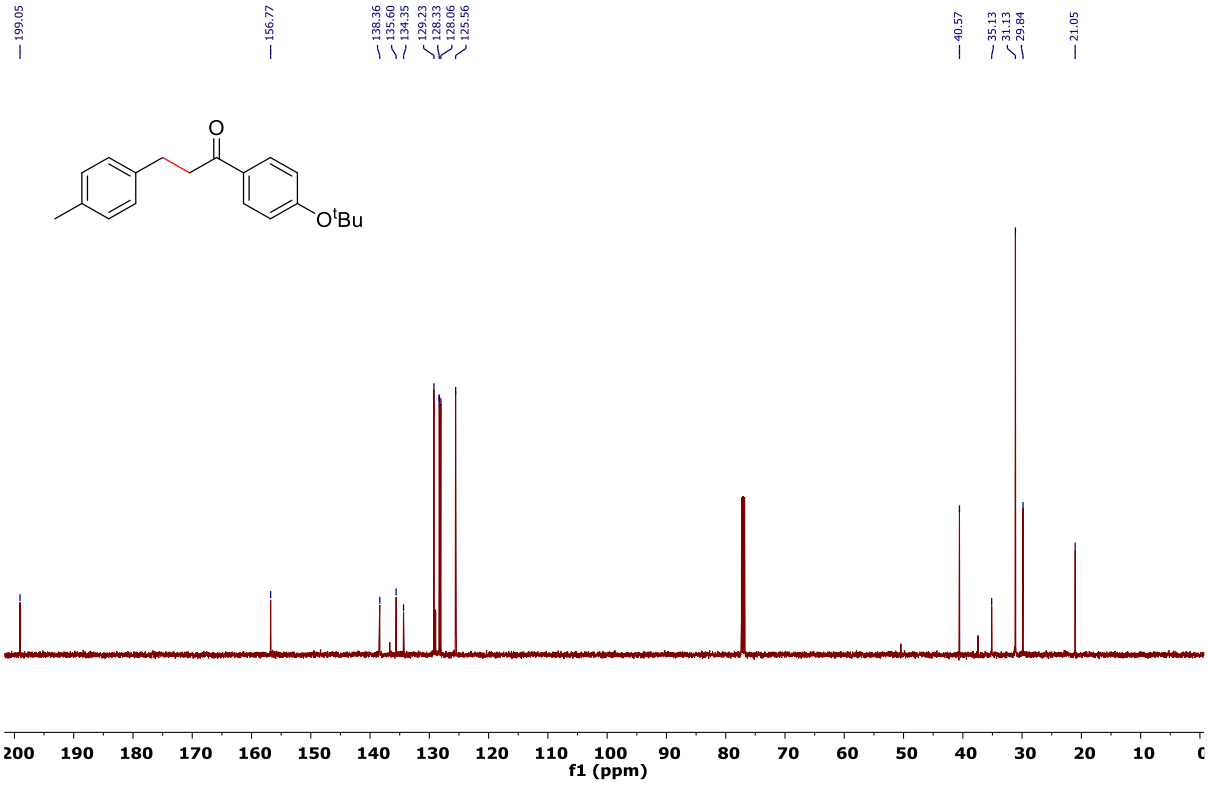

12.31
7.76
7.74
7.48
7.46
7.45
7.16
7.14
7.13
7.11
7.00
6.98
6.90
6.88
6.87
3.33
3.31
3.30
3.05
3.03
3.02
2.33
O
OH
0.85
0.98
0.97
3.83
0.94
0.96
2.00
2.01
3.08
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

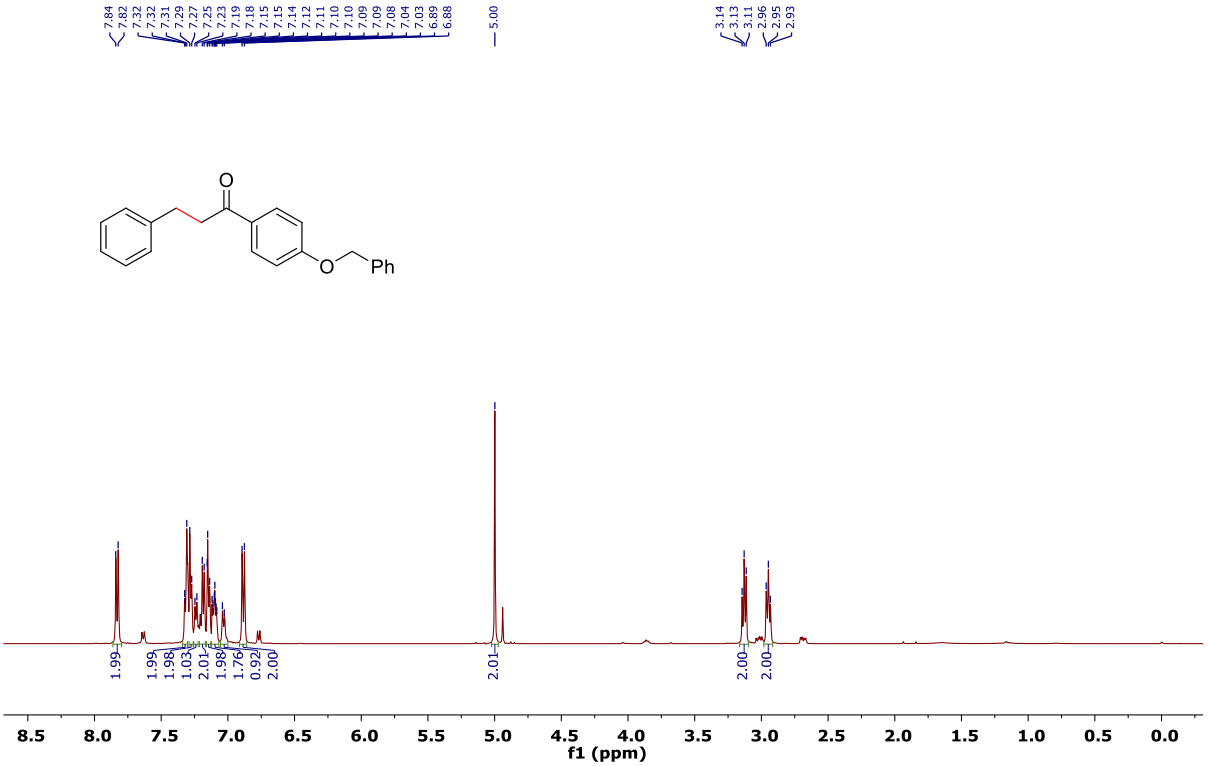

205.91
162.86
138.00
136.70
136.24
130.22
129.67
128.64
119.68
119.30
118.95
40.59
30.02
21.40
O
OH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


9.16
8.77
8.77
8.24
8.22
7.43
7.42
7.42
7.41
7.15
7.13
7.12
7.10
3.31
3.30
3.28
3.06
3.05
3.03
2.32
O
Me
N
1.00
1.00
0.99
1.04
3.95
1.99
2.00
3.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


198.28
153.50
149.66
137.85
136.08
135.74
132.37
129.51
128.50
123.94
41.09
29.61
21.23
O
Me
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


7.93
7.91
7.49
7.47
7.17
7.16
7.13
7.12
7.04
3.29
3.28
3.26
3.06
3.04
3.02
2.34
1.35
O
O^{t}Bu
1.96
1.96
2.00
1.98
0.72
2.00
2.00
2.94
8.98
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)


199.05
156.77
138.36
135.60
134.35
129.23
128.33
128.06
125.56
40.57
35.13
31.13
29.84
21.05
O
O^{t}Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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7.82
7.32
7.32
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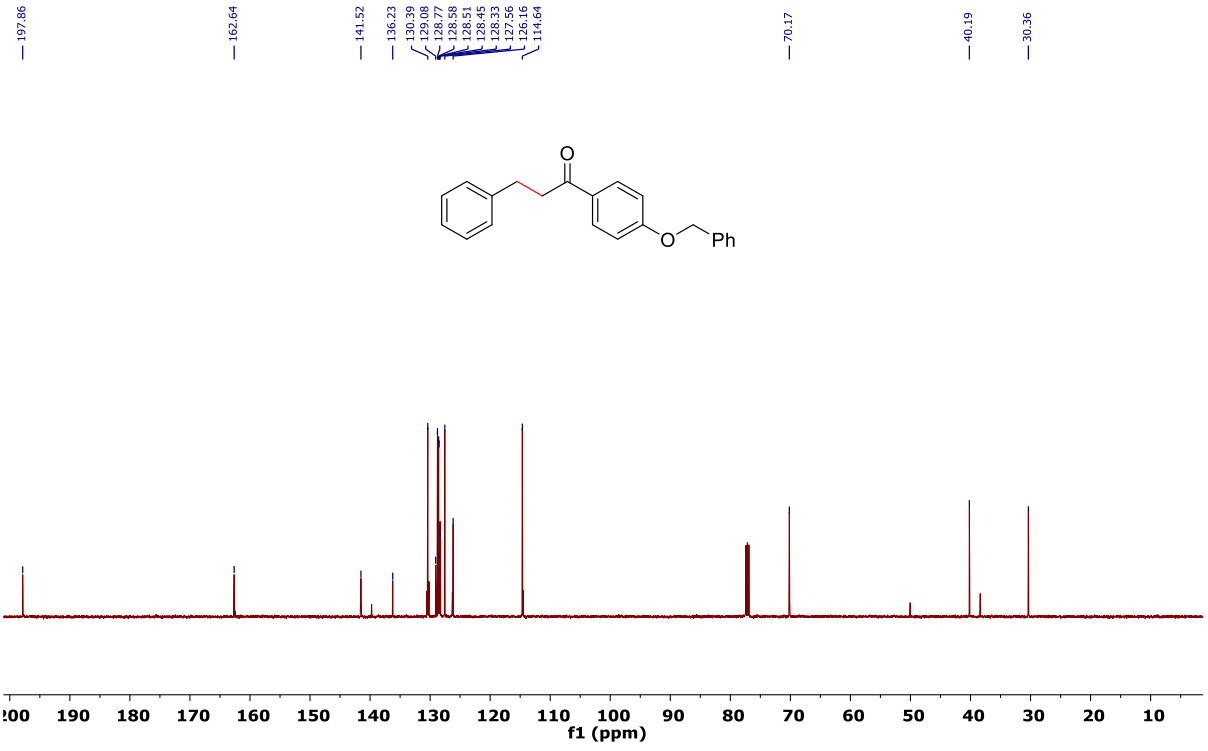
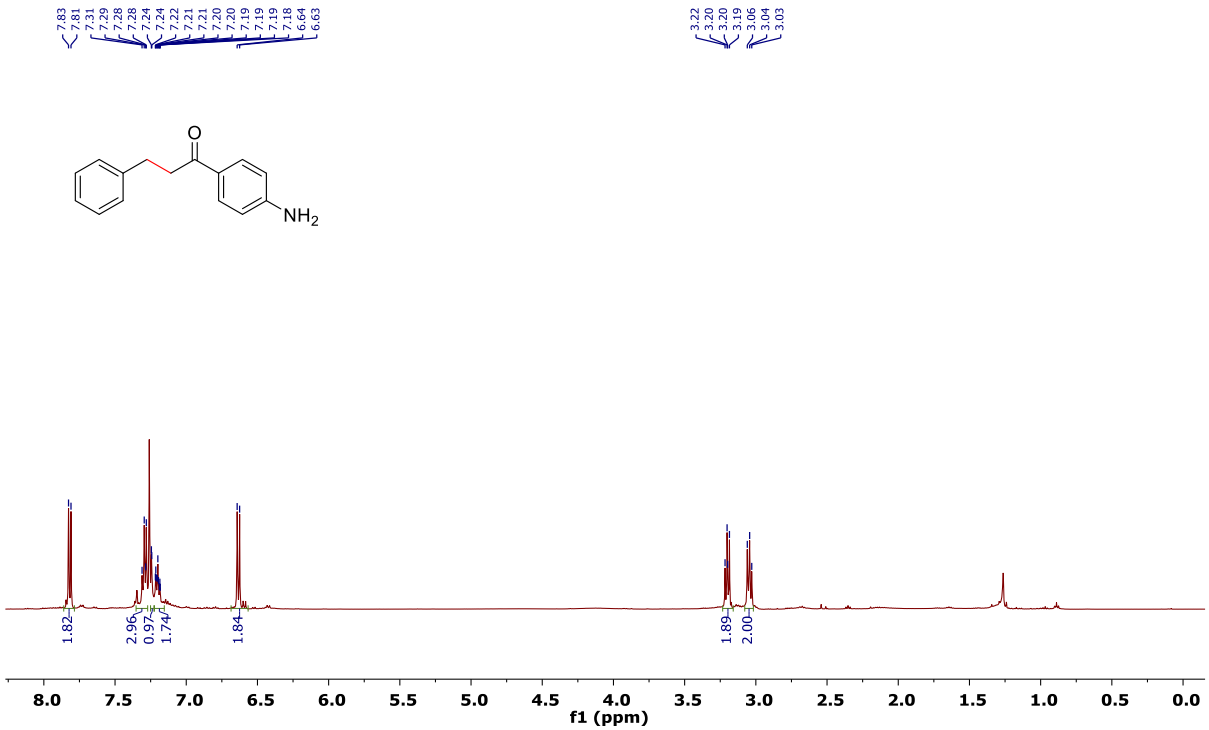
7.25
7.23
7.19
7.18
7.15
7.15
7.14
7.12
7.11
7.10
7.10
7.09
7.09
7.08
7.04
7.03
6.89
6.88
5.00
3.14
3.13
3.11
2.96
2.95
2.93
O
O
Ph
1.99
1.99
1.98
1.03
2.01
1.98
1.76
0.92
2.00
2.01
2.00
2.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


197.86
162.64
141.52
136.23
130.39
129.08
128.77
128.58
128.51
128.45
128.33
127.56
126.16
114.64
70.17
40.19
30.36
O
O
Ph
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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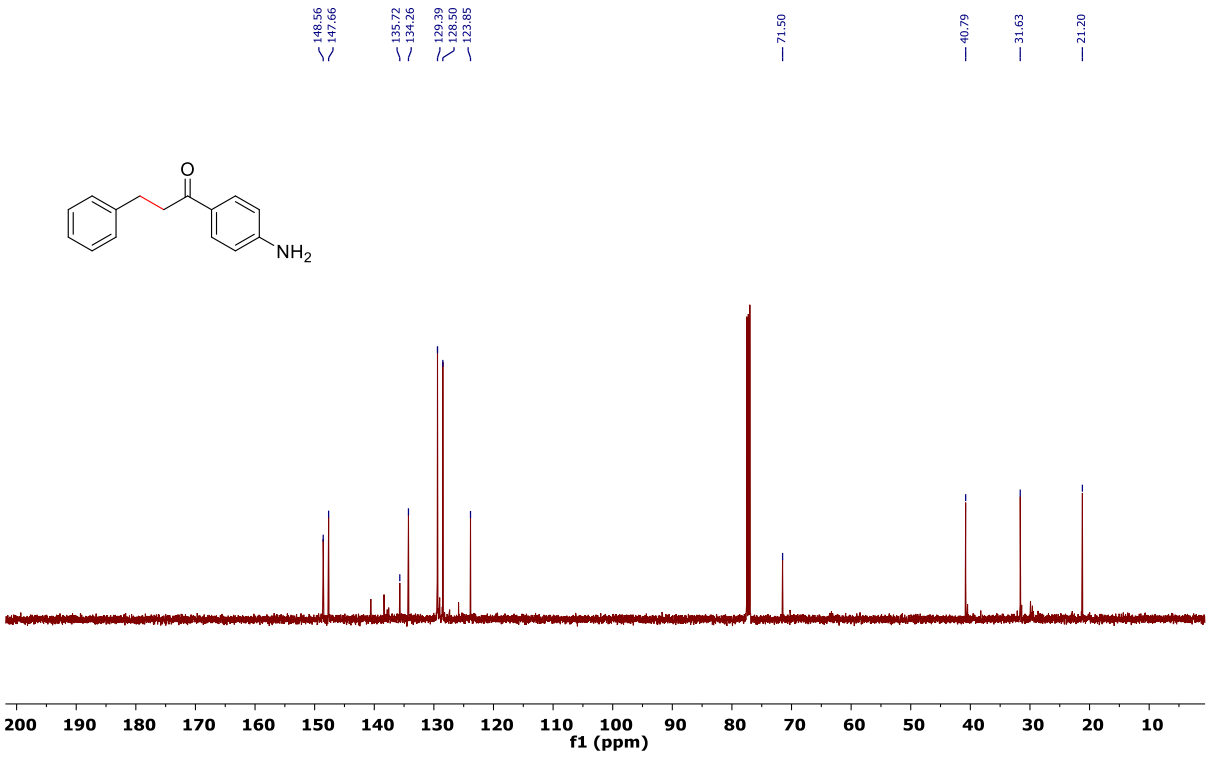
7.29
7.28
7.28
7.24
7.24
7.22
7.21
7.21
7.20
7.20
7.19
7.19
7.19
7.18
6.64
6.63
3.22
3.20
3.20
3.19
3.06
3.04
3.03
O
NH2
1.82
2.96
0.97
1.74
1.84
1.89
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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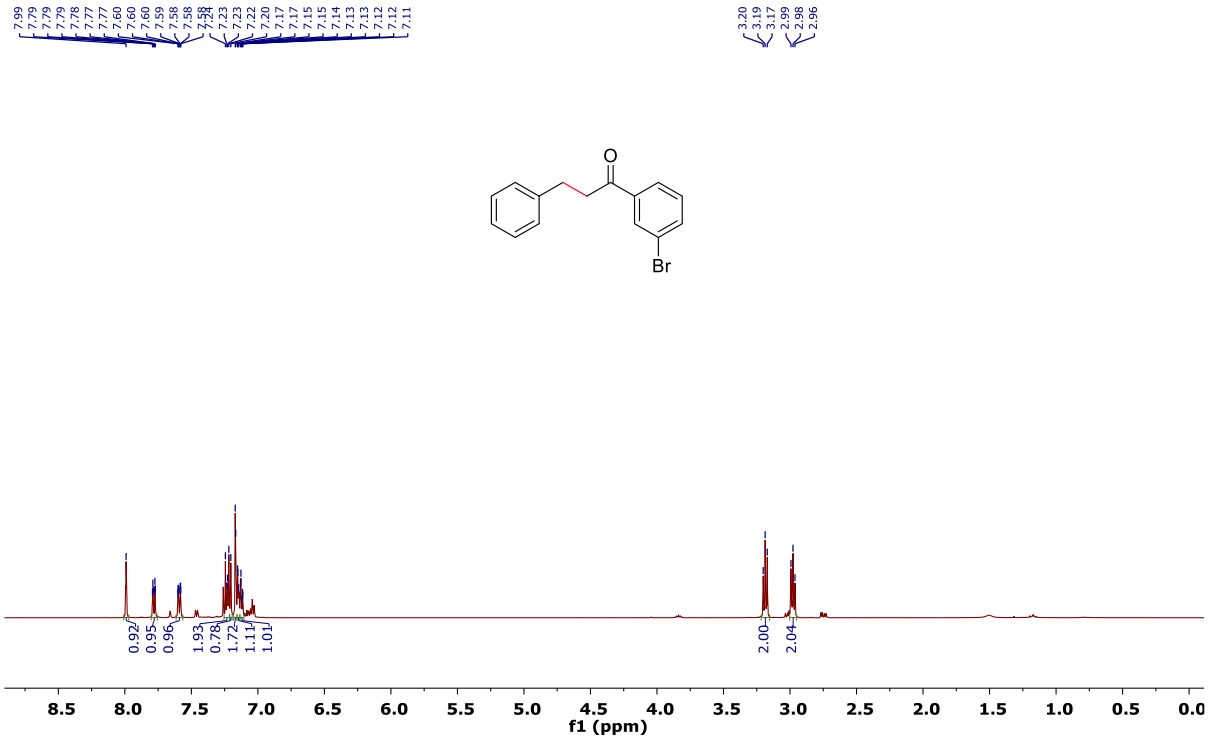
147.66
135.72
134.26
129.39
128.50
123.85
71.50
40.79
31.63
21.20
O
NH2
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.79
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7.77
7.60
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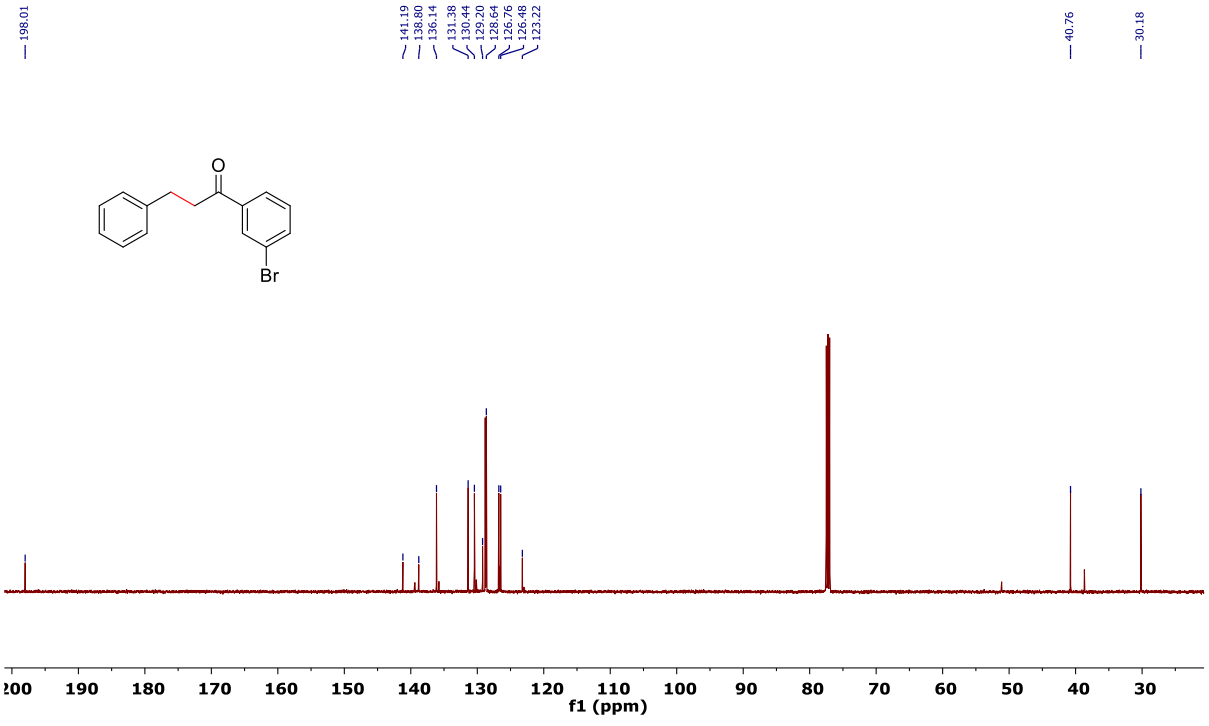
7.60
7.59
7.58
7.58
7.58
7.24
7.23
7.23
7.22
7.20
7.17
7.17
7.15
7.15
7.14
7.13
7.13
7.12
7.12
7.11
3.20
3.19
3.17
2.99
2.98
2.96
O
Br
0.92
0.95
0.96
1.93
0.78
1.72
1.11
1.01
2.00
2.04
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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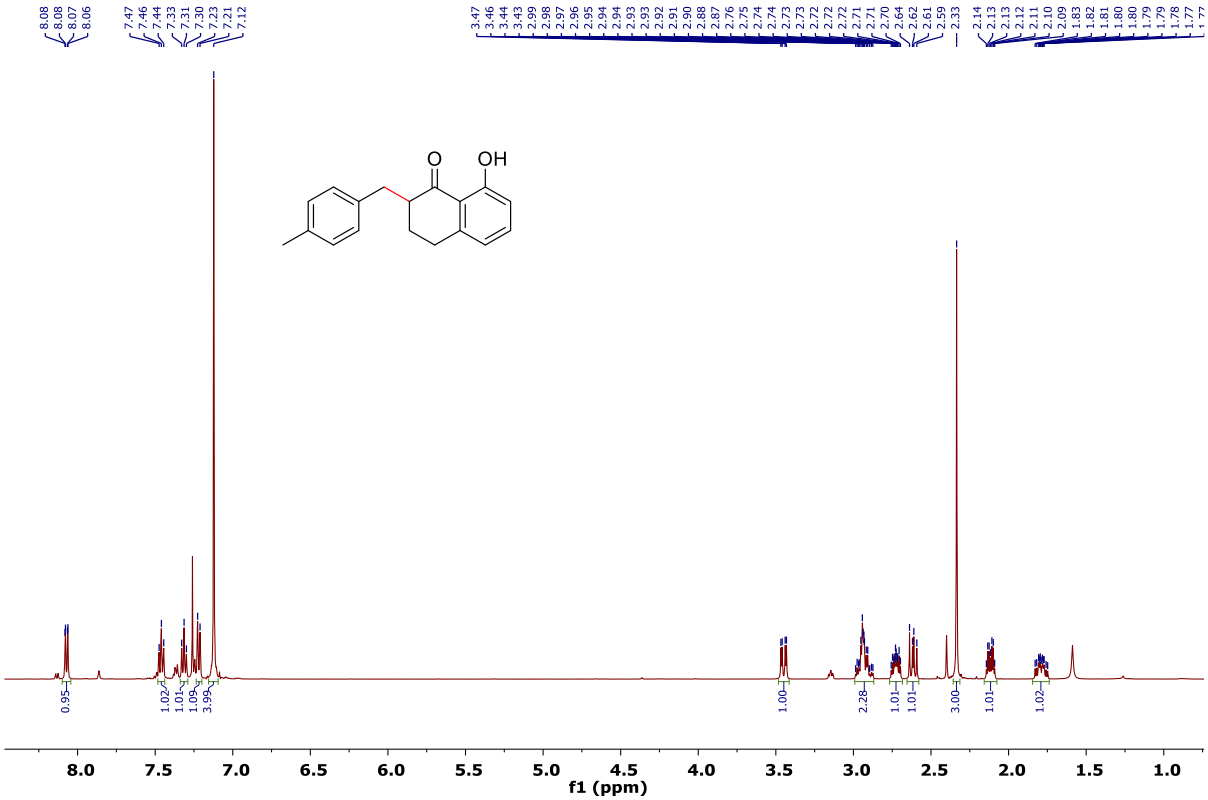
141.19
138.80
136.14
131.38
130.44
129.20
128.64
126.76
126.48
123.22
40.76
30.18
O
Br
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (ppm)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8.08
8.07
8.06
7.47
7.46
7.44
7.33
7.31
7.30
7.23
7.21
7.12
3.47
3.46
3.44
3.43
2.99
2.98
2.97
2.96
2.95
2.94
2.94
2.93
2.93
2.92
2.91
2.90
2.88
2.87
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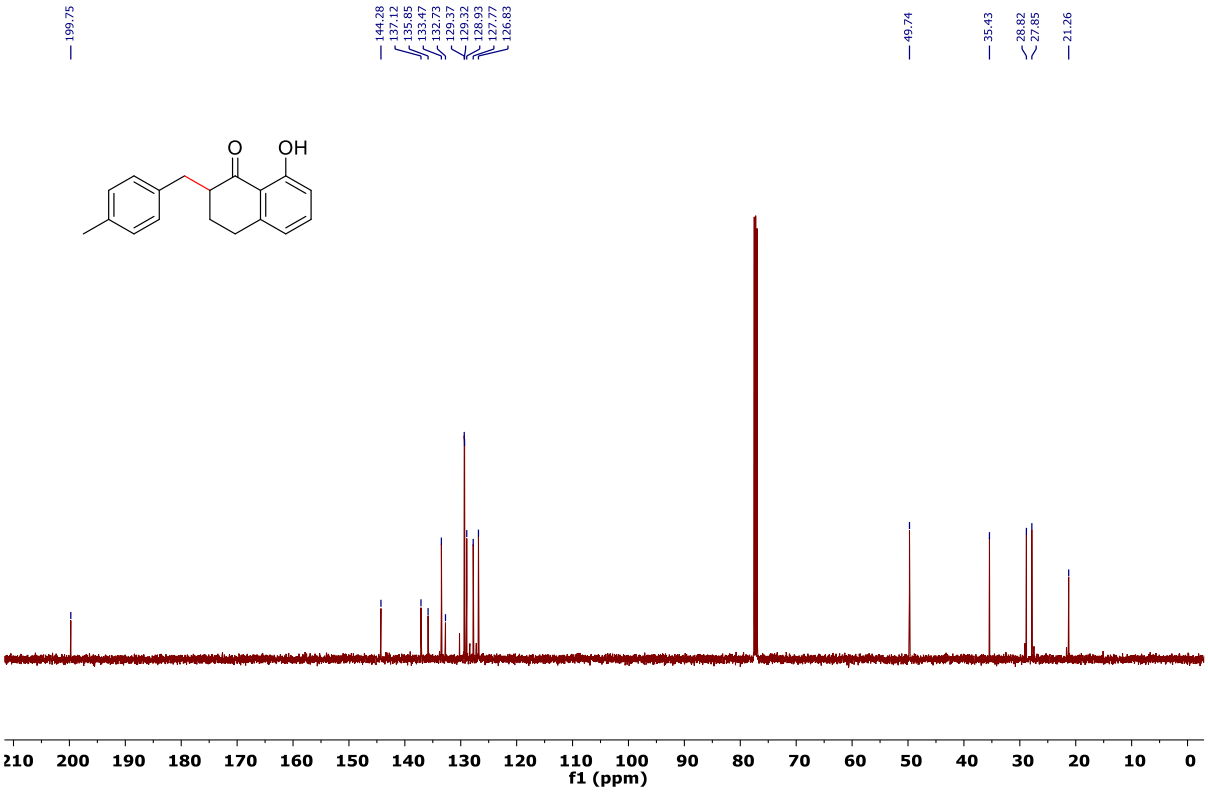
2.74
2.73
2.72
2.72
2.72
2.71
2.71
2.70
2.64
2.62
2.61
2.59
2.33
2.14
2.13
2.13
2.12
2.11
2.10
2.09
1.83
1.82
1.81
1.80
1.80
1.79
1.79
1.78
1.77
O
OH
0.95
1.02
1.01
1.09
3.99
1.00
2.28
1.01
1.01
3.00
1.01
1.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)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144.28
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135.85
133.47
132.73
129.37
129.32
128.93
127.77
126.83
49.74
35.43
28.82
27.85
21.26
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)


8.24
8.22
8.20
8.19
8.19
8.17
8.17
8.17
7.89
7.88
7.84
7.84
7.83
7.83
7.76
7.75
7.74
7.74
7.74
7.73
7.72
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7.49
7.48
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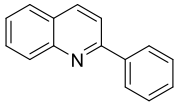
7.47
7.46
7.46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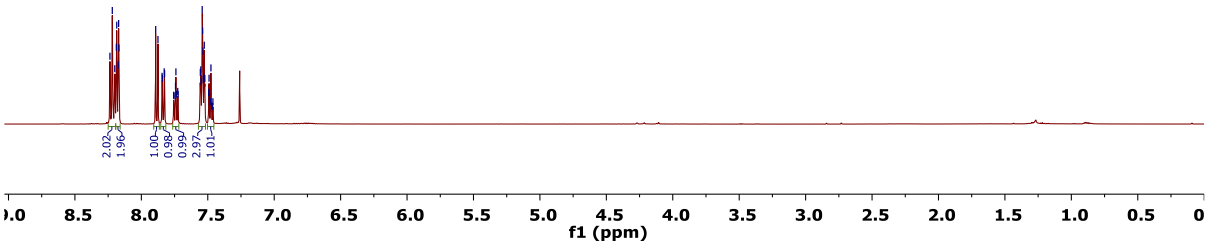
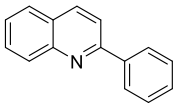
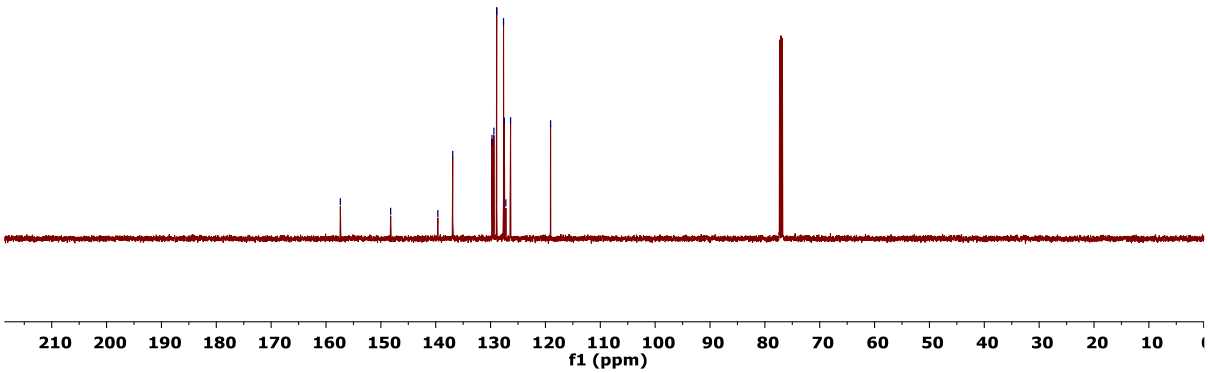
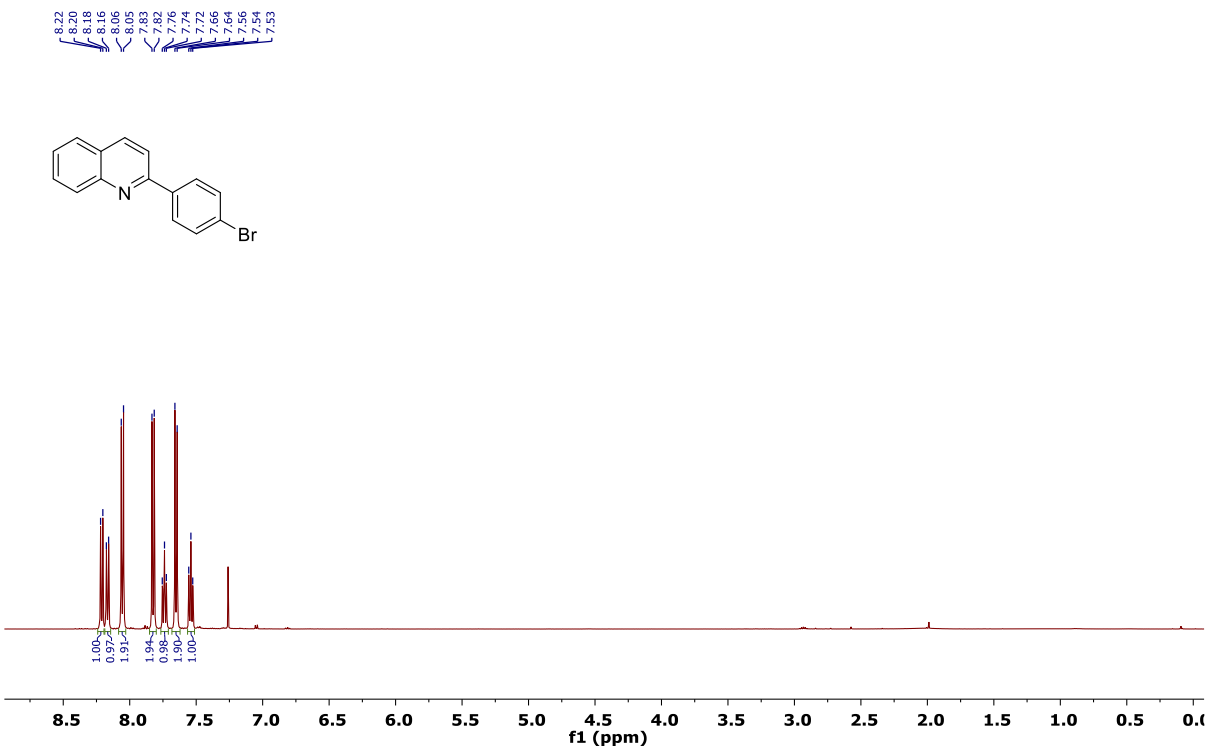
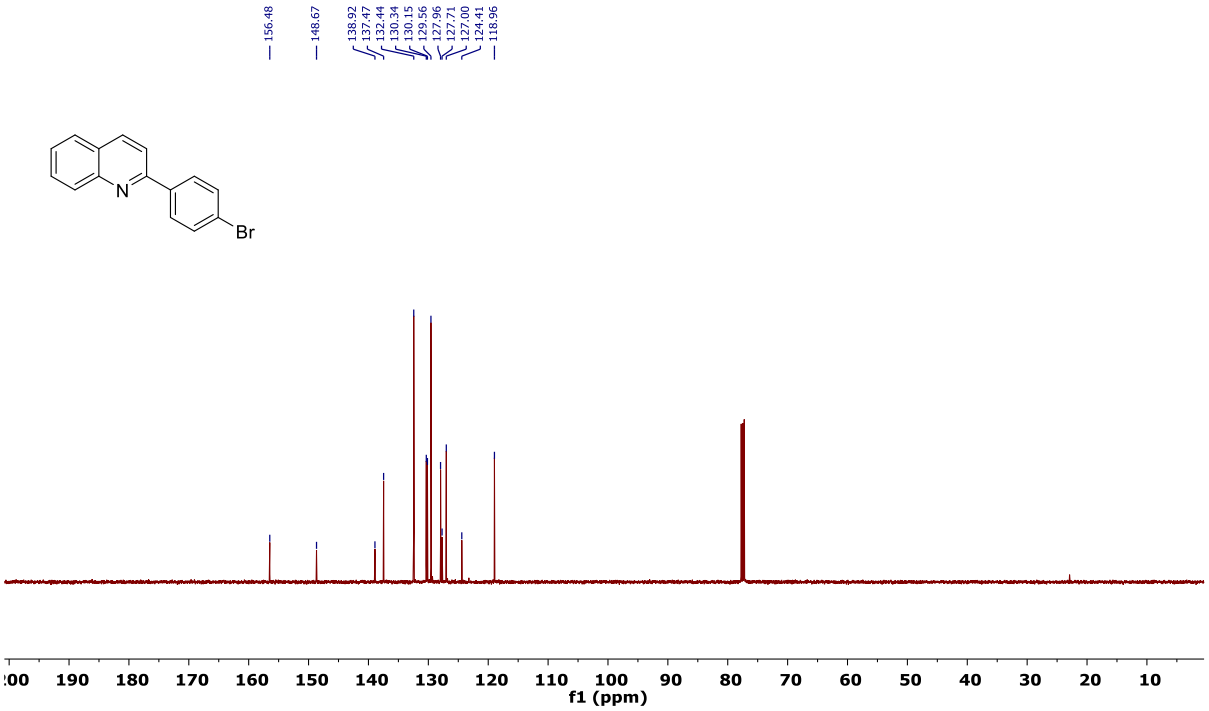

2.02
1.96
1.00
0.98
0.99
2.97
1.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

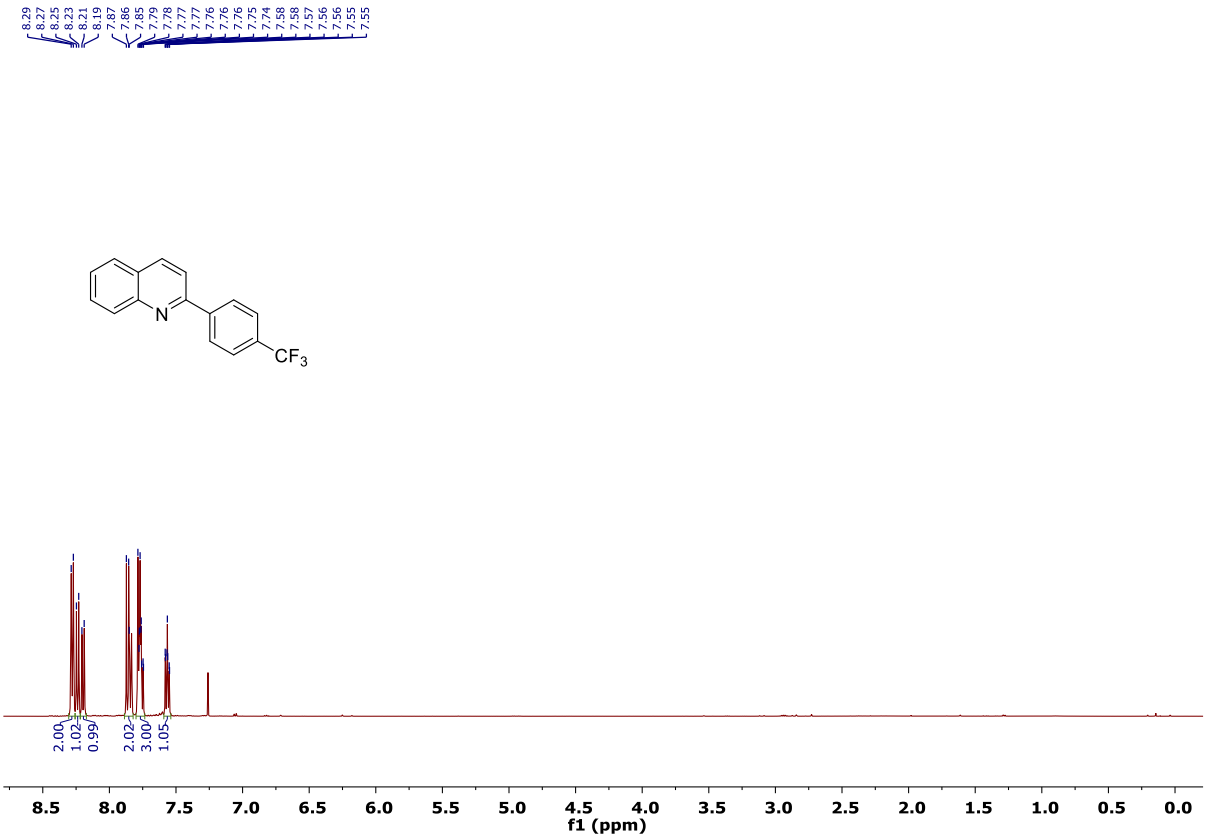

157.38
148.22
139.61
136.90
129.75
129.70
129.40
128.88
127.64
127.49
127.22
126.35
119.06

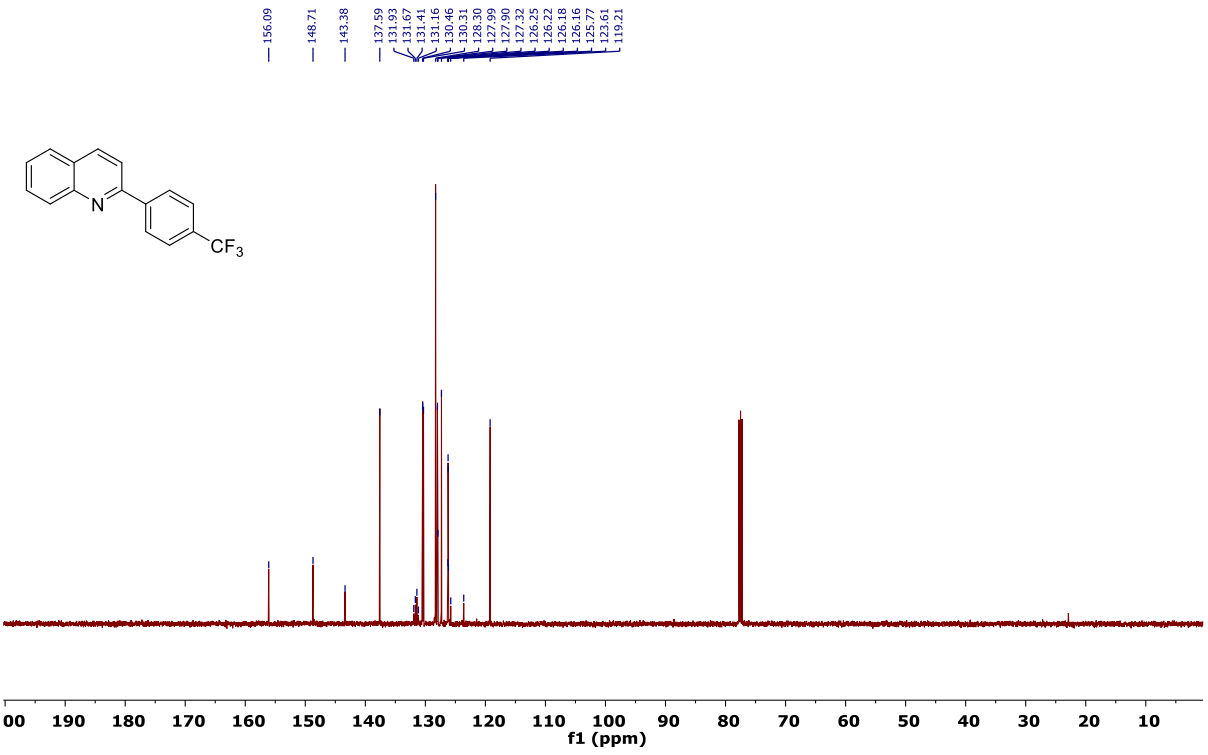

N


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

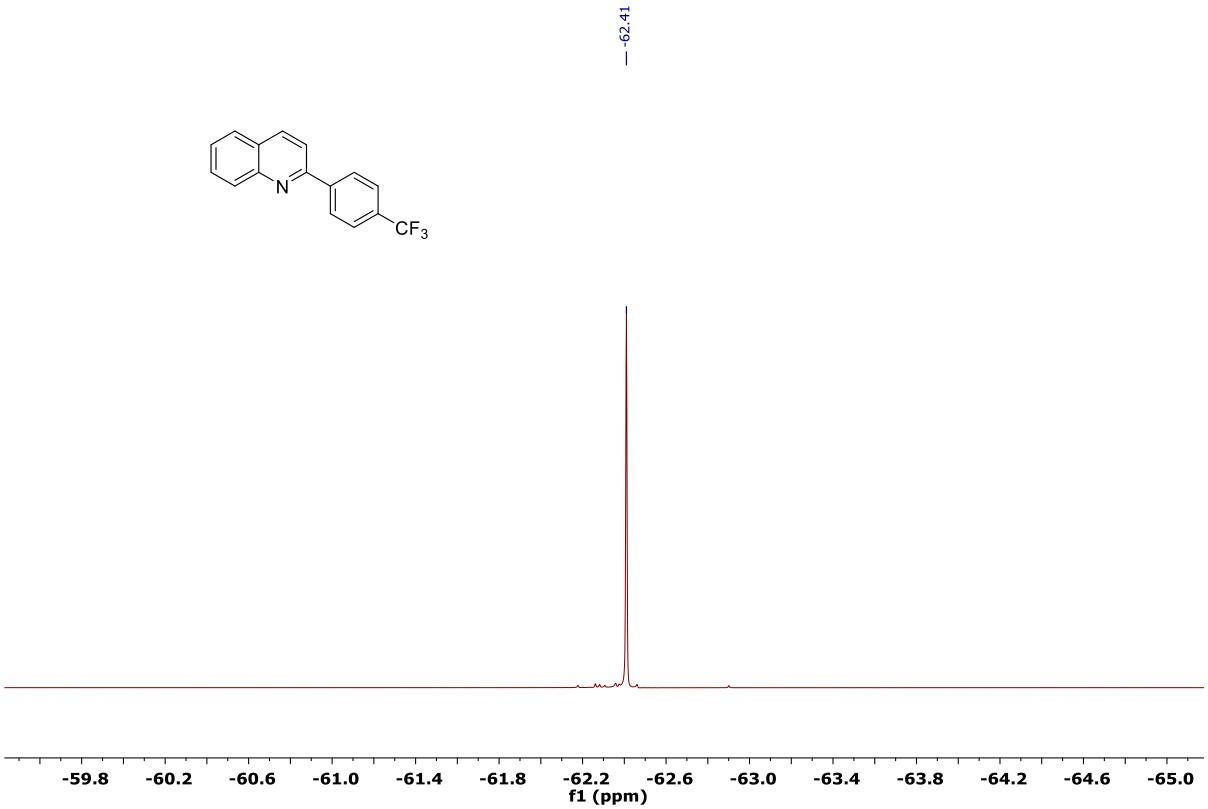

8.22
8.20
8.18
8.16
8.06
8.05
7.83
7.82
7.76
7.74
7.72
7.66
7.64
7.56
7.54
7.53
N
Br
1.00
0.97
1.91
1.94
0.98
1.90
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

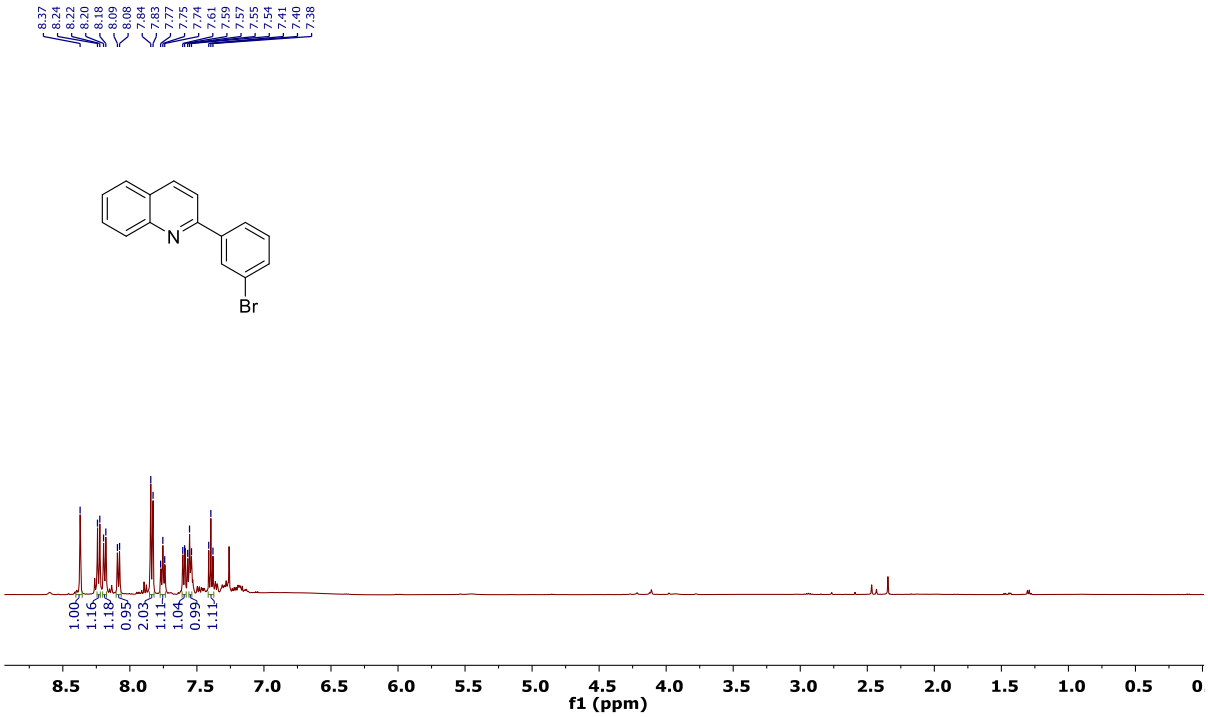

156.48
148.67
138.92
137.47
132.44
130.34
130.15
129.56
127.96
127.71
127.00
124.41
118.96
N
Br
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

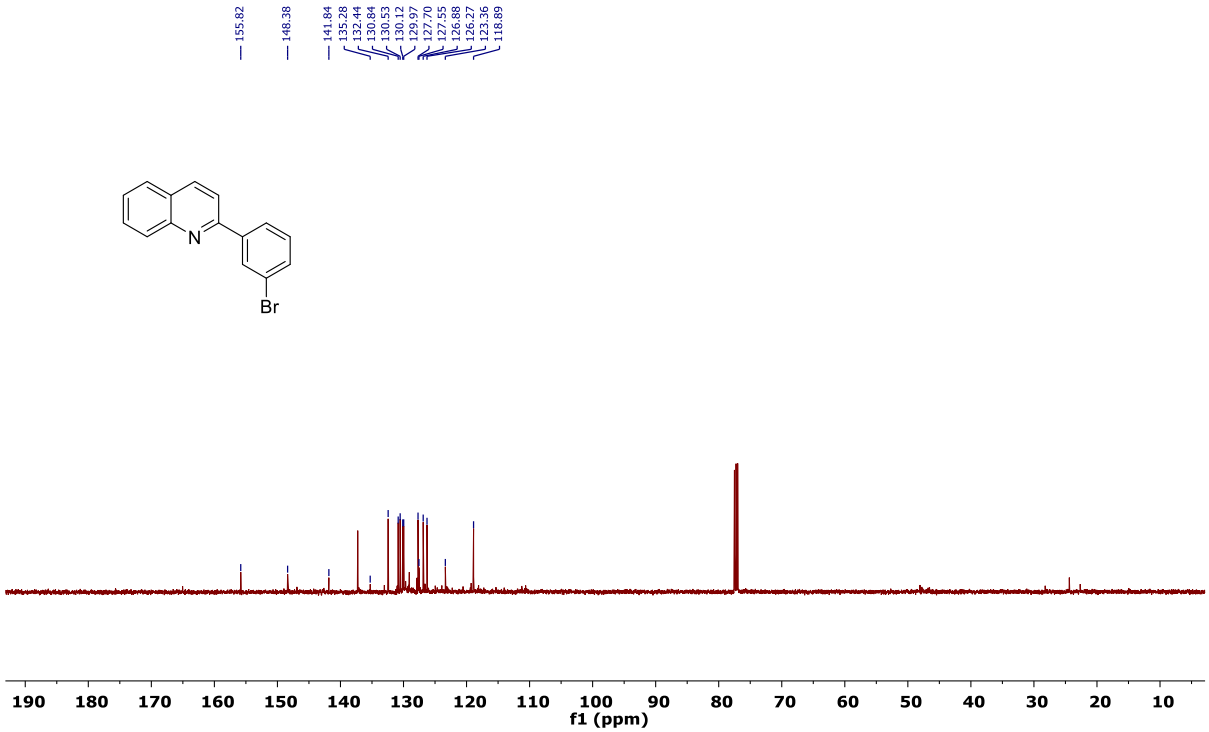

8.29
8.27
8.25
8.23
8.21
8.19
7.87
7.86
7.85
7.79
7.78
7.77
7.77
7.76
7.76
7.76
7.75
7.74
7.58
7.58
7.57
7.56
7.56
7.55
7.55
N
CF3
2.00
1.02
0.99
2.02
3.00
1.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

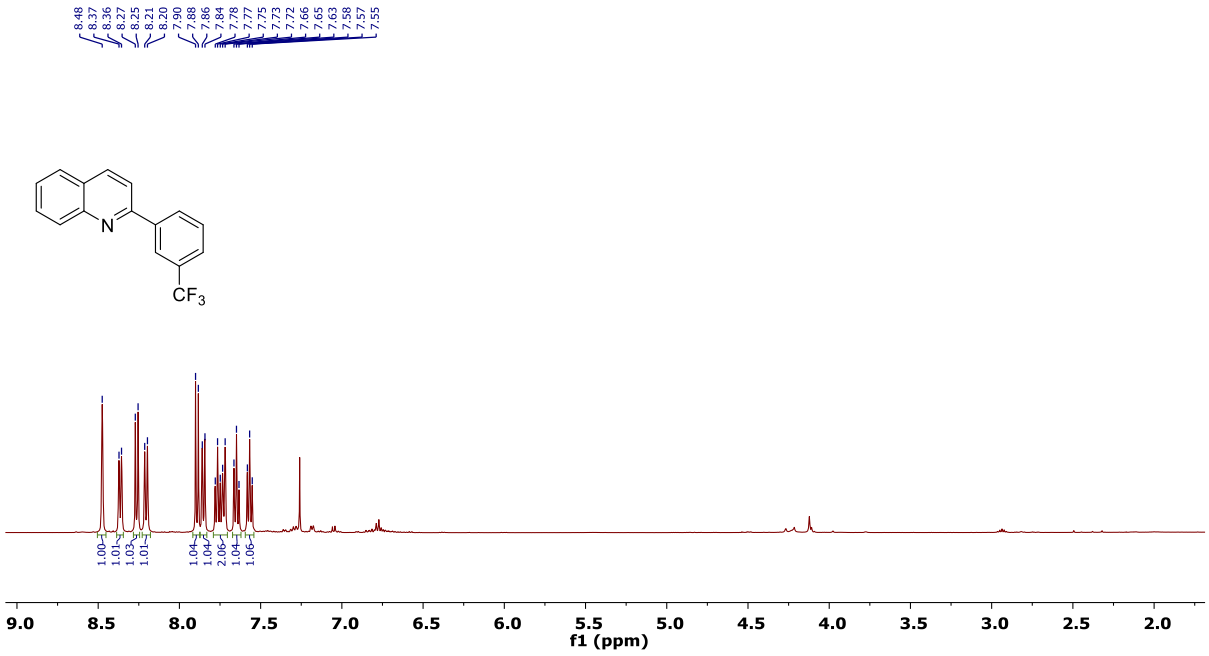

156.09
148.71
143.38
137.59
131.93
131.67
131.41
131.16
130.46
130.31
128.30
127.99
127.90
127.32
126.25
126.22
126.18
126.16
125.77
123.61
119.21
N
CF3
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

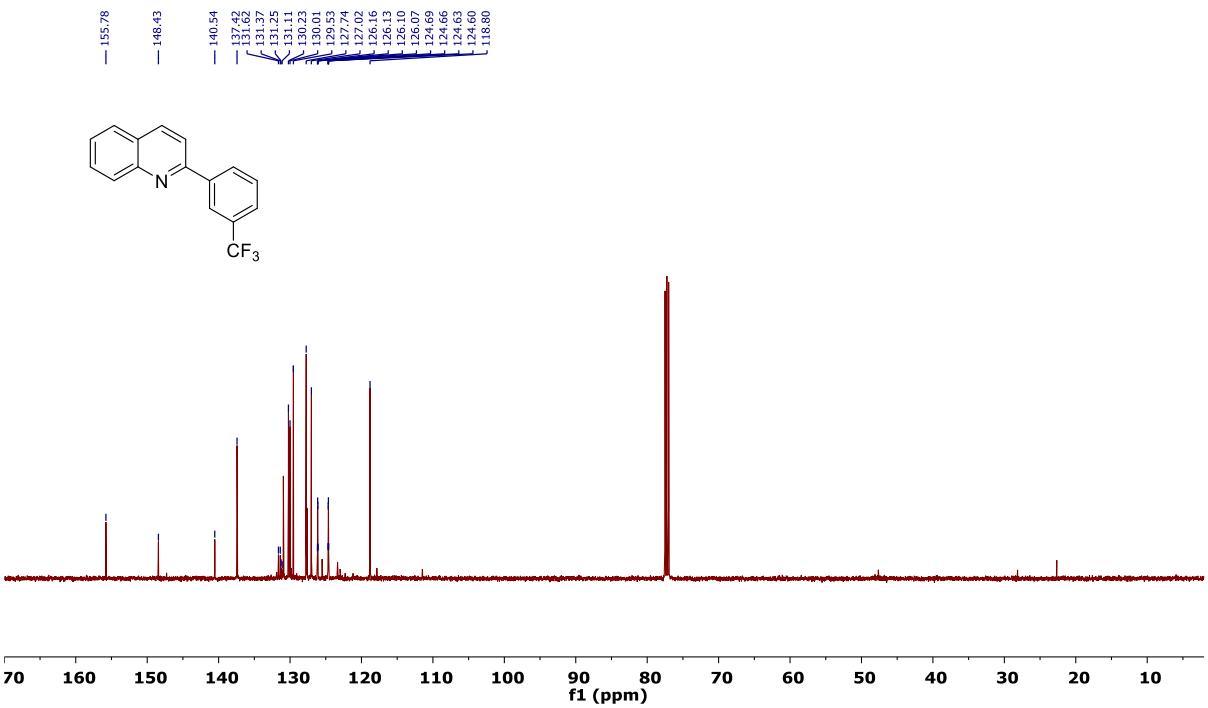
^{19}F NMR


-62.41
N
CF3
-59.8
-60.2
-60.6
-61.0
-61.4
-61.8
-62.2
-62.6
-63.0
-63.4
-63.8
-64.2
-64.6
-65.0
f1 (ppm)

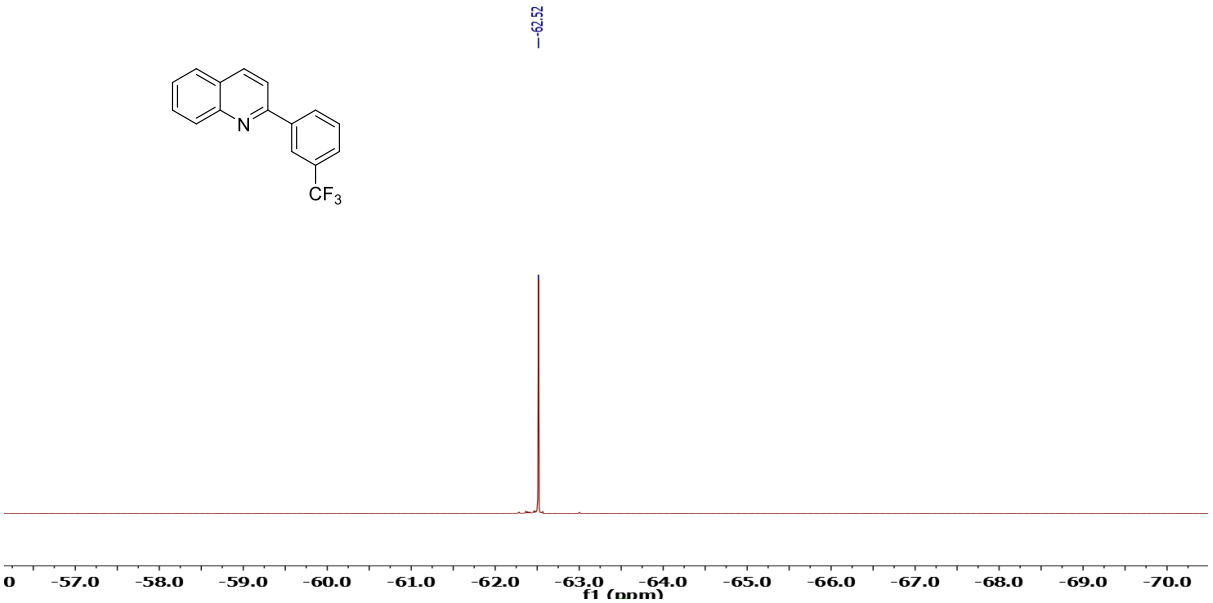

8.37
8.24
8.22
8.20
8.18
8.09
8.08
7.84
7.83
7.77
7.75
7.74
7.61
7.59
7.57
7.55
7.54
7.41
7.40
7.38
N
Br
1.00
1.16
1.18
0.95
2.03
1.11
1.04
0.99
1.11
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

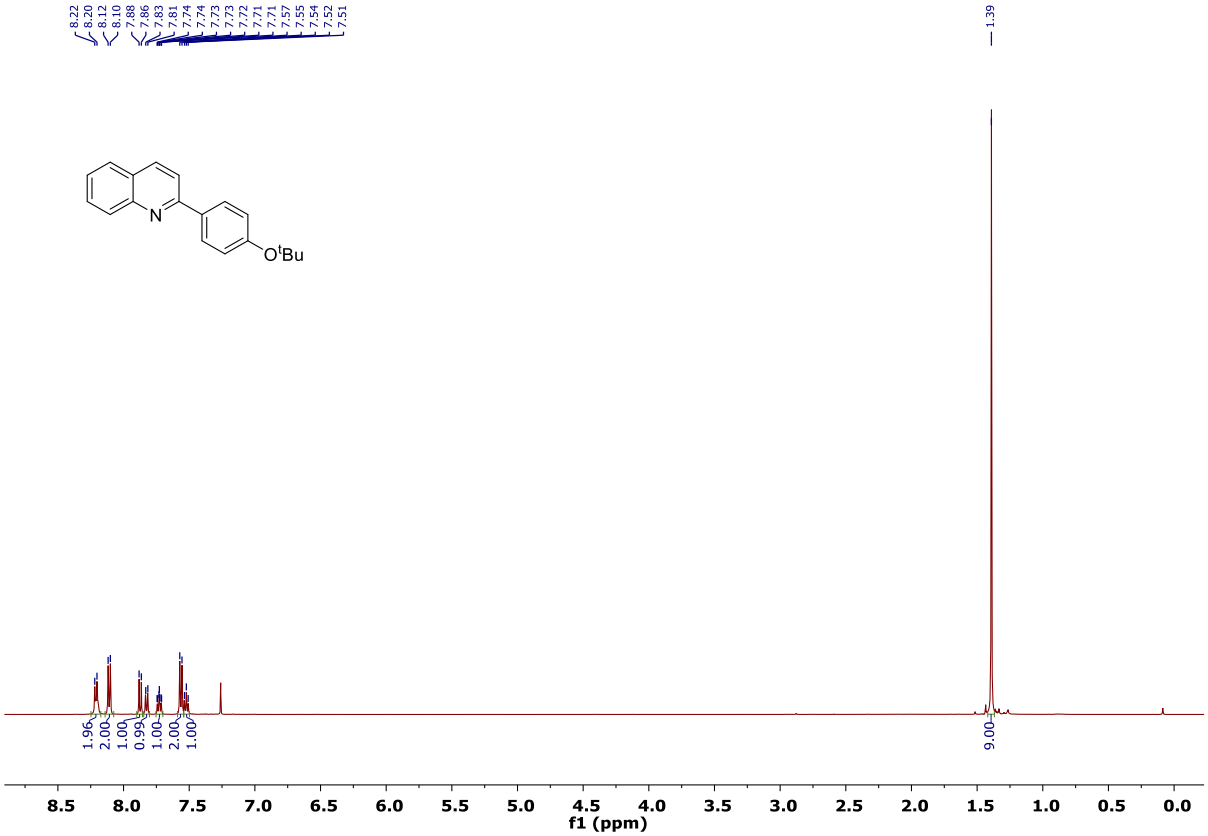

155.82
148.38
141.84
135.28
132.44
130.84
130.53
130.12
129.97
127.70
127.55
126.88
126.27
123.36
116.89
N
Br
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

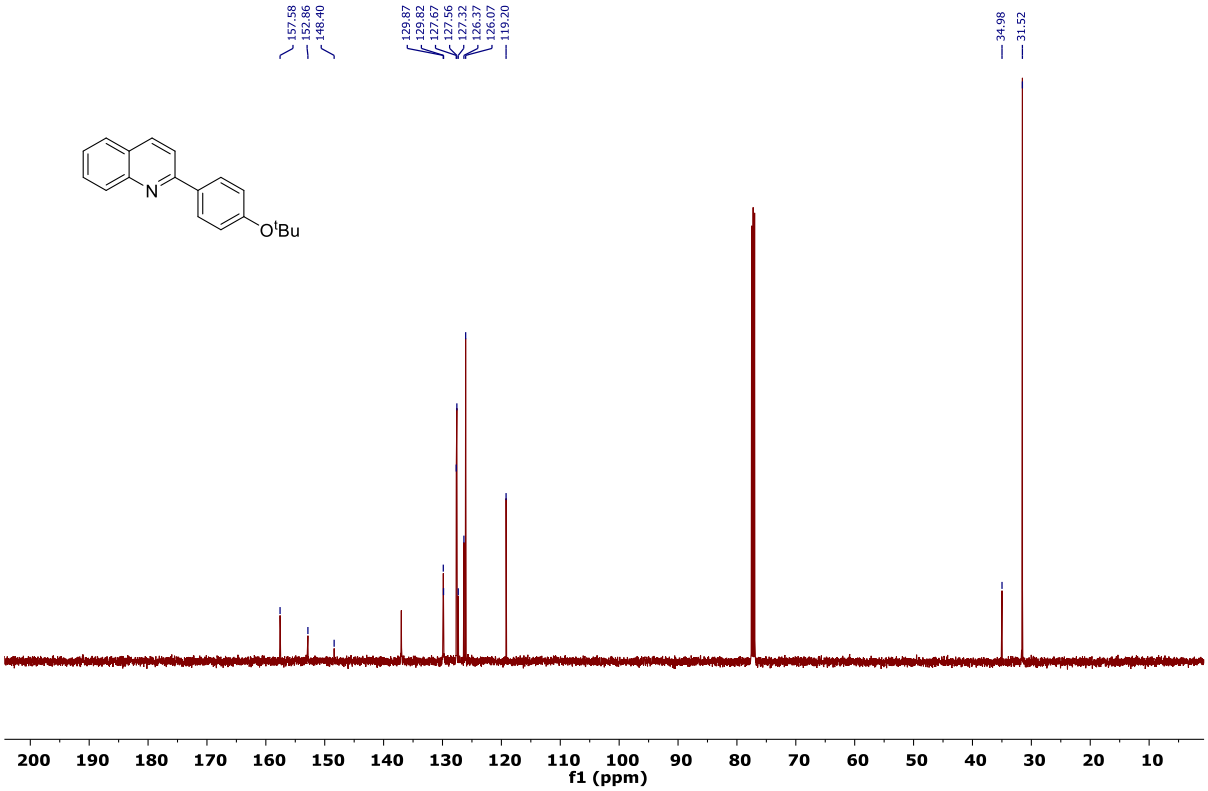

8.48
8.37
8.36
8.27
8.25
8.21
8.20
7.90
7.88
7.86
7.84
7.78
7.77
7.75
7.73
7.72
7.66
7.65
7.63
7.58
7.57
7.55
N
CF3
1.00
1.01
1.03
1.01
1.04
1.04
2.06
1.04
1.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
f1 (ppm)

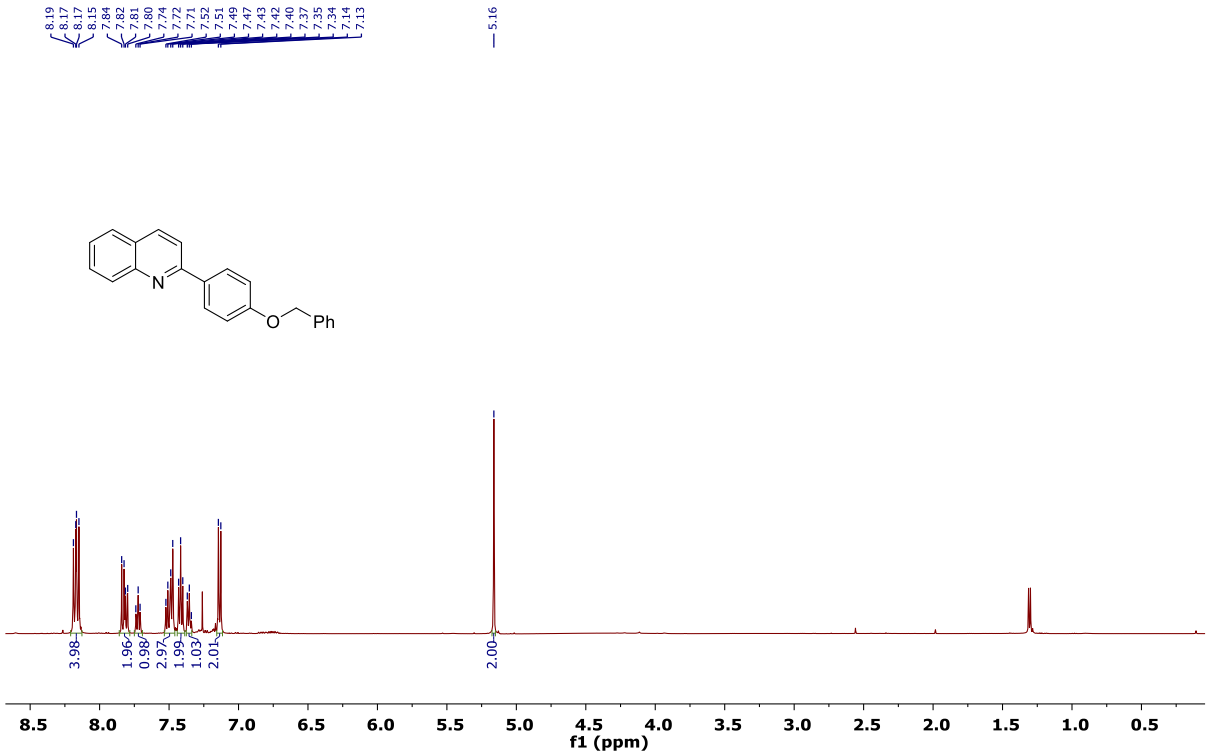

155.78
148.43
140.54
137.42
131.62
131.37
131.25
131.11
130.23
130.01
129.53
127.74
127.02
126.16
126.13
126.10
126.07
124.69
124.66
124.63
124.60
118.80
N
CF3
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

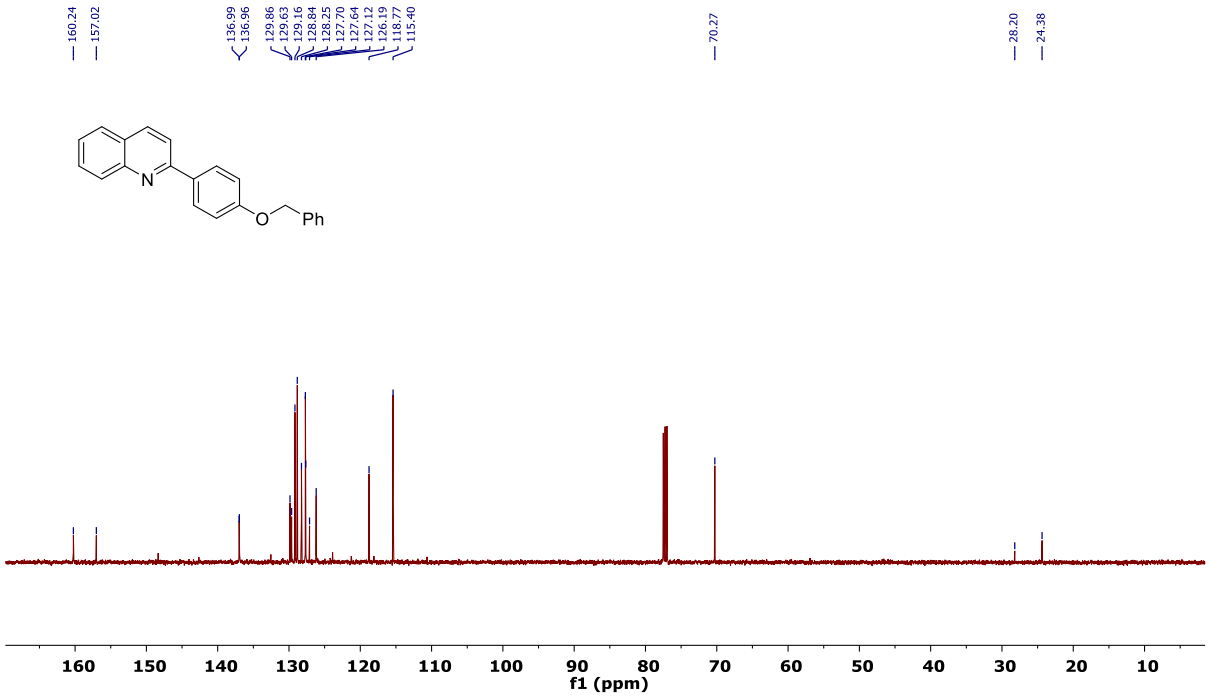
^{19}F NMR

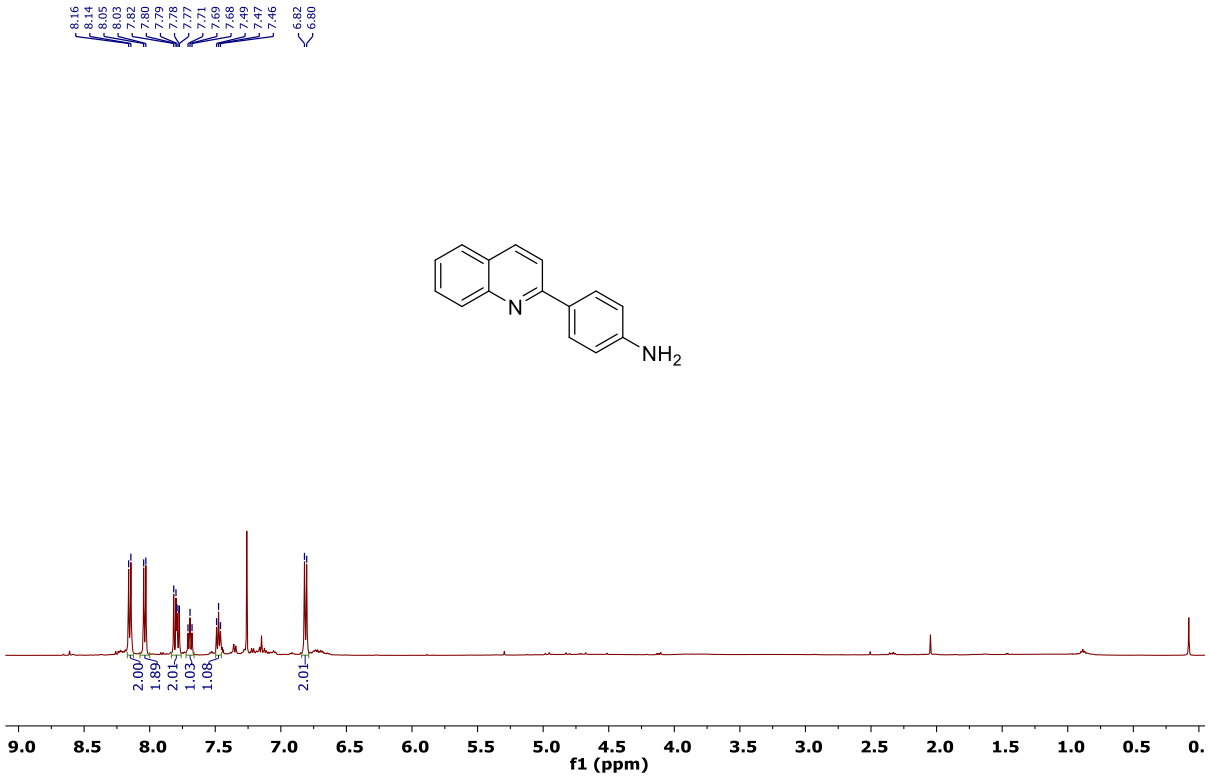

-62.52
N
CF3
0 -57.0 -58.0 -59.0 -60.0 -61.0 -62.0 -63.0 -64.0 -65.0 -66.0 -67.0 -68.0 -69.0 -70.0
f1 (ppm)

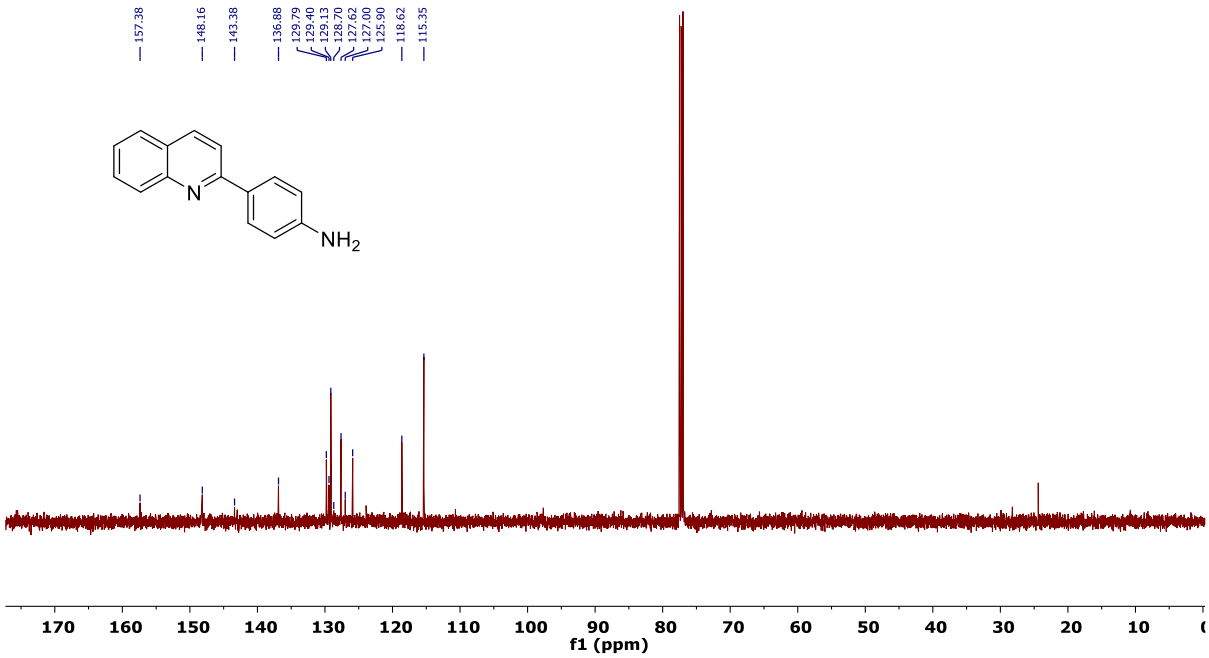

8.22
8.20
8.12
8.10
7.88
7.86
7.83
7.81
7.74
7.74
7.73
7.73
7.72
7.71
7.71
7.57
7.55
7.54
7.52
7.51
1.39
N
O^{t}Bu
1.96
2.00
1.00
0.99
1.00
2.00
1.00
9.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


157.58
152.86
148.40
129.87
129.82
127.67
127.56
127.32
126.37
126.07
119.20
34.98
31.52
N
O^{t}Bu
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


8.19
8.17
8.17
8.15
7.84
7.82
7.81
7.80
7.74
7.72
7.71
7.52
7.51
7.49
7.47
7.43
7.42
7.40
7.37
7.35
7.34
7.14
7.13
5.16
N
O
Ph
3.98
1.96
0.98
2.97
1.99
1.03
2.01
2.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


160.24
157.02
136.99
136.96
129.86
129.63
129.16
128.84
128.25
127.70
127.64
127.12
126.19
118.77
115.40
70.27
28.20
24.38
N
O
Ph
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


8.16
8.14
8.05
8.03
7.82
7.80
7.79
7.78
7.77
7.71
7.69
7.68
7.49
7.47
7.46
6.82
6.80
N
NH2
2.00
1.89
2.01
1.03
1.08
2.01
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.
f1 (ppm)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143.38
136.88
129.79
129.40
129.13
128.70
127.62
127.00
125.90
118.62
115.35
N
NH2
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


8.27
8.26
8.04
8.03
7.95
7.95
7.94
7.94
7.83
7.83
7.76
7.76
7.75
7.74
7.74
7.73
7.73
7.57
7.56
7.55
7.55
7.54
7.53
7.39
7.38
7.37
7.37
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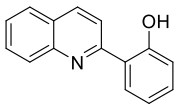
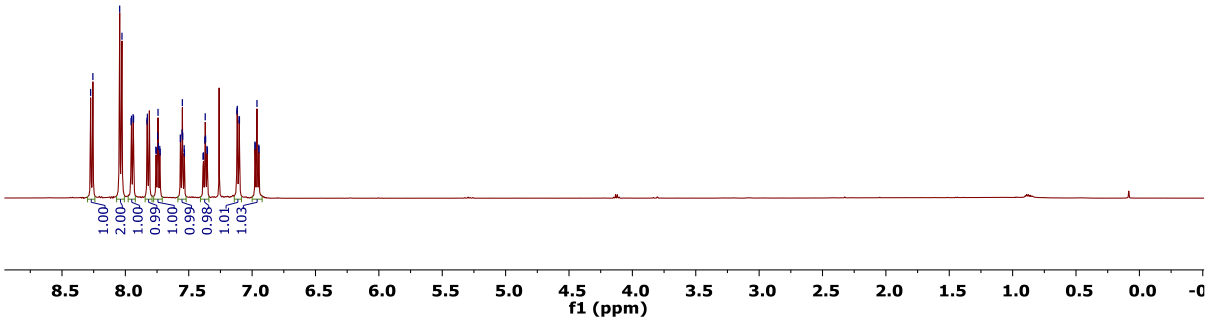
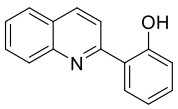
7.10
6.98
6.98
6.96
6.95
6.95

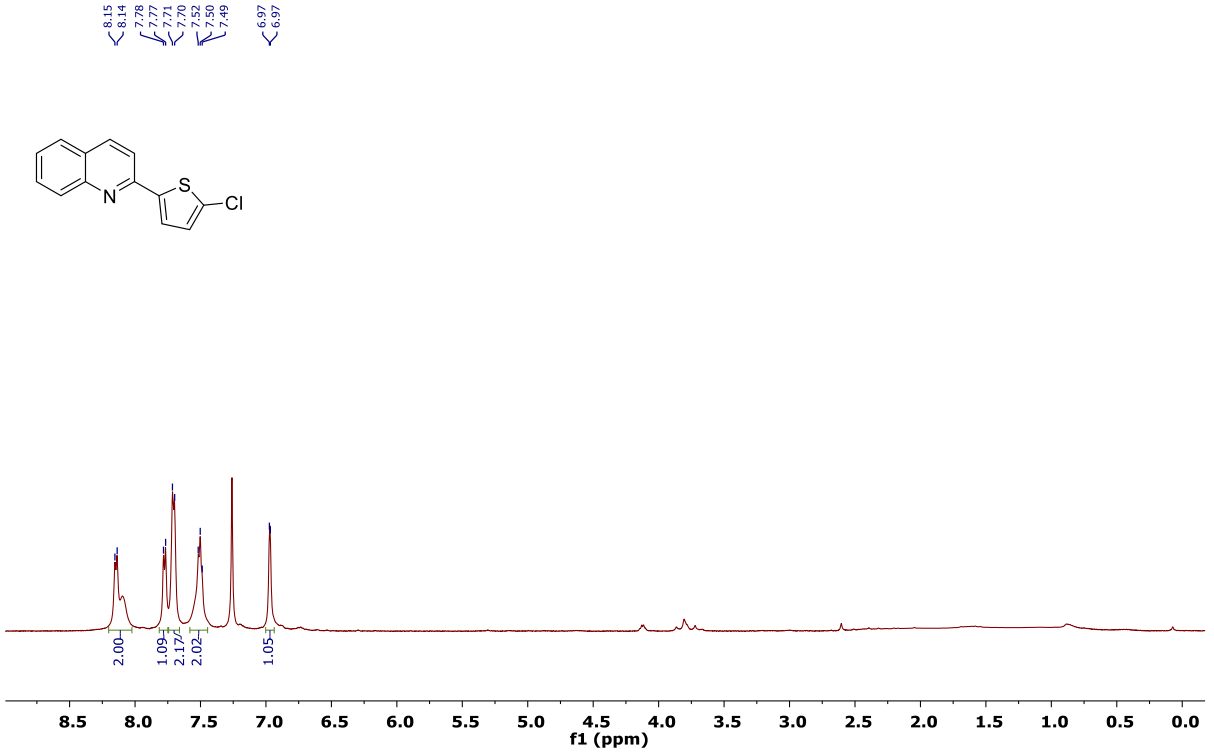

OH
N

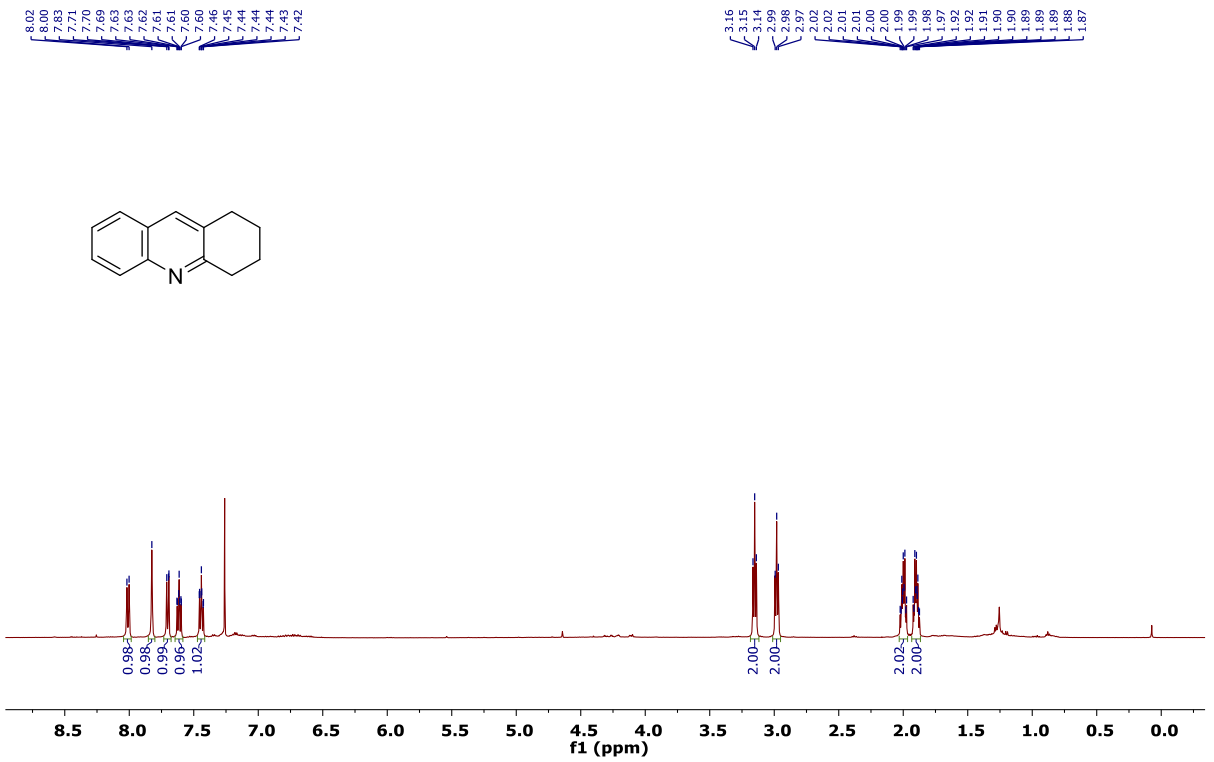

1.00
2.00
1.00
0.99
1.00
0.99
0.98
1.01
1.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


161.46
158.38
145.06
138.21
132.59
131.02
128.02
127.93
127.45
127.20
127.00
119.39
119.25
119.16
117.76


OH
N

.90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


8.15
8.14
7.78
7.77
7.71
7.70
7.52
7.50
7.49
6.97
6.97
N
S
Cl
2.00
1.09
2.17
2.02
1.05
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
f1 (ppm)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8.00
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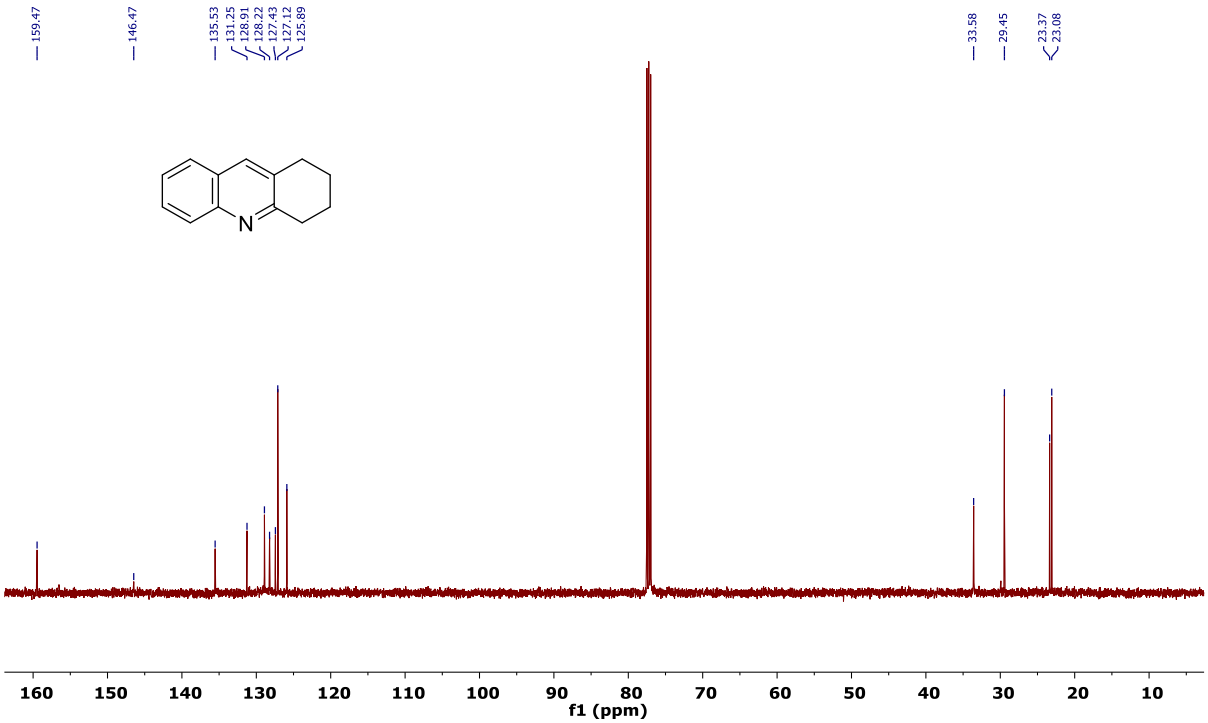
7.70
7.69
7.63
7.63
7.62
7.61
7.61
7.60
7.60
7.46
7.45
7.44
7.44
7.44
7.43
7.42
3.16
3.15
3.14
2.99
2.98
2.97
2.02
2.02
2.01
2.01
2.00
2.00
1.99
1.99
1.98
1.97
1.92
1.92
1.91
1.90
1.90
1.89
1.89
1.89
1.88
1.87
N
0.98
0.98
0.99
0.96
1.02
2.00
2.00
2.02
2.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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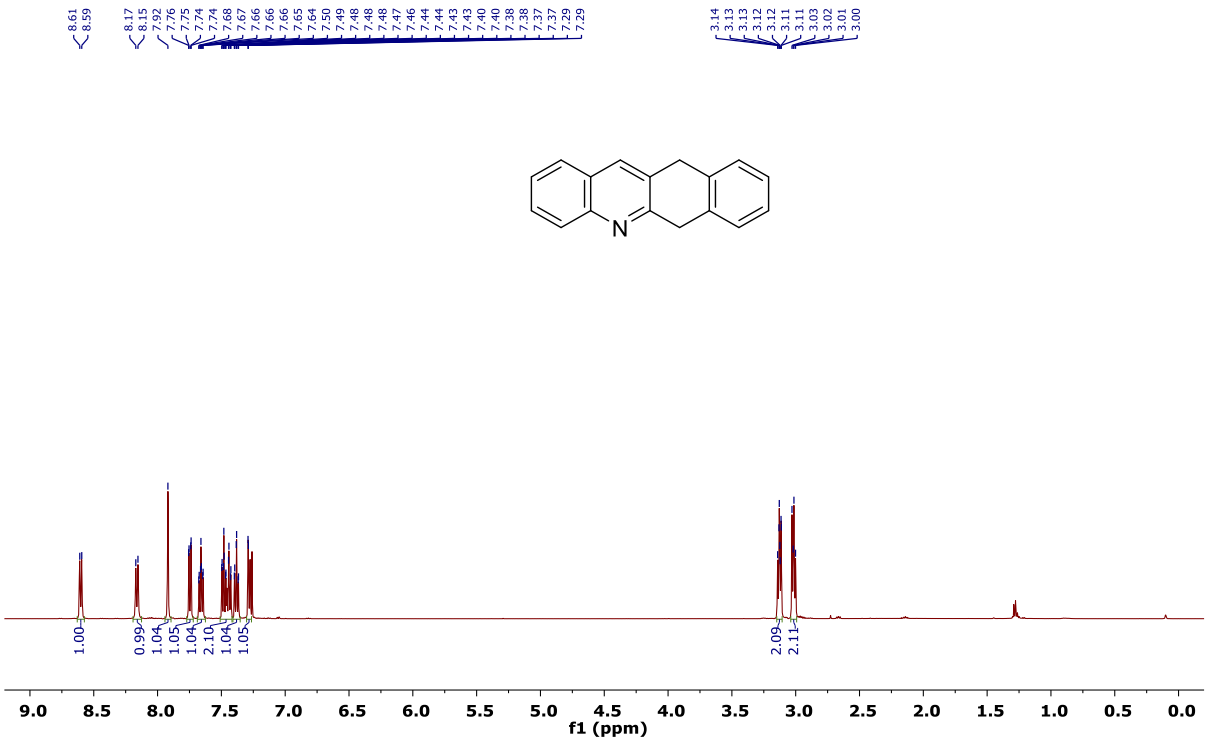
146.47
135.53
131.25
128.91
128.22
127.43
127.12
125.89
33.58
29.45
23.37
23.08
N
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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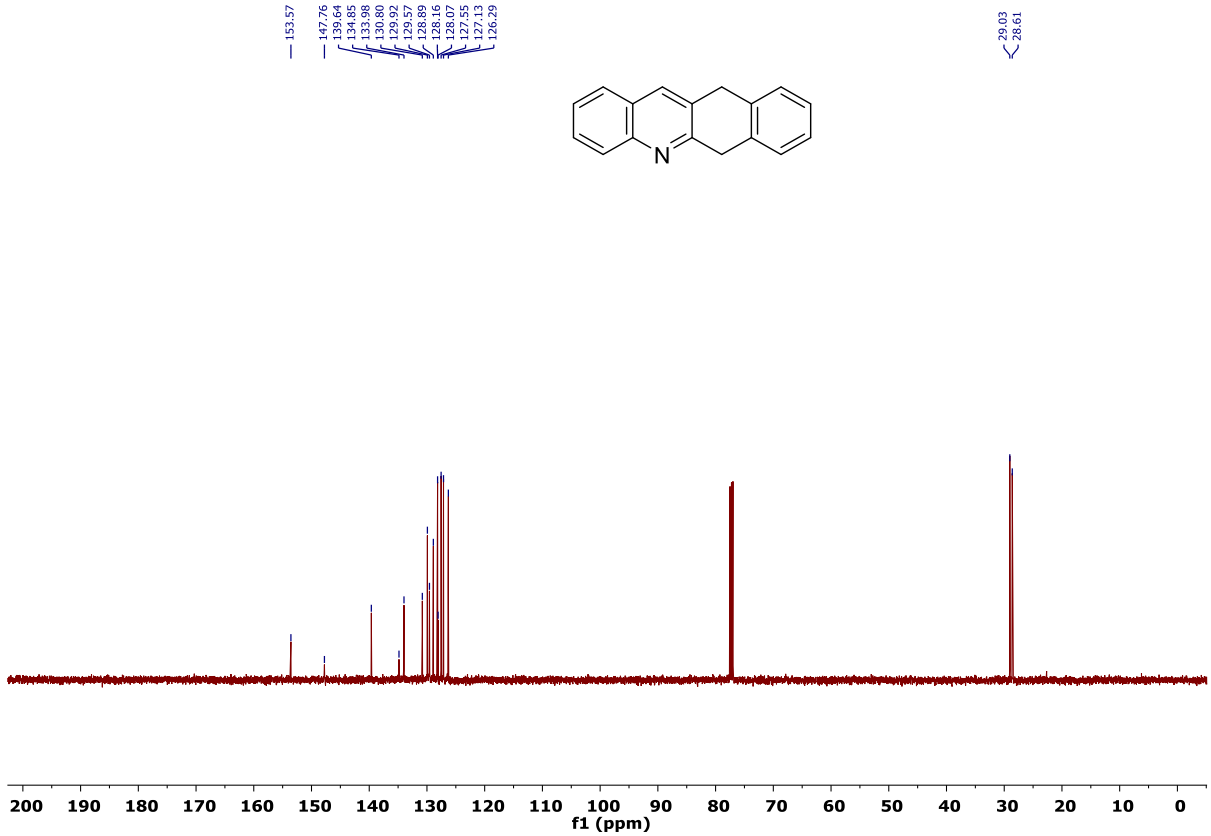
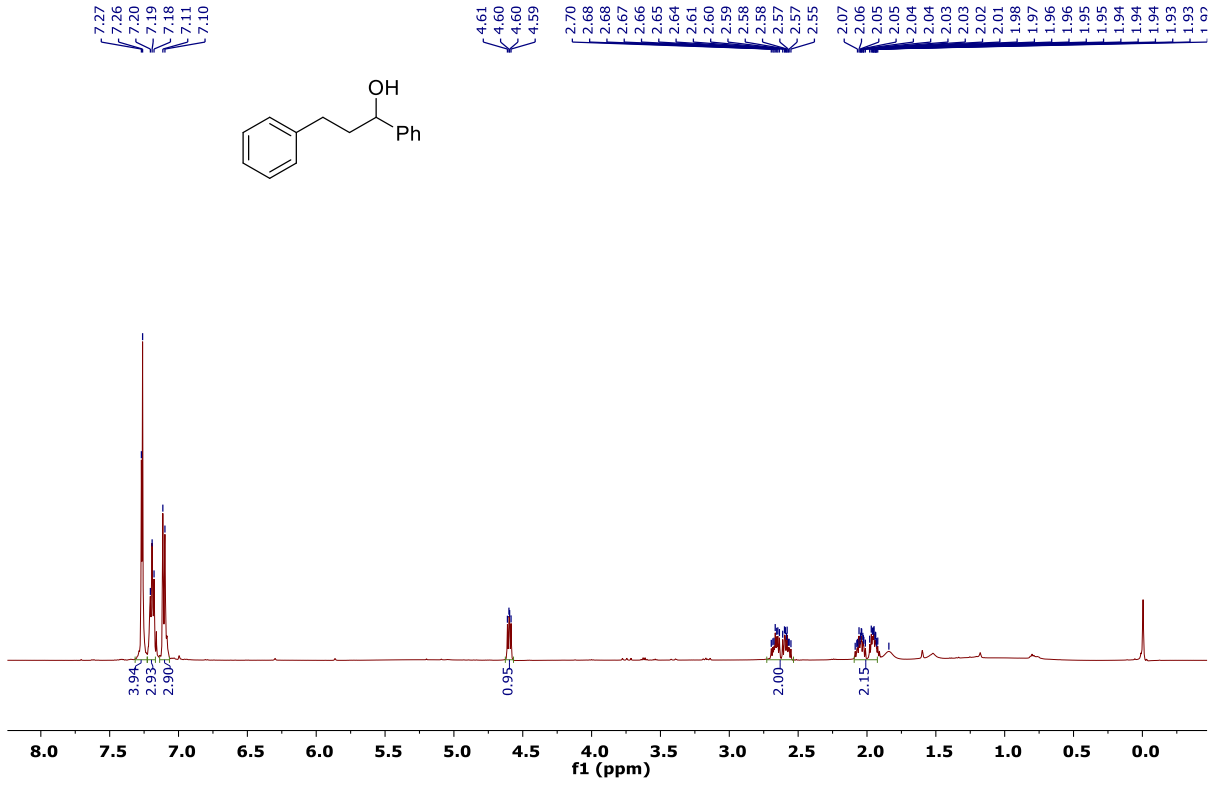
8.59
8.17
8.15
7.92
7.76
7.75
7.74
7.74
7.68
7.67
7.66
7.66
7.65
7.64
7.50
7.49
7.48
7.48
7.48
7.47
7.46
7.44
7.44
7.43
7.43
7.40
7.40
7.38
7.37
7.37
7.29
7.29
3.14
3.13
3.13
3.12
3.12
3.11
3.11
3.03
3.02
3.01
3.00
N
1.00
0.99
1.04
1.05
1.04
2.10
1.04
1.05
2.09
2.11
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


153.57
147.76
139.64
134.85
133.98
130.80
129.92
129.57
128.89
128.16
128.07
127.55
127.13
126.29
29.03
28.61
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.27
7.26
7.20
7.19
7.18
7.11
7.10
4.61
4.60
4.60
4.59
2.70
2.68
2.68
2.67
2.66
2.65
2.64
2.61
2.60
2.59
2.58
2.58
2.57
2.57
2.55
2.07
2.06
2.05
2.05
2.04
2.04
2.03
2.03
2.02
2.01
1.98
1.97
1.96
1.96
1.95
1.95
1.94
1.94
1.94
1.93
OH
Ph
3.94
2.93
2.90
0.95
2.00
2.15
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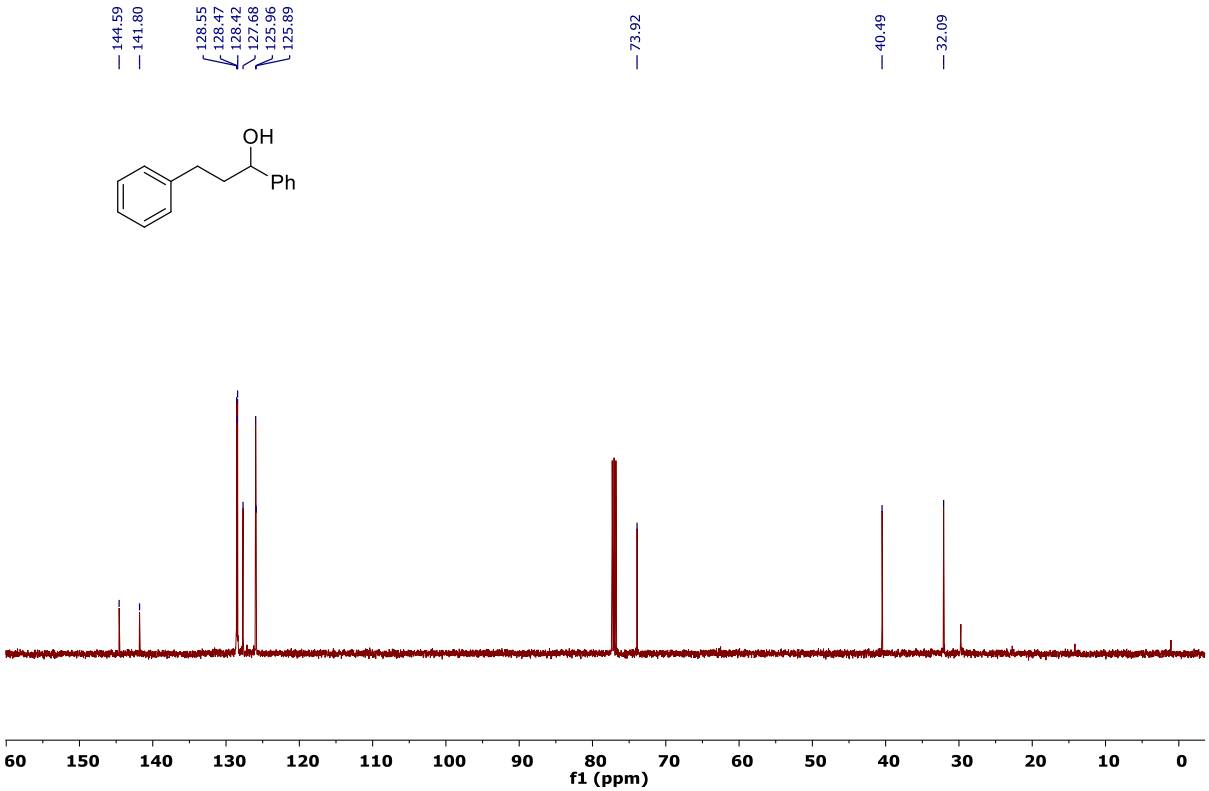
f1 (ppm)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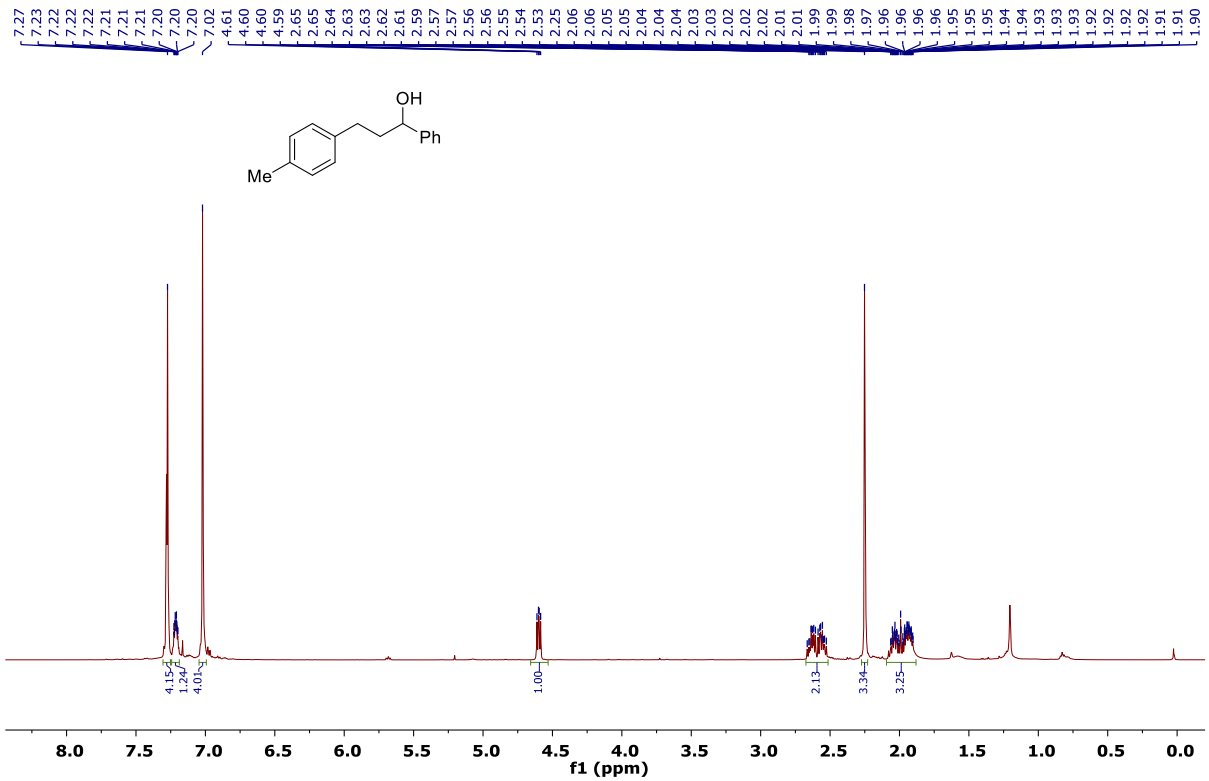
OH
Ph
f1 (ppm)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Ph
Me
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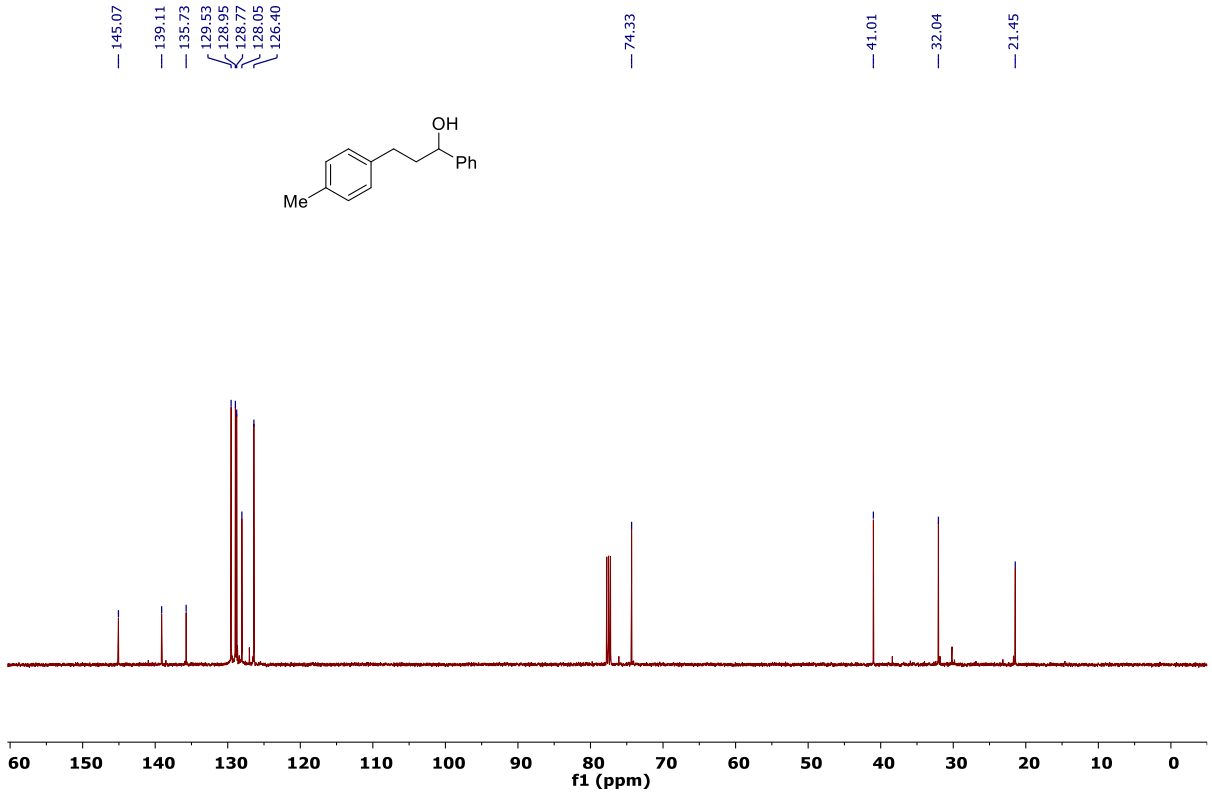
4.01
1.00
2.13
3.34
3.25
f1 (ppm)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41.01
32.04
21.45
OH
Ph
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110
100
90
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50
40
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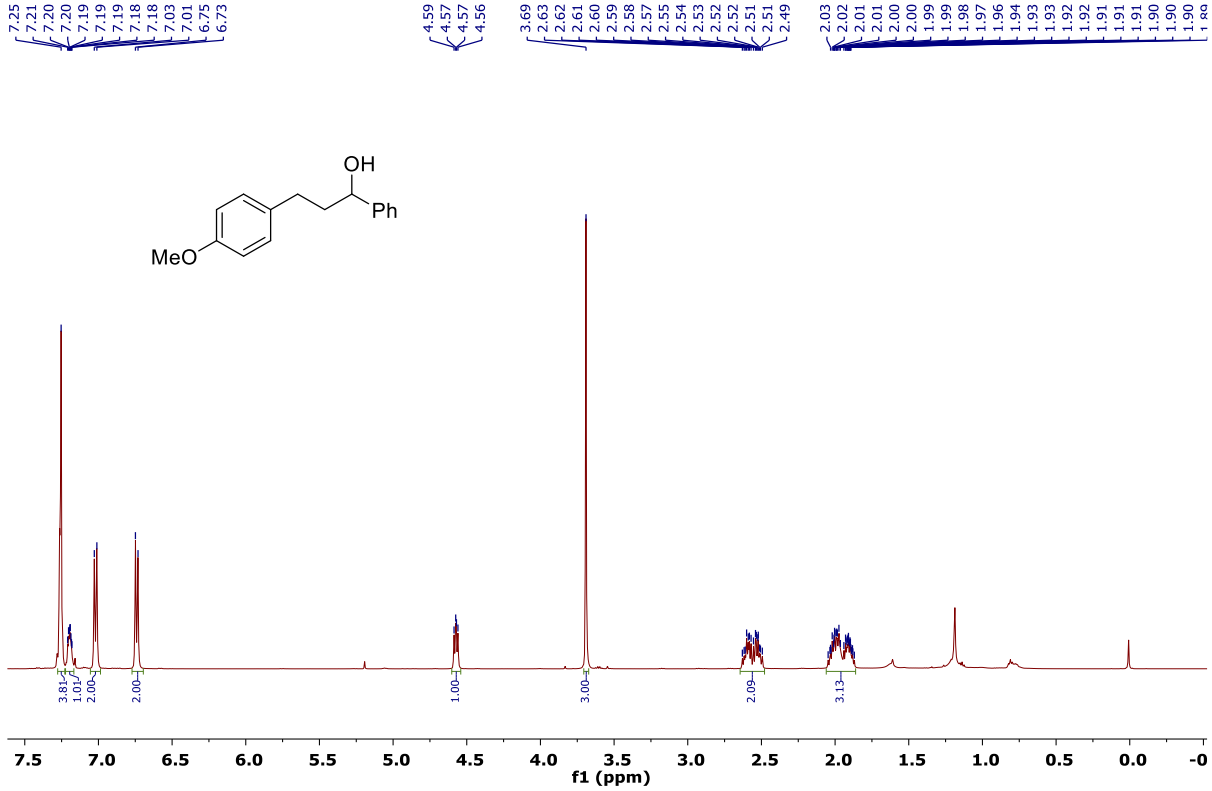
10
0
f1 (ppm)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7.18
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7.01
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6.73
4.59
4.57
4.57
4.56
3.69
2.63
2.62
2.61
2.60
2.59
2.58
2.57
2.55
2.54
2.53
2.52
2.52
2.51
2.51
2.49
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2.01
2.01
2.00
2.00
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1.96
1.94
1.93
1.93
1.92
1.92
1.91
1.91
1.91
1.90
1.90
1.90
OH
Ph
MeO
3.81
1.01
2.00
2.00
1.00
3.00
2.09
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
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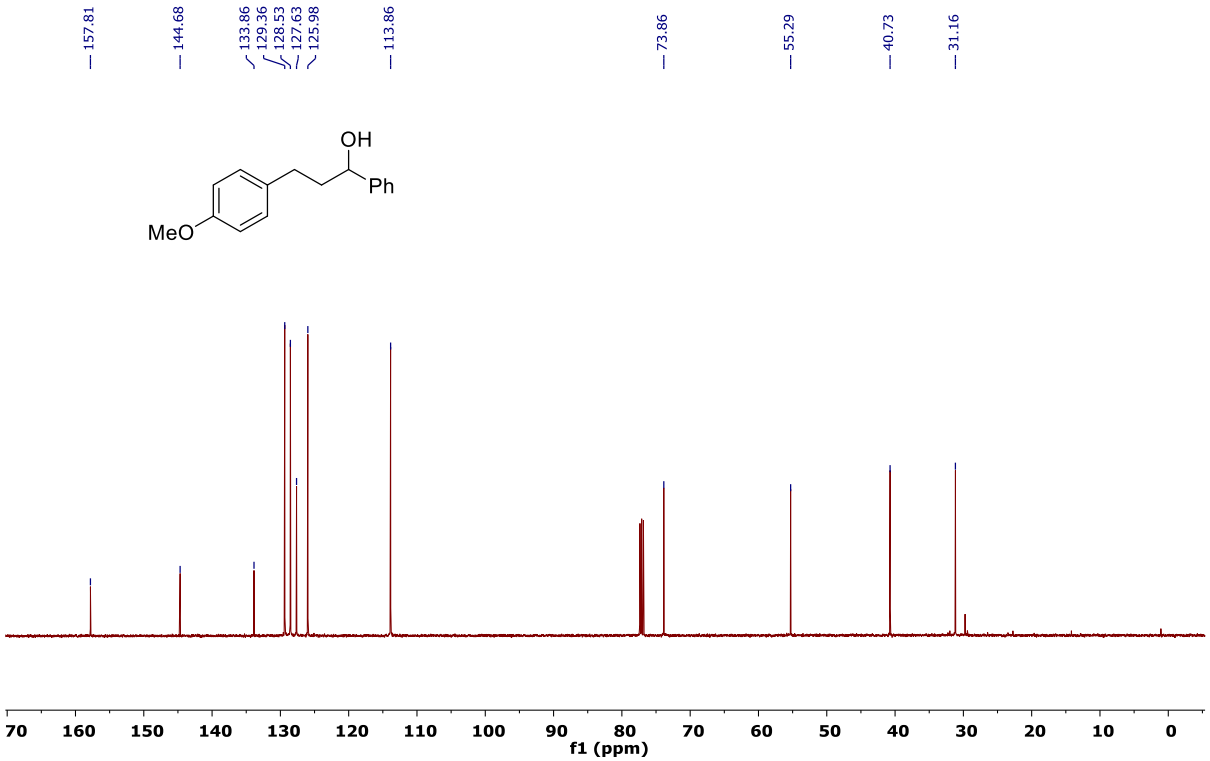
-0
f1 (ppm)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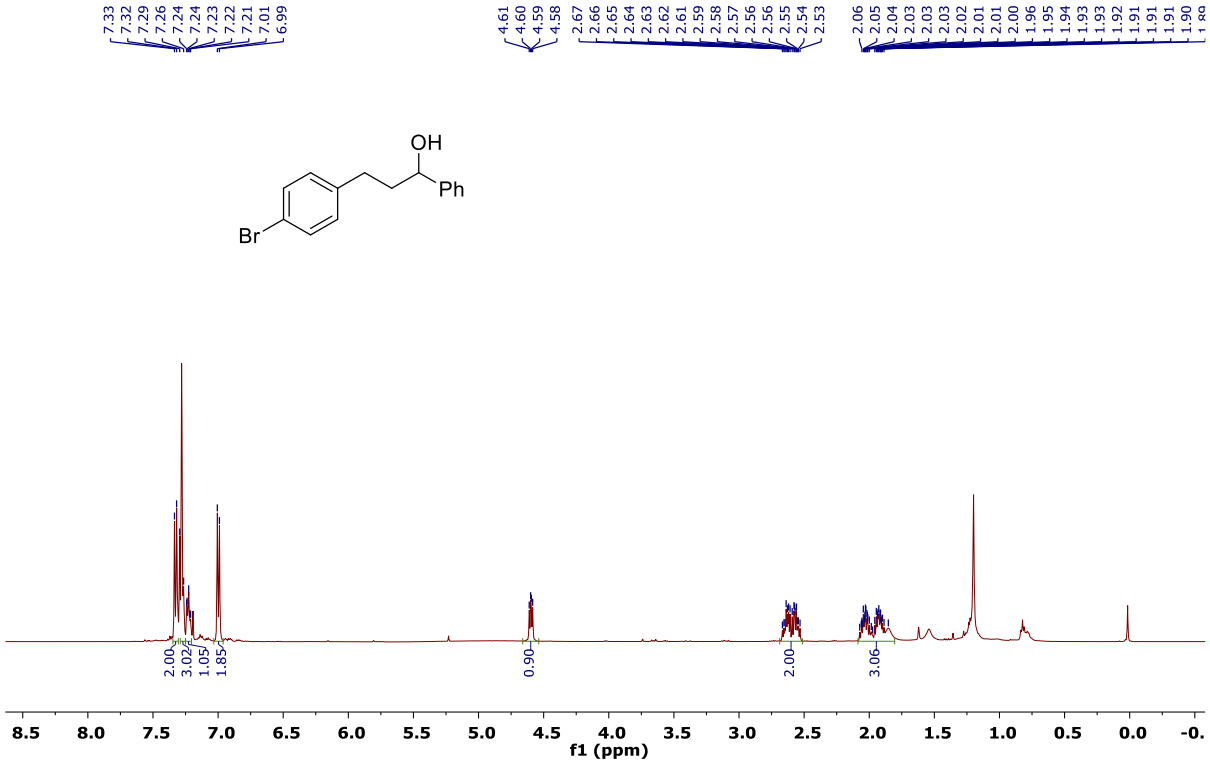
OH
Ph
MeO
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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4.59
4.58
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2.64
2.63
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2.59
2.58
2.57
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2.55
2.54
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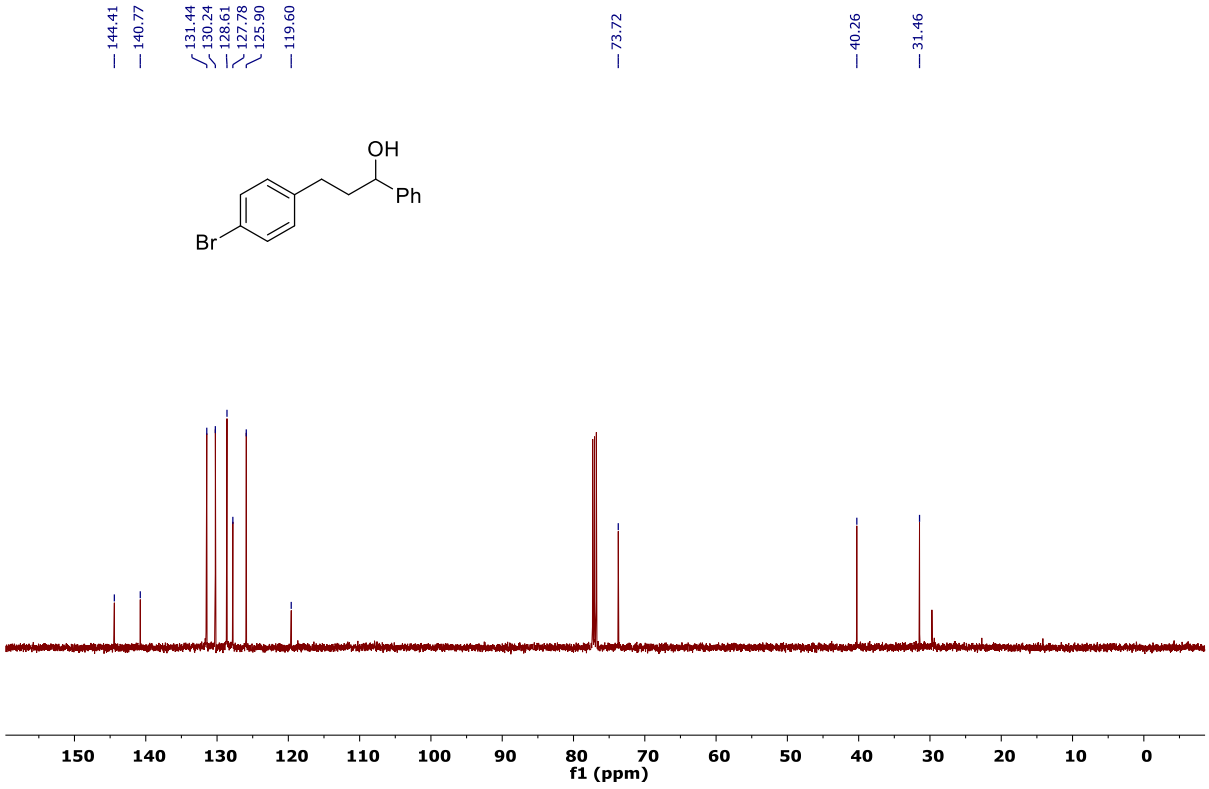
2.06
2.05
2.04
2.03
2.03
2.02
2.01
2.01
2.00
1.96
1.95
1.94
1.93
1.93
1.92
1.91
1.91
1.91
1.90
OH
Ph
Br
2.00
3.02
1.05
1.85
0.90
2.00
3.06
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)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127.78
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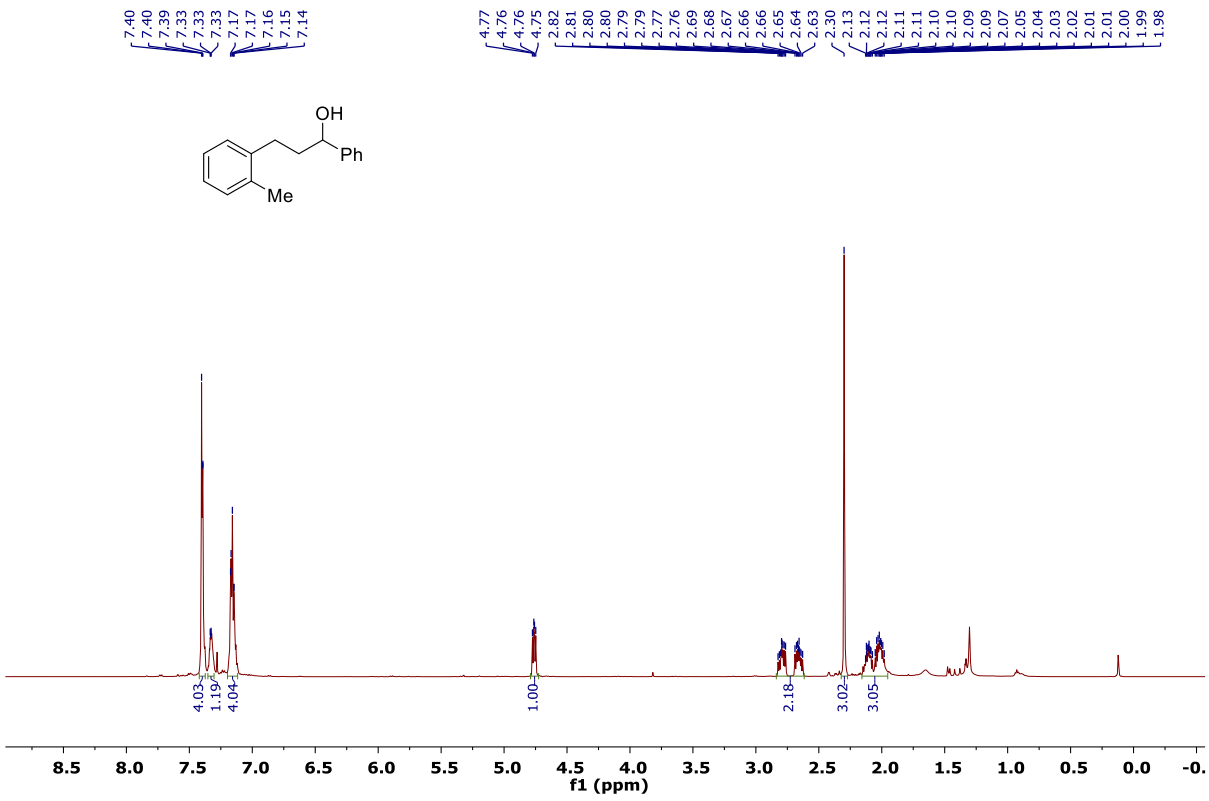
31.46
OH
Ph
Br
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.40
7.39
7.33
7.33
7.17
7.17
7.16
7.15
7.14
4.77
4.76
4.76
4.75
2.82
2.81
2.80
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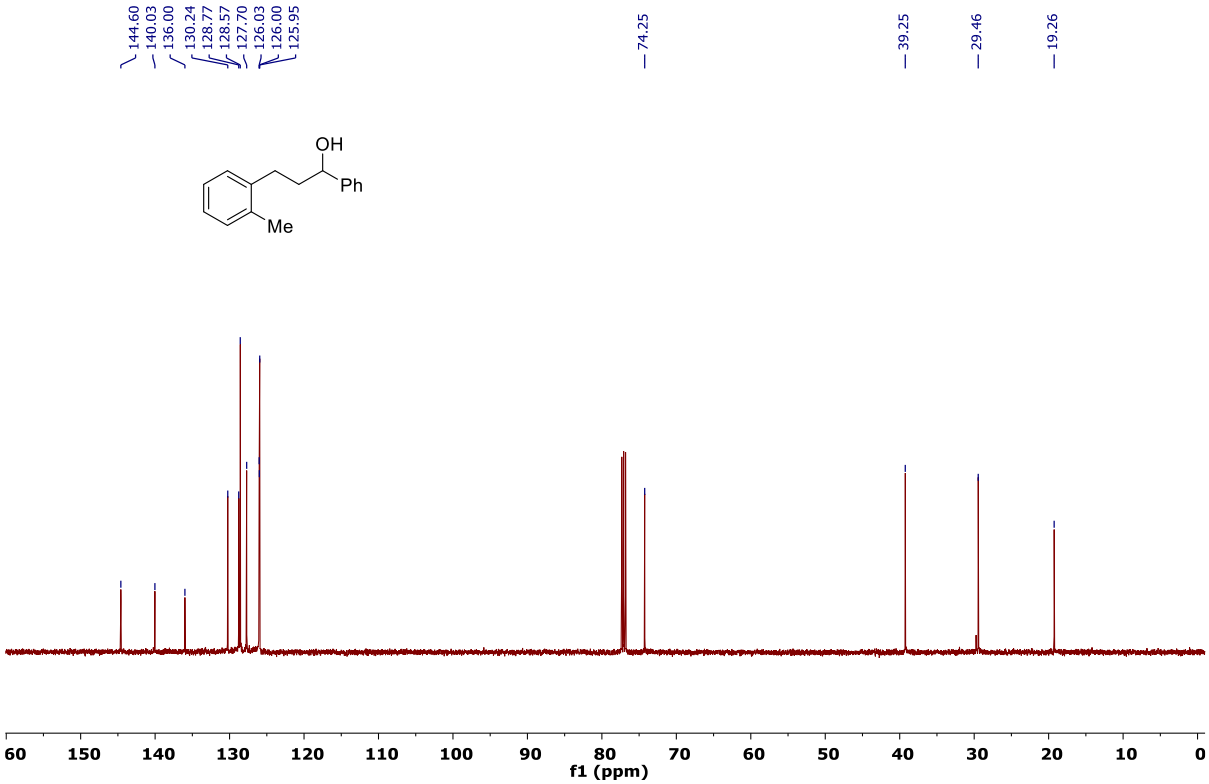
2.10
2.09
2.09
2.07
2.05
2.04
2.03
2.02
2.01
2.01
2.00
1.99
1.98
OH
Ph
Me
4.03
1.19
4.04
1.00
2.18
3.02
3.05
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)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140.03
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126.03
126.00
125.95
74.25
39.25
29.46
19.26
OH
Ph
Me
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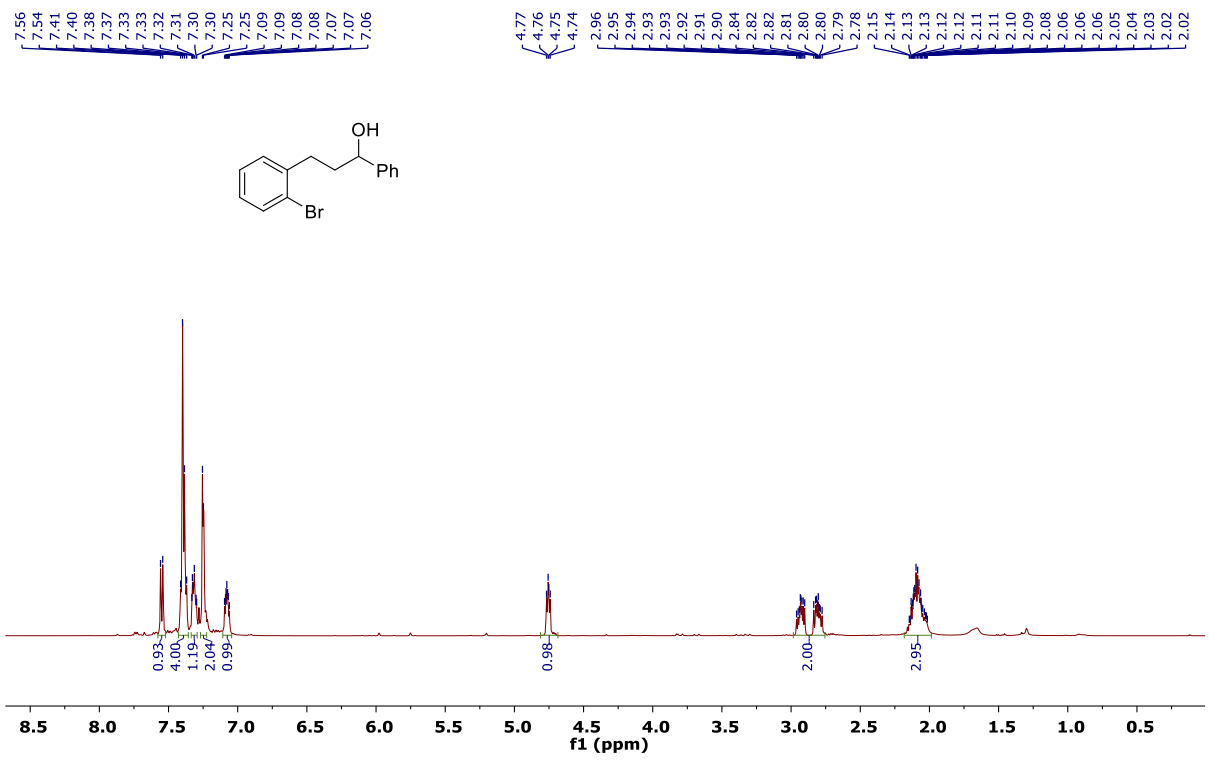
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.40
7.38
7.37
7.33
7.33
7.32
7.31
7.30
7.30
7.25
7.25
7.09
7.09
7.08
7.08
7.07
7.07
7.06
4.77
4.76
4.75
4.74
2.96
2.95
2.94
2.93
2.93
2.92
2.91
2.90
2.84
2.82
2.82
2.81
2.80
2.80
2.79
2.78
2.15
2.14
2.13
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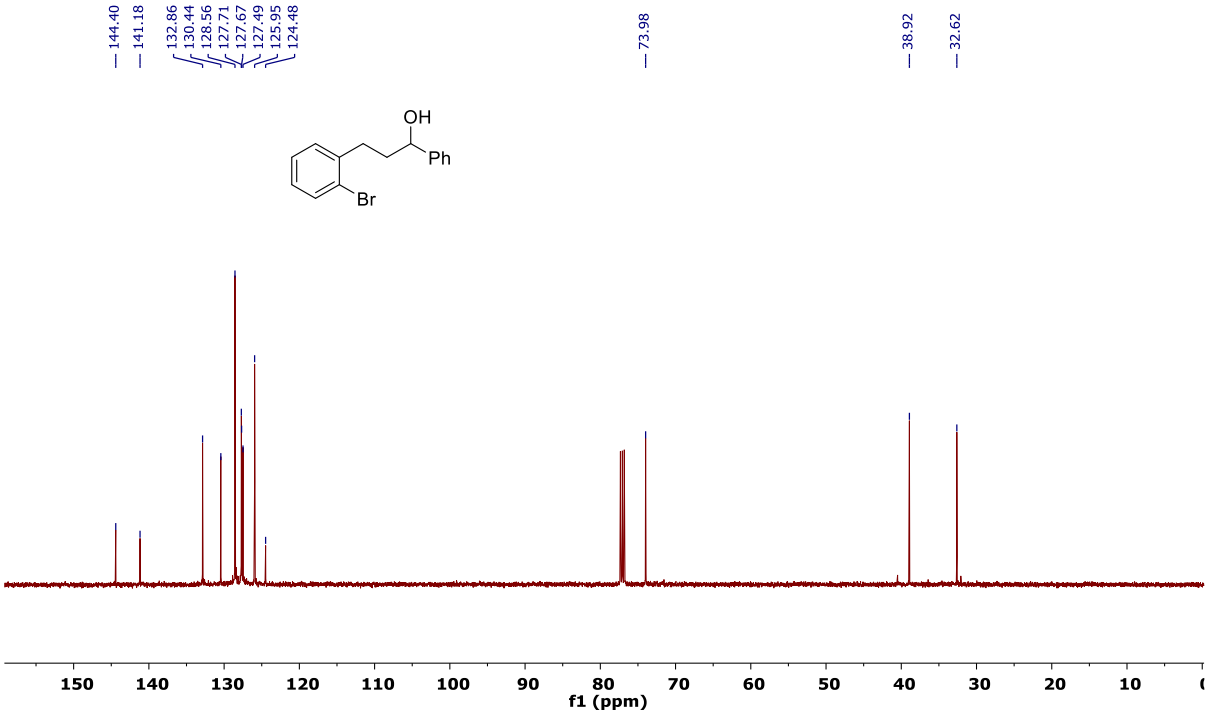
2.12
2.12
2.11
2.11
2.10
2.09
2.08
2.06
2.06
2.06
2.05
2.04
2.03
2.02
2.02
OH
Ph
Br
0.93
4.00
1.19
2.04
0.99
0.98
2.00
2.95
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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127.49
125.95
124.48
73.98
38.92
32.62
OH
Ph
Br
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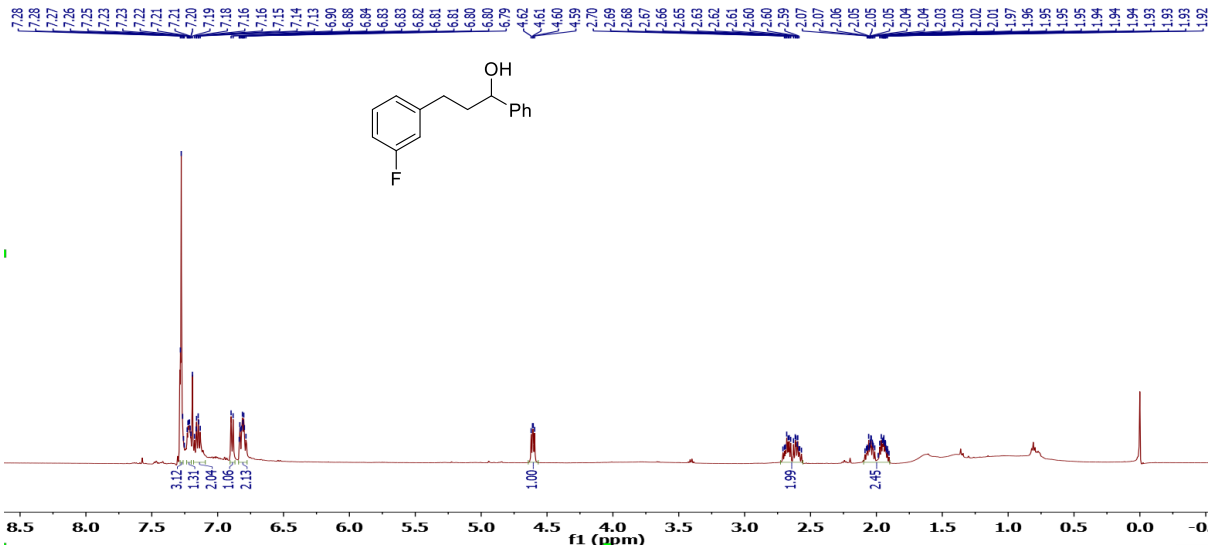
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


OH
Ph
F
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1.31
2.04
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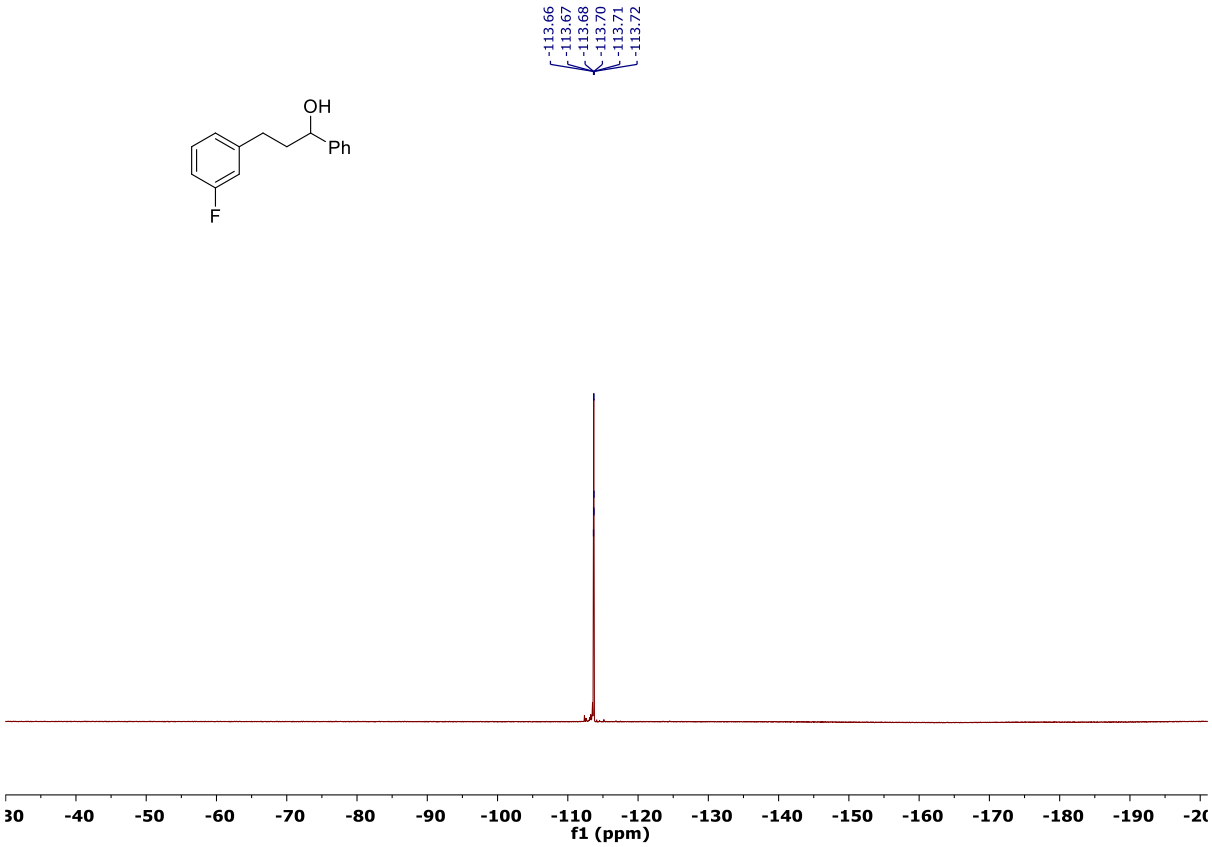
2.13
1.00
1.99
2.45
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

^{19}F NMR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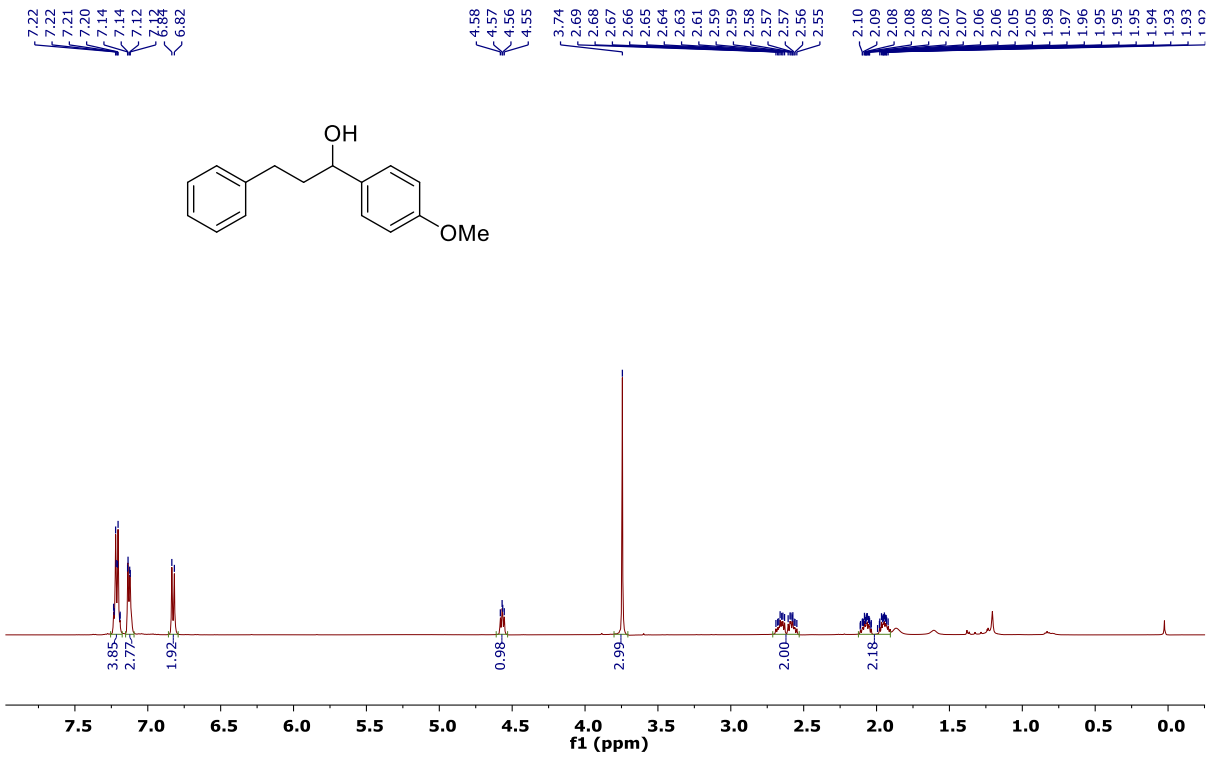
-113.68
-113.70
-113.71
-113.72
OH
Ph
F
f1 (ppm)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7.22
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7.14
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6.84
6.82
4.58
4.57
4.56
4.55
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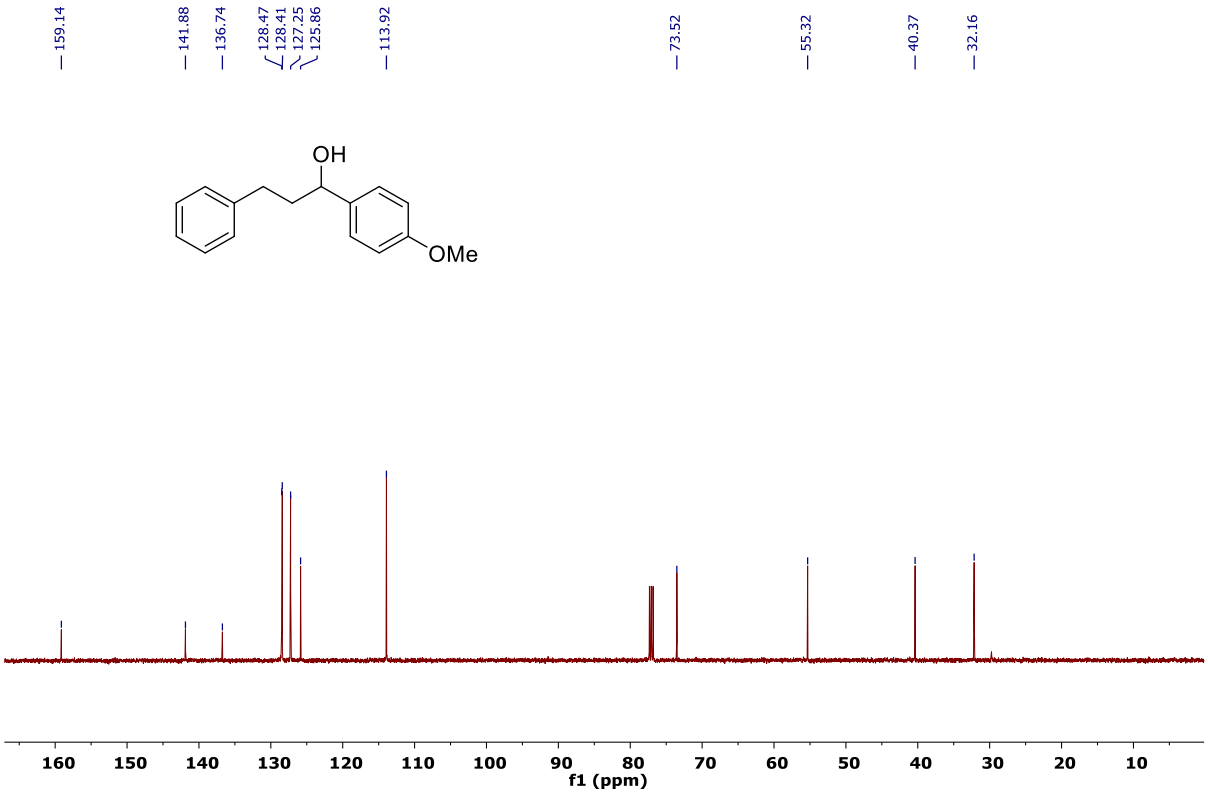
1.97
1.96
1.95
1.95
1.95
1.94
1.93
1.93
OH
OMe
3.85
2.77
1.92
0.98
2.99
2.00
2.18
f1 (ppm)


159.14
141.88
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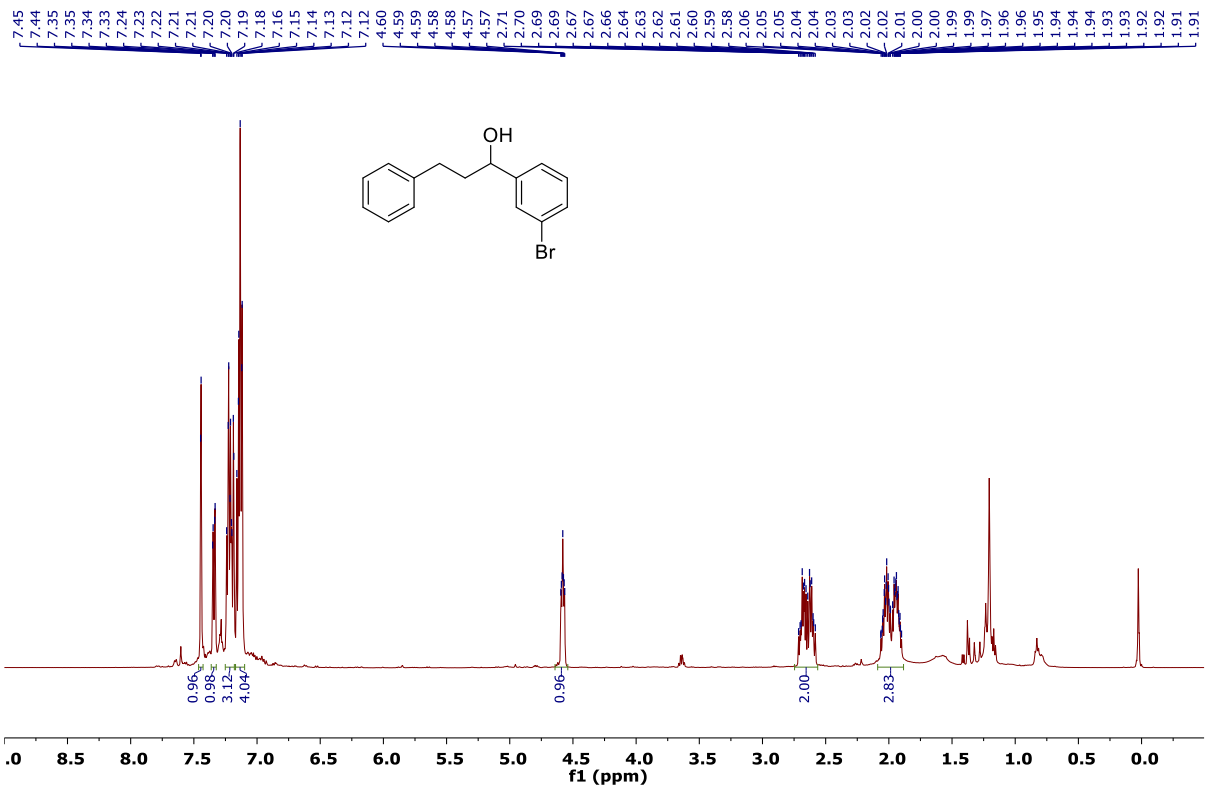
128.47
128.41
127.25
125.86
113.92
73.52
55.32
40.37
32.16
OH
OMe
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


OH
Br
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0.98
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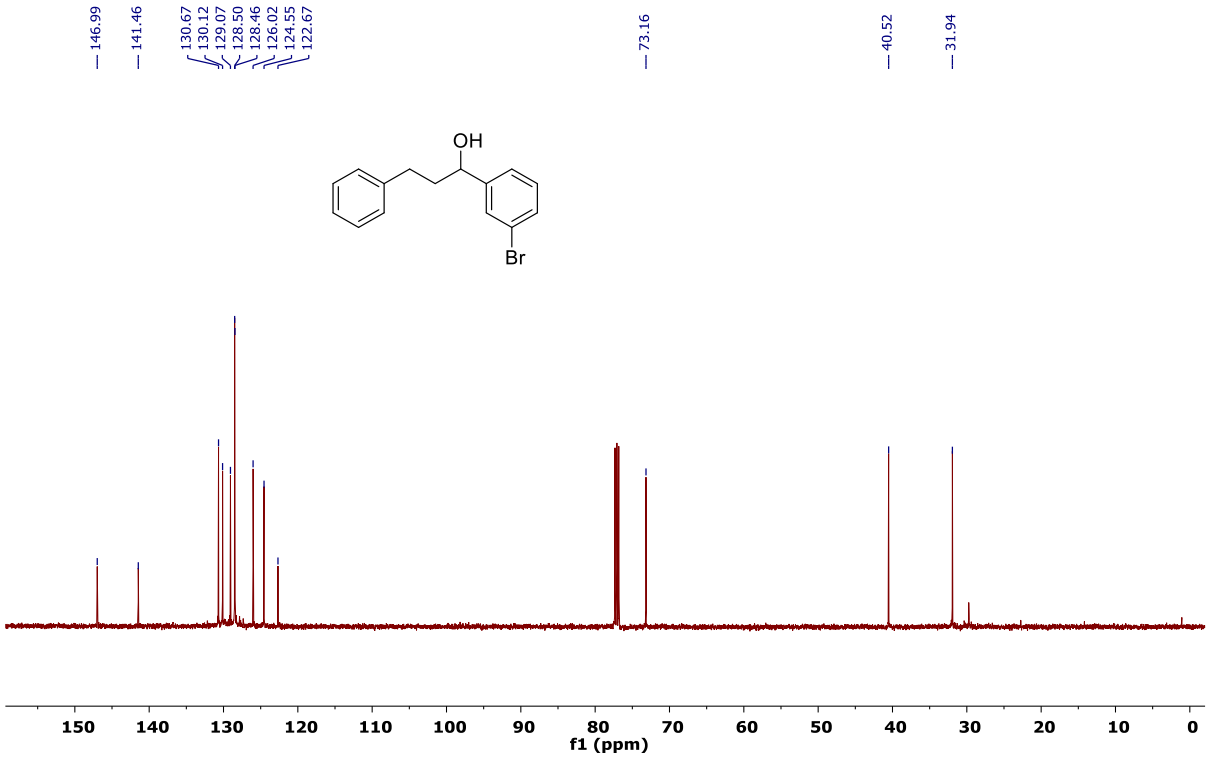
4.04
0.96
2.00
2.83
.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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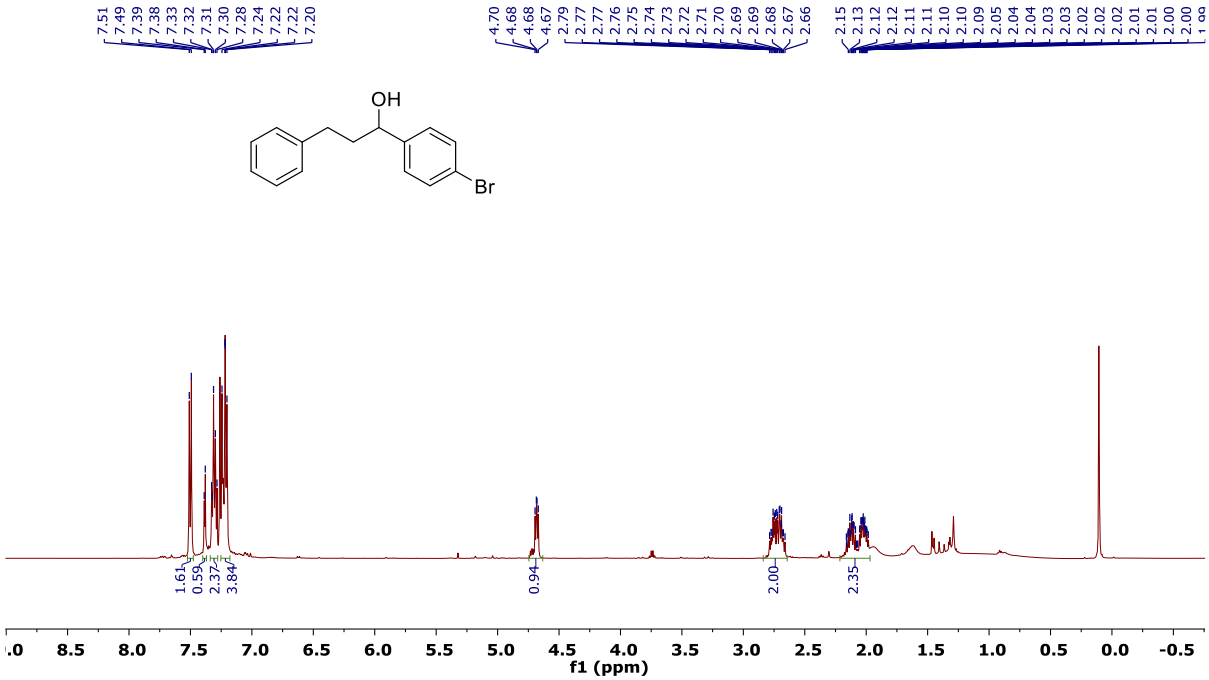
130.12
129.07
128.50
128.46
126.02
124.55
122.67
73.16
40.52
31.94
OH
Br
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.49
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7.32
7.31
7.30
7.28
7.24
7.22
7.22
7.20
4.70
4.68
4.68
4.67
2.79
2.77
2.77
2.76
2.75
2.74
2.73
2.72
2.71
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2.69
2.69
2.68
2.67
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2.12
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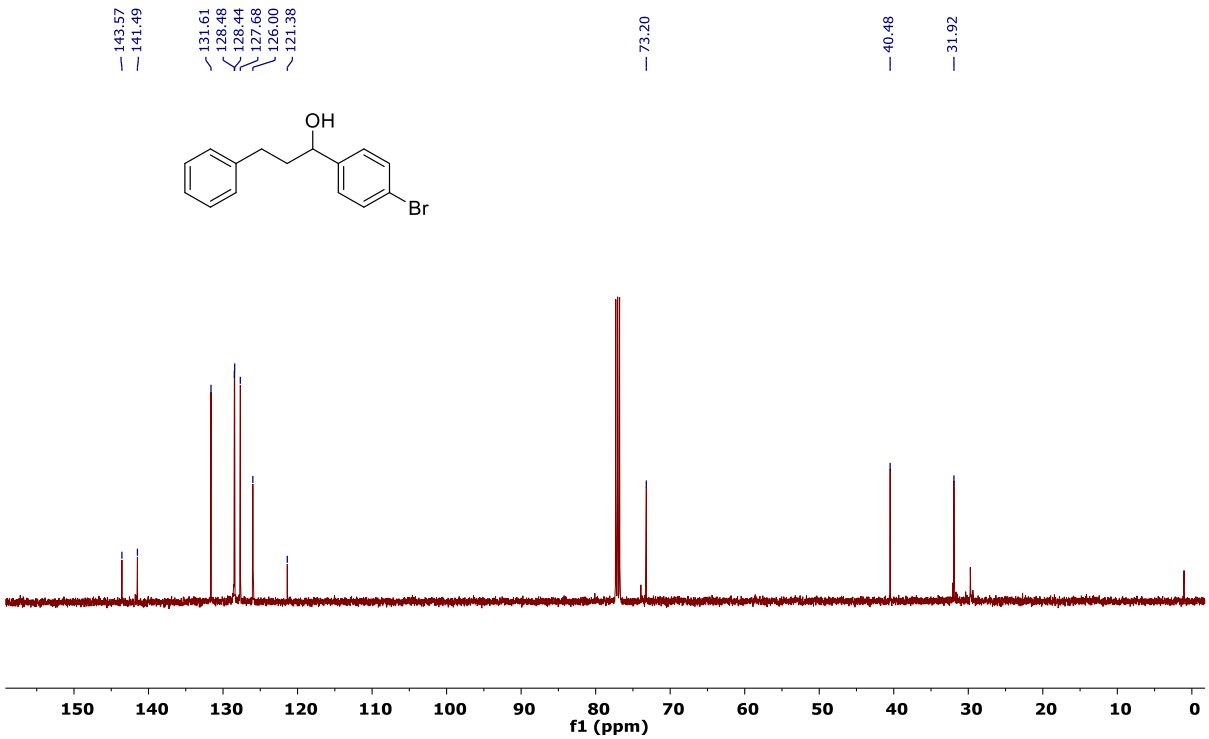
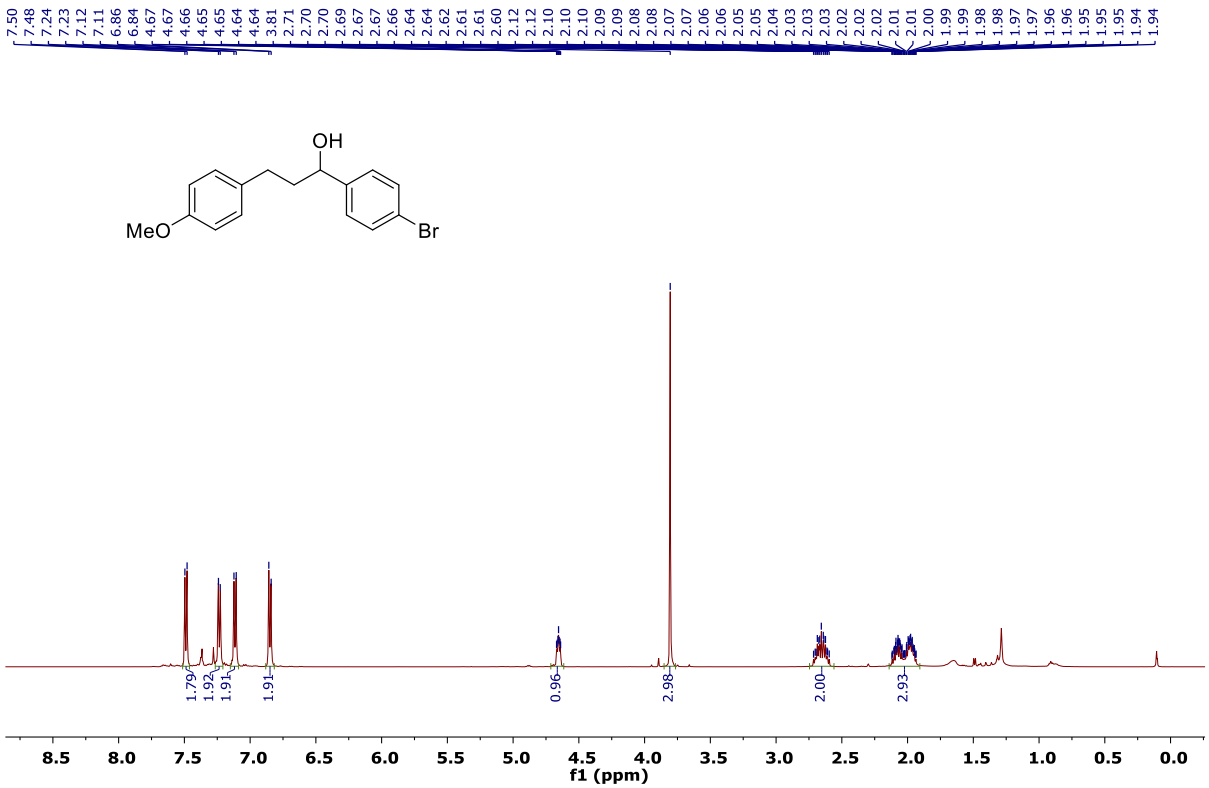
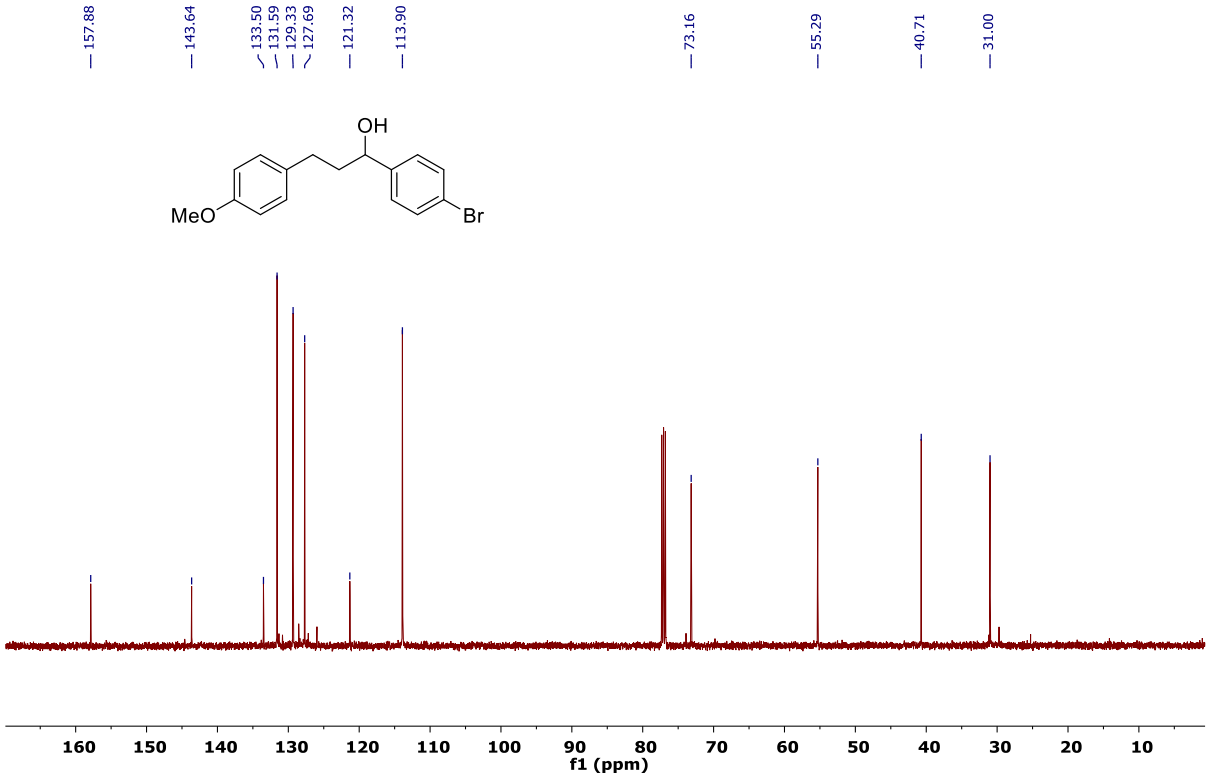
2.09
2.05
2.04
2.04
2.03
2.03
2.02
2.02
2.02
2.01
2.01
2.00
2.00
OH
Br
1.61
0.59
2.37
3.84
0.94
2.00
2.35
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


143.57
141.49
131.61
128.48
128.44
127.68
126.00
121.38
73.20
40.48
31.92
OH
Br
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


OH
MeO
Br
1.79
1.92
1.91
1.91
0.96
2.98
2.00
2.93
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)


157.88
143.64
133.50
131.59
129.33
127.69
121.32
113.90
73.16
55.29
40.71
31.00
OH
MeO
Br
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

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