Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Visible light mediated synthesis of 4-phenyl-1,2-dihydronaphthalene derivatives via single-electron oxidation or MHAT from methylenecyclopropanes

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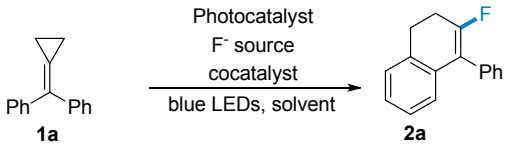
Contents

General remarks

^{1}H, ^{13}C and ^{19}F NMR spectra were recorded at 400 MHz, respectively. HRMS spectra were recorded by EI, ESI method. Infrared spectra were recorded on a Perkin-Elmer PE-983 spectrometer with absorption in cm^{-1}. Mass spectra were recorded by EI, ESI, and HRMS was measured on an Agilent Technologies 6224 TOF LC/MS instrument and a Waters Micromass GCT Permier. Melting points were determined on a digital melting point apparatus and temperatures were uncorrected. X-ray structure was determined on a Bruker Smart-1000 X-ray Diffraction meter. The employed solvents were dried up by standard methods when necessary. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel coated plates (Huanghai GF254). Flash column chromatography was performed by using 300-400 mesh silica gel eluting with ethyl acetate and petroleum ether at increased pressure.

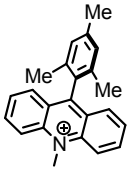
1. Optimization of reaction conditions

Table S1. Optimization of reaction conditions using **1a** as a template substrate to furnish **2a**.

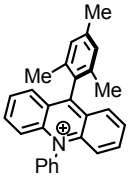


1a **2a**

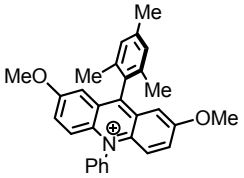
entry	photocatalyst	F⁻ source	cocatalyst	solvent	yield (%)
1	cat **I**	Et_3N•3HF	4,4'-dinitrodiphenyl disulfide	CH_3CN	21
2	cat **I**	Et_3N•3HF	$Co(dmgH)_2PyCl$	CH_3CN	44
3	cat **I**	CsF	$Co(dmgH)_2PyCl$	CH_3CN	-
4	cat **I**	TBAF	$Co(dmgH)_2PyCl$	CH_3CN	-
5	cat **I**	KHF_2	$Co(dmgH)_2PyCl$	CH_3CN	-
6[b]	cat **I**	Et_3N•3HF	$Co(dmgH)_2PyCl$	CH_3CN	55
7[b]	cat **I**	Et_3N•3HF	$Co(dmgH)_2PyCl$	DCM	27
8[b]	cat **I**	Et_3N•3HF	$Co(dmgH)_2PyCl$	1,4-Dioxane	14
9[b]	cat **I**	Et_3N•3HF	$Co(dmgH)_2PyCl$	Toluene	trace
10[b]	cat **I**	Et_3N•3HF	$Co(dmgH)_2Cl_2$	CH_3CN	-
11[b]	cat **I**	Et_3N•3HF	$Co(dmgH)_2(DMAP)Cl$	CH_3CN	34
12[b]	cat **II**	Et_3N•3HF	$Co(dmgH)_2PyCl$	CH_3CN	27
13[b]	cat **III**	Et_3N•3HF	$Co(dmgH)_2PyCl$	CH_3CN	8
14[b]	$Ir(dFCF_3ppy)_2(dtbbpy)PF_6$	Et_3N•3HF	$Co(dmgH)_2PyCl$	CH_3CN	-
15[c]	cat **I**	Et_3N•3HF	$Co(dmgH)_2PyCl$	CH_3CN	-
16[b]	cat **I**	Et_3N•3HF	-	CH_3CN	trace
17[d]	cat **I**	Et_3N•3HF	$Co(dmgH)_2PyCl$	CH_3CN	-



cat **I**



cat **II**

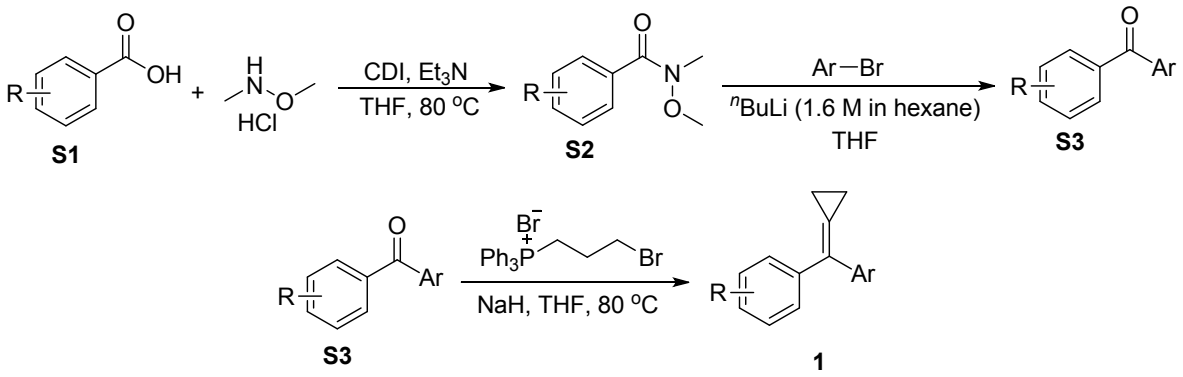


cat **III**

Initially, we utilized Mes-Arc$^+ClO_4^-$ as photocatalyst and 4,4'-dintriodiphenyl disulfide as co-catalyst to investigate the reactivity of MCP **1a** with 1.0 equiv of $Et_3N{\cdot}3HF$. The desired product **2a** was obtained in 21% yield under 12 W blue LEDs irradiation and its structure was confirmed by ^1H NMR, ^{13}C NMR, HRMS spectroscopic data. Then, the related reaction conditions were optimized with regard to catalyst, solvent, and the source of fluorine anion in a sequence to get the highest yield of **2a**.

To further increase the acidity of the solvent, the mixture of HFIP and CH_3CN was used as the solvent. In addition, 4Å molecular sieves were added to get rid of the impact of H_2O. In this case, we acquired **3a** in 76% yield unexpectedly.

2. General procedure for the preparation of 1[1]



General synthetic method for **1**:

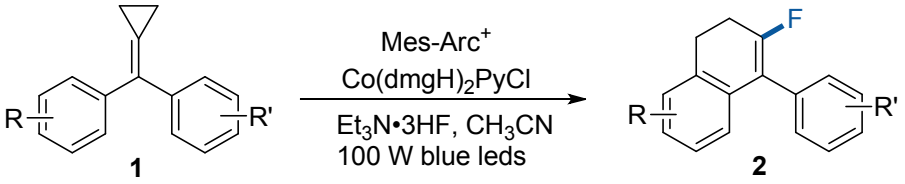
To a 250 mL round bottom flask were added anthranilic acid **S1** (1.0 equiv), 1,1'-carbonyldiimidazole (CDI) (1.0 equiv) and THF (1.0 mL/mol of anthranilic acid), and then the reaction mixture was heated to 70 °C for 0.5 hours. To another 50 mL round bottom flask were added *N*, *O*-dimethylhydroxylamine hydrochloride (1.0 equiv), Et_3N (1.0 equiv), and the reaction mixture was stirred for 10 min. Then, the solution of *N, O*-dimethylhydroxylamine hydrochloride in THF was added into the above THF solution of **S1** and CDI in a 250 mL round bottom flask. Next, the reaction mixture was stirred at 80 °C for 3 hours. After that, the mixture was filtered and extracted with CH_2Cl_2 for three times. The solvent was removed in vacuum and the residue was purified by a column chromatography to give the desired Weinreb amide **S2**.

To an oven dried 250 mL round bottom flask were added a solution of aryl bromide in anhydrous THF under inert atmosphere and then the reaction mixture was cooled to -78 °C. A solution of ^{n}BuLi in anhydrous hexane was added to the reaction mixture for 0.5 h at -78 °C. Subsequently, a solution of Weinreb amide **S2** in anhydrous THF was added to the mixture for 0.5 h at -78 °C. The reaction was quenched by addition of 5% HCl aqueous solution, extracted with ethyl acetate for three times. The organic extracts were dried over Na_2SO_4 and concentrated in vacuum. The product was purified by a column chromatography to give the desired benzophenones **S3**.

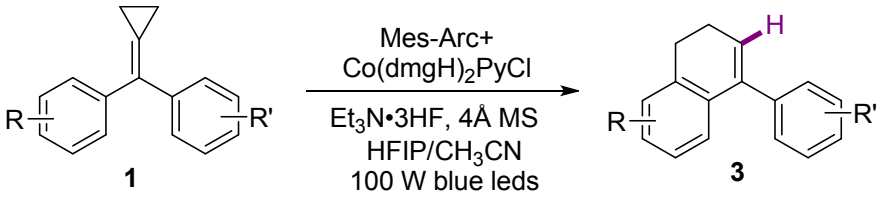
To an oven-dried round bottom flask was added sodium hydride (3.0 equiv) and (3-bromopropyl)triphenylphosphonium bromide (1.5 equiv), and then anhydrous THF (2.0 mL/mol of NaH) was added under inert atmosphere. The reaction mixture was stirred for 2 hours under reflux. Then, a solution of benzophenone **S3** (1.0 equiv) in THF (1.0 mL/mol) was added to the reaction mixture and the resulting mixture was stirred for another 3 hours. The reaction mixture was subsequently cooled to room temperature and filtered through a Celite and washed with petroleum

ether for three times. Next, the solvent was removed under vacuum and the residue was purified by a column chromatography to give methylenecyclopropanes **1**.

3. General procedure for the preparation of 2 & 3.



Reactions were carried out with **1** (0.2 mmol), photocatalyst (3 mol%), Co catalyst (3 mol%) in solvents (2 mL) at ambient temperature using 100 W blue light irradiation for 12 hours. The product was purified by a column chromatography to give the desired products **2**. Yields were determined by isolated product.



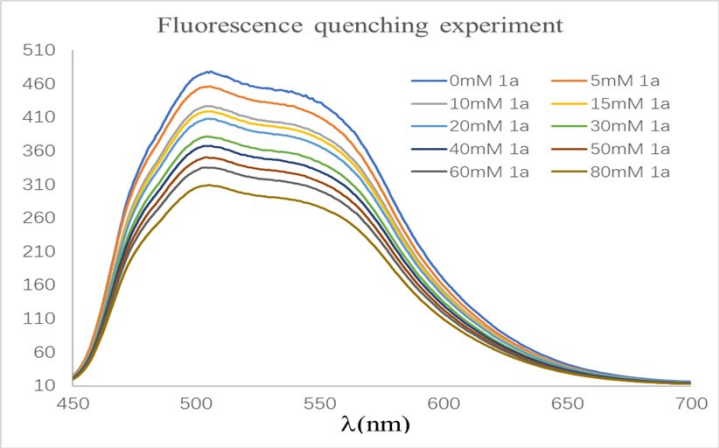
Reactions were carried out with oven-dried 4Å MS 50 mg, **1a** (0.2 mmol), photocatalyst (3 mol%), Co catalyst (3 mol%) in 2 mL solvents (HFIP/CH_3CN = 4:1) at ambient temperature using 100 W blue light irradiation for 12 hours. The mixture was filtered through a Celite and washed with petroleum ether. Next, the solvent was removed under vacuum and the residue was purified by a column chromatography to give **3**. Yields were determined by isolated product.

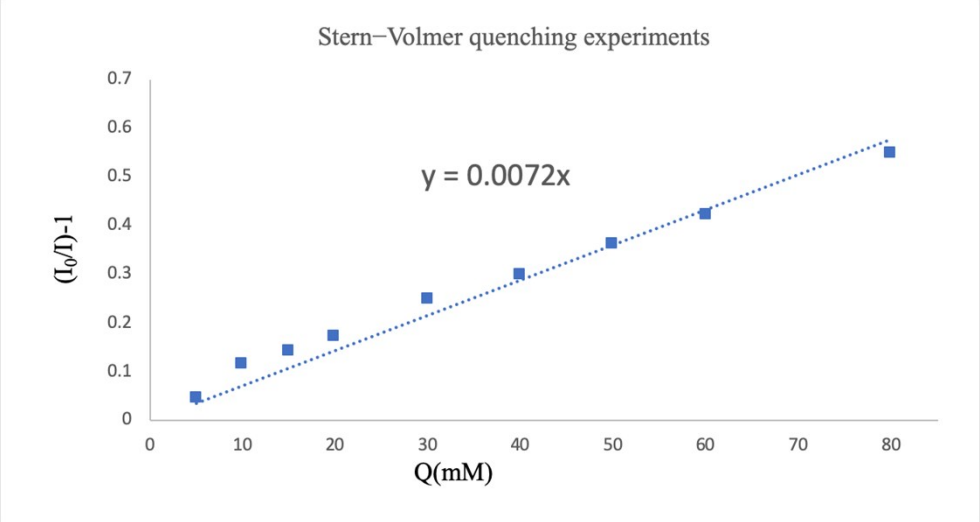
The above reaction mixtures were stirred and irradiated with a Kelo 100 W blue LED lamp (5 cm away, with cooling fan to keep the reaction temperature no more than 30 °C) for 12 hours.

5. Luminescence Quenching Experiments (Stern-Volmer Studies)

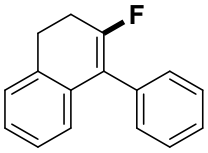
Emission intensities were recorded using a Hitachi F-2700 fluorescence spectrophotometer with a 5 nm band width. Solutions of different concentration of **1a** were prepared and introduced to a 1 cm path length quartz cuvette equipped with a Teflon® septum under Ar atmosphere. In a typical experiment, to a 0.005 M solution of photocatalyst in MeCN was added the appropriate amount **1a** in screw-top quartz cuvette under Ar atmosphere and the emission of the sample was collected.

Photocatalyst was excited at 430 nm and the emission intensity was collected at 440 nm. The solutions mixtures were stirred for 30s in dark, then filtered to 1.0 cm path length quartz cuvettes, sealed with Teflon® septum.

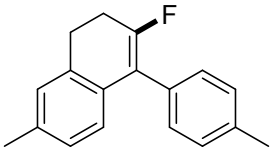




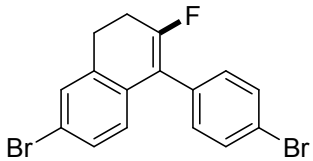
6. Spectroscopy of compounds 2



Compound 2a: colorless oil (24.6 mg, 55%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.42 (t, *J* = 7.6 Hz, 2H), 7.38-7.26 (m, 3H), 7.16 (d, *J* = 7.0 Hz, 1H), 7.14-7.03 (m, 2H), 6.81 (d, *J* = 7.5 Hz, 1H), 3.08 (td, *J* = 8.4, 2.4 Hz, 2H), 2.70 (td, *J* = 8.2, 5.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0 (d, *J* = 267.3 Hz), 135.3 (d, *J* = 5.4 Hz), 133.7, 132.9, 130.2, 128.3, 128.2 (d, *J* = 4.7 Hz), 127.3 (d, *J* = 15.9 Hz), 126.5, 126.2 (d, *J* = 2.1 Hz), 125.3 (d, *J* = 6.8 Hz), 117.3 (d, *J* = 13.2 Hz), 28.7, 25.2 (d, *J* = 24.4 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ -102.2. IR (neat) ν 3058, 2943, 1671, 1483, 1151, 941, 764, 753 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{13}F$ requires $[M]^+$: 224.0996, Found: 224.0995.

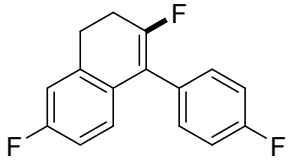


Compound 2b: colorless oil (26.7 mg, 53%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.24-7.17 (m, 4H), 6.98 (s, 1H), 6.87 (d, *J* = 7.2 Hz, 1H), 6.71 (d, *J* = 7.7 Hz, 1H), 3.03 (td, *J* = 8.0, 2.0 Hz, 3H), 2.66 (td, *J* = 8.1, 5.6 Hz, 3H), 2.39 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3 (d, *J* = 265.6 Hz), 137.0, 135.8 (d, *J* = 2.4 Hz), 132.8, 132.6 (d, *J* = 5.4 Hz), 130.7 (d, *J* = 1.0 Hz), 130.0 (d, *J* = 1.8 Hz), 129.0, 128.2, 127.0, 125.3 (d, *J* = 6.8 Hz), 116.9 (d, *J* = 13.2 Hz), 28.8 (d, *J* = 6.7 Hz), 25.3 (d, *J* = 24.7 Hz), 21.3, 21.0. ^{19}F NMR (376 MHz, $CDCl_3$) δ -103.8. IR (neat) ν 3024, 2921, 1668, 1512, 1359, 1114, 934, 812, 742 cm^{-1}. HRMS (EI) Calcd. for $C_{18}H_{17}F$ requires $[M]^+$: 252.1309, Found: 252.1307.

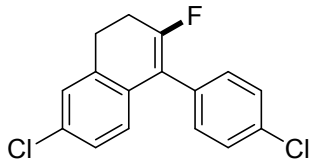


Compound 2c: colorless oil (30.6 mg, 40%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.59-7.52 (m, 2H), 7.30 (d, *J* = 2.1 Hz, 1H), 7.22-7.13 (m, 3H), 6.64 (d, *J* = 8.4 Hz, 1H), 3.04 (td, *J* = 7.7, 2.4 Hz, 2H), 2.67 (td, *J* = 8.1, 5.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.3 (d, *J* = 269.7 Hz), 135.0, 133.8 (d, *J* = 5.4 Hz), 132.0, 131.8 (d, *J* = 1.9 Hz), 131.7, 130.3, 129.5, 126.6 (d, *J* = 6.9 Hz), 121.7, 119.9 (d, *J* = 2.8 Hz), 115.9 (d, *J* = 13.5 Hz), 28.3 (d, *J* = 6.6 Hz), 25.0 (d, *J* = 24.5 Hz). ^{19}F NMR (376 MHz,

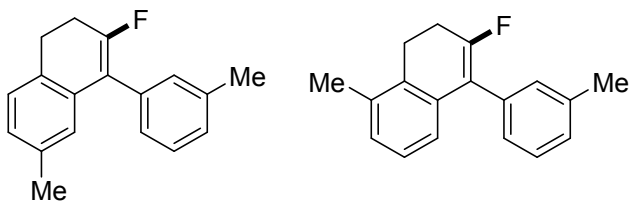
$CDCl_3$) δ -100.5. IR (neat) ν 2922, 2841, 1660, 1483, 1228, 929, 876, 824, 806 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{11}Br_2F$ requires $[M]^+$: 379.9206, Found: 379.9203.



Compound 2d: colorless oil (24.5 mg, 47%). ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.30-7.21 (m, 2H), 7.11 (t, *J* = 8.5 Hz, 2H), 6.88 (dd, *J* = 8.7, 1.6 Hz, 1H), 6.82-6.66 (m, 2H), 3.08-2.99 (m, 2H), 2.68 (td, *J* = 8.1, 5.9 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2 (d, *J* = 245.1 Hz), 161.3 (dd, *J* = 244.6, 2.5 Hz), 157.5 (dd, *J* = 266.9, 2.1 Hz), 135.2 (d, *J* = 7.6 Hz), 131.8 (dd, *J* = 8.0, 1.9 Hz), 131.1 (dd, *J* = 5.3, 3.2 Hz), 129.3 (dd, *J* = 3.5, 1.0 Hz), 126.5 (dd, *J* = 7.8, 7.1 Hz), 115.7 (d, *J* = 13.7 Hz), 115.4 (d, *J* = 21.2 Hz), 114.6 (d, *J* = 22.0 Hz), 112.9 (d, *J* = 21.1 Hz), 28.6 (dd, *J* = 6.7, 1.6 Hz), 24.9 (d, *J* = 24.9 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ -103.3, -114.5, -116.4. IR (neat) ν 3040, 2951, 1671, 1494, 1245, 1222, 1097, 830, 800 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{11}F_3$ requires $[M]^+$: 260.0807, Found: 260.0808.

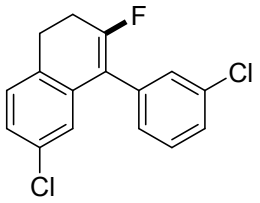


Compound 2e: colorless oil (26.0 mg, 44%). ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.39 (d, *J* = 8.3 Hz, 2H), 7.23 (t, *J* = 6.9 Hz, 2H), 7.17-7.10 (m, 1H), 7.06-6.98 (m, 1H), 6.69 (d, *J* = 8.3 Hz, 1H), 3.03 (dt, *J* = 8.5, 4.2 Hz, 2H), 2.67 (td, *J* = 8.2, 5.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.3 (d, *J* = 269.2 Hz), 134.7, 133.5, 133.4 (d, *J* = 5.3 Hz), 131.8 (d, *J* = 2.8 Hz), 131.6 (d, *J* = 0.8 Hz), 131.4 (d, *J* = 2.0 Hz), 128.7, 127.5, 126.5, 126.3 (d, *J* = 7.0 Hz), 115.8 (d, *J* = 13.5 Hz), 28.4 (d, *J* = 6.7 Hz), 25.0 (d, *J* = 24.6 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ -100.9. IR (neat) ν 3026, 2961, 1667, 1438, 1087, 999, 930, 825, 812, 723 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{11}Cl_2F$ requires $[M]^+$: 292.0216, Found: 292.0220.

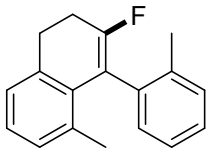


Compound 2f & 2f': (4 : 1), colorless oil (27.3 g, 54%). ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.32 (t, *J* = 7.4 Hz, 1H), 7.21-6.90 (m, 2H), 6.68-6.65 (m, 1H), 3.06-2.96 (m, 1H), 2.73-2.62 (m, 1H), 2.40-

2.35 (m, 1H). 2.33 (s, 2.4H), 2.18 (s, 0.6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0 (d, *J* = 266.7 Hz), 157.4 (d, *J* = 266.0 Hz), 137.9, 137.8, 136.0, 135.4 (d, *J* = 5.3 Hz), 135.2 (d, *J* = 5.3 Hz), 134.7, 133.9, 133.6, 131.0, 130.84, 130.82, 129.8, 128.3 (d, *J* = 1.8 Hz), 128.2, 128.14, 128.10, 127.25 (d, *J* = 1.6 Hz), 127.19 (d, *J* = 1.8 Hz), 127.1, 126.7 (d, *J* = 2.1 Hz), 126.1, 126.01, 125.98, 123.6 (d, *J* = 6.7 Hz), 117.3 (d, *J* = 13.0 Hz), 28.3 (d, *J* = 6.7 Hz), 25.4 (d, *J* = 24.7 Hz), 24.8 (d, *J* = 24.1 Hz), 24.7 (d, *J* = 7.9 Hz), 21.5, 21.4, 21.1, 19.8. ^{19}F NMR (376 MHz, $CDCl_3$) δ -102.1, -102.7. IR (neat) ν 3037, 2919, 1601, 1513, 1409, 1230, 974, 802, 189, 715 cm^{-1}. HRMS (EI) Calcd. for $C_{18}H_{17}F$ requires $[M]^+$: 252.1309, Found: 252.1307.

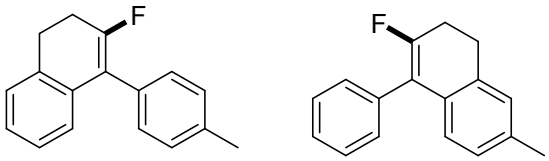


Compound 2g: colorless oil (21.6 mg, 37%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.38-7.27 (m, 3H), 7.21-7.12 (m, 2H), 7.01 (t, *J* = 7.9 Hz, 1H), 6.66 (d, *J* = 7.6 Hz, 1H), 3.21 (td, *J* = 8.2, 2.3 Hz, 2H), 2.72 (td, *J* = 8.3, 5.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8 (d, *J* = 270.1 Hz), 136.7 (d, *J* = 5.2 Hz), 135.2 (d, *J* = 0.9 Hz), 134.3, 133.1, 130.4, 130.2 (d, *J* = 2.0 Hz), 129.7, 128.4 (d, *J* = 1.8 Hz), 127.8, 127.5 (d, *J* = 2.0 Hz), 127.4, 123.8 (d, *J* = 6.7 Hz), 115.9 (d, *J* = 13.6 Hz), 25.1 (d, *J* = 6.8 Hz), 24.4 (d, *J* = 24.3 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ -99.7. IR (neat) ν 3060, 2940, 2840, 1671, 1495, 1246, 1223, 946, 834, 760, 700 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{11}Cl_2F$ requires $[M]^+$: 292.0216, Found: 292.0210.

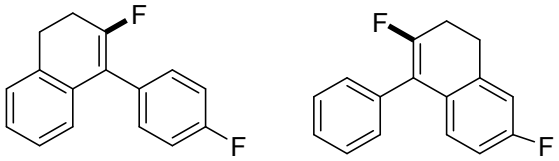


Compound 2h: colorless oil (18.2 mg, 36%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.23-7.14 (m, 4H), 7.06 (d, *J* = 7.2 Hz, 1H), 7.01 (t, *J* = 7.4 Hz, 1H), 6.87 (d, *J* = 7.4 Hz, 1H), 3.08-2.91 (m, 2H), 2.66-2.50 (m, 2H), 2.14 (s, 3H), 1.56 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4 (d, *J* = 268.5 Hz), 136.9 (d, *J* = 1.0 Hz), 135.8 (d, *J* = 1.4 Hz), 135.3 (d, *J* = 6.3 Hz), 135.0, 133.4 (d, *J* = 5.1 Hz), 130.7, 130.1 (d, *J* = 1.9 Hz), 129.9, 127.3, 126.1 (d, *J* = 1.6 Hz), 125.5, 125.3, 116.7 (d, *J* = 14.0 Hz), 30.5 (d, *J* = 4.4 Hz), 25.2 (d, *J* = 25.5 Hz), 21.7, 19.9 (d, *J* = 1.9 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ -97.3. IR (neat) ν 3058, 2951, 1656, 1461, 1360, 928, 761, 737 cm^{-1}. HRMS (EI) Calcd. for $C_{18}H_{17}F$ requires $[M]^+$: 252.1309,

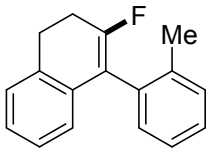
Found: 252.1309.

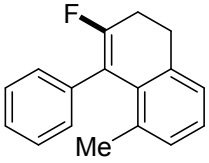


Compound 2j & 2j': (1.1: 1): colorless oil (23.4 mg, 53%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.41 (t, *J* = 7.2 Hz, 1H), 7.37-7.28 (m, 1.5H), 7.26-7.18 (m, 1.5H), 7.17-7.01 (m, 2H), 6.99 (s, 0.5H), 6.91-6.80 (m, 1H), 6.70 (d, *J* = 7.8 Hz, 0.5H), 3.11-3.00 (m, 2H), 2.79-2.63 (m, 2H), 2.40 (s, 1.6H), 2.29 (s, 1.4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9 (d, *J* = 268.3 Hz), 157.3 (d, *J* = 267.8 Hz), 137.1, 135.9 (d, *J* = 2.3 Hz), 135.4 (d, *J* = 5.4 Hz), 133.8 (d, *J* = 1.0 Hz), 132.9, 132.8, 132.4 (d, *J* = 5.2 Hz), 130.6 (d, *J* = 1.0 Hz), 130.2 (d, *J* = 1.9 Hz), 130.0 (d, *J* = 1.8 Hz), 129.1, 128.6, 128.3, 128.2, 127.3, 127.2, 127.0, 126.4, 126.09, 126.07, 125.31 (d, *J* = 6.8 Hz), 125.26 (d, *J* = 6.8 Hz), 28.8 (d, *J* = 1.6 Hz), 28.7 (d, *J* = 1.6 Hz), 25.3 (d, *J* = 24.7 Hz), 25.2 (d, *J* = 24.8 Hz), 21.3, 21.0. ^{19}F NMR (376 MHz, $CDCl_3$) δ -102.4, -103.7. IR (neat) ν 3026, 2943, 1669, 1483, 1360, 1151, 817, 760, 743 cm^{-1}. HRMS (EI) Calcd. for $C_{17}H_{15}F$ requires $[M]^+$: 238.1152, Found: 238.1150.

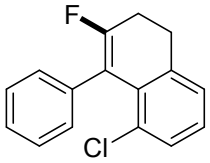


Compound 2k & 2k': (1 : 1.3), colorless oil (21.8 mg, 45%); M.p. 83-85 °C. ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.44-7.22 (m, 4H), 7.19-7.02 (m, 2H), 6.87 (dd, *J* = 9.0, 2.0 Hz, 0.5H), 6.80-6.70 (m, 1.5 H), 3.10-3.00 (m, 2H), 2.67 (td, *J* = 8.2, 5.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.1 (d, *J* = 244.8 Hz), 161.3 (dd, *J* = 244.3, 2.6 Hz), 158.3 (d, *J* = 267.6 Hz), 157.3 (dd, *J* = 264.0, 2.6 Hz), 135.2 (d, *J* = 7.5 Hz), 135.1 (d, *J* = 5.2 Hz), 133.5 (d, *J* = 0.8 Hz), 132.8, 131.8 (dd, *J* = 8.0, 1.9 Hz), 131.3 (dd, *J* = 5.3, 3.0 Hz), 130.1 (d, *J* = 1.9 Hz), 129.4 (d, *J* = 4.5 Hz), 128.4, 127.5, 127.3, 126.7 (dd, *J* = 7.8, 0.6 Hz), 126.5, 126.3 (d, *J* = 2.2 Hz), 125.1 (d, *J* = 6.8 Hz), 116.6 (d, *J* = 14.2 Hz), 116.3 (d, *J* = 13.0 Hz), 115.3 (d, *J* = 21.3 Hz), 114.5 (d, *J* = 22.1 Hz), 112.8 (d, *J* = 21.0 Hz), 28.7 (dd, *J* = 6.8, 1.6 Hz), 28.6 (d, *J* = 6.6 Hz), 25.2 (d, *J* = 25.0 Hz), 24.8 (d, *J* = 25.2 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ -101.9, -103.5, -114.7, -116.6. IR (neat) ν 3024, 2922, 1733, 1494, 1246, 1223, 1158, 834, 760 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{12}F_2$ requires $[M]^+$: 242.0902, Found: 242.0901.



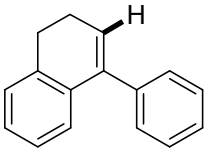


Compound 2l & 2l': (2 : 1), colorless oil (18.1 mg, 38%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.38-7.20 (m, 3.7H), 7.16 (d, *J* = 7.1 Hz, 1.3H), 7.12-6.99 (m, 2H), 6.92 (d, *J* = 6.1 Hz, 0.3H), 6.55 (d, *J* = 7.5 Hz, 0.7H), 3.11 (dt, *J* = 8.3, 4.1 Hz, 1.3H), 3.02-2.93 (m, 0.7H), 2.71 (td, *J* = 8.3, 4.6 Hz, 1.3H), 2.63-2.50 (m, 0.7H), 2.16 (s, 2H), 1.64 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7 (d, *J* = 271.6 Hz), 157.8 (d, *J* = 265.5 Hz), 137.4 (d, *J* = 0.9 Hz), 136.2, 135.9, 135.5 (d, *J* = 6.0 Hz), 135.0 (d, *J* = 5.9 Hz), 133.7 (d, *J* = 4.9 Hz), 133.1 (d, *J* = 1.7 Hz), 132.4, 130.54, 130.50 (d, *J* = 1.5 Hz), 130.0, 129.2 (d, *J* = 2.5 Hz), 128.1, 127.8, 127.2, 126.7, 126.6, 126.2 (d, *J* = 1.6 Hz), 126.1 (d, *J* = 2.2 Hz), 125.8, 124.9, 124.7 (d, *J* = 6.8 Hz), 117.1 (d, *J* = 11.6 Hz), 116.5 (d, *J* = 15.1 Hz), 30.4 (d, *J* = 3.6 Hz), 28.7 (d, *J* = 7.0 Hz), 25.6 (d, *J* = 25.4 Hz), 25.0 (d, *J* = 24.4 Hz), 22.6, 19.6. ^{19}F NMR (376 MHz, $CDCl_3$) δ -100.0, -100.8. IR (neat) ν 3019, 2945, 1674, 1483, 1361, 1151, 931, 762, 752, 732 cm^{-1}. HRMS (EI) Calcd. for $C_{18}H_{17}F$ requires $[M]^+$: 238.1152, Found: 238.1147.

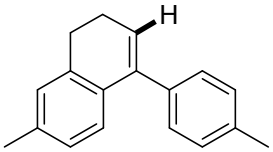


Compound 2m: colorless oil (18.3 mg, 35%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.49 (dt, *J* = 7.4, 3.6 Hz, 1H), 7.36-7.31 (m, 3H), 7.29-7.25 (m, 1H), 7.17 (d, *J* = 7.2 Hz, 1H), 7.14-7.03 (m, 2H), 6.56 (d, *J* = 8.1 Hz, 1H), 3.18-3.04 (m, 2H), 2.76-2.69 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8 (d, *J* = 268.3 Hz), 134.6, 134.2 (d, *J* = 5.3 Hz), 132.7 (d, *J* = 1.5 Hz), 132.2 (d, *J* = 2.2 Hz), 129.7, 129.2, 127.3, 126.8, 126.6, 126.3 (d, *J* = 2.1 Hz), 124.5 (d, *J* = 6.7 Hz), 28.6 (d, *J* = 7.0 Hz), 25.0 (d, *J* = 23.7 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ -98.6. IR (neat) ν 3060, 2943, 1679, 1485, 1363, 1231, 1153, 1060, 935, 752, 733, 715 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{12}ClF$ requires $[M]^+$: 258.0606, Found: 258.0600.

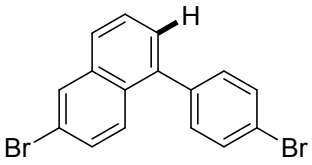
7. Spectroscopy of compounds 3



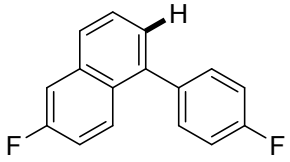
Compound 3a: colorless oil (31.2 mg, 76%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.41-7.29 (m, 5H), 7.23-7.08 (m, 3H), 7.00 (d, *J* = 7.5 Hz, 1H), 6.09 (t, *J* = 4.6 Hz, 1H), 2.85 (t, *J* = 8.0 Hz, 2H), 2.41 (td, *J* = 7.9, 4.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.8, 139.9, 136.8, 135.1, 128.7, 128.2, 127.6, 127.5, 127.1, 127.0, 126.2, 125.4, 28.3, 23.5. IR (neat) ν 3026, 2917, 1716, 1660, 1447, 1268, 927, 756, 699 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{14}$ requires $[M]^+$: 206.1090, Found: 206.1088.



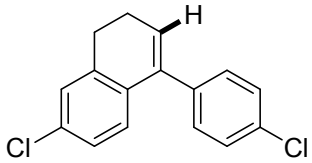
Compound 3b: colorless oil (29.9 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.23 (d, *J* = 8.1 Hz, 2H), 7.17 (d, *J* = 7.8 Hz, 2H), 7.02 (s, 1H), 6.93-6.87 (m, 2H), 6.00 (t, *J* = 4.6 Hz, 1H), 2.80 (t, *J* = 7.9 Hz, 2H), 2.37 (m, 5H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.5, 138.0, 136.8, 136.63, 136.61, 132.5, 128.8, 128.6, 128.4, 126.7, 126.2, 125.4, 28.4, 23.6, 21.2, 21.1. IR (neat) ν 2921, 2859, 1713, 1655, 1605, 1276, 1040, 820, 794 cm^{-1}. HRMS (EI) Calcd. for $C_{18}H_{18}$ requires $[M]^+$: 234.1403, Found: 234.1401.



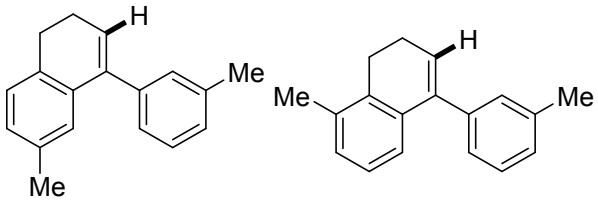
Compound 3c: colorless oil (39.1 mg, 54%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 8.06 (d, *J* = 1.8 Hz, 1H), 7.77 (d, *J* = 8.2 Hz, 1H), 7.70 (d, *J* = 9.0 Hz, 1H), 7.66-7.59 (m, 2H), 7.58-7.47 (m, 2H), 7.39 (d, *J* = 7.0 Hz, 1H), 7.36-7.29 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 139.0, 134.9, 131.6, 130.3, 129.9, 129.6, 127.5, 127.2, 127.1, 126.5, 121.8, 120.1. IR (neat) ν 3045, 2919, 1583, 1494, 1070, 958, 829, 777, 740 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{10}Br_2$ requires $[M]^+$: 359.9144, Found: 359.9145.



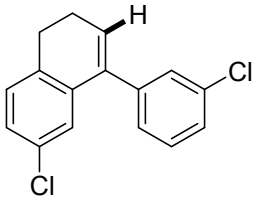
Compound 3d: colorless oil (32.2 mg, 67%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.86-7.75 (m, 2H), 7.56-7.48 (m, 2H), 7.46-7.38 (m, 2H), 7.34 (d, *J* = 7.0 Hz, 1H), 7.24-7.14 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4 (d, *J* = 245.0 Hz), 160.5 (d, *J* = 245.3 Hz), 139.4 (d, *J* = 1.0 Hz), 136.4 (d, *J* = 3.4 Hz), 134.7 (d, *J* = 9.1 Hz), 131.5 (d, *J* = 7.9 Hz), 128.7, 128.5 (d, *J* = 8.8 Hz), 127.1 (d, *J* = 5.2 Hz), 126.5, 126.2 (d, *J* = 2.1 Hz), 116.3 (d, *J* = 24.8 Hz), 115.3 (d, *J* = 21.2 Hz), 111.2 (d, *J* = 20.1 Hz). ^{19}F NMR (376 MHz, $CDCl_3$) δ -115.1, -115.3. IR (neat) ν 3050, 2924, 1626, 1607, 1506, 1220, 863.95, 839, 826, 784 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{10}F_2$ requires $[M]^+$: 240.0745, Found: 240.0742.



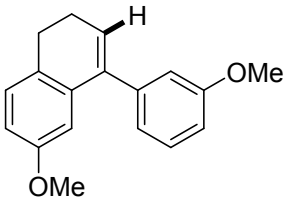
Compound 3e: colorless oil (31.4 mg, 57%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.34 (d, *J* = 7.7 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.18 (s, 1H), 7.07 (d, *J* = 8.2 Hz, 1H), 6.87 (d, *J* = 8.2 Hz, 1H), 6.06 (d, *J* = 5.1 Hz, 1H), 2.81 (t, *J* = 7.8 Hz, 2H), 2.39 (q, *J* = 5.9 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 138.6, 138.1, 133.2, 133.1, 132.5, 129.9, 128.5, 128.2, 127.7, 126.4, 126.2, 28.0, 23.2. IR (neat) ν 3032, 2948, 2830, 1550, 1491, 1090, 1012, 959, 840, 823, 808 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{12}Cl_2$ requires $[M]^+$: 274.0311, Found: 274.0309.



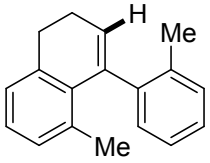
Compound 3f & 3f': (2 : 1), colorless oil (34.2 g, 73%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.27-7.22 (m, 1H), 7.18-6.95 (m, 5H), 6.88-6.80 (m, 1H), 6.05 (t, *J* = 4.7 Hz, 1H), 2.80 (t, *J* = 8.0 Hz, 2H), 2.42-2.28 (m, 7.1H), 2.22 (s, 0.9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.2, 140.9, 140.2, 140.0, 137.8, 137.7, 135.6, 135.1, 135.0, 134.7, 133.7, 129.5, 129.4, 129.1, 128.05, 128.00, 127.73, 127.68, 127.6, 127.5, 127.4, 127.0, 126.2, 125.9, 125.8, 125.4, 123.1, 27.9, 23.9, 23.7, 23.2, 21.5, 21.4, 21.2, 19.8. IR (neat) ν 2942, 2914, 1674, 1460, 1227, 1151, 961, 781, 725, 702 cm^{-1}. HRMS (EI) Calcd. for $C_{18}H_{18}$ requires $[M]^+$: 234.1403, Found: 234.1398.



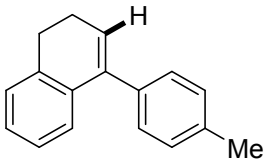
Compound 3g: colorless oil (39.6 mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.33-7.28 (m, 2H), 7.24 (d, *J* = 8.5 Hz, 1H), 7.20-7.15 (m, 1H), 7.04 (t, *J* = 7.9 Hz, 1H), 6.85 (d, *J* = 7.7 Hz, 1H), 6.12 (t, *J* = 4.7 Hz, 1H), 2.98 (t, *J* = 8.1 Hz, 2H), 2.42 (td, *J* = 8.1, 4.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.3, 138.4, 136.5, 134.2, 134.1, 133.1, 129.5, 129.2, 128.8, 128.2, 127.4, 126.9, 126.8, 123.9, 24.3, 22.8. IR (neat) ν 3060, 2930, 1589, 1561, 1389, 1094, 1001, 779, 707 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{12}Cl_2$ requires $[M]^+$: 274.0311, Found: 274.0309.

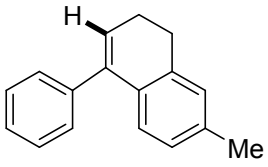


Compound 3h: colorless oil (35.2 mg, 66%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.26 (t, *J* = 7.7 Hz, 1H), 7.07 (t, *J* = 8.0 Hz, 1H), 6.94-6.83 (m, 2H), 6.79 (d, *J* = 8.2 Hz, 1H), 6.67 (d, *J* = 7.7 Hz, 1H), 6.09 (t, *J* = 4.6 Hz, 1H), 3.85 (s, 3H), 3.80 (s, 3H), 2.85 (t, *J* = 8.1 Hz, 2H), 2.36 (td, *J* = 8.1, 4.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 156.0, 142.5, 139.6, 136.0, 129.0, 127.9, 126.1, 124.6, 121.3, 118.6, 114.2, 112.7, 109.7, 55.6, 55.2, 22.9, 19.8. IR (neat) ν 2932, 2833, 1573, 1460, 1435, 1258, 1041, 782, 763, 702 cm^{-1}. HRMS (EI) Calcd. for $C_{18}H_{18}O_2$ requires $[M]^+$: 266.1301, Found: 266.1301.

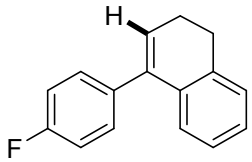


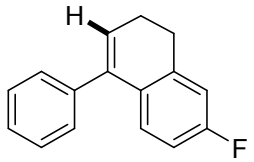
Compound 3i: colorless oil (24.8 mg, 53%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.24-7.16 (m, 3H), 7.14-7.01 (m, 3H), 6.89 (d, *J* = 7.3 Hz, 1H), 6.10 (dd, *J* = 5.8, 4.3 Hz, 1H), 2.81-2.73 (m, 2H), 2.35-2.21 (m, 2H), 1.99 (s, 3H), 1.61 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.6, 140.3, 138.2, 135.4, 134.4, 133.9, 131.6, 130.2, 129.6, 129.0, 126.7, 126.6, 125.9, 125.4, 30.2, 23.1, 22.1, 20.0. IR (neat) ν 3021, 2926, 1664, 1459, 1163, 1033, 752, 727 cm^{-1}. HRMS (EI) Calcd. for $C_{18}H_{18}$ requires $[M]^+$: 234.1403, Found: 234.1399.



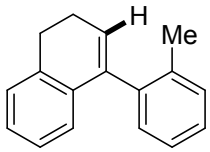


Compound 3j & 3j': (1.1 : 1), colorless oil (32.6 mg, 74%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.38-7.28 (m, 2H), 7.25-7.06 (m, 4H), 7.01 (d, *J* = 6.9 Hz, 1H), 6.89 (d, *J* = 8.1 Hz, 1H), 6.08-6.00 (m, 1H), 2.87-2.78 (m, 2H), 2.43-2.34 (m, 3.6H), 2.32 (s, 1.4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.9, 139.73, 139.68, 137.8, 136.8, 136.75, 136.71, 136.68, 135.2, 132.4, 128.9, 128.7, 128.65, 128.60, 128.4, 128.1, 127.5, 127.2, 127.0, 126.9, 126.7, 126.6, 126.1, 125.42, 125.38, 28.34, 28.30, 23.6, 23.5, 21.2, 21.1. IR (neat) ν 3021, 2924, 1728, 1659, 1446, 1180, 815, 757, 741 cm^{-1}. HRMS (EI) Calcd. for $C_{17}H_{16}$ requires $[M]^+$: 220.1247, Found: 220.1243.



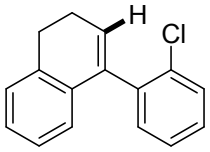


Compound 3k & 3k': (1 : 2.5), colorless oil (29.4 mg, 66%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.40-7.27 (m, 3.6H), 7.22-7.03 (m, 2H), 6.98-6.88 (m, 1.7H), 6.77 (t, *J* = 8.6 Hz, 0.7H), 6.04 (t, *J* = 5.7 Hz, 1H), 2.83 (t, *J* = 7.0 Hz, 2H), 2.40 (q, *J* = 6.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3, 161.6 (d, *J* = 246.5 Hz), 140.6, 139.3 (d, *J* = 7.5 Hz), 139.1, 138.9, 136.73, 136.68, 134.9, 131.2 (d, *J* = 3.0 Hz), 130.3 (d, *J* = 7.9 Hz), 128.6, 128.3, 127.8, 127.6, 127.2, 127.1, 126.9 (d, *J* = 8.1 Hz), 126.6 (d, *J* = 2.1 Hz), 126.2, 125.2, 115.0 (d, *J* = 21.2 Hz), 114.6 (d, *J* = 21.5 Hz), 112.5 (d, *J* = 21.0 Hz), 28.5 (d, *J* = 1.3 Hz), 28.2, 23.5, 23.2. ^{19}F NMR (376 MHz, $CDCl_3$) δ -115.5, -115.7. IR (neat) ν 3024, 2935, 1603, 1493, 1240, 1222, 828, 763, 740, 701 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{13}F$ requires $[M]^+$: 224.0996, Found: 224.0990.



Compound 3l: colorless oil (22.3 mg, 51%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.28-7.15 (m, 5H), 7.15-7.10 (m, 1H), 7.04 (t, *J* = 7.9 Hz, 1H), 6.61 (d, *J* = 7.6 Hz, 1H), 5.93 (t, *J* = 4.5 Hz, 1H), 2.94-2.82 (m, 2H), 2.47-2.39 (m, 2H), 2.10 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.4, 139.0, 136.5, 135.8,

135.2, 130.0, 129.8, 127.6, 127.4, 127.2, 126.8, 126.4, 125.7, 124.8, 28.2, 23.4, 19.9. IR (neat) ν 3016, 2927, 1485, 1040, 907, 755, 738, 727 cm^{-1}. HRMS (EI) Calcd. for $C_{18}H_{18}$ requires $[M]^+$: 234.1403, Found: 234.1397.

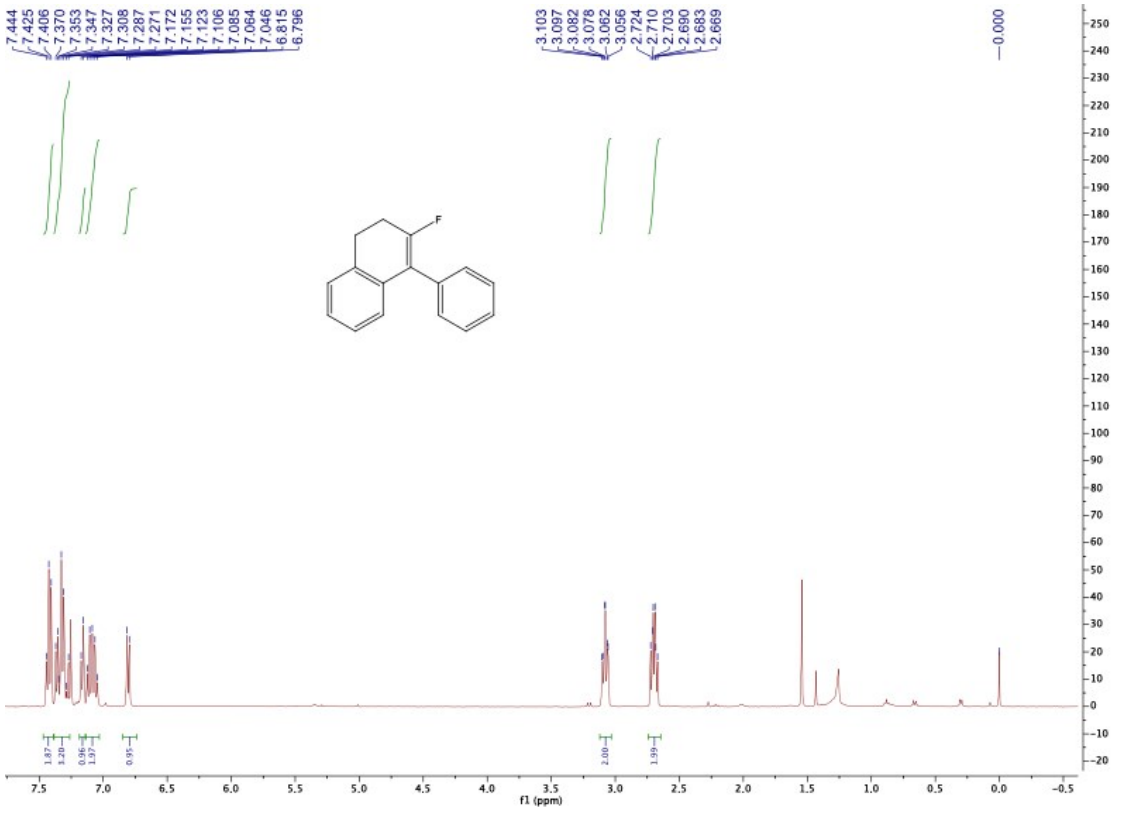


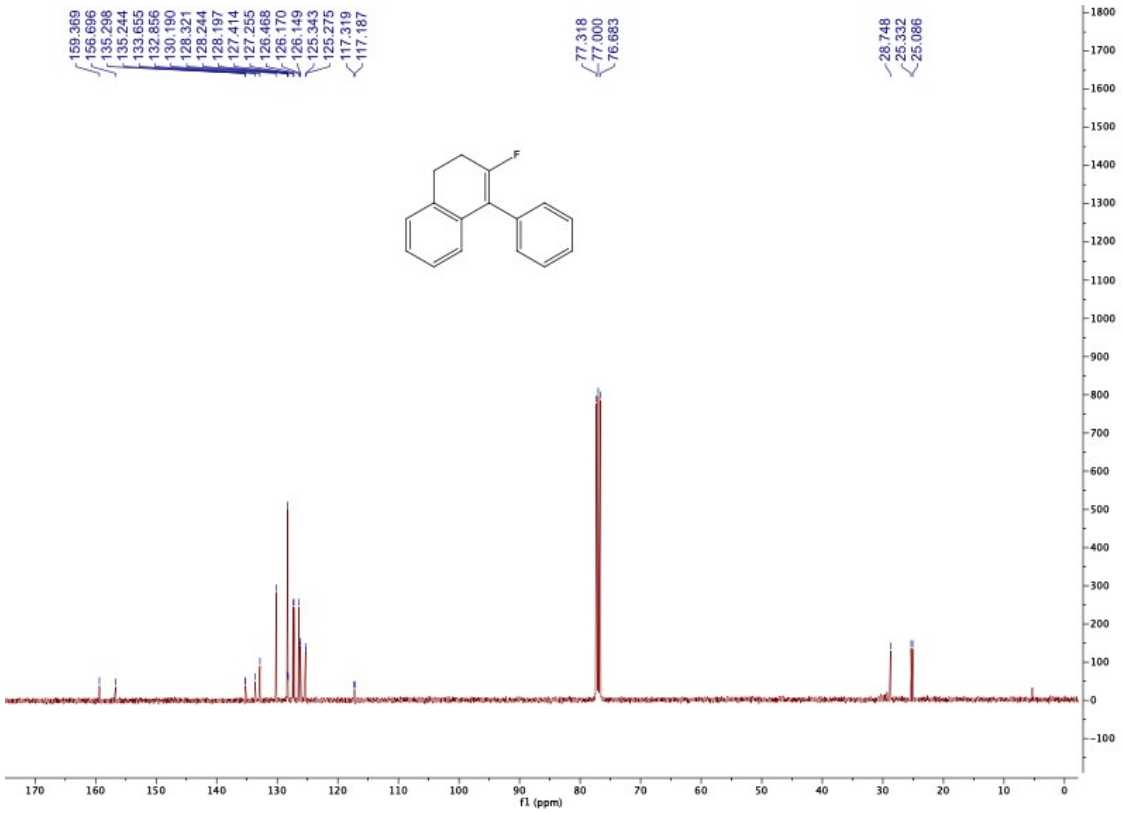
Compound 3m: colorless oil (22.1 mg, 46%). ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.34-7.28 (m, 2H), 7.28-7.22 (m, 3H), 7.20-7.14 (m, 2H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.32 (t, *J* = 5.3 Hz, 1H), 2.76 (t, *J* = 7.6 Hz, 2H), 2.33-2.25 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.2, 141.6, 139.1, 132.82, 132.76, 131.8, 129.5, 128.0, 127.7, 126.9, 126.5, 125.8, 30.1, 23.2. IR (neat) ν 3019, 2932, 1441, 1135, 876, 813, 776, 760 cm^{-1}. HRMS (EI) Calcd. for $C_{16}H_{13}Cl$ requires $[M]^+$: 240.0700, Found: 240.0693.

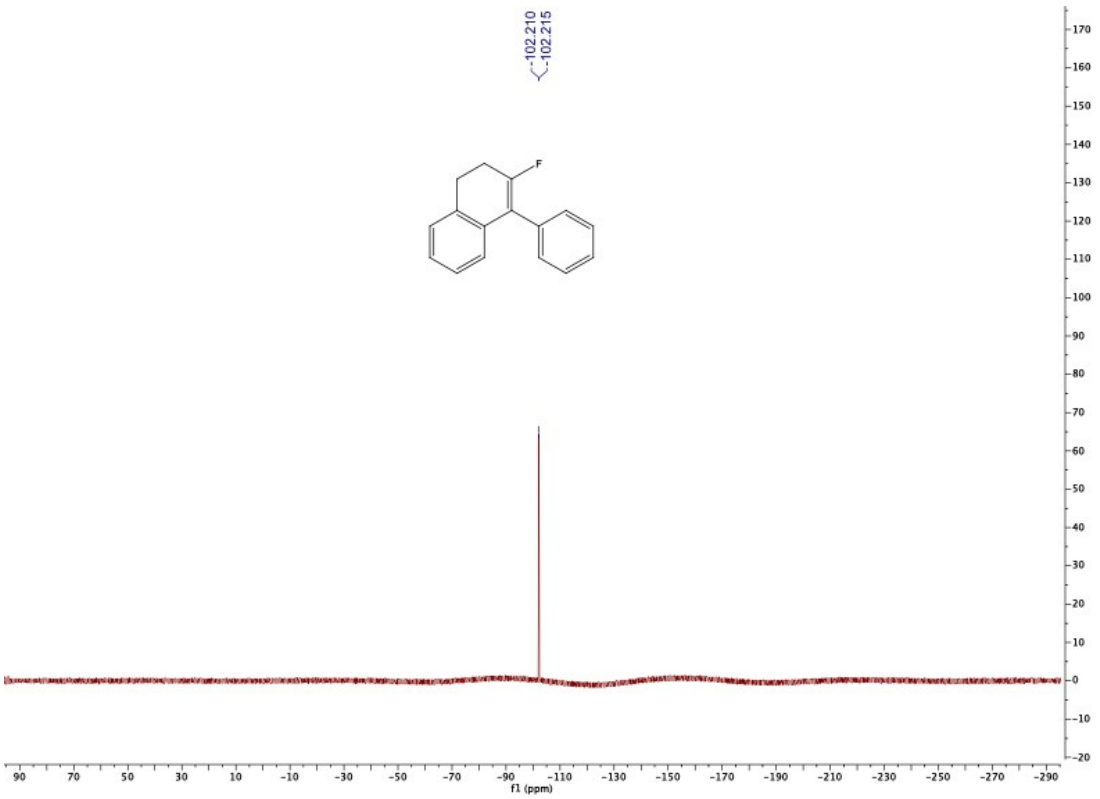
8. Reference

[1] J. Liu, Q. Li, Y. Wei, M. Shi, *Org. Lett*. 2020, **22**, 2494.

9. NMR Spectroscopy



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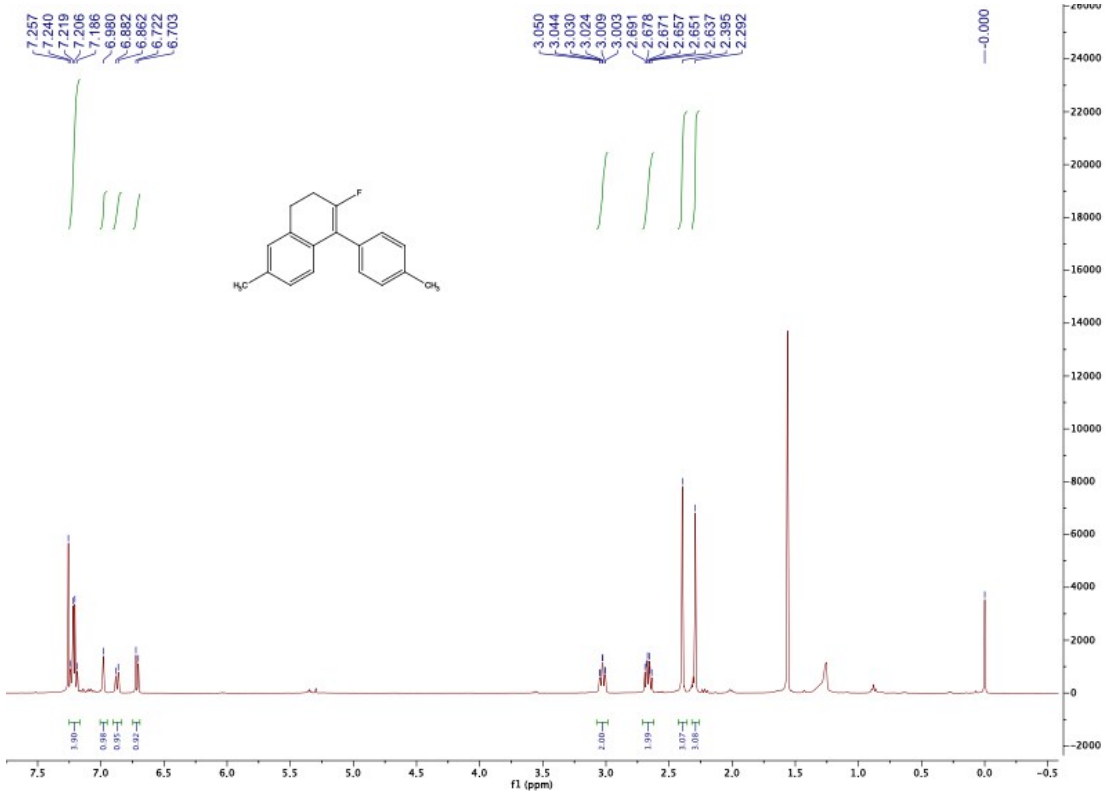
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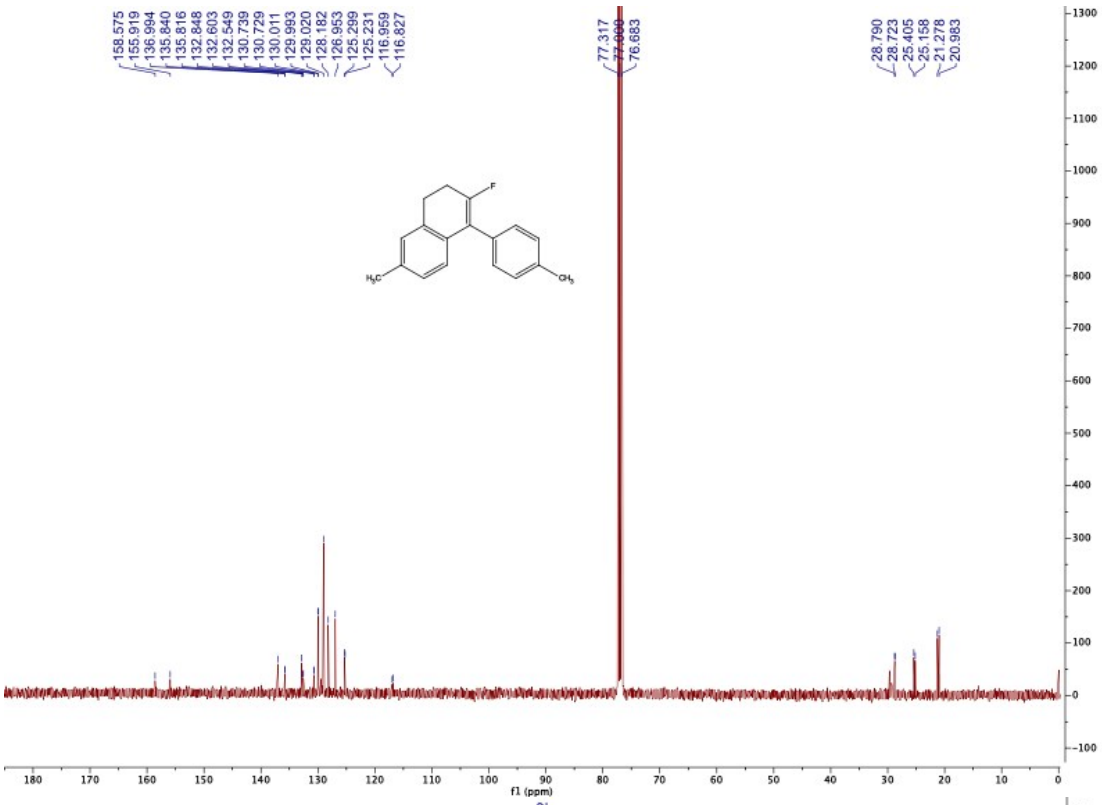
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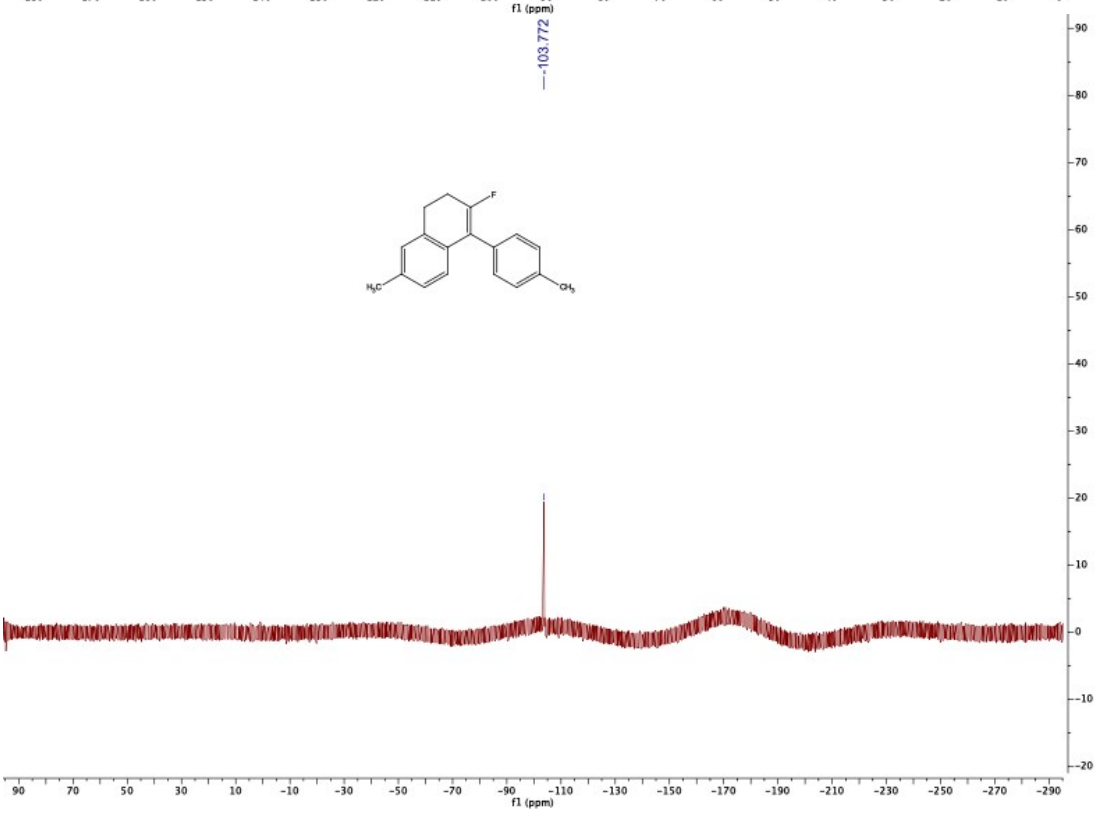
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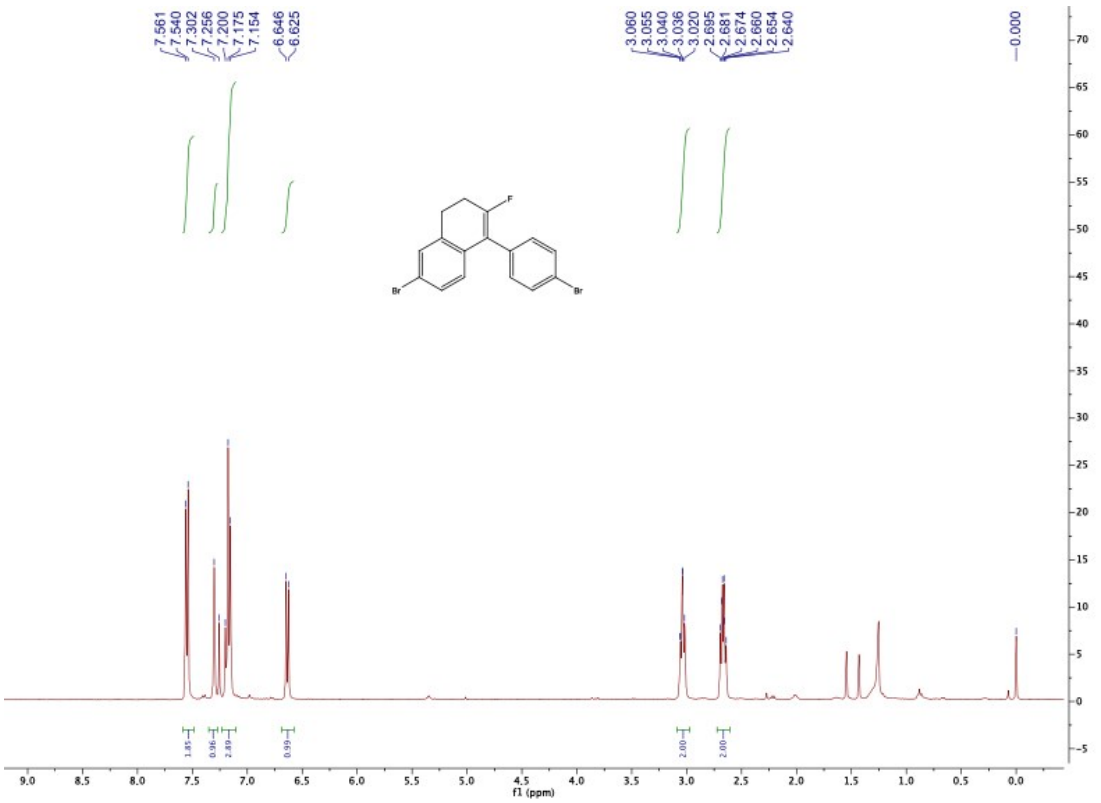
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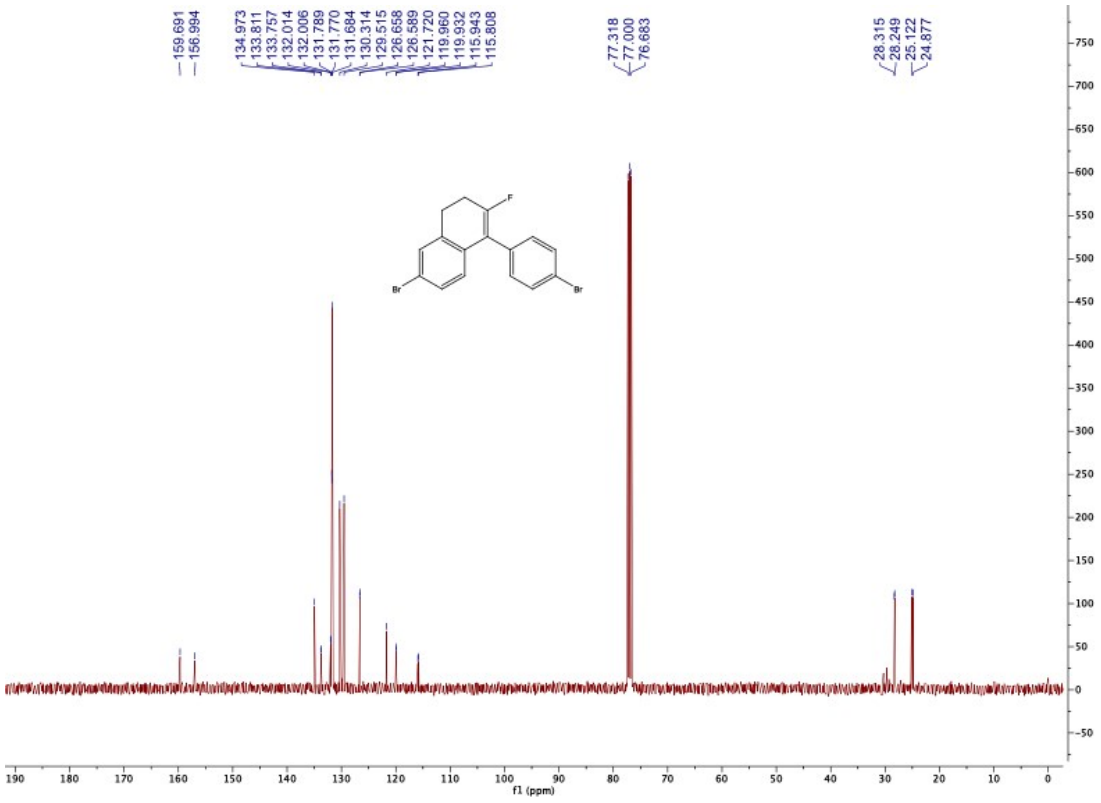
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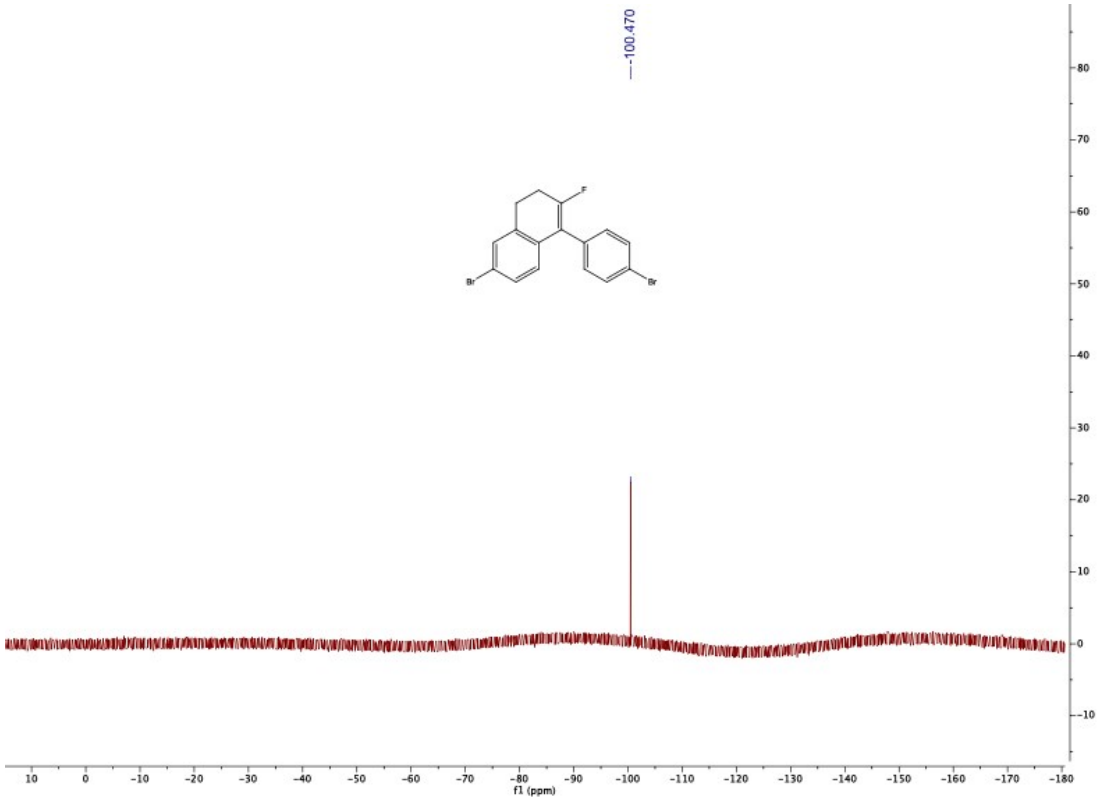
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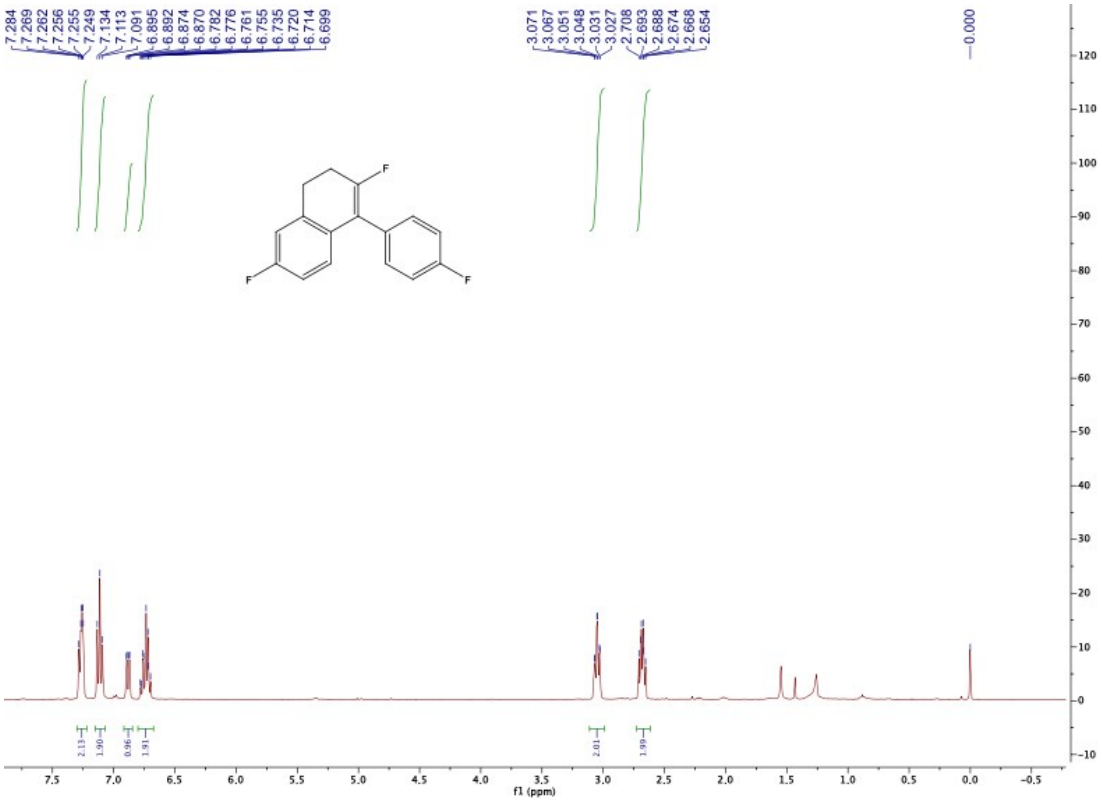
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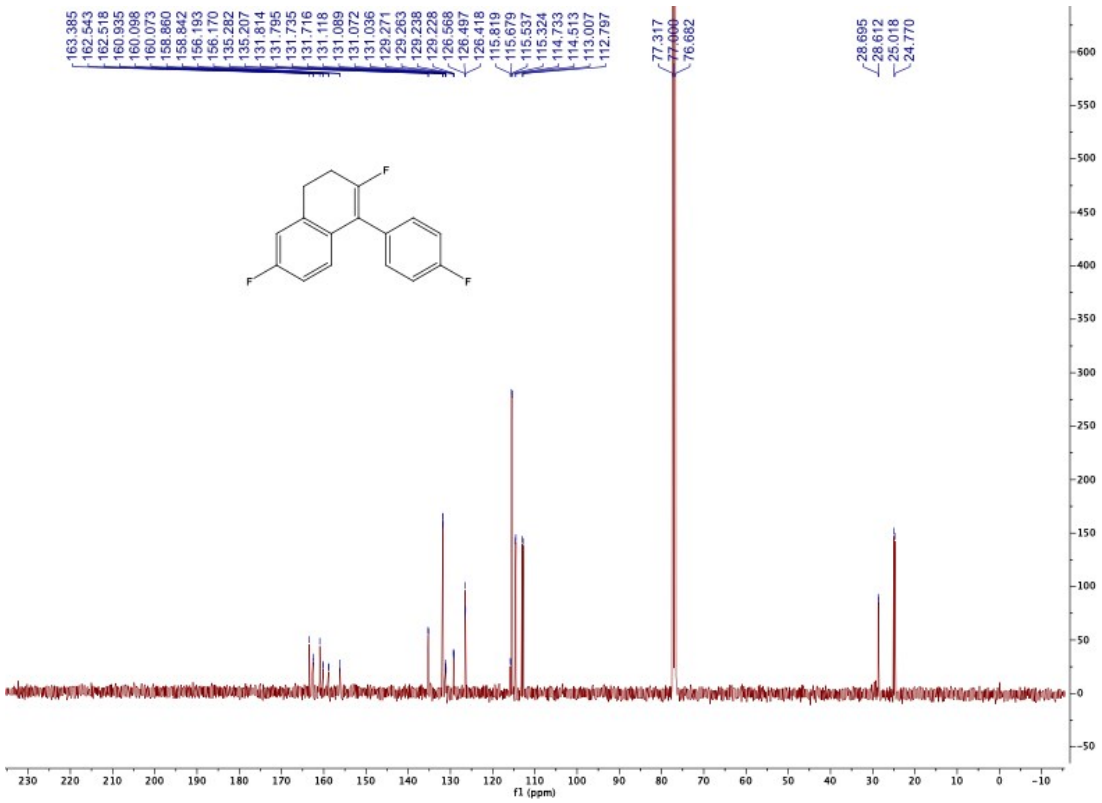
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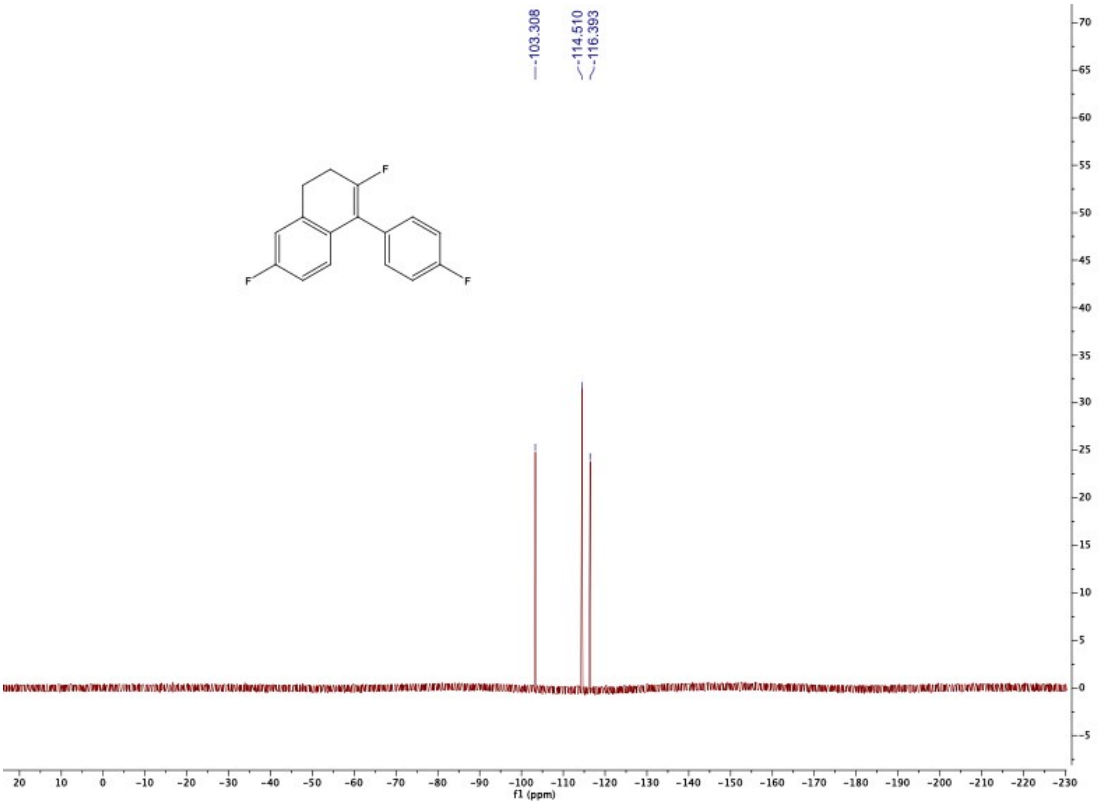
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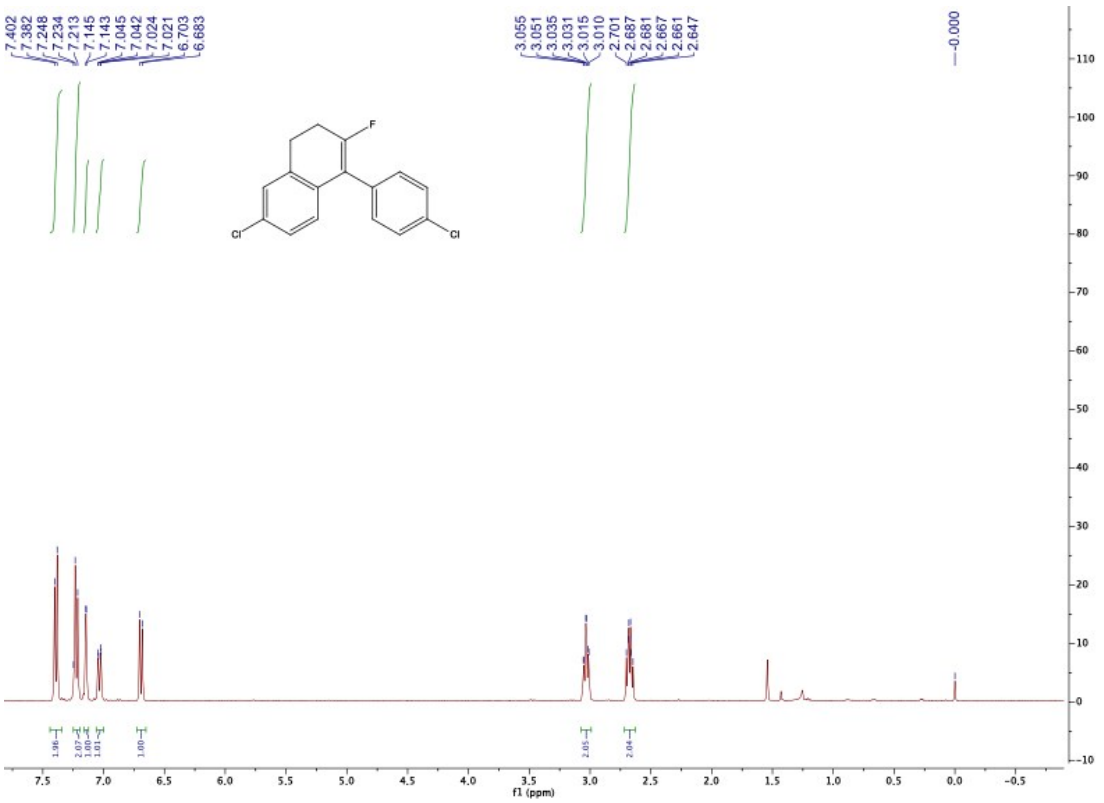
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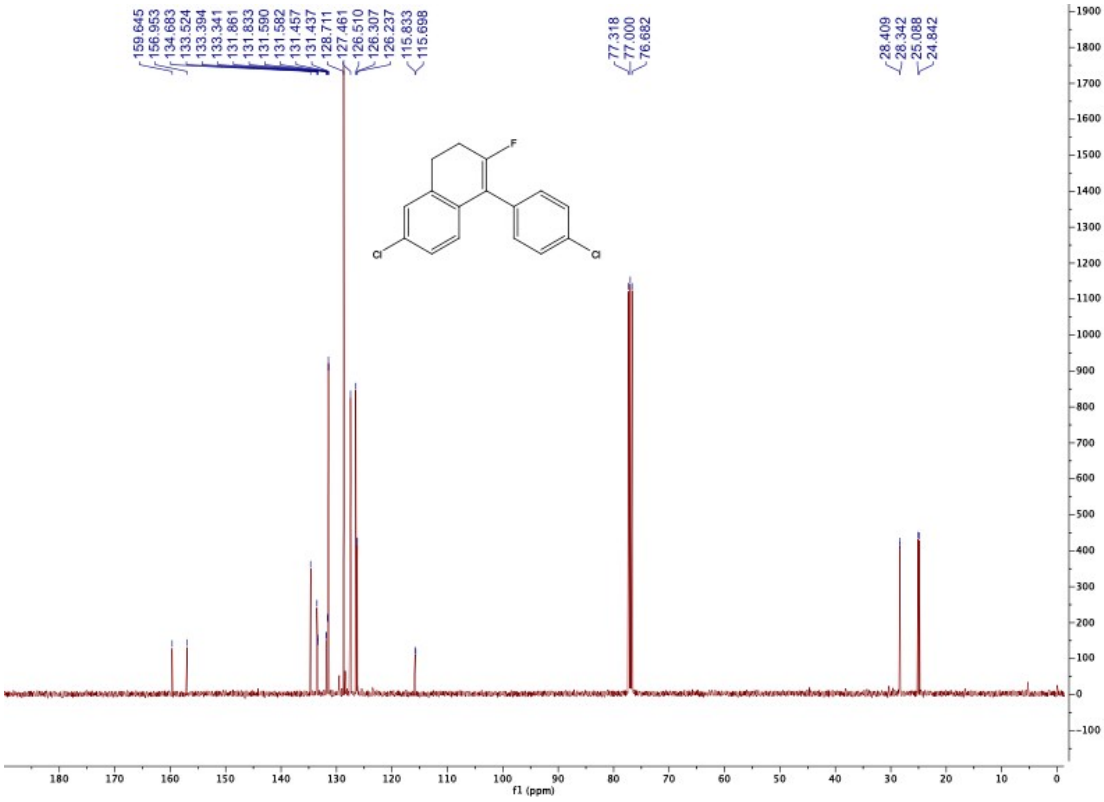
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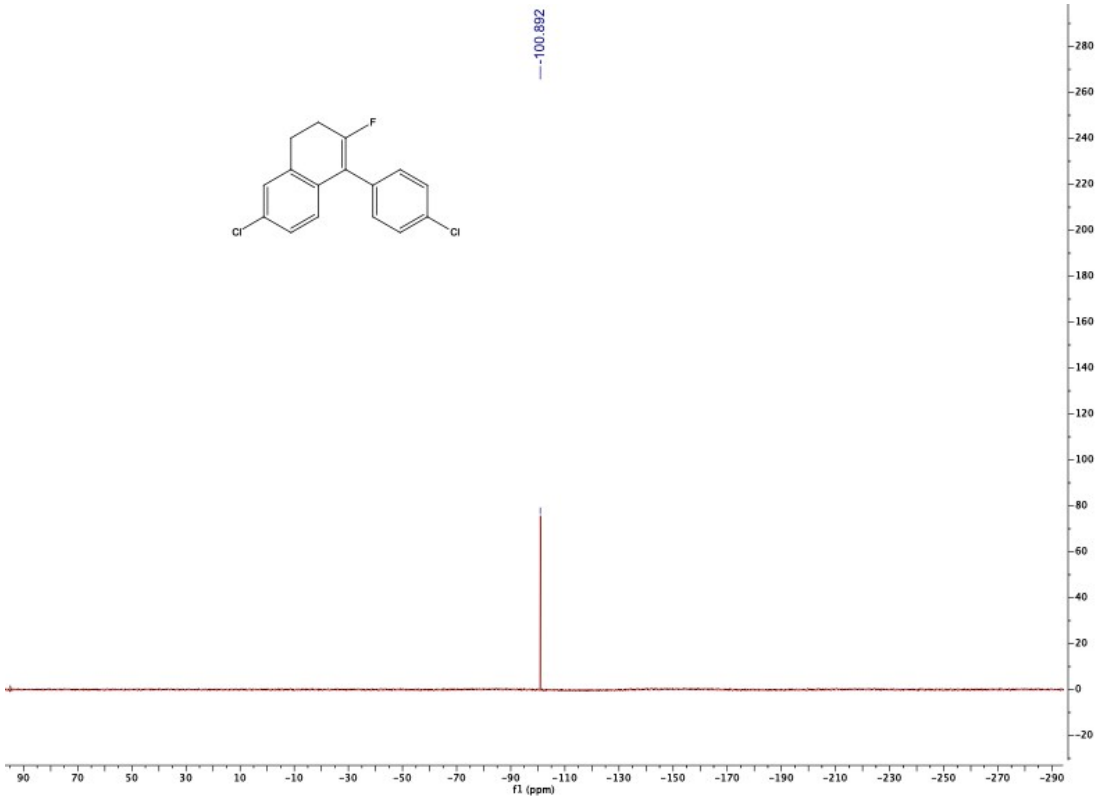
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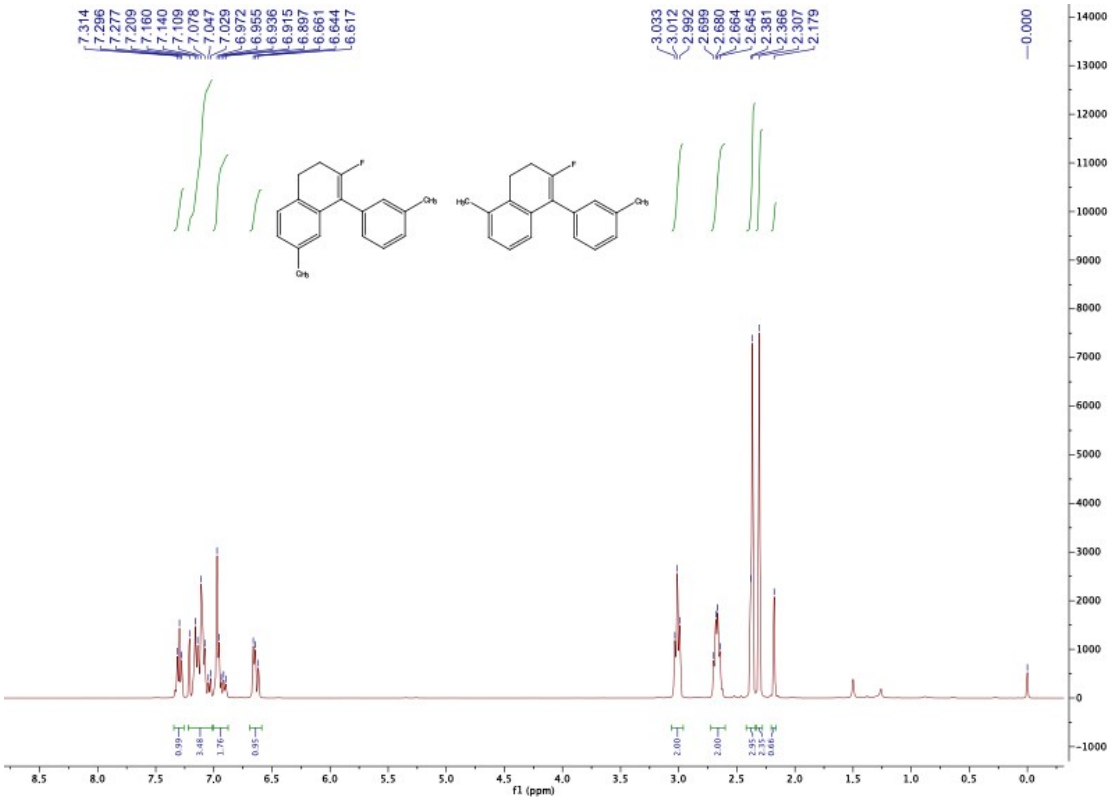
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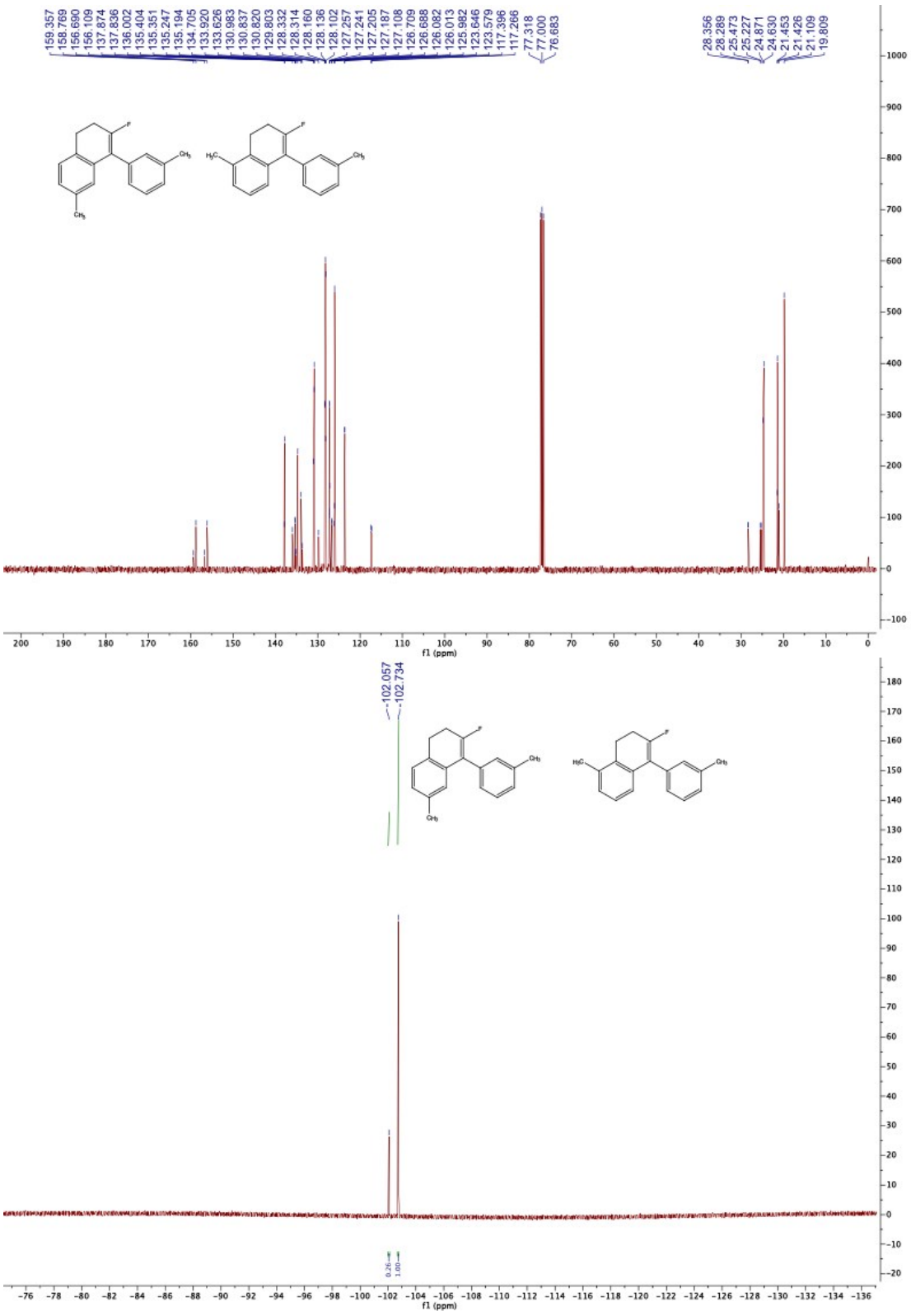
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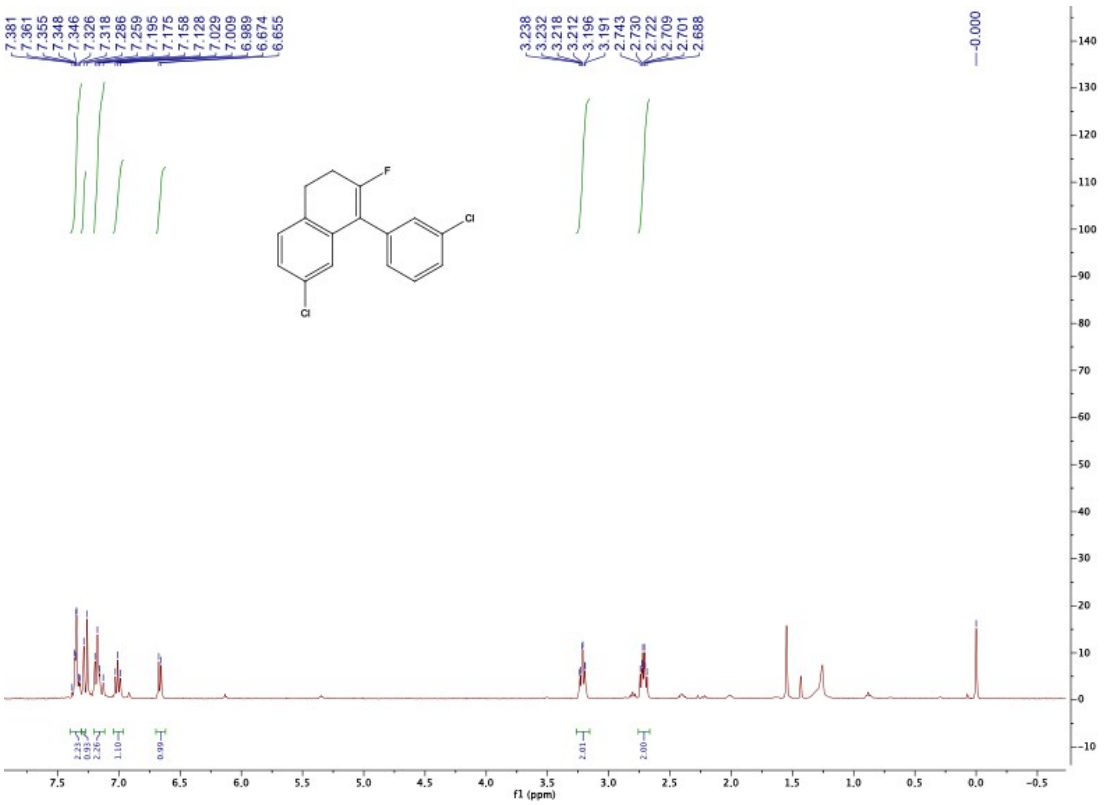
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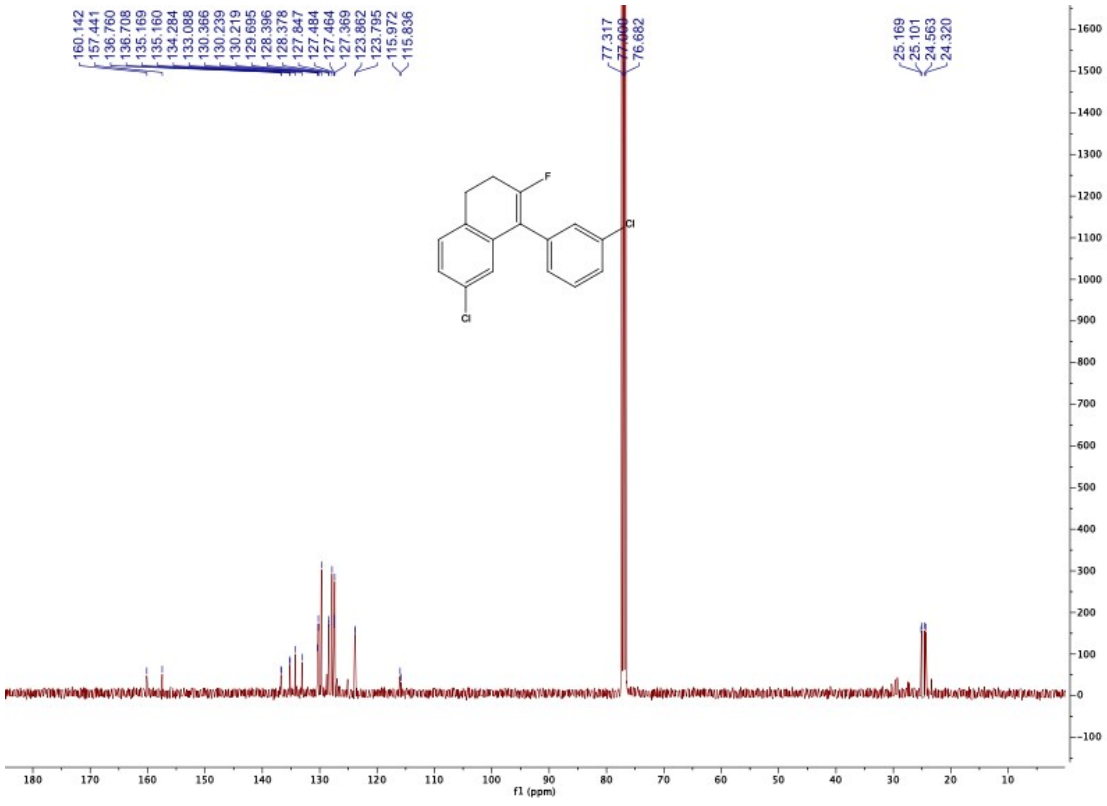
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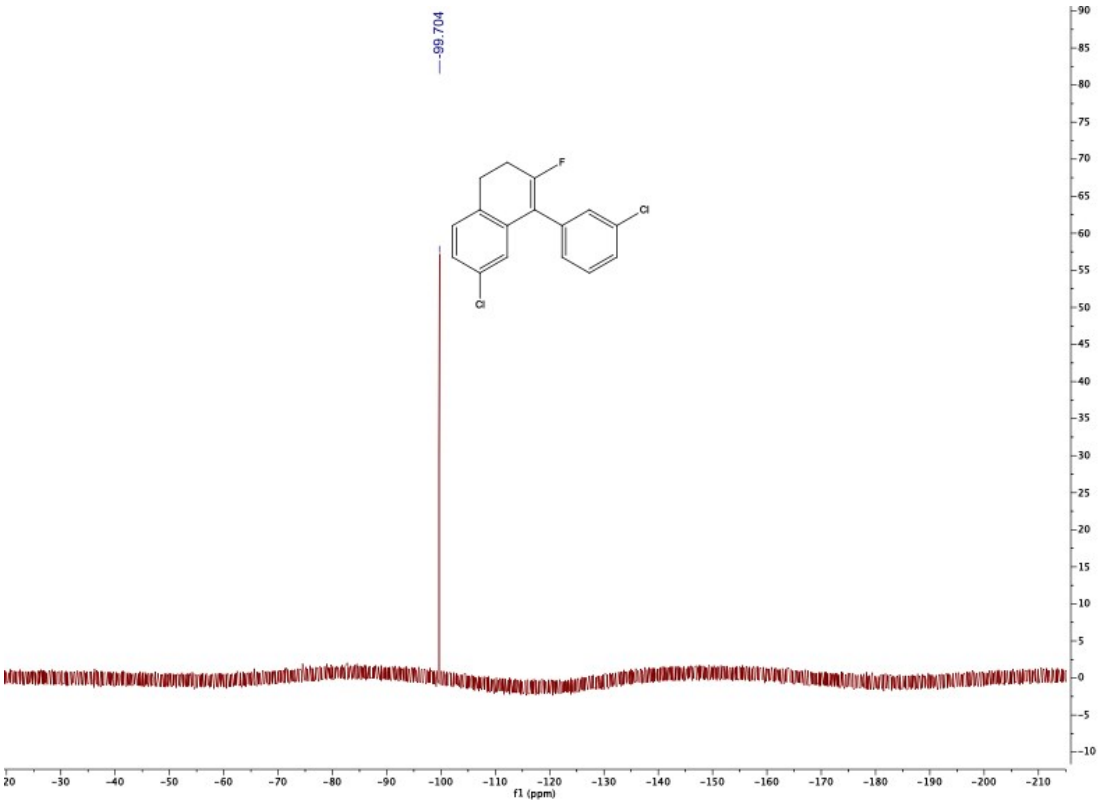
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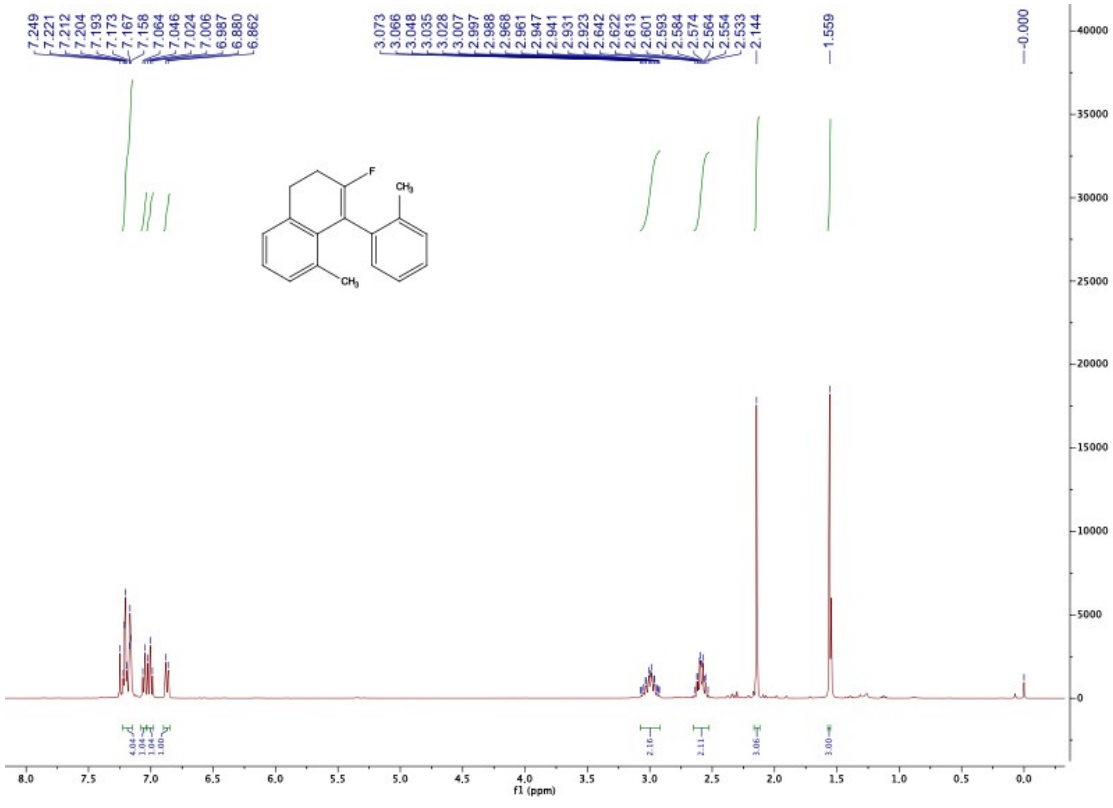
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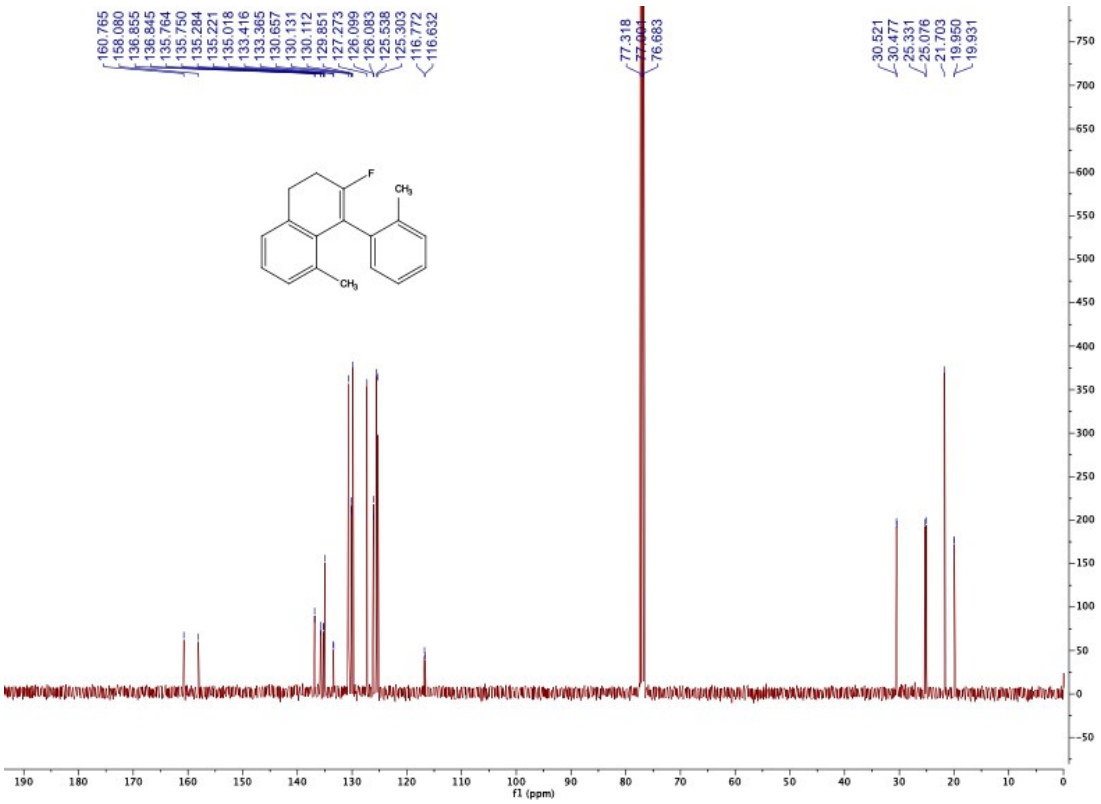
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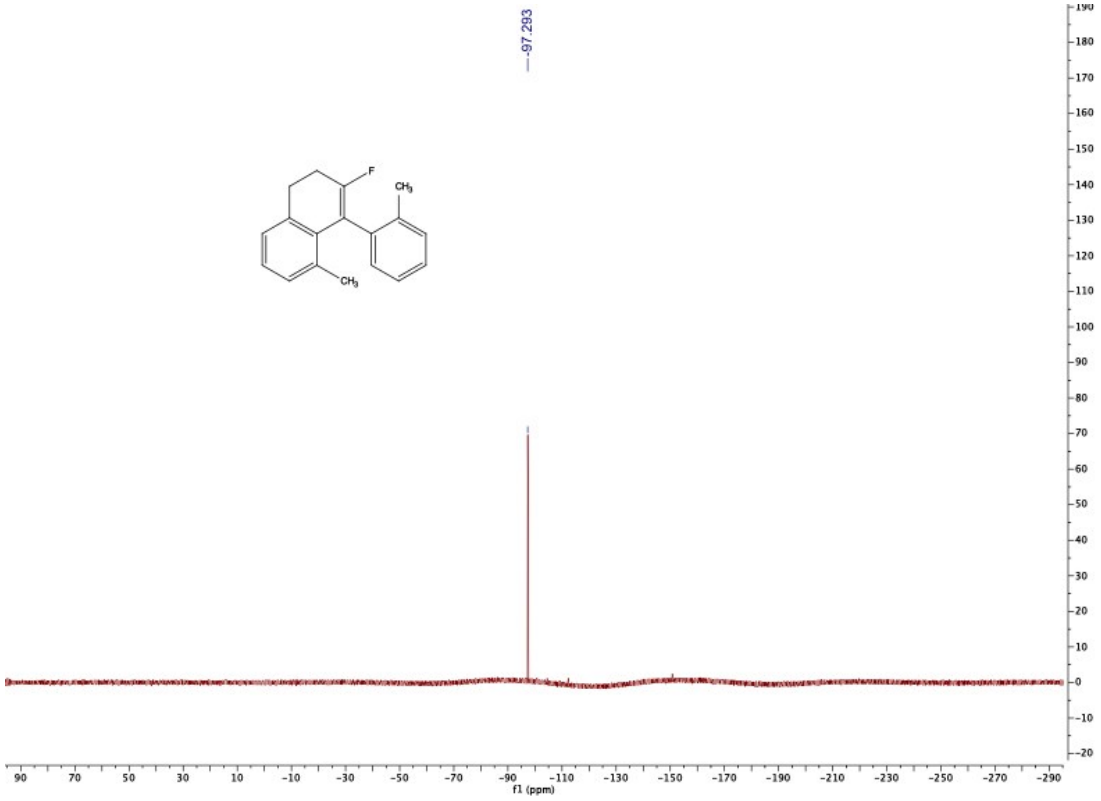
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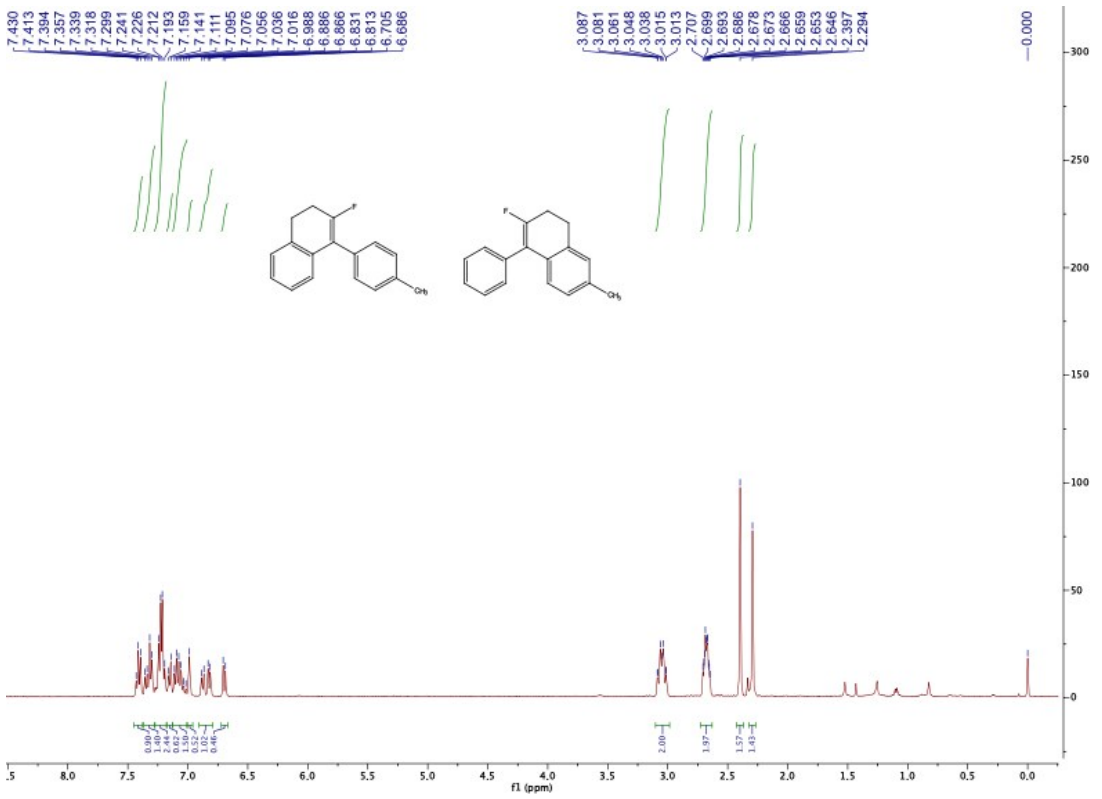
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7.212
7.193
7.159
7.141
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7.095
7.076
7.056
7.036
7.016
6.988
6.886
6.866
6.831
6.813
6.705
6.686
3.087
3.081
3.061
3.048
3.038
3.015
3.013
2.707
2.699
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2.666
2.659
2.653
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2.397
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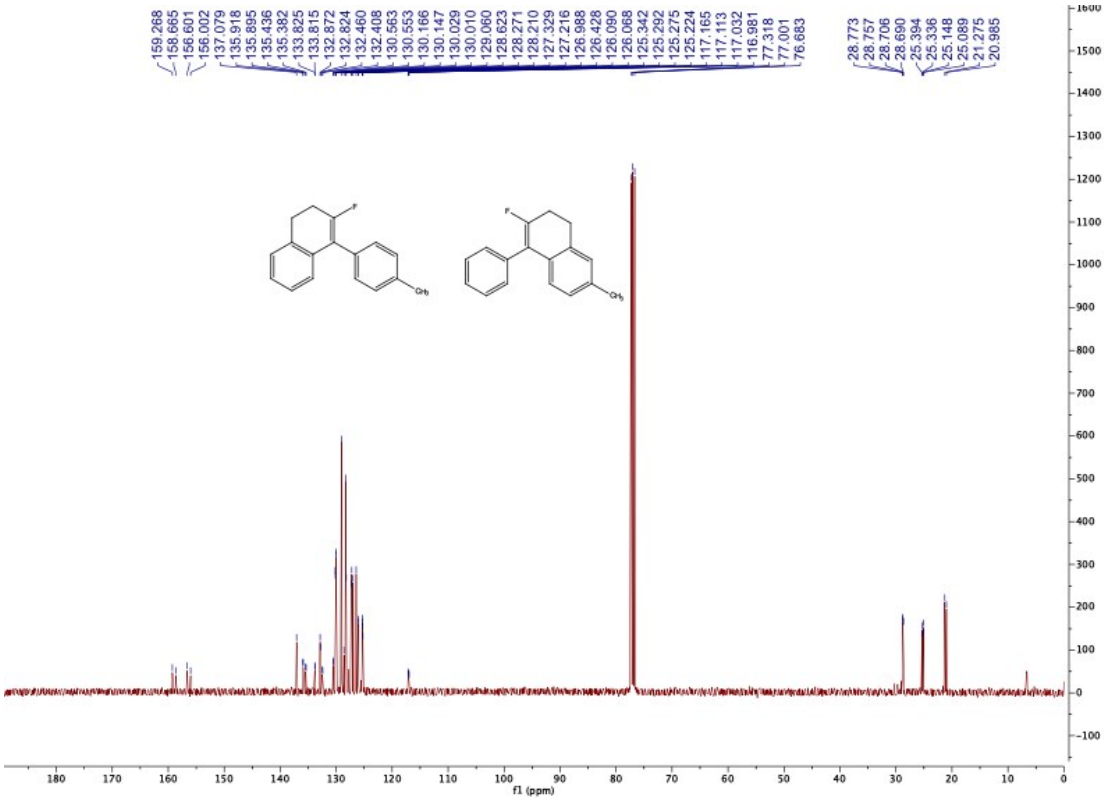
f1 (ppm)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132.460
132.408
130.563
130.553
130.166
130.147
130.029
130.010
129.060
128.623
128.271
128.210
127.329
127.216
126.988
126.428
126.090
126.068
125.342
125.292
125.275
125.224
117.165
117.113
117.032
116.981
77.318
77.001
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28.706
28.690
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25.336
25.148
25.089
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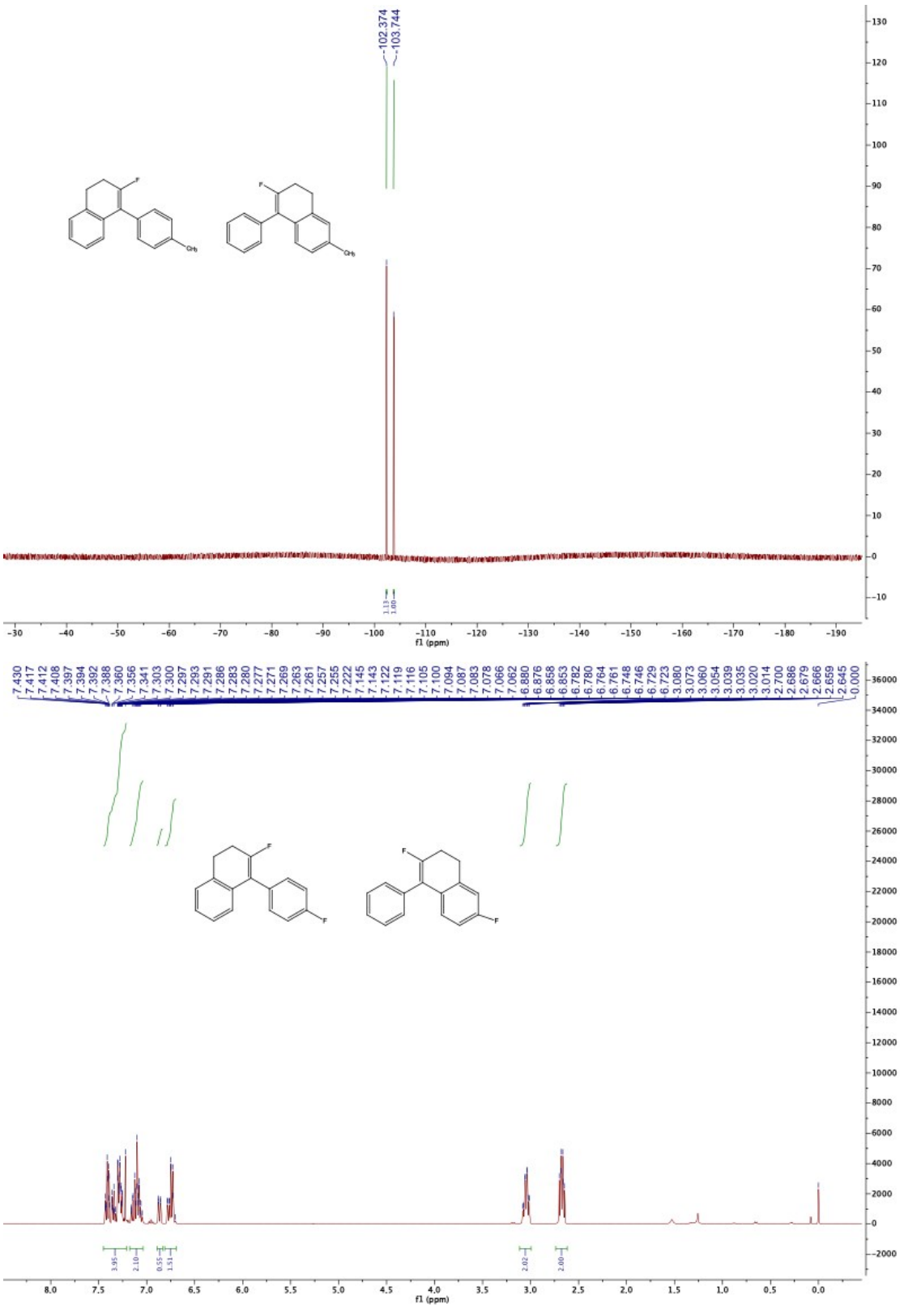
f1 (ppm)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7.303
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7.297
7.293
7.291
7.286
7.283
7.280
7.277
7.271
7.269
7.263
7.261
7.257
7.255
7.222
7.145
7.143
7.122
7.119
7.116
7.105
7.100
7.094
7.087
7.083
7.078
7.066
7.062
6.880
6.876
6.858
6.853
6.782
6.780
6.764
6.761
6.748
6.746
6.729
6.723
3.080
3.073
3.060
3.054
3.039
3.035
3.020
3.014
2.700
2.686
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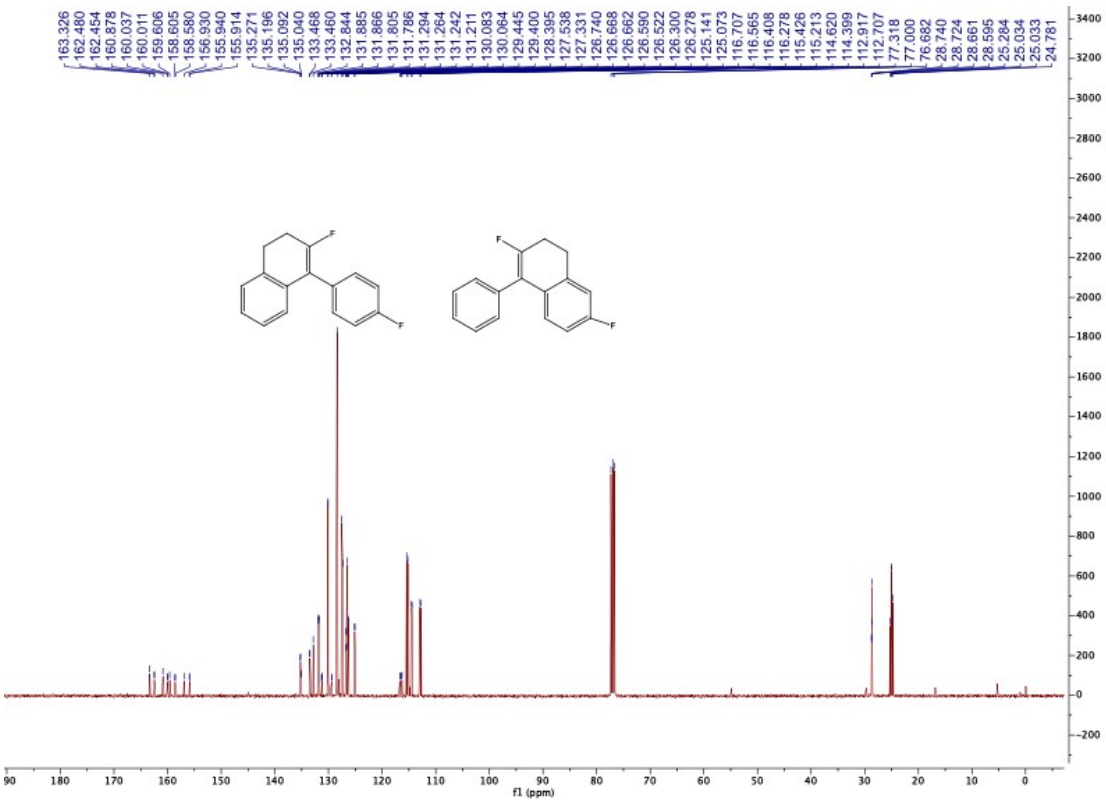
2.00
f1 (ppm)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135.092
135.040
133.468
133.460
132.844
131.885
131.866
131.805
131.786
131.294
131.264
131.242
131.211
130.083
130.064
129.445
129.400
128.395
127.538
127.331
126.740
126.668
126.662
126.590
126.522
126.300
126.278
125.141
125.073
116.707
116.565
116.408
116.278
115.426
115.213
114.620
114.399
112.917
112.707
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28.661
28.595
25.284
25.034
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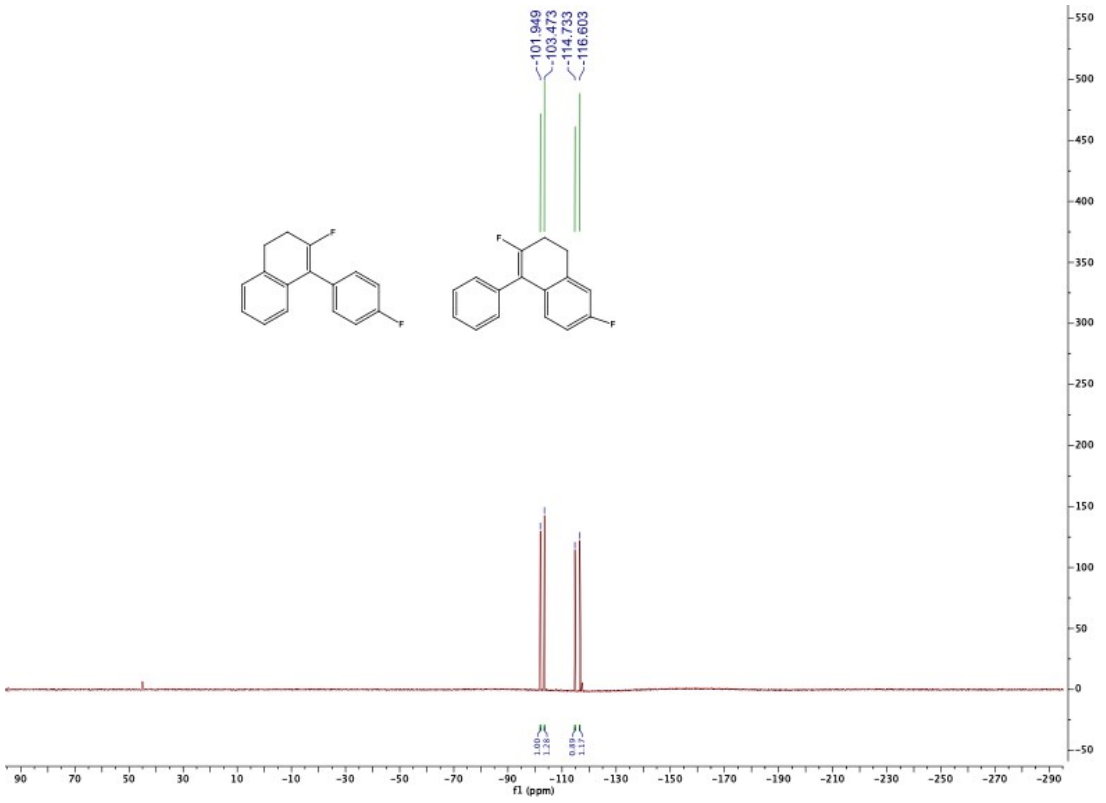
24.781
f1 (ppm)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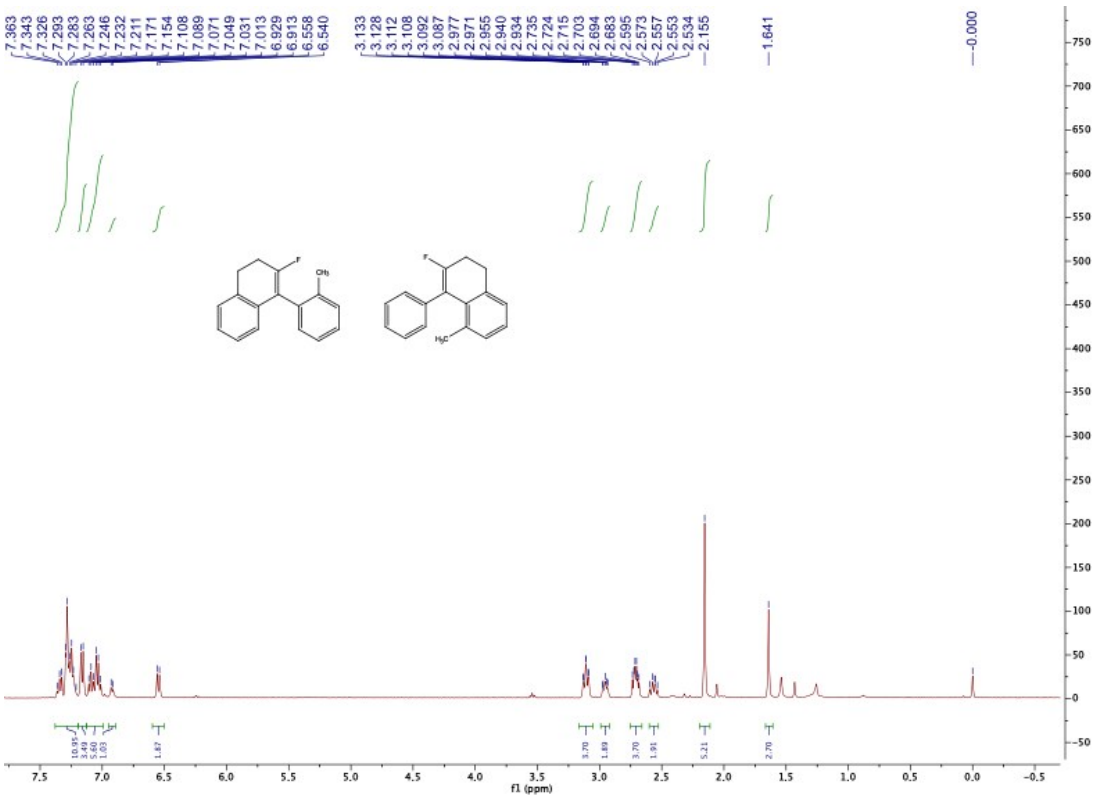
0.89
1.17
f1 (ppm)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7.263
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7.232
7.211
7.171
7.154
7.108
7.089
7.071
7.049
7.031
7.013
6.929
6.913
6.558
6.540
3.133
3.128
3.112
3.108
3.092
3.087
2.977
2.971
2.955
2.940
2.934
2.735
2.724
2.715
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2.573
2.557
2.553
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2.155
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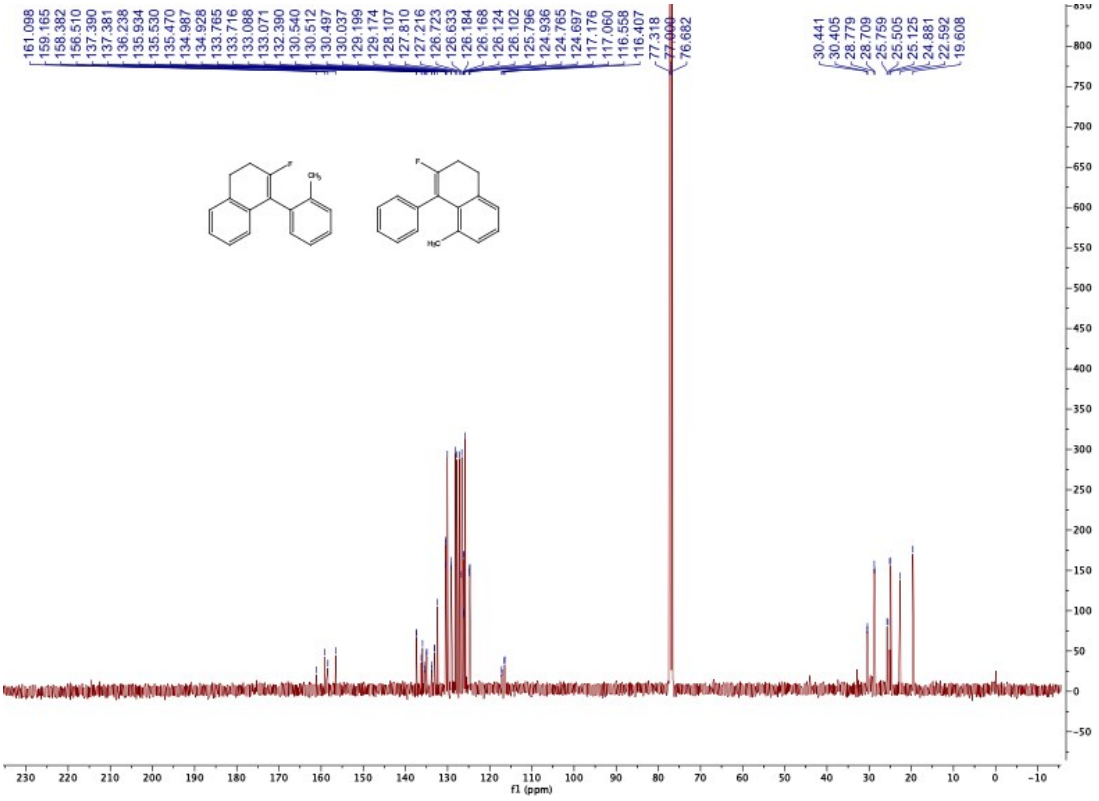
0.000
f1 (ppm)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135.470
134.987
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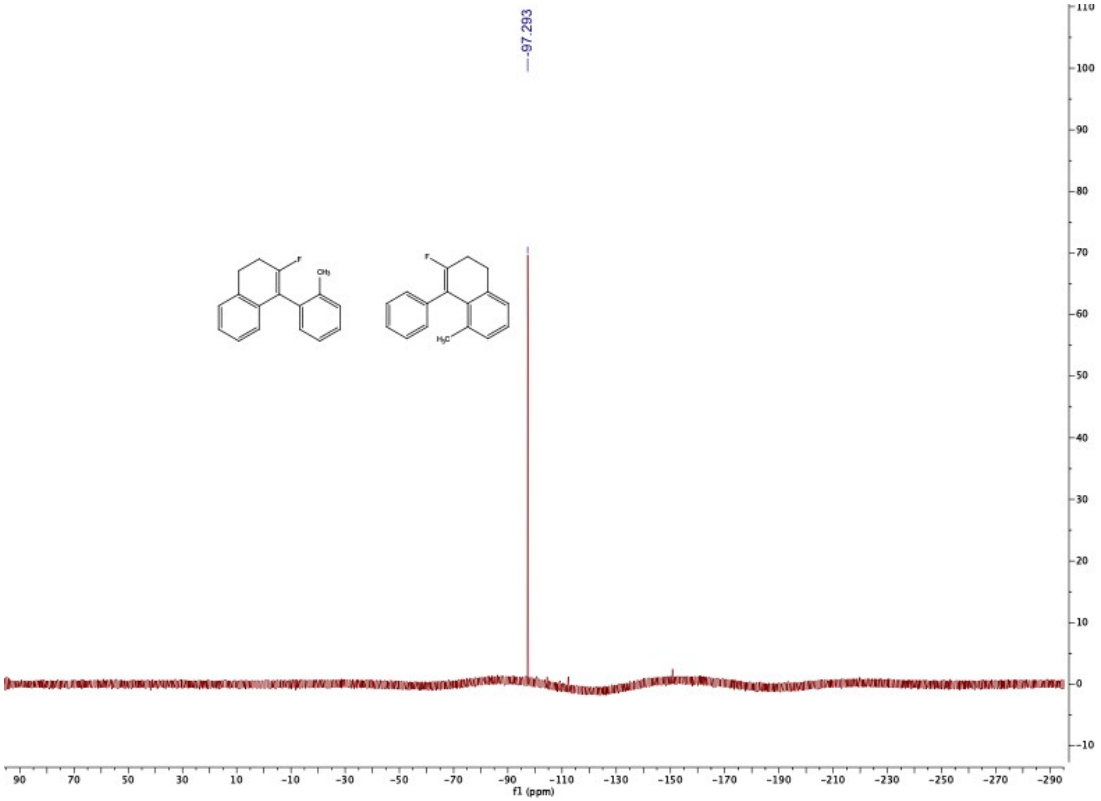
133.765
133.716
133.088
133.071
132.390
130.540
130.512
130.497
130.037
129.199
129.174
128.107
127.810
127.216
126.723
126.633
126.184
126.168
126.124
126.102
125.796
124.936
124.765
124.697
117.176
117.060
116.558
116.407
77.318
77.000
76.682
30.441
30.405
28.779
28.709
25.759
25.505
25.125
24.881
22.592
19.608
f1 (ppm)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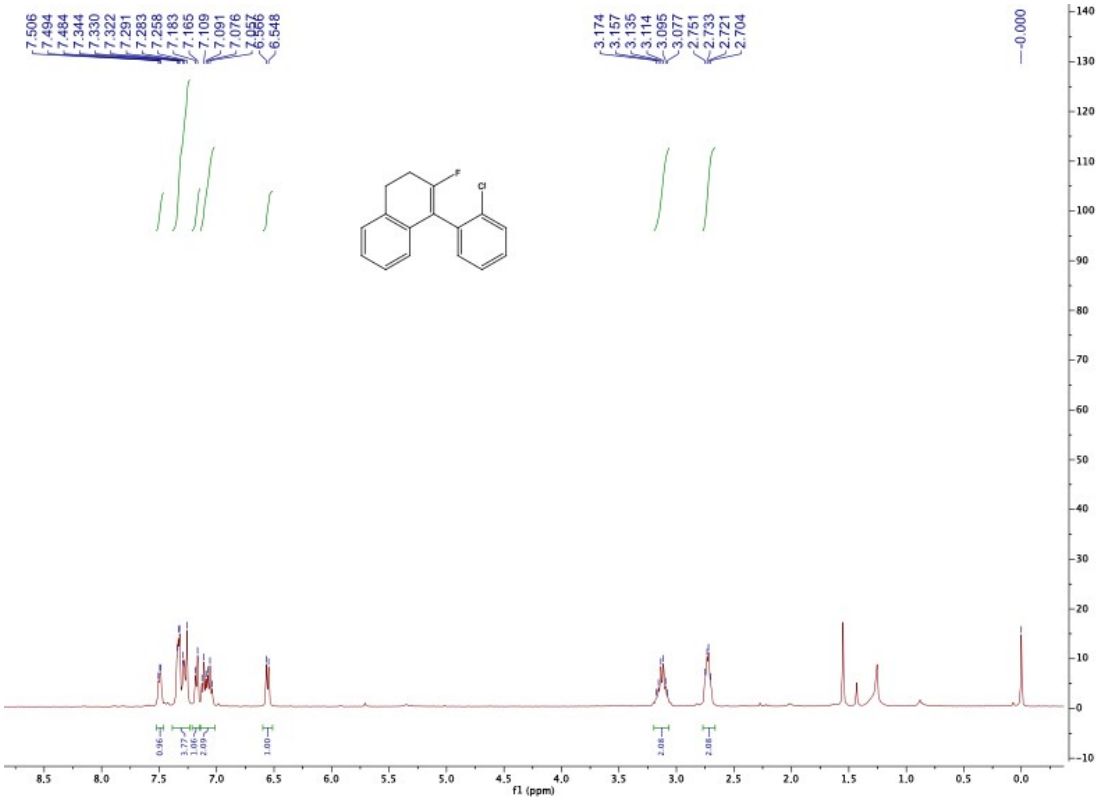
F
CH3
F
H3C
f1 (ppm)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7.484
7.344
7.330
7.322
7.291
7.283
7.258
7.183
7.165
7.109
7.091
7.076
6.556
6.548
3.174
3.157
3.135
3.114
3.095
3.077
2.751
2.733
2.721
2.704
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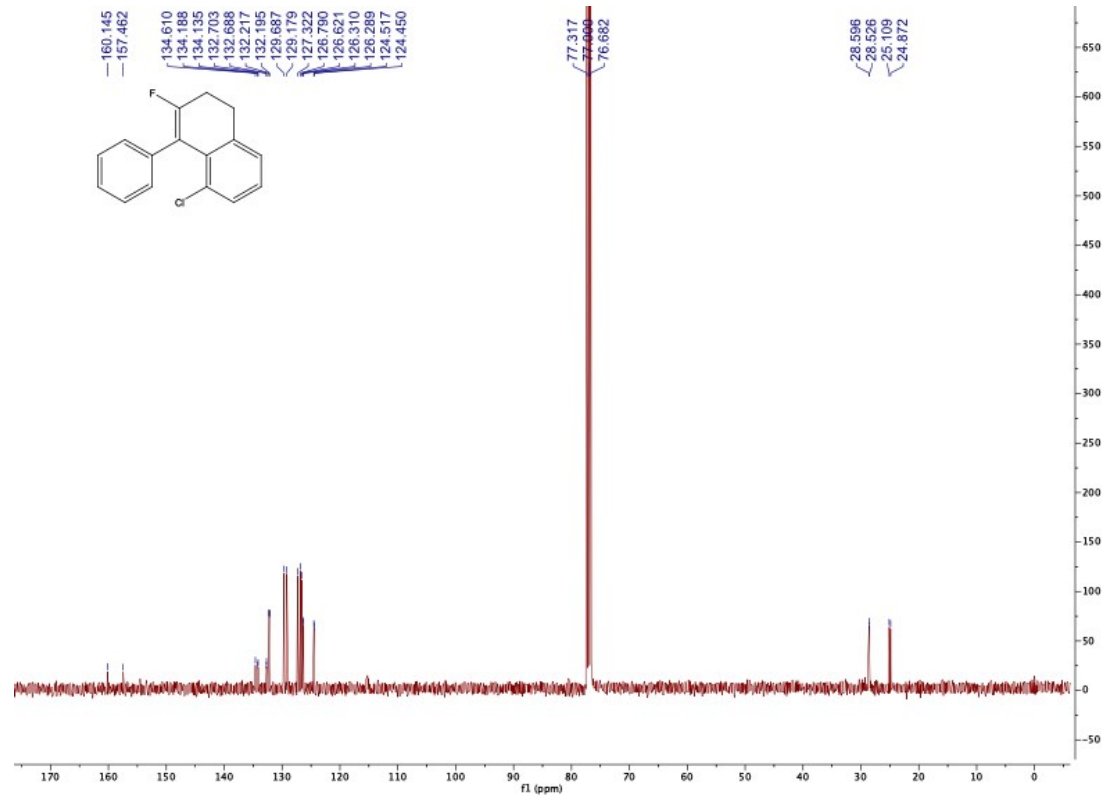
F
Cl
0.96
3.77
1.06
2.09
1.00
2.08
2.08
f1 (ppm)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132.703
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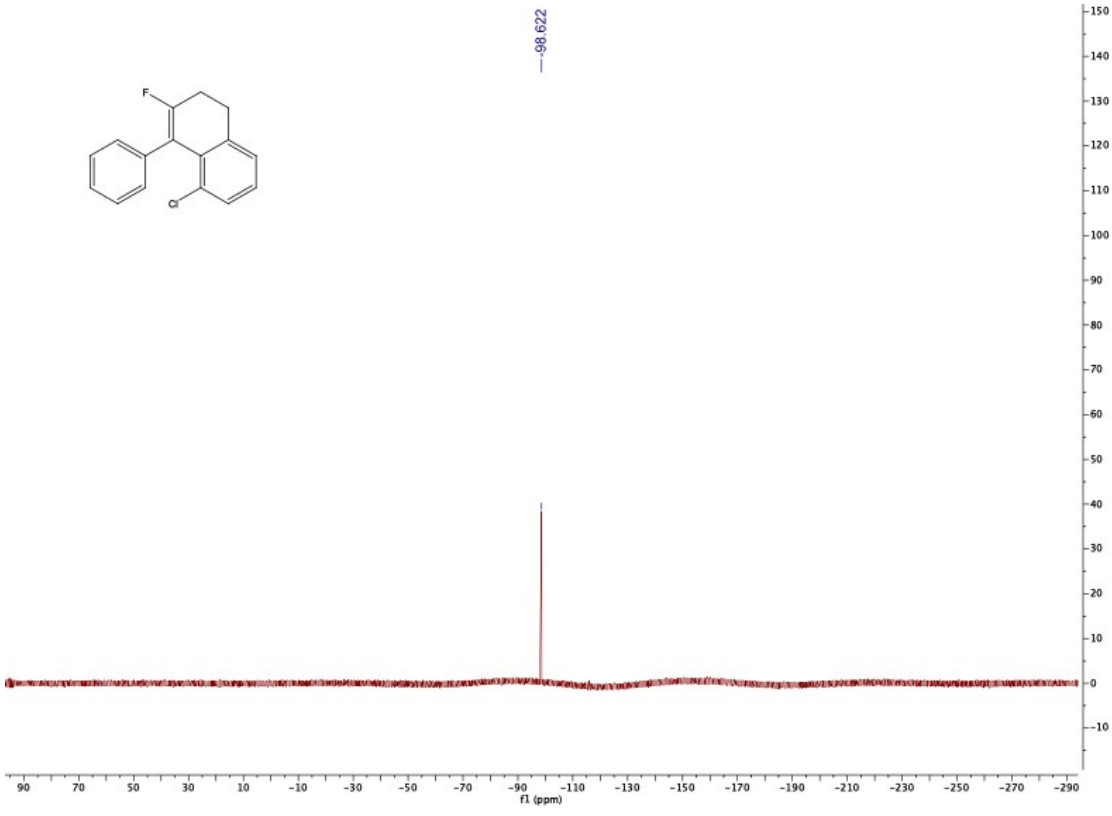
132.217
132.195
129.687
129.179
127.322
126.790
126.621
126.310
126.289
124.517
124.450
77.317
77.000
76.682
28.596
28.526
25.109
24.872
F
Cl
f1 (ppm)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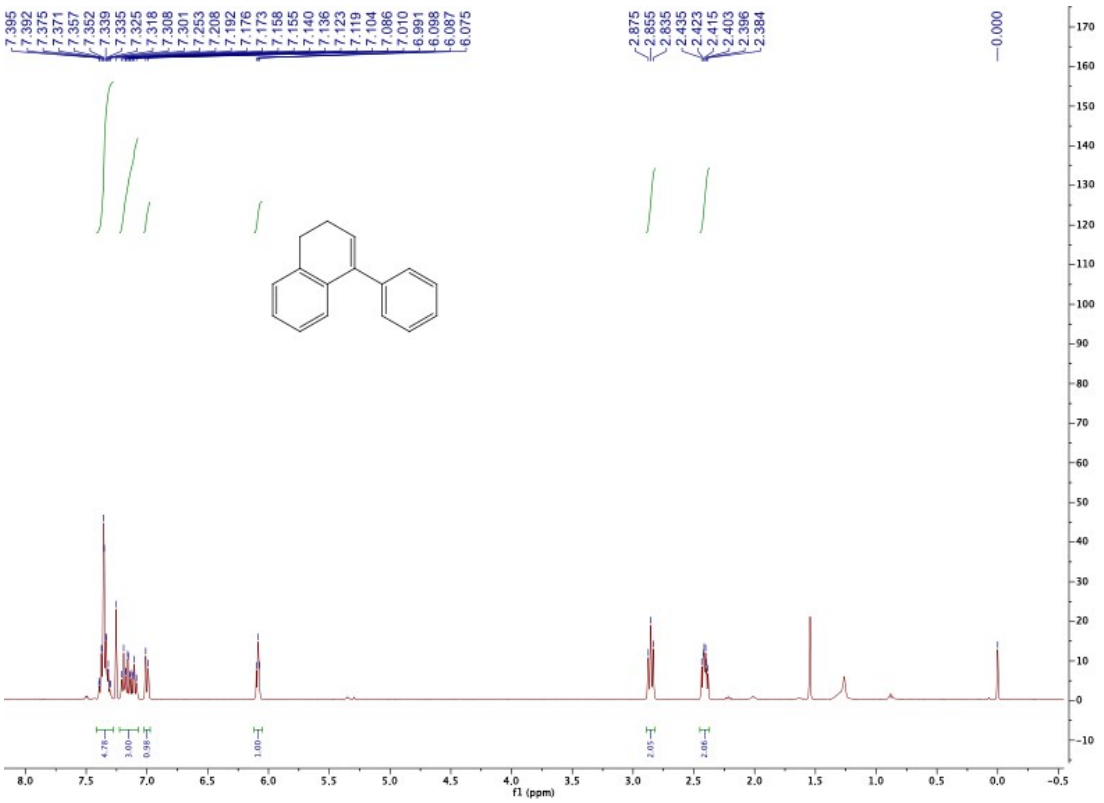
F
Cl
f1 (ppm)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7.339
7.335
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7.318
7.308
7.301
7.253
7.208
7.192
7.176
7.173
7.158
7.155
7.140
7.136
7.123
7.119
7.104
7.086
7.010
6.991
6.098
6.087
6.075
2.875
2.855
2.835
2.435
2.423
2.415
2.403
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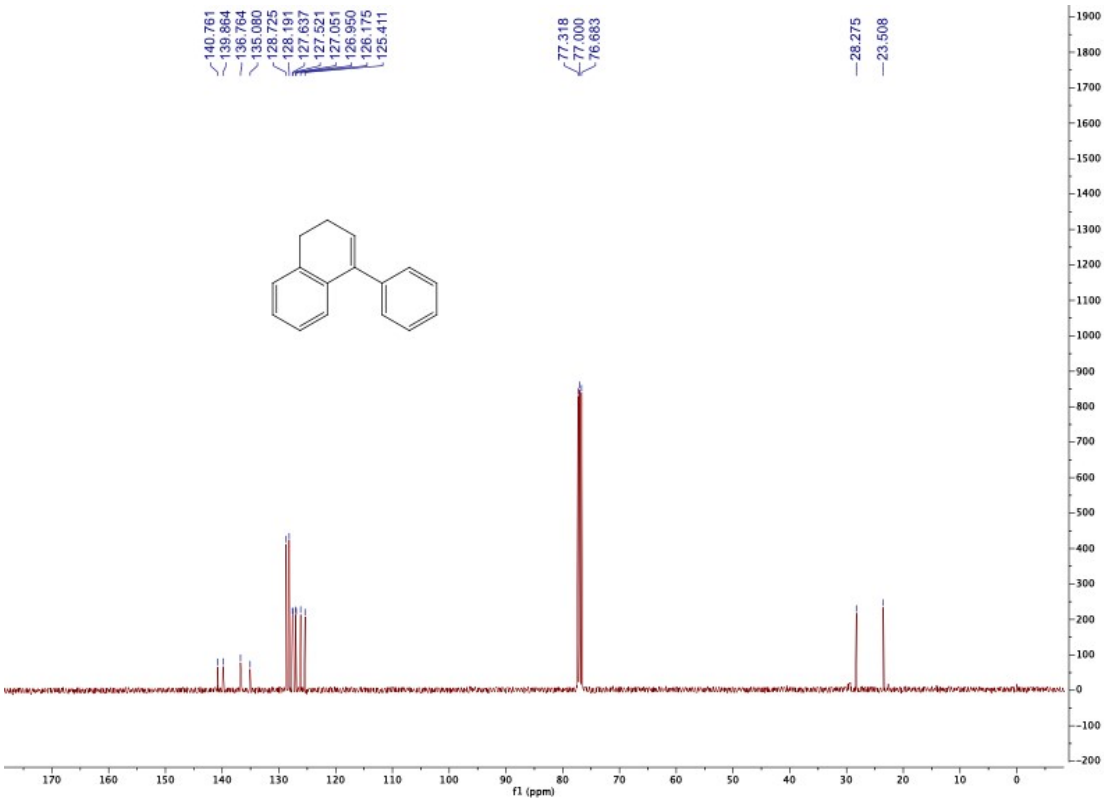
2.384
0.000
4.78
3.00
0.98
1.00
2.05
2.06
f1 (ppm)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139.864
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128.725
128.191
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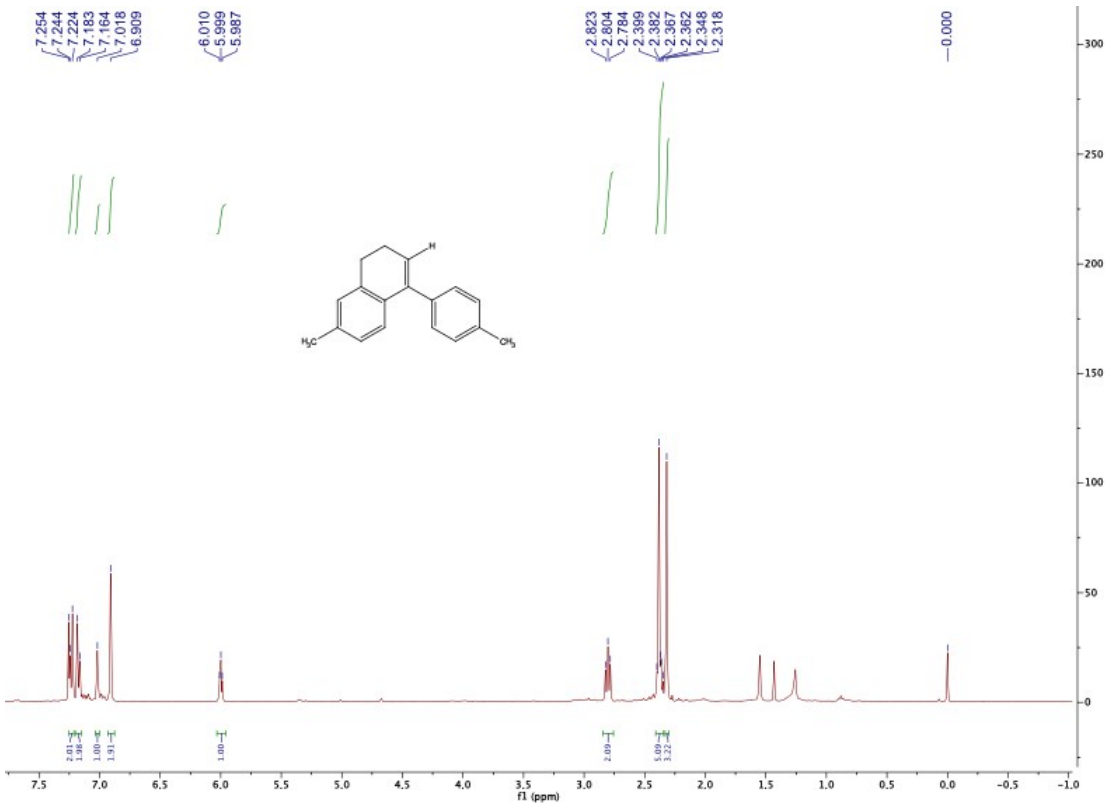
127.521
127.051
126.950
126.175
125.411
77.318
77.000
76.683
28.275
23.508
f1 (ppm)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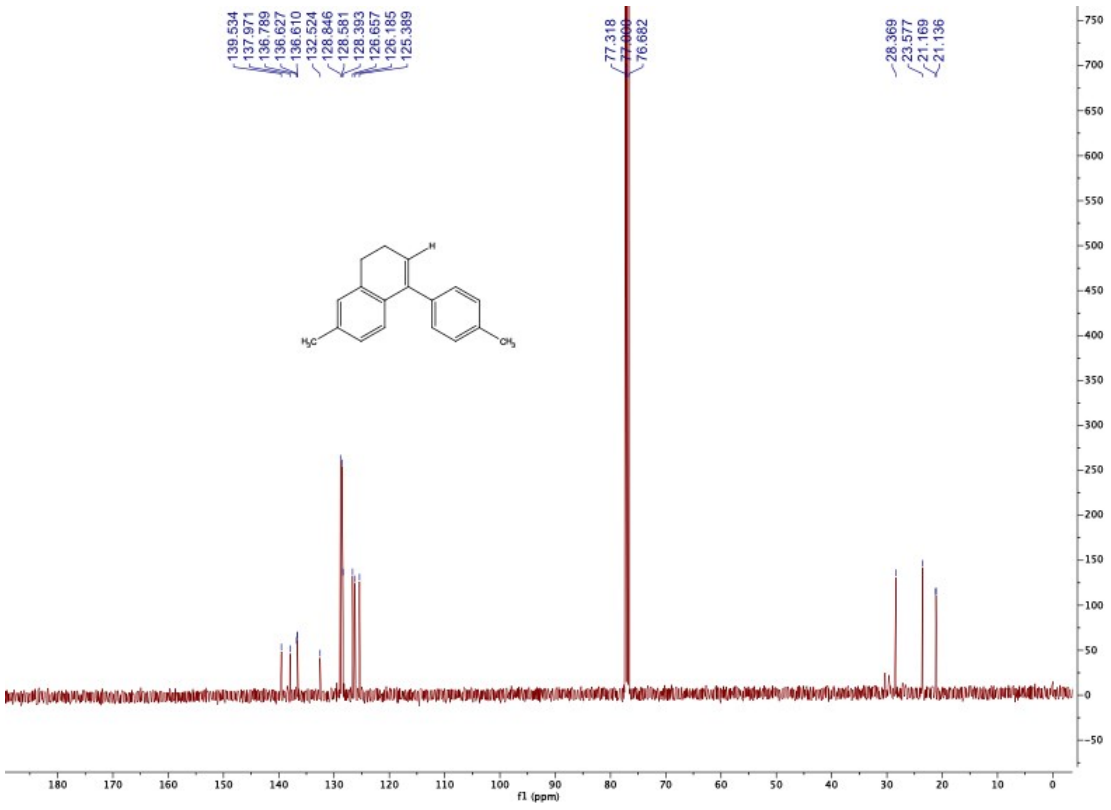
7.244
7.224
7.183
7.164
7.018
6.909
6.010
5.999
5.987
2.823
2.804
2.784
2.399
2.382
2.367
2.362
2.348
2.318
0.000
H
H3C
CH3
f1 (ppm)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136.789
136.627
136.610
132.524
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128.581
128.393
126.657
126.185
125.389
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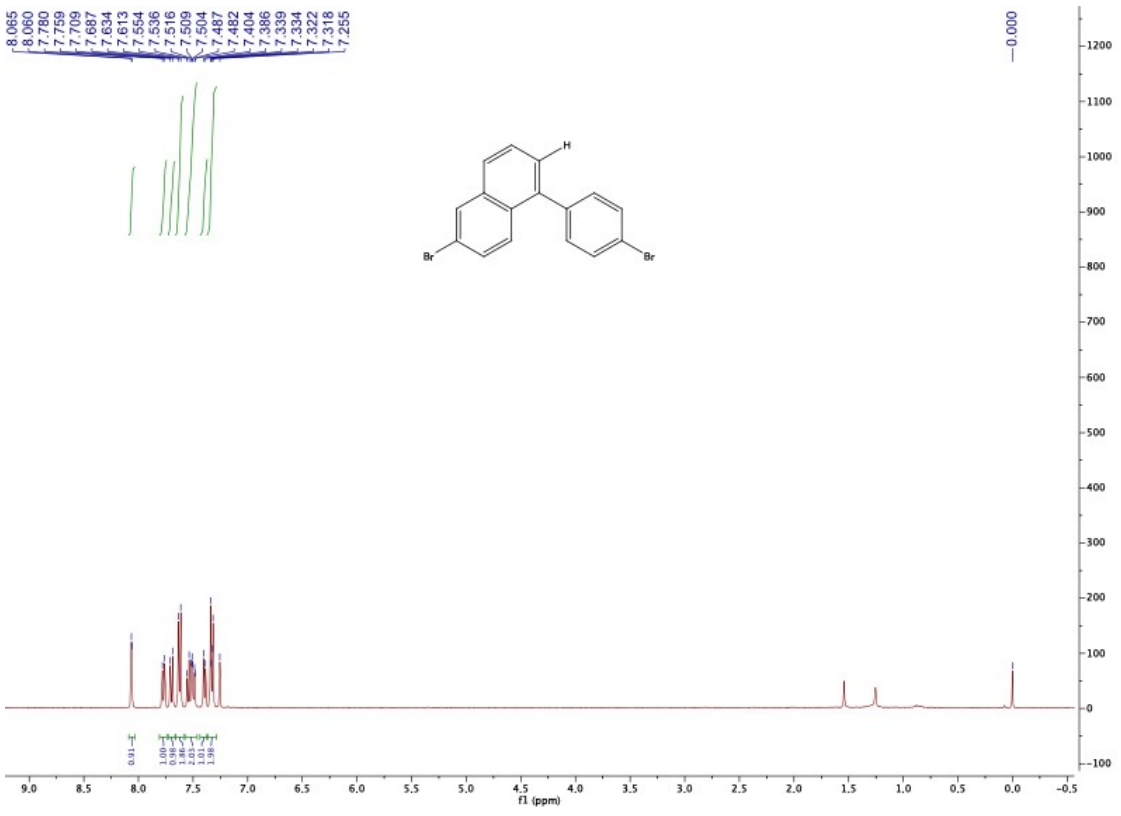
77.000
76.682
28.369
23.577
21.169
21.136
H
H3C
CH3
f1 (ppm)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8.060
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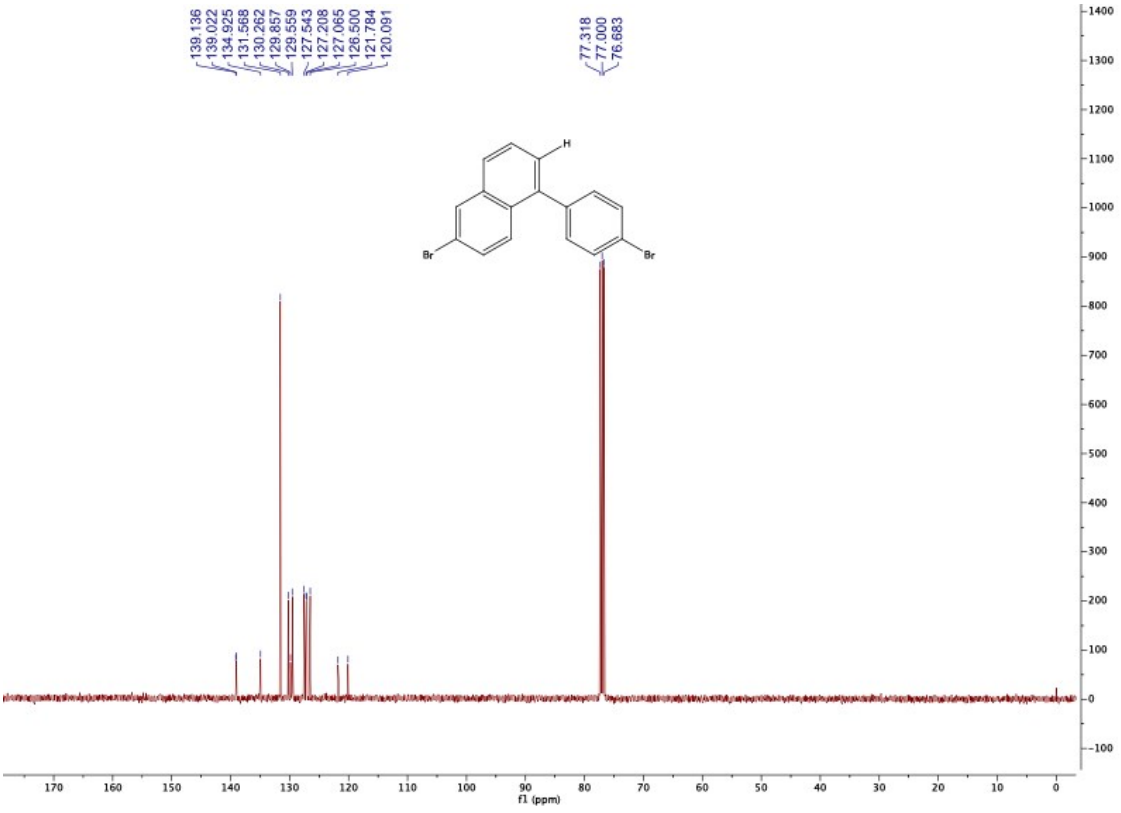
7.759
7.709
7.687
7.634
7.613
7.554
7.536
7.516
7.509
7.504
7.487
7.482
7.404
7.386
7.339
7.334
7.322
7.318
7.255
0.000
H
Br
Br
f1 (ppm)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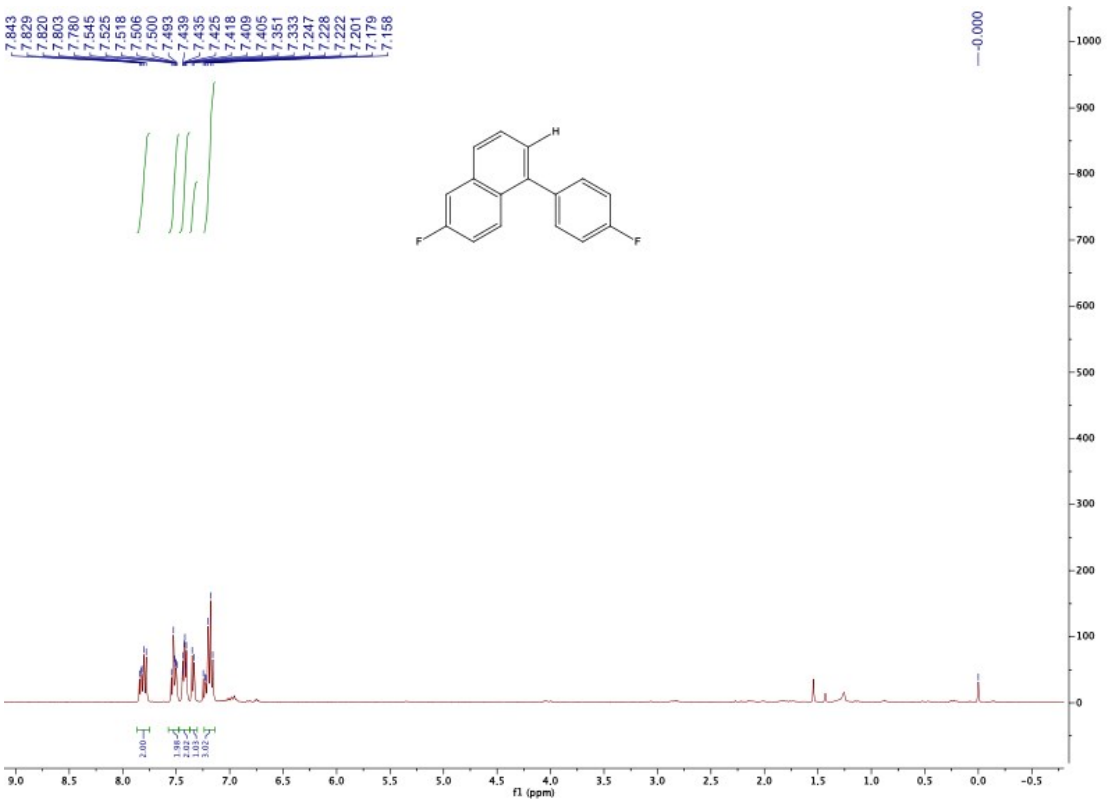
129.559
127.543
127.208
127.065
126.500
121.784
120.091
77.318
77.000
76.683
H
Br
Br
f1 (ppm)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7.500
7.493
7.439
7.435
7.425
7.418
7.409
7.405
7.351
7.333
7.247
7.228
7.222
7.201
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H
F
F
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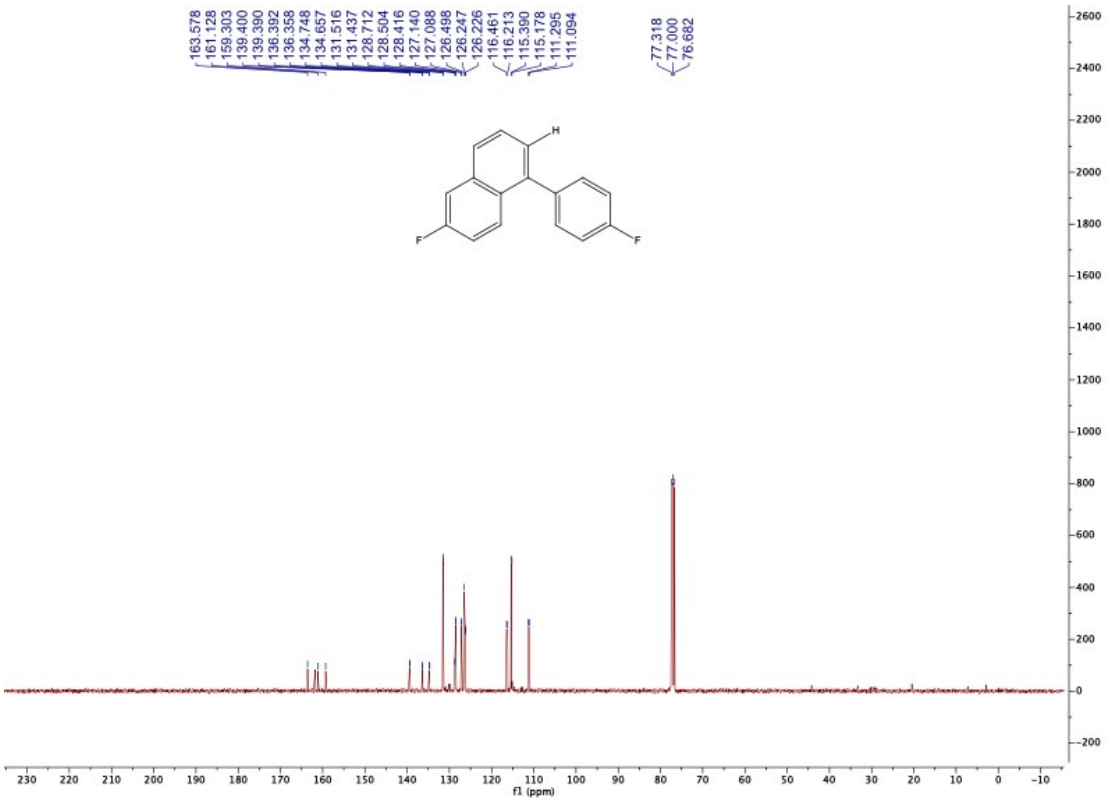
2.02
1.03
3.02
f1 (ppm)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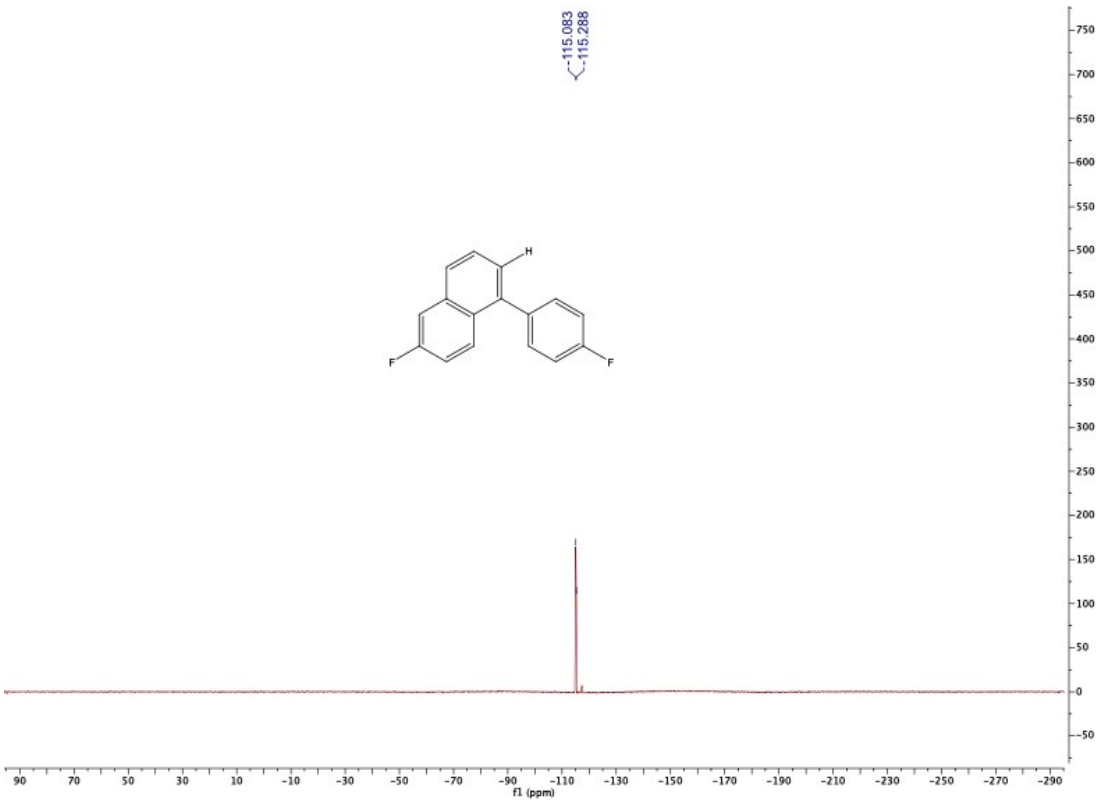
131.516
131.437
128.712
128.504
128.416
127.140
127.088
126.498
126.247
126.226
116.461
116.213
115.390
115.178
111.295
111.094
77.318
77.000
76.682
H
F
F
f1 (ppm)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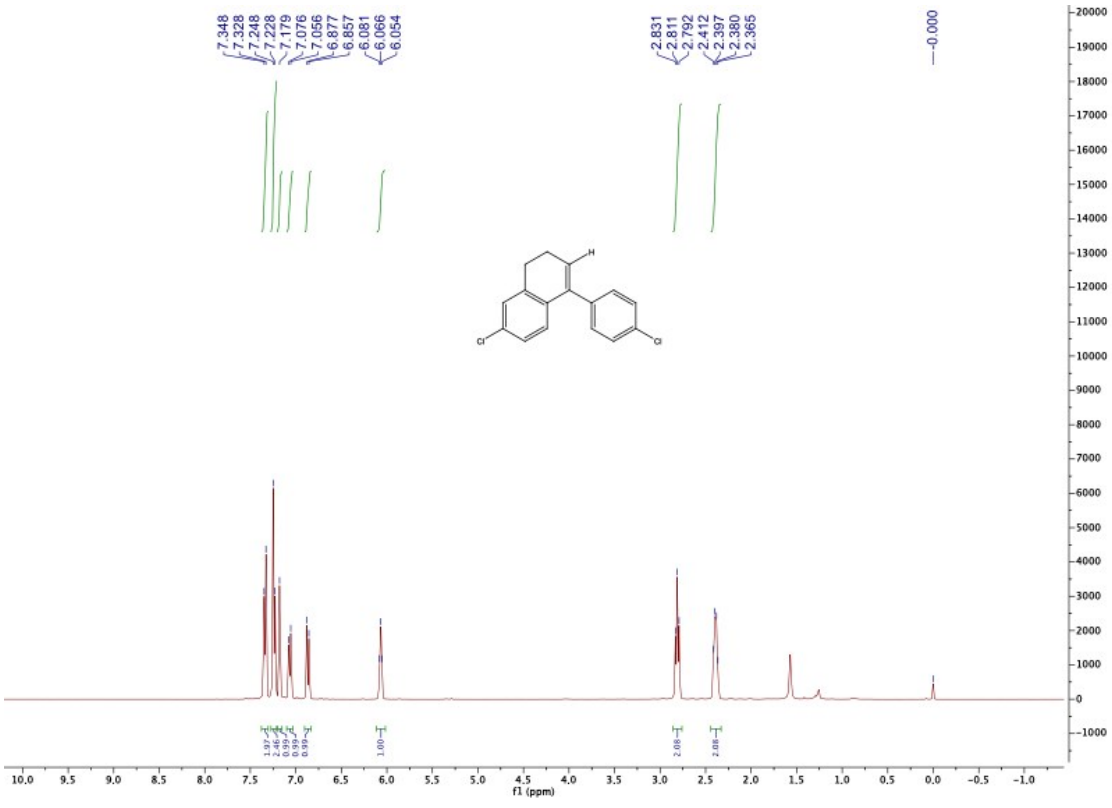
-115.288
H
F
F
f1 (ppm)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7.328
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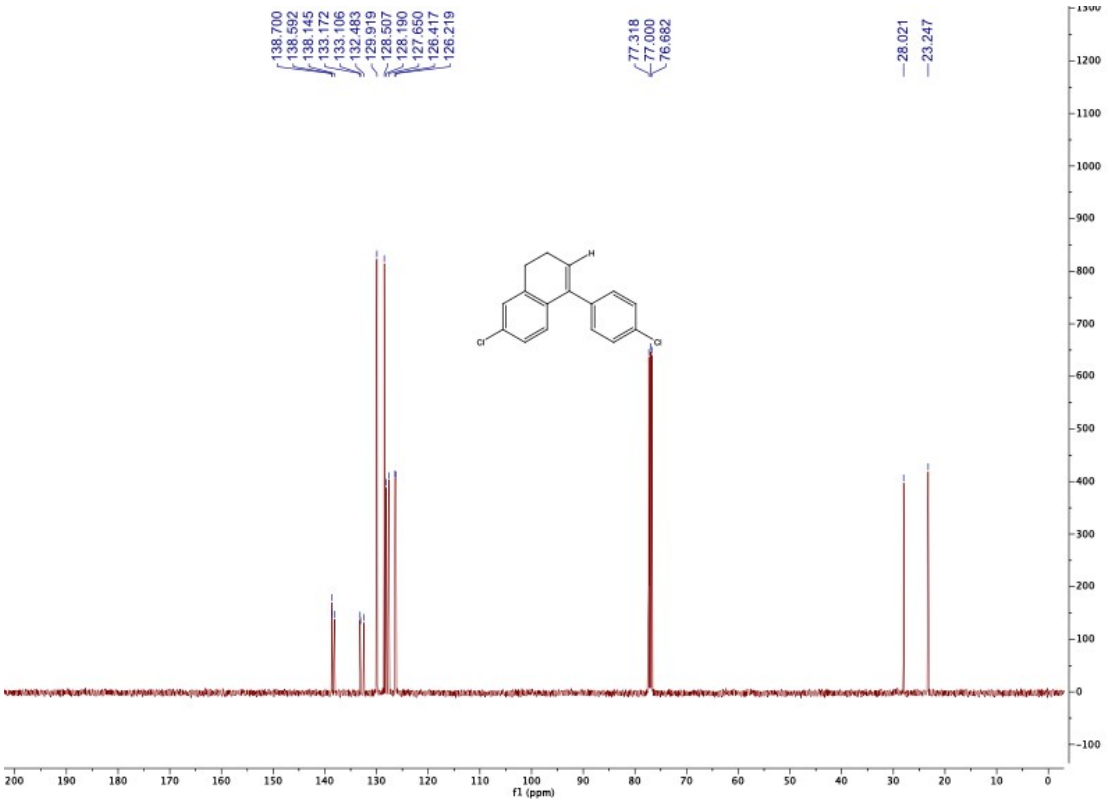
7.179
7.076
7.056
6.877
6.857
6.081
6.066
6.054
2.831
2.811
2.792
2.412
2.397
2.380
2.365
0.000
H
Cl
Cl
f1 (ppm)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138.592
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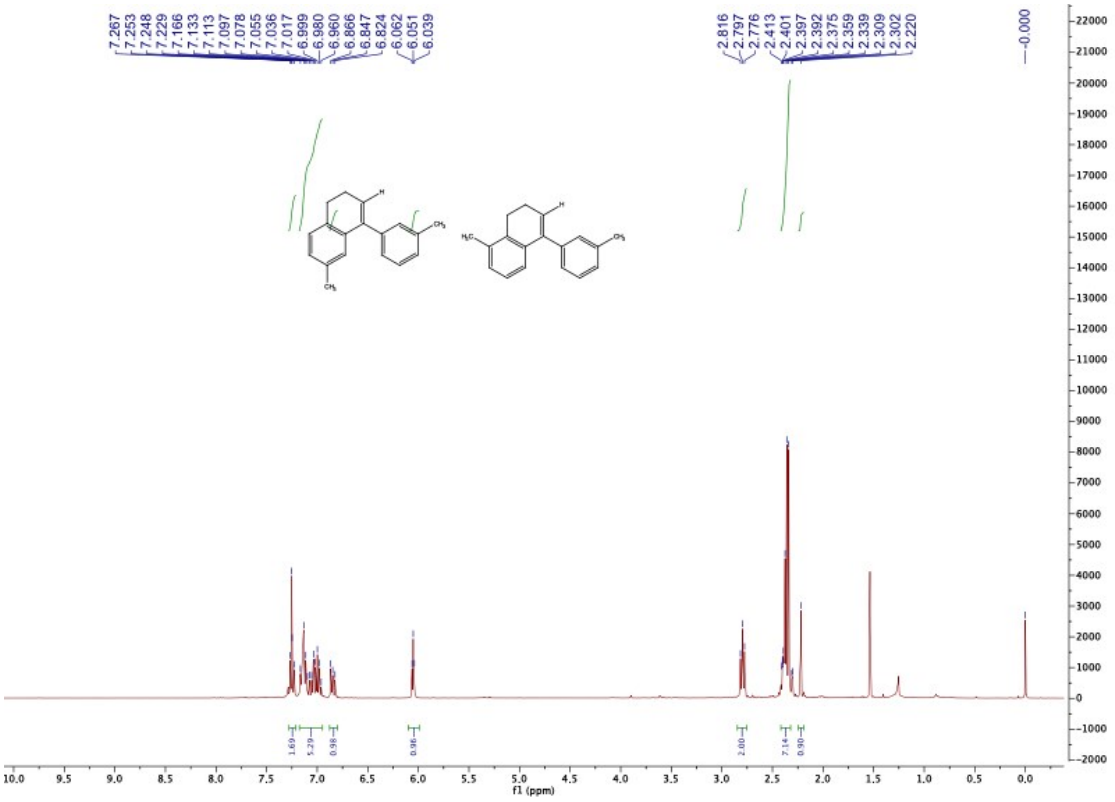
132.483
129.919
128.507
128.190
127.650
126.417
126.219
77.318
77.000
76.682
28.021
23.247
f1 (ppm)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7.133
7.113
7.097
7.078
7.055
7.036
7.017
6.999
6.980
6.960
6.866
6.847
6.824
6.062
6.051
6.039
2.816
2.797
2.776
2.413
2.401
2.397
2.392
2.375
2.359
2.339
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2.00
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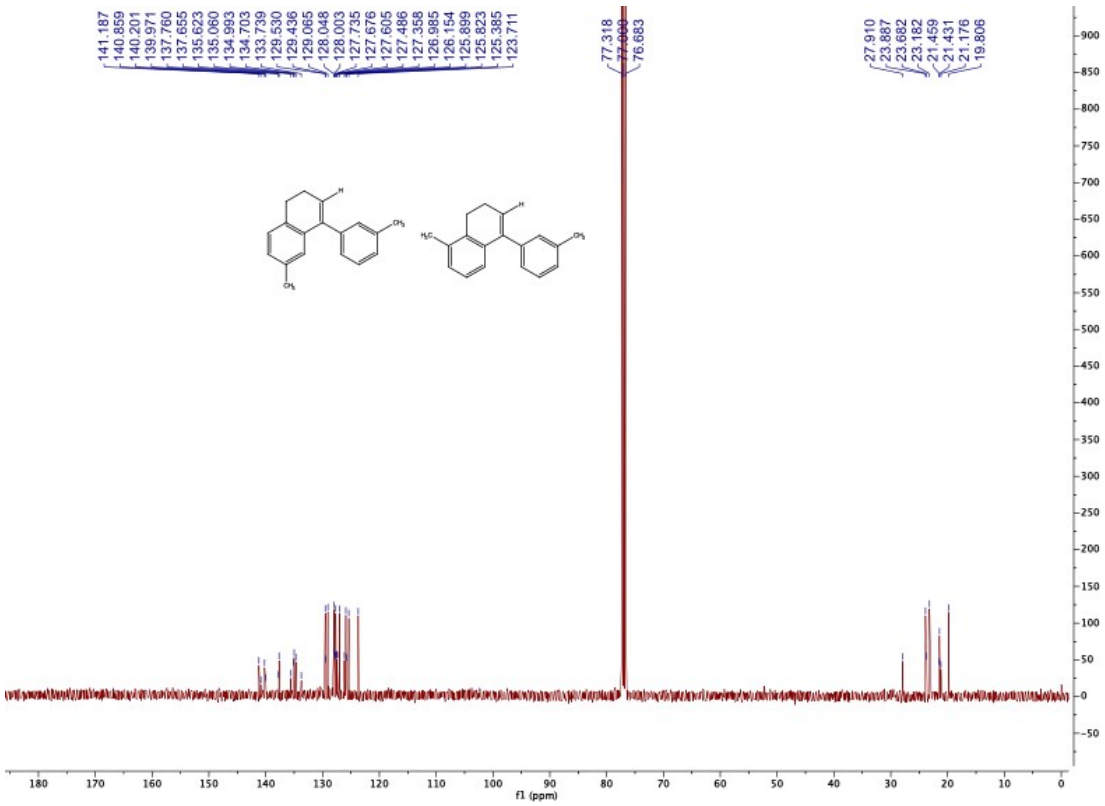
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129.436
129.065
128.048
128.003
127.735
127.676
127.605
127.486
127.358
126.985
126.154
125.899
125.823
125.385
123.711
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27.910
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23.682
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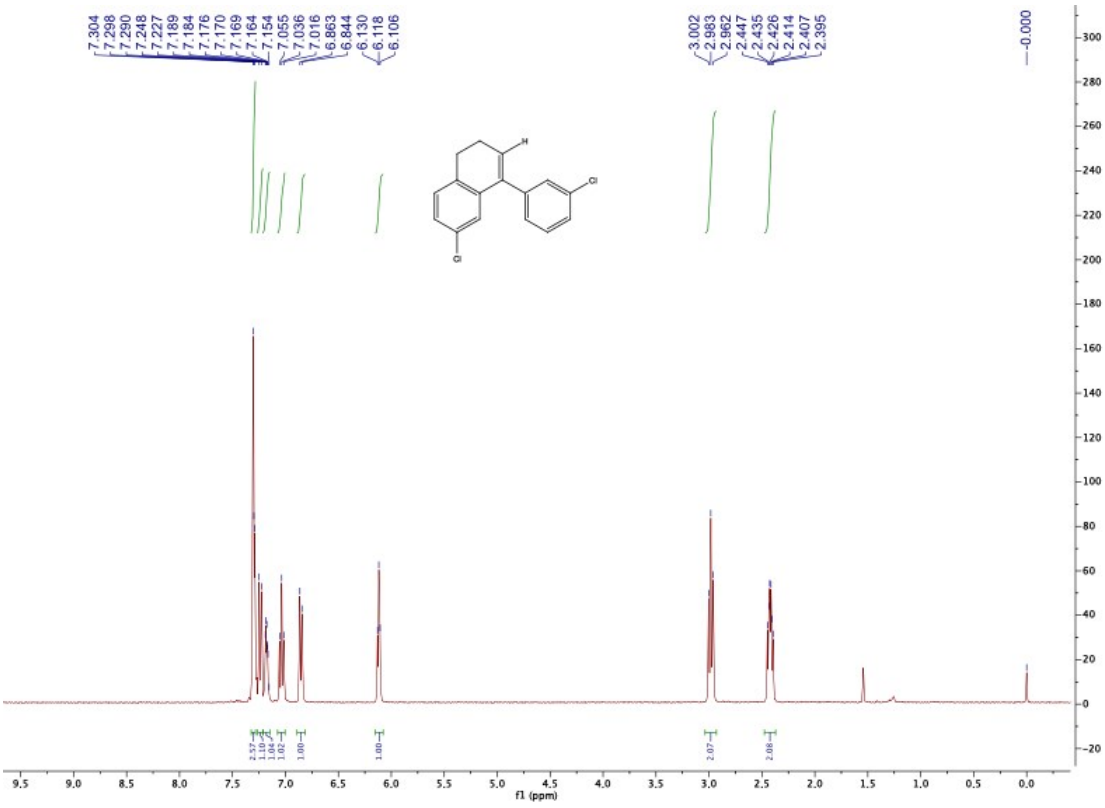
21.431
21.176
19.806
f1 (ppm)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7.184
7.176
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7.169
7.164
7.154
7.055
7.036
7.016
6.863
6.844
6.130
6.118
6.106
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2.447
2.435
2.426
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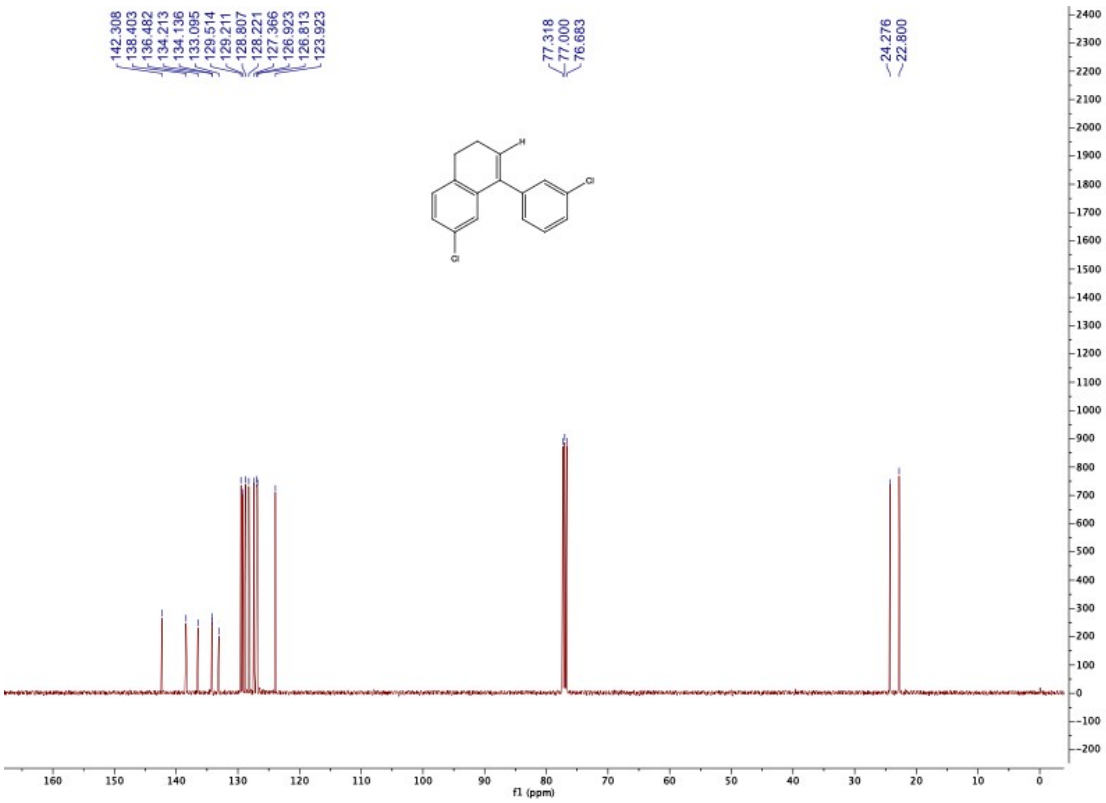
2.407
2.395
-0.000
f1 (ppm)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134.213
134.136
133.095
129.514
129.211
128.807
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126.923
126.813
123.923
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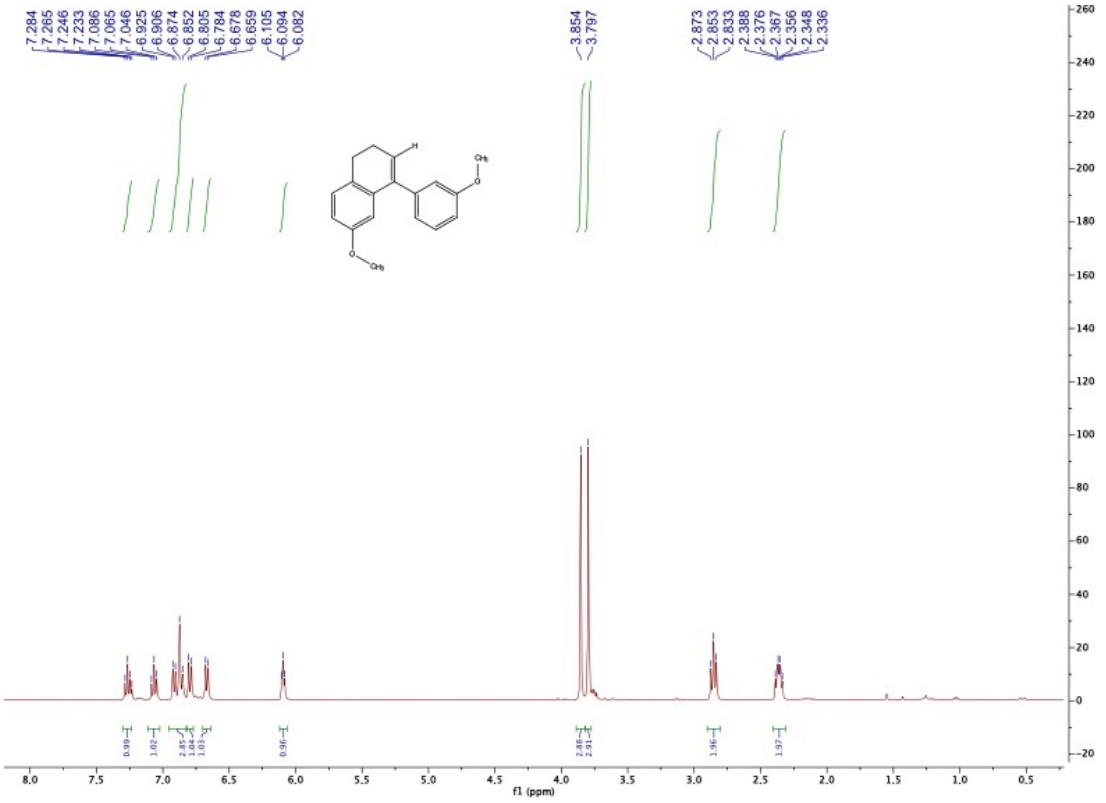
77.000
76.683
24.276
22.800
f1 (ppm)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7.233
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6.925
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6.874
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6.678
6.659
6.105
6.094
6.082
3.854
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2.376
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2.356
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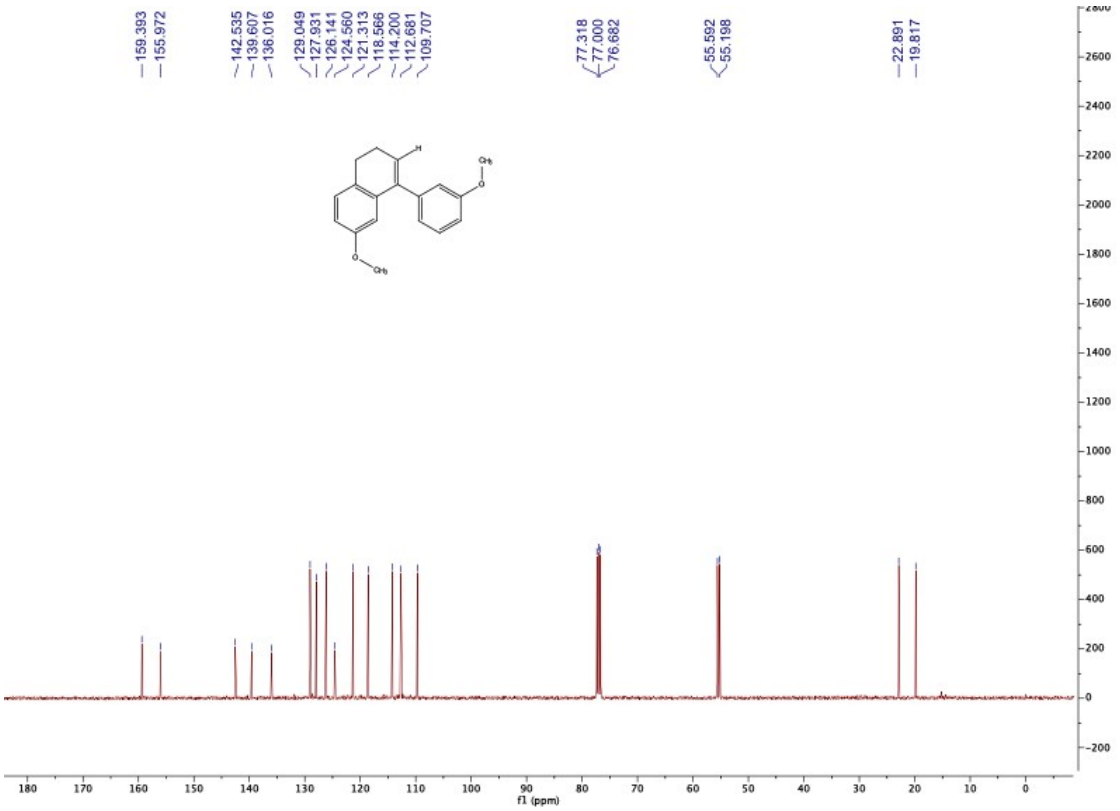
2.336
f1 (ppm)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127.931
126.141
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121.313
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55.198
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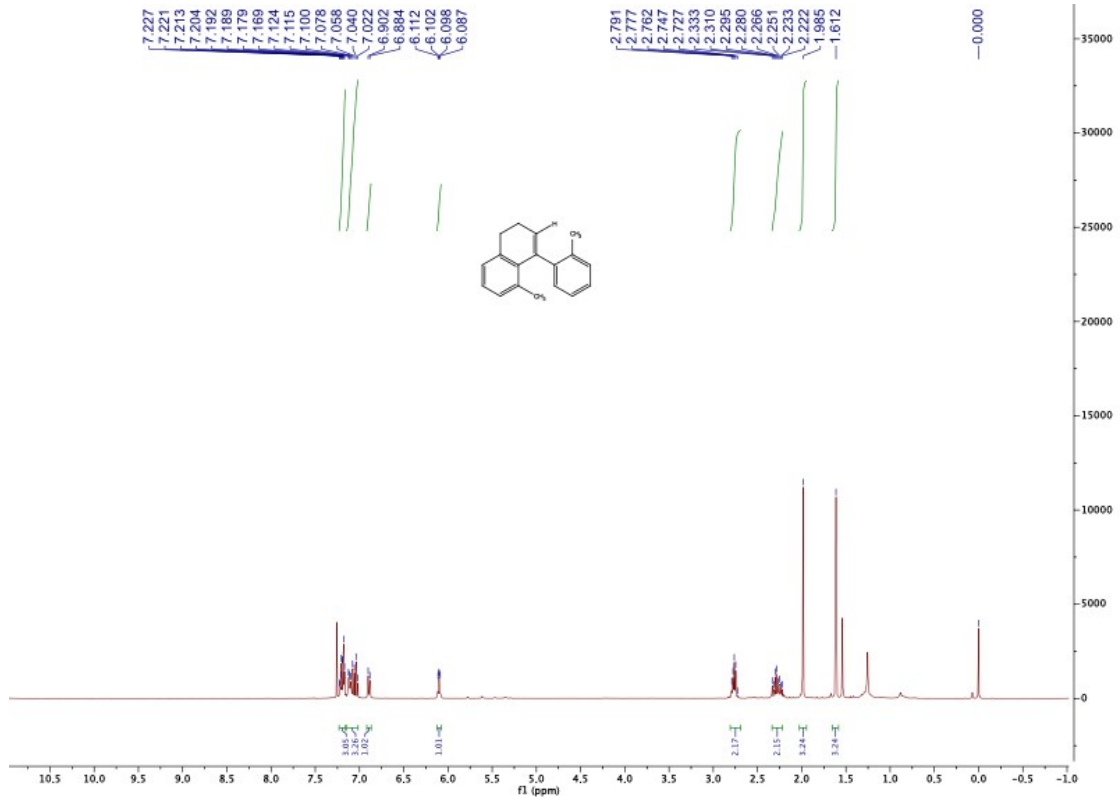
19.817
f1 (ppm)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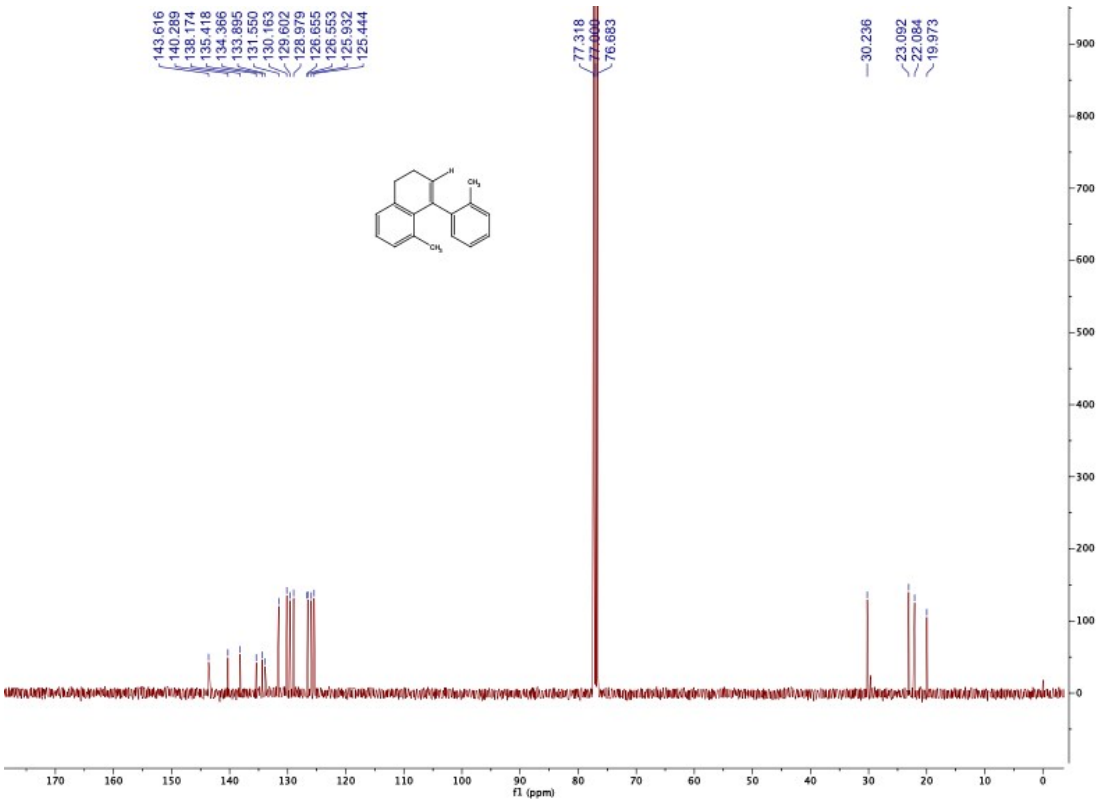
7.040
7.022
6.902
6.884
6.112
6.102
6.098
6.087
2.791
2.777
2.762
2.747
2.727
2.333
2.310
2.295
2.280
2.266
2.251
2.233
2.222
1.985
1.612
0.000
f1 (ppm)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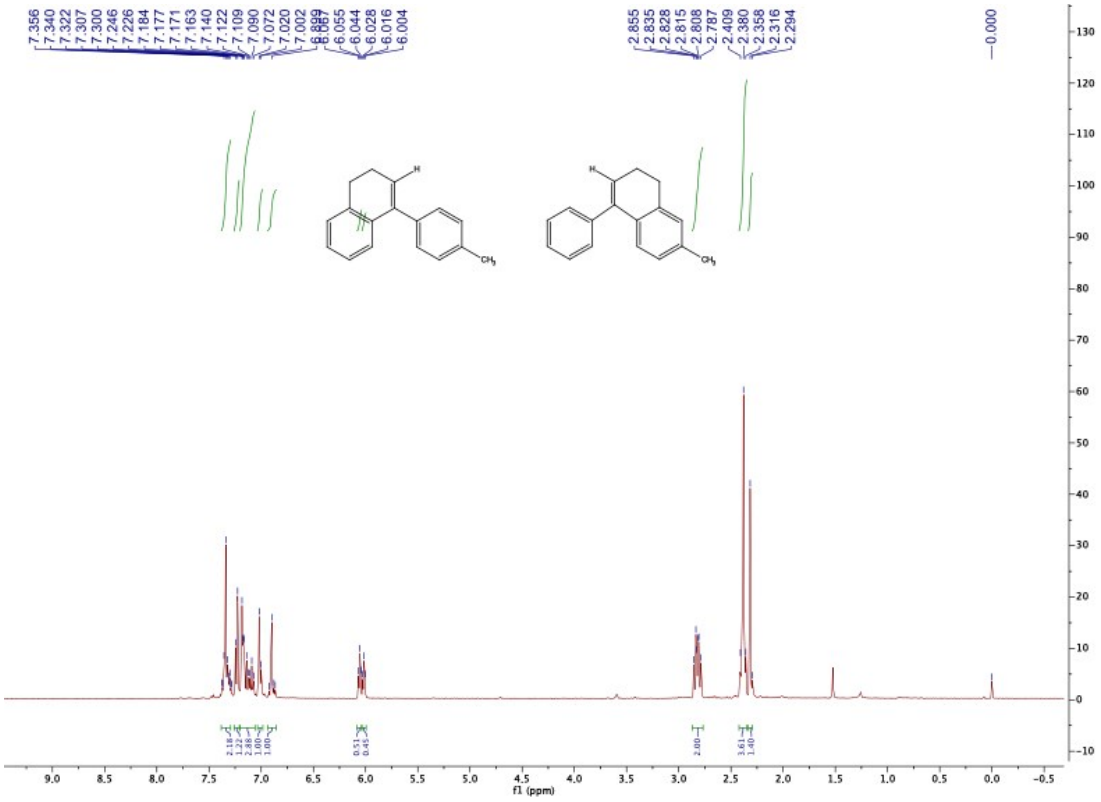
140.289
138.174
135.418
134.366
133.895
131.550
130.163
129.602
128.979
126.655
126.553
125.932
125.444
77.318
77.000
76.683
30.236
23.092
22.084
19.973
f1 (ppm)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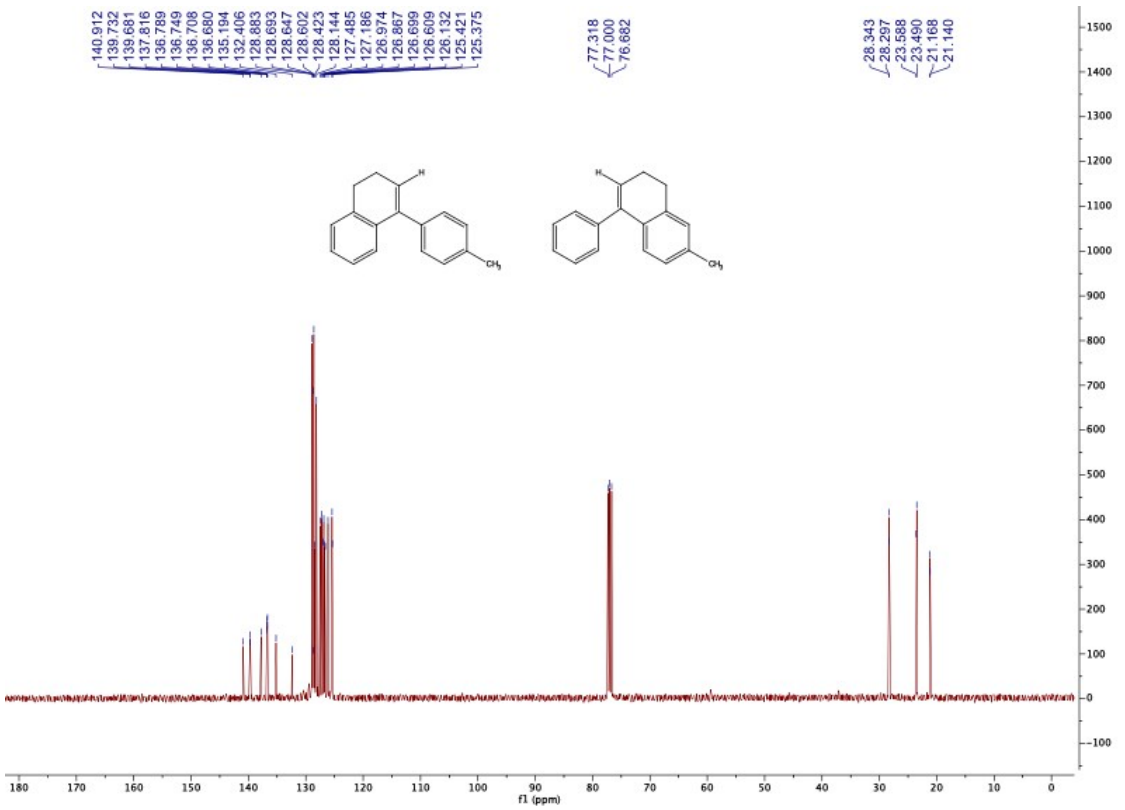
7.340
7.322
7.307
7.300
7.246
7.226
7.184
7.177
7.171
7.163
7.140
7.122
7.109
7.090
7.072
7.020
7.002
6.055
6.044
6.028
6.016
6.004
2.855
2.835
2.828
2.815
2.808
2.787
2.409
2.380
2.358
2.316
2.294
0.000
f1 (ppm)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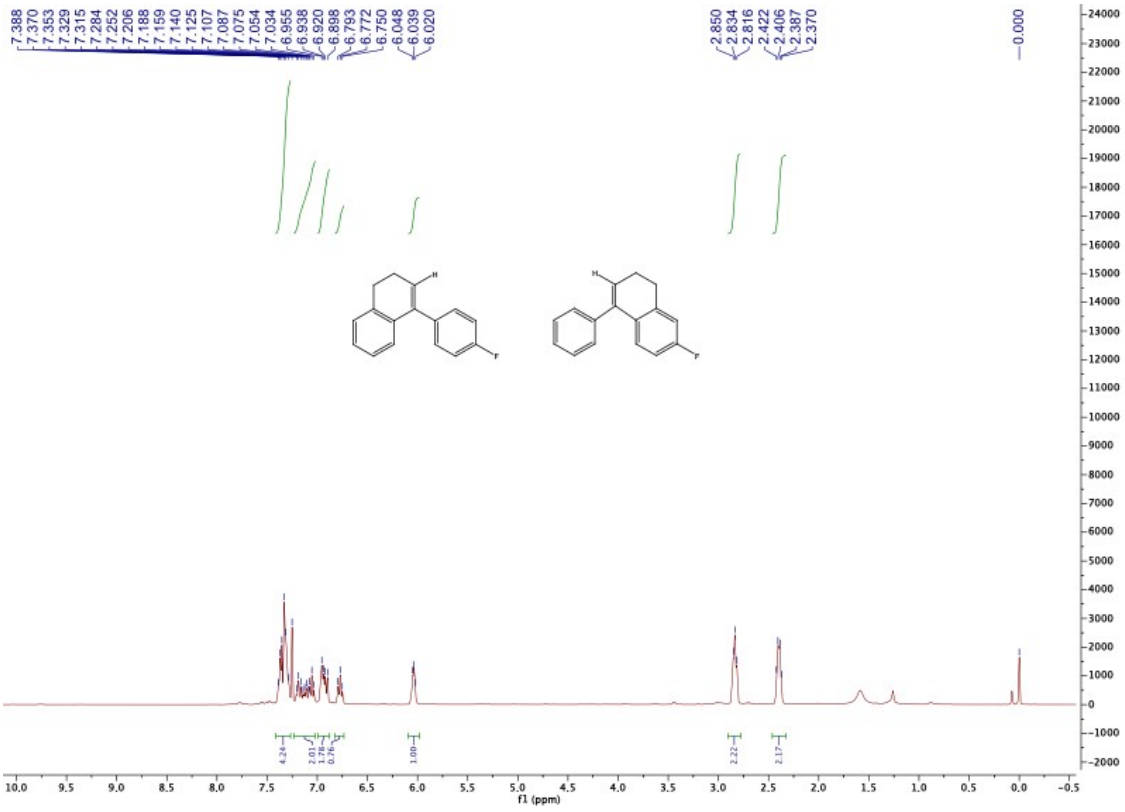
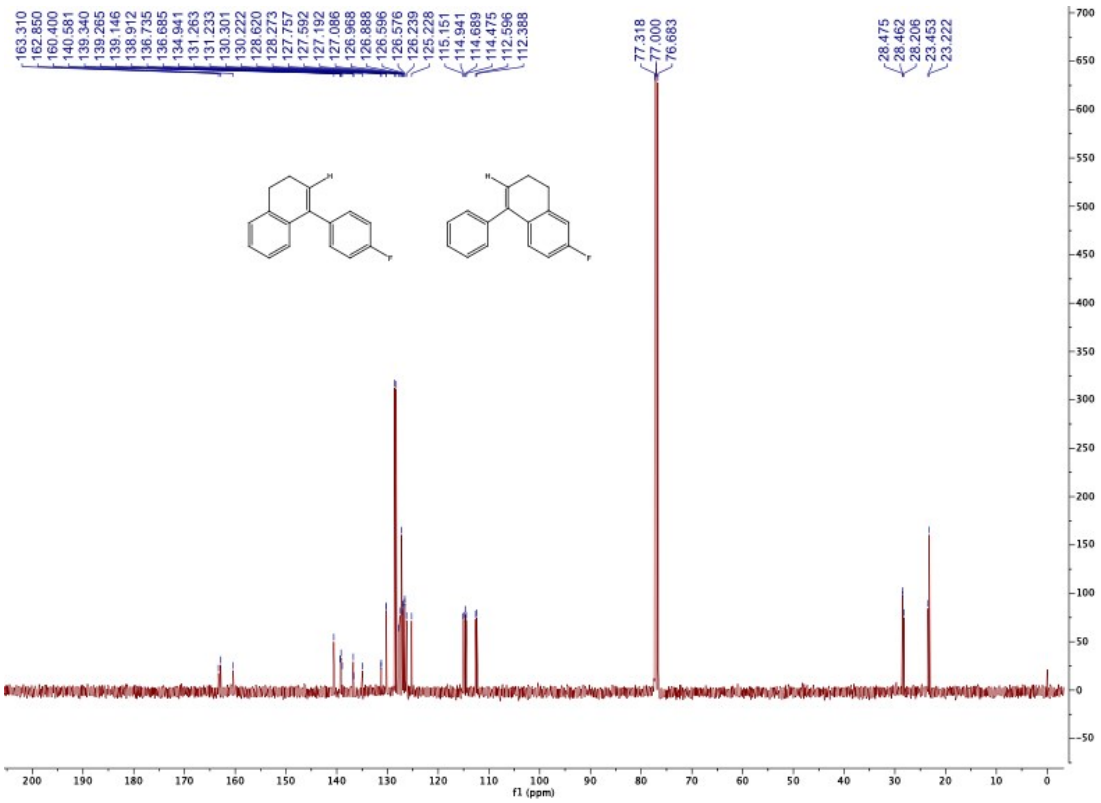
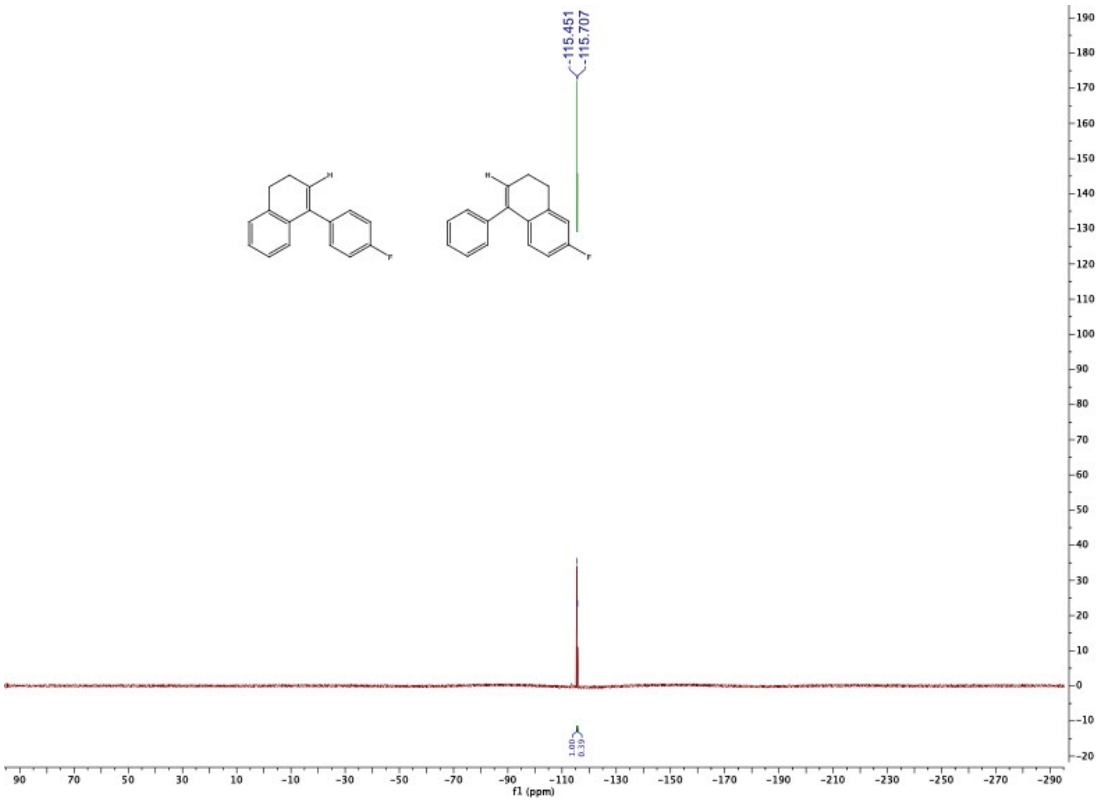
139.732
139.681
137.816
136.789
136.749
136.708
136.680
135.194
132.406
128.883
128.693
128.647
128.602
128.423
128.144
127.485
127.186
126.974
126.867
126.699
126.609
126.132
125.421
125.375
77.318
77.000
76.682
28.343
28.297
23.588
23.490
21.168
21.140
f1 (ppm)


7.388
7.370
7.353
7.329
7.315
7.284
7.252
7.206
7.188
7.159
7.140
7.125
7.107
7.087
7.075
7.054
7.034
6.955
6.938
6.920
6.898
6.793
6.772
6.750
6.048
6.039
6.020
2.850
2.834
2.816
2.422
2.406
2.387
2.370
0.000
4.24
2.01
1.76
0.76
1.00
2.22
2.17
f1 (ppm)


163.310
162.850
160.400
140.581
139.340
139.265
139.146
138.912
136.735
136.685
134.941
131.263
131.233
130.301
130.222
128.620
128.273
127.757
127.592
127.192
127.086
126.968
126.888
126.596
126.576
126.239
125.228
115.151
114.941
114.689
114.475
112.596
112.388
77.318
77.000
76.683
28.475
28.462
28.206
23.453
23.222
f1 (ppm)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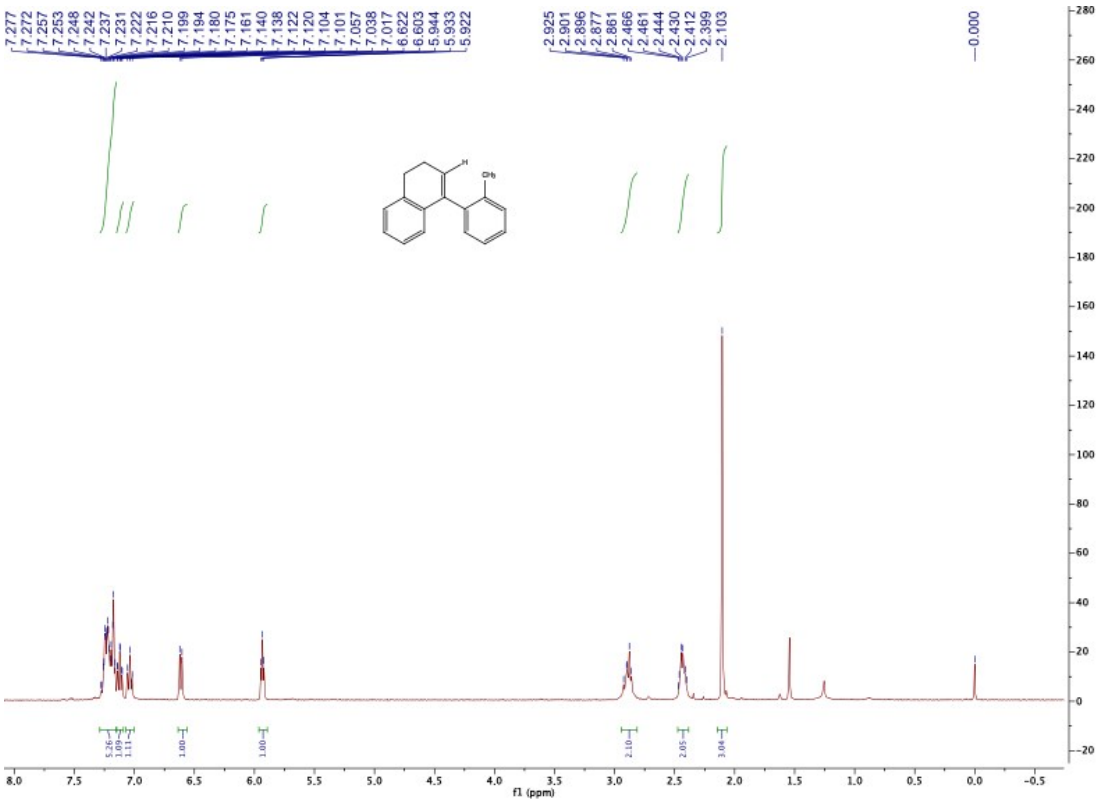
-115.707
1.00
0.39
f1 (ppm)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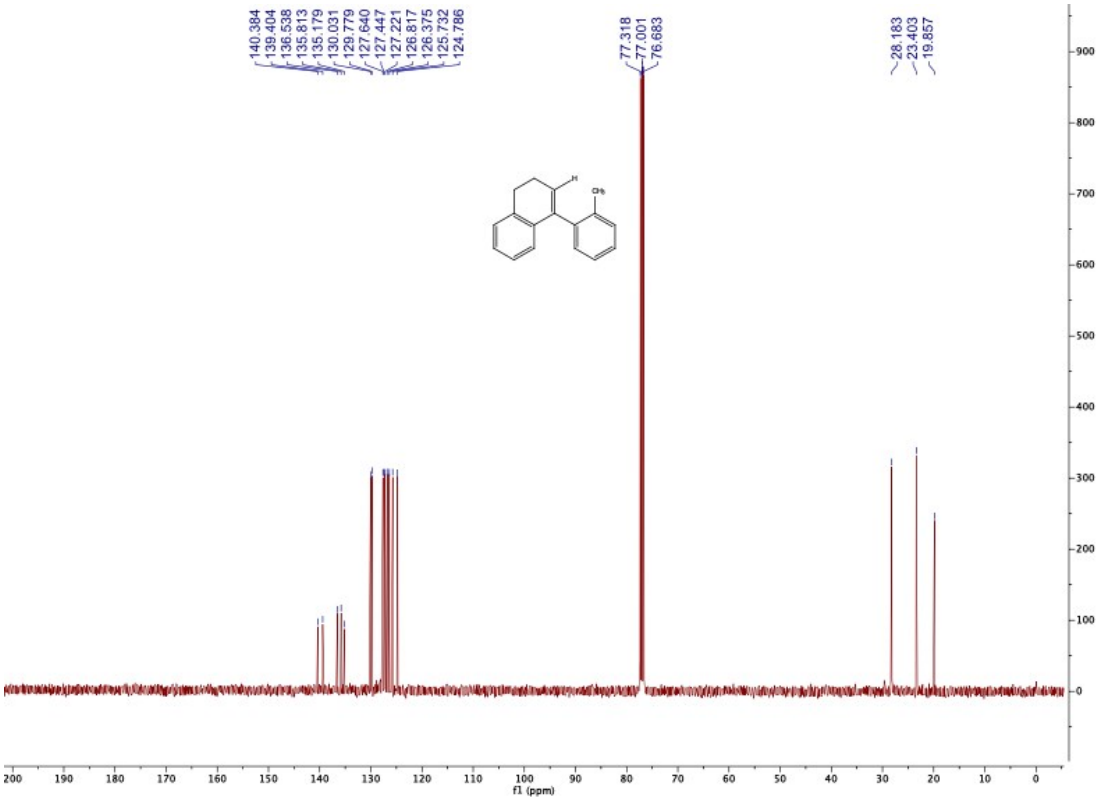
7.272
7.257
7.253
7.248
7.242
7.237
7.231
7.222
7.216
7.210
7.199
7.194
7.180
7.175
7.161
7.140
7.138
7.122
7.120
7.104
7.101
7.057
7.038
7.017
6.622
6.603
5.944
5.933
5.922
2.925
2.901
2.896
2.877
2.861
2.466
2.461
2.444
2.430
2.412
2.399
2.103
0.000
f1 (ppm)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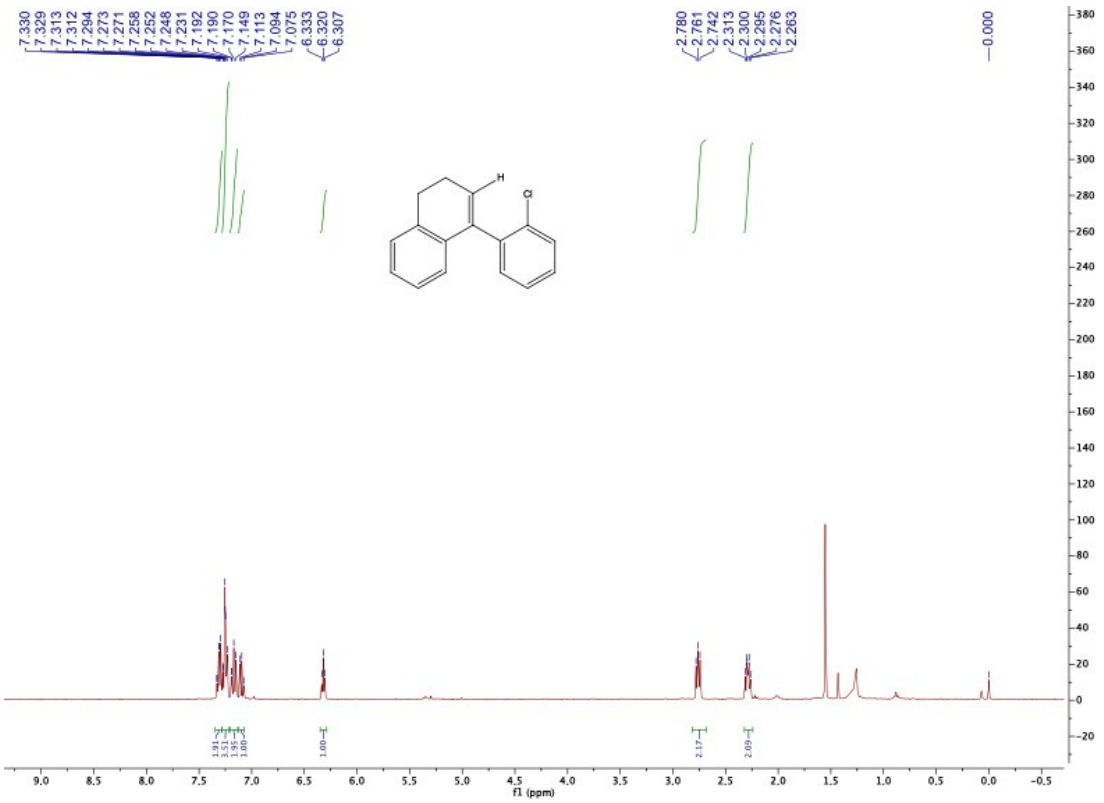
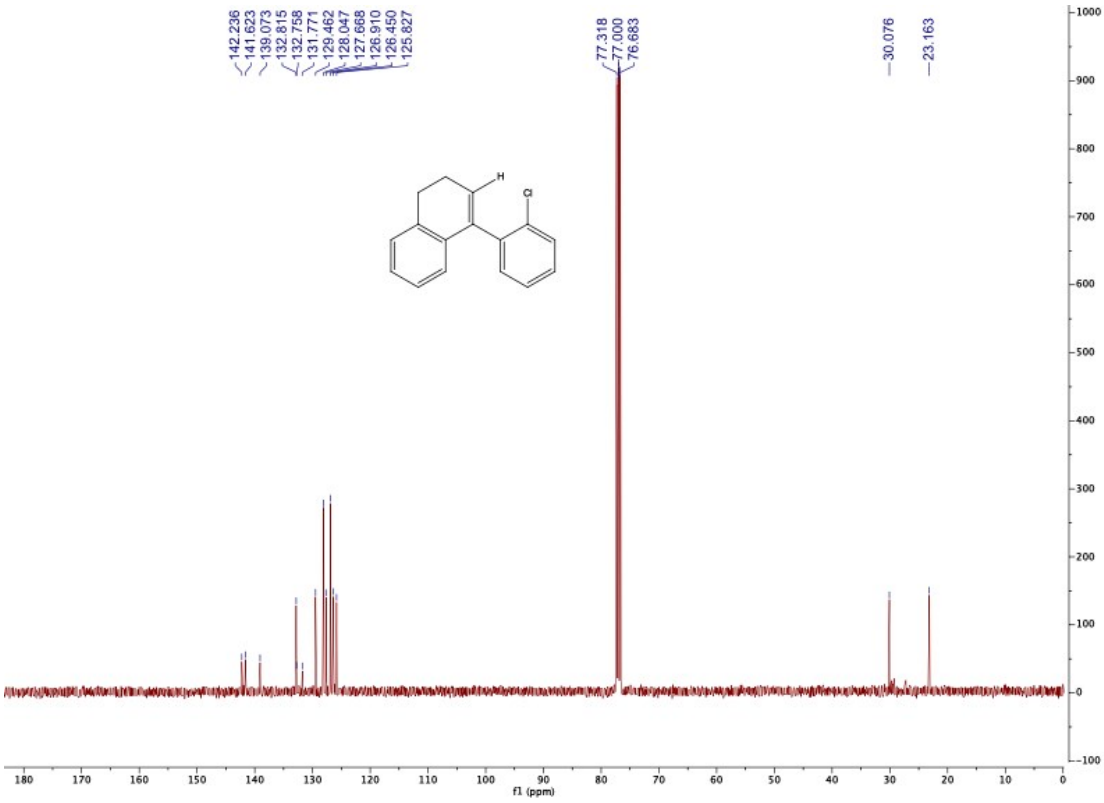
139.404
136.538
135.813
135.179
130.031
129.779
127.640
127.447
127.221
126.817
126.375
125.732
124.786
77.318
77.001
76.683
28.183
23.403
19.857
f1 (ppm)


7.330
7.329
7.313
7.312
7.294
7.273
7.271
7.258
7.252
7.248
7.231
7.192
7.190
7.170
7.149
7.113
7.094
7.075
6.333
6.320
6.307
2.780
2.761
2.742
2.313
2.300
2.295
2.276
2.263
0.000
H
Cl
f1 (ppm)


142.236
141.623
139.073
132.815
132.758
131.771
129.462
128.047
127.688
126.910
126.450
125.827
77.318
77.000
76.683
30.076
23.163
H
Cl
f1 (ppm)