S-Trifluoromethyl Thioesters as Bifunctional Reagents for Acyl-Trifluoromethylthiolation of Alkenes and 1,3-Enynes via Photoredox/Copper Dual Catalysis

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Supporting Information

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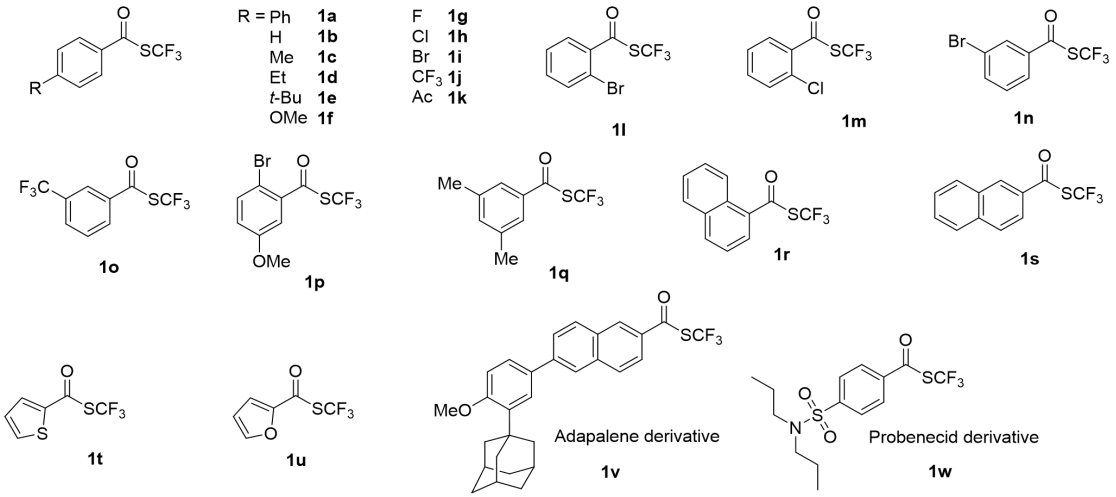
I. General Information

All reactions were performed at 6W Blue LEDs irradiation under argon atmosphere with magnetic stirring unless otherwise indicated. All reagents were purchased from Commercially available chemicals were obtained from bidepharm, Energy Chemical, Innochem, Admas, Alfa Aesar, J&K, Sigma-Aldrich, TCI and used without further purification. Anhydrous THF were purchased from Energy Chemical and stored over molecular sieves under nitrogen.

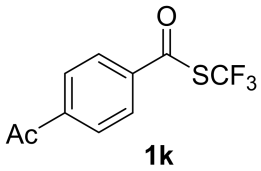
Melting points were determined using a digital melting point apparatus and uncorrected. ^{1}H, ^{19}F and ^{13}C NMR spectra were recorded on Bruker AV NEO 600 spectrometer (^{1}H: 600 MHz, ^{13}C: 151 MHz, ^{19}F: 565 MHz), usually in $CDCl_3$ with TMS as an internal standard. All chemical shifts were reported as δ values (ppm) relative to TMS (δ_H = 0 ppm) and $CDCl_3$ (δ_C = 77 ppm), respectively and observed coupling constants (*J*) are given in Hertz (Hz). The following abbreviations were used to explain fthe multiplicities: s = singlet, d = doublet, t = triplet, q = quartet. High resolution mass spectra (HRMS) of product were recorded on a Thermo fisher Q Exactive instrument. Melting points were obtained on a Yanaco MP-500 melting point apparatus with uncorrected.

II. Starting Materials



Scheme-S1 The scope of trifluoromethyl thioesters.

S-Trifluoromethyl thioesters were prepared according to reported methods.[1, 18] **1a**,[2] **1b**, [1] **1c**,[3] **1d**,[4] **1e**,[2] **1f-1j**,[2] **1l-1m**,[2] **1n**,[3] **1o**,[3] **1r**,[2] **1s**,[2] **1t**,[5] **1u**,[1] **1v**,[2] **1w**,[2] are known compounds, and the NMR are consistent with reported articles.

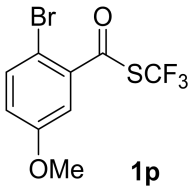


^{1}H NMR (600 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.6 Hz, 2H), 7.95 (d, *J* = 8.6 Hz, 2H), 2.66 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 196.8, 182.9, 141.6, 138.1, 128.9, 127.9, 127.7 (q, *J* = 310.0 Hz), 26.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.61.

HRMS (APCI) calcd for $C_{10}H_6F_3O_2S^-$ $[M-H]^-$ *m/z*: 247.0046, found: 247.0046.

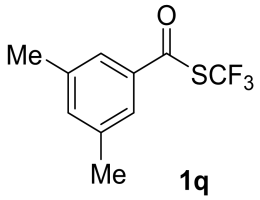


^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.8 Hz, 1H), 7.12 (d, *J* = 3.0 Hz, 1H), 6.96 (dd, *J* = 8.8, 3.0 Hz, 1H), 3.84 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 183.4, 158.8, 137.3, 137.2, 135.5, 127.4 (q, *J* = 311.2 Hz), 119.78, 114.7, 109.4, 55.8.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.53.

HRMS (APCI) calcd for $C_9H_7BrF_3O_2S^+$ $[M+H]^+$ *m/z*: 314.9297, found: 314.9286.



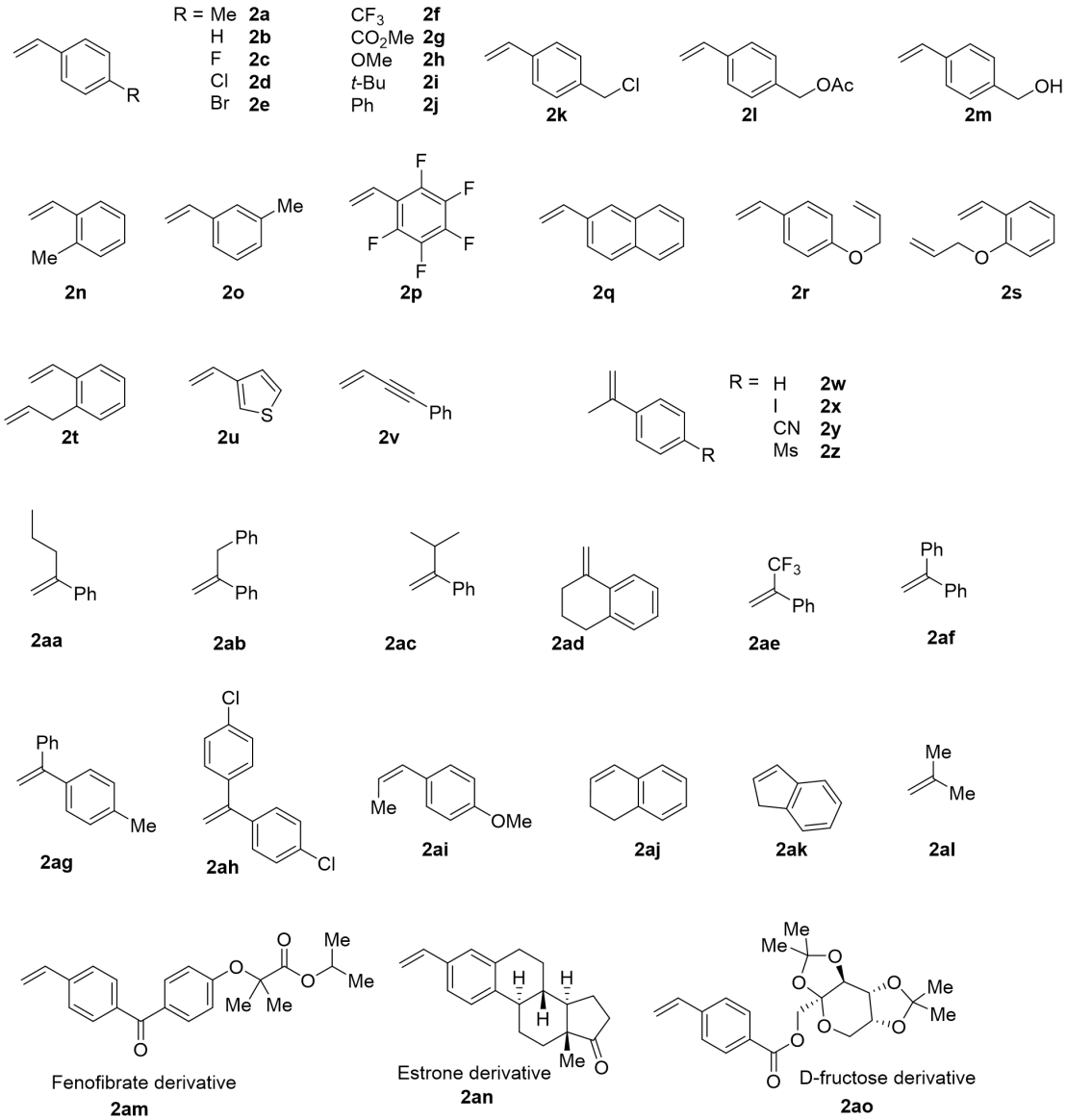
1q

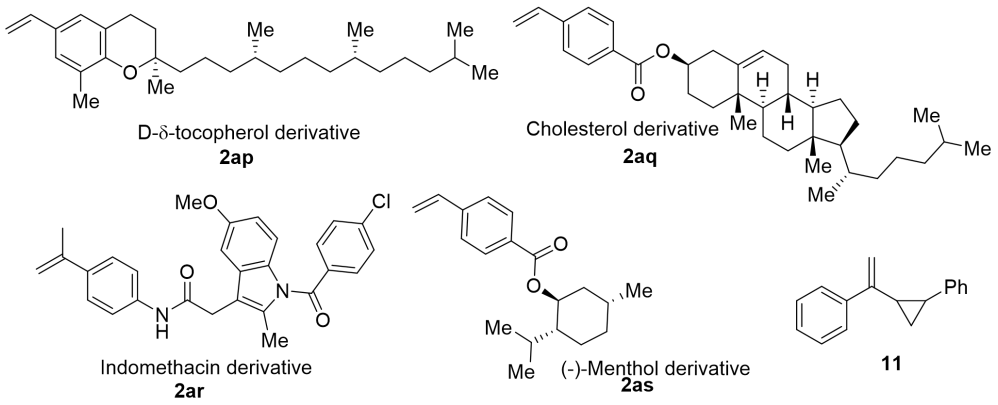
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.47 – 7.42 (m, 2H), 7.30 – 7.27 (m, 1H), 2.38 (d, *J* = 0.5 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 183.4, 139.1, 136.7, 135.2 (q, *J* = 2.5 Hz), 128.1 (q, *J* = 309.5 Hz), 125.3, 21.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.75.

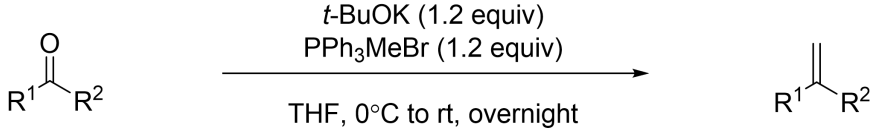
HRMS (APCI) calcd for $C_{10}H_{10}F_3OS^+$ $[M+H]^+$ *m/z*: 235.0399, found: 235.0397.





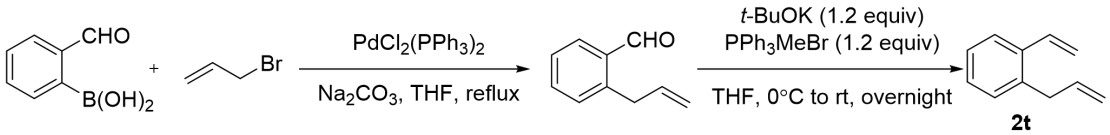
Scheme-S2 The scope of alkenes.

Alkenes **2a-2l**, **2n-2q**, **2w**, **2af**, **2ai-2al**, **2u, 2w**, **2af** and **2ai-2al** were commercial available. Alkenes **2m**,[6] **2r-2s**,[7] **2t**,[8] **2v**,[9] **2x-2ae**,[10] **2ag-2ah,**[10] **2am,**[11] **2an,**[11] **2ao**[12]**, 2ap,**[11] **2aq,**[12] **2as,**[13] and **19**[14] were synthesized according to the literature, and the NMR are consistent with reported articles.



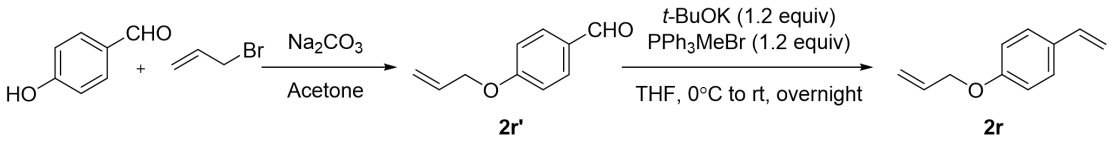
General procedure A: To a suspension of *t*-BuOK (1.35 g, 12mmol, 1.2 equiv.) in anhydrous THF (20 mL) was added $MePPh_3Br$ (4.28g, 12 mmol, 1.2 equiv.) under argon atmosphere. The reaction was stirred at room temperature for 1 h. Then ketones (12 mmol, 1,2 equiv.) was added at 0 °C and stirred at room temperature overnight. The reaction mixture was quenched by addition of saturated aqueous ammonium chloride and extracted by diethyl ether (30 mL × 3). The combined organic layer was washed with brine, dried with anhydrous sodium sulfate and concentrated in vacuum. The residues were purified by column chromatograph using silica gel with petroleum ether/ethyl acetate eluents to give the corresponding alkenes.

The procedure for the preparation of compound **2t.**



To a round-bottom flask charged with a stir bar, 2-formylphenylboronic acid (750 mg, 5 mmol, 1 equiv.) and allyl bromide (0.65 mL, 7.5 mmol, 1.5 equiv.), $PdCl_2(PPh_3)_2$ (97.7 mg, 2.5 mol%), and THF (25 mL) were added. The reaction mixture was heated to 50 °C, then aq Na_2CO_3 (1 M, 2 equiv.) solution was added drop wise over a period of 1 h and reflux for4 h. The reaction mixture was quenched with water and extracted with CH_2Cl_2 (30 mL × 3). The combined organic layer was washed with brine, dried with anhydrous sodium sulfate and concentrated in vacuum. The residue was purified by column chromatograph using silica gel with petroleum ether/ethyl acetate eluents to give the product **2t'**. Then **2t** was prepared from **2t'** according to the **General procedure A**.

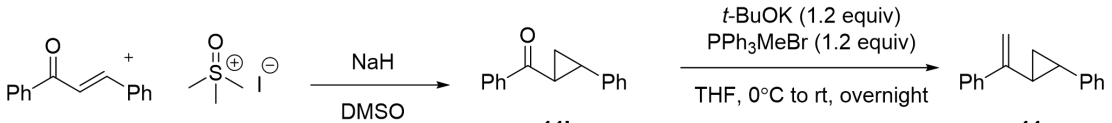
The procedure for the preparation of compounds **2r** and **2s.**



2r' **2r**

To a round-bottom flask charged with a stir bar, 4-hydroxybenzaldehyde (611 mg, 5 mmol, 1 equiv.), and Acetone (10 mL) were added. Then Na_2CO_3 (1.59 g, 15 mmol, 3 equiv.) and allyl bromide (0.65 mL, 7.5 mmol, 1.5 equiv.) added and the reaction mixture was stirred at room temperature for 2 h and further heated to reflux another 2 h. After cooling to room temperature, the solution was filtered, washed with acetone and concentrated in vacuum. The residue was purified by column chromatograph using silica gel with petroleum ether/ethyl acetate eluents to give the product **2r'**. Then **2t** was prepared from **2r'** according to the **General procedure A**.

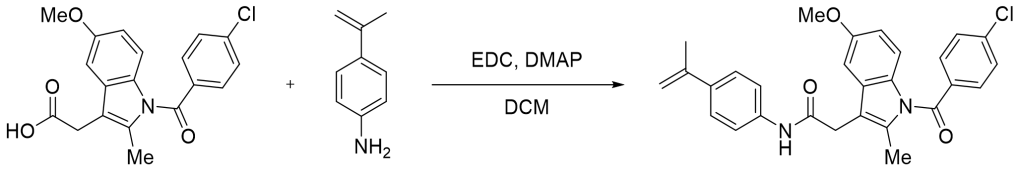
The procedure for the preparation of compound **11.**



11' **11**

To a two-necked round-bottom flask charged with a stir bar, NaH (0.48 g, 12 mmol, 60% dispersion in paraffin liquid) was added and washed with petroleum ether. Then, anhydrous DMSO (20 mL) and trimethylsulfonium iodide (2.42 g, 11 mmol) were added to the flask under argon atmosphere. A solution of (*E*)-chalcone (2.08 g, 10 mmol) in DMSO was added to the flask at 0 °C. The reaction mixture was stirred at room temperature for 2 h. Then the reaction mixture was heated to 50 °C and stirred another 2 h. After cooling to room temperature, the reaction was quenched with water and extracted with ethyl acetate. The combined organic layer was washed with brine, dried with anhydrous sodium sulfate and concentrated in vacuum. The residue was purified by column chromatograph using silica gel with petroleum ether/ethyl acetate eluents to give the product **11'**. Then **11** was prepared from **11'** according to the **General procedure A**.

The procedure for the preparation of compound **2ar.**[7]

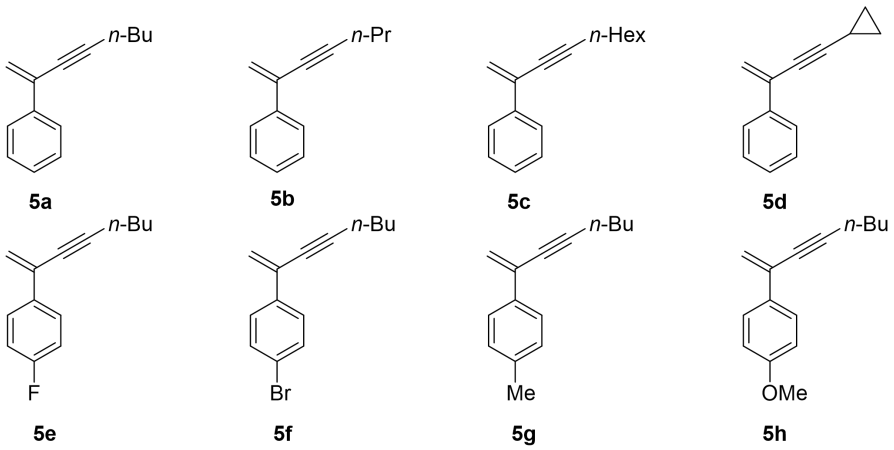


2ar

According to a literature, the solution of the Indomethacin (2 mmol, 716 mg), 4-vinylaniline (267 mg, 2.04 mmol), 1-(3- dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (575 mg, 3 mmol), and DMAP (5 mg, 0.04 mmol) in CH_2Cl_2 (20.0 mL) was stirred at room temperature for 2.5 h. After the reaction, the system extracted with CH_2Cl_2 (20mL × 3). The combined organic layers were dried over Na_2SO_4, and concentrated under reduced pressure. The resulting crude products were purified by flash column chromatography (petroleum ether/ethyl acetate = 5/1) to afford **2ar** (681 mg, 72%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.71 – 7.67 (m, 2H), 7.52 – 7.48 (m, 2H), 7.40 – 7.36 (m, 2H), 7.35 (d, *J* = 8.8 Hz, 2H), 7.28 (brs, 1H), 6.95 (d, *J* = 2.4 Hz, 1H), 6.88 (d, *J* = 9.0 Hz, 1H), 6.72 (dd, *J* = 9.0, 2.5 Hz, 1H), 5.31 (s, 1H), 5.07 – 4.99 (m, 1H), 3.81 (s, 5H), 2.45 (s, 3H), 2.11 (d, *J* = 0.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 168.3, 168.0, 156.4, 142.4, 139.7, 137.6, 136.7, 136.6, 133.5, 131.2, 131.0, 130.1, 129.3, 126.0, 119.8, 115.2, 112.5, 112.3, 112.0, 100.7, 55.8, 33.4, 21.7, 13.3.
HRMS (APCI) calcd for $C_{28}H_{26}ClN_2O_3^+$ $[M+H]^+$ *m/z*: 473.1626, found: 473.1620.



5a **5b** **5c** **5d**

5e **5f** **5g** **5h**

5a-5b,[15] **5c,**[16] **5e-5g**[15], **5h**[17] were synthesized according to the literature, and the NMR are consistent with reported articles.

General procedure B for preparation of 1,3-enynes **5a-5h**.
To a round-bottom flask charged with a stir bar, alkyne (5 mmol) and anhydrous THF (20 mL) were added. Then *n*-BuLi (2.0 M in hexane, 5 mmol, 2.5 mL) was added dropwise at -78 °C. Na_2CO_3 (1.59 g, 15 mmol, 3 equiv.) The reaction mixture was stirred at room temperature for 1 h. After cooling to -78 °C, ketone (5 mmol) in THF (10 mL) was added dropwise. The reaction mixture was allowed to warm to room temperature until the reaction is complete. The resulting mixture wasquenched with satureated aqueous NH_4Cl and extracted with ethyl acetate. The combined organic layer was washed with brine, dried with anhydrous sodium sulfate and concentrated in vacuum to give the crude propargyl alcohol **5'**.
The resulting propargyl alcohol **5'** was dissolved in DCM (30 mL) and TEA (25 mmol, 5 equiv) was added to this solution at 0 °C. Then methylsulfonyl chloride (12.5 mmol, 2.5 equiv) was added dropwise. After the reaction is complete, the reaction was quenched with saturated aqueous NH_4Cl, and extracted with ethyl acetate. The combined organic layer was washed with brine, dried with anhydrous sodium sulfate and concentrated in vacuum. The residue was purified by column chromatograph using silica gel with petroleum ether/dichloromethane eluents to give the product **5**.

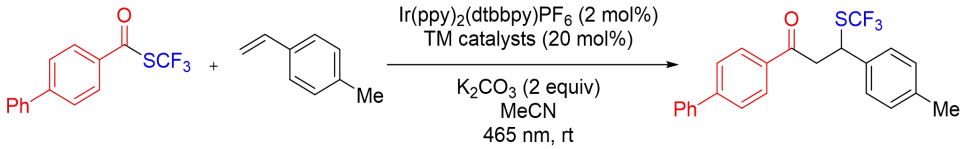
III. Optimization Studies

General procedure for the optimization of the reaction conditions

To an oven-dried 10 mL vial charged with a stir bar, *S*-trilfluoromethyl thioesters **1a**, Photocatalysis, and base were added. The $Cu(MeCN)_4PF_6$ was added into the vial in the glove box. Then, the septum capped vial was transferred out of the glovebox. Alkene **2a** and anhydrous THF were added under the argon atmosphere. After degassed by three cycles of freeze-pump thaw, the mixture was placed on a photoreactor equipped with 6W LED lamps and stirred for 12 hours at room temperature. The solvents were evaporated and the NMR yields were determined by using 1,3,5-trimethoxybenzene as internal

standard.

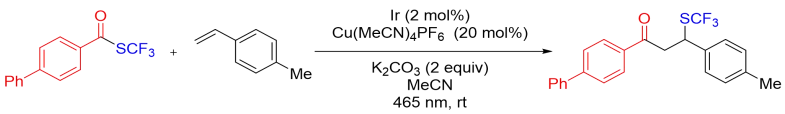
Table S1. Effect of transition-metal catalysts on the reaction



Entry	TM catalysts	Yield (%)
1	$Cu(MeCN)_4BF_4$	69
2	CuBr	64
3	CuCN	41
4	CuCl	59
5	CuI	8
6	CuOAc	3
7	Copper(I) thiophene-2-carboxylate	20
8	$Cu(MeCN)_4PF_6$	78
9	$Cu(OAc)_2$	0
10	$Cu(acac)_2$	0
11	$CuCl_2$	37
12	$CuCl_2•H_2O$	38
13	$CuBr_2$	58
14	$Cu(OTf)_2$	18
15	$Cu(OTf)_2•H_2O$	14
16	$Cu(OH)_2$	1
17	$NiCl_2$	0
18	$NiCl_2$•glyme	14
19	$Cu(BF_4)_2•6H_2O$	18

a

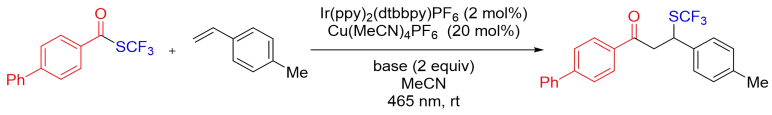
Table S2. Effect of photo catalysts on the reaction



Entry	Photocatalysts	Yield (%)
1	*fac*-[$Ir(ppy)_3$]	47
2	4CzIPN	19

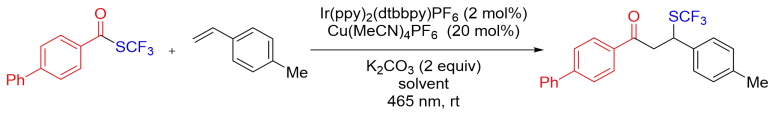
3	$[Ru(bpy)_3]Cl_2$	2
4	$Ir(ppy)_2(dtbppy)PF_6$	78
5	$Ir(tbbpy)_2(bpy)PF_6$	46
6	$Ir(dFppy)_3PF_6$	74
7	$Ir[dF(Me)_2ppy]_2(ddtbpy)PF_6$	18
8	$Ir[dF(CF_3)_2ppy]_2(bpy)PF_6$	0
9	$Ir(dFppy)_2(bpy)PF_6$	20
10	$Ir(Fppy)_2(dtbbpy)PF_6$	72
11	$Ir[dF(Me)ppy)_2(bpy)PF_6$	28
12	$Ir(Fppy)_2(bpy)PF_6$	63
13	$Ir(tbbpy)_2(bpy)PF_6$	53
14	Mes-Acr(ClO_4)	0
15	$Ir(dFppy)_2(dtbbpy)PF_6$	40
16	$Ir[dF(CF_3)_2ppy]_2(dtbpy)PF_6$	5

Table S3. Effect of bases on the reaction



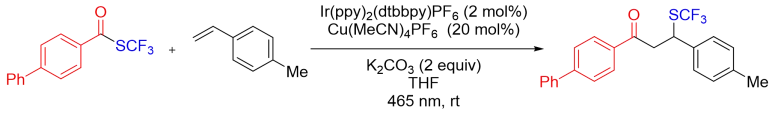
Entry	base	Yield (%)
1	K_2CO_3	78
2	Na_2CO_3	76
3	Cs_2CO_3	0
4	Li_2CO_3	52
5	K_3PO_4	62
6	$K_2PO_4•3H_2O$	64
7	$NaHCO_3$	72
8	LiOH	0
9	Et_3N	0
10	2,6-lutidine	0
11	KOAc	17

Table S4. Effect of solvents on the reaction



Entry	solvent	Yield (%)
1	MeCN	78
2	DCM	73
3	MeOH	0
4	THF	99
5	1,4-Dioxane	73
6	DMF	1
7	DMSO	6
8	THF (0.2 M)	83
9	THF (0.05 M)	99

Table S5. Effect of other conditions on the reaction

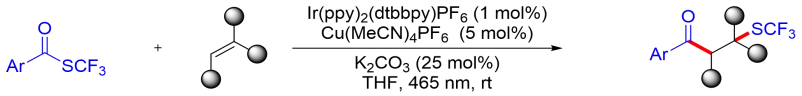


Entry	Deviation	Yield (%)
1	$Ir(ppy)_2(dtbppy)PF_6$ (1 mol%)	99
2	$Ir(ppy)_2(dtbppy)PF_6$ (0.5 mol%)	84
3[a]	$Cu(MeCN)_4PF_6$ (10 mol%)	99
4[a]	$Cu(MeCN)_4PF_6$ (5 mol%)	98
5[a]	$Cu(MeCN)_4PF_6$ (2.5 mol%)	40
6[a]	$Cu(MeCN)_4PF_6$ (0 mol%)	0
7[a, b]	K_2CO_3 (1 equiv.)	97
8[a, b]	K_2CO_3 (0.5 equiv.)	100
9[a, b]	K_2CO_3 (0.25 equiv.)	99 (94)
10[a, b]	K_2CO_3 (0 equiv.)	0
10[a, b, c]	$ArCOSCF_3$ (1.2 equiv.)	92

[a] $Ir(ppy)_2(dtbppy)PF_6$ (1 mol%); [b] $Cu(MeCN)_4PF_6$ (5 mol%); [c] K_2CO_3 (0.25 equiv.).

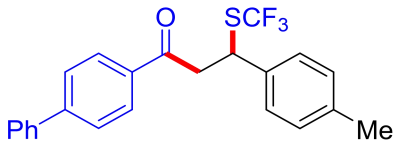
IV. Experimental Procedures and Characterization Data

General procedure for the preparation of compounds 3/4



An oven-dried 10 mL vial was charged with a stir bar, *S*-trilfluoromethyl thioesters **1** (0.3 mmol, 1.5 equiv.), $Ir(ppy)_2(dtbppy)PF_6$ (1.8 mg, 0.002 mmol, 1 mol%), and potassium carbonates (6.9 mg, 0.05 mmol, 25 mol%) was added. The $Cu(MeCN)_4PF_6$ (3.7 mg, 0.01 mmol, 5 mol%) was added into the vial in the glove box. Then, the septum capped vial was transferred out of the glovebox and alkene **2** (0.2 mmol, 1 equiv.) (if solid, added before sealed), THF (2 mL) were added under the argon atmosphere. After degassed by three cycles of freeze-pump thaw, the mixture was placed on a photoreactor equipped with 6W LED lamps and stirred for 12 hours. The solvent was evaporated and the residue was purified by column chromatography to afford the desired products **3/4**.

Characterization Data of compounds



3a

1-([1,1'-Biphenyl]-4-yl)-3-(p-tolyl)-3-((trifluoromethyl)thio)propan-1-one (**3a**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 75.2 mg product as a white solid in 94% yield.

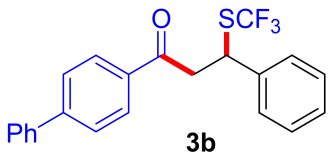
Mp = 110-112 °C

^{1}H NMR (600 MHz, cdcl$_3$) δ 7.98 (d, *J* = 8.2 Hz, 2H), 7.67 (d, *J* = 8.2 Hz, 2H), 7.61 (d, *J* = 7.8 Hz, 2H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.3 Hz, 1H), 7.32 (d, *J* = 7.9 Hz, 2H), 7.14 (d, *J* = 7.9 Hz, 2H), 5.11 (dd, *J* = 8.5, 5.4 Hz, 1H), 3.82 (dd, *J* = 17.4, 8.6 Hz, 1H), 3.69 (dd, *J* = 17.4, 5.3 Hz, 1H), 2.31 (s, 3H).

^{13}C NMR (151 MHz, cdcl$_3$) δ 195.1, 146.2, 139.6, 137.9, 136.6, 134.9, 130.3 (q, *J* = 307.8 Hz), 129.5, 129.0, 128.6, 128.4, 127.4, 127.3, 127.2, 45.2, 44.1, 21.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.07.

HRMS (APCI) calcd for $C_{23}H_{20}F_3OS^+$ $[M+H]^+$ *m/z*: 401.1181, found: 401.1182.



3b

1-([1,1'-Biphenyl]-4-yl)-3-phenyl-3-((trifluoromethyl)thio)propan-1-one (**3b**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 93.3 mg product as a white solid in 95% yield.

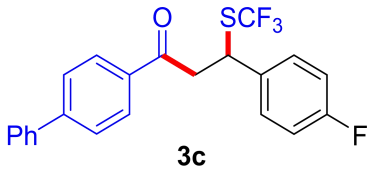
Mp = 92-94 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 – 7.94 (m, 2H), 7.67 – 7.63 (m, 2H), 7.61 – 7.58 (m, 2H), 7.47 – 7.41 (m, 4H), 7.41 – 7.37 (m, 1H), 7.34 – 7.30 (m, 2H), 7.27 – 7.23 (m, 1H), 5.13 (dd, *J* = 8.4, 5.5 Hz, 1H), 3.80 (dd, *J* = 17.4, 8.5 Hz, 1H), 3.69 (dd, *J* = 17.4, 5.5 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.0, 146.3, 139.8, 139.6, 134.9, 130.3 (q, *J* = 307.5 Hz), 129.0, 128.8, 128.6, 128.4, 128.1, 127.6, 127.3, 127.2, 45.1, 44.3.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.06.

HRMS (ESI) calcd for $C_{22}H_{16}F_3OS^+$ $[M-H]^-$ *m/z*: 385.0879, found: 385.0878.



3c

1-([1,1'-Biphenyl]-4-yl)-3-(4-fluorophenyl)-3-((trifluoromethyl)thio)propan-1-one (**3c**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 70.0 mg product as a white solid in 87% yield.

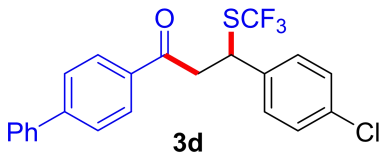
Mp = 88-90 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 – 7.94 (m, 2H), 7.69 – 7.65 (m, 2H), 7.62 – 7.58 (m, 2H), 7.49 – 7.44 (m, 2H), 7.43 – 7.38 (m, 3H), 7.04 – 6.98 (m, 2H), 5.12 (dd, *J* = 8.7, 5.2 Hz, 1H), 3.77 (dd, *J* = 17.4, 8.7 Hz, 1H), 3.68 (dd, *J* = 17.4, 5.2 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.8, 162.2 (d, *J* = 247.3 Hz), 146.4, 139.6, 135.6 (d, *J* = 3.2 Hz), 134.8, 130.2 (q, *J* = 307.7 Hz), 129.3 (d, *J* = 8.1 Hz), 129.0, 128.6, 128.4, 127.4, 127.2, 115.8 (d, *J* = 21.6 Hz), 45.1, 43.6.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.09, -113.52 – -113.83 (m).

HRMS (APCI) calcd for $C_{22}H_{17}F_4OS^+$ $[M+H]^+$ *m/z*: 405.0931, found: 405.0924.



3d

1-([1,1'-Biphenyl]-4-yl)-3-(4-chlorophenyl)-3-((trifluoromethyl)thio)propan-1-one (**3d**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 75.2 mg product as a white solid in 89% yield.

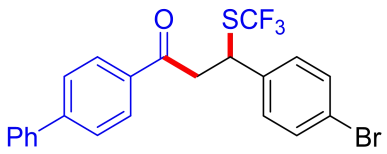
Mp = 86-88 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 – 7.94 (m, 2H), 7.70 – 7.65 (m, 2H), 7.62 – 7.58 (m, 2H), 7.49 – 7.44 (m, 2H), 7.42 – 7.38 (m, 1H), 7.38 – 7.35 (m, 2H), 7.32 – 7.28 (m, 2H), 5.09 (dd, *J* = 8.6, 5.2 Hz, 1H), 3.76 (dd, *J* = 17.5, 8.7 Hz, 1H), 3.67 (dd, *J* = 17.5, 5.2 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.7, 146.5, 139.5, 138.4, 134.7, 133.9, 130.2 (q, *J* = 307.6 Hz), 129.0, 128.6, 128.4, 127.4, 127.2, 44.9, 43.6.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.07.

HRMS (APCI) calcd for $C_{22}H_{17}ClF_3OS^+$ $[M+H]^+$ *m/z*: 421.0635, found: 421.0628.



3e

1-([1,1'-Biphenyl]-4-yl)-3-(4-bromophenyl)-3-((trifluoromethyl)thio)propan-1-one (**3e**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 69.0 mg product as a white solid in 74% yield.

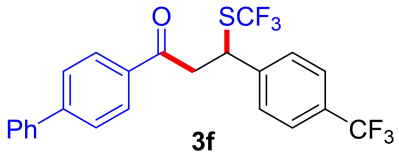
Mp = 82-84 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 – 7.92 (m, 2H), 7.69 – 7.64 (m, 2H), 7.62 – 7.58 (m, 2H), 7.48 – 7.43 (m, 4H), 7.42 – 7.37 (m, 1H), 7.33 – 7.29 (m, 2H), 5.08 (dd, *J* = 8.6, 5.2 Hz, 1H), 3.75 (dd, *J* = 17.5, 8.6 Hz, 1H), 3.67 (dd, *J* = 17.5, 5.2 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.7, 146.4, 139.5, 139.0, 134.7, 131.9, 130.1 (q, *J* = 308.2 Hz), 129.3, 129.0, 128.6, 128.4, 127.4, 127.2, 122.0, 44.8, 43.6.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.06.

HRMS (APCI) calcd for $C_{22}H_{17}F_3OBrS^+$ $[M+H]^+$ *m/z*: 465.0130, found: 465.0122.



3f

1-([1,1'-Biphenyl]-4-yl)-3-(4-(trifluoromethyl)phenyl)-3-((trifluoromethyl)thio)propan-1-one (**3f**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 75.6 mg product as a white solid in 83% yield.

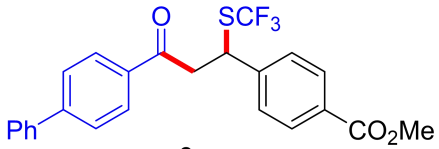
Mp = 104-106 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 – 7.85 (m, 2H), 7.62 – 7.56 (m, 2H), 7.53 – 7.46 (m, 6H), 7.39 – 7.35 (m, 2H), 7.34 – 7.29 (m, 1H), 5.07 (dd, *J* = 8.5, 5.2 Hz, 1H), 3.70 (dd, *J* = 17.6, 8.5 Hz, 1H), 3.63 (dd, *J* = 17.6, 5.2 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.5, 146.6, 144.1, 139.5, 134.6, 130.2 (q, *J* = 32.6 Hz), 130.1 (q, *J* = 307.6 Hz), 129.0, 128.6, 128.5, 128.1, 127.4, 127.2, 125.8 (q, *J* = 3.9 Hz) 123.9 (q, *J* = 272.1 Hz), 44.8, 43.6.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.14, -62.65.

HRMS (APCI) calcd for $C_{23}H_{17}F_6OS^+$ $[M+H]^+$ *m/z*: 455.0899, found: 455.0894.



3g

Methyl 4-(3-([1,1'-biphenyl]-4-yl)-3-oxo-1-((trifluoromethyl)thio)propyl)benzoate (**3g**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 69.3 mg product as a white solid in 83% yield.

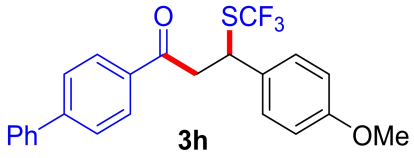
Mp = 92-94 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 – 7.90 (m, 2H), 7.89 – 7.84 (m, 2H), 7.60 – 7.56 (m, 2H), 7.53 – 7.49 (m, 2H), 7.45 – 7.42 (m, 2H), 7.40 – 7.35 (m, 2H), 7.33 – 7.28 (m, 1H), 5.07 (dd, *J* = 8.4, 5.4 Hz, 1H), 3.80 (s, 3H), 3.71 (dd, *J* = 17.5, 8.5 Hz, 1H), 3.62 (dd, *J* = 17.5, 5.4 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.6, 166.5, 146.4, 145.0, 139.5, 134.6, 130.1 (q, *J* = 308.2 Hz), 130.1, 129.8, 1289.0, 128.6, 128.4, 127.7, 127.4, 127.2, 52.1, 44.7, 43.8.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.06.

HRMS (APCI) calcd for $C_{24}H_{20}F_3O_3S^+$ $[M+H]^+$ *m/z*: 445.1080, found: 445.1074.



3h

1-([1,1'-Biphenyl]-4-yl)-3-(4-methoxyphenyl)-3-((trifluoromethyl)thio)propan-1-one (**3h**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 68.3 mg product as a white solid in 82% yield.

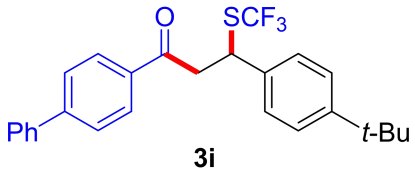
Mp = 91-93 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 – 7.94 (m, 2H), 7.69 – 7.65 (m, 2H), 7.62 – 7.57 (m, 2H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.41 – 7.37 (m, 1H), 7.35 – 7.32 (m, 2H), 6.87 – 6.82 (m, 2H), 5.11 (dd, *J* = 8.7, 5.3 Hz, 1H), 3.79 (dd, *J* = 17.3, 8.8 Hz, 1H), 3.76 (s, 3H), 3.67 (dd, *J* = 17.3, 5.3 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.2, 159.2, 146.2, 139.6, 134.9, 131.5, 130.3 (q, *J* = 308.1 Hz), 129.0, 128.7, 128.6, 128.4, 127.3, 127.2, 114.2, 55.2, 45.2, 44.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.02.

HRMS (APCI) calcd for $C_{22}H_{19}O_2^+$ $[M-SCF_3]^+$ *m/z*: 315.1380, found: 315.1375.



3i

1-([1,1'-Biphenyl]-4-yl)-3-(4-(tert-butyl)phenyl)-3-((trifluoromethyl)thio)propan-1-one (**3i**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 79.1mg product as a white solid in 89% yield.

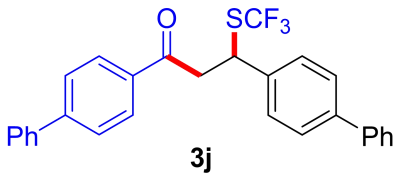
Mp = 89-91 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 – 7.94 (m, 2H), 7.67 – 7.64 (m, 2H), 7.61 – 7.58 (m, 2H), 7.48 – 7.43 (m, 2H), 7.41 – 7.37 (m, 1H), 7.36 – 7.31 (m, 4H), 5.13 (dd, *J* = 8.4, 5.3 Hz, 1H), 3.83 (dd, *J* = 17.5, 8.5 Hz, 1H), 3.70 (dd, *J* = 17.5, 5.3 Hz, 1H), 1.28 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.2, 151.0, 146.2, 139.6, 136.5, 134.9, 130.4 (q, *J* = 307.5 Hz), 129.0, 128.6, 128.4, 127.3, 127.2, 127.2, 125.8, 45.3, 44.0 (q, *J* = 1.3 Hz), 34.5, 31.2.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.09.

HRMS (APCI) calcd for $C_{26}H_{24}F_3OS^-$ $[M-H]^-$ *m/z*: 441.1505, found: 441.1508.



3j

1,3-Di([1,1'-biphenyl]-4-yl)-3-((trifluoromethyl)thio)propan-1-one (**3j**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 77.6 mg product as a white solid in 84% yield.

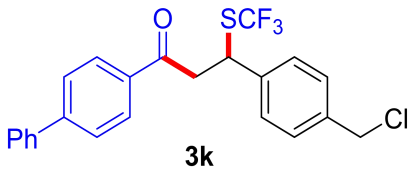
Mp = 163-165 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 – 7.95 (m, 2H), 7.70 – 7.66 (m, 2H), 7.63 – 7.59 (m, 2H), 7.58 – 7.53 (m, 4H), 7.49 (d, *J* = 8.3 Hz, 2H), 7.48 – 7.44 (m, 2H), 7.44 – 7.38 (m, 3H), 7.36 – 7.31 (m, 1H), 5.18 (dd, *J* = 8.6, 5.3 Hz, 1H), 3.86 (dd, *J* = 17.5, 8.6 Hz, 1H), 3.74 (dd, *J* = 17.5, 5.3 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.1, 146.4, 141.0, 140.4, 139.6, 138.7, 134.9, 130.3 (q, *J* = 307.5 Hz), 129.0, 128.8, 128.7, 128.4, 128.0, 127.54, 127.45, 127.4, 127.3, 127.0, 45.2, 44.0 (q, *J* = 1.5 Hz).

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.03.

HRMS (APCI) calcd for $C_{28}H_{22}F_3OS^+$ $[M+H]^+$ *m/z*: 463.1338, found: 463.1330.



3k

1-([1,1'-Biphenyl]-4-yl)-3-(4-(chloromethyl)phenyl)-3-((trifluoromethyl)thio)propan-1-one (**3k**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 64.3 mg product as a white solid in 74% yield.

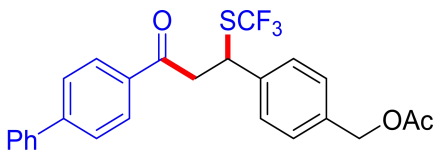
Mp = 102-103 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 – 7.95 (m, 2H), 7.69 – 7.66 (m, 2H), 7.63 – 7.59 (m, 2H), 7.50 – 7.45 (m, 2H), 7.44 – 7.38 (m, 3H), 7.36 (d, *J* = 8.2 Hz, 2H), 5.13 (dd, *J* = 8.5, 5.4 Hz, 1H), 4.54 (s, 2H), 3.80 (dd, *J* = 17.5, 8.5 Hz, 1H), 3.70 (dd, *J* = 17.5, 5.3 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.8, 146.4, 140.1, 139.6, 137.3, 134.8, 130.2 (q, *J* = 307.5 Hz), 129.0, 129.0, 128.6, 128.4, 128.0, 127.4, 127.3, 45.6, 45.0, 43.9, 43.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.10.

HRMS (APCI) calcd for $C_{23}H_{19}ClF_3OS^+$ $[M+H]^+$ *m/z*: 435.0792, found: 435.0786.



3l

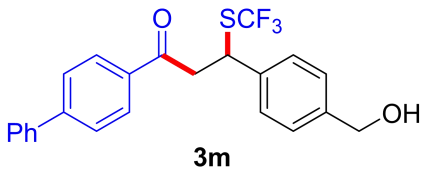
4-(3-([1,1'-Biphenyl]-4-yl)-3-oxo-1-((trifluoromethyl)thio)propyl)benzyl acetate (**3l**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 10/1) to give 79.9 mg product as a pale yellow oil in 87% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 – 7.95 (m, 2H), 7.70 – 7.66 (m, 2H), 7.63 – 7.58 (m, 2H), 7.48 – 7.45 (m, 2H), 7.43 (d, *J* = 8.2 Hz, 2H), 7.42 – 7.38 (m, 1H), 7.32 (d, *J* = 8.2 Hz, 2H), 5.13 (dd, *J* = 8.5, 5.3 Hz, 1H), 5.06 (s, 2H), 3.81 (dd, *J* = 17.5, 8.6 Hz, 1H), 3.70 (dd, *J* = 17.5, 5.3 Hz, 1H), 2.08 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.9, 170.8, 146.4, 139.8, 139.6, 135.8, 134.8, 130.2 (q, *J* = 307.7 Hz), 129.0, 128.7, 128.6, 128.4, 127.8, 127.3, 127.2, 65.7, 45.1, 43.9, 20.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.10.

HRMS (APCI) calcd for $C_{24}H_{21}O_3^+$ [M-SCF_3]$^+$ *m/z*: 357.1485, found: 357.1481.



3m

1-([1,1'-Biphenyl]-4-yl)-3-(4-(hydroxymethyl)phenyl)-3-((trifluoromethyl)thio)propan-1-one (**3m**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 4/1) to give 63.8 mg product as a white solid in 77% yield.

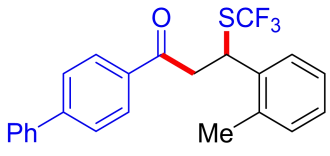
Mp = 111-113 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 – 7.91 (m, 2H), 7.68 – 7.63 (m, 2H), 7.61 – 7.57 (m, 2H), 7.48 – 7.44 (m, 2H), 7.43 – 7.37 (m, 3H), 7.31 (d, *J* = 8.2 Hz, 2H), 5.12 (dd, *J* = 8.6, 5.3 Hz, 1H), 4.62 (s, 2H), 3.80 (dd, *J* = 17.4, 8.7 Hz, 1H), 3.69 (dd, *J* = 17.4, 5.3 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.1, 146.3, 140.8, 139.5, 139.0, 134.8, 130.3 (q, *J* = 307.7 Hz), 129.0, 128.6, 128.4, 127.7, 127.33, 127.32, 127.2, 64.7, 45.0, 44.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.03.

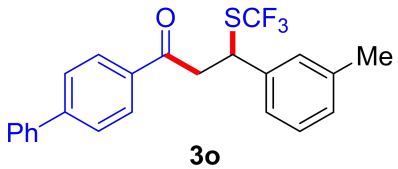
HRMS (APCI) calcd for $C_{23}H_{20}F_3O_2S^+$ [M+H]$^+$ *m/z*: 417.1131, found: 417.1128.



3n

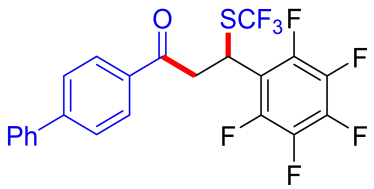
(R)-1-([1,1'-biphenyl]-4-yl)-3-(o-tolyl)-3-((trifluoromethyl)thio)propan-1-one (**3n**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 77.7 mg product as a colorless oil in 97% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 8.3 Hz, 2H), 7.60 (d, *J* = 7.3 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.34 – 7.31 (m, 1H), 7.20 – 7.13 (m, 3H), 5.37 (dd, *J* = 9.1, 5.1 Hz, 1H), 3.98 (dd, *J* = 17.6, 9.1 Hz, 1H), 3.75 (dd, *J* = 17.6, 5.1 Hz, 1H), 2.55 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 195.4, 146.3, 139.6, 137.1, 136.2, 134.9, 131.0, 130.4 (q, *J* = 307.9 Hz), 129.0, 128.6, 128.4, 128.1, 127.4, 127.3, 126.6, 126.2, 45.3, 40.2, 19.3.
^{19}F NMR (565 MHz, $CDCl_3$) δ -40.14.
HRMS (APCI) calcd for $C_{23}H_{20}F_3OS^+$ $[M+H]^+$ *m/z*: 401.1181, found: 401.1180.



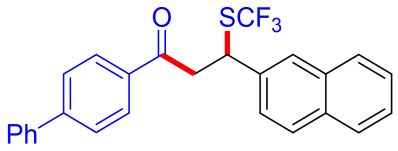
3o

(R)-1-([1,1'-biphenyl]-4-yl)-3-(m-tolyl)-3-((trifluoromethyl)thio)propan-1-one (**3o**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 63.0 mg product as a pale yellow oil in 79% yield.
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91 – 7.86 (m, 2H), 7.57 (d, *J* = 8.3 Hz, 2H), 7.53 – 7.48 (m, 2H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.33 – 7.28 (m, 1H), 7.16 – 7.10 (m, 3H), 7.00 – 6.95 (m, 1H), 5.04 – 4.97 (m, 1H), 3.72 (dd, *J* = 17.4, 8.4 Hz, 1H), 3.64 – 3.57 (m, 1H), 2.24 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 195.1, 146.2, 139.6, 138.6, 134.9, 130.3 (q, *J* = 307.9 Hz), 129.0, 128.9, 128.7, 128.6, 128.4, 128.3, 127.3, 127.2, 124.5, 45.2, 44.3, 21.4.
^{19}F NMR (565 MHz, $CDCl_3$) δ -40.08.
HRMS (APCI) calcd for C23H20F3OS$^+$ $[M+H]^+$ *m/z*: 401.1181, found: 401.1177.



3p

1-([1,1'-Biphenyl]-4-yl)-3-(perfluorophenyl)-3-((trifluoromethyl)thio)propan-1-one (**3p**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 74.9 mg product as a pale yellow oil in 79% yield.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 – 7.95 (m, 2H), 7.71 – 7.67 (m, 2H), 7.63 – 7.59 (m, 2H), 7.50 – 7.45 (m, 2H), 7.44 – 7.39 (m, 1H), 5.41 (dd, *J* = 9.8, 5.1 Hz, 1H), 3.96 (dd, *J* = 18.0, 9.9 Hz, 1H), 3.75 (dd, *J* = 18.0, 5.2 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 194.1, 146.8, 139.5, 134.1, 130.1 (q, *J* = 307.4 Hz), 129.0, 128.6, 128.5, 127.5, 127.3, 42.4, 32.9.
^{19}F NMR (565 MHz, $CDCl_3$) δ -41.07 (s, 3F), -140.77 (d, *J* = 17.2 Hz, 2F), -153.75 (t, *J* = 20.9 Hz, 1F), -161.14 (td, *J* = 21.0, 6.6 Hz, 2F).
HRMS (APCI) calcd for $C_{22}H_{13}F_8OS^+$ $[M+H]^+$ *m/z*: 477.0554, found: 477.0547.



3q

1-([1,1'-Biphenyl]-4-yl)-3-(naphthalen-2-yl)-3-((trifluoromethyl)thio)propan-1-one (**3q**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 36.4 mg product as a white solid in 42% yield.

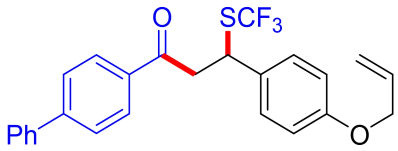
Mp = 132-134 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.86 (s, 1H), 7.83 (d, *J* = 8.6 Hz, 1H), 7.82 – 7.77 (m, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.62 – 7.58 (m, 2H), 7.56 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.49 – 7.43 (m, 4H), 7.39 (t, *J* = 7.3 Hz, 1H), 5.31 (dd, *J* = 8.4, 5.5 Hz, 1H), 3.91 (dd, *J* = 17.5, 8.5 Hz, 1H), 3.78 (dd, *J* = 17.5, 5.4 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.0, 146.3, 139.6, 137.0, 134.9, 133.2, 132.9, 130.3 (q, *J* = 307.8 Hz), 129.0, 128.9, 128.7, 128.4, 128.0, 127.7, 127.4, 127.3, 126.6, 126. 5, 126.4, 125.2, 45.1, 44.5.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.01.

HRMS (APCI) calcd for $C_{26}H_{18}F_3OS^-$ $[M-H]^-$ *m/z*: 435.1036, found: 435.1030.



3r

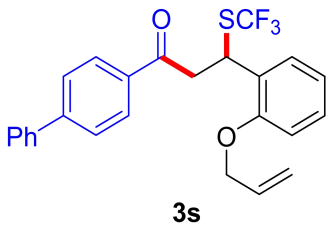
1-([1,1'-Biphenyl]-4-yl)-3-(4-(allyloxy)phenyl)-3-((trifluoromethyl)thio)propan-1-one (**3r**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 49.9 mg product as a colorless oil in 56% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 – 7.93 (m, 2H), 7.68 – 7.63 (m, 2H), 7.60 (dd, *J* = 5.2, 3.3 Hz, 2H), 7.46 (dd, *J* = 10.4, 4.8 Hz, 2H), 7.42 – 7.37 (m, 1H), 7.35 – 7.30 (m, 2H), 6.88 – 6.82 (m, 2H), 6.01 (ddt, *J* = 17.2, 10.6, 5.3 Hz, 1H), 5.38 (dq, *J* = 17.2, 1.5 Hz, 1H), 5.26 (dq, *J* = 10.5, 1.3 Hz, 1H), 5.11 (dd, *J* = 8.7, 5.3 Hz, 1H), 4.49 (dt, *J* = 5.3, 1.4 Hz, 2H), 3.79 (dd, *J* = 17.3, 8.7 Hz, 1H), 3.67 (dd, *J* = 17.3, 5.3 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.2, 146.2, 139.6, 134.9, 133.0, 131.7, 130.3 (d, *J* = 307.6 Hz), 129.0, 128.7, 128.6, 128.4, 127.3, 127.2, 117.7, 114.9, 68.8, 45.2, 44.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.01.

HRMS (APCI) calcd for $C_{25}H_{22}F_3O_2S^+$ $[M+H]^+$ *m/z*: 443.1287, found: 443.1278.



3s

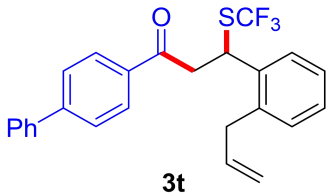
1-([1,1'-Biphenyl]-4-yl)-3-(2-(allyloxy)phenyl)-3-((trifluoromethyl)thio)propan-1-one (**3s**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 57.8 mg product as a colorless oil in 65% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 – 7.94 (m, 2H), 7.67 – 7.62 (m, 2H), 7.61 – 7.57 (m, 2H), 7.48 – 7.42 (m, 2H), 7.42 – 7.36 (m, 2H), 7.24 – 7.18 (m, 1H), 6.91 (td, *J* = 7.5, 0.9 Hz, 1H), 6.85 (d, *J* = 8.2 Hz, 1H), 6.15 – 6.04 (m, 1H), 5.48 (dq, *J* = 17.3, 1.6 Hz, 1H), 5.33 (dq, *J* = 10.6, 1.3 Hz, 1H), 5.29 (dd, *J* = 8.4, 5.7 Hz, 1H), 4.62 (dt, *J* = 5.1, 1.5 Hz, 2H), 3.97 (dd, *J* = 17.4, 8.4 Hz, 1H), 3.76 (dd, *J* = 17.4, 5.7 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.8, 155.6, 146.0, 139.7, 135.1, 132.9, 131.1 (q, *J* = 307.3 Hz), 129.21, 129.17, 128.9, 128.6, 128.3, 128.2, 127.24, 127.21, 120.9, 117.6, 112.3, 69.1, 44.7, 40.5.

^{19}F NMR (565 MHz, $CDCl_3$) δ -41.08.

HRMS (APCI) calcd for $C_{24}H_{21}O_2^+$ $[M\text{-}SCF_3]^+$ *m/z*: 341.1536, found: 341.1533.



3t

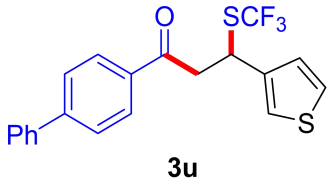
1-([1,1'-Biphenyl]-4-yl)-3-(2-allylphenyl)-3-((trifluoromethyl)thio)propan-1-one (**3t**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 62.5 mg product as a colorless oil in 73% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 – 7.95 (m, 2H), 7.68 – 7.65 (m, 2H), 7.61 – 7.58 (m, 2H), 7.48 – 7.43 (m, 2H), 7.41 – 7.37 (m, 2H), 7.23 – 7.18 (m, 3H), 6.04 (ddt, *J* = 14.3, 10.5, 6.4 Hz, 1H), 5.42 (dd, *J* = 8.1, 5.8 Hz, 1H), 5.15 – 5.06 (m, 2H), 3.88 (dd, *J* = 17.6, 8.1 Hz, 1H), 3.77 (dd, *J* = 17.6, 5.8 Hz, 1H), 3.70 – 3.61 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.3, 146.2, 139.6, 137.8, 137.1, 136.2, 134.9, 130.4, 130.3 (q, *J* = 308.0 Hz), 129.0, 128.6, 128.4, 128.2, 127.3, 127.2, 127.01, 126.94, 116.6, 45.7, 39.9, 37.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.00.

HRMS (APCI) calcd for $C_{25}H_{22}F_3OS^+$ $[M+H]^+$ *m/z*: 427.1338, found: 427.1334.



3u

1-([1,1'-Biphenyl]-4-yl)-3-(thiophen-3-yl)-3-((trifluoromethyl)thio)propan-1-one (**3u**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 40.6 mg product as a pale yellow solid in 52% yield.

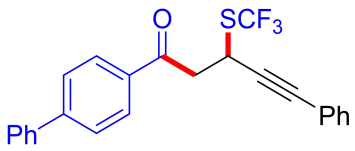
Mp = 82-85 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.02 – 7.98 (m, 2H), 7.70 – 7.67 (m, 2H), 7.63 – 7.59 (m, 2H), 7.48 – 7.44 (m, 2H), 7.42 – 7.38 (m, 1H), 7.21 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.09 – 7.06 (m, 1H), 6.90 (dd, *J* = 5.1, 3.6 Hz, 1H), 5.41 (dd, *J* = 8.1, 5.1 Hz, 1H), 3.86 (dd, *J* = 17.5, 8.1 Hz, 1H), 3.76 (dd, *J* = 17.5, 5.1 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.8, 146.4, 143.2, 139.6, 134.7, 130.3 (q, *J* = 308.1 Hz).129.0, 128.7, 128.4, 127.4, 127.2, 126.8, 126.3, 125.5, 46.1, 39.7, 39.7.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.43.

HRMS (APCI) calcd for $C_{20}H_{14}F_3OS_2^-$ $[M-H]^-$ *m/z*: 391.0444, found: 391.0441.



3v

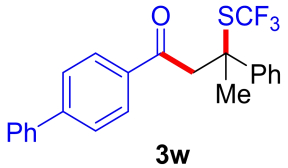
1-([1,1'-Biphenyl]-4-yl)-5-phenyl-3-((trifluoromethyl)thio)pent-4-yn-1-one (**3v**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 4/1) to give 35.3 mg product as a yellow oil in 43% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.07 – 8.04 (m, 2H), 7.74 – 7.69 (m, 2H), 7.64 – 7.61 (m, 2H), 7.49 – 7.45 (m, 3H), 7.44 – 7.40 (m, 1H), 7.38 – 7.35 (m, 2H), 7.31 – 7.25 (m, 3H), 4.88 (dd, *J* = 7.2, 5.9 Hz, 1H), 3.73 (dd, *J* = 17.2, 7.2 Hz, 1H), 3.67 (dd, *J* = 17.2, 5.9 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.4, 146.5, 139.6, 134.7, 131.7, 130.4 (d, *J* = 308.0 Hz), 129.0, 128.8, 128.7, 128.4, 128.2, 127.4, 127.3, 122.1, 85.9, 85.2, 44.7, 31.0 (q, *J* = 2.3 Hz).

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.70.

HRMS (APCI) calcd for $C_{24}H_{18}F_3OS^+$ $[M+H]^+$ *m/z*: 411.1025, found: 411.1021.



3w

1-([1,1'-Biphenyl]-4-yl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**3w**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 75.2 mg product as a white solid in 94% yield.

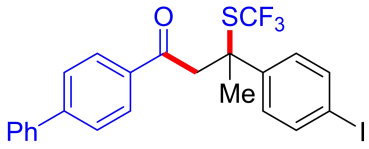
Mp = 76-78 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 – 7.92 (m, 2H), 7.69 – 7.64 (m, 2H), 7.63 – 7.59 (m, 2H), 7.56 – 7.52 (m, 2H), 7.50 – 7.45 (m, 2H), 7.42 – 7.38 (m, 1H), 7.36 – 7.31 (m, 2H), 7.28 – 7.24 (m, 1H), 4.23 (d, *J* = 17.3 Hz, 1H), 3.83 (d, *J* = 17.3 Hz, 1H), 2.26 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.9, 146.1, 141.5, 139.7, 135.6, 130.2 (q, *J* = 309.6 Hz). 129.0, 128.5, 128.5, 128.3, 127.8, 127.3, 127.2, 126.4, 54.8, 49.8, 27.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.02.

HRMS (APCI) calcd for $C_{22}H_{19}O^+$ $[M+H]^+$ *m/z*: 299.1430, found: 299.1427.



3x

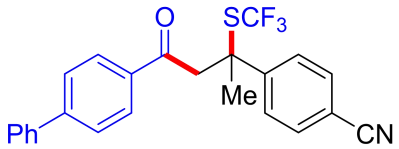
1-([1,1'-Biphenyl]-4-yl)-3-(4-iodophenyl)-3-((trifluoromethyl)thio)butan-1-one (**3x**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 88.5 mg product as a pale yellow oil in 84% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.68 – 7.63 (m, 4H), 7.63 – 7.59 (m, 2H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.43 – 7.39 (m, 1H), 7.30 – 7.25 (m, 2H), 4.15 (d, *J* = 17.4 Hz, 1H), 3.80 (d, *J* = 17.4 Hz, 1H), 2.20 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.5, 146.2, 141.6, 139.6, 137.5, 135.3, 130.0 (q, *J* = 309.8 Hz), 129.0, 128.5, 128.4, 127.3, 127.3, 93.6, 54.3, 49.6, 26.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -35.86.

HRMS (APCI) calcd for $C_{22}H_{18}IO^+$ $[M\text{-}SCF_3]^+$ *m/z*: 425.0397, found: 425.0390.



3y

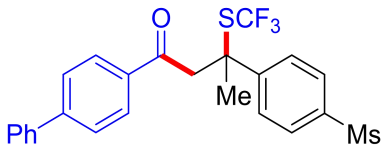
4-(4-([1,1'-Biphenyl]-4-yl)-4-oxo-2-((trifluoromethyl)thio)butan-2-yl)benzonitrile (**3y**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 10/1) to give 69.0 mg product as a colorless oil in 81% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95 – 7.91 (m, 2H), 7.70 – 7.58 (m, 8H), 7.50 – 7.45 (m, 2H), 7.43 – 7.39 (m, 1H), 4.14 (d, *J* = 17.6 Hz, 1H), 3.85 (d, *J* = 17.6 Hz, 1H), 2.23 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.1, 147.4, 146.5, 139.4, 135.0, 132.1, 129.8 (q, *J* = 309.6 Hz), 129.0, 128.5, 127.4, 127.3, 127.2, 118.4, 111.5, 54.0, 49.4, 26.8.

^{19}F NMR (565 MHz, $CDCl_3$) δ -35.72.

HRMS (APCI) calcd for $C_{21}H_{19}F_3NOS^+$ $[M+H]^+$ *m/z*: 426.1134, found: 426.1126.



3z

1-([1,1'-Biphenyl]-4-yl)-3-(4-(methylsulfonyl)phenyl)-3-((trifluoromethyl)thio)butan-1-one (**3z**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 3/1) to give 92.2 mg product as a colorless oil in 96% yield.

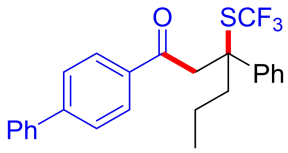
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 – 7.92 (m, 2H), 7.92 – 7.89 (m, 2H), 7.77 – 7.73 (m, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.61 (dd, *J* = 5.2, 3.3 Hz, 2H), 7.50 – 7.45 (m, 2H), 7.43 – 7.39 (m, 1H), 4.18 (d, *J* =

17.6 Hz, 1H), 3.87 (d, J = 17.6 Hz, 1H), 3.03 (s, 3H), 2.25 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.2, 148.4, 146.4, 139.5, 139.4, 135.0, 129.9 (q, J = 309.6 Hz), 129.0, 128.5, 127.6, 127.5, 127.4, 127.2, 53.9, 49.5, 44.4, 27.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -35.68.

HRMS (APCI) calcd for $C_{24}H_{22}F_3O_3S_2^+$ $[M+H]^+$ *m/z*: 479.0957, found: 479.0952.



3aa

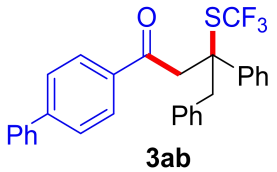
1-([1,1'-Biphenyl]-4-yl)-3-phenyl-3-((trifluoromethyl)thio)hexan-1-one (**3aa**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 75.8 mg product as a pale yellow oil in 88% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 – 7.95 (m, 2H), 7.68 – 7.64 (m, 2H), 7.63 – 7.60 (m, 2H), 7.56 – 7.52 (m, 2H), 7.49 – 7.45 (m, 2H), 7.42 – 7.38 (m, 1H), 7.34 – 7.30 (m, 2H), 7.26 – 7.22 (m, 1H), 3.96 (q, J = 17.4 Hz, 2H), 2.51 (tdd, J = 26.2, 14.2, 4.4 Hz, 2H), 1.58 – 1.50 (m, 1H), 1.33 – 1.25 (m, 1H), 0.95 (t, J = 7.3 Hz, 3H)

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.0, 146.0, 142.1, 139.7, 135.8, 130.3 (q, J = 309.1 Hz), 129.0, 128.5, 128.3, 128.2, 127.4, 127.26, 127.25, 126.8, 58.7, 44.7, 40.4, 18.1, 14.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -35.80.

HRMS (APCI) calcd for $C_{25}H_{24}F_3OS^+$ $[M+H]^+$ *m/z*: 429.1494, found: 429.1485.



3ab

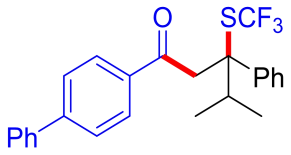
1-([1,1'-Biphenyl]-4-yl)-3,4-diphenyl-3-((trifluoromethyl)thio)butan-1-one (**3ab**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 40.3 mg product as a colorless oil in 42% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, J = 8.3 Hz, 2H), 7.60 (d, J = 8.3 Hz, 2H), 7.56 – 7.52 (m, 2H), 7.46 (d, J = 7.6 Hz, 2H), 7.39 (t, J = 7.6 Hz, 2H), 7.32 (t, J = 7.4 Hz, 1H), 7.28 (t, J = 7.7 Hz, 2H), 7.21 (t, J = 7.3 Hz, 1H), 7.13 – 7.08 (m, 1H), 7.06 (t, J = 7.4 Hz, 2H), 6.85 (d, J = 7.5 Hz, 2H), 3.84 (d, J = 13.5 Hz, 1H), 3.76 (d, J = 13.5 Hz, 1H), 3.71 (s, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.6, 146.0, 141.8, 139.7, 135.9, 135.4, 130.9, 130.2 (q, J = 308.7 Hz), 129.0, 128.4, 128.3, 128.2, 128.0, 127.6, 127.30, 127.25, 127.22, 127.17, 58.2, 44.1, 42.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -35.49.

HRMS (APCI) calcd for $C_{28}H_{23}O^+$ $[M-SCF_3]^+$ *m/z*: 375.1743, found: 375.1736.



3ac

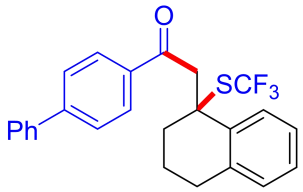
1-([1,1'-Biphenyl]-4-yl)-4-methyl-3-phenyl-3-((trifluoromethyl)thio)pentan-1-one (**3ac**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 28.7 mg product as a colorless oil in 33% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 – 7.91 (m, 2H), 7.62 – 7.58 (m, 2H), 7.57 – 7.52 (m, 2H), 7.51 – 7.47 (m, 2H), 7.42 – 7.37 (m, 2H), 7.35 – 7.30 (m, 1H), 7.29 – 7.24 (m, 2H), 7.22 – 7.18 (m, 1H), 4.09 (d, J = 17.9 Hz, 1H), 3.98 (d, J = 17.9 Hz, 1H), 2.96 (hept, J = 6.7 Hz, 1H), 1.49 (s, 1H), 0.97 (d, J = 6.7 Hz, 3H), 0.82 (d, J = 6.8 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.2, 145.8, 139.8, 138.9, 136.2, 130.7 (q, J = 309.0 Hz), 129.0, 128.4, 128.3, 128.1, 127.7, 127.34, 127.25, 64.2, 43.9, 35.2, 18.7, 18.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -34.56.

HRMS (APCI) calcd for $C_{24}H_{23}O^+$ $[M\text{-}CF_3S]^+$ *m/z*: 327.1743, found: 327.1738.



3ad

1-([1,1'-Biphenyl]-4-yl)-2-(1-((trifluoromethyl)thio)-1,2,3,4-tetrahydronaphthalen-1-yl)ethan-1-one (**3ad**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 55.5 mg product as a white solid in 65% yield.

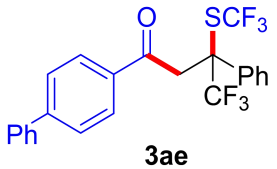
Mp = 94-96 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, J = 8.4 Hz, 2H), 7.69 – 7.65 (m, 2H), 7.63 – 7.59 (m, 2H), 7.49 – 7.44 (m, 2H), 7.43 – 7.38 (m, 1H), 7.29 (d, J = 7.9 Hz, 1H), 7.17 – 7.12 (m, 2H), 7.08 – 7.03 (m, 1H), 4.57 (d, J = 18.0 Hz, 1H), 3.93 (d, J = 17.9 Hz, 1H), 3.07 – 2.99 (m, 1H), 2.92 – 2.85 (m, 1H), 2.77 – 2.66 (m, 1H), 2.40 (d, J = 14.5 Hz, 1H), 2.29 (qdd, J = 12.8, 4.9, 2.6 Hz, 1H), 2.00 – 1.91 (m, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.0, 146.0, 139.7, 138.0, 135.7, 135.3, 130.0 (q, J = 310.5 Hz), 129.8, 129.0, 128.4, 128.3, 127.9, 127.3, 127.2, 126.5, 126.2, 56.7, 49.5, 34.3, 29.8, 19.4.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.14.

HRMS (APCI) calcd for $C_{25}H_{23}F_3OS^+$ $[M+H]^+$ *m/z*: 427.1338, found: 427.1331.



3ae

1-([1,1'-Biphenyl]-4-yl)-4,4,4-trifluoro-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**3ae**) was

synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 5/1) to give 49.3 mg product as a white solid in 46% yield.

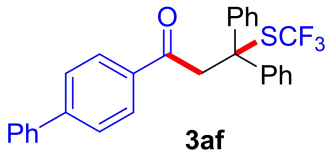
Mp = 106-109 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.2 Hz, 2H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.62 (d, *J* = 7.7 Hz, 2H), 7.54 – 7.50 (m, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.4 Hz, 1H), 7.39 – 7.36 (m, 3H), 4.31 (d, *J* = 18.5 Hz, 1H), 4.22 (d, *J* = 18.5 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 191.6, 146.5, 139.6, 135.1, 132.6, 129.2, 129.0, 129.0 (q, *J* = 310.6 Hz), 128.6, 128.50, 128.45, 127.52, 127.46, 127.3, 125.2 (q, *J* =282.9 Hz), 60.0 (q, *J* = 28.3 Hz), 39.6.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.17 (q, *J* = 8.0 Hz), -70.33 (q, *J* = 8.2 Hz).

HRMS (APCI) calcd for $C_{23}H_{17}F_6OS^+$ $[M+H]^+$ *m/z*: 455.0899, found: 455.0893.



3af

1-([1,1'-Biphenyl]-4-yl)-3,3-diphenyl-3-((trifluoromethyl)thio)propan-1-one (**3af**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 80.1 mg product as a yellow solid in 87% yield.

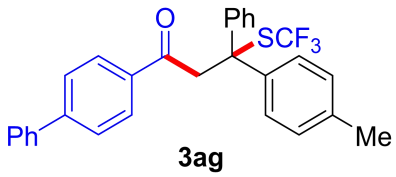
Mp = 102-104 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.59 – 7.55 (m, 2H), 7.51 – 7.47 (m, 4H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 1H), 7.32 – 7.28 (m, 4H), 7.27 – 7.23 (m, 2H), 4.56 (s, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.6, 145.9, 141.3, 139.6, 135.5, 129.8 (q, *J* = 309.7 Hz), 128.9, 128.8, 128.5, 128.3, 128.0, 127.7, 127.19, 127.16, 62.2, 50.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.45.

HRMS (APCI) calcd for $C_{28}H_{20}F_3OS^-$ $[M-H]^-$ *m/z*: 461.1192, found: 461.1190.



3ag

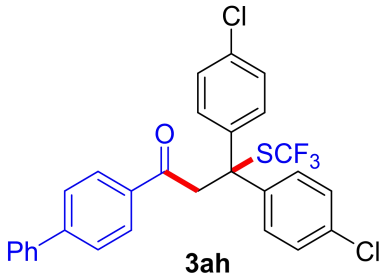
1-([1,1'-Biphenyl]-4-yl)-3-phenyl-3-(p-tolyl)-3-((trifluoromethyl)thio)propan-1-one (**3ag**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 3/1) to give 84.2 mg product as a colorless oil in 88% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 – 7.88 (m, 2H), 7.64 – 7.55 (m, 4H), 7.52 – 7.43 (m, 4H), 7.41 – 7.34 (m, 3H), 7.33 – 7.28 (m, 2H), 7.27 – 7.23 (m, 1H), 7.11 (d, *J* = 8.1 Hz, 2H), 4.54 (s, 2H), 2.31 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.7, 145.9, 141.4, 139.7, 138.3, 137.5, 135.6, 129.9 (q, *J* = 309.7 Hz), 128.9, 128.8, 128.7, 128.6, 128.5, 128.3, 127.9, 127.6, 127.20, 127.16, 62.1, 50.1, 21.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.46.

HRMS (APCI) calcd for $C_{28}H_{23}O^+$ $[M-SCF_3]^+$ *m/z*: 375.1743, found: 375.1739.



3ah

1-([1,1'-Biphenyl]-4-yl)-3,3-bis(4-chlorophenyl)-3-((trifluoromethyl)thio)propan-1-one (**3ah**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 3/1) to give 98.5 mg product as a white solid in 93% yield.

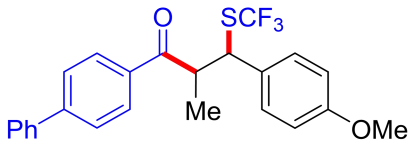
Mp = 129-131 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 – 7.90 (m, 2H), 7.69 – 7.65 (m, 2H), 7.64 – 7.60 (m, 2H), 7.50 – 7.47 (m, 2H), 7.44 – 7.40 (m, 5H), 7.34 – 7.28 (m, 4H), 4.53 (s, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.2, 146.3, 139.5, 139.4, 135.1, 133.9, 130.2, 129.5 (q, J = 309.6 Hz), 129.0, 128.5, 128.4, 128.3, 127.3, 127.2, 61.2, 50.2.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.34.

HRMS (APCI) calcd for $C_{29}H_{19}Cl_2O^+$ $[M\text{-}SCF_3]^+$ *m/z*: 429.0807, found: 429.0805.



3ai

1-([1,1'-Biphenyl]-4-yl)-3-(4-methoxyphenyl)-2-methyl-3-((trifluoromethyl)thio)propan-1-one (**3ai**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to give 35.6 mg product (*E*/*Z* = 7/3) as a white solid in 41% yield.

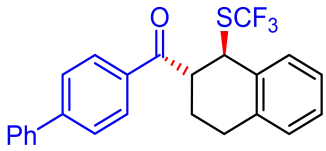
Mp = 70-72 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03 (d, J = 8.4 Hz, 2H, minor), 7.84 (d, J = 8.5 Hz, 2H, major), 7.70 (d, J = 8.5 Hz, 2H, minor), 7.64 – 7.60 (m, 2H), 7.59 – 7.56 (m, 2H, major), 7.50 – 7.43 (m, 2H), 7.43 – 7.37 (m, 1H), 7.32 – 7.29 (m, 1H, minor), 7.27 – 7.22 (m, 2H, major), 6.88 (d, J = 8.7 Hz, 1H, minor), 6.74 (d, J = 8.8 Hz, 1H, major), 4.75 (d, J = 10.5 Hz, 1H, major), 4.67 (d, J = 8.8 Hz, 1H, minor), 4.10 – 4.03 (m, 1H, major), 4.01 – 3.96 (m, 1H, minor), 3.79 (s, 3H, minor), 3.70 (s, 3H, major), 1.48 (d, J = 6.9 Hz, 3H, major), 1.15 (d, J = 7.0 Hz, 3H, minor).

^{13}C NMR (151 MHz, $CDCl_3$) δ 200.5 (200.7), 158.9 (159.2), 146.0 (146.3), 139.6 (139.7), 134.6 (134.8), 132.7 (131.6), 130.37 (q, J = 307.4 Hz), 128.9, 128.8 (129.2), 128.7 (129.0), 128.3 (128.4), 127.3 (127.5), 127.2 (127.3), 113.9 (114.0), 55.1 (55.2), 52.2 (51.2), 46.0 (45.9), 17.5 (17.4).

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.35 (major), -40.59 (minor).

HRMS (APCI) calcd for $C_{24}H_{20}F_3O_2S^+$ $[M+H]^+$ *m/z*: 429.1142, found: 429.1138.



3aj

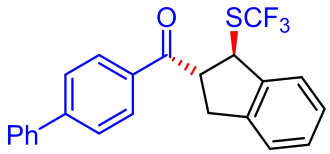
cis-[1,1'-Biphenyl]-4-yl(1-((trifluoromethyl)thio)-1,2,3,4-tetrahydronaphthalen-2-yl)methanone (**3aj**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 55.5 mg product as a colorless oil in 65% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.3 Hz, 2H), 7.71 (d, *J* = 8.2 Hz, 2H), 7.63 (d, *J* = 7.4 Hz, 2H), 7.56 (d, *J* = 7.8 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.3 Hz, 1H), 7.24 (t, *J* = 7.5 Hz, 1H), 7.18 (t, *J* = 7.4 Hz, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 5.16 (d, *J* = 4.1 Hz, 1H), 4.26 (dt, *J* = 6.1, 4.3 Hz, 1H), 2.83 (dt, *J* = 16.8, 5.3 Hz, 1H), 2.68 – 2.60 (m, 1H), 2.44 (ddt, *J* = 14.3, 9.5, 4.7 Hz, 1H), 2.19 – 2.11 (m, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 199.8, 146.2, 139.7, 136.4, 134.1, 132.7, 130.7, 130.6 (q, *J* = 308.4 Hz), 129.0, 128.9, 128.9, 128.3, 127.6, 127.5, 127.3, 126.7, 48.8, 44.9, 26.4, 24.2.

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.43.

HRMS (APCI) calcd for $C_{24}H_{20}F_3OS^+$ $[M+H]^+$ *m/z*: 413.1181, found: 413.1175.



3ak

cis-[1,1'-Biphenyl]-4-yl(1-((trifluoromethyl)thio)-2,3-dihydro-1H-inden-2-yl)methanone (**3ak**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 40.2 mg product as a white solid in 50% yield.

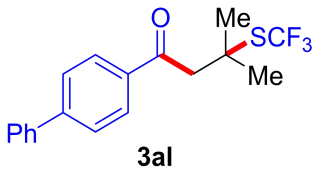
Mp = 105-107 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03 – 7.98 (m, 2H), 7.67 – 7.63 (m, 2H), 7.59 – 7.54 (m, 2H), 7.42 – 7.37 (m, 3H), 7.36 – 7.31 (m, 1H), 7.24 – 7.17 (m, 2H), 7.10 (d, *J* = 6.6 Hz, 1H), 5.50 (d, *J* = 5.1 Hz, 1H), 4.47 (dt, *J* = 10.2, 5.8 Hz, 1H), 3.56 (dd, *J* = 16.2, 9.9 Hz, 1H), 3.03 (dd, *J* = 16.2, 6.3 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.0, 146.3, 140.3, 139.7, 139.3, 134.3, 130.5 (d, *J* = 308.1 Hz), 129.2, 129.0, 128.9, 128.4, 127.7, 127.5, 127.3, 125.4, 124.6, 55.1, 49.7, 36.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.62.

HRMS (APCI) calcd for $C_{23}H_{18}F_3OS^+$ $[M+H]^+$ *m/z*: 399.1025, found: 399.1020.



3al

1-([1,1'-Biphenyl]-4-yl)-3-methyl-3-((trifluoromethyl)thio)butan-1-one (**3al**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 14.9 mg product as a

white solid in 22% yield.

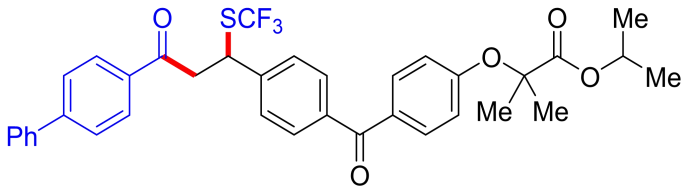
Mp = 90-92 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.5 Hz, 2H), 7.71 – 7.68 (m, 2H), 7.64 – 7.61 (m, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.4 Hz, 1H), 3.50 (s, 2H), 1.70 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 196.2, 146.1, 139.7, 136.0, 131.0 (q, *J* = 307.9 Hz), 129.0, 128.7, 128.3, 127.3, 127.3, 49.8, 49.5, 29.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -35.40.

HRMS (APCI) calcd for $C_{18}H_{18}F_3OS^+$ $[M+H]^+$ *m/z*: 339.1025, found: 339.1020.



3am

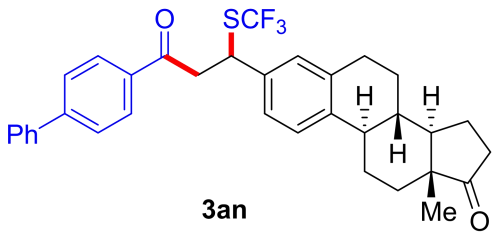
Isopropyl 2-(4-(4-(3-([1,1'-biphenyl]-4-yl)-3-oxo-1-((trifluoromethyl)thio)propyl)benzoyl)phenoxy)-2-methylpropanoate (**3am**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 25/1) to give 106.2 mg product as a yellow oil in 84% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 – 7.95 (m, 2H), 7.75 – 7.71 (m, 4H), 7.69 – 7.65 (m, 2H), 7.62 – 7.59 (m, 2H), 7.55 (d, *J* = 8.3 Hz, 2H), 7.48 – 7.43 (m, 2H), 7.42 – 7.37 (m, 1H), 6.87 – 6.83 (m, 2H), 5.19 (dd, *J* = 8.5, 5.2 Hz, 1H), 5.08 (hept, *J* = 6.2 Hz, 1H), 3.85 (dd, *J* = 17.6, 8.5 Hz, 1H), 3.74 (dd, *J* = 17.6, 5.2 Hz, 1H), 1.66 (s, 6H), 1.19 (d, *J* = 6.3 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.62, 194.61, 173.0, 159.6, 146.4, 144.0, 139.5, 137.7, 134.6, 131.9, 130.3, 130.22, 130.15 (q, *J* = 308.0 Hz), 128.9, 128.6, 128.4, 127.5, 127.3, 127.2, 117.1, 79.3, 69.2, 44.8, 43.8, 25.31 (25.27), 21.43.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.06.

HRMS (APCI) calcd for $C_{36}H_{34}F_3O_5S^+$ $[M+H]^+$ *m/z*: 635.2074, found: 635.2065.



3an

(8*S*,9*R*,13*R*,14*R*)-3-(3-([1,1'-Biphenyl]-4-yl)-3-oxo-1-((trifluoromethyl)thio)propyl)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[a]phenanthren-17-one (**3an**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 99.8 mg product as a white solid in 88% yield.

Mp = 66-67 °C

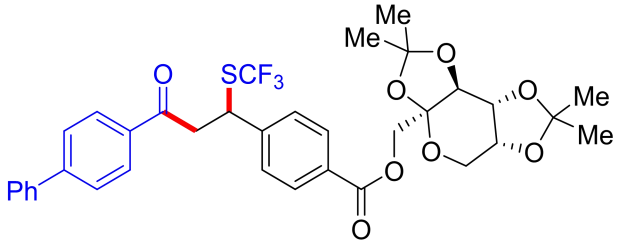
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.69 – 7.65 (m, 2H), 7.62 – 7.57 (m, 2H), 7.49 – 7.44 (m, 2H), 7.42 – 7.38 (m, 1H), 7.23 (dd, *J* = 8.1, 2.0 Hz, 1H), 7.18 (t, *J* = 7.1 Hz, 1H), 7.13 (d, *J*

= 2.9 Hz, 1H), 5.08 (dd, *J* = 8.2, 5.5 Hz, 1H), 3.84 (dd, *J* = 17.5, 8.6 Hz, 1H), 3.70 (dd, *J* = 17.5, 5.2 Hz, 1H), 2.95 – 2.82 (m, 2H), 2.49 (dd, *J* = 19.1, 8.8 Hz, 1H), 2.40 – 2.34 (m, 1H), 2.25 (t, *J* = 10.4 Hz, 1H), 2.17 – 2.08 (m, 1H), 2.08 – 1.97 (m, 2H), 1.94 (dd, *J* = 11.5, 1.9 Hz, 1H), 1.66 – 1.38 (m, 6H), 0.88 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 220.7, 195.21 (195.19), 146.2, 139.7, 139.6, 137.05 (137.03), 136.9, 134.9, 130.4 (q, *J* = 307.9 Hz), 129.0, 128.6, 128.4, 128.09 (128.08), 127.3, 127.2, 125.83 (125.81), 124.66 (124.65), 50.5, 47.9, 45.30 (45.25), 44.3, 44.0, 37.9, 35.8, 31.5, 29.30 (29.28), 26.3, 25.5, 21.5, 13.8.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.10.

HRMS (APCI) calcd for $C_{34}H_{34}F_3O_2S^+$ $[M+H]^+$ *m/z*: 563.2226, found: 563.2216.



3ao

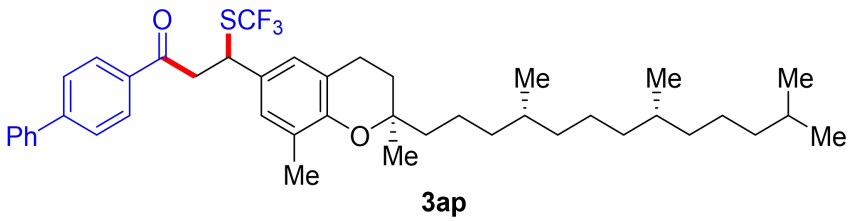
((3a*S*,5a*R*,8a*R*,8b*S*)-2,2,7,7-Tetramethyltetrahydro-3aH-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-3a-yl)methyl 4-(3-([1,1'-biphenyl]-4-yl)-3-oxo-1-((trifluoromethyl)thio)propyl)benzoate (**3ao**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 4/1) to give 102.6 mg product as a sticky colorless oil in 76% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.4 Hz, 2H), 7.98 – 7.94 (m, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.62 – 7.59 (m, 2H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.49 – 7.45 (m, 2H), 7.43 – 7.39 (m, 1H), 5.15 (dd, *J* = 8.4, 5.4 Hz, 1H), 4.64 (d, *J* = 11.9 Hz, 1H), 4.62 (dd, *J* = 5.3, 2.6 Hz, 1H), 4.44 (d, *J* = 2.6 Hz, 1H), 4.32 (d, *J* = 11.8 Hz, 1H), 4.25 (dd, *J* = 7.9, 1.2 Hz, 1H), 3.94 (dd, *J* = 13.0, 1.9 Hz, 1H), 3.82 – 3.75 (m, 2H), 3.74 – 3.67 (m, 1H), 1.54 (s, 3H), 1.43 (s, 3H), 1.37 (s, 3H), 1.33 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194. 6, 165.4, 146.5, 145.3, 139.5, 134.6, 130.3, 130.1 (q, *J* = 308.1 Hz), 129.6, 129.0, 128.6, 128.4, 127.7, 127.4, 127.2, 109.1, 108.8, 101.6, 70.7, 70.6, 70.1, 65.5, 61.3, 44.7, 43.8, 26.5, 25.8, 25.5, 24.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.08.

HRMS (APCI) calcd for $C_{35}H_{36}F_3O_8S^+$ $[M+H]^+$ *m/z*: 673.2077, found: 673.2065.



3ap

1-([1,1'-Biphenyl]-4-yl)-3-((*R*)-2,8-dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl)-3-((trifluoromethyl)thio)propan-1-one (**3ap**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 78.5 mg product as a sticky yellow oil in 56% yield.

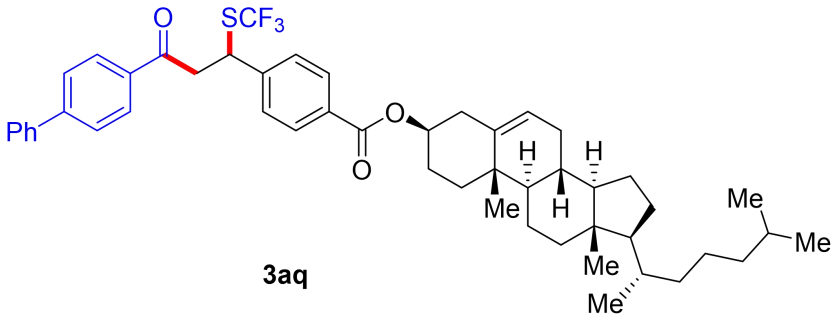
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.68 – 7.64 (m, 2H), 7.61 – 7.58 (m, 2H), 7.48

– 7.43 (m, 2H), 7.41 – 7.36 (m, 1H), 6.97 (s, 1H), 6.92 (s, 1H), 5.05 (dd, *J* = 8.6, 5.3 Hz, 1H), 3.83 (dd, *J* = 17.4, 8.6 Hz, 1H), 3.68 (dd, *J* = 17.4, 5.2 Hz, 1H), 2.76 – 2.62 (m, 2H), 2.13 (s, 3H), 1.77 (dt, *J* = 13.8, 7.0 Hz, 1H), 1.73 – 1.67 (m, 1H), 1.55 – 1.49 (m, 3H), 1.45 – 1.02 (m, 18H), 0.86 (d, *J* = 6.6 Hz, 6H), 0.85 (d, *J* = 3.6 Hz, 3H), 0.84 (d, *J* = 3.6 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.5, 152.0, 146.1, 139.7, 135.1, 130.4 (q, *J* = 308.0 Hz), 129.1, 129.0, 128.6, 128.3, 127.3, 127.2, 126.7, 126.1, 126.0, 120.6, 76.3, 45.7, 44.2, 40.34 (40.29), 39.4, 37.43, 37.41, 37.39, 37.3, 32.8, 32.7, 30.96 (30.95), 28.0, 24.8, 24.4, 24.22 (24.18), 22.7, 22.6, 22.3, 21.0, 19.7, 19.6, 16.2.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.06.

HRMS (APCI) calcd for $C_{42}H_{57}O_2^+$ [M-SCF_3]$^+$ *m/z*: 593.4353, found: 593.4346.



3aq

(3*R*,8*R*,9*R*,10*S*,13*S*,14*R*,17*S*)-10,13-Dimethyl-17-((*S*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 4-(3-([1,1'-biphenyl]-4-yl)-3-oxo-1-((trifluoromethyl)thio)propyl)benzoate (**3aq**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 80.2 mg product as a white solid in 50% yield.

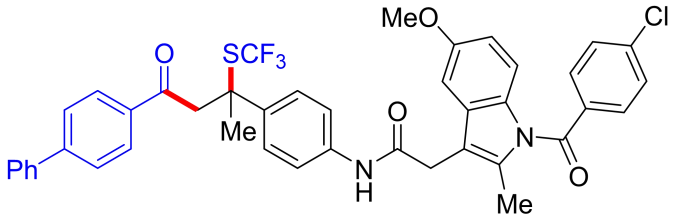
Mp = 162-164 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03 – 7.98 (m, 2H), 7.98 – 7.93 (m, 2H), 7.68 – 7.65 (m, 2H), 7.62 – 7.58 (m, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.46 (dd, *J* = 10.3, 4.8 Hz, 2H), 7.42 – 7.38 (m, 1H), 5.40 (d, *J* = 4.4 Hz, 1H), 5.15 (dd, *J* = 8.5, 5.4 Hz, 1H), 4.90 – 4.79 (m, 1H), 3.80 (dd, *J* = 17.5, 8.6 Hz, 1H), 3.71 (dd, *J* = 17.5, 5.3 Hz, 1H), 2.43 (d, *J* = 7.8 Hz, 2H), 2.04 – 1.93 (m, 3H), 1.90 (dt, *J* = 13.3, 3.4 Hz, 1H), 1.87 – 1.80 (m, 1H), 1.75 – 1.67 (m, 1H), 1.61 – 1.42 (m, 6H), 1.41 – 1.31 (m, 3H), 1.30 – 1.04 (m, 11H), 1.03 – 0.94 (m, 3H), 0.8 (d, *J* = 6.5 Hz, 3H), 0.87 (d, *J* = 6.6 Hz, 3H), 0.86 (d, *J* = 6.6 Hz, 3H), 0.68 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.6, 165.4, 146.5, 144.8, 139.6, 139.5, 134.7, 133.2, 131.2, 130.6, 130.1, 129.1, 129.0, 128.6, 128.4, 128.2, 127.6, 127.4, 127.2, 127.1, 122.8, 74.7, 56.7, 56.1, 50.0, 44.8, 43.9, 42.3, 39.7, 39.5, 38.2, 37.0, 36.6, 36.2, 35.8, 31.9, 31.9, 28.2, 28.0, 27.8, 24.3, 23.8, 22.8, 22.5, 21.0, 19.3, 18.7, 11.8.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.05.

HRMS (APCI) calcd for $C_{50}H_{60}F_3O_3S^-$ [M-H]$^-$ *m/z*: 797.4221, found: 797.4208.



3ar

N-(4-(4-([1,1'-biphenyl]-4-yl)-4-oxo-2-((trifluoromethyl)thio)butan-2-yl)phenyl)-2-(1-(4-chloro benzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetamide (**3ar**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 137.1 mg product as a white solid in 91% yield.

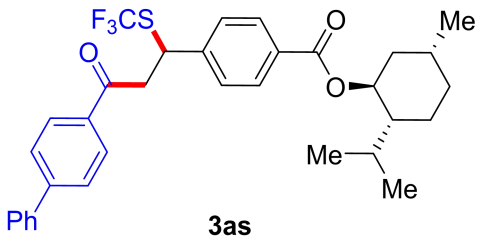
Mp = 100-102°C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 – 7.89 (m, 2H), 7.66 – 7.61 (m, 4H), 7.60 – 7.57 (m, 2H), 7.51 (s, 1H), 7.48 – 7.43 (m, 4H), 7.43 – 7.35 (m, 5H), 6.93 (d, *J* = 2.5 Hz, 1H), 6.87 (d, *J* = 9.0 Hz, 1H), 6.69 (dd, *J* = 9.0, 2.5 Hz, 1H), 4.16 (d, *J* = 17.4 Hz, 1H), 3.83 – 3.77 (m, 4H), 3.75 (s, 2H), 2.40 (s, 3H), 2.19 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.8, 168.33, 168.29, 156.4, 146.1, 139.64, 139.62, 137.6, 137.0, 136.7, 135.5, 133.5, 131.2, 130.2 (q, *J* = 309.6 Hz).131.0, 130.2, 129.3, 129.0, 128.5, 128.4, 127.32, 127.27, 127.15, 119.8, 115.3, 112.5, 112.3, 100.81, 55.8, 54.5, 49.7, 33.3 27.12, 13.4.

^{19}F NMR (565 MHz, $CDCl_3$) δ -35.97.

HRMS (APCI) calcd for $C_{42}H_{35}ClF_3N_2O_4S^+$ $[M+H]^+$ *m/z*: 755.1953, found: 755.1936.



3as

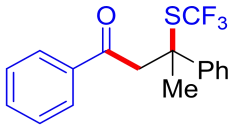
(1*S*,2*R*,5*R*)-2-Isopropyl-5-methylcyclohexyl 4-(3-([1,1'-biphenyl]-4-yl)-3-oxo-1-((trifluoromethyl)thio) propyl)benzoate (**3as**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to give 96.7 mg product as a sticky yellow oil in 85% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.4 Hz, 2H), 7.96 (d, *J* = 8.0 Hz, 2H), 7.67 (d, *J* = 8.3 Hz, 2H), 7.61 – 7.58 (m, 2H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.48 – 7.44 (m, 2H), 7.41 – 7.38 (m, 1H), 5.16 (dd, *J* = 8.4, 5.3 Hz, 1H), 4.91 (td, *J* = 10.9, 4.3 Hz, 1H), 3.81 (ddd, *J* = 17.5, 8.6, 3.1 Hz, 1H), 3.71 (dt, *J* = 17.5, 5.0 Hz, 1H), 2.09 (d, *J* = 12.2 Hz, 1H), 1.99 – 1.90 (m, 1H), 1.73 – 1.69 (m, 2H), 1.56 – 1.50 (m, 2H), 1.14 – 1.04 (m, 2H), 0.94 – 0.89 (m, 7H), 0.77 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.60 (194.57), 165.4, 146.4, 144.75 (144.73), 139.5, 134.6, 130.6, 130.14 (q, *J* = 308.1 Hz), 130.08, 129.0, 128.6, 128.4, 127.6, 127.4, 127.2, 74.9, 47.2, 44. 8, 43.8, 40.9, 34.2, 31.4, 26.39 (26.38), 23.53 (23.51), 22.0, 20.7, 16.42 (16.40).

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.067, -40.074.

HRMS (APCI) calcd for $C_{33}H_{35}F_3O_3S^-$ $[M-H]^-$ *m/z*: 567.2186, found: 567.2172.



4a

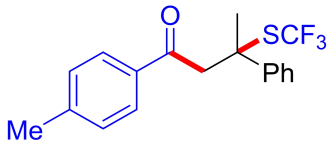
1,3-Diphenyl-3-((trifluoromethyl)thio)butan-1-one (**4a**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 52.4 mg product as a yellow oil in 81% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 – 7.76 (m, 2H), 7.48 – 7.41 (m, 3H), 7.37 – 7.32 (m, 2H), 7.25 – 7.21 (m, 2H), 7.18 – 7.14 (m, 1H), 4.12 (d, J = 17.4 Hz, 1H), 3.72 (d, J = 17.4 Hz, 1H), 2.15 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.3, 141.5, 136.9, 133.3, 130.2 (q, J = 309.6 Hz), 128.6, 128.4, 127.9, 127.7, 126.3, 54.7, 49.7, 27.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.01.

HRMS (APCI) calcd for $C_{16}H_{15}O^+$ $[M\text{-}SCF_3]^+$ *m/z*: 223.1117, found: 223.1117.



4b

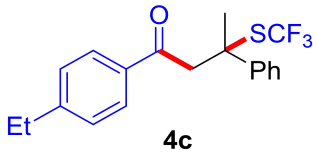
3-Phenyl-1-(p-tolyl)-3-((trifluoromethyl)thio)butan-1-one (**4b**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 44.4 mg product as a colorless oil in 66% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (d, J = 8.2 Hz, 2H), 7.46 – 7.41 (m, 2H), 7.26 – 7.21 (m, 2H), 7.19 – 7.13 (m, 3H), 4.09 (d, J = 17.3 Hz, 1H), 3.69 (d, J = 17.3 Hz, 1H), 2.31 (s, 3H), 2.15 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.9, 144.3, 141.6, 134.5, 130.2 (q, J = 309.6 Hz), 129.3, 128.4, 128.0, 127.7, 126.4, 54.8, 49.6, 27.0, 21.6.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.04.

HRMS (APCI) calcd for $C_{17}H_{17}O^+$ $[M\text{-}SCF_3]^+$ *m/z*: 237.1274, found: 237.1270.



4c

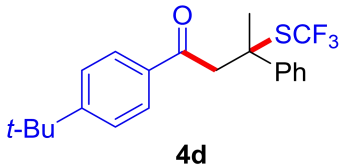
1-(4-Ethylphenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4c**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 5/1) to give 49.5 mg product as a colorless oil in 70% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, J = 8.3 Hz, 2H), 7.55 – 7.48 (m, 2H), 7.31 (dd, J = 10.5, 4.9 Hz, 2H), 7.27 – 7.21 (m, 3H), 4.17 (d, J = 17.4 Hz, 1H), 3.78 (d, J = 17.3 Hz, 1H), 2.69 (q, J = 7.6 Hz, 2H), 2.23 (s, 3H), 1.24 (t, J = 7.6 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.9, 141.6, 134.7, 130.2 (q, J = 309.5 Hz), 128.4, 128.1, 127.7, 126.4, 54.8, 49.6, 28.9, 27.0, 15.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.03.

HRMS (APCI) calcd for $C_{18}H_{19}O^+$ $[M\text{-}SCF_3]^+$ *m/z*: 251.1430, found: 251.1430.



4d

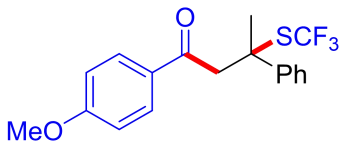
1-(4-(Tert-butyl)phenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4d**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 5/1) to give 59.3 mg product as a yellow oil in 78% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 – 7.71 (m, 2H), 7.45 – 7.41 (m, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.23 (t, *J* = 7.7 Hz, 2H), 7.17 – 7.13 (m, 1H), 4.10 (d, *J* = 17.4 Hz, 1H), 3.70 (dd, *J* = 17.4, 0.6 Hz, 1H), 2.15 (s, 3H), 1.24 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.9, 157.2, 141.6, 134.4, 130.2 (q, *J* = 309.5 Hz), 128.4, 127.9, 127.7, 126.3, 125.6, 54.8, 49.6, 35.1, 31.0, 27.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.01.

HRMS (APCI) calcd for $C_{20}H_{23}O^+$ $[M\text{-}SCF_3]^+$ *m/z*: 279.1743, found: 279.1742.



4e

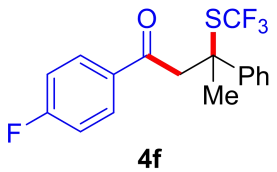
1-(4-Methoxyphenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4e**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 3/1) to give 17.9 mg product as a colorless oil in 25% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 – 7.82 (m, 2H), 7.55 – 7.47 (m, 2H), 7.34 – 7.29 (m, 2H), 7.26 – 7.22 (m, 1H), 6.92 – 6.88 (m, 2H), 4.13 (d, *J* = 17.1 Hz, 1H), 3.86 (s, 3H), 3.74 (d, *J* = 17.1 Hz, 1H), 2.23 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 193.8, 163.7, 141.7, 130.24, 130.24 (q, *J* = 309.5 Hz), 130.0, 128.4, 127.7, 126.4, 113.8, 55.5, 54.9, 49.4, 27.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.05.

HRMS (APCI) calcd for $C_{18}H_{18}F_3O_2S^+$ $[M+H]^+$ *m/z*: 355.0974, found: 355.0971.



4f

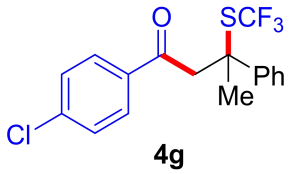
1-(4-Fluorophenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4f**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 58.4 mg product as a colorless oil in 85% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 – 7.77 (m, 2H), 7.47 – 7.39 (m, 2H), 7.27 – 7.21 (m, 2H), 7.19 – 7.14 (m, 1H), 7.05 – 6.97 (m, 2H), 4.08 (d, *J* = 17.3 Hz, 1H), 3.68 (d, *J* = 17.3 Hz, 1H), 2.15 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 193.7, 165.8 (d, J = 255.2 Hz), 141.4, 133.3 (d, J = 2.8 Hz), 130.2 (q, J = 309.6 Hz), 130.6 (d, J = 9.4 Hz), 128.5, 127.8, 126.3, 115.8 (d, J = 22.1 Hz), 54.7, 49.7, 27.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.02 (s, 3F), -104.34 – -104.68 (m, 1F).

HRMS (APCI) calcd for $C_{16}H_{14}FO^+$ [M-SCF_3]$^+$ *m/z*: 241.1023, found: 241.1018.



4g

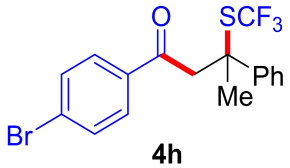
1-(4-Chlorophenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4g**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 50/1) to give 58.7 mg product as a colorless oil in 82% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74 – 7.67 (m, 2H), 7.44 – 7.39 (m, 2H), 7.33 – 7.29 (m, 2H), 7.24 (t, J = 7.8 Hz, 2H), 7.19 – 7.14 (m, 1H), 4.07 (d, J = 17.3 Hz, 1H), 3.67 (d, J = 17.3 Hz, 1H), 2.14 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.1, 141.3, 139.9, 135.2, 130.2 (q, J = 309.6 Hz), 129.3, 129.0, 128.5, 127.8, 126.3, 54.6, 49.7, 27.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.01.

HRMS (APCI) calcd for $C_{16}H_{14}ClO^+$ [M-SCF_3]$^+$ *m/z*: 257.0728, found: 257.0725.



4h

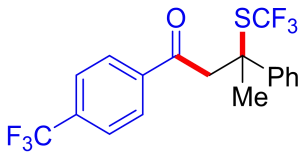
1-(4-Bromophenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4h**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 65.8 mg product as a yellow oil in 82% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 – 7.68 (m, 2H), 7.58 – 7.54 (m, 2H), 7.52 – 7.47 (m, 2H), 7.34 – 7.29 (m, 2H), 7.27 – 7.23 (m, 1H), 4.14 (d, J = 17.3 Hz, 1H), 3.74 (d, J = 17.3 Hz, 1H), 2.22 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.3, 141.3, 135.6, 131.9, 130.1 (q, J = 309.6 Hz), 129.4, 128.6, 128.5, 127.8, 126.3, 54.6, 49.7, 27.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.00.

HRMS (APCI) calcd for $C_{16}H_{14}BrO^+$ [M-SCF_3]$^+$ *m/z*: 301.0223, found: 301.0220.



4i

3-Phenyl-1-(4-(trifluoromethyl)phenyl)-3-((trifluoromethyl)thio)butan-1-one (**4i**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 51.6 mg product as a colorless oil in 66% yield.

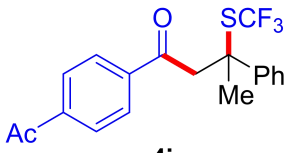
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 (d, J = 8.1 Hz, 2H), 7.69 (d, J = 8.2 Hz, 2H), 7.52 – 7.48 (m, 2H),

7.35 – 7.29 (m, 2H), 7.28 – 7.24 (m, 1H), 4.22 (d, J = 17.4 Hz, 1H), 3.81 (d, J = 17.4 Hz, 1H), 2.24 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.5, 141.1, 139.5, 134.6 (q, J = 32.8 Hz), 130.1 (q, J = 309.6 Hz), 128.6, 128.2, 128.0, 126.3, 125.7 (q, J = 3.8 Hz), 123.5 (q, J = 272.7 Hz), 54.5, 50.1, 27.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.05, -63.20.

HRMS (APCI) calcd for $C_{17}H_{14}F_3O^+$ $[M\text{-}SCF_3]^+$ *m/z*: 291.0991, found: 291.0990.



4j

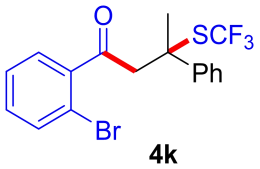
1-(4-Acetylphenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4j**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 2/1) to give 18.6 mg product as a colorless oil in 25% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 – 7.96 (m, 2H), 7.96 – 7.88 (m, 2H), 7.53 – 7.46 (m, 2H), 7.35 – 7.30 (m, 2H), 7.28 – 7.24 (m, 1H), 4.22 (d, J = 17.4 Hz, 1H), 3.81 (d, J = 17.3 Hz, 1H), 2.63 (s, 3H), 2.24 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 197.3, 194.9, 141.2, 140.3, 140.0, 130.1 (q, J = 309.6 Hz), 128.5, 128.1, 127.9, 126.3, 54.6, 50.2, 27.0, 26.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.04.

HRMS (APCI) calcd for $C_{19}H_{16}F_3O_2S^-$ $[M\text{-}H]^-$ *m/z*: 365.0829, found: 365.0821.



4k

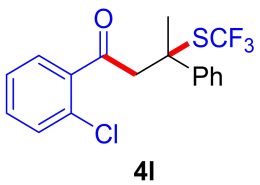
1-(2-Bromophenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4k**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 5/1) to give 61.7 mg product as a yellow oil in 77% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.55 – 7.49 (m, 3H), 7.33 – 7.28 (m, 2H), 7.27 – 7.20 (m, 3H), 7.05 – 7.01 (m, 1H), 4.11 (d, J = 16.9 Hz, 1H), 3.76 (d, J = 16.9 Hz, 1H), 2.24 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 199.6, 141.8, 140.9, 133.5, 131.6, 130.1 (q, J = 309.6 Hz), 128.4, 128.2, 127.9, 127.4, 126.5, 118.3, 54.5, 53.9, 26.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.15.

HRMS (APCI) calcd for $C_{16}H_{14}BrO^+$ $[M\text{-}SCF_3]^+$ *m/z*: 301.0223, found: 301.0221.



4l

1-(2-Chlorophenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4l**) was synthesized according to

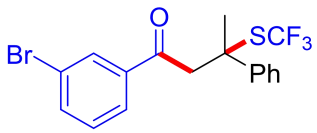
General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 5/1) to give 69.3 mg product as a colorless oil in 97% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.50 (d, *J* = 7.9 Hz, 2H), 7.37 – 7.27 (m, 4H), 7.26 – 7.22 (m, 1H), 7.20 (td, *J* = 7.4, 1.4 Hz, 1H), 7.10 (dd, *J* = 7.7, 1.4 Hz, 1H), 4.15 (d, *J* = 16.8 Hz, 1H), 3.76 (d, *J* = 16.8 Hz, 1H), 2.23 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.9, 141.0, 139.6, 131.7, 130.4, 130.3, 130.1 (q, *J* = 309.6 Hz), 128.6, 128.4, 127.9, 126.9, 126.5, 54.6, 54.1, 26.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.17.

HRMS (APCI) calcd for $C_{17}H_{13}ClF_3OS^-$ $[M-H]^-$ *m/z*: 357.0333, found: 357.0328.



4m

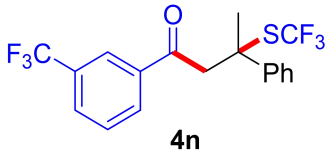
1-(3-Bromophenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4m**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 5/1) to give 62.7 mg product as a colorless oil in 78% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95 (t, *J* = 1.8 Hz, 1H), 7.80 – 7.75 (m, 1H), 7.69 – 7.65 (m, 1H), 7.52 – 7.46 (m, 2H), 7.35 – 7.28 (m, 3H), 7.25 (tt, *J* = 6.4, 1.1 Hz, 1H), 4.15 (d, *J* = 17.4 Hz, 1H), 3.74 (d, *J* = 17.4 Hz, 1H), 2.22 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.0, 141.2, 138.6, 136.2, 131.0, 130.2, 130.1 (q, *J* = 309.5 Hz), 128.5, 127.9, 126.4, 126.3, 123.0, 54.5, 49.9, 27.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.01.

HRMS (APCI) calcd for $C_{16}H_{14}BrO^+$ $[M-SCF_3]^+$ *m/z*: 301.0223, found: 301.0220.



4n

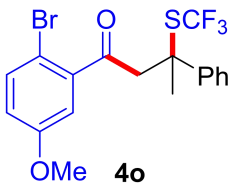
3-Phenyl-1-(3-(trifluoromethyl)phenyl)-3-((trifluoromethyl)thio)butan-1-one (**4n**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 5/1) to give 65.7 mg product as a colorless oil in 84% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.07 (s, 1H), 8.03 (d, *J* = 7.9 Hz, 1H), 7.79 (d, *J* = 7.8 H z, 1H), 7.57 (t, *J* = 7.8 Hz, 1H), 7.54 – 7.49 (m, 2H), 7.35 – 7.29 (m, 2H), 7.25 (d, *J* = 7.3 Hz, 1H), 4.20 (d, *J* = 17.3 Hz, 1H), 3.79 (d, *J* = 17.2 Hz, 1H), 2.24 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.1, 141.2, 137.4, 131.3 (q, *J* = 32.8 Hz), 131.0, 130.1 (q, *J* = 309.5 Hz), 129.7 (q, *J* = 3.5 Hz), 129.4, 128.5, 127.9, 126.3, 124.8 (q, *J* = 3.4 Hz), 123.5 (q, *J* = 272.8 Hz), 54.5, 50.0, 26.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.03, -62.88.

HRMS (APCI) calcd for $C_{17}H_{14}F_3O^+$ $[M-SCF_3]^+$ *m/z*: 291.0991, found: 291.0990.



4o

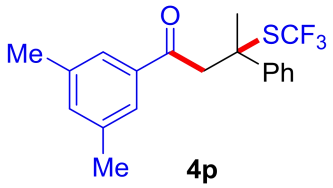
1-(2-Bromo-5-methoxyphenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4o**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to give 64.2 mg product as a colorless oil in 77% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.45 – 7.41 (m, 2H), 7.31 (d, *J* = 8.8 Hz, 1H), 7.25 – 7.21 (m, 2H), 7.19 – 7.14 (m, 1H), 6.70 (dd, *J* = 8.8, 3.0 Hz, 1H), 6.38 (d, *J* = 3.0 Hz, 1H), 4.03 (d, *J* = 16.8 Hz, 1H), 3.67 (d, *J* = 16.7 Hz, 1H), 3.62 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 199.6, 158.7, 142.6, 1401.0, 134.2, 130.0 (q, *J* = 309.6 Hz), 128.4, 127.9, 126.6, 117.8, 113.5, 108.3, 55.5, 54.5, 53.9, 26.9

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.15.

HRMS (APCI) calcd for $C_{17}H_{16}BrO_2^+$ $[M\text{-}SCF_3]^+$ *m/z*: 331.0328, found: 331.0326.



4p

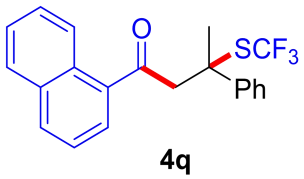
1-(3,5-Dimethylphenyl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4p**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 45.2 mg product as a colorless oil in 64% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 – 7.49 (m, 2H), 7.46 (s, 2H), 7.33 – 7.29 (m, 2H), 7.26 – 7.22 (m, 1H), 7.18 (s, 1H), 4.17 (d, *J* = 17.4 Hz, 1H), 3.76 (d, *J* = 17.4 Hz, 1H), 2.34 (s, 6H), 2.22 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.6, 141.6, 138.3, 137.0, 135.0, 130.2 (q, *J* = 309.5 Hz), 128.4, 127.7, 126.4, 125.7, 54.8, 49.9, 27.0, 21.2.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.03.

HRMS (APCI) calcd for $C_{18}H_{19}O^+$ $[M\text{-}SCF_3]^+$ *m/z*: 251.1430, found: 251.1433.



4q

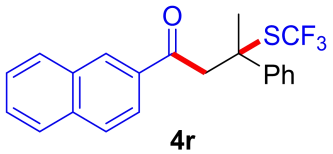
1-(naphthalen-1-yl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4q**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 5/1) to give 61.3 mg product as a colorless oil in 82% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.16 – 8.10 (m, 1H), 7.92 (d, *J* = 8.2 Hz, 1H), 7.83 – 7.79 (m, 1H), 7.71 (dd, *J* = 7.2, 1.0 Hz, 1H), 7.55 – 7.50 (m, 2H), 7.49 – 7.44 (m, 2H), 7.42 (dd, *J* = 8.1, 7.3 Hz, 1H), 7.31 – 7.24 (m, 2H), 7.24 – 7.19 (m, 1H), 4.21 (d, *J* = 16.6 Hz, 1H), 3.84 (d, *J* = 16.6 Hz, 1H), 2.29 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 199.9, 141.3, 136.4, 133.9, 132.7, 130.2 (q, J = 309.6 Hz), 129.7, 128.4, 128.3, 127.8, 127.8, 126.9, 126.5, 125.4, 124.2, 55.0, 53.4, 27.2.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.00.

HRMS (APCI) calcd for $C_{20}H_{17}O^+$ [M-SCF_3]$^+$ *m/z*: 273.1274, found: 273.1270.



4r

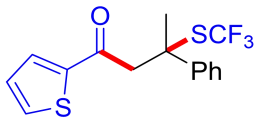
1-(naphthalen-2-yl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4r**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 52.2 mg product as a colorless oil in 70% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.30 (d, J = 0.7 Hz, 1H), 7.86 (d, J = 8.1 Hz, 1H), 7.82 (dd, J = 8.6, 1.8 Hz, 1H), 7.79 – 7.74 (m, 2H), 7.54 – 7.50 (m, 1H), 7.50 – 7.44 (m, 3H), 7.27 – 7.22 (m, 2H), 7.20 – 7.13 (m, 1H), 4.25 (d, J = 17.2 Hz, 1H), 3.84 (d, J = 17.2 Hz, 1H), 2.20 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.3, 141.6, 135.6, 134.3, 133.3, 132.4, 130.3 (q, J = 309.6 Hz), 129.7, 129.6, 128.7, 128.6, 128.5, 127.77, 127.75, 126.9, 126.4, 123.5, 54.9, 49.9, 27.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -35.97.

HRMS (APCI) calcd for $C_{21}H_{16}F_3OS^-$ [M-H]$^-$ *m/z*: 373.0879, found: 373.0875.



4s

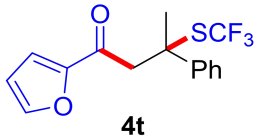
3-Phenyl-1-(thiophen-2-yl)-3-((trifluoromethyl)thio)butan-1-one (**4s**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 5/1) to give 30.0 mg product as a colorless oil in 45% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (d, J = 3.7 Hz, 1H), 7.54 (d, J = 4.9 Hz, 1H), 7.46 (d, J = 7.9 Hz, 2H), 7.25 (t, J = 7.7 Hz, 2H), 7.18 (t, J = 7.3 Hz, 1H), 7.02 (t, J = 4.4 Hz, 1H), 3.97 (d, J = 16.6 Hz, 1H), 3.64 (d, J = 16.6 Hz, 1H), 2.16 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 188.2, 144.4, 141.4, 134.2, 132.1, 130.1 (q, J = 309.5 Hz), 128.5, 128.1, 127.9, 126.4, 54.6, 50.5, 27.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.10.

HRMS (APCI) calcd for $C_{15}H_{12}F_3OS_2^-$ [M-H]$^-$ *m/z*: 329.0287, found: 329.0285.



4t

1-(Furan-2-yl)-3-phenyl-3-((trifluoromethyl)thio)butan-1-one (**4t**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 3/1) to give 14.2 mg product as a colorless oil in 23% yield.

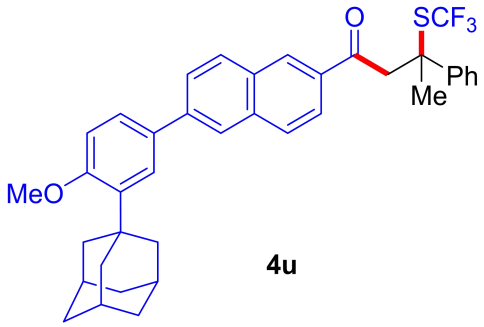
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 – 7.50 (m, 3H), 7.36 – 7.29 (m, 2H), 7.26 – 7.22 (m, 1H), 7.10 (dd,

J = 3.6, 0.6 Hz, 1H), 6.50 (dd, *J* = 3.6, 1.7 Hz, 1H), 3.97 (d, *J* = 16.4 Hz, 1H), 3.61 (d, *J* = 16.4 Hz, 1H), 2.21 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 184.6, 152.7, 146.5, 141.4, 133.2, 131.2, 129.1, 128.4, 127.8, 127.1, 126.5, 117.3, 112.5, 54.6, 49.5, 27.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -36.18.

HRMS (APCI) calcd for $C_{14}H_{13}O_2^+$ [M-SCF_3]$^+$ *m/z*: 213.0910, found: 213.0909.



4u

1-(6-(3-((3r,5r,7r)-Adamantan-1-yl)-4-methoxyphenyl)naphthalen-2-yl)-3-phenyl-3-((trifluoro methyl)thio)butan-1-one (**4u**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 64.7 mg product as a pale yellow solid in 53% yield.

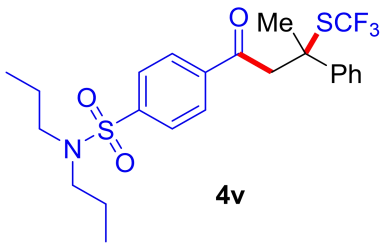
Mp =148-151 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.99 (s, 1H), 7.97 (d, *J* = 8.6 Hz, 1H), 7.91 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.88 (d, *J* = 8.7 Hz, 1H), 7.81 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.60 (d, *J* = 2.3 Hz, 1H), 7.59 – 7.55 (m, 2H), 7.54 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.35 – 7.30 (m, 2H), 7.27 – 7.22 (m, 1H), 6.99 (d, *J* = 8.5 Hz, 1H), 4.34 (d, *J* = 17.1 Hz, 1H), 3.92 (d, *J* = 17.1 Hz, 1H), 3.90 (s, 3H), 2.28 (s, 3H), 2.18 (d, *J* = 2.6 Hz, 6H), 2.10 (s, 3H), 1.80 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.1, 159.0, 141.9, 141.6, 139.1, 136.1, 133.8, 132.3, 131.1, 130.3 (q, *J* = 309.6 Hz), 130.0, 129.5, 128.6, 128.5, 127.8, 126.7, 126.4, 125.9, 125.7, 124.7, 123.9, 112.1, 55.2, 54.9, 49.8, 40.6, 37.2, 37.1, 29.1, 27.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -35.97.

HRMS (APCI) calcd for $C_{38}H_{38}F_3O_2S^+$ [M+H]$^+$ *m/z*: 615.2539, found: 615.2523.



4v

4-(3-Phenyl-3-((trifluoromethyl)thio)butanoyl)-N,N-dipropylbenzenesulfonamide (**4v**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 30/1) to give 52.2 mg product as a colorless oil in 54% yield.

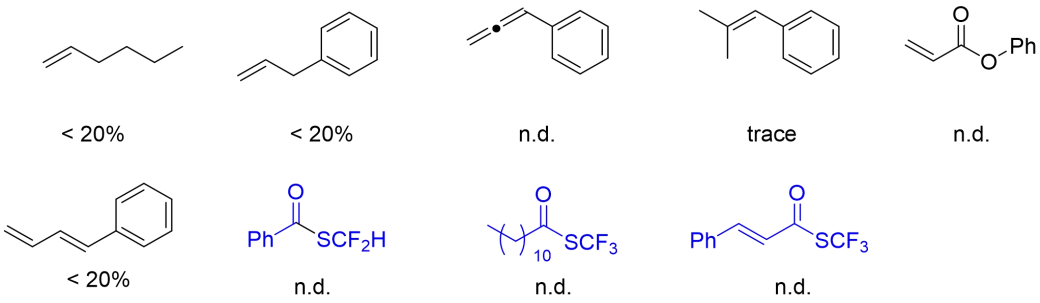
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 – 7.92 (m, 2H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.51 (dt, *J* = 3.0, 1.8 Hz, 2H), 7.35 – 7.29 (m, 2H), 7.29 – 7.23 (m, 1H), 4.21 (d, *J* = 17.3 Hz, 1H), 3.80 (d, *J* = 17.3 Hz, 1H), 3.12 – 3.04 (m, 4H), 2.23 (s, 3H), 1.59 – 1.50 (m, 4H), 0.87 (t, *J* = 7.4 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 194.4, 144.4, 141.1, 139.4, 130.1 (q, J = 309.7 Hz), 128.5, 128.4, 127.9, 127.3, 126.3, 54.5, 50.1, 49.9, 26.9, 21.9, 11.1.

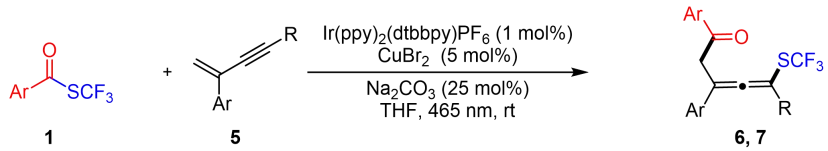
^{19}F NMR (565 MHz, $CDCl_3$) δ -36.02.

HRMS (APCI) calcd for $C_{23}H_{29}F_3NO_3S_2^+$ $[M+H]^+$ *m/z*: 488.1535, found: 488.1534.

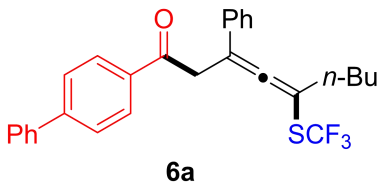
Failed examples



General procedure for the preparation of compounds 6/7



An oven-dried 10 mL vial was charged with a stir bar, *S*-trilfluoromethyl thioesters **1** (0.3 mmol, 1.5 equiv.), $Ir(ppy)_2(dtbppy)PF_6$ (1.8 mg, 0.002 mmol, 1 mol%), and sodium carbonates (5.3 mg, 0.05 mmol, 25 mol%) was added. The $CuBr_2$ (3.7 mg, 0.01 mmol, 5 mol%) was added into the vial in the glove box. Then, the septum capped vial was transferred out of the glovebox. 1,3-Enynes **5** (0.2 mmol, 1 equiv.) and THF (2 mL) were added under the argon atmosphere. After degassed by three cycles of freeze-pump thaw, the mixture was placed on a photoreactor equipped with 6W LED lamps and stirred for 12 hours. The solvent was evaporated and the residue was purified by column chromatography to afford the desired products **6/7**.

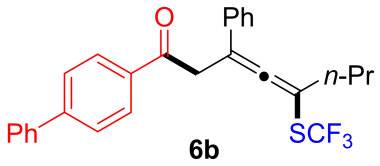


6a

1-([1,1'-biphenyl]-4-yl)-3-phenyl-5-((trifluoromethyl)thio)nona-3,4-dien-1-one (**6a**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 50.0 mg product as a brown oil in 54% yield.

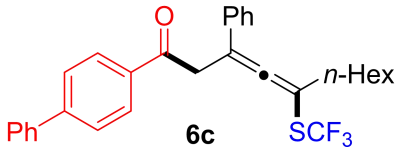
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, J = 8.4 Hz, 2H), 7.61 (d, J = 8.4 Hz, 2H), 7.57 – 7.51 (m, 2H), 7.39 (t, J = 7.6 Hz, 2H), 7.35 – 7.29 (m, 3H), 7.27 (t, J = 7.7 Hz, 2H), 7.22 – 7.17 (m, 1H), 4.11 – 4.04 (m, 2H), 2.32 – 2.16 (m, 2H), 1.36 – 1.28 (m, 2H), 1.18 (dt, J = 22.4, 7.3 Hz, 2H), 0.74 (t, J = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 208.9, 195.7, 146.1, 139.8, 134.9, 134.0, 130.1 (dd, *J* = 618.5, 309.3 Hz), 129.2, 129.0, 128.7, 128.3, 128.0, 127.3, 127.2, 126.4, 102.8, 97.3, 40.5, 35.3, 29.6, 22.0, 13.7.
^{19}F NMR (565 MHz, $CDCl_3$) δ -40.35.
HRMS (ESI) calcd for $C_{28}H_{26}F_3OS^+$ $[M+H]^+$ *m/z*: 467.1651, found: 467.1650.



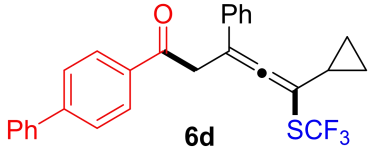
6b

(S)-1-([1,1'-biphenyl]-4-yl)-3-phenyl-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**6b**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 62.0 mg product as a brown oil in 67% yield.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.64 – 7.60 (m, 2H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.42 – 7.37 (m, 3H), 7.35 (t, *J* = 7.7 Hz, 2H), 7.30 – 7.25 (m, 1H), 4.16 (s, 2H), 2.36 – 2.24 (m, 2H), 1.52 – 1.44 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 209.0, 195.7, 146.1, 139.8, 134.9, 134.0, 130.1 (q, *J* = 309.4 Hz), 129.2, 129.0, 128.7, 128.3, 128.0, 127.3, 127.3, 126.4, 102.8, 97.0, 40.6, 37.6, 20.9, 13.5.
^{19}F NMR (565 MHz, $CDCl_3$) δ -40.34.
HRMS (ESI) calcd for $C_{27}H_{24}F_3OS^+$ $[M+H]^+$ *m/z*: 453.1494, found: 453.1492.



6c

1-([1,1'-biphenyl]-4-yl)-3-phenyl-5-((trifluoromethyl)thio)undeca-3,4-dien-1-one (**6c**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 50.4 mg product as a brown oil in 51% yield.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.07 – 8.02 (m, 2H), 7.71 – 7.67 (m, 2H), 7.64 – 7.61 (m, 2H), 7.49 – 7.44 (m, 2H), 7.42 – 7.37 (m, 3H), 7.37 – 7.32 (m, 2H), 7.30 – 7.25 (m, 1H), 4.22 – 4.09 (m, 2H), 2.37 – 2.23 (m, 2H), 1.45 – 1.36 (m, 2H), 1.25 – 1.14 (m, 6H), 0.80 (t, *J* = 6.9 Hz, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 209.0, 195.7, 146.1, 139.7, 134.9, 134.0, 130.1 (q, *J* = 309.6 Hz), 129.2, 129.0, 128.7, 128.3, 128.0, 127.3, 127.2, 126.4, 102.8, 97.3, 40.6, 35.6, 31.5, 28.6, 27.5, 22.5, 14.0.
^{19}F NMR (565 MHz, $CDCl_3$) δ -40.34.
HRMS (ESI) calcd for $C_{30}H_{30}F_3OS^+$ $[M+H]^+$ *m/z*: 495.1964, found: 495.1961.



6d

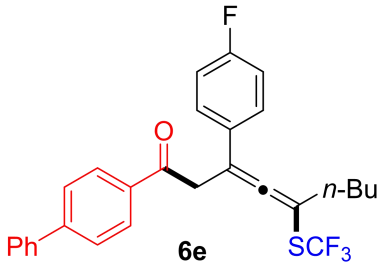
1-([1,1'-biphenyl]-4-yl)-5-cyclopropyl-3-phenyl-5-((trifluoromethyl)thio)penta-3,4-dien-1-one (**6d**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 48.7 mg product as a brown oil in 54% yield.
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.65 – 7.61 (m, 2H),

7.47 (t, J = 7.6 Hz, 2H), 7.40 (t, J = 7.4 Hz, 1H), 7.36 – 7.32 (m, 4H), 7.30 – 7.25 (m, 1H), 4.15 (d, J = 16.0 Hz, 1H), 4.11 (d, J = 15.9 Hz, 1H), 1.57 – 1.46 (m, 1H), 0.83 – 0.70 (m, 2H), 0.66 – 0.60 (m, 1H), 0.54 – 0.47 (m, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 207.9, 195.7, 146.1, 139.7, 134.8, 133.8, 123.0 (d, J = 309.9 Hz), 129.2, 129.0, 128.8, 128.3, 128.1, 127.3, 127.3, 126.3, 104.1, 100.6, 40.6, 15.4, 8.1, 7.9.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.25.

HRMS (ESI) calcd for $C_{27}H_{22}F_3OS^+$ $[M+H]^+$ *m/z*: 451.1338, found: 451.1335.



6e

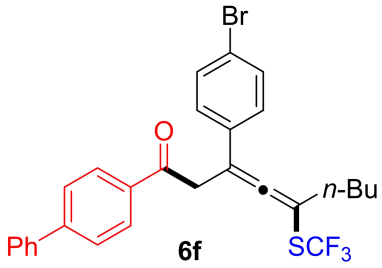
1-([1,1'-biphenyl]-4-yl)-3-(4-fluorophenyl)-5-((trifluoromethyl)thio)nona-3,4-dien-1-one (**6e**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 50.0 mg product as a brown oil in 52% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.07 – 8.02 (m, 2H), 7.72 – 7.68 (m, 2H), 7.65 – 7.60 (m, 2H), 7.50 – 7.46 (m, 2H), 7.43 – 7.39 (m, 1H), 7.37 – 7.32 (m, 2H), 7.06 – 7.01 (m, 2H), 4.18 – 4.08 (m, 2H), 2.37 – 2.26 (m, 2H), 1.43 – 1.36 (m, 2H), 1.29 – 1.23 (m, 2H), 0.83 (t, J = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 208.7, 195.5, 162.4 (d, J = 248.4 Hz), 146.2, 139.7, 134.8, 130.1 (q, J = 309.4 Hz), 130.0 (d, J = 3.2 Hz), 129.2, 129.0, 128.4, 128.1 (d, J = 8.2 Hz), 127.3, 127.2, 115.7 (d, J = 21.8 Hz), 102.1, 97.3, 40.7, 35.3, 29.6, 22.0, 13.7.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.40 (s, 3F), -113.40 – -113.84 (m, 1F).

HRMS (ESI) calcd for $C_{28}H_{25}F_4OS^+$ $[M+H]^+$ *m/z*: 485.1557, found: 485.1559.



6f

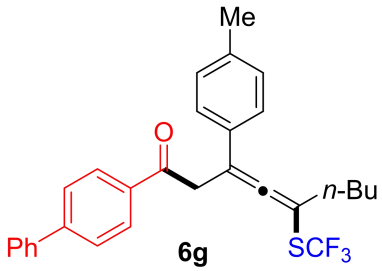
1-([1,1'-biphenyl]-4-yl)-3-(4-bromophenyl)-5-((trifluoromethyl)thio)nona-3,4-dien-1-one (**6f**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 43.4 mg product as a brown oil in 40% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.06 – 8.00 (m, 2H), 7.73 – 7.67 (m, 2H), 7.65 – 7.61 (m, 2H), 7.49 – 7.44 (m, 4H), 7.44 – 7.39 (m, 1H), 7.25 – 7.22 (m, 3H), 4.13 (s, 2H), 2.39 – 2.23 (m, 2H), 1.45 – 1.36 (m, 2H), 1.29 – 1.23 (m, 2H), 0.83 (t, J = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 208.8, 195.4, 146.3, 139.7, 134.8, 133.1, 131.9, 130.1 (d, J = 309.5 Hz), 129.2, 129.0, 128.4, 128.0, 127.4, 127.3, 122.1, 102.2, 97.7, 40.5, 35.3, 29.7, 22.1, 13.7.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.39.

HRMS (ESI) calcd for $C_{28}H_{25}F_3OSBr^+$ $[M+H]^+$ *m/z*: 545.0756, found: 545.0755.



6g

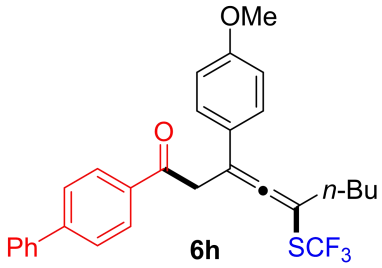
1-([1,1'-biphenyl]-4-yl)-3-(p-tolyl)-5-((trifluoromethyl)thio)nona-3,4-dien-1-one (**6g**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 63.7 mg product as a brown oil in 66% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 2H), 7.70 – 7.66 (m, 2H), 7.63 – 7.60 (m, 2H), 7.49 – 7.43 (m, 2H), 7.41 – 7.38 (m, 1H), 7.28 (d, J = 8.2 Hz, 2H), 7.16 (d, J = 8.0 Hz, 2H), 4.17 – 4.06 (m, 2H), 2.38 – 2.23 (m, 5H), 1.43 – 1.35 (m, 2H), 1.28 – 1.21 (m, 2H), 0.82 (t, J = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 208.9, 195.9, 146.1, 139.8, 138.1, 135.0, 131.0, 130.2 (q, J = 309.4 Hz), 129.5, 129.3, 129.0, 128.4, 127.3, 127.3, 126.3, 102.9, 97.1, 40.7, 35.4, 29.7, 22.1, 21.2, 13.8.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.35.

HRMS (ESI) calcd for $C_{29}H_{28}F_3OS^+$ $[M+H]^+$ *m/z*: 481.1807, found: 481.1805.



6h

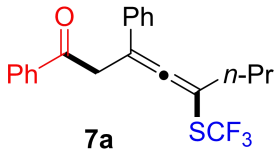
1-([1,1'-biphenyl]-4-yl)-3-(4-methoxyphenyl)-5-((trifluoromethyl)thio)nona-3,4-dien-1-one (**6h**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 2/1) to give 42.6 mg product as a brown oil in 43% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (d, J = 8.4 Hz, 2H), 7.71 – 7.66 (m, 2H), 7.65 – 7.60 (m, 2H), 7.49 – 7.45 (m, 2H), 7.42 – 7.38 (m, 1H), 7.33 – 7.29 (m, 2H), 6.92 – 6.87 (m, 2H), 4.12 (s, 2H), 3.80 (s, 3H), 2.37 – 2.23 (m, 2H), 1.42 – 1.35 (m, 2H), 1.28 – 1.22 (m, 2H), 0.82 (t, J = 7.3 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 208.7, 195.9, 159.5, 146.0, 139.8, 134.9, 130.2 (q, J = 309.3 Hz), 129.2, 129.0, 128.3, 127.7, 127.3, 127.2, 126.0, 114.2, 102.6, 97.0, 55.3, 40.7, 35.4, 29.7, 22.0, 13.7.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.37.

HRMS (ESI) calcd for $C_{29}H_{28}F_3O_2S^+$ $[M+H]^+$ *m/z*: 497.1757, found: 497.1755.



7a

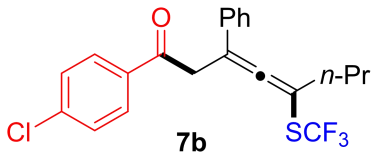
1,3-diphenyl-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**7a**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 38.3 mg product as a brown oil in 51% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 – 7.95 (m, 2H), 7.60 – 7.55 (m, 1H), 7.49 – 7.45 (m, 2H), 7.38 – 7.31 (m, 4H), 7.28 – 7.25 (m, 1H), 4.13 (s, 2H), 2.34 – 2.18 (m, 2H), 1.46 (hept, J = 7.4 Hz, 2H), 0.86 (t, J = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 209.0, 196.1, 136.2, 134.0, 133.4, 130.1 (q, J = 309.4 Hz), 128.7, 128.7, 128.6, 128.0, 126.3, 102.7, 97.0, 40.5, 37.6, 20.9, 13.4.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.41.

HRMS (ESI) calcd for $C_{21}H_{20}F_3OS^+$ $[M+H]^+$ *m/z*: 377.1181, found: 377.1181.



7b

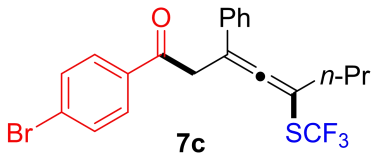
1-(4-chlorophenyl)-3-phenyl-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**7b**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 49.1 mg product as a brown oil in 60% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 – 7.87 (m, 2H), 7.47 – 7.41 (m, 2H), 7.35 (d, J = 4.3 Hz, 4H), 7.29 – 7.25 (m, 1H), 4.09 (s, 2H), 2.37 – 2.23 (m, 2H), 1.47 (hept, J = 7.4 Hz, 2H), 0.87 (t, J = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 208.9, 195.0, 139.9, 134.5, 133.8, 130.0 (d, J = 309.5 Hz), 130.0, 129.0, 128.8, 128.1, 126.3, 102.6, 97.2, 40.5, 37.5, 20.9, 13.4.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.37.

HRMS (ESI) calcd for $C_{21}H_{19}ClF_3OS^+$ $[M+H]^+$ *m/z*: 411.0792, found: 411.0793.



7c

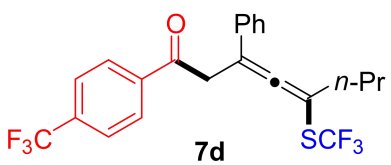
1-(4-bromophenyl)-3-phenyl-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**7c**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 56.2 mg product as a brown oil in 62% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, J = 8.5 Hz, 2H), 7.61 (d, J = 8.5 Hz, 2H), 7.38 – 7.32 (m, 4H), 7.30 – 7.25 (m, 1H), 4.09 (s, 2H), 2.37 – 2.22 (m, 2H), 1.47 (hept, J = 7.4 Hz, 2H), 0.87 (t, J = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 208.9, 195.2, 134.9, 133.8, 132.0, 130.1, 130.0 (q, J = 309.5 Hz), 128.8, 128.6, 128.1, 126.3, 102.6, 97.2, 40.5, 37.5, 20.9, 13.4.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.36.

HRMS (ESI) calcd for $C_{21}H_{19}BrF_3OS^+$ $[M+H]^+$ *m/z*: 455.0287, found: 455.0287.



7d

3-phenyl-1-(4-(trifluoromethyl)phenyl)-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**7d**) was synthesized according to General Procedure and purified by flash silica column chromatography

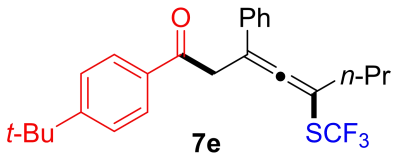
(petroleum ether/dichloromethane = 4/1) to give 60.6 mg product as a brown oil in 68% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.1 Hz, 2H), 7.74 (d, *J* = 8.2 Hz, 2H), 7.38 – 7.33 (m, 4H), 7.31 – 7.26 (m, 1H), 4.15 (s, 2H), 2.34 – 2.20 (m, 2H), 1.45 (hept, *J* = 7.3 Hz, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 208.8, 195.3, 138.8, 134.7 (q, *J* = 32.7 Hz), 133.7, 123.0 (q, *J* = 309.7 Hz), 128.9, 128.8, 128.2, 126.3, 125.8 (q, *J* = 3.7 Hz), 123.5 (q, *J* = 272.7 Hz), 102.4, 97.5, 40.8, 37.5, 20.9, 13.4.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.41, -63.19.

HRMS (ESI) calcd for $C_{22}H_{19}F_6OS^+$ $[M+H]^+$ *m/z*: 445.1055, found: 445.1054.



7e

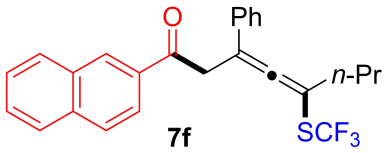
1-(4-(tert-butyl)phenyl)-3-phenyl-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**7e**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 41.1 mg product as a brown oil in 48% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 – 7.88 (m, 2H), 7.51 – 7.46 (m, 2H), 7.38 – 7.31 (m, 4H), 7.28 – 7.24 (m, 1H), 4.12 (d, *J* = 16.0 Hz, 1H), 4.09 (d, *J* = 16.0 Hz, 1H), 2.34 – 2.20 (m, 2H), 1.45 (hept, *J* = 7.4 Hz, 2H), 1.34 (s, 9H), 0.85 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 209.1, 195.8, 157.2, 134.0, 133.7, 130.1 (q, *J* = 309.3 Hz), 128.7, 128.6, 127.9, 126.4, 125.6, 102.9, 96.9, 40.5, 37.6, 35.1, 31.0, 20.9, 13.5.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.43.

HRMS (ESI) calcd for $C_{25}H_{28}F_3OS^+$ $[M+H]^+$ *m/z*: 433.1807, found: 433.1806.



7f

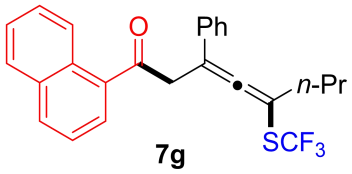
1-(naphthalen-2-yl)-3-phenyl-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**7f**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 39.6 mg product as a brown oil in 46% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.50 (s, 1H), 8.02 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.95 (d, *J* = 8.1 Hz, 1H), 7.90 (d, *J* = 8.7 Hz, 1H), 7.88 (d, *J* = 8.2 Hz, 1H), 7.64 – 7.58 (m, 1H), 7.58 – 7.53 (m, 1H), 7.42 – 7.38 (m, 2H), 7.37 – 7.33 (m, 2H), 7.29 – 7.25 (m, 1H), 4.26 (s, 2H), 2.34 – 2.17 (m, 2H), 1.43 (hept, *J* = 7.4 Hz, 2H), 0.79 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 209.1, 196.1, 135.7, 134.0, 133.6, 132.5, 130.4, 130.1 (q, *J* = 309.4 Hz), 129.6, 128.72, 128.66, 128.6, 128.0, 127.8, 126.9, 126.4, 124.1, 102.9, 97.0, 40.6, 37.6, 20.9, 13.4.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.39.

HRMS (ESI) calcd for $C_{25}H_{22}F_3OS^+$ $[M+H]^+$ *m/z*: 427.1338, found: 427.1336.



7g

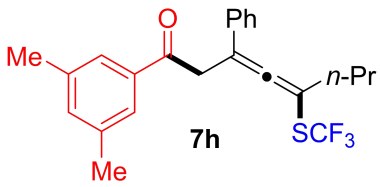
1-(naphthalen-1-yl)-3-phenyl-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**7g**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 40.7 mg product as a brown oil in 48% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.54 (d, *J* = 8.5 Hz, 1H), 8.00 (d, *J* = 8.2 Hz, 1H), 7.93 (dd, *J* = 7.2, 1.0 Hz, 1H), 7.87 (dd, *J* = 8.3, 1.0 Hz, 1H), 7.58 – 7.46 (m, 3H), 7.41 – 7.36 (m, 2H), 7.36 – 7.30 (m, 2H), 7.27 – 7.24 (m, 1H), 4.25 (d, *J* = 16.1 Hz, 1H), 4.22 (d, *J* = 16.1 Hz, 1H), 2.20 (ddd, *J* = 15.4, 9.2, 6.1 Hz, 1H), 2.08 (ddd, *J* = 15.3, 9.1, 6.0 Hz, 1H), 1.42 – 1.28 (m, 2H), 0.79 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 209.1, 200.0, 134.7, 134.0, 134.0, 133.1, 130.4, 130.1 (d, *J* = 309.3 Hz), 128.7, 128.4, 128.2, 128.1, 128.0, 126.5, 126.4, 125.7, 124.3, 102.9, 96.9, 43.7, 37.6, 20.8, 13.4.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.45.

HRMS (ESI) calcd for $C_{25}H_{22}F_3OS^+$ $[M+H]^+$ *m/z*: 427.1338, found: 427.1340.



7h

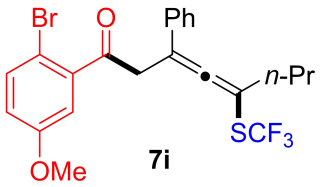
1-(3,5-dimethylphenyl)-3-phenyl-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**7h**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 4/1) to give 38.9 mg product as a brown oil in 48% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (s, 2H), 7.39 – 7.29 (m, 4H), 7.28 – 7.23 (m, 1H), 7.22 (s, 1H), 4.10 (s, 2H), 2.37 (s, 6H), 2.35 – 2.22 (m, 2H), 1.47 (hept, *J* = 7.4 Hz, 2H), 0.87 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 209.1, 196.6, 138.3, 136.4, 135.0, 134.1, 130.1 (q, *J* = 309.3 Hz), 128.7, 127.9, 126.4, 126.3, 102.9, 96.8, 40.7, 37.6, 21.2, 20.9, 13.5.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.44.

HRMS (ESI) calcd for $C_{23}H_{24}F_3OS^+$ $[M+H]^+$ *m/z*: 405.1494, found: 405.1493.



7i

1-(2-bromo-5-methoxyphenyl)-3-phenyl-5-((trifluoromethyl)thio)octa-3,4-dien-1-one (**7i**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 2/1) to give 48.9 mg product as a brown oil in 50% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.8 Hz, 1H), 7.37 – 7.32 (m, 4H), 7.28 – 7.24 (m, 1H), 6.84 (dd, *J* = 8.8, 3.0 Hz, 1H), 6.76 (d, *J* = 3.0 Hz, 1H), 4.16 (d, *J* = 16.5 Hz, 1H), 4.12 (d, *J* = 16.5 Hz, 1H), 3.71 (s, 3H), 2.36 – 2.26 (m, 2H), 1.53 – 1.46 (m, 2H), 0.90 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 209.3, 200.0, 158.8, 141.4, 134.4, 133.8, 130.1 (q, *J* = 309.5 Hz), 128.7,

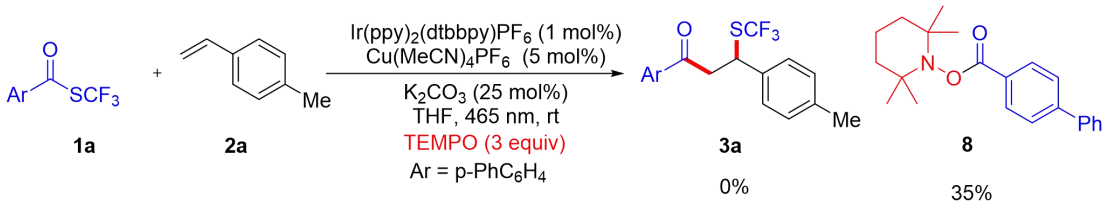
128.0, 126.4, 118.0, 114.2, 108.9, 101.6, 96.5, 55.5, 44.3, 37.6, 20.9, 13.5.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.50.

HRMS (ESI) calcd for $C_{22}H_{21}BrF_3O_2S^+$ $[M+H]^+$ *m/z*: 485.0392, found: 485.0391.

V. Mechanistic investigations

Radical inhibition experiments



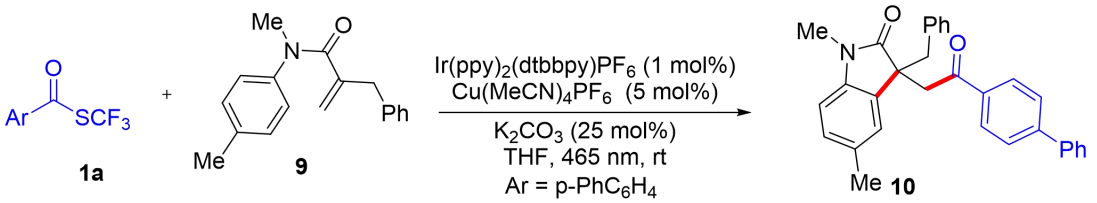
To an oven-dried 10 mL vial charged with a stir bar, *S*-trilfluoromethyl thioester **1a** (0.3 mmol, 1.5 equiv.), $Ir(ppy)_2(dtbppy)PF_6$ (1.8 mg, 0.002 mmol, 1 mol%), potassium carbonate (6.9 mg, 0.05 mmol, 25 mol%), and TEMPO (93.7 mg, 0.6 mmol, 3 equiv.) were added. The $Cu(MeCN)_4PF_6$ (3.7 mg, 0.01 mmol, 5 mol%) was added into the vial in the glove box. Then, the septum capped vial was transferred out of the glovebox and alkene **2a** (23.6 mg, 0.2 mmol, 1 equiv.) and THF (2 mL) were added under the argon atmosphere. After degassed by three cycles of freeze-pump thaw, the mixture was placed on a photoreactor equipped with 6W LED lamps and stirred for 12 hours. The solvent was evaporated and the residue was purified by column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to afford the **8 (**35.7 mg, 35%) as yellow oil.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.17 – 8.12 (m, 2H), 7.69 – 7.66 (m, 2H), 7.64 – 7.60 (m, 2H), 7.49 – 7.45 (m, 2H), 7.42 – 7.38 (m, 1H), 1.80 (t, *J* = 13.4 Hz, 2H), 1.75 – 1.68 (m, 1H), 1.63 – 1.57 (m, 2H), 1.50 – 1.44 (m, 1H), 1.30 (s, 6H), 1.15 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.1, 145.5, 139.9, 129.9, 128.8, 128.2, 128.0, 127.1, 127.0, 60.3, 38.9, 31.8, 20.7, 16.8.

HRMS (APCI) calcd for $C_{22}H_{28}NO_2^+$ $[M+H]^+$ *m/z*: 338.2115, found: 338.2110.

Radical probe experiment



To an oven-dried 10 mL vial charged with a stir bar, *S*-trilfluoromethyl thioester **1a** (0.3 mmol, 1.5 equiv.), $Ir(ppy)_2(dtbppy)PF_6$ (1.8 mg, 0.002 mmol, 1 mol%), potassium carbonate (6.9 mg, 0.05 mmol, 25 mol%) and alkene **9** (53.1 mg, 0.2 mmol, 1 equiv.) were added. The $Cu(MeCN)_4PF_6$ (3.7 mg, 0.01 mmol, 5 mol%) was added into the vial in the glove box. Then, the septum capped vial was transferred out of the glovebox and THF (2 mL) was added under the argon atmosphere. After degassed by three cycles of freeze-pump thaw, the mixture was placed on a photoreactor equipped with 6W LED lamps

and stirred for 12 hours. The solvent was evaporated and the residue was purified by column chromatography (petroleum ether/ethyl acetate = 5/1) to afford the **10** (23.7 mg, 22%) as white solid. (NMR yield is 28% using 1,3,5-trimethoxybenzene as internal standard.

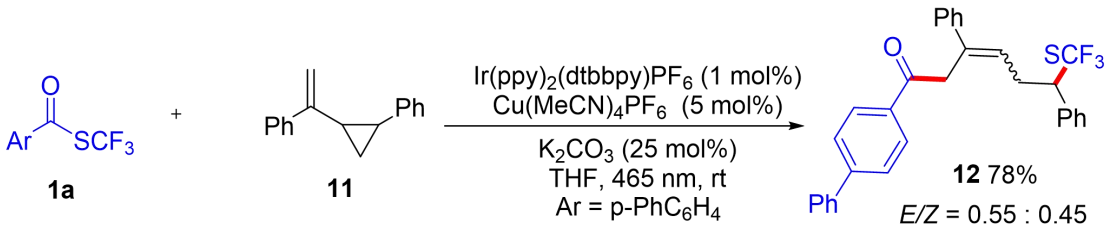
Mp = 211-213 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 – 7.89 (m, 2H), 7.64 – 7.61 (m, 2H), 7.59 (dd, *J* = 5.1, 3.3 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.40 – 7.36 (m, 1H), 7.14 – 7.06 (m, 3H), 6.97 (dd, *J* = 7.9, 0.7 Hz, 1H), 6.88 – 6.83 (m, 3H), 6.52 (d, *J* = 7.9 Hz, 1H), 3.81 (d, *J* = 1.3 Hz, 2H), 3.14 (d, *J* = 12.7 Hz, 1H), 3.08 (d, *J* = 12.7 Hz, 1H), 3.02 (s, 3H), 2.27 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.4, 179.0, 145.8, 142.0, 139.7, 135.1, 135.0, 131.0, 130.9, 130.1, 128.9, 128.6, 128.2, 128.1, 127.4, 127.2, 127.1, 126.6, 123.6, 107.5, 50.9, 44.9, 44.5, 26.0, 21.2.

HRMS (APCI) calcd for $C_{31}H_{28}NO_2^+$ $[M+H]^+$ *m/z*: 446.2115, found: 446.2109.

Radical clock experiment



To an oven-dried 10 mL vial charged with a stir bar, *S*-trilfluoromethyl thioester **1a** (0.3 mmol, 1.5 equiv.), $Ir(ppy)_2(dtbppy)PF_6$ (1.8 mg, 0.002 mmol, 1 mol%), and potassium carbonate (6.9 mg, 0.05 mmol, 25 mol%) were added. The $Cu(MeCN)_4PF_6$ (3.7 mg, 0.01 mmol, 5 mol%) was added into the vial in the glove box. Then, the septum capped vial was transferred out of the glovebox and alkene **11** (44.1 mg, 0.2 mmol, 1 equiv.) and THF (2 mL) were added under the argon atmosphere. After degassed by three cycles of freeze-pump thaw, the mixture was placed on a photoreactor equipped with 6W LED lamps and stirred for 12 hours. The solvent was evaporated and the residue was purified by column chromatography (petroleum ether/ethyl acetate = 5/1) to afford the *E*-**12** (42.9 mg, 43%, pale yellow oil) and *Z*-**12** (35.3 mg, 35%, yellow oil), respectively.

***E*-isomer**

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.70 – 7.66 (m, 2H), 7.65 – 7.61 (m, 2H), 7.49 – 7.45 (m, 2H), 7.41 (dt, *J* = 9.1, 4.2 Hz, 1H), 7.39 – 7.34 (m, 4H), 7.33 – 7.28 (m, 1H), 7.27 – 7.23 (m, 2H), 7.22 – 7.18 (m, 3H), 5.87 (t, *J* = 7.3 Hz, 1H), 4.49 (dd, *J* = 8.4, 6.6 Hz, 1H), 4.14 – 4.05 (m, 2H), 2.93 – 2.80 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.9, 146.0, 142.3, 140.0, 139.8, 136.3, 135.3, 130.5 (q, *J* = 307.4 Hz), 129.0, 128.8, 128.4, 128.3, 128.1, 127.5, 127.4, 127.28, 127.26, 127.24, 126.1, 49.4, 40.8, 36.4.

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.71.

***Z*-isomer**

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 – 7.89 (m, 2H), 7.63 – 7.59 (m, 4H), 7.48 – 7.45 (m, 2H), 7.41 – 7.38 (m, 1H), 7.32 – 7.28 (m, 2H), 7.27 – 7.22 (m, 4H), 7.17 – 7.14 (m, 2H), 7.06 – 7.03 (m, 2H), 5.54 – 5.43 (m, 1H), 4.32 – 4.25 (m, 1H), 3.98 – 3.90 (m, 2H), 2.75 – 2.60 (m, 2H).

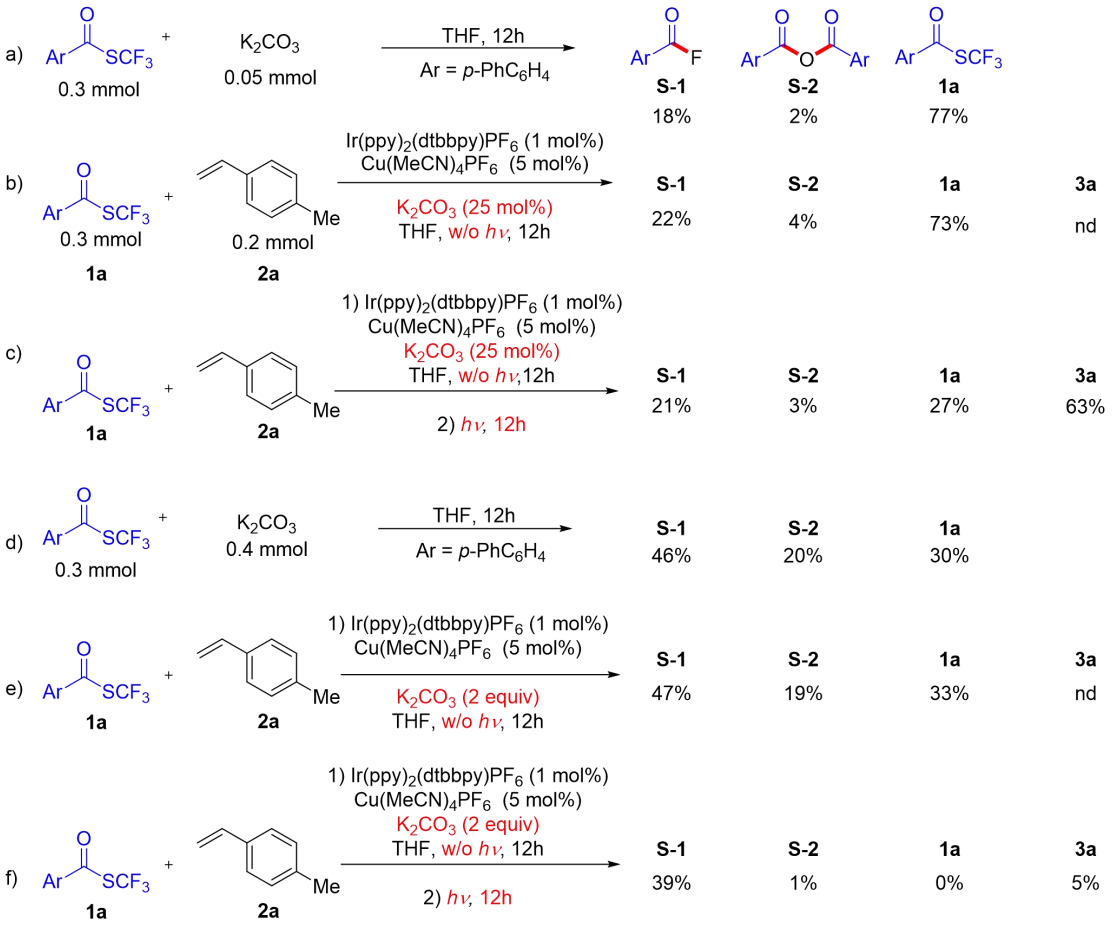
^{13}C NMR (151 MHz, $CDCl_3$) δ 197.0, 145.7, 139.82, 139.80, 139.5, 138.2, 135.4, 130.4 (q, *J* = 307.3 Hz), 128.96, 128.95, 128.6, 128.3, 128.23, 128.15, 127.9, 127.5, 127.3, 127.2, 127.1, 126.9, 49.53,

48.74, 36.08.

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.82.

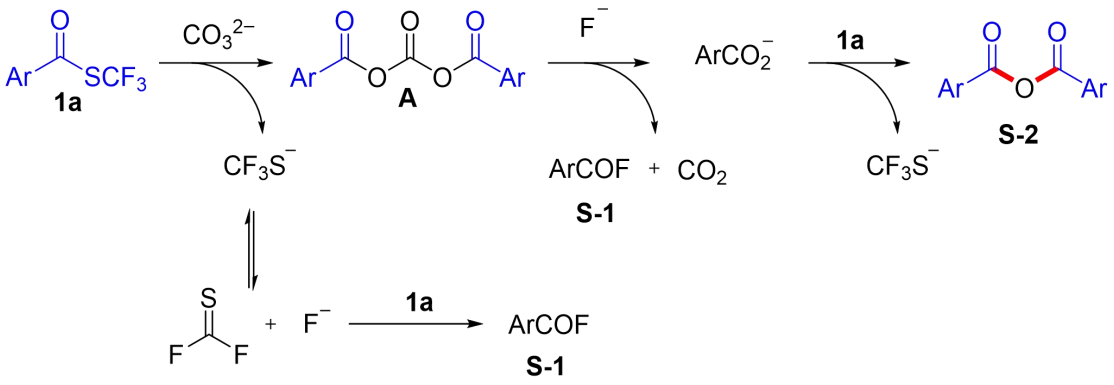
HRMS (APCI) calcd for $C_{31}H_{26}F_3OS^+$ $[M+H]^+$ *m/z*: 503.1651, found: 503.1646.

Controlled experiments



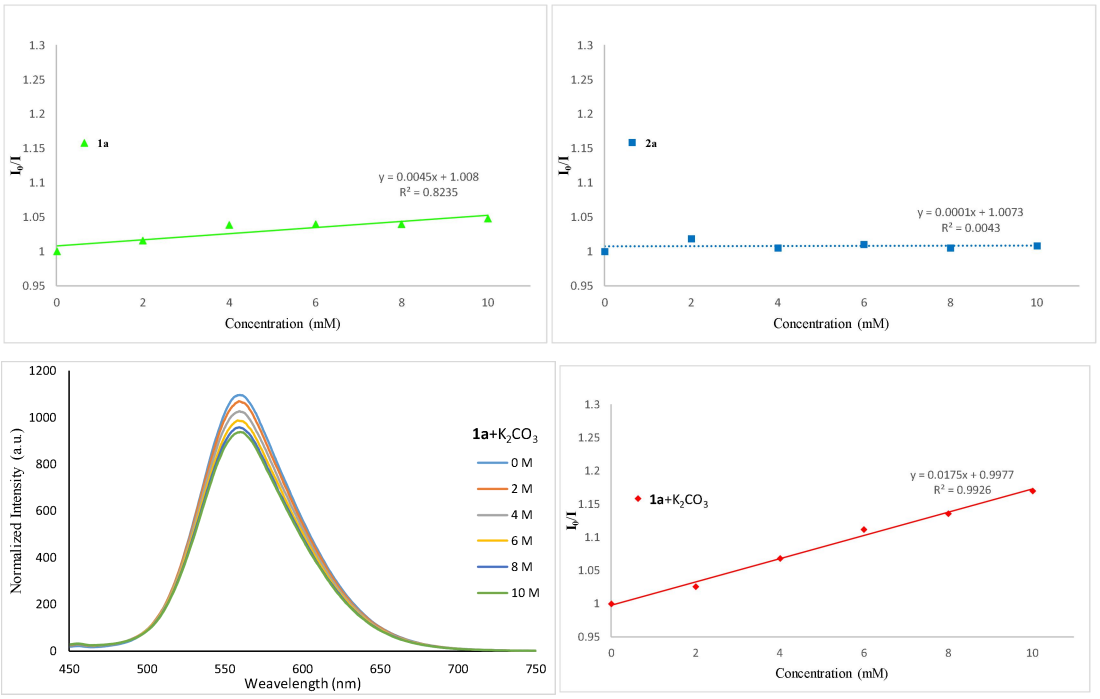
Scheme S3 The reaction of trifluoromethyl thioesters and K_2CO_3.

The reaction between trifluoromethyl thioesters and K_2CO_3 was performed. By using 0.05 mmol K_2CO_3, 19% of **14** and 2% of **15** were generated along with 77% of **1a** (**Scheme S3a**). The possible mechanism was shown in **Scheme-S4**. We tried to stirred the reaction mixture in the dark in the presence of 25 mol% K_2CO_3, the desired product **3a** was not detected (**Scheme S3b)**. Then, the mixture continued to stir for 12 h under blue LEDs irradiation and the product (**3a**) was obtained in 63% NMR yields (**Scheme S3c)**. This demonstrated that the product **3a** was derived from the residue **1a** rather the **S-1** or **S-2.** When the amount of K_2CO_3 increased to 2 equiv.alents, yields of **S-1** and **S-2** increased (**Scheme S3d** and **S3e**). On the contrary, the yield of **3a** decreased a lot (**Scheme S3f)** indicated that the generated potassium acetate may inhibited the generation of product **3a.** And it also showed that **S-1** and **S-2** are not the intermediates in this reaction.



Scheme-S4 The possible mechanism for formation of **S-1** and **S-2**.

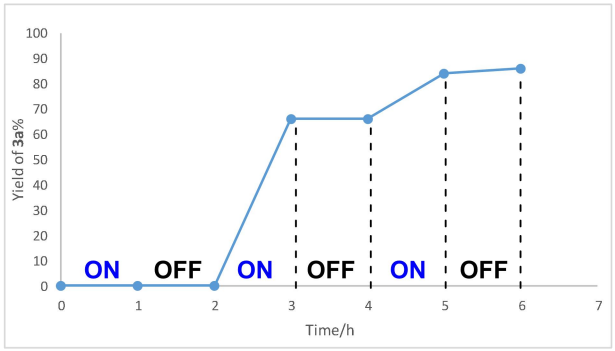
Stern-Volmer quenching experiments



Scheme-S5 Stern-Volmer quenching experiments.

The luminescence quenching experiments were taken using a F-4600 FL Spectrophotometer (Hitachi, Japan). The experiments were carried out in 1 x 10^{-4} mol/L of $[Ir(ppy)_2dtbbpy]PF_6$ in anhydrous degassed THF at room temperature under air atmosphere. The excitation wavelength was 420 nm and the emission intensity was collected at 525 nm. The concentrations of quencher in THF were 2 mM, 4 mM, 6 mM, 8 mM, 10 mM.

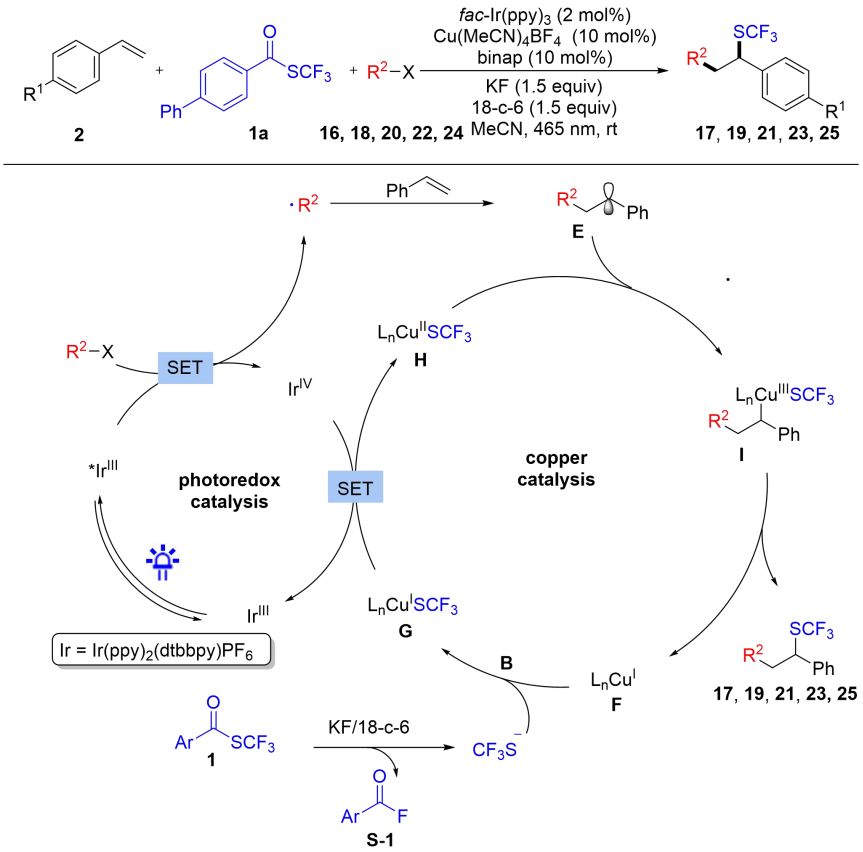
Light on-off experiments



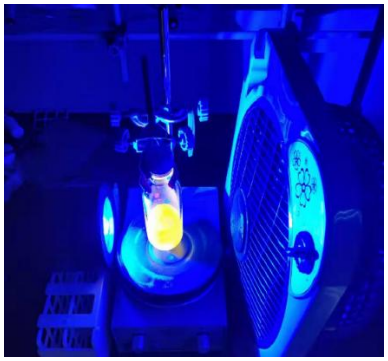
Scheme-S6 Light on-off experiments

The experiments were implied according to the general procedure for the preparation of compounds **3a**. Yields were determined via ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.

Mechanism of fluoroalkylation and arylation of alkenes with related radical precursors



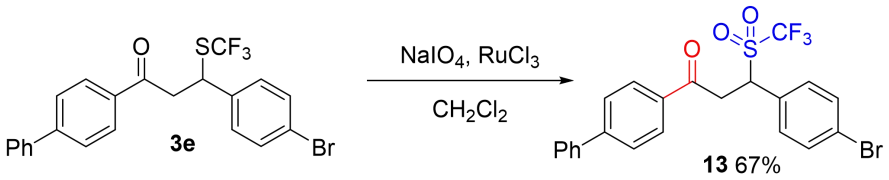
VI. Scale-up experiment (3e)



Light Sources. Reactions were carried out using 90W Blue LED lamps (Kessil A360WE-Series tuna blue) purchased from Kessil (λmax = 450 nm flanked by a second peak at λ = 422 nm). Kessil lamps were placed approximately 3 cm away from the flask. A fan was placed 4 cm away from the flask for cooling when the Kessil lamp was in use.

To an oven-dried 100 mL a heavy-wall pressure vessel charged with a stir bar, *S*-trilfluoromethyl thioester **1a** (1.13 g, 6 mmol, 1.5 equiv.), $Ir(ppy)_2(dtbppy)PF_6$ (36.6 mg, 0.04 mmol, 1 mol%), and potassium carbonate (138.2 mg, 0.1 mmol, 25 mol%) were added. The vessel was then transported inside a glove box under argon atmosphere. The $Cu(MeCN)_4PF_6$ (74.5 mg, 0.2 mmol, 5 mol%), alkene **2e** (732 mg, 4 mmol, 1 equiv.) and degassed THF (40 mL) were added into the vessel in the glove box, which was then sealed with a cap. The closed vessel was removed from the glove box and was placed on a photoreactor equipped with 90 W LED lamps and stirred for 24 hours. The solvent was evaporated and the residue was purified by column chromatography to afford the desired products **3e** (1.35 g, 73%).

VII. Derivatization reactions of 3e.



A 10 mL vial was charged with a stir bar, **3e** (46.5 mg, 0.1 mmol, 1 equiv.), CCl_4 (0.2 ml), MeCN (0.2 ml) and H_2O (0.4 ml), $NaIO_4$ (64.2 mg, 0.3 mmol, 3 equiv.) and, $RuCl_3$ (2 mg, 0.01 mmol, 1 mol%) were added. The mixture was continuously stirred at room temperature for 14 h. Then the mixture was extracted with CH_2Cl_2. The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuum. The residues were purified by column chromatograph using silica gel with petroleum ether/ethyl acetate eluents to afford the desired product **13** (33.3 mg, 67%) as white solid.

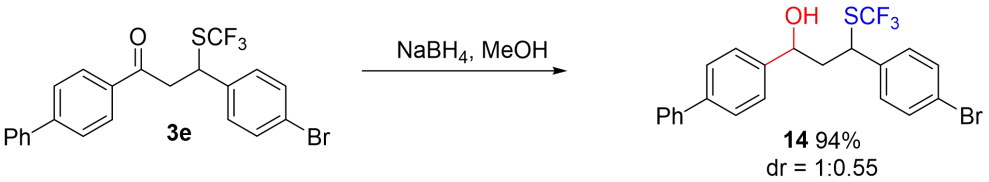
Mp = 148-150 °C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.63 – 7.59 (m, 2H), 7.57 – 7.53 (m, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.44 – 7.39 (m, 3H), 5.33 (dd, *J* = 9.7, 3.2 Hz, 1H), 4.04 (dd, *J* = 17.7, 3.3 Hz, 1H), 3.94 (dd, *J* = 17.7, 9.8 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 192.4, 147.0, 139.4, 134.0, 132.4, 131.5, 129.0, 128.8, 128.6, 128.5, 127.5, 127.3, 124.6, 119.8 (q, J = 329.4 Hz), 61.9, 37.1.

^{19}F NMR (565 MHz, $CDCl_3$) δ -73.14.

HRMS (ESI) calcd for $C_{22}H_{15}BrF_3O_3S^+$ $[M\text{-}H]^-$ *m/z*: 494.9883, found: 494.9875.



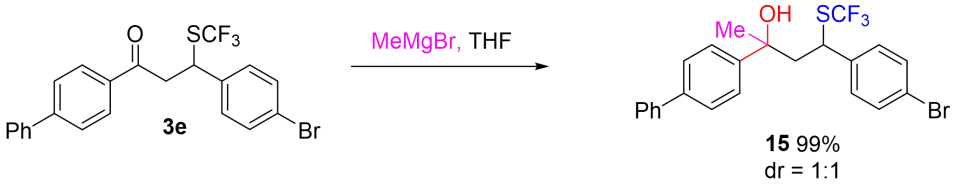
To an oven-dried 10 mL vial charged with a stir bar, **3e** (46.5 mg, 0.1 mmol, 1 equiv.), and EtOH (1 mL) were added. Then $NaBH_4$ (45.4 mg, 1.2 mmol, 1.2 equiv.) was added at 0 °. The resulting mixture was stirred at room temperature for 2 h. The reaction was quenched with water. Then the mixture was extracted with CH_2Cl_2. The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuum. The residues were purified by column chromatograph using silica gel with petroleum ether/ethyl acetate eluents to afford the desired product **14** (.43.7 mg, 94) as pale yellow oil.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 – 7.45 (m, 4H), 7.45 – 7.33 (m, 4H), 7.30 – 7.21 (m, 3H), 7.18 (d, J = 8.4 Hz, 2H, minor), 7.12 (d, J = 8.4 Hz, 2H, major), 4.75 (dd, J = 8.6, 4.7 Hz, 1H, major), 4.57 (dd, J = 10.9, 4.9 Hz, 1H, minor), 4.41 (dd, J = 8.9, 6.1 Hz, 1H, major), 4.29 (dd, J = 9.9, 3.1 Hz, 1H, minor), 2.42 – 2.34 (m, 1H, minor), 2.34 – 2.26 (m, 1H, major), 2.22 – 2.15 (m, 1H, major), 2.13 – 2.06 (m, 1H, minor), 2.00 (brs, 1H, major), 1.83 (brs, 1H, minor).

^{13}C NMR (151 MHz, $CDCl_3$) δ 142.4, 142.0, 141.2, 141.1, 140.5, 140.2, 139.0, 132.1, 132.0, 130.3 (q, J = 307.7 Hz), 130.2 (q, J = 307.7 Hz). 129.4, 129.1, 128.8, 127.49, 127.47, 127.43, 127.1, 126.4, 126.1, 122.0, 121.8, 71.2, 70.9, 45. 8, 45.6, 45.1, 45.0.

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.52 (major), -39.62 (minor).

HRMS (ESI) calcd for $C_{22}H_{17}BrF_3OS^+$ $[M\text{-}H]^-$ *m/z*: 465.0141, found: 465.0134.



To a stirred solution of **3e** (46.5 mg, 0.1 mmol, 1 equiv.) in THF (1 mL) was slowly added 1 M methyl magnesium bromide in THF (0.15 mL) at -78 °C. Then, reaction temperature was raised to room temperature. After stirring for 1 h, the reaction mixture was quenched by water. Then the mixture was extracted with ethyl acetate. The combined organic layers were dried over anhydrous sodium sulfate and concentrated in vacuum. The residues were purified by column chromatograph using silica gel with petroleum ether/ethyl acetate eluents to afford the desired product **15** (48.0 mg, 99%) as yellow oil.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.55 – 7.47 (m, 6H), 7.40 – 7.33 (m, 8H), 7.33 – 7.30 (m, 2H), 7.30 – 7.25 (m, 2H), 7.24 – 7.20 (m, 2H), 7.20 – 7.17 (m, 2H), 7.03 – 6.98 (m, 2H), 6.86 – 6.82 (m, 2H), 4.31 (dd, J = 7.5, 5.5 Hz, 1H), 4.25 (dd, J = 9.1, 4.9 Hz, 1H), 2.54 – 2.45 (m, 3H), 2.39 (dd, J = 14.8, 5.5 Hz, 1H), 1.92 (s, 1H), 1.51 (s, 3H), 1.47 (s, 1H), 1.44 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 145.2, 144.6, 140.6, 140.5, 140.1, 139.9, 139.8, 132.0, 131.5, 129.92 (q, J = 307.5 Hz), 129.89 (q, J = 308.1 Hz), 129.4, 129.2, 128.80, 128.75, 127.4, 127.3, 127.14, 127.12,

127.05, 126.9, 125.13, 125.11, 121.98, 121.26, 74.8, 74.3, 50.1, 49.4, 45.2, 44.9, 31.5, 31.1.

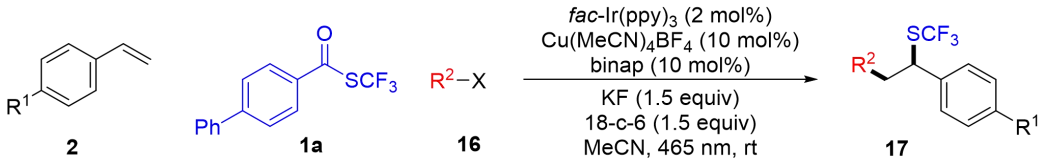
^{19}F NMR (565 MHz, $CDCl_3$) δ -39.91, -40.02.

HRMS (ESI) calcd for $C_{22}H_{19}BrF_3OS^+$ $[M-H]^-$ *m/z*: 479.0298, found: 479.0291.

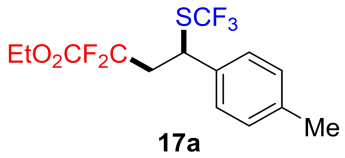
VIII. Carbo-trifluoromethylthiolation of alkenes with other radical precursors

The study of trifluoromethylthio-difluoroalkylation of alkenes

The general procedure for the synthesis of **17** and **19**.



An oven-dried 10 mL vial was charged with a stir bar, *S*-trilfluoromethyl thioester **1a** (42.3 mg, 0.15 mmol, 1.5 equiv.), *fac*-Ir(ppy)$_3$ (1.3 mg, 0.002 mmol, 2 mol%) and, binap (6.2 mg, 0.01 mmol, 10 mol%) were added. Then, $Cu(MeCN)_4BF_4$ (3.1 mg, 0.01 mmol, 10 mol%), KF (8.7 mg, 0.15 mmol, 1.5 equiv.), 18-Crown-6 (39.7 mg, 0.15 mmol, 1.5 equiv.) were added into the vial in the glove box. The septum capped vial was transferred out of the glovebox. Alkene **2** (0.1 mmol, 1 equiv.), **16** (0.15 mmol, 1.5 equiv.), (if solid, added before sealed), and anhydrous MeCN (2 mL) were added under the argon atmosphere. The mixture was placed on a photoreactor equipped with 6W LED lamps and stirred for 12 hours. The solvent was evaporated and the residue was purified by column chromatography to afford the desired products **17**.



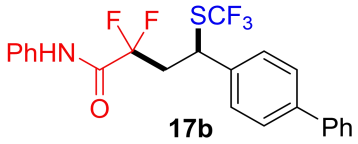
Ethyl (R)-2,2-difluoro-4-(p-tolyl)-4-((trifluoromethyl)thio)butanoate (**17a**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to 4/1) to give 28.1 mg product as a colorless oil in 82% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.19 (d, *J* = 8.1 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 4.58 (dd, *J* = 9.8, 4.9 Hz, 1H), 4.07 – 3.97 (m, 2H), 2.99 – 2.88 (m, 1H), 2.83 (dtd, *J* = 14.7, 14.7, 4.9 Hz, 1H), 2.33 (s, 3H), 1.23 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 163.09 (t, *J* = 32.1 Hz), 138.58, 134.89, 129.87 (q, *J* = 308.2 Hz), 129.57, 127.48, 114.15 (dd, *J* = 253.3), 63.05, 42.73, 41.31 (t, *J* = 23.9 Hz), 21.10, 13.64.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.36 (s), -102.39 (dt, *J* = 266.1, 13.9 Hz), -105.46 (dt, *J* = 266.1, 15.8 Hz).

HRMS (APCI) calcd for $C_{13}H_{15}O_2F_2^+$ $[M-SCF_3]^+$ *m/z*: 241.1035, found: 241.1032.



17b

4-([1,1'-Biphenyl]-4-yl)-2,2-difluoro-N-phenyl-4-((trifluoromethyl)thio)butanamide (**17b**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to give 33.0 mg product as a white solid in 73% yield.

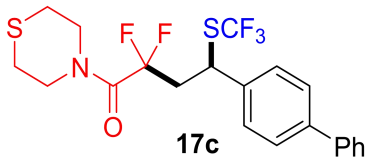
Mp = 113-114 °C

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 (brs, 1H), 7.51 (d, *J* = 7.9 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.42 – 7.38 (m, 4H), 7.38 – 7.31 (m, 3H), 7.28 (t, *J* = 7.9 Hz, 2H), 7.14 (dd, *J* = 10.9, 3.8 Hz, 1H), 4.76 – 4.62 (m, 1H), 3.13 (tdd, *J* = 15.0, 15.0, 10.1 Hz, 1H), 2.99 (tdd, *J* = 15.1, 15.1, 5.4 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 160.78 (t, *J* = 28.0 Hz), 141.66, 140.07, 136.89, 135.52, 129.91 (q, *J* = 308.1 Hz), 129.15, 128.71, 127.99, 127.62, 127.58, 127.06, 125.70, 120.14, 116.23 (t, *J* = 256.5 Hz), 42.93. 40.20 (t, *J* = 23.6 Hz).

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.13 (s, 3F), -100.94 (dt, *J* = 260.9, 14.9 Hz, 1F), -104.90 (dt, *J* = 260.9, 16.1 Hz, 1F).

HRMS (APCI) calcd for $C_{22}H_{18}ONF_2^+$ $[M\text{-}SCF_3]^+$ *m/z*: 350.1351, found: 350.1344.



17c

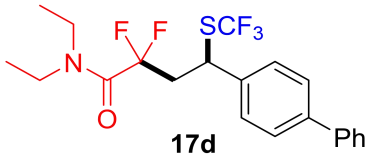
4-([1,1'-Biphenyl]-4-yl)-2,2-difluoro-1-thiomorpholino-4-((trifluoromethyl)thio)butan-1-one (**17c**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to give 38.9 mg product as a pale yellow oil in 84% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 – 7.54 (m, 4H), 7.45 – 7.42 (m, 2H), 7.42 – 7.39 (m, 2H), 7.37 – 7.33 (m, 1H), 4.82 (dd, *J* = 7.2, 6.6 Hz, 1H), 3.93 – 3.79 (m, 4H), 3.07 – 2.95 (m, 2H), 2.63 (t, *J* = 5.2 Hz, 2H), 2.62 – 2.59 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 161.14 (t, *J* = 28.6 Hz), 141.13, 140.25, 138.52, 129.99 (q, *J* = 308.0 Hz), 128.79, 127.82, 127.53, 127.51, 127.02, 117.97 (t, *J* = 258.1 Hz), 48.58 (t, *J* = 6.0 Hz), 46.04, 42.86, 41.33 (t, *J* = 22.4 Hz), 28.04, 27.23.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.23 (s), -97.04 (ddd, *J* = 285.4, 20.5, 13.5 Hz, 1H), -98.73 (ddd, *J* = 285.4, 20.5, 14.6 Hz, 1H).

HRMS (APCI) calcd for $C_{20}H_{20}ONSF_2^+$ $[M\text{-}SCF_3]^+$ *m/z*: 360.1228, found: 360.1221.



17d

4-([1,1'-Biphenyl]-4-yl)-*N*,*N*-diethyl-2,2-difluoro-4-((trifluoromethyl)thio)butanamide (**17d**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to give 26.0 mg

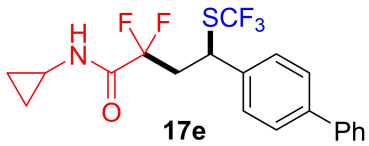
product as a pale yellow oil in 61% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.54 (m, 4H), 7.46 – 7.39 (m, 4H), 7.37 – 7.32 (m, 1H), 4.83 (dd, *J* = 8.0, 6.3 Hz, 1H), 3.51 – 3.40 (m, 2H), 3.40 – 3.26 (m, 2H), 3.07 – 2.93 (m, 2H), 1.14 (t, *J* = 7.0 Hz, 3H), 1.12 (t, *J* = 7.1 Hz, 3H)

^{13}C NMR (151 MHz, $CDCl_3$) δ 161.91 (t, *J* = 28.2 Hz), 141.01, 140.35, 138.81, 130.06 (q, *J* = 307.6 Hz),128.77, 127.86, 127.46, 127.03, 118.01 (t, *J* = 257.9 Hz), 43.01, 41.79 (t, *J* = 6.2 Hz), 41.63, 41.43 (t, *J* = 22.9 Hz), 14.16, 12.22.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.24 (s), -97.54 (ddd, *J* = 283.2, 20.1, 12.9 Hz), -98.75 – -99.39 (ddd, *J* = 283.2, 20.4, 14.7 Hz).

HRMS (APCI) calcd for $C_{20}H_{22}ONF_2^+$ $[M\text{-}SCF_3]^+$ *m/z*: 330.1664, found: 330.1657.



17e

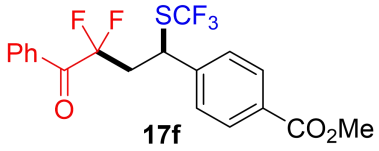
4-([1,1'-Biphenyl]-4-yl)-*N*-cyclopropyl-2,2-difluoro-4-((trifluoromethyl)thio)butanamide (**17e**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 4/1) to give 30.0 mg product as a yellow oil in 61% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.1 Hz, 4H), 7.47 – 7.41 (m, 2H), 7.38 (d, *J* = 8.2 Hz, 2H), 7.37 – 7.34 (m, 1H), 6.26 (brs, 1H), 4.66 (dd, *J* = 9.3, 5.9 Hz, 1H), 3.00 (tdd, *J* = 15.4, 15.4, 9.3 Hz, 1H), 2.88 (tdd, *J* = 15.4, 15.4, 5.9 Hz, 1H), 2.64 – 2.55 (m, 1H), 0.81 – 0.70 (m, 2H), 0.52 – 0.45 (m, 1H), 0.44 – 0.36 (m, 1H).

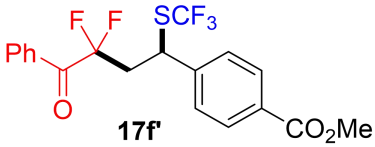
^{13}C NMR (151 MHz, $CDCl_3$) δ 141.46, 140.14, 137.40, 128.82, 127.97, 127.61, 127.54, 127.01, 42.87, 22.43, 6.39, 6.27.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.20 (d, *J* = 5.3 Hz, 3F), -102.06 (dt, *J* = 261.7, 16.4 Hz, 1F), -104.84 (dt, *J* = 261.9, 16.9 Hz, 1F).

HRMS (APCI) calcd for $C_{19}H_{18}ONF_2^+$ $[M\text{-}SCF_3]^+$ *m/z*: 314.1351, found: 314.1345.



17f



17f'

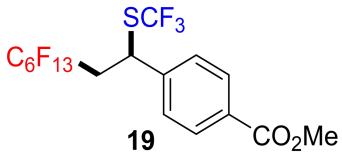
Methyl -4-(3,3-difluoro-4-oxo-4-phenyl-1-((trifluoromethyl)thio)butyl)benzoate (**17f**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to give 29.3 mg mixture product (1:0.15) as a yellow oil. The yield of **5f** is 61%.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.02 – 7.97 (m, 4H), 7.65 – 7.59 (m, 1H), 7.47 – 7.40 (m, 4H), 4.82 (t, *J* = 7.2 Hz 1H), 3.91 (s, 3H), 3.05 – 2.94 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 188.09 (t, *J* = 30.7 Hz), 166.39, 144.32, 134.63, 131.21 (t, *J* = 2.5 Hz), 130.21, 130.19, 130.10 (t, *J* = 2.8 Hz), 129.73 (d, *J* = 310.3 Hz), 128.71, 127.56, 117.81 (t, *J* = 256.5 Hz), 52.18, 42.64, 40.00 (t, *J* = 22.4 Hz).

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.22 (s, 3F), -97.43 (dt, *J* = 297.2, 16.5 Hz, 1F), -98.46 (dt, *J* = 297.2, 16.7 Hz, 1H).

HRMS (APCI) calcd for $C_{18}H_{15}O_3F_2^+$ [M-SCF_3]$^+$ *m/z*: 317.0984 found: 317.0980.



19

Methyl -4-(3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluoro-1-((trifluoromethyl)thio)octyl)benzoate (**19**) was synthesized according to General Procedure and purified by flash silica column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to give 42.2 mg product as a yellow oil in 72% yield.

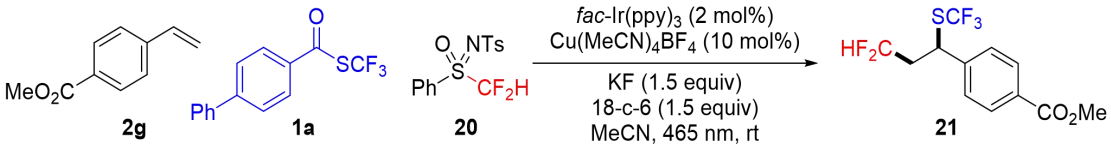
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.09 – 8.04 (m, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 4.78 (dd, *J* = 8.7, 5.5 Hz, 1H), 3.93 (s, 3H), 2.98 – 2.81 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.27, 143.19, 130.65, 130.49, 127.30, 52.26, 41.59.

^{19}F NMR (565 MHz, $CDCl_3$) δ -40.44 (s, 3F), -80.86 (t, *J* = 10.1 Hz, 3F), -111.38 – -112.12 (m, 1F), -113.20 – -113.90 (m, 1F), -121.84 (s, 2F), -122.90 (s, 2F), -123.46 (s, 2F), -126.10 126.27 (m, 2F).

HRMS (APCI) calcd for $C_{13}H_{15}O_2F_2^+$ [M+H]$^+$ *m/z*: 583.0219, found: 583.0210.

The study of trifluoromethylthio-difluoromethylation of alkenes



2g **1a** **20** **21**

An oven-dried 10 mL vial was charged with a stir bar, *S*-trilfluoromethyl thioester **1a** (42.3 mg, 0.15 mmol, 1.5 equiv.), *fac*-Ir(ppy)$_3$ (1.3 mg, 0.002 mmol, 2 mol%), Hu's reagent **20** (51.8 mg, 0.15 mmol, 1.5 equiv.), and alkene **2g** (16.2 mg, 0.15 mmol, 1 equiv.) were added. Then, $Cu(MeCN)_4BF_4$ (3.1 mg, 0.01 mmol, 10 mol%), KF (8.7 mg, 0.15 mmol, 1.5 equiv.), 18-Crown-6 (39.7 mg, 0.15 mmol, 1.5 equiv.) were added into the vial in the glove box. The septum capped vial was transferred out of the glovebox. Anhydrous MeCN (2 mL) was added under the argon atmosphere. The mixture was placed on a photoreactor equipped with 6W LED lamps and stirred for 12 hours. The solvent was evaporated and the residue was purified by column chromatography (petroleum ether/dichloromethane = 10/1 to petroleum ether/ethyl acetate = 20/1) to give 24.6 mg product **21** as a colorless oil in 78% yield.

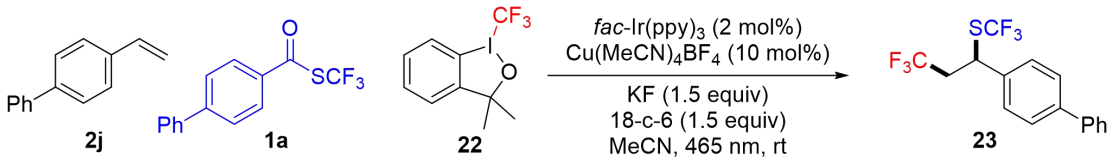
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08 – 8.03 (m, 2H), 7.44 – 7.40 (m, 2H), 5.67 (tt, *J* = 55.8, 4.6 Hz, 1H), 4.53 (dd, *J* = 8.2, 7.6 Hz, 1H), 3.93 (s, 3H), 2.63 – 2.44 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.28, 143.61, 130.52, 130.50, 129.74 (q, *J* = 308.1 Hz), 127.30, 114.49 (t, *J* = 240.8 Hz), 52.27, 43.12, 40.44 (t, *J* = 23.0 Hz).

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.95 (s, 3F), -117.12 (dddd, *J* = 66.7, 55.8, 16.8, 11.5 Hz, 1H), -118.00 (dddd, *J* = 289.2, 56.2, 20.1, 12.1 Hz, 1H).

HRMS (APCI) calcd for $C_{11}H_{11}O_2F_2^+$ [M-SCF_3]$^+$ *m/z*: 213.0722, found: 213.0725.

The study of trifluoromethylthio-trifluoromethylation of alkenes



To an oven-dried 10 mL vial charged with a stir bar, *S*-trilfluoromethyl thioester **1a** (42.3 mg, 0.15 mmol, 1.5 equiv.), *fac*-Ir(ppy)$_3$ (1.3 mg, 0.002 mmol, 2 mol%), Togni-I reagnent **22** (49.5 mg, 0.15 mmol, 1.5 equiv.), and alkene **2j** (18.0 mg, 0.10 mmol, 1 equiv.) were added. Then, Cu(MeCN)$_4$BF$_4$ (3.1 mg, 0.01 mmol, 10 mol%), KF (8.7 mg, 0.15 mmol, 1.5 equiv.), 18-Crown-6 (39.7 mg, 0.15 mmol, 1.5 equiv.) were added into the vial in the glove box. The septum capped vial was transferred out of the glovebox. Anhydrous MeCN (2 mL) was added under the argon atmosphere. The mixture was placed on a photoreactor equipped with 6W LED lamps and stirred for 12 hours. The solvent was evaporated and the residue was purified by column chromatography (petroleum ether/dichloromethane = 20/1) to give 19.6 mg product **23** as a white solid in 56% yield.

Mp = 101-103 °C

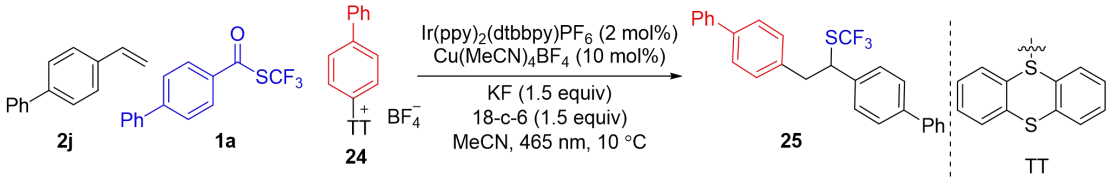
^{1}H NMR (600 MHz, CDCl$_3$) δ 7.59 (t, *J* = 8.5 Hz, 4H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.39 (d, *J* = 8.2 Hz, 2H), 7.38 – 7.33 (m, 1H), 4.66 (dd, *J* = 9.3, 5.4 Hz, 1H), 3.03 – 2.84 (m, 2H).

^{13}C NMR (151 MHz, CDCl$_3$) δ 141.70, 140.10, 136.60, 129.81 (q, *J* = 308.2 Hz), 128.85, 127.80, 127.69, 127.66, 127.08, 124.83 (q, *J* = 278.5 Hz), 42.68, 40.90 (q, *J* = 28.4 Hz).

^{19}F NMR (565 MHz, CDCl$_3$) δ -40.27 (s), -63.85 (t, *J* = 10.6 Hz).

HRMS (APCI) calcd for $C_{15}H_2F_3^+$ [M-SCF$_3$]$^+$ *m/z*: 249.0886, found: 249.0883.

The study of trifluoromethylthio-arylation of alkenes



To an oven-dried 10 mL vial charged with a stir bar, *S*-trilfluoromethyl thioester **1a** (42.3 mg, 0.15 mmol, 1.5 equiv.), Ir(ppy)$_2$(dtbbpy)PF$_6$ (1.8 mg, 0.002 mmol, 1 mol%), sulfonium salt **24** (51.8 mg, 0.15 mmol, 1.5 equiv.), and alkene **2j** (18.0 mg, 0.10 mmol, 1 equiv.) were added. Then, Cu(MeCN)$_4$BF$_4$ (3.1 mg, 0.01 mmol, 10 mol%), KF (8.7 mg, 0.15 mmol, 1.5 equiv.), 18-Crown-6 (39.7 mg, 0.15 mmol, 1.5 equiv.) were added into the vial in the glove box. The septum capped vial was transferred out of the glovebox. Anhydrous MeCN (2 mL) was added under the argon atmosphere. The mixture was placed on a photoreactor equipped with 6W LED lamps and stirred for 12 hours at 10 °C. The solvent was evaporated and the residue was purified by column chromatography (petroleum ether/dichloromethane = 20/1) to give 17.2 mg product **25** as a colorless oil in 40% yield.

^{1}H NMR (600 MHz, CDCl$_3$) δ 8.06 – 7.86 (m, 2H), 7.56 – 7.51 (m, 2H), 7.47 – 7.38 (m, 4H), 7.36 – 7.30 (m, 3H), 7.04 (d, *J* = 8.2 Hz, 2H), 4.60 (dd, *J* = 9.1, 6.2 Hz, 1H), 3.90 (s, 3H), 3.34 (dd, *J* = 13.9, 6.2 Hz, 1H), 3.23 (dd, *J* = 13.9, 9.1 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.59, 144.94, 140.49, 139.92, 135.63, 130.19 (q, J = 307.5 Hz), 129.95, 129.82, 129.54, 128.74, 127.80, 127.31, 127.12, 126.94, 52.15, 50.58, 42.50.

^{19}F NMR (565 MHz, $CDCl_3$) δ -39.68.

HRMS (APCI) calcd for $C_{22}H_{19}O_2^+$ $[M\text{-}SCF_3]^+$ *m/z*: 315.1380, found: 315.1375.

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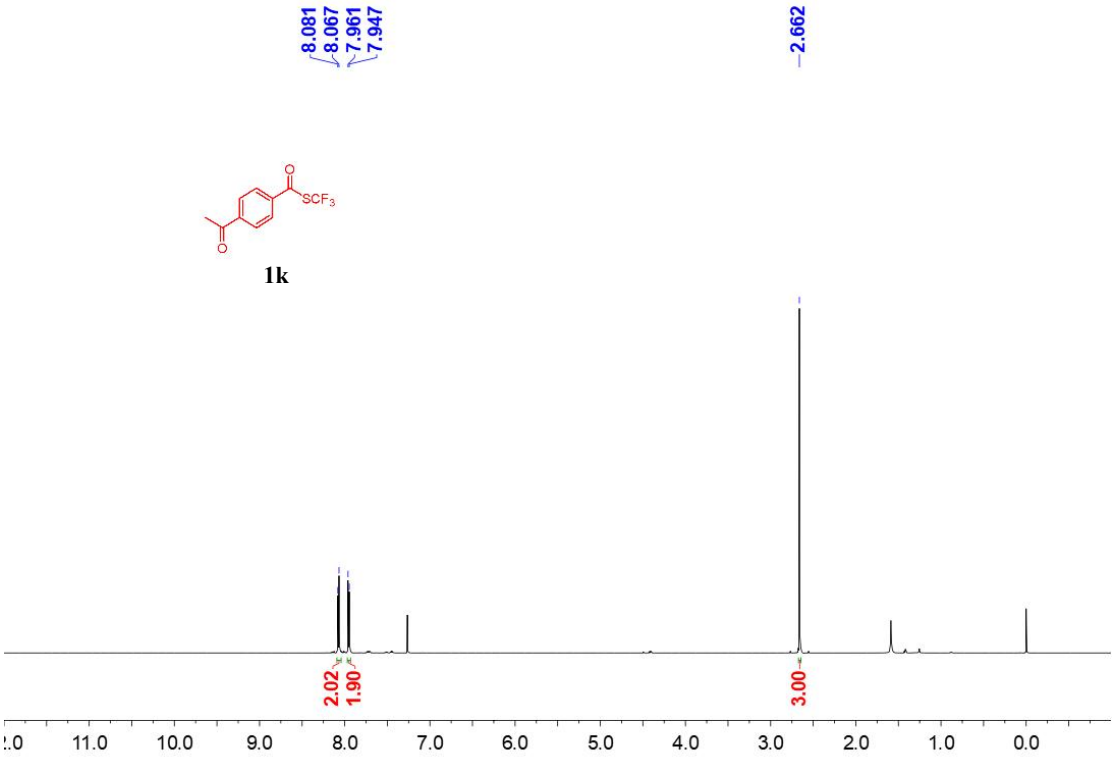
18. D. Shi; Y, Tian; R. Liu; X. Li, CN112358427B, **2021.**

X. Copies of ^{1}H, ^{13}C and ^{19}F NMR Spectra



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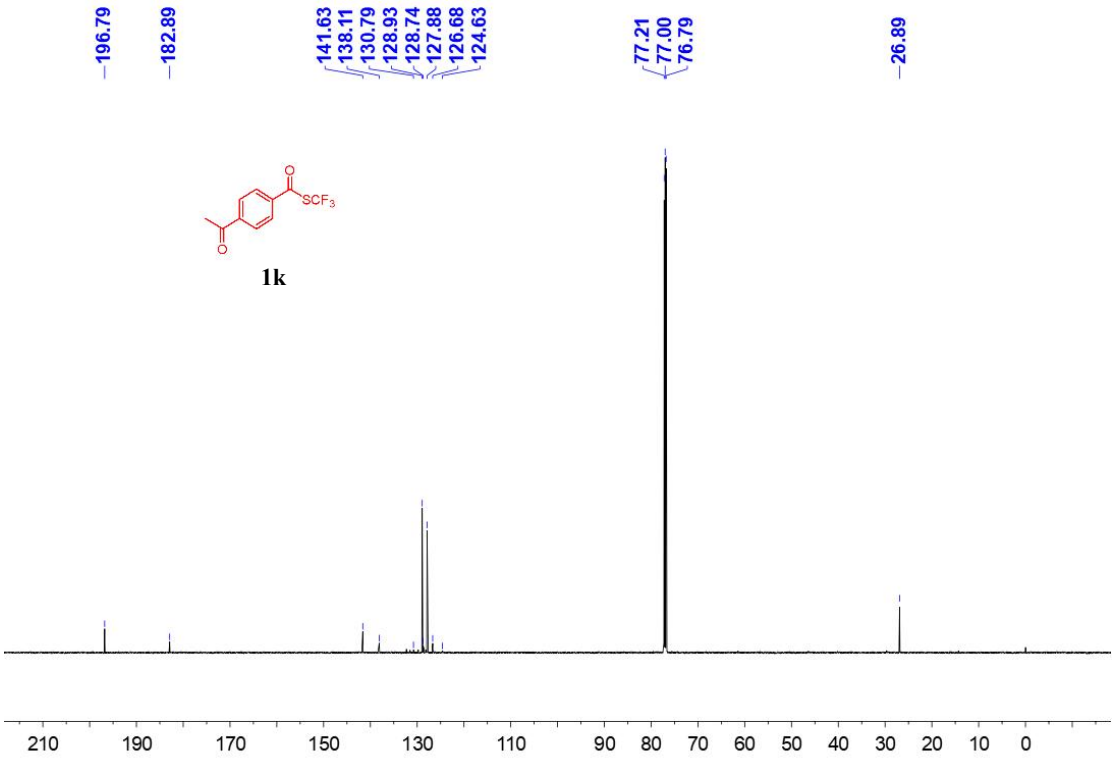

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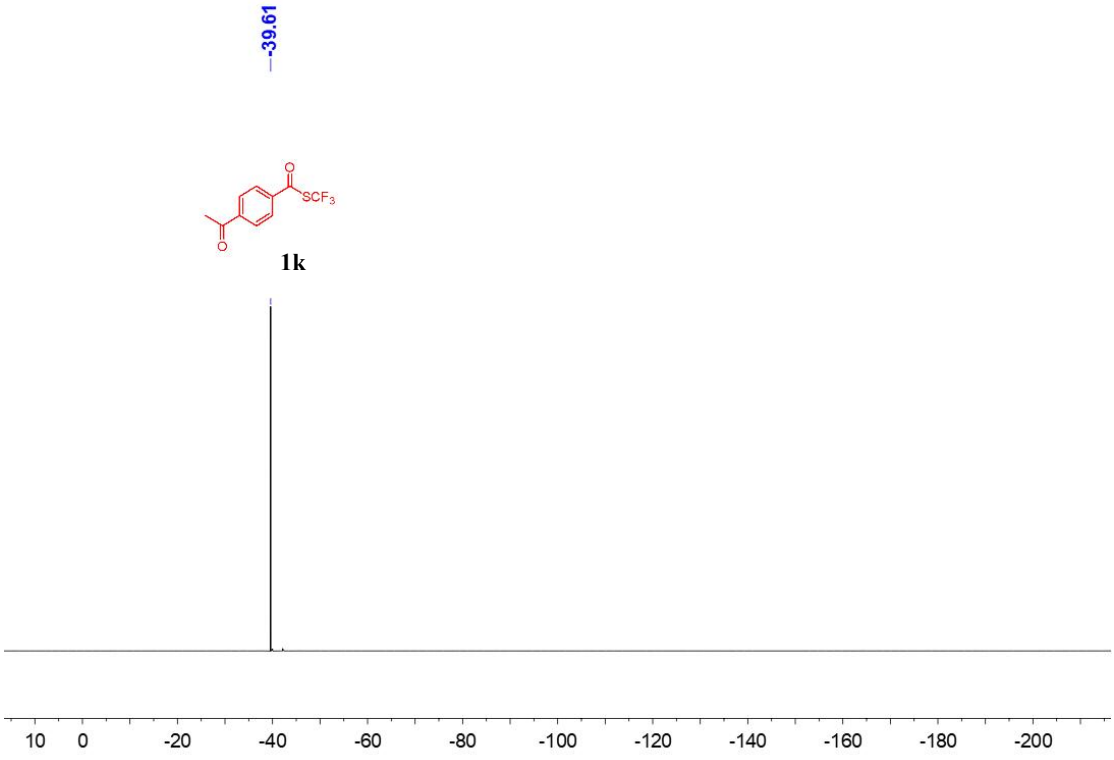
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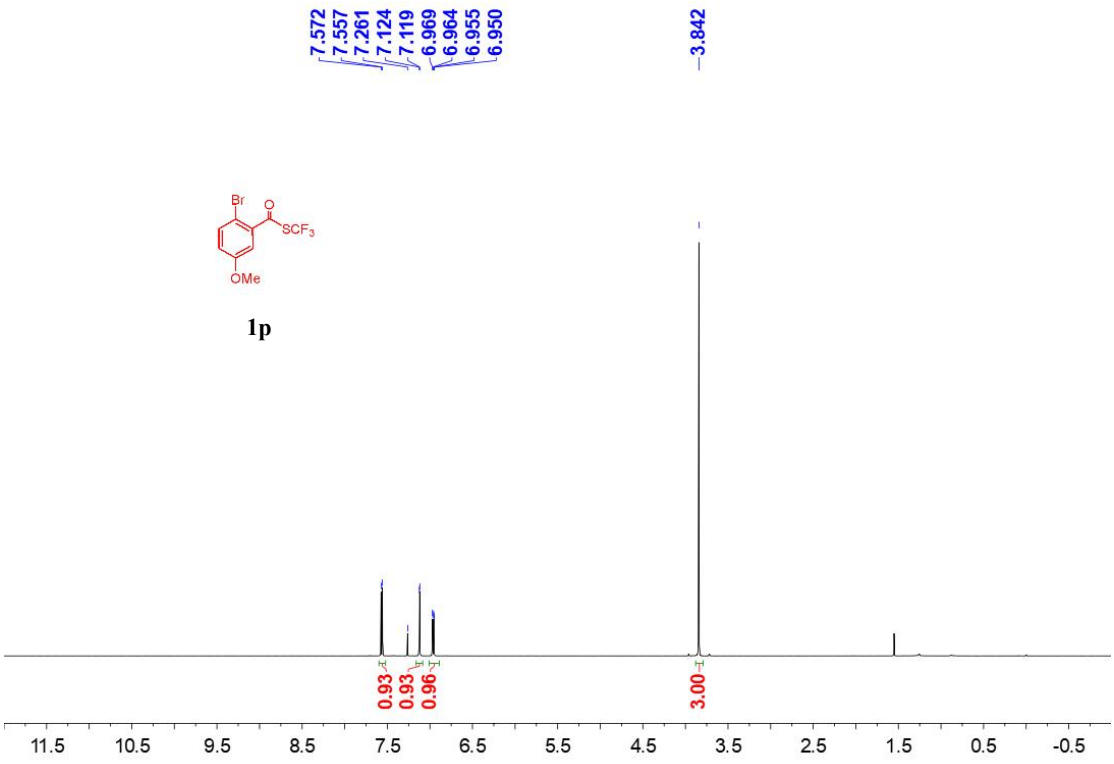
1k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0.93
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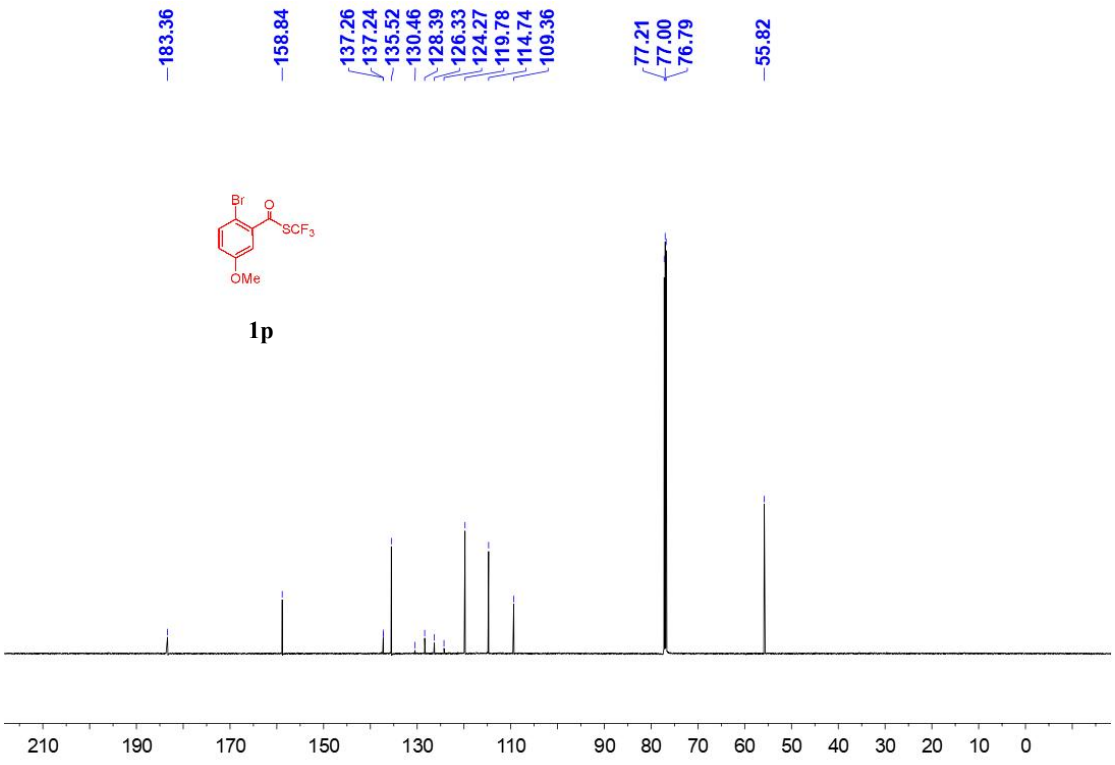
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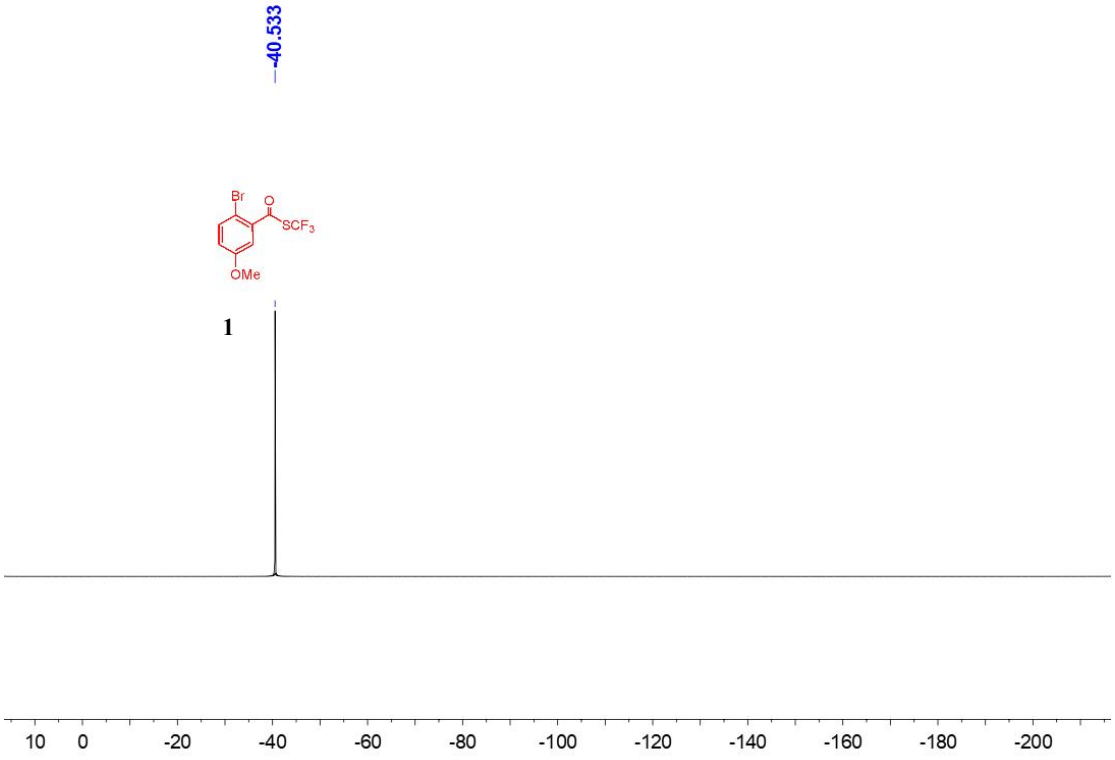
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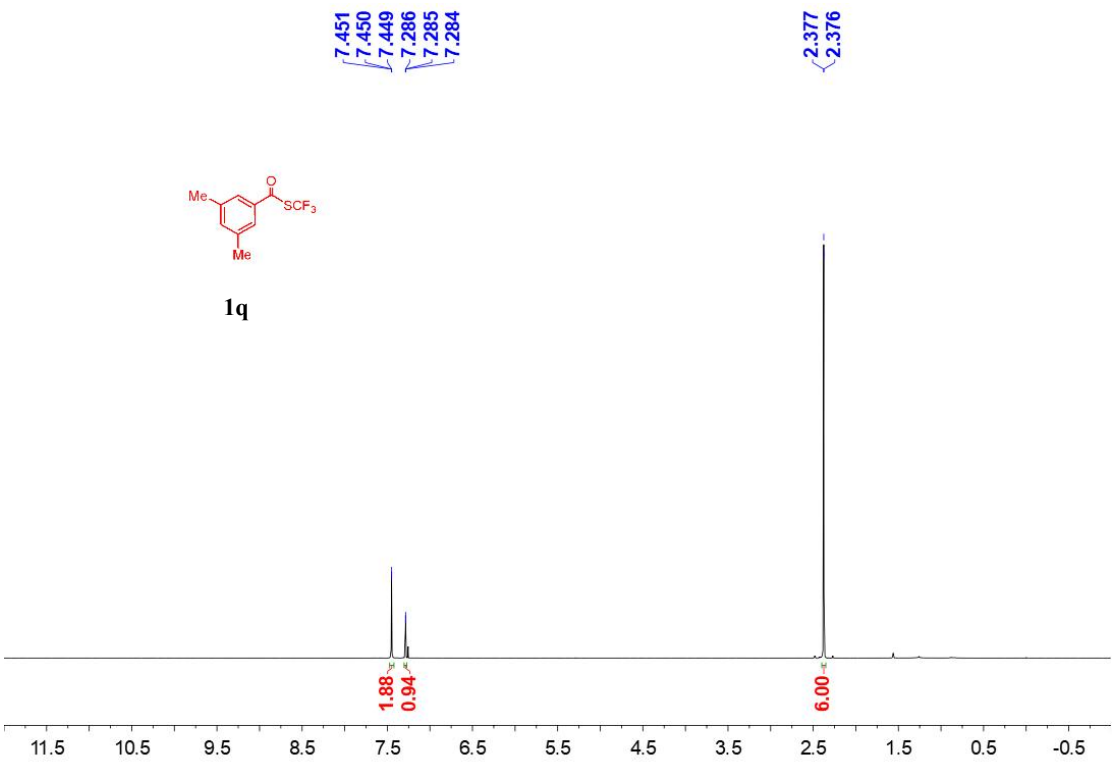
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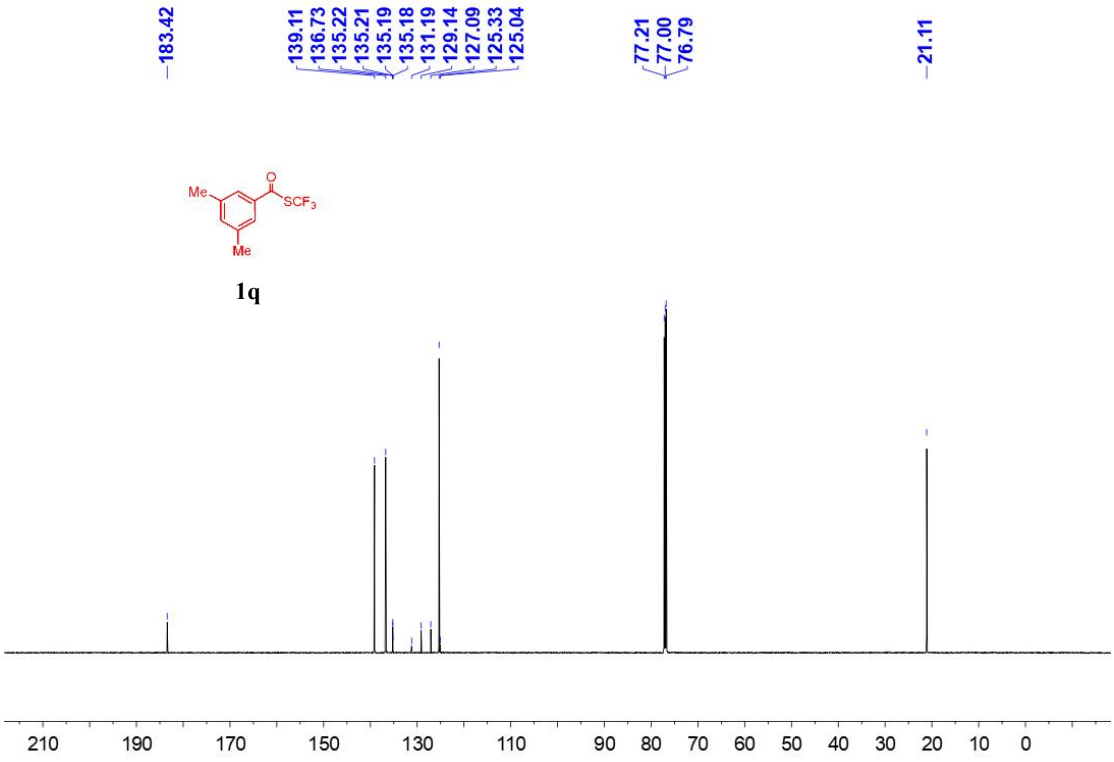
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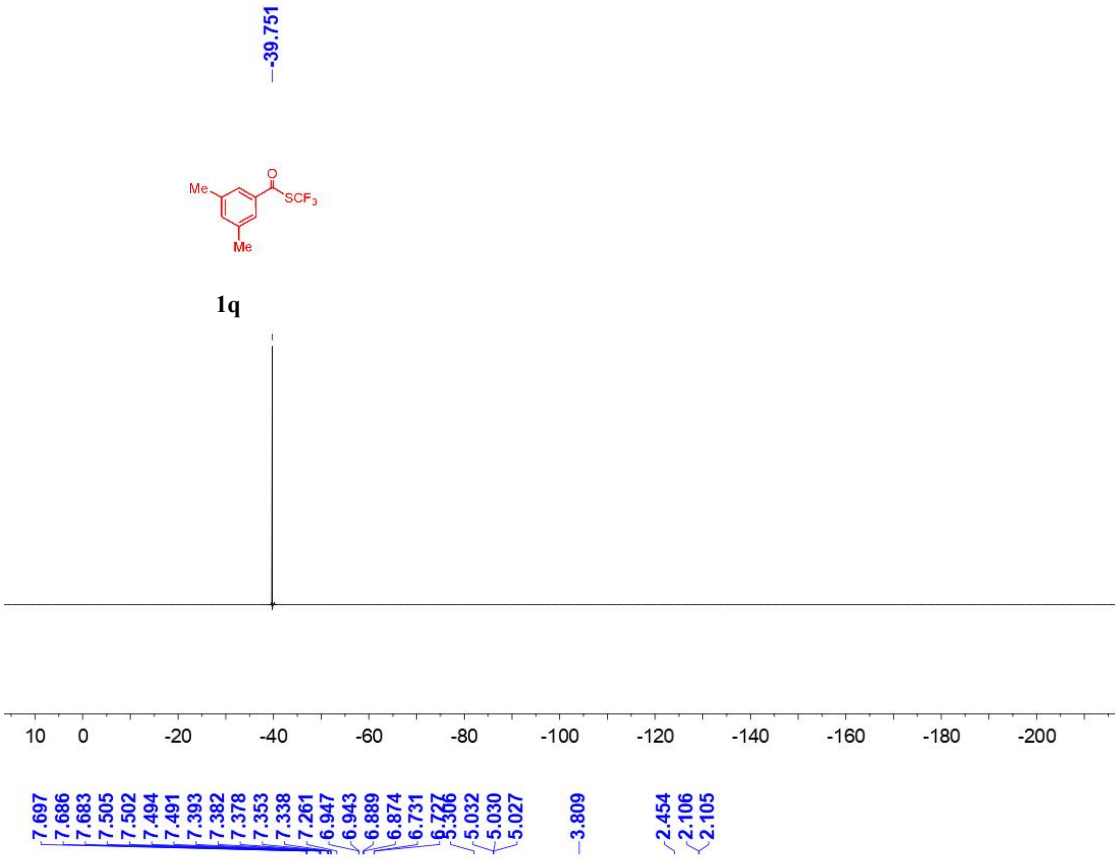
1q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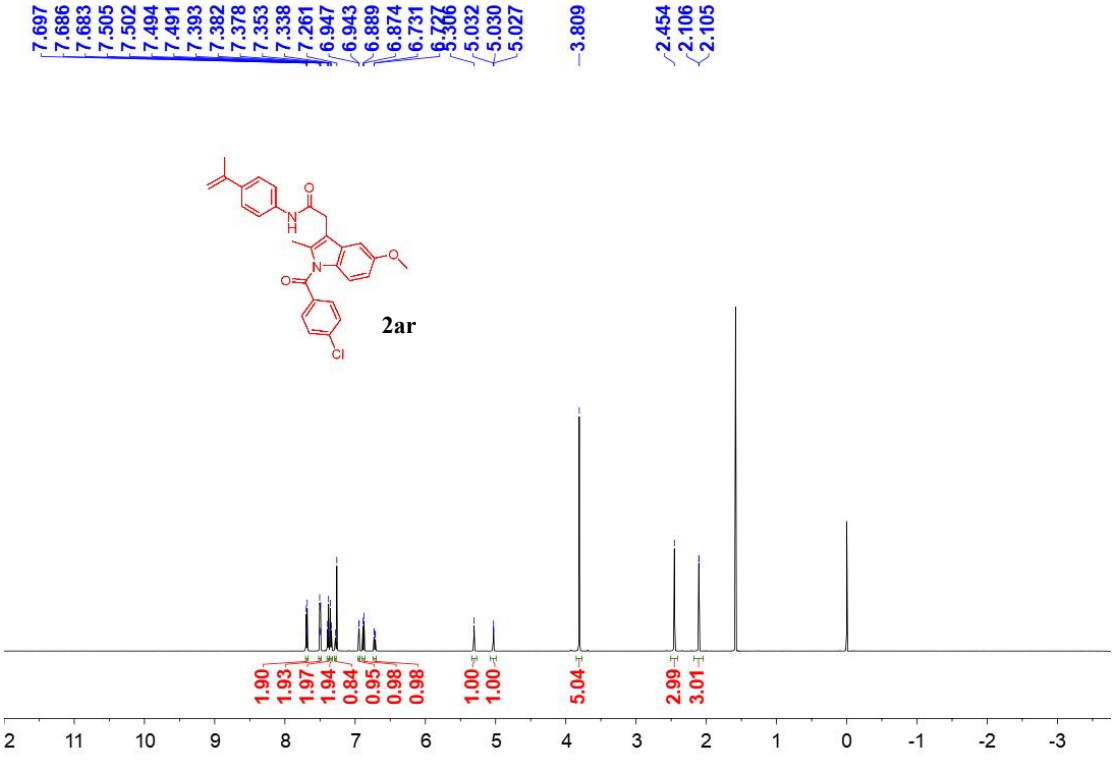
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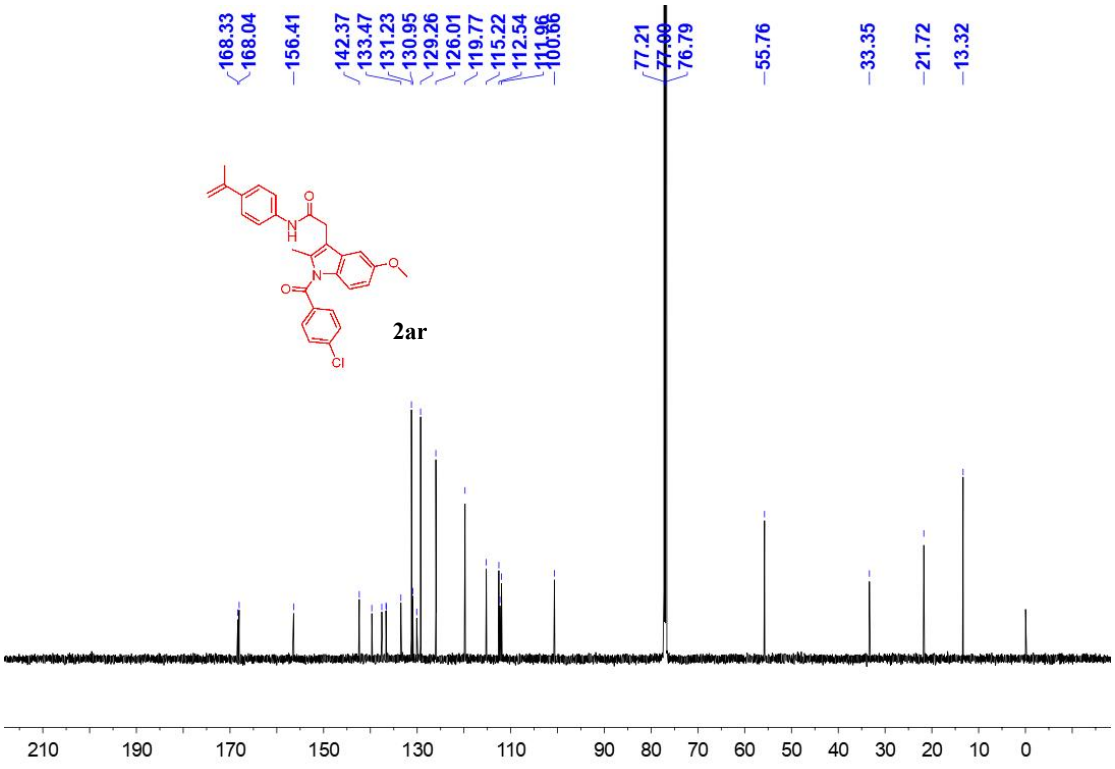
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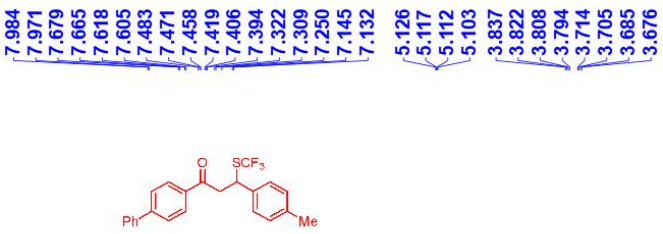
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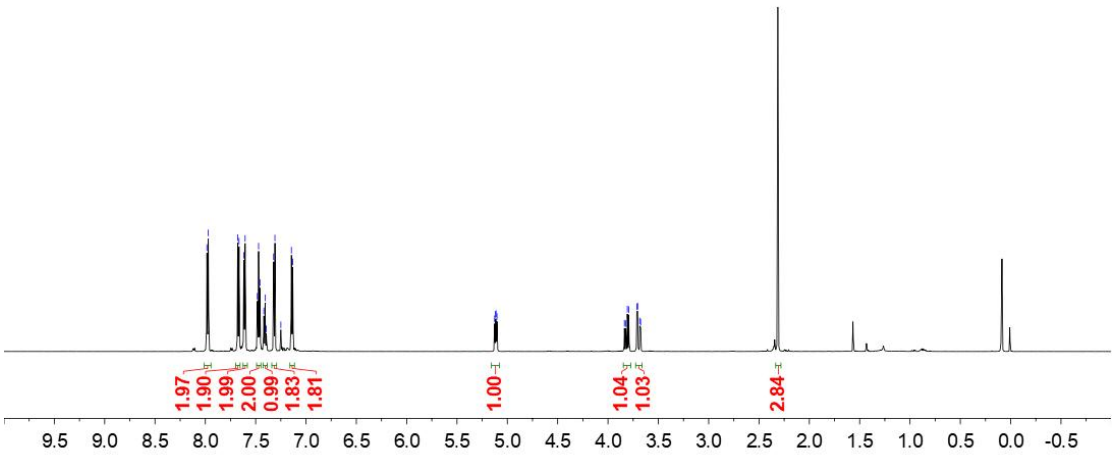
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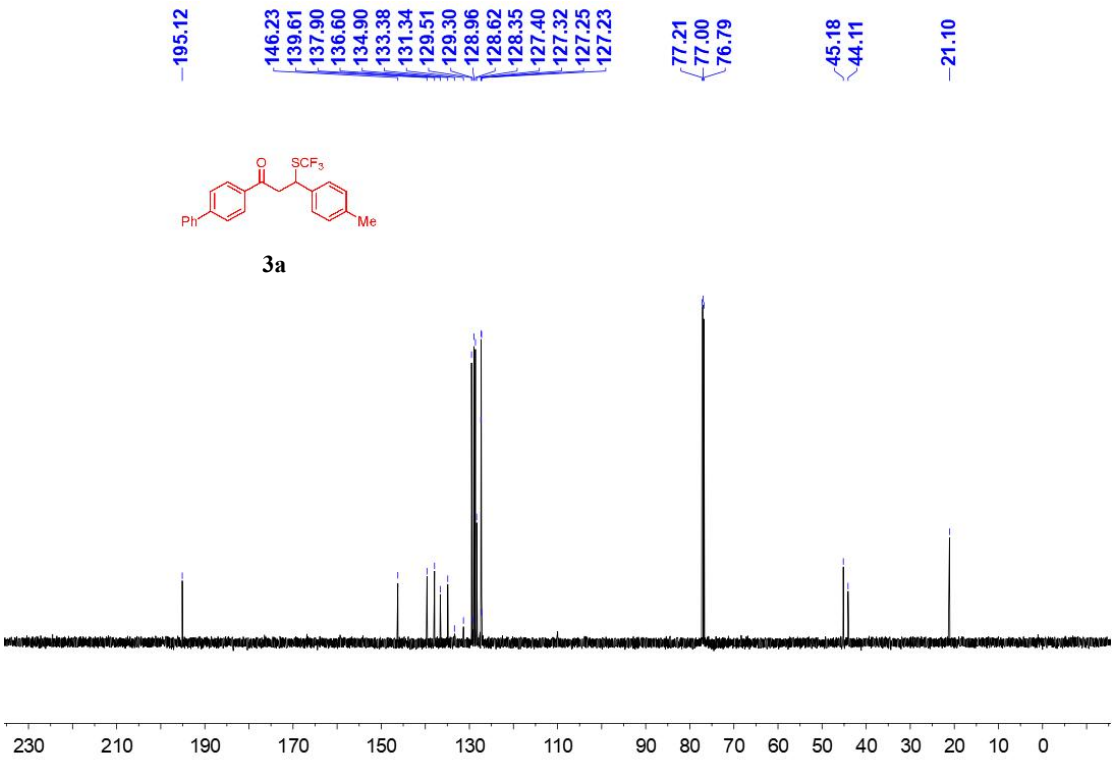
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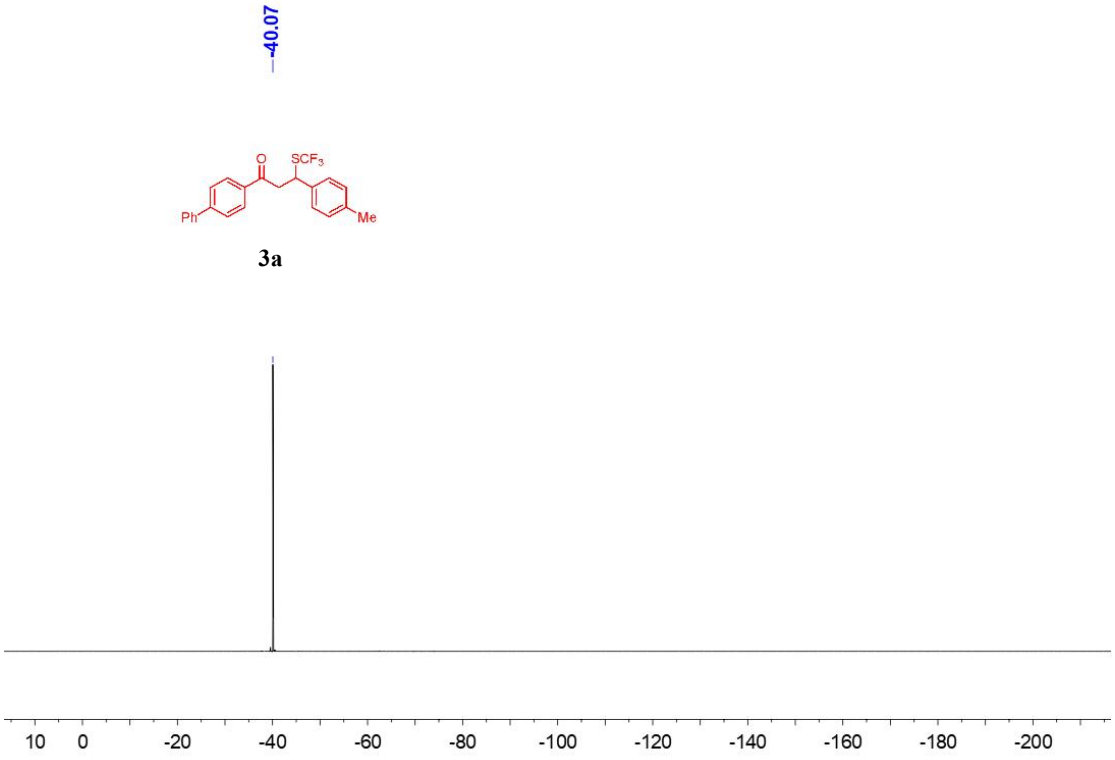
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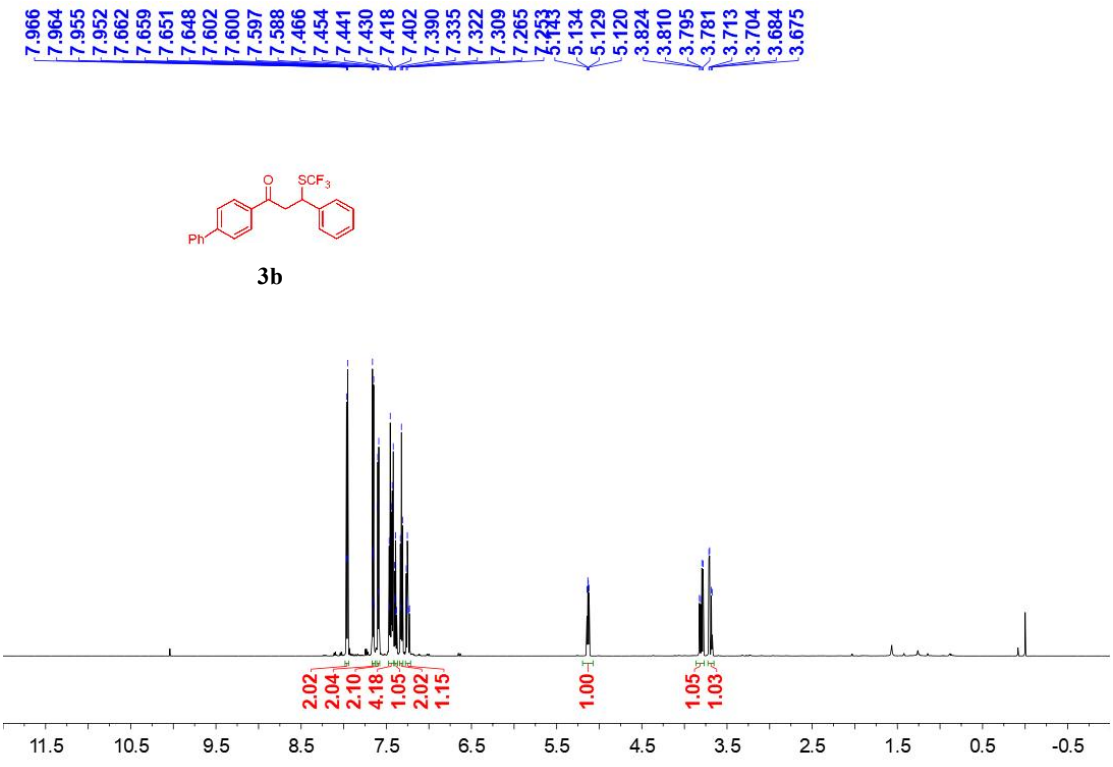
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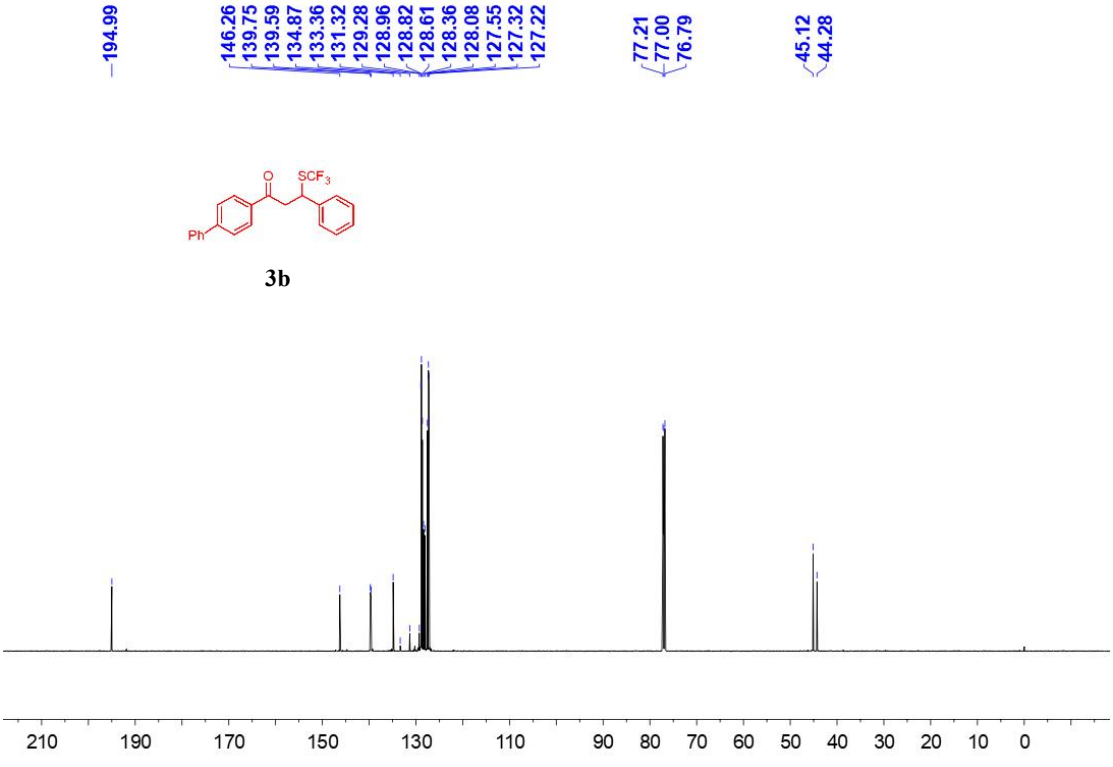
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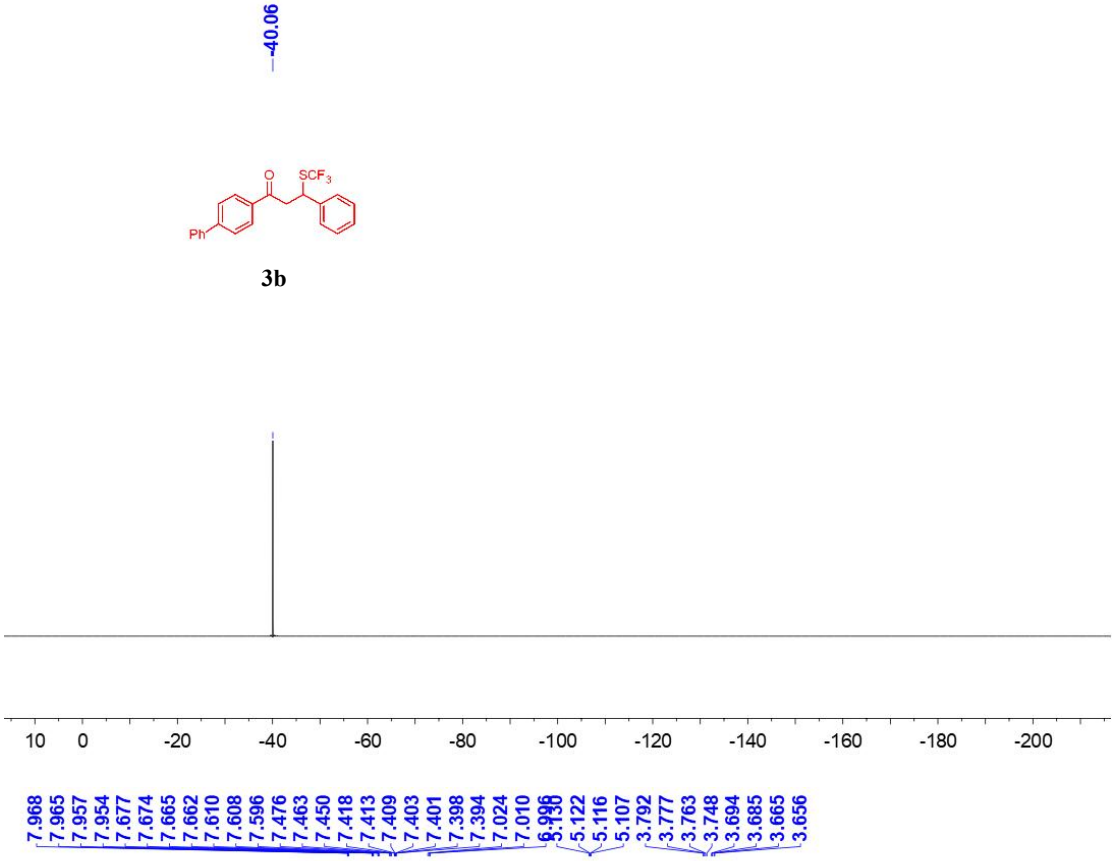
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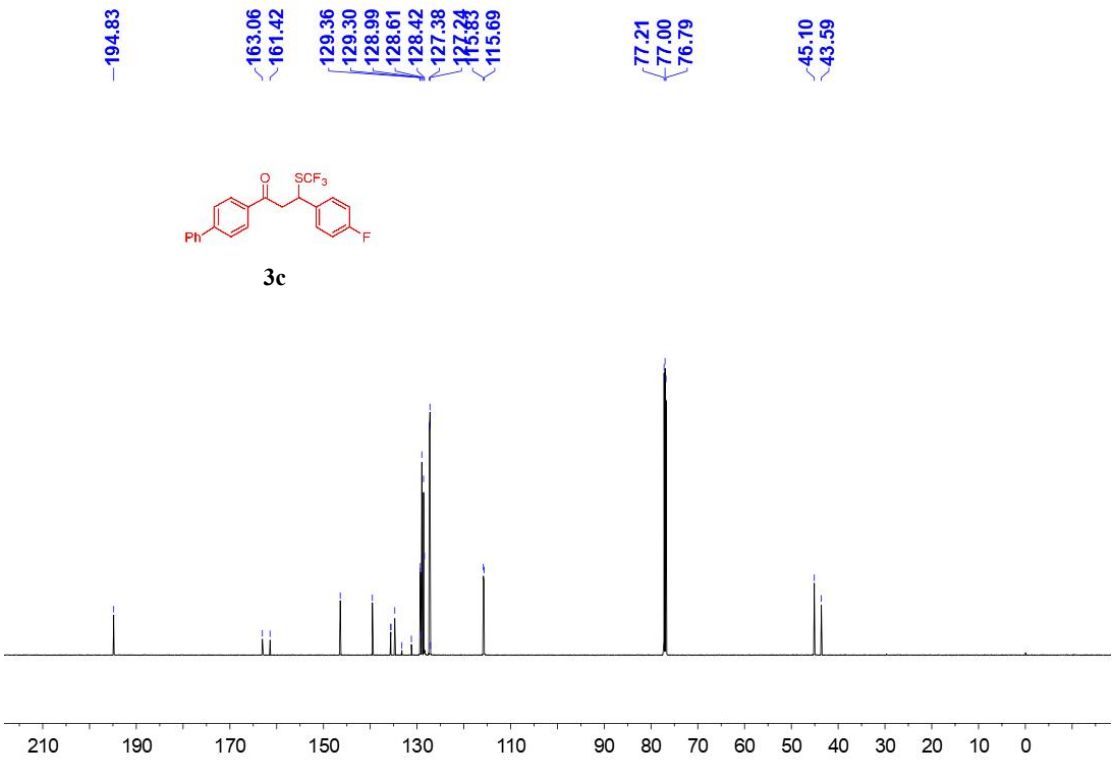
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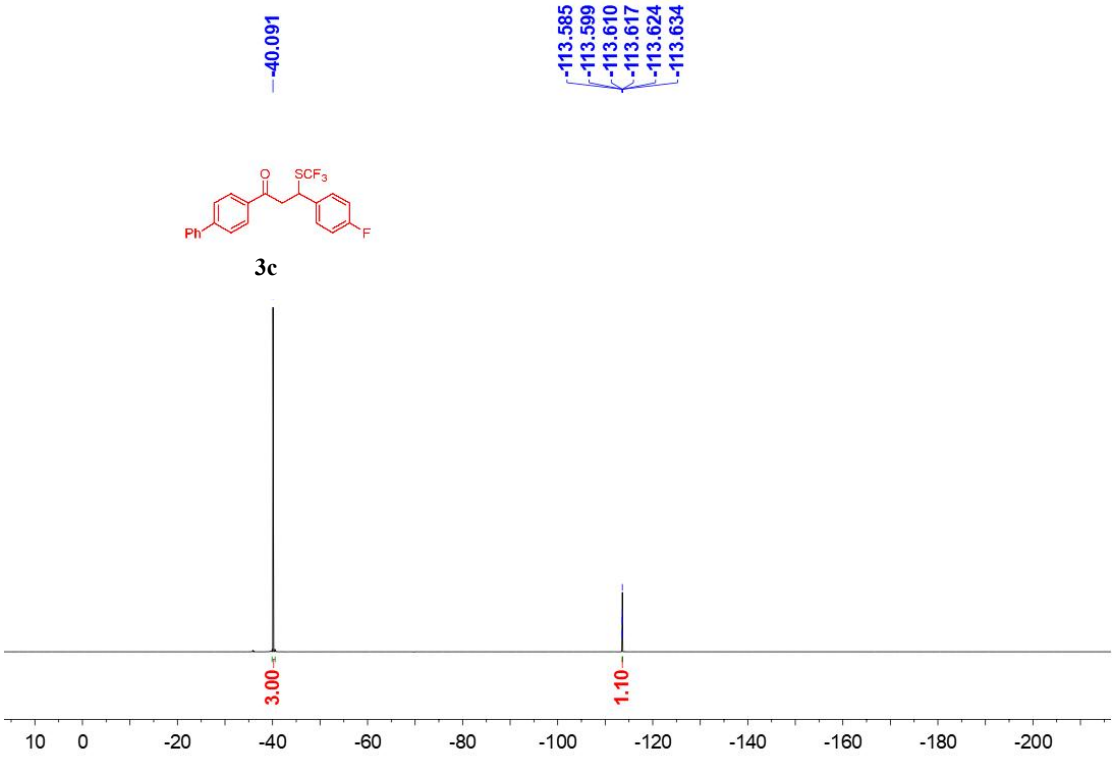
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3c


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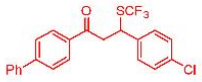
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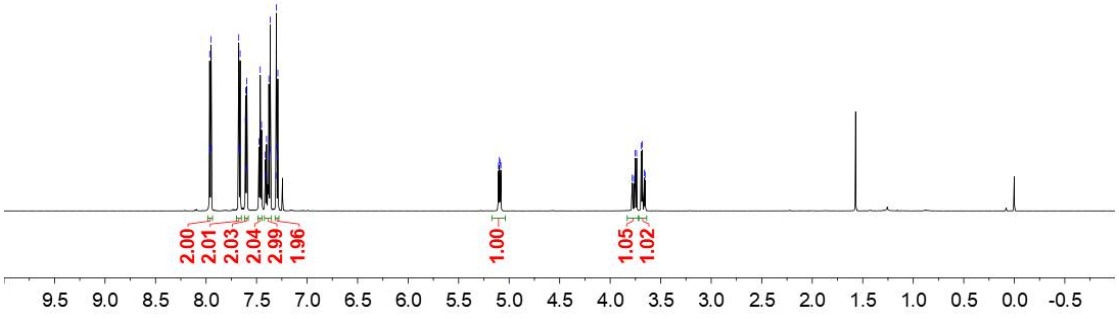
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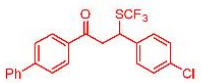
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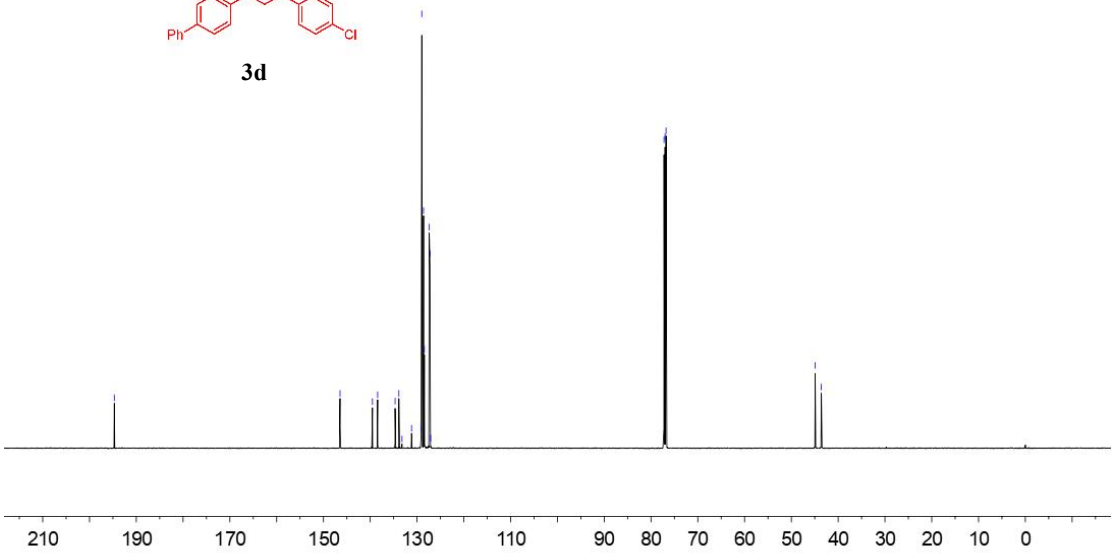
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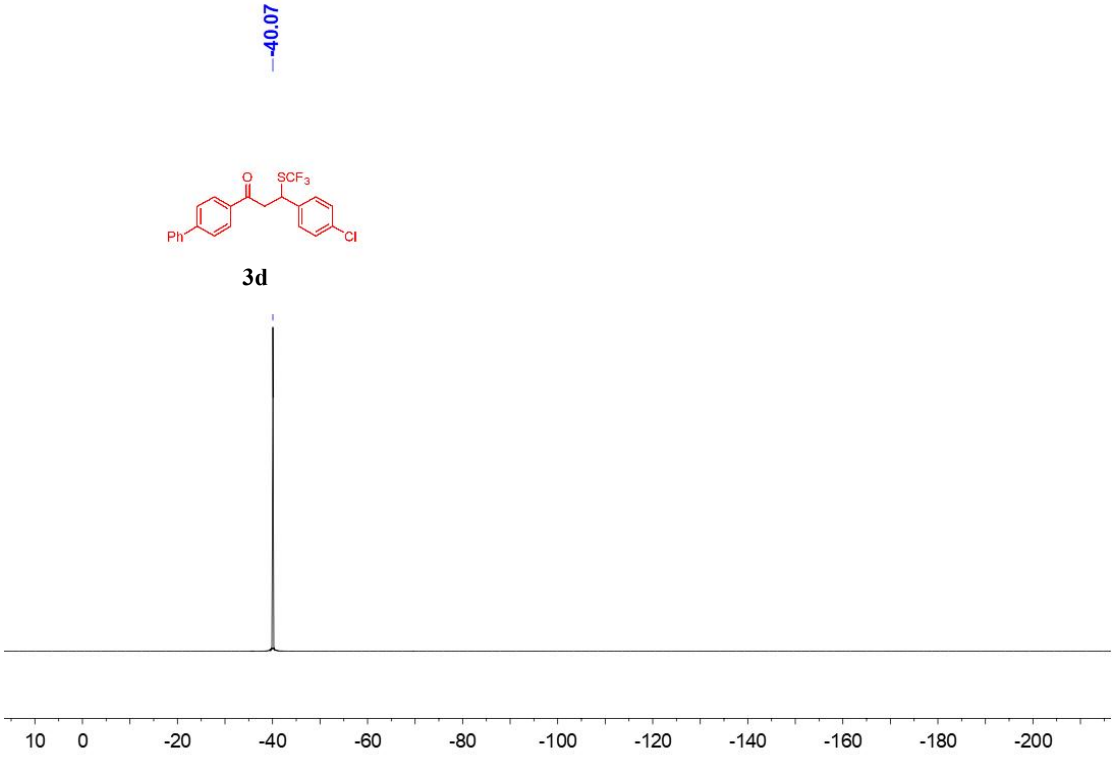
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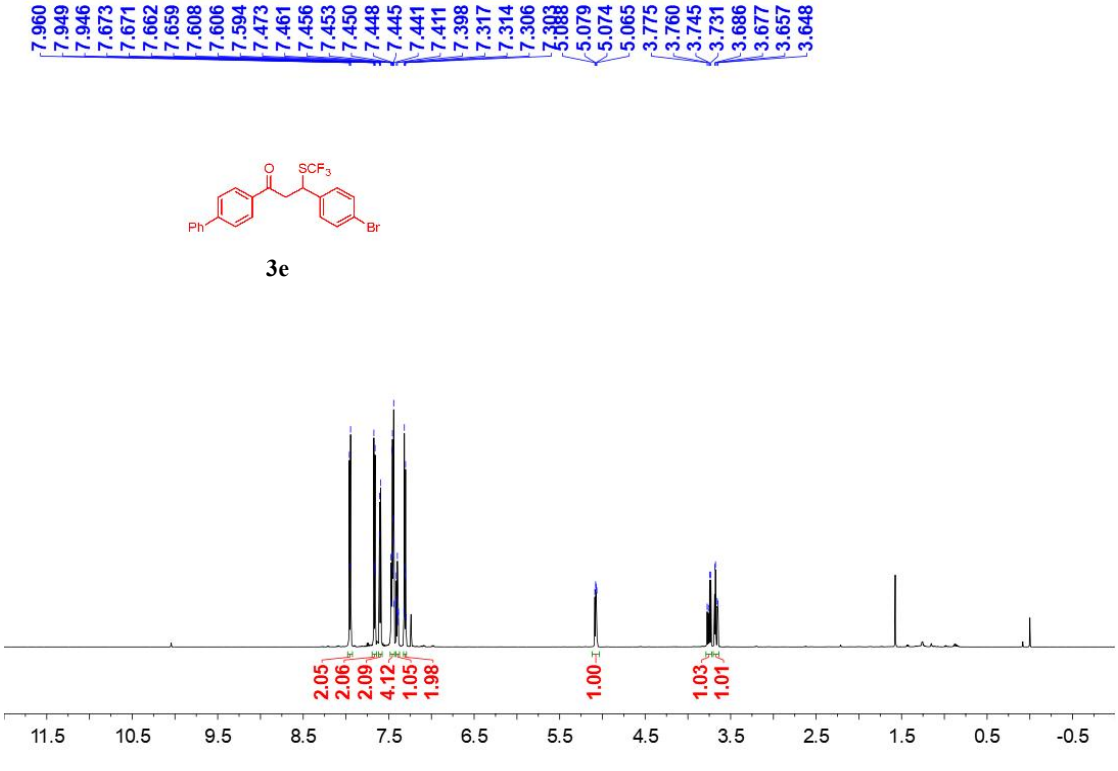
3d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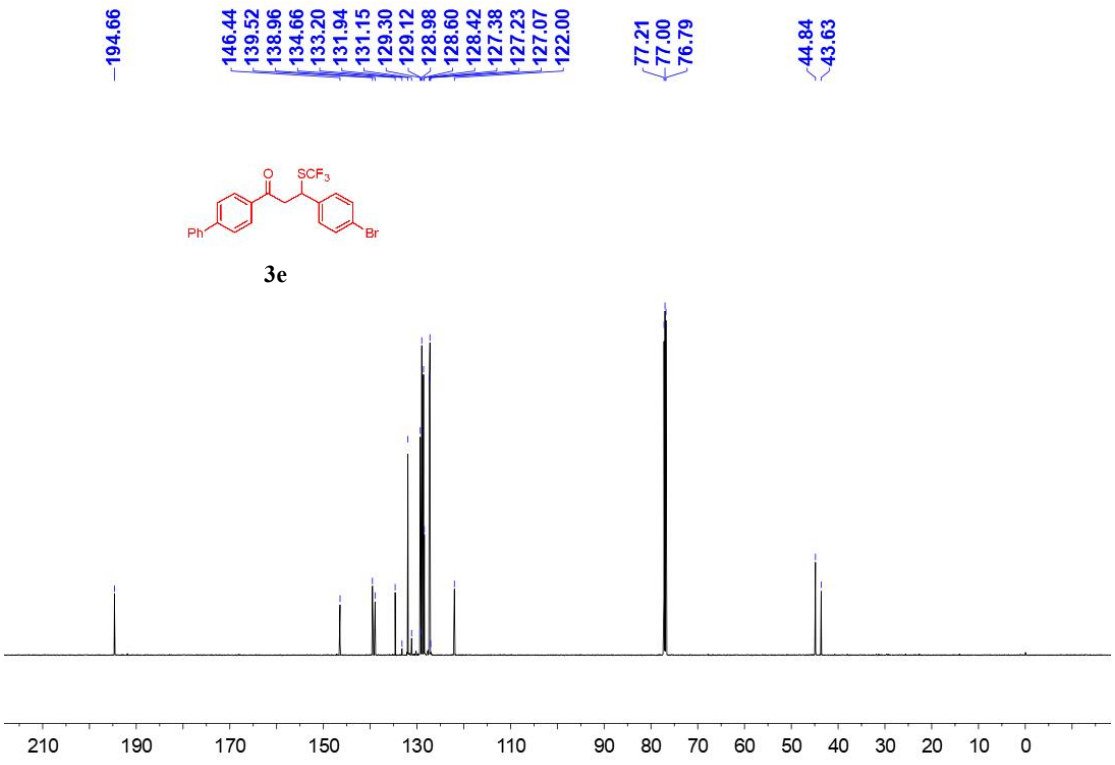
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O
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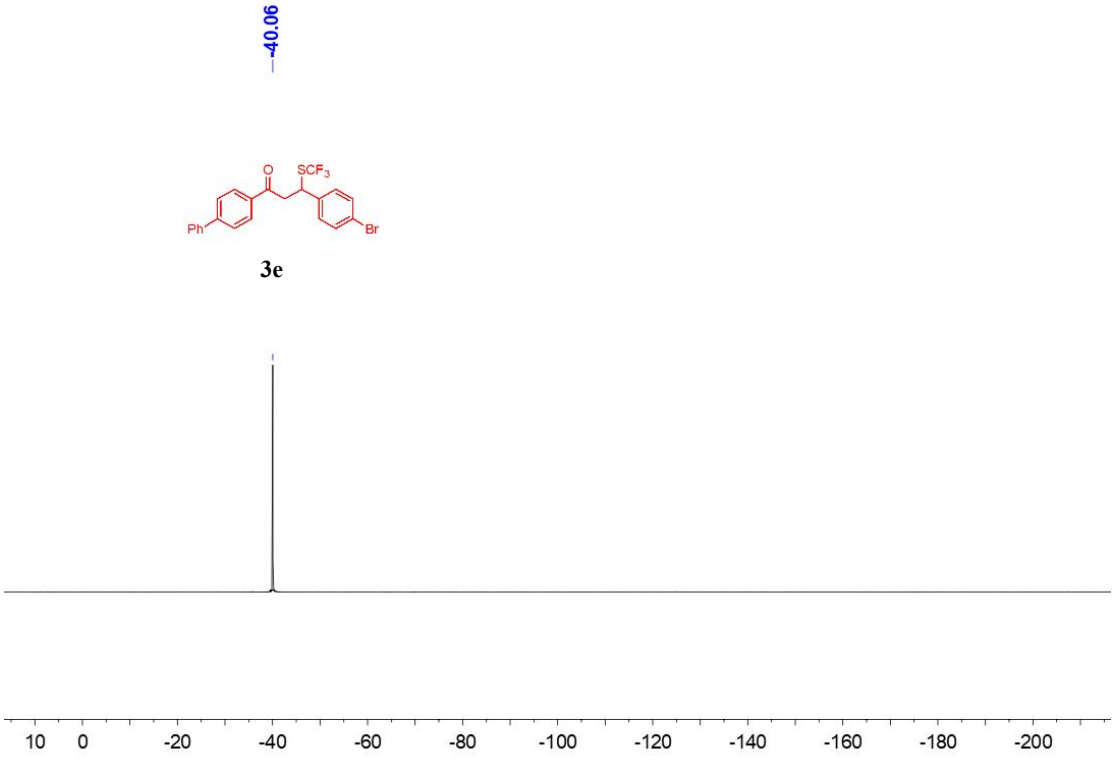
3e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-200

3e


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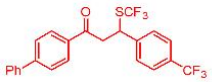
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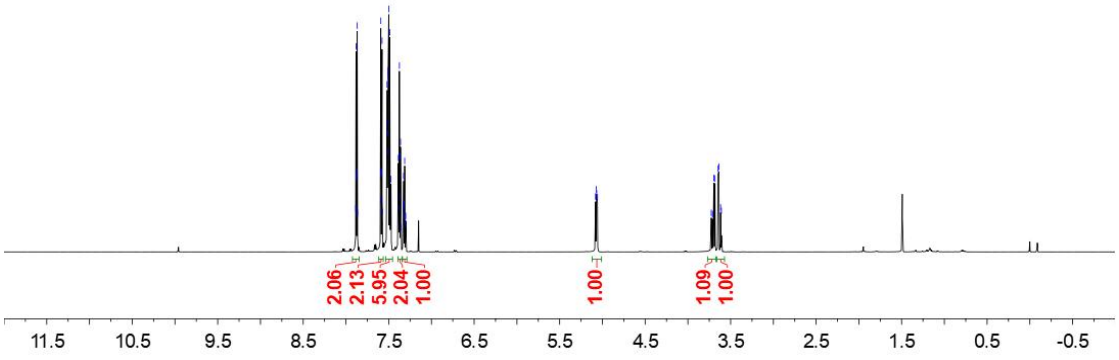
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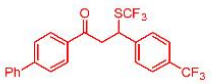
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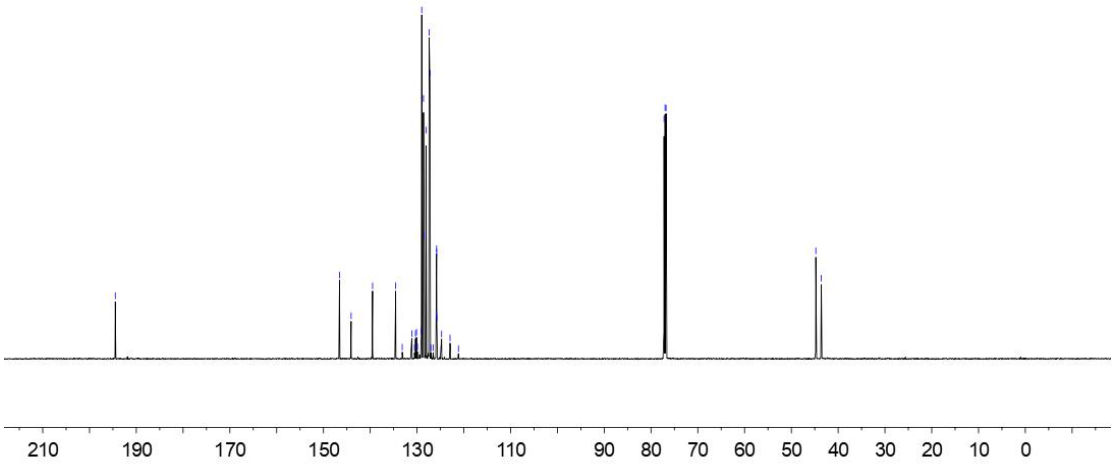
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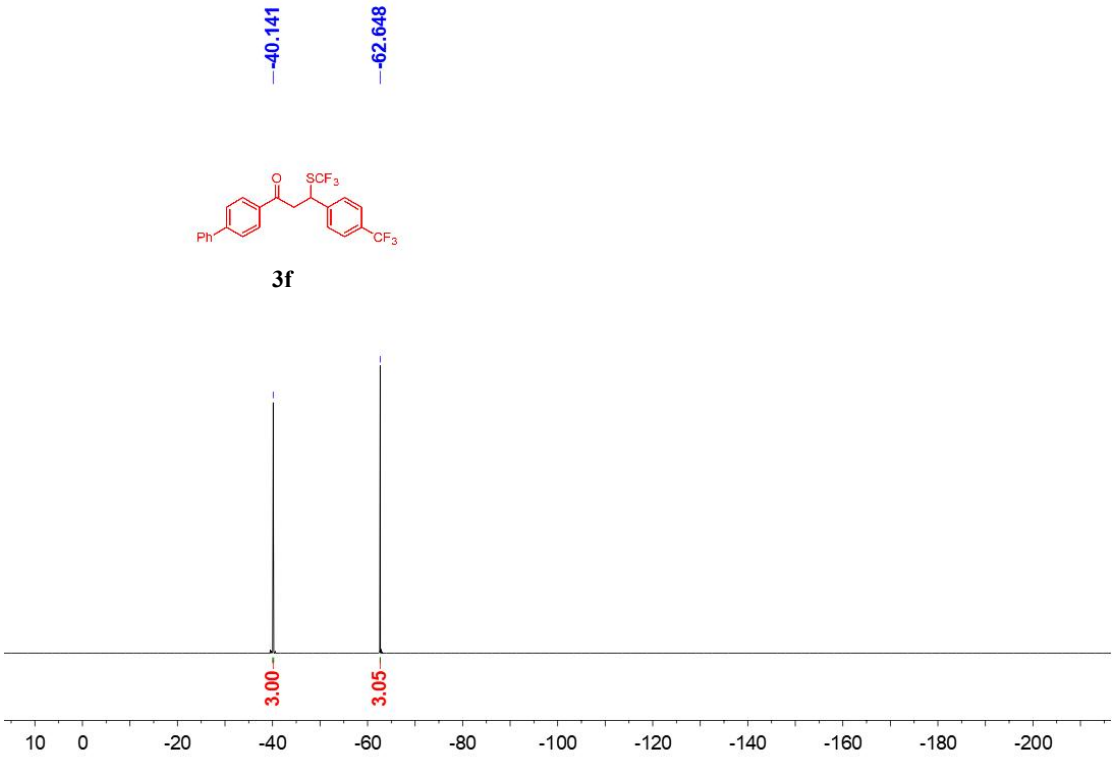
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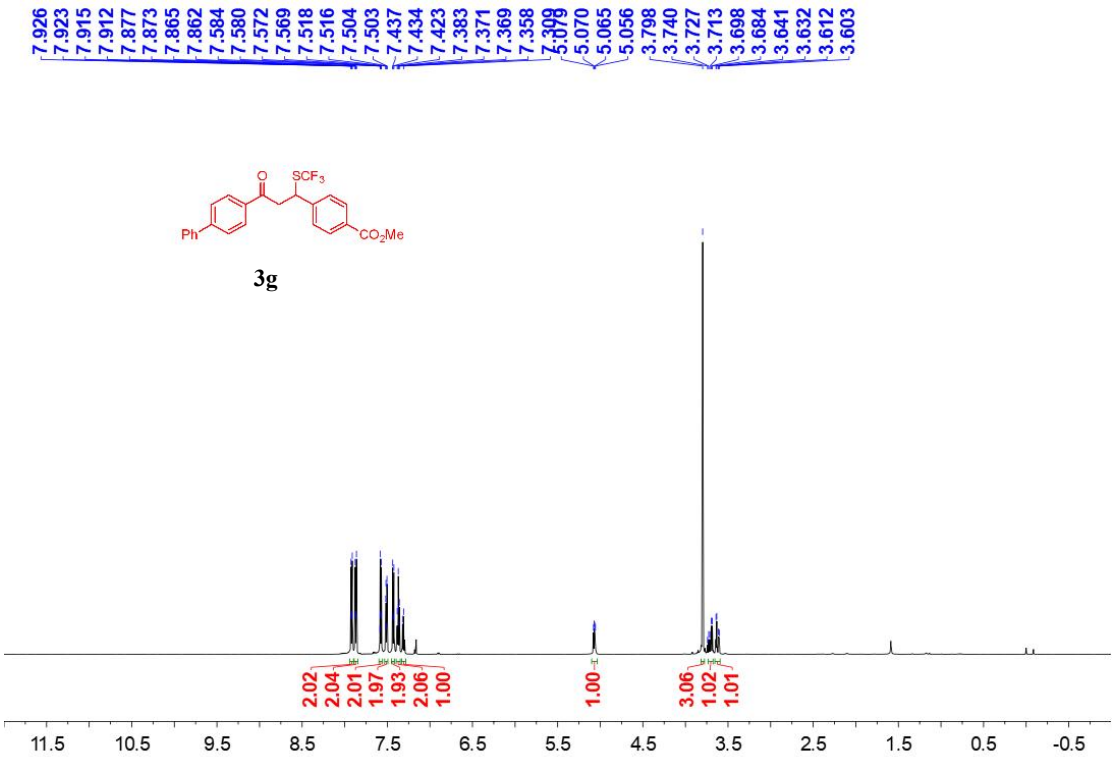
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3.603
O
SCF3
Ph
CO2Me
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2.06
1.00
1.00
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1.02
1.01
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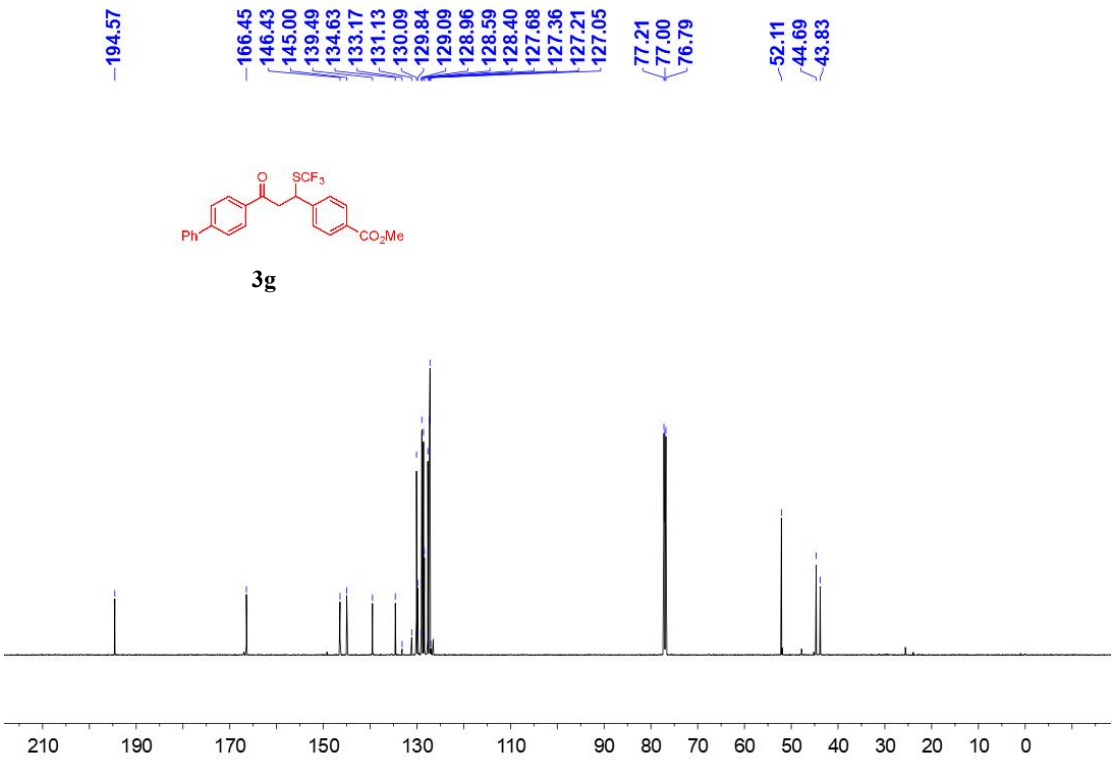
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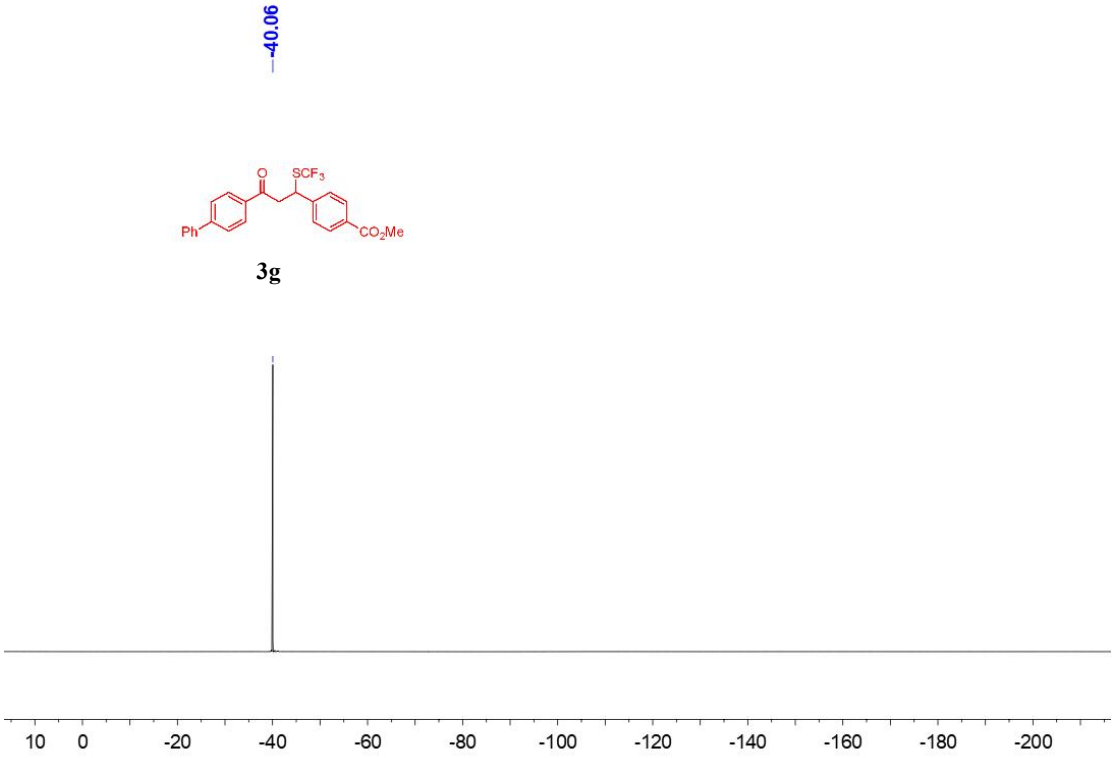
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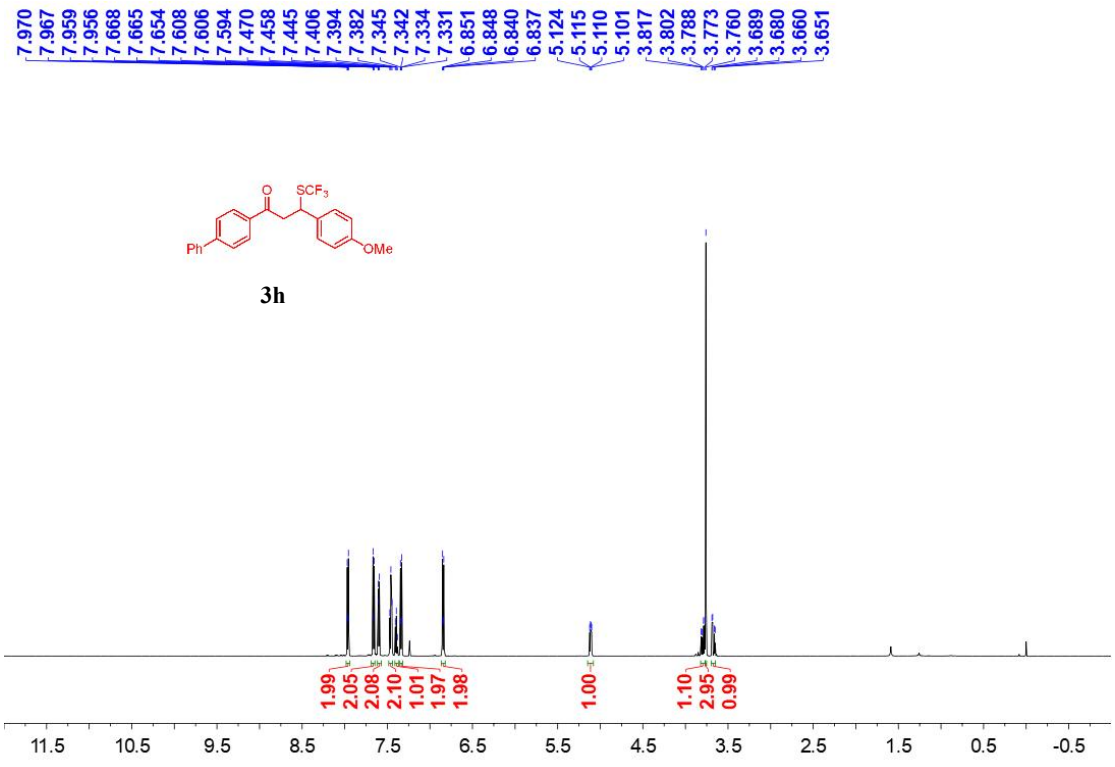
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3.651
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SCF3
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1.00
1.10
2.95
0.99
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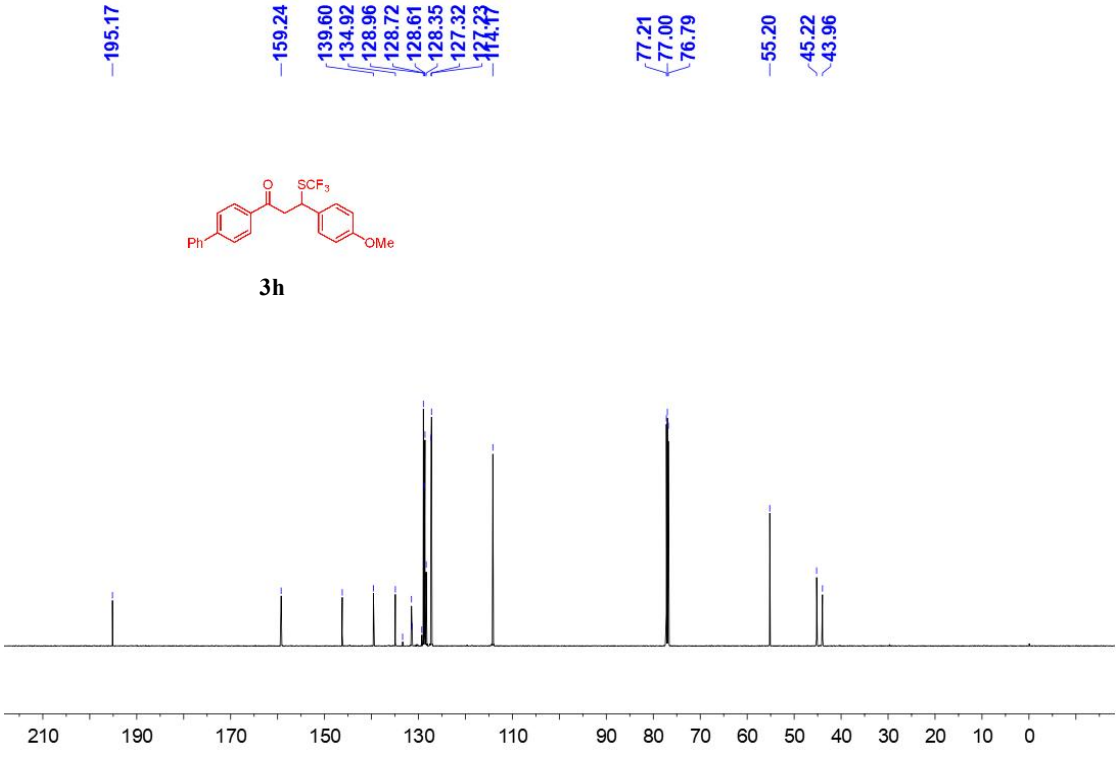
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SCF3
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OMe
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20
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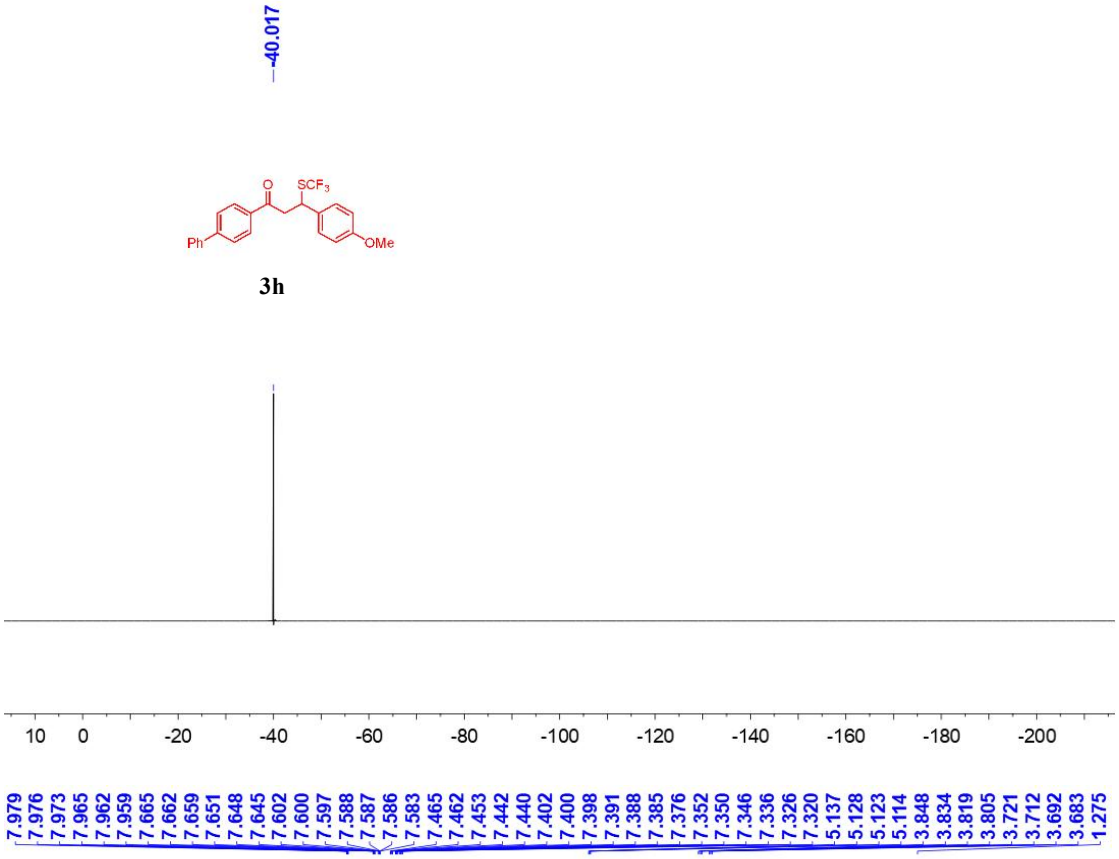
3h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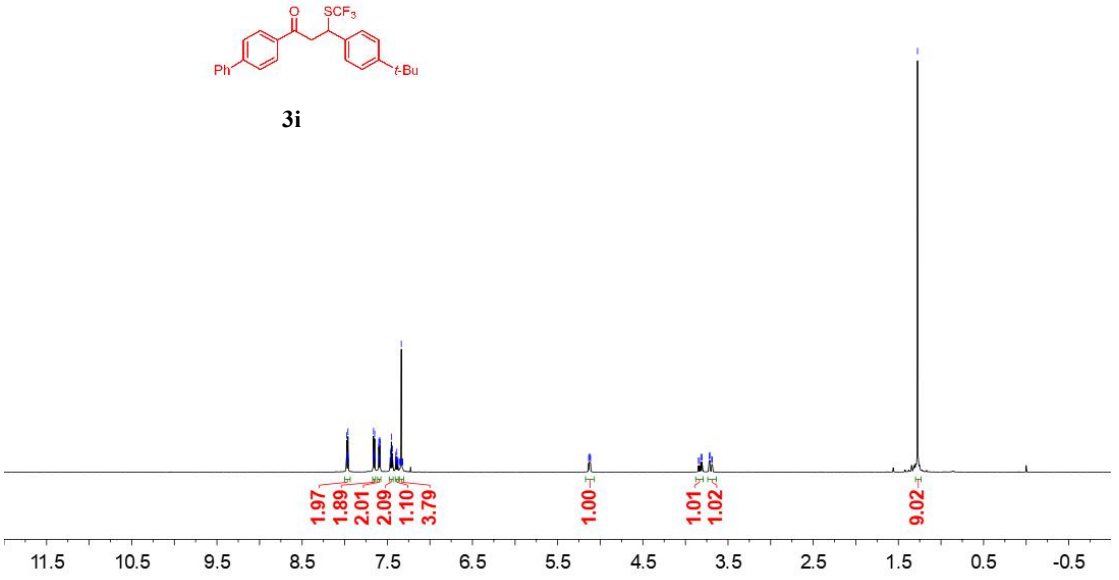
3h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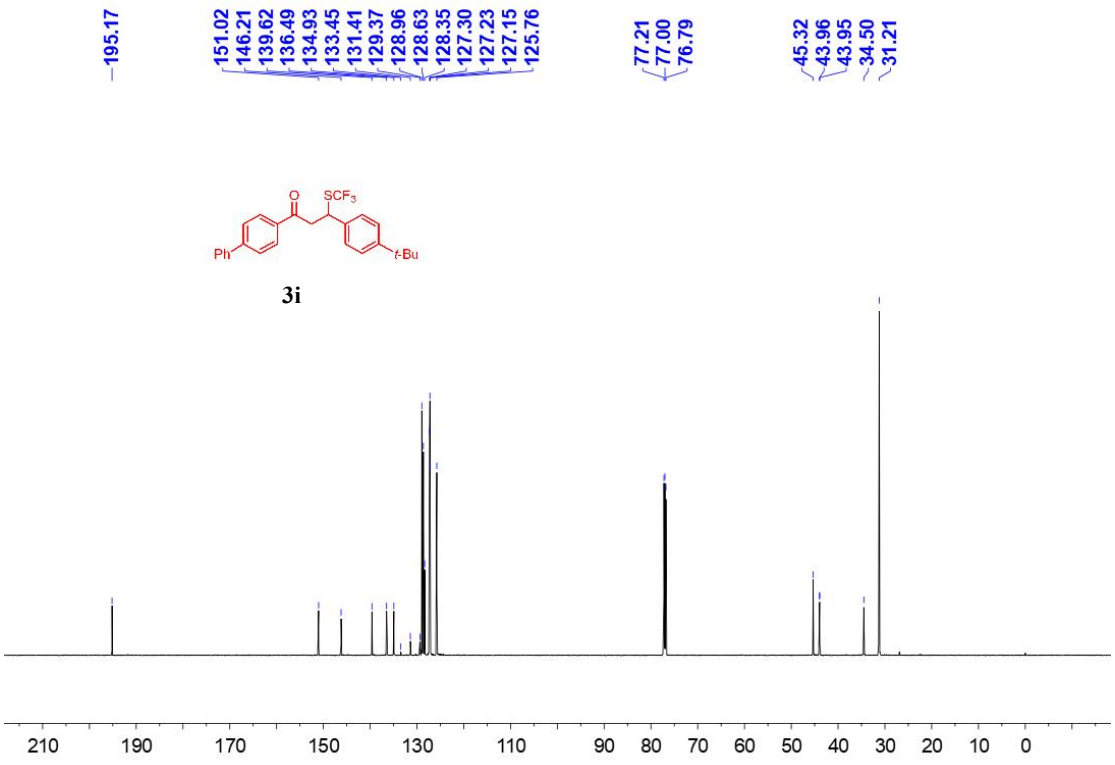
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SCF3
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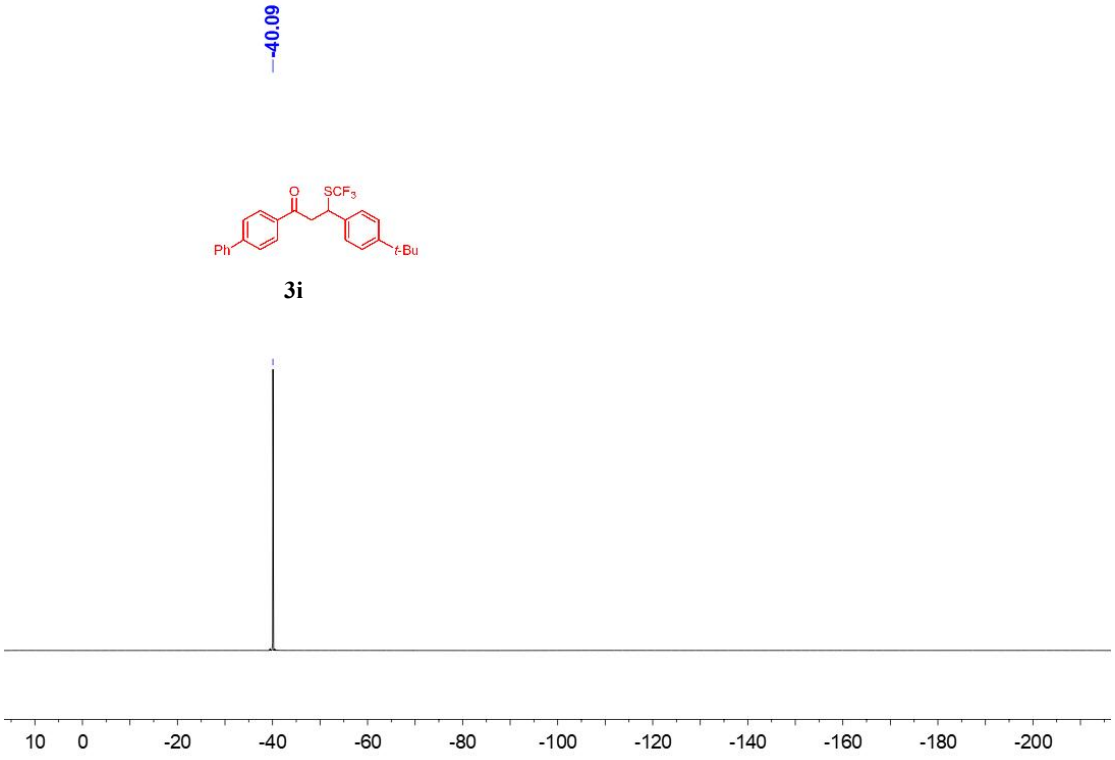
0

3i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3i


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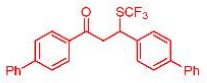
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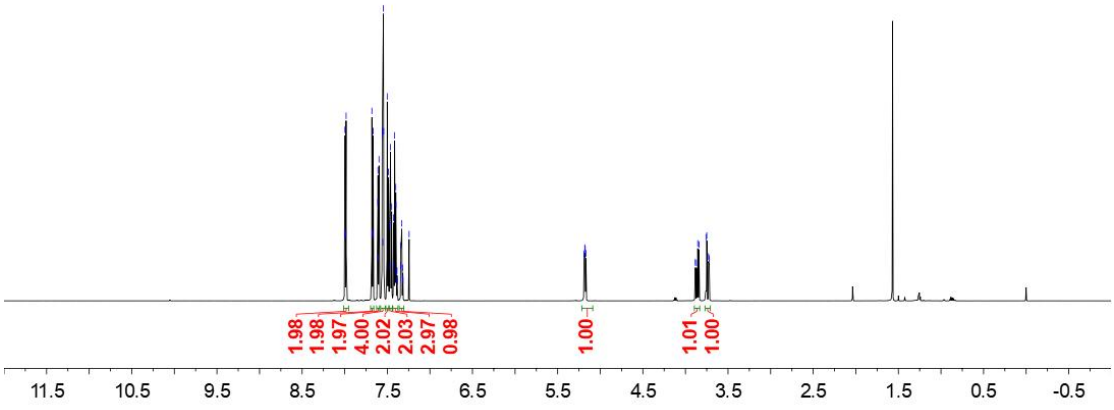
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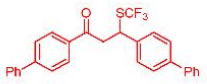
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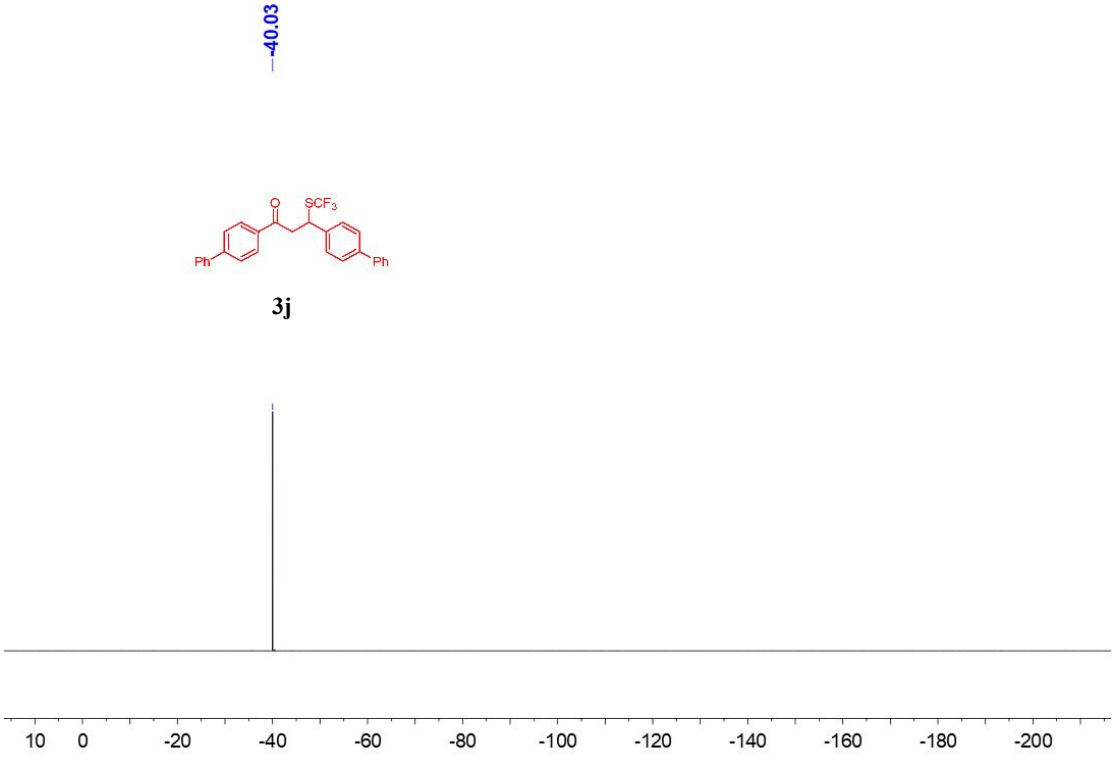
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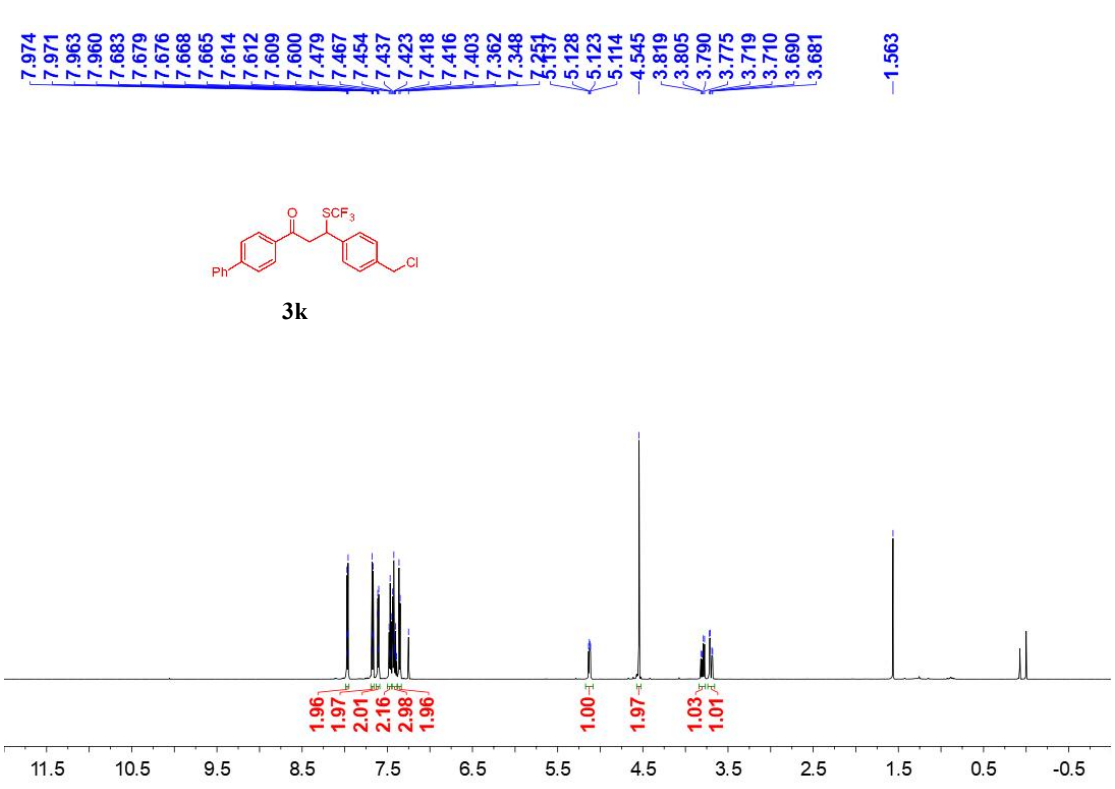
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SCF3
Ph
Cl
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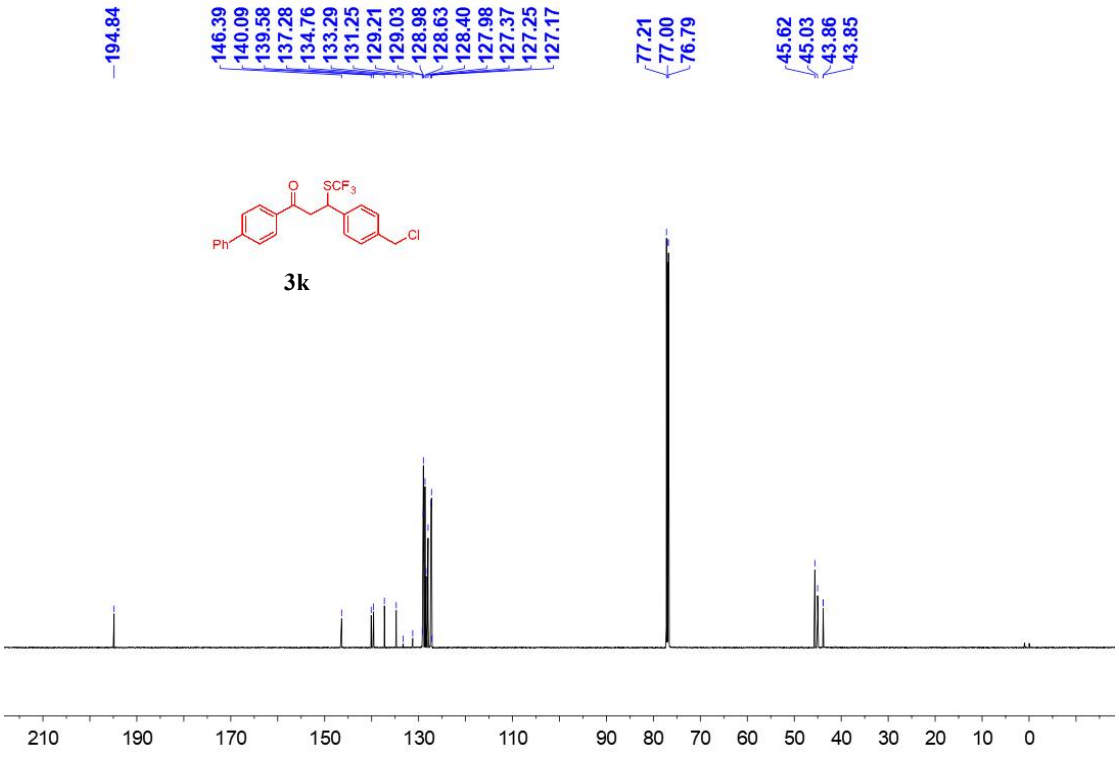
3k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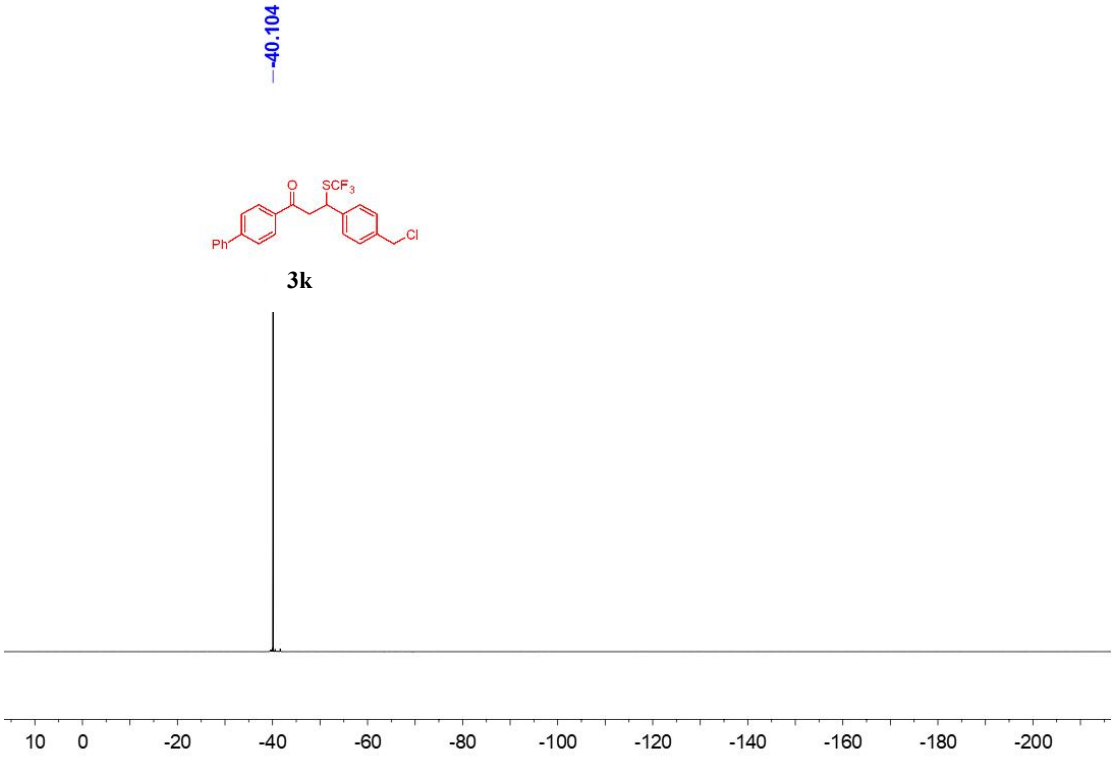
3k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3k


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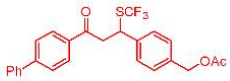
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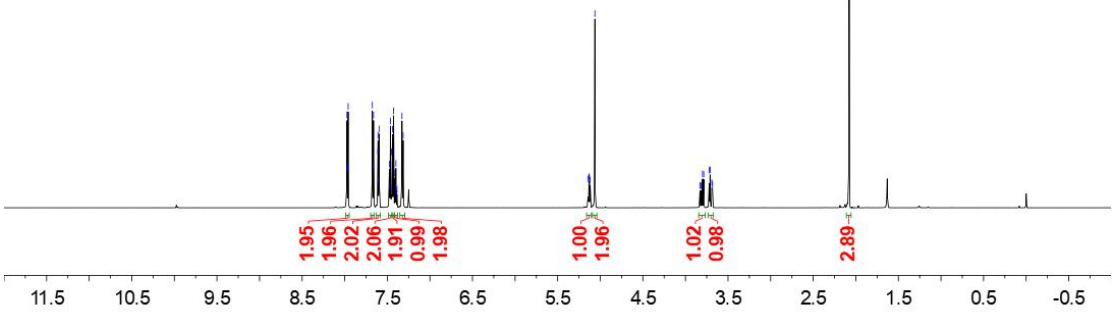
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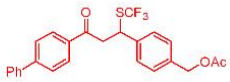
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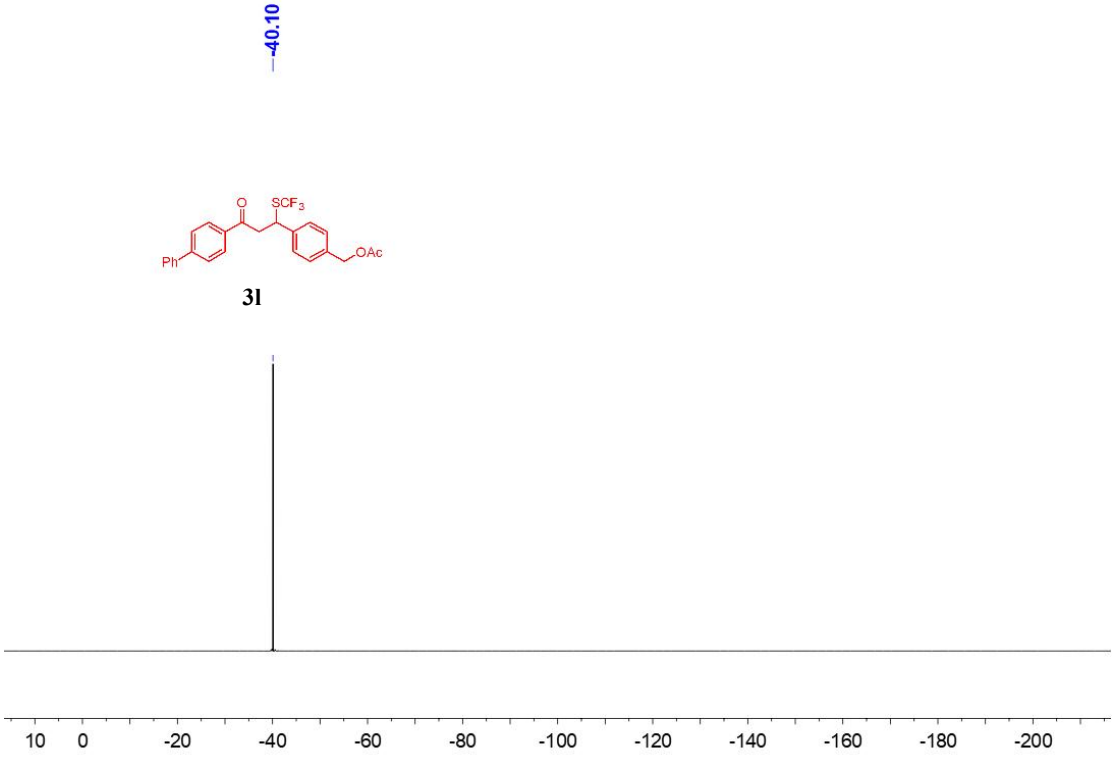
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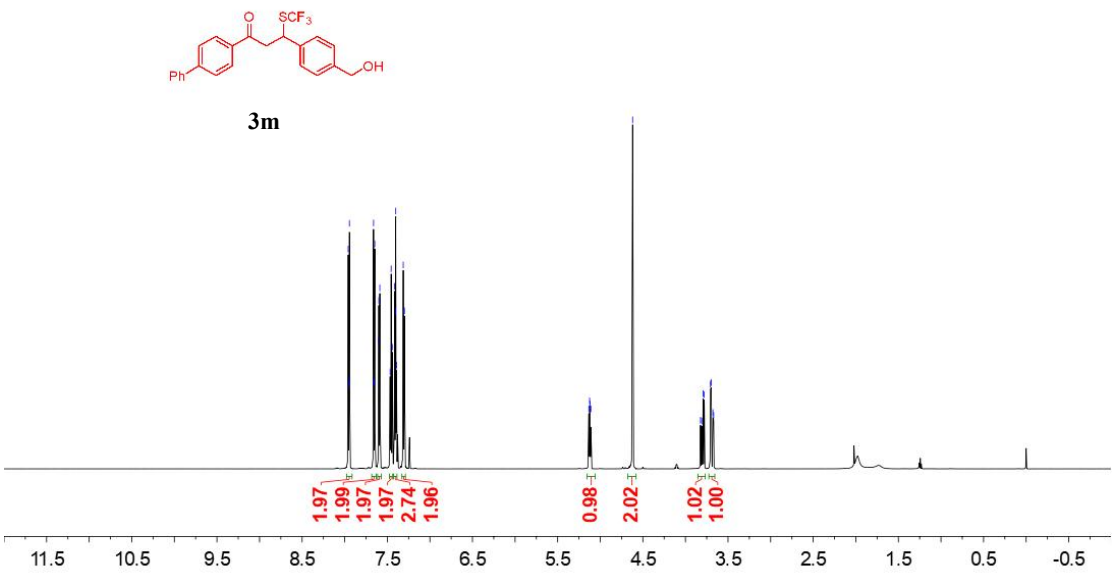
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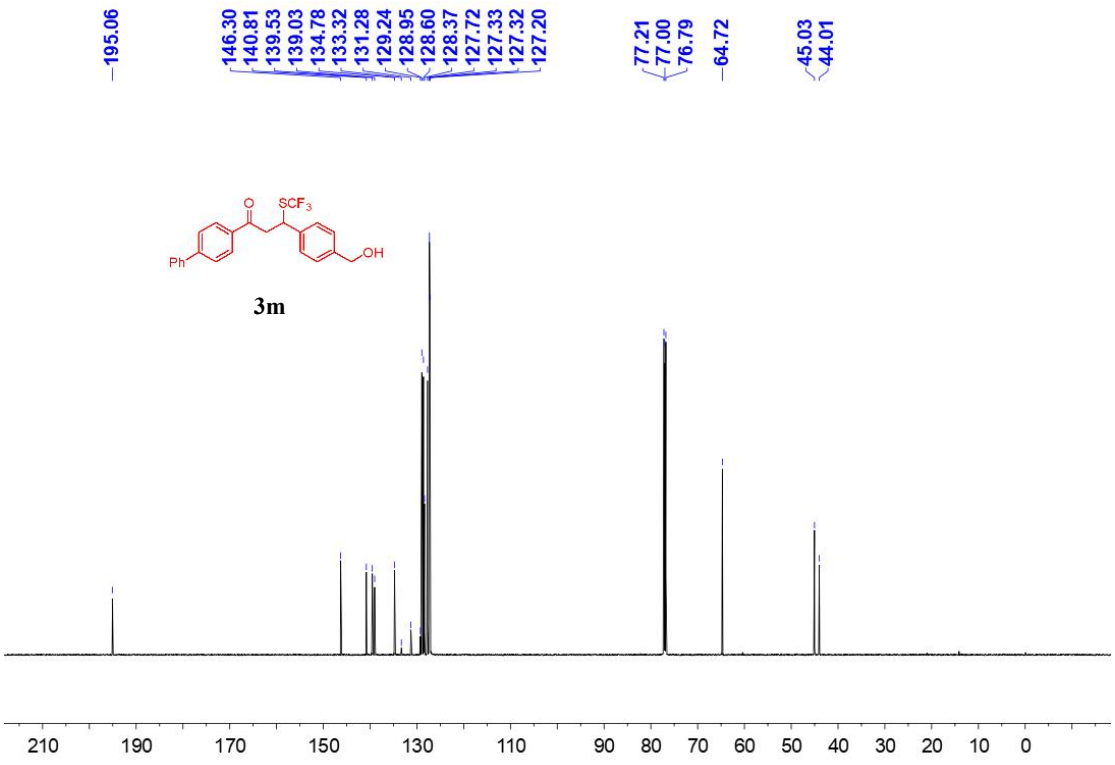
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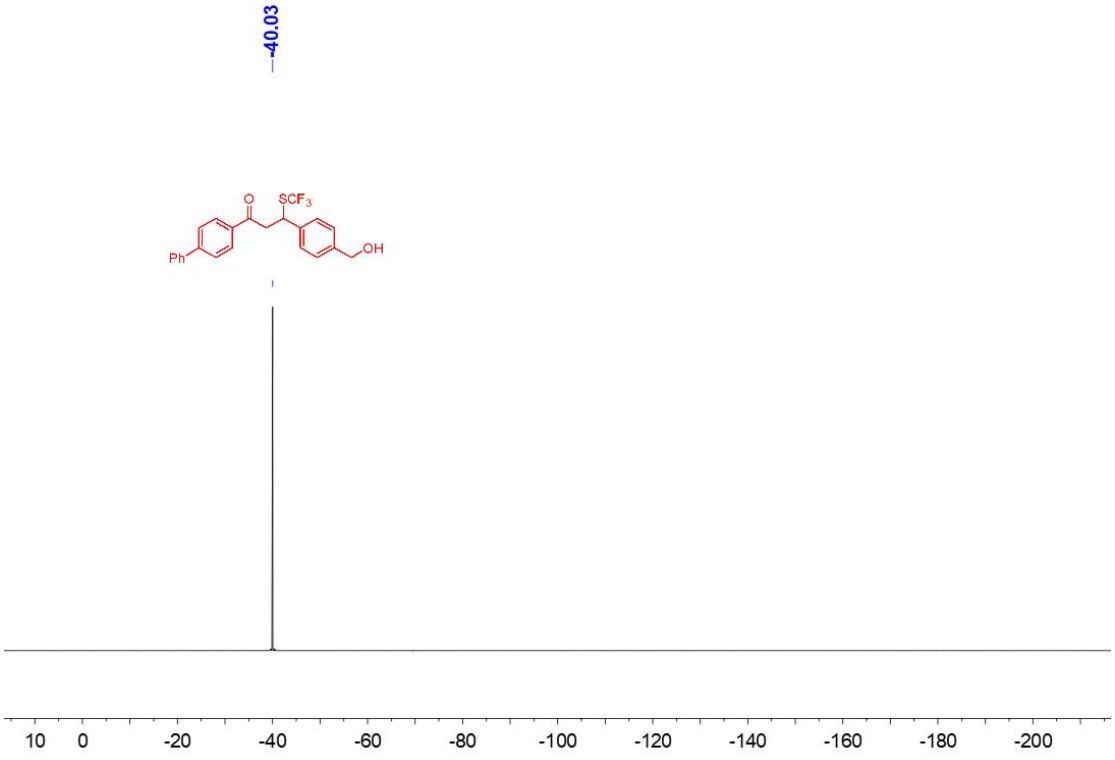
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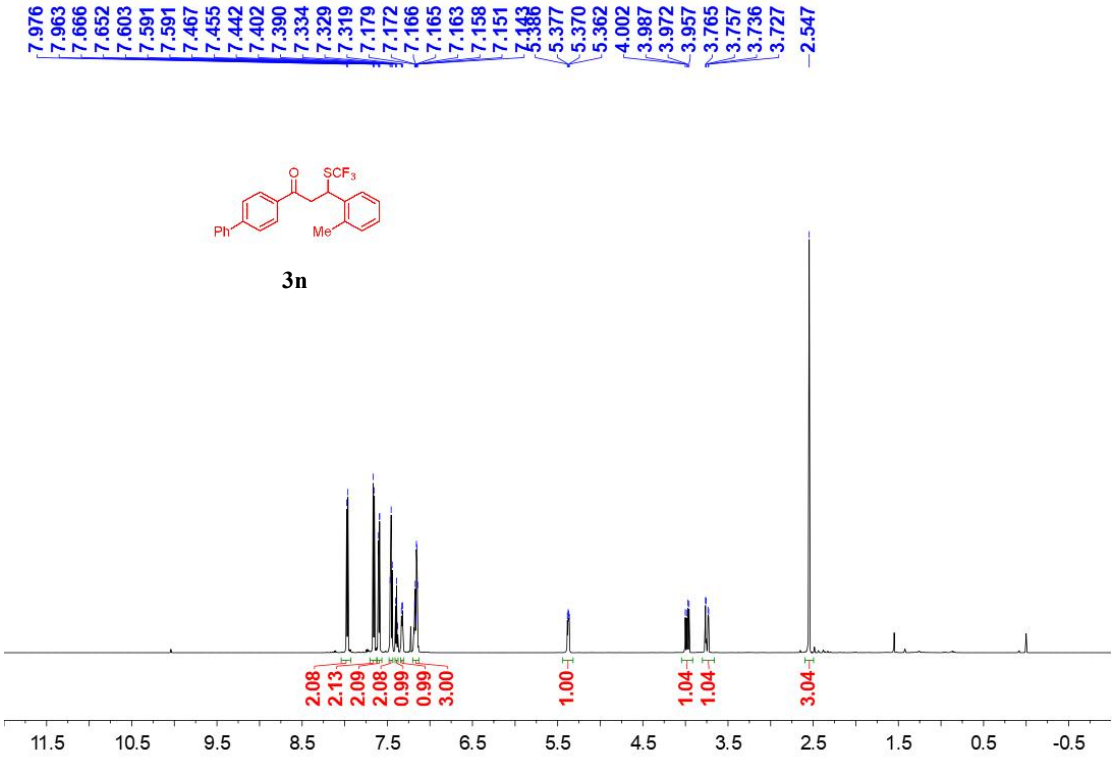
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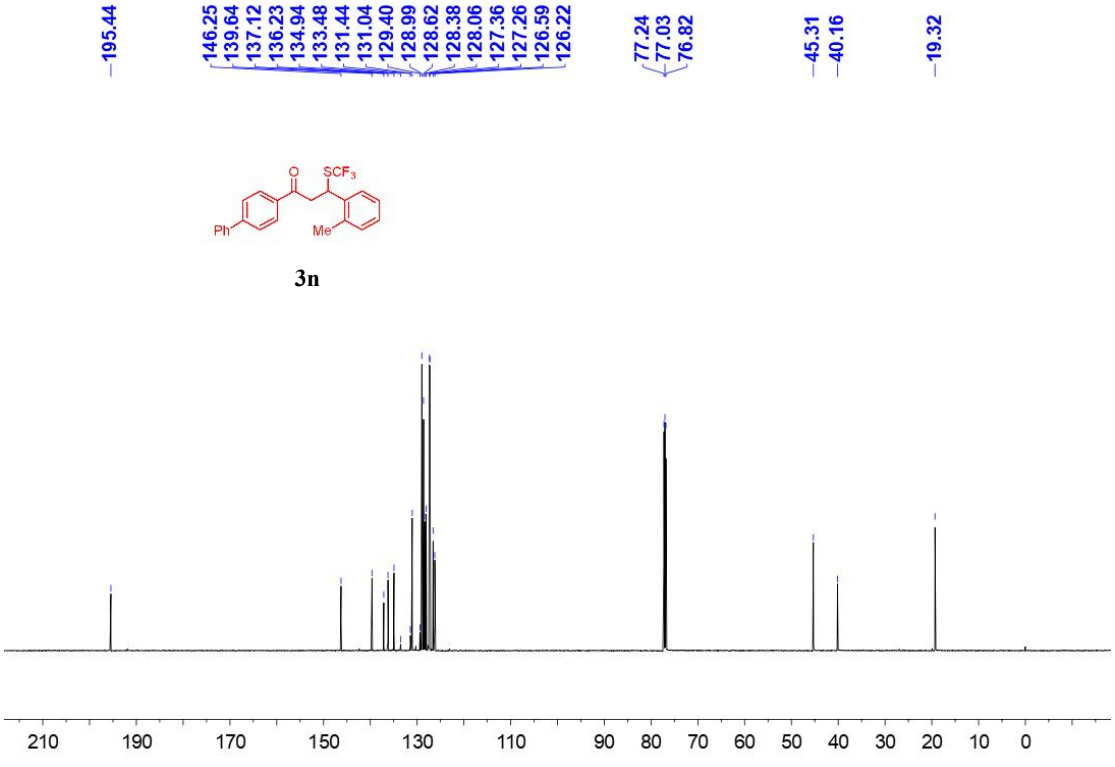
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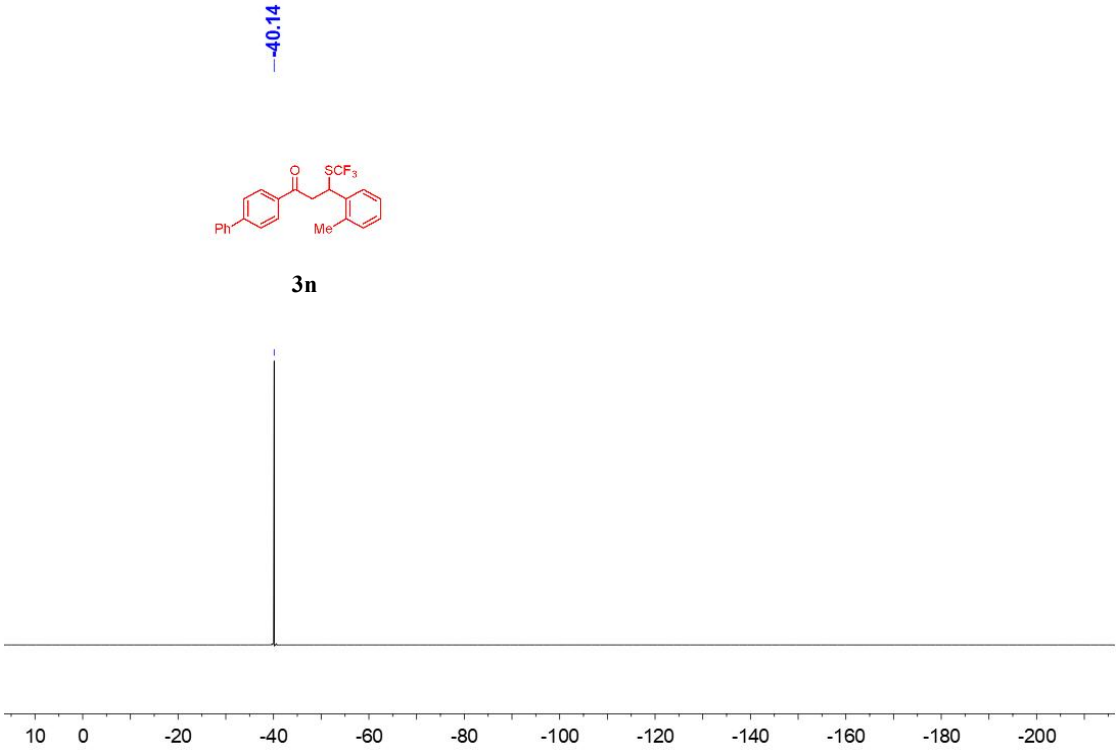
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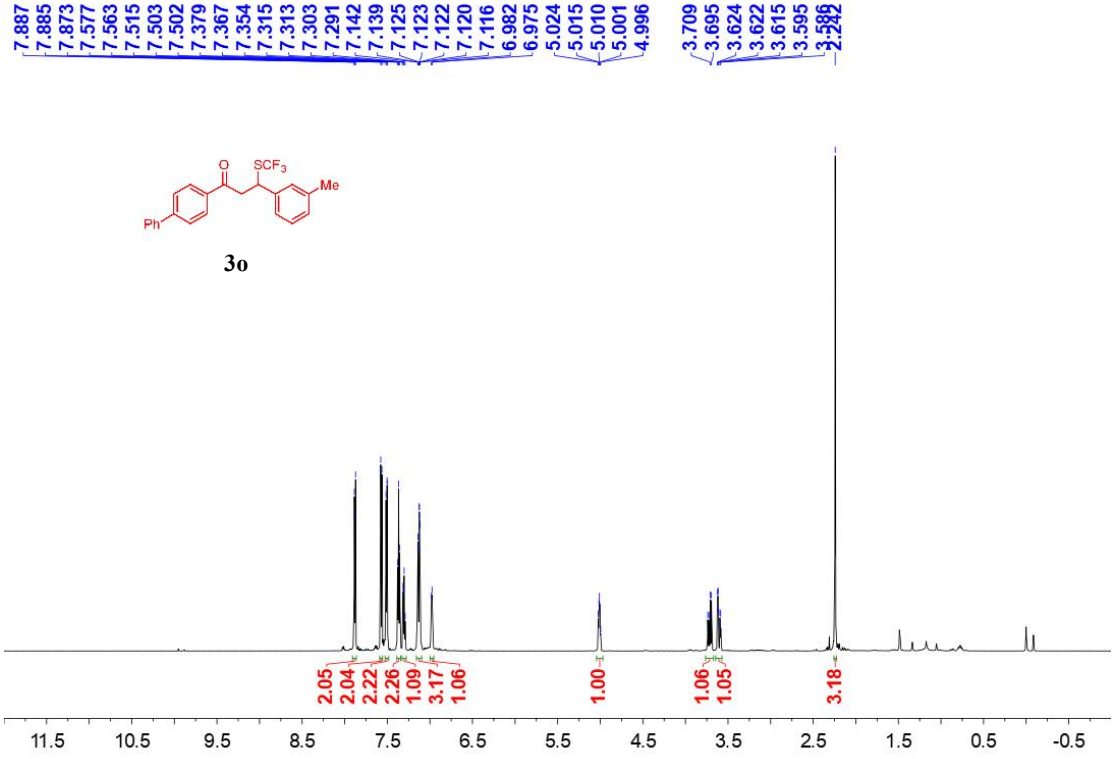
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SCF3
Me
Ph
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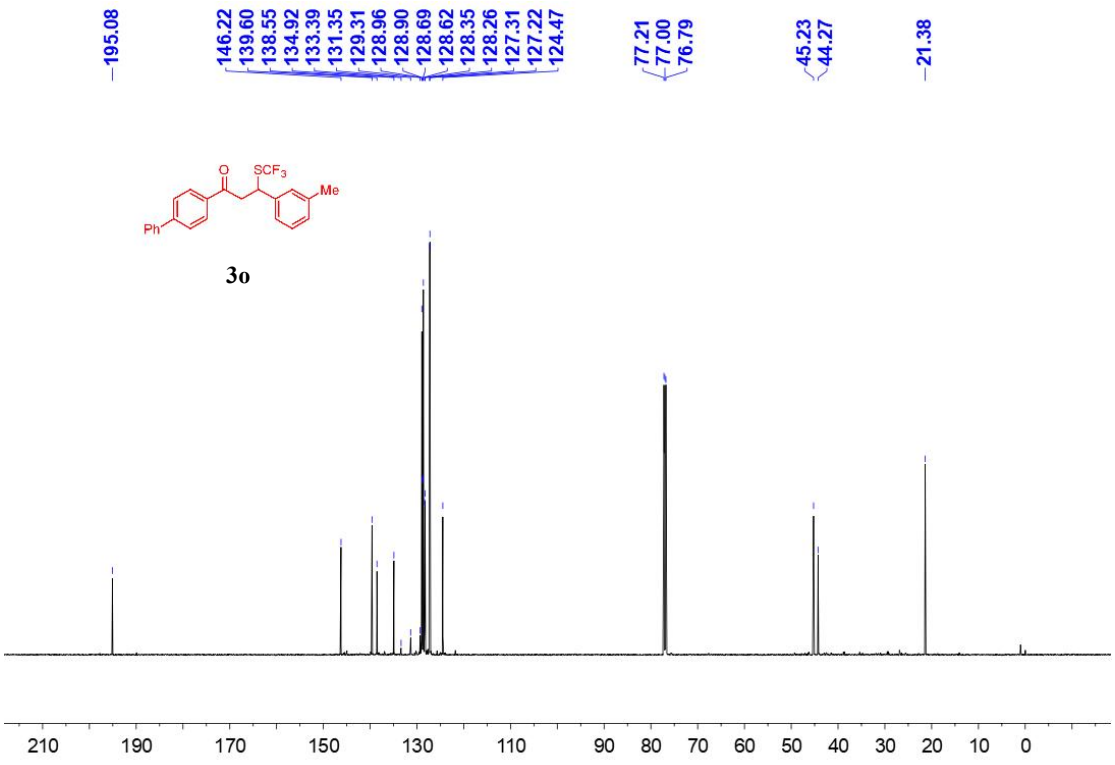
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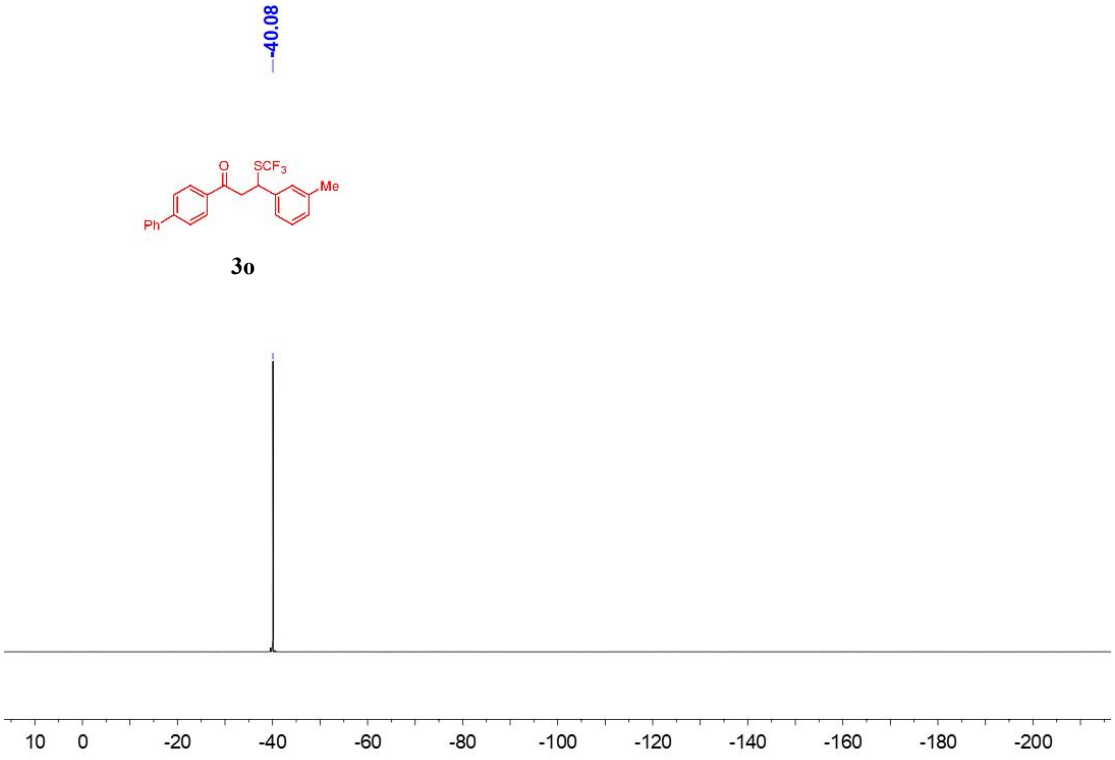
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3o


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F
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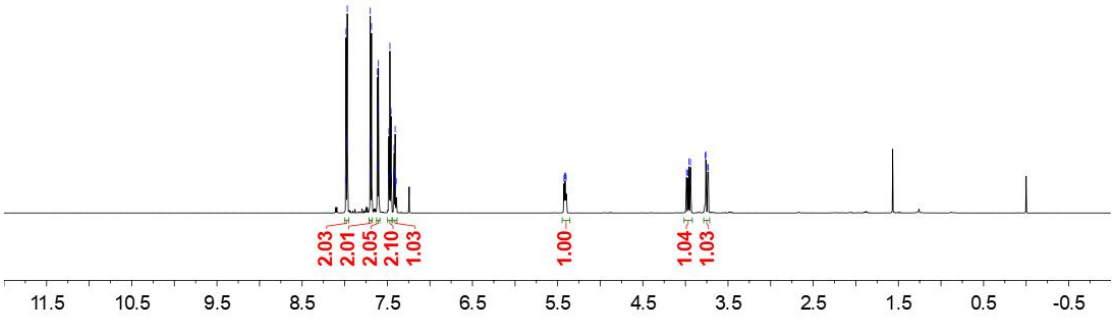
F
Ph

3p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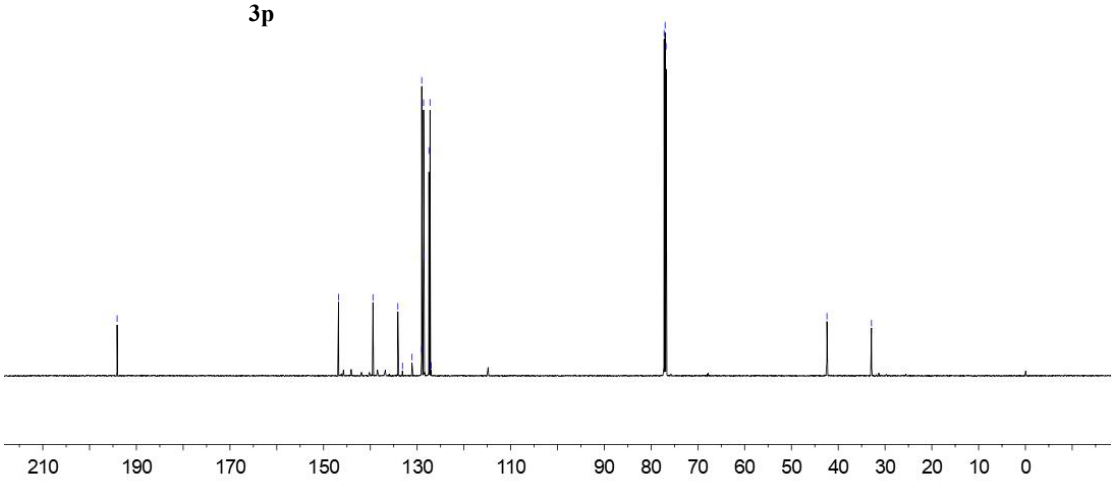
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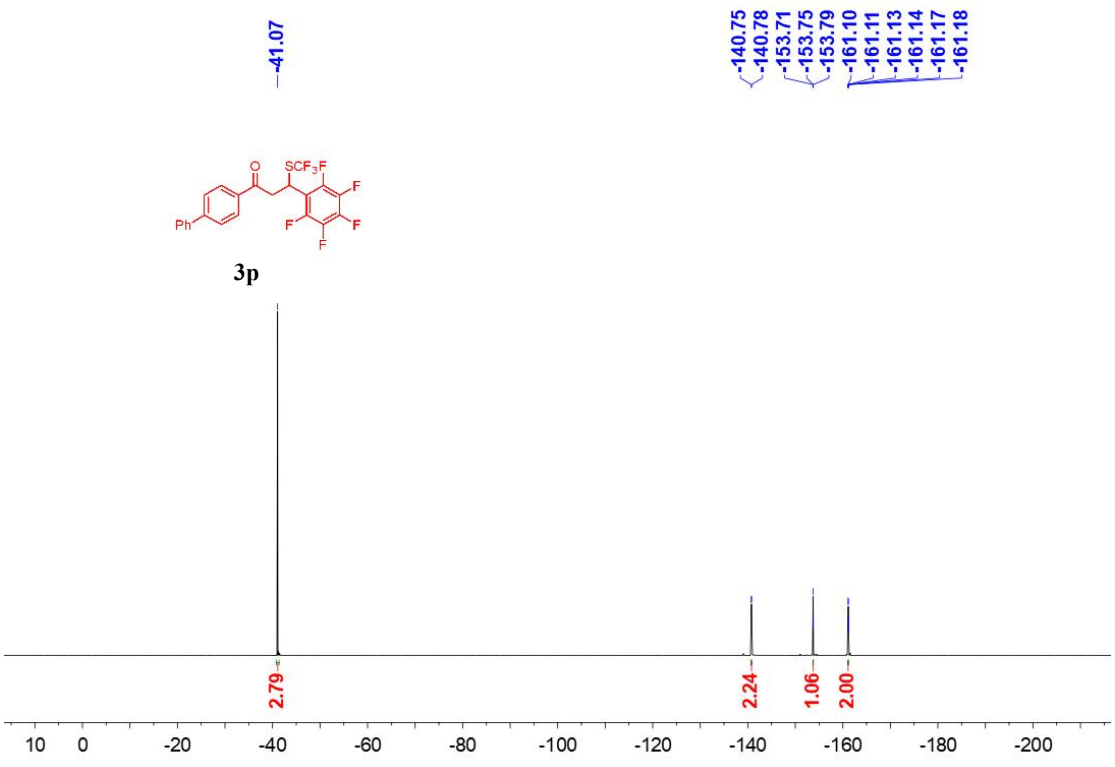
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SCF3
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F
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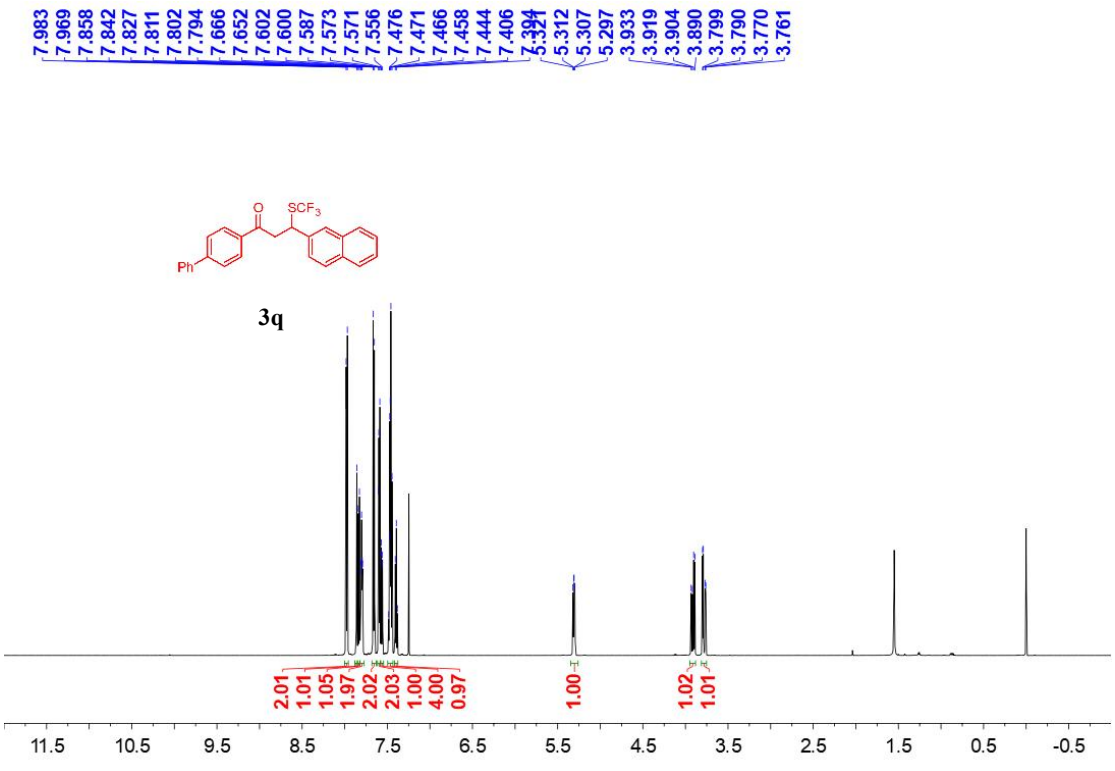
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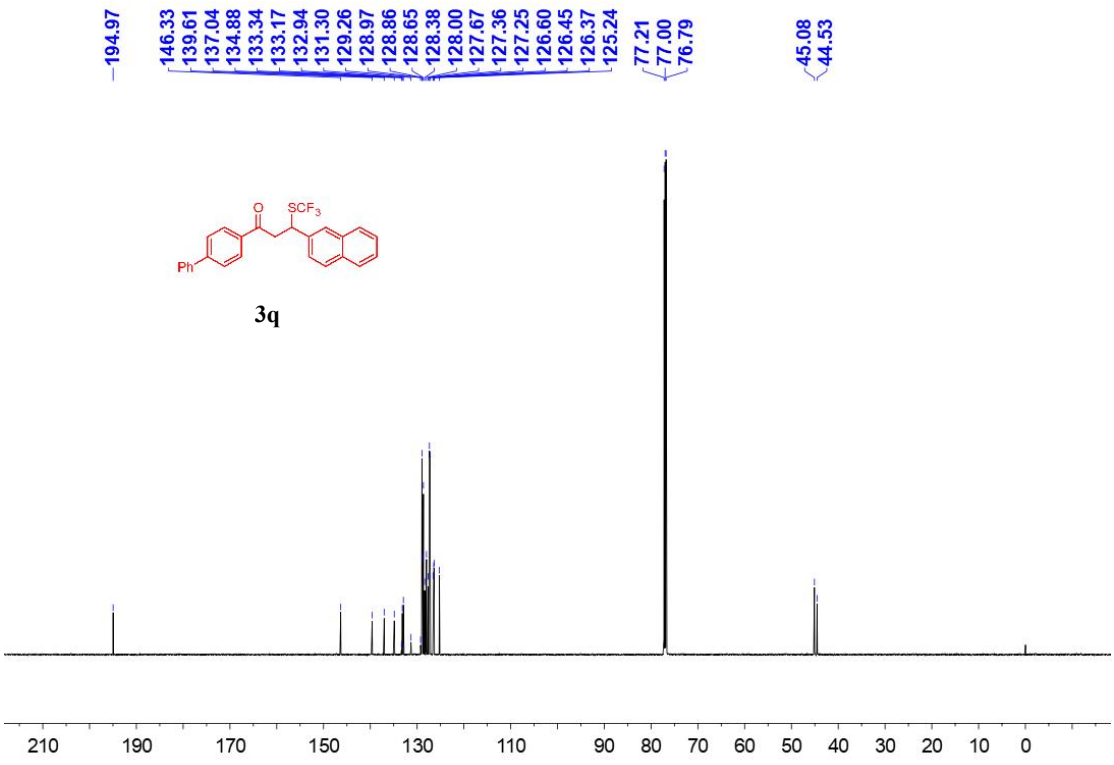
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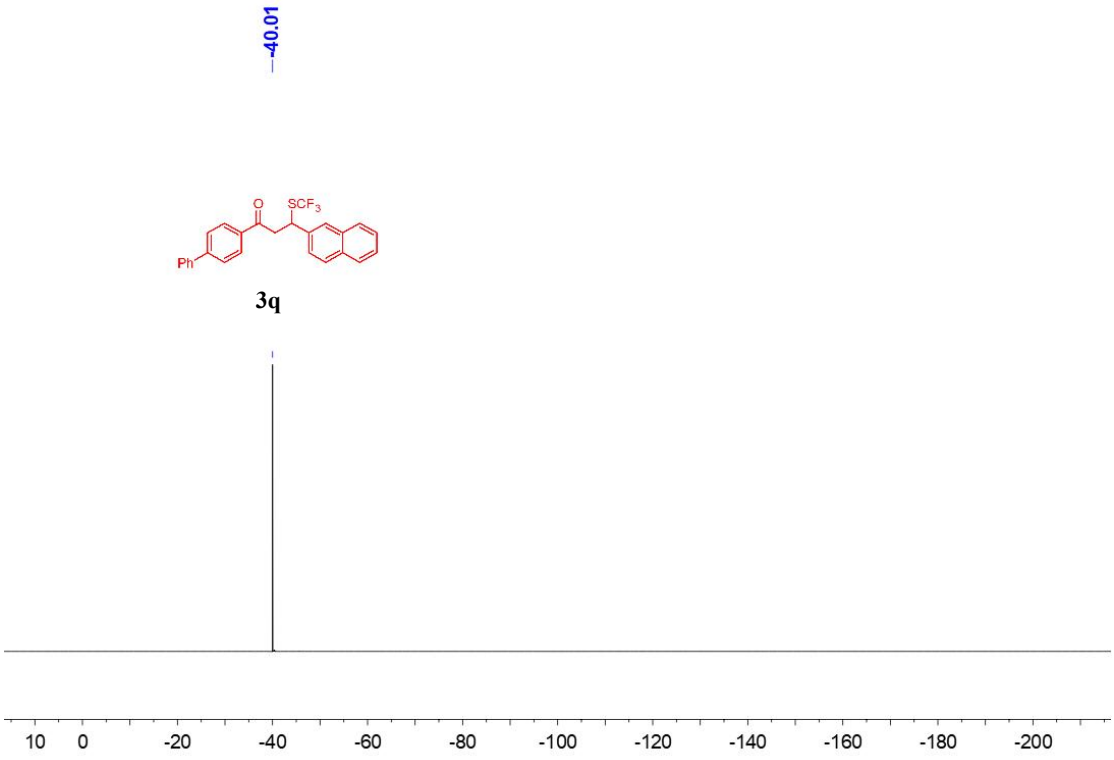
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3q


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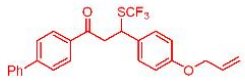
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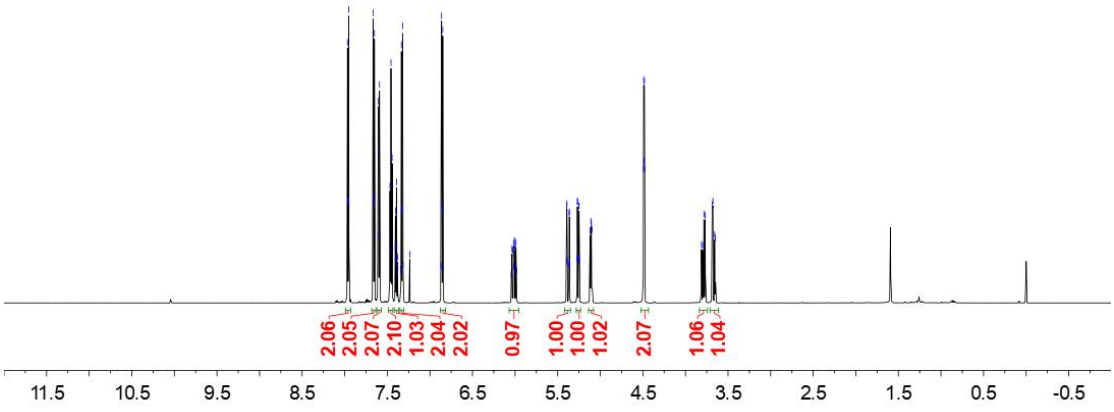
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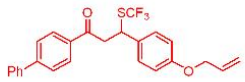
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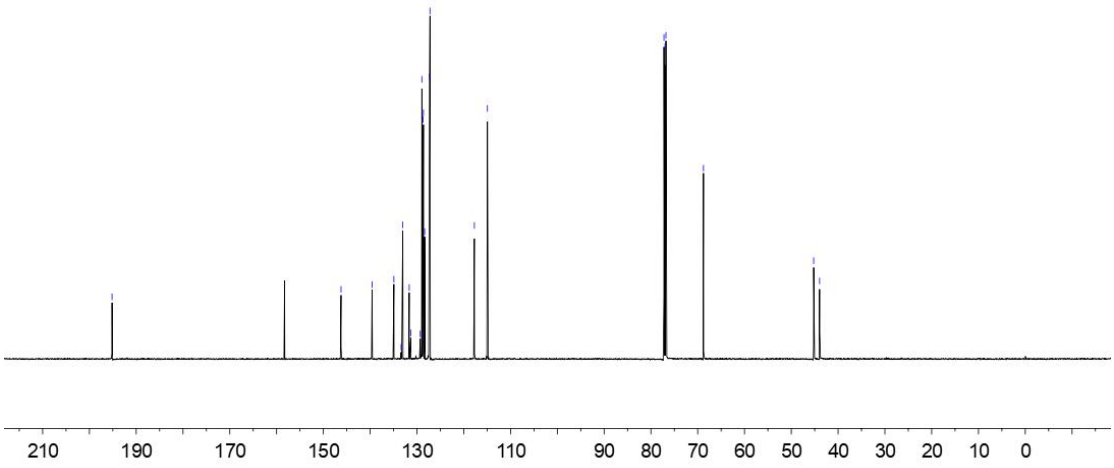
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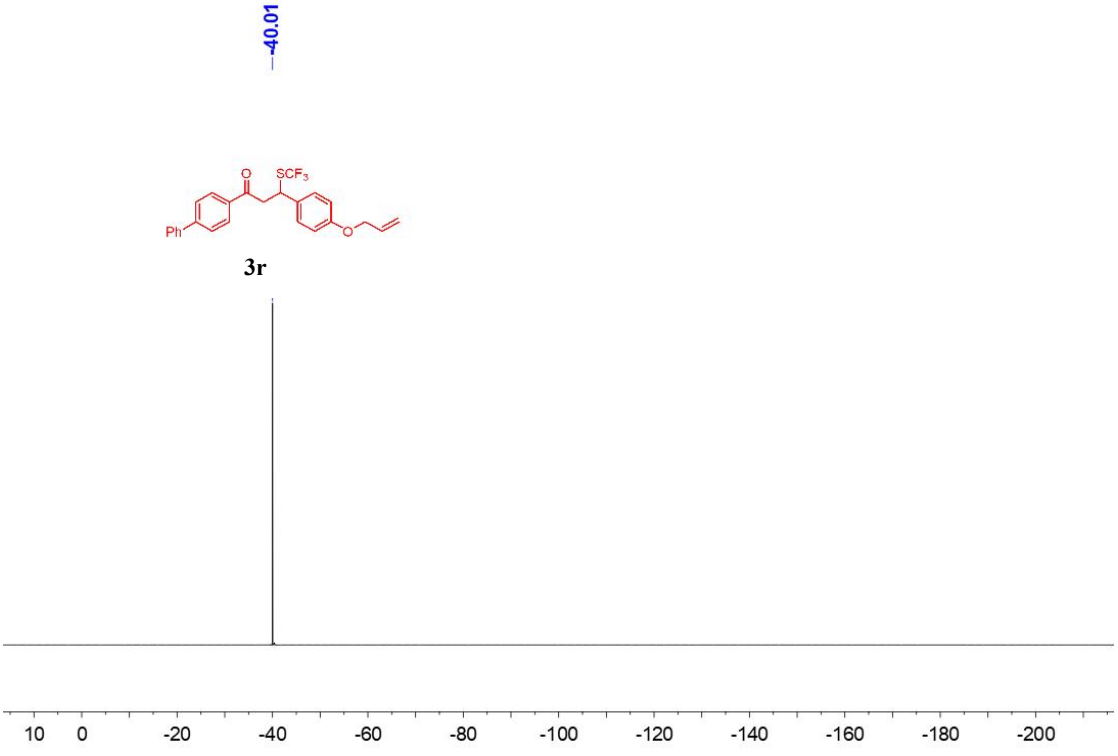
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3r


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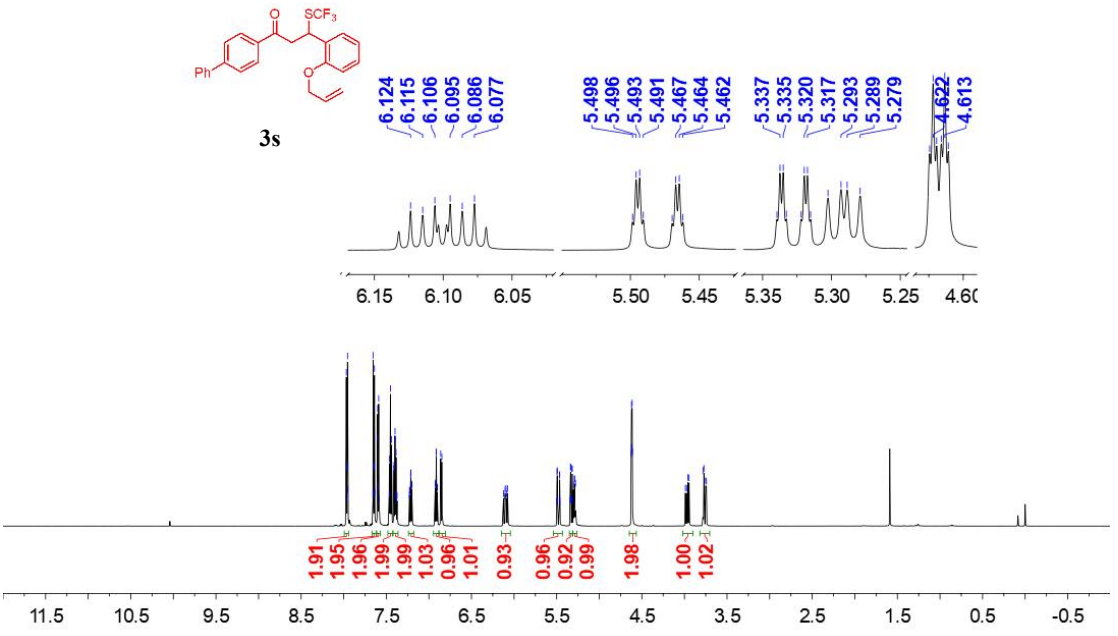
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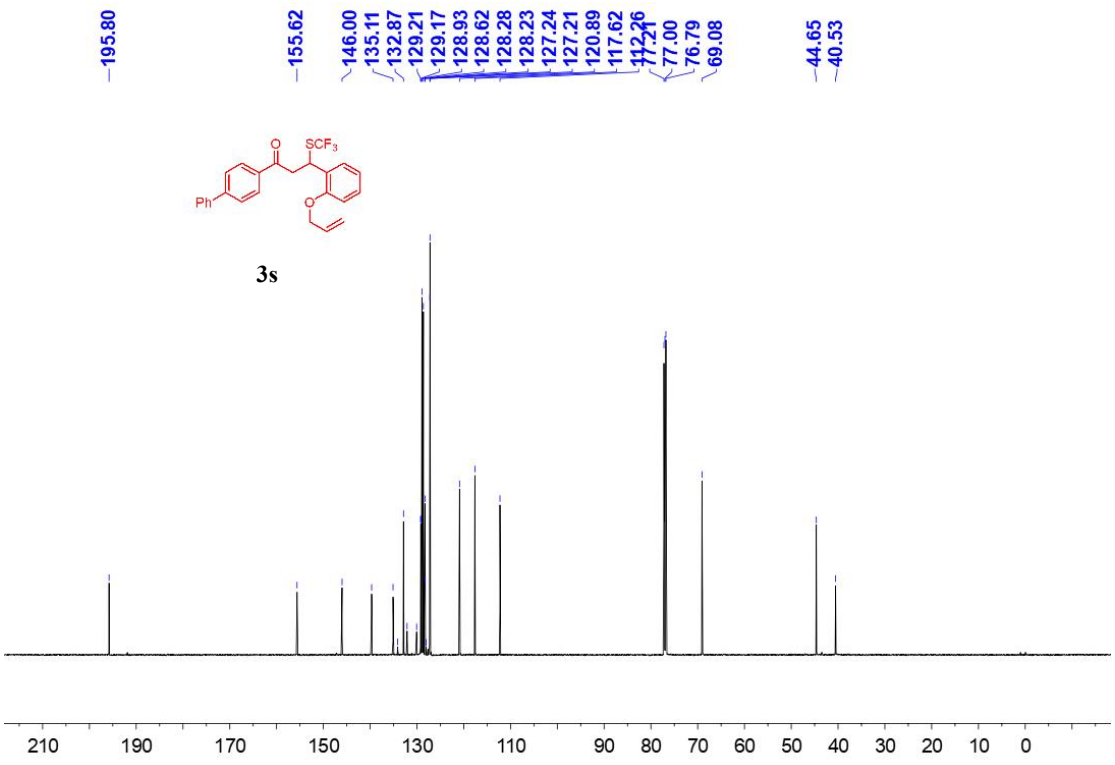
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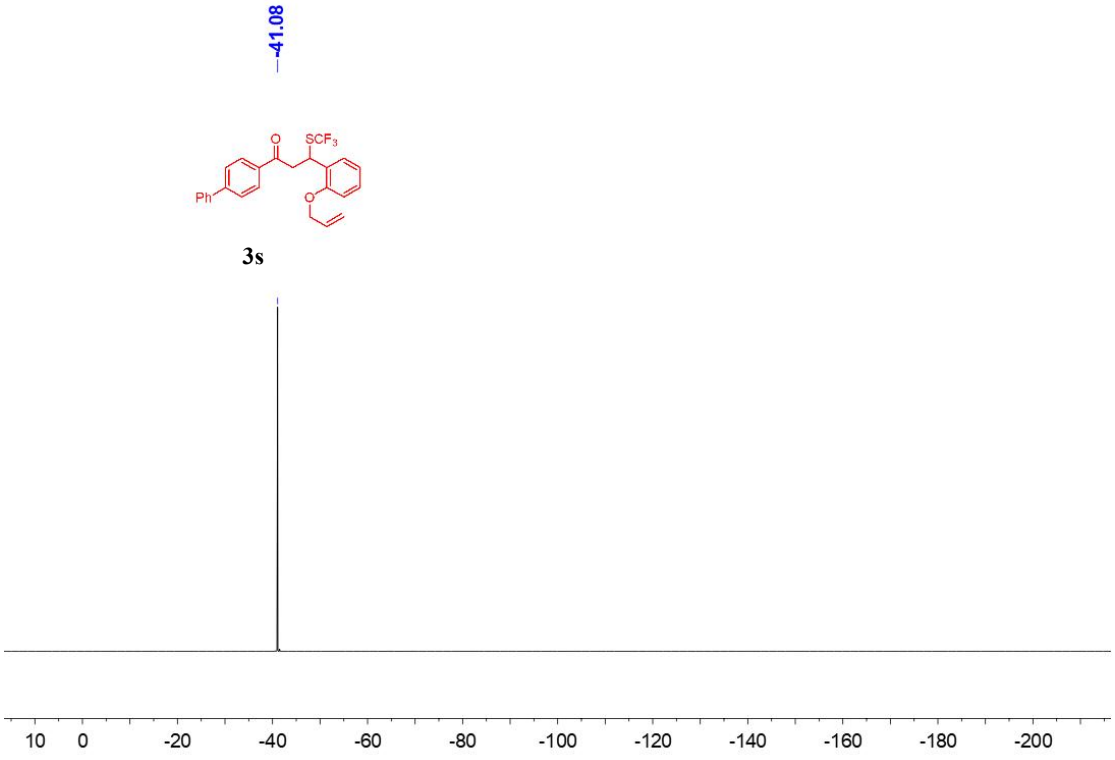
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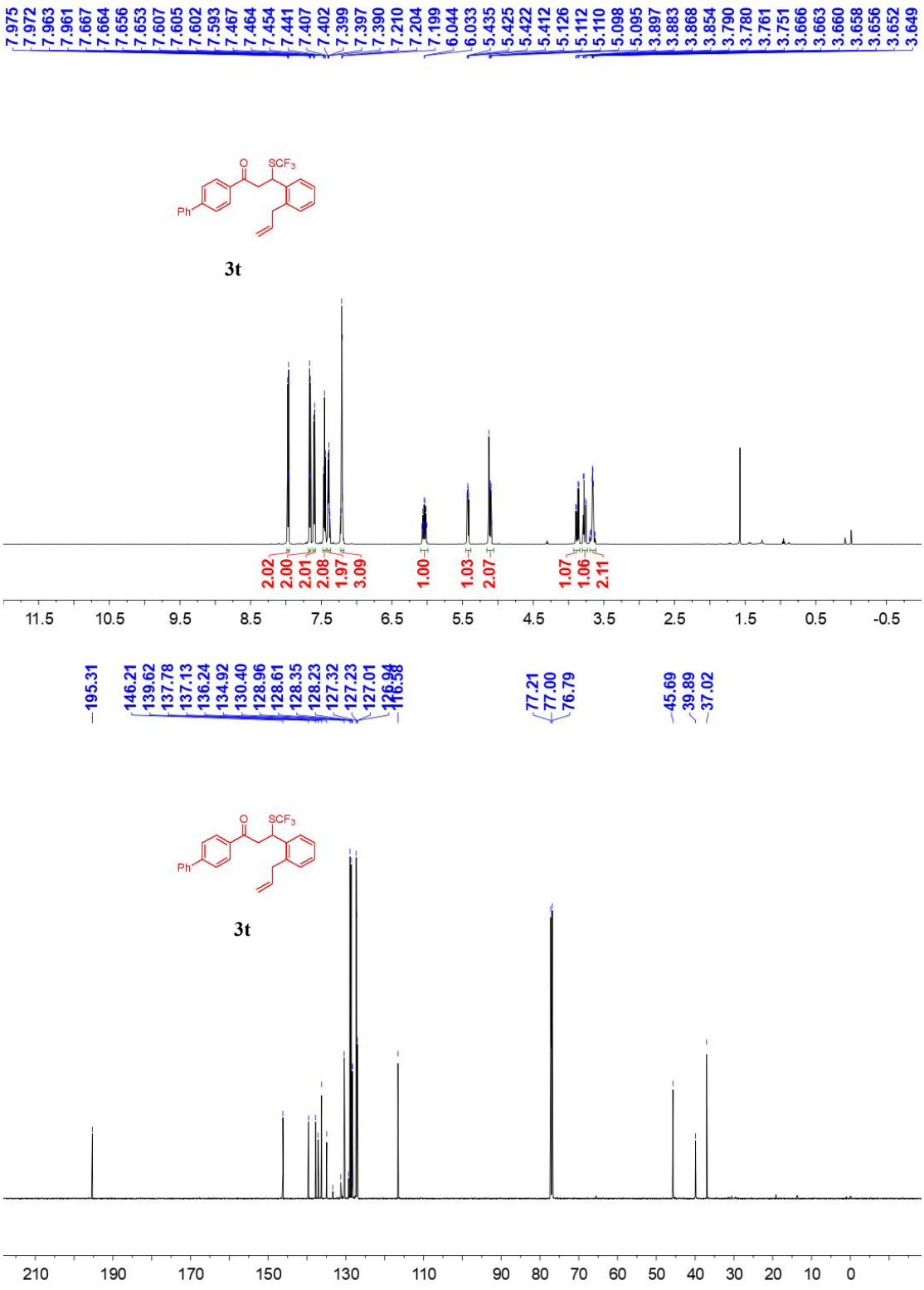
3s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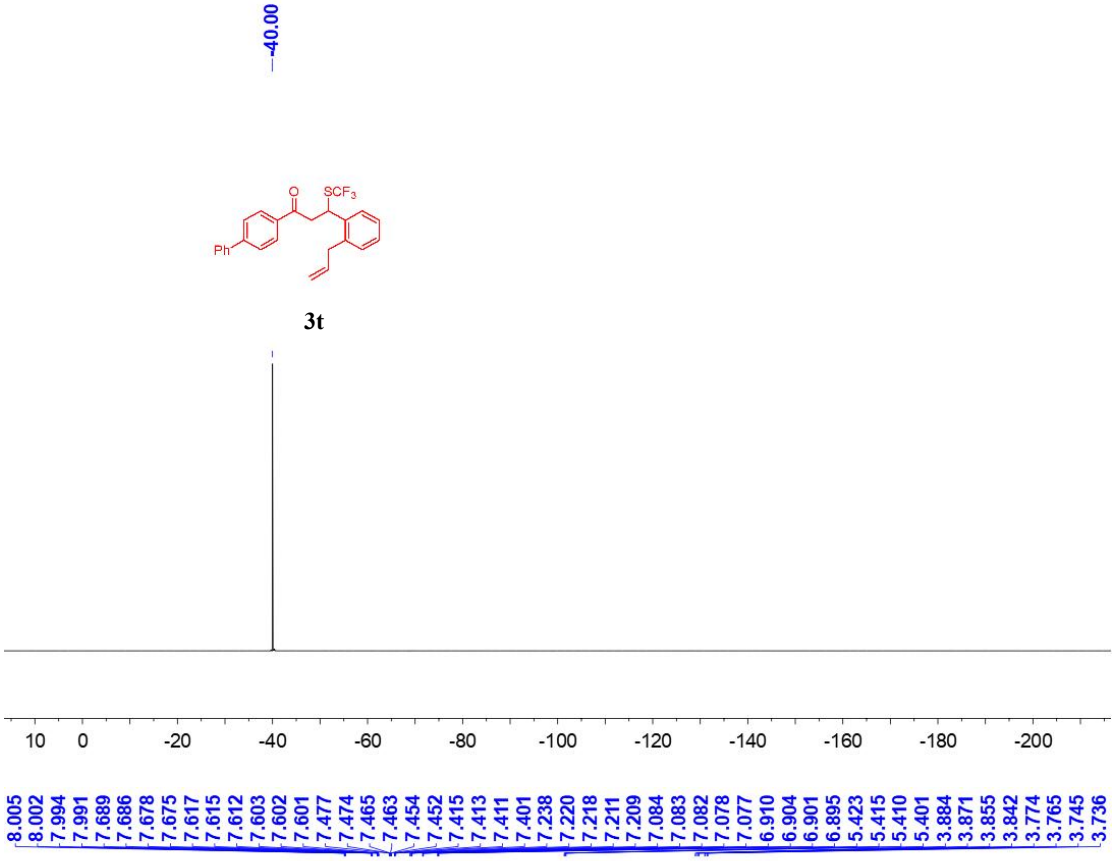
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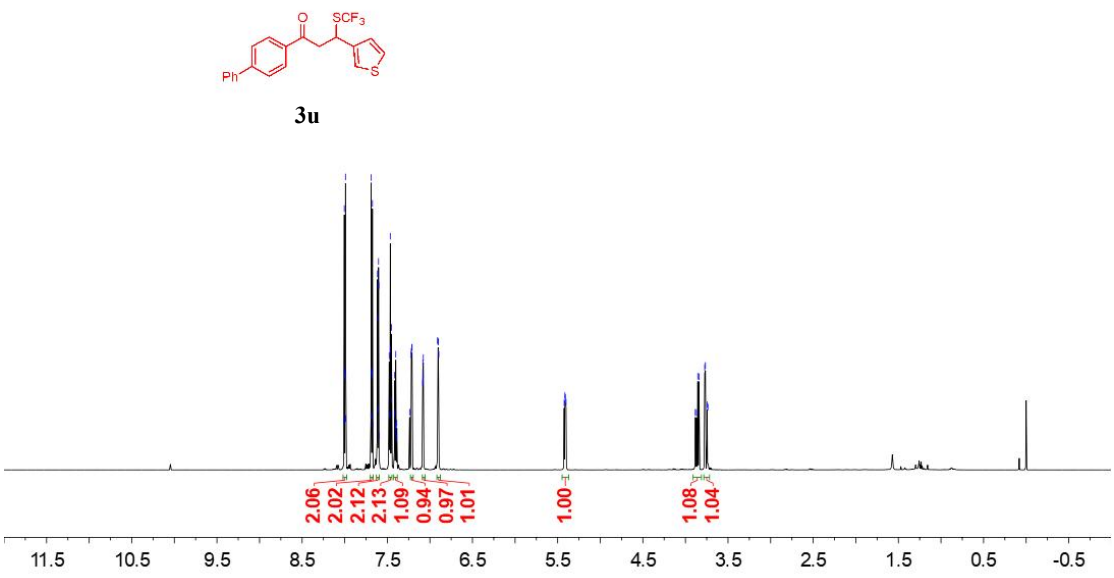
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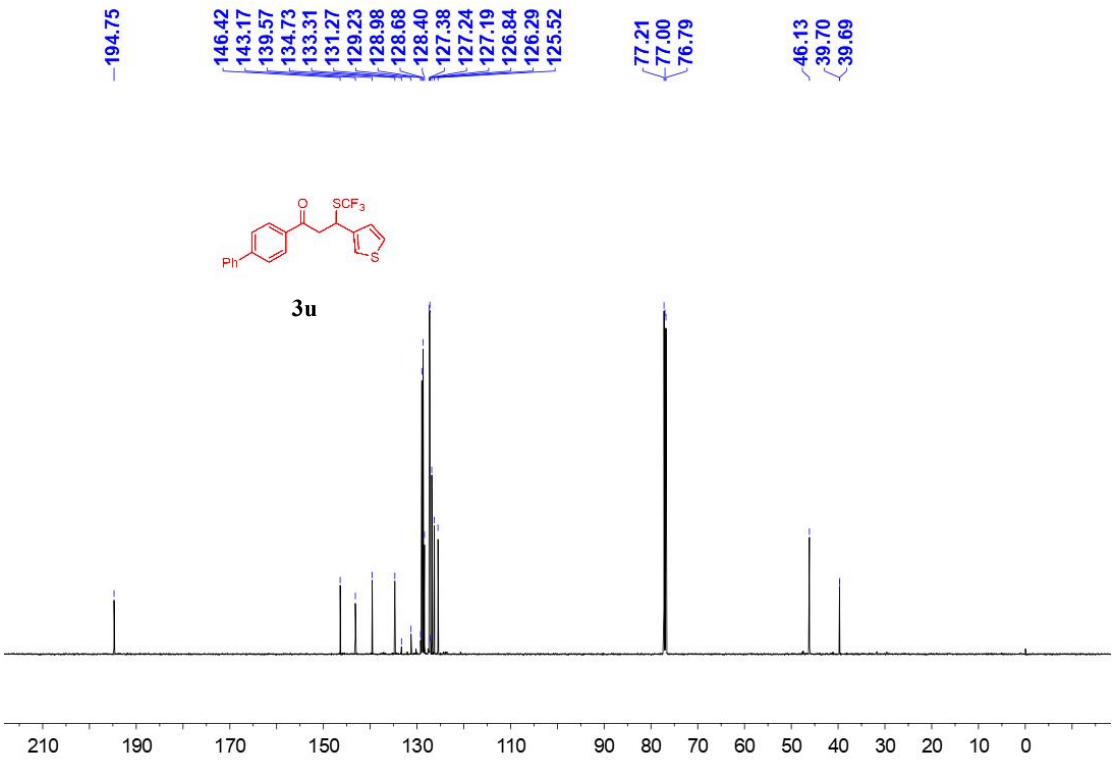
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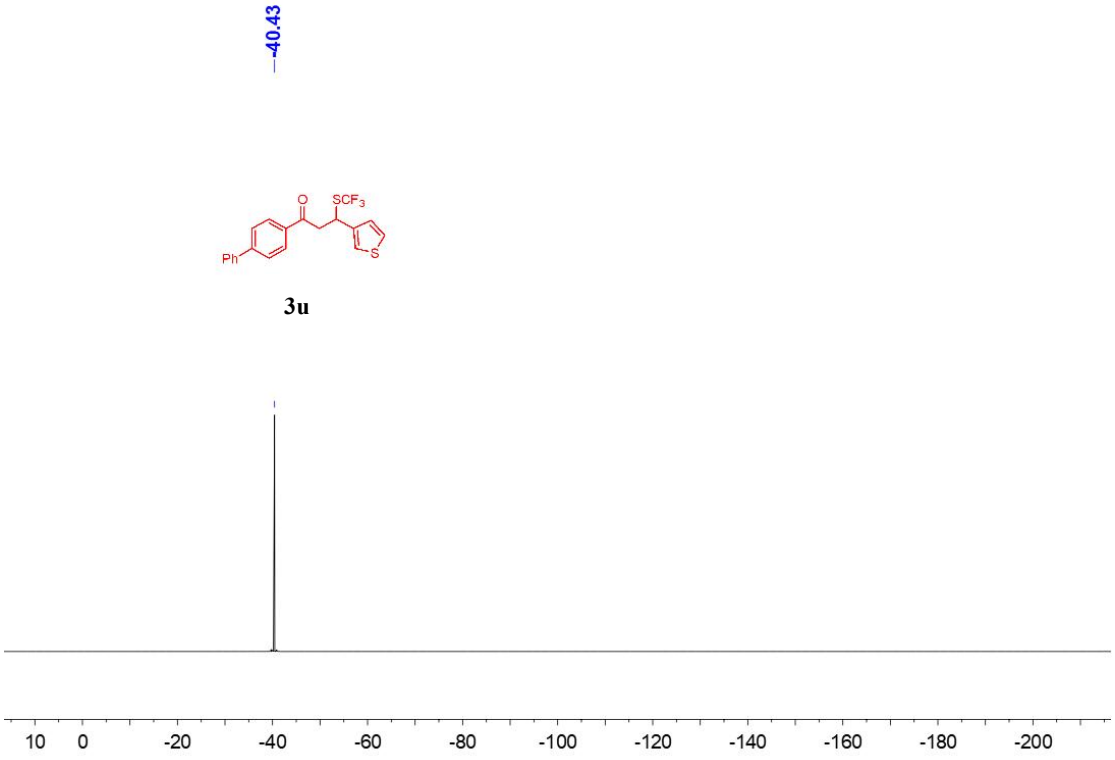
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3u


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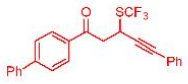
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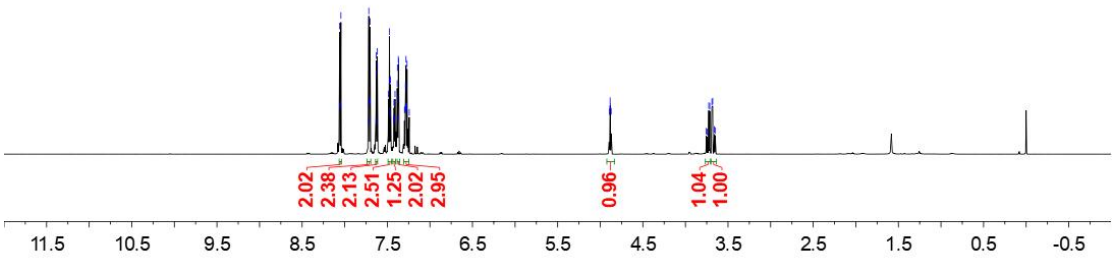
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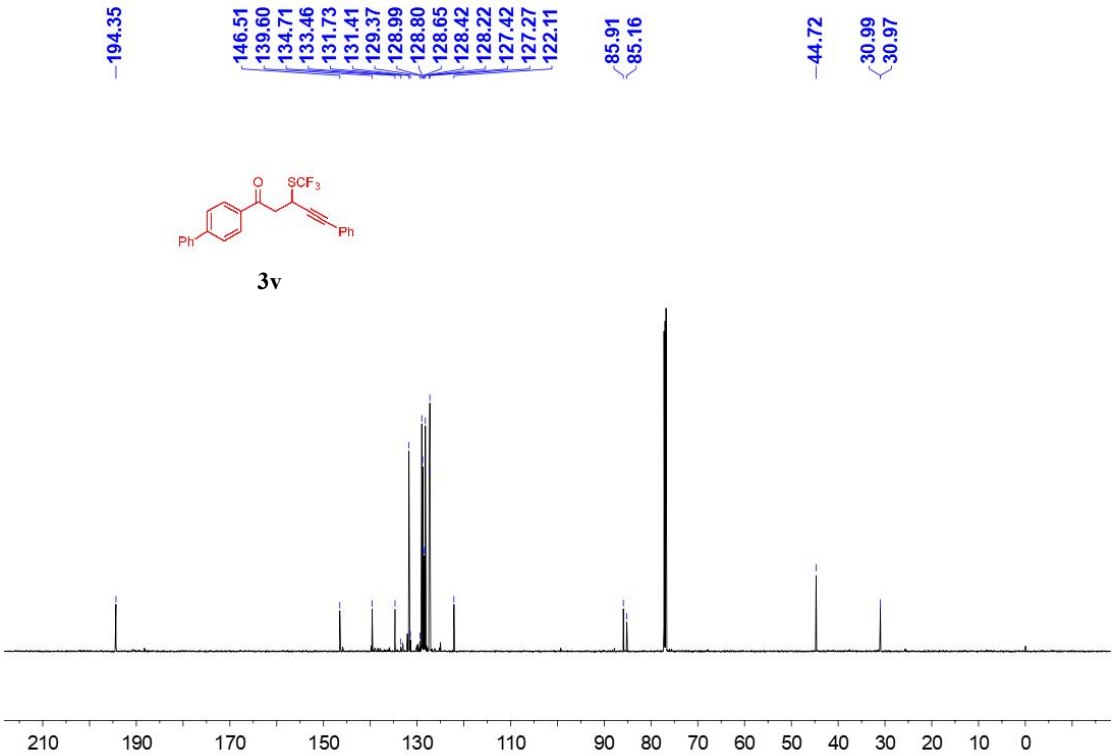
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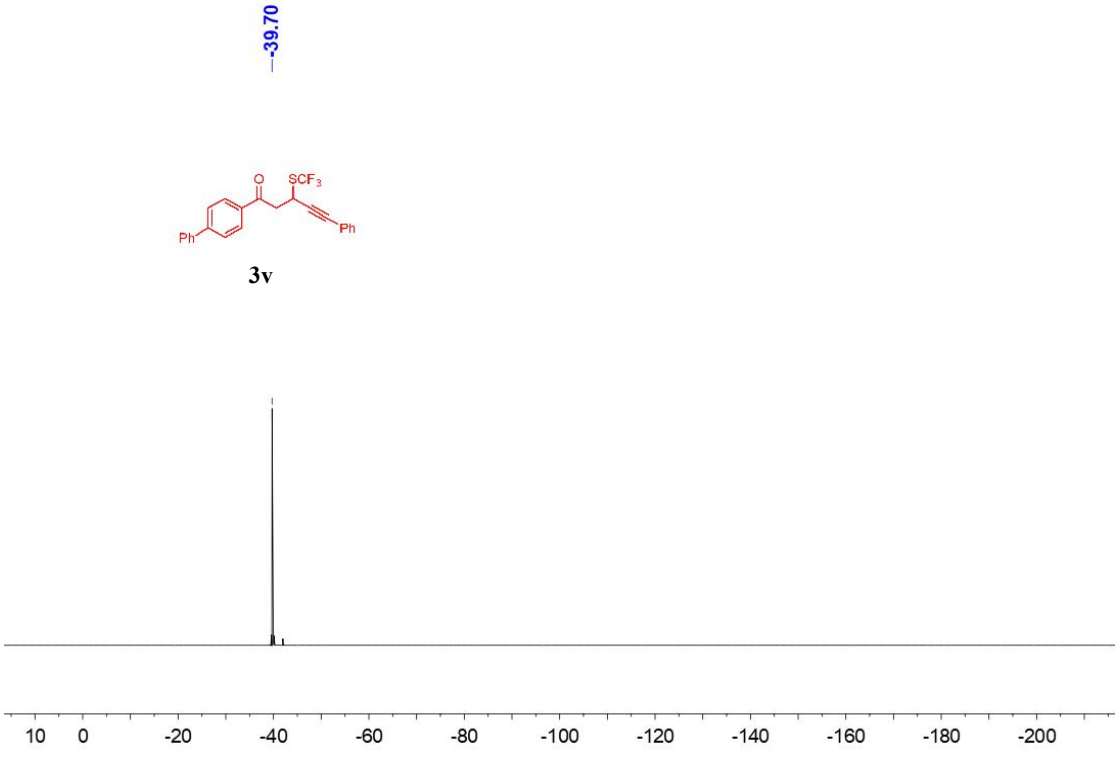
3v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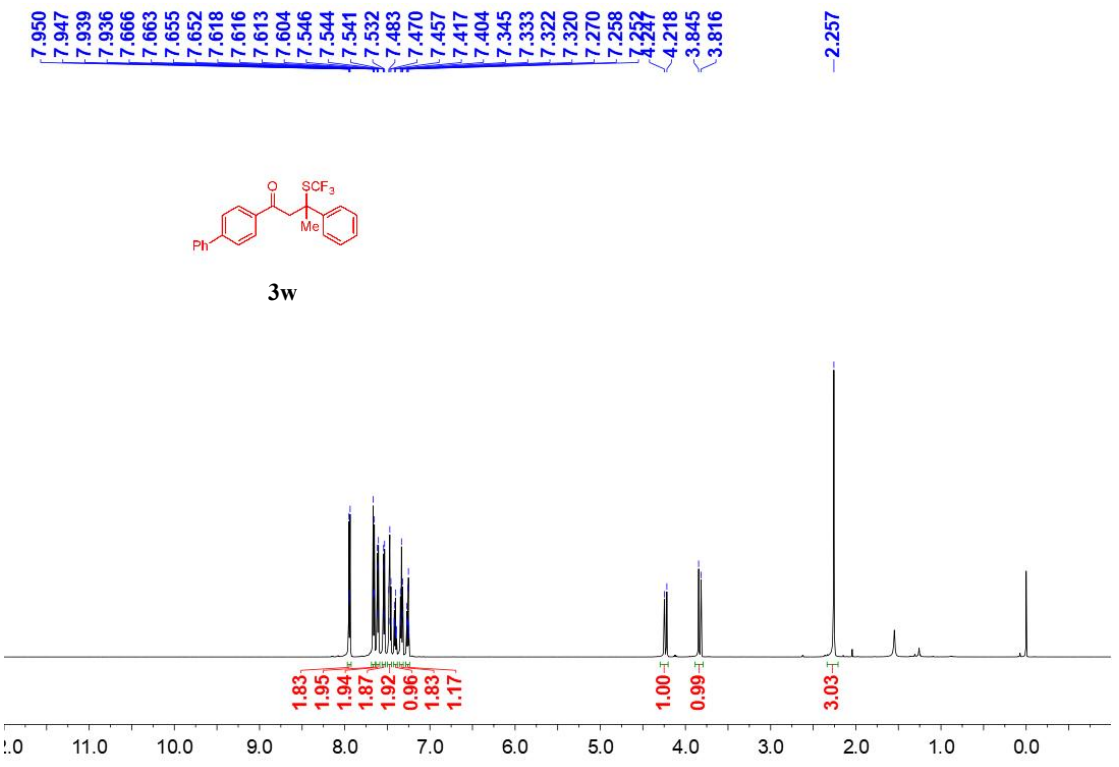
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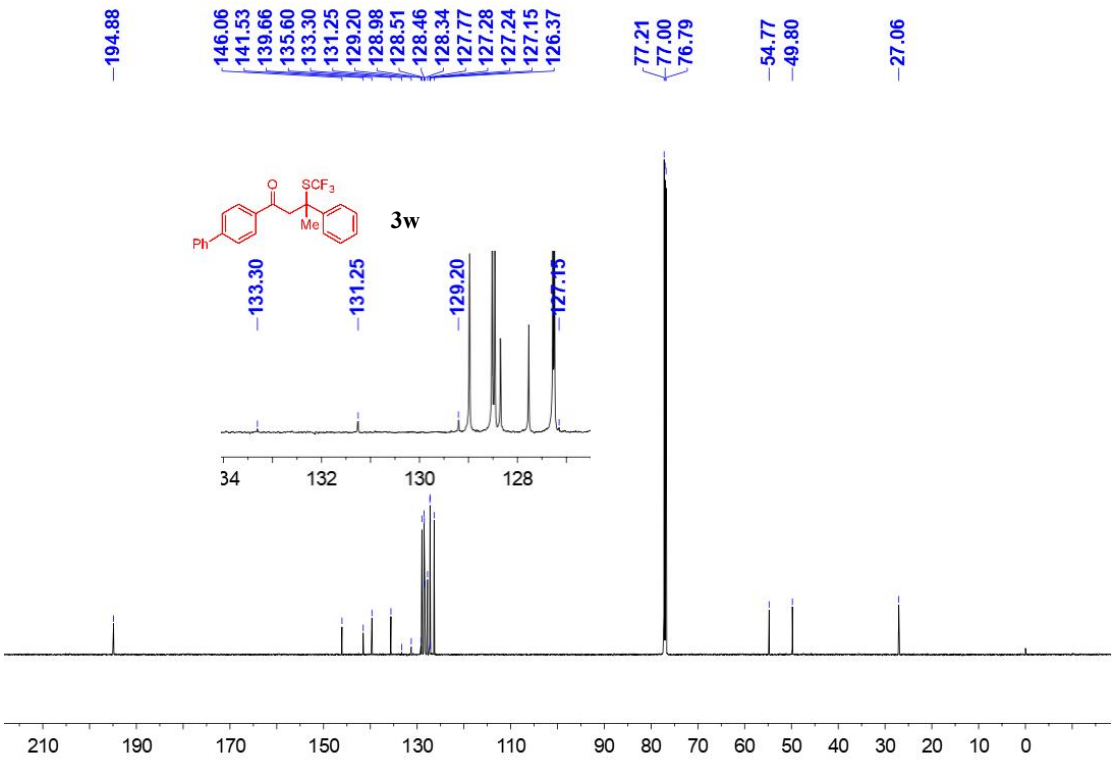
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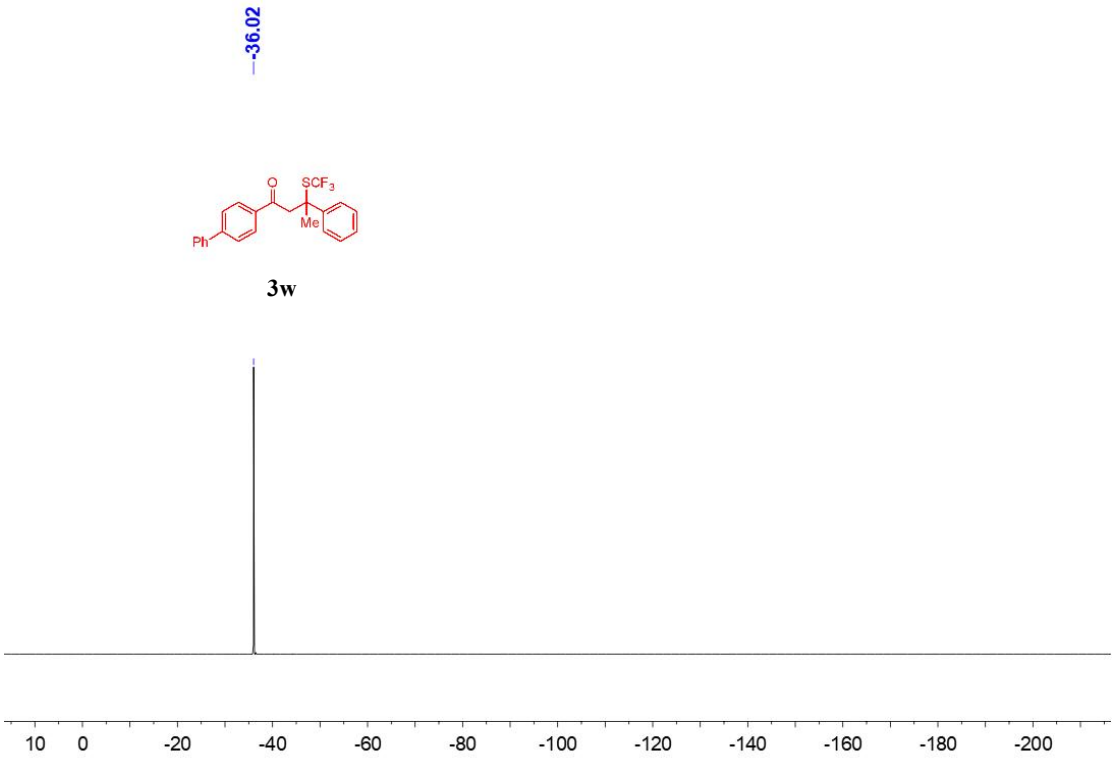
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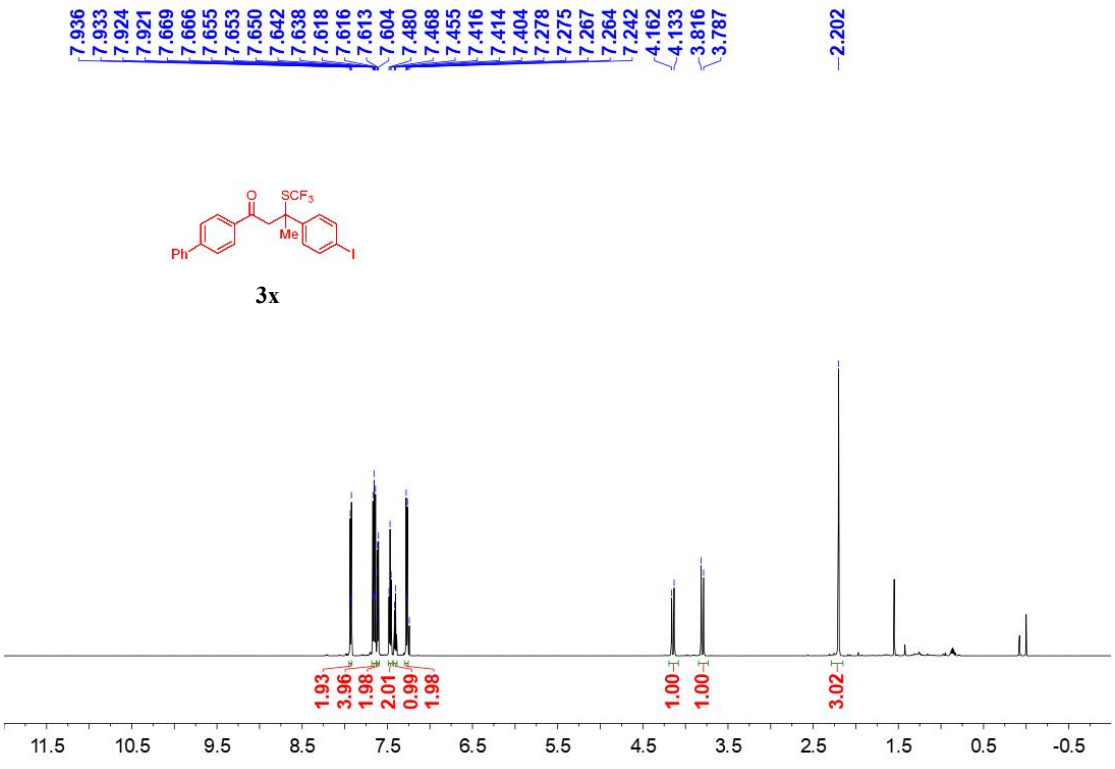
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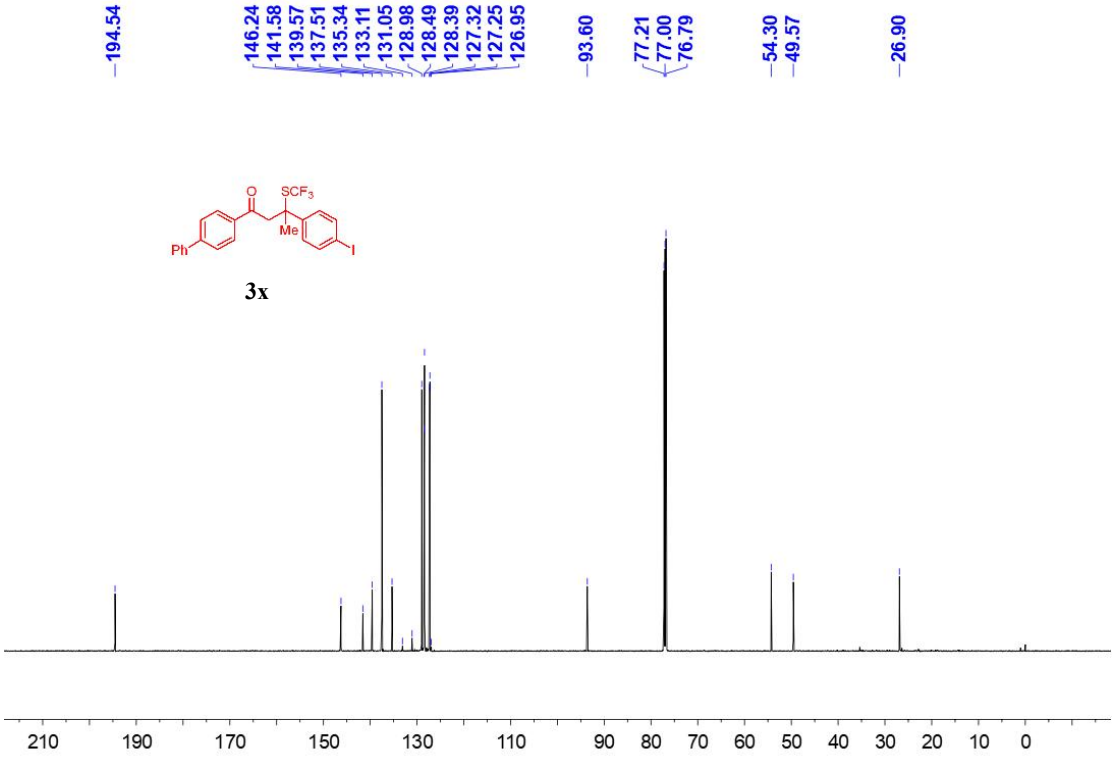
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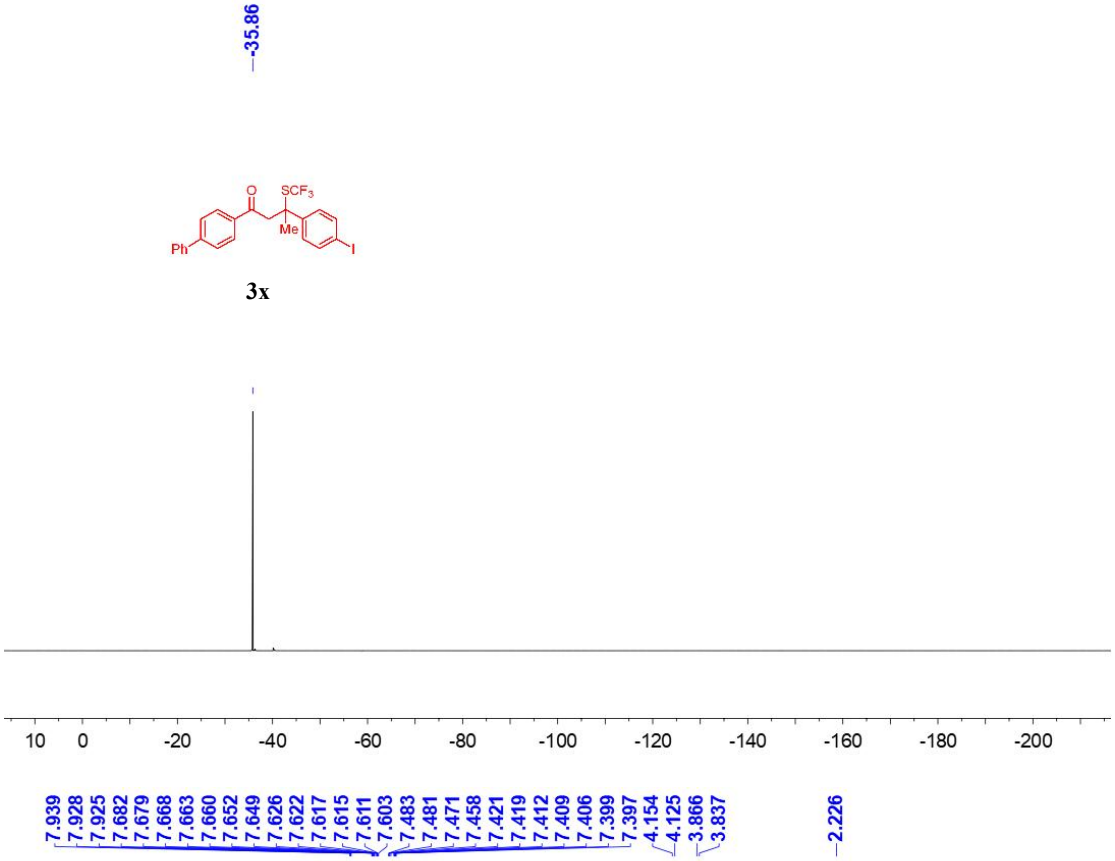
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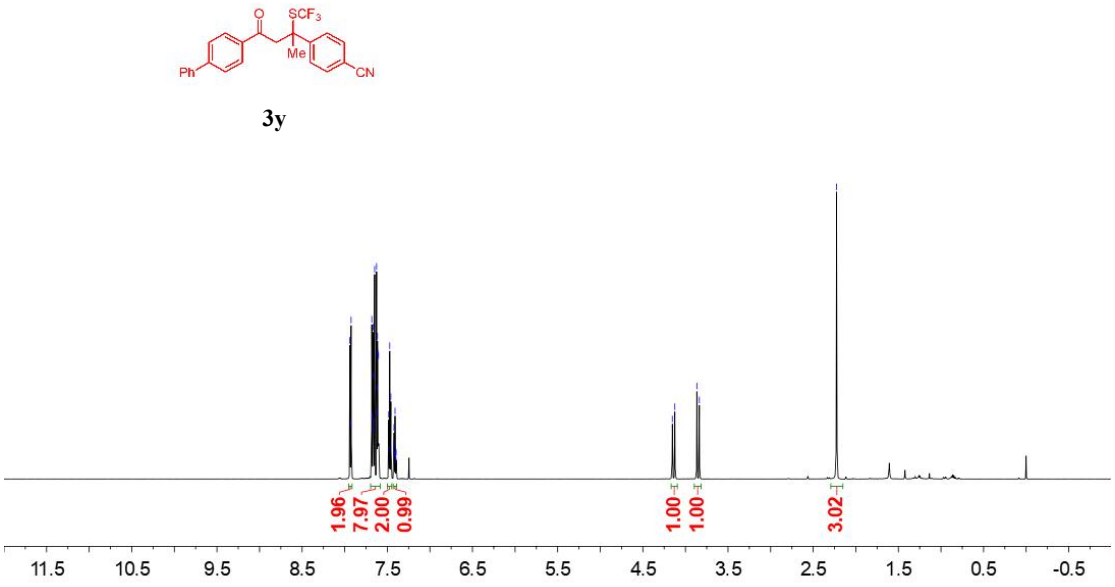
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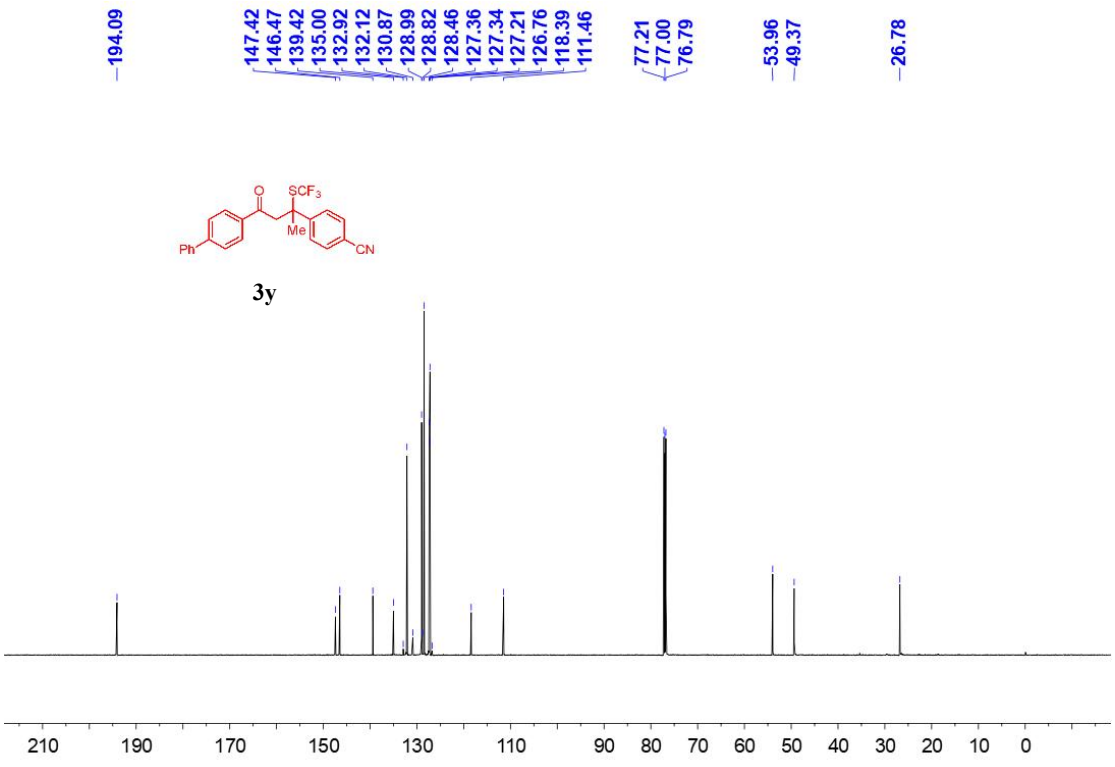
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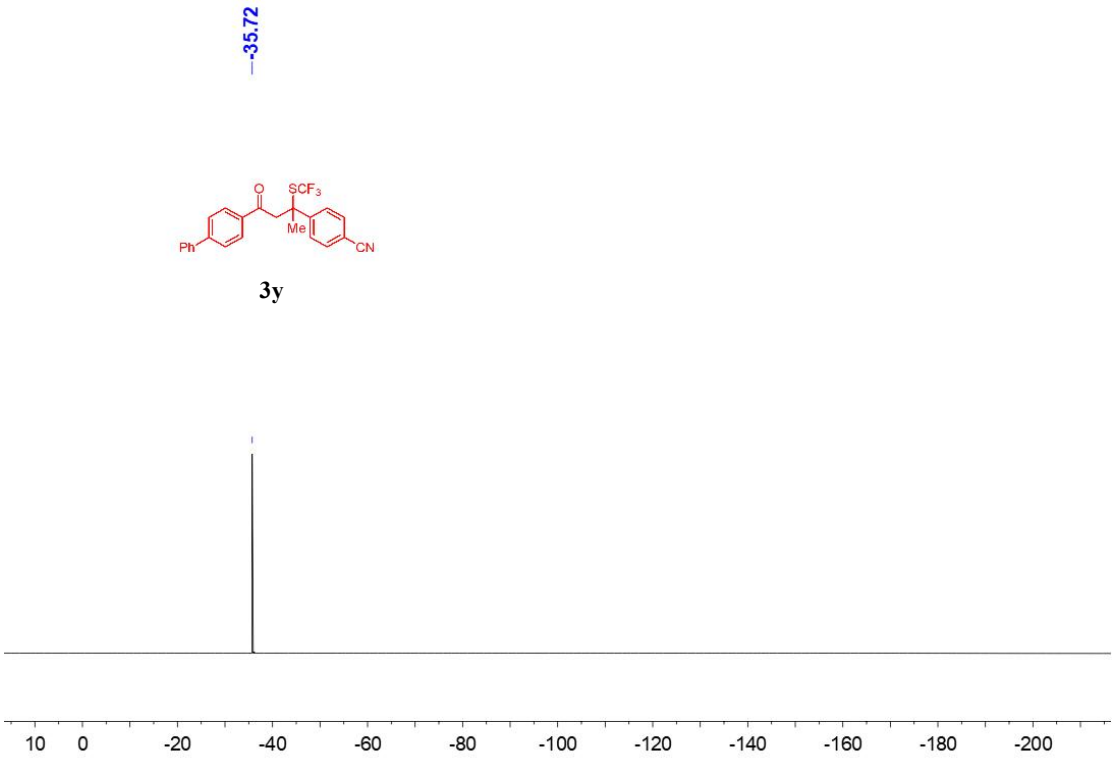
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3y


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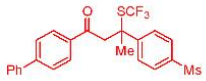
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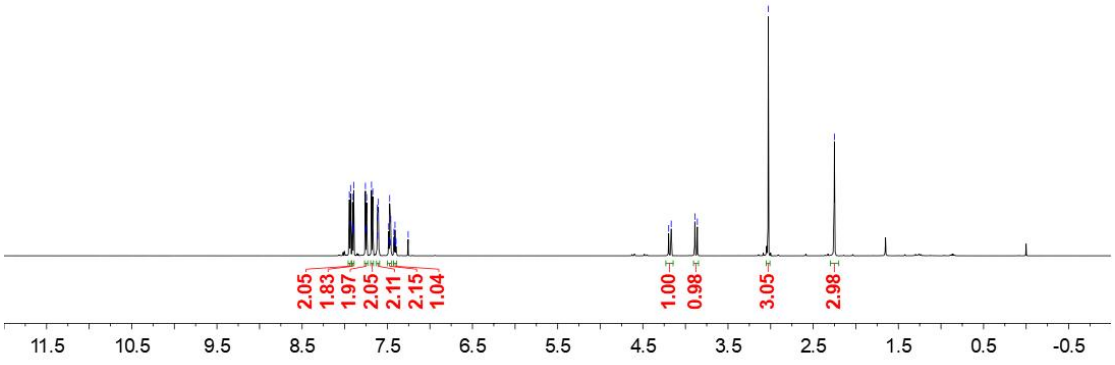
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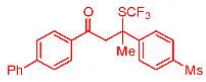
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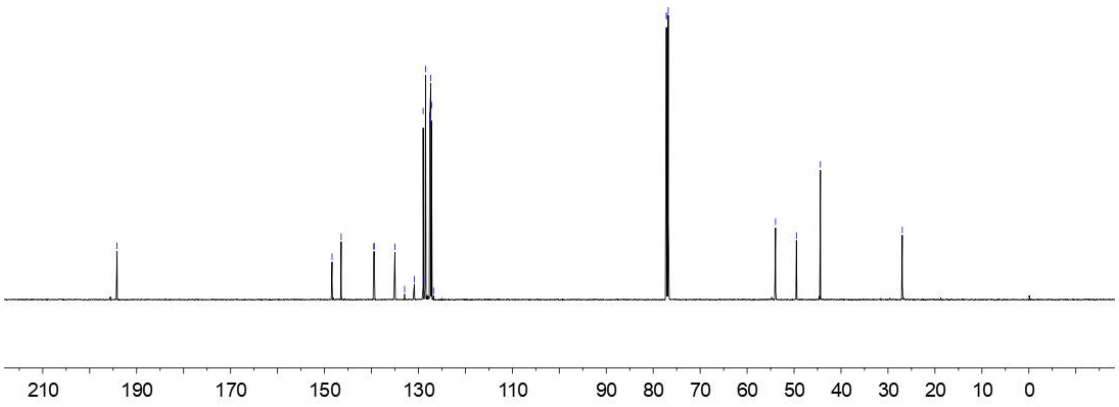
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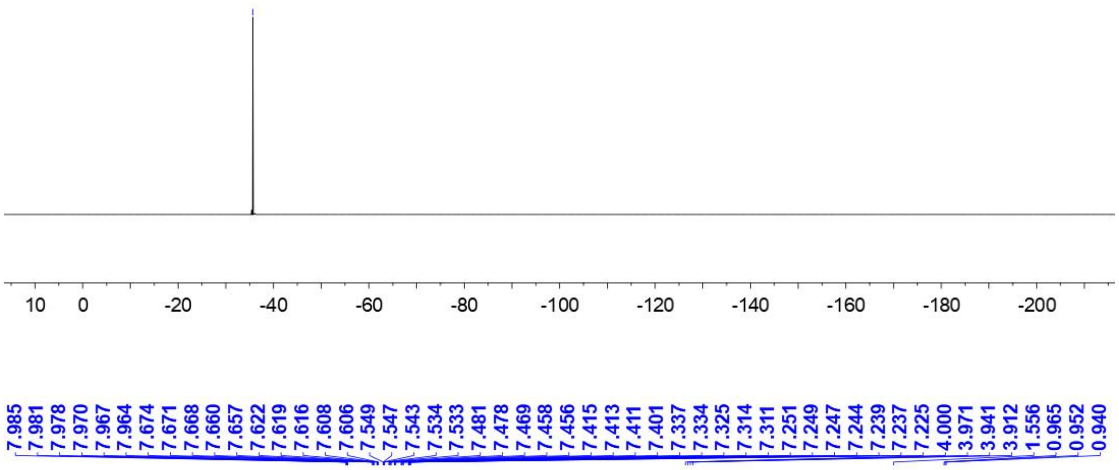
3z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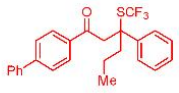
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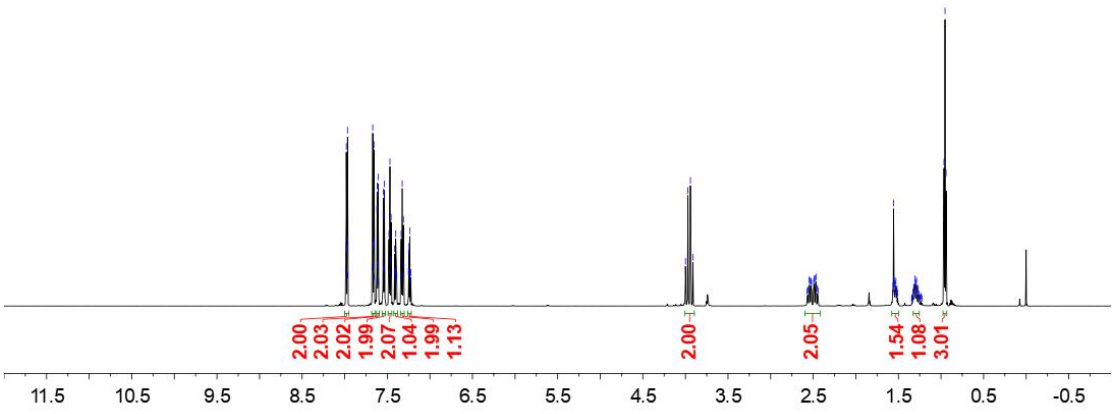
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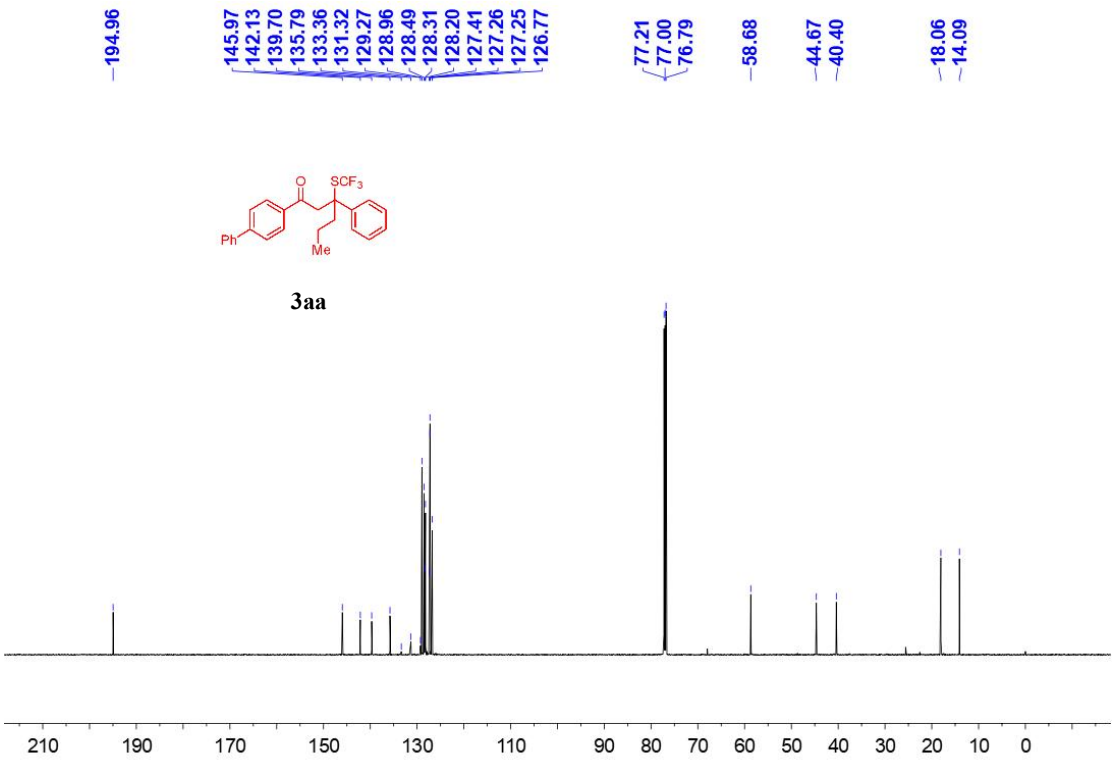
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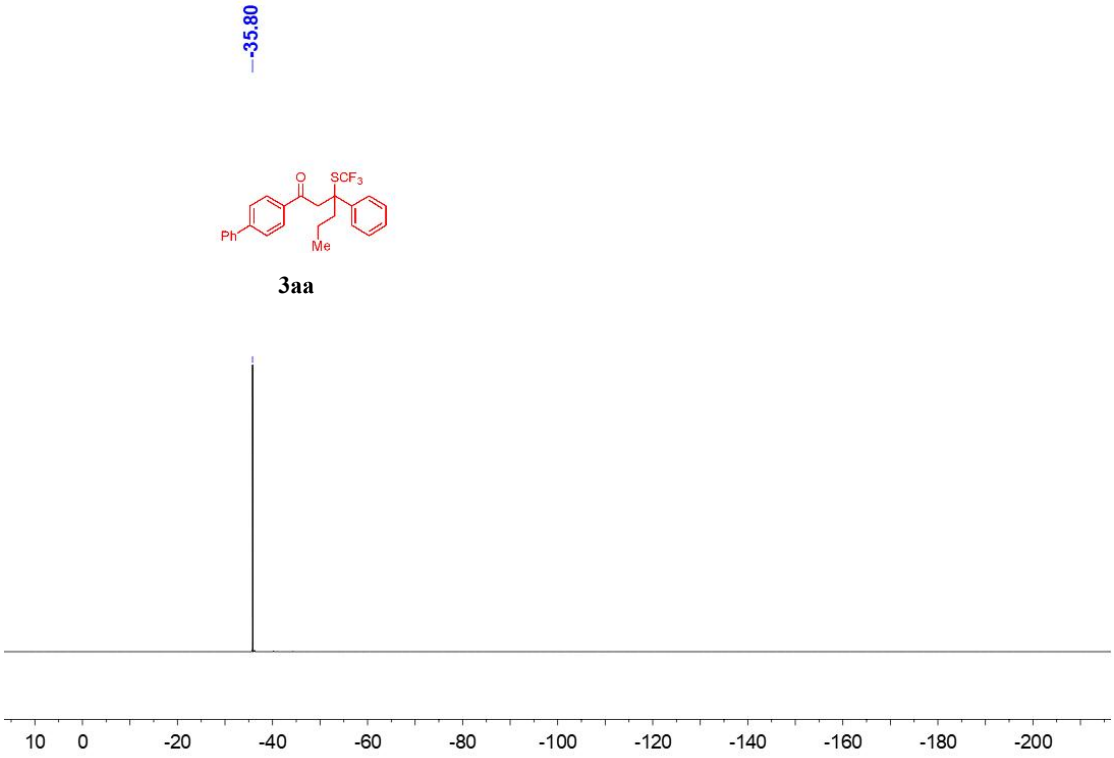
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3aa


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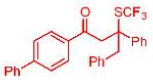
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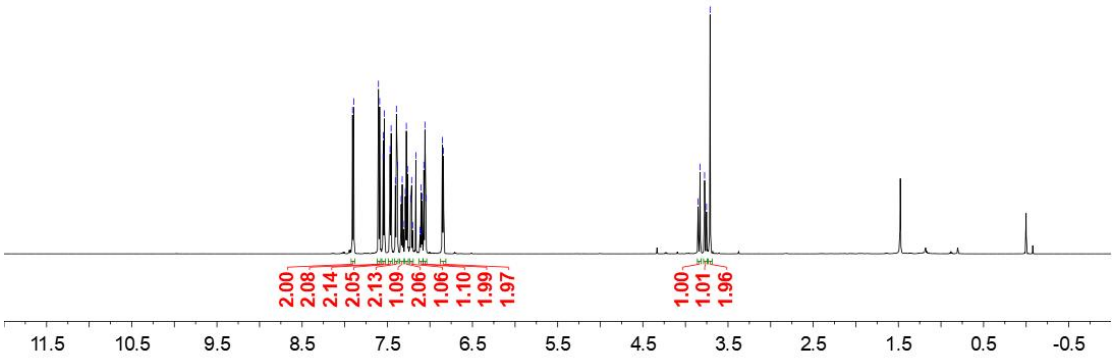
3ab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4.5
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2.5
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-0.5


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146.03
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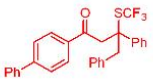
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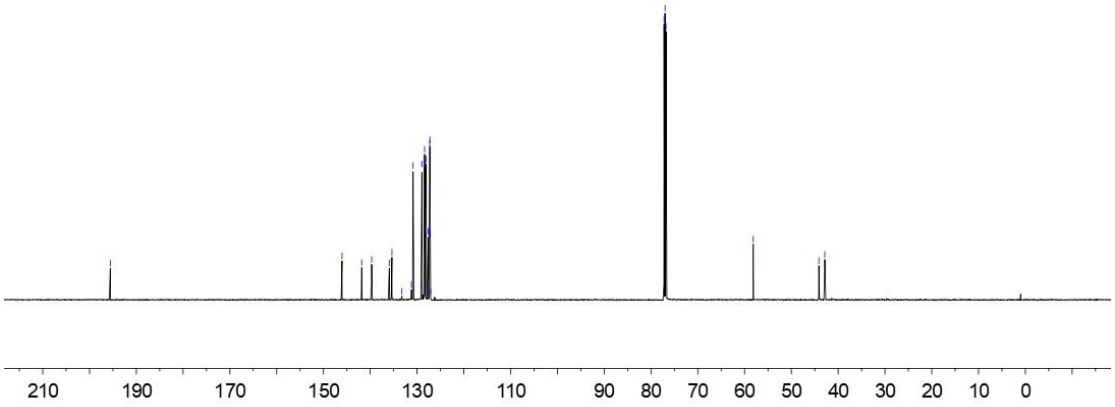
Ph
Ph
Ph

3ab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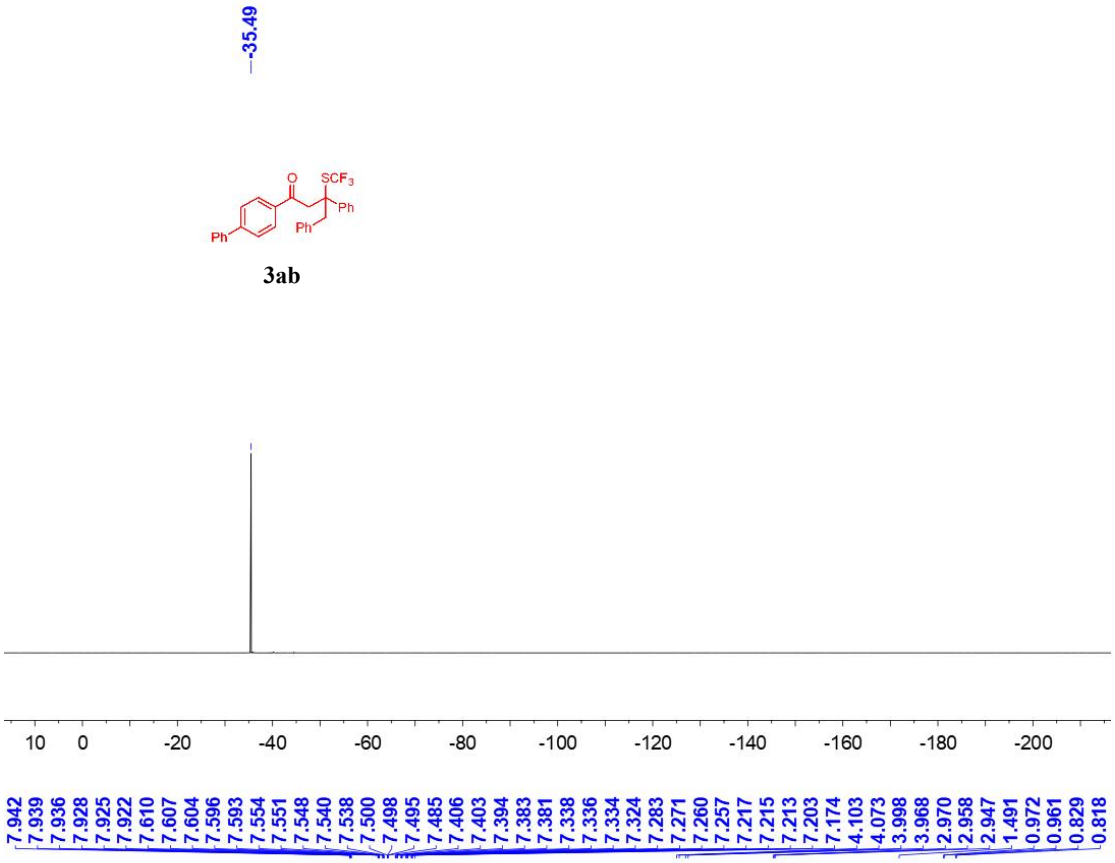
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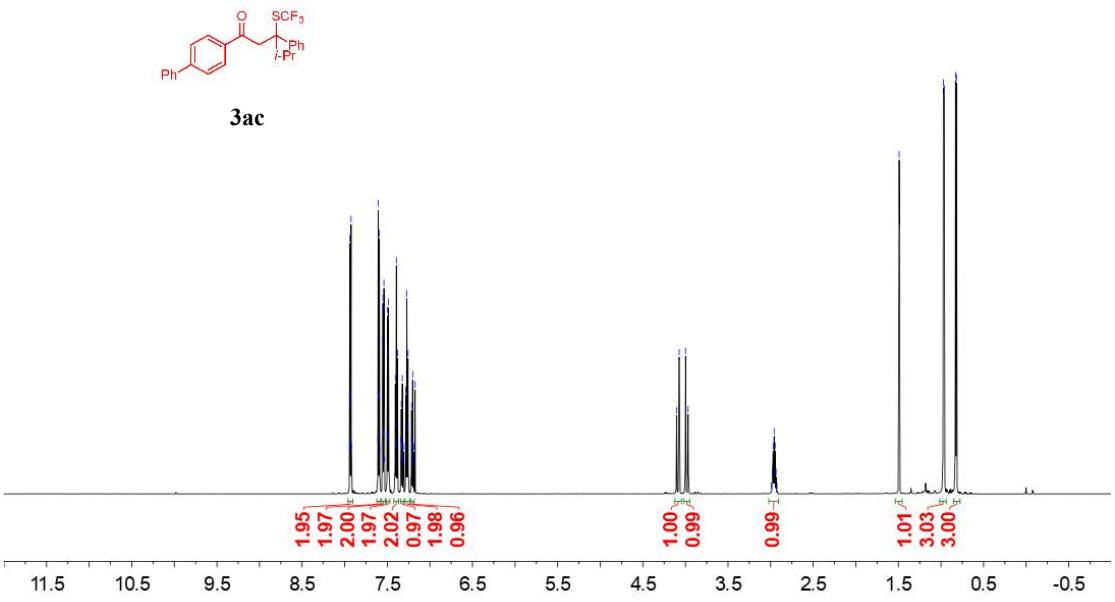
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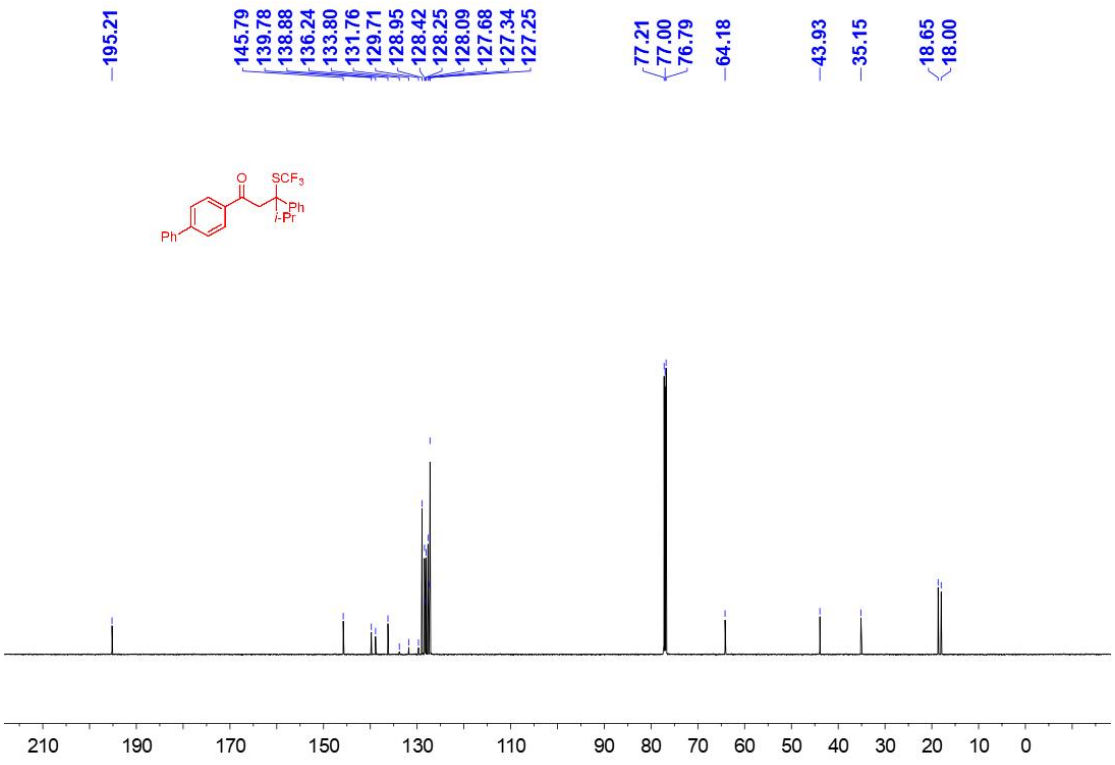
3ac

3ac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18.00
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SCF3
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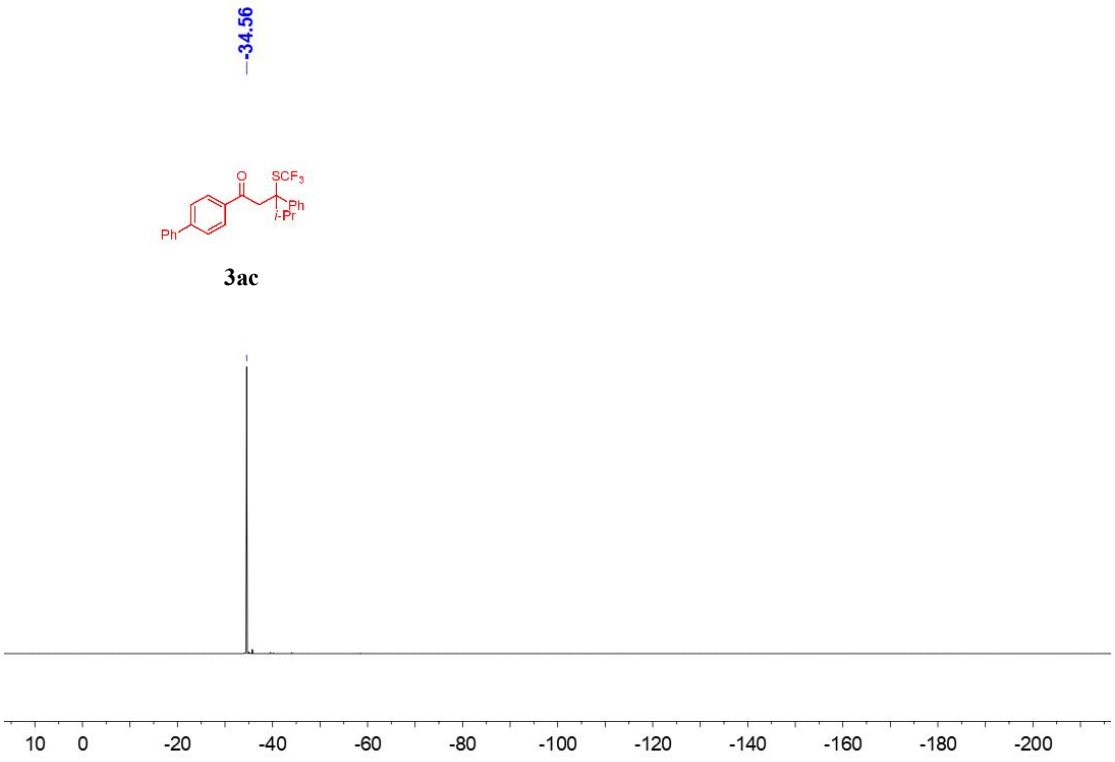
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3ac


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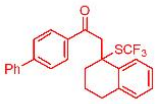
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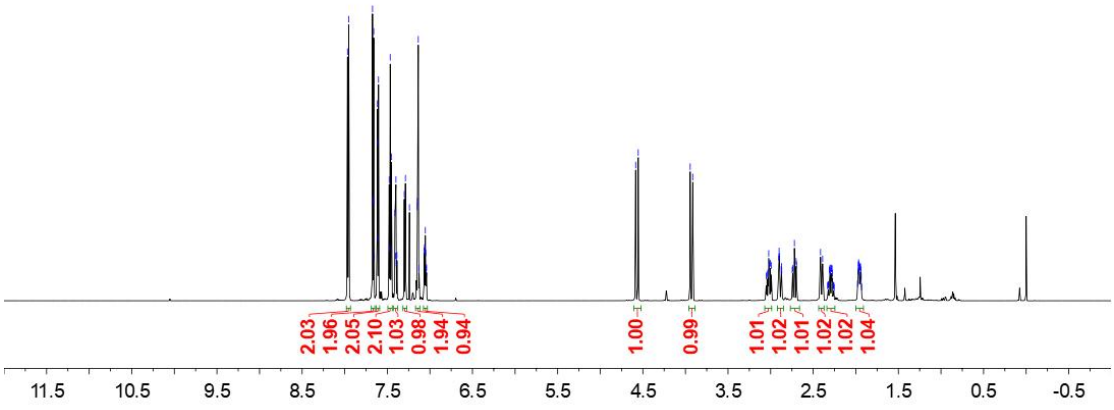
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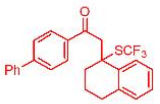
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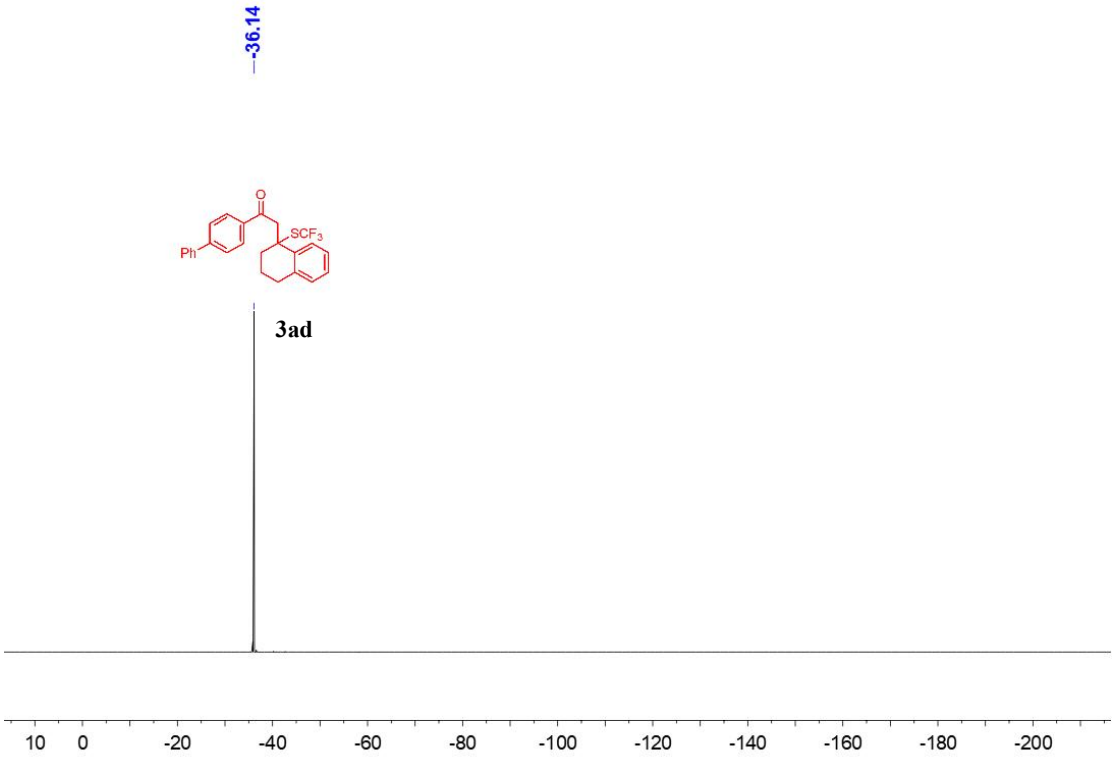
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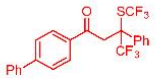
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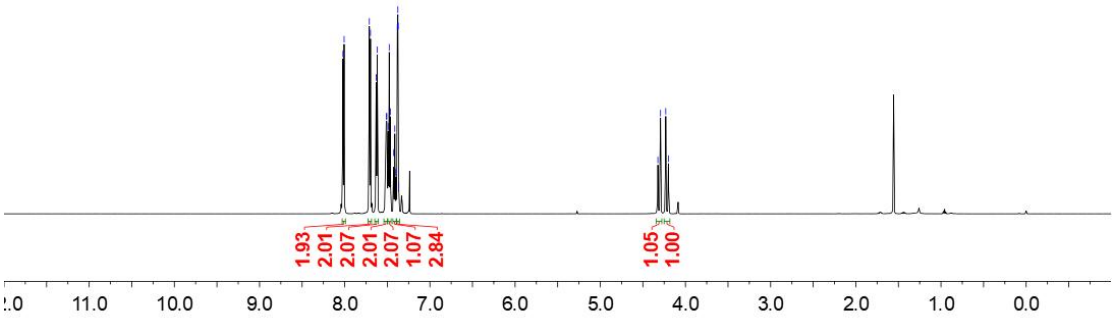
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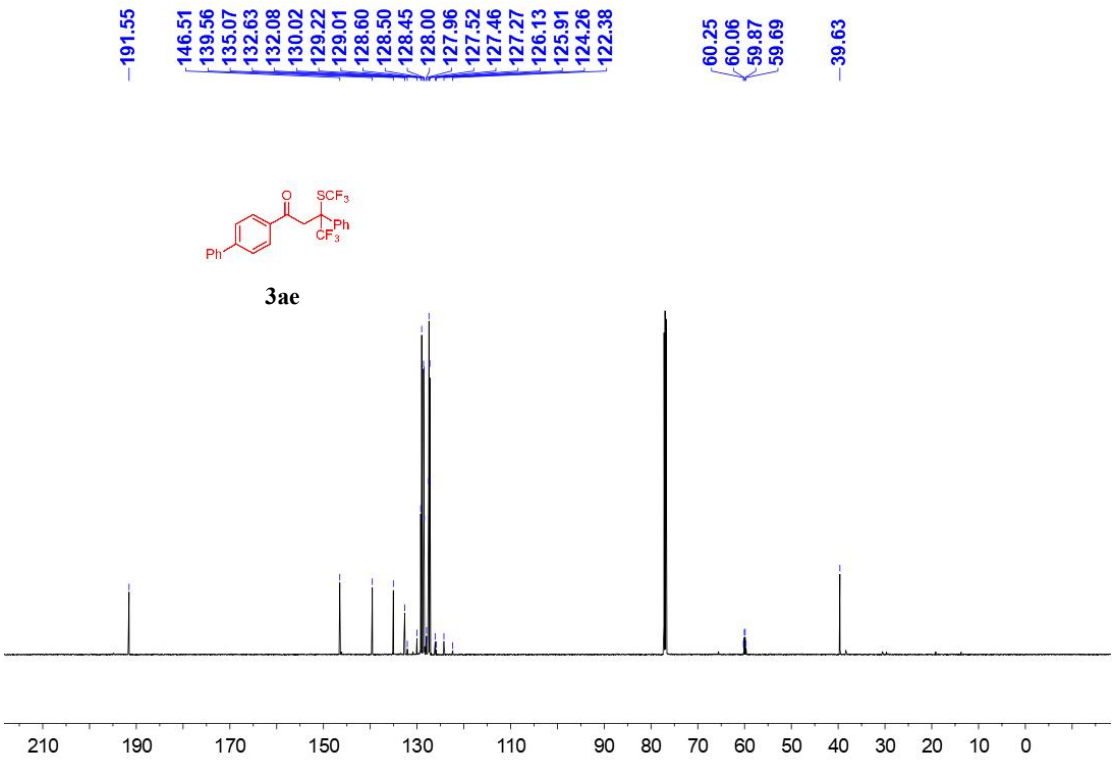
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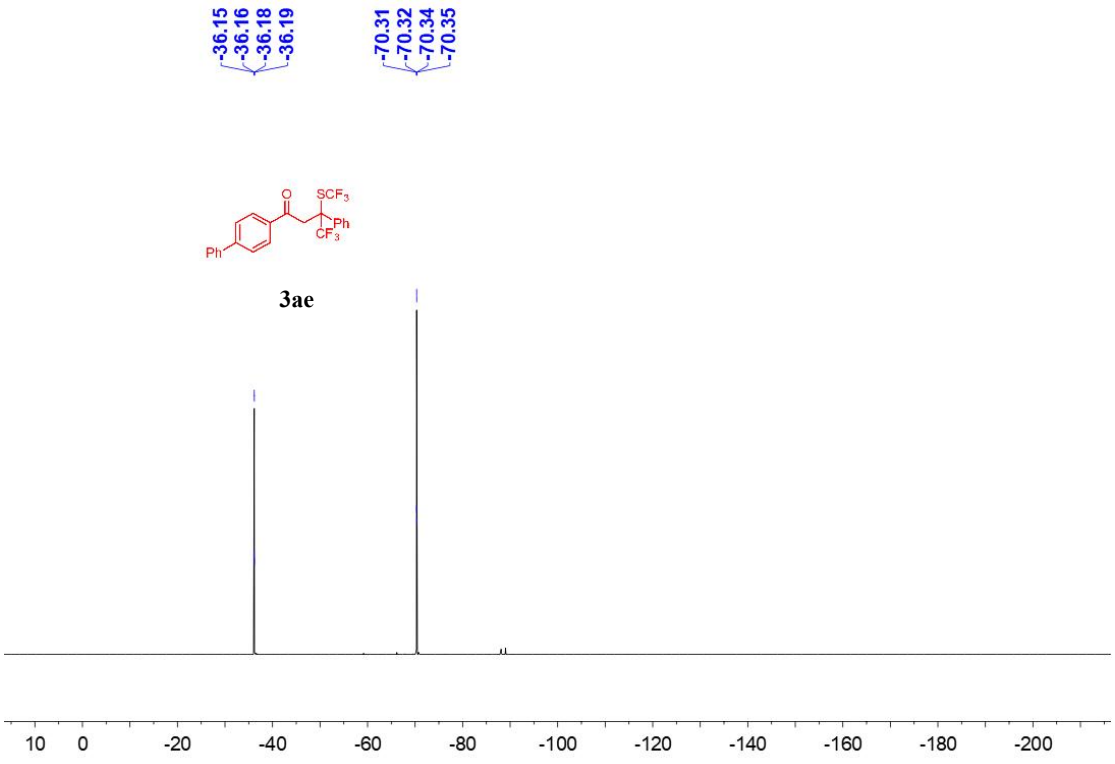
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CF3
Ph
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-200


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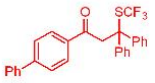
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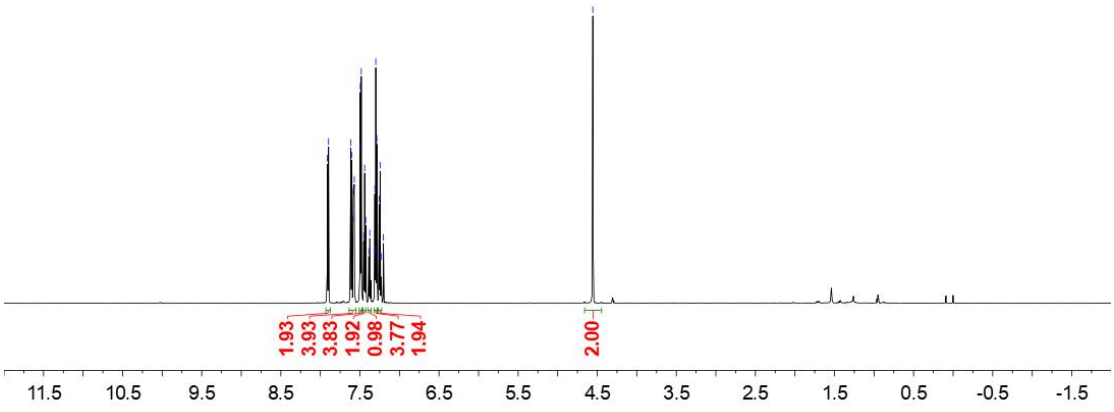
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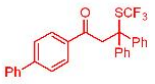
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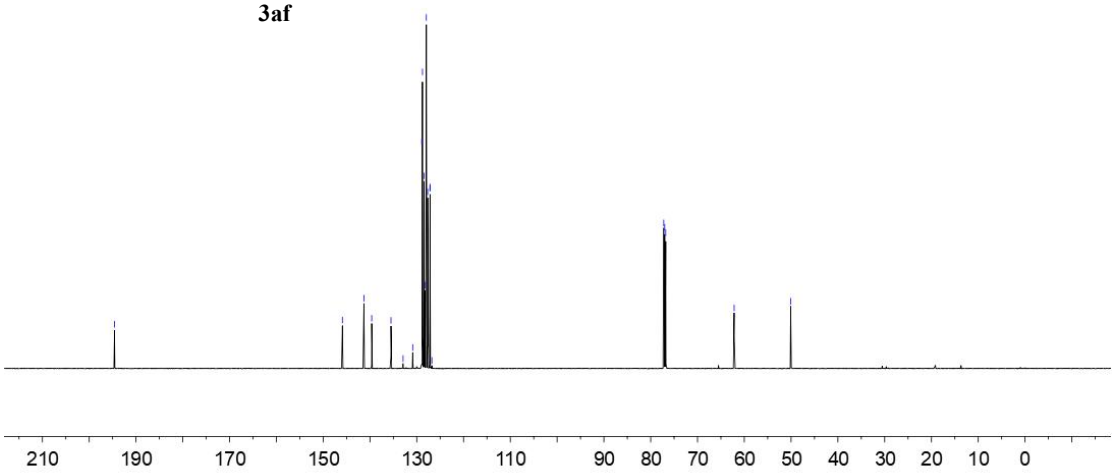
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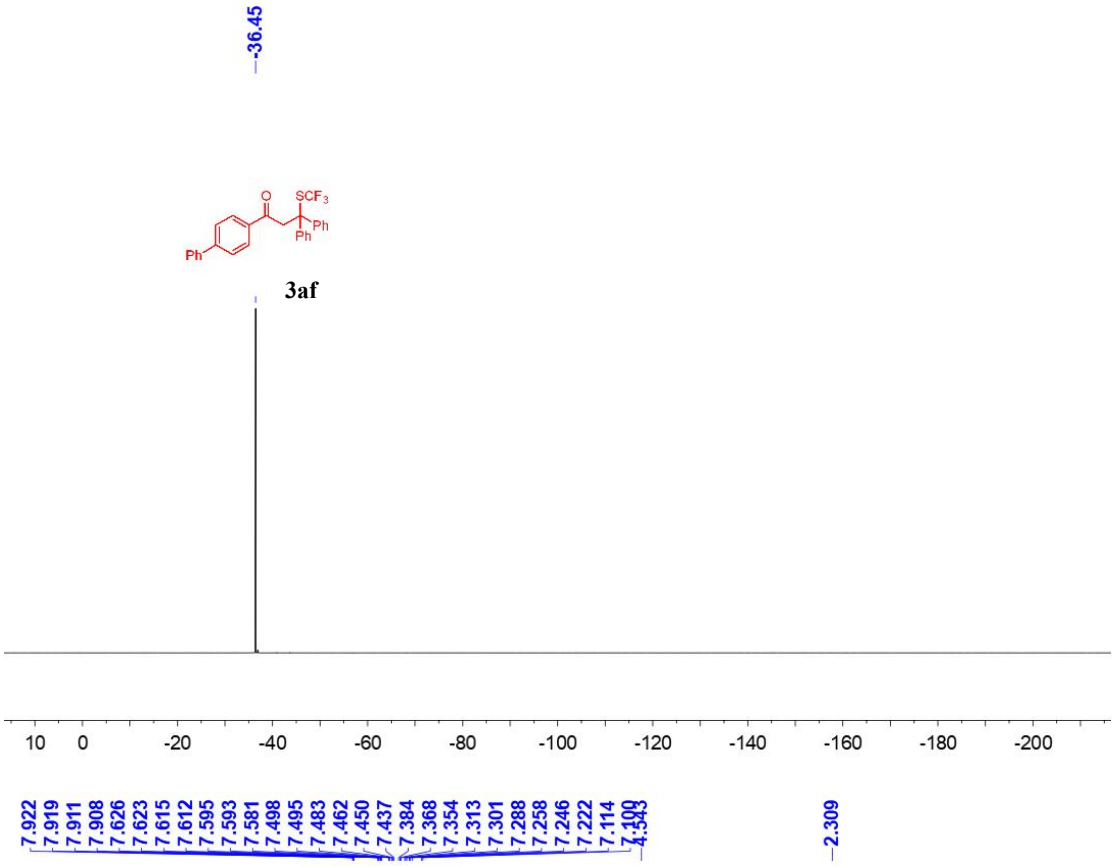
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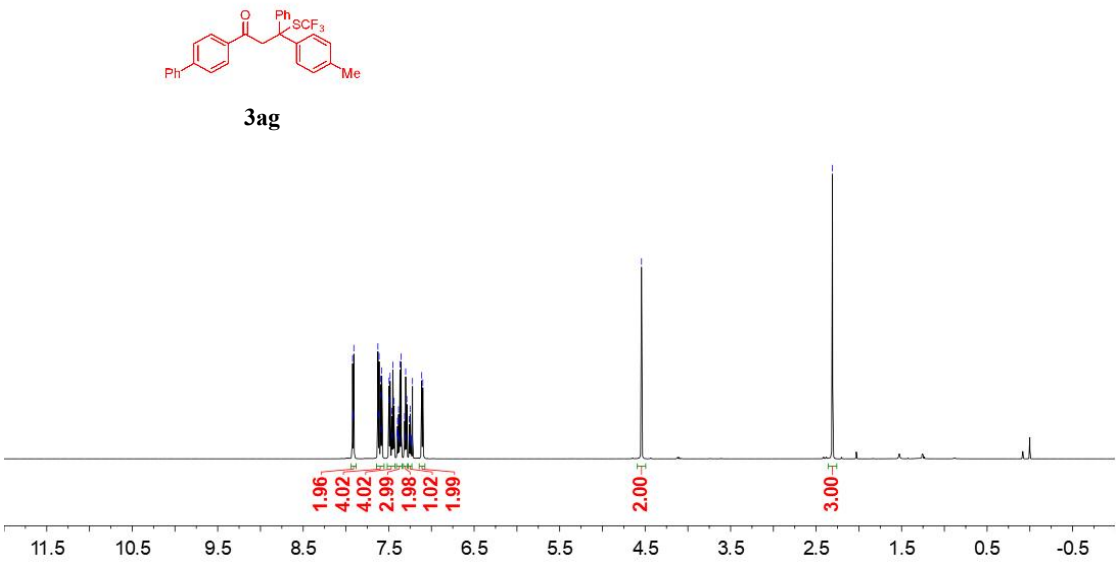
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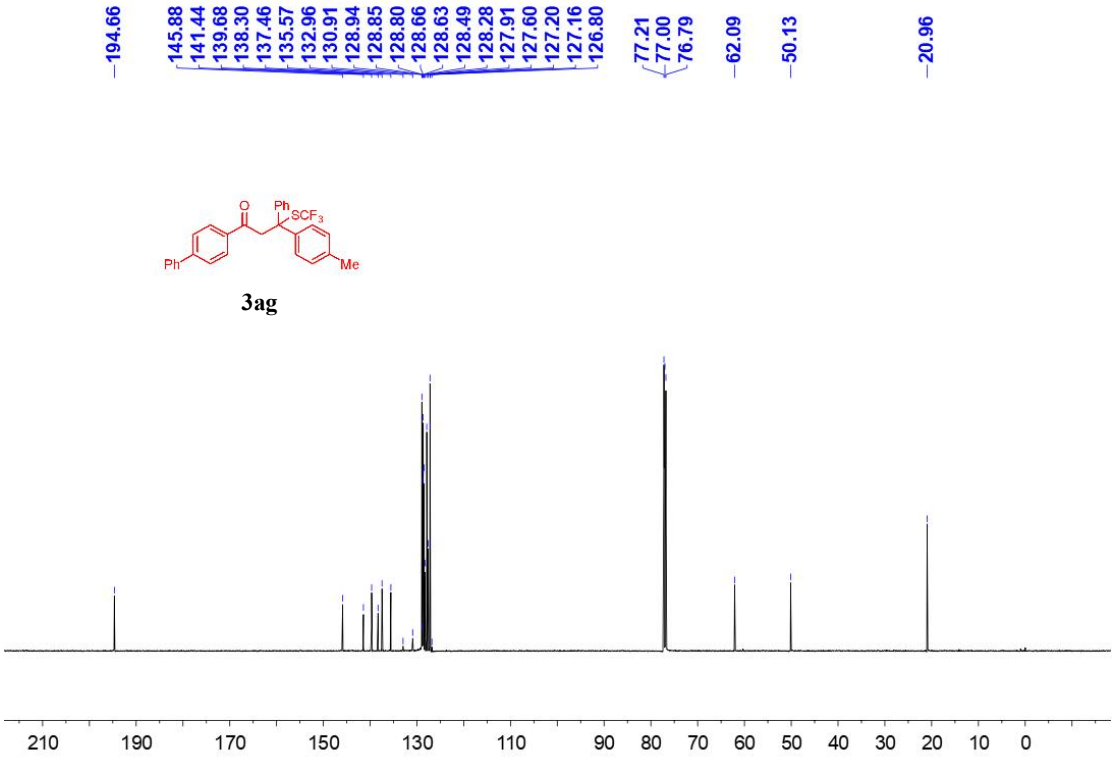
3ag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O
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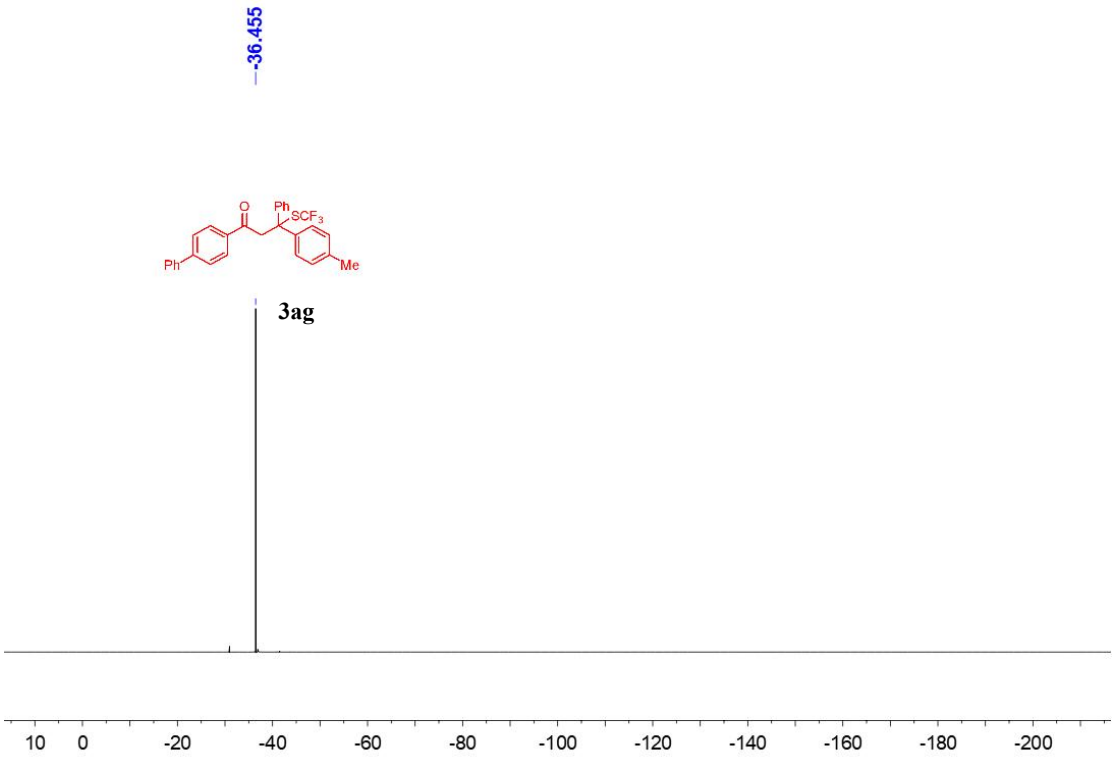
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3ag


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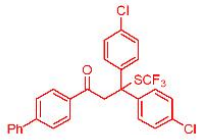
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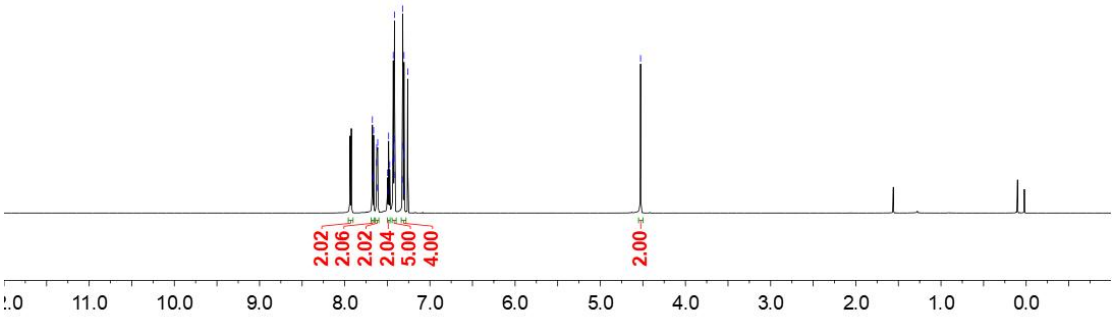
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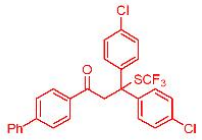
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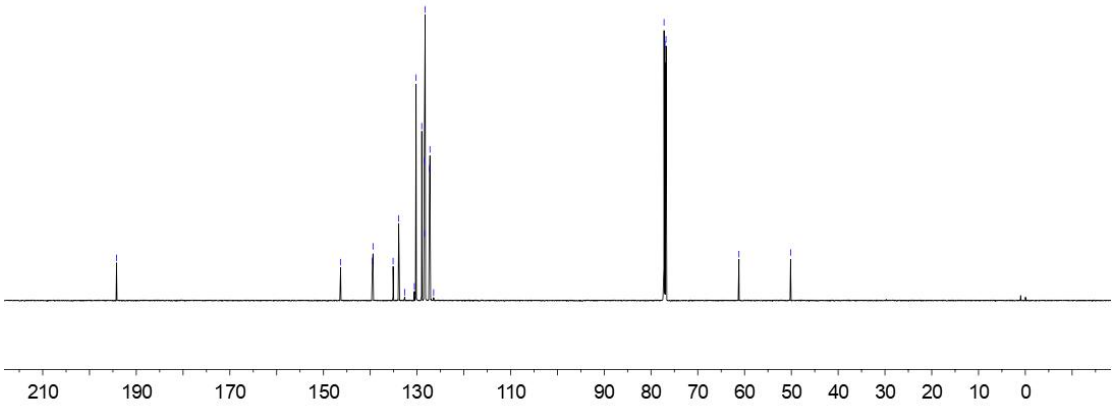
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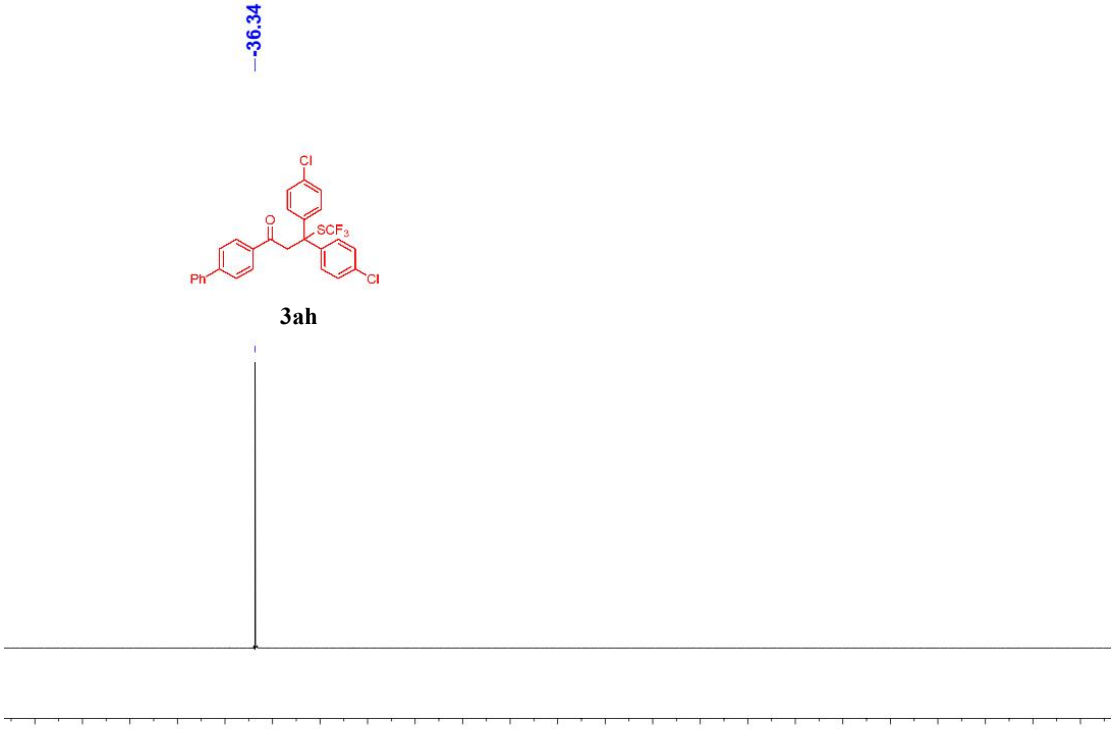
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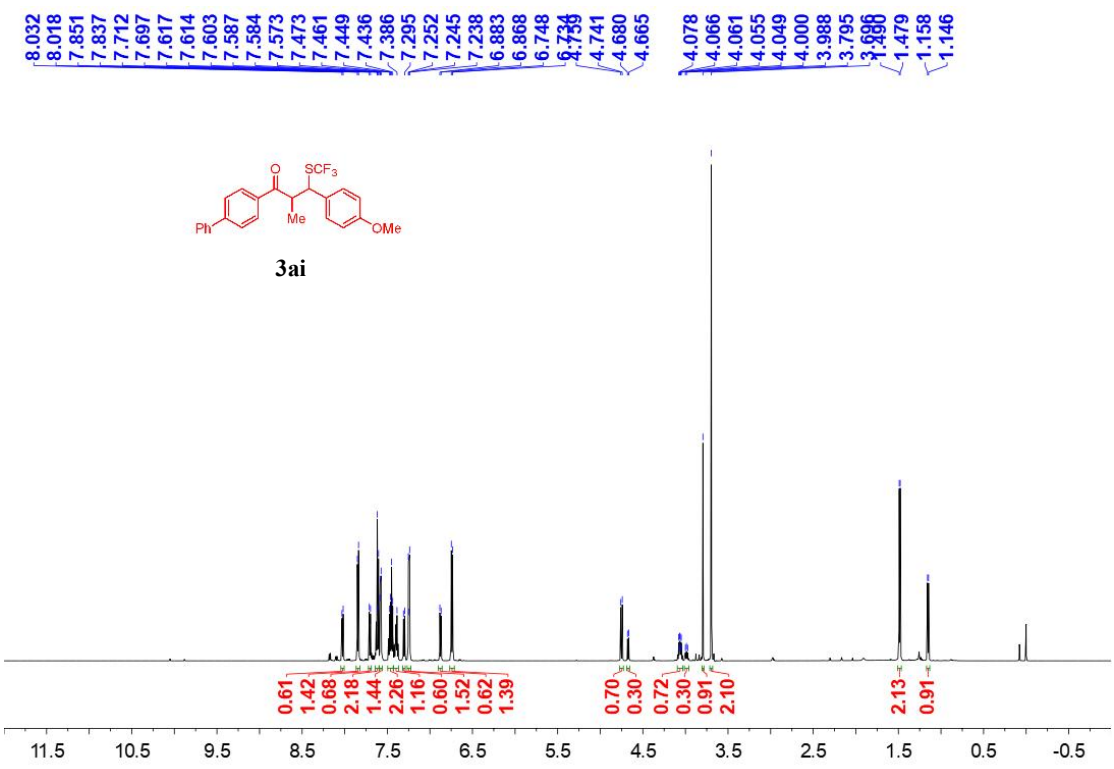
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SCF3
Me
Ph
OMe
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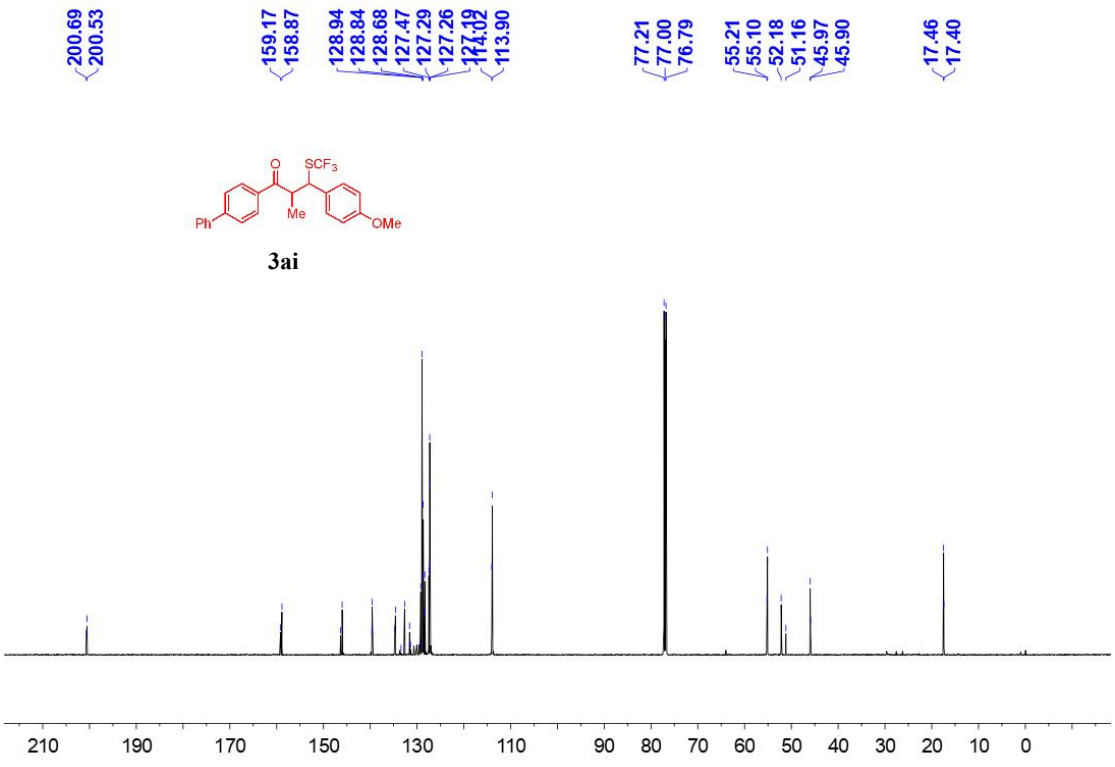
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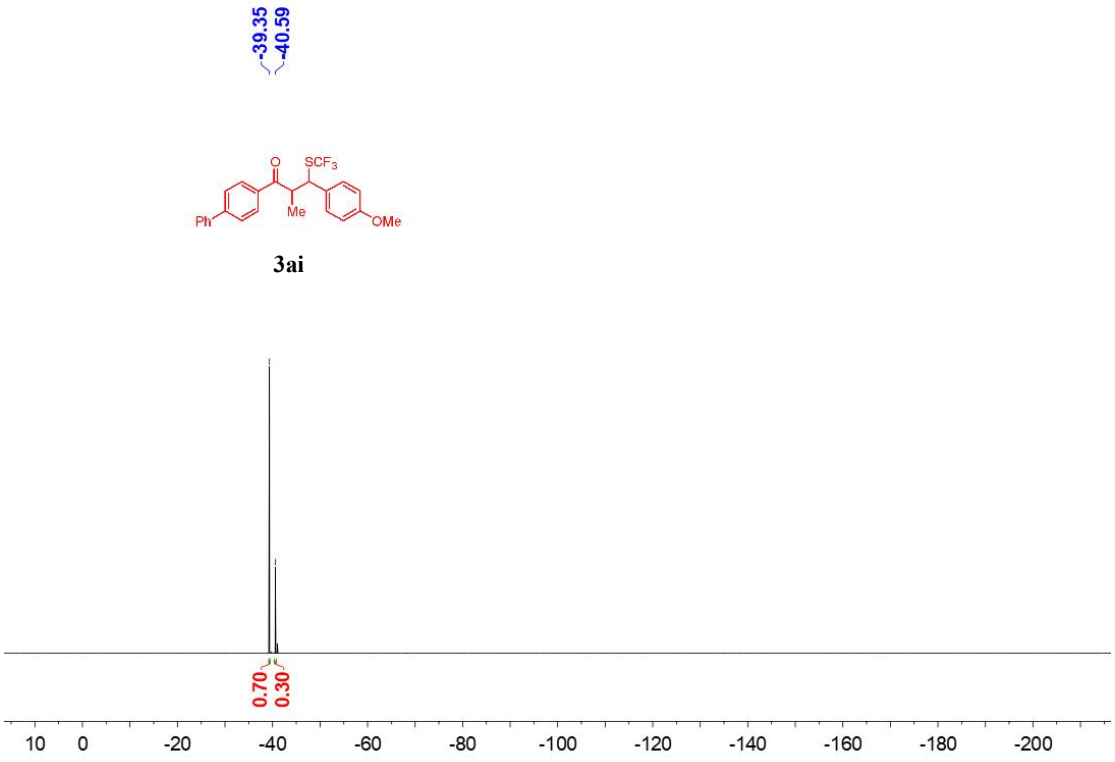
3ai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3ai


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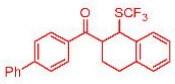
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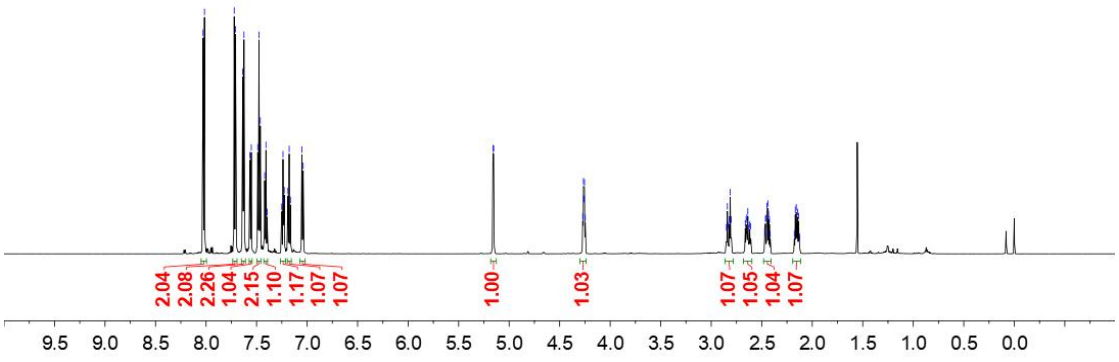
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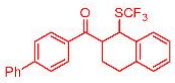
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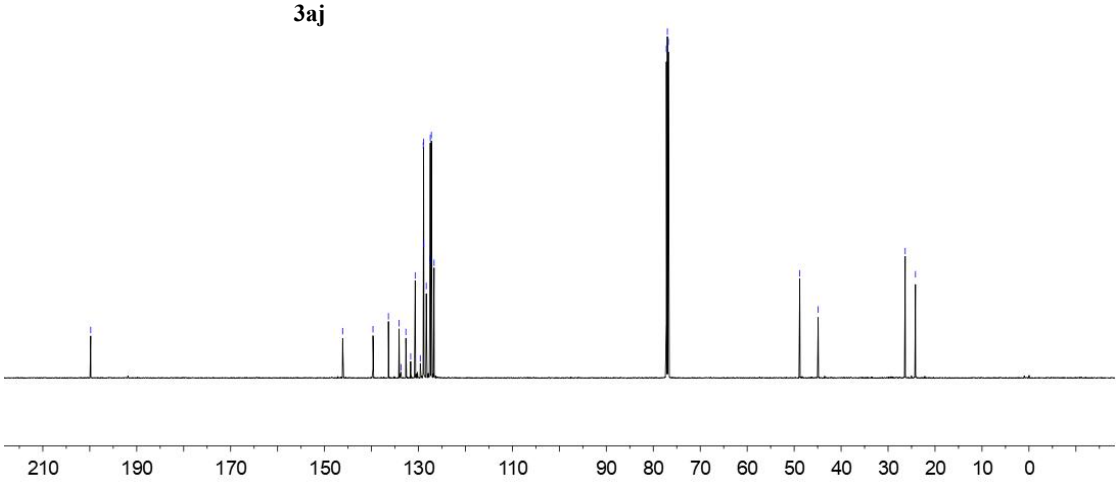
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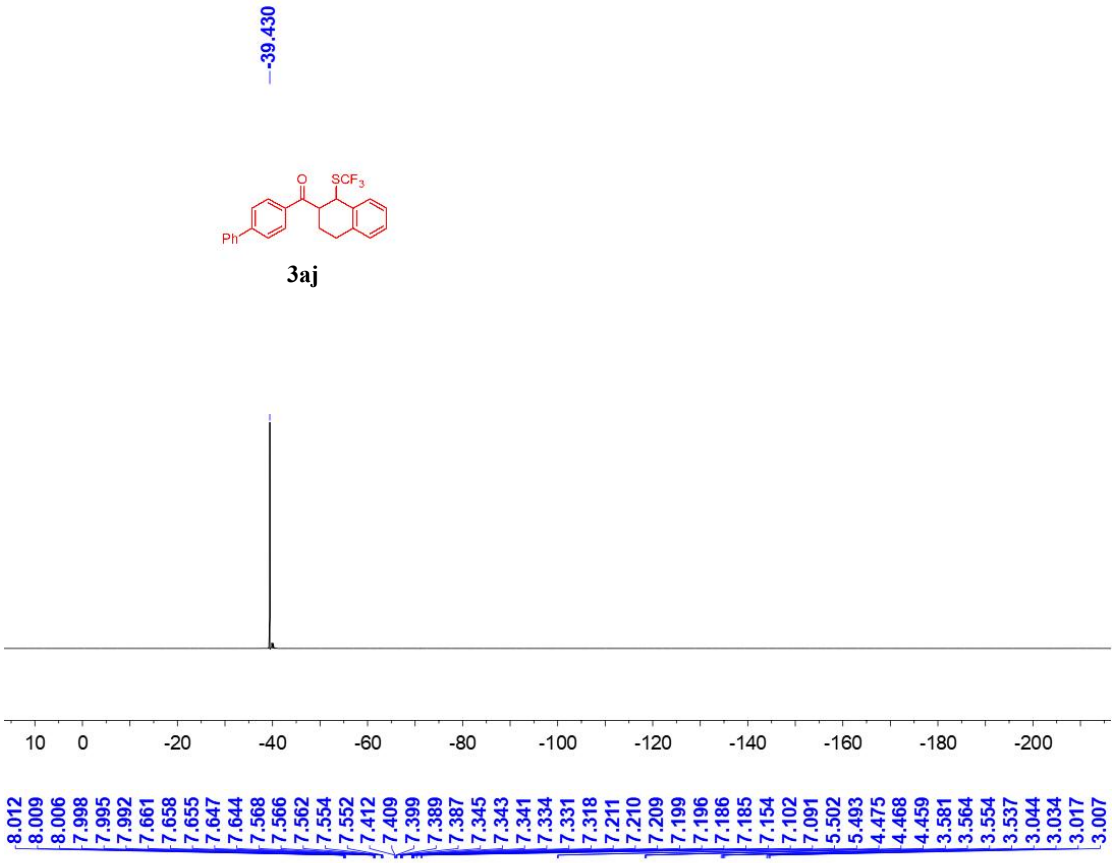
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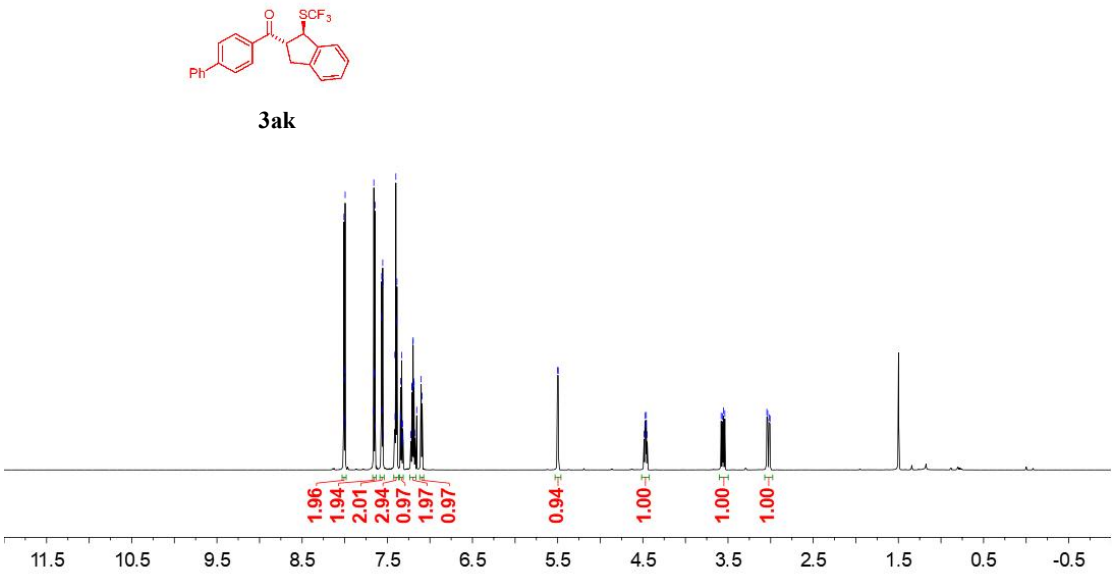
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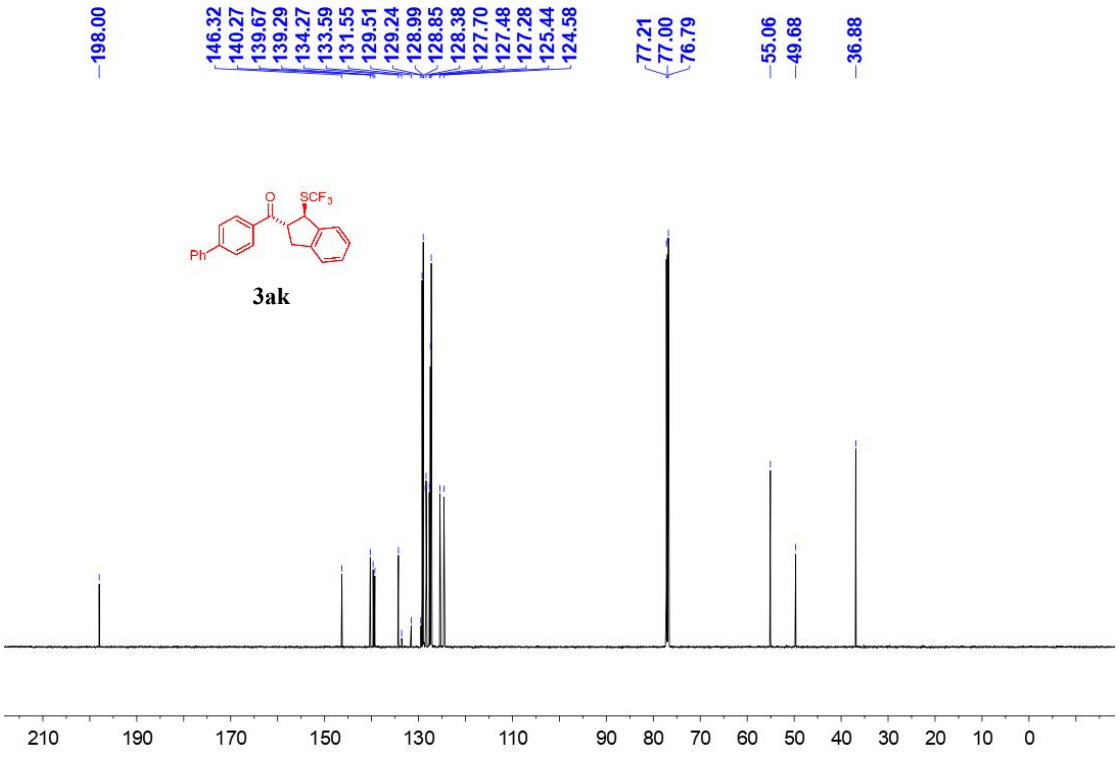
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49.68
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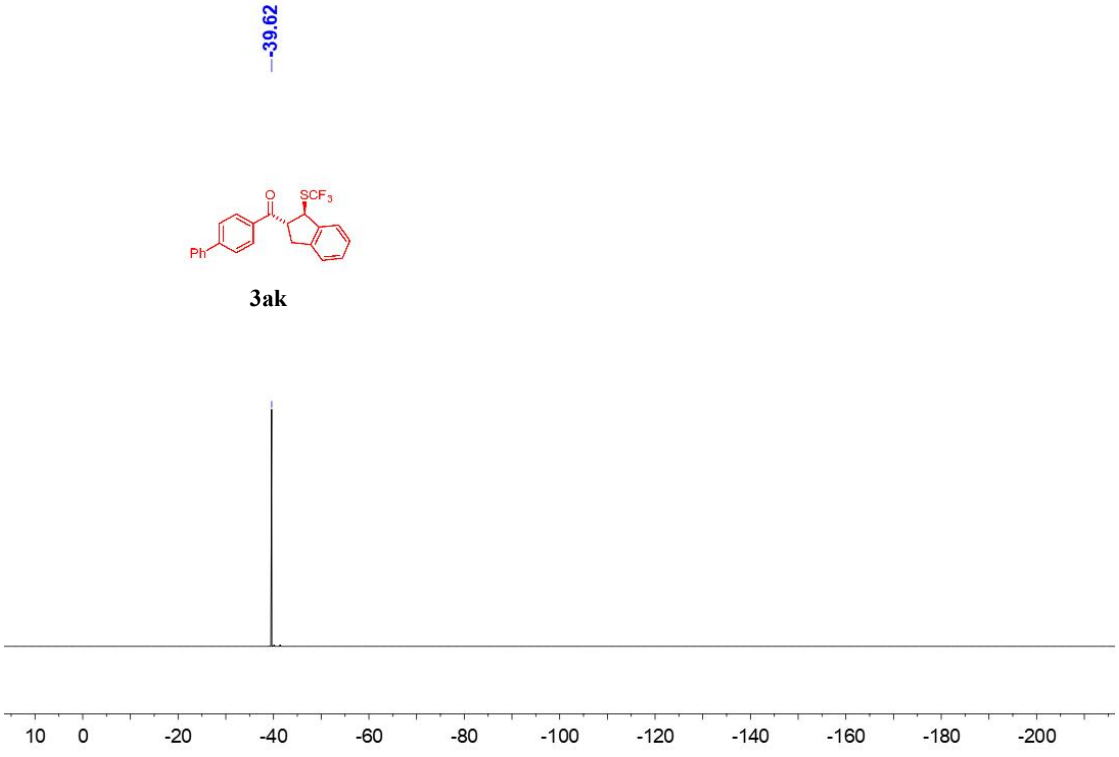
3ak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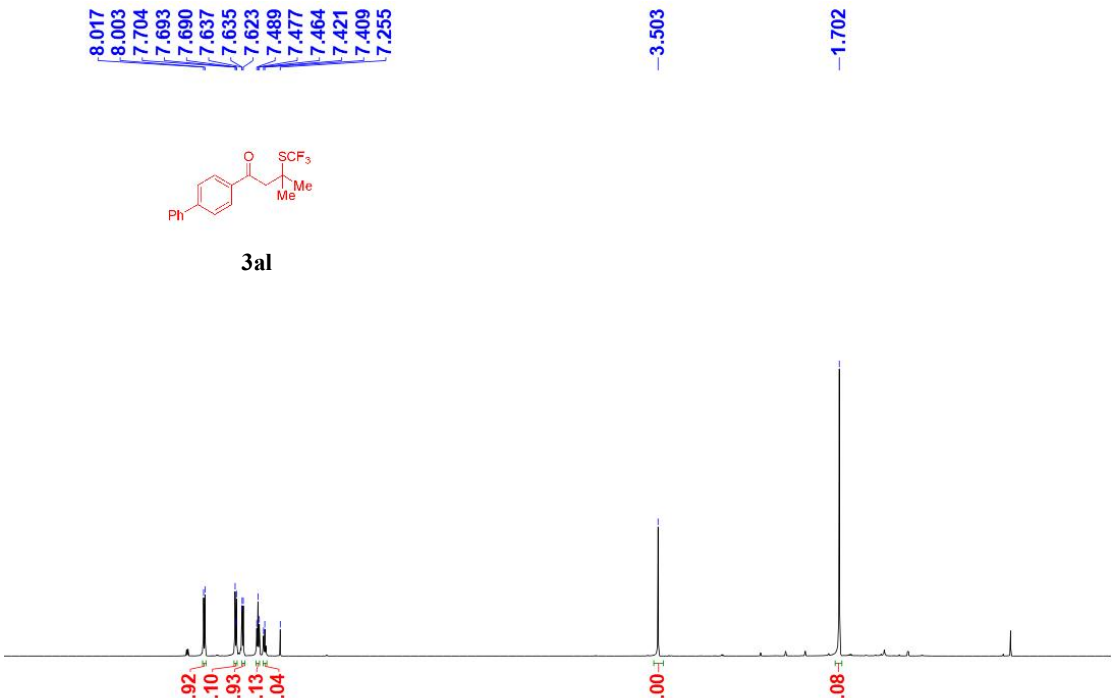
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2.00
6.08
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

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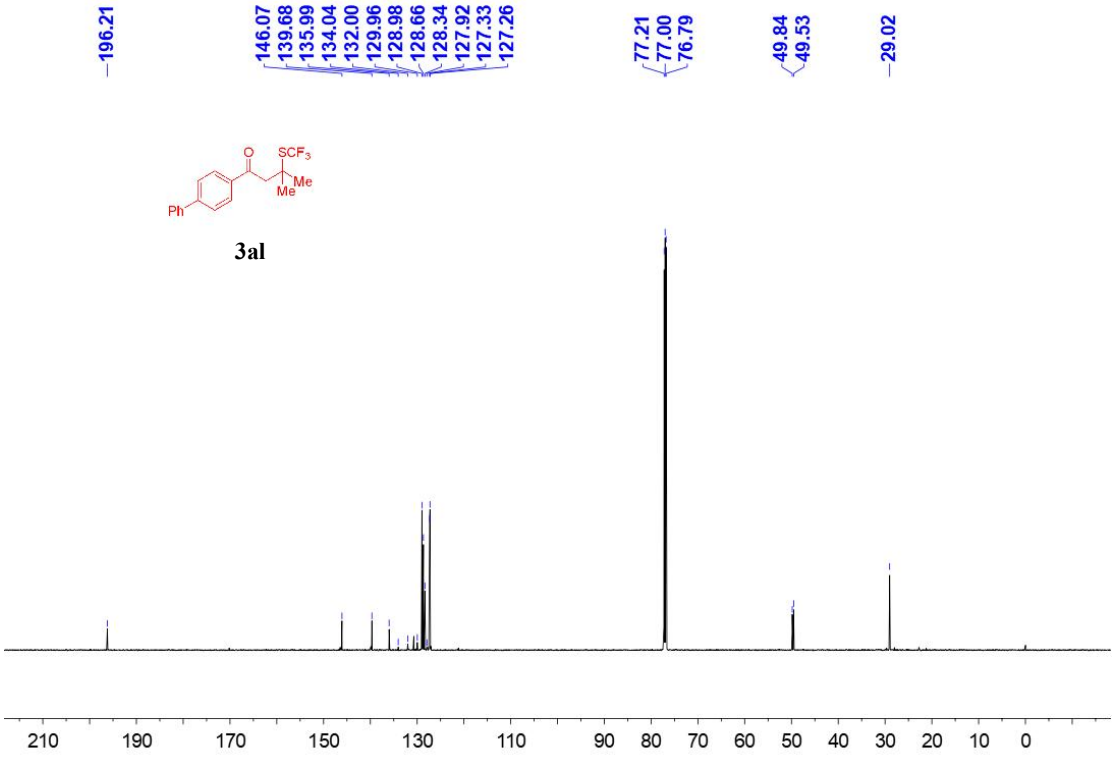
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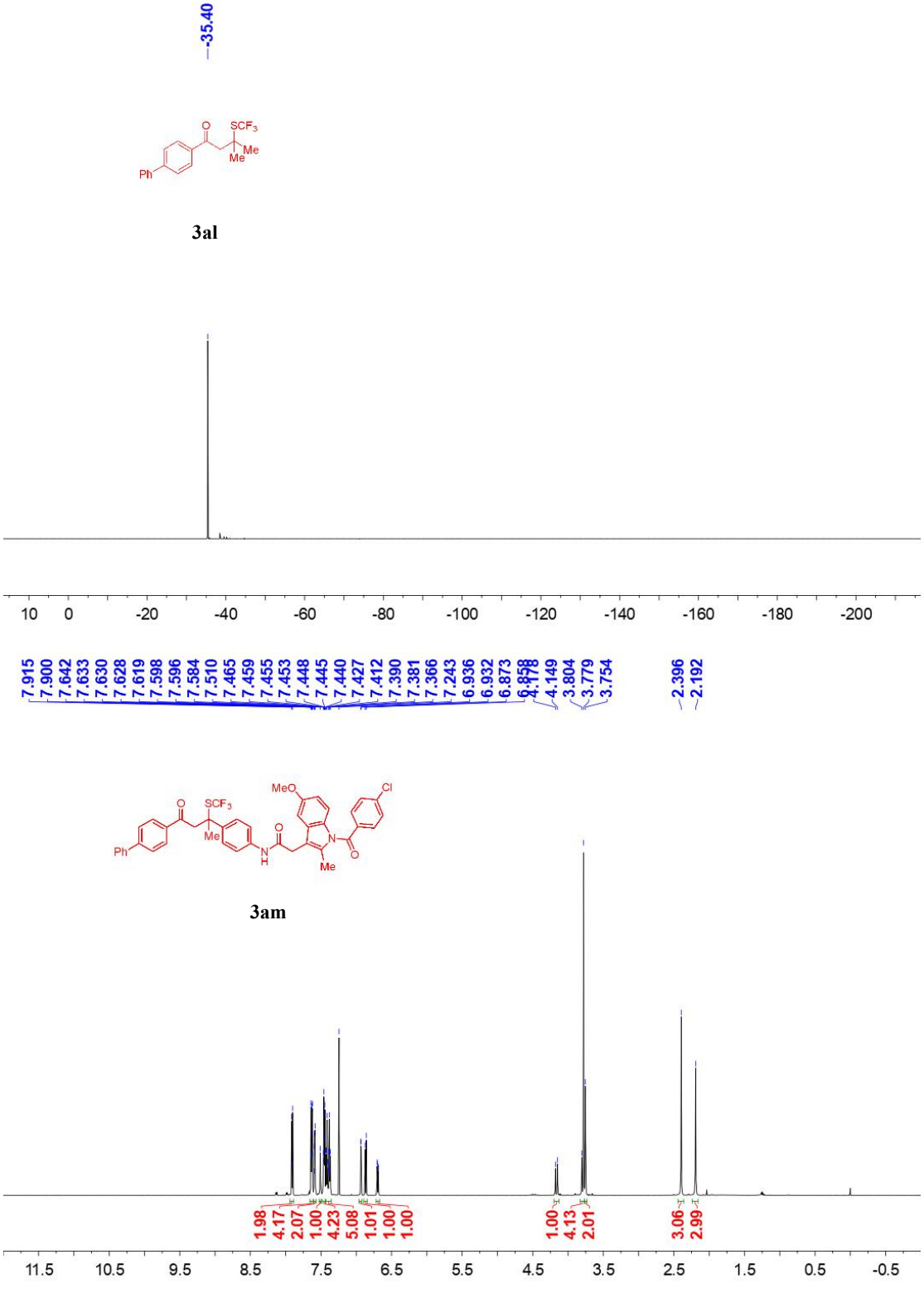
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SCF3
Me
Ph
N
H
O
N
O
Me
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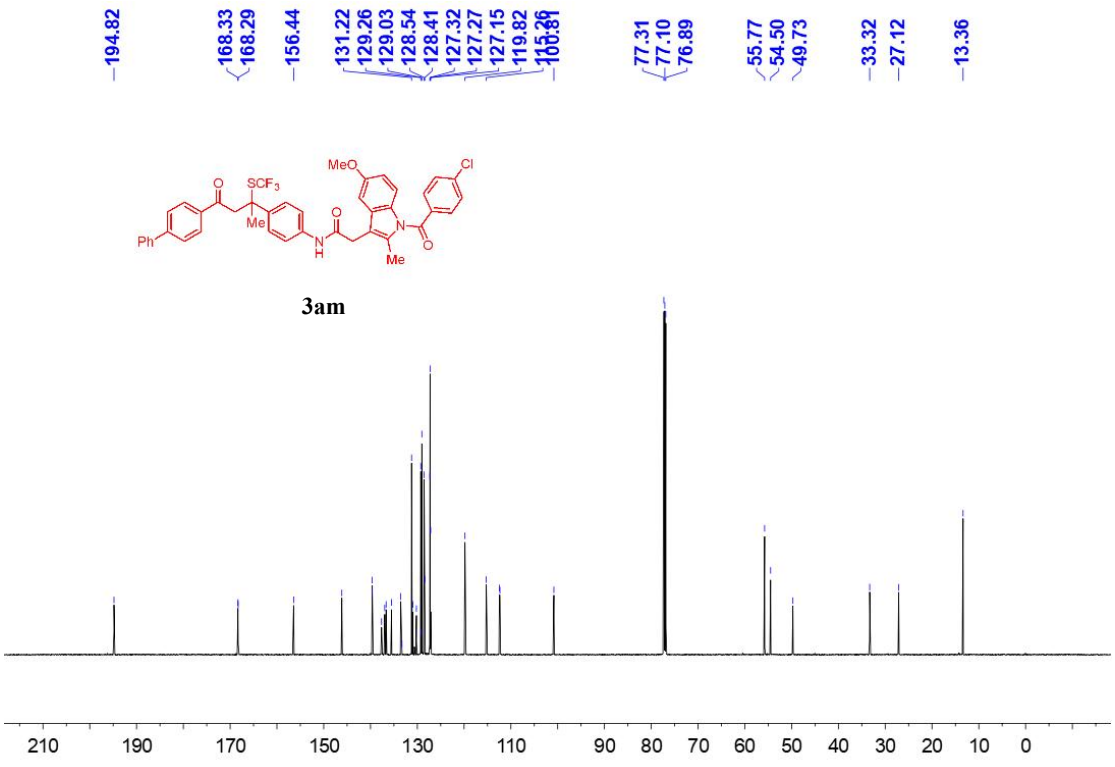
3al

3am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33.32
27.12
13.36
MeO
Cl
SCF3
Me
Ph
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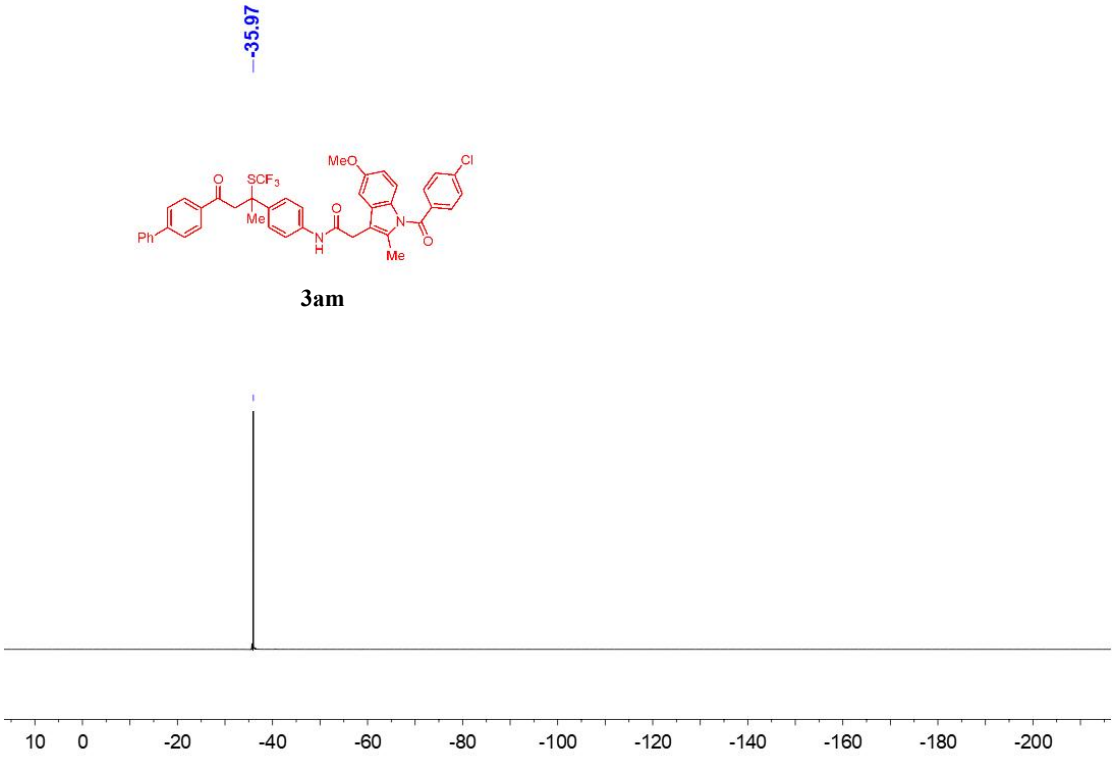
10
0

3am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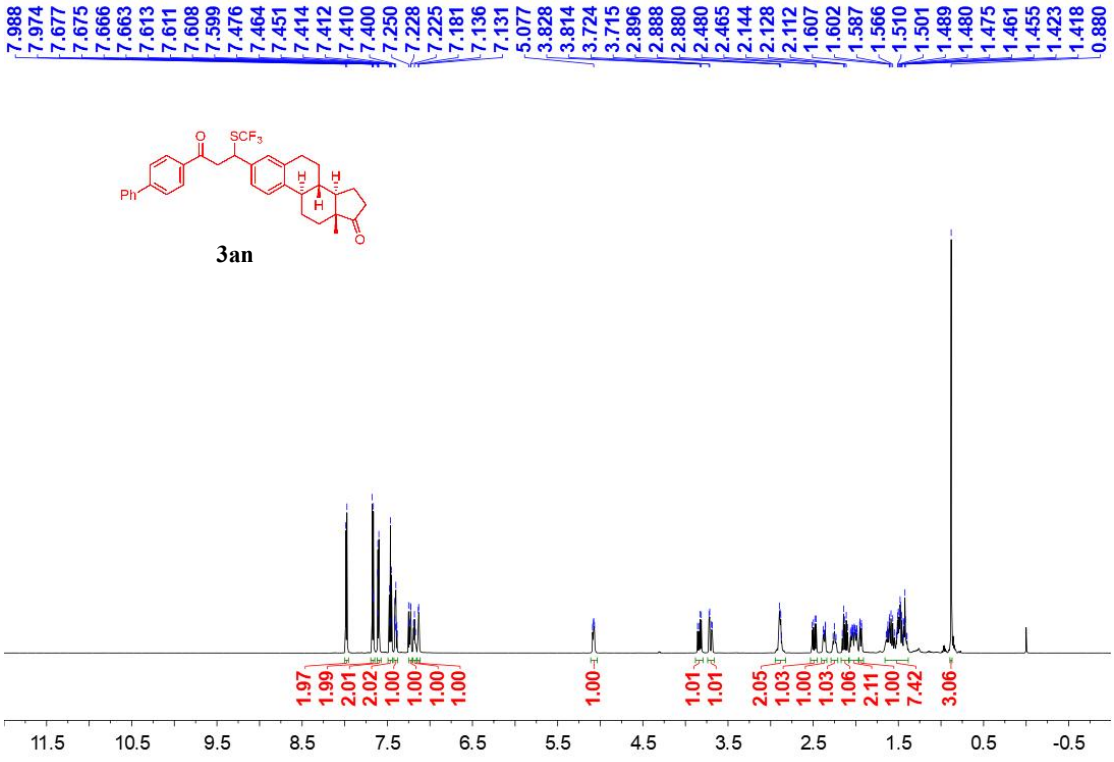
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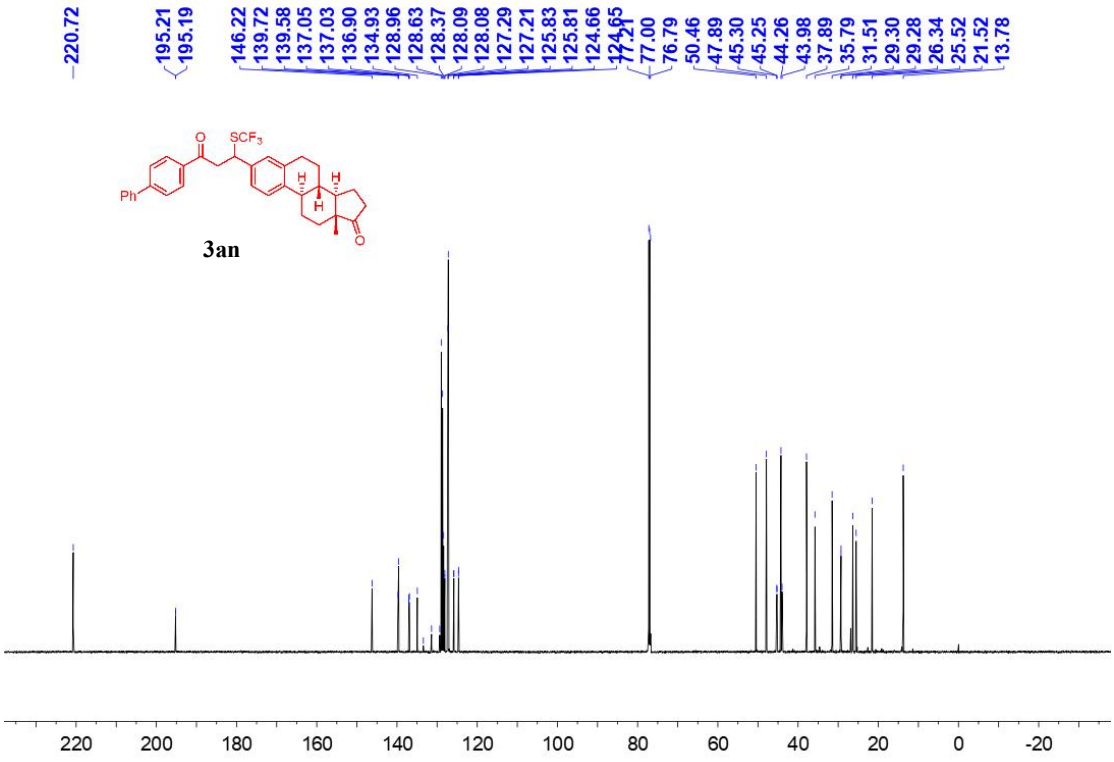
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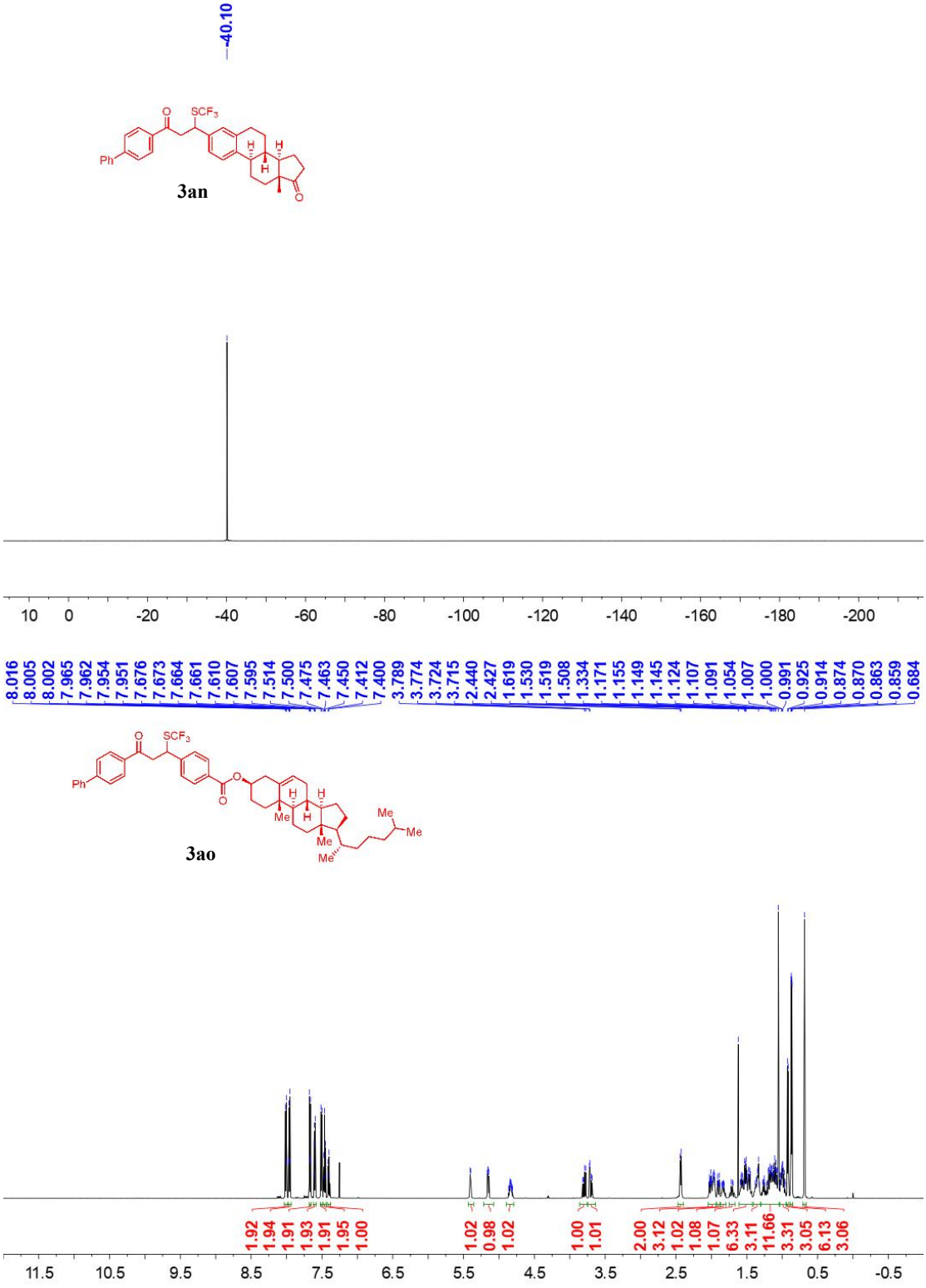
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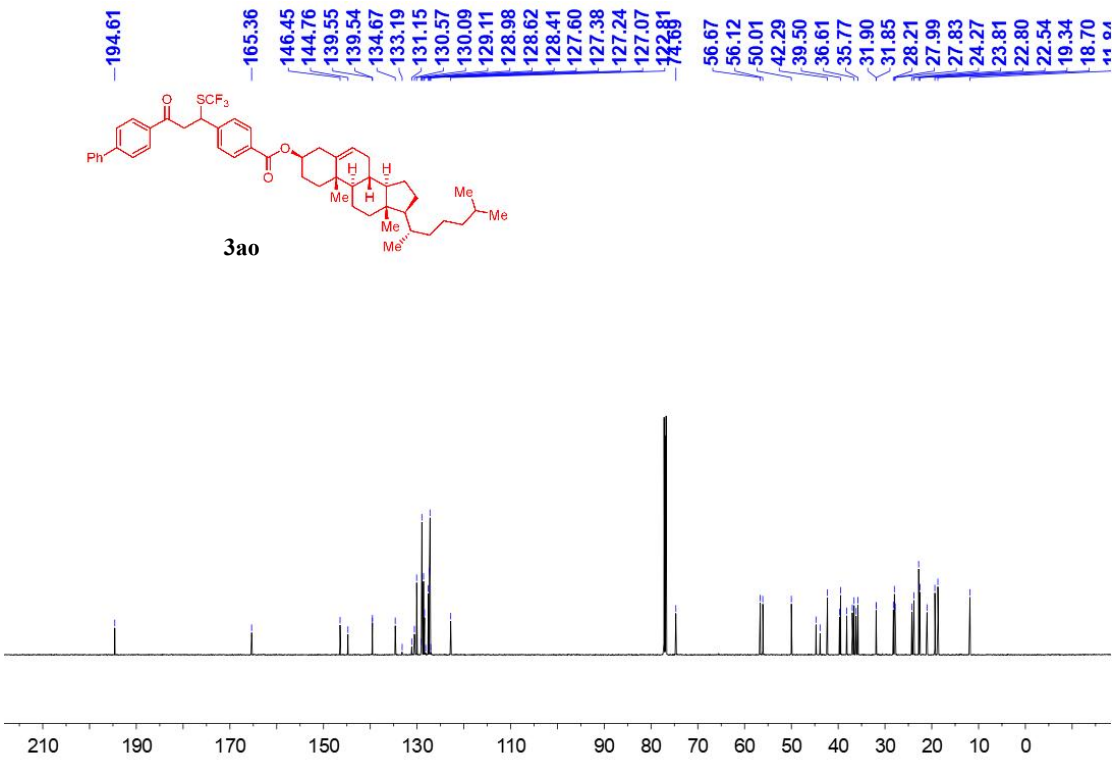
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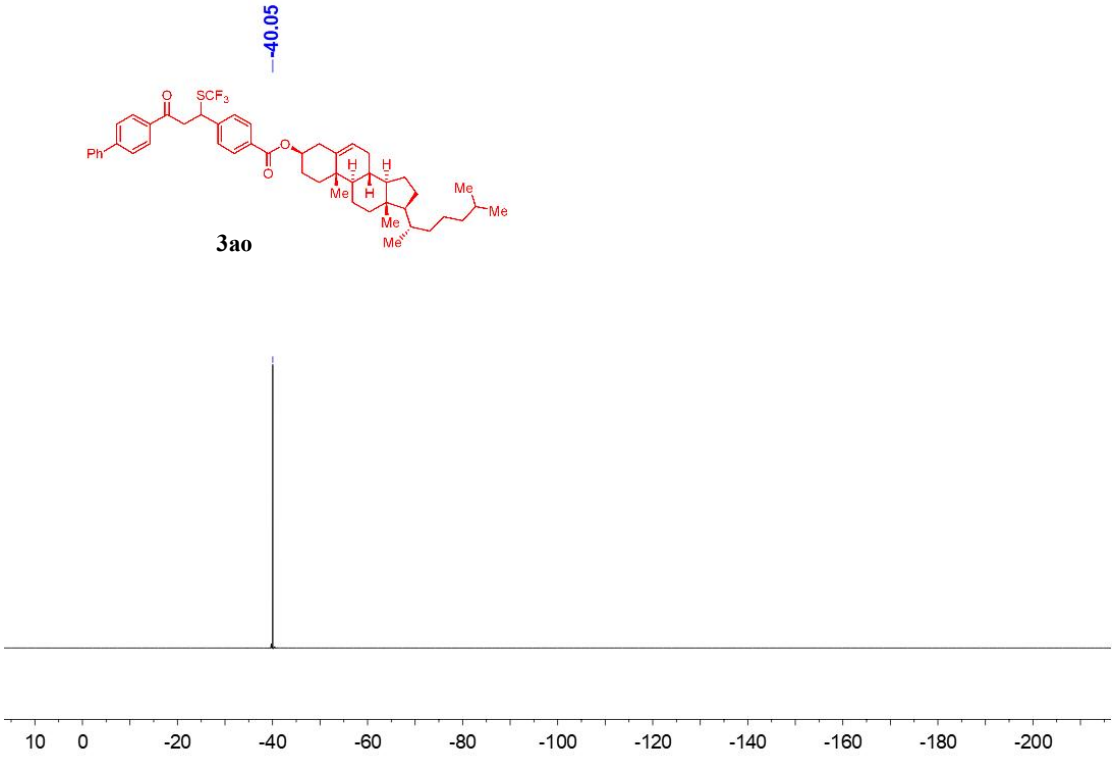
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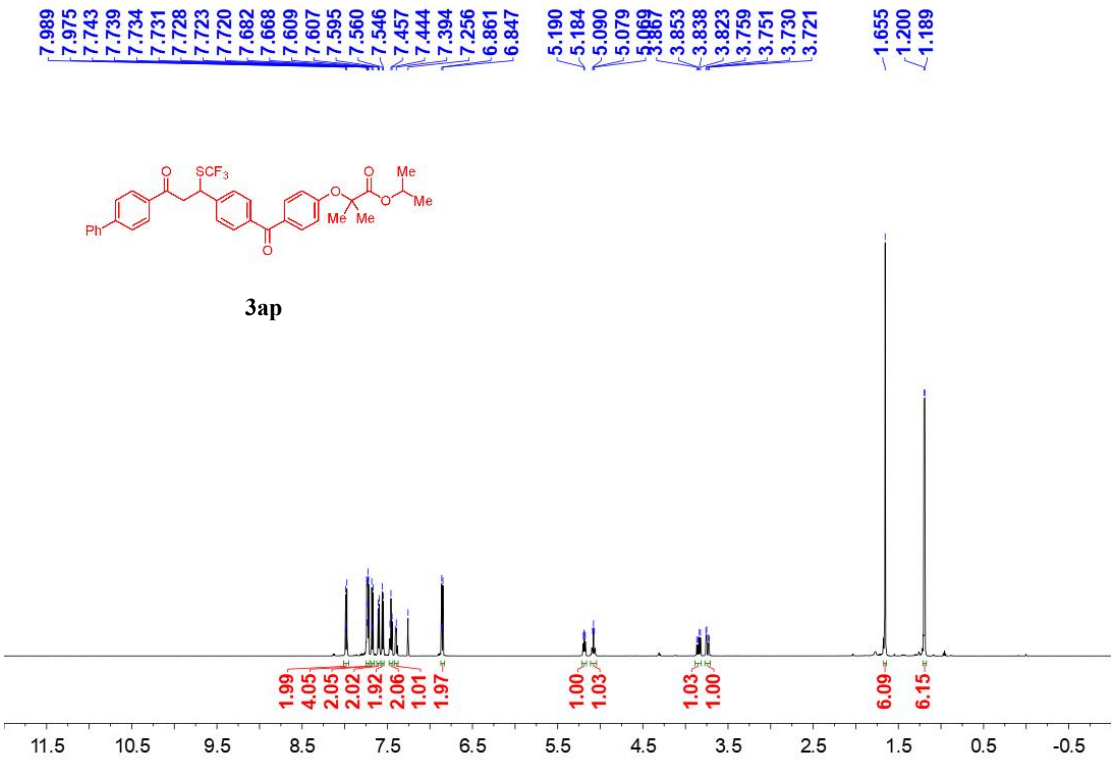
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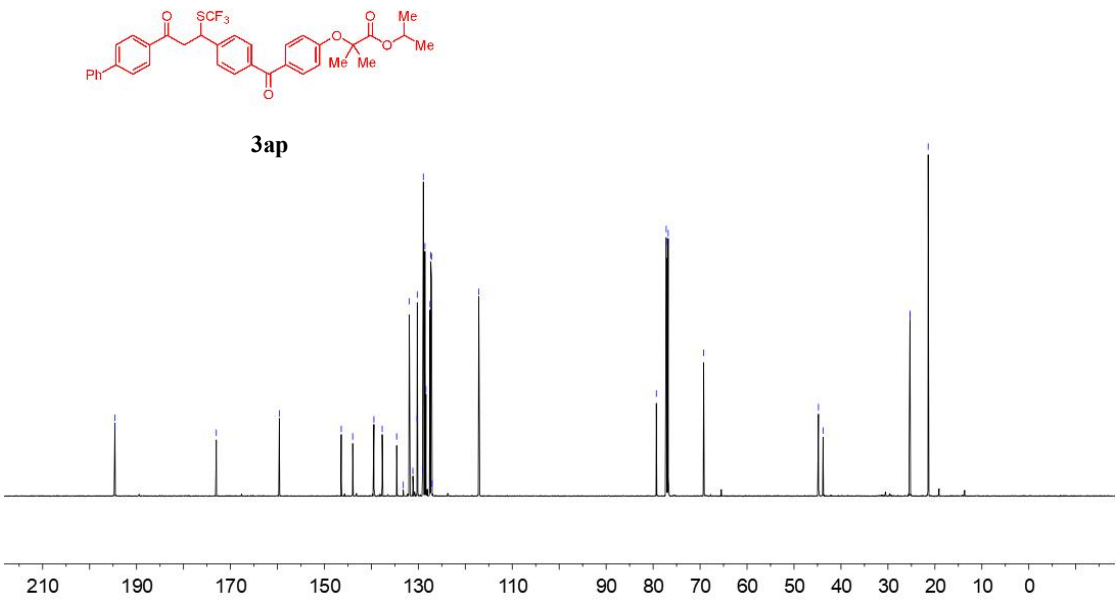
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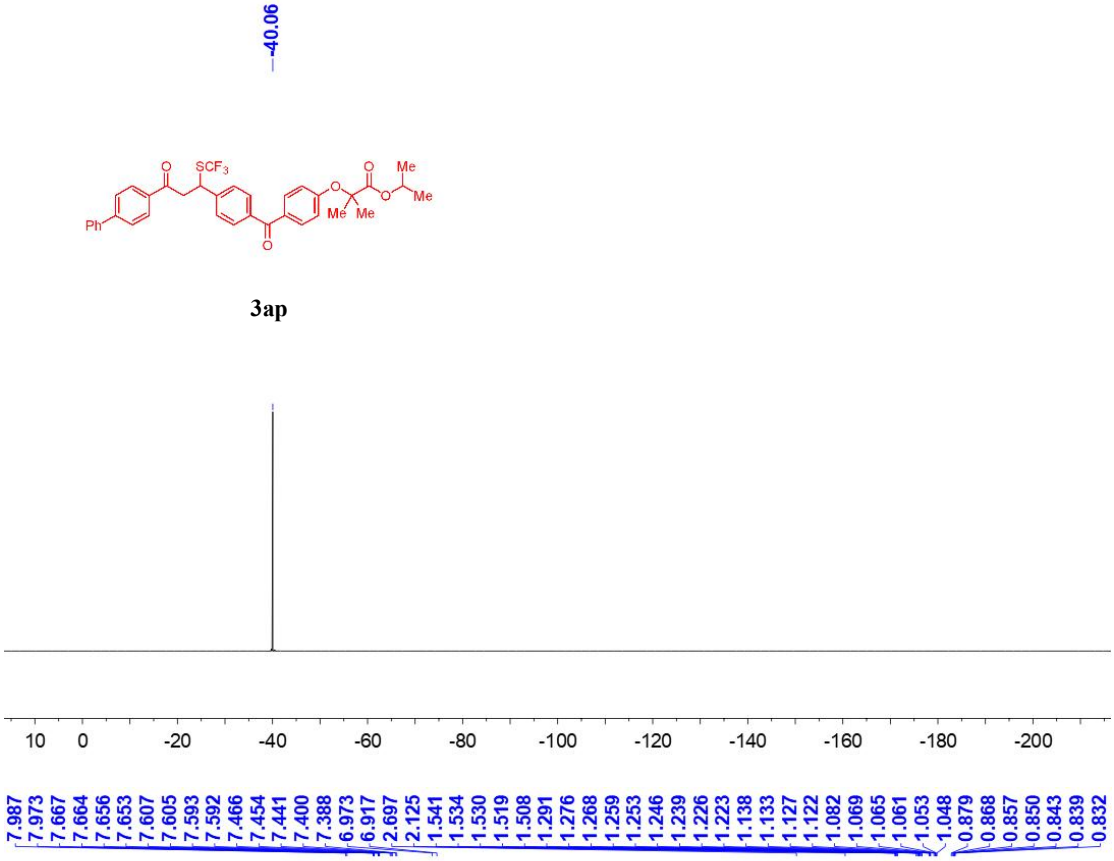
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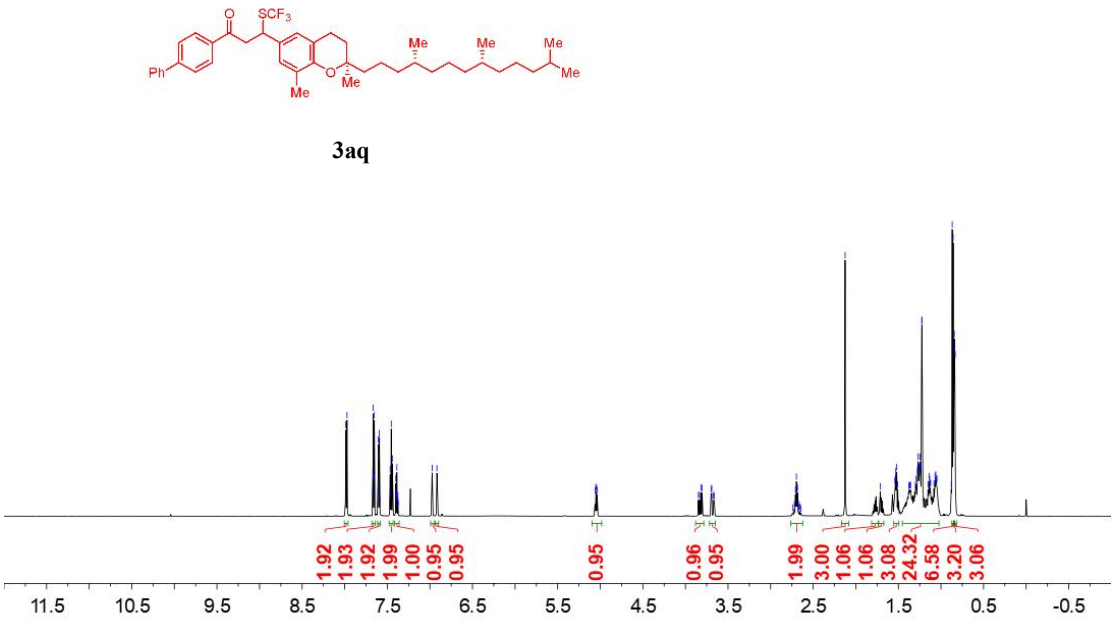
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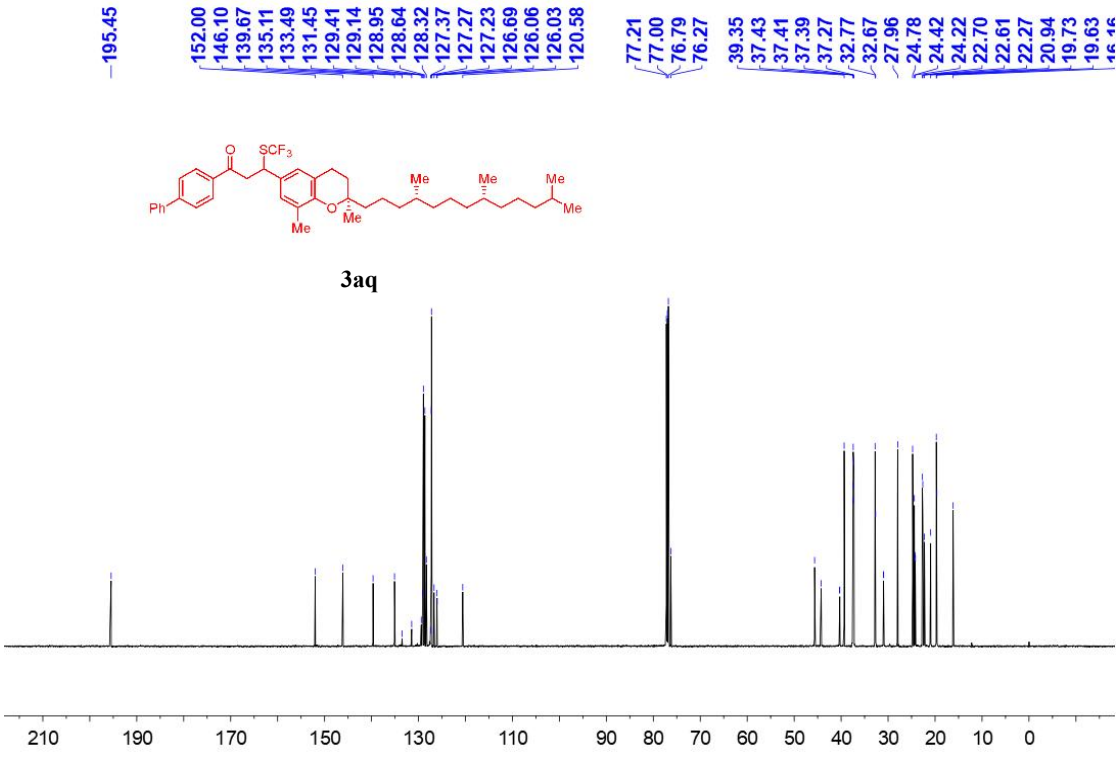
3aq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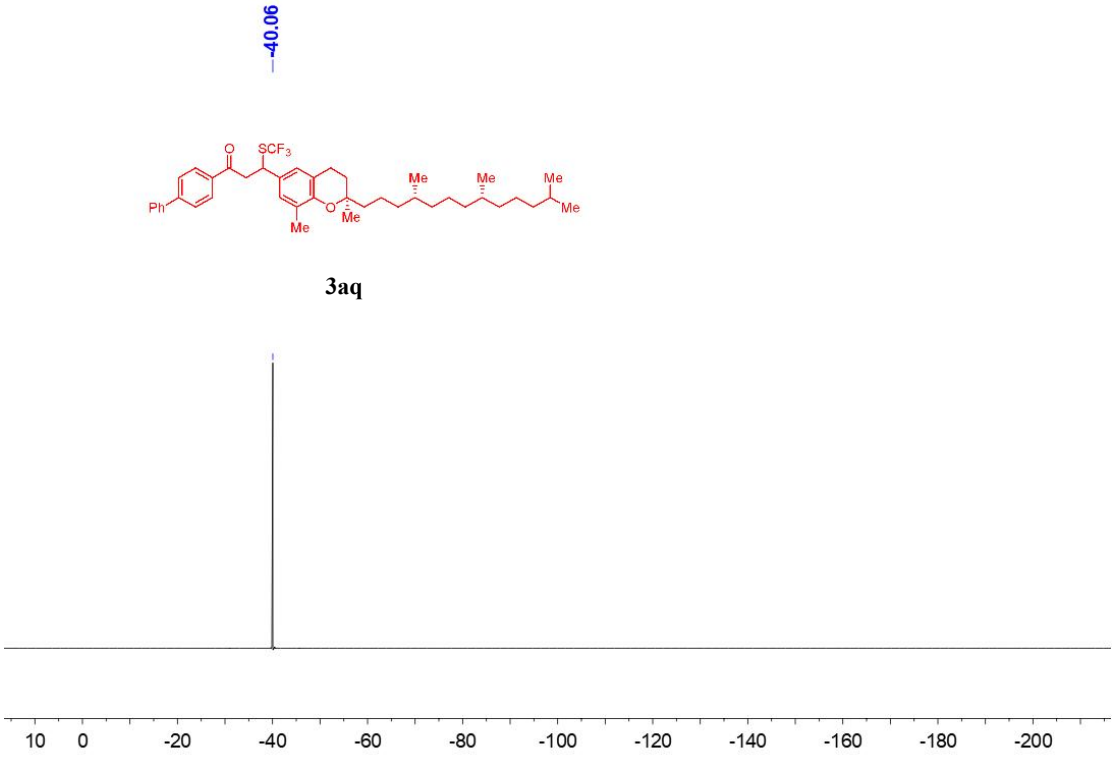
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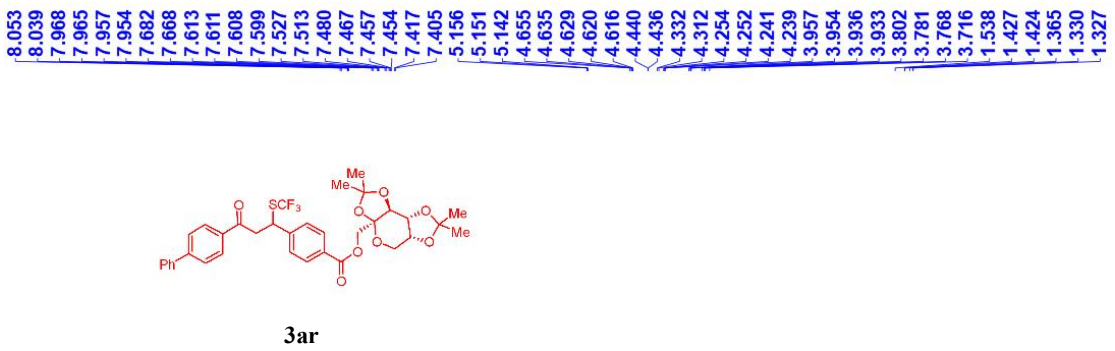
3aq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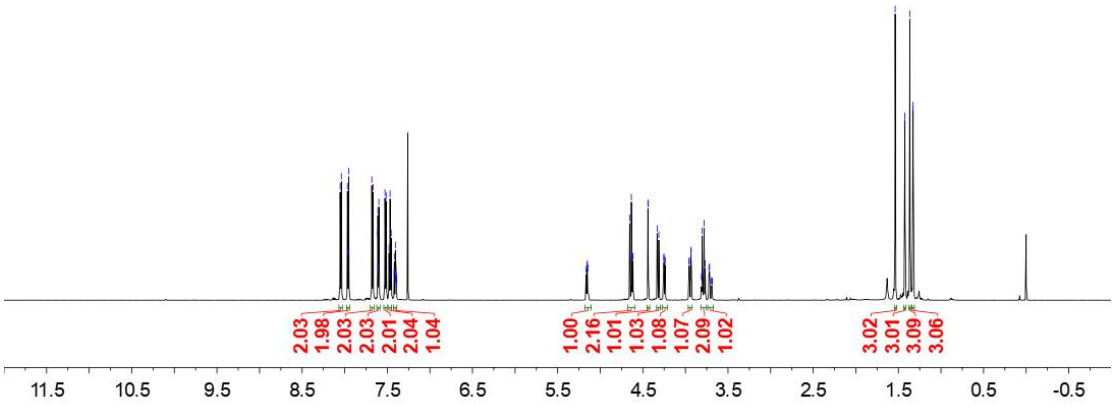
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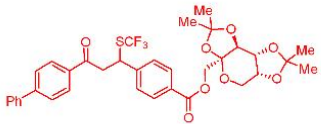
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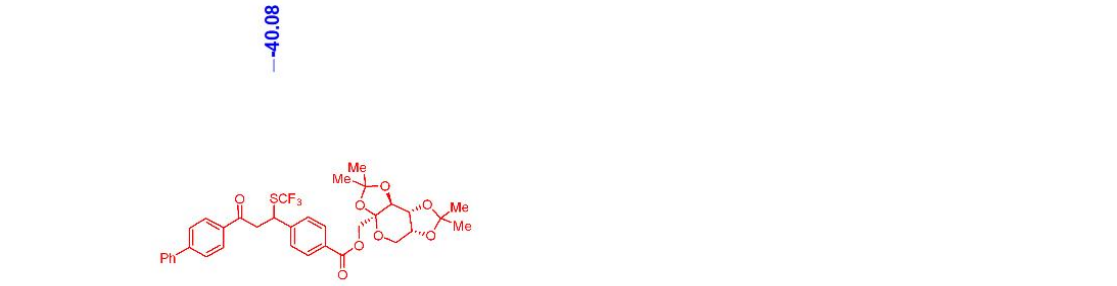
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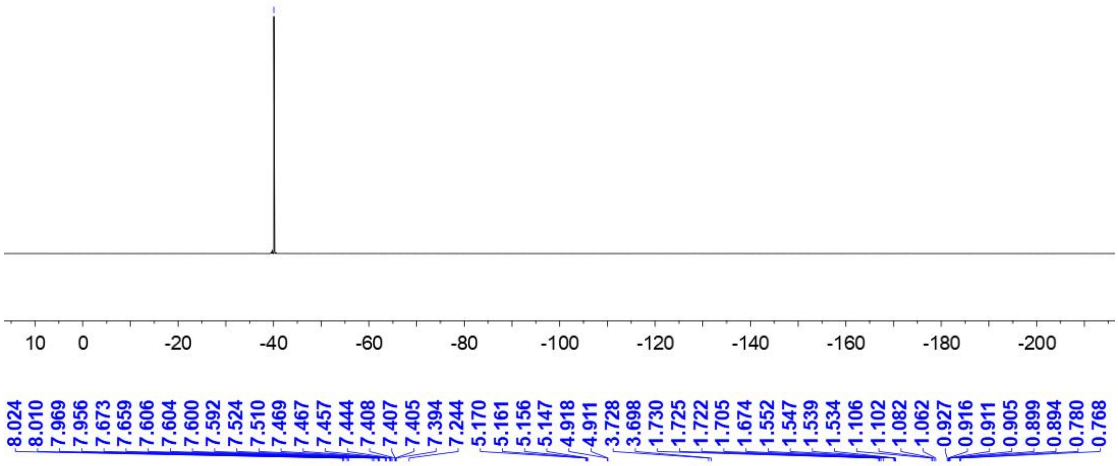
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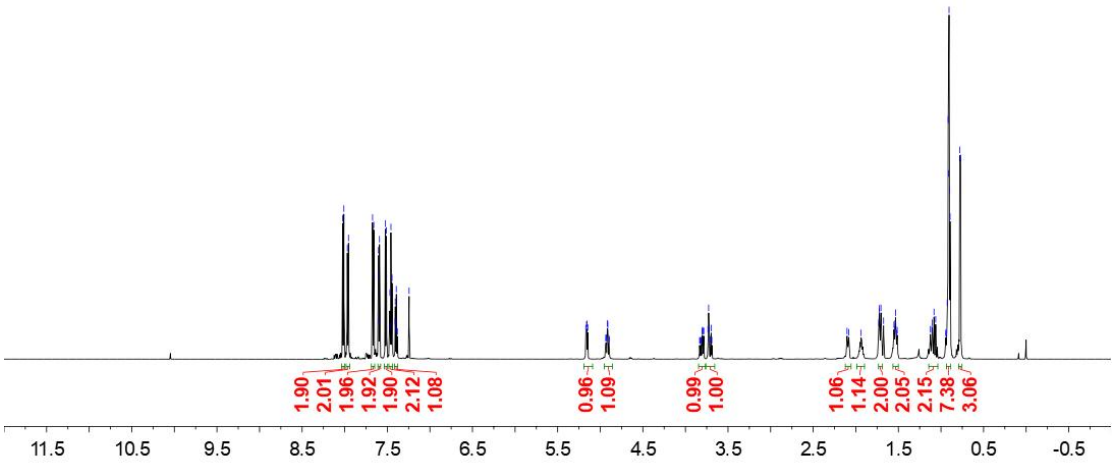
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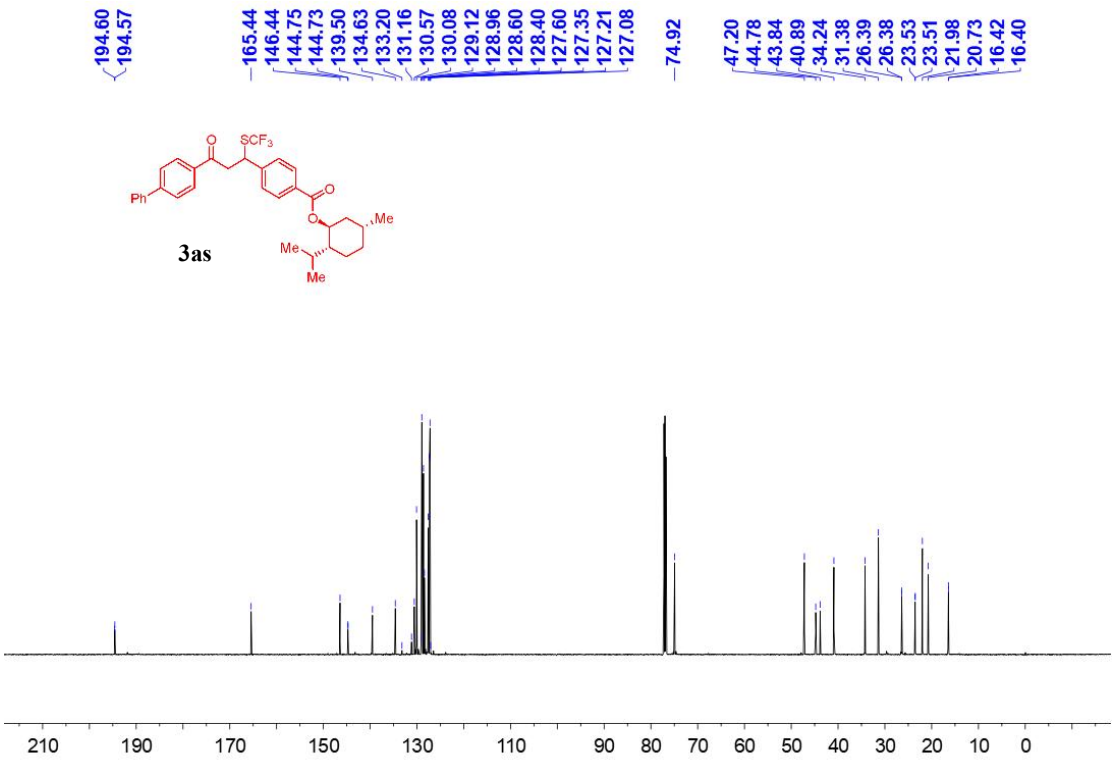
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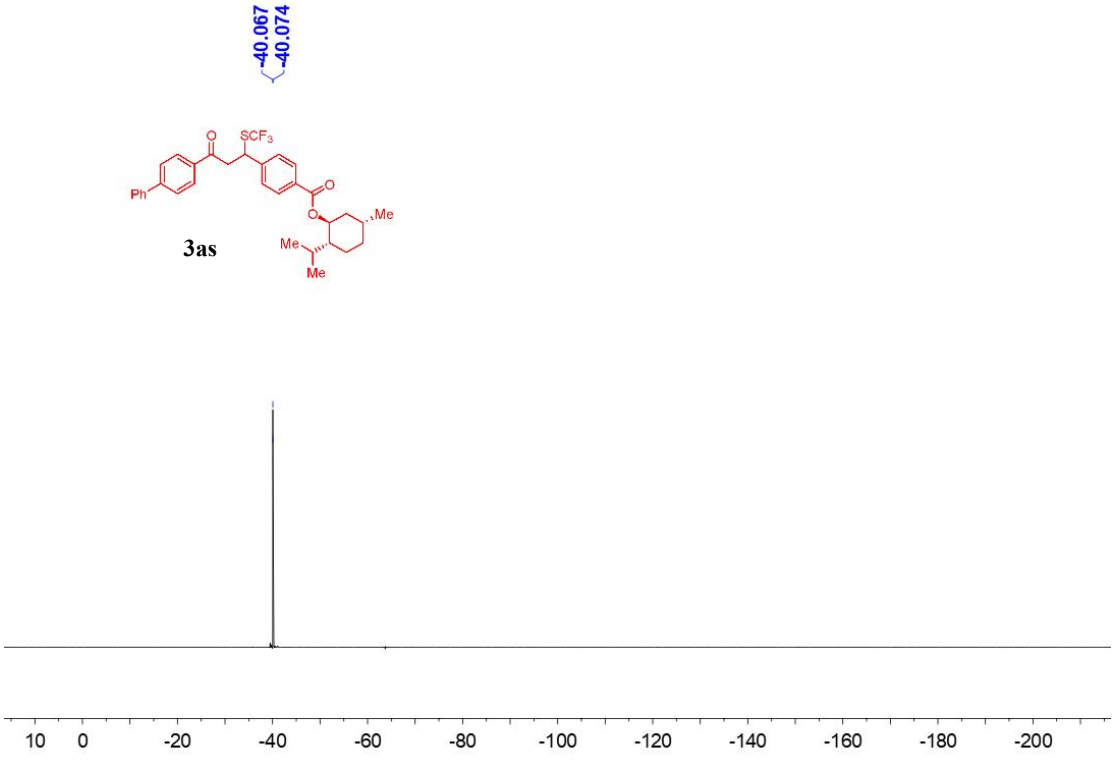
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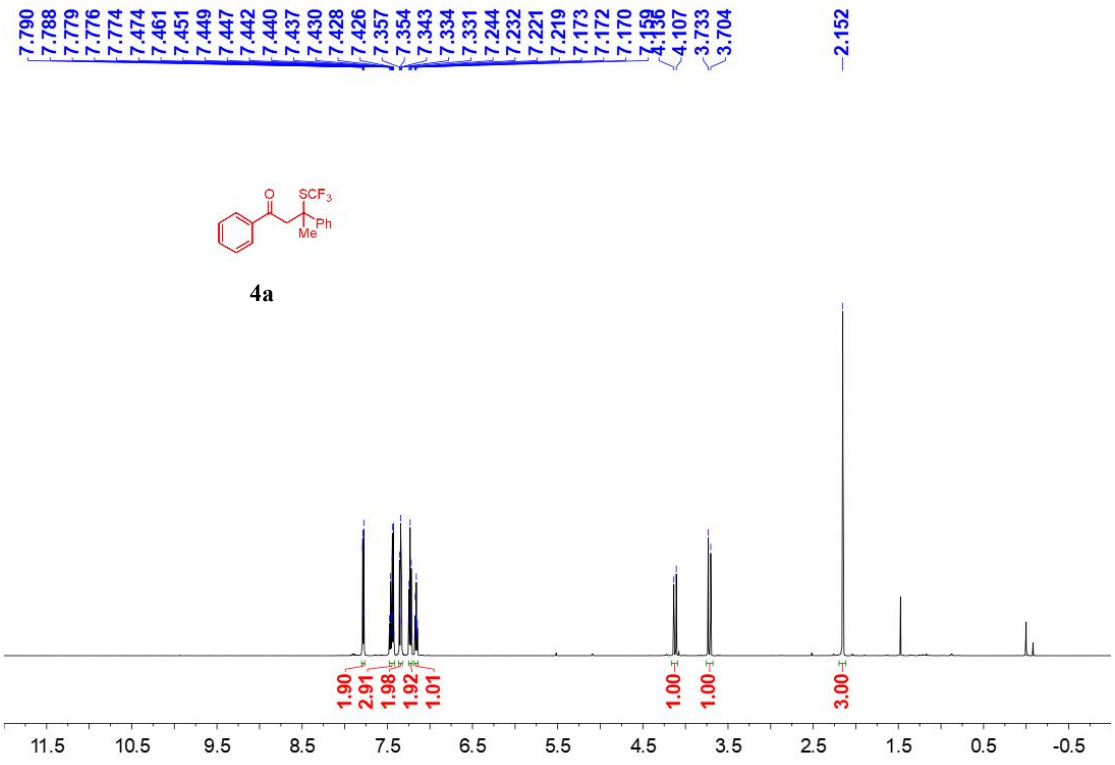
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7.170
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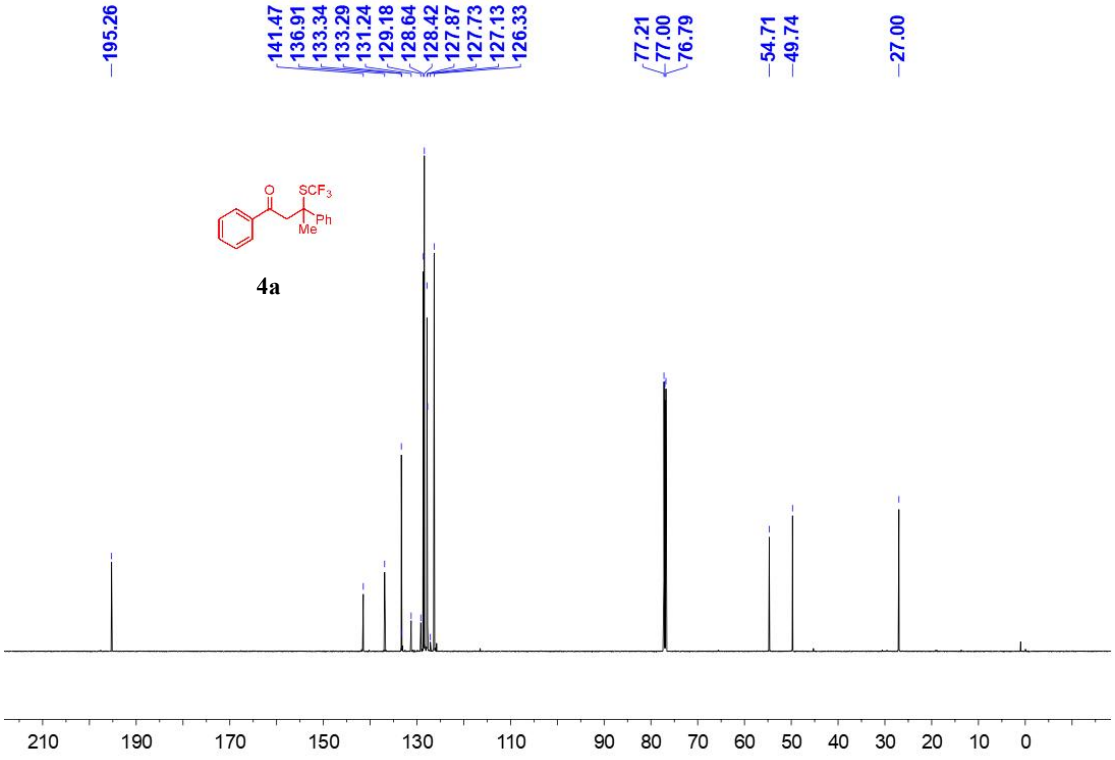
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27.00
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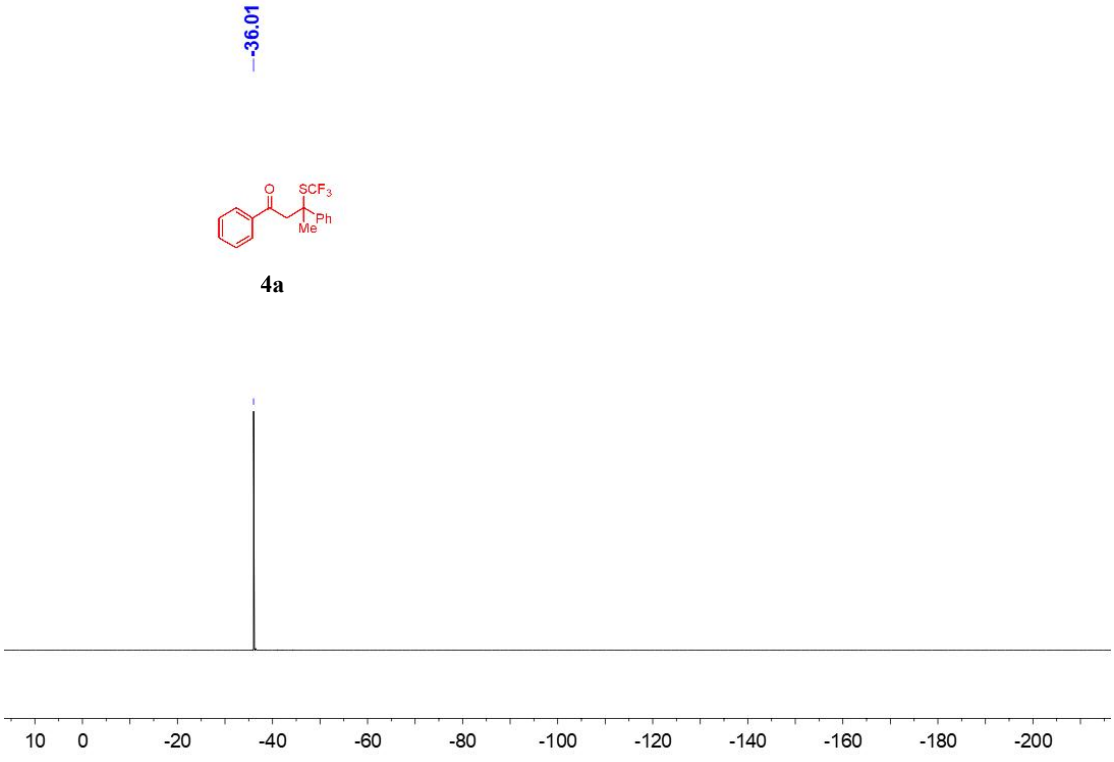
4a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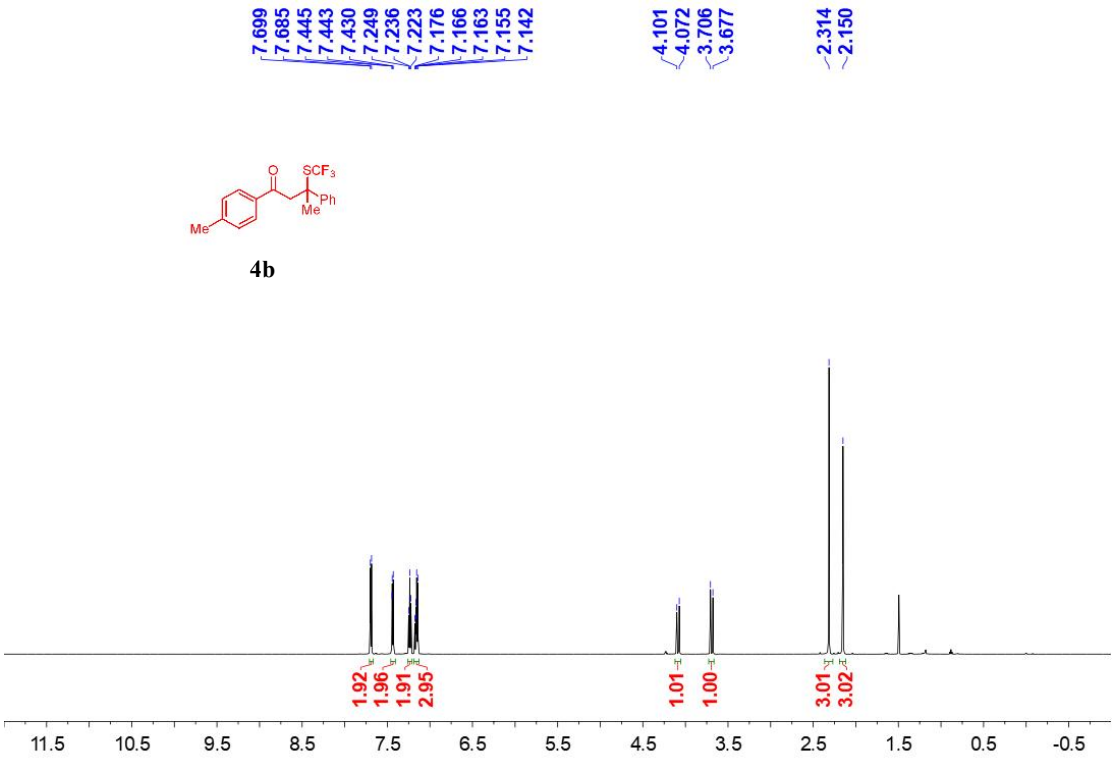
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Ph
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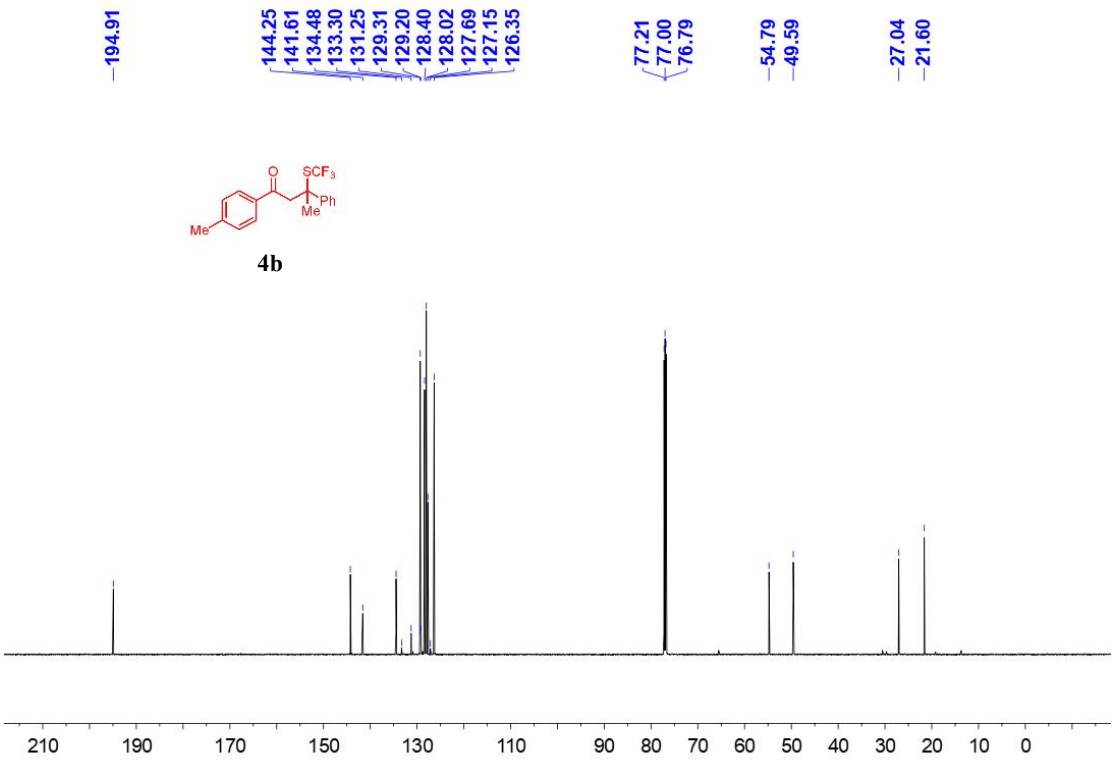
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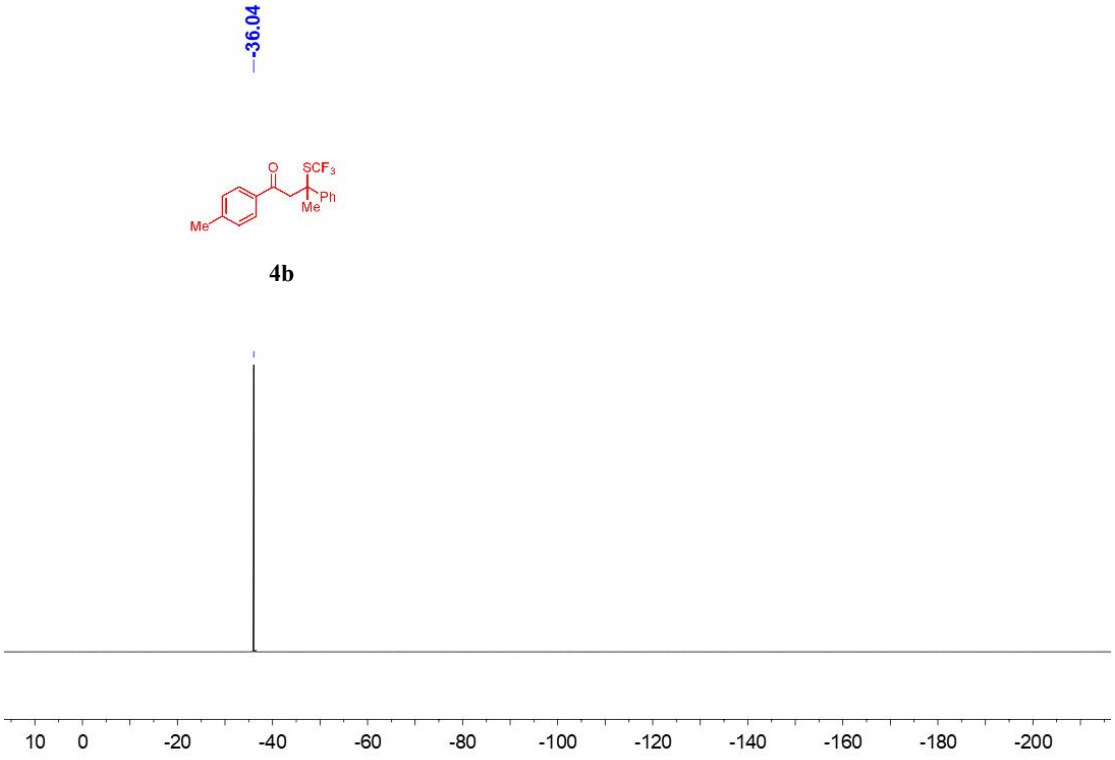
4b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4b


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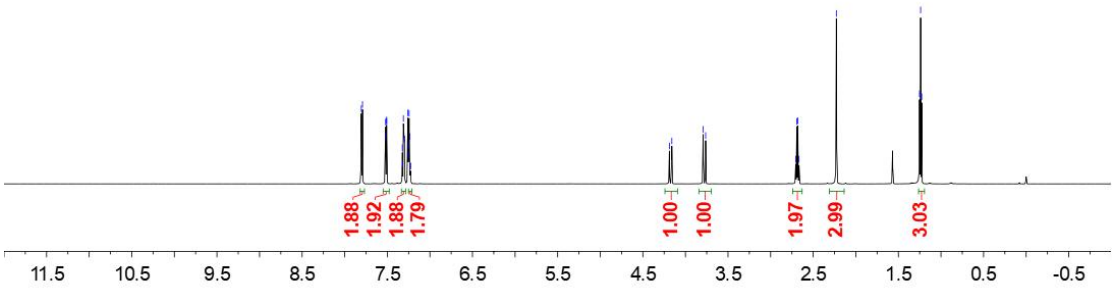
Et

4c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0.5
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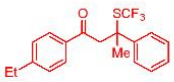
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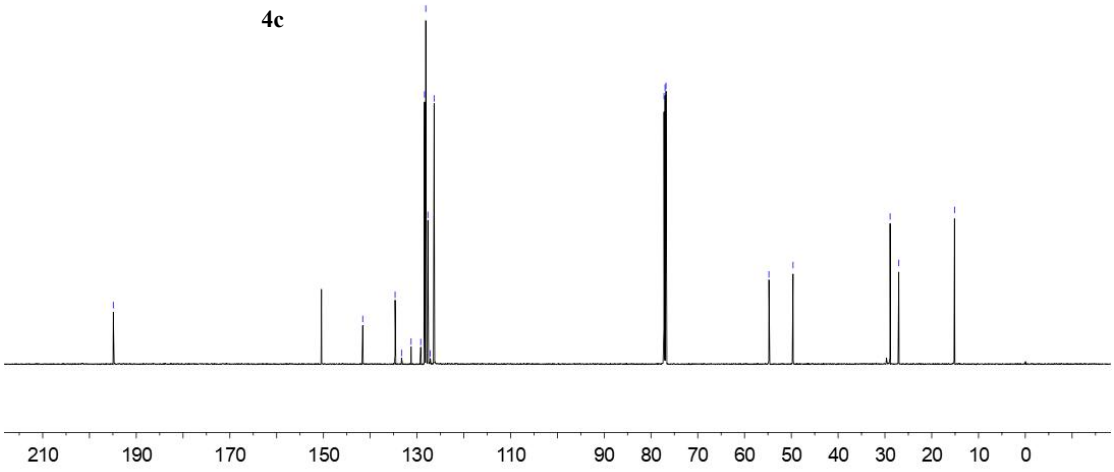
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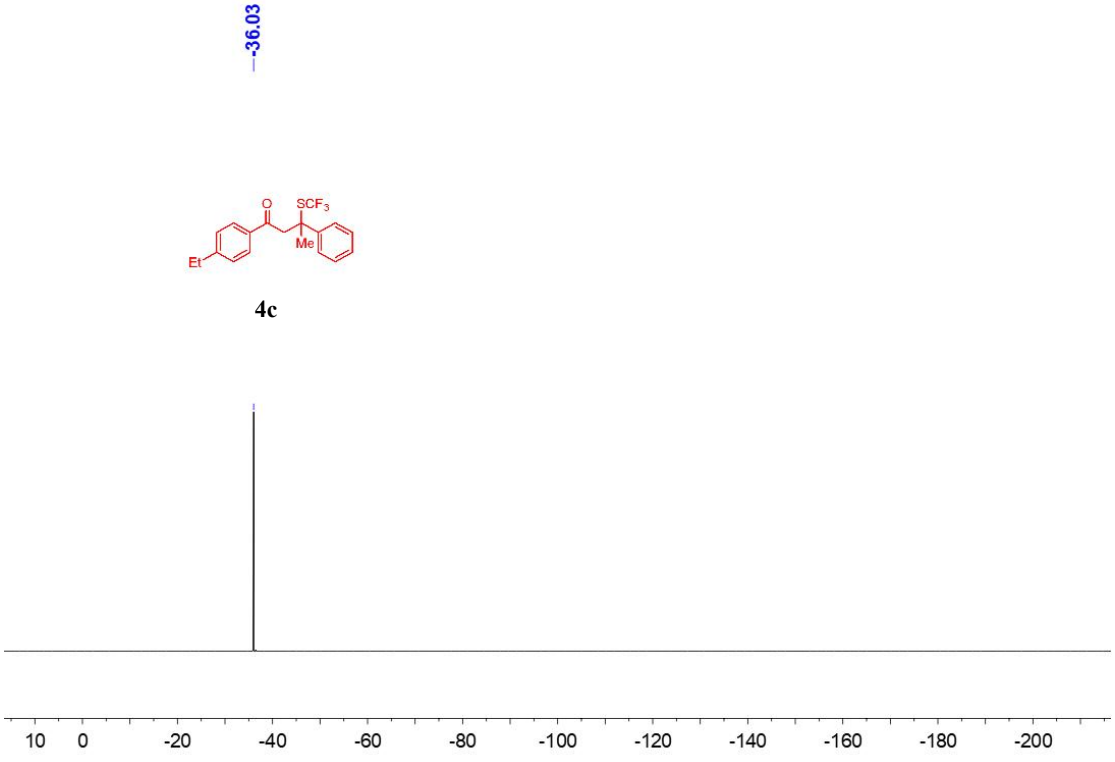
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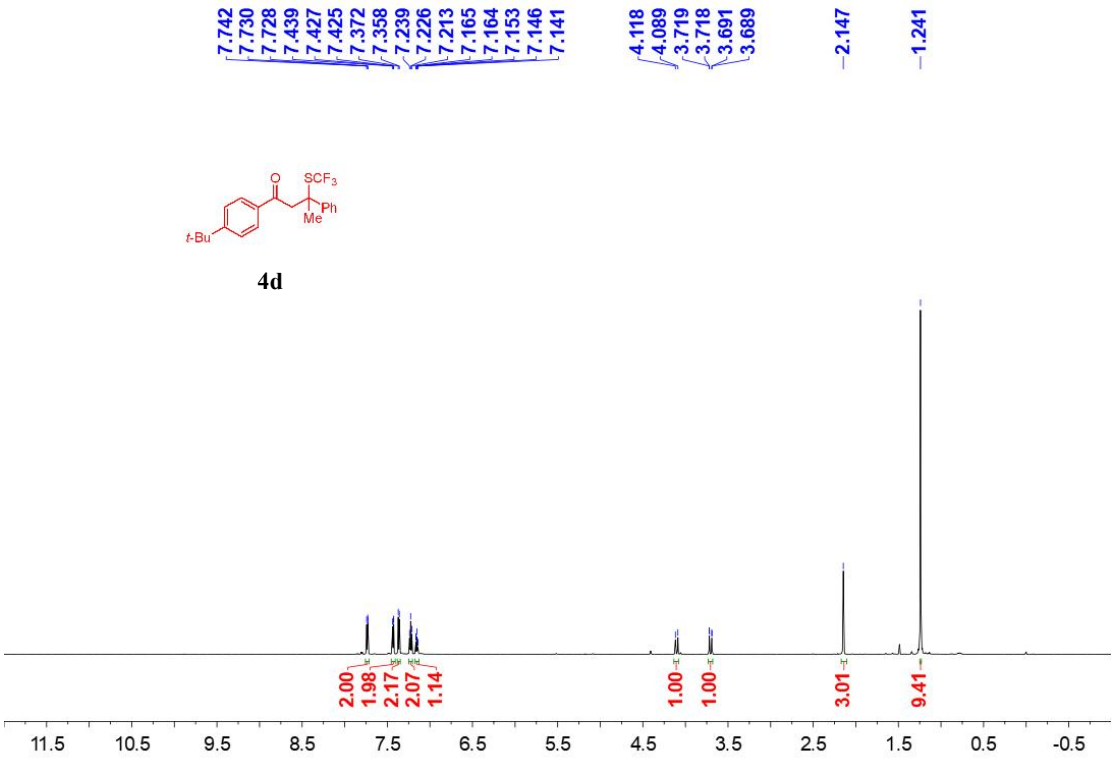
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t-Bu
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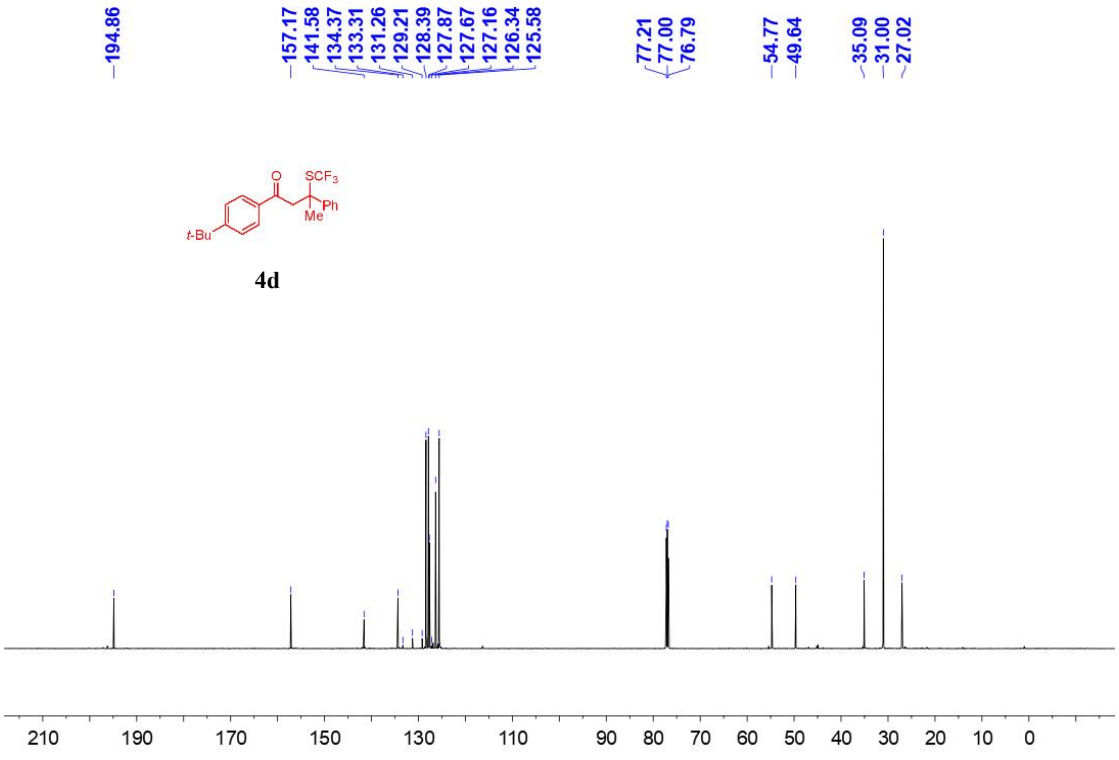
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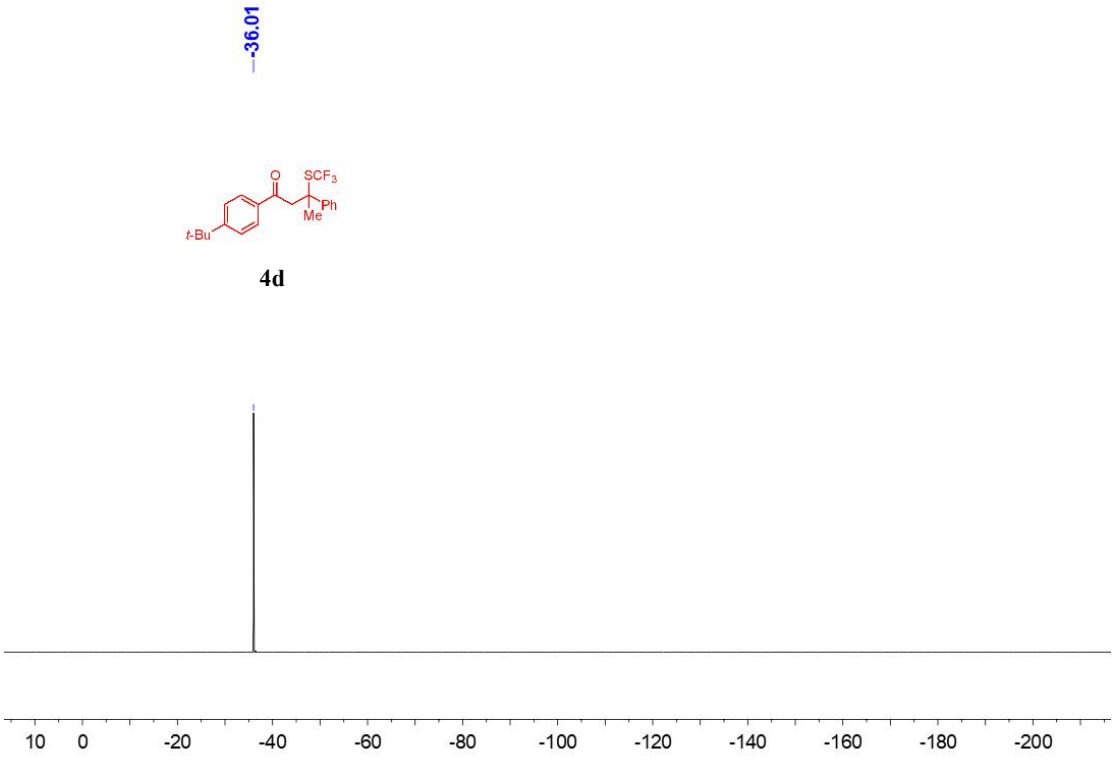
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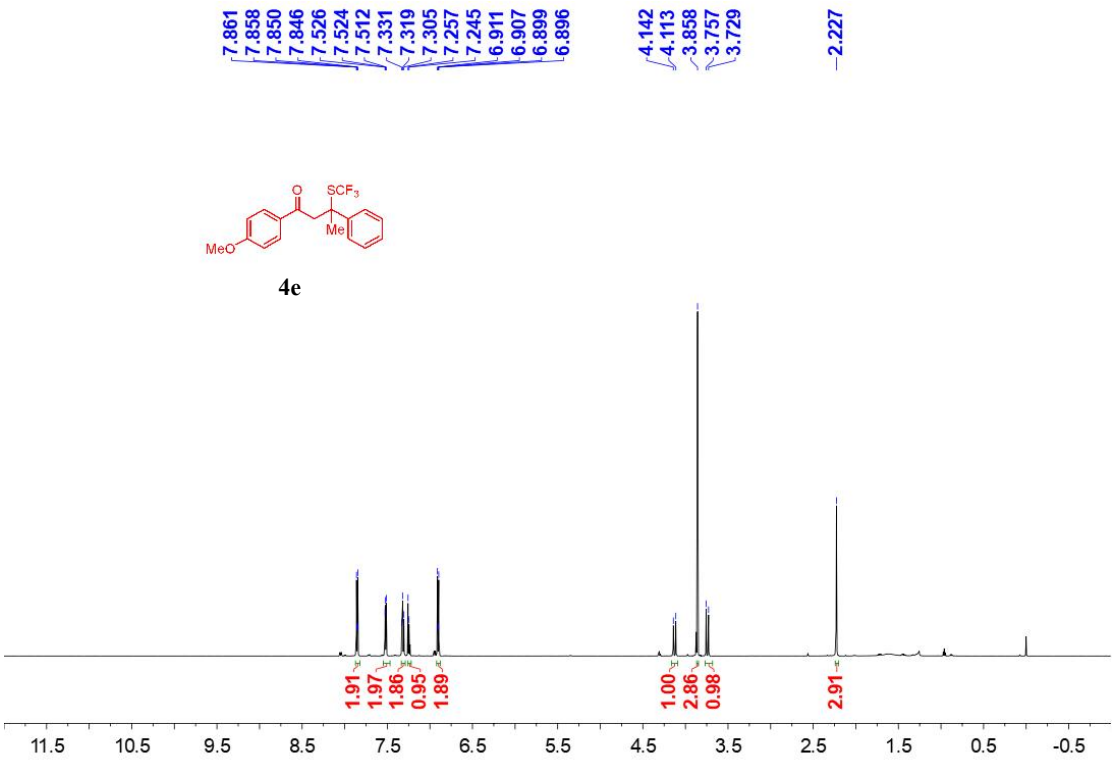
4d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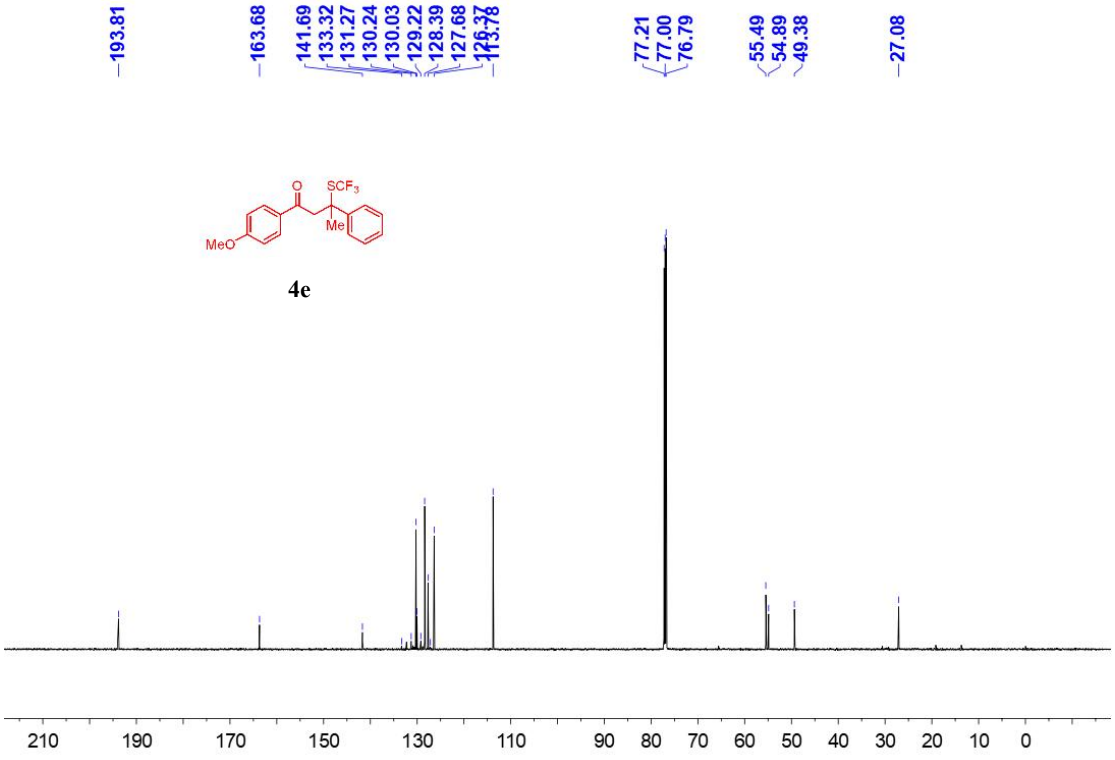
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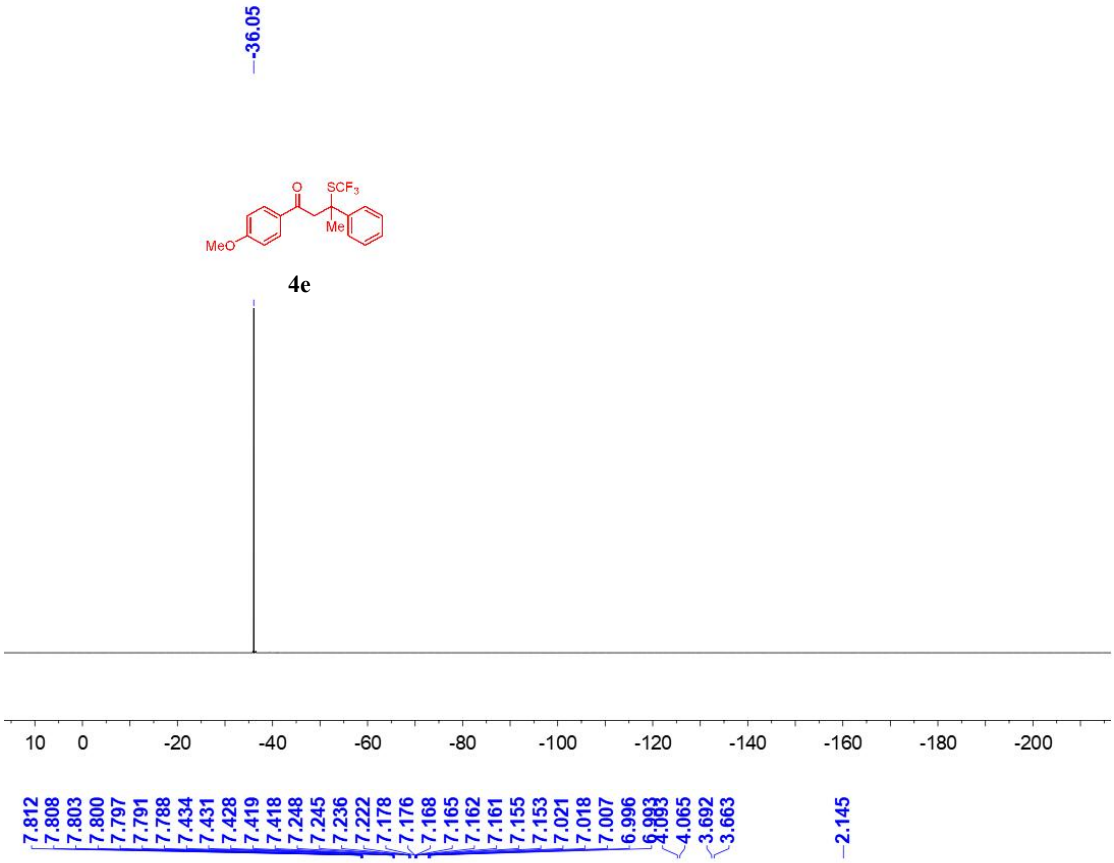
4e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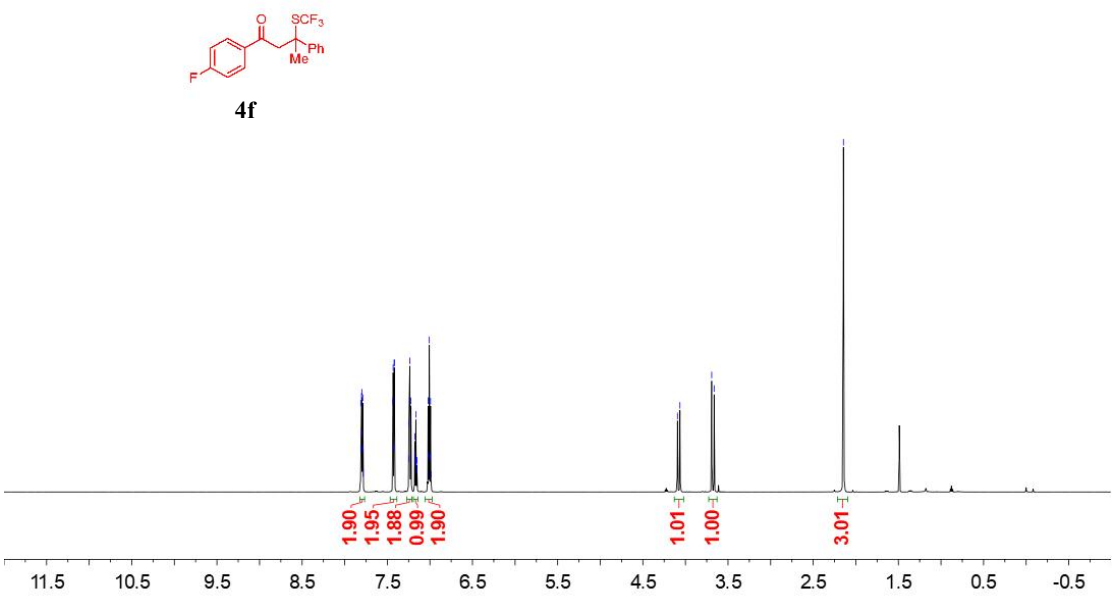
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F
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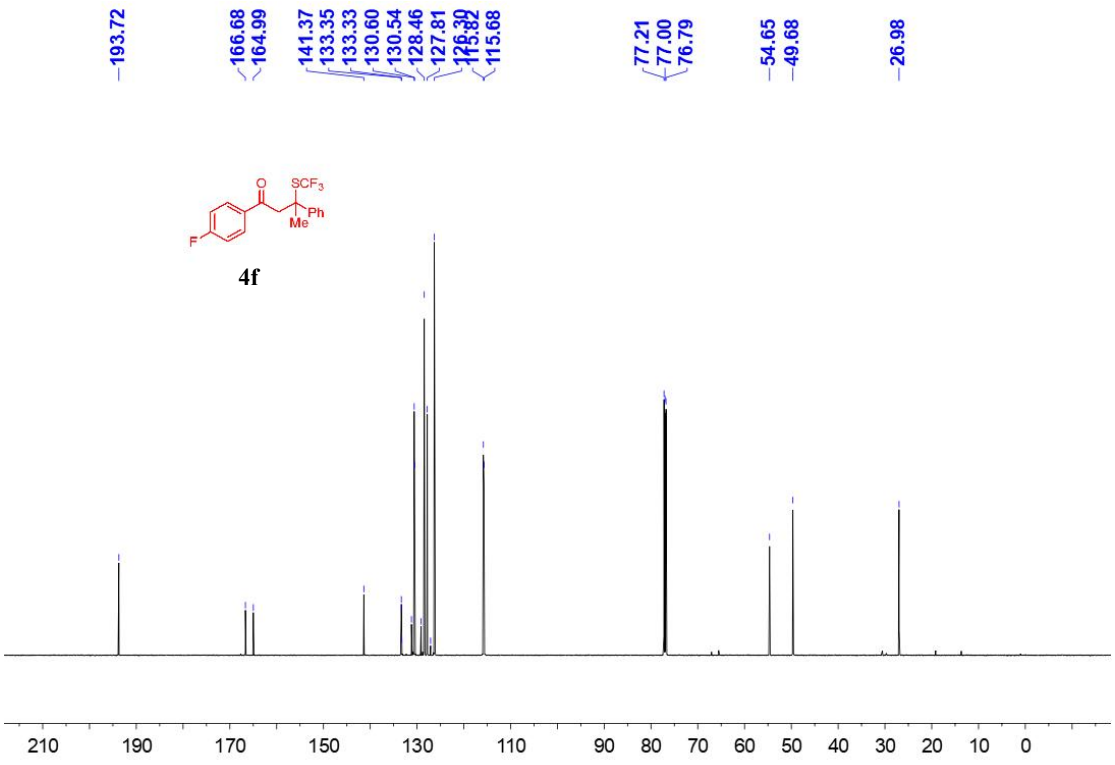
4f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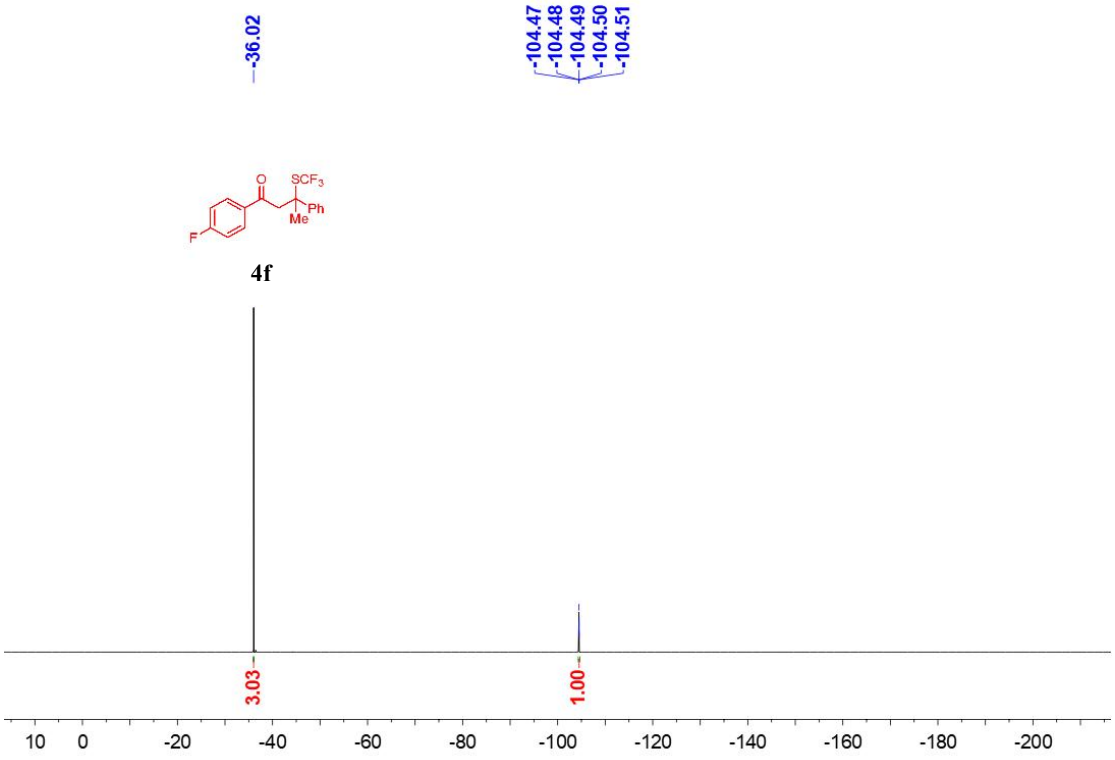
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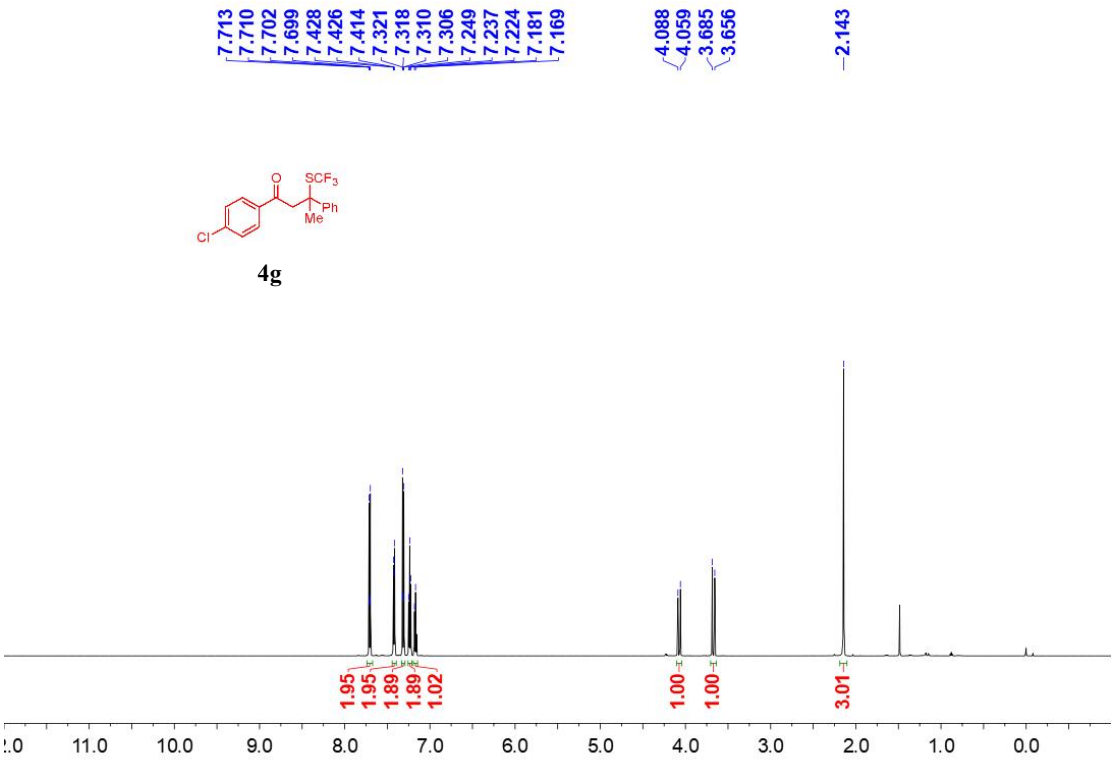
4f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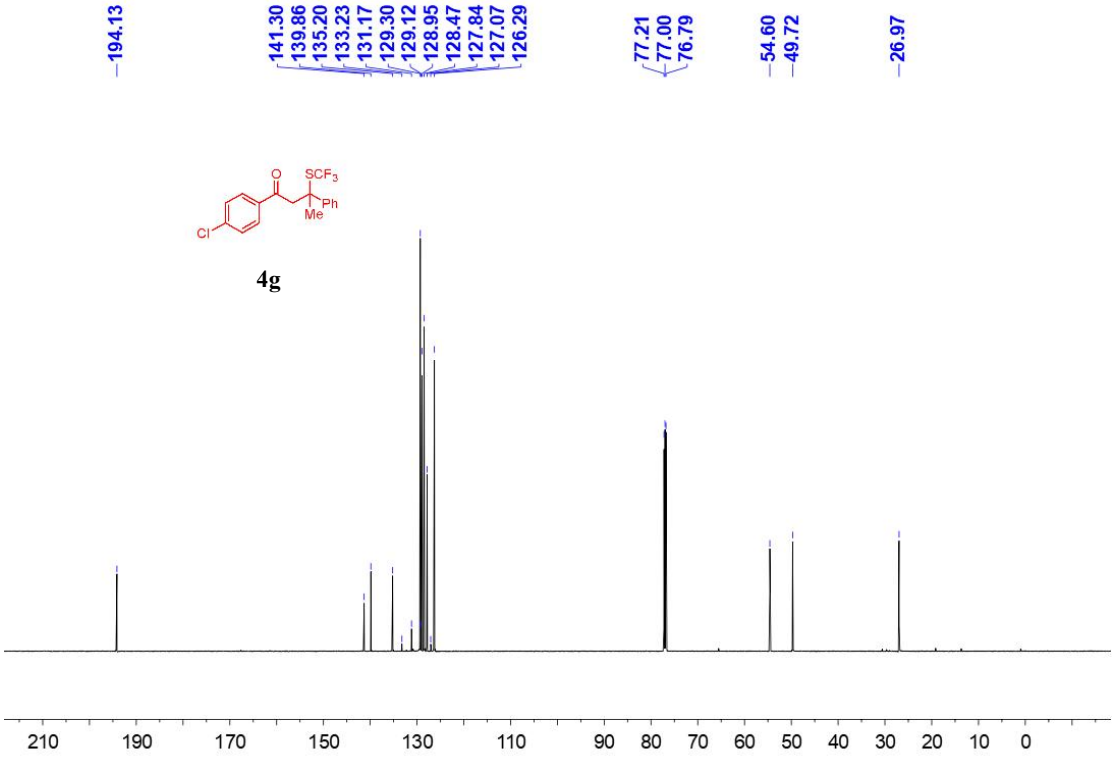
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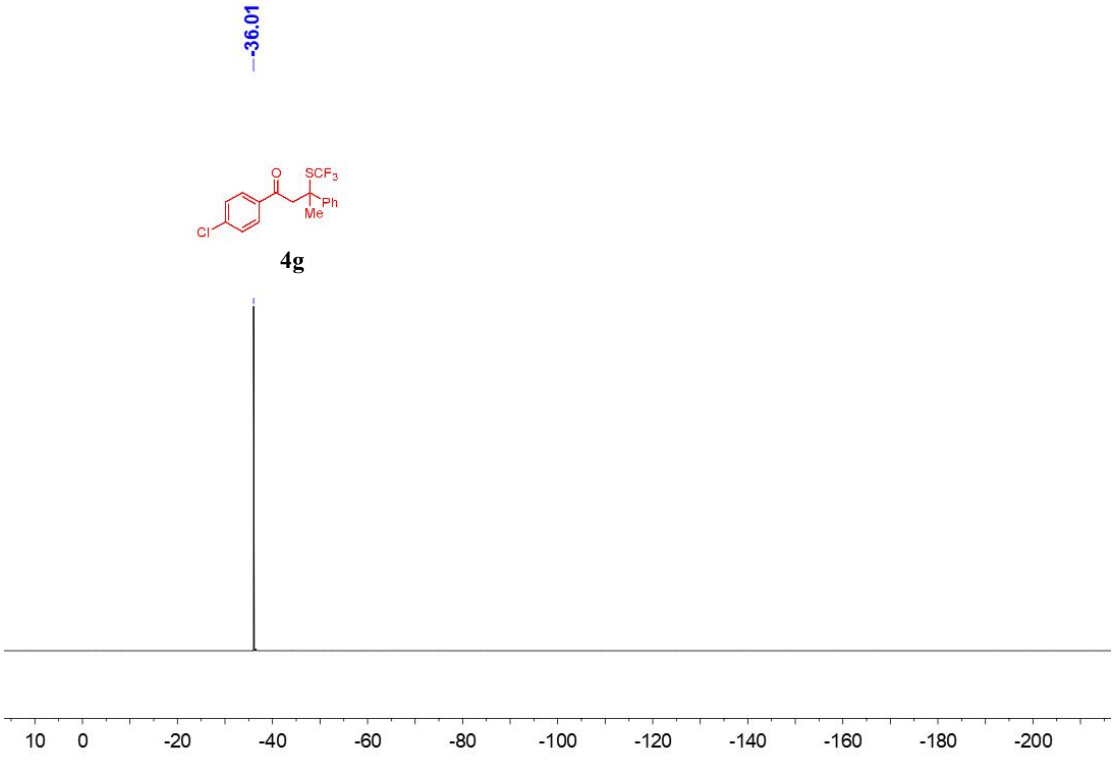
4g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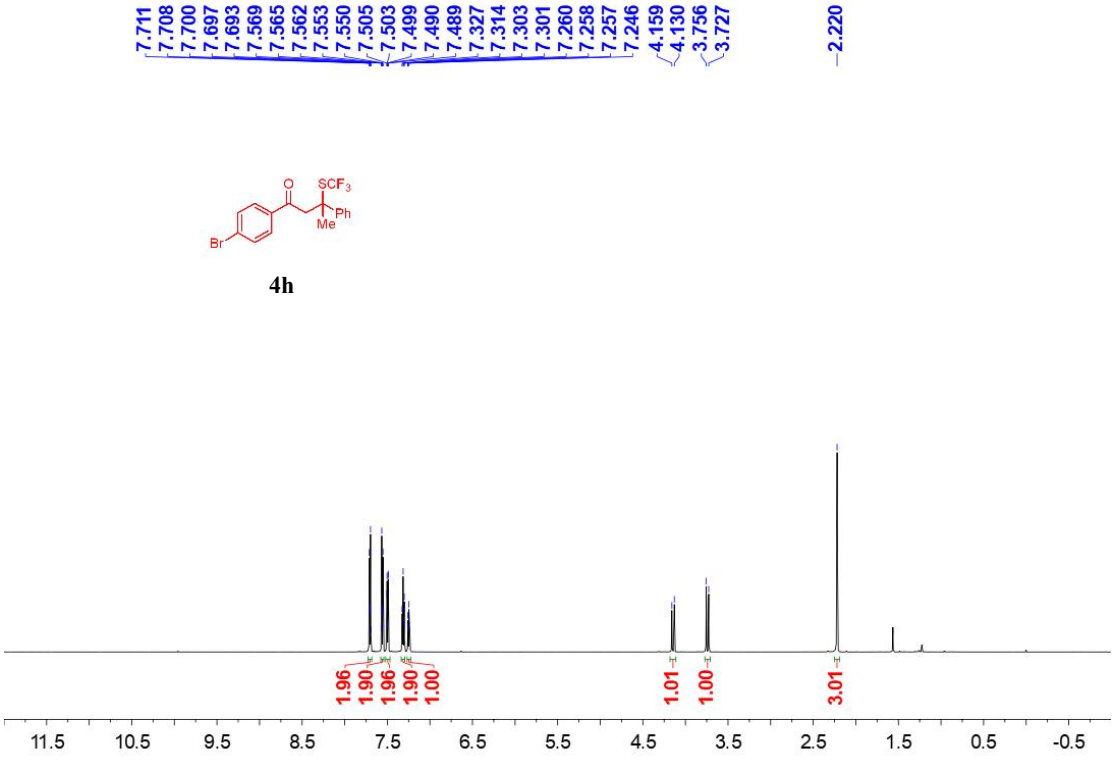
4g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2.220
1.96 1.90 1.96 1.90 1.00 1.01 1.00 3.01
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

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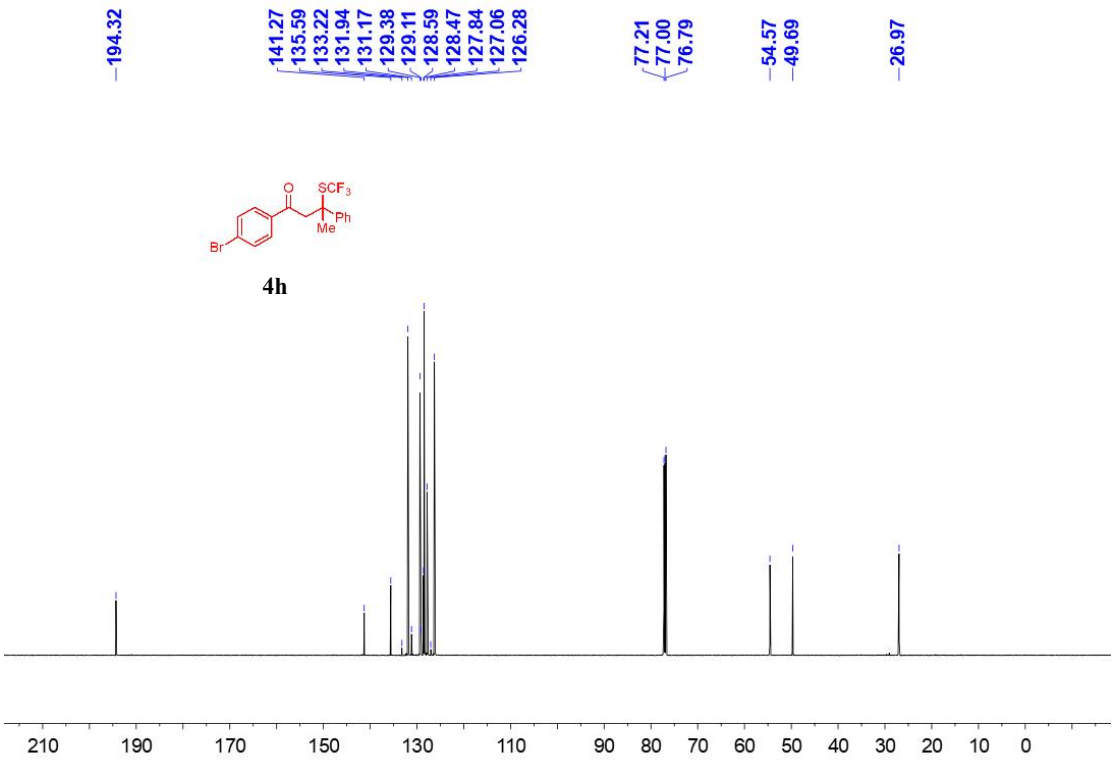
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77.00
76.79
54.57
49.69
26.97
O
SCF3
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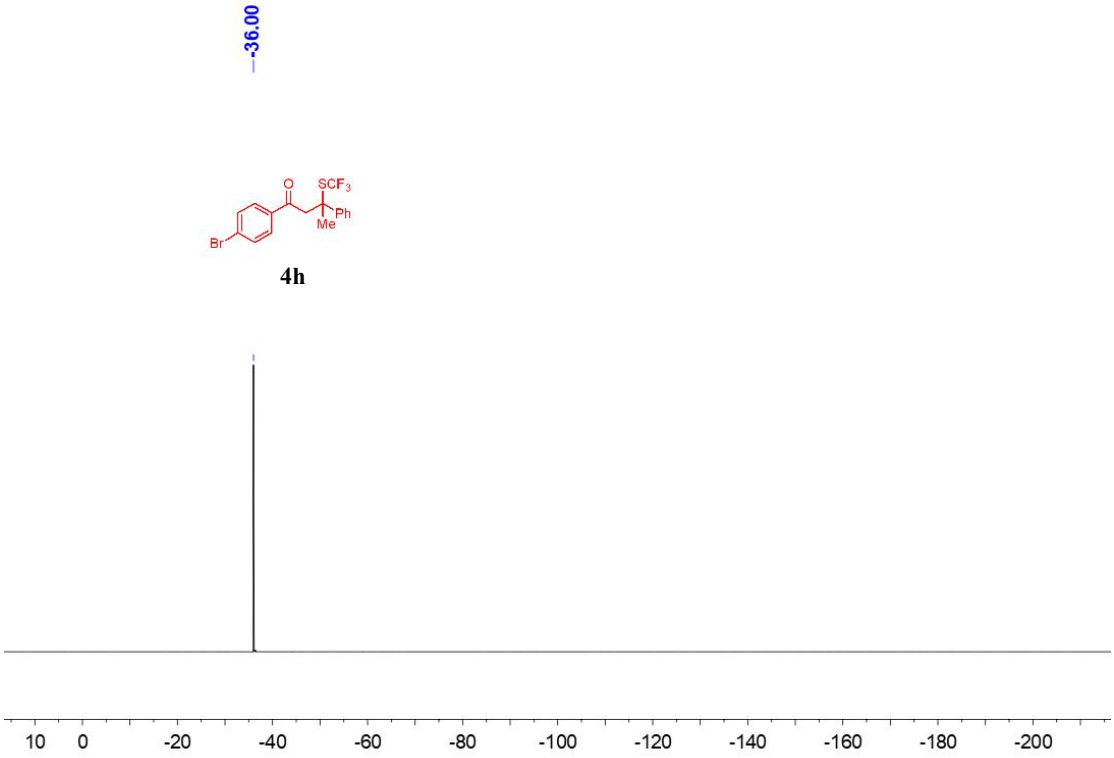
4h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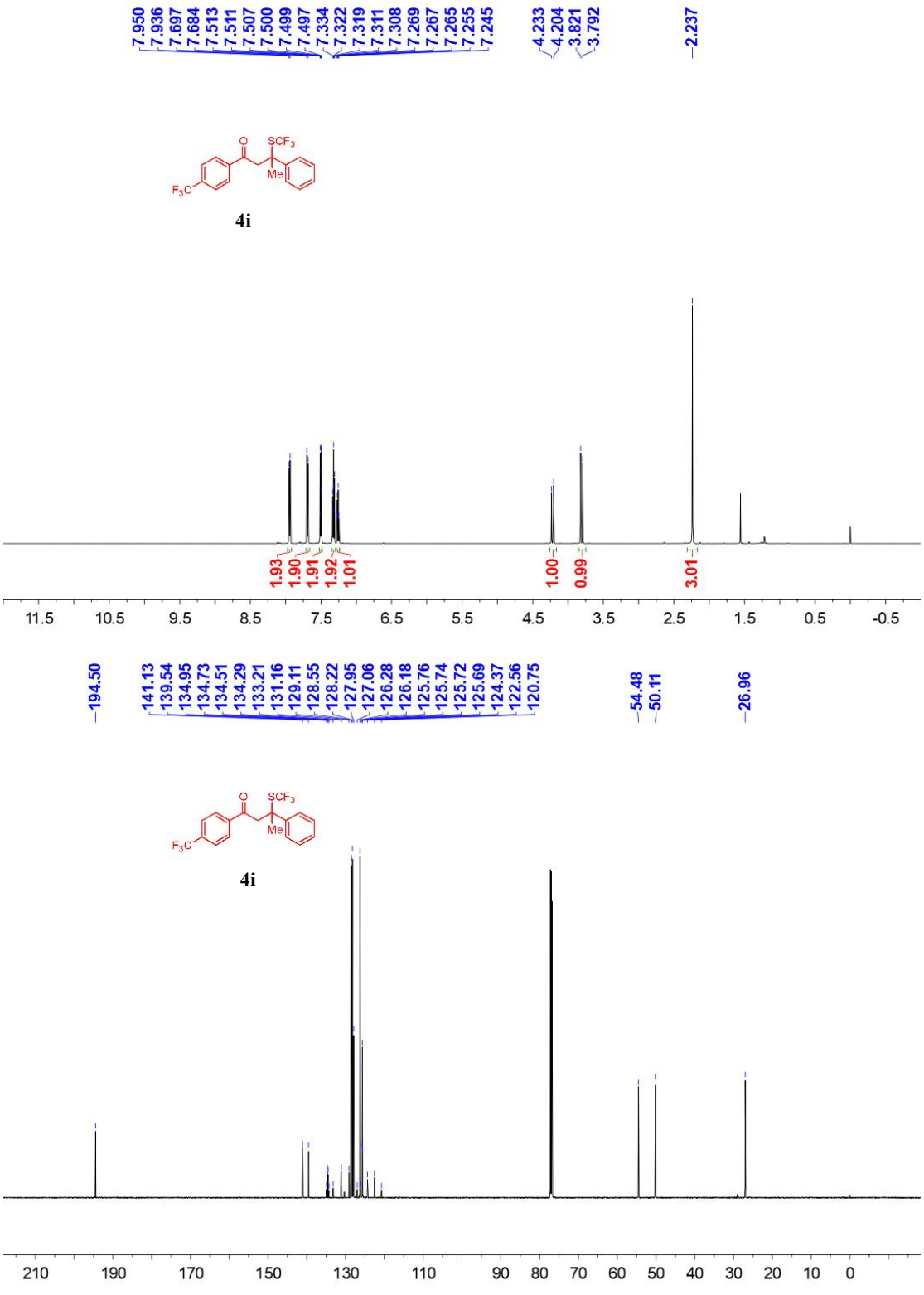
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7.308
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2.237
SCF3
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F3C
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1.91
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1.00
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3.01
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4i


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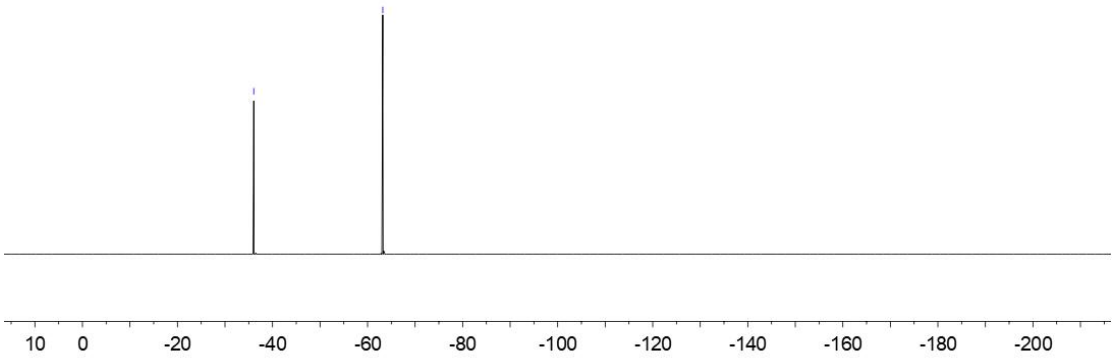
4i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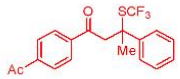
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Me
Ac

4j

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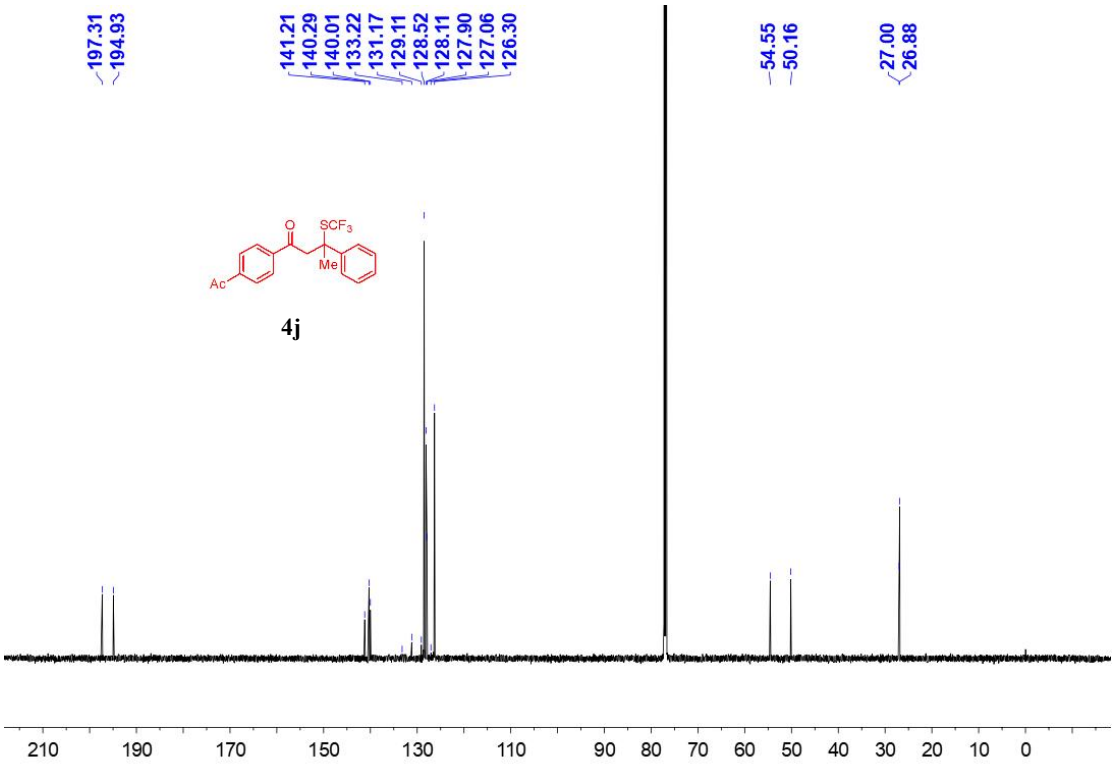
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27.00
26.88
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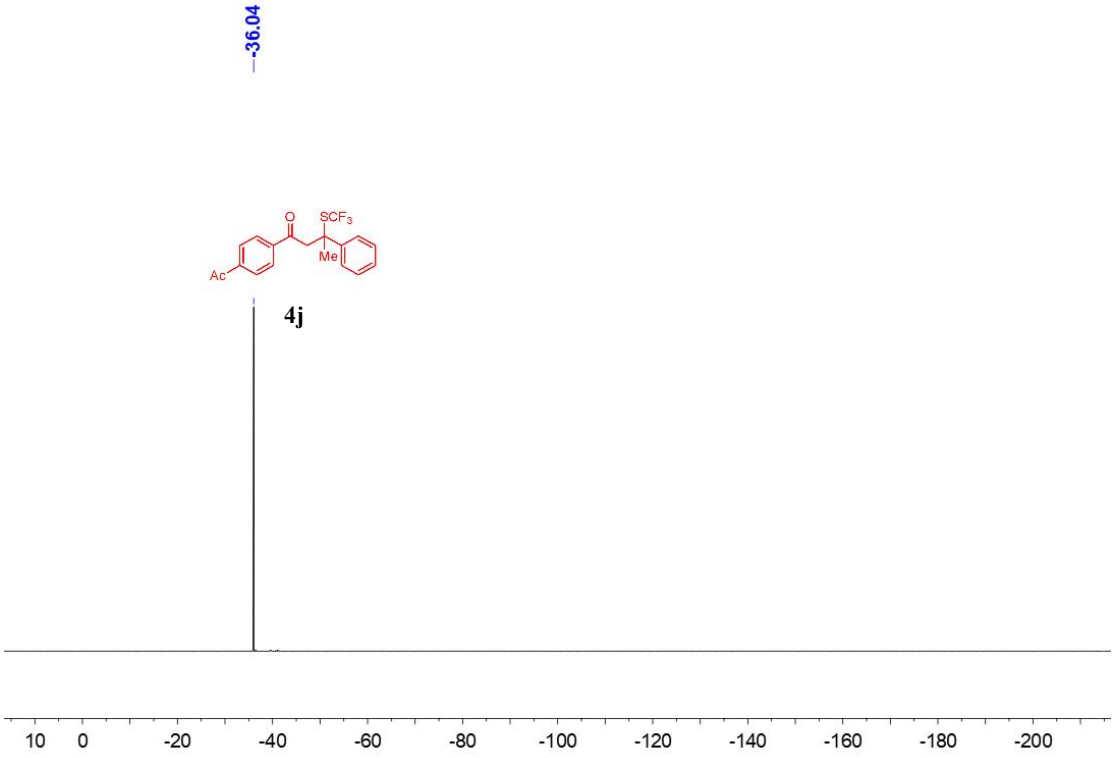
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4j


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7.233
7.228
7.225
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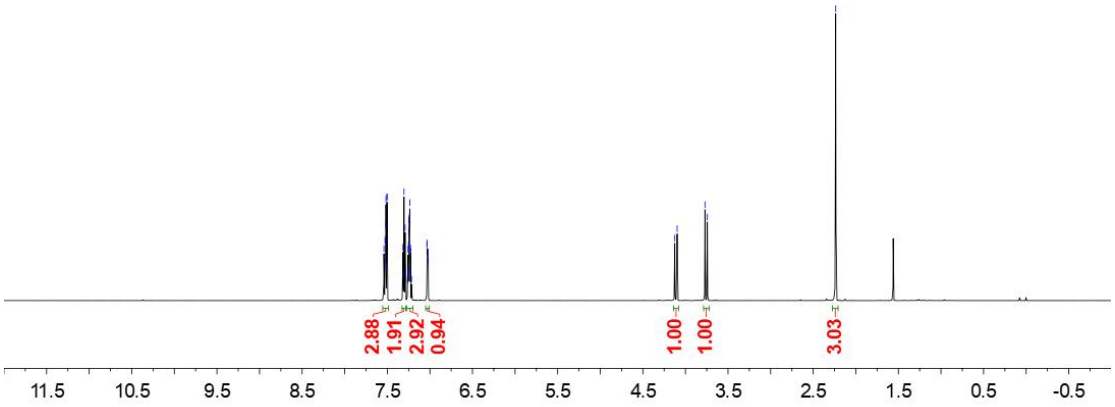
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5.5
4.5
3.5
2.5
1.5
0.5
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199.58
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133.13
131.59
131.08
129.03
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127.92
127.38
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53.89
26.86


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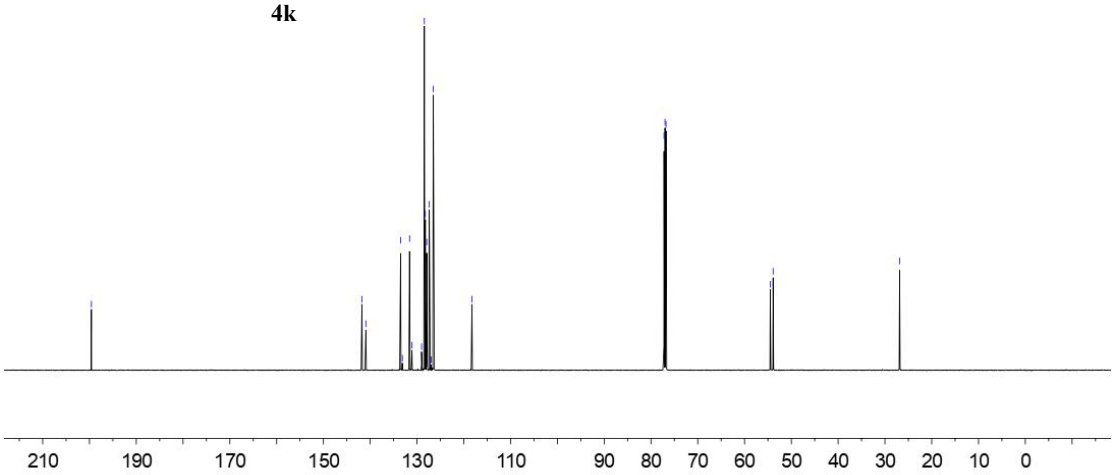
SCF3
Ph
Me
Br

4k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Br
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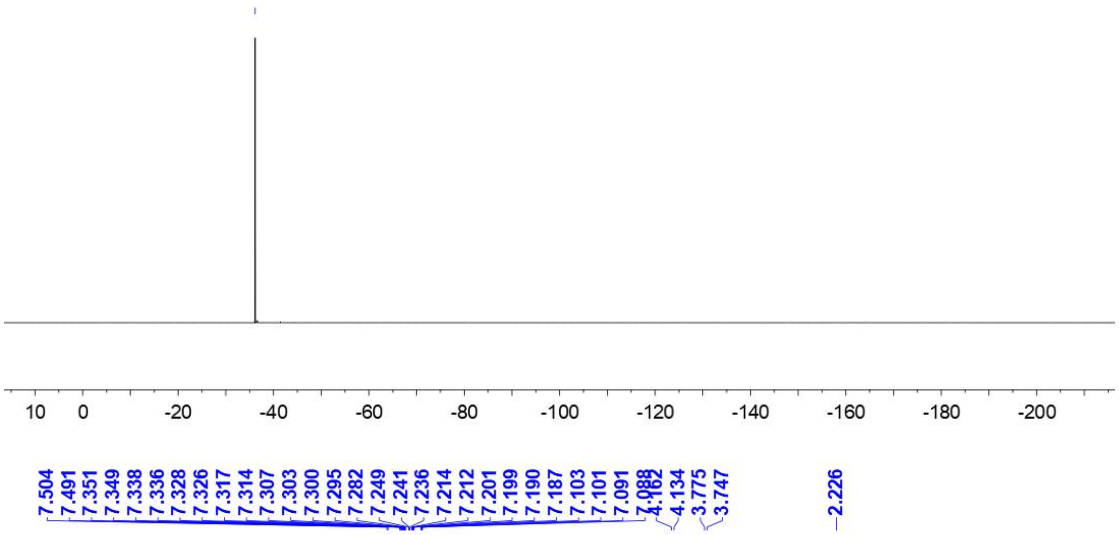
4k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7.249
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7.236
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7.212
7.201
7.199
7.190
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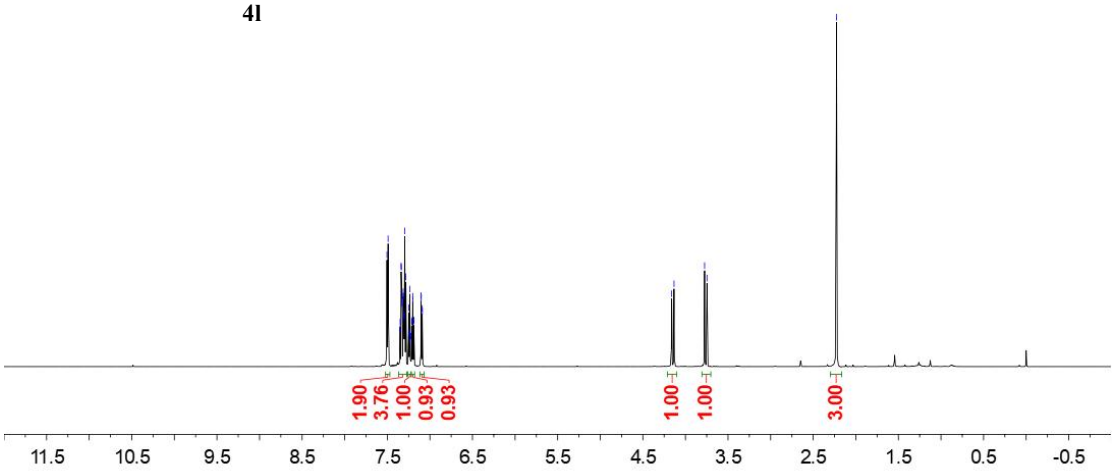
Cl

4l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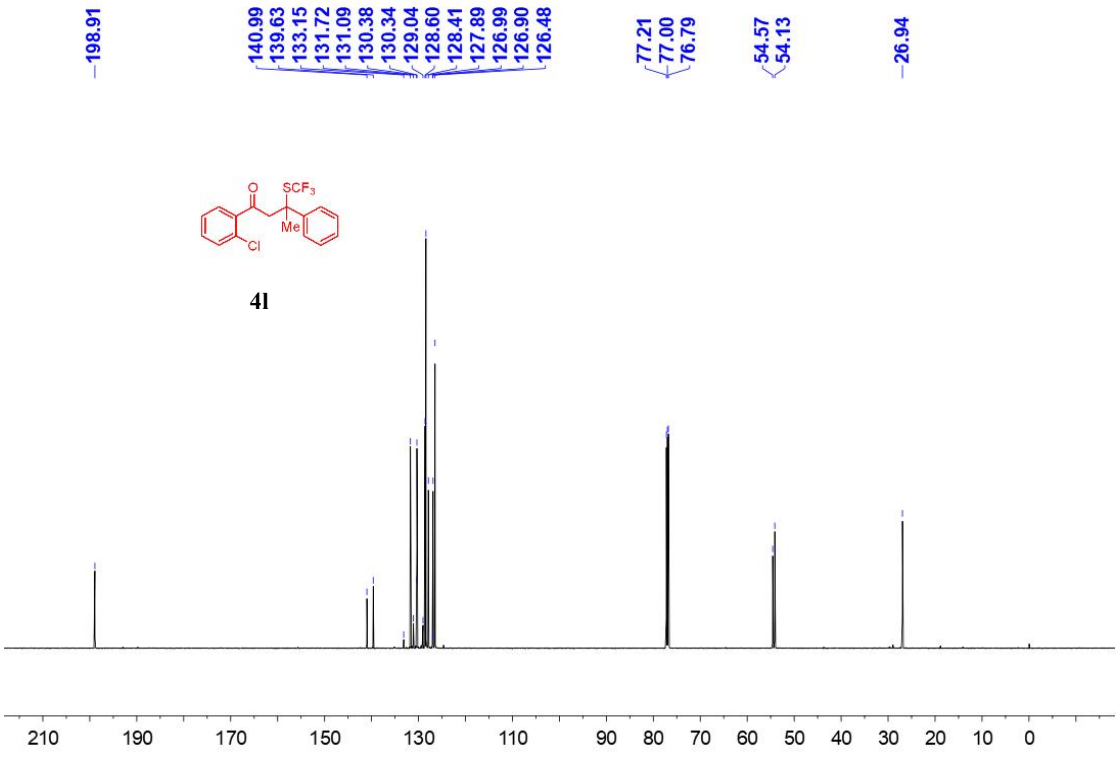
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76.79
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54.13
26.94
O
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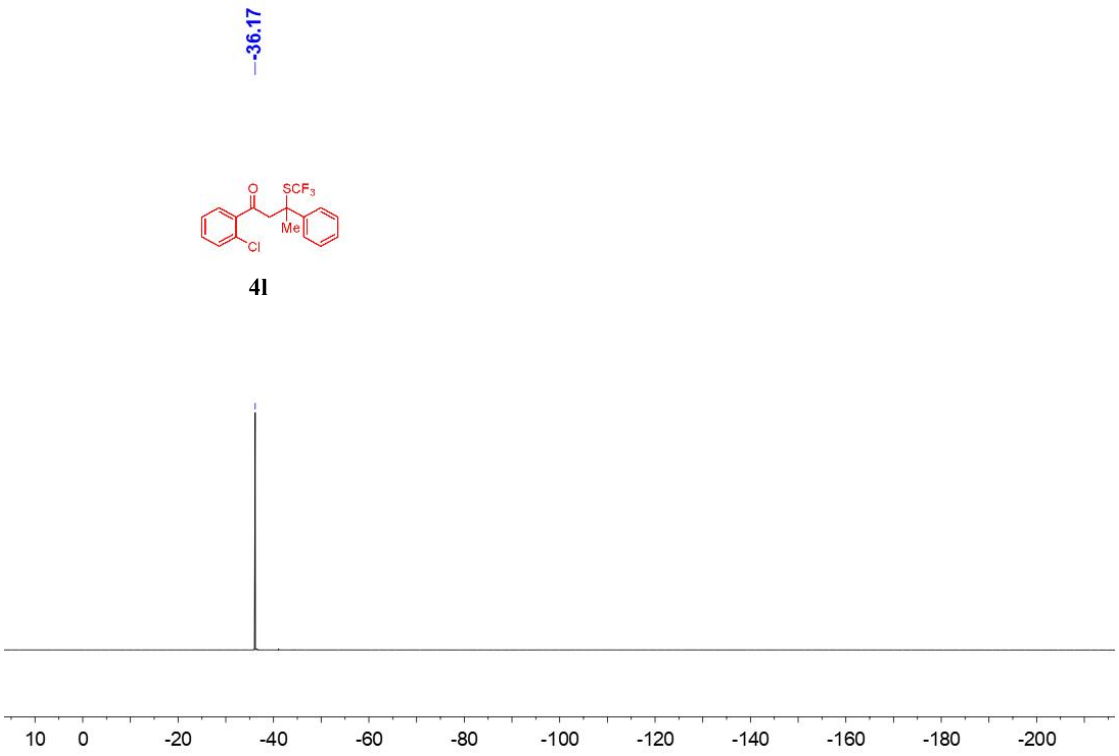
0

4l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-200

4l


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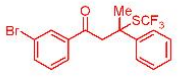
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SCF3

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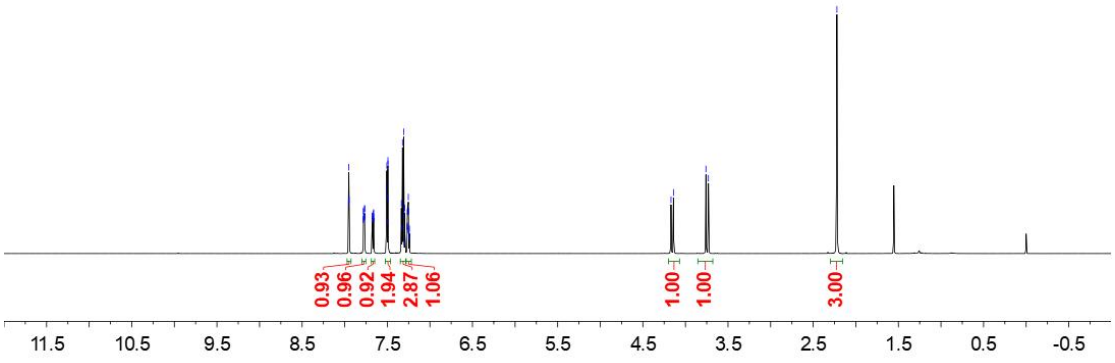
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7.5
6.5
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4.5
3.5
2.5
1.5
0.5
-0.5


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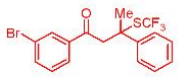
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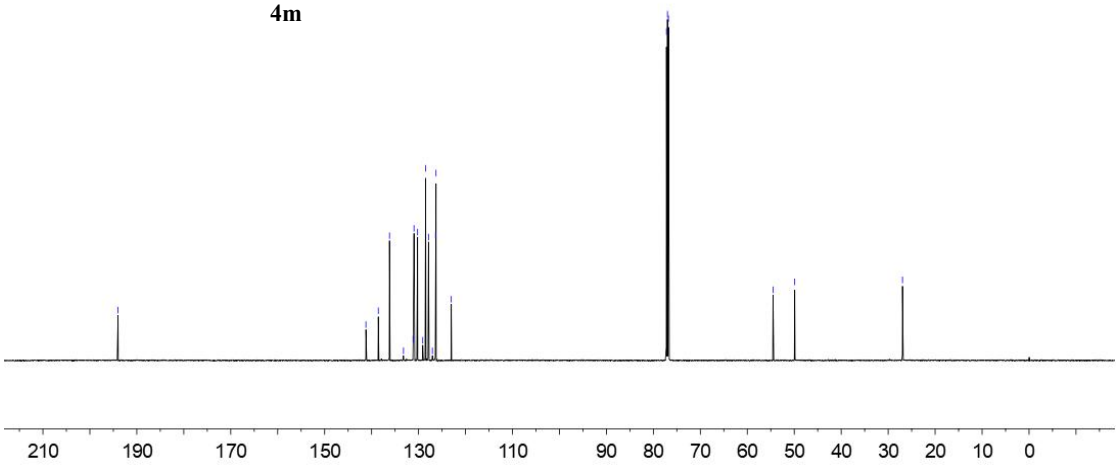
Me
SCF3

4m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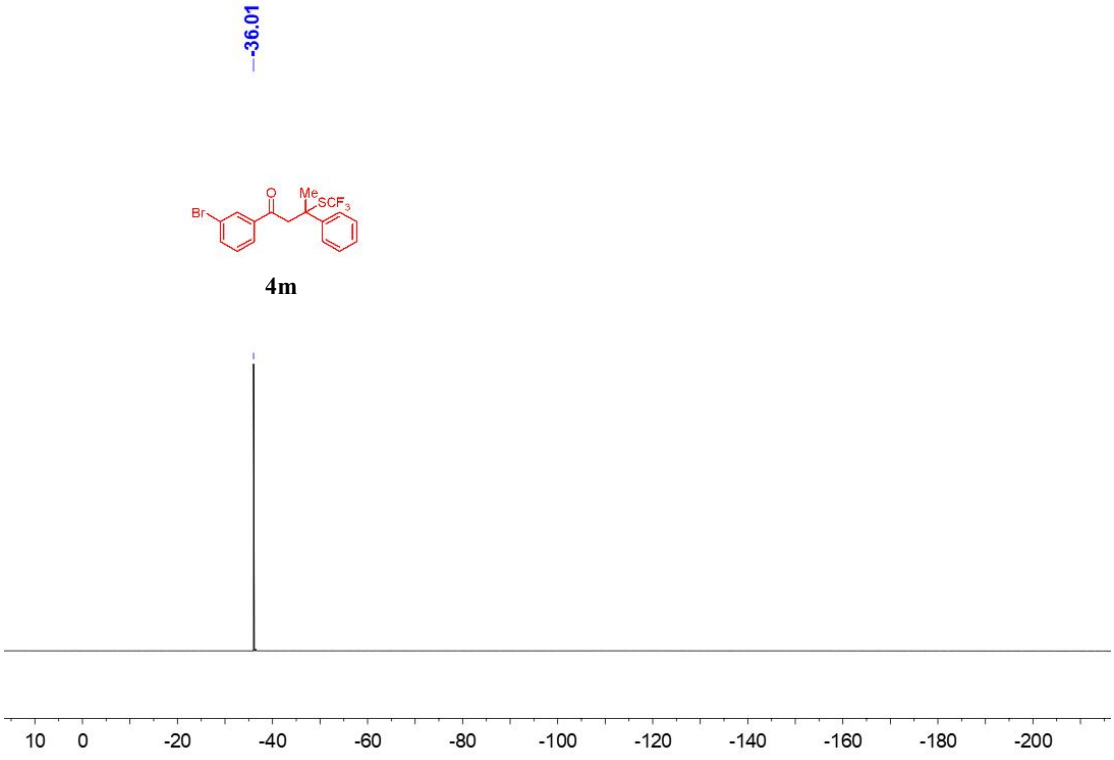
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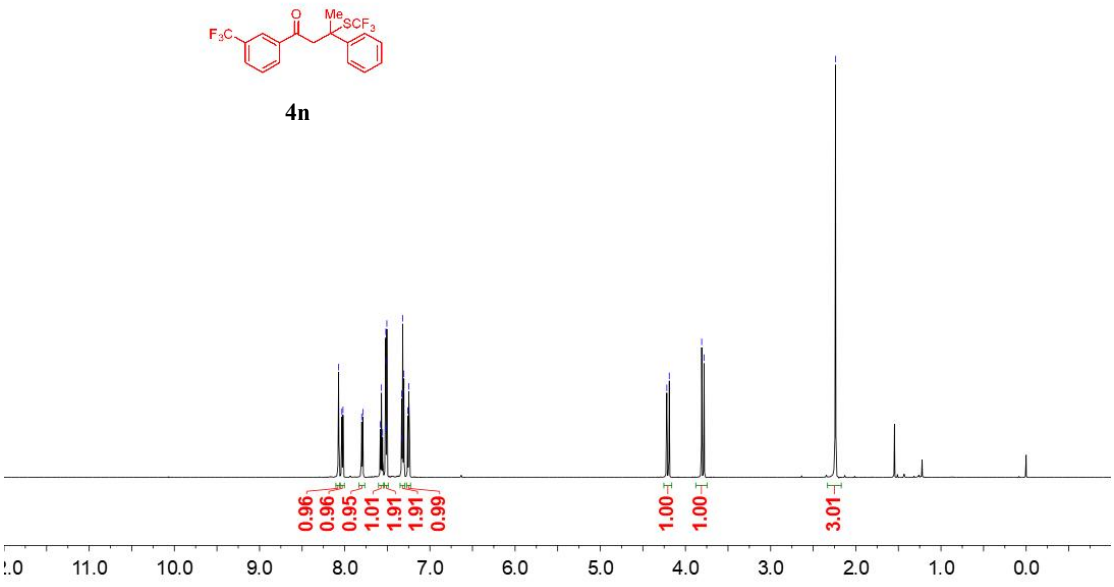
2.238

4m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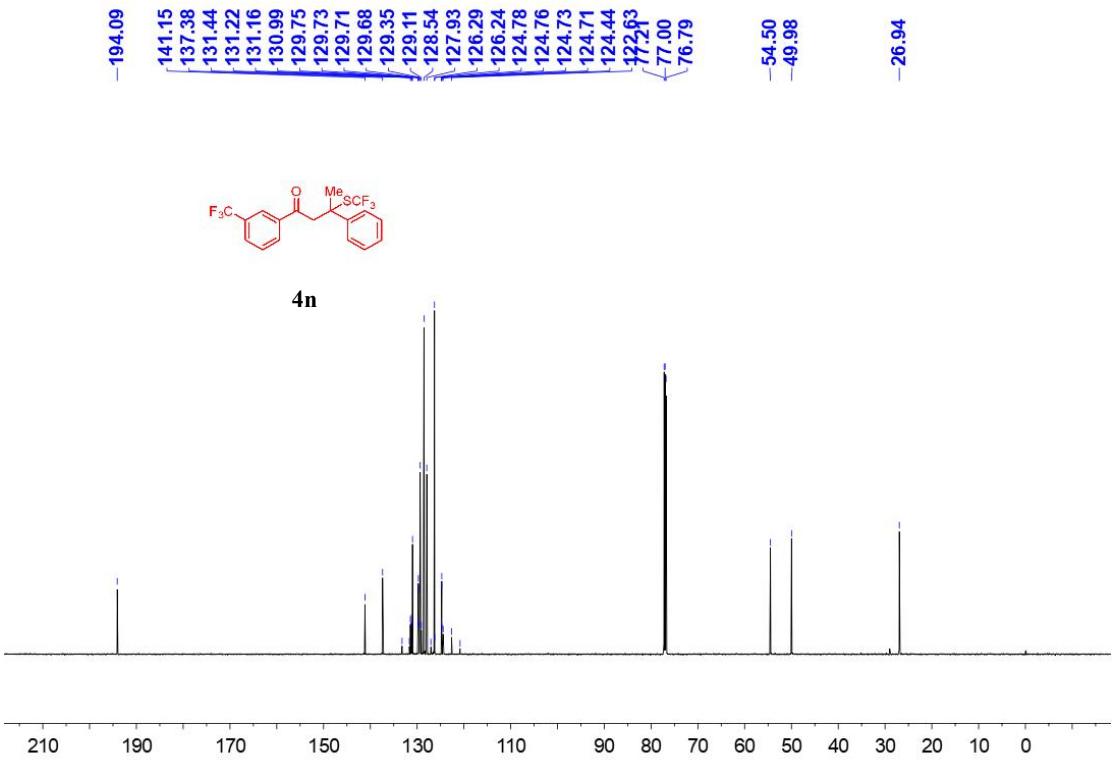
4n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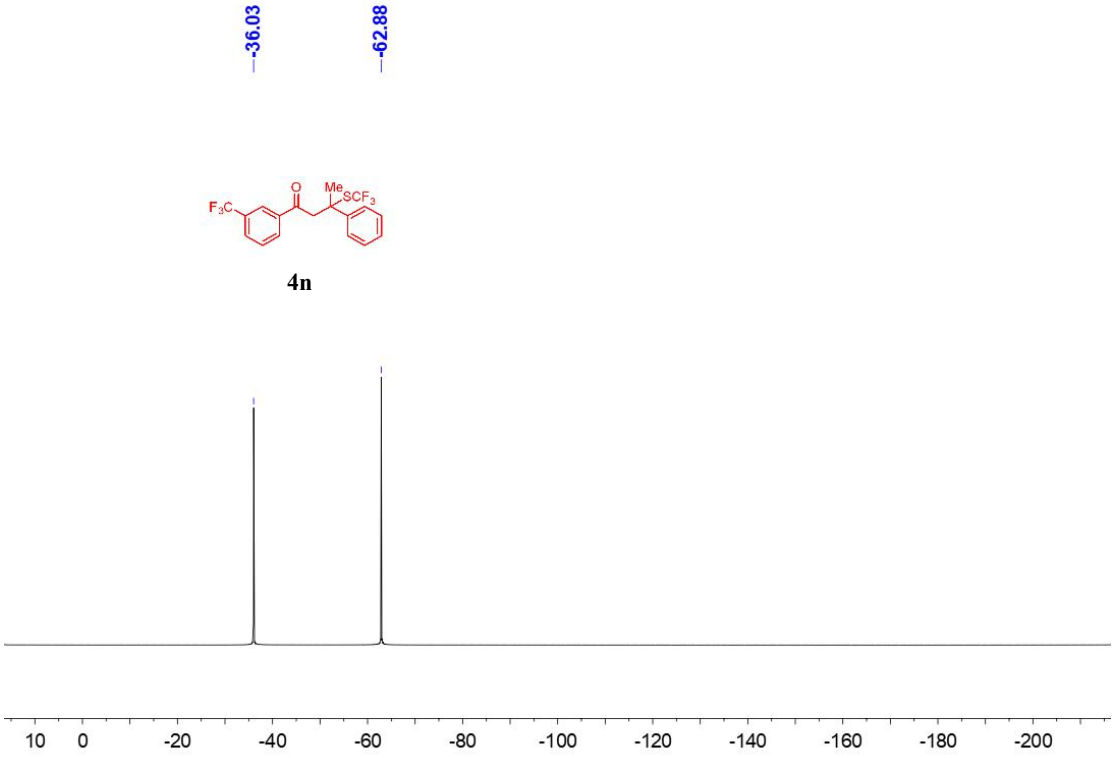
4n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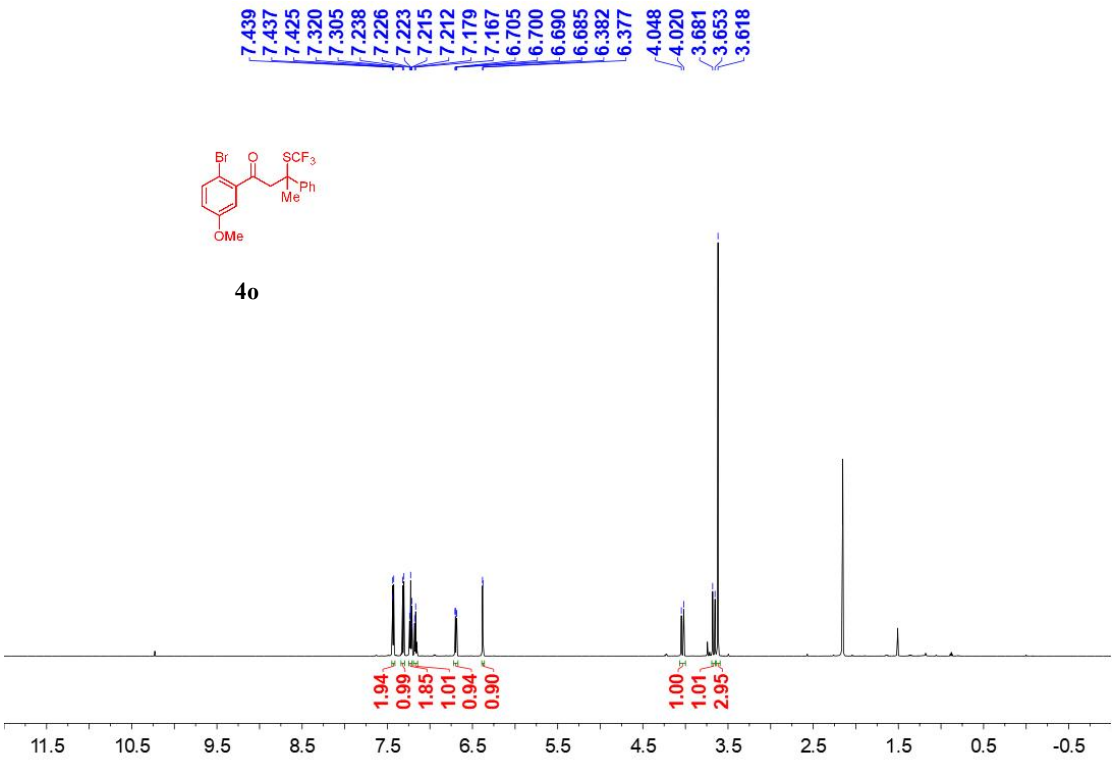
4n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OMe
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1.01
2.95
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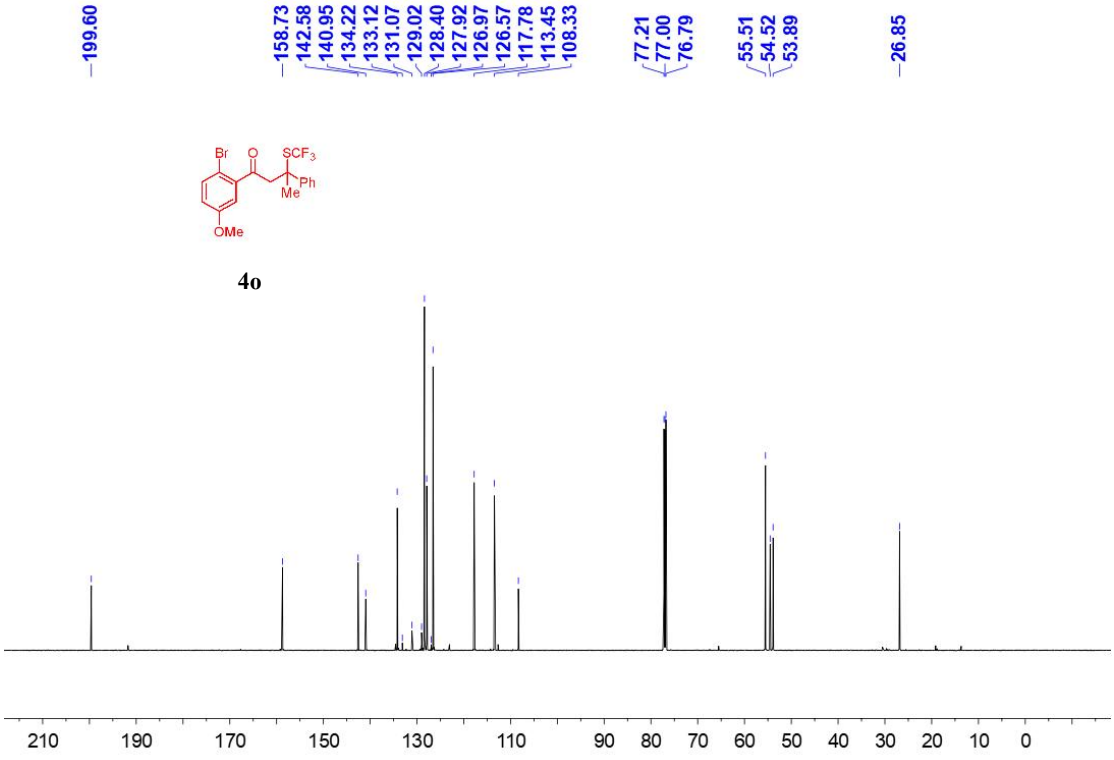
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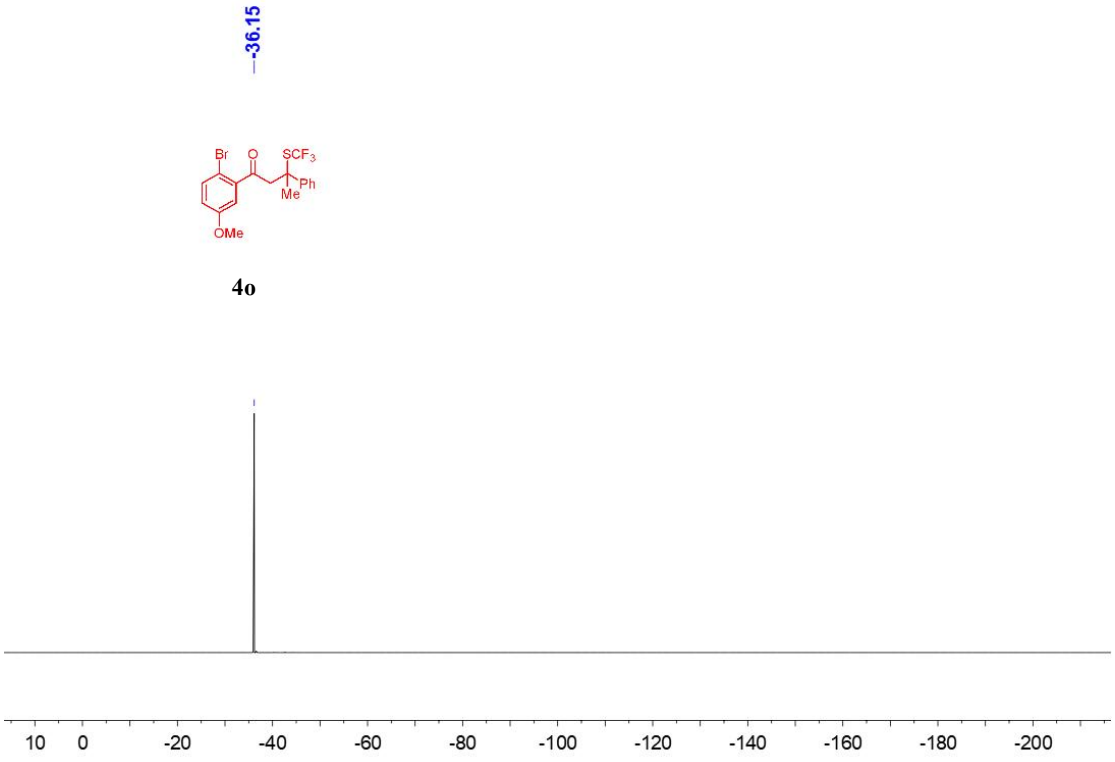
4o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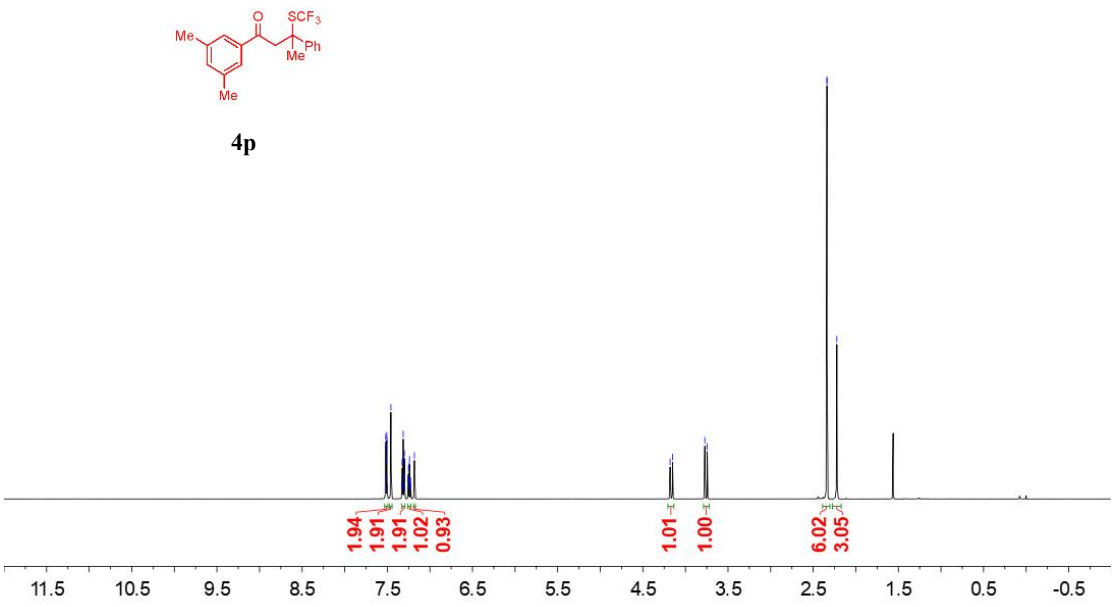
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6.02
3.05
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10.5
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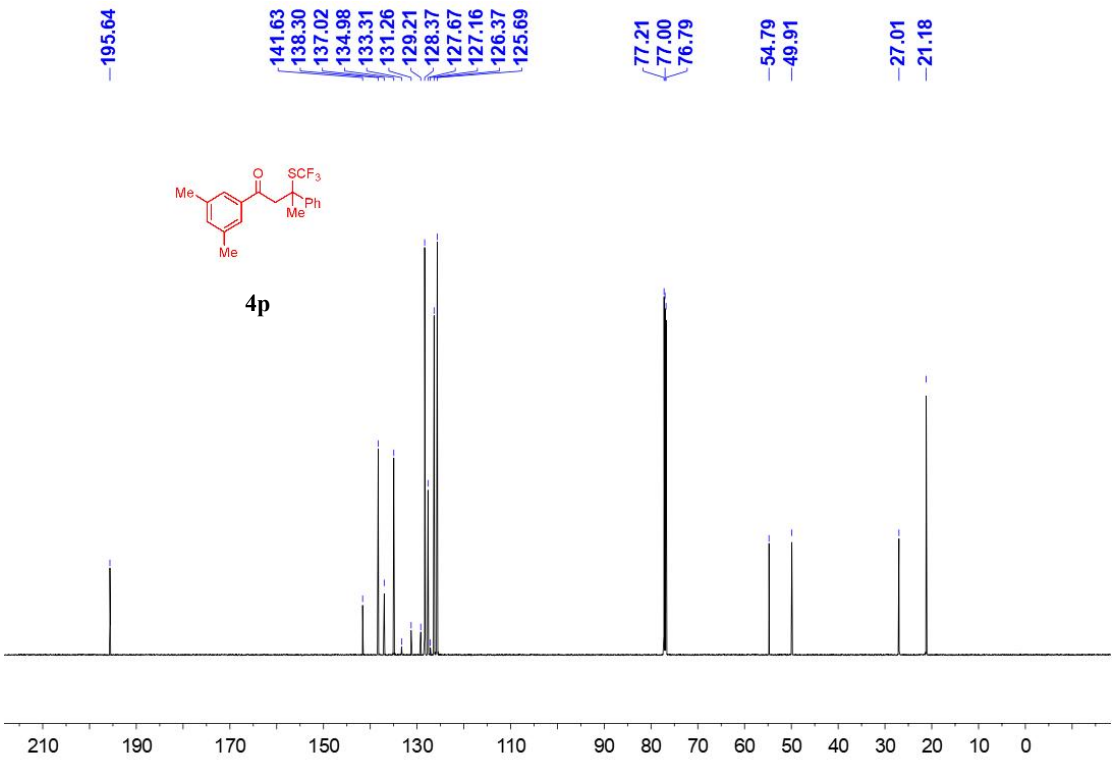
4p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27.01
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SCF3
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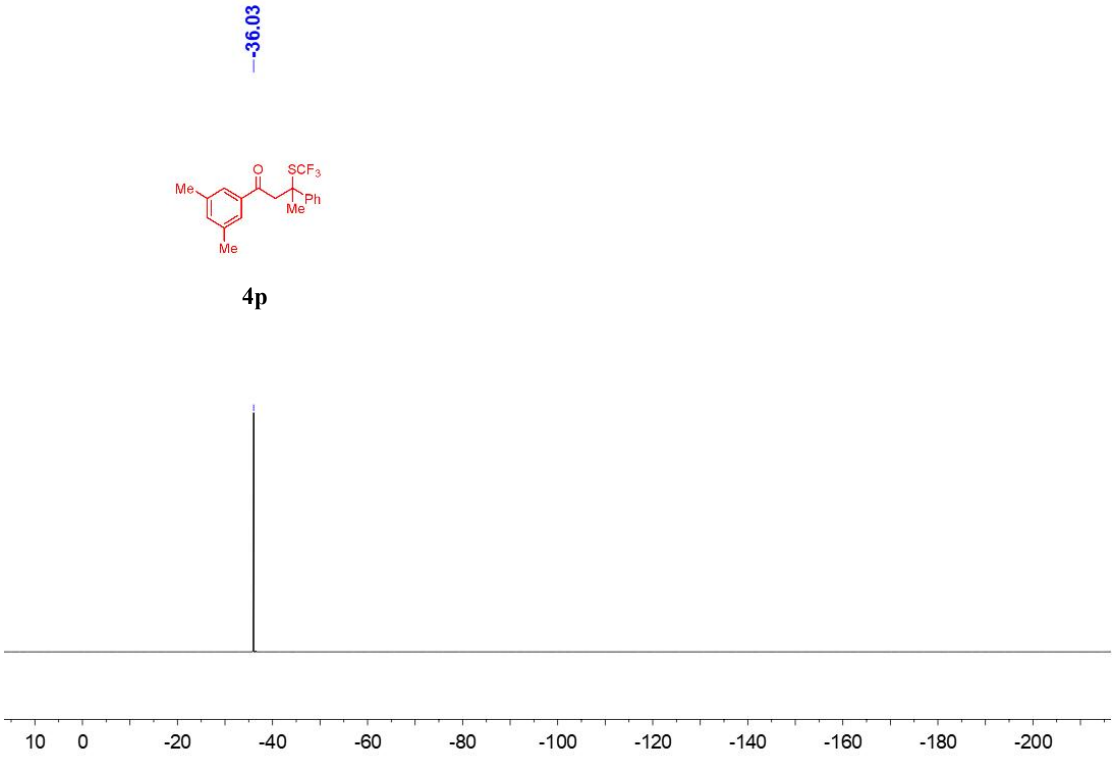
4p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SCF3
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Me
Me
Me
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-100
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-140
-160
-180
-200

4p


7.931
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7.815
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7.712
7.702
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7.520
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7.471
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7.436
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3.826
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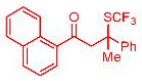
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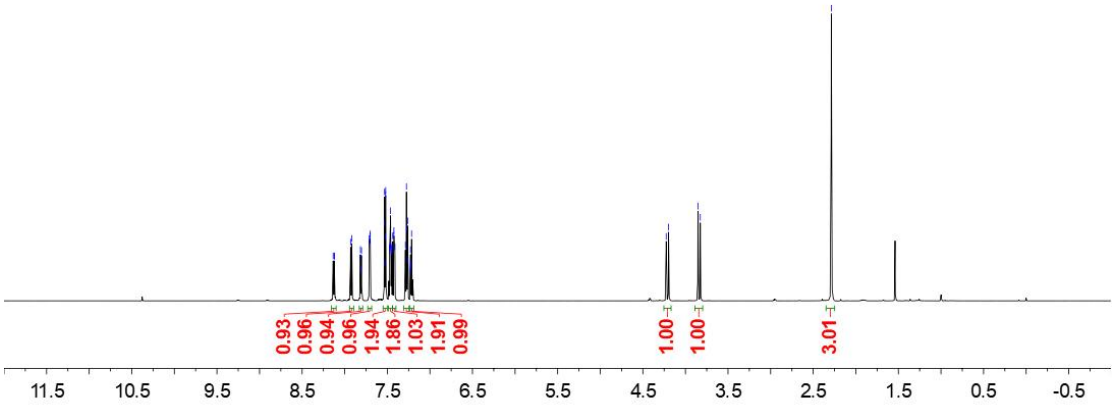
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1.00
1.00
3.01
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5


199.91
141.31
136.37
133.85
133.27
132.68
131.22
129.68
129.16
128.43
128.34
127.84
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77.00
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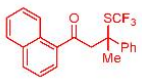
53.36
27.20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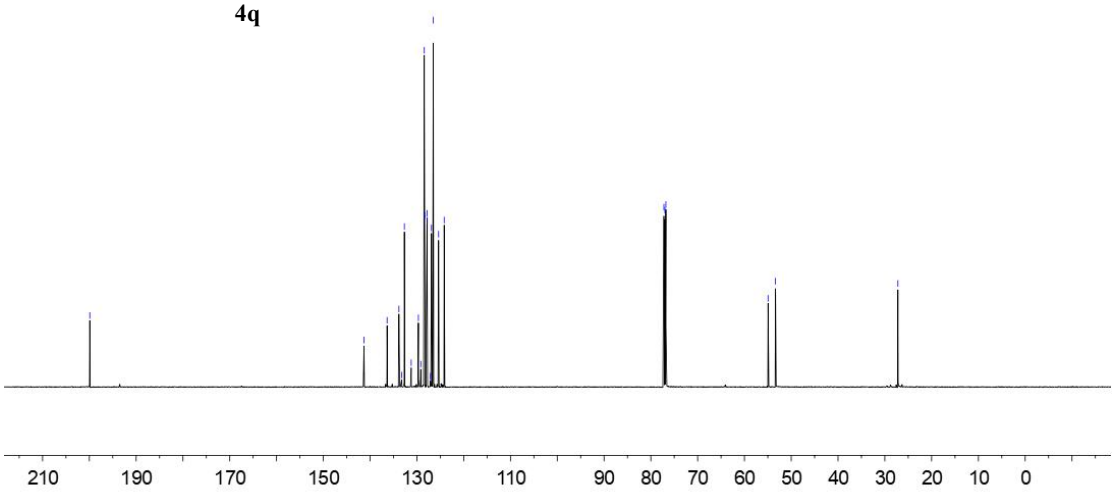
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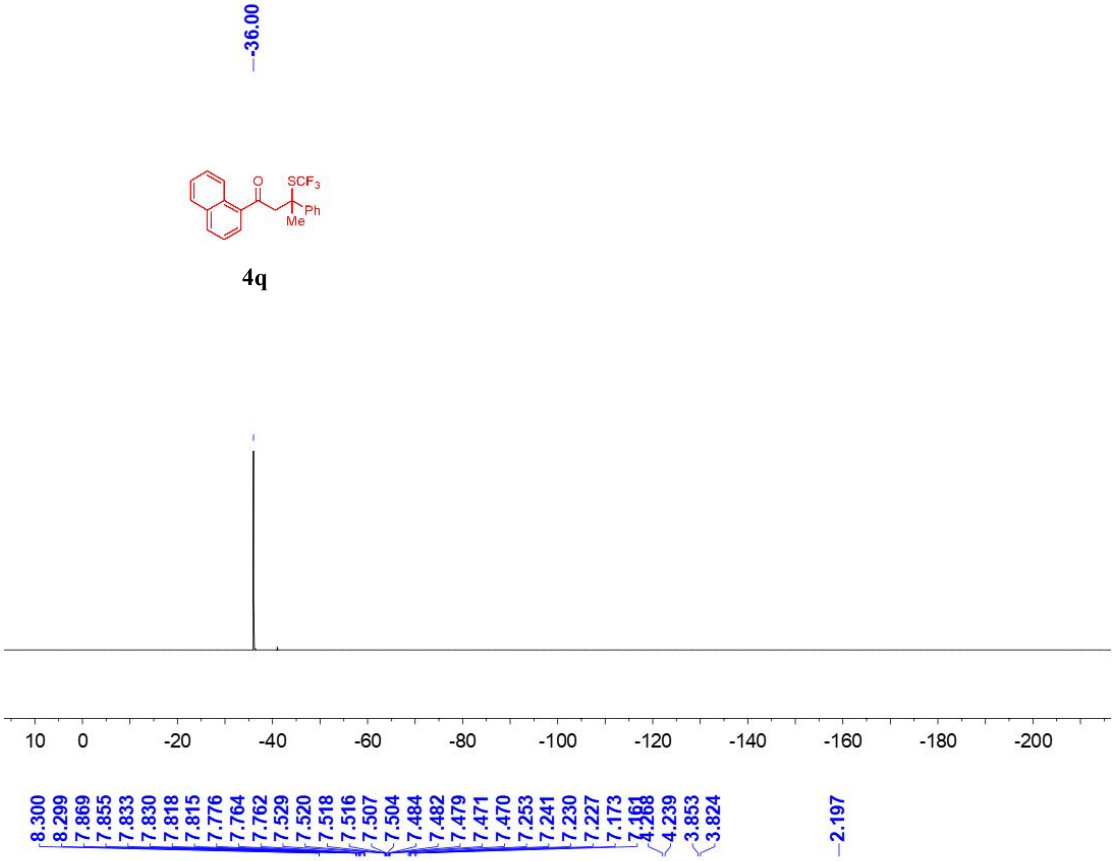
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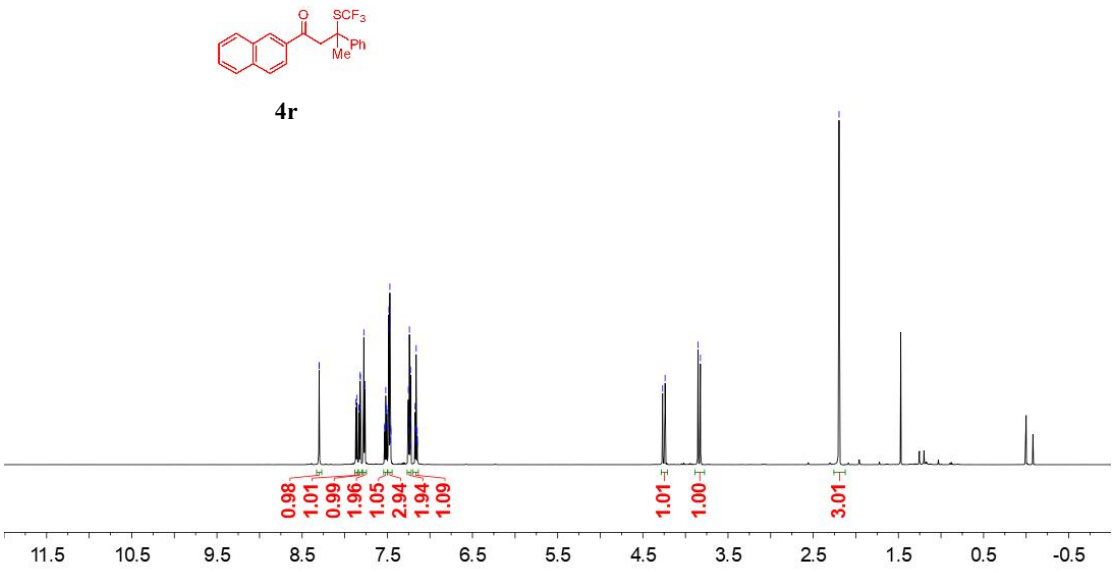
2.197

4q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SCF3
Ph
Me
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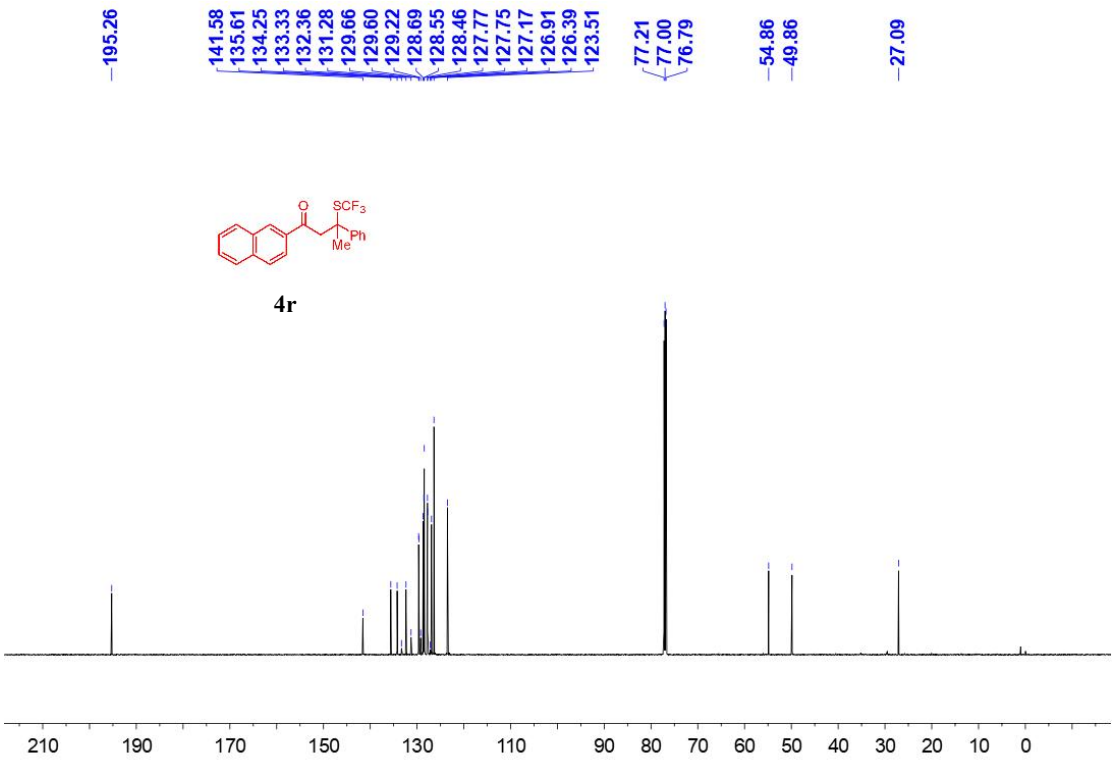
4r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77.00
76.79
54.86
49.86
27.09
O
SCF3
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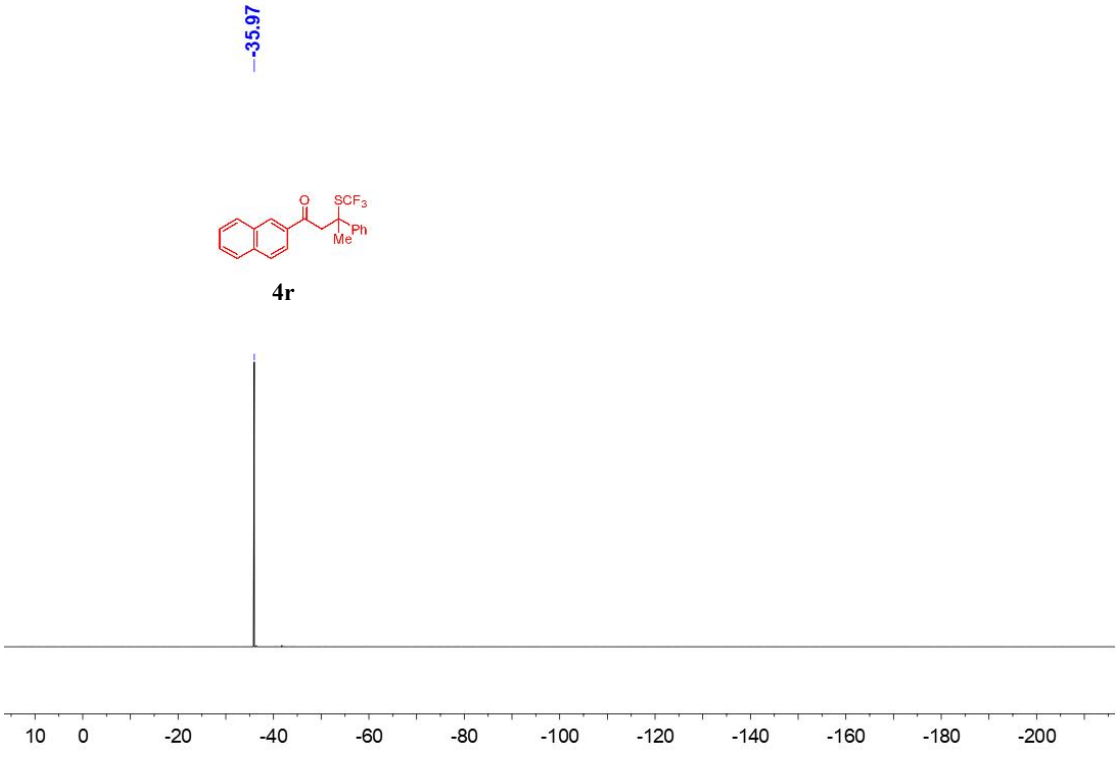
4r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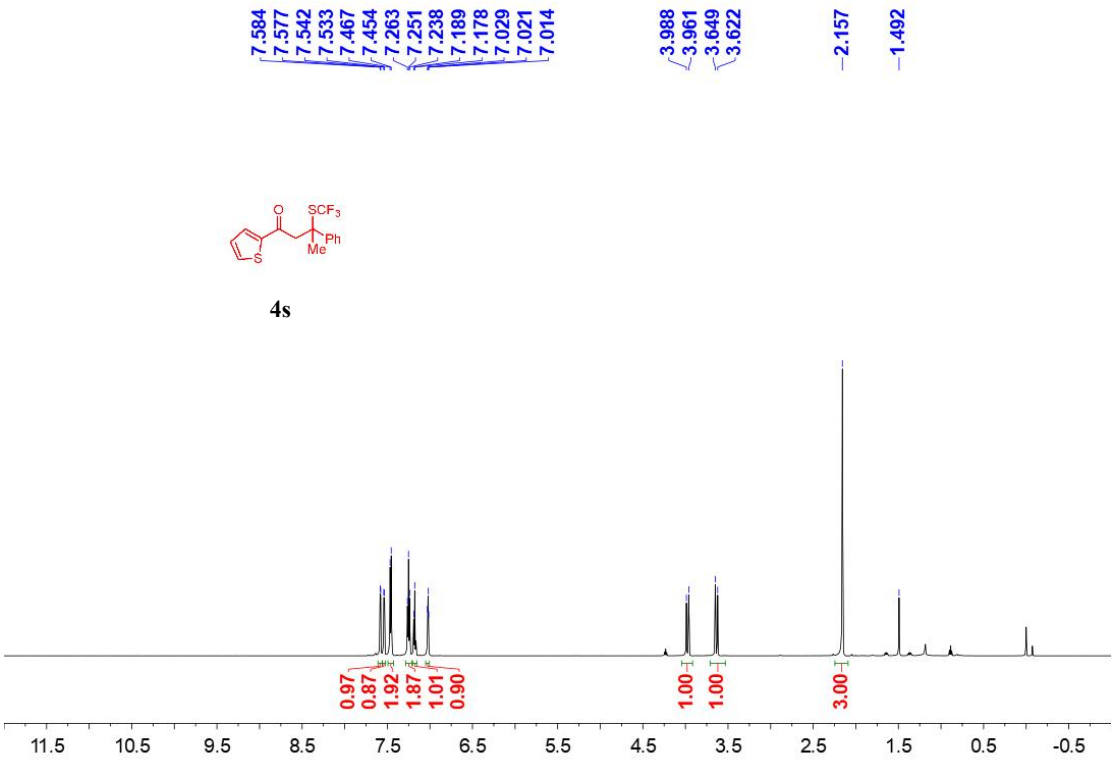
4r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7.021
7.014
3.988
3.961
3.649
3.622
2.157
1.492
SCF3
Ph
Me
S
O
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0.87
1.92
1.87
1.01
0.90
1.00
1.00
3.00
11.5
10.5
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6.5
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4.5
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2.5
1.5
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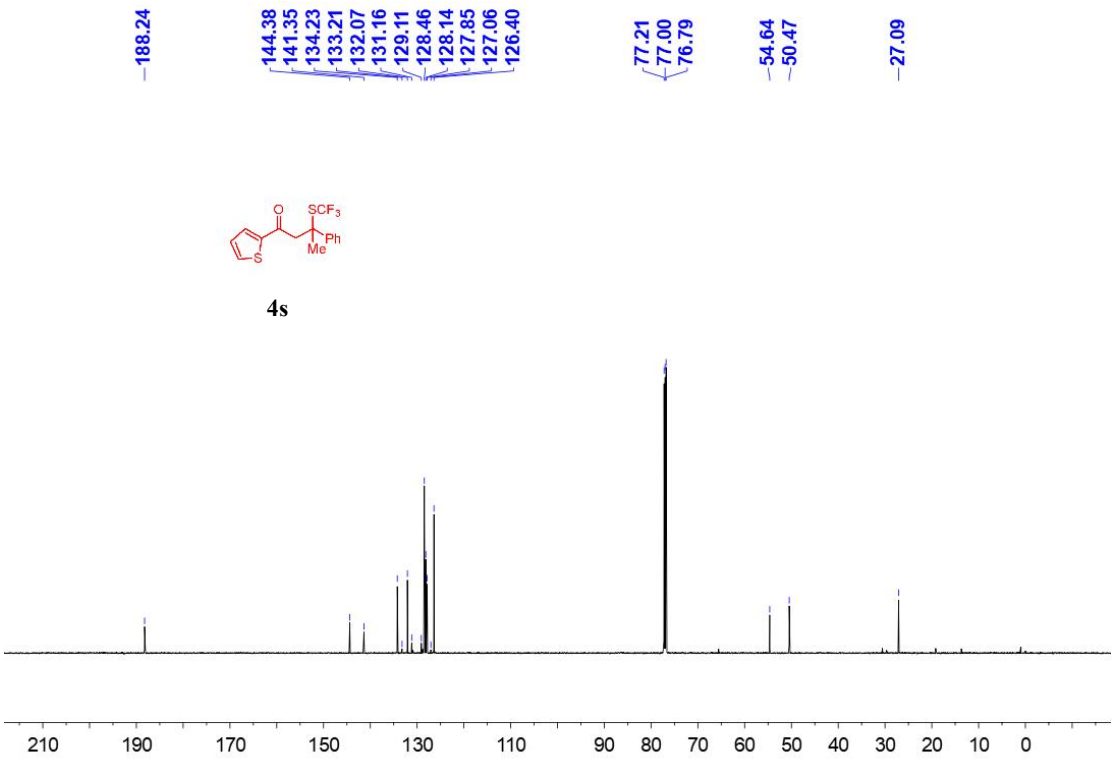
4s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129.11
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127.06
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77.00
76.79
54.64
50.47
27.09
SCF3
Ph
Me
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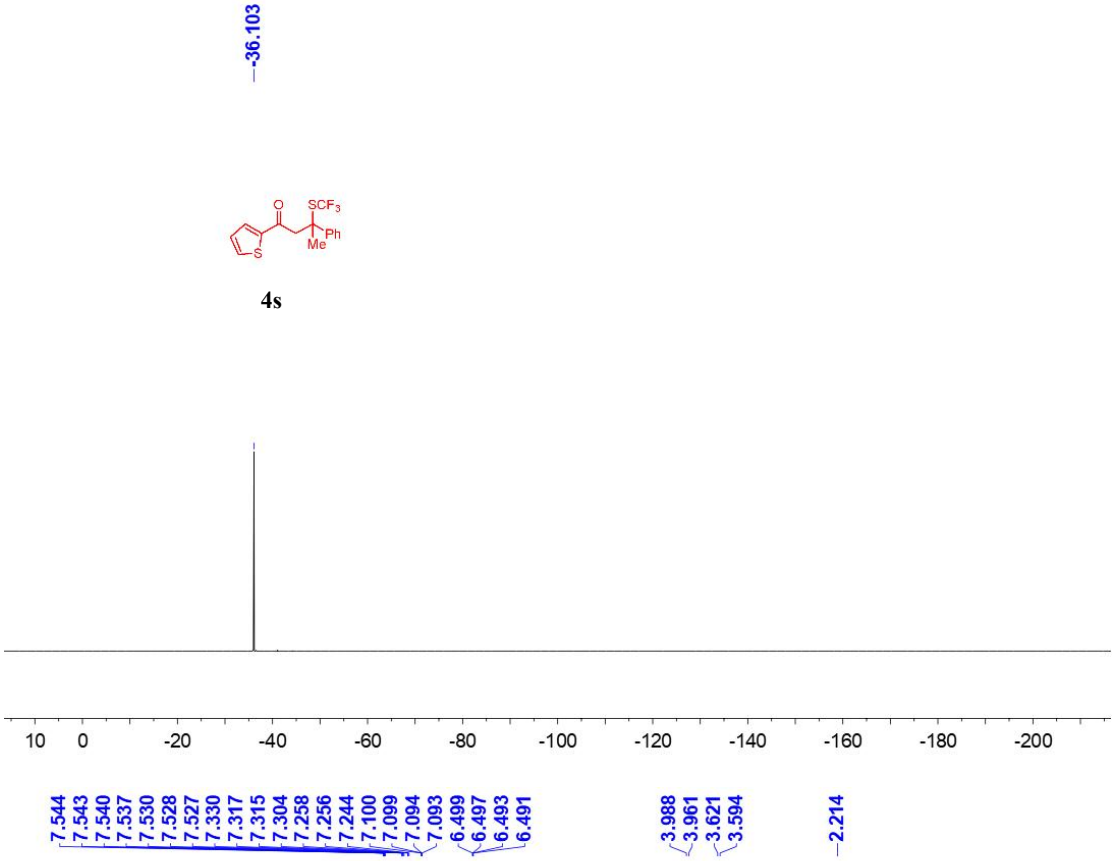
20
10
0

4s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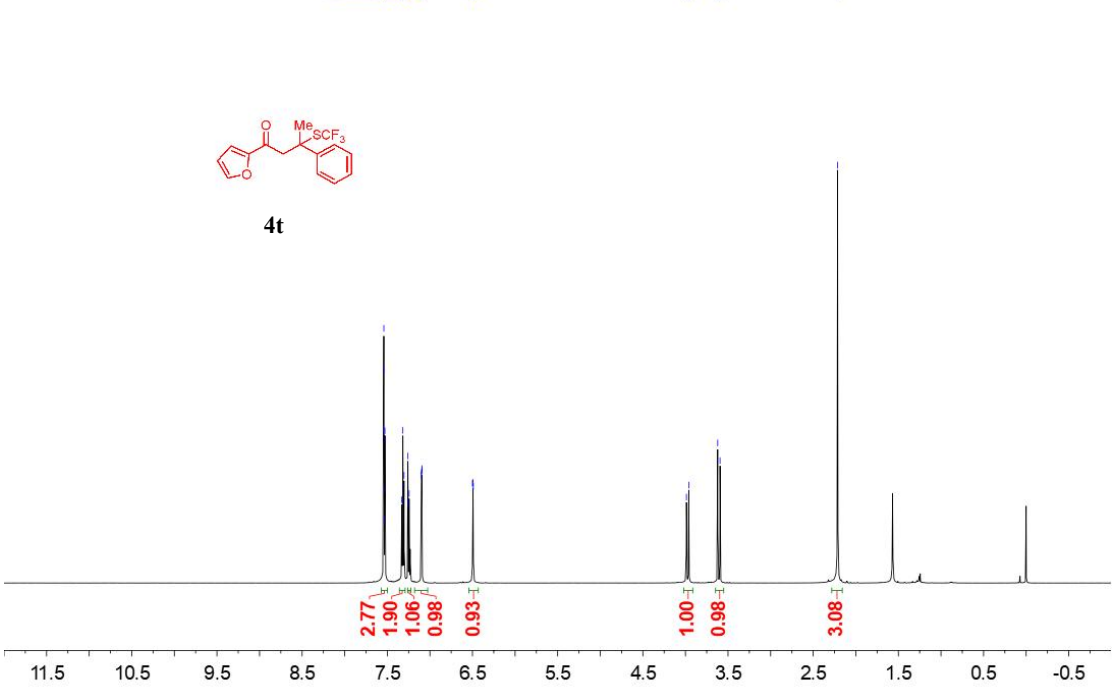
4s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7.315
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7.256
7.244
7.100
7.099
7.094
7.093
6.499
6.497
6.493
6.491
3.988
3.961
3.621
3.594
2.214
Me
SCF3
O
2.77
1.90
1.06
0.98
0.93
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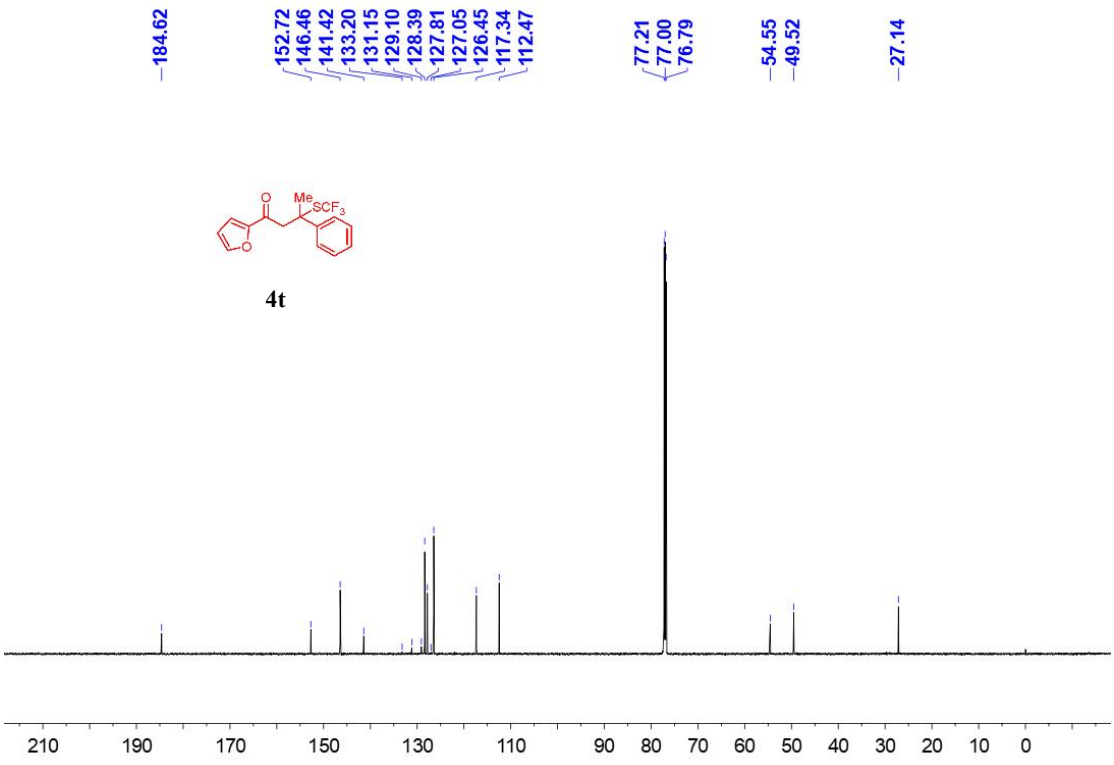
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

4t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117.34
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77.21
77.00
76.79
54.55
49.52
27.14
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60
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20
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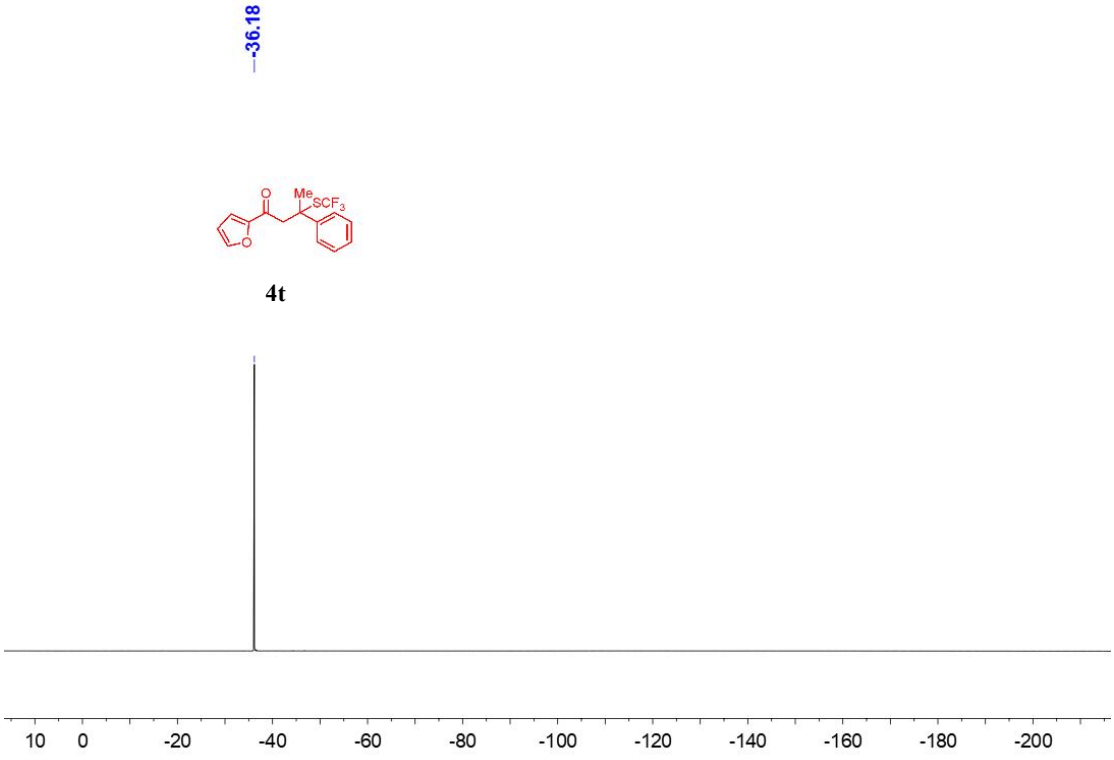
0

4t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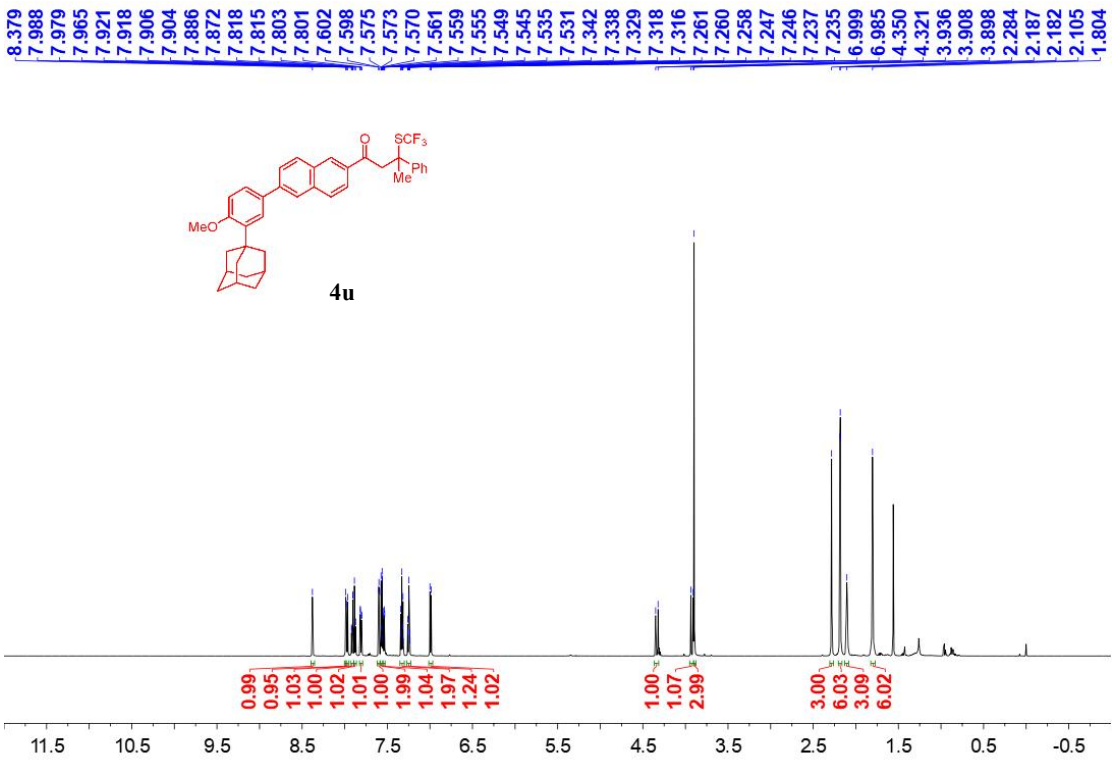
4t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7.815
7.803
7.801
7.602
7.598
7.575
7.573
7.570
7.561
7.559
7.555
7.549
7.545
7.535
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7.329
7.318
7.316
7.261
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7.246
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1.804
SCF3
Ph
Me
MeO
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1.00
1.02
1.01
1.00
1.99
1.04
1.97
1.24
1.02
1.00
1.07
2.99
3.00
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3.09
6.02
11.5
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7.5
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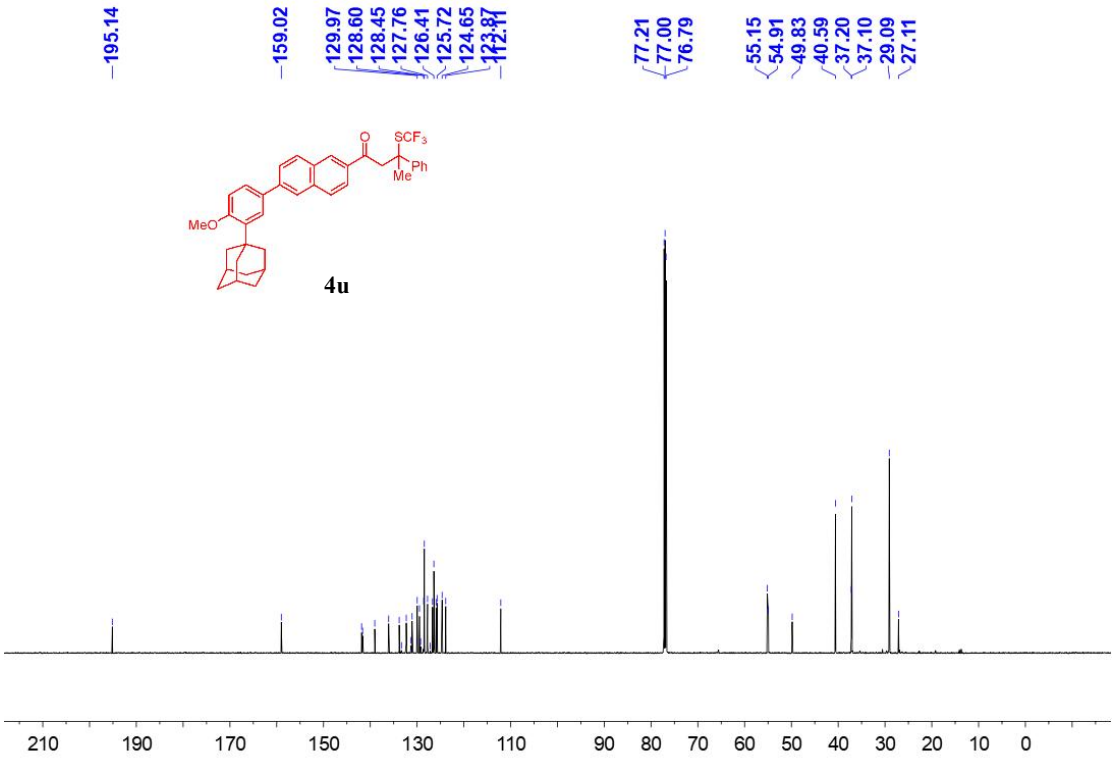
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112.11
77.21
77.00
76.79
55.15
54.91
49.83
40.59
37.20
37.10
29.09
27.11
SCF3
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MeO
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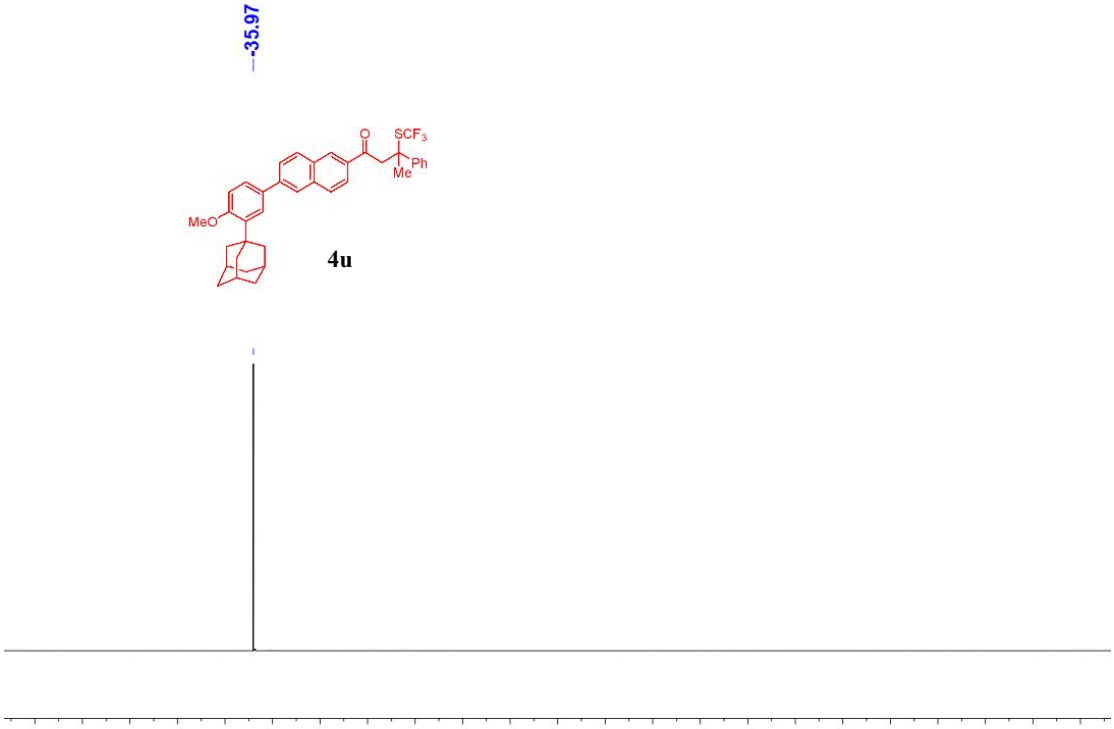
0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Me
MeO
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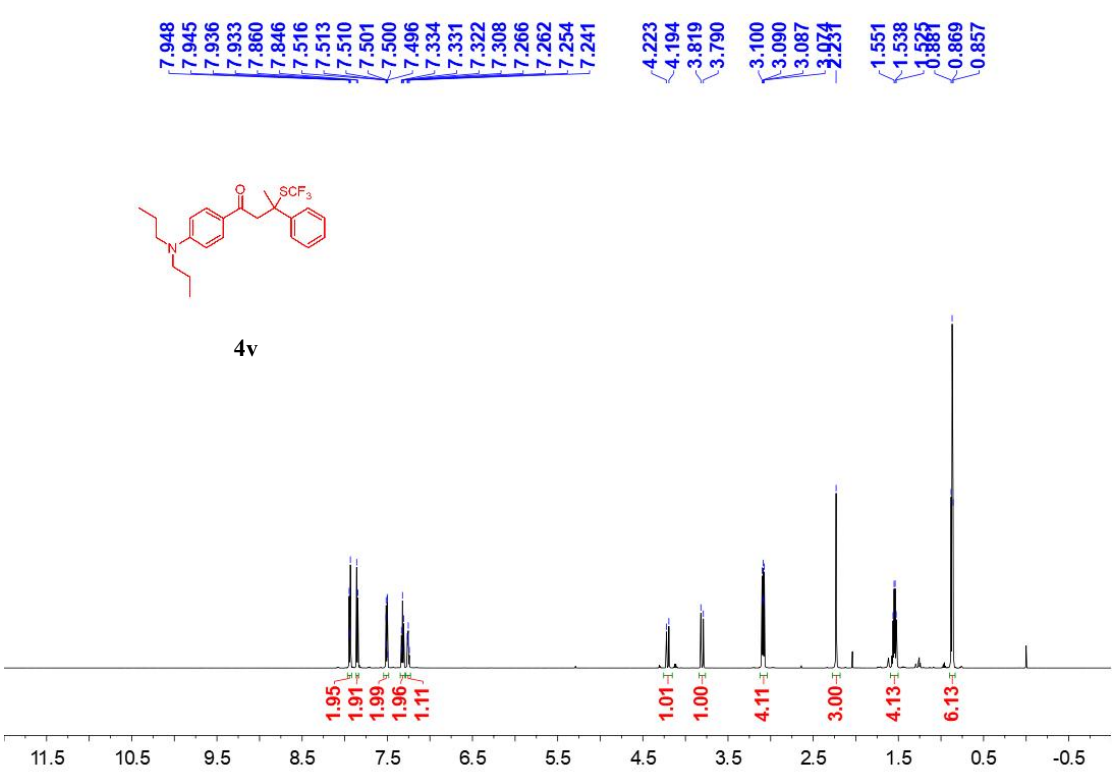
4u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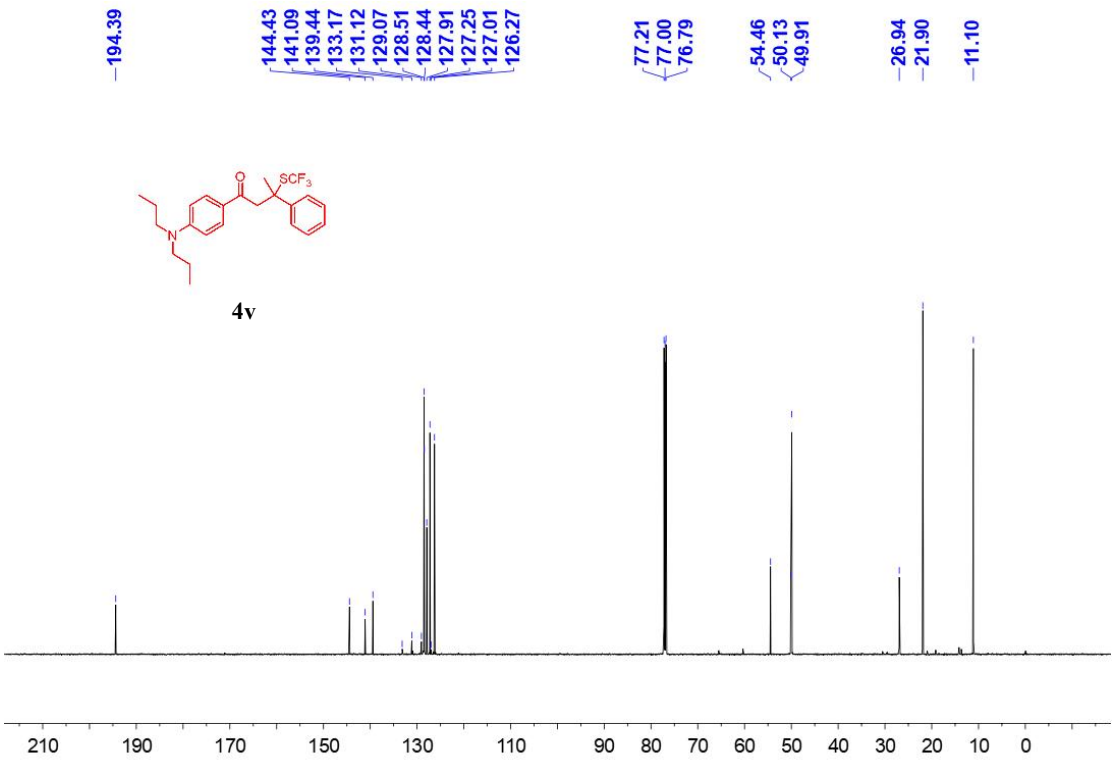
4.223 4.194 3.819 3.790
3.100 3.090 3.087
1.551 1.538
O
SCF3
N
1.95 1.91 1.99 1.96 1.11 1.01 1.00 4.11 3.00 4.13 6.13
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

4v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127.01
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77.00
76.79
54.46
50.13
49.91
26.94
21.90
11.10
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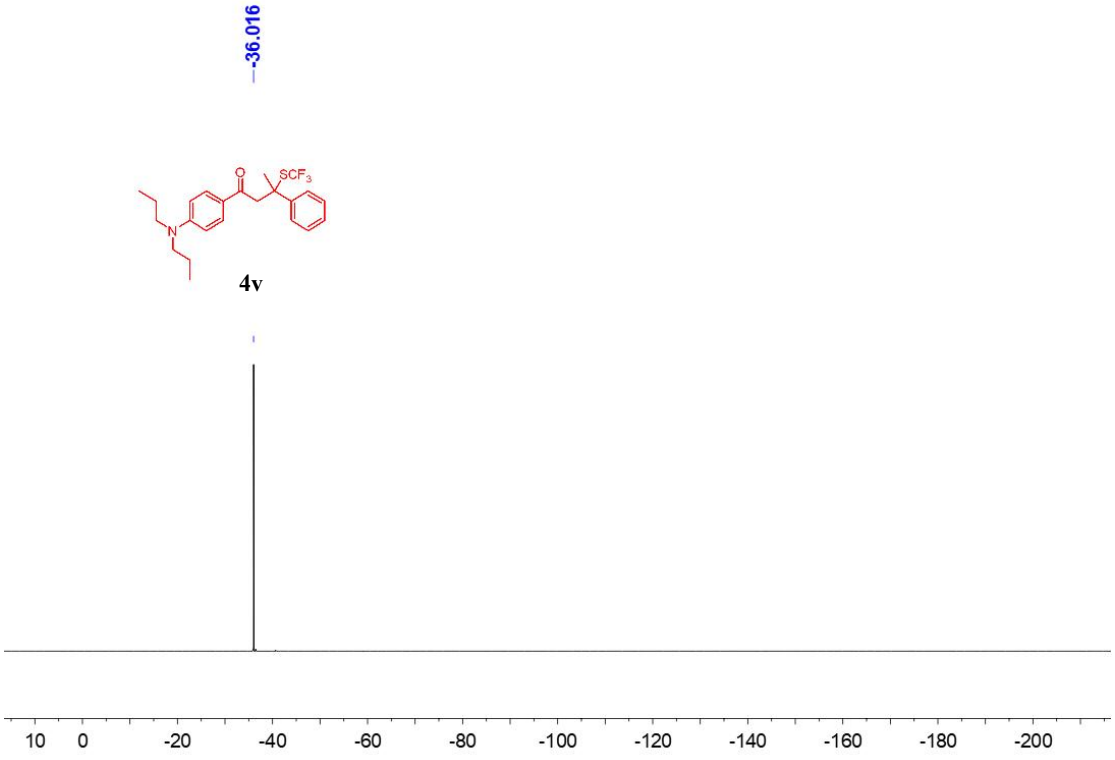
0

4v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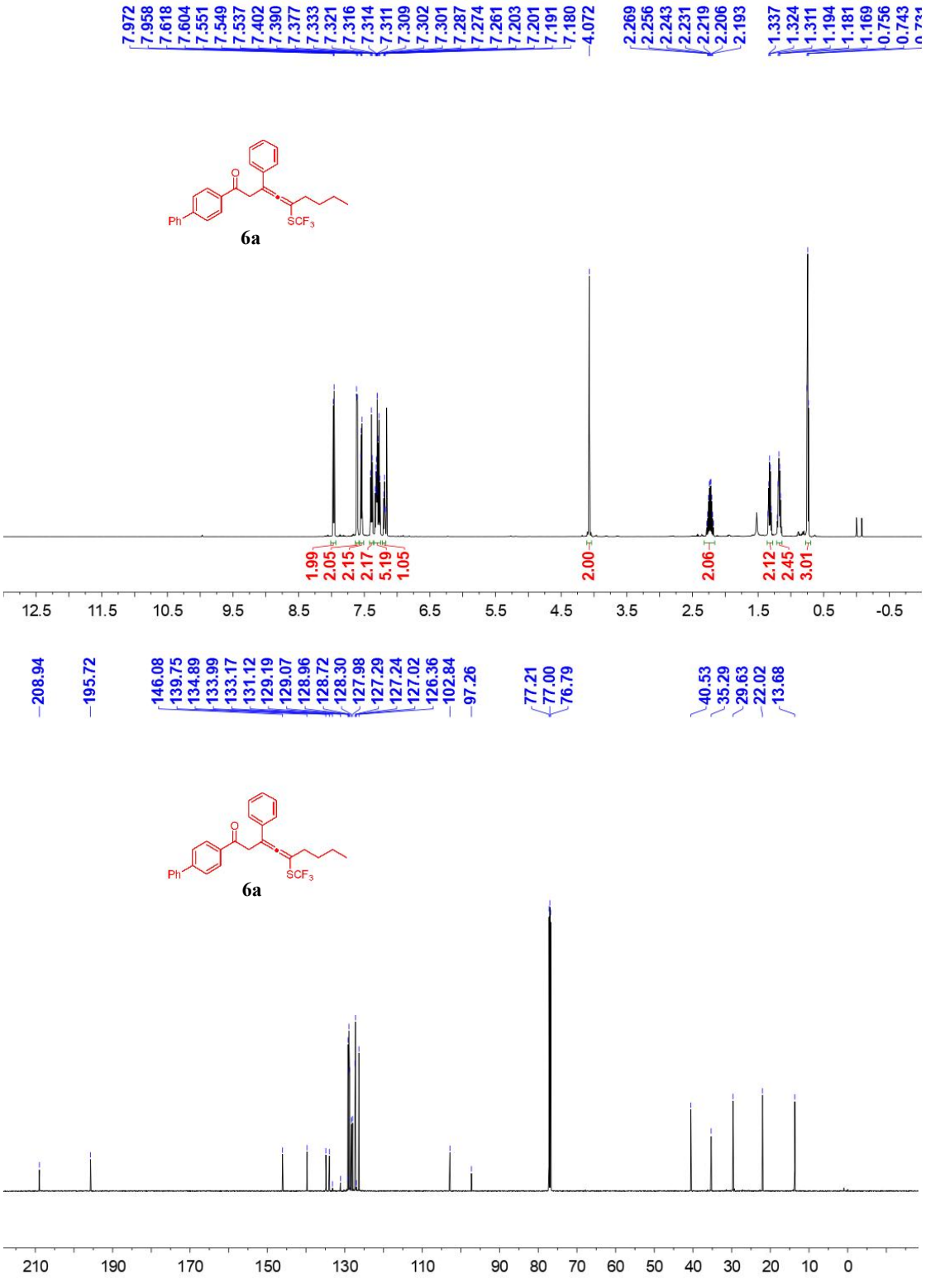
-180
-200

4v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7.302
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7.261
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7.201
7.191
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1.181
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0.743
Ph
SCF3
6a
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2.17
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1.05
2.00
2.06
2.12
2.45
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12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
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146.08
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133.17
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127.29
127.24
127.02
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22.02
13.68
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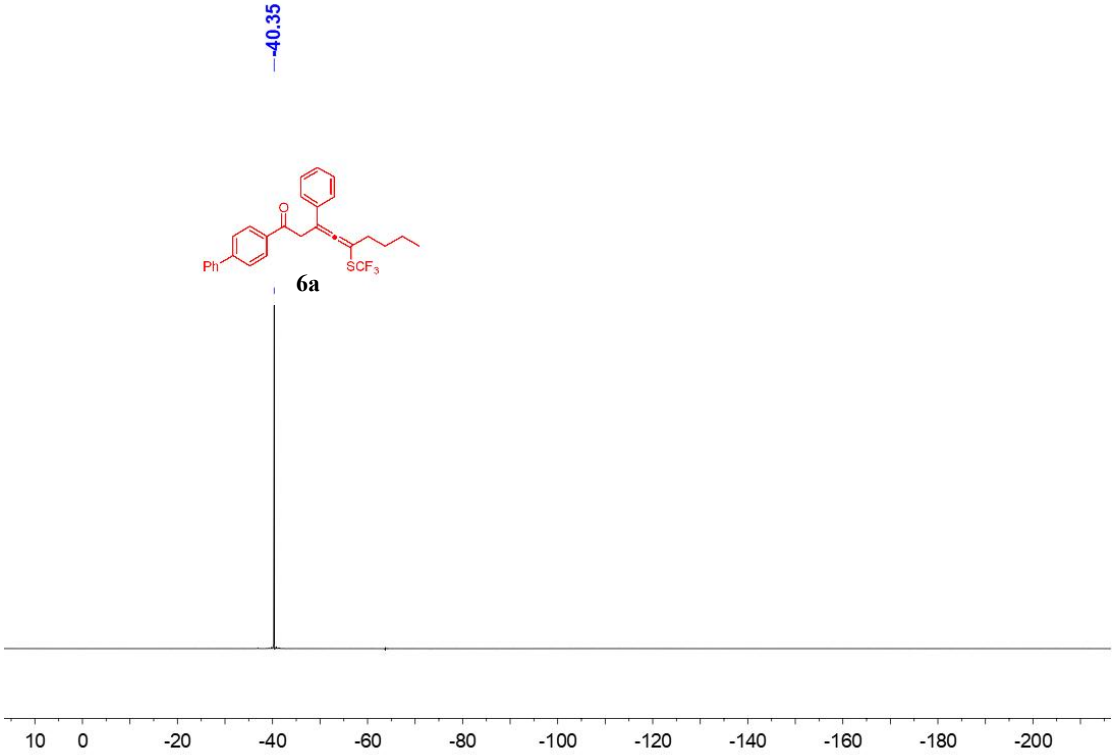
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6a
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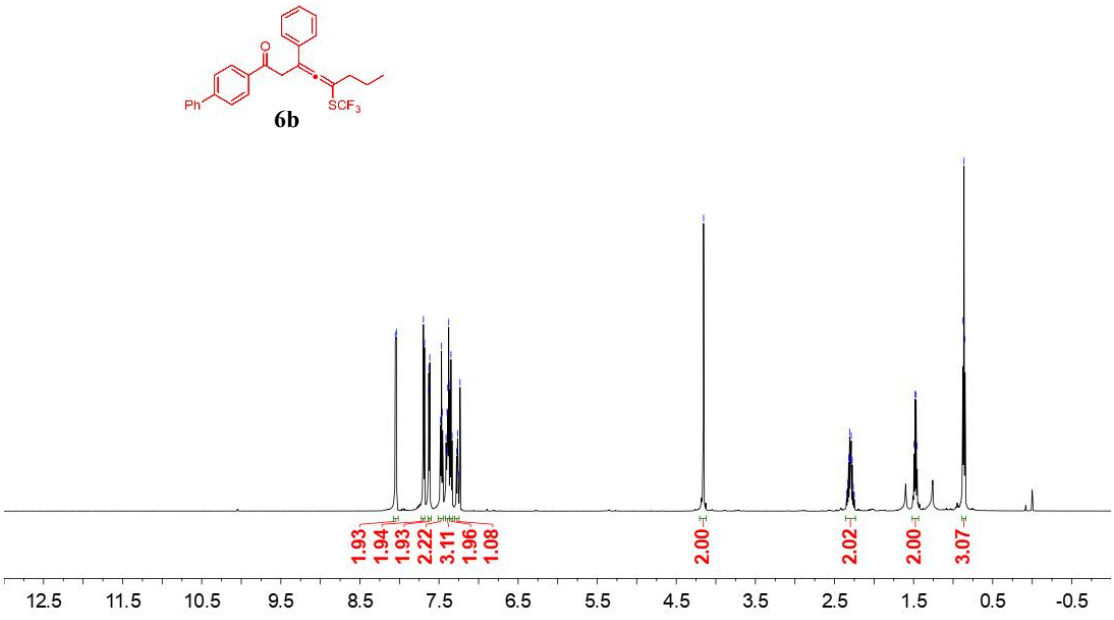
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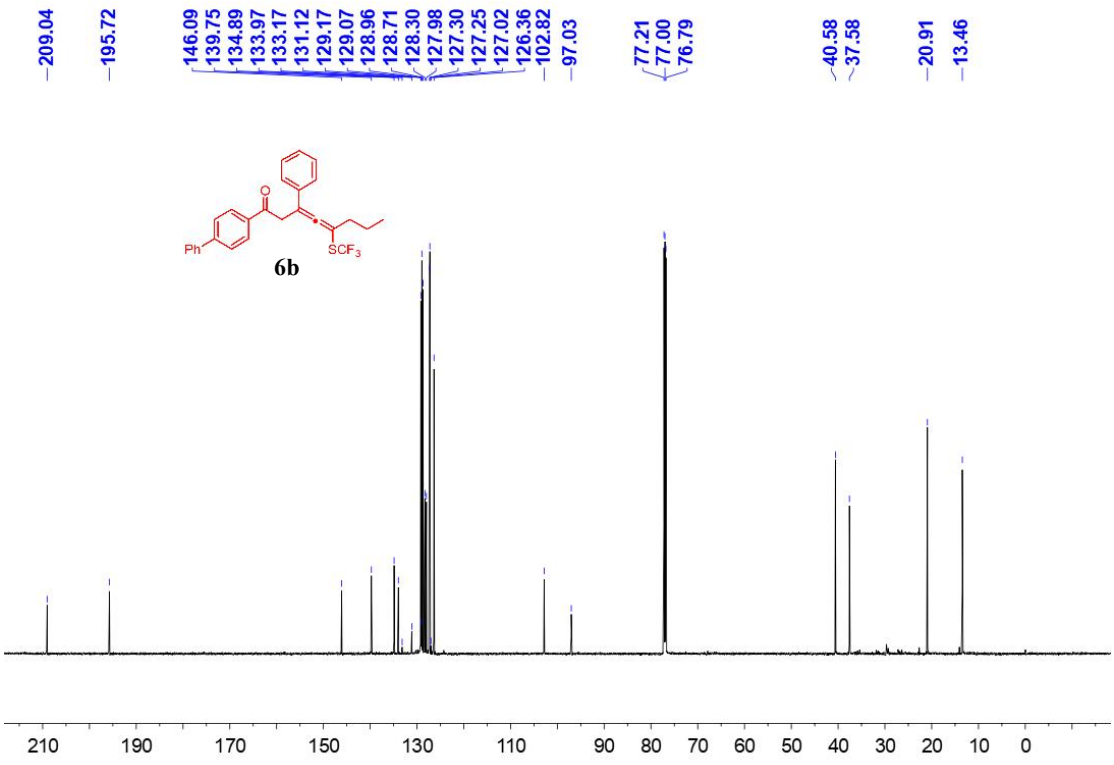
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77.00
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13.46
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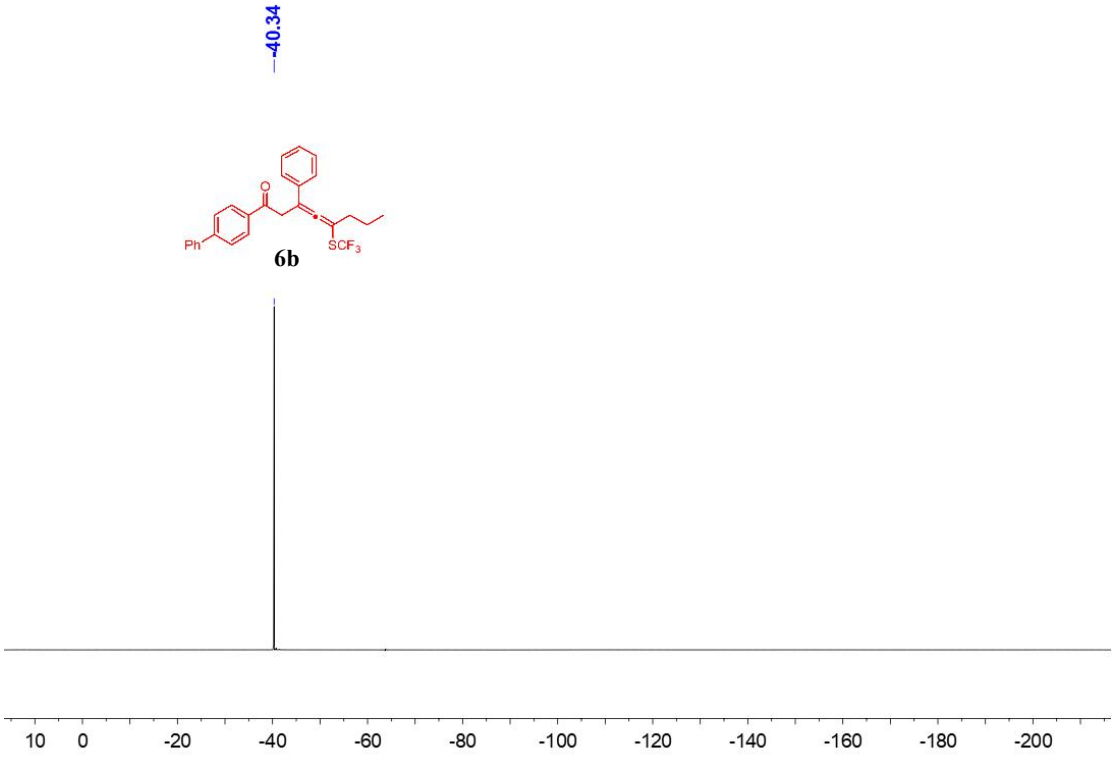
20
10
0

6b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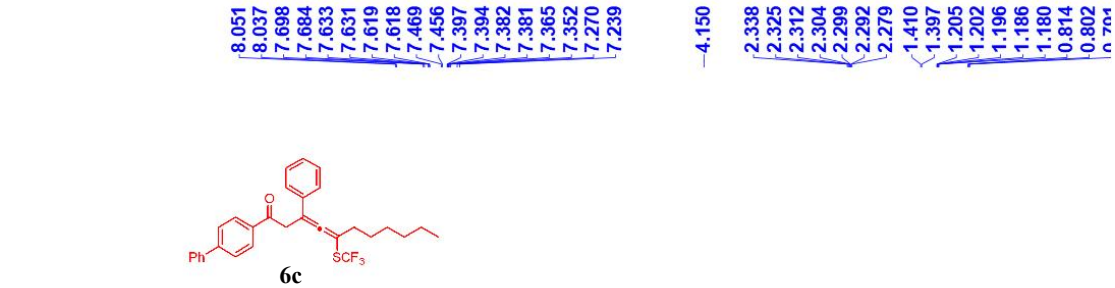
-180
-200

6b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2.312
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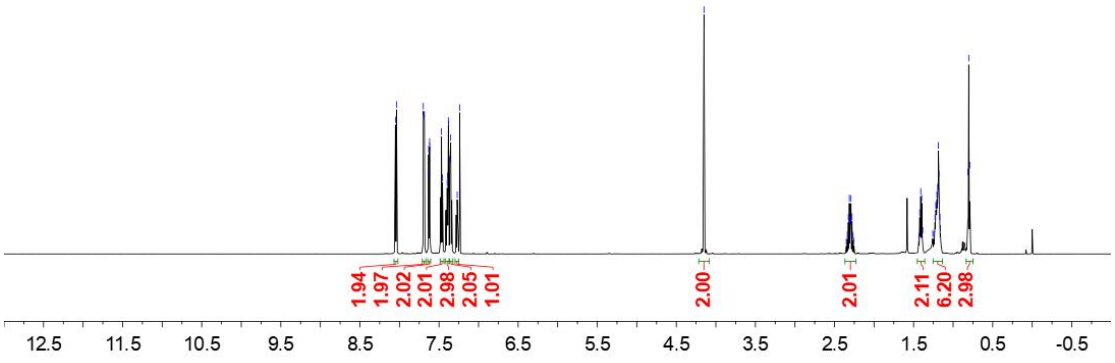
0.802

O
Ph
SCF3

6c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2.00
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2.11
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12.5
11.5
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7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5


208.95
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146.06
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Ph
SCF3

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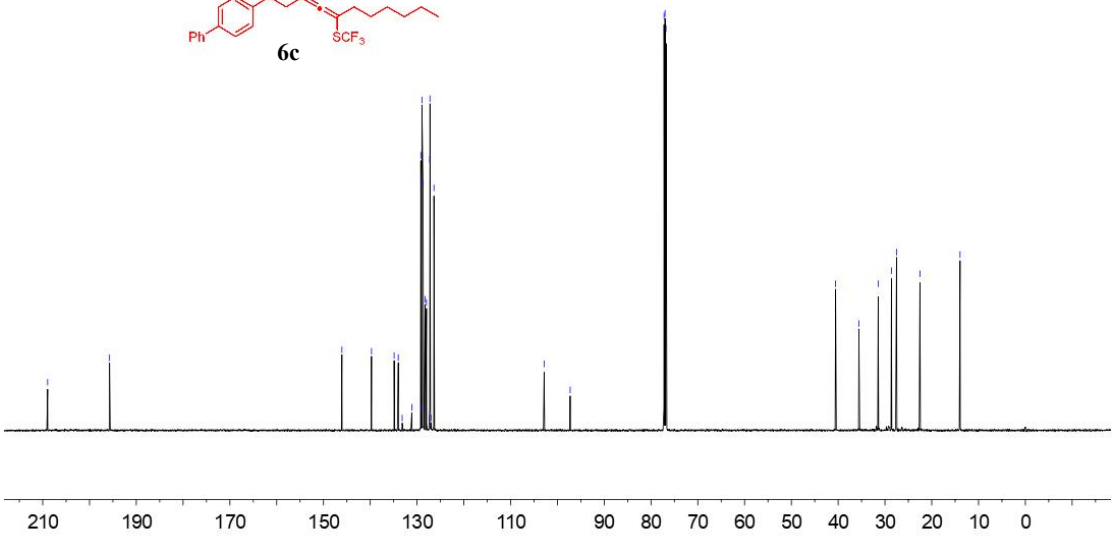
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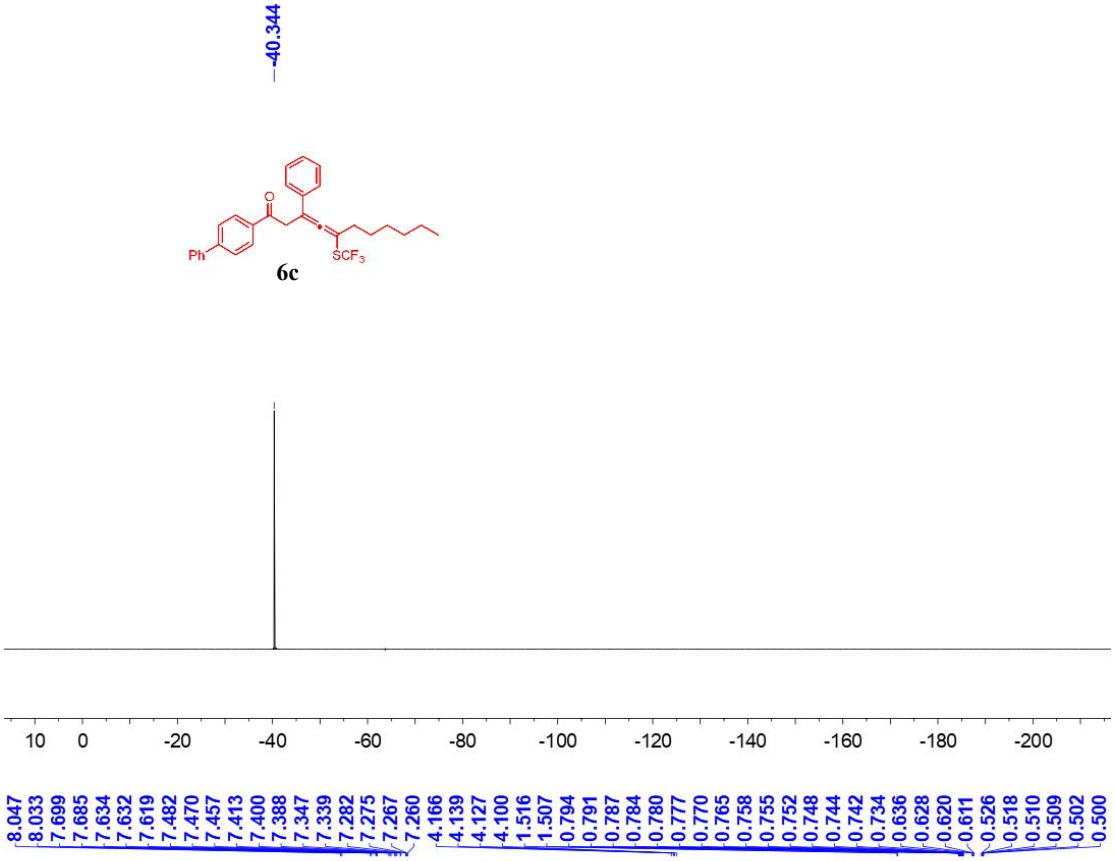
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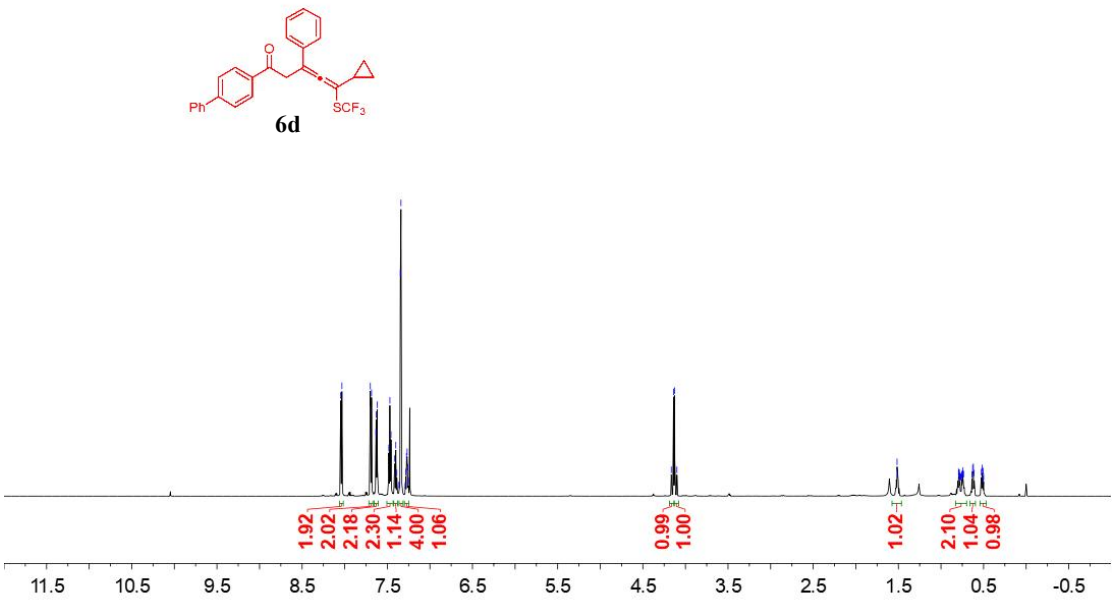
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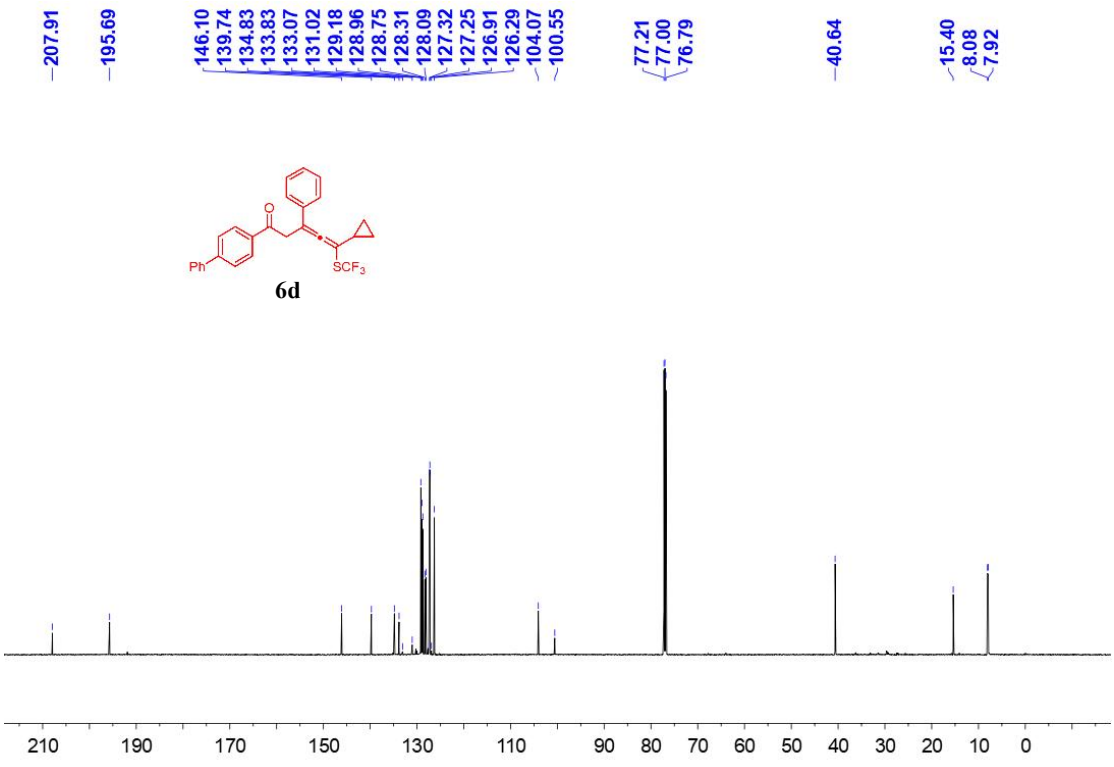
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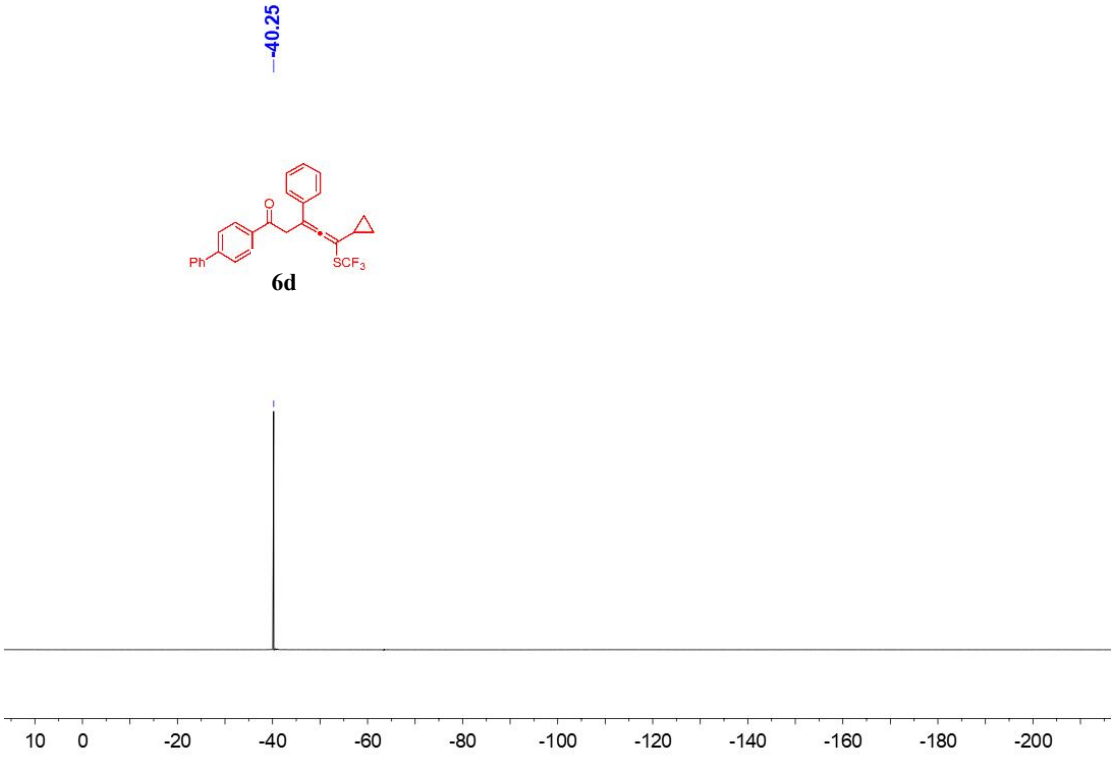
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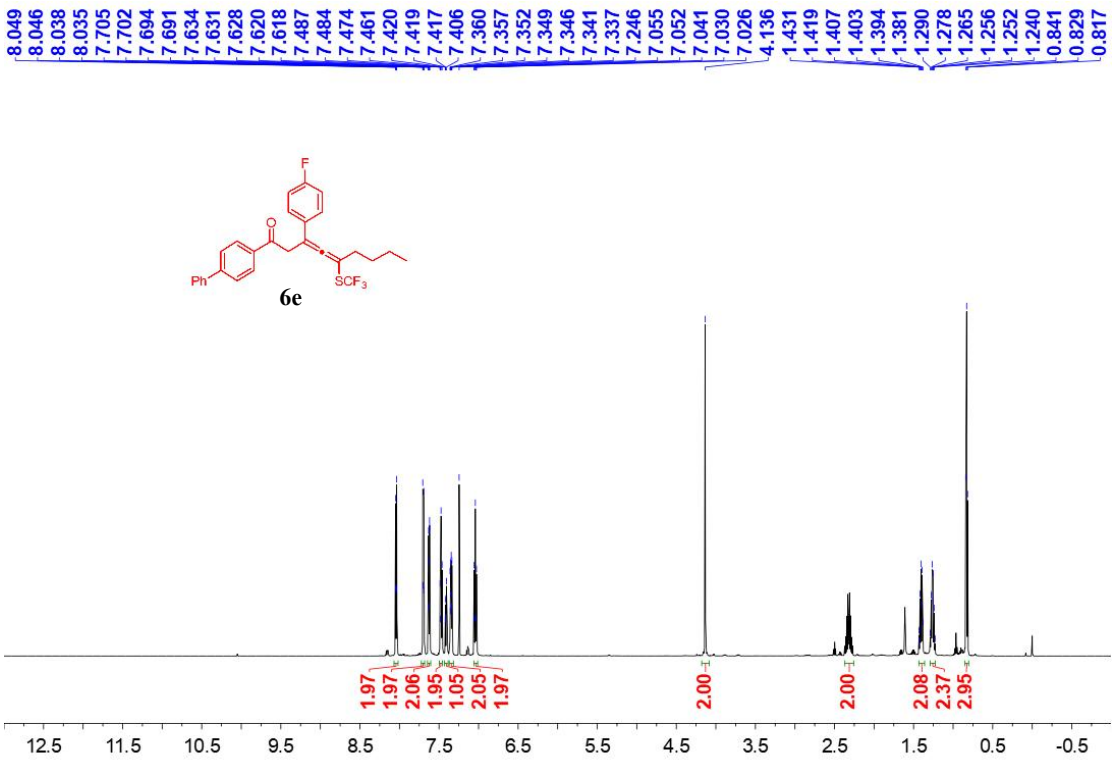
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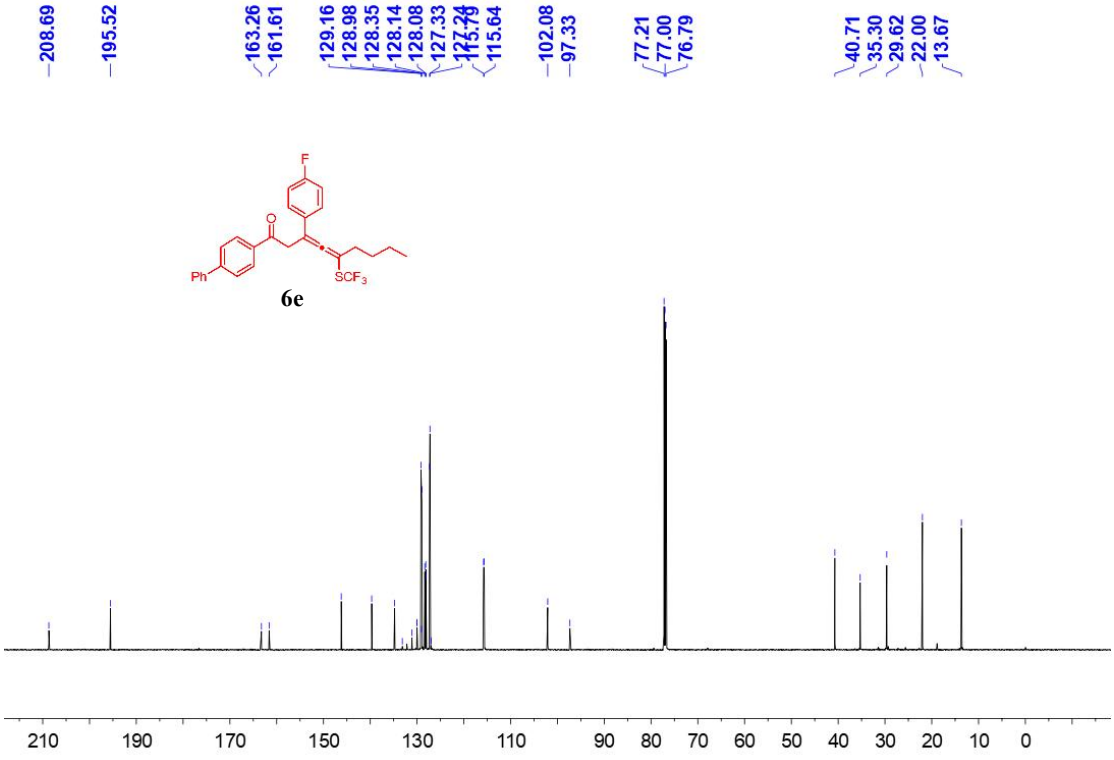
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22.00
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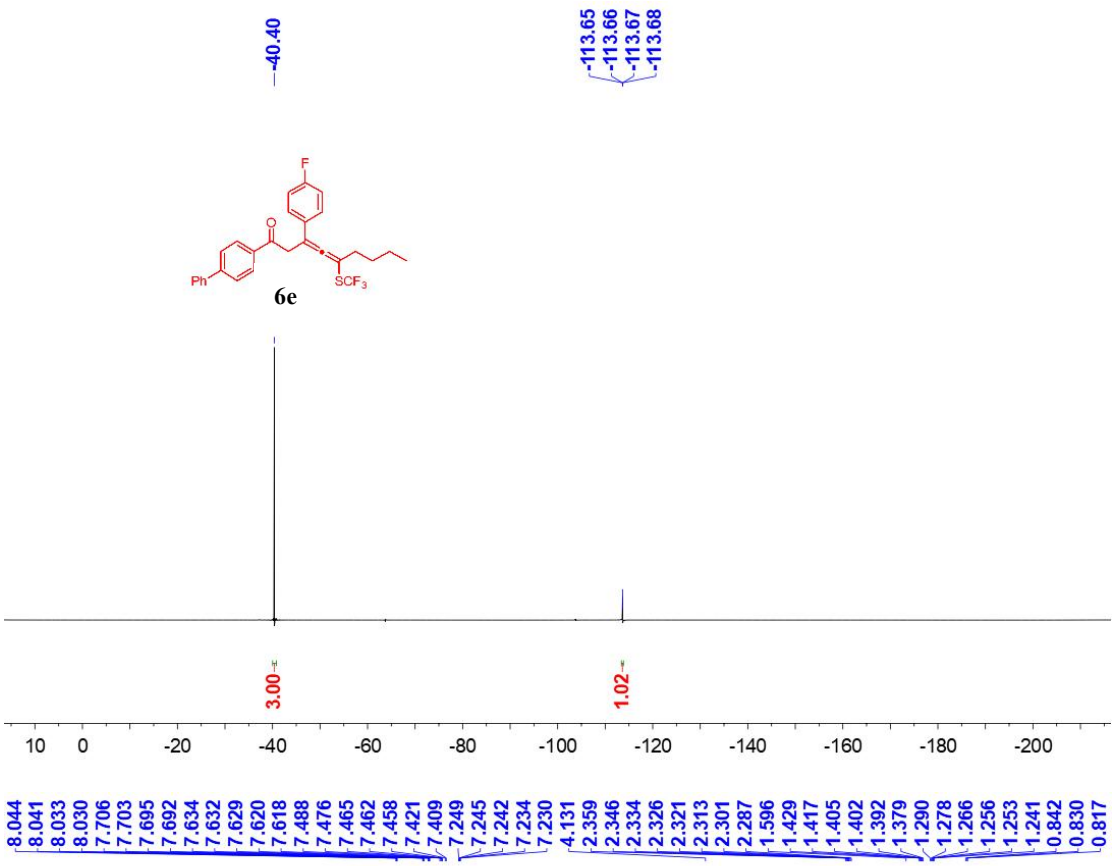
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1.02
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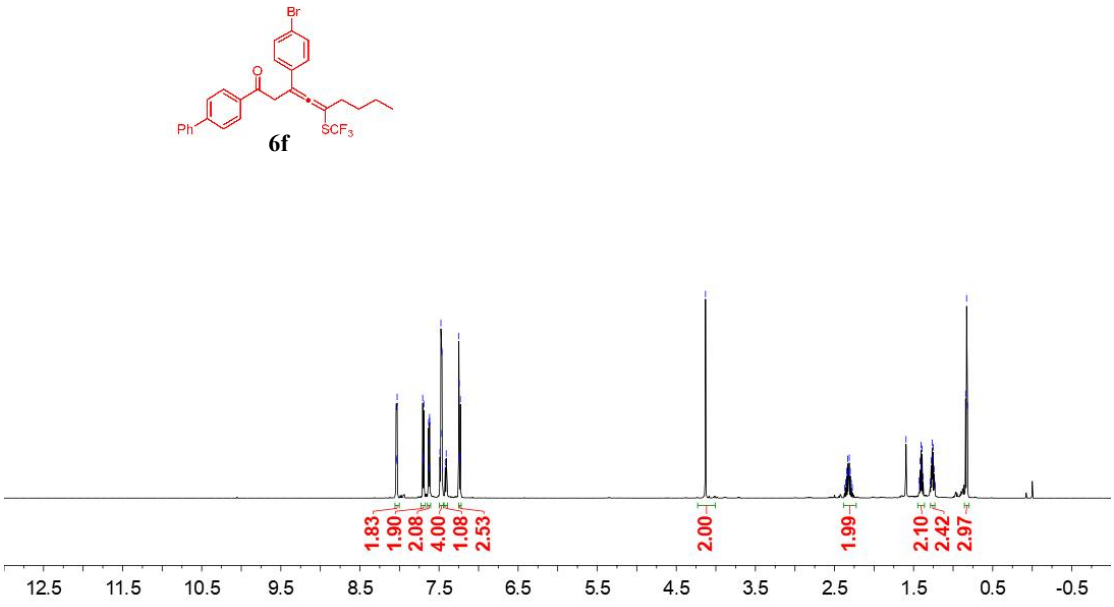
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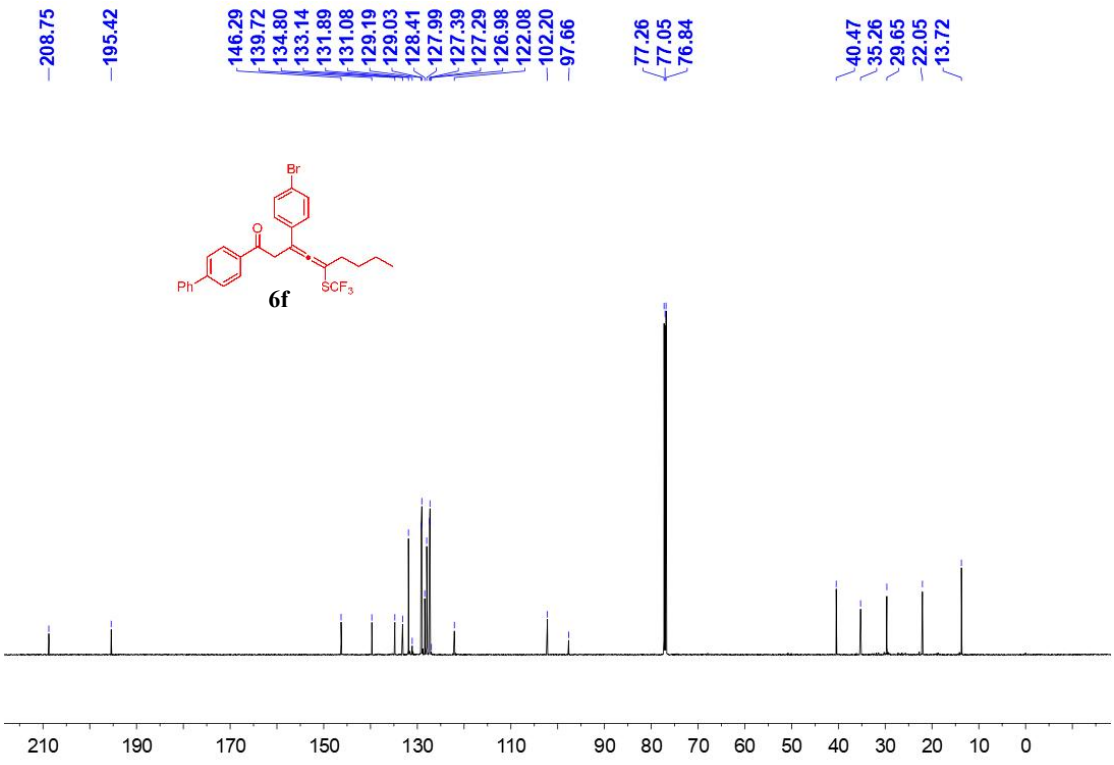
6f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29.65
22.05
13.72
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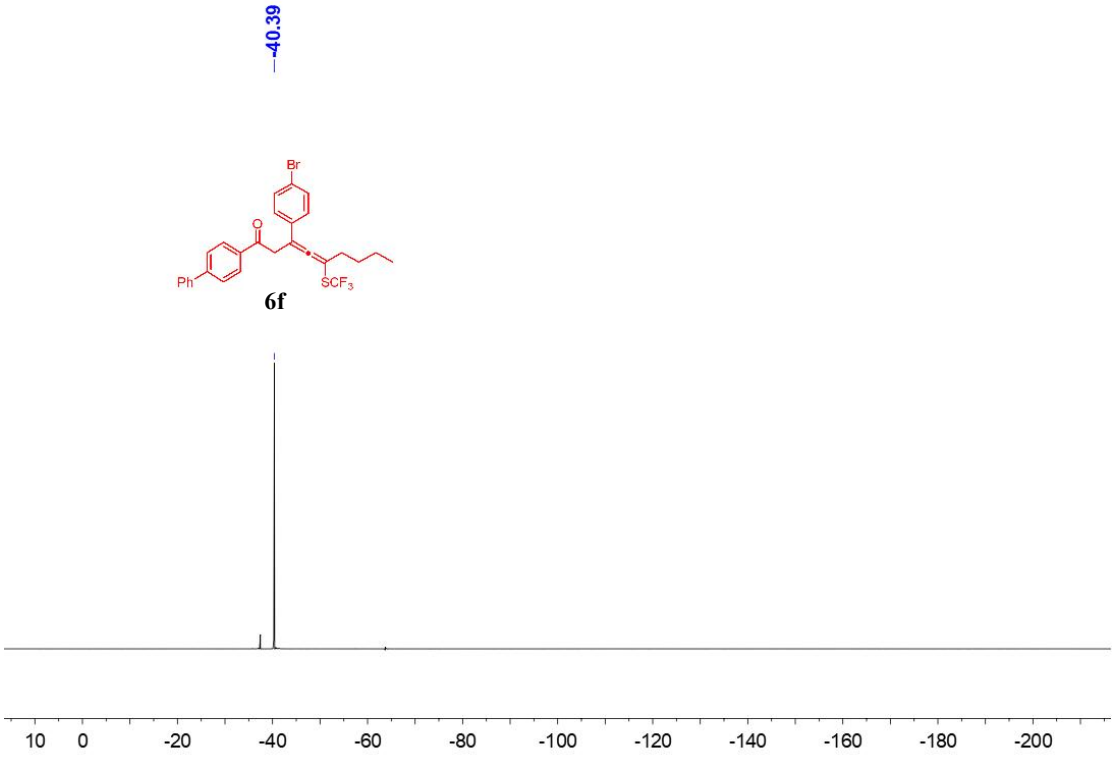
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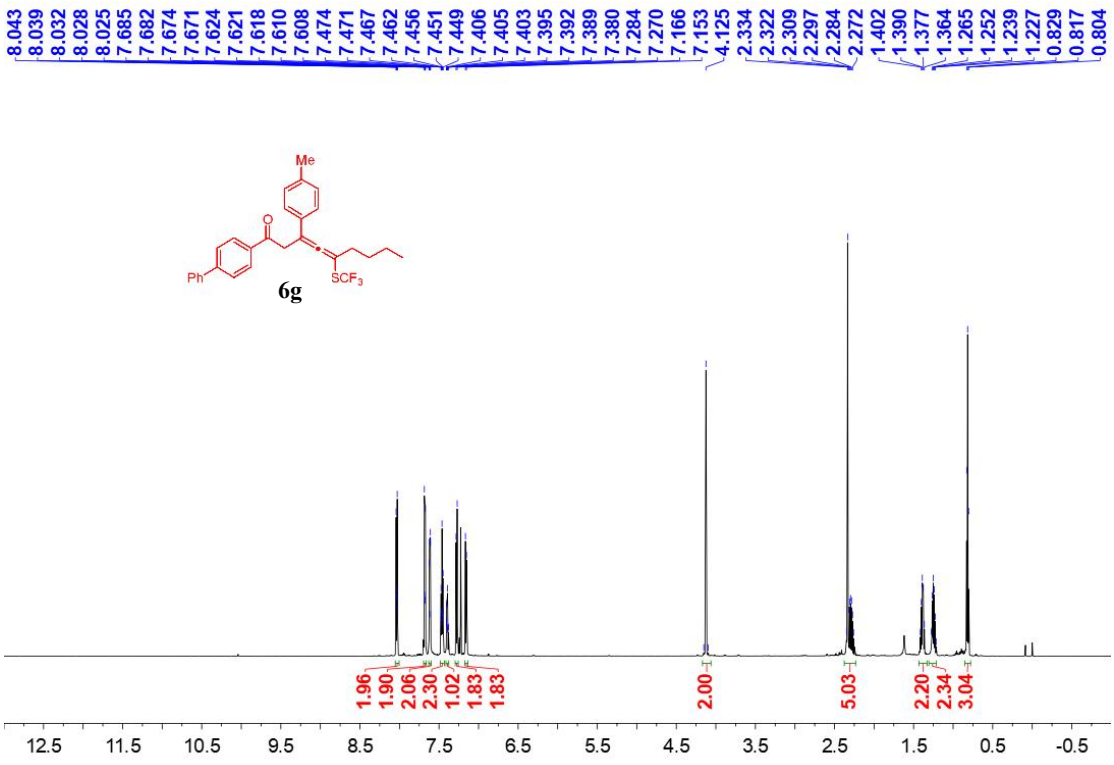
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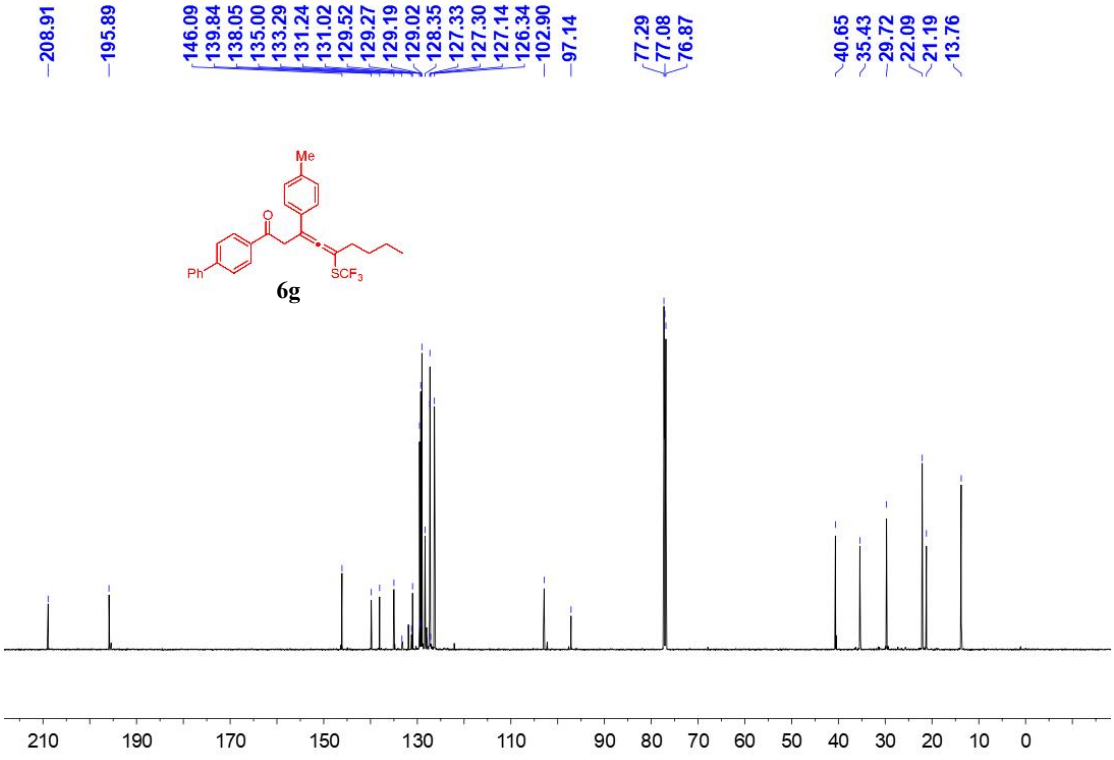
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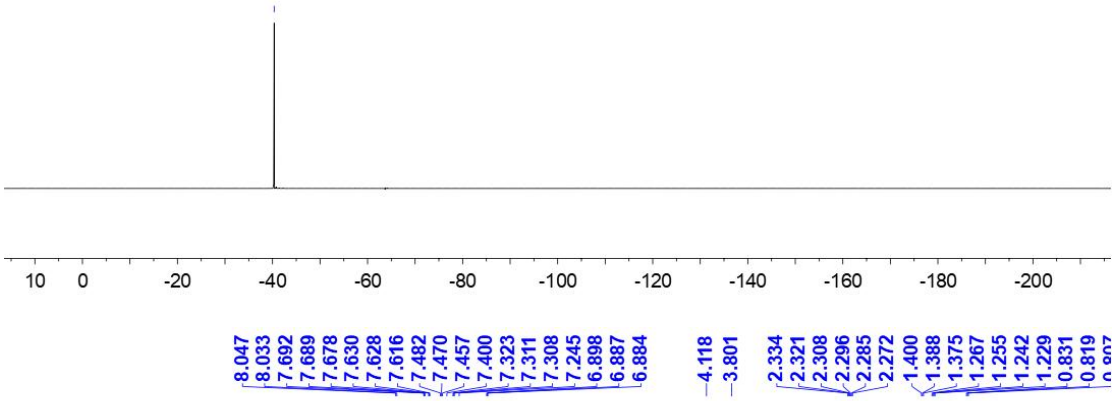
6g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0.819


O
O
Ph
SCF3

6h

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1.99
3.08
2.08
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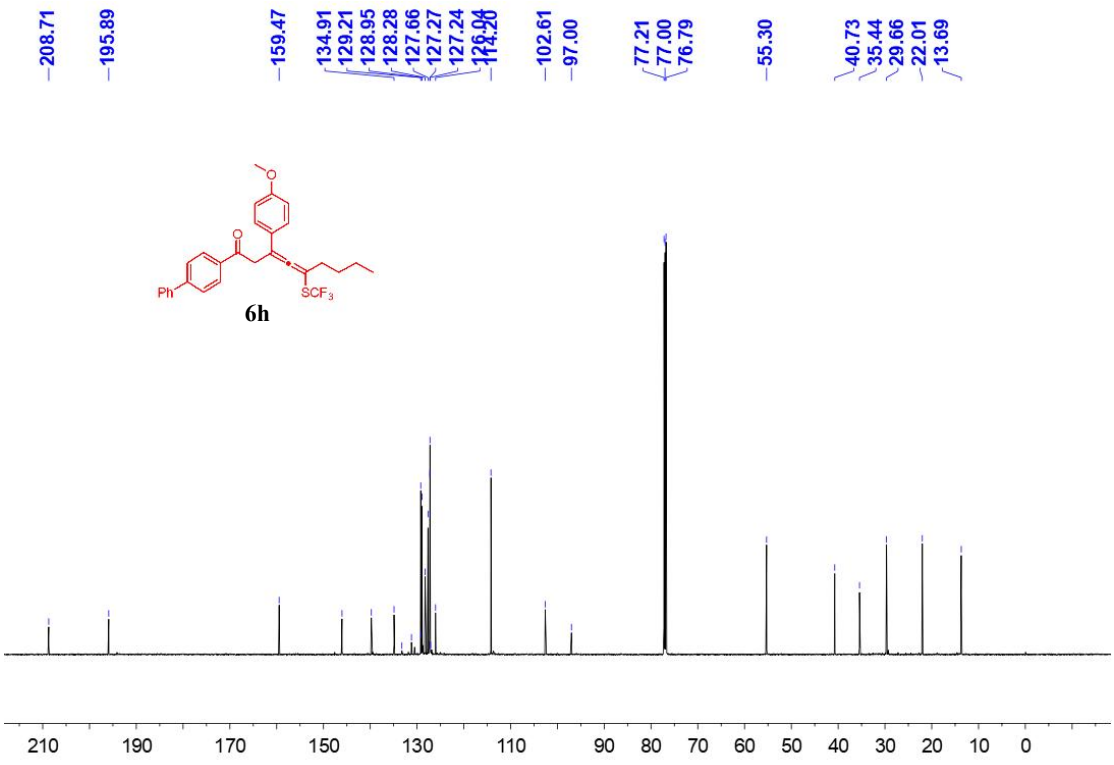
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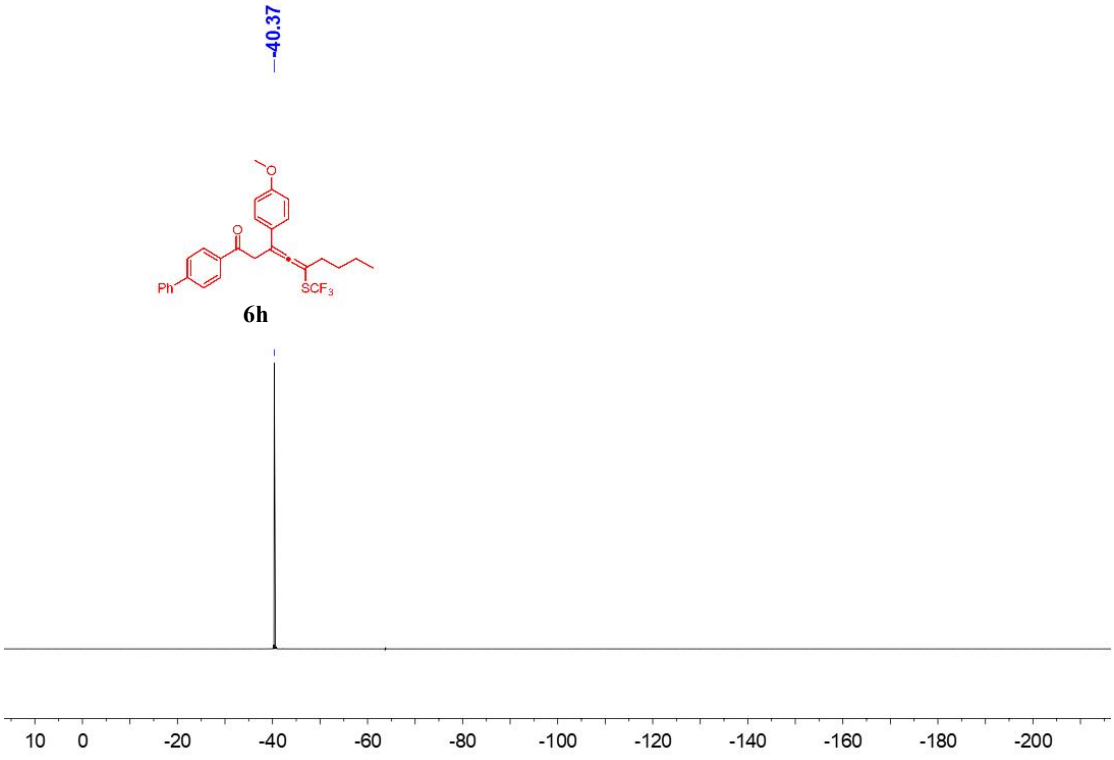
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6h


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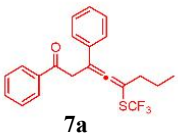
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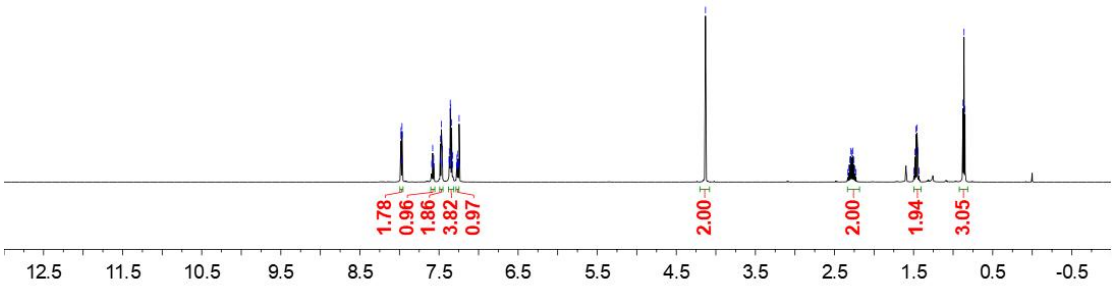
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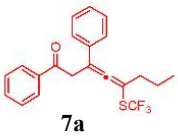
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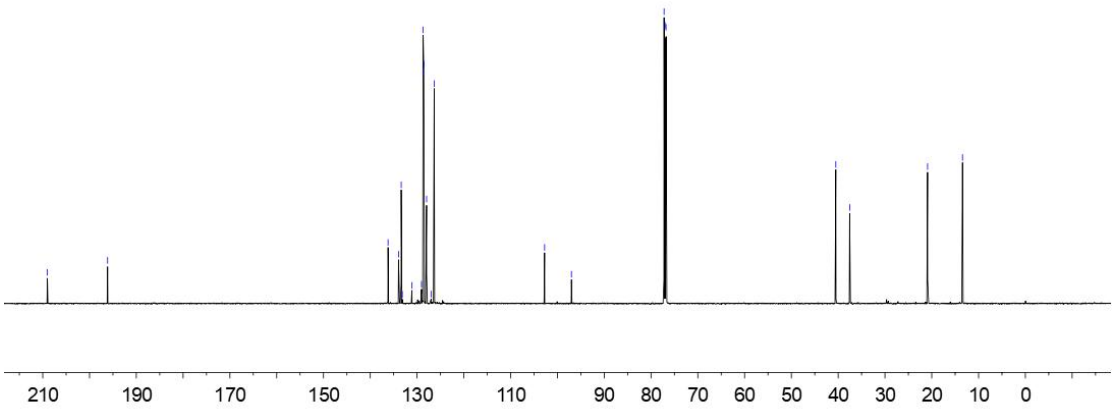
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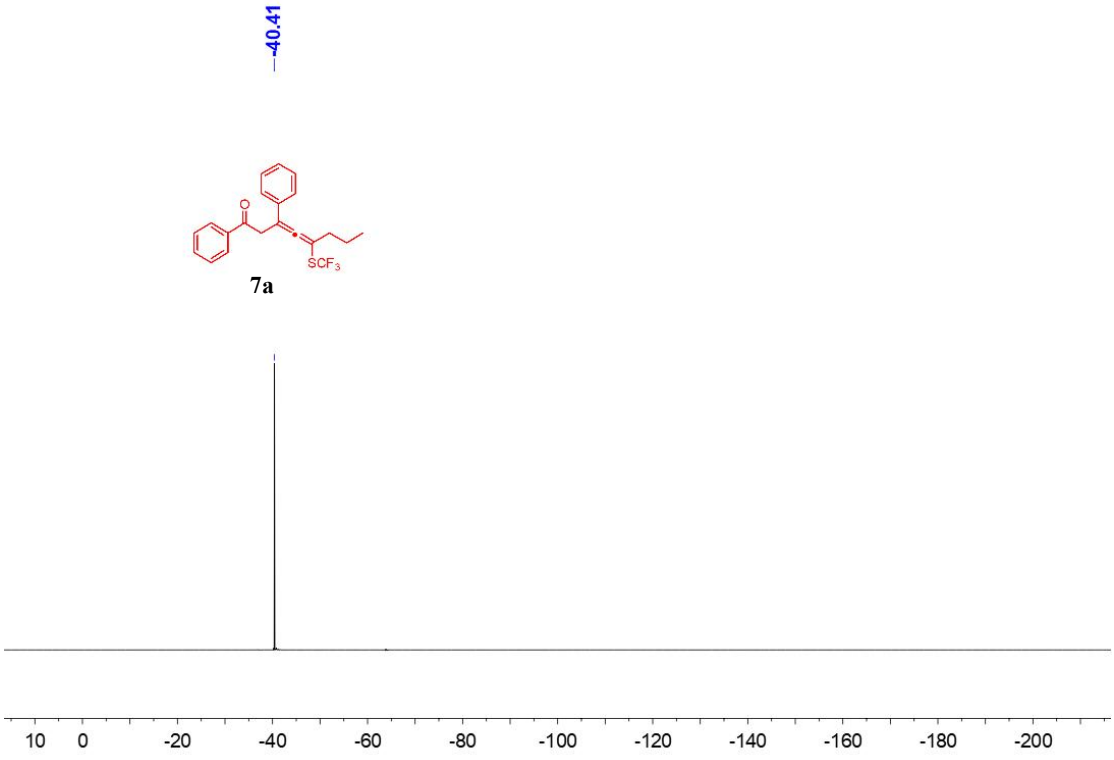
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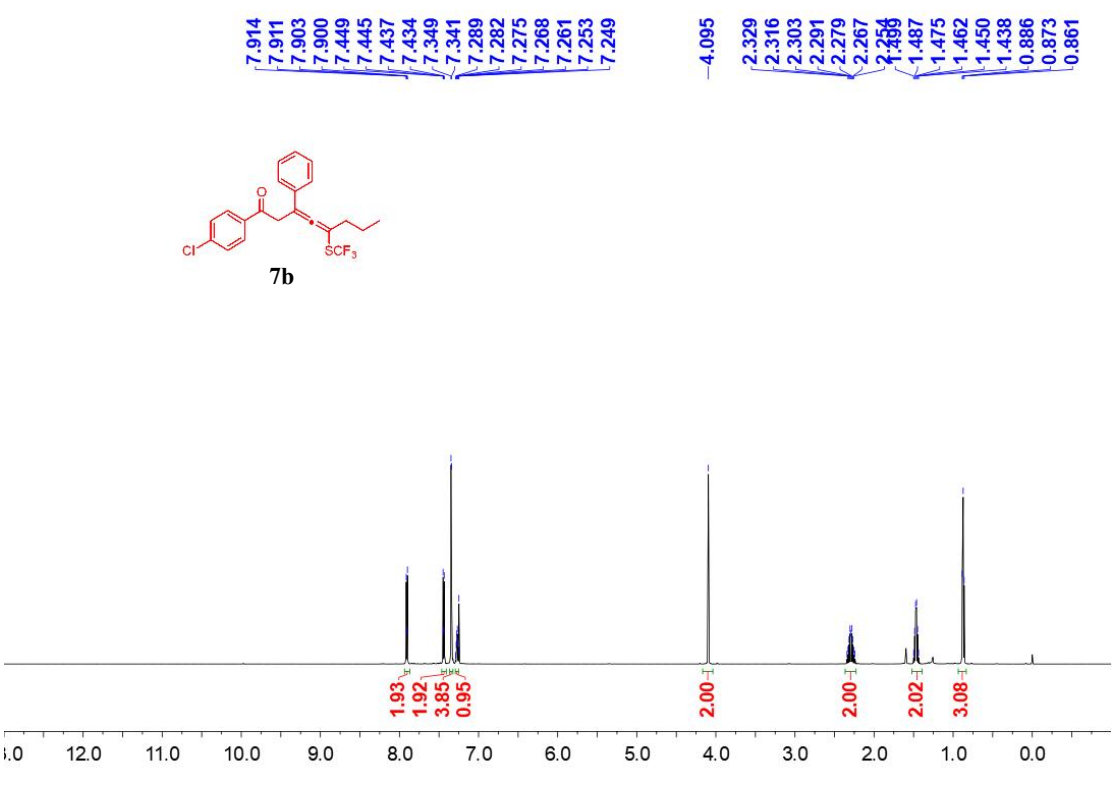
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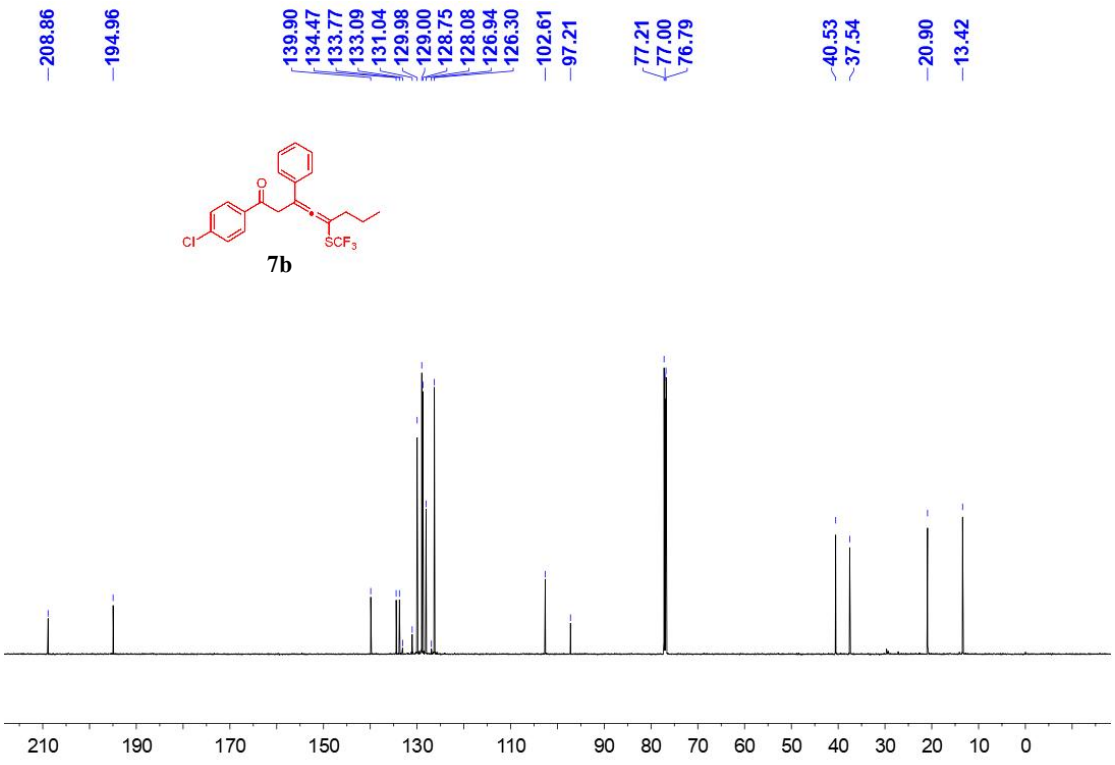
7b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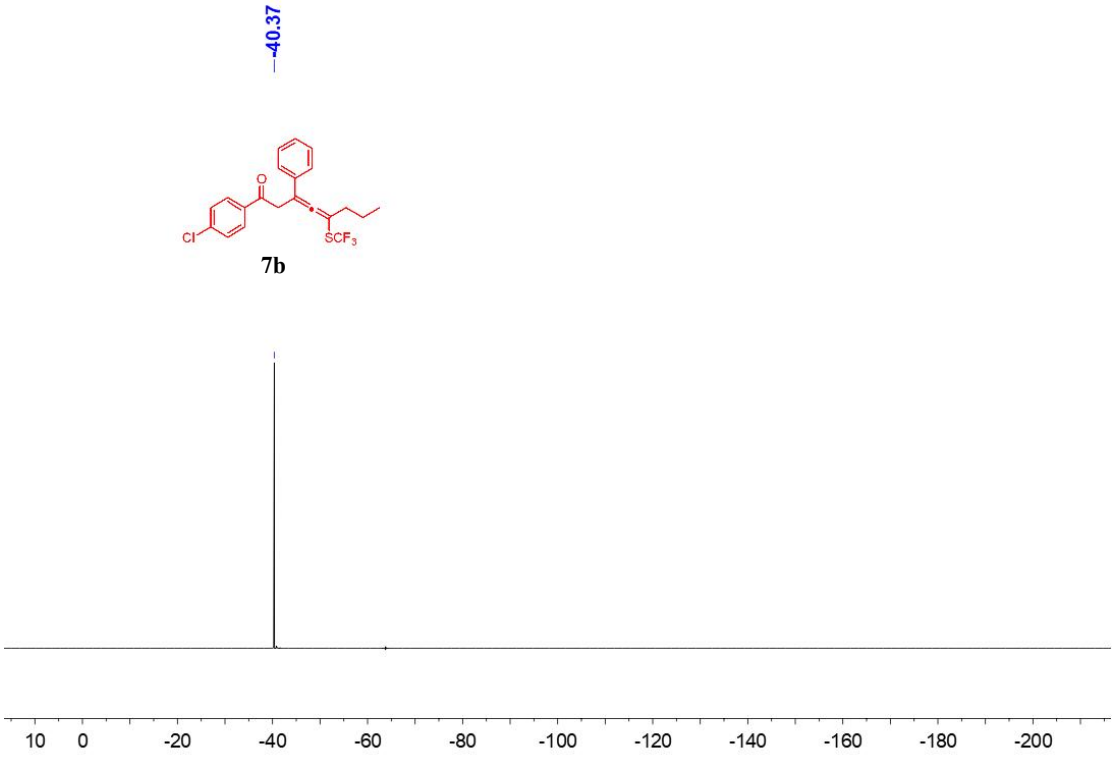
7b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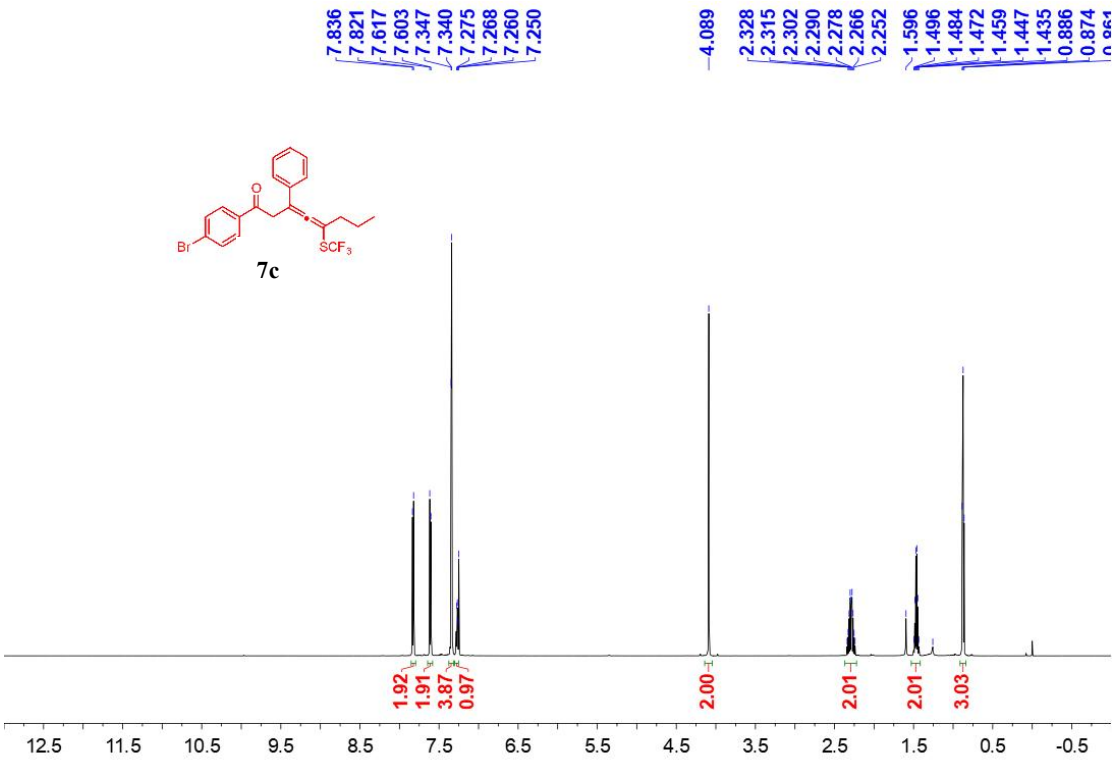
7b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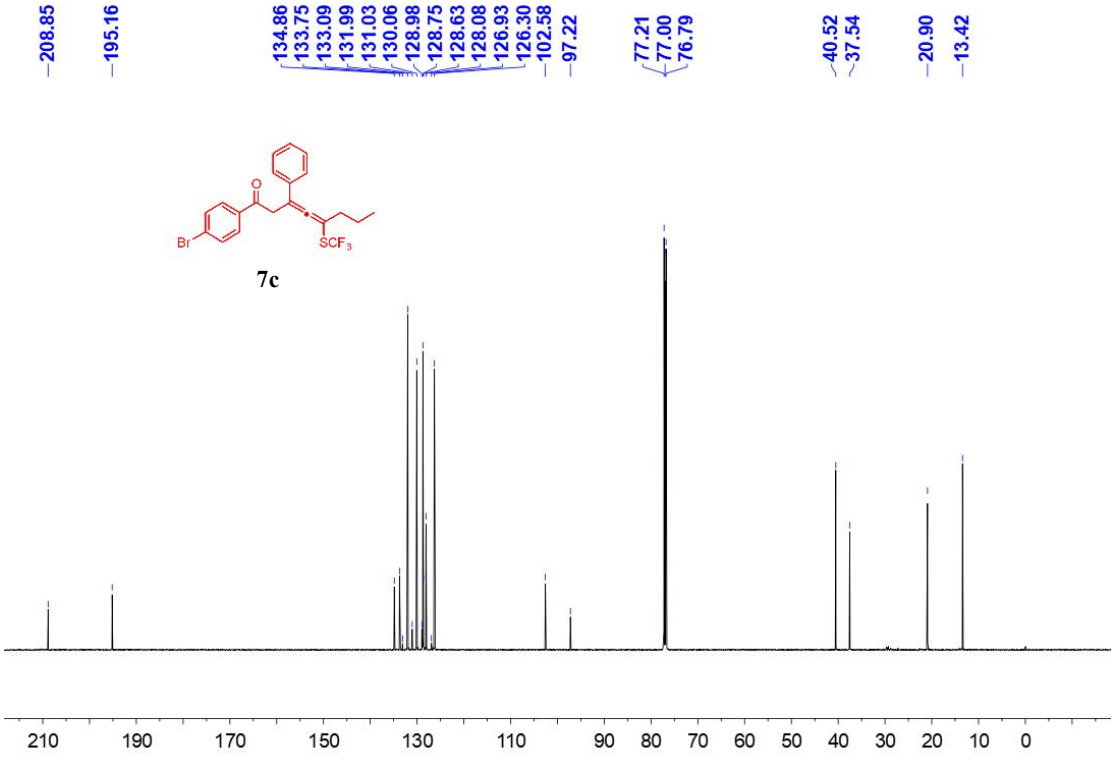
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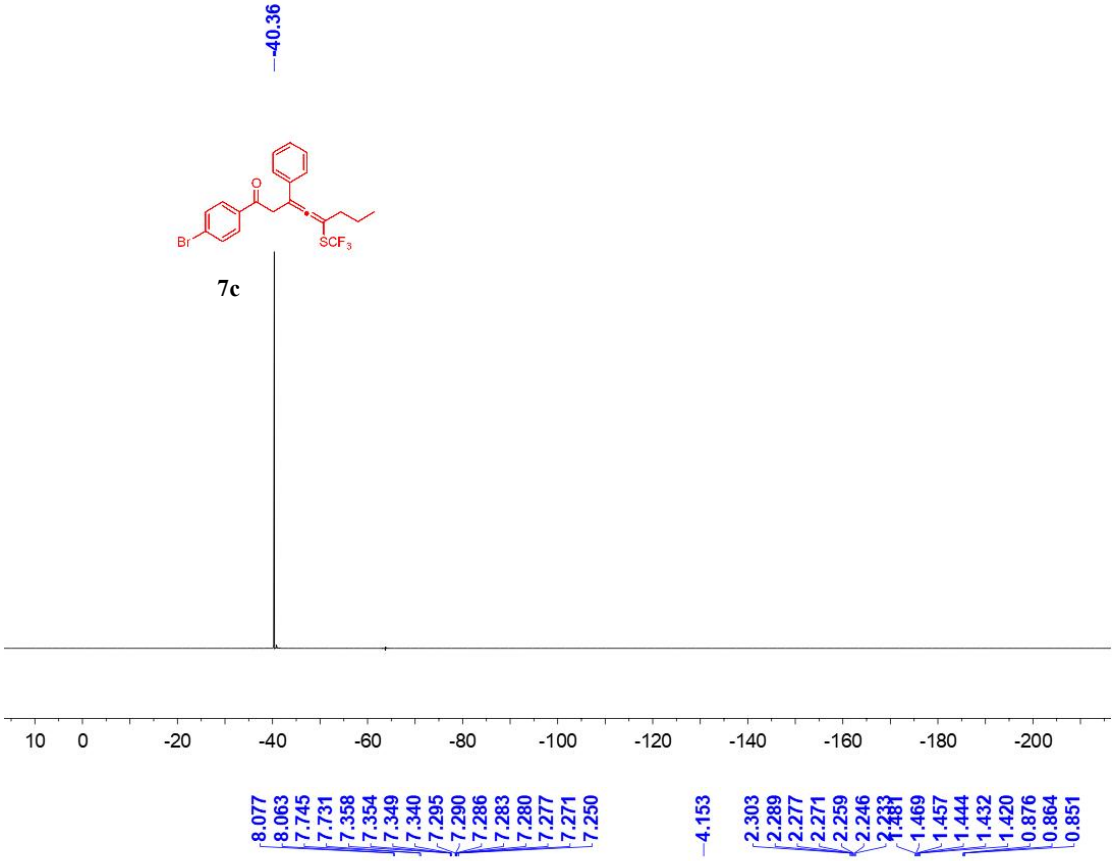
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8.077 8.063 7.745 7.731 7.358 7.354 7.349 7.340 7.295 7.290 7.286 7.283 7.280 7.277 7.271 7.250
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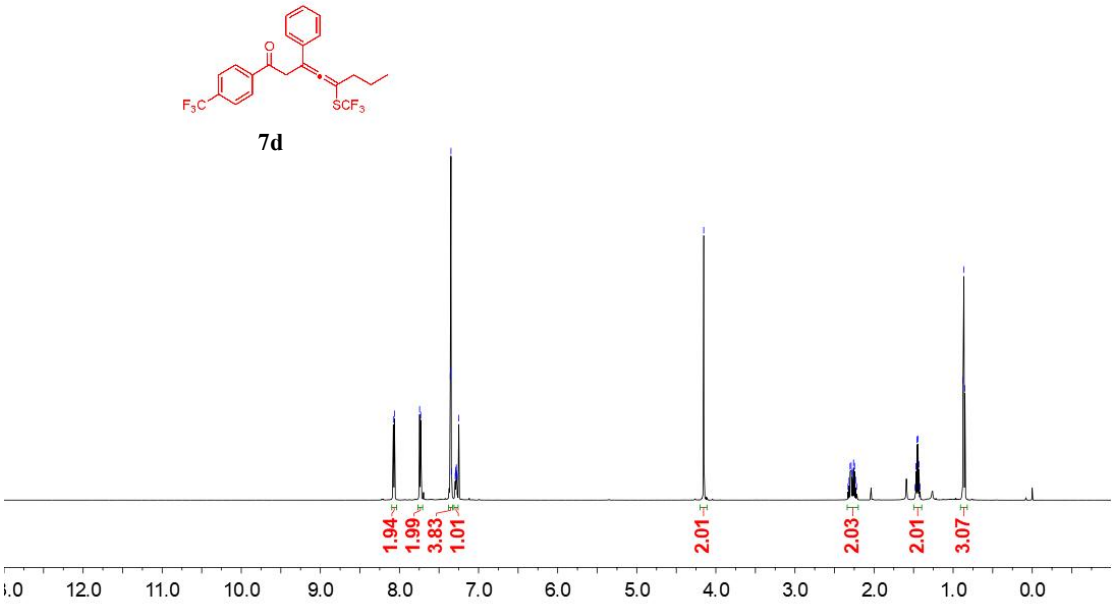
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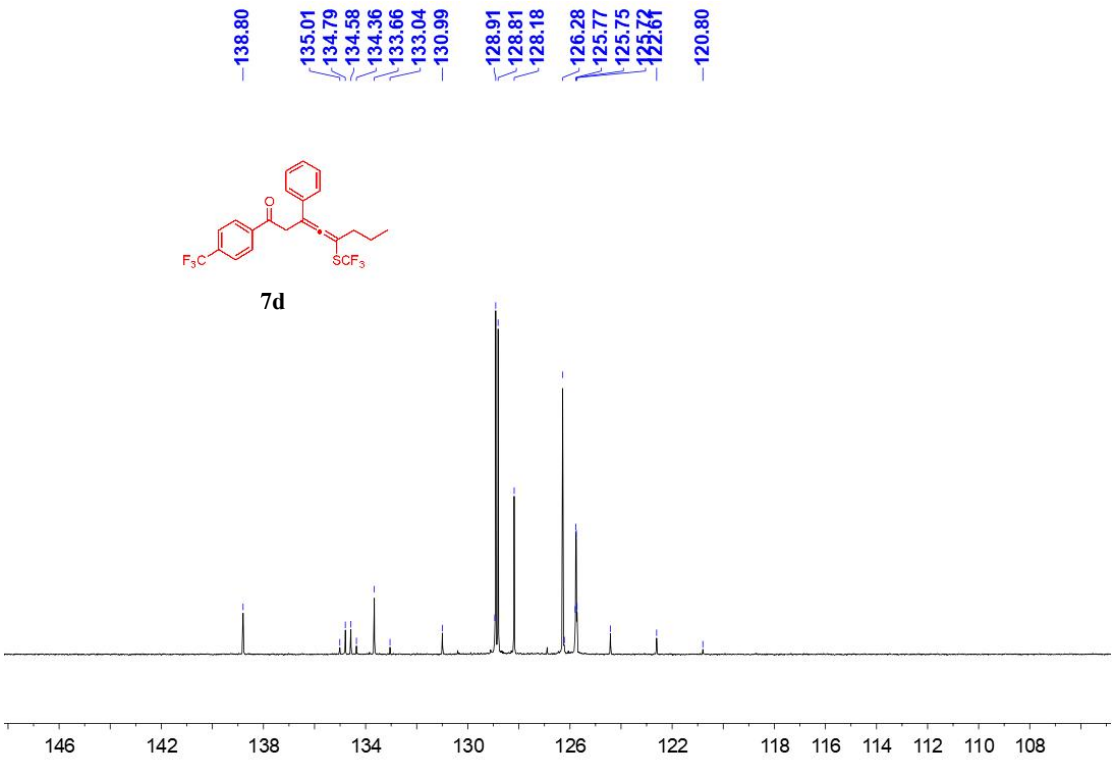
7d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SCF3
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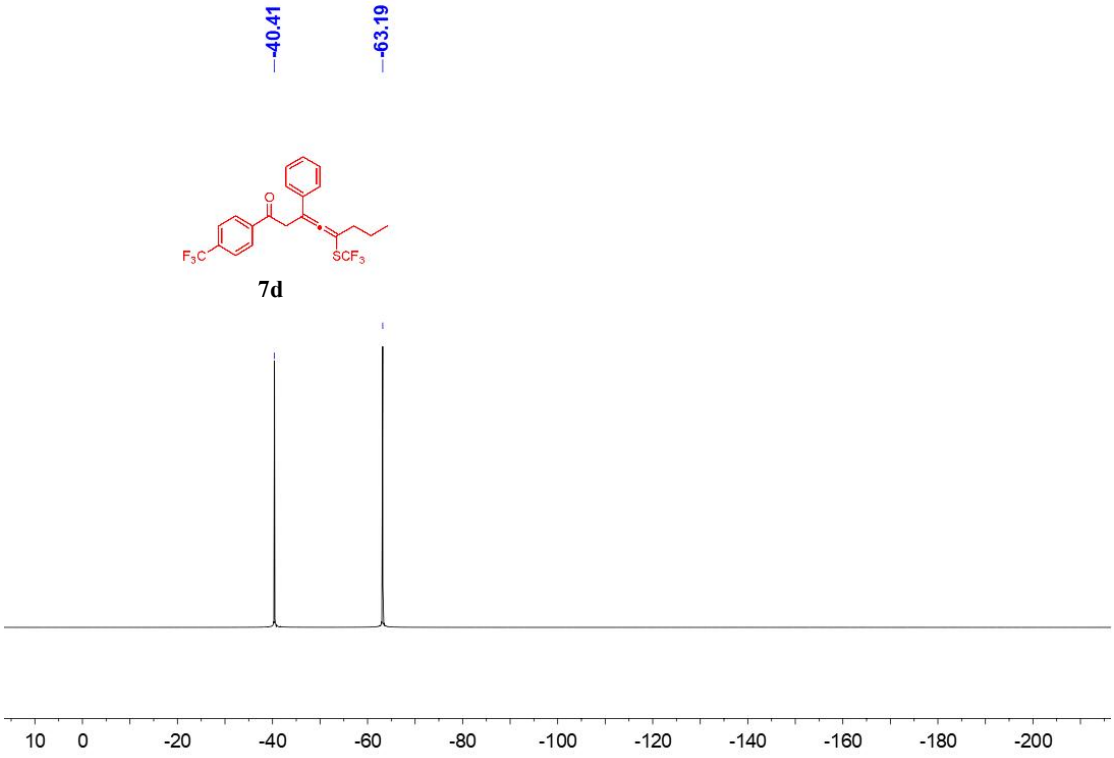
7d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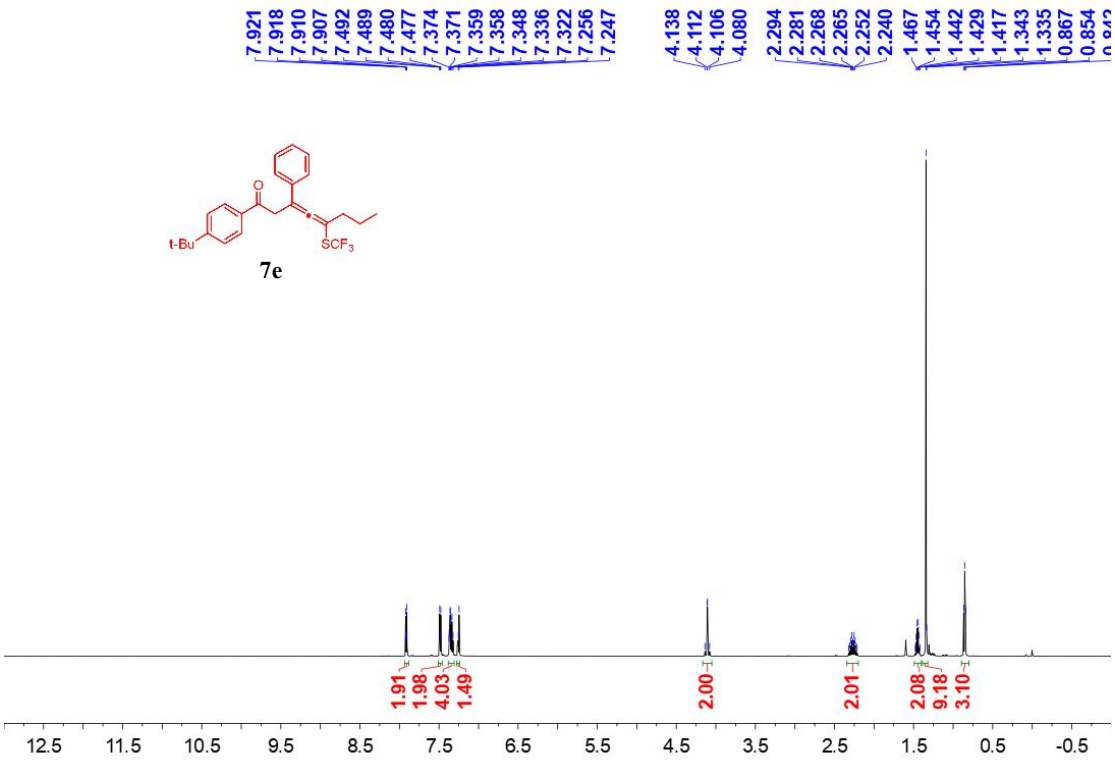
-200

7d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SCF3
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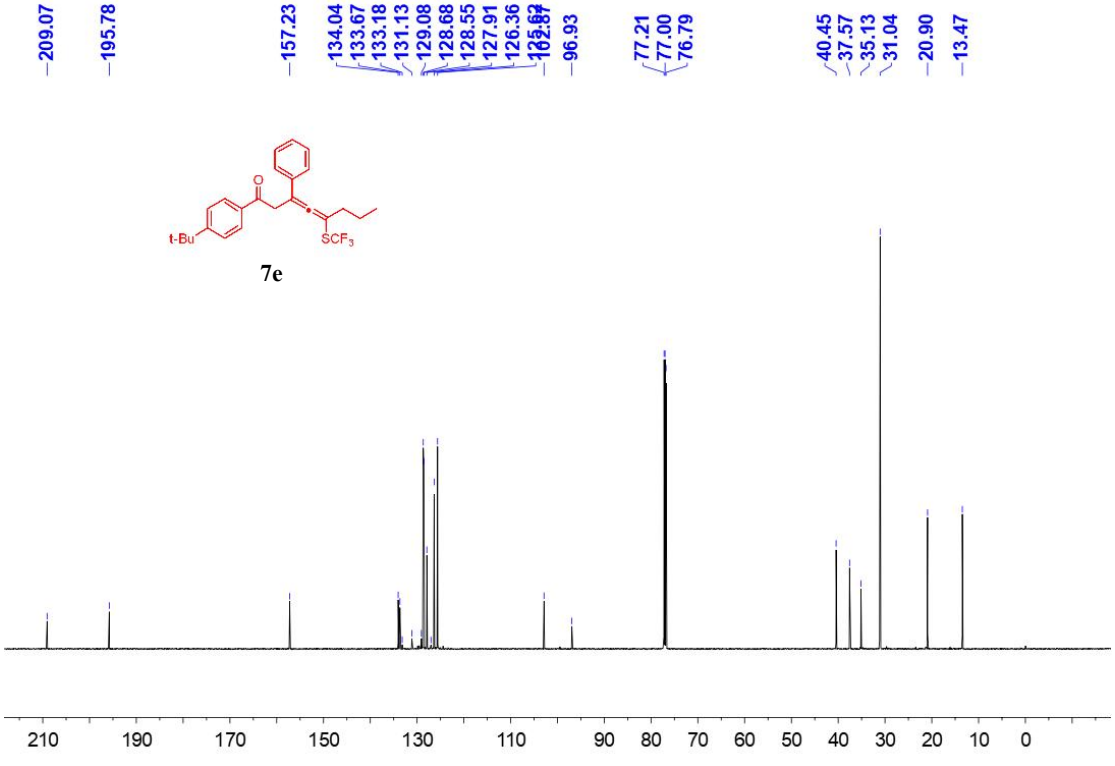
7e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20.90
13.47
t-Bu
SCF3
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20
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7e


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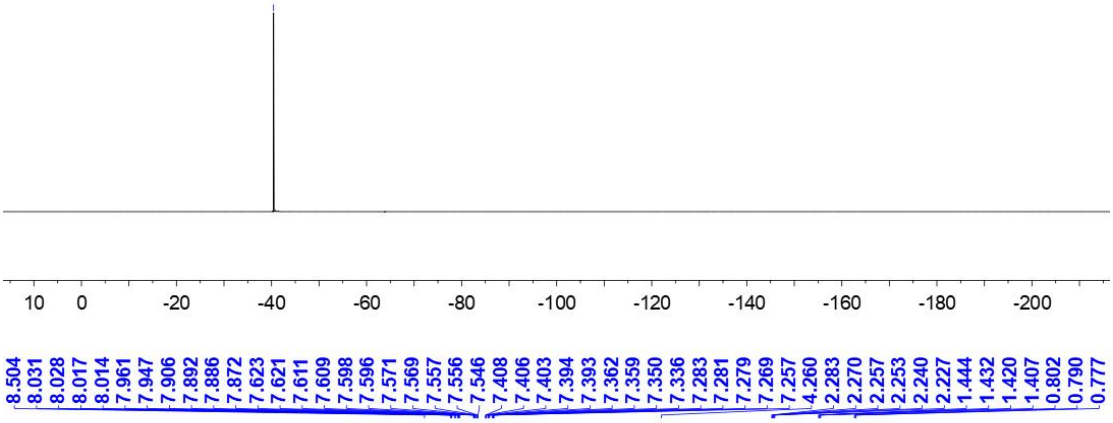
SCF3
O

7e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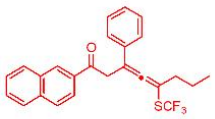
SCF3
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7f

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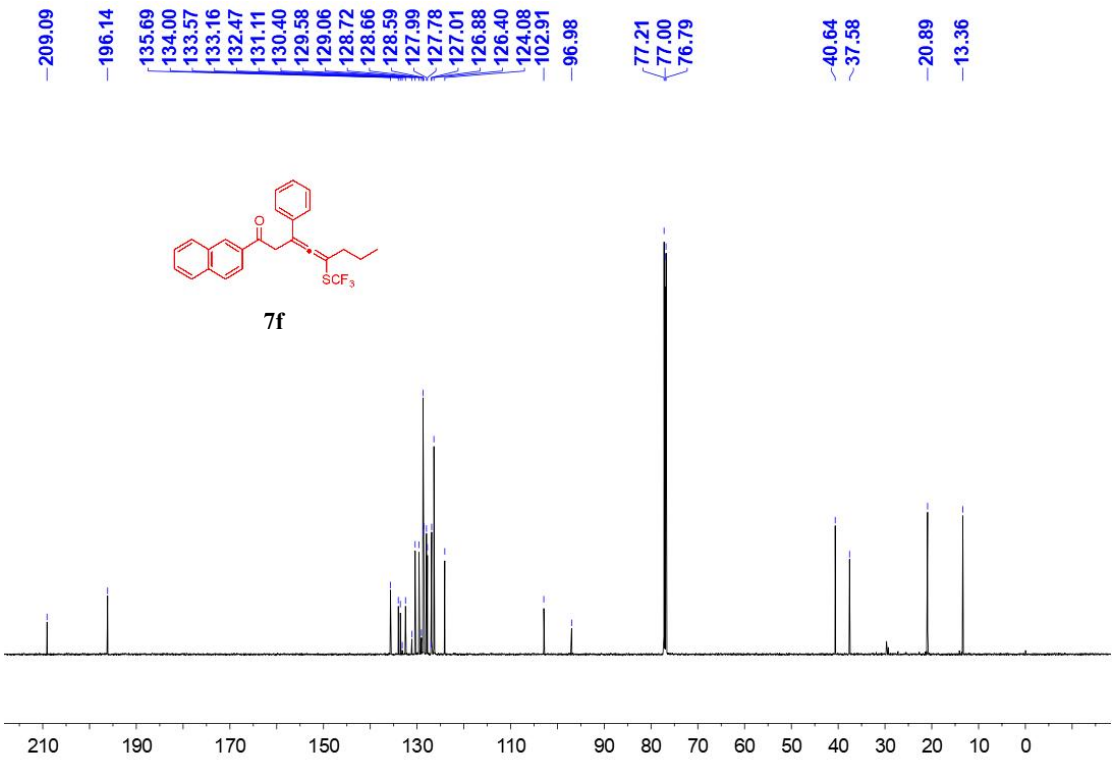
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126.40
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76.79
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37.58
20.89
13.36
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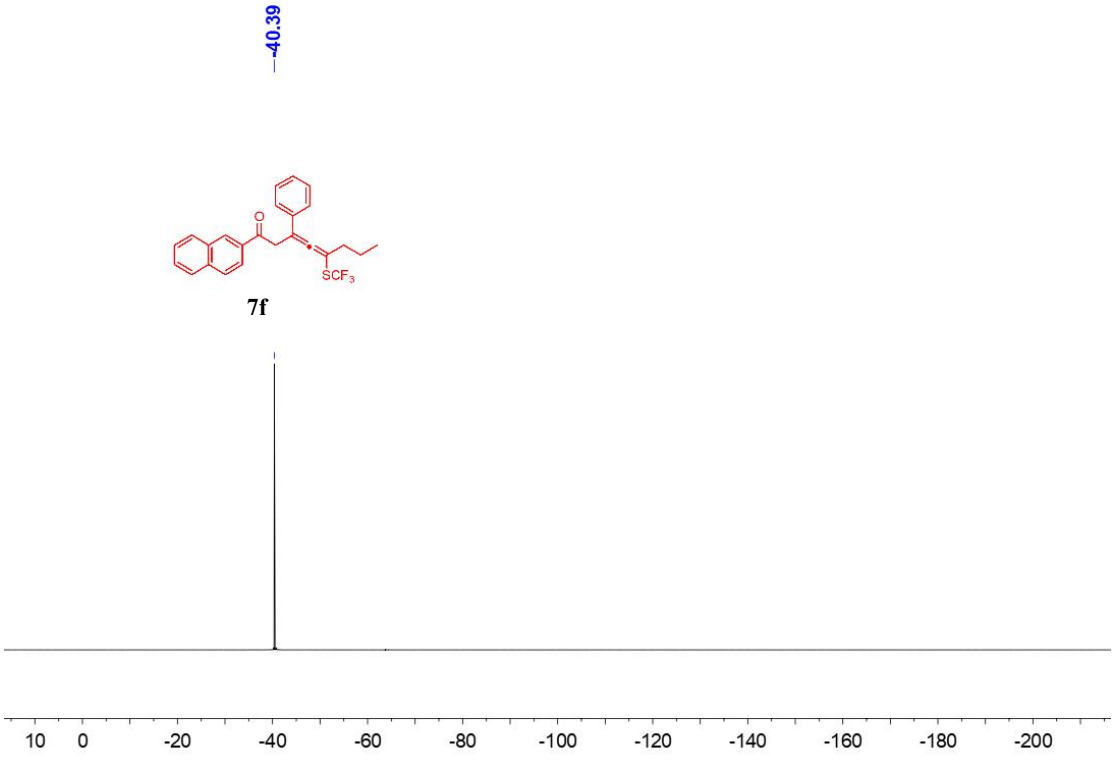
0

7f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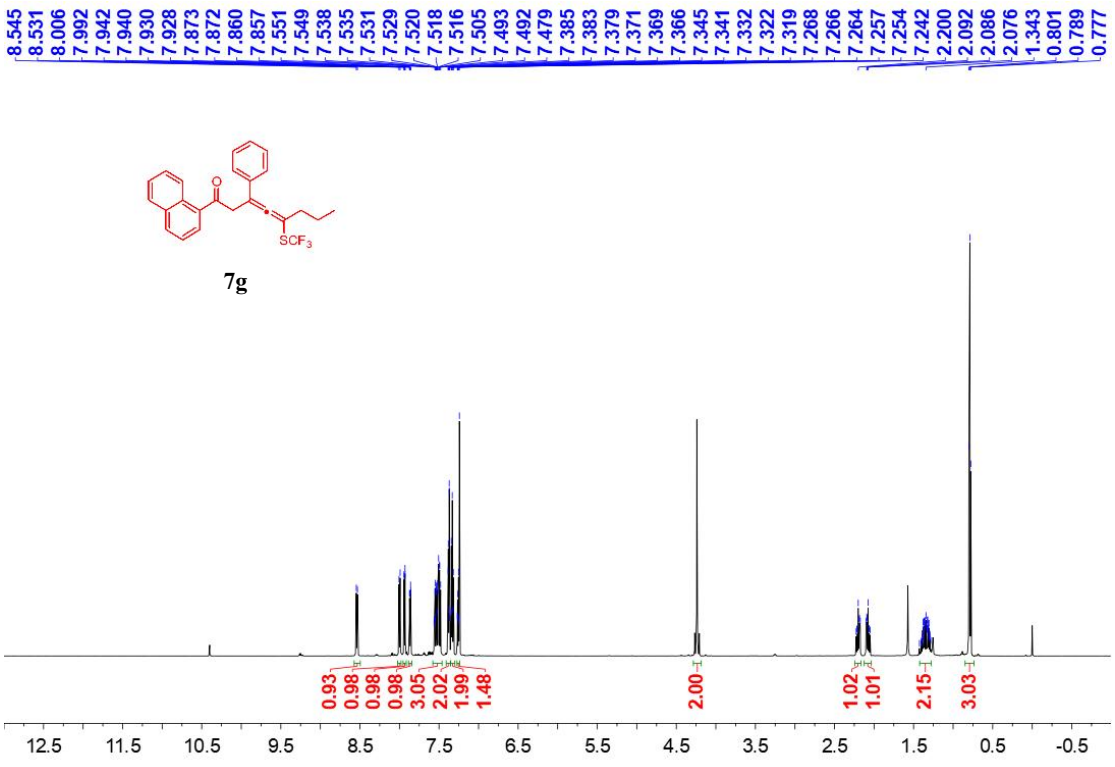
7f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SCF3
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1.01
2.15
3.03
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11.5
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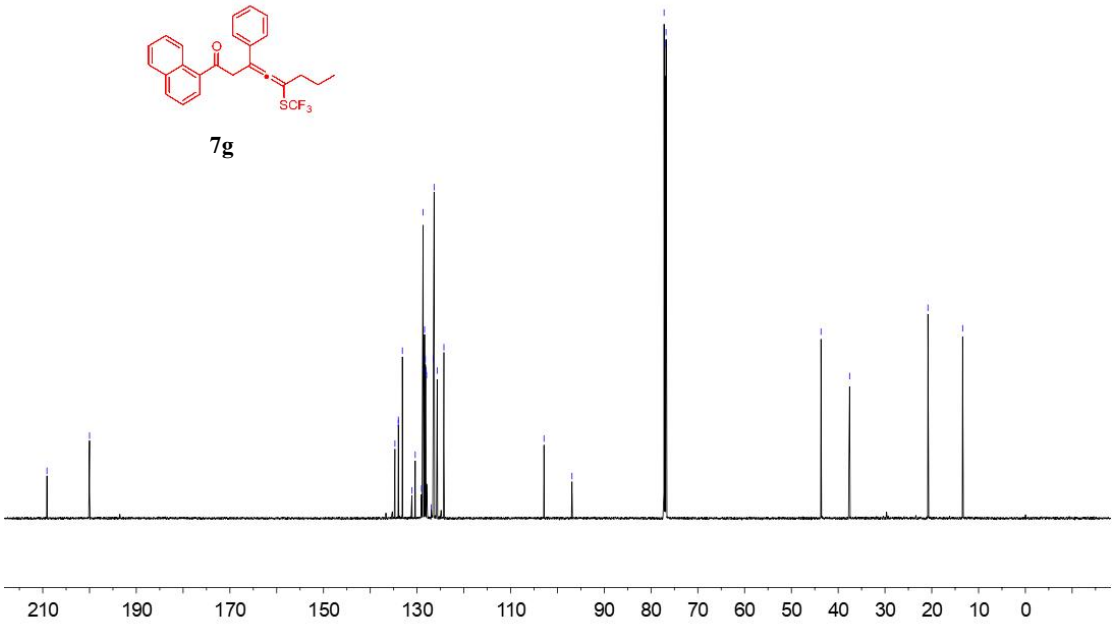
7g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124.26
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76.79
43.67
37.57
20.82
13.40
SCF3
210
190
170
150
130
110
90
80
70
60
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7g


-40.45
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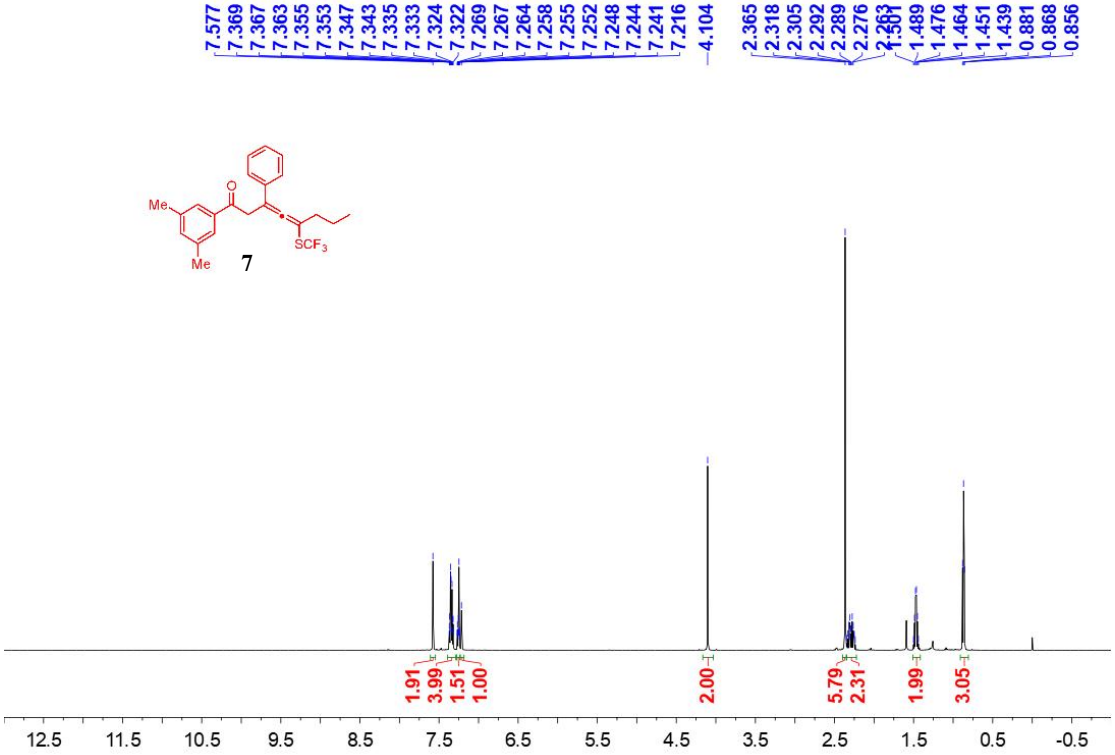
7g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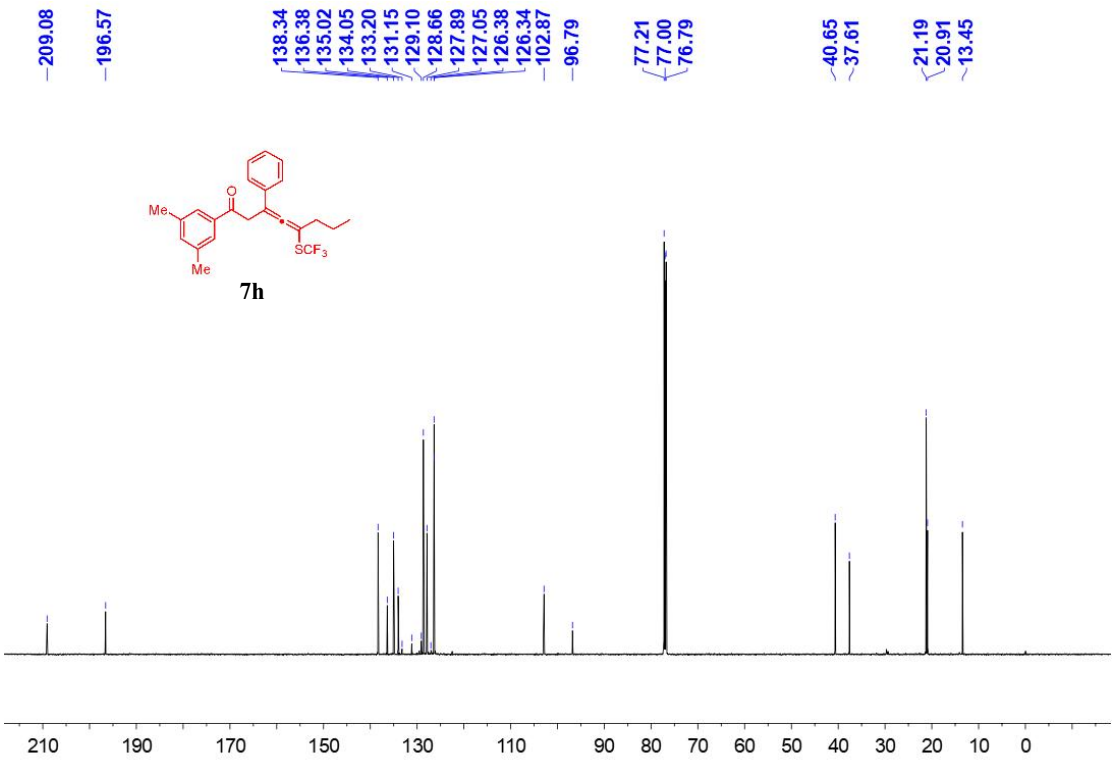
Me
Me
SCF3
1.91 3.99 1.51 1.00 2.00 5.79 2.31 1.99 3.05
12.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

7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20.91
13.45
Me
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60
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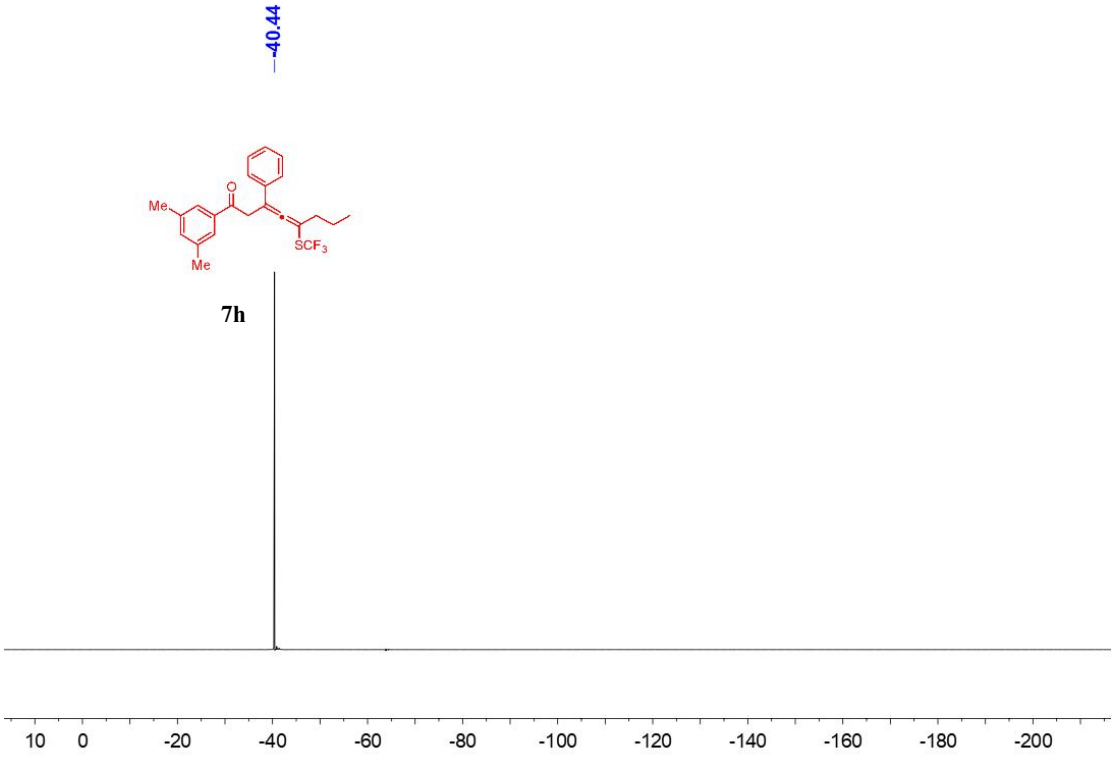
0

7h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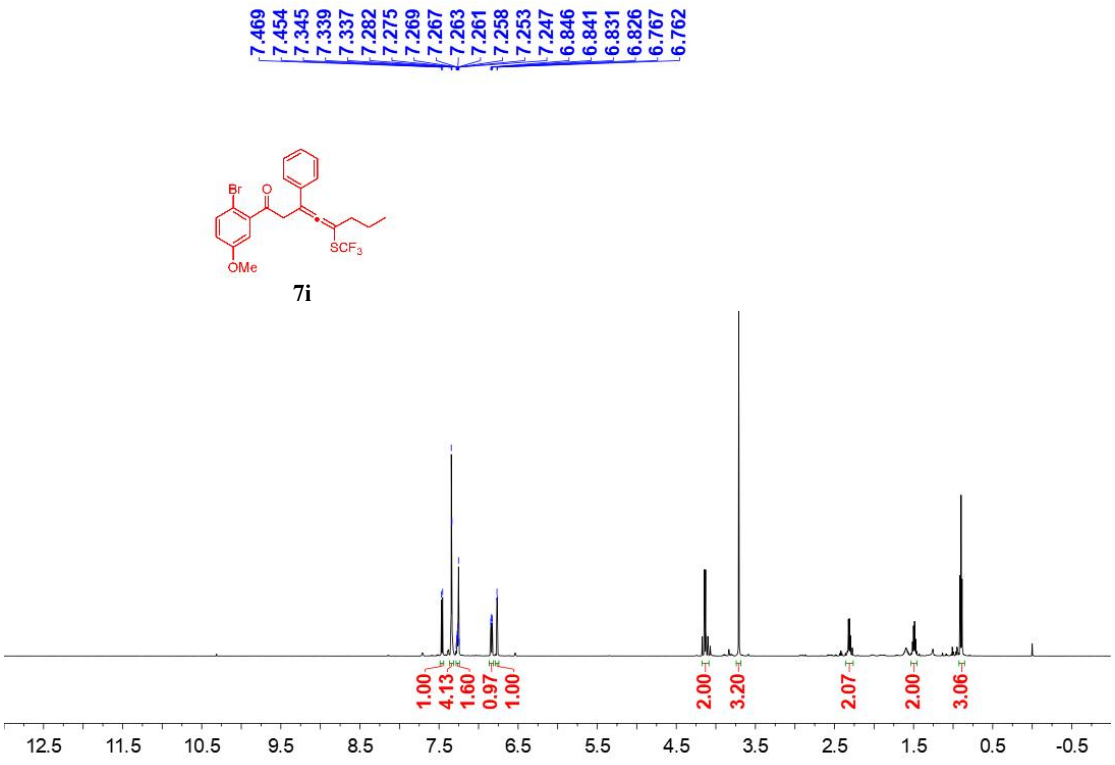
7h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7.247
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6.841
6.831
6.826
6.767
6.762
Br
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SCF3
OMe
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1.60
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1.00
2.00
3.20
2.07
2.00
3.06
12.5
11.5
10.5
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2.5
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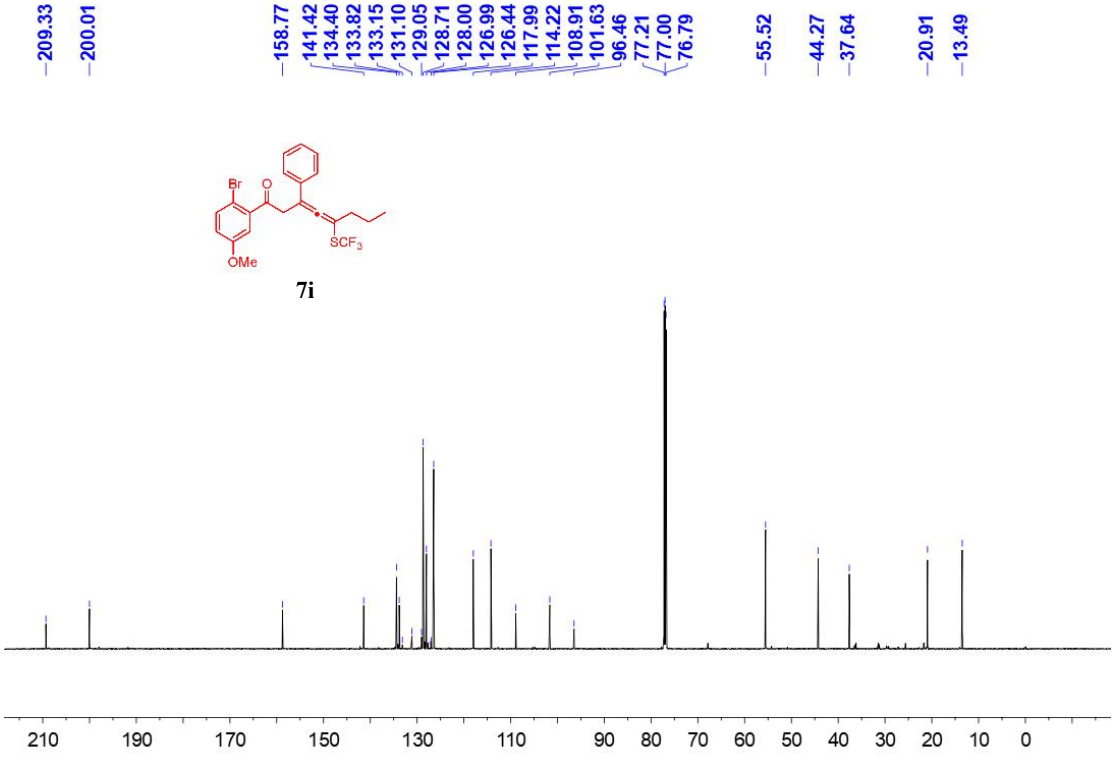
7i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101.63
96.46
77.21
77.00
76.79
55.52
44.27
37.64
20.91
13.49
Br
O
SCF3
OMe
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150
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110
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80
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60
50
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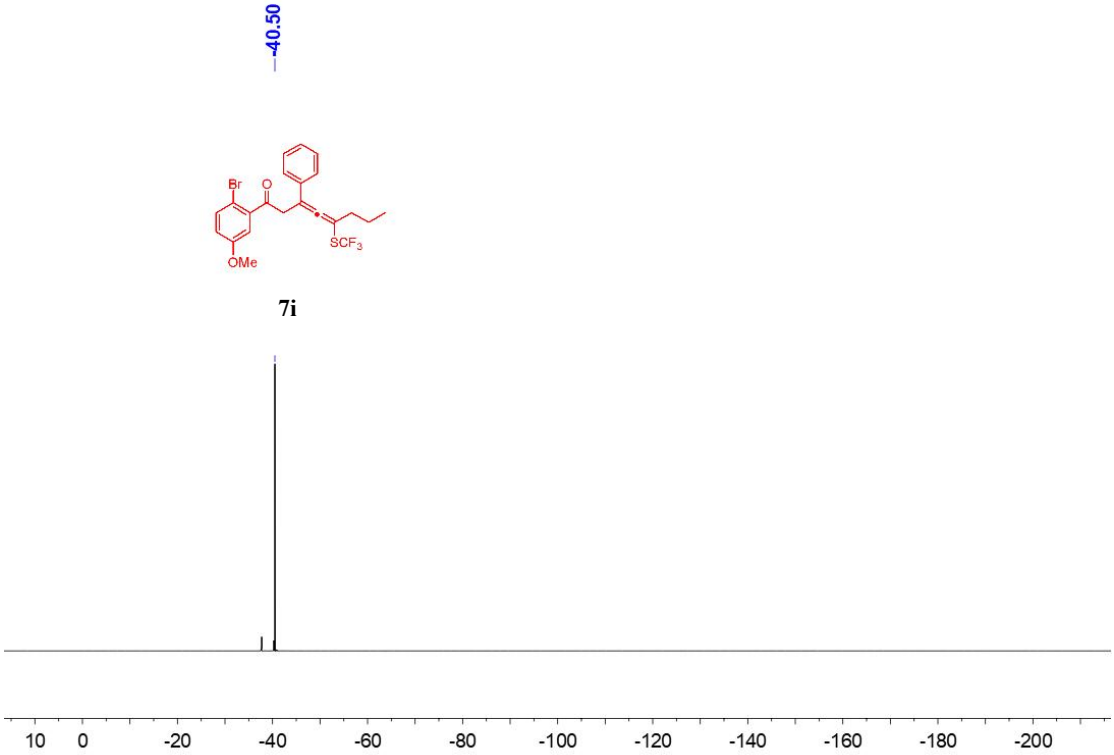
20
10
0

7i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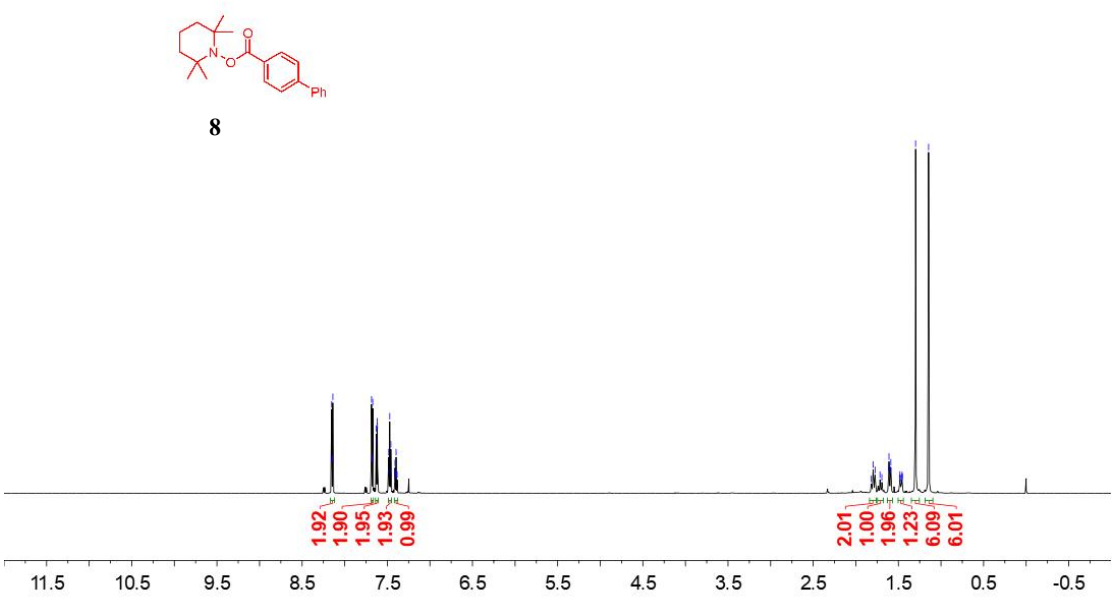
7i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11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

8


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20.71
16.84

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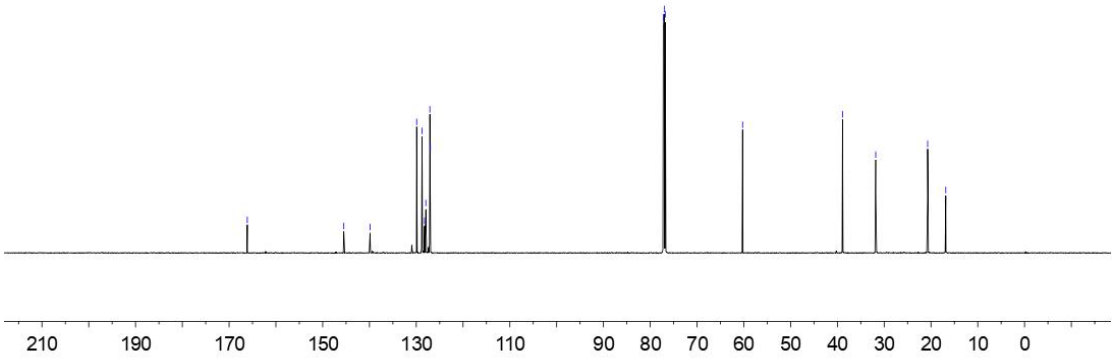
O
O
Ph

8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190
170
150
130
110
90
80
70
60
50
40
30
20
10
0


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7.100
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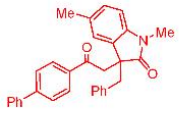
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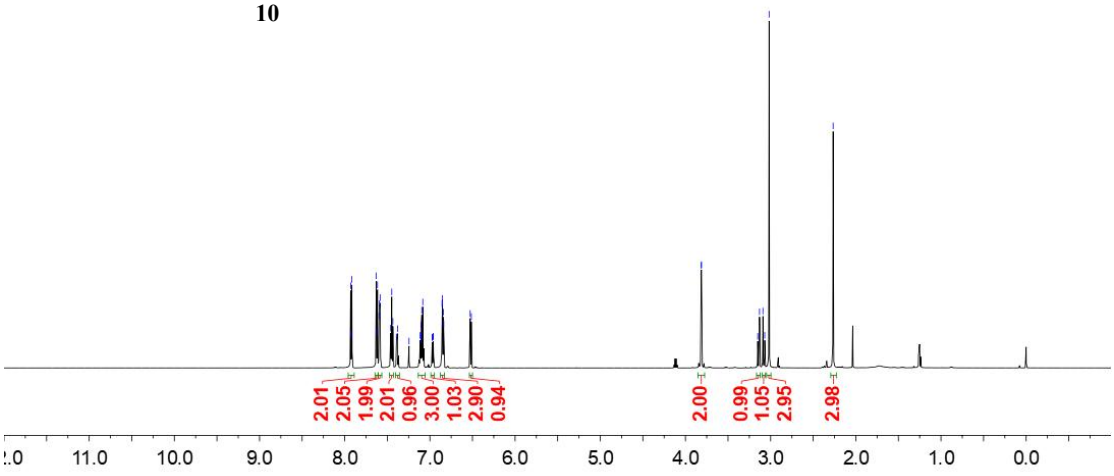
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2.95
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4.0
3.0
2.0
1.0
0.0


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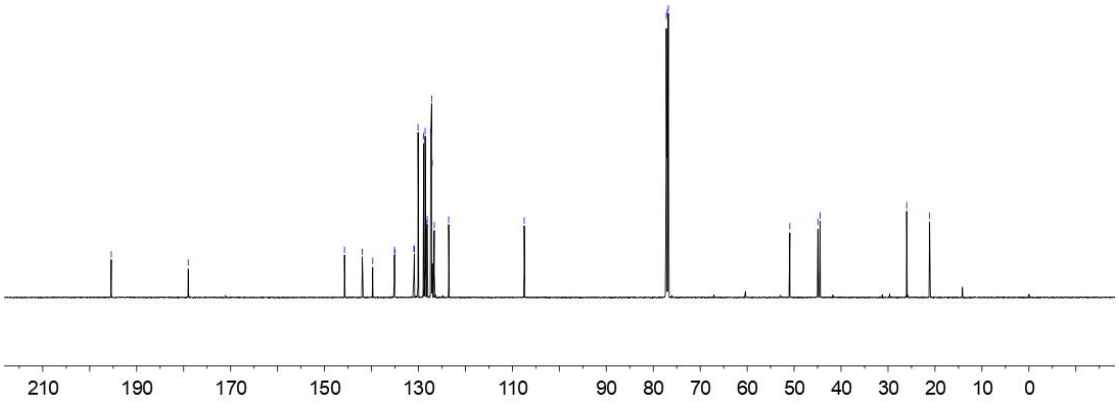
O
O
Ph
Ph

10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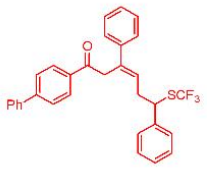
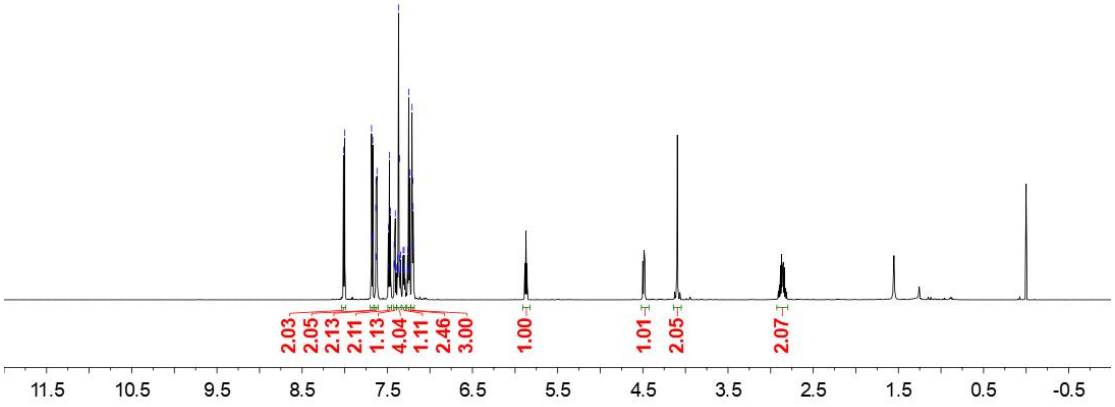

O
Ph
SCF3

(*E*)-12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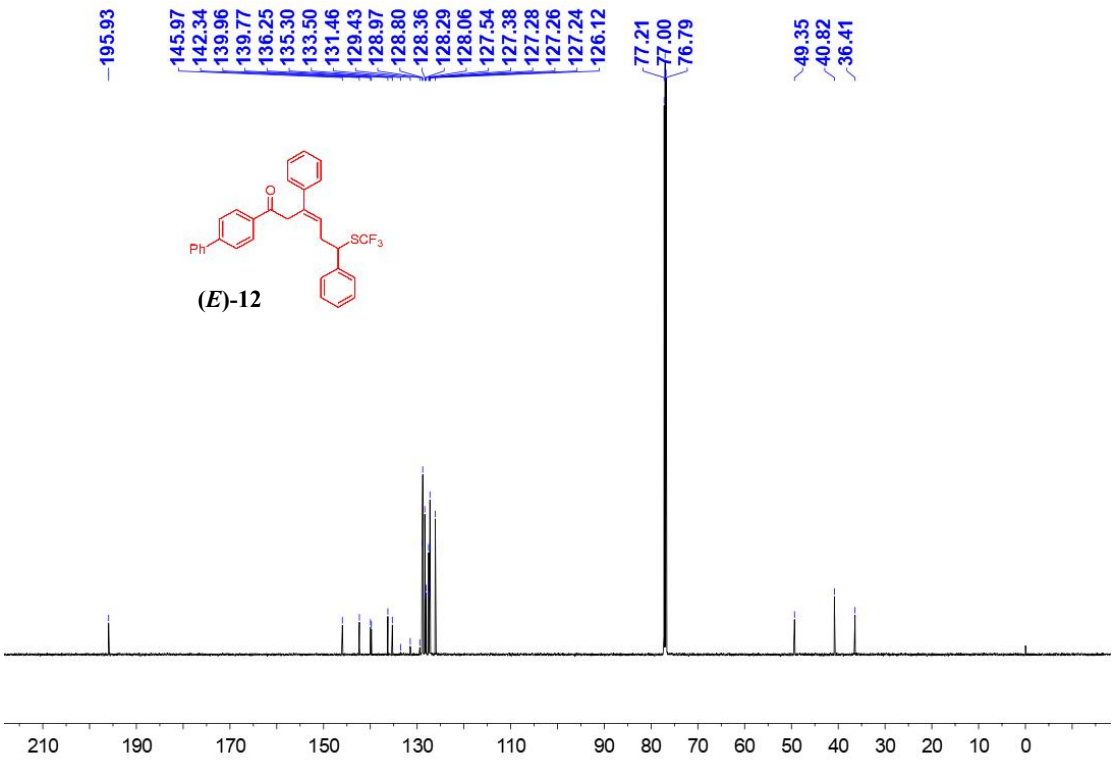
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77.00
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40.82
36.41
Ph
SCF3
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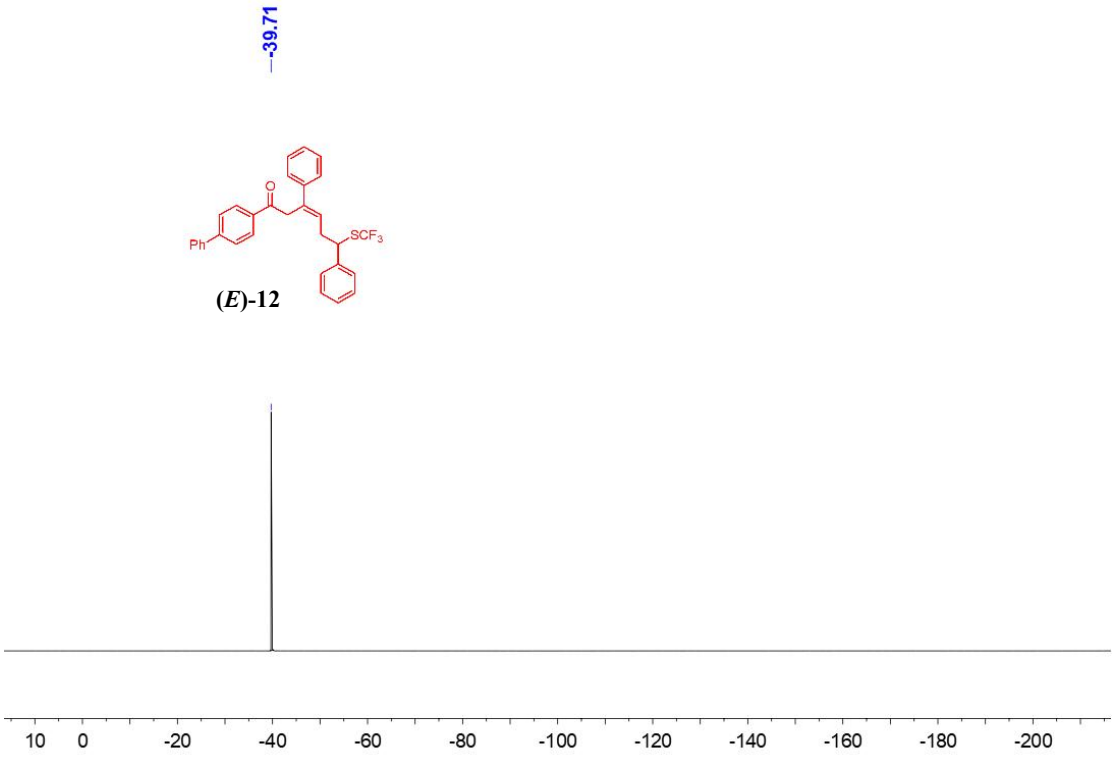
(*E*)-12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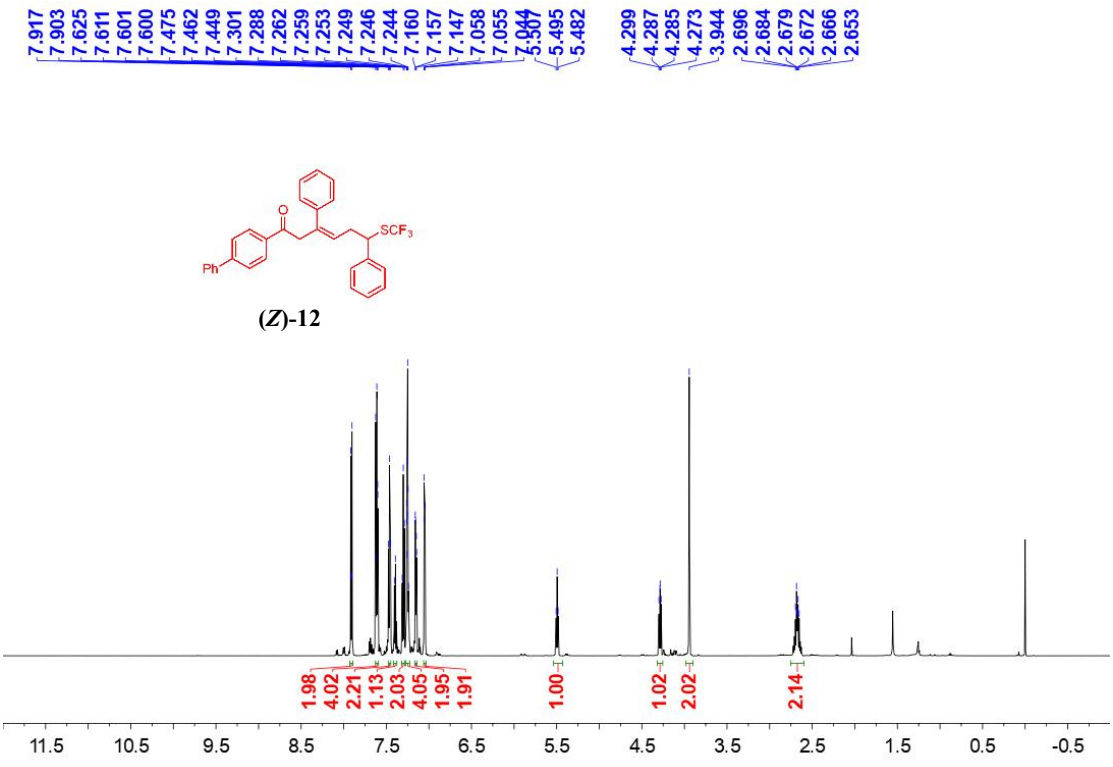
(*E*)-12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7.055
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4.285
4.273
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2.684
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2.672
2.666
2.653
SCF3
Ph
1.98
4.02
2.21
1.13
2.03
4.05
1.95
1.91
1.00
1.02
2.02
2.14
11.5
10.5
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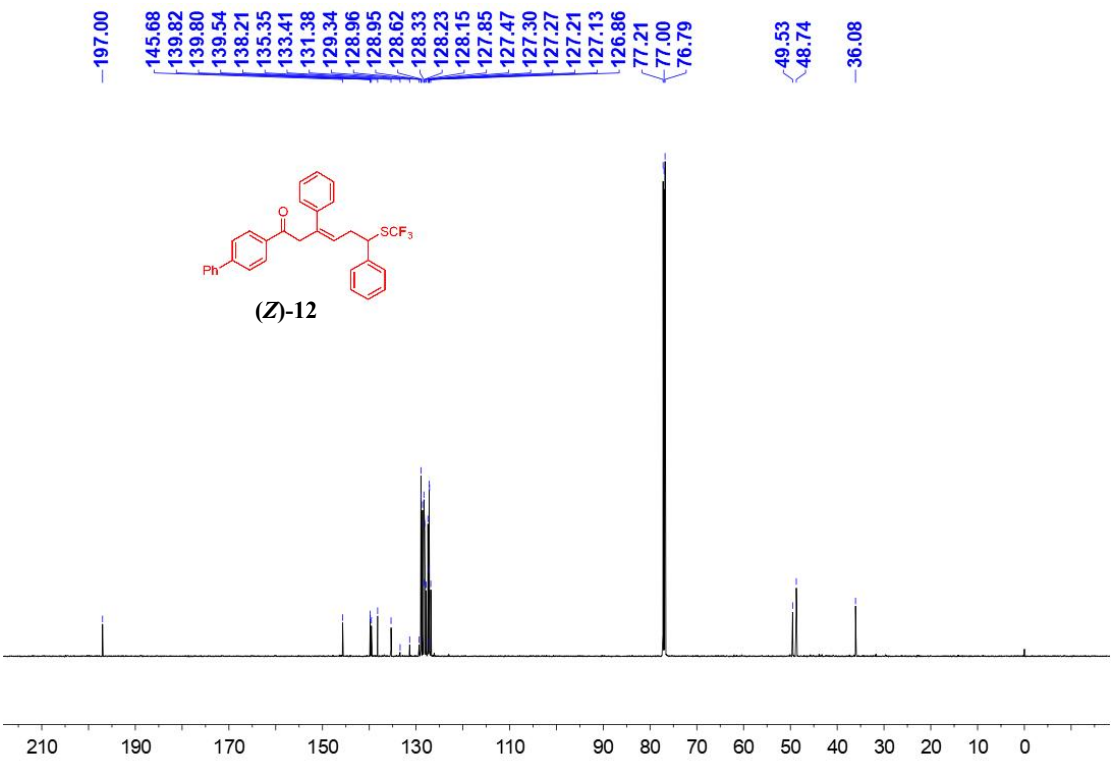
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127.27
127.21
127.13
126.86
77.21
77.00
76.79
49.53
48.74
36.08
SCF3
Ph
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20
10
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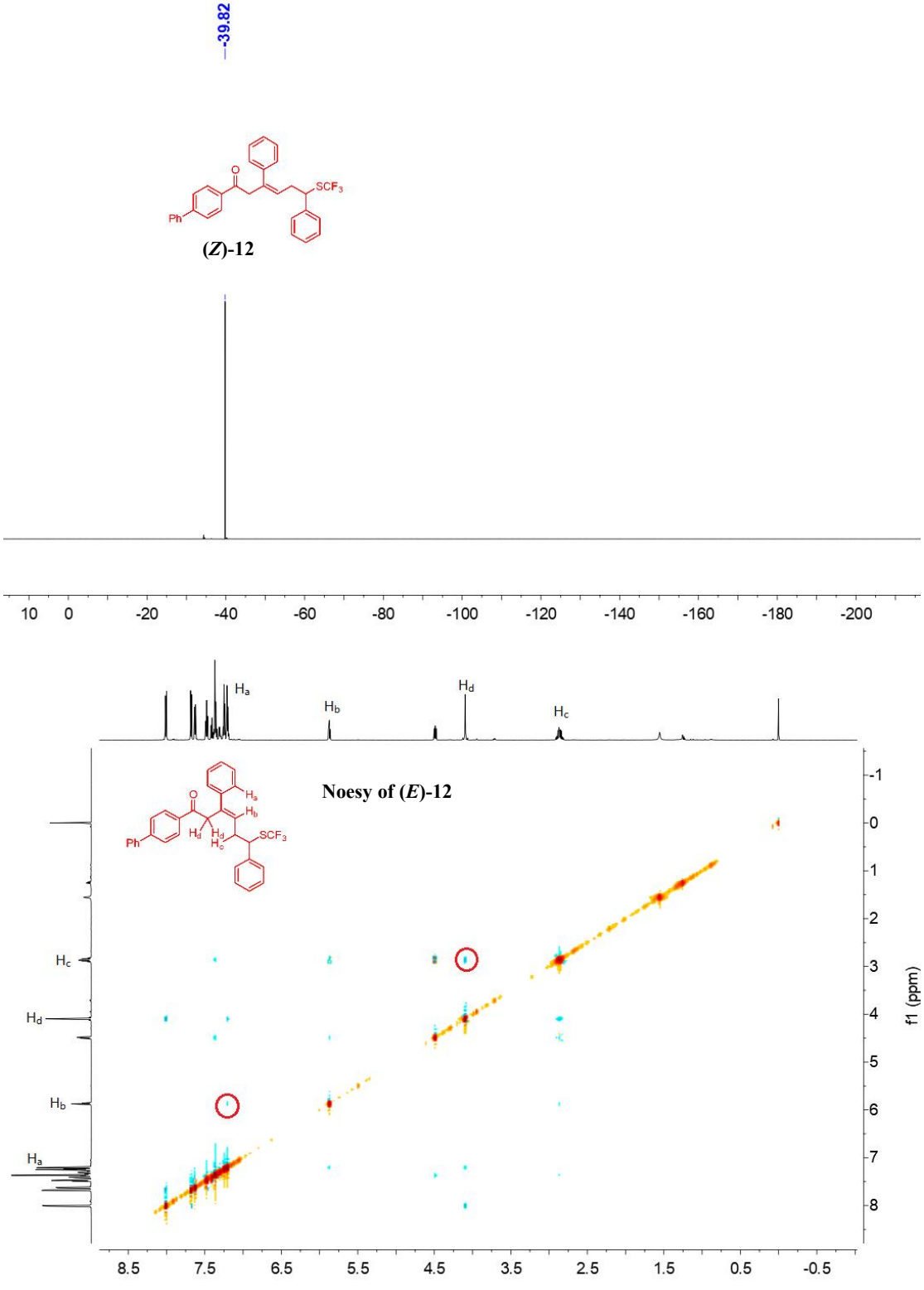
(*Z*)-12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Ha
Hb
Hd
Hc
Noesy of (E)-12
Ph
SCF3
Hc
Hd
Hb
Ha
8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
f1 (ppm)

(*Z*)-12


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7.553
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7.537
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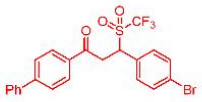
3.937
3.921


O
O
S
CF3
O
Ph
Br

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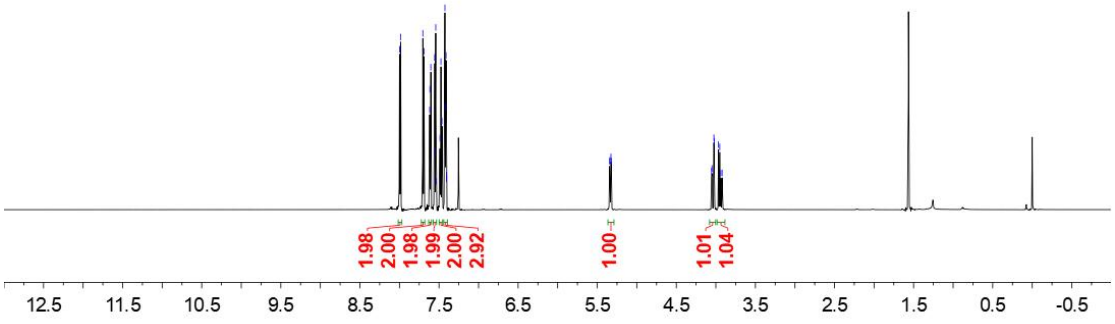
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6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5


192.35
147.01
139.38
134.04
132.44
131.47
129.03
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128.58
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123.08
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77.00
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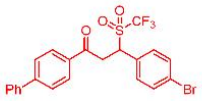
37.11


O
O
S
CF3
O
Ph
Br

13

210
190
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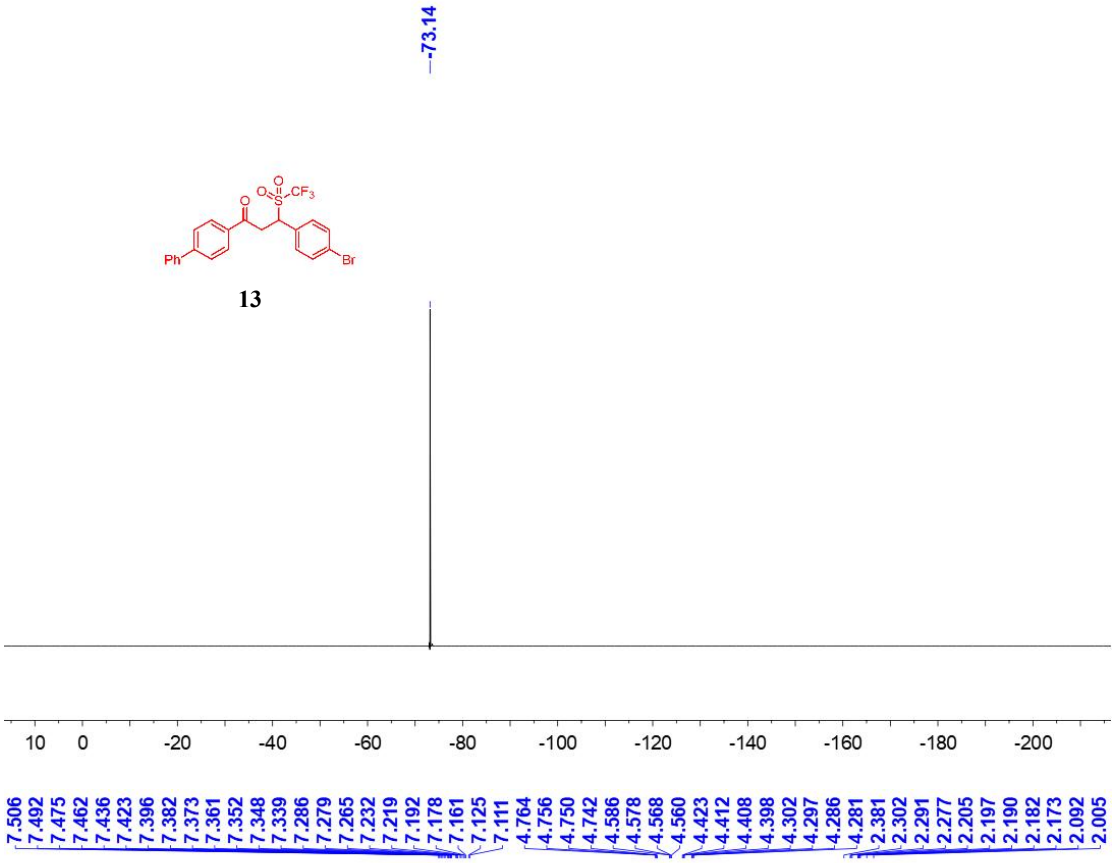
30
20
10
0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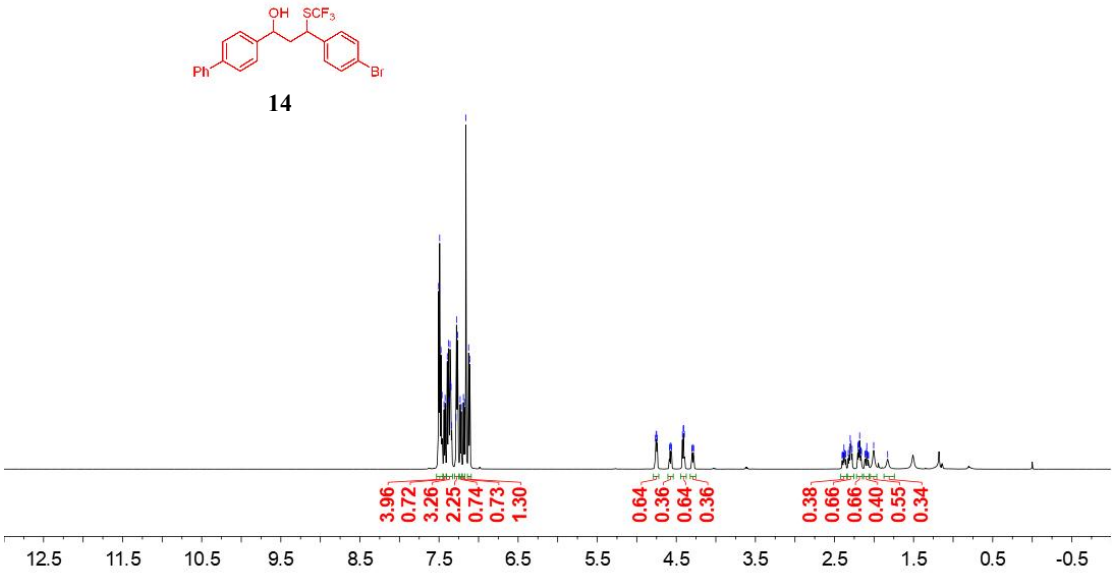
13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14


142.41
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138.98
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132.06
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131.20
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129.26
129.16
129.09
128.82
127.49
127.47
127.43
127.22
127.05
126.35
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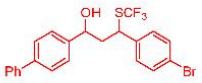
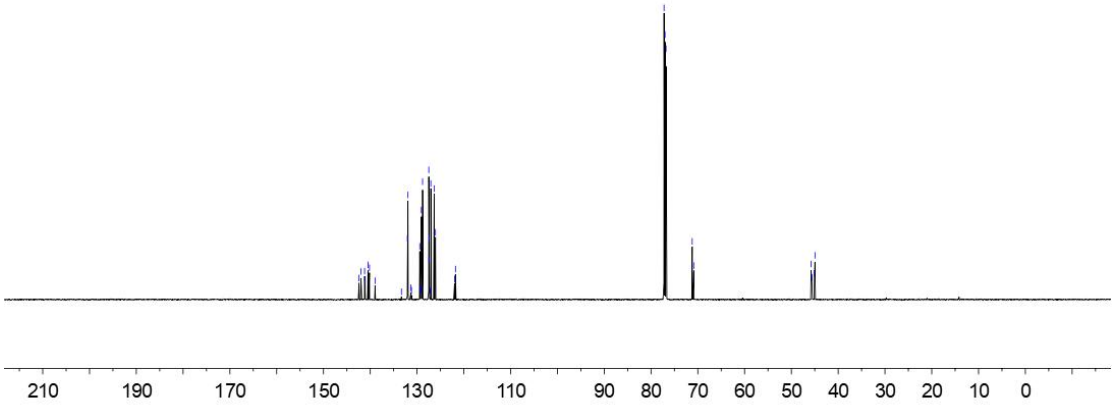
45.09
44.96


OH
SCF3
Ph
Br

14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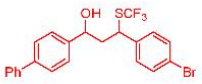
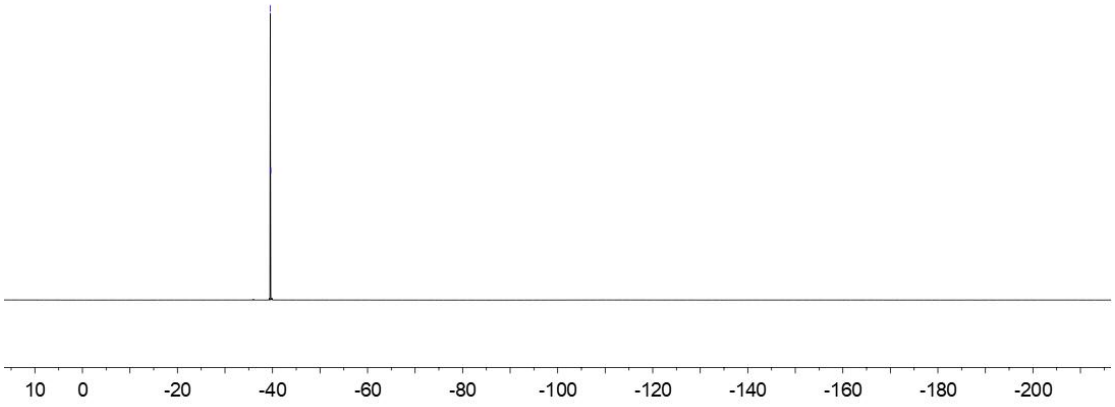
130
110
90
80
70
60
50
40
30
20
10
0


-39.52
-39.62


OH
SCF3
Ph
Br

14


10
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-40
-60
-80
-100
-120
-140
-160
-180
-200


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7.518
7.515
7.504
7.493
7.491
7.480
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7.374
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7.358
7.354
7.350
7.347
7.343
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7.291
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7.279
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7.267
7.220
7.217
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7.186
7.178
7.175
7.013
7.010
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6.850
6.839
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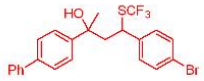
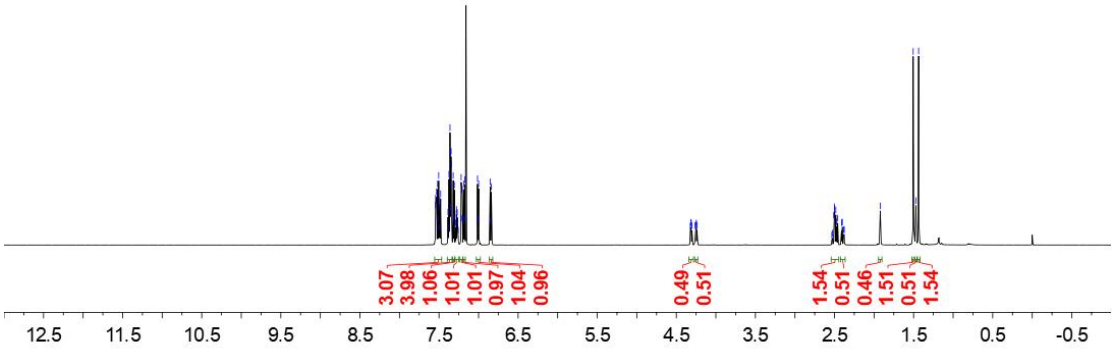

HO
SCF3
Ph
Br

15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0.46
1.51
0.51
1.54
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5


145.21
144.57
140.59
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140.07
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139.84
132.04
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125.11
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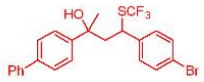
31.05


HO
SCF3
Ph
Br

15

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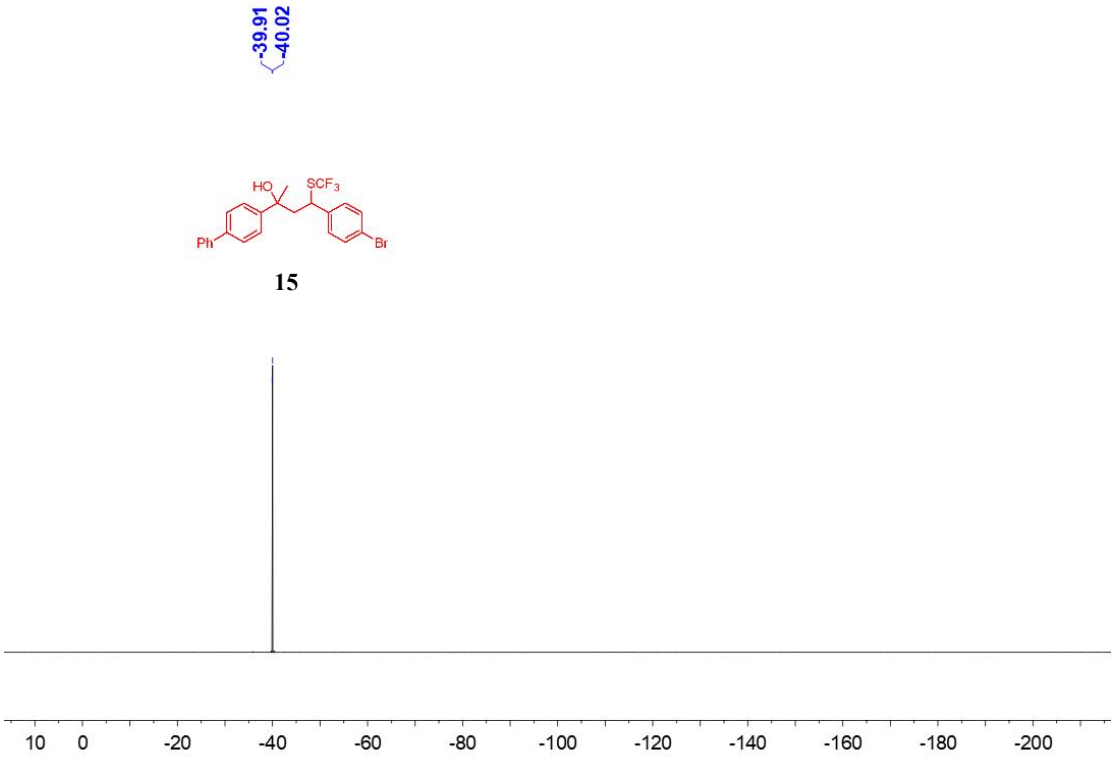
20
10
0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HO
SCF3
Ph
Br
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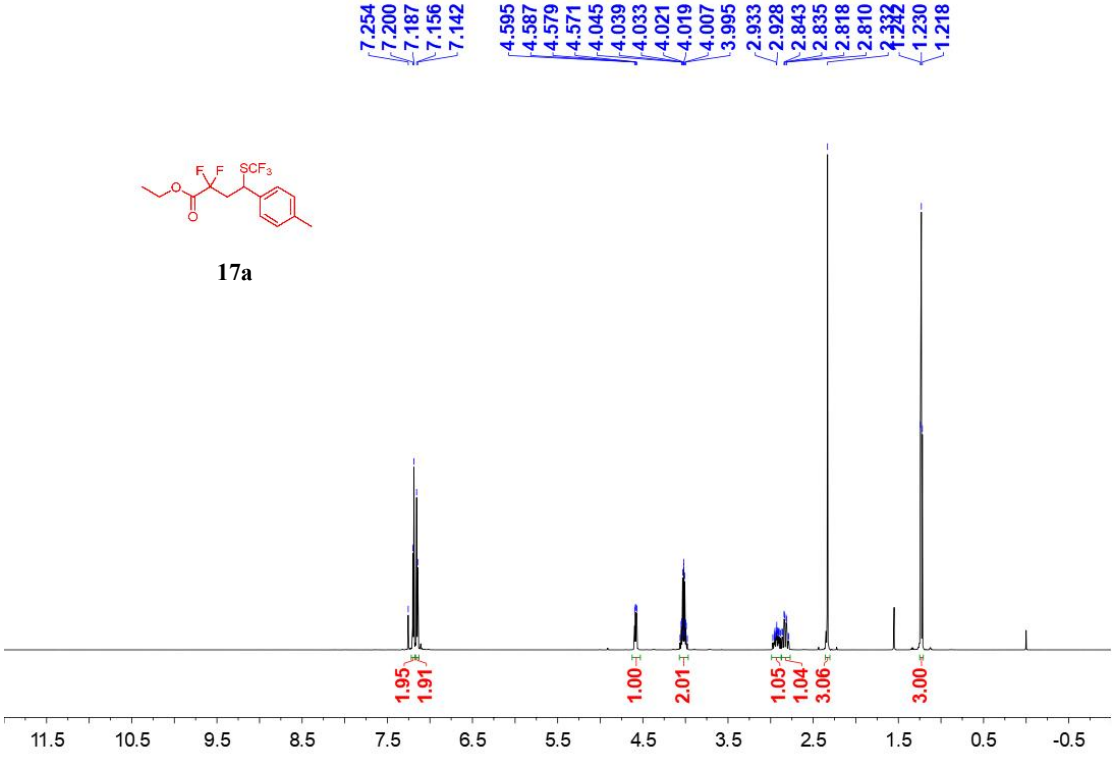
15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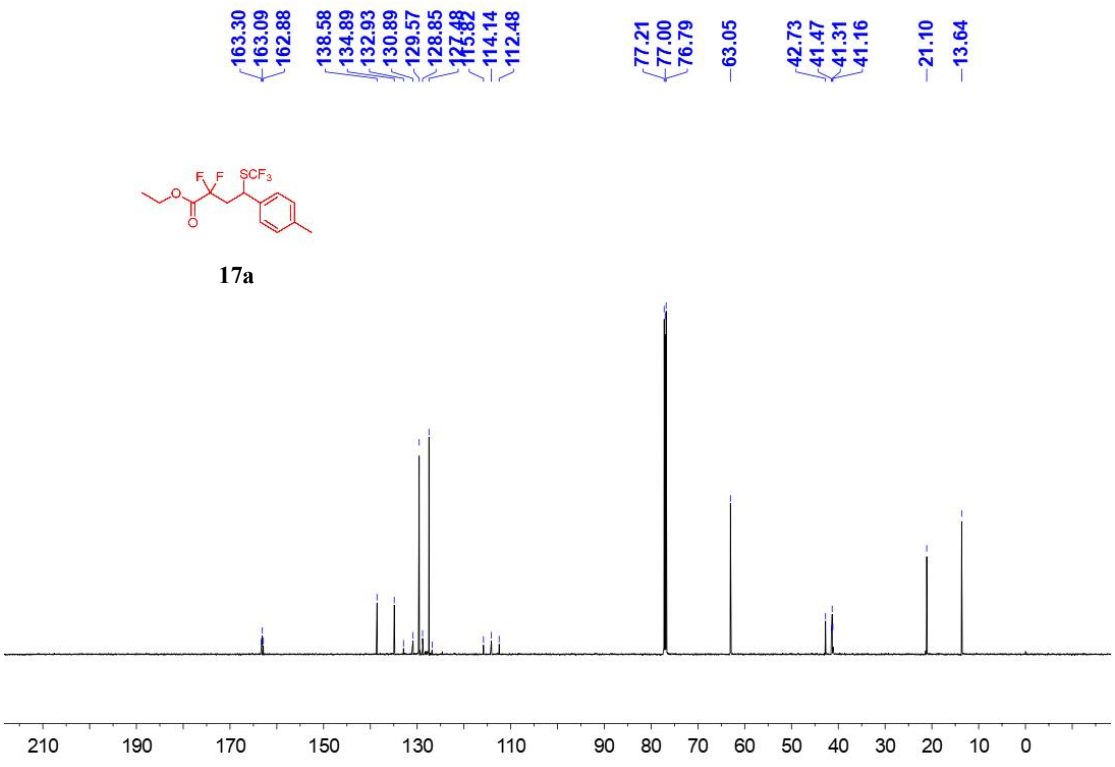
4.039
4.033
4.021
4.019
4.007
3.995
2.933
2.928
2.843
2.835
2.818
2.810
1.230
1.218
F F SCF3
O
O
1.95
1.91
1.00
2.01
1.05
1.04
3.06
3.00
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

17a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129.57
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114.14
112.48
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77.00
76.79
63.05
42.73
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21.10
13.64
F F SCF3
O
O
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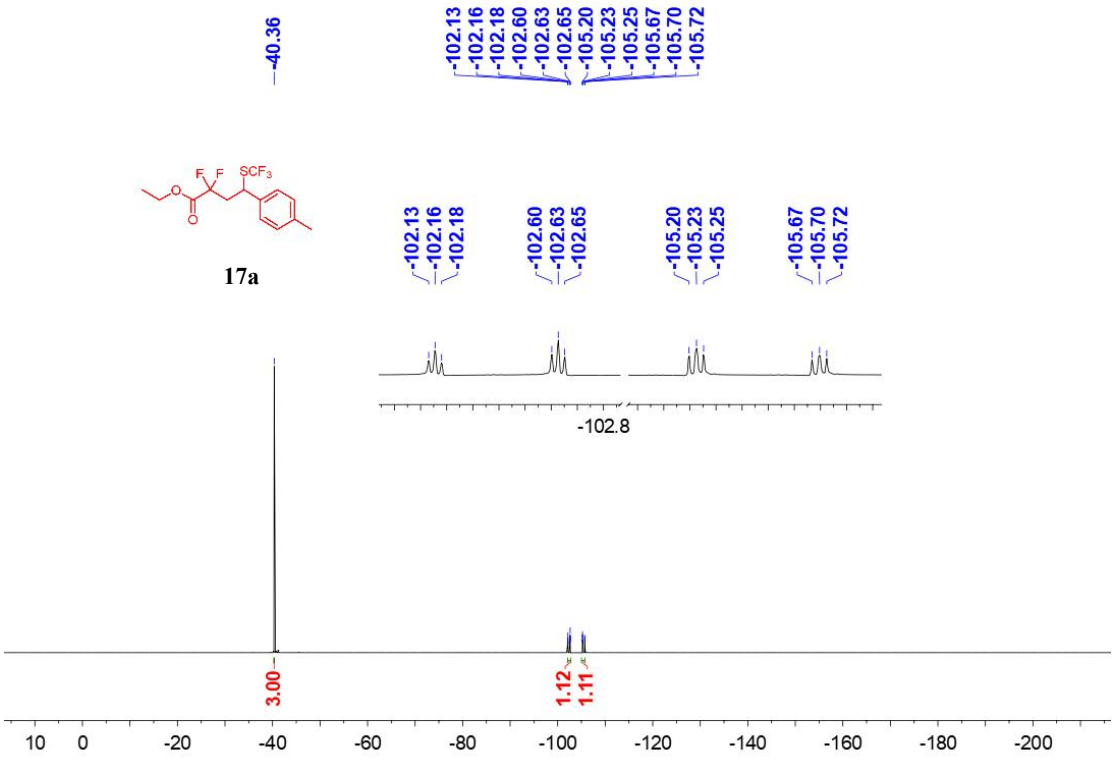
17a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102.63
102.65
105.20
105.23
105.25
105.67
105.70
105.72
F F SCF3
O
O
-102.8
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1.12
1.11
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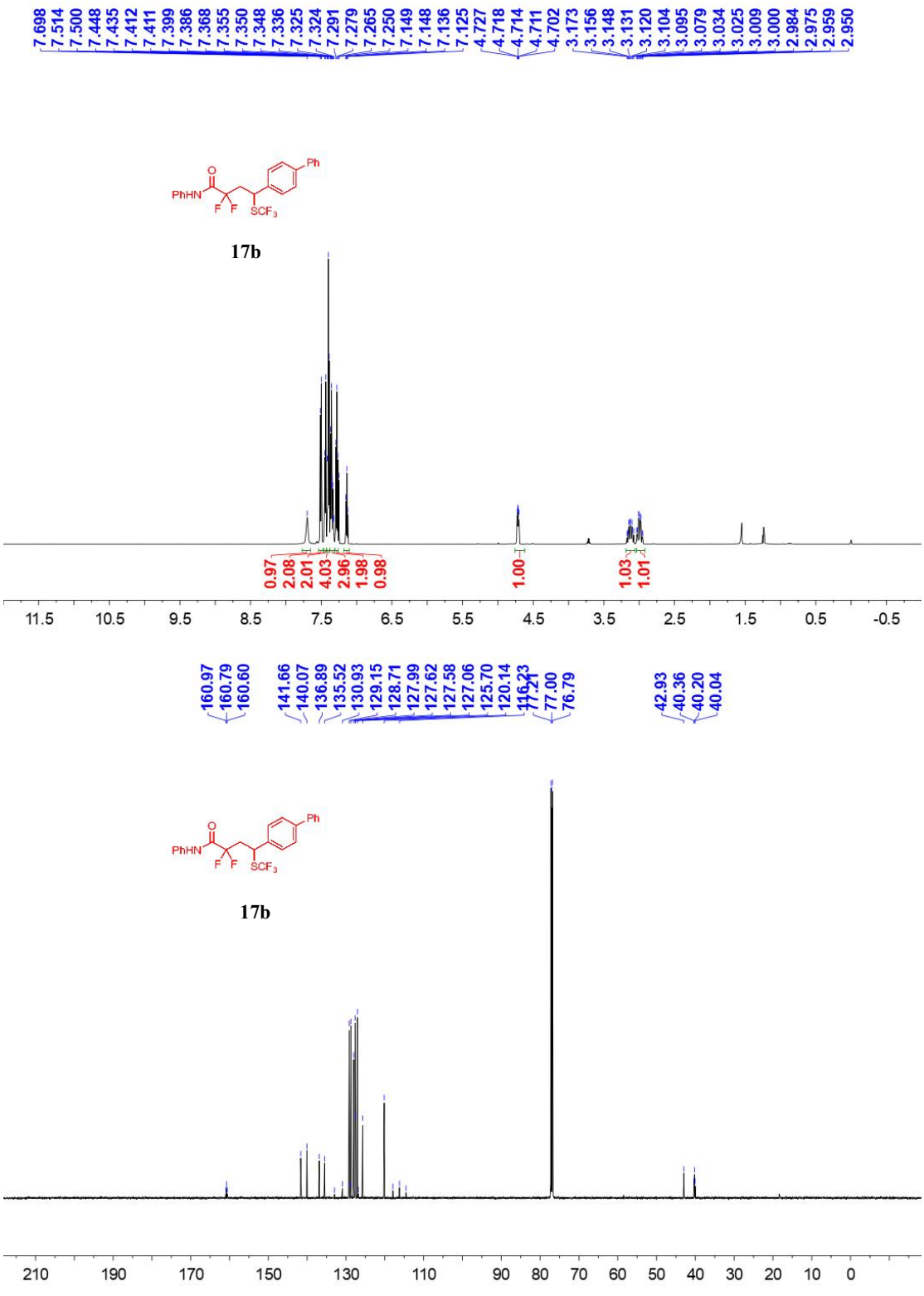
17a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7.348
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7.324
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3.034
3.025
3.009
3.000
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2.950
PhHN
O
F F
SCF3
Ph
17b
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4.03
2.96
1.98
0.98
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1.03
1.01
11.5
10.5
9.5
8.5
7.5
6.5
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3.5
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135.52
130.93
129.15
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127.99
127.62
127.58
127.06
125.70
120.14
116.23
77.41
77.00
76.79
42.93
40.36
40.20
40.04
PhHN
O
F F
SCF3
Ph
17b
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190
170
150
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70
60
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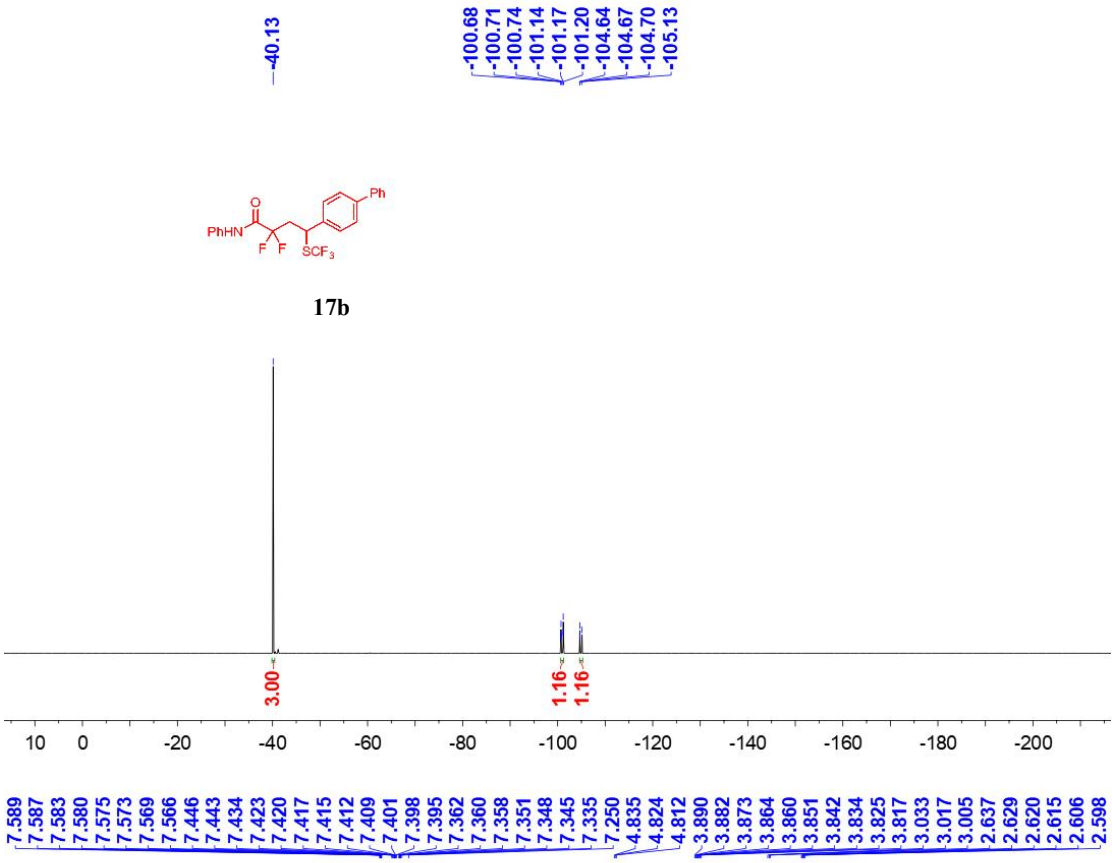
30
20
10
0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PhHN
O
F F
SCF3
Ph
3.00
1.16
1.16
10 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200
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7.587
7.583
7.580
7.575
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7.566
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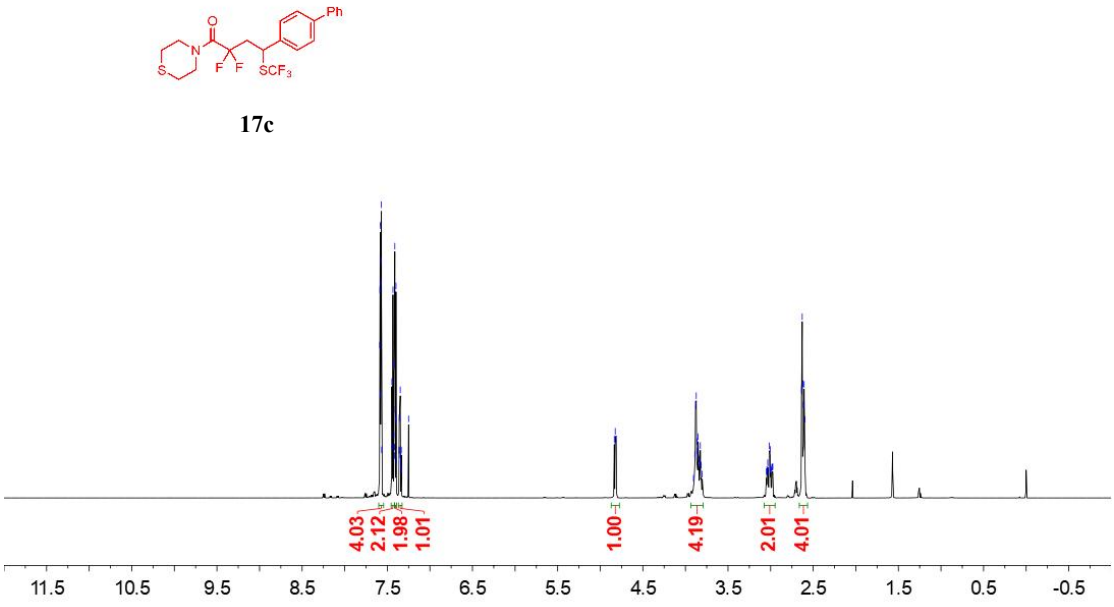
17b


N
O
F F
SCF3
Ph
S
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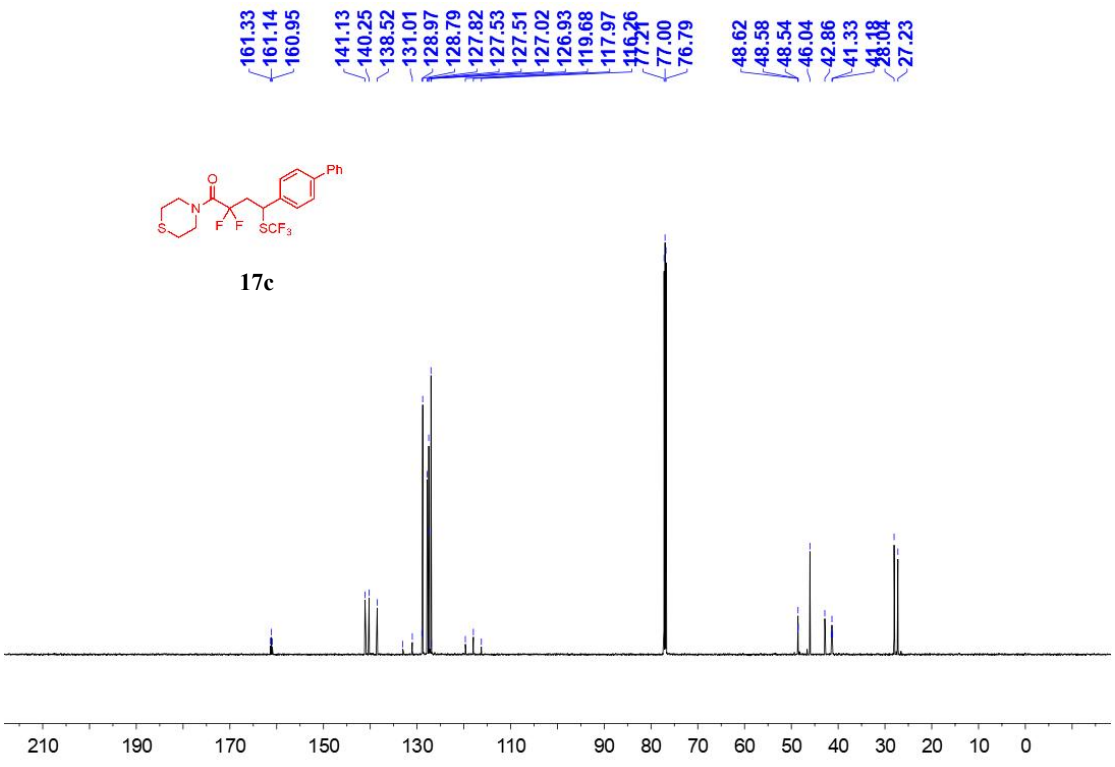
2.12
1.98
1.01
1.00
4.19
2.01
4.01
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

17c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128.79
127.82
127.53
127.51
127.02
126.93
119.68
117.97
116.26
77.21
77.00
76.79
48.62
48.58
48.54
46.04
42.86
41.33
28.04
27.23
Ph
O
N
S
F
F
SCF3
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20
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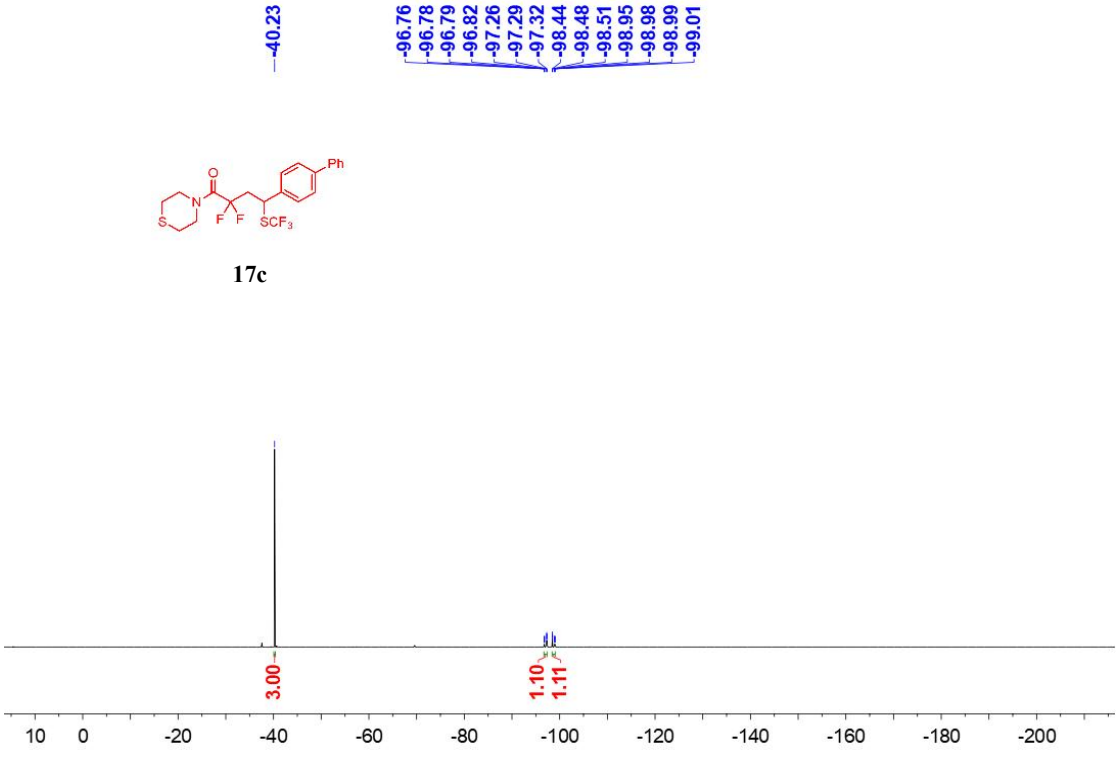
0

17c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-98.98
-98.99
-99.01
Ph
O
N
S
F
F
SCF3
3.00
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10
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-20
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-80
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-140
-160
-180
-200

17c


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7.585
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1.130
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Ph
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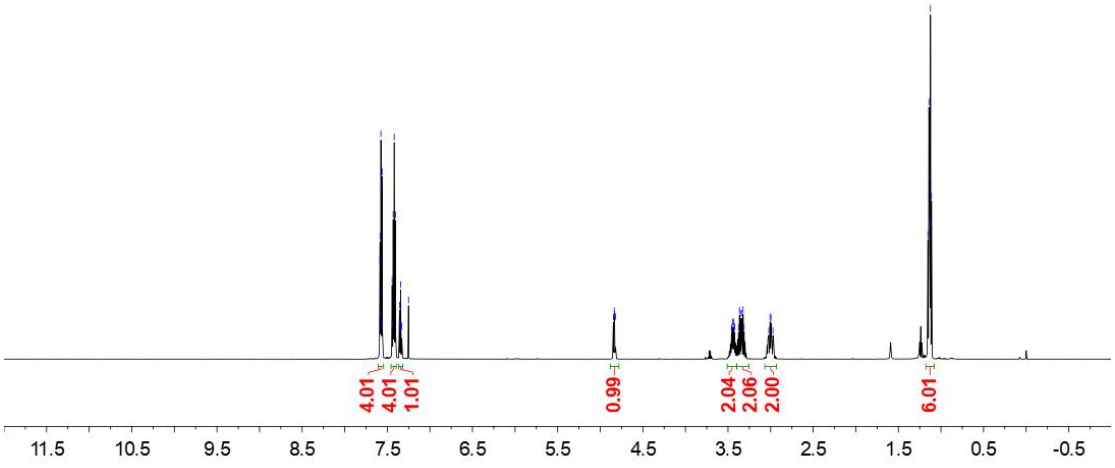
N
F
O
SCF3

17d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6.01
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7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5


162.09
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161.72
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127.03
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118.01
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77.00
76.79
43.01
41.83
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41.58
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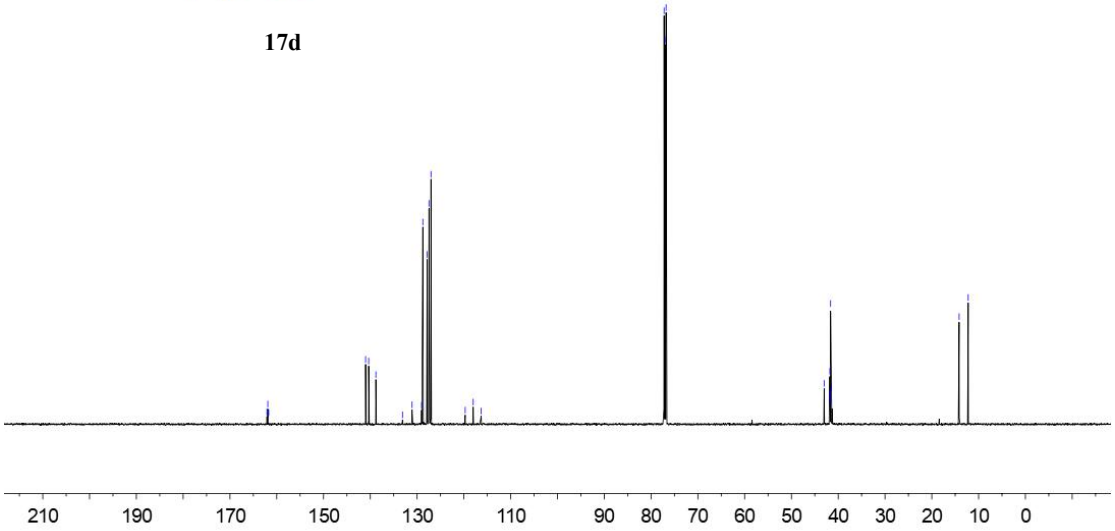
12.22


Ph
F
N
F
O
SCF3

17d


210
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170
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130
110
90
80
70
60
50
40
30
20
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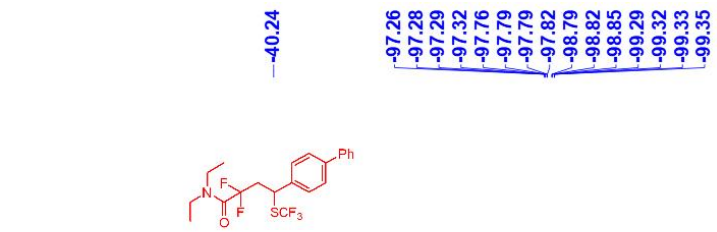
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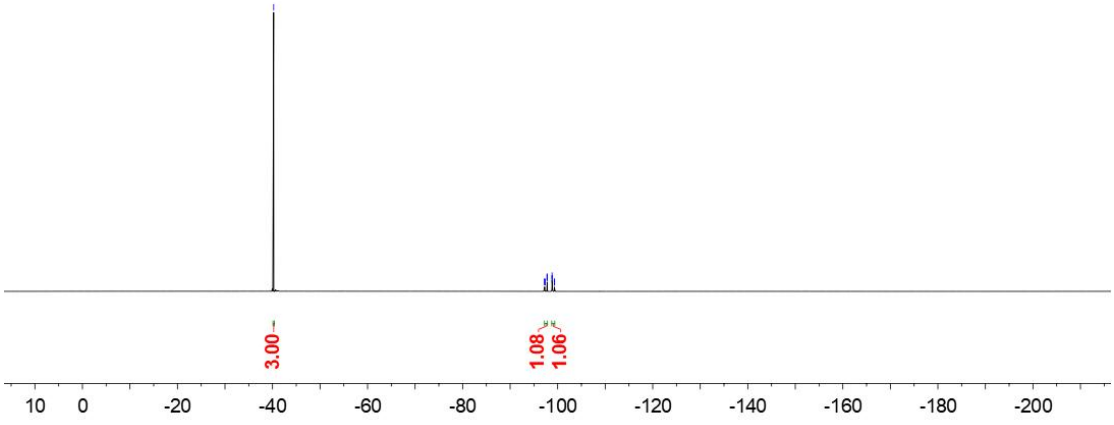
F
O
SCF3

17d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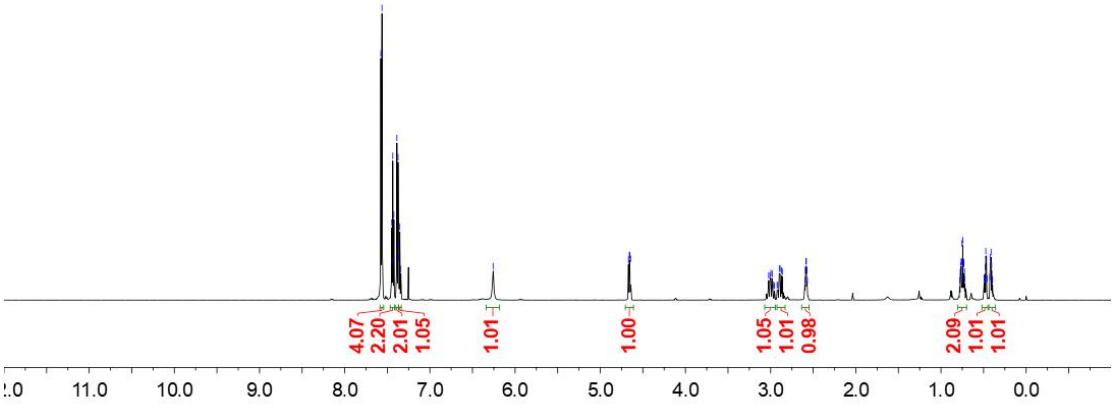
F
F
O
SCF3

17e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1.01
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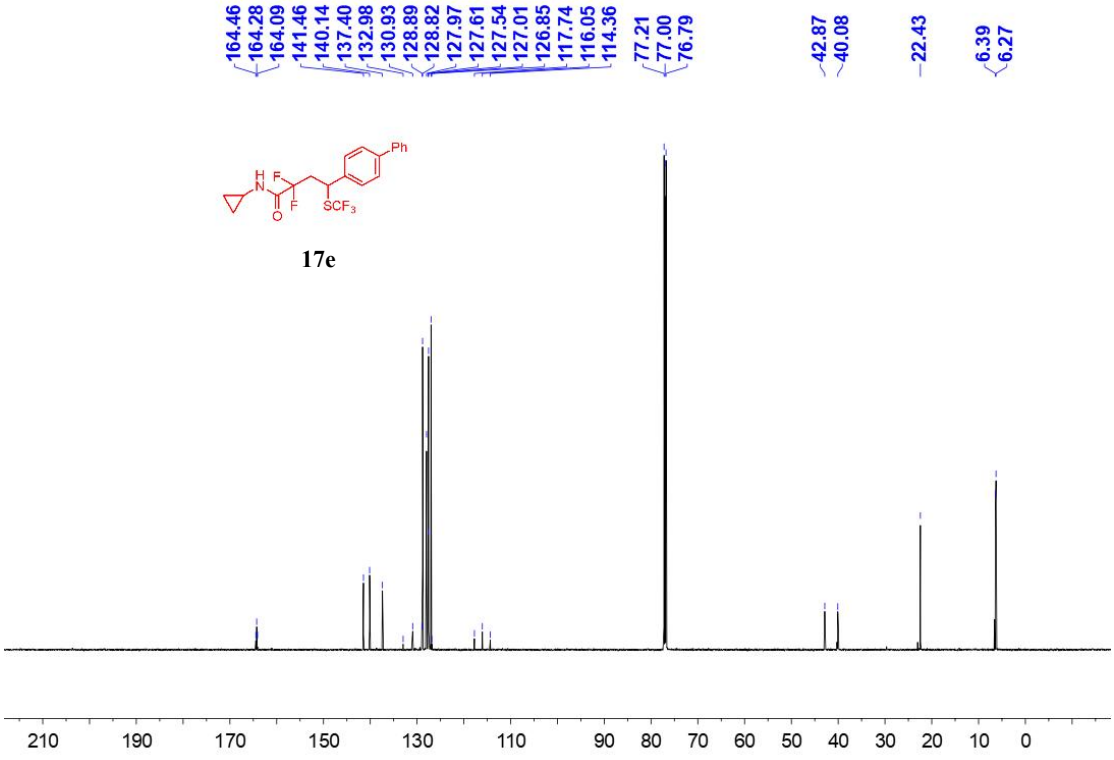
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40.08
22.43
6.39
6.27
Ph
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H
N
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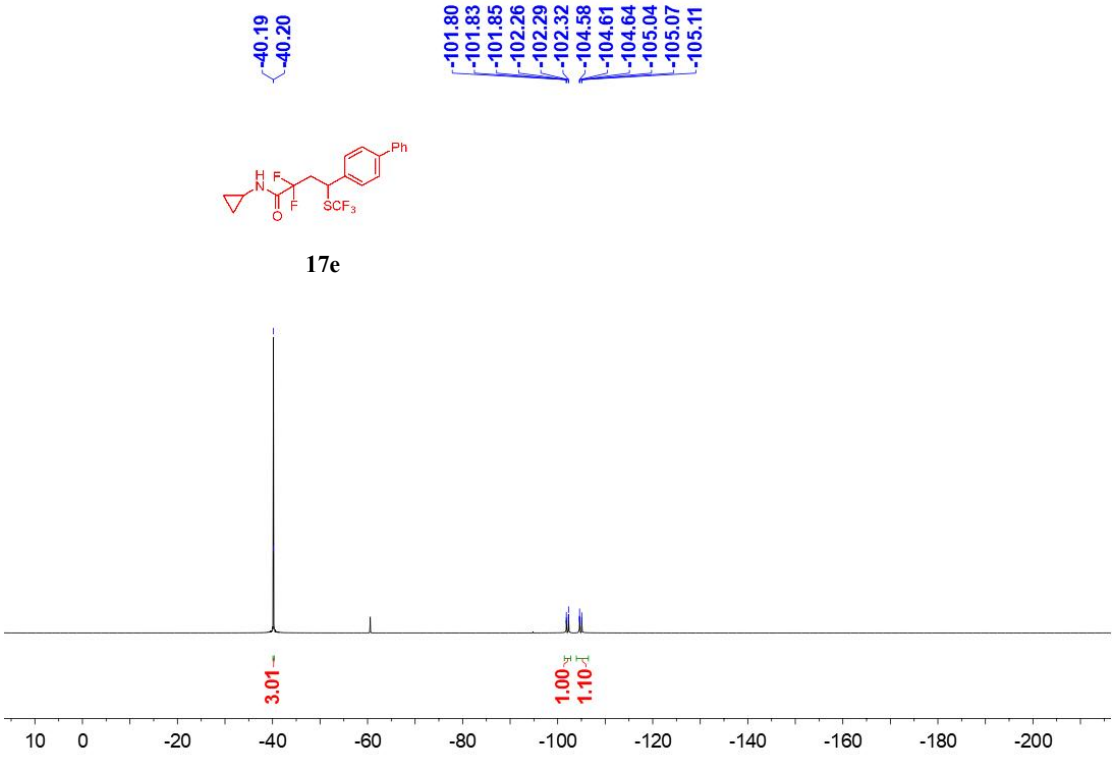
17e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Ph
F
H
N
F
O
SCF3
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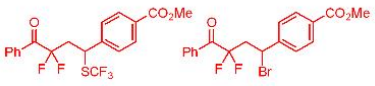
17e



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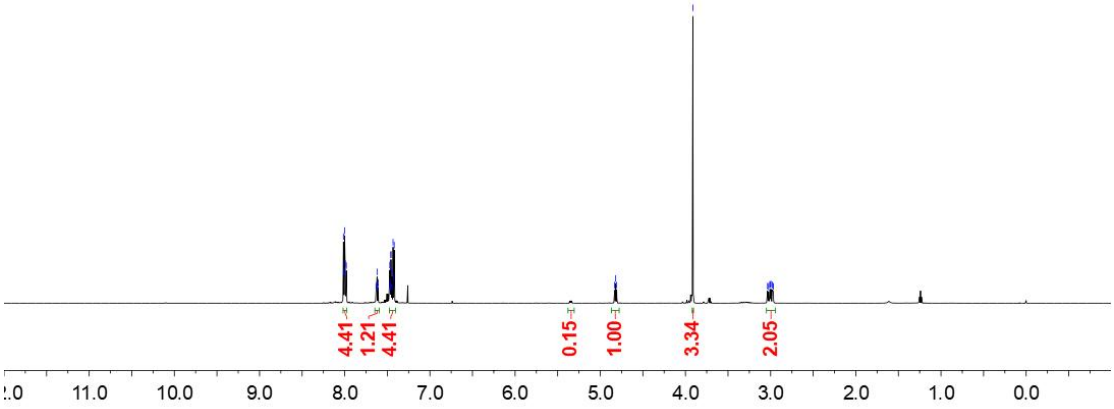

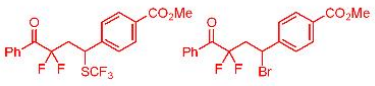

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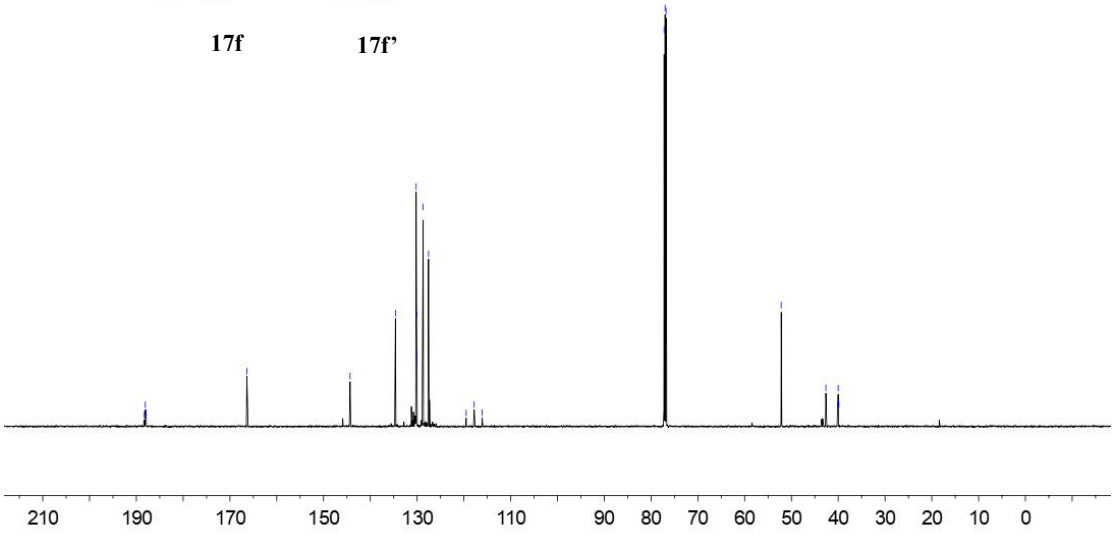

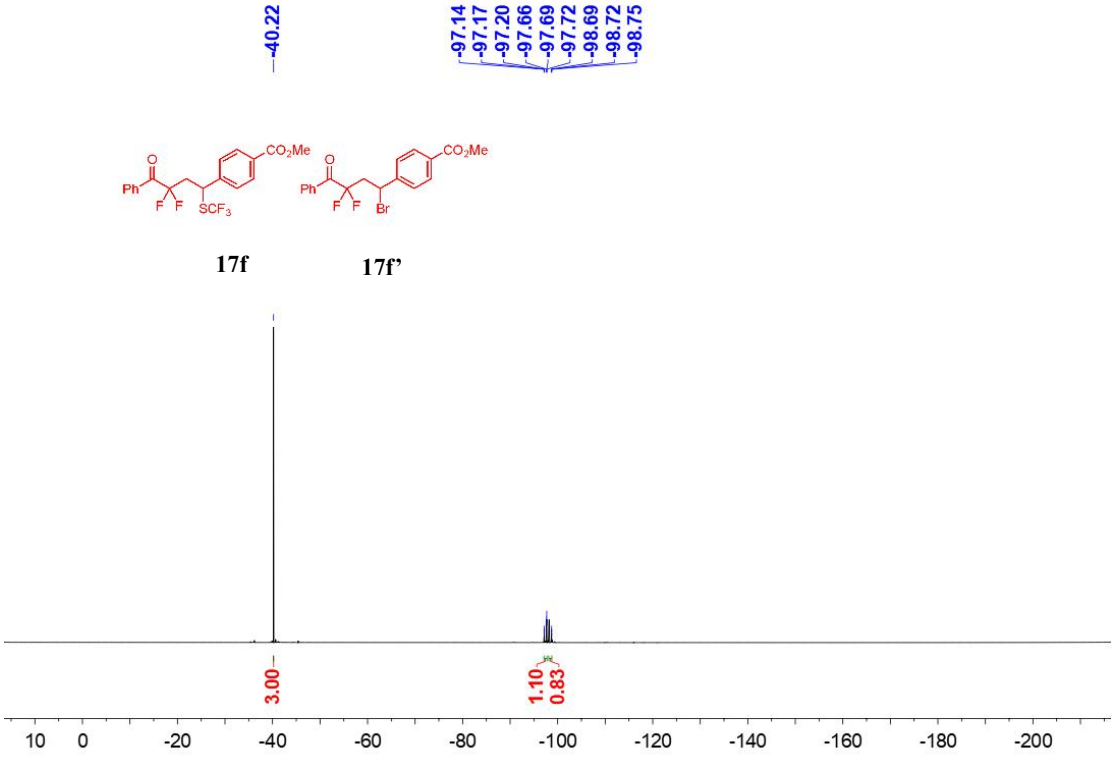
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CO2Me
Ph
F F
SCF3
Br
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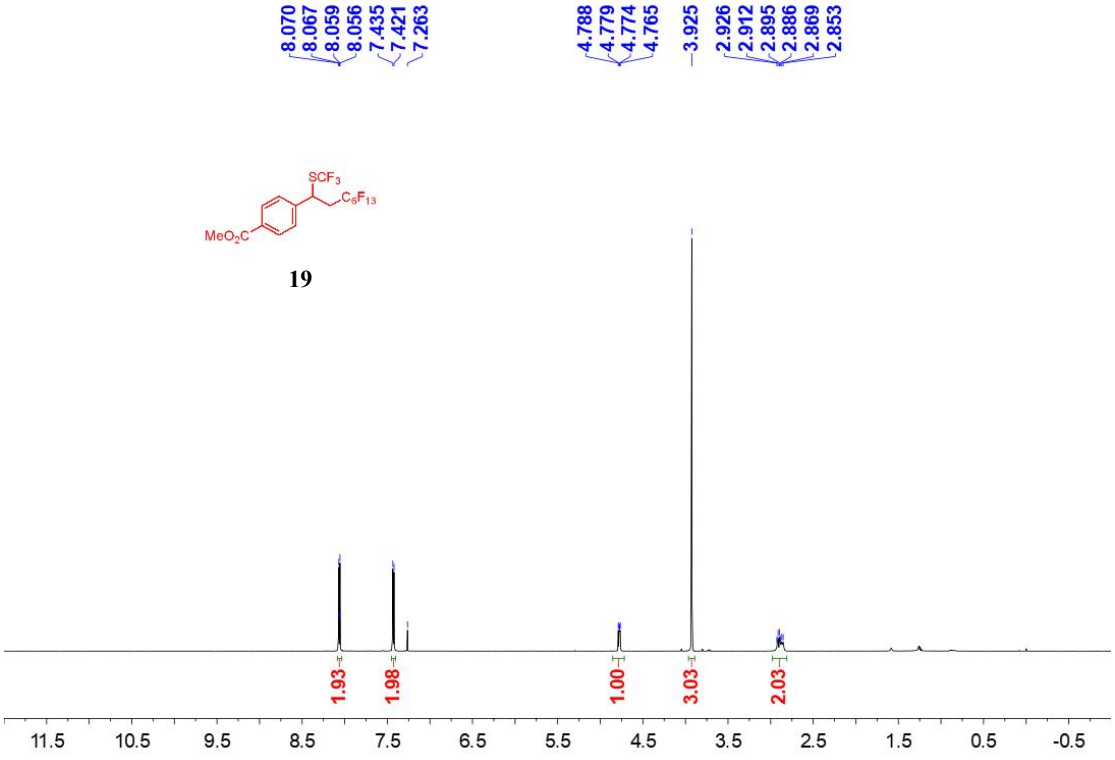
1.10
0.83
10 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200

17f **17f'**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2.895
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2.853
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C6F13
MeO2C
1.93
1.98
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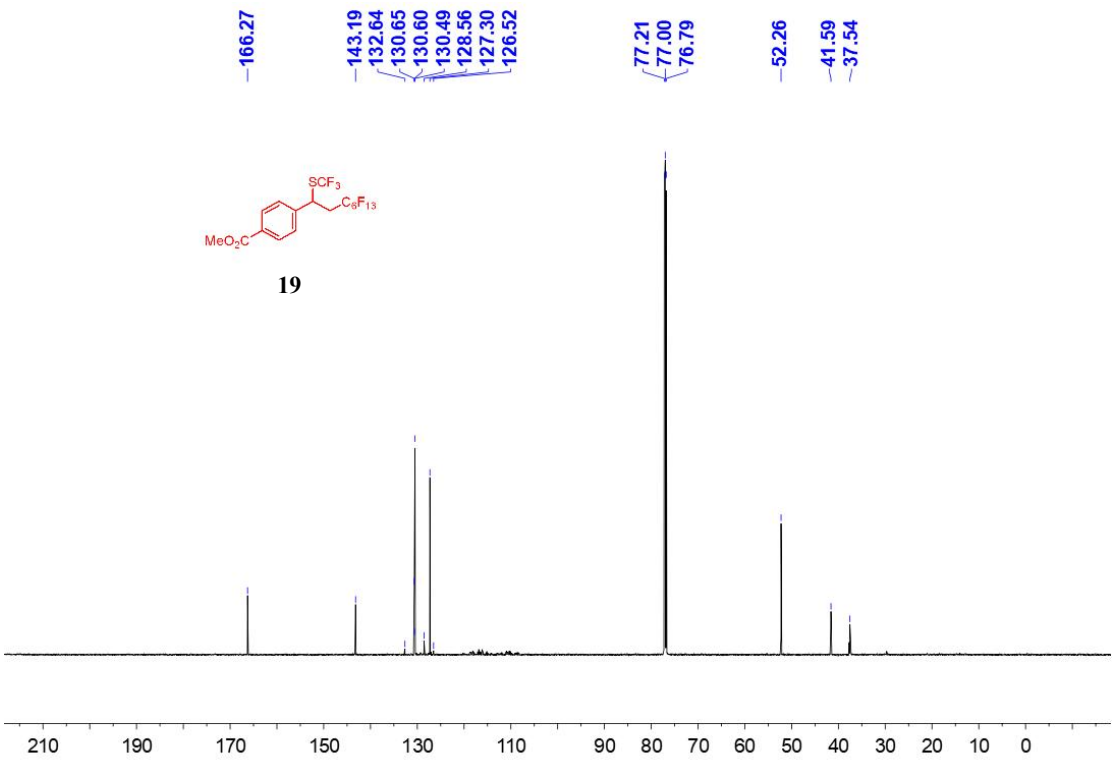
2.03
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5

19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52.26
41.59
37.54
SCF3
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170
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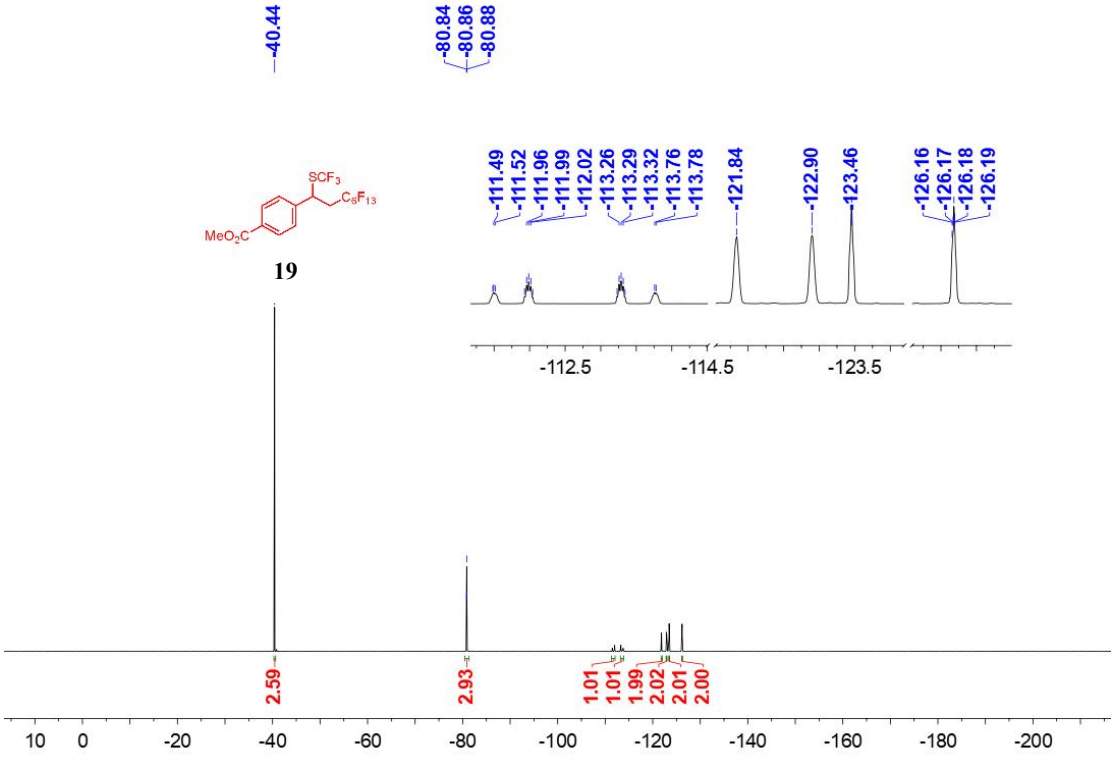
80
70
60
50
40
30
20
10
0

19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122.90
123.46
126.16
126.17
126.18
126.19
SCF3
C6F13
MeO2C
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-114.5
-123.5
2.59
2.93
1.01
1.01
1.99
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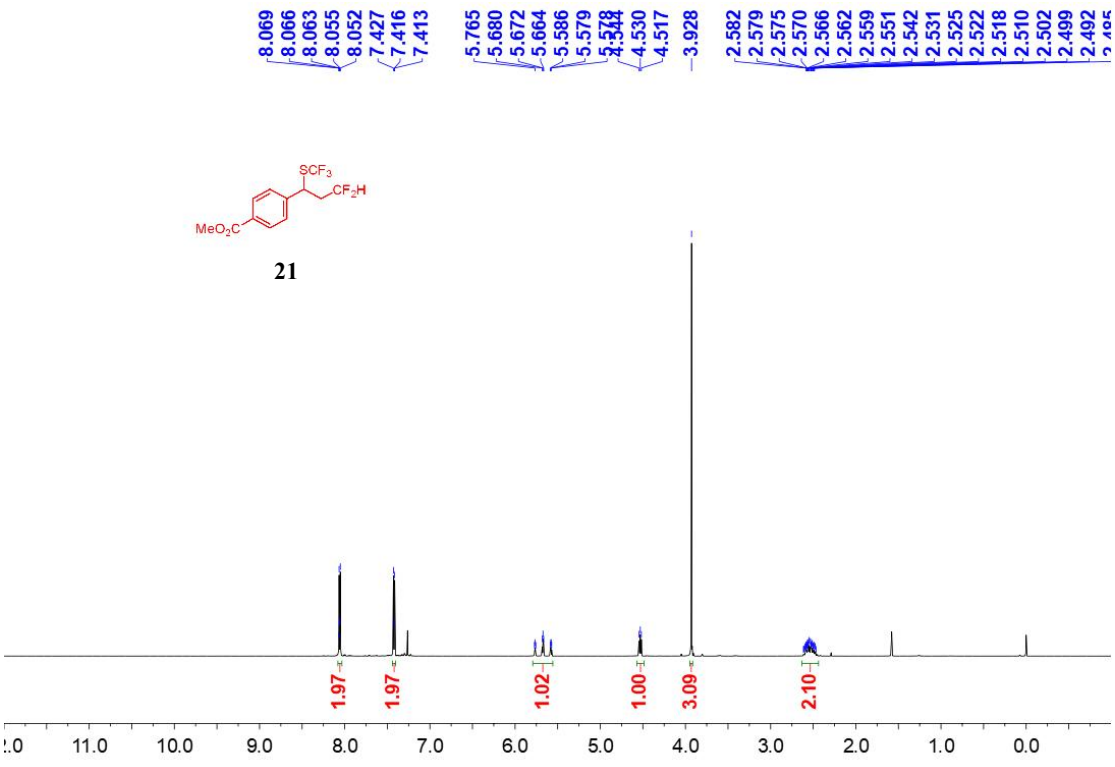
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5.579
4.530
4.517
3.928
2.582
2.579
2.575
2.570
2.566
2.562
2.559
2.551
2.542
2.531
2.525
2.522
2.518
2.510
2.502
2.499
2.492
SCF3
CF2H
MeO2C
1.97
1.97
1.02
1.00
3.09
2.10
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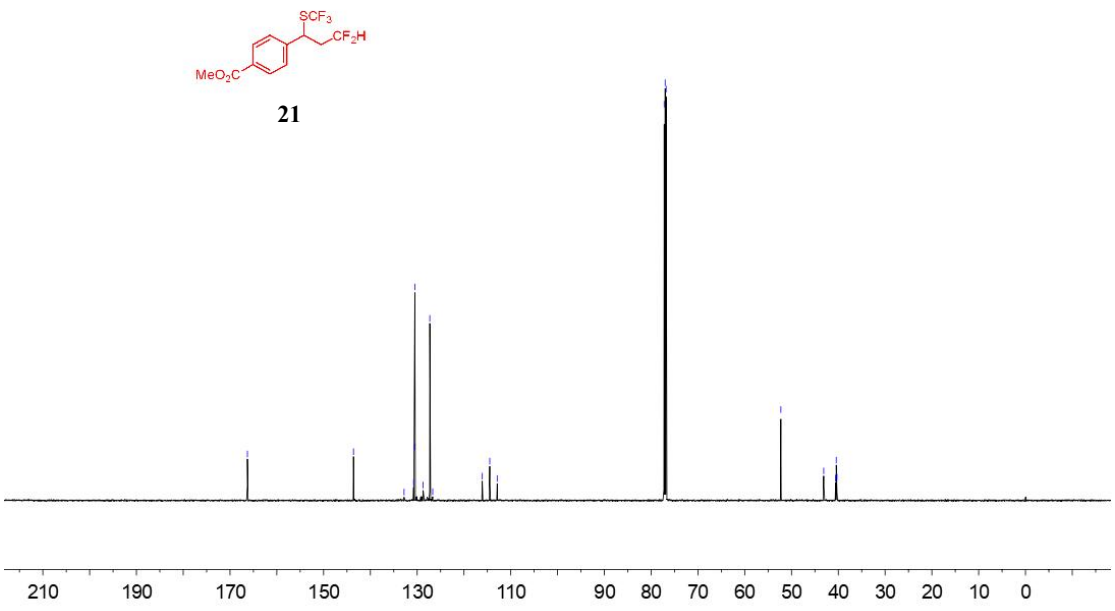
21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40.59
40.44
40.29
SCF3
CF2H
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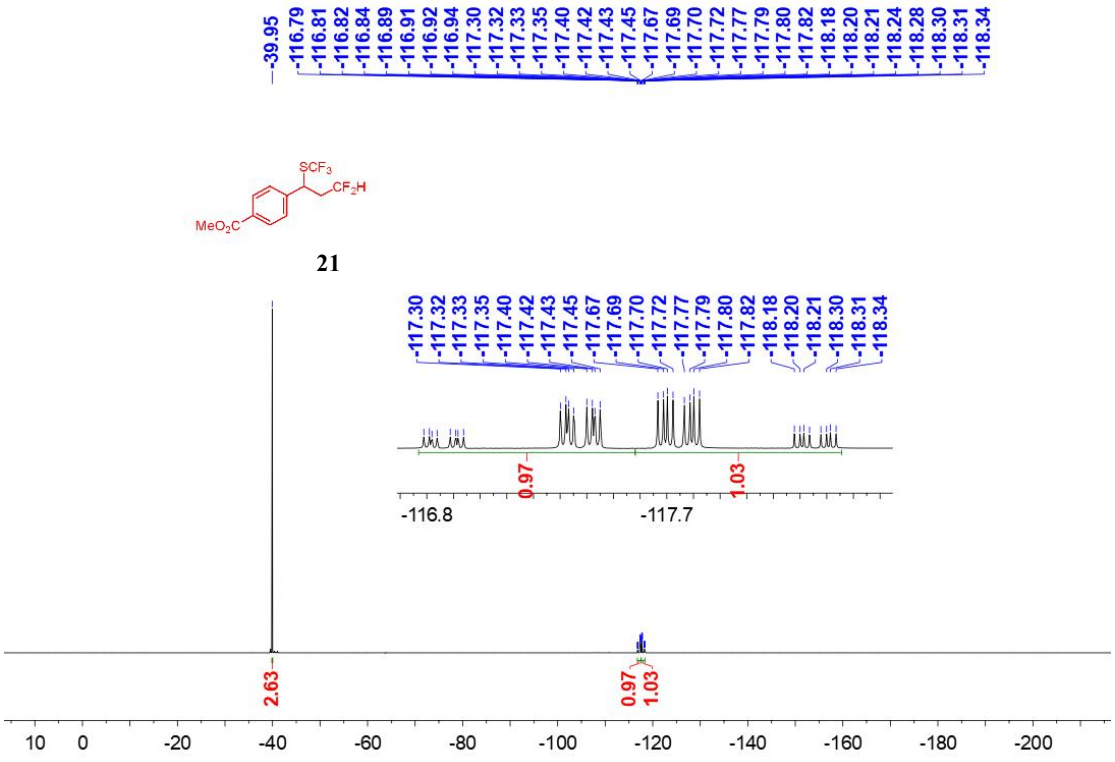
10
0

21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CF2H
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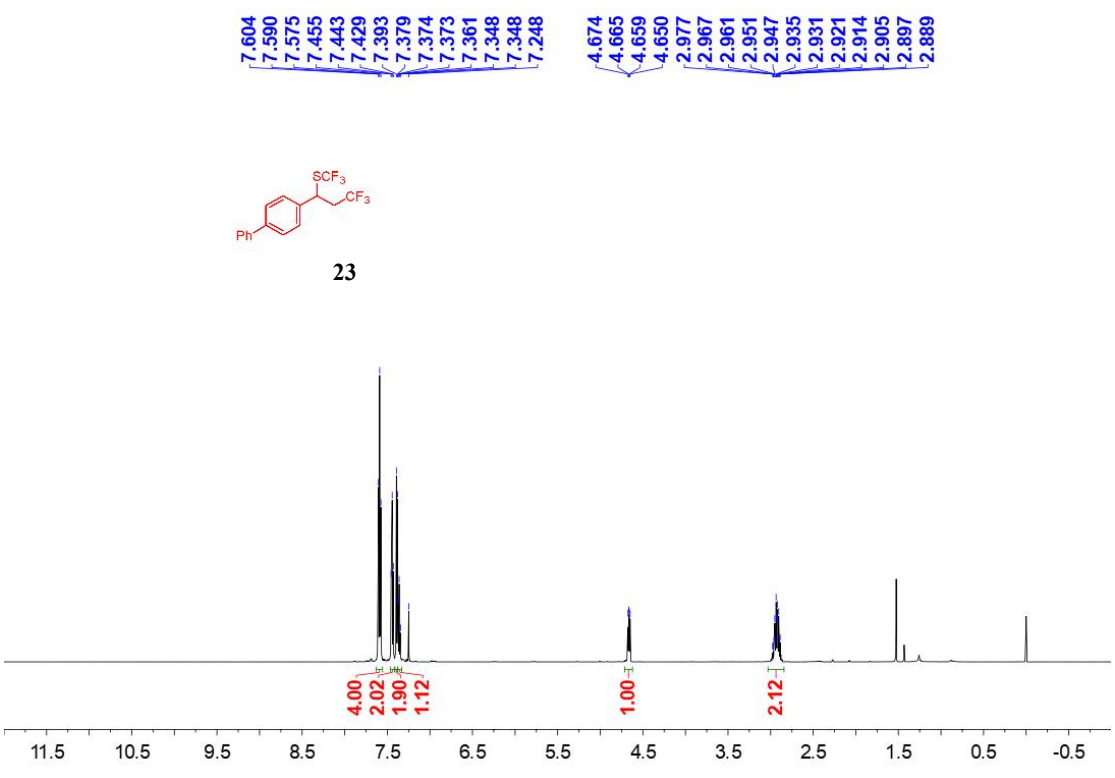
21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4.650
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2.931
2.921
2.914
2.905
2.897
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CF3
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2.12
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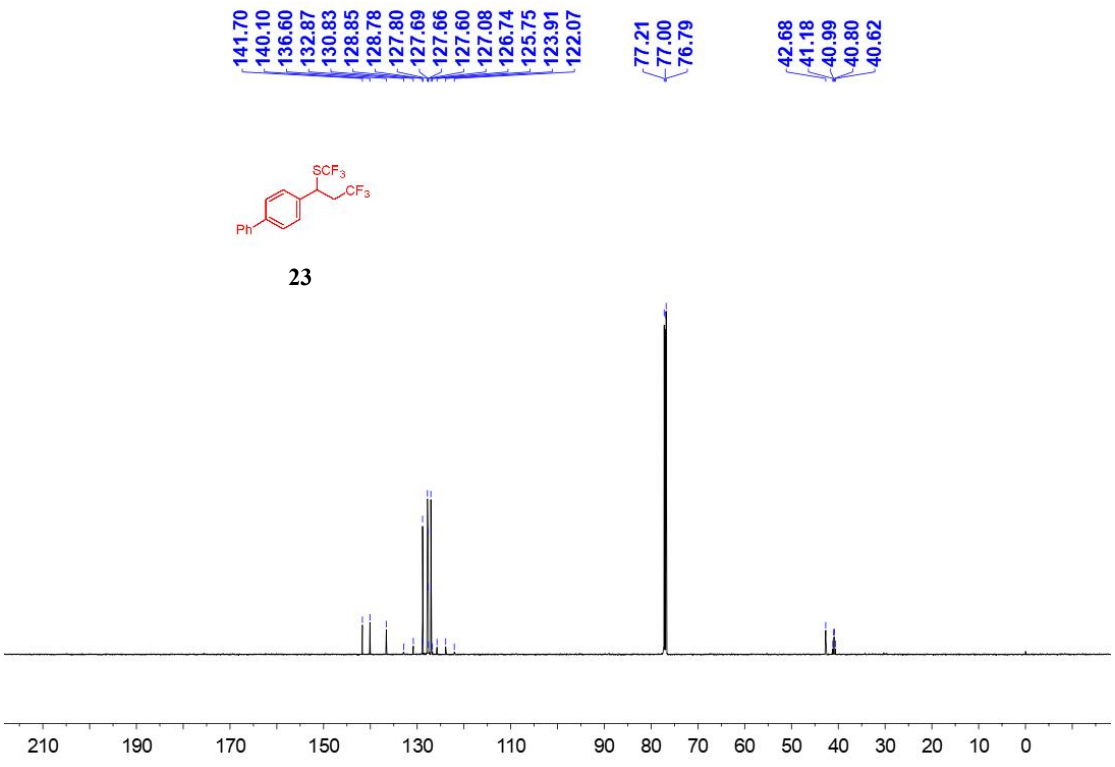
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76.79
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41.18
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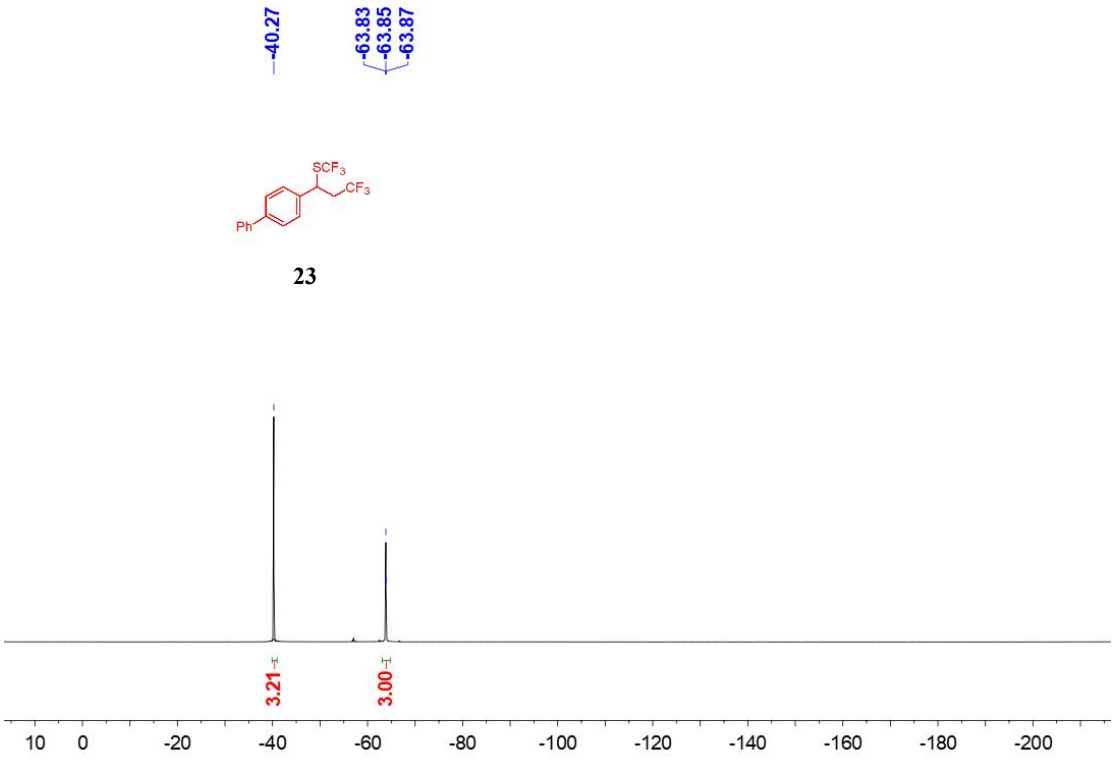
0

23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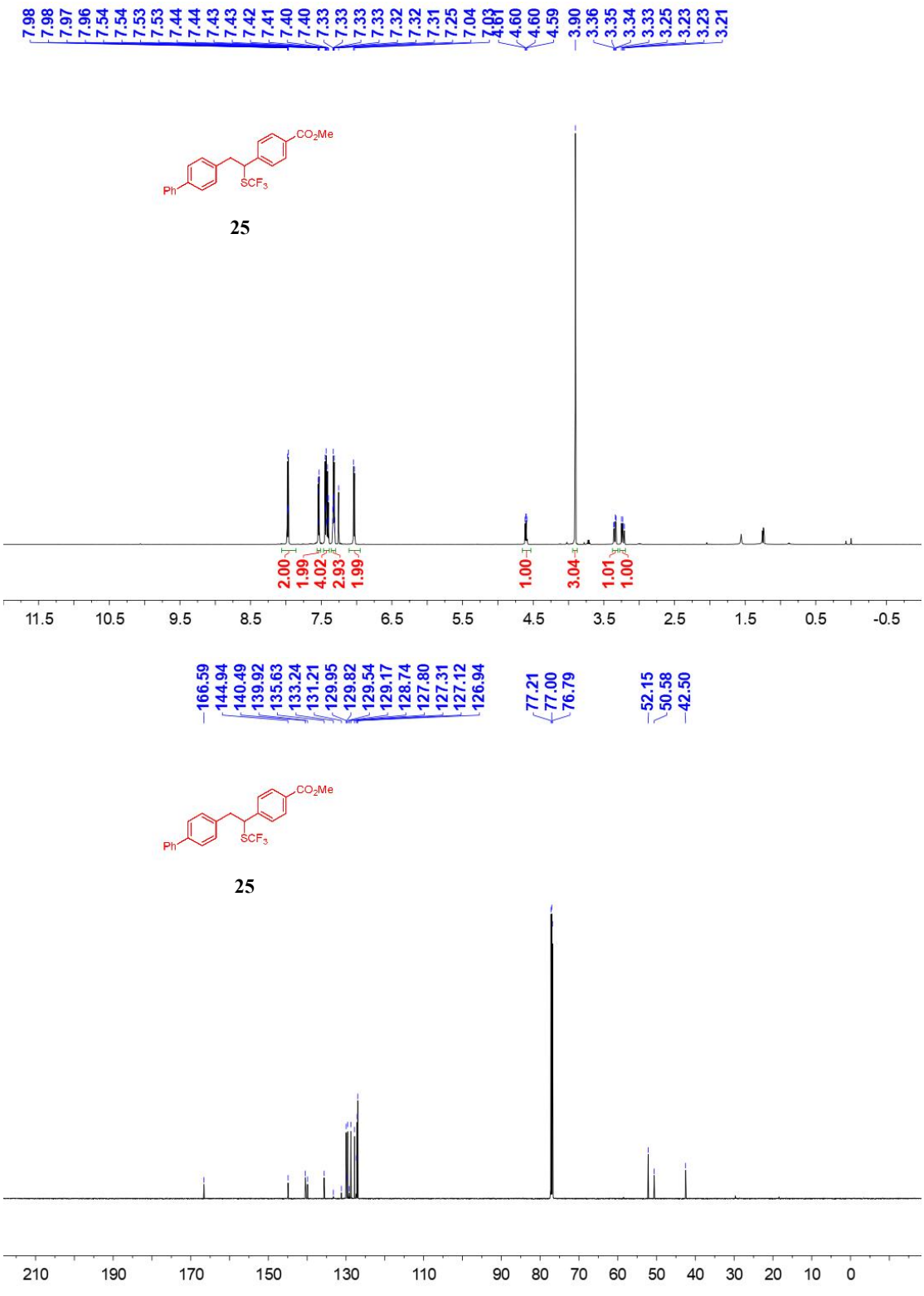
23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CO2Me
SCF3
Ph
2.00 1.99 4.02 2.93 1.99 1.00 3.04 1.01 1.00
11.5 10.5 9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5 -0.5
166.59 144.94 140.49 139.92 135.63 133.24 131.21 129.95 129.82 129.54 129.17 128.74 127.80 127.31 127.12 126.94 77.21 77.00 76.79 52.15 50.58 42.50
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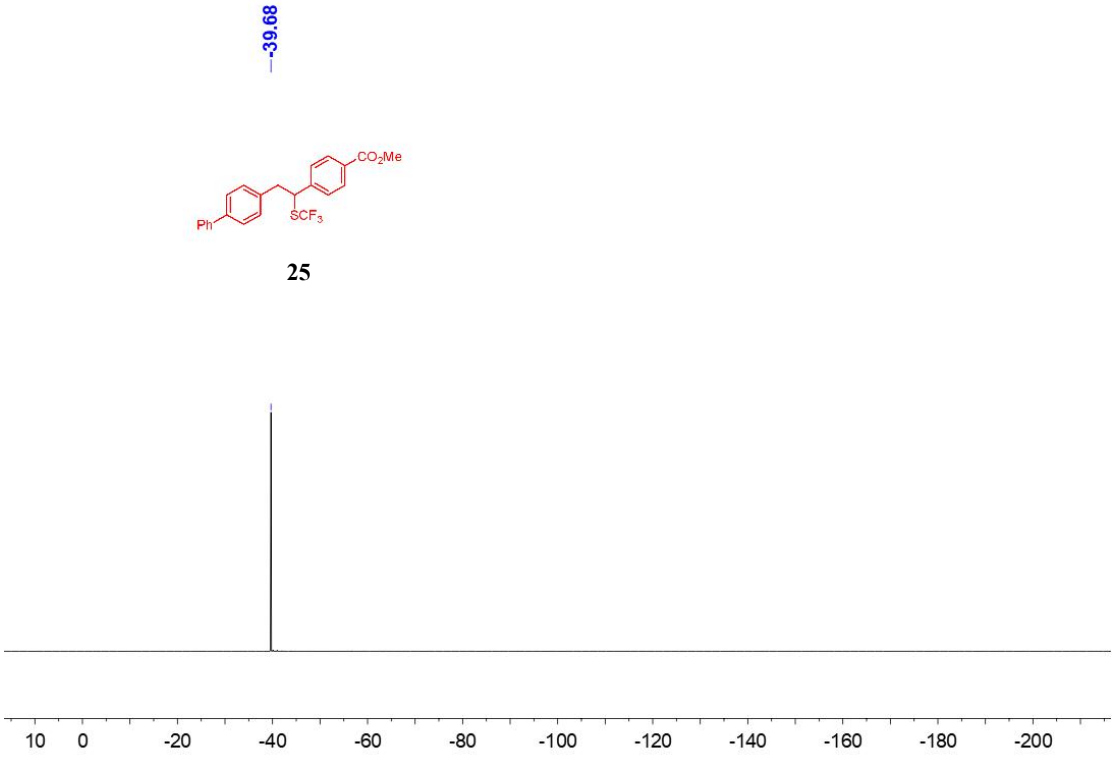

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