

Electronic Supplementary Information

Metal-Free and Site-Selective α -C-H Functionalization of Tetrahydrofuran Enabled by Photocatalytic Generation of Bromine Radical

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1. Experimental section

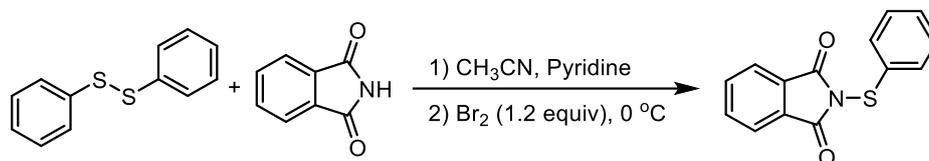
1) General information

All chemicals, unless otherwise noted, were purchased from commercial sources and were used without further purification. Unless stated otherwise, all reactions were carried out under air atmosphere. The substrates *N*-phenyl-sulfonyl phthalimides (**a**) and α -trifluoromethyl arylalkenes (**c**) were synthesized according to the literature methods with slight modification.¹⁻⁵ Irradiation with visible light was performed using blue LEDs ($\lambda = 450 \pm 10$ nm) illumination instruments (The instruments were designed by ourselves and the actual output power density of the LEDs at 0.5 cm distance is 33.70 mW/cm² detected by CEL-NP2000-10 (Beijing Ceau Light Co. Ltd., China) light power meter). For irradiation, the material of the reaction vessel is common glass; the distance from the light source is about 0.5 cm.

The nuclear magnetic resonance spectra were recorded on the Bruker Ascend™ 400 MHz NMR spectrometer with tetramethylsilane (TMS) as an internal standard. High resolution mass spectra were recorded using a Q Exactive mass spectrometer (Thermo Fisher Scientific, USA). Cyclic voltammogram experiments were measured on the CHI-Instrument CHI660E.

2) Preparation of of *N*-phenyl-sulfenyl phthalimides¹⁻³

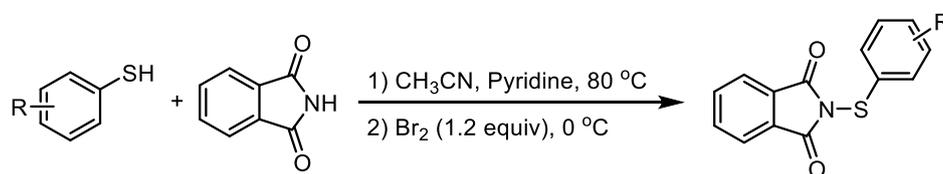
Method A:



A suspension of phthalimide (1.047 g, 10.0 mmol) and diphenyl disulfide (1.30 g, 6.0 mmol) in CH₃CN (5.0 mL) and pyridine (4.0 mL) were treated with a solution of Br₂ (615 μ L in 5.0 mL CH₃CN, 12.0 mmol, 1.2 equiv) dropwise over 30 mins. Upon complete addition of Br₂ solution, the mixture was stirred for 1 h at 0 °C, and was subsequently quenched by dropwise addition of CH₃OH (15.0 mL). Filtration of the suspension and washing of the precipitate with pre-cooled CH₃OH (0 °C, 3 \times 10.0 mL). Further purification was achieved by recrystallization in ethyl acetate through hot cooling to room temperature.

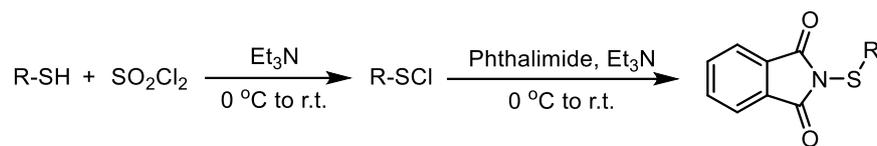
a1 was prepared according to the above procedure.

Method B:



A suspension of phthalimide (1.047 g, 10.0 mmol) and thiophenols (11.0 mmol, 1.1 equiv) in CH₃CN (5.0 mL) and pyridine (4.0 mL) was heated to 80 °C and then cooled to room temperature. The mixture was treated with a solution of Br₂ (615 μ L in 5.0 mL CH₃CN, 12.0 mmol, 1.2 equiv) dropwise over 30 mins. Upon complete addition of Br₂ solution, the mixture was stirred for 1 h at 0 °C and was subsequently quenched by dropwise addition of H₂O (15.0 mL). Filtration of the suspension and washing of the precipitate with pre-cooled CH₃OH (0 °C, 3 \times 10.0 mL). Further purification was achieved by recrystallization in ethyl acetate through hot cooling to room temperature.

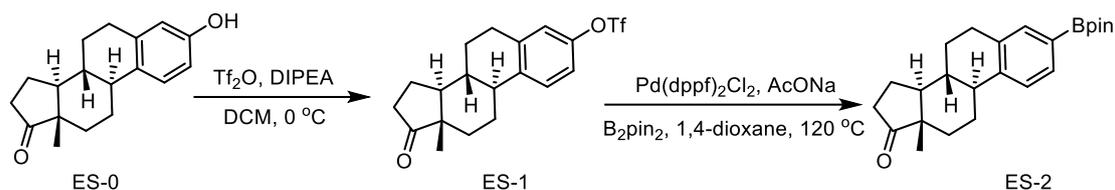
a2-a29 were prepared according to the above procedure.

Method C:

Sulfuryl chloride (5 mmol in 10 mL CH₂Cl₂) was added dropwise via a dropping funnel to a solution of thiol (1.0 equiv) in CH₂Cl₂ (1.0 M) and Et₃N (0.1 mL) at 0 °C. After stirring for 15 min, the mixture was warmed to room temperature for 30 mins and then cooled to 0 °C. The resulting solution was transferred dropwise via cannula to a solution of phthalimide (1.0 equiv) in CH₂Cl₂ (1.0 M) and Et₃N (1.3 equiv) at 0 °C and the mixture was then warmed to room temperature over 1 h. The solution was diluted with H₂O, extracted with hot ethyl acetate before being dried over Na₂SO₄, and then concentrated to give crude product that was purified by recrystallization.

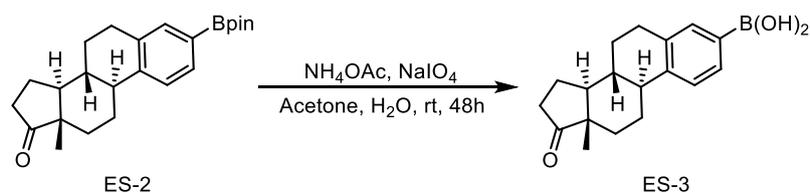
a30-a33 were prepared according to the above procedure.

3) Preparation of boronic acid of Estrone⁴



Estrone (10.0 mmol, 1.0 equiv., 2.70 g), and DIPEA (12.0 mmol, 1.2 equiv.) were dissolved in DCM (30.0 mL) in a two-neck flask with a stir bar under argon atmosphere. The reaction mixture was stirred at $0\text{ }^\circ\text{C}$, and Tf_2O (12.0 mmol, 1.2 equiv., 2.0 mL) was dropwise added into reaction system over 5 min. The reaction mixture was then allowed to warm to room temperature and stirred for 30 min. Upon completion, water (50.0 mL) was added to quench the reaction. The reaction mixture was then extracted with ethyl acetate (30.0 mL x 3). The combined organic extracts were dried with anhydrous MgSO_4 and concentrated under vacuum. The crude product was purified by flash column chromatography on silica gel to afford the corresponding trifluoromethanesulfonic ester **ES-1** (81%, 3.21 g).

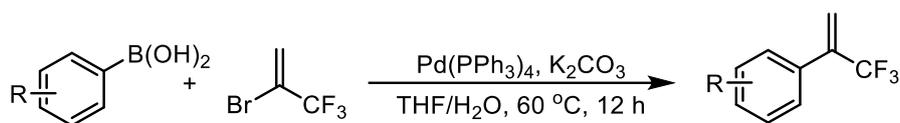
Trifluoromethanesulfonic ester **ES-1** (5.0 mmol, 1.0 equiv.), B_2pin_2 (10.0 mmol, 2.0 equiv., 2.7 g), $\text{Pd}(\text{dppf})\text{Cl}_2$ (0.5 mmol, 10 mol%, 0.367 g), and AcONa (15.0 mmol, 3.0 equiv., 1.5 g) were dissolved in 1,4-dioxane (20.0 mL) in a two-neck flask with a stir bar under argon atmosphere. The reaction mixture was stirred at $120\text{ }^\circ\text{C}$ for 8 h. Upon completion, water (50.0 mL) was added to quench the reaction. The reaction mixture was then extracted with ethyl acetate (30.0 mL x 3). The combined organic extracts were dried with anhydrous MgSO_4 and concentrated under vacuum. The desired product **ES-2** was obtained through silica gel chromatography.



ES-2 (2.0 mmol, 1.0 equiv., 0.76 g), NH_4OAc (12.0 mmol, 6.0 equiv., 0.92 g) and NaIO_4 (12.0 mmol, 6.0 equiv., 2.57 g) in acetone (50 mL) and water (20 mL) in a 200 mL round-bottom flask with a stir bar under argon atmosphere. Then the reaction

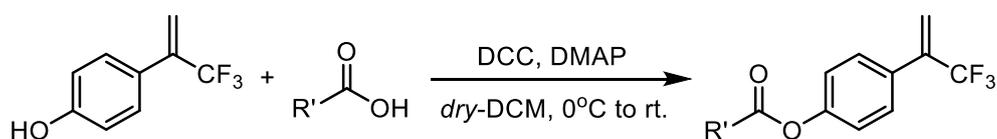
mixture was stirred at room temperature for 48 h. Upon completion, the resulting mixture was filtered through a pad of Celite. The filtrate was extracted with ethyl acetate (20.0 mL x 3). The combined organic layer was dried with anhydrous MgSO₄ and concentrated under vacuum. The corresponding boronic acid **ES-3** was obtained (57% yield) without further purification

4) Preparation of α -trifluoromethyl arylalkenes⁵



Arylboronic acid (10.0 mmol), Pd(PPh₃)₄ (0.3 mmol, 3 mol%), K₂CO₃ (2.0 M) were dissolved in THF (30.0 mL) in a two-neck flask under argon atmosphere. Then, 2-bromo-3,3,3-trifluoroprop-1-ene (20.0 mmol, 2.1 mL) was added dropwise into the mixture. The mixture was heated to 60 °C in an oil bath for at least 12 h. Then the mixed solution was extracted with ethyl acetate (3 × 15.0 mL). The organic layer was washed with brine (20.0 mL), dried over Na₂SO₄, and then concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 200/1) to afford the desired products.

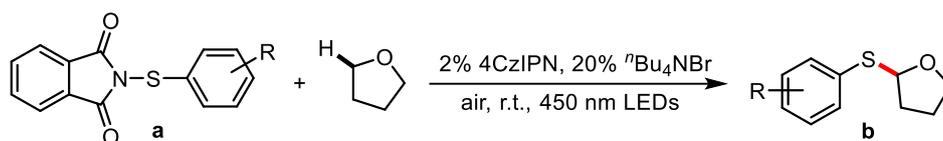
c1-c33, c39 were prepared according to the above procedure.



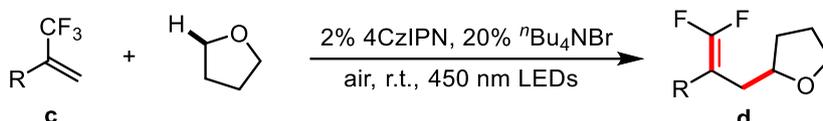
Carboxylic acid (2.2 mmol in dry DCM) was added dropwise via a dropping funnel to a solution of 4-(3,3,3-trifluoroprop-1-en-2-yl)phenol (2.0 mmol), DCC (2.2 mmol, 1.1 equiv.), DMAP (20 mol%) were dissolved in *dry*-DCM (10.0 mL) in a two-neck flask under argon atmosphere at 0 °C. After stirring for 5 min, the mixture was allowed to warm to room temperature over 3h. Upon completion, the resulting mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography to afford the desired products.

c34-c38 were prepared according to the above procedure.

5) General procedure for the photochemical reactions

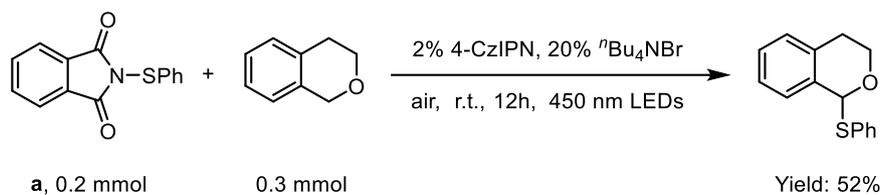


a (0.2 mmol), tetra-butyl ammonium bromide tBu₄NBr (12.9 mg, 20 mol%), 4-CzIPN (3.1 mg, 2.0 mol%) were dissolved in 2.0 mL THF in a 10.0 mL flask equipped with magnetic stirring bar, then the reaction tube was irradiated by blue LEDs ($\lambda = 450 \pm 10$ nm) at room temperature for 12 h. After reaction, the solvent was removed by rotary evaporation and purified by column-chromatography on silica gel using petroleum ether/ethyl acetate as the eluent to afford the desired product **b**.

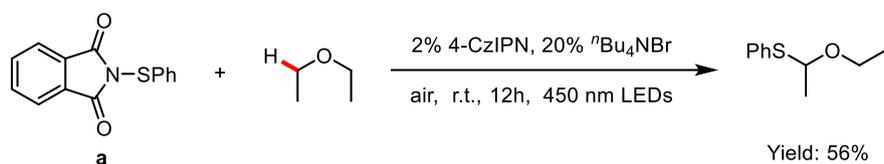


c (0.2 mmol), tetra-butyl ammonium bromide tBu₄NBr (12.9 mg, 20 mol%), 4-CzIPN (3.1 mg, 2 mol%) were dissolved in 2.0 mL THF in a 10.0 mL flask equipped with magnetic stirring bar, then the reaction tube was irradiated by blue LEDs ($\lambda = 450 \pm 10$ nm) at room temperature for 12 h. After reaction, the solvent was removed by rotary evaporation and purified by column-chromatography on silica gel using petroleum ether/ethyl acetate as the eluent to afford the desired product **d**.

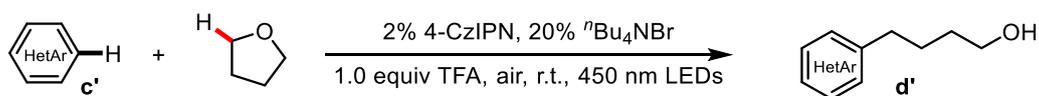




a (0.2 mmol), tetra-butyl ammonium bromide $t\text{Bu}_4\text{NBr}$ (12.9 mg, 20 mol%), 4-CzIPN (3.1 mg, 2 mol%) and isochroman (0.3 mmol) were dissolved in 2.0 mL acetone in a 10.0 mL flask equipped with magnetic stirring bar, then the reaction tube was irradiated by blue LEDs ($\lambda = 450 \pm 10 \text{ nm}$) at room temperature for 12 h. After reaction, the solvent was removed by rotary evaporation and purified by column-chromatography on silica gel using petroleum ether/ethyl acetate as the eluent to afford the desired product **b34**.



a (0.2 mmol), tetra-butyl ammonium bromide $t\text{Bu}_4\text{NBr}$ (12.9 mg, 20 mol%), 4-CzIPN (3.1 mg, 2 mol%) were dissolved in 2.0 mL Diethyl ether in a 10.0 mL flask equipped with magnetic stirring bar, then the reaction tube was irradiated by blue LEDs ($\lambda = 450 \pm 10 \text{ nm}$) at room temperature for 12 h. After reaction, the solvent was removed by rotary evaporation and purified by column-chromatography on silica gel using petroleum ether/ethyl acetate as the eluent to afford the desired product **b35**.



c' (0.2 mmol), tetra-butyl ammonium bromide $t\text{Bu}_4\text{NBr}$ (12.9 mg, 20 mol%), 4-CzIPN (3.1 mg, 2 mol%) and Trifluoroacetic acid (1.0 equiv., 20 μL) were dissolved in 2.0 mL THF in a 10.0 mL flask equipped with magnetic stirring bar, then the reaction tube was irradiated by blue LEDs ($\lambda = 450 \pm 10 \text{ nm}$) at room temperature for 12 h. After reaction, the solvent was removed by rotary evaporation and purified by column-chromatography on silica gel using hexane/ethyl acetate as the eluent to afford the desired product **d'**.

6) Crystal structure determination of **b15**

A suitable crystal of **b15** was mounted with glue at the end of a glass fiber. Data collection for **b15** was performed on a Rigaku OD (Enhance Cu X-ray Source, $K\alpha$, $\lambda = 1.54184 \text{ \AA}$) with CCD Plate (XtaLAB Pro: Kappa single) under 293 K. Data were processed with the CrysAlisPro 1.171.39.7e (Rigaku Oxford Diffraction, 2015).

Structure was solved by ShelXT⁶ in Olex2 1.5⁷ and refined on F^2 using full-matrix least-squares (SHELXL-2018 in Olex2 1.5). Anisotropic thermal parameters were applied to all non-hydrogen atoms. The hydrogen atoms were generated geometrically. Crystal data and structure refinement parameters are summarized in Table S1. CCDC No. **2161362**.

Single crystals of **b15** were prepared in acetonitrile solution of **b15**. Colourless block crystals formed.

Table S1 Crystal data and structure refinements for **b15**

Compound	b15
Formula	C ₁₆ H ₁₆ O ₈
Formula weight	256.35
Temperature (K)	293(2) K
Crystal system	Monoclinic
Space group	$P2_1/n$
$a/\text{\AA}$	6.0752(3)
$b/\text{\AA}$	9.8777(5)
$c/\text{\AA}$	22.1887(13)
$\beta/^\circ$	91.536(5)
$V/\text{\AA}^3$	1331.04(12)
Z	4
$D_c/\text{g cm}^{-3}$	1.279
reflns coll.	5767

unique reflns	2471
R_{int}	0.0235
$R_1 [I > 2\sigma(I)]$	0.0538
$wR_2 [I > 2\sigma(I)]$	0.1380
R_1 (all data)	0.0621
wR_2 (all data)	0.1429
GOF	1.069

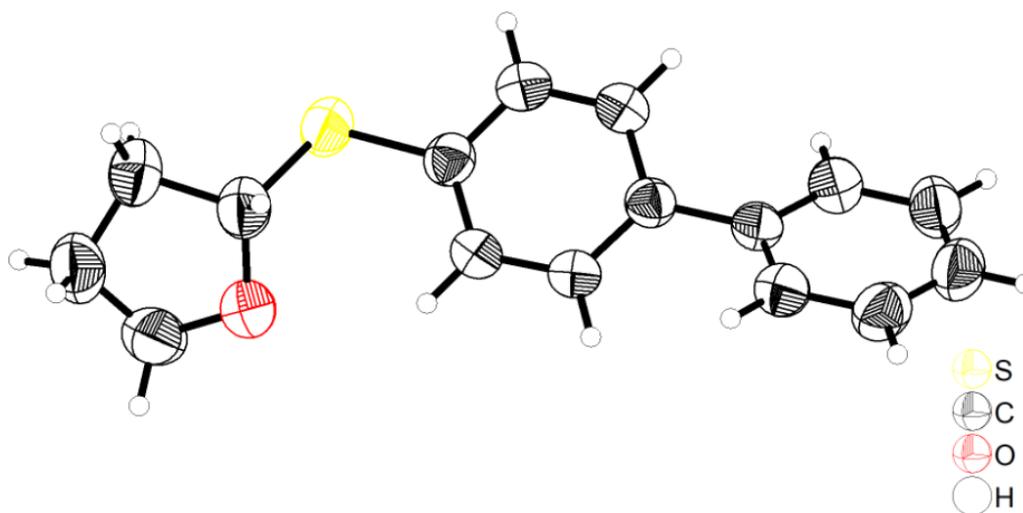


Figure S1. Molecular structure of **b15** with 50% thermal ellipsoid.

CCDC Number: 2161362

7) Luminescence quenching experiments

General procedure: The luminescence quenching experiments were measured with excitation at 450 nm. A THF solution of 1×10^{-4} M 4-CzIPN and 1.0×10^{-1} M **a1** or ${}^n\text{Bu}_4\text{NBr}$ respectively were prepared. The experiments were conducted in 1.25 cm x 1.25 cm x 4.5 cm quartz cuvette at room temperature. Appropriate volume (the whole solution volume change < 5%) of the quencher **a1** or ${}^n\text{Bu}_4\text{NBr}$ was respectively injected to the THF solution (3.0 mL) of 1×10^{-4} M 4-CzIPN in the quartz cuvette by microsyringe.

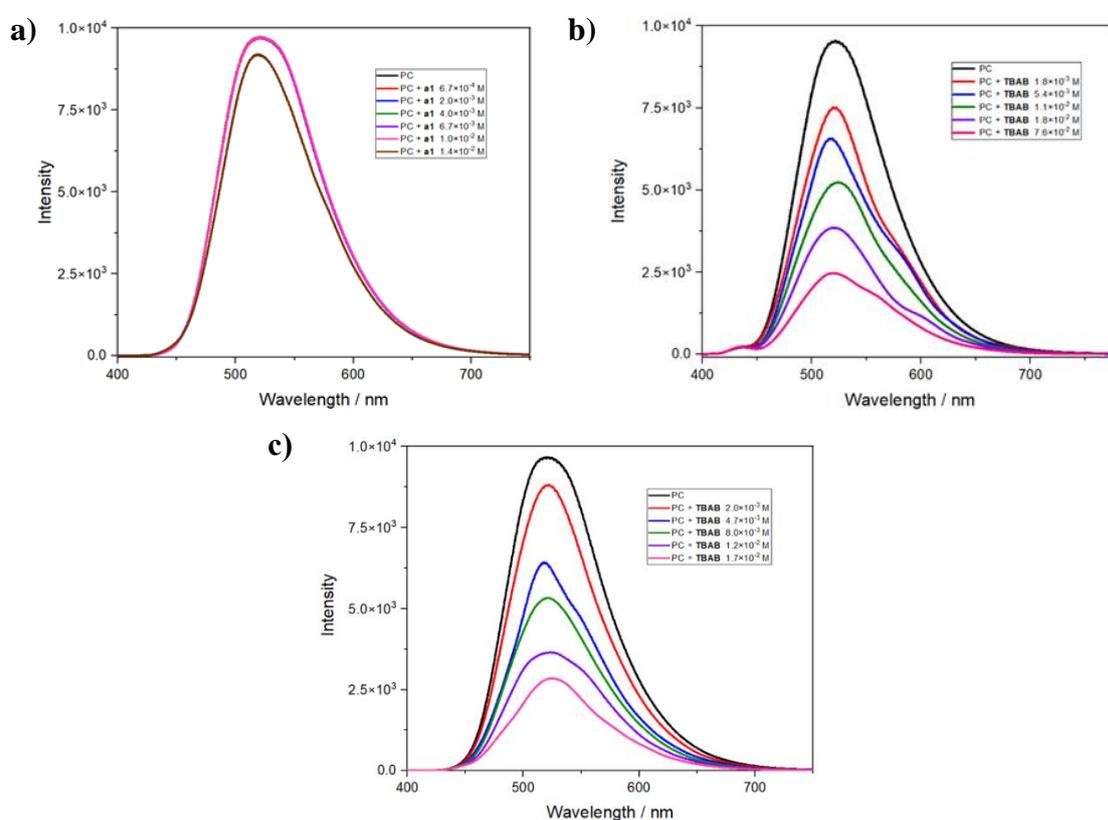


Figure S2. Luminescence quenching spectra of 4-CzIPN (1.0×10^{-4} M) **a)** by various concentration of **a1**; **b)** by various concentration of ${}^n\text{Bu}_4\text{NBr}$ under argon atmosphere; **c)** by various concentration of ${}^n\text{Bu}_4\text{NBr}$ under air atmosphere with excitation at 450 nm.

8) Radical inhibition experiment

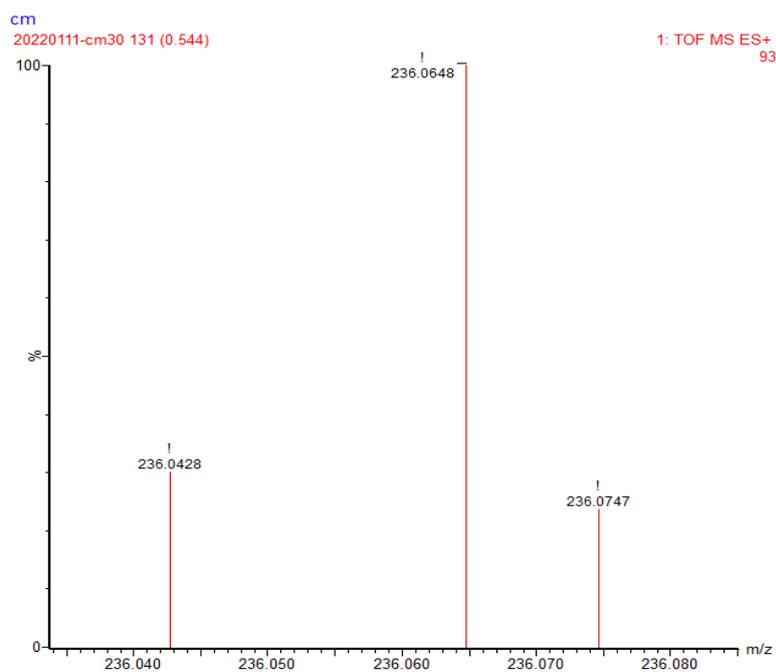
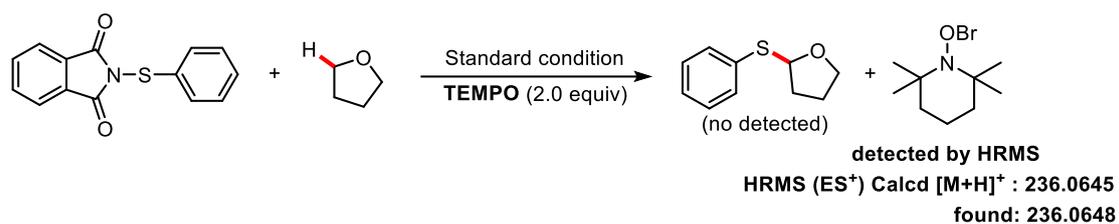
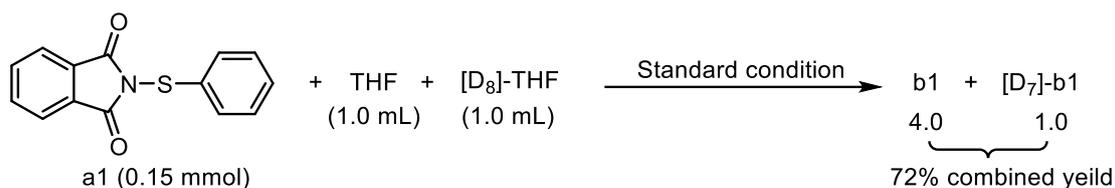


Figure S3. HRMS spectra for Radical inhibition experiment.

9) Kinetic isotope effect experiment



a1 (0.15 mmol), ⁿBu₄NBr (9.7 mg, 20 mol%), 4-CzIPN (2.4 mg, 2.0 mol%) were dissolved in THF/D₈-THF (1.0 mL/1.0 mL) in a 10.0 mL flask equipped with magnetic stirring bar, then the reaction tube was irradiated by blue LEDs ($\lambda = 450 \pm 10$ nm) at room temperature for 8 h. After reaction, the solvent was removed by rotary evaporation and purified by column-chromatography on silica gel using petroleum ether/ethyl acetate as the eluent to afford the desired product **b1**.

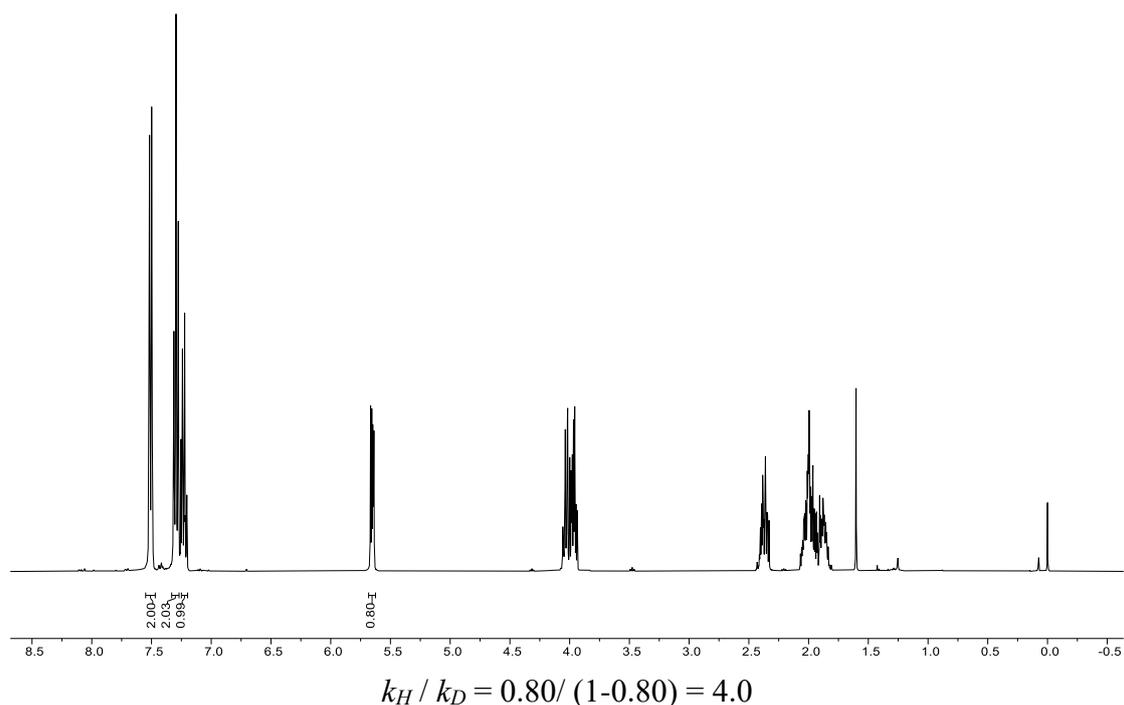
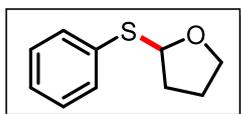


Figure S4. ¹H NMR spectra for kinetic isotope effect experiment.

10) References

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2. Characterization data of the products



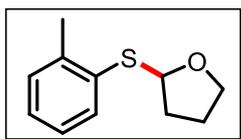
b1

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b1** as a colorless oil (33.5 mg, 93% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.50 (d, $J = 7.1$ Hz, 2H), 7.28 (t, $J = 7.4$ Hz, 2H), 7.20 (t, $J = 7.4$ Hz, 1H), 5.64 (dd, $J = 7.2, 3.8$ Hz, 1H), 4.04 – 3.91 (m, 2H), 2.39 – 2.29 (m, 1H), 2.04 – 1.92 (m, 2H), 1.90 – 1.79 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 135.8, 131.0, 128.8, 126.8, 87.1, 67.3, 32.7, 24.9.

HRMS (EI) calculated for $\text{C}_{10}\text{H}_{12}\text{OS}$ $[\text{M}]^+$: 180.0604; Found: 180.0605



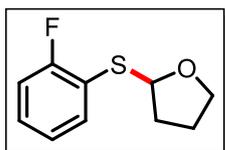
b2

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b2** as a colorless oil (30.3 mg, 78% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.60 (d, $J = 7.4$ Hz, 1H), 7.16 (d, $J = 6.3$ Hz, 1H), 7.14 (d, $J = 4.7$ Hz, 1H), 7.13 – 7.09 (m, 1H), 5.63 (dd, $J = 7.2, 3.7$ Hz, 1H), 4.04 – 3.91 (m, 2H), 2.38 (s, 3H), 2.37 – 2.30 (m, 1H), 2.06 – 1.97 (m, 2H), 1.91 – 1.82 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 138.4, 135.2, 130.8, 130.0, 126.7, 126.5, 86.4, 67.4, 32.8, 24.9, 20.8.

HRMS (ESI) calculated for $\text{C}_{11}\text{H}_{14}\text{OS}$ $[\text{M}+\text{H}]^+$: 195.0839; Found: 195.0841



b3

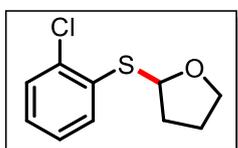
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b3** as a pale-yellow oil (33.3 mg, 84% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.60 (td, *J* = 7.6, 1.8 Hz, 1H), 7.29 – 7.19 (m, 1H), 7.13 – 7.03 (m, 2H), 5.70 (dd, *J* = 7.1, 3.4 Hz, 1H), 4.07 – 3.93 (m, 2H), 2.42 – 2.33 (m, 1H), 2.12 – 1.97 (m, 2H), 1.93 – 1.84 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 161.5 (d, *J* = 245.9 Hz), 133.9, 129.0 (d, *J* = 7.8 Hz), 124.5 (d, *J* = 3.8 Hz), 122.3 (d, *J* = 17.9 Hz), 115.6 (d, *J* = 22.9 Hz), 86.4 (d, *J* = 7.8 Hz), 67.37, 32.70, 24.62.

¹⁹F NMR (376 MHz, CDCl₃) δ -109.35 (s, 1F).

HRMS (ESI) calculated for C₁₀H₁₁FOS [M+H]⁺: 199.0588; Found: 199.0592



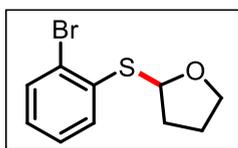
b4

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b4** as a pale-yellow oil (37.7 mg, 88% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.68 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.35 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.22 (td, *J* = 7.6, 1.5 Hz, 1H), 7.12 (td, *J* = 7.6, 1.6 Hz, 1H), 5.75 (dd, *J* = 7.2, 3.6 Hz, 1H), 4.04 – 3.94 (m, 2H), 2.46 – 2.37 (m, 1H), 2.12 – 2.00 (m, 2H), 1.94 – 1.85 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 135.5, 133.9, 130.7, 129.5, 127.2, 127.2, 85.5, 67.5, 32.6, 24.8.

HRMS (ESI) calculated for C₁₀H₁₁ClOS [M+H]⁺: 215.0292; Found: 215.0297



b5

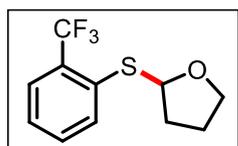
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b5** as a pale-yellow oil (42.8 mg, 83% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.70 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.55 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.29 (td, *J* = 7.6, 1.3 Hz, 1H), 7.06 (td, *J* = 7.7, 1.6 Hz, 1H), 5.78 (dd, *J* = 7.3,

3.6 Hz, 1H), 4.11 – 3.94 (m, 2H), 2.52 – 2.38 (m, 1H), 2.15 – 2.00 (m, 2H), 2.01 – 1.84 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 137.7, 132.8, 130.3, 127.9, 127.2, 123.9, 85.6, 67.5, 32.5, 24.8.

HRMS (ESI) calculated for C₁₀H₁₁BrOS [M+H]⁺: 258.9787; Found: 258.9783



b6

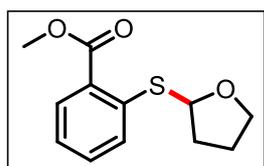
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b6** as a colorless oil (42.2 mg, 85% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.88 (d, *J* = 7.9 Hz, 1H), 7.66 (d, *J* = 7.9 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 1H), 7.32 (t, *J* = 7.7 Hz, 1H), 5.71 (dd, *J* = 7.2, 3.5 Hz, 1H), 4.10 – 3.96 (m, 2H), 2.48 – 2.36 (m, 1H), 2.13 – 2.01 (m, 2H), 1.97 – 1.86 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 135.6 (d, *J* = 1.8 Hz), 133.1 (d, *J* = 3.7 Hz), 132.0 (d, *J* = 1.9 Hz), 126.5 (q, *J* = 275.2 Hz), 126.4, 125.2, 122.5, 87.1, 67.4, 32.7, 24.7.

¹⁹F NMR (376 MHz, CDCl₃) δ -60.62 (s, 3F).

HRMS (ESI) calculated for C₁₁H₁₁F₃OS [M+H]⁺: 249.0556; Found: 249.0559



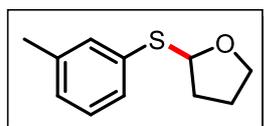
b7

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b7** as a colorless oil (38.6 mg, 81% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.92 (d, *J* = 8.7 Hz, 1H), 7.86 (d, *J* = 8.1 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 5.75 (dd, *J* = 7.4, 3.9 Hz, 1H), 4.04 – 3.94 (m, 2H), 3.89 (s, 3H), 2.47 – 2.38 (m, 1H), 2.13 – 2.03 (m, 2H), 1.96 – 1.86 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 167.0, 141.1, 132.4, 130.9, 128.0, 124.6, 122.9, 84.5, 67.6, 52.1, 32.4, 25.1.

HRMS (ESI) calculated for C₁₂H₁₄O₃S [M+H]⁺: 239.0737; Found: 239.0731



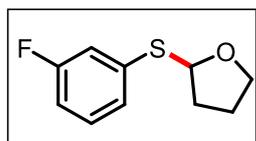
b8

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b8** as a colorless oil (32.2 mg, 83% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.32 (s, 1H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.03 (d, *J* = 7.8 Hz, 1H), 5.64 (dd, *J* = 7.2, 3.9 Hz, 1H), 4.03 – 3.93 (m, 2H), 2.40 – 2.34 (m, 1H), 2.32 (s, 3H), 2.05 – 1.94 (m, 2H), 1.90 – 1.82 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 138.6, 135.4, 131.6, 128.7, 128.0, 127.7, 87.1, 67.3, 32.7, 24.9, 21.4.

HRMS (ESI) calculated for C₁₁H₁₄OS [M+H]⁺: 195.0839; Found: 195.0840



b9

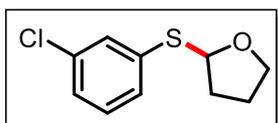
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b9** as a colorless oil (30.9 mg, 77% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.25 (d, *J* = 2.9 Hz, 1H), 7.24 (d, *J* = 3.1 Hz, 1H), 7.23 (d, *J* = 5.7 Hz, 1H), 6.94 – 6.85 (m, 1H), 5.68 (dd, *J* = 7.3, 3.9 Hz, 1H), 4.04 – 3.93 (m, 2H), 2.44 – 2.32 (m, 1H), 2.08 – 1.94 (m, 2H), 1.93 – 1.84 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 162.7 (d, *J* = 248.8 Hz), 138.3 (d, *J* = 3.0 Hz), 130.0 (d, *J* = 8.7 Hz), 125.9 (d, *J* = 3.2 Hz), 117.2 (d, *J* = 22.4 Hz), 113.5 (d, *J* = 21.2 Hz), 86.8, 67.4, 32.6, 24.8.

¹⁹F NMR (376 MHz, CDCl₃) δ -112.37 (s, 1F).

HRMS (ESI) calculated for C₁₀H₁₁FOS [M+H]⁺: 199.0588; Found: 199.0581



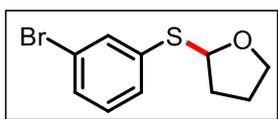
b10

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b10** as a pale-yellow oil (38.1 mg, 89% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.56 – 7.50 (m, 1H), 7.38 (dt, $J = 6.9, 1.9$ Hz, 1H), 7.25 – 7.21 (m, 1H), 7.20 – 7.18 (m, 1H), 5.68 (dd, $J = 7.3, 3.8$ Hz, 1H), 4.05 – 3.96 (m, 2H), 2.44 – 2.34 (m, 1H), 2.09 – 1.95 (m, 2H), 1.94 – 1.85 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 138.0, 134.5, 130.2, 129.8, 128.6, 126.7, 86.9, 67.4, 32.6, 24.8.

HRMS (ESI) calculated for $\text{C}_{10}\text{H}_{11}\text{ClOS}$ $[\text{M}+\text{H}]^+$: 215.0292; Found: 215.0297



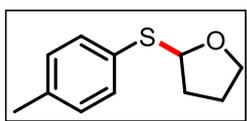
b11

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b11** as a pale-yellow oil (41.3 mg, 80% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.66 (t, $J = 1.8$ Hz, 1H), 7.41 (d, $J = 8.5$ Hz, 1H), 7.34 (d, $J = 8.9$ Hz, 1H), 7.14 (t, $J = 7.9$ Hz, 1H), 5.66 (dd, $J = 7.3, 3.8$ Hz, 1H), 4.03 – 3.95 (m, 2H), 2.41 – 2.33 (m, 1H), 2.08 – 1.93 (m, 2H), 1.93 – 1.83 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 138.3, 133.0, 130.1, 129.7, 129.1, 122.6, 86.9, 67.4, 32.6, 24.8.

HRMS (ESI) calculated for $\text{C}_{10}\text{H}_{11}\text{BrOS}$ $[\text{M}+\text{H}]^+$: 258.9787; Found: 258.9778



b12

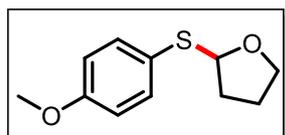
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 30/1, v/v) afforded **b12** as a colorless oil (32.6 mg, 84% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.40 (d, $J = 8.2$ Hz, 2H), 7.10 (d, $J = 7.9$ Hz, 2H), 5.57

(dd, $J = 7.2, 3.8$ Hz, 1H), 4.05 – 3.90 (m, 2H), 2.38 – 2.32 (m, 1H), 2.31 (s, 3H), 2.05 – 1.92 (m, 2H), 1.91 – 1.82 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 137.0, 131.9, 131.8, 129.6, 87.6, 67.2, 33.6, 24.8, 21.1.

HRMS (ESI) calculated for $\text{C}_{11}\text{H}_{14}\text{OS}$ $[\text{M}+\text{H}]^+$: 195.0838; Found: 195.0843



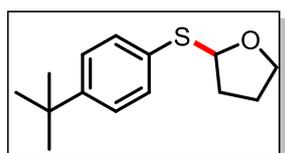
b13

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 30/1, v/v) afforded **b13** as a colorless oil (36.1 mg, 86% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.45 (d, $J = 8.8$ Hz, 2H), 6.84 (d, $J = 8.8$ Hz, 2H), 5.46 (dd, $J = 7.2, 3.7$ Hz, 1H), 4.06 – 3.96 (m, 1H), 3.92 (td, $J = 8.1, 4.1$ Hz, 1H), 3.78 (s, 3H), 2.34 – 2.26 (m, 1H), 2.02 – 1.92 (m, 2H), 1.88 – 1.79 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 159.4, 134.6, 125.6, 114.4, 88.2, 67.2, 55.3, 32.5, 24.8.

HRMS (ESI) calculated for $\text{C}_{11}\text{H}_{14}\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$: 211.0788; Found: 211.0786



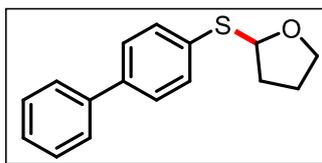
b14

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b14** as a colorless oil (42.5 mg, 90% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.44 (d, $J = 8.4$ Hz, 2H), 7.32 (d, $J = 8.4$ Hz, 2H), 5.61 (dd, $J = 7.2, 3.9$ Hz, 1H), 4.05 – 3.91 (m, 2H), 2.40 – 2.28 (m, 1H), 2.03 – 1.91 (m, 2H), 1.91 – 1.79 (m, 1H), 1.29 (s, 9H).

^{13}C NMR (101 MHz, CDCl_3) δ 150.2, 132.0, 131.4, 126.0, 87.5, 67.3, 34.6, 32.7, 31.4, 25.0.

HRMS (ESI) calculated for $\text{C}_{14}\text{H}_{20}\text{OS}$ $[\text{M}+\text{H}]^+$: 237.1308; Found: 237.1312



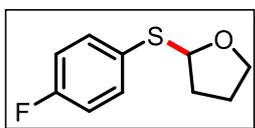
b15

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b15** as a white solid (35.3 mg, 69% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.57 (dd, $J = 3.9, 2.4$ Hz, 2H), 7.56 (d, $J = 3.2$ Hz, 2H), 7.52 (d, $J = 8.5$ Hz, 2H), 7.42 (t, $J = 7.5$ Hz, 2H), 7.33 (t, $J = 7.4$ Hz, 1H), 5.69 (dd, $J = 7.2, 3.8$ Hz, 1H), 4.08 – 3.95 (m, 2H), 2.43 – 2.34 (m, 1H), 2.08 – 1.96 (m, 2H), 1.95 – 1.85 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 140.6, 139.8, 134.8, 131.4, 128.8, 127.5, 127.4, 127.0, 87.2, 67.4, 32.7, 24.9.

HRMS (ESI) calculated for $\text{C}_{16}\text{H}_{16}\text{OS}$ $[\text{M}+\text{H}]^+$: 257.0995; Found: 257.0999



b16

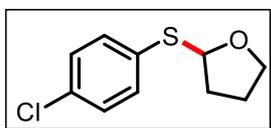
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b16** as a pale-yellow oil (36.4 mg, 92% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.49 (dd, $J = 8.8, 5.3$ Hz, 2H), 7.00 (t, $J = 8.7$ Hz, 2H), 5.54 (dd, $J = 7.2, 3.8$ Hz, 1H), 4.08 – 3.89 (m, 2H), 2.41 – 2.28 (m, 1H), 2.06 – 1.92 (m, 2H), 1.91 – 1.82 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 162.3 (d, $J = 247.8$ Hz), 134.0 (d, $J = 8.2$ Hz), 130.4 (d, $J = 3.4$ Hz), 115.8 (d, $J = 21.7$ Hz), 87.8, 67.2, 32.5, 24.8.

$^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -114.81 (s, 1F).

HRMS (ESI) calculated for $\text{C}_{10}\text{H}_{11}\text{FOS}$ $[\text{M}+\text{H}]^+$: 199.0588; Found: 199.0588



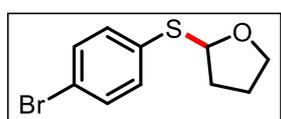
b17

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b17** as a pale-yellow oil (39.4 mg, 92% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.49 (dd, $J = 8.8, 5.3$ Hz, 2H), 6.99 (t, $J = 8.7$ Hz, 2H), 5.53 (dd, $J = 7.2, 3.8$ Hz, 1H), 4.04 – 3.89 (m, 2H), 2.40 – 2.27 (m, 1H), 2.06 – 1.92 (m, 2H), 1.90 – 1.80 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 161.1, 134.0, 130.5, 116.0, 87.8, 67.2, 32.6, 24.8.

HRMS (ESI) calculated for $\text{C}_{10}\text{H}_{11}\text{ClOS}$ $[\text{M}+\text{H}]^+$: 215.0292; Found: 215.0300



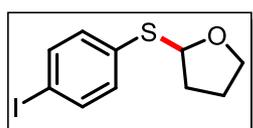
b18

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b18** as a pale-yellow oil (47.0 mg, 91% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.40 (d, $J = 8.7$ Hz, 2H), 7.36 (d, $J = 8.7$ Hz, 2H), 5.60 (dd, $J = 7.2, 3.8$ Hz, 1H), 4.03 – 3.92 (m, 2H), 2.42 – 2.29 (m, 1H), 2.06 – 1.93 (m, 2H), 1.92 – 1.83 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 135.0, 132.6, 131.8, 120.9, 87.1, 67.3, 32.6, 24.8.

HRMS (ESI) calculated for $\text{C}_{10}\text{H}_{11}\text{BrOS}$ $[\text{M}+\text{H}]^+$: 258.9787; Found: 258.9785



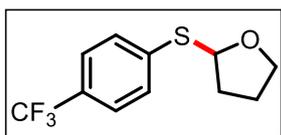
b19

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b19** as a yellow oil (54.5 mg, 89% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.58 (d, $J = 8.4$ Hz, 2H), 7.22 (d, $J = 8.4$ Hz, 2H), 5.61 (dd, $J = 7.3, 3.8$ Hz, 1H), 4.02 – 3.92 (m, 2H), 2.40 – 2.30 (m, 1H), 2.05 – 1.92 (m, 2H), 1.90 – 1.82 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 137.6, 136.0, 132.6, 92.1, 87.0, 67.4, 32.6, 24.8.

HRMS (ESI) calculated for $\text{C}_{10}\text{H}_{11}\text{IOS}$ $[\text{M}+\text{H}]^+$: 306.9649; Found: 306.9439



b20

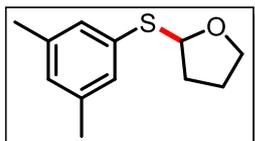
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b20** as a colorless oil (43.6 mg, 88% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.58 (d, $J = 8.2$ Hz, 2H), 7.52 (d, $J = 8.3$ Hz, 2H), 5.75 (dd, $J = 7.3, 3.8$ Hz, 1H), 4.08 – 3.95 (m, 2H), 2.42 (tdd, $J = 12.0, 5.2, 3.4$ Hz, 1H), 2.10 – 1.97 (m, 2H), 1.96 – 1.86 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 141.48 (d, $J = 1.5$ Hz), 129.49, 128.2 (d, $J = 32.5$ Hz), 125.5 (q, $J = 3.8$ Hz), 122.8, 86.2, 67.4, 32.6, 24.8.

$^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -62.49 (s, 3F).

HRMS (ESI) calculated for $\text{C}_{11}\text{H}_{11}\text{F}_3\text{OS}$ $[\text{M}+\text{H}]^+$: 249.0556; Found: 249.0552



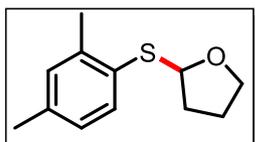
b22

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b22** as a colorless oil (36.2 mg, 87% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.12 (s, 2H), 6.85 (s, 1H), 5.65 (dd, $J = 7.3, 4.0$ Hz, 1H), 4.05 – 3.92 (m, 2H), 2.40 – 2.32 (m, 1H), 2.28 (s, 6H), 2.05 – 1.93 (m, 2H), 1.91 – 1.82 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 138.4, 135.2, 128.7, 128.5, 87.1, 67.3, 32.7, 24.9, 21.3.

HRMS (ESI) calculated for $\text{C}_{12}\text{H}_{16}\text{OS}$ $[\text{M}+\text{H}]^+$: 209.0995; Found: 209.0995



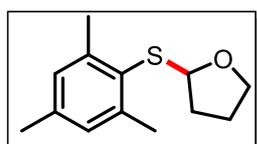
b23

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b23** as a colorless oil (34.1 mg, 82% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.48 (d, *J* = 7.9 Hz, 1H), 7.01 (s, 1H), 6.97 (d, *J* = 8.5 Hz, 1H), 5.59 (dd, *J* = 7.1, 3.6 Hz, 1H), 4.08 – 3.94 (m, 2H), 2.37 (s, 3H), 2.36 – 2.30 (m, 1H), 2.28 (s, 3H), 2.08 – 1.95 (m, 2H), 1.91 – 1.80 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 139.1, 137.0, 132.2, 131.2, 131.0, 127.3, 86.9, 67.3, 32.7, 24.9, 21.0, 20.8.

HRMS (ESI) calculated for C₁₂H₁₆OS [M+H]⁺: 209.0995; Found: 209.1002



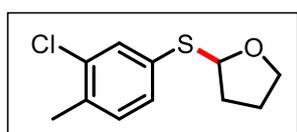
b24

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b24** as a colorless oil (23.1 mg, 52% yield).

¹H NMR (400 MHz, CDCl₃) δ 6.96 (s, 2H), 5.40 – 5.31 (m, 1H), 4.06 – 3.87 (m, 2H), 2.53 (s, 6H), 2.37 – 2.29 (m, 1H), 2.27 (s, 3H), 2.13 – 1.97 (m, 2H), 1.90 – 1.82 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 143.2, 138.4, 129.1, 129.0, 88.1, 67.3, 32.9, 25.0, 22.3, 21.1.

HRMS (ESI) calculated for C₁₃H₁₈OS [M+H]⁺: 223.1152; Found: 223.1159



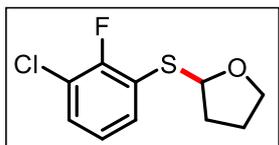
b25

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b25** as a colorless oil (28.3 mg, 65% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.53 (d, *J* = 1.9 Hz, 1H), 7.31 (dd, *J* = 7.9, 1.9 Hz, 1H), 7.16 (d, *J* = 7.2 Hz, 1H), 5.62 (dd, *J* = 7.2, 3.8 Hz, 1H), 4.07 – 3.95 (m, 2H), 2.43 – 2.36 (m, 1H), 2.35 (s, 3H), 2.08 – 1.95 (m, 2H), 1.94 – 1.86 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 134.8, 134.5, 134.2, 131.6, 131.1, 129.7, 87.4, 67.3, 32.6, 24.8, 19.7.

HRMS (ESI) calculated for C₁₁H₁₃ClOS [M+H]⁺: 229.0449; Found: 229.0439



b26

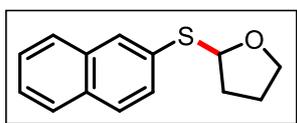
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b26** as a colorless oil (29.2 mg, 63% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.50 (ddd, $J = 8.0, 6.4, 1.7$ Hz, 1H), 7.28 (ddd, $J = 8.2, 6.8, 1.6$ Hz, 1H), 7.03 (td, $J = 8.0, 1.2$ Hz, 1H), 5.71 (dd, $J = 7.2, 3.4$ Hz, 1H), 4.06 – 3.93 (m, 2H), 2.43 – 2.34 (m, 1H), 2.12 – 1.98 (m, 2H), 1.96 – 1.86 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 156.7 (d, $J = 247.2$ Hz), 131.6, 124.8, 124.6 (q, $J = 4.5, 3.8$ Hz), 121.4 (d, $J = 19.2$ Hz), 86.4, 67.4, 32.7, 24.6.

$^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -110.73 (s, 1F).

HRMS (ESI) calculated for $\text{C}_{13}\text{H}_{18}\text{OS}$ $[\text{M}+\text{H}]^+$: 233.0198; Found: 233.0190



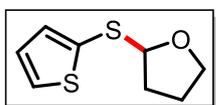
b27

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b27** as a pale-yellow oil (28.1 mg, 61% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.01 (s, 1H), 7.81 (d, $J = 6.8$ Hz, 2H), 7.78 (d, $J = 8.5$ Hz, 1H), 7.58 (dd, $J = 8.6, 1.8$ Hz, 1H), 7.51 – 7.43 (m, 2H), 5.78 (dd, $J = 7.2, 3.8$ Hz, 1H), 4.12 – 3.98 (m, 2H), 2.49 – 2.37 (m, 1H), 2.11 – 2.00 (m, 2H), 1.98 – 1.87 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 133.7, 133.2, 132.2, 129.4, 128.7, 128.2, 127.7, 127.4, 126.4, 125.8, 87.2, 67.4, 32.7, 24.9.

HRMS (ESI) calculated for $\text{C}_{14}\text{H}_{14}\text{OS}$ $[\text{M}+\text{H}]^+$: 231.0839; Found: 231.0833



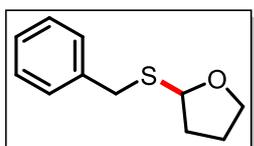
b28

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b28** as a colorless oil (17.5 mg, 47% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.37 (d, $J = 5.4$ Hz, 1H), 7.17 (d, $J = 3.5$ Hz, 1H), 7.00 (dd, $J = 5.3, 3.6$ Hz, 1H), 5.43 (dd, $J = 7.2, 3.0$ Hz, 1H), 4.07 – 3.92 (m, 2H), 2.34 – 2.25 (m, 1H), 2.04 – 1.92 (m, 2H), 1.93 – 1.81 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 134.3, 132.4, 129.9, 127.5, 89.5, 67.5, 32.1, 24.6.

HRMS (ESI) calculated for $\text{C}_8\text{H}_{10}\text{OS}_2$ $[\text{M}+\text{H}]^+$: 187.0246; Found: 187.0247



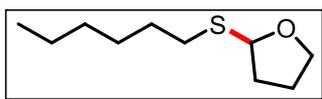
b29

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b29** as a colorless oil (28.3 mg, 73% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.35 (d, $J = 7.6$ Hz, 2H), 7.30 (d, $J = 7.7$ Hz, 2H), 7.26 (s, 1H), 5.22 (dd, $J = 7.3, 3.6$ Hz, 1H), 4.01 – 3.92 (m, 2H), 3.91 – 3.73 (m, 2H), 2.23 – 2.15 (m, 1H), 2.05 – 1.95 (m, 2H), 1.87 – 1.75 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 138.6, 129.0, 128.6, 126.9, 83.0, 66.8, 36.0, 31.0, 26.2.

HRMS (ESI) calculated for $\text{C}_{11}\text{H}_{14}\text{OS}$ $[\text{M}+\text{H}]^+$: 195.0839; Found: 195.0833



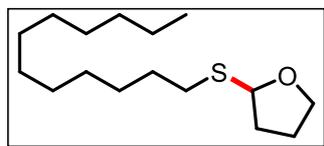
b30

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b30** as a colorless oil (26.3 mg, 70% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 5.36 (dd, $J = 7.3, 3.9$ Hz, 1H), 3.98 – 3.86 (m, 2H), 2.73 – 2.54 (m, 2H), 2.32 – 2.22 (m, 1H), 2.04 – 1.94 (m, 1H), 1.89 – 1.78 (m, 2H), 1.67 – 1.58 (m, 2H), 1.42 – 1.35 (m, 2H), 1.33 – 1.26 (m, 4H), 0.88 (t, $J = 6.8$ Hz, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 84.5, 66.8, 32.7, 31.5, 31.3, 30.0, 28.8, 24.9, 22.6, 14.1.

HRMS (ESI) calculated for C₁₀H₂₀OS [M+H]⁺: 189.1308; Found: 189.1308



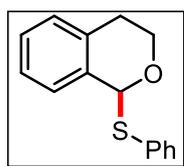
b31

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b31** as a colorless oil (29.9 mg, 55% yield).

¹H NMR (400 MHz, CDCl₃) δ 5.36 (dd, *J* = 7.3, 3.9 Hz, 1H), 3.97 – 3.86 (m, 2H), 2.74 – 2.53 (m, 2H), 2.33 – 2.20 (m, 1H), 2.03 – 1.94 (m, 1H), 1.88 – 1.78 (m, 2H), 1.67 – 1.59 (m, 2H), 1.41 – 1.33 (m, 2H), 1.31 – 1.23 (m, 16H), 0.92 – 0.82 (t, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 84.4, 66.7, 32.6, 31.9, 31.2, 30.0, 29.65, 29.63, 29.60, 29.5, 29.3, 29.2, 29.0, 24.8, 22.3, 14.1.

HRMS (ESI) calculated for C₁₆H₃₂OS [M+H]⁺: 273.2247; Found: 273.2238



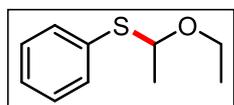
b34

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b34** as a colorless oil (25.2 mg, 52% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.60 (d, *J* = 6.9 Hz, 2H), 7.35 (d, *J* = 7.0 Hz, 2H), 7.32 (s, 1H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.24 – 7.16 (m, 2H), 7.12 (dd, *J* = 5.4, 3.6 Hz, 1H), 6.49 (s, 1H), 4.55 (td, *J* = 11.6, 3.2 Hz, 1H), 4.01 (dd, *J* = 10.9, 5.7 Hz, 1H), 3.11 (ddd, *J* = 16.7, 12.3, 6.3 Hz, 1H), 2.70 (dd, *J* = 16.0, 3.0 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 136.0, 133.9, 133.8, 131.2, 128.9, 128.8, 127.7, 127.2, 127.1, 126.1, 85.9, 58.2, 27.8.

HRMS (ESI) calculated for C₁₅H₁₄OS [M+H]⁺: 243.0839; Found: 243.0844



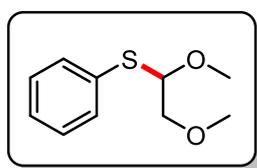
b35

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **b35** as a colorless oil (20.4 mg, 56% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.48 (d, $J = 9.5$ Hz, 2H), 7.30 (d, $J = 6.5$ Hz, 2H), 7.28 (d, $J = 4.5$ Hz, 1H), 4.89 (q, $J = 6.3$ Hz, 1H), 4.02 – 3.90 (m, 1H), 3.56 – 3.44 (m, 1H), 1.50 (d, $J = 6.3$ Hz, 3H), 1.23 (t, $J = 7.0$ Hz, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 133.8, 128.7, 127.5, 84.5, 63.4, 22.7, 14.9.

HRMS (ESI) calculated for $\text{C}_{10}\text{H}_{14}\text{OS}$ $[\text{M}+\text{H}]^+$: 183.0839; Found: 183.0841



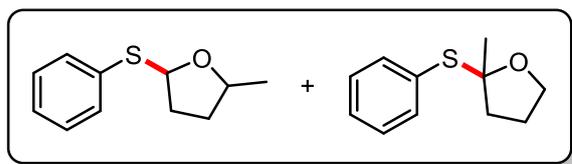
b36

Purification by column chromatography on silica gel (hexane/ethyl acetate = 50/1, v/v) afforded **b36** as a colorless oil (23.8 mg, 60% yield).

$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.55 – 7.46 (m, 2H), 7.31 (dd, $J = 5.1, 2.0$ Hz, 3H), 4.76 (dd, $J = 7.8, 4.1$ Hz, 1H), 3.61 (dd, $J = 10.6, 4.1$ Hz, 1H), 3.56 (s, 3H), 3.53 (dd, $J = 10.6, 7.8$ Hz, 1H), 3.37 (s, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 133.9, 132.3, 128.8, 127.9, 88.9, 74.7, 59.1, 56.3.

HRMS (ESI) calculated for $\text{C}_{10}\text{H}_{15}\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$: 199.0787; Found: 199.0788



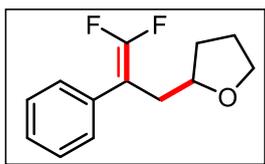
b37/ b37'

Due to the minimal polarity difference of **b37** and **b37'**, the isolation is difficult. Therefore, the mixture of **b37** and **b37'** was obtained. Purification by column chromatography on silica gel (hexane/ethyl acetate = 50/1, v/v) afforded the mixture of **b37/ b37'** as a colorless oil (34.5 mg, 89% yield).

$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.53 (dt, $J = 7.0, 2.9$ Hz, 7H), 7.51 – 7.48 (m, 2H), 7.36 – 7.28 (m, 11H), 7.28 – 7.25 (m, 2H), 7.24 – 7.18 (m, 2H), 5.69 (dd, $J = 7.4, 4.7$ Hz, 1H), 5.48 (dd, $J = 7.1, 4.0$ Hz, 1H), 4.31 (dp, $J = 8.7, 6.0$ Hz, 1H), 4.24 – 4.17

(m, 1H), 4.17 – 4.10 (m, 3H), 4.02 (ddd, $J = 10.1, 7.5, 4.4$ Hz, 3H), 2.47 (dddd, $J = 13.5, 9.7, 7.3, 3.9$ Hz, 1H), 2.39 – 2.29 (m, 1H), 2.22 – 2.14 (m, 3H), 2.11 (ddd, $J = 12.9, 6.6, 3.8$ Hz, 5H), 2.06 – 2.03 (m, 1H), 2.01 – 1.96 (m, 4H), 1.96 – 1.91 (m, 3H), 1.73 – 1.62 (m, 1H), 1.55 (s, 9H), 1.50 – 1.38 (m, 1H), 1.35 (d, $J = 6.2$ Hz, 3H), 1.29 (d, $J = 6.1$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 136.1, 135.5, 133.4, 131.6, 130.8, 128.8, 128.5, 128.1, 126.9, 126.6, 95.0, 87.1, 86.8, 74.5, 67.7, 39.5, 33.6, 33.2, 32.5, 28.2, 25.1, 22.0, 20.1.



d1

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d1** as a colorless oil (24.7 mg, 55% yield).

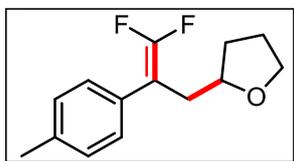
¹H NMR (400 MHz, CDCl₃) δ 7.34 (d, *J* = 4.8 Hz, 4H), 7.30 – 7.22 (m, 1H), 3.90 – 3.75 (m, 2H), 3.73 – 3.63 (m, 1H), 2.71 (ddt, *J* = 14.2, 6.7, 2.4 Hz, 1H), 2.49 (ddt, *J* = 14.2, 6.8, 2.3 Hz, 1H), 1.93 – 1.84 (m, 2H), 1.83 – 1.75 (m, 1H), 1.52 – 1.43 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.3 (t, *J* = 286.6 Hz), 133.6, 128.5, 128.4 (t, *J* = 3.2 Hz), 127.3, 90.1 (dd, *J* = 20.1, 16.6 Hz), 77.0 (t, *J* = 3.0 Hz), 67.8, 33.9, 30.9, 25.6

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -90.80 (s, 1F), -90.81 (s, 1F).

¹⁹F NMR (376 MHz, CDCl₃) δ -90.72 (d, *J* = 42.8 Hz, 1F), -90.85 (d, *J* = 44.0 Hz, 1F)

HRMS (ESI) calculated for C₁₃H₁₄F₂O [M+H]⁺: 225.1086; Found: 225.1081



d2

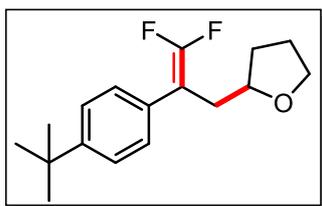
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d2** as a colorless oil (40.0 mg, 84% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.23 (d, *J* = 7.1 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 3.88 – 3.77 (m, 2H), 3.72 – 3.65 (m, 1H), 2.70 (ddt, *J* = 14.1, 6.6, 2.4 Hz, 1H), 2.47 (ddt, *J* = 14.2, 7.0, 2.3 Hz, 1H), 2.34 (s, 3H), 1.93 – 1.84 (m, 2H), 1.83 – 1.75 (m, 1H), 1.53 – 1.43 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.3 (t, *J* = 289.5 Hz), 137.2, 130.6, 129.3, 128.3 (t, *J* = 3.0 Hz), 89.9 (dd, *J* = 20.9, 15.4 Hz), 70.1, 67.9, 33.9, 30.9, 25.7, 21.2.

¹⁹F NMR (376 MHz, CDCl₃) δ -90.96 (d, *J* = 43.2 Hz, 1F), -91.26 (d, *J* = 44.0 Hz, 1F).

HRMS (ESI) calculated for C₁₄H₁₆F₂O [M+H]⁺: 239.1242; Found: 239.1240



d3

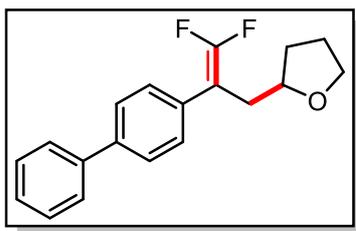
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d3** as a colorless oil (49.3 mg, 88% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.42 – 7.38 (m, 2H), 7.31 (d, *J* = 7.6 Hz, 2H), 3.95 – 3.81 (m, 2H), 3.78 – 3.68 (m, 1H), 2.74 (ddt, *J* = 14.1, 6.6, 2.5 Hz, 1H), 2.52 (ddt, *J* = 14.1, 6.9, 2.3 Hz, 1H), 1.98 – 1.89 (m, 2H), 1.88 – 1.80 (m, 1H), 1.58 – 1.48 (m, 1H), 1.35 (s, 9H).

¹³C NMR (101 MHz, CDCl₃) δ 154.3 (dd, *J* = 290.9, 287.8 Hz), 150.2, 130.4, 127.9 (t, *J* = 3.3 Hz), 125.4, 89.8 (dd, *J* = 20.3, 15.5 Hz), 77.0, 67.8, 34.5, 33.8, 31.3, 30.9, 25.6.

¹⁹F NMR (376 MHz, CDCl₃) δ -90.81 (d, *J* = 41.7 Hz, 1F), -90.96 (d, *J* = 42.2 Hz, 1F).

HRMS (ESI) calculated for C₁₇H₂₂F₂O [M+H]⁺: 281.1712; Found: 281.1707



d4

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d4** as a colorless oil (53.9 mg, 90% yield).

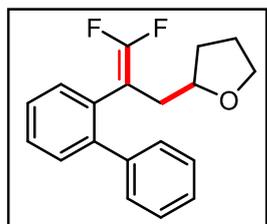
¹H NMR (400 MHz, CDCl₃) δ 7.61 – 7.56 (m, 4H), 7.43 (t, *J* = 7.7 Hz, 4H), 7.34 (t, *J* = 7.3 Hz, 1H), 3.91 – 3.83 (m, 2H), 3.70 (td, *J* = 8.1, 5.9 Hz, 1H), 2.74 (ddt, *J* = 14.3, 6.7, 2.4 Hz, 1H), 2.53 (ddt, *J* = 14.3, 6.8, 2.3 Hz, 1H), 1.96 – 1.87 (m, 2H), 1.86 – 1.76 (m, 1H), 1.56 – 1.46 (m, 1H).

¹³C NMR (400 MHz, CDCl₃) δ 154.4 (t, *J* = 290.1 Hz), 140.6, 140.1, 132.5, 128.8,

128.7 (t, $J = 3.4$ Hz), 127.4, 127.2, 127.0, 89.8 (t, $J = 17.3$ Hz), 67.8, 33.8, 31.0, 25.6.

^{19}F NMR (376 MHz, CDCl_3) δ -90.04 (d, $J = 40.6$ Hz, 1F), -90.16 (d, $J = 40.6$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{19}\text{H}_{18}\text{F}_2\text{O}$ $[\text{M}+\text{H}]^+$: 301.1399; Found: 301.1392



d5

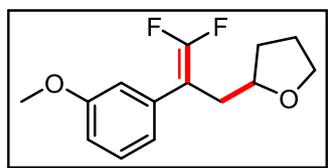
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d5** as a colorless oil (51.6 mg, 86% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.40 – 7.28 (m, 9H), 3.78 – 3.69 (m, 1H), 3.65 – 3.54 (m, 2H), 2.18 – 2.01 (m, 1H), 1.90 – 1.79 (m, 1H), 1.79 – 1.71 (m, 2H), 1.71 – 1.65 (m, 1H), 1.32 – 1.20 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 154.3 (t, $J = 286.8$ Hz), 141.6 (d, $J = 2.9$ Hz), 141.2, 131.9 (d, $J = 4.9$ Hz), 131.3, 130.4, 128.6 (d, $J = 41.9$ Hz), 127.4 (d, $J = 5.6$ Hz), 125.8, 120.4, 119.4, 110.7, 90.4 (dd, $J = 22.0, 17.8$ Hz), 67.7, 34.1, 30.9, 25.6.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -89.35 (d, $J = 43.1$ Hz, 1F), -93.06 (d, $J = 42.5$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{19}\text{H}_{18}\text{F}_2\text{O}$ $[\text{M}+\text{H}]^+$: 301.1399; Found: 301.1392



d6

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d6** as a colorless oil (34.6 mg, 68% yield).

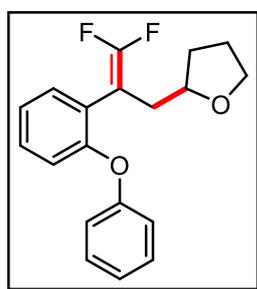
^1H NMR (400 MHz, CDCl_3) δ 7.29 – 7.25 (m, 1H), 6.92 (d, $J = 7.8$ Hz, 1H), 6.89 (s, 1H), 6.82 (dd, $J = 8.3, 2.3$ Hz, 1H), 3.91 – 3.80 (m, 2H), 3.80 (s, 3H), 3.69 (td, $J = 8.1, 5.9$ Hz, 1H), 2.69 (ddt, $J = 14.2, 6.7, 2.4$ Hz, 1H), 2.47 (ddt, $J = 14.2, 6.8, 2.4$ Hz, 1H),

1.95 – 1.84 (m, 2H), 1.86 – 1.75 (m, 1H), 1.54 – 1.42 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 159.6, 154.4 (t, *J* = 288.4 Hz), 135.0 (t, *J* = 3.8 Hz), 129.6 (d, *J* = 275 Hz), 120.8 (t, *J* = 3.0 Hz), 114.4 (t, *J* = 3.3 Hz), 112.8, 90.1 (dd, *J* = 21.6, 14.5 Hz), 67.8, 55.3, 33.9, 31.0, 25.6, 8.1.

¹⁹F NMR (376 MHz, CDCl₃) δ -90.02 (d, *J* = 41.0 Hz, 1F), -90.54 (d, *J* = 40.9 Hz, 1F).

HRMS (ESI) calculated for C₁₄H₁₆F₂O₂ [M+H]⁺: 255.1192; Found: 255.1199



d7

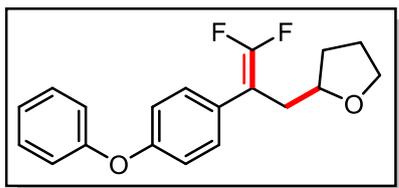
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d7** as a colorless oil (50.0 mg, 79% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.31 (d, *J* = 2.1 Hz, 1H), 7.31 – 7.27 (m, 2H), 7.27 – 7.21 (m, 1H), 7.12 – 7.04 (m, 2H), 6.96 – 6.92 (m, 2H), 6.90 (dd, *J* = 8.2, 1.2 Hz, 1H), 3.85 – 3.74 (m, 2H), 3.70 – 3.62 (m, 1H), 2.65 (ddt, *J* = 14.1, 6.5, 2.2 Hz, 1H), 2.47 (ddt, *J* = 14.1, 7.1, 2.1 Hz, 1H), 1.91 – 1.82 (m, 2H), 1.81 – 1.72 (m, 1H), 1.50 – 1.41 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 157.3, 154.9, 153.9 (t, *J* = 287.7 Hz), 131.8 (t, *J* = 2.3 Hz), 129.7, 129.3, 125.4, 123.6, 123.1, 119.3, 118.3, 86.6 (dd, *J* = 23.8, 17.6 Hz), 77.2, 67.7, 34.0, 30.9, 25.6.

¹⁹F NMR (376 MHz, CDCl₃) δ -88.47 (d, *J* = 40.3 Hz, 1F), -92.32 (d, *J* = 40.2 Hz, 1F).

HRMS (ESI) calculated for C₁₉H₁₈F₂O₂ [M+H]⁺: 317.1348; Found: 317.1346



d8

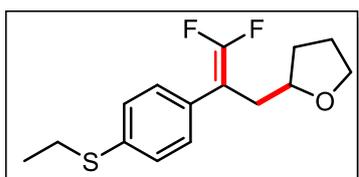
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d8** as a colorless oil (46.8 mg, 74% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.34 (dd, *J* = 8.6, 7.4 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H), 7.12 (t, *J* = 7.4 Hz, 1H), 7.03 (d, *J* = 7.5 Hz, 2H), 6.98 (d, *J* = 8.7 Hz, 2H), 3.89 – 3.79 (m, 2H), 3.70 (td, *J* = 8.1, 4.5 Hz, 1H), 2.67 (ddt, *J* = 14.2, 6.8, 2.4 Hz, 1H), 2.48 (ddt, *J* = 14.2, 6.7, 2.4 Hz, 1H), 1.98 – 1.85 (m, 2H), 1.89 – 1.76 (m, 1H), 1.55 – 1.44 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 156.8, 156.6, 154.2 (t, *J* = 290.5 Hz), 129.8, 129.7 (t, *J* = 3.2 Hz), 128.2, 123.5, 119.2, 118.5, 89.5 (dd, *J* = 20.2, 16.6 Hz), 77.0, 67.8, 34.0, 31.0, 25.6.

¹⁹F NMR (376 MHz, CDCl₃) δ -90.88 (d, *J* = 42.9 Hz, 1F), -91.00 (d, *J* = 42.1 Hz, 1F)

HRMS (ESI) calculated for C₁₉H₁₈F₂O₂ [M+H]⁺: 317.1348; Found: 317.1343



d9

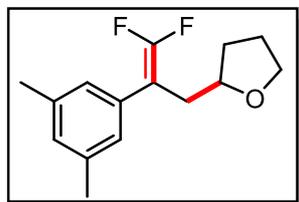
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d9** as a pale-yellow oil (35.7 mg, 63% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.29 (d, *J* = 8.8 Hz, 2H), 7.27 (d, *J* = 2.9 Hz, 2H), 3.88 – 3.77 (m, 2H), 3.72 – 3.65 (m, 1H), 2.95 (q, *J* = 7.4 Hz, 2H), 2.68 (ddt, *J* = 14.3, 6.7, 2.4 Hz, 1H), 2.48 (ddt, *J* = 14.2, 6.7, 2.4 Hz, 1H), 1.95 – 1.86 (m, 2H), 1.86 – 1.77 (m, 1H), 1.54 – 1.43 (m, 1H), 1.33 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 159.2, 154.3 (t, *J* = 290.2 Hz), 136.0, 130.9, 128.7 (t, *J* = 3.6 Hz), 128.6, 89.6 (t, *J* = 18.2 Hz), 67.8, 33.7, 30.9, 27.4, 25.6, 14.3.

¹⁹F NMR (376 MHz, CDCl₃) δ -90.15 (s, 2F)

HRMS (ESI) calculated for C₁₅H₁₈F₂OS [M+H]⁺: 285.1120; Found: 285.1126



d10

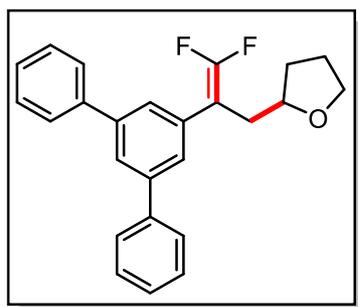
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d10** as a colorless oil (38.3 mg, 76% yield).

¹H NMR (400 MHz, CDCl₃) δ 6.94 (s, 2H), 6.90 (s, 1H), 3.89 – 3.76 (m, 2H), 3.73 – 3.63 (m, 1H), 2.69 (ddt, *J* = 14.1, 6.6, 2.5 Hz, 1H), 2.45 (ddt, *J* = 14.1, 7.1, 2.3 Hz, 1H), 2.30 (s, 6H), 1.94 – 1.85 (m, 2H), 1.84 – 1.74 (m, 1H), 1.55 – 1.40 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.2 (dd, *J* = 289.8, 286.9 Hz), 137.9, 133.3, 129.1, 126.2 (t, *J* = 3.0 Hz), 90.10 (dd, *J* = 21.0, 15.0 Hz), 77.0, 67.8, 34.0, 30.9, 25.6, 21.3.

¹⁹F NMR (376 MHz, CDCl₃) δ -90.86 (d, *J* = 42.9 Hz, 1F), -91.36 (d, *J* = 42.9 Hz, 1F).

HRMS (ESI) calculated for C₁₅H₁₈F₂O [M+H]⁺: 253.1399; Found: 253.1403



d11

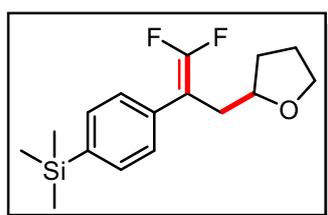
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d11** as a pale-yellow oil (52.6 mg, 70% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.71 (t, *J* = 1.8 Hz, 1H), 7.64 (d, *J* = 7.0 Hz, 4H), 7.54 (s, 2H), 7.46 (t, *J* = 7.6 Hz, 4H), 7.37 (t, *J* = 7.3 Hz, 2H), 3.93 – 3.84 (m, 2H), 3.70 (td, *J* = 8.1, 5.9 Hz, 1H), 2.79 (ddt, *J* = 14.2, 6.8, 2.3 Hz, 1H), 2.57 (ddt, *J* = 14.2, 6.7, 2.3 Hz, 1H), 1.98 – 1.86 (m, 2H), 1.86 – 1.76 (m, 1H), 1.57 – 1.47 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 154.56 (t, $J = 289.1$ Hz), 142.1, 140.9, 134.6, 128.9, 127.6, 127.4, 126.3 (t, $J = 3.2$ Hz), 125.31, 90.22 (dd, $J = 21.6, 14.8$ Hz), 77.0, 67.9, 34.1, 31.1, 25.6.

^{19}F NMR (376 MHz, CDCl_3) δ -89.82 (d, $J = 40.6$ Hz, 1F), -90.17 (d, $J = 40.8$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{25}\text{H}_{22}\text{F}_2\text{O}$ $[\text{M}+\text{H}]^+$: 377.1712; Found: 377.1718



d12

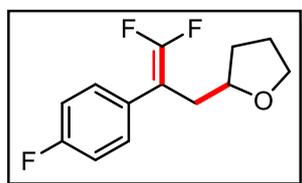
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d12** as a colorless oil (40.9 mg, 69% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.50 (d, $J = 8.1$ Hz, 2H), 7.33 (d, $J = 8.4$ Hz, 2H), 3.89 – 3.78 (m, 2H), 3.68 (td, $J = 8.1, 6.0$ Hz, 1H), 2.71 (ddt, $J = 14.2, 6.7, 2.4$ Hz, 1H), 2.50 (ddt, $J = 14.2, 6.9, 2.3$ Hz, 1H), 1.93 – 1.85 (m, 2H), 1.83 – 1.74 (m, 1H), 0.26 (s, 9H).

^{13}C NMR (101 MHz, CDCl_3) δ 154.4 (t, $J = 289.2$ Hz), 139.6, 133.9, 133.5, 127.6 (t, $J = 3.1$ Hz), 90.09 (t, $J = 17.9$ Hz), 77.0, 67.8, 33.8, 30.9, 25.6, -1.2.

^{19}F NMR (376 MHz, CDCl_3) δ -90.34 (s, 2F)

HRMS (ESI) calculated for $\text{C}_{16}\text{H}_{22}\text{F}_2\text{OSi}$ $[\text{M}+\text{H}]^+$: 297.1481; Found: 297.1489



d13

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d13** as a colorless oil (29.5 mg, 61% yield).

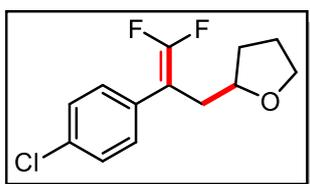
^1H NMR (400 MHz, CDCl_3) δ 7.34 – 7.28 (m, 2H), 7.07 – 7.01 (m, 2H), 3.88 – 3.76 (m, 2H), 3.69 (td, $J = 8.1, 5.9$ Hz, 1H), 2.66 (ddt, $J = 14.3, 6.9, 2.3$ Hz, 1H), 2.47 (ddt,

$J = 14.3, 6.5, 2.5$ Hz, 1H), 1.95 – 1.87 (m, 2H), 1.86 – 1.77 (m, 1H), 1.54 – 1.42 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 162.0 (d, $J = 247.1$ Hz), 154.3 (t, $J = 292.8$), 130.17 (dt, $J = 7.6, 3.2$ Hz), 129.6, 115.5 (d, $J = 21.3$ Hz), 89.44 (t, $J = 18.3$ Hz), 77.0, 67.9, 34.1, 31.0, 25.6.

^{19}F NMR (376 MHz, CDCl_3) δ -90.70 (s, 2F), -114.53 (s, 1F).

HRMS (ESI) calculated for $\text{C}_{13}\text{H}_{13}\text{F}_3\text{O}$ $[\text{M}+\text{H}]^+$: 243.0992; Found: 243.0993



d14

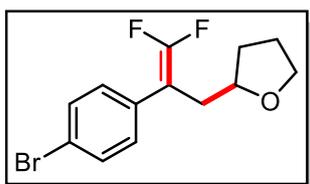
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d14** as a colorless oil (28.4 mg, 55% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.32 (d, $J = 8.8$ Hz, 2H), 7.28 (d, $J = 9.0$ Hz, 2H), 3.87 – 3.76 (m, 2H), 3.68 (td, $J = 8.1, 5.9$ Hz, 1H), 2.65 (ddt, $J = 14.3, 7.0, 2.3$ Hz, 1H), 2.48 (ddt, $J = 14.3, 6.5, 2.5$ Hz, 1H), 1.96 – 1.84 (m, 2H), 1.87 – 1.75 (m, 1H), 1.52 – 1.43 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 154.3 (t, $J = 290.6$ Hz), 133.2, 132.1, 129.7 (t, $J = 3.3$ Hz), 128.6, 89.4 (dd, $J = 20.3, 16.5$ Hz), 76.90 (t, $J = 2.9$ Hz), 77.0, 67.8, 33.8, 31.0, 25.6.

^{19}F NMR (376 MHz, CDCl_3) δ -89.85 (d, $J = 35.3$ Hz, 1F), -89.98 (d, $J = 39.9$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{13}\text{H}_{13}\text{ClF}_2\text{O}$ $[\text{M}+\text{H}]^+$: 259.0696; Found: 259.0690



d15

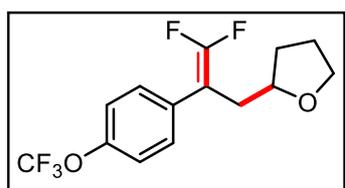
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d15** as a colorless oil (30.4 mg, 50% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.48 (d, *J* = 8.5 Hz, 2H), 7.22 (d, *J* = 7.6 Hz, 2H), 3.88 – 3.76 (m, 2H), 3.69 (td, *J* = 8.1, 5.9 Hz, 1H), 2.65 (ddt, *J* = 14.4, 6.9, 2.3 Hz, 1H), 2.48 (ddt, *J* = 14.3, 6.4, 2.5 Hz, 1H), 1.95 – 1.86 (m, 2H), 1.86 – 1.77 (m, 1H), 1.52 – 1.43 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.2 (dd, *J* = 290.6, 288.6 Hz), 132.6 (dd, *J* = 3.5, 1.9 Hz), 131.6, 130.0 (t, *J* = 3.3 Hz), 121.3, 89.5 (dd, *J* = 21.0, 15.7 Hz), 77.0, 67.8, 33.7, 31.0, 25.6.

¹⁹F NMR (376 MHz, CDCl₃) δ -89.68 (d, *J* = 39.5 Hz, 1F), -89.82 (d, *J* = 39.9 Hz, 1F).

HRMS (ESI) calculated for C₁₃H₁₃BrF₂O [M+H]⁺: 303.0191; Found: 303.0190



d16

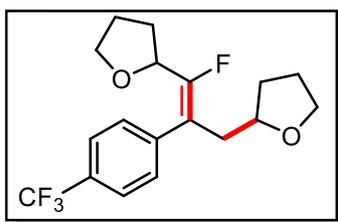
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d2** as a pale-yellow oil (37.0 mg, 60% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.38 (d, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 7.9 Hz, 2H), 3.88 – 3.77 (m, 2H), 3.70 (td, *J* = 8.2, 6.0 Hz, 1H), 2.66 (ddt, *J* = 14.3, 7.1, 2.3 Hz, 1H), 2.50 (ddt, *J* = 14.4, 6.3, 2.5 Hz, 1H), 1.96 – 1.88 (m, 2H), 1.87 – 1.79 (m, 1H), 1.54 – 1.45 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.4 (t, *J* = 291.6 Hz), 148.2, 132.4, 129.8 (t, *J* = 3.3 Hz), 121.7, 120.9, 119.2, 89.3 (dd, *J* = 21.8, 15.1 Hz), 67.8, 33.4, 31.0, 25.6.

¹⁹F NMR (376 MHz, CDCl₃) δ -57.88 (s, 3F), -89.81 (d, *J* = 40.1 Hz, 1F), -90.00 (d, *J* = 40.1 Hz, 1F).

HRMS (ESI) calculated for C₁₄H₁₃F₅O₂ [M+H]⁺: 309.0909; Found: 309.0902



d17

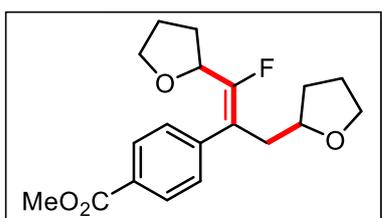
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **d17** as a colorless oil (38.5 mg, 56% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.60 (d, *J* = 7.9 Hz, 2H), 7.40 (t, *J* = 8.3 Hz, 2H), 4.40 – 4.25 (m, 1H), 3.98 – 3.91 (m, 1H), 3.85 – 3.62 (m, 4H), 2.75 (ddd, *J* = 13.6, 6.6, 3.0 Hz, 1H), 2.63 (tdd, *J* = 13.5, 7.0, 3.0 Hz, 1H), 2.11 – 1.98 (m, 2H), 1.94 – 1.76 (m, 5H), 1.59 – 1.39 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 157.0 (d, *J* = 260.8 Hz), 156.9 (d, *J* = 260.4 Hz), 141.8 (d, *J* = 8.1 Hz), 141.3 (d, *J* = 8.1 Hz), 129.6, 125.4 (t, *J* = 4.6 Hz), 119.4 (t, *J* = 18.0 Hz), 74.2 (d, *J* = 26.9 Hz), 69.1, 67.8 (d, *J* = 11.7 Hz), 36.4 (d, *J* = 4.2 Hz), 35.9 (d, *J* = 4.6 Hz), 31.0 (d, *J* = 22.6 Hz), 28.5, 26.8, 25.6 (d, *J* = 9.3 Hz).

¹⁹F NMR (376 MHz, CDCl₃) δ -62.53 (d, *J* = 5.9 Hz, 3F), -125.37 (d, *J* = 29.6 Hz, 0.5F), -126.07 (d, *J* = 29.9 Hz, 0.5F).

HRMS (ESI) calculated for C₁₈H₂₀F₄O₂ [M+H]⁺: 345.1473; Found: 345.1477



d18

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **d18** as a colorless oil (34.7 mg, 52% yield).

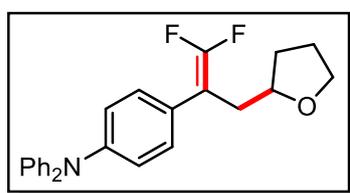
¹H NMR (400 MHz, CDCl₃) δ 8.01 (d, *J* = 7.9 Hz, 2H), 7.35 (t, *J* = 7.4 Hz, 2H), 4.43 – 4.28 (m, 1H), 3.98 – 3.93 (m, 1H), 3.92 (s, 3H), 3.86 – 3.81 (m, 1H), 3.80 – 3.76 (m, 1H), 3.75 – 3.60 (m, 2H), 2.77 (ddt, *J* = 13.3, 6.4, 3.2 Hz, 1H), 2.68 – 2.59 (m, 1H), 2.10 – 1.98 (m, 2H), 1.93 – 1.76 (m, 5H), 1.56 – 1.37 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 166.8 (d, *J* = 2.4 Hz), 156.8 (d, *J* = 260.7 Hz), 156.7

(d, $J = 260.4$ Hz), 142.8 (d, $J = 8.3$ Hz), 142.3 (d, $J = 8.1$ Hz), 129.6 (d, $J = 4.5$ Hz), 129.3, 119.6 (dd, $J = 18.6, 12.9$ Hz), 74.2 (d, $J = 27.0$ Hz), 69.1, 67.7 (d, $J = 11.6$ Hz), 52.2, 36.0 (dd, $J = 39.5, 4.5$ Hz), 30.9 (d, $J = 19.6$ Hz), 28.5, 26.8, 25.6 (d, $J = 8.6$ Hz).

^{19}F NMR (376 MHz, CDCl_3) δ -125.50 (d, $J = 29.6$ Hz, 0.5F), -126.20 (d, $J = 29.7$ Hz, 0.5F).

HRMS (ESI) calculated for $\text{C}_{19}\text{H}_{23}\text{FO}_4$ $[\text{M}+\text{H}]^+$: 335.1654; Found: 335.1661



d19

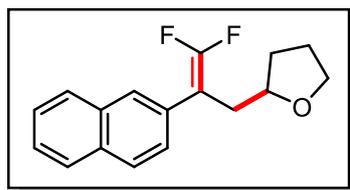
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d19** as a green oil (39.1 mg, 52% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.28 (dd, $J = 8.5, 7.2$ Hz, 4H), 7.23 (d, $J = 7.6$ Hz, 2H), 7.12 (d, $J = 7.3$ Hz, 4H), 7.05 (dd, $J = 8.0, 6.3$ Hz, 4H), 3.95 – 3.84 (m, 2H), 3.74 (td, $J = 8.1, 5.9$ Hz, 1H), 2.69 (ddt, $J = 14.1, 6.6, 2.5$ Hz, 1H), 2.50 (ddt, $J = 14.1, 6.8, 2.3$ Hz, 1H), 2.00 – 1.92 (m, 2H), 1.91 – 1.81 (m, 1H), 1.58 – 1.49 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 154.3 (t, $J = 215.6$ Hz), 147.6, 146.8, 129.3, 129.0 (t, $J = 3.9$ Hz), 127.05, 127.01, 124.55, 123.08 (d, $J = 4.5$ Hz), 114.50, 89.6 (dd, $J = 20.6, 14.6$ Hz), 67.8, 33.8, 31.0, 25.6.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -90.70 (d, $J = 42.8$ Hz, 1F), -90.89 (d, $J = 42.9$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{25}\text{H}_{23}\text{F}_2\text{NO}$ $[\text{M}+\text{H}]^+$: 392.1821; Found: 392.1826



d20

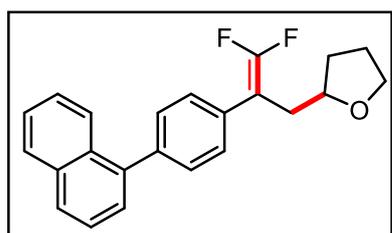
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d20** as a colorless oil (35.6 mg, 65% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.81 (dd, *J* = 8.7, 2.4 Hz, 4H), 7.50 – 7.44 (m, 3H), 3.89 – 3.79 (m, 2H), 3.67 (td, *J* = 8.1, 6.0 Hz, 1H), 2.81 (ddt, *J* = 14.3, 6.7, 2.4 Hz, 1H), 2.59 (ddt, *J* = 14.3, 6.9, 2.3 Hz, 1H), 1.94 – 1.85 (m, 2H), 1.84 – 1.72 (m, 1H), 1.58 – 1.43 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.6 (dd, *J* = 290.6, 287.8 Hz), 133.3, 132.6, 131.0 (t, *J* = 3.5 Hz), 128.2, 128.0, 127.5 (t, *J* = 3.4 Hz), 127.5, 126.4, 126.2, 90.2 (dd, *J* = 21.3, 14.8 Hz), 77.3, 76.8, 67.9, 34.0, 31.0, 25.6.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -90.12 (d, *J* = 40.3 Hz, 1F), -90.36 (d, *J* = 40.5 Hz, 1F).

HRMS (ESI) calculated for C₁₇H₁₆F₂O [M+H]⁺: 275.1242; Found: 275.1252



d21

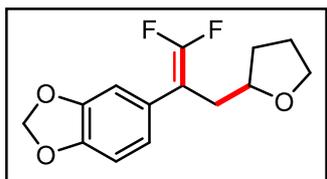
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d21** as a colorless oil (46.9 mg, 67% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.95 – 7.82 (m, 3H), 7.51 (d, *J* = 7.0 Hz, 1H), 7.49 – 7.45 (m, 5H), 7.42 (ddd, *J* = 8.6, 7.0, 1.5 Hz, 2H), 3.96 – 3.85 (m, 2H), 3.73 (td, *J* = 8.0, 5.9 Hz, 1H), 2.77 (ddt, *J* = 14.3, 6.8, 2.4 Hz, 1H), 2.57 (ddt, *J* = 14.3, 6.7, 2.3 Hz, 1H), 2.00 – 1.89 (m, 2H), 1.89 – 1.80 (m, 1H), 1.61 – 1.48 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.5 (t, *J* = 290.6 Hz) 139.8, 139.7, 133.9, 132.5, 131.6, 130.2, 128.4, 128.2 (t, *J* = 3.3 Hz), 77.1, 127.8, 127.0, 126.1, 126.0, 125.9, 125.4, 90.0 (dd, *J* = 19.4, 16.6 Hz), 67.9, 33.9, 31.1, 25.7.

¹⁹F NMR (376 MHz, CDCl₃) δ -89.91, -90.01, -90.03, -90.14.

HRMS (ESI) calculated for C₂₃H₂₀F₂O [M+H]⁺: 351.1555; Found: 355.1547



d23

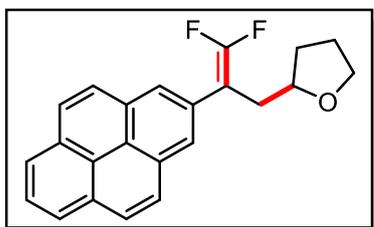
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d23** as a colorless oil (32.2 mg, 60% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.83 (s, 1H), 6.79 (s, 2H), 5.96 (s, 2H), 3.90 – 3.78 (m, 2H), 3.69 (td, $J = 8.0, 5.9$ Hz, 1H), 2.63 (ddt, $J = 14.2, 6.8, 2.4$ Hz, 1H), 2.43 (ddt, $J = 14.2, 6.7, 2.4$ Hz, 1H), 1.95 – 1.86 (m, 2H), 1.85 – 1.76 (m, 1H), 1.54 – 1.42 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 154.2 (t, $J = 289.3$ Hz), 147.8, 146.8, 127.2 (t, $J = 3.7$ Hz), 122.0 (t, $J = 3.2$ Hz), 109.0 (t, $J = 3.3$ Hz), 108.4, 101.2, 89.9 (dd, $J = 22.0, 14.8$ Hz), 77.0, 67.8, 34.2, 31.0, 25.6.

$^{19}\text{F NMR}$ (376 MHz, Chloroform-*d*) δ -90.91 (d, $J = 43.4$ Hz, 1F), -91.38 (d, $J = 43.3$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{14}\text{H}_{14}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 269.0984; Found: 269.0980



d23

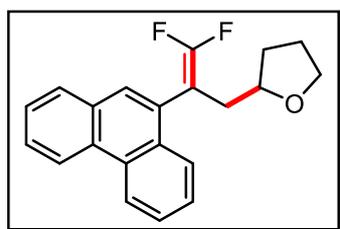
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d23** as a pale-yellow oil (48.7 mg, 70% yield).

$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 8.21 (dd, $J = 7.7, 4.3$ Hz, 4H), 8.16 – 8.03 (m, 3H), 8.03 (t, $J = 7.6$ Hz, 2H), 3.93 (dt, $J = 8.5, 6.6$ Hz, 1H), 3.82 (p, $J = 6.6$ Hz, 1H), 3.72 (td, $J = 8.0, 5.9$ Hz, 1H), 3.08 – 2.97 (m, 1H), 2.70 (ddt, $J = 14.0, 5.9, 2.7$ Hz, 1H), 1.94 – 1.83 (m, 2H), 1.83 – 1.71 (m, 1H), 1.59 – 1.46 (m, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 154.0 (t, $J = 294.1$ Hz), 139.56, 131.29, 131.09, 130.93, 128.04, 127.76, 127.29, 126.14, 125.79, 125.40, 125.28, 124.97, 124.76, 120.32, 119.34, 110.61, 88.6 (dd, $J = 21.9, 18.7$ Hz), 67.71, 36.19, 31.16, 25.62.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -87.32 (t, J = 36.7 Hz, 1F), -91.84 (d, J = 40.5 Hz, 1F).

HRMS (ESI) calculated for C₂₃H₁₈F₂O [M+H]⁺: 349.1399; Found: 349.1399



d24

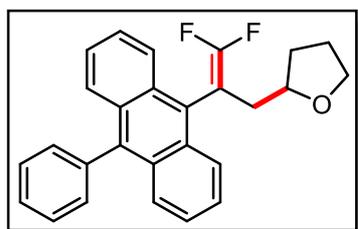
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d24** as a colorless oil (60.2 mg, 93% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.71 (dd, J = 21.5, 8.1 Hz, 2H), 8.10 – 7.84 (m, 2H), 7.76 – 7.68 (m, 1H), 7.68 – 7.62 (m, 3H), 7.62 – 7.57 (m, 1H), 3.93 – 3.76 (m, 2H), 3.74 – 3.61 (m, 1H), 2.92 – 2.76 (m, 1H), 2.64 – 2.41 (m, 1H), 1.98 – 1.82 (m, 2H), 1.81 – 1.72 (m, 1H), 1.57 – 1.39 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 131.4, 130.8, 130.3, 129.1, 128.8, 127.03, 126.9, 126.0, 125.6, 123.2, 123.0, 122.6, 88.2 (dd, J = 22.0, 18.9 Hz), 77.0, 67.7, 35.2, 31.2, 25.6.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -87.23 (dd, J = 135.5, 41.0 Hz, 1F), -92.14 (dd, J = 63.1, 41.0 Hz, 1F).

HRMS (ESI) calculated for C₂₁H₁₈F₂O [M+H]⁺: 325.1398; Found: 325.1407



d25

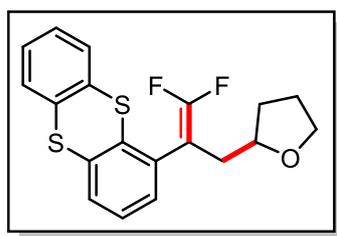
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d25** as a yellow oil (52.0 mg, 65% yield).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.35 (d, *J* = 8.8 Hz, 1H), 8.08 (d, *J* = 8.8 Hz, 1H), 7.67 (t, *J* = 9.3 Hz, 2H), 7.62 – 7.46 (m, 5H), 7.43 (t, *J* = 7.1 Hz, 2H), 7.35 (dd, *J* = 8.8, 6.5 Hz, 2H), 3.94 (td, *J* = 7.8, 5.6 Hz, 1H), 3.71 (dq, *J* = 14.5, 7.1, 6.3 Hz, 2H), 2.99 (ddd, *J* = 14.1, 8.3, 2.3 Hz, 1H), 2.62 – 2.54 (m, 1H), 1.97 – 1.72 (m, 3H), 1.55 – 1.39 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.2 (dd, *J* = 291.6, 286.7 Hz), 138.8, 138.2, 131.3, 131.1, 130.4, 130.1 (d, *J* = 3.0 Hz), 129.9, 129.7, 129.6 (d, *J* = 3.2 Hz), 128.4, 128.4, 127.7, 127.6, 127.3, 127.0, 126.1, 125.9, 125.8, 125.4, 125.3, 125.0, 85.9 (dd, *J* = 22.1, 19.6 Hz), 67.5, 36.6, 31.3, 25.6.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -85.96 (d, *J* = 38.8 Hz, 1F), -91.06 (d, *J* = 38.8 Hz, 1F).

HRMS (ESI) calculated for C₂₇H₂₂F₂O [M+H]⁺: 401.1712; Found: 401.1710



d26

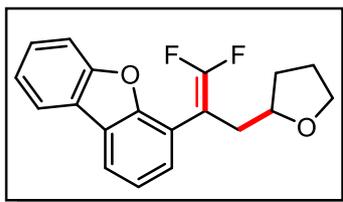
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d26** as a colorless oil (68.8 mg, 95% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.51 – 7.44 (m, 3H), 7.26 – 7.19 (m, 4H), 3.83 (q, *J* = 7.2, 6.8 Hz, 1H), 3.74 – 3.61 (m, 2H), 2.69 (dd, *J* = 14.1, 6.5 Hz, 1H), 2.49 (ddt, *J* = 14.1, 6.8, 2.4 Hz, 1H), 1.97 – 1.85 (m, 2H), 1.83 – 1.73 (m, 1H), 1.58 – 1.45 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.0 (t, *J* = 288.4 Hz), 136.3 (d, *J* = 9.5 Hz), 136.1, 135.4, 133.7, 129.5, 128.8, 128.6 (d, *J* = 6.9 Hz), 127.8 (d, *J* = 13.1 Hz), 127.3, 88.8 (dd, *J* = 23.7, 18.2 Hz), 67.7, 34.7, 31.1, 25.6.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -88.66 (d, *J* = 42.8 Hz, 1F), -93.11 (d, *J* = 42.6 Hz, 1F).

HRMS (ESI) calculated for C₁₉H₁₆F₂OS₂ [M+H]⁺: 363.0684; Found: 363.0677



d27

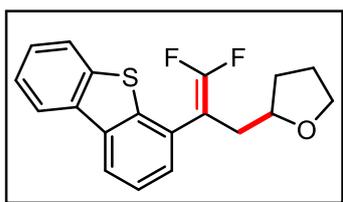
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d27** as a colorless oil (51.5 mg, 82% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.91 (dd, *J* = 20.1, 8.3 Hz, 2H), 7.58 (d, *J* = 8.2 Hz, 1H), 7.48 – 7.42 (m, 1H), 7.38 (d, *J* = 7.1 Hz, 1H), 7.33 (td, *J* = 7.4, 3.2 Hz, 2H), 3.85 – 3.73 (m, 2H), 3.64 (tdd, *J* = 8.3, 5.9, 1.5 Hz, 1H), 2.93 (ddq, *J* = 14.0, 6.6, 2.4 Hz, 1H), 2.72 (ddq, *J* = 14.1, 7.0, 2.4 Hz, 1H), 1.93 – 1.81 (m, 2H), 1.79 – 1.70 (m, 1H), 1.56 – 1.43 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 156.1, 154.3 (t, *J* = 290.0 Hz), 153.8, 128.0 (t, *J* = 2.5 Hz), 127.4, 124.6, 124.1, 122.9 (d, *J* = 4.6 Hz), 120.7, 120.2, 118.1, 111.9, 86.0 (dd, *J* = 24.5, 16.6 Hz), 77.2 (t, *J* = 3.0 Hz), 67.8, 33.7, 30.9, 25.6.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -87.13 (d, *J* = 36.8 Hz, 1F), -90.58 (d, *J* = 36.7 Hz, 1F).

HRMS (ESI) calculated for C₁₉H₁₆F₂O₂ [M+H]⁺: 315.1192; Found: 315.1189



d28

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d28** as a colorless oil (61.4 mg, 93% yield).

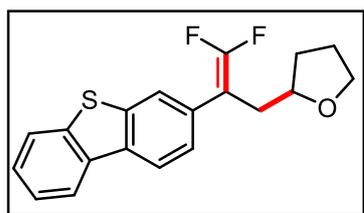
¹H NMR (400 MHz, CDCl₃) δ 8.15 – 8.06 (m, 2H), 7.87 – 7.80 (m, 1H), 7.50 – 7.41 (m, 3H), 7.36 (d, *J* = 6.2 Hz, 1H), 3.87 – 3.72 (m, 2H), 3.66 (td, *J* = 7.9, 6.0 Hz, 1H), 2.81 (dddd, *J* = 14.1, 6.7, 2.6, 1.6 Hz, 1H), 2.60 (ddt, *J* = 14.1, 6.9, 2.4 Hz, 1H), 1.96 – 1.82 (m, 2H), 1.83 – 1.71 (m, 1H), 1.54 – 1.43 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 153.9 (t, *J* = 291.4 Hz), 139.8, 139.2, 135.9 (d, *J* =

26.6 Hz), 128.6 (d, $J = 4.5$ Hz), 127.8 (d, $J = 1.9$ Hz), 127.0, 124.8, 124.5, 122.8, 121.8, 121.1, 89.2 (dd, $J = 23.2, 17.5$ Hz), 76.9, 67.8, 34.0, 31.1, 25.6.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -85.80 (d, $J = 36.9$ Hz, 1F), -90.86 (d, $J = 36.3$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{19}\text{H}_{16}\text{F}_2\text{OS}$ $[\text{M}+\text{H}]^+$: 331.0963; Found: 331.0973



d29

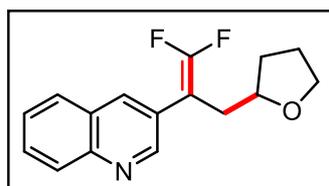
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d29** as a colorless oil (41.1 mg, 61% yield).

^1H NMR (400 MHz, CDCl_3) δ 8.16 – 8.09 (m, 2H), 7.81 (dd, $J = 8.8, 6.5$ Hz, 2H), 7.47 – 7.39 (m, 3H), 3.90 – 3.80 (m, 2H), 3.68 (td, $J = 8.2, 5.9$ Hz, 1H), 2.80 (ddt, $J = 14.3, 6.9, 2.3$ Hz, 1H), 2.58 (ddt, $J = 14.2, 6.6, 2.5$ Hz, 1H), 1.96 – 1.83 (m, 2H), 1.83 – 1.74 (m, 1H), 1.55 – 1.45 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 154.4 (t, $J = 288.8$ Hz), 139.8, 138.5, 135.8, 135.3, 130.0, 127.0 (t, $J = 3.1$ Hz), 126.96, 124.5, 122.8, 121.7, 121.6 (t, $J = 3.2$ Hz), 90.2 (dd, $J = 20.1, 16.5$ Hz), 77.0 (t, $J = 2.9$ Hz), 67.8, 34.3, 31.0, 25.6.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -90.55 (d, $J = 42.1$ Hz, 1F), -90.68 (d, $J = 40.8$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{19}\text{H}_{16}\text{F}_2\text{OS}$ $[\text{M}+\text{H}]^+$: 331.0963; Found: 331.0964



d30

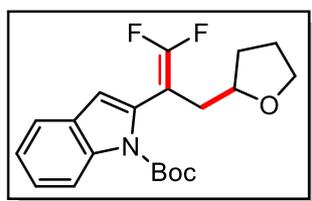
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d30** as a colorless oil (33.0 mg, 60% yield).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.92 (s, 1H), 8.17 – 8.08 (m, 2H), 7.82 (d, *J* = 8.2 Hz, 1H), 7.72 (t, *J* = 7.9 Hz, 1H), 7.57 (t, *J* = 7.6 Hz, 1H), 3.88 – 3.80 (m, 2H), 3.69 (td, *J* = 8.0, 6.1 Hz, 1H), 2.83 – 2.73 (m, 1H), 2.70 – 2.61 (m, 1H), 1.95 – 1.88 (m, 2H), 1.86 – 1.80 (m, 1H), 1.58 – 1.48 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.8 (t, *J* = 292.8 Hz), 150.4, 147.1, 135.0, 129.6, 129.2, 127.8, 127.6, 127.0, 87.9 (dd, *J* = 22.9, 13.6 Hz), 76.9 (t, *J* = 2.9 Hz), 67.8, 33.7, 31.1, 25.6.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -88.13 (d, *J* = 37.3 Hz, 1F), -88.97 (d, *J* = 37.0 Hz, 1F).

HRMS (ESI) calculated for C₁₆H₁₅F₂NO [M+H]⁺: 276.1195; Found: 276.1189



d31

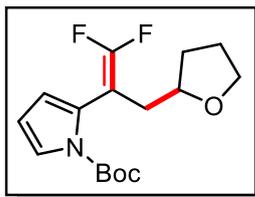
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d31** as a colorless oil (53.0 mg, 73% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.17 (d, *J* = 7.2 Hz, 1H), 7.51 (d, *J* = 7.7 Hz, 1H), 7.30 (t, *J* = 7.1 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 6.55 (s, 1H), 3.88 – 3.79 (m, 2H), 3.69 (td, *J* = 8.0, 6.0 Hz, 1H), 2.65 (dddd, *J* = 14.2, 6.9, 2.5, 1.3 Hz, 1H), 2.42 – 2.32 (m, 1H), 1.97 – 1.86 (m, 2H), 1.85 – 1.77 (m, 1H), 1.63 (s, 9H), 1.53 – 1.43 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.4 (t, *J* = 290.0 Hz), 149.8, 136.9, 131.0 (d, *J* = 7.9 Hz), 128.9, 124.5, 122.9, 120.6, 115.7, 111.5, 85.1 (dd, *J* = 28.3, 17.0 Hz), 84.0, 67.7, 34.6, 31.1, 28.1, 25.6.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -87.04 (d, *J* = 36.5 Hz, 1F), -91.30 (d, *J* = 36.4 Hz, 1F).

HRMS (ESI) calculated for C₂₀H₂₃F₂NO₃ [M+H]⁺: 364.1719; Found: 364.1712



d32

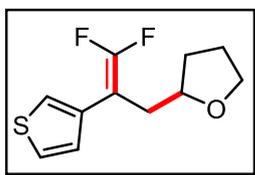
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d32** as a colorless oil (35.7 mg, 57% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.30 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.18 – 6.09 (m, 2H), 3.89 – 3.75 (m, 2H), 3.69 (td, *J* = 8.0, 6.0 Hz, 1H), 2.58 (dddd, *J* = 14.1, 6.6, 2.6, 1.6 Hz, 1H), 2.31 (ddt, *J* = 14.1, 7.0, 2.4 Hz, 1H), 1.97 – 1.87 (m, 2H), 1.86 – 1.79 (m, 1H), 1.57 (s, 9H), 1.50 – 1.41 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.4 (t, *J* = 296.7 q Hz), 151.4, 148.7, 125.0, 122.4, 115.5, 110.4, 84.0 (dd, *J* = 28.6, 17.5 Hz), 83.9, 67.6, 34.53, 30.92, 27.9, 25.6.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -87.47 (d, *J* = 37.0 Hz, 1F), -91.41 (d, *J* = 36.9 Hz, 1F).

HRMS (ESI) calculated for C₁₆H₂₁F₂NO₃ [M+H]⁺: 314.1563; Found: 314.1565



d33

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1, v/v) afforded **d33** as a colorless oil (20.7 mg, 45% yield).

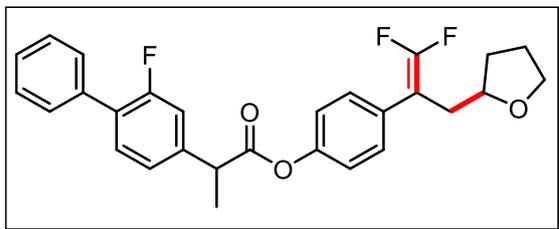
¹H NMR (400 MHz, CDCl₃) δ 7.31 (dd, *J* = 5.1, 3.0 Hz, 2H), 7.19 (ddd, *J* = 5.2, 2.3, 1.3 Hz, 1H), 4.03 – 3.85 (m, 2H), 3.72 (td, *J* = 8.0, 6.0 Hz, 1H), 2.67 (ddt, *J* = 14.3, 6.6, 2.6 Hz, 1H), 2.52 (dddd, *J* = 14.3, 6.8, 2.9, 1.4 Hz, 1H), 2.00 – 1.89 (m, 2H), 1.88 – 1.81 (m, 1H), 1.59 – 1.48 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 154.8 (t, *J* = 294.5 Hz) 133.7, 127.2, 127.1 (dd, *J* = 6.4, 2.4 Hz), 122.2 (t, *J* = 5.3 Hz), 86.4 (t, *J* = 13.6), 67.9, 33.4 (d, *J* = 2.1 Hz), 31.0, 25.6.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ -86.39 (d, *J* = 38.6 Hz, 1F), -90.68 (d, *J* = 39.3

Hz, 1F).

HRMS (ESI) calculated for $C_{13}H_{14}F_2O$ $[M+H]^+$: 231.0650; Found: 231.0640



d34

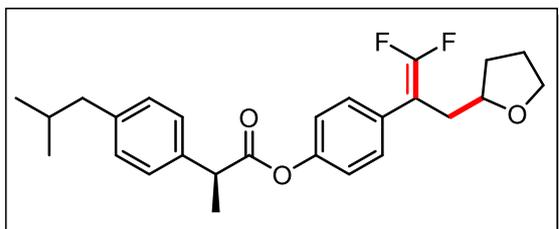
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **d34** as a colorless oil (48.4 mg, 52% yield).

1H NMR (400 MHz, Chloroform-*d*) δ 7.59 (d, $J = 7.3$ Hz, 2H), 7.48 (t, $J = 7.5$ Hz, 3H), 7.41 (d, $J = 7.2$ Hz, 1H), 7.37 (d, $J = 8.5$ Hz, 2H), 7.32 – 7.26 (m, 1H), 7.29 – 7.22 (m, 1H), 7.07 (d, $J = 8.7$ Hz, 2H), 4.03 (q, $J = 7.1$ Hz, 1H), 3.99 – 3.76 (m, 2H), 3.71 (td, $J = 8.1, 5.8$ Hz, 1H), 2.70 (ddt, $J = 14.3, 6.9, 2.3$ Hz, 1H), 2.50 (ddt, $J = 14.2, 6.5, 2.4$ Hz, 1H), 1.98 – 1.88 (m, 2H), 1.88 – 1.78 (m, 1H), 1.69 (d, $J = 7.2$ Hz, 3H), 1.55 – 1.44 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.4, 159.8 (d, $J = 248.8$ Hz), 154.3 (t, $J = 290.4$ Hz), 149.8, 141.2 (d, $J = 7.8$ Hz), 135.4, 131.3, 131.0 (d, $J = 3.8$ Hz), 129.4 (t, $J = 3.3$ Hz), 129.0 (d, $J = 3.0$ Hz), 128.5, 128.2 (d, $J = 13.4$ Hz), 127.8, 123.6 (d, $J = 3.3$ Hz), 121.4, 115.4 (d, $J = 23.8$ Hz), 89.5 (dd, $J = 20.2, 16.5$ Hz), 67.8, 45.2, 33.9, 31.0, 25.6, 18.4.

^{19}F NMR (376 MHz, $CDCl_3$) δ -90.26 (s, 2F), -117.28 (s, 1F).

HRMS (ESI) calculated for $C_{28}H_{25}F_3O_3$ $[M+H]^+$: 467.1829; Found: 467.1831



d35

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1, v/v) afforded **d35** as a colorless oil (47.9 mg, 56% yield).

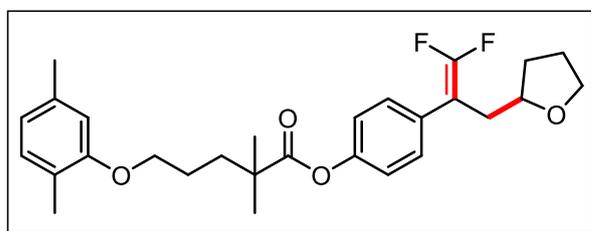
1H NMR (400 MHz, Chloroform-*d*) δ 7.30 (dd, $J = 8.4, 4.9$ Hz, 4H), 7.14 (d, $J = 7.7$

Hz, 2H), 6.99 (d, $J = 8.6$ Hz, 2H), 3.93 (q, $J = 7.1$ Hz, 1H), 3.88 – 3.74 (m, 2H), 3.71 – 3.63 (m, 1H), 2.66 (ddt, $J = 16.2, 6.5, 1.9$ Hz, 1H), 2.47 (d, $J = 7.2$ Hz, 3H), 1.94 – 1.78 (m, 4H), 1.60 (d, $J = 7.2$ Hz, 3H), 1.51 – 1.39 (m, 1H), 0.91 (d, $J = 6.6$ Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 173.2, 154.3 (t, $J = 290.1$ Hz), 145.0, 140.9, 137.2, 131.0, 130.8, 129.9, 129.6, 129.39 (t, $J = 3.3$ Hz), 127.3, 121.5, 89.5 (dd, $J = 20.2, 16.5$ Hz), 67.8, 45.2 (d, $J = 20.8$ Hz), 33.9, 31.0, 30.3, 26.7, 25.6, 22.5, 18.6.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -91.62 (d, $J = 44.1$ Hz, 1F), -91.78 (d, $J = 44.2$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{26}\text{H}_{30}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 429.2236; Found: 429.2233



d36

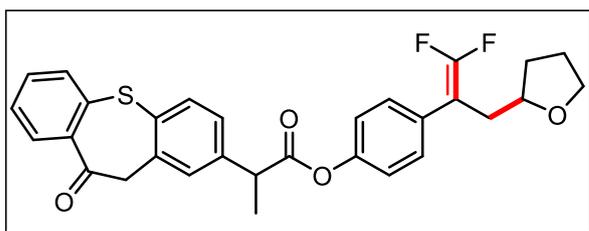
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1, v/v) afforded **d36** as a pale-yellow oil (47.2 mg, 50% yield).

^1H NMR (400 MHz, Chloroform-*d*) δ 7.33 (d, $J = 8.3$ Hz, 2H), 7.02 (d, $J = 8.7$ Hz, 2H), 6.99 (s, 1H), 6.66 (d, $J = 7.5$ Hz, 1H), 6.62 (s, 1H), 3.98 (s, 2H), 3.88 – 3.77 (m, 2H), 3.68 (td, $J = 8.1, 5.8$ Hz, 1H), 2.68 (ddt, $J = 14.3, 6.9, 2.3$ Hz, 1H), 2.47 (ddt, $J = 14.2, 6.6, 2.4$ Hz, 1H), 2.30 (s, 3H), 2.17 (s, 3H), 1.87 (s, 6H), 1.85 – 1.75 (m, 1H), 1.53 – 1.42 (m, 1H), 1.37 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 176.3, 156.9, 154.4 (t, $J = 289.9$ Hz), 150.1, 136.6, 131.0, 130.4, 129.5 (t, $J = 3.3$ Hz), 123.7, 121.6, 120.8, 112.0, 89.6 (t, $J = 16.3$ Hz), 77.3, 77.0, 67.8 (d, $J = 3.2$ Hz), 42.5, 37.2, 34.0, 31.0, 29.6 (d, $J = 28.6$ Hz), 25.6, 25.3 (d, $J = 13.3$ Hz), 21.5, 15.9.

^{19}F NMR (376 MHz, CDCl_3) δ -90.33 (d, $J = 41.0$ Hz, 1F), -90.40 (d, $J = 41.2$ Hz, 1F)

HRMS (ESI) calculated for $\text{C}_{28}\text{H}_{34}\text{F}_2\text{O}_4$ $[\text{M}+\text{H}]^+$: 473.2498; Found: 473.2450



d37

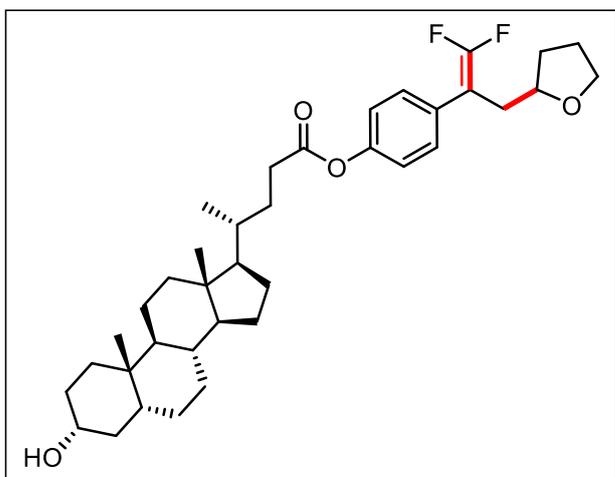
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 5/1, v/v) afforded **d37** as a colorless oil (57.2 mg, 55% yield).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.21 (d, $J = 7.8$ Hz, 1H), 7.63 (dd, $J = 16.3, 7.9$ Hz, 2H), 7.48 (s, 1H), 7.43 (t, $J = 7.1$ Hz, 1H), 7.32 (t, $J = 8.4$ Hz, 3H), 7.26 (s, 1H), 6.98 (d, $J = 8.4$ Hz, 2H), 4.39 (s, 2H), 3.96 (q, $J = 7.1$ Hz, 1H), 3.88 – 3.70 (m, 2H), 3.72 – 3.62 (m, 1H), 2.65 (ddt, $J = 14.2, 6.9, 2.3$ Hz, 1H), 2.46 (ddt, $J = 14.2, 6.5, 2.4$ Hz, 1H), 1.94 – 1.81 (m, 2H), 1.84 – 1.71 (m, 1H), 1.60 (d, $J = 7.1$ Hz, 3H), 1.52 – 1.39 (m, 1H), 1.34 – 1.25 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 191.4, 172.4, 154.3 (t, $J = 290.1$ Hz), 149.7, 142.1, 140.2, 138.2, 136.2, 133.6, 132.6, 131.7 (d, $J = 15.1$ Hz), 131.2, 130.9, 129.4 (t, $J = 3.3$ Hz), 128.8, 127.0, 126.4, 125.8, 125.3, 121.4, 89.5 (t, $J = 18.3$ Hz), 67.8, 51.1, 45.3, 33.9, 31.0, 25.6, 18.6.

¹⁹F NMR (376 MHz, CDCl₃) δ -90.25 (s, 2F).

HRMS (ESI) calculated for C₃₀H₂₆F₂O₄S [M+Na]⁺: 543.1413; Found: 543.1413



d38

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate =

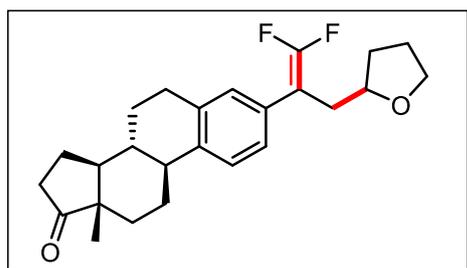
2/1, v/v) afforded **d38** as a pale-yellow oil (53.8 mg, 45% yield).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.35 (d, $J = 8.4$ Hz, 2H), 7.07 (d, $J = 8.6$ Hz, 2H), 3.90 – 3.76 (m, 2H), 3.74 – 3.56 (m, 1H), 2.73 – 2.54 (m, 1H), 2.53 – 2.41 (m, 1H), 2.02 – 1.72 (m, 3H), 1.69 – 1.57 (m, 1H), 1.55 – 1.45 (m, 1H), 1.39 (q, $J = 3.5$ Hz, 1H), 1.34 – 1.03 (m, 3H), 0.98 (d, $J = 6.2$ Hz, 2H), 0.92 (s, 2H), 0.67 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 172.7, 154.4, (t, $J = 290.0$ Hz), 150.0, 131.0, 129.4 (t, $J = 3.3$ Hz), 121.6, 89.5 (dd, $J = 21.1, 16.2$ Hz), 77.3, 76.9, 71.9, 67.8, 56.6, 56.0, 49.2, 42.8, 42.1, 40.5, 40.2, 36.5, 35.9, 35.4, 34.6, 33.9, 31.4, 31.0, 30.6, 28.3, 27.2, 26.5, 25.6, 24.3, 23.4, 20.9, 18.4, 12.1.

¹⁹F NMR (376 MHz, CDCl₃) δ -90.23 (d, $J = 41.0$ Hz, 1F) -90.36 (d, $J = 40.6$ Hz, 1F).

HRMS (ESI) calculated for C₃₇H₅₂F₂O₄ [M+H]⁺: 599.3907; Found: 599.3913



d39

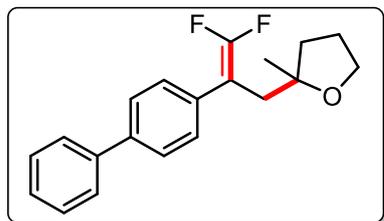
Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 10/1, v/v) afforded **d39** as a colorless oil (48.1 mg, 60% yield).

¹H NMR (400 MHz, Chloroform-*d*) δ 7.27 (d, $J = 6.1$ Hz, 1H), 7.12 (d, $J = 7.7$ Hz, 1H), 7.07 (s, 1H), 3.92 – 3.77 (m, 2H), 3.70 (td, $J = 8.1, 5.9$ Hz, 1H), 2.91 (dd, $J = 9.0, 4.3$ Hz, 2H), 2.69 (ddt, $J = 14.0, 6.6, 2.5$ Hz, 1H), 2.55 – 2.40 (m, 3H), 2.30 (td, $J = 10.5, 3.8$ Hz, 1H), 2.11 – 1.79 (m, 7H), 1.71 – 1.60 (m, 2H), 1.58 – 1.51 (m, 3H), 1.49 – 1.44 (m, 2H), 0.91 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 220.9, 154.3 (t, $J = 288.6$ Hz), 139.0, 136.6, 130.9 (t, $J = 3.2$ Hz), 128.8, 125.7, 125.5, 110.3, 110.1, 89.8 (dd, $J = 21.2, 15.0$ Hz), 67.8, 50.6, 48.0, 44.4, 38.1, 35.9, 33.8, 31.6, 31.0, 29.4, 26.5, 25.6 (d, $J = 3.0$ Hz), 21.6, 13.9.

¹⁹F NMR (376 MHz, Chloroform-*d*) δ - 90.54 (d, $J = 43.6$ Hz, 1F), -90.78 (d, $J = 43.7$ Hz, 1F).

HRMS (ESI) calculated for $C_{25}H_{30}F_2O_2$ $[M+Na]^+$: 423.2107; Found: 423.2109



d40

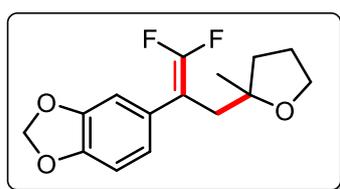
Purification by column chromatography on silica gel (hexane/ethyl acetate = 50/1, v/v) afforded **d40** as a colorless oil (44.0 mg, 70% yield).

1H NMR (400 MHz, Chloroform-*d*) δ 7.62 – 7.54 (m, 3H), 7.41 (q, J = 7.0, 6.6 Hz, 3H), 7.32 (t, J = 7.3 Hz, 1H), 3.71 (dq, J = 33.0, 8.1, 7.5 Hz, 2H), 2.65 (q, J = 14.3 Hz, 1H), 1.85 (p, J = 7.1 Hz, 2H), 1.69 (dd, J = 12.5, 7.3 Hz, 1H), 1.51 (dt, J = 12.2, 7.3 Hz, 1H), 1.12 (s, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 154.8 (t, J = 289.5 Hz), 140.6, 139.8, 133.8 (dd, J = 5.0, 3.4 Hz), 128.8 (d, J = 3.6 Hz), 127.4, 127.1 (d, J = 3.7 Hz), 90.0 (dd, J = 21.3, 14.1 Hz), 83.1 (t, J = 3.3 Hz), 67.1, 38.4, 36.6, 26.3, 26.0.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -89.15 (d, J = 38.9 Hz, 1F), -90.66 (d, J = 38.9 Hz, 1F).

HRMS (ESI) calculated for $C_{20}H_{20}F_2O$ $[M+H]^+$: 315.1555; Found: 315.1558



d41

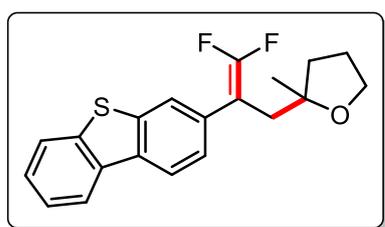
Purification by column chromatography on silica gel (hexane/ethyl acetate = 50/1, v/v) afforded **d41** as a colorless oil (34.9 mg, 62% yield).

1H NMR (400 MHz, Chloroform-*d*) δ 6.79 (d, J = 9.9 Hz, 3H), 5.95 (s, 2H), 3.82 – 3.64 (m, 2H), 2.61 – 2.48 (m, 2H), 1.95 – 1.80 (m, 2H), 1.65 (dt, J = 12.4, 7.5 Hz, 1H), 1.49 (ddd, J = 12.4, 8.1, 6.5 Hz, 1H), 1.10 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 154.7 (t, $J = 289.2$ Hz), 147.6, 146.6, 128.5 (d, $J = 4.2$ Hz), 122.0 (t, $J = 3.0$ Hz), 109.0 (t, $J = 3.0$ Hz), 108.2, 101.1, 89.9 (dd, $J = 21.8, 14.5$ Hz), 82.9 (d, $J = 3.3$ Hz), 67.0, 38.8, 36.5, 26.2, 25.9.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -90.26 (d, $J = 40.9$ Hz, 1F), -91.26 (d, $J = 40.8$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{15}\text{H}_{17}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 283.1141; Found: 283.1139



d42

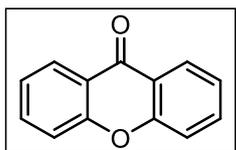
Purification by column chromatography on silica gel (hexane/ethyl acetate = 50/1, v/v) afforded **d42** as a colorless oil (41.3 mg, 60% yield).

^1H NMR (400 MHz, Chloroform-*d*) δ 8.20 – 8.08 (m, 2H), 7.87 – 7.77 (m, 2H), 7.47 – 7.37 (m, 3H), 3.71 (ddt, $J = 29.1, 8.3, 6.8$ Hz, 2H), 2.80 – 2.63 (m, 2H), 1.83 (p, $J = 7.1$ Hz, 2H), 1.66 (dt, $J = 12.3, 7.5$ Hz, 1H), 1.49 (dt, $J = 12.4, 7.3$ Hz, 1H), 1.11 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 154.9 (t, $J = 291.6$ Hz), 139.9, 138.3, 135.7, 135.3, 131.3 (dd, $J = 5.2, 3.3$ Hz), 127.2 (t, $J = 2.9$ Hz), 126.9, 124.4, 122.9, 122.6, 121.6, 121.5 (t, $J = 3.0$ Hz), 90.25 (dd, $J = 21.6, 14.3$ Hz), 83.1, 67.1, 39.0, 36.7, 26.3, 25.9.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -89.35 (d, $J = 39.3$ Hz, 1F), -90.93 (d, $J = 39.5$ Hz, 1F).

HRMS (ESI) calculated for $\text{C}_{20}\text{H}_{19}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 345.1120; Found: 345.1113



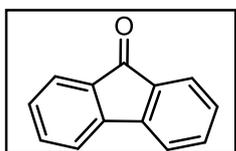
f1

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **f1** as a white solid (38.9 mg, 99% yield).

$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 8.32 (dd, $J = 8.0, 1.7$ Hz, 1H), 7.74 – 7.66 (m, 1H), 7.46 (d, $J = 8.4$ Hz, 1H), 7.36 (t, $J = 7.5$ Hz, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 177.2, 156.1, 134.8, 126.7, 123.9, 121.8, 118.0.

HRMS (EI) calculated for $\text{C}_{13}\text{H}_8\text{O}_2$ $[\text{M}]^+$: 196.0519; Found: 196.0519



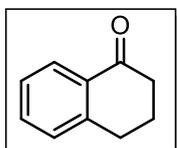
f2

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **f2** as a white solid (33.4 mg, 93% yield).

$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.63 (d, $J = 7.3$ Hz, 1H), 7.46 (q, $J = 7.4$ Hz, 2H), 7.27 (td, $J = 7.1, 1.7$ Hz, 1H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 193.9, 144.4, 134.7, 134.1, 129.1, 124.3, 120.3.

HRMS (EI) calculated for $\text{C}_{13}\text{H}_8\text{O}$ $[\text{M}]^+$: 180.0570; Found: 180.0570



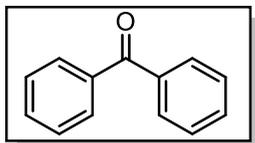
f3

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **f3** as a white solid (18.3 mg, 62% yield).

$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 8.03 (dd, $J = 7.8, 1.5$ Hz, 1H), 7.47 (td, $J = 7.5, 1.5$ Hz, 1H), 7.30 (t, $J = 7.0$ Hz, 1H), 7.25 (d, $J = 7.7$ Hz, 1H), 2.97 (t, $J = 6.1$ Hz, 2H), 2.66 (dd, $J = 7.3, 5.8$ Hz, 2H), 2.14 (p, $J = 6.5$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 198.4, 144.5, 133.4, 132.6, 128.8, 127.2, 126.6, 39.2, 29.7, 23.3.

HRMS (EI) calculated for $\text{C}_{10}\text{H}_{10}\text{O}$ $[\text{M}]^+$: 146.0627; Found: 146.0627



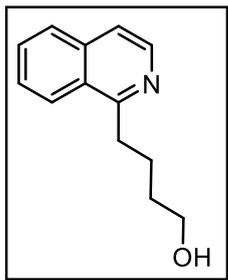
f4

Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20/1, v/v) afforded **f4** as a white solid (16.4 mg, 45% yield).

^1H NMR (400 MHz, Chloroform-*d*) δ 7.81 (d, J = 6.8 Hz, 2H), 7.59 (t, J = 7.4 Hz, 1H), 7.48 (d, J = 15.3 Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 196.8, 137.6, 132.4, 130.1, 128.3.

HRMS (EI) calculated for $\text{C}_{13}\text{H}_{10}\text{O}$ $[\text{M}]^+$: 182.0727; Found: 182.0727



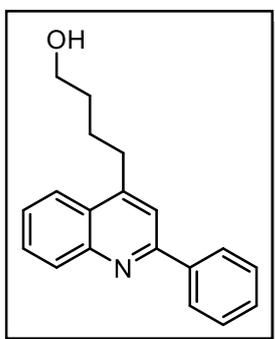
d'1

Purification by column chromatography on silica gel (hexane/ethyl acetate = 2/1, v/v) afforded **d'1** as a colorless oil (33.5 mg, 83% yield).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.39 (d, $J = 5.8$ Hz, 1H), 8.16 (d, $J = 8.4$ Hz, 1H), 7.81 (d, $J = 8.1$ Hz, 1H), 7.63 (dt, $J = 31.7, 6.9$ Hz, 1H), 7.51 (d, $J = 5.8$ Hz, 1H), 3.72 (t, $J = 6.2$ Hz, 2H), 3.36 (t, $J = 7.6$ Hz, 2H), 3.28 – 3.03 (1H), 2.00 (p, $J = 7.3$ Hz, 2H), 1.74 (p, $J = 6.4$ Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 161.8, 141.4, 136.3, 130.0, 127.4, 127.2, 127.0, 125.3, 119.4, 62.1, 34.3, 32.4, 25.3.

HRMS (ESI) calculated for C₁₃H₁₅NO [M+H]⁺: 202.1226; Found: 202.1222



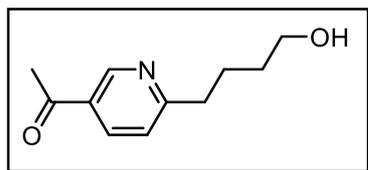
d'2

Purification by column chromatography on silica gel (hexane/ethyl acetate = 3/1, v/v) afforded **d'2** as a colorless oil (42.3 mg, 76% yield).

¹H NMR (400 MHz, Chloroform-*d*) δ 8.21 (d, $J = 7.2$ Hz, 1H), 8.11 (d, $J = 5.1$ Hz, 2H), 8.00 (d, $J = 8.5$ Hz, 1H), 7.71 (t, $J = 6.9$ Hz, 1H), 7.66 (s, 1H), 7.55 – 7.43 (m, 4H), 3.67 (t, $J = 6.4$ Hz, 2H), 3.10 (t, $J = 7.7$ Hz, 2H), 2.18 (1H), 1.85 (p, $J = 7.6$ Hz, 2H), 1.68 (dt, $J = 13.2, 6.5$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 157.2, 148.9, 148.4, 139.8, 130.4, 130.0, 129.2, 128.8, 127.6, 126.5, 126.1, 123.4, 118.8, 62.4, 32.6, 32.2, 26.3.

HRMS (ESI) calculated for $\text{C}_{19}\text{H}_{19}\text{NO}$ $[\text{M}+\text{H}]^+$: 278.1539; Found: 278.1543



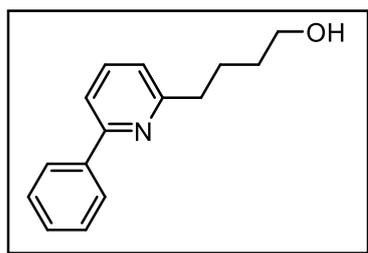
d'3

Purification by column chromatography on silica gel (hexane/ethyl acetate = 5/1, v/v) afforded **d'3** as a pale-yellow oil (17.0 mg, 44% yield).

^1H NMR (400 MHz, Chloroform-*d*) δ 9.05 (d, $J = 2.3$ Hz, 1H), 8.16 (dd, $J = 8.2, 2.3$ Hz, 1H), 7.29 (d, $J = 8.0$ Hz, 1H), 3.69 (t, $J = 6.3$ Hz, 2H), 2.91 (t, $J = 7.7$ Hz, 2H), 2.79 – 2.75 (1H), 2.62 (s, 3H), 1.86 (p, $J = 7.5$ Hz, 2H), 1.65 (dt, $J = 13.0, 6.5$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 196.6, 166.9, 149.5, 136.1, 130.2, 122.9, 62.1, 37.8, 32.0, 26.6, 25.6.

HRMS (ESI) calculated for $\text{C}_{11}\text{H}_{15}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 194.1176; Found: 194.1170



d'4

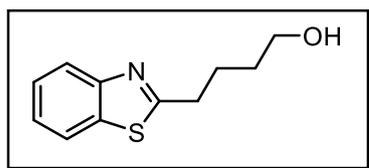
Purification by column chromatography on silica gel (hexane/ethyl acetate = 5/1, v/v) afforded **d'4** as a pale-yellow oil (15.5 mg, 34% yield).

^1H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, $J = 7.0$ Hz, 2H), 7.65 (t, $J = 7.7$ Hz, 1H), 7.51 (d, $J = 7.8$ Hz, 1H), 7.46 (t, $J = 7.5$ Hz, 2H), 7.39 (t, $J = 7.3$ Hz, 1H), 7.08

(d, $J = 7.6$ Hz, 1H), 3.66 (t, $J = 6.4$ Hz, 2H), 2.89 (t, $J = 7.6$ Hz, 2H), 2.57 – 2.28 (1H), 1.88 (p, $J = 7.4$ Hz, 2H), 1.65 (dt, $J = 13.0, 6.6$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 161.9, 157.0, 139.7, 137.1, 128.8, 128.7, 127.1, 121.2, 118.1, 62.4, 37.6, 32.0, 25.9.

HRMS (ESI) calculated for $\text{C}_{15}\text{H}_{17}\text{NO}$ $[\text{M}+\text{H}]^+$: 228.1383; Found: 228.1375



d'5

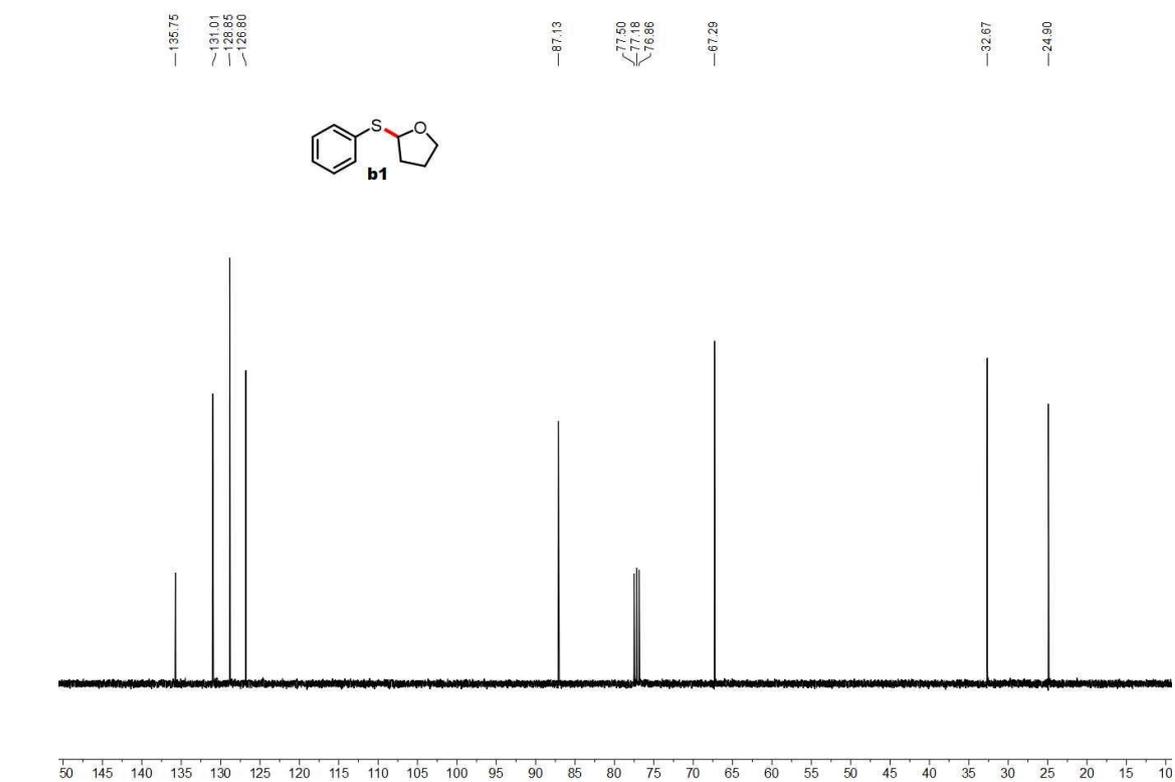
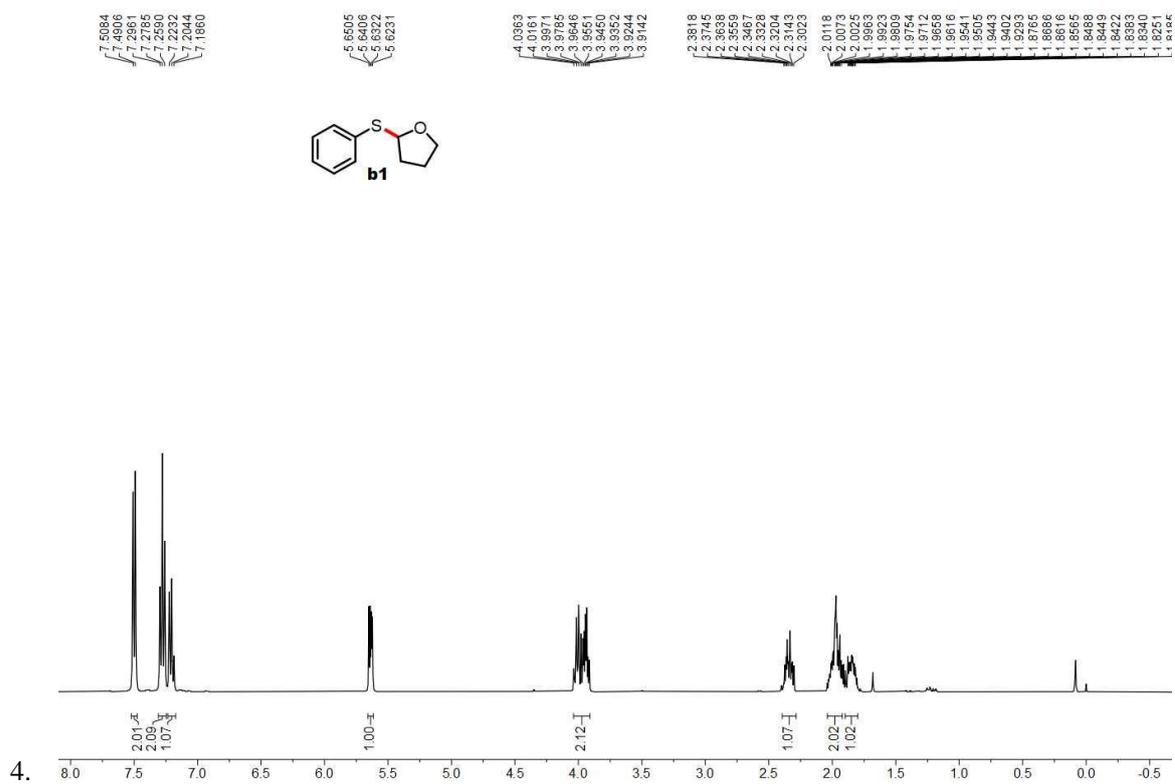
Purification by column chromatography on silica gel (hexane/ethyl acetate = 2/1, v/v) afforded **d'5** as a pale-yellow oil (25.1mg, 61% yield).

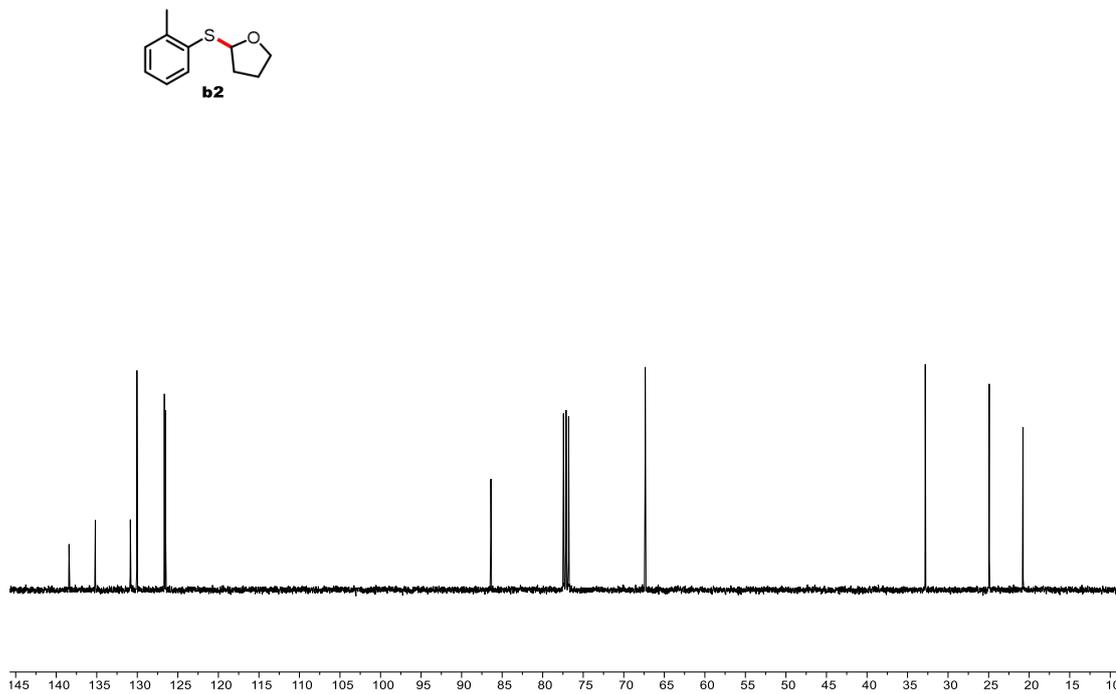
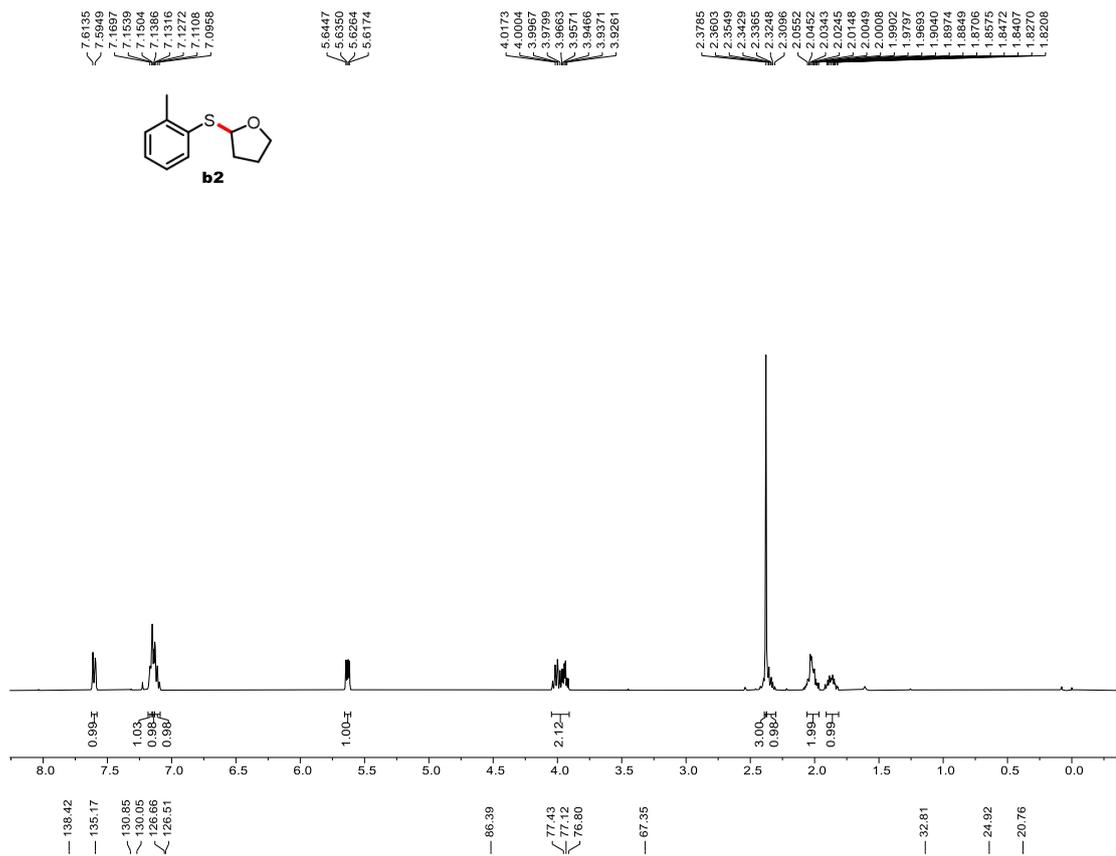
^1H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, $J = 8.1$ Hz, 1H), 7.82 (d, $J = 8.0$ Hz, 1H), 7.44 (dd, $J = 8.3, 7.1$ Hz, 1H), 7.34 (t, $J = 7.6$ Hz, 1H), 3.70 (t, $J = 6.3$ Hz, 2H), 3.15 (t, $J = 7.5$ Hz, 2H), 2.63 (1H), 1.98 (p, $J = 7.5$ Hz, 2H), 1.71 (dt, $J = 13.2, 6.5$ Hz, 2H).

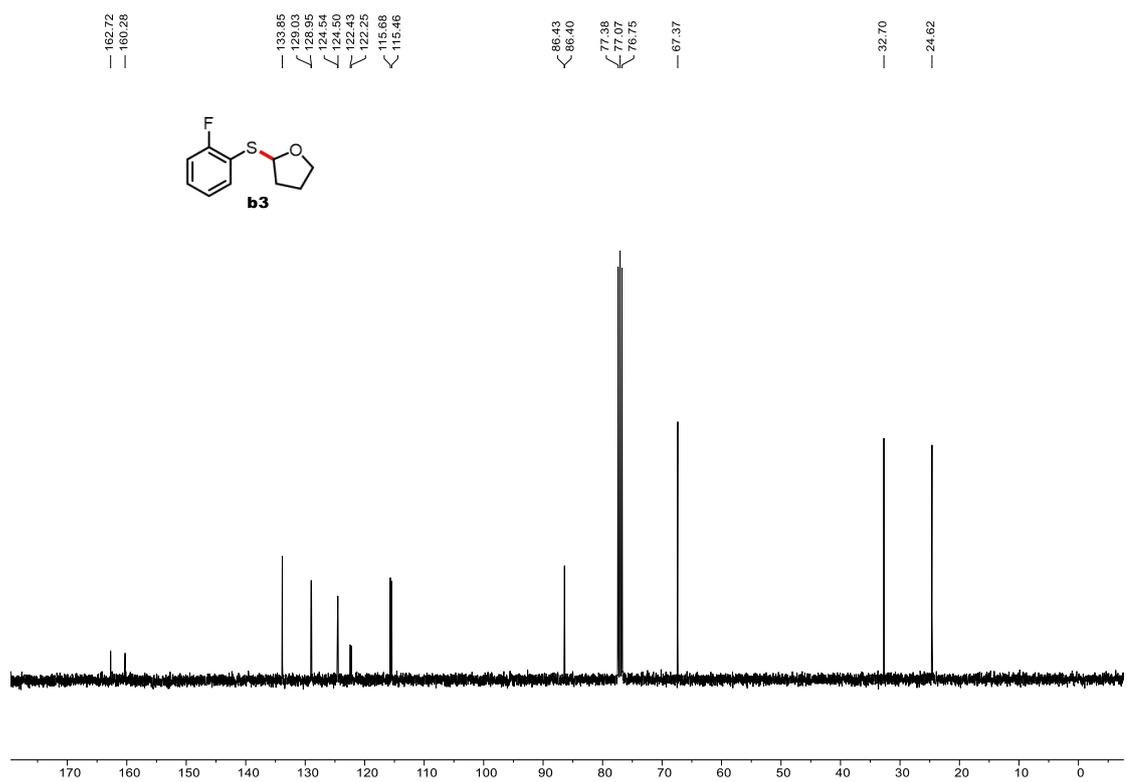
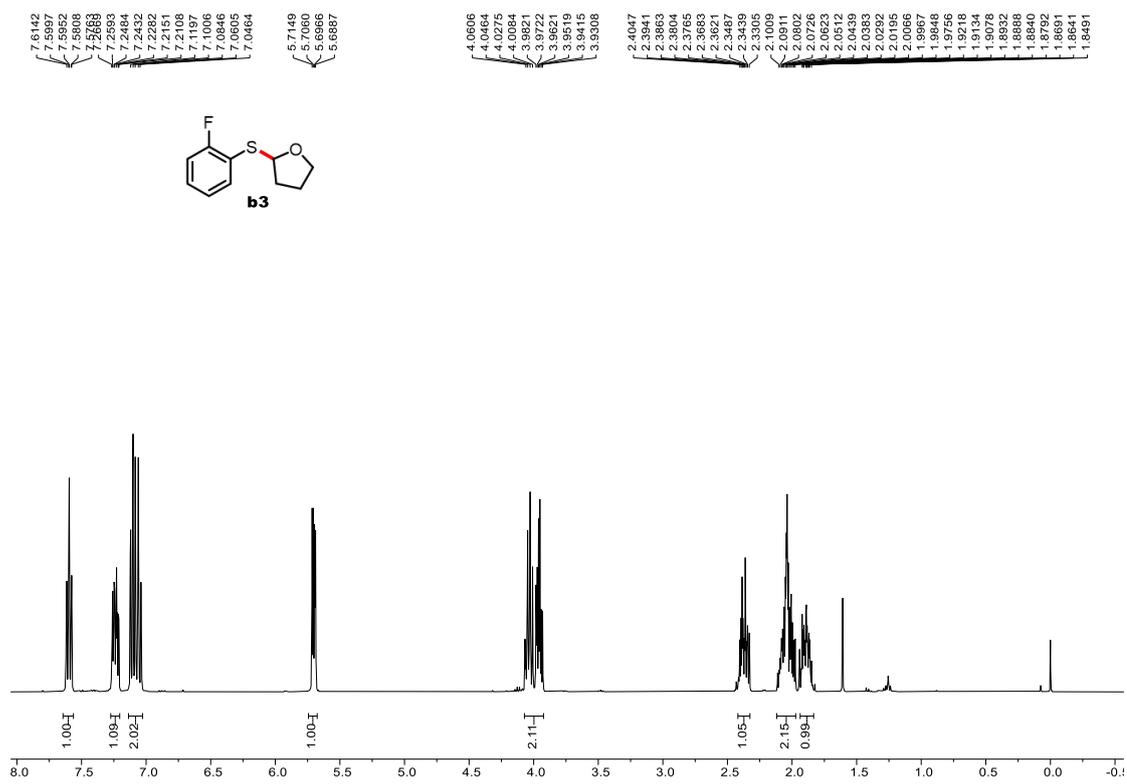
^{13}C NMR (101 MHz, CDCl_3) δ 172.2, 153.0, 135.0, 126.0, 124.8, 122.4, 121.5, 62.0, 33.8, 32.0, 25.7.

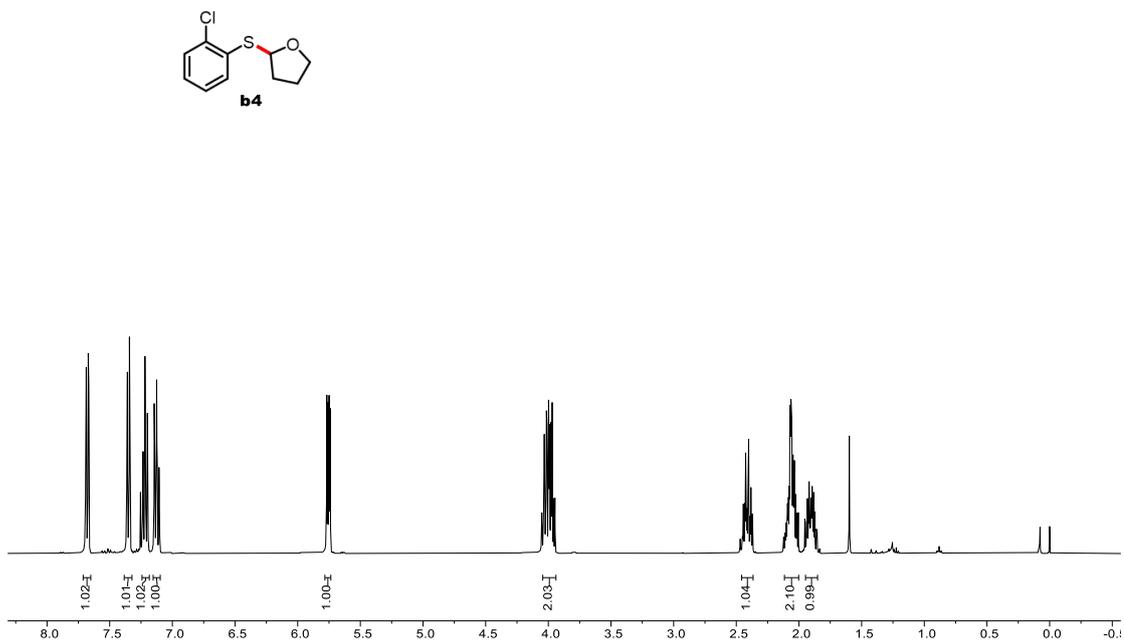
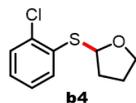
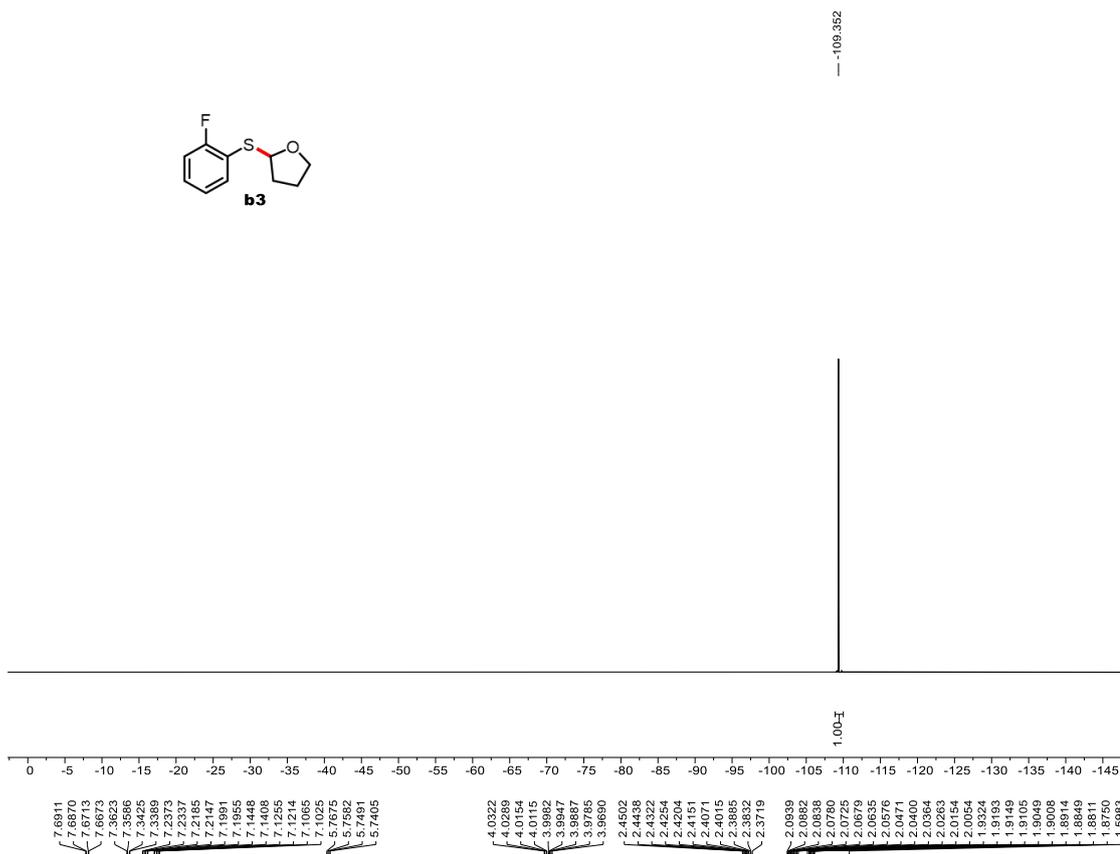
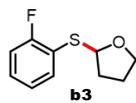
HRMS (ESI) calculated for $\text{C}_{11}\text{H}_{13}\text{NOS}$ $[\text{M}+\text{H}]^+$: 208.0791; Found: 208.0795

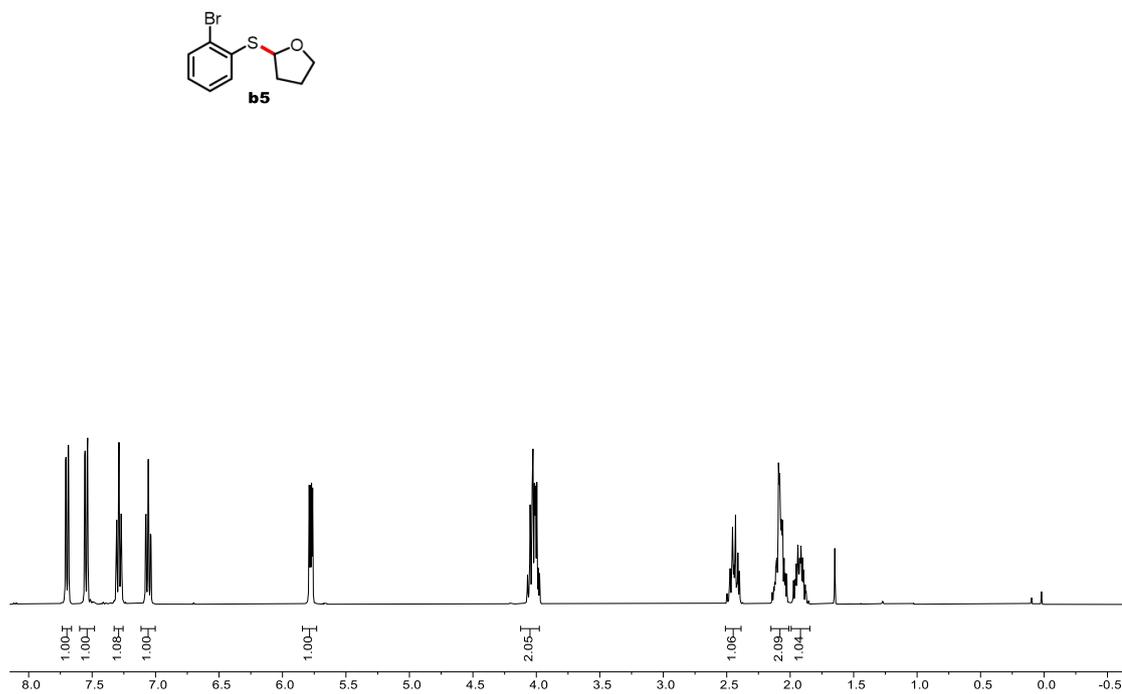
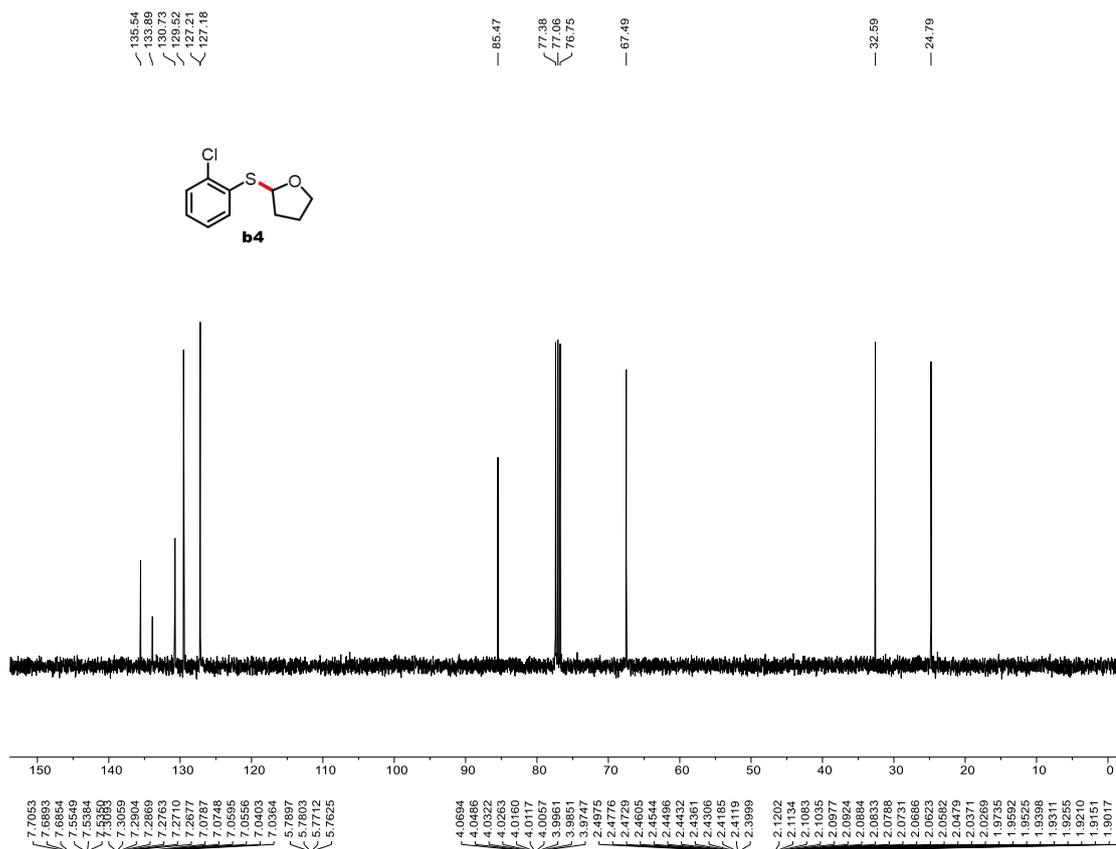
3. NMR spectra for the products







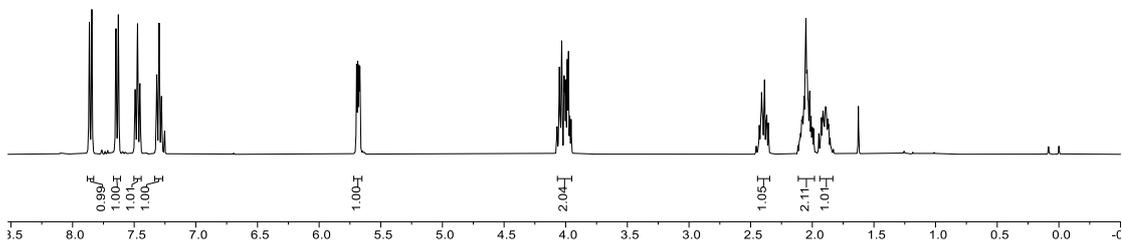
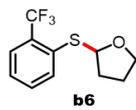
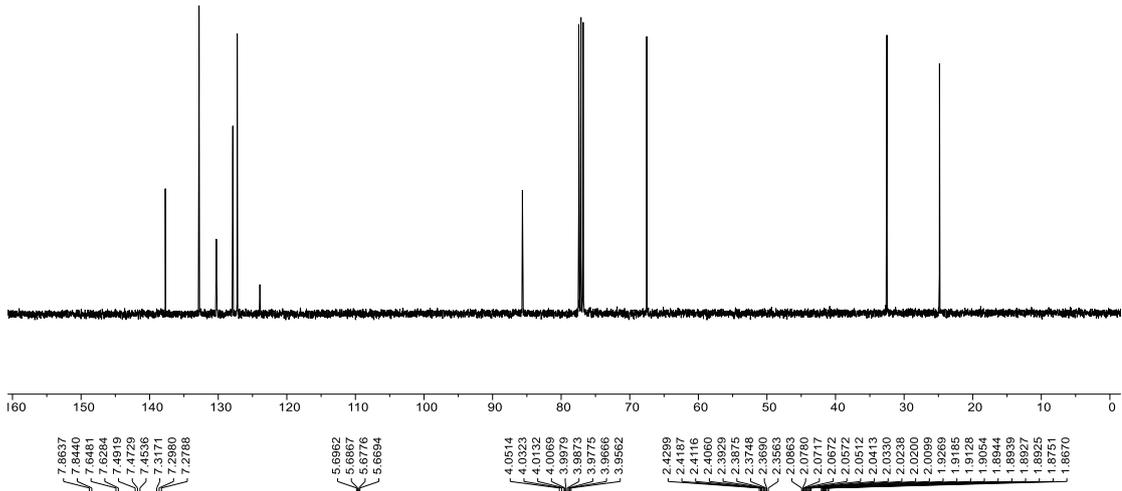
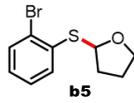


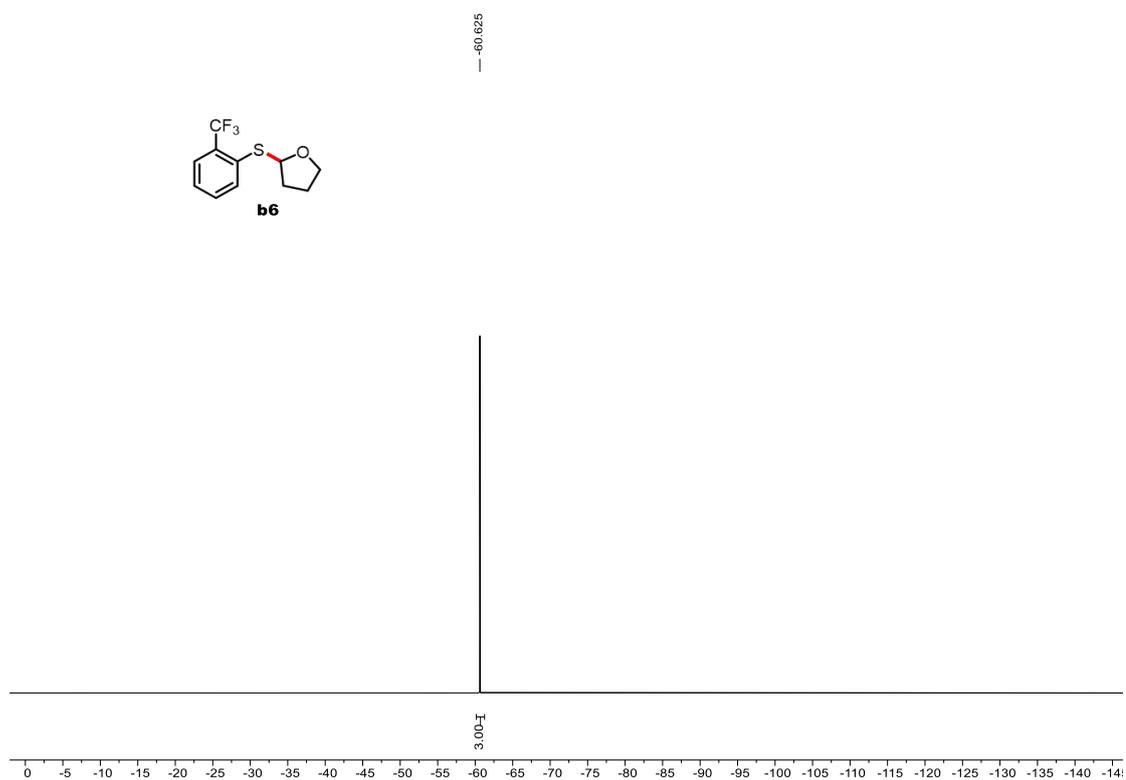
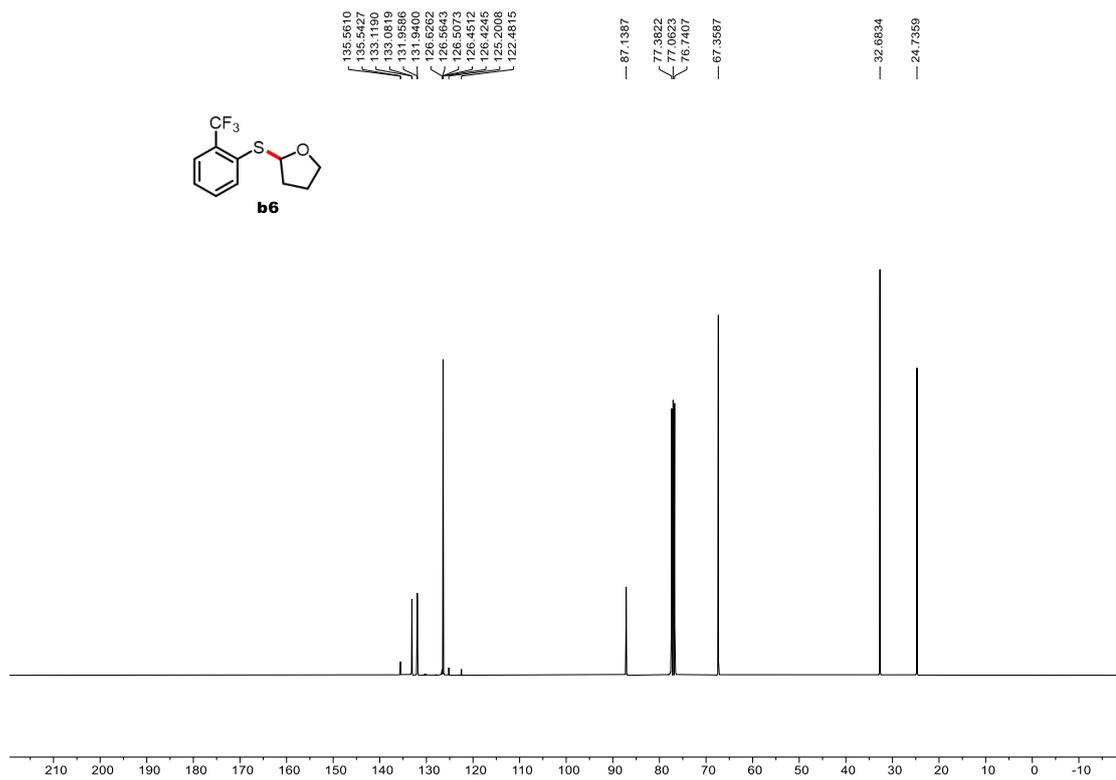


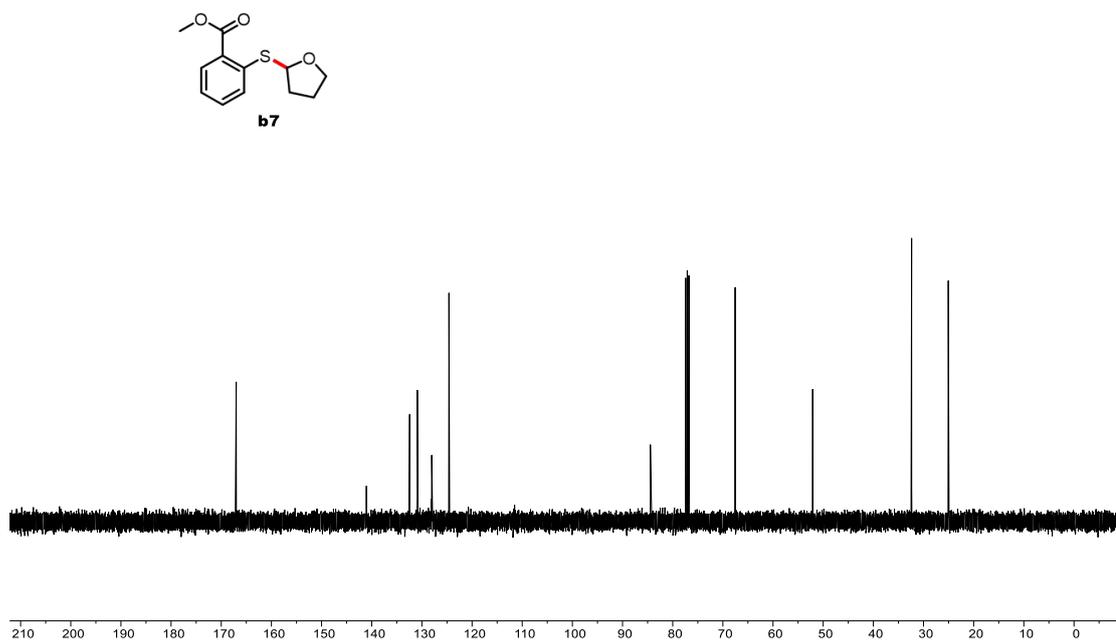
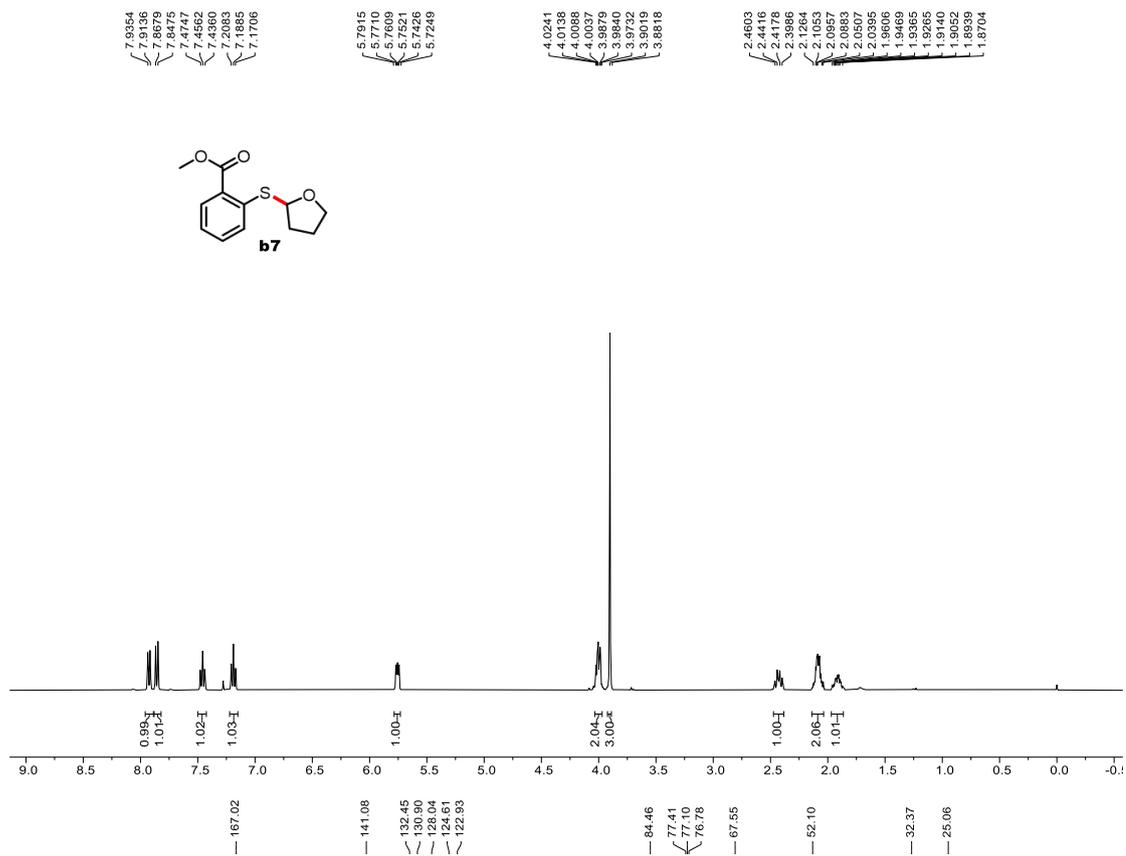
137.72
132.80
131.76
127.45
127.23
123.92

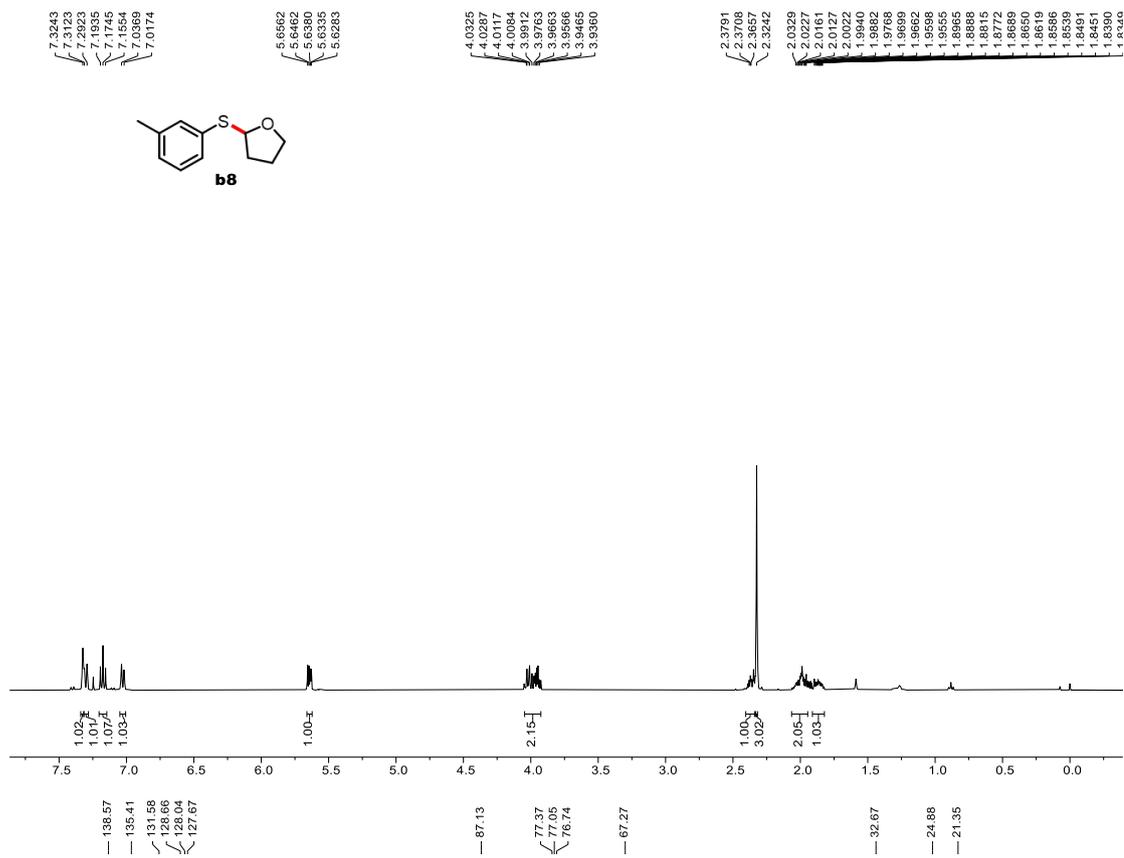
85.64
77.43
77.00
76.80
67.54

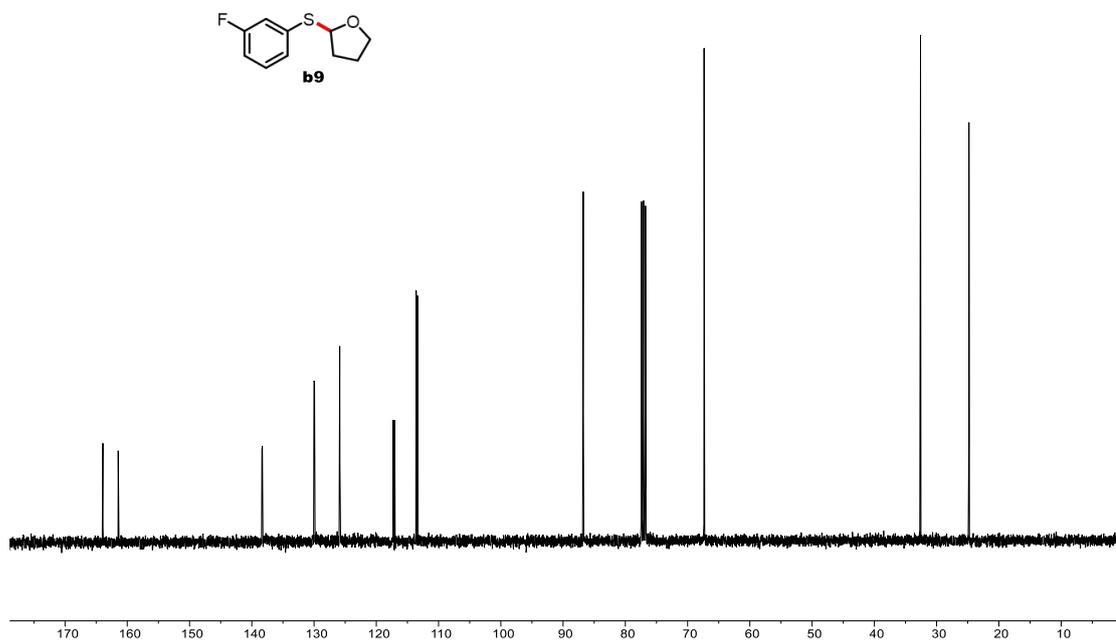
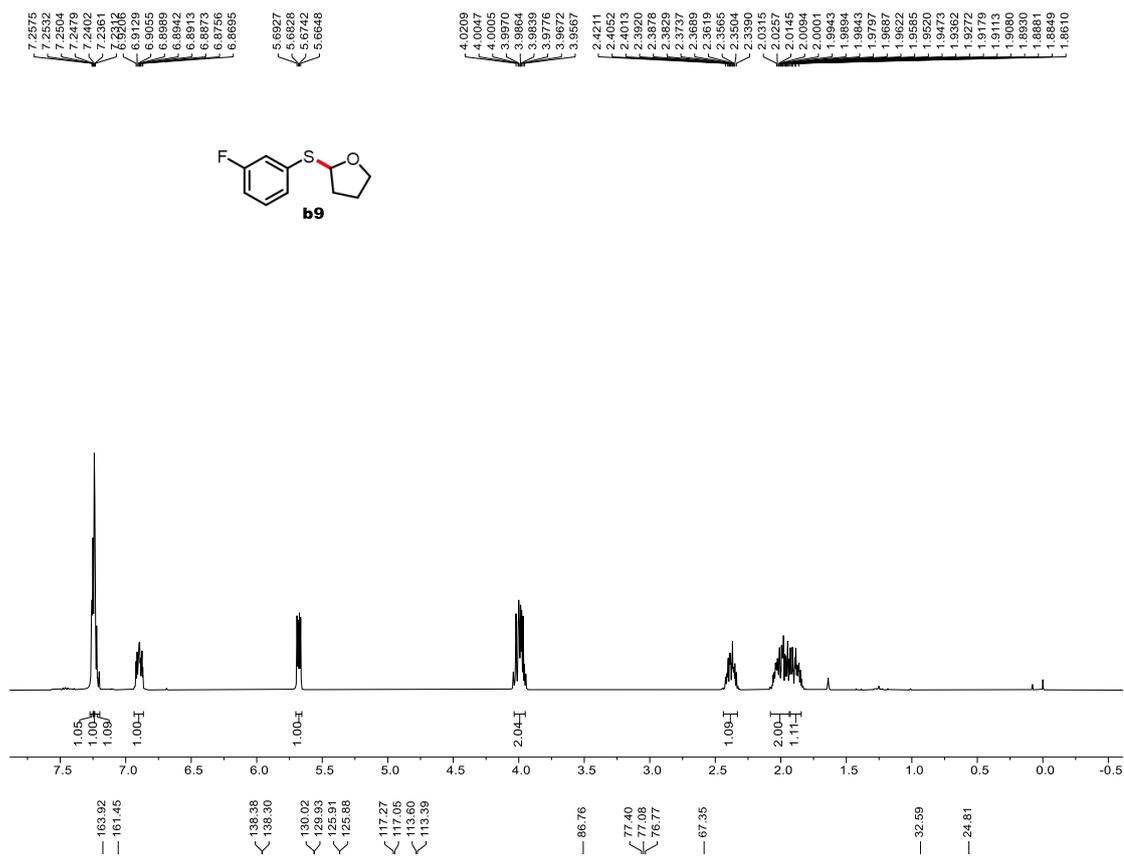
32.51
24.84

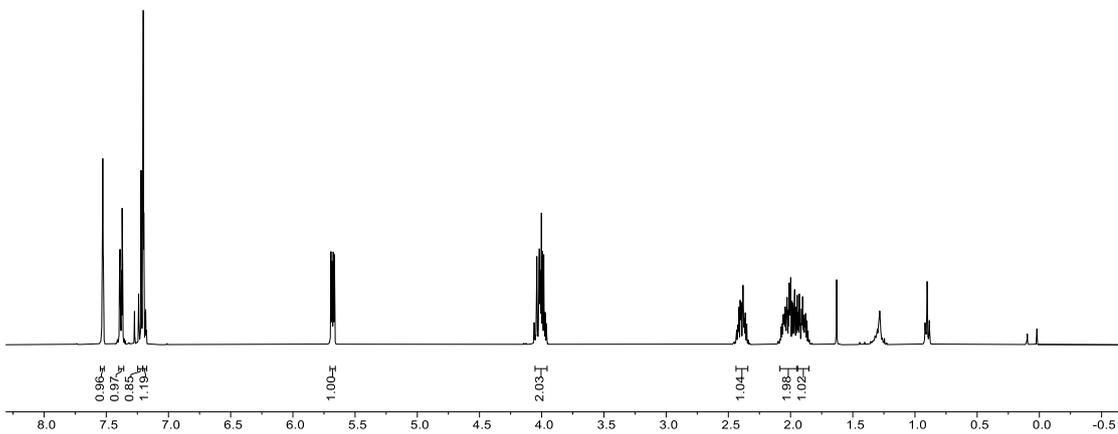
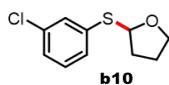
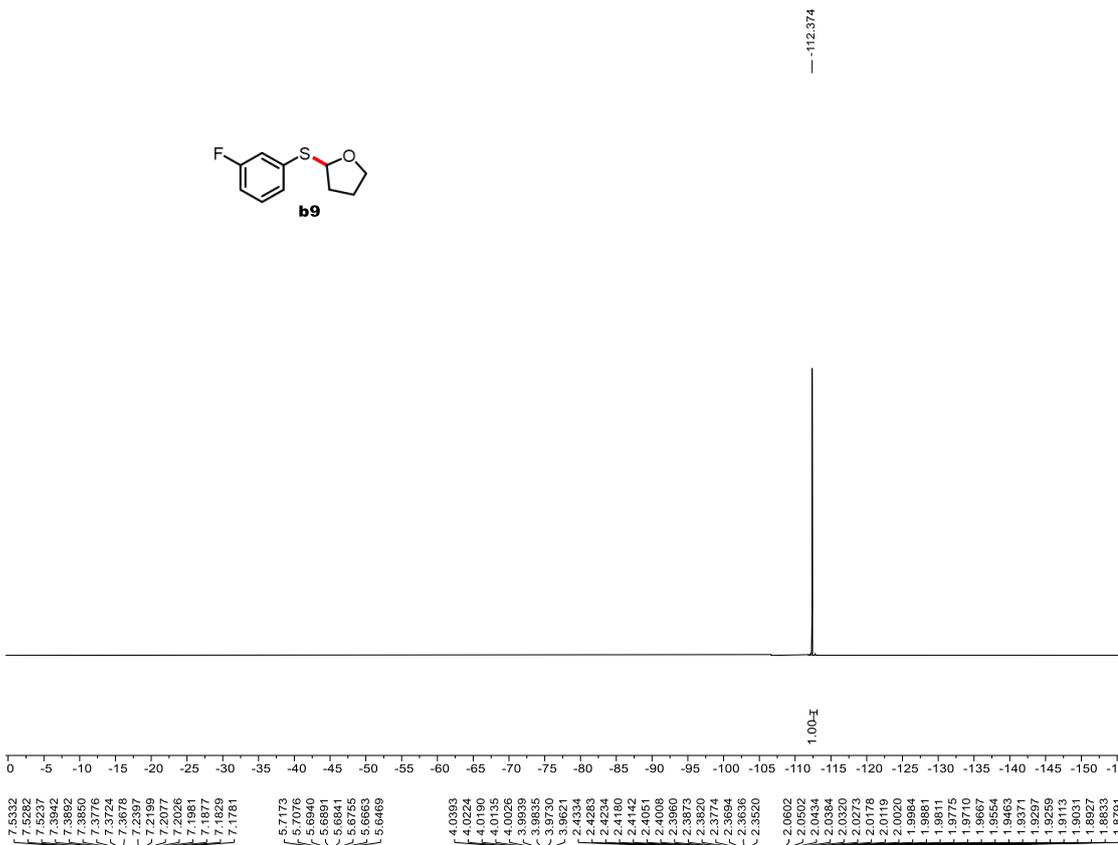
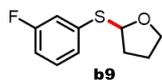








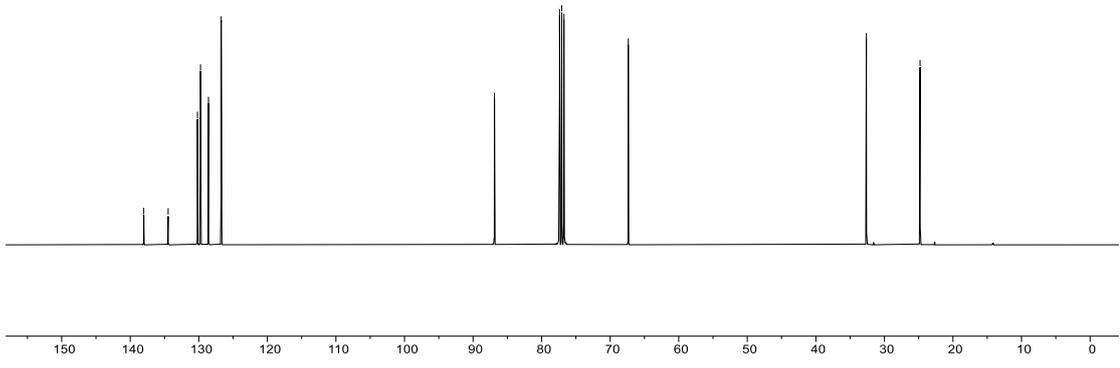
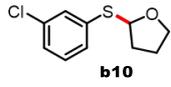




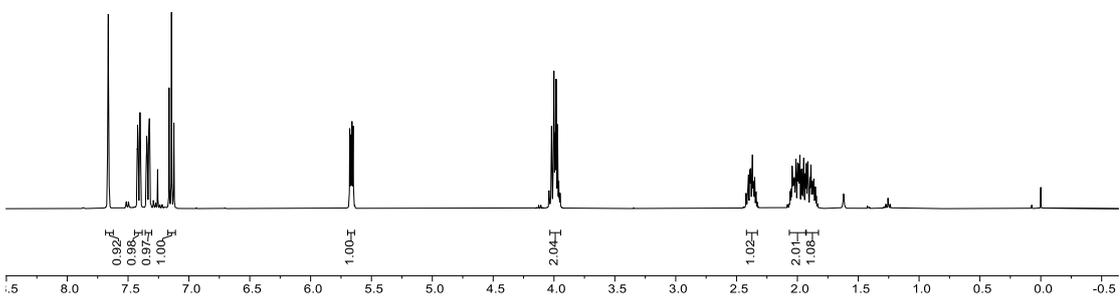
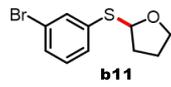
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134.47
130.20
128.75
128.59
126.74

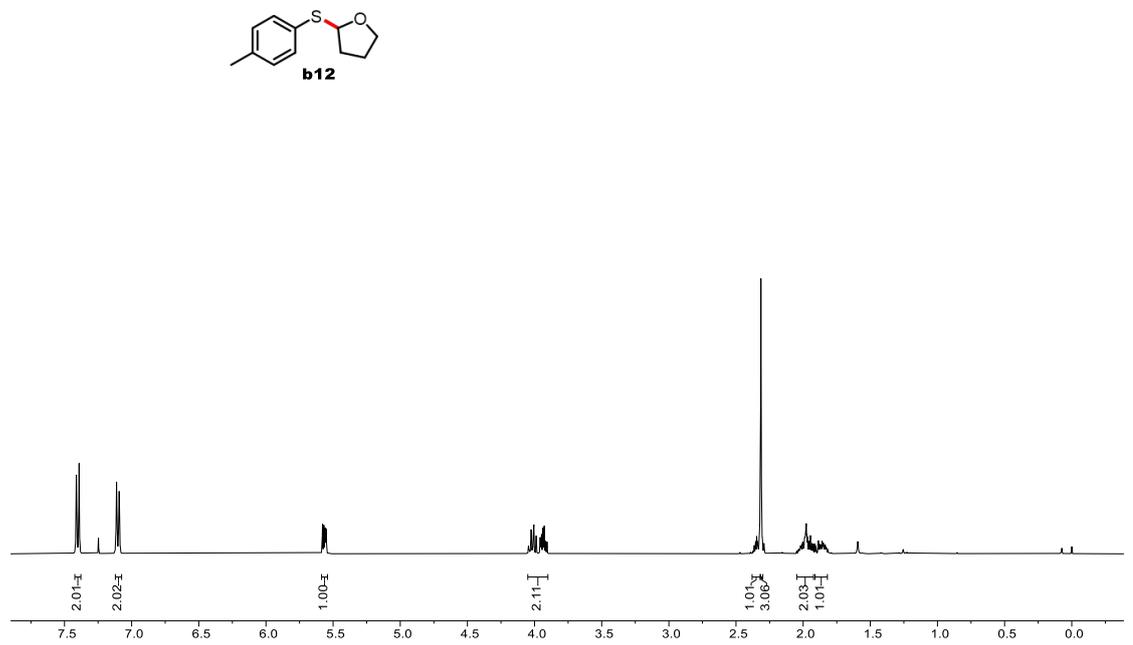
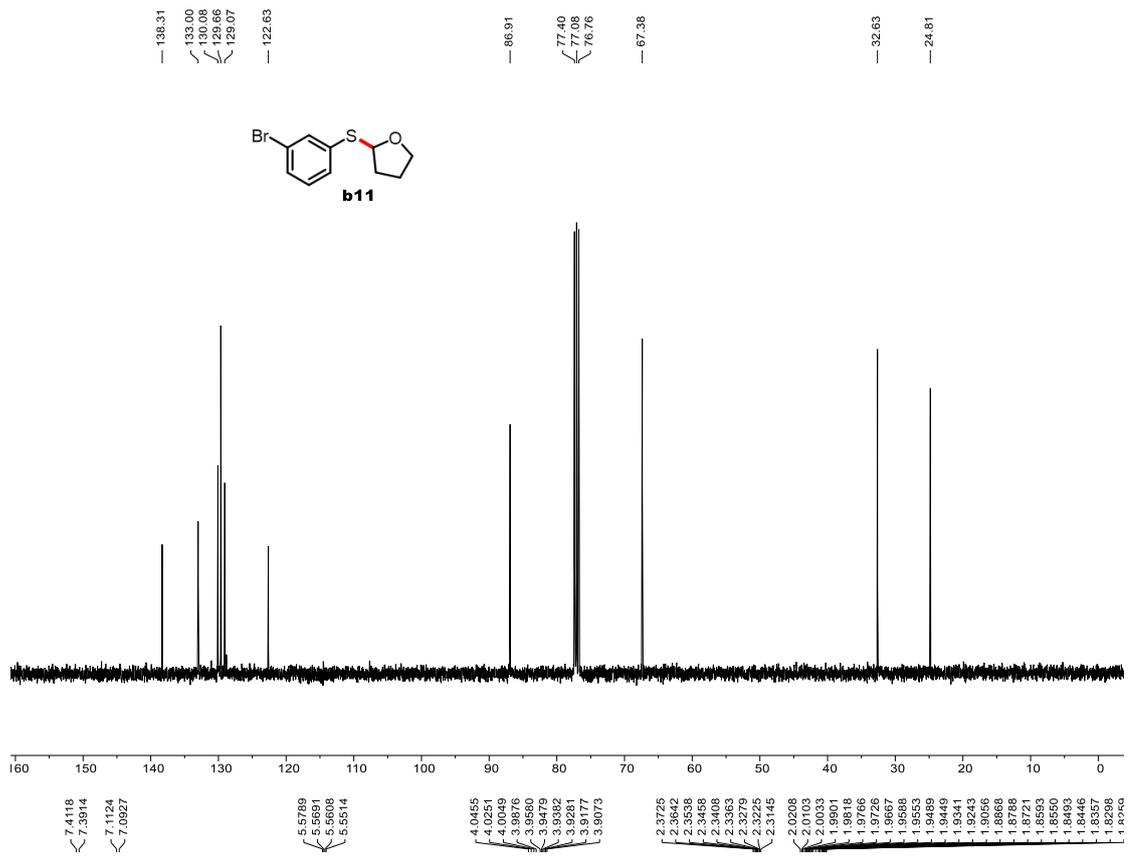
86.87
77.38
77.06
76.74
67.35

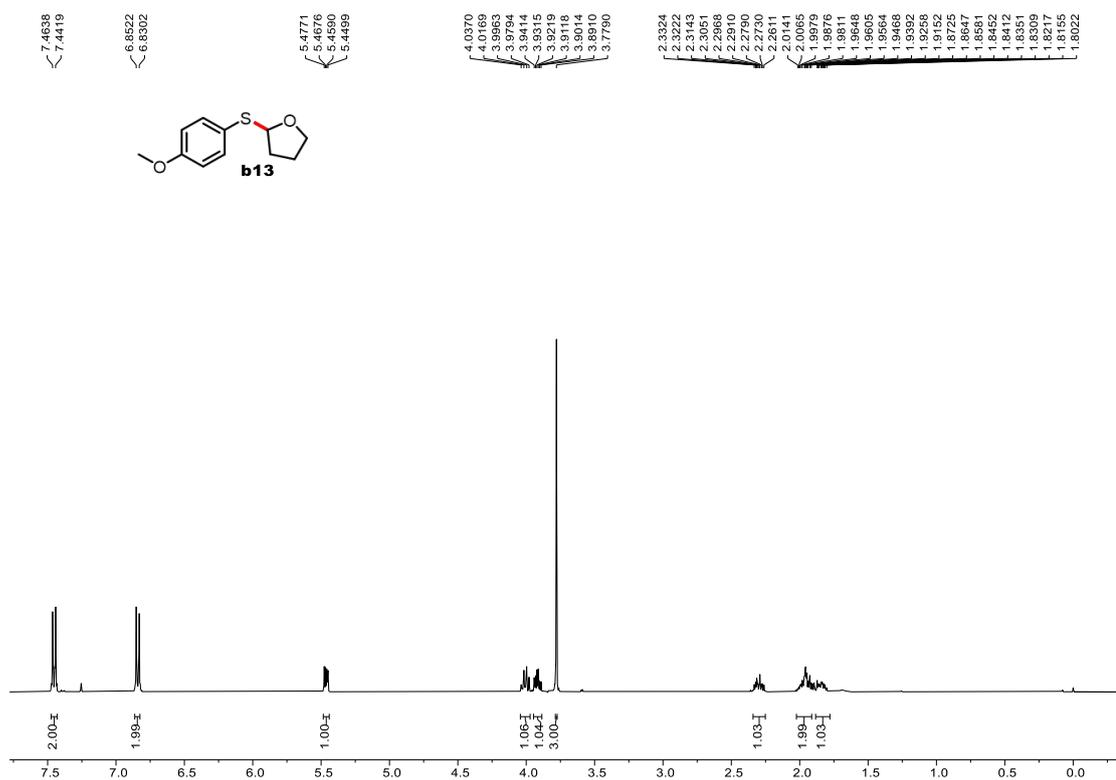
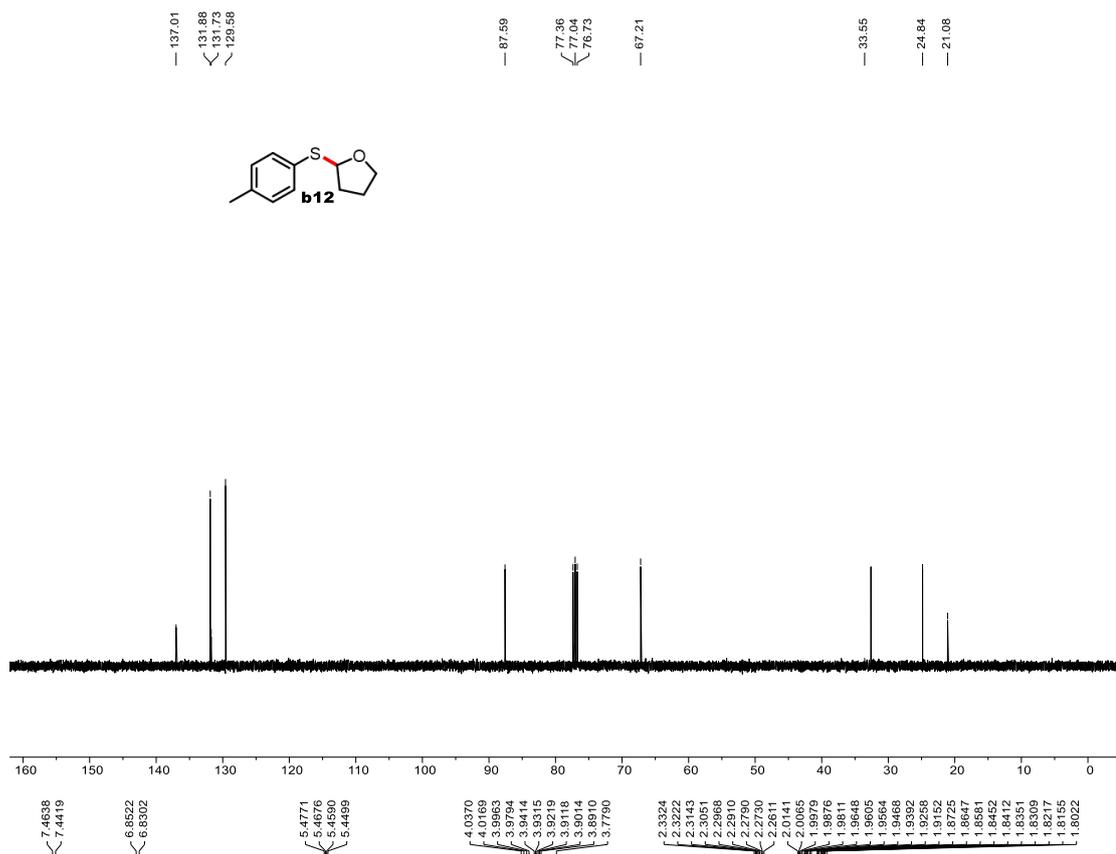
32.63
24.80

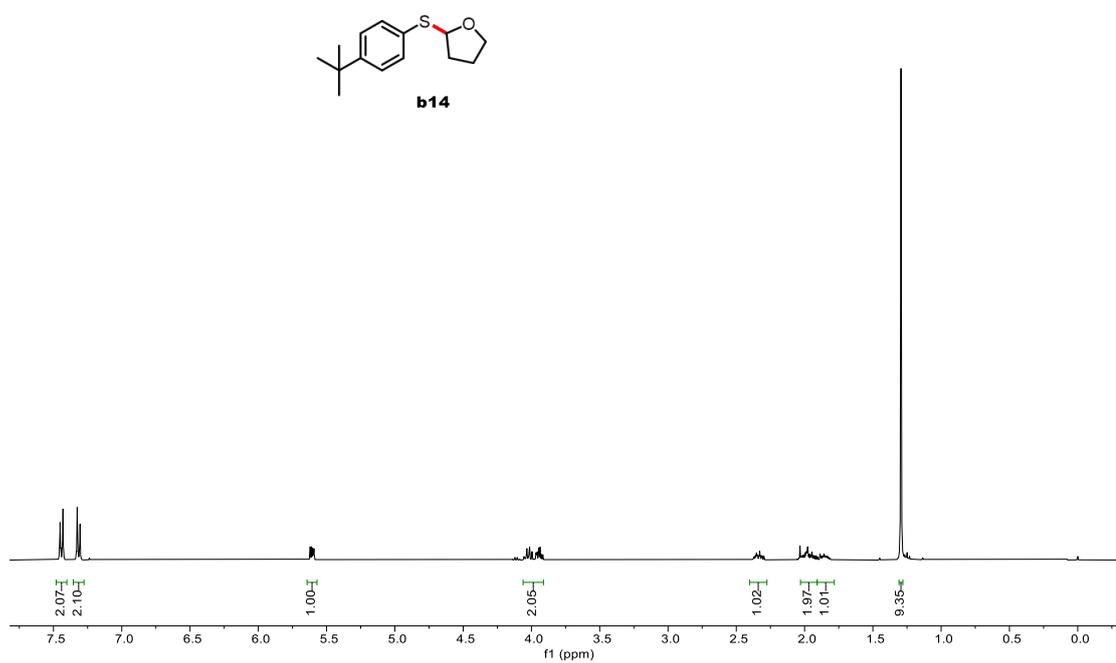
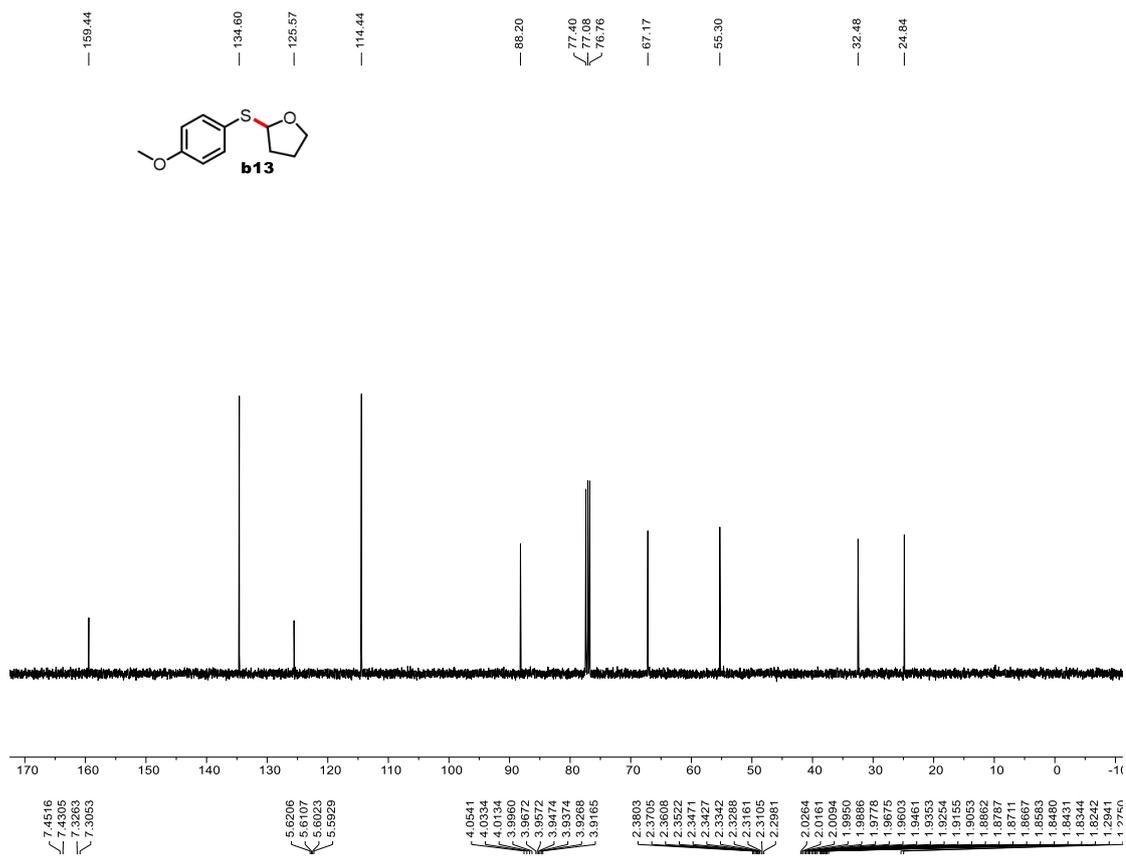


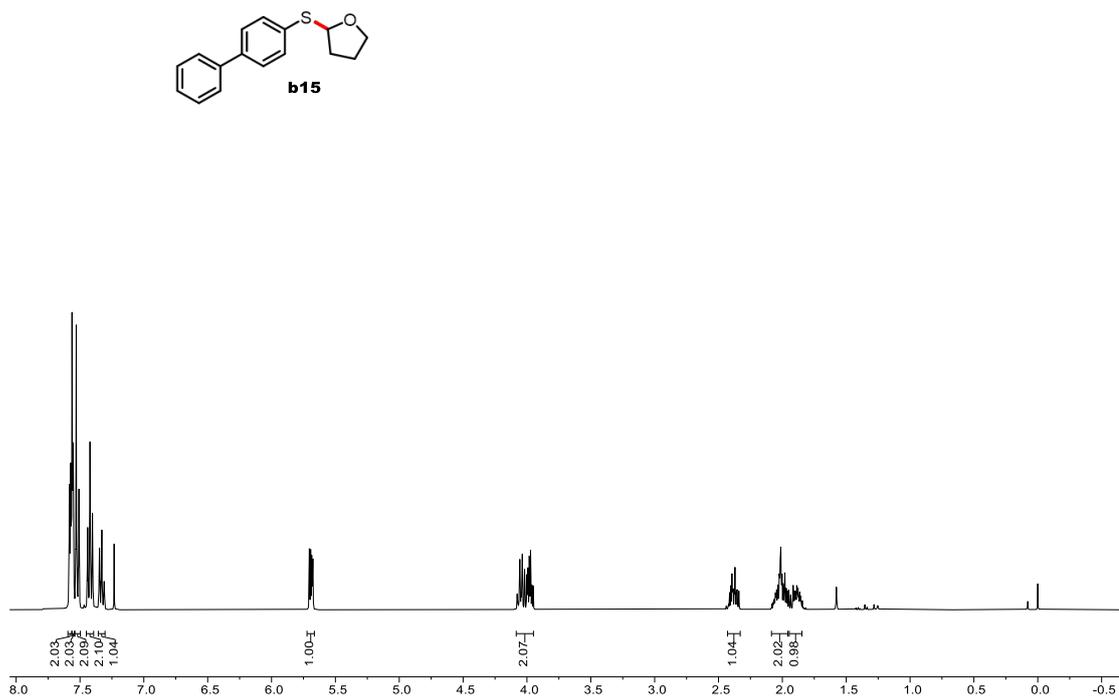
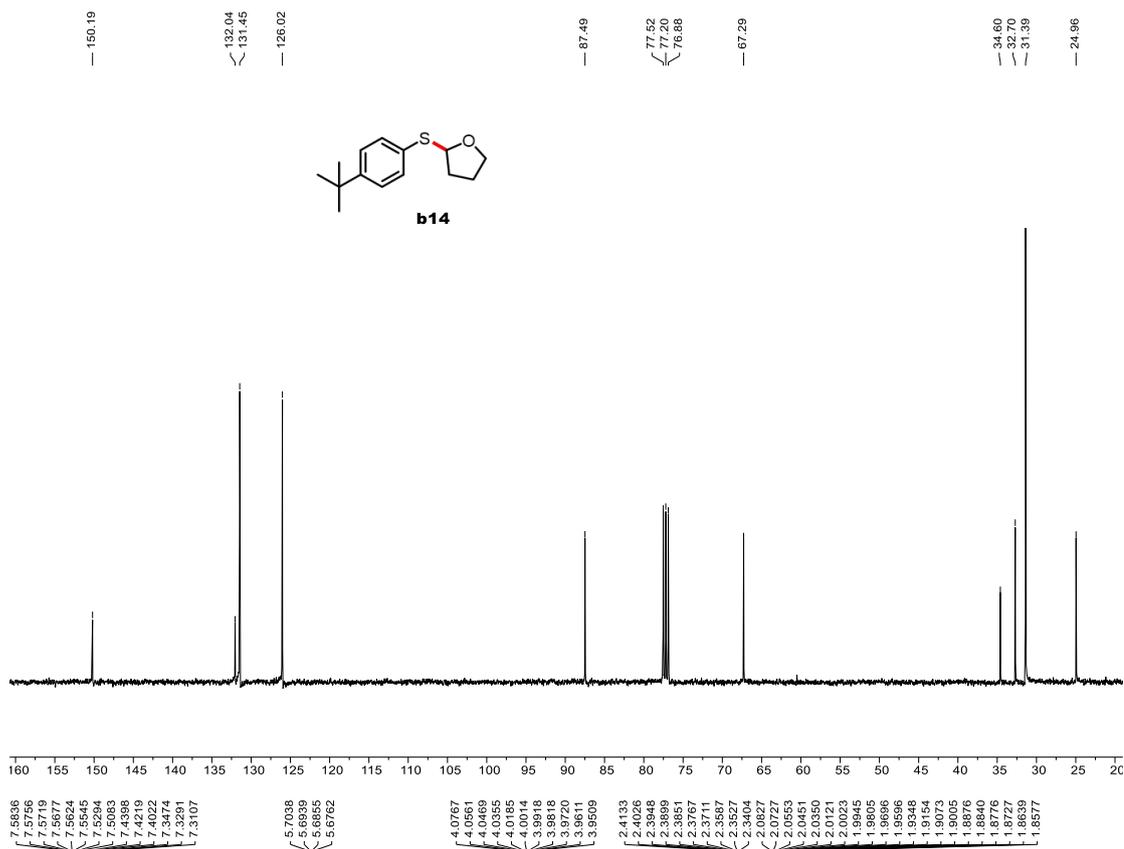
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7.4219
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7.3470
7.3247
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7.1237
5.6783
5.6686
5.6600
5.6506
4.0203
4.0048
4.0007
3.9833
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3.9533
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3.9500
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2.4015
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2.3881
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2.3746
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2.3619
2.3569
2.3518
2.3468
2.3292
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2.0433
2.0330
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2.0228
2.0170
2.0118
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1.8624
1.8577
1.8522



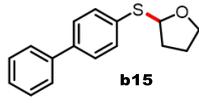








140.57
139.76
134.80
131.41
128.82
127.54
127.37
127.00



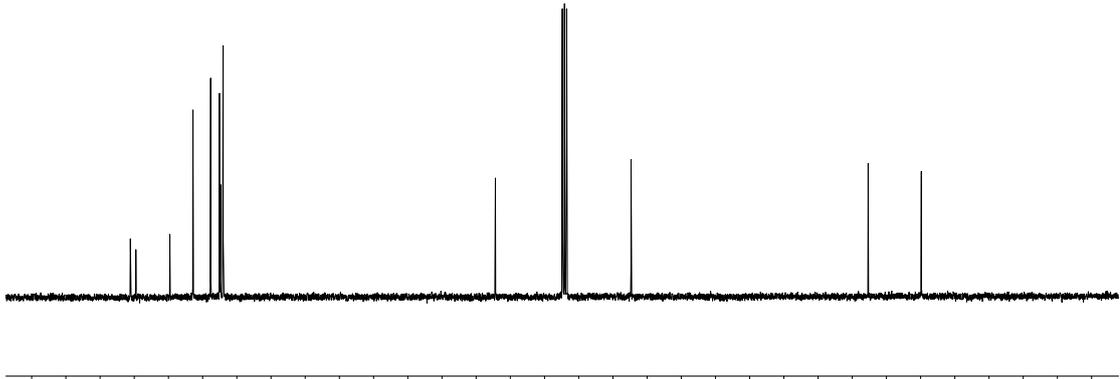
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77.40
76.98
76.77

87.95

32.67

24.90



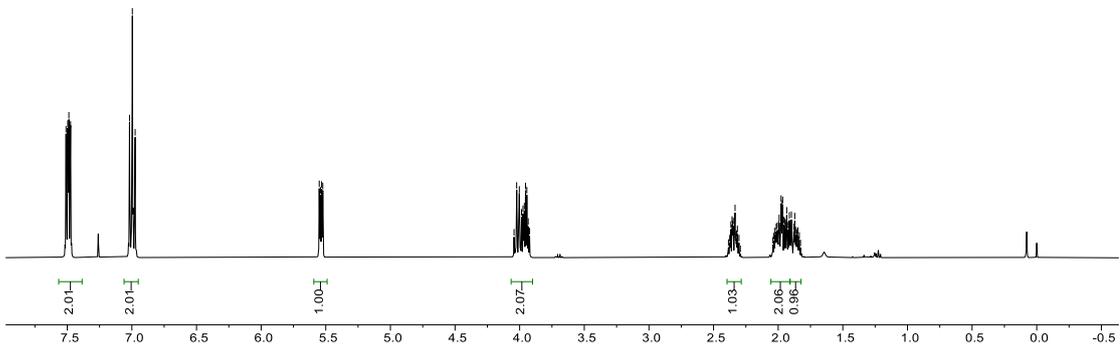
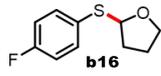
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7.4876
7.4744

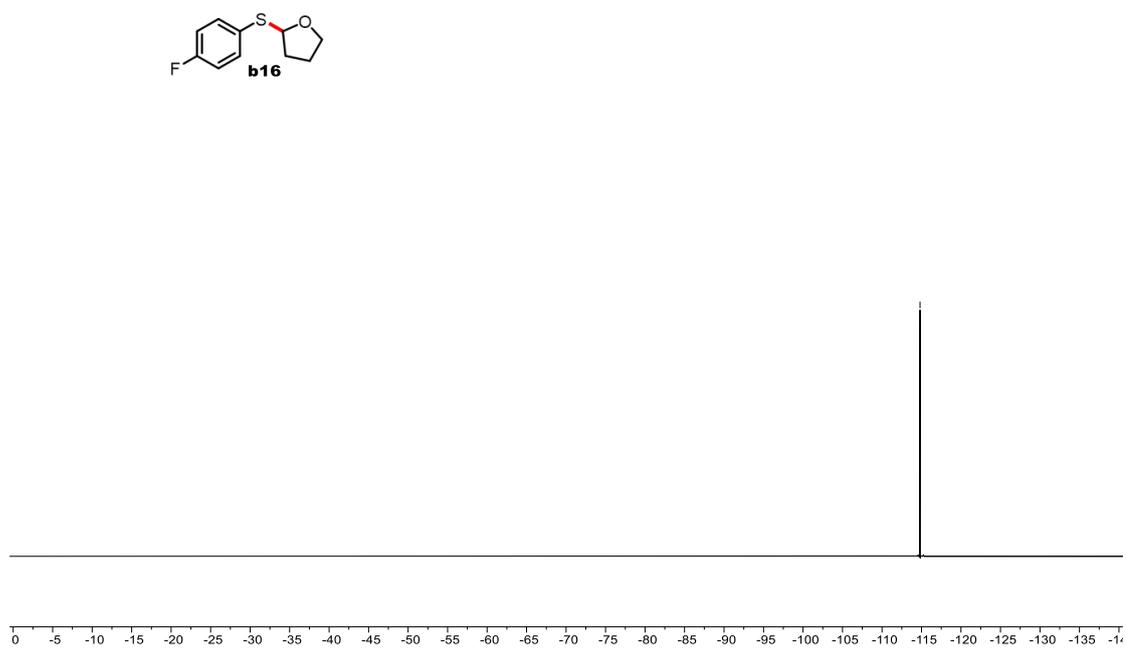
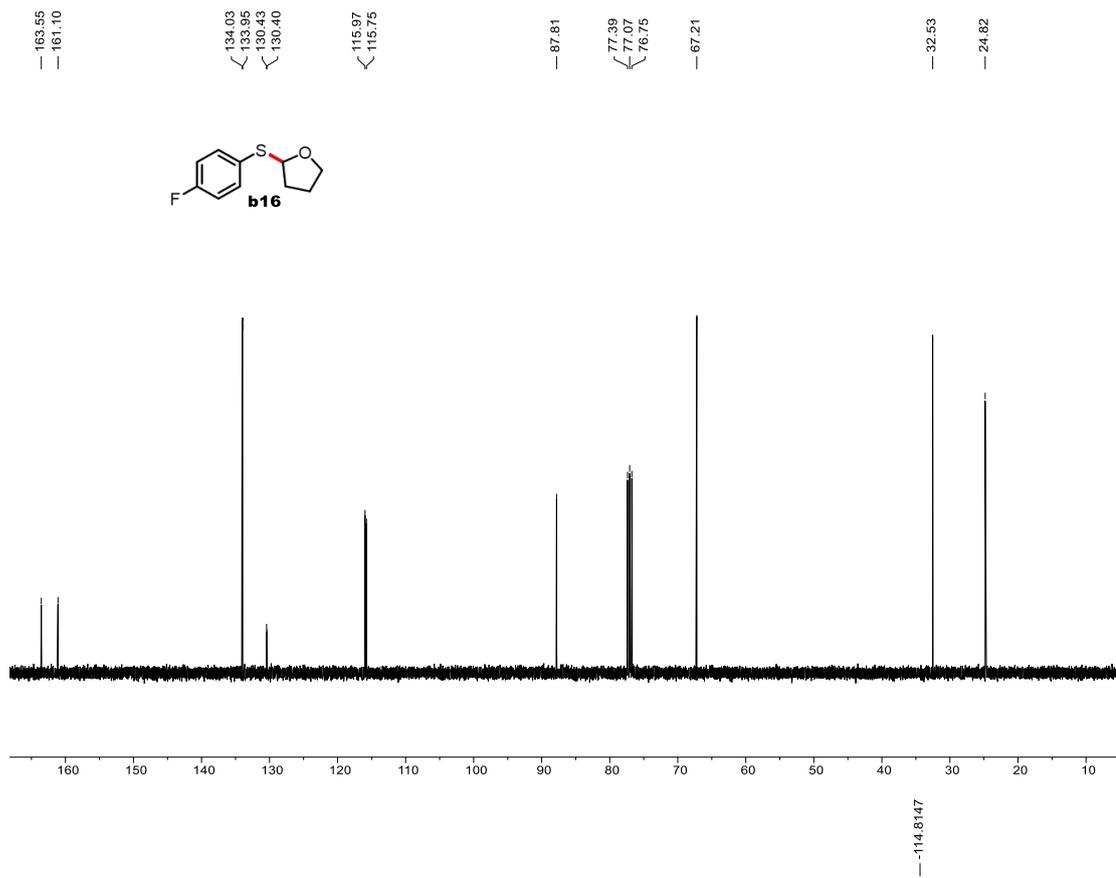
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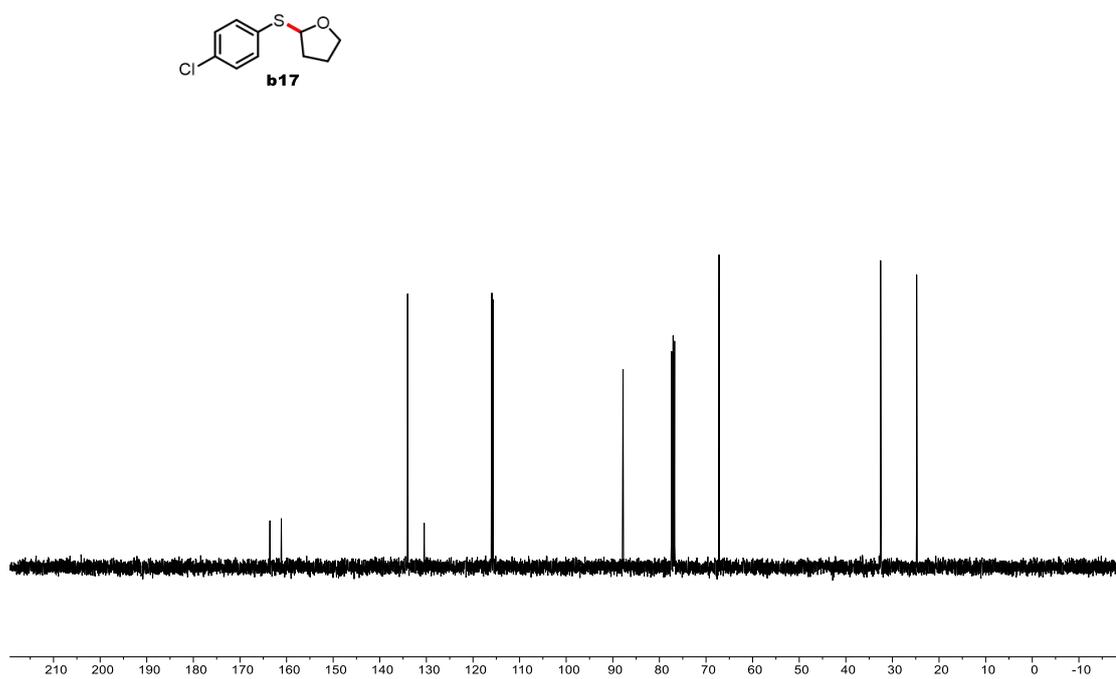
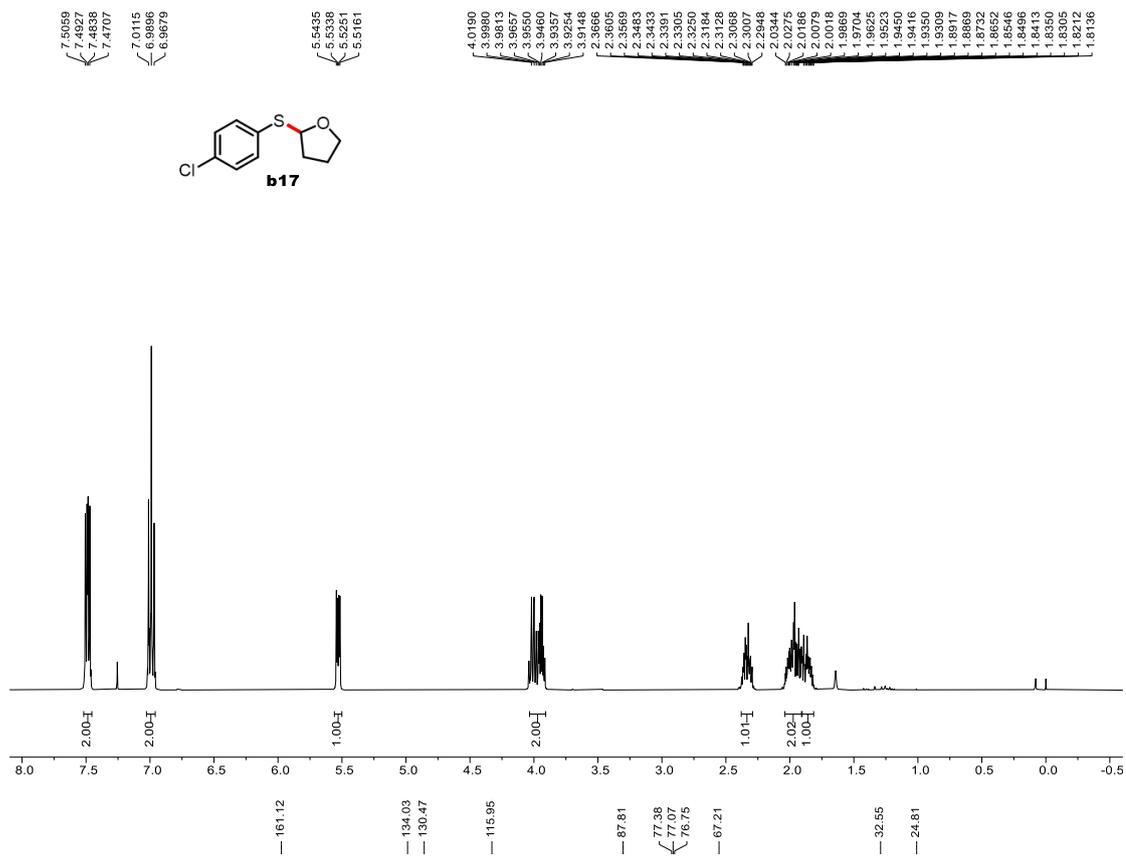
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5.5322
5.5231

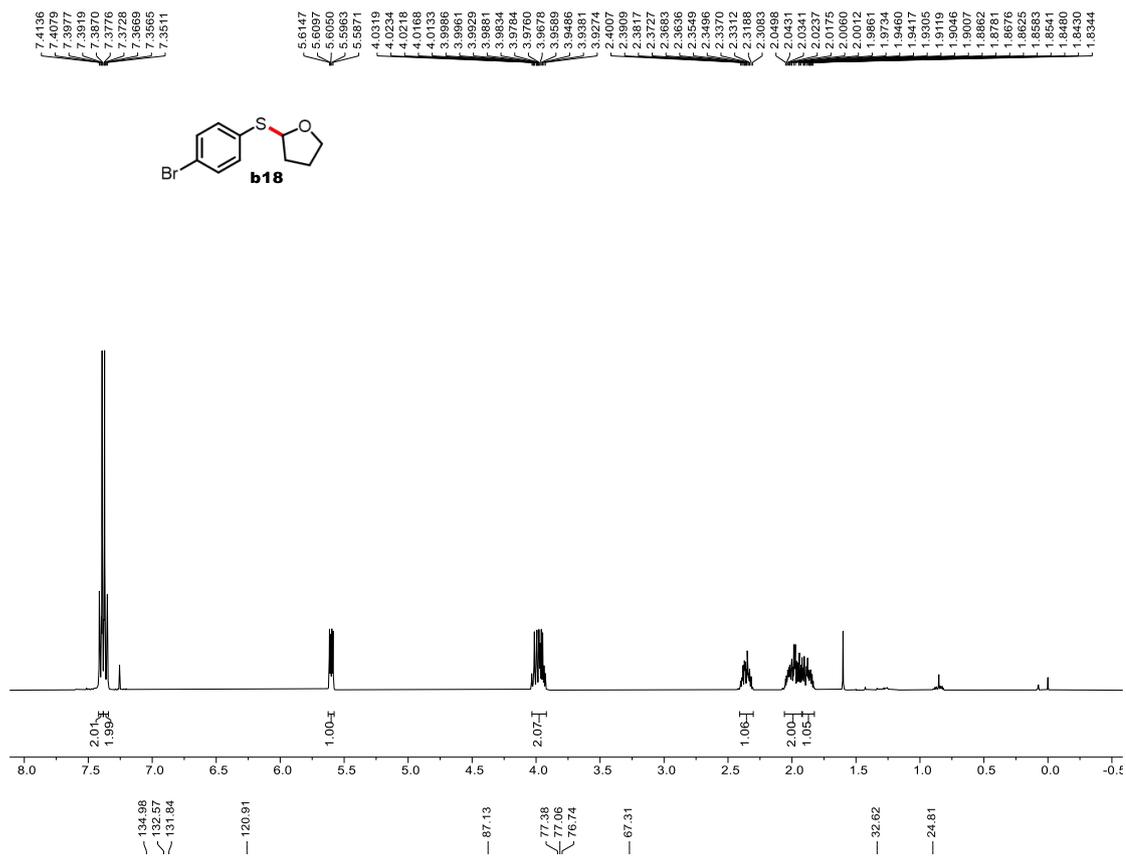
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3.9146
3.9060
3.8967
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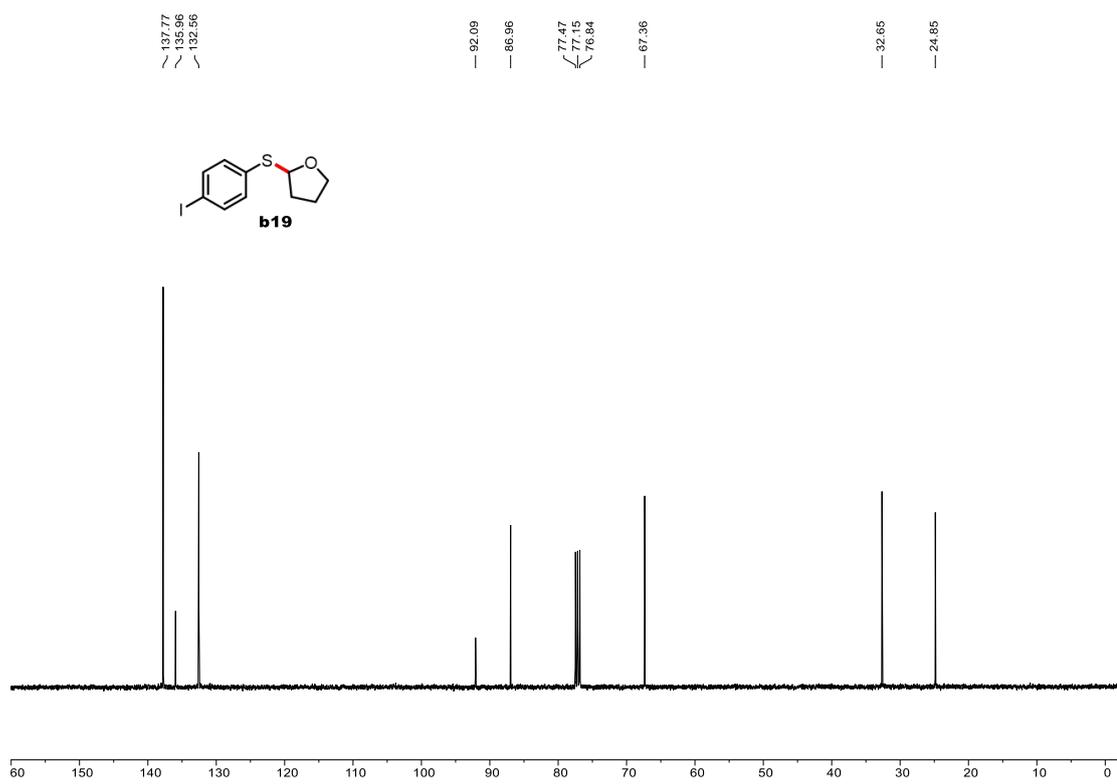
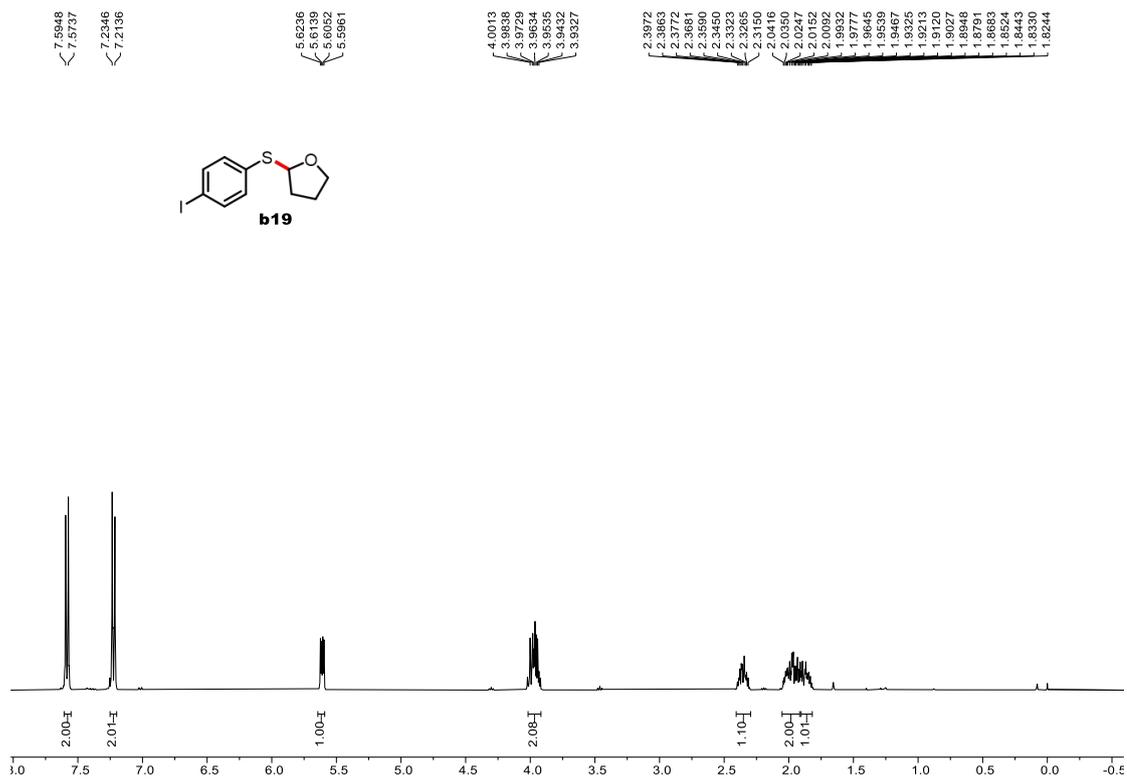
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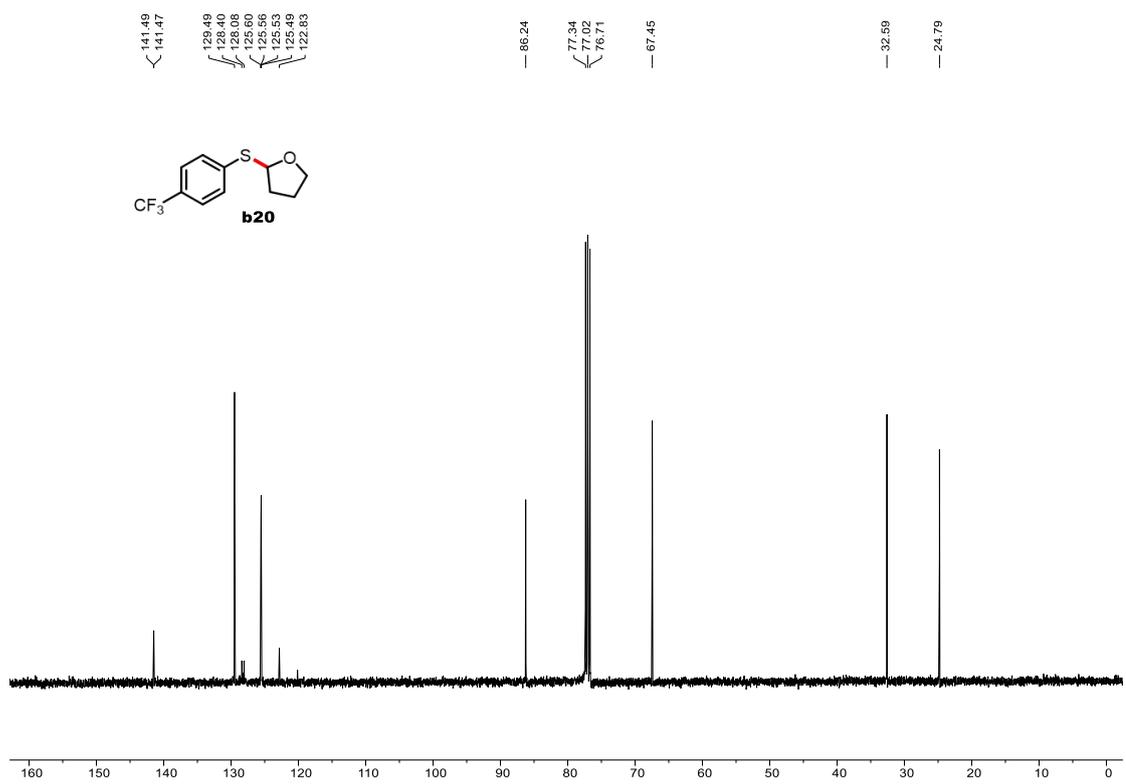
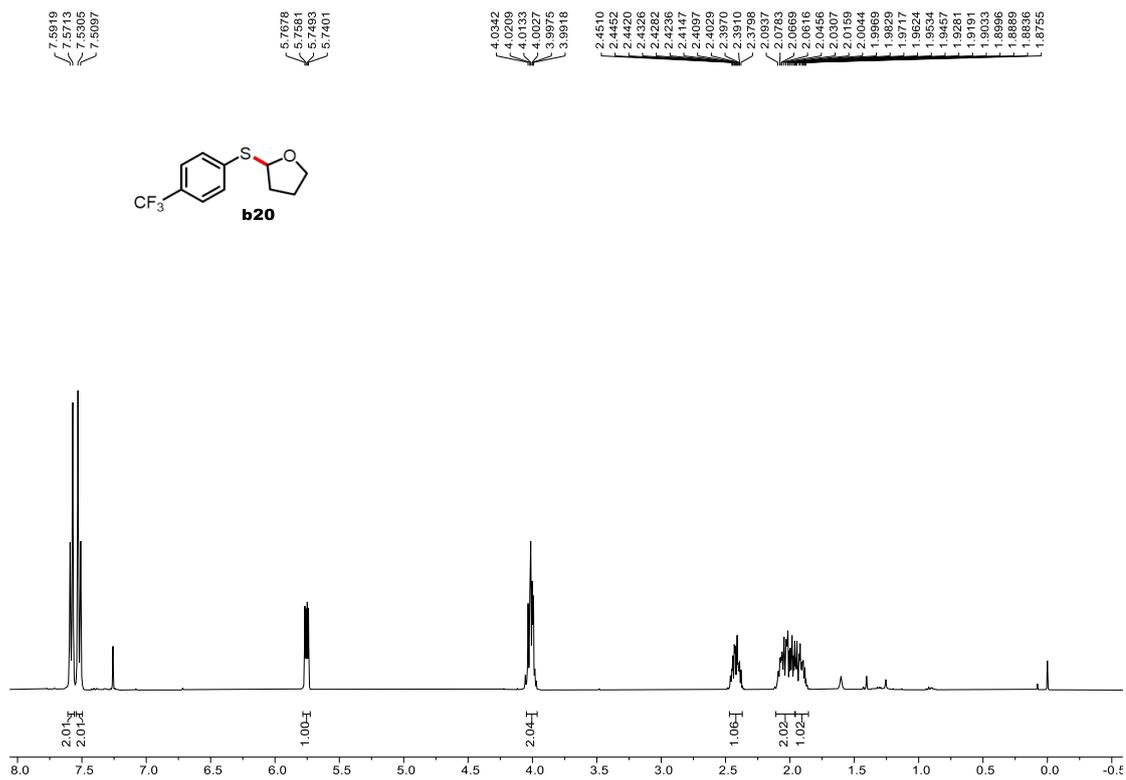


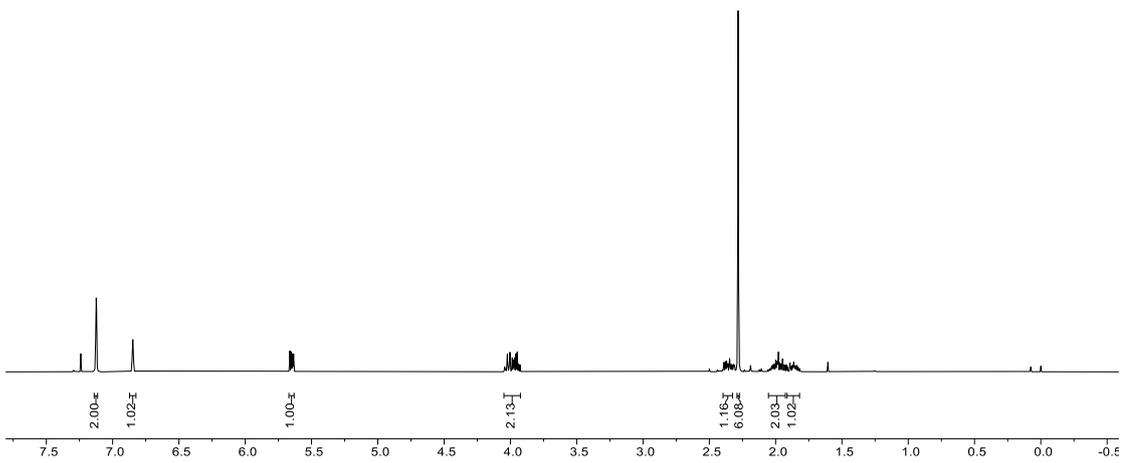
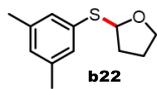
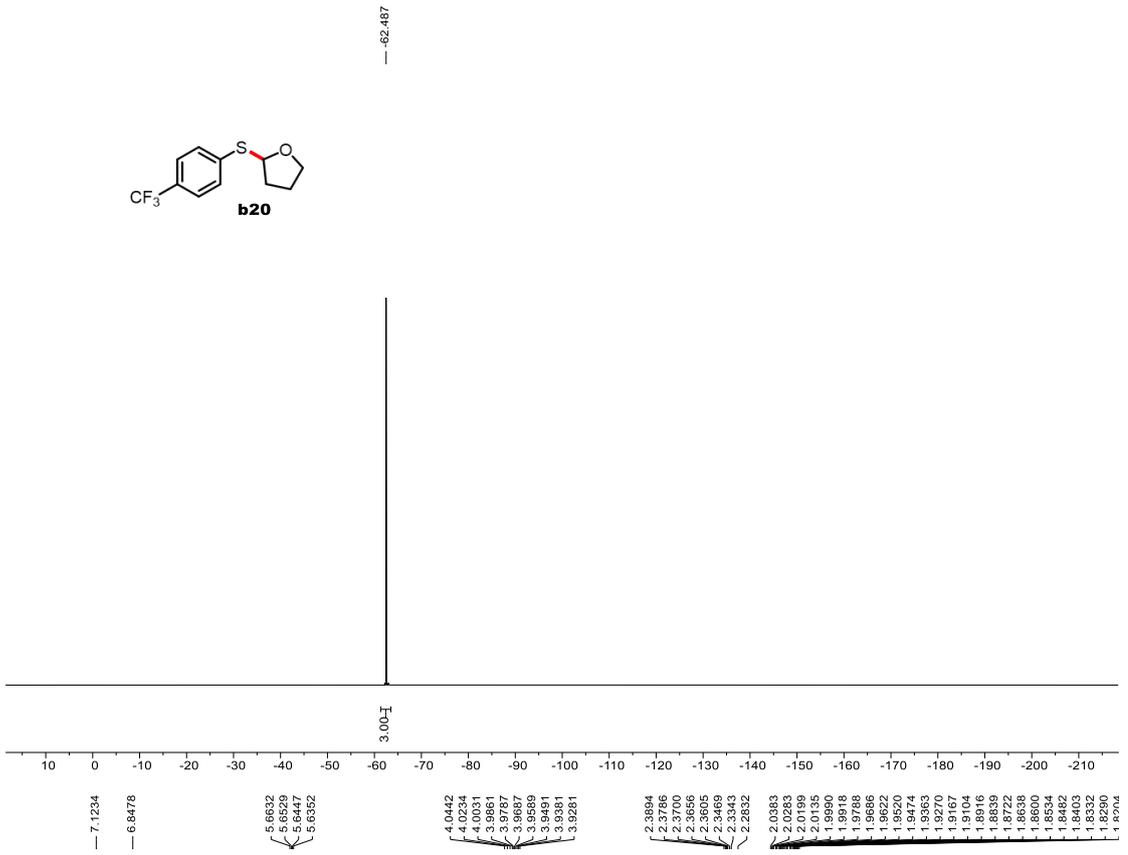
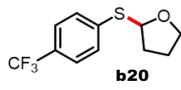


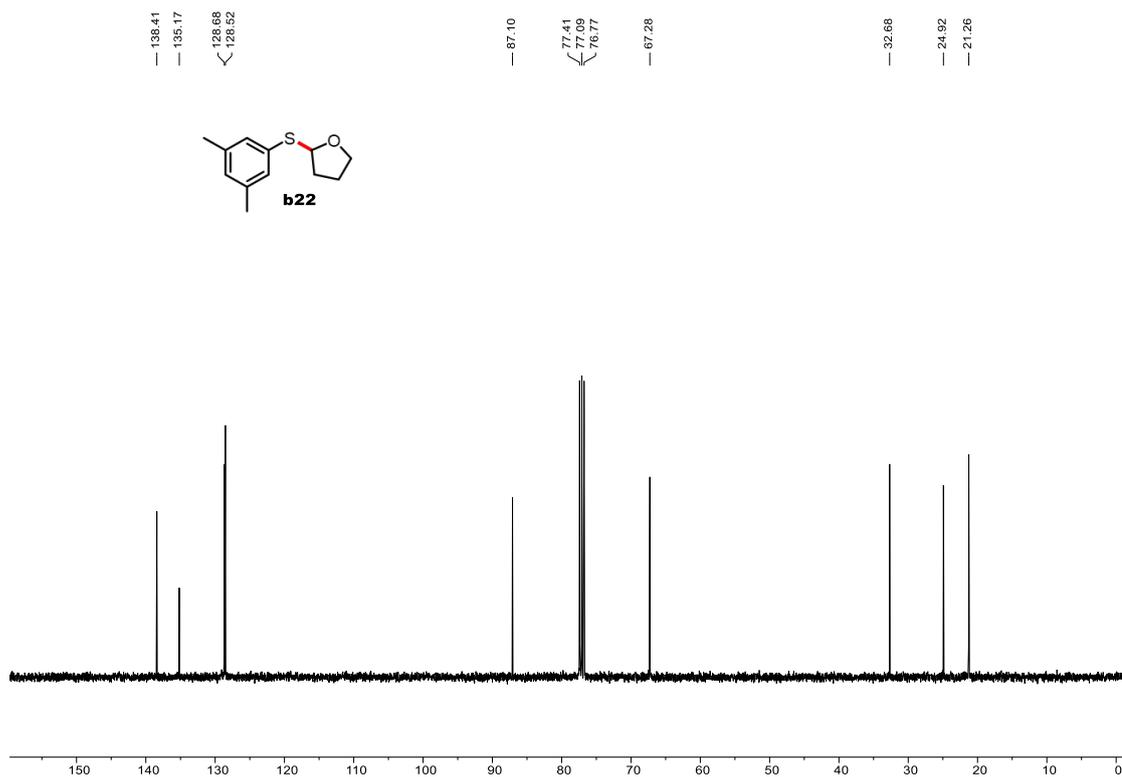












139.07
136.97
132.15
131.06
127.27

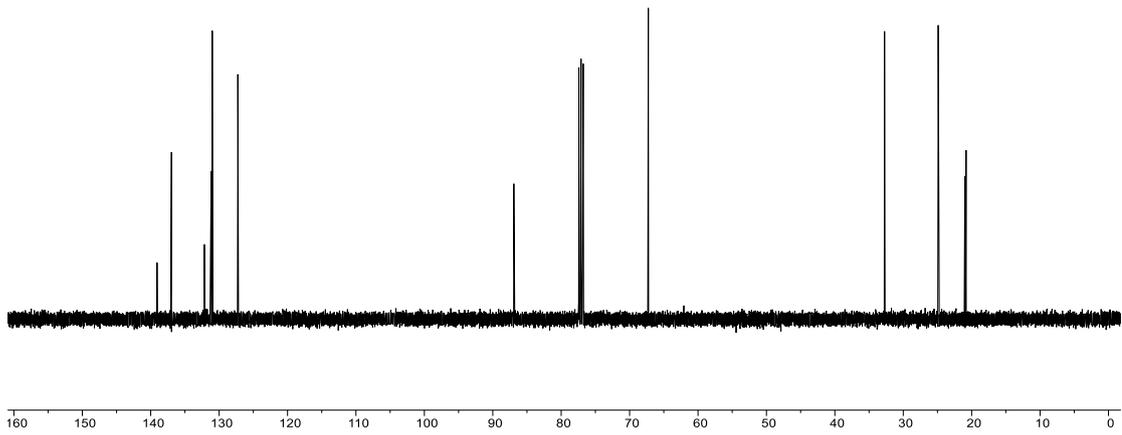
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77.14
77.09
76.78

67.26

32.73

24.89
21.00
20.61



6.9551

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5.3483
5.3358

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4.0414
4.0234

4.0018
3.9222
3.9025

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3.8709

2.5445
2.5291
2.5137

2.3352
2.3298
2.3259

2.3175
2.3117
2.3082

2.2937
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2.2746

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2.1080
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1.9857

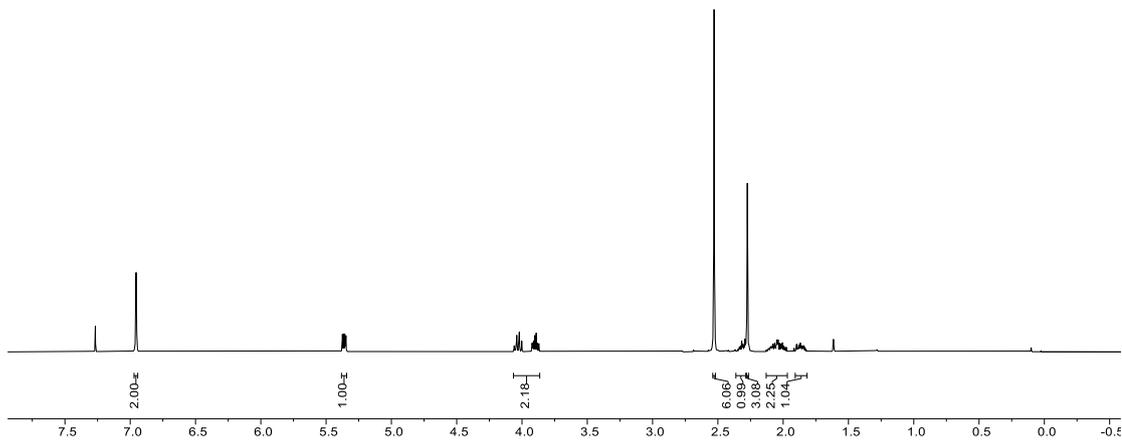
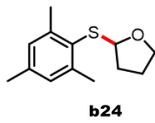
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1.8332
1.8245

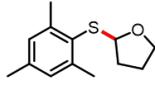


— 143.24
 — 138.37
 < 129.13
 < 128.99

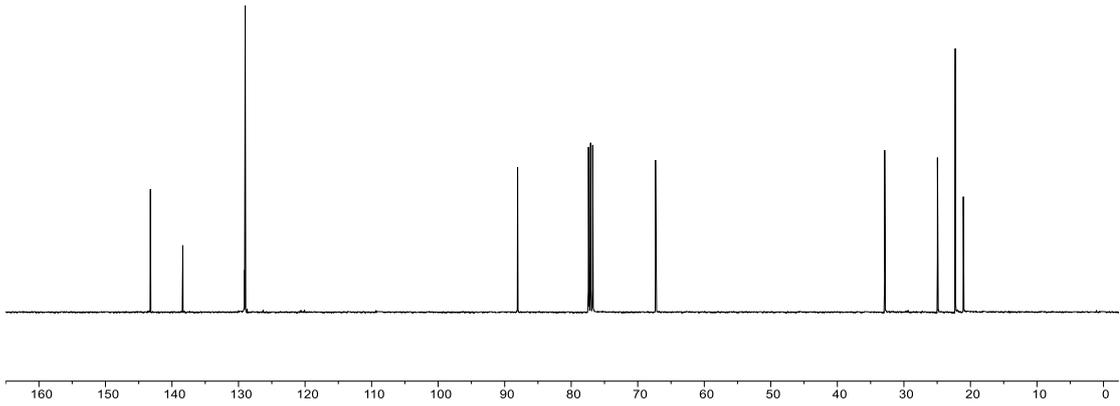
— 86.06
 < 77.40
 < 77.08
 < 76.77

— 87.33

— 32.89
 — 24.85
 < 22.28
 < 21.07



b24

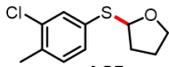


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 7.3183
 7.3136
 7.2988
 7.1877 CDCl₃
 7.1498

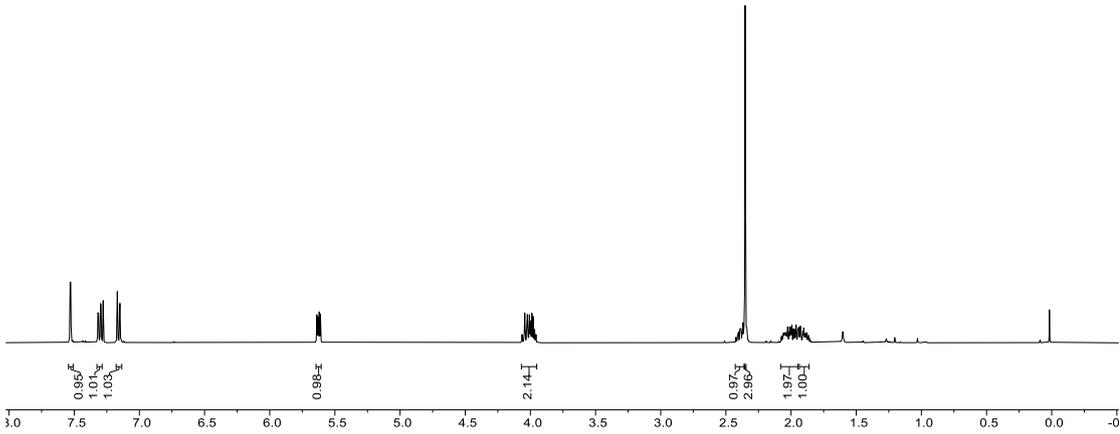
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 5.6203
 5.6110

4.0637
 4.0431
 4.0228
 4.0082
 3.9935
 3.9785

2.4250
 2.4152
 2.4072
 2.3934
 2.3886
 2.3844
 2.3755
 2.3702
 2.3622 H₂O
 2.1675
 2.0588
 2.0468
 2.0314
 2.0261
 2.0161
 2.0111
 2.0053
 1.9926
 1.9821
 1.9750
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 1.9494
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 1.8579



b25



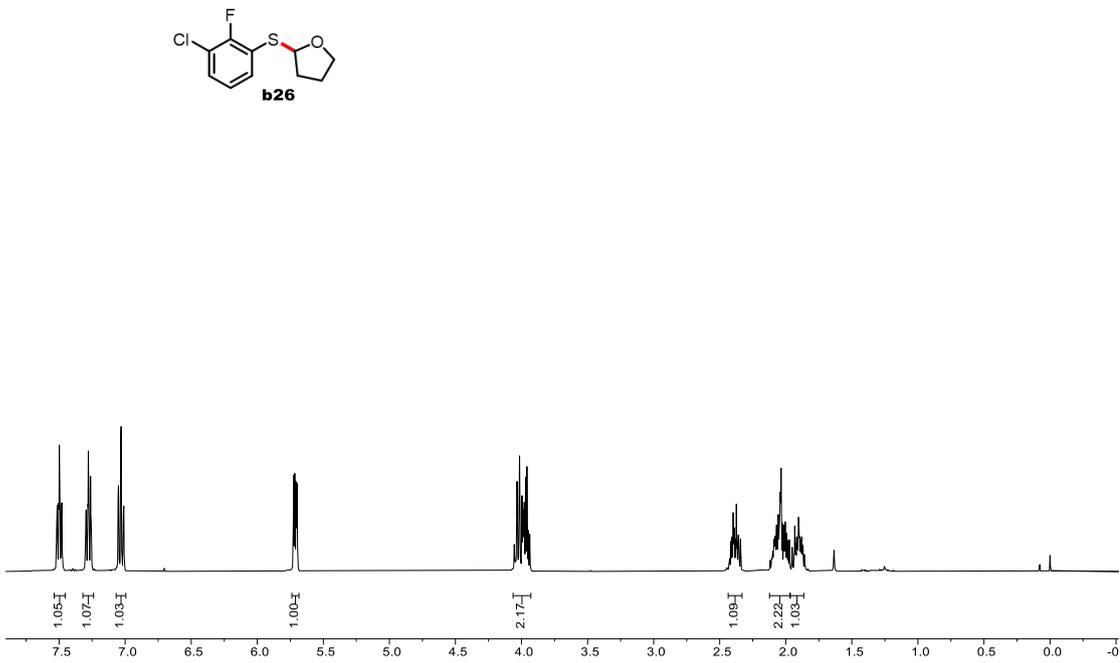
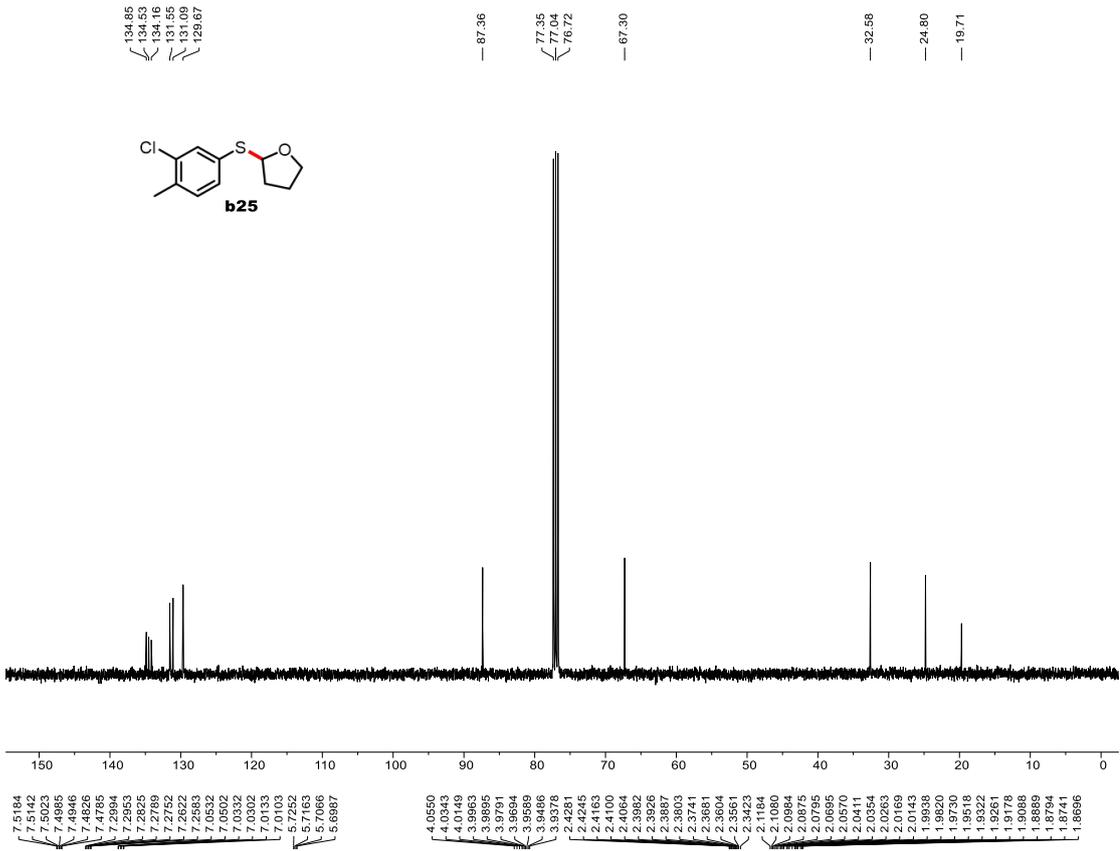
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0.98

2.14

0.97
 2.96

1.87
 1.00



157.97
155.52

131.64
129.38
124.77
124.70
124.67
124.62
124.57
121.46
121.27

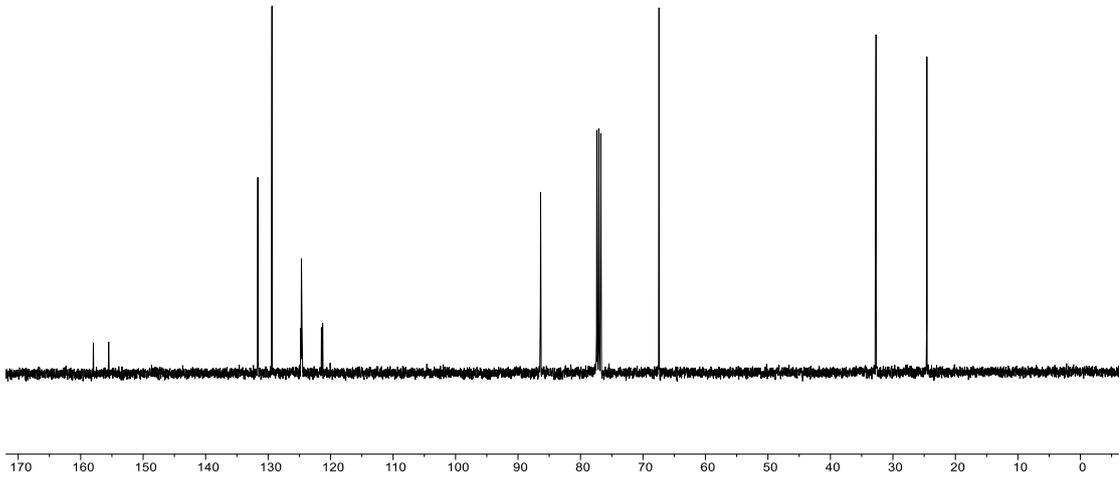
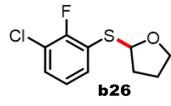
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77.39
77.08
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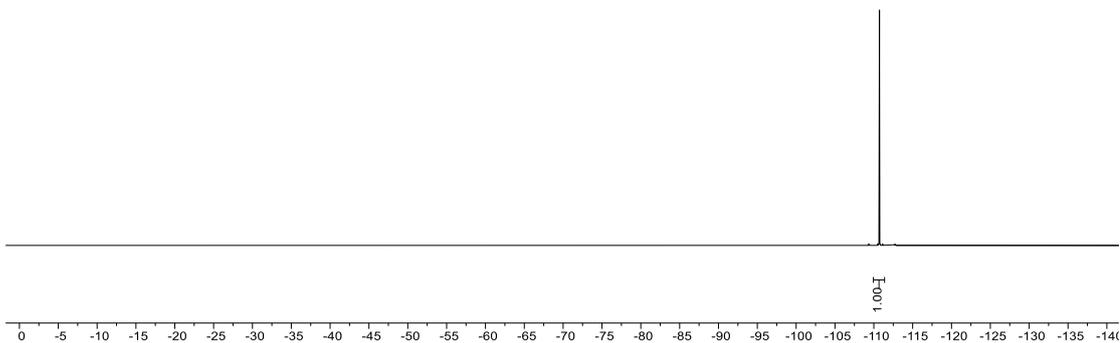
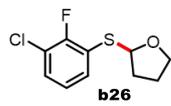
67.44

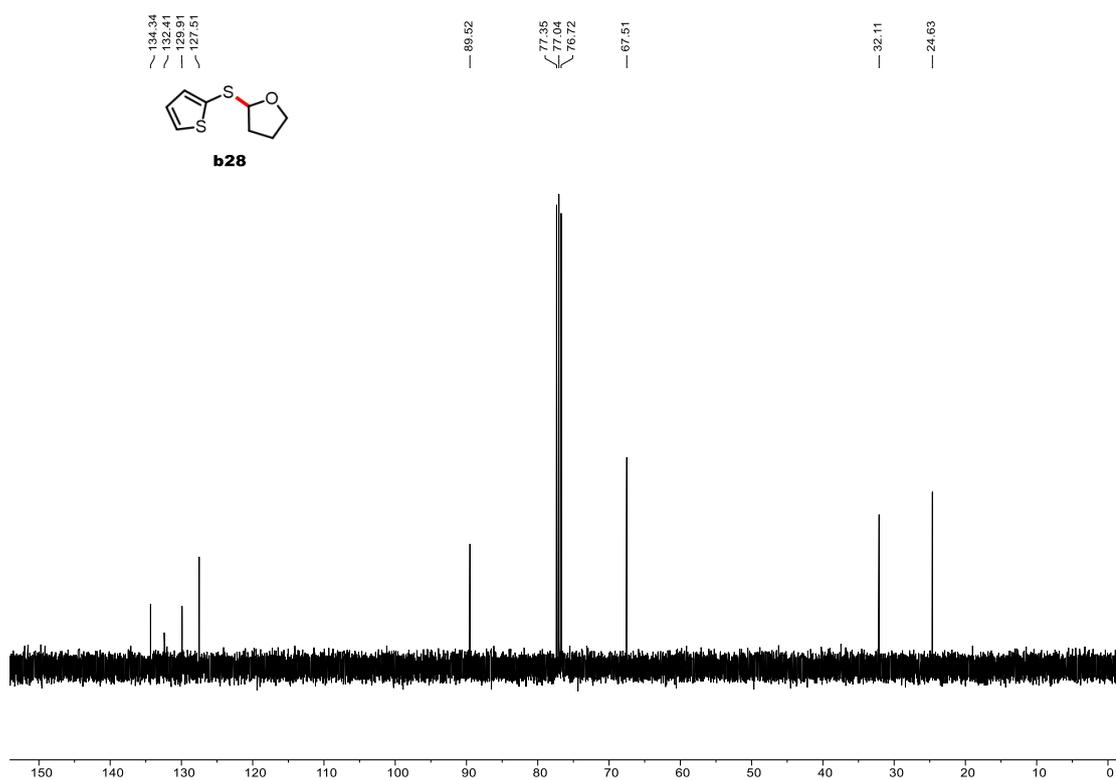
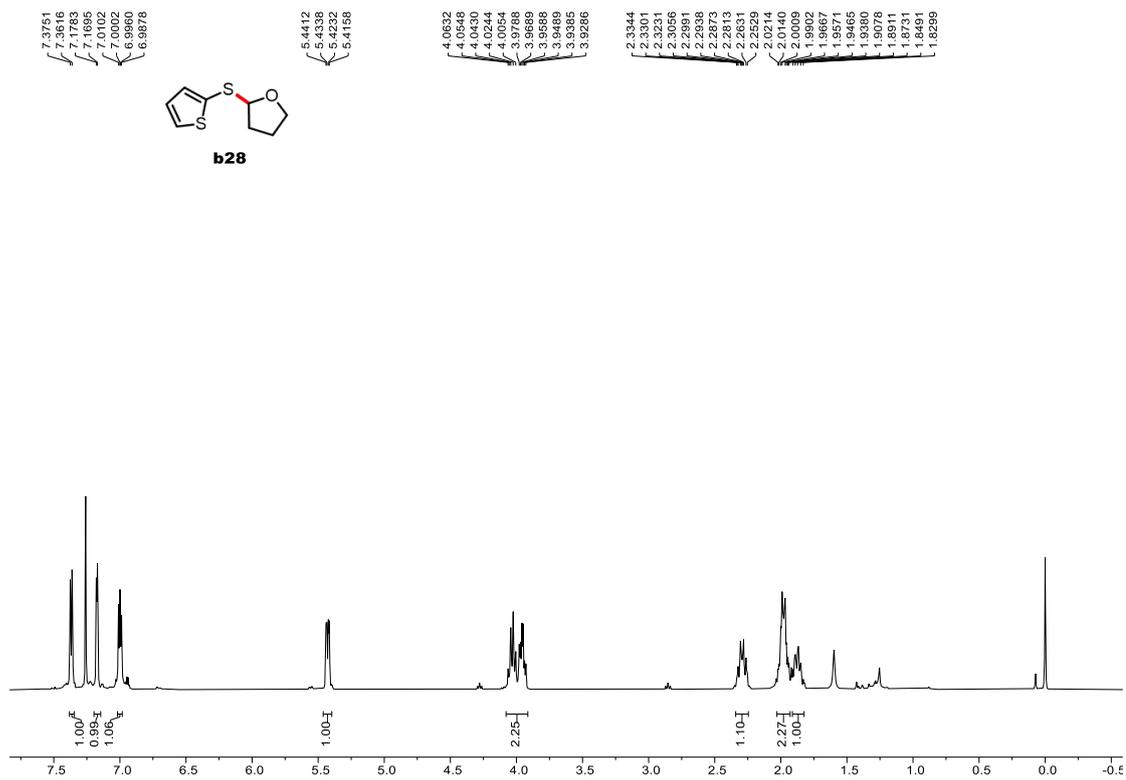
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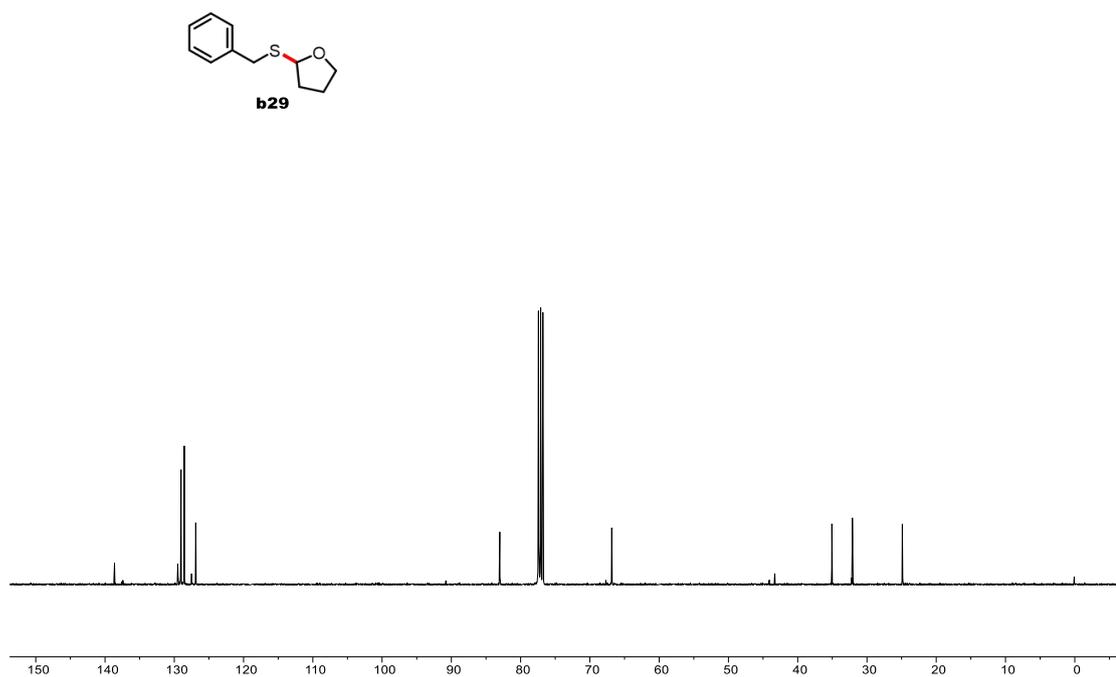
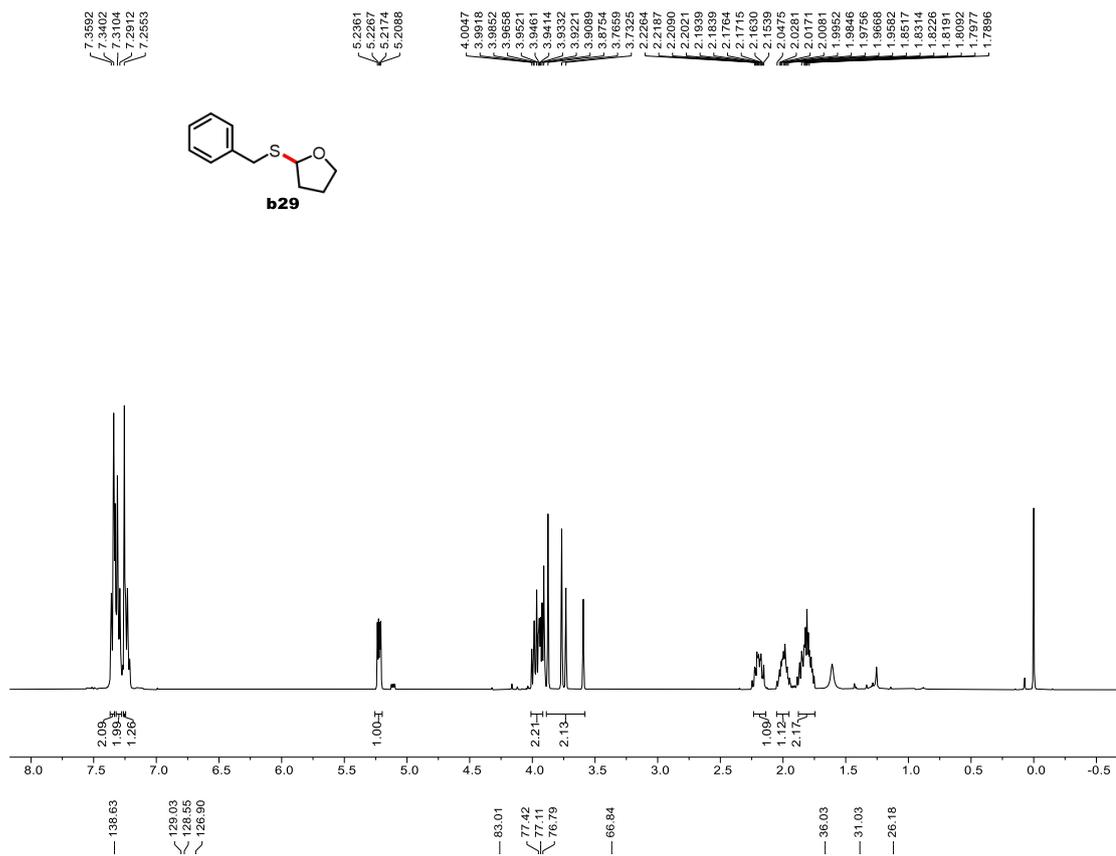
24.57



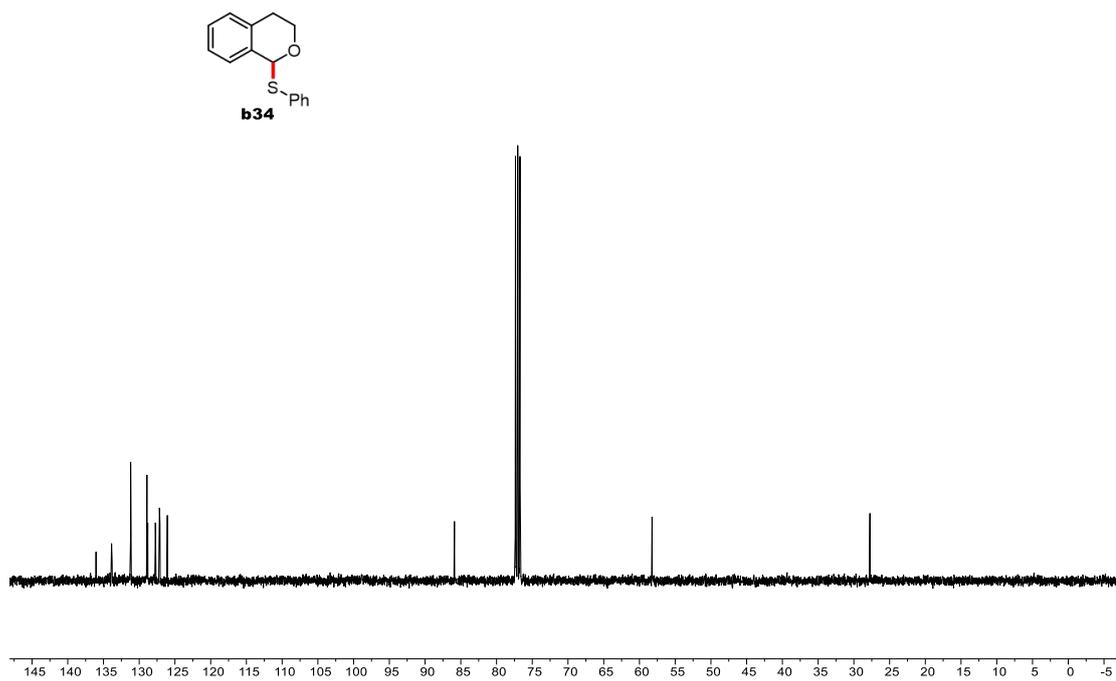
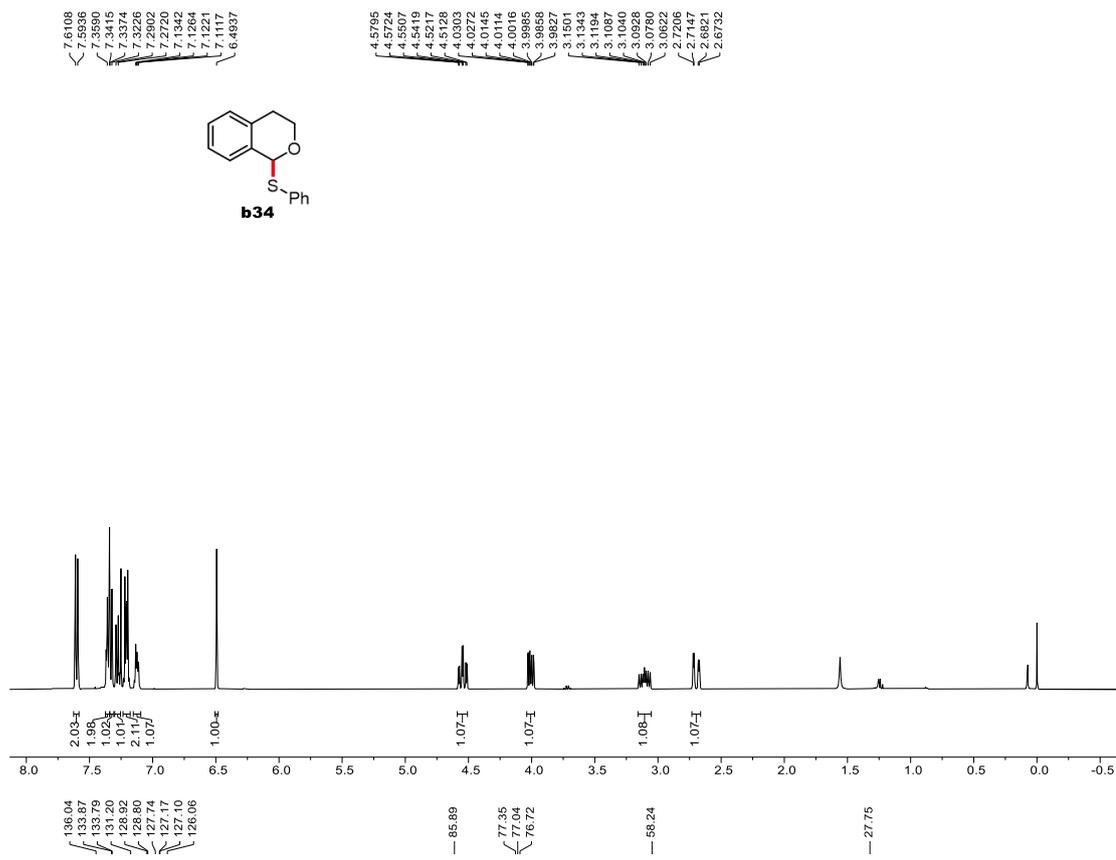
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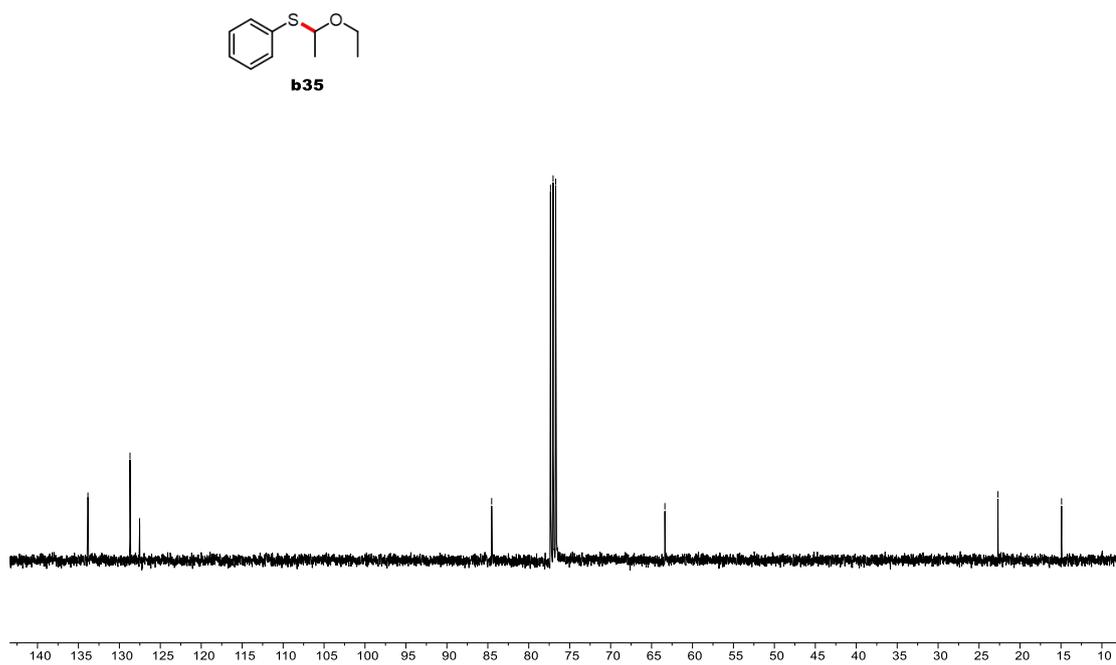
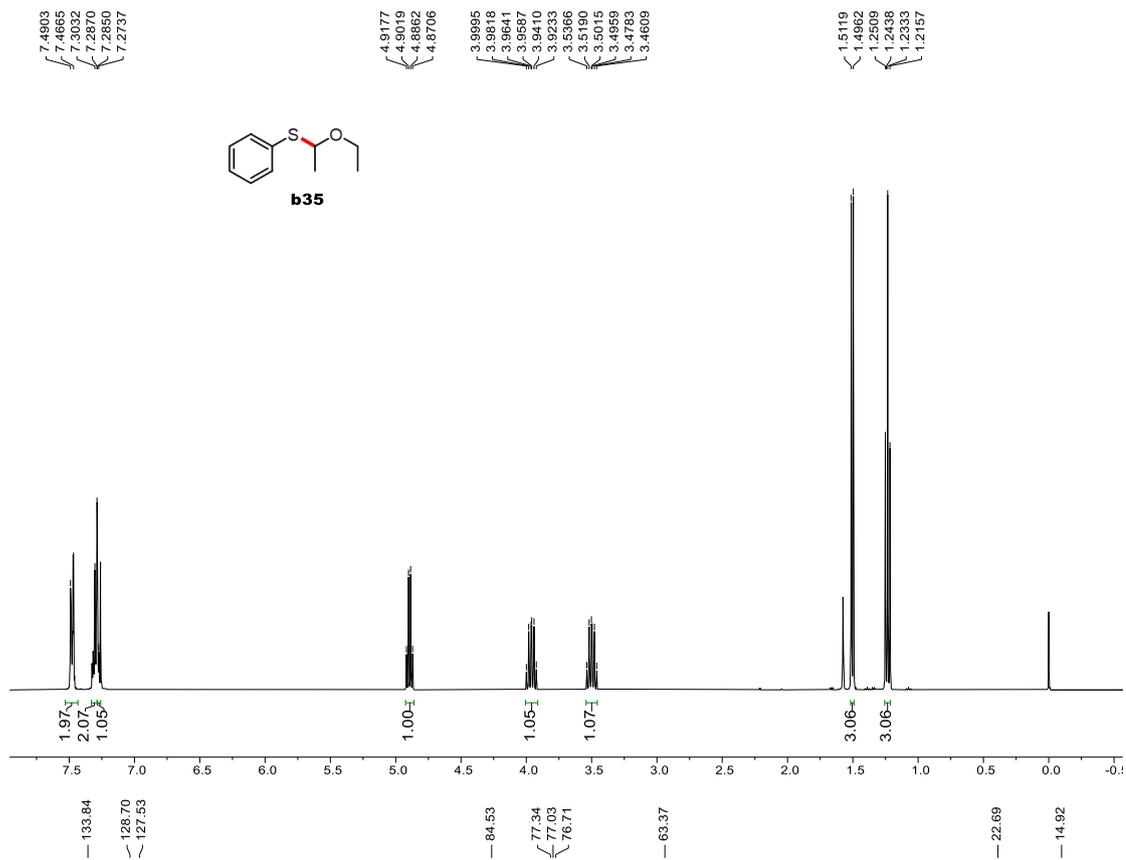






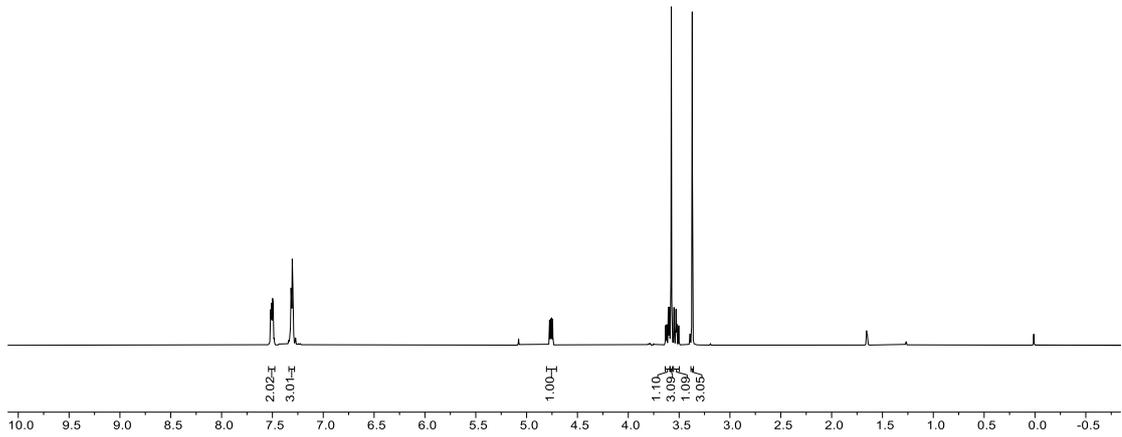
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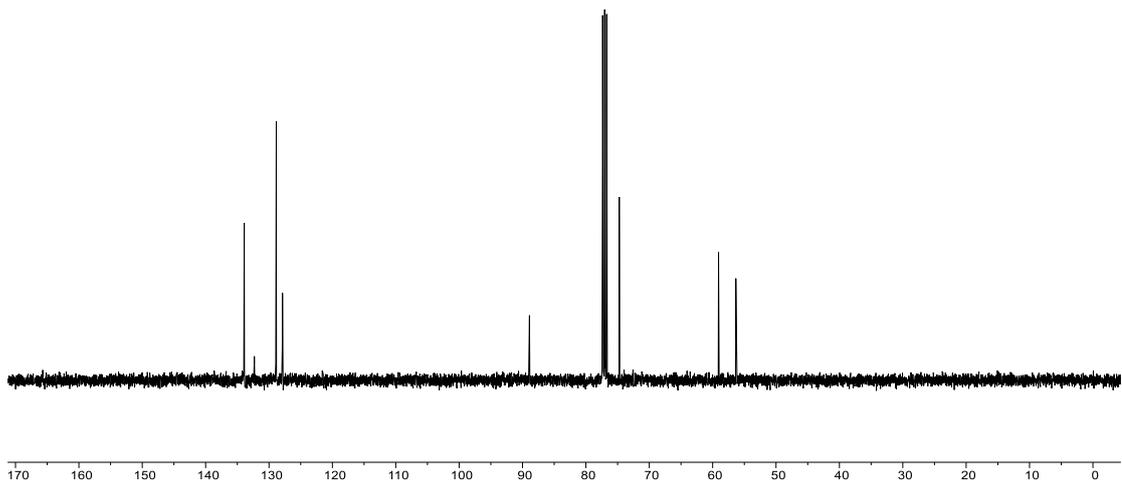


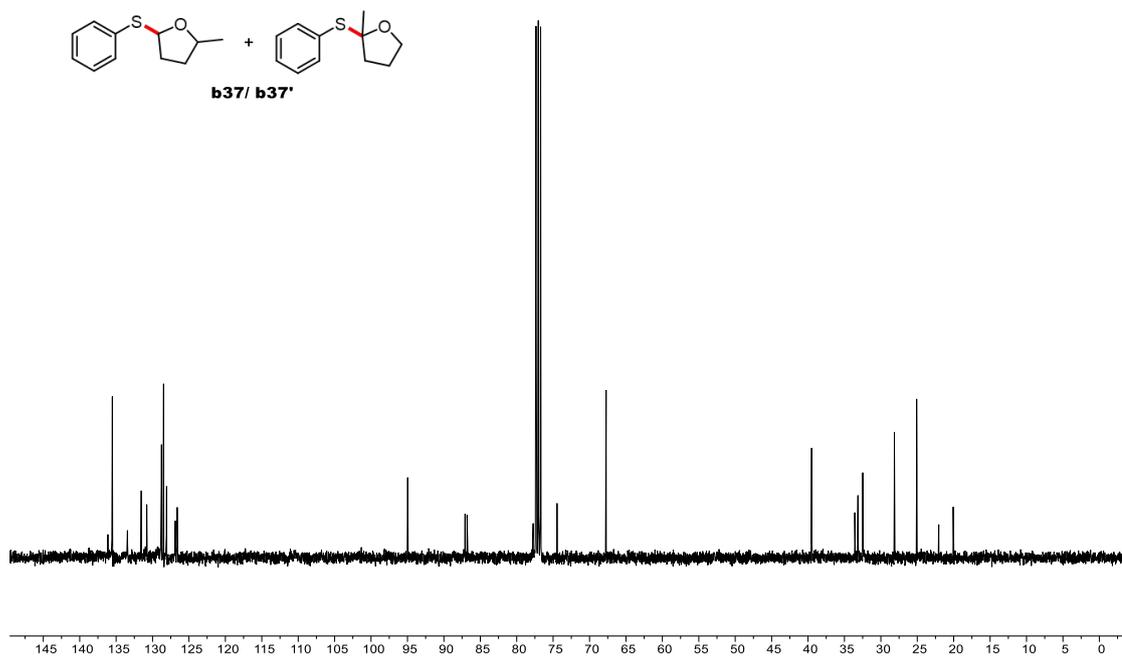
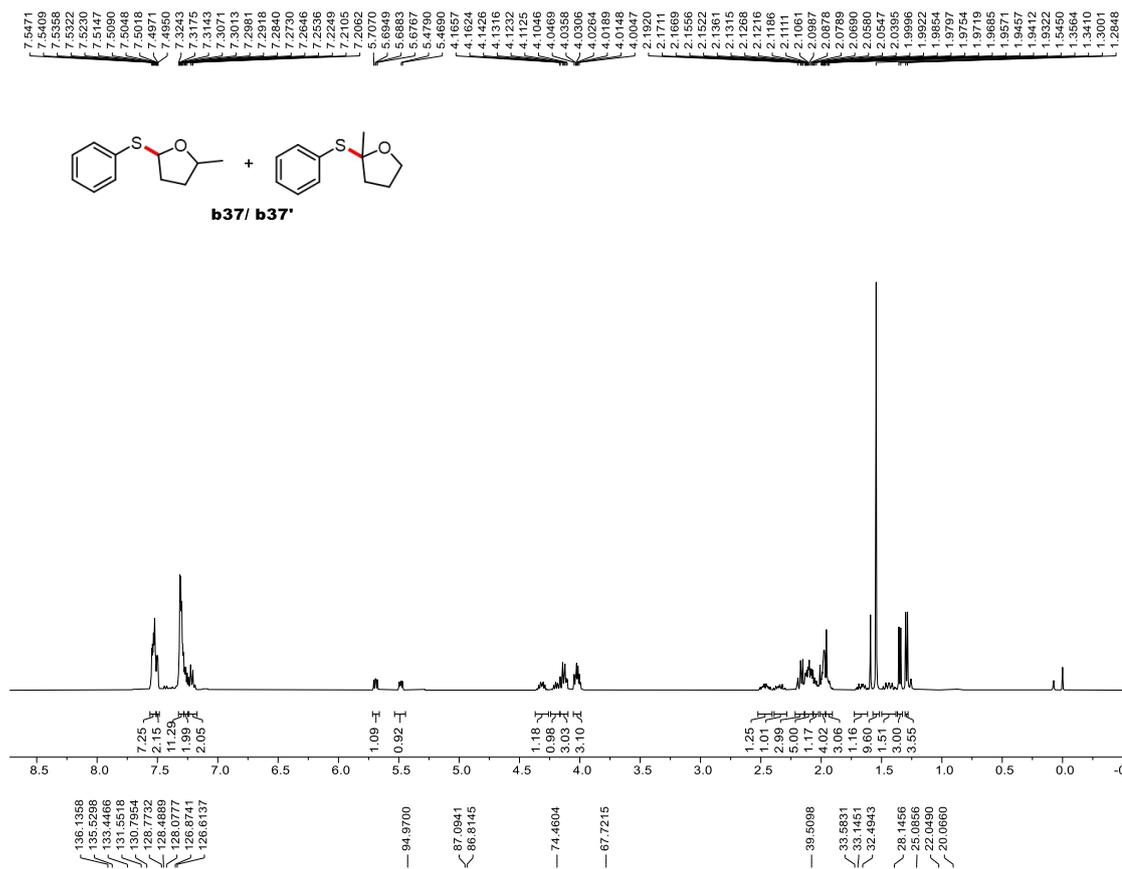
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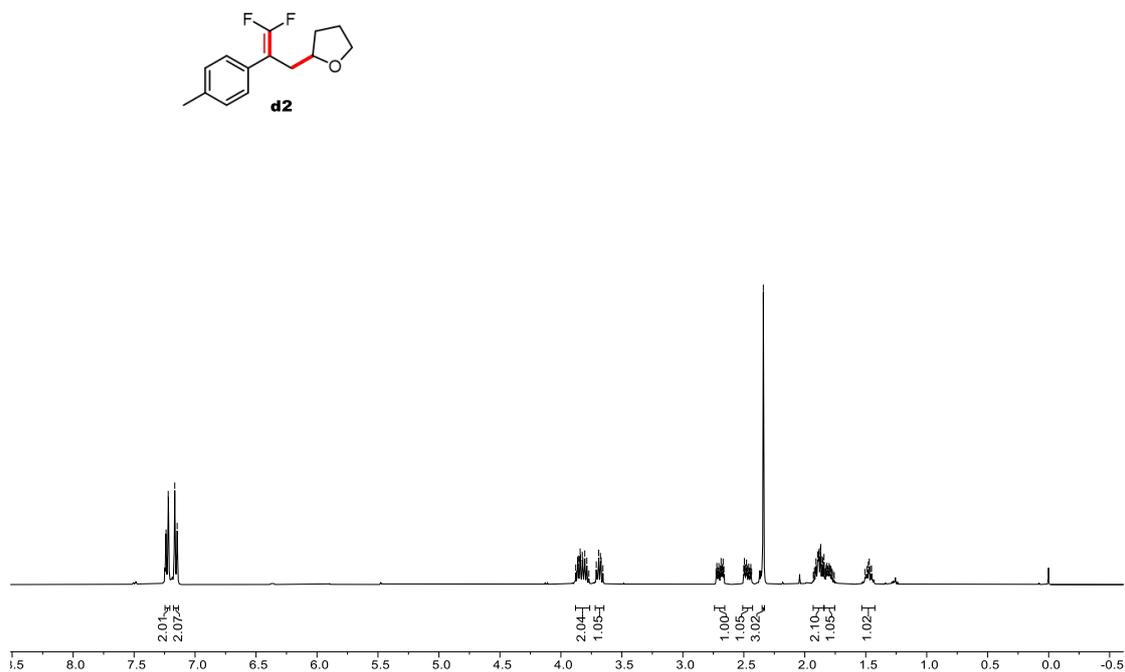
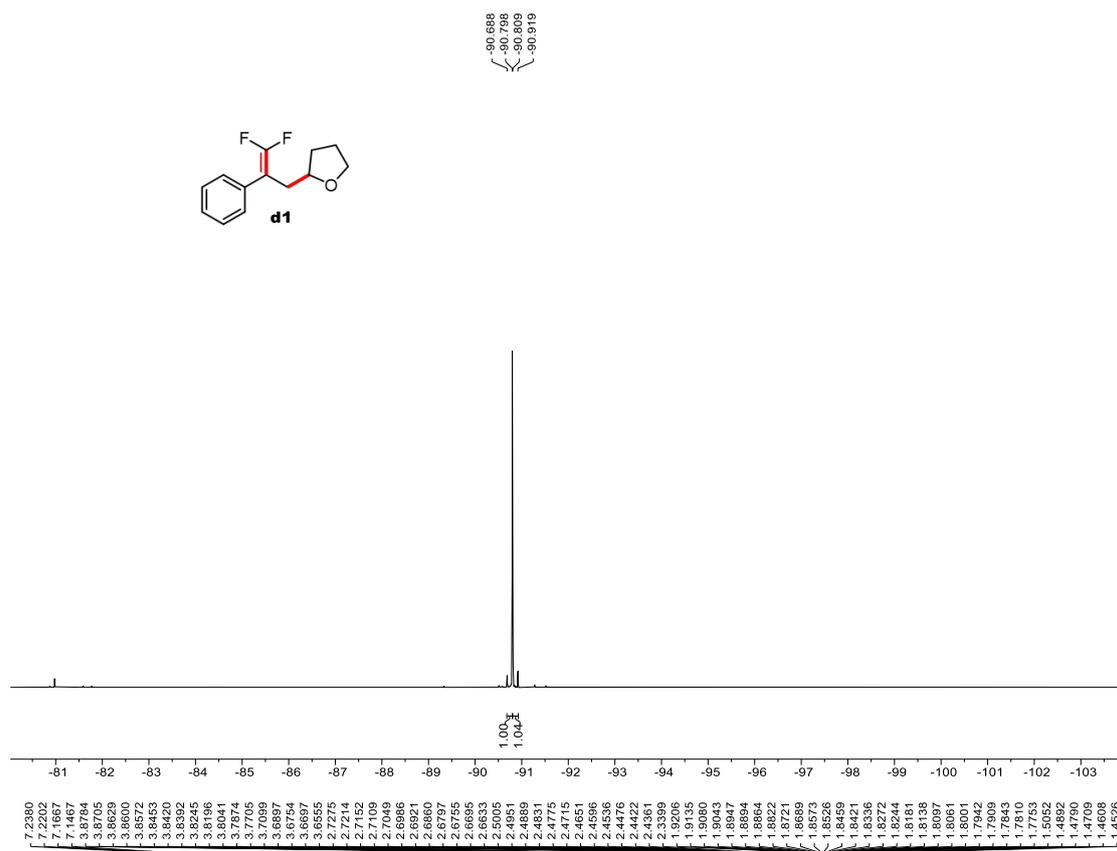
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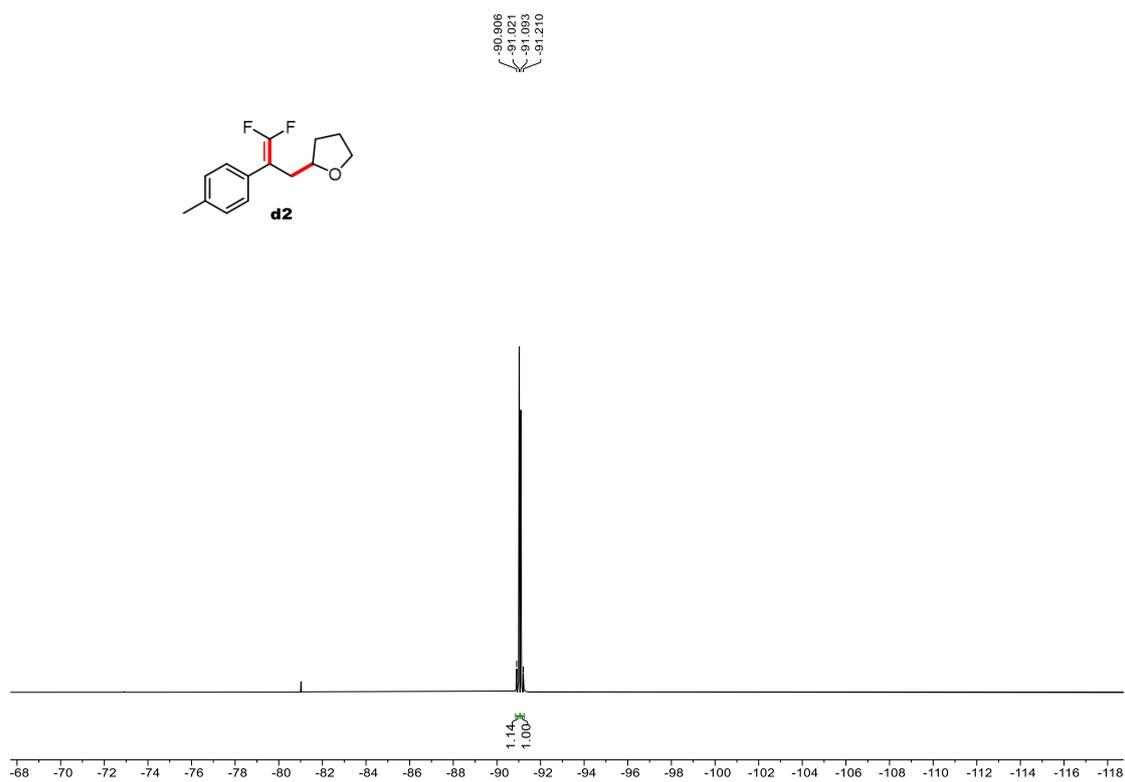
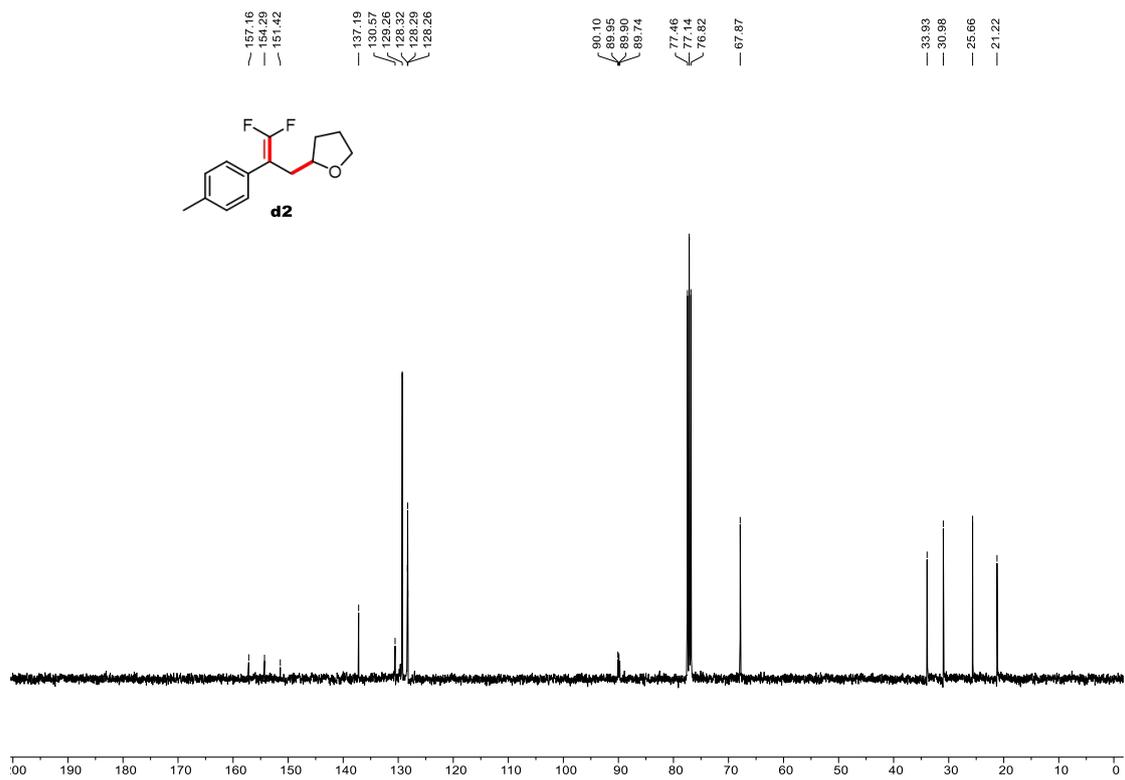
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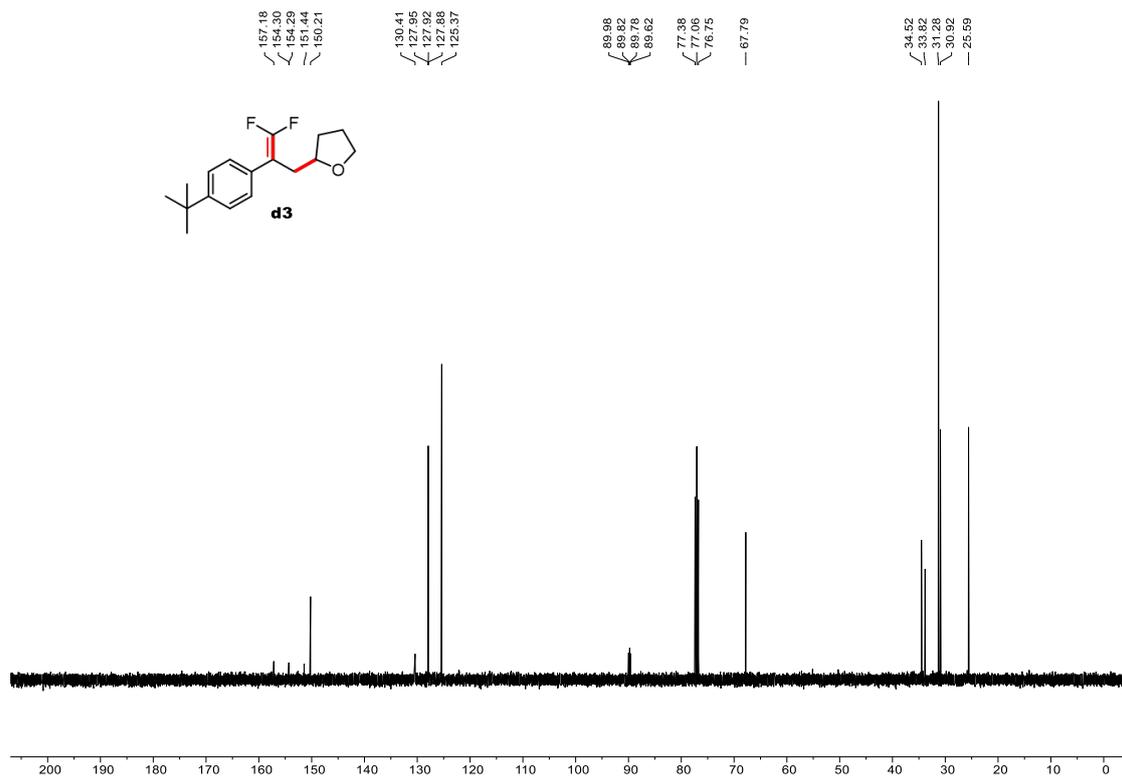
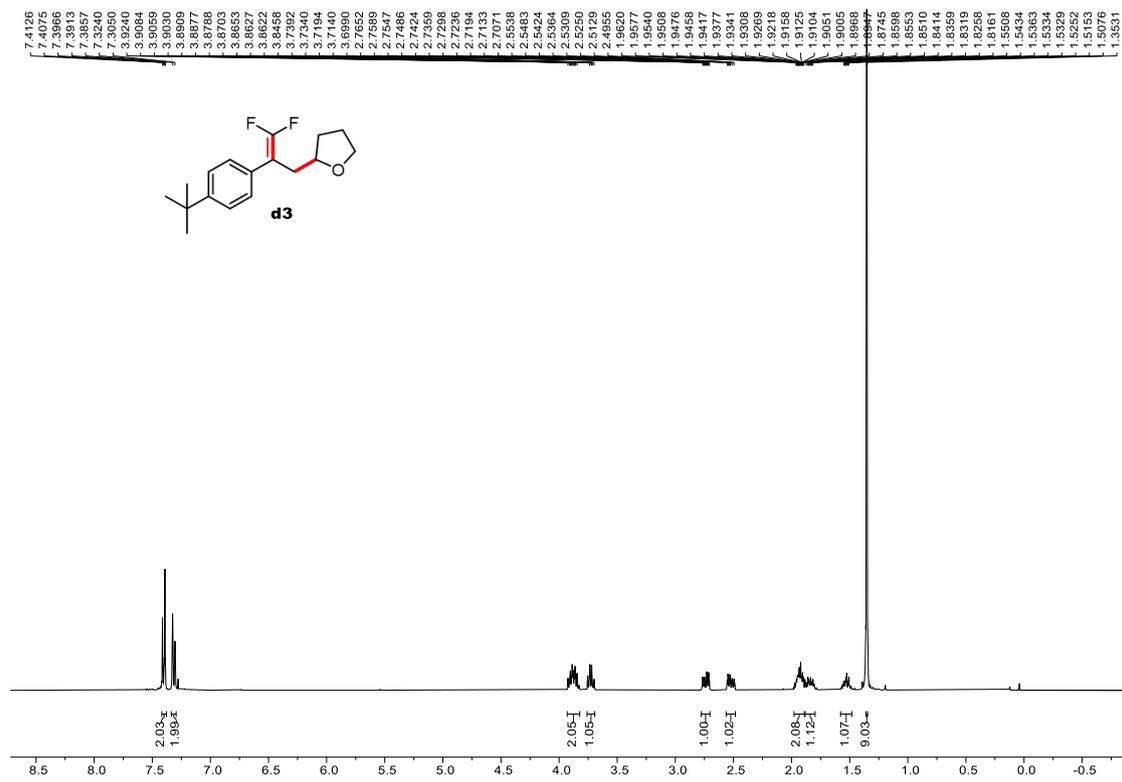


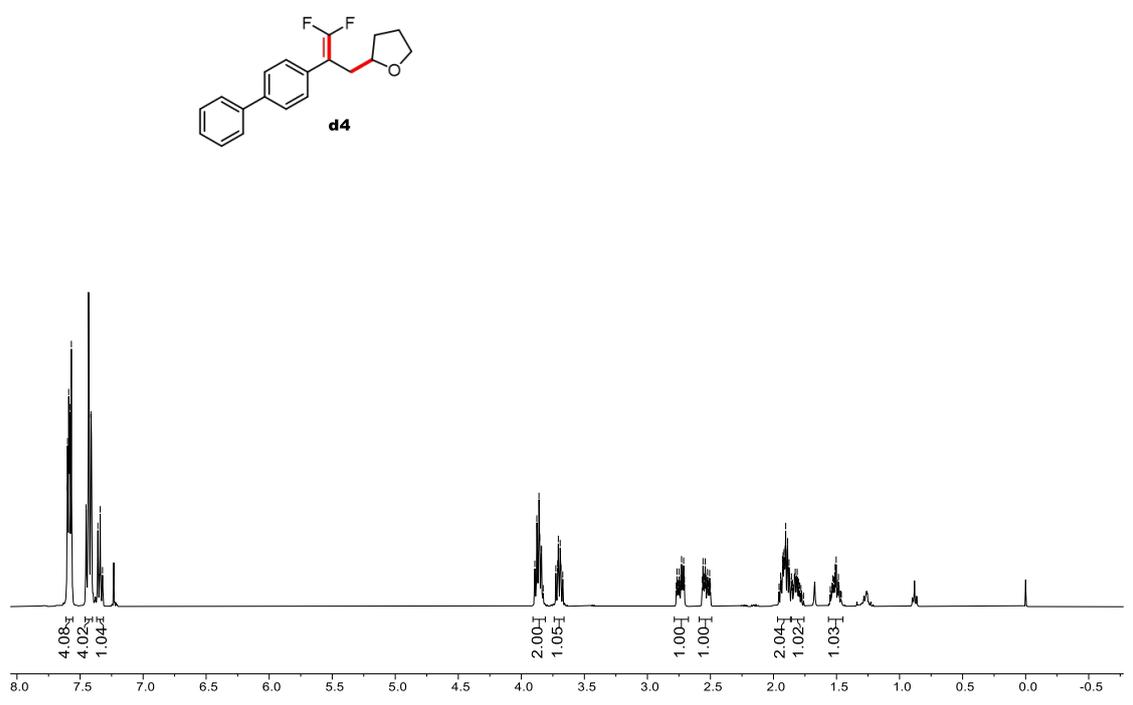
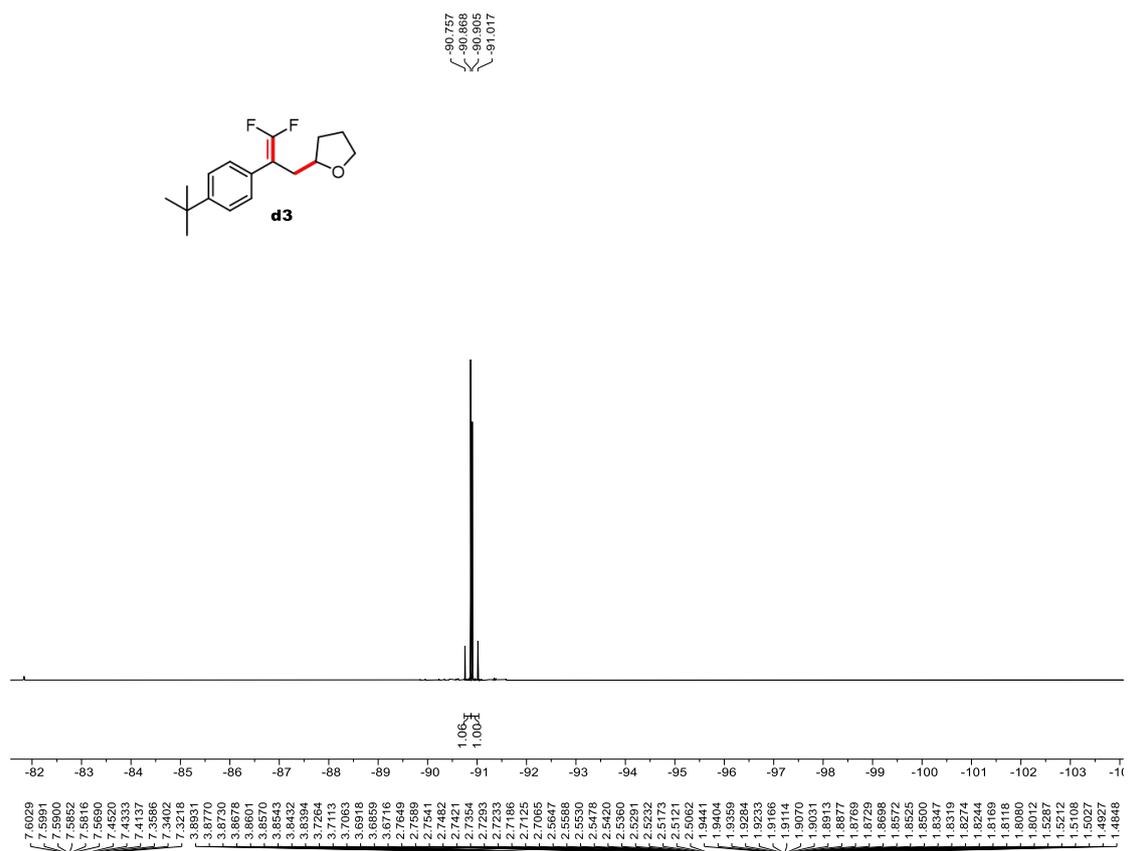


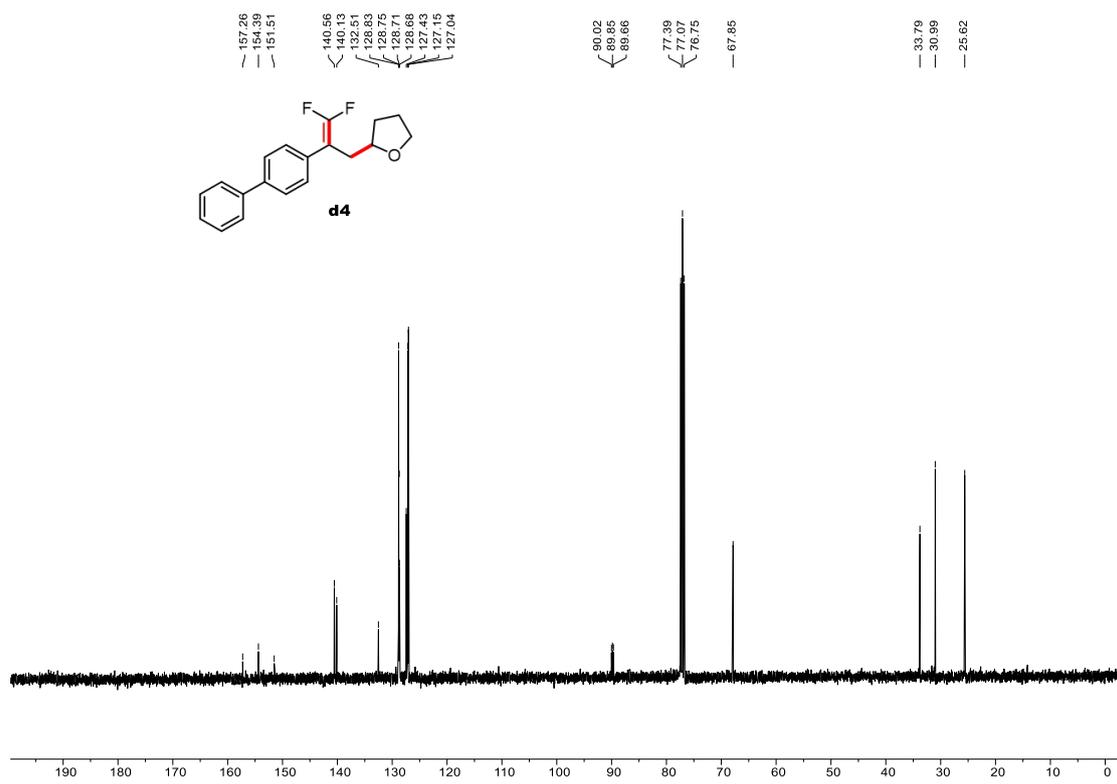
Due to the minimal polarity difference of **b37** and **b37'**, the isolation is difficult. Therefore, the mixture of **b37** and **b37'** was obtained.

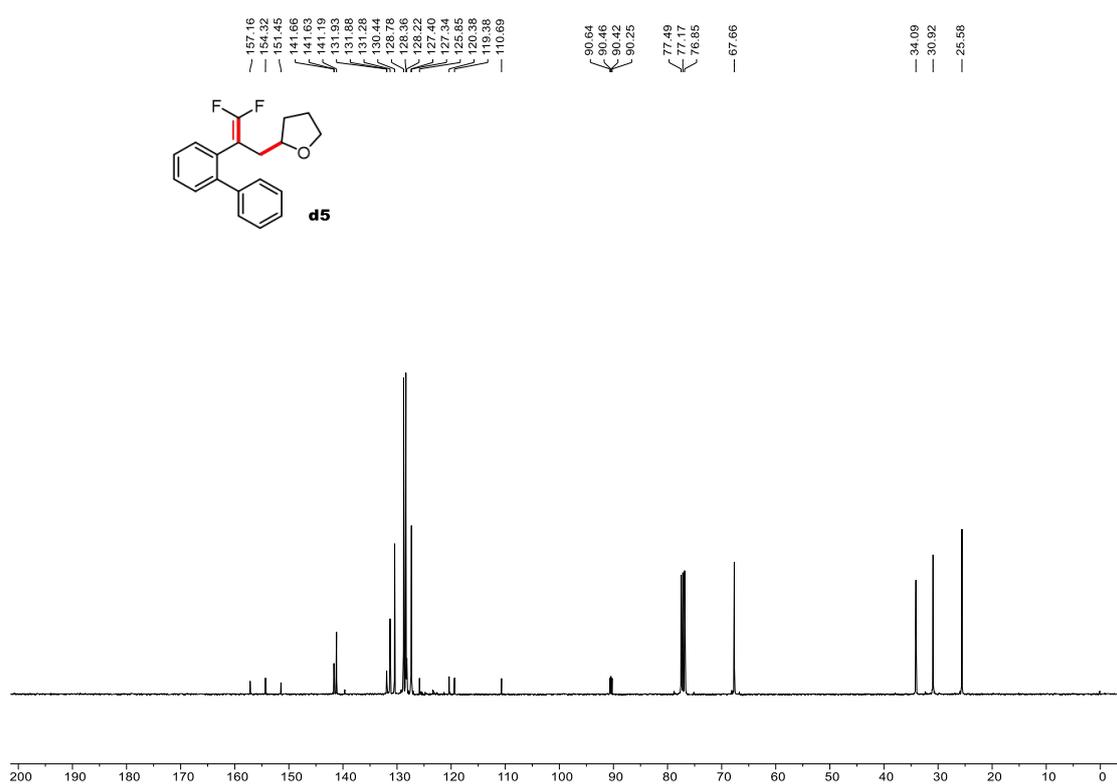
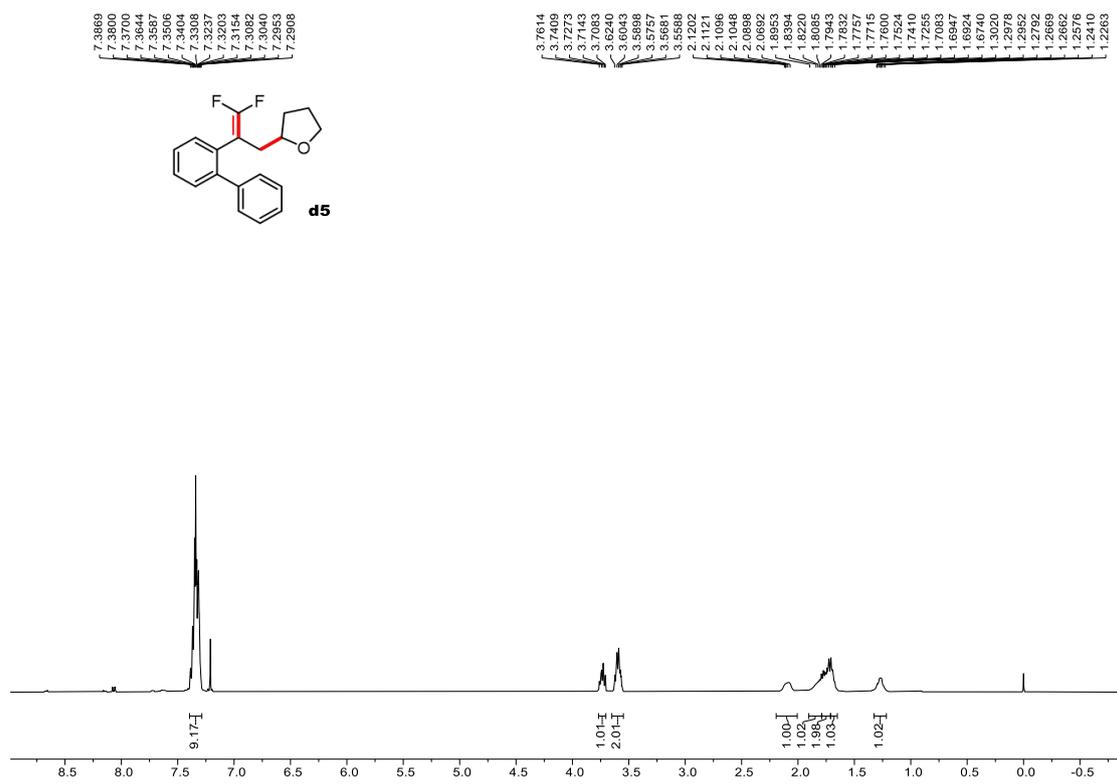


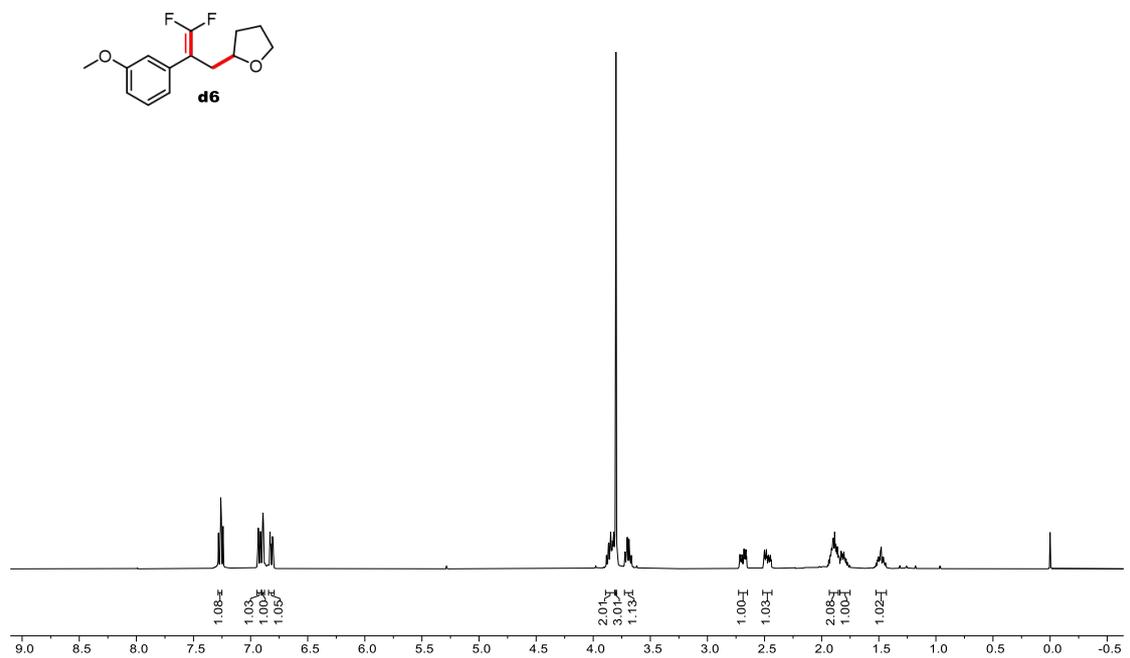
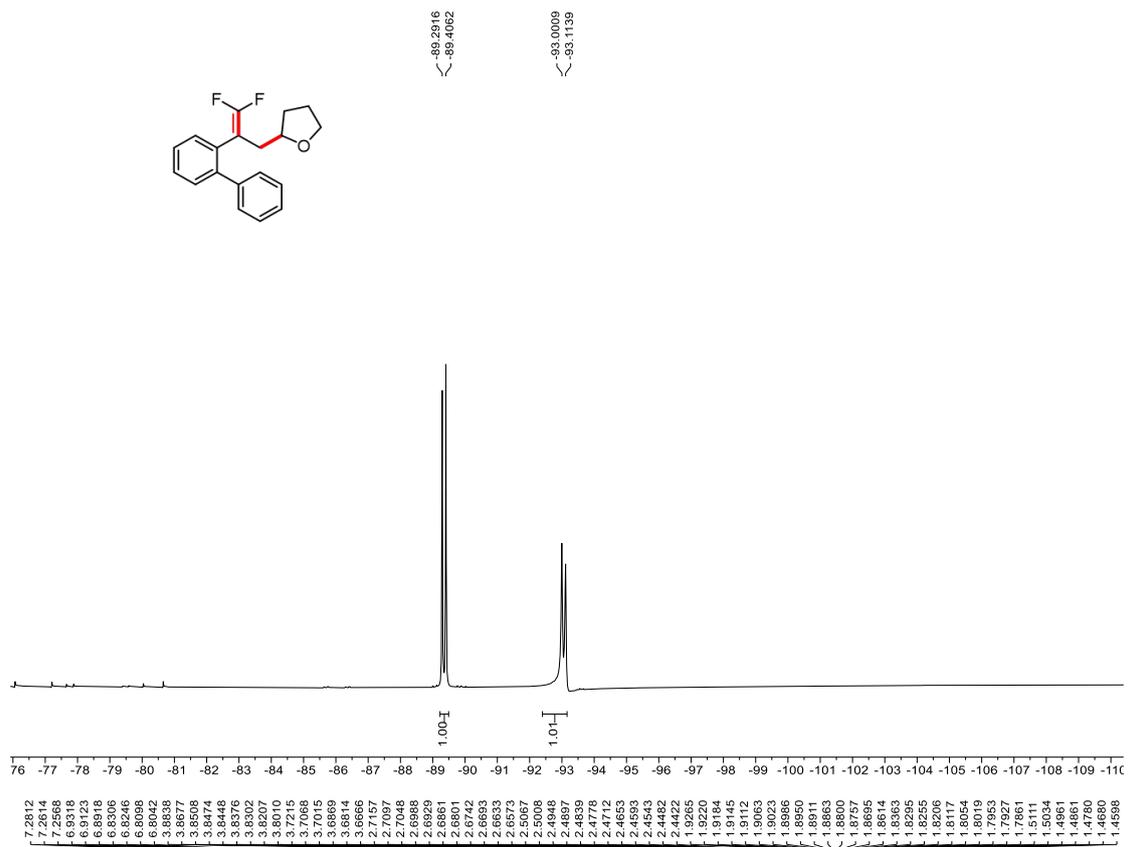


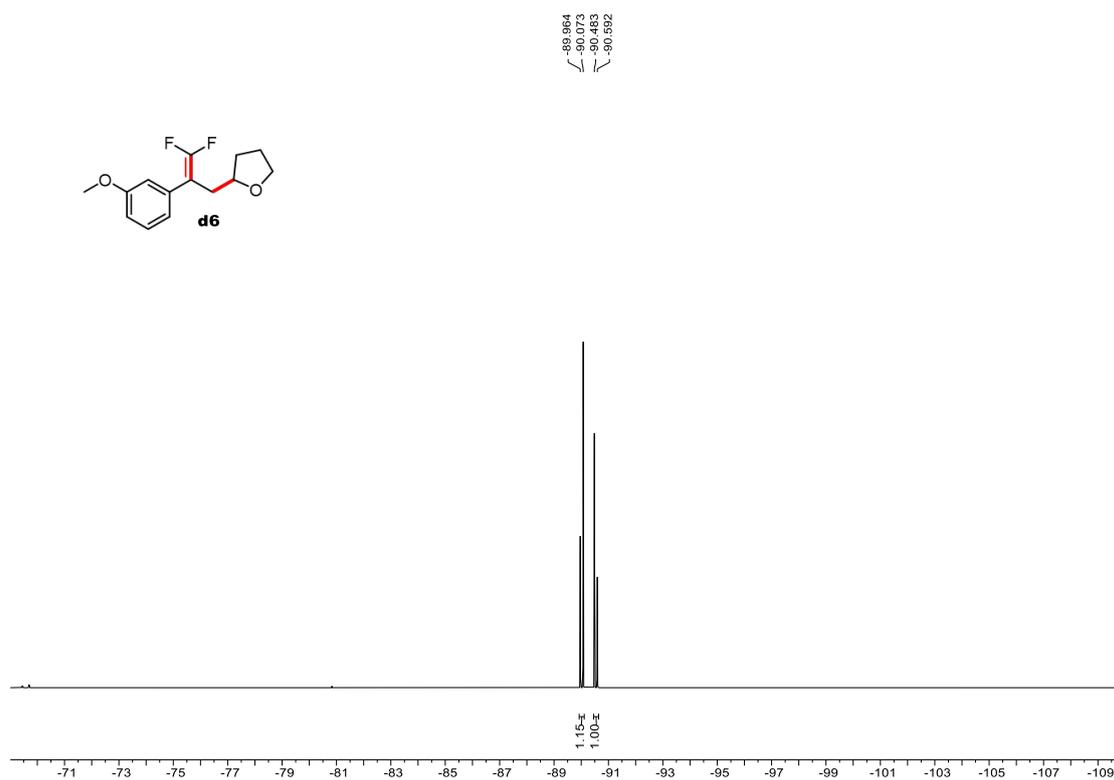
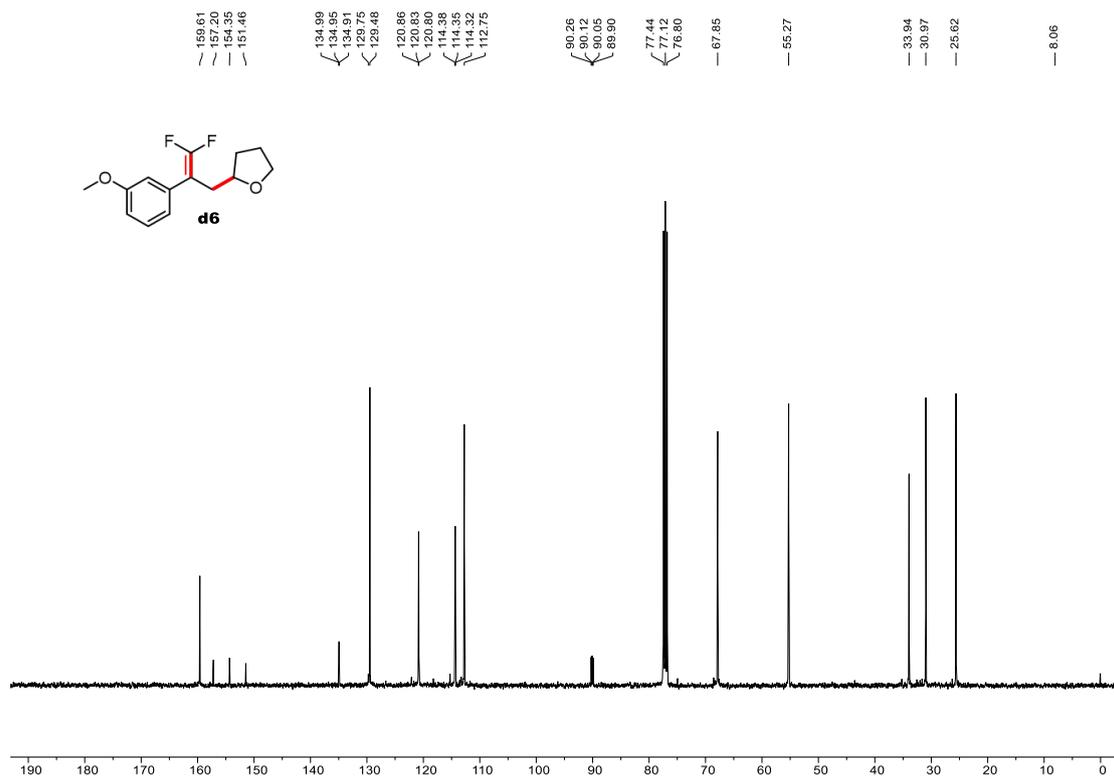


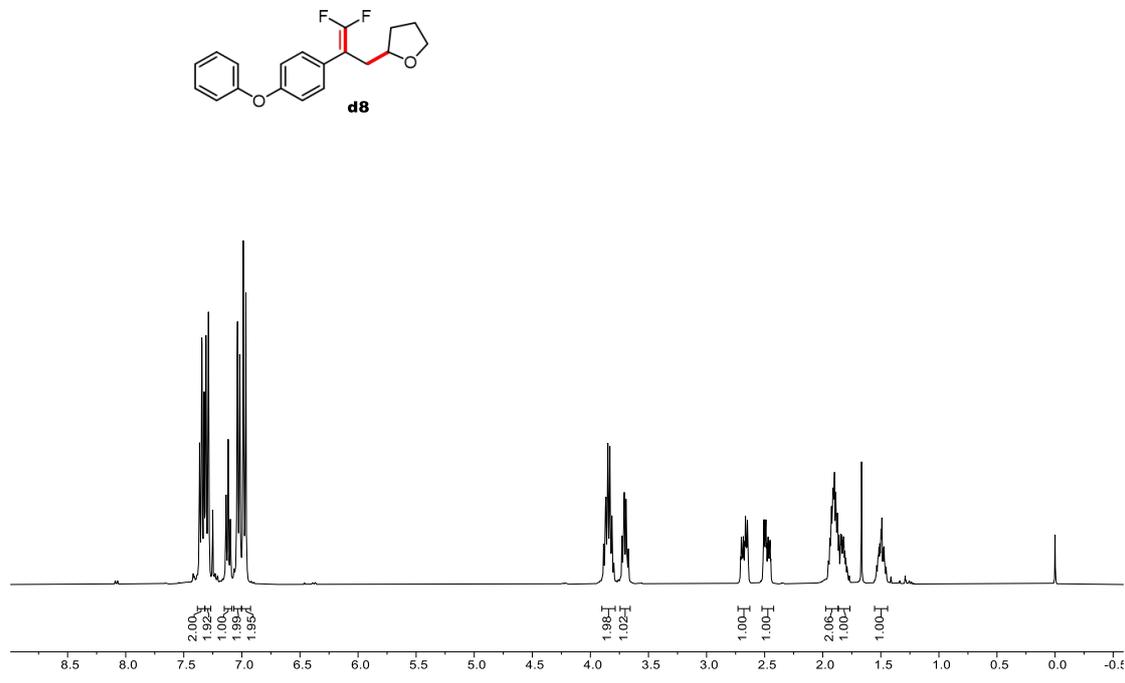
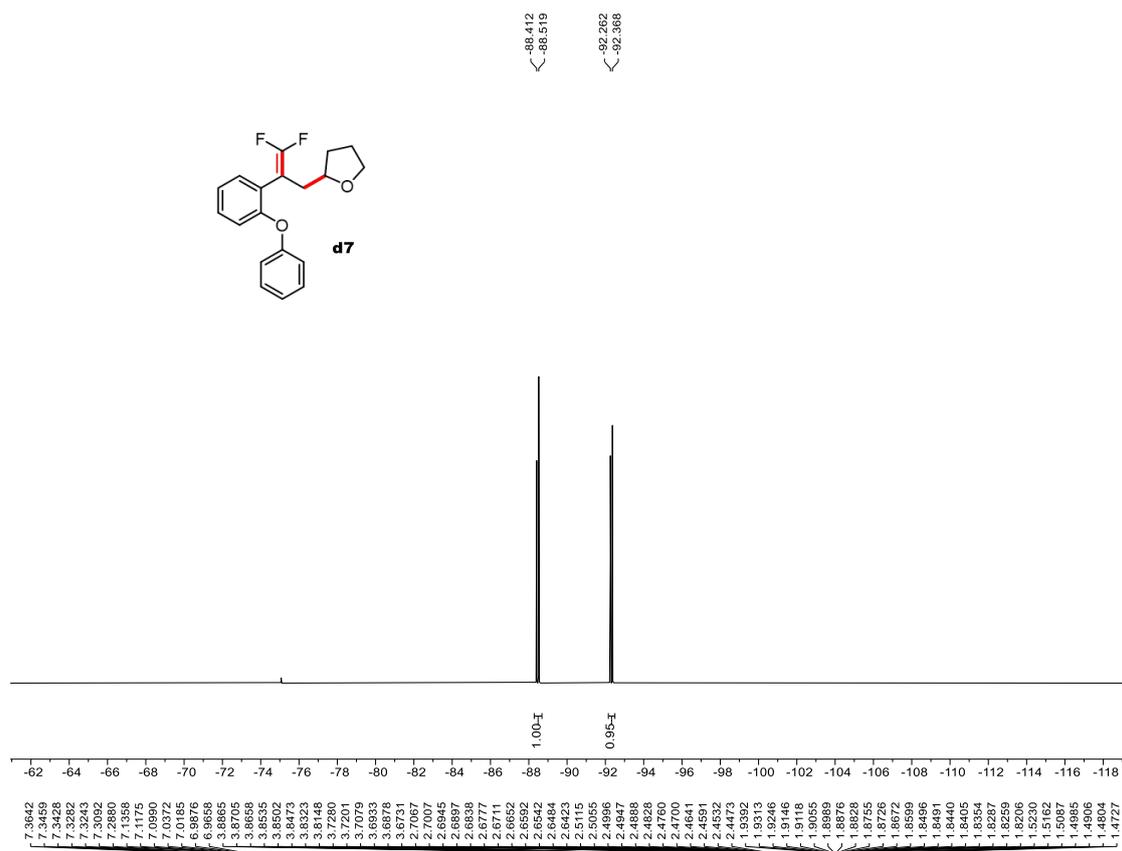


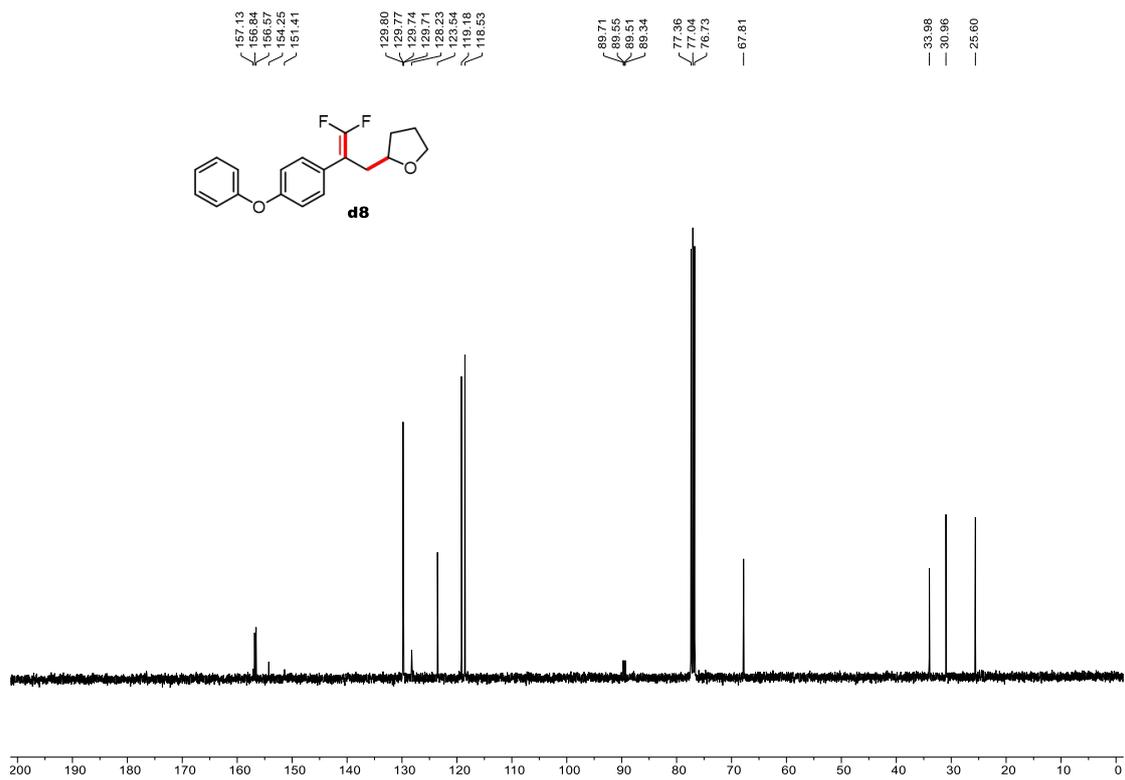


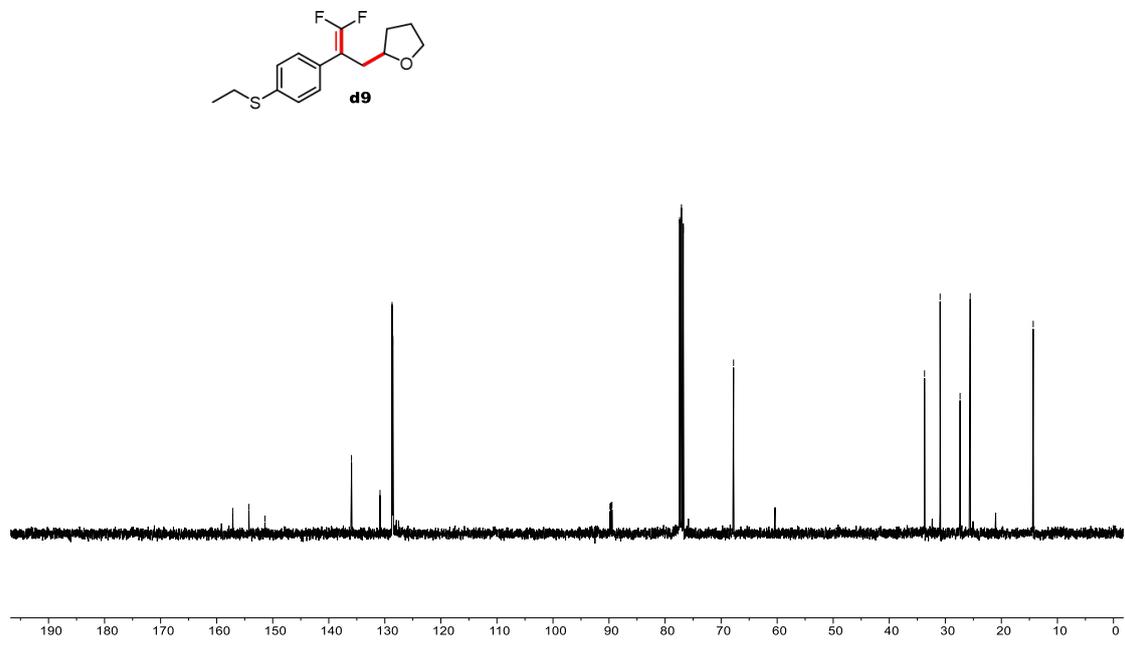
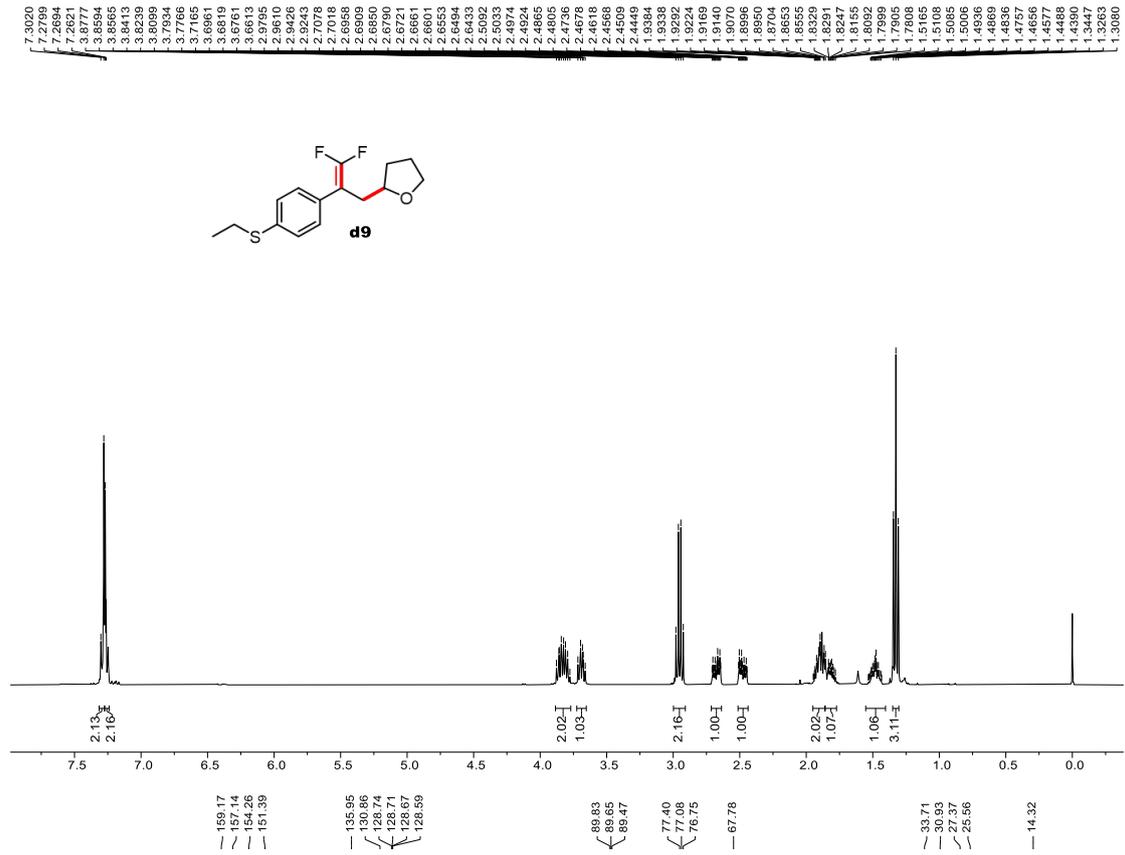


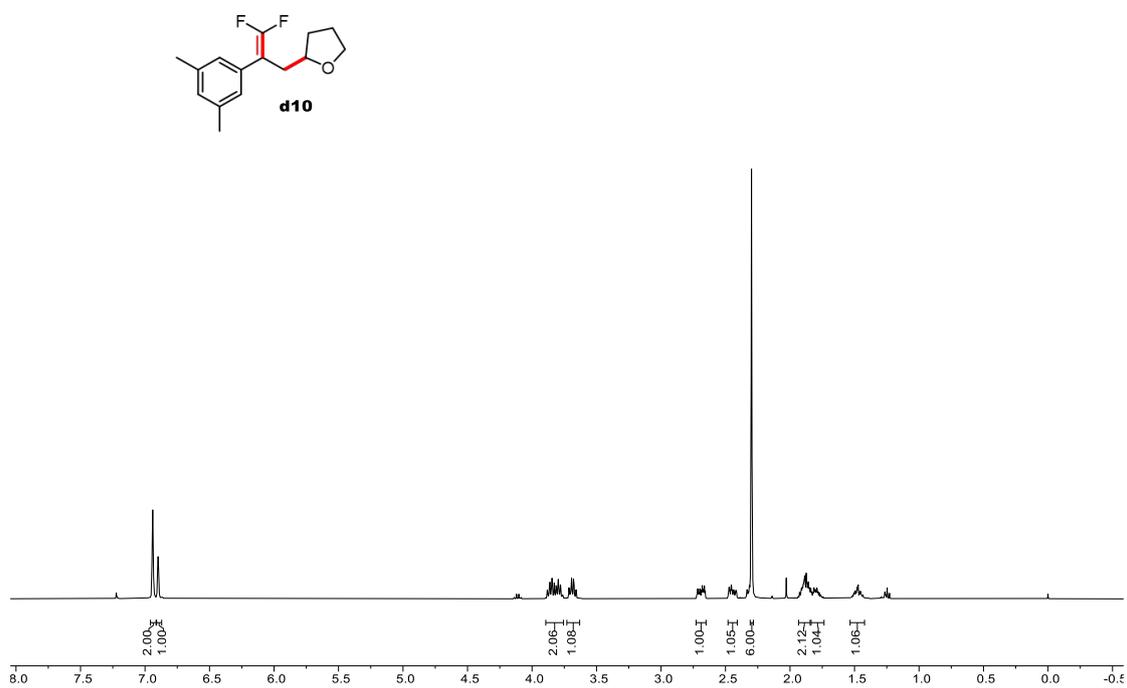
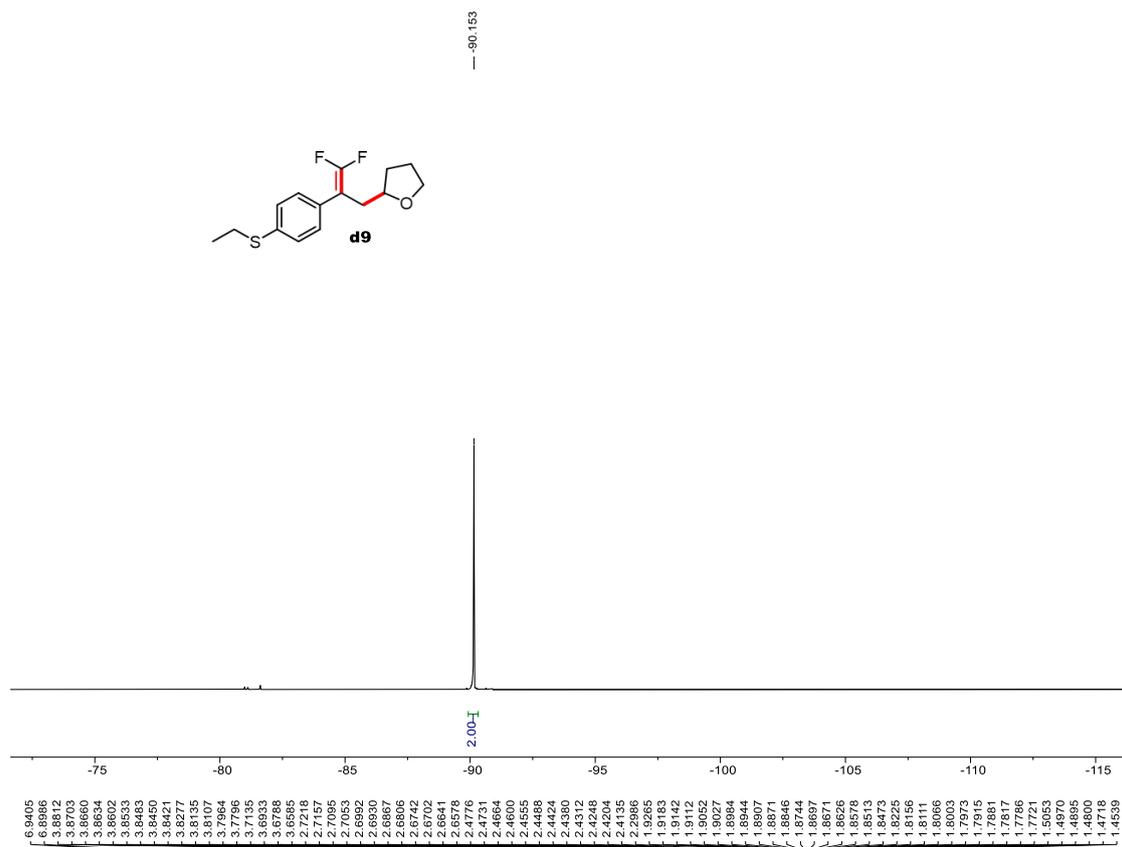


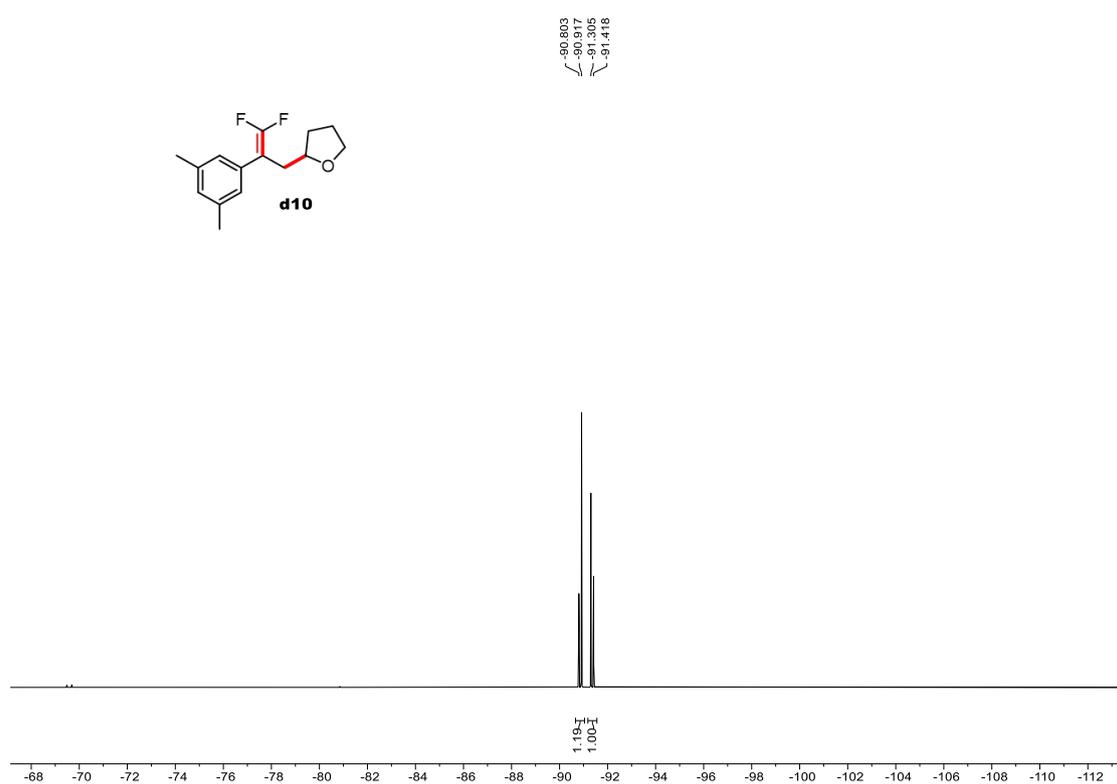
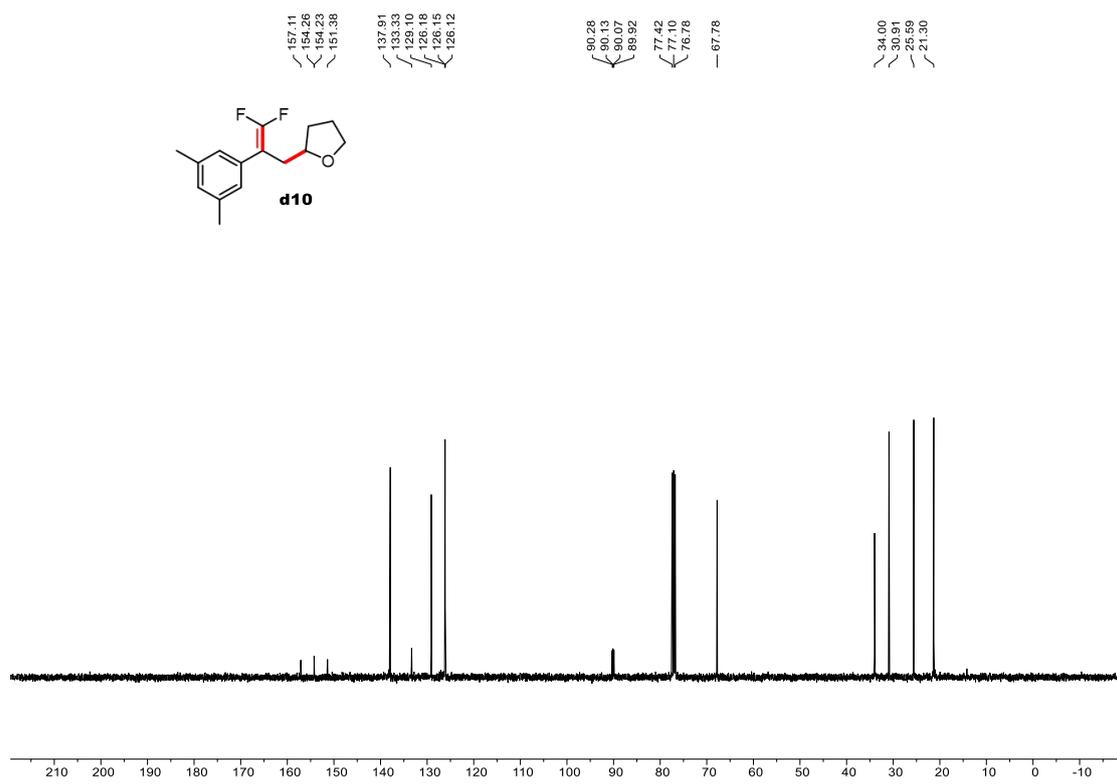


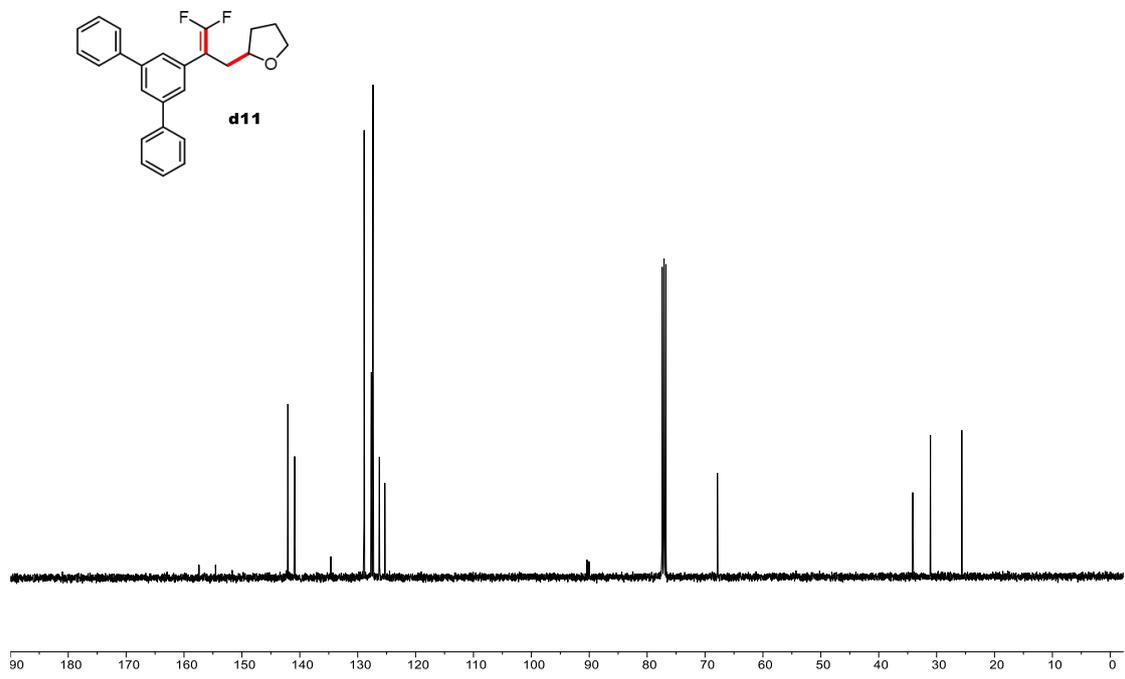
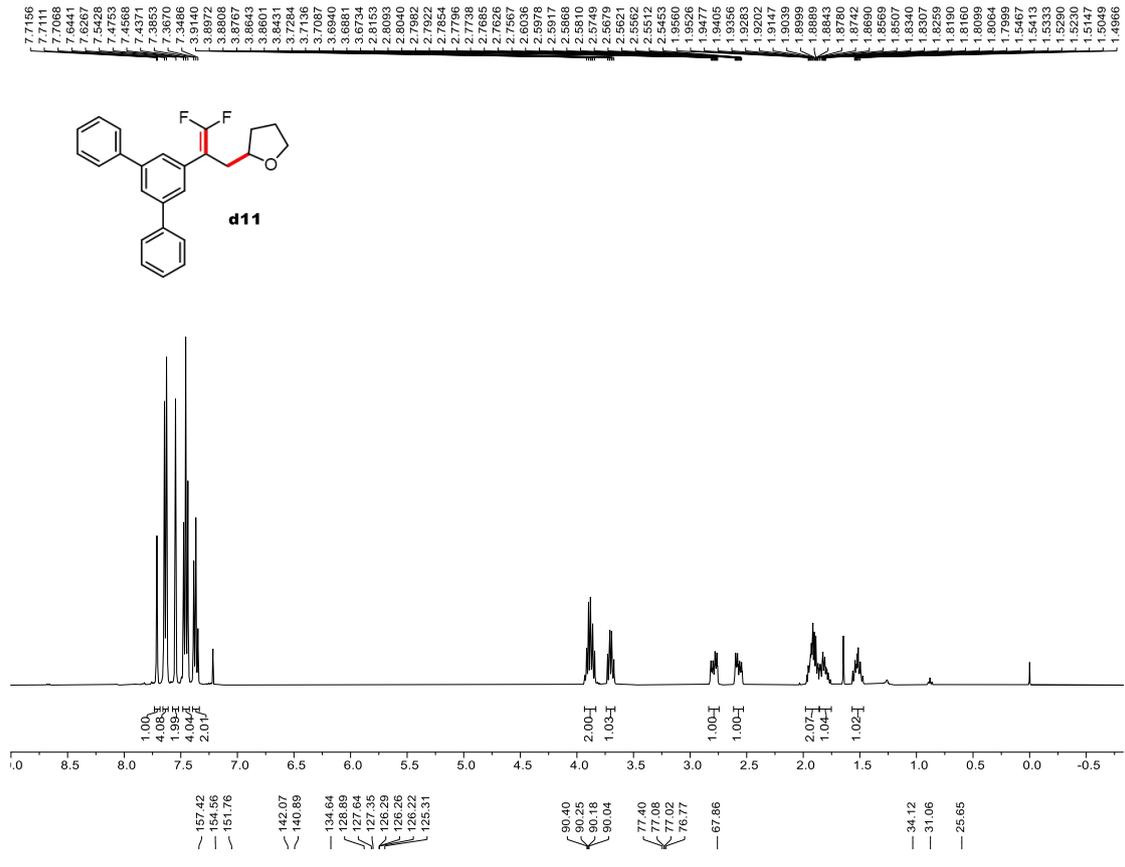


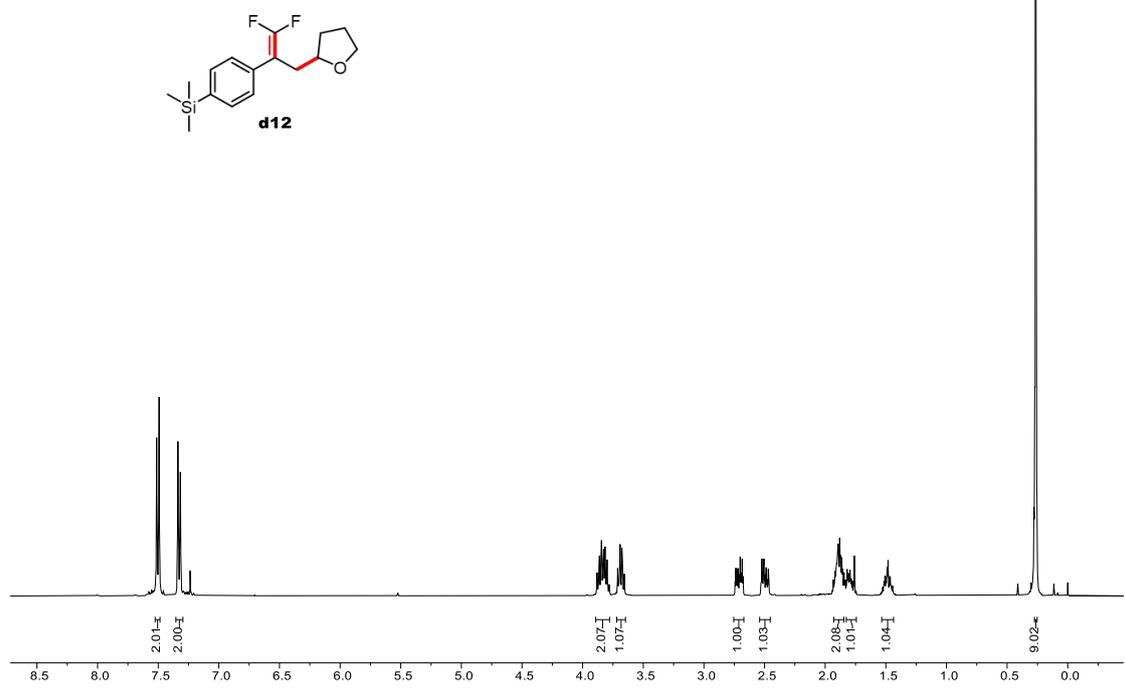
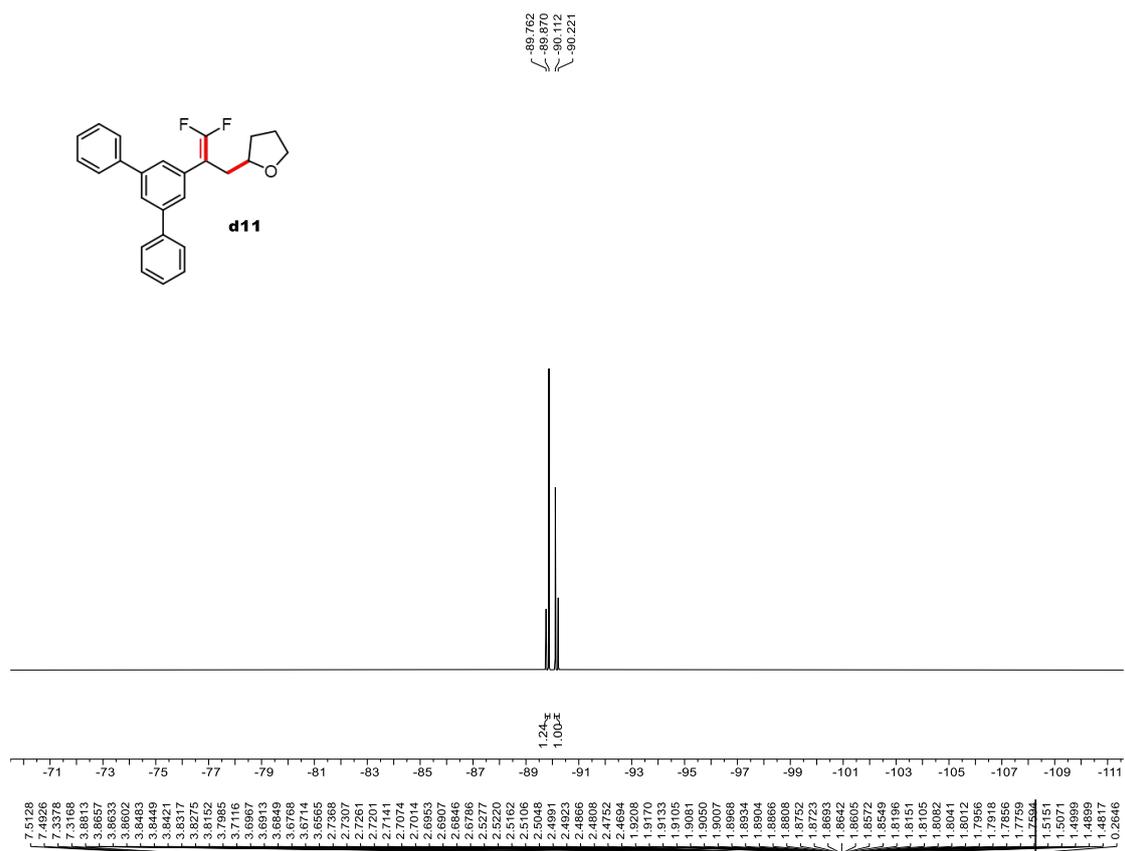


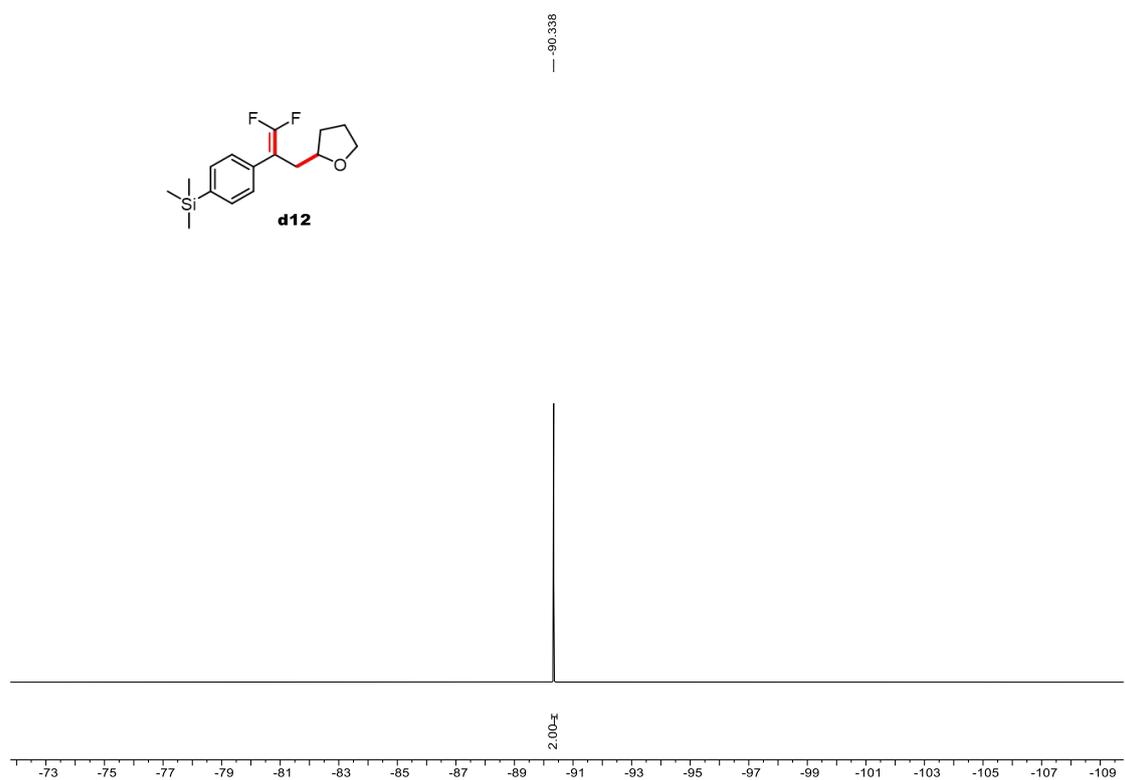
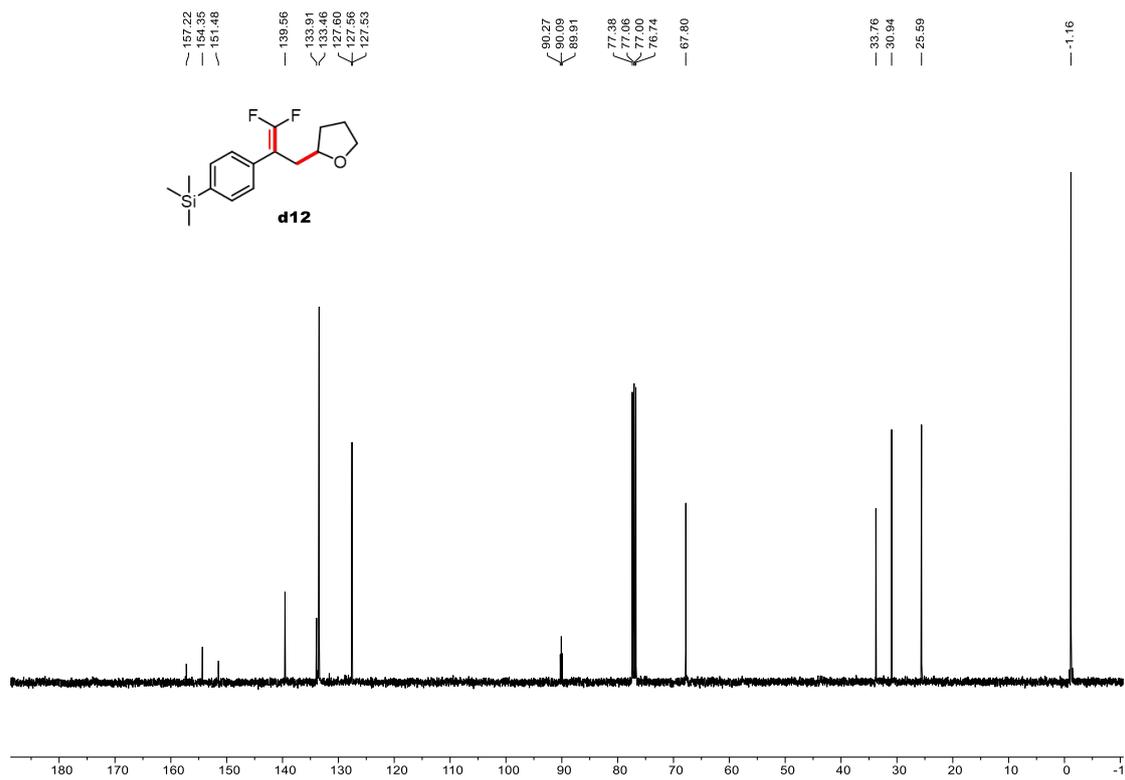


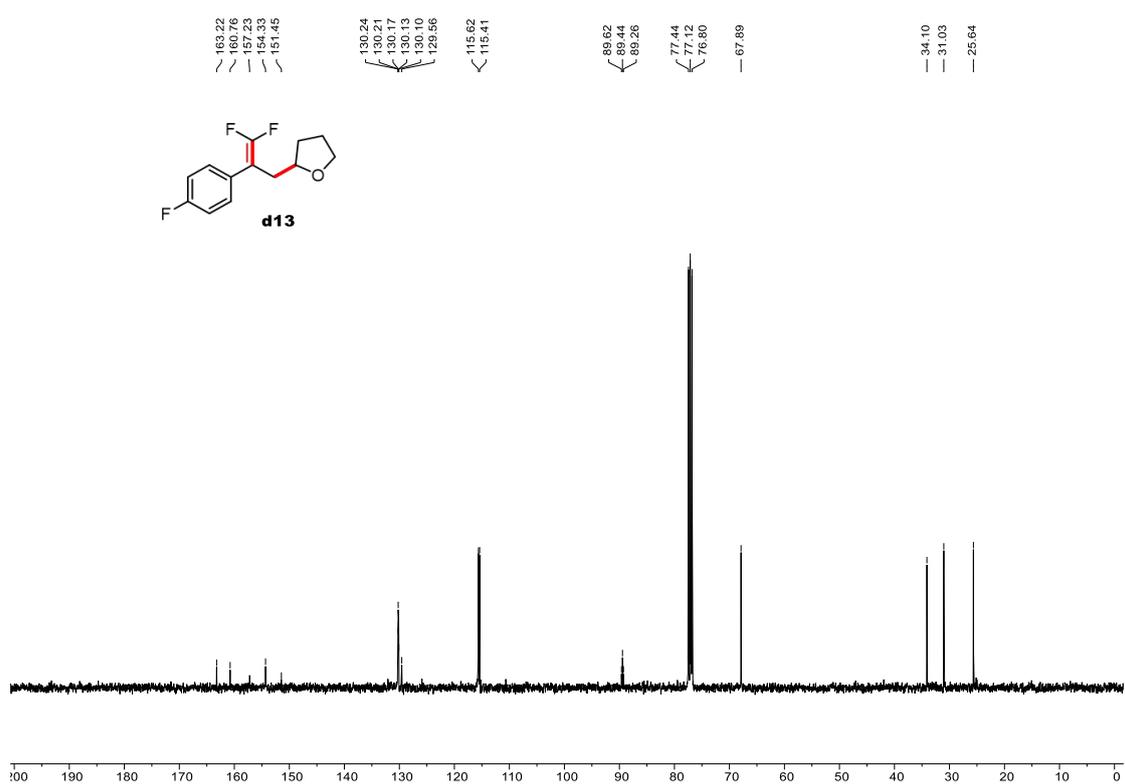
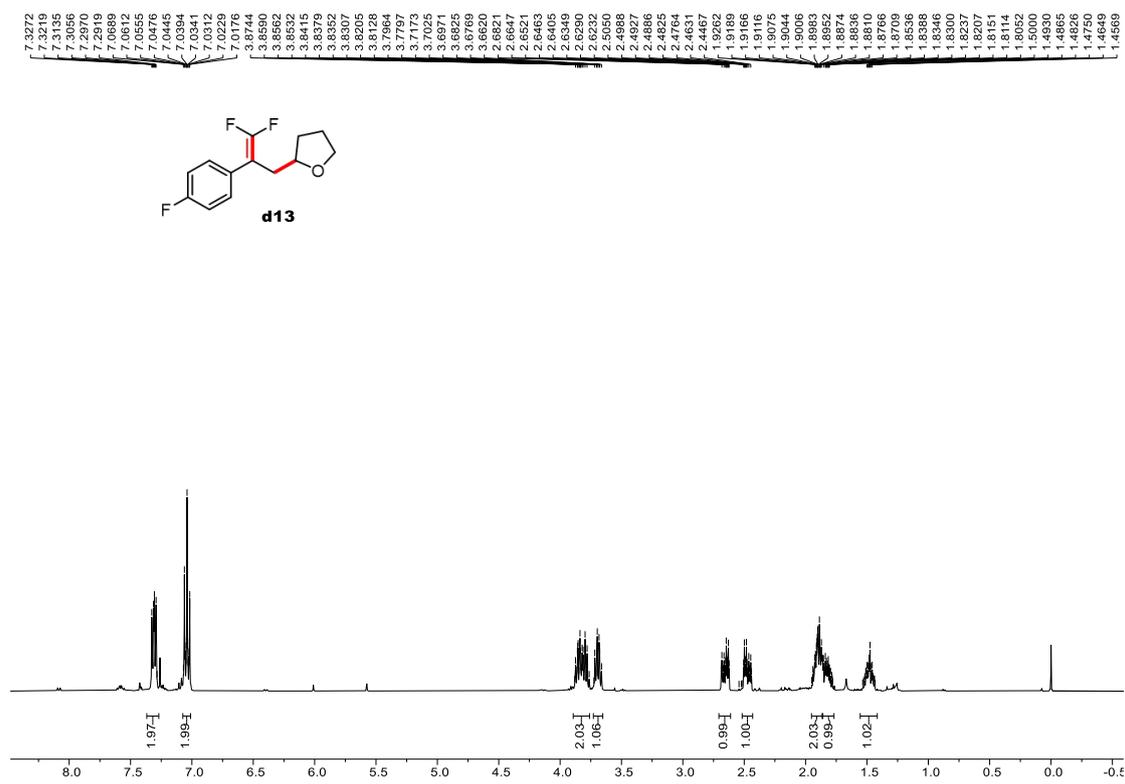


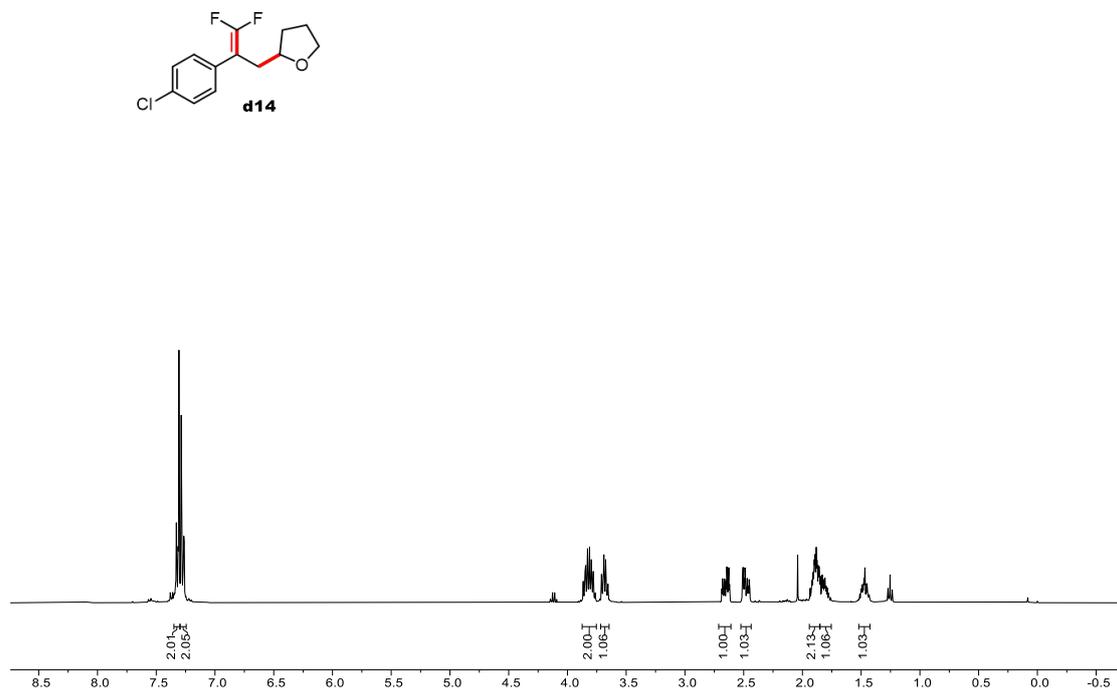
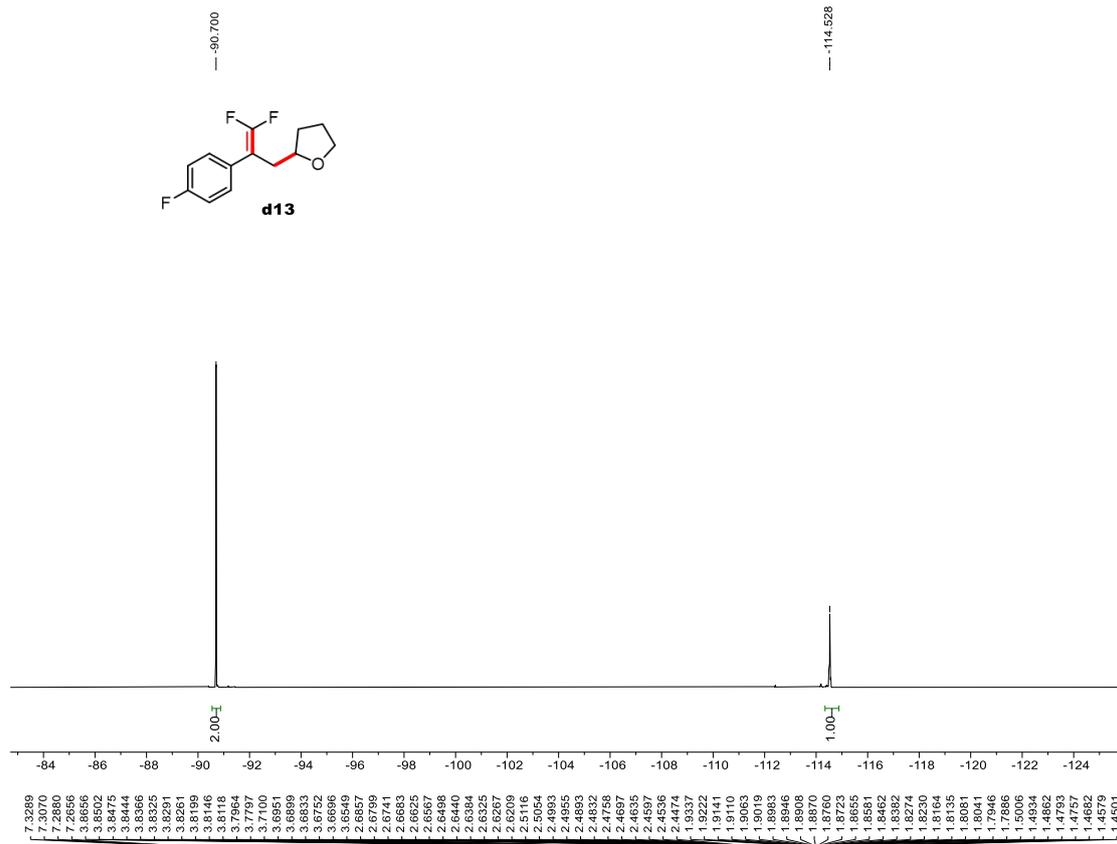


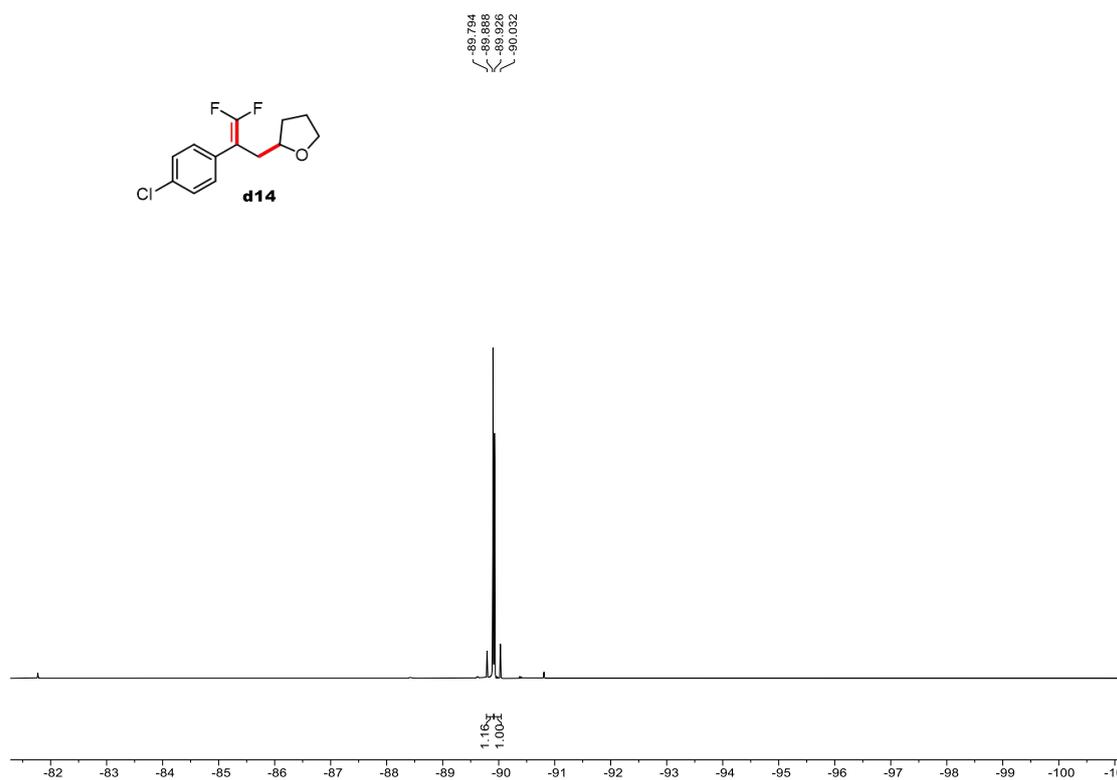
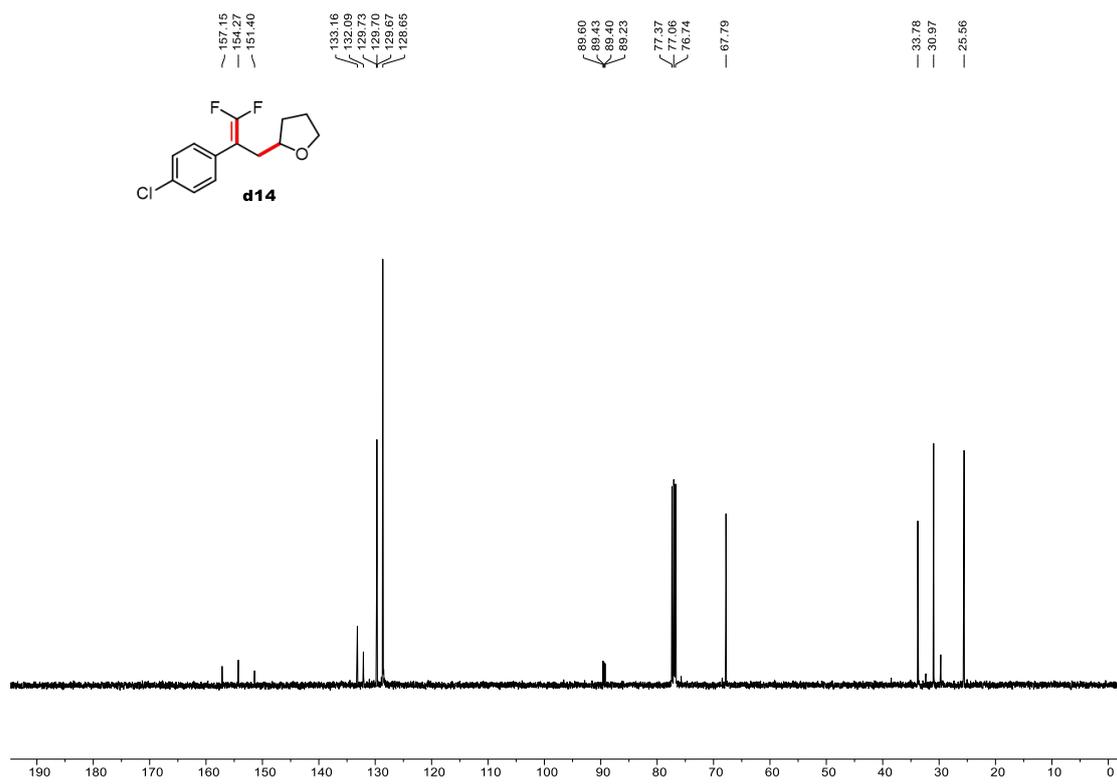


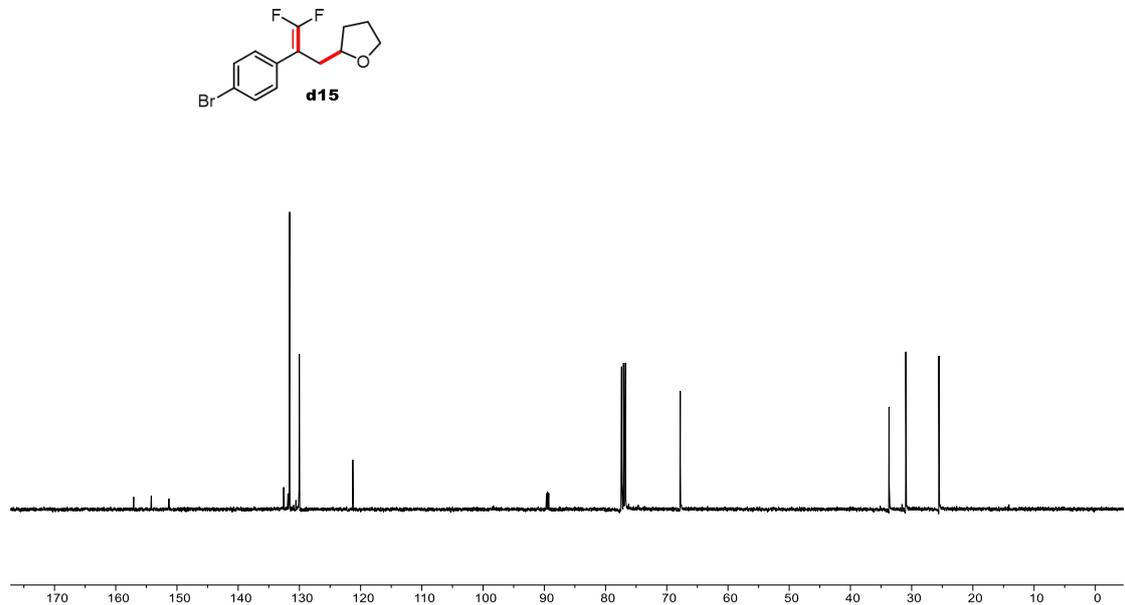
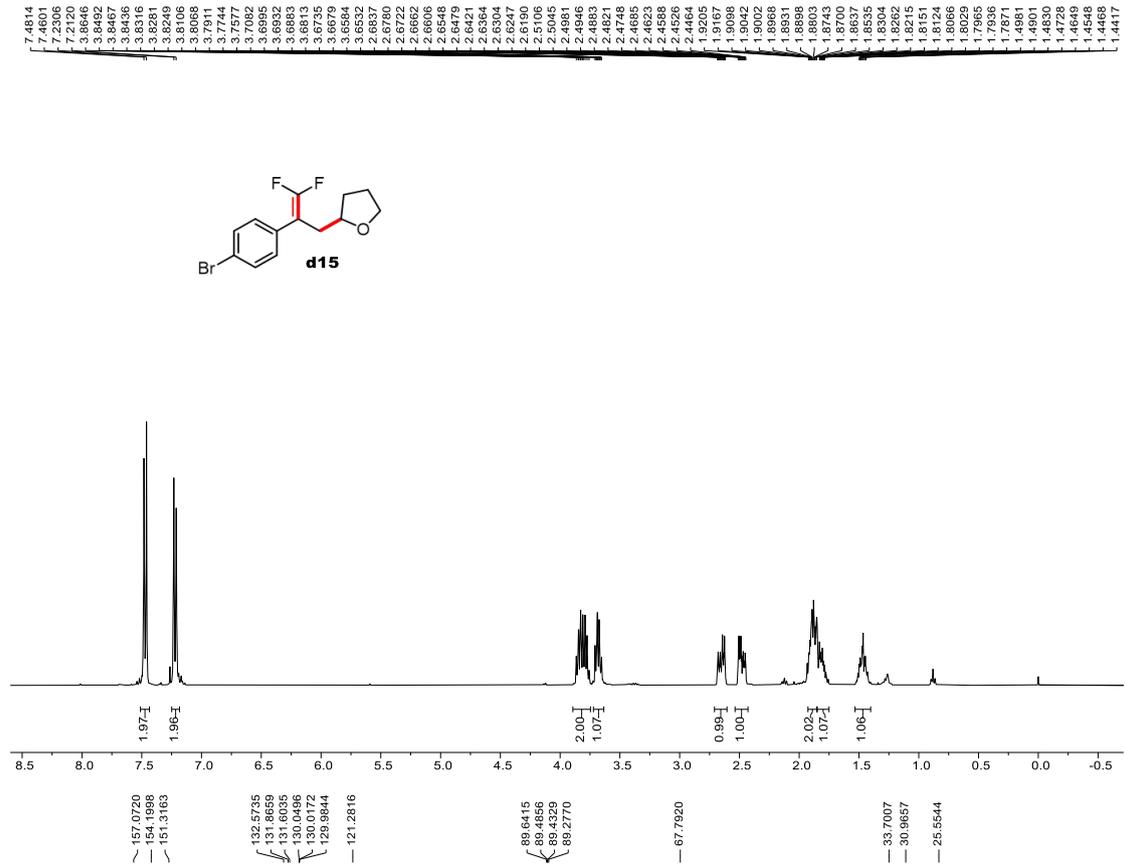


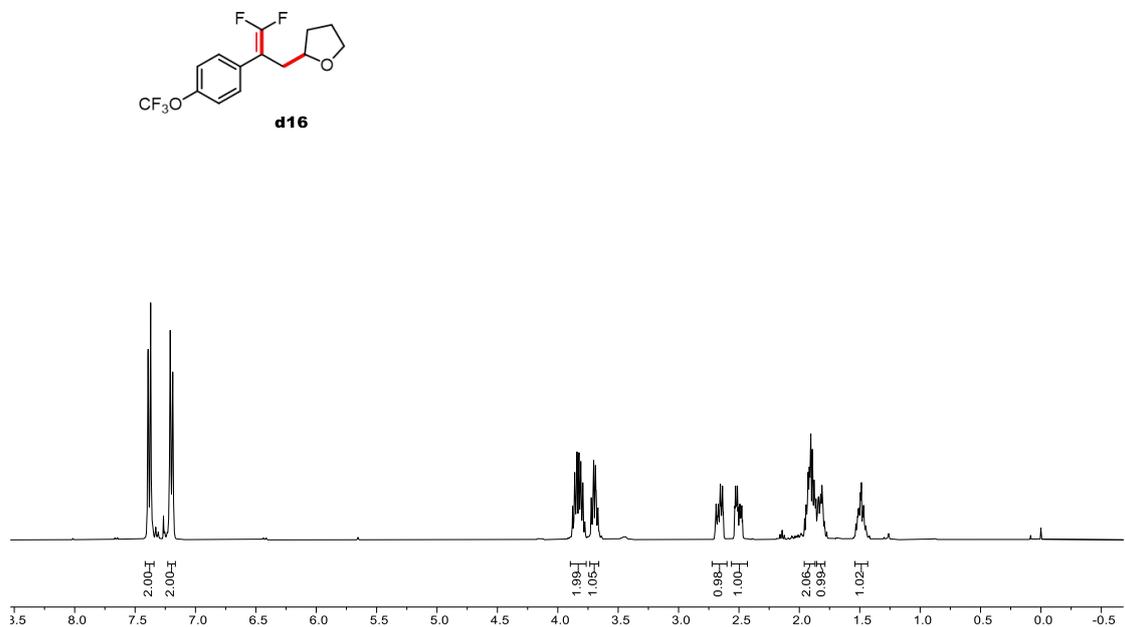
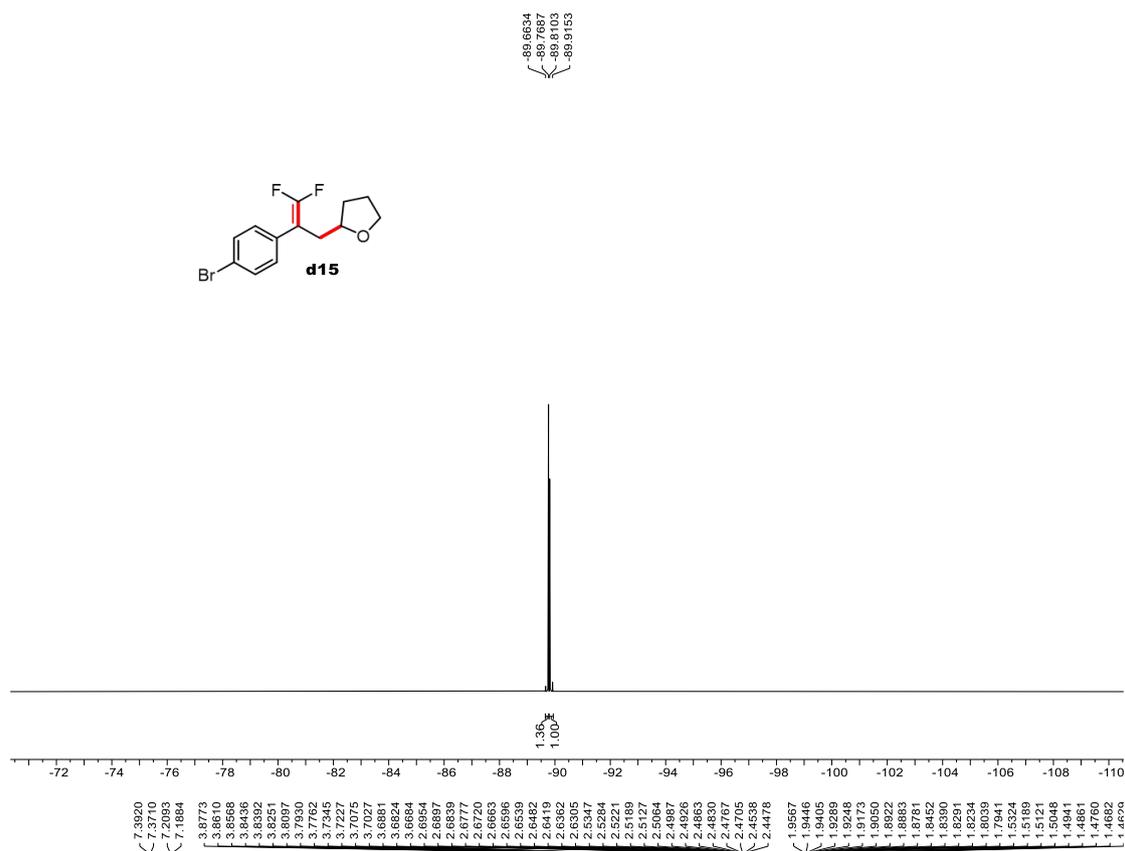












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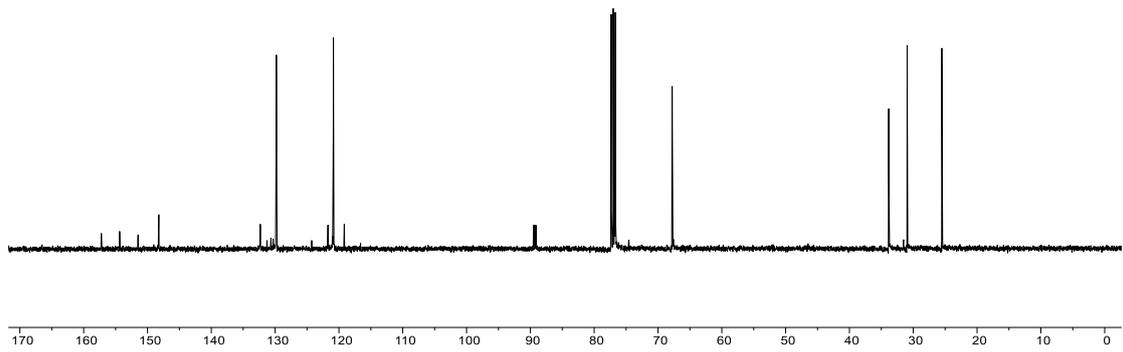
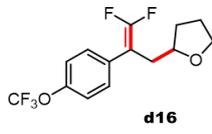
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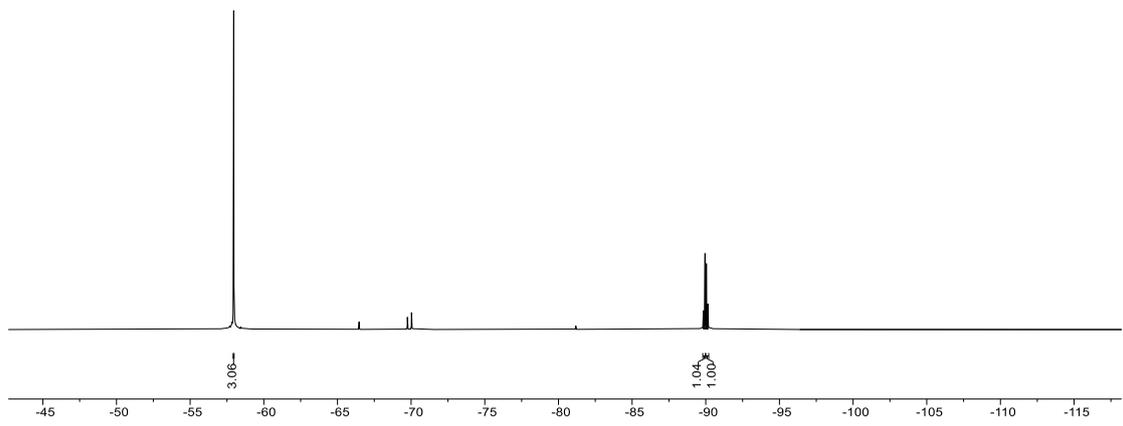
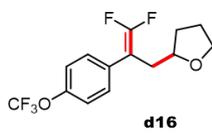
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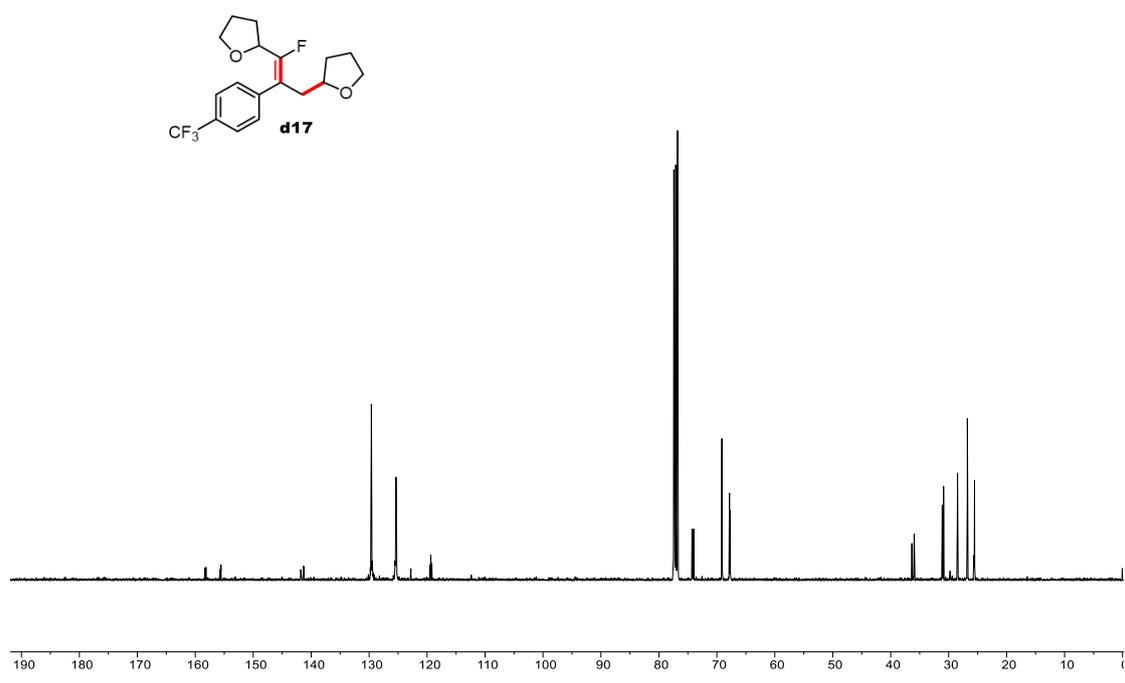
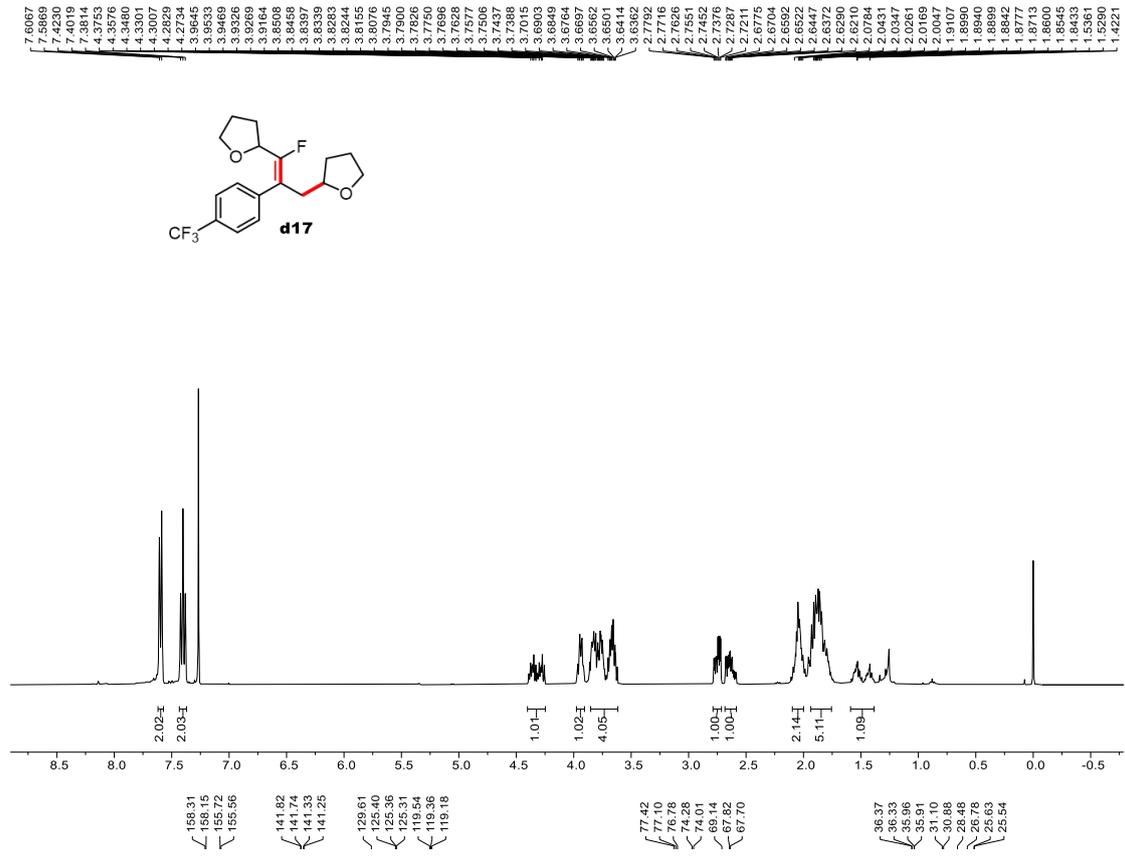
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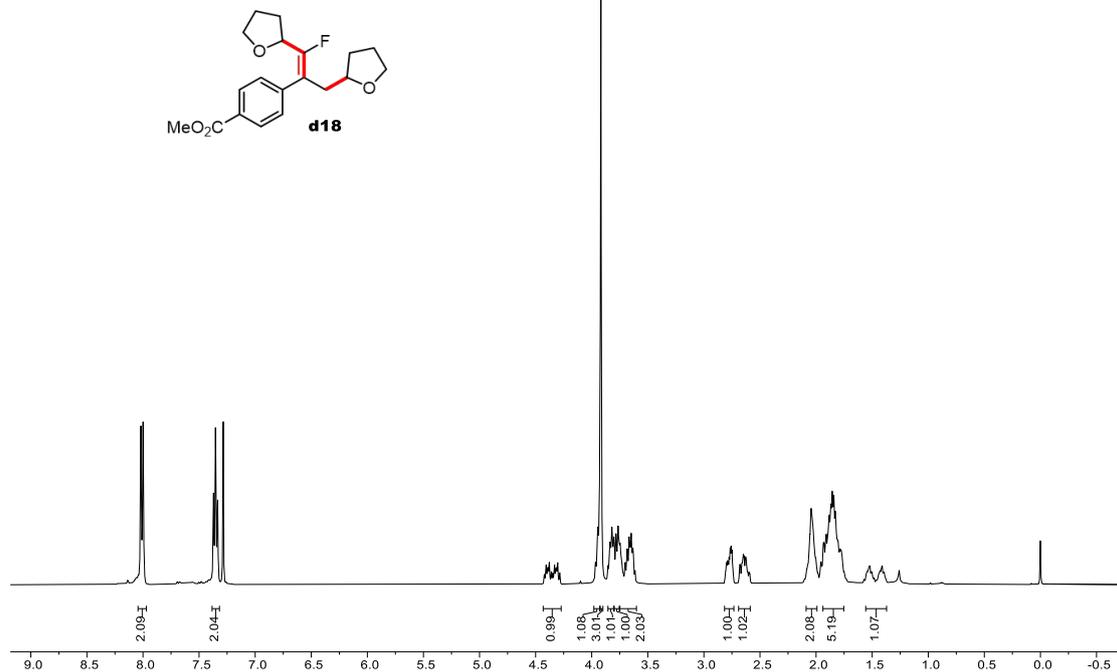
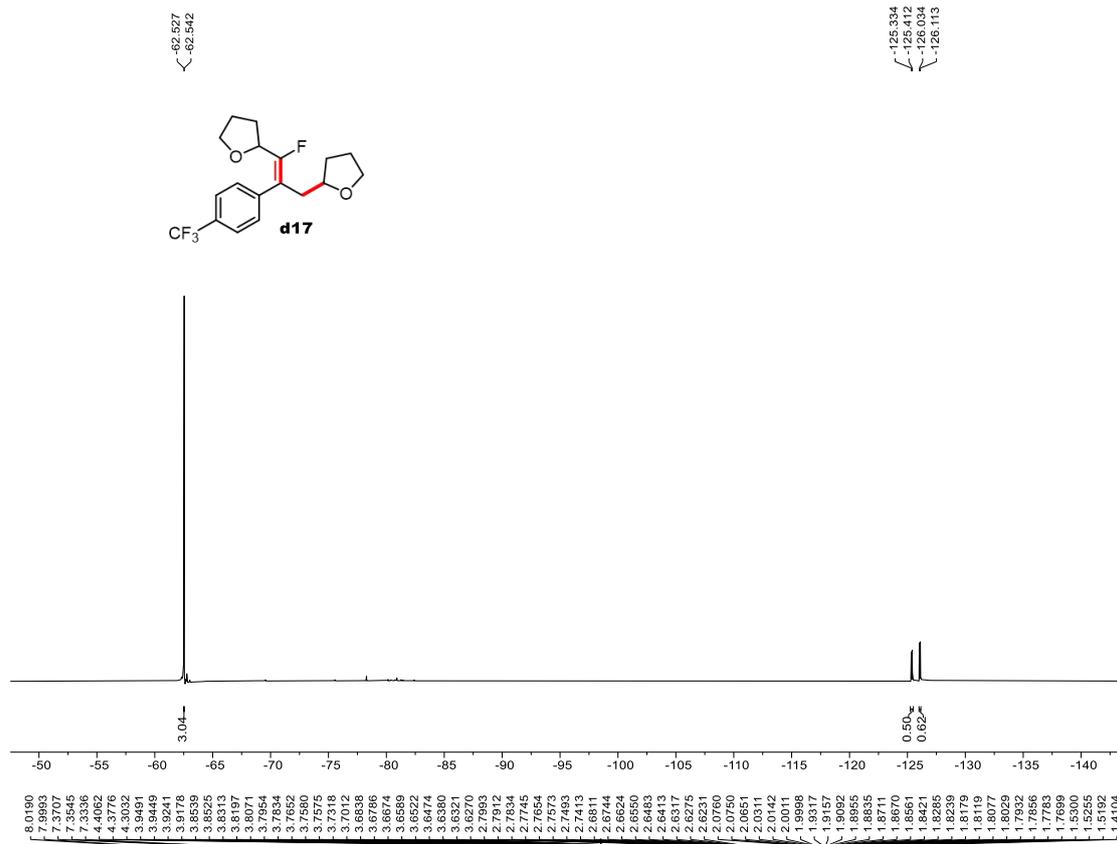


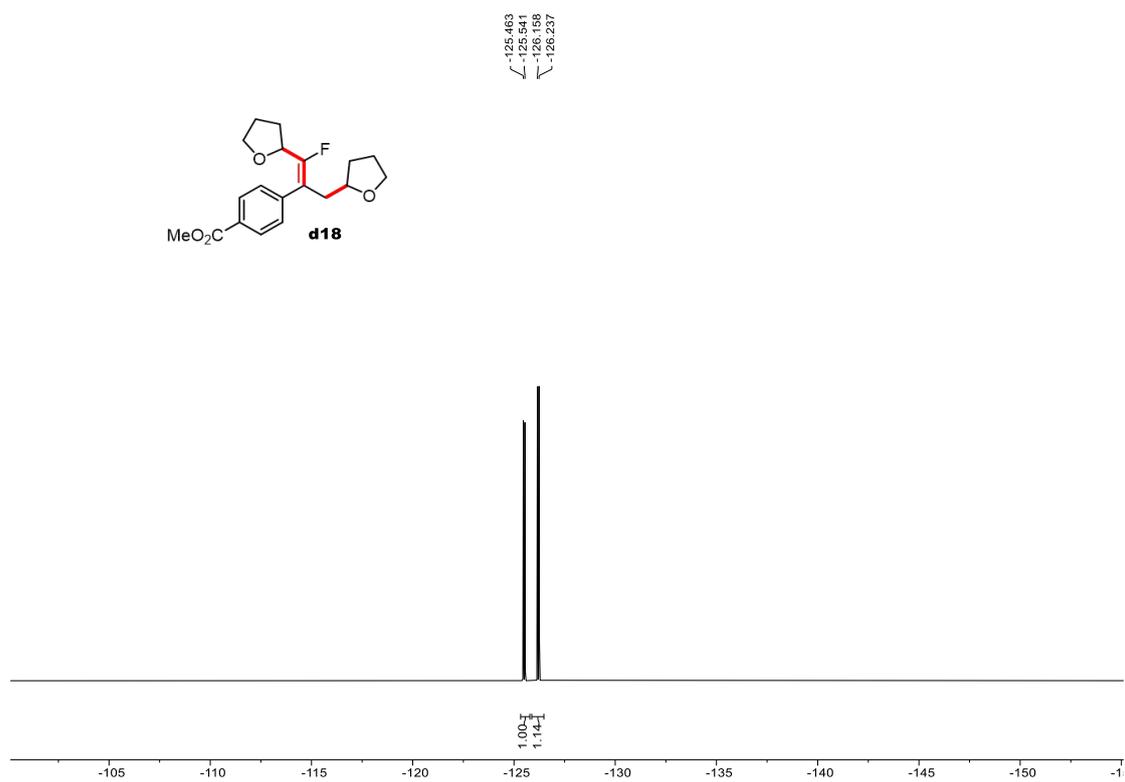
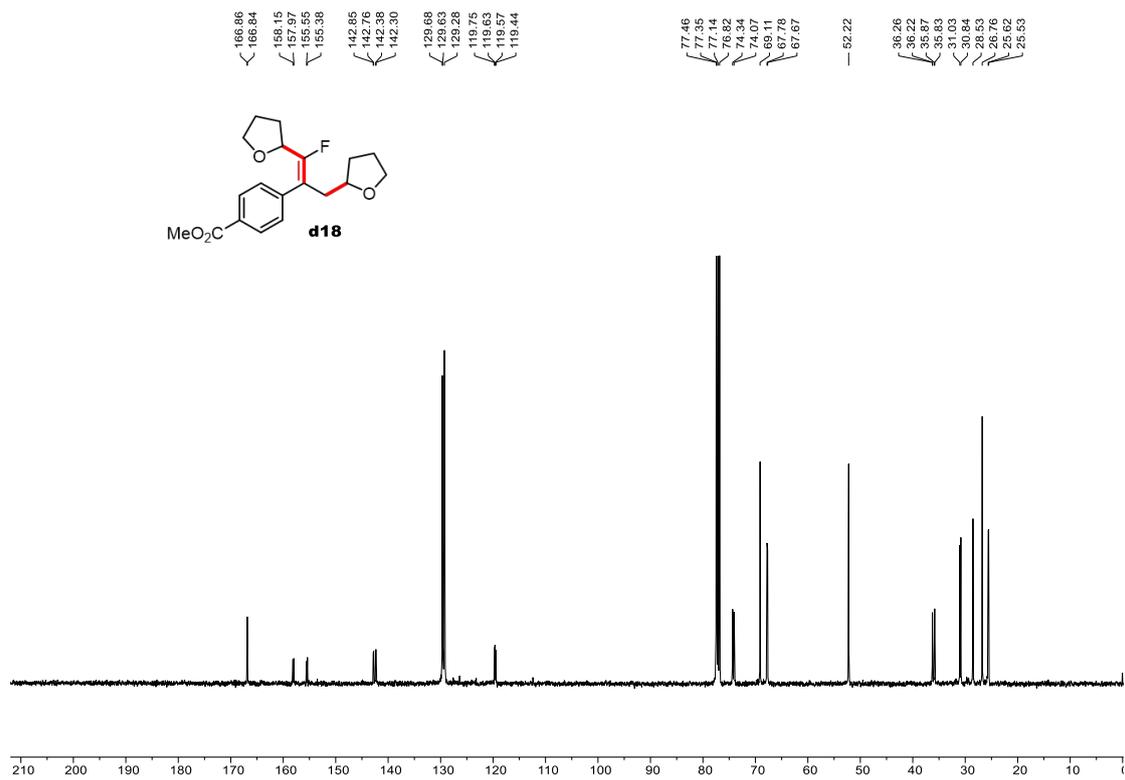
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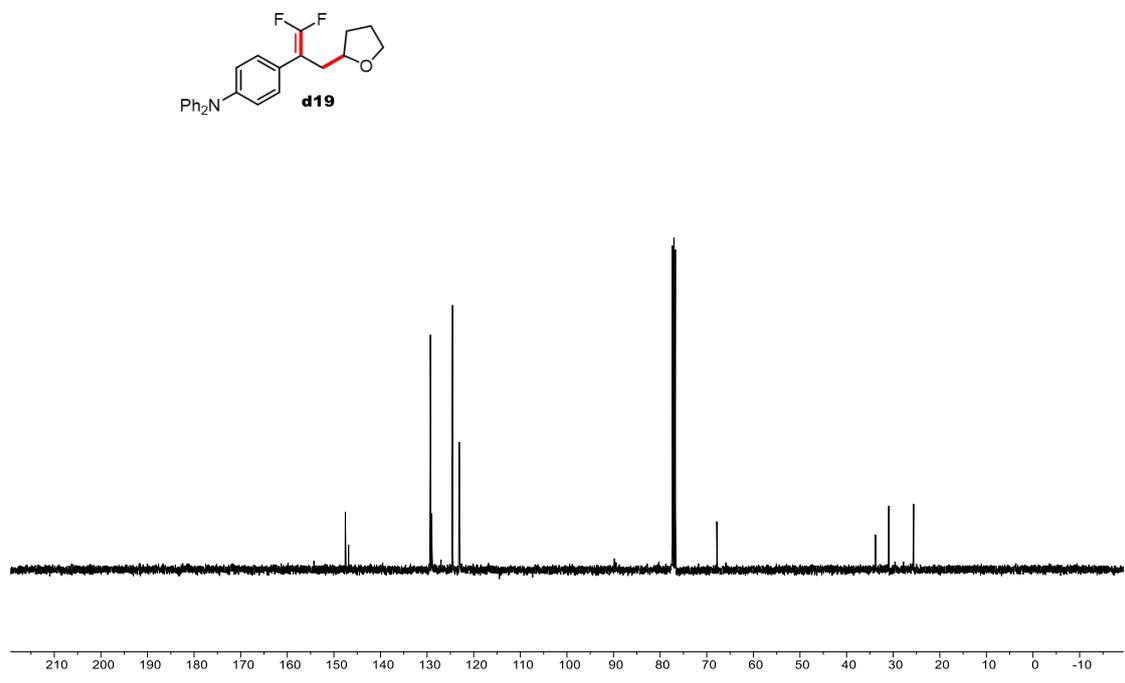
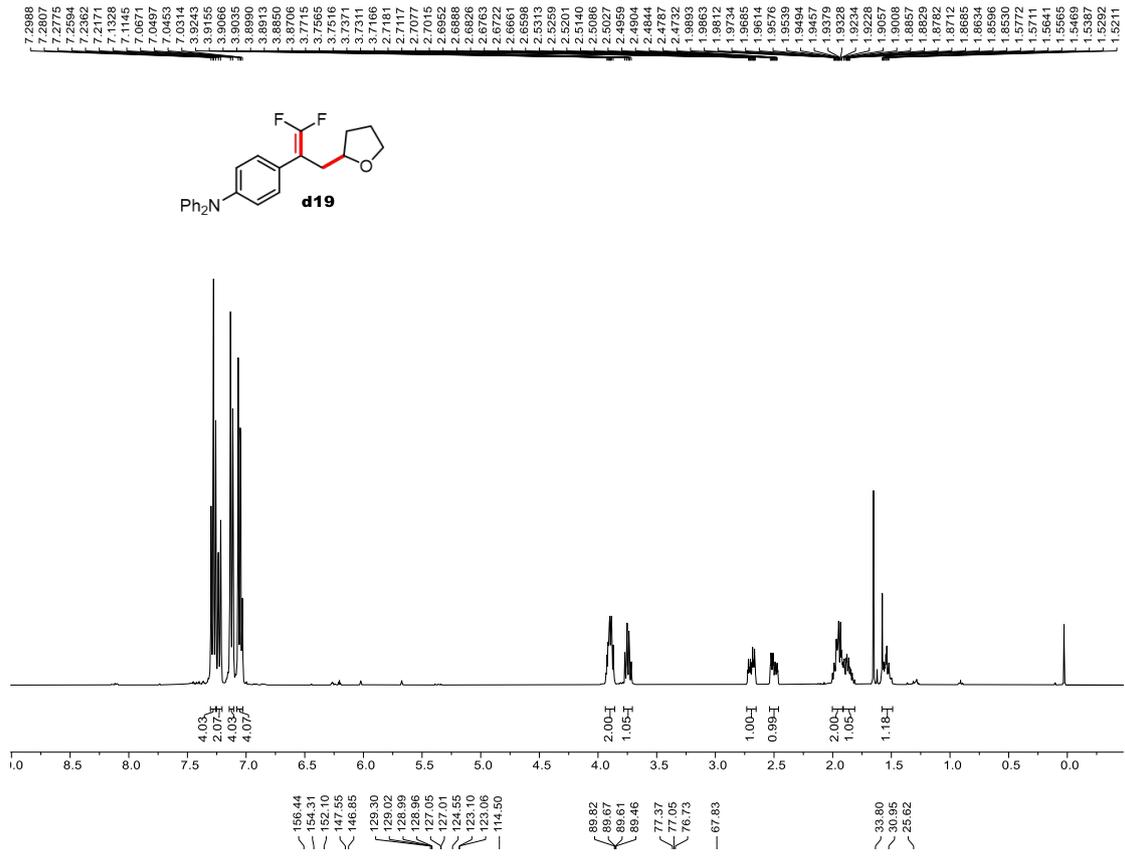
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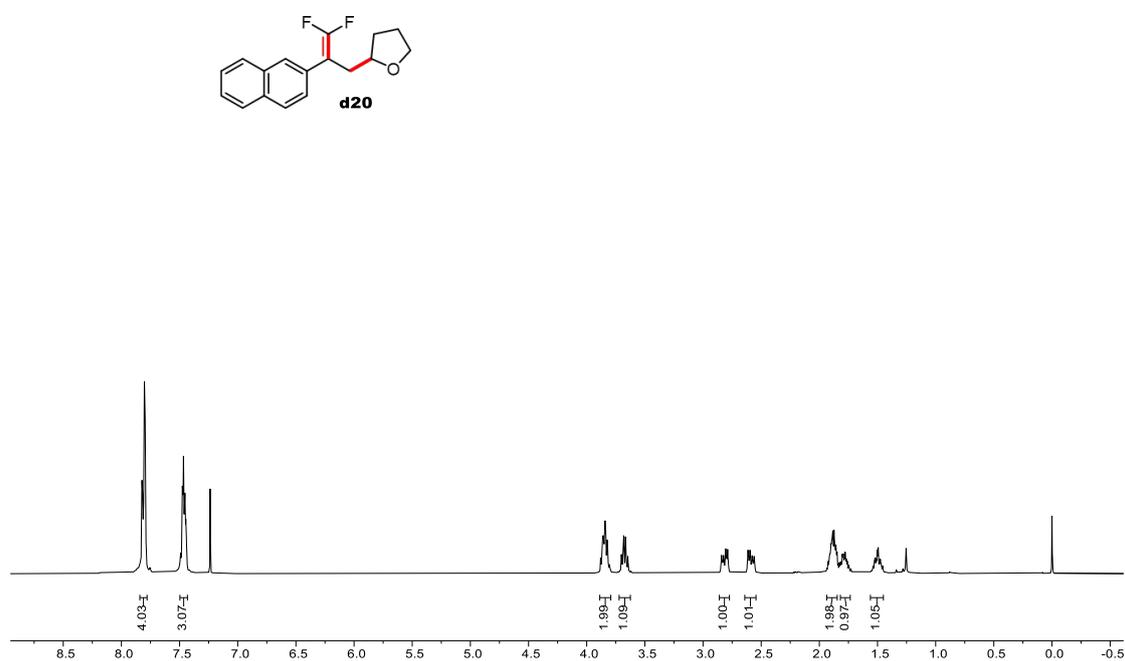
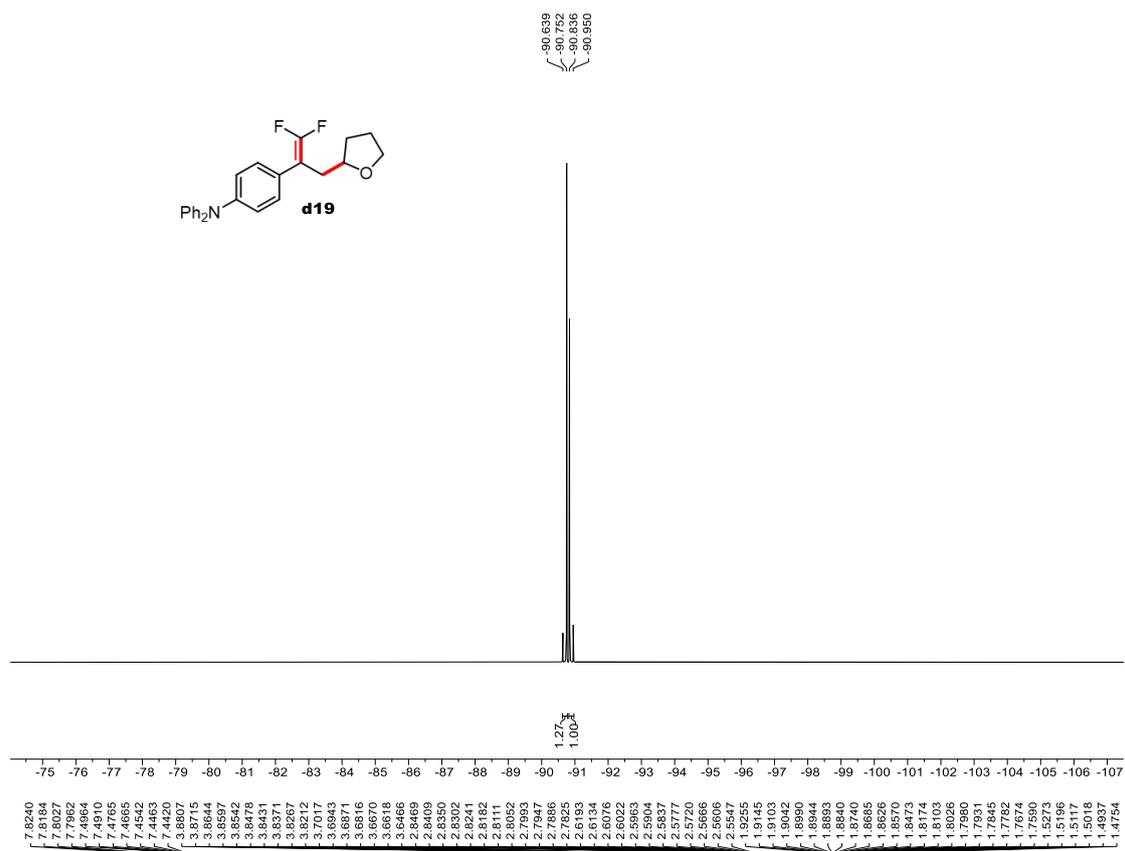


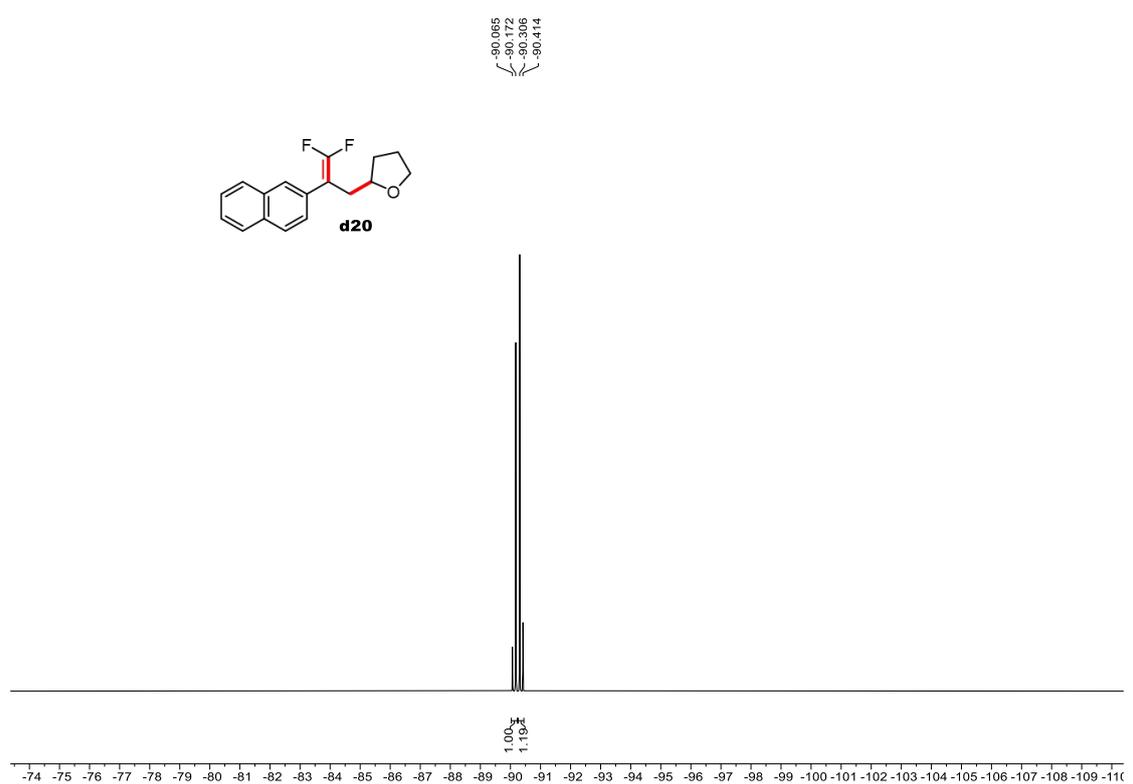
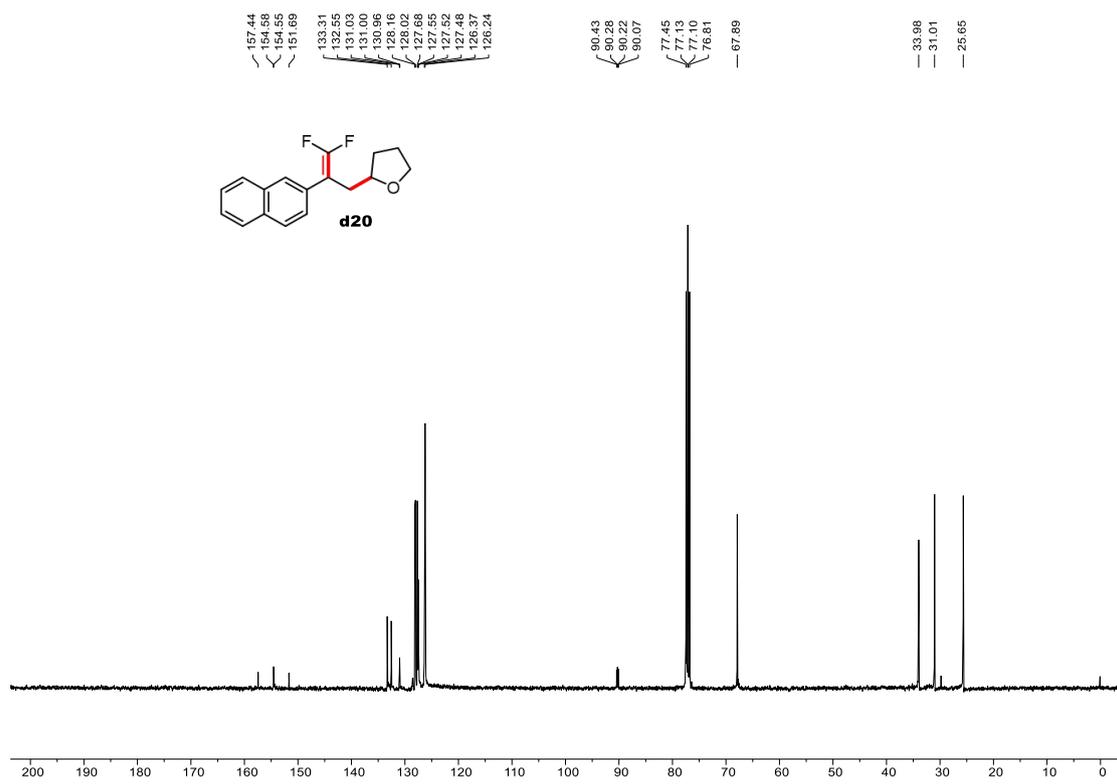


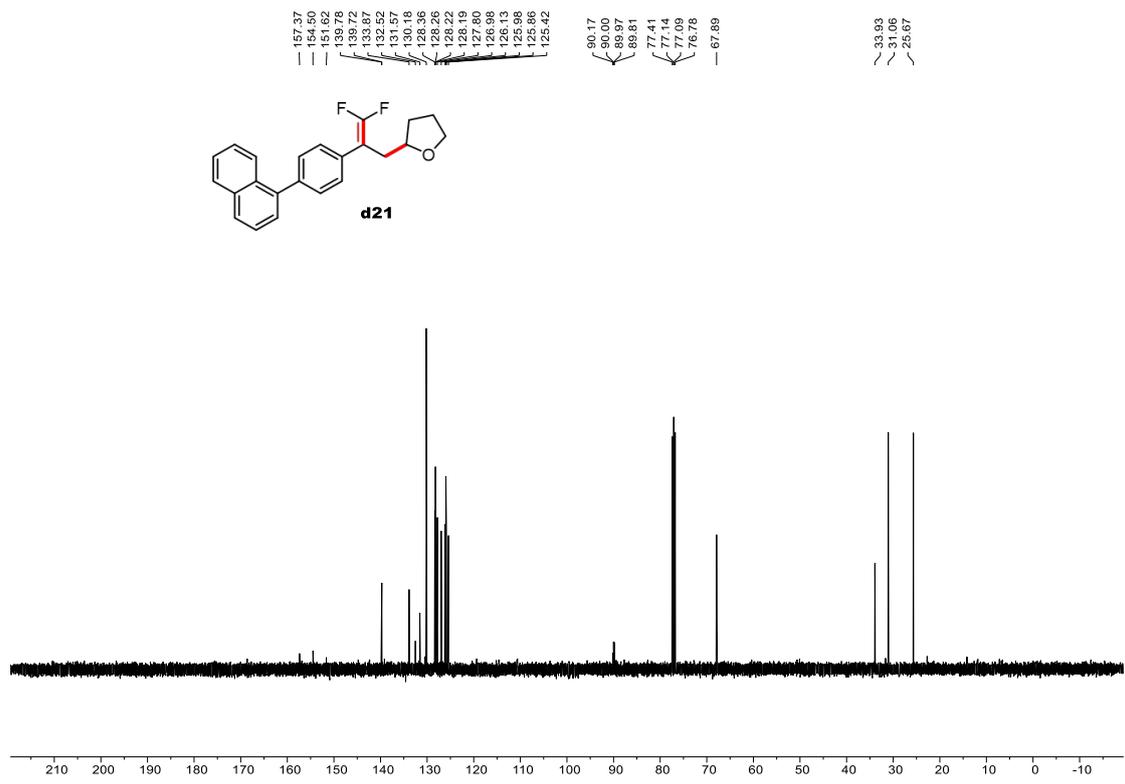
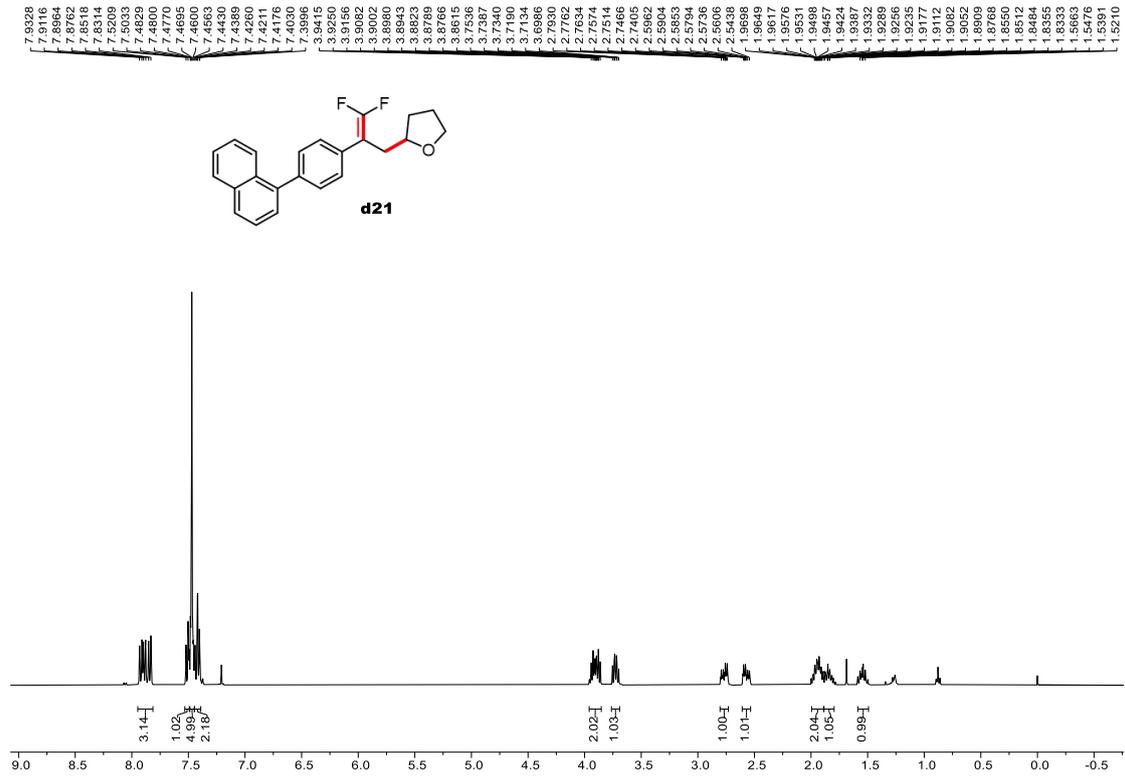


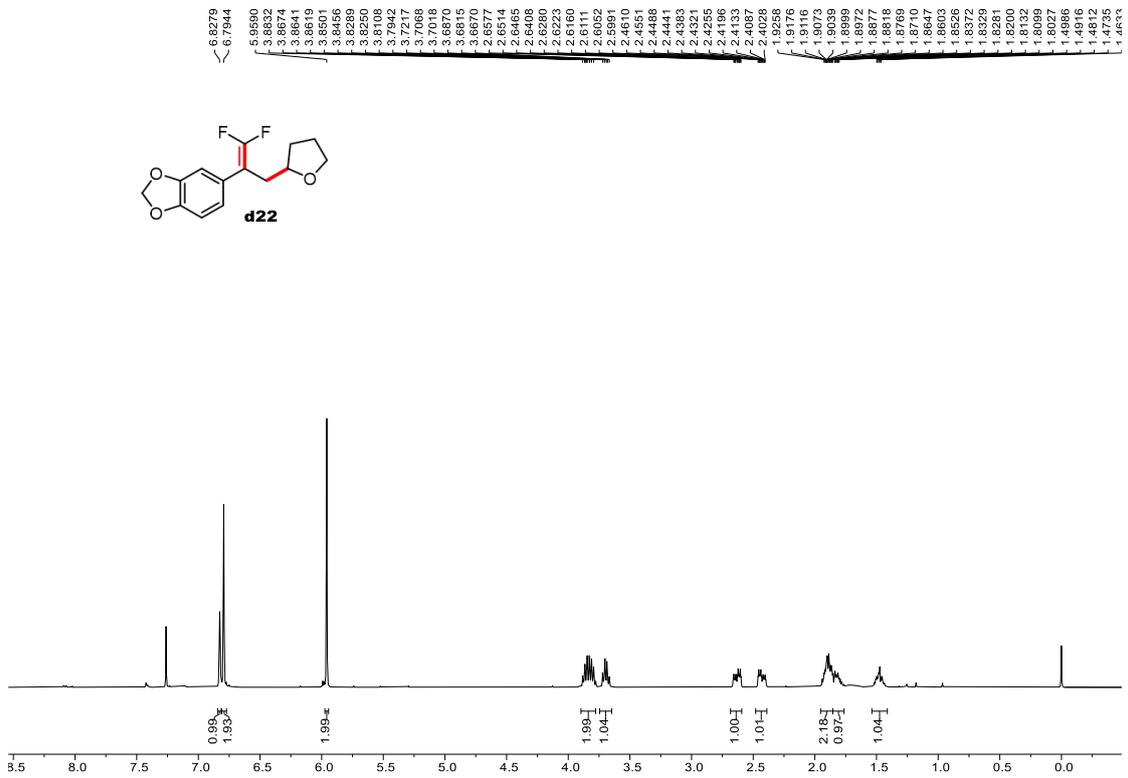
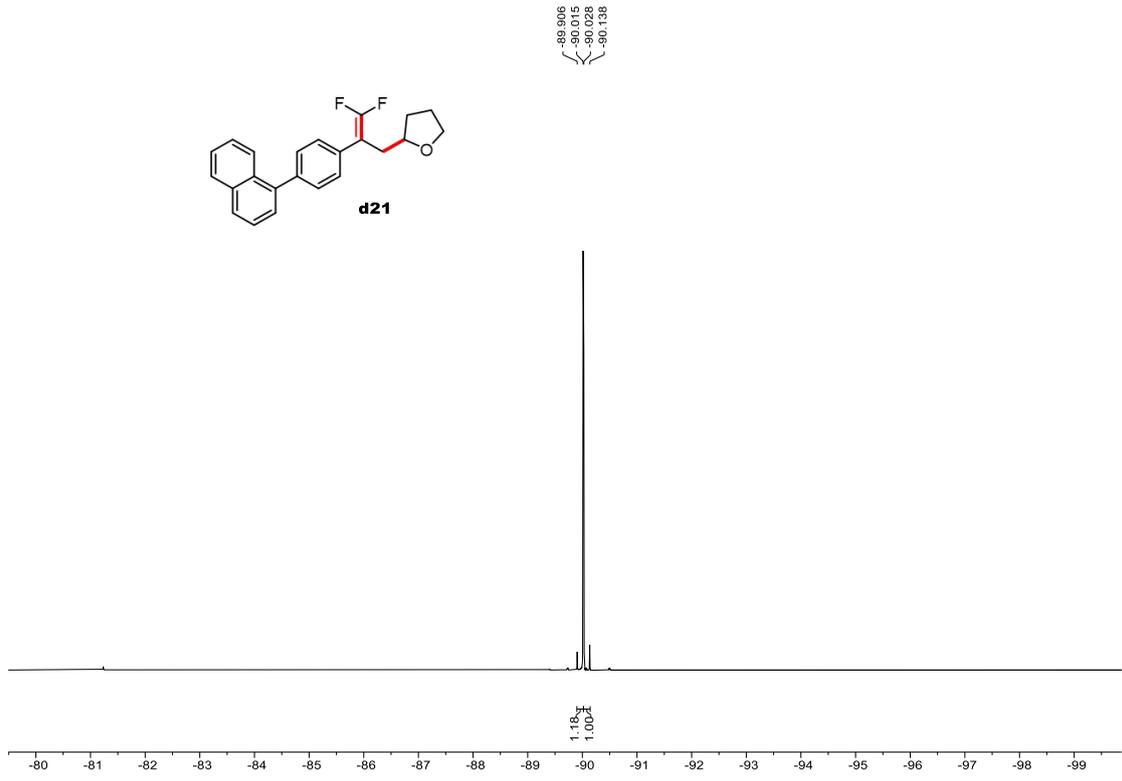
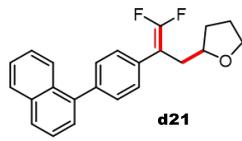












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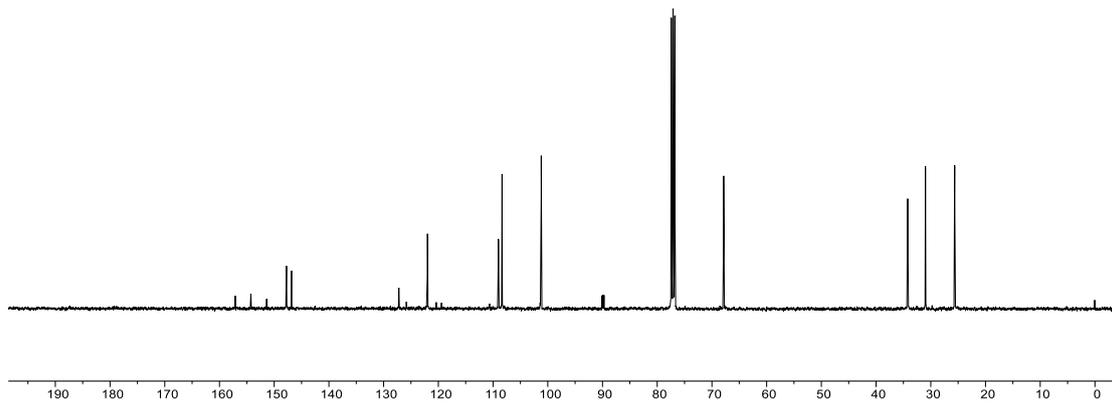
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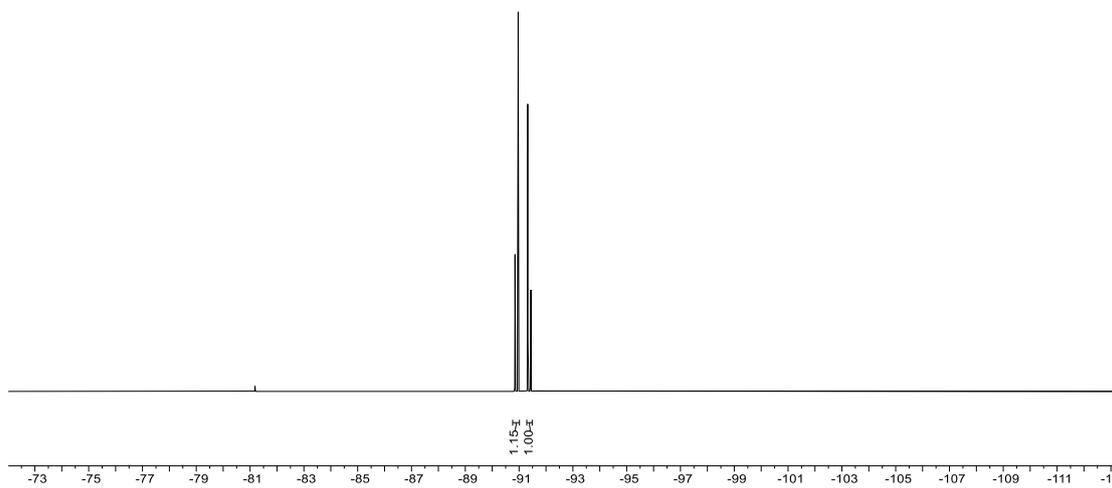
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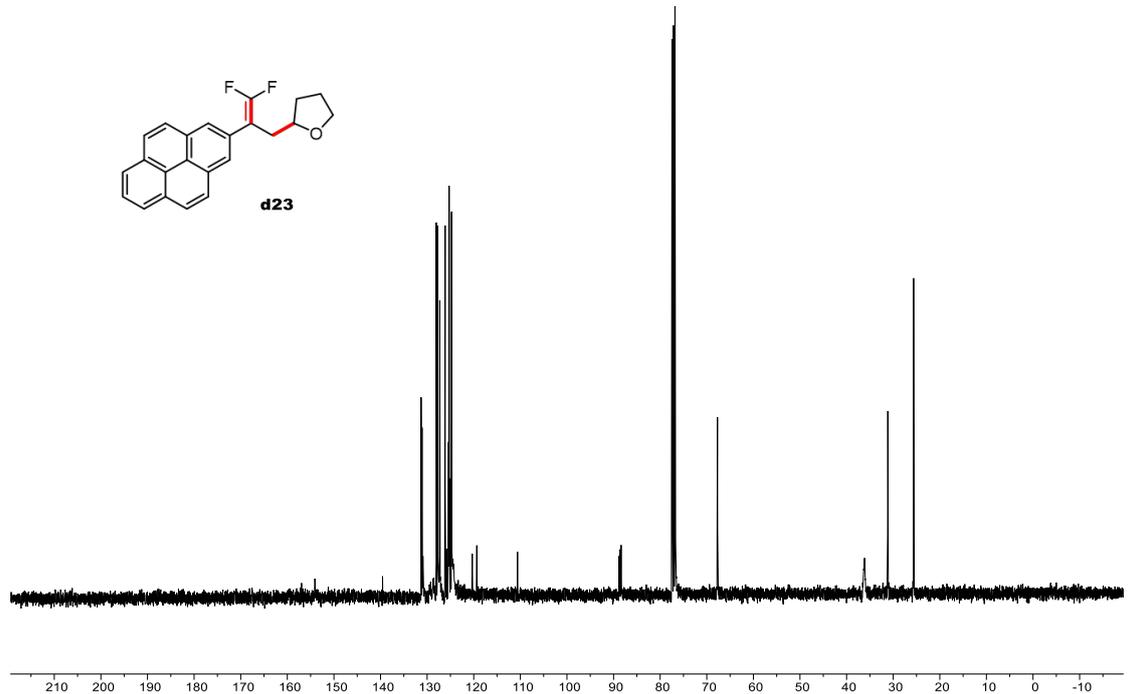
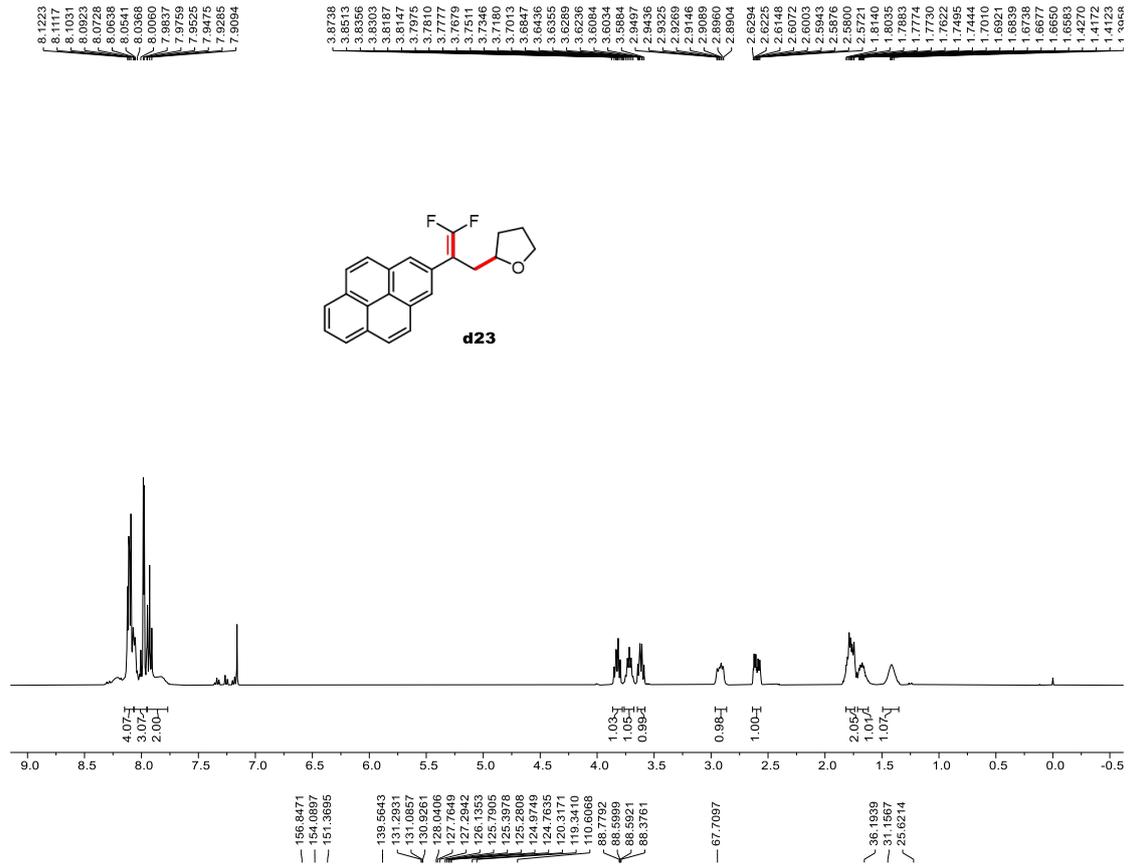
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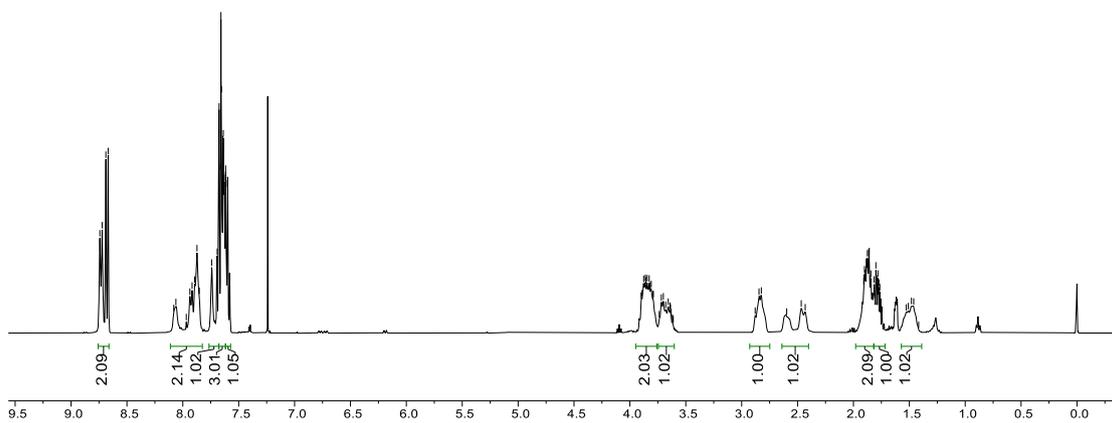
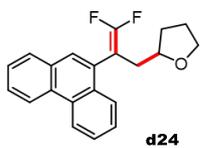
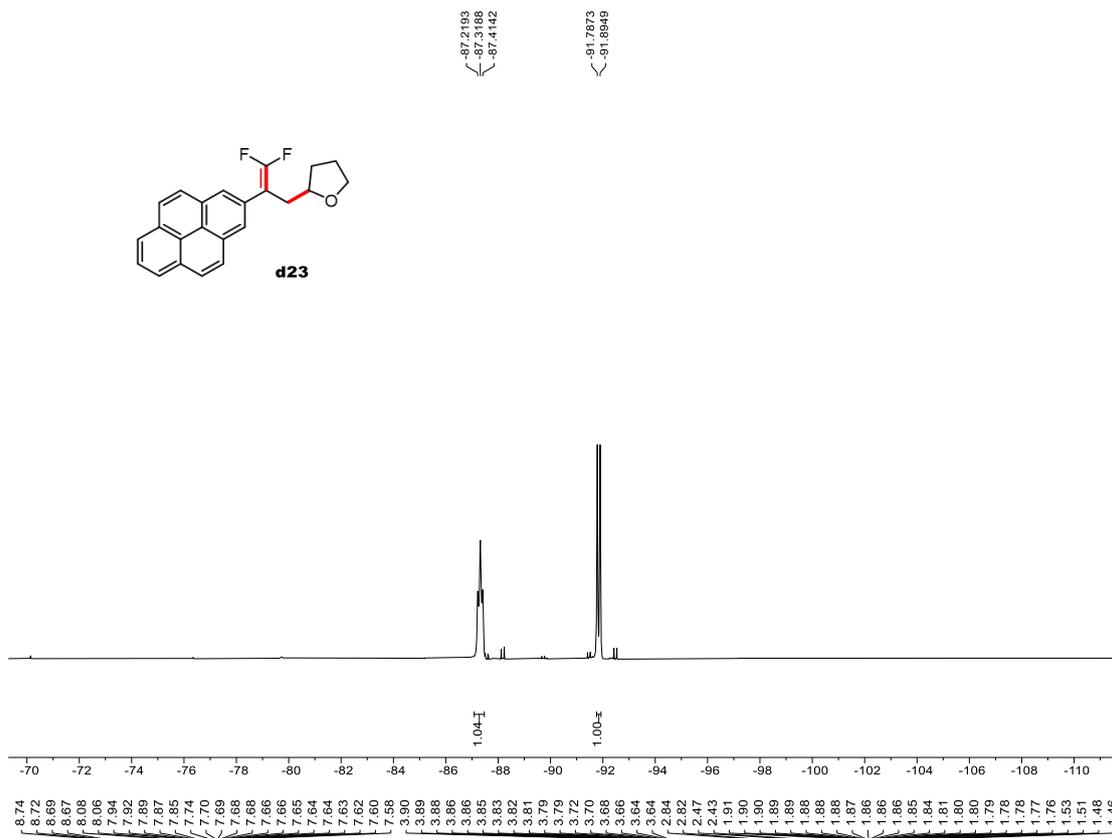
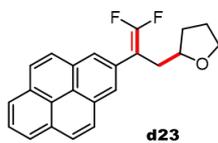
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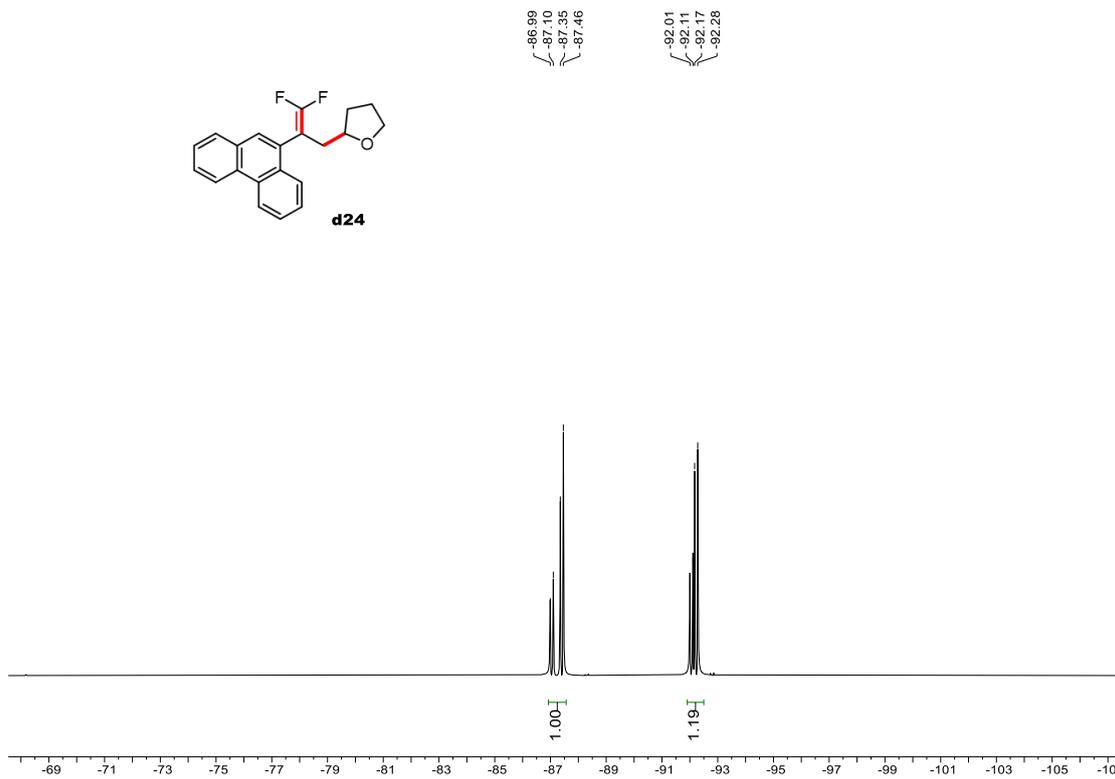
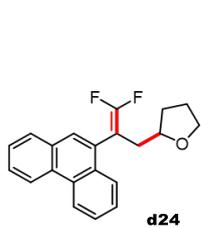
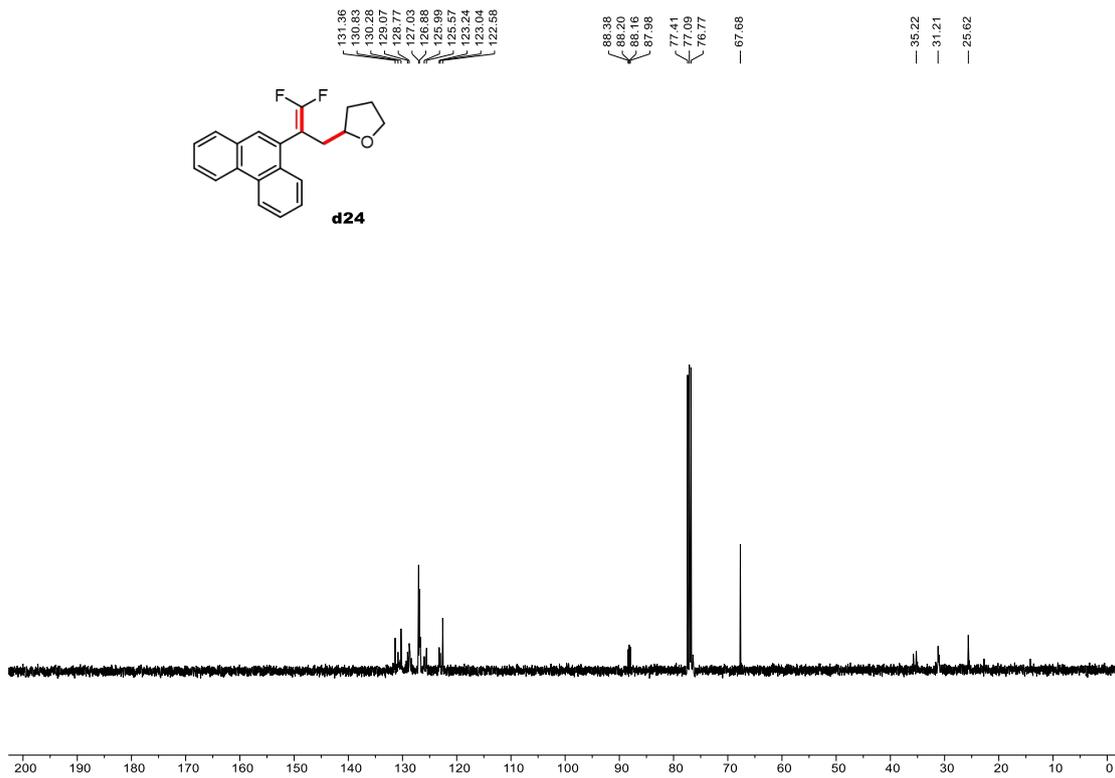
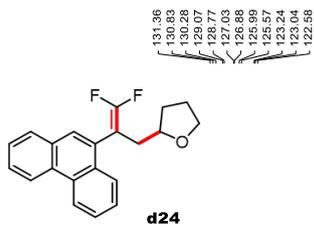


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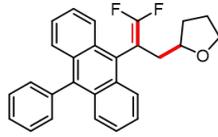




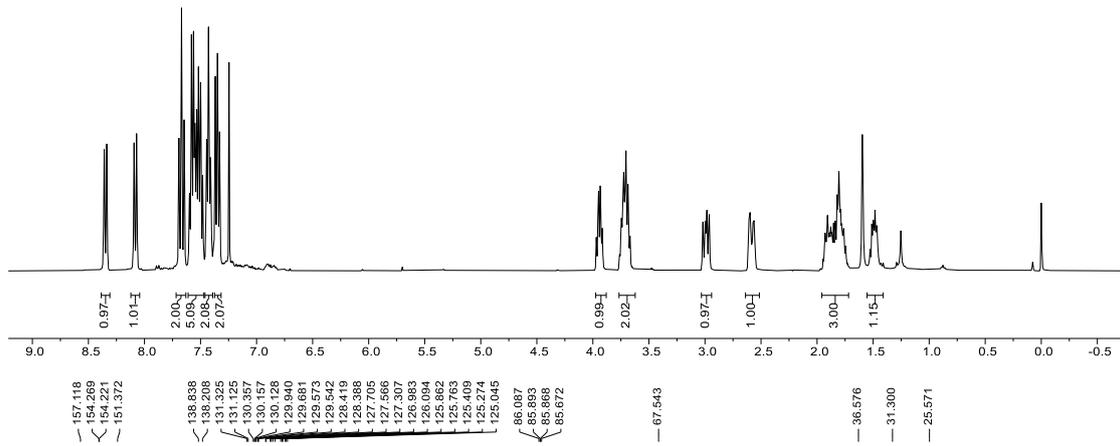




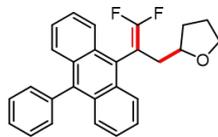
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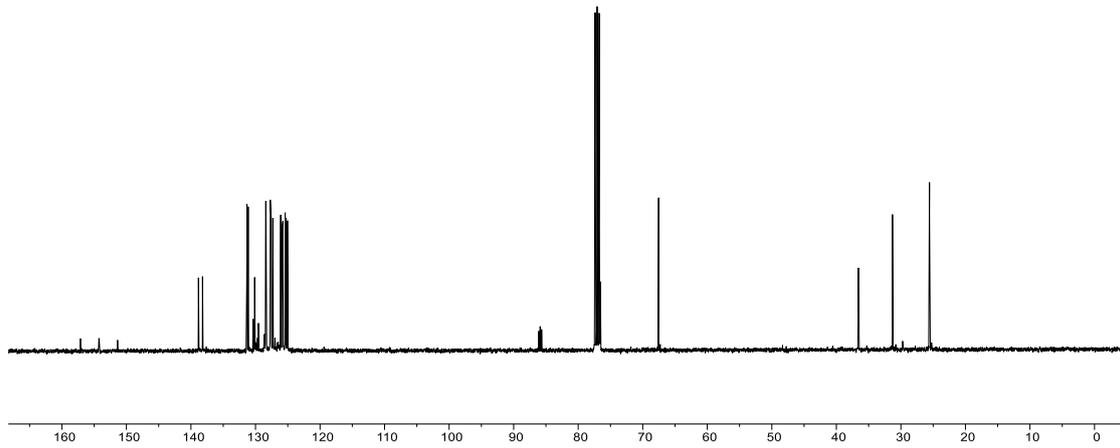
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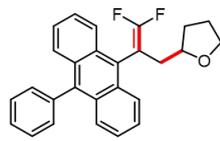


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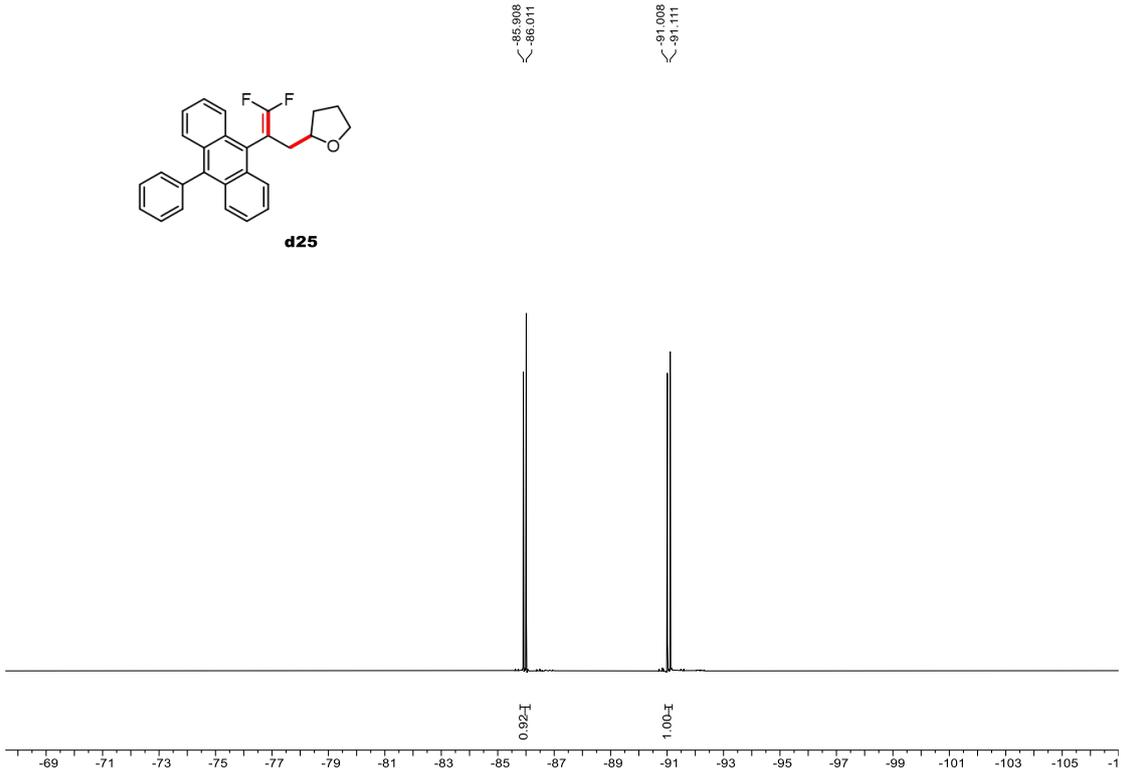


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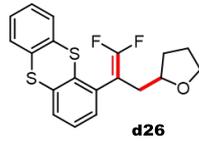




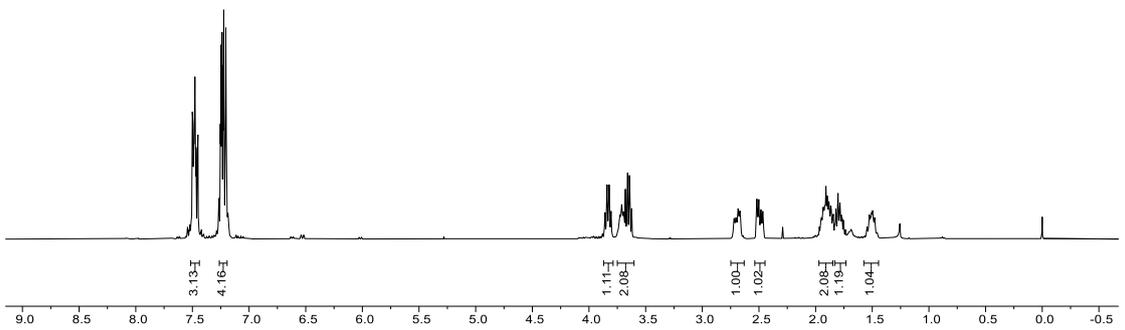
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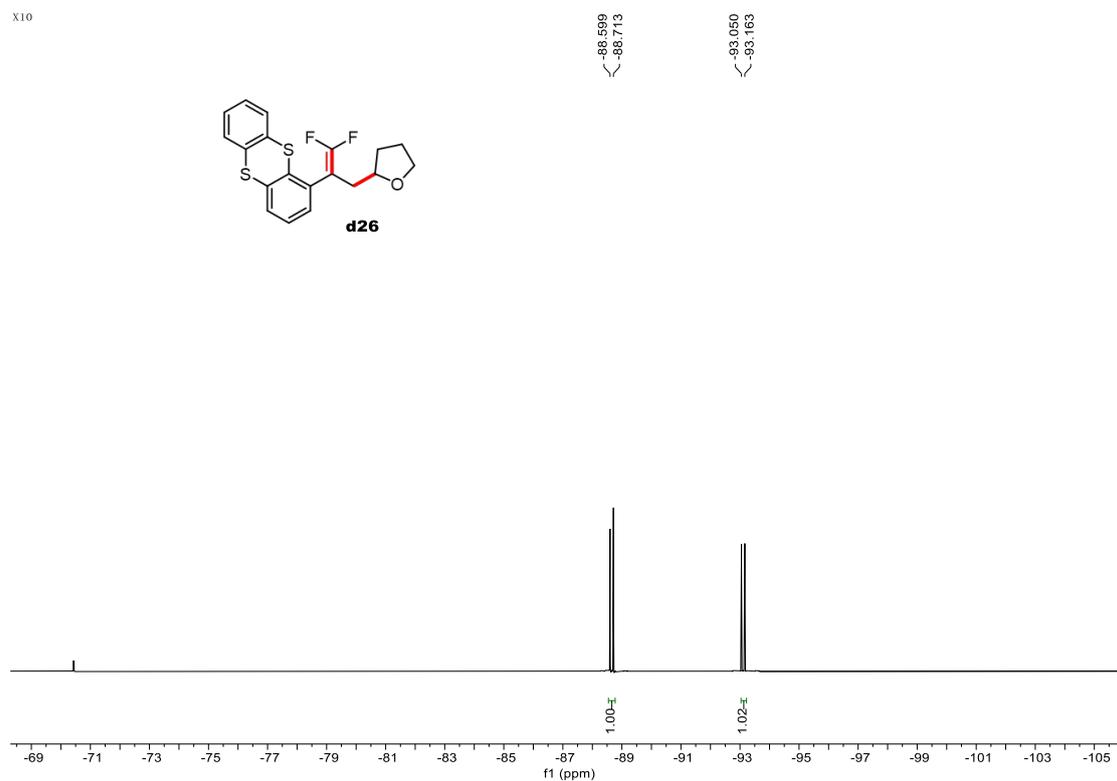
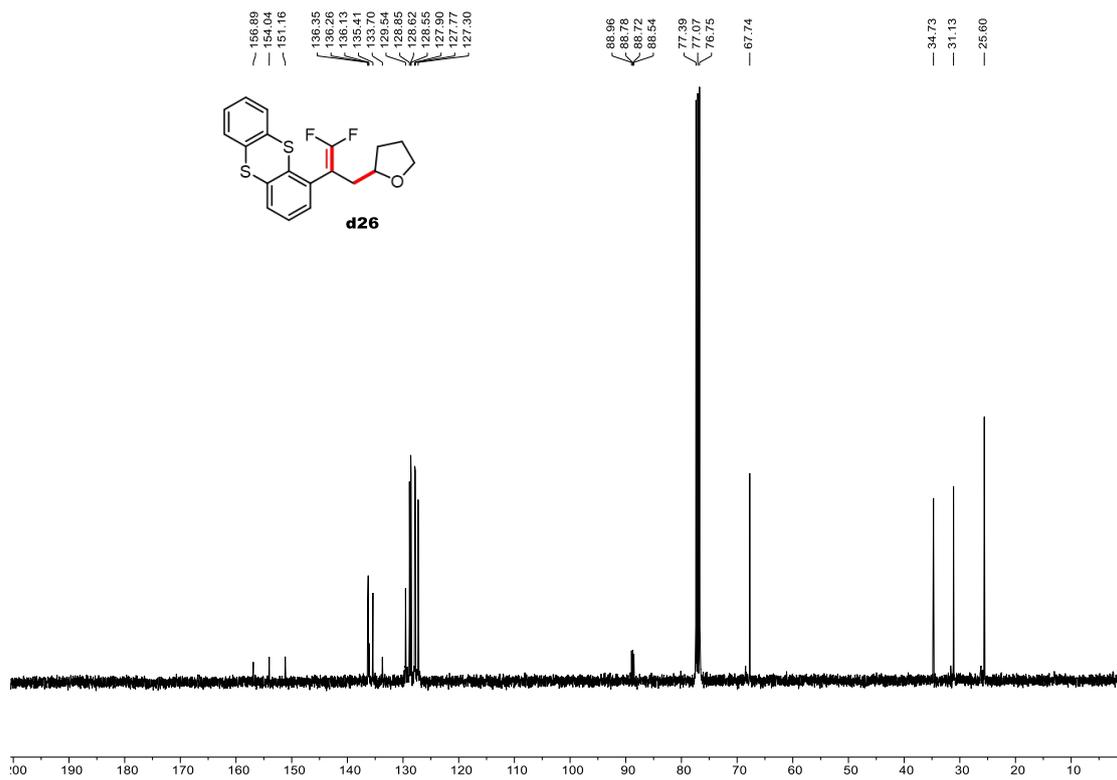


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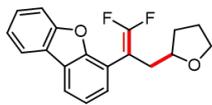


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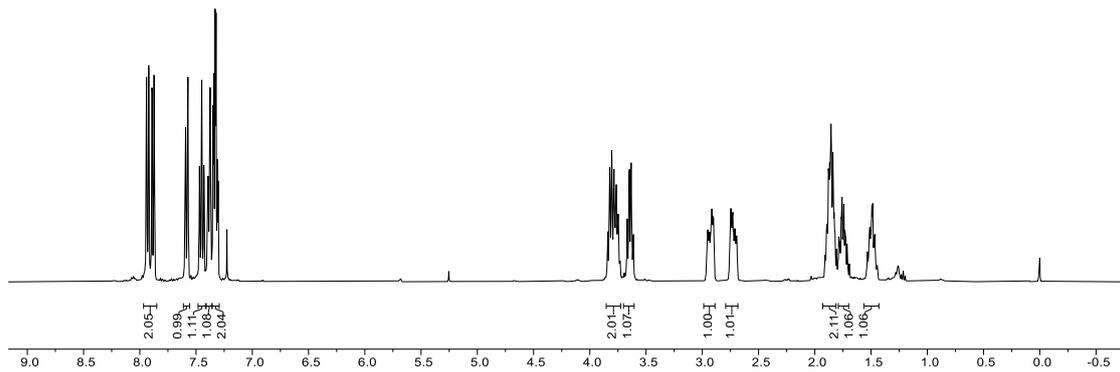




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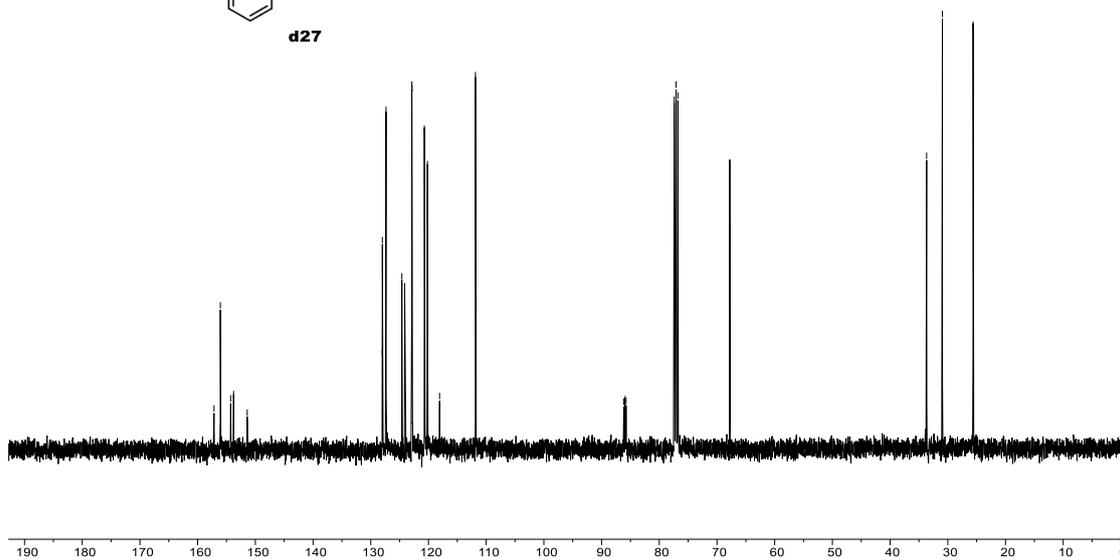
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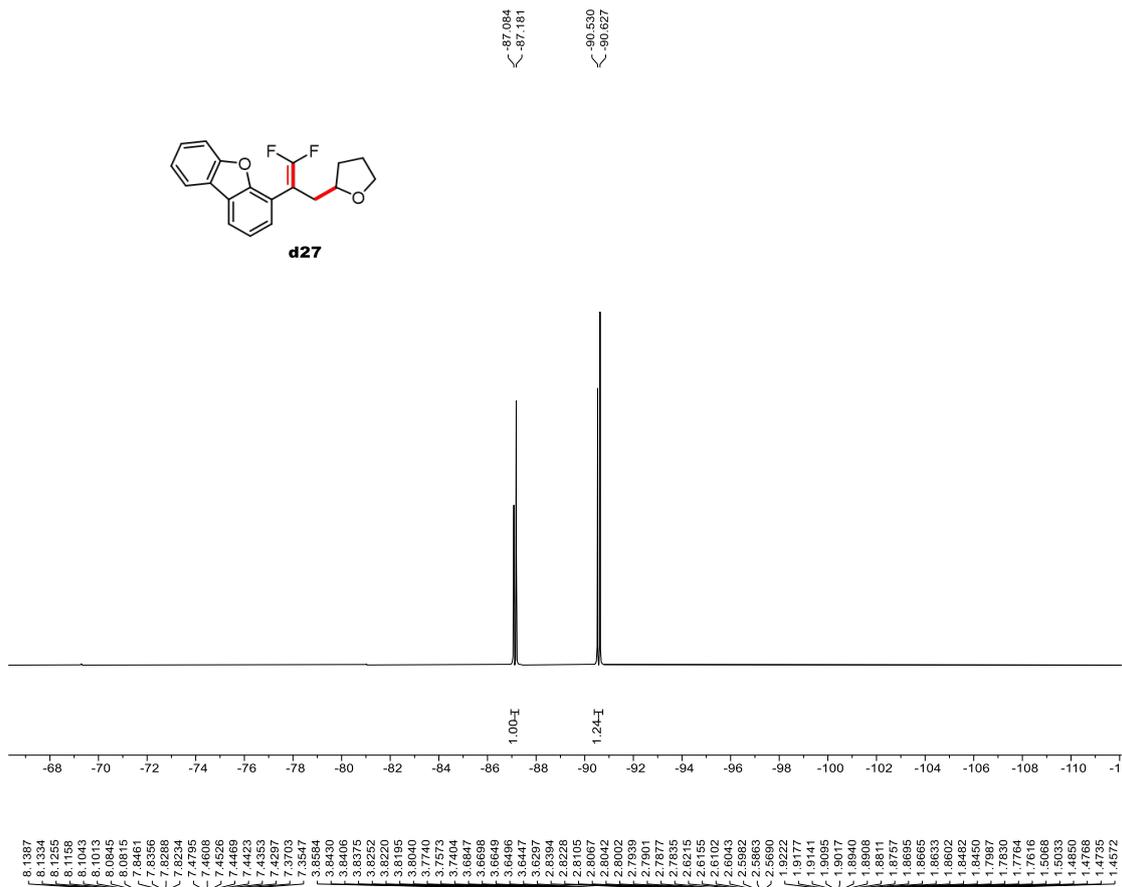


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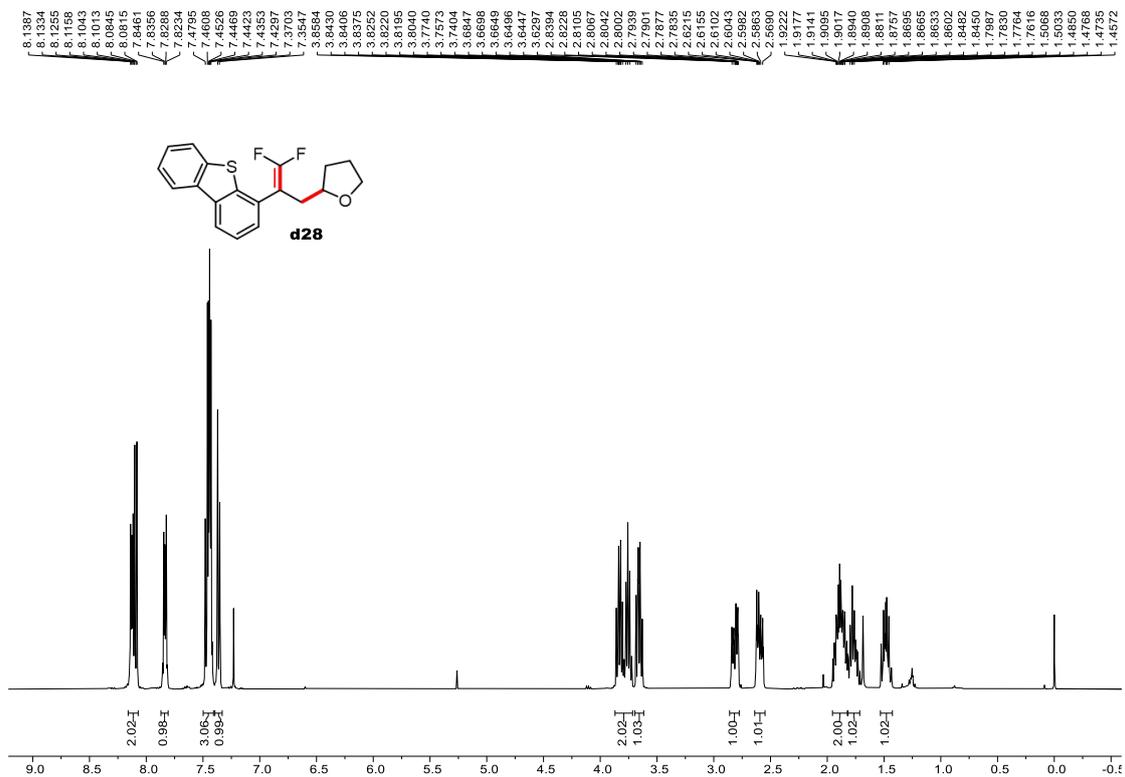


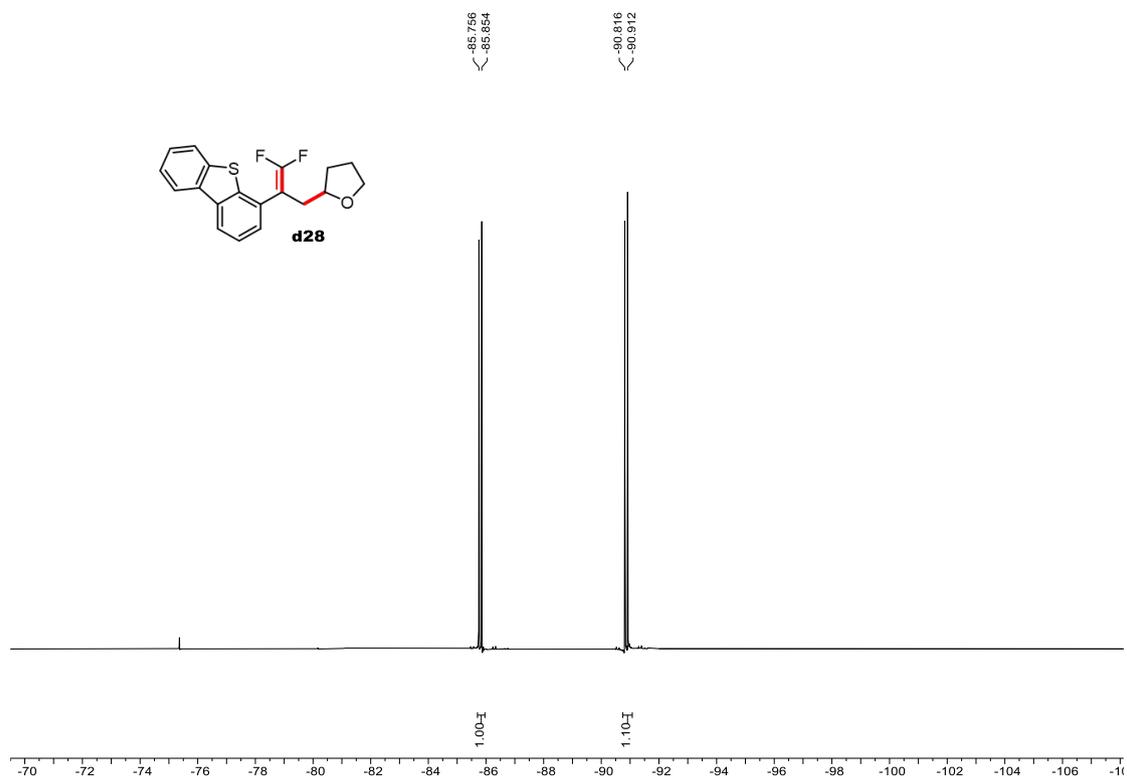
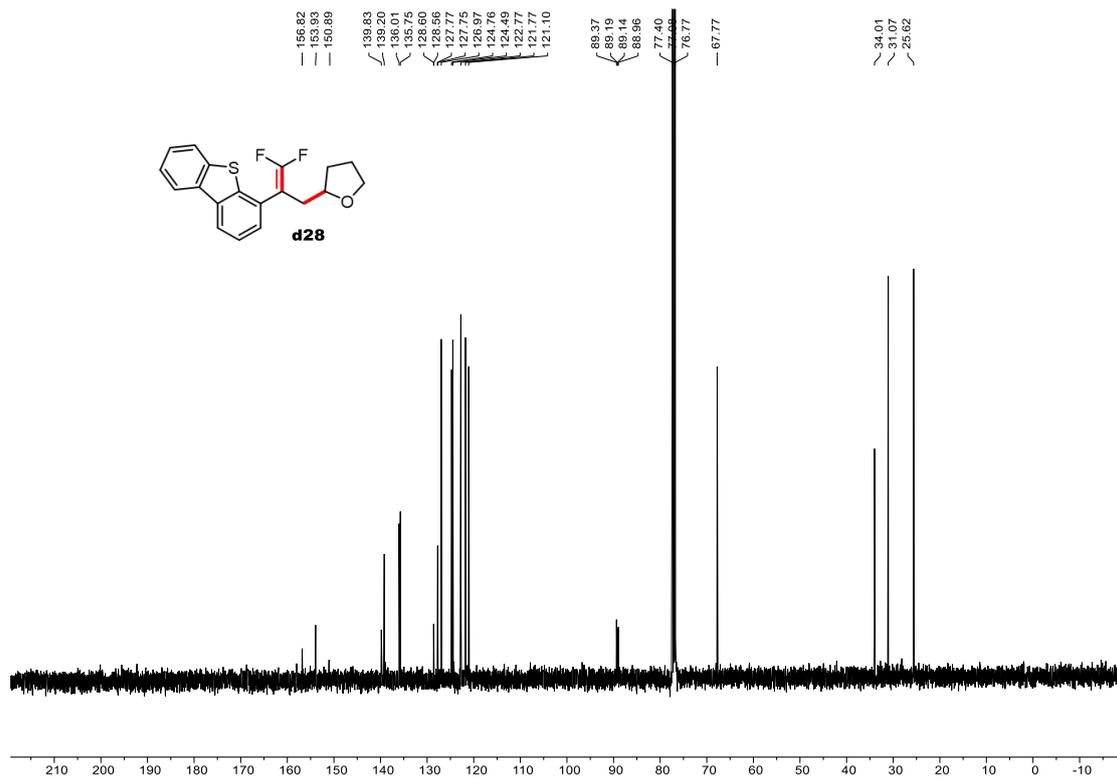


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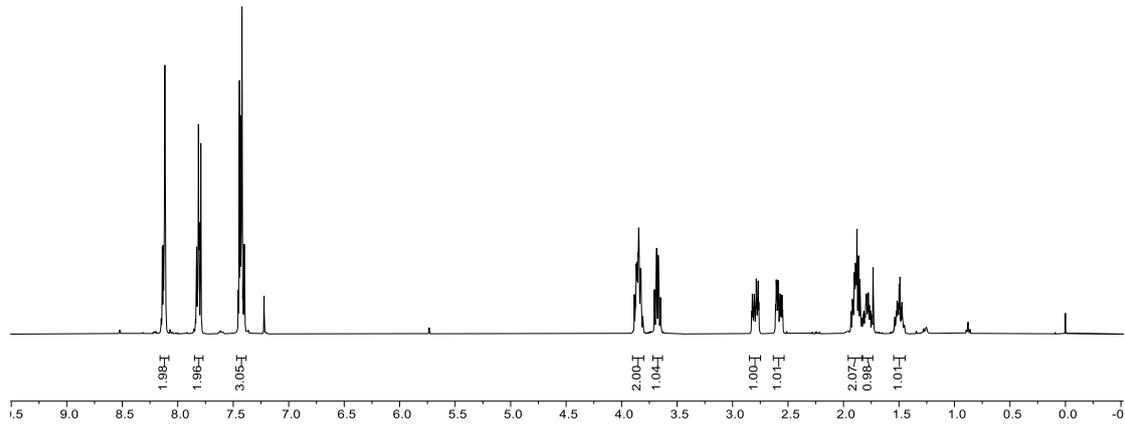
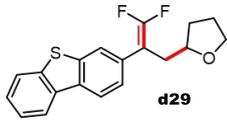


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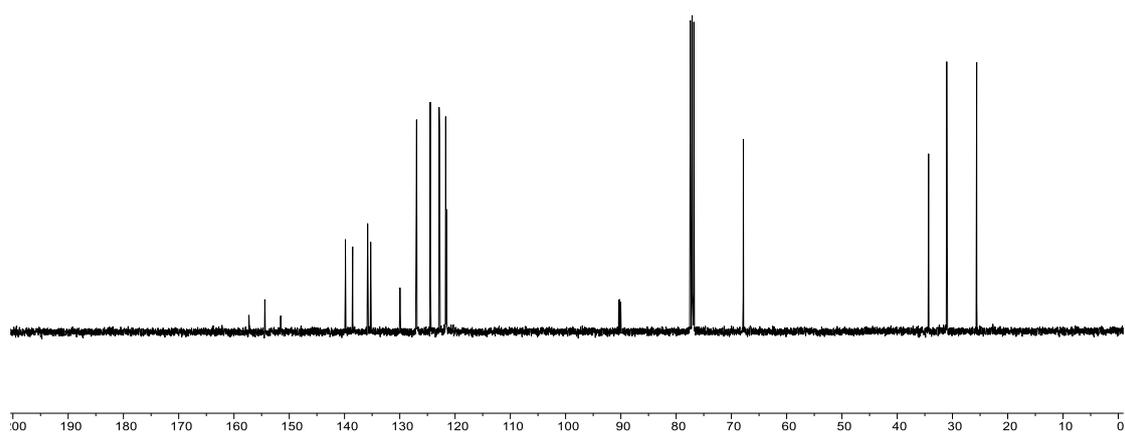
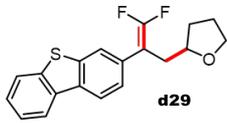


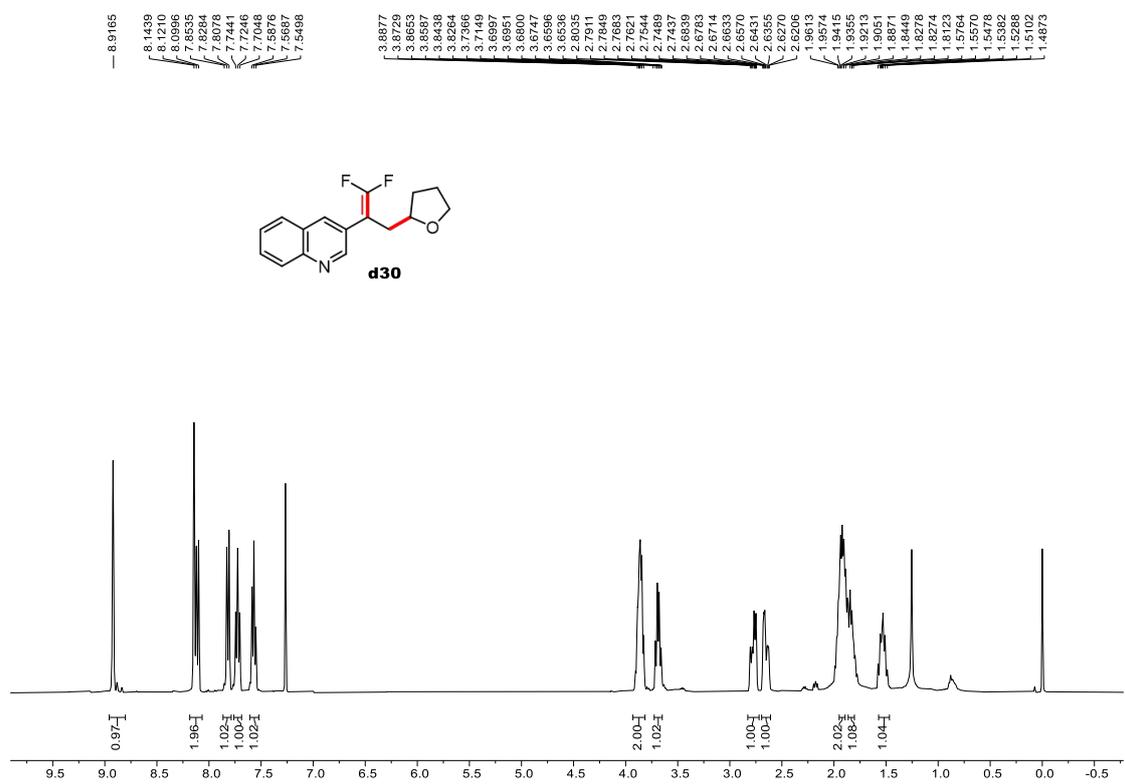
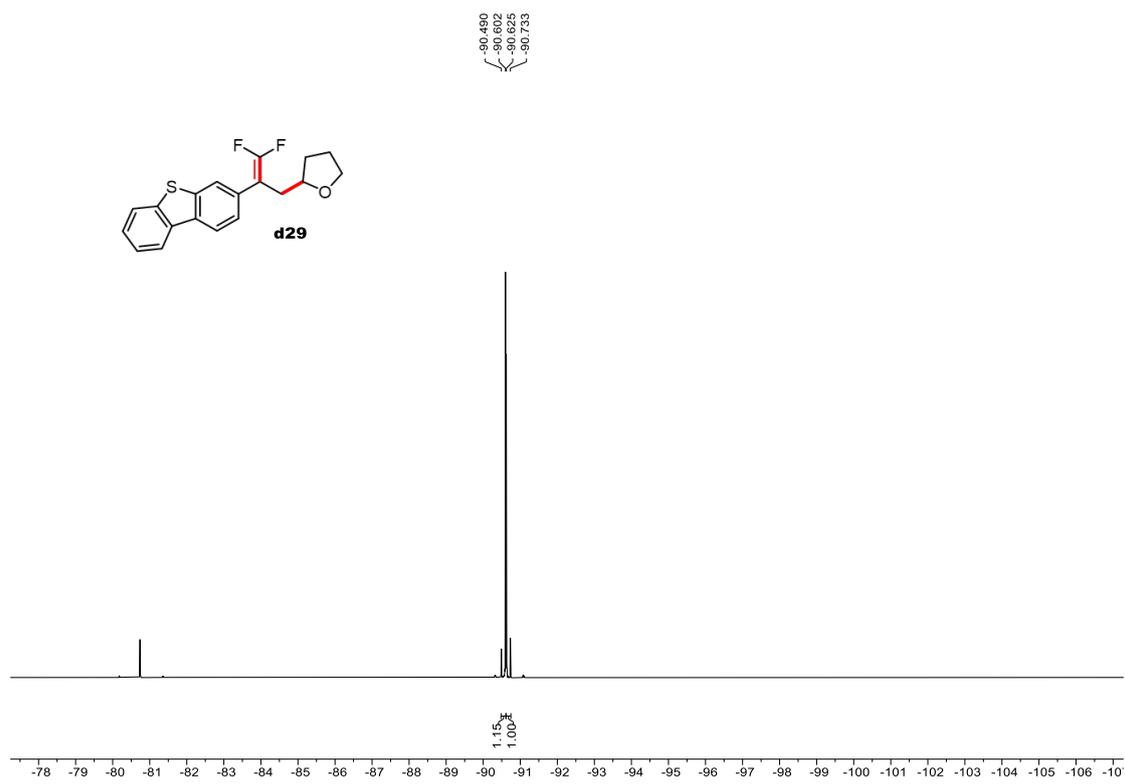


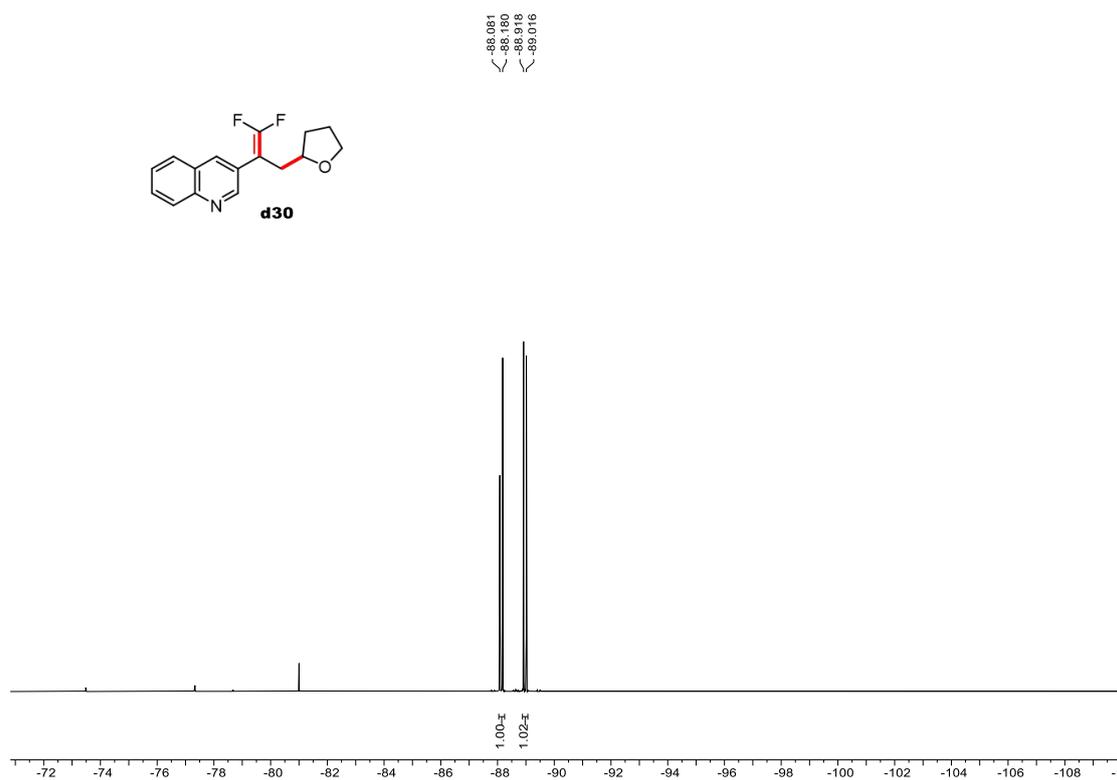
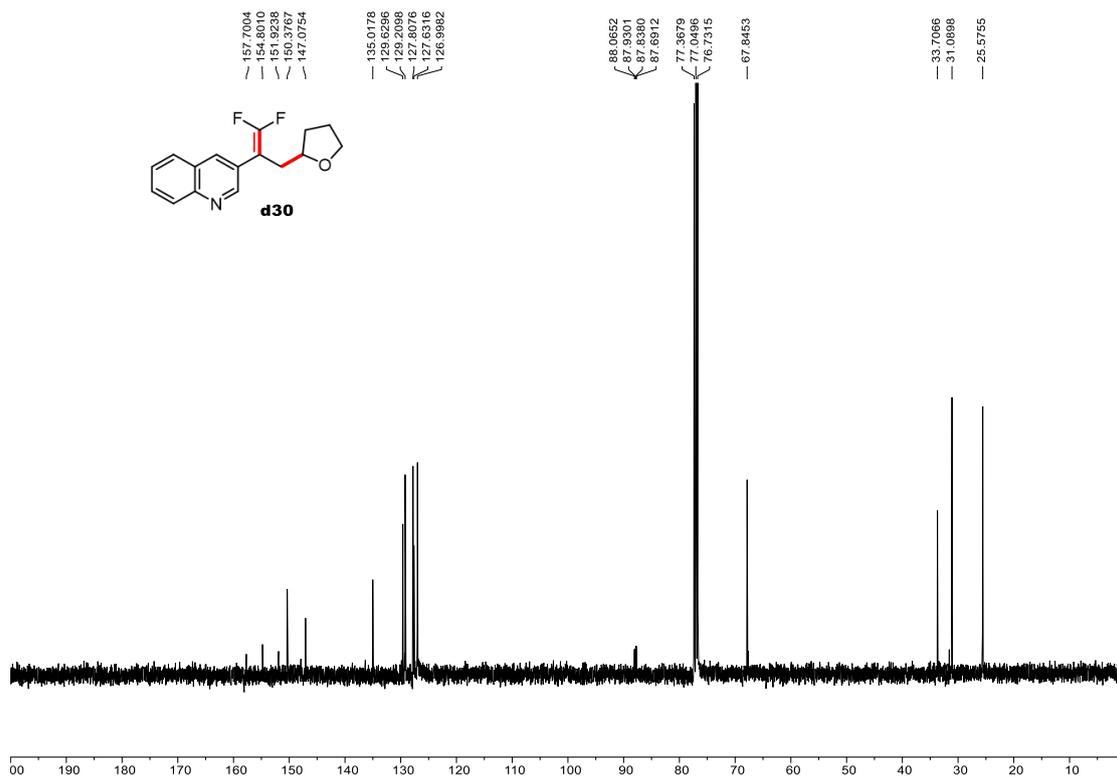
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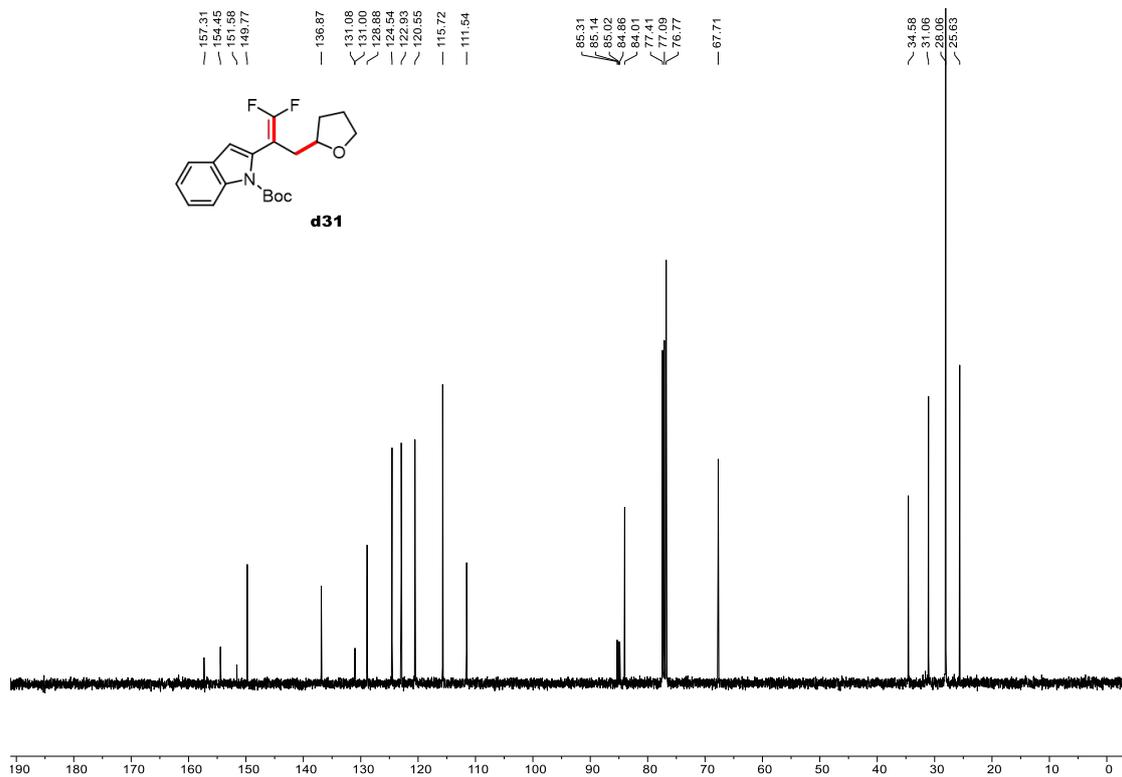
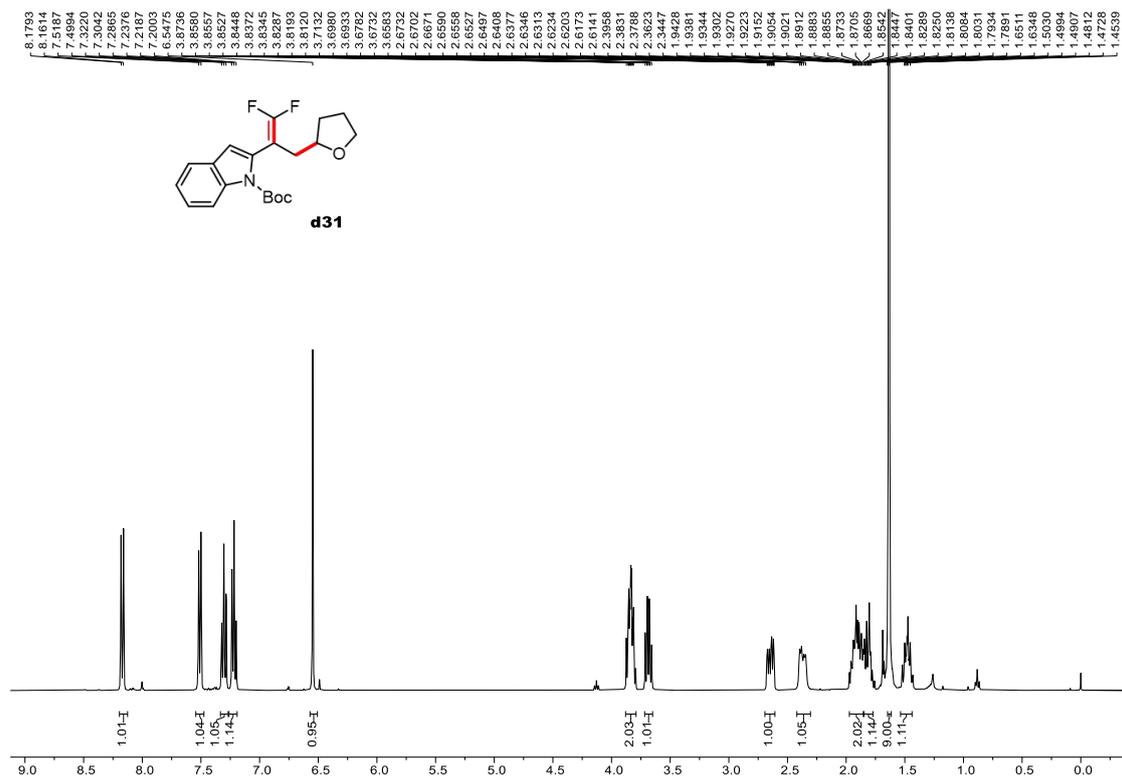


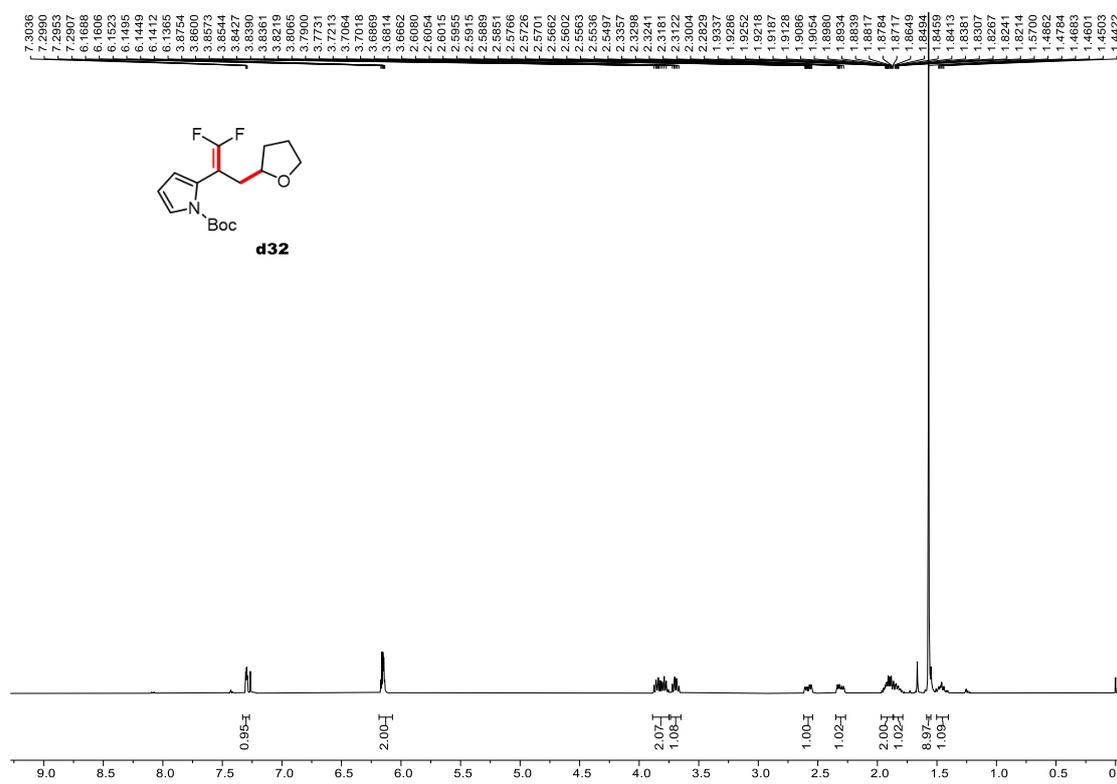
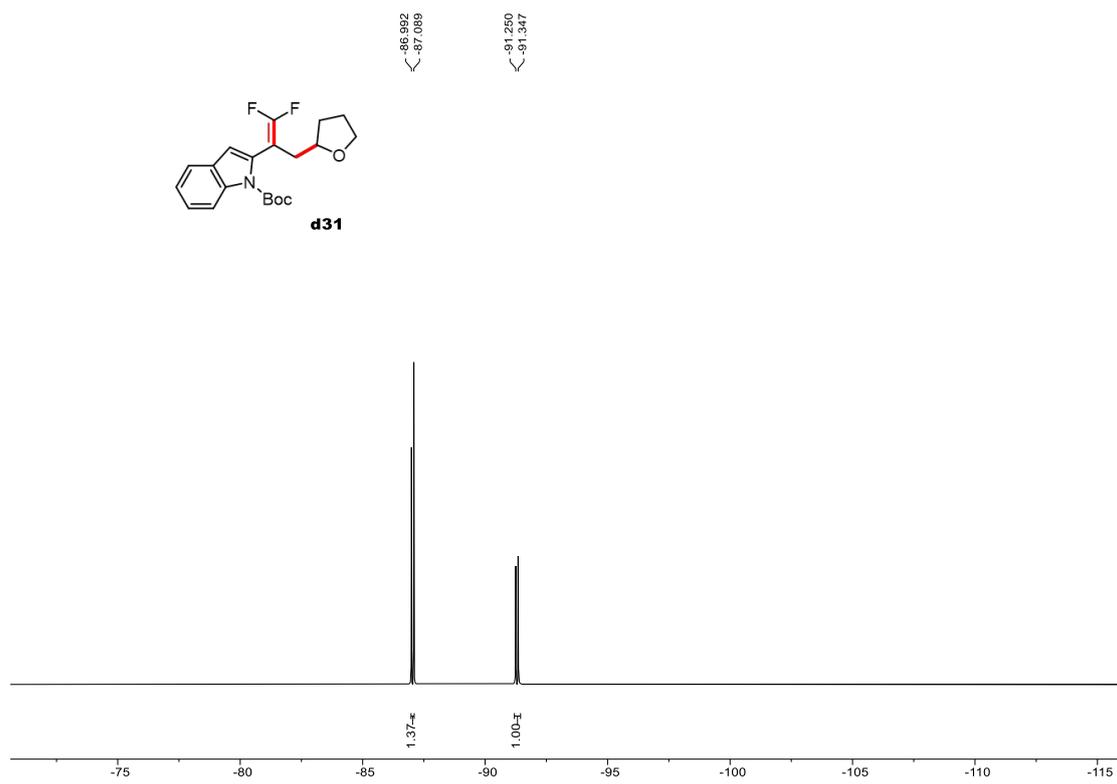
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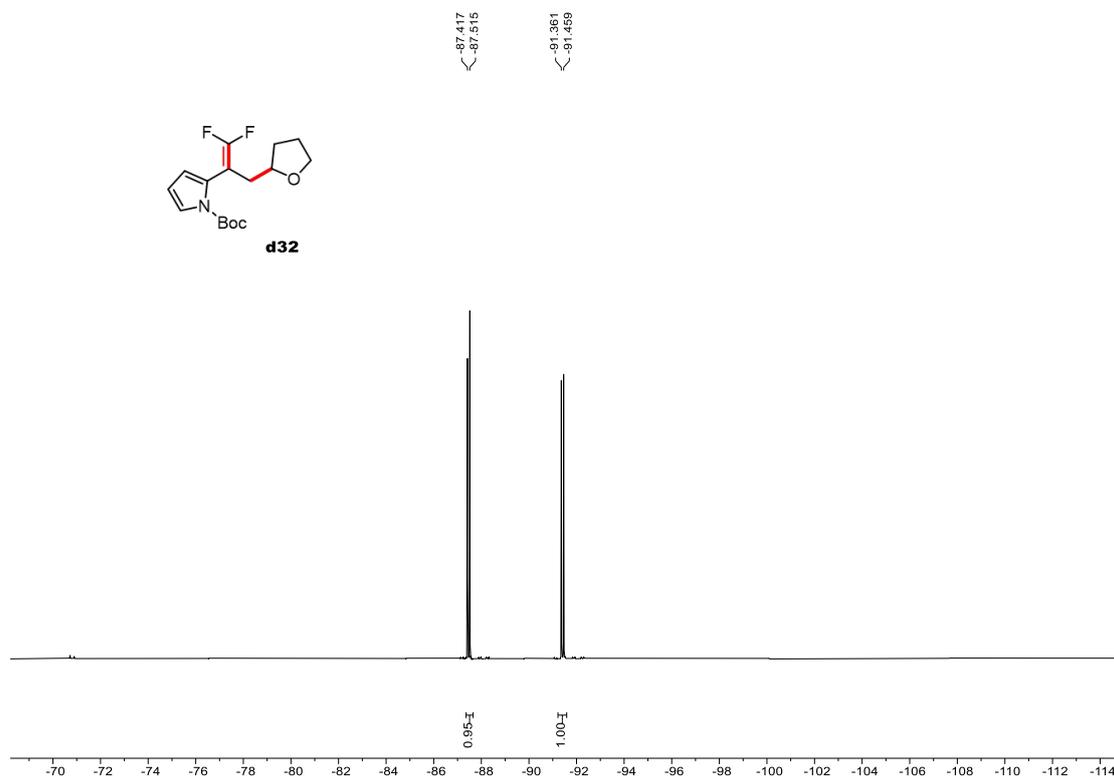
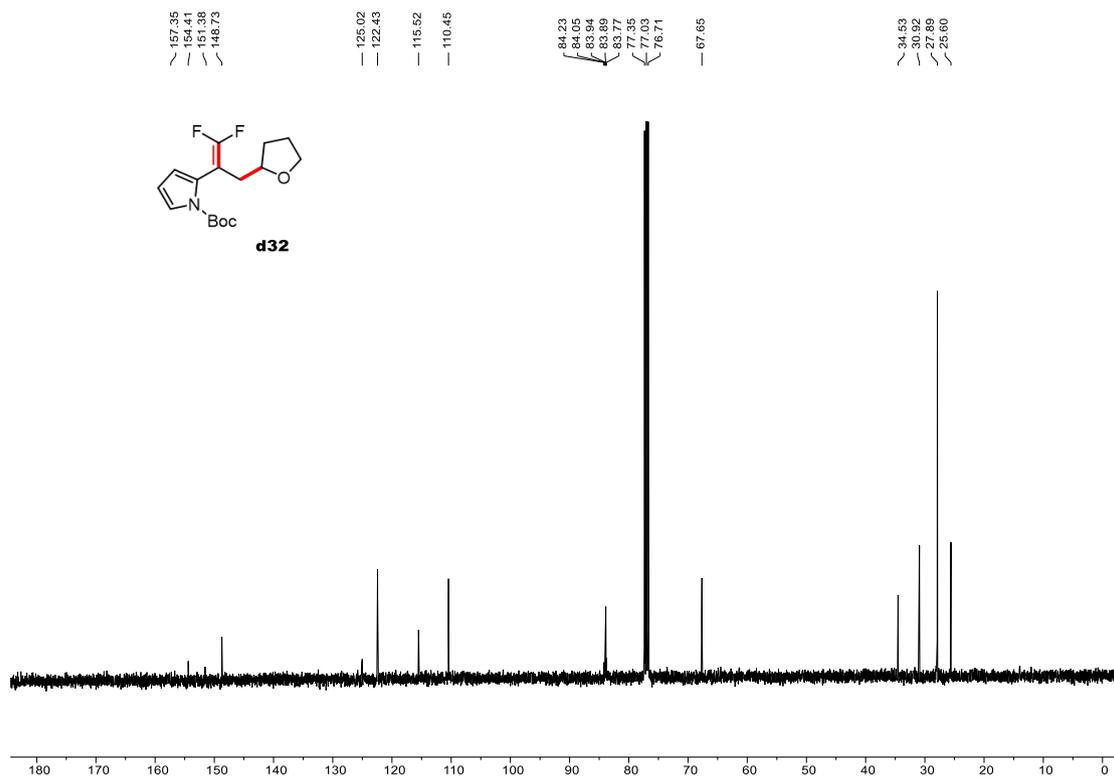


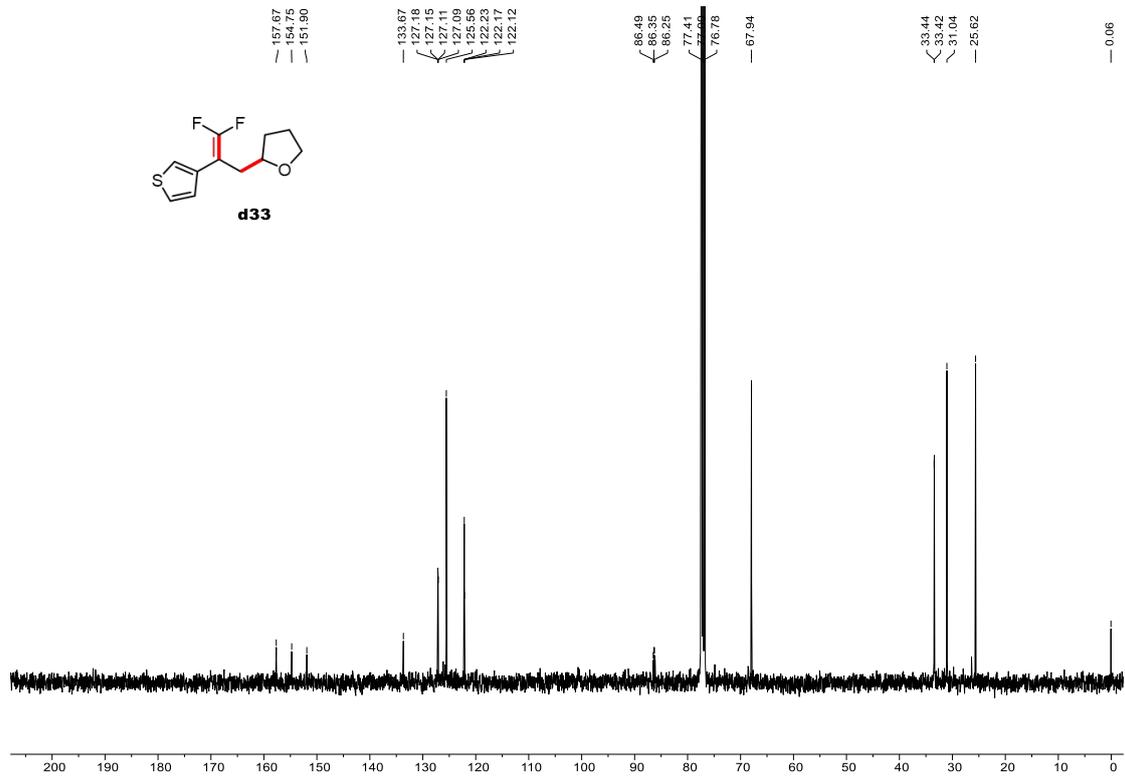
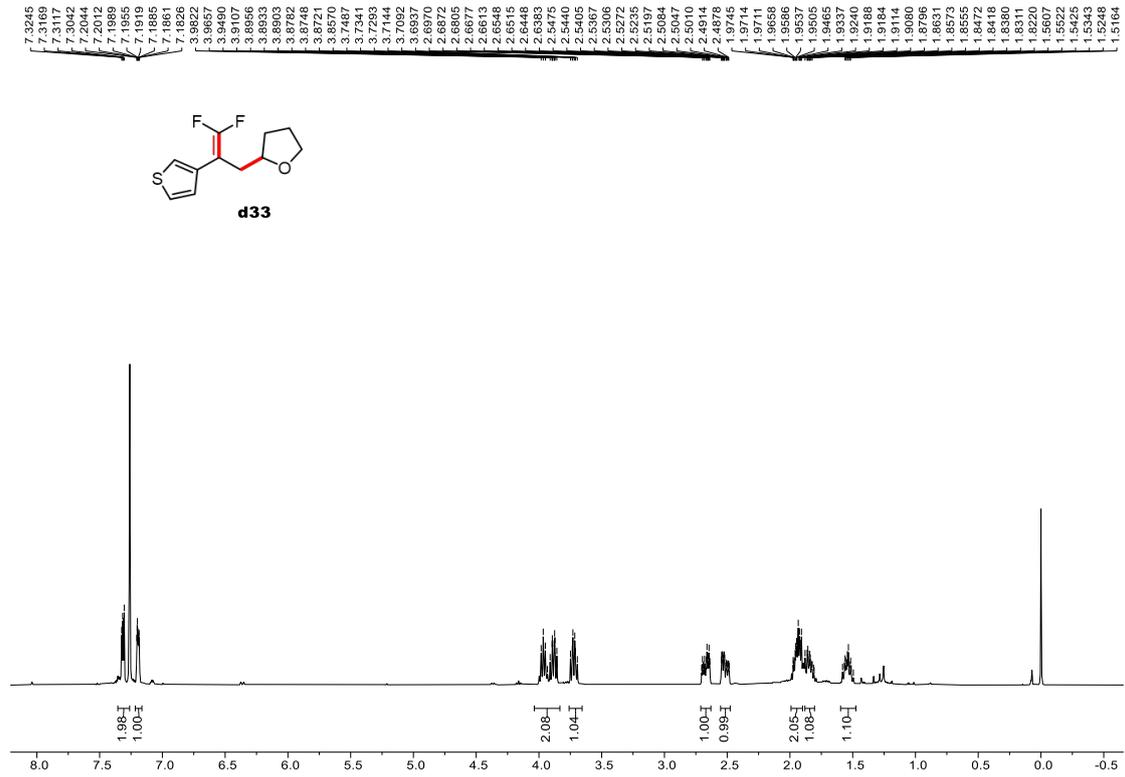


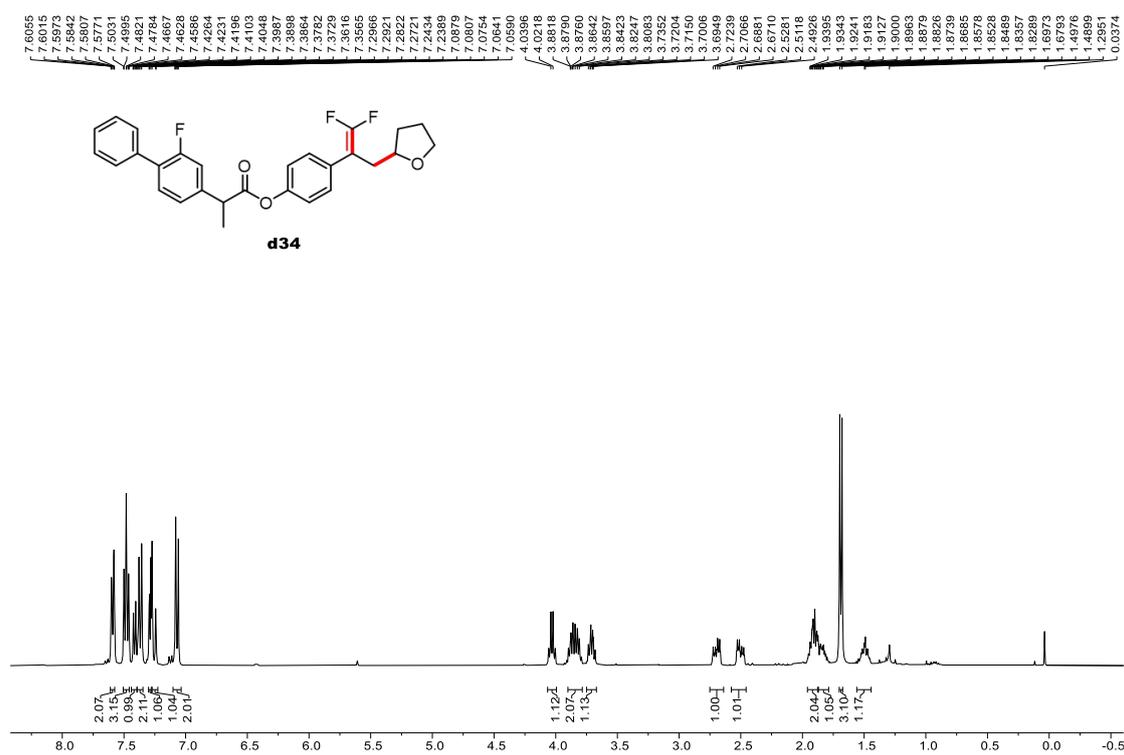
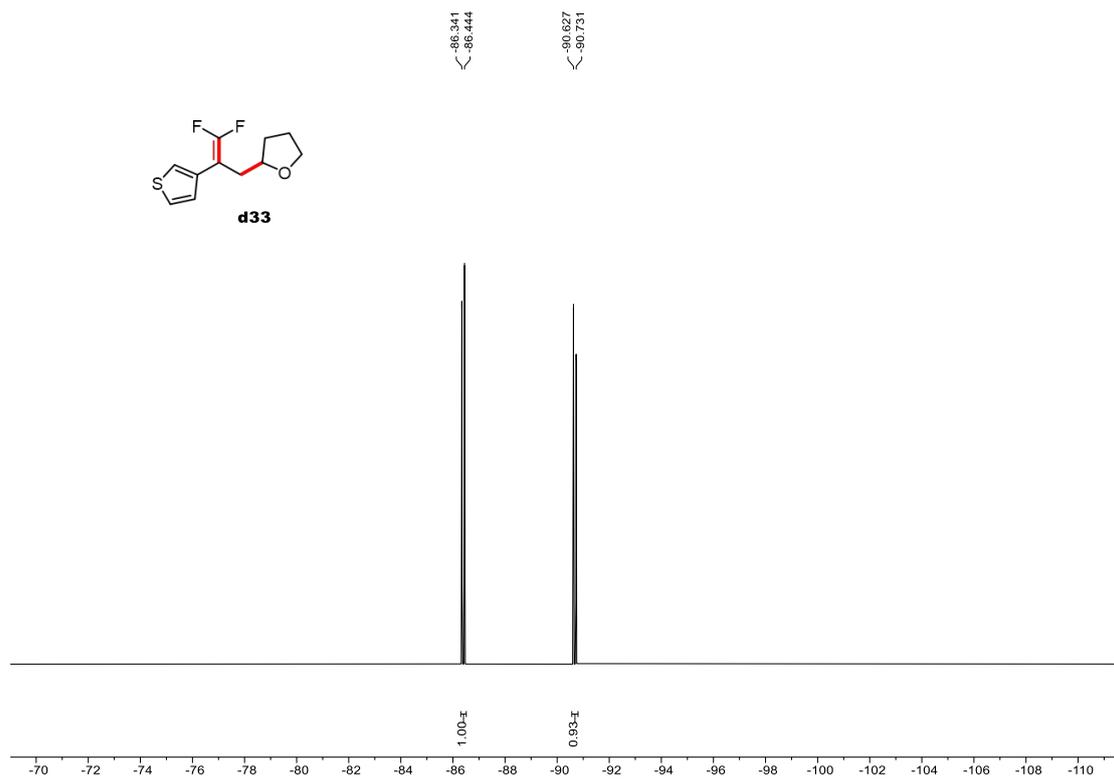


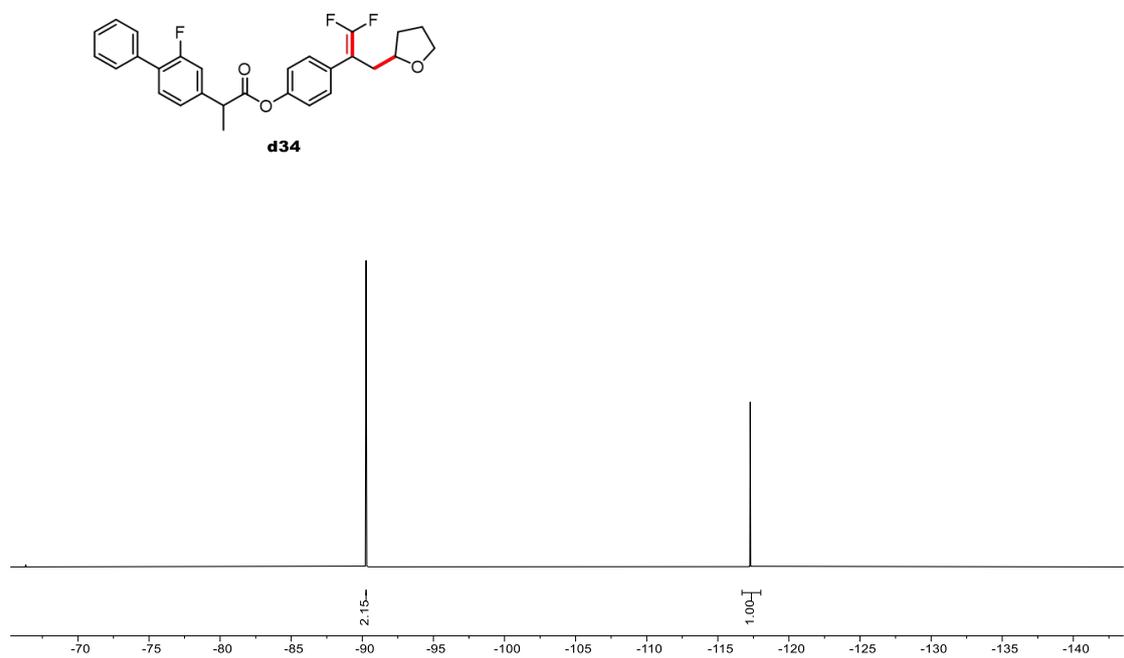
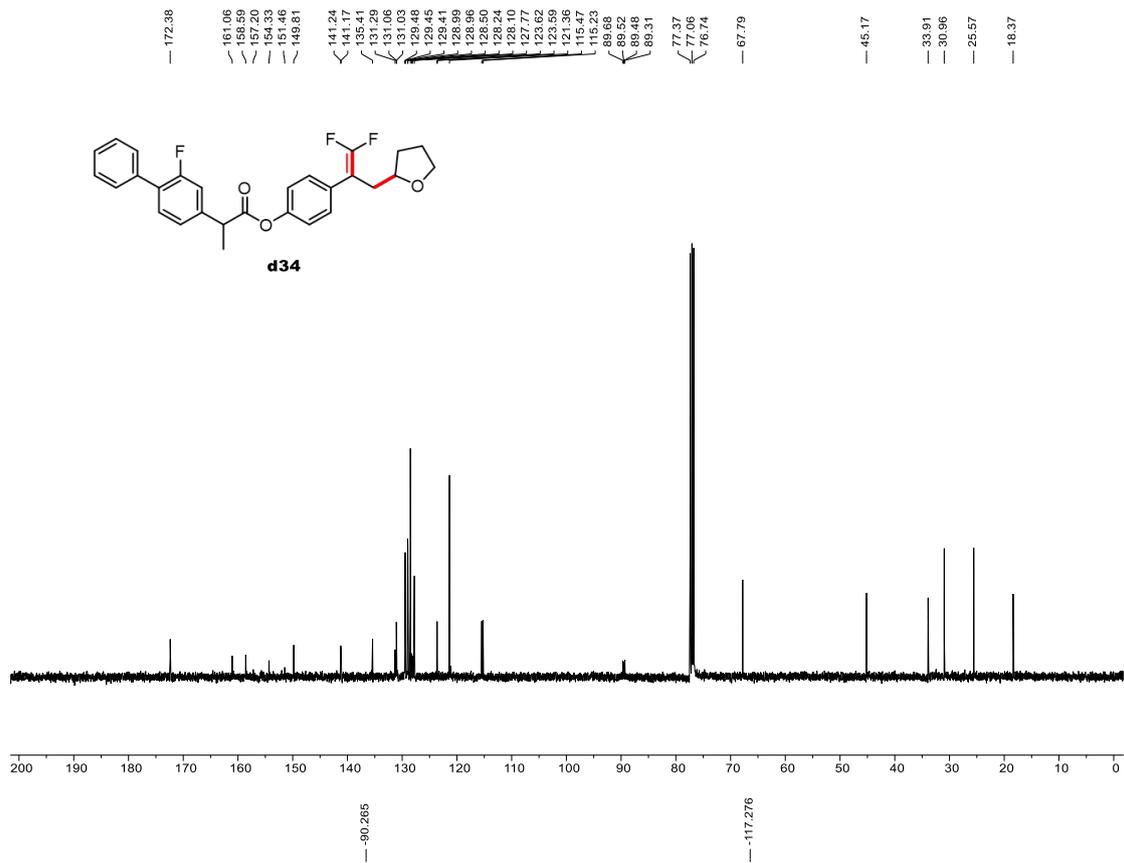


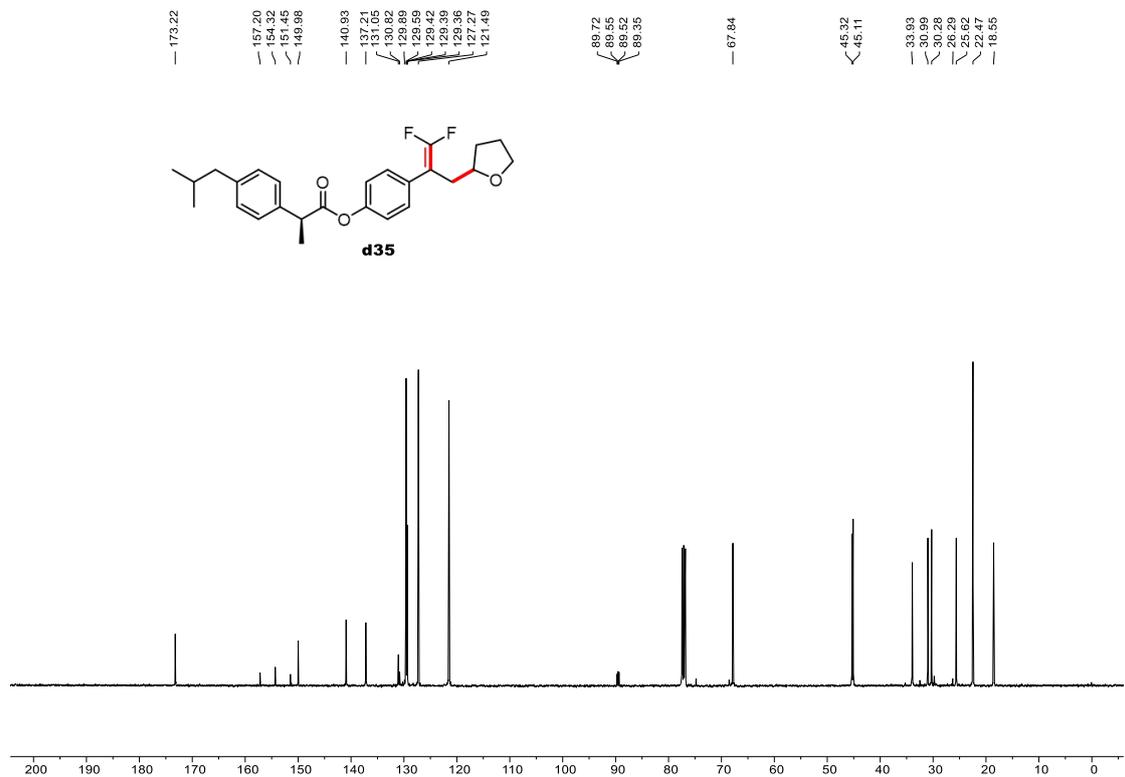
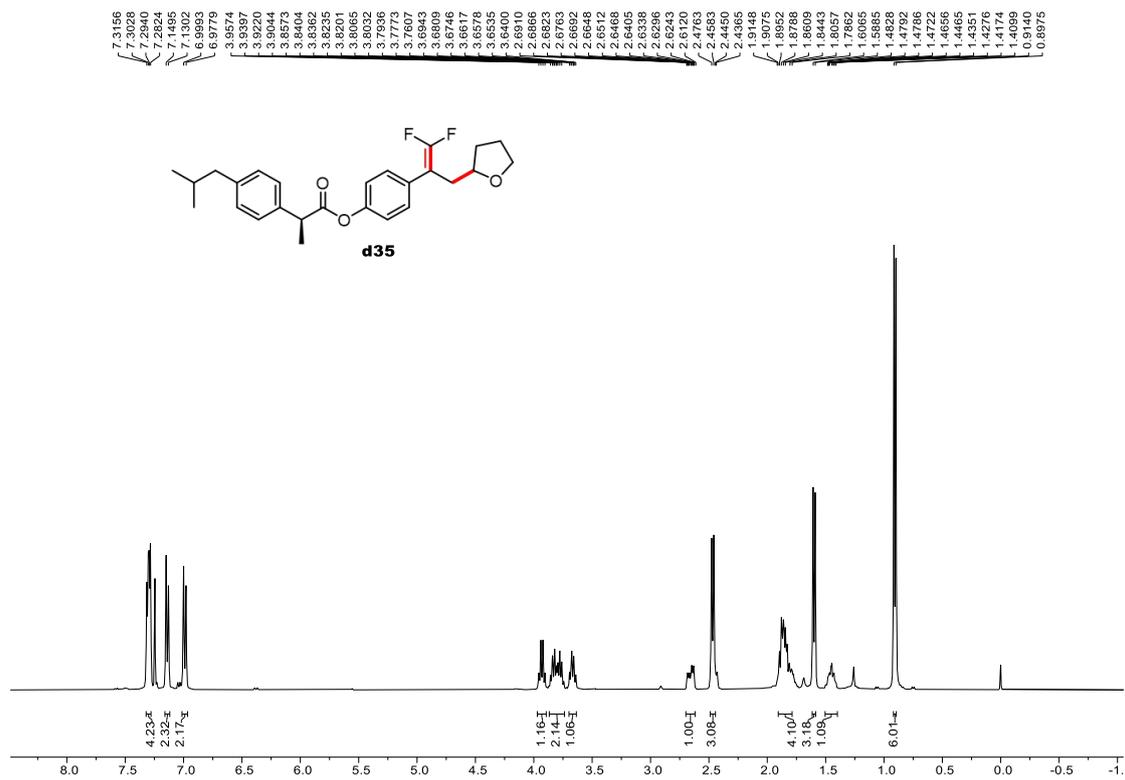


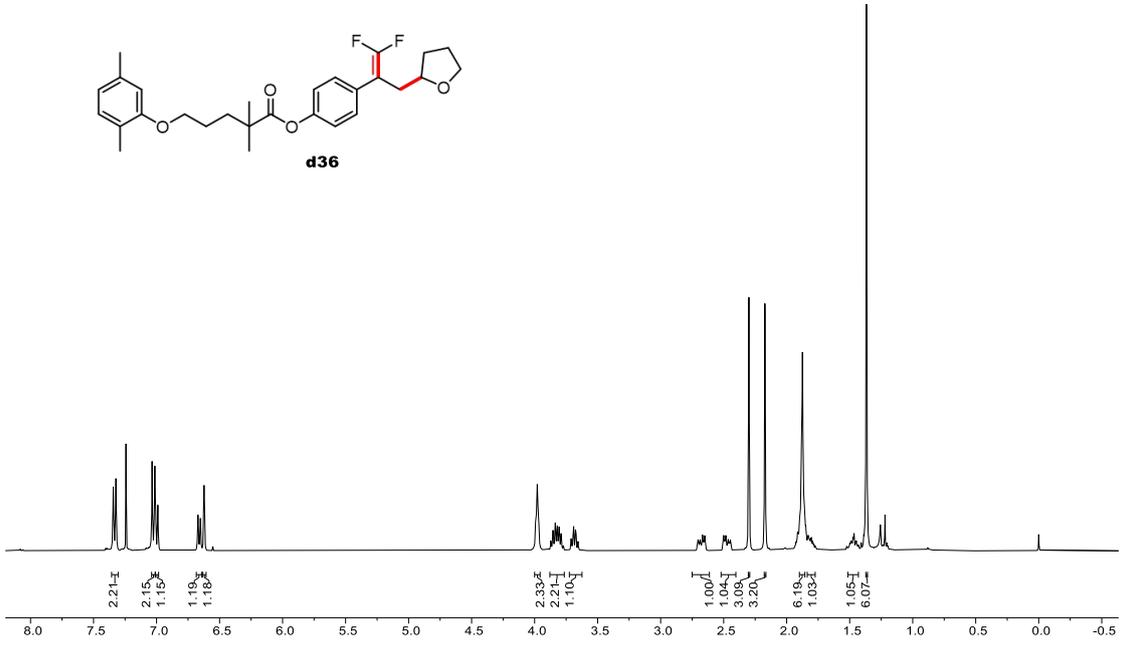
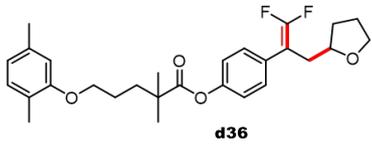
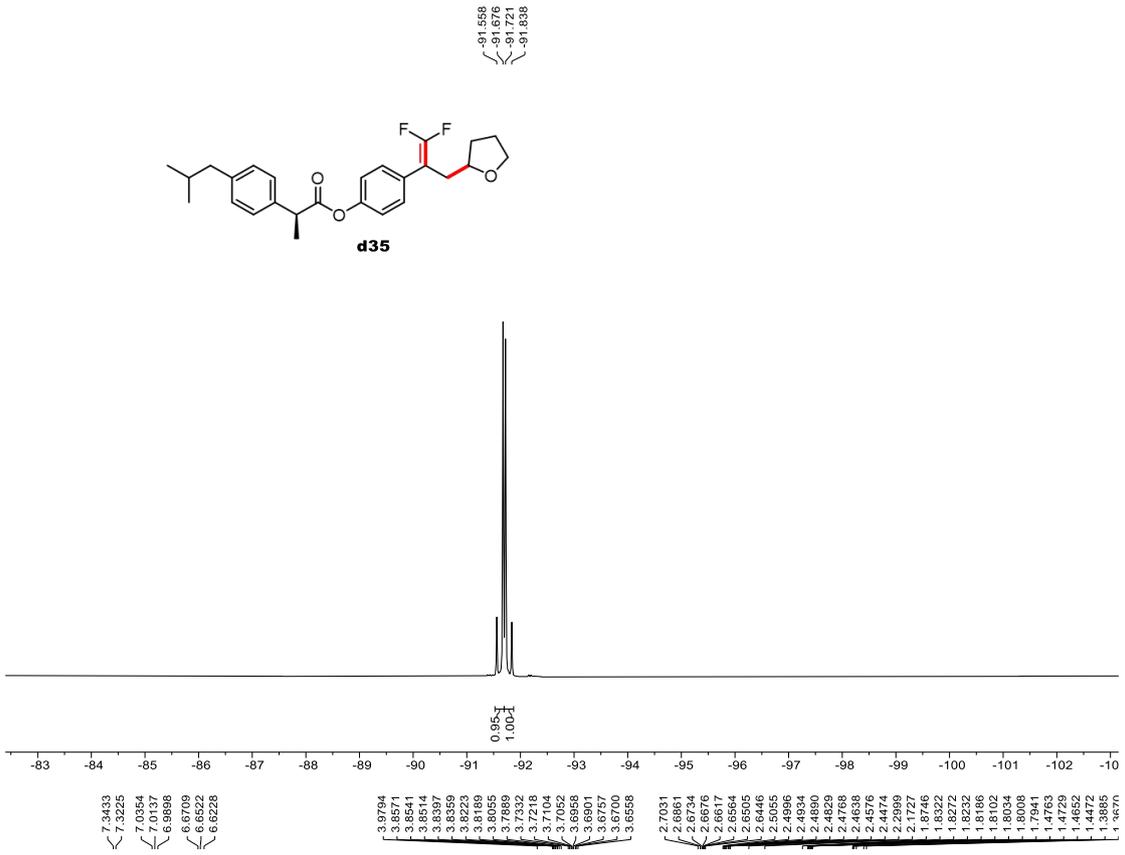
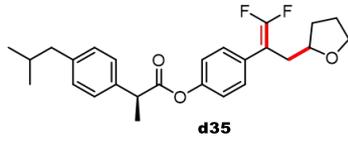


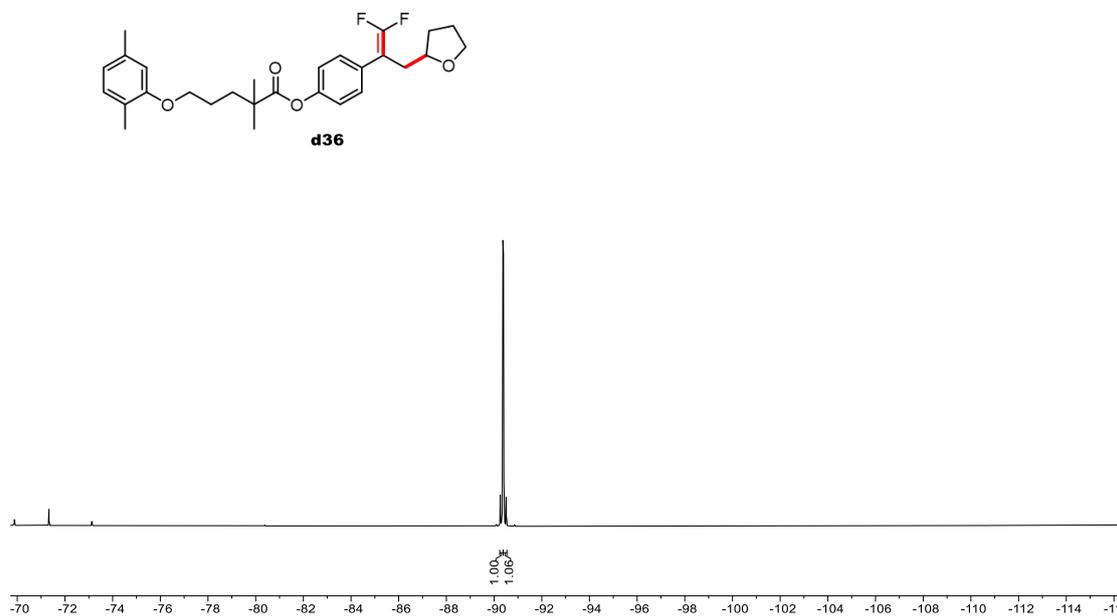
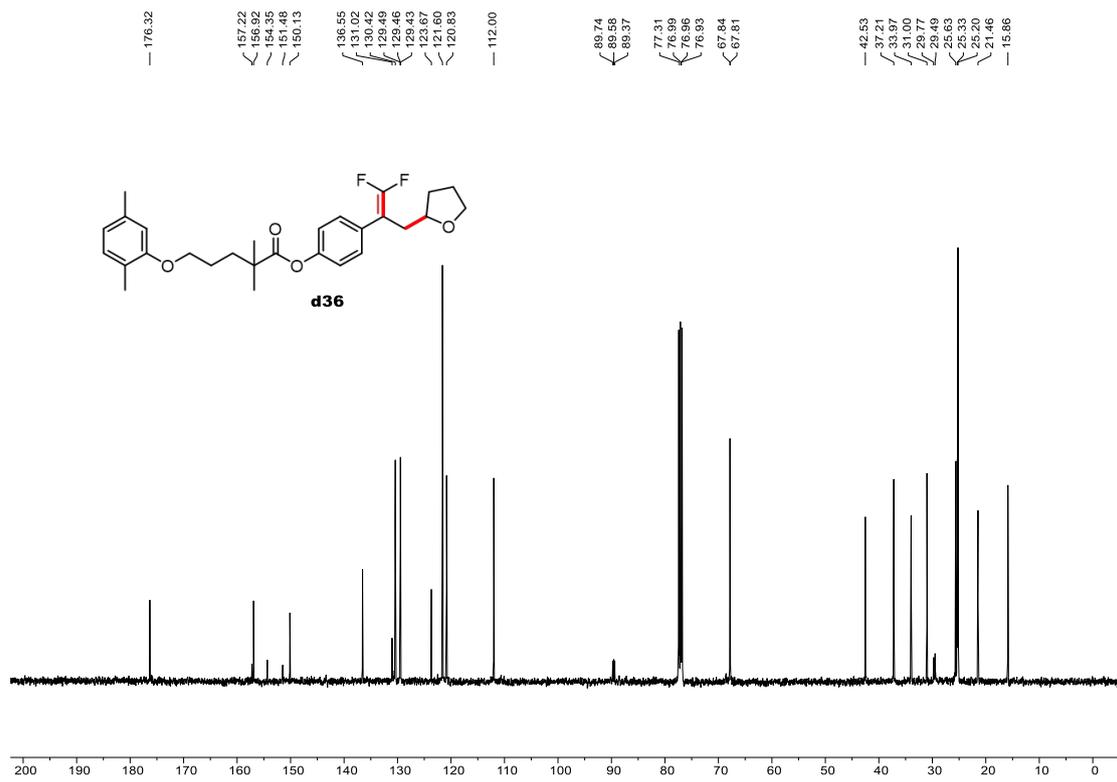




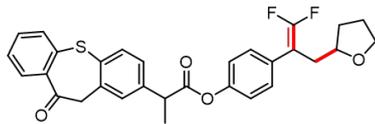




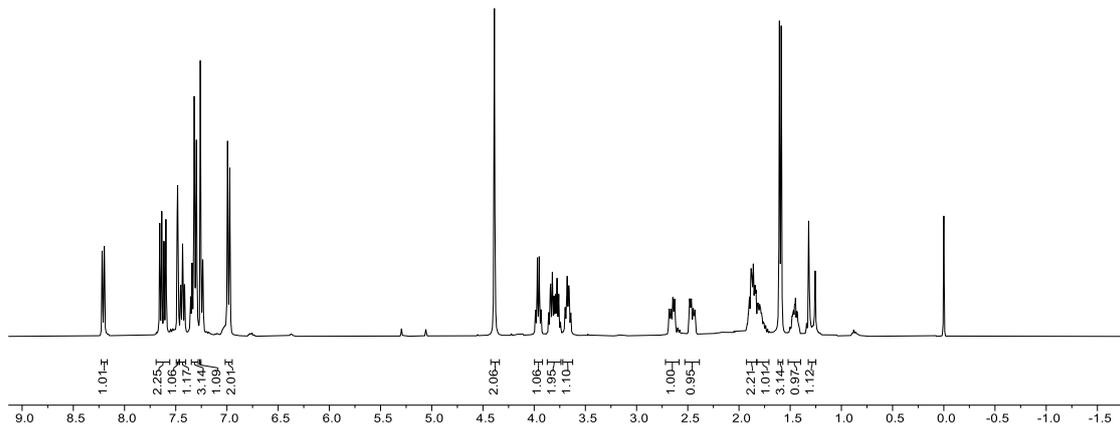




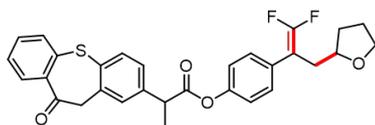
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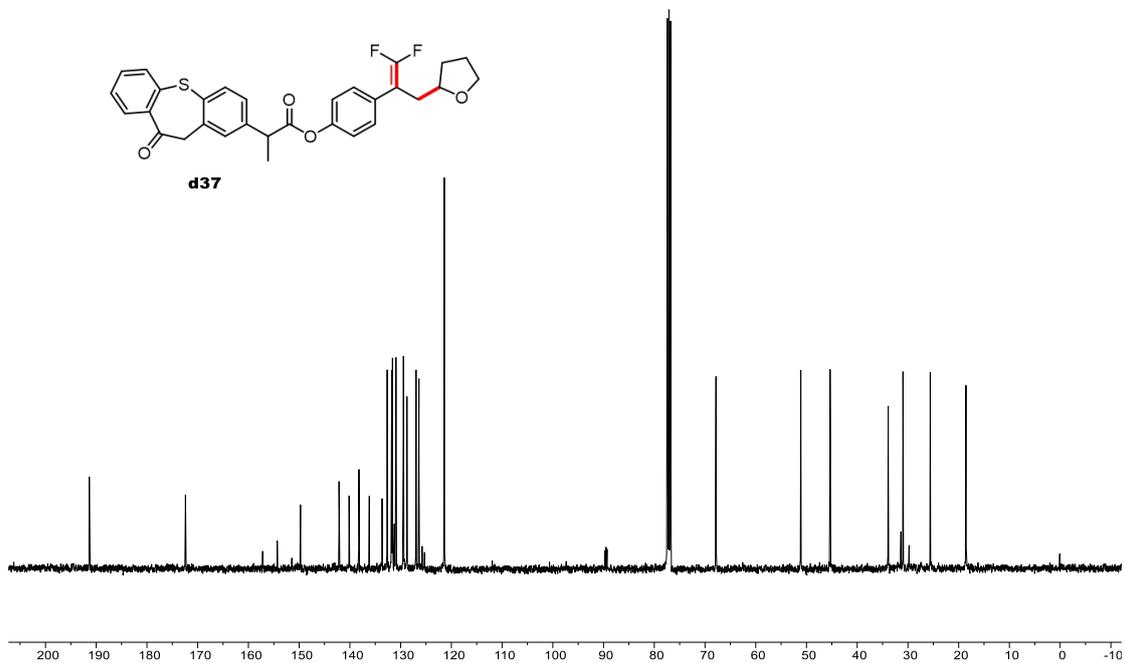
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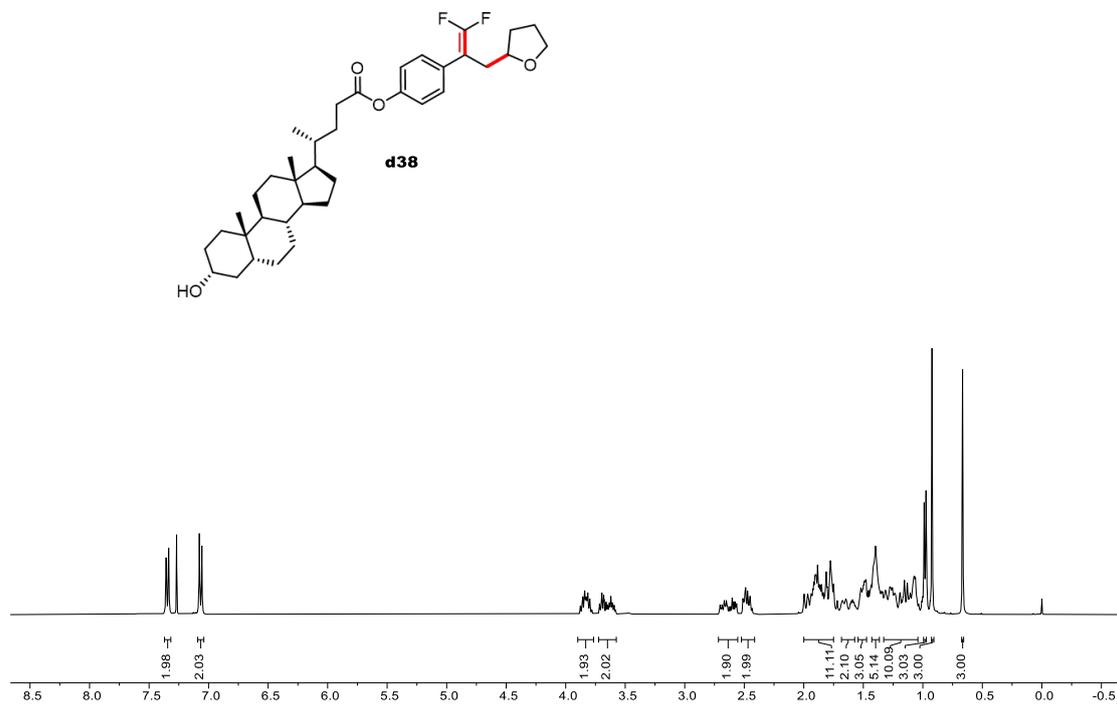
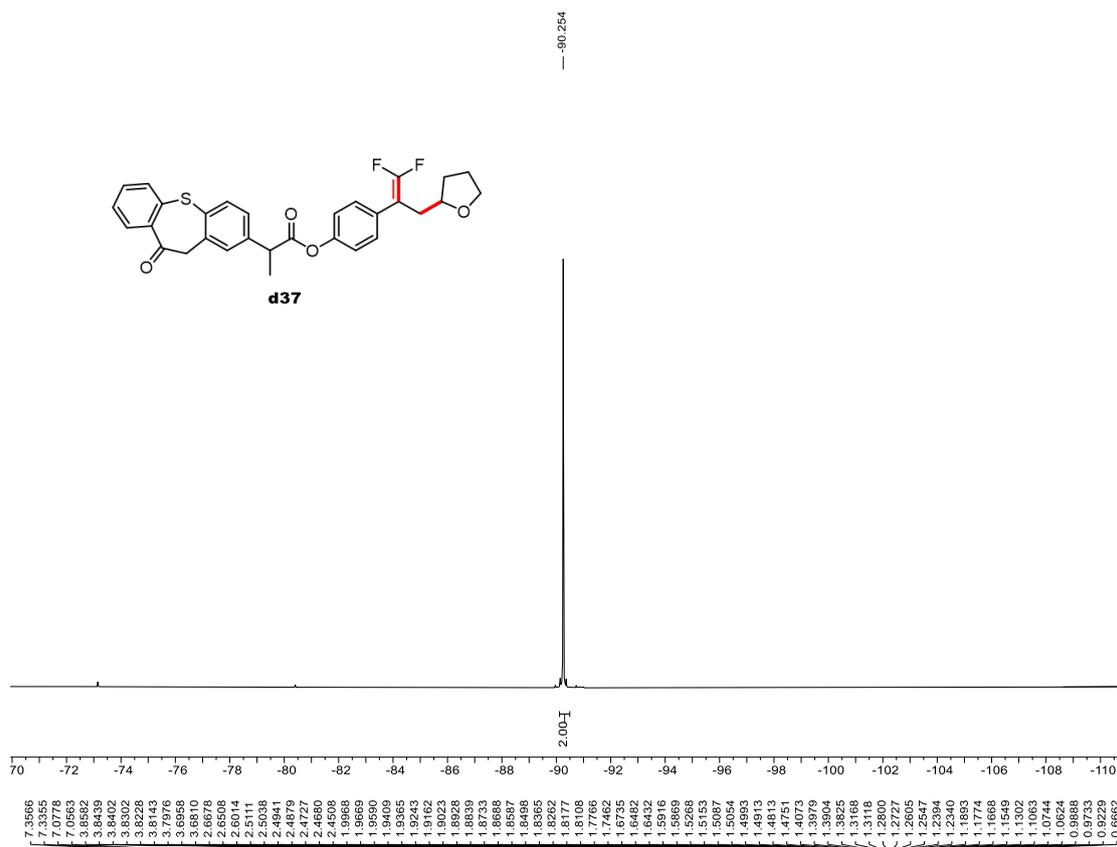


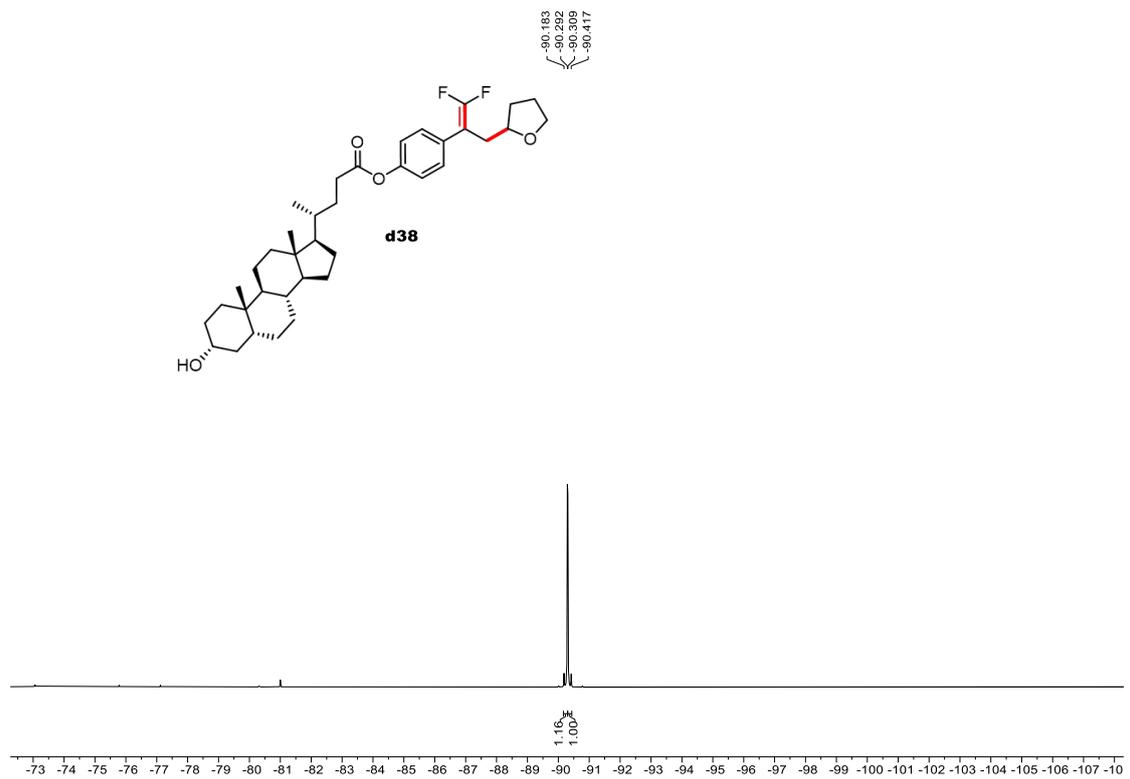
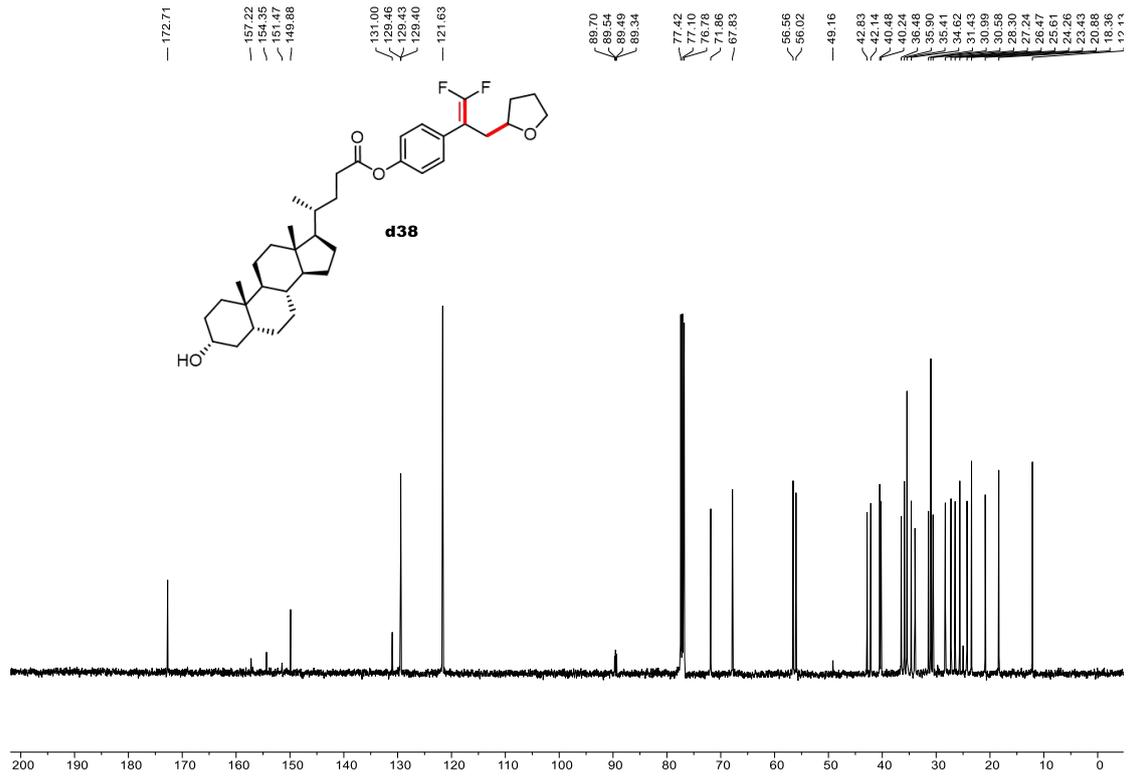
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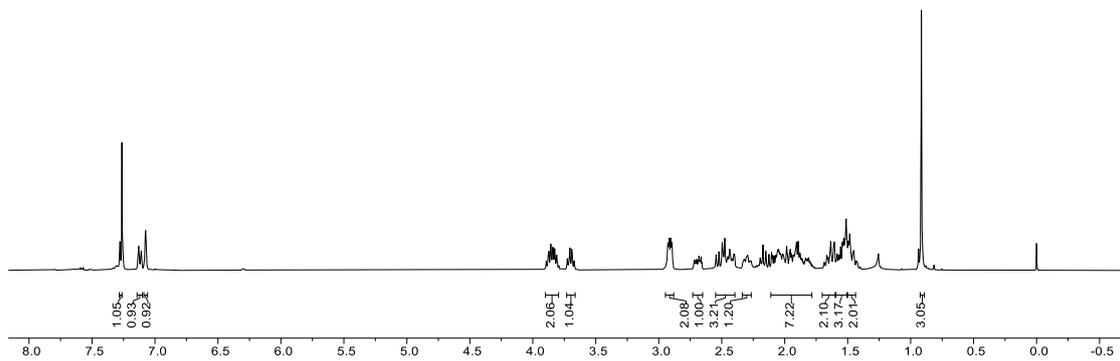
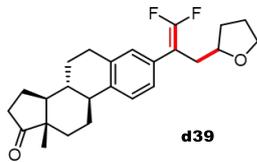
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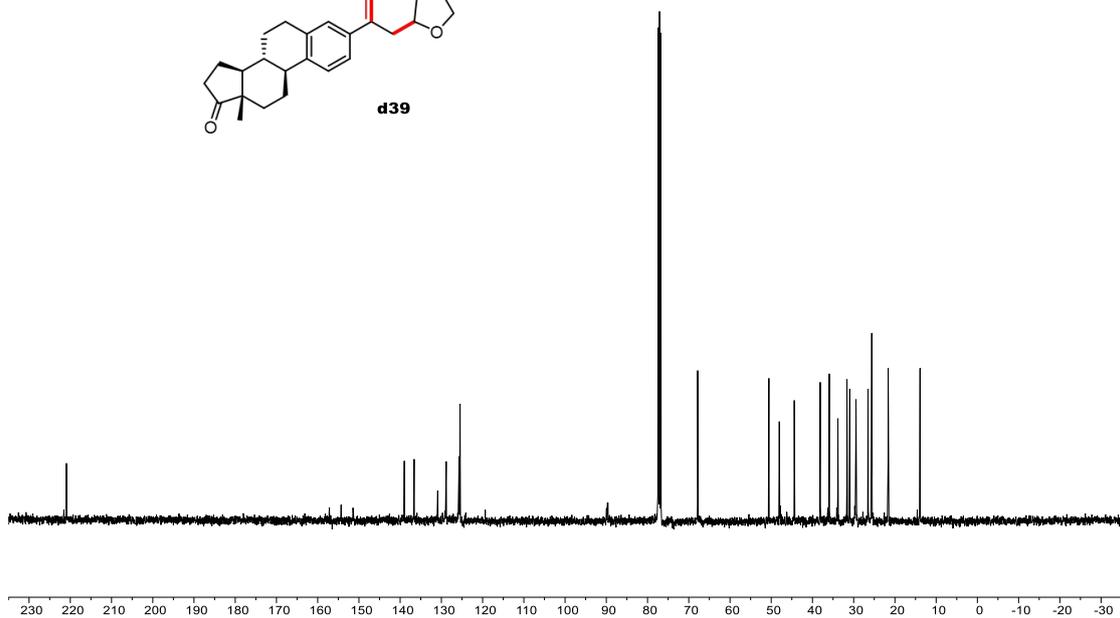
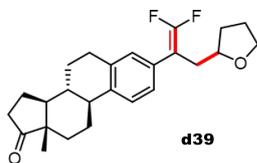


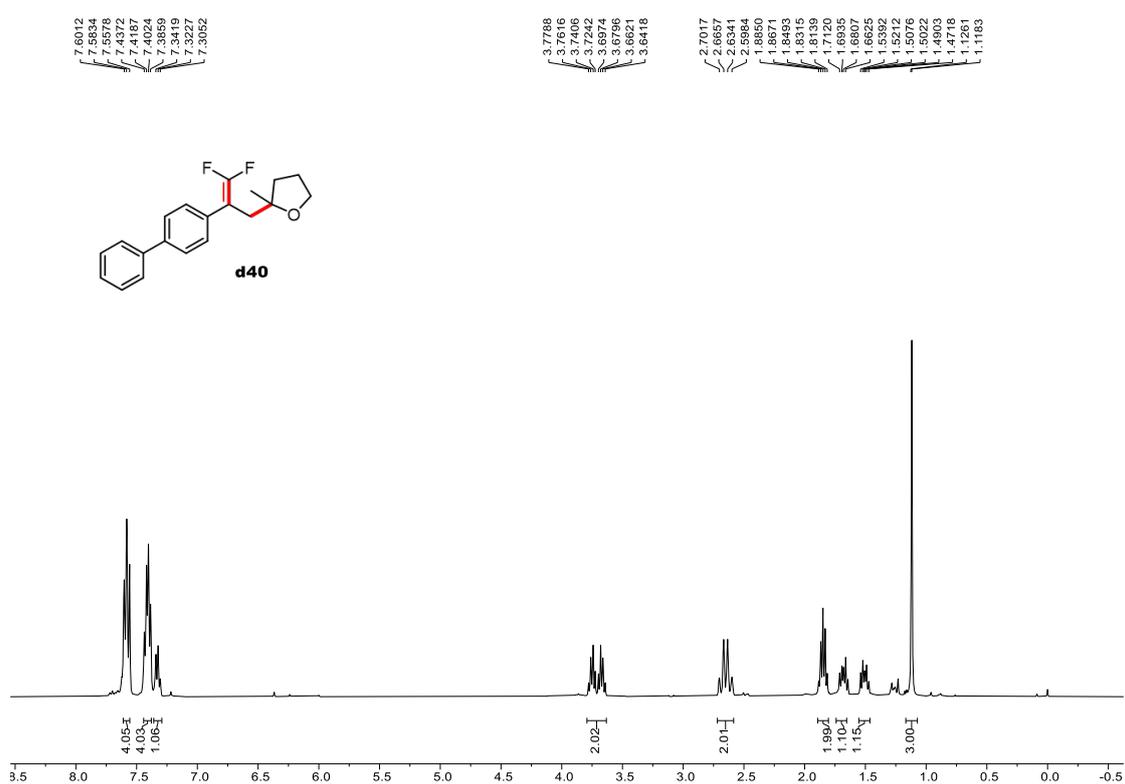
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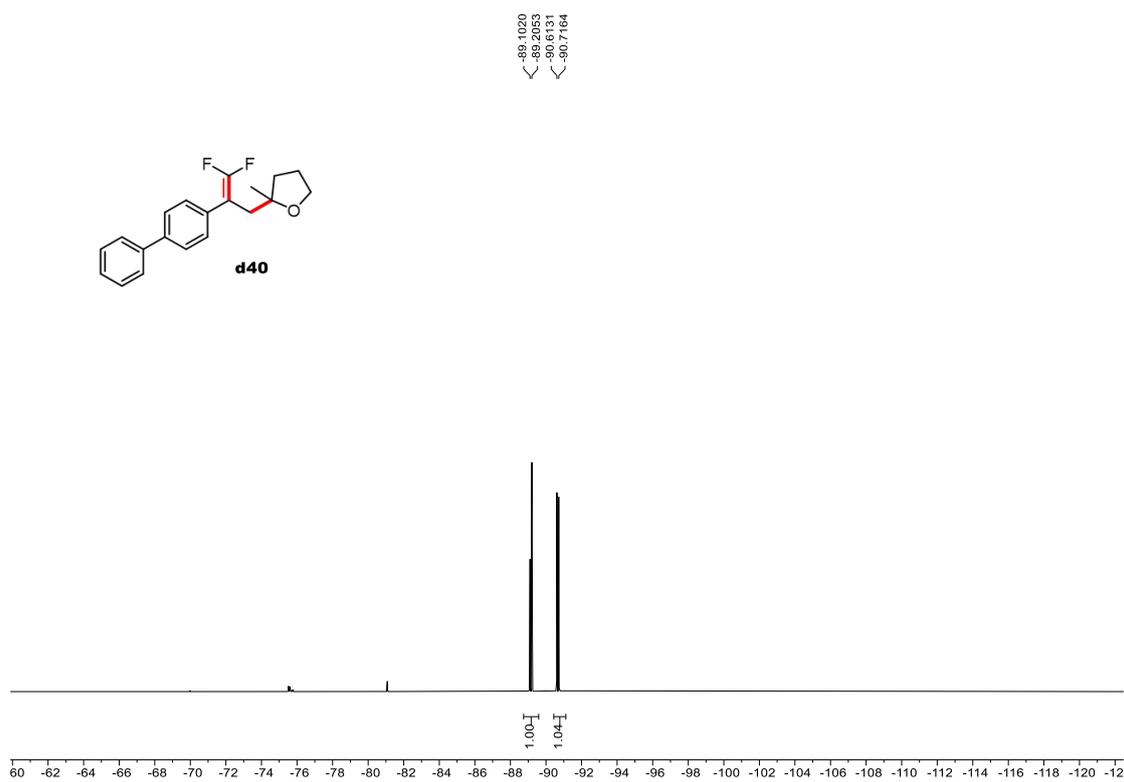
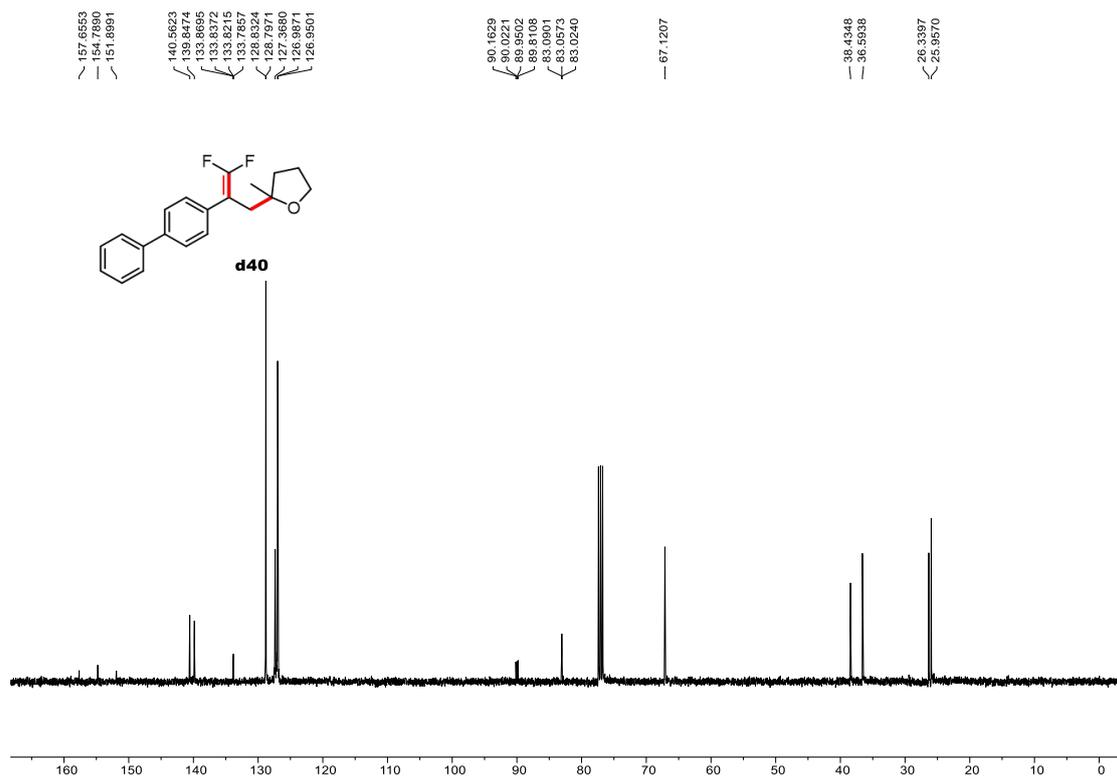
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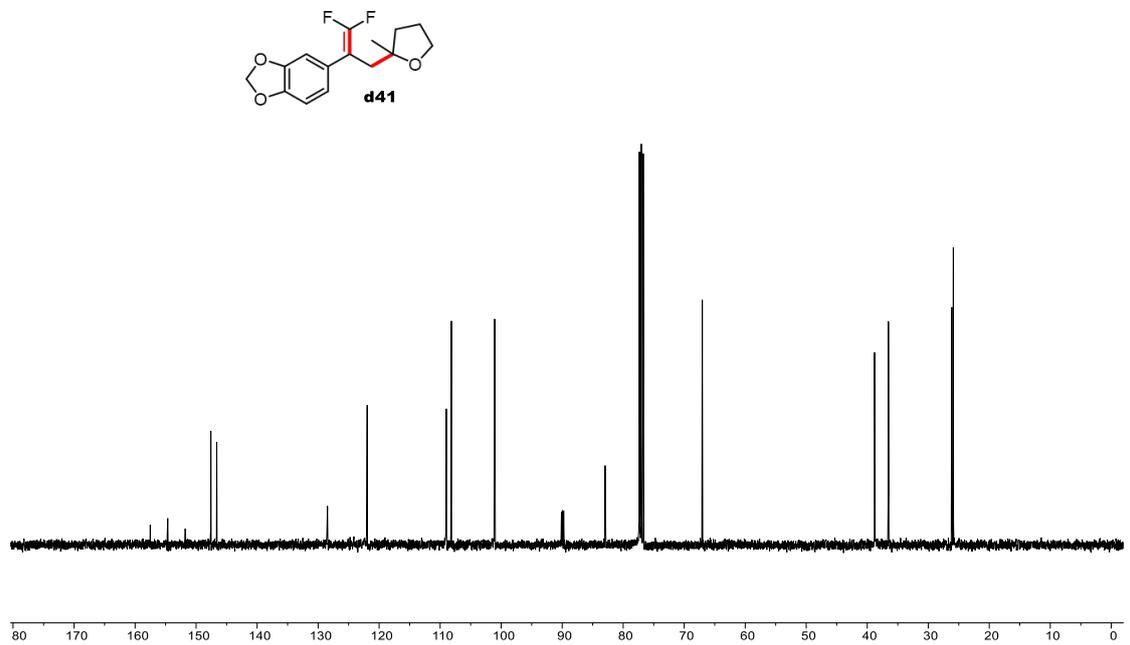
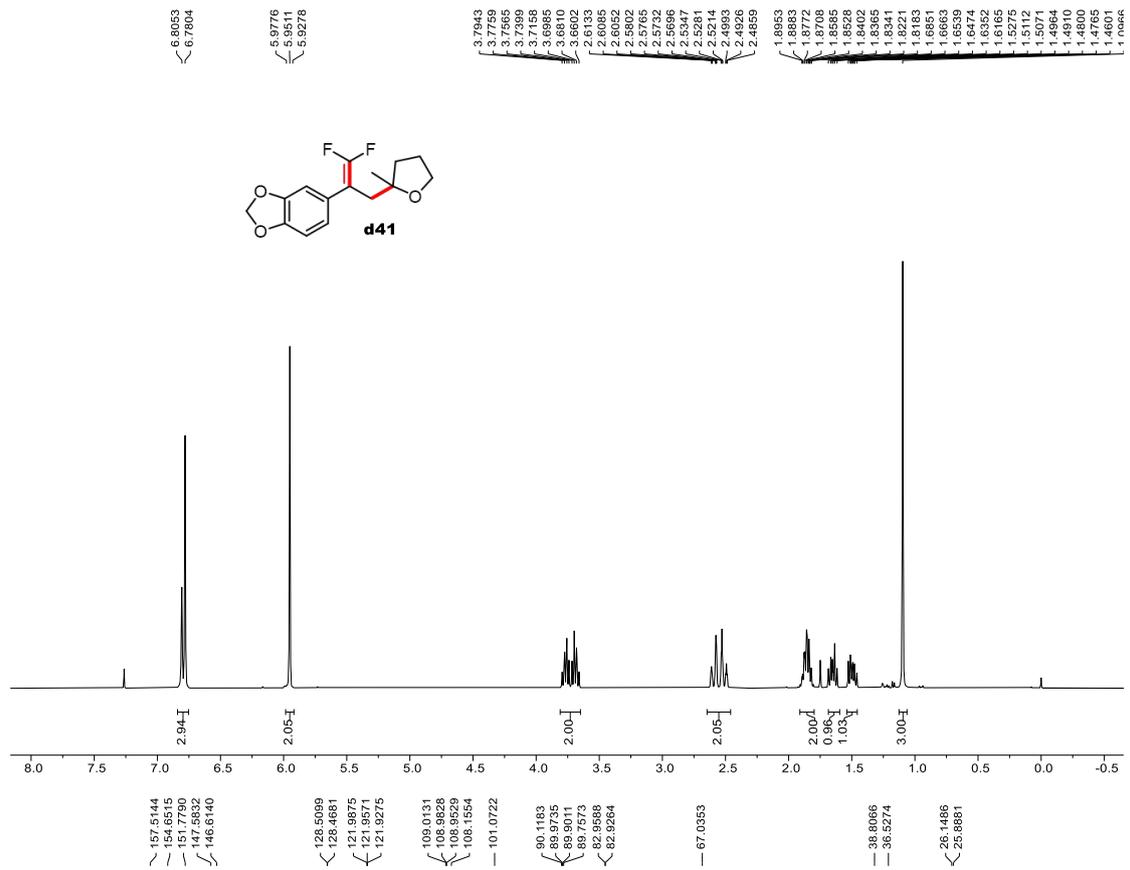
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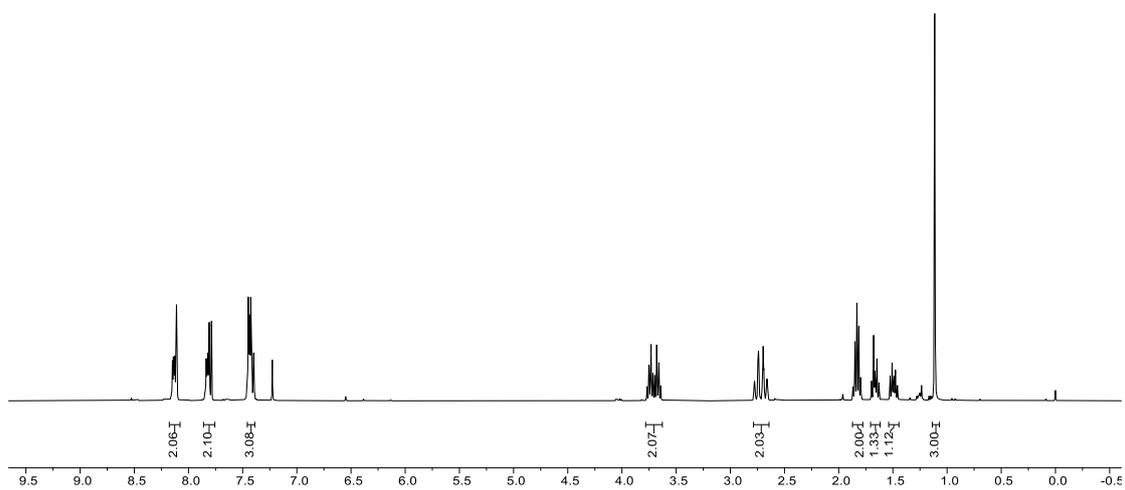
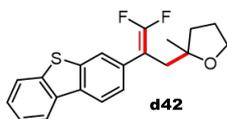
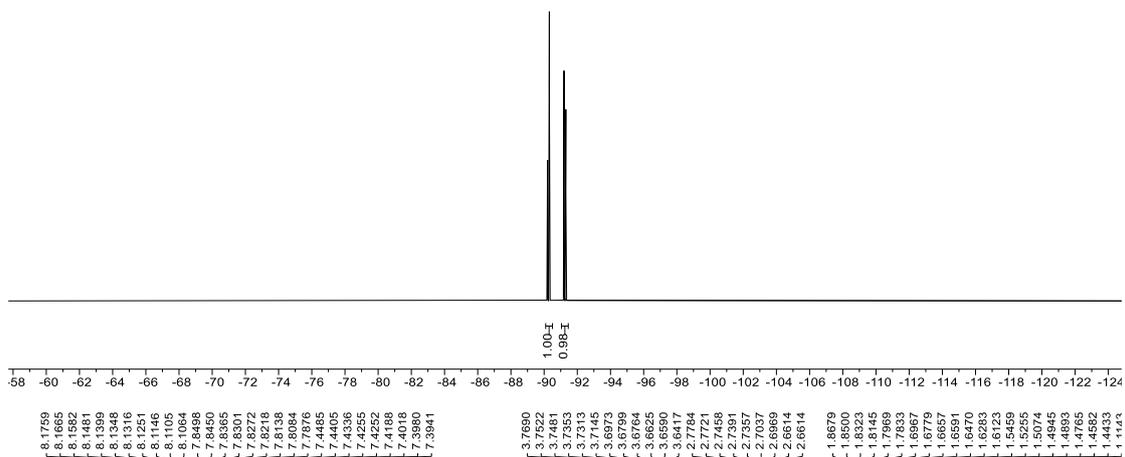


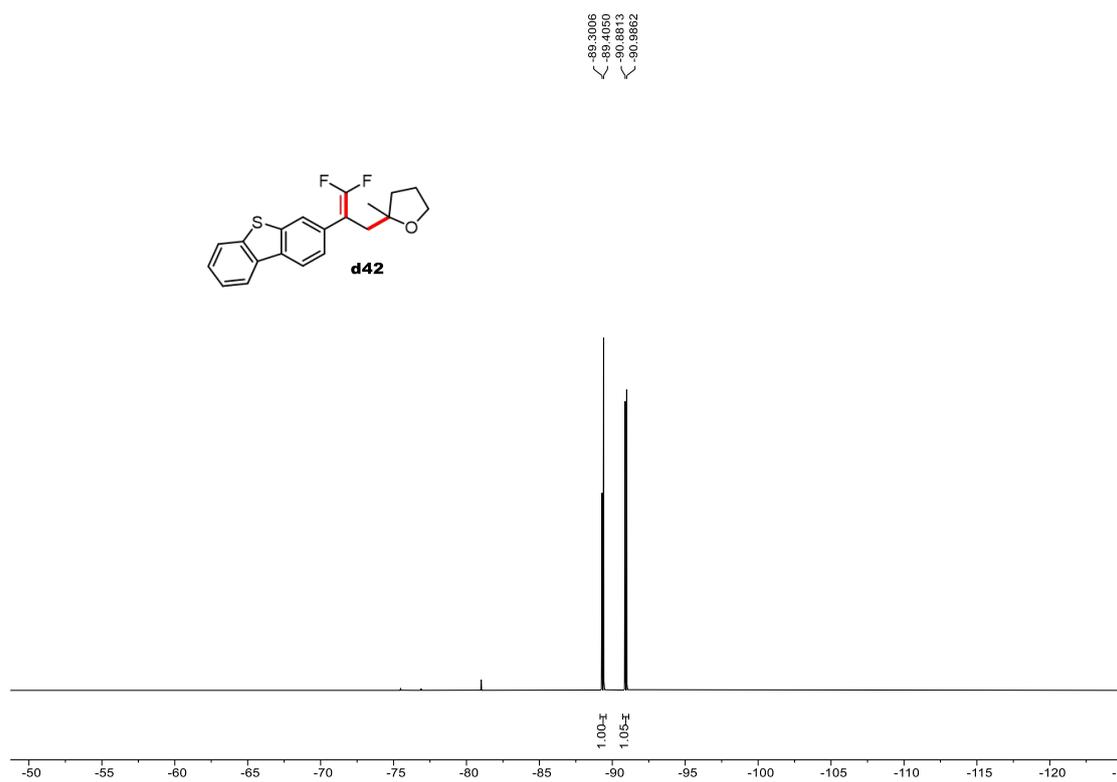
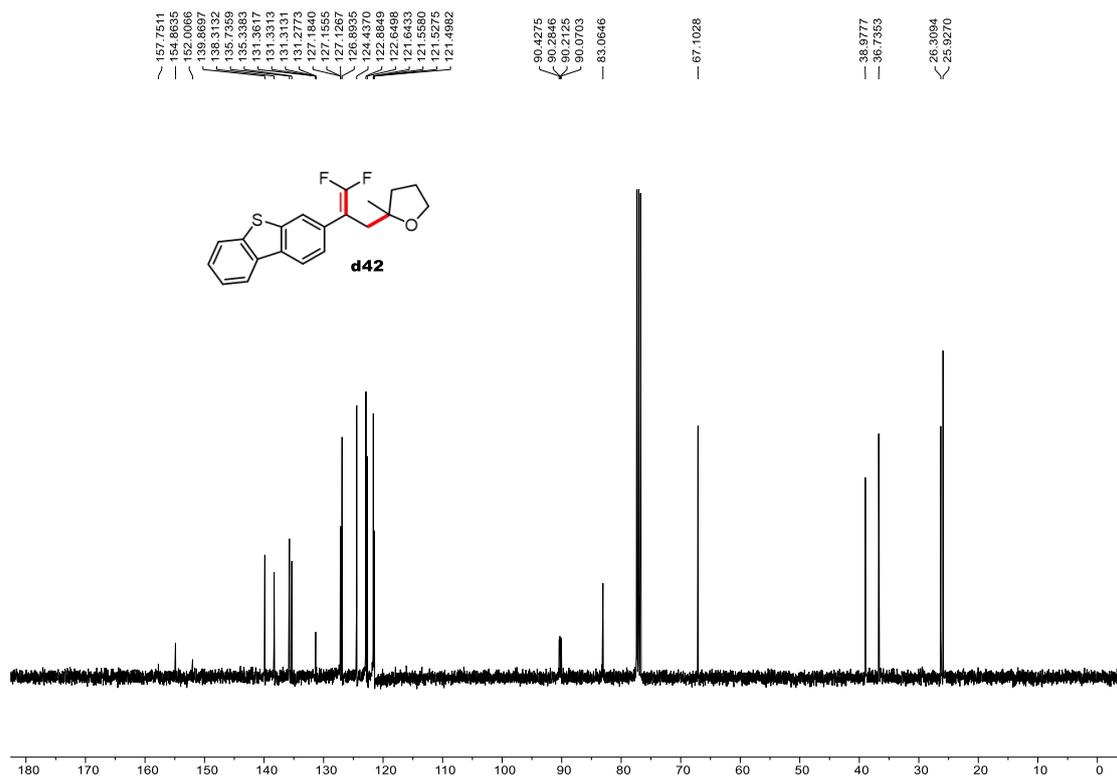


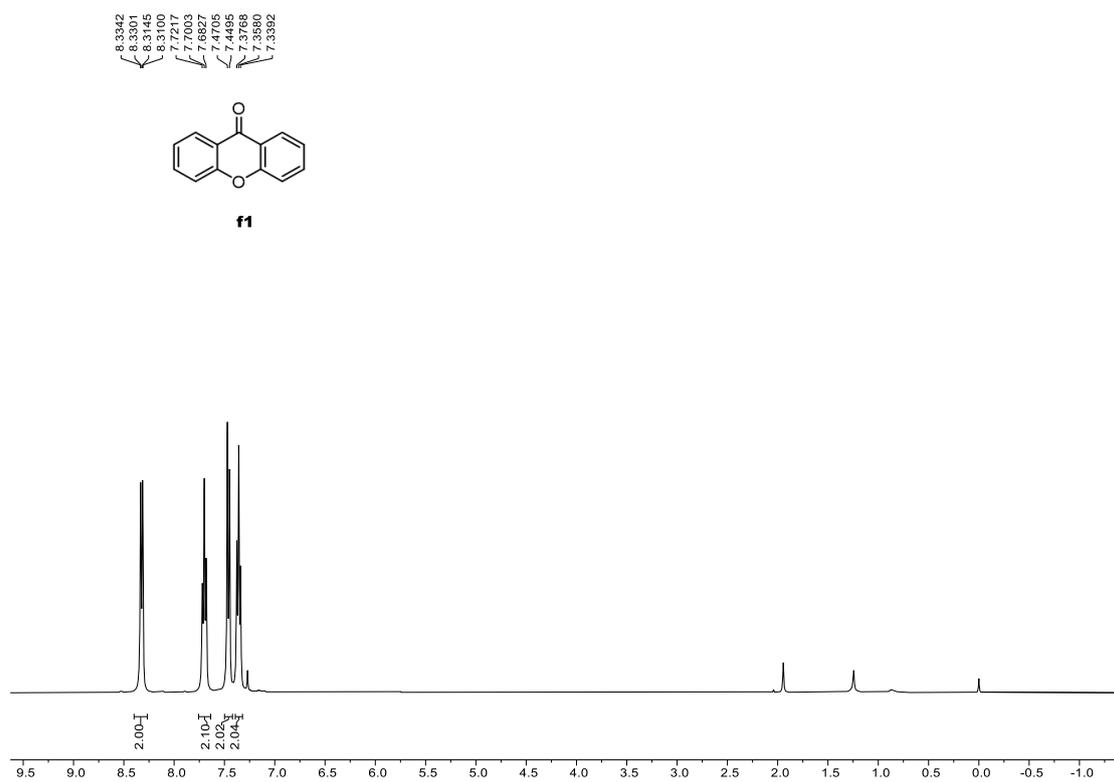
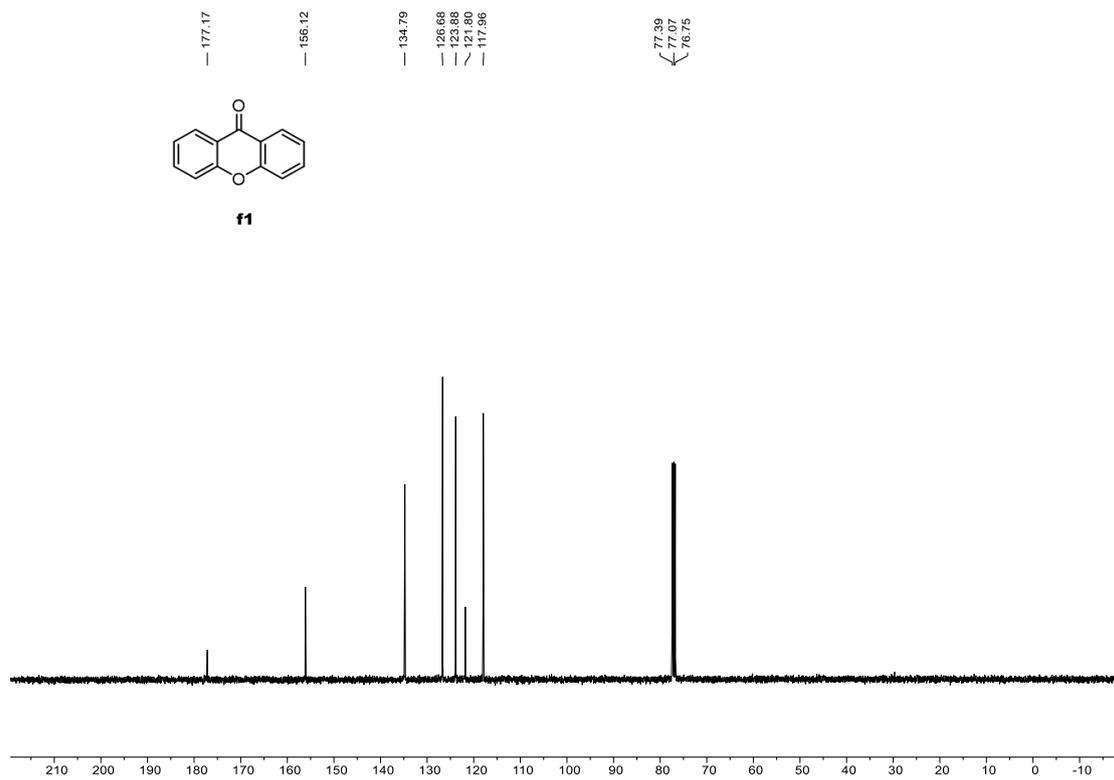


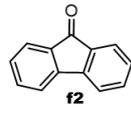


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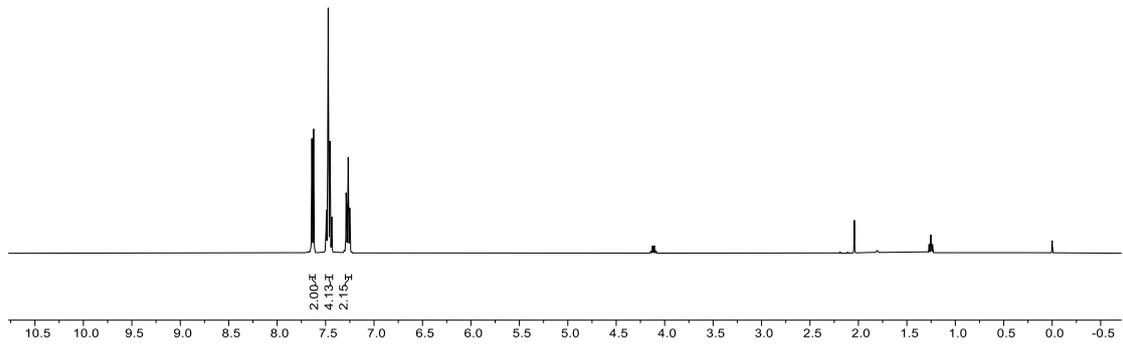




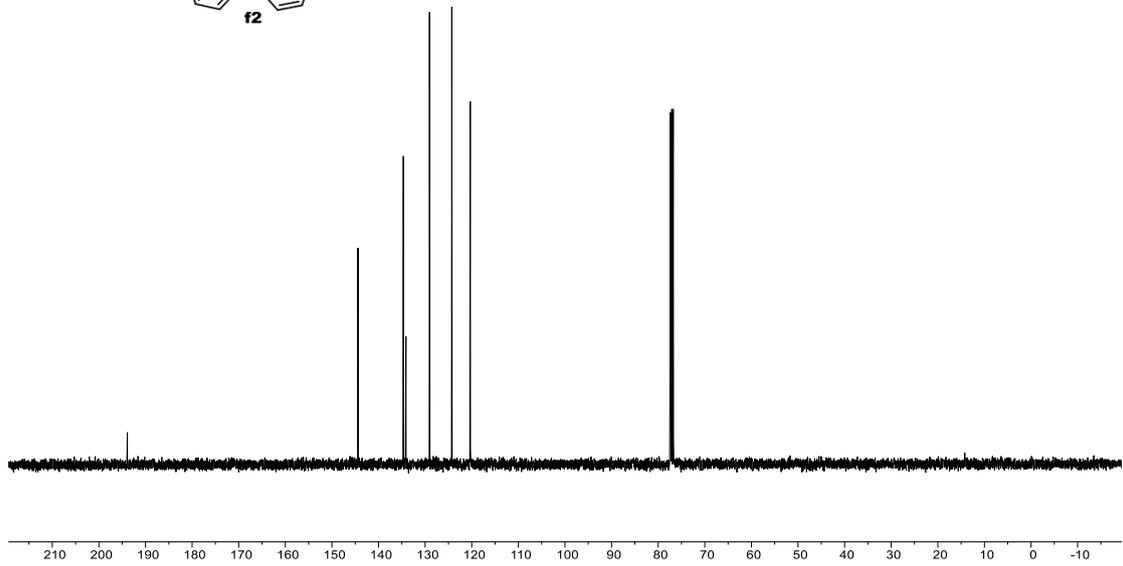
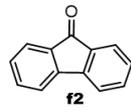




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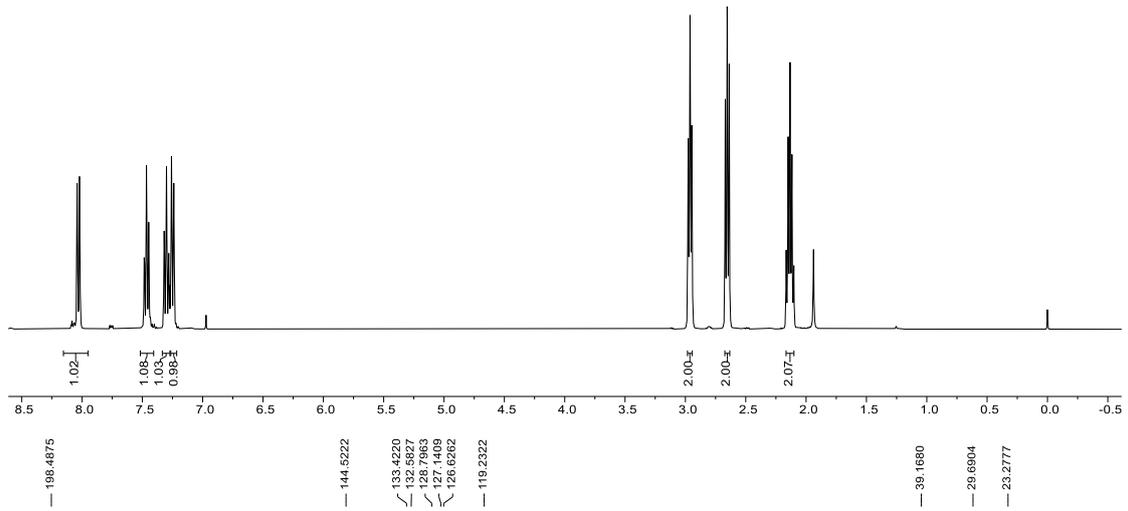


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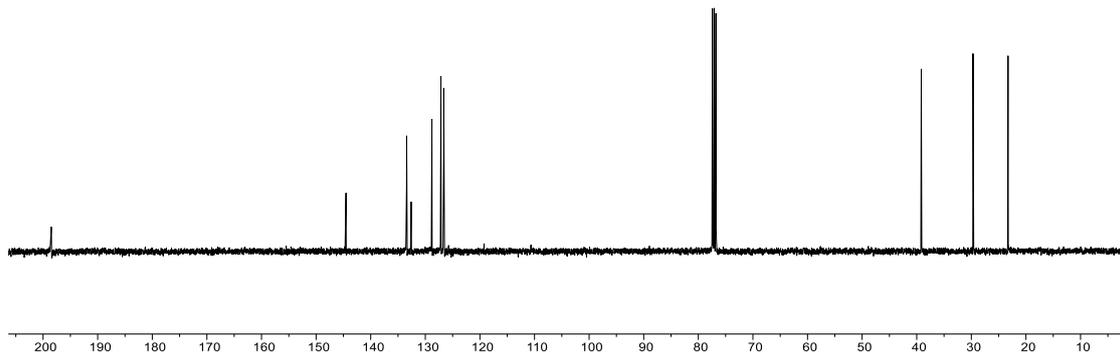
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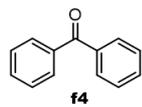


f3



f3





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