

Supporting Information

for

Photoredox-Catalyzed Hydroxydifluoroacetylation of Alkenes with FSO₂CF₂CO₂Me and H₂O: Simple Synthesis of CF₂CO₂Me-containing Alcohol and Difluorolactone

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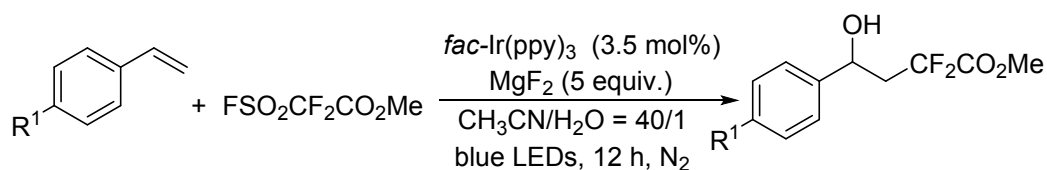
1. General Comments

All the reactions were carried out in oven-dried sealed tube with Teflon-lined septum under N₂ atmosphere. Unless indicated, all materials were obtained from commercial sources and used as received. Superdry acetonitrile with molecular sieves in it was use in the reaction. ¹H NMR, ¹³C NMR and ¹⁹F NMR spectra were recorded on 400 MHz at ambient temperature with CDCl₃ as the solvent. Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of CDCl₃ (7.26), to the carbon resonance of CDCl₃ (77.16). Coupling constants (*J*) were given in Hertz (Hz). The term m, q, t, d, s referred to multiplet, quartet, triplet, doublet, singlet. The reaction progress was monitored by GC-MS if applicable. Column chromatography was performed with silica gel (200-300 meshes). Thin layer chromatography (TLC) was visualized using UV light. Fluorescence quenching experiments were measured on an Ahilent Technologies Cary Eclipse Fluorescence Spectrophotometer.

1-Methyl-4-vinylbenzene **1c**,¹ 1-Methyl-2-vinylbenzene **1i**,¹ 2-vinylnaphthalene **1q**,¹ 3-vinylestrone **1s**,³ 1,2-di-p-tolylolethene **1z**,² and 1-methyl-4-styrylbenzene **1za**² were synthesized according to literature procedures.

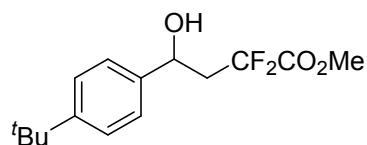
2. Experimental Section

2-1. General procedure for hydroxydifluoroacetylation of alkenes

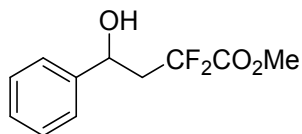


An oven-dried Schleck tube equipped with a stirrer bar was charged with 4.58 mg *fac*-Ir(ppy)₃ (3.5 mol%), which was degassed and refilled with N₂ for 5 times. The alkenes **1a-1s** (0.2 mmol, 1.0 equiv.), Chen reagent (FSO₂CF₂COOMe) (134.4 mg, 0.7 mmol, 3.5 equiv.), MgF₂ (62.3 mg, 1.0 mmol, 5 equiv.), H₂O (0.1 mL) and dry CH₃CN (4 mL) were added under N₂. The reaction mixture was irradiated for 12 h under room temperature by 3 W blue LEDs. Quenching the reaction with H₂O and the aqueous layer was extracted with ethyl acetate (EA) twice. The combined organic layer dried by

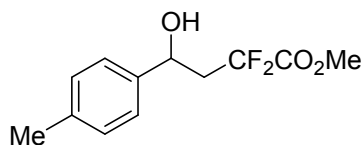
Na₂SO₄ and concentrated in *vacuo*. The residue was purified by chromatography on silica gel to give product **3a-3s**, which were identified by ¹H, ¹³C and ¹⁹F NMR.



Methyl 4-(4-(*tert*-butyl)phenyl)-2,2-difluoro-4-hydroxybutanoate (3a): white solid, 44.0 mg (77% yield), mp: 49.1-50.2 °C. ¹H NMR (400 MHz, CHLOROFORM-D) δ 7.41 – 7.36 (m, 2H), 7.30 – 7.27 (m, 2H), 4.99 (dt, *J* = 10.0, 3.2 Hz, 1H), 3.84 (s, 3H), 2.77 – 2.62 (m, 1H), 2.46 – 2.34 (m, 1H), 2.06 (dd, *J* = 3.6, 1.1 Hz, 1H), 1.31 (s, 9H). ¹³C NMR (101 MHz, CHLOROFORM-D) δ 164.91 (t, *J* = 35.9 Hz), 151.52, 139.71, 125.81, 125.58, 115.42 (dd, *J* = 251.7, 248.8 Hz), 68.74 (dd, *J* = 8.5, 3.6 Hz), 53.49, 43.81 (t, *J* = 22.7 Hz), 34.73, 31.44. ¹⁹F NMR (376 MHz, CHLOROFORM-D) δ -104.55 (dddd, *J* = 264.3, 36.7, 19.0, 10.9 Hz). HRMS (ESI) Exact mass calculated for C₁₅H₂₀F₂O₃ [M+H]⁺: 287.1453; found: 287.1450.

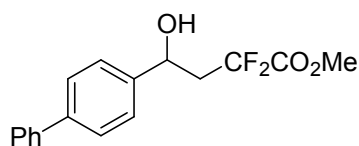


Methyl 2,2-difluoro-4-hydroxy-4-phenylbutanoate (3b): colorless oil liquid, 32.2 mg (70% yield). ¹H NMR (400 MHz, CHLOROFORM-D) δ 7.38 – 7.27 (m, 5H), 5.03 – 4.97 (m, 1H), 3.84 (s, 3H), 2.74 – 2.60 (m, 1H), 2.44 – 2.33 (m, 1H), 2.23 (dd, *J* = 3.6, 0.7 Hz, 1H). ¹³C NMR (101 MHz, CHLOROFORM-D) δ 164.89 (t, *J* = 32.6 Hz), 142.72, 128.88, 128.38, 125.78, 115.35 (dd, *J* = 251.8, 248.8 Hz), 68.91 (dd, *J* = 8.4, 3.5 Hz), 53.51, 43.90 (t, *J* = 22.7 Hz). ¹⁹F NMR (376 MHz, CHLOROFORM-D) δ -104.54 (dddd, *J* = 262.4, 36.2, 18.7, 10.4 Hz). HRMS (ESI) Exact mass calculated for C₁₁H₁₂F₂O₃ [M+H]⁺: 231.0827; found: 231.0831.

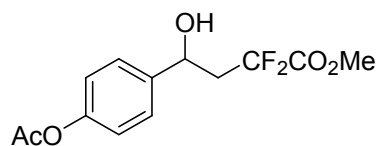


Methyl 2,2-difluoro-4-hydroxy-4-(*p*-tolyl)butanoate (3c): colorless oil liquid, 35.1 mg (72% yield). ¹H NMR (400 MHz, CHLOROFORM-D) δ 7.25 – 7.22 (m, 2H), 7.17

(d, $J = 8.0$ Hz, 2H), 4.96 (dt, $J = 10.0, 3.1$ Hz, 1H), 3.84 (s, 3H), 2.67 (ddd, $J = 24.4, 9.8, 5.0$ Hz, 1H), 2.42 – 2.34 (m, 4H), 2.14 (d, $J = 3.5$ Hz, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.94 (t, $J = 35.9$ Hz), 139.79, 138.18, 129.52, 125.74, 115.39 (dd, $J = 251.6, 248.5$ Hz), 68.75 (dd, $J = 8.4, 3.5$ Hz), 53.48, 43.86 (t, $J = 22.7$ Hz), 21.23. ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.55 (dddd, $J = 263.5, 36.1, 19.8, 10.9$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{12}\text{H}_{14}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 245.0894; found: 245.0888.

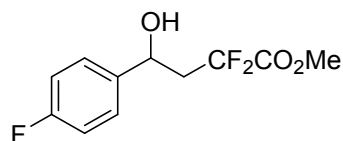


Methyl 4-([1,1'-biphenyl]-4-yl)-2,2-difluoro-4-hydroxybutanoate (3d): white solid, 40.4 mg (66% yield), mp: 48.3-49.2 °C. ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.61 – 7.56 (m, 4H), 7.46 – 7.42 (m, 4H), 7.38 – 7.33 (m, 1H), 5.08 (dt, $J = 10.0, 3.1$ Hz, 1H), 3.87 (s, 3H), 2.73 (ddd, $J = 23.9, 9.9, 4.9$ Hz, 1H), 2.43 (ddd, $J = 15.4, 8.2, 6.4$ Hz, 1H), 2.15 (d, $J = 3.6$ Hz, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.90 (dd, $J = 33.2, 31.9$ Hz), 141.68, 141.27, 140.60, 128.94, 127.60, 127.54, 127.17, 126.25, 115.35 (dd, $J = 251.6, 248.8$ Hz), 68.61 (dd, $J = 8.4, 3.2$ Hz), 53.52, 43.84 (t, $J = 22.7$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.50 (dddd, $J = 264.3, 36.7, 19.2, 11.8$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{17}\text{H}_{16}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 307.1140; found: 307.1145.

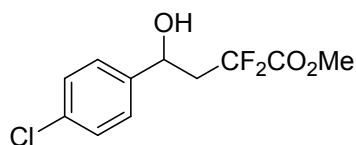


Methyl 4-(4-acetoxyphenyl)-2,2-difluoro-4-hydroxybutanoate (3e): pale yellow oil liquid, 34.6 mg (60% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.40 – 7.36 (m, 2H), 7.12 – 7.06 (m, 2H), 5.03 (dt, $J = 9.9, 3.1$ Hz, 1H), 3.86 (s, 3H), 2.73 – 2.60 (m, 1H), 2.38 (dt, $J = 12.2, 6.5$ Hz, 1H), 2.30 (s, 3H), 2.16 (dd, $J = 3.6, 0.7$ Hz, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 169.65, 164.78 (t, $J = 35.9$ Hz), 150.45, 140.36, 127.12, 126.93, 122.52, 121.94, 115.23 (dd, $J = 251.7, 248.9$ Hz), 68.25 (dd, $J = 8.0, 3.4$ Hz), 53.49, 43.83 (t, $J = 22.8$ Hz), 21.14. ^{19}F NMR (376 MHz,

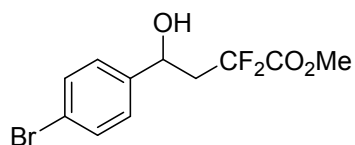
CHLOROFORM-D) δ -104.53 (dddd, $J = 264.4, 36.7, 19.1, 11.8$ Hz). HRMS (ESI) Exact mass calculated for $C_{13}H_{14}F_2O_5$ $[M+H]^+$: 289.0882; found: 289.0886.



Methyl 2,2-difluoro-4-(4-fluorophenyl)-4-hydroxybutanoate (3f): colorless oil liquid, 32.7 mg (66% yield). 1H NMR (400 MHz, CHLOROFORM-D) δ 7.32 (ddd, $J = 8.2, 5.2, 2.5$ Hz, 2H), 7.07 – 7.00 (m, 2H), 5.00 (dt, $J = 9.8, 2.8$ Hz, 1H), 3.85 (s, 3H), 2.71 – 2.57 (m, 1H), 2.41 – 2.29 (m, 2H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.85 (dd, $J = 33.0, 31.8$ Hz), 162.59 (d, $J = 246.6$ Hz), 138.56 (d, $J = 2.9$ Hz), 127.54 (d, $J = 8.3$ Hz), 115.81, 115.71 (d, $J = 21.5$ Hz), 115.24 (dd, $J = 251.5, 249.3$ Hz), 68.23 (dd, $J = 8.3, 3.5$ Hz), 53.55, 43.92 (t, $J = 22.7$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.95 (dddd, $J = 264.4, 35.0, 23.0, 11.9$ Hz, 2F), -113.71 (td, $J = 12.0, 3.8$ Hz, 1F). HRMS (ESI) Exact mass calculated for $C_{11}H_{11}F_3O_3$ $[M+H]^+$: 249.0733; found: 249.0737.

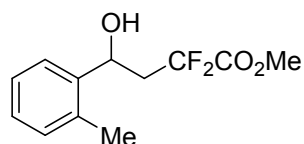


Methyl 4-(4-chlorophenyl)-2,2-difluoro-4-hydroxybutanoate (3g): colorless oil liquid, 41.2 mg (78% yield). 1H NMR (400 MHz, CHLOROFORM-D) δ 7.35 – 7.31 (m, 2H), 7.31 – 7.27 (m, 2H), 5.00 (d, $J = 10.0$ Hz, 1H), 3.86 (s, 3H), 2.70 – 2.56 (m, 1H), 2.41 – 2.30 (m, 2H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.81 (t, $J = 35.7$ Hz), 141.22, 134.08, 129.02, 127.18, 115.19 (dd, $J = 251.8, 249.2$ Hz), 68.24 (dd, $J = 8.4, 3.5$ Hz), 53.59, 43.88 (t, $J = 22.9$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.53 (dddd, $J = 264.4, 36.7, 19.1, 12.0$ Hz). HRMS (ESI) Exact mass calculated for $C_{11}H_{11}ClF_2O_3$ $[M+H]^+$: 265.0438; found: 265.0442.

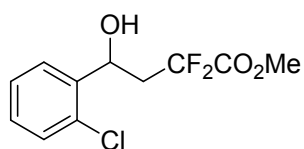


Methyl 4-(4-bromophenyl)-2,2-difluoro-4-hydroxybutanoate (3h): white solid, 33.9 mg (55% yield), mp: 45.2-46.1 °C. 1H NMR (400 MHz, CHLOROFORM-D) δ 7.52 –

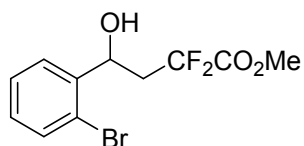
7.46 (m, 2H), 7.25 – 7.21 (m, 2H), 4.98 (d, $J = 10.0$ Hz, 1H), 3.85 (s, 3H), 2.68 – 2.55 (m, 1H), 2.37 (ddd, $J = 30.4, 13.5, 2.7$ Hz, 2H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.81 (t, $J = 36.0$ Hz), 141.74, 131.95, 127.50, 122.15, 115.16 (dd, $J = 251.7, 249.3$ Hz), 68.25 (dd, $J = 8.3, 3.4$ Hz), 53.59, 43.81 (t, $J = 22.8$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -105.54 (dddd, $J = 264.5, 36.1, 23.4, 12.9$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{11}\text{H}_{11}\text{BrF}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 308.9932; found: 308.9938.



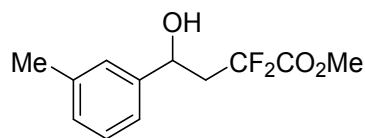
Methyl 2,2-difluoro-4-hydroxy-4-(o-tolyl)butanoate (3i): colorless oil liquid, 24.9 mg (51% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.24 (t, $J = 5.0$ Hz, 1H), 7.13 (dd, $J = 15.9, 7.9$ Hz, 3H), 4.97 (dt, $J = 10.1, 3.1$ Hz, 1H), 3.85 (s, 3H), 2.74 – 2.59 (m, 1H), 2.42 – 2.33 (m, 4H), 2.18 (dd, $J = 3.6, 0.7$ Hz, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.92 (dd, $J = 33.3, 32.0$ Hz), 142.70, 138.64, 129.10, 128.78, 126.44, 122.81, 115.39 (dd, $J = 251.7, 248.7$ Hz), 68.91 (dd, $J = 8.4, 3.5$ Hz), 53.49, 43.90 (t, $J = 22.7$ Hz), 21.52. ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.58 (dddd, $J = 262.7, 35.6, 19.3, 10.9$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{12}\text{H}_{14}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 245.0984; found: 245.0980.



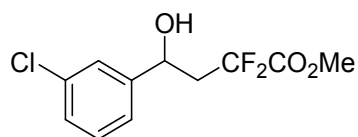
Methyl 4-(2-chlorophenyl)-2,2-difluoro-4-hydroxybutanoate (3j): colorless oil liquid, 31.7 mg (60% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.60 (dd, $J = 7.7, 1.7$ Hz, 1H), 7.33 (ddd, $J = 15.4, 7.7, 1.3$ Hz, 2H), 7.25 (dd, $J = 7.2, 5.4$ Hz, 1H), 5.48 – 5.42 (m, 1H), 3.90 (s, 3H), 2.59 – 2.48 (m, 2H), 2.30 (d, $J = 4.0$ Hz, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.95 (dd, $J = 33.3, 31.9$ Hz), 140.04, 131.48, 129.70, 129.28, 127.48, 127.06, 115.34 (dd, $J = 252.3, 249.0$ Hz), 65.62 (dd, $J = 8.5, 3.4$ Hz), 53.58, 42.19 (t, $J = 22.9$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -93.13 – -116.52 (m). HRMS (ESI) Exact mass calculated for $\text{C}_{11}\text{H}_{11}\text{ClF}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 265.0438; found: 265.0439.



Methyl 4-(2-bromophenyl)-2,2-difluoro-4-hydroxybutanoate (3k): colorless oil liquid, 30.8 mg (50% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.59 (dd, $J = 7.8, 1.6$ Hz, 1H), 7.52 (dd, $J = 8.0, 1.1$ Hz, 1H), 7.35 (td, $J = 7.7, 1.1$ Hz, 1H), 7.19 – 7.13 (m, 1H), 5.38 (dt, $J = 9.0, 4.5$ Hz, 1H), 3.89 (s, 3H), 2.54 – 2.45 (m, 3H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.96 (dd, $J = 33.2, 31.9$ Hz), 141.59, 132.94, 129.61, 128.10, 127.33, 121.39, 115.28 (dd, $J = 252.4, 248.7$ Hz), 67.79 (dd, $J = 8.6, 3.3$ Hz), 53.58, 42.23 (t, $J = 23.1$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.77 (ddt, $J = 263.8, 36.1, 15.1$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{11}\text{H}_{11}\text{BrF}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 308.9932; found: 308.9938.

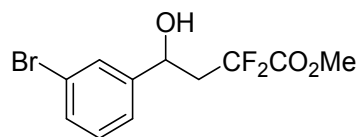


Methyl 2,2-difluoro-4-hydroxy-4-(m-tolyl)butanoate (3l): colorless oil liquid, 32.2 mg (66% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.47 (dd, $J = 7.4, 1.5$ Hz, 1H), 7.25 – 7.11 (m, 3H), 5.24 (d, $J = 10.1$ Hz, 1H), 3.85 (s, 3H), 2.66 – 2.52 (m, 1H), 2.38 – 2.24 (m, 4H), 2.16 – 2.13 (m, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.90 (t, $J = 35.9$ Hz), 140.84, 134.23, 130.74, 128.02, 126.64, 125.19, 115.46 (dd, $J = 251.5, 249.1$ Hz), 65.33 (dd, $J = 8.5, 3.3$ Hz), 53.50, 42.97 (t, $J = 22.7$ Hz), 18.83. ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.77 (dddd, $J = 262.9, 37.2, 19.0, 11.3$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{12}\text{H}_{14}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 245.0984; found: 245.0985.

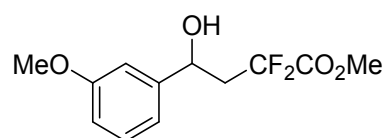


Methyl 4-(3-chlorophenyl)-2,2-difluoro-4-hydroxybutanoate (3m): colorless oil liquid, 18.5 mg (35% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.37 (s, 1H), 7.33 – 7.20 (m, 3H), 5.01 (d, $J = 10.1$ Hz, 1H), 3.87 (s, 3H), 2.70 – 2.56 (m, 1H), 2.44 – 2.31 (m, 2H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.80 (dd, $J = 33.4, 31.7$

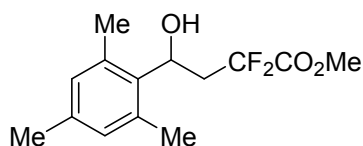
Hz), 144.77, 134.80, 130.18, 128.46, 125.99, 123.91, 115.16 (dd, $J = 251.9, 249.4$ Hz), 68.29 (dd, $J = 8.3, 3.3$ Hz), 53.62, 43.88 (t, $J = 22.9$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.58 (dddd, $J = 264.5, 35.2, 18.4, 12.0$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{11}\text{H}_{11}\text{ClF}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 265.0438; found: 265.0439.



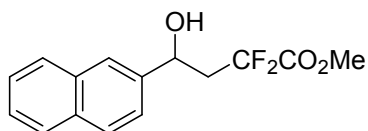
Methyl 4-(3-bromophenyl)-2,2-difluoro-4-hydroxybutanoate (3n): colorless oil liquid, 37.0 mg (60% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.53 (s, 1H), 7.43 (d, $J = 7.5$ Hz, 1H), 7.25 (dd, $J = 13.7, 7.6$ Hz, 2H), 4.99 (d, $J = 10.1$ Hz, 1H), 3.86 (s, 3H), 2.71 – 2.56 (m, 1H), 2.38 (ddd, $J = 28.4, 11.7, 2.9$ Hz, 2H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.88 (dd, $J = 33.6, 31.6$ Hz), 145.02, 131.38, 130.46, 128.90, 124.39, 122.93, 115.14 (dd, $J = 251.6, 249.3$ Hz), 68.20 (dd, $J = 8.3, 3.2$ Hz), 53.62, 43.86 (t, $J = 22.8$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -94.84 – -113.49 (m). HRMS (ESI) Exact mass calculated for $\text{C}_{11}\text{H}_{11}\text{BrF}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 308.9932; found: 308.9938.



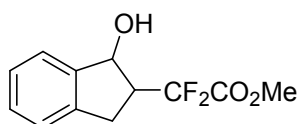
Methyl 2,2-difluoro-4-hydroxy-4-(3-methoxyphenyl)butanoate (3o): 164.88 (dd, $J = 33.6, 31.6$ Hz), 38.8 mg (31% yield). ^1H NMR (400 MHz, CDCl_3) δ 7.28 (t, $J = 7.3$ Hz, 1H), 6.92 (d, $J = 8.0$ Hz, 2H), 6.84 (d, $J = 8.1$ Hz, 1H), 4.99 (d, $J = 10.0$ Hz, 1H), 3.86 (s, 3H), 3.81 (s, 3H), 2.65 (ddd, $J = 19.7, 14.4, 7.4$ Hz, 1H), 2.39 (q, $J = 13.8$ Hz, 1H), 2.19 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 164.88 (dd, $J = 33.6, 31.6$ Hz), 160.06, 144.43, 129.97, 117.83, 115.34 (dd, $J = 251.8, 248.6$ Hz), 113.84, 111.25, 68.85 (dd, $J = 8.5, 3.5$ Hz), 55.42, 53.53, 43.94 (t, $J = 22.7$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.89 (dddd, $J = 263.0, 36.7, 24.5, 12.6$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{12}\text{H}_{14}\text{F}_2\text{O}_4$ $[\text{M}+\text{H}]^+$: 261.0933; found: 261.0935.



Methyl 2,2-difluoro-4-hydroxy-4-mesitylbutanoate (3p): colorless oil liquid, 49.0 mg (85% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 6.81 (s, 2H), 5.44 (dt, $J = 10.4, 2.8$ Hz, 1H), 3.84 (s, 3H), 3.03 – 2.89 (m, 1H), 2.39 (s, 6H), 2.30 (ddd, $J = 14.8, 7.7, 2.8$ Hz, 1H), 2.23 (s, 3H), 2.01 – 1.97 (m, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.86(t, $J = 35.9$ Hz), 137.49, 136.15, 134.75, 130.37, 1115.55 (dd, $J = 251.3, 248.9$ Hz), 65.57 (dd, $J = 8.4, 3.3$ Hz), 53.41, 40.51 (t, $J = 22.5$ Hz), 20.82, 20.39. ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -105.22 (dddd, $J = 262.5, 36.8, 18.8, 11.3$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{14}\text{H}_{18}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 273.1297; found: 273.125.

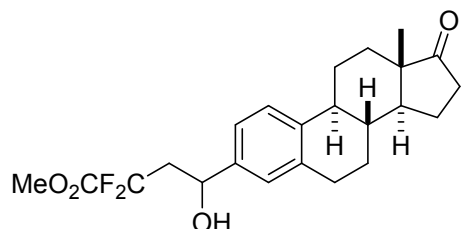


Methyl 2,2-difluoro-4-hydroxy-4-(naphthalen-2-yl)butanoate (3q): yellow solid, 21.8 mg (42% yield), mp: 135.3-136.1 °C. ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.85 – 7.78 (m, 4H), 7.52 – 7.41 (m, 3H), 5.16 (d, $J = 10.0$ Hz, 1H), 3.82 (s, 3H), 2.75 (ddd, $J = 24.2, 9.8, 5.0$ Hz, 1H), 2.47 (ddd, $J = 27.3, 14.7, 2.9$ Hz, 1H), 2.36 (s, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.91 (t, $J = 32.5$ Hz), 140.00, 133.29, 133.26, 128.81, 128.11, 127.83, 126.57, 126.39, 124.68, 123.57, 115.38 (dd, $J = 251.8, 249.1$ Hz), 69.01 (dd, $J = 8.4, 3.5$ Hz), 53.53, 43.84 (t, $J = 22.8$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.46 (dddd, $J = 264.3, 36.8, 18.4, 11.1$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{15}\text{H}_{14}\text{F}_2\text{O}_3$ $[\text{M}+\text{H}]^+$: 281.0984; found: 281.0982.



methyl 2,2-difluoro-2-(1-hydroxy-2,3-dihydro-1H-inden-2-yl)acetate (3r): colorless oil liquid, 27.1 mg (56% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.40 – 7.36 (m, 1H), 7.30 – 7.26 (m, 2H), 7.22 (dd, $J = 4.9, 3.6$ Hz, 1H), 5.41 (t, $J = 6.5$ Hz, 1H), 3.91 (s, 3H), 3.15 (dd, $J = 15.6, 8.7$ Hz, 1H), 3.08 – 2.93 (m, 2H), 2.64 (d, $J =$

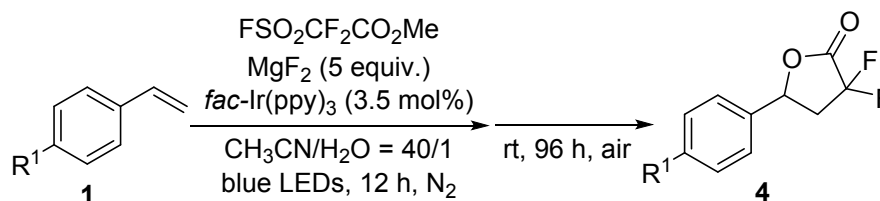
6.4 Hz, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 165.02 (t, $J = 32.7$ Hz), 142.67, 139.39, 128.89, 127.54, 124.86, 124.35, 116.29 (t, $J = 252.3$ Hz), 75.59 (dd, $J = 6.8, 3.3$ Hz), 53.92 (t, $J = 20.7$ Hz), 29.86 (t, $J = 4.2$ Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -110.44 (ddd, $J = 281.6, 261.8, 17.5$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{11}\text{H}_{12}\text{F}_2\text{O}_2$ $[\text{M}+\text{H}]^+$: 242.0755; found: 242.0754.



Methyl 2,2-difluoro-4-hydroxy-4-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)butanoate (3s): yellow oil liquid, 63.3 mg (78% yield).

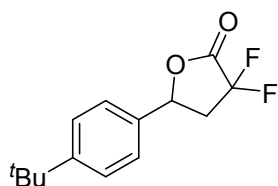
^1H NMR (400 MHz, CHLOROFORM-D) δ 7.28 (d, $J = 8.1$ Hz, 1H), 7.14 – 7.07 (m, 2H), 4.93 (d, $J = 10.2$ Hz, 1H), 3.85 (s, 3H), 2.91 (dd, $J = 8.8, 3.9$ Hz, 2H), 2.74 – 2.61 (m, 1H), 2.54 – 2.26 (m, 5H), 2.15 – 2.00 (m, 3H), 1.97 – 1.91 (m, 1H), 1.65 – 1.41 (m, 6H), 0.89 (s, 3H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 221.13, 164.85 (t, $J = 32.5$ Hz), 140.31, 139.76, 136.96, 126.30, 125.75, 123.11, 115.33 (dd, $J = 251.7, 248.3$ Hz), 68.51 – 64.62 (m), 53.41, 50.44, 47.99, 44.36, 43.75 (t, $J = 22.7$ Hz), 38.10, 35.85, 31.55, 29.42, 26.46, 25.74, 21.60, 13.83. ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -104.63 (dddd, $J = 262.5, 36.9, 18.7, 11.1$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{23}\text{H}_{28}\text{F}_2\text{O}_4$ $[\text{M}+\text{H}]^+$: 407.2028; found: 407.2025.

2-2. General procedure for hydroxydifluoroacetylation/esterification of alkenes

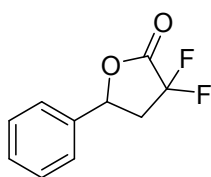


An oven-dried Schleck tube equipped with a stirrer bar was charged with 4.58 mg fac-Ir(ppy)_3 (3.5 mol%) which was degassed and refilled with N_2 for 5 times. The alkenes **1a-1c** (0.2 mmol, 1.0 equiv.), Chen's reagent ($\text{FSO}_2\text{CF}_2\text{COOMe}$) (134.4 mg,

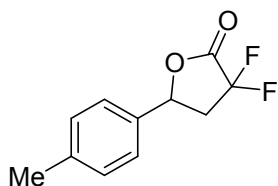
0.7 mmol, 3.5 equiv.), MgF₂ (62.3 mg, 1.0 mmol, 5 equiv.), H₂O (0.1 mL) and dry CH₃CN (4 mL) were added under N₂. The resulting mixture was irradiated for 12 h under room temperature by 3 W blue LEDs. Quenching the reaction with H₂O and the aqueous layer was extracted with ethyl acetate (EA) twice. The combined organic layer dried by Na₂SO₄, and concentrated in *vacuo* and the residue was set under the air for 4 days and then purified by chromatography on silica gel to give product **4a-4c**, which were identified by ¹H, ¹³C, and ¹⁹F NMR.



5-(4-(*tert*-Butyl)phenyl)-3,3-difluorodihydrofuran-2(3H)-one (4a): white solid, 33.5 mg (66% yield), mp: 71.6-72.3 °C. ¹H NMR (400 MHz, CHLOROFORM-D) δ 7.47 – 7.44 (m, 2H), 7.30 – 7.26 (m, 2H), 5.59 (dd, *J* = 8.9, 6.2 Hz, 1H), 3.15 – 3.05 (m, 1H), 2.74 – 2.59 (m, 1H), 1.33 (s, 9H). ¹³C NMR (101 MHz, CHLOROFORM-D) δ 165.13 (t, *J* = 32.7 Hz), 153.14, 133.17, 126.23, 125.79, 115.87 (dd, *J* = 258.9, 249.9 Hz), 77.25 (d, *J* = 8.1 Hz), 39.93 (d, *J* = 21.4 Hz), 34.90, 31.35. ¹⁹F NMR (376 MHz, CHLOROFORM-D) δ -107.70 (dddd, *J* = 279.2, 18.5, 16.0, 9.7 Hz). HRMS (ESI) Exact mass calculated for C₁₄H₁₆F₂O₂ [M+H]⁺: 255.1191; found: 255.1192.

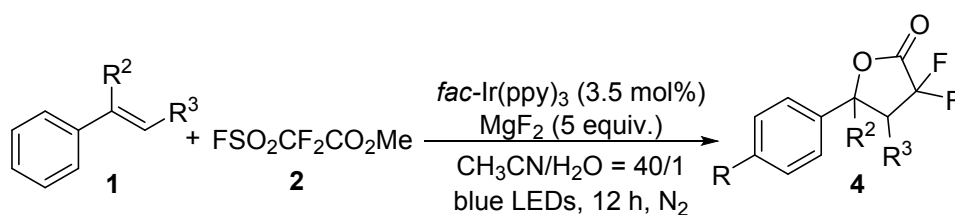


3,3-Difluoro-5-phenyldihydrofuran-2(3H)-one (4b): colorless oil liquid, 21.8 mg (55% yield). ¹H NMR (400 MHz, CHLOROFORM-D) δ 7.48 – 7.41 (m, 3H), 7.37 – 7.32 (m, 2H), 5.61 (dd, *J* = 8.8, 6.3 Hz, 1H), 3.19 – 3.09 (m, 1H), 2.72 – 2.58 (m, 1H). ¹³C NMR (101 MHz, CHLOROFORM-D) δ 165.05 (dd, *J* = 33.5, 32.2 Hz), 136.32, 129.76, 129.30, 125.81, 115.77 (dd, *J* = 258.7, 250.0 Hz), 77.21, 40.16 (t, *J* = 21.6 Hz). ¹⁹F NMR (376 MHz, CHLOROFORM-D) δ -107.70 (dddd, *J* = 279.5, 18.3, 17.3, 9.6 Hz). HRMS (ESI) Exact mass calculated for C₁₀H₈F₂O₂ [M+H]⁺: 199.0565; found: 199.0563.

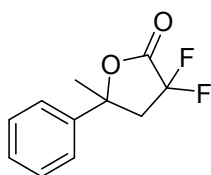


3,3-Difluoro-5-(p-tolyl)dihydrofuran-2(3H)-one (4c): white solid, 24.2 mg (57% yield), mp: 46.5-47.8 °C. ¹H NMR (400 MHz, CHLOROFORM-D) δ 7.24 (s, 4H), 5.57 (dd, *J* = 9.0, 6.1 Hz, 1H), 3.16 – 3.05 (m, 1H), 2.73 – 2.57 (m, 1H), 2.39 – 2.37 (m, 3H). ¹³C NMR (101 MHz, CHLOROFORM-D) δ 165.14 (t, *J* = 32.6 Hz), 139.93, 133.22, 129.94, 125.95, 115.86 (dd, *J* = 259.1, 250.3 Hz), 77.31 (d, *J* = 7.4 Hz), 40.00 (t, *J* = 21.6 Hz), 21.35. ¹⁹F NMR (376 MHz, CHLOROFORM-D) δ -107.73 (dddd, *J* = 279.0, 18.7, 15.8, 9.7 Hz). HRMS (ESI) Exact mass calculated for C₁₁H₁₀F₂O₂ [M+H]⁺: 213.0722; found: 213.0726.

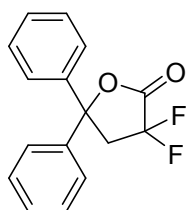
2-3. General procedure for hydroxydifluoroacetylation of disubstituted alkenes



An oven-dried Schleck tube equipped with a stirrer bar was charged with 4.58 mg *fac*-Ir(ppy)₃ (3.5 mol%) which was degassed and refilled with N₂ for 5 times. The alkenes **1v-1za** (0.2 mmol, 1.0 equiv.), Chen reagent (FSO₂CF₂COOMe) (134.4 mg, 0.7 mmol, 3.5 equiv.), MgF₂ (62.3 mg, 1.0 mmol, 5 equiv.), H₂O (0.1 mL) and dry CH₃CN (4 mL) were added under N₂. The resulting mixture was irradiated for 12 h under room temperature by 3 W blue LEDs. Quenching the reaction with H₂O and the aqueous layer was extracted with ethyl acetate (EA) twice. The combined organic layer dried by Na₂SO₄, and concentrated in *vacuo* and the residue was purified by chromatography on silica gel to give product **4v-4za**, which were identified by ¹H, ¹³C and ¹⁹F NMR.

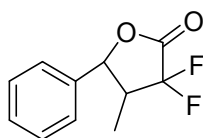


3,3-Difluoro-5-methyl-5-phenyldihydrofuran-2(3H)-one (4v): colorless oil liquid, 21.2 mg (50% yield). ¹H NMR (400 MHz, CHLOROFORM-D) δ 7.44 – 7.38 (m, 2H), 7.35 (dd, *J* = 3.8, 2.7 Hz, 2H), 7.33 (t, *J* = 2.6 Hz, 1H), 2.96 (dd, *J* = 16.6, 12.5 Hz, 2H), 1.82 (s, 3H). ¹³C NMR (101 MHz, CHLOROFORM-D) δ 164.84 (t, *J* = 33.2 Hz), 142.63, 129.06, 128.54, 123.87, 116.09 (dd, *J* = 255.5, 252.7 Hz), 84.04 (dd, *J* = 4.8, 3.8 Hz), 45.04 (t, *J* = 20.6 Hz), 30.28. ¹⁹F NMR (376 MHz, CHLOROFORM-D) δ -103.24 (ddt, *J* = 281.8, 24.7, 14.5 Hz). HRMS (ESI) Exact mass calculated for C₁₁H₁₀F₂O₂ [M+H]⁺: 213.0722; found: 213.0726.



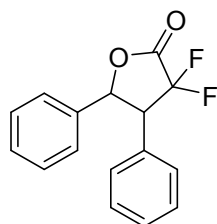
3,3-Difluoro-5,5-diphenyldihydrofuran-2(3H)-one (4w): colorless oil liquid, 30.1 mg (55% yield). ¹H NMR (400 MHz, CHLOROFORM-D) δ 7.40 – 7.30 (m, 10H), 3.43 (t, *J* = 13.8 Hz, 2H). ¹³C NMR (101 MHz, CHLOROFORM-D) δ 164.58 (t, *J* = 32.8 Hz), 141.72, 129.02, 128.75, 125.37, 115.65 (t, *J* = 254.3 Hz), 86.66 (t, *J* = 4.5 Hz), 44.66 (t, *J* = 20.8 Hz). ¹⁹F NMR (376 MHz, CHLOROFORM-D) δ -104.43 (t, *J* = 14.0 Hz).

HRMS (ESI) Exact mass calculated for C₁₆H₁₂F₂O₂ [M+H]⁺: 275.0878; found: 275.0877.

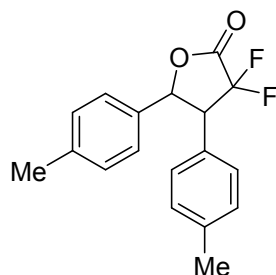


3,3-difluoro-4-methyl-5-phenyldihydrofuran-2(3H)-one (4x): colorless oil liquid, 27.6 mg (65% yield). ¹H NMR (400 MHz, CDCl₃) δ 7.48 – 7.41 (m, 3H), 7.38 – 7.32 (m, 2H), 5.06 (d, *J* = 9.6 Hz, 1H), 2.69 – 2.52 (m, 1H), 1.24 (d, *J* = 6.8 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 165.25 (dd, *J* = 34.2, 31.3 Hz), 134.89, 130.03, 129.27, 126.49, 115.76 (dd, *J* = 261.4, 250.7 Hz), 83.68 (d, *J* = 9.4 Hz), 46.04 (t, *J* = 20.5 Hz), 6.88 (d, *J* = 6.9 Hz). ¹⁹F NMR (376 MHz, CHLOROFORM-D) δ -118.65 (ddd, *J* =

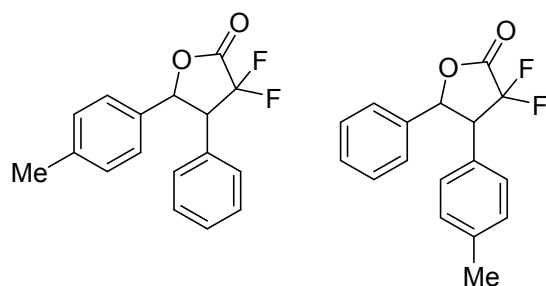
296.9, 273.5, 16.7 Hz). HRMS (ESI) Exact mass calculated for $C_{11}H_{10}F_2O_2$ $[M+H]^+$: 213.0722; found: 213.0723.



3,3-Difluoro-4,5-diphenyldihydrofuran-2(3H)-one (4y): white solid, 31.8 mg (58% yield), mp: 72.1-73.4 °C. 1H NMR (400 MHz, CHLOROFORM-D) δ 7.42 – 7.38 (m, 3H), 7.36 – 7.28 (m, 5H), 7.26 – 7.22 (m, 2H), 5.77 (d, J = 10.0 Hz, 1H), 3.76 (ddd, J = 19.1, 16.5, 10.0 Hz, 1H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.56 (t, J = 32.6 Hz), 134.85, 129.80, 129.66, 129.39, 129.25, 129.10, 128.26, 126.08, 114.58 (t, J = 257.6 Hz), 81.55 (t, J = 4.1 Hz), 56.59 (t, J = 19.7 Hz). ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -114.23 (dd, J = 18.3, 5.4 Hz). HRMS (ESI) Exact mass calculated for $C_{16}H_{12}F_2O_2$ $[M+H]^+$: 275.0878; found: 275.0877.

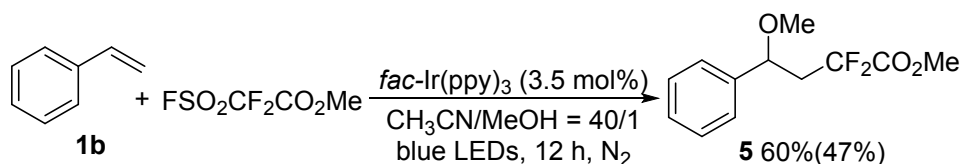


3,3-Difluoro-4,5-di-p-tolyldihydrofuran-2(3H)-one (4z): colorless oil liquid, 23.4 mg (39% yield). 1H NMR (400 MHz, CHLOROFORM-D) δ 7.21 (d, J = 1.7 Hz, 4H), 7.17 (s, 4H), 5.74 (dd, J = 10.0, 2.2 Hz, 1H), 3.82 – 3.69 (m, 1H), 2.35 (d, J = 11.5 Hz, 6H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.77 (t, J = 32.4 Hz), 139.84, 139.26, 131.78, 129.89, 129.72, 129.45, 126.22, 125.17, 114.66 (t, J = 257.0 Hz), 81.67 (t, J = 4.5 Hz), 56.12 (t, J = 19.5 Hz), 21.28, 21.23. ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -114.49 (d, J = 17.5 Hz). HRMS (ESI) Exact mass calculated for $C_{18}H_{16}F_2O_2$ $[M+H]^+$: 303.1191; found: 303.1193.

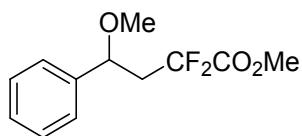


3,3-Difluoro-4-phenyl-5-(p-tolyl)dihydrofuran-2(3H)-one (4za) and 3,3-difluoro-5-phenyl-4-(p-tolyl)dihydrofuran-2(3H)-one(4za'): colorless oil liquid, 22.7 mg (40% yield). ^1H NMR (400 MHz, CHLOROFORM-D) δ 7.39 – 7.29 (m, 4H), 7.25 – 7.13 (m, 5H), 5.74 (dd, $J = 10.1, 3.6$ Hz, 1H), 3.82 – 3.64 (m, 1H), 2.35 (s, 1H), 2.32 (s, 2H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 164.66 (t, $J = 32.5$ Hz), 139.95, 139.39, 134.99, 131.74, 129.97, 129.79, 129.74, 129.64, 129.53, 129.33, 129.22, 129.08, 128.38, 126.24, 126.09, 125.15, 114.68 (t, $J = 257.5$ Hz), 81.68-81.54(m), 56.39 (td, $J = 19.4, 12.2$ Hz), 21.33, 21.30. ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -114.37 (ddd, $J = 34.3, 18.6, 9.6$ Hz). HRMS (ESI) Exact mass calculated for $\text{C}_{17}\text{H}_{14}\text{F}_2\text{O}_2$ $[\text{M}+\text{H}]^+$:285.1035; found: 285.1036.

2-4. Procedure for alkoxydifluoroacetylation of styrene (1b)

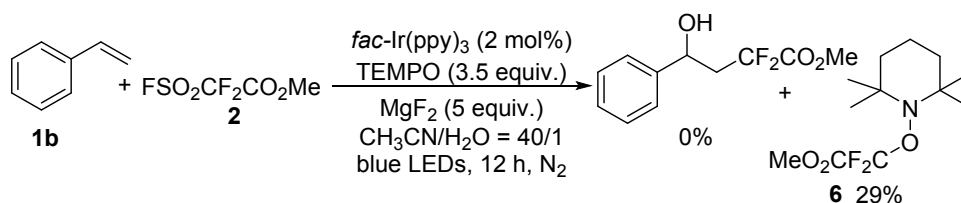


An oven-dried Schleck tube equipped with a stirrer bar was charged with 4.58 mg *fac*-Ir(ppy)₃ (3.5 mol%) which was degassed and refilled with N₂ for 5 times. The alkenes **1b** (0.2 mmol, 1.0 equiv.), Chen's reagent FSO₂CF₂COOMe (115.2 mg, 0.6 mmol, 3 equiv), MgF₂ (62.3 mg, 1.0 mmol, 5 equiv.), MeOH (0.1 mL) and dry CH₃CN (4 mL) were added under N₂. The resulting mixture was irradiated for 12 h under room temperature by 3 W blue LEDs. Quenching the reaction with H₂O and the aqueous layer was extracted with ethyl acetate (EA) twice. The combined organic layer dried by Na₂SO₄, and concentrated in *vacuo* and the residue was purified by chromatography on silica gel to give product **5**, which is identified by ^1H , ^{13}C and ^{19}F NMR.

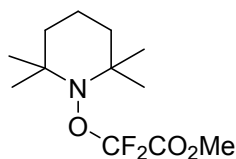


Methyl 2,2-difluoro-4-methoxy-4-phenylbutanoate (5): colorless oil liquid, 22.9 mg (47% yield). ¹H NMR (400 MHz, CHLOROFORM-D) δ 7.40 – 7.28 (m, 5H), 4.35 (d, *J* = 10.5 Hz, 1H), 3.86 (s, 3H), 3.12 (s, 3H), 2.68 (dddd, *J* = 28.4, 14.7, 10.5, 6.3 Hz, 1H), 2.34 – 2.23 (m, 1H). ¹³C NMR (101 MHz, CHLOROFORM-D) δ 164.30 (t, *J* = 35.5 Hz), 139.88, 128.81, 128.41, 126.54, 115.14 (dd, *J* = 251.5, 248.0 Hz), 77.90 (dd, *J* = 10.0, 2.0 Hz), 56.75, 53.18, 43.74 (t, *J* = 23.6 Hz). ¹⁹F NMR (376 MHz, CHLOROFORM-D) δ -105.13 (dddd, *J* = 259.9, 39.0, 20.3, 8.6 Hz). HRMS (ESI) Exact mass calculated for C₁₂H₁₄F₂O₃ [M+H]⁺:245.0984; found: 245.0985.

2-5. Control experiment with TEMPO



An oven-dried Schleck tube equipped with a stirrer bar was charged with 4.58 mg *fac*-Ir(ppy)₃ (3.5 mol%) and TEMPO (109.2 mg, 0.7 mmol, 3.5 equiv.) which was degassed and refilled with N₂ for 5 times. The alkenes **1b** (0.2 mmol, 1.0 equiv.), Chen's reagent (FSO₂CF₂COOMe) (134.4 mg, 0.7 mmol, 3.5 equiv.) and dry DMSO (4 mL) were added under N₂. The resulting mixture was irradiated for 12 h under room temperature by 3 W blue LEDs. Quenching the reaction with H₂O and the aqueous layer was extracted with ethyl acetate (EA) twice. The combined organic layer dried by Na₂SO₄, and concentrated in *vacuo* and the residue was purified by chromatography on silica gel to afford product **6** in 29% isolated yield.

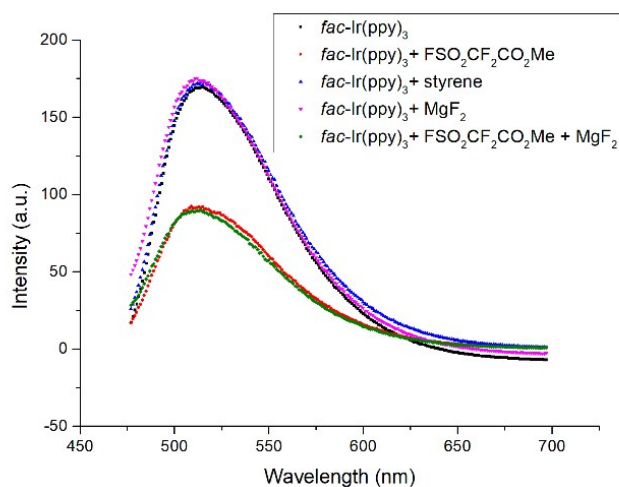


Methyl 2,2-difluoro-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)acetate (6): colorless oil liquid, 54 mg (29%). ¹H NMR (400 MHz, CHLOROFORM-D) δ 3.91 (s,

3H), 1.57 (dd, $J = 11.3, 8.4$ Hz, 5H), 1.39 – 1.34 (m, 1H), 1.18 (d, $J = 8.2$ Hz, 12H). ^{13}C NMR (101 MHz, CHLOROFORM-D) δ 161.37 (t, $J = 42.8$ Hz), 115.66 (t, $J = 271.2$ Hz), 61.54, 53.61, 40.33, 33.57 (t, $J = 4.2$ Hz), 20.91, 17.03. ^{19}F NMR (376 MHz, CHLOROFORM-D) δ -72.94 (s).

2-6. Fluorescence quenching experiments

Fluorescence quenching experiments were measured on an Ahilent Technologies Cary Eclipse Fluorescence Spectrophotometer. The complex *fac*-Ir(ppy)₃ was excited at 375 nm and the emission spectrum $\lambda_{\text{max}} = 518$ nm was recorded. Gradient dilution to get 3.0×10^{-5} M *fac*-Ir(ppy)₃ solution in CH₃CN, 0.06 M FSO₂CF₂COOMe (**2**) solution in CH₃CN and 0.06 M styrene (**1b**) solution in CH₃CN. 2.0 mL 3.0×10^{-5} M *fac*-Ir(ppy)₃ solution in CH₃CN and a stirrer bar were added into the 2.0 mL quartz cuvette covered with Teflon cap. 1.0×10^{-2} mL 0.06 M FSO₂CF₂COOMe solution, 0.06 M styrene (**1b**) solution, 1.0 mg MgF₂ were added, separately. Then the emission spectrum of the



solution was collected at each addition **Fig S1**.

Fig S1. Fluorescence quenching experiment between photocatalyst and substrate

2.0 mL 3.0×10^{-5} M *fac*-Ir(ppy)₃ solution in CH₃CN and a stirrer bar were added into the 2.0 mL quartz cuvette covered with Teflon cap. Adding 2.0×10^{-3} mL 0.06 M FSO₂CF₂COOMe solution each time until 2.0×10^{-2} mL solution was added. Then the emission spectrum of the solution was collected at each addition **Fig S2**.

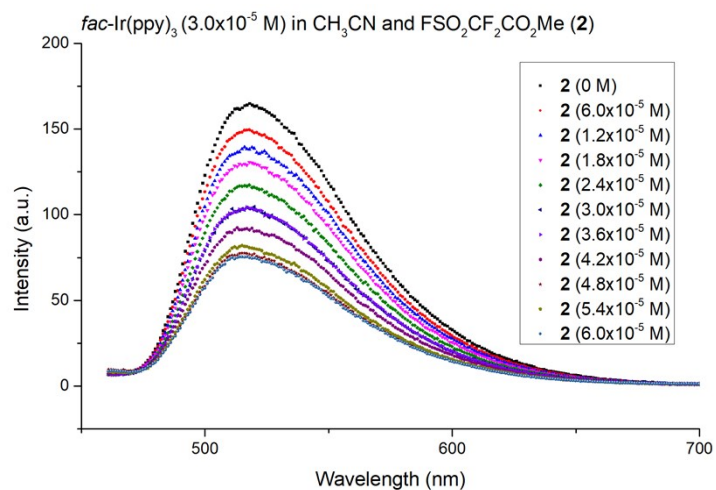
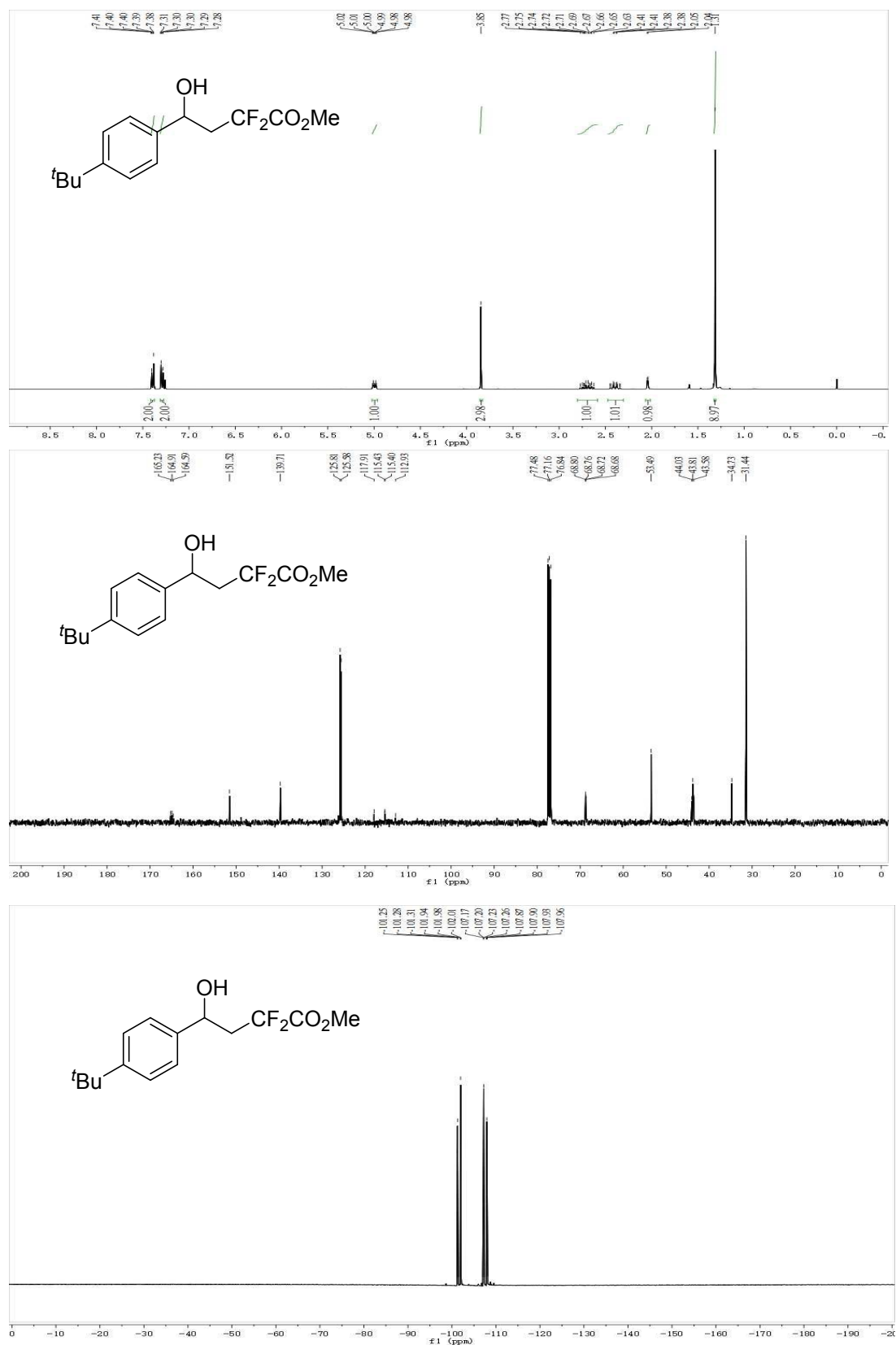


Fig S2. Fluorescence quenching experiment between photocatalyst and FSO₂CF₂CO₂Me

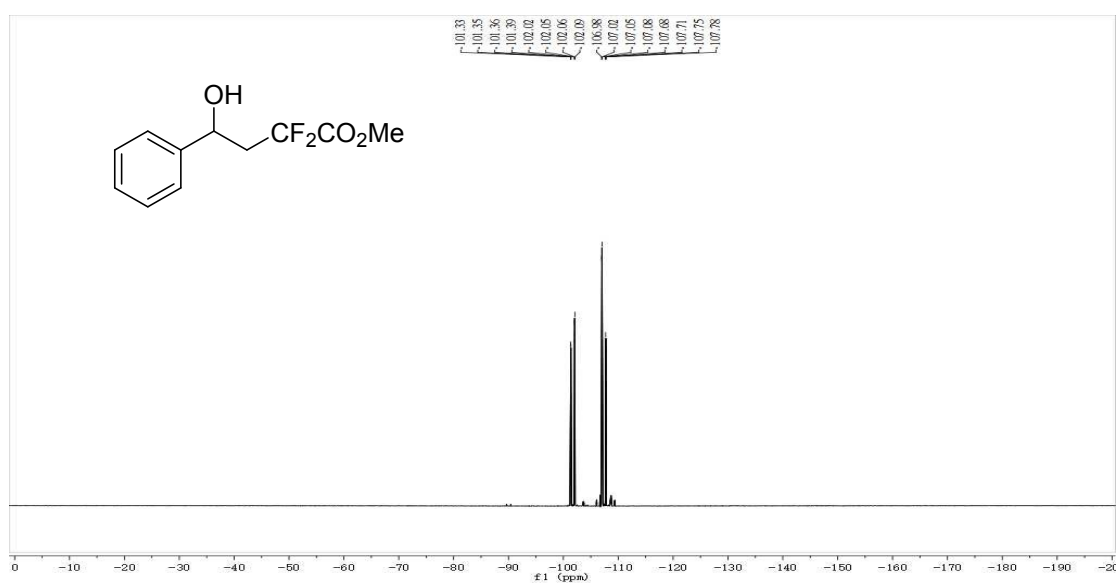
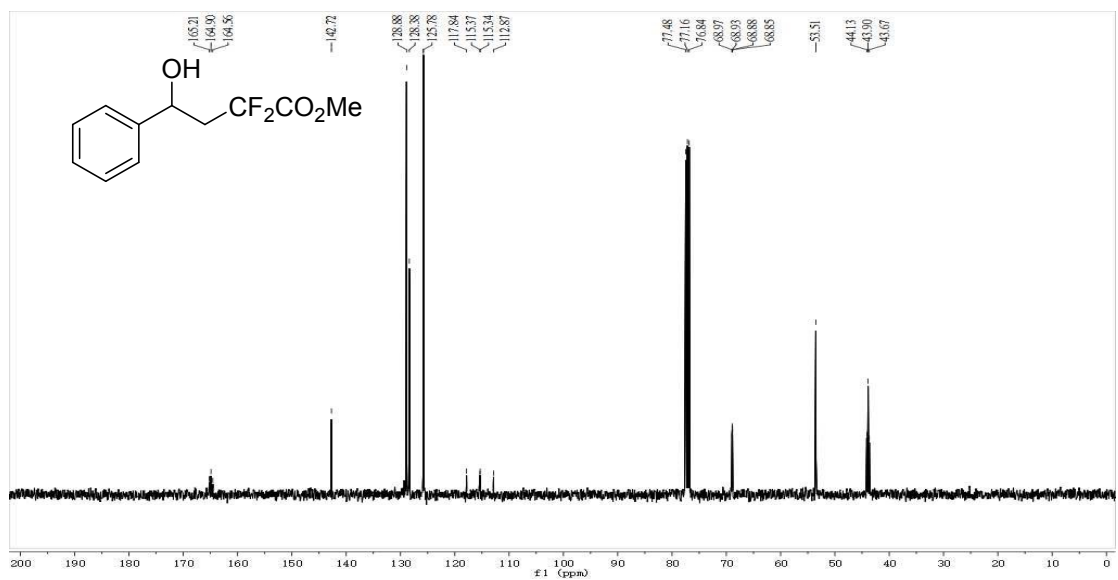
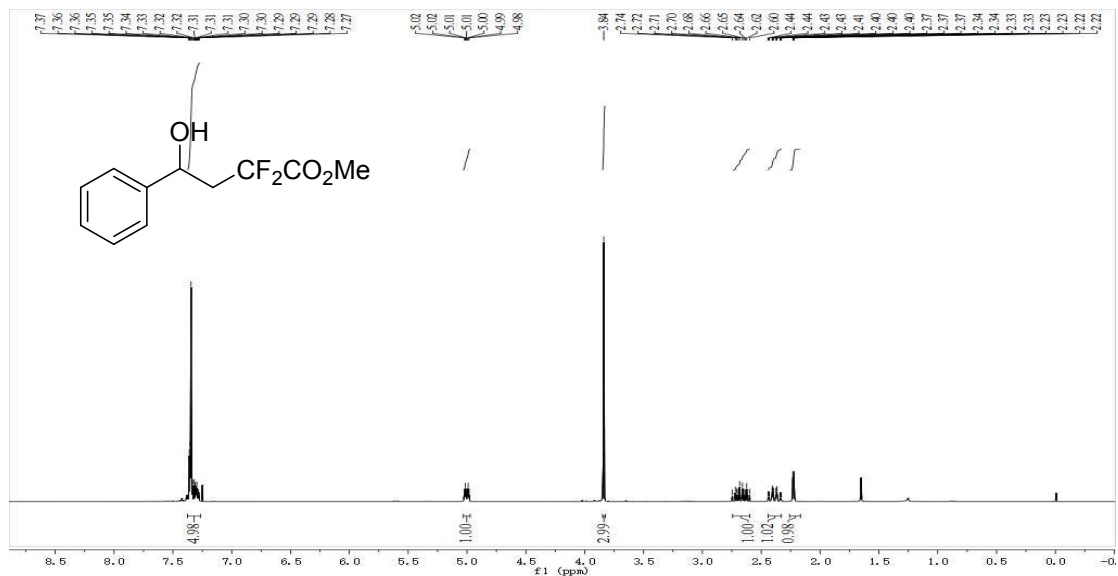
3. Reference

- [1] Gallagher, K. J.; Webster, R. L., *Chem. Commun.* **2014**, *50*, 12109.
- [2] Nojima, M.; Ohta, Y.; Yokozawa, T., *J. Am. Chem. Soc.* **2015**, *137*, 5682.
- [3] C.-Y. Huang, A. G. Doyle, *J. Am. Chem. Soc.* **2012**, *134*, 9541

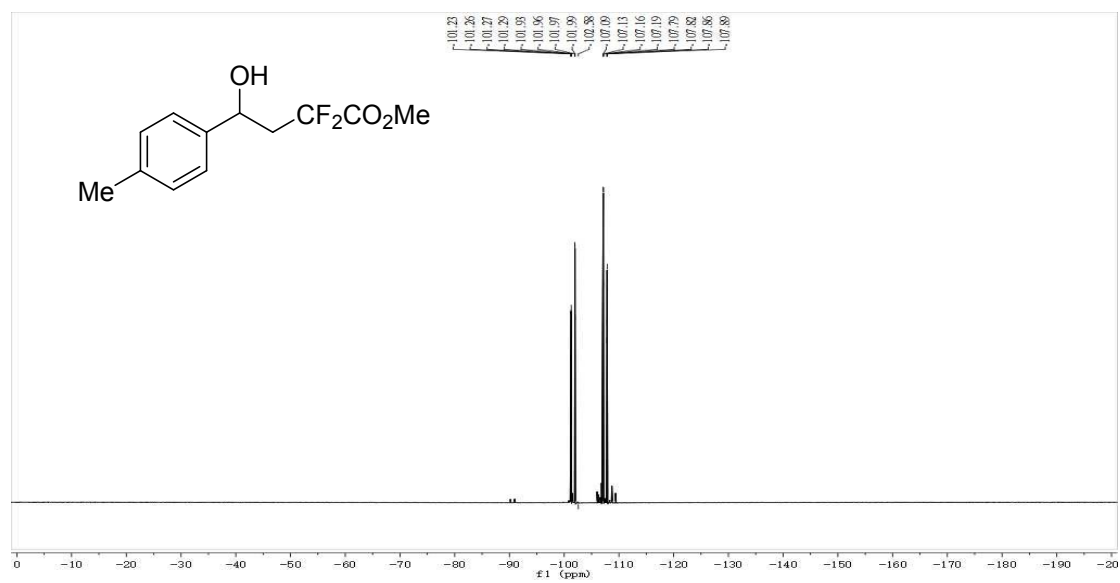
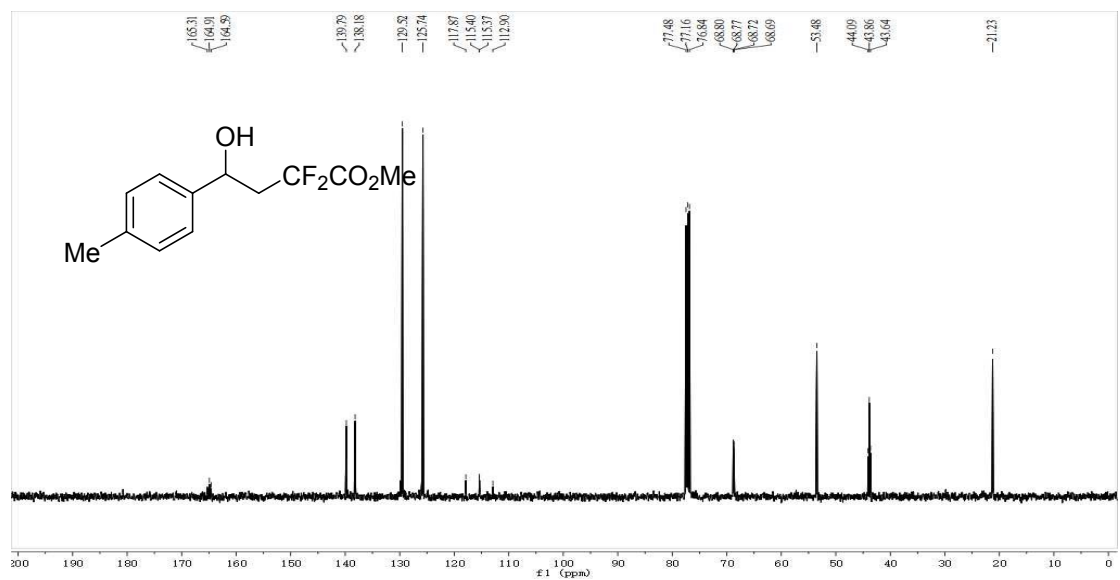
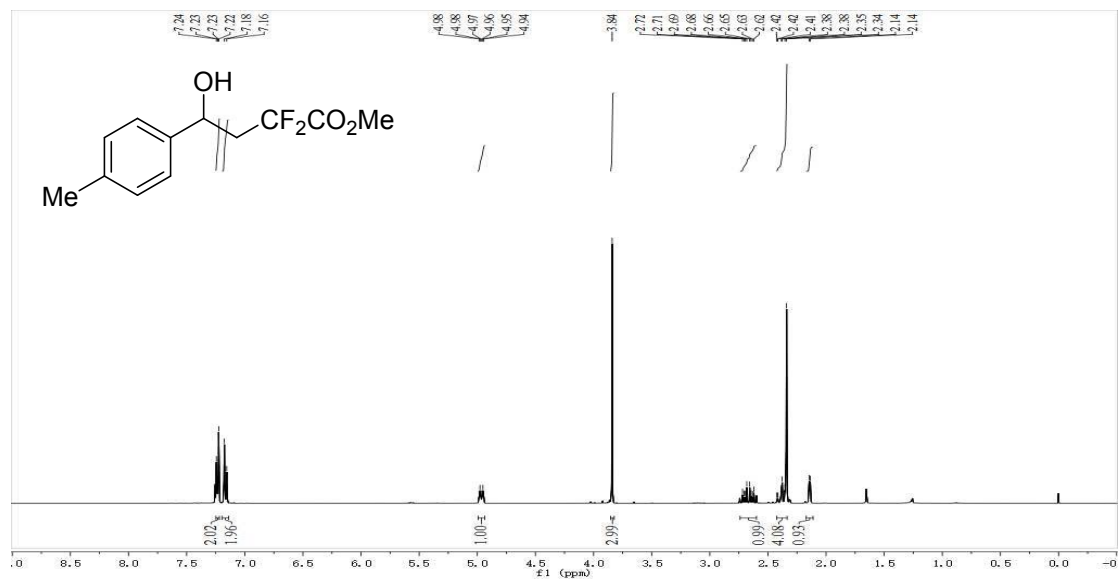
4. Copies of ^1H , ^{13}C , and ^{19}F NMR Spectra



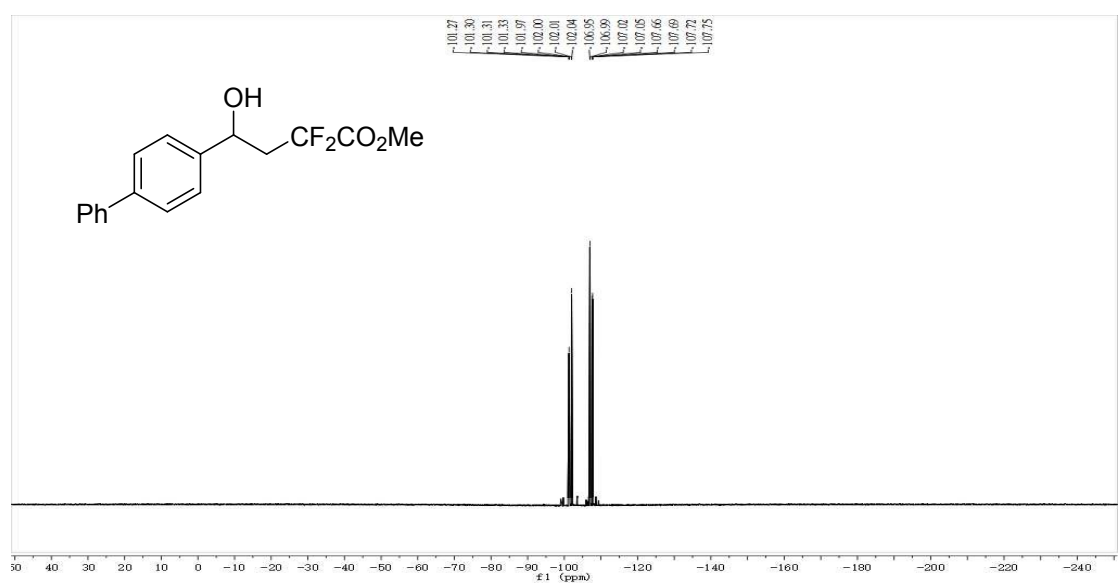
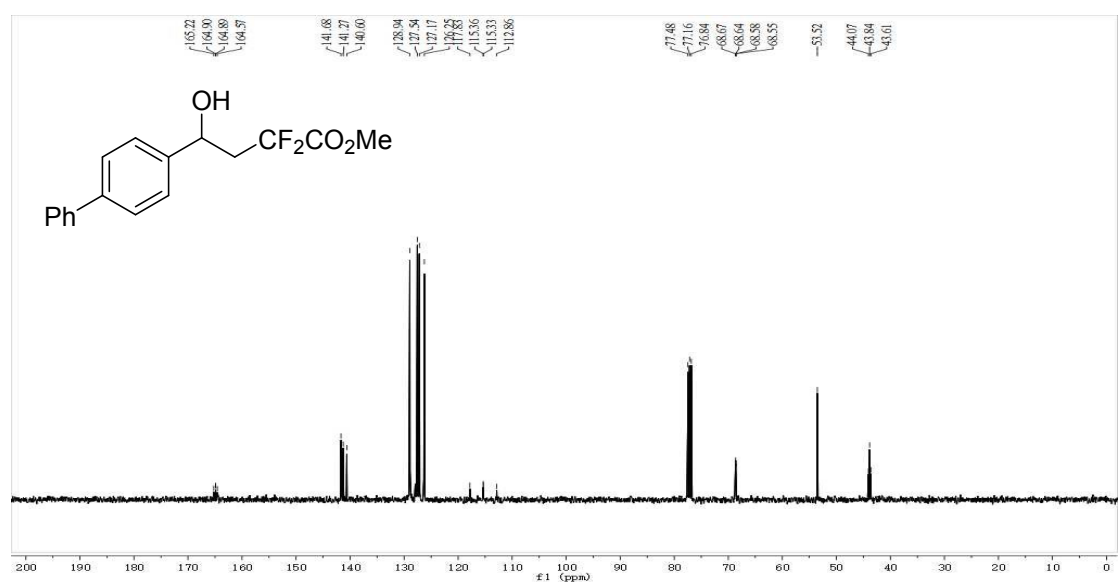
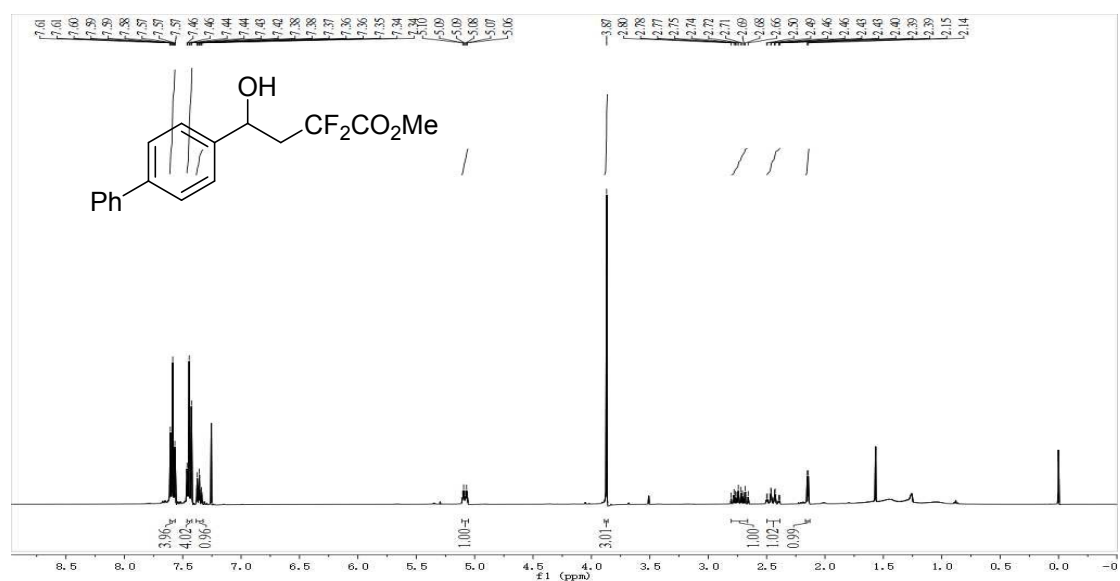
^1H NMR, ^{13}C NMR and ^{19}F NMR of **3a**



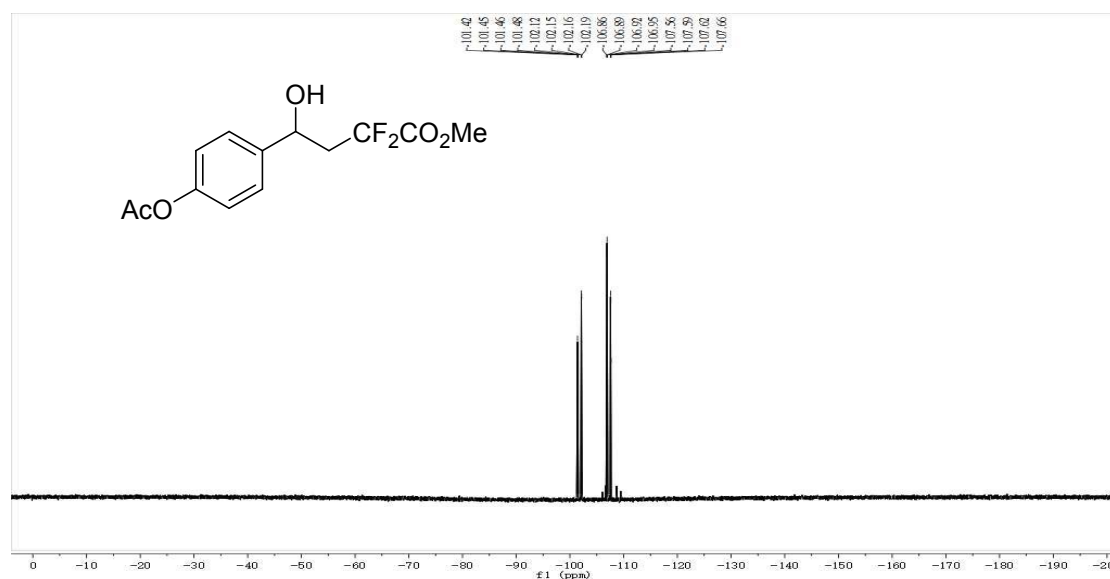
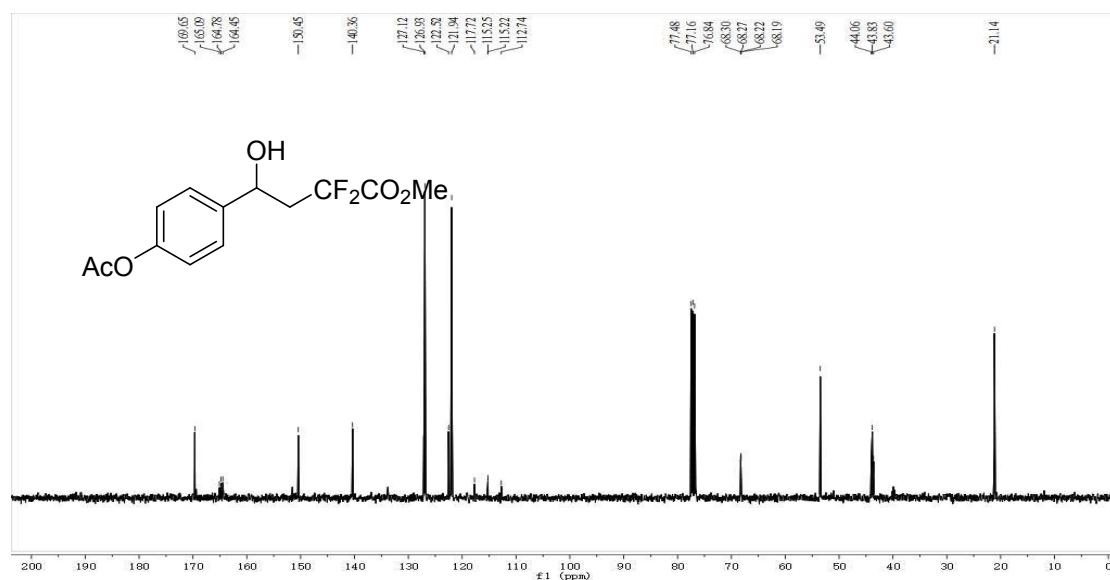
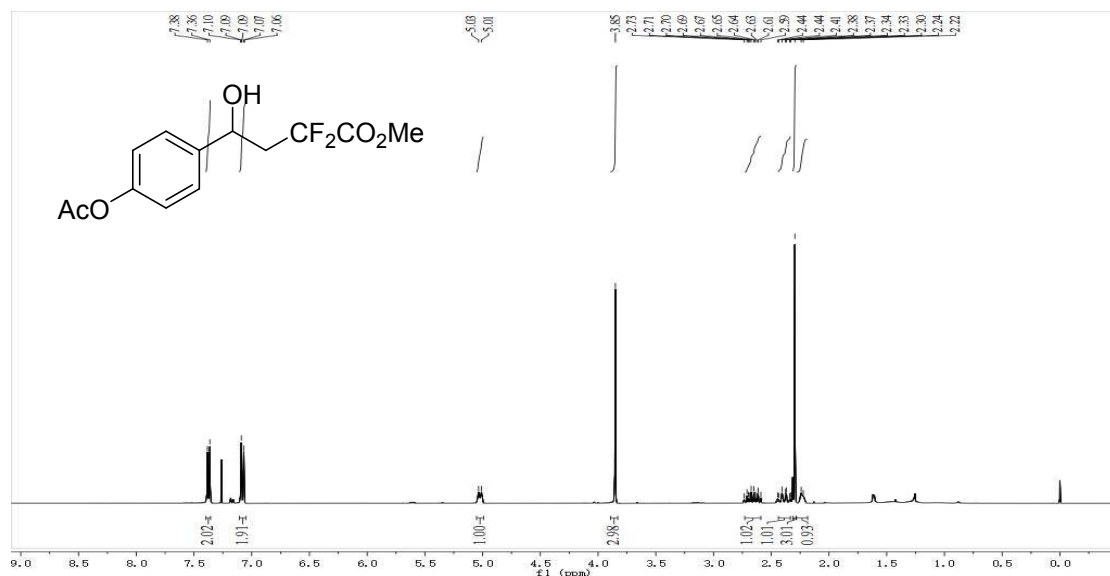
¹H NMR, ¹³C NMR and ¹⁹F NMR of **3b**



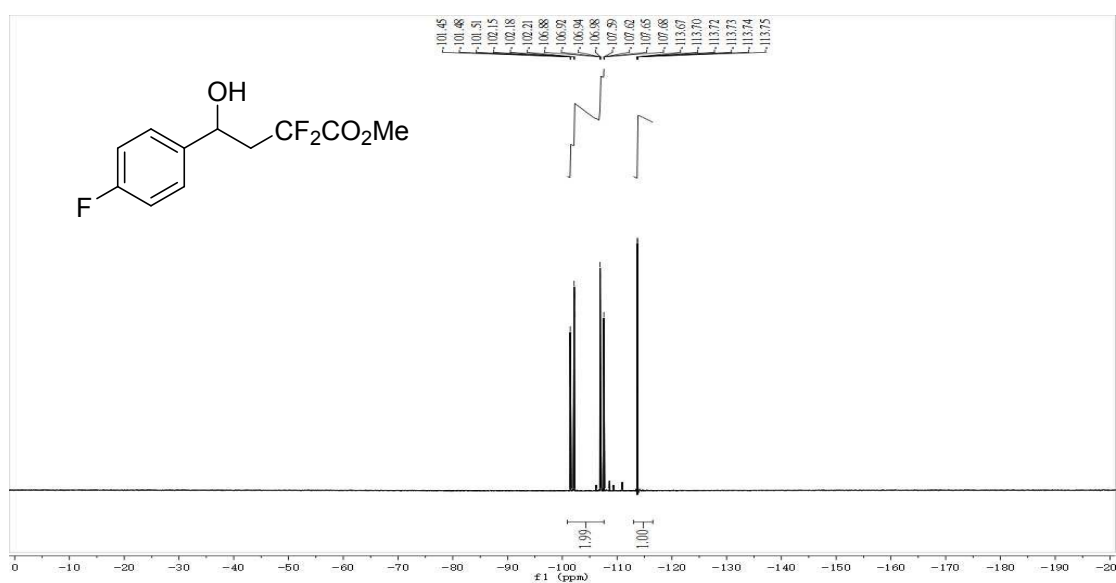
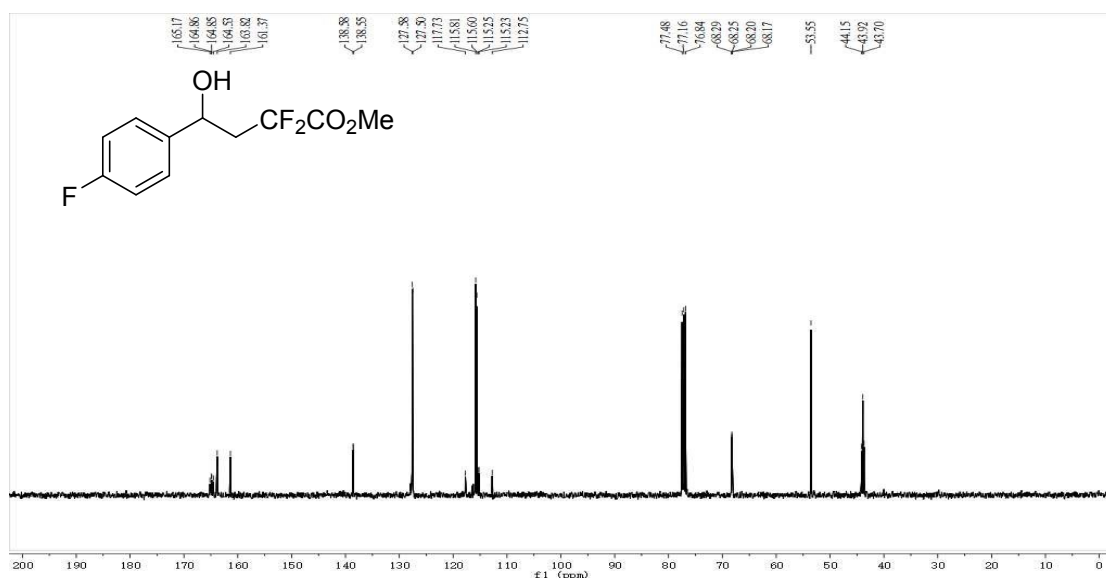
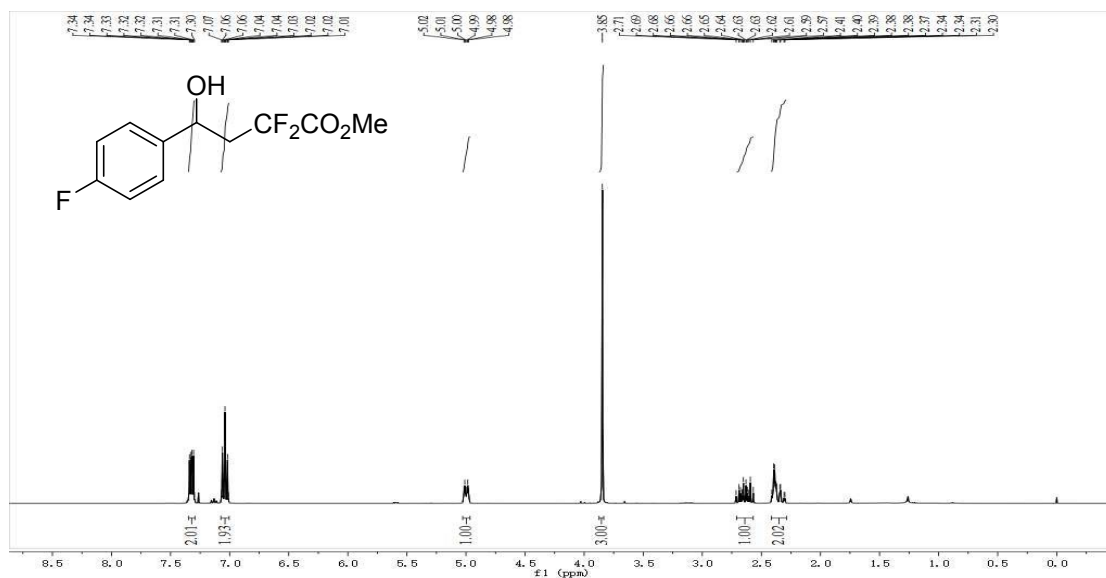
¹H NMR, ¹³C NMR and ¹⁹F NMR of 3c



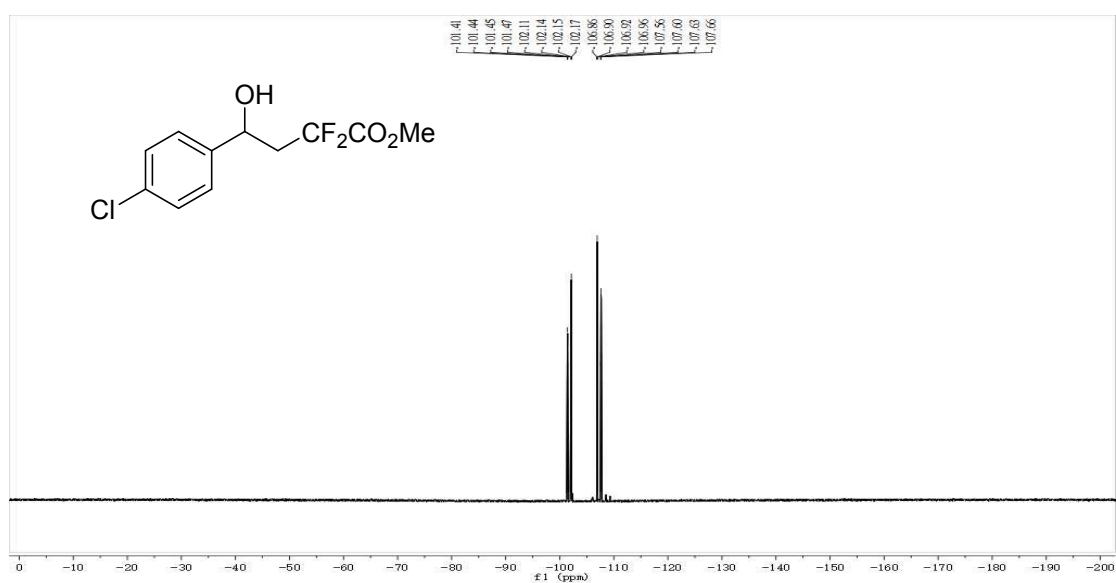
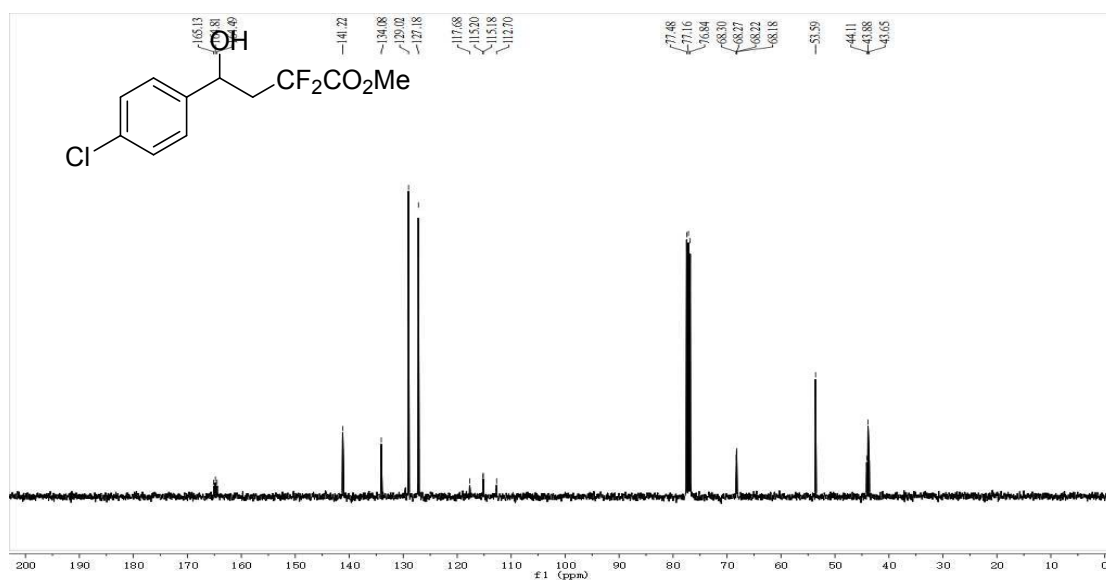
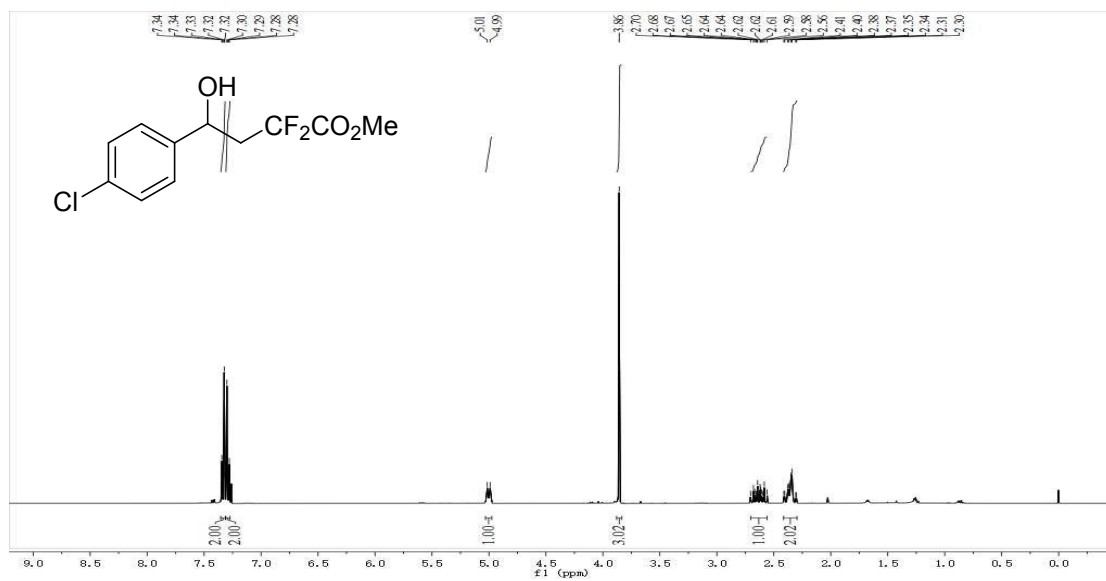
¹H NMR, ¹³C NMR and ¹⁹F NMR of 3d



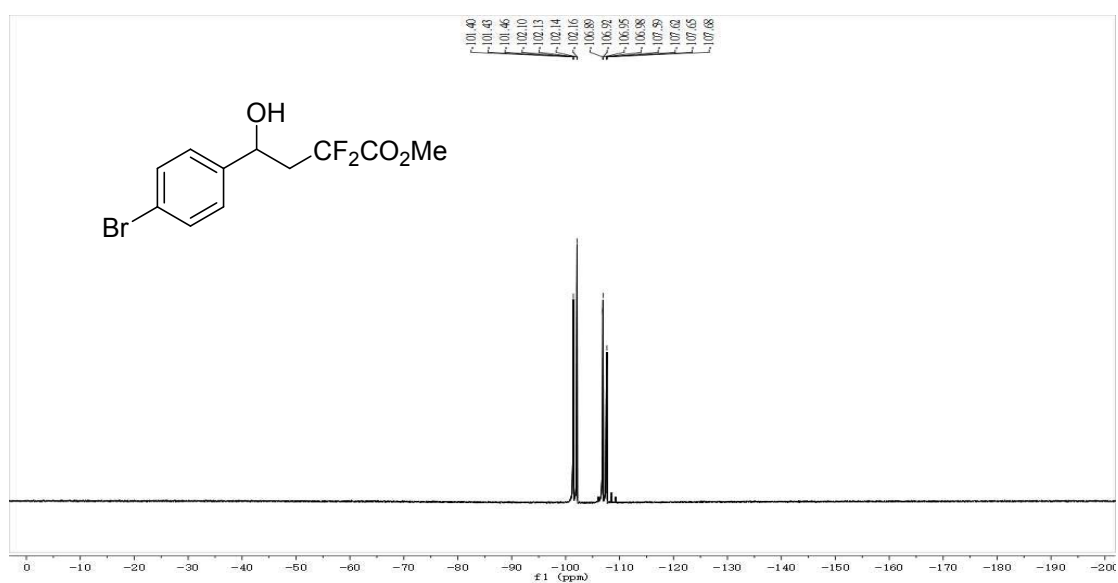
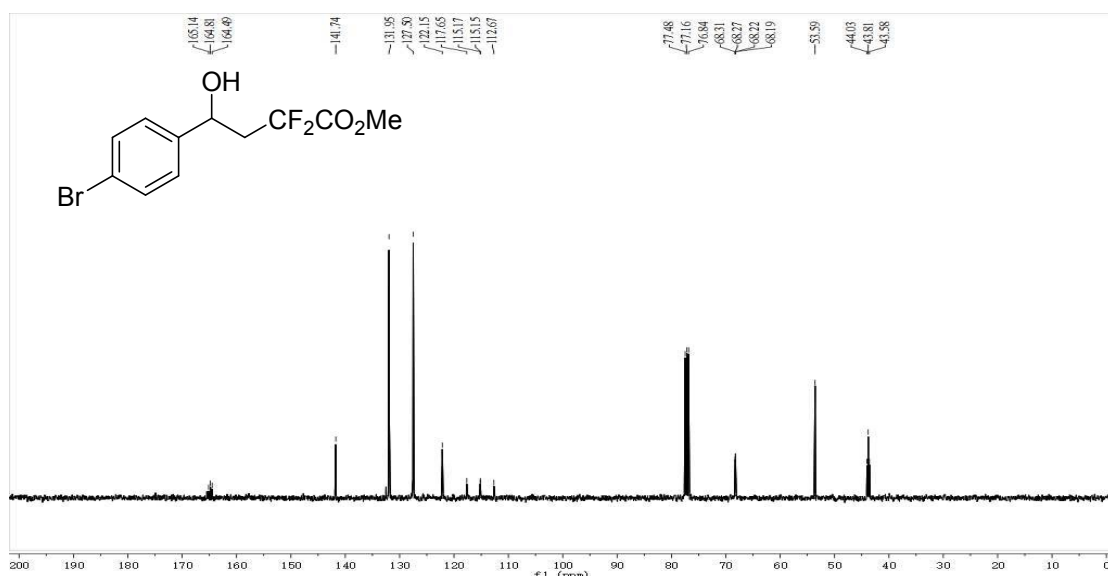
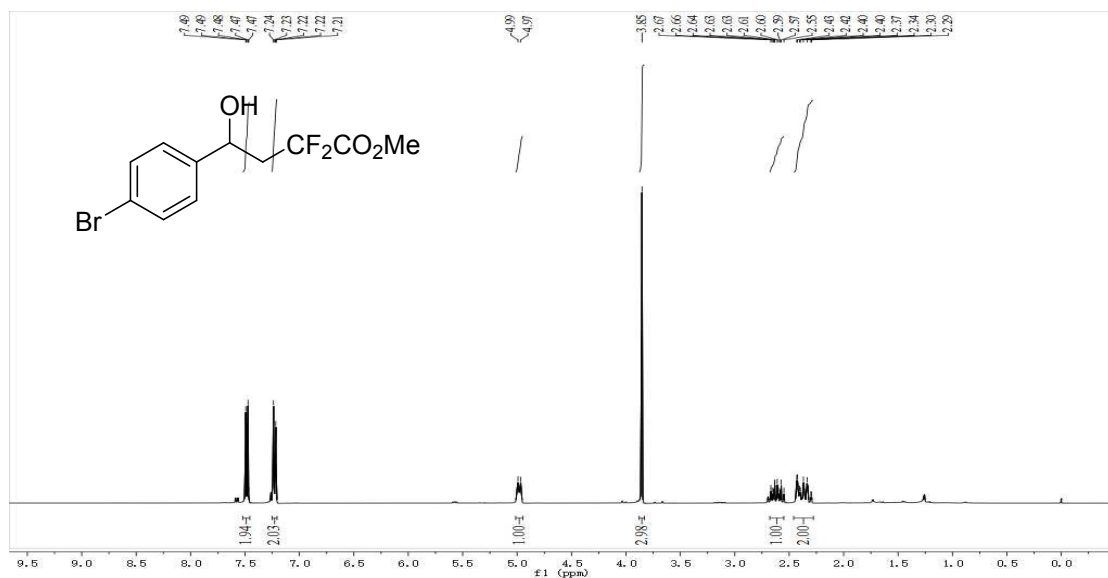
¹H NMR, ¹³C NMR and ¹⁹F NMR of **3e**



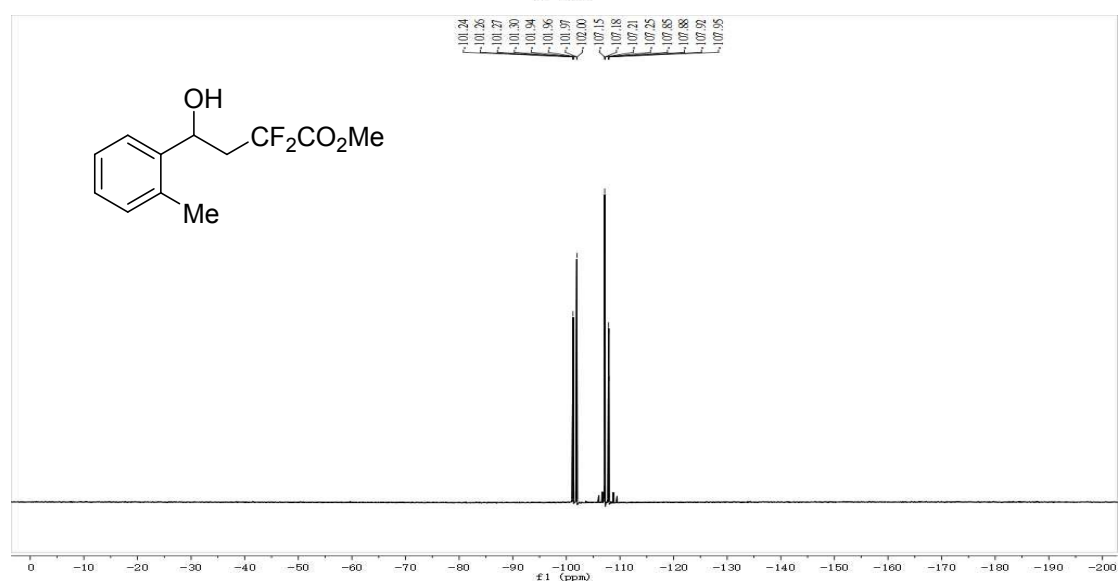
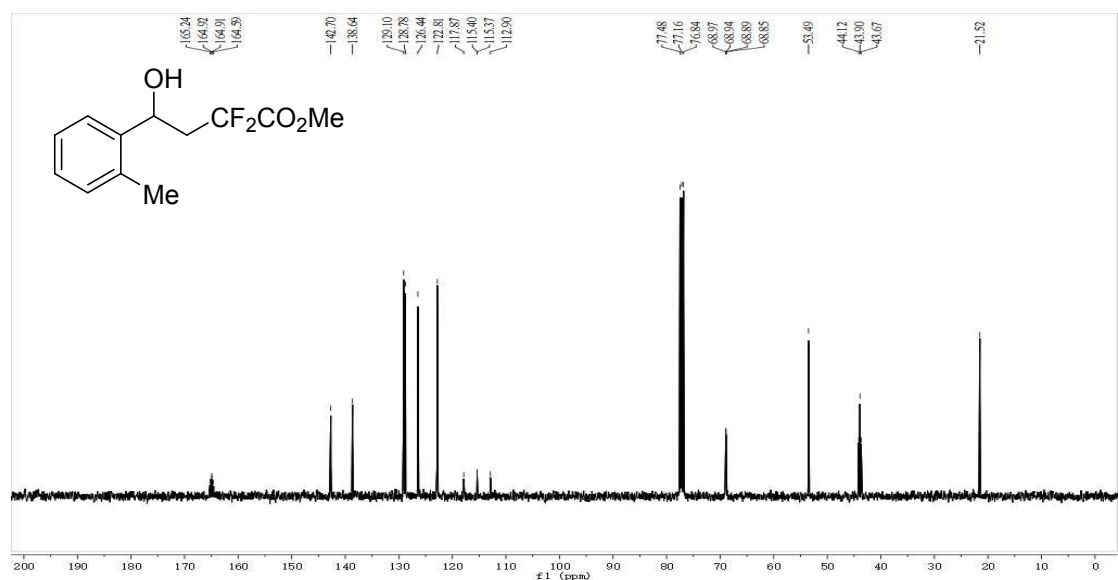
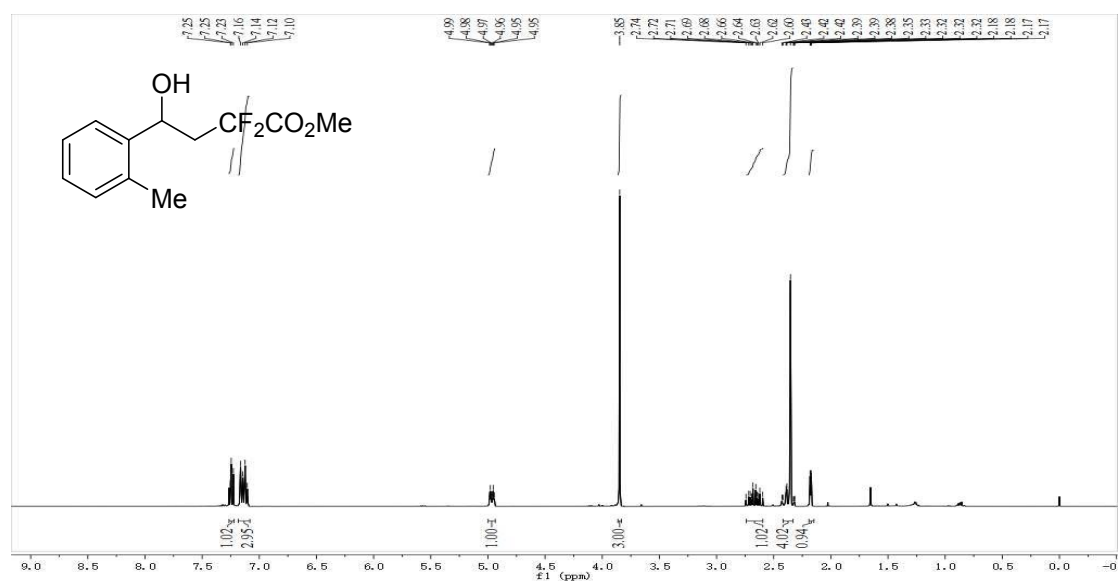
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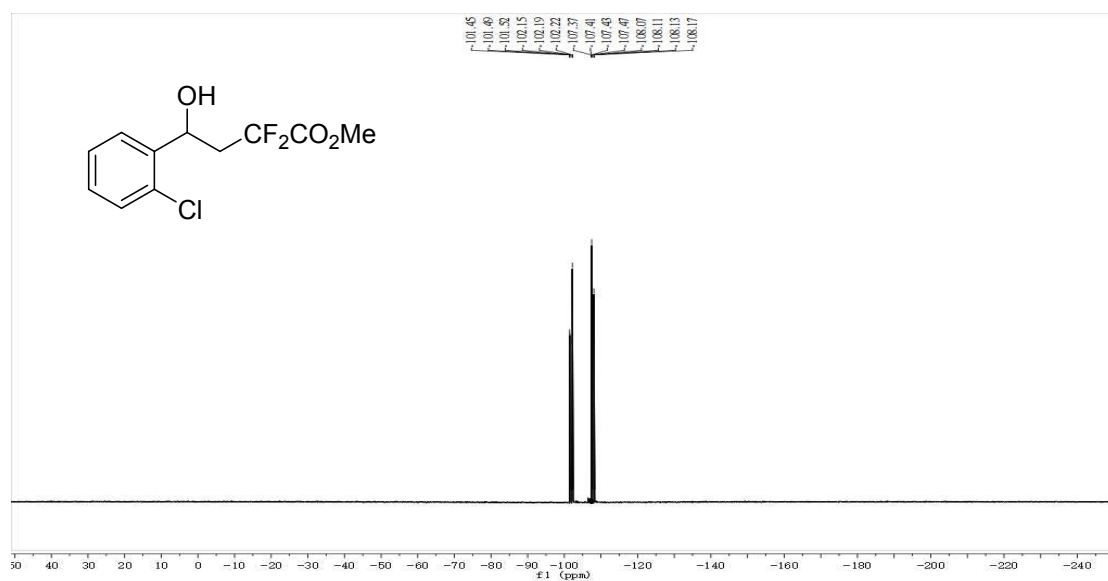
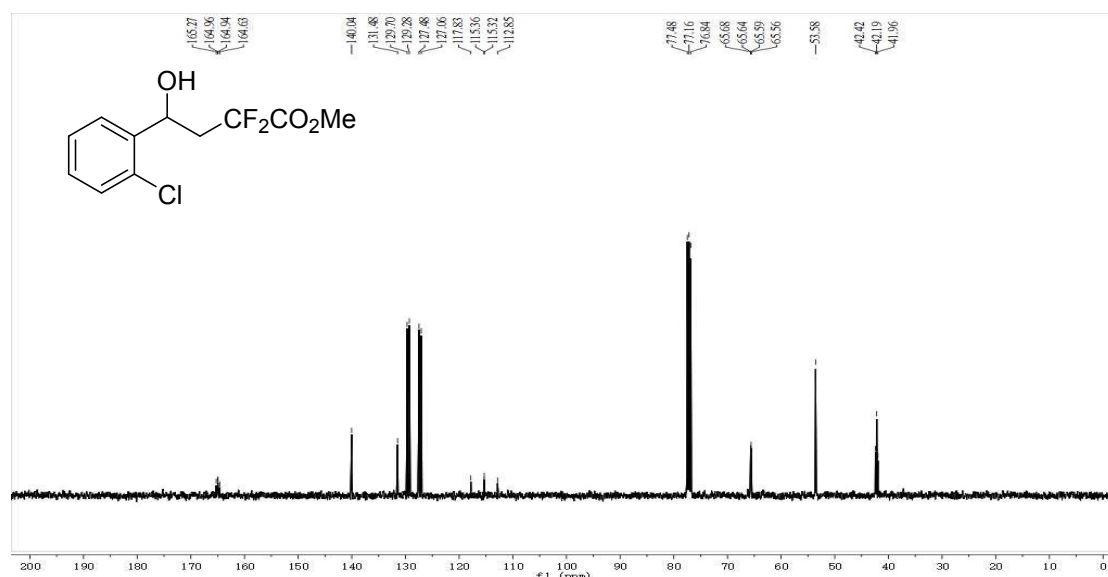
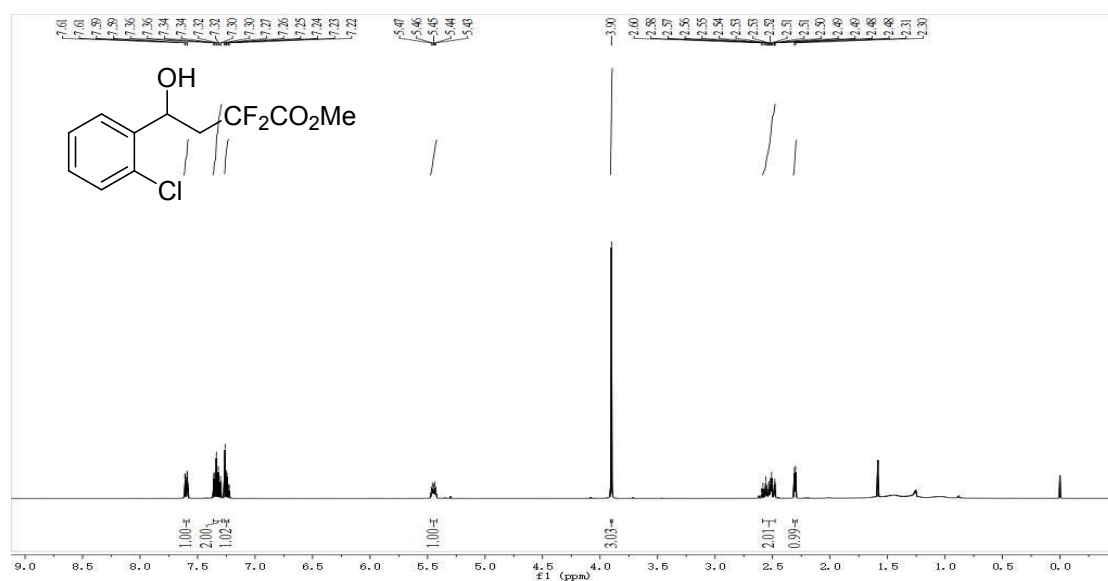
¹H NMR, ¹³C NMR and ¹⁹F NMR of 3g



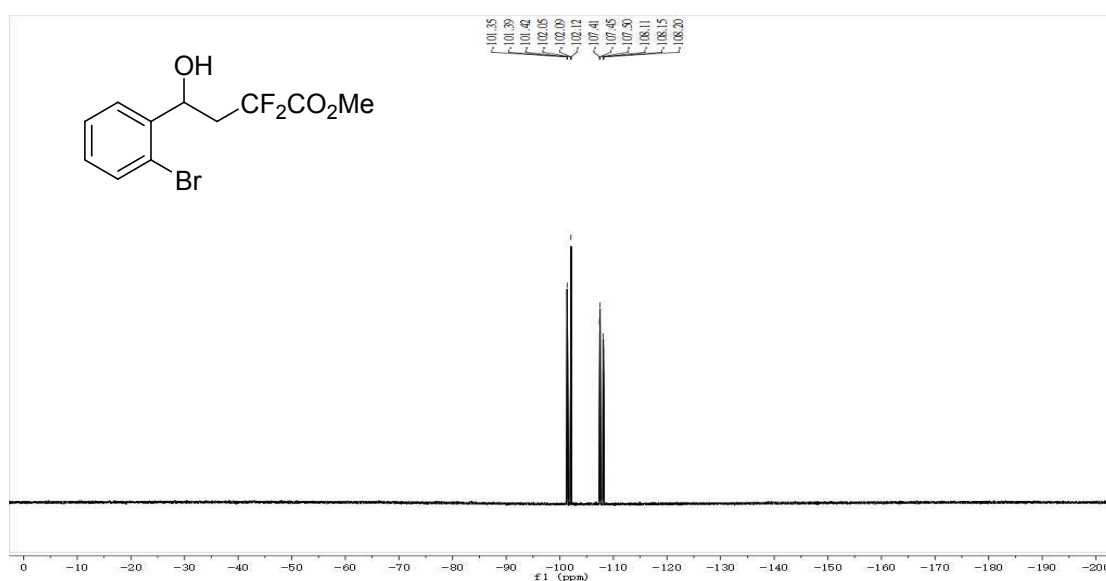
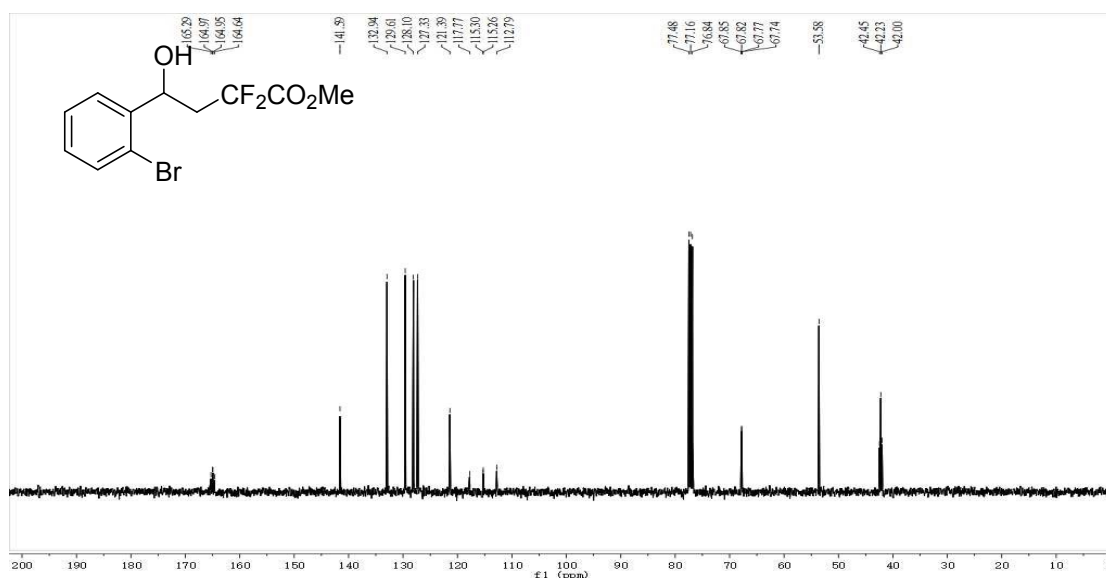
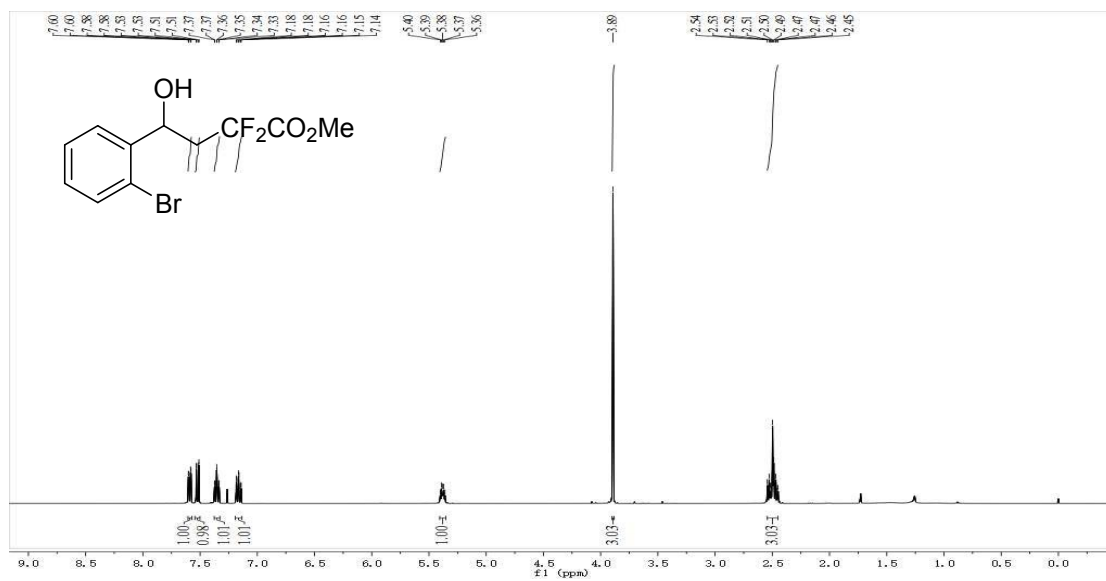
¹H NMR, ¹³C NMR and ¹⁹F NMR of 3h



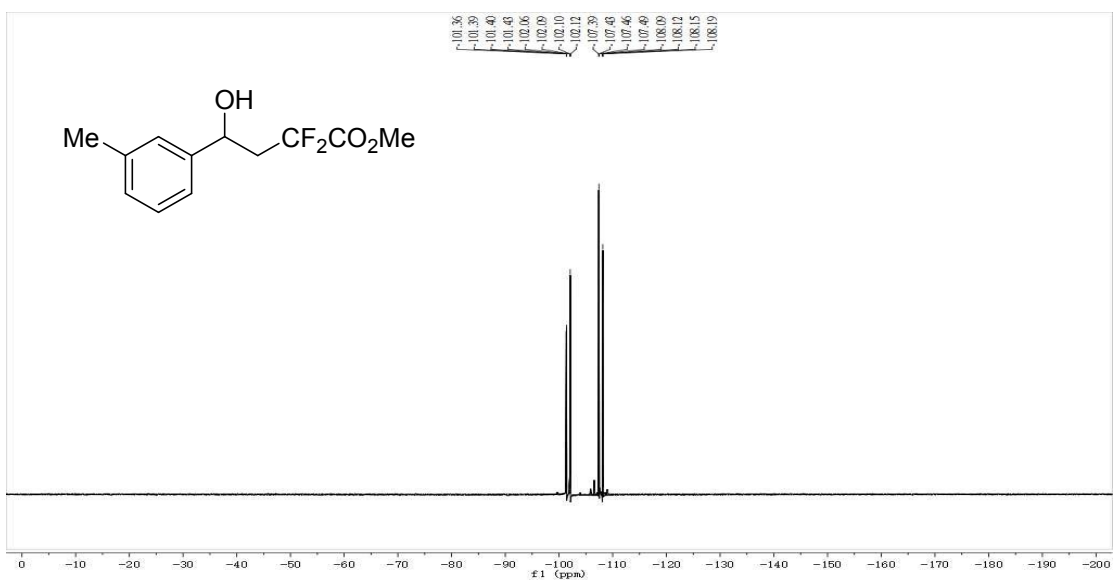
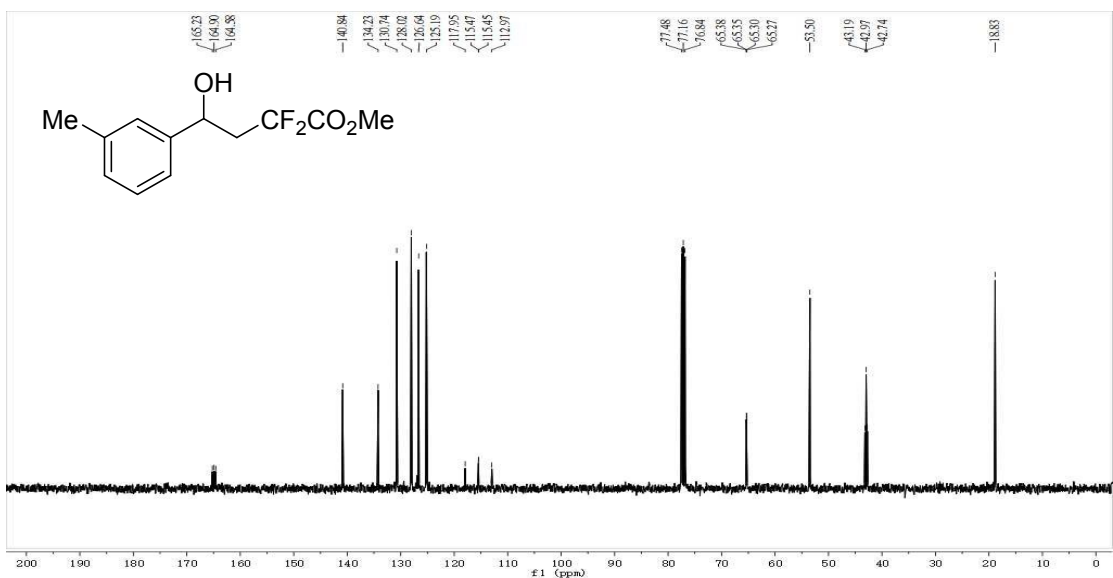
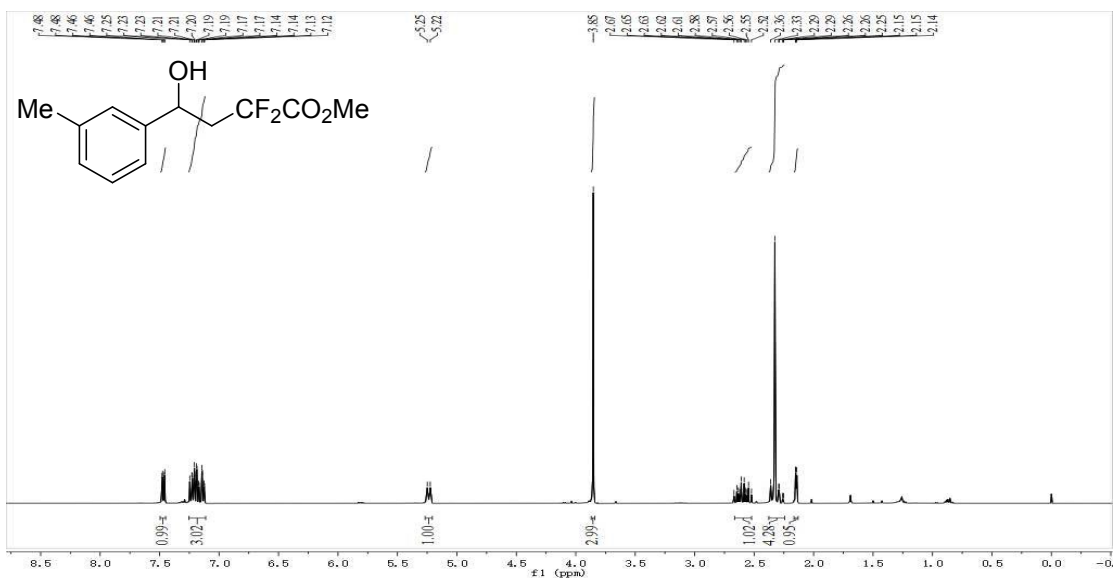
¹H NMR, ¹³C NMR and ¹⁹F NMR of **3i**



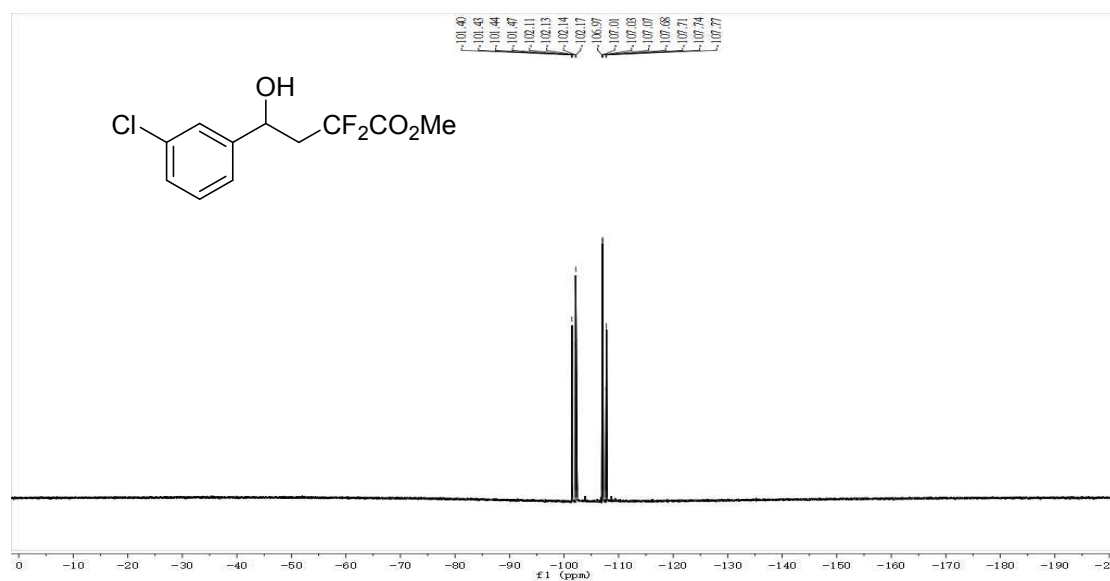
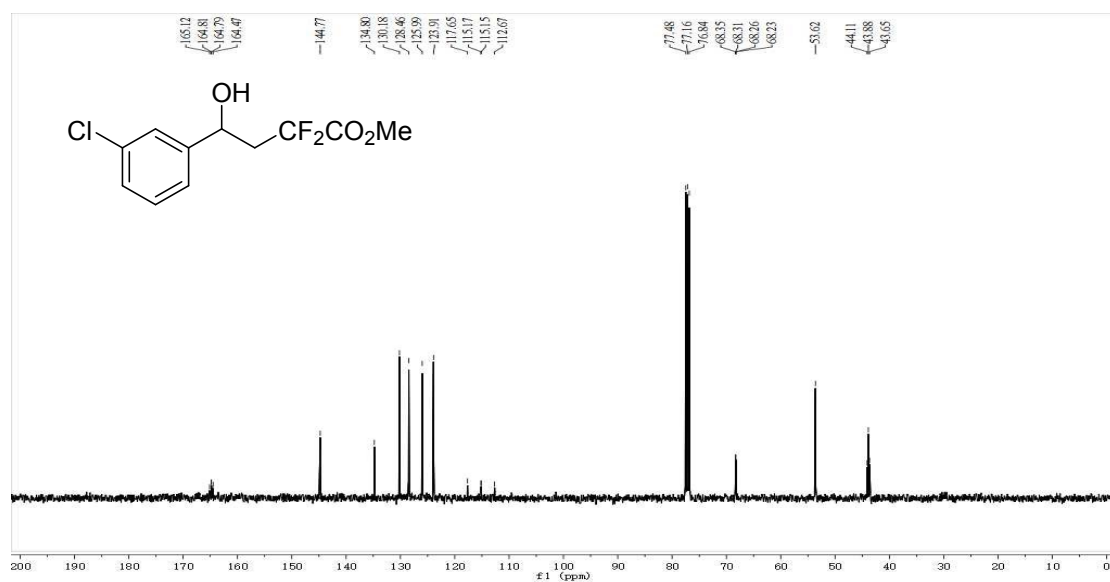
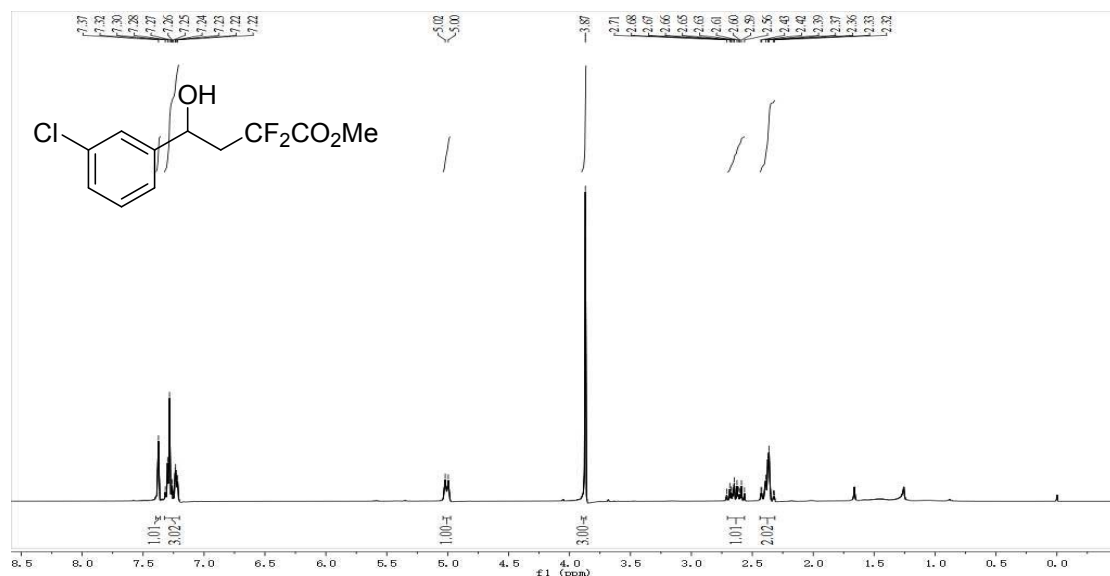
¹H NMR, ¹³C NMR and ¹⁹F NMR of **3j**



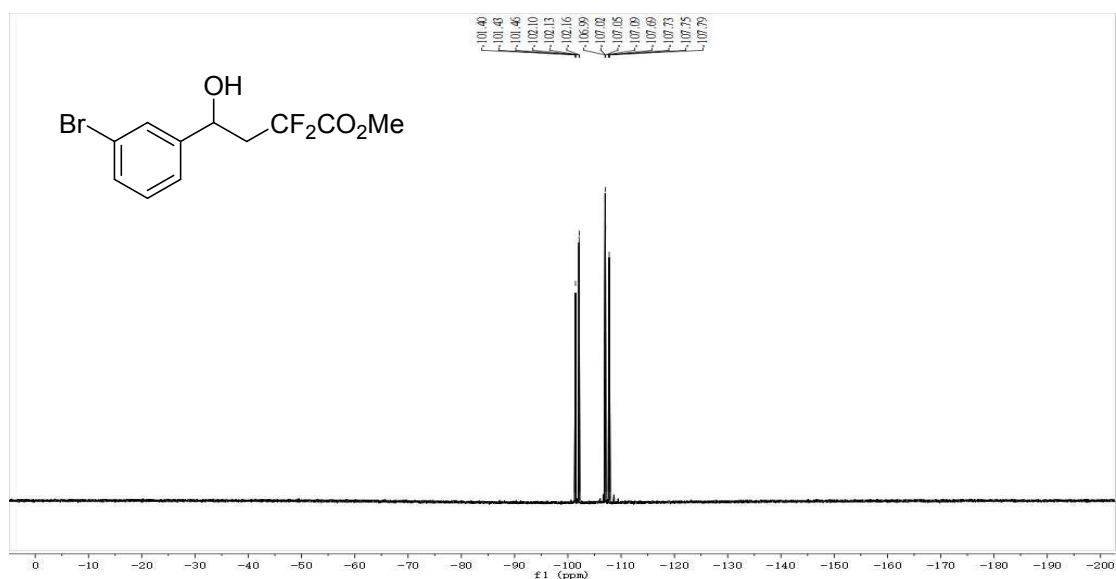
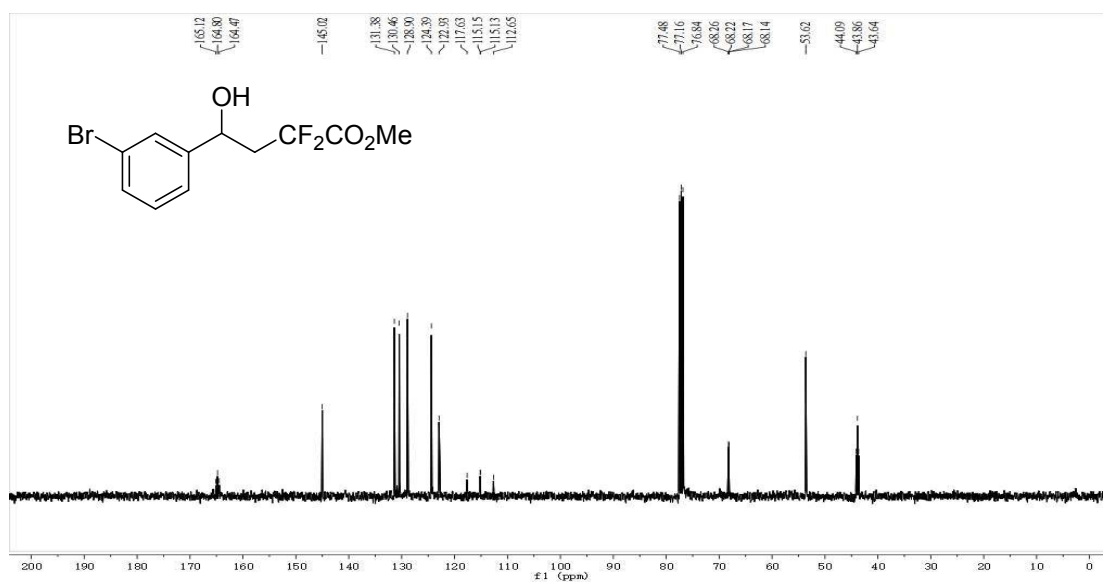
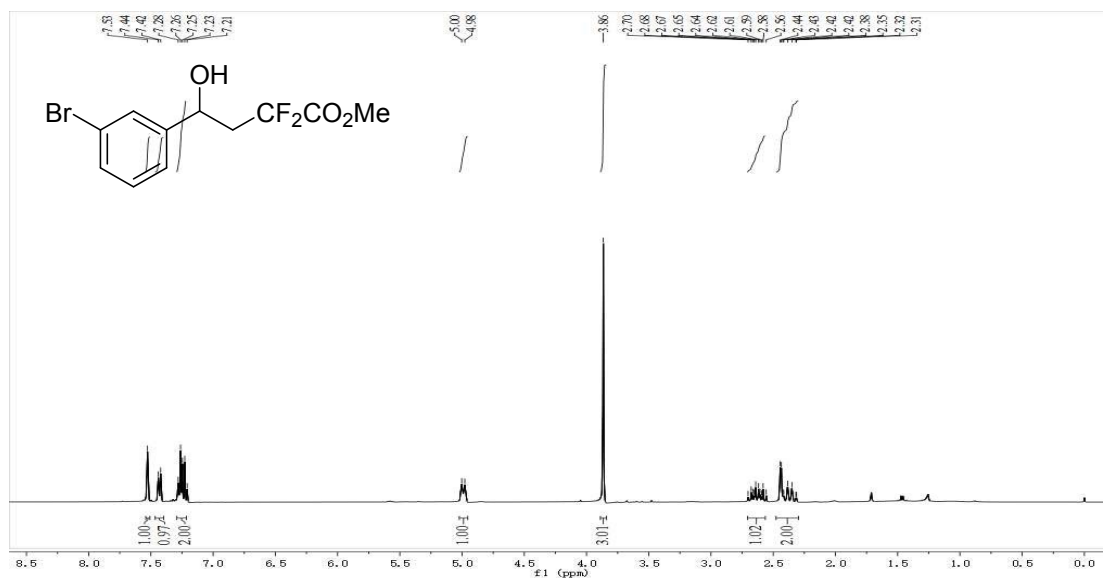
¹H NMR, ¹³C NMR and ¹⁹F NMR of 3k



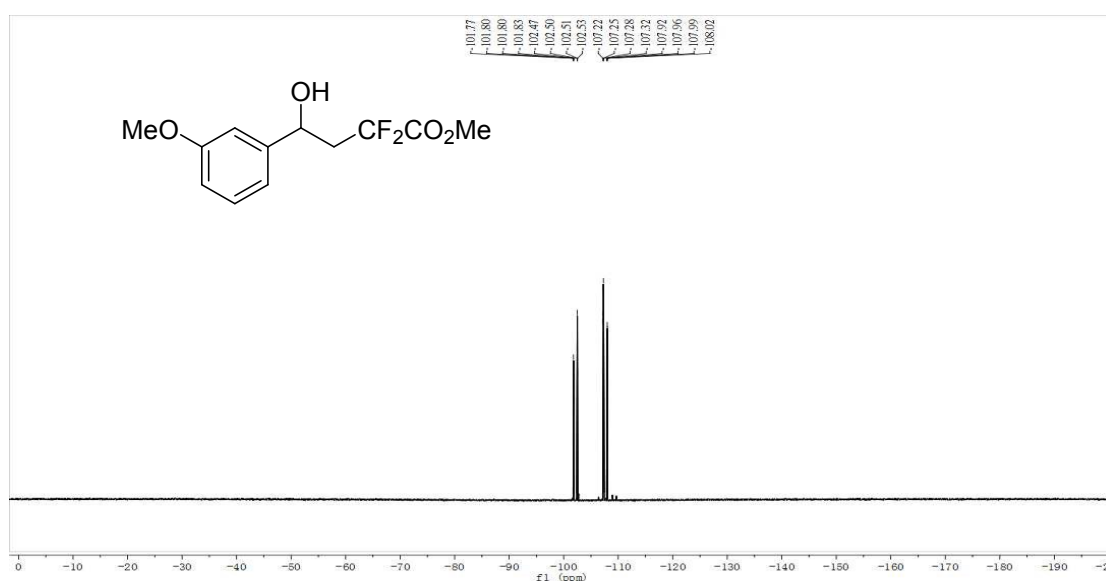
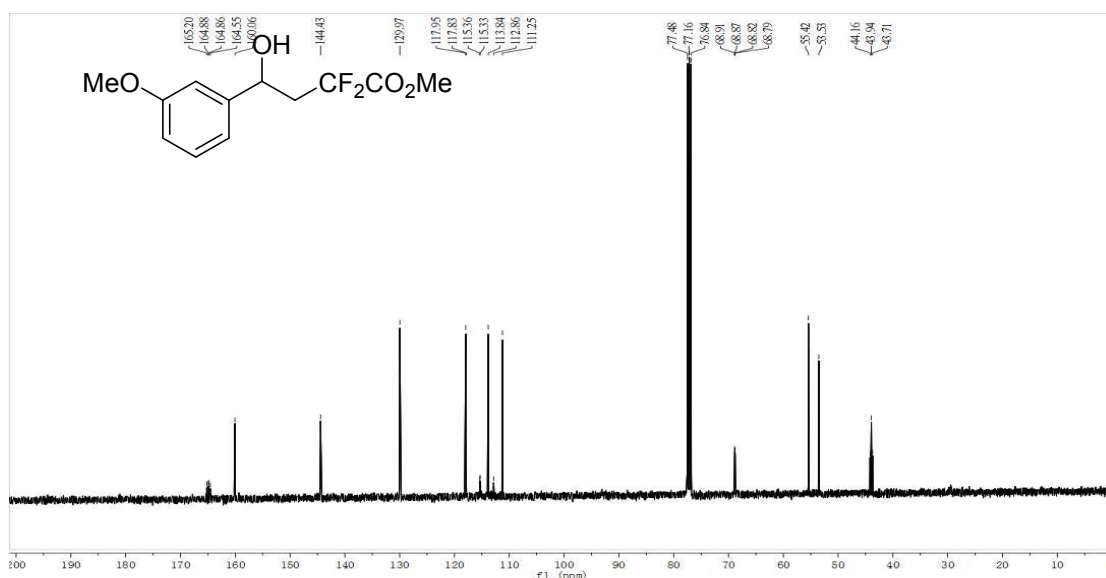
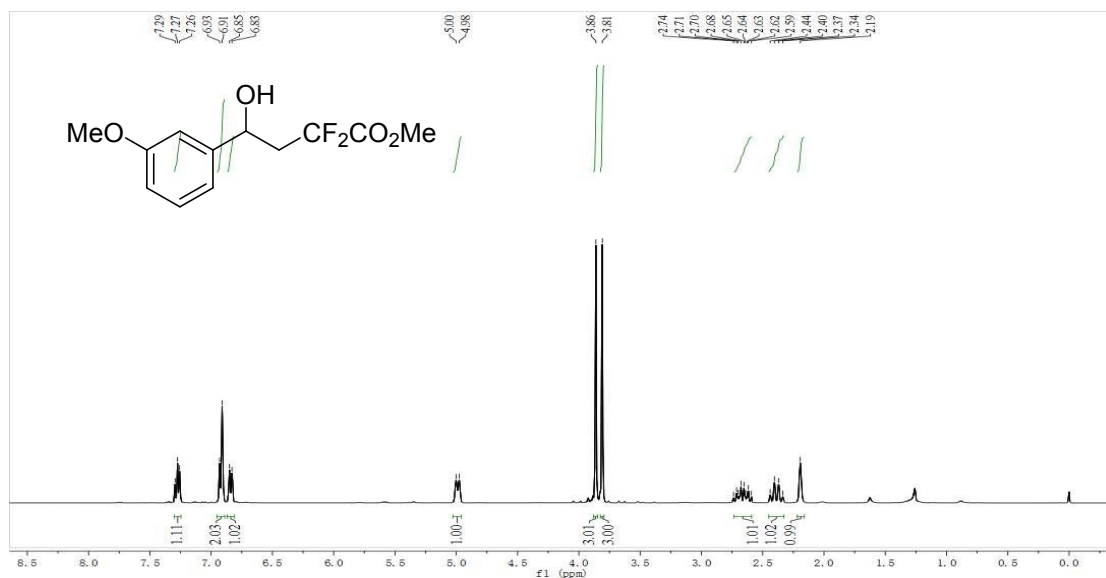
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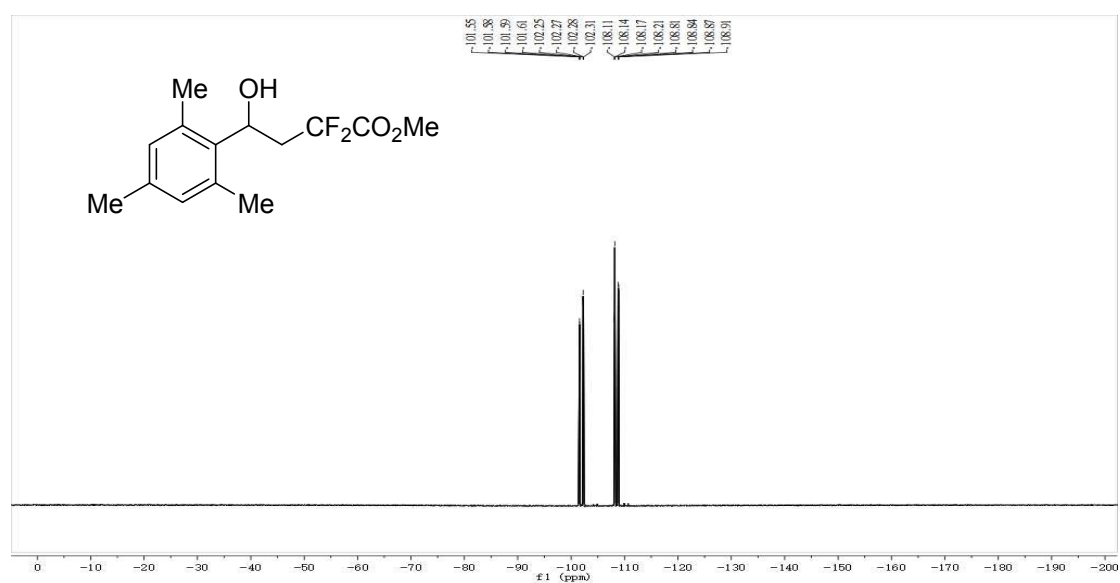
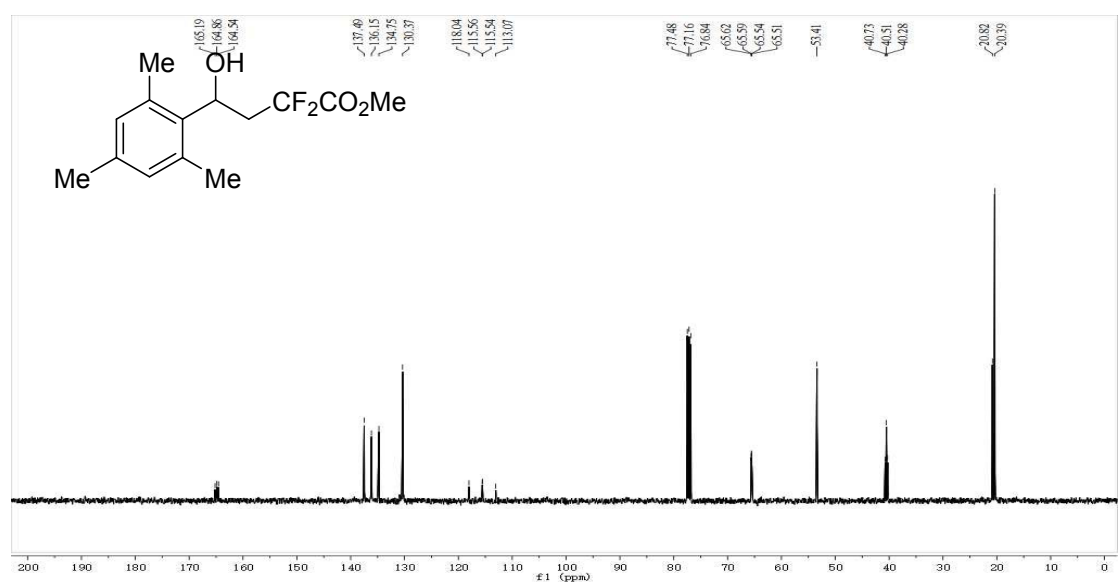
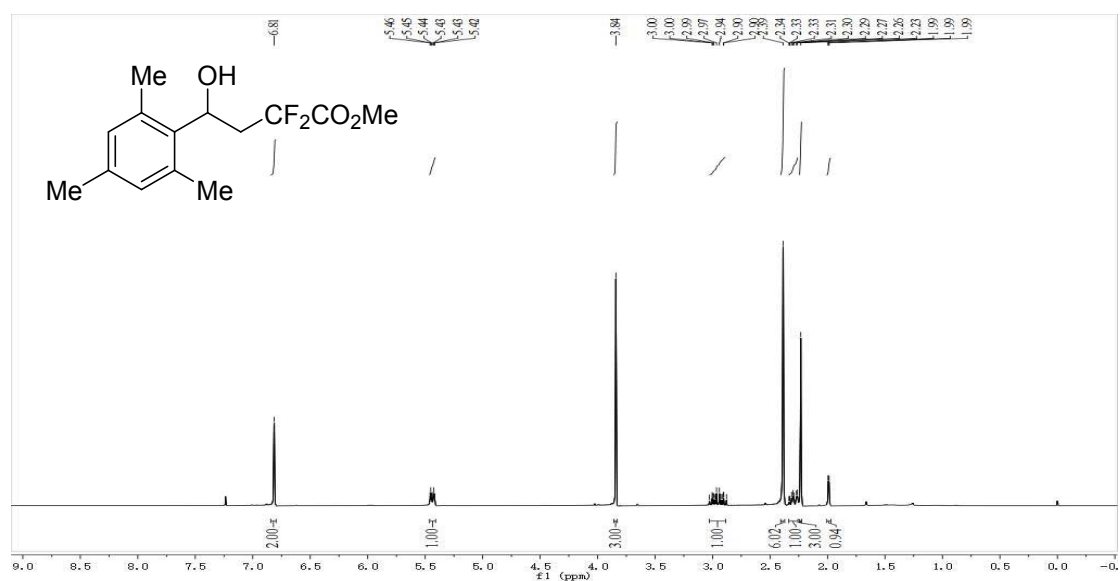
¹H NMR, ¹³C NMR and ¹⁹F NMR of **3m**



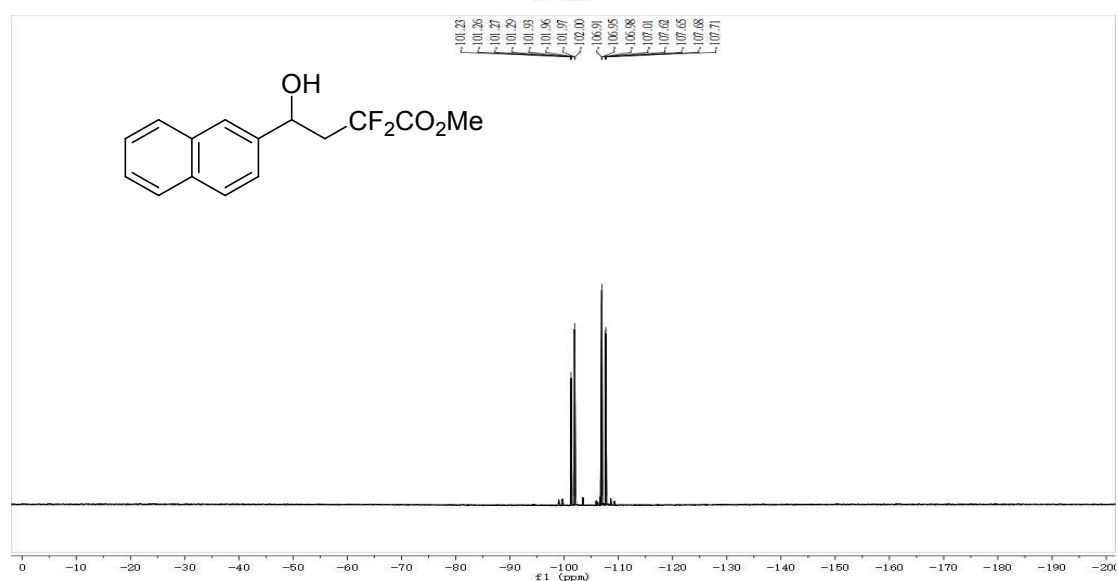
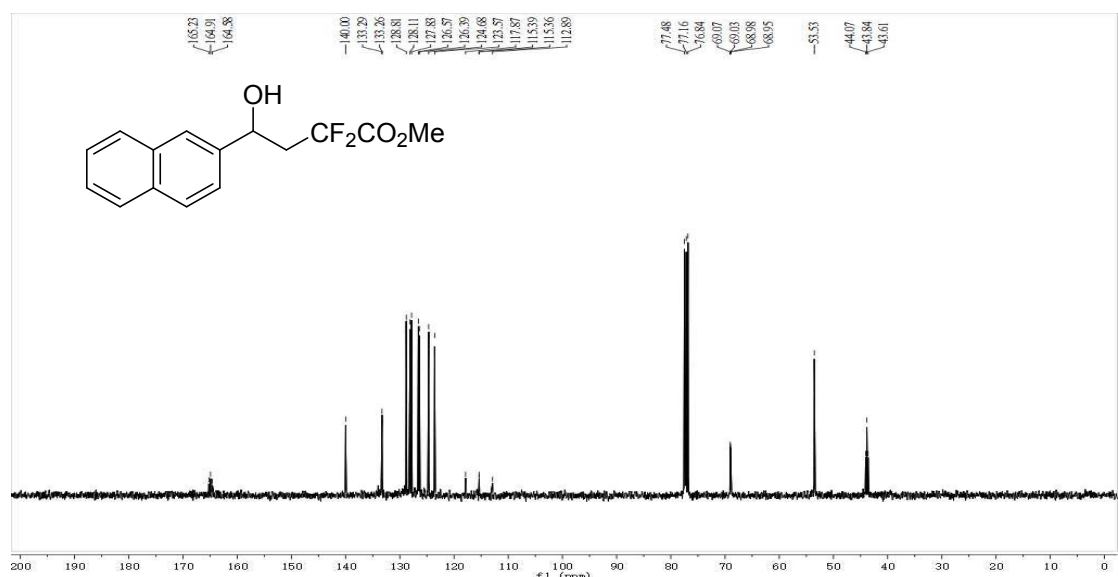
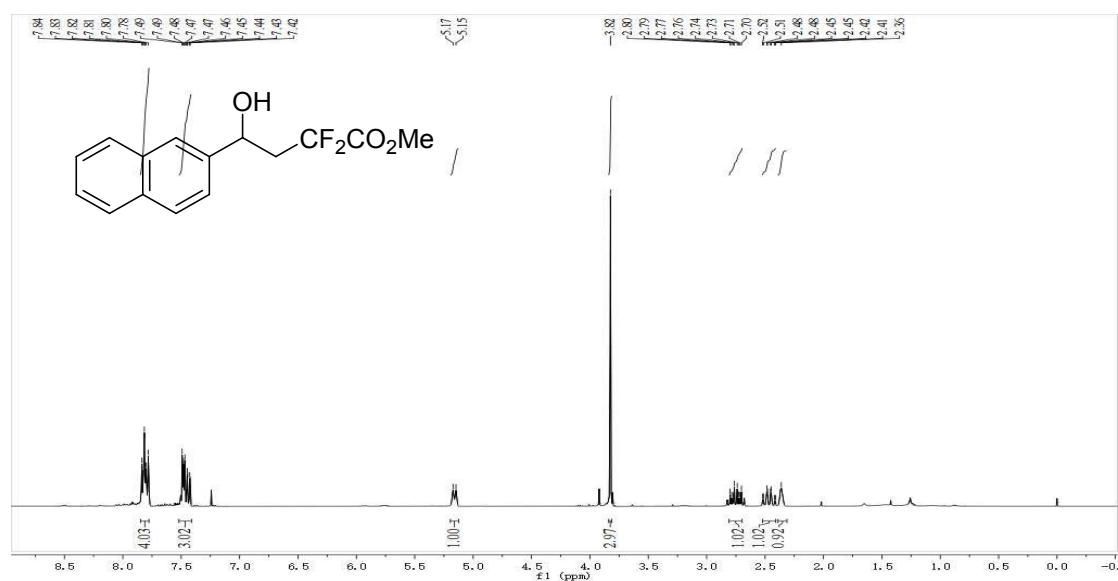
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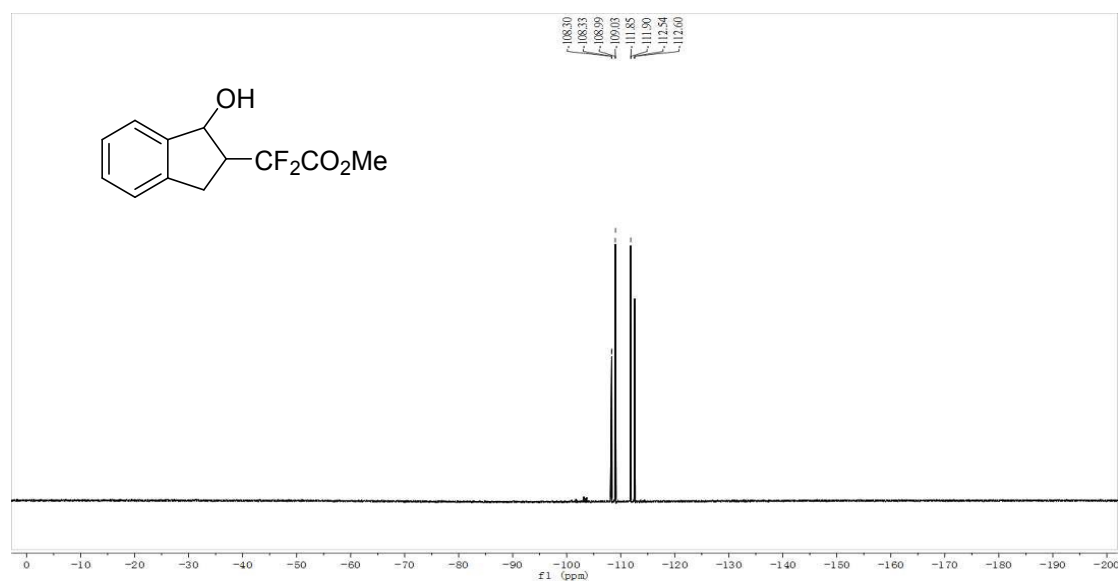
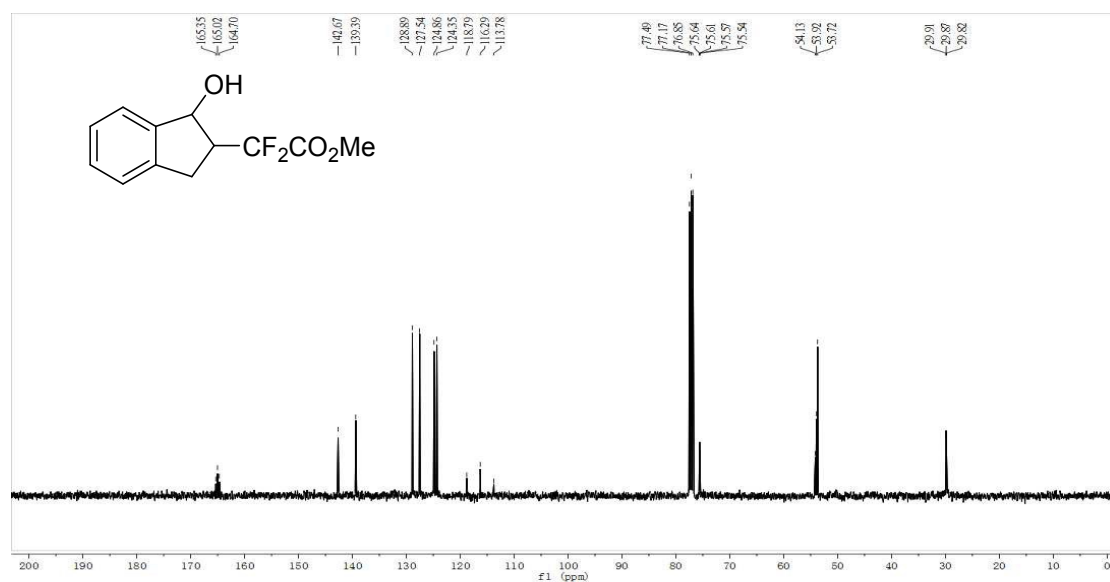
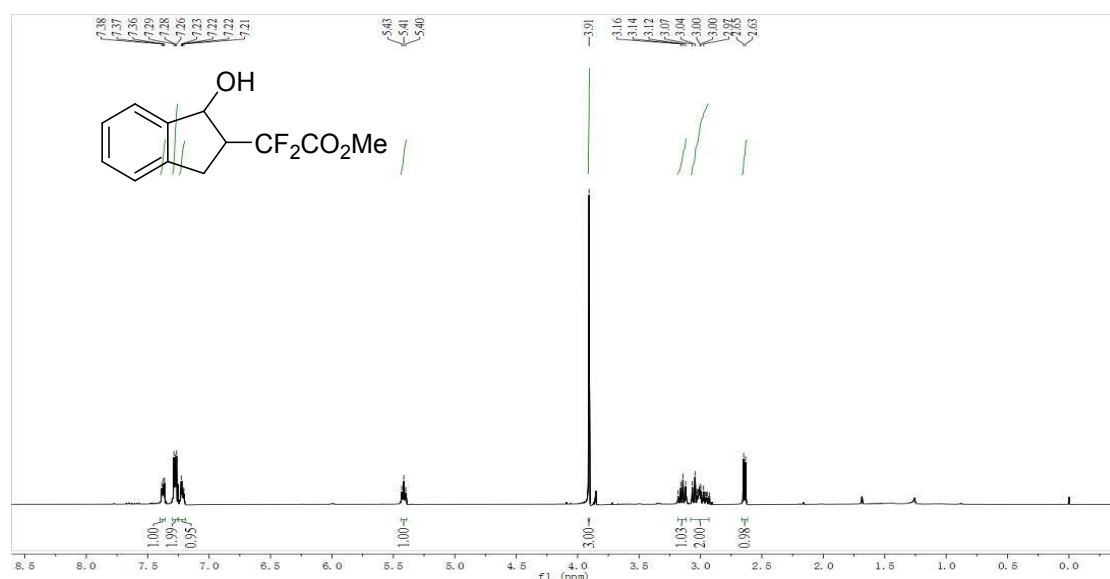
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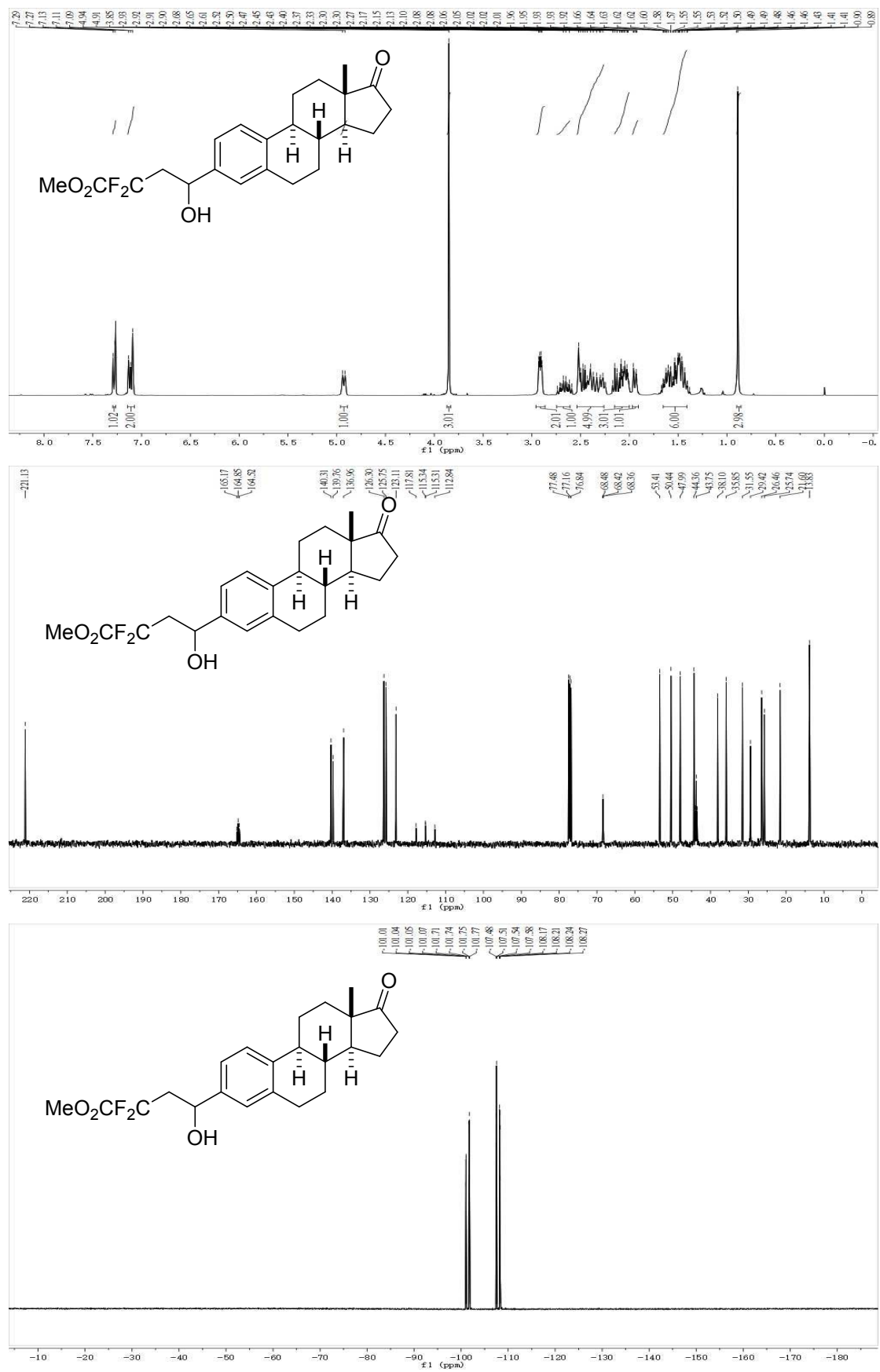
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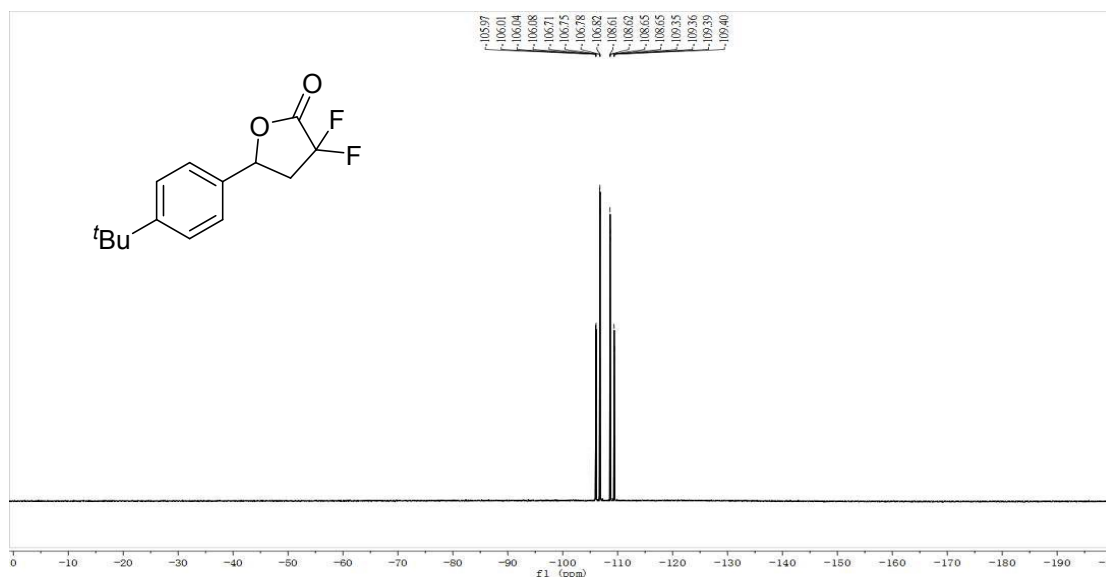
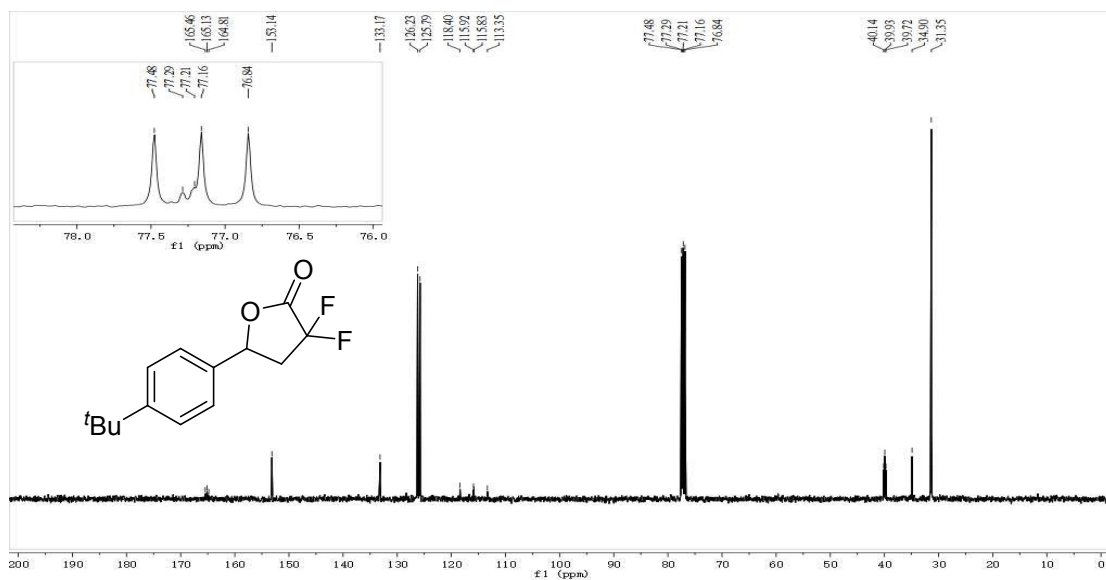
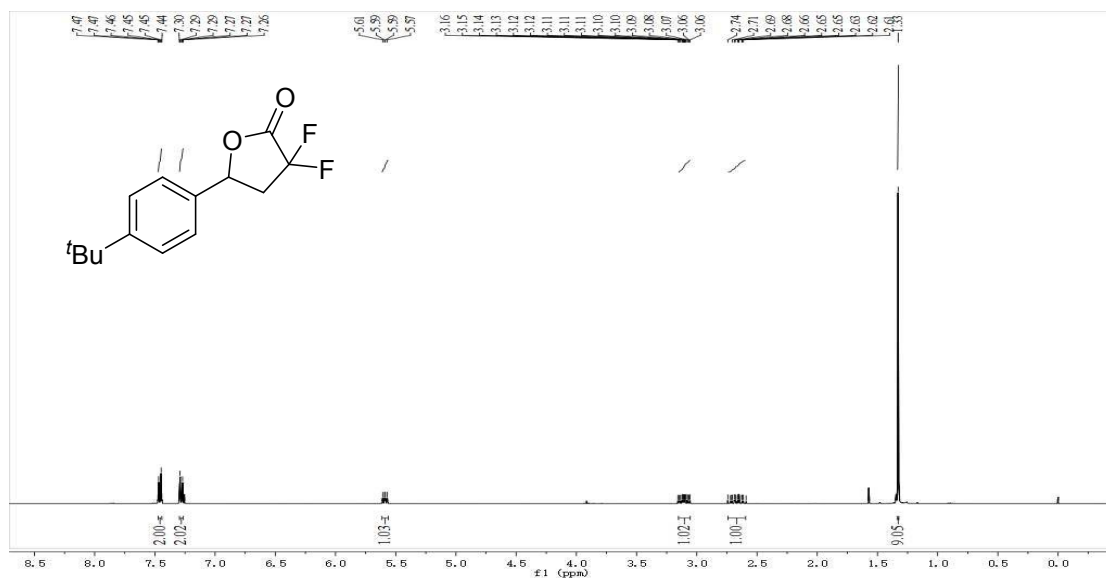
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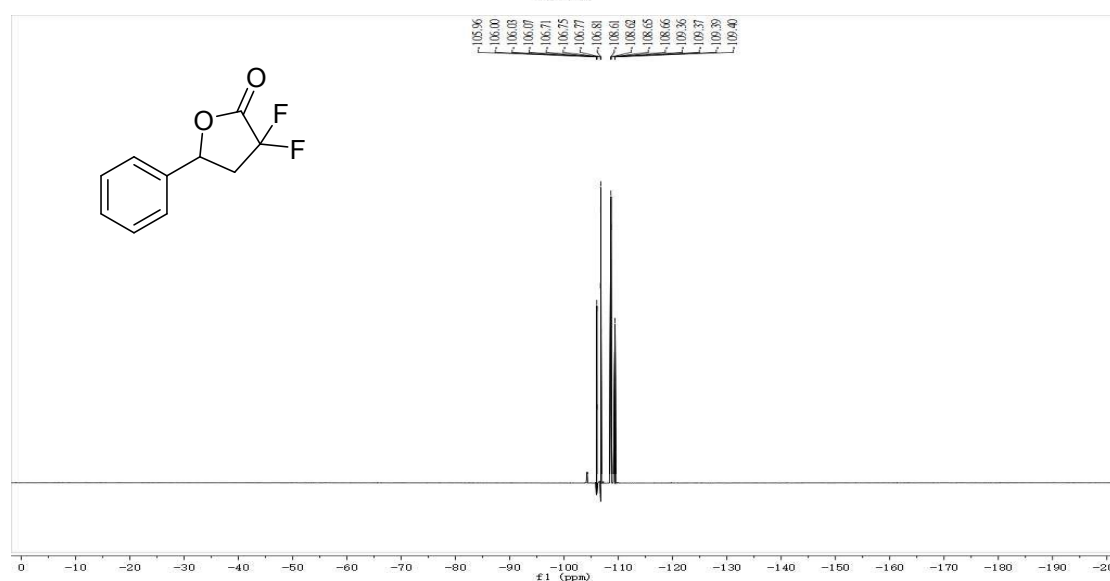
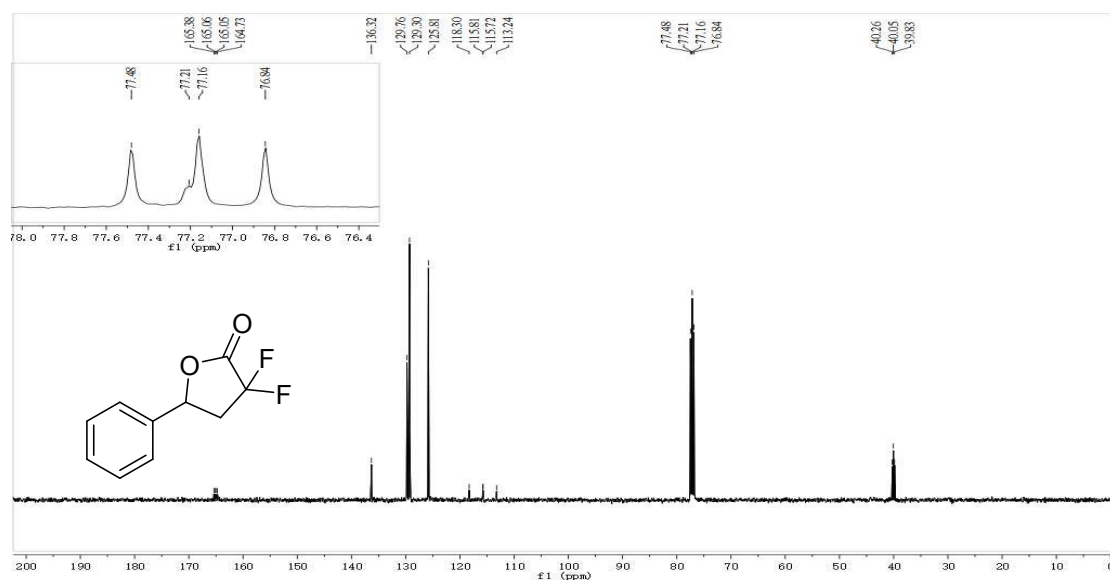
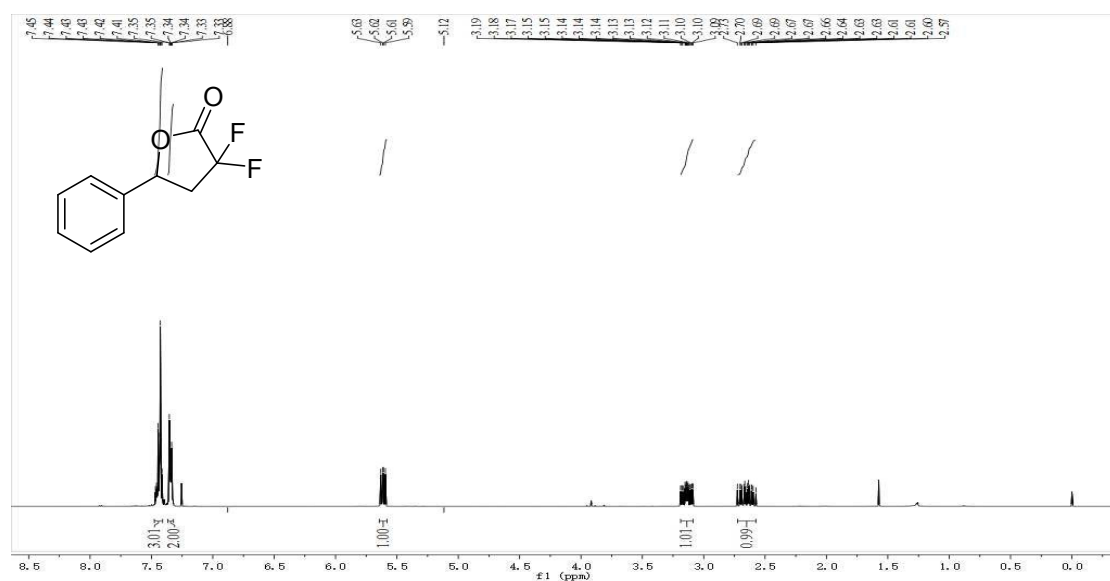
¹H NMR, ¹³C NMR and ¹⁹F NMR of **3r**



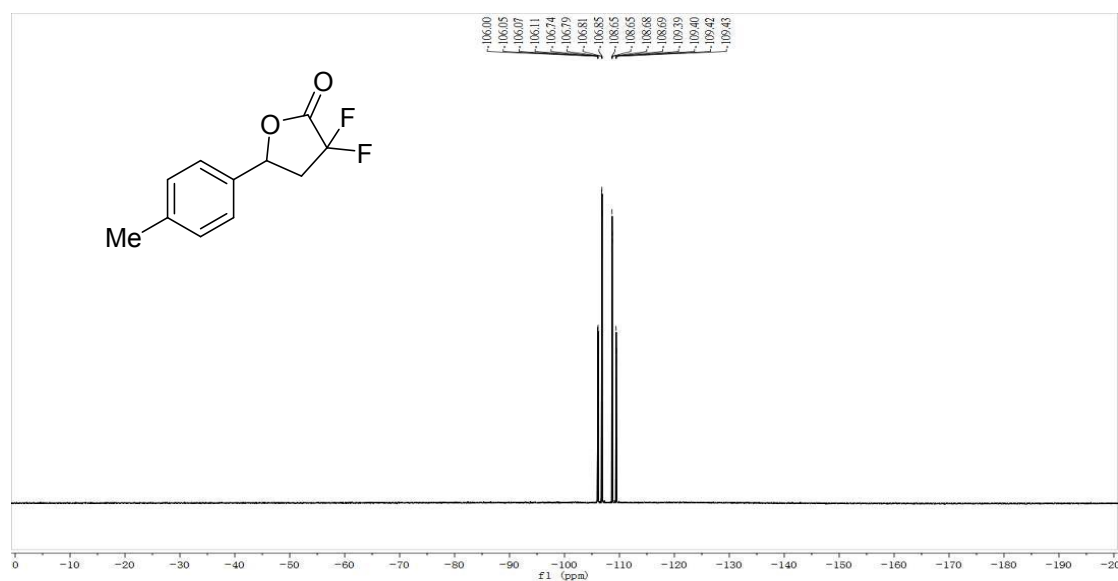
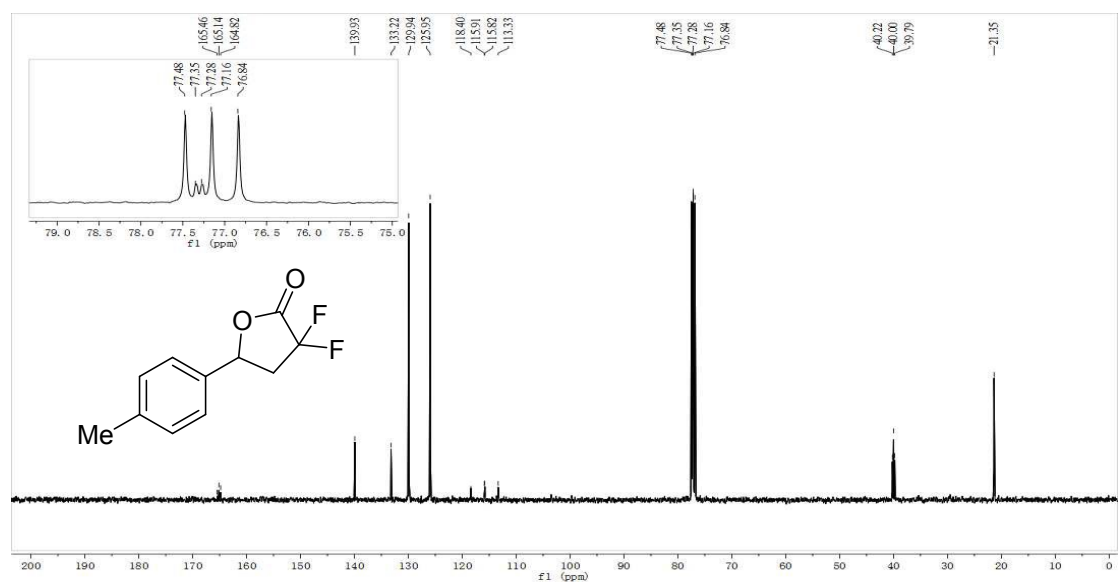
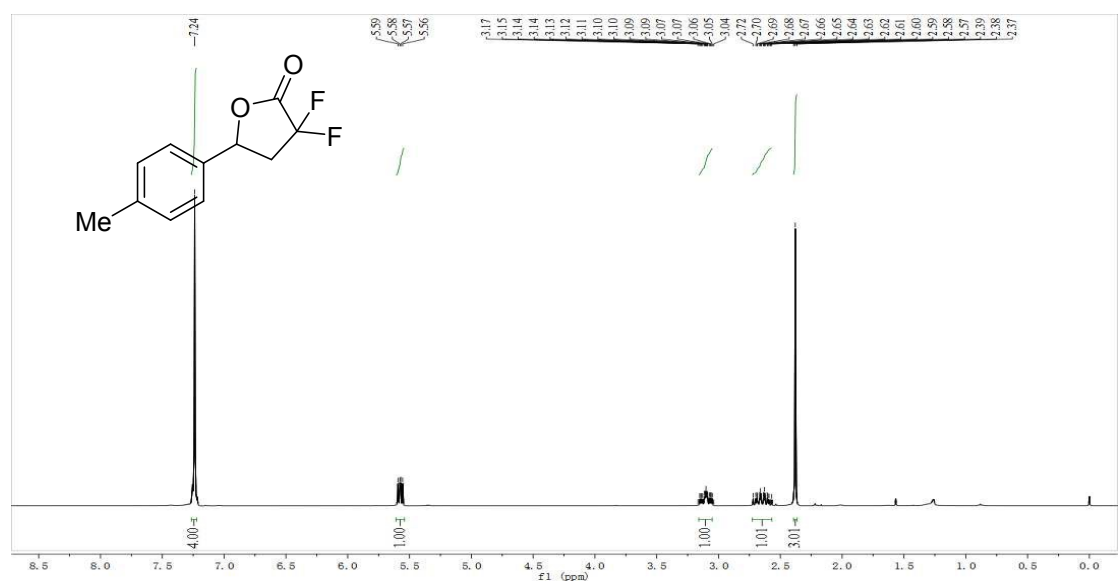
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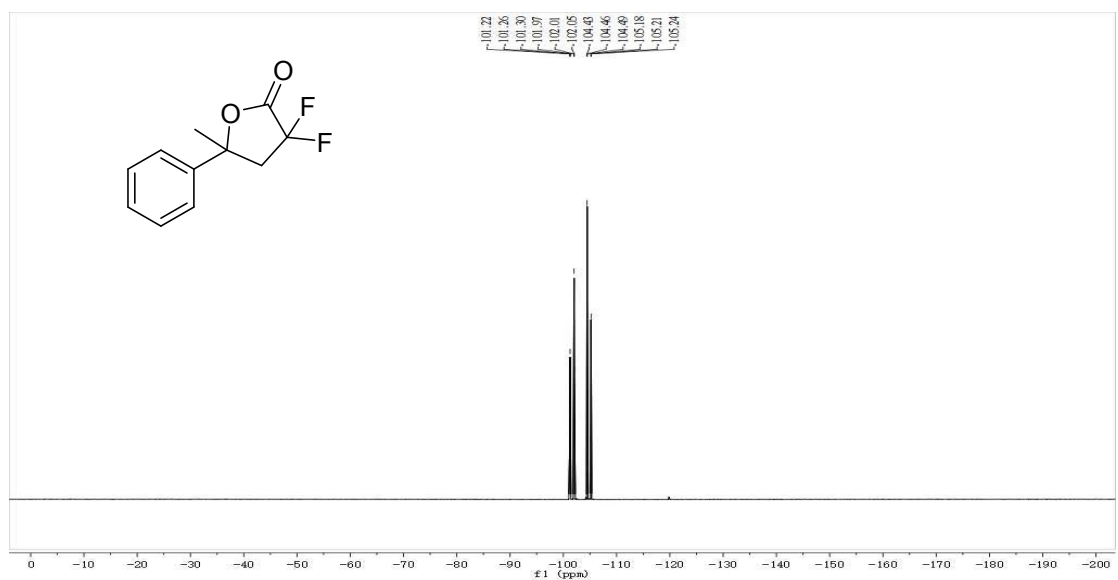
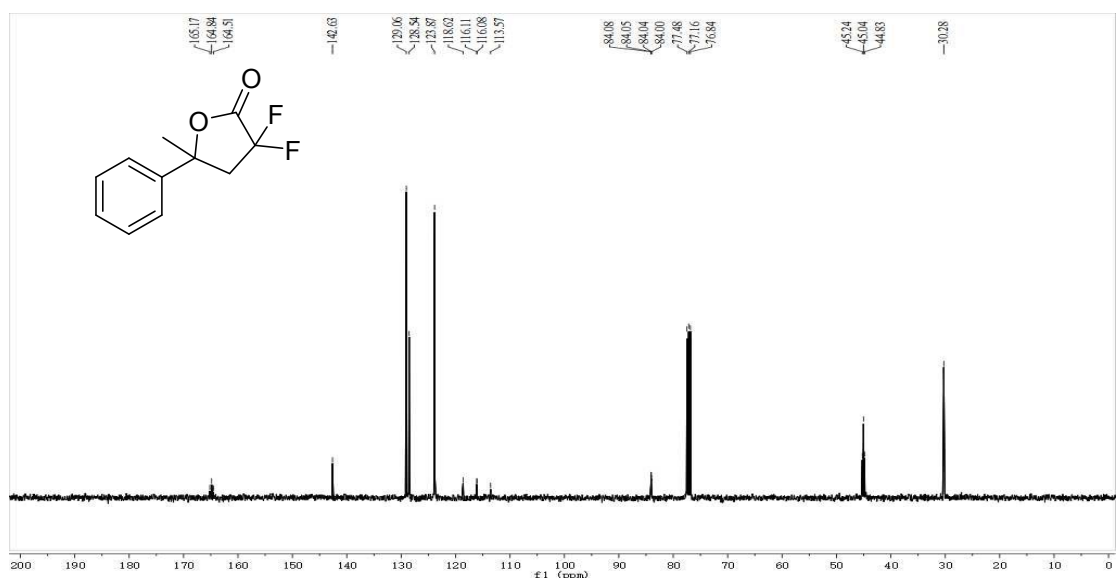
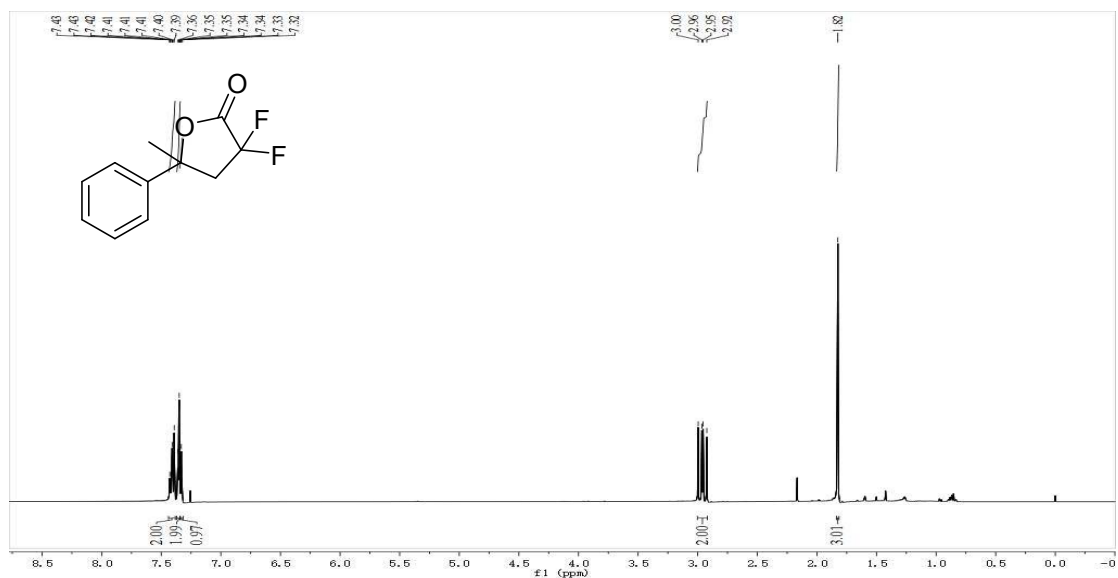
¹H NMR, ¹³C NMR and ¹⁹F NMR of 4a



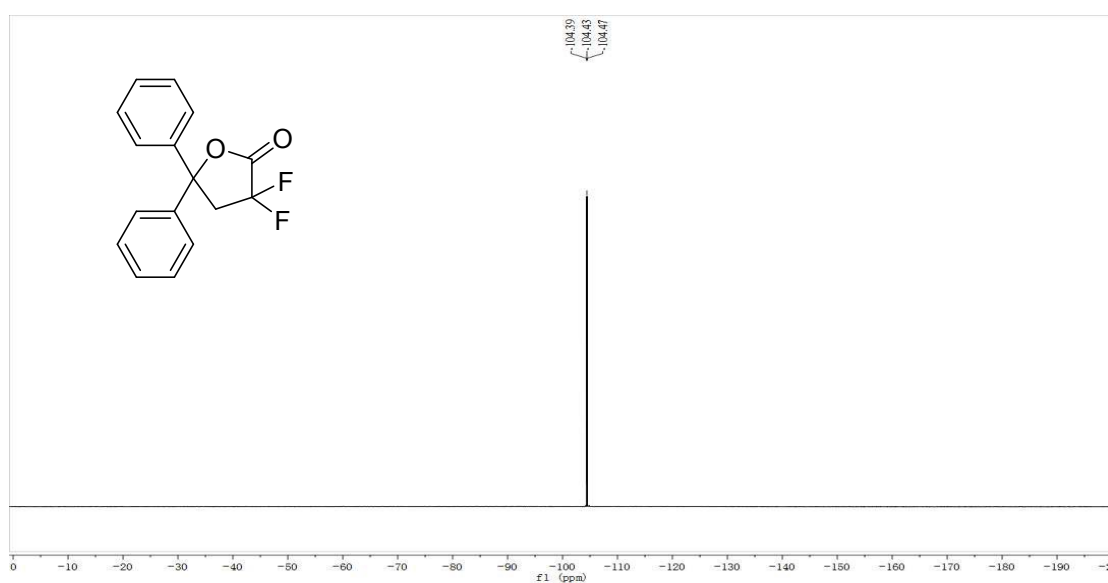
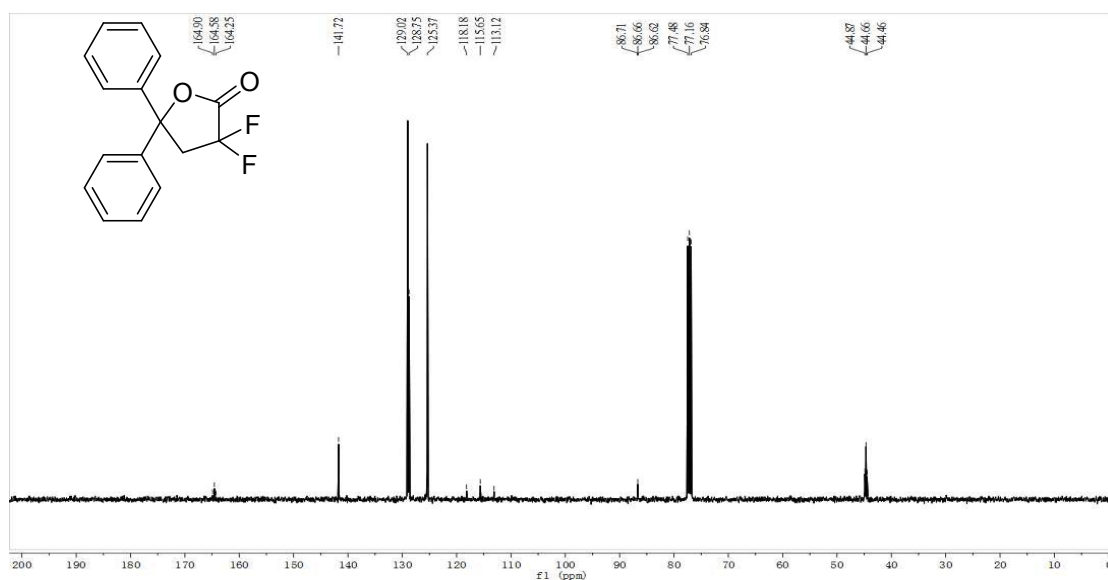
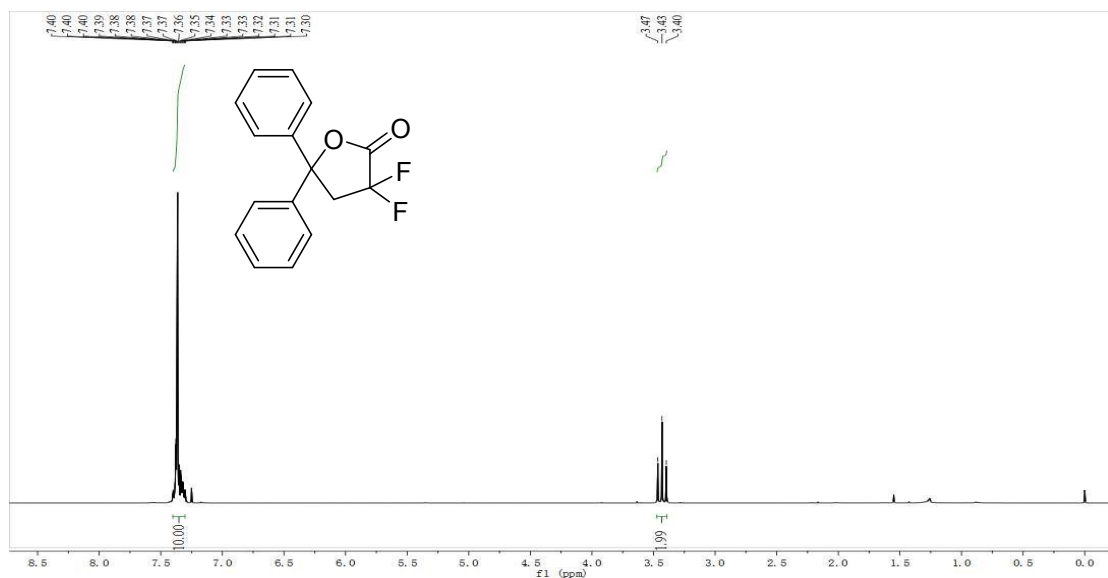
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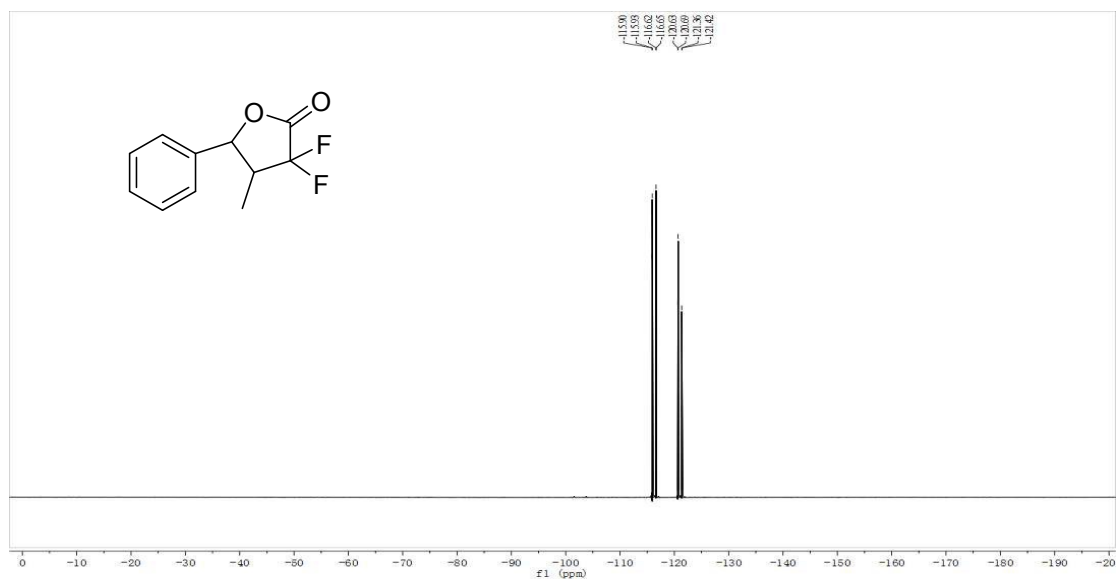
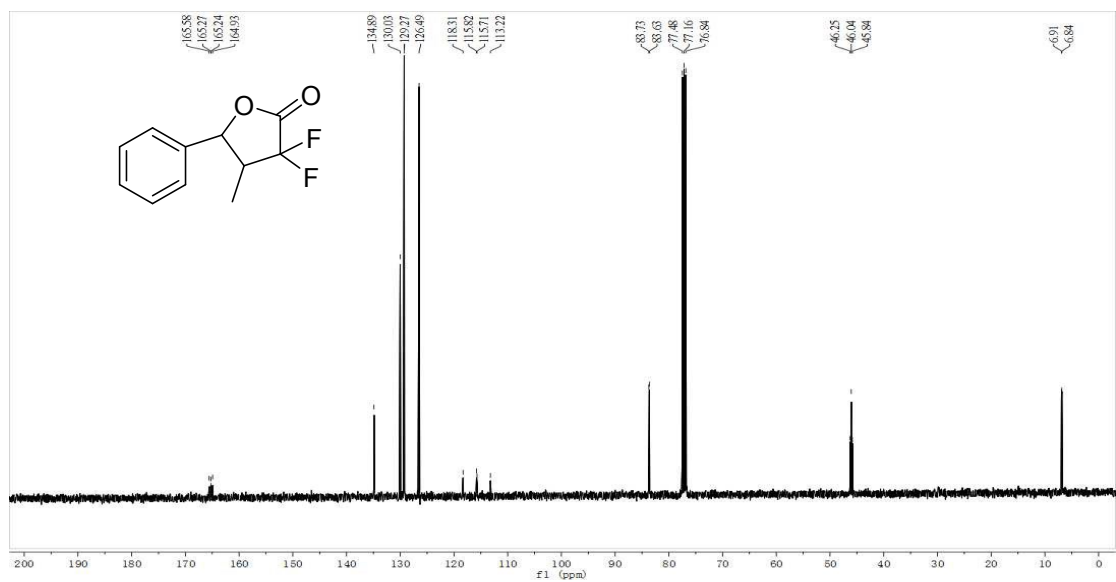
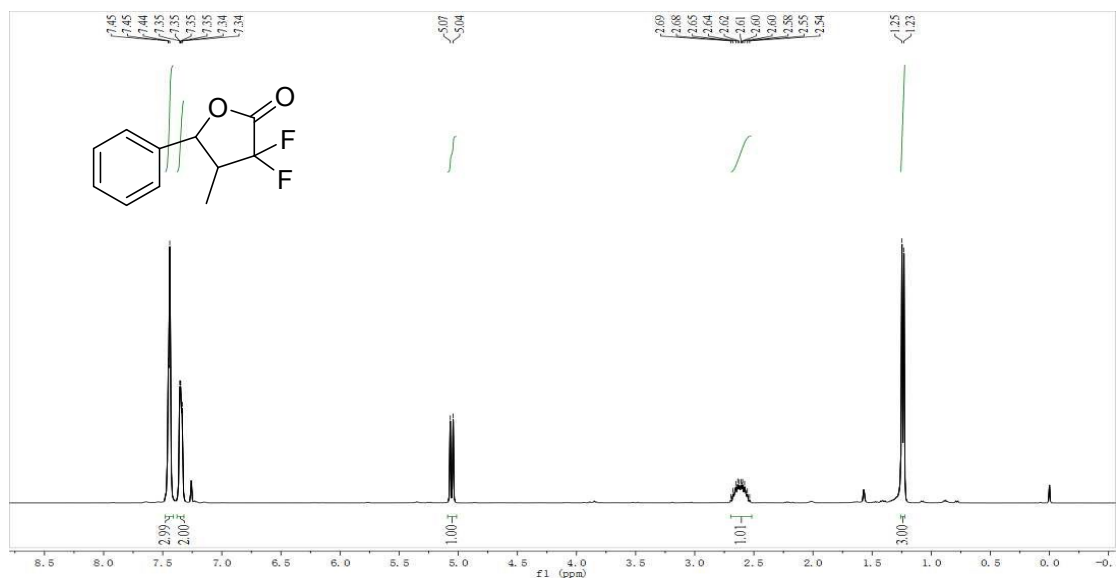
¹H NMR, ¹³C NMR and ¹⁹F NMR of 4c



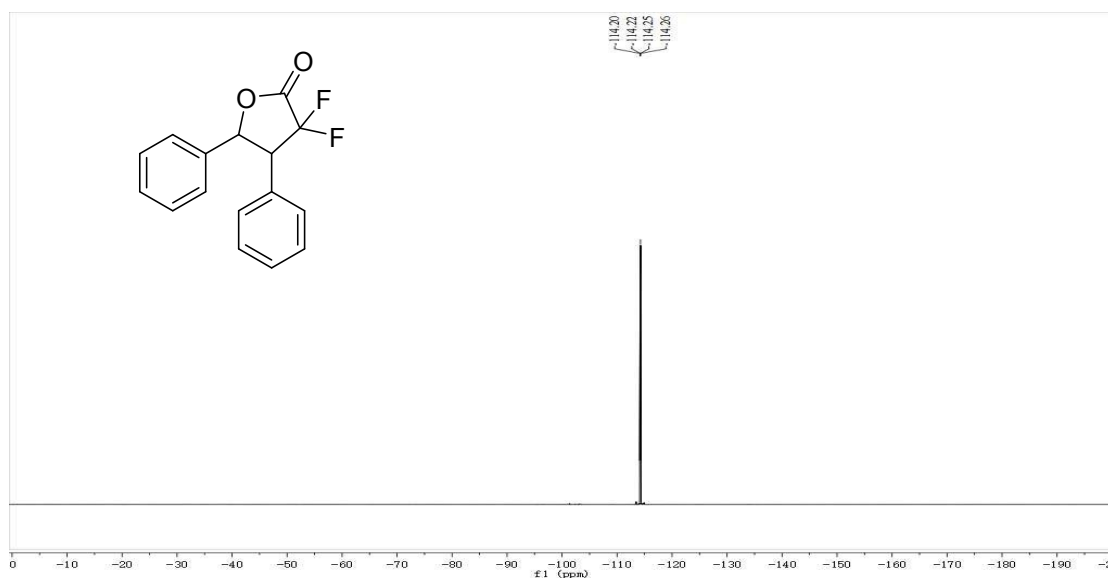
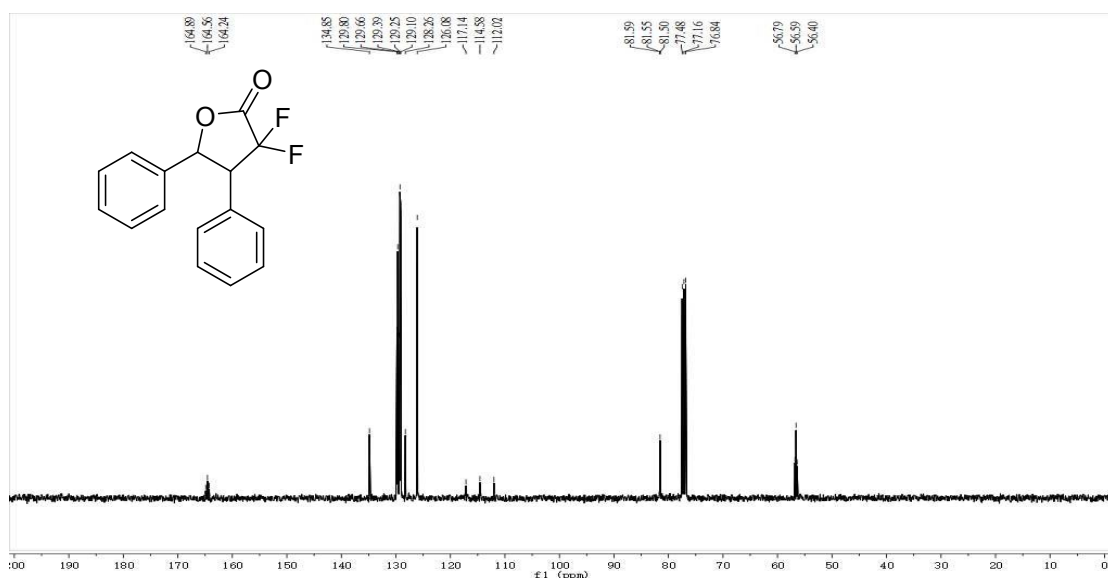
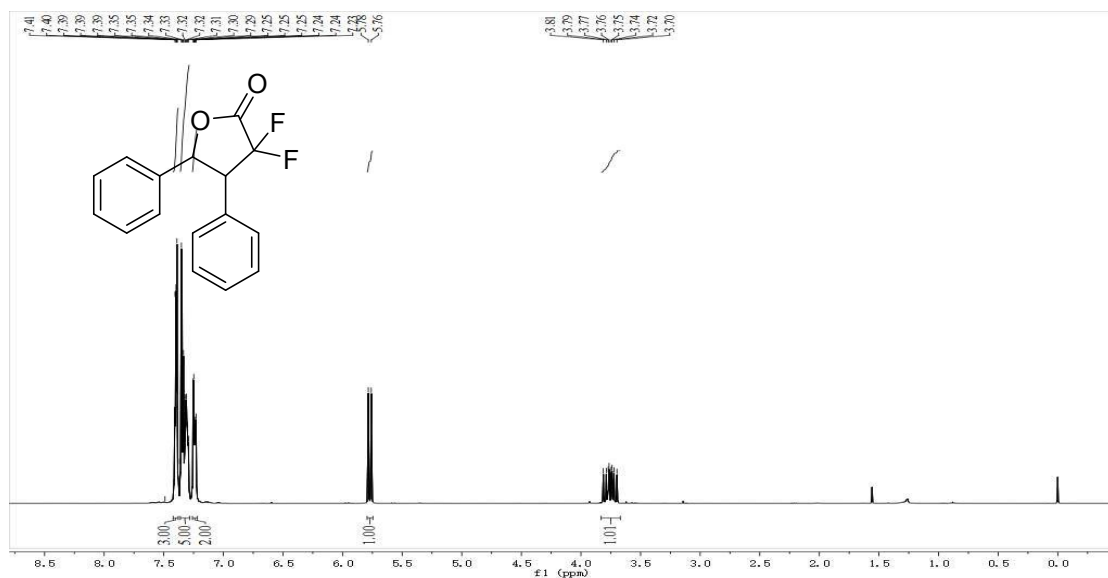
¹H NMR, ¹³C NMR and ¹⁹F NMR of 4v



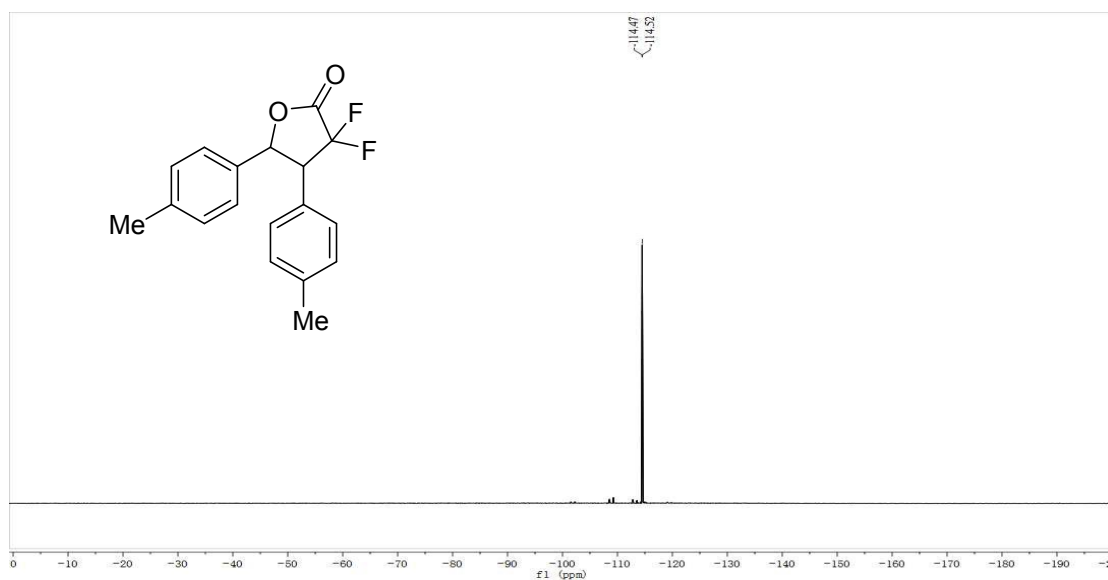
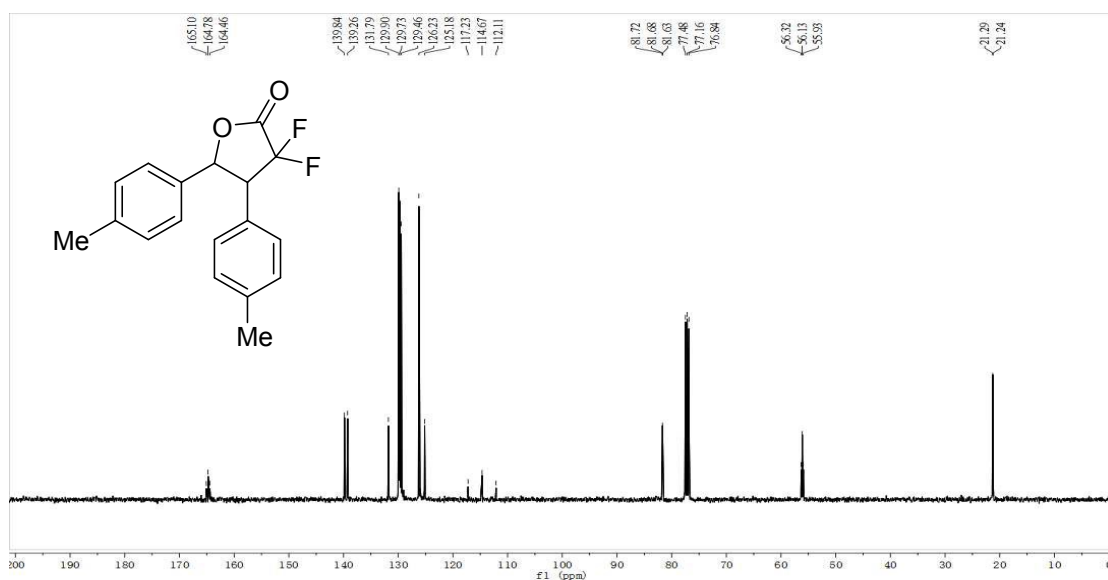
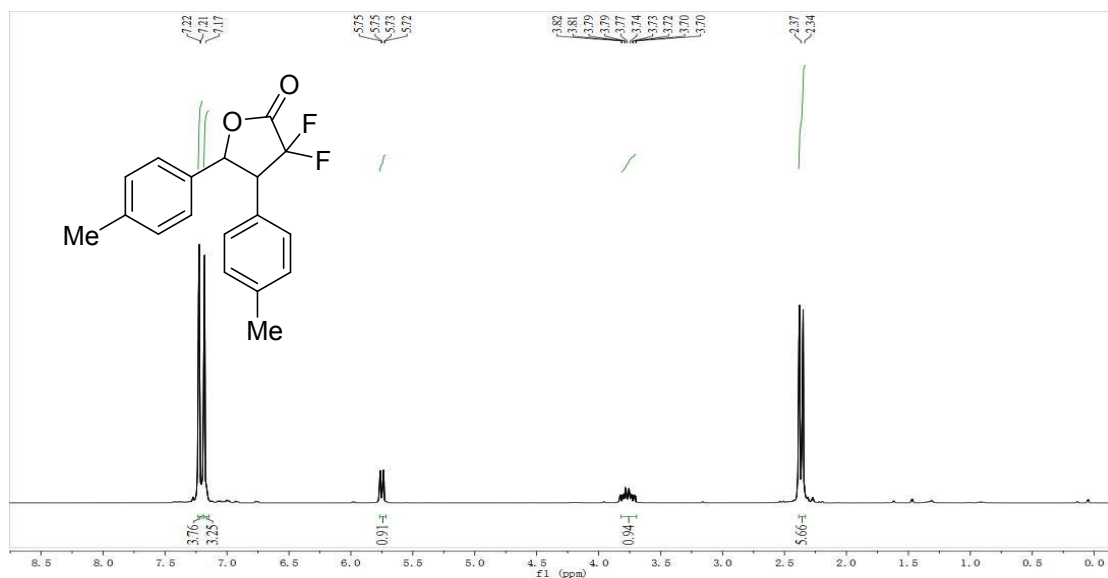
¹H NMR, ¹³C NMR and ¹⁹F NMR of 4w



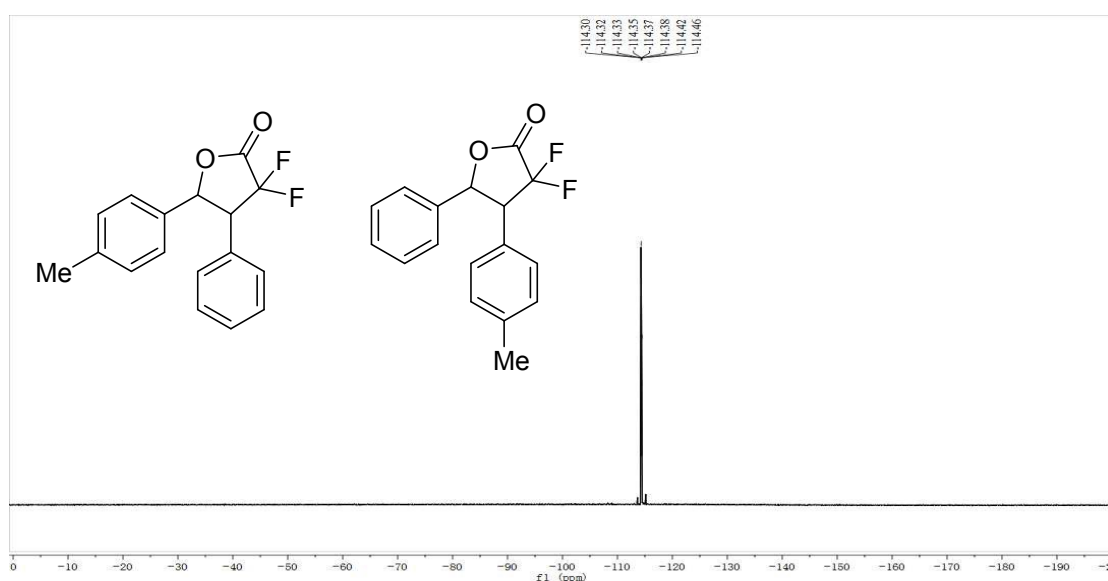
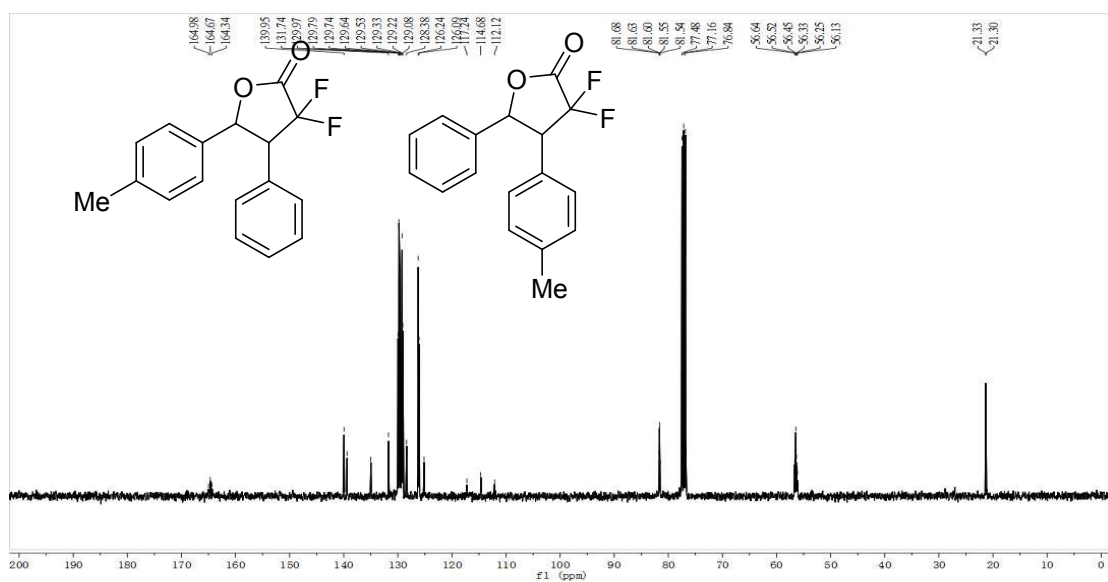
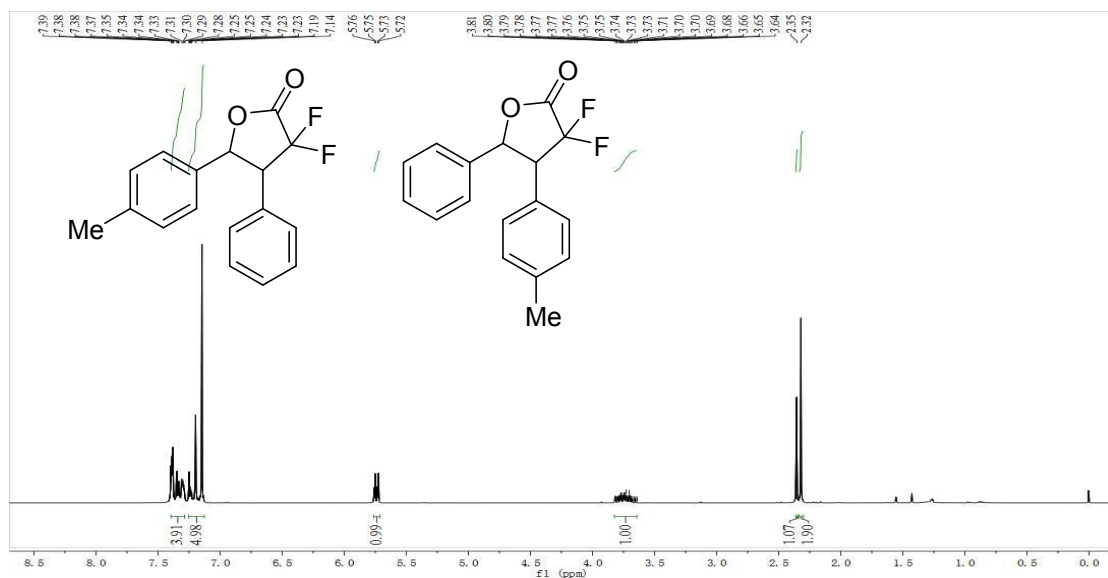
¹H NMR, ¹³C NMR and ¹⁹F NMR of 4x



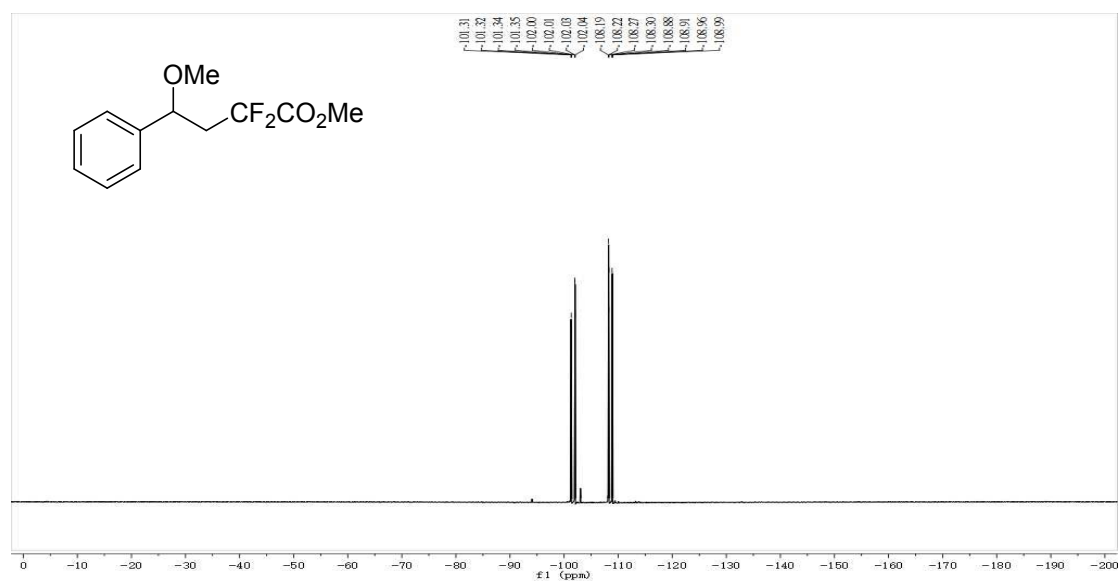
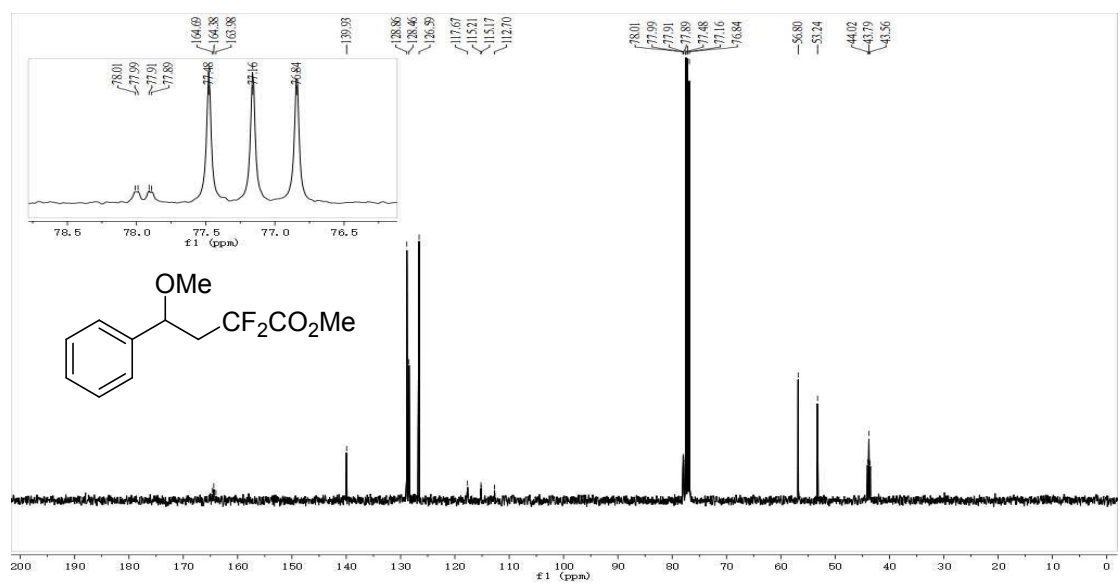
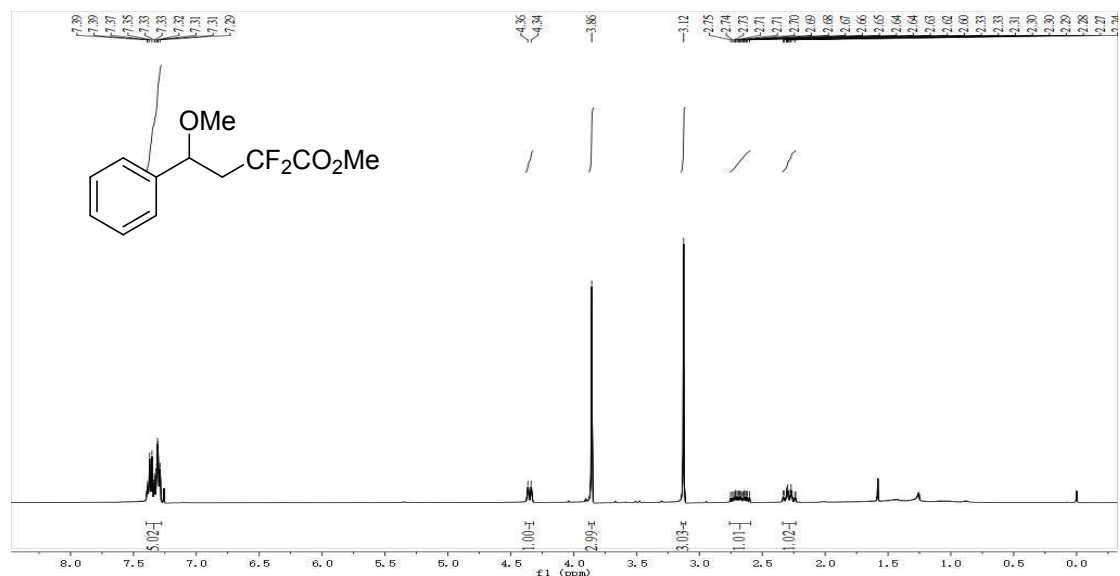
¹H NMR, ¹³C NMR and ¹⁹F NMR of 4y



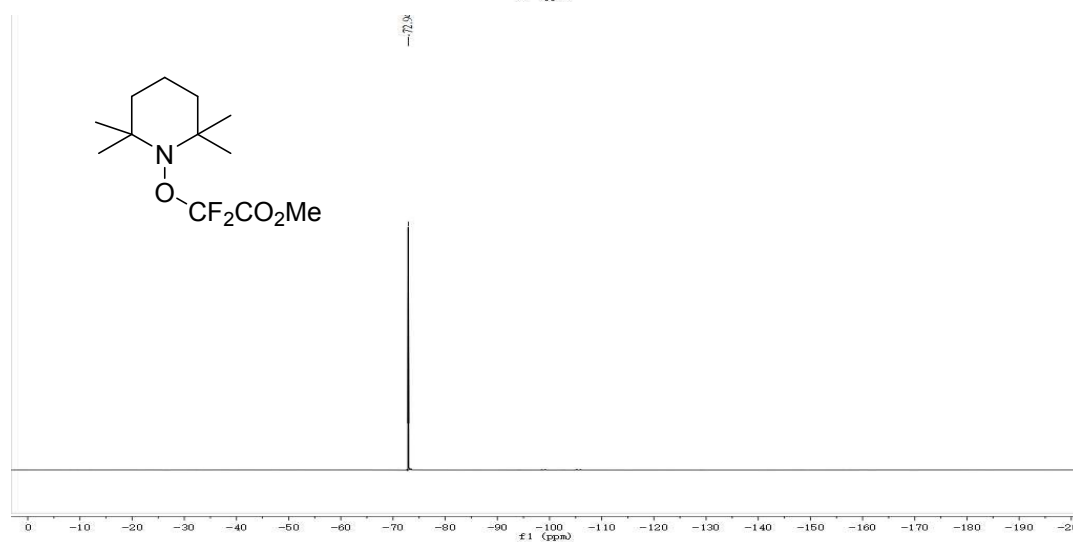
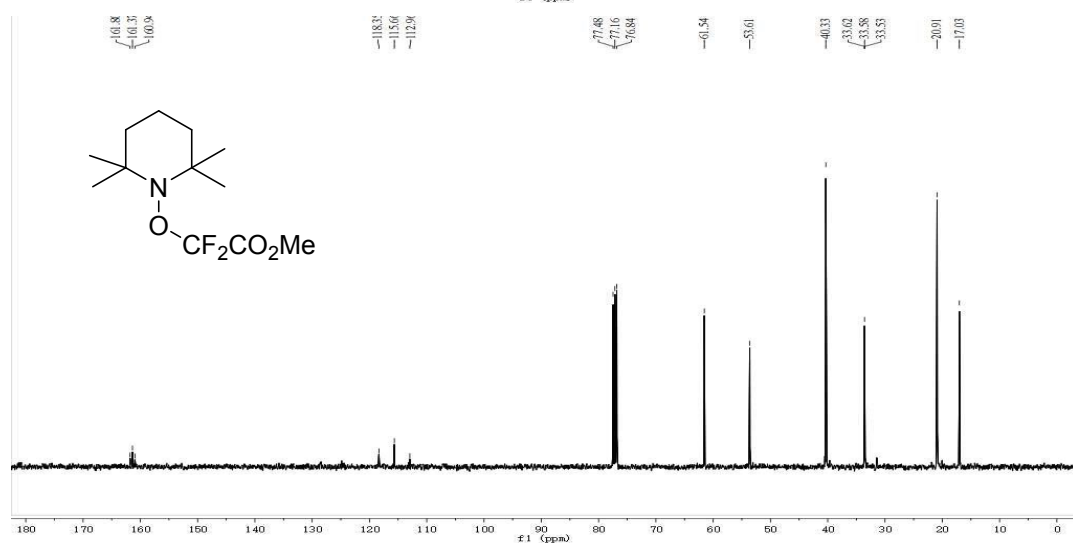
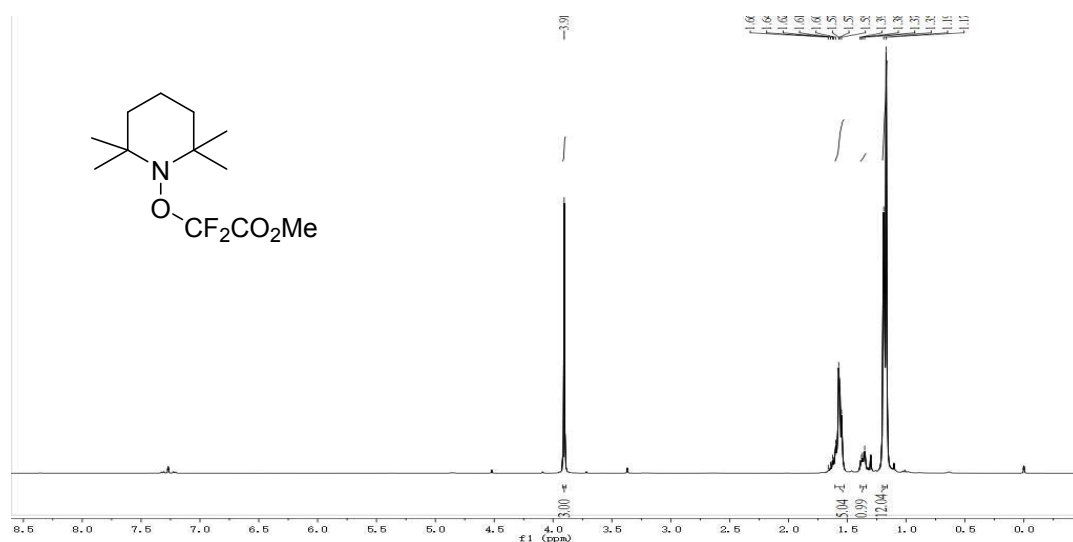
¹H NMR, ¹³C NMR and ¹⁹F NMR of **4z**



¹H NMR, ¹³C NMR and ¹⁹F NMR of the mixture of **4za** and **4za'**



¹H NMR, ¹³C NMR and ¹⁹F NMR of **5**



¹H NMR, ¹³C NMR and ¹⁹F NMR of 6