Supporting Information

Photoinduced radical-initiated carboxylative cyclization of allyl amines with carbon dioxide

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General Considerations

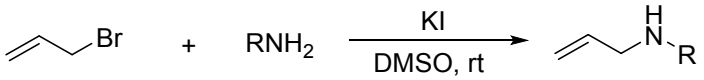
All reactions were carried out with standard Schlenk techniques. The products were isolated by column chromatography on silica gel (200-300 mesh) using petroleum ether (60-90 ^{o}C) and ethyl acetate. Photoreactions were carried out using a Xe lamp (CEL-HXF300, Radiant output 300W, visible output 5000 Lu, color temperature 5600K). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz spectrometer at 20 ^{o}C. All ^{1}H NMR spectra are reported in parts per million (ppm) downfield of TMS and were measured relative to the signals for $CDCl_3$ (7.26 ppm), DMSO-d_6 (2.50 ppm), CD_3CN (1.94 ppm) with ^{1}H decoupling. Coupling constants, *J*, are reported in hertz (Hz). Multiplets were assigned as singlet (s), doublet (d), triplet (t), doublet of doublet (dd) and multiplet (m). ^{13}C NMR was recorded at 101 MHz relative to $CDCl_3$ (77.16 ppm), DMSO-d_6 (39.52 ppm), CD_3CN (118.26 ppm). FT-IR was recorded on a Bruker Tensor 27 FT-IR spectrophotometer with KBr pellets. Mass spectra were recorded on a Shimadzu GCMS-QP2010 equipped with a RTX-5MS capillary column at an ionization voltage of 70 eV. The data are given as mass units per charge (m/z). Electrospray ionization mass spectra (ESI-MS) were recorded with an Agilent 6520 Q-TOF mass spectrometer. High resolution mass spectrometry was conducted using a Varian 7.0 T FTICR-MS by ESI technique.

Materials

Anhydrous DMSO and DMF were purchased from *J&K* chemical Co.. Anhydrous CH_3CN, THF and anhydrous $CHCl_3$ were purified according to *Purification of Common Laboratory Chemicals*. Unless otherwise noted, carbon dioxide (99.999%) was used. Allyl amines **1a** – **1b**, **1d** – **1i** were synthesized as previously described.[1,2] Other allyl amines/alcohols and chemicals were obtained from TCI, Aladdin or Alfa Aesar and used as received. The $[TBDH]^{+}I^{-}$ was synthesized via the neutralization of TBD and anhydrous hydriodic acid according to the reported procedures.[3]

General Procedure for Synthesis of Allyl Amines

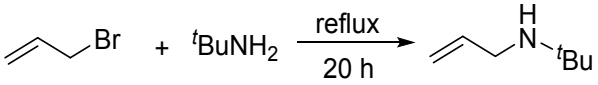
(1) A general procedure for synthesis of allyl amines 1a – 1b, 1e – 1i[1]



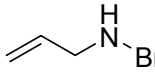
To a solution of corresponding amine (25.1 mmol) and KI (21.0 mg, 0.125 mmol) in DMSO (15 mL) was added dropwise 3-bromopropene or 3-bromo-2-methylprop-1-ene (12.7 mmol) via a

syringe at 0 °C. After stirring for 18 h at room temperature, to the reaction mixture was added 1M aqueous $NaHCO_3$ (30 mL), and the aqueous layer was extracted with Et_2O (5 × 15 mL). The combined organic layer was washed with brine (15 mL), dried over anhydrous $MgSO_4$ and filtered. The solution was concentrated in vacuo followed by silica gel flash column chromatography (eluent; petroleum ether/ethyl acetate = 4/1 to 1/1) to provide the corresponding allyl amines (yield 47% ~ 75%).

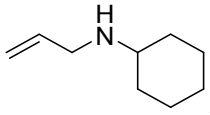
(2) A general procedure for synthesis of allyl amine 1d[2]



3-Bromopropene (1.09 g, 0.75 mL, 9 mmol) was added dropwise over 3 min to stirred and cooled (0 °C) n-butylamine (4.6 mL, 90 mmol). The resulting mixture was stirred and heated under reflux for 20 h, and then cooled to 0 °C. Concentrated hydrochloric acid was added dropwise to it over 5 min until pH 1 was attained. Water (25 mL) was added to the resulting suspension, which was then washed with diethyl ether (2 × 25 mL). The remaining aqueous solution was cooled to 0 °C and then brought to pH 10 by slow addition of potassium hydroxide pellets. The liberated amine was extracted into diethyl ether (3 × 25 mL). The combined organic phases were dried, filtered and the solvent removed under reduced pressure at 2 °C to leave a pale yellow liquid. The liquid was further purified by Kugelrohr distillation to yield the *N*-(*tert*-butyl)prop-2-en-1-amine (0.43 g, 38%) as a colourless oil.

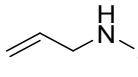


***N*-benzylprop-2-en-1-amine 1a**: yield 75%; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 4.4 Hz, 4H), 7.28 – 7.23 (m, 1H), 5.94 (dt, *J* = 16.3, 6.0 Hz, 1H), 5.20 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.12 (dd, *J* = 10.3, 1.4 Hz, 1H), 3.80 (s, 2H), 3.28 (dt, *J* = 5.9, 1.2 Hz, 2H), 1.54 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.3, 136.9, 128.4, 128.2, 127.0, 116.0, 53.3, 51.8; MS (ESI): calcd. for $C_{10}H_{14}N^+$ $[M + H]^+$ = 148.11, found 148.07.

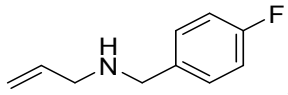


***N*-allylcyclohexanamine 1b**: yield 47%; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.90 (ddt, *J* = 16.6, 10.4, 6.1 Hz, 1H), 5.14 (d, *J* = 18.0 Hz, 1H), 5.06 (d, *J* = 10.2 Hz, 1H), 3.26 (d, *J* = 6.0 Hz, 2H), 2.44 (tt, *J* = 10.4, 3.7 Hz, 1H), 1.87 (d, *J* = 10.4 Hz, 2H), 1.72 – 1.61 (m, 5H), 1.25 – 1.18 (m, 2H), 1.09 – 1.04 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.9, 116.0, 57.9, 56.2, 49.4, 33.4,

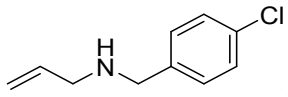
26.1, 25.1, 18.4; MS (ESI): calcd. for $C_9H_{18}N^+$ $[M + H]^+$ = 140.14, found 140.13.



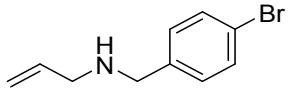
***N*-(*tert*-butyl)prop-2-en-1-amine 1d**: yield 38%; colourless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.89 (dq, *J* = 11.0, 6.0 Hz, 1H), 5.15 (d, *J* = 17.2 Hz, 1H), 5.06 (d, *J* = 10.2 Hz, 1H), 3.23 (d, *J* = 5.8 Hz, 2H), 2.59 (t, *J* = 7.1 Hz, 2H), 1.50 – 1.42 (m, 2H), 1.37 – 1.24 (m, 3H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.2, 115.7, 52.7, 49.3, 32.4, 20.6, 14.1; MS (ESI): calcd. for $C_7H_{16}N^+$ $[M + H]^+$ = 114.13, found 114.13.



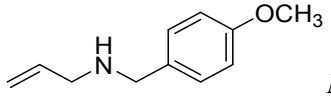
***N*-(4-fluorobenzyl)prop-2-en-1-amine 1e**: yield 70%; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.00 (t, *J* = 8.6 Hz, 2H), 5.92 (dq, *J* = 10.6, 6.0 Hz, 1H), 5.19 (d, *J* = 17.2 Hz, 1H), 5.11 (d, *J* = 10.2 Hz, 1H), 3.75 (s, 2H), 3.25 (d, *J* = 5.9 Hz, 2H), 1.50 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.9 (d, *J* = 244.5 Hz), 136.7, 136.0 (d, *J* = 3.1 Hz), 129.8 (d, *J* = 7.9 Hz), 116.2, 115.2 (d, *J* = 21.2 Hz), 52.5, 51.8; MS (ESI): calcd. for $C_{10}H_{13}FN^+$ $[M + H]^+$ = 166.21, found 166.07.



***N*-(4-chlorobenzyl)prop-2-en-1-amine 1f**: yield 72%; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.24 (m, 1H), 5.91 (ddt, *J* = 16.3, 10.3, 6.0 Hz, 1H), 5.19 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.12 (dd, *J* = 10.2, 1.2 Hz, 1H), 3.75 (s, 1H), 3.25 (d, *J* = 6.0 Hz, 2H), 1.45 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.8, 136.7, 132.7, 129.6, 128.6, 116.3, 52.5, 51.8; MS (ESI): calcd. for $C_{10}H_{13}ClN^+$ $[M + H]^+$ = 182.07, found 182.00.

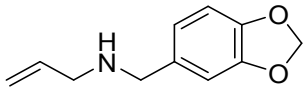


***N*-(4-bromobenzyl)prop-2-en-1-amine 1g**: yield 70%; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 8.2 Hz, 2H), 5.91 (ddt, *J* = 16.3, 10.3, 6.0 Hz, 1H), 5.18 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.11 (dd, *J* = 10.2, 1.3 Hz, 1H), 3.73 (s, 2H), 3.25 (d, *J* = 6.0 Hz, 2H), 1.54 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.3, 136.6, 131.5, 130.0, 120.8, 116.3, 52.6, 51.7; MS (ESI): calcd. for $C_{10}H_{13}BrN^+$ $[M + H]^+$ = 226.02, 228.02, found 226.00, 228.00.



***N*-(4-methoxybenzyl)prop-2-en-1-amine 1h**: yield 69%; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ7.26 (d, *J* = 7.5 Hz, 2H), 6.88 (d, *J* = 7.3 Hz, 2H), 5.95 (dq, *J* = 11.1, 6.0 Hz, 1H), 5.17 (dd, *J* = 33.1, 13.7 Hz, 2H), 3.81 (s, 3H), 3.74 (s, 2H), 3.28 (d, *J* = 5.9 Hz, 2H),

1.53 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.6, 136.9, 132.4, 129.4, 116.0, 113.8, 55.3, 52.7, 51.7; MS (ESI): calcd. for $C_{11}H_{16}NO^+$ $[M + H]^+$ = 178.12, found 178.00.

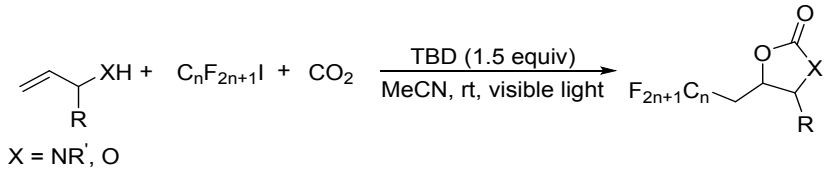


***N*-(benzo[*d*][1,3]dioxol-5-ylmethyl)prop-2-en-1-amine 1i**: yield 51%; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.85 (s, 1H), 6.79 – 6.74 (m, 2H), 5.96 – 5.89 (m, 3H), 5.20 (dd, *J* = 17.2, 1.5 Hz, 1H), 5.13 (dd, *J* = 10.2, 1.1 Hz, 1H), 3.72 (s, 2H), 3.27 (d, *J* = 6.0 Hz, 2H), 2.52 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.7, 146.7, 136.0, 133.3, 121.5, 116.7, 108.9, 108.1, 100.9, 52.7, 51.3; MS (ESI): calcd. for $C_{11}H_{14}NO_2^+$ $[M + H]^+$ = 192.10, 192.01

The Procedure for Synthesis of $[TBDH]^+I^-$

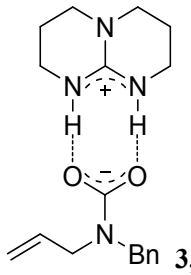
In a 25 mL round bottom flask, anhydrous hydriodic acid (55% w/w, 1.2 mL, 5 mmol HI,) was dropped into the solution of TBD (0.6950 g, 5 mmol) in 5 mL water at 0 °C within 20 minutes. After the addition, the reaction mixture was stirred for an addition period of 2 hours at room temperature to ensure the reaction had proceeded to completion. The solution was concentrated in vacuo and the small amount of water was removed by heating the residue at 80 °C in high vacuum until the weight of the residue remained constant. The white crystalline $[TBDH]^+I^-$ was obtained in 99% yield (1.3216 g). Melting point: 106 – 108 °C; IR (KBr) *v* 3310, 3282, 3220, 3040, 2871, 1638, 1534, 1375, 1065 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.36 (s, 2H), 3.28 (t, *J* = 6 Hz, 4H), 3.19 – 3.16 (m, 4H), 1.89 – 1.83 (m, 4H), ^{13}C NMR (101 MHz, DMSO-d_6) δ 150.5, 46.2, 37.5, 20.2.

General procedure for photoinduced radical-initiated carboxylative cyclization of allyl amines/alcohols with CO_2

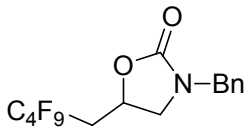


TBD (0.1044 g, 0.75 mmol) was charged to the tube in a 100 mL steel autoclave. Then the autoclave was subjected to three cycles of pressurization and depressurization with CO_2. In CO_2 atmosphere, allyl amines/alcohol (0.5 mmol), MeCN/DMSO (2 mL) was added and the mixture was stirred for 2 h. Then perfluoroalky iodide (1 mmol) was added and the contents was stirred for appropriate time under a 300 W Xe lamp (420 – 700nm). Thereafter, the autoclave was cooled to room

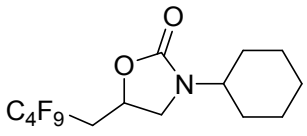
temperature and carefully depressurized. The crude reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 4/1) to provide the corresponding products (yield 47% ~ 99%).



3,4,6,7,8,9-Hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidin-1-ium allyl(benzyl)carbamate (E), ^{1}H NMR (400 MHz, CD_3CN) δ 11.32 (s, 2H), 7.56 – 6.88 (m, 5H), 5.98 – 5.60 (m, 1H), 5.00 (d, *J* = 12.3 Hz, 2H), 4.39 (s, 2H), 3.76 (d, *J* = 5.4 Hz, 2H), 3.22 (t, *J* = 5.7 Hz, 4H), 3.13 (t, *J* = 5.3 Hz, 4H), 1.90 – 1.82 (m, 4H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 164.4, 152.7, 142.2, 137.5, 129.0, 128.3, 127.1, 115.2, 49.6, 49.2, 47.3, 38.2, 21.7.

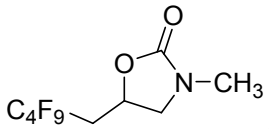


3-Benzyl-5-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)oxazolidin-2-one (2a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.26 (m, 5H), 4.95-4.80 (m, 1H), 4.49 (d, *J* = 14.8 Hz, 1H), 4.38 (d, *J* = 14.8 Hz, 1H), 3.63 (t, *J* = 8.7 Hz, 1H), 3.19 (dd, *J* = 9.0, 7.5 Hz, 1H), 2.75-2.61 (m, 1H), 2.39 (ddd, *J* = 24.2, 16.5, 7.2 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.04, 135.33, 129.13, 128.39, 128.34, 66.94 (t, *J* = 3.3 Hz), 49.71, 48.49, 36.15 (t, *J* = 21.0 Hz), (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, $CDCl_3$) δ -81.0 (t, *J* = 9.1 Hz, 3F), -112.7 – -112.8 (m, 2F), -124.5 (m, 2F), -126.0 (t, *J* = 12.4 Hz, 2F); HRMS (ESI): $C_{15}H_{13}F_9NO_2^+$ for $[M + H]^+$ calculated 410.0797, found 410.0797.

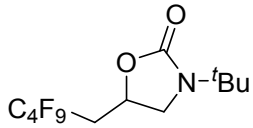


3-Cyclohexyl-5-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)oxazolidin-2-one (2b): ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.86 (qd, *J* = 7.4, 5.5 Hz, 1H), 3.74 – 3.65 (m, 2H), 3.28 (dd, *J* = 8.9, 7.3 Hz, 1H), 2.76 – 2.61(m, 1H), 2.42 (ddd, *J* = 24.1, 17.3, 7.4 Hz, 1H), 1.81 – 1.80 (m, 4H), 1.69 – 1.66 (m, 1H), 1.41 – 1.28 (m, 4H), 1.12 – 1.03 (m, 1H); ^{13}C NMR (101 MHz, DMSO-d_6) δ155.9, 66.8, 52.1, 45.4, 34.7 (t, *J* = 20.2 Hz), 29.9, 29.5, 25.0 (m), (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, $CDCl_3$) δ -81.0 (t, *J* = 9.1 Hz, 3F), -112.7 – -112.8 (m, 2F), -124.5 (s, 2F), -126.0 (t, *J* = 12.4 Hz, 2F); HRMS (ESI): $C_{14}H_{17}F_9NO_2^+$ for

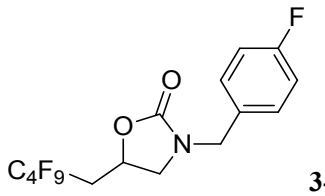
$[M + H]^+$ calculated 402.1110, found 402.1106.



3-Methyl-5-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)oxazolidin-2-one (2c): ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.89 – 4.82 (m, 1H), 3.76 (t, *J* = 8.7 Hz, 1H), 3.33 (t, *J* = 8.1 Hz, 1H), 2.87 (s, 3H), 2.67 (ddd, *J* = 38.1, 15.7, 5.6 Hz, 1H), 2.43 (ddd, *J* = 36.1, 15.4, 6.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.3, 66.6, 52.4, 36.1 (t, *J* = 20.9 Hz), 31.0, (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (376 MHz, $CDCl_3$) δ -81.1 (t, *J* = 8.2 Hz, 3F), -112.7 – -113.2 (m, 2F), -124.4 – -124.7 (m, 2F), -125.9 – -126.2 (m, 2F); HRMS (ESI): $C_8H_9F_9NO_2^+$ for $[M + H]^+$ calculated 334.0484, found 334.0492.

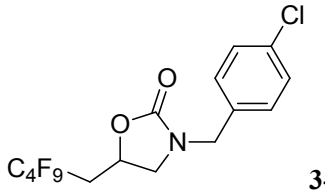


3-(*Tert*-butyl)-5-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)oxazolidin-2-one (2d): ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.96 – 4.86 (m, 1H), 3.79 (t, *J* = 8.6 Hz, 1H), 3.36 – 3.19 (m, 3H), 2.70 (td, *J* = 20.5, 4.9 Hz, 1H), 2.46 (qd, *J* = 16.6, 7.0 Hz, 1H), 1.58 – 1.51 (m, 2H), 1.40 – 1.31 (m, 2H), 0.95 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.0, 66.7, 50.3, 44.0, 36.2 (t, *J* = 21.0 Hz), 29.4, 19.9, 13.7, (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (376 MHz, $CDCl_3$) δ -81.2 (t, *J* = 9.1 Hz, 3F), -112.8 – -112.9 (m, 2F), -124.5 (s, 2F), -126.0 (t, *J* = 12.4 Hz, 2F); HRMS (ESI): $C_{12}H_{15}F_9NO_2^+$ for $[M + H]^+$ calculated 376.0954, found 376.0952.

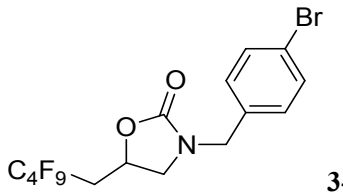


3-(4-Fluorobenzyl)-5-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)oxazolidin-2-one (2e): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.26 (m, 2H), 7.09 – 7.03 (m, 2H), 4.89 (dt, *J* = 14.2, 7.2 Hz, 1H), 4.47 (d, *J* = 14.9 Hz, 1H), 4.36 (d, *J* = 14.9 Hz, 1H), 3.64 (t, *J* = 8.7 Hz, 1H), 3.20 (dd, *J* = 8.9, 7.5 Hz, 1H), 2.76 – 2.61 (m, 1H), 2.41 (ddd, *J* = 36.2, 15.7, 7.0 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.7 (d, *J* = 247.0 Hz), 157.0, 131.2 (d, *J* = 2.9 Hz), 130.10 (d, *J* = 8.1 Hz), 116.02 (d, *J* = 21.7 Hz), 67.0, 49.6, 47.8, 36.1 (t, *J* = 20.9 Hz), (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, $CDCl_3$) δ -81.1 (t, *J* = 9.5 Hz, 3F), -112.7 – -112.9 (m, 2F), -113.7 – -113.8 (m, 1F), -124.4 – -124.5 (m, 2F), -126.0

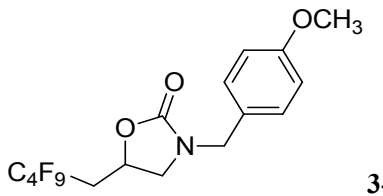
(t, J = 11.9 Hz, 2F); HRMS (ESI): $C_{15}H_{12}F_{10}NO_2^+$ for $[M + H]^+$ calculated 428.0703, found 428.0709.



3-(4-Chlorobenzyl)-5-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)oxazolidin-2-one (2f): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, J = 8.3 Hz, 2H), 7.22 (d, J = 8.2 Hz, 2H), 4.91 – 4.86 (m, 1H), 4.45 (d, J = 15.0 Hz, 1H), 4.34 (d, J = 15.0 Hz, 1H), 3.62 (t, J = 8.7 Hz, 1H), 3.18 (t, J = 8.7 Hz, 1H), 2.75 – 2.61 (m, 1H), 2.39 (ddd, J = 35.0, 17.3, 7.2 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.0, 134.3, 133.9, 129.7, 129.3, 67.0, 49.7, 47.8, 36.1 (t, J = 20.9 Hz), (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, $CDCl_3$) δ -81.0 (t, J = 8.8 Hz, 3F), -112.7 – -112.9 (m, 2F), -124.4 – -124.5 (m, 2F), -126.0 (t, J = 12.6 Hz, 2F); HRMS (ESI): $C_{15}H_{12}ClF_9NO_2^+$ for $[M + H]^+$ calculated 444.0407, found 444.0413.

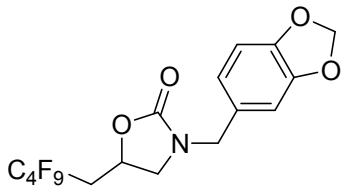


3-(4-Bromobenzyl)-5-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)oxazolidin-2-one (2g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, J = 8.1 Hz, 2H), 7.17 (d, J = 8.1 Hz, 2H), 4.91 – 4.84 (m, 1H), 4.44 (d, J = 14.9 Hz, 1H), 4.33 (d, J = 15.0 Hz, 1H), 3.62 (t, J = 8.7 Hz, 1H), 3.18 (t, J = 8.2 Hz, 1H), 2.69 (qd, J = 19.5, 5.4 Hz, 1H), 2.39 (ddd, J = 33.3, 17.3, 7.3 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.0, 134.4, 132.3, 130.0, 122.4, 67.0, 49.7, 47.9, 36.1 (t, J = 20.9 Hz), (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, $CDCl_3$) δ -81.0 (t, J = 9.2 Hz, 3F), -112.7 – -112.9 (m, 2F), -124.4 (s, 2F), -126.0 (t, J = 12.5 Hz, 2F); HRMS (ESI): $C_{15}H_{12}BrF_9NO_2^+$ for $[M + H]^+$ calculated 487.9902, 489.9882, found 487.9899, 489.9878.

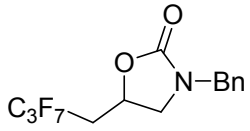


3-(4-Methoxybenzyl)-5-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)oxazolidin-2-one (2h): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (d, J = 8.1 Hz, 2H), 6.89 (d, J = 8.2 Hz, 2H), 4.88 – 4.81 (m, 1H), 4.41 (d, J = 14.7 Hz, 1H), 4.32 (d, J = 14.7 Hz, 1H),

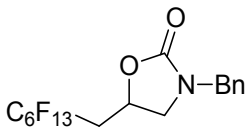
3.80 (s, 3H), 3.60 (t, J = 8.7 Hz, 1H), 3.16 (t, J = 8.2 Hz, 1H), 2.67 (td, J = 19.7, 5.2 Hz, 1H), 2.37 (ddd, J = 33.9, 17.3, 7.3 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.7, 157.0, 129.8, 127.3, 114.4, 66.9, 55.4, 49.6, 47.9, 36.3, 36.1 (t, J= 21.0 Hz), 35.9, (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, $CDCl_3$) δ -81.0 (t, J = 9.4 Hz, 3F), -112.7 (dd, J = 19.7, 9.3 Hz, 2F), -124.4 (dd, J = 12.4, 6.9 Hz, 2F), -125.9 (dd, J = 13.4, 10.8 Hz, 2F); HRMS (ESI): $C_{16}H_{15}F_9NO_3^+$ for $[M + H]^+$ calculated 440.0903, found 440.0895.



3-(Benzo[*d*][1,3]dioxol-5-ylmethyl)-5-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)oxazolidin-2-one (2i): ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.78 – 6.71 (m, 3H), 5.96 (s, 2H), 4.89 – 4.82 (m, 1H), 4.37 (d, J = 14.7 Hz, 1H), 4.27 (d, J = 14.7 Hz, 1H), 3.62 (t, J = 8.7 Hz, 1H), 3.17 (t, J = 8.2 Hz, 1H), 2.74 – 2.60 (m, 1H), 2.39 (ddd, J = 23.9, 17.3, 7.1 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.9, 148.4, 147.8, 129.1, 121.9, 108.7, 108.6, 101.4, 66.9, 49.6, 48.3, 36.1 (t, J = 21.1 Hz), (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (376 MHz, $CDCl_3$) δ -81.0 (t, J = 8.3 Hz, 3F), -112.7 – -112.8 (m, 2F), -124.5 (s, 2F), -126.0 (t, J = 12.6 Hz, 2F); HRMS (ESI): $C_{16}H_{13}F_9NO_4^+$ for $[M + H]^+$ calculated 454.0695, found 454.0694.

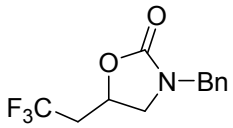


3-Benzyl-5-(2,2,3,3,4,4,4-heptafluorobutyl)oxazolidin-2-one (2j): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.28 (m, 5H), 4.92 – 4.85 (m, 1H), 4.51 (d, J = 14.8 Hz, 1H), 4.40 (d, J = 14.8 Hz, 1H), 3.65 (t, J = 8.7 Hz, 1H), 3.21 (dd, J = 9.0, 7.4 Hz, 1H), 2.76 – 2.61 (m, 1H), 2.40 (ddd, J = 34.5, 17.0, 7.1 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.0, 135.4, 129.1, 128.4, 128.3, 66.9, 49.7, 48.5, 36.0 (t, J = 21.2 Hz), (carbon peaks of $–C_3F_7$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, $CDCl_3$) δ -80.6 (t, J = 9.6 Hz, 3F), -113.5 – -113.6 (m, 2F), -127.7 – -127.8 (t, J = 18.5 Hz, 2F); HRMS (ESI): $C_{14}H_{13}F_7NO_2^+$ for $[M + H]^+$ calculated 360.0829, found 360.0834.

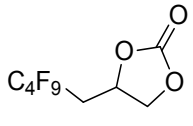


3-Benzyl-5-(2,2,3,3,4,4,5,5,6,6,7,7,7-tridecafluoroheptyl)oxazolidin-2-one (2k): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 5H), 4.94 – 4.81 (m, 1H), 4.50 (d, J = 14.8

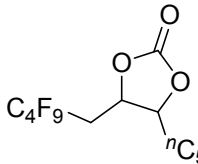
Hz, 1H), 4.38 (d, J = 14.7 Hz, 1H), 3.63 (t, J = 8.5 Hz, 1H), 3.19 (t, J = 7.8 Hz, 1H), 2.69 (q, J = 16.7 Hz, 1H), 2.46 – 2.32 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.0, 135.3, 129.1, 128.4, 128.3, 67.0, 49.7, 48.5, 36.2 (t, J = 21.2 Hz), (carbon peaks of $–C_6F_{13}$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, $CDCl_3$) δ -80.8 (t, J = 9.9 Hz, 3F), -112.4 – -112.6 (m, 2F), -121.8 (s, 2F), -122.8 (s, 2F), -123.5 (s, 2F), -126.1 (td, J = 14.8, 6.8 Hz, 2F); HRMS (ESI): $C_{17}H_{13}F_{13}NO_2^+$ for $[M + H]^+$ calculated 510.0733, found 510.0734.



3-benzyl-5-(2,2,2-trifluoroethyl)oxazolidin-2-one (2l): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.27 (m, 5H), 4.75 (dt, J = 14.3 Hz, 1H), 4.48 (d, J = 14.8 Hz, 1H), 4.38 (d, J = 14.9 Hz, 1H), 3.60 (t, J = 8.7 Hz, 1H), 3.24 - 3.11 (m, 1H), 2.78 – 2.58 (m, 1H), 2.49 – 2.31 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.1, 135.3, 129.1, 128.34, 128.29, 124.9 (q, J = 276.9 Hz), 67.4 (q, J = 3.3 Hz), 49.2, 48.4, 39.0 (q, J = 28.4 Hz); ^{19}F NMR (377 MHz, $CDCl_3$) δ -63.74 (t, J = 10.3 Hz, 3F); HRMS (ESI): $C_{12}H_{13}F_3NO_2^+$ for $[M + H]^+$ calculated 260.0893, found 260.0891.

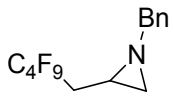


4-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-1,3-dioxolan-2-one (2m): ^{1}H NMR (400 MHz, DMSO-d_6) δ 5.18 (ddd, J = 16.4, 8.1, 3.0 Hz, 1H), 4.64 (t, J = 8.3 Hz, 1H), 4.22 (t, J = 8.0 Hz, 1H), 3.12 – 2.80 (m, 2H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 33.6 (t, J = 20.1 Hz), 68.6, 70.13, 154.4, (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, DMSO-d_6) δ -80.6 (dd, J = 11.6, 8.0 Hz, 3F), -111.2 (ddd, J = 27.6, 22.4, 9.4 Hz, 1F), -113.3 (ddd, J = 272.1, 18.5, 8.1 Hz, 1F), -123.2 – -124.7 (m, 2F), -125.2 – -126.4 (m, 2F); calcd. for $C_8H_6F_9O_3^+$ $[M + H]^+$ = 321.0168 Found: 321.0172.



4-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)-5-pentyl-1,3-dioxolan-2-one (2n): ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.08 (dd, J = 12.0, 7.5 Hz, 0.28 H), 4.76 (t, J = 8.7 Hz, 0.28 H), 4.65 (q, J = 5.9 Hz, 0.72 H), 4.41 (dd, J = 12.2, 6.0 Hz, 0.72 H), 2.67 (ddd, J = 35.2, 18.4, 6.2 Hz, 1 H), 2.46 (ddd, J = 22.4, 17.3, 5.8 Hz, 1 H), 1.81 – 1.34 (m, 8 H), 0.92 – 0.91 (m, 3 H); ^{13}C NMR (101

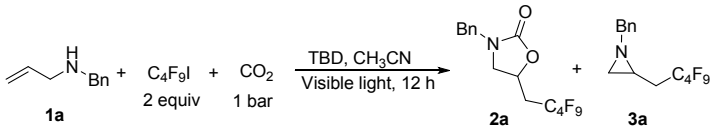
MHz, $CDCl_3$) δ 153.5, 82.0, 79.3, 74.6 (t, *J* = 2.7 Hz), 72.3 (d, *J* = 4.4 Hz), 35.4 (t, *J* = 21.4 Hz), 33.4, 31.33, 31.30, 31.0 (t, *J* = 21.6 Hz), 29.3, 25.1, 24.3, 22.5, 14.0, (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting);^{19}F NMR (377 MHz, $CDCl_3$) δ -81.0 (t, *J* = 9.3 Hz, 3F), -112.5 – -112.6 (m, 2F), -124.2 – 124.3 (m, 2F), -125.9 (t, *J* = 12.1 Hz); calcd. for $C_{13}H_{16}F_9O_3^+$ [M + H]$^+$ = 391.0950, Found: 391.0945



1-Benzyl-2-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)aziridine (3a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.23 (m, 5H), 3.63 (d, *J* = 13.3 Hz, 1H), 3.19 (d, *J* = 13.4 Hz, 1H), 2.31 (td, *J* = 19.3, 5.8 Hz, 2H), 1.84 (dd, *J* = 5.9, 3.2 Hz, 1H), 1.65 (d, *J* = 3.0 Hz, 1H), 1.53 (d, *J* = 6.3 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.1, 128.1, 128.0, 126.9, 109.0, 62.9, 34.1(t, *J* = 20.74 Hz), 31.8, 30.8 (t, *J* = 4.74 Hz), (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); GC-MS (EI, 70 eV) m/z (%) = 364.96 (6), 274.06 (100), 132.15 (27), 91 (41).

Control Experiments

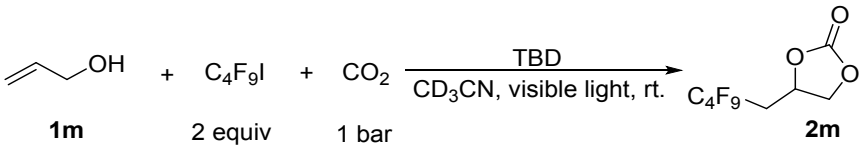
Table S1 Control experiments[a]



Entry	Base (equiv.)	Visible light (nm)	Yield of **2a** (%)[b]	Yield of **3a** (%)[b]
1	–	420-700	21	35
2	TBD (1.5)	420-700	92	trace
3[c]	TBD (1.5)	420-700	0	81

[a] Reaction conditions: **1a** (0.5 mmol), C_4F_9I (1.0 mmol), MeCN (2 mL), CO_2 (1 bar), rt.. [b] Isolated yield.
[c] Without CO_2, and Ar (1 bar).

^{1}H NMR investigation



TBD (0.0522 g, 0.375 mmol), allyl alcohol (0.0145 g, 0.25 mmol) and 1.2 mL deuterium acetonitrile were added to a 10 mL Schlenk tube equipped with a magnetic stir bar. Then the flask was subjected to three cycles of pressurization and depressurization with CO_2 using the "freeze–pump–thaw" method. In CO_2 atmosphere, perfluorobutyl iodide (86 μL, 0.5 mmol) was added and the contents were stirred under a 300 W Xe lamp (420 – 700nm). The reaction system was monitored with ^{1}H NMR spectroscopy at intervals. Before light output, most of allyl alcohol, TBD and CO_2 converted into allylic carbonate suspend in acetonitrile (signals of allylic carbonate emerged at δ = 6.03 - 5.92 (m, 1H), 5.24 (ddd, J = 17.3, 3.7, 1.8 Hz, 1H), 5.08 (ddd, J = 10.5, 3.4, 1.6 Hz, 1H), 4.34 (dt, J = 5.2, 1.5 Hz, 2H); signals of remainder allylic alcohol appeared at δ = 5.96 (ddt, J = 17.2, 10.2, 4.8 Hz, 1H), 5.22 (ddd, J = 17.2, 3.8, 1.8 Hz, 1H), 5.06 (ddd, J = 10.4, 3.4, 1.6 Hz, 1H), 4.04 (dt, J = 4.8, 1.7 Hz, 2H)) (Figure 1, 0 h). With light output, the solubility of allyl carbonate increased due to a little increase in reaction temperature, assigned to an enhancement on the intensity of ^{1}H NMR signal 4.34 ppm. Meanwhile, peaks corresponding to 4-nonafluoropentyl-ethylene carbonate **2m** appeared at δ = 5.17, 4.64, 4.22 ppm, indicating the start of this reaction (Figure 1, 1 h). As the reaction progressed at 4 h, a set of new signals emerged at δ = 4.31, 3.82, 3.67 ppm, assigned to the iodo-perfluoroalkylated carbonate intermediate **D**, which was corroborated indirectly in mass spectra (Figure S1). At 6 h, the raw materials converted completely, following a rapid

transformation from intermediate **D** to **2m** only in 10 min.

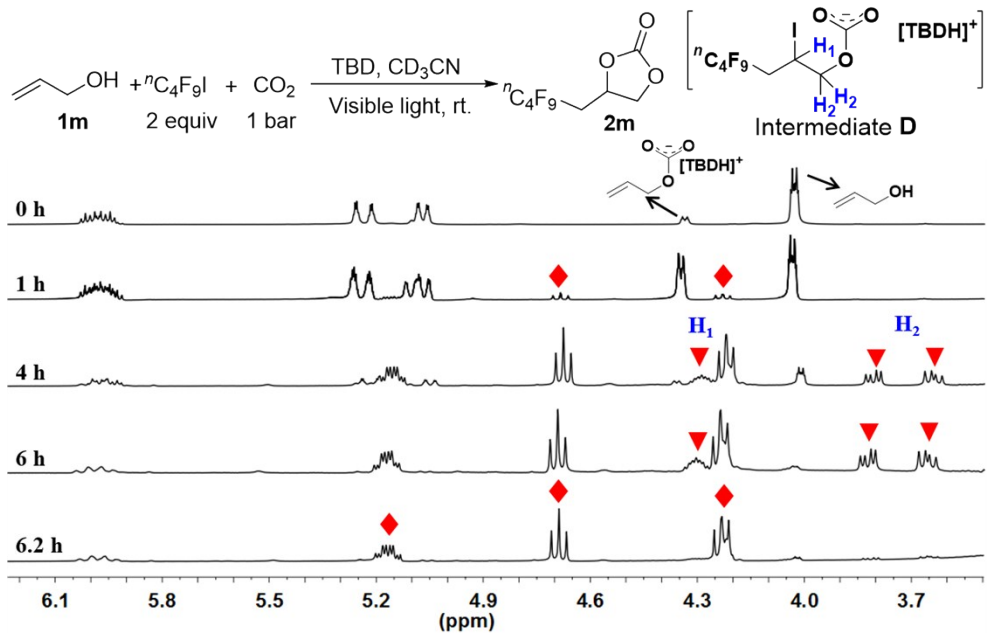
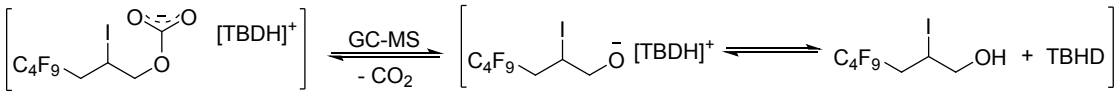
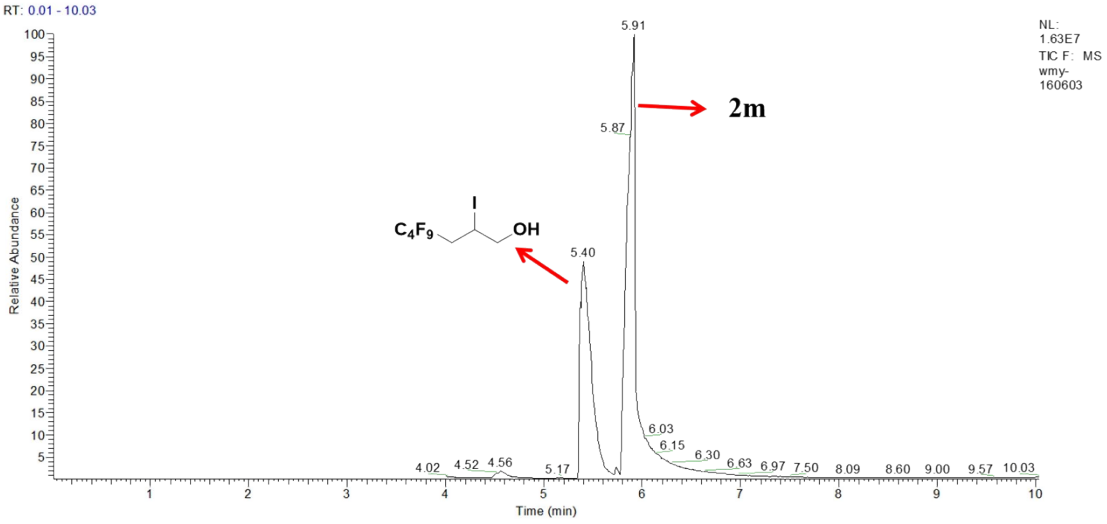


Fig. 1 ^{1}H NMR spectra for the visible light-driven carboxylative cyclization reaction of allyl alcohol **1m**, CO_2 and C_4F_9I in CD_3CN.

GC-MS (EI, 70 eV) m/z (%)

As the reaction progressed at 4 h, the crude reaction mixture was analyzed by GC-MS rapidly, as follows.





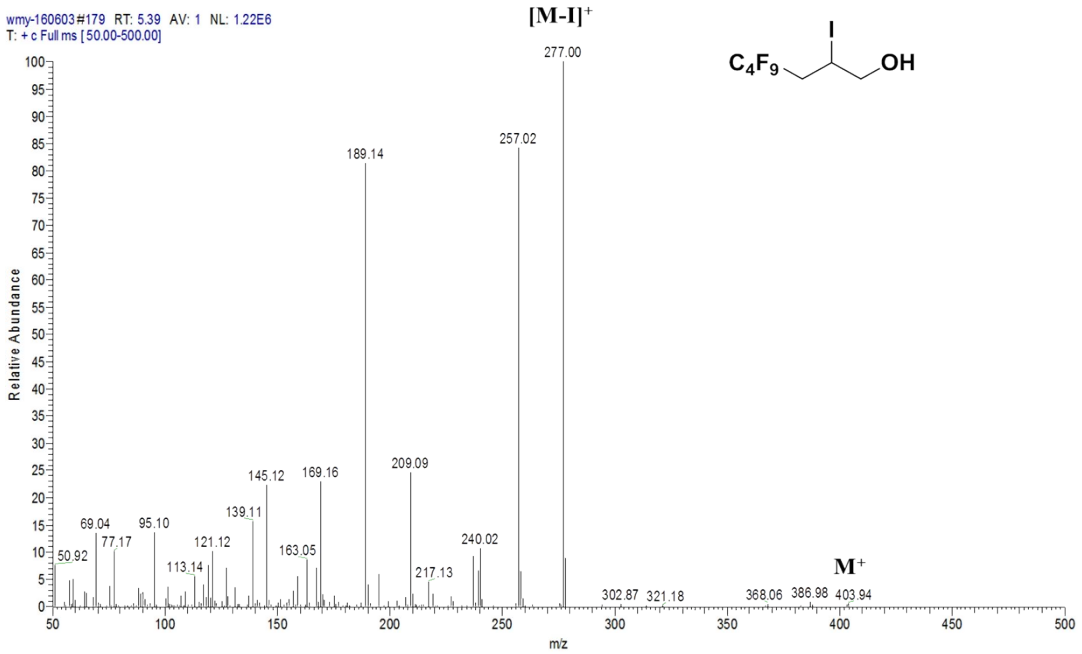
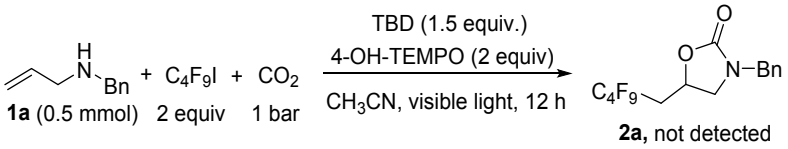
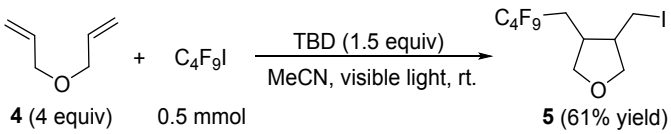


Fig. S1 GC-MS spectra for nonafluoro-2-iodoheptan-1-ol

Radical-Trapping Experiments

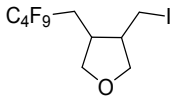


TBD (0.1044 g, 0.75 mmol) and 4-OH-TEMPO (0.1722 g, 1 mmol) were charged to the tube in a 100 mL steel autoclave. Then the autoclave was subjected to three cycles of pressurization and depressurization with CO_2. In CO_2 atmosphere, *N*-benzylprop-2-en-1-amine **1a** (0.0736 g, 0.5 mmol), MeCN (2 mL) and perfluoroalky iodide (172 μL, 1 mmol) were added and the contents were stirred for 12 h under a 300 W Xe lamp (420 nm – 700nm). Thereafter, the autoclave was cooled to room temperature and carefully depressurized. The crude reaction mixture was then analyzed by GC-MS.



TBD (0.1044 g, 0.75 mmol) was charged to the tube in a 100 mL steel autoclave. Then the autoclave was subjected to three cycles of pressurization and depressurization with CO_2. In CO_2 atmosphere, allyl ether **4** (0.1963 g, 2 mmol), MeCN (2 mL) and perfluoroalkyl iodide (86 μL, 0.5 mmol) were added and the contents were stirred for 12 h under a 300 W Xe lamp (420 nm – 700nm). Thereafter,

the autoclave was cooled to room temperature and carefully depressurized. The crude reaction mixture was purified by column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 25/1) to provide the colourless oil **5** (yield 61%).



5

3-(Iodomethyl)-4-(2,2,3,3,4,4,5,5,5-nonafluoropentyl)tetrahydrofuran (5): (dr = 50:11): ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.20 – 4.19 (m, 0.22H), 4.08 – 4.00 (m, 2.22H), 3.78 (dd, *J* = 9.1, 4.6 Hz, 1H), 3.67 (t, *J* = 8.2 Hz, 1H), 3.64 – 3.56 (m, 0.44H), 3.33 (dd, *J* = 10.0, 5.1 Hz, 0.22H), 3.21 (dd, *J* = 9.8, 5.3 Hz, 1H), 3.23 – 3.18 (t, *J* = 10.1 Hz, 0.22H), 3.11 (t, *J* = 10.1 Hz, 1H), 2.88 – 2.82 (m, 1H), 2.78 – 2.69 (m, 1H), 2.49 – 2.27 (m, 1.66H), 2.22 – 2.02 (m, 1.22H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 74.2 (d, *J* = 3.5 Hz), 73.9, 73.6, 71.8 (d, *J* = 3.9 Hz), 47.9, 45.2, 39.4, 35.8, 33.8 (t, *J* = 21.6 Hz), 28.3 (t, *J* = 21.8 Hz), 6.7, 2.8, (carbon peaks of $–C_4F_9$ are omitted due to complicated C-F splitting); ^{19}F NMR (377 MHz, $CDCl_3$) δ -81.0 – -81.2 (m, 3F), -112.5 – -115.2 (m, 2F), -124.36 (s, 2F), -125.9 – -126.0 (m, 2F); HRMS (ESI): $C_{10}H_{11}F_9IO$ for $[M + H]^+$ calculated 444.9705, found 444.9702.

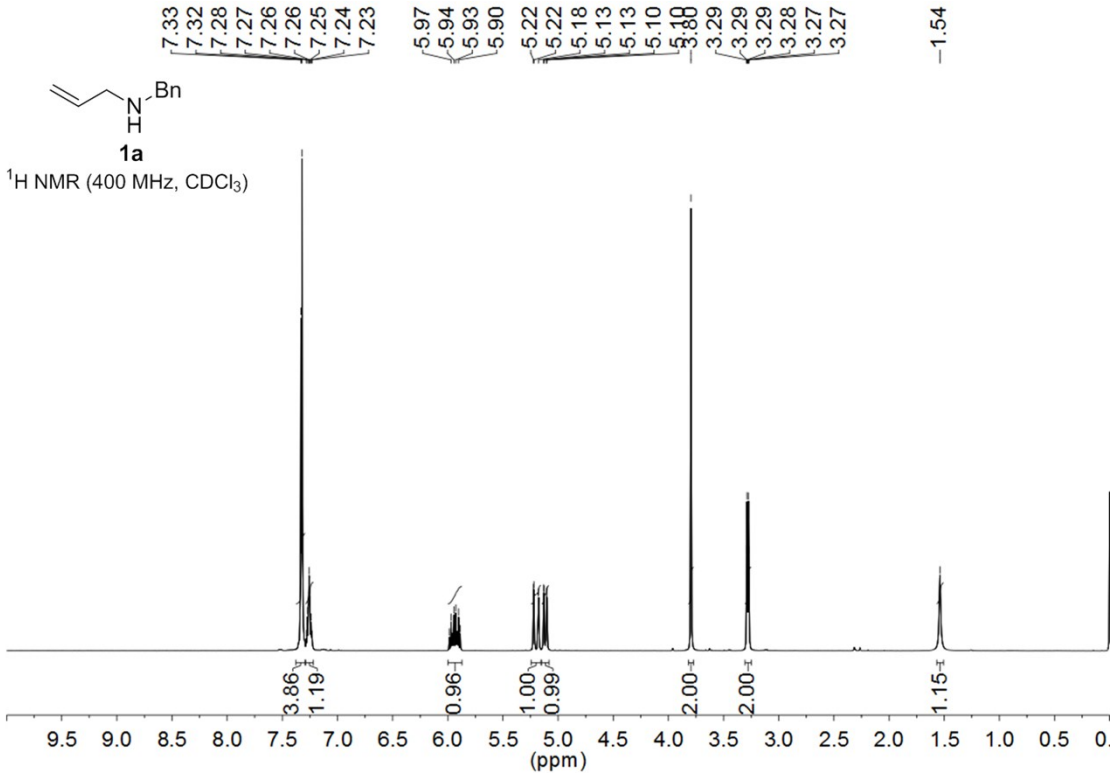
References

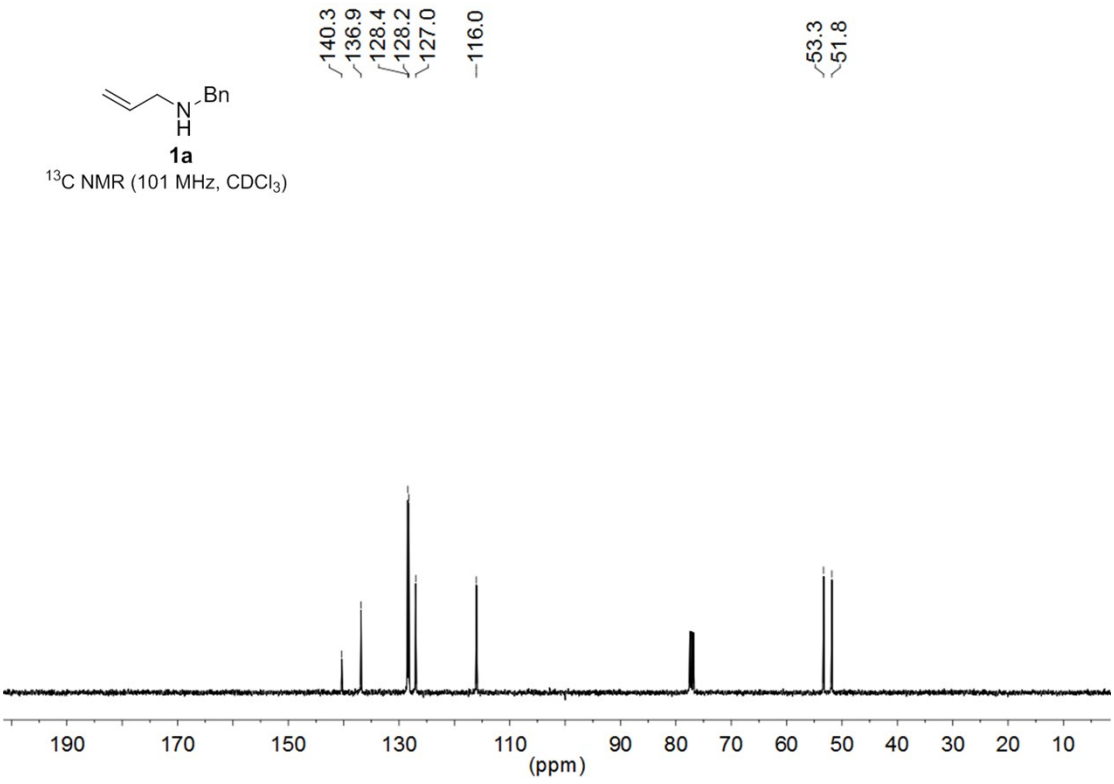
1. Makino, T.; Itoh, K. *J. Org. Chem.* **2004**, 69, 395-405.

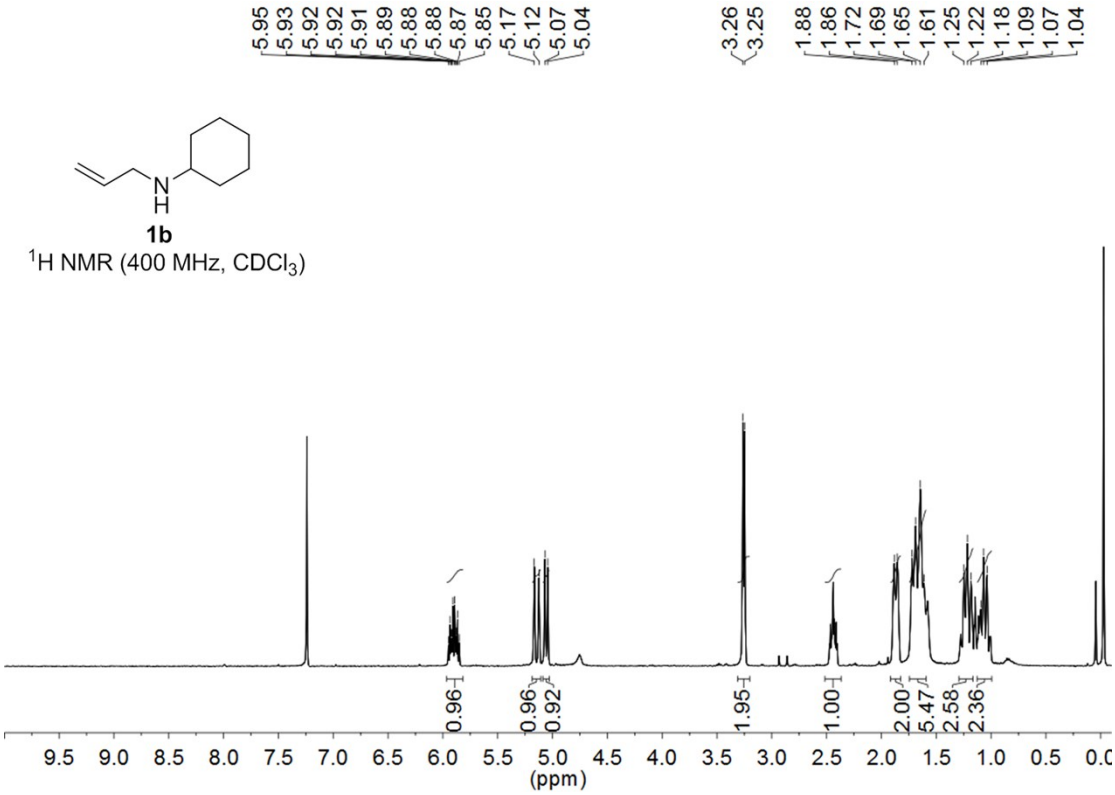
2. Gill, G. B.; Pattenden, G.; Reynolds, S. J. *J. Chem. Soc. Perkin Trans.*1 **1994**, 369-378.

3. a) Wang, C. M.; Guo, L. P.; Li, H. R.; Wang, Y.; Weng, J. Y.; Wu, L. H. *Green Chem.* **2006**, 8, 603; b) Wang, C. M.; Zhao, W. J.; Li, H. R.; Guo, L. P. *Green Chem.* **2009**, 11, 843.

^{1}H NMR, ^{13}C NMR and ^{19}F NMR

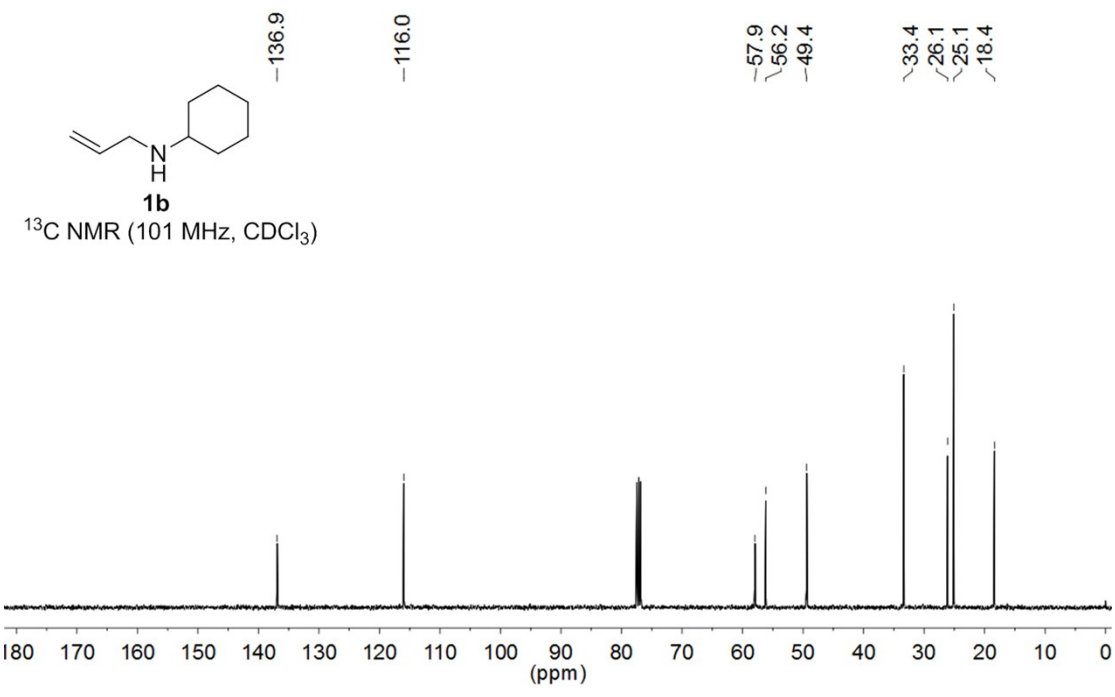






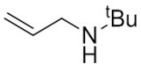
1b

^{1}H NMR (400 MHz, $CDCl_3$)



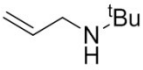
1b

^{13}C NMR (101 MHz, $CDCl_3$)



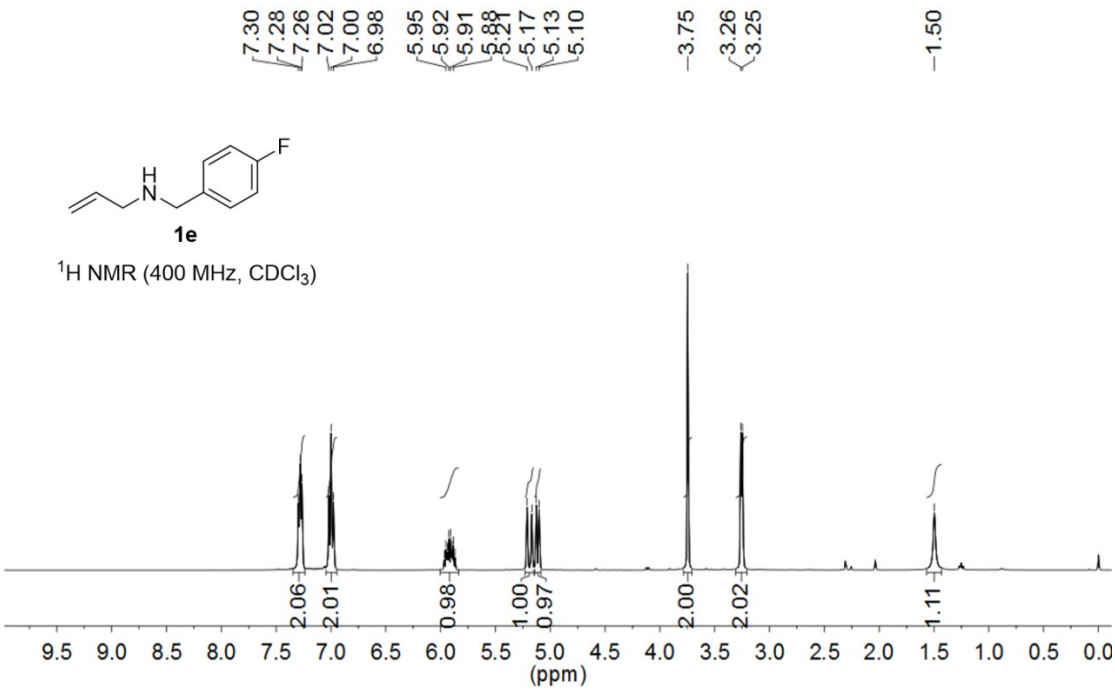
1d

^{1}H NMR (400 MHz, $CDCl_3$)



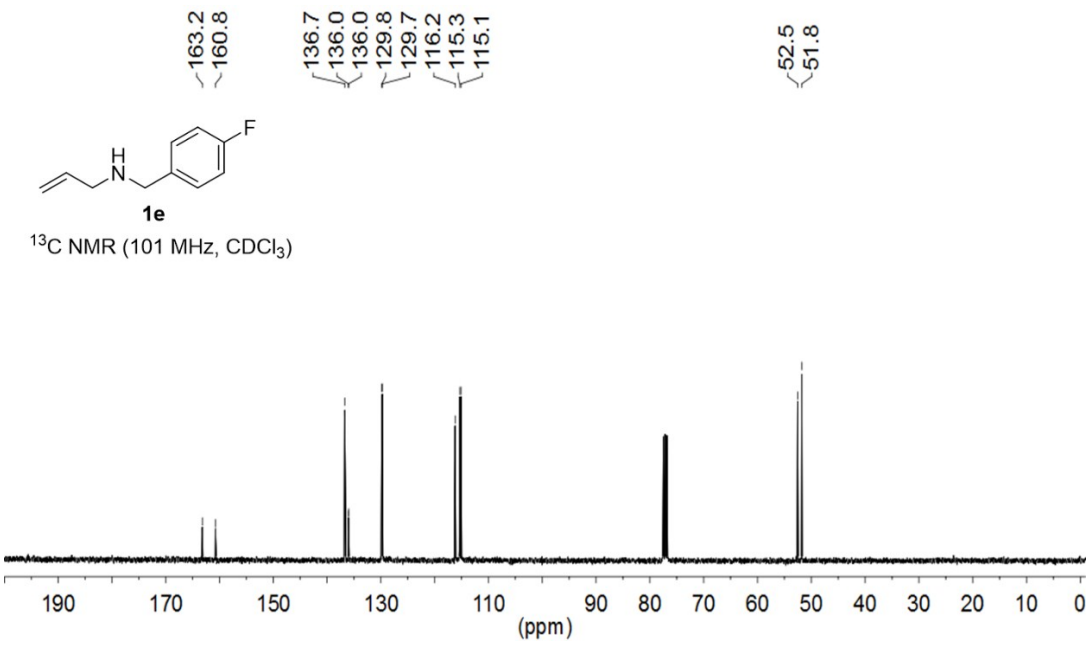
1d

^{13}C NMR (101 MHz, $CDCl_3$)



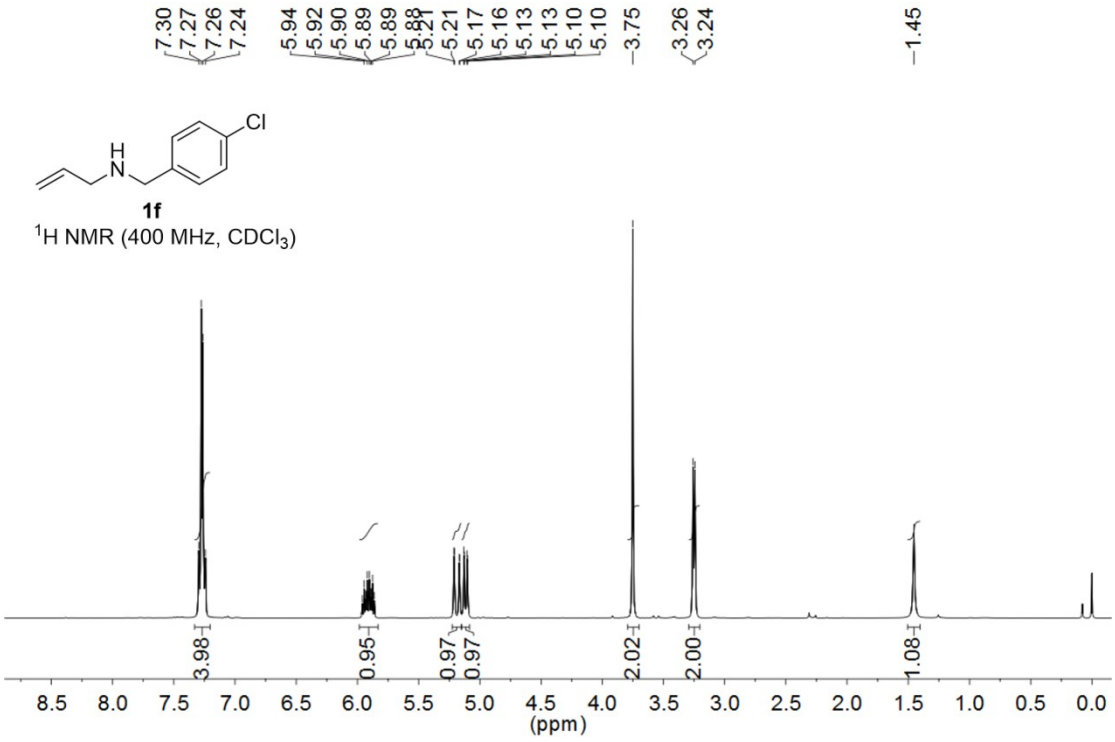
1e

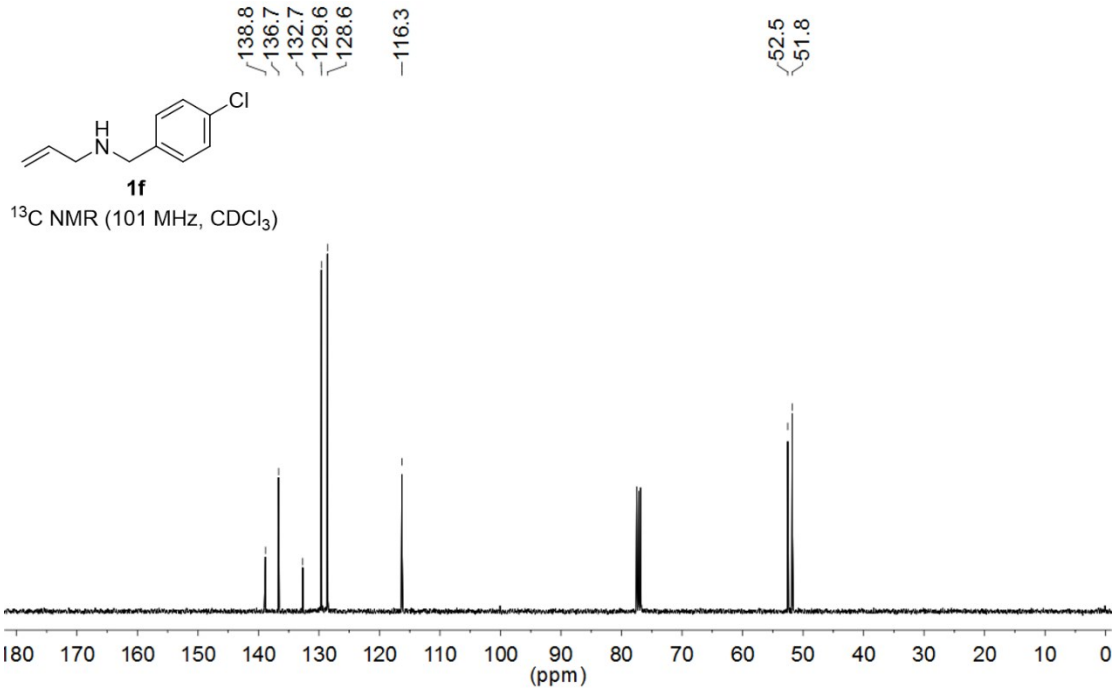
^{1}H NMR (400 MHz, $CDCl_3$)

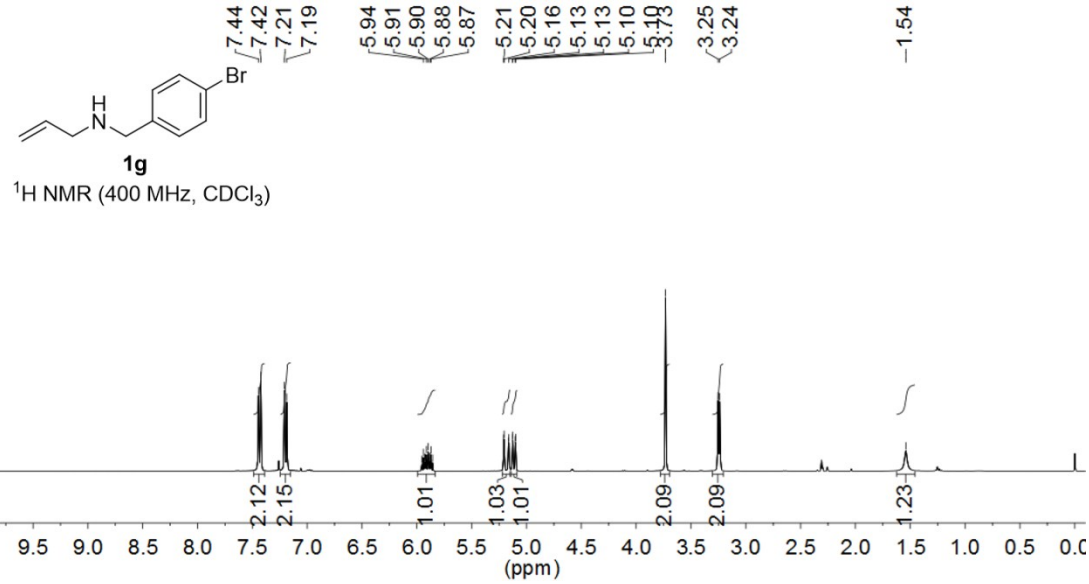


1e

^{13}C NMR (101 MHz, $CDCl_3$)

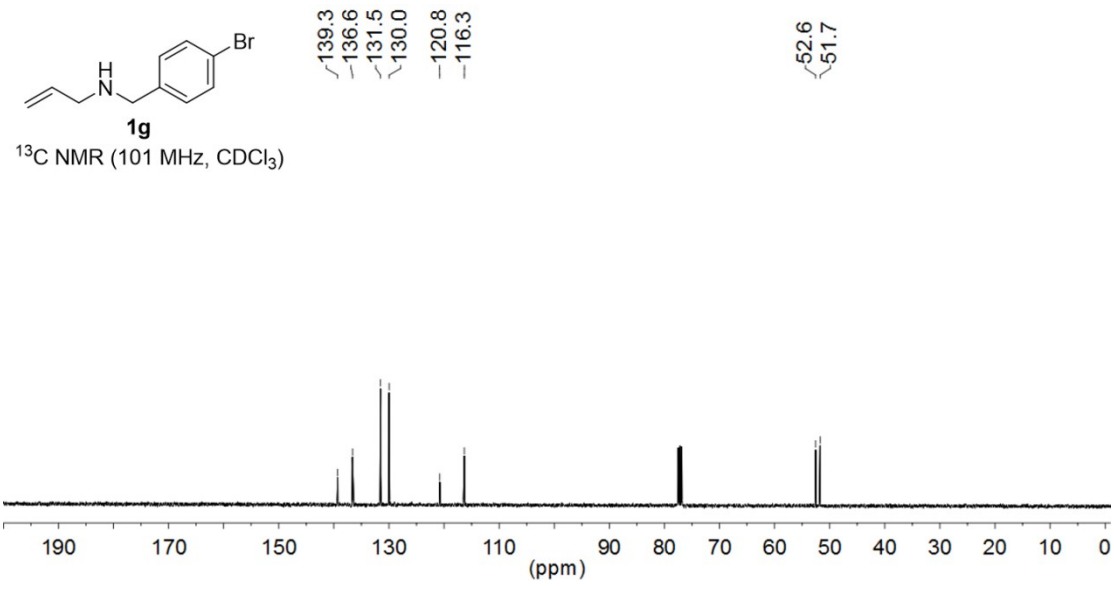

7.30
7.27
7.26
7.24
5.94
5.92
5.90
5.89
5.89
5.88
5.21
5.21
5.17
5.16
5.13
5.13
5.10
5.10
3.75
3.26
3.24
1.45
Cl
H
N
1f
1H NMR (400 MHz, CDCl3)
3.98
0.95
0.97
0.97
2.02
2.00
1.08
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)


138.8
136.7
132.7
129.6
128.6
116.3
52.5
51.8
Cl
H
N
1f
13C NMR (101 MHz, CDCl3)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)



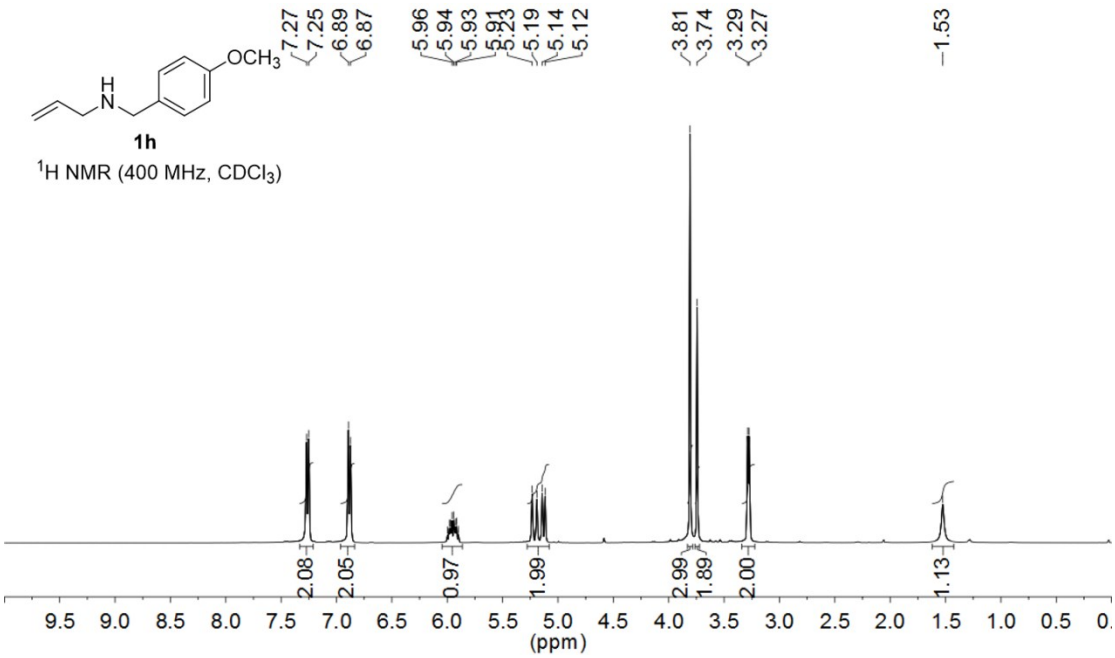
1g

^{1}H NMR (400 MHz, $CDCl_3$)



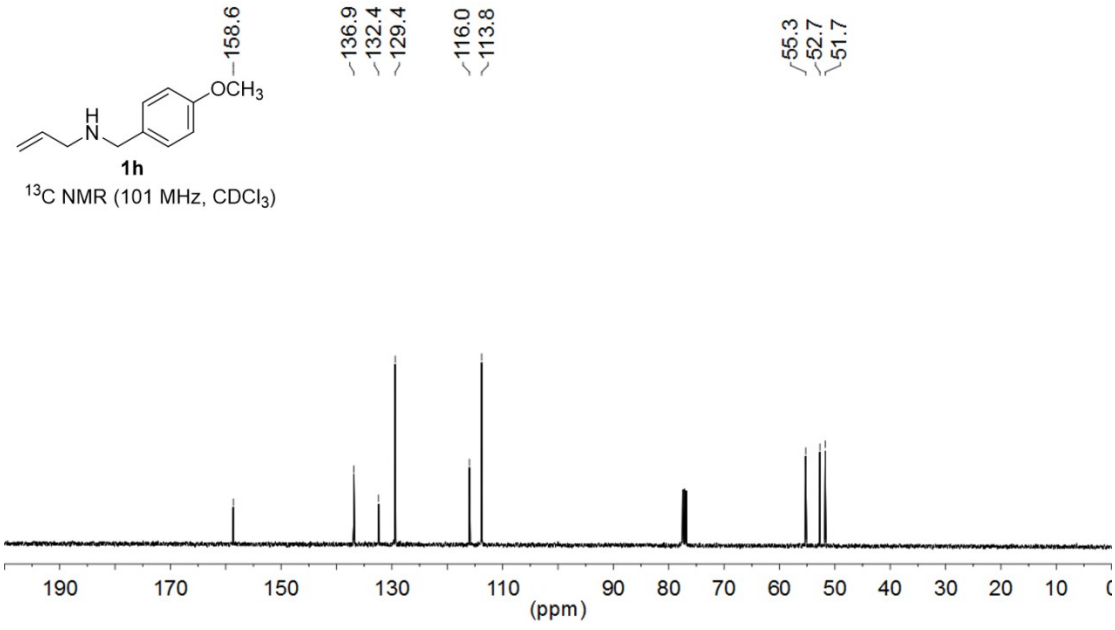
1g

^{13}C NMR (101 MHz, $CDCl_3$)



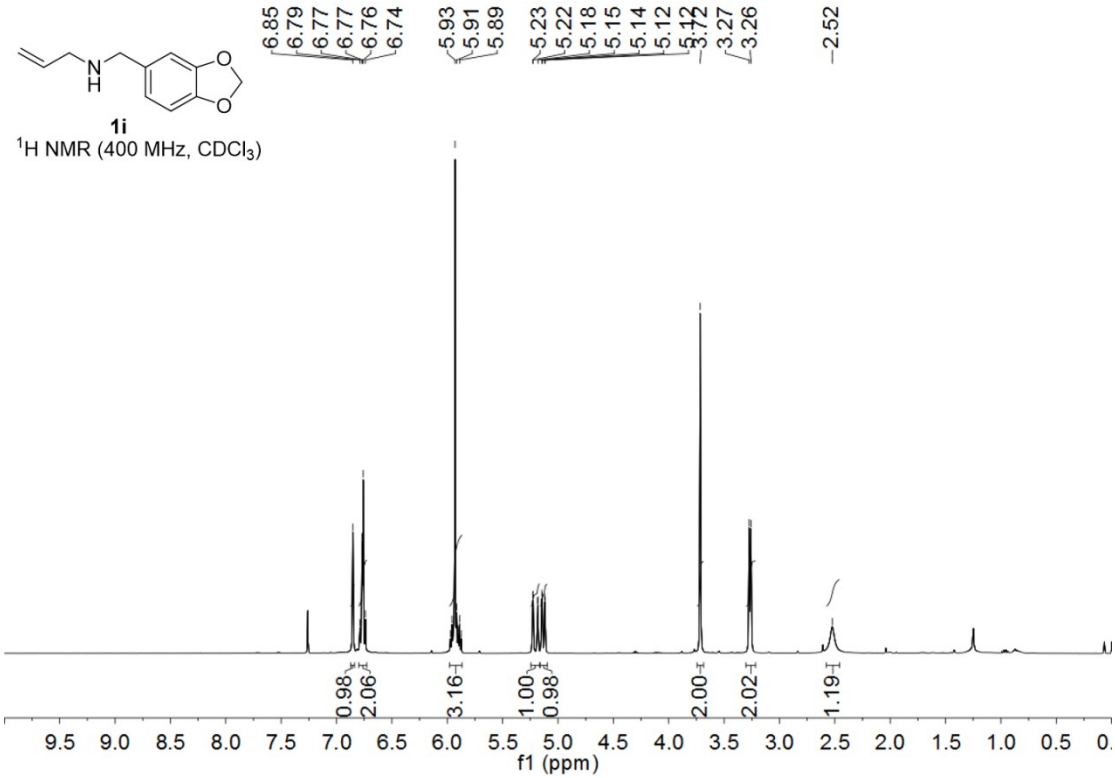
1h

^{1}H NMR (400 MHz, $CDCl_3$)



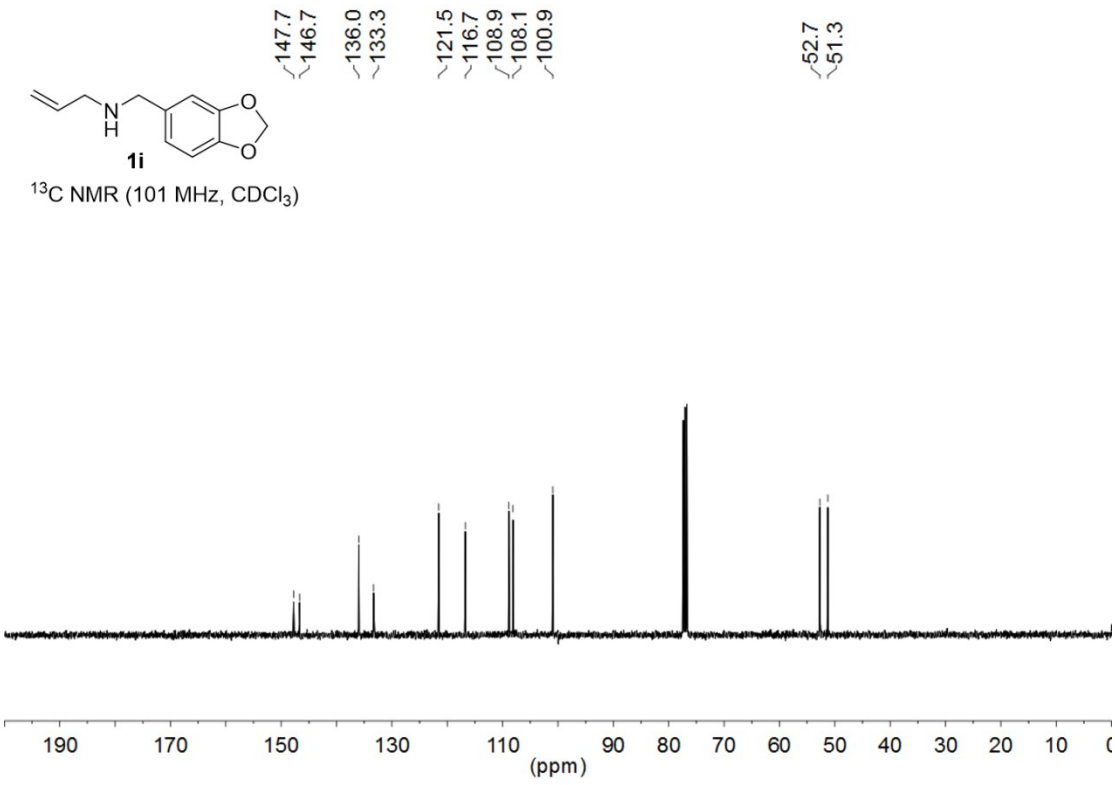
1h

^{13}C NMR (101 MHz, $CDCl_3$)



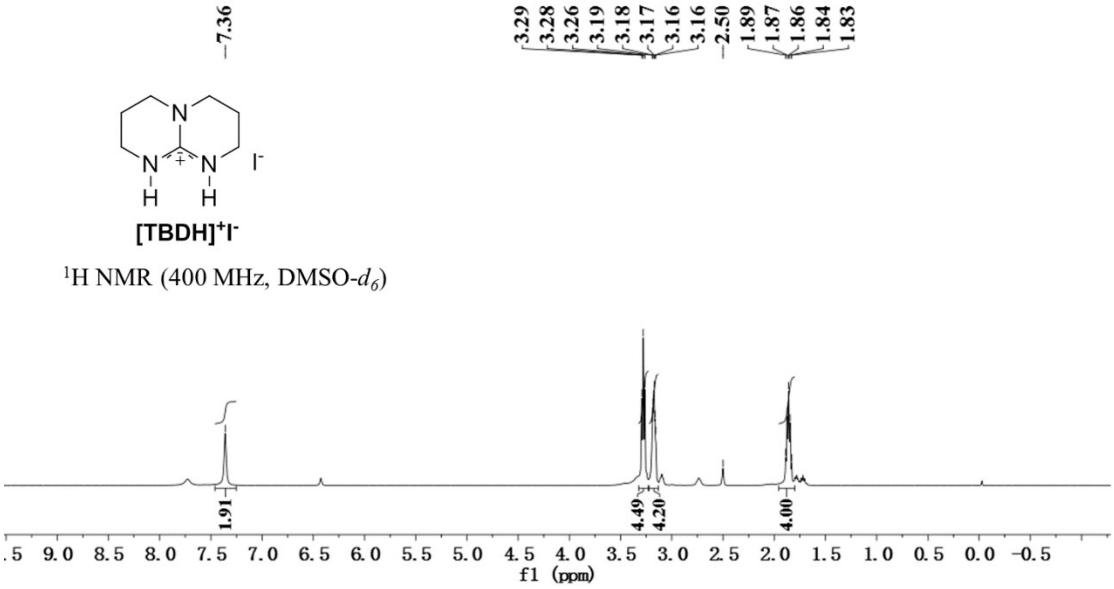
1i

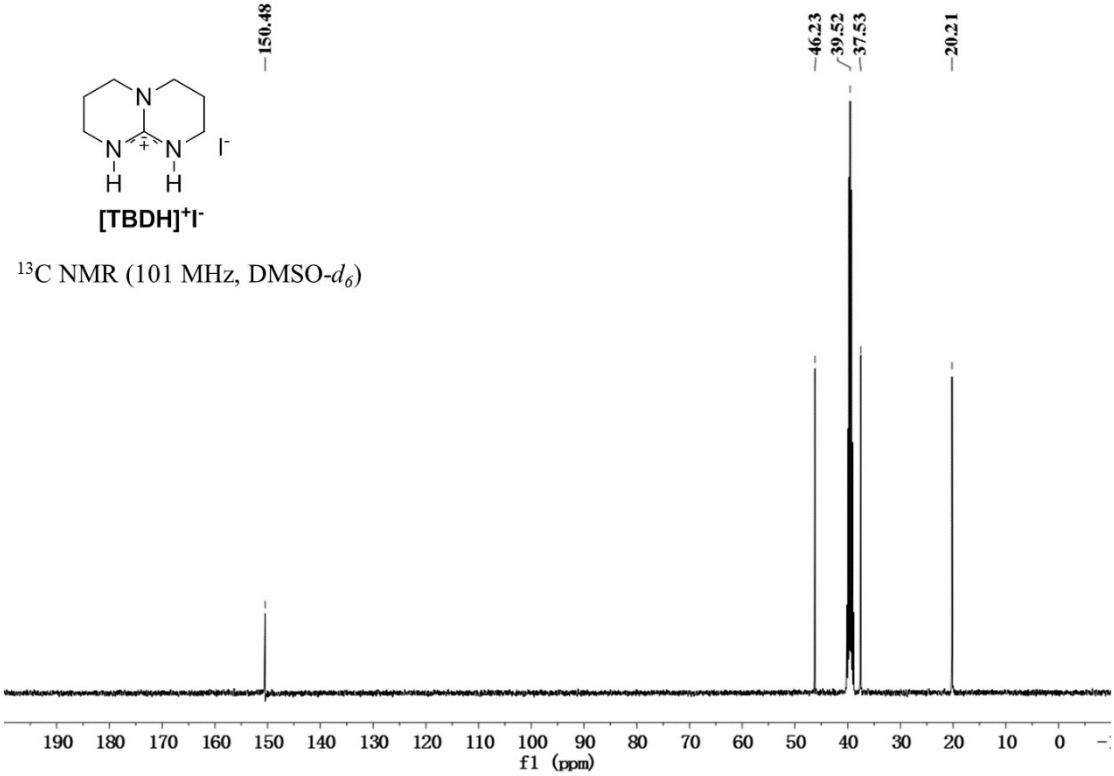
^{1}H NMR (400 MHz, $CDCl_3$)

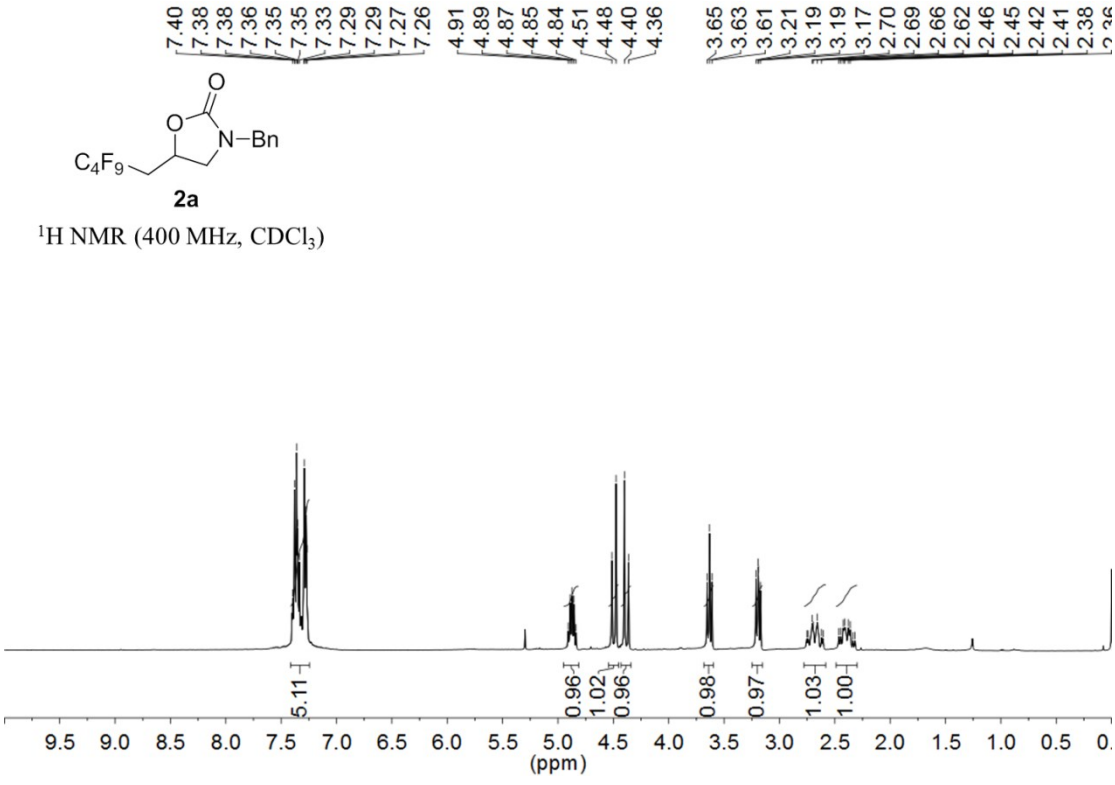


1i

^{13}C NMR (101 MHz, $CDCl_3$)

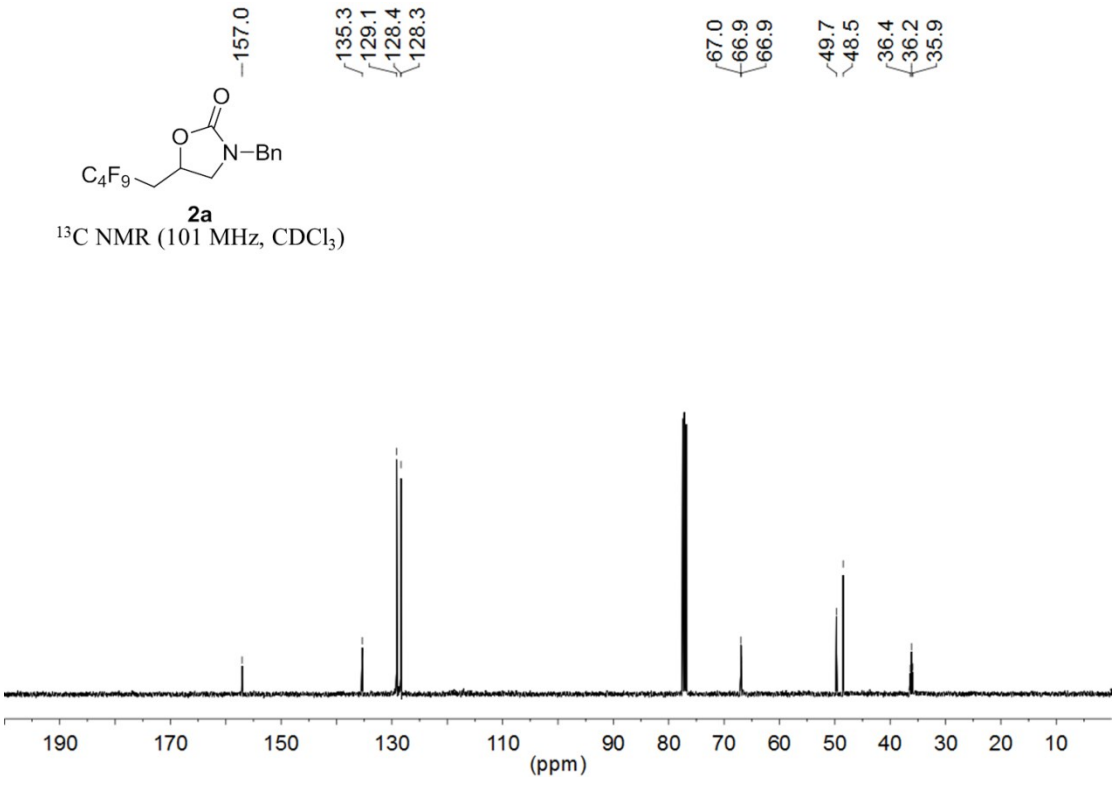

7.36
3.29
3.28
3.26
3.19
3.18
3.17
3.16
3.16
2.50
1.89
1.87
1.86
1.84
1.83
N
N
H
N
H
I-
[TBDH]+I-
1H NMR (400 MHz, DMSO-d6)
1.91
4.49
4.20
4.00
f1 (ppm)


150.48
46.23
39.52
37.53
20.21
N
N
H
N
H
I-
[TBDH]+I-
13C NMR (101 MHz, DMSO-d6)
f1 (ppm)



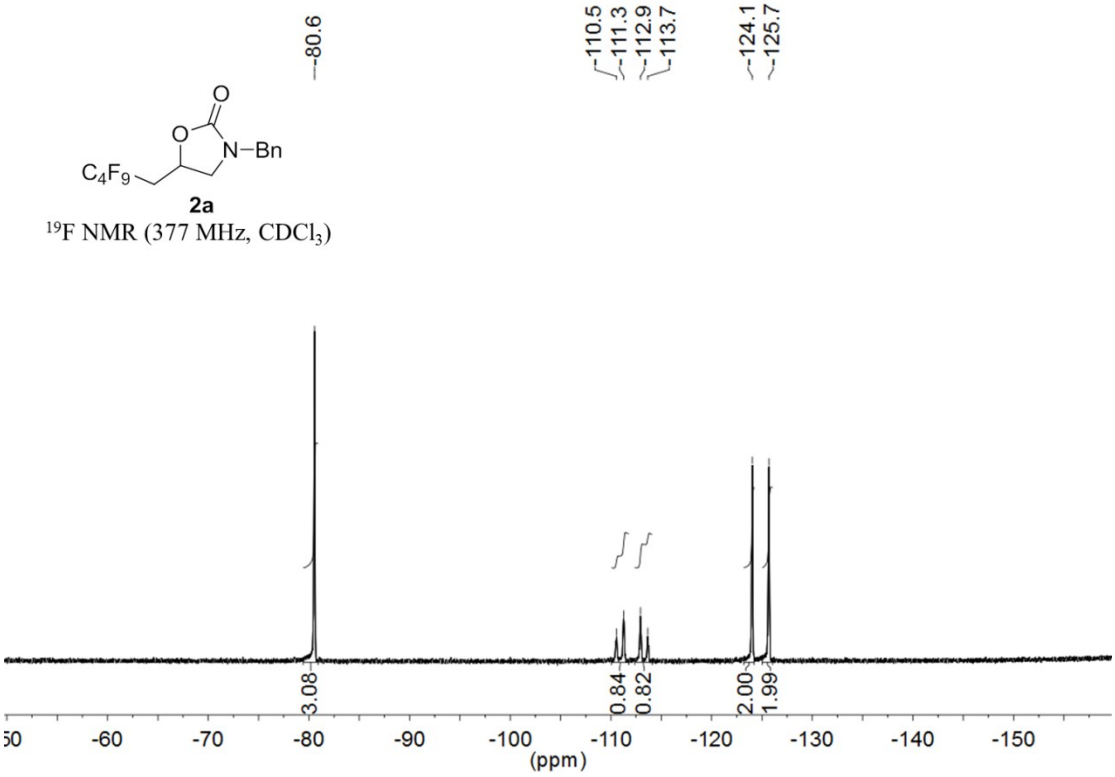
2a

^{1}H NMR (400 MHz, $CDCl_3$)



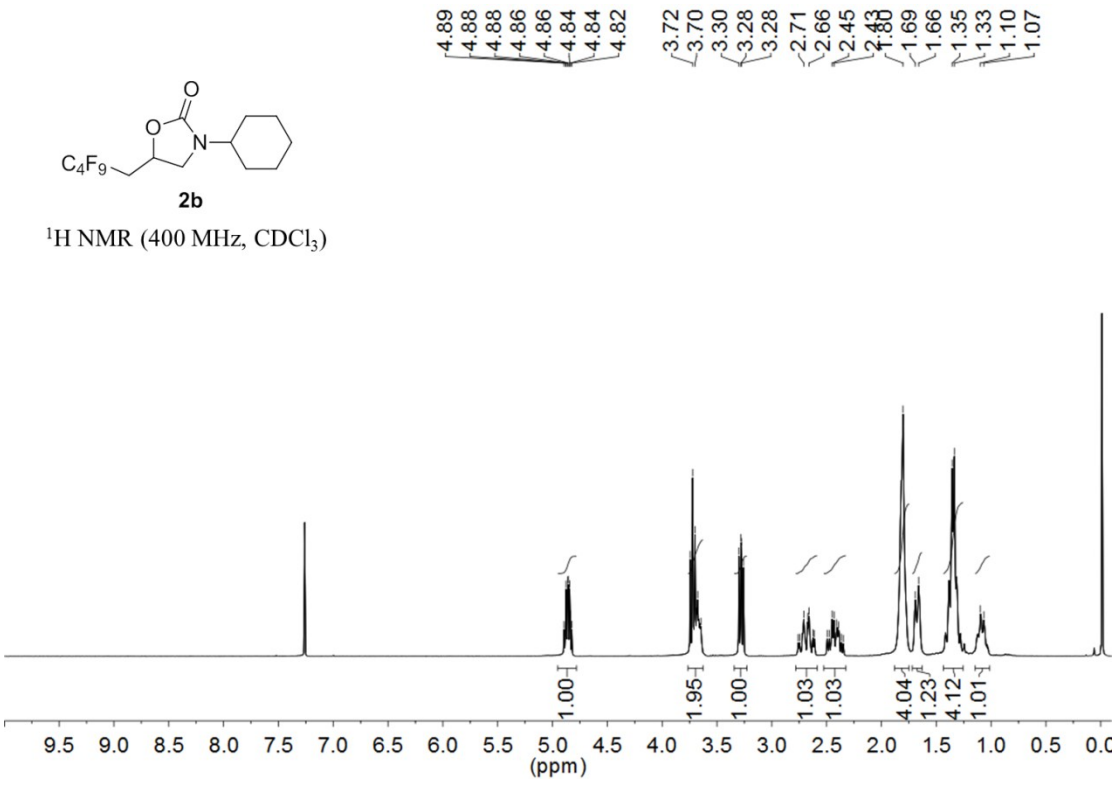
2a

^{13}C NMR (101 MHz, $CDCl_3$)



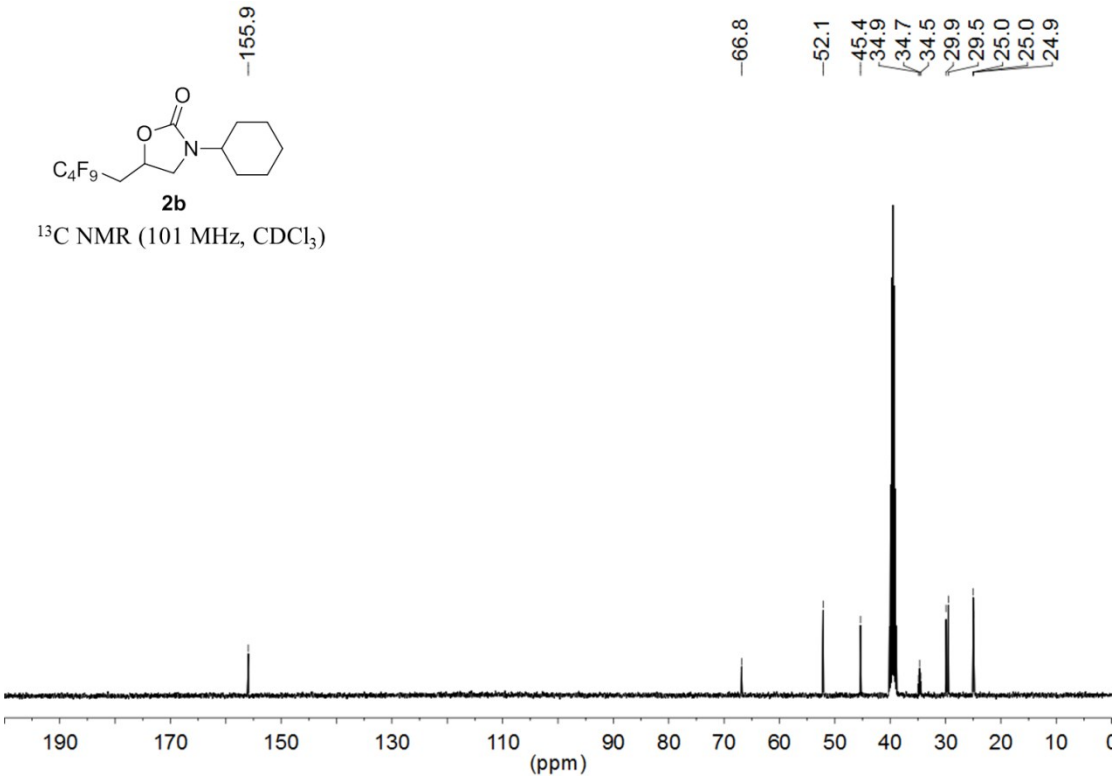
2a

^{19}F NMR (377 MHz, $CDCl_3$)

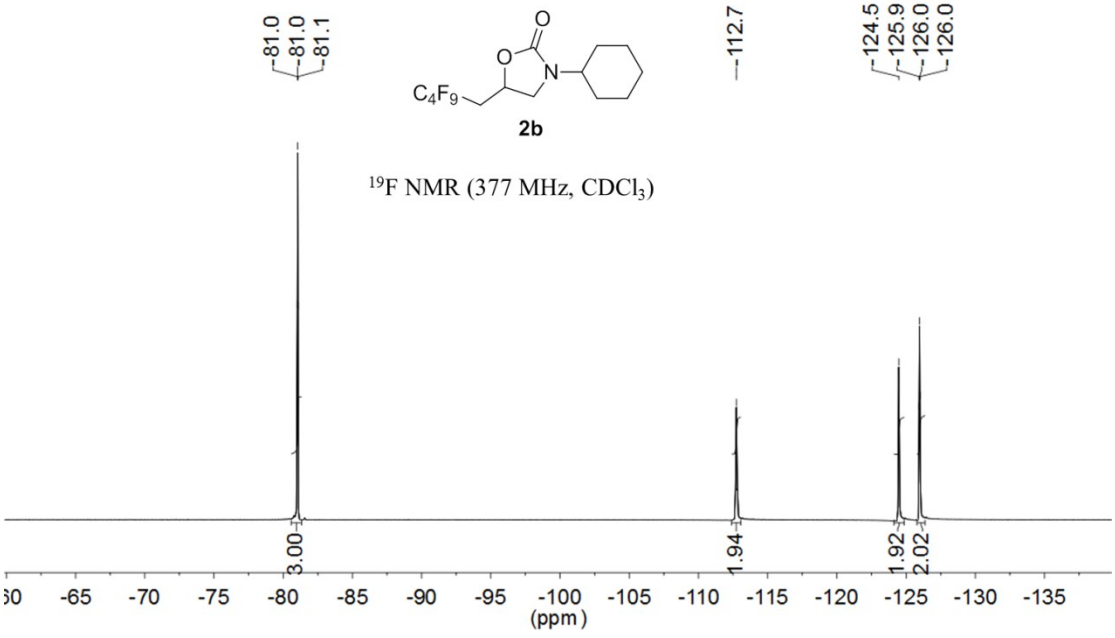


2b

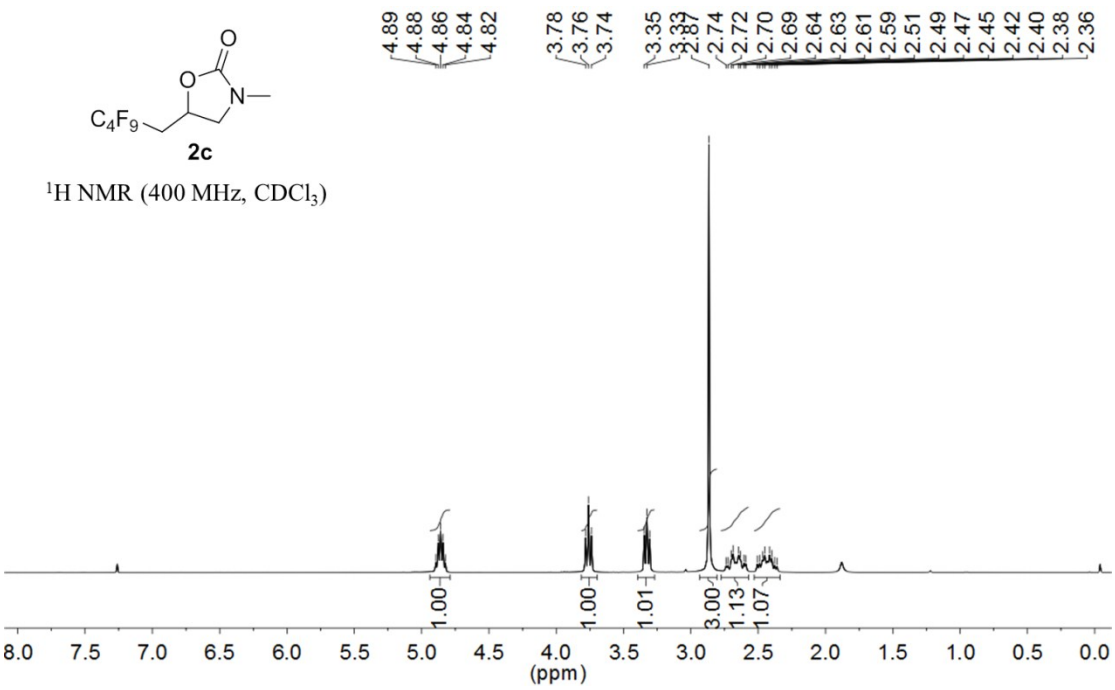
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)

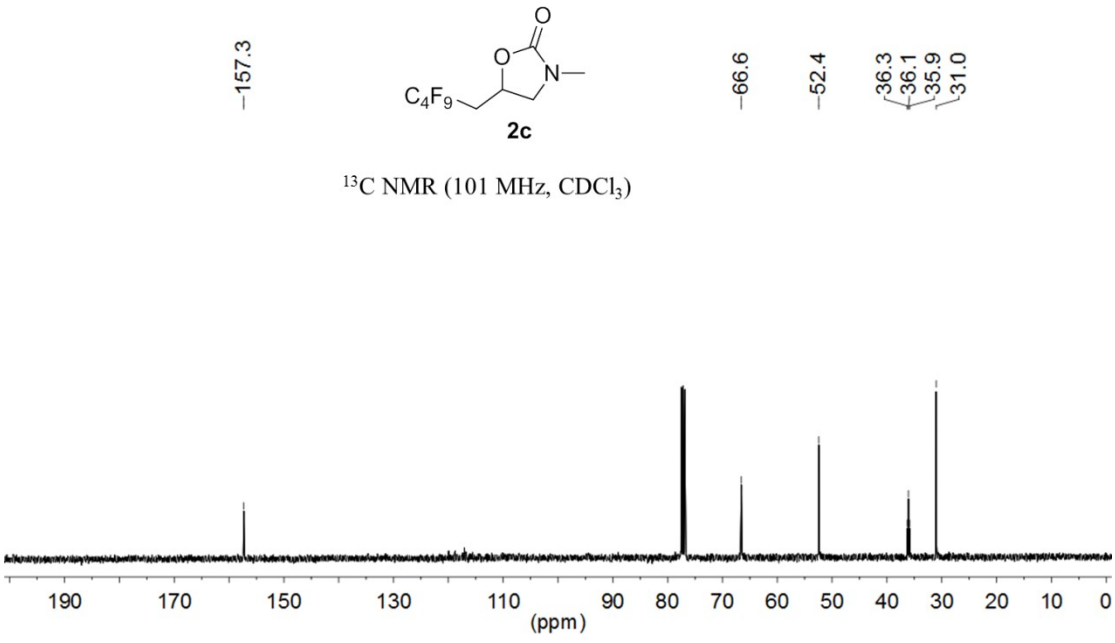


^{19}F NMR (377 MHz, $CDCl_3$)



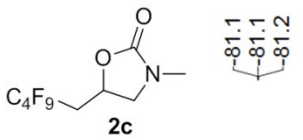
2c

^{1}H NMR (400 MHz, $CDCl_3$)



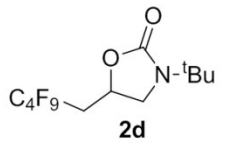
2c

^{13}C NMR (101 MHz, $CDCl_3$)



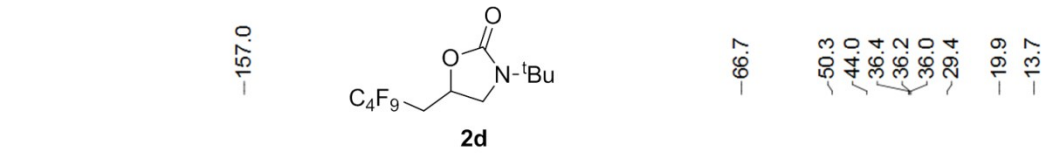
2c

^{19}F NMR (377 MHz, $CDCl_3$)



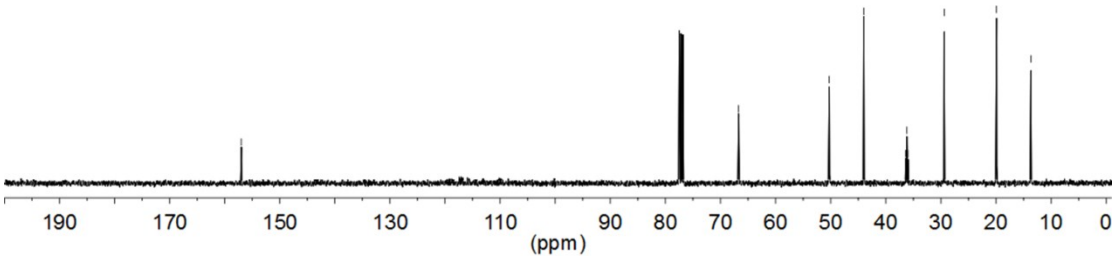
2d

^1H NMR (400 MHz, $CDCl_3$)



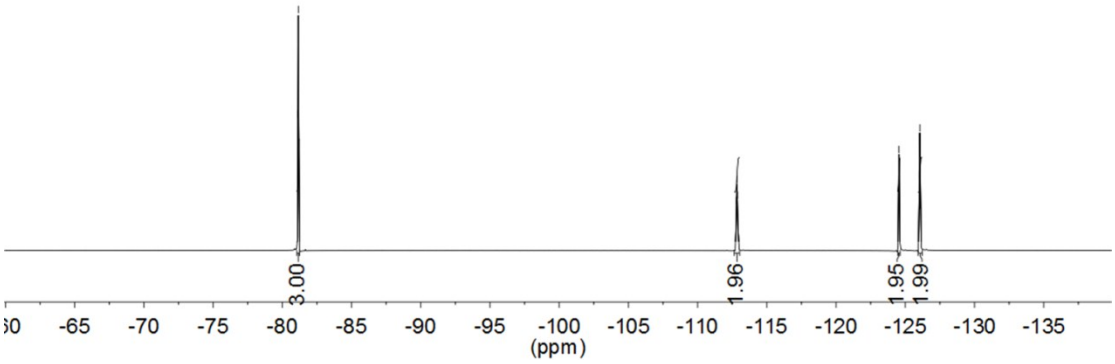
2d

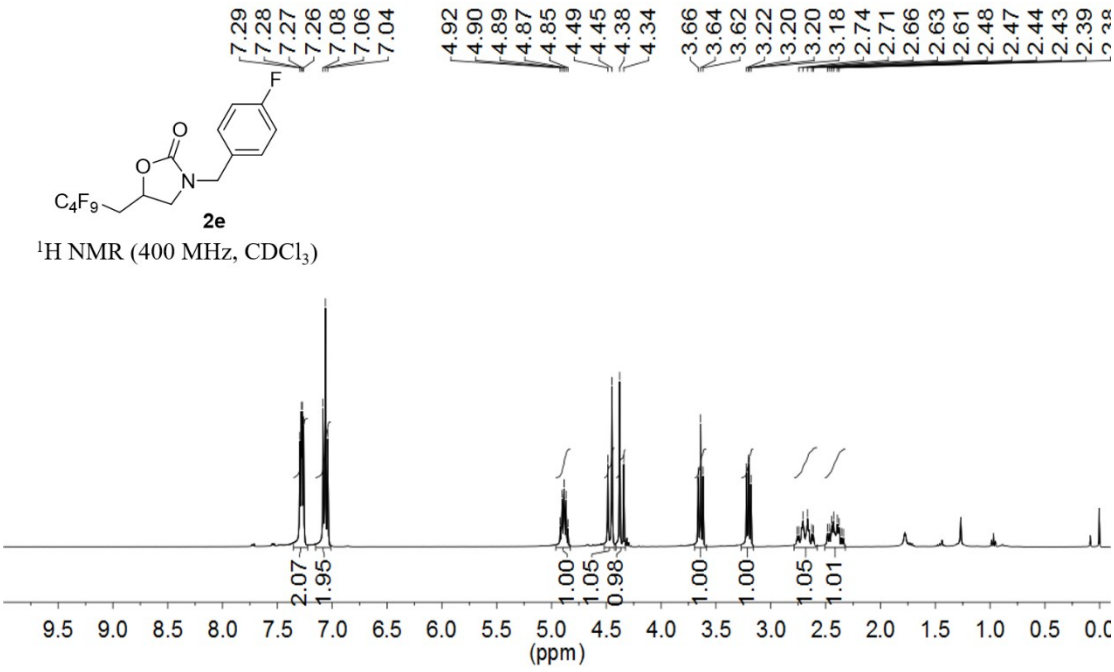
^{13}C NMR (101 MHz, $CDCl_3$)



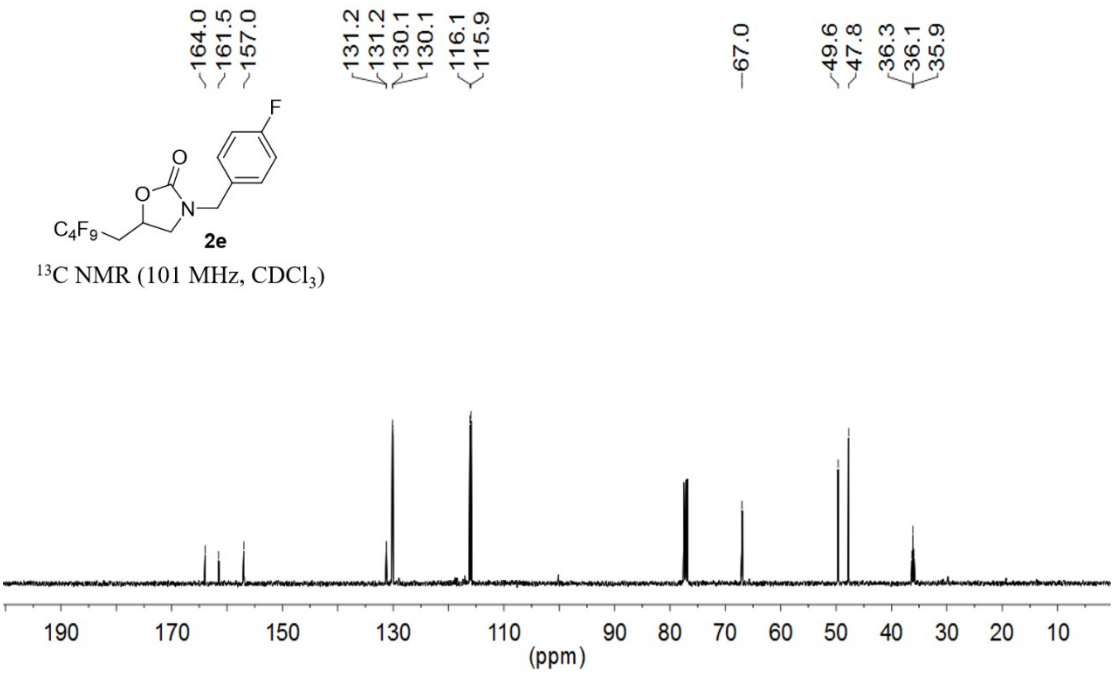
2d

^{19}F NMR (377 MHz, $CDCl_3$)

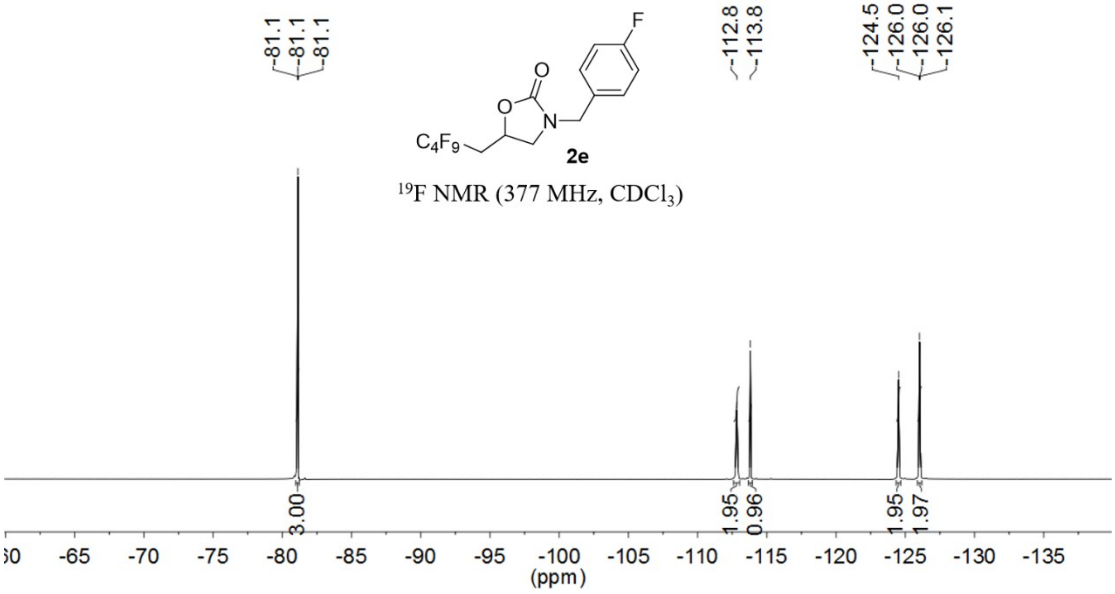




^{1}H NMR (400 MHz, $CDCl_3$)

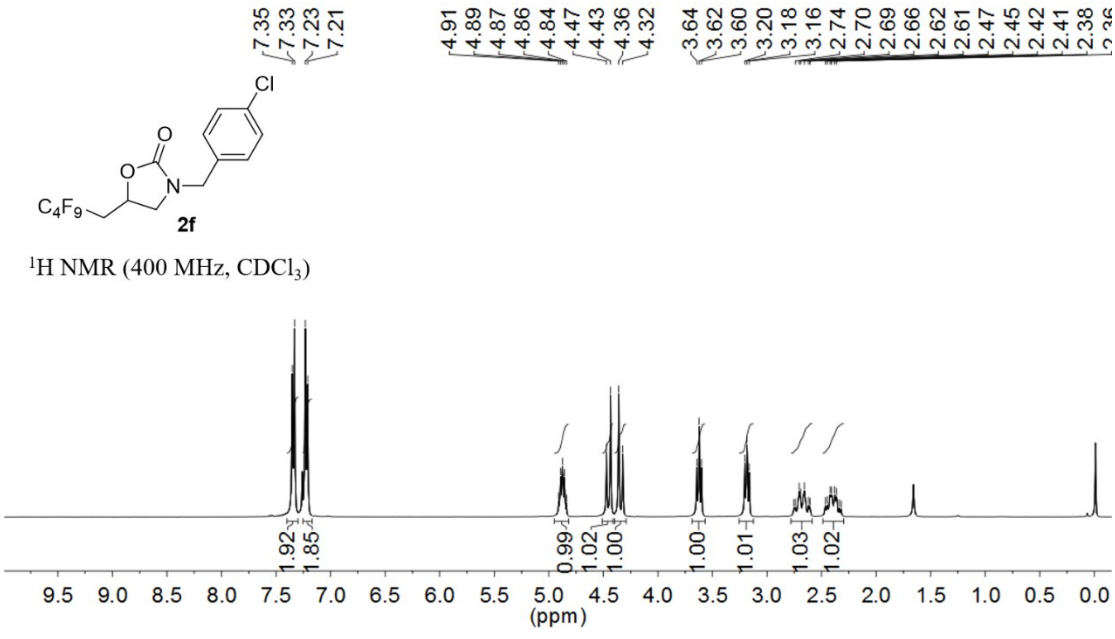


^{13}C NMR (101 MHz, $CDCl_3$)



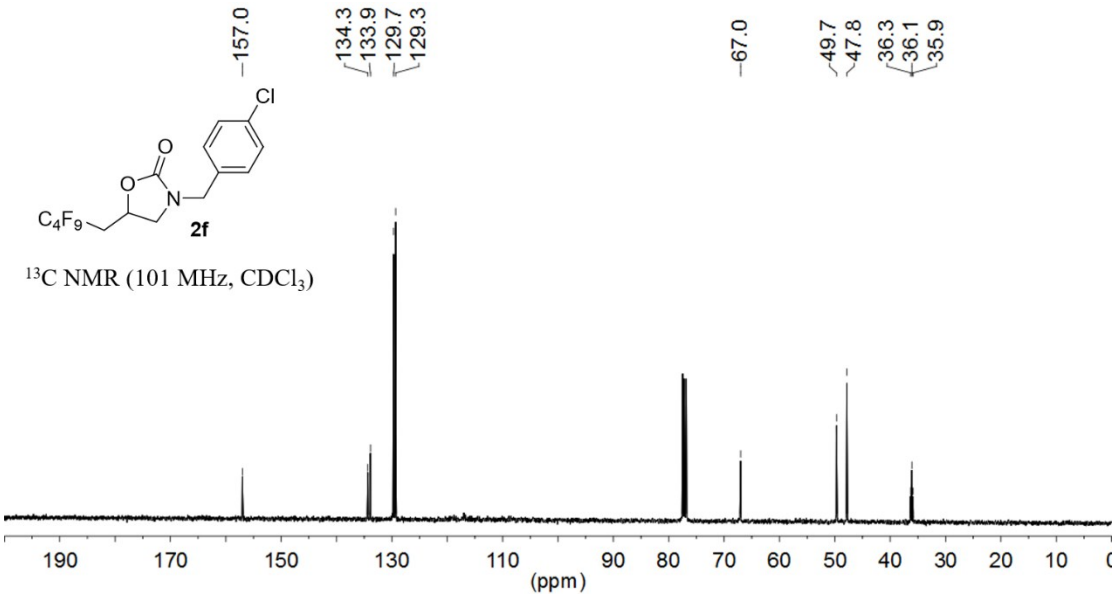
2e

^{19}F NMR (377 MHz, $CDCl_3$)

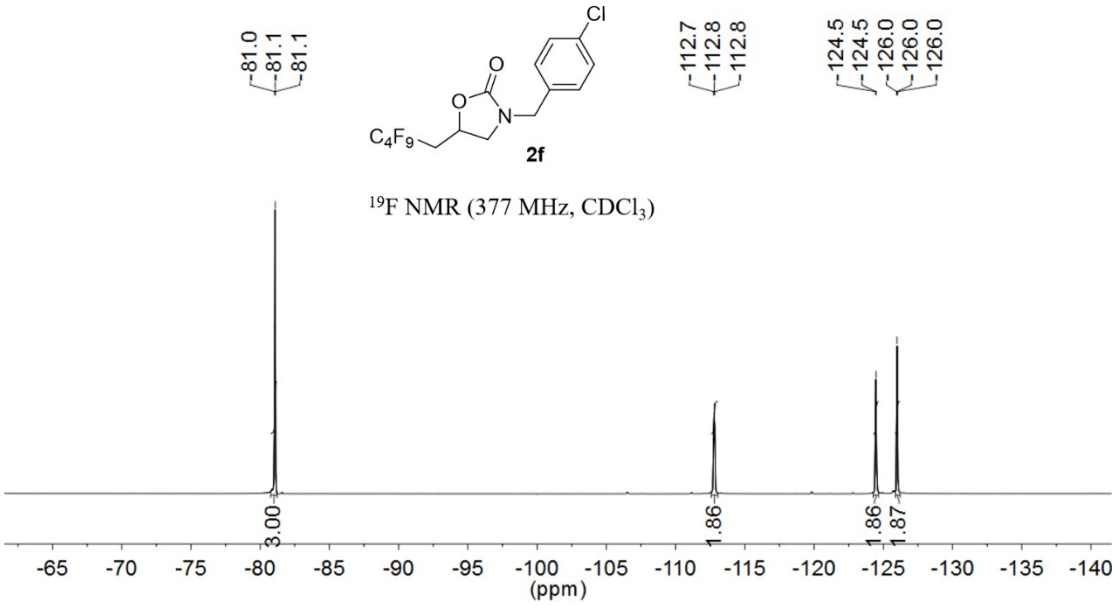


2f

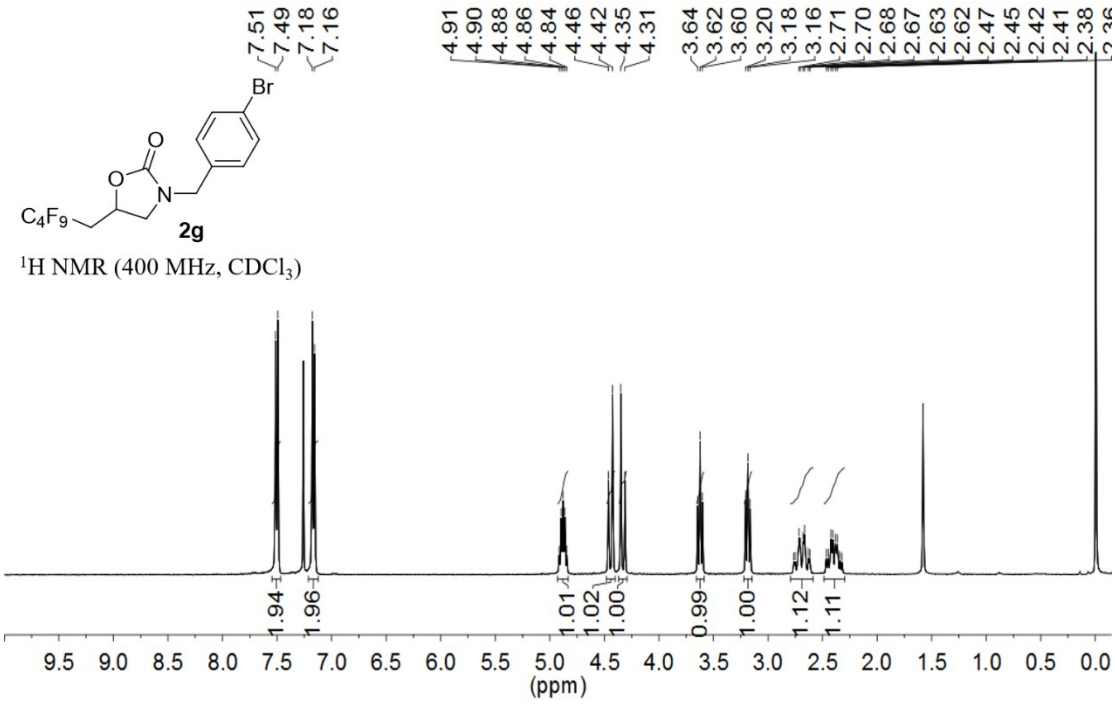
^{1}H NMR (400 MHz, $CDCl_3$)



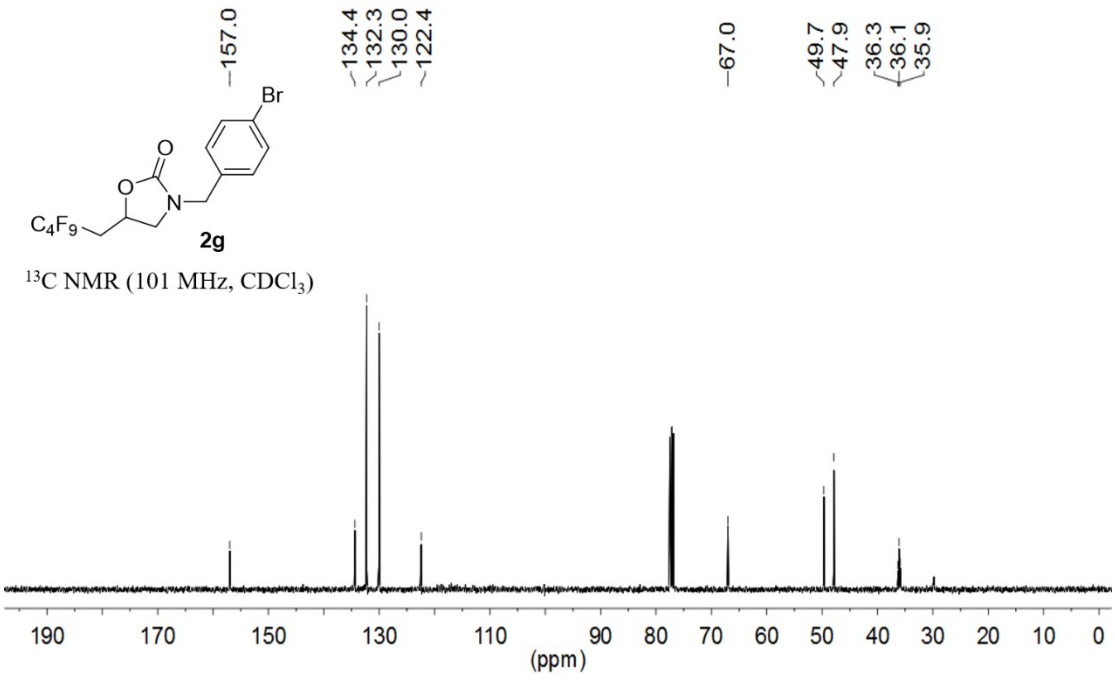
^{13}C NMR (101 MHz, $CDCl_3$)



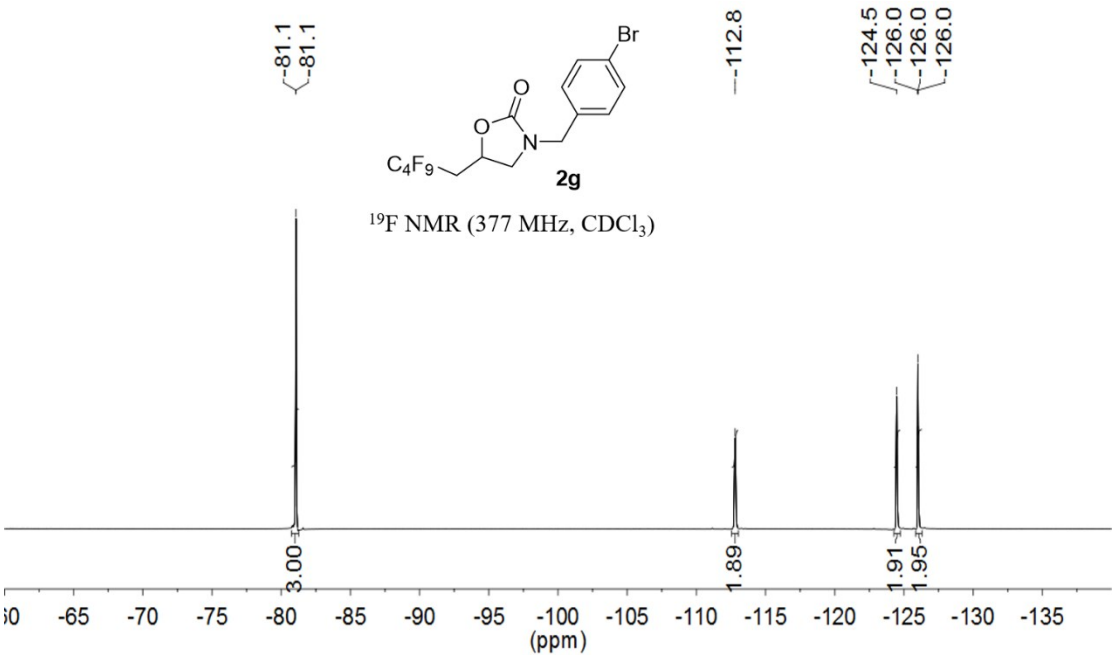
^{19}F NMR (377 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)

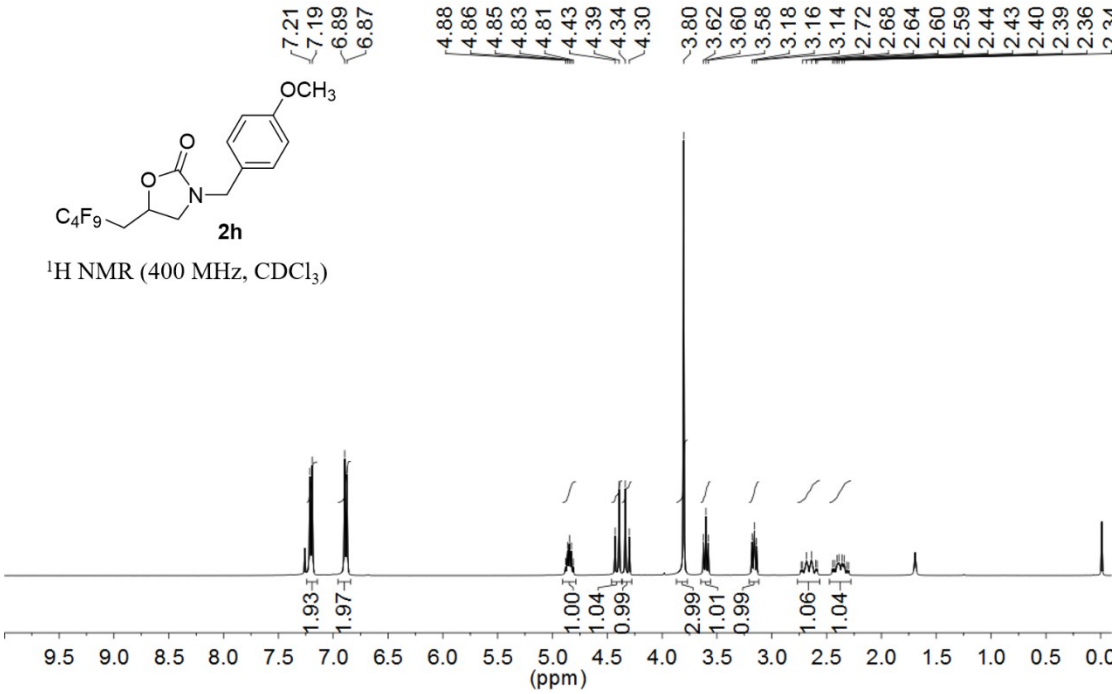


^{13}C NMR (101 MHz, $CDCl_3$)



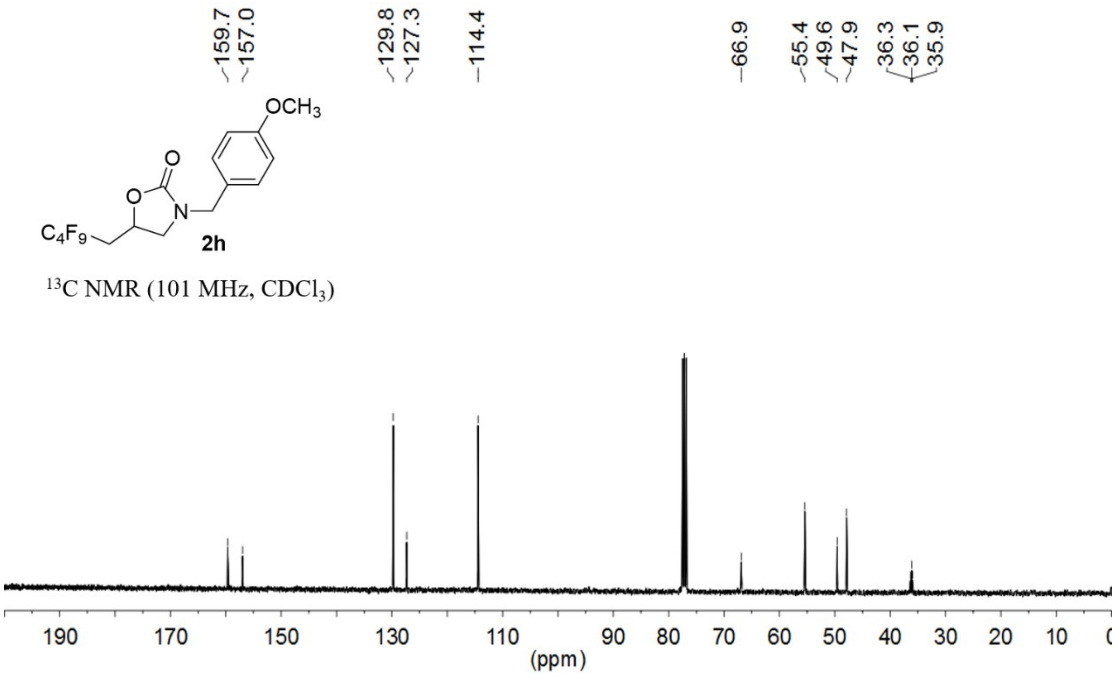
2g

^{19}F NMR (377 MHz, $CDCl_3$)

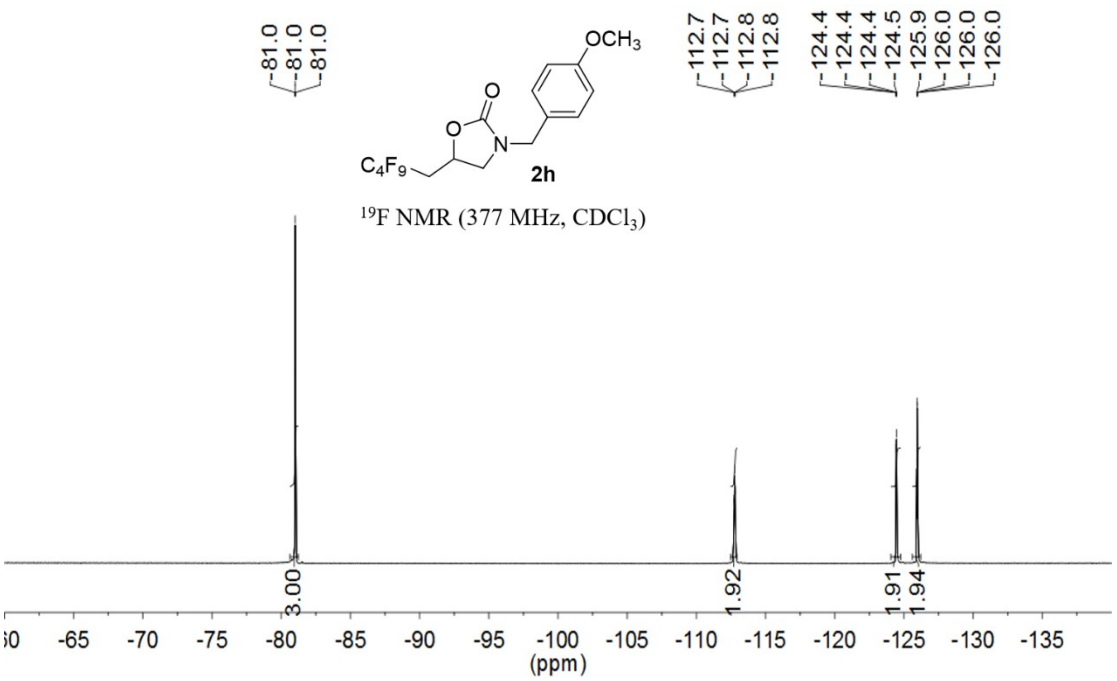


2h

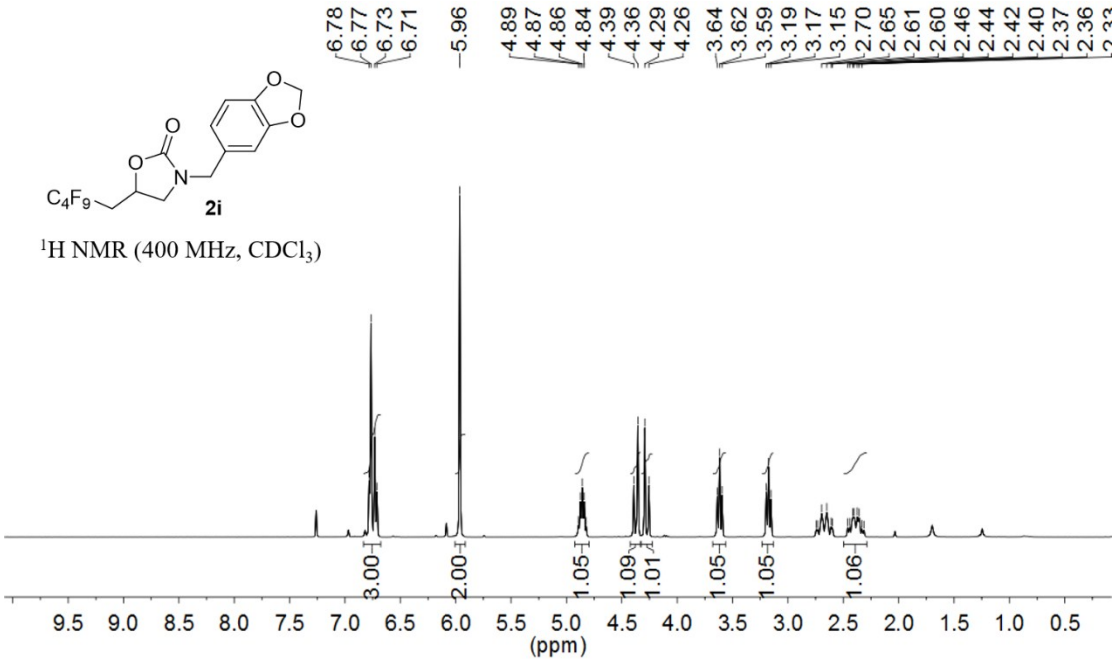
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)

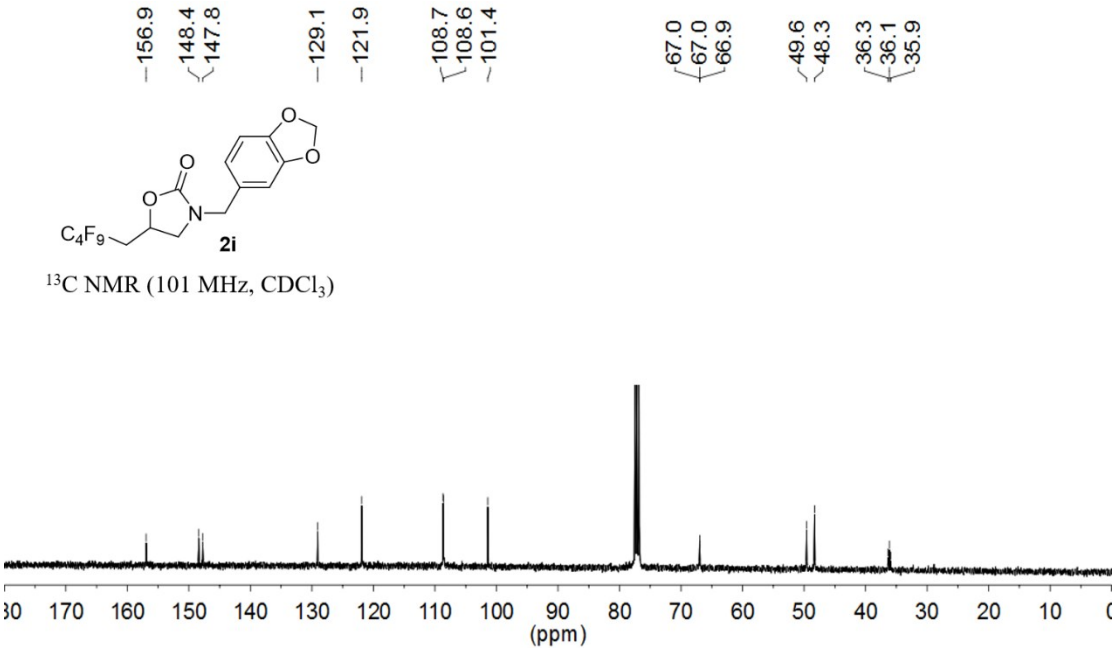


^{19}F NMR (377 MHz, $CDCl_3$)



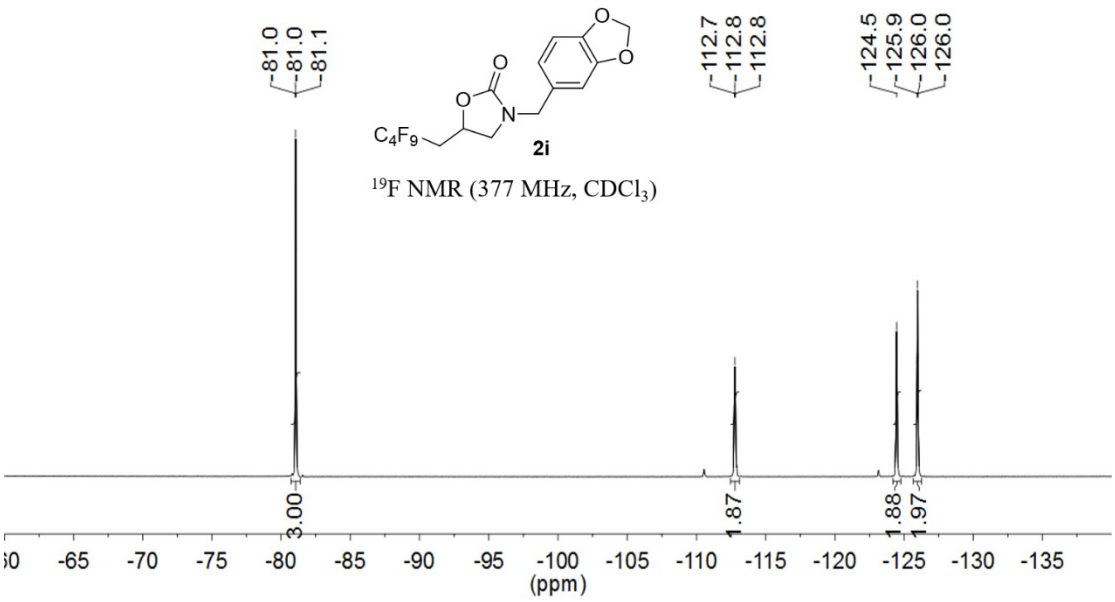
2i

^{1}H NMR (400 MHz, $CDCl_3$)



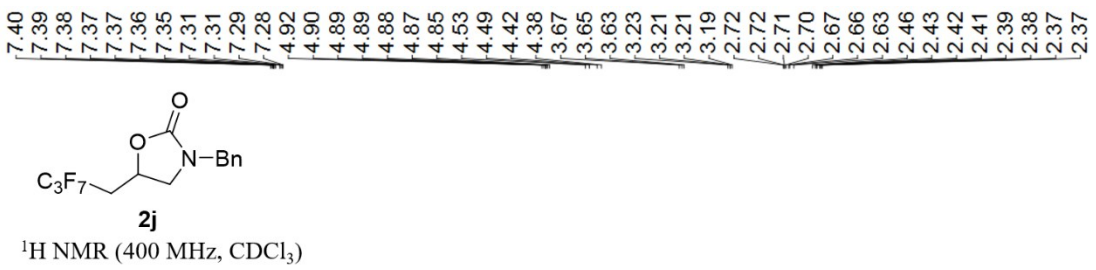
2i

^{13}C NMR (101 MHz, $CDCl_3$)



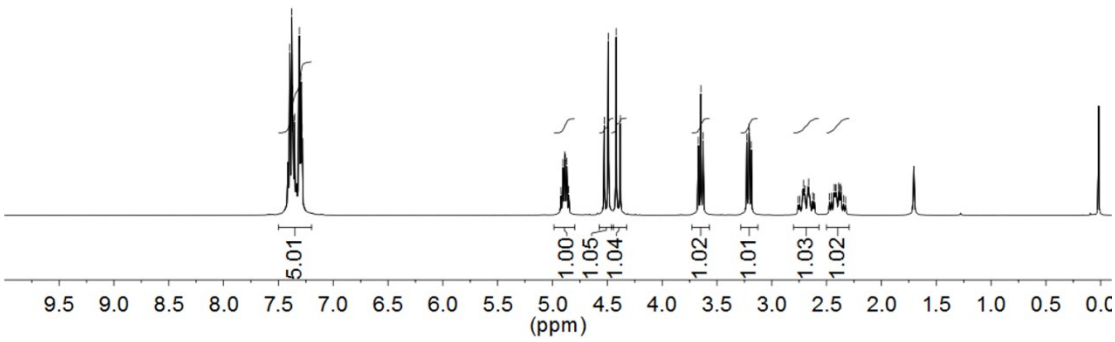
2i

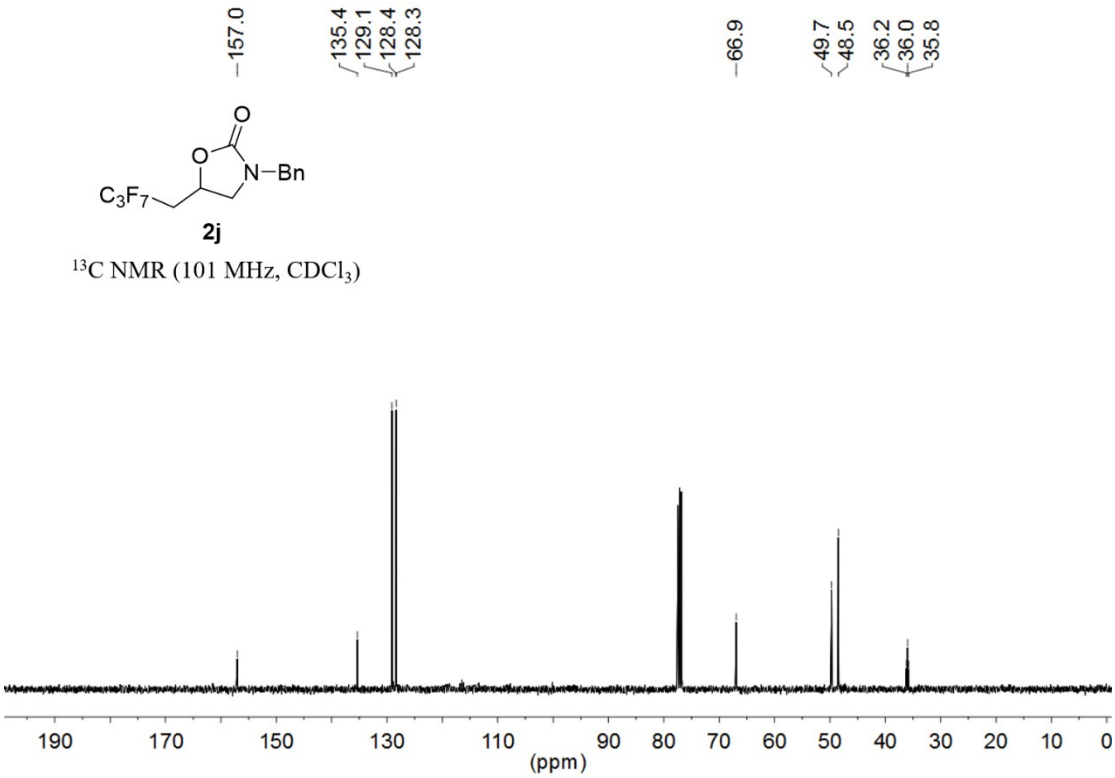
^{19}F NMR (377 MHz, $CDCl_3$)



2j

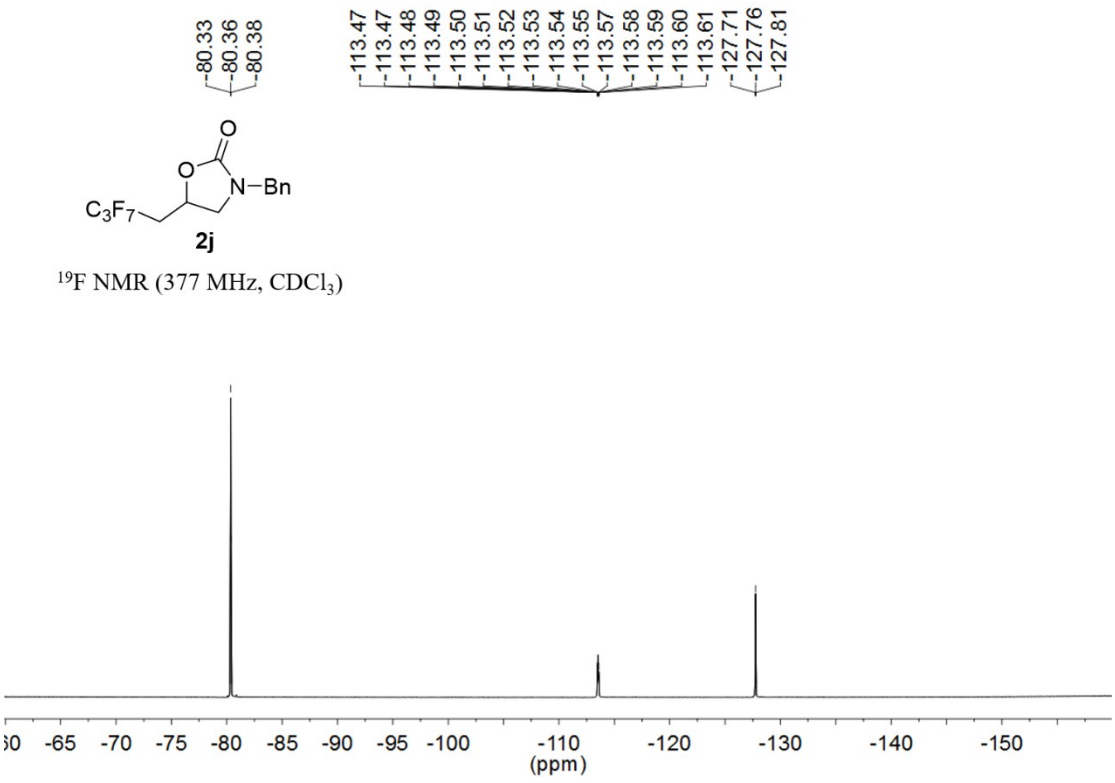
^{1}H NMR (400 MHz, $CDCl_3$)





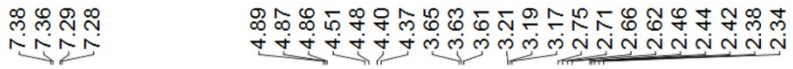
2j

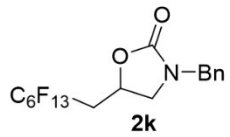
^{13}C NMR (101 MHz, $CDCl_3$)



2j

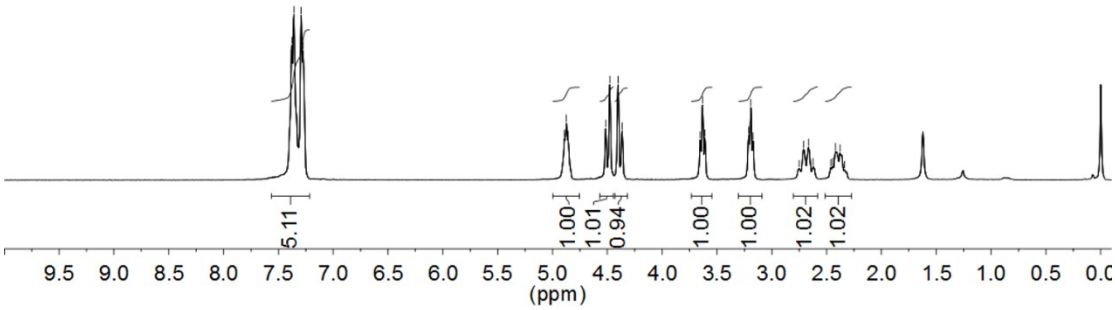
^{19}F NMR (377 MHz, $CDCl_3$)

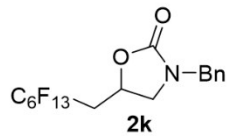




2k

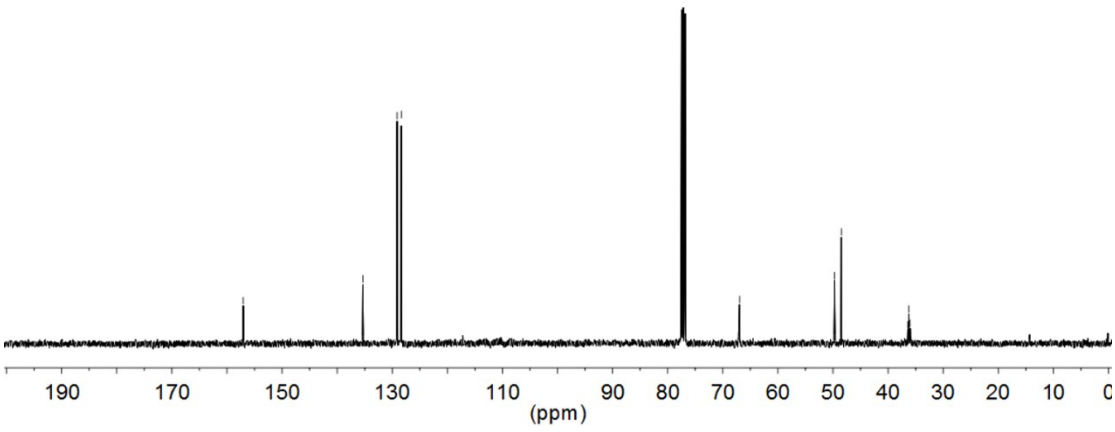
^{1}H NMR (400 MHz, $CDCl_3$)

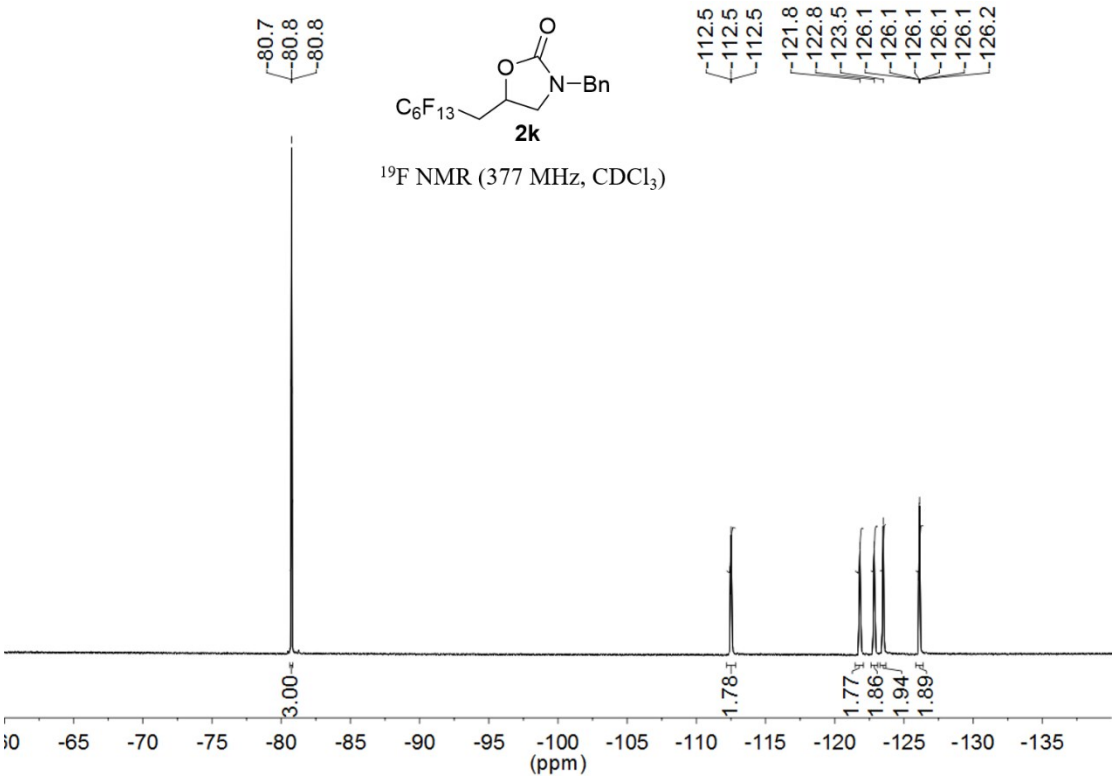




2k

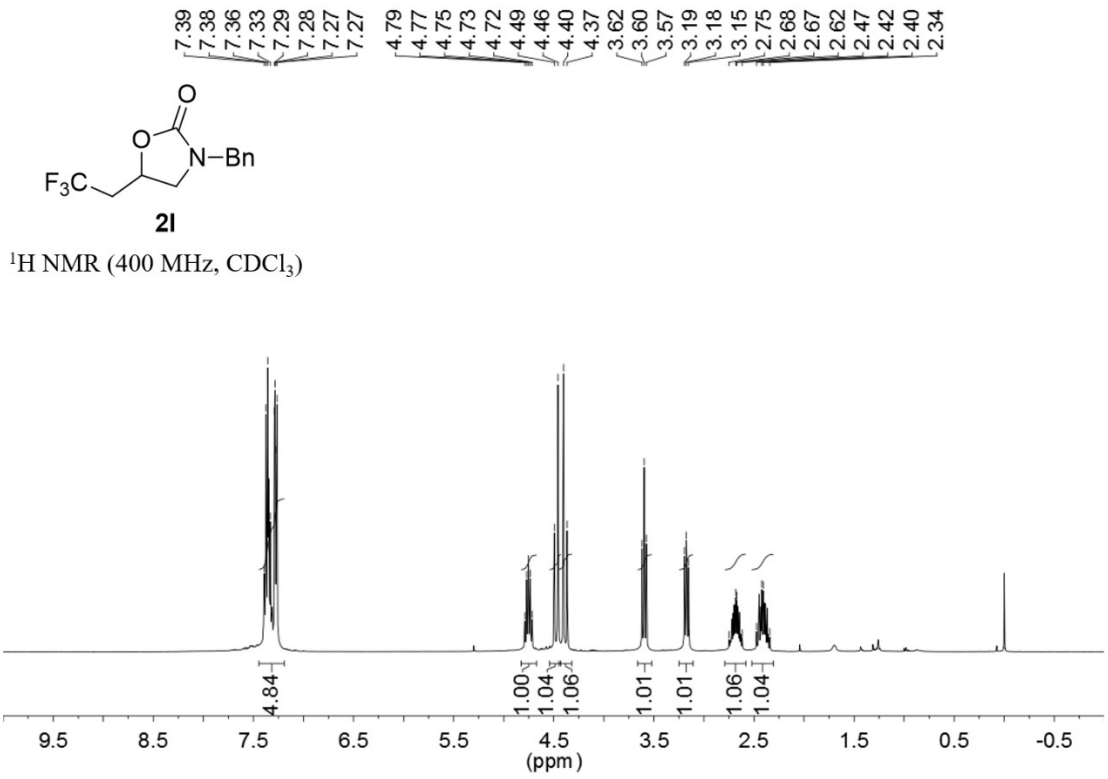
^{13}C NMR (101 MHz, $CDCl_3$)





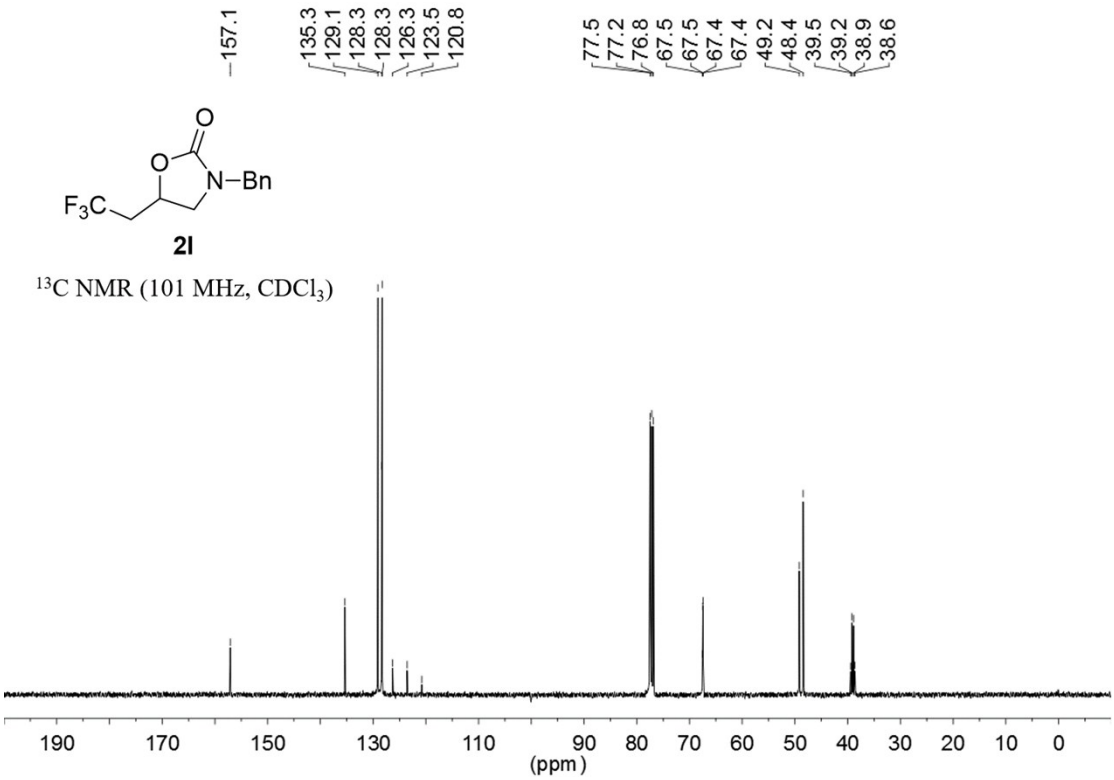
2k

^{19}F NMR (377 MHz, $CDCl_3$)



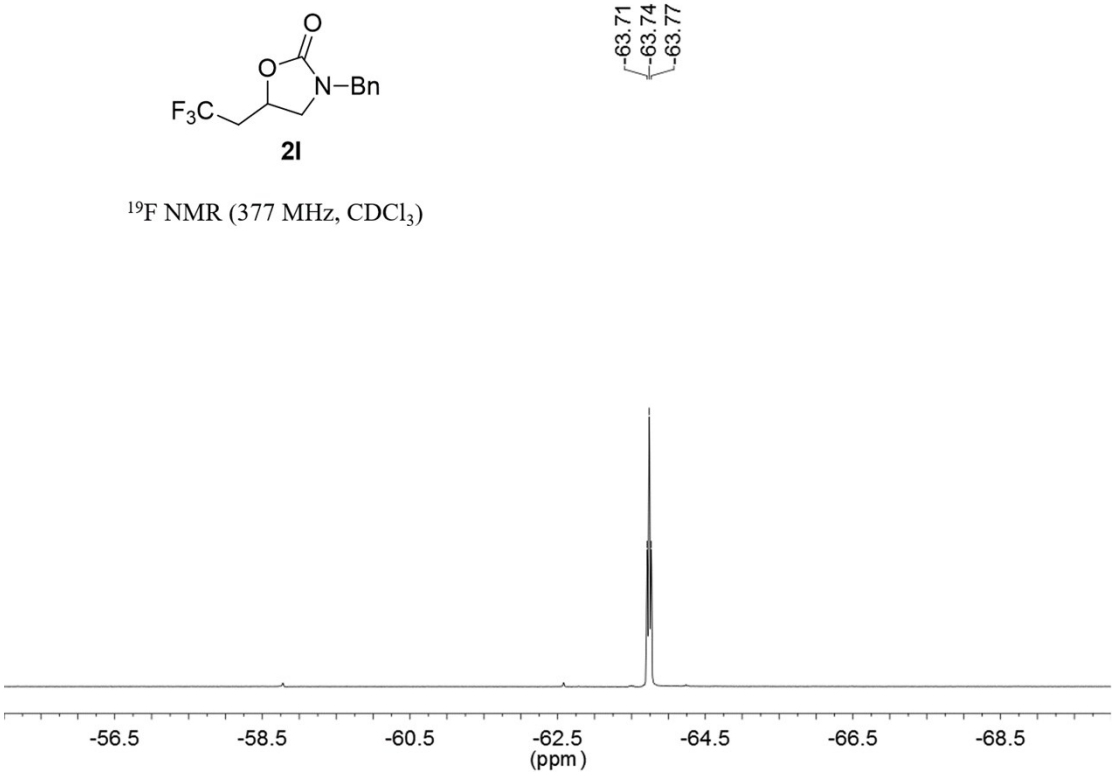
2l

^{1}H NMR (400 MHz, $CDCl_3$)



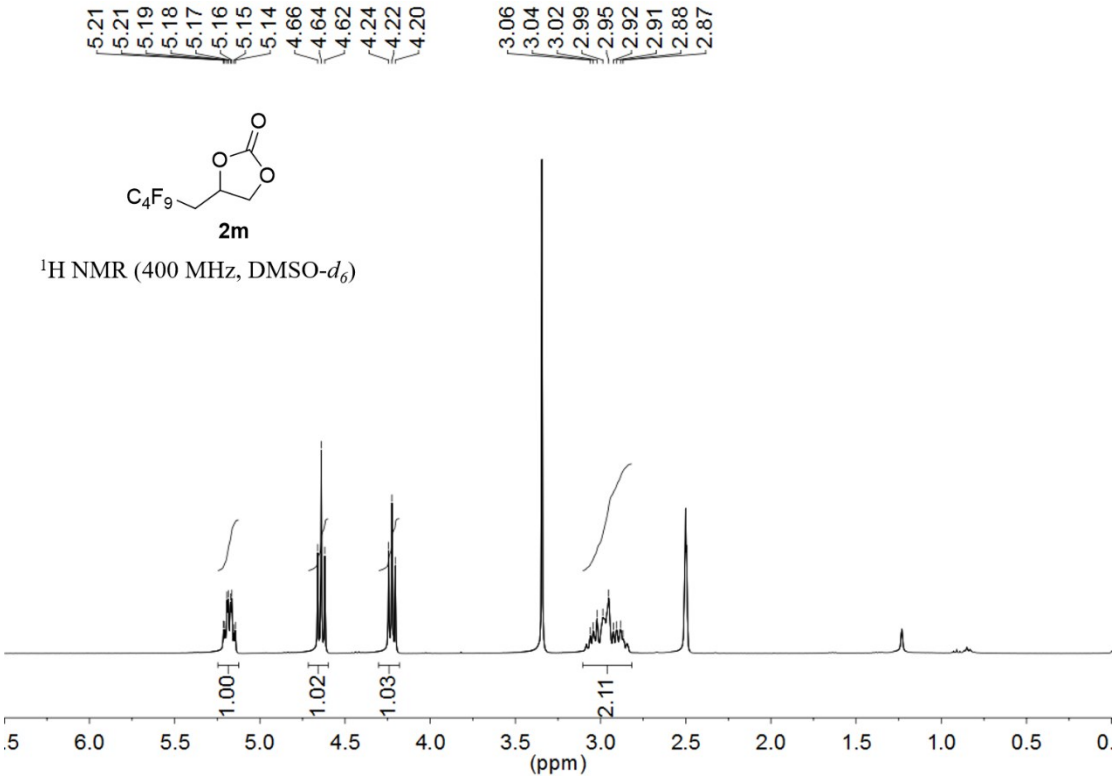
2l

^{13}C NMR (101 MHz, $CDCl_3$)



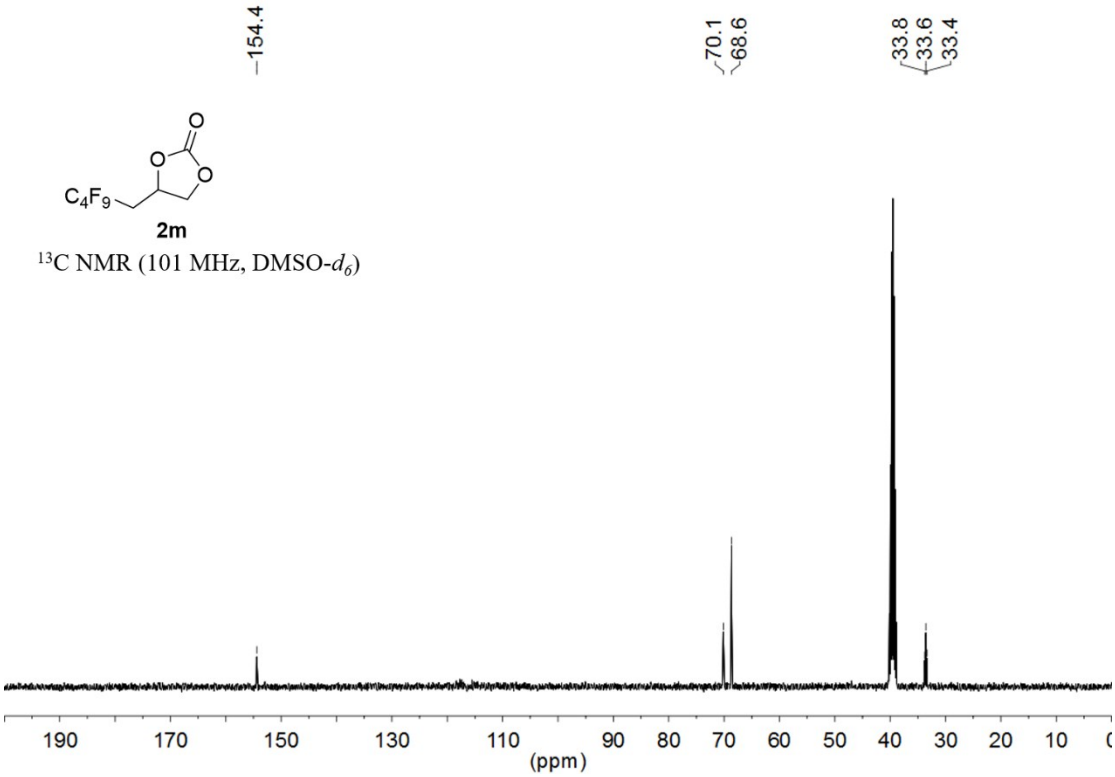
2l

^{19}F NMR (377 MHz, $CDCl_3$)



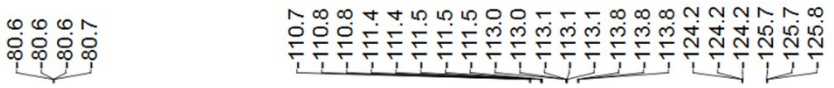
2m

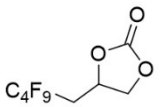
^{1}H NMR (400 MHz, DMSO-d_6)



2m

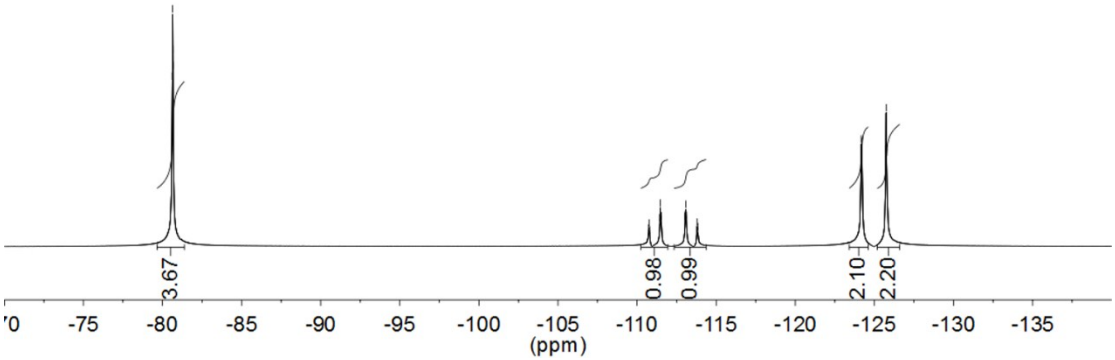
^{13}C NMR (101 MHz, DMSO-d_6)

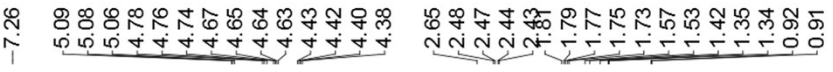


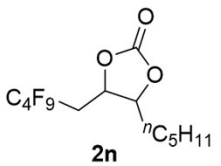


2m

^{19}F NMR (377 MHz, DMSO-d_6)



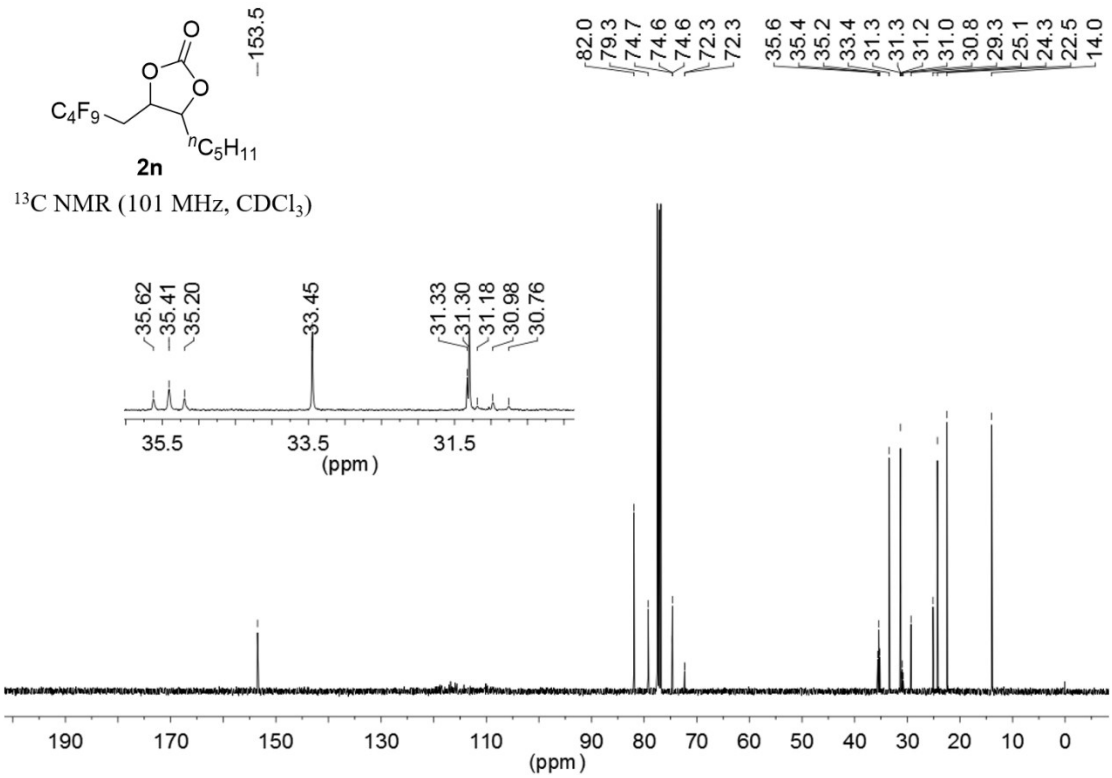




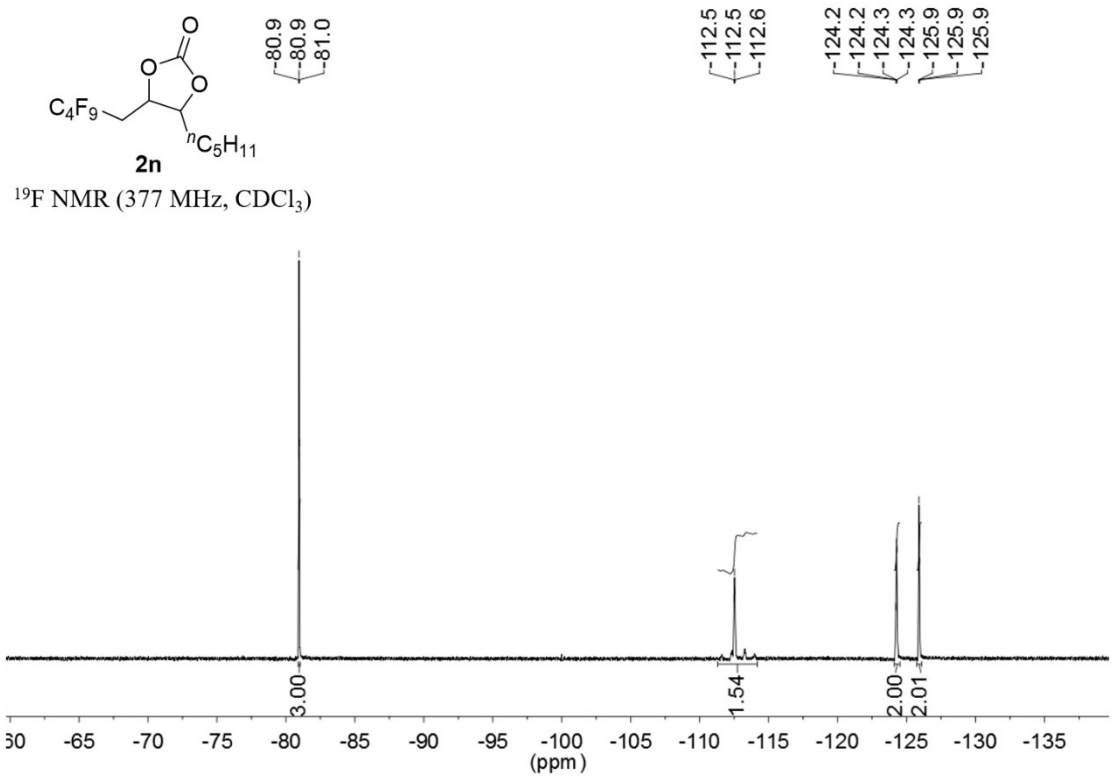
2n

dr = 18:7

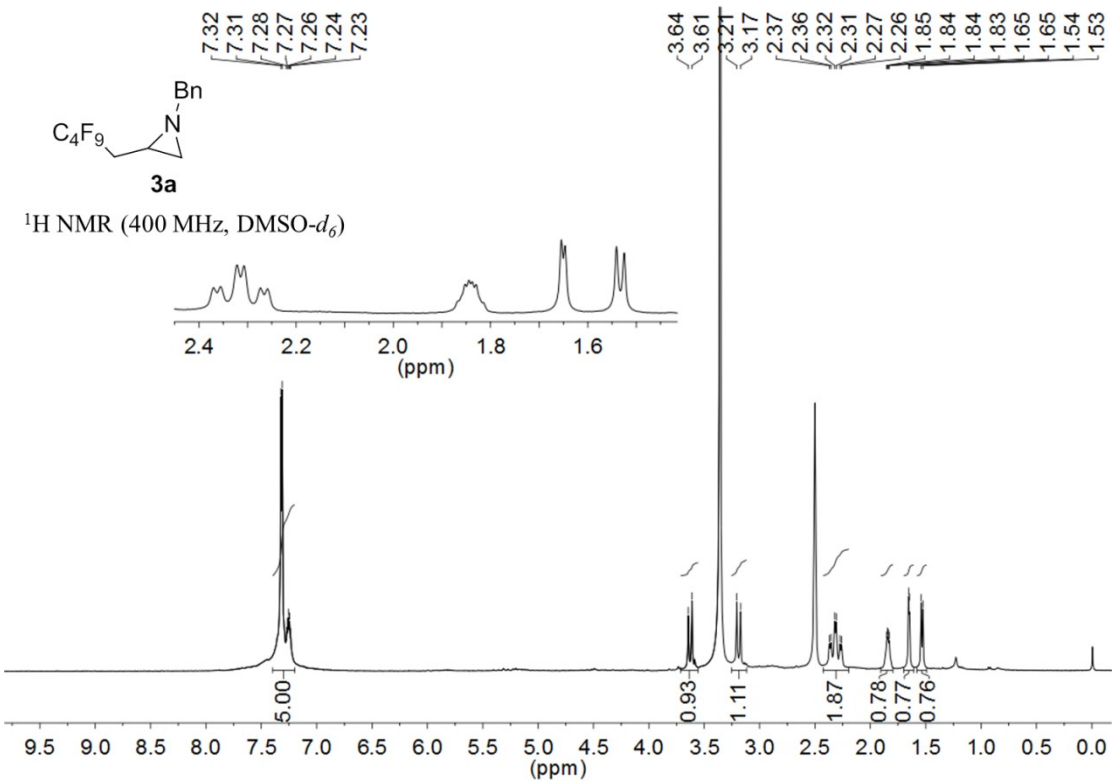
^{1}H NMR (400 MHz, $CDCl_3$)



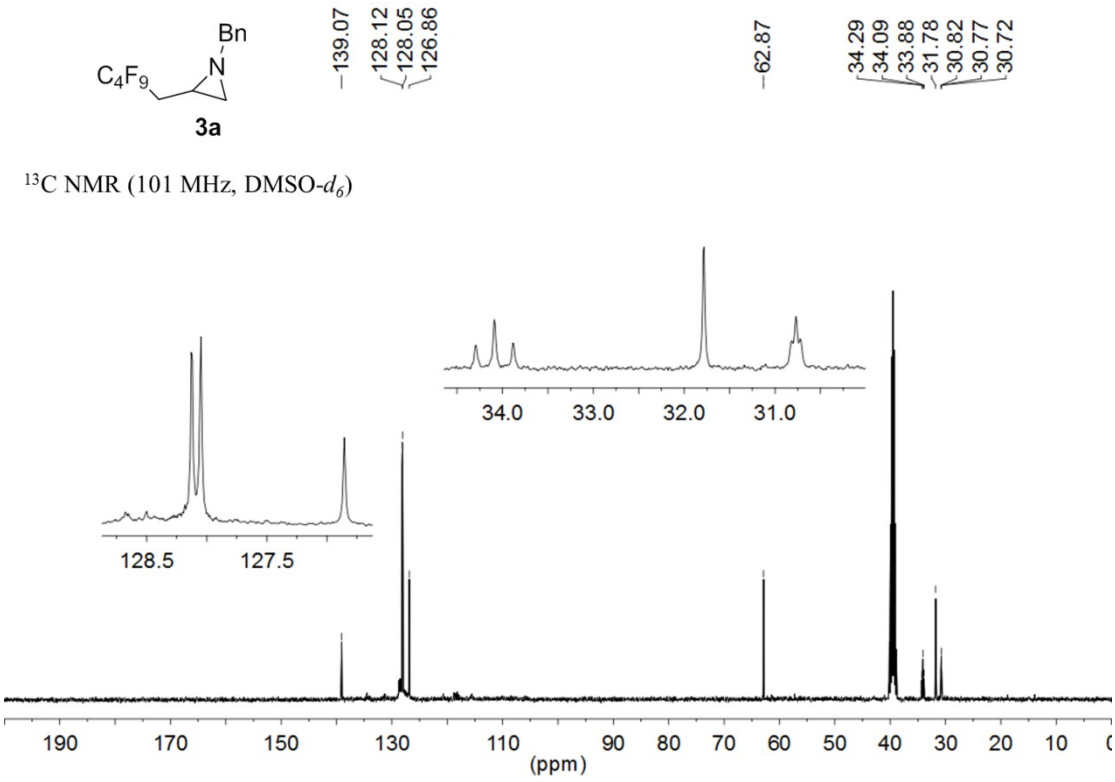
^{13}C NMR (101 MHz, $CDCl_3$)



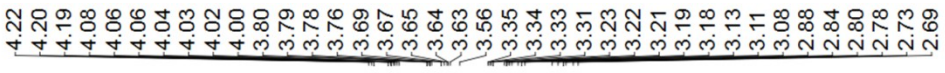
^{19}F NMR (377 MHz, $CDCl_3$)

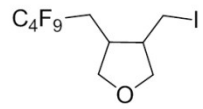


^{1}H NMR (400 MHz, DMSO-d_6)



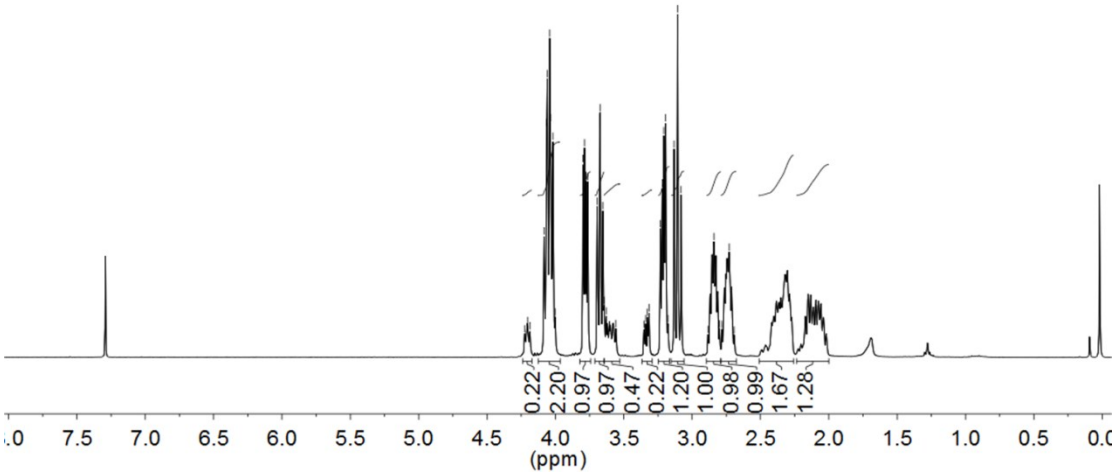
^{13}C NMR (101 MHz, DMSO-d_6)

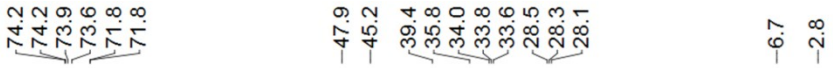


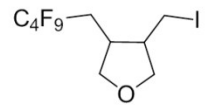


5 (*d.r.* = 50 : 11)

^{1}H NMR (400 MHz, $CDCl_3$)

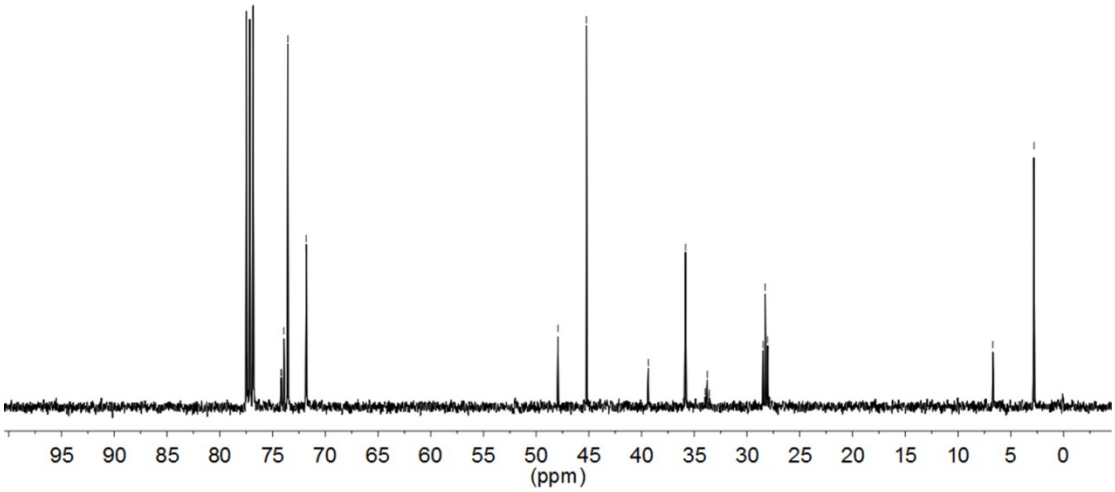


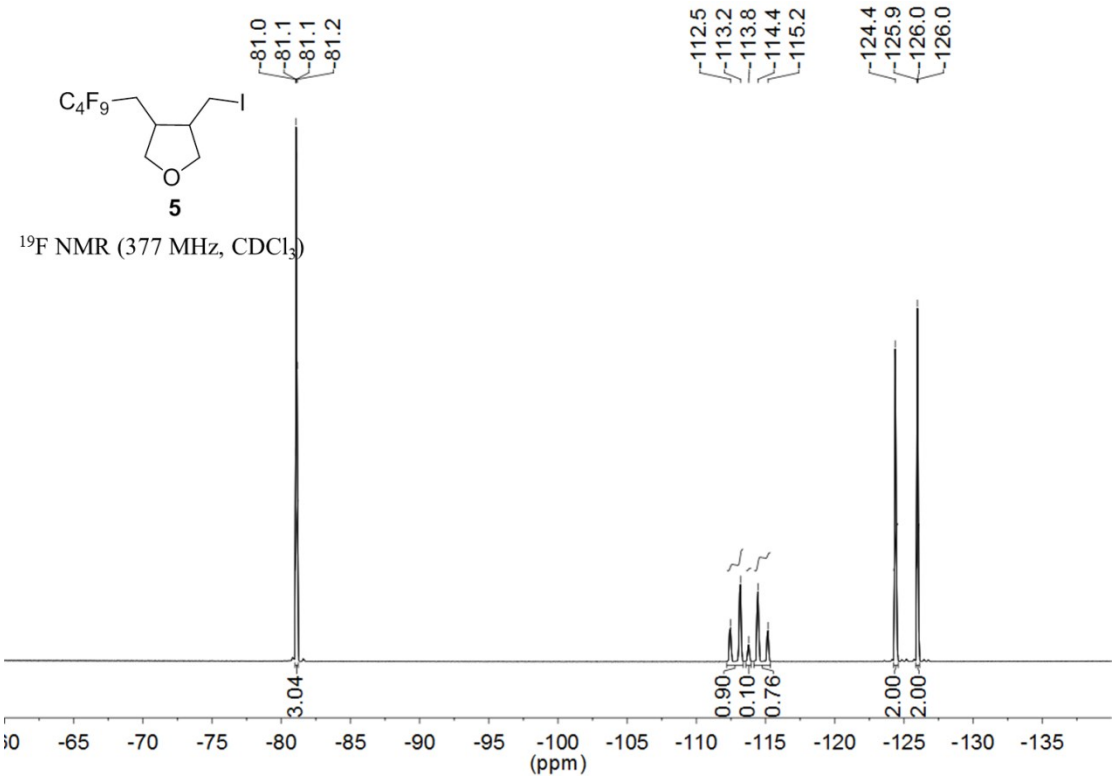


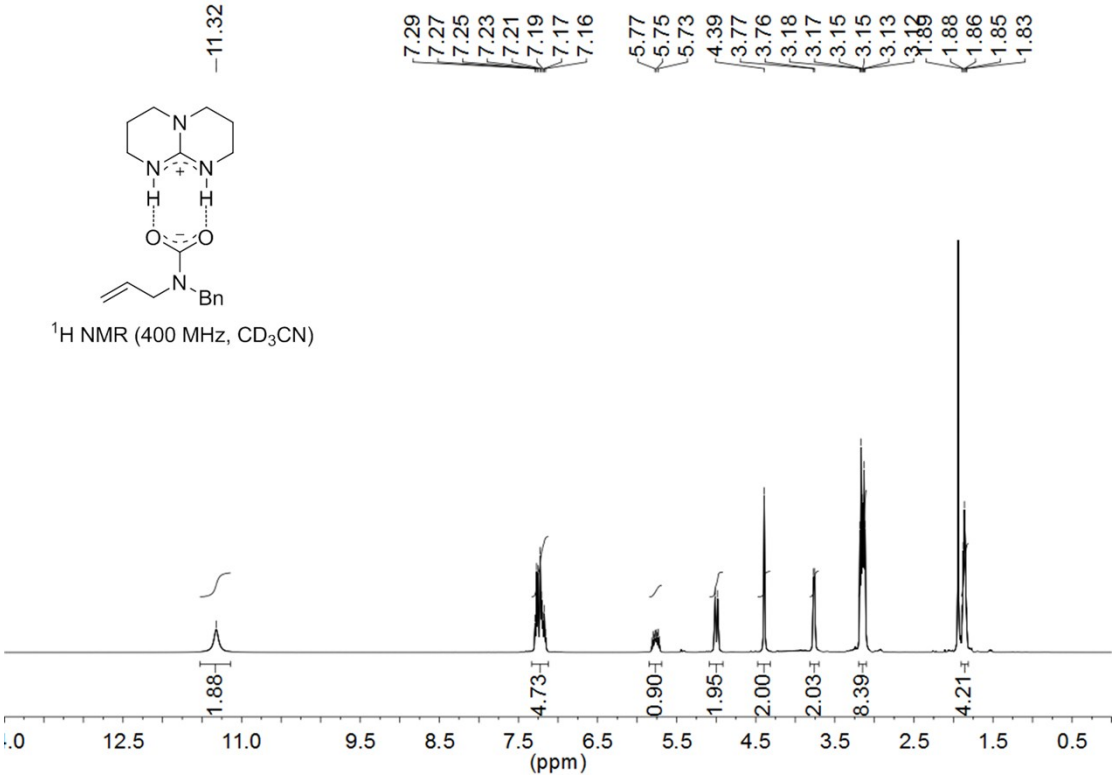


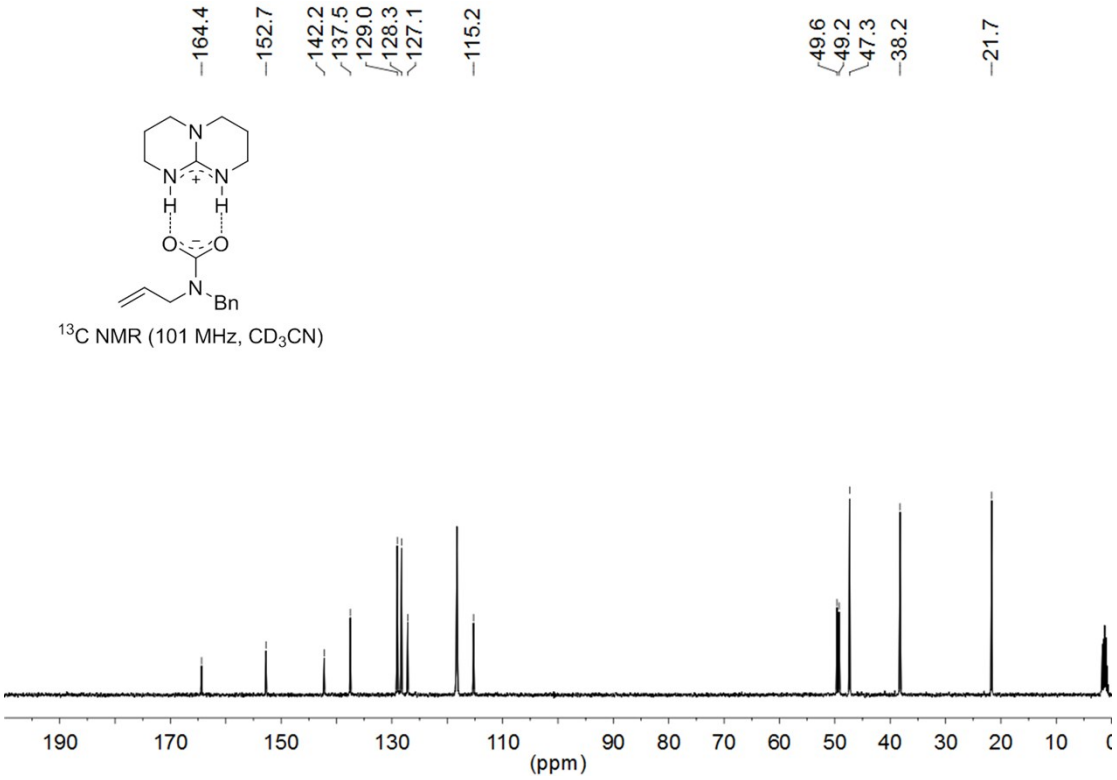
5 (*d.r.* = 50 : 11)

^{13}C NMR (101 MHz, $CDCl_3$)




-81.0
-81.1
-81.1
-81.2
-112.5
-113.2
-113.8
-114.4
-115.2
-124.4
-125.9
-126.0
-126.0
C4F9
I
O
5
19F NMR (377 MHz, CDCl3)
3.04
0.90
0.10
0.76
2.00
2.00
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
-135
(ppm)


11.32
7.29
7.27
7.25
7.23
7.21
7.19
7.17
7.16
5.77
5.75
5.73
4.39
3.77
3.76
3.18
3.17
3.15
3.15
3.13
1.88
1.86
1.85
1.83
N
H
O
Bn
1H NMR (400 MHz, CD3CN)
1.88
4.73
0.90
1.95
2.00
2.03
8.39
4.21
12.5
11.0
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
(ppm)



^{13}C NMR (101 MHz, CD_3CN)