

Supporting Information

Palladium-Catalyzed Decarboxylative Rearrangements of Allyl 2,2,2-Trifluoroethyl Malonates: Direct Access to Homoallylic Esters

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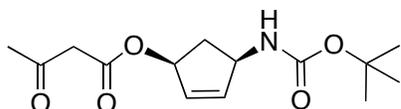
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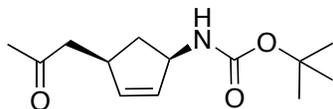
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General Experimental: Commercially available reagents and anhydrous solvents were used without further purification unless otherwise specified. Tetrahydrofuran (THF) was distilled from sodium and benzophenone prior to use. THF was also degassed with argon (Ar) prior to use in palladium reactions. Toluene was dried with a Dean-Stark apparatus prior to use. Reactions were monitored by thin-layer chromatography (TLC) on silica gel 60 F254 (0.2 mm) precoated aluminum foil and visualized with an ethanolic solution of KMnO_4 . Flash chromatography was performed with silica gel 60 (230–400 mesh). ^1H NMR and ^{13}C NMR spectra were recorded at ambient temperature with the residual solvent peaks as internal standards. The line positions of multiplets are given in ppm (δ) and the coupling constants (J) are given as absolute values in Hertz. Infrared spectra were recorded by a FT-IR spectrometer and reported as cm^{-1} . All melting points were recorded uncorrected. High-resolution mass spectra (HRMS) data were obtained as specified.

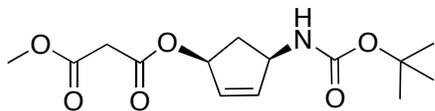


(±)-(1S*,4R*)-4-(tert-Butoxycarbonylamino)cyclopent-2-enyl 3-oxobutanoate (5): A solution of alcohol (±)-4 (140 mg, 0.703 mmol) and NaOAc (30 mg, 0.0351 mmol) in anhydrous THF (3 mL) was heated to 75 °C. To the solution was added diketene (0.108 mL, 1.4 mmol). The reaction mixture was heated at 75 °C for 1 h. To the solution was added EtOAc (5 mL) and washed with sat. NaCl (2 × 10 mL). The organic layer was dried over anhydrous sodium sulfate and concentrated to an oil. Purification by column chromatography (silica gel; eluted with 50% EtOAc/hexanes) yielded an off-white solid. Recrystallization from EtOAc/hexanes gave 119 mg of pure white crystals (60%). mp 68–69 °C; ^1H NMR (300 MHz, CDCl_3) δ 1.45 (s, 9H), 1.54–1.59 (m, 1H), 2.27 (s, 3H), 2.80–2.88 (m, 1H), 3.45 (s, 3H), 4.59–4.73 (m, 2H), 5.94 (ddd, $J = 1.8, 1.9, 5.6$ Hz, 1H), 6.04–6.07 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 28.6, 30.4, 38.7, 50.3, 54.5, 78.8, 79.8, 131.6, 137.9, 155.2, 166.9, 200.7; IR (thin film) 1514, 1710, 1737, 2978, 3351 cm^{-1} ; HRMS (FAB) m/z $[M]^+$: calcd. for $\text{C}_{14}\text{H}_{21}\text{NO}_5^+$, 283.1420; found 283.1437.

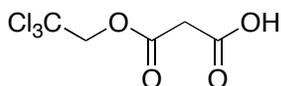


(±)-tert-Butyl (1R*,4R*)-4-(2-oxopropyl)cyclopent-2-enylcarbamate (6): A solution of $\text{Pd}(\text{OAc})_2$ (2 mg, 0.00238 mmol) and PPh_3 (8 mg, 0.0317 mmol) in 1 mL anhydrous THF was stirred at rt for 10 min. To the mixture was added a solution of (±)-5 (30 mg, 0.105 mmol) in 1 mL anhydrous THF. The reaction was stirred at rt overnight. The reaction mixture was concentrated and purified by column chromatography (silica gel; eluted with 10–15% EtOAc/hexanes) to provide 16.5 mg of a pure tan oil (65%). ^1H NMR (500 MHz, CDCl_3) δ 1.09 (ddd, $J = 6.2, 6.2, 13.4$ Hz, 1H), 1.44 (s, 9H), 2.14 (s, 3H), 2.49 (dd, $J = 7.4, 16.8$ Hz, 1H), 2.57 (dd, $J = 6.8, 16.8$ Hz, 1H), 2.61–2.69 (m, 1H), 2.98–3.06 (m, 1H), 4.48–4.60 (m, 1H), 4.65–4.74

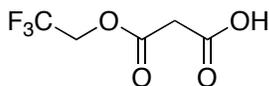
(m, 1H), 5.65–5.71 (m, 1H), 5.77 (ddd, $J = 2.0, 2.0, 5.6$, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 28.6, 30.5, 38.9, 39.9, 50.3, 56.6, 79.5, 132.5, 136.8, 155.4, 207.9; IR (thin film) 1456, 1518, 1599, 1709, 2930, 2977, 3342 cm^{-1} ; HRMS (FAB) m/z $[\text{M}^+]^+$: calcd for $\text{C}_{13}\text{H}_{21}\text{NO}_3^+$, 239.1521; found 239.1535.



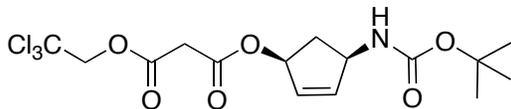
(±)-(1S*,4R*)-4-(tert-Butoxycarbonylamino)cyclopent-2-enyl methyl malonate (7): A solution of (±)-4 (50 mg, 0.251 mmol), methyl malonate (29 mg, 0.251 mmol), DMAP (3 mg, 0.02451 mmol) and EDC·HCl (57.7 mg, 0.301 mmol) in anhydrous CH_2Cl_2 (2 mL) was stirred at rt for 16 h. The reaction mixture was diluted with CH_2Cl_2 (2 mL), washed with sat. NaHCO_3 (3×4 mL), 10% citric acid (3×4 mL) and water (2×4 mL). The organic layer was then washed with sat. NaCl, dried over anhydrous sodium sulfate and concentrated to a yellow oil. Purification by column chromatography (silica gel; eluted with 50% EtOAc/hexanes) provided 68 mg of product as a pure white solid (91%). mp 67–68 °C; ^1H NMR (500 MHz, CDCl_3) δ 1.44 (s, 9H), 1.58 (ddd, $J = 3.8, 3.8, 14.6$ Hz, 1H), 2.81 (ddd, $J = 7.6, 7.6, 14.7$ Hz, 1H), 3.37 (s, 2H), 3.75 (s, 3H), 4.65–4.69 (m, 2H), 5.56–5.59 (m, 1H), 5.93–5.96 (m, 1H), 6.02–6.04 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 28.6, 38.6, 41.6, 52.8, 54.4, 79.0, 79.8, 131.6, 138.0, 155.1, 166.2, 167.2; IR (thin film) 1438, 1514, 1710, 1734, 1747, 2978, 3379 cm^{-1} ; HRMS (FAB) m/z $[\text{M}^+]^+$: calcd for $\text{C}_{14}\text{H}_{21}\text{NO}_6^+$, 299.1369; found, 299.1379.



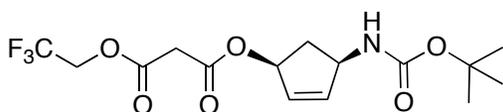
3-Oxo-3-(2,2,2-trichloroethoxy)propanoic acid (10a): In a clean oven dried 15 mL sealed tube, a mixture of 2,2-dimethyl-1,3-dioxane-4,6-dione (Meldrum's acid) (250 mg, 1.73 mmol) and 2,2,2-trichloroethanol (0.166 mL, 1.73 mmol) in anhydrous CH_3CN (5 mL) was stirred at 80 °C for 16 h. The reaction mixture was concentrated to give 390 mg of product as a pure clear oil (95%). ^1H NMR (300 MHz, CDCl_3) δ 3.61 (s, 2H), 4.82 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 40.8, 74.8, 94.4, 164.9, 171.2; HRMS (FAB) m/z $[\text{M}+\text{H}]^+$: calcd for $\text{C}_5\text{H}_6\text{Cl}_3\text{O}_4^+$, 235.9342; found, 235.9332.



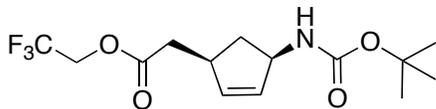
3-Oxo-3-(2,2,2-trifluoroethoxy)propanoic acid (10b): In a clean oven dried 50 mL sealed tube, a mixture of 2,2-dimethyl-1,3-dioxane-4,6-dione (Meldrum's acid) (1.5 g, 10.4 mmol) and 2,2,2-trifluoroethanol (1.5 mL, 20.8 mmol) in anhydrous toluene (15 mL) was stirred at 100 °C for 16 h. The reaction mixture was concentrated to give 1.88 g of product as a pure white solid (97%). ^1H NMR (300 MHz, CDCl_3) δ 3.56 (s, 2H), 4.55 (q, $J = 8.4$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 40.6, 61.4 (q, $J = 37.2$ Hz), 122.7 (q, $J = 275$ Hz), 164.8, 171.5; HRMS (FAB) m/z $[\text{M}+\text{H}]^+$: calcd for $\text{C}_5\text{H}_6\text{F}_3\text{O}_4^+$, 187.0213; found, 187.0230.



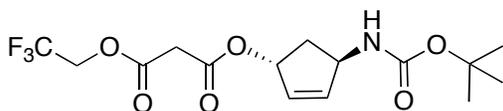
(±)-(1S*,4R*)-4-(tert-Butoxycarbonylamino)cyclopent-2-enyl-2,2,2-trichloroethyl malonate (11a): A solution of 3-oxo-3-(2,2,2-trichloroethoxy)propanoic acid **10a** (310 mg, 1.32 mmol), alcohol (±)-**4** (250 mg, 1.25 mmol) and EDC·HCl (359 mg, 1.88 mmol) in anhydrous CH₂Cl₂ (10 mL) was stirred at rt for 16h. The reaction mixture was diluted with CH₂Cl₂ (5 mL), washed with sat. NaHCO₃ (3 × 5 mL), 10% citric acid (3 × 5 mL) and water (2 × 5 mL). The organic layer was then washed with sat. NaCl, dried over anhydrous sodium sulfate and concentrated to a yellow oil. The material was purified by column chromatography (silica gel; eluted with 10–15% EtOAc/hexanes) to give 500 mg of a clear oil (97%). ¹H NMR (600 MHz, CDCl₃) δ 1.45 (s, 9H), 1.60 (ddd, *J*= 3.7, 3.7, 14.7 Hz, 1H), 2.83 (ddd, *J*= 7.3, 7.4, 14.7 Hz, 1H), 3.52 (s, 2H), 4.57–4.75 (m, 2H), 4.80 (s, 2H), 5.58–5.63 (m, 1H), 5.96 (ddd, *J*= 1.8, 2.2, 5.5 Hz, 1H), 6.01–6.05 (m, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 28.6, 38.6, 41.5, 54.5, 74.7, 79.4, 79.9, 94.5, 131.6, 138.1, 155.1, 165.2, 165.4; IR (thin film) 1455, 1510, 1709, 1736, 1767, 2779, 3348 cm⁻¹; HRMS (FAB) *m/z* [M+H]⁺: calcd for C₁₅H₂₁Cl₃NO₆⁺, 416.0434; found, 416.0428.



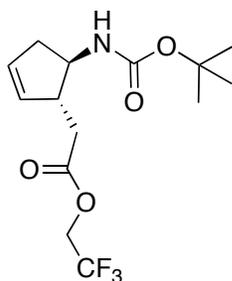
(-)-(1S*,4R*)-4-(tert-Butoxycarbonylamino)cyclopent-2-enyl 2,2,2-trifluoroethyl malonate (11b): A solution of alcohol (-)-**4** (2.42 g, 12.1 mmol), 3-oxo-3-(2,2,2-trifluoroethoxy)propanoic acid **10b** (2.71g, 14.6 mmol), DMAP (0.148 g, 1.21 mmol) and EDC·HCl (4.66 g, 24.3 mmol) in anhydrous CH₂Cl₂ (60 mL) was stirred at rt for 16 h. The reaction mixture was transferred to a separatory funnel and washed with sat. NaHCO₃ (3 × 25 mL), 10% citric acid (3 × 25 mL) and water (2 × 25 mL). The organic layer was then washed with sat. NaCl, dried over anhydrous sodium sulfate and concentrated to a yellow oil. Purification by chromatography (silica gel; eluted with 10-15% EtOAc/hexanes) provided 4.3 g of product as a pure white solid (96%). mp 52–53 °C; [α]_D²⁰ -27.9 (c 0.1, CH₂Cl₂); ¹H NMR (CDCl₃, 600 MHz) δ 1.44 (s, 9H), 1.57 (*J*= 3.8, 3.8, 14.8 Hz, 1H), 2.83 (ddd, *J*= 7.8, 7.8, 14.8 Hz, 1H), 3.48 (s, 2H), 4.53 (q, *J*= 8.4 Hz, 2H), 4.60–4.64 (m, 1H), 4.66–4.72 (m, 1H), 5.58–5.61 (m, 1H), 5.94 (ddd, *J*= 1.9, 1.9, 5.6, 1H), 6.03–6.05 (m, 1H); ¹³C NMR (CDCl₃, 150 MHz) δ 28.6, 38.6, 41.2, 54.5, 61.1 (q, *J*= 36.2 Hz), 79.4, 79.9, 122.5 (q, *J*= 278.3 Hz), 131.4, 138.1, 155.1, 165.2, 165.3; IR (thin film): 1456, 1514, 1713, 1738, 1770, 2981, 3348 cm⁻¹; HRMS (FAB) *m/z* [M+H]⁺: calcd for C₁₅H₂₁F₃O₆N⁺, 368.1321; found, 368.1316.



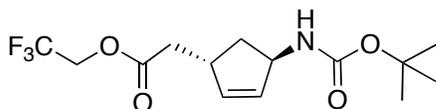
(-)-2,2,2-Trifluoroethyl 2-((1R*,4R*)-4-(tert-butoxycarbonylamino)cyclopent-2-enyl)acetate (12b): In a clean oven dried 15 mL sealed tube equipped with a 19/22 septa, a solution of Pd(dba)₂ (9.4 mg, 0.0163 mmol) and 1,2-bis(diphenylphosphino)ethane (19.5 mg, 0.0490 mmol) in 2 mL anhydrous THF was stirred at rt for 10 min. To the mixture was added a solution of (-)-**11b** (60 mg, 0.163 mmol) in 2 mL anhydrous THF. The septum was removed from the tube (maintaining Ar stream) and the tube was sealed with a teflon screw cap and heated at 75 °C for 6 h. The reaction mixture was concentrated and purified by column chromatography (silica gel; eluted with 100% hexanes to 10% EtOAc/hexanes) to give 43 mg of a pure white powder (82%). mp 49–50 °C; $[\alpha]_D^{20}$ -1.9 (c 0.1, CH₂Cl₂); ¹H NMR (600 MHz, CDCl₃) δ 1.20 (ddd, *J* = 6.5, 6.5, 13.5 Hz, 1H), 1.45 (s, 9H), 2.47 (dd, *J* = 7.3, 15.6 Hz, 1H), 2.55 (dd, *J* = 6.8, 15.6 Hz, 1H), 2.68 (ddd, *J* = 7.8, 7.8, 13.5 Hz, 1H), 3.00–3.07 (m, 1H), 4.48 (q, *J* = 8.5 Hz, 2H), 4.53–4.60 (m, 1H), 4.69–4.76 (m, 1H), 5.73 (ddd, *J* = 2.1, 2.2, 5.6 Hz, 1H), 5.79 (ddd, *J* = 1.8, 2.1, 5.6 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 28.6, 38.5, 40.1, 40.7, 56.6, 60.4 (q, *J* = 36.6 Hz), 79.6, 123.1 (q, *J* = 275.7 Hz), 133.3, 135.8, 155.4, 170.8; IR (thin film) 1413, 1455, 1510, 1702, 1758, 2933, 2978, 3338 cm⁻¹; HRMS (FAB) *m/z* [M+H]⁺: calcd for C₁₄H₂₁F₃NO₄⁺, 324.1423; found, 324.1433.



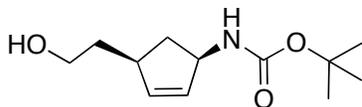
(±)-(1R*,4R*)-4-(tert-Butoxycarbonylamino)cyclopent-2-enyl 2,2,2-trifluoroethyl malonate (14): A solution of alcohol (±)-**4** (100 mg, 0.501 mmol), 3-oxo-3-(2,2,2-trifluoroethoxy) propanoic acid **10b** (93 mg, 0.501 mmol), and PS-PPh₃ (Biotage, 1.73 mmol/g)(290 mg, 0.501 mmol) in anhydrous THF (4.5 mL) was stirred at 0 °C. To the mixture was added DIAD (0.099 mL, 0.501 mmol). The solution was allowed to warm slowly to rt and stirred for 16 h at rt. The resin was filtered and washed with CH₂Cl₂ (10 mL). The filtrate was washed with sat. NaHCO₃ (3 × 10 mL). The organic layer was then washed with sat. NaCl, dried over anhydrous sodium sulfate, and concentrated to an oil. The crude product was adsorbed on silica gel and purified by column chromatography (silica gel; eluted with 100% hexanes to 20% EtOAc/hexanes) to give 156 mg of product (85%). mp 71–73 °C; ¹H NMR (600 MHz, CDCl₃) δ 1.44 (s, 9H), 1.97 (ddd, *J* = 4.7, 7.3, 15.0 Hz, 1H), 2.34 (ddd, *J* = 2.1, 7.3, 15.0 Hz, 1H), 3.46 (s, 2H), 4.45–4.52 (overlapping m, 1H), 4.52 (overlapping q, *J* = 8.5, 2H), 4.87–4.95 (m, 1H), 5.82 (dddd, *J* = 2.4, 2.4, 2.4, 7.2 Hz, 1H), 5.97 (ddd, *J* = 2.1, 2.1, 5.6 Hz, 1H), 6.08 (dd, *J* = 1.8, 5.7 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 28.6, 38.8, 41.2, 55.8, 61.2 (q, *J* = 37 Hz), 79.4, 80.5, 122.8 (q, *J* = 270 Hz), 131.5, 139.7, 155.5, 165.1, 165.7; IR (thin film) 1520, 1678, 1732, 1762, 2359, 2990, 3356 cm⁻¹; HRMS (FAB) *m/z* [M+H]⁺: calcd for C₁₅H₂₁F₃NO₆⁺, 368.1321; found, 368.1324.



(±)-2,2,2-Trifluoroethyl 2-((1R*,5R*)-5-(tert-butoxycarbonylamino)cyclopent-2-enyl)acetate (15): In a clean oven dried 15 mL sealed tube equipped with a 19/22 septa, a solution of (±)-**14** (25 mg, 0.0680 mmol) and KOAc (1.3 mg, 0.0136 mmol) in 2 mL anhydrous toluene was stirred at rt. *N,O*-Bis(trimethylsilyl)acetamide (0.0169 mL, 0.0689 mmol) was added dropwise. The septum was removed from the tube (maintaining Ar stream) and the tube was sealed with a teflon screw cap and heated at 100 °C for 16 h. The reaction mixture was concentrated and purified by column chromatography (silica gel; eluted with 100% hexanes to 15% EtOAc/hexanes) to give 11.5 mg of a pure white powder (52%). mp 52–53 °C; ¹H NMR (600 MHz, CDCl₃) δ 1.44 (s, 9H), 2.16 (dddd, *J*= 2.1, 2.1, 2.1, 5.6, 17.0 Hz, 1H), 2.46 (dd, *J*= 8.5, 16.0 Hz, 1H), 2.71 (dd, *J*= 6.5, 16.0, 1H), 2.76–2.84 (m, 1H), 2.88–2.93 (m, 1H), 3.92–3.99 (m, 1H), 4.42–4.53 (m, 2H), 4.66–4.75 (m, 1H), 5.65 (dddd, *J*= 2.1, 2.4, 2.4, 5.9 Hz, 1H), 5.74 (dddd, *J*= 1.8, 2.4, 2.4, 5.9 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 28.6, 38.0, 39.6, 49.5, 56.1, 69.5 (q, *J*= 36.2), 79.6, 123.2 (q, *J*= 276), 130.1, 132.0, 155.8, 171.0; IR (thin film) 1412, 1455, 1520, 1694, 1759, 2979, 3346 cm⁻¹; HRMS (ESI) *m/z* [M+Na]⁺: calcd for C₁₄H₂₀F₃NO₄Na⁺, 346.1242; found, 346.1237.

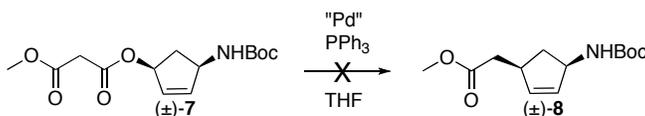


(±)-2,2,2-Trifluoroethyl 2-((1S*,4R*)-4-(tert-butoxycarbonylamino)cyclopent-2-enyl)acetate (16): In a clean oven dried 15 mL sealed tube equipped with a 19/22 septa, a solution of Pd(dba)₂ (4.7 mg, 0.00817 mmol) and 1,2-bis(diphenylphosphino)ethane (9.7 mg, 0.0245 mmol) in 1 mL anhydrous THF was stirred at rt for 10 min. To the mixture was added a solution of (±)-**14** (30 mg, 0.0817 mmol) in 1 mL anhydrous THF. The septum was removed from the tube (maintaining Ar stream) and the tube was sealed with a teflon screw cap and heated at 75 °C for 6 h. The reaction mixture was concentrated and purified by column chromatography (silica gel; eluted with 100% hexanes to 10% EtOAc/hexanes) to give 20 mg of a pure white powder (76%). mp 64–66 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.44 (s, 9H), 1.87–1.97 (m, 2H), 2.43 (dd, *J*= 8.0, 16.0 Hz, 1H), 2.48 (dd, *J*= 7.2, 16.0 Hz, 1H), 3.20–3.27 (m, 1H), 4.47 (q, *J*=8.4 Hz, 2H), 4.45–4.53 (m, 1H), 4.73–4.80 (m, 1H), 5.76 (ddd, *J*= 2.3, 2.4, 5.4 Hz, 1H), 5.85 (ddd, *J*= 1.8, 2.2, 5.8 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 28.6, 38.5, 39.4, 40.6, 56.3, 60.4 (q, *J*= 36.4 Hz), 79.6, 123.1 (q, *J*= 275.8 Hz), 132.9, 136.9, 155.5, 171.0; IR (thin film) 1415, 1505, 1694, 1738, 2930, 2978, 3362, 3409 cm⁻¹; HRMS (FAB) *m/z* [M+H]⁺: calcd for C₁₄H₂₁F₃NO₄⁺, 324.1423; found, 324.1422.



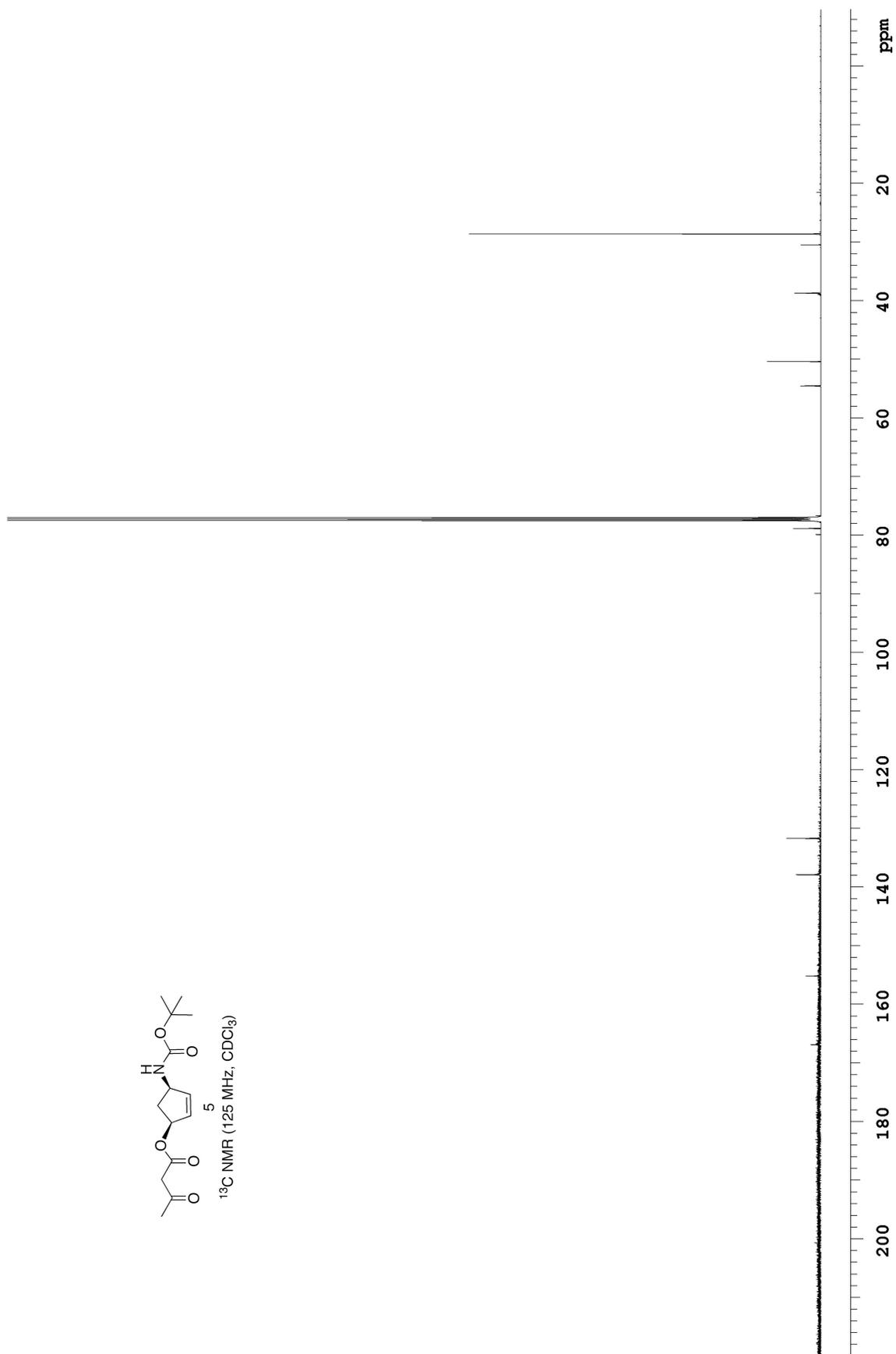
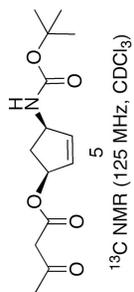
(-)-tert-Butyl (1*R,4*R**)-4-(2-hydroxyethyl)cyclopent-2-enylcarbamate (17):** A solution of (-)-**12b** (25 mg, 0.077 mmol) in THF (0.4 mL) was cooled to -78 °C. The mixture was treated dropwise with a 1M solution of DIBAL-H in THF (0.232 mL, 0.232 mmol) and allowed to stir for 3 h while warming to rt. To the reaction mixture was added sat. aqueous NH_4Cl (5 mL). The resultant gummy precipitate was filtered and the aqueous layer was extracted with CH_2Cl_2 (3×8 mL). The combined organic extracts were washed with sat. NaCl, dried over anhydrous sodium sulfate, and concentrated to an oil. The crude product was purified by column chromatography (silica gel; eluted with 100% hexanes to 33% EtOAc/hexanes) to give 15 mg of a pure colorless oil (85%). ^1H NMR (500 MHz, CDCl_3) δ 1.15 (ddd, $J= 6.4, 6.4, 13.2$, 1H), 1.44 (s, 9H), 1.53–1.60 (m, 2H), 1.74 (dddd, $J= 6.6, 6.8, 6.8, 13.4$ Hz, 1H), 2.58 (ddd, $J= 7.6, 7.6, 13.6$ Hz, 1H), 2.68–2.75 (m, 1H), 3.65–3.74 (m, 2H), 4.55–4.62 (m, 1H), 4.64–4.71 (m, 1H), 5.66 (ddd, $J= 2.0, 2.0, 5.6$ Hz, 1H), 5.82 (ddd, $J= 2.0, 2.0, 5.6$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 28.6, 39.0, 39.5, 41.3, 56.7, 61.6, 79.5, 131.9, 137.7, 155.5; IR (thin film): 1456, 1521, 1693, 2854, 2925, 2968, 3343 cm^{-1} ; HRMS (FAB) m/z $[\text{M}+\text{H}]^+$: calcd for $\text{C}_{12}\text{H}_{22}\text{O}_3\text{N}^+$, 228.1600; found, 228.1609.

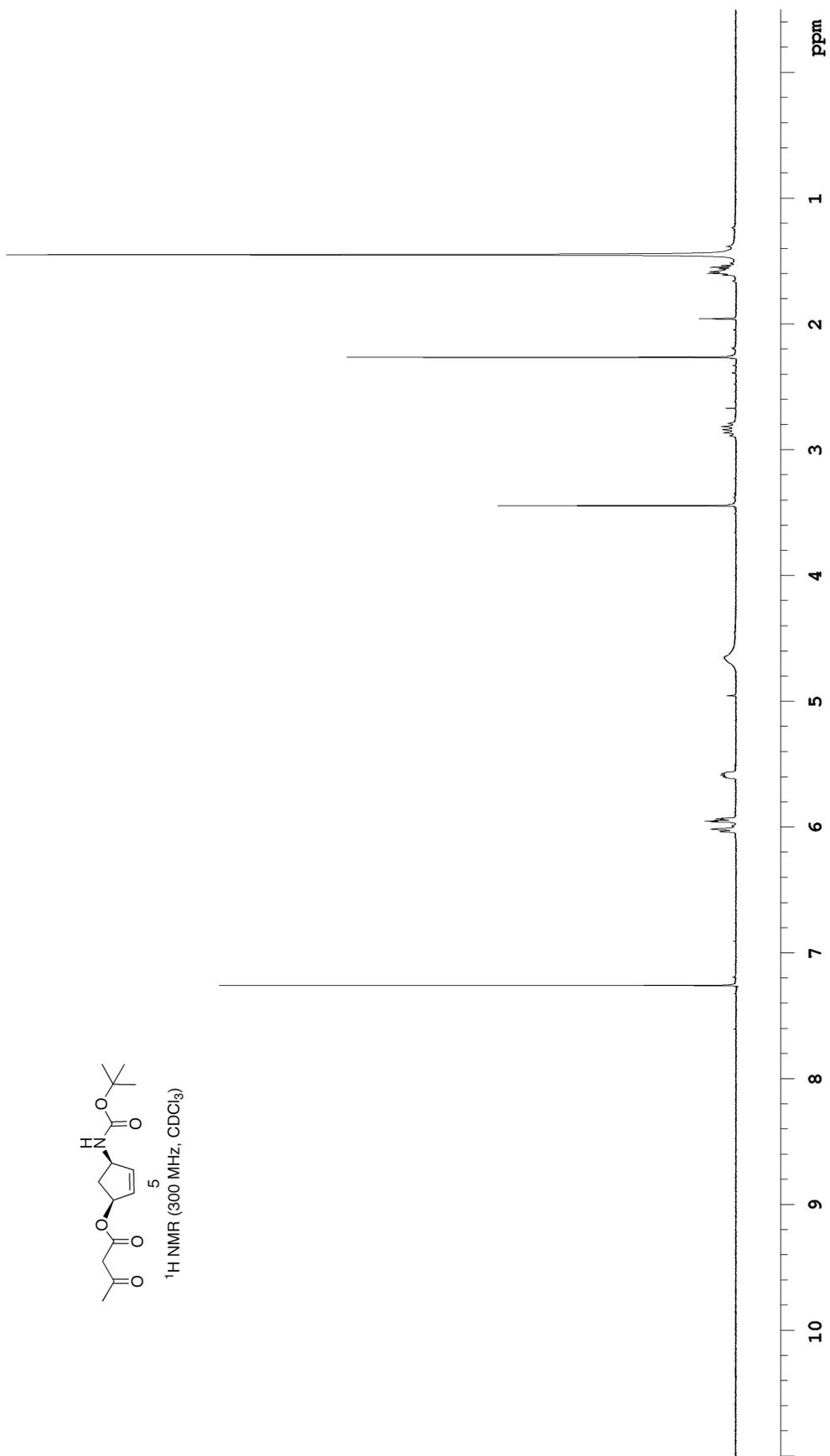
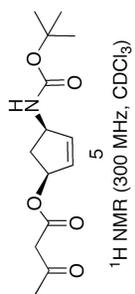
Scheme 1. Attempted Decarboxylative Allylation of (\pm)-**7**.

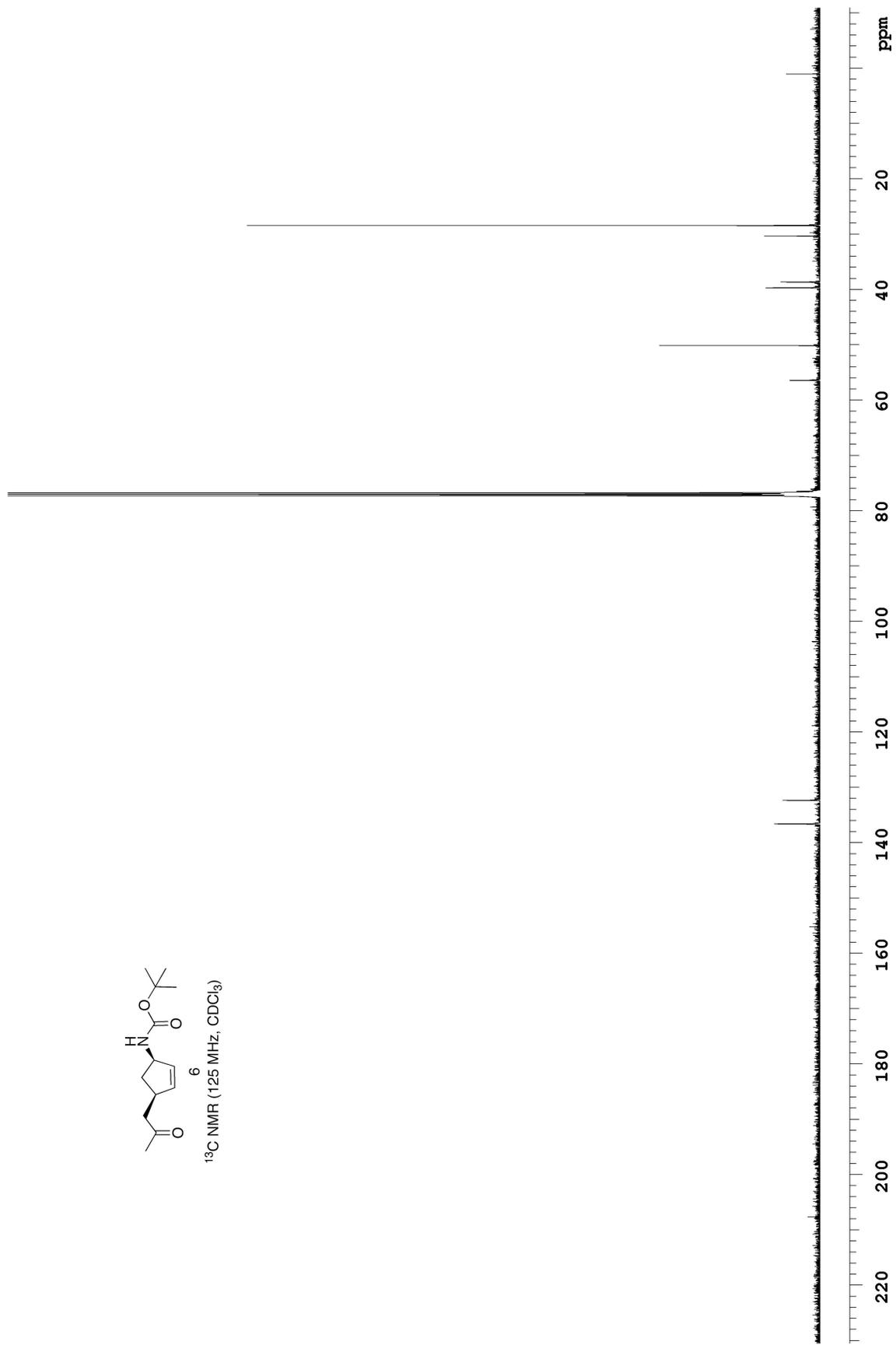
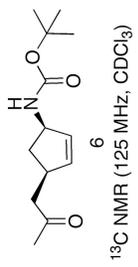


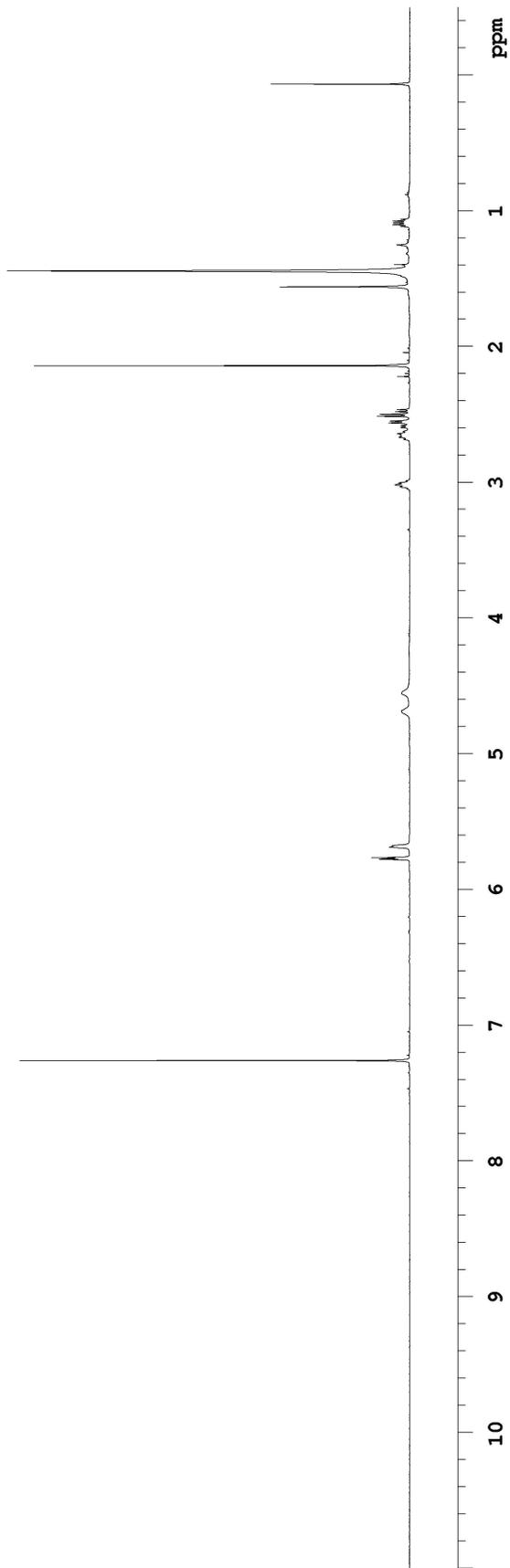
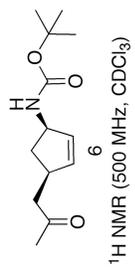
entry	substrate	Pd source	temp	time	yield
1	7	$\text{Pd}(\text{OAc})_2$	75 °C	16 h	0% ^a
2	7	$\text{Pd}(\text{dba})_2$	75 °C	16 h	0% ^a
3	7	$\text{Pd}_2(\text{dba})_3 \cdot \text{CHCl}_3$	75 °C	16 h	0% ^a
4	7	$\text{Pd}_2(\text{dba})_3$	75 °C	16h	0% ^a

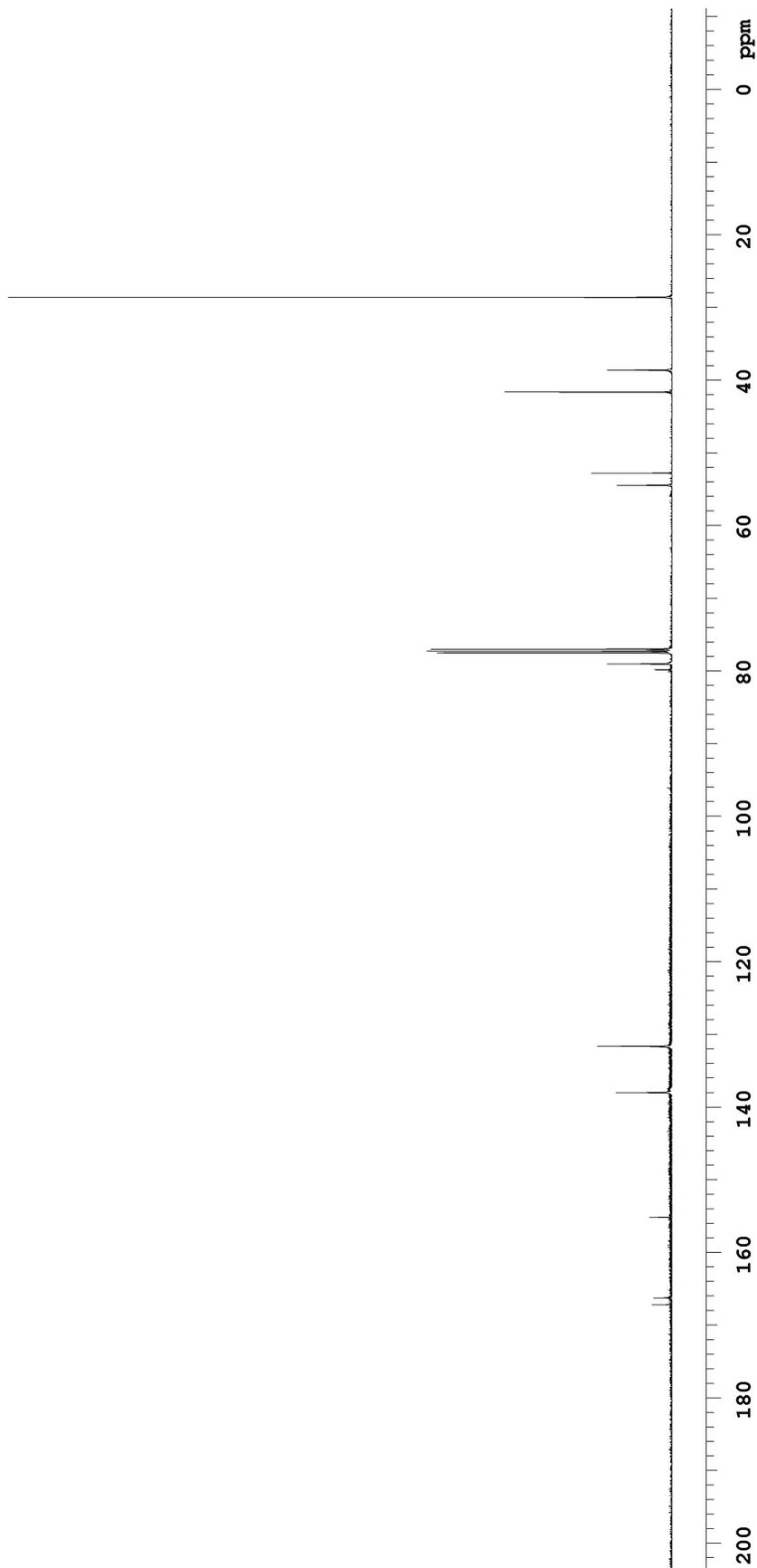
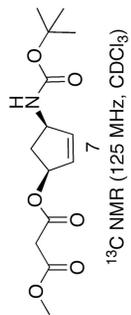
^a Starting material was recovered.

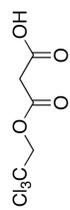






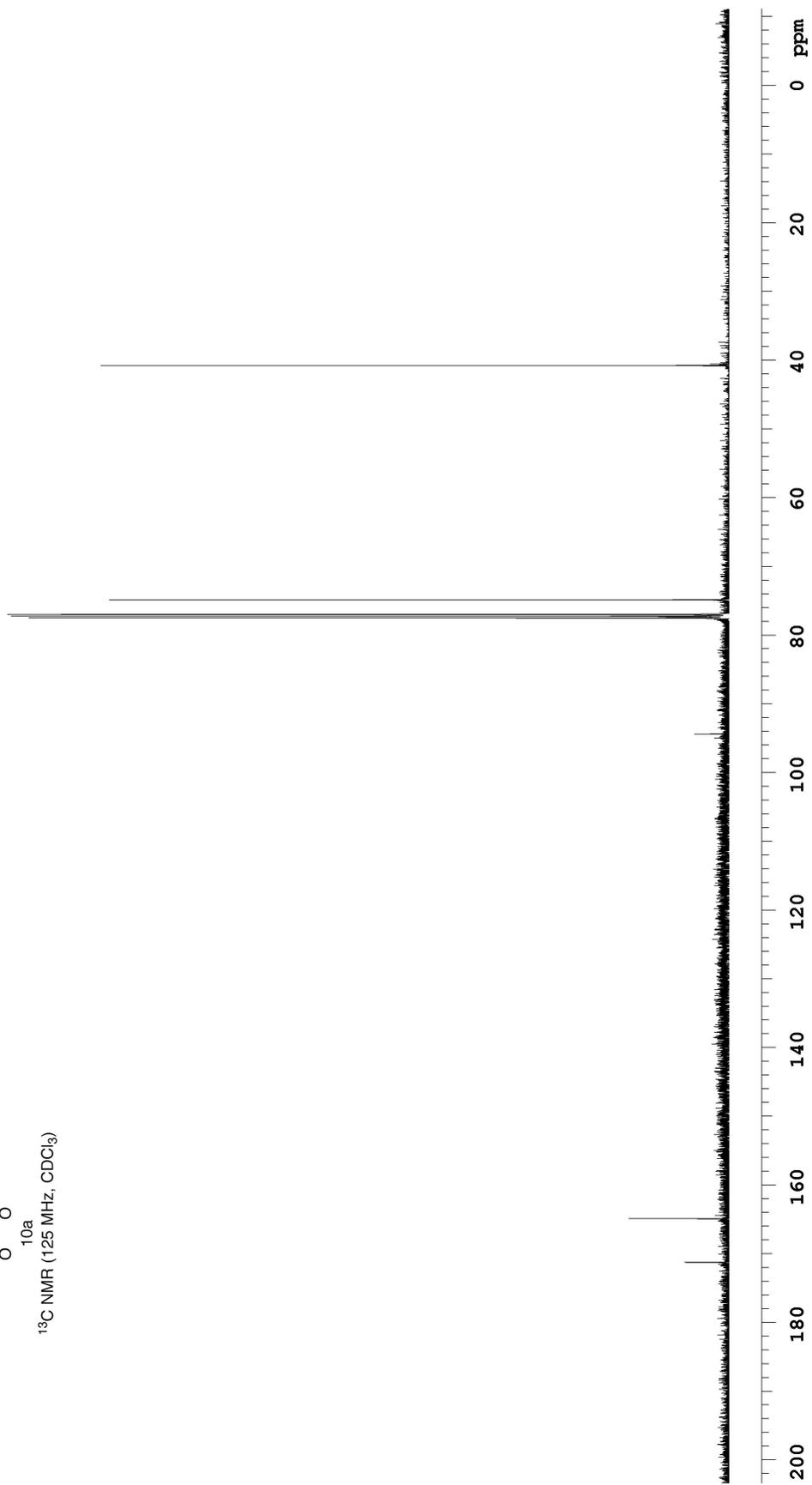


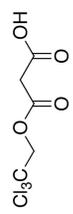




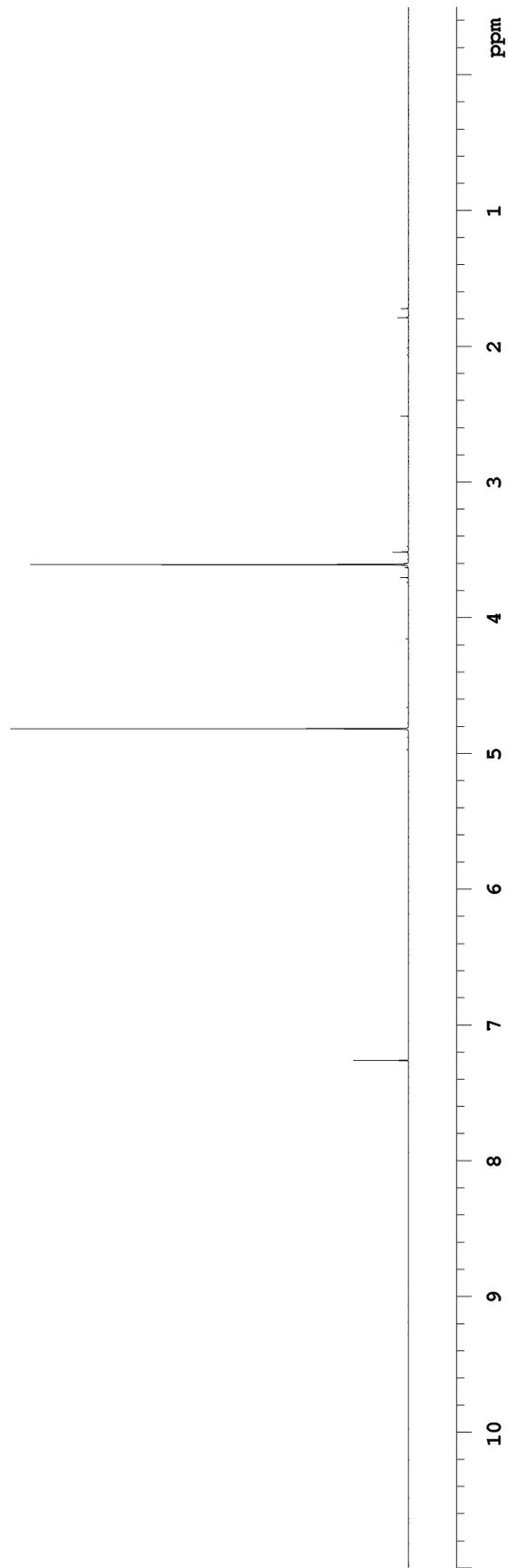
10a

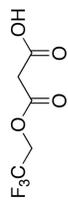
¹³C NMR (125 MHz, CDCl₃)





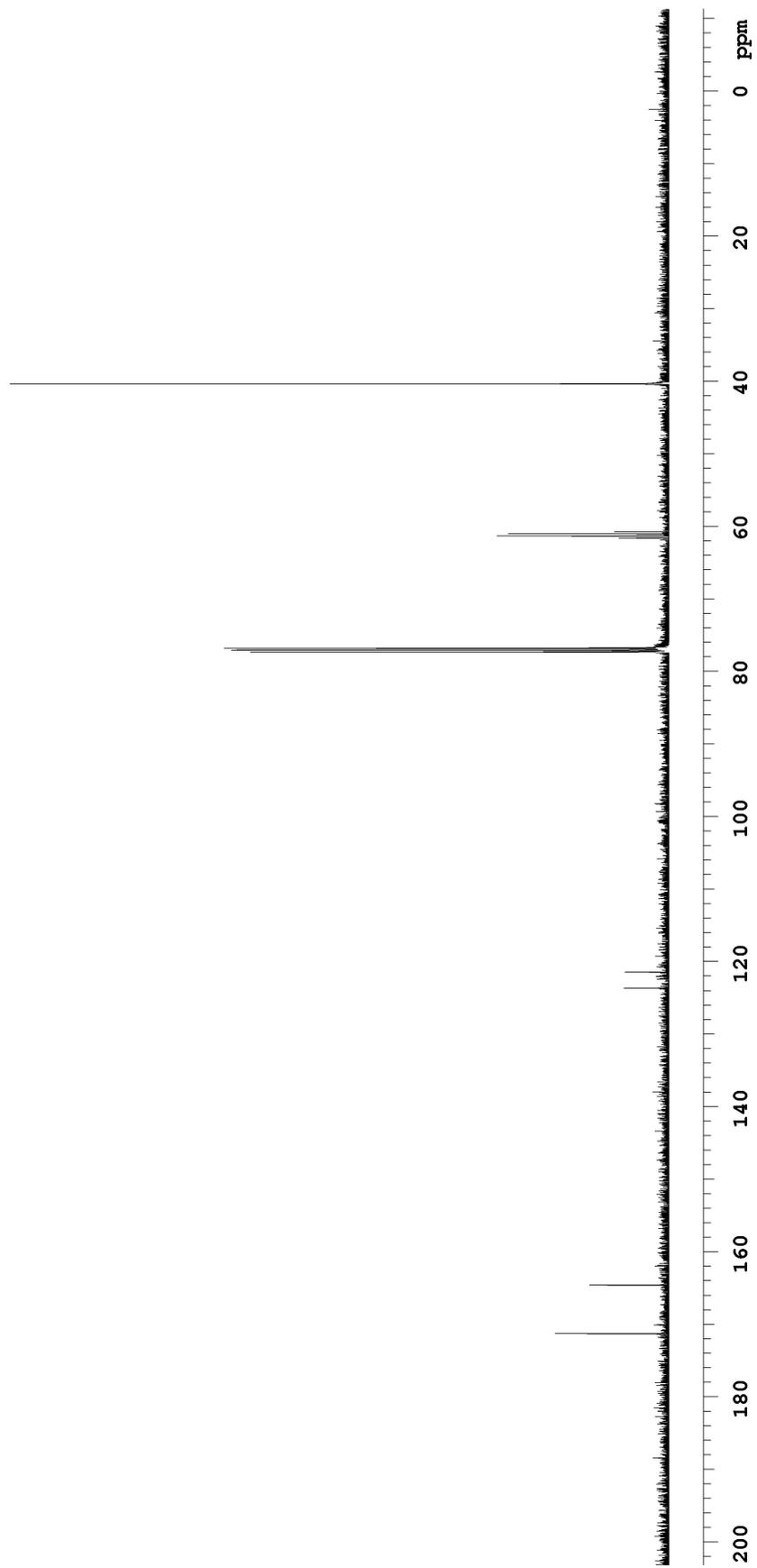
10a
¹H NMR (300 MHz, CDCl₃)

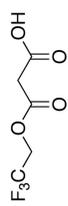




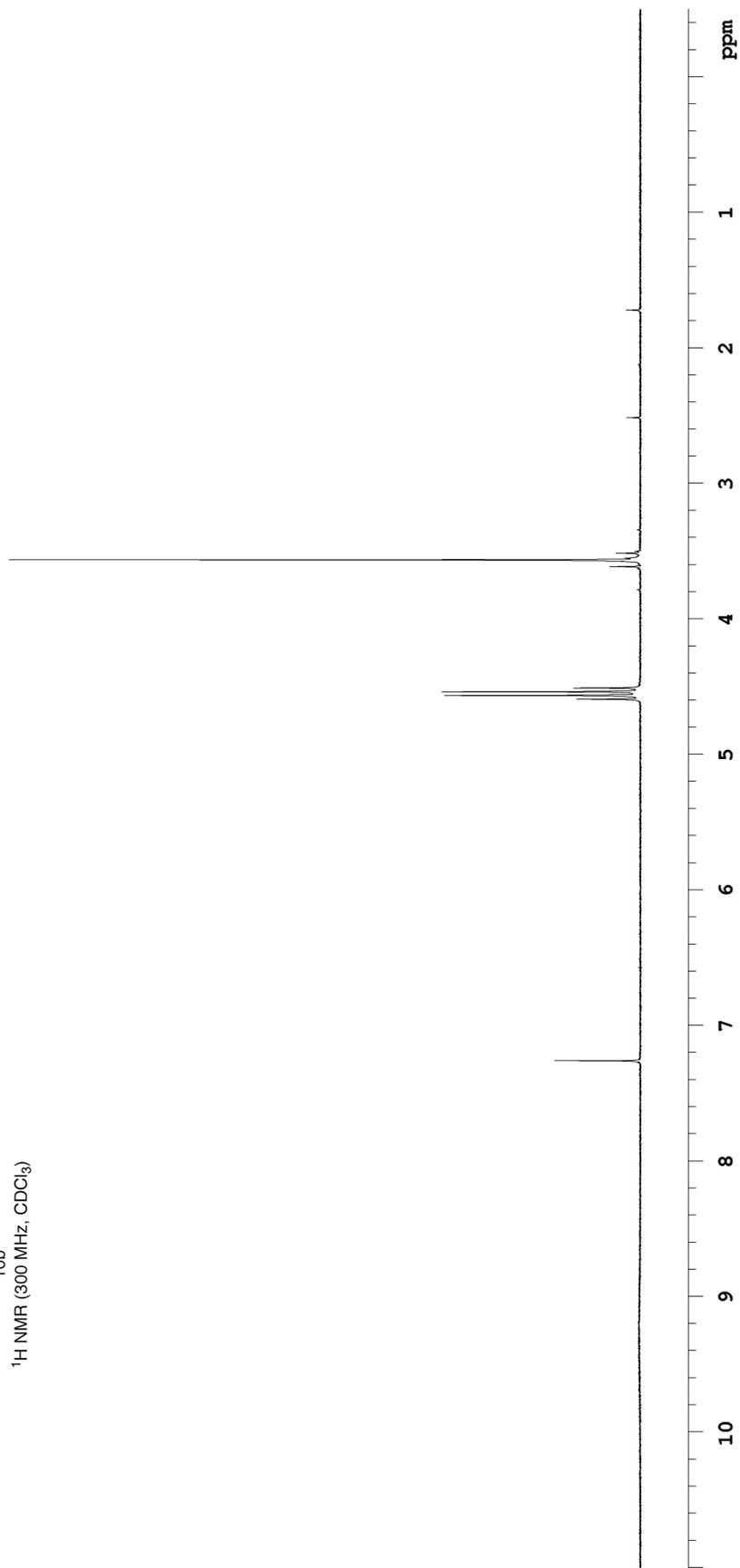
10b

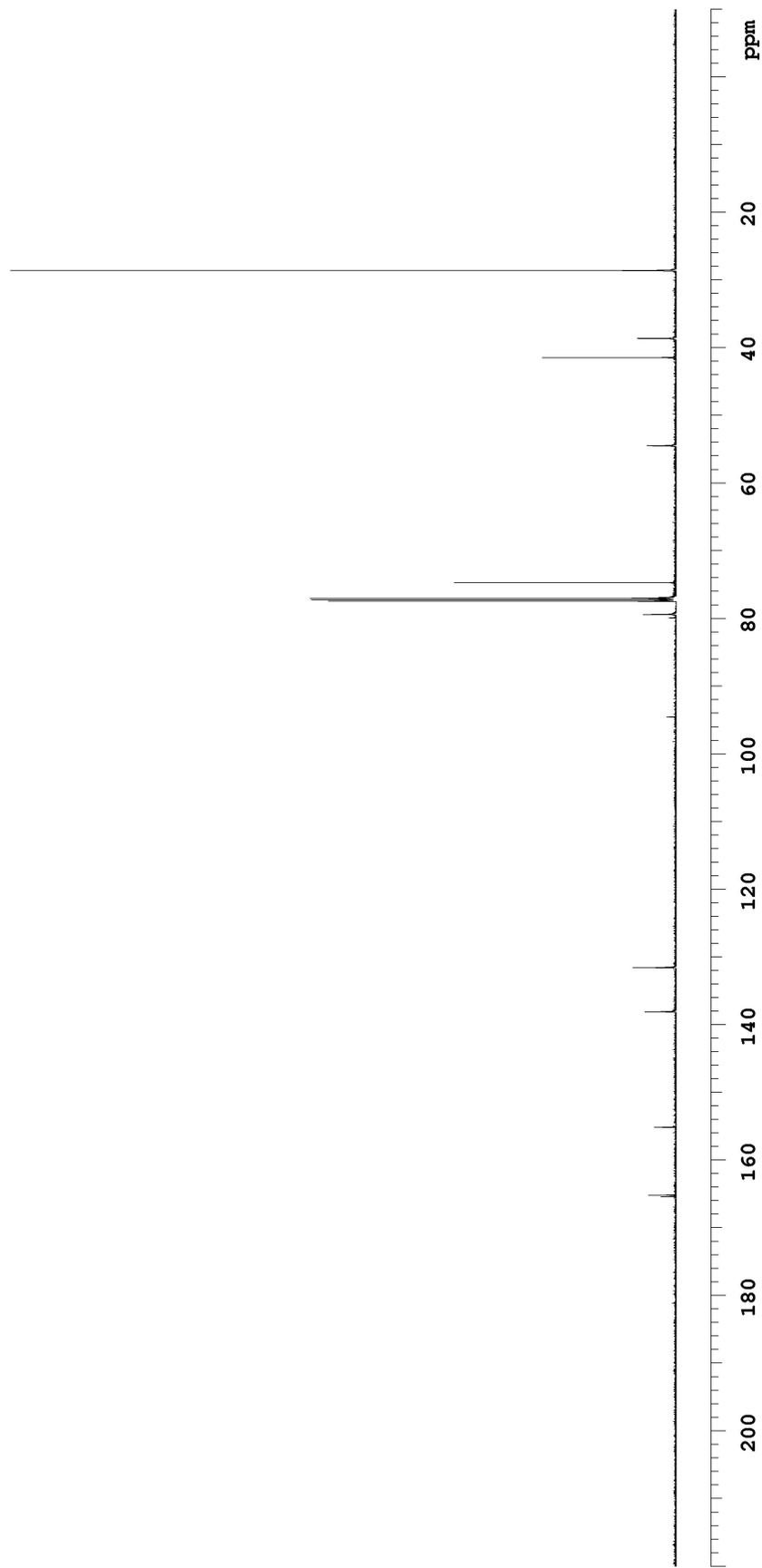
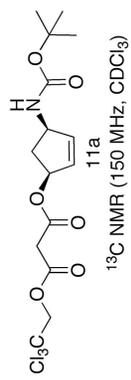
^{13}C NMR (125 MHz, CDCl_3)

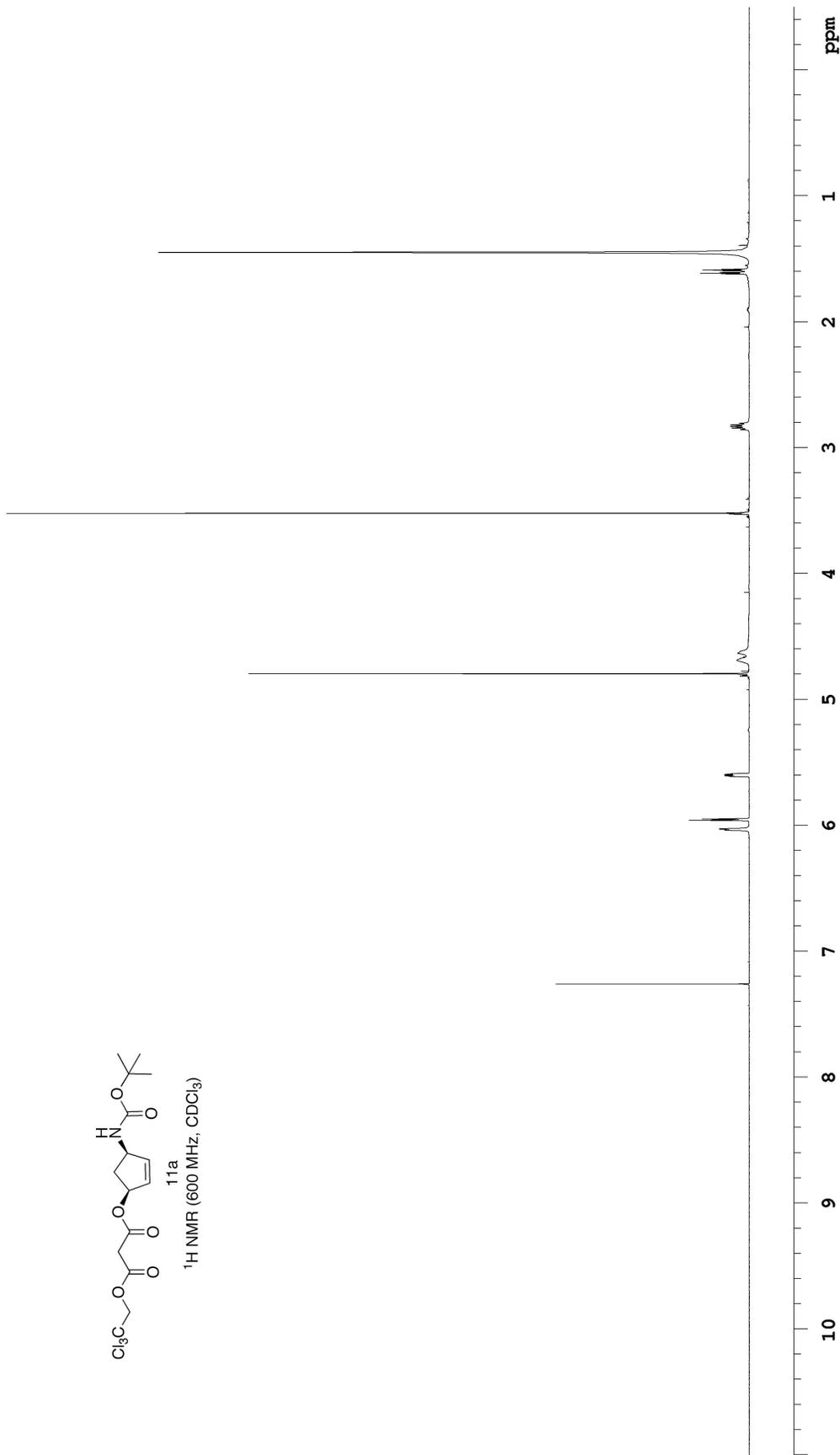
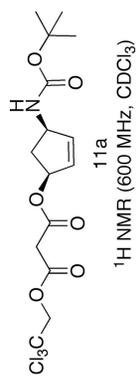


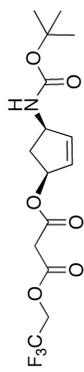


^{10b}
¹H NMR (300 MHz, CDCl₃)



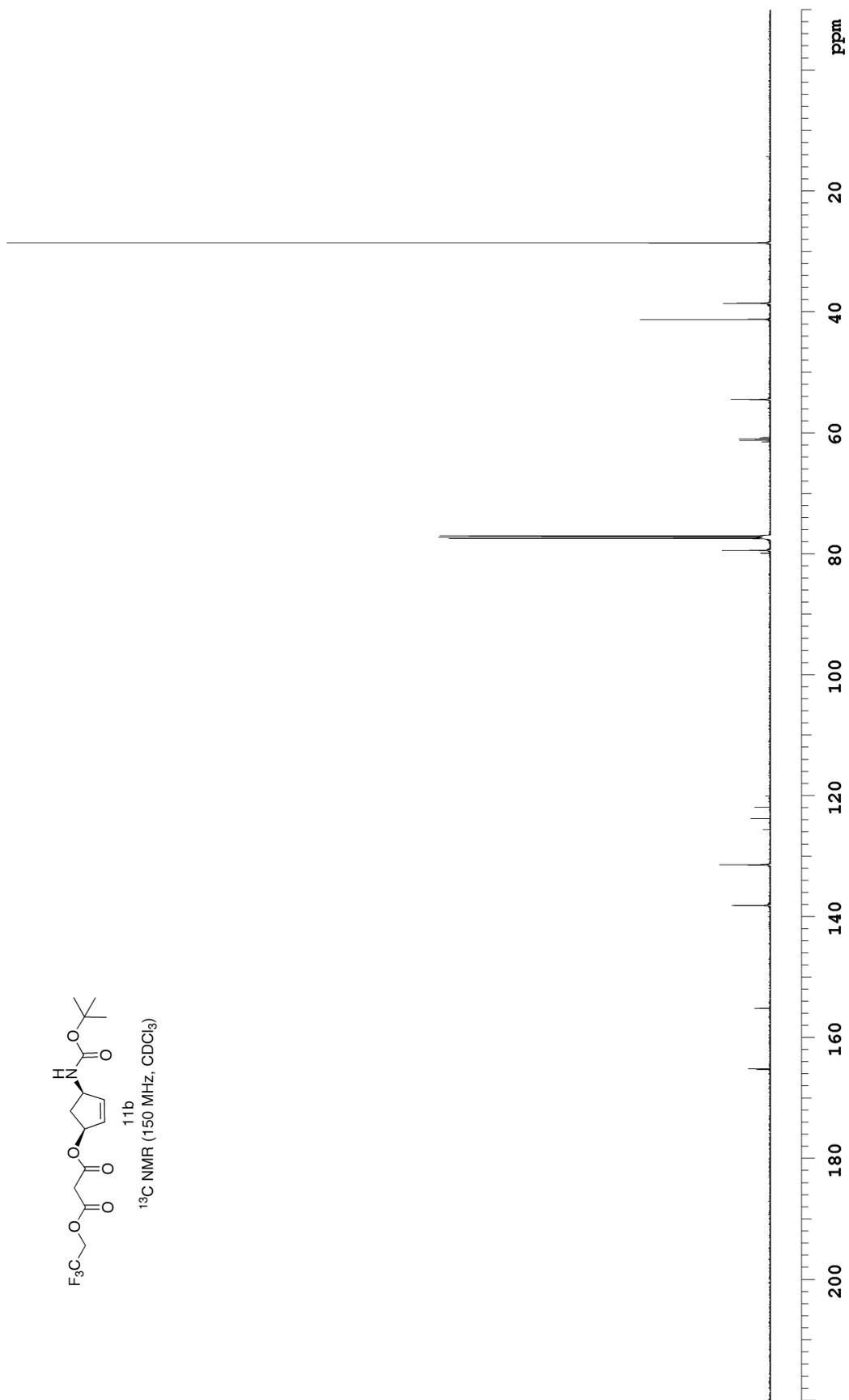


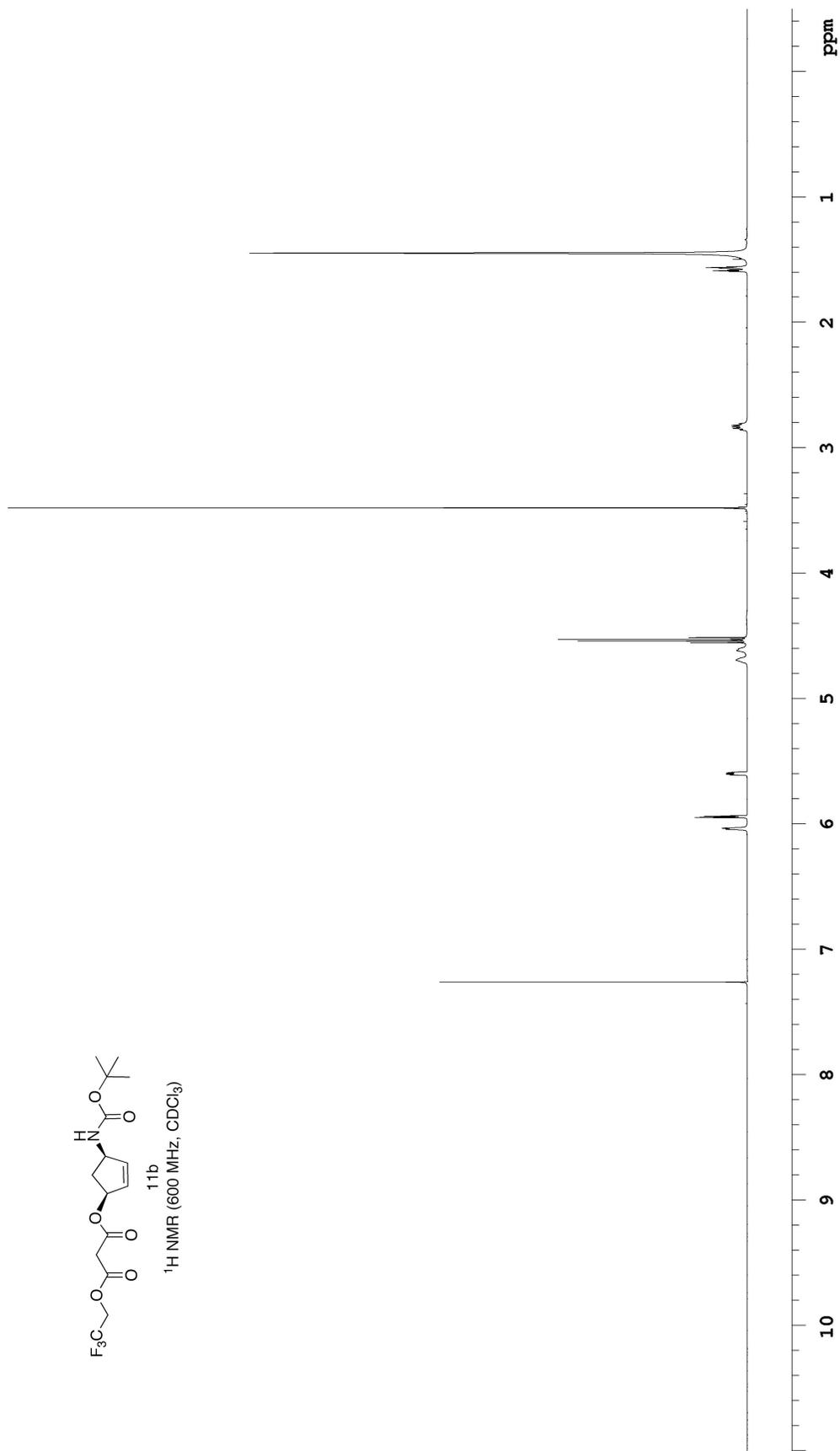
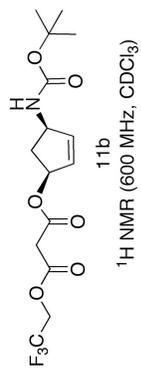


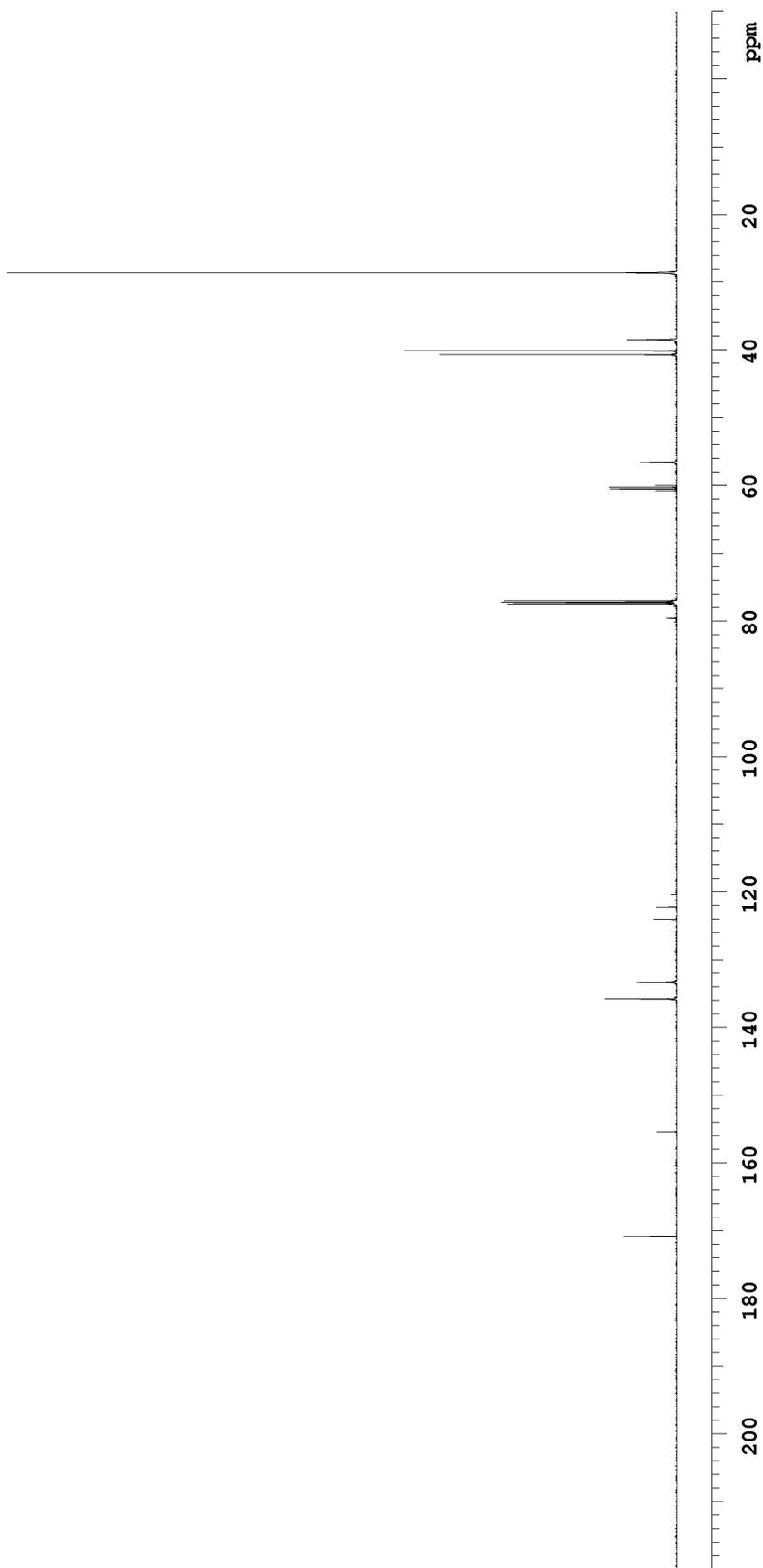
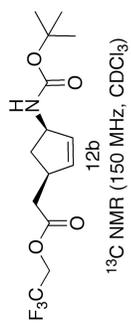


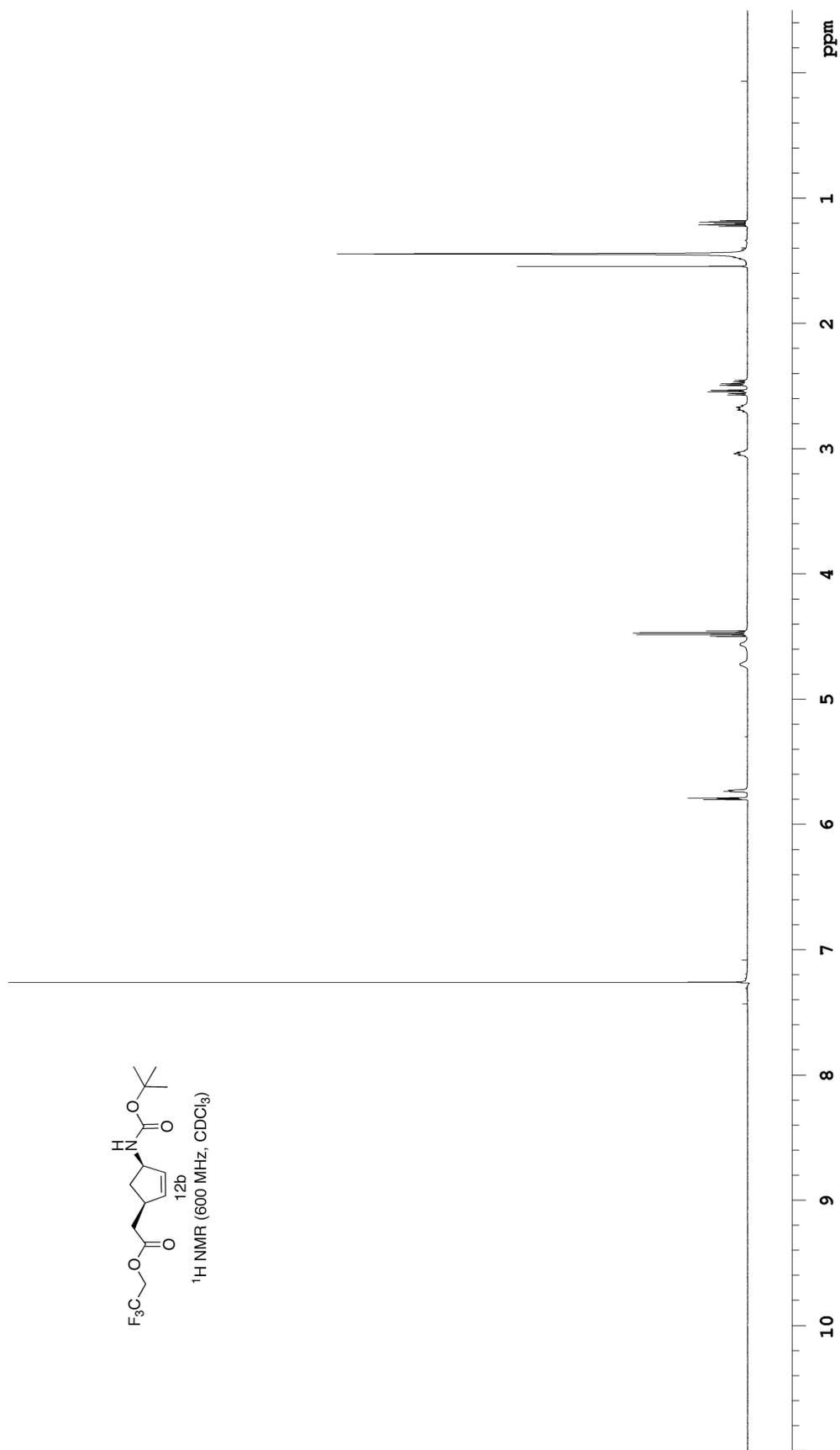
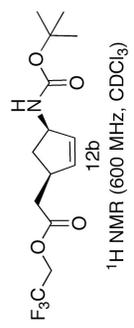
11b

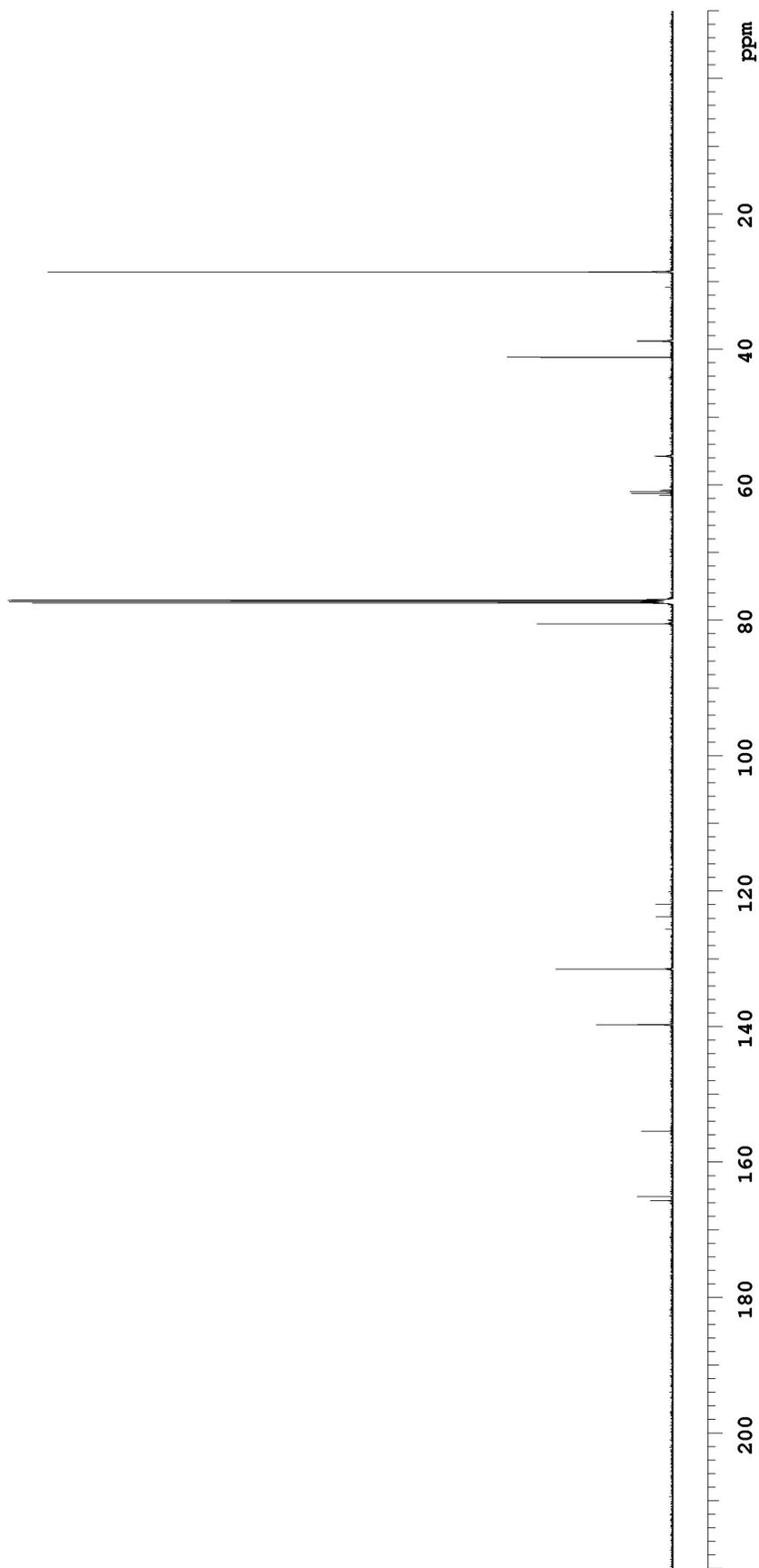
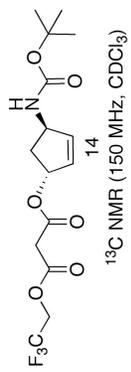
¹³C NMR (150 MHz, CDCl₃)

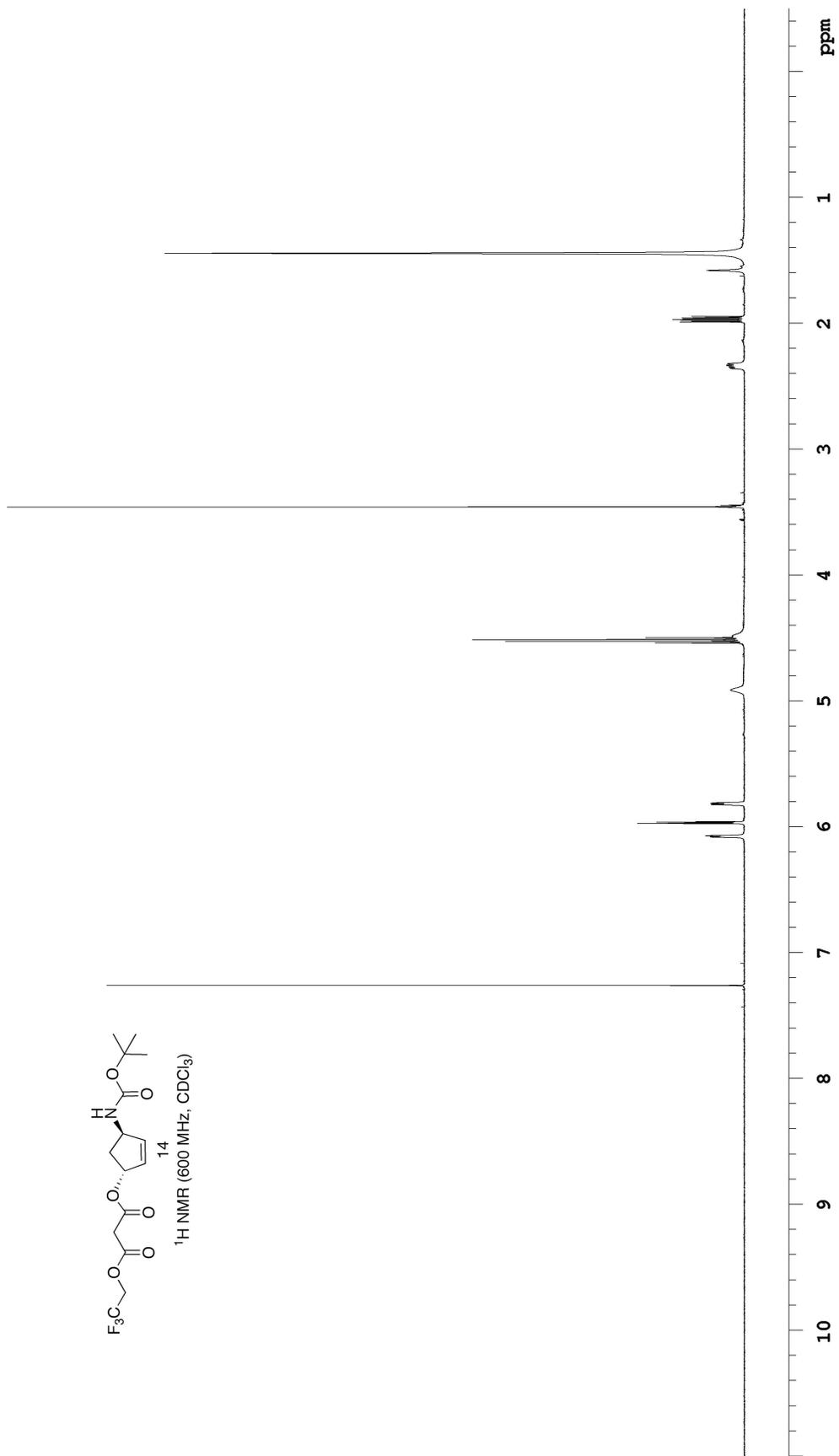
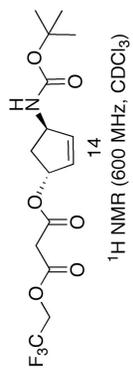


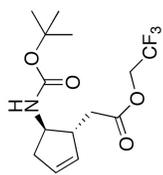






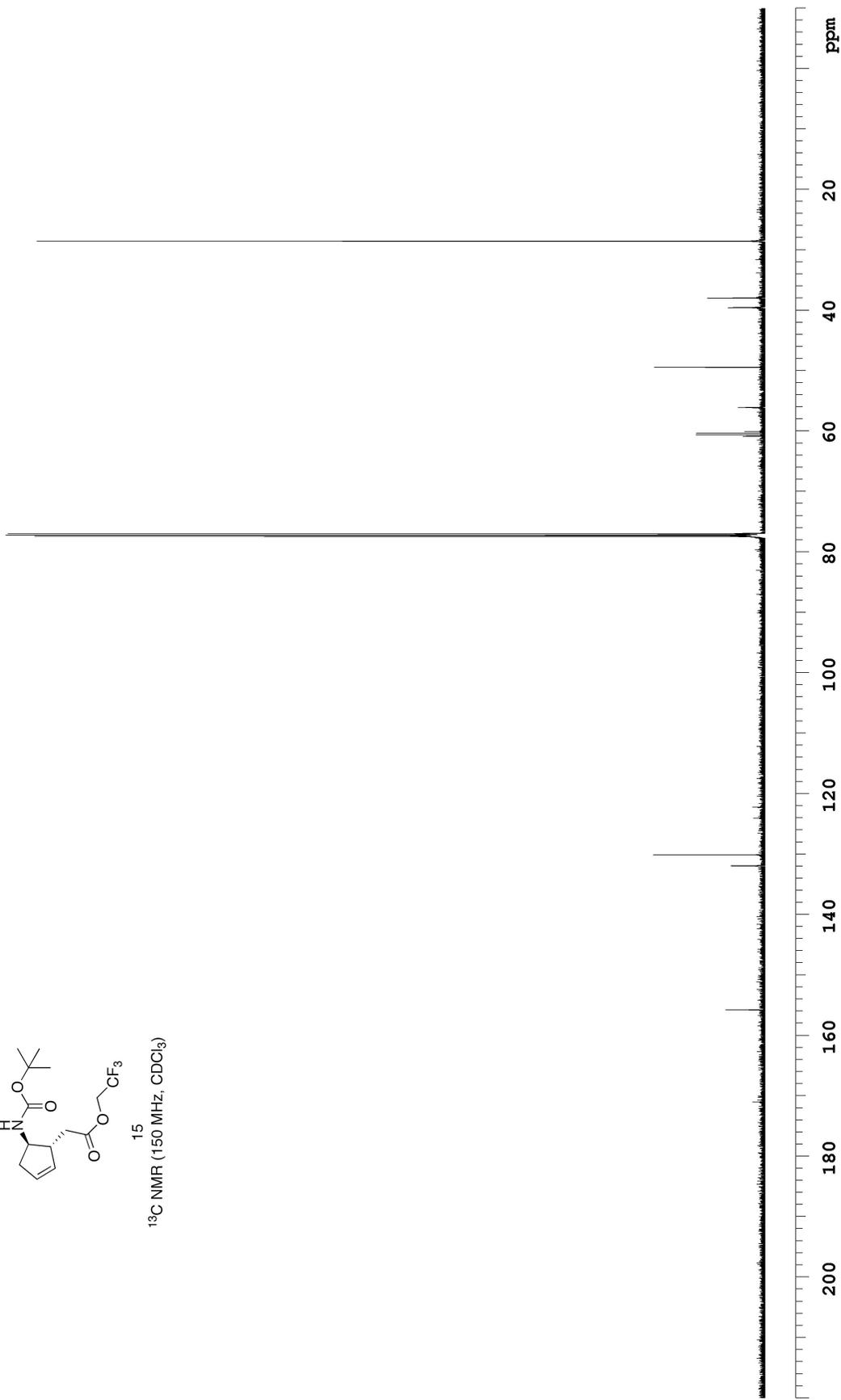


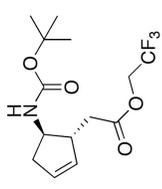




15

^{13}C NMR (150 MHz, CDCl_3)





15
¹H NMR (600 MHz, CDCl₃)

