



Title	Efficient synthesis of α, β -dichlorinated ketones from α, β -dichlorinated Weinreb amides through a simple work-up procedure
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Citation	Organic & Biomolecular Chemistry, 19(36), 7822-7826 https://doi.org/10.1039/d1ob01379c
Issue Date	2021-09-28
Doc URL	https://hdl.handle.net/2115/86845
Type	journal article
File Information	Supplementary files.pdf, Supplementary information



General Methods.

The IR spectra were recorded on a JASCO FTIR-4100 Type A spectrometer using a NaCl cell. The ^1H NMR and ^{13}C NMR spectra were recorded using a JNM-EX 400 (400 MHz and 100 MHz) spectrometer. Chemical shifts were reported in ppm relative to CHCl_3 in CDCl_3 for ^1H NMR ($\delta = 7.26$) and ^{13}C NMR ($\delta = 77.0$) and CHD_2OH in CD_3OD for ^1H NMR ($\delta = 3.35$) and ^{13}C NMR ($\delta = 49.3$). Splitting patterns for ^1H NMR were designated as “s, d, t, q, m, dt, dd, and td”. These symbols indicate “singlet, doublet, triplet, quartet, multiplet, doublettriplet, doubletdoublet, and tripletdoublet” respectively. All commercially obtained reagents were employed as received. Analytical TLC was carried out using pre-coated silica gel plates (Wako TLC Silicagel 70F₂₅₄). Wakogel 60N 63-212 μm was used for column chromatography. Reversed-phase high performance liquid chromatography (HPLC) was carried out using HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm , $\phi 8.0 \times 250$ mm).

Ketone **3a** and **4a**

To a solution of 1-hexyne (0.109 mL, 0.957 mmol) in THF (5.0 mL) was added *n*BuLi (0.601 mL, 0.957 mmol, 1.59 M) at 0 °C to give **1a**. After 30 minutes, a solution of **2** (111 mg, 0.319 mmol) in THF (3.0 mL) was added. The mixture was stirred for 10 minutes at 0 °C, quenched with 0.390 mL of 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (116 mg, 98%) of dichloro ketone **3a** and unsaturated ketone **4a** with ratio 85:15. For further purification, the partial (ca. 10.0 mg) of mixture products were separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm , $\phi 8.0 \times 250$ mm, elution with H_2O :Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **3a** as a colorless oil and **4a** as a colorless oil.

3a: IR (neat) 3019, 2960, 2936, 2214, 1726, 1683, 1647, 1215, 756, 699, 669 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 0.94 (3H, t, $J = 7.3$ Hz), 1.46-1.81 (9H, m), 2.06-2.18 (1H, m), 2.48 (2H, t, $J = 6.8$ Hz), 3.51 (2H, t, $J = 5.8$ Hz), 4.35 (1H, td, $J = 9.2, 2.4$ Hz), 4.46 (1H, d, $J = 8.8$ Hz), 4.49 (2H, s), 7.24-7.33 (5H, m); ^{13}C NMR (CD_3OD , 100 MHz) δ 13.7, 19.2, 22.9, 23.4, 29.9, 30.7, 34.4, 61.6, 66.9, 70.8, 73.9, 78.8, 100.2, 128.6, 128.8, 129.3, 139.8, 179.8; HRMS (ESI) m/z : [$\text{M} + \text{Na}$] $^+$; Calcd for $\text{C}_{20}\text{H}_{26}\text{O}_2\text{Cl}_2\text{Na}$ 391.1202; Found 391.1201.

4a: IR (neat) 3065, 3019, 2959, 2936, 2865, 2214, 1716, 1649, 1617, 1216, 759, 698, 667 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 0.94 (3H, t, $J = 7.3$ Hz), 1.43-1.50 (2H, m), 1.46-1.66 (6H, m), 2.45-2.52 (4H, m), 3.51 (2H, t, $J = 6.3$ Hz), 4.49 (2H, s), 7.25-7.32 (5H, m), 7.46 (1H, t, $J = 7.3$ Hz); ^{13}C NMR (CD_3OD , 100 MHz) δ 13.8, 19.2, 23.0, 25.5, 30.3, 30.6, 30.8, 70.7, 73.9, 78.5, 98.6, 128.6, 128.8, 129.3, 136.1, 139.7, 149.3, 173.0; HRMS (ESI) m/z : [$\text{M} + \text{Na}$] $^+$; Calcd for

C₂₀H₂₅O₂ClNa 355.1435; Found 355.1438.

Ketone **3b**

To a solution of **2** (157 mg, 0.451 mmol) in THF (5.0 mL) was added *n*BuLi (**1b**) (0.427 mL, 0.680 mmol, 1.59 M) at -20 °C. The mixture was stirred for 30 minutes, quenched with 0.560 mL 4.00 M HCl in 1,4-dioxane then excess of H₂O, extracted with EtOAc, washed with brine, dried over Na₂SO₄, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (155 mg, 98%) of dichloro ketone **3b** and unsaturated ketone **4b**. For further purification to obtain pure **3b**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm, φ8.0×250 mm, elution with H₂O:Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **3b** as a colorless oil: IR (neat) 3063, 3029, 2957, 2933, 2868, 1729, 1495, 1454, 1103, 735, 698, 664 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 0.91 (3H, t, *J* = 7.3 Hz), 1.31-1.38 (2H, m), 1.53-1.79 (7H, m), 2.08-2.10 (1H, m), 2.64-2.75 (2H, m), 3.50 (2H, t, *J* = 5.8 Hz), 4.32 (1H, td, *J* = 9.2, 2.4 Hz), 4.49 (2H, s), 4.51 (1H, d, *J* = 8.7 Hz), 7.24-7.33 (5H, m); ¹³C NMR (CD₃OD, 100 MHz) δ 14.1, 23.1, 23.3, 26.4, 29.9, 34.6, 40.7, 61.5, 64.3, 70.9, 73.9, 128.6, 128.8, 129.3, 139.7, 203.7; HRMS (ESI) *m/z*: [M + Na]⁺; Calcd for C₁₈H₂₆O₂Cl₂Na 367.1202; Found 367.1202.

Ketone **3c**

To a solution of **2** (50.0 mg, 0.144 mmol) in THF (2.0 mL) was added PhLi (**1c**) (0.120 mL, 0.216 mmol, 1.80 M) at -20 °C. The mixture was stirred for 30 minutes, quenched with 0.200 mL 4.00 M HCl in 1,4-dioxane then excess of H₂O, extracted with EtOAc, washed with brine, dried over Na₂SO₄, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (49.0 mg, 94%) of dichloro ketone **3c** and unsaturated ketone **4c**. For further purification to obtain pure **3c**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm, φ8.0×250 mm, elution with H₂O:Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **3c** as a colorless oil: IR (neat) 3062, 3030, 2941, 2864, 1695, 1596, 1580, 1449, 1100, 738, 688, 663 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.27-1.93 (5H, m), 2.24-2.30 (1H, m), 3.53 (2H, t, *J* = 5.8 Hz), 4.50 (2H, s), 4.51 (1H, td, *J* = 9.2, 2.4 Hz), 5.53 (1H, d, *J* = 9.7 Hz), 7.24-7.33 (5H, m), 7.54 (2H, dd, *J* = 8.7, 7.8 Hz), 7.64-7.68 (1H, m), 8.05 (2H, dd, *J* = 7.3, 1.4 Hz); ¹³C NMR (CD₃OD, 100 MHz) δ 23.3, 30.0, 34.5, 58.5, 61.4, 70.9, 73.9, 127.7, 128.6, 128.8, 129.3, 130.0, 135.3, 136.0, 139.7, 193.2; HRMS (ESI) *m/z*: [M + Na]⁺; Calcd for C₂₀H₂₂O₂Cl₂Na 387.0889; Found 387.0888.

Ketone **3d**

To a solution of **2** (114 mg, 0.327 mmol) in THF (4.0 mL) was added CH_3MgBr (**1d**) (0.163 mL, 0.491 mmol, 3.00 M) at 0 °C. The mixture was stirred for 2 hours, quenched with 0.460 mL 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (97.0 mg, 98%) of dichloro ketone **3d** and unsaturated ketone **4d**. For further purification to obtain pure **3d**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm , $\phi 8.0 \times 250$ mm, elution with H_2O :Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **3d** as a colorless oil: IR (neat) 3031, 2940, 2866, 1721, 1495, 1454, 1100, 739, 714, 698 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 1.53-1.80 (5H, m), 2.07-2.17 (1H, m), 2.31 (3H, s), 3.50 (2H, t, $J = 5.8$ Hz), 4.33 (1H, td, $J = 9.2, 2.4$ Hz), 4.49 (2H, s), 4.50 (1H, d, $J = 7.3$ Hz), 7.26-7.34 (5H, m); ^{13}C NMR (CD_3OD , 100 MHz) δ 23.4, 26.9, 29.9, 34.6, 61.6, 65.4, 70.9, 73.9, 128.6, 128.8, 129.3, 139.7, 201.4; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$; Calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2\text{Cl}_2\text{Na}$ 325.0732; Found 325.0728.

Ketone **3e**

To a solution of **2** (131 mg, 0.376 mmol) in THF (4.0 mL) was added VinylMgCl (**1e**) (0.427 mL, 0.680 mmol, 1.35 M) at 0 °C. The mixture was stirred for 2 hours, quenched with 0.460 mL 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (115 mg, 97%) of dichloro ketone **3e** and unsaturated ketone **4e**. For further purification to obtain pure **3e**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm , $\phi 8.0 \times 250$ mm, elution with H_2O :Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **3e** as a colorless oil: IR (neat) 3063, 3029, 2930, 2866, 1725, 1558, 1455, 1275, 1101, 714, 700, 667 cm^{-1} ; ^1H NMR (CDCl_3 , 400 MHz) δ 1.61-1.81 (5H, m), 2.13-2.17 (1H, m), 3.50 (2H, t, $J = 5.8$ Hz), 4.31 (1H, td, $J = 9.2, 2.4$ Hz), 4.49 (2H, s), 4.51 (1H, d, $J = 8.8$ Hz), 5.96 (1H, dd, $J = 10.4, 0.9$ Hz), 6.45 (1H, dd, $J = 17.5, 0.9$ Hz), 6.62 (1H, dd, $J = 17.5, 10.2$ Hz), 7.26-7.35 (5H, m); ^{13}C NMR (CDCl_3 , 100 MHz) δ 22.2, 29.1, 33.6, 59.7, 61.5, 69.8, 72.9, 127.5, 127.6, 128.3, 131.5, 132.1, 138.4, 190.7; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$; Calcd for $\text{C}_{16}\text{H}_{20}\text{O}_2\text{Cl}_2\text{Na}$ 337.0732; Found 337.0728.

Ketone **3f**

To a solution of **2** (110 mg, 0.315 mmol) in THF (5.0 mL) was added $n\text{C}_5\text{H}_{11}\text{MgBr}$ (**1f**) (0.236 mL, 0.473 mmol, 2.00 M) at 0 °C. The mixture was stirred for 2 hours, quenched with 0.390 mL 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (109 mg, 96%) of dichloro ketone **3f** and unsaturated ketone **4f**. For further purification to obtain pure **3f**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm , $\phi 8.0 \times 250$ mm, elution with H_2O :Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **3f** as a colorless oil: IR (neat) 3064, 3031, 2931, 2862, 1730, 1558, 1455, 1274, 1101, 739, 698, 663 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 0.90 (3H, t, J = 6.8 Hz), 1.27-1.36 (4H, m), 1.53-1.79 (7H, m), 2.07-2.16 (1H, m), 2.63-2.74 (2H, m), 3.51 (2H, t, J = 5.8 Hz), 4.32 (1H, td, J = 9.2, 2.4 Hz), 4.49 (2H, s), 4.51 (1H, d, J = 9.3 Hz), 7.23-7.33 (5H, m); ^{13}C NMR (CD_3OD , 100 MHz) δ 14.3, 23.3, 23.5, 24.1, 29.9, 32.2, 34.6, 40.9, 61.5, 64.3, 70.9, 73.9, 128.6, 128.8, 129.3, 139.7, 203.7; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$; Calcd for $\text{C}_{19}\text{H}_{28}\text{O}_2\text{Cl}_2\text{Na}$ 381.1358; Found 381.1353.

Ketone **3g**

To a solution of **2** (111 mg, 0.319 mmol) in THF (4.0 mL) was added PhMgBr (**1g**) (0.159 mL, 0.478 mmol, 3.00 M) at 0 °C. The mixture was stirred for 4 hours, quenched with 0.390 mL 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (111 mg, 96%) of dichloro ketone **3g** and unsaturated ketone **4g**. For further purification to obtain pure **3g**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm , $\phi 8.0 \times 250$ mm, elution with H_2O :Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **3g** as a colorless oil: IR (neat) 3063, 3030, 2939, 2865, 1695, 1596, 1580, 1449, 1273, 1100, 746, 688, 663 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 1.27-1.93 (5H, m), 2.24-2.30 (1H, m), 3.53 (2H, t, J = 5.8 Hz), 4.50 (2H, s), 4.51 (1H, td, J = 9.2, 2.4 Hz), 5.53 (1H, d, J = 9.7 Hz), 7.24-7.33 (5H, m), 7.54 (2H, dd, J = 8.7, 7.8 Hz), 7.64-7.68 (1H, m), 8.05 (2H, dd, J = 7.3, 1.4 Hz); ^{13}C NMR (CD_3OD , 100 MHz) δ 23.3, 30.0, 34.5, 58.5, 61.4, 70.9, 73.9, 127.7, 128.6, 128.8, 129.3, 130.0, 135.3, 136.0, 139.7, 193.2; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$; Calcd for $\text{C}_{20}\text{H}_{22}\text{O}_2\text{Cl}_2\text{Na}$ 387.0889; Found 387.0888.

Ketone **3h** and **4h**

To a solution of **2** (64.2 mg, 0.184 mmol) in THF (2.0 mL) was added HC≡CMgCl (**1h**) (0.553 mL, 0.277 mmol, 0.500 M) at 0 °C. The mixture was stirred for 4 hours, quenched with 0.230 mL 4.00 M HCl in 1,4-dioxane then excess of H₂O, extracted with EtOAc, washed with brine, dried over Na₂SO₄, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (52.0 mg, 90%) of dichloro ketone **3h** and unsaturated ketone **4h** with ratio 35:65. For further purification, the partial (ca. 10.0 mg) of mixture products were separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm, φ8.0×250 mm, elution with H₂O:Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **3h** as a colorless oil and **4h** as a colorless oil.

3h: IR (neat) 3273, 3066, 2921, 2851, 2098, 1697, 1660, 1587, 1455, 1259, 1101, 909, 735, 698 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.55-1.80 (5H, m), 2.10-2.16 (1H, m), 3.41 (1H, s), 3.50 (2H, t, *J* = 5.8 Hz), 4.31-4.32 (2H, m), 4.51 (2H, s), 7.25-7.35 (5H, m); ¹³C NMR (CDCl₃, 100 MHz) δ 22.1, 28.9, 33.3, 59.5, 64.8, 69.8, 72.9, 78.1, 82.5, 127.5, 127.6, 128.3, 138.4, 177.7; HRMS (ESI) *m/z*: [M + Na]⁺; Calcd for C₁₆H₁₈O₂Cl₂Na 335.0576; Found 335.0581.

4h: IR (neat) 3251, 3064, 2925, 2855, 2096, 1717, 1659, 1614, 1455, 1233, 1101, 883, 733, 698 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.67-1.70 (4H, m), 2.53 (2H, q, *J* = 7.3 Hz), 3.34 (1H, s), 3.51 (2H, t, *J* = 5.8 Hz), 4.51 (2H, s), 7.26-7.35 (5H, m), 7.46 (1H, t, *J* = 7.3 Hz); ¹³C NMR (CDCl₃, 100 MHz) δ 24.3, 29.3, 29.8, 69.6, 72.9, 78.5, 81.3, 127.5, 127.5, 128.3, 134.9, 138.3, 149.3, 171.1; HRMS (ESI) *m/z*: [M + Na]⁺; Calcd for C₁₆H₁₇O₂ClNa 299.0809; Found 299.0801.

Amide **6**

To a solution of **2** (182 mg, 0.524 mmol) in THF (6.0 mL) was added *i*PrMgBr (**1i**) (1.07 mL, 0.786 mmol, 0.730 M) at 0 °C. The mixture was stirred for 4 hours, quenched with 0.650 mL 4.00 M HCl in 1,4-dioxane then excess of H₂O, extracted with EtOAc, washed with brine, dried over Na₂SO₄, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give unsaturated amide **6** (135 mg, 0.487 mmol, 93%) as a colorless oil: IR (neat) 3063, 3029, 2936, 2857, 1664, 1633, 1495, 1455, 1412, 1379, 1273, 1104, 997, 746, 737, 698 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.54-1.60 (2H, m), 1.61-1.67 (2H, m), 2.25 (2H, q, *J* = 7.3 Hz), 3.23 (3H, s), 3.48 (2H, t, *J* = 6.3 Hz), 3.68 (3H, s), 4.49 (2H, s), 6.39 (1H, d, *J* = 15.6 Hz), 6.97 (1H, dt, *J* = 15.7, 6.8 Hz), 7.26-7.34 (5H, m); ¹³C NMR (CDCl₃, 100 MHz) δ 24.8, 29.1, 32.1, 32.7, 61.5, 69.8, 72.7, 118.7, 127.4, 127.5, 128.2, 138.3, 147.4, 166.8; HRMS (ESI) *m/z*: [M + Na]⁺; Calcd for C₁₆H₂₃NO₃Na 300.1567; Found 300.1567.

Ketone **8a**

To a solution of 1-hexyne (0.083 mL, 0.722 mmol) in THF (4.0 mL) was added *n*BuLi (0.453 mL,

0.722 mmol, 1.59 M) at 0 °C to give **1a**. The mixture was cooled at -40 °C and added cold solution of **7** (84.0 mg, 0.241 mmol) in THF (3.0 mL), stirred for 10 minutes at -40 °C, quenched with 0.310 mL 4.00 M HCl in 1,4-dioxane then excess of H₂O, extracted with EtOAc, washed with brine, dried over Na₂SO₄, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (84.0 mg, 95%) of dichloro ketone **8a** and unsaturated ketone **4a**. For further purification to obtain pure **8a**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 µm, φ8.0×250 mm, elution with H₂O:Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **8a** as a colorless oil: IR (neat) 3030, 2956, 2933, 2863, 2211, 1697, 1671, 1653, 1102, 771, 736, 698 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 0.75 (3H, t, *J* = 7.3 Hz), 1.25-1.46 (8H, m), 1.71 (2H, q, *J* = 7.3 Hz), 2.29 (2H, t, *J* = 6.8 Hz), 3.32 (2H, t, *J* = 5.8 Hz), 4.30 (2H, s), 4.49 (1H, td, *J* = 7.1, 3.4 Hz), 4.70 (1H, d, *J* = 3.4 Hz), 7.26-7.33 (5H, m); ¹³C NMR (CD₃OD, 100 MHz) δ 13.7, 19.3, 22.9, 24.2, 29.9, 30.6, 36.6, 62.6, 70.8, 70.9, 73.9, 79.3, 101.1, 128.6, 128.8, 129.3, 139.7, 180.4; HRMS (ESI) *m/z*: [M + Na]⁺; Calcd for C₂₀H₂₆O₂Cl₂Na 391.1202; Found 391.1203.

Ketone **8b**

To a solution of **7** (54.0 mg, 0.155 mmol) in THF (2.0 mL) was added *n*BuLi (**1b**) (0.145 mL, 0.232 mmol, 1.59 M) at -40 °C. The mixture was stirred for 30 minutes, quenched with 0.200 mL 4.00 M HCl in 1,4-dioxane then excess of H₂O, extracted with EtOAc, washed with brine, dried over Na₂SO₄, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (51.0 mg, 95%) of dichloro ketone **8b** and unsaturated ketone **4b**. For further purification to obtain pure **8b**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 µm, φ8.0×250 mm, elution with H₂O:Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **8b** as a colorless oil: IR (neat) 3063, 3030, 2956, 2933, 2865, 1717, 1540, 1455, 1397, 1100, 772, 737, 698 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 0.91 (3H, t, *J* = 7.3 Hz), 1.28-1.37 (2H, m), 1.49-1.66 (6H, m), 1.86 (2H, q, *J* = 6.8 Hz), 2.71 (2H, t, *J* = 7.3 Hz), 3.50 (2H, t, *J* = 5.8 Hz), 4.48 (2H, s), 4.54 (1H, td, *J* = 7.1, 3.4 Hz), 4.77 (1H, d, *J* = 3.4 Hz), 7.24-7.33 (5H, m); ¹³C NMR (CD₃OD, 100 MHz) δ 14.2, 23.1, 24.2, 26.5, 29.9, 36.6, 40.7, 63.1, 69.6, 70.9, 73.9, 128.6, 128.8, 129.3, 139.7, 205.1; HRMS (ESI) *m/z*: [M + Na]⁺; Calcd for C₁₈H₂₆O₂Cl₂Na 367.1202; Found 367.1200.

Ketone **4c**

To a solution of **7** (87.0 mg, 0.249 mmol) in THF (3.0 mL) was added PhLi (**1c**) (0.207 mL, 0.373 mmol, 1.80 M) at $-20\text{ }^{\circ}\text{C}$. The mixture was stirred for 30 minutes, quenched with 0.310 mL 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give unsaturated ketone **4c** (65.0 mg, 0.197 mmol, 78%) as a colorless oil: IR (neat) 3063, 3031, 2941, 2861, 1718, 1688, 1670, 1449, 1273, 1177, 713, 696, 666 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 1.34-1.66 (4H, m), 2.49 (2H, td, $J = 7.3, 6.8\text{ Hz}$), 3.50 (2H, t, $J = 5.8\text{ Hz}$), 4.47 (2H, s), 6.70 (1H, t, $J = 7.3\text{ Hz}$), 7.24-7.30 (5H, m), 7.47 (2H, dd, $J = 8.7, 7.8\text{ Hz}$), 7.57-7.61 (1H, m), 7.65 (2H, dd, $J = 7.3, 1.4\text{ Hz}$); ^{13}C NMR (CD_3OD , 100 MHz) δ 25.5, 30.3, 30.5, 70.8, 73.9, 128.6, 128.8, 129.3, 129.6, 130.3, 133.7, 134.1, 138.2, 139.7, 147.1, 191.9; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$; Calcd for $\text{C}_{20}\text{H}_{21}\text{O}_2\text{ClNa}$ 351.1122; Found 351.1123.

Ketone **8d**

To a solution of **7** (88.0 mg, 0.253 mmol) in THF (3.0 mL) was added CH_3MgBr (**1d**) (0.126 mL, 0.378 mmol, 3.00 M) at $-20\text{ }^{\circ}\text{C}$. The mixture was stirred for 2 hours, quenched with 0.310 mL 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (73.0 mg, 95%) of dichloro ketone **8d** and unsaturated ketone **4d**. For further purification to obtain pure **8d**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm , $\phi 8.0 \times 250\text{ mm}$, elution with H_2O :Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **8d** as a colorless oil: IR (neat) 3032, 2937, 2864, 1717, 1496, 1455, 1099, 1026, 737, 714, 699 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 1.40-1.55 (4H, m), 1.77-1.88 (2H, m), 2.24 (3H, s), 3.41 (2H, t, $J = 5.8\text{ Hz}$), 4.39 (2H, s), 4.46 (1H, td, $J = 9.2, 2.4\text{ Hz}$), 4.69 (1H, d, $J = 2.9\text{ Hz}$), 7.16-7.23 (5H, m); ^{13}C NMR (CD_3OD , 100 MHz) δ 24.2, 27.9, 29.9, 36.7, 62.8, 69.9, 70.9, 73.9, 128.6, 128.8, 129.3, 139.7, 202.9; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$; Calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2\text{Cl}_2\text{Na}$ 325.0732; Found 325.0733.

Ketone **4e**

To a solution of **7** (71.4 mg, 0.205 mmol) in THF (3.0 mL) was added VinylMgCl (**1e**) (0.146 mL, 0.308 mmol, 2.10 M) at $0\text{ }^{\circ}\text{C}$. The mixture was stirred for 2 hours, quenched with 0.250 mL 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give unsaturated ketone **4e** (52.0 mg, 0.187 mmol, 90%) as a colorless oil: IR (neat) 3029, 2932, 2862, 1717, 1652, 1616, 1455, 1274, 1101, 714,

700 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 1.60-1.70 (4H, m), 2.42-2.52 (2H, m), 3.51 (2H, t, J = 5.8 Hz), 4.48 (2H, s), 5.84 (1H, dd, J = 10.4, 1.9 Hz), 6.31 (1H, dd, J = 16.8, 1.9 Hz), 7.11 (1H, dd, J = 17.5, 10.2 Hz), 7.18 (1H, t, J = 6.8 Hz), 7.25-7.32 (5H, m); ^{13}C NMR (CD_3OD , 100 MHz) δ 25.4, 30.3, 30.6, 70.8, 73.9, 128.6, 128.8, 129.3, 130.7, 131.9, 135.2, 139.7, 145.1, 185.7; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$; Calcd for $\text{C}_{16}\text{H}_{19}\text{O}_2\text{ClNa}$ 301.0965; Found 301.0959.

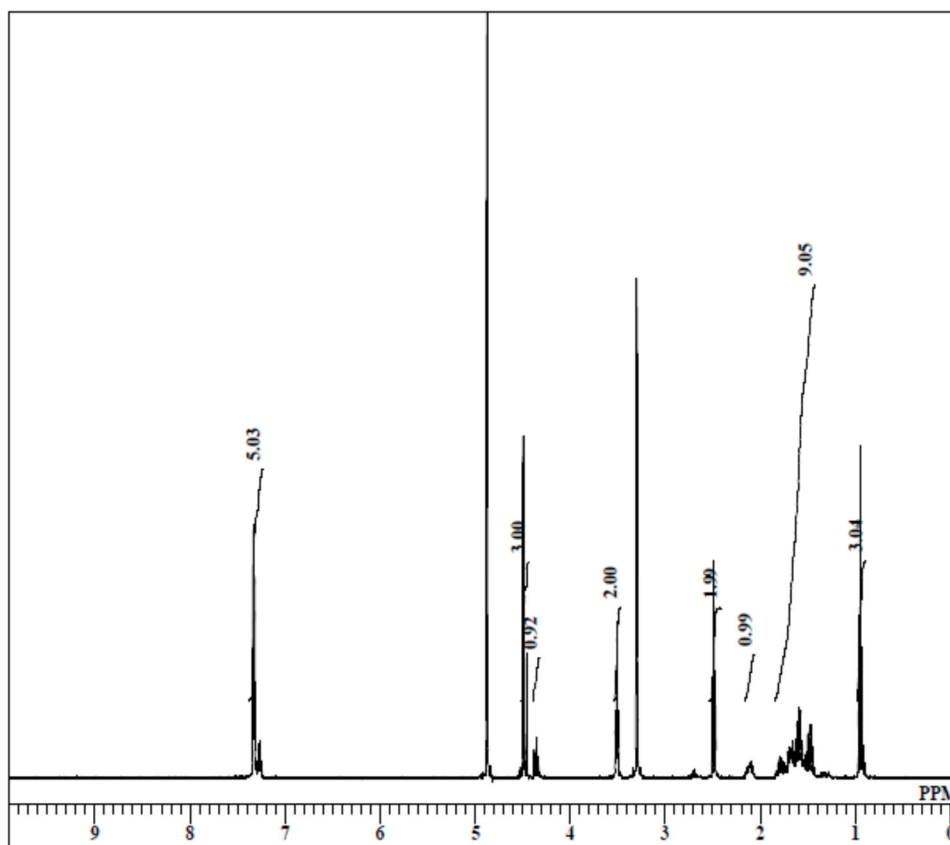
Ketone **8f**

To a solution of **7** (83.0 mg, 0.238 mmol) in THF (3.0 mL) was added $n\text{C}_5\text{H}_{11}\text{MgBr}$ (**1f**) (0.178 mL, 0.358 mmol, 2.00 M) at -20°C . The mixture was stirred for 2 hours, quenched with 0.290 mL 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give a mixture (77.0 mg, 90%) of dichloro ketone **8f** and unsaturated ketone **4f**. For further purification to obtain pure **8f**, a partial (ca. 10.0 mg) of the mixture was separated by HPLC (NOMURA CHEMICAL, DEVELOSIL C30-UG 5 μm , $\phi 8.0 \times 250$ mm, elution with H_2O :Acetonitrile = 80:20 to 0:100 gradiently for 90 min, 2 mL/min) to afford **8f** as a colorless oil: IR (neat) 3068, 3030, 2931, 2859, 1717, 1558, 1455, 1362, 1100, 772, 735, 697 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 0.90 (3H, t, J = 6.8 Hz), 1.27-1.34 (4H, m), 1.48-1.66 (6H, m), 1.87 (2H, q, J = 6.8 Hz), 2.70 (2H, t, J = 6.8 Hz), 3.50 (2H, t, J = 5.8 Hz), 4.48 (2H, s), 4.53 (1H, td, J = 6.8, 3.4 Hz), 4.76 (1H, d, J = 2.9 Hz), 7.24-7.33 (5H, m); ^{13}C NMR (CD_3OD , 100 MHz) δ 14.2, 23.5, 24.1, 24.2, 29.9, 32.2, 36.6, 40.9, 63.0, 69.6, 70.9, 73.9, 128.6, 128.8, 129.3, 139.8, 205.0; HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$; Calcd for $\text{C}_{19}\text{H}_{28}\text{O}_2\text{Cl}_2\text{Na}$ 381.1358; Found 381.1358.

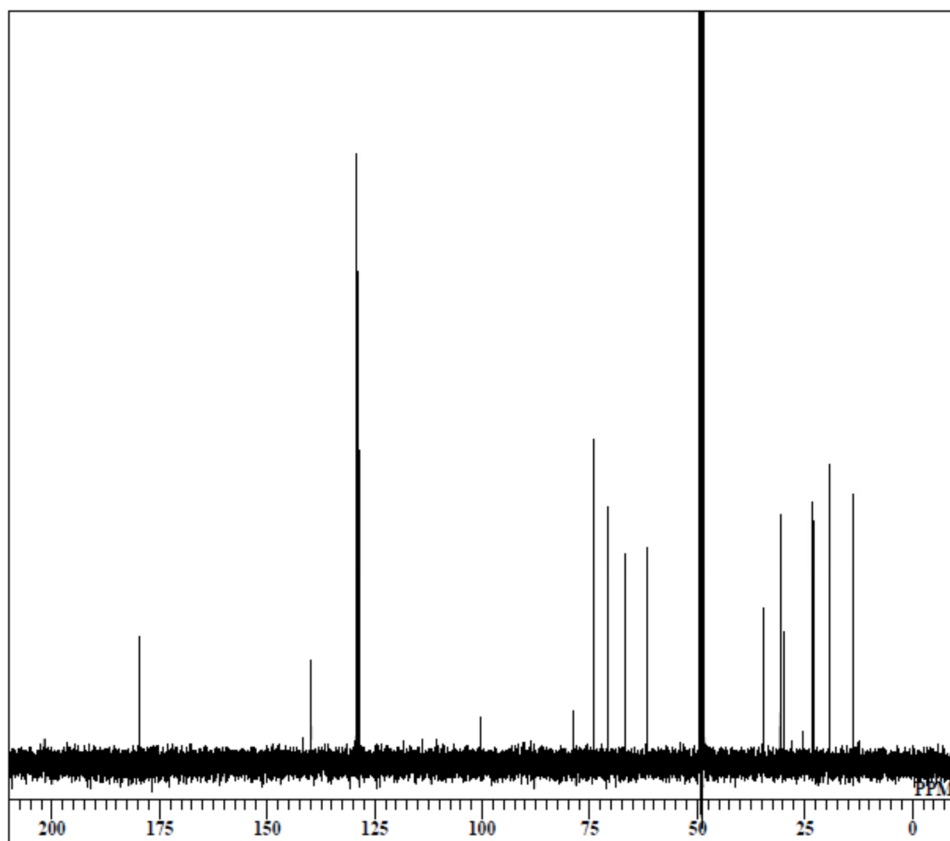
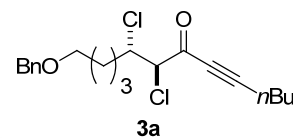
Ketone **4g**

To a solution of **7** (103 mg, 0.295 mmol) in THF (3.0 mL) was added PhMgBr (**1g**) (0.147 mL, 0.442 mmol, 3.00 M) at -20°C . The mixture was stirred for 2 hours, quenched with 0.360 mL 4.00 M HCl in 1,4-dioxane then excess of H_2O , extracted with EtOAc, washed with brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (EtOAc:Hexane = 2:98) to give unsaturated ketone **4g** (62.0 mg, 0.189 mmol, 74%) as a colorless oil: IR (neat) 3063, 3031, 2941, 2861, 1718, 1688, 1670, 1449, 1273, 1177, 713, 696, 666 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 1.34-1.66 (4H, m), 2.49 (2H, q, J = 6.8 Hz), 3.50 (2H, t, J = 5.8 Hz), 4.47 (2H, s), 6.70 (1H, t, J = 7.3 Hz), 7.24-7.30 (5H, m), 7.47 (2H, dd, J = 8.7, 7.8 Hz), 7.57-7.61 (1H, m), 7.65 (2H, dd, J = 7.3, 1.4 Hz); ^{13}C NMR (CD_3OD , 100 MHz) δ 25.5, 30.3, 30.5, 70.8, 73.9, 128.6, 128.8, 129.3, 129.6, 130.3, 133.7, 134.1, 138.2, 139.7, 147.1,

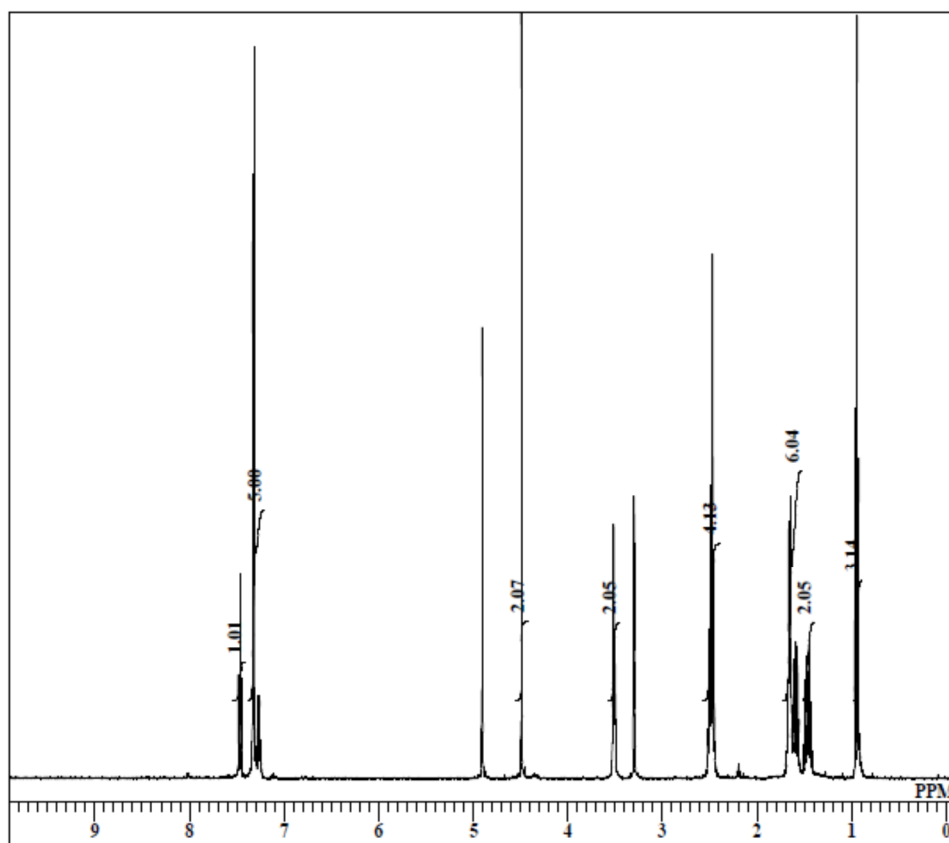
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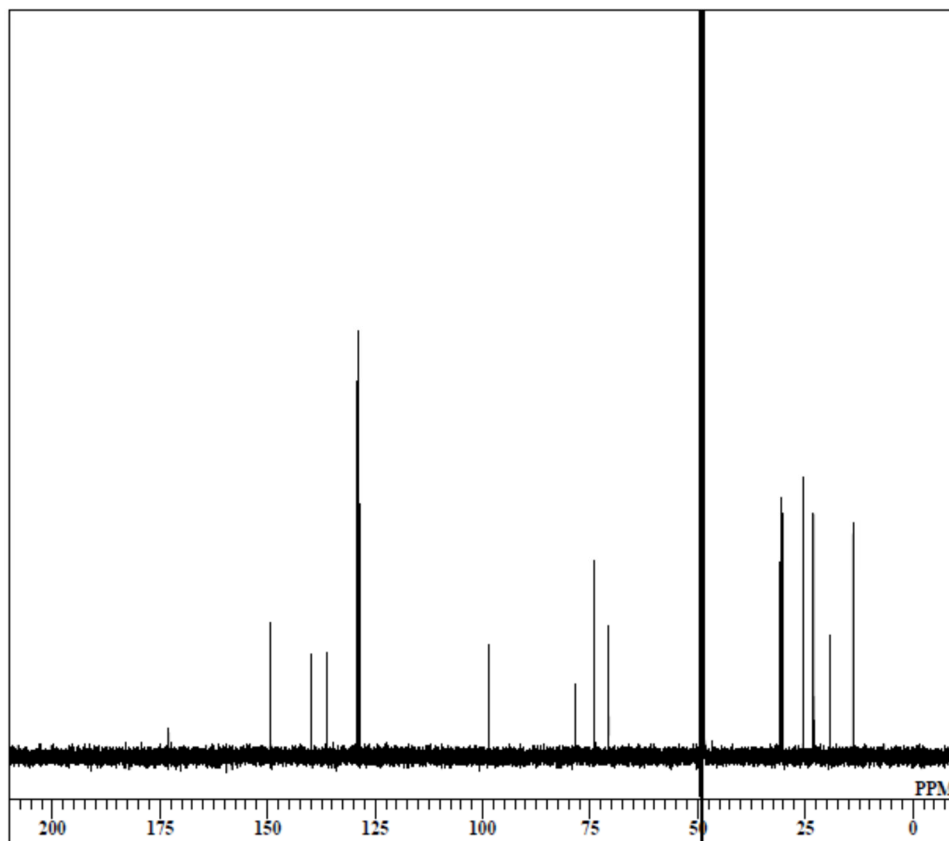
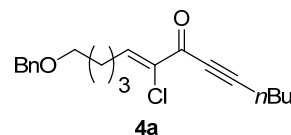
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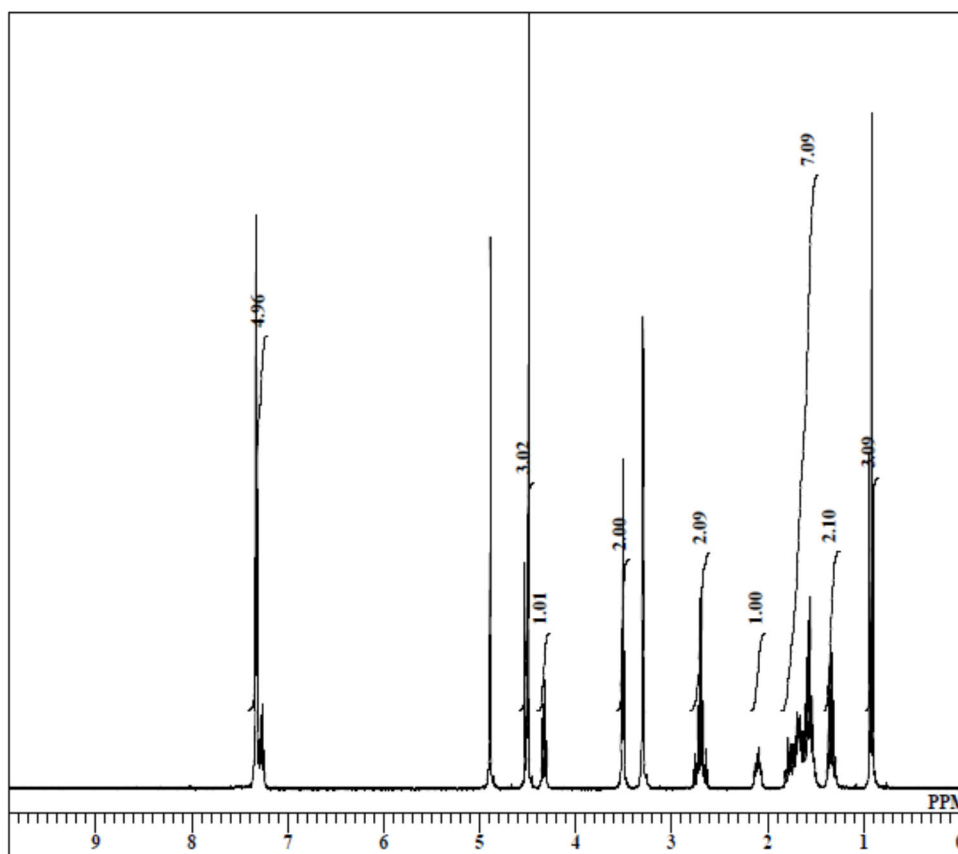
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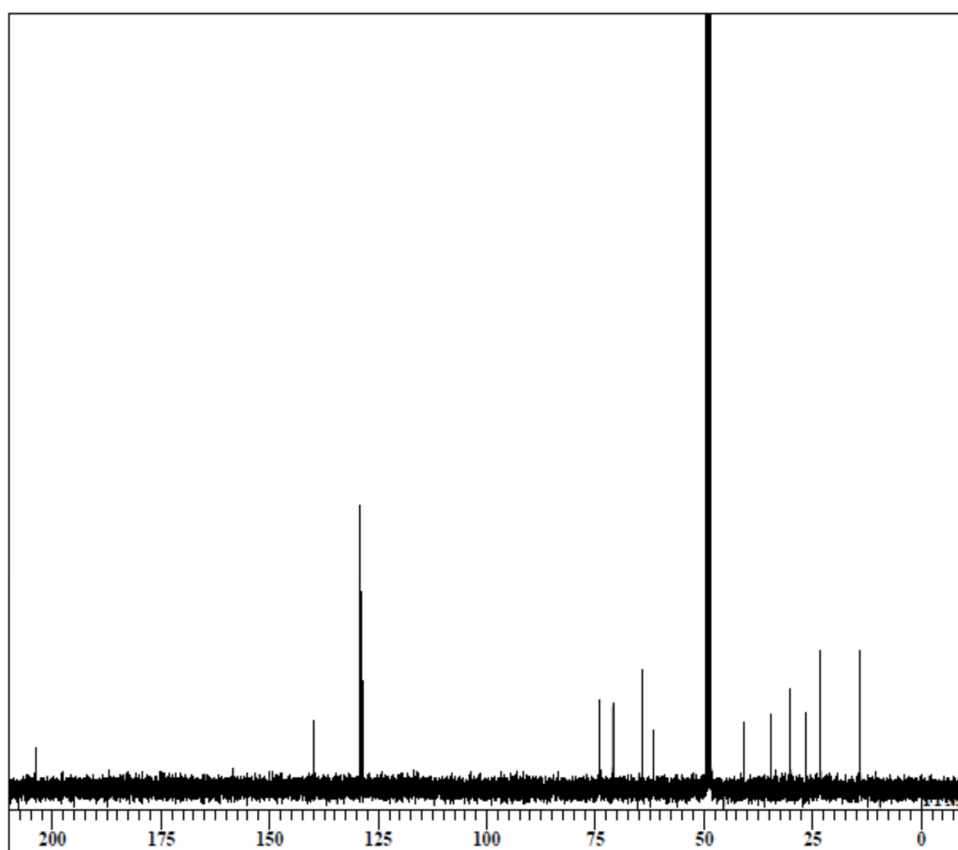
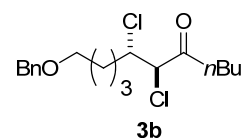
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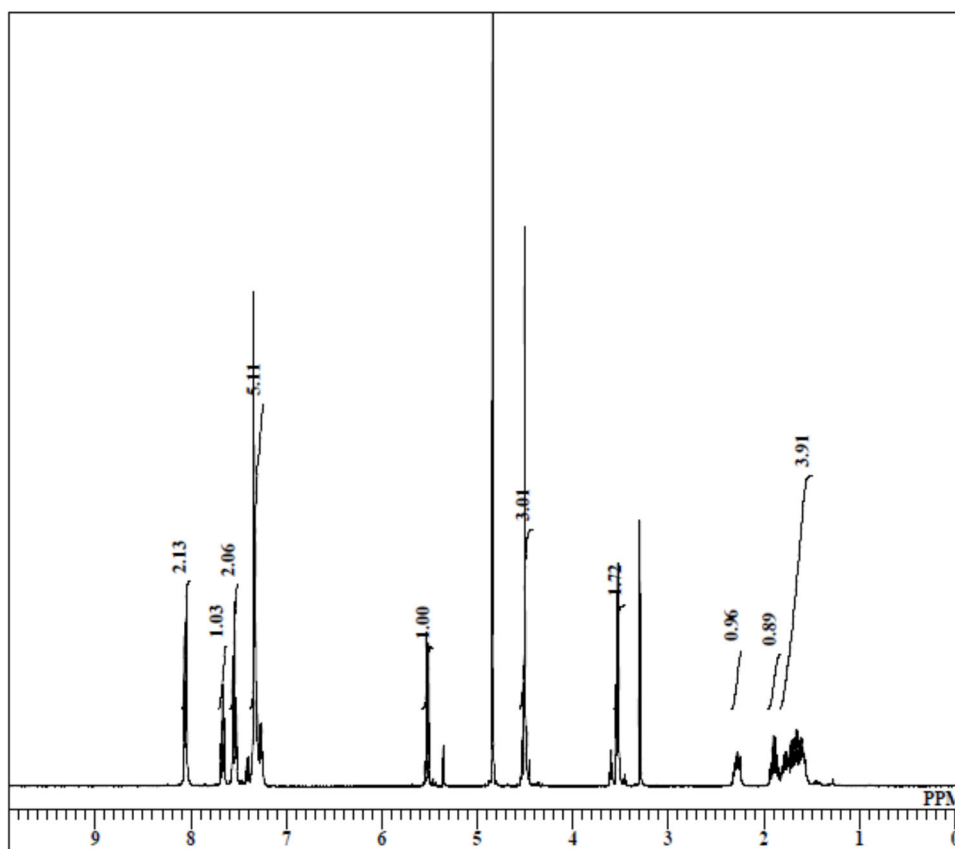
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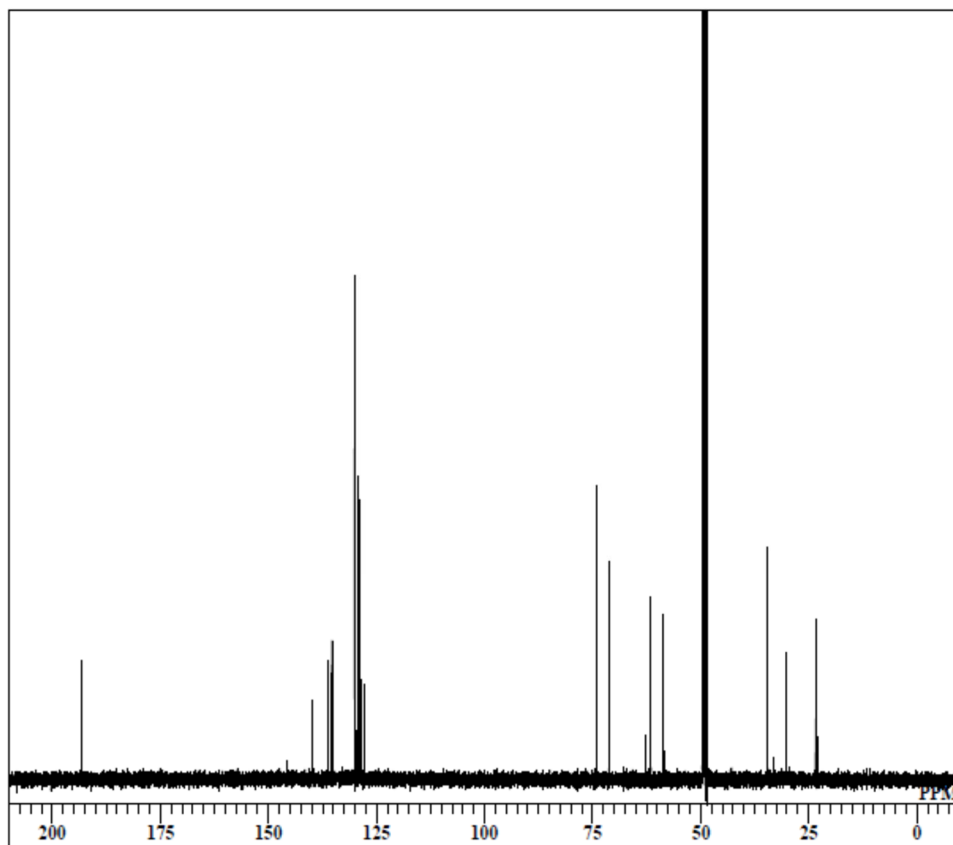
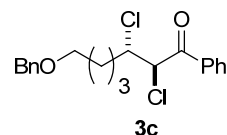
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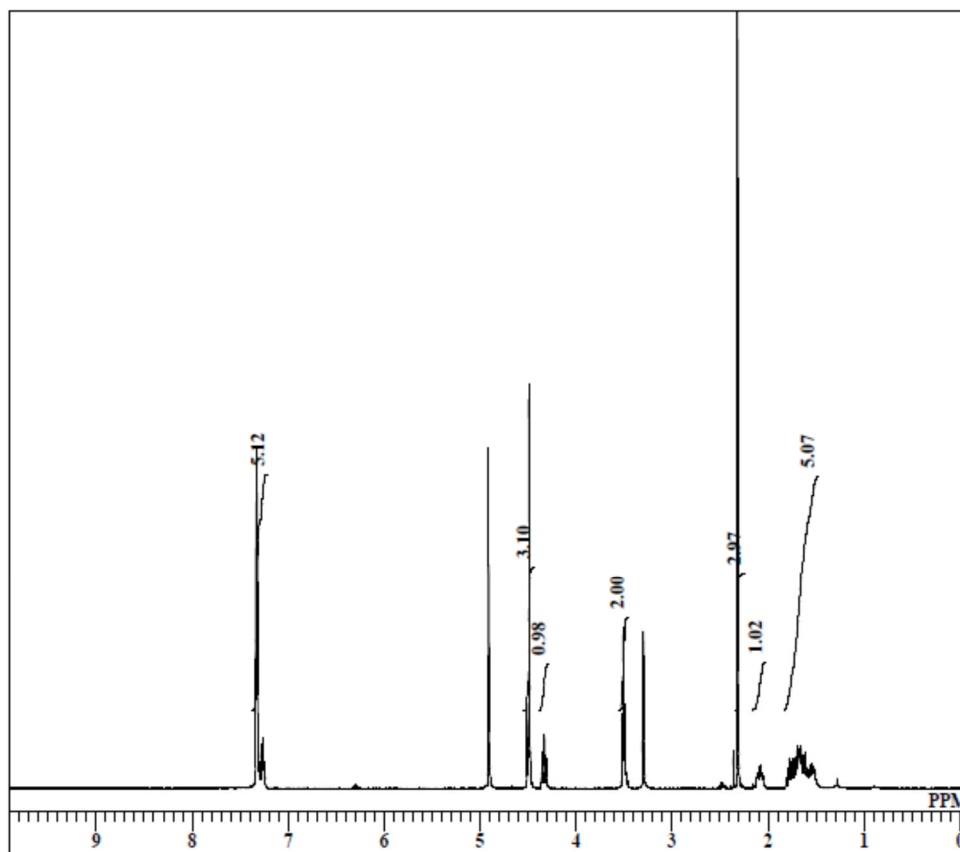
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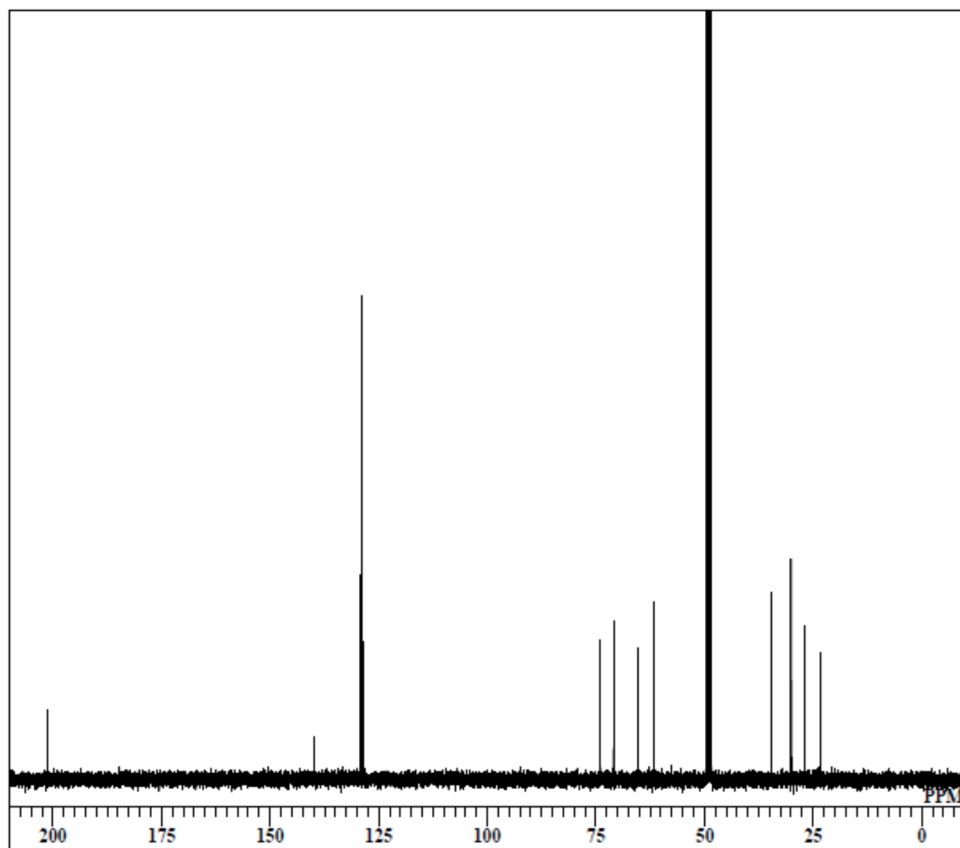
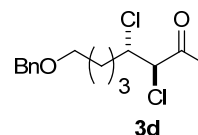
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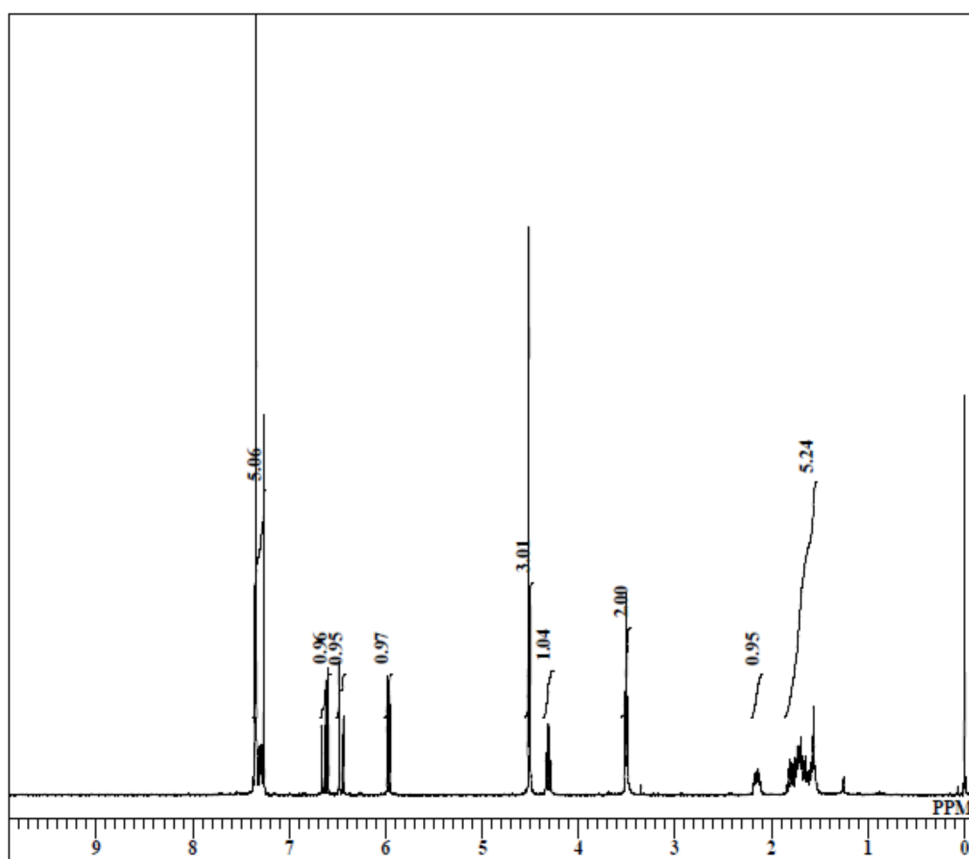
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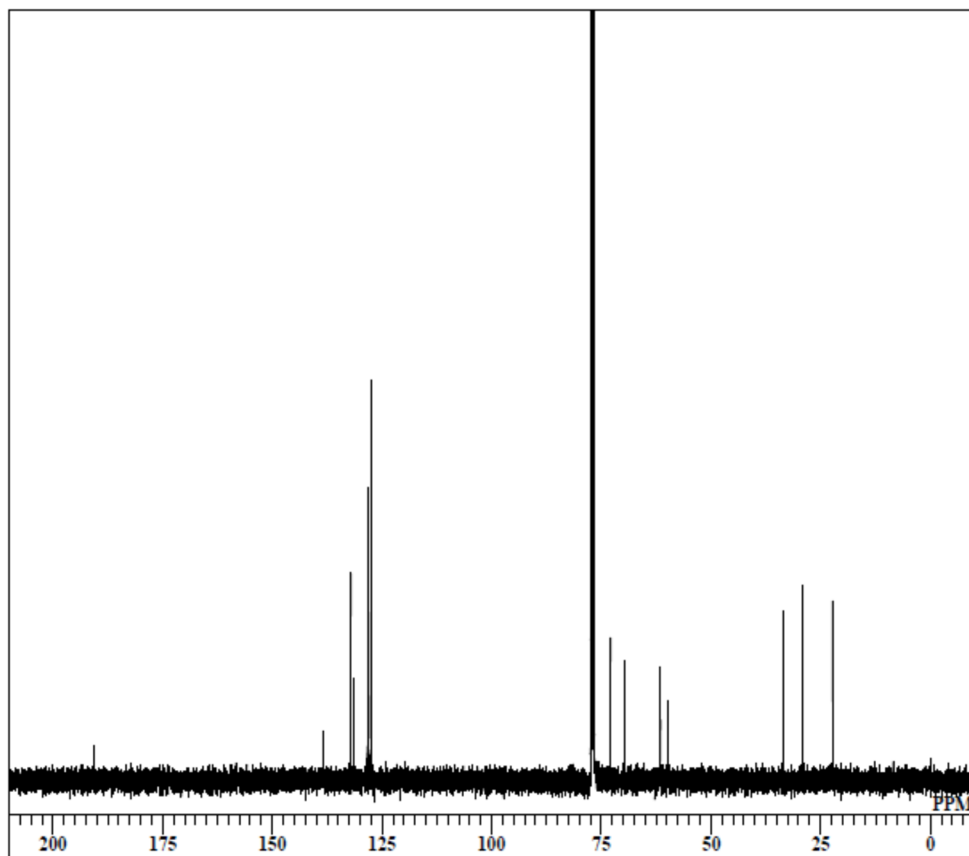
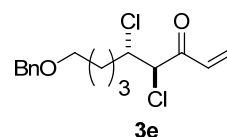
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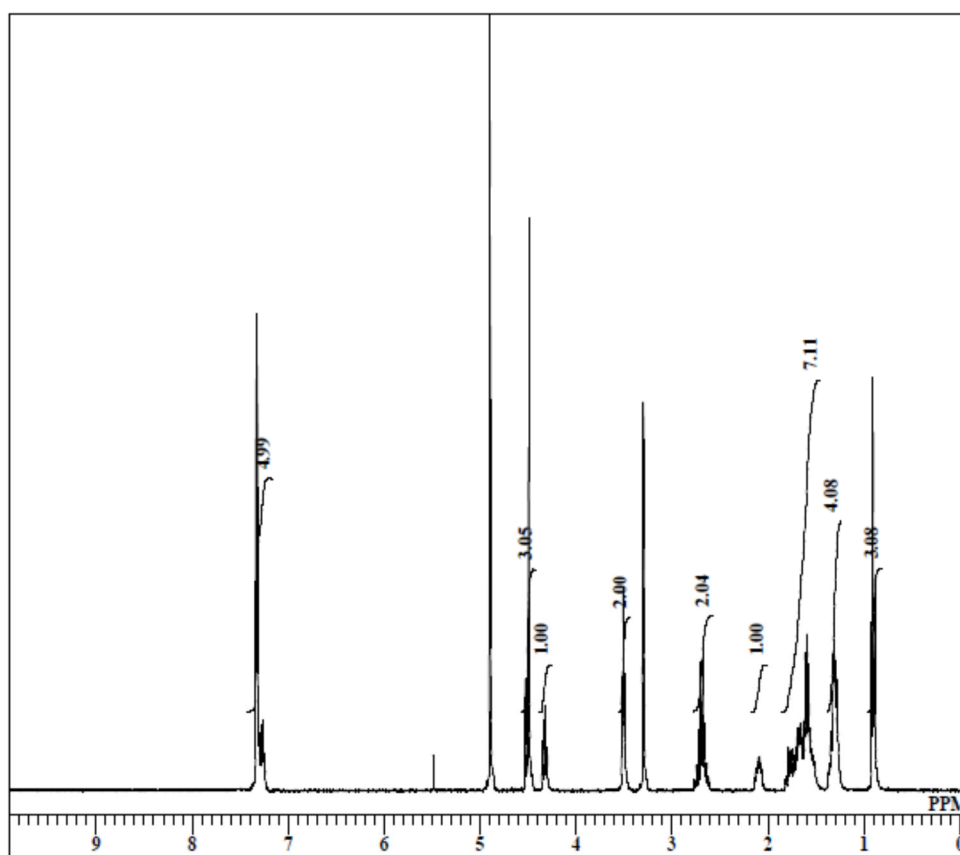
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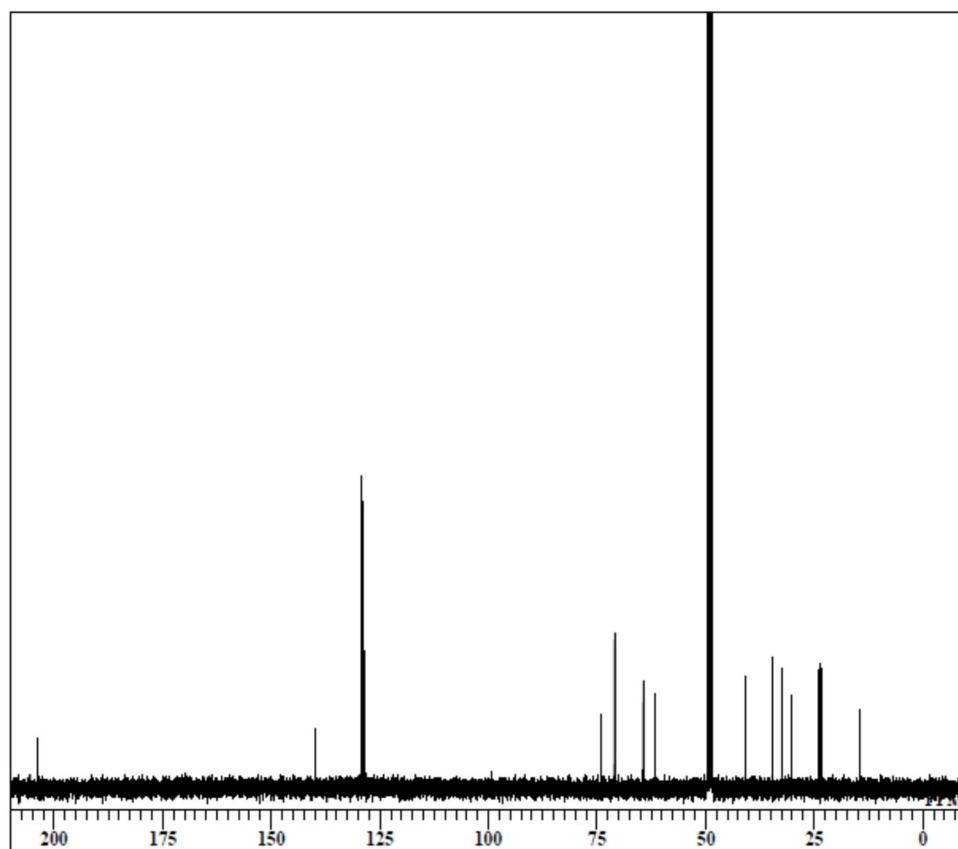
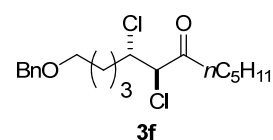
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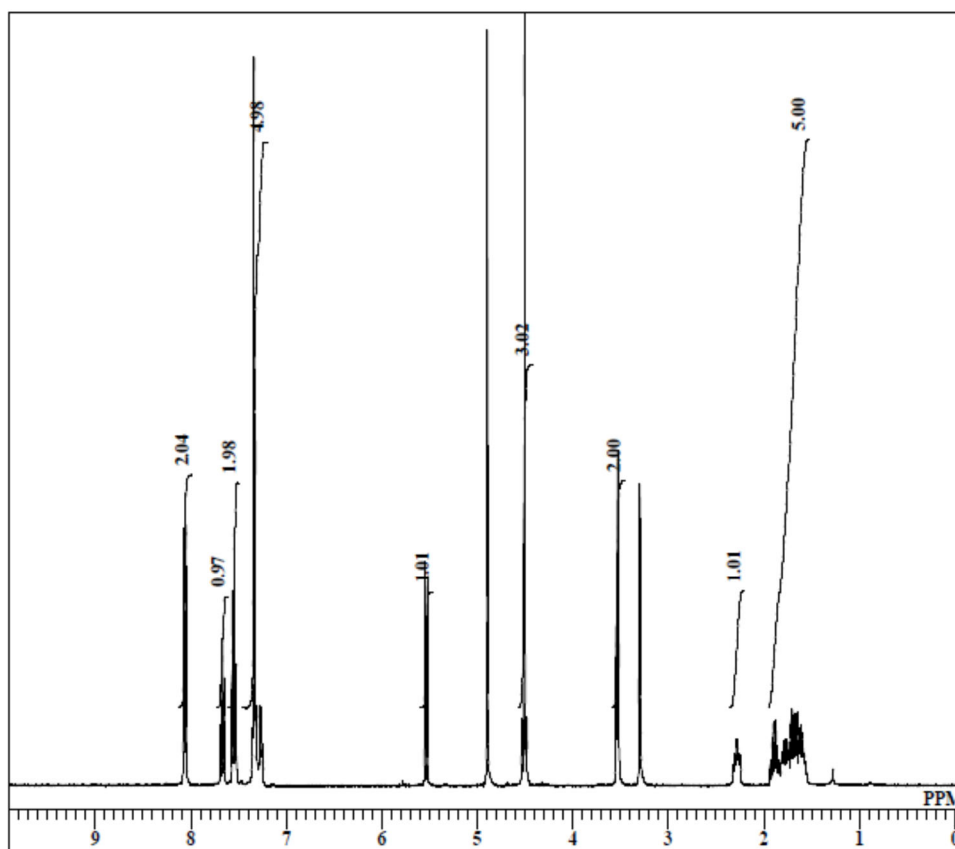
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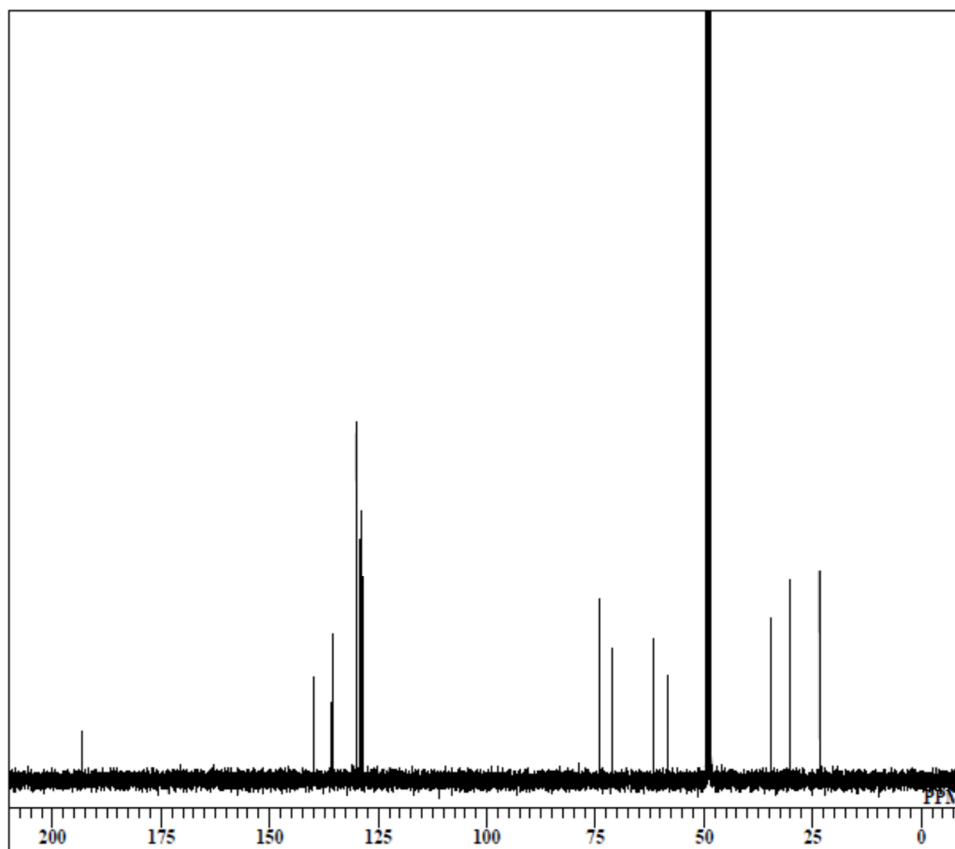
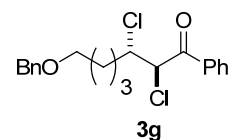
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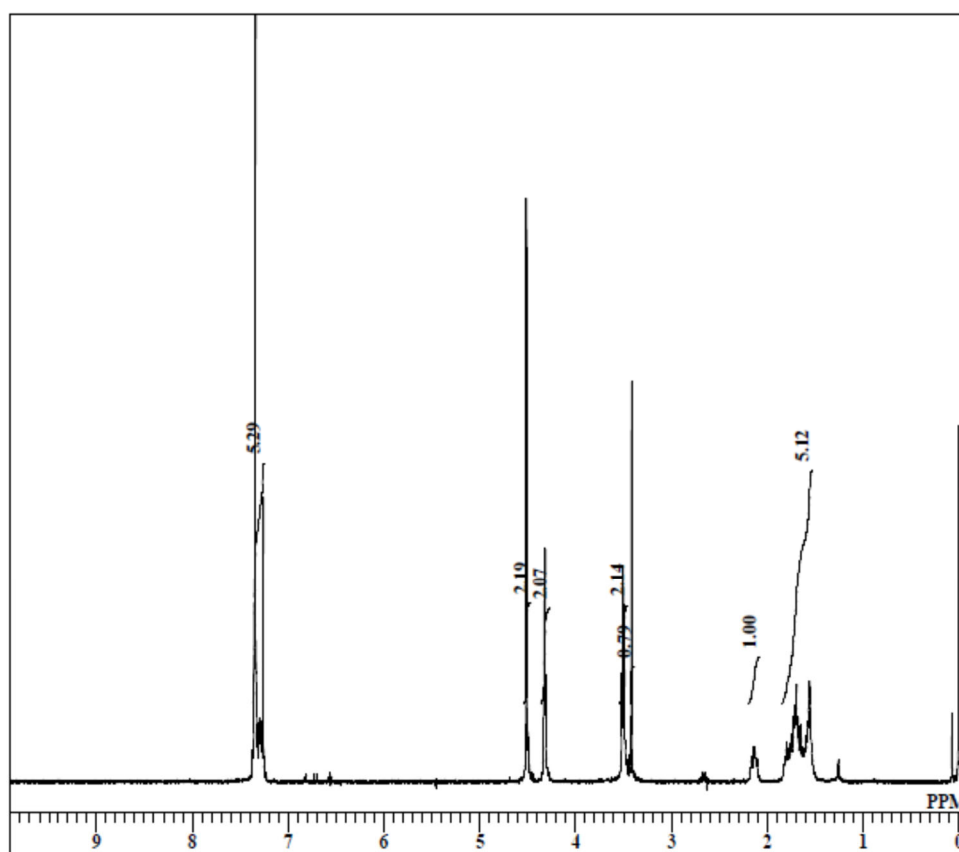
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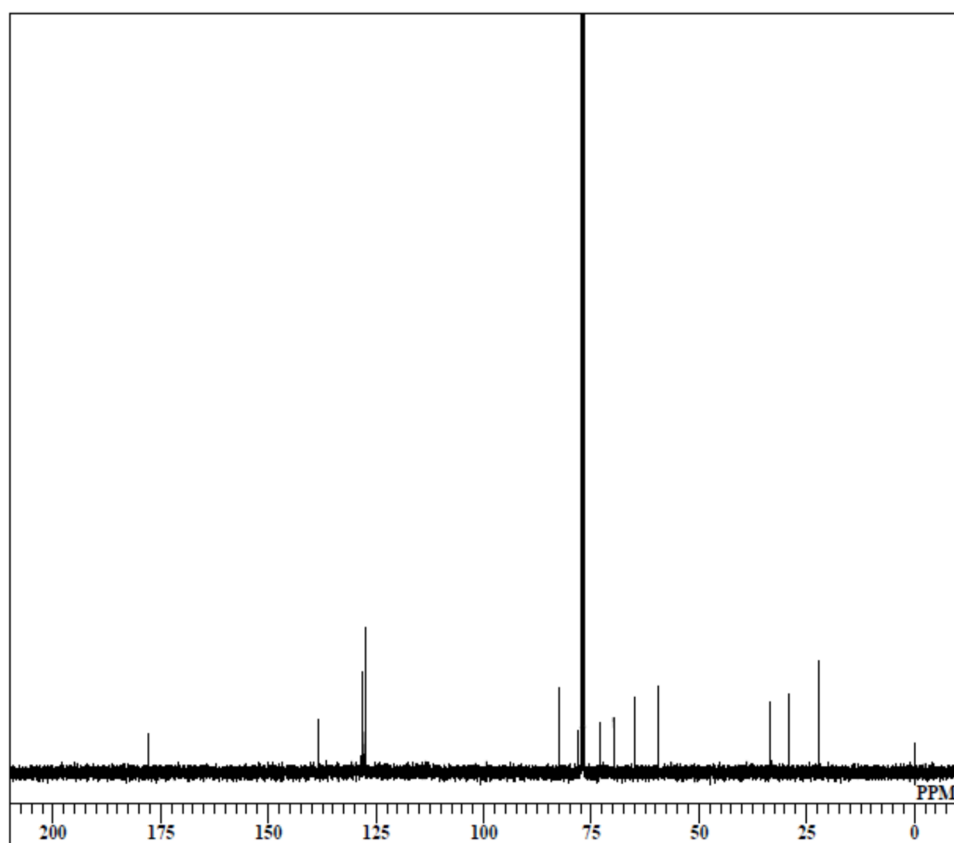
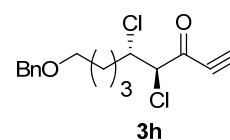
DFILE 20210117 AntiCl-Phenyl-1H.
 COMNT 20210117 AntiCl-Phenyl-1H
 DATIM Sun Jan 17 15:59:52 2021
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 2.0500 sec
 PD 4.9500 sec
 PW1 6.00 usec
 IRNUC 1H
 CTEMP 17.4 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.12 Hz
 RGAIN 21



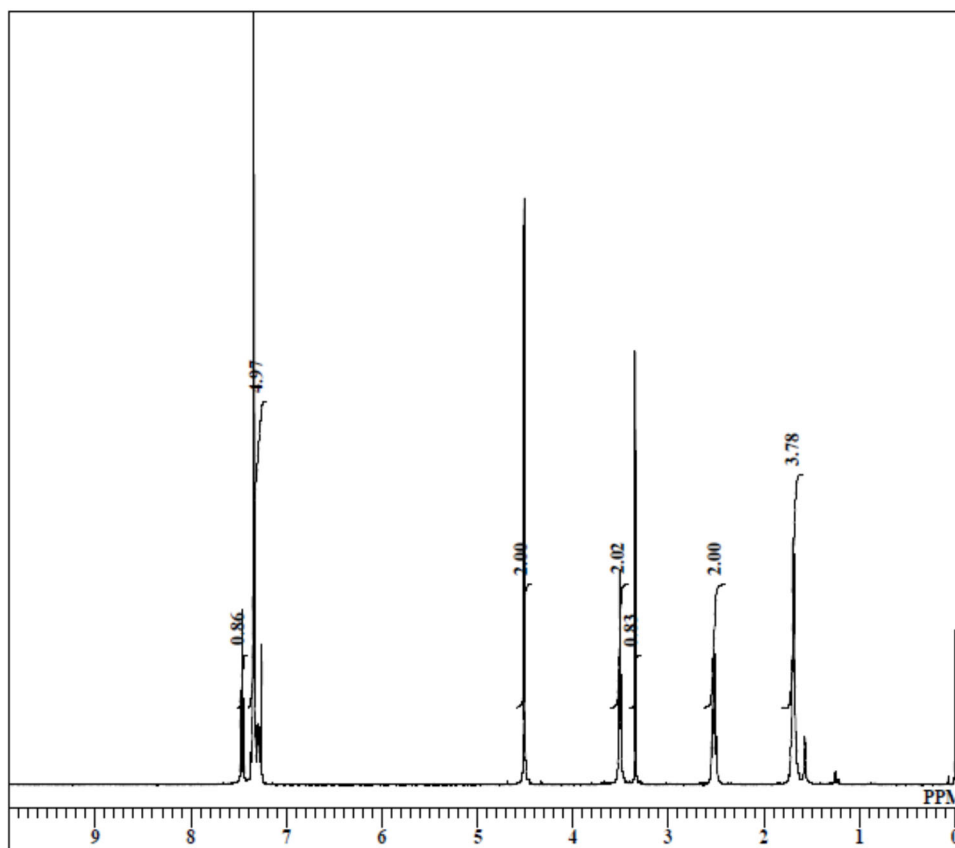
DFILE 20210117 AntiCl-Phenyl-13C
 COMNT 20210117 AntiCl-Phenyl-13C
 DATIM Sun Jan 17 17:21:08 2021
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 1536
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 5.00 usec
 IRNUC 1H
 CTEMP 14.9 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.12 Hz
 RGAIN 28



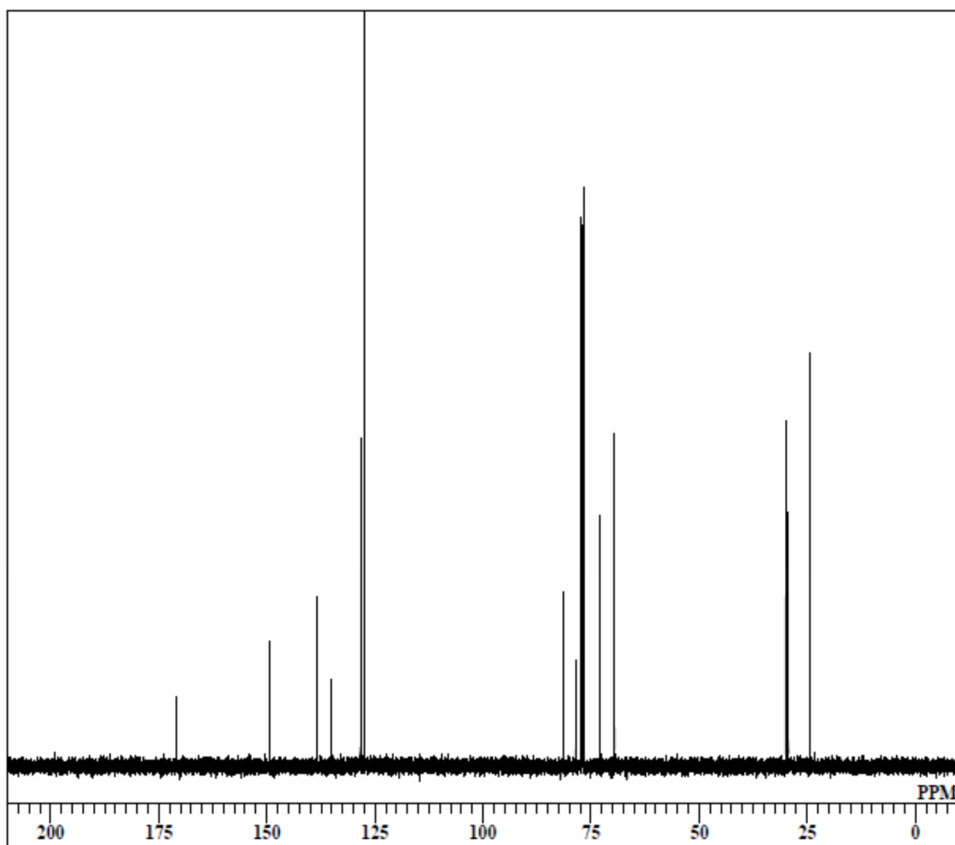
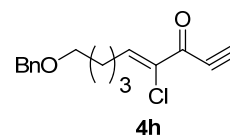
DFILE 20210620 AntiCl-ethynyl 1H
 COMNT 20210620 AntiCl-ethynyl 1H
 DATIM Sun Jun 20 21:10:32 2021
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 4
 ACQTM 2.0500 sec
 PD 4.9500 sec
 PW1 6.00 usec
 IRNUC 1H
 CTEMP 22.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.10 Hz
 RGAIN 24



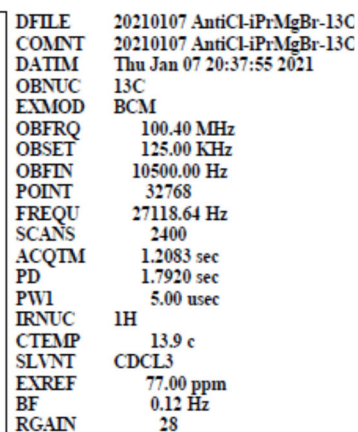
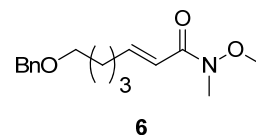
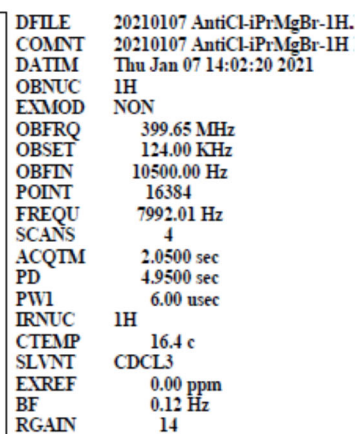
DFILE 20210620 AntiCl-ethynyl-13C
 COMNT 20210620 AntiCl-ethynyl-13C
 DATIM Tue Jun 22 00:22:19 2021
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 5400
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 5.00 usec
 IRNUC 1H
 CTEMP 21.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.10 Hz
 RGAIN 29

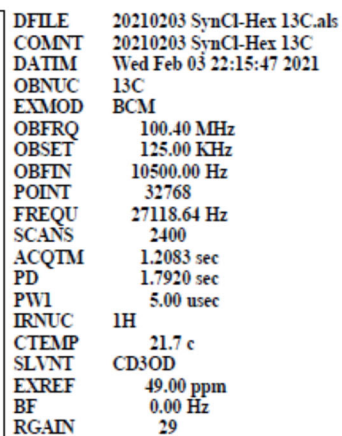
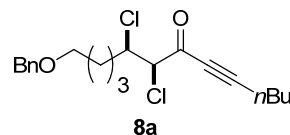
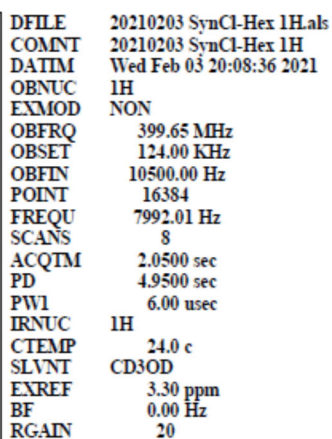


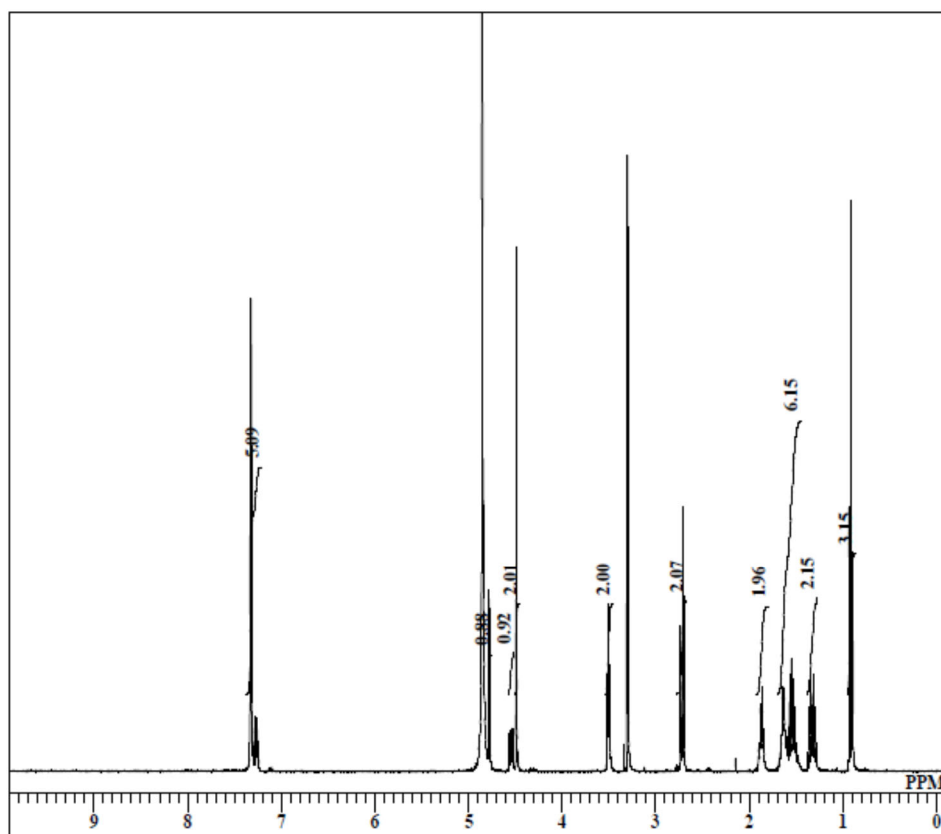
DFILE 20210529 AntiCl-Ethynyl 1H
 COMNT 20210529 AntiCl-Ethynyl 1H
 DATIM Sun May 30 19:16:38 2021
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 2.0500 sec
 PD 4.9500 sec
 PW1 6.00 usec
 IRNUC 1H
 CTEMP 22.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.10 Hz
 RGAIN 21



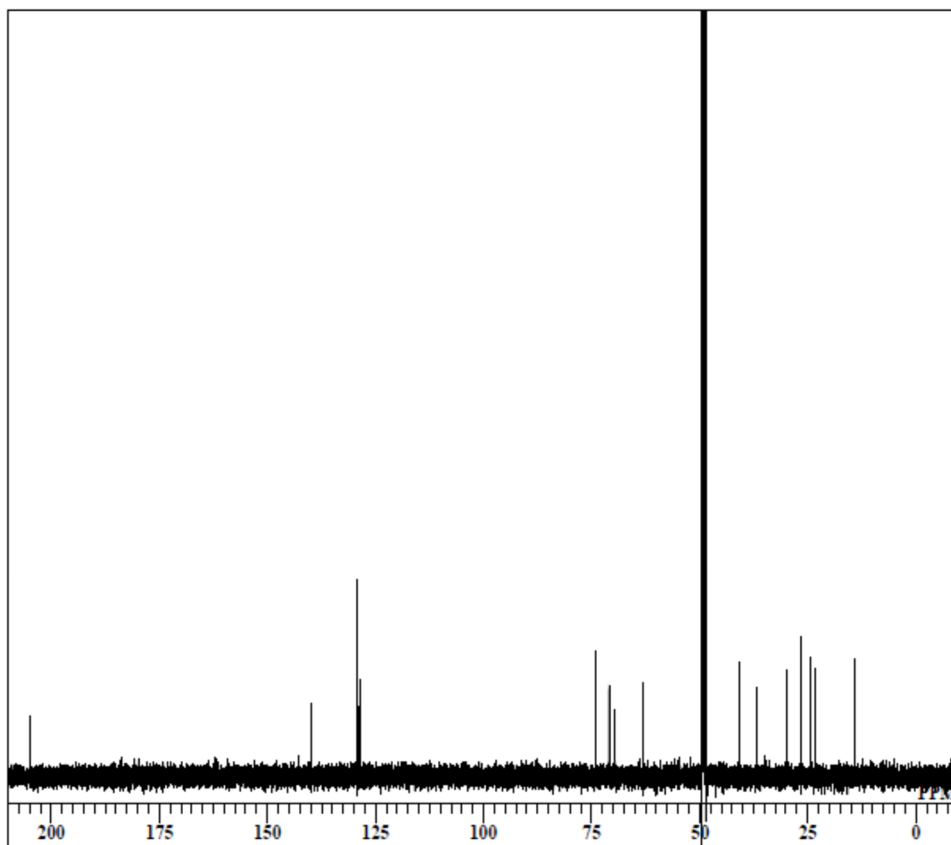
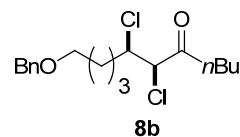
DFILE 20210529 AntiCl-Ethynyl 13 C
 COMNT 20210529 AntiCl-Ethynyl 13C
 DATIM Sat May 29 15:59:43 2021
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 700
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 5.00 usec
 IRNUC 1H
 CTEMP 20.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.10 Hz
 RGAIN 29



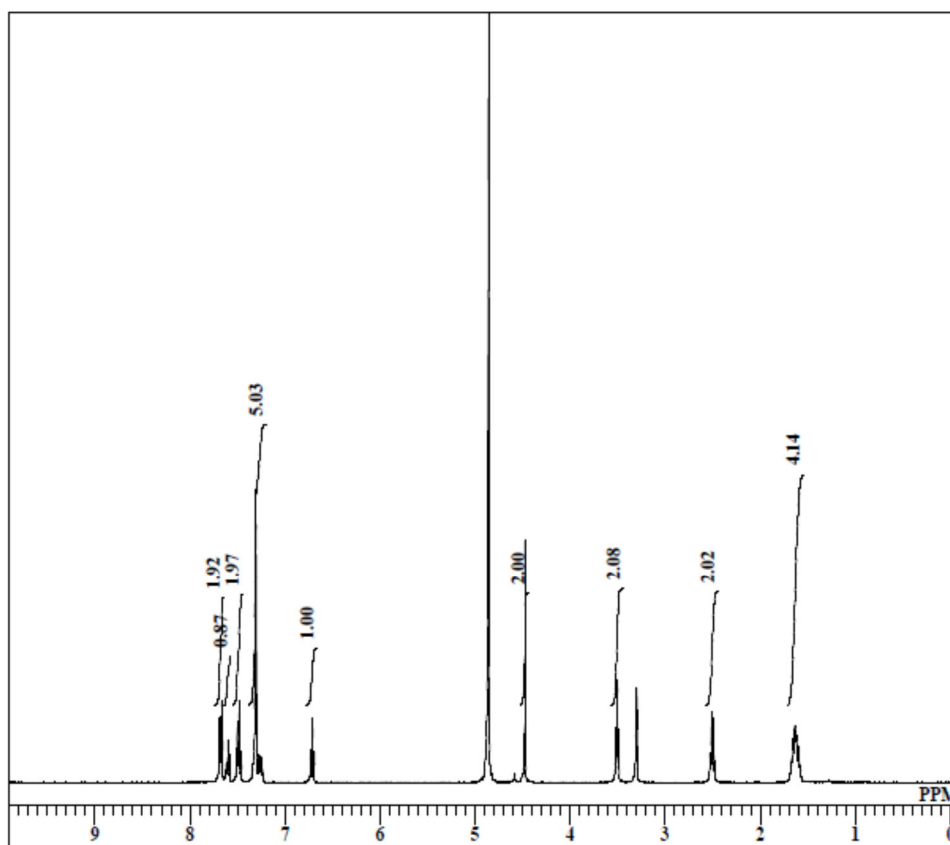




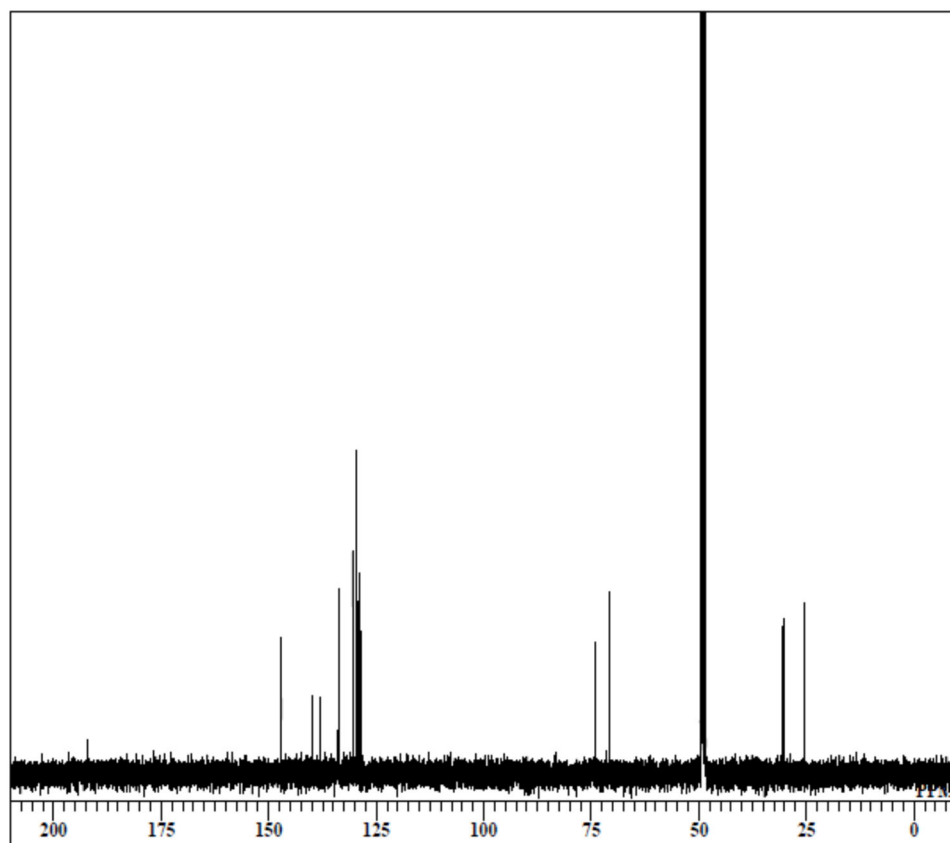
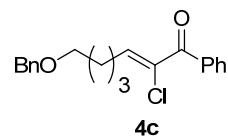
DFILE 20210202 SynCl-nBu1.als
 COMNT 20210202 SynCl-nBu
 DATIM Tue Feb 02 18:15:38 2021
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 2.0500 sec
 PD 4.9500 sec
 PW1 6.00 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.00 Hz
 RGAIN 20



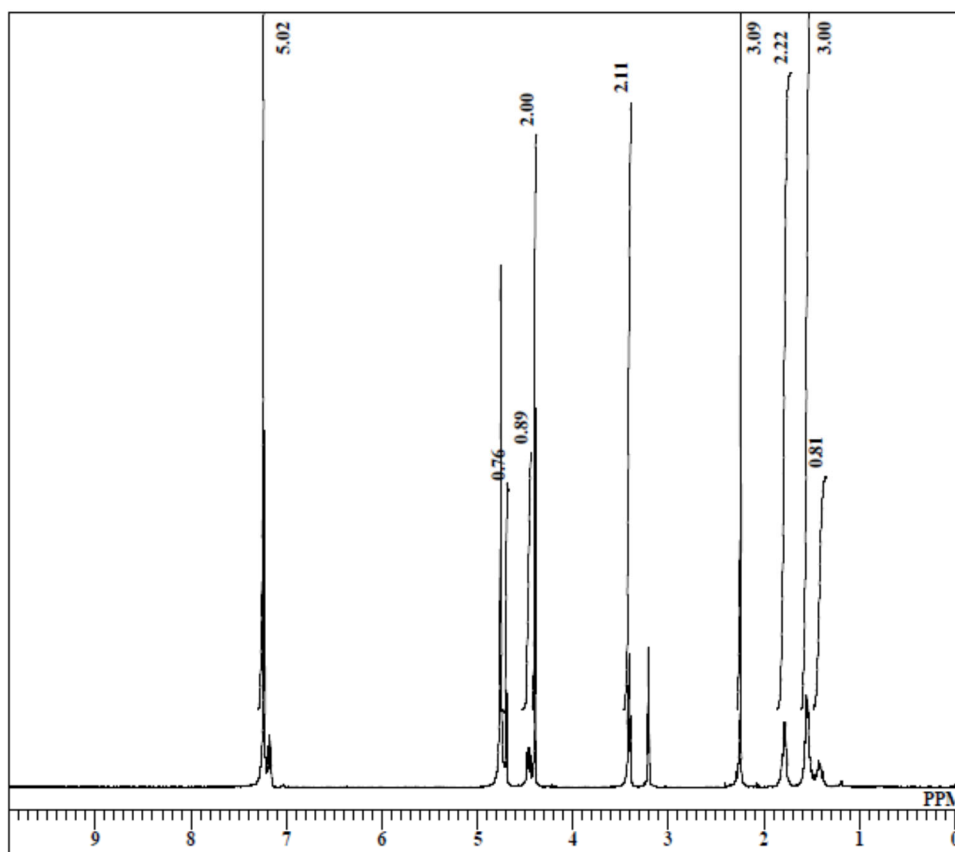
DFILE 20210202 SynCl-nBu13Cl.als
 COMNT 20210202 SynCl-nBu13C
 DATIM Tue Feb 02 20:21:04 2021
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 2400
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 5.00 usec
 IRNUC 1H
 CTEMP 21.6 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.00 Hz
 RGAIN 29



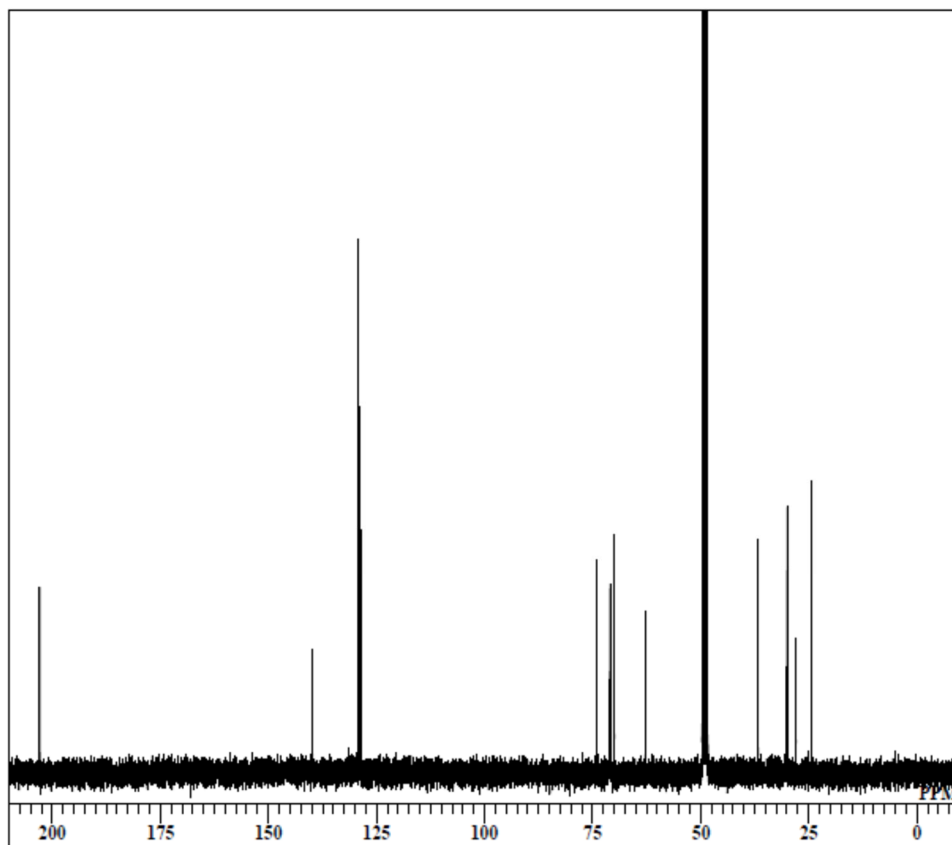
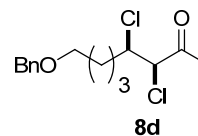
DFILE 20210208 SynCl Ph-Li 1H.als
 COMNT 20210208 SynCl Ph-Li 1H
 DATIM Mon Feb 08 11:54:11 2021
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 2.0500 sec
 PD 4.9500 sec
 PW1 6.00 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.12 Hz
 RGAIN 18



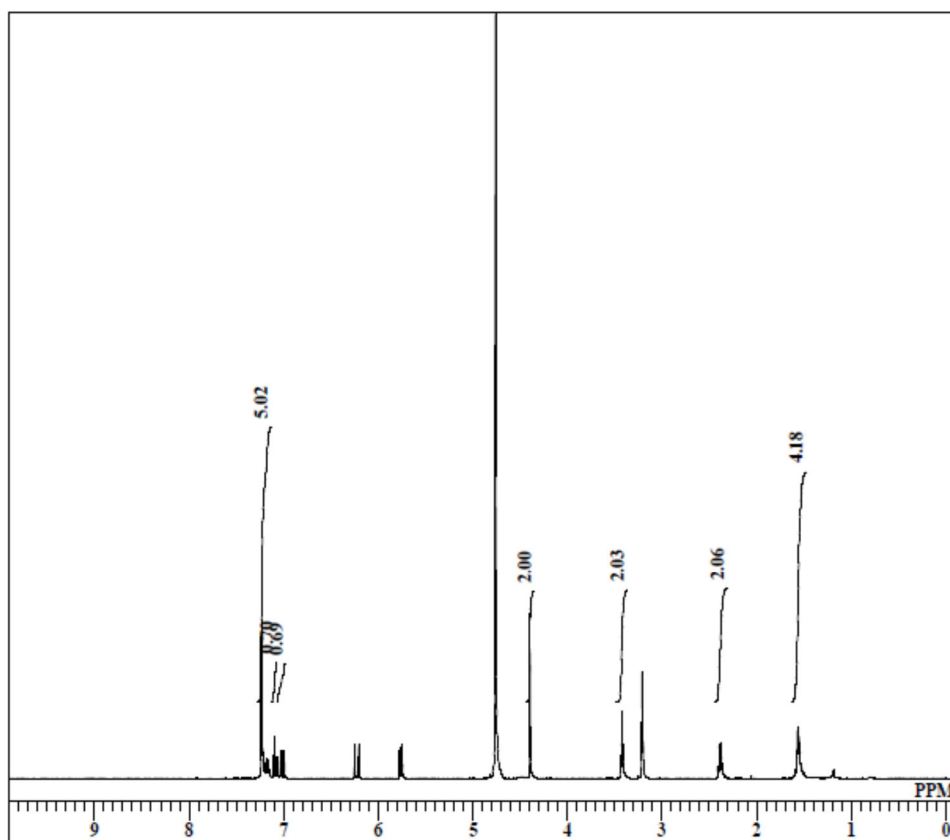
DFILE 20210208 SynCl Ph-Li 13C.als
 COMNT 20210208 SynCl Ph-Li 13C
 DATIM Mon Feb 08 22:52:23 2021
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 2500
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 5.00 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.12 Hz
 RGAIN 29



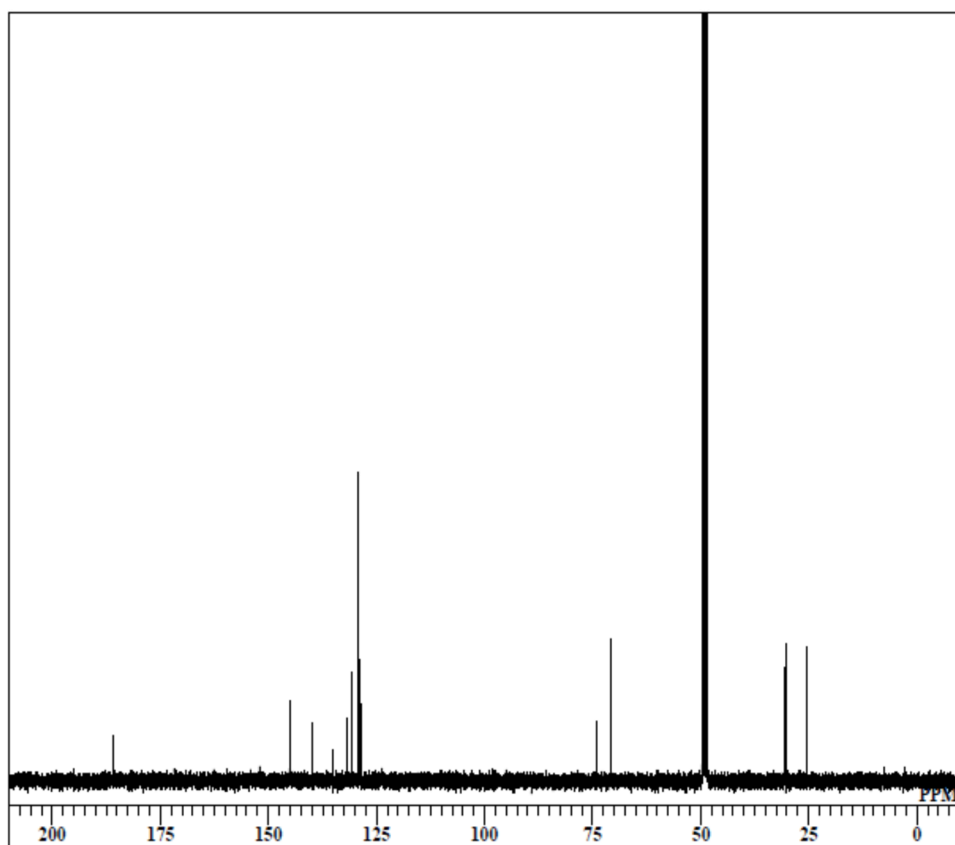
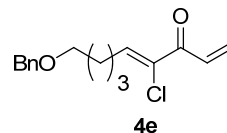
DFILE 20210205 SynCl-Methyl- 1H.
 COMENT 20210205 SynCl-Methyl 1H
 DATIM Fri Feb 05 16:13:44 2021
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 2.0500 sec
 PD 4.9500 sec
 PW1 6.00 usec
 IRNUC 1H
 CTEMP 22.6 c
 SLVNT CD3OD
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 19



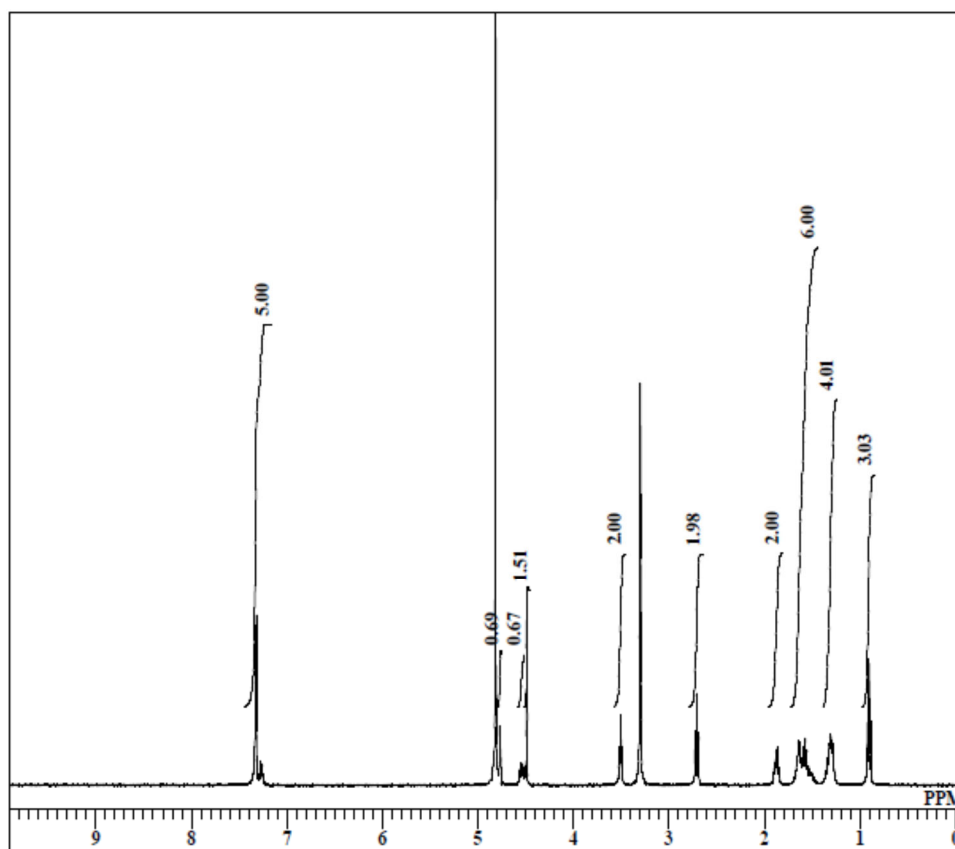
DFILE 20210205 SynCl-Methyl- 13C.
 COMENT 20210205 SynCl-Methyl- 13C
 DATIM Fri Feb 05 19:59:13 2021
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 2400
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 5.00 usec
 IRNUC 1H
 CTEMP 21.9 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.12 Hz
 RGAIN 29



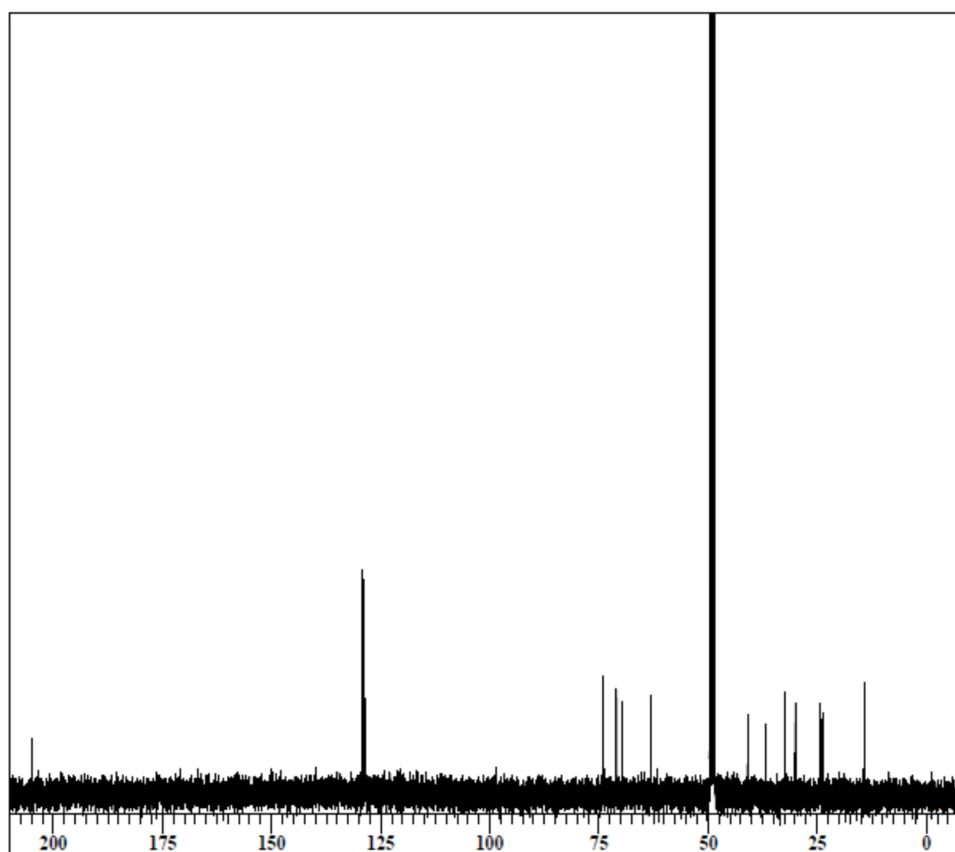
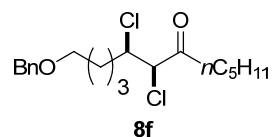
DFILE 20210206 SynCl-VinylMgX 1H
 COMNT 20210208 SynCl-Vinyl-Li 1H
 DATIM Mon Feb 08 11:40:06 2021
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 2.0500 sec
 PD 4.9500 sec
 PW1 6.00 usec
 IRNUC 1H
 CTEMP 22.7 c
 SLVNT CD3OD
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 20



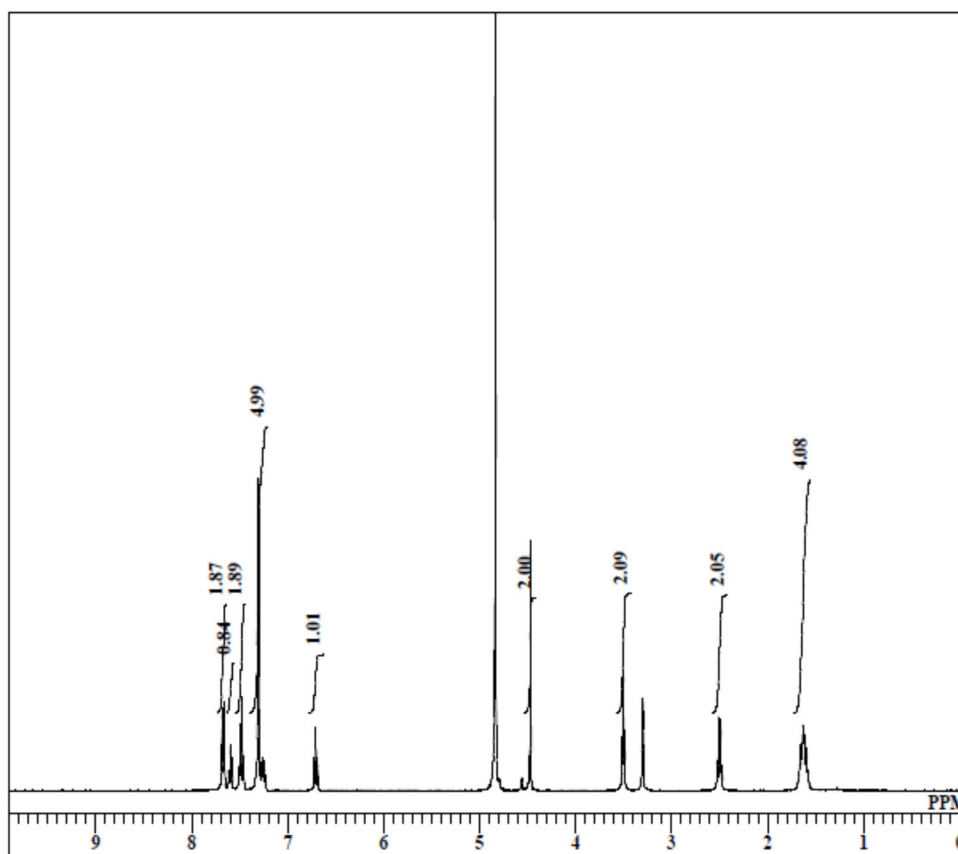
DFILE 20210206 SynCl-VinylMgX 13
 COMNT 20210206 SynCl-VinylMgX 13
 DATIM Mon Feb 08 20:16:10 2021
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 3000
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 5.00 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.12 Hz
 RGAIN 29



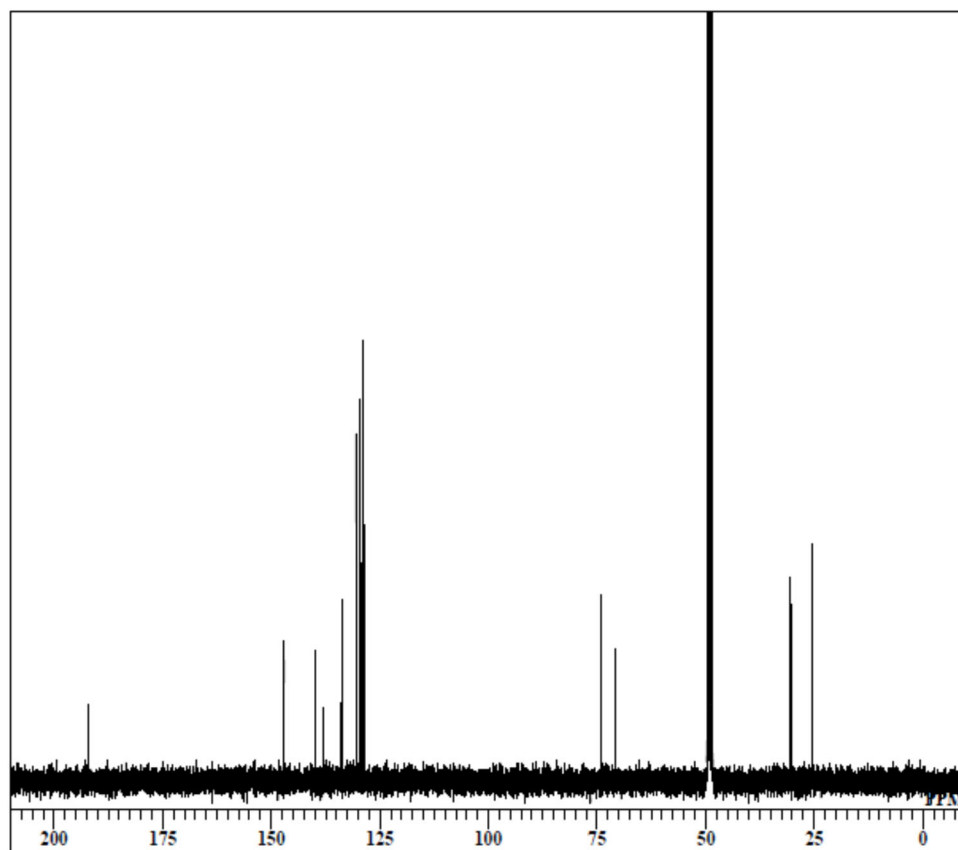
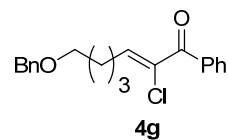
DFILE 20210223 SynCl-PentylMgX
 COMNT 20210223 SynCl-PentylMgX
 DATIM Tue Feb 23 21:10:22 2021
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 2.0500 sec
 PD 4.9500 sec
 PW1 6.00 usec
 IRNUC 1H
 CTEMP 26.2 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.10 Hz
 RGAIN 24



DFILE 20210223 SynCl-PentylMgX
 COMNT 20210223 SynCl-PentylMgX
 DATIM Wed Feb 24 05:37:53 2021
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 10000
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 5.00 usec
 IRNUC 1H
 CTEMP 22.8 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.10 Hz
 RGAIN 29



DFILE 20210207 SynCl PhMgX 1H.
 COMNT 20210207 SynCl-PhMgX 1H
 DATIM Sun Feb 07 19:36:34 2021
 OBNUC 1H
 EXMOD NON
 OBFRQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10500.00 Hz
 POINT 16384
 FREQU 7992.01 Hz
 SCANS 8
 ACQTM 2.0500 sec
 PD 4.9500 sec
 PW1 6.00 usec
 IRNUC 1H
 CTEMP 24.1 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.12 Hz
 RGAIN 18



DFILE 20210207 SynCl PhMgX 13C.
 COMNT 20210207 SynCl-PhMgX 13C
 DATIM Sun Feb 07 21:46:12 2021
 OBNUC 13C
 EXMOD BCM
 OBFRQ 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 POINT 32768
 FREQU 27118.64 Hz
 SCANS 2500
 ACQTM 1.2083 sec
 PD 1.7920 sec
 PW1 5.00 usec
 IRNUC 1H
 CTEMP 22.4 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.12 Hz
 RGAIN 29