Supplementary Material (ESI) for New Journal of Chemistry

Supporting Information

Gold-Catalyzed Cyclizations of Alkyne-Propargylic Acetates to 2,3-Bisalkyledenecycloakanones and Their Related Benzene Derivatives

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Characterization of new compounds **2a-d, 4a-f,** and **5h-i** are therein.

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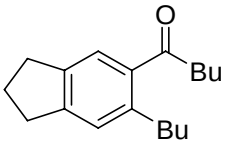
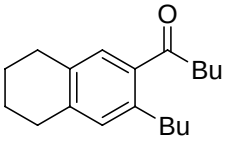
1. General experimental procedure :

To a mixture of $AuCl(PPh_3)$ (80.0 mg, 0.16 mmol) and AgSbF6 (54.0 mg, 0,16 mmol) in dry acetonitrile (0.5 mL) was added a solution of yne-propargylic acetate **1** (100.0 mg, 3.2 mmol) in dry dichloromethane (0.5 mL) at 0℃ under argon atmosphere. The resulting mixture was stirred for 5~10h at 25~80 ℃. Upon completion of the reaction, the solvent was removed under vacuum and the crude product was subjected for flash column chromatography(SiO_2, *n*-hexane/EtOAc = 10:1) to afford the pure products.

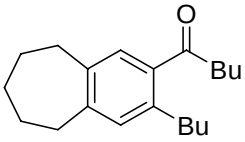
2. Spectroscopic data of compounds



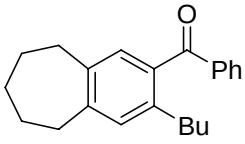
Spectroscopic data of compound 2a: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (s, 1H), 7.11 (s, 1H), 2.90 (t, *J* = 7.2 Hz, 4H), 2.85 (t, *J* =7.8 Hz, 2H), 2.74(t, *J* = 7.6 Hz, 2H), 2.09 (quint, *J* = 7.2 Hz, 2H), 1.68 (quint, *J* = 7.2 Hz, 2H), 1,52 (m, 2H), 1.38 (m ,4H), 0.94 (t, *J* = 7.2 Hz, 3H), 0.92 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.79, 147.74, 141.63, 140.93, 137.20, 127.06, 124.18, 42.11, 34.67, 33.82, 33.10, 32.61, 26.91, 25.62, 23.05, 22.71, 14.23, 14.18; FTIR (NaCl, cm^{-1}) 1684, 1559, 1541, 1507; HRMS (ES) calcd for $C_{18}H_{26}O\ Na^+$ ($M+Na^+$) 281.1876, found 281.1883.



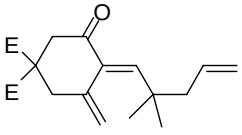
Spectroscopic data of compound 2b: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (s, 1H), 6.93 (s, 1H), 2.85 (t, *J* = 7.2 Hz, 2H), 2.80-2.70 (m ,6H), 1.82-1.78 (m, 4H), 1.72-1.64 (m, 2H), 1.56-1.48 (m, 2H), 1.44-1.34 (m, 4H), 0.94 (t, *J* = 7.6 Hz, 3H), 0.91 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.17, 140.70, 139.90, 136.16, 134.46, 131.89, 129.49, 41.78, 34.52, 29.55, 29.19, 26.90, 23.41, 23.23, 23.10, 22.71, 14.25, 14.21; FTIR (NaCl, cm^{-1}) 1684, 1559, 1458; HRMS (ES) calcd for $C_{19}H_{28}O\ Na^+$ ($M+Na^+$) 295.2132, found 295.2033.



Spectroscopic data of compound 2c: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (s, 1H) 6.97 (s, 1H), 2.86 (t, *J* = 7.2 Hz, 2H), 2.82-2.71 (m, 5H), 1.89-1.81 (m, 2H), 1.72-1.61 (m, 6H), 1.57-1.49 (m, 3H), 1.44-1.34 (m, 5H), 0.95 (t, *J* = 7.2 Hz, 3H), 0.92 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.23, 146.89, 140.71, 140.64, 136.28, 132.11, 129.27, 41.82, 36.83, 36.55, 34.46, 33.50, 32.85, 29.93, 28.52, 28.33, 26.85, 23.12, 22.71, 14.24, 14.20; FTIR (NaCl, cm^{-1}) 1683, 1609, 1557; HRMS (ES) calcd for $C_{20}H_{30}ONa^+$ ($M+Na^+$) 309.2189; found 309.2194.

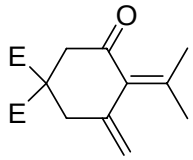


Spectroscopic data of compound 2d: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.79 (m, 2H), 7.56 (m, 1H), 7.46-7.42 (m, 2H), 7.03 (s, 1H), 7.01 (s, 1H), 2.81 (dd, *J* = 7.0, 2.0 Hz, 2H), 2.74 (dd, *J* = 7.2, 2.0 Hz, 2H), 2.59 (t, *J* = 8.0 Hz, 2H), 1.84 (m, 2H), 1.70-1.60 (m, 4H), 1.53-1.45 (m, 2H), 1.29-1.22 (m, 2H), 0.82 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.01, 145.89, 140.12, 139.72, 138.38, 135.86, 132.78, 130.93, 130.11, 129.40, 128.27, 36.67, 36.10, 34.02, 32.61, 28.24, 28.15, 22.61, 13.82; FTIR (NaCl, cm^{-1}) 1663, 1596; HRMS (ES) calcd for $C_{22}H_{26}O\ Na^+$ ($M+Na^+$) 329.1876, found 329.1886.

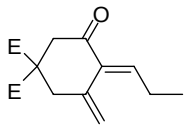


Spectroscopic data of compound 4a: ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.99 (s, 1H), 5.81-5.70 (m, 1H), 5.03-4.98 (m, 3H), 4.81 (q, *J* = 1.2Hz,

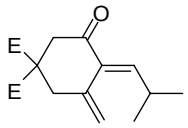
1H), 4.25-4.13 (m, 4H), 2.98 (s, 2H), 2.87 (s, 2H), 2.28 (d, *J* = 7.2 Hz, 2H), 1.14(s, 6H), 1.23(t, J=6.8, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.78, 170.06, 149.44, 144.193, 138.61, 135.65, 117.48, 112.86, 62.19, 55.20, 47.52, 46.98, 38.91, 36.85, 27.73, 14.23; FTIR (NaCl, cm^{-1}) 1745, 1670; HRMS (ES) calcd for $C_{20}H_{28}O_5Na^+$ (M+Na$^+$) 371.1834, found 371.1830.



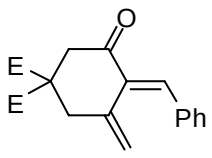
Spectroscopic data of compound 4b: ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.21 (s, 1H), 4.82 (s, 1H), 4.18 (q, *J* = 7.2 Hz, 4H), 3.00 (s, 1H), 2.83 (s, 1H), 2.07 (s, 2H), 1.97 (s, 2H), 1.23 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.06, 170.30, 146.64, 139.74, 135.01, 117.80, 62.11, 55.10, 46.53, 39.94, 24.34, 22.90, 14.25; FTIR (NaCl, cm^{-1}) 1750, 1686; HRMS (ES) calcd for $C_{16}H_{22}O_5Na^+$ (M+Na$^+$) 317.1365, found 317.1361.



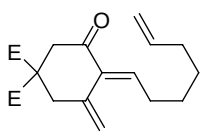
Spectroscopic data of compound 4c: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.73 (t, *J* = 7.6 Hz, 1H), 5.23 (d, *J* = 1.2 Hz, 1H), 4.95 (s, 1H), 4.19 (q, *J* = 7.2 Hz, 4H), 2.96 (s, 2H), 2.85 (s, 1H), 2.35 (m, 2H), 1.23 (t, *J* = 7.2 Hz, 6H), 1.08 (t, *J* = 7.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.67, 169.92, 144.16, 137.16, 136.13, 117.39, 61.91, 54.47, 44.36, 39.70, 22.89, 14.00, 13.73; FTIR (NaCl, cm^{-1}) 1740, 1692; HRMS (ES) calcd for $C_{16}H_{22}O_5Na^+$ (M+Na$^+$) 317.1365, found 317.1369.



Spectroscopic data of compound 4d : ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.50 (t, *J* = 7.6 Hz, 1H), 5.18 (d, *J* = 1.2 Hz, 1H), 4.97 (s, 1H), 4.19 (q, *J* = 7.2 Hz, 4H), 2.97 (s, 2H), 2.86 (m, 1H), 2.85 (s, 2H), 1.23(t, *J* = 7.2 Hz, 6H), 1.04(d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.32, 170.16, 148.63, 137.64, 135.13, 116.98, 62.16, 54.72, 44.68, 40.02, 27.93, 22.73, 14.25 FTIR (NaCl, cm^{-1}) 1755, 1685; HRMS (ES) calcd for $C_{17}H_{24}O_5Na^+$ (M+Na$^+$) 331.1521, found 331.1527.

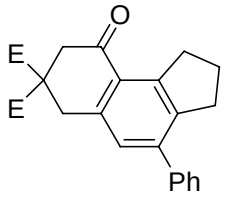


Spectroscopic data of compound 4e : ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.54(m, 2H), 7.45 (s, 1H), 7.32-7.27 (m, 3H), 5.14(s, 1H), 5.08 (d, *J* = 1.2 Hz, 1H), 4.20 (q, J=7.6, 4H), 3.12 (s, 1H), 2.95 (s, 1H), 1.23 (t, J = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.46, 169.87, 136.81, 136.36, 134.82, 129.59, 128.85, 128.42, 119.00, 62.02, 54.38, 44.71, 39.34, 13.99; FTIR (NaCl, cm^{-1}) 1745, 1680, 1591; HRMS (ES) calcd for $C_{20}H_{22}O_5Na^+$ (M+Na$^+$) 365.1365, found 365.1361.

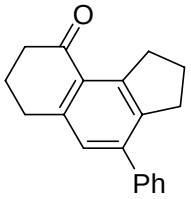


Spectroscopic data of compound 4f : ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.74 (t, J=7.6, 1H), 5.83-5.73(m, 1H), 5.23 (d, *J* = 1.2 Hz, 1H), 5.02-4.92(m, 3H), 4.19 (q, J = 7.2 Hz, 4H), 4.18 (q, J = 7.2 Hz, 4H), 2.96 (s, 1H), 2.85 (s, 1H),

2.34 (q, J = 7.2 Hz, 2H), 2.07-2.01 (m, 2H), 1.52-1.37 (m, 4H), 1.23 (t, *J* = 7.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.60, 169.90, 142.64, 138.49, 137.17, 117.46, 114.63, 61.91, 54.46, 44.35, 39.70, 33.43, 29.67, 29.28, 28.61, 28.54, 13.99; FTIR (NaCl, cm^{-1}) 1748, 1679; HRMS (ES) calcd for $C_{20}H_{28}O_5Na^+$ ($M+Na^+$) 371.1834, found 371.1840.

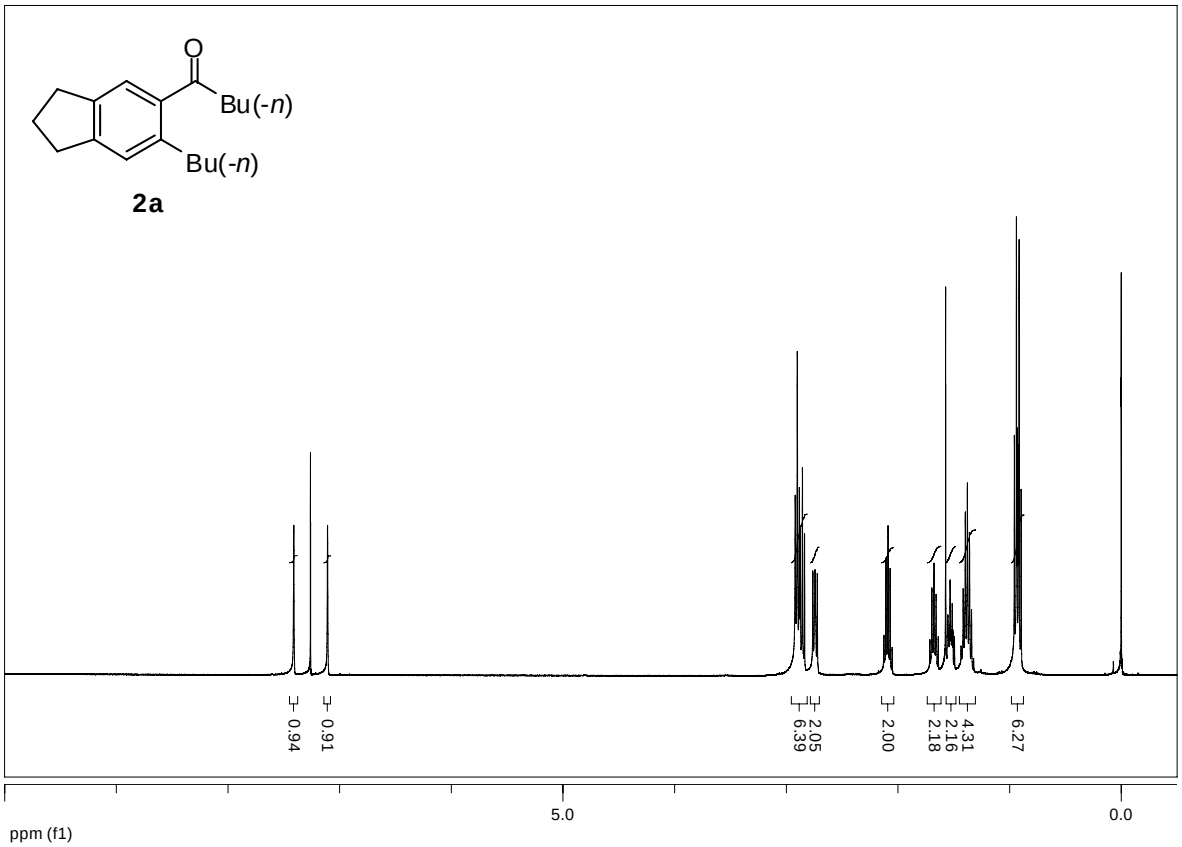


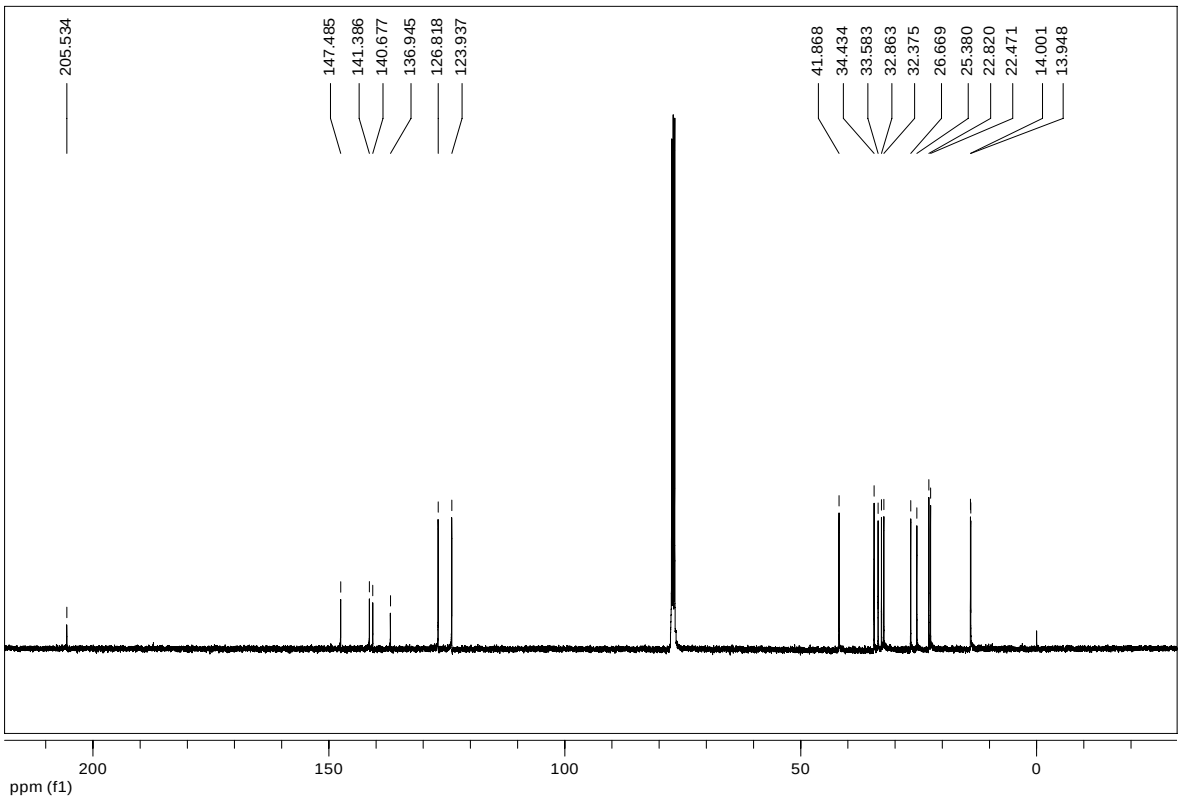
Spectroscopic data of compound 5h: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.41 (m, 3H), 7.39-7.35(m, 2H), 7.11 (s, 1H), 4.17 (q, J = 6.8 Hz, 4H), 3.53 (s, 2H), 3.38 (t, J = 7.6 Hz, 2H), 3.10 (s, 1H), 2.88 (t, J = 7,2 Hz, 2H), 2.04 (m, 2H), 1.19 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.52, 170.68, 148.50, 143.87, 143.49, 140.92, 138.75, 129.05, 128.39, 128.27, 127.22, 62.63, 55.97, 45.27, 36.13, 35.19, 32.64, 26.20, 14.62; FTIR (NaCl, cm^{-1}) 1750, 1729, 1682, 1593; HRMS (ES) calcd for $C_{25}H_{26}O_5Na^+$ ($M+Na^+$) 429.1678, found 429.1680

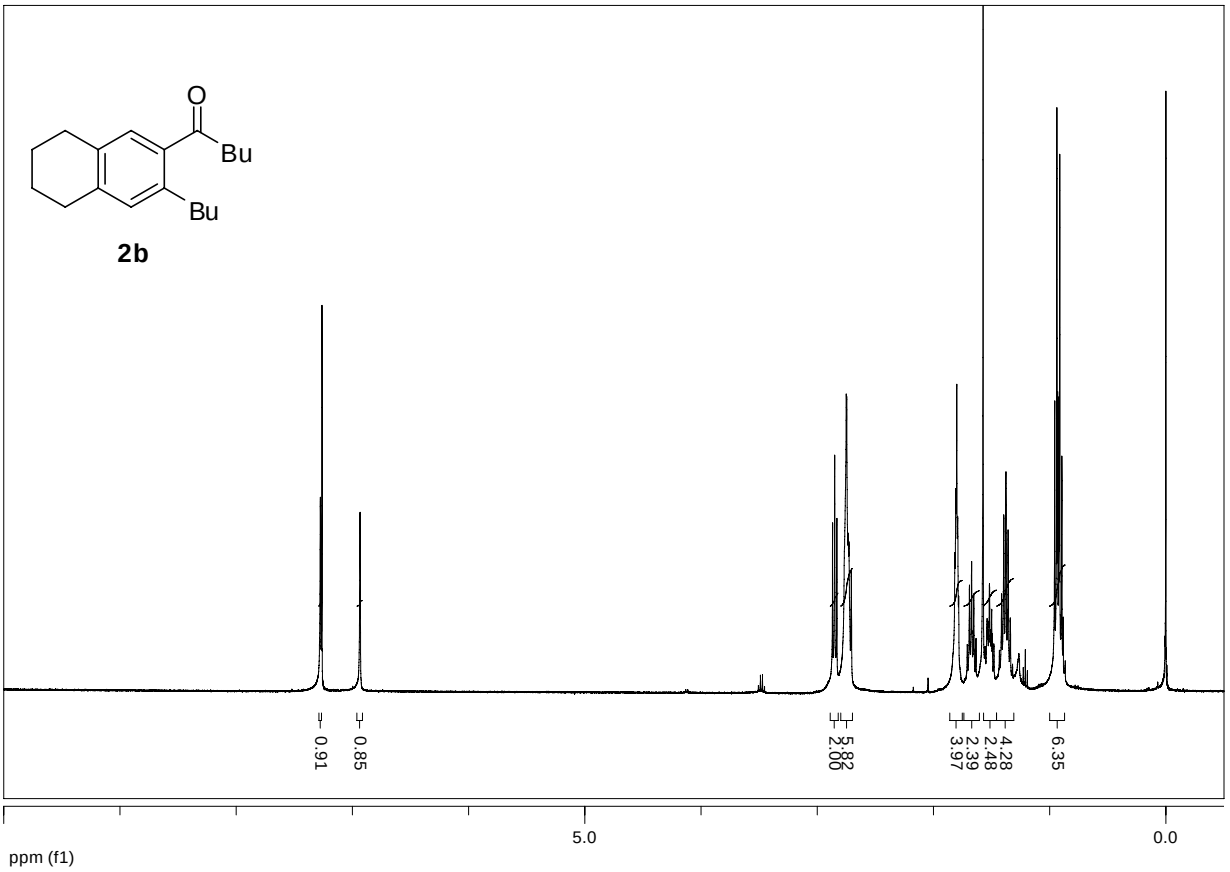


Spectroscopic data of compound 5i: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (m, 4H), 7.40-7.34 (m, 1H), 7.10 (s, 1H), 3.39 (t, *J* = 6.8 Hz, 2H), 2.99 (t, *J* = 6 Hz, 2H), 2.89 (t, *J* = 7.6 Hz, 2H), 2.66 (t, *J* = 6.4 Hz, 2H), 2.13 (q, *J* = 6.4 Hz, 2H), 2.04 (q, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.53, 147.64, 143.34, 142.51, 141.80, 140.51, 128.36, 128.29, 127.51, 127.32, 40.46, 34.70, 31.89, 30.22, 25.64, 23.36; FTIR (NaCl, cm^{-1}) 1678, 1593; HRMS (ES) calcd for $C_{19}H_{18}ONa^+$ ($M+Na^+$) 285.1255, found 285.1260

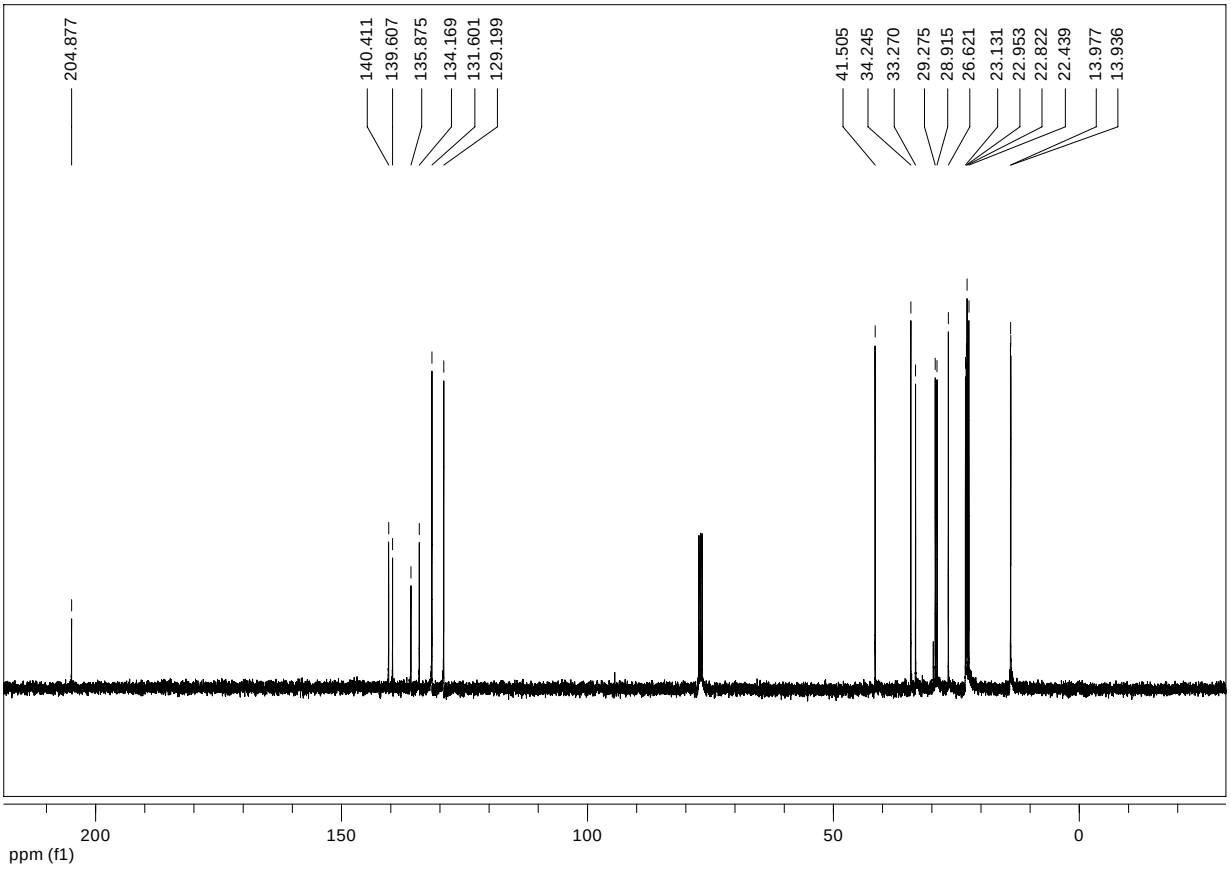
3. ^{1}H and ^{13}C NMR Spectra of Compounds **2a-d, 4a-f,** and **5h-i**

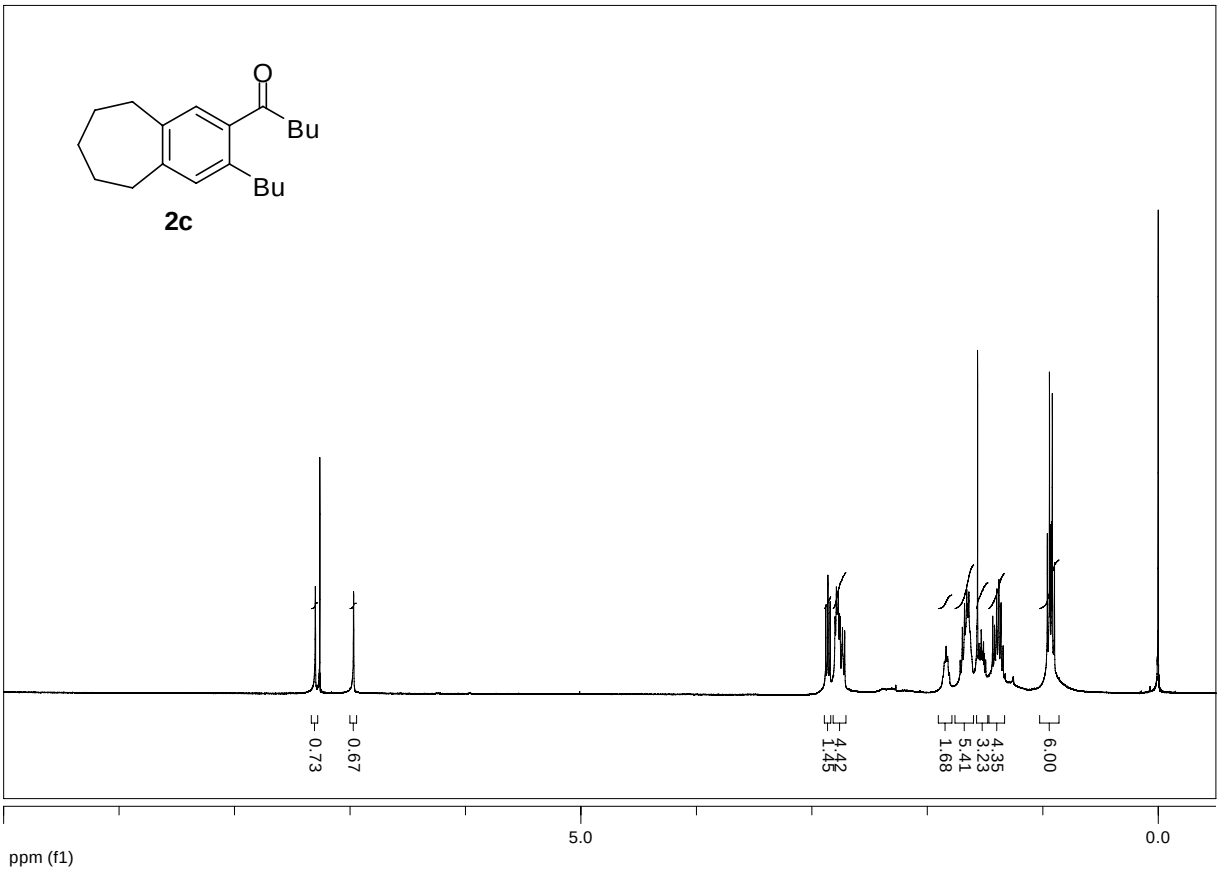


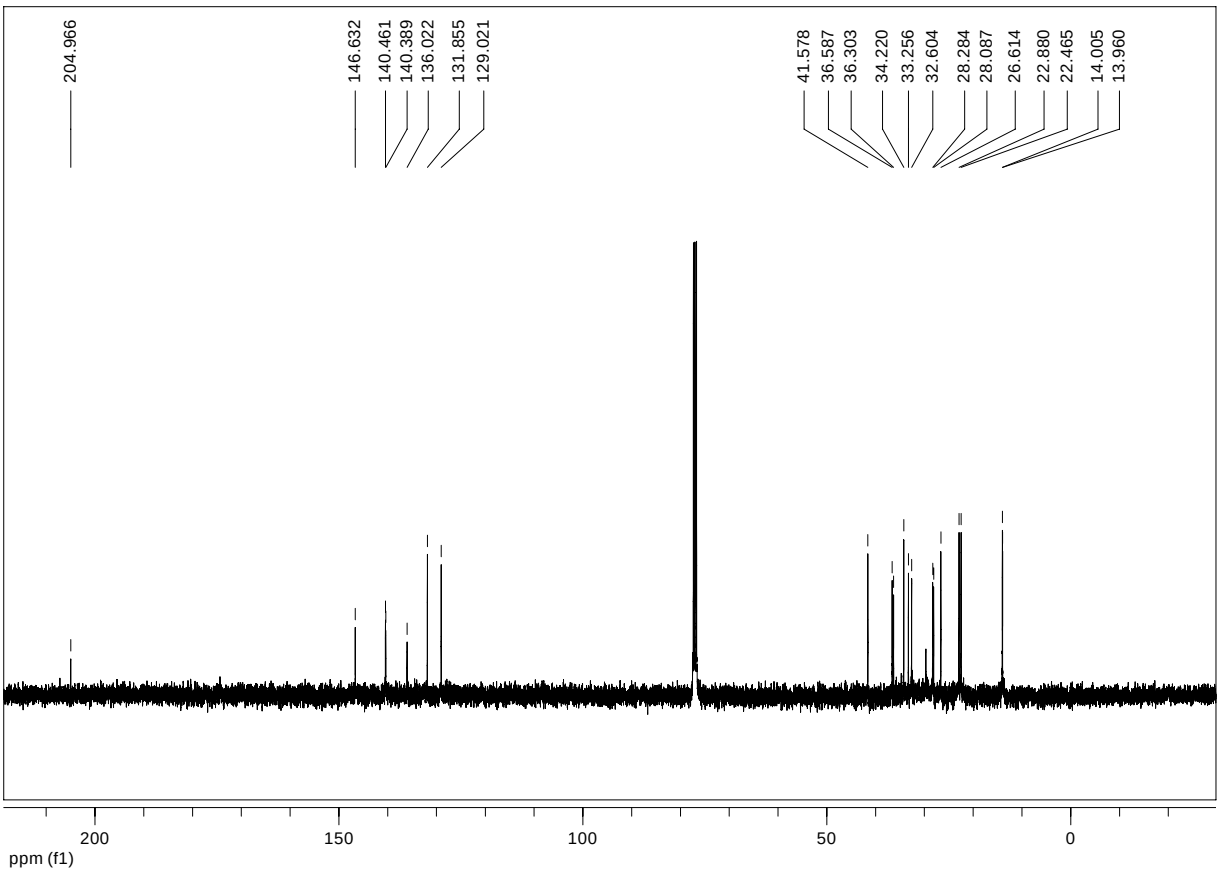


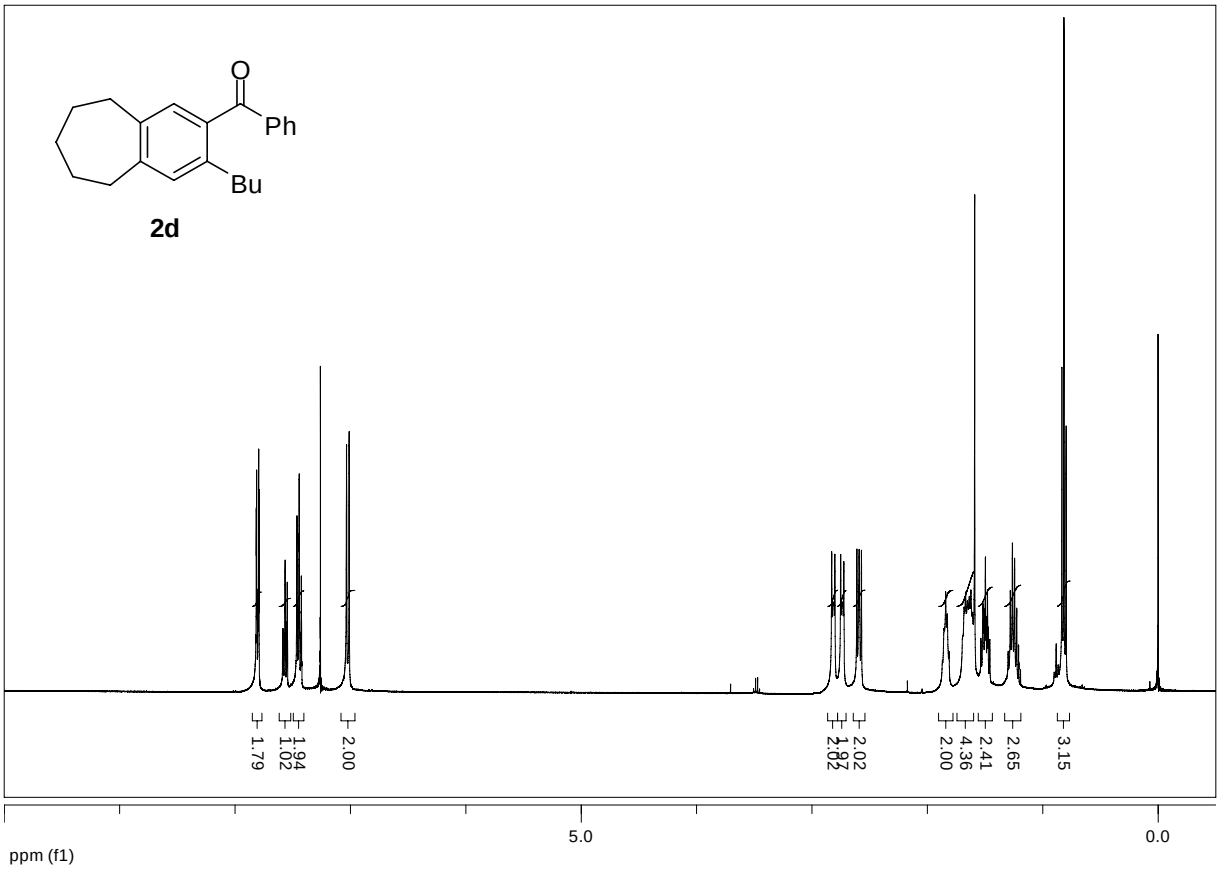


2b

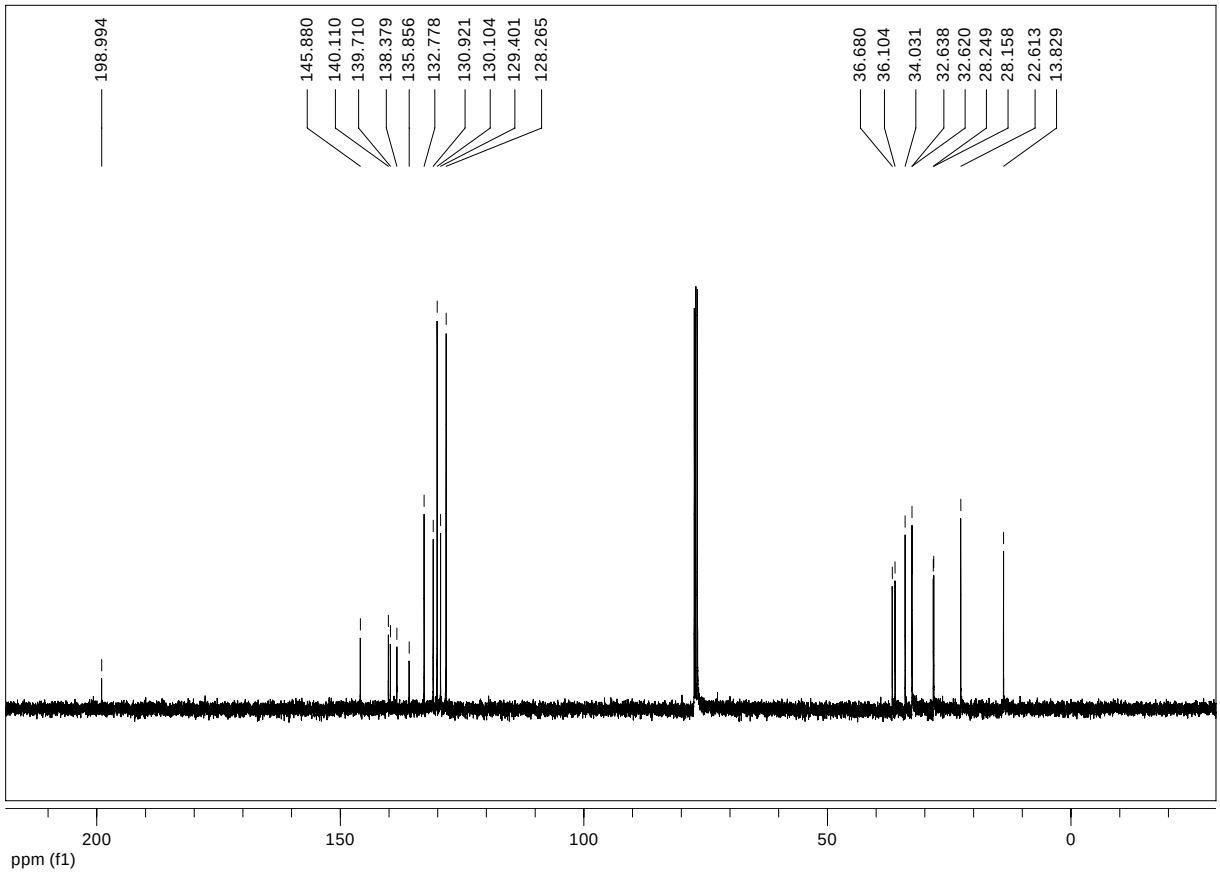


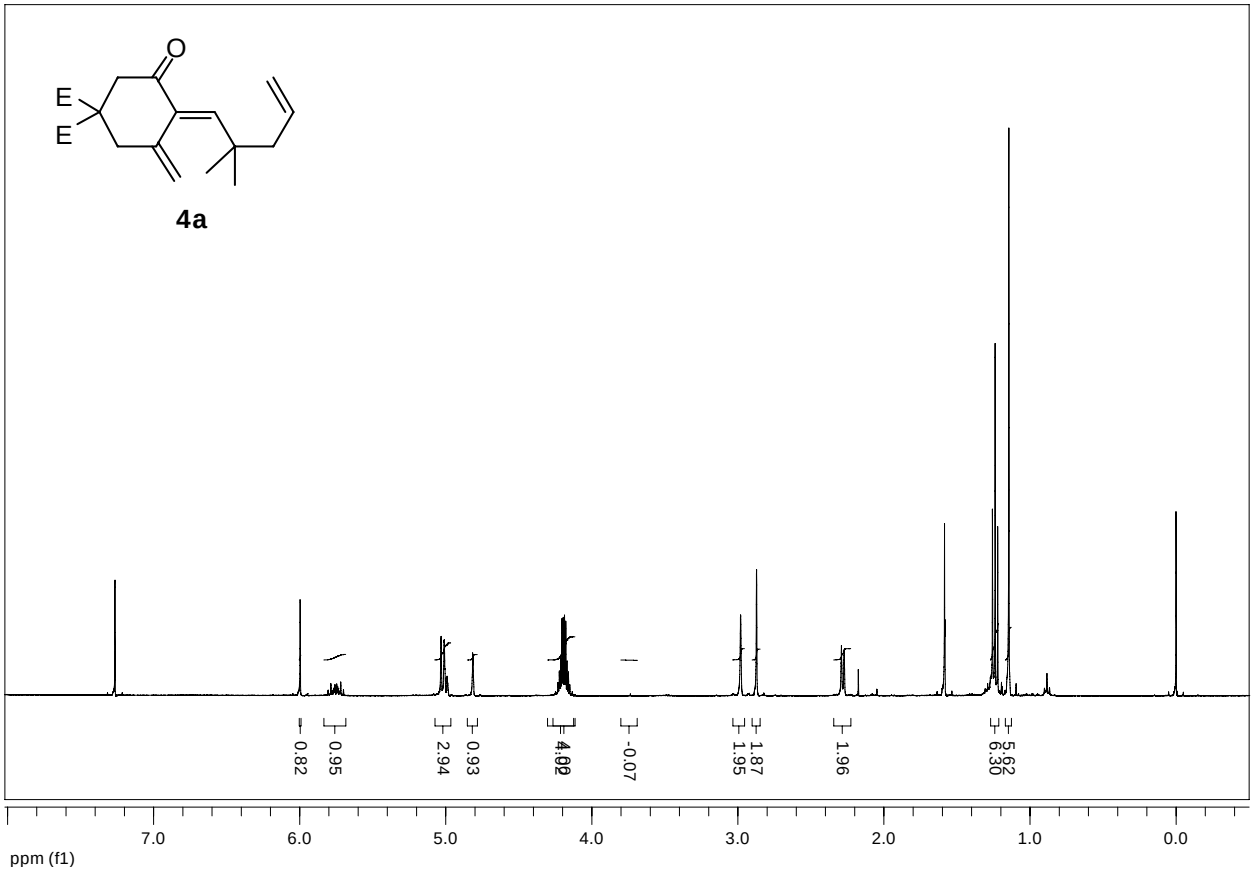


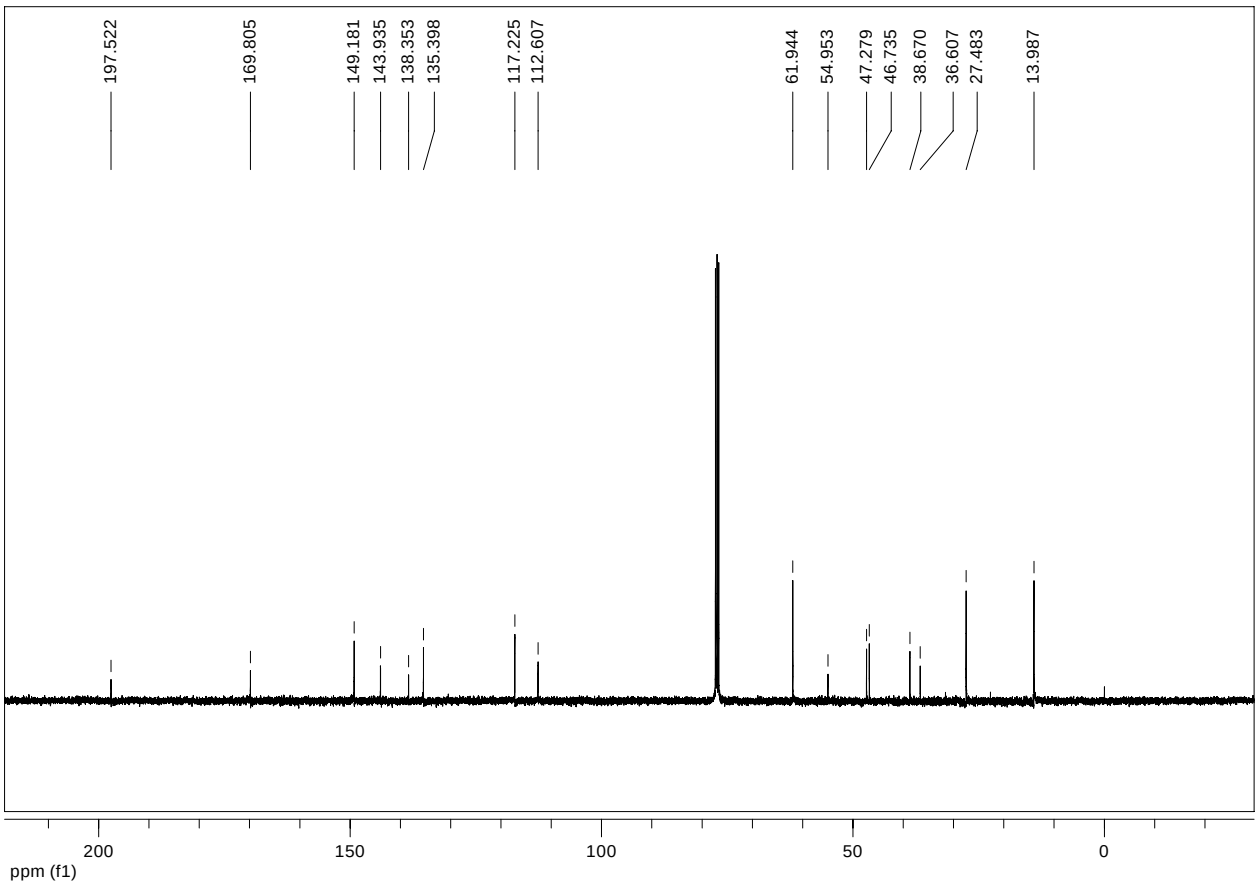


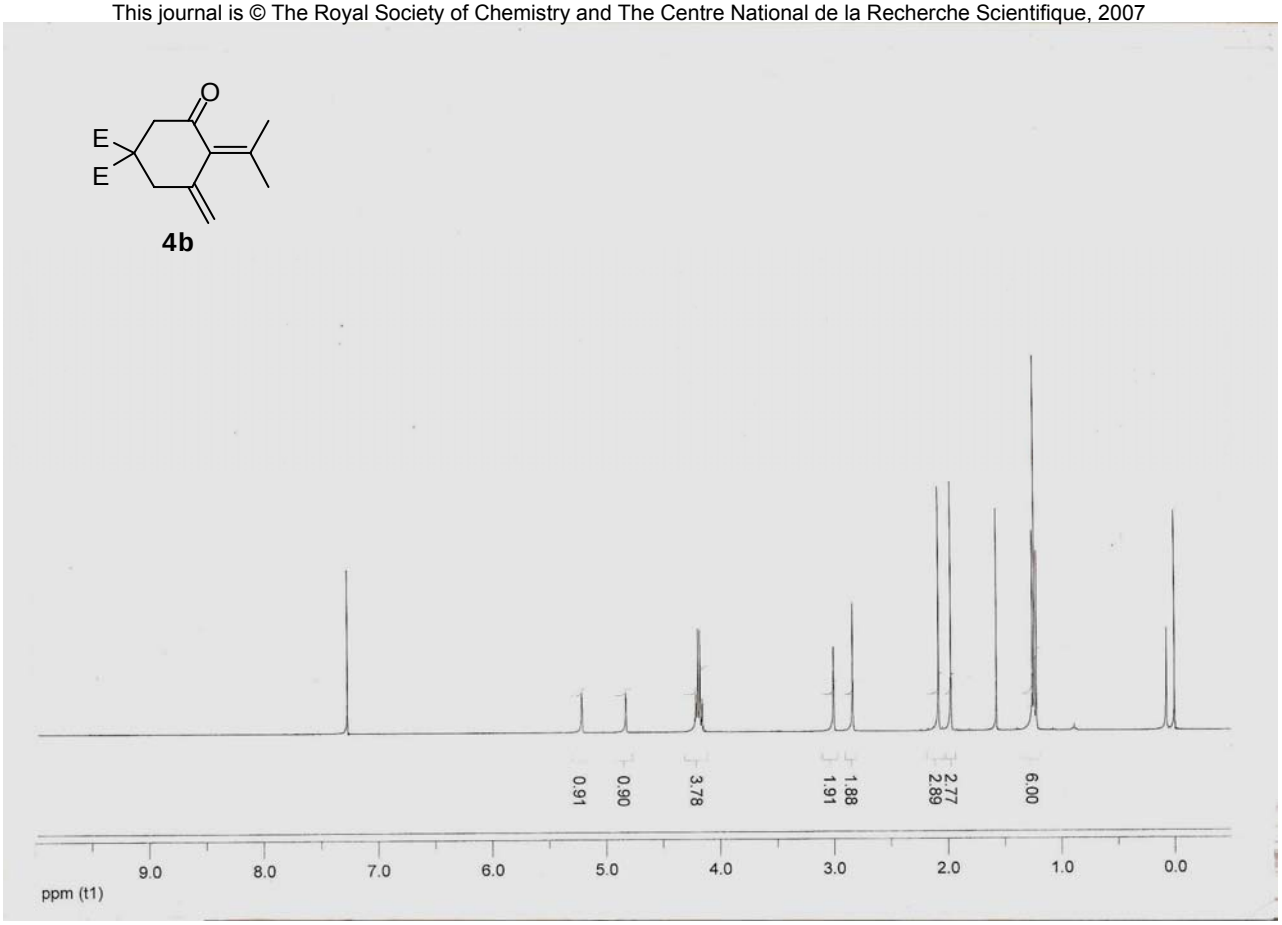


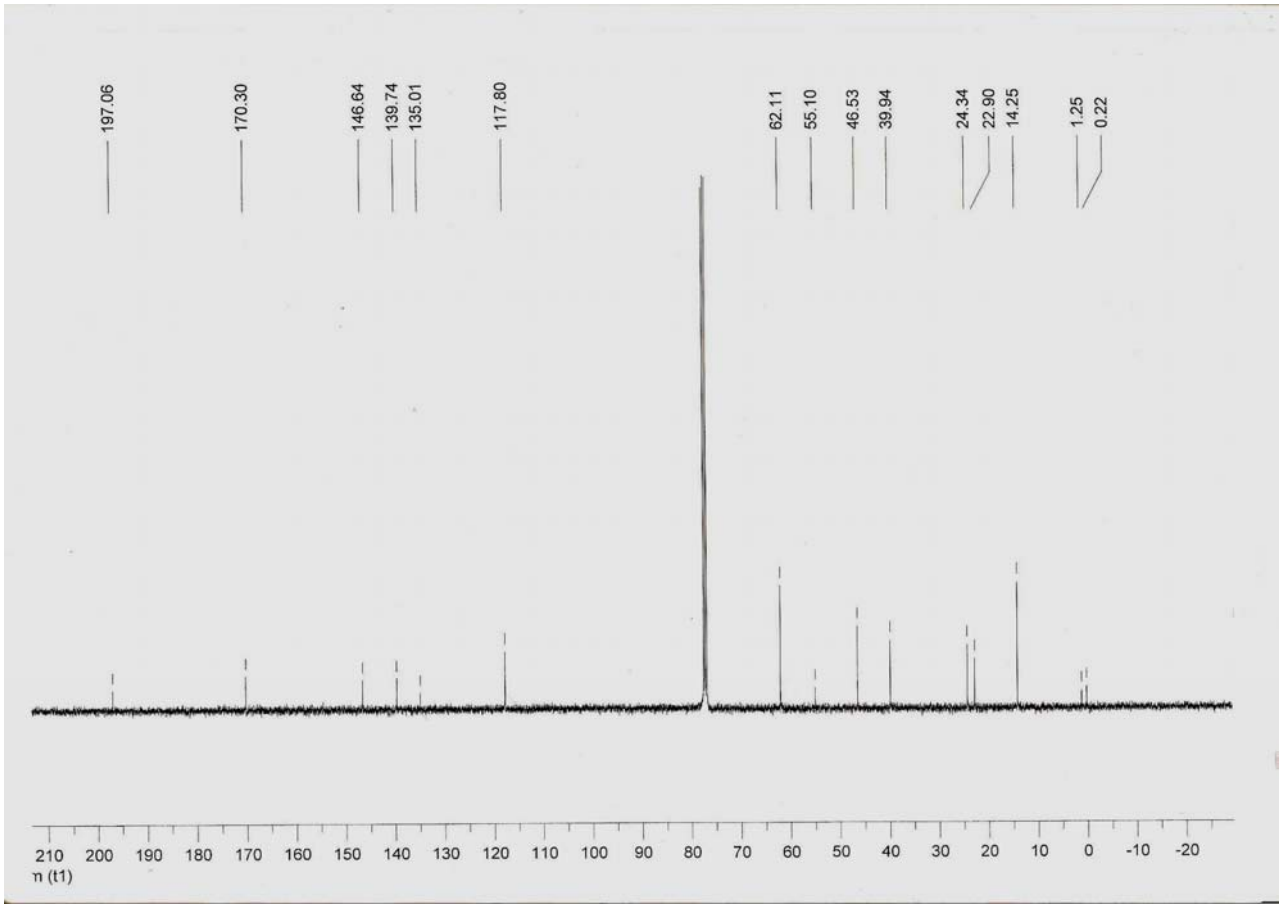
2d

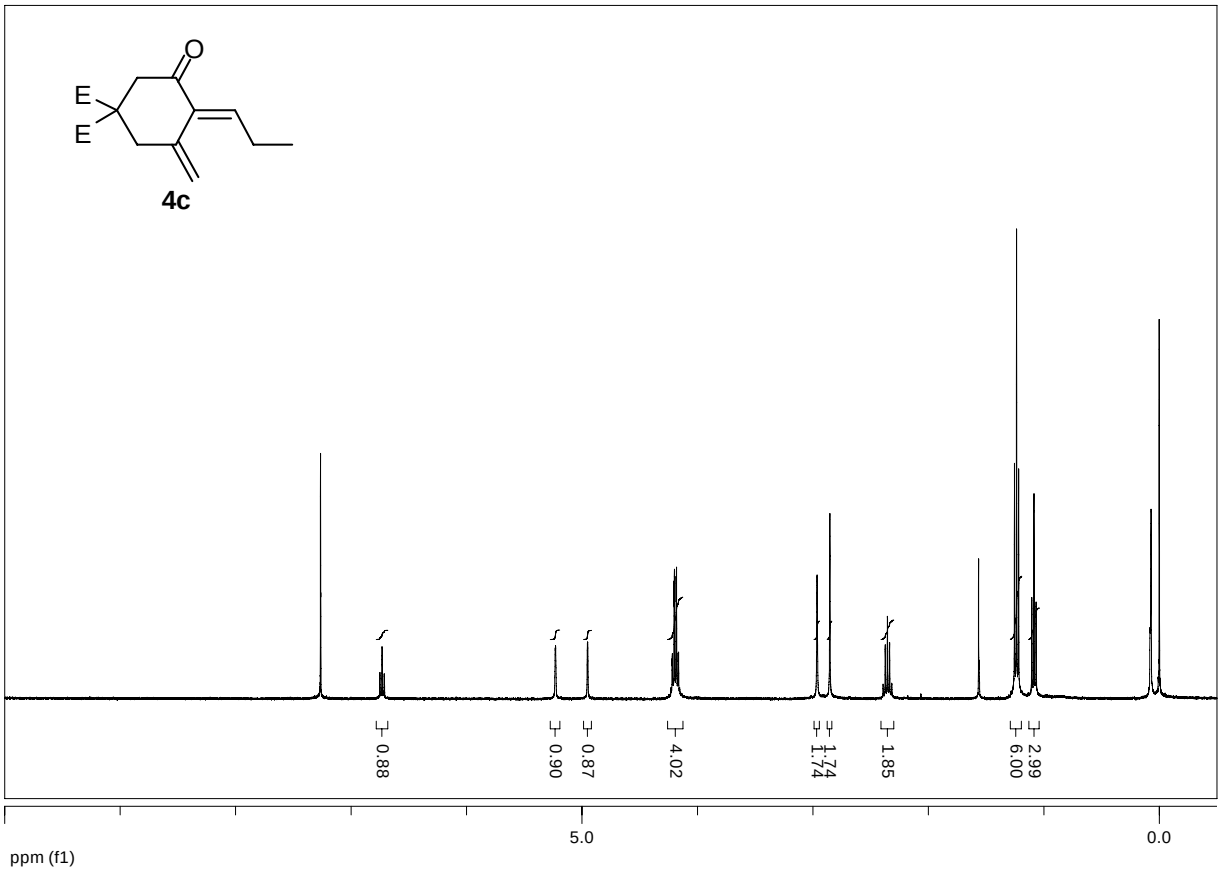












4c