Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Allylic and benzylic sp^3 C-H oxidation in water

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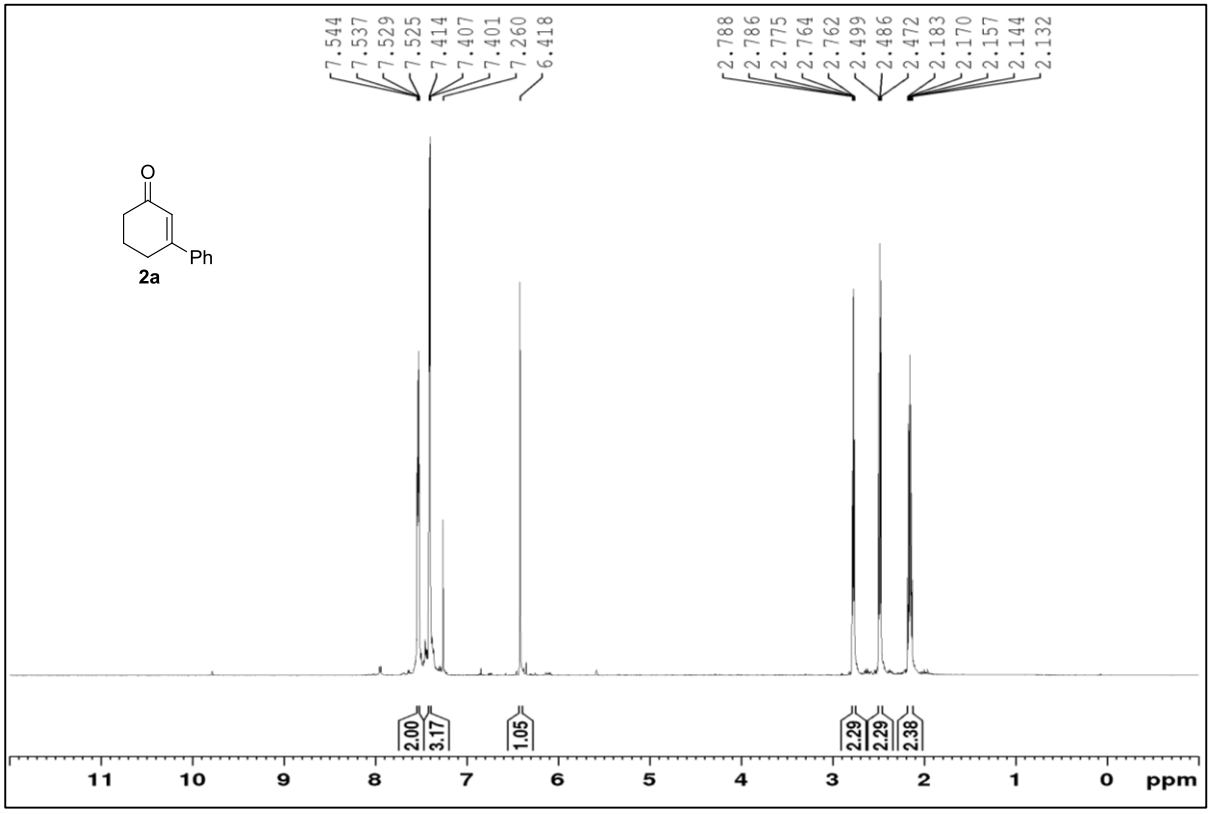
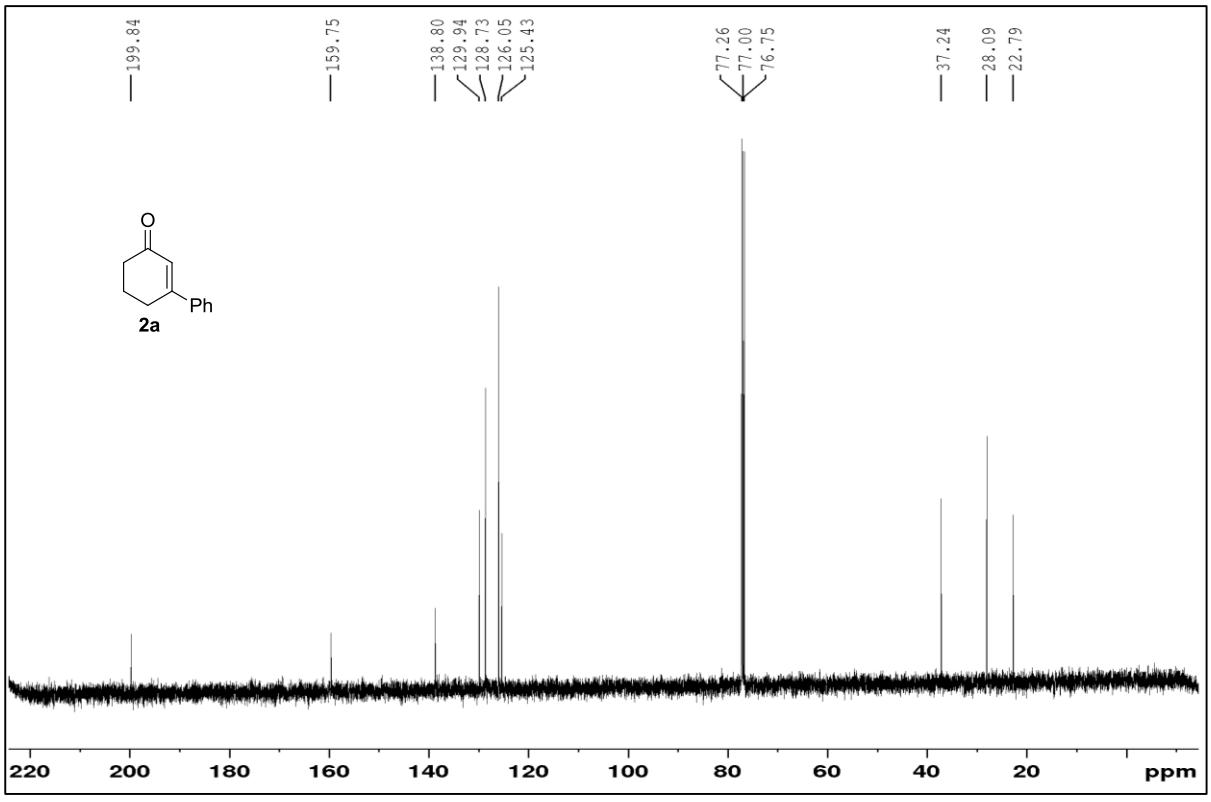
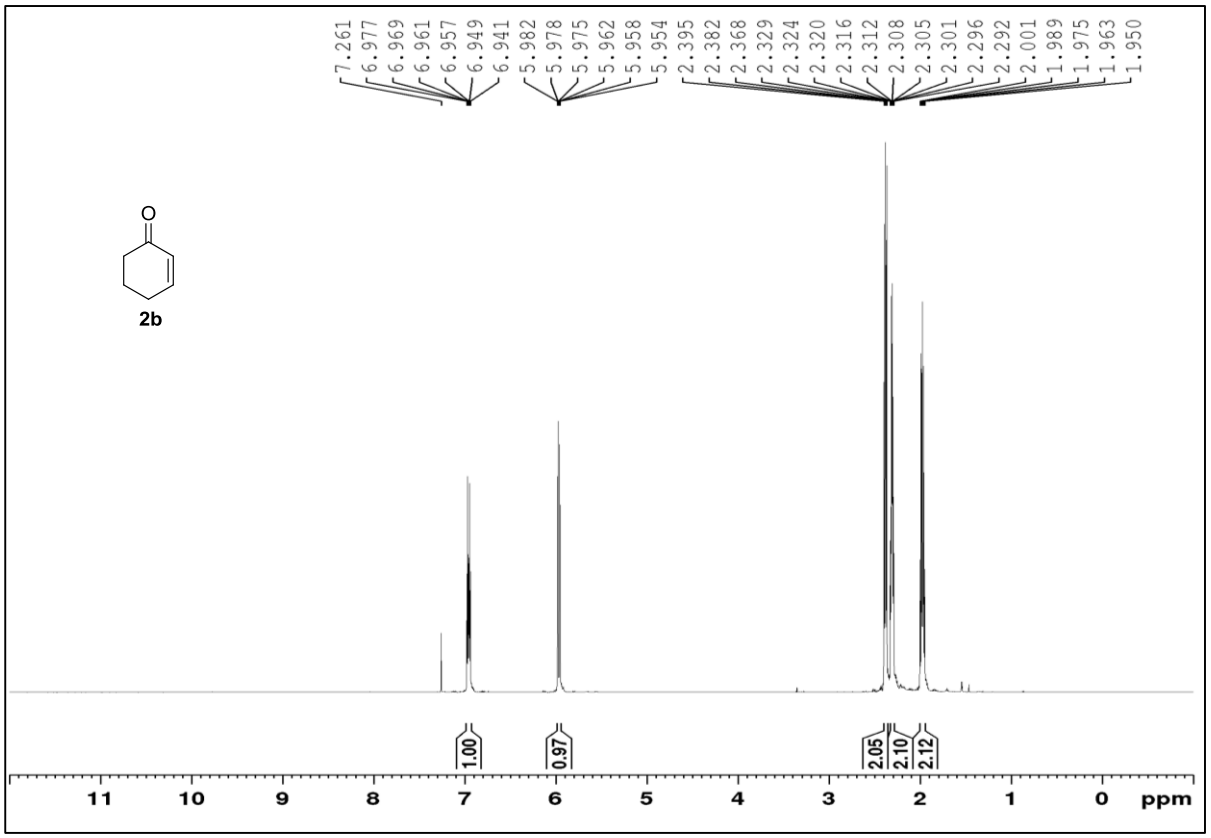
NMR and mass spectrometry characterization data

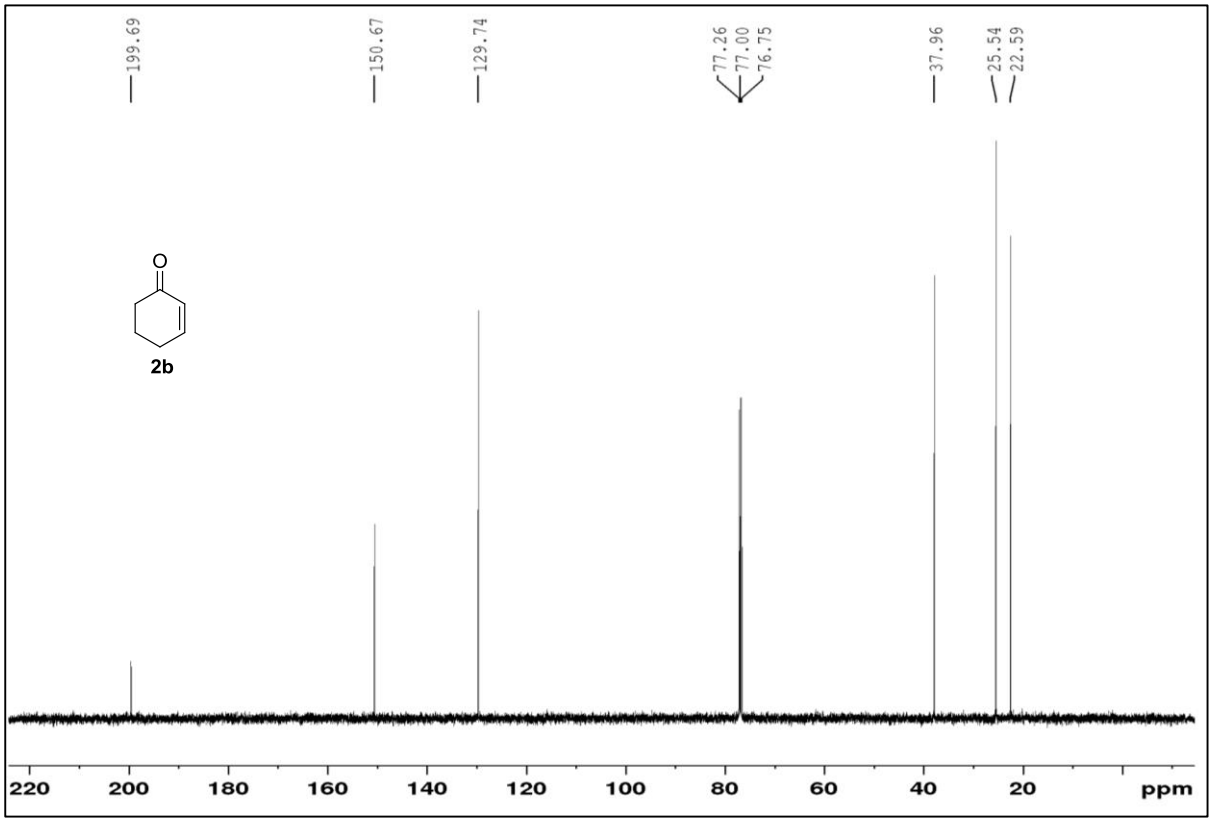
3-Phenylcyclohex-2-enone (**2a**). Pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (dd, *J* = 6.7, 3.1 Hz, 2H), 7.43 – 7.39 (m, 3H), 6.42 (s, 1H), 2.80 – 2.74 (m, 2H), 2.48 (d, *J* = 7.0 Hz, 2H), 2.15 (dd, *J* = 12.8, 6.4 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.8, 159.8, 138.8, 129.9, 128.7, 126.1, 125.4, 37.2, 28.1, 22.8. HRMS (EI): calcd for $C_{12}H_{12}O$ 172.0888, found 172.0895.



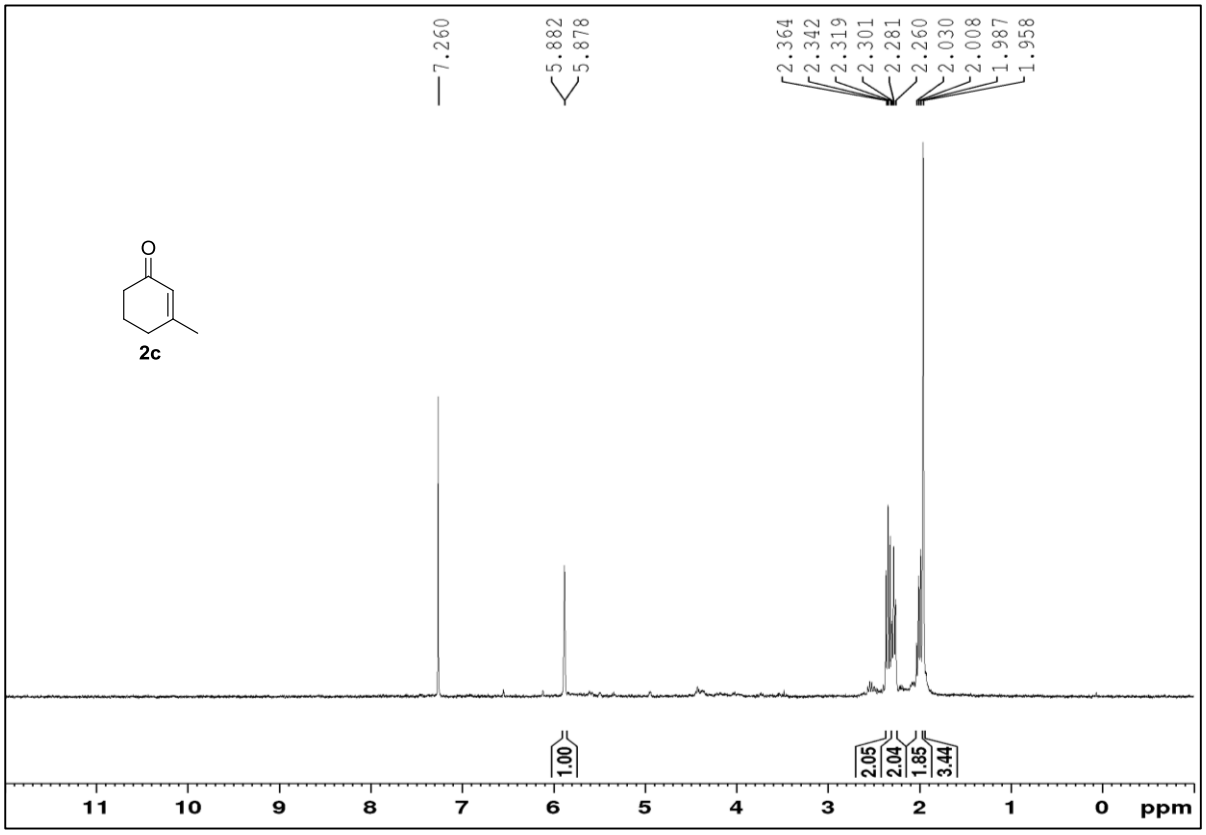


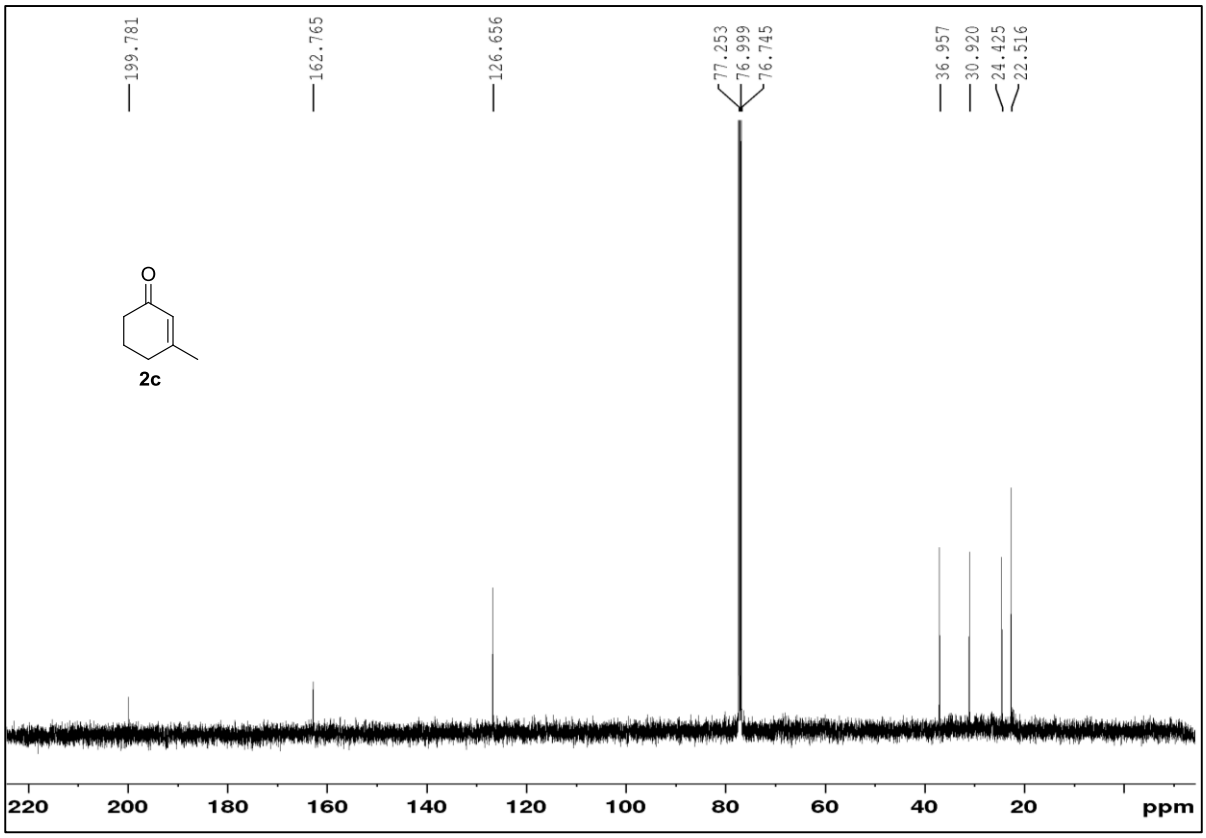
Cyclohex-2-enone (**2b**). Colorless liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.96 (dt, *J* = 10.0, 4.0 Hz, 1H), 5.97 (dt, *J* = 10.1, 2.0 Hz, 1H), 2.41 – 2.33 (m, 2H), 2.31 (tdd, *J* = 6.1, 4.2, 2.0 Hz, 2H), 1.98 (dt, *J* = 12.4, 6.1 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.7, 150.7, 129.7, 38.0, 25.5, 22.6. HRMS (EI): calcd for C_6H_8O 96.0575, found 96.0578.



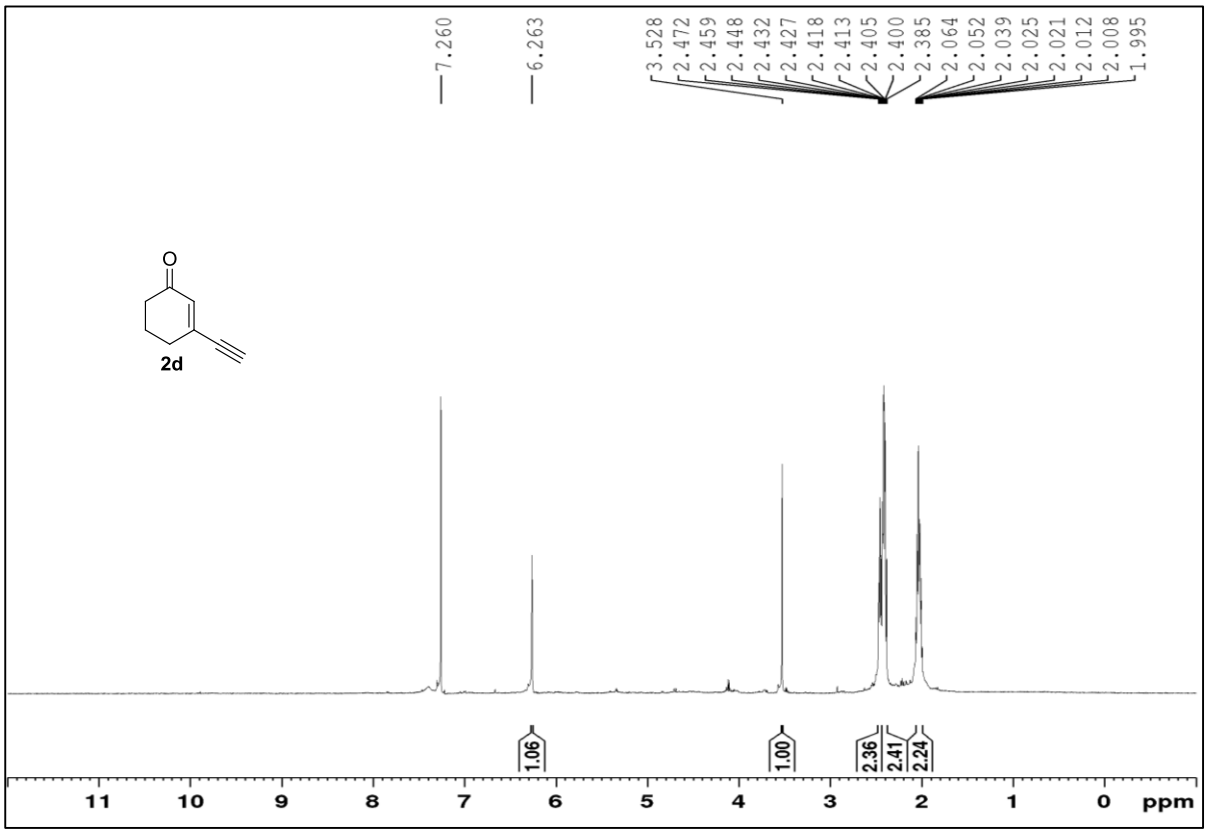


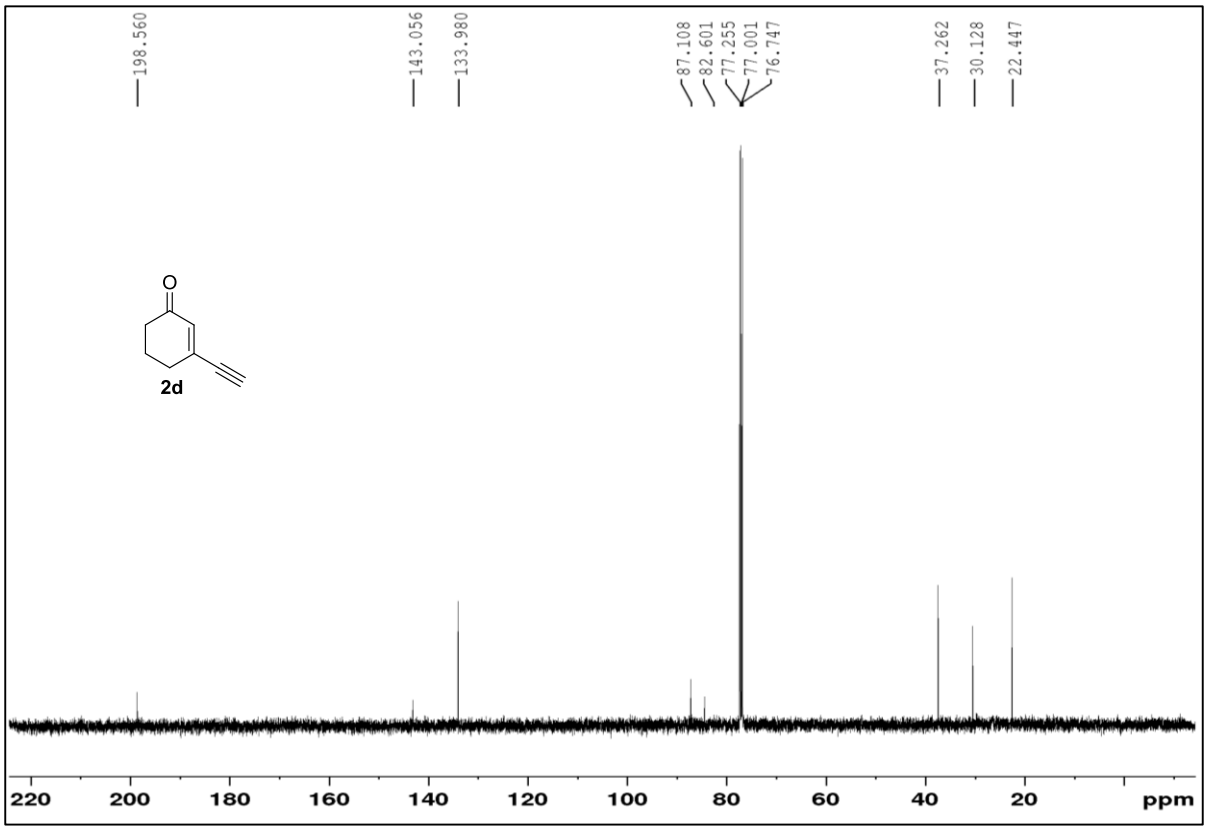
3-Methylcyclohex-2-enone (**2c**). Pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.88 (d, *J* = 1.2 Hz, 1H), 2.38 – 2.31 (m, 2H), 2.28 (t, *J* = 6.2 Hz, 2H), 2.04 – 1.97 (m, 2H), 1.96 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.8, 162.8, 126.7, 37.0, 30.9, 24.4, 22.5. HRMS (EI): calcd for $C_7H_{10}O$ 110.0732, found 110.0730.



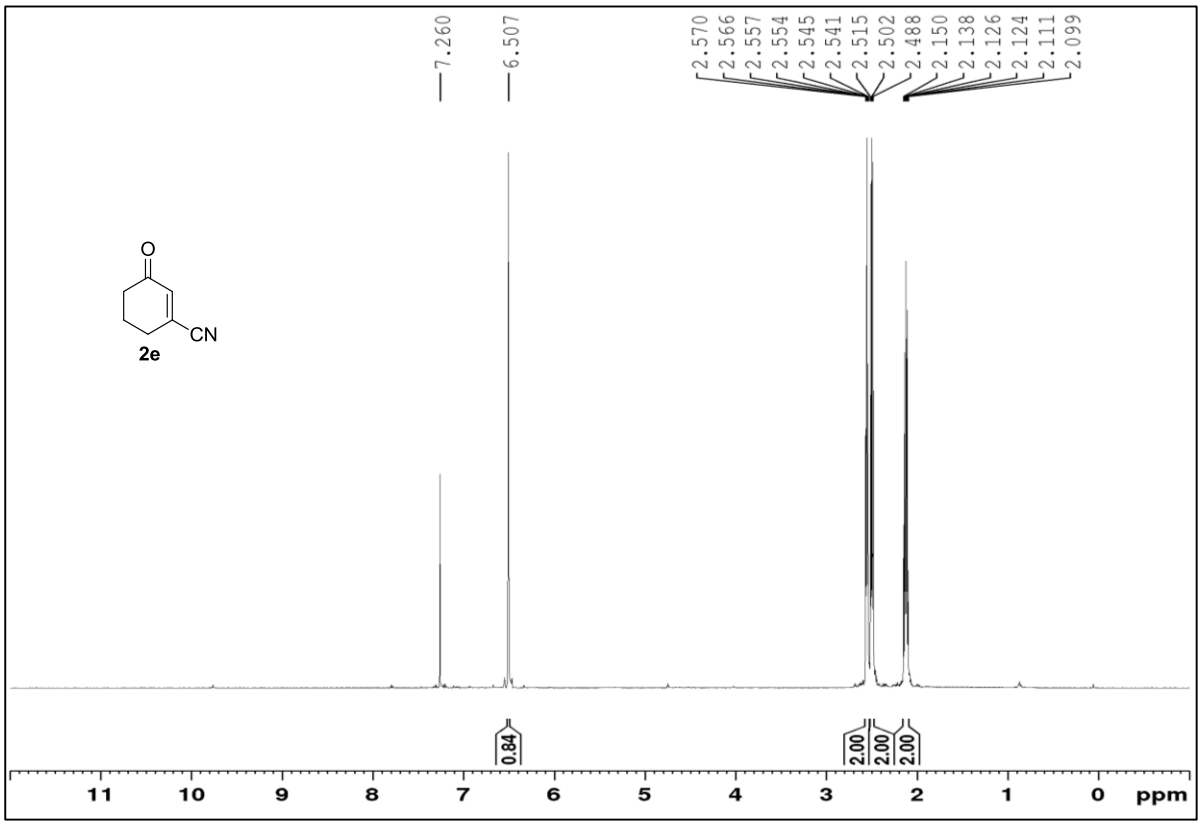


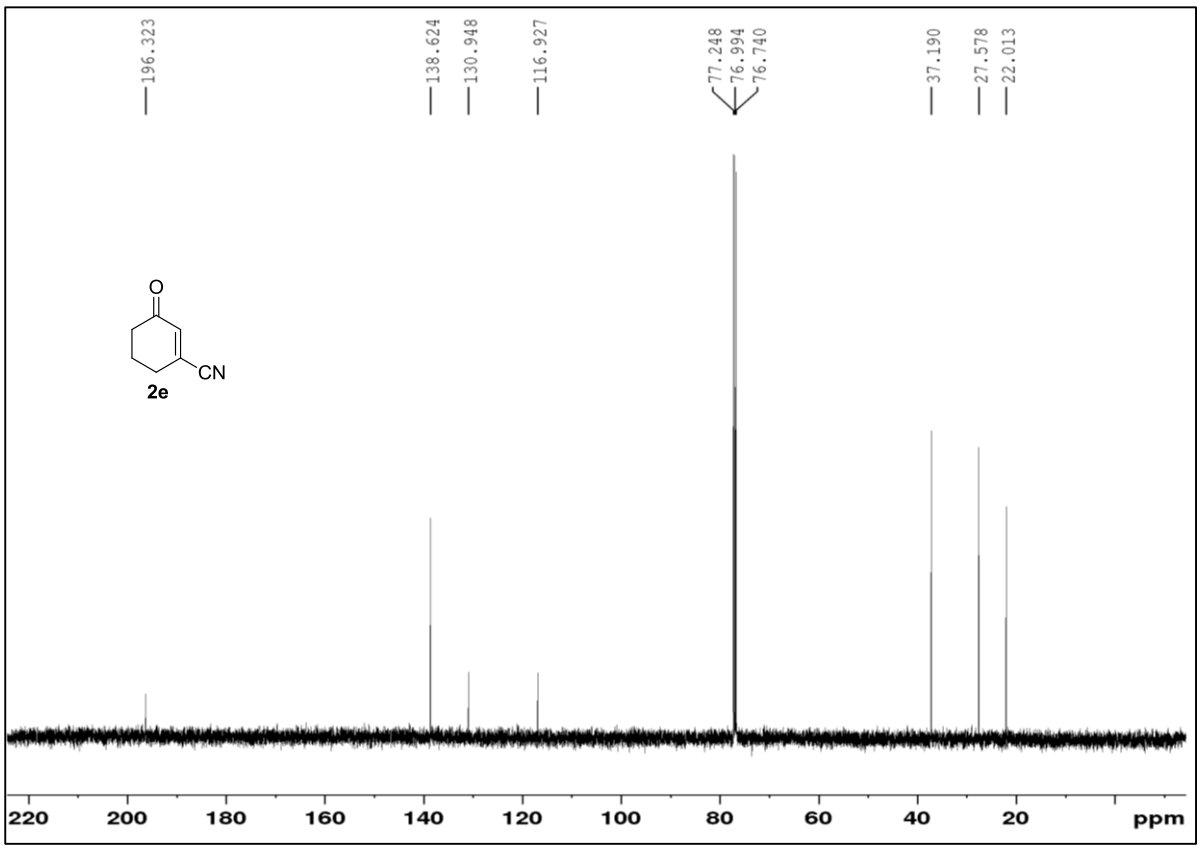
3-Ethynylcyclohex-2-enone (**2d**). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.26 (s, 1H), 3.53 (s, 1H), 2.46 (t, *J* = 6.0 Hz, 2H), 2.44 – 2.38 (m, 2H), 2.07 – 1.99 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 198.6, 143.1, 134.0, 87.1, 82.6, 37.3, 30.1, 22.4. HRMS (EI): calcd for C_8H_8O 120.0575, found 120.0573.



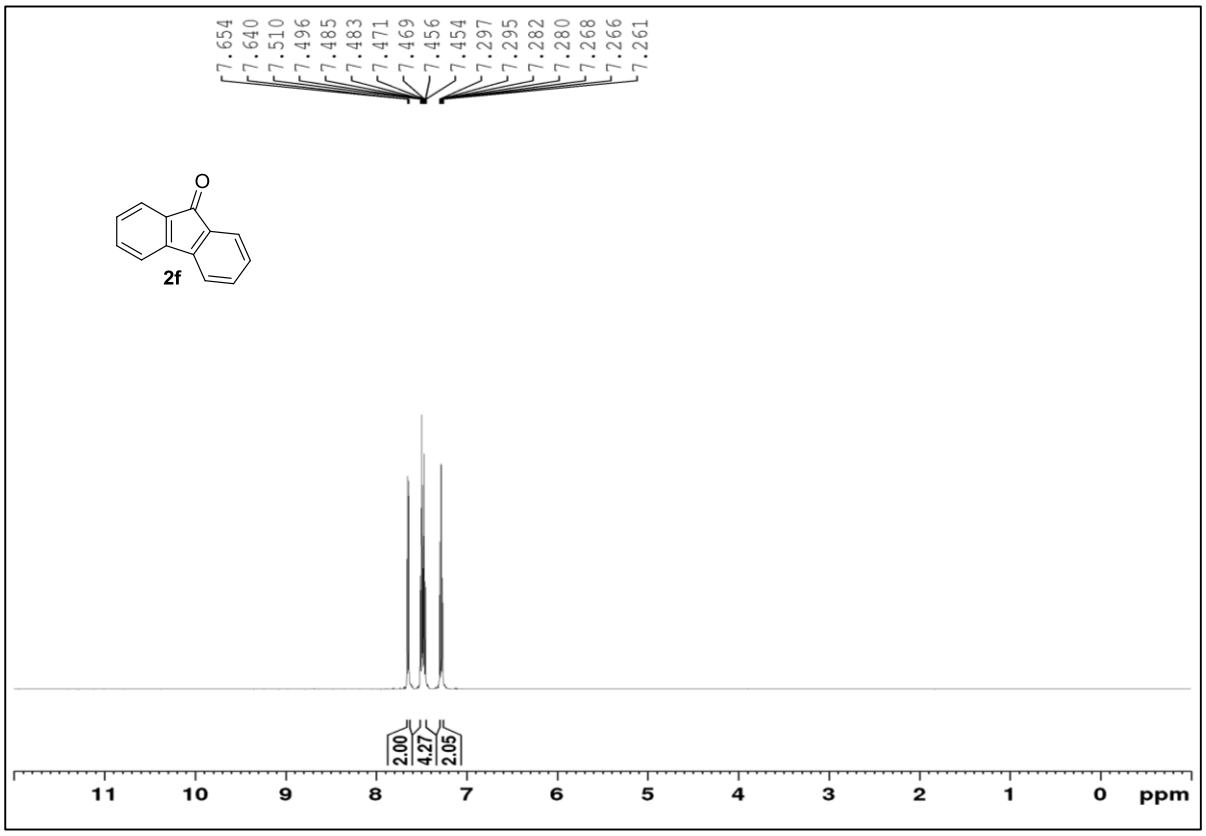


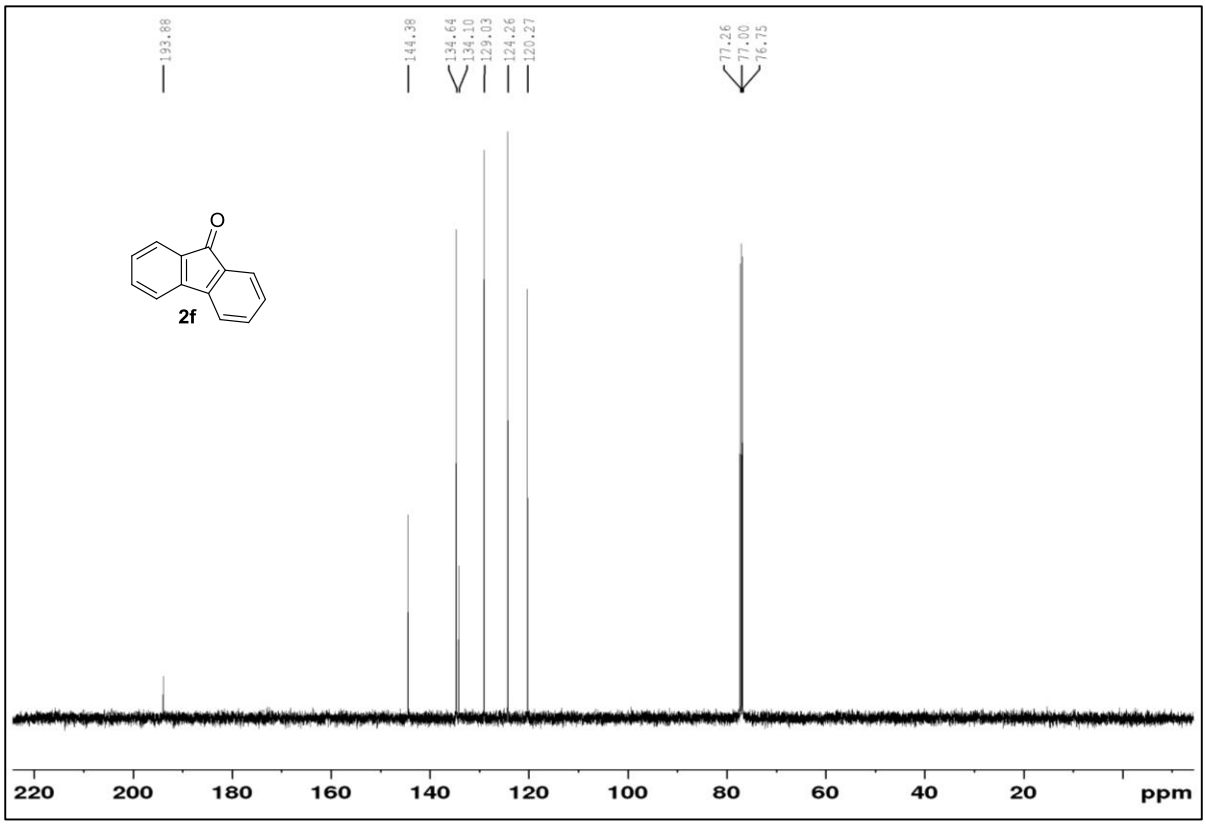
3-Oxocyclohex-1-enecarbonitrile (**2e**). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.51 (s, 1H), 2.56 (td, *J* = 6.0, 1.9 Hz, 2H), 2.52 – 2.48 (m, 2H), 2.13 (td, *J* = 12.3, 6.1 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 196.3, 138.6, 130.9, 116.9, 37.2, 27.6, 22.0. HRMS (EI): calcd for C_7H_7NO 121.0528, found 121.0528.



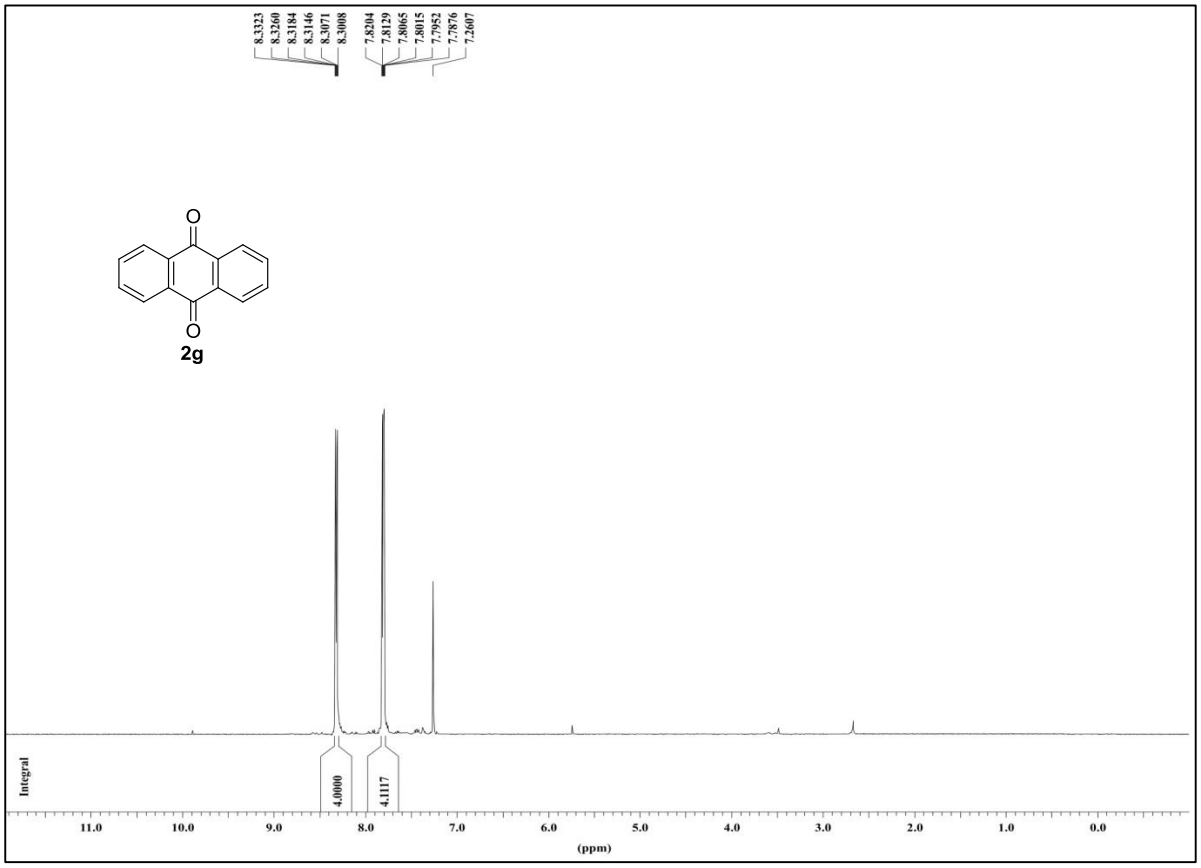


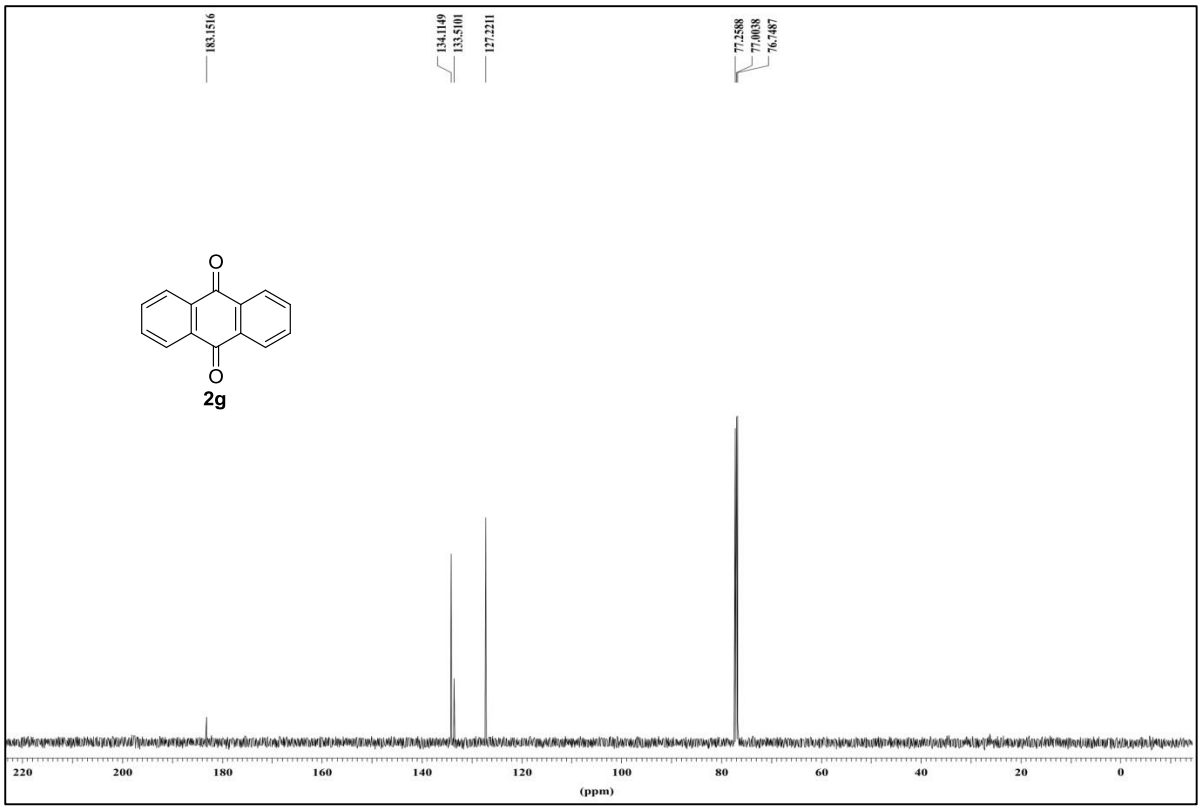
9H-Fluoren-9-one (**2f**). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.4 Hz, 2H), 7.48 (ddd, *J* = 10.0, 8.5, 4.2 Hz, 4H), 7.28 (td, *J* = 7.3, 1.3 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 193.9, 144.4, 134.6, 134.1, 129.0, 124.3, 120.3. HRMS (EI): calcd for $C_{13}H_8O$ 180.0575, found 180.0575.



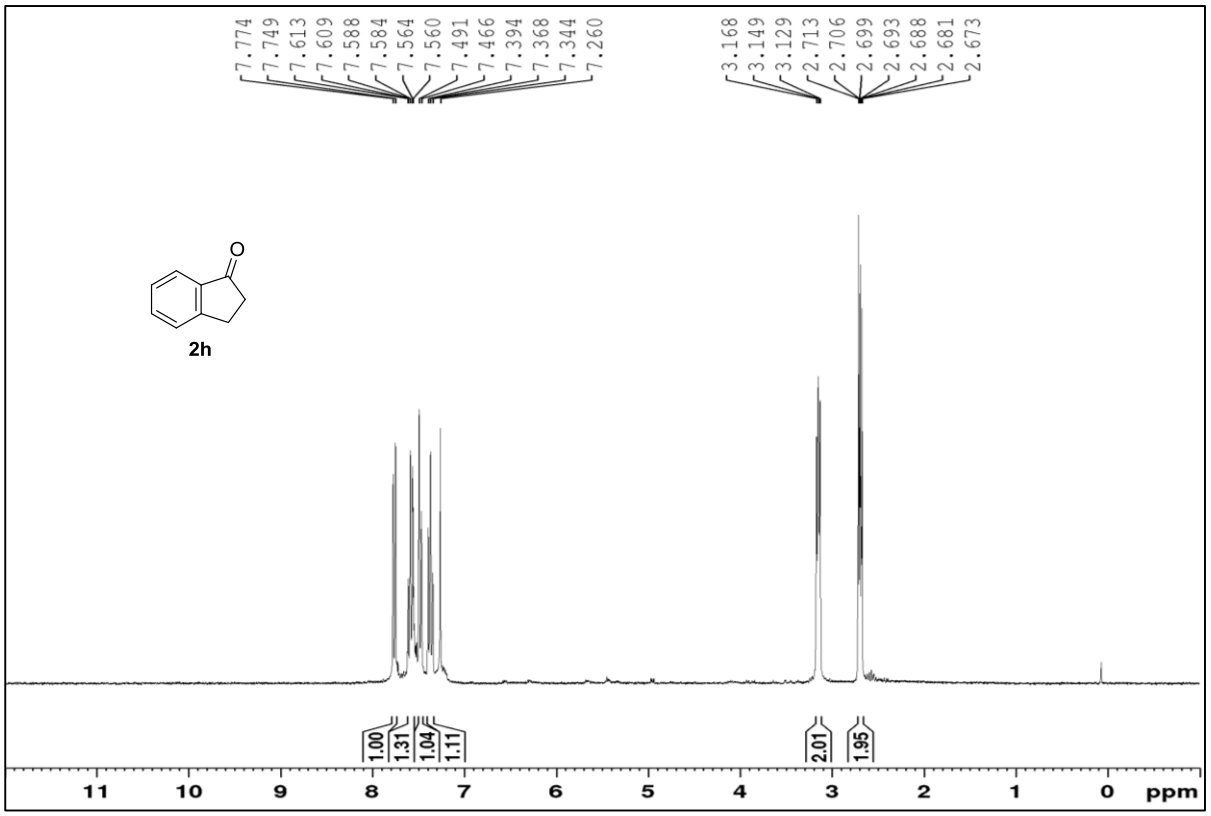


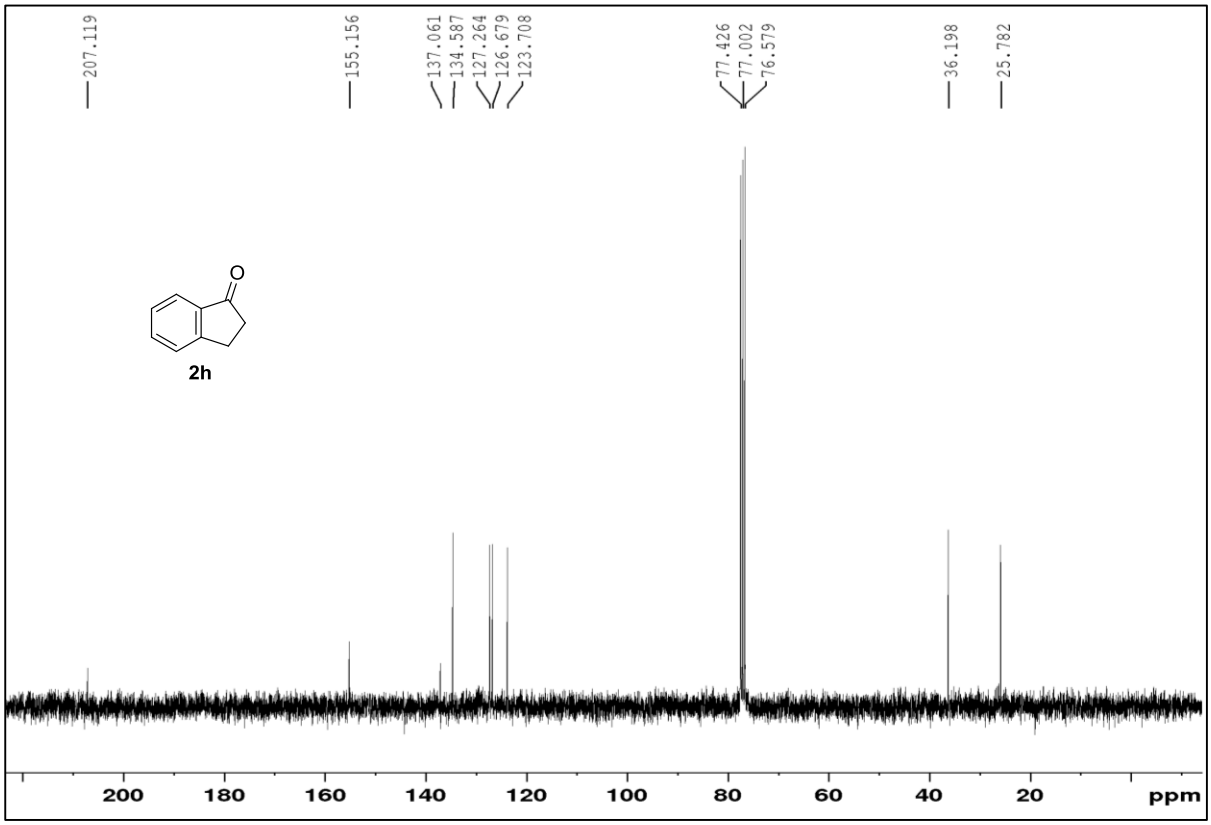
Anthracene-9,10-dione (**2g**). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.34 – 8.29 (m, 4H), 7.83 – 7.78 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 183.2, 134.1, 133.5, 127.2. HRMS (EI): calcd for $C_{14}H_8O_2$ 208.0524, found 208.0527.



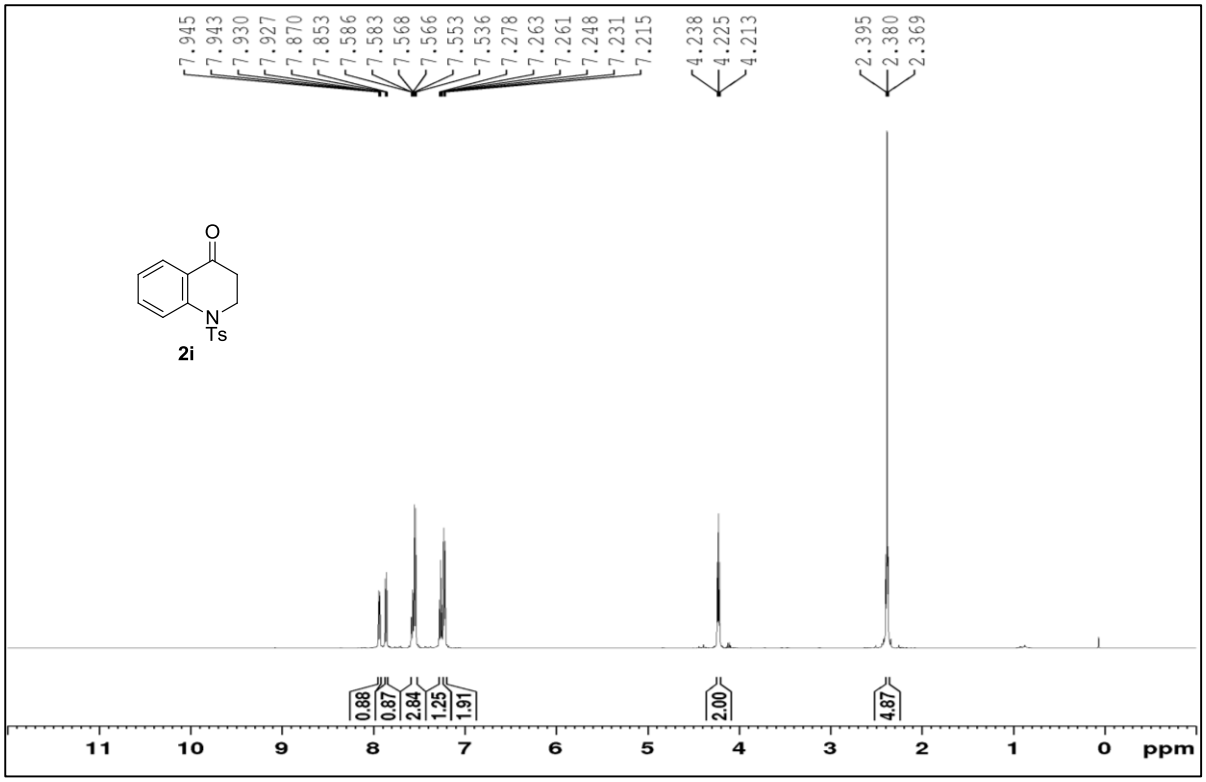


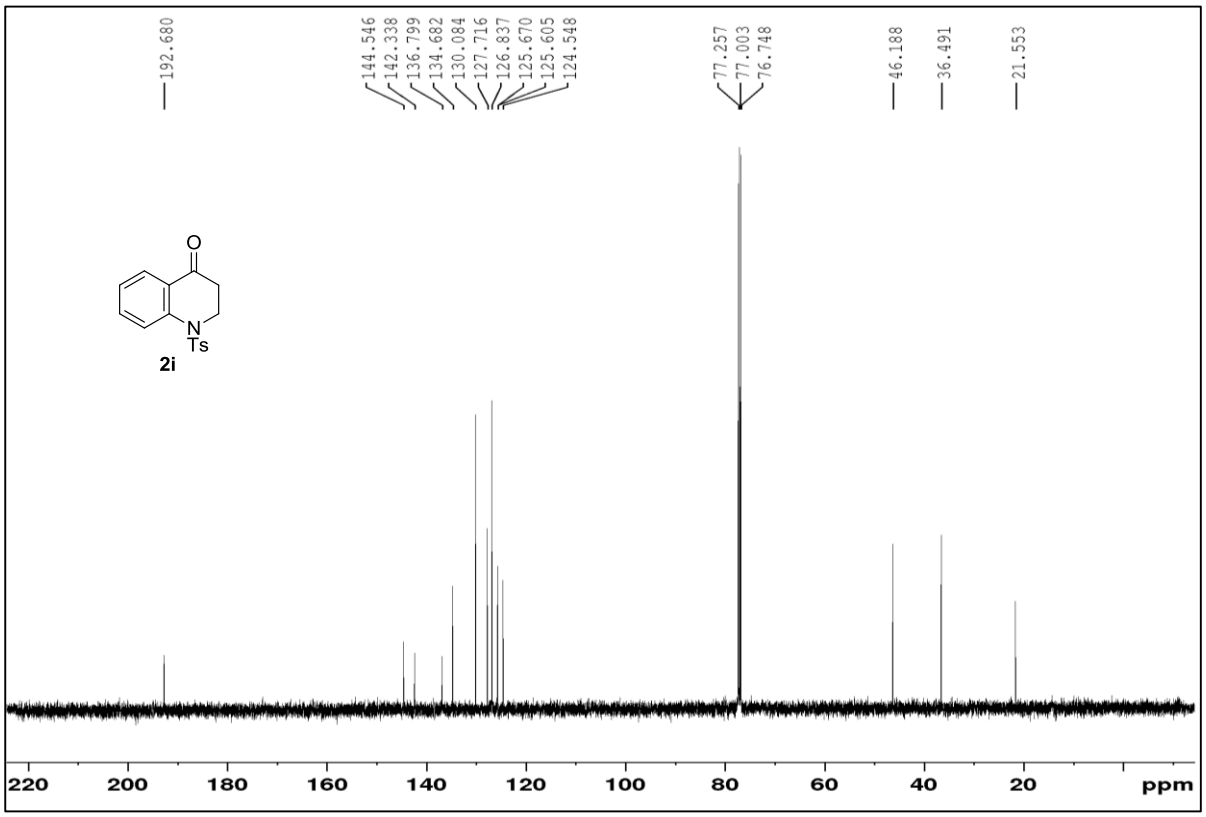
2,3-Dihydro-1H-inden-1-one (**2h**). Brown solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.7 Hz, 1H), 7.61 – 7.53 (m, 1H), 7.48 (d, *J* = 7.7 Hz, 1H), 7.37 (t, *J* = 7.4 Hz, 1H), 3.18 – 3.11 (m, 2H), 2.73 – 2.67 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.1, 155.2, 137.1, 134.6, 127.3, 126.7, 123.7, 36.2, 25.8. HRMS (EI): calcd for C_9H_8O 132.0575, found 132.0570.



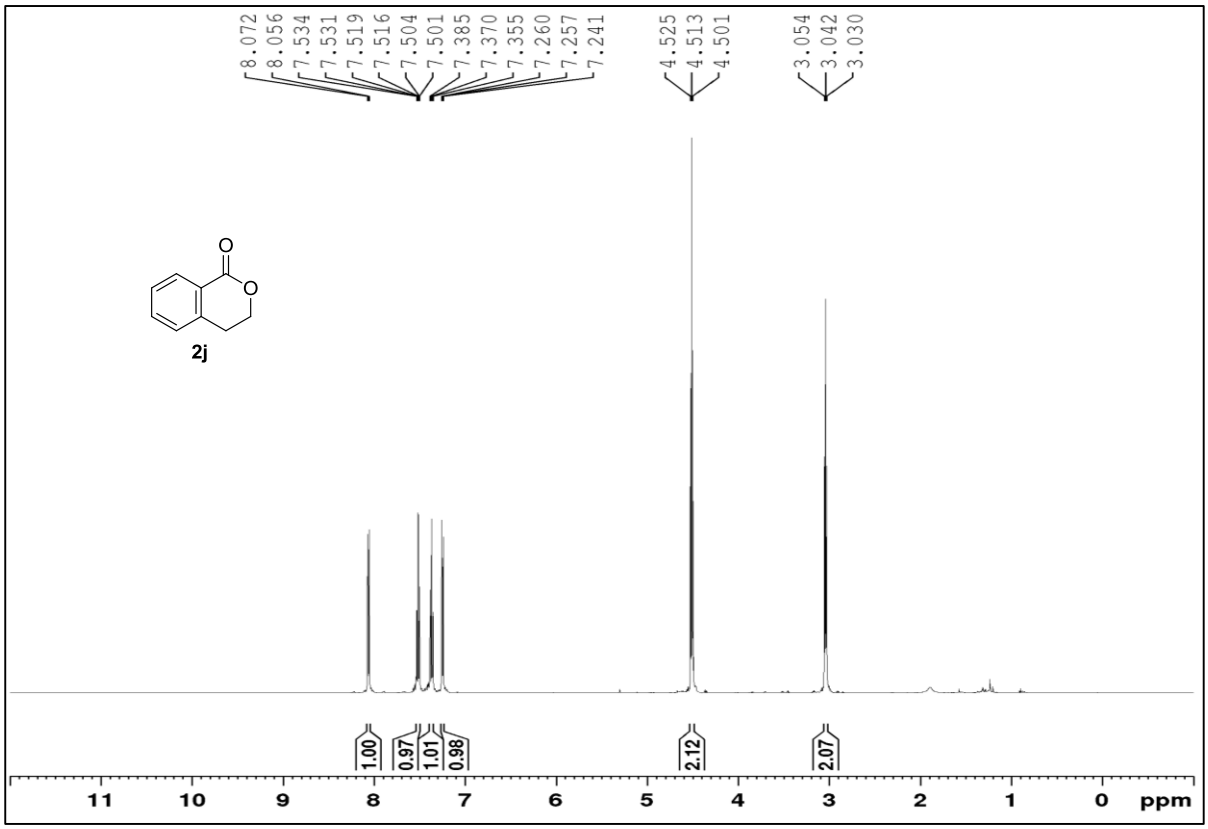


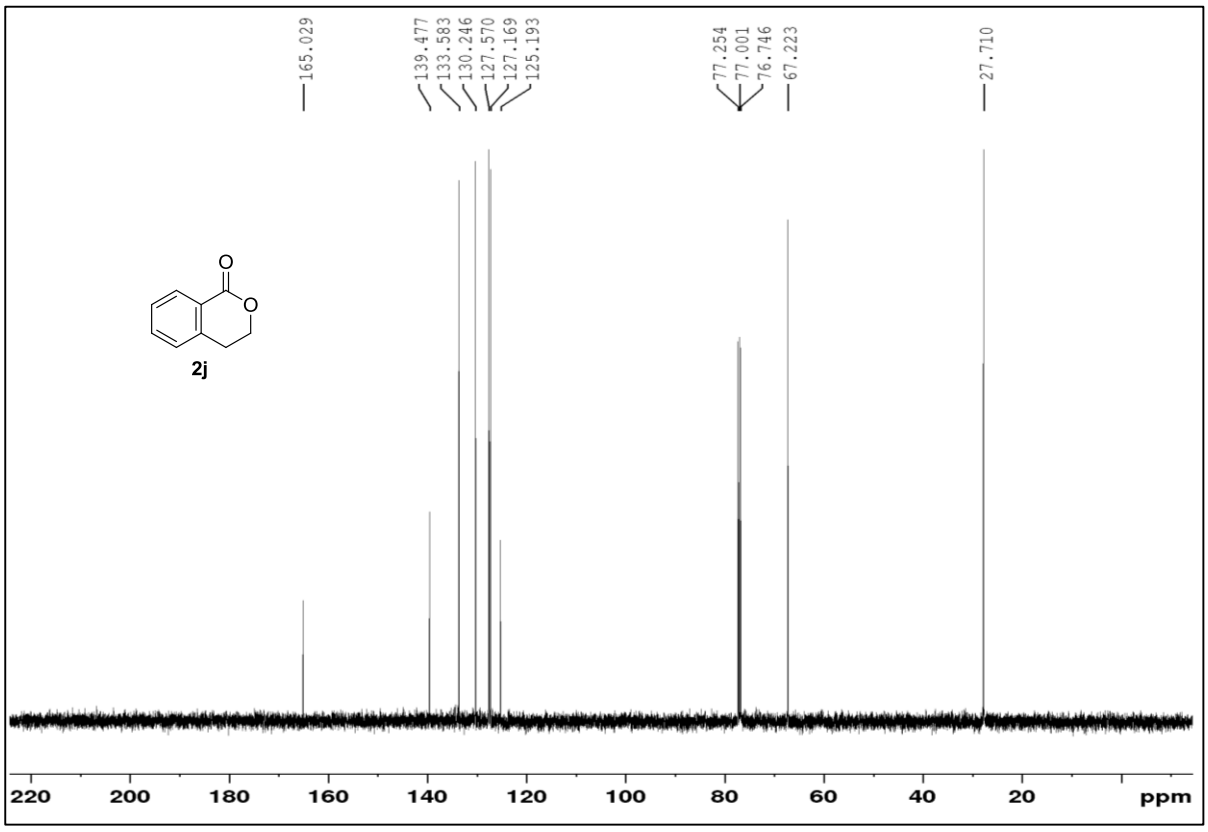
1-Tosyl-2,3-dihydroquinolin-4(1H)-one (**2i**). Brown solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.86 (d, *J* = 8.4 Hz, 1H), 7.59 – 7.53 (m, 3H), 7.26 (dd, *J* = 8.2, 7.0 Hz, 1H), 7.22 (d, *J* = 8.2 Hz, 2H), 4.23 (t, *J* = 6.5 Hz, 2H), 2.40 – 2.36 (m, 5H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 192.7, 144.5, 142.3, 136.8, 134.7, 130.1, 127.7, 126.8, 125.7, 125.6, 124.5, 46.2, 36.5, 21.6. HRMS (EI): calcd for $C_{16}H_{15}NO_3S$ 301.0773, found 301.0772.



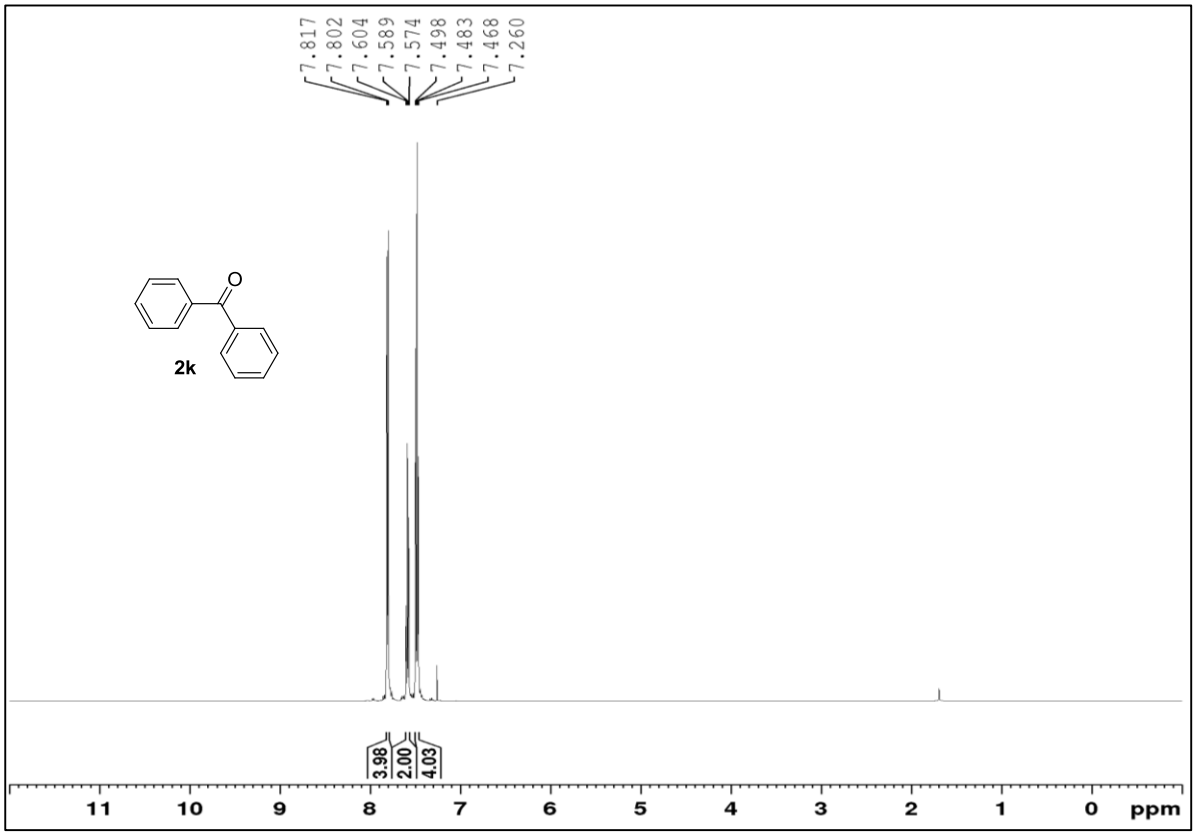


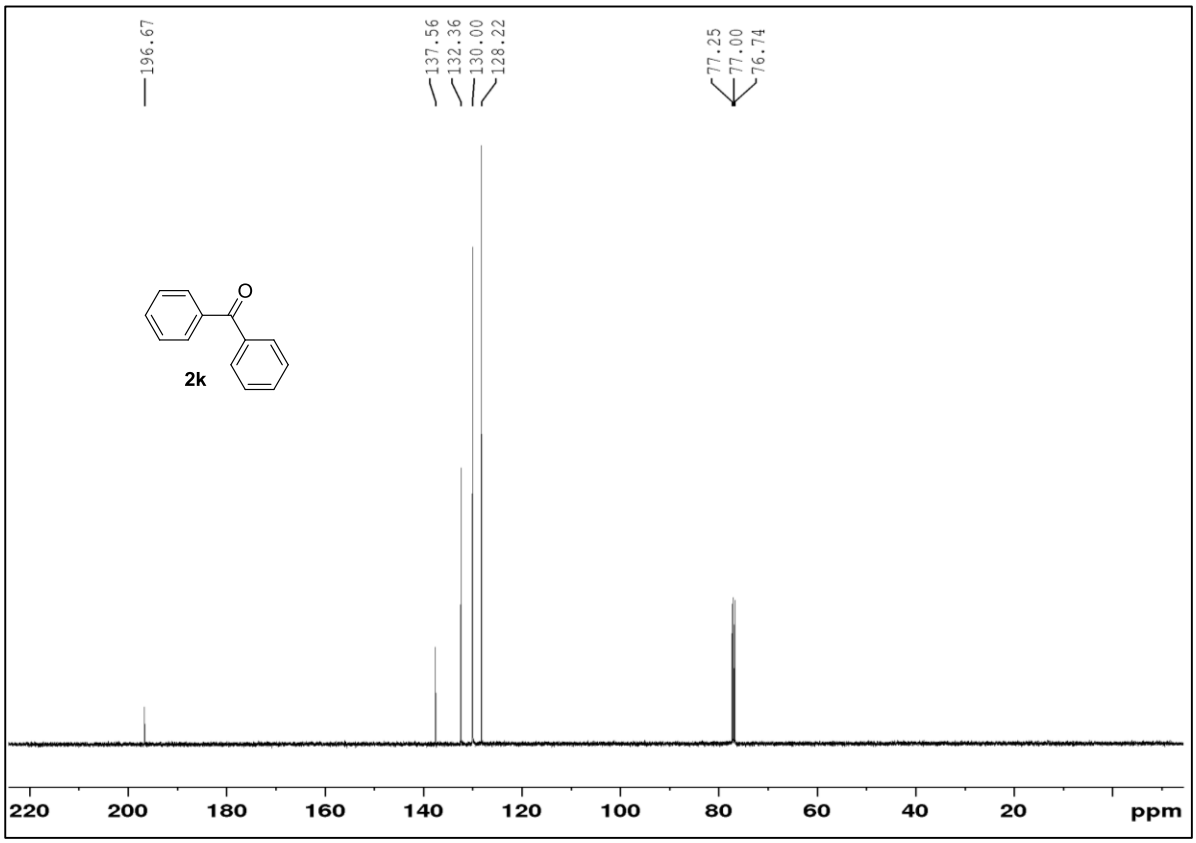
Isochroman-1-one (**2j**). Brown oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 (d, *J* = 7.8 Hz, 1H), 7.52 (td, *J* = 7.5, 1.4 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 7.7 Hz, 1H), 4.53 – 4.49 (m, 2H), 3.04 (t, *J* = 6.0 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.0, 139.5, 133.6, 130.2, 127.6, 127.2, 125.2, 67.2, 27.7. HRMS (EI): calcd for $C_9H_8O_2$ 148.0524, found 148.0523.



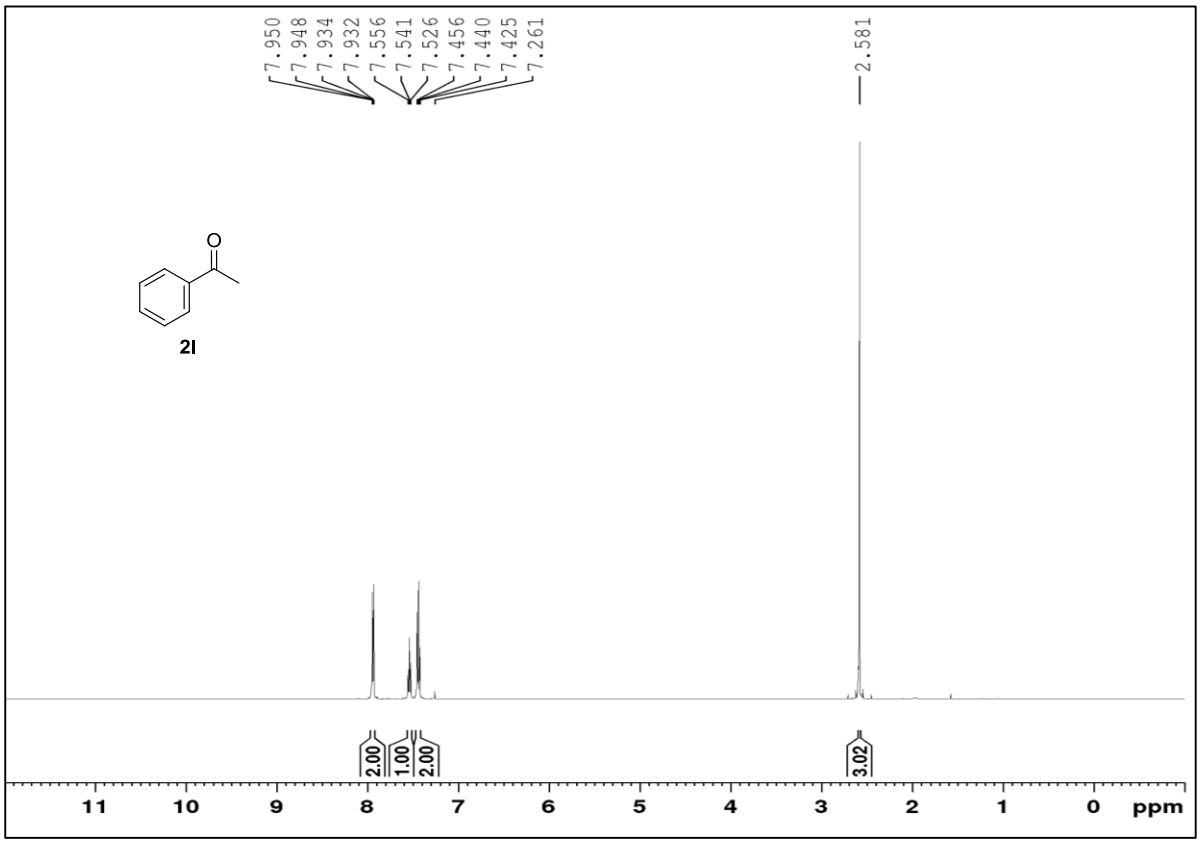


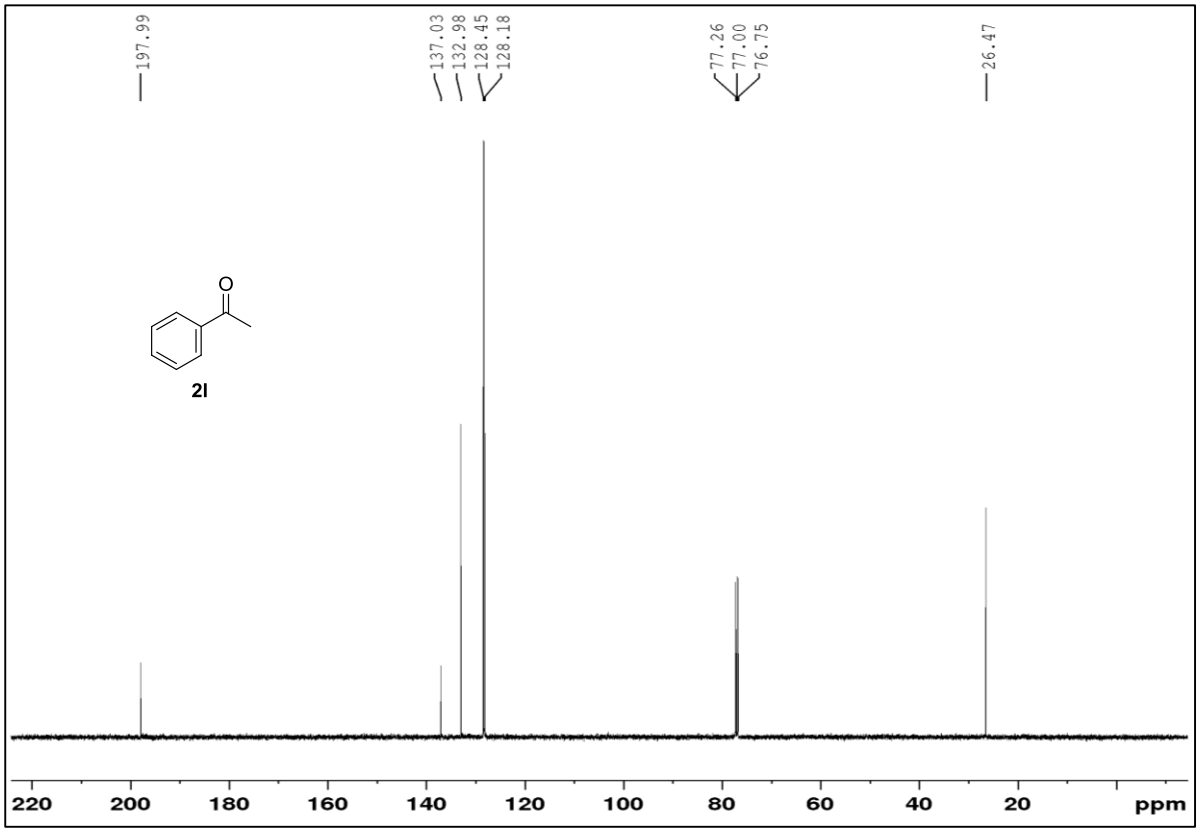
Benzophenone (**2k**). White solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.1 Hz, 4H), 7.59 (t, *J* = 7.4 Hz, 2H), 7.48 (t, *J* = 7.7 Hz, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 196.7, 137.6, 132.4, 130.0, 128.2. HRMS (EI): calcd for $C_{13}H_{10}O$ 182.0732, found 182.0732.



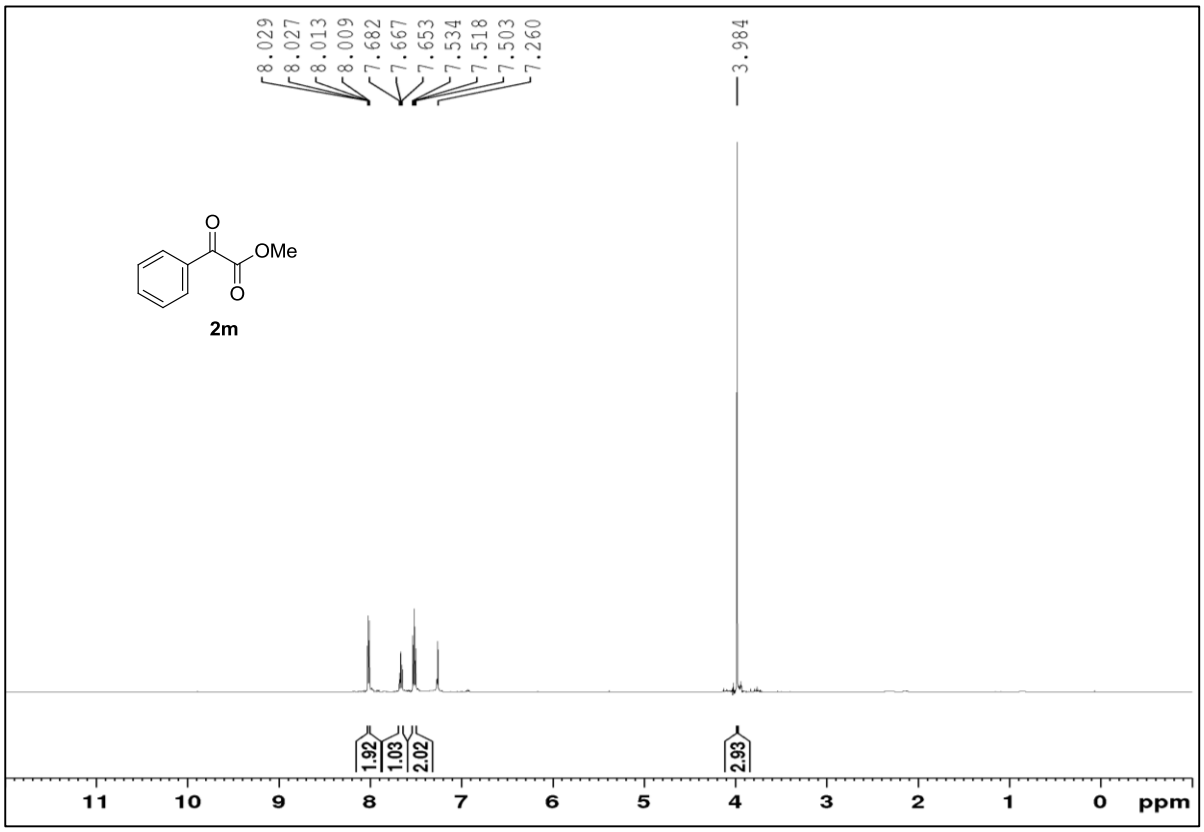


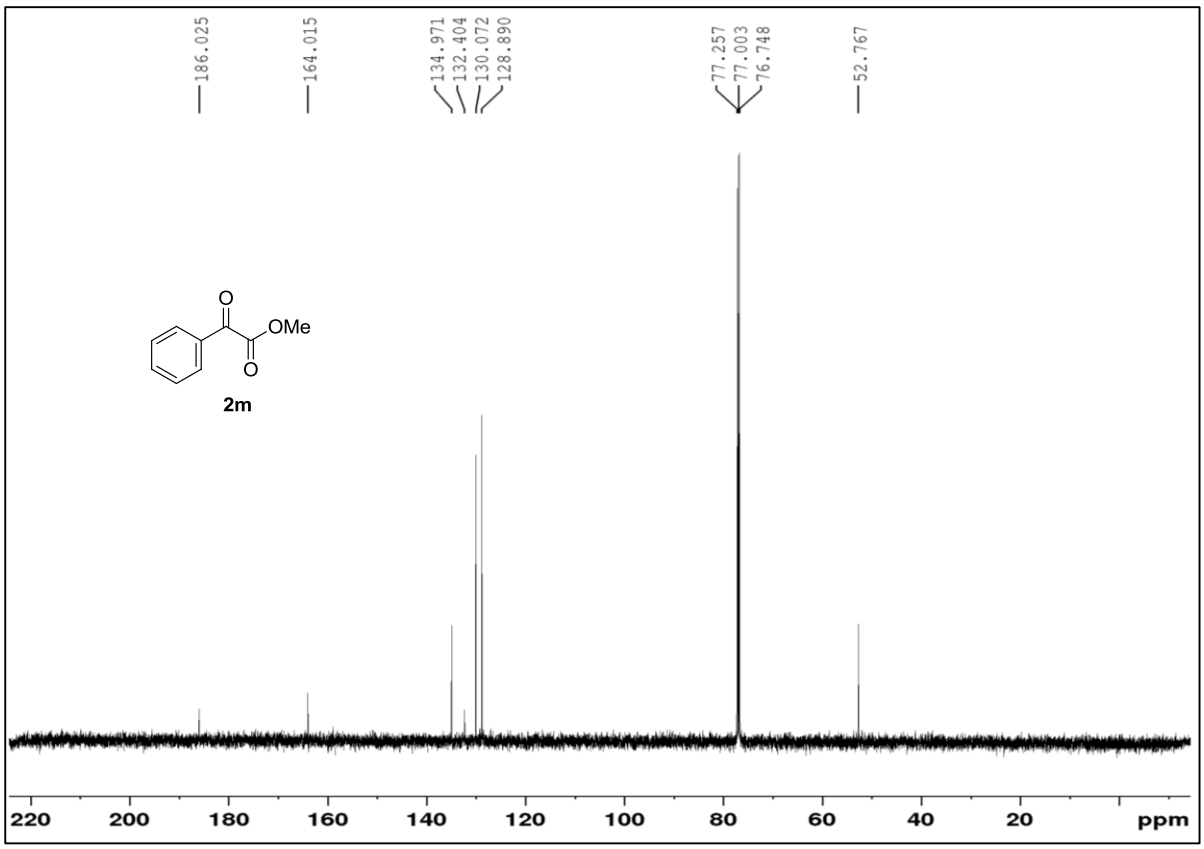
Acetophenone (**2l**). Colorless liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 2.58 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 198.0, 137.0, 133.0, 128.5, 128.2, 26.5. HRMS (EI): calcd for C_8H_8O 120.0575, found 120.0577.



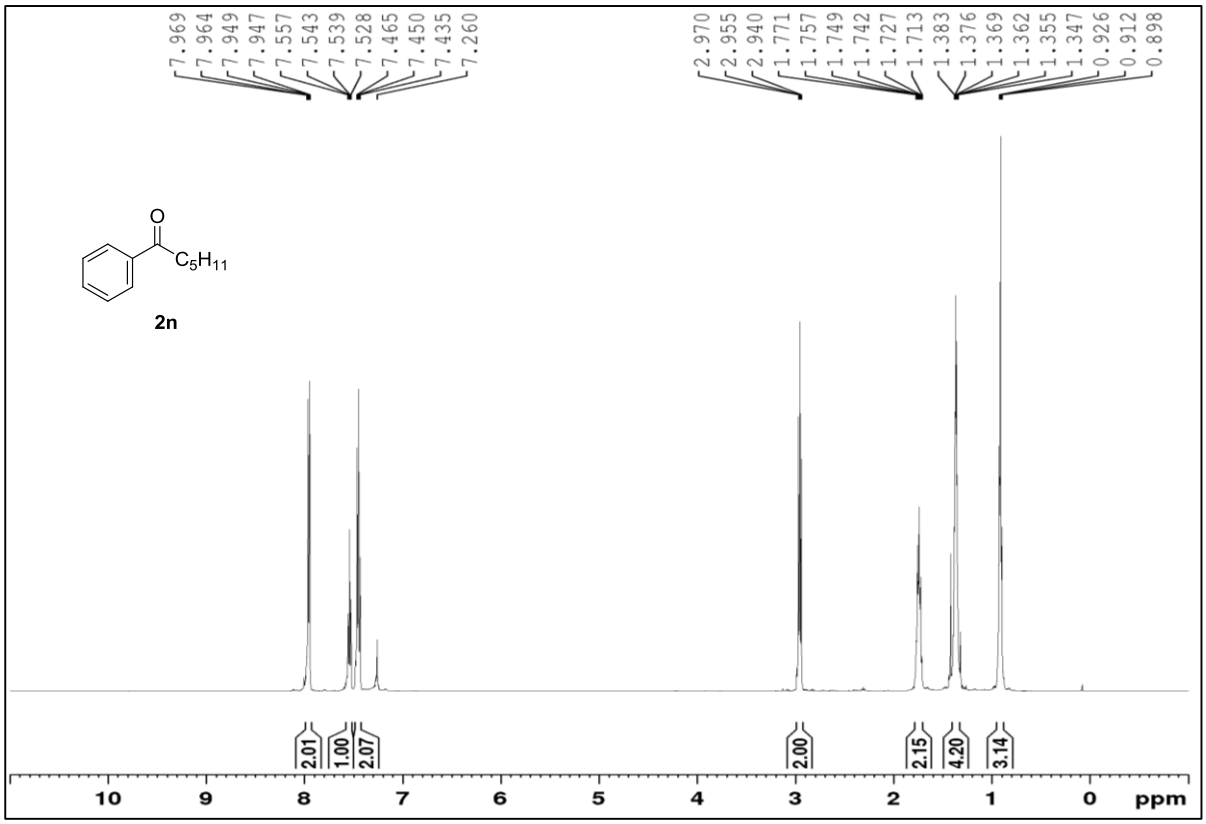


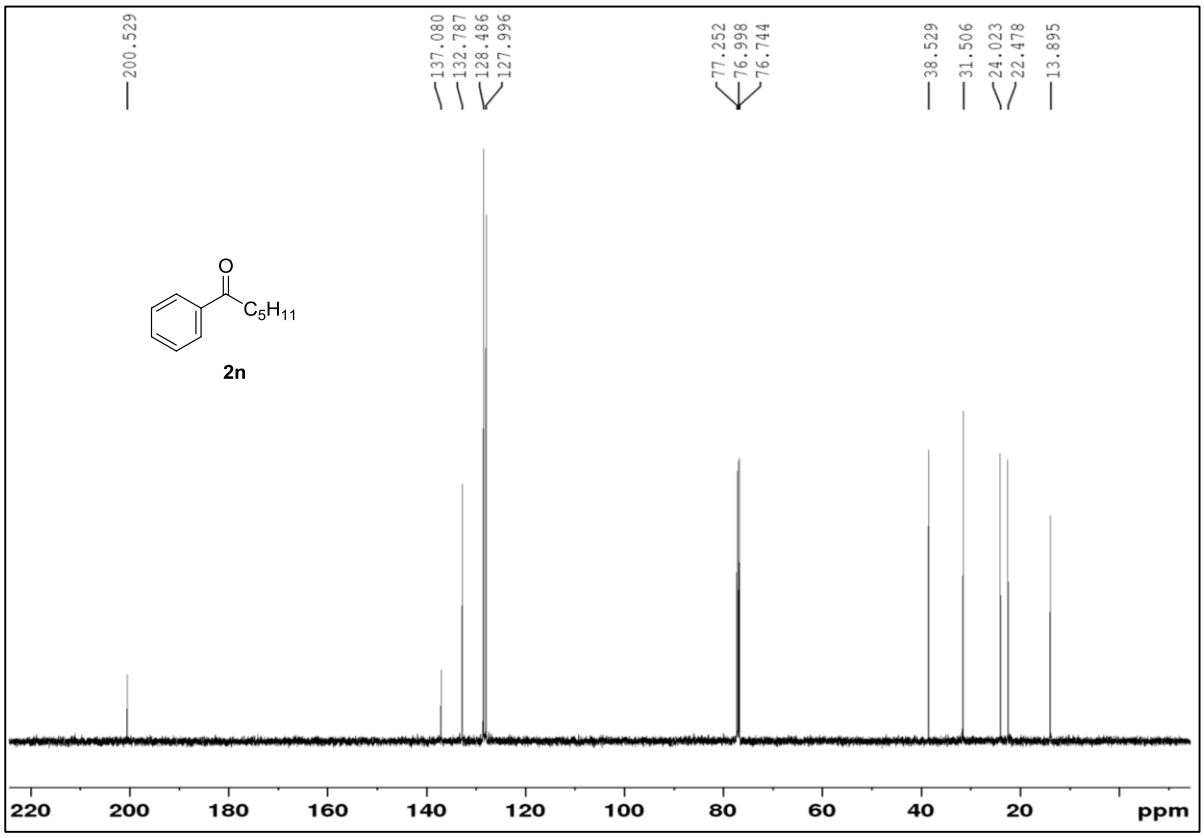
Methyl 2-oxo-2-phenylacetate (**2m**). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.67 (t, *J* = 7.5 Hz, 1H), 7.52 (t, *J* = 7.9 Hz, 2H), 3.98 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 186.0, 164.0, 135.0, 132.4, 130.1, 128.9, 52.8. HRMS (EI): calcd for $C_9H_8O_3$ 164.0473, found 164.0481.



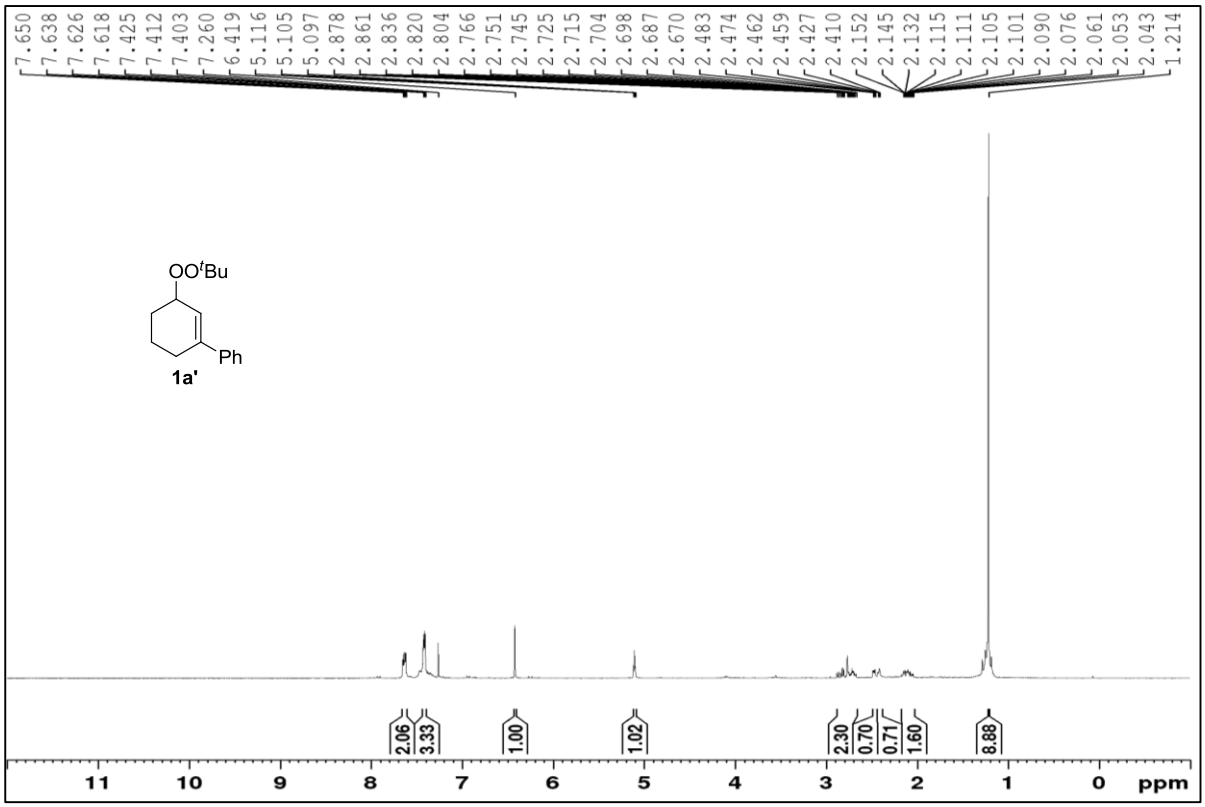


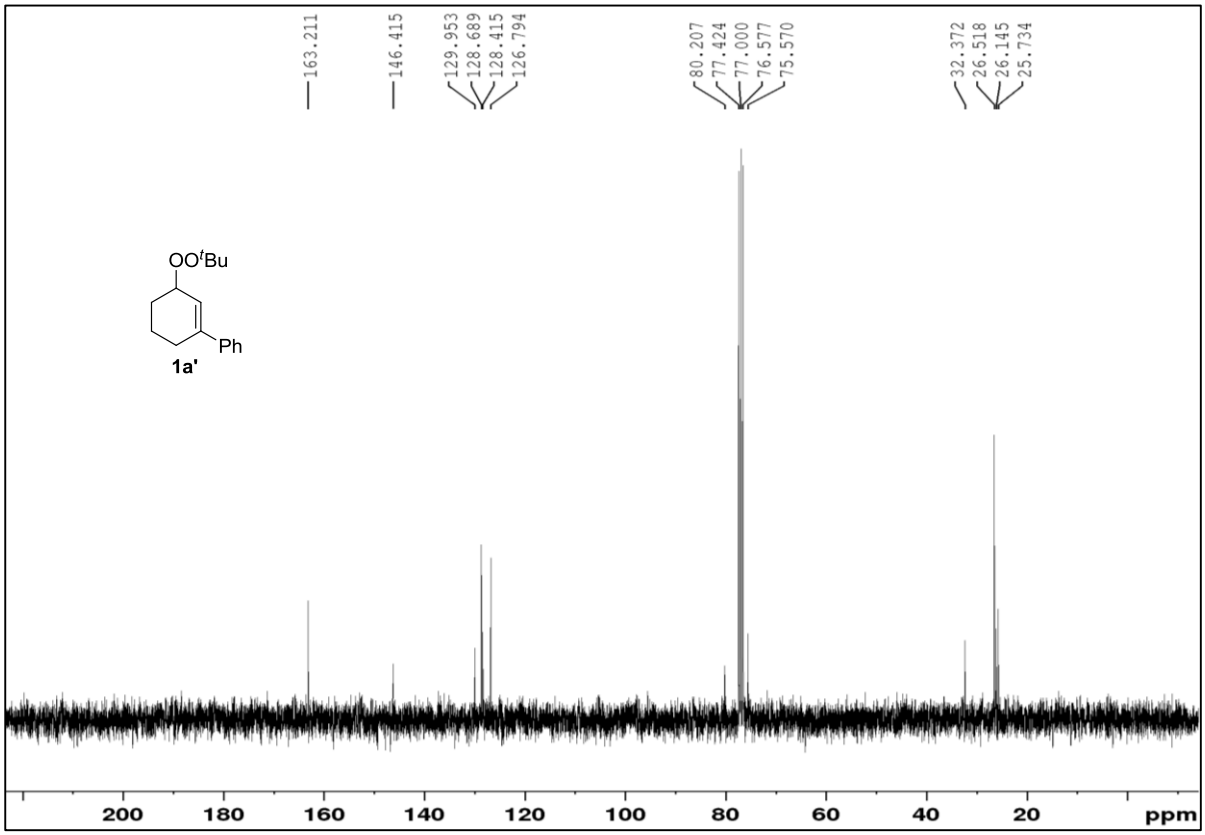
1-Phenylhexan-1-one (**2n**). Pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.55 (d, *J* = 7.3 Hz, 2H), 7.45 (t, *J* = 7.7 Hz, 2H), 2.99 – 2.92 (m, 2H), 1.74 (dq, *J* = 14.8, 7.4 Hz, 2H), 1.37 (td, *J* = 7.2, 3.6 Hz, 4H), 0.91 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 200.5, 137.1, 132.8, 128.5, 128.0, 38.5, 31.5, 24.0, 22.5, 13.9. HRMS (EI): calcd for $C_{12}H_{16}O$ 176.1201, found 176.1206.



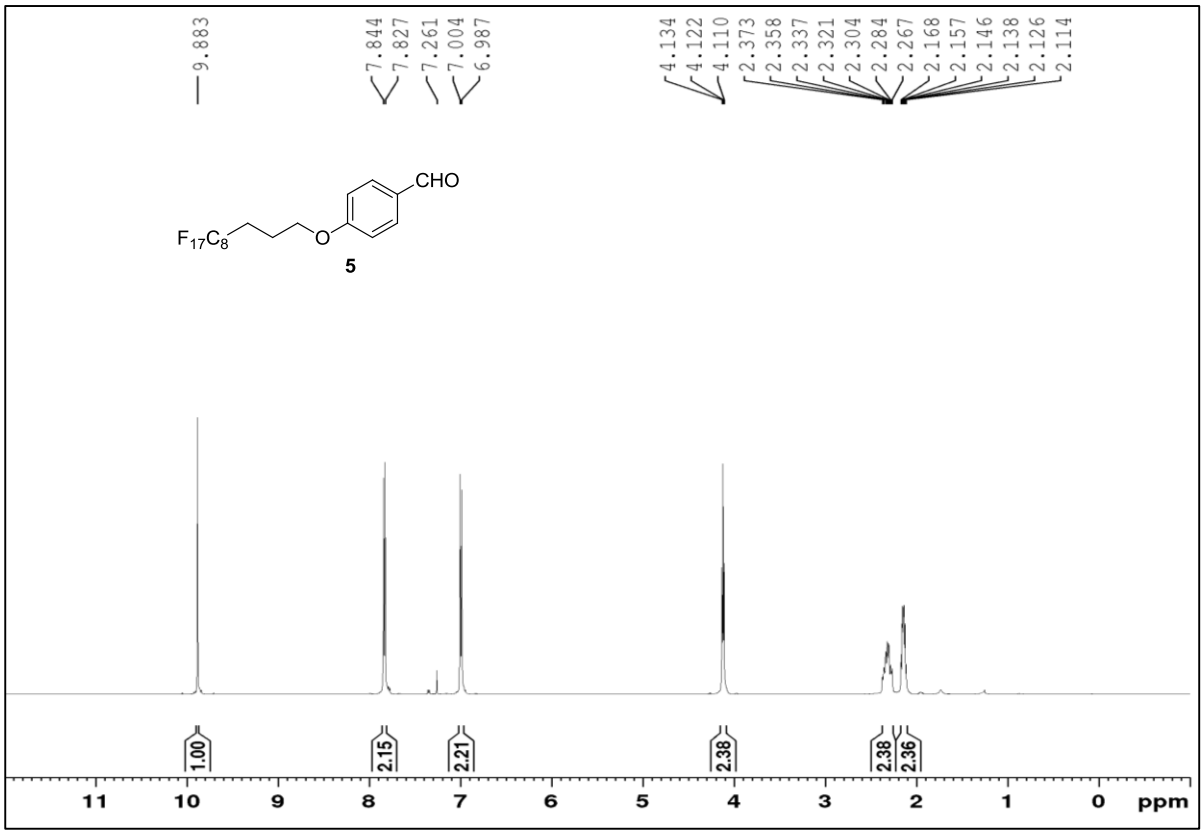


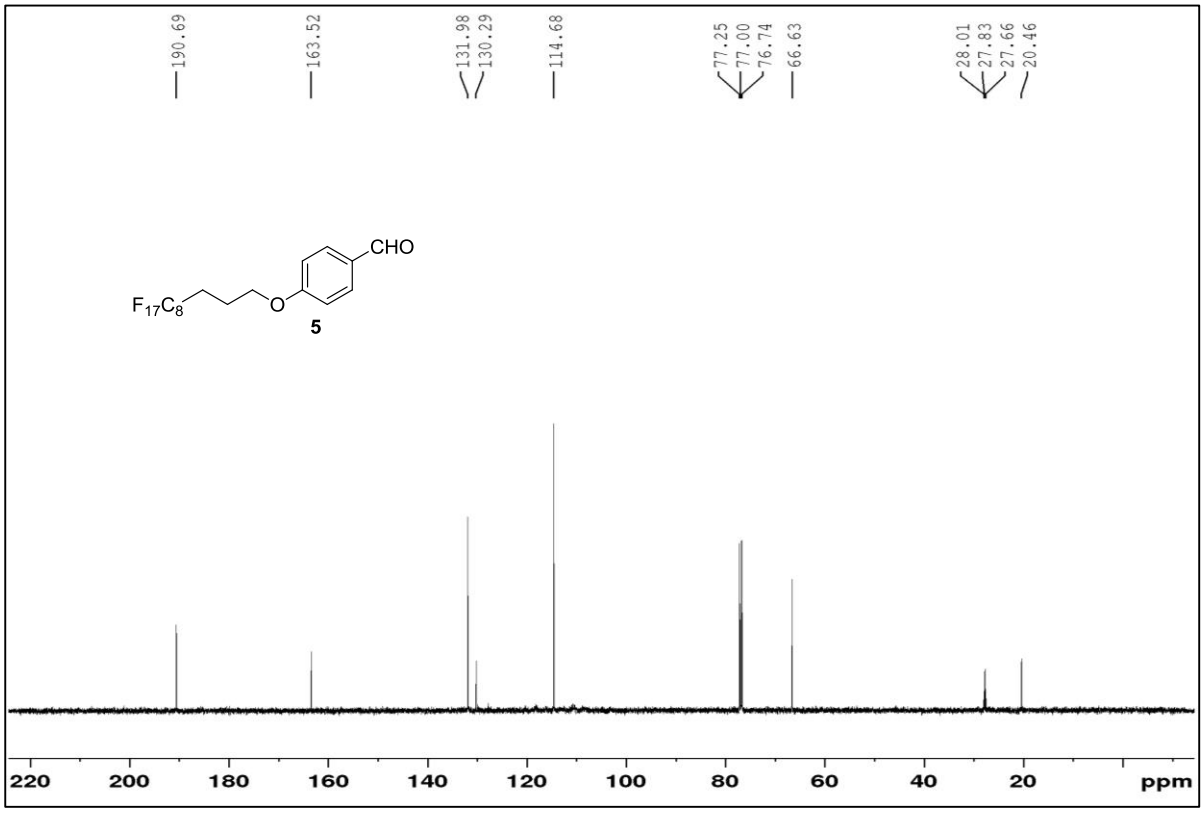
3-t-Butylperoxy-1-phenylcyclohexene (**1a'**). Pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.66 – 7.60 (m, 2H), 7.44 – 7.39 (m, 3H), 6.42 (s, 1H), 5.11 (t, *J* = 2.8 Hz, 1H), 2.89 – 2.66 (m, 2H), 2.47 (dd, *J* = 5.6, 1.8 Hz, 1H), 2.42 (d, *J* = 4.8 Hz, 1H), 2.17 – 2.03 (m, 2H), 1.21 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.2, 146.4, 130.0, 128.7, 128.4, 126.8, 80.2, 75.6, 32.4, 26.5, 26.1, 25.7.



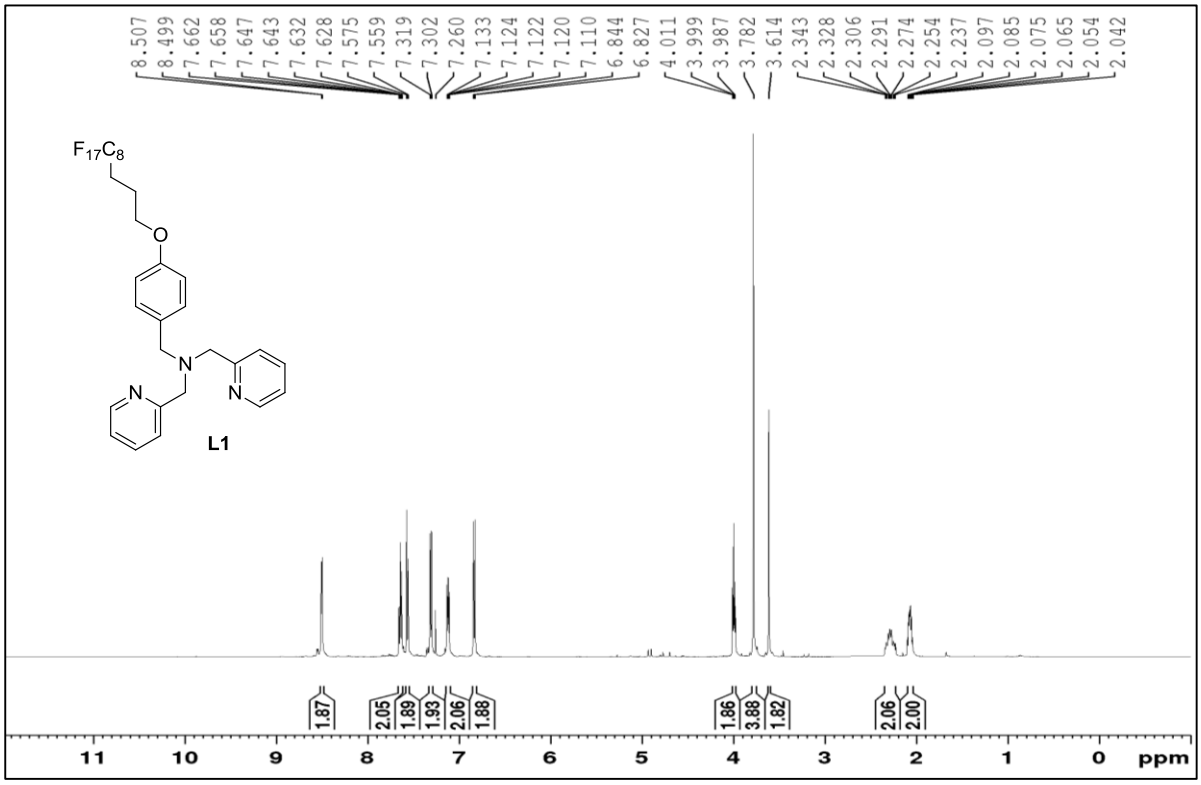


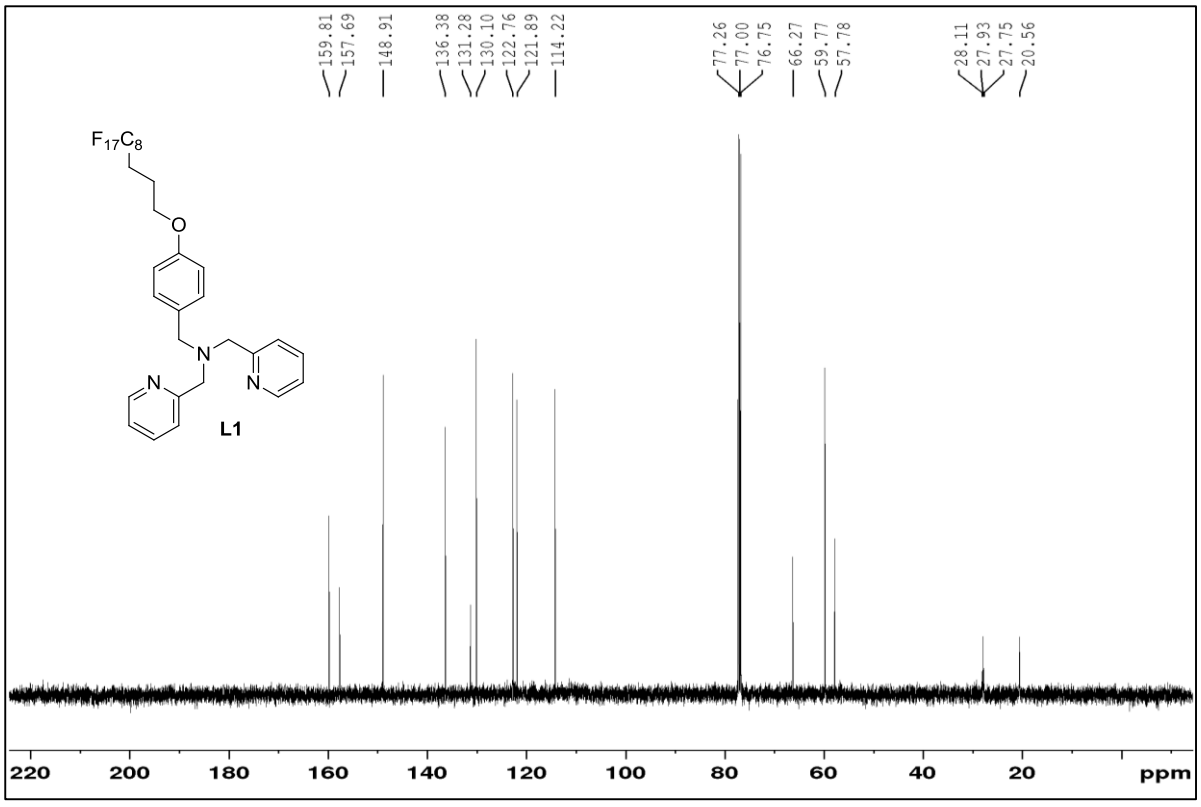
4-(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoroundecyloxy)benzaldehyde (**5**). White solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.88 (s, 1H), 7.83 (d, *J* = 8.7 Hz, 2H), 6.99 (d, *J* = 8.7 Hz, 2H), 4.12 (t, *J* = 5.9 Hz, 2H), 2.38 – 2.25 (m, 2H), 2.14 (dq, *J* = 12.0, 6.0 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 190.7, 163.5, 132.0, 130.3, 114.7, 66.6, 27.8 (t, *J* = 22.5 Hz), 20.5. HRMS (ESI): calcd for $C_{18}H_{12}F_{17}O_2$ (M + H) 583.0560, found 583.0560.



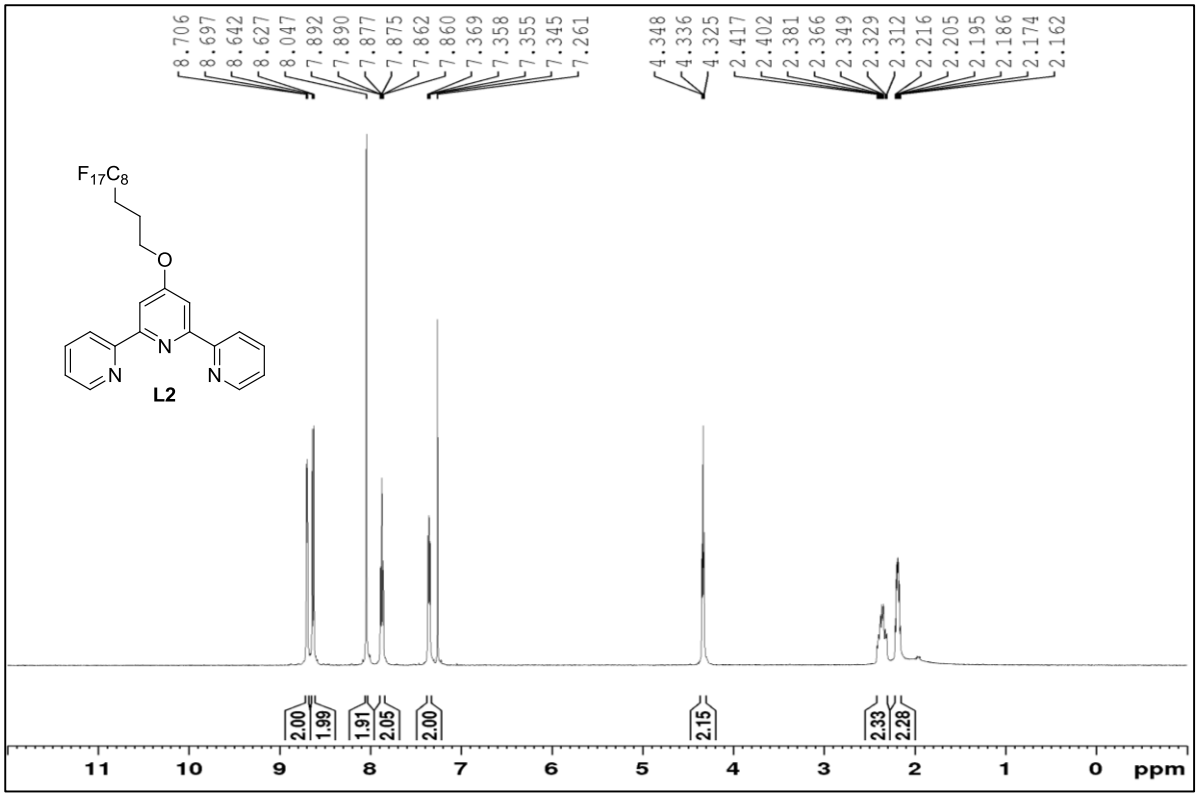


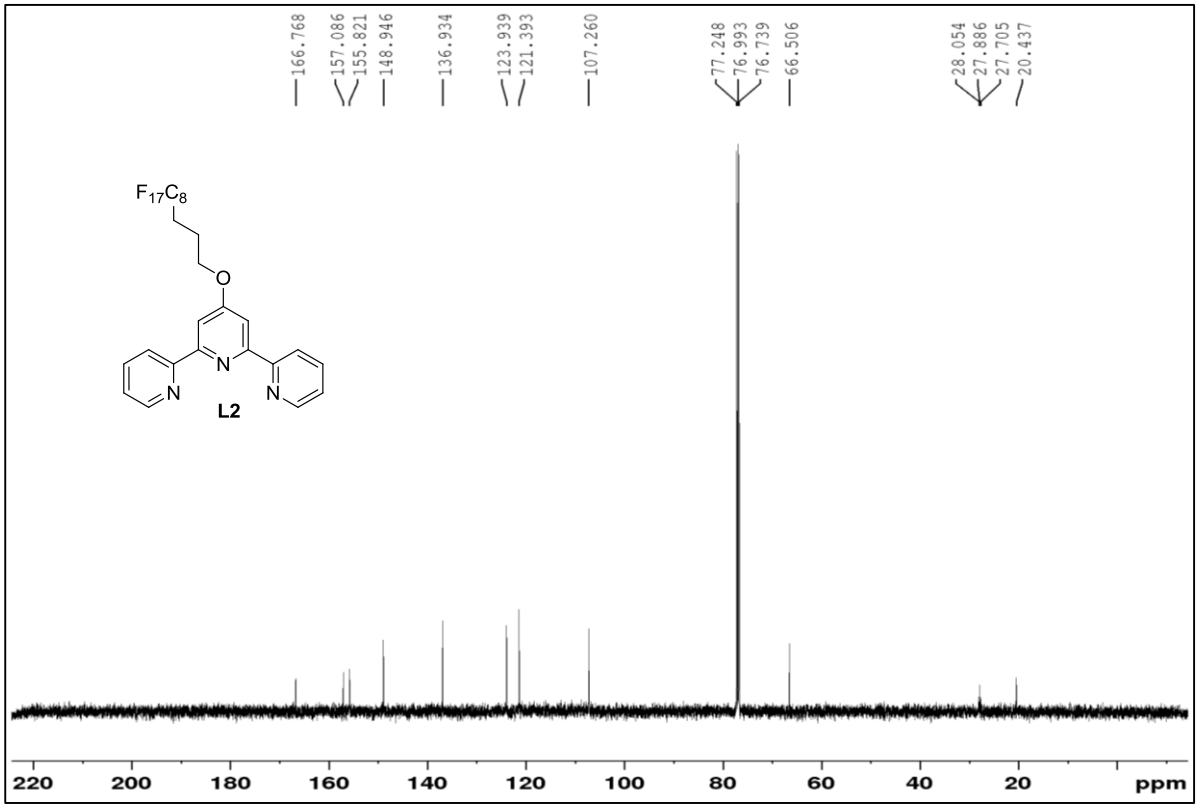
N-(4-(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-heptadecafluoroundecyloxy)benzyl)-1-(pyridin-2-yl)-N-(pyridin-2-ylmethyl)methanamine (**L1**). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.7 Hz, 2H), 7.65 (td, *J* = 7.6, 1.7 Hz, 2H), 7.57 (d, *J* = 7.8 Hz, 2H), 7.31 (d, *J* = 8.6 Hz, 2H), 7.15 – 7.10 (m, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.00 (t, *J* = 5.9 Hz, 2H), 3.78 (s, 4H), 3.61 (s, 2H), 2.35 – 2.22 (m, 2H), 2.07 (td, *J* = 11.4, 5.9 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.8, 157.7, 148.9, 136.4, 131.3, 130.1, 122.8, 121.9, 114.2, 66.3, 59.8, 57.8, 27.9 (t, *J* = 22.5 Hz), 20.6. HRMS (ESI): calcd for $C_{30}H_{25}F_{17}N_3O$ (M + H) 766.1721, found 766.1729.





4-(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoroundecyloxy)-2,6-di(2-pyridyl)pyridine (**L2**). White solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.70 (d, *J* = 4.5 Hz, 2H), 8.63 (d, *J* = 7.9 Hz, 2H), 8.05 (s, 2H), 7.88 (td, *J* = 7.8, 1.4 Hz, 2H), 7.36 (dd, *J* = 6.8, 5.2 Hz, 2H), 4.34 (t, *J* = 5.8 Hz, 2H), 2.43 – 2.30 (m, 2H), 2.19 (dt, *J* = 16.4, 5.8 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.8, 157.1, 155.8, 148.9, 136.9, 123.9, 121.4, 107.3, 66.5, 27.9 (t, *J* = 21.0 Hz), 20.4. HRMS (ESI): calcd for $C_{26}H_{16}F_{17}N_3ONa$ (M + Na) 732.0914, found 732.0922.





2,2'-(4-(4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoroundecyloxy)pyridine-2,6-diyl)bis(1H-benzo[d]imidazole) (**L3**). Off-white solid. ^{1}H NMR (500 MHz, DMSO) δ 7.86 (s, 2H), 7.75 (d, *J* = 8.6 Hz, 4H), 7.32 (s, 4H), 4.43 (t, *J* = 6.1 Hz, 2H), 2.89 (s, 2H), 2.73 (s, 2H). HRMS (ESI): calcd for $C_{30}H_{17}F_{17}N_5O$ (M – H) 786.1167, found 786.1159.