Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Electrochemical synthesis of 7-membered carbocycles through cascade 5-*exo*/7-*endo* radical cyclization

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1. General Information

Anhydrous MeOH was purchased from Thermo Fisher, and tetrahydrofuran were obtained by distillation under argon from sodium/benzophenone, respectively. Other solvents and commercially available reagents were used without purification. Flash column chromatography was performed with silica gel (200–300 mesh). NMR spectra were recorded on Bruker AV-500, Bruker AV-600 and Bruker AV-850 instruments. Data were reported as chemical shifts in ppm relative to TMS (0.00 ppm) for ^{1}H and $CDCl_3$ (77.2 ppm) for ^{13}C. The abbreviations used for explaining the multiplicities were as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. High resolution mass spectra (ESI HRMS) were recorded on a Micromass QTOF2 Quadruple/Time-of-Flight Tandem mass spectrometer by the instrumentation center of Department of Chemistry, Xiamen University. Cyclic voltammograms were obtained on a CHI 760E potentiostat. Infrared spectra (IR) were recorded on a Nicolet AVATER FTIR330 spectrometer.

2. General Procedure for the Electrolysis

A 10 mL three-necked round-bottomed flask was charged with the substrate (0.2 mmol, 1.0 equiv), Cp_2Fe (0.01 mmol, 0.05 equiv), nBu$_4$NBF$_4$ (0.6 mmol, 3.0 equiv), and Na_2CO_3 (0.2 mmol, 1.0 equiv). The flask was equipped with a condenser, a reticulated vitreous carbon (100 PPI) anode (1.2 cm x 1.0 cm x 1.0 cm) and a platinum plate (1 cm x 1 cm) cathode. The flask was flushed with argon. 1,4-Cyclohexadiene (1.0 mmol, 5.0 equiv), THF (5.0 mL) and MeOH (1.0 mL) were added. The constant current (5.0 mA) electrolysis was carried out at reflux (oil bath temperature, 80 °C) until complete consumption of the substrate (monitored by TLC or ^{1}H NMR). The reaction mixture was cooled to rt and concentrated under reduced pressure. The residue was chromatographed through silica gel eluting with ethyl acetate/hexanes to give the desired product.

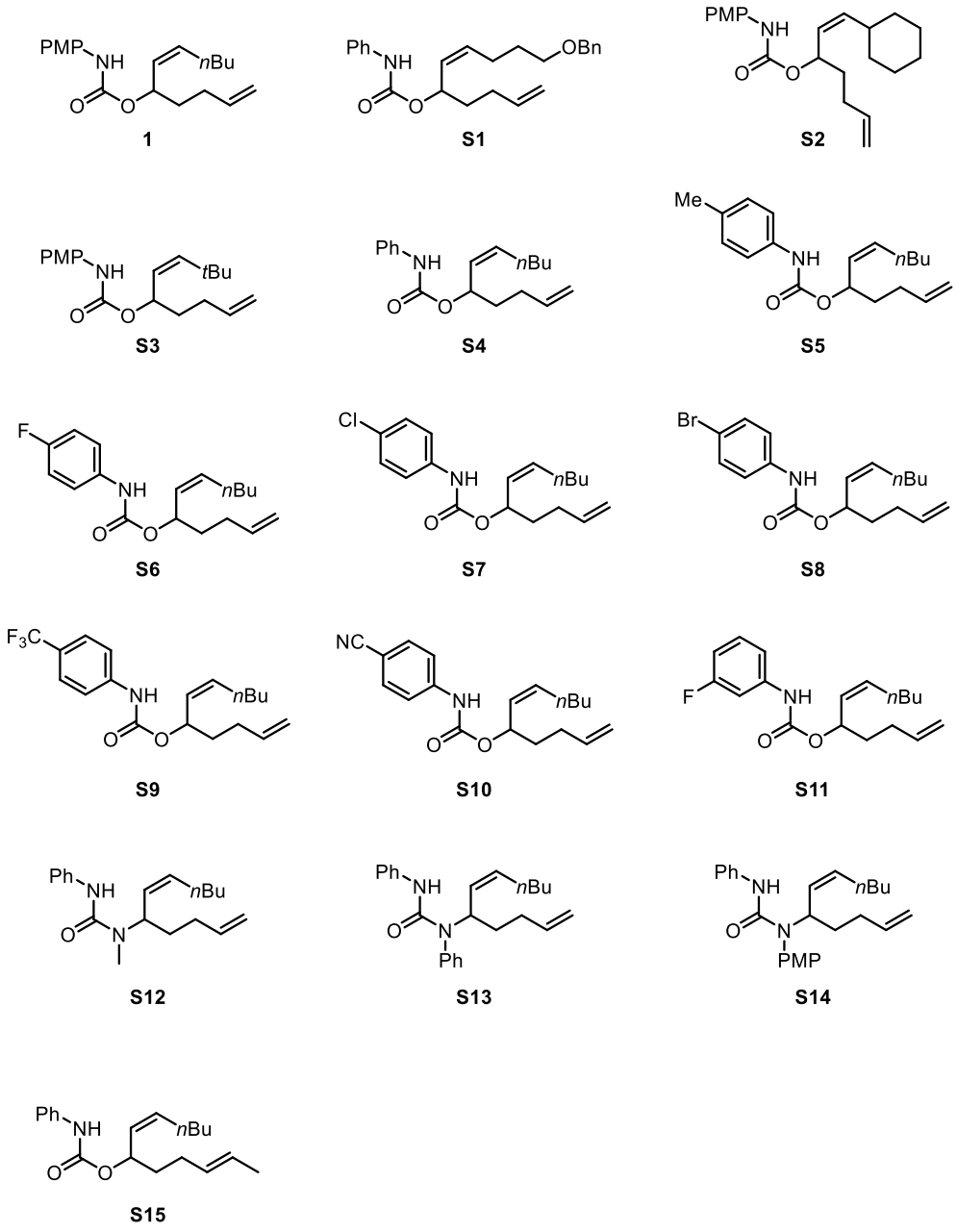
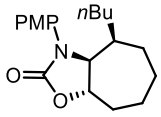


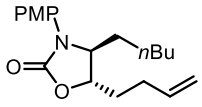
Fig. S1 Substrates employed for the electrolysis.

3. Characterization Data for Electrolysis Products

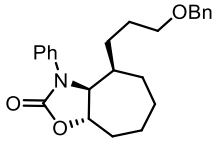


4-Butyl-3-(4-methoxyphenyl)octahydro-2*H*-cyclohepta[*d*]oxazol-2-one (2). The title compound was obtained as a yellow oil in 52% yield (33 mg) starting from **1** (63 mg, 0.2 mmol) by following the General Procedure A. Electricity = 1.9 F mol^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.22–7.17 (m, 2H), 6.94–6.89 (m, 2H), 4.43 (td, *J* = 10.1,

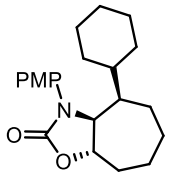
4.2 Hz, 1H), 4.24 (dd, *J* = 10.1, 4.9 Hz, 1H), 3.80 (s, 3H), 2.40–2.30 (m, 1H), 2.03–1.89 (m, 2H), 1.87–1.79 (m, 1H), 1.76–1.70 (m, 1H), 1.65–1.46 (m, 3H), 1.43–1.36 (m, 1H), 1.34–1.30 (m, 1H), 1.25–1.20 (m, 1H), 1.15–0.98 (m, 3H), 0.91–0.84 (m, 1H), 0.73 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.5, 157.3, 130.0, 124.6, 114.5, 74.9, 65.3, 55.6, 32.7, 32.2, 31.7, 29.1, 28.0, 25.7, 22.6, 22.4, 13.9. IR (neat, cm^{-1}): 2928, 1703, 1518, 1221, 1033, 828. ESI HRMS *m/z* $(M+Na)^+$ calcd 340.1883, obsd 340.1878.



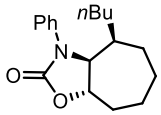
5-(But-3-en-1-yl)-3-(4-methoxyphenyl)-4-pentyloxazolidin-2-one (3). The title compound was obtained as a yellow oil in 9% yield (6 mg) starting from **1** (63 mg, 0.2 mmol) by following the General Procedure A. Electricity = 1.9 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31–7.28 (m, 2H), 6.93–6.89 (m, 2H), 5.84 (ddt, *J* = 16.9, 10.1, 6.6 Hz, 1H), 5.11 (dd, *J* = 17.1, 1.7 Hz, 1H), 5.05 (dd, *J* = 10.1, 1.6 Hz, 1H), 4.30–4.25 (m, 1H), 3.91–3.86 (m, 1H), 3.81 (s, 3H), 2.37–2.22 (m, 2H), 1.96–1.88 (m, 1H), 1.82–1.76 (m, 1H), 1.31–1.18 (m, 8H), 0.84 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.6, 155.9, 137.0, 129.8, 124.7, 116.1, 114.7, 77.9, 62.4, 55.7, 35.0, 32.2, 31.7, 29.1, 23.8, 22.6, 14.1. IR (neat, cm^{-1}): 2978, 1728, 1210, 1053, 734. ESI HRMS *m/z* $(M+Na)^+$ calcd 340.1883, obsd 340.1874.



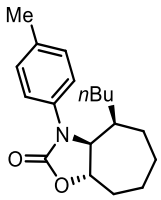
4-(3-(Benzyloxy)propyl)-3-phenyloctahydro-2*H*-cyclohepta[*d*]oxazol-2-one (4). The title compound was obtained as a yellow oil in 52% yield (38 mg) starting from **S1** (73 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.0 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.21 (m, 9H), 7.20–7.13 (m, 1H), 4.43 (td, *J* = 10.6, 4.0 Hz, 1H), 4.36–4.29 (m, 3H), 3.24–3.14 (m, 2H), 2.41–2.29 (m, 1H), 2.18–2.07 (m, 1H), 2.05–1.96 (m, 1H), 1.90–1.81 (m, 1H), 1.77–1.62 (m, 2H), 1.60–1.48 (m, 3H), 1.46–1.38 (m, 1H), 1.33–1.24 (m, 1H), 1.20–1.09 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.8, 138.5, 137.0, 129.1, 128.5, 127.7, 127.6, 125.6, 122.7, 74.8, 72.9, 70.3, 64.6, 32.5, 32.4, 31.7, 27.1, 25.8, 25.4, 22.4. IR (neat, cm^{-1}): 2927, 1738, 1385, 1133, 1070, 758. ESI HRMS *m/z* $(M+Na)^+$ calcd 402.2040, obsd 402.2037.



4-Cyclohexyl-3-(4-methoxyphenyl)octahydro-2*H*-cyclohepta[*d*]oxazol-2-one (5). The title compound was obtained as a yellow oil in 47% yield (32 mg) starting from **S2** (68 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.0 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25–7.21 (m, 2H), 6.94–6.88 (m, 2H), 4.59 (td, *J* = 10.1, 3.2 Hz, 1H), 4.38 (dd, *J* = 10.1, 6.7 Hz, 1H), 3.81 (s, 3H), 2.39–2.29 (m, 1H), 2.07–2.01 (m, 1H), 1.99–1.85 (m, 2H), 1.75–1.61 (m, 6H), 1.47–1.33 (m, 4H), 1.18–1.07 (m, 3H), 1.04–0.97 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.3, 157.2, 131.0, 123.4, 114.5, 76.0, 65.1, 55.6, 37.8, 37.4, 34.5, 31.9, 28.3, 27.1, 26.8, 26.6 (2C), 26.5, 23.7. IR (neat, cm^{-1}): 2925, 1751, 1512, 1247, 1136, 829. ESI HRMS *m/z* $(M+Na)^+$ calcd 366.2040, obsd 366.2039.

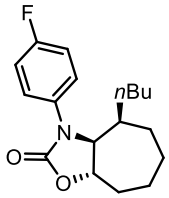


4-Butyl-3-phenyloctahydro-2*H*-cyclohepta[*d*]oxazol-2-one (7). The title compound was obtained as a yellow oil in 75% yield (43 mg) starting from **S4** (58 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.4 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41–7.34 (m, 2H), 7.32–7.26 (m, 2H), 7.21–7.14 (m, 1H), 4.44 (td, *J* = 10.1, 3.9 Hz, 1H), 4.34 (dd, *J* = 10.1, 5.2 Hz, 1H), 2.39–2.30 (m, 1H), 2.15–2.04 (m, 1H), 2.05–1.94 (m, 1H), 1.92–1.82 (m, 1H), 1.79–1.69 (m, 1H), 1.71–1.61 (m, 1H), 1.61–1.46 (m, 2H), 1.23–1.16 (m, 3H), 1.13–0.95 (m, 3H), 0.90–0.82 (m, 1H), 0.68 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.9, 137.1, 129.1, 125.5, 122.7, 74.9, 64.8, 32.5, 32.4, 31.8, 28.9, 28.1, 25.9, 22.5 (2C), 13.9. IR (neat, cm^{-1}): 2930, 1754, 1385, 1131, 751. ESI HRMS *m/z* $(M+Na)^+$ calcd 310.1778, obsd 310.1775.

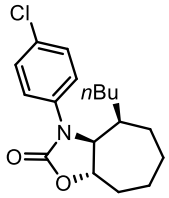


4-Butyl-3-(*p*-tolyl)octahydro-2*H*-cyclohepta[*d*]oxazol-2-one (8). The title compound was obtained as a yellow oil in 64% yield (38 mg) starting from **S5** (60 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.0 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.19–7.15 (m, 4H), 4.43 (td, *J* = 10.1, 4.1 Hz, 1H), 4.29 (dd, *J* = 10.1, 5.1 Hz,

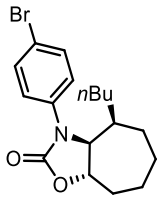
1H), 2.38–2.30 (m, 4H), 2.09–2.02 (m, 1H), 2.00–1.93 (m, 1H), 1.90–1.82 (m, 1H), 1.79–1.71 (m, 1H), 1.58–1.45 (m, 2H), 1.39–1.32 (m, 1H), 1.30–1.19 (m, 3H), 1.14–1.07 (m, 1H), 1.06–0.98 (m, 2H), 0.93–0.81 (m, 1H), 0.71 (t, J = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.1, 135.4, 134.5, 129.7, 122.8, 74.9, 65.0, 32.5, 32.4, 31.7, 29.0, 28.1, 25.8, 22.6, 22.4, 21.1, 13.9. IR (neat, cm^{-1}): 2928, 1753, 1515, 1134, 1016, 652. ESI HRMS *m/z* $(M+Na)^+$ calcd 324.1934, obsd 324.1930.



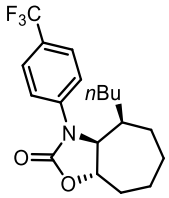
4-Butyl-3-(4-fluorophenyl)octahydro-2*H*-cyclohepta[*d*]oxazol-2-one (9). The title compound was obtained as a yellow oil in 58% yield (35 mg) starting from **S6** (61 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.0 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22–7.15 (m, 2H), 7.04–6.97 (m, 2H), 4.36 (td, J = 10.1, 4.0 Hz, 1H), 4.21 (dd, J = 10.1, 5.0 Hz, 1H), 2.31–2.23 (m, 1H), 1.99–1.88 (m, 2H), 1.82–1.74 (m, 1H), 1.68–1.63 (m, 1H), 1.60–1.55 (m, 1H), 1.52–1.37 (m, 2H), 1.27–1.13 (m, 3H), 1.06–0.93 (m, 3H), 0.84–0.75 (m, 1H), 0.64 (t, J = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.3 (d, $J_{C\text{-}F}$ = 245.1 Hz), 156.9, 133.1 (d, $J_{C\text{-}F}$ = 3.0 Hz), 124.5 (d, $J_{C\text{-}F}$ = 8.3 Hz) 116.0 (d, $J_{C\text{-}F}$ = 22.7 Hz), 75.0, 65.0, 32.4, 32.3, 31.7, 28.9, 28.0, 25.8, 22.5, 22.4, 13.9. ^{19}F NMR (471 MHz, $CDCl_3$) δ −116.6. IR (neat, cm^{-1}): 2930, 1720, 1511, 1138, 834. ESI HRMS *m/z* $(M+Na)^+$ calcd 328.1683, obsd 328.1681.



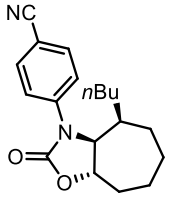
4-Butyl-3-(4-chlorophenyl)octahydro-2*H*-cyclohepta[*d*]oxazol-2-one (10). The title compound was obtained as a yellow oil in 71% yield (43 mg) starting from **S7** (60 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.3 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37–7.33 (m, 2H), 7.28–7.22 (m, 2H), 4.43 (td, J = 10.0, 3.9 Hz, 1H), 4.30 (dd, J = 10.0, 5.2 Hz, 1H), 2.40–2.28 (m, 1H), 2.13–1.97 (m, 2H), 1.92–1.80 (m, 1H), 1.77–1.68 (m, 1H), 1.61–1.44 (m, 2H), 1.30–1.16 (m, 4H), 1.14–0.98 (m, 3H), 0.90–0.80 (m, 1H), 0.71 (t, J = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.6, 135.8, 130.8, 129.2, 123.7, 75.0, 64.6, 32.5, 32.3, 31.7, 28.9, 28.1, 25.9, 22.6, 22.4, 13.9. IR (neat, cm^{-1}): 2927, 1752, 1494, 1184, 874. ESI HRMS *m/z* $(M+Na)^+$ calcd 344.1388, obsd 344.1385.



3-(4-Bromophenyl)-4-butyloctahydro-2*H*-cyclohepta[*d*]oxazol-2-one (11). The title compound was obtained as a yellow oil in 67% yield (49 mg) starting from **S8** (73 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.0 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52–7.46 (m, 2H), 7.22–7.16 (m, 2H), 4.43 (td, *J* = 9.9, 3.9 Hz, 1H), 4.30 (dd, *J* = 9.9, 5.2 Hz, 1H), 2.39–2.31 (m, 1H), 2.11–1.97 (m, 2H), 1.92–1.83 (m, 1H), 1.75–1.65 (m, 2H), 1.56–1.49 (m, 2H), 1.30–1.21 (m, 3H), 1.14–1.07 (m, 1H), 1.05–0.97 (m, 2H), 0.91–0.84 (m, 1H), 0.71 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.5, 136.3, 132.2, 124.0, 118.5, 74.9, 64.6, 32.5, 32.3, 31.7, 28.9, 28.1, 25.9, 22.6, 22.4, 13.9. IR (neat, cm^{-1}): 2929, 1728, 1491, 1133, 1075, 756. ESI HRMS *m/z* $(M+Na)^+$ calcd 388.0883, obsd 388.0879.

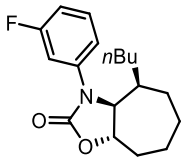


4-Butyl-3-(4-(trifluoromethyl)phenyl)octahydro-2*H*-cyclohepta[*d*]oxazol-2-one (12). The title compound was obtained as a white solid in 69% yield (47 mg) starting from **S9** (71 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.3 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.44 (d, *J* = 8.4 Hz, 2H), 4.46 (td, *J* = 9.9, 3.7 Hz, 1H), 4.38 (dd, *J* = 9.9, 5.3 Hz, 1H), 2.41–2.31 (m, 1H), 2.21–2.11 (m, 1H), 2.11–2.01 (m, 1H), 1.96–1.85 (m, 1H), 1.79–1.68 (m, 2H), 1.60–1.49 (m, 2H), 1.30–1.16 (m, 3H), 1.12–0.95 (m, 3H), 0.90–0.84 (m, 1H), 0.67 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.3, 140.5, 127.1 (q, J_{C-F} = 32.9 Hz), 126.3 (q, J_{C-F} = 3.9 Hz), 124.1 (d, J_{C-F} = 271.7 Hz), 122.0, 75.0, 64.4, 32.6, 32.2, 31.7, 28.7, 28.2, 25.9, 22.5, 22.4, 13.8. ^{19}F NMR (471 MHz, $CDCl_3$) δ –62.3. IR (neat, cm^{-1}): 2983, 1755, 1315, 1125, 840. ESI HRMS *m/z* $(M+Na)^+$ calcd 378.1651, obsd 378.1649.

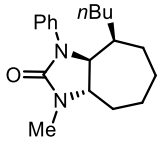


4-Butyl-2-oxohexahydro-2*H*-cyclohepta[*d*]oxazol-3(3a*H*)-yl)benzonitrile (13). The title compound was obtained as a yellow oil in 62% yield (38 mg) starting from **S10**

(62 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.8 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63–7.57 (m, 2H), 7.42–7.35 (m, 2H), 4.39 (td, *J* = 10.5, 3.6 Hz, 1H), 4.31 (dd, *J* = 10.5, 5.3 Hz, 1H), 2.36–2.25 (m, 1H), 2.17–2.07 (m, 1H), 2.05–1.96 (m, 1H), 1.87–1.79 (m, 1H), 1.71–1.60 (m, 2H), 1.52–1.36 (m, 2H), 1.22–1.08 (m, 3H), 1.01–0.89 (m, 3H), 0.82–0.77 (m, 1H), 0.61 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 155.9, 141.5, 133.1, 121.9, 118.7, 108.2, 75.0, 64.2, 32.7, 32.1, 31.6, 28.7, 28.2, 26.0, 22.5, 22.4, 13.8. IR (neat, cm^{-1}): 2931, 2225, 1758, 1382, 1201, 1133, 828. ESI HRMS *m/z* $(M+Na)^+$ calcd 335.1730, obsd 335.1727.

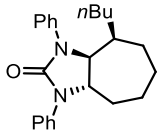


4-Butyl-3-(3-fluorophenyl)octahydro-2*H*-cyclohepta[*d*]oxazol-2-one (14). The title compound was obtained as a yellow oil in 62% yield (38 mg) starting from **S11** (61 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.2 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37–7.30 (m, 1H), 7.13–7.06 (m, 2H), 6.91–6.84 (m, 1H), 4.43 (td, *J* = 10.0, 3.8 Hz, 1H), 4.31 (dd, *J* = 10.0, 5.3 Hz, 1H), 2.40–2.30 (m, 1H), 2.20–2.11 (m, 1H), 2.07–1.99 (m, 1H), 1.92–1.85 (m, 1H), 1.77–1.68 (m, 2H), 1.58–1.46 (m, 2H), 1.29–1.20 (m, 3H), 1.13–0.97 (m, 3H), 0.92–0.84 (m, 1H), 0.70 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.0 (d, $J_{C\text{-}F}$ = 245.9 Hz), 156.4, 138.8 (d, $J_{C\text{-}F}$ = 10.3 Hz), 130.2 (d, $J_{C\text{-}F}$ = 9.3 Hz), 117.7 (d, $J_{C\text{-}F}$ = 3.1 Hz), 112.3 (d, $J_{C\text{-}F}$ = 21.2 Hz), 109.9 (d, $J_{C\text{-}F}$ = 24.8 Hz), 74.9, 64.6, 32.6, 32.2, 31.7, 28.8, 28.1, 25.9, 22.5, 22.4, 13.8. ^{19}F NMR (471 MHz, $CDCl_3$) δ −111.5. IR (neat, cm^{-1}): 2931, 1718, 1610, 1498, 1200, 775. ESI HRMS *m/z* $(M+Na)^+$ calcd 328.1683, obsd 328.1682.

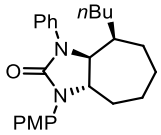


4-Butyl-1-methyl-3-phenyloctahydrocyclohepta[*d*]imidazol-2(1*H*)-one (15). The title compound was obtained as a yellow oil in 40% yield (24 mg) starting from **S12** (60 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.7 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.29 (m, 4H), 7.12–7.07 (m, 1H), 4.11 (dd, *J* = 9.5, 5.2 Hz, 1H), 3.31 (td, *J* = 9.5, 3.4 Hz, 1H), 2.82 (s, 3H), 2.27–2.18 (m, 1H), 2.17–2.09 (m, 1H), 2.02–1.88 (m, 3H), 1.58–1.48 (m, 2H), 1.44–1.36 (m, 1H), 1.14–0.83 (m, 7H), 0.70 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.4, 138.9, 128.7, 124.2, 122.7, 62.6, 56.2, 33.1, 32.4, 32.0, 29.5, 29.1, 28.8, 27.4, 22.9, 22.7, 14.0. IR (neat,

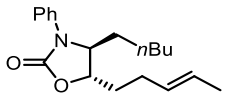
cm^{-1}): 2925, 1707, 1425, 1134, 760. ESI HRMS *m/z* (M+Na)$^+$ calcd 323.2094, obsd 323.2092.



4-Butyl-1,3-diphenyloctahydrocyclohepta[*d*]imidazol-2(1*H*)-one (16). The title compound was obtained as a yellow oil in 58% yield (40 mg) starting from **S13** (70 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.5 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33–7.24 (m, 8H), 7.09–7.03 (m, 2H), 4.28 (dd, *J* = 10.0, 5.1 Hz, 1H), 4.08 (td, *J* = 10.0, 2.9 Hz, 1H), 2.29–2.22 (m, 1H), 2.19–2.11 (m, 1H), 2.06–1.98 (m, 1H), 1.87–1.78 (m, 1H), 1.72–1.64 (m, 1H), 1.55–1.42 (m, 2H), 1.38–1.30 (m, 2H), 1.26–1.21 (m, 1H), 1.17–1.11 (m, 1H), 1.07–0.91 (m, 3H), 0.87–0.79 (m, 1H), 0.62 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.9, 138.5, 138.3, 128.9, 128.8, 124.6 (2C), 122.9 (2C), 62.1, 53.8, 33.1, 32.4, 31.9, 28.9, 28.8, 27.5, 23.0, 22.6, 13.9. IR (neat, cm^{-1}): 2922, 1709, 1400, 1134, 1076, 651. ESI HRMS *m/z* (M+Na)$^+$ calcd 385.2250, obsd 385.2247.



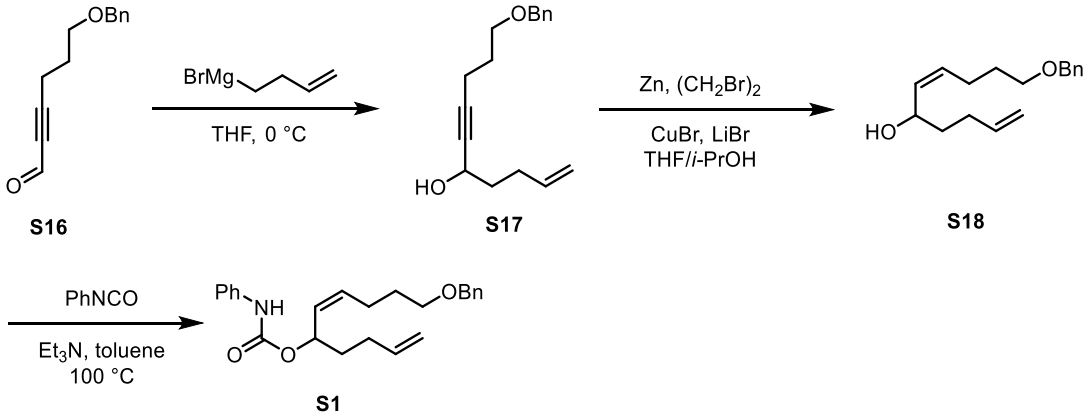
4-Butyl-1-(4-methoxyphenyl)-3-phenyloctahydrocyclohepta[*d*]imidazol-2(1*H*)-one (17). The title compound was obtained as a yellow oil in 52% yield (31 mg) starting from **S14** (61 mg, 0.2 mmol) by following the General Procedure A. Electricity = 2.6 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31–7.25 (m, 4H), 7.20–7.14 (m, 2H), 7.09–7.01 (m, 1H), 6.90–6.80 (m, 2H), 4.26 (dd, *J* = 10.5, 5.2 Hz, 1H), 3.96 (td, *J* = 10.5, 3.1 Hz, 1H), 3.73 (s, 3H), 2.20–2.11 (m, 2H), 2.05–1.95 (m, 1H), 1.85–1.76 (m, 1H), 1.70–1.61 (m, 1H), 1.52–1.44 (m, 1H), 1.41–1.32 (m, 3H), 1.26–1.22 (m, 2H), 1.16–0.93 (m, 3H), 0.89–0.79 (m, 1H), 0.63 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.4, 157.2, 138.5, 131.5, 128.7, 125.3, 124.5, 122.7, 114.3, 62.3, 55.7, 54.7, 33.1, 32.6, 31.9, 28.9 (2C), 27.5, 23.0, 22.6, 13.9. IR (neat, cm^{-1}): 2927, 2360, 1707, 1457, 1136, 638. ESI HRMS *m/z* (M+Na)$^+$ calcd 415.2356, obsd 415.2354.



5-(Pent-3-en-1-yl)-4-pentyl-3-phenyloxazolidin-2-one (18). The title compound was obtained as a yellow oil in 62% yield (36 mg) starting from **S15** (68 mg, 0.2 mmol) by

following the General Procedure A. Electricity = 2.0 F mol^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.33 (m, 2H), 7.34–7.27 (m, 2H), 7.14–7.06 (m, 1H), 5.53–5.42 (m, 1H), 5.37–5.27 (m, 1H), 4.24–4.17 (m, 1H), 3.96–3.89 (m, 1H), 2.21 (q, *J* = 7.4 Hz, 2H), 1.87–1.75 (m, 1H), 1.72–1.61 (m, 1H), 1.62–1.56 (m, 4H), 1.21–1.14 (m, 7H), 0.77 (t, *J* = 6.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 155.4, 137.1, 129.3, 128.5, 125.9, 125.2, 121.9, 78.0, 61.6, 35.6, 31.9, 31.6, 23.7, 22.5, 22.3, 14.0, 13.0. IR (neat, cm^{-1}): 2920, 1688, 1508, 1220, 768. ESI HRMS *m/z* $(M+Na)^+$ calcd 324.1934, obsd 324.1923.

4. Synthesis and Characterization of New Substrates

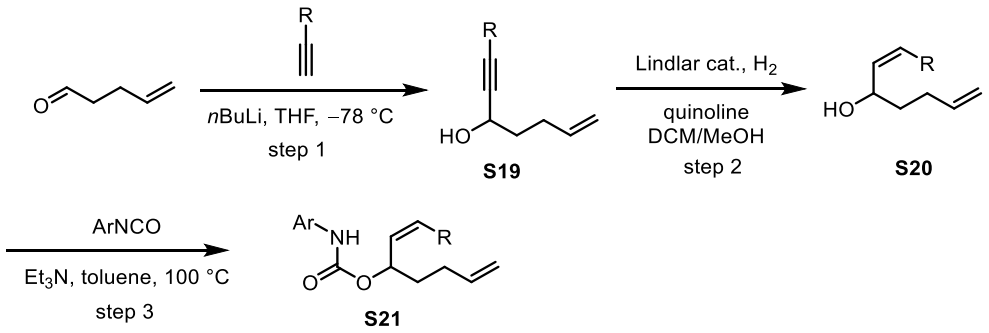


10-(Benzyloxy)dec-1-en-6-yn-5-ol (S17). To a solution of **S16** (1.0 g, 5.0 mmol, 1.0 equiv) in THF (50 mL) at 0 °C was added but-3-en-1-ylmagnesium bromide (0.5 M in THF, 6 mmol, 1.2 equiv) dropwise over 10 min.[1] The reaction mixture was warmed to ambient temperature and stirred for 2 h. Saturated aqueous NH_4Cl (20 mL) was added to quench the reaction. The resulting mixture was extracted three times with EtOAc (3 x 20 mL). The combined organic solution was washed with brine (20 mL), dried over anhydrous $MgSO_4$, filtered, and chromatographed through silica gel eluting with ethyl acetate/hexanes to afford the title compound as a pale yellow oil in 87% yield (1.1 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.24 (m, 5H), 5.88–5.76 (m, 1H), 5.08–5.02 (m, 1H), 5.01–4.94 (m, 1H), 4.51 (s, 2H), 4.37–4.30 (m, 1H), 3.55 (t, *J* = 6.2 Hz, 2H), 2.34 (td, *J* = 7.1, 1.9 Hz, 2H), 2.24–2.14 (m, 2H), 2.00–1.87 (m, 1H), 1.84–1.67 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.6, 138.0, 128.5, 127.8, 127.7, 115.2, 85.1, 81.5, 73.1, 68.8, 62.3, 37.3, 29.6, 28.9, 15.7.

(*Z*)-10-(Benzyloxy)deca-1,6-dien-5-ol (S18). To a suspension of Zn dust (3.2 g, 50 mmol, 25 equiv) in *i*-PrOH (20 mL) was added $(CH_2Br)_2$ (0.82 g, 4.4 mmol, 2.2 equiv). The resulting reaction mixture was heated at reflux for 15 min.[2] A solution of CuBr (0.86 g, 6.0 mmol, 3.0 equiv) and LiBr (0.90 g, 10.4 mmol, 5.3 equiv) in 20 mL of THF was added slowly. The reaction mixture was heated at reflux for an additional 20 min. A solution of **S17** (0.50 g, 2.0 mmol, 1.0 equiv) in *i*-PrOH was added dropwise over 5

min. The reaction mixture was then stirred for 8 h at reflux under Ar. The reaction mixture was filtered through a pad of celite. The filtrate was oncentrated under reduced pressure. The residue was chromatographed through silica gel eluting with ethyl acetate/hexanes to afford the title compound as a yellow oil in 84% yield (0.43 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.31 (m, 4H), 7.30–7.26 (m, 1H), 5.89–5.77 (m, 1H), 5.51–5.38 (m, 2H), 5.06–4.99 (m, 1H), 4.99–4.90 (m, 1H), 4.50 (s, 2H), 4.45 (q, J = 7.2 Hz, 1H), 3.53–3.39 (m, 2H), 2.35–2.24 (m, 1H), 2.20–2.06 (m, 3H), 1.81–1.60 (m, 4H), 1.56–1.49 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.6 (2C), 133.4, 131.7, 128.6, 127.9, 127.8, 114.9, 73.0, 69.4, 67.1, 36.6, 29.9, 29.5, 24.4. IR (neat, cm^{-1}): 3418, 2925, 1717, 1274, 1074, 914, 712. ESI HRMS *m/z* $(M+Na)^+$ calcd 283.1669, obsd 283.1671.

(*Z*)-10-(Benzyloxy)deca-1,6-dien-5-yl phenylcarbamate (S1). To a solution of **S18** (0.35 g, 1.3 mmol, 1.0 equiv) in toluene (15 mL) was added aryl isocyanate (0.19 g, 1.6 mmol, 1.2 equiv) at rt, followed by Et_3N (0.4 mL, 2.6 mmol, 2.0 equiv). The resulting reaction mixture was stirred at 100 °C until complete consumption of the starting material (monitored by TLC). The solvent was removed under reduced pressure. The residue was chromatographed through silica gel eluting with ethyl acetate/hexanes to afford the title compound as a yellow oil in 94% yield (0.46 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.22 (m, 9H), 7.07–7.00 (m, 1H), 6.60 (s, 1H), 5.86–5.75 (m, 1H), 5.63–5.50 (m, 2H), 5.42–5.33 (m, 1H), 5.08–5.00 (m, 1H), 5.00–4.96 (m, 1H), 4.49 (s, 2H), 3.58–3.44 (m, 2H), 2.44–2.19 (m, 2H), 2.17–2.02 (m, 2H), 1.88–1.76 (m, 1H), 1.76–1.67 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.1, 138.7, 138.2, 137.8, 133.9, 129.2, 128.6, 128.5, 127.8, 127.7, 123.4, 118.8, 115.3, 73.1, 71.1, 70.0, 34.3, 29.8, 29.5, 24.9. IR (neat, cm^{-1}): 3328, 2927, 1727, 1217, 1076, 746. ESI HRMS *m/z* $(M+Na)^+$ calcd 402.2040, obsd 402.2031.



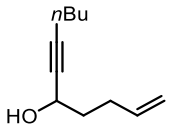
General procedure B

Step 1: To a solution of alkyne (1.2 equiv) in THF (40 mL) at −78 °C was added *n*-butyllithium (2.5 M in hexane, 1.2 equiv) dropwise over 10 min. Upon complete addition, the reaction mixture was stirred for 30 min at −78 °C. Pent-4-enal (1.0 equiv) was added dropwise over 30 min. The reaction mixture was warmed to ambient

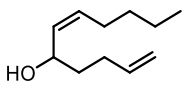
temperature and stirred for 1 h. Saturated aqueous NH_4Cl (20 mL) was added to quench the reaction. The resulting mixture was extracted with EtOAc (3 x 20 mL). The combined organic solution was washed with brine (20 mL), dried over anhydrous $MgSO_4$, and filtered. The filtrate was concentrated under reduced pressure. The residue was chromatographed through silica gel eluting with ethyl acetate/hexanes to give the desired product **S19**.

Step 2: **S19** was dissolved in DCM (12 mL) and MeOH (3 mL) under argon atmosphere. Quinoline (0.4 equiv) and Lindlar catalyst (5% Pd on $CaCO_3$ poisoned with Pb, 0.05 equiv) were added. The reaction mixture was then stirred until complete consumption of the starting material (monitored by ^{1}H NMR) under hydrogen atmosphere (balloon). The reaction mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure. The crude **S20** was used in the following step without purification.[3]

Step 3: To a solution of **S20** (1.0 equiv) in toluene (0.1 M) was added aryl isocyanate (1.2 equiv) at rt, followed by Et_3N (2.0 equiv). The resulting reaction mixture was stirred at 100 °C until complete consumption of the starting material (monitored by TLC). The solvent was removed under reduced pressure. The residue was chromatographed through silica gel eluting with ethyl acetate/hexanes to give the product **S21**.

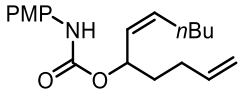


Undec-1-en-6-yn-5-ol (S22). The title compound was obtained as a light yellow oil in 87% yield (4.6 g) starting from 1-hexyne (4.4 mL, 38 mmol, 1.2 equiv) and pent-4-enal (2.7 g, 32 mmol, 1.0 equiv) by following the General Procedure B, step 1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.91–5.76 (m, 1H), 5.11–5.02 (m, 1H), 5.02–4.95 (m, 1H), 4.42–4.31 (m, 1H), 2.28–2.18 (m, 4H), 2.13–2.01 (m, 1H), 1.83–1.69 (m, 2H), 1.54–1.46 (m, 2H), 1.44–1.37 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.0, 115.2, 85.9, 81.2, 62.3, 37.4, 30.9, 29.6, 22.1, 18.5, 13.7. IR (neat, cm^{-1}): 3388, 2936, 2866, 1508, 856. ESI HRMS *m/z* $(M+Na)^+$ calcd 166.1358, obsd 166.1360.

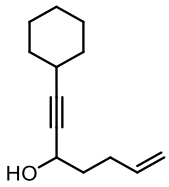


(*Z*)-Undeca-1,6-dien-5-ol (S23). The title compound was obtained as a light yellow oil in 78% yield (0.7 g) starting from **S22** (0.9 g, 38 mmol, 1.2 equiv) by following the General Procedure B, step 2. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.84 (ddt, *J* = 16.9, 10.2, 6.6 Hz, 1H), 5.56–5.45 (m, 1H), 5.42–5.33 (m, 1H), 5.09–5.00 (m, 1H), 4.98 (dd, *J* =

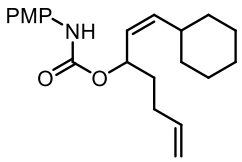
10.2, 1.8 Hz, 1H), 4.50–4.41 (m, 1H), 2.17–2.03 (m, 4H), 1.74–1.64 (m, 1H), 1.58–1.49 (m, 1H), 1.39–1.30 (m, 5H), 0.92–0.87 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.5, 132.6, 132.4, 114.8, 67.3, 36.7, 32.0, 29.8, 27.6, 22.5, 14.1.



(*Z*)-Undeca-1,6-dien-5-yl (4-methoxyphenyl)carbamate (1). The title compound was obtained as a yellow oil in 76% yield (0.70 g) starting from **S23** (0.44 g, 2.9 mmol) following the General Procedure B, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24–7.19 (m, 2H), 6.82–6.73 (m, 2H), 6.41 (s, 1H), 5.83–5.68 (m, 1H), 5.58–5.41 (m, 2H), 5.32–5.21 (m, 1H), 5.07–4.96 (m, 1H), 4.96–4.86 (m, 1H), 3.70 (s, 3H), 2.15–2.07 (m, 2H), 2.07–2.01 (m, 2H), 1.82–1.66 (m, 1H), 1.58–1.51 (m, 1H), 1.37–0.98 (m, 4H), 0.82 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.0, 153.6, 137.9, 134.7, 131.3, 128.1, 120.8, 115.1, 114.4, 71.0, 55.7, 34.4, 31.9, 29.5, 27.8, 22.5, 14.1. IR (neat, cm^{-1}): 3332, 2928, 1704, 1518, 1220, 828. ESI HRMS *m/z* $(M+Na)^+$ calcd 340.1883, obsd 340.1880.

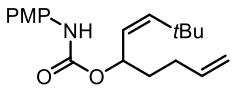


Cyclohexylhept-6-en-1-yn-3-ol (S24). The title compound was obtained as a yellow oil in 64% yield (0.90 g) starting from ethynylcyclohexane (1.0 g, 9.3 mmol, 1.2 equiv) and pent-4-enal (0.68 g, 8.0 mmol, 1.0 equiv) by following the General Procedure B, step1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.92–5.77 (m, 1H), 5.11–5.03 (m, 1H), 5.01–4.94 (m, 1H), 4.44–4.35 (m, 1H), 2.44–2.36 (m, 1H), 2.27–2.20 (m, 2H), 1.86–1.75 (m, 5H), 1.73–1.62 (m, 3H), 1.47–1.37 (m, 2H), 1.36–1.23 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.1, 115.2, 90.1, 81.1, 62.4, 37.5, 32.8, 29.7, 29.2, 26.0, 25.0. IR (neat, cm^{-1}): 3378, 2930, 2855, 1447, 911. ESI HRMS *m/z* $(M+Na)^+$ calcd 215.1406, obsd 215.1406.

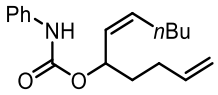


(*Z*)-1-Cyclohexylhepta-1,6-dien-3-yl (4-methoxyphenyl)carbamate (S2). The title compound was obtained as a yellow oil in 80% yield (2 steps, 1.2 g) starting from **S24** (0.88 g, 4.3 mmol) by following the General Procedure B, steps 2 and 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30–7.24 (m, 2H), 6.86–6.82 (m, 2H), 6.45 (s, 1H), 5.89–5.77 (m, 1H), 5.59–5.52 (m, 1H), 5.44–5.37 (m, 1H), 5.24 (dd, *J* = 10.9, 9.1 Hz, 1H), 5.07–4.96 (m, 2H), 3.77 (s, 3H), 2.45–2.37 (m, 1H), 2.13–2.09 (m, 2H), 1.88–1.77 (m, 1H), 1.70–1.53

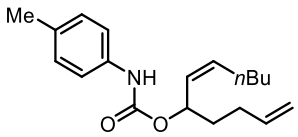
(m, 5H), 1.39–1.22 (m, 3H), 1.21–1.03 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.0, 153.5, 140.2, 138.0, 131.3, 126.1, 120.8, 115.1, 114.4, 71.3, 55.7, 37.2, 34.7, 33.5, 33.2, 29.5, 26.1, 25.9, 25.8. IR (neat, cm^{-1}): 3331, 2925, 1705, 1518, 1219, 1083. ESI HRMS *m/z* $(M+Na)^+$ calcd 366.2040, obsd 366.2037.



(*Z*)-8,8-Dimethylnona-1,6-dien-5-yl (4-methoxyphenyl)carbamate (S3). The title compound was obtained as a yellow oil in 23% yield (3 steps, 0.7 g) starting from pent-4-enal (0.8 g, 10 mmol) and 3,3-dimethylbut-1-yne (1.6 g, 20 mmol) by following the General Procedure B. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34–7.26 (m, 2H), 6.85–6.82 (m, 2H), 6.42 (s, 1H), 5.89–5.77 (m, 2H), 5.48 (dd, *J* = 12.3, 1.0 Hz, 1H), 5.17 (dd, *J* = 12.3, 9.7 Hz, 1H), 5.09–5.02 (m, 1H), 5.01–4.96 (m, 1H), 3.78 (s, 3H), 2.23–2.03 (m, 2H), 1.87–1.76 (m, 1H), 1.68–1.60 (m, 1H), 1.16 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.2, 137.9, 126.5, 115.2, 114.4, 55.7, 35.1, 34.2, 31.4, 31.3, 29.6. IR (neat, cm^{-1}): 3329, 2908, 1710, 1508, 1210, 788. ESI HRMS *m/z* $(M+Na)^+$ calcd 340.1883, obsd 340.1878.

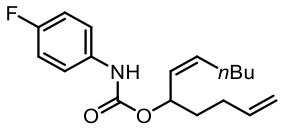


(*Z*)-Undeca-1,6-dien-5-yl phenylcarbamate (S4). The title compound was obtained as a yellow oil in 85% yield (0.95 g) starting from **S23** (0.62 g, 3.9 mmol) by following the General Procedure B, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.0 Hz, 2H), 7.23–7.18 (m, 2H), 7.00–6.93 (m, 1H), 6.52 (s, 1H), 5.83–5.68 (m, 1H), 5.57–5.46 (m, 2H), 5.36–5.23 (m, 1H), 5.01–4.95 (m, 1H), 4.95–4.88 (m, 1H), 2.16–2.08 (m, 2H), 2.07–2.00 (m, 2H), 1.81–1.72 (m, 1H), 1.61–1.51 (m, 1H), 1.33–1.23 (m, 4H), 0.82 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.2, 138.2, 137.9, 134.8, 129.2, 128.0, 123.4, 118.8, 115.2, 71.1, 34.4, 31.9, 29.5, 27.8, 22.5, 14.1. IR (neat, cm^{-1}): 3335, 2920, 1705, 1608, 1442, 1134, 752. ESI HRMS *m/z* $(M+Na)^+$ calcd 310.1778, obsd 310.1775.

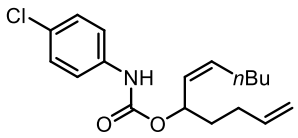


(*Z*)-Undeca-1,6-dien-5-yl p-tolylcarbamate (S5). The title compound was obtained as a yellow oil in 83% yield (0.50 g) starting from **S23** (0.30 g, 2.0 mmol) by following the General Procedure B, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29–7.22 (m, 2H), 7.09 (d, *J* = 8.1 Hz, 2H), 6.51 (s, 1H), 5.90–5.76 (m, 1H), 5.62–5.53 (m, 2H), 5.39–5.30

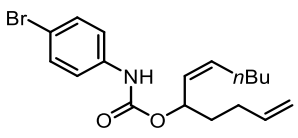
(m, 1H), 5.09–5.01 (m, 1H), 4.98 (dd, *J* = 10.2, 1.6 Hz, 1H), 2.29 (s, 3H), 2.22–2.05 (m, 4H), 1.89–1.79 (m, 1H), 1.69–1.57 (m, 1H), 1.39–1.30 (m, 4H), 0.90 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.3, 137.9, 135.6, 134.7, 133.0, 129.7, 128.1, 118.9, 115.2, 71.0, 34.4, 31.9, 29.5, 27.9, 22.5, 20.9, 14.1. IR (neat, cm^{-1}): 3327, 2925, 1705, 1518, 1205, 1046, 815. ESI HRMS *m/z* $(M+Na)^+$ calcd 324.1934, obsd 324.1931.



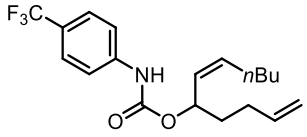
(*Z*)-Undeca-1,6-dien-5-yl (4-fluorophenyl)carbamate (S6). The title compound was obtained as a pale yellow oil in 90% yield (0.30 g) starting from **S23** (0.20 g, 1.1 mmol) by following the General Procedure B, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36–7.30 (m, 2H), 7.02–6.92 (m, 2H), 6.58 (s, 1H), 5.88–5.77 (m, 1H), 5.63–5.51 (m, 2H), 5.34 (dd, *J* = 11.0, 9.0 Hz, 1H), 5.04 (dd, *J* = 17.0, 2.1 Hz, 1H), 4.99 (d, *J* = 11.0 Hz, 1H), 2.23–2.07 (m, 4H), 1.87–1.79 (m, 1H), 1.68–1.60 (m, 1H), 1.39–1.31 (m, 4H), 0.90 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.1 (d, *J* = 242.4 Hz), 153.4, 137.8, 134.9, 134.2 (d, $J_{C\text{-}F}$ = 2.7 Hz), 127.9, 120.6 (d, $J_{C\text{-}F}$ = 8.5 Hz), 115.8 (d, $J_{C\text{-}F}$ = 22.6 Hz), 115.2, 71.2, 34.4, 31.9, 29.4, 27.8, 22.5, 14.1. ^{19}F NMR (471 MHz, $CDCl_3$) δ −119.8. IR (neat, cm^{-1}): 3323, 2928, 1705, 1515, 1208, 1046, 832. ESI HRMS *m/z* $(M+Na)^+$ calcd 328.1683, obsd 328.1682.



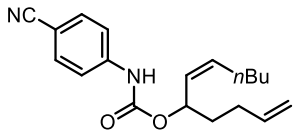
(*Z*)-Undeca-1,6-dien-5-yl (4-chlorophenyl)carbamate (S7). The title compound was obtained as a yellow oil in 77% yield (0.49 g) starting from **S23** (0.33 g, 2.0 mmol) by following the General Procedure B, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.5 Hz, 2H), 7.27–7.23 (m, 2H), 6.56 (s, 1H), 5.90–5.75 (m, 1H), 5.63–5.49 (m, 2H), 5.37–5.30 (m, 1H), 5.07–5.02 (m, 1H), 4.99 (dd, *J* = 10.2, 1.7 Hz, 1H), 2.23–2.06 (m, 4H), 1.89–1.79 (m, 1H), 1.69–1.61 (m, 1H), 1.39–1.32 (m, 4H), 0.90 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.0, 137.8, 136.8, 135.0, 129.2, 128.4, 127.8, 120.0, 115.3, 71.4, 34.4, 31.9, 29.5, 27.9, 22.5, 14.1. IR (neat, cm^{-1}): 3323, 2957, 1705, 1520, 1207, 826. ESI HRMS *m/z* $(M+Na)^+$ calcd 344.1388, obsd 344.1382.



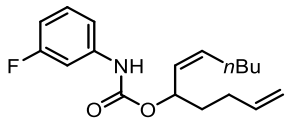
(*Z*)-Undeca-1,6-dien-5-yl (4-bromophenyl)carbamate (S8). The title compound was obtained as a yellow oil in 80% yield (0.58 g) starting from **S23** (0.33 g, 2.0 mmol) by following the General Procedure B, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42–7.36 (m, 2H), 7.29–7.25 (m, 2H), 6.59 (s, 1H), 5.91–5.74 (m, 1H), 5.65–5.49 (m, 2H), 5.37–5.18 (m, 1H), 5.08–5.01 (m, 1H), 4.99 (dd, *J* = 10.2, 1.7 Hz, 1H), 2.22–2.08 (m, 4H), 1.88–1.78 (m, 1H), 1.72–1.58 (m, 1H), 1.44–1.25 (m, 4H), 0.90 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.0, 137.8, 137.4, 135.0, 132.1, 127.8, 120.3, 115.9, 115.3, 71.4, 34.4, 31.9, 29.4, 27.9, 22.5, 14.1. IR (neat, cm^{-1}): 3328, 2957, 1668, 1508, 1218, 823. ESI HRMS *m/z* $(M+Na)^+$ calcd 388.0883, obsd 388.0881.



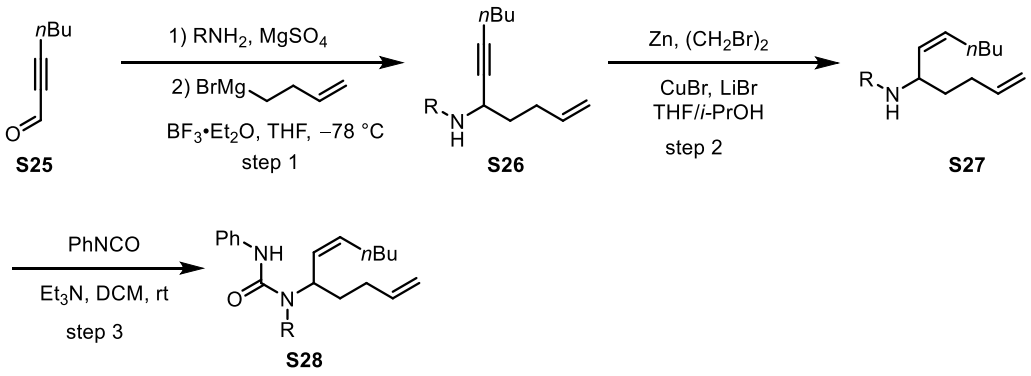
(*Z*)-Undeca-1,6-dien-5-yl (4-(trifluoromethyl)phenyl)carbamate (S9). The title compound was obtained as a pale yellow oil in 72% yield (0.51 g) starting from **S23** (0.35 g, 2.0 mmol) by following the General Procedure B, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.5 Hz, 2H), 7.49 (d, *J* = 8.5 Hz, 2H), 6.73 (s, 1H), 5.89–5.78 (m, 1H), 5.68–5.54 (m, 2H), 5.45–5.31 (m, 1H), 5.10–5.02 (m, 1H), 5.02–4.97 (m, 1H), 2.26–2.08 (m, 4H), 1.90–1.80 (m, 1H), 1.69–1.62 (m, 1H), 1.40–1.32 (m, 4H), 0.90 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 152.8, 141.4, 137.7, 135.1, 127.6, 126.5 (q, $J_{C\text{-}F}$ = 4.0 Hz), 125.2 (q, $J_{C\text{-}F}$ = 32.7 Hz), 124.4 (q, $J_{C\text{-}F}$ = 271.2 Hz), 118.2, 115.3, 71.6, 34.3, 31.9, 29.4, 27.9, 22.5, 14.1. ^{19}F NMR (471 MHz, $CDCl_3$) δ −62.0. IR (neat, cm^{-1}): 3322, 2929, 1708, 1537, 1308, 1121, 841. ESI HRMS *m/z* $(M+Na)^+$ calcd 378.1651, obsd 378.1648.



(*Z*)-Undeca-1,6-dien-5-yl (4-cyanophenyl)carbamate (S10). The title compound was obtained as a pale yellow oil in 89% yield (1.0 g) starting from **S23** (0.66 g, 3.9 mmol) by following the General Procedure B, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62–7.55 (m, 2H), 7.55–7.47 (m, 2H), 6.81 (s, 1H), 5.88–5.76 (m, 1H), 5.66–5.53 (m, 2H), 5.39–5.29 (m, 1H), 5.08–5.02 (m, 1H), 5.02–4.98 (m, 1H), 2.26–2.13 (m, 2H), 2.16–2.06 (m, 2H), 1.90–1.76 (m, 1H), 1.71–1.62 (m, 1H), 1.43–1.25 (m, 4H), 0.90 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 152.5, 142.4, 137.6, 135.3, 133.5, 127.5, 119.1, 118.4, 115.4, 106.3, 71.9, 34.3, 31.8, 29.4, 27.9, 22.5, 14.1. IR (neat, cm^{-1}): 3318, 2926, 2225, 1735, 1508, 1218, 840. ESI HRMS *m/z* $(M+Na)^+$ calcd 335.1730, obsd 335.1729.



(*Z*)-Undeca-1,6-dien-5-yl (3-fluorophenyl)carbamate (S11). The title compound was obtained as a pale yellow oil in 86% yield (0.47 g) starting from **S23** (0.30 g, 1.8 mmol) by following the General Procedure B, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 (d, *J* = 11.1 Hz, 1H), 7.17–7.10 (m, 1H), 6.93 (dd, *J* = 8.1, 1.9 Hz, 1H), 6.69–6.63 (m, 1H), 6.58 (s, 1H), 5.80–5.70 (m, 1H), 5.57–5.46 (m, 2H), 5.30–5.24 (m, 1H), 5.00–4.94 (m, 1H), 4.92 (dd, *J* = 10.2, 1.7 Hz, 1H), 2.16–2.01 (m, 4H), 1.81–1.72 (m, 1H), 1.64–1.55 (m, 1H), 1.33–1.23 (m, 4H), 0.83 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.4 (d, $J_{C\text{-}F}$ = 244.4 Hz), 152.9, 139.9 (d, $J_{C\text{-}F}$ = 11.0 Hz), 137.8, 135.0, 130.2 (d, $J_{C\text{-}F}$ = 9.5 Hz), 127.8, 115.3, 114.0, 110.1 (d, $J_{C\text{-}F}$ = 21.3 Hz), 106.2 (d, $J_{C\text{-}F}$ = 27.0 Hz), 71.4, 34.4, 31.9, 29.4, 27.9, 22.5, 14.1. ^{19}F NMR (471 MHz, $CDCl_3$) δ −111.7. IR (neat, cm^{-1}): 3323, 2958, 1707, 1608, 1507, 1218, 771. ESI HRMS *m/z* $(M+Na)^+$ calcd 328.1683, obsd 328.1681.

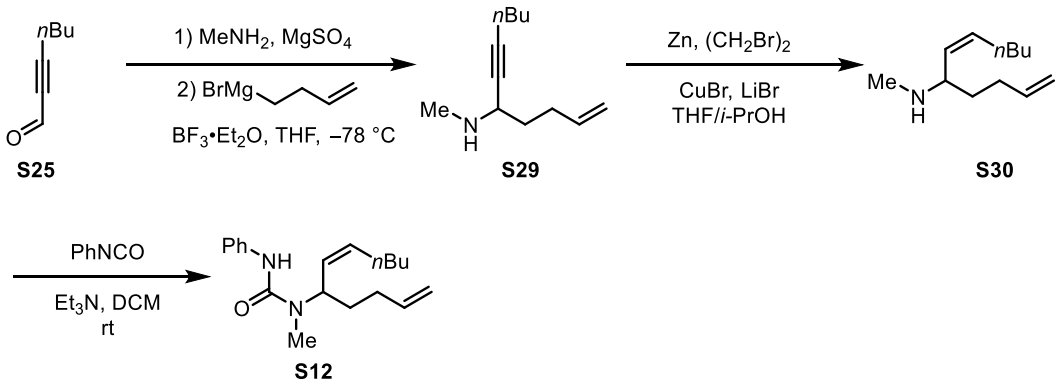


General Procedure C

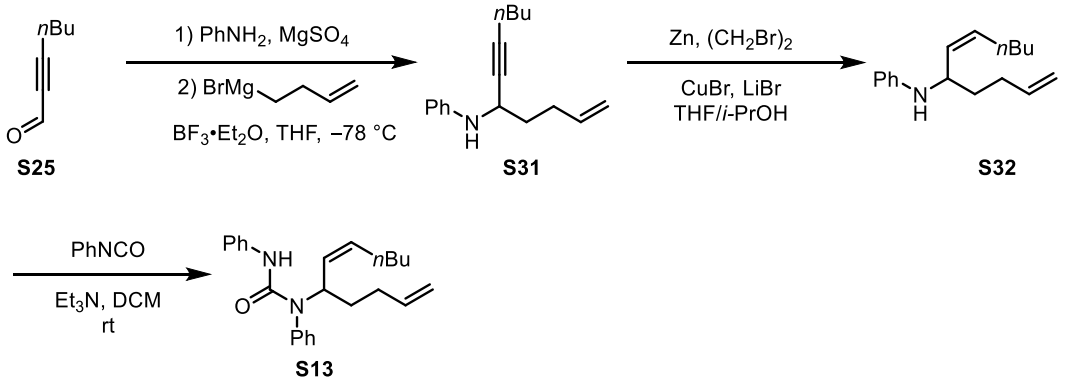
Step 1: To a solution of hept-2-ynal (**S25**)[4] (1.0 equiv) in THF (50 mL) at rt was added amine (1.0 equiv) dropwise over 5 min. Upon complete addition, the reaction mixture was stirred for 2 h at rt. The reaction mixture was filtered through a pad of celite and the filtrate was cooled to −78 °C. $BF_3{\cdot}Et_2O$ (1.5 equiv) was added dropwise. The resulting reaction mixture was stirred for 1 h at −78 °C. But-3-en-1-ylmagnesium bromide (0.5 M in THF, 2.0 equiv) was added dropwise over 10 min at the same temperature. The reaction mixture was warmed to ambient temperature and stirred for 2 h. Saturated aqueous NH_4Cl (20 mL) was added to quench the reaction. The resulting mixture was extracted with EtOAc (3 x 20 mL). The combined organic solution was washed with brine (20 mL), dried over anhydrous $MgSO_4$, and filtered. The filtrate was concentrated under reduced pressure. The residue was chromatographed through silica gel eluting with ethyl acetate/hexanes to give the desired product **S26**.

Step 2: To a suspension of Zn dust (25 equiv) in *i*-PrOH (20 mL) was added $(CH_2Br)_2$ (2.2 equiv). The mixture was heated at reflux for 15 min. A solution of CuBr (3.0 equiv) and LiBr (5.3 equiv) in 20 mL of THF was added slowly. The resulting mixture was heated at reflux for 20 min. A solution of **S26** (1.0 equiv) in *i*-PrOH was added dropwise over 5 min. The reaction mixture was then heated under Ar until complete consumption of the starting material (monitored by ^{1}H-NMR). The reaction mixture was filtered through a pad of celite. The filtrate was concentrated under reduced pressure. The residue was chromatographed through silica gel eluting with ethyl acetate/hexanes to give the product **S27**.

Step 3: To a solution of **S27** (1.0 equiv) in DCM (0.1 M) was added aryl isocyanate (1.2 equiv) at rt, followed by Et_3N (2 equiv). The resulting reaction mixture was stirred at rt until complete consumption of the starting material (monitored by TLC). The solvent was removed under reduced pressure. The residue was chromatographed through silica gel eluting with ethyl acetate/hexanes to give the product **S28**.

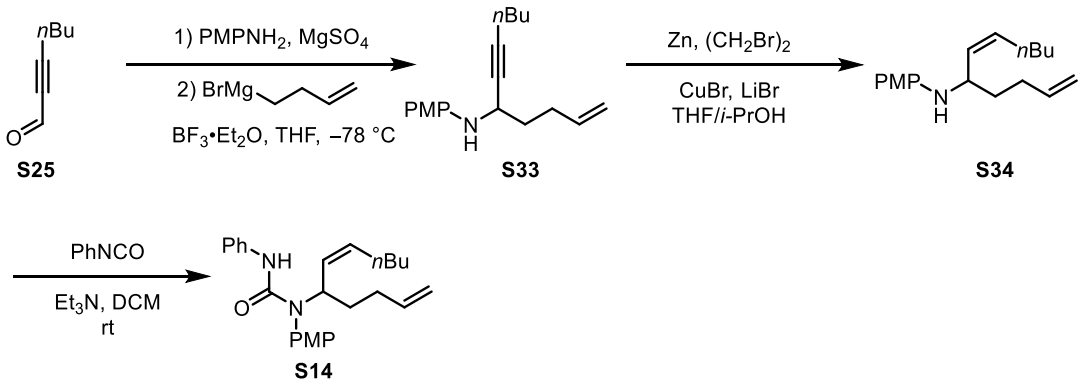


(*Z*)-1-Methyl-3-phenyl-1-(undeca-1,6-dien-5-yl)urea (S12). The title compound (0.25 g) was obtained as a yellow oil in 10% yield (3 steps) starting from **S25** (1.1 g, 10 mmol) by following the General Procedure C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (d, J = 8.1 Hz, 2H), 7.27–7.20 (m, 2H), 7.01–6.95 (m, 1H), 6.46 (s, 1H), 5.89–5.78 (m, 1H), 5.60–5.52 (m, 1H), 5.38–5.30 (m, 1H), 5.09–4.94 (m, 3H), 2.80 (s, 3H), 2.16–2.00 (m, 4H), 1.72–1.65 (m, 1H), 1.61–1.53 (m, 1H), 1.36–1.27 (m, 4H), 0.86 (t, J = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 155.3, 139.5, 138.0, 134.8, 128.8, 127.7, 122.8, 120.1, 115.1, 50.9, 32.7, 31.7, 30.3, 28.8, 27.8, 22.3, 14.0. IR (neat, cm^{-1}): 3304, 2922, 1618, 1508, 1244, 747. ESI HRMS *m/z* $(M+Na)^+$ calcd 323.2094, obsd 323.2100.

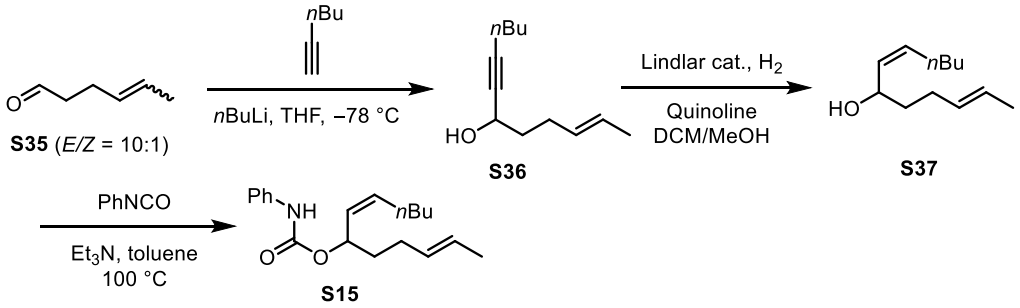


(*Z*)-*N*-(Undeca-1,6-dien-5-yl)aniline (S32). The title compound (0.40 g) was obtained as a yellow oil in 8% yield (2 steps) starting from **S25** (2.2 g, 20 mmol) by following the General Procedure C, steps 1 and 2. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.16–7.11 (m, 2H), 6.70–6.65 (m, 1H), 6.59–6.53 (m, 2H), 5.92–5.77 (m, 1H), 5.54–5.44 (m, 1H), 5.21–5.15 (m, 1H), 5.08–5.01 (m, 1H), 5.01–4.95 (m, 1H), 4.16–4.05 (m, 1H), 3.54 (s, 1H), 2.20–2.09 (m, 4H), 1.83–1.71 (m, 1H), 1.63–1.46 (m, 2H), 1.38–1.35 (m, 3H), 0.93–0.87 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.8, 138.4, 132.5, 132.2, 129.3, 117.4, 115.1, 113.6, 50.6, 35.5, 32.0, 30.2, 28.0, 22.6, 14.2. IR (neat, cm^{-1}): 3356, 2961, 2925, 1602, 1502, 1260, 1133, 799. ESI HRMS *m/z* $(M+H)^+$ calcd 244.2060, obsd 244.2062.

(*Z*)-1,3-Diphenyl-1-(undeca-1,6-dien-5-yl)urea (S13). The title compound was obtained as a yellow oil in 70% yield (0.21 g) starting from **S32** (0.23 g, 1.0 mmol) by following the General Procedure C, step 3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51–7.39 (m, 3H), 7.27–7.17 (m, 6H), 6.99–6.93 (m, 1H), 5.92–5.81 (m, 2H), 5.56–5.49 (m, 1H), 5.47–5.40 (m, 1H), 5.07–5.00 (m, 2H), 4.96 (d, *J* = 10.2 Hz, 1H), 2.28–2.18 (m, 2H), 2.16–2.09 (m, 2H), 1.86–1.78 (m, 1H), 1.51–1.45 (m, 1H), 1.40–1.31 (m, 4H), 0.90 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.0, 139.2, 138.4, 138.3, 134.3, 131.2, 130.0, 128.9, 122.9, 119.5, 115.0, 52.1, 34.0, 31.9, 30.7, 28.0, 22.5, 14.2. IR (neat, cm^{-1}): 3426, 2924, 1677, 1500, 1438, 1235, 748. ESI HRMS *m/z* $(M+Na)^+$ calcd 385.2250, obsd 385.2247.



(*Z*)-1-(4-Methoxyphenyl)-3-phenyl-1-(undeca-1,6-dien-5-yl)urea (S14). The title compound (0.10 g) was obtained as a yellow oil in 4% yield (3 steps) starting from **S25** (1.1 g, 10 mmol) by following the General Procedure C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25–7.18 (m, 4H), 7.17–7.12 (m, 2H), 6.98–6.92 (m, 3H), 5.90 (s, 1H), 5.87–5.80 (m, 1H), 5.55–5.48 (m, 1H), 5.46–5.40 (m, 1H), 5.06–4.92 (m, 3H), 3.86 (s, 3H), 2.29–2.19 (m, 2H), 2.16–2.08 (m, 2H), 1.84–1.74 (m, 1H), 1.48–1.41 (m, 1H), 1.39–1.30 (m, 4H), 0.90 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.7, 154.3, 139.3, 138.4, 134.2, 132.4, 130.5, 129.0, 128.9, 122.8, 119.4, 115.1, 114.9, 55.7, 51.8, 34.0, 32.0, 30.7, 28.0, 22.5, 14.2. IR (neat, cm^{-1}): 3424, 2925, 1674, 1508, 1240, 1134, 750. ESI HRMS *m/z* (M+Na)$^+$ calcd 415.2356, obsd 415.2353.



(2*E*,7*Z*)-Dodeca-2,7-dien-6-yl phenylcarbamate (S15). The title compound (1.30 g) was obtained as a yellow oil in 32% yield (3 steps) starting from **S35** (1.36 g, 13.6 mmol) by following the General Procedure B. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41–7.34 (m, 2H), 7.31–7.27 (m, 2H), 7.07–7.00 (m, 1H), 6.56 (s, 1H), 5.63–5.52 (m, 2H), 5.51–5.44 (m, 1H), 5.43–5.32 (m, 2H), 2.26–2.15 (m, 2H), 2.13–2.08 (m, 2H), 1.85–1.76 (m, 1H), 1.63–1.57 (m, 5H), 1.36–1.31 (m, 3H), 0.90 (t, J = 6.8 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.2, 138.2, 134.7, 129.5, 129.2, 128.1, 124.9, 123.4, 118.8, 71.3, 35.0, 31.9, 27.8, 22.8, 22.5, 14.1, 12.9. IR (neat, cm^{-1}): 3320, 2898, 1688, 1528, 1210, 808. ESI HRMS *m/z* (M+Na)$^+$ calcd 324.1934, obsd 324.1923.

5. References

1. E. D. Slack, C. M. Gabriel and B. H. Lipshutz, *Angew. Chem., Int. Ed*., 2014, **53**, 14051.

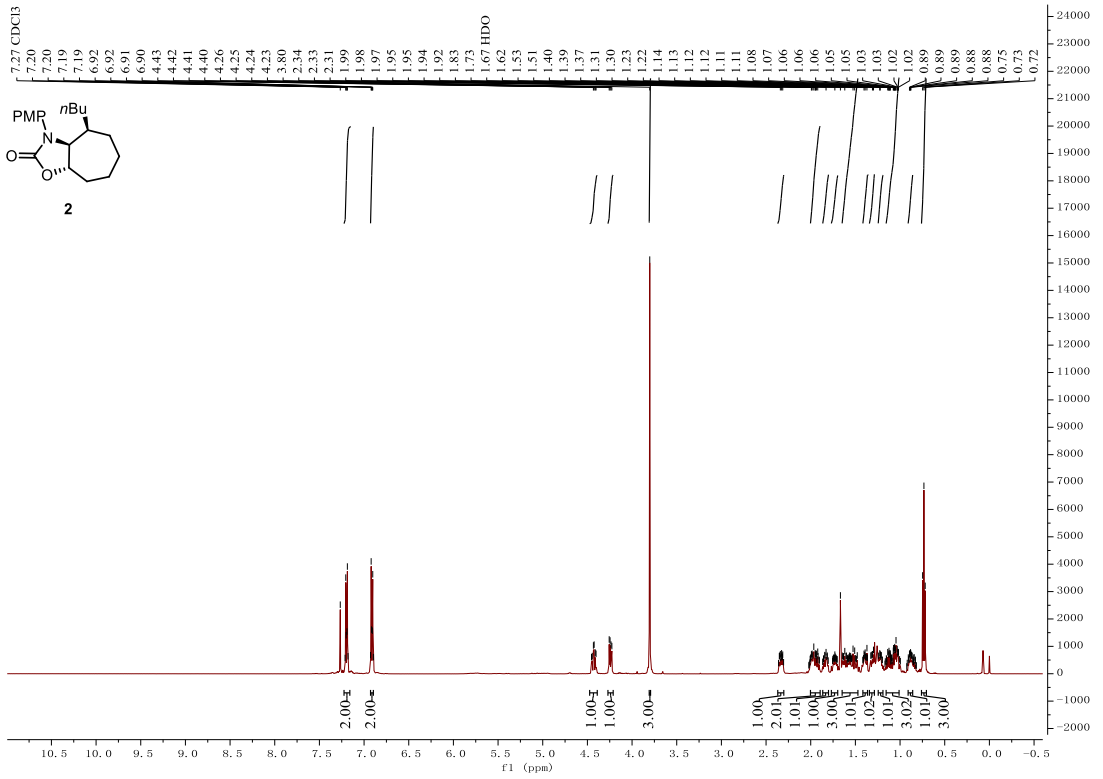
2. J. Cossy, N. Blanchard and C. Meyer, *Tetrahedron Lett.*, 2002, **43**, 1801.

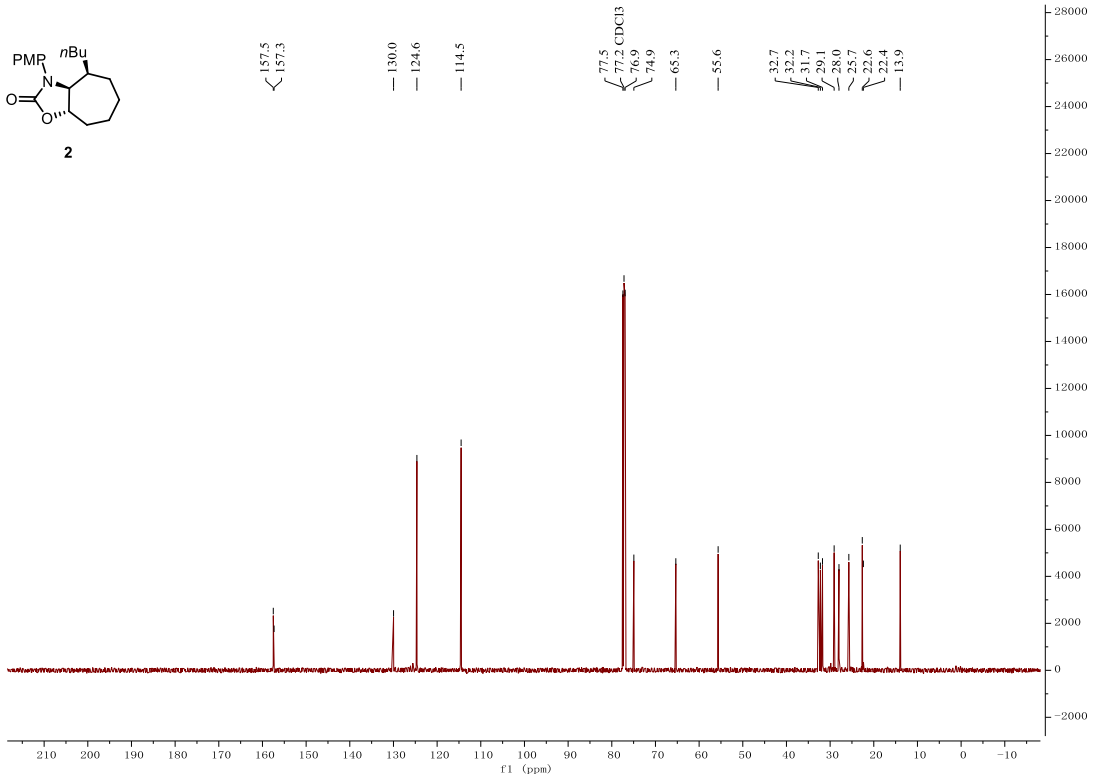
3. L. Zhu, P. Xiong, Z. Y. Mao, Y. H. Wang, X. Yan, X. Lu, and H.-C. Xu, *Angew. Chem., Int. Ed.*, 2016, **55**, 2226.

4. E. S. Sattely, G. A. Cortez, D. C. Moebius, R. R. Schrock and A. H. Hoveyda, *J. Am. Chem. Soc.*, 2005, **127**, 8526.

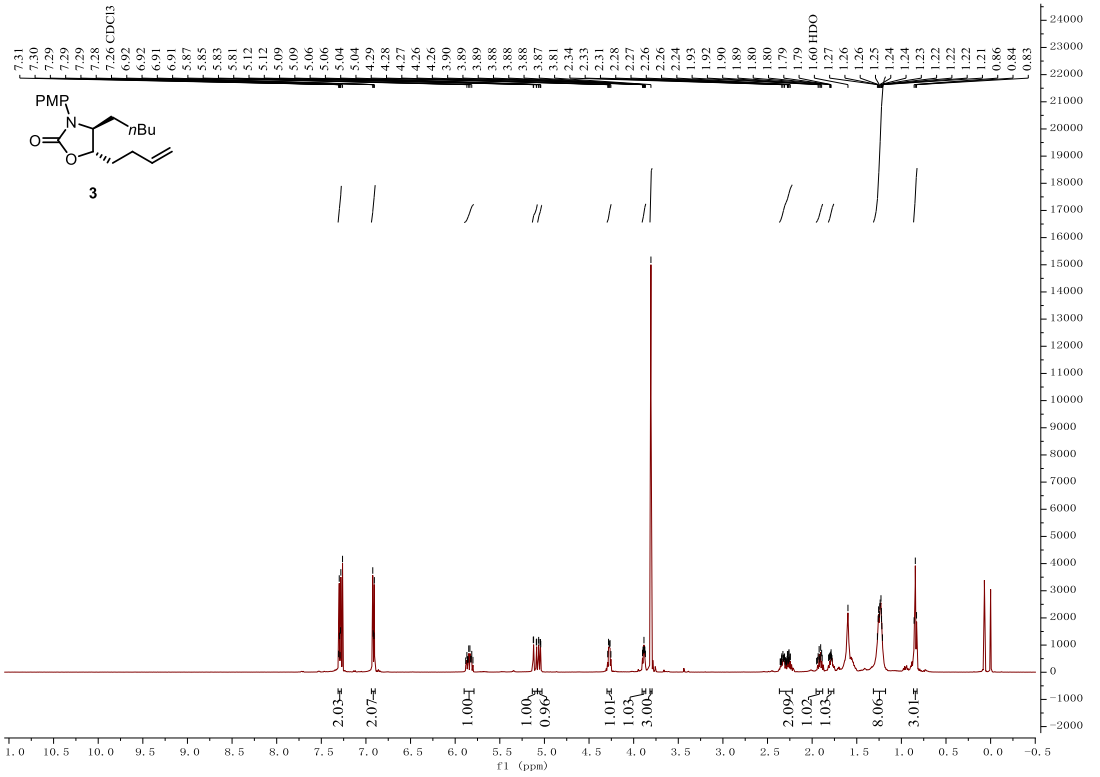
6. NMR Spectra for New Compounds

Compound 2





Compound 3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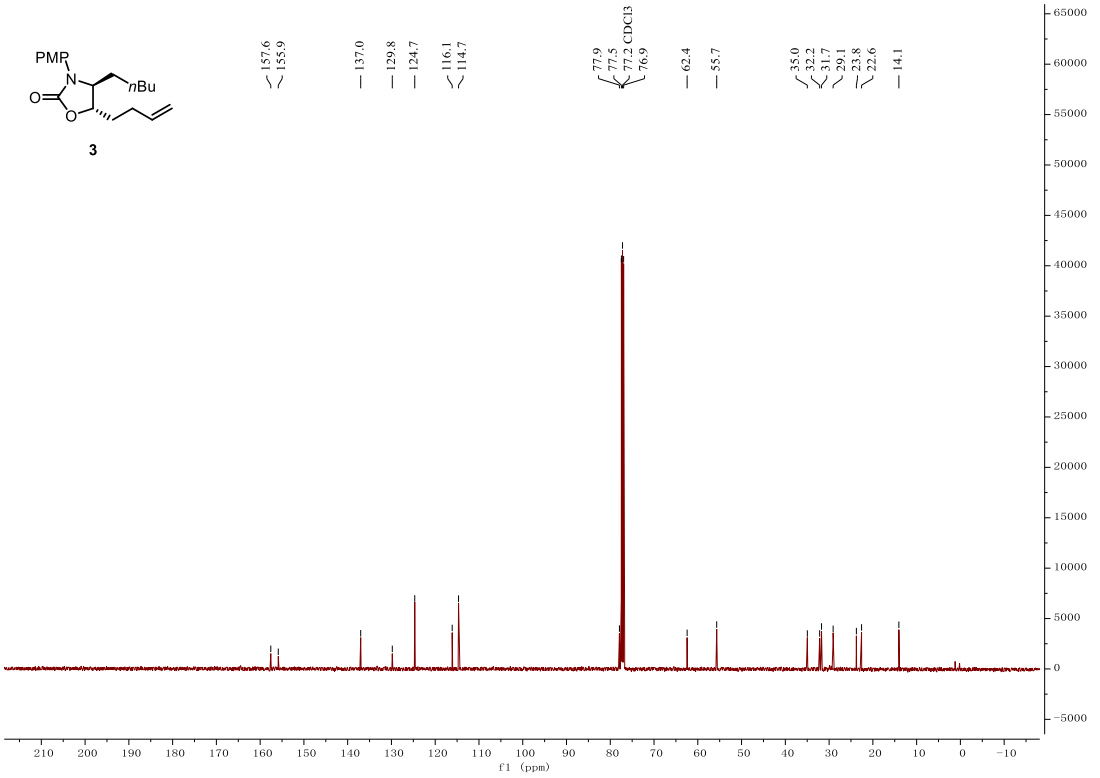
3
7.31 7.30 7.29 7.29 7.29 7.28 7.26 CDCl3 6.92 6.92 6.91 6.91 5.87 5.85 5.83 5.81 5.12 5.12 5.09 5.09 5.06 5.06 5.04 5.04 4.29 4.28 4.27 4.26 4.26 3.90 3.89 3.89 3.88 3.88 3.87 3.81 2.34 2.33 2.31 2.28 2.27 2.26 2.26 2.24 1.93 1.92 1.90 1.89 1.80 1.80 1.79 1.79 1.60 HDO 1.27 1.26 1.26 1.25 1.24 1.24 1.23 1.22 1.22 1.22 1.21 0.86 0.84 0.83
2.03 2.07 1.00 1.00 0.96 1.01 1.03 3.00 2.09 1.02 1.03 8.06 3.01
f1 (ppm)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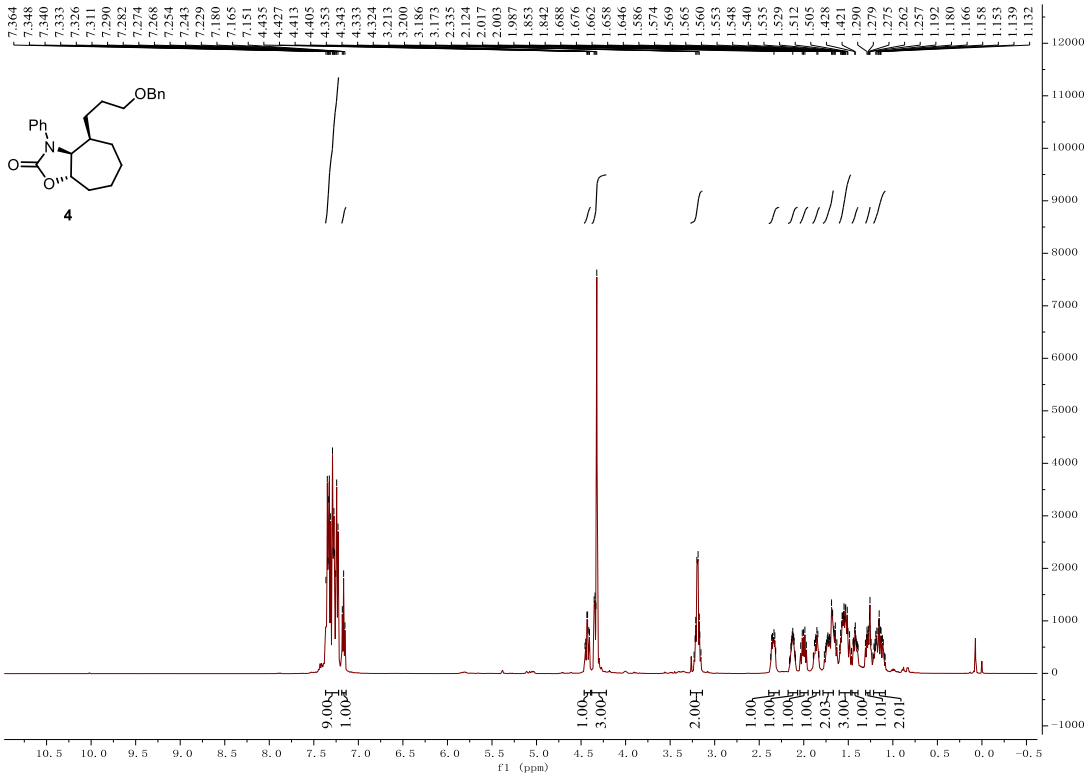
nBu
3
157.6 155.9 137.0 129.8 124.7 116.1 114.7 77.9 77.5 77.2 CDCl3 76.9 62.4 55.7 35.0 32.2 31.7 29.1 23.8 22.6 14.1
f1 (ppm)

Compound 4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Ph
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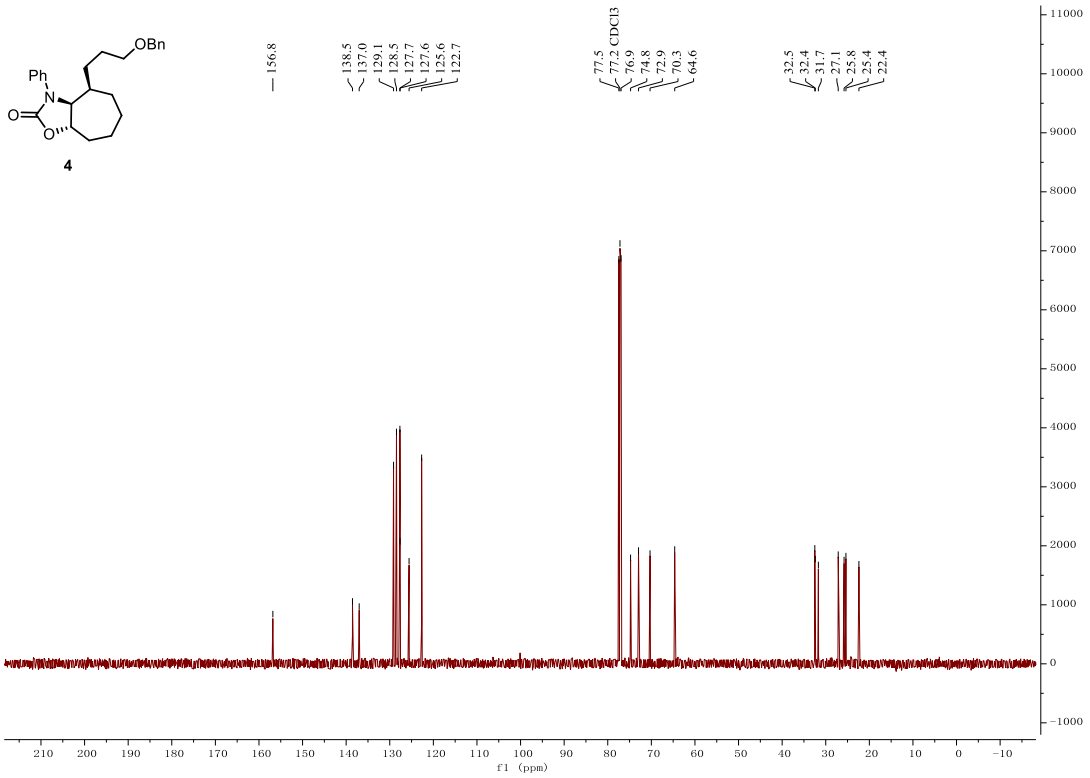
O
O
4
f1 (ppm)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Ph
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O
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129.1
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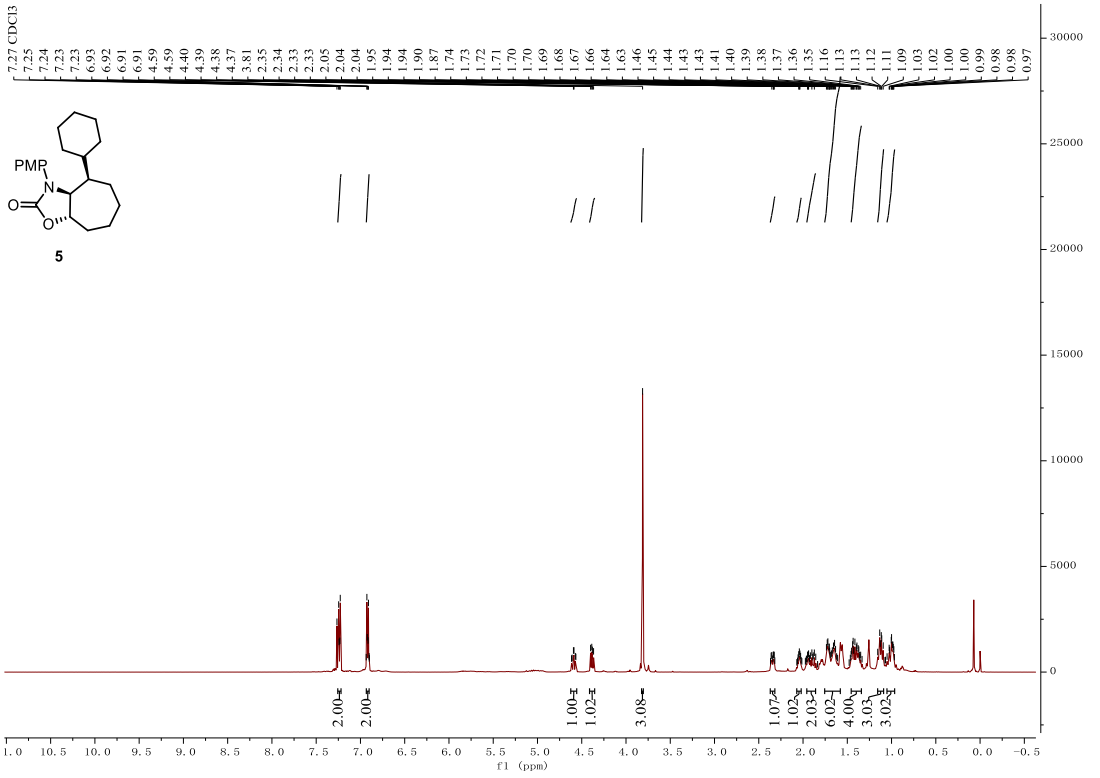
127.7
127.6
125.6
122.7
77.5
77.2 CDCl3
76.9
74.8
72.9
70.3
64.6
32.5
32.4
31.7
27.1
25.8
25.4
22.4
f1 (ppm)

Compound 5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6.92
6.91
6.91
4.59
4.59
4.40
4.39
4.38
4.37
3.81
2.35
2.34
2.33
2.33
2.05
2.04
2.04
1.95
1.94
1.94
1.90
1.87
1.74
1.73
1.72
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1.36
1.35
1.16
1.13
1.13
1.12
1.11
1.09
1.03
1.02
1.00
1.00
0.99
0.98
0.98
0.97
2.00
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1.07
1.02
2.03
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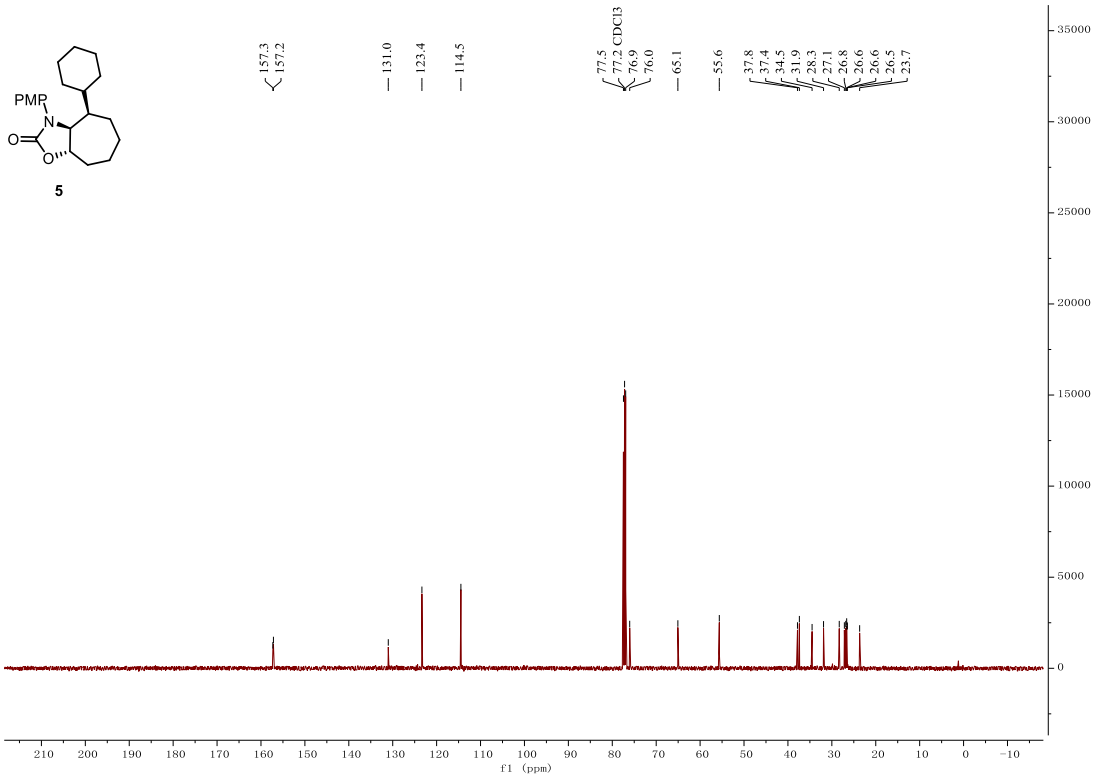
4.00
3.03
3.02
f1 (ppm)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65.1
55.6
37.8
37.4
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31.9
28.3
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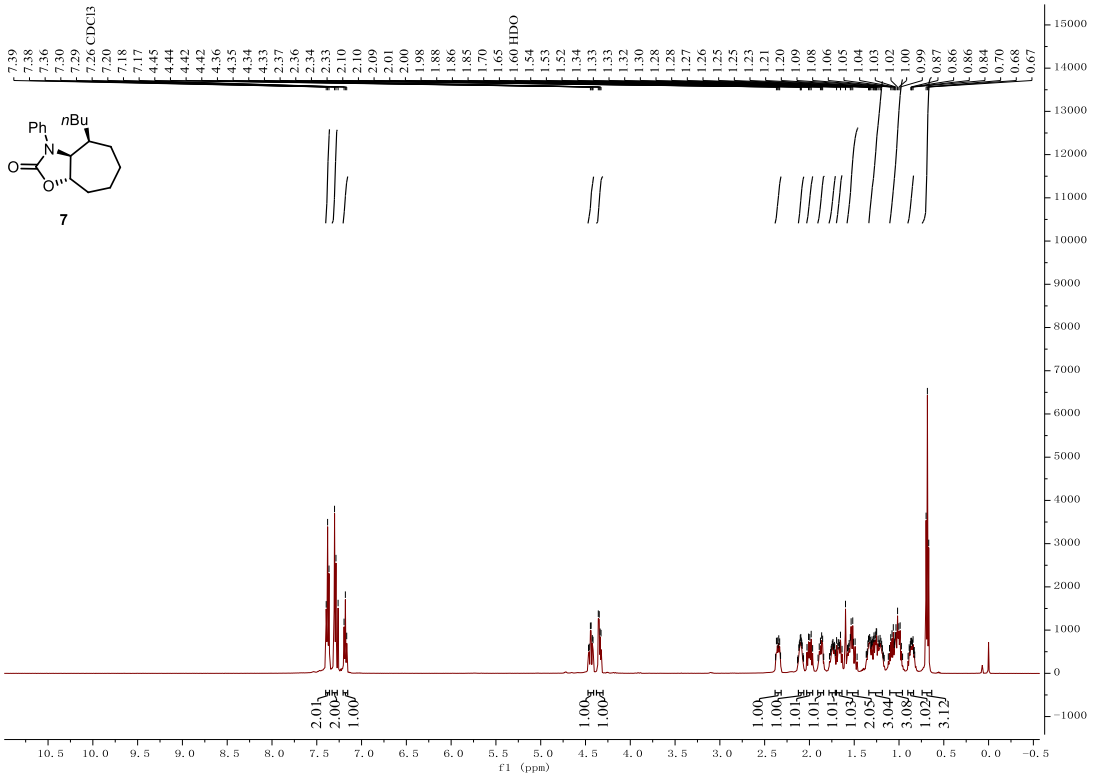
26.8
26.6
26.6
26.5
23.7
f1 (ppm)

Compound 7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1.33
1.33
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1.30
1.28
1.28
1.27
1.26
1.25
1.25
1.23
1.21
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1.05
1.04
1.03
1.02
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Ph
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7
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3.08
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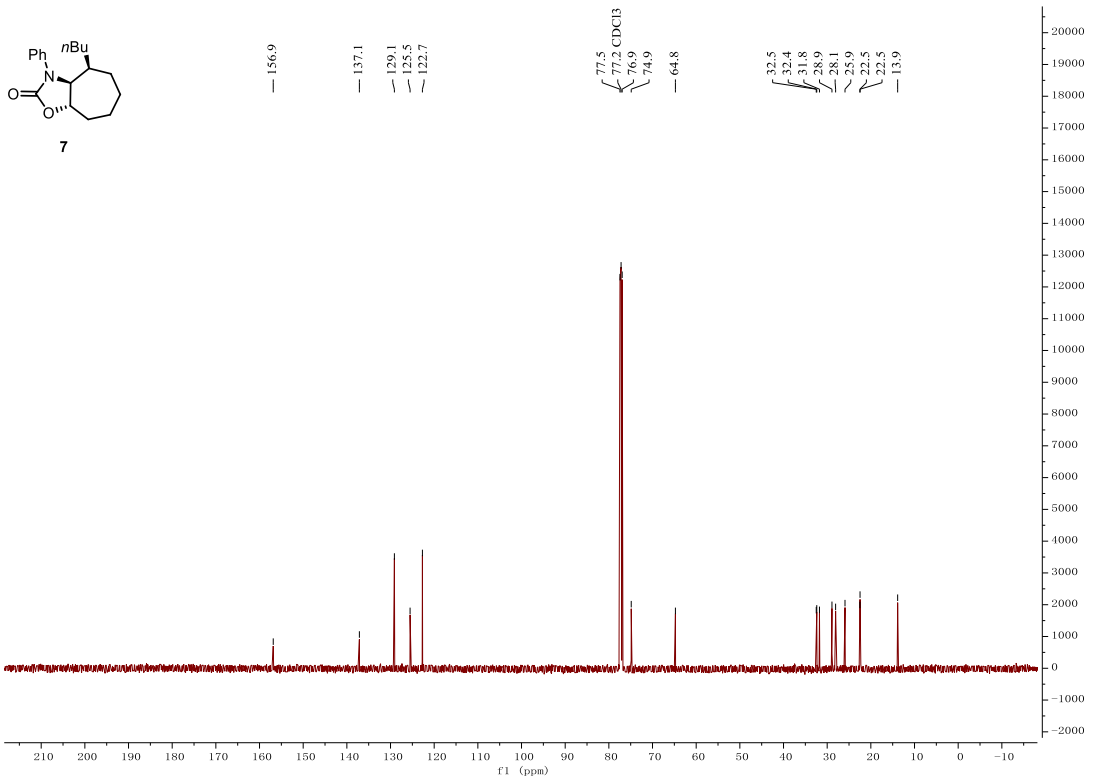
f1 (ppm)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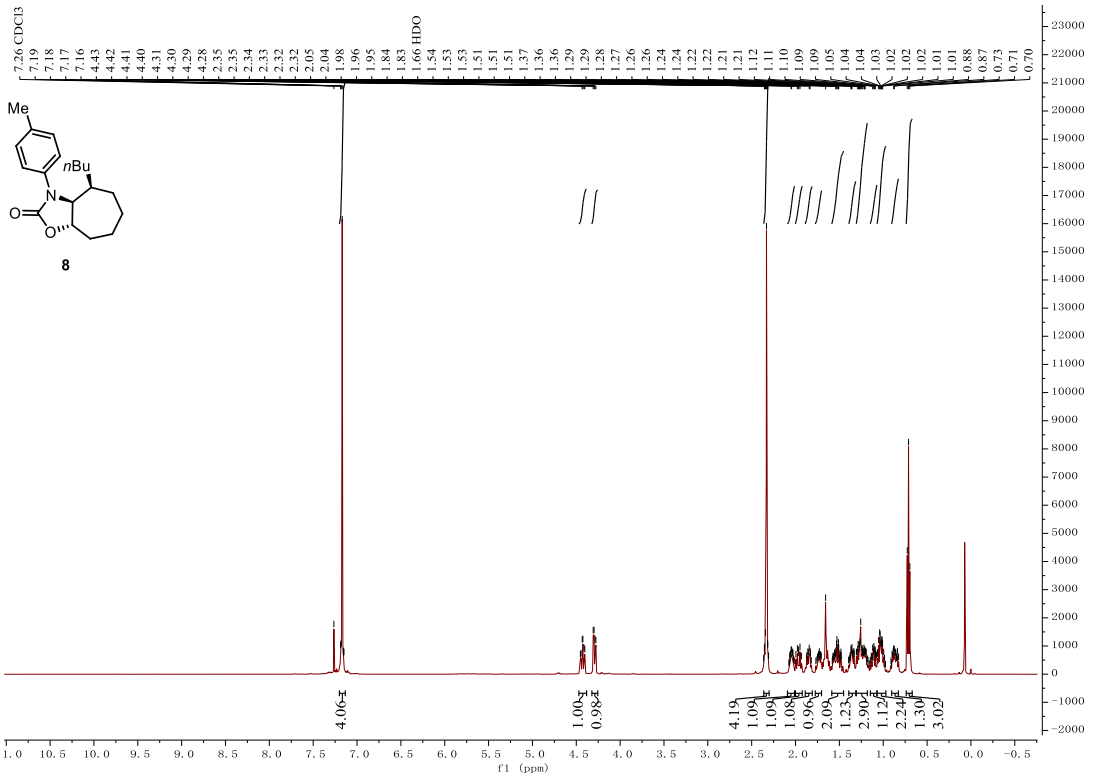
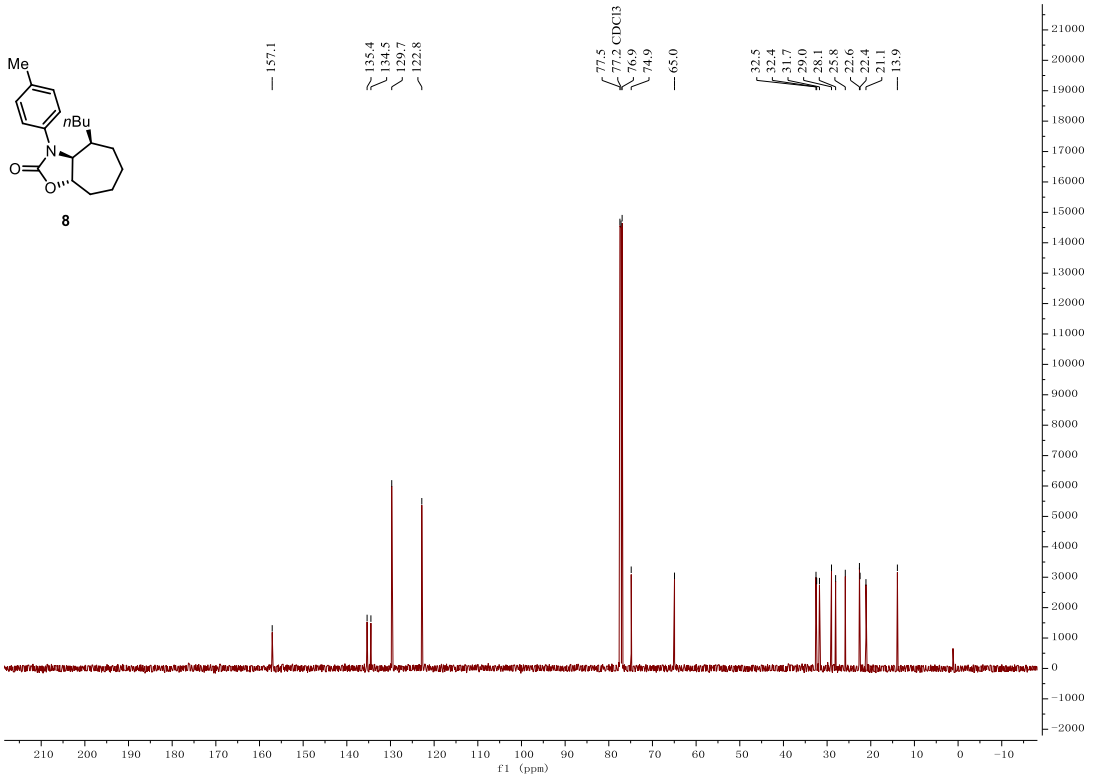
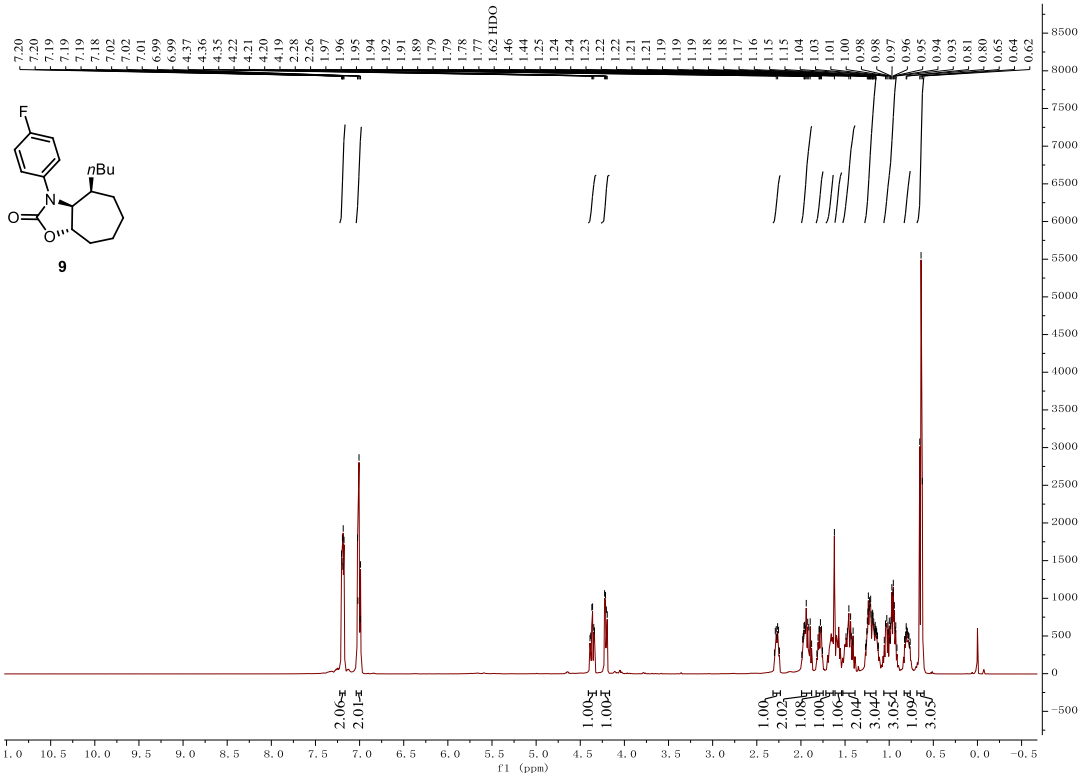
77.2 CDCl3
76.9
74.9
64.8
32.5
32.4
31.8
28.9
28.1
25.9
22.5
22.5
13.9
f1 (ppm)

Compound 8





Compound 9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N
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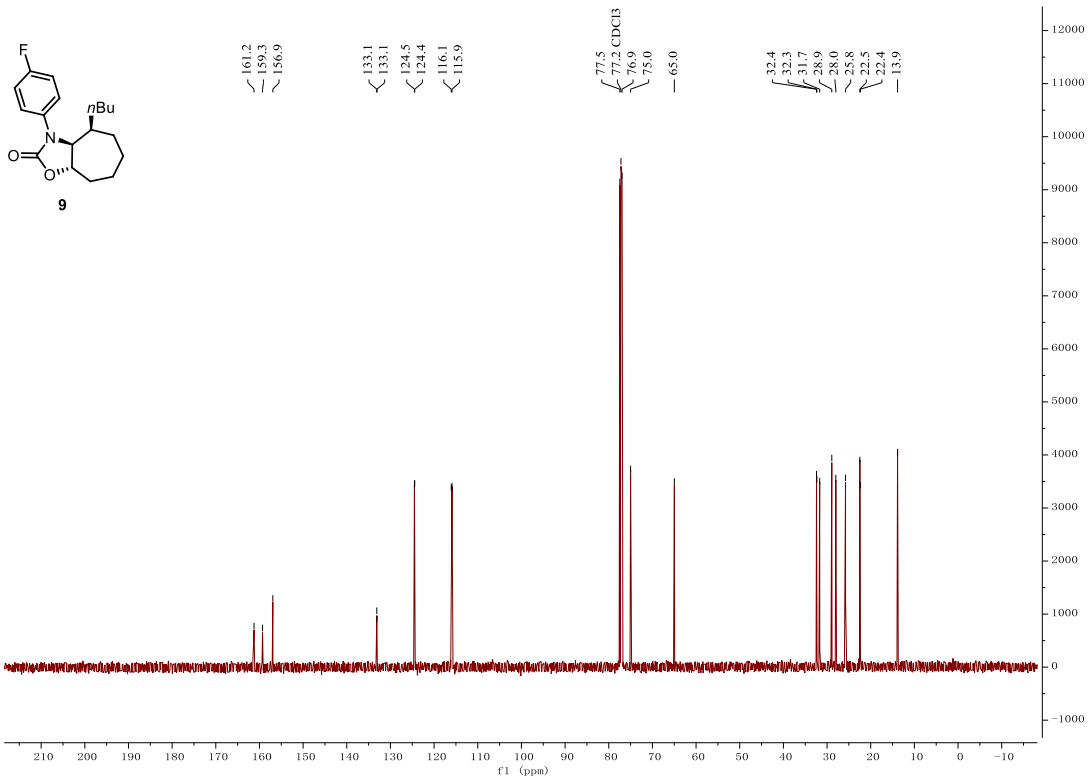
O
9
HDO
f1 (ppm)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115.9
77.5
77.2 CDCl3
76.9
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32.3
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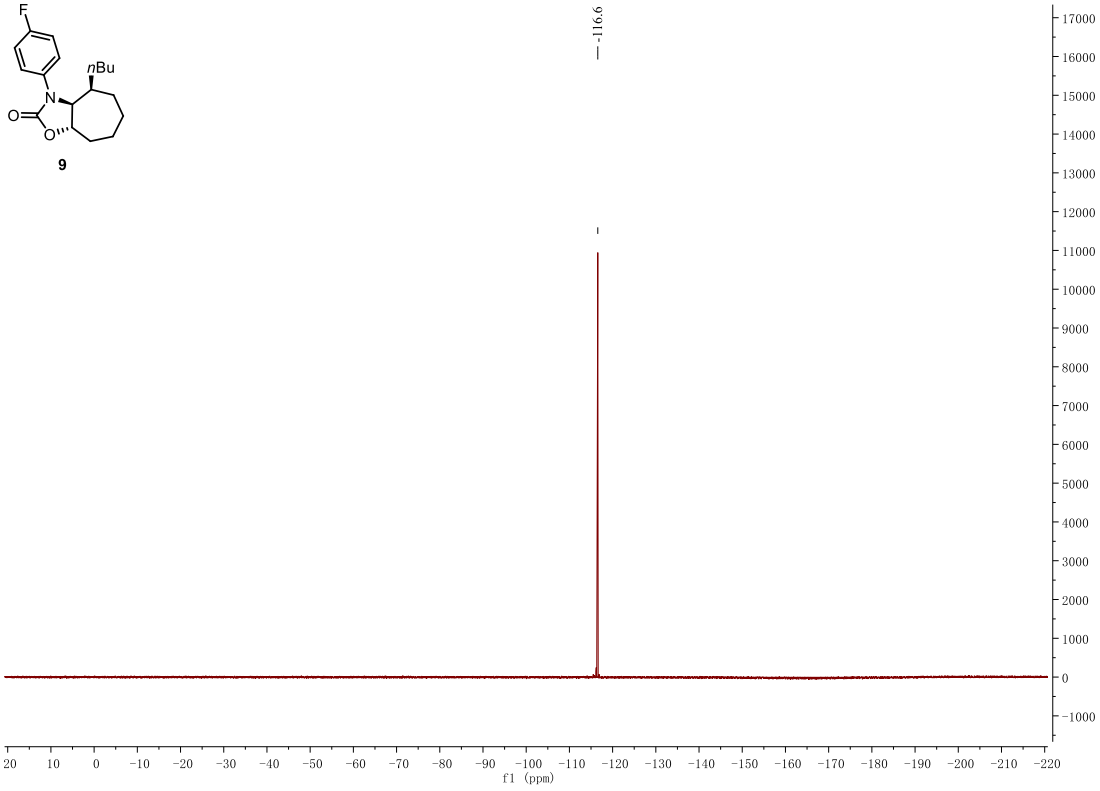
22.5
22.4
13.9
f1 (ppm)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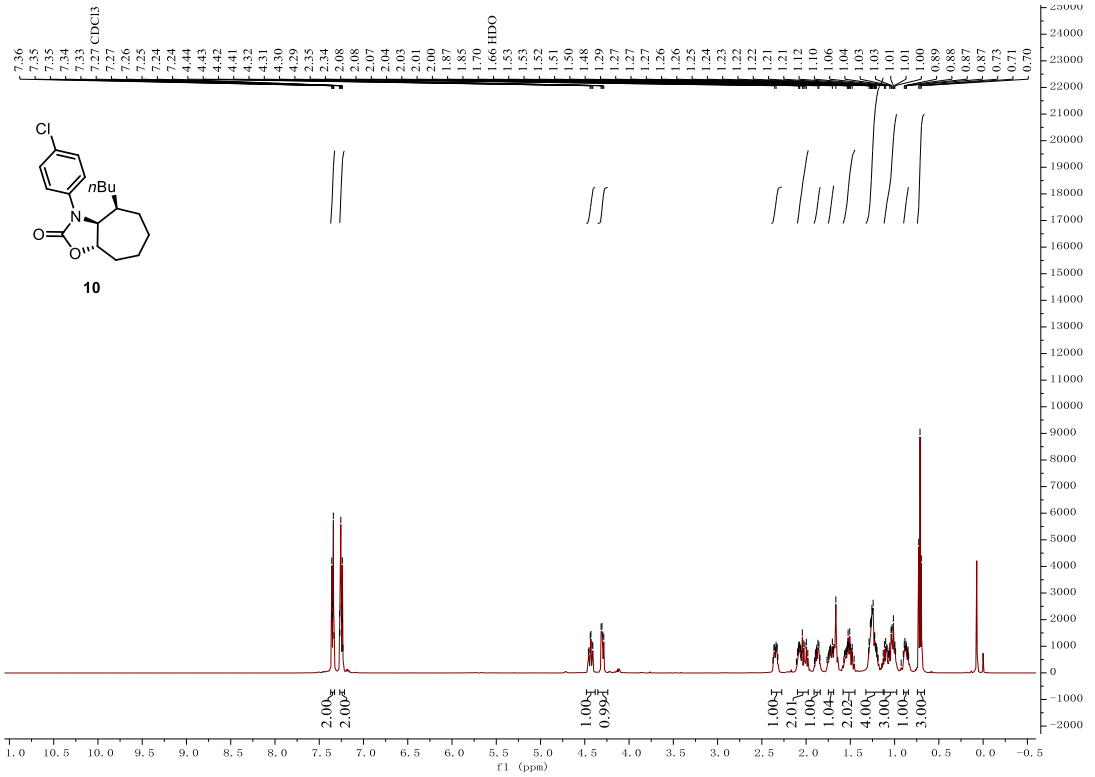
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

Compound 10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1.04
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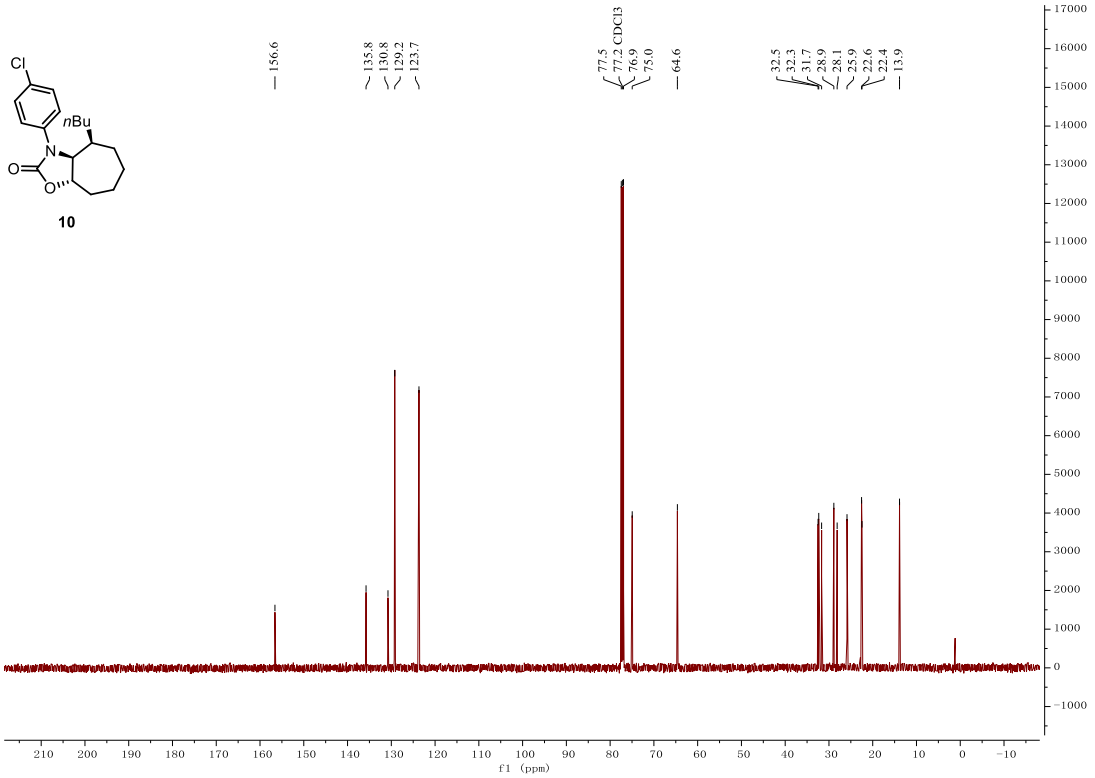
f1 (ppm)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77.5
77.2 CDCl3
76.9
75.0
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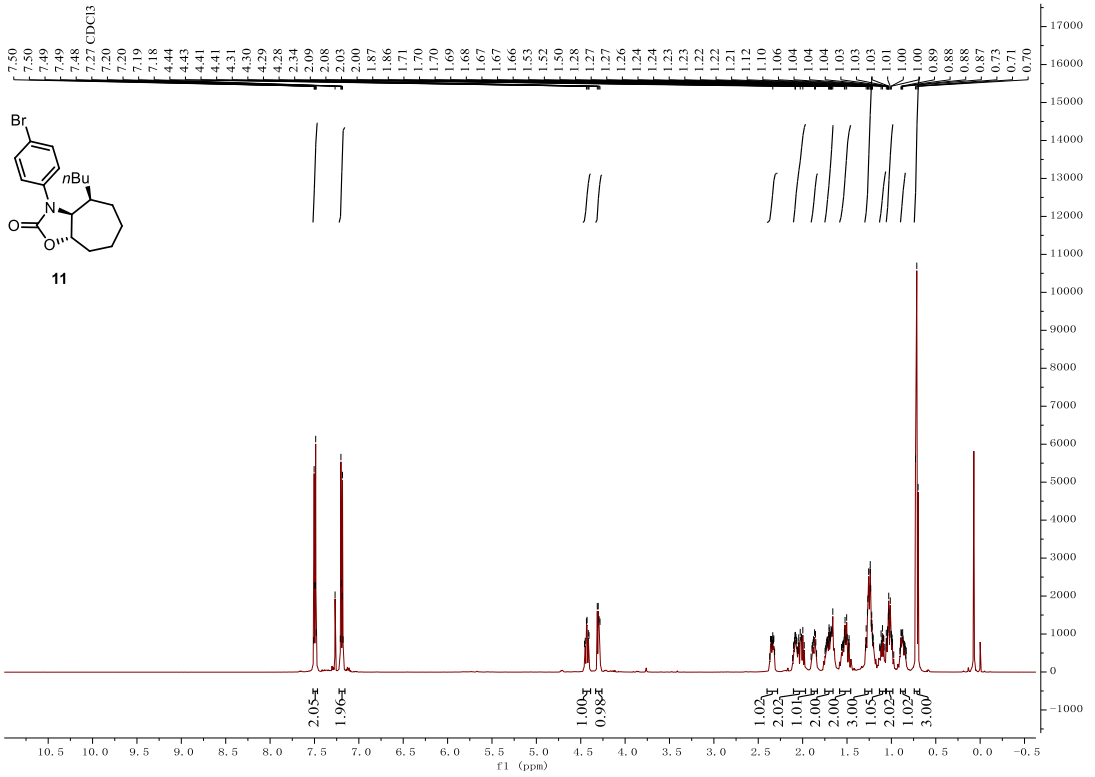
31.7
28.9
28.1
25.9
22.6
22.4
13.9
f1 (ppm)

Compound 11


Br
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N
O
O
11
7.50 7.50 7.49 7.49 7.48 7.27 CDCl3 7.20 7.20 7.19 7.18 4.44 4.43 4.41 4.41 4.31 4.30 4.29 4.28 2.34 2.09 2.08 2.03 2.00 1.87 1.86 1.71 1.70 1.70 1.69 1.68 1.67 1.67 1.66 1.53 1.52 1.50 1.28 1.27 1.27 1.26 1.24 1.24 1.23 1.23 1.22 1.22 1.21 1.12 1.10 1.06 1.04 1.04 1.04 1.03 1.03 1.03 1.01 1.00 1.00 0.89 0.88 0.88 0.87 0.73 0.71 0.70
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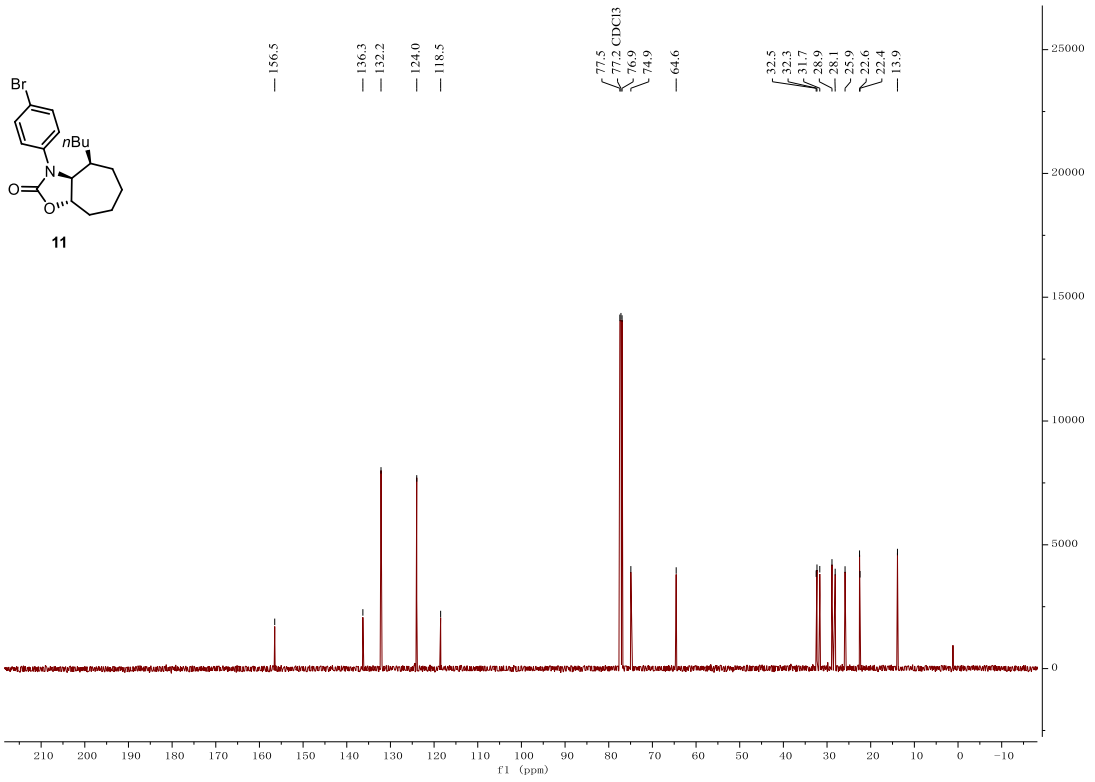
f1 (ppm)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77.2 CDCl3
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74.9
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28.1
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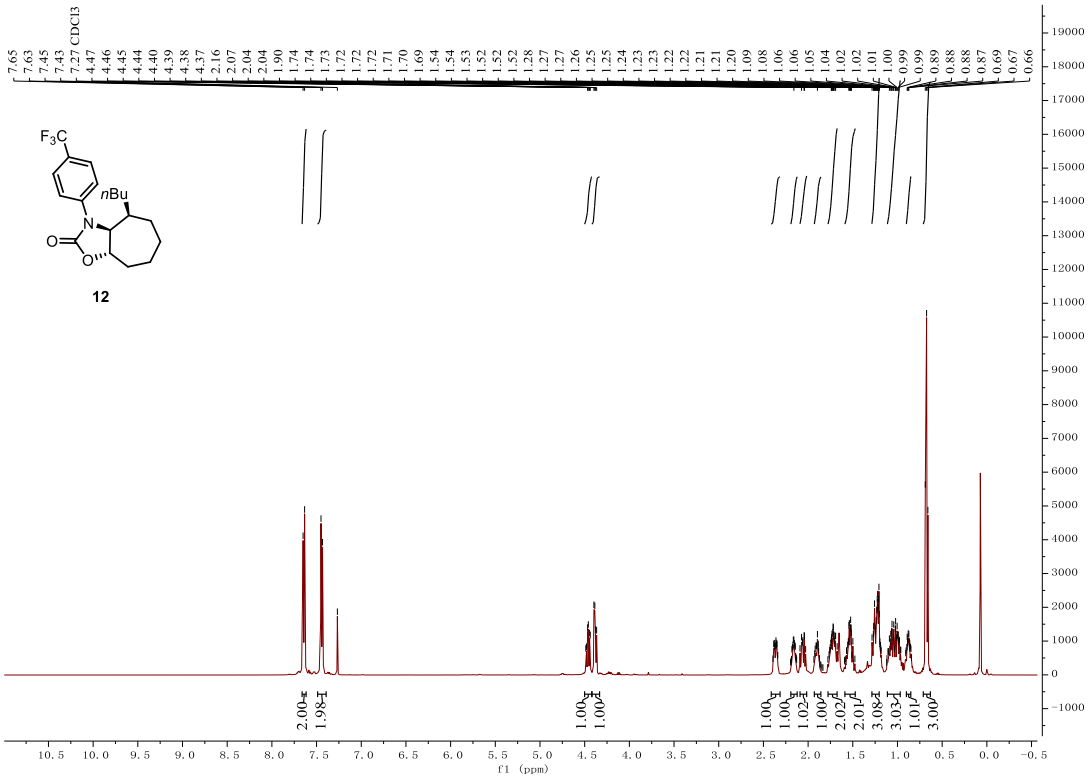
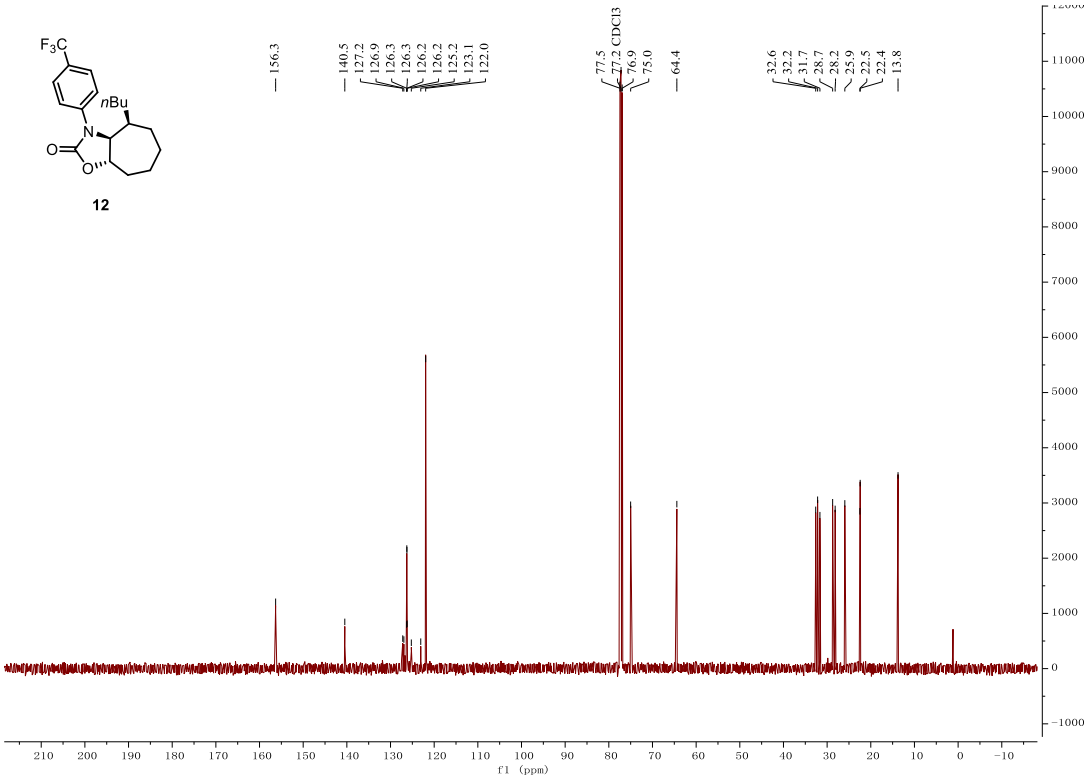
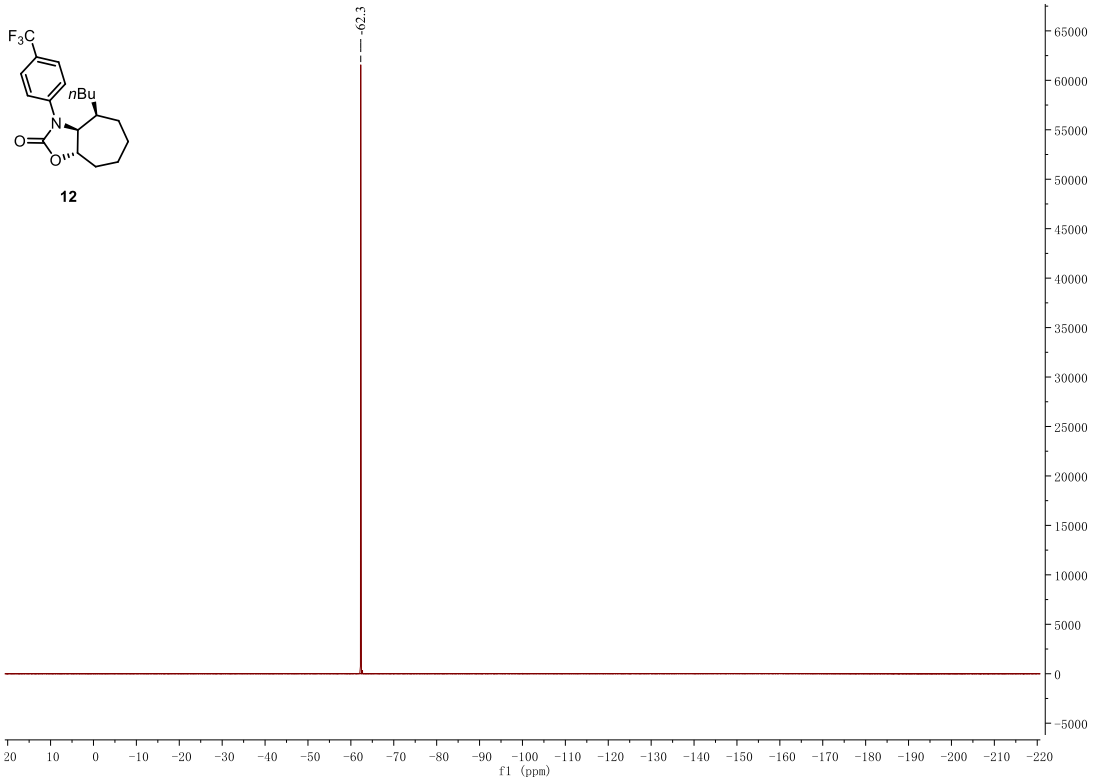
22.6
22.4
13.9
f1 (ppm)

Compound 12



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60000
55000
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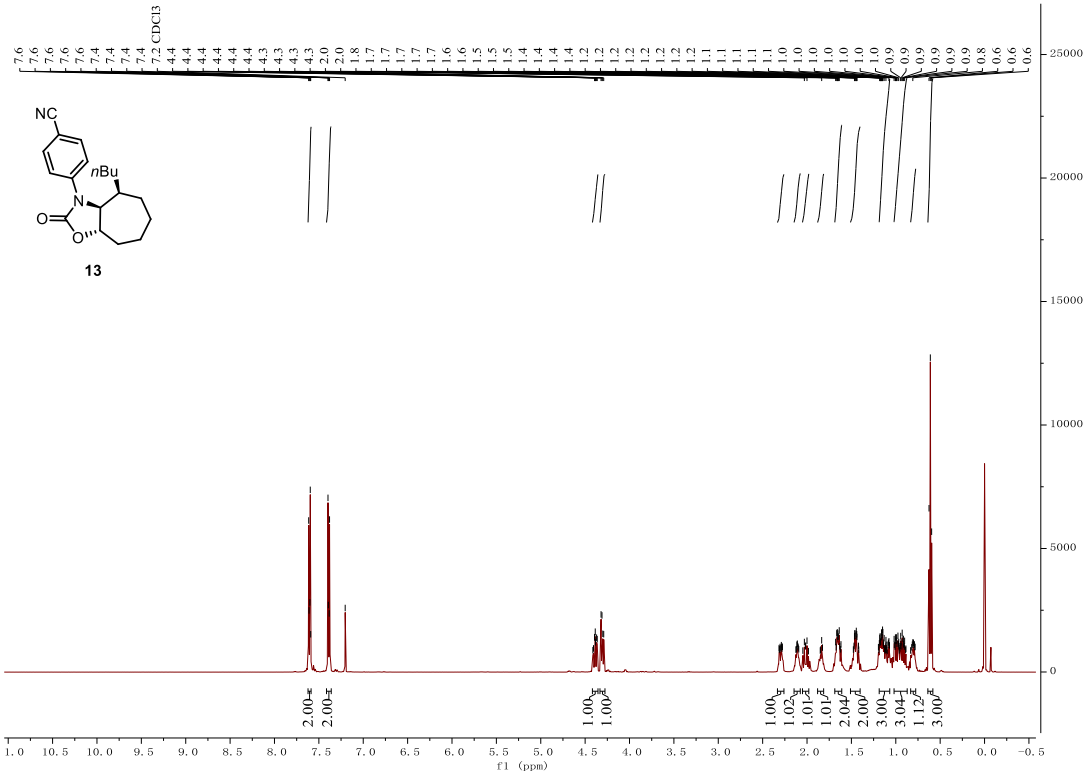
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

Compound 13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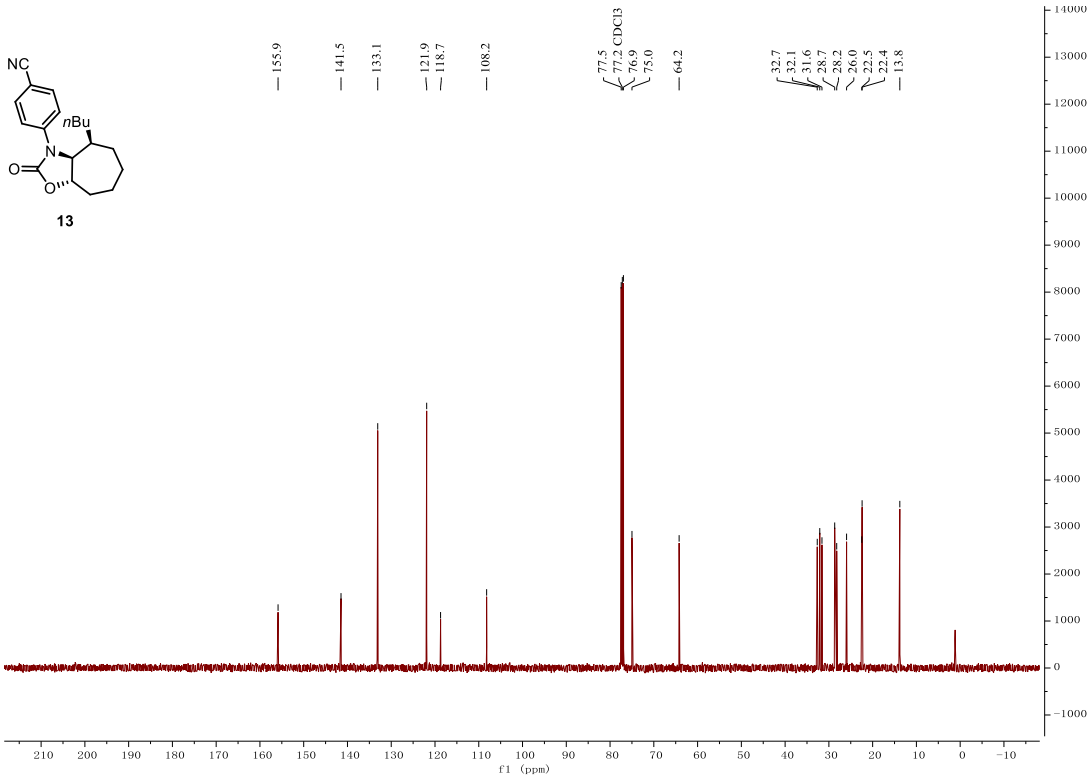
O
O
13
7.2 CDCl3
f1 (ppm)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76.9
75.0
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31.6
28.7
28.2
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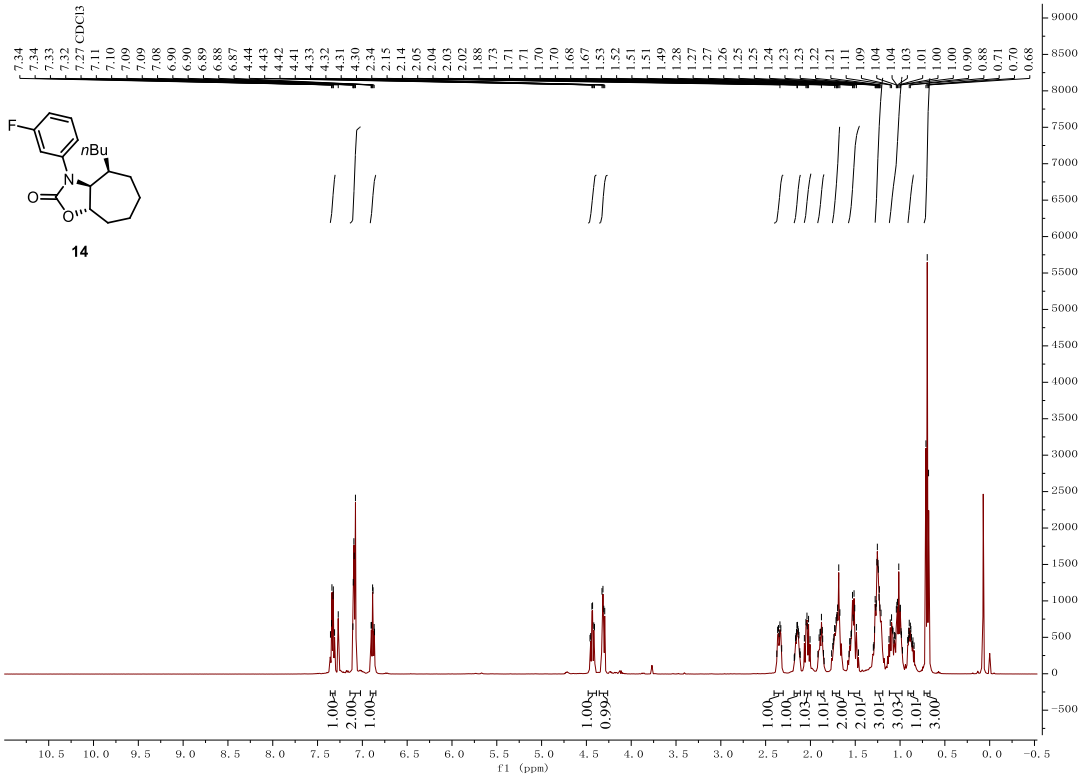
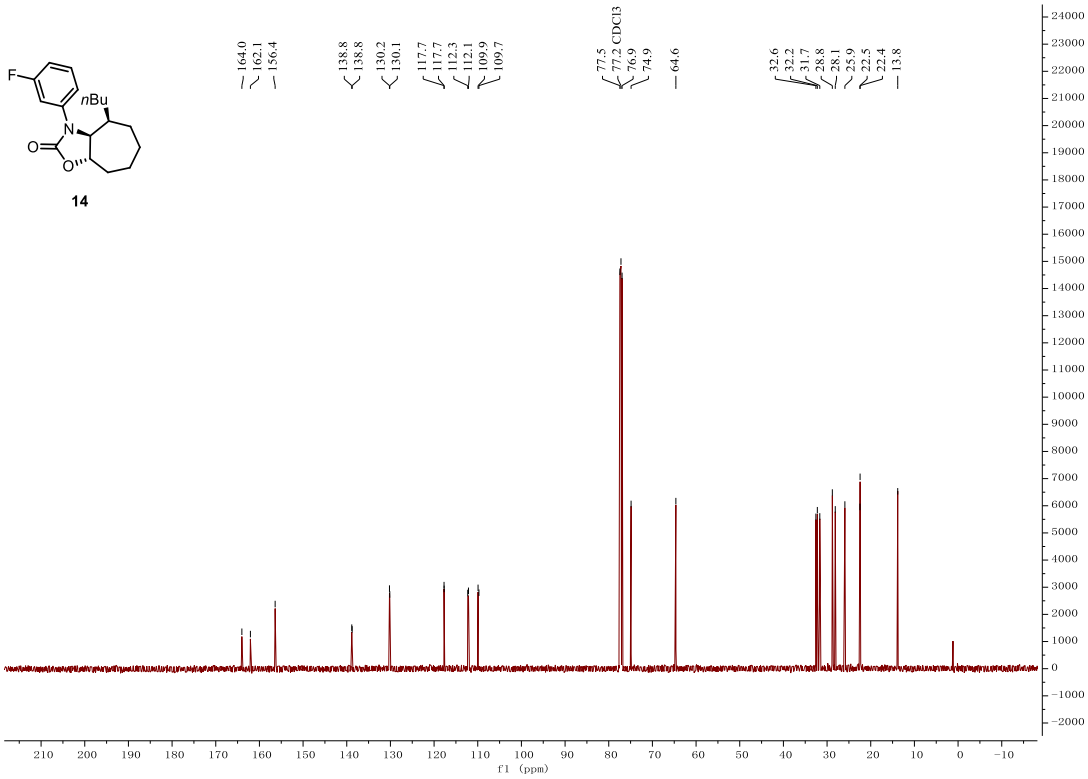
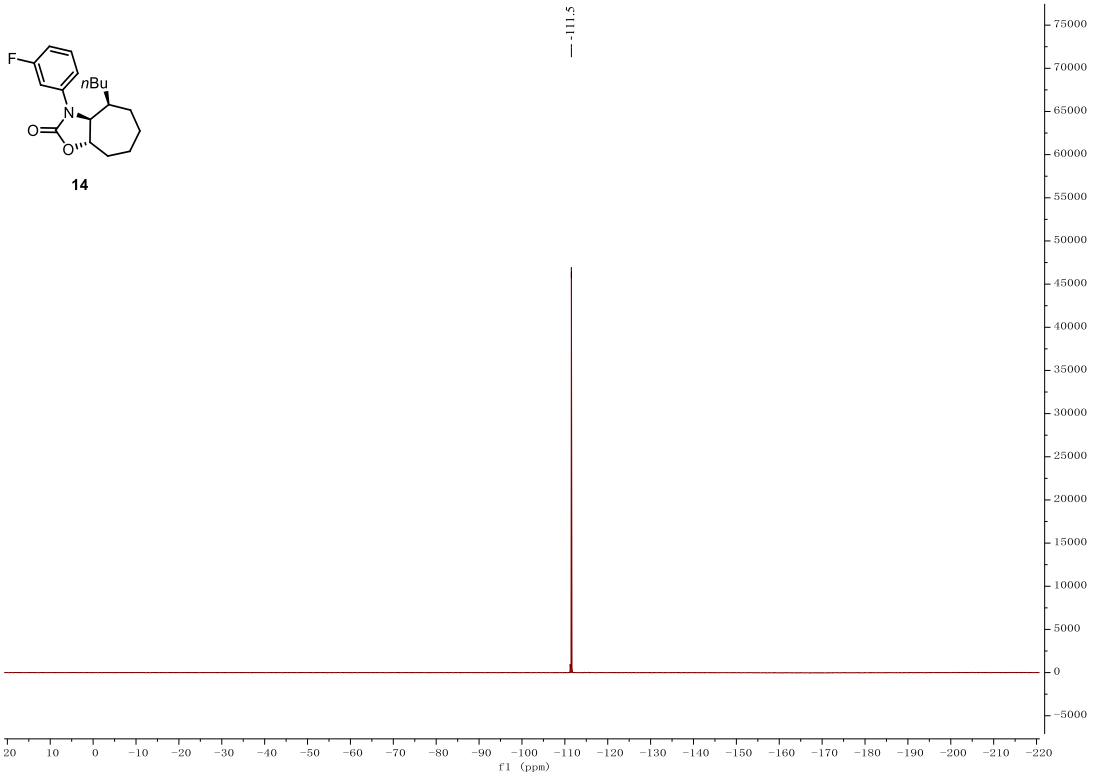
22.4
13.8
f1 (ppm)

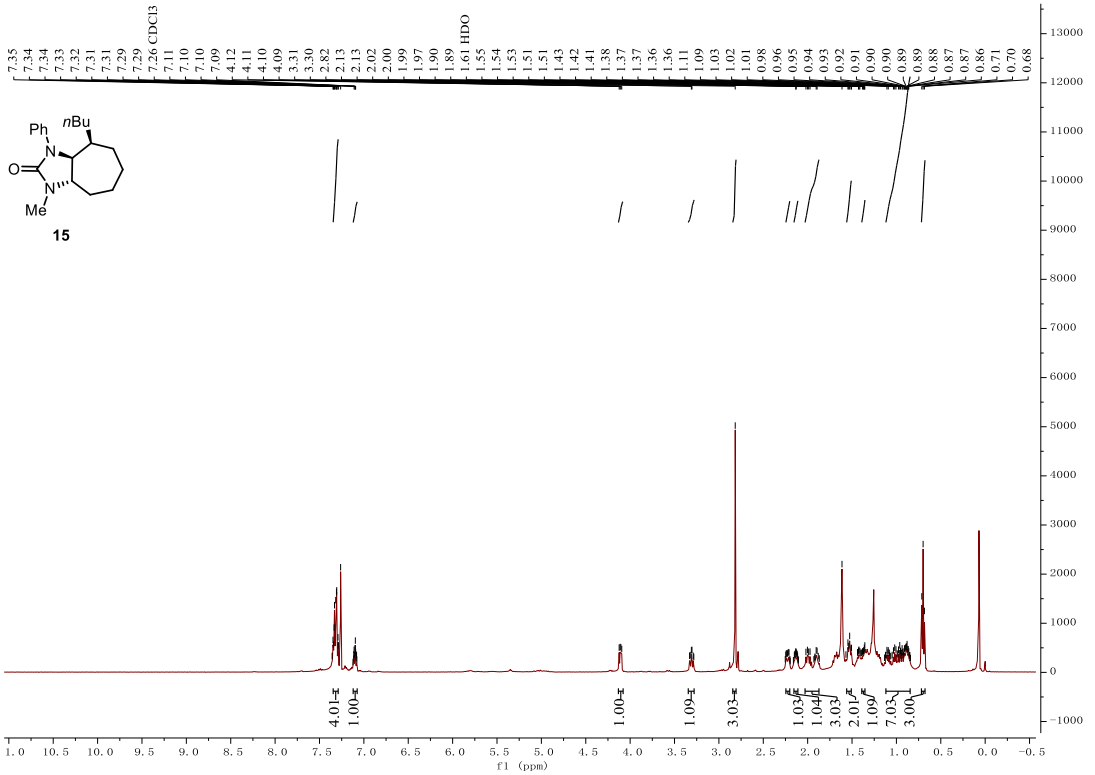
Compound 14

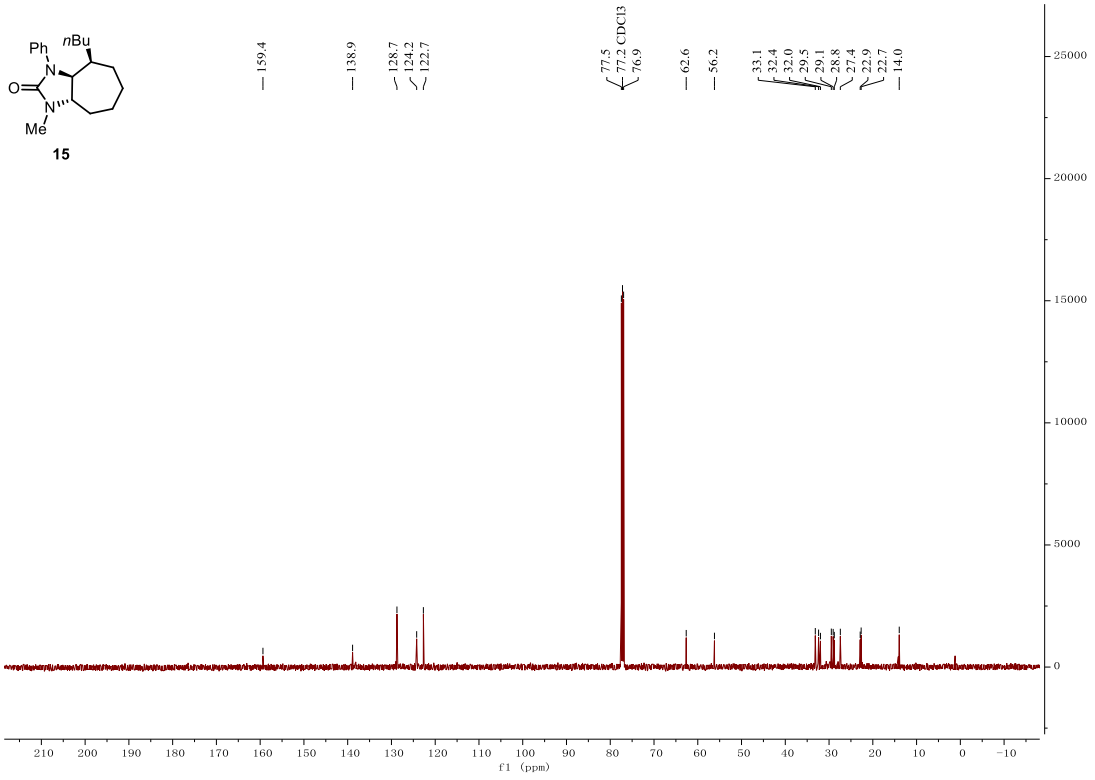




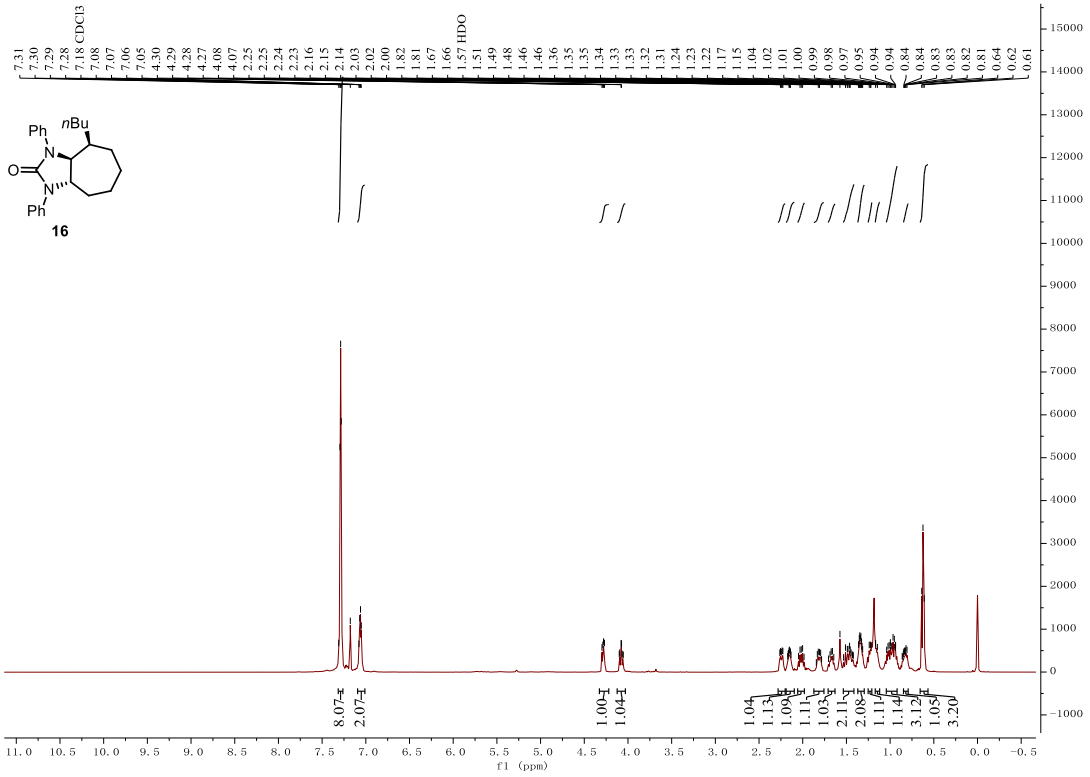

F
nBu
N
O
O
14
-111.5
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

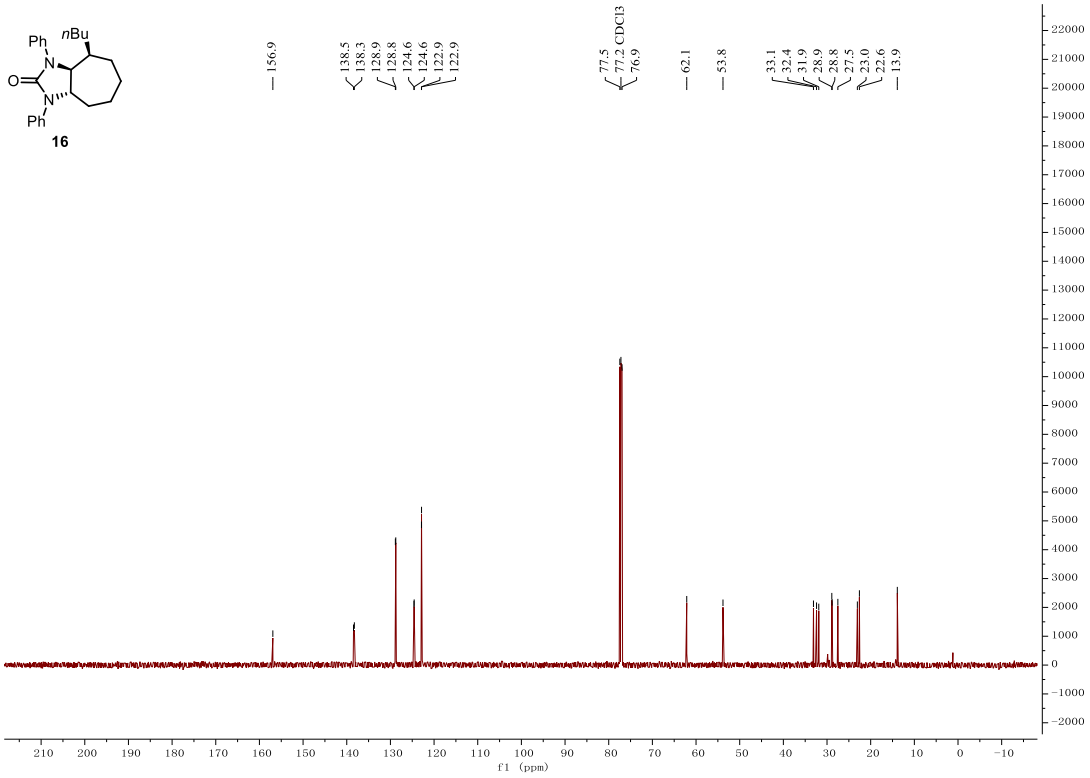
Compound 15


Ph
nBu
N
O
N
Me
15
7.35 7.34 7.34 7.33 7.32 7.31 7.31 7.29 7.29 7.26 CDCl3 7.11 7.10 7.10 7.09 4.12 4.11 4.10 4.09 3.31 3.30 2.82 2.13 2.02 2.00 1.99 1.97 1.90 1.89 1.61 HDO 1.55 1.54 1.53 1.51 1.51 1.43 1.42 1.41 1.38 1.37 1.37 1.36 1.36 1.11 1.09 1.03 1.02 1.01 0.98 0.96 0.95 0.94 0.93 0.92 0.91 0.90 0.90 0.89 0.89 0.88 0.87 0.87 0.86 0.71 0.70 0.68
4.01 1.00 1.00 1.09 3.03 1.03 1.04 3.03 2.01 1.09 7.03 3.00
f1 (ppm)

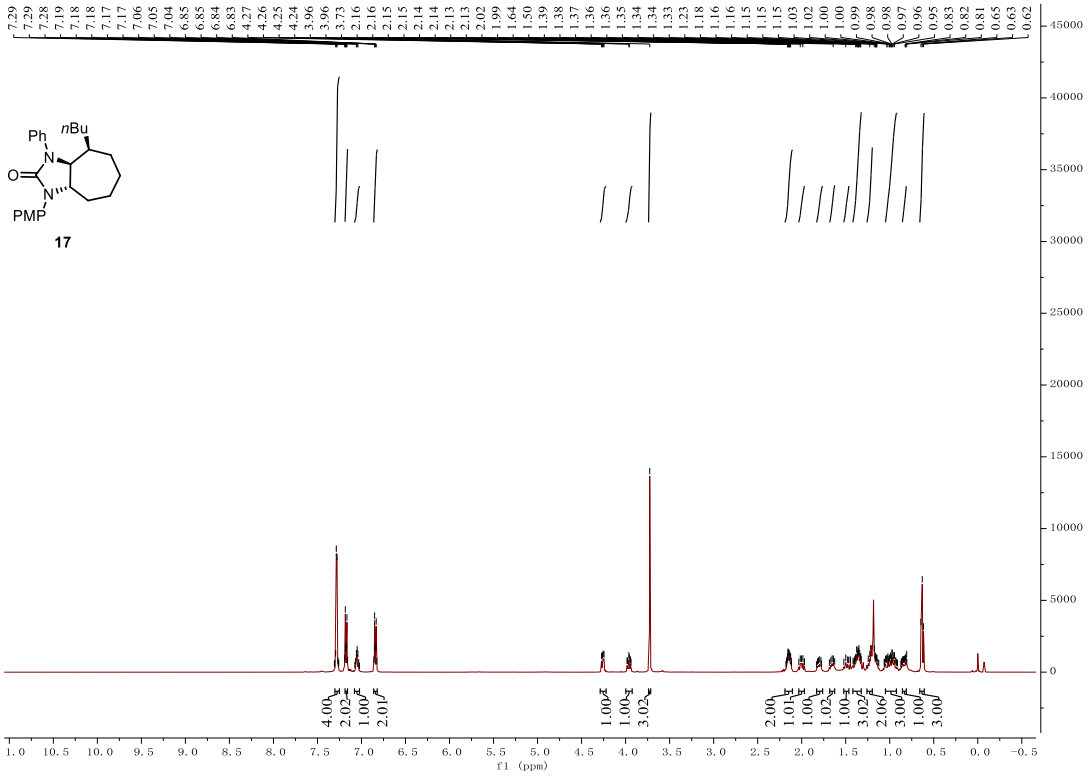

Ph
nBu
N
O
N
Me
15
159.4 138.9 128.7 124.2 122.7 77.5 77.2 CDCl3 76.9 62.6 56.2 33.1 32.4 32.0 29.5 29.1 28.8 27.4 22.9 22.7 14.0
f1 (ppm)

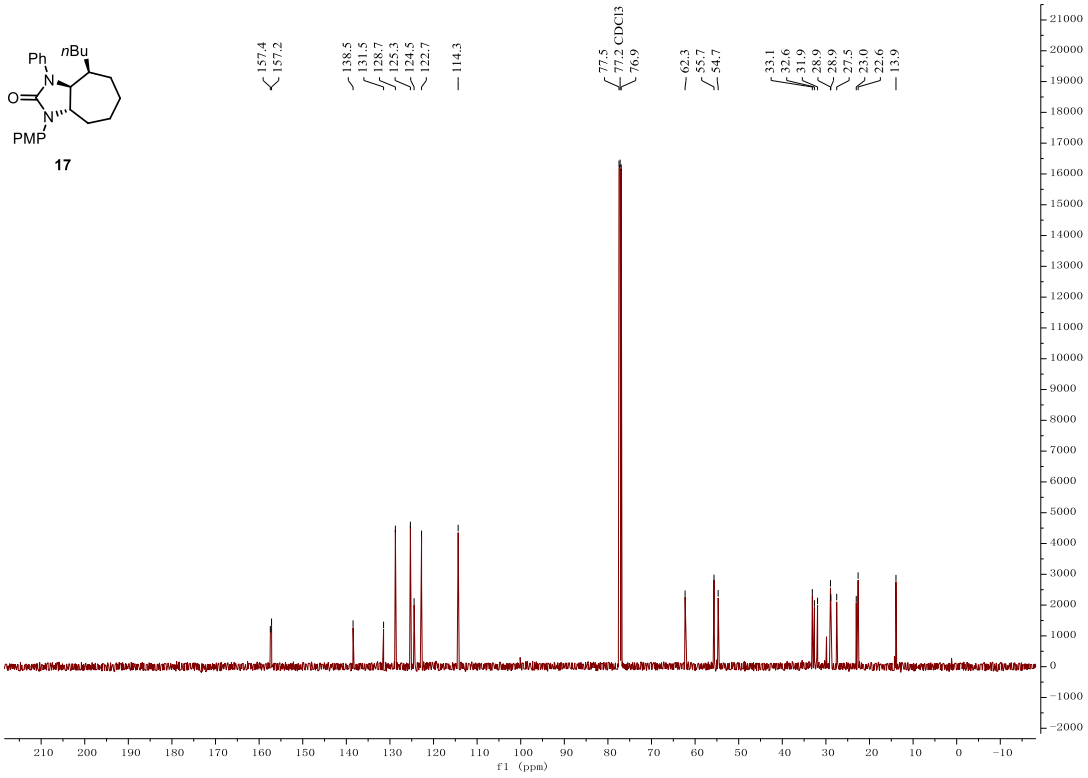
Compound 16


Ph
nBu
N
O
N
Ph
16
7.31 7.30 7.29 7.28 7.18 CDCl3 7.08 7.07 7.06 7.05 4.30 4.29 4.28 4.27 4.08 4.07 2.25 2.25 2.24 2.23 2.16 2.15 2.14 2.03 2.02 2.00 1.82 1.81 1.67 1.66 1.57 HDO 1.51 1.49 1.48 1.46 1.46 1.36 1.35 1.35 1.34 1.33 1.33 1.32 1.31 1.24 1.23 1.22 1.17 1.15 1.04 1.02 1.01 1.00 0.99 0.98 0.97 0.95 0.94 0.94 0.84 0.84 0.83 0.83 0.82 0.81 0.64 0.62 0.61
8.07 2.07 1.00 1.04 1.04 1.13 1.09 1.11 1.03 2.11 2.08 1.11 1.14 3.12 1.05 3.20
f1 (ppm)

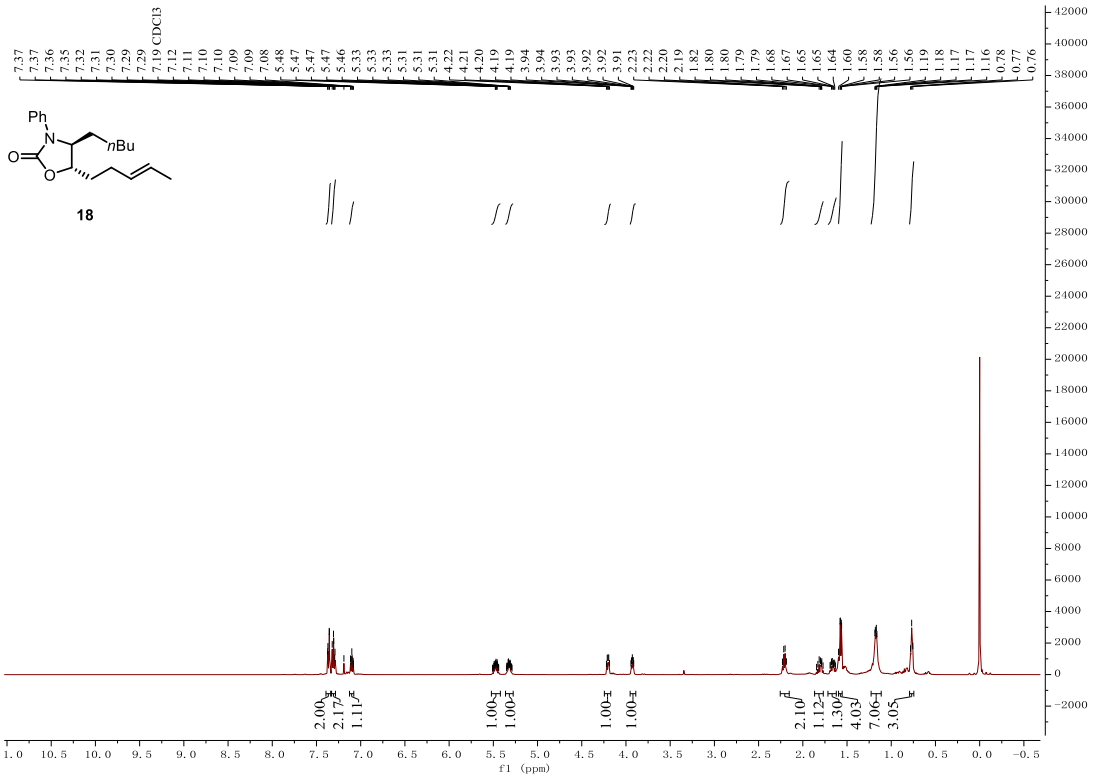

Ph
nBu
N
O
N
Ph
16
156.9
138.5 138.3 128.9 128.8 124.6 124.6 122.9 122.9
77.5 77.2 CDCl3 76.9
62.1
53.8
33.1 32.4 31.9 28.9 28.8 27.5 23.0 22.6
13.9
f1 (ppm)

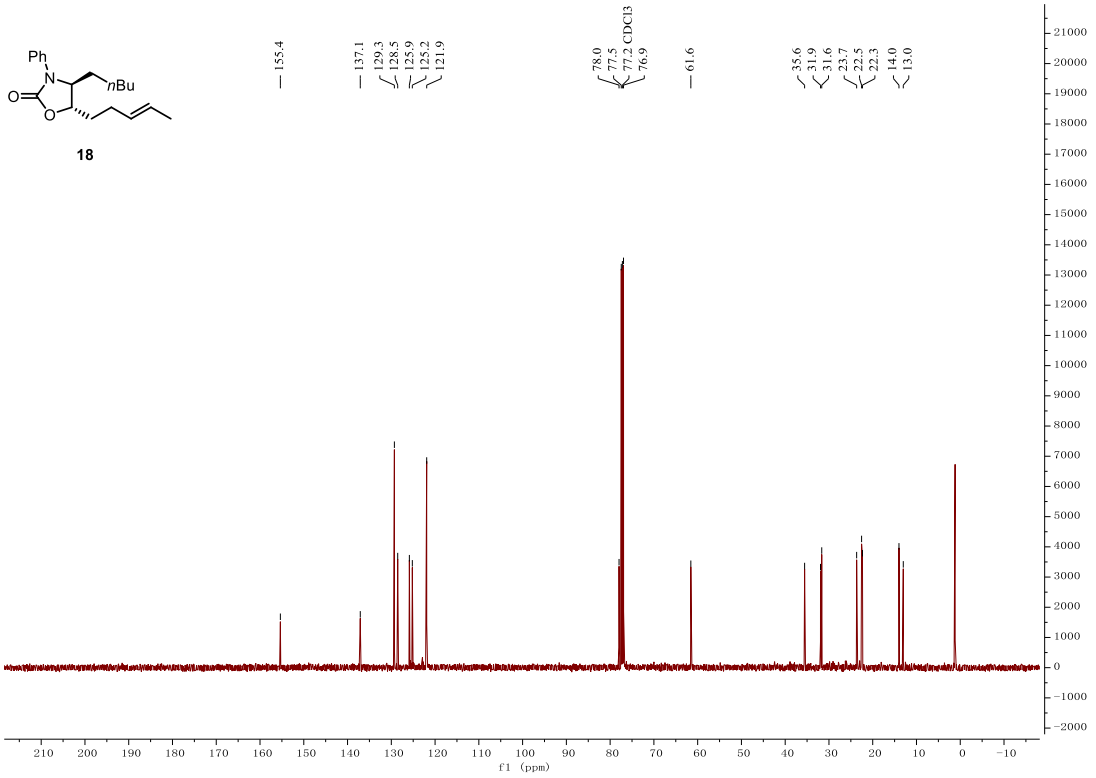
Compound 17


Ph
nBu
O
N
N
PMP
17
f1 (ppm)

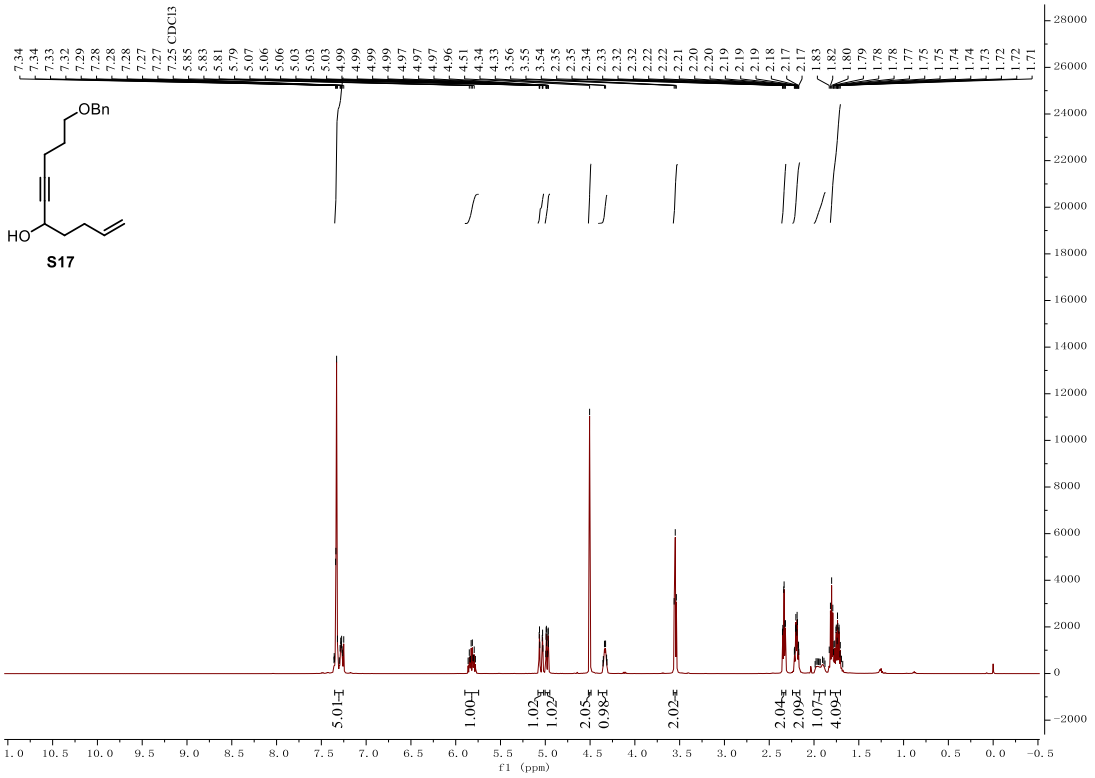

Ph
nBu
O
N
N
PMP
17
157.4
157.2
138.5
131.5
128.7
125.3
124.5
122.7
114.3
77.5
77.2 CDCl3
76.9
62.3
55.7
54.7
33.1
32.6
31.9
28.9
28.9
27.5
23.0
22.6
13.9
f1 (ppm)

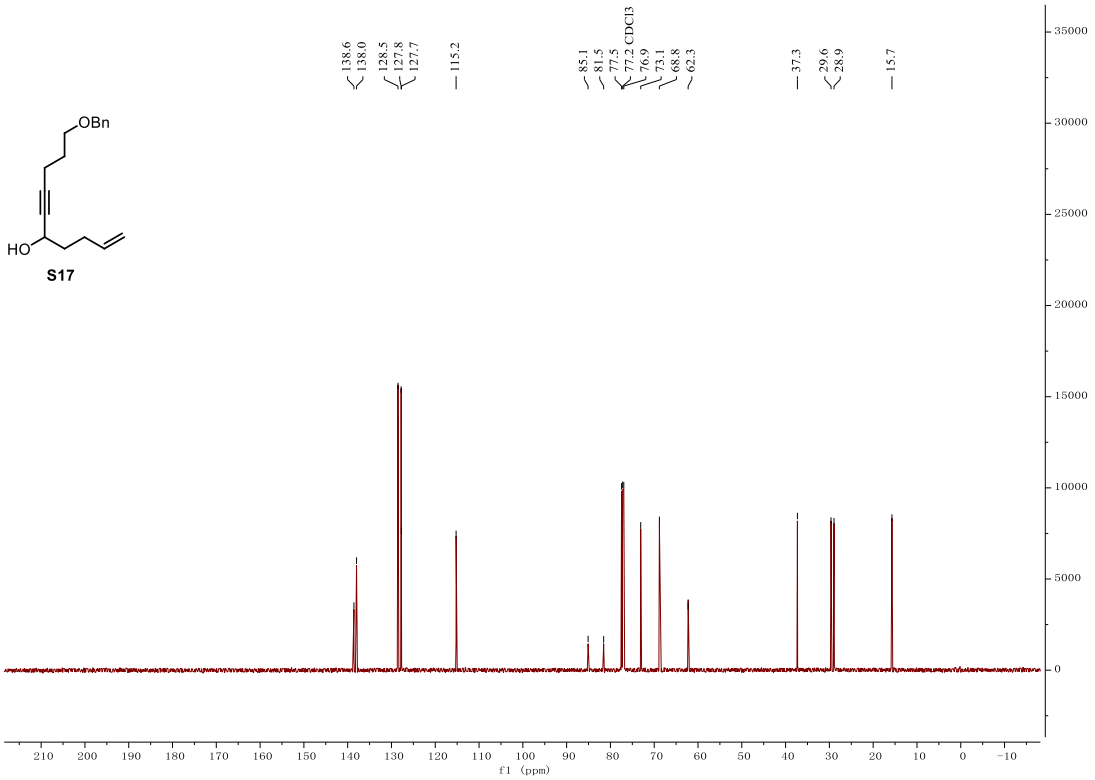
Compound 18



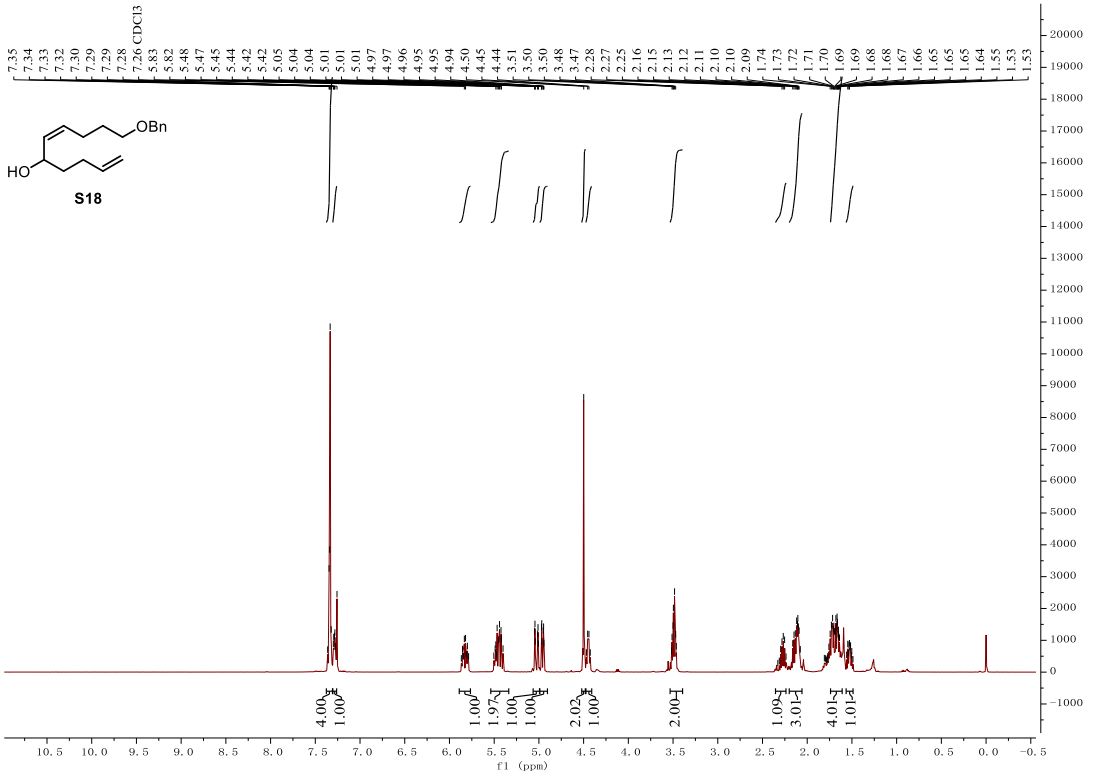


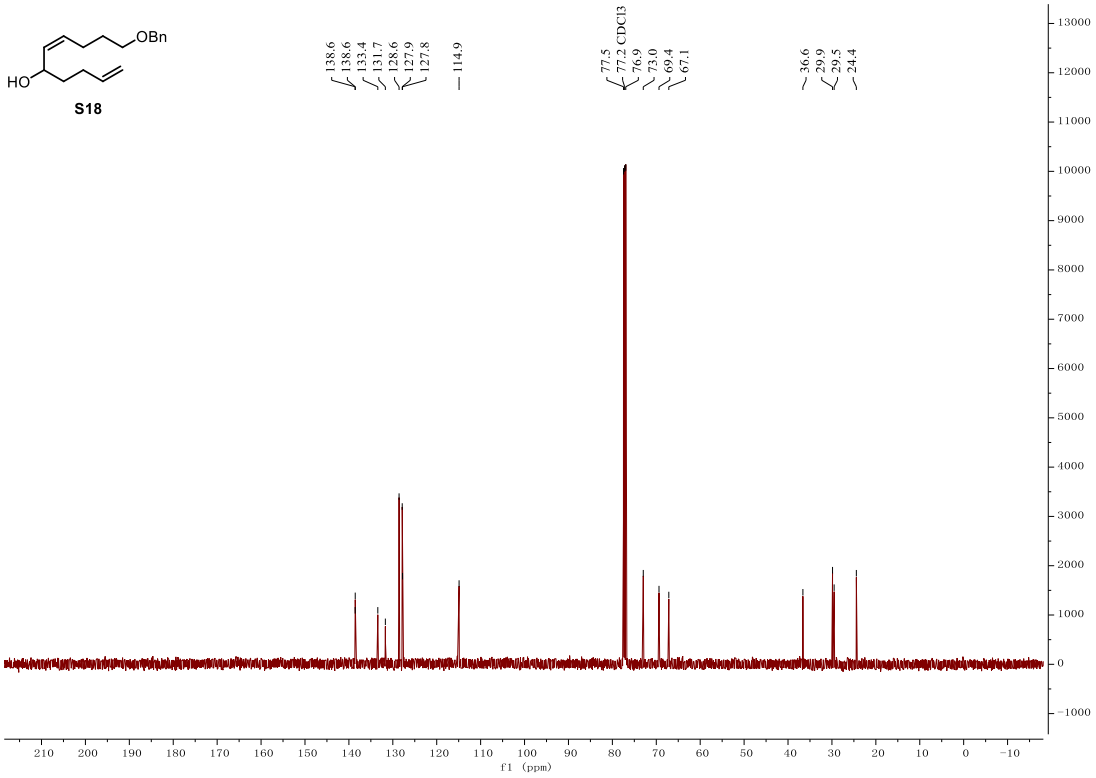
Compound S17



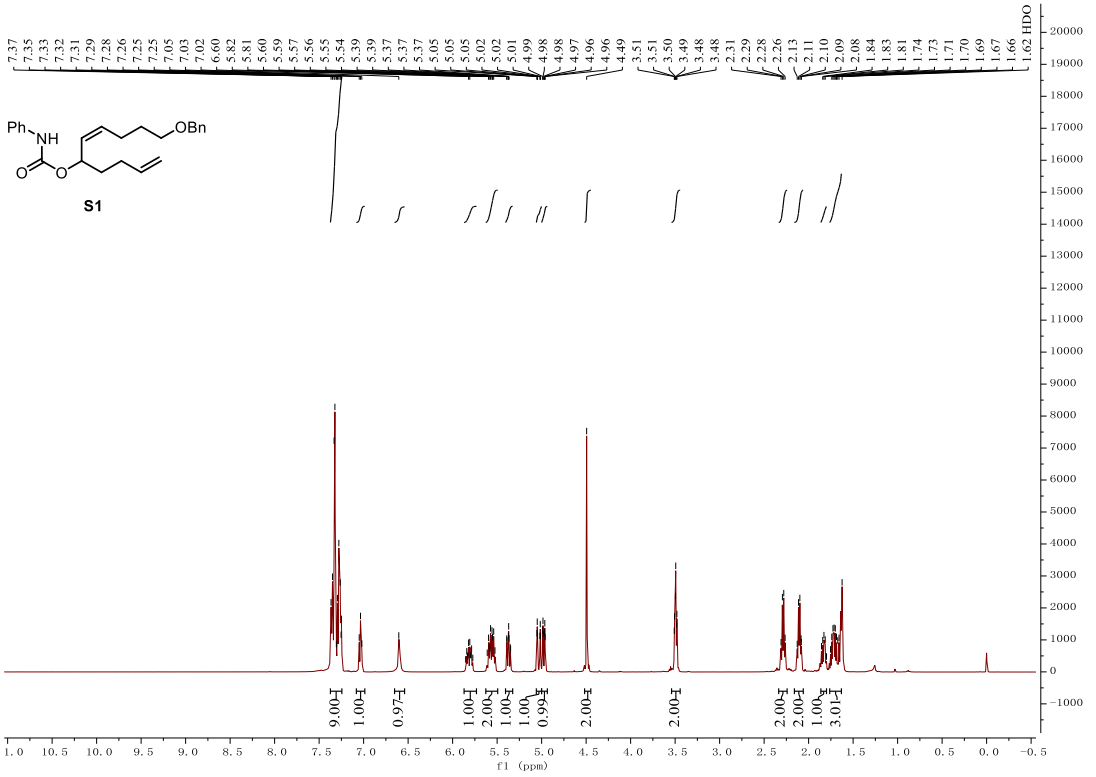


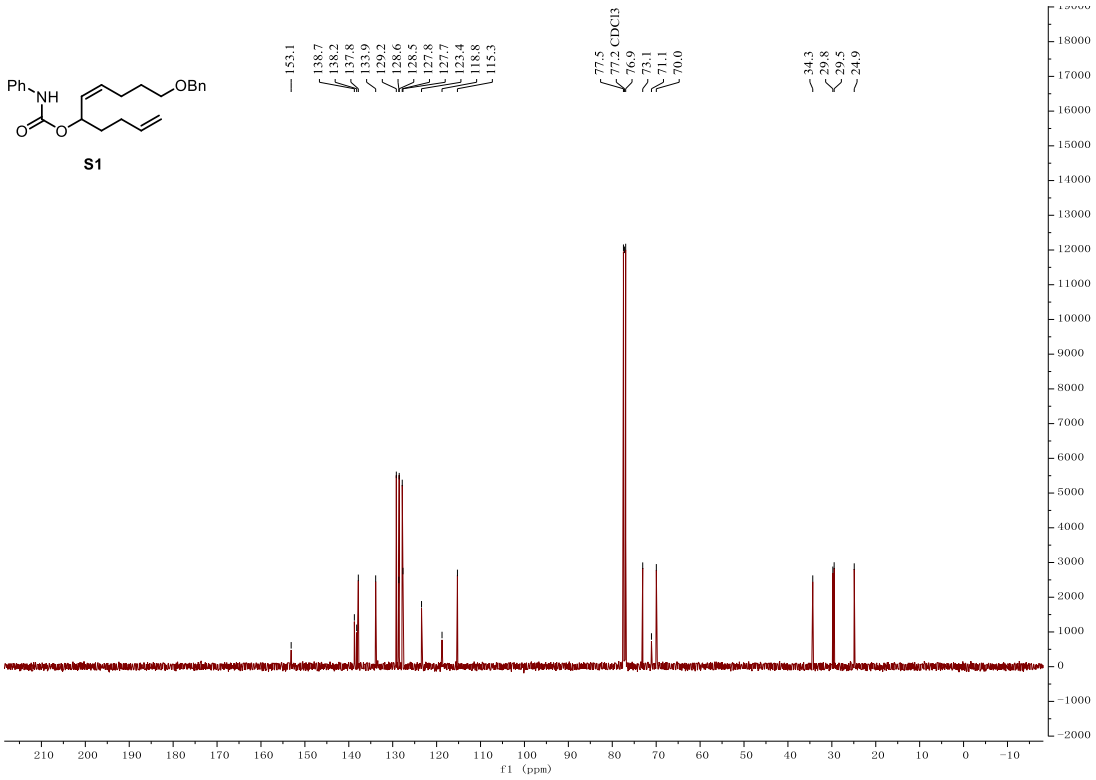
Compound S18



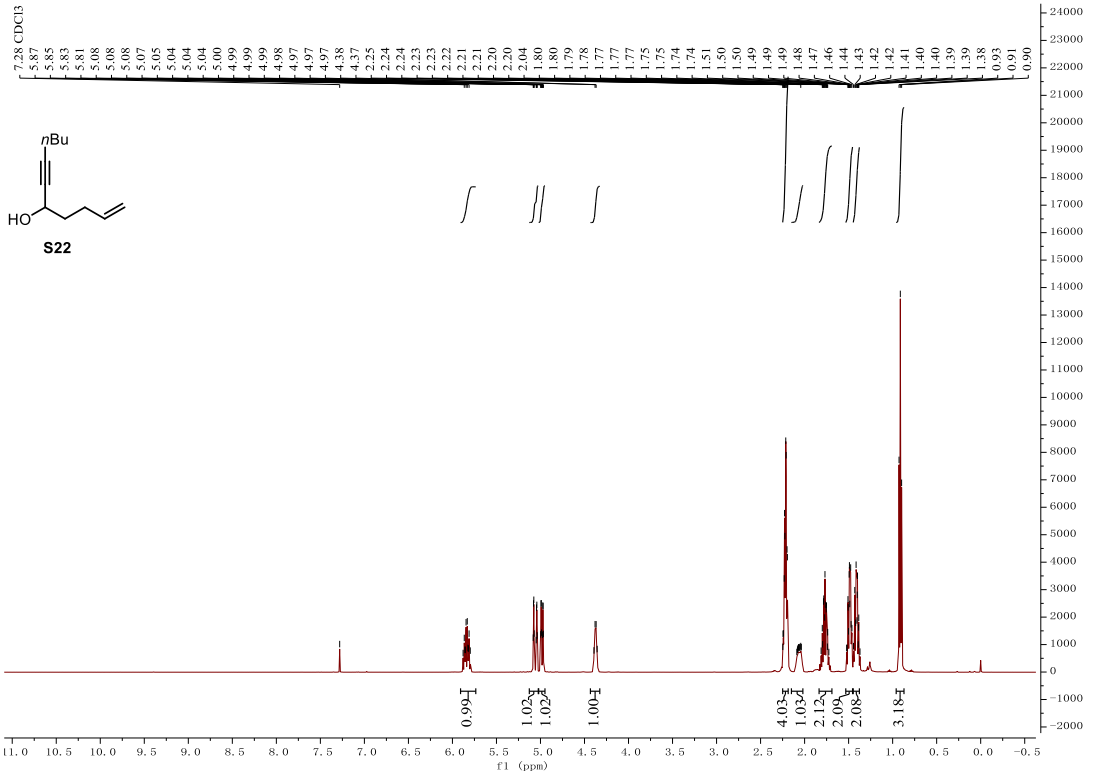


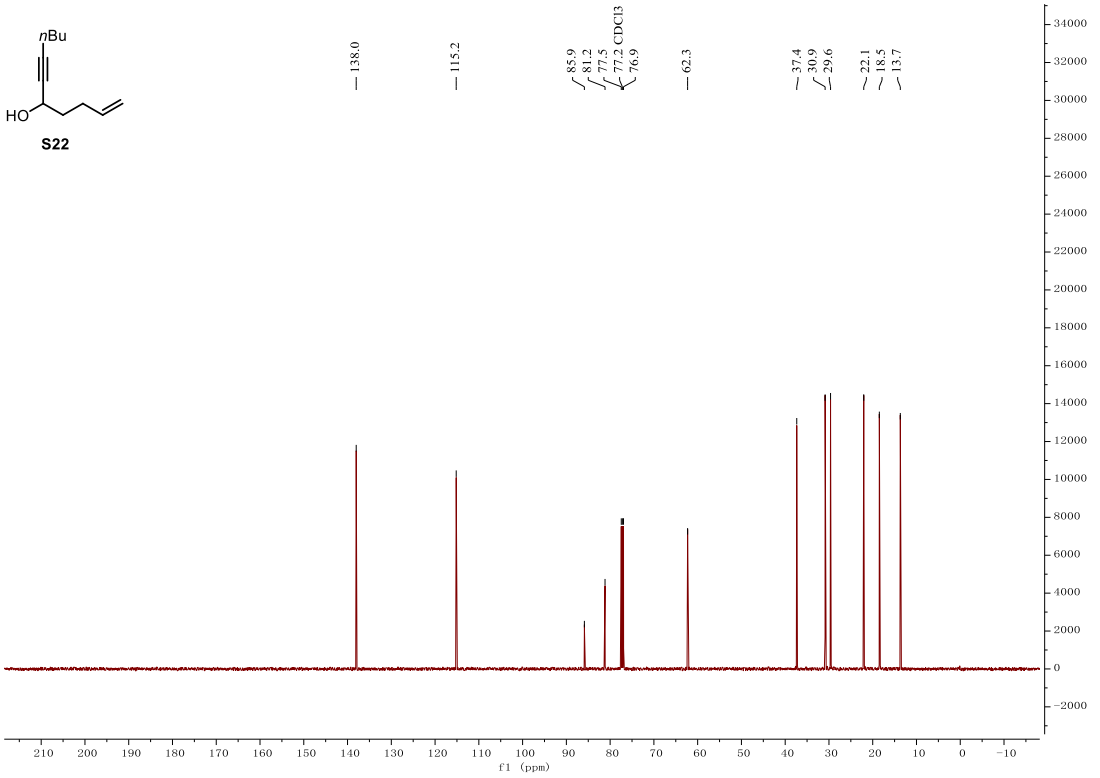
Compound S1


Ph
NH
O
O
OBn
S1
7.37 7.35 7.33 7.32 7.31 7.29 7.28 7.26 7.25 7.25 7.05 7.03 7.02 6.60 5.82 5.81 5.60 5.59 5.57 5.56 5.55 5.54 5.39 5.39 5.37 5.37 5.05 5.05 5.02 5.02 5.01 4.99 4.98 4.98 4.97 4.96 4.96 4.49 3.51 3.51 3.50 3.49 3.48 3.48 2.31 2.29 2.28 2.26 2.13 2.11 2.10 2.09 2.08 1.84 1.83 1.81 1.74 1.73 1.71 1.70 1.69 1.67 1.66 1.62 HDO
9.00 1.00 0.97 1.00 2.00 1.00 1.00 0.99 2.00 2.00 2.00 2.00 1.00 3.01
f1 (ppm)

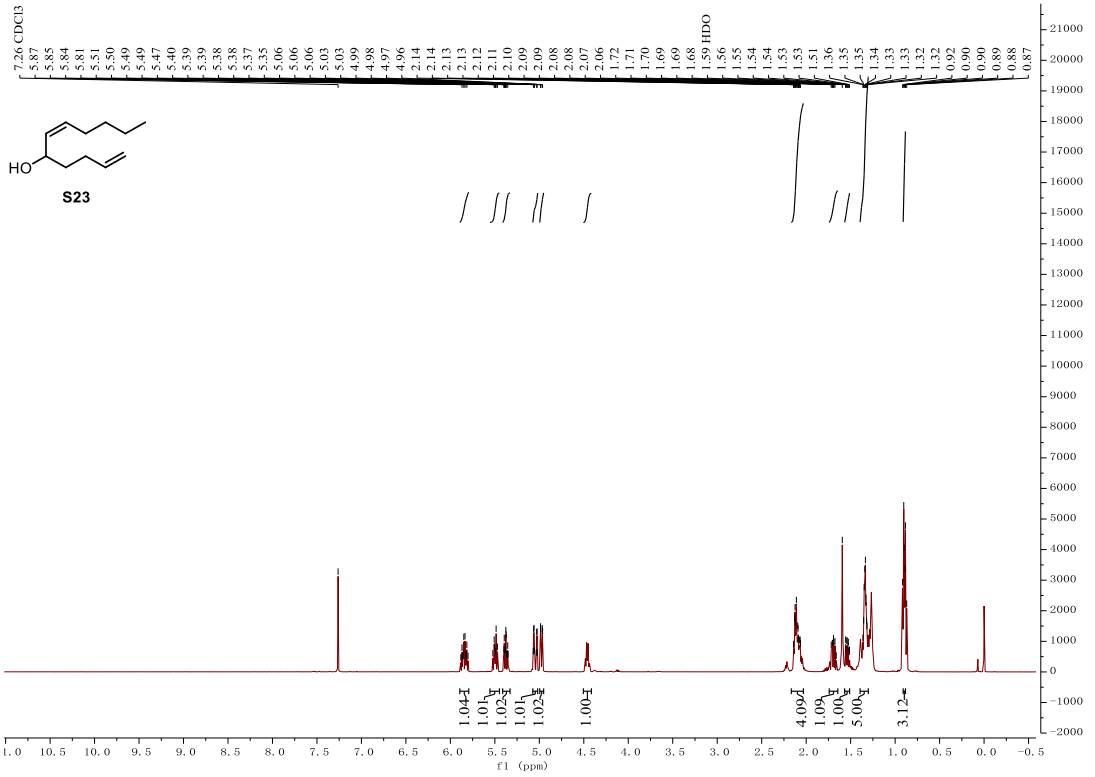

Ph
NH
O
O
OBn
S1
153.1
138.7 138.2 137.8 133.9 129.2 128.6 128.5 127.8 127.7 123.4 118.8 115.3
77.5 77.2 CDCl3 76.9 73.1 71.1 70.0
34.3 29.8 29.5 24.9
f1 (ppm)

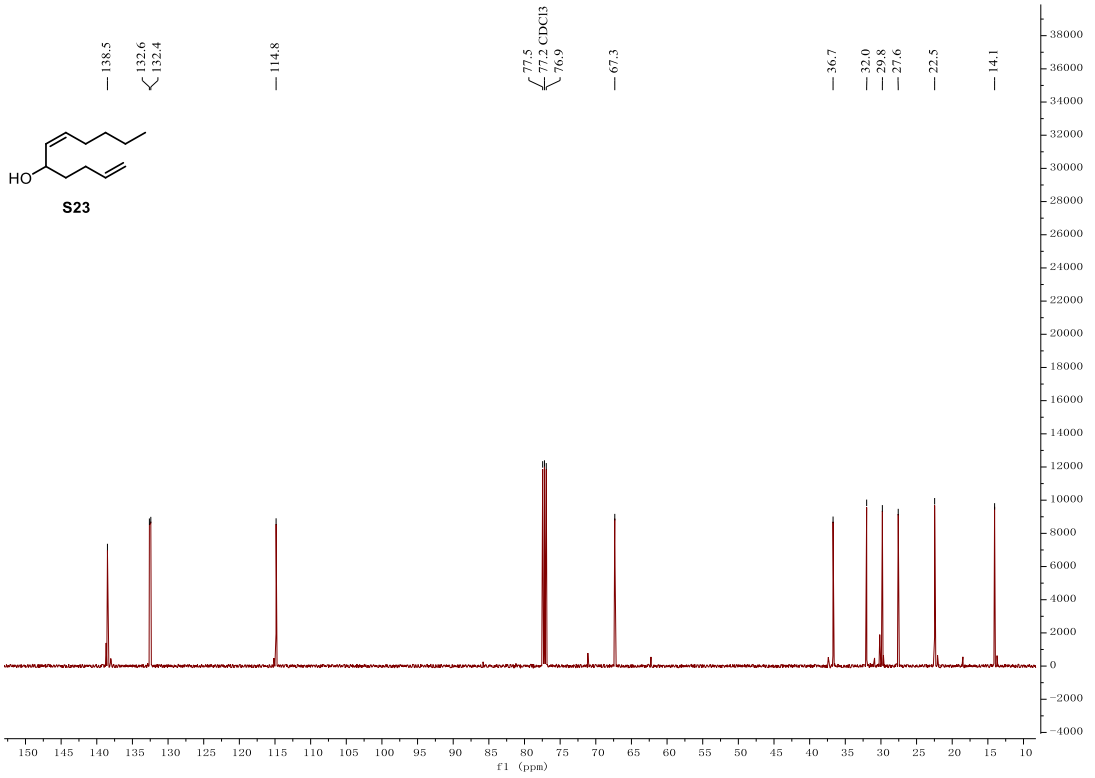
Compound S22



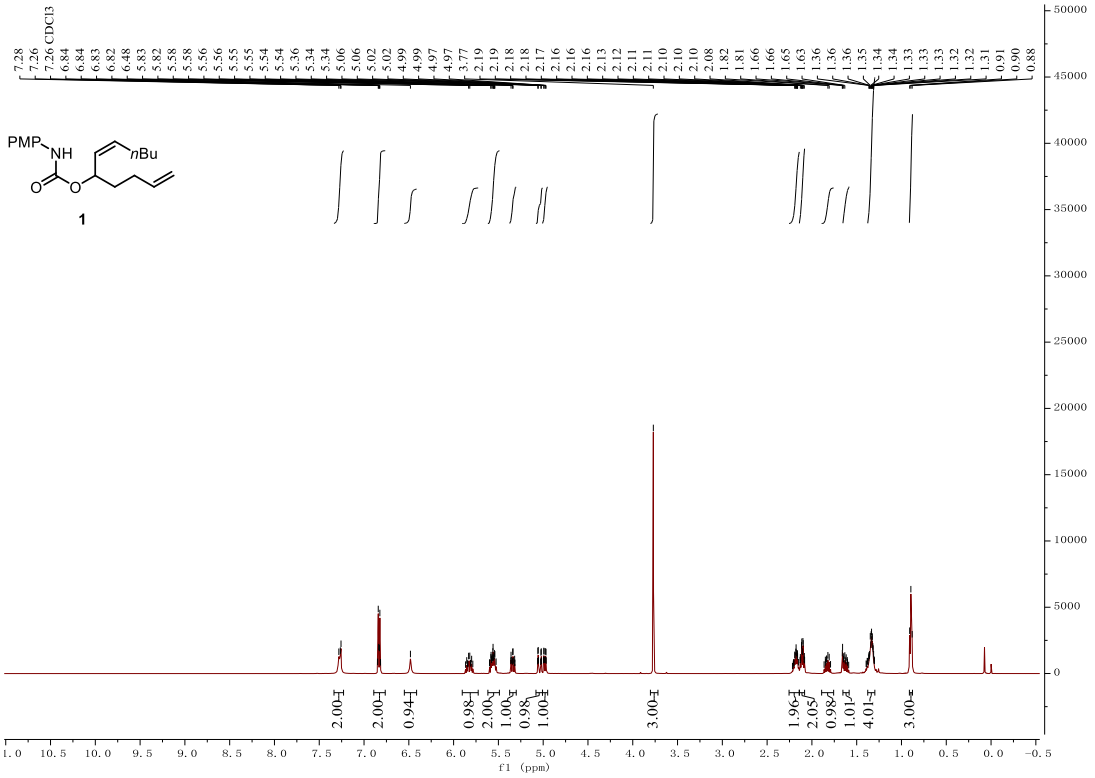


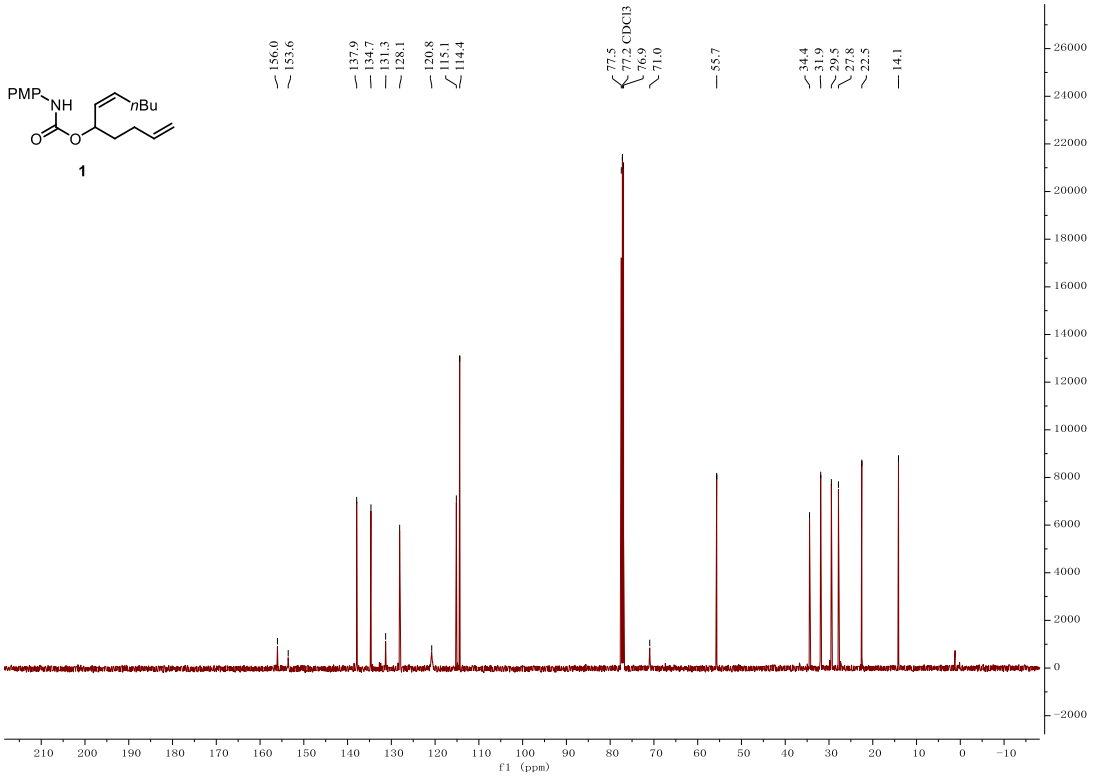
Compound S23



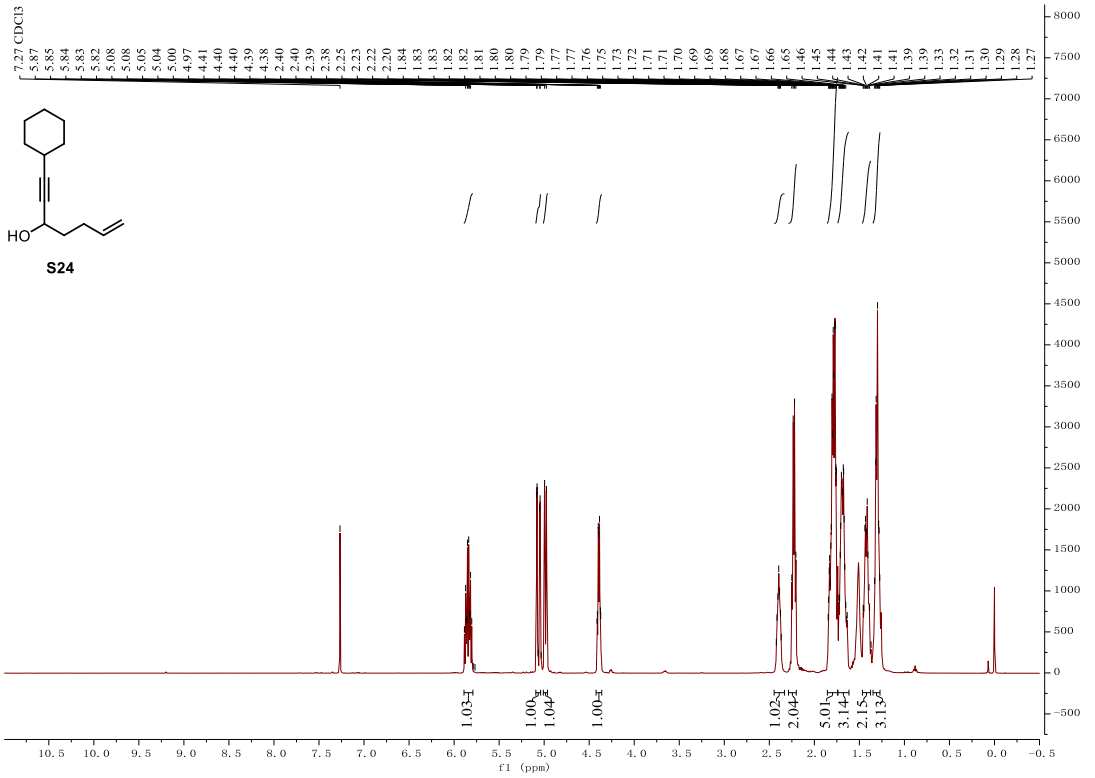


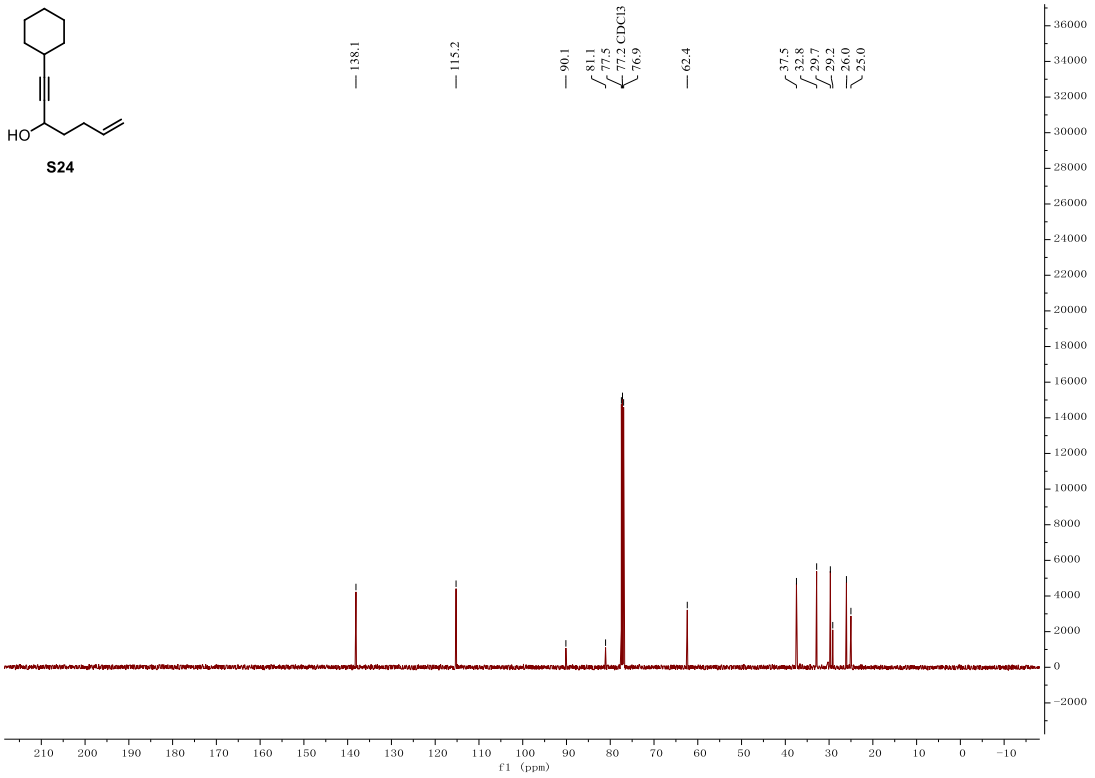
Compound 1


7.28
7.26
7.26 CDCl3
6.84
6.83
6.82
6.48
5.83
5.82
5.58
5.56
5.55
5.54
5.36
5.34
5.06
5.02
4.99
4.97
3.77
2.19
2.18
2.17
2.16
2.13
2.12
2.11
2.10
2.08
1.82
1.81
1.66
1.65
1.63
1.36
1.35
1.34
1.33
1.32
1.31
0.91
0.90
0.88
PMP
NH
O
O
nBu
1
2.00
2.00
0.94
0.98
2.00
1.00
0.98
1.00
3.00
1.96
2.05
0.98
1.01
4.01
3.00
f1 (ppm)

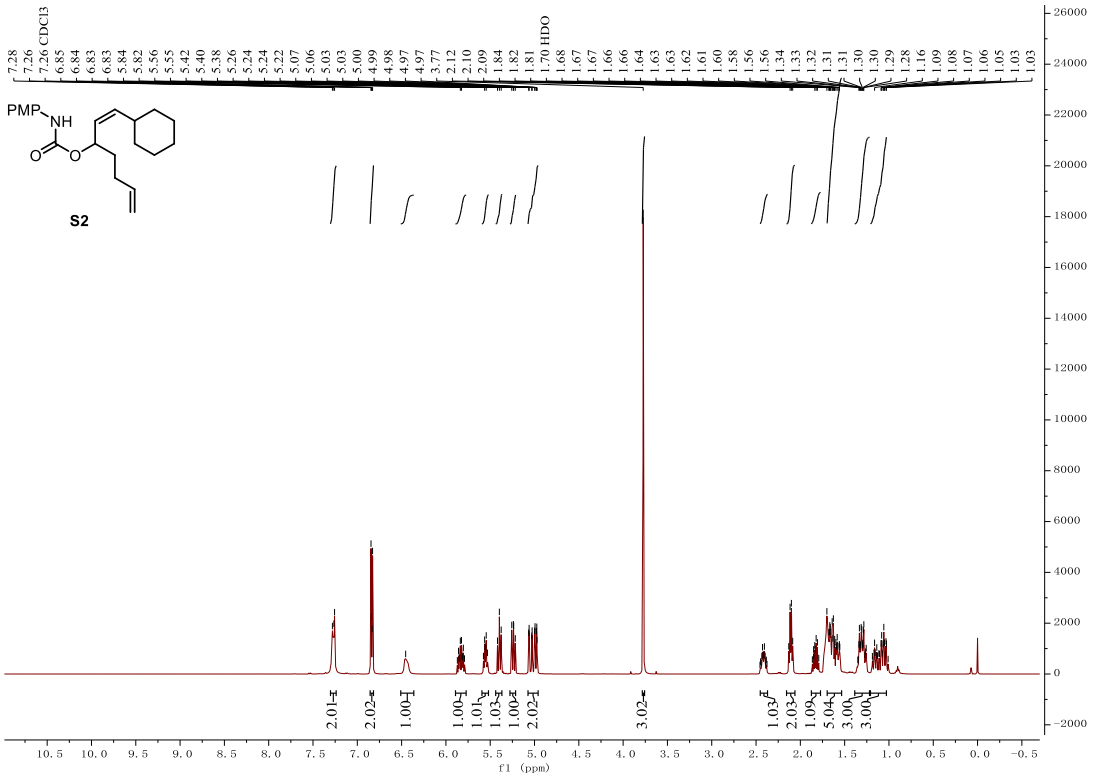

156.0
153.6
137.9
134.7
131.3
128.1
120.8
115.1
114.4
77.5
77.2 CDCl3
76.9
71.0
55.7
34.4
31.9
29.5
27.8
22.5
14.1
PMP
NH
O
O
nBu
1
f1 (ppm)

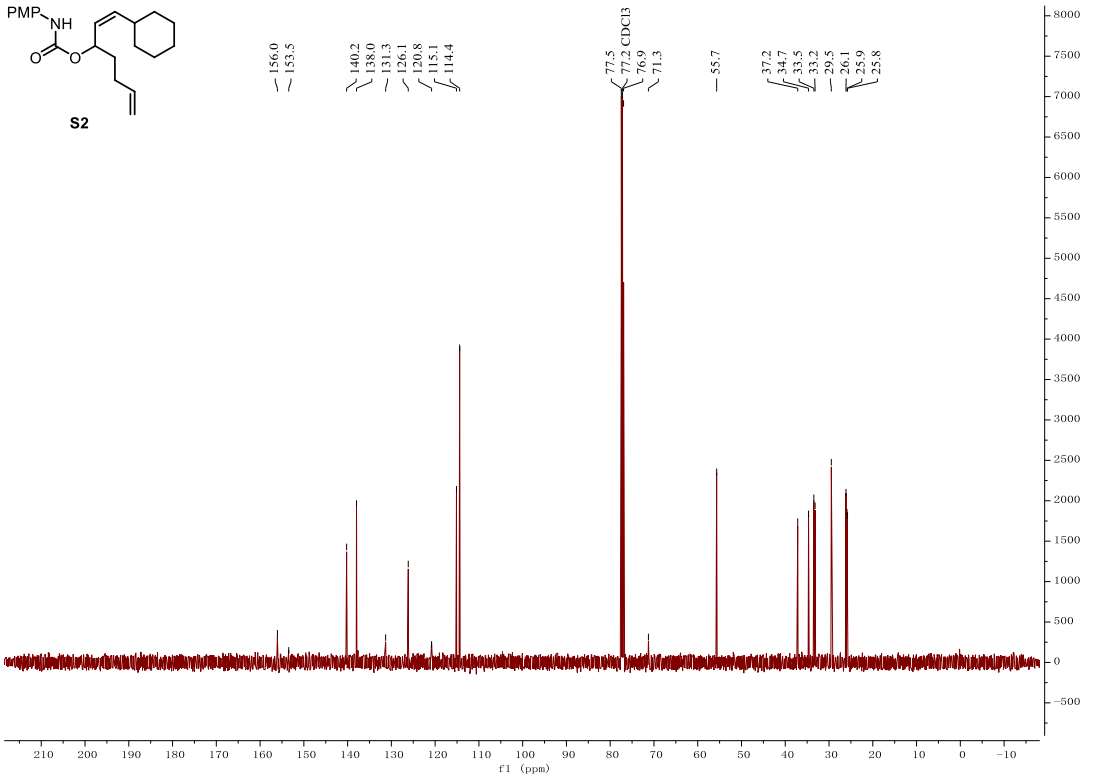
Compound S24



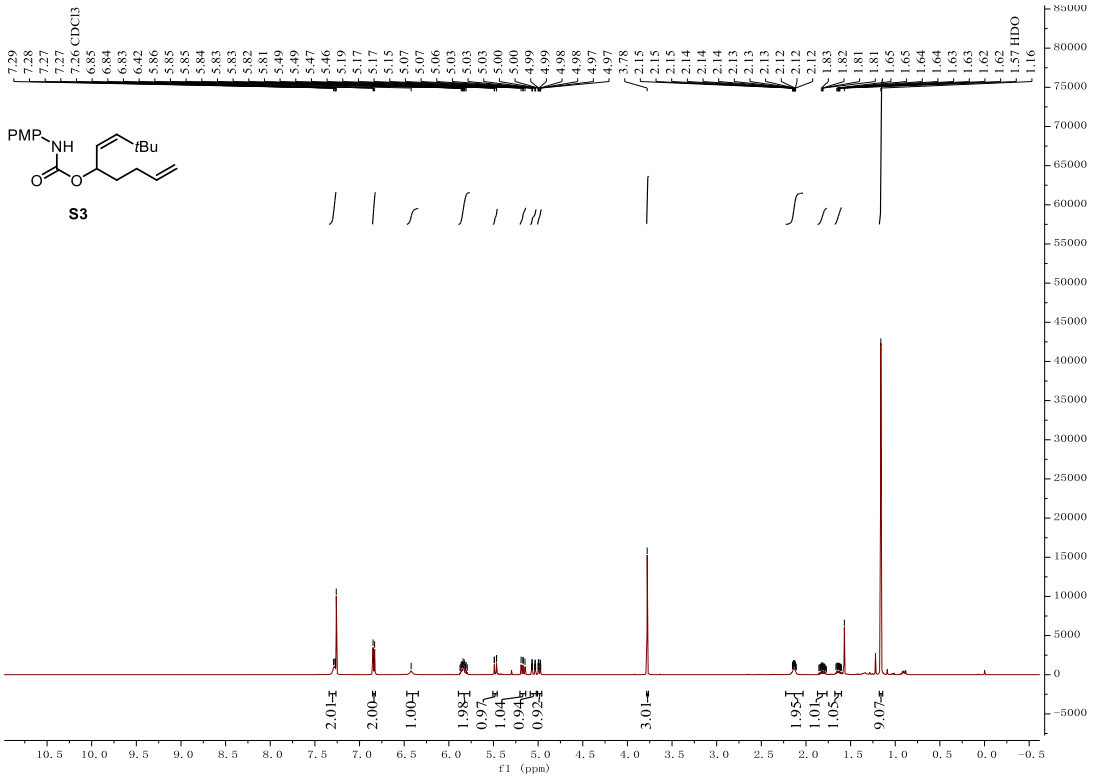


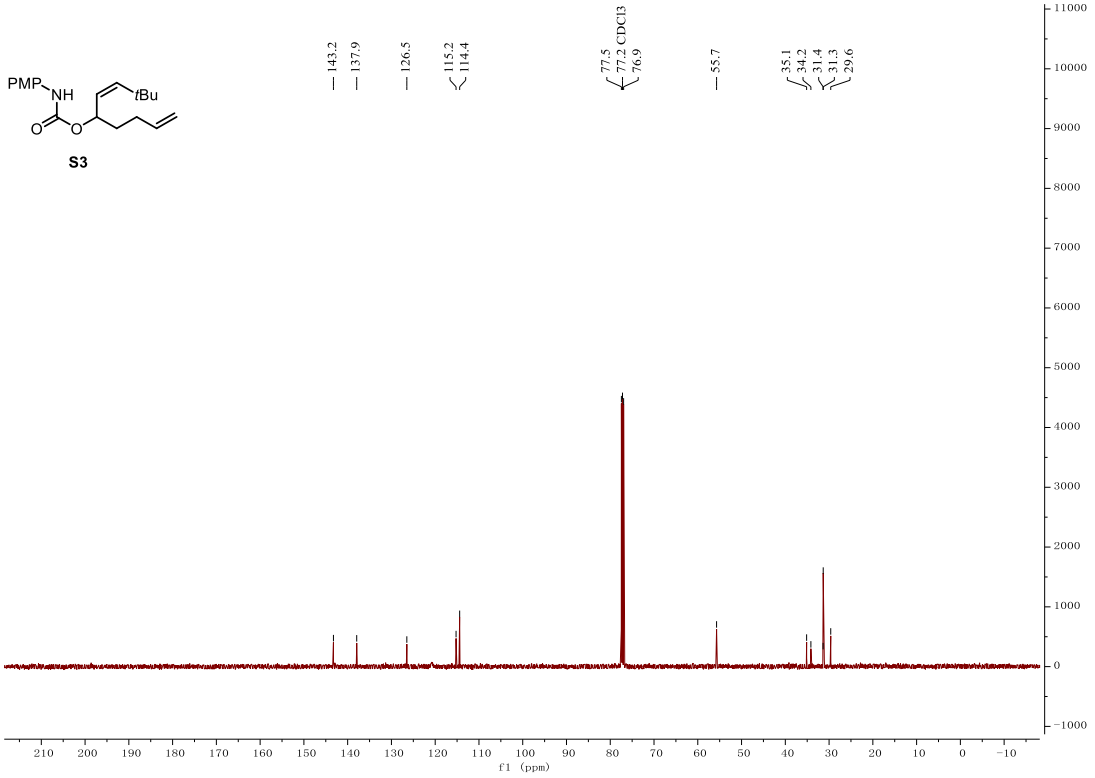
Compound S2


PMP
NH
O
O
S2
7.28
7.26
7.26 CDCl3
6.85
6.84
6.83
6.83
5.84
5.82
5.56
5.55
5.42
5.40
5.38
5.26
5.24
5.24
5.22
5.07
5.06
5.03
5.03
5.00
4.99
4.98
4.97
4.97
3.77
2.12
2.10
2.09
1.84
1.82
1.81
1.70 HDO
1.68
1.67
1.67
1.66
1.66
1.64
1.63
1.63
1.62
1.61
1.60
1.58
1.56
1.56
1.34
1.33
1.32
1.31
1.31
1.30
1.30
1.29
1.28
1.16
1.09
1.08
1.07
1.06
1.05
1.03
1.03
2.01
2.02
1.00
1.00
1.01
1.03
1.00
2.02
3.02
1.03
2.03
1.09
5.04
3.00
3.00
f1 (ppm)

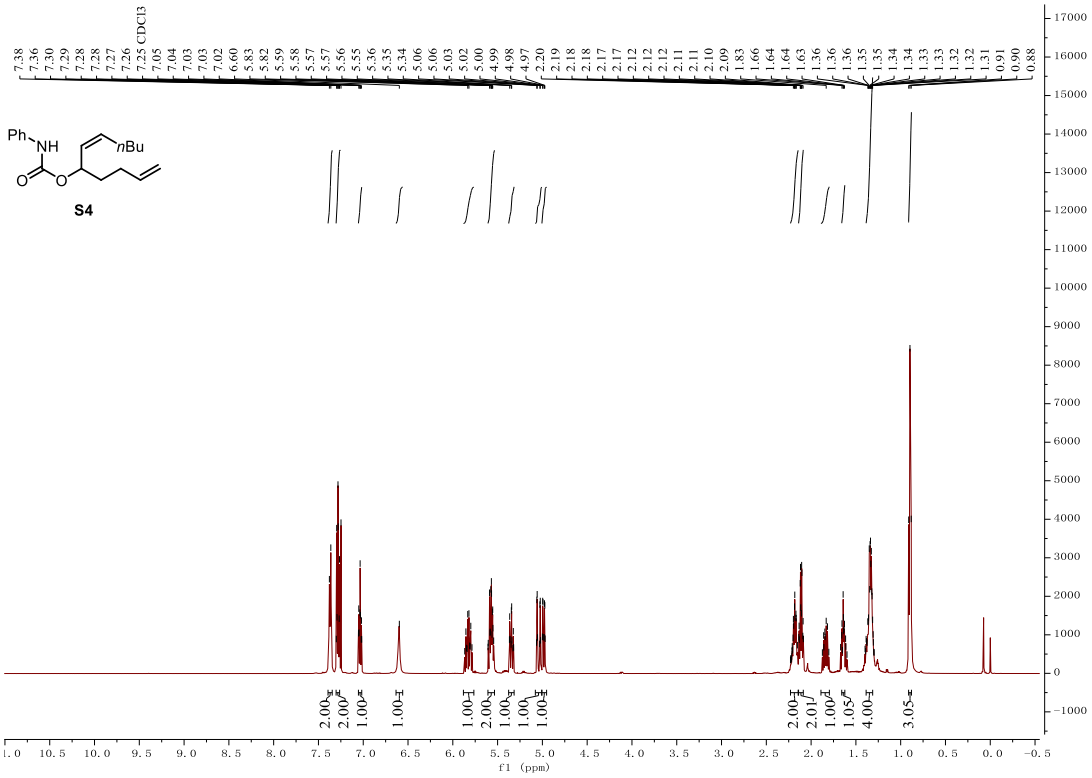

PMP
NH
O
O
S2
156.0
153.5
140.2
138.0
131.3
126.1
120.8
115.1
114.4
77.5
77.2 CDCl3
76.9
71.3
55.7
37.2
34.7
33.5
33.2
29.5
26.1
25.9
25.8
f1 (ppm)

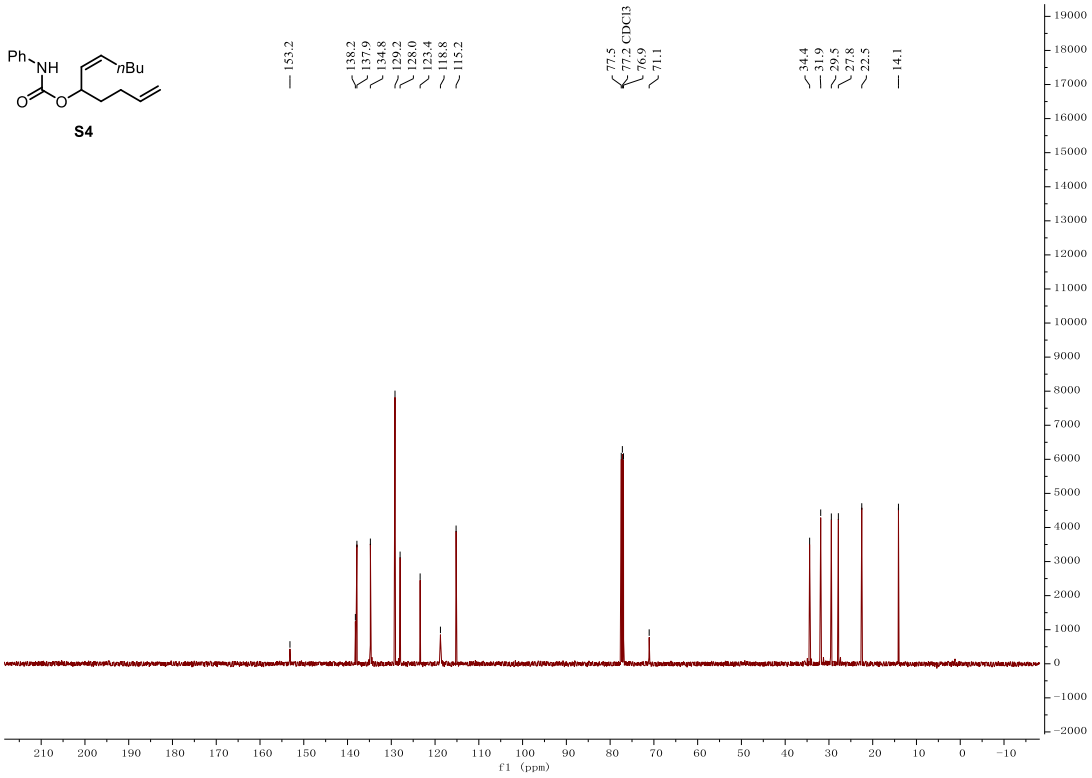
Compound S3


PMP
NH
O
O
tBu
S3
7.29 7.28 7.27 7.27 7.26 CDCl3 6.85 6.84 6.83 6.42 5.86 5.85 5.85 5.84 5.83 5.83 5.82 5.81 5.49 5.49 5.47 5.46 5.19 5.17 5.17 5.15 5.07 5.07 5.06 5.03 5.03 5.03 5.00 5.00 4.99 4.99 4.98 4.98 4.97 4.97 3.78 2.15 2.15 2.15 2.14 2.14 2.13 2.13 2.12 2.12 2.12 1.83 1.82 1.81 1.81 1.65 1.65 1.64 1.64 1.63 1.62 1.62 1.57 HDO 1.16
2.01 2.00 1.00 1.98 0.97 1.04 0.94 0.92 3.01 1.95 1.01 1.05 9.07
f1 (ppm)

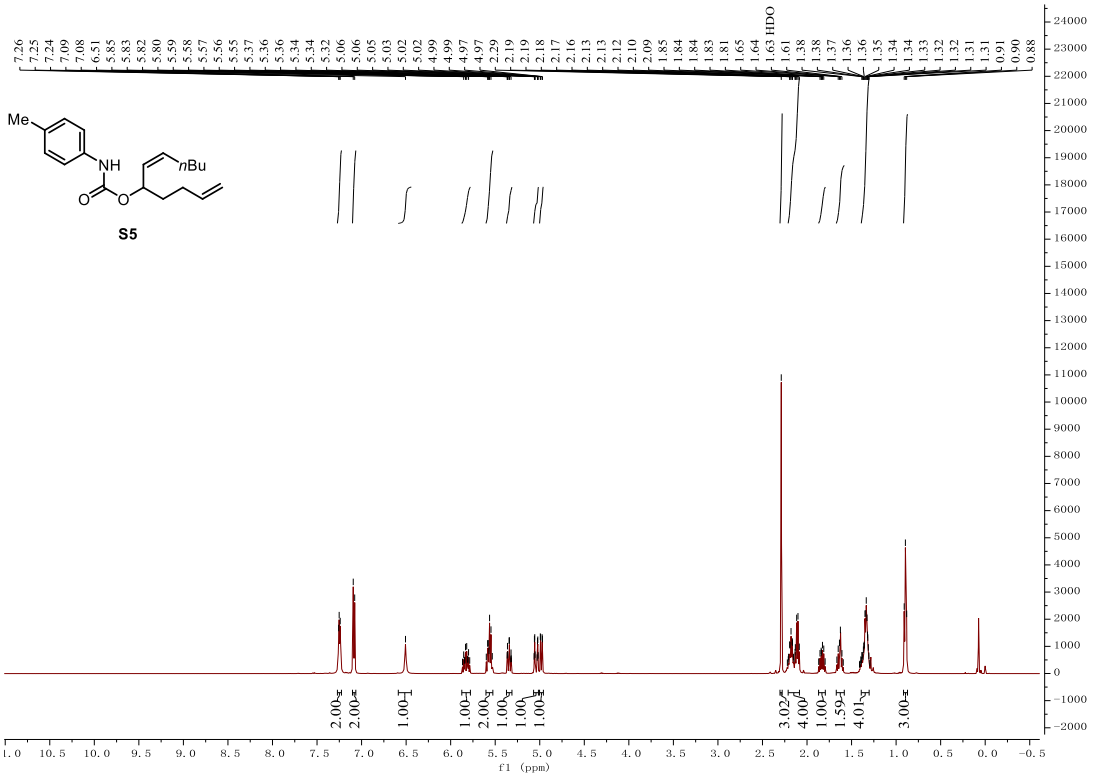

PMP
NH
O
O
tBu
S3
143.2 137.9 126.5 115.2 114.4 77.5 77.2 CDCl3 76.9 55.7 35.1 34.2 31.4 31.3 29.6
f1 (ppm)

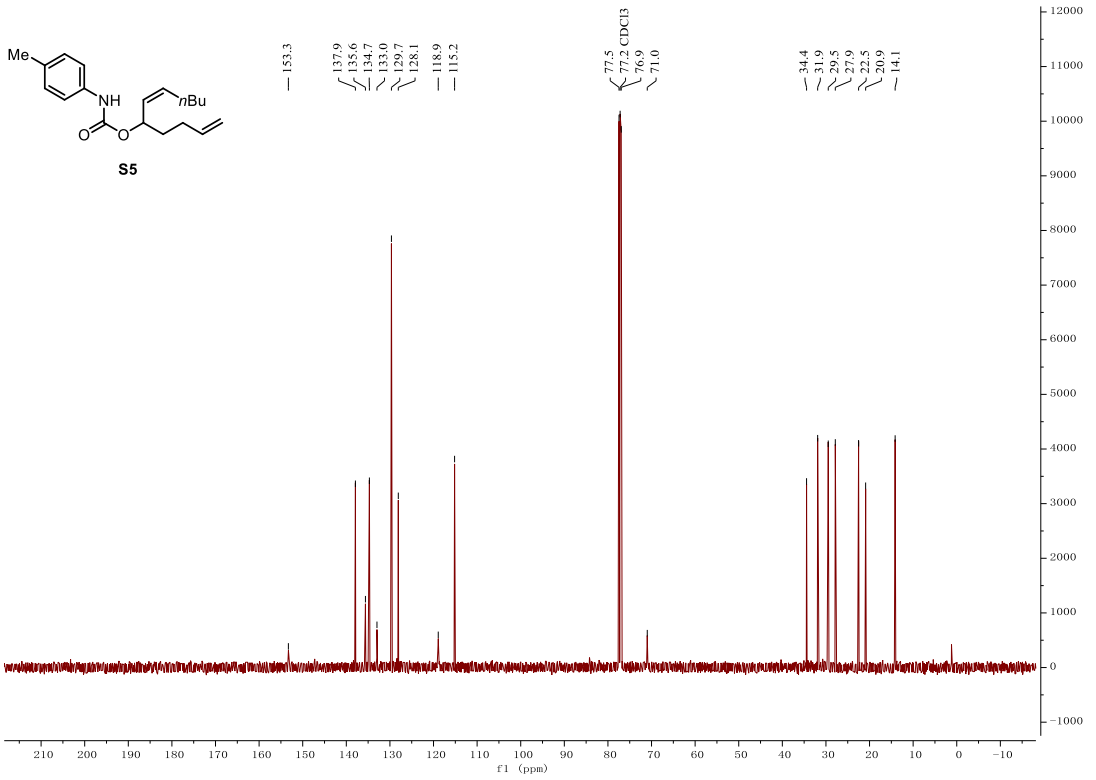
Compound S4


Ph
NH
O
O
nBu
S4
7.38 7.36 7.30 7.29 7.28 7.28 7.27 7.26 7.25 CDCl3 7.05 7.04 7.03 7.03 7.02 6.60 5.83 5.82 5.59 5.58 5.57 5.57 5.56 5.55 5.36 5.35 5.34 5.06 5.06 5.03 5.02 5.00 4.99 4.98 4.97 2.20 2.19 2.18 2.18 2.17 2.17 2.12 2.12 2.12 2.11 2.11 2.10 2.09 1.83 1.66 1.64 1.64 1.63 1.36 1.36 1.36 1.35 1.35 1.34 1.34 1.33 1.33 1.32 1.31 0.91 0.90 0.88
2.00 2.00 1.00 1.00 1.00 2.00 1.00 1.00 2.00 2.01 1.00 1.05 4.00 3.05
f1 (ppm)

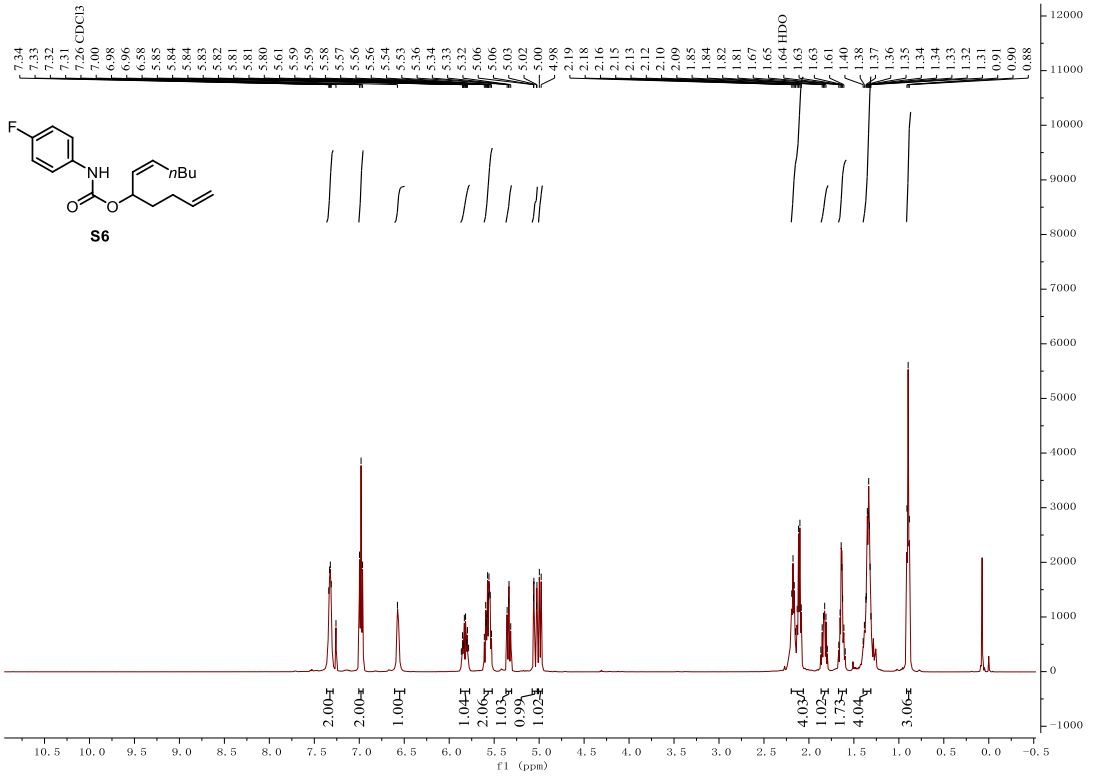

Ph
NH
O
O
nBu
S4
153.2
138.2 137.9 134.8 129.2 128.0 123.4 118.8 115.2
77.5 77.2 CDCl3 76.9 71.1
34.4 31.9 29.5 27.8 22.5 14.1
f1 (ppm)

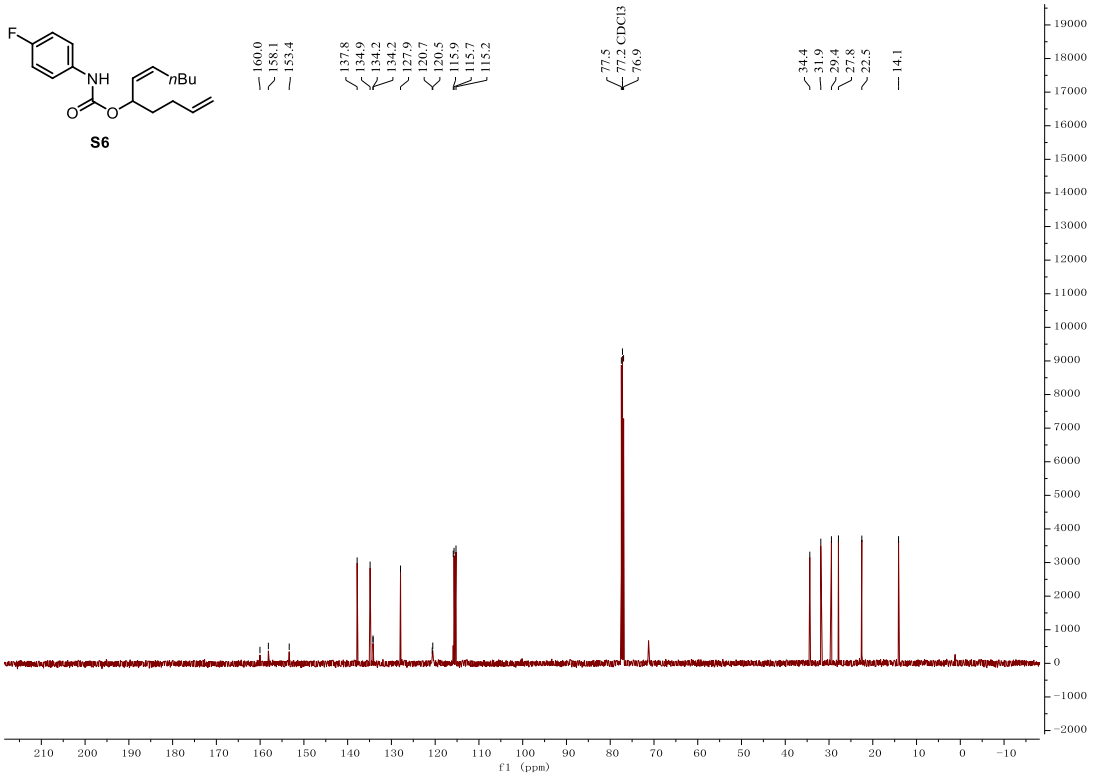
Compound S5

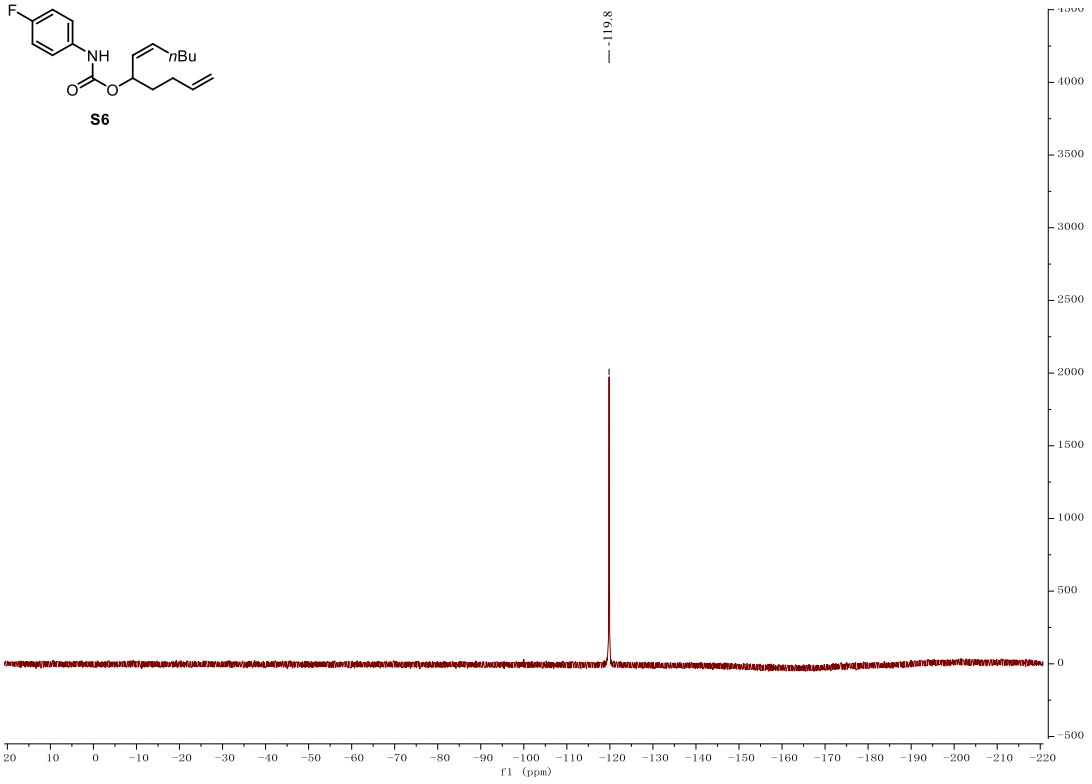

Me
NH
O
O
nBu
S5
7.26 7.25 7.24 7.09 7.08 6.51 5.85 5.83 5.82 5.80 5.59 5.58 5.57 5.56 5.55 5.37 5.36 5.36 5.34 5.34 5.32 5.06 5.06 5.05 5.03 5.02 5.02 4.99 4.99 4.97 4.97 2.29 2.19 2.19 2.18 2.17 2.16 2.13 2.13 2.12 2.10 2.09 1.85 1.84 1.84 1.83 1.81 1.65 1.64 1.63 HDO 1.61 1.38 1.38 1.37 1.36 1.36 1.35 1.34 1.34 1.33 1.32 1.32 1.31 1.31 0.91 0.90 0.88
2.00 2.00 1.00 1.00 2.00 1.00 1.00 1.00 3.02 4.00 1.00 1.59 4.01 3.00
f1 (ppm)


Me
NH
O
O
nBu
S5
153.3
137.9 135.6 134.7 133.0 129.7 128.1 118.9 115.2
77.5 77.2 CDCl3 76.9 71.0
34.4 31.9 29.5 27.9 22.5 20.9 14.1
f1 (ppm)

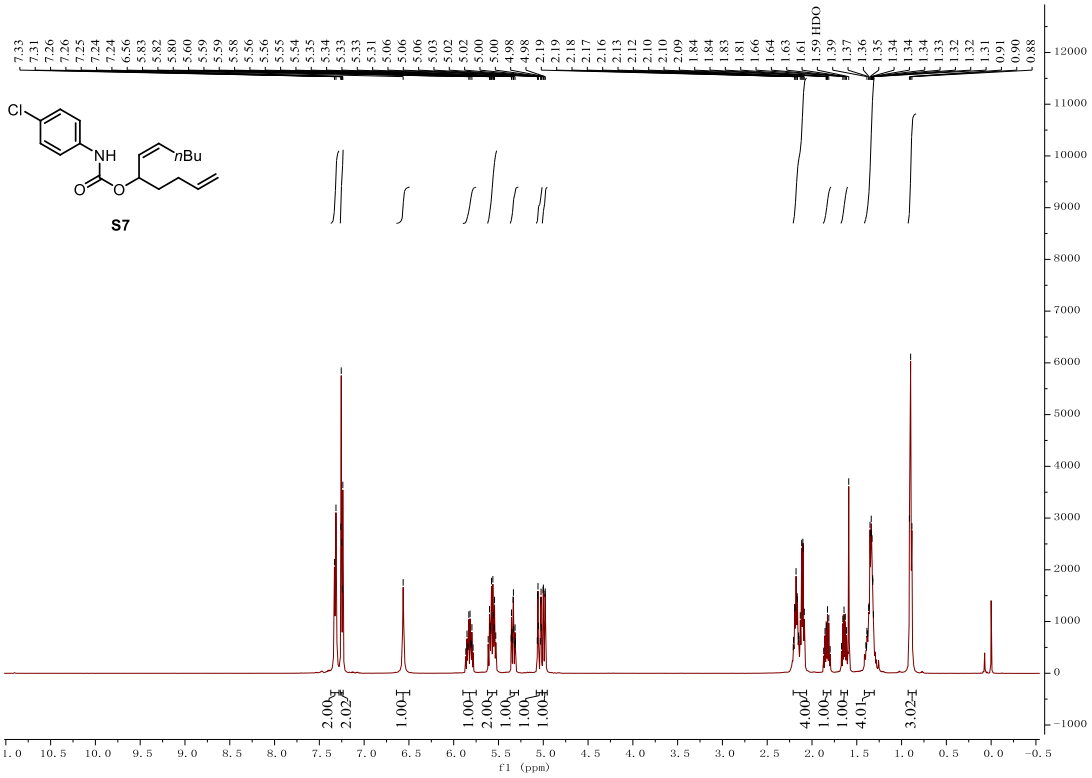
Compound S6

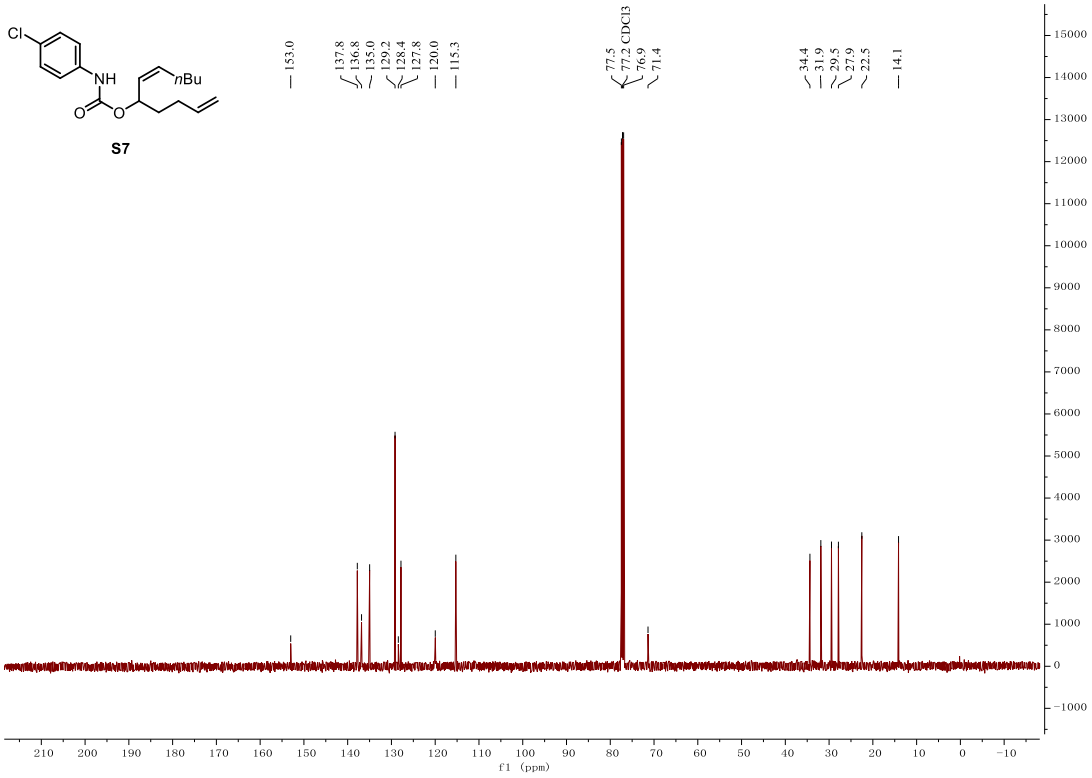

F
NH
O
O
nBu
S6
7.34 7.33 7.32 7.31 7.26 CDCl3 7.00 6.98 6.96 6.58 5.85 5.84 5.84 5.83 5.82 5.81 5.81 5.80 5.61 5.59 5.59 5.58 5.57 5.56 5.56 5.54 5.53 5.36 5.34 5.33 5.32 5.06 5.06 5.03 5.02 5.00 4.98 2.19 2.18 2.16 2.15 2.13 2.12 2.10 2.09 1.85 1.84 1.82 1.81 1.67 1.65 1.64 HDO 1.63 1.63 1.61 1.40 1.38 1.37 1.36 1.35 1.34 1.34 1.33 1.32 1.31 0.91 0.90 0.88
2.00 2.00 1.00 1.04 2.06 1.03 0.99 1.02 4.03 1.02 1.73 4.04 3.06
f1 (ppm)


F
NH
O
O
nBu
S6
160.0 158.1 153.4 137.8 134.9 134.2 134.2 127.9 120.7 120.5 115.9 115.7 115.2 77.5 77.2 CDCl3 76.9 34.4 31.9 29.4 27.8 22.5 14.1
f1 (ppm)

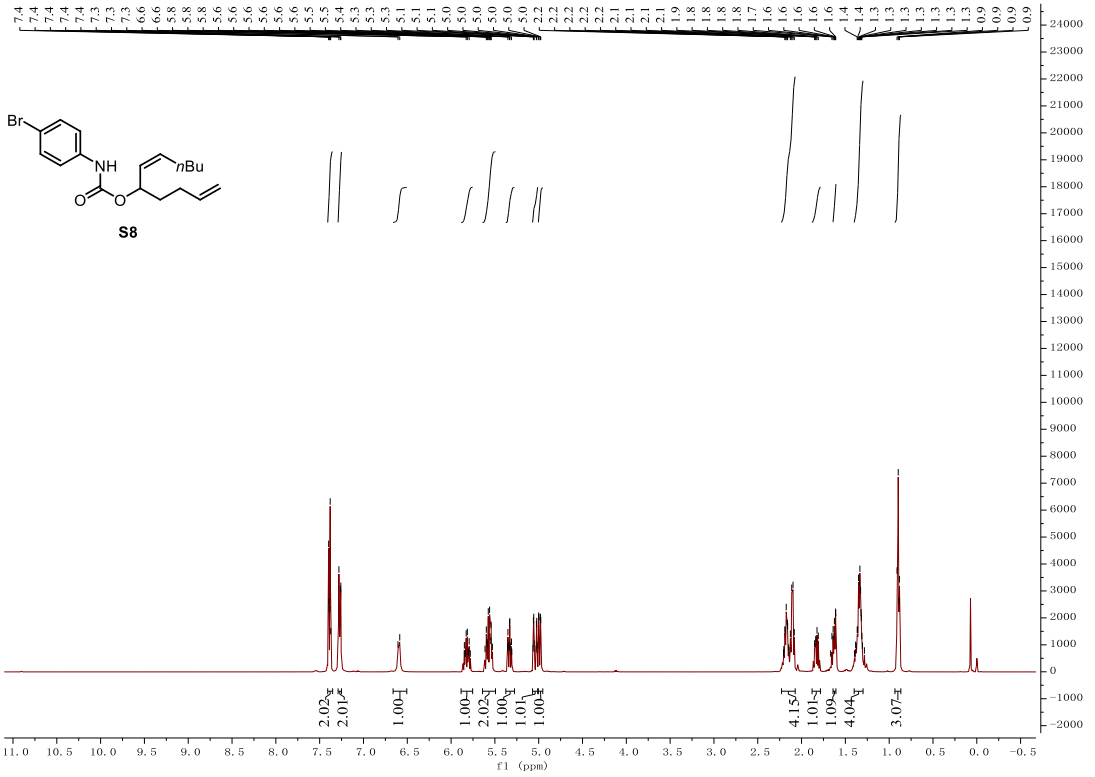

F
NH
O
O
nBu
S6
-119.8
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

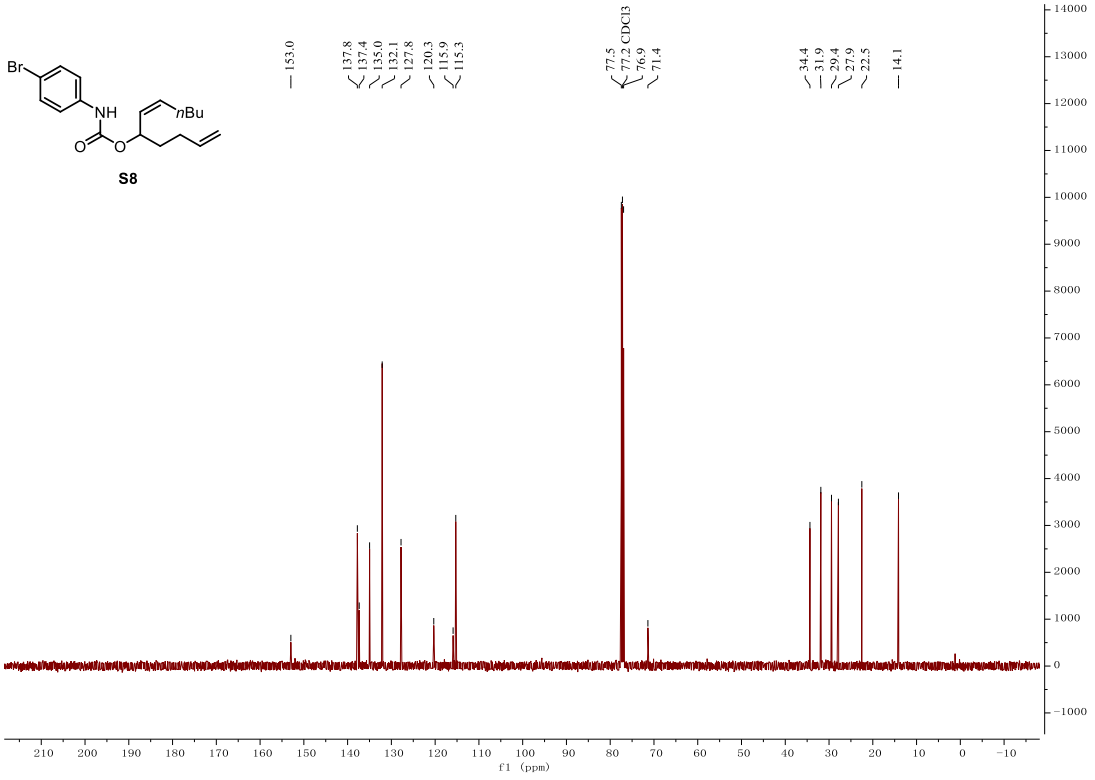
Compound S7


Cl
NH
O
O
nBu
S7
HDO
f1 (ppm)

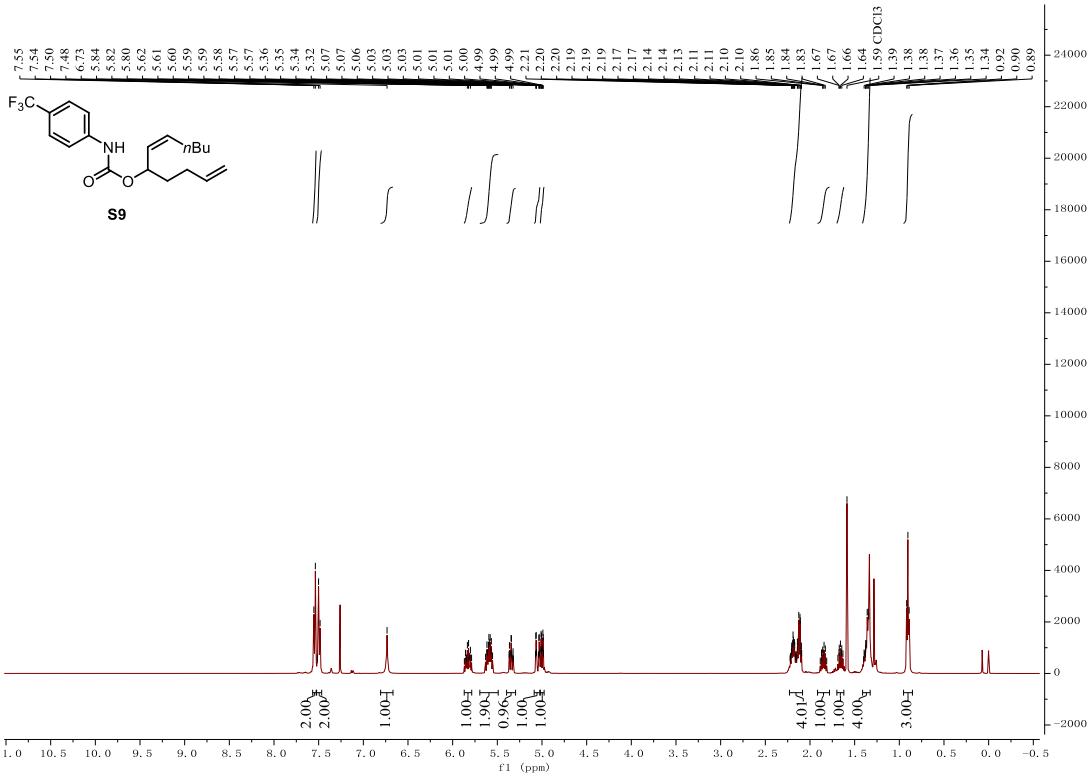

Cl
NH
O
O
nBu
S7
153.0
137.8
136.8
135.0
129.2
128.4
127.8
120.0
115.3
77.5
77.2 CDCl3
76.9
71.4
34.4
31.9
29.5
27.9
22.5
14.1
f1 (ppm)

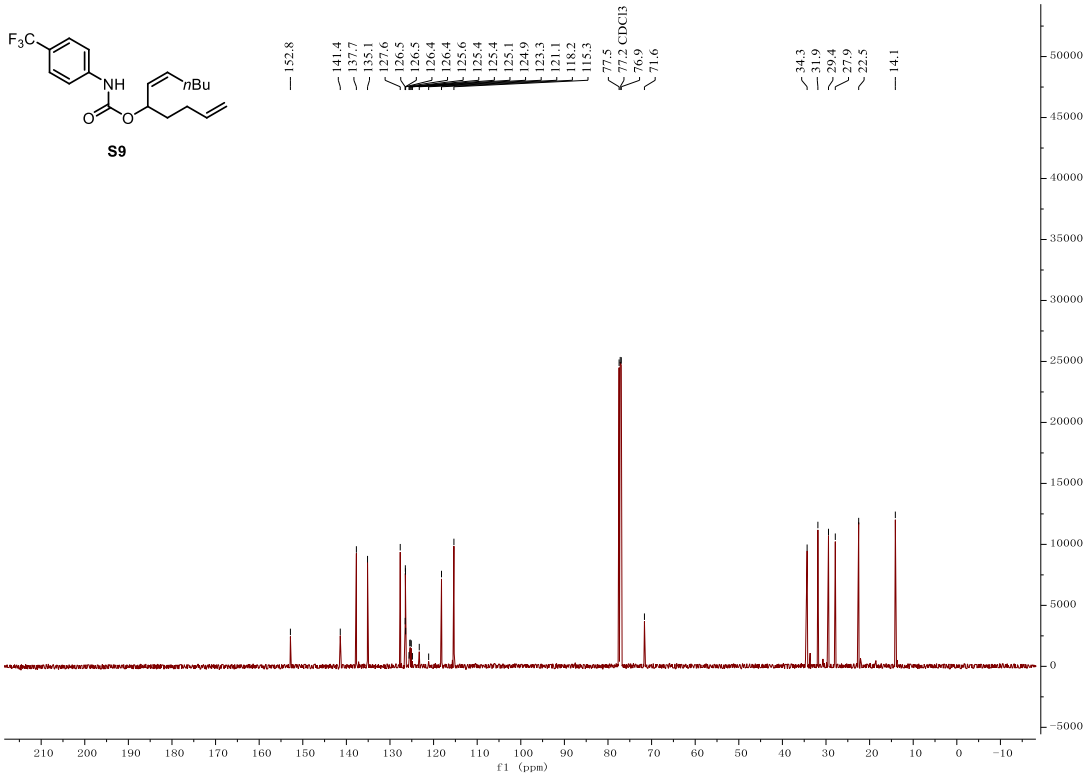
Compound S8

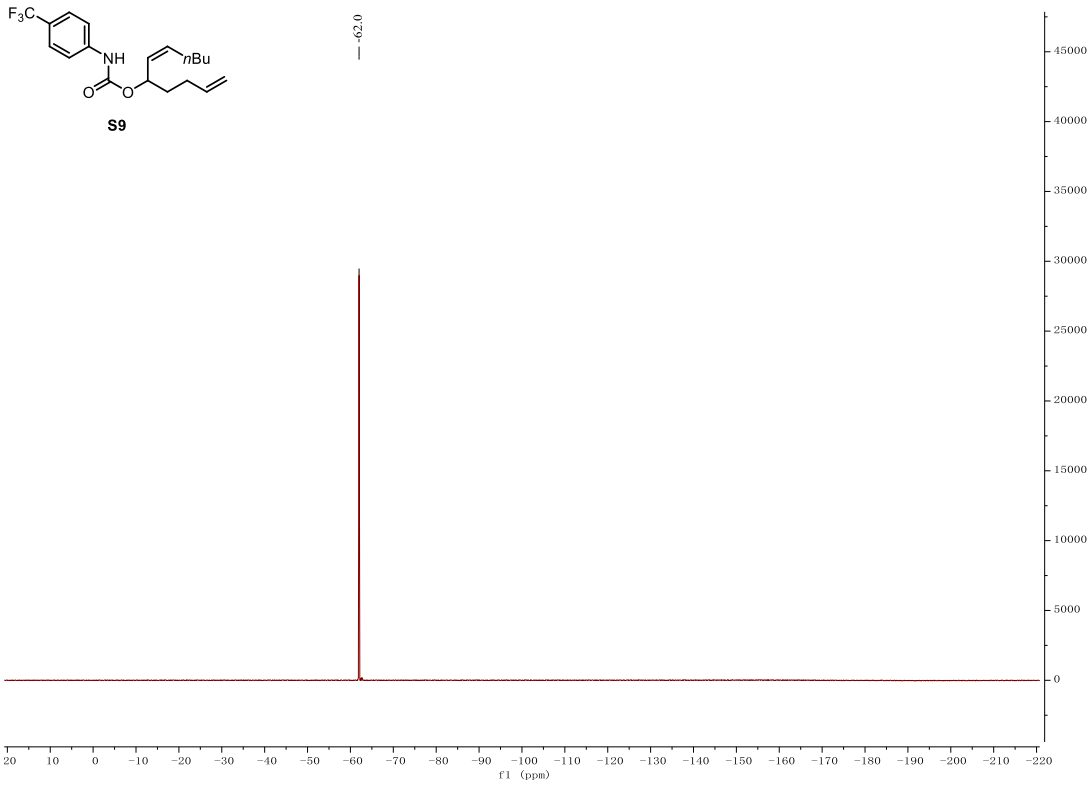

Br
NH
O
O
nBu
S8
f1 (ppm)


Br
NH
O
O
nBu
S8
153.0
137.8
137.4
135.0
132.1
127.8
120.3
115.9
115.3
77.5
77.2 CDCl3
76.9
71.4
34.4
31.9
29.4
27.9
22.5
14.1
f1 (ppm)

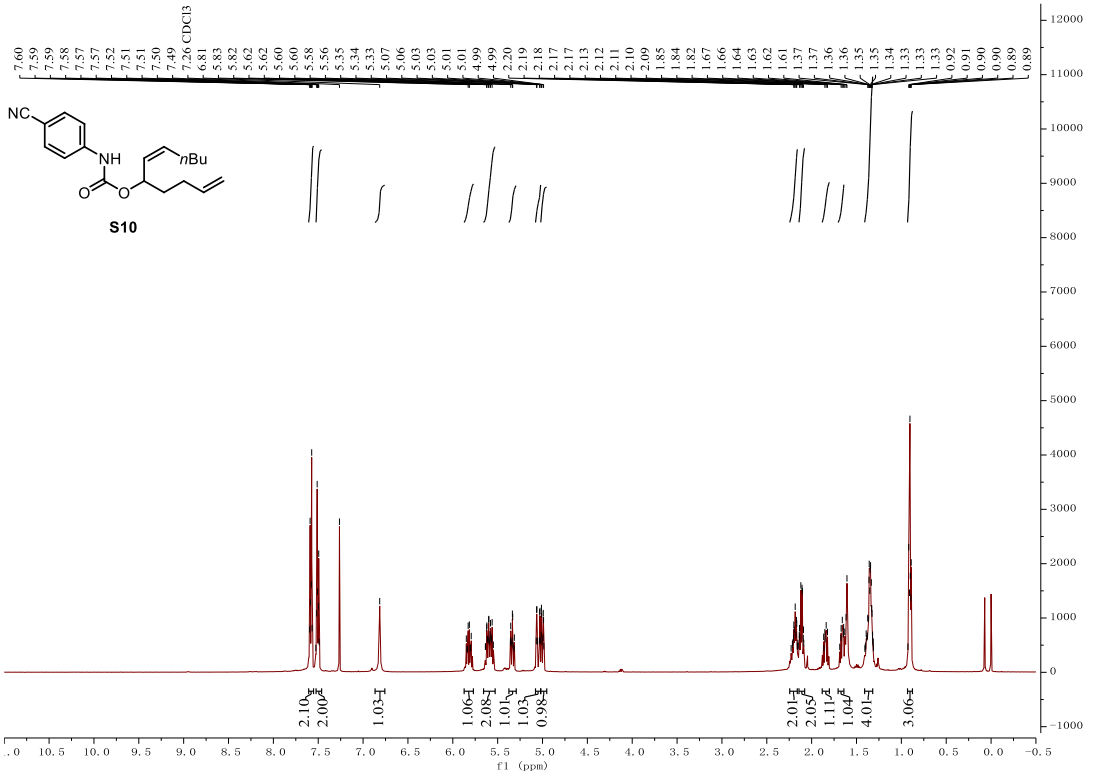
Compound S9

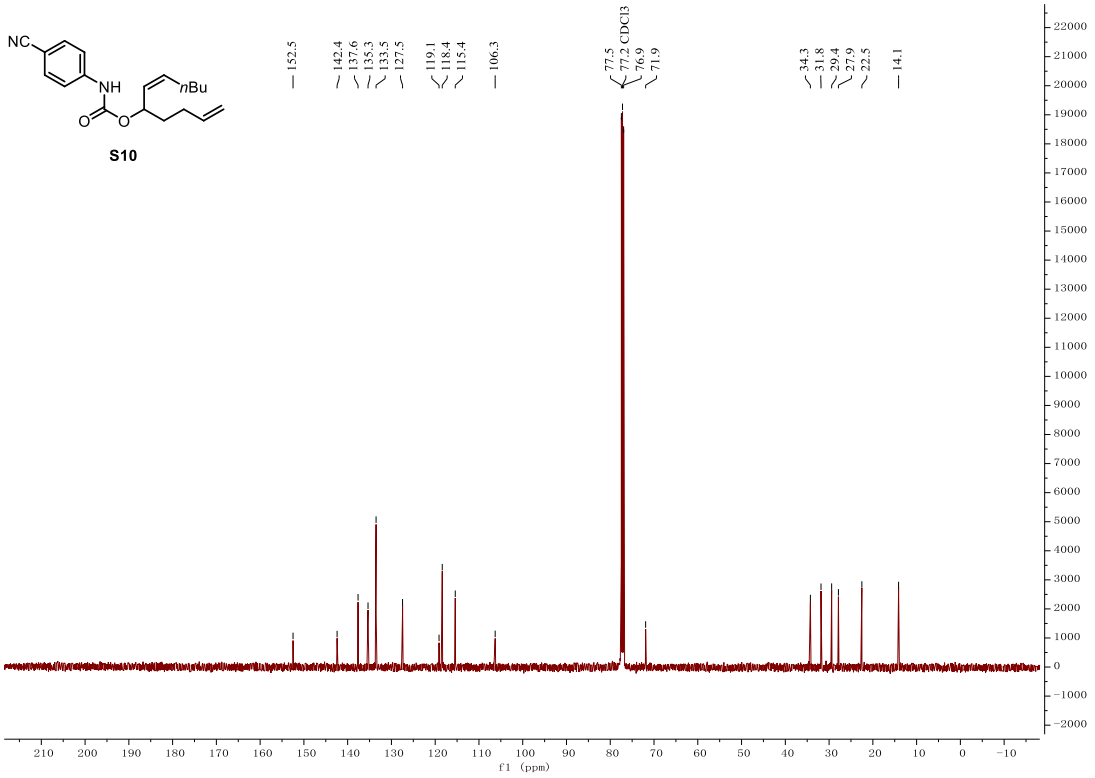

F3C
NH
O
O
nBu
S9
CDCl3
2.00
2.00
1.00
1.00
1.90
0.96
1.00
1.00
4.01
1.00
1.00
4.00
3.00
f1 (ppm)


F3C
NH
O
O
nBu
S9
152.8
141.4
137.7
135.1
127.6
126.5
126.5
126.4
126.4
125.6
125.4
125.4
125.1
124.9
123.3
121.1
118.2
115.3
77.5
77.2 CDCl3
76.9
71.6
34.3
31.9
29.4
27.9
22.5
14.1
f1 (ppm)

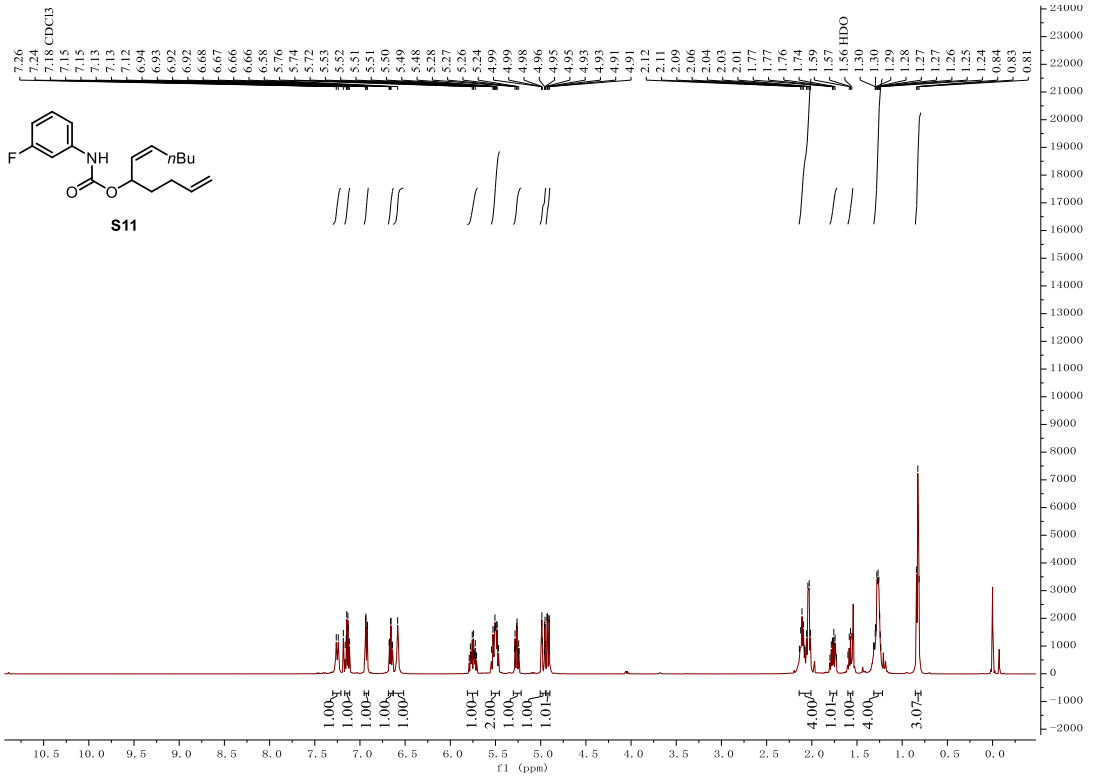

F3C
NH
O
O
nBu
S9
-62.0
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

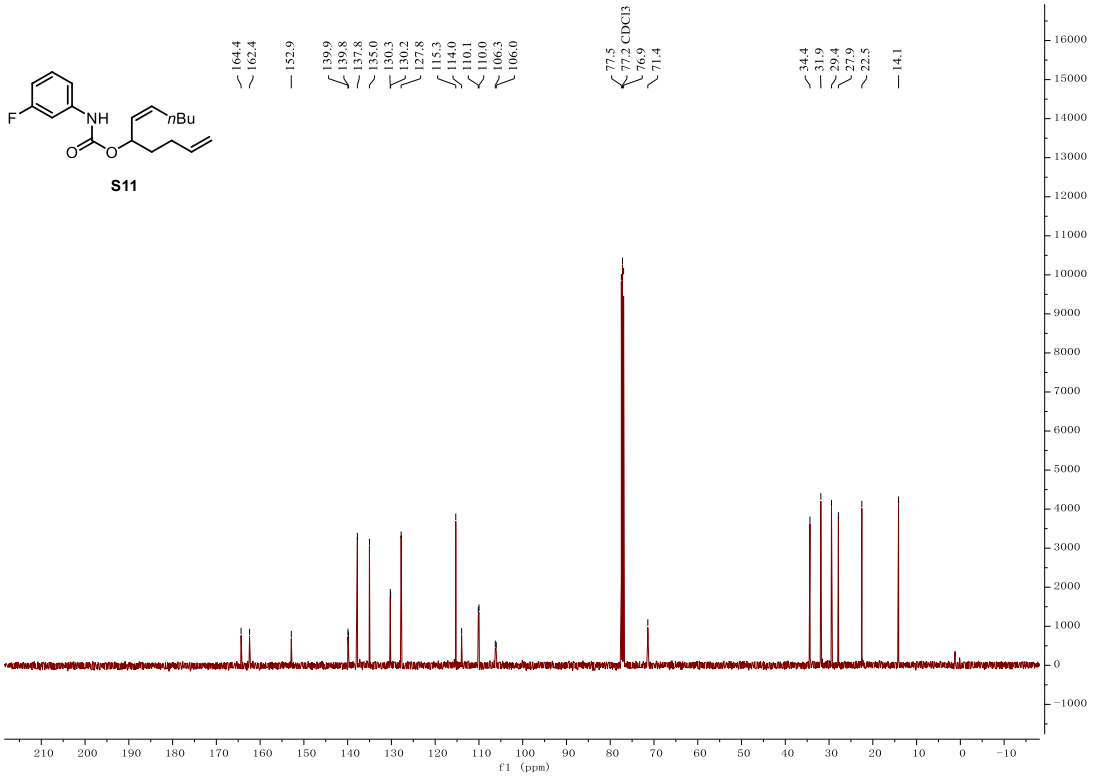
Compound S10

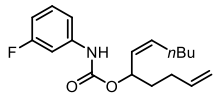




Compound S11



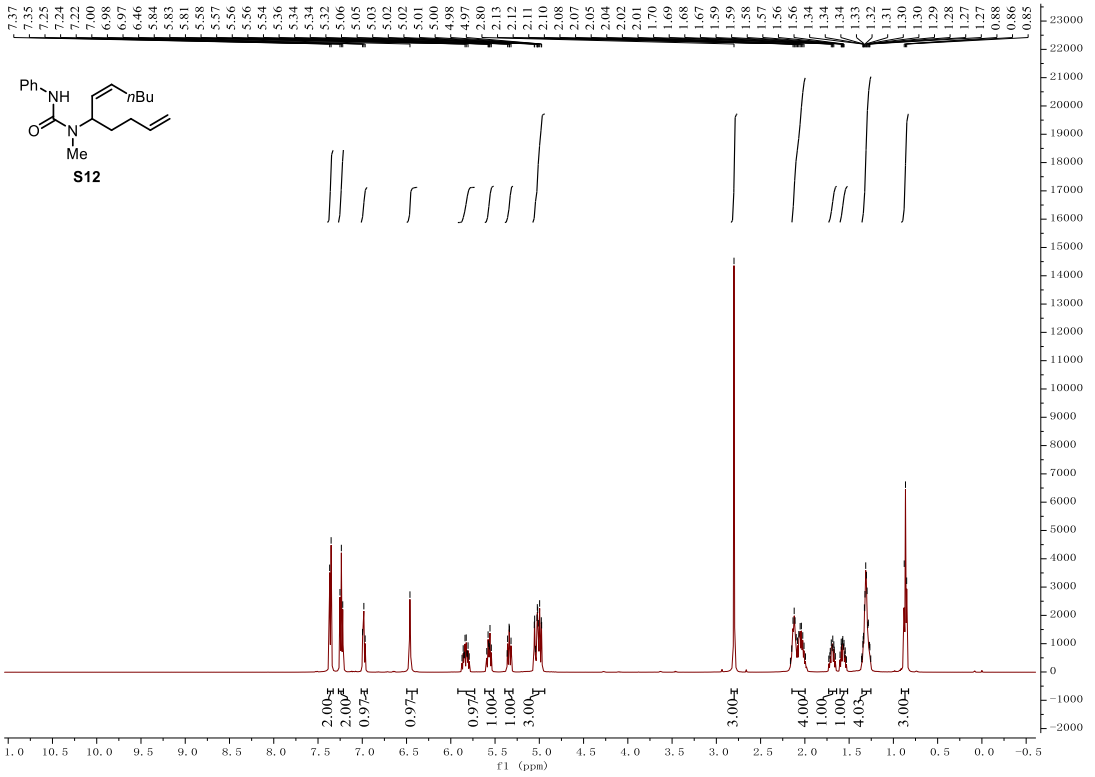


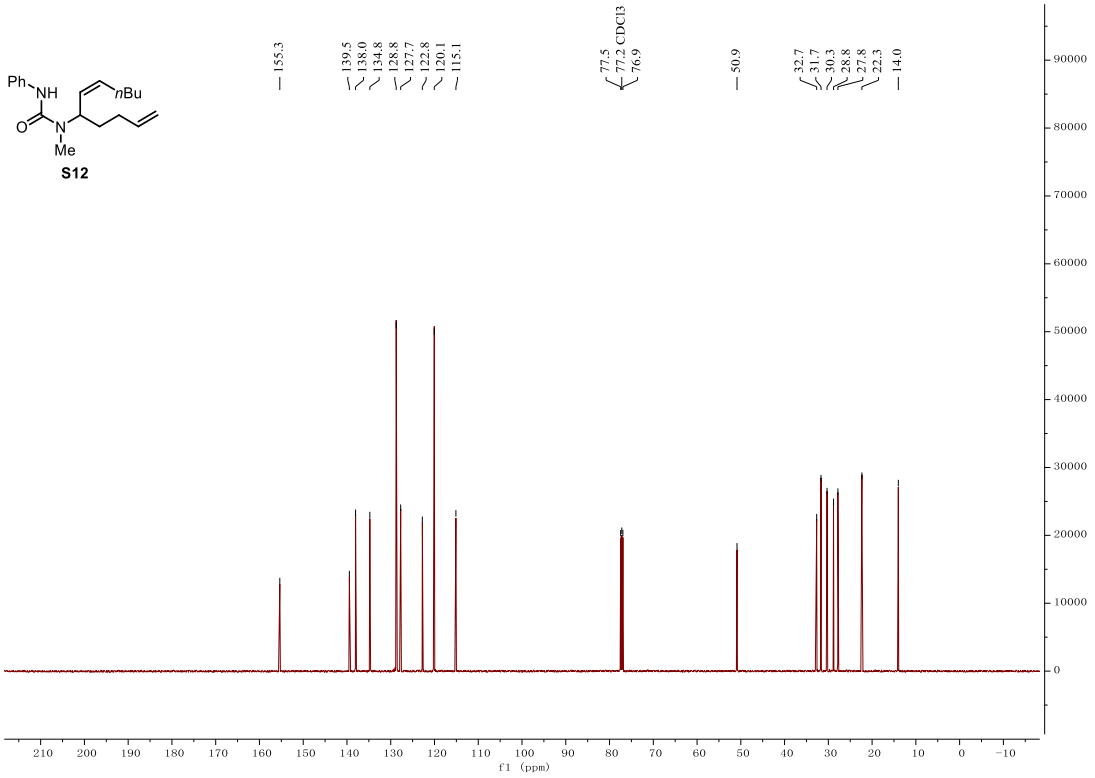

F
NH
O
O
nBu

S11

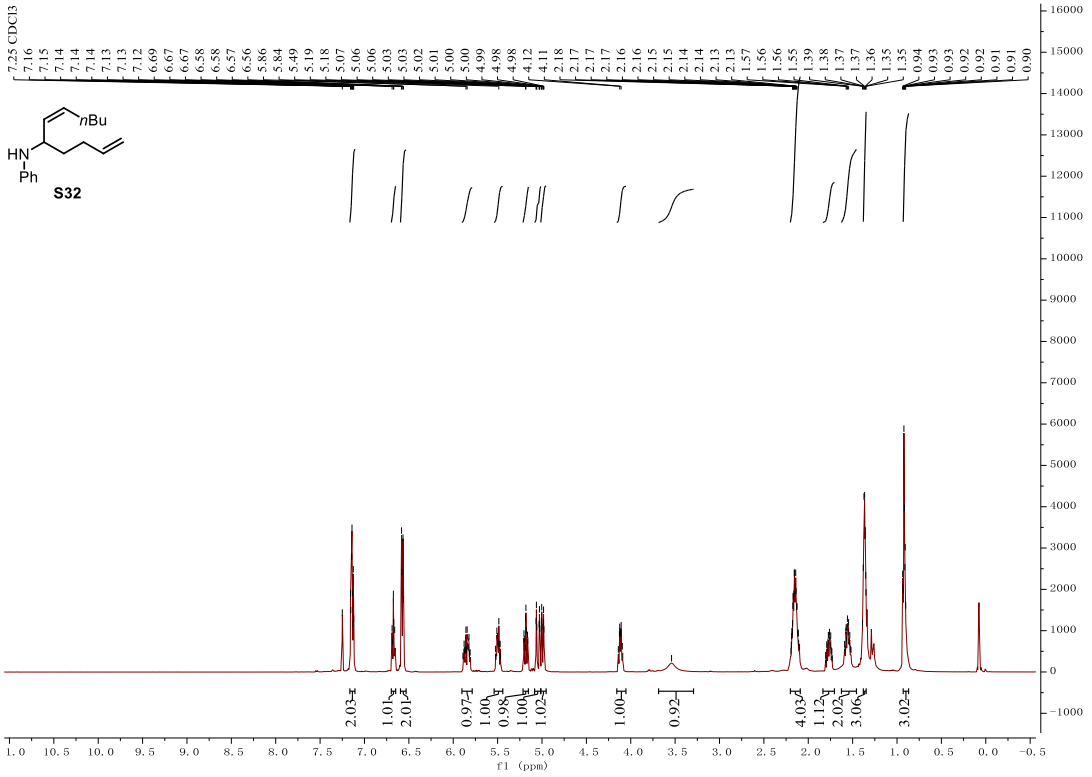
-111.7
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

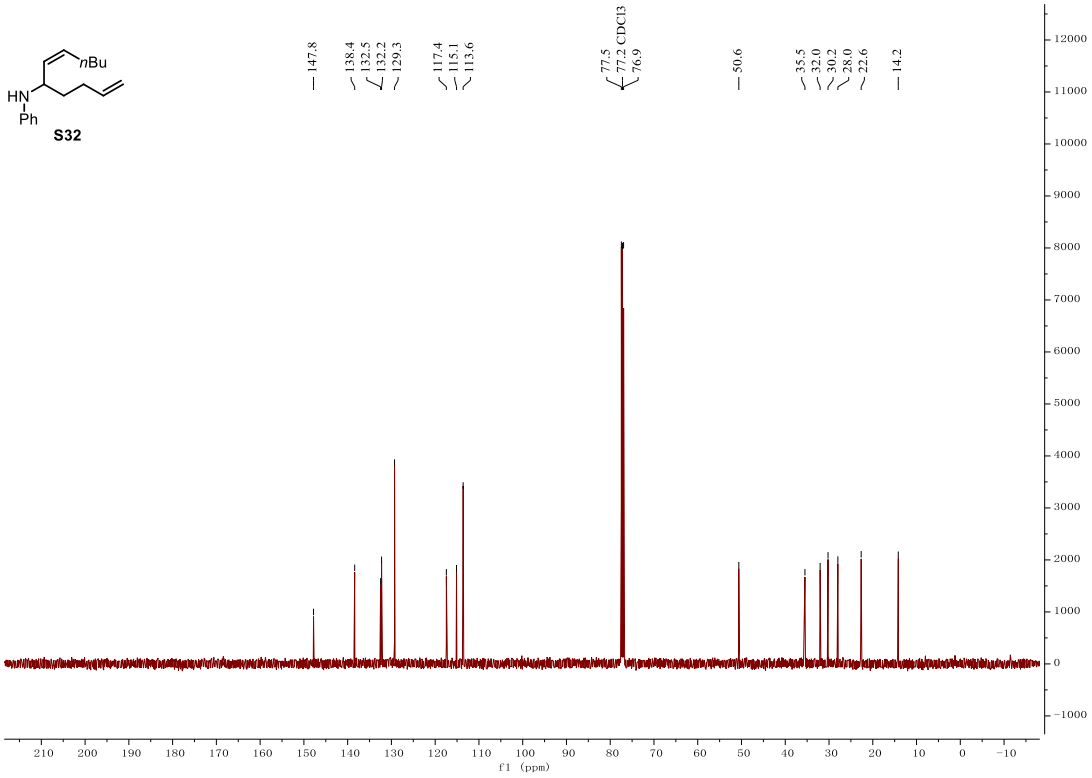
Compound S12



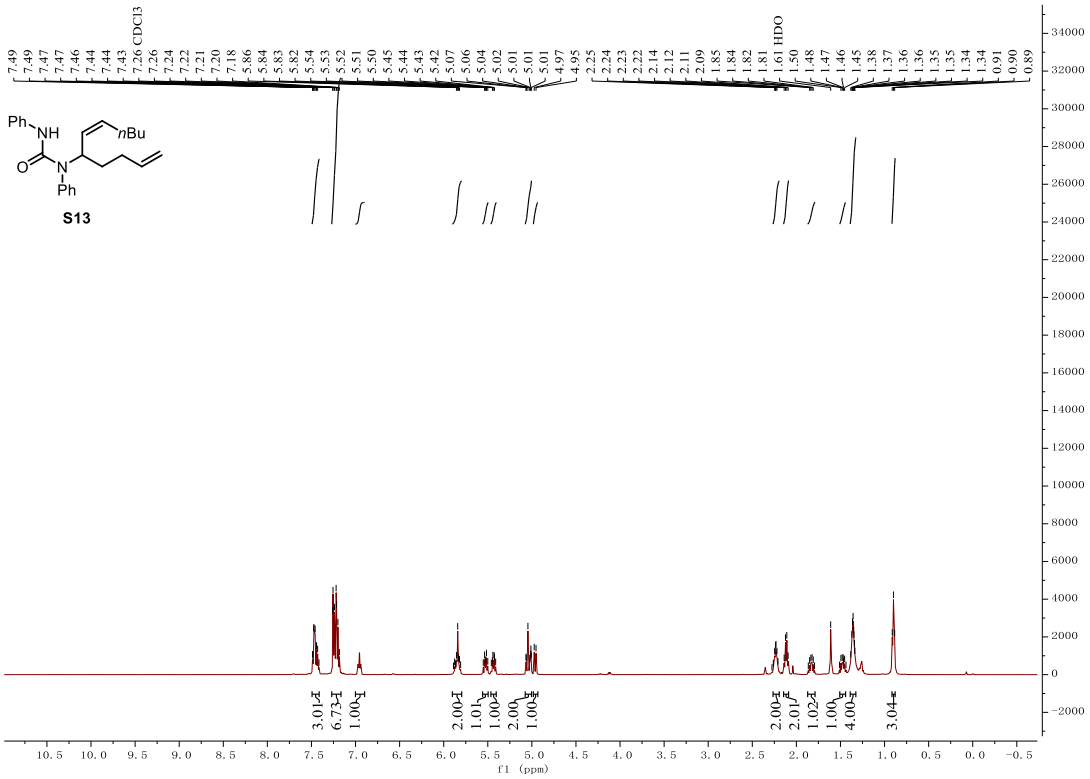


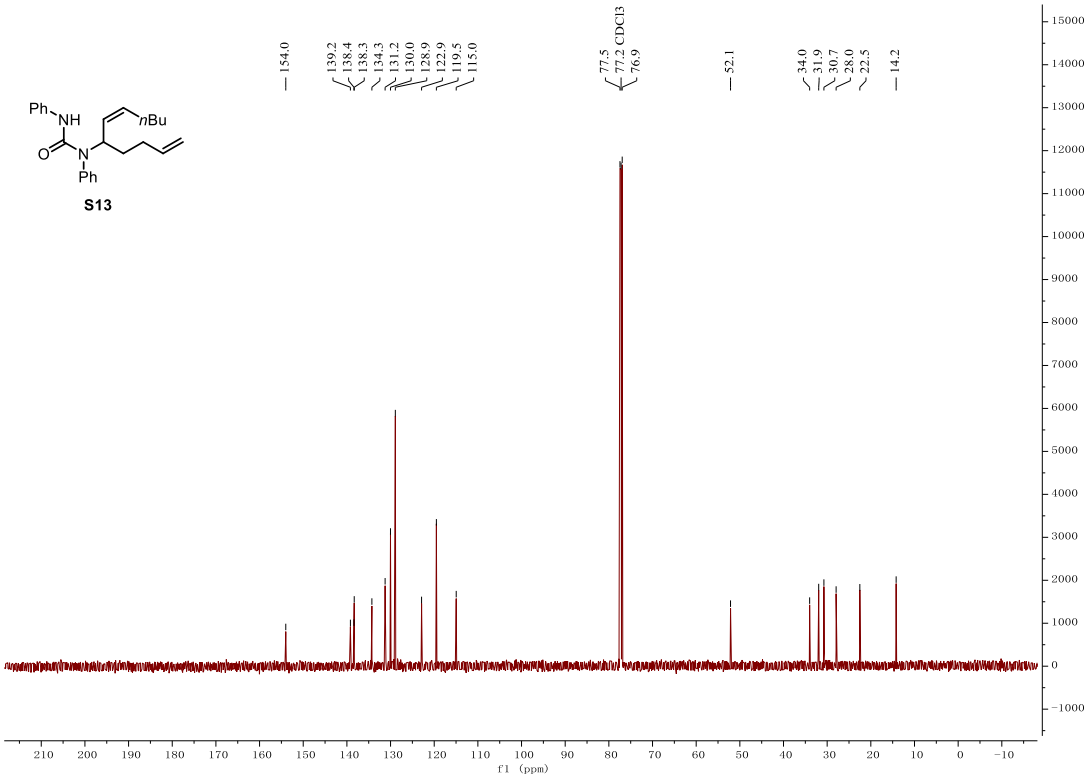
Compound S32



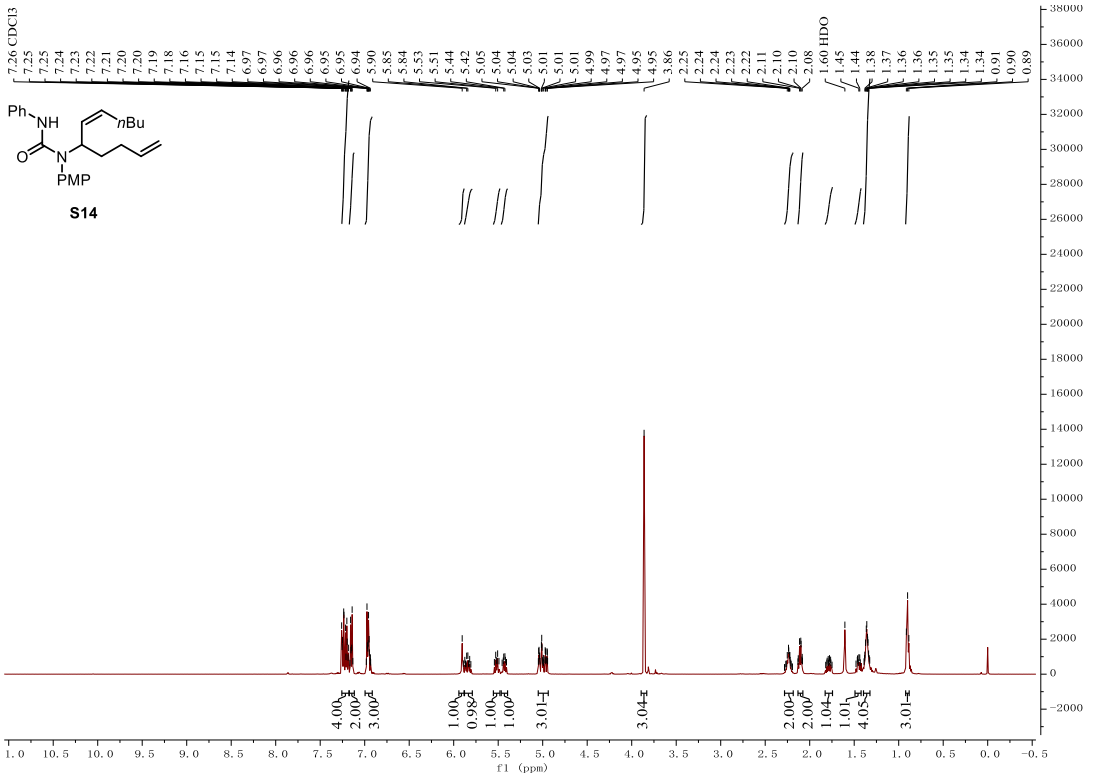


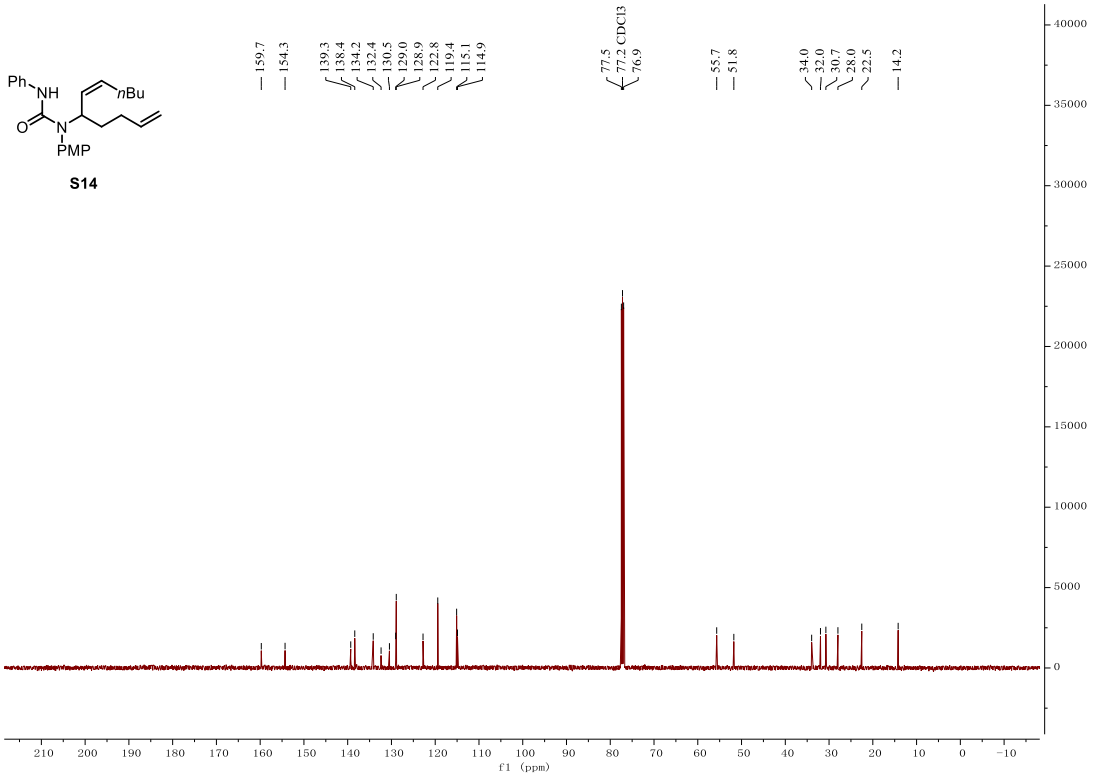
Compound S13





Compound S14





Compound S15