Electronic Supplementary Material (ESI) for ChemComm.


# Supporting Information

## (E)-α,β-Unsaturated Amides from Tertiary Amines, Olefins and CO via Pd / Cu-Catalyzed Aerobic Oxidative *N*-Dealkylation

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## 1. General

All reactions were carried out in oven-dried Schlenk tube under a mixed oxygen and carbon monoxide atmosphere with a balloon. Toluene was distilled from sodium and DMF was dried and distilled from $CaH_2$. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. $PdCl_2(PPh_3)_2$ was synthesized according to literature procedures. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. HPLC yields were recorded with a Dionex P680A LPG-4 high performance liquid chromatography instrument with a UV detector and biphenyl was added as an internal standard. $^1H$ and $^{13}C$ NMR data were recorded with Bruker Advanced II (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts are reported relative to tetramethylsilane and d-solvent peaks (77.00 ppm, chloroform), respectively. High resolution mass spectra (HRMS) were measured with a Waters Micromass GCT instrument and accurate masses were reported for the molecular ion ($[M+H]^+$).

## 2. (E)-α, β-Unsaturated Amides from Tertiary Amines, Olefins and CO via Pd / Cu-Catalyzed Aerobic Oxidative N-Dealkylation

In an oven-dried Schlenk tube equipped with a stir-bar, $PdCl_2(PPh_3)_2$ (8.4 mg, 3 mol%), $Cu(OAc)_2 \cdot H_2O$ (16.0 mg, 20 mol%) were combined. A balloon filled with CO and $O_2$ (the ratio is 1:7) was connected to the Schlenk tube via the side tube and purged for 3 times. Then DMSO (0.2 mL) and toluene (2.0mL) were added to the tube via a syringe. At last, Styrene (0.48 mmol) and amine (0.4 mmol) were added to the tube. The Schlenk tube was heated at 110 $^oC$ for 24 hours and then cooled to room temperature. After the balloon gas was released carefully, the reaction was quenched by water and extracted with $CH_2Cl_2$ three times. The combined organic layers were dried over anhydrous $Na_2SO_4$ and evaporated in vacuum. The desired products were obtained in the corresponding yields after purification by flash chromatography on silica gel(petroleumether : ethyl acetate 10 : 1).

## 3. Preparation of dipiperidin-1-ylmethane and dimorpholinomethane

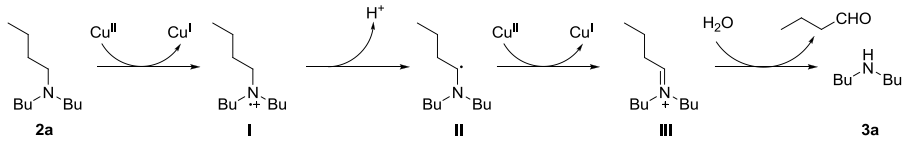
Piperidine or morpholine (20 mol, 40% aqueous solution) was added dropwise to stirred ice-cooled formaldehyde (10 mol, 36% aqueous solution). The mixture was allowed to stand overnight and then saturated with solid potassium hydroxide. The upper layer was separated, dried over potassium hydroxide pellets and the residual liquid was fractionally distilled.

## 4. Preparation of 5-vinylbenzo[d][1,3]dioxole

NaH(12.5 mmol) was added dropwise to a stirred solution of methyltriphenylphosphonium iodide (7 mmol) in THF (40 mL) at 0 ℃. After stirring for 2 h, a cold solution of Piperonyl aldehyde (5 mmol) in THF (8 mL) was added dropwise from a dropping funnel to the reaction mixture. The

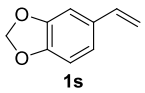
suspension produced was stirred overnight, then treated with saturated ammonium chloride solution, dried, filtered and concentrated under vacuum. The resulting viscous solution was purified by flash chromatography using hexane as eluent.

## 5. Proposed mechanism for the Cu-catalyzed C-N bond activation




Based on the observations vide supra and related mechanistic studies, a plausible mechanism for Cu-catalyzed C-N bongd activation has been proposed. Taking tributyl amine as an example, the tertiary amine is oxidized by Cu(II) salt to afford cation radical **I**. After deprotonation, a carbon radical is obtained which would be oxidized to imine cation. Lastly, **III** would go hydrolysis to give dibutyl amine

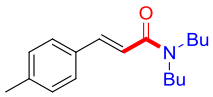
## 6. Analytical Data of Products




**1s**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.01 (d, $J$ = 1.6 Hz, 1 H), 6.87 (dd, $J_1$ = 8.0 Hz, $J_2$ = 1.6 Hz, 1 H), 6.80 (d, $J$ = 8.0 Hz, 1 H), 6.70-6.63 (m, 1 H), 5.99 (s, 2H), 5.62 (dd, $J_1$ = 17.6 Hz, $J_2$ = 0.8 Hz, 1 H), 5.17 (dd, $J_1$ = 10.8 Hz, $J_2$ = 0.6 Hz, 1 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 147.95, 147.31, 136.30, 132.07, 120.97, 111.92, 108.14, 105.34, 101.01.

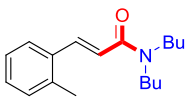



**3a**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, $J$ = 15.6 Hz, 1 H), 7.43 (d, $J$ = 8.0 Hz, 2 H), 7.20 (d, $J$ = 8.0 Hz, 2 H), 6.81 (d, $J$ = 15.2 Hz, 1 H), 3.47-3.39 (m. 4 H), 2.39 (s, 3H), 1.64-1.60 (m, 4 H), 1.41-1.28 (m, 4 H), 0.99-0.95 (dt, $J_1$ = 11.2 Hz, $J_2$ = 5.8 Hz, 6 H)

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 166.15, 142.12, 139.62, 132.75, 129.46, 127.67, 116.72, 47.91, 46.66, 31.94, 30.09, 21.38, 20.30, 20.09, 13.91, 13.84.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 274.2171; found: 274.2744.



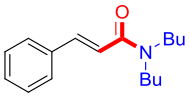

**3b**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, $J$ = 15.2 Hz, 1 H), 7.53 (d, $J$ = 7.2 Hz, 1 H), 7.29-7.22 (m 3 H), 6.76 (d, $J$

= 15.2 Hz, 1 H), 3.48-3.40 (m. 4 H), 2.46 (s, 3H), 1.69-1.58 (m, 4 H), 1.42-1.28 (m, 4 H), 1.01-0.96 (m, 6 H)

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 166.04, 140.01, 137.44, 134.65, 130.65, 129.14, 126.09, 126.04, 119.05, 47.98, 46.73, 31.95, 30.07, 20.32, 20.09, 19.86, 13.90, 13.83.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 274.2171; found: 274.2156.

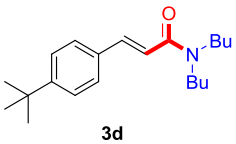



**3c**

$^1$H NMR (400 MHz, $CDCl_3$): δ 7.72 (d, $J$ = 15.2 Hz, 1 H), 7.53 (dd, $J_1$ = 8.0 Hz, $J_2$ = 1.6 Hz, 2 H), 7.41-7.36 (m 3 H), 6.86 (d, $J$ = 15.6 Hz, 1 H), 3.47-3.39 (m. 4 H), 1.68-1.57 (m, 4 H), 1.41-1.33 (m, 4 H), 1.01-0.95 (m, 6 H)

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.96, 142.12, 135.49, 129.36, 128.72, 127.67, 117.77, 47.91, 46.65, 31.93, 30.05, 20.28, 20.07, 13.89, 13.81.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 260.2014; found: 260.2004.

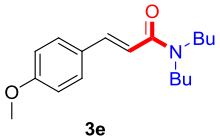



**3d**

$^1$H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, $J$ = 15.2 Hz, 1 H), 7.48 (d, $J$ = 8.4 Hz, 2 H), 7.42 (d, $J$ = 8.4 Hz, 2 H), 6.82 (d, $J$ = 15.6 Hz, 1 H), 3.47-3.39 (m. 4 H), 1.68-1.56 (m, 4 H), 1.43-1.32 (m, 13 H), 0.99-0.95 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 166.13, 152.75, 141.99, 132.75, 127.48, 125.67, 116.91, 47.92, 46.66, 34.74, 31.93, 31.16, 30.08, 20.29, 20.08, 13.90, 13.82.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 316.2640; found: 316.2629.

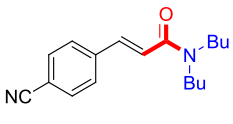



**3e**

$^1$H NMR (400 MHz, $CDCl_3$): δ 7.67 (d, $J$ = 15.2 Hz, 1 H), 7.48 (d, $J$ = 8.8 Hz, 2 H), 6.91 (d, $J$ = 8.8 Hz, 2 H), 6.72 (d, $J$ = 15.6 Hz, 1 H), 3.84 (s, 3 H), 3.46-3.38 (m. 4 H), 1.66-1.55 (m, 4 H), 1.42-1.33 (m, 4 H), 0.99-0.94 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ166.23, 160.63, 141.77, 129.19, 128.21, 115.32, 114.13, 55.28, 47.86, 46.62, 31.91, 30.08, 20.27, 20.07, 13.88, 13.81.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 290.2120; found: 290.2105.



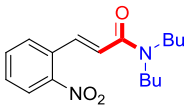

**3f**

$^1$H NMR (400 MHz, $CDCl_3$): δ 7.68-7.65 (m, 3 H), 7.59 (d, $J$ = 8.4 Hz, 2 H), 6.92 (d, $J$ = 15.2 Hz, 1 H), 3.45-3.38

(m. 4 H), 1.64-1.54 (m, 4 H), 1.41-1.35 (m, 4 H), 0.99-0.93 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.03, 139.85, 139.81, 132.47, 128.01, 121.28, 118.49, 112.41, 47.90, 46.68, 31.91, 29.89, 20.20, 19.99, 13.82, 13.76.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 285.1967; found: 285.1954.

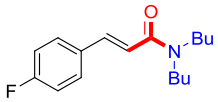



**3g**

$^1$H NMR (400 MHz, $CDCl_3$): δ 8.04-7.98 (m, 2 H), 7.66-7.59 (m 2 H), 7.53-7.49 (m, 1 H), 6.72 (d, *J* = 15.6 Hz, 1 H), 3.45-3.39 (m. 4 H), 1.66-1.58 (m, 4 H), 1.41-1.32 (m, 4 H), 0.98-0.94 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.10, 148.27, 137.07, 133.21, 131.80, 129.44, 129.21, 124.70, 123.44, 48.04, 46.54, 31.84, 29.90, 20.27, 20.02, 13.86, 13.78.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 305.1865; found: 305.1854.



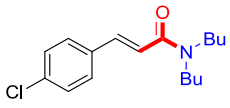

**3h**

$^1$H NMR (400 MHz, $CDCl_3$): δ 7.66 (d, *J* = 15.6 Hz, 1 H), 7.49 (dd, $J_1$ = 5.6 Hz, $J_2$ = 8.8 Hz, 2 H), 7.07-7.03 (m, 2 H), 6.76 (d, *J* = 15.2 Hz, 1 H), 3.44-3.36 (m. 4 H), 1.66-1.53 (m, 4 H), 1.42-1.30 (m, 4 H), 0.98-0.92 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.79, 163.31(d, *J* = 250.4 Hz), 140.88, 129.40(d, *J* = 9.1 Hz), 117.50(d, *J* = 2.0 Hz), 115.77(d, *J* = 22.2 Hz), 47.87, 46.64, 31.91, 30.02, 20.25, 20.05, 13.86, 13.79.

19F NMR (377 MHz, CDCl3) δ -111.2.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 278.1920; found: 278.1914.

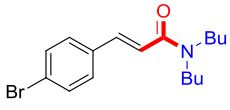



**3i**

$^1$H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 15.2 Hz, 1 H), 7.45 (d, *J* = 8.8 Hz, 2 H), 7.34 (d, *J* = 8.4 Hz, 2 H), 6.82 (d, *J* = 15.6 Hz, 1 H), 3.46-3.38 (m. 4 H), 1.67-1.54 (m, 4 H), 1.41-1.26 (m, 4 H), 1.00-0.94 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.64, 140.74, 135.13, 133.97, 128.95, 128.84, 118.32, 47.88, 46.65, 31.92, 30.00, 20.25, 20.04, 13.86, 13.80.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 294.1625; found: 294.1616.

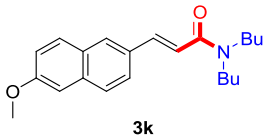



**3j**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, *J* = 15.2 Hz, 1 H), 7.50 (d, *J* = 8.4 Hz, 2 H), 7.37 (d, *J* = 8.0 Hz, 2 H), 6.83 (d, *J* = 15.2k Hz, 1 H), 3.45-3.37 (m. 4 H), 1.66-1.54 (m, 4 H), 1.43-1.31 (m, 4 H), 1.00-0.93 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.63, 140.80, 134.39, 131.90, 129.08, 123.40, 118.42, 47.89, 46.66, 31.92, 29.99, 20.25, 20.04, 13.87, 13.80.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 338.1120; found: 338.1110.

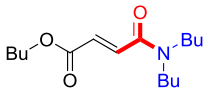



**3k**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.87-7.84 (m, 2 H), 7.78-7.72 (m, 2 H), 7.66-7.64 (m, 1 H), 7.18-7.13 (m, 2 H), 6.92 (d, *J* = 15.6 Hz, 1 H), 3.94 (s, 3 H), 3.49-3.42 (m. 4 H), 1.69-1.58 (m, 4 H), 1.43-1.36 (m, 4 H), 1.00-0.96 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 166.12, 158.38, 142.35, 135.13, 130.78, 129.89, 128.89, 128.72, 127.22, 124.21, 119.21, 116.75, 105.84, 55.28, 47.89, 46.65, 31.92, 30.07, 20.28, 20.08, 13.89, 13.82.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 340.2277; found: 330.2268.

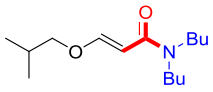



**3l**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.37 (d, *J* = 15.2 Hz, 1 H), 6.83 (d, *J* = 15.2 Hz, 1 H), 4.21 (t, *J* = 6.4 Hz, 2 H), 3.42-3.32 (m. 4 H), 1.71-1.64 (m, 2 H), 1.62-1.52 (m, 4 H), 1.45-1.31 (m, 6 H), 0.98-0.93 (m, 9 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 166.00, 164.13, 134.07, 131.01, 64.91, 48.03, 46.44, 31.82, 30.53, 29.76, 20.22, 19.92, 19.09, 13.84, 13.71, 13.66.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 274.2171; found: 274.2164.

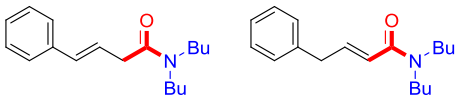



**3m**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 12.0 Hz, 1 H), 5.61 (d, *J* = 11.6 Hz, 1 H), 3.65 (d, *J* = 6.8 Hz, 2 H), 3.30 (td, $J_1$ = 7.2 Hz, $J_2$ = 50.4 Hz, 4 H), 2.05-1.95 (m, 1 H), 1.57-1.52 (m, 4 H), 1.35-1.33 (m, 4 H), 0.99-0.91 (m, 12 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 166.90, 161.67, 96.09, 78.42 , 47.83, 46.17, 31.61, 30.21, 29.69, 28.36, 20.26, 20.11, 18.92, 13.90.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 256.2277; found: 256.2270.

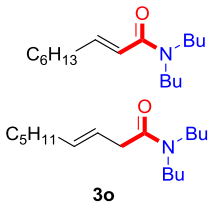


**3n**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.05 (d, *J* =7.2 Hz, 0.15 H), 7.99 (d, *J* = 14.8 Hz, 0.11 H), 7.60 (d, *J* = 7.2 Hz, 0.15 H), 7.59-7.50 (m, 0.39 H), 7.37 (d, *J* = 8.0 Hz, 2 H), 7.31-7.28 (m, 2.18 H), 7.23-7.19 (m, 1.15 H), 7.09-7.03 (m, 0.16 H), 6.48-6.36 (m, 2 H), 6.10 (d, *J* = 15.2 Hz, 0.14 H), 3.53 (d, *J* = 6.8 Hz, 0.31 H), 3.45-3.23 (m. 6.72 H), 1.57-1.51 (m, 4.92 H), 1.37-1.31 (m, 4.95 H), 0.97-0.91 (m, 7.35 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 170.37, 165.89, 143.99, 137.08, 132.26, 128.80, 128.78, 128.49, 128.43, 127.26, 126.38, 126.16, 123.86, 121.87, 48.11, 47.89, 46.39, 45.72, 38.55, 37.88, 31.24, 30.01, 29.84, 29.67, 20.24, 20.20, 20.10, 19.96, 13.87, 13.82, 13.73, 13.66.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 284.2226; found: 284.2217.

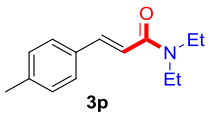


**3o**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.29-6.88 (m, 1 H), 6.19 (d, *J* = 14.8 Hz, 1 H), 5.58-5.53 (m, 2 H), 3.39-3.32 (m, 8 H), 3.07 (d, *J* = 5.6 Hz, 2 H), 2.23-2.19 (m, 2 H), 2.07-2.02 (m, 2 H), 1.55-1.51 (m, 8 H), 1.36-1.27 (m, 22 H), 0.98-0.87 (m, 18 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 171.06, 166.26, 146.13, 133.63, 123.26, 120.36, 47.80, 47.78, 47.72, 46.40, 45.57, 37.67, 32.49, 31.75, 31.63, 31.36, 31.20, 30.04, 29.82, 29.68, 28.92, 28.85, 28.34, 22.58, 22.50, 20.29, 20.24, 20.10, 20.05, 14.07, 14.04, 13.88, 13.83, 13.81.

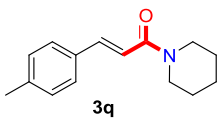
HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 268.2640; found: 268.2635.



**3p**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, *J* = 15.2 Hz, 1 H), 7.43 (d, *J* = 8.0 Hz, 2 H), 7.18 (d, *J* = 8.0 Hz, 2 H), 6.78 (d, *J* = 15.6 Hz, 1 H), 3.52-3.44 (m. 4 H), 2.37 (s, 3 H), 1.28-1.17 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.84, 142.24, 139.63, 132.65, 129.42, 127.67, 116.58, 42.24, 41.03, 21.35, 15.02, 13.19.



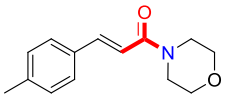
**3q**

$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, *J* = 15.2 Hz, 1 H), 7.42 (d, *J* = 8.0 Hz, 2 H), 7.17 (d, *J* = 8.0 Hz, 2 H), 6.87 (d, *J* = 15.2 Hz, 1 H), 3.62 (d, *J* = 33.6 Hz, 4 H), 2.36 (s, 3 H), 1.68-1.60 (m, 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.36, 142.01, 139.46, 132.52, 129.30, 127.49, 116.40, 46.83, 43.17, 26.58,

25.46, 24.49, 21.23.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 230.1545; found: .230.1536

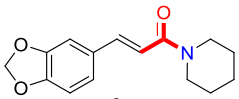



**3r**

$^1$H NMR (400 MHz, $CDCl_3$): δ 7.67 (d, *J* = 15.2 Hz, 1 H), 7.42 (d, *J* = 7.2 Hz, 2 H), 7.17 (d, *J* = 7.2 Hz, 2 H), 6.81 (d, *J* = 15.6 Hz, 1 H), 3.71-3.66 (m. 8 H), 2.36 (s, 3 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.55, 143.00, 139.85, 132.11, 129.34, 127.58, 115.17, 66.62, 45.98, 42.24, 21.22.

HRMS (ESI) calcd for $C_{16}H_{13}NO$ $[M+H]^+$: 232.1338; found: 232.1332.

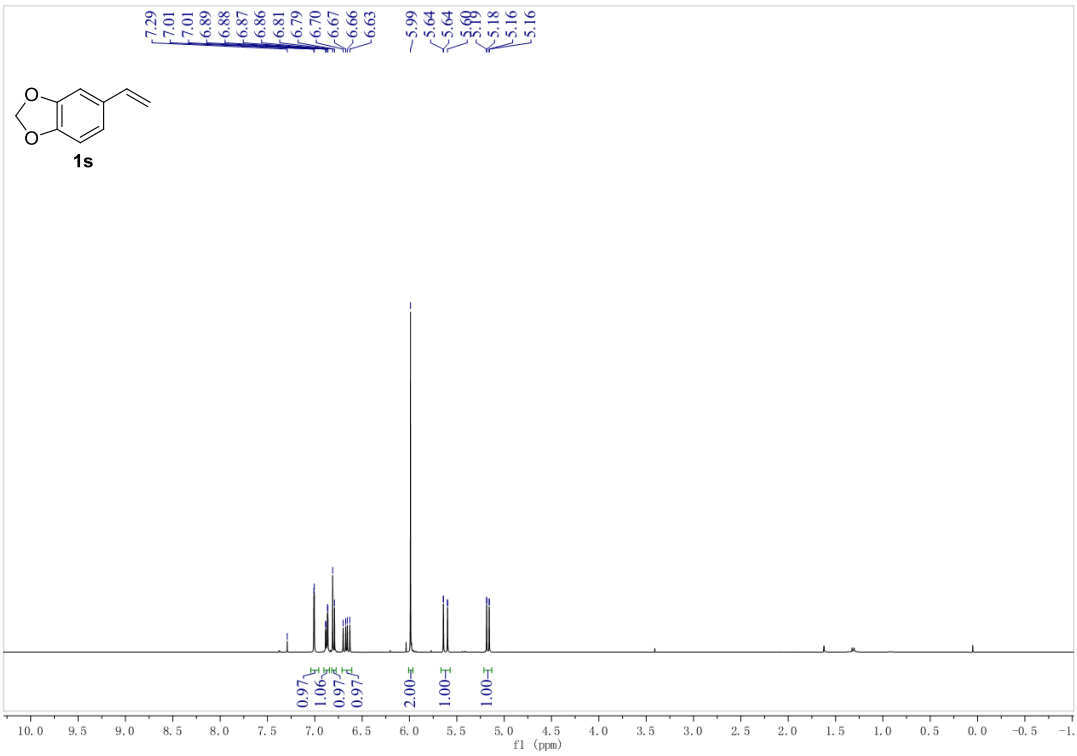


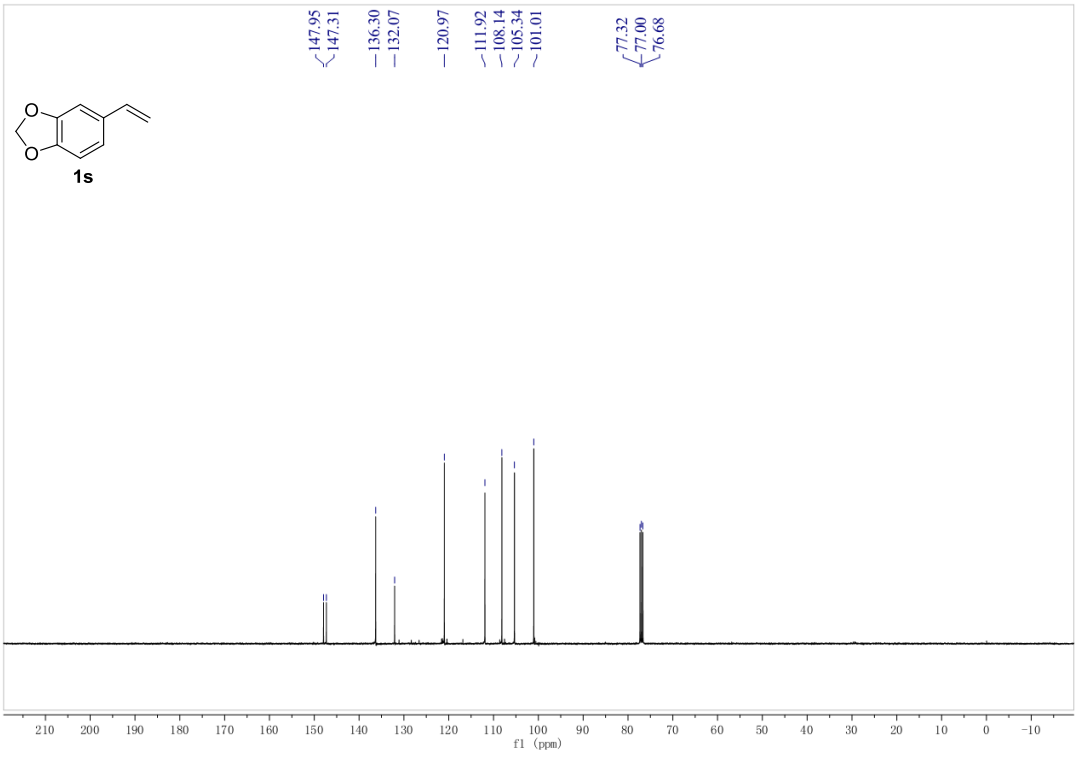

**3s**

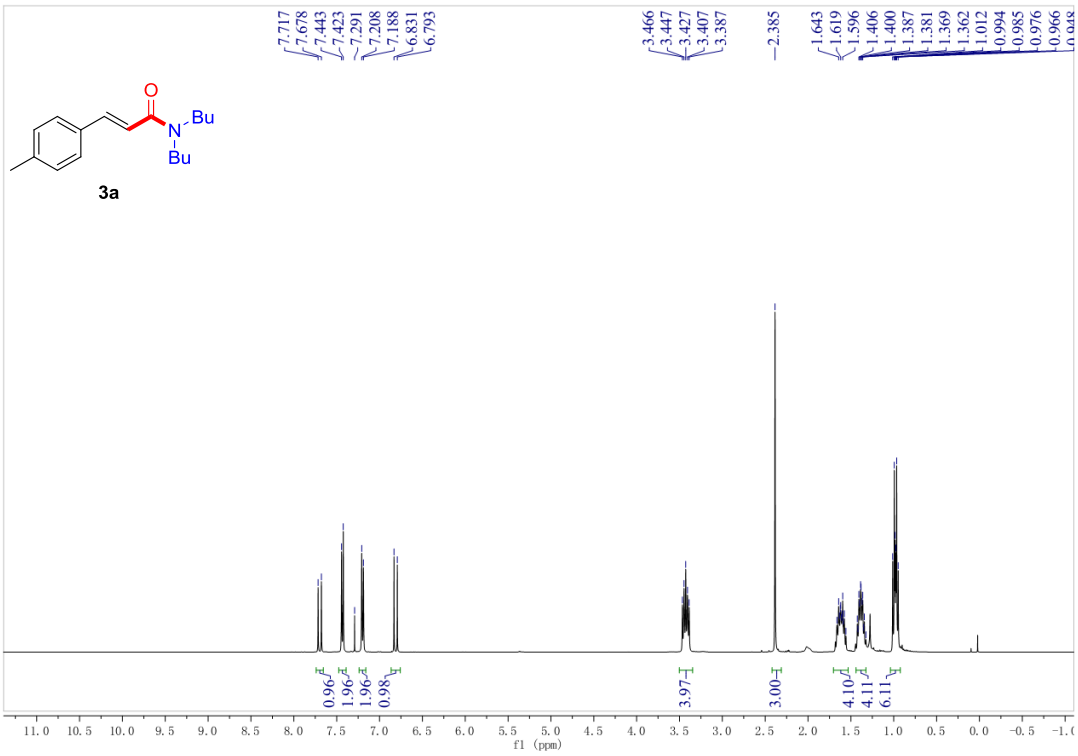
$^1$H NMR (400 MHz, $CDCl_3$): δ 7.55 (d, *J* = 15.2 Hz, 1 H), 7.02 (d, *J* = 1.6 Hz, 1 H), 6.98 (dd, $J_1$ = 8.0 Hz, $J_2$ = 1.6 Hz, 1 H), 6.78 (d, *J* = 8.0 Hz, 1 H), 6.73 (d, *J* = 15.2 Hz, 1 H), 5.97 (s, 2H), 3.60 (d, *J* = 33.2 Hz, 4 H ), 1.67-1.59 (m. 6 H).

$^{13}$C NMR (101 MHz, $CDCl_3$): δ 165.23, 148.64, 147.99, 141.77, 129.70, 123.42, 115.46, 108.28, 106.14, 101.23, 46.80, 43.16, 26.59, 25.46, 24.49.

# 7. NMR Spectra of Products

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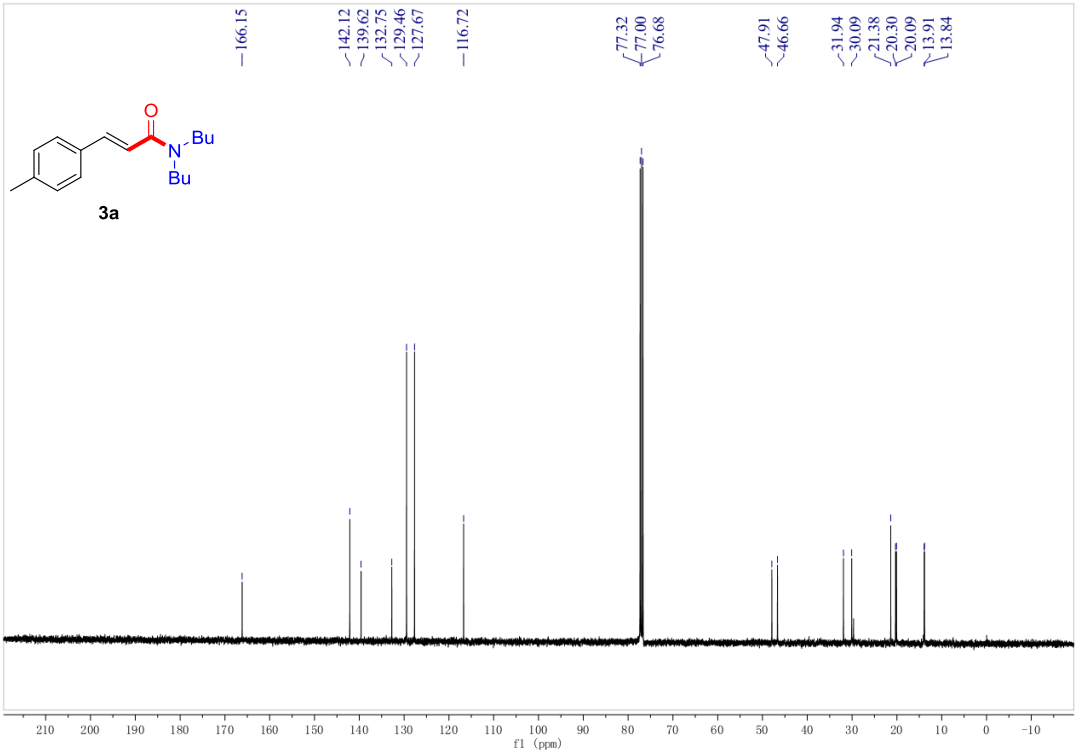
7.423
7.291
7.208
7.188
6.831
6.793
3.466
3.447
3.427
3.407
3.387
2.385
1.643
1.619
1.596
1.406
1.400
1.387
1.381
1.369
1.362
1.012
0.994
0.985
0.976
0.966
O
N
Bu
Bu
3a
0.96
1.96
1.96
0.98
3.97
3.00
4.10
4.11
6.11
f1 (ppm)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142.12
139.62
132.75
129.46
127.67
116.72
77.32
77.00
76.68
47.91
46.66
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20.30
20.09
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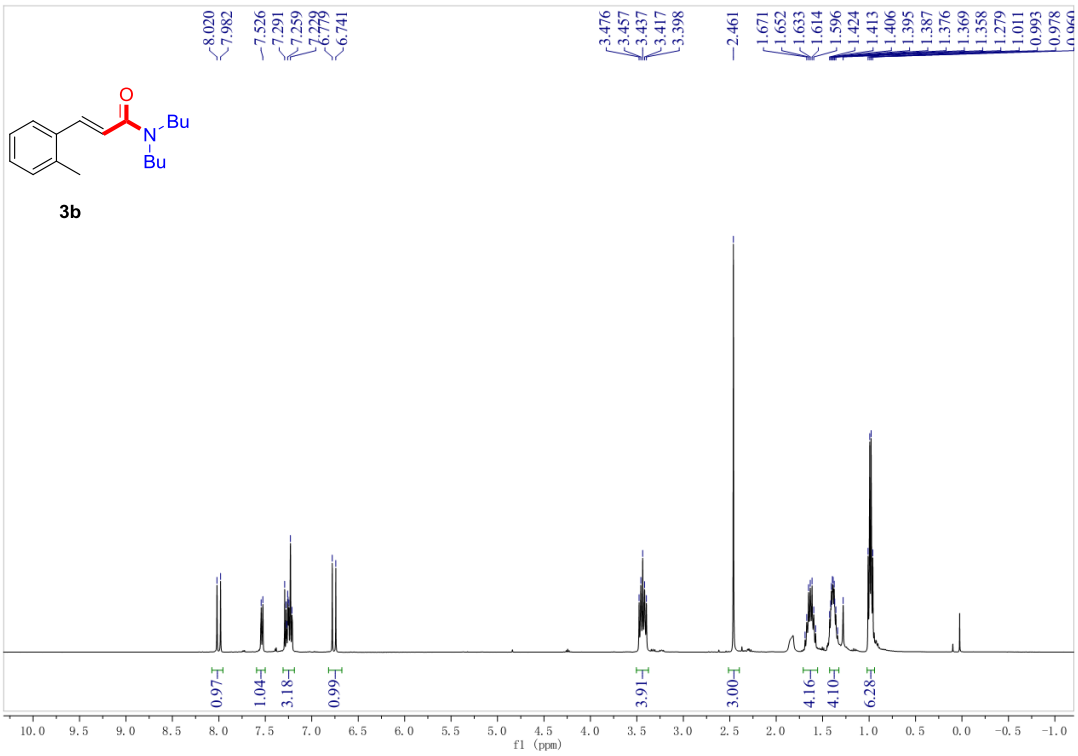
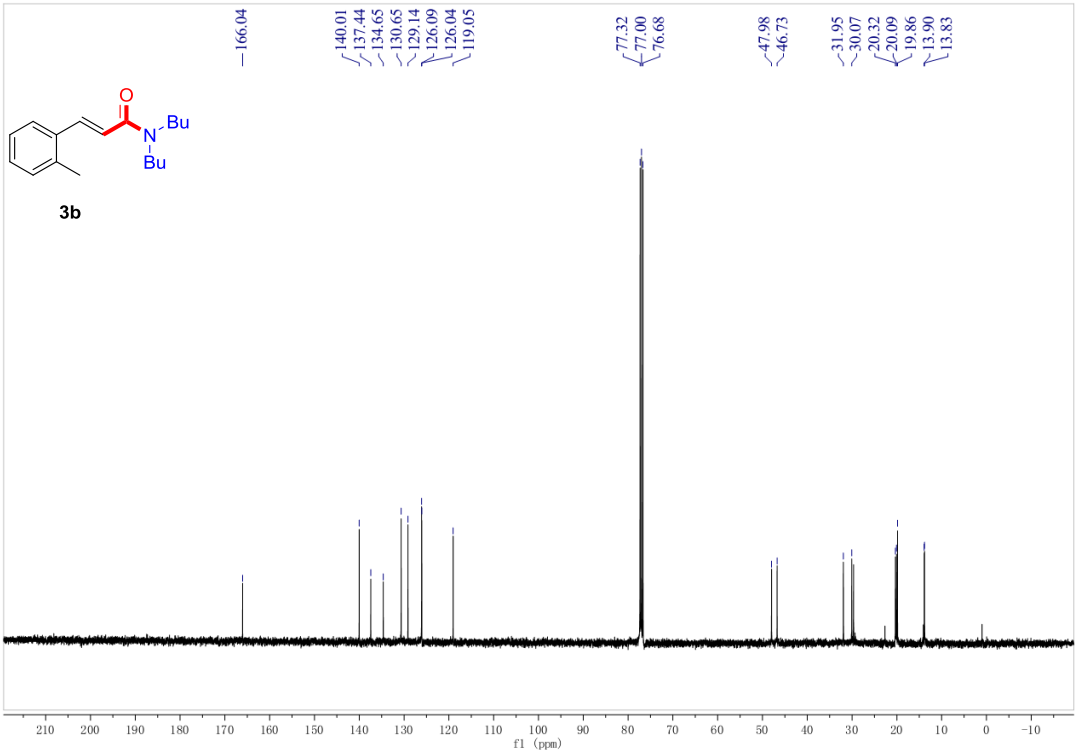
13.84
O
N
Bu
Bu
3a
f1 (ppm)


8.020
7.982
7.526
7.291
7.259
7.229
6.779
6.741
3.476
3.457
3.437
3.417
3.398
2.461
1.671
1.652
1.633
1.614
1.596
1.424
1.413
1.406
1.395
1.387
1.376
1.369
1.358
1.279
1.011
0.993
0.978
O
N
Bu
Bu
3b
0.97
1.04
3.18
0.99
3.91
3.00
4.16
4.10
6.28
f1 (ppm)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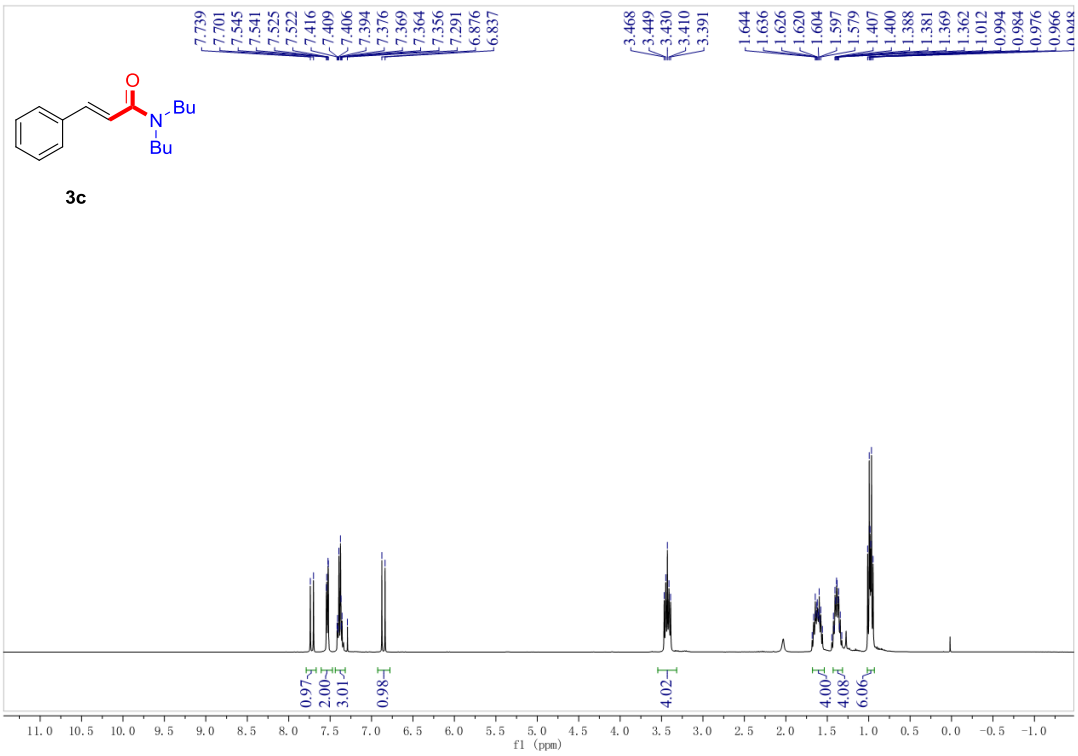
137.44
134.65
130.65
129.14
126.09
126.04
119.05
77.32
77.00
76.68
47.98
46.73
31.95
30.07
20.32
20.09
19.86
13.90
13.83
O
N
Bu
Bu
3b
f1 (ppm)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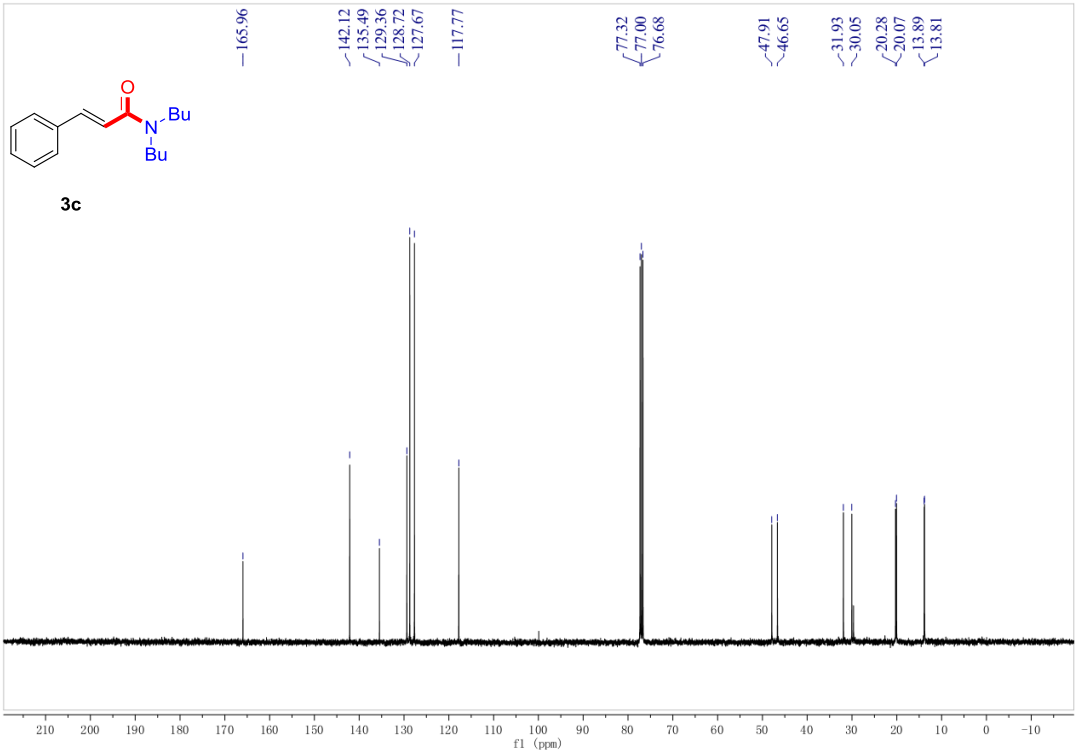
7.545
7.541
7.525
7.522
7.416
7.409
7.406
7.394
7.376
7.369
7.364
7.356
7.291
6.876
6.837
3.468
3.449
3.430
3.410
3.391
1.644
1.636
1.626
1.620
1.604
1.597
1.579
1.407
1.400
1.388
1.381
1.369
1.362
1.012
0.994
0.984
0.976
0.966
O
N
Bu
Bu
3c
0.97
2.00
3.01
0.98
4.02
4.00
4.08
6.06
f1 (ppm)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117.77
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77.00
76.68
47.91
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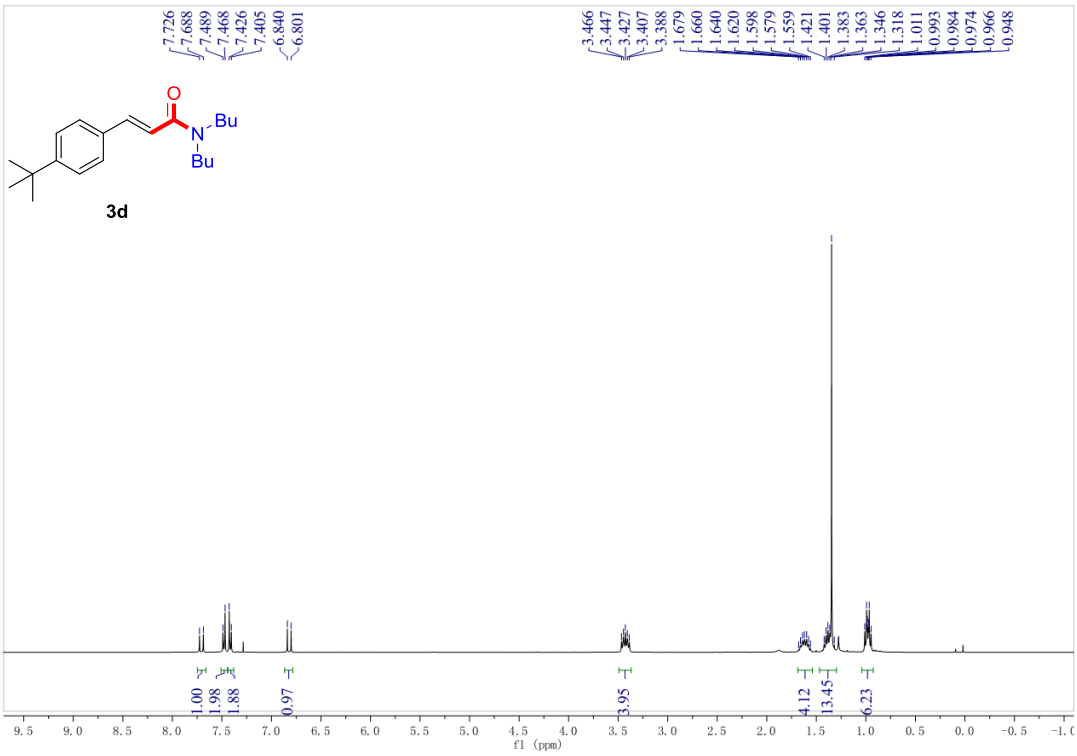
30.05
20.28
20.07
13.89
13.81
O
N
Bu
Bu
3c
f1 (ppm)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7.489
7.468
7.426
7.405
6.840
6.801
3.466
3.447
3.427
3.407
3.388
1.679
1.660
1.640
1.620
1.598
1.579
1.559
1.421
1.401
1.383
1.363
1.346
1.318
1.011
0.993
0.984
0.974
0.966
0.948
O
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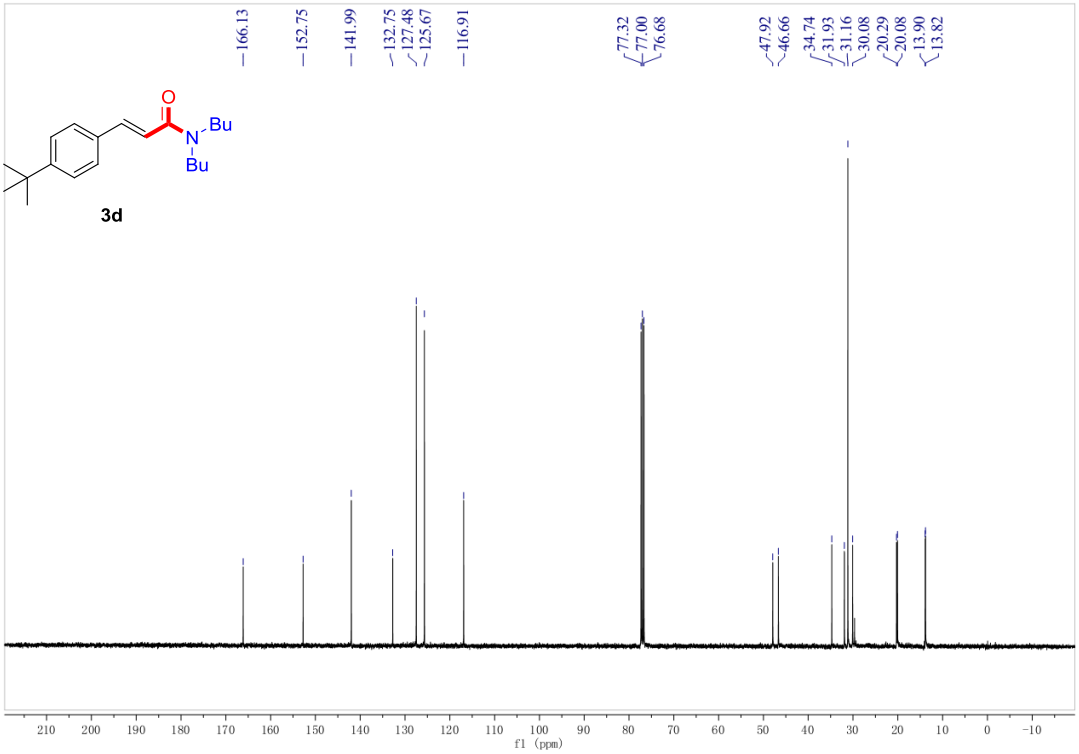
Bu
Bu
3d
1.00
1.98
1.88
0.97
3.95
4.12
13.45
6.23
f1 (ppm)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77.00
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47.92
46.66
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31.93
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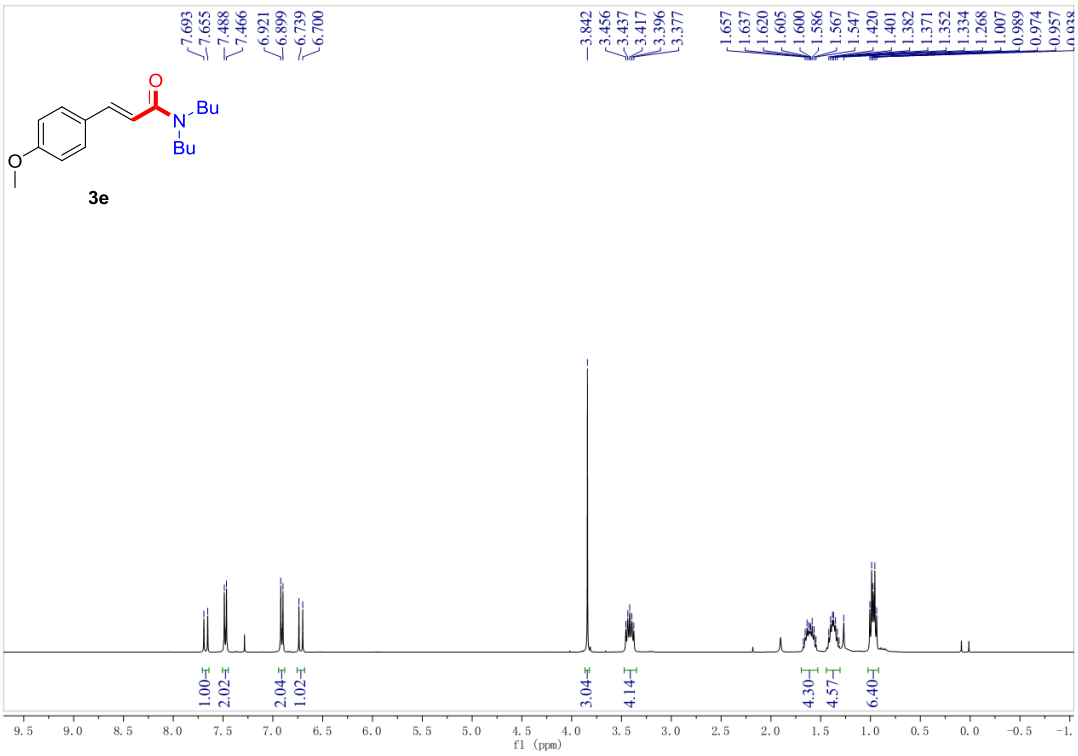
30.08
20.29
20.08
13.90
13.82
O
N
Bu
Bu
3d
f1 (ppm)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6.899
6.739
6.700
3.842
3.456
3.437
3.417
3.396
3.377
1.657
1.637
1.620
1.605
1.600
1.586
1.567
1.547
1.420
1.401
1.382
1.371
1.352
1.334
1.268
1.007
0.989
0.974
0.957
O
N
Bu
Bu
O
3e
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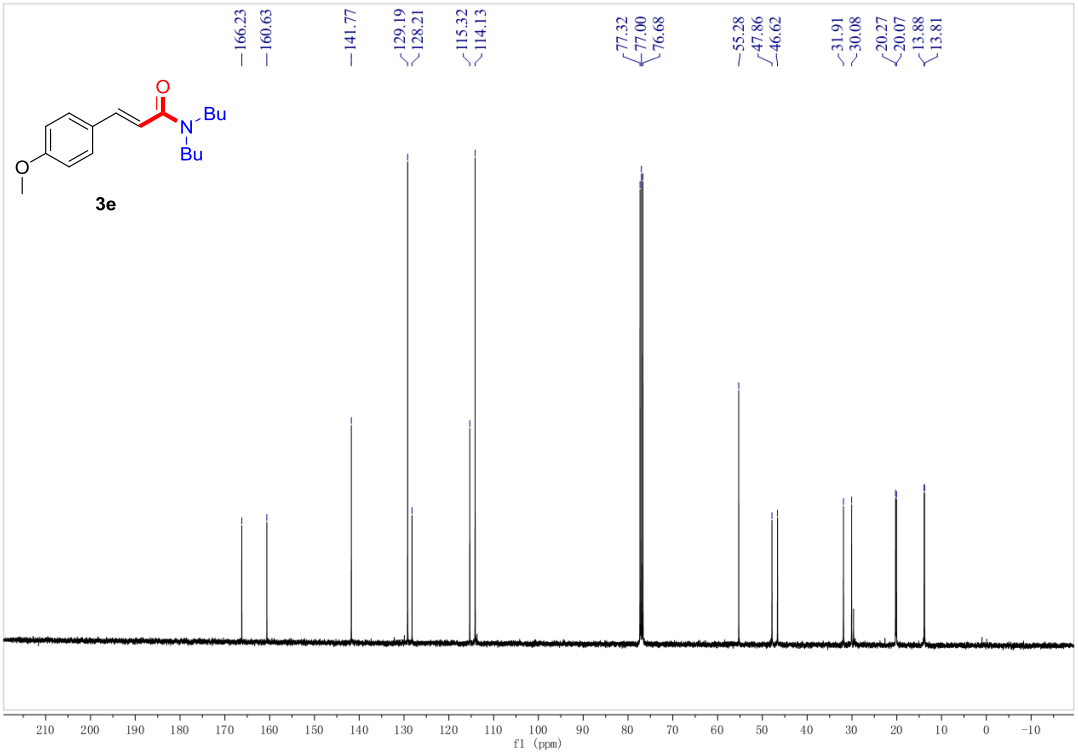
2.02
2.04
1.02
3.04
4.14
4.30
4.57
6.40
f1 (ppm)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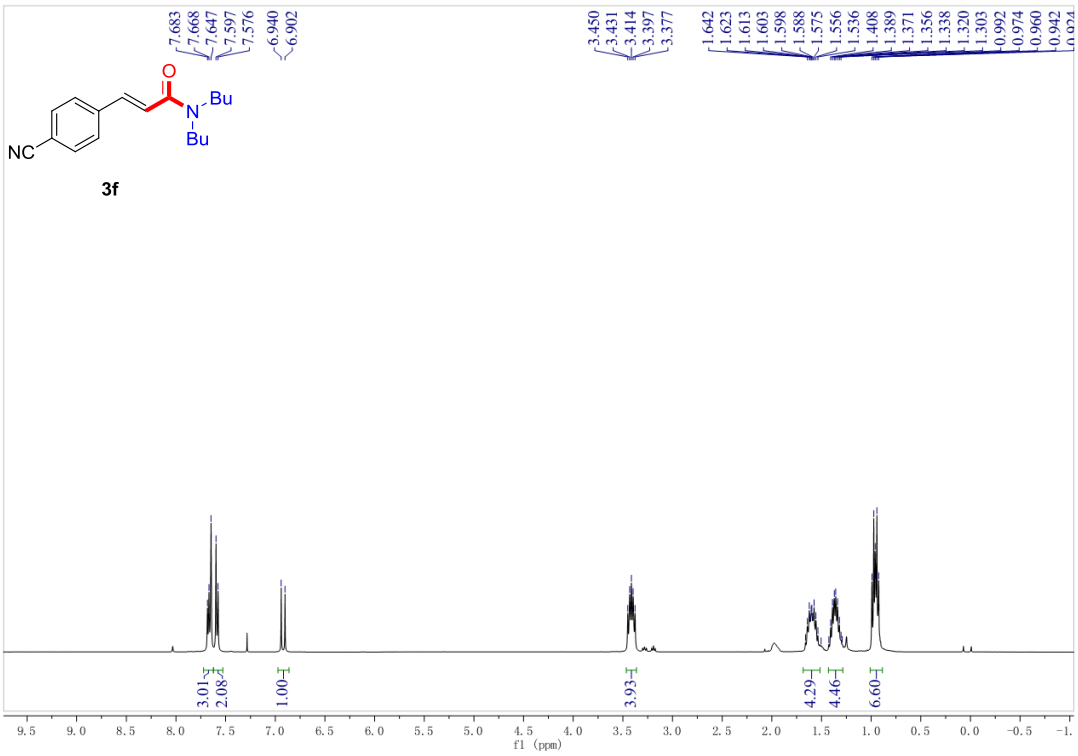
160.63
141.77
129.19
128.21
115.32
114.13
77.32
77.00
76.68
55.28
47.86
46.62
31.91
30.08
20.27
20.07
13.88
13.81
O
N
Bu
Bu
O
3e
f1 (ppm)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6.902
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3.431
3.414
3.397
3.377
1.642
1.623
1.613
1.603
1.598
1.588
1.575
1.556
1.536
1.408
1.389
1.371
1.356
1.338
1.320
1.303
0.992
0.974
0.960
0.942
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Bu
Bu
NC
3f
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2.08
1.00
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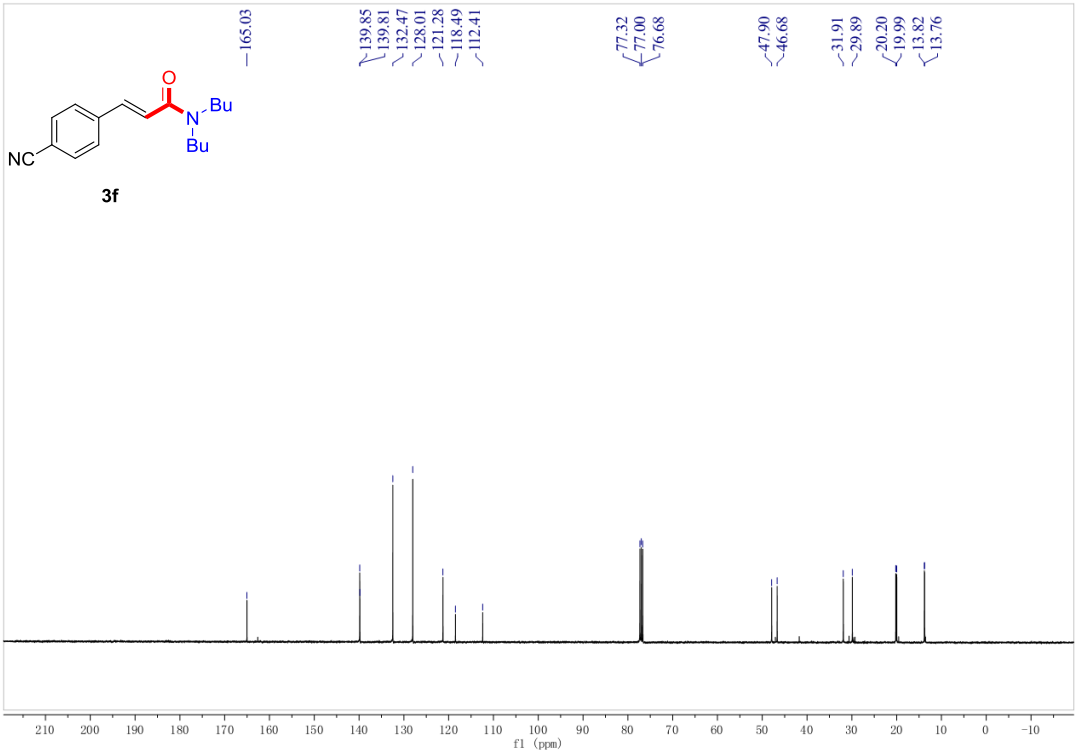
4.29
4.46
6.60
f1 (ppm)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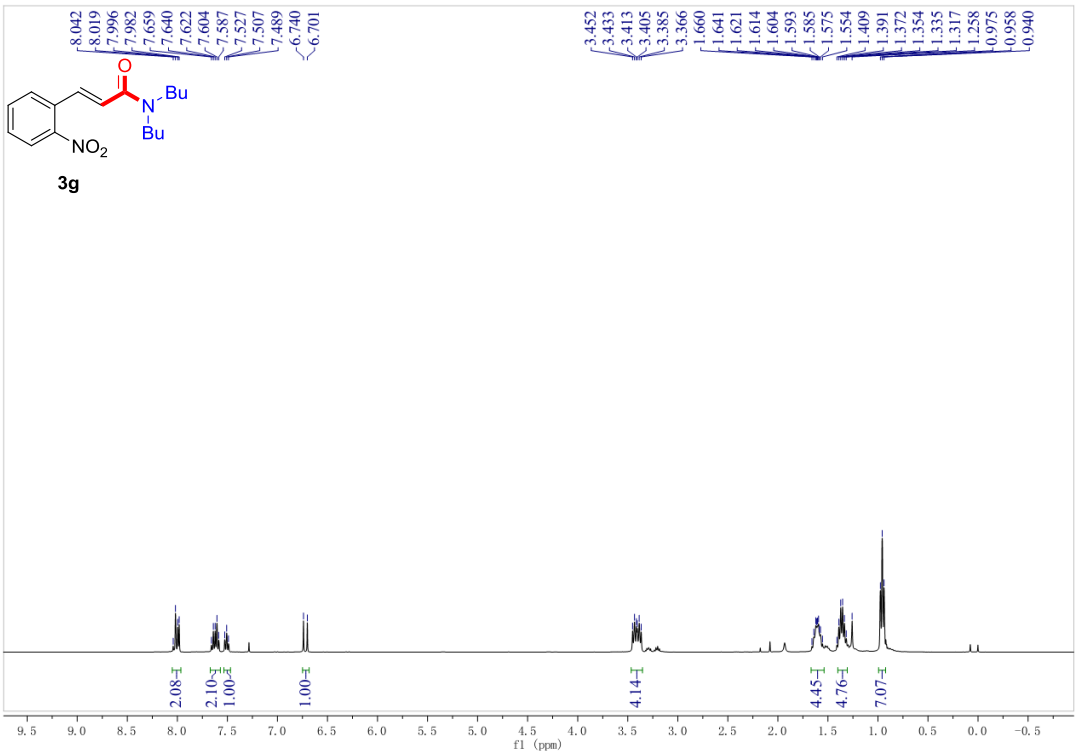
128.01
121.28
118.49
112.41
77.32
77.00
76.68
47.90
46.68
31.91
29.89
20.20
19.99
13.82
13.76
O
N
Bu
Bu
NC
3f
f1 (ppm)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7.640
7.622
7.604
7.587
7.527
7.507
7.489
6.740
6.701
3.452
3.433
3.413
3.405
3.385
3.366
1.660
1.641
1.621
1.614
1.604
1.593
1.585
1.575
1.554
1.409
1.391
1.372
1.354
1.335
1.317
1.258
0.975
0.958
0.940
O
N
Bu
Bu
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2.10
1.00
1.00
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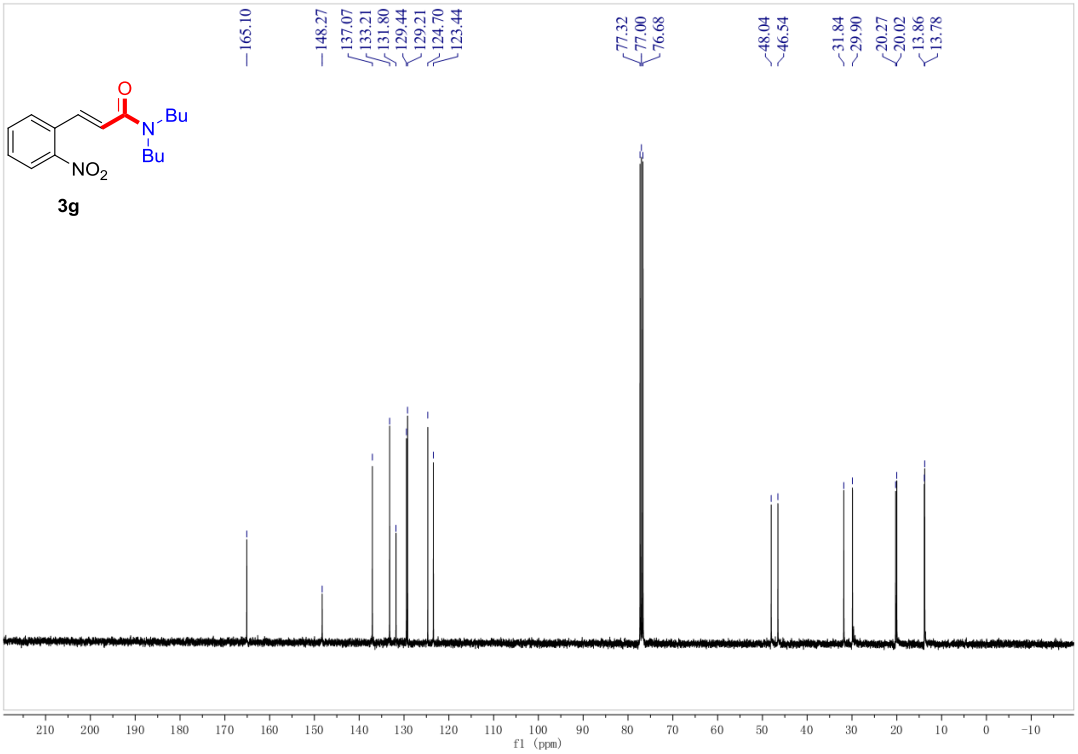
4.45
4.76
7.07
f1 (ppm)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133.21
131.80
129.44
129.21
124.70
123.44
77.32
77.00
76.68
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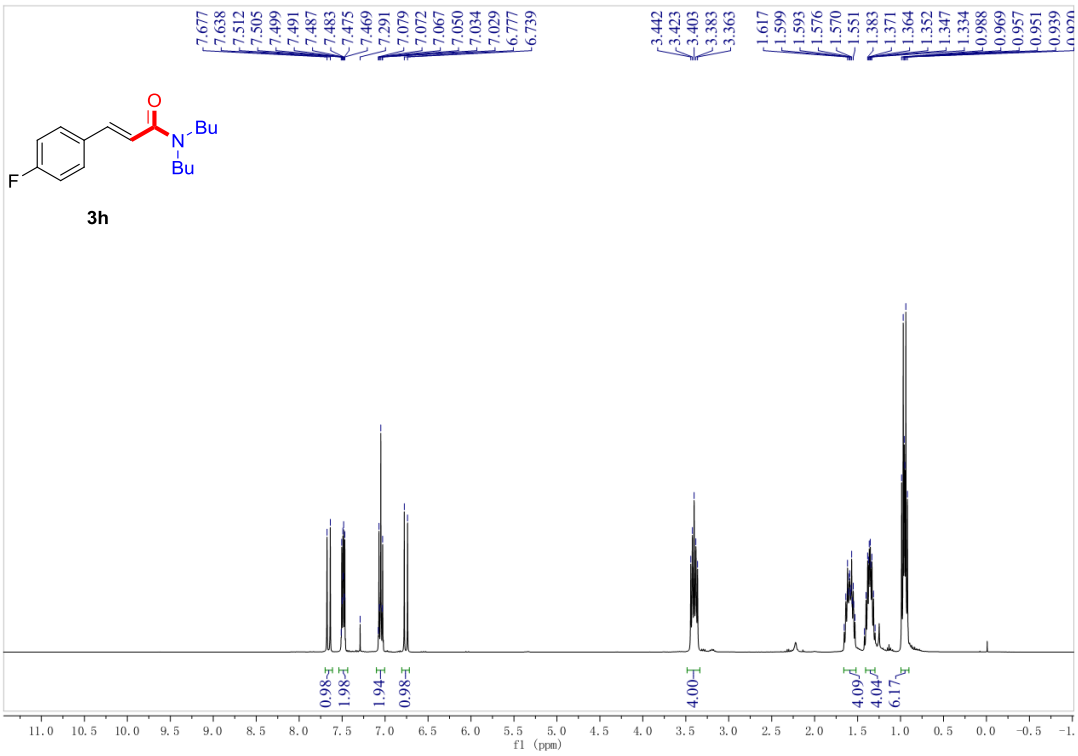
O
N
Bu
Bu
NO2
3g
f1 (ppm)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7.291
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7.072
7.067
7.050
7.034
7.029
6.777
6.739
3.442
3.423
3.403
3.383
3.363
1.617
1.599
1.593
1.576
1.570
1.551
1.383
1.371
1.364
1.352
1.347
1.334
0.988
0.969
0.957
0.951
0.939
O
N
Bu
Bu
F
3h
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1.98
1.94
0.98
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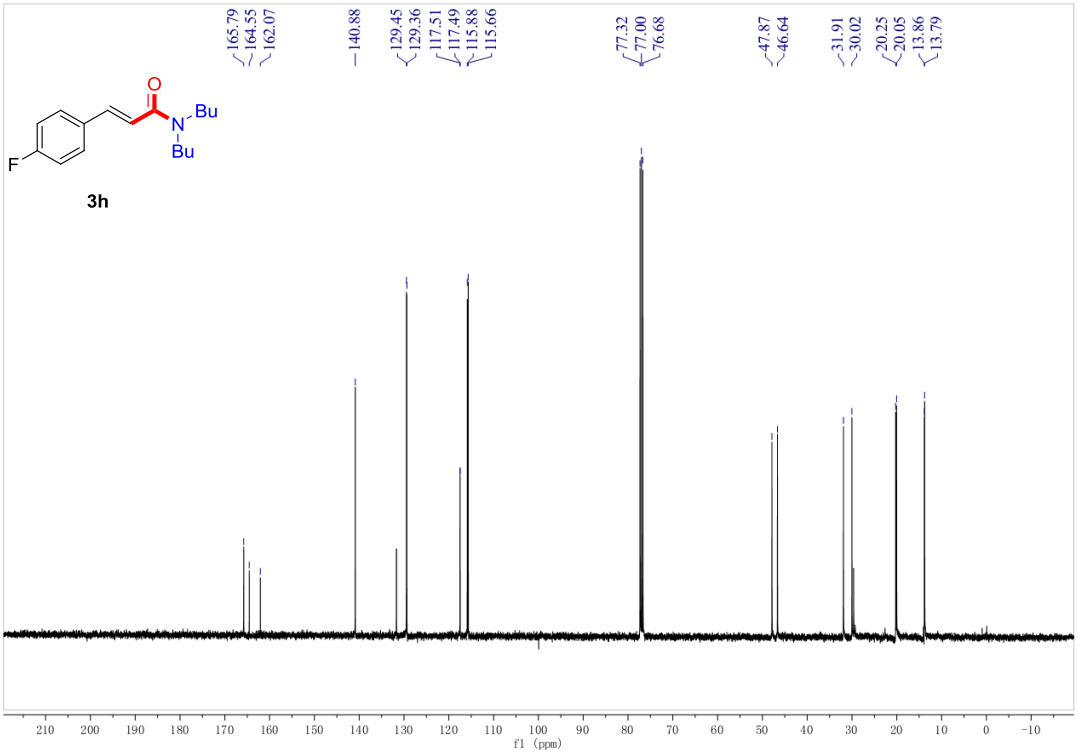
4.09
4.04
6.17
f1 (ppm)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117.49
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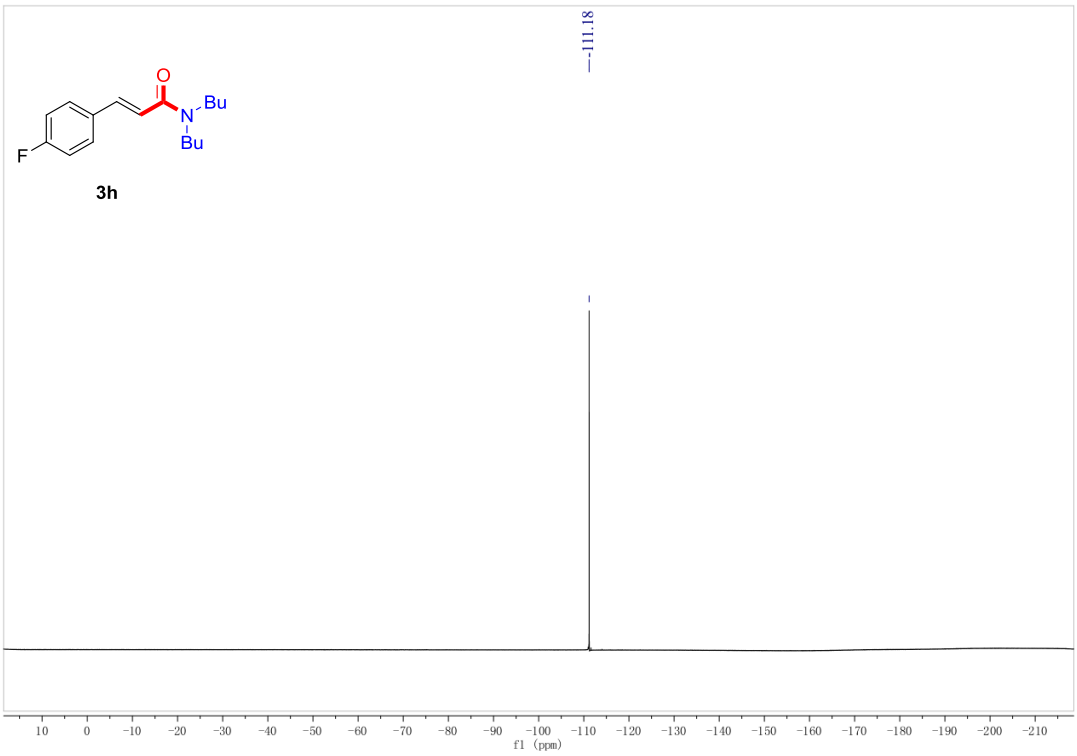
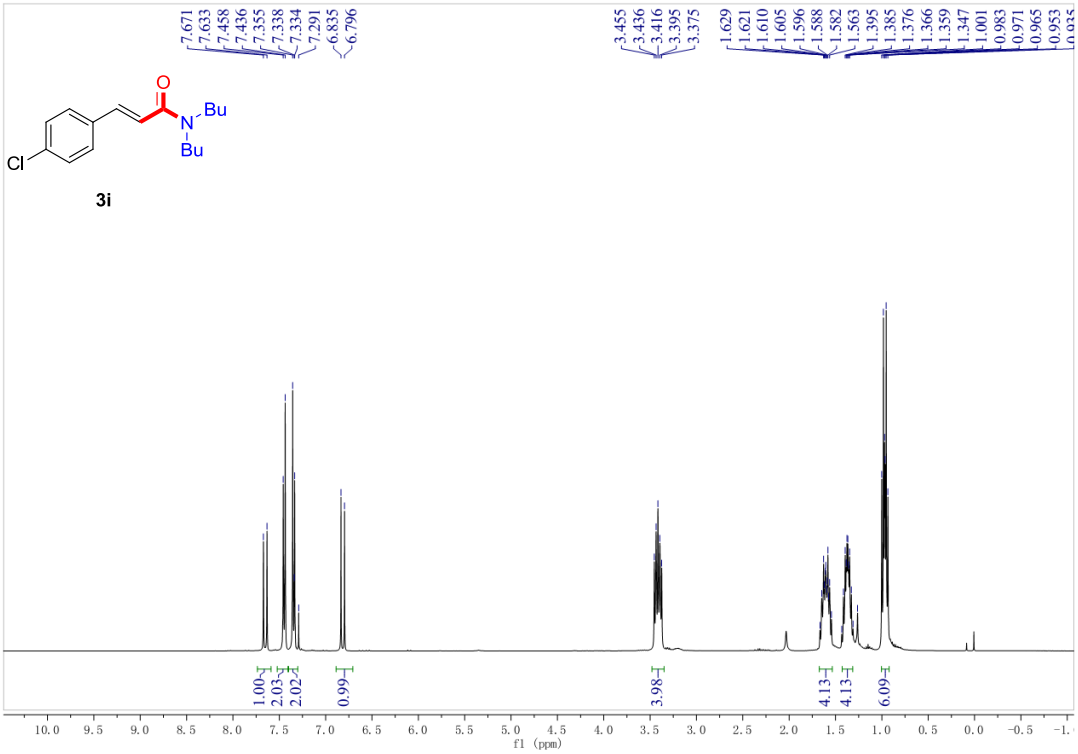
115.66
77.32
77.00
76.68
47.87
46.64
31.91
30.02
20.25
20.05
13.86
13.79
O
N
Bu
Bu
F
3h
f1 (ppm)


O
N
Bu
Bu
F
-111.18
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

**3h**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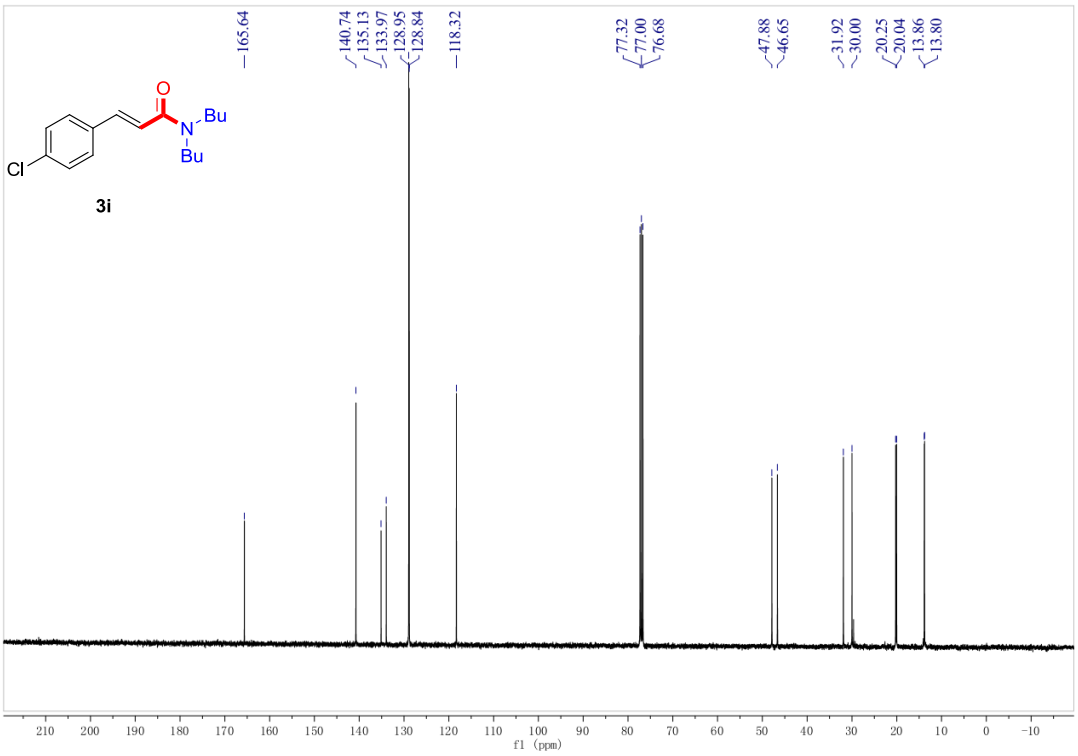
3.416
3.395
3.375
1.629
1.621
1.610
1.605
1.596
1.588
1.582
1.563
1.395
1.385
1.376
1.366
1.359
1.347
1.001
0.983
0.971
0.965
0.953
O
N
Bu
Bu
Cl
1.00
2.03
2.02
0.99
3.98
4.13
4.13
6.09
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

**3i**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133.97
128.95
128.84
118.32
77.32
77.00
76.68
47.88
46.65
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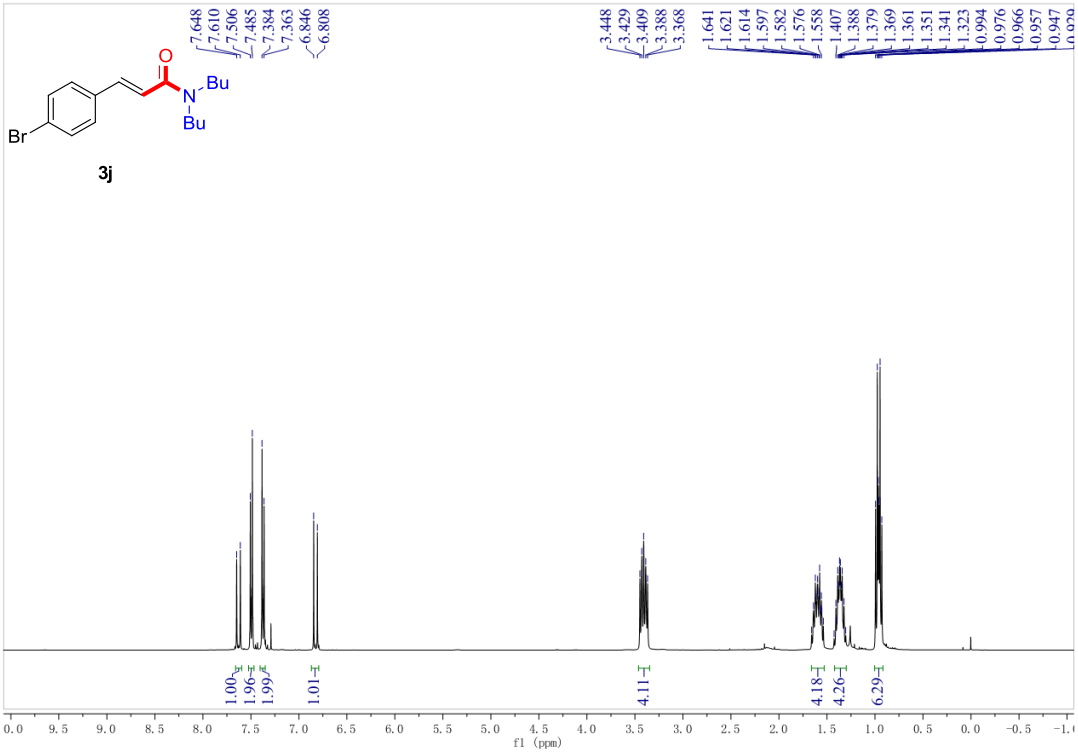
20.25
20.04
13.86
13.80
O
N
Bu
Bu
Cl
f1 (ppm)

**3i**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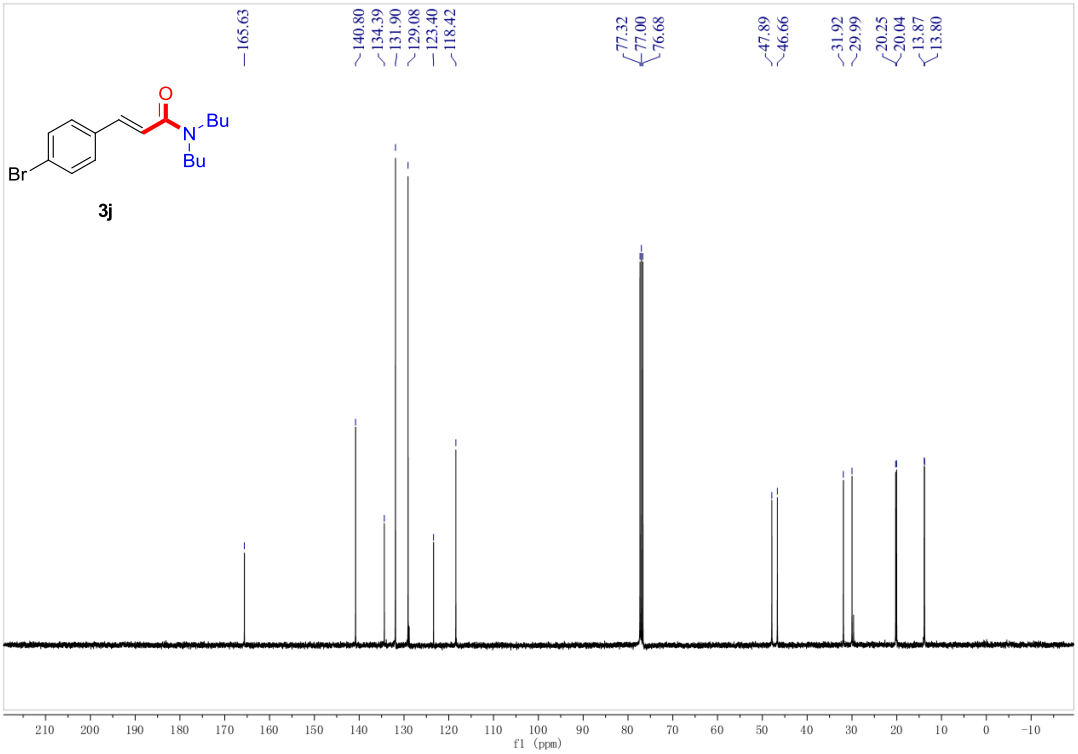
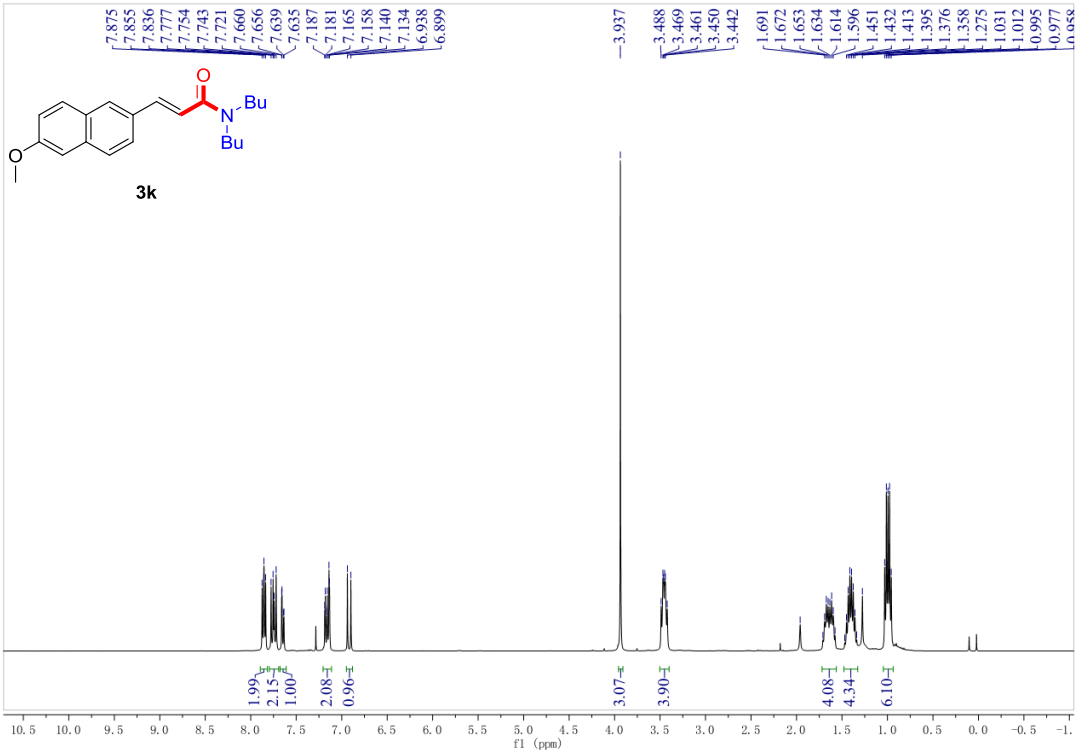
3.429
3.409
3.388
3.368
1.641
1.621
1.614
1.597
1.582
1.576
1.558
1.407
1.388
1.379
1.369
1.361
1.351
1.341
1.323
0.994
0.976
0.966
0.957
0.947
O
N
Bu
Bu
Br
1.00
1.96
1.99
1.01
4.11
4.18
4.26
6.29
f1 (ppm)

**3j**


165.63
140.80
134.39
131.90
129.08
123.40
118.42
77.32
77.00
76.68
47.89
46.66
31.92
29.99
20.25
20.04
13.87
13.80
O
N
Bu
Bu
Br
3j
f1 (ppm)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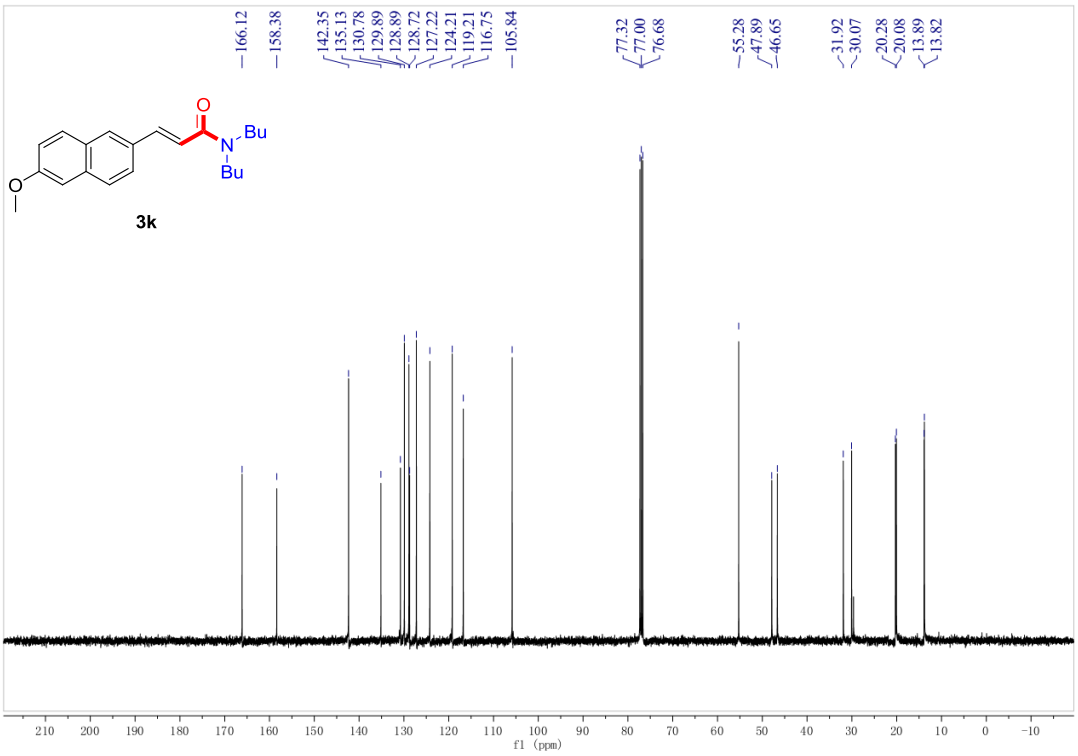
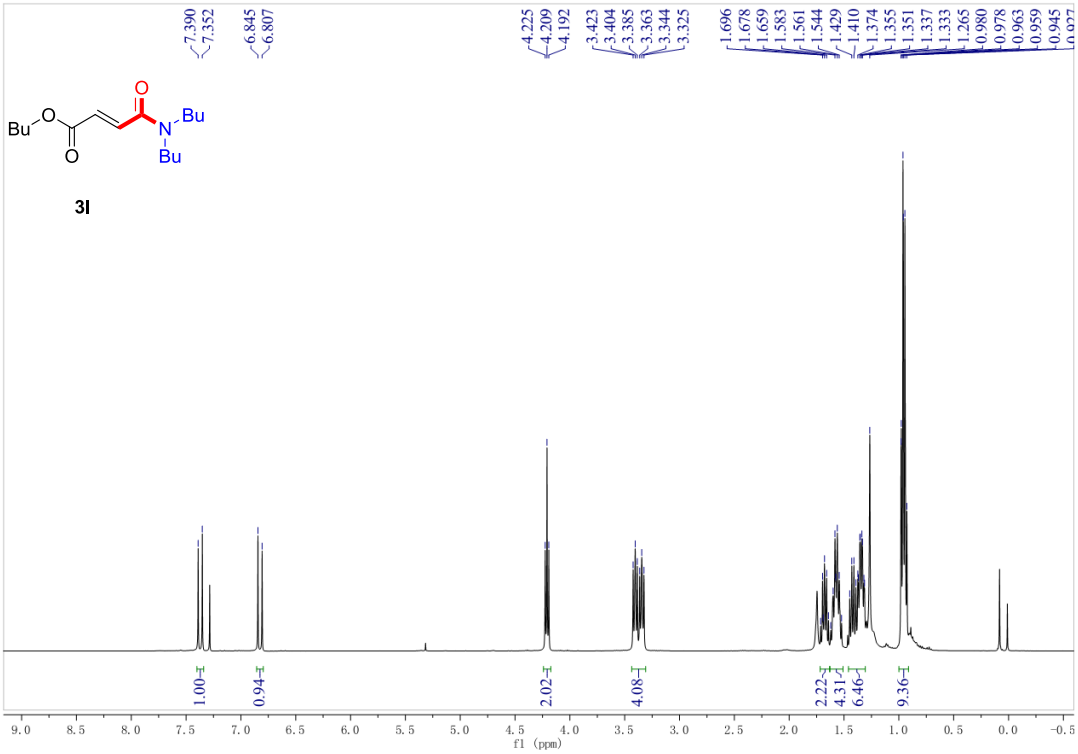
7.855
7.836
7.777
7.754
7.743
7.721
7.660
7.656
7.639
7.635
7.187
7.181
7.165
7.158
7.140
7.134
6.938
6.899
3.937
3.488
3.469
3.461
3.450
3.442
1.691
1.672
1.653
1.634
1.614
1.596
1.451
1.432
1.413
1.395
1.376
1.358
1.275
1.031
1.012
0.995
0.977
O
N
Bu
Bu
O
3k
1.99
2.15
1.00
2.08
0.96
3.07
3.90
4.08
4.34
6.10
f1 (ppm)


166.12
158.38
142.35
135.13
130.78
129.89
128.89
128.72
127.22
124.21
119.21
116.75
105.84
77.32
77.00
76.68
55.28
47.89
46.65
31.92
30.07
20.28
20.08
13.89
13.82
O
N
Bu
Bu
O
3k
f1 (ppm)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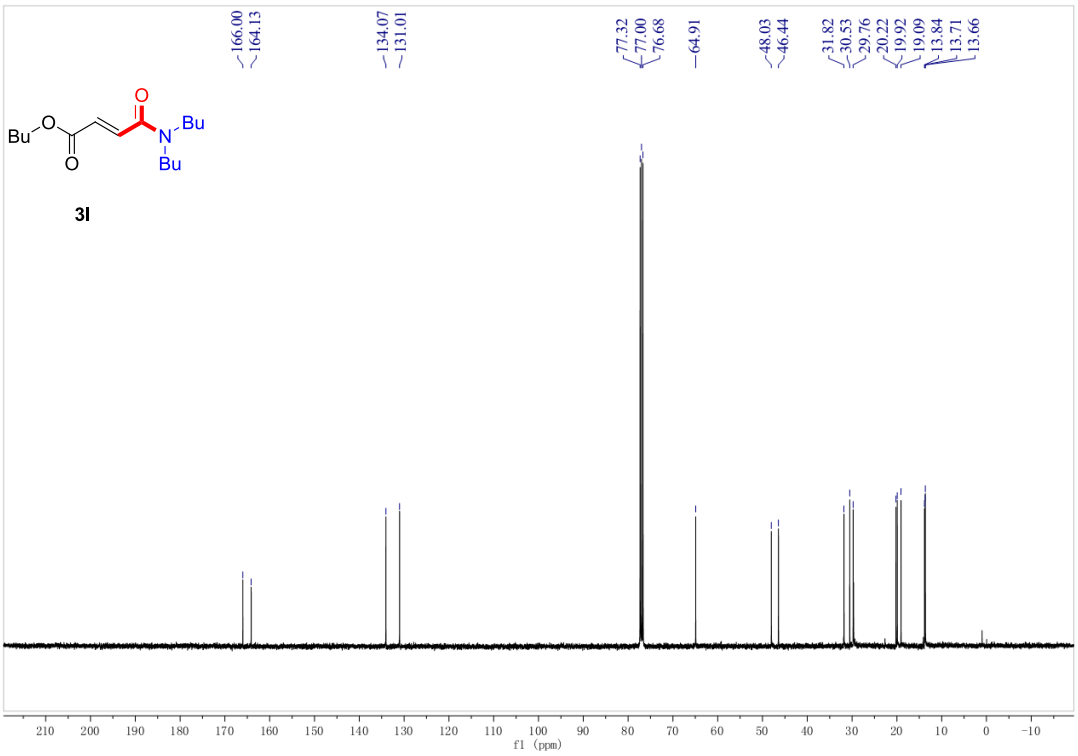
7.352
6.845
6.807
4.225
4.209
4.192
3.423
3.404
3.385
3.363
3.344
3.325
1.696
1.678
1.659
1.583
1.561
1.544
1.429
1.410
1.374
1.355
1.351
1.337
1.333
1.265
0.980
0.978
0.963
0.959
0.945
Bu
O
O
O
N
Bu
Bu
3l
1.00
0.94
2.02
4.08
2.22
4.31
6.46
9.36
f1 (ppm)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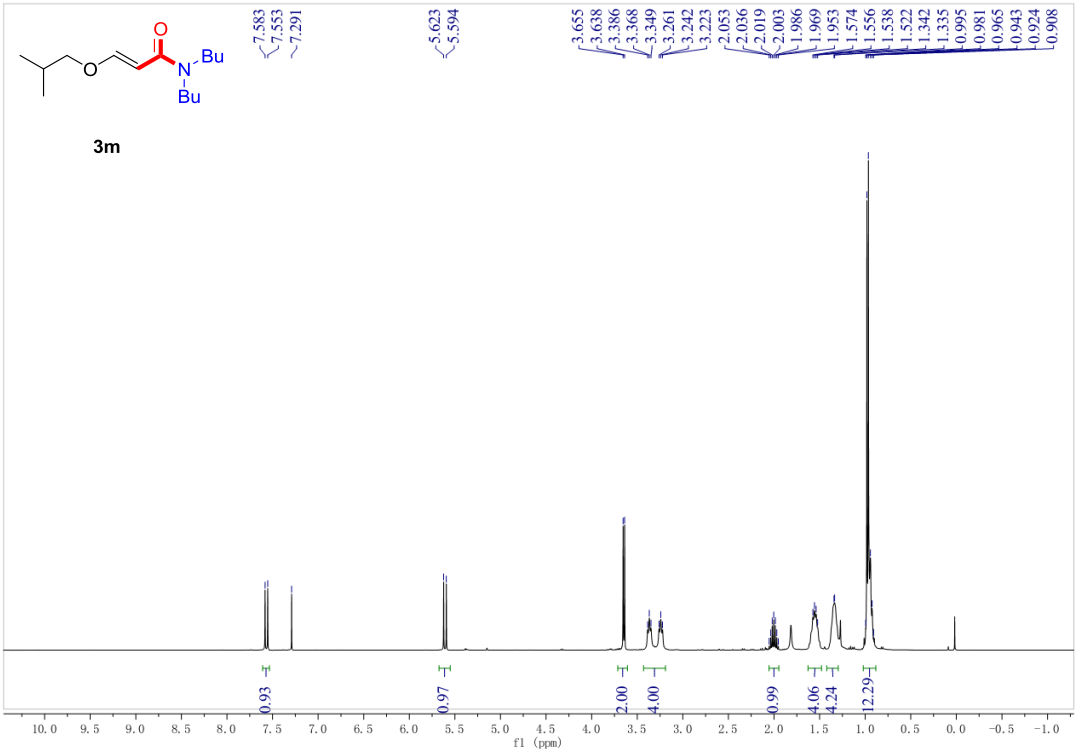
164.13
134.07
131.01
77.32
77.00
76.68
64.91
48.03
46.44
31.82
30.53
29.76
20.22
19.92
19.09
13.84
13.71
13.66
Bu
O
O
O
N
Bu
Bu
f1 (ppm)

**3l**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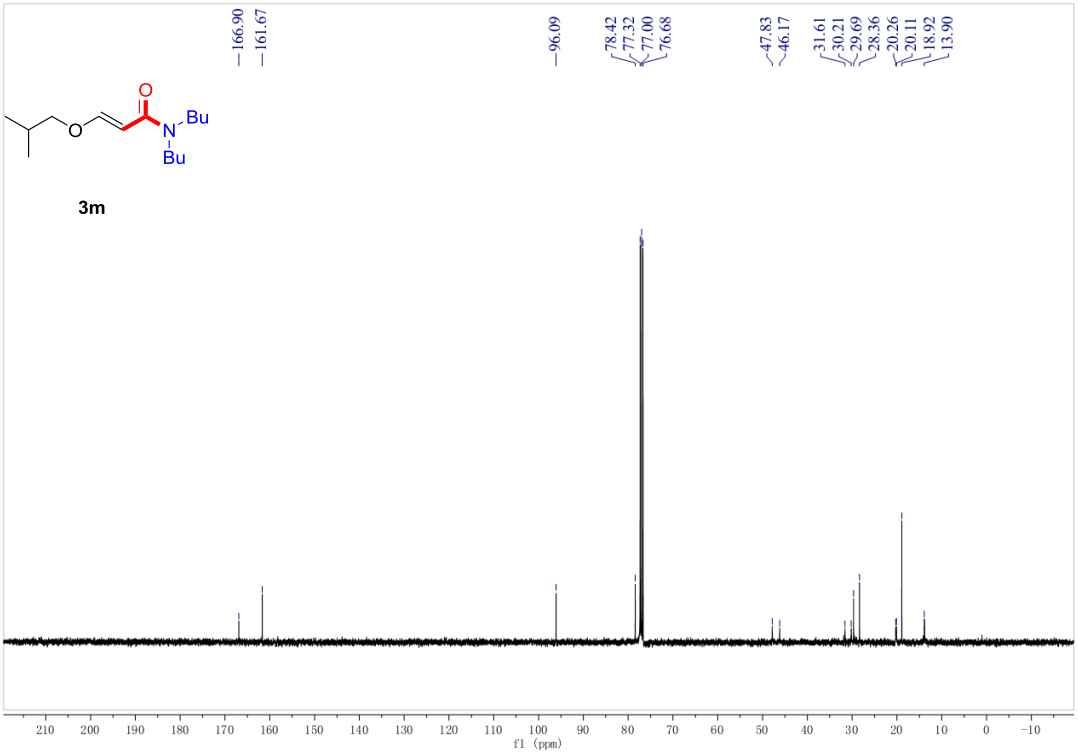
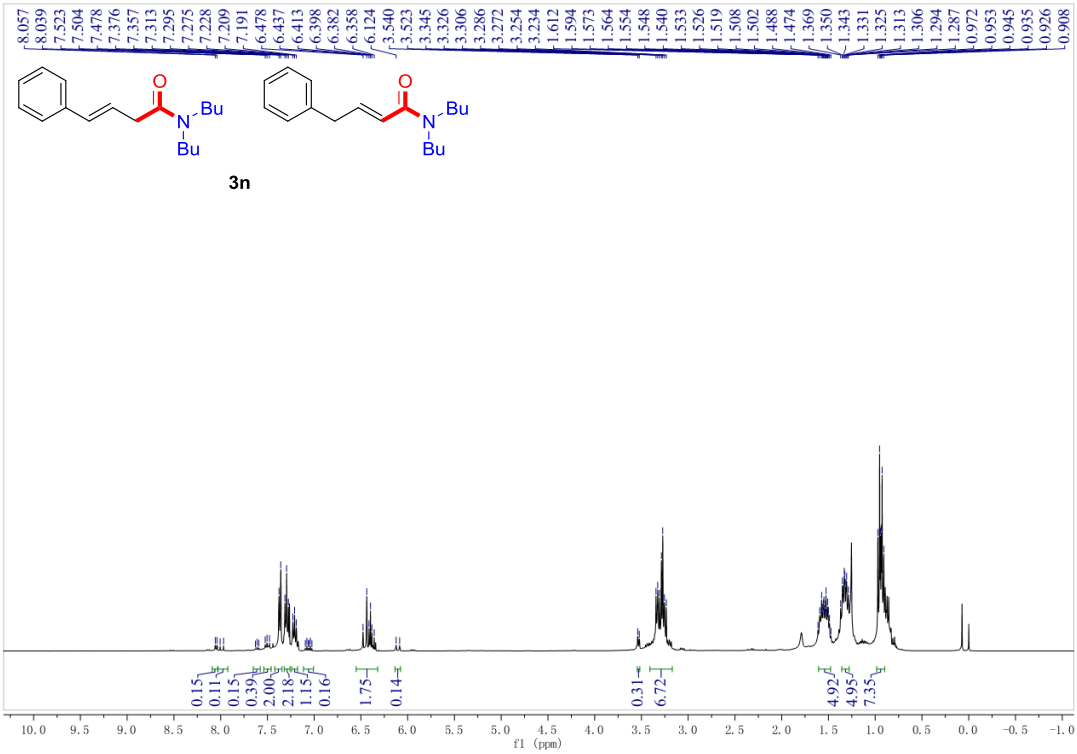
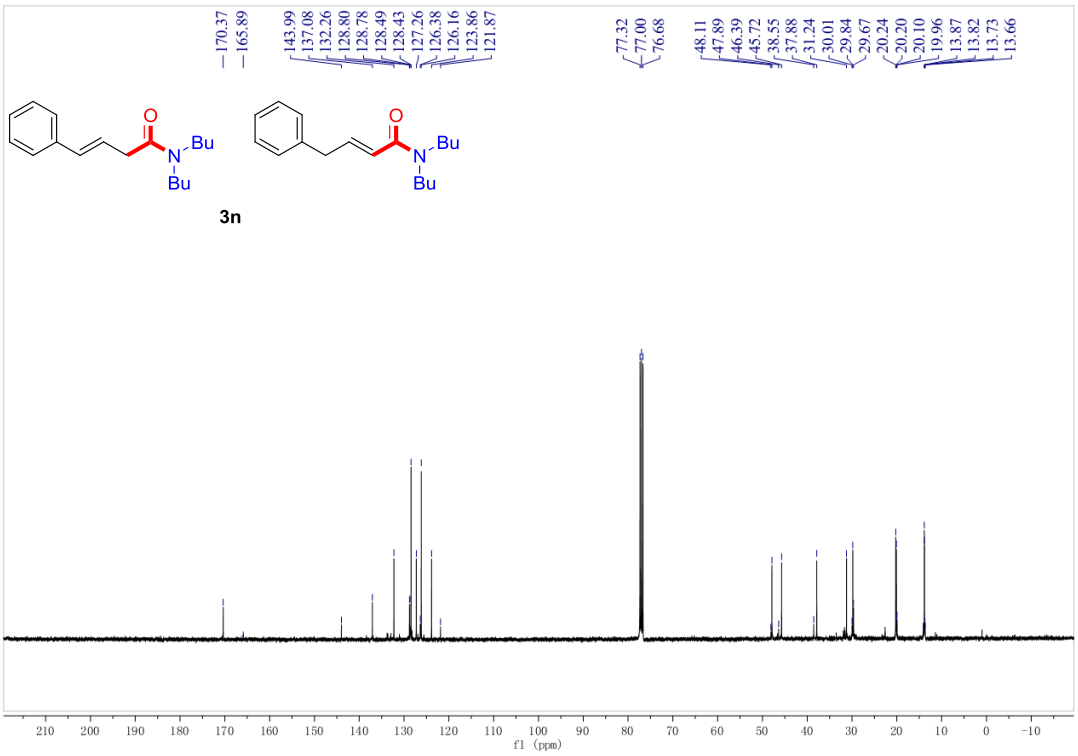
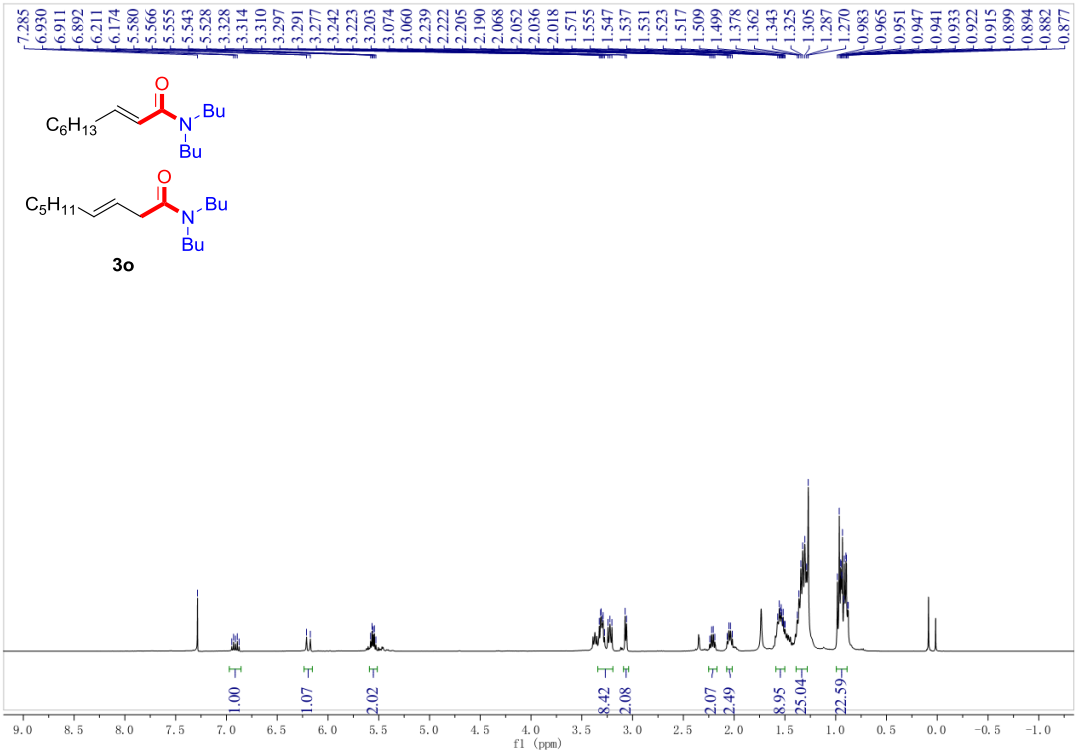
7.553
7.291
5.623
5.594
3.655
3.638
3.386
3.368
3.349
3.261
3.242
3.223
2.053
2.036
2.019
2.003
1.986
1.969
1.953
1.574
1.556
1.538
1.522
1.342
1.335
0.995
0.981
0.965
0.943
0.924
0.908
O
O
N
Bu
Bu
0.93
0.97
2.00
4.00
0.99
4.06
4.24
12.29
f1 (ppm)

**3m**


166.90
161.67
96.09
78.42
77.32
77.00
76.68
47.83
46.17
31.61
30.21
29.69
28.36
20.26
20.11
18.92
13.90
O
N
Bu
Bu
O
3m
f1 (ppm)


8.057
8.039
7.523
7.504
7.478
7.376
7.357
7.313
7.295
7.275
7.228
7.209
7.191
6.478
6.437
6.413
6.398
6.382
6.358
6.124
3.540
3.523
3.345
3.326
3.306
3.286
3.272
3.254
3.234
1.612
1.594
1.573
1.564
1.554
1.548
1.540
1.533
1.526
1.519
1.508
1.502
1.488
1.474
1.369
1.350
1.343
1.331
1.325
1.313
1.306
1.294
1.287
0.972
0.953
0.945
0.935
0.926
0.908
O
N
Bu
Bu
3n
0.15
0.11
0.15
0.39
2.00
2.18
1.15
0.16
1.75
0.14
0.31
6.72
4.92
4.95
7.35
f1 (ppm)


170.37
165.89
143.99
137.08
132.26
128.80
128.78
128.49
128.43
127.26
126.38
126.16
123.86
121.87
77.32
77.00
76.68
48.11
47.89
46.39
45.72
38.55
37.88
31.24
30.01
29.84
29.67
20.24
20.20
20.10
19.96
13.87
13.82
13.73
13.66
O
N
Bu
Bu
Bu
Bu
3n
f1 (ppm)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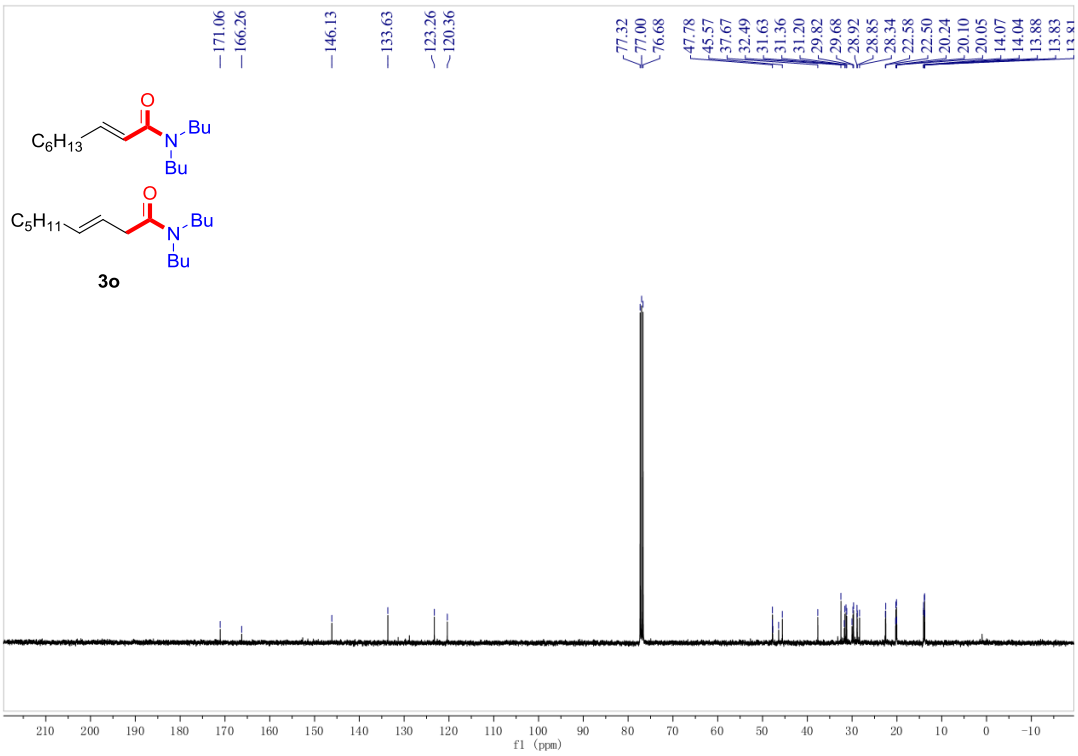
6.930
6.911
6.892
6.211
6.174
5.580
5.566
5.555
5.543
5.528
3.328
3.314
3.310
3.297
3.291
3.277
3.242
3.223
3.203
3.074
3.060
2.239
2.222
2.205
2.190
2.068
2.052
2.036
2.018
1.571
1.555
1.547
1.537
1.531
1.523
1.517
1.509
1.499
1.378
1.362
1.343
1.325
1.305
1.287
1.270
0.983
0.965
0.951
0.947
0.941
0.933
0.922
0.915
0.899
0.894
0.882
0.877
C6H13
C5H11
O
N
Bu
Bu
Bu
Bu
3o
1.00
1.07
2.02
8.42
2.08
2.07
2.49
8.95
25.04
22.59
f1 (ppm)


171.06
166.26
146.13
133.63
123.26
120.36
77.32
77.00
76.68
47.78
45.57
37.67
32.49
31.63
31.36
31.20
29.82
29.68
28.92
28.85
28.34
22.58
22.50
20.24
20.10
20.05
14.07
14.04
13.88
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C6H13
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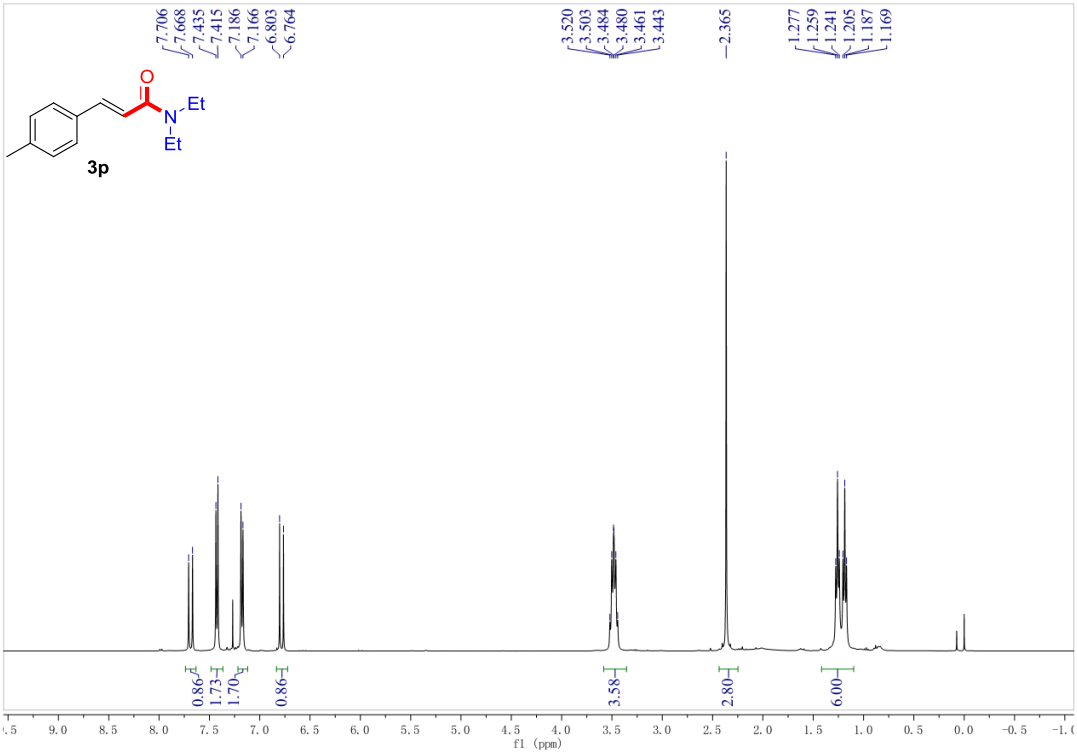
Bu
Bu
C5H11
O
N
Bu
Bu
3o
f1 (ppm)


7.706
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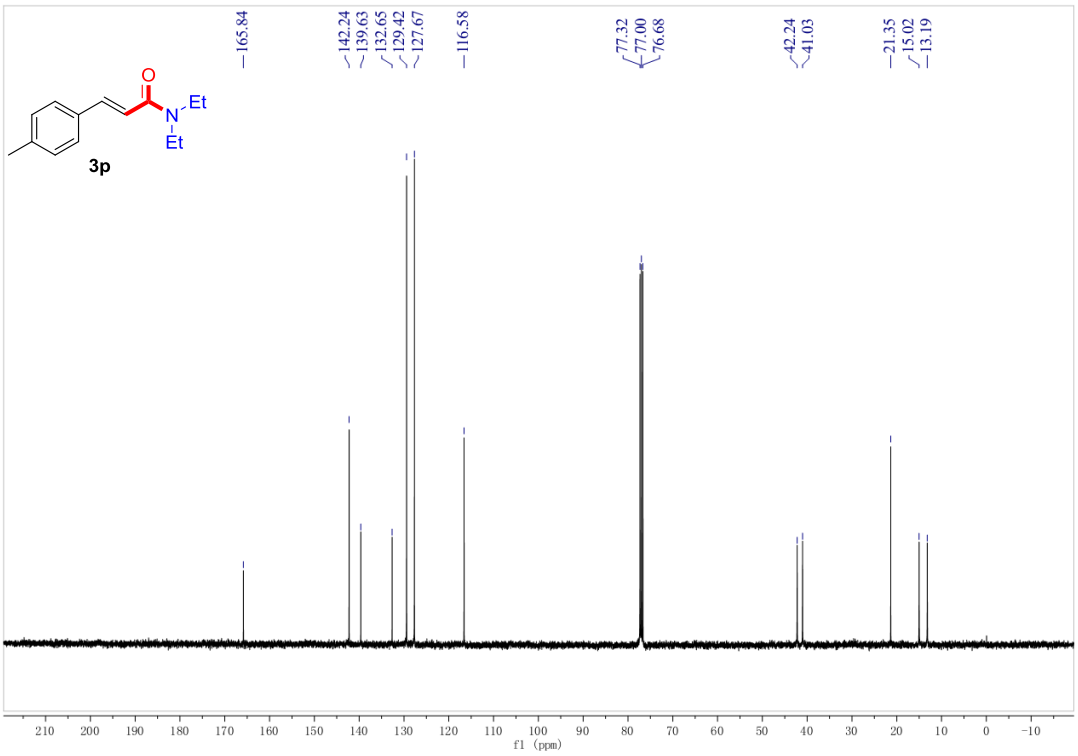
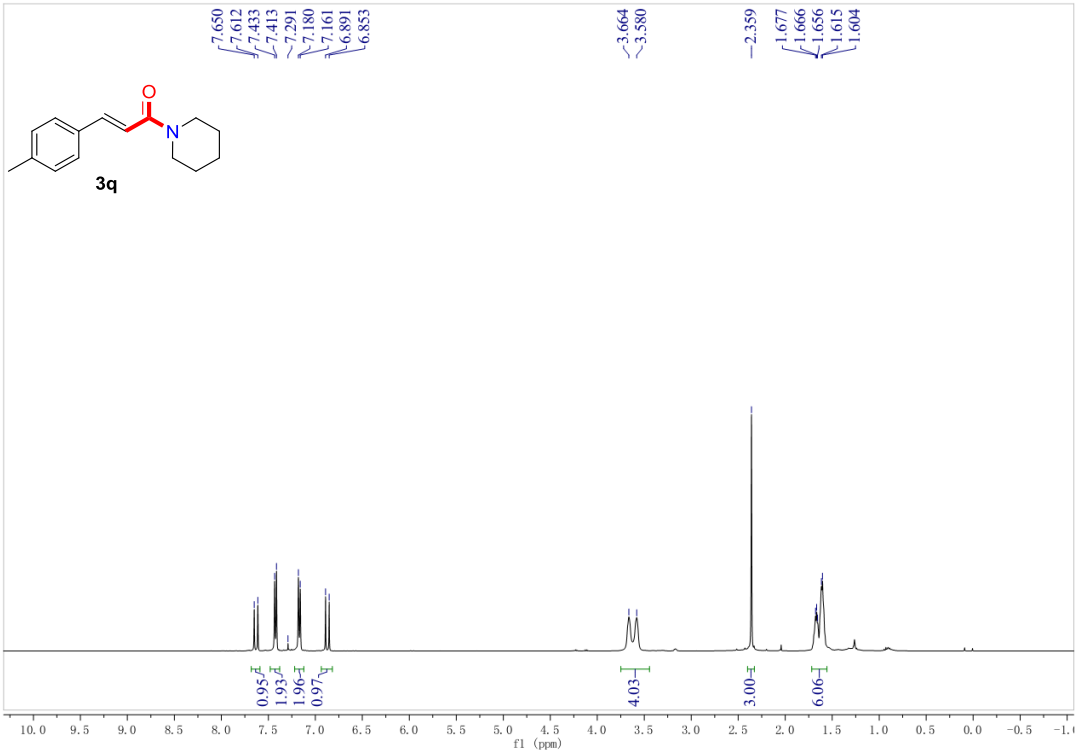
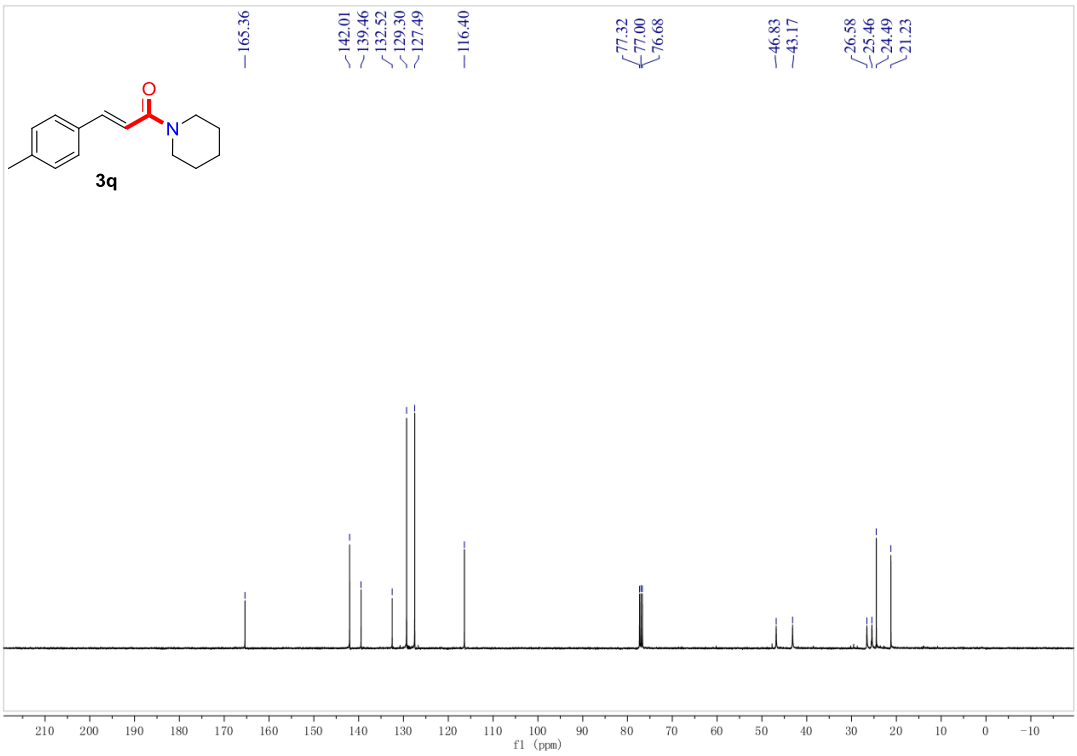
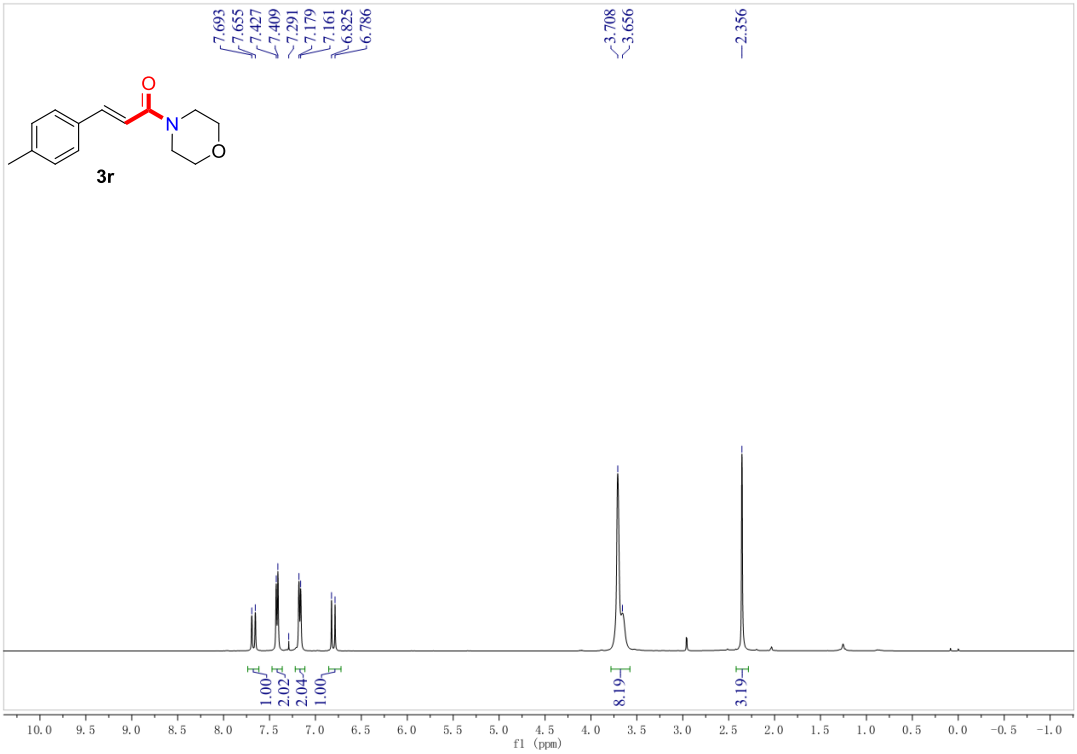
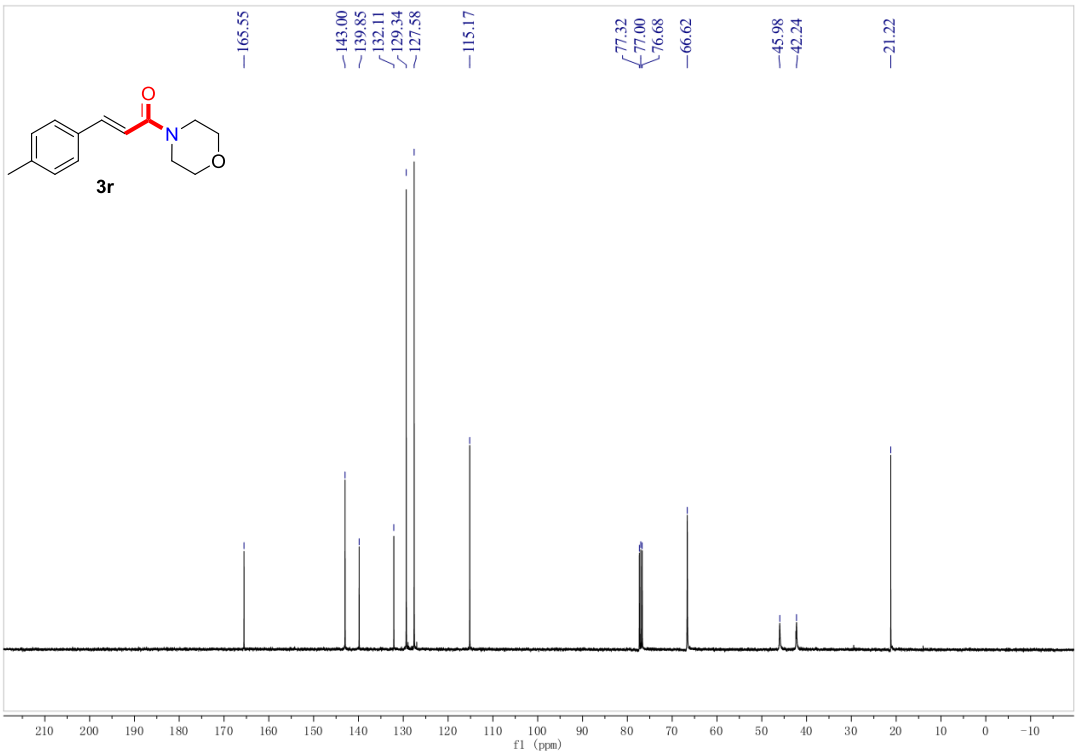
7.435
7.415
7.186
7.166
6.803
6.764
3.520
3.503
3.484
3.480
3.461
3.443
2.365
1.277
1.259
1.241
1.205
1.187
1.169
O
N
Et
Et
3p
0.86
1.73
1.70
0.86
3.58
2.80
6.00
f1 (ppm)


165.84
142.24
139.63
132.65
129.42
127.67
116.58
77.32
77.00
76.68
42.24
41.03
21.35
15.02
13.19
O
N
Et
Et
3p
f1 (ppm)


7.650
7.612
7.433
7.413
7.291
7.180
7.161
6.891
6.853
3.664
3.580
2.359
1.677
1.666
1.656
1.615
1.604
O
N
3q
0.95
1.93
1.96
0.97
4.03
3.00
6.06
f1 (ppm)


165.36
142.01
139.46
132.52
129.30
127.49
116.40
77.32
77.00
76.68
46.83
43.17
26.58
25.46
24.49
21.23
O
N
3q
f1 (ppm)


7.693
7.655
7.427
7.409
7.291
7.179
7.161
6.825
6.786
3.708
3.656
2.356
O
N
O
3r
1.00
2.02
2.04
1.00
8.19
3.19
f1 (ppm)


165.55
143.00
139.85
132.11
129.34
127.58
115.17
77.32
77.00
76.68
66.62
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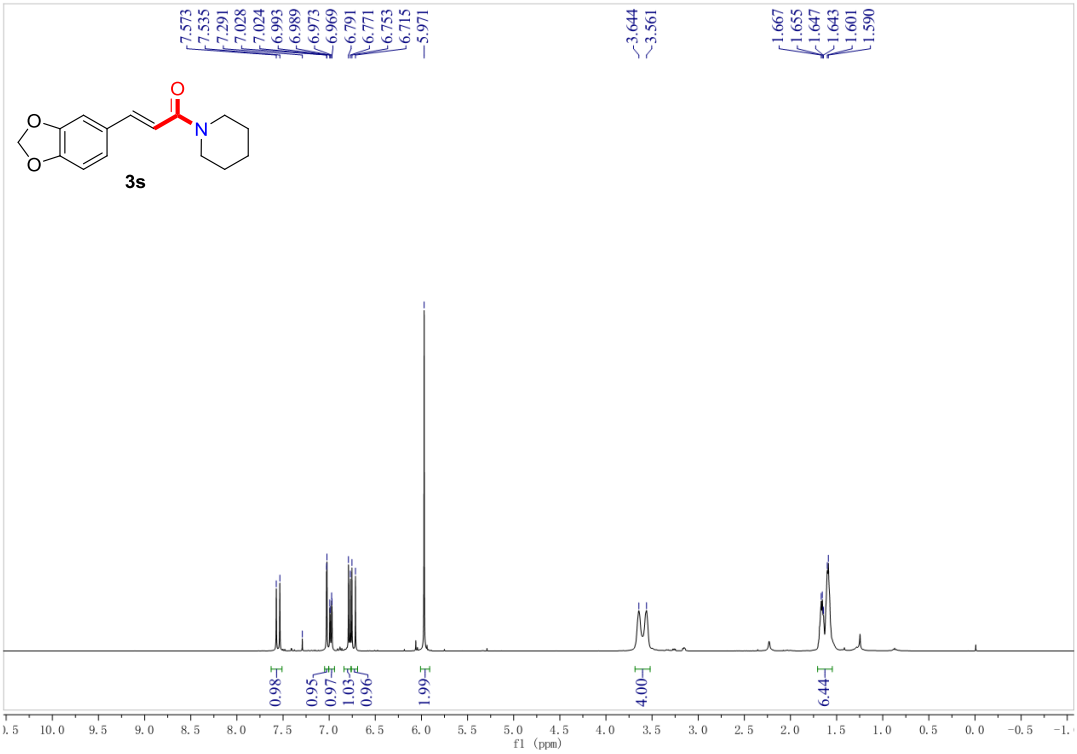
42.24
21.22
3r
f1 (ppm)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7.024
6.993
6.989
6.973
6.969
6.791
6.771
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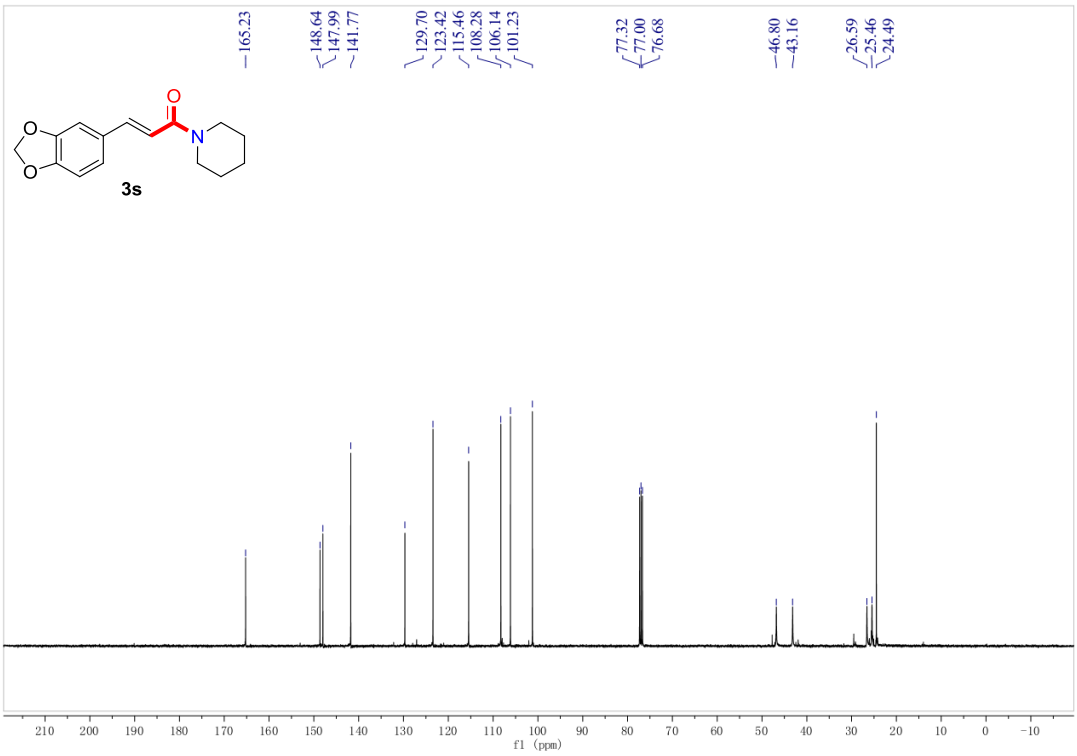
6.715
5.971
3.644
3.561
1.667
1.655
1.647
1.643
1.601
1.590
3s
0.98
0.95
0.97
1.03
0.96
1.99
4.00
6.44
f1 (ppm)




# 8. References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