Supporting Information

Palladium-Catalyzed Dehydration of Amides to Nitriles

Wandi Zhang,[†] Christopher W. Haskins,[†] Yang Yang, and Mingji Dai[*]

Department of Chemistry and Center for Cancer Research, Purdue University, 560 Oval Drive, West Lafayette, IN 47907, United States

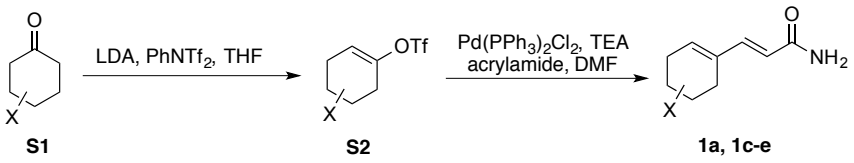
CONTENTS

Part 1. Experimental Procedure and Analytical Data

General Methods: NMR spectra were recorded on (^{1}H at 400 MHz, 500 MHz and ^{13}C at 100 MHz, 125 MHz) spectrometers. Chemical shifts (δ) were given in ppm with reference to solvent signals [^{1}H NMR: $CHCl_3$ (7.26); ^{13}C NMR: $CDCl_3$ (77.2)]. Column chromatography was performed on silica gel. All reactions sensitive to air or moisture were carried out under argon or nitrogen atmosphere in dry and freshly distilled solvents under anhydrous conditions, unless otherwise noted. Anhydrous MeCN was distilled over calcium hydride under N_2. All other solvents and reagents were used as obtained from commercial sources without further purification.

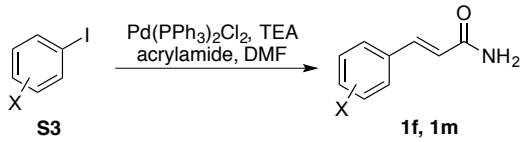
General Procedure for the Synthesis of Amides

I. General Procedure for the Synthesis of Amides **1a, 1c-e**.



To a stirred solution of diisopropyl amine (1.1 equiv) in THF cooled to 0 °C was added *n*-butyllithium (1.1 equiv). The mixture was stirred at -78 °C for 30 min before ketone **S1** (1.0 equiv) was added dropwise as a solution of THF to the LDA solution. The mixture was stirred at -78 °C for 1 hour. *N*-Phenyl-bis(trifluoromethanesulfonimide) (1.05 equiv) was added at -78 °C. The mixture was warmed to 0 °C and stirred for 2 h. The reaction was quenched with H_2O and extracted with Et_2O. The organic layers were washed with 1M aqueous NaOH, dried over $MgSO_4$, filtered and concentrated. The crude product was concentrated and purified by flash chromatography to yield the corresponding triflate **S2**. To a stirring solution of $Pd(PPh_3)_2Cl_2$ (0.1 equiv) in DMF, a solution of vinyl triflate **S2** (1.0 equiv), triethylamine (3.4 equiv), and acrylamide (1.8 equiv) in DMF was added dropwise. The mixture was heated to 75 °C for 5 h. The mixture was diluted with H_2O and extracted using Et_2O and CH_2Cl_2. The organic layers were washed with brine and dried over Na_2SO_4. The crude product was concentrated and recrystallized using Hex:EtOAc (4:1) to yield amide **1a, 1c-e**.

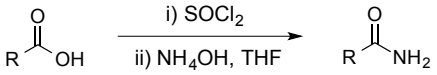
II. General Procedure for the Synthesis of Amides **1f** and **1m**.



To a stirring solution of $Pd(PPh_3)_2Cl_2$ (0.1 equiv) in DMF, a solution of the corresponding iodide **S3** (1.0 equiv), triethylamine (3.4 equiv), and acrylamide (1.8 equiv) in DMF was added dropwise.

The mixture was heated to 75 °C for 5 h. The mixture was diluted with H_2O and extracted with Et_2O and CH_2Cl_2. The organic layers were washed with brine and dried over Na_2SO_4. The crude product was concentrated and recrystallized using Hex:EtOAc (4:1) to yield amides **1f** and **1m**.

VI. General Procedure for the Synthesis of Other Amides



S4 **1g-i, 4a-j**

To corresponding carboxylic acid **S4** (3.0 mmol), an excess of $SOCl_2$ (5 mL) was added to the reaction flask. The solution was then refluxed for several hours (1-5 h) or until solution was no longer fuming. The excess $SOCl_2$ was removed under vacuum. The crude product was dissolved in 5 mL of THF and cooled to 0 °C. An excess amount of NH_4OH (28-30% NH_3, 15 mL) was added to the solution slowly. The solution was allowed to stir for 0.5 h after addition at 0 °C. The solution was extracted using (1x10 mL) THF and (3x10 mL) EtOAc. The product was dried over anhydrous Na_2SO_4 or $MgSO_4$, filtered and concentrated to give the desired amides **1g-i**, and **4a-j**.

Compounds **1b**[1], **1j**[2], **1k**[2], and **1i**[2] were synthesized according to the literature route.

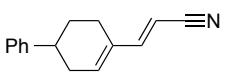
General Procedure for the Dehydration of Amides and Analytical Data

General Procedure **A** for the Dehydration of Amides

A mixture of amide (50 mg), $PdCl_2$ (0.1 equiv), AgOAc (3.0 equiv), and LiCl (2.0 equiv) in MeCN (2.5 mL) in a 2 dram vial were heated at 80 °C for 24 h or until the starting material was consumed. The palladium catalyst was filtered off through a plug of celite and washed with EtOAc. The crude solution was concentrated and purfied via column chromatography (Hex/EtOAc = 10/1).

General Procedure **B** for Dehydration of Amides

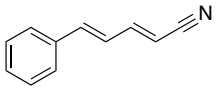
A mixture of amide (50 mg), $Pd(PPh_3)_2Cl_2$ (0.1 equiv), $Cu(OAc)_2$ (3.0 equiv) in MeCN (2.5 mL) in a 2 dram vial were heated at 80 °C for 24 hours or until the starting material was consumed. The palladium catalyst was filtered off through a plug of celite and washed with EtOAc. The crude solution was concentrated and purified via column chromatography (Hex/EtOAc = 10/1).



3a (96%)

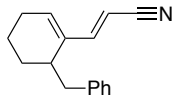
3a was prepared using general procedure **A**; 96% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.33 (t, J = 7.2 Hz, 2H), 7.26-7.22 (m, 3H), 7.06 (d, J = 16.4 Hz, 1H), 6.25 (s, 1H), 5.23 (d, J = 16.4 Hz,

1H), 2.87-2.80 (m, 1H), 2.56 (d, J = 19.6 Hz, 1H), 2.37 (dd, J = 19.6, 10.4 Hz, 1H), 2.26 (d, J = 3.2 Hz, 1H), 2.08 (dd, J = 12.6, 3.2 Hz, 1H), 1.87-1.77 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.1, 145.7 139.2, 134.5, 128.7, 126.9, 126.6, 118.9, 93.2, 39.6, 34.4, 29.1; IR (film): 2921, 2209, 1626, 1597, 1452, 1181 cm^{-1}; MS (ESI): *m/z* 232.0 $[M+Na]^+$.



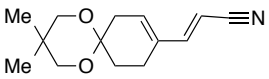
3b (69%)

3b was prepared using general procedure **A**; 69% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.46 (dd, J = 6.4, 2.0 Hz, 2H), 7.40-7.34 (m, 3H), 7.15 (dd, J = 15.6, 12.0 Hz, 1H), 6.91-6.78 (m, 2H), 5.44 (d, J = 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.4, 141.5, 135.4, 129.8, 129.0, 127.5, 125.5, 118.4, 98.4. Our spectra are consistent with literature report.[3]



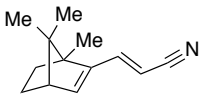
3c (82%)

3c was prepared using general procedure **A**; 82%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.34-7.30 (m, 2H), 7.26-7.24 (m, 1H), 7.18-7.16 (m, 1H), 6.96 (d, J =16.8 Hz, 1H), 6.17 (t, J = 4.0 Hz, 1H), 5.34 (d, J = 16.4 Hz, 1H), 2.87 (dd, J = 13.2, 2.8 Hz, 1H), 2.57-2.48 (m, 2H), 2.37-2.17 (m, 2H), 1.78-1.60 (m, 3H), 1.43 (tt, J = 13.6, 4.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 153.1, 140.5, 140.4, 138.4, 129.0, 128.6, 126.4, 119.0, 92.7, 38.6, 34.3, 26.6, 24.3, 16.6; IR (film): 2936, 2215, 1622, 1599, 1266, 968 cm^{-1}; MS (ESI): *m/z* 246.0 $[M+Na]^+$.



3d (49%)

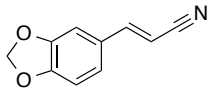
3d was prepared using general procedure **A**; 49%. ^{1}H NMR(400 MHz, $CDCl_3$): δ = 7.00 (d, J = 16.0 Hz, 1H), 5.99 (t, J = 4.4 Hz, 1H), 5.20 (d, J = 16.4 Hz, 1H), 3.57 (d, J = 11.2 Hz, 2H), 3.47 (d, J = 11.6 Hz, 2H), 2.56 (s, 2H), 2.22-2.20 (m, 2H), 2.03 (t, J = 6.4 Hz, 2H), 1.03 (s, 3H), 0.93 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 152.4, 135.6, 134.1, 118.1, 96.6, 93.8, 70.5, 35.9, 30.3, 26.9. 22.8, 22.6, 21.9; IR (film): 2959, 2217, 1633, 1266, 1039, 968 cm^{-1}; MS (ESI): *m/z* 256.1 $[M+Na]^+$.



3e (51%)

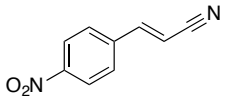
3e was prepared using general procedure **A**; 51%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.93 (dd, J =16.8, 1.2 Hz, 1H), 6.42 (d, J =3.2 Hz, 1H), 5.46 (dd, J =16.4, 1.2 Hz, 1H), 2.41 (t, J =3.6 Hz, 1H), 1.96-1.90 (m, 1H), 1.64-1.59 (m, 1H), 1.14 (s, 3H), 1.09-0.95 (m, 2H), 0.08 (s, 3H), 0.76 (s,

3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.2, 145.5, 142.8, 119.2, 94.0, 57.6, 54.2, 52.3, 31.4, 25.0, 19.5, 19.3, 12.6; IR (film): 2959, 2214, 1606, 1265, 966, 910 cm^{-1}; MS (ESI): *m/z* 210.1 $[M+Na]^+$.



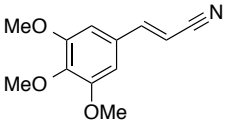
3f (59%)

3f was prepared using general procedure **A**; 59%. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.28 (d, *J* = 16.5 Hz, 1H), 6.93-6.92 (m, 2H), 6.82 (dd, *J* =7.0, 1.0 Hz, 1H), 6.02 (s, 2H), 5.67 (d, *J* = 16.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.5, 150.1, 148.7, 128.1, 124.2, 118.6, 108.8, 105.7, 101.9, 94.0. Our spectra are consistent with literature report.[4]



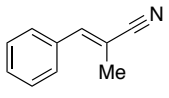
3g (80%)

3g was prepared using general procedure **A**; 80%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.28 (d, *J* = 8.8 Hz, 2H), 7.63 (dd, *J* = 6.8, 2.0 Hz, 2H), 7.47 (d, *J* = 16.8 Hz, 1H) 6.05 (d, *J* = 16.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 149.2, 147.9, 139.3, 128.3, 124.5, 117.1, 101.2. Our spectra are consistent with literature report.[5]



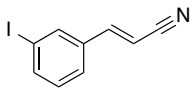
3h (87%)

3h was prepared using general procedure **A**; 87%. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.29 (d, *J* = 16.5 Hz, 1H), 6.64 (s, 2H), 5.78 (d, *J* = 16.5 Hz, 1H), 3.86 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.7, 150.5, 141.0, 129.1, 118.3, 104.8, 95.5, 61.1, 56.3. Our spectra are consistent with literature report.[6]



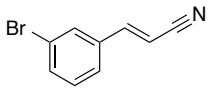
3i (78%)

3i was prepared using general procedure **A**; 78%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.45-7.37(m, 3H), 7.34 (d, *J* = 1.7 Hz, 1H), 7.32 (s, 1H), 7.22 (s, 1H), 2.15 (d, *J* = 1.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.3, 134.0, 129.2, 129.2, 128.6, 121.2, 109.6, 16.7. Our spectra are consistent with literature report.[7]



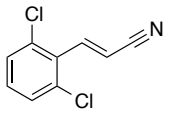
3j (59%)

3j was prepared using general procedure **A**; 59%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.74 (s, 1H), 7.70 (d, J = 7.89 Hz, 1H), 7.35 (d, J = 7.80 Hz, 1H), 7.21 (d, J = 6.10, 1H), 7.1 (t, J = 7.83, 1H), 5.8 (d, J = 16.6, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.7, 139.8, 135.9, 135.4, 130.6, 126.4, 117.5, 97.79, 94.71; IR(film): 2957, 2931, 2220, 1620, 1560, 1409 cm^{-1}; MS (ESI): *m/z* 277.9 $[M+Na]^{+}$.



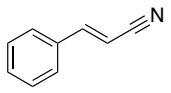
3k (60%)

3k was prepared using general procedure **A**; 60%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.60 (t, J = 1.8 Hz, 1H), 7.57 (d, J = 7.93 Hz, 1H), 7.38-7.27 (m, 3H), 5.89 (d, J = 16.7, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 149.0, 135.6, 134.1, 130.7, 130.2, 126.1, 123.4, 117.7, 98.2. Our spectra are consistent with literature report.[8]



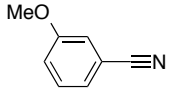
3l (56%)

3l was prepared using general procedure **A**; 56%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.55 (d, J = 17.0, 1H), 7.38 (d, J = 8.1 Hz, 2H), 7.24 (t, J = 8.4 Hz, 1H), 6.19 (d, J = 17.0, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.3, 134.9, 130.9, 130.6, 129.2, 117.3, 105.9. Our spectra are consistent with literature report.[9]



3m (91%)

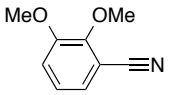
3m was prepared using general procedure **A**; 91%. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.46-7.38 (m, 6H), 5.88 (d, J = 17.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.7, 133.6, 131.3, 129.2, 127.4, 118.3, 96.4. Our spectra are consistent with literature report.[10]



5a (87%)

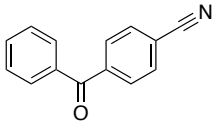
5a was prepared using general procedure **B**; 87%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36 (t, J = 8.48 Hz, 1H), 7.23(t, J = 10.57 Hz, 1H), 7.12 (br s, 2H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.5, 130.2,124.4, 119.2, 118.6, 116.7, 113.1, 55.4. Our spectra are consistent with

literature report.[11]



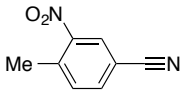
5b (83%)

5b was prepared using general procedure **B**; 83%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.14-7.04 (m, 3H), 4.00 (s, 3H), 3.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 152.6, 151.6, 124.3, 116.9, 116.2, 106.9, 61.6, 56.0. Our spectra are consistent with literature report.[12]



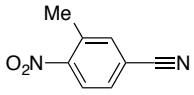
5c (53%)

5c was prepared using general procedure **B**; 53%. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.87 (d, J = 7.5 Hz, 2H), 7.80-7.78 (m, 4H), 7.64 (t, J = 7.5 Hz, 1H), 7.51 (t, J = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.2, 141.3, 136.4, 133.5, 132.3, 130.4, 130.2, 128.8, 118.1, 115.8. Our spectra are consistent with literature report.[13]



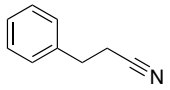
5d (77%)

5d was prepared using general procedure **B**; 77%. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.26 (d, J = 2.0 Hz, 1H), 7.78 (dd, J = 3.0, 2.0 Hz, 1H), 7.51 (d, J = 8.0 Hz, 1H), 2.68 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 149.4, 139.1, 135.8, 134.1, 128.5, 116.7, 111.5. Our spectra are consistent with literature report.[14]



5e (77%)

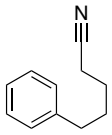
5e was prepared using general procedure **B**; 77%. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.02 (d, J = 8.5 Hz, 1H), 7.68-7.65 (m, 2H), 2.62 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 151.6, 136.6, 134.7, 130.8, 125.3, 117.0, 116.7, 20.0. Our spectra are consistent with literature report.[14]



5f (88%)

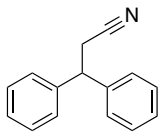
5f was prepared using general procedure **B**; 88%. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.36 (t, J = 6.85 Hz, 2H), 7.29 (m, 1H), 7.26 (d, J = 6.91 Hz, 2H), 2.97 (t, J = 7.09 Hz, 2H), 2.62 (t, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 138.0, 128.8, 128.2, 127.1, 119.1, 31.5, 19.2. Our spectra are

consistent with literature report.[15]



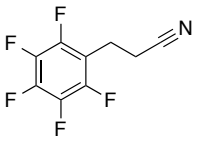
5g (95%)

5g was prepared using general procedure **B**; 95%. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.30 (t, J = 7.17 Hz, 2H), 7.19 (m, 3H), 2.67 (t, J = 7.23 Hz, 2H), 2.35 (t, J = 7.09 Hz, 2H), 2.27 (s, 3H), 1.80 (m, 2H), 1.71 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 141.1, 128.4, 128.3, 126.0, 119.5, 34.9, 30.2, 24.7, 17.0. Our spectra are consistent with literature report.[15]



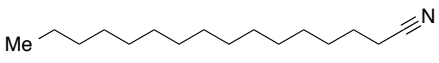
5h (93%)

5h was prepared using general procedure **B**; 93%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38-7.34 (m, 4H), 7.31-7.25 (m, 6H), 4.40 (t, J = 9.5 Hz, 1H), 3.04 (d, J = 9.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 141.3, 129.0, 127.6, 127.5, 47.2, 24.3. Our spectra are consistent with literature report.[16]



5i (93%)

5i was prepared using general procedure **B**; 93%. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.09 (t, J = 7.28 Hz, 2H), 2.68 (t, J = 7.32 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.7, 144.2, 142.3, 139.8,139.2, 136.7, 117.9, 111.2, 109.0, 19.1, 17.4; ^{19}F NMR (376 MHz, $CDCl_3$): -144.68, -155.95, -162.75; IR (film): 2252, 1522, 1502, 1040, 941, 876 cm^{-1}; MS (ESI): *m/z* 244.0 $[M+Na]^{+}$.



5j (78%)

5j was prepared using general procedure **B**; 78%. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.33 (t, J = 7.0 Hz, 2H), 1.68-1.62 (m, 2H), 1.47-1.41 (m, 2H), 1.31-1.25 (m, 22H), 0.88 (t, J = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 120.0, 32.0, 29.8, 29.8, 29.7, 29.6, 29.5, 29.4, 28.9, 28.8, 25.5, 22.8, 17.2, 14.2. Our spectra are consistent with literature report.[17]

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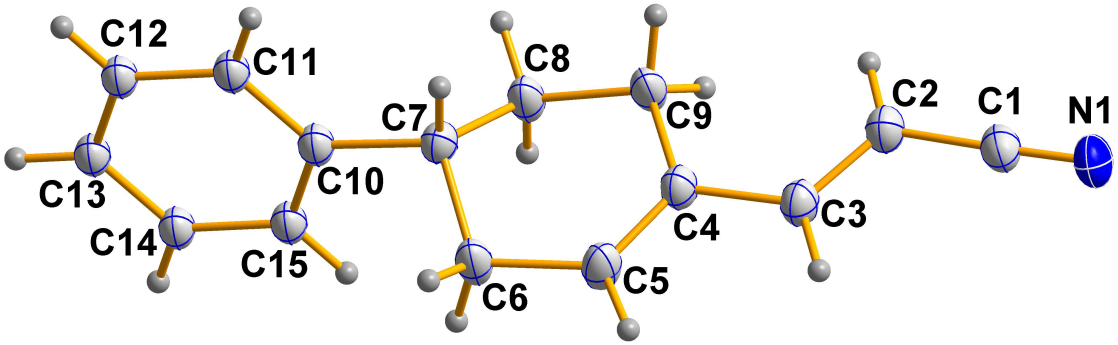
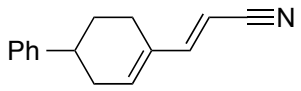
Part 2. X–Ray Structure of 6a





3a

Figure S1. X–Ray Structure of **3a**

Part 3. NMR Spectra of New Compounds

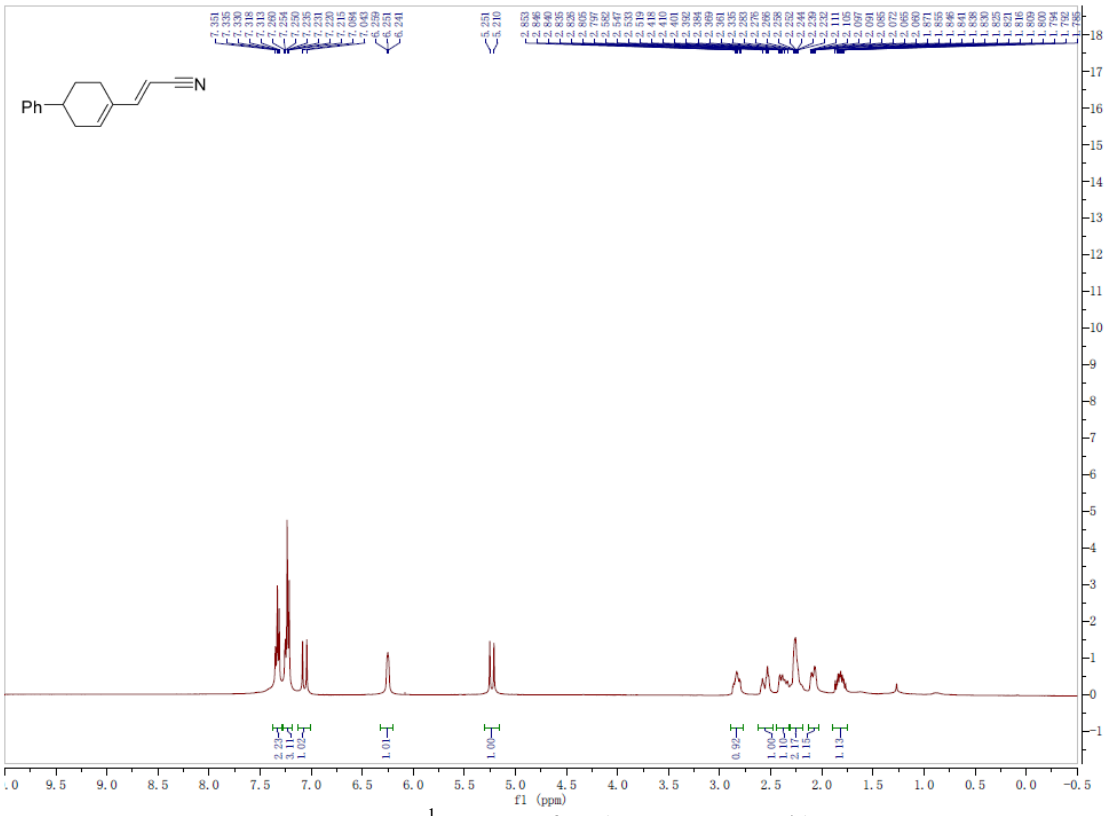


Figure S2. ^{1}H NMR of **3a** (400 MHz, $CDCl_3$)

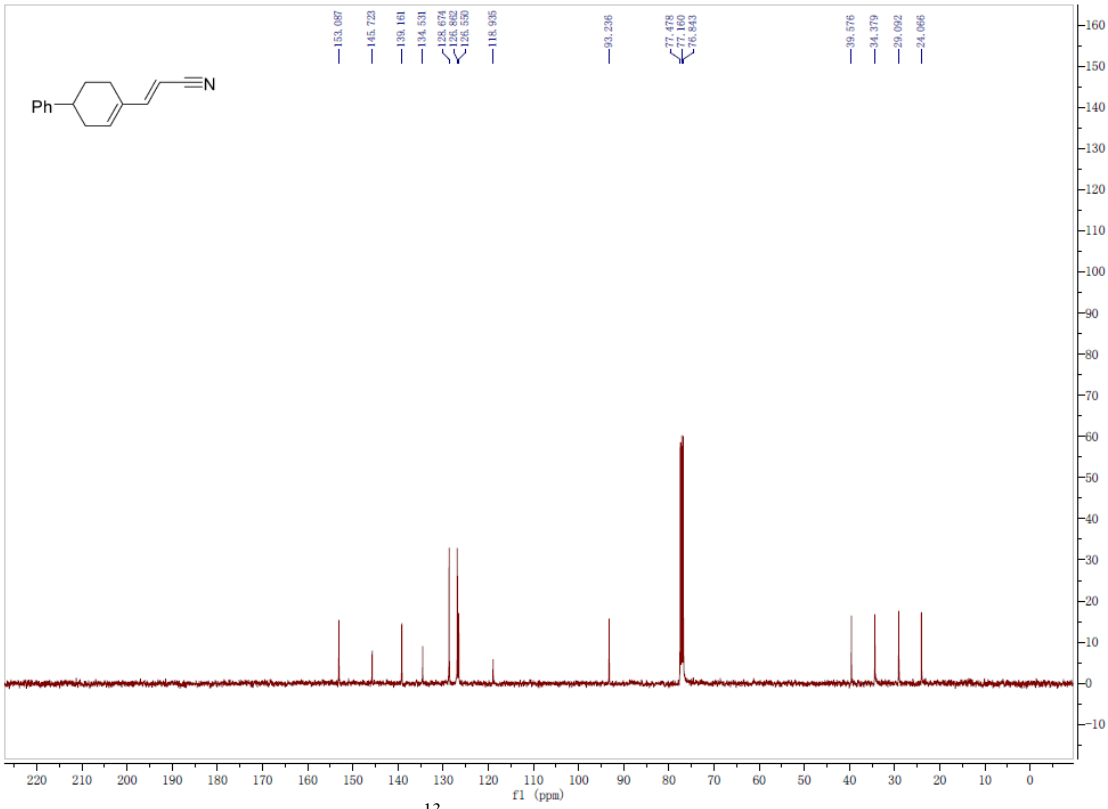


Figure S3. ^{13}C NMR of **3a** (100 MHz, $CDCl_3$)

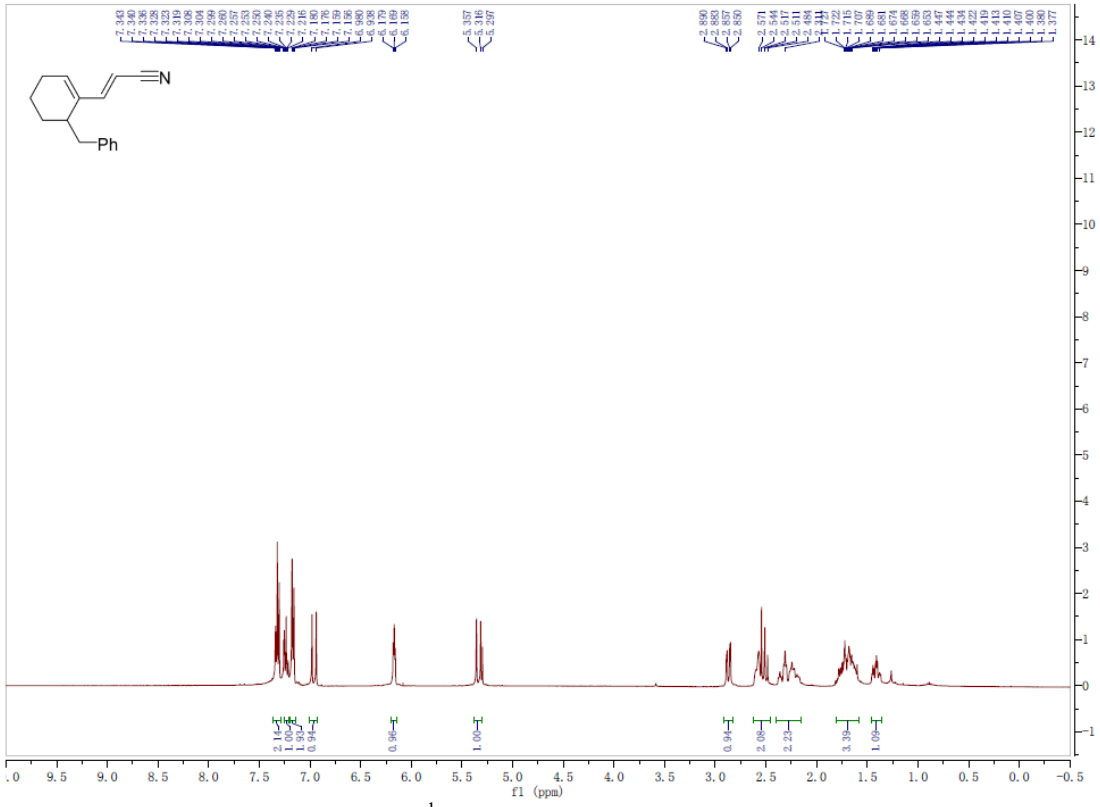


Figure S4. ^{1}H NMR of **3c** (400 MHz, $CDCl_3$)

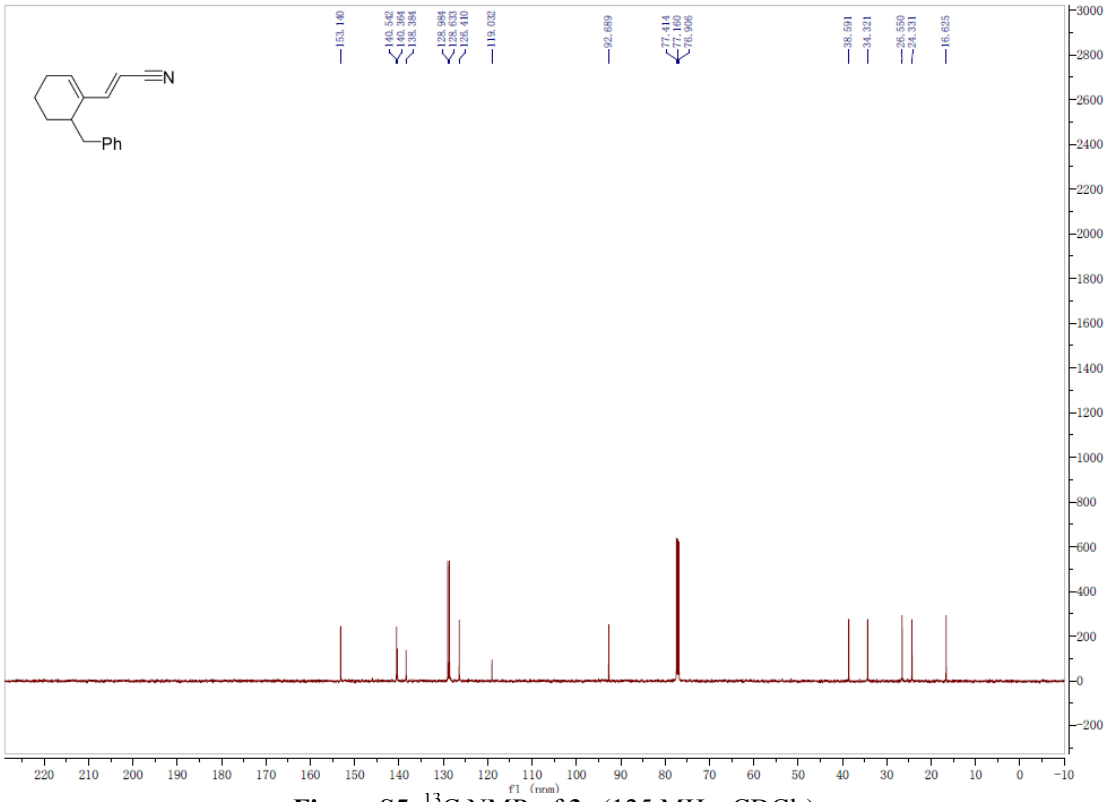


Figure S5. ^{13}C NMR of **3c** (125 MHz, $CDCl_3$)

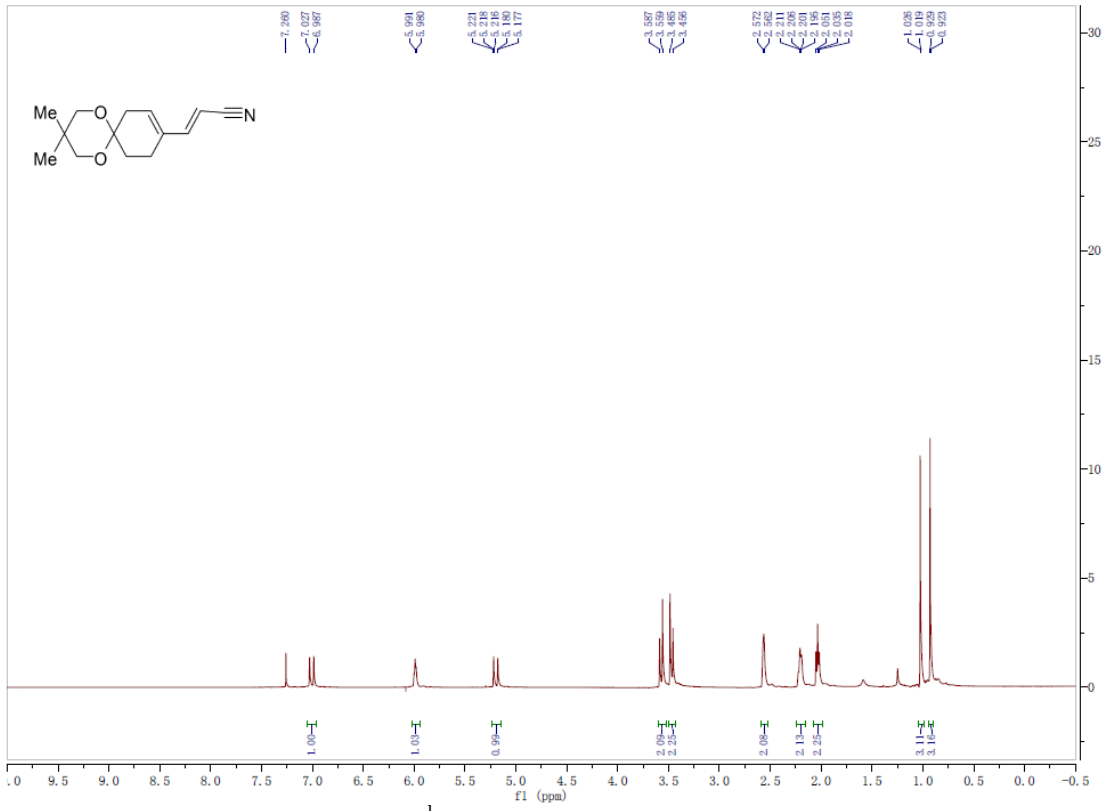


Figure S6. ^{1}H NMR of **3d** (400 MHz, $CDCl_3$)

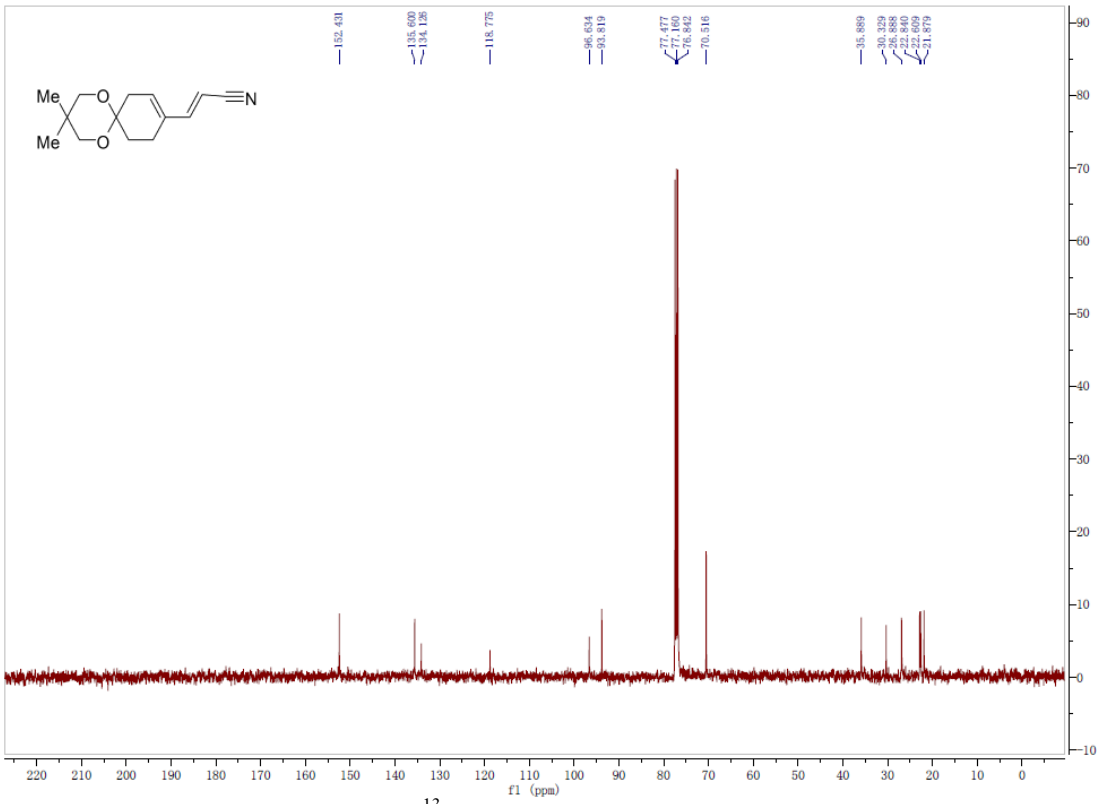


Figure S7. ^{13}C NMR of **3d** (100 MHz, $CDCl_3$)

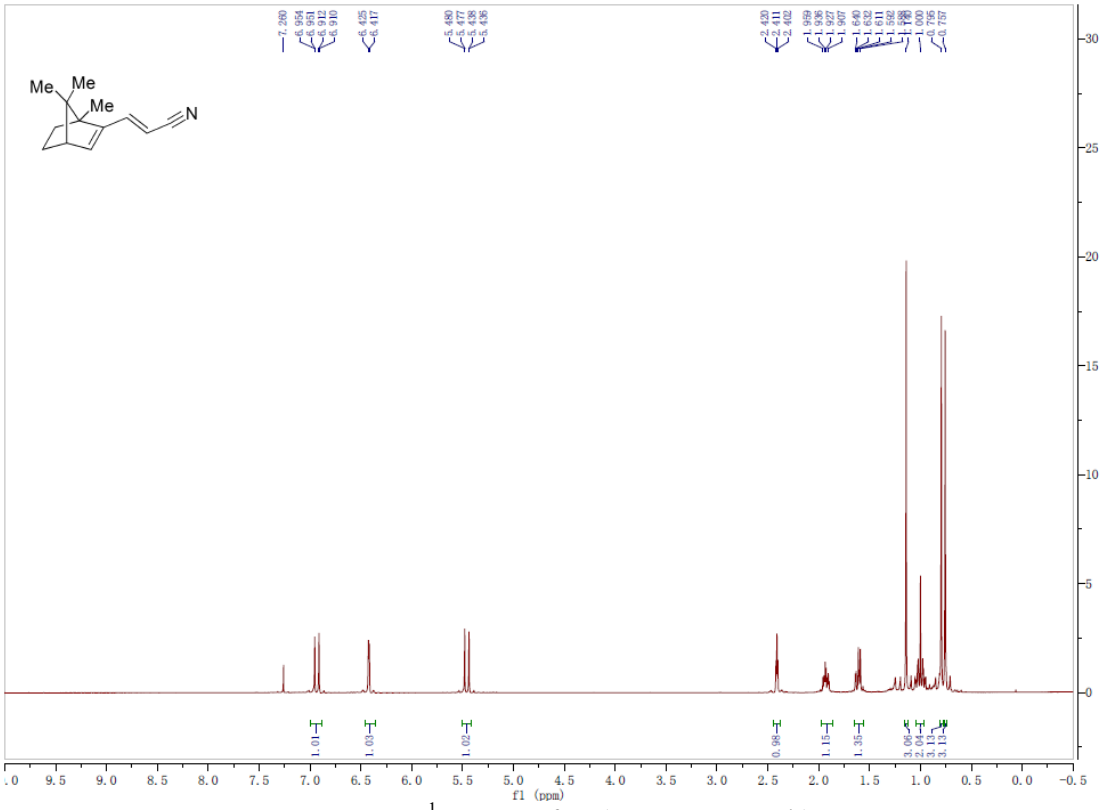


Figure S8. ^{1}H NMR of **3e** (400 MHz, $CDCl_3$)

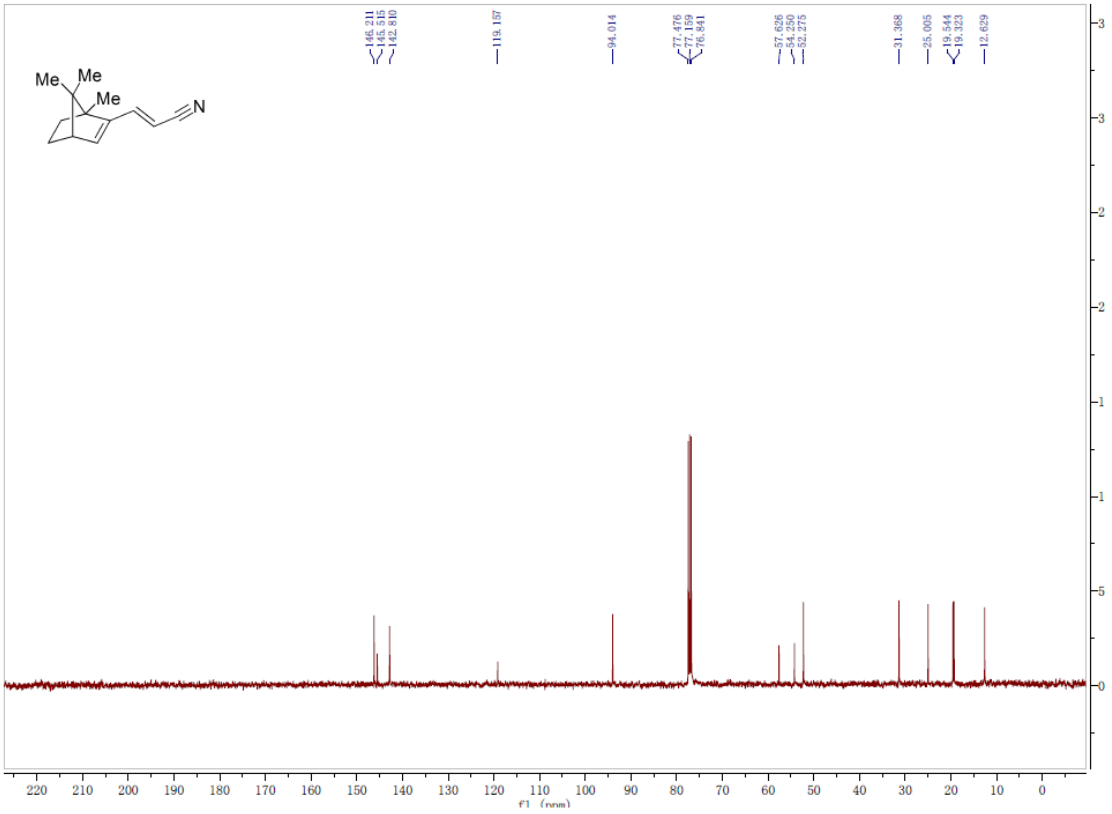


Figure S9. ^{13}C NMR of **3e** (100 MHz, $CDCl_3$)

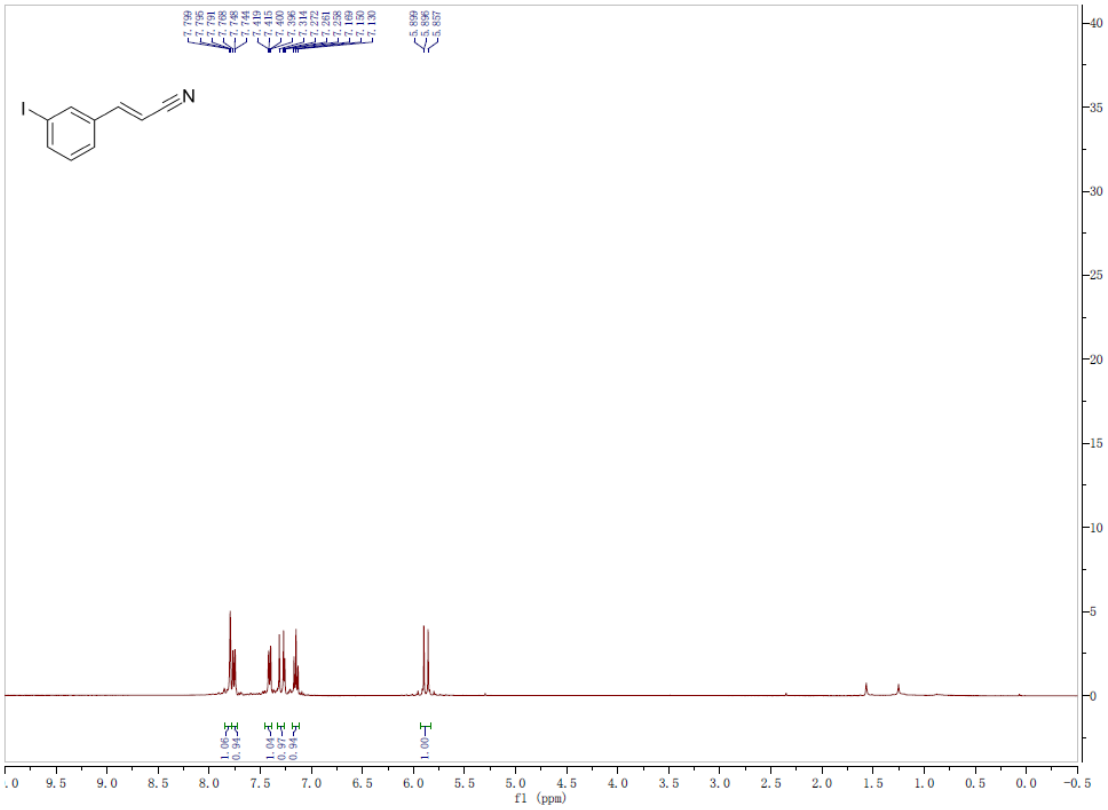


Figure S10. ^{1}H NMR of **3j** (400 MHz, $CDCl_3$)

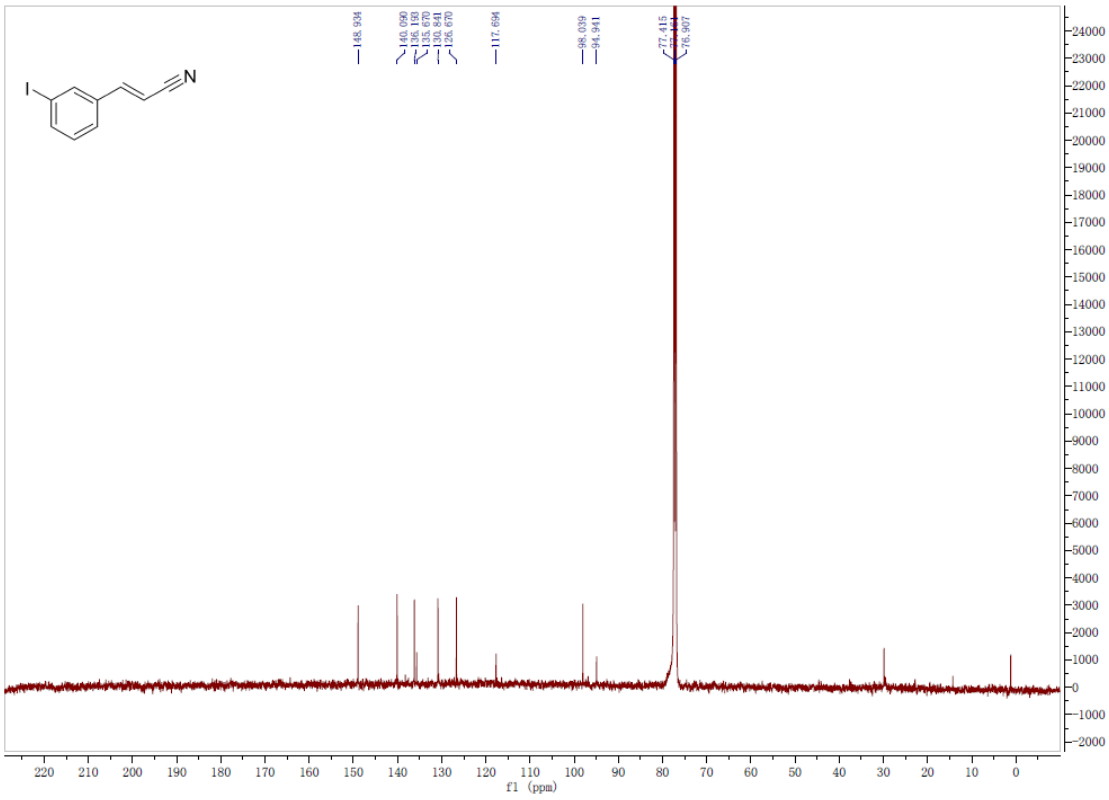


Figure S11. ^{13}C NMR of **3j** (125 MHz, $CDCl_3$)

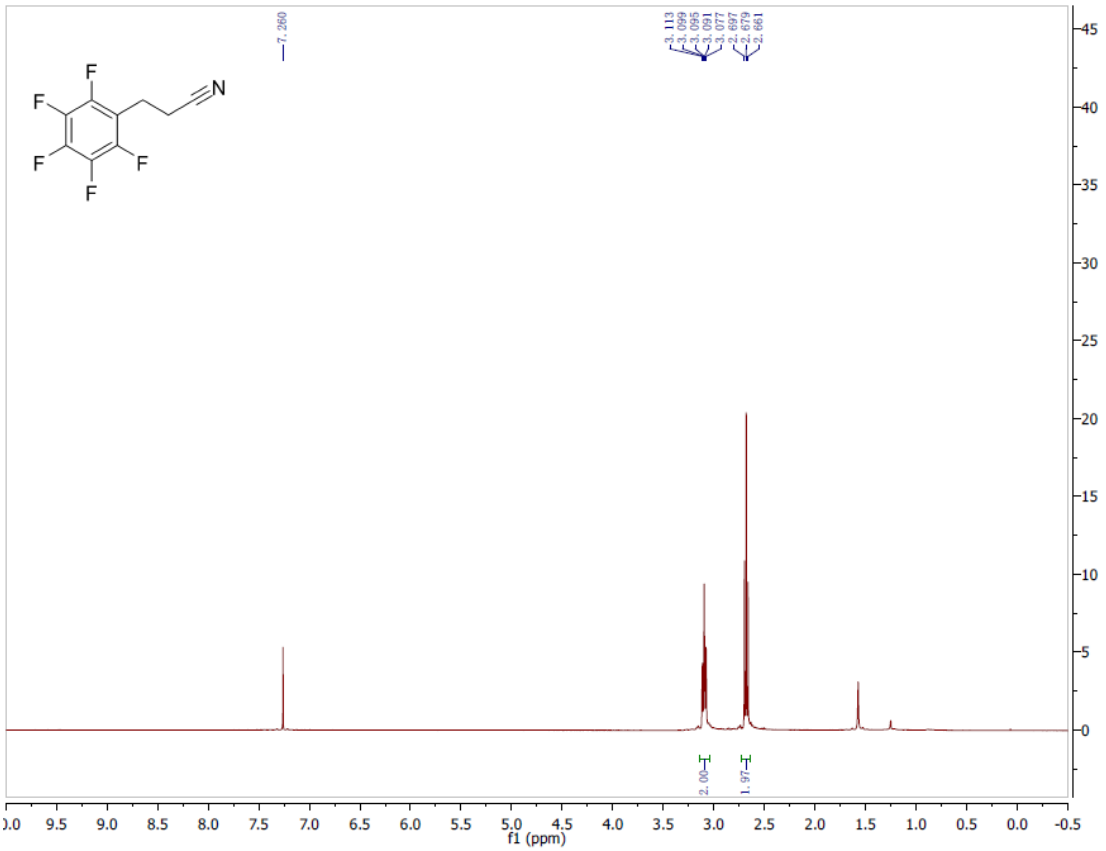


Figure S12. ^{1}H NMR of **5i** (400 MHz, $CDCl_3$)

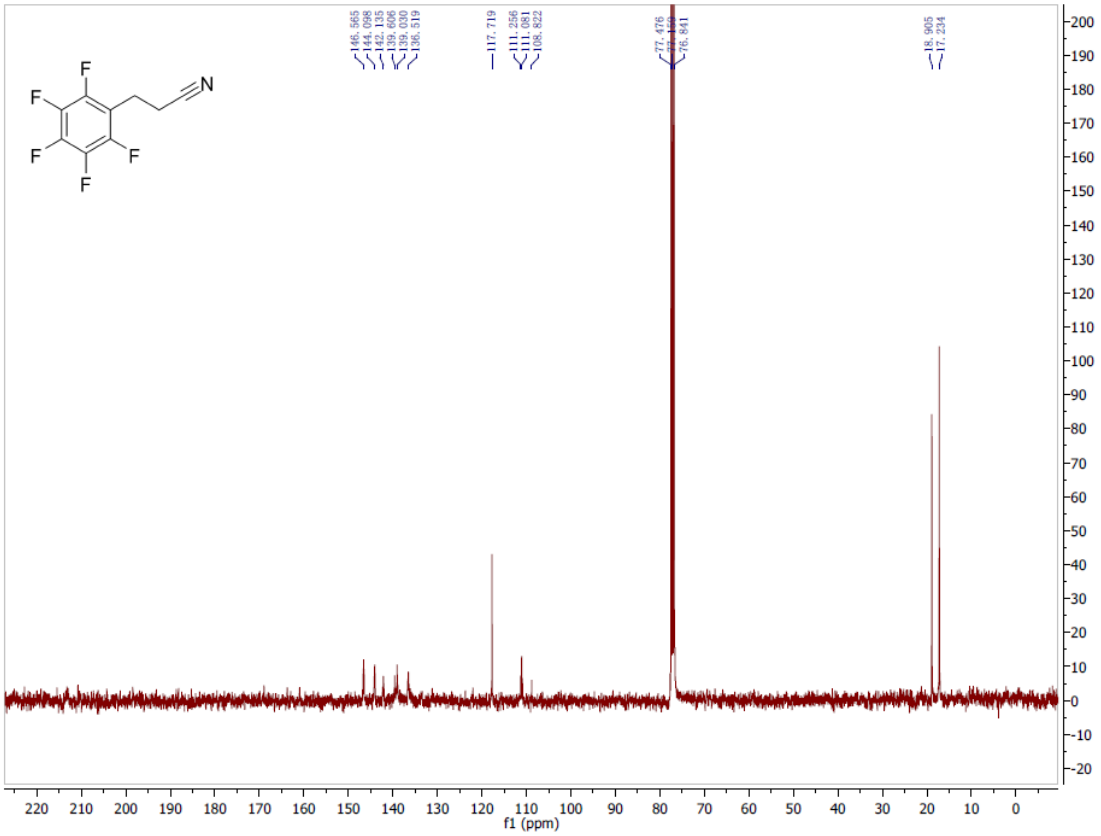


Figure S13. ^{13}C NMR of **5i** (100 MHz, $CDCl_3$)