Supporting Information

Nickel-Catalysed Novel β,γ-Unsaturated Nitriles Synthesis

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General information

All reactions were isolated from moisture and oxygen by a nitrogen atmosphere with a balloon fitted on a Schlenk tube. All glassware was oven dried at 110 ^{o}C for hours and cooled down under vacuum. Toluene and mesitylene was purified by distillation with sodium. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. $Ni(PPh_3)_4$[1] was prepared following literature procedures. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp. 60-90 ^{o}C). Gas chromatographic analyses were performed on Varian GC 2000 gas chromatography instrument with a FID detector and biphenyl was added as internal standard. GC-MS spectra were recorded on a Varian GC-MS 3900-2100T. ^{1}H and ^{13}C NMR data were recorded with Varian Mercury (300 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) 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. EPR spectra were recorded on a Bruker A200 spectrometer.

General procedure for the Nickel-catalysed Heck-type alkenylation of α-bromide nitriles

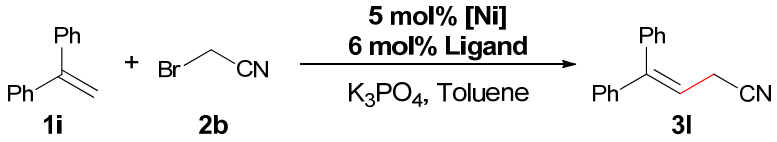
Method A: In a glove box, $Ni(PPh_3)_4$ (27.7 mg, 0.025 mmol), dppp (12.4 mg, 0.030 mmol) and K_3PO_4 (212.3 mg, 1.0 mmol) were added in a Shlenck tube. The tube was then sealed with septa and taken out of the glove box. Mesitylene (2 mL) was injected in the tube via a syringe. The reaction was then heated up to 160 ^{o}C with stirring. Then, alkyl bromide (0.75 mmol) and alkene (0.50 mmol) were consequently injected in the reaction tube. After stirring for 3 hours, it was quenched by diluted HCl solution and

extracted with ethyl ether (3 x 10 mL). The organic layers were combined and the pure product was obtained by flash column chromatography on silica gel (petroleum: ethyl acetate = 50:1).

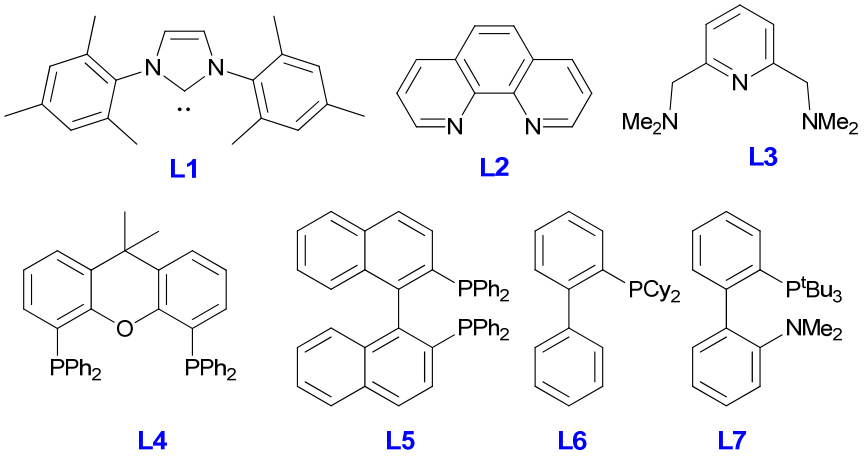
Method B: In a glove box, $Ni(PPh_3)_4$ (27.7 mg, 0.025 mmol), dppp (12.4 mg, 0.030 mmol) and K_3PO_4 (212.3 mg, 1.0 mmol) were added in a Shlenck tube. The tube was then sealed with septa and taken out of the glove box. Toluene (2 mL) was injected in the tube via a syringe. After stirring for 5 min, alkyl bromide (0.75 mmol) and alkene (0.50 mmol) were consequently injected in the reaction tube. The reaction was then heated up to 100 ^oC and kept stirring for 10 hours. After the completion of the reaction, it was quenched by diluted HCl solution and extracted with ethyl ether (3 x 10 mL). The organic layers were combined and the pure product was obtained by flash column chromatography on silica gel.

Method C: In a glove box, $Ni(acac)_2$ (6.4 mg, 0.025 mmol), dppf (16.6 mg, 0.030 mmol) and K_3PO_4 (212.3 mg, 1.0 mmol) were added in a Shlenck tube. The tube was then sealed with septa and taken out of the glove box. Toluene (2 mL) was injected in the tube via a syringe. After stirring for 5 min, DIBAL-H (40 μL, 1.5 M, 0.060 mmol) was added dropwise under ice bath. Then alkyl bromide (0.75 mmol) and alkene (0.50 mmol) were consequently injected in the reaction tube. The reaction was then heated up to 100 ^oC and kept stirring for 10 hours. After the completion of the reaction, it was quenched by diluted HCl solution and extracted with ethyl ether (3 x 10 mL). The organic layers were combined and the pure product was obtained by flash column chromatography on silica gel.

Ligand optimization for primary α-cyano alkyl bromides by using $Ni(acac)_2$ as the catalyst precursor [a]



Entry	Catalyst precursor	Ligand	Yield(%)[b]
1	$Ni(PPh_3)_4$	dppp	28
2	$Ni(acac)_2$ / DIBAL-H	dppp	57
3	**$Ni(acac)_2$ / DIBAL-H**	**dppf**	**69**
4	$Ni(acac)_2$ / DIBAL-H	L1	13
5	$Ni(acac)_2$ / DIBAL-H	L2	*n.d.*
6	$Ni(acac)_2$ / DIBAL-H	L3	2
7	$Ni(acac)_2$ / DIBAL-H	L4	46
8	$Ni(acac)_2$ / DIBAL-H	L5	10
9[c]	$Ni(acac)_2$ / DIBAL-H	L6	14
10[c]	$Ni(acac)_2$ / DIBAL-H	L7	33



[a]The reactions were carried out with **1i** (0.50 mmol), **2b** (0.75 mmol), [Ni] (5 mol%), Ligand (6 mol%), K_3PO_4 (1.0 mmol), toluene (2 mL), 100 °C, 12 h. [b]The yield was determined by GC analysis with biphenyl as the internal standard. *n.d.* = no desired product. [c]Ligand (10 mol%)

EPR experiments

Method for the EPR detection of different nickel species: In a glove box, nickel catalyst (0.05 mmol) was added to a texting tube. The tube was then sealed with septa and taken out of the glove box. Toluene (0.5 mL) was injected in the tube via a syringe. EPR detection was conducted at 150 K on a Bruker A-200 spectrometer operating at 9.435 GHz.

Method for detecting the reaction between $Ni(PPh_3)_4$ and α-cyano alkyl bromides: In a glove box, $Ni(PPh_3)_4$ (27.7 mg, 0.025 mmol), dppp (12.4 mg, 0.030 mmol) was added to a texting tube. The tube was then sealed with septa and taken out of the glove box. Toluene (0.5 mL) and 2-bromopentanenitrile (16.2 mg, 0.10 mmol) was consequently injected in the tube. Immediately, EPR detection was conducted at 170 K on a Bruker A-200 spectrometer operating at 9.435 GHz.

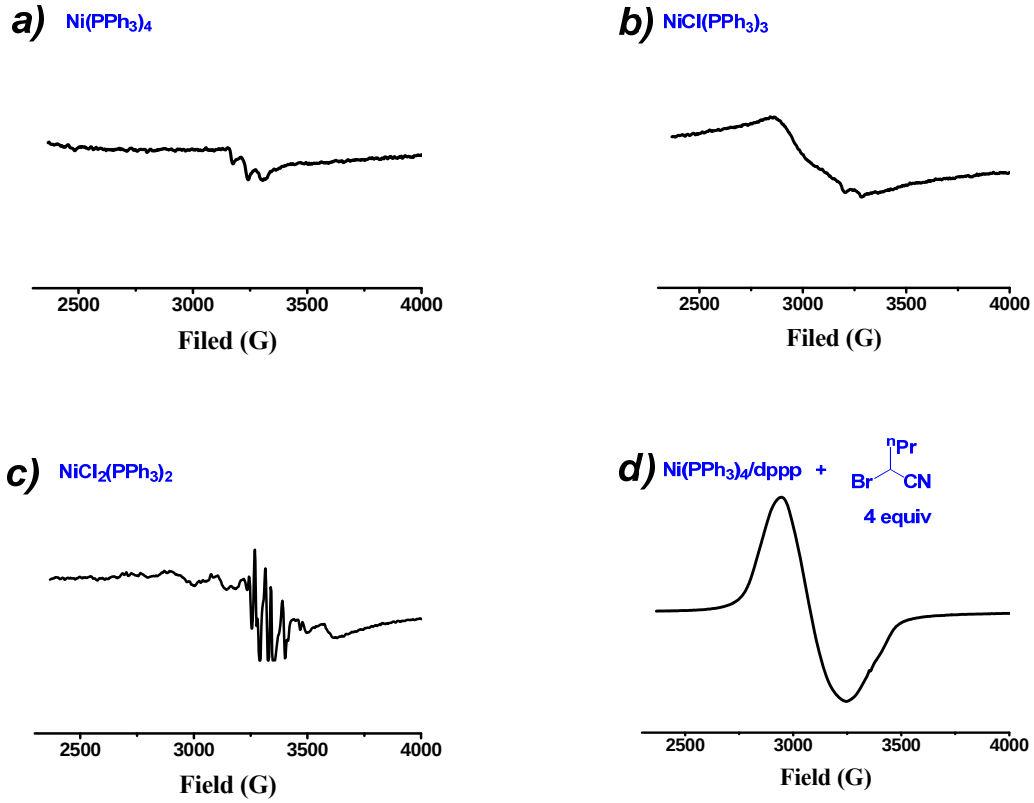
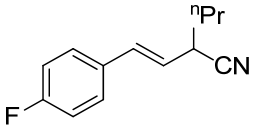


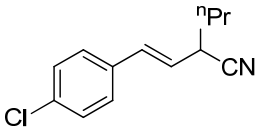
Figure S1. Experimental spectra of nickel species

EPR experiments shown in Figure S1 indicated that Ni^0, Ni^I and Ni^{II} complexes have their characteristic peaks that differ from each other (Figures S1, a, b and c). When the mixture of $Ni(PPh_3)_4$/dppp and excess amount of 2-bromopentanenitrile were taken to do EPR detection immediately, the resulting spectrum obviously showed a rather strong signal of Ni^I species (Figures S1, d). This result clearly proved that Ni(0) initiated the reaction via a single electron transfer process.

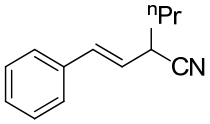
Detail descriptions for products



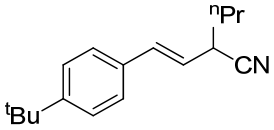
(*E*)-2-(4-Fluorostyryl)pentanenitrile (**3a**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.25 (m, 2H), 7.20-6.85 (m, 2H), 6.68 (d, *J* = 15.8 Hz,1H), 5.96 (dd, *J* = 15.7, 6.0 Hz, 1H), 3.55-3.30 (m, 1H), 1.85-1.70 (m, 2H), 1.65-1.50 (m, 2H), 0.99 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.4, 161.1, 132.1, 129.9, 128.4, 128.3, 123.2, 120.3, 116.0, 115.7, 35.4, 34.3, 20.3, 13.7. HRMS (EI) calcd for $C_{13}H_{14}FN$ $[M]^+$: 203.1111; found: 203.1110



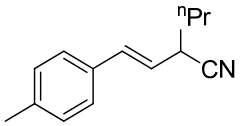
(*E*)-2-(4-Chlorostyryl)pentanenitrile (**3b**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.25 (m, 4H), 6.66 (d, *J* = 15.9, 1H), 6.01 (dd, *J* = 15.8, 6.4 Hz, 1H), 3.50-3.35 (m, 1H), 1.80-1.69 (m, 2H), 1.61-1.46 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 134.1, 133.7, 131.7, 128.7, 127.6, 123.8, 119.9, 35.0, 34.0, 20.0, 13.4. HRMS (ESI) calcd for $C_{13}H_{14}ClN$ $[M+H]^+$: 219.0893; found: 219.0891.



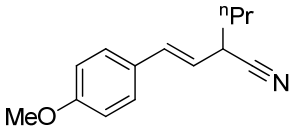
(*E*)-2-Styrylpentanenitrile (**3c**)[1]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28-7.10 (m, 5H), 6.63 (d, *J* = 15.3, 1H), 5.94 (dd, *J* = 15.6, 6.6 Hz, 1H), 3.40-3.25 (m, 1H), 1.72-1.58 (m, 2H), 1.56-1.46 (m, 2H), 0.89 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.0, 134.3, 131.7, 129.5, 127.8, 124.5, 121.5, 36.5, 35.4, 21.3, 14.8.



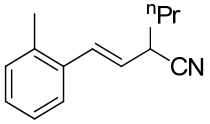
(*E*)-2-(4-(*tert*-Butyl)styryl)pentanenitrile (**3d**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.25 (m, 4H), 6.68 (d, *J* = 15.8 Hz, 1H), 5.98 (dd, *J* = 16.6, 7.2 Hz, 1H), 3.55-3.27 (m, 1H), 1.77-1.63 (m, 2H), 1.62-1.45 (m, 2H), 1.31 (s, 9H), 0.96 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.6, 133.3, 133.1, 126.5, 125.9, 122.7, 120.6, 35.6, 34.9, 34.4, 31.5, 20.3, 13.8. HRMS (ESI) calcd for $C_{17}H_{23}N$ $[M+H]^+$: 242.1909; found: 242.1906.



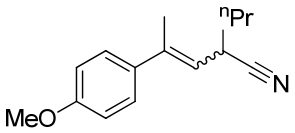
(*E*)-2-(4-Methylstyryl)pentanenitrile (**3e**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 6.67 (d, *J* = 15.8 Hz, 1H), 5.97 (dd, *J* = 15.8, 6.5 Hz, 1H), 3.39 (q, *J* = 6.5 Hz, 1H), 2.33 (s, 3H), 1.78-1.68 (m, 2H), 1.61-1.45 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.3, 133.2, 129.6, 128.5, 126.6, 122.4, 120.5, 35.5, 34.4, 21.4, 20.3, 13.7. HRMS (ESI) calcd for $C_{14}H_{17}N$ $[M+H]^+$: 200.1439; found: 200.1436.



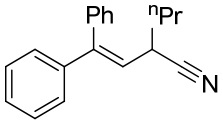
(*E*)-2-(4-Methoxystyryl)pentanenitrile (**3f**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 6.63 (d, *J* = 15.8 Hz, 1H), 5.88 (dd, *J* = 15.8, 6.5 Hz, 1H), 3.79 (s, 3H), 3.38 (q, *J* = 6.5 Hz, 1H), 1.82-1.66 (m, 2H), 1.61-1.43 (m, 2H), 0.97 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.5, 132.4, 128.4, 127.6, 120.8, 120.3, 113.9, 55.2, 35.3, 34.0, 20.0, 13.4. HRMS (ESI) calcd for $C_{14}H_{17}NO$ $[M+H]^+$: 216.1388; found: 216.1388.



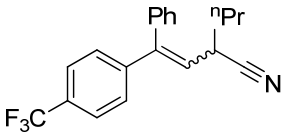
(*E*)-2-(2-Methylstyryl)pentanenitrile (**3g**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.31 (m, 1H), 7.27 - 7.03 (m, 3H), 6.92 (d, *J* = 15.7 Hz, 1H), 5.90 (dd, *J* = 15.7, 6.4 Hz, 1H), 3.50-3.30 (m, 1H), 2.35 (s, 3H), 1.67-1.82 (m, 2H), 1.45-1.64 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.8, 135.1, 131.3, 130.5, 128.2, 126.3, 125.9, 124.8, 120.4, 35.4, 34.5, 20.2, 19.9, 13.6. HRMS (ESI) calcd for $C_{14}H_{17}N$ $[M+H]^+$: 200.1439; found: 200.1435.



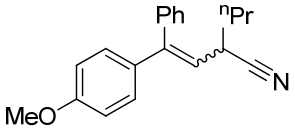
(*Z, E*)-4-Methoxyphenyl)-2-propylpent-3-enenitrile (**3h**): ^{1}H NMR (300 MHz, $CDCl_3$) (one isomer presented) δ 7.24 (d, *J* = 7.6 Hz, 2H), 6.79 (d, *J* = 7.5 Hz, 2H), 5.48 (d, *J* = 8.3 Hz, 1H), 3.73 (s, 3H), 3.50-3.30 (m, 1H), 1.99 (s, 3H), 1.77-1.39 (m, 4H), 0.98-0.80 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.2, 139.1, 134.4, 126.9, 121.0, 120.0, 113.6, 55.2, 35.3, 30.0, 20.1, 16.4, 13.5. HRMS (ESI) calcd for $C_{15}H_{19}NO$ $[M+H]^+$: 230.1545; found: 230.1545.



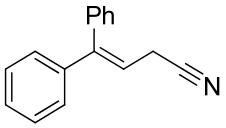
2-(2,2-Diphenylvinyl)pentanenitrile (**3i**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48-7.09 (m, 10H), 5.94 (d, *J* = 10.0 Hz, 1H), 3.29 (dt, *J* = 9.9, 7.4 Hz, 1H), 1.82-1.54 (m, 2H), 1.49-1.32 (m, 2H), 0.84 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.1, 140.7, 138.3, 129.3, 128.7, 128.3, 128.1, 128.0, 127.4, 122.3, 120.9, 35.5, 31.1, 20.0, 13.4. HRMS (ESI) calcd for $C_{19}H_{19}N$ $[M+H]^+$: 262.1596; found: 262.1593.



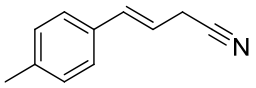
(*Z, E*)-2-(2-Phenyl-2-(4-(trifluoromethyl)phenyl)vinyl)pentanenitrile (**3j**): ^{1}H NMR (300 MHz, CDCl3) δ 7.60 (d, *J* = 7.9 Hz, 1.3H), 7.44 (d, *J* = 8.2 Hz, 0.8H), 7.38 - 7.29 (d, *J* = 7.7 Hz, 1H), 7.29 - 7.16 (m, 4H), 7.16 - 7.01 (m, 2H), 5.96 5.89 (m, 1H), 3.24 (dt, *J* = 10.0, 7.6 Hz, 0.4H), 3.12 (dt, *J* = 10.0, 7.4 Hz, 0.7H), 1.77 - 1.47 (m, 2H), 1.43 - 1.28 (m, 2H), 0.85 – 0.72 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.0, 144.9, 144.1, 142.1, 139.8, 137.4, 130.4, 130.0, 129.7, 129.2, 128.9, 128.4, 128.3, 127.6, 127.3, 125.7, 125.7, 125.2, 125.2, 124.3, 123.2, 120.5, 120.4, 35.4, 35.3, 31.0, 20.0, 13.3. HRMS (ESI) calcd for $C_{20}H_{19}F_3N$ $[M+H]^+$: 330.1470; found: 330.1471.



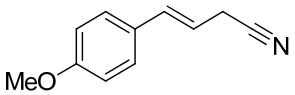
(*Z, E*)-2-(2-(4-Methoxyphenyl)-2-phenylvinyl)pentanenitrile (**3k**): ^{1}H NMR (300 MHz, CDCl3) δ 7.46 - 7.34 (m, 2H), 7.33 - 7.06 (m, 5H), 6.94 (d, *J* = 8.5 Hz, 0.7H), 6.81 (d, *J* = 8.6 Hz, 1.3H), 5.96 - 5.80 (m, 1H), 3.82 (s, 1.1H), 3.76 (s, 1.8H), 3.43 - 3.31 (m, 0.4H), 3.31 - 3.20 (m, 0.6H), 1.83 - 1.55 (m, 2H), 1.53 - 1.34 (m, 2H), 0.91 - 0.81 (q, J = 7.0 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.4, 159.1, 145.7, 145.4, 141.0, 138.4, 133.0, 130.4, 129.1, 128.5, 128.4, 128.1, 127.9, 127.7, 127.3, 121.9, 120.9, 120.3, 113.9, 113.5, 55.0, 35.4, 30.9, 19.9, 13.2. HRMS (ESI) calcd for $C_{20}H_{21}NO$ $[M+H]^+$: 292.1701; found: 292.1697.



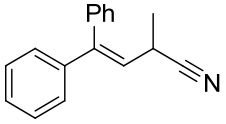
4,4-Diphenylbut-3-enenitrile (**3l**)[2]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44-7.34 (m, 3H), 7.30-7.13 (m, 7H), 6.02 (t, *J* = 7.3 Hz, 1H), 3.12 (d, *J* = 7.3 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.4, 140.6, 137.9, 129.3, 128.7, 128.3, 128.1, 128.0, 127.4, 118.1, 115.4, 18.3.



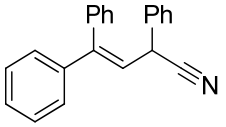
(*E*)-4-(p-tolyl)but-3-enenitrile (**3m**)[3]**:** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 7.9 Hz, 2H), 6.68 (d, *J* = 15.8 Hz, 1H), 5.97 (dt, *J* = 15.8, 5.7 Hz, 1H), 3.24 (dd, *J* = 5.5, 1.3 Hz, 2H), 2.33 (s, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 138.1, 134.3, 132.8, 129.3, 126.2, 117.4, 115.5, 21.1, 20.6.



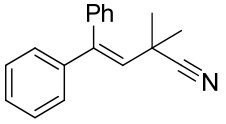
(*E*)-4-(4-Methoxyphenyl)but-3-enenitrile (**3n**)[3]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.4 Hz, 2H), 6.86 (d, *J* = 8.3 Hz, 2H), 6.65 (d, *J* = 15.7 Hz, 1H), 5.89 (dt, *J* = 15.7, 5.7 Hz, 1H), 3.80 (s, 3H), 3.24 (d, *J* = 5.6 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.6, 134.0, 128.4, 127.6, 117.5, 114.3, 114.0, 55.2, 20.7.



2-Methyl-4,4-diphenylbut-3-enenitrile (**3o**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.05 (m, 10H), 5.96 (d, *J* = 9.9 Hz, 1H), 3.39 (dq, *J* = 9.7, 7.1 Hz, 1H), 1.40 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.5, 140.5, 138.1, 129.2, 128.7, 128.2, 128.1, 128.0, 127.3, 123.3, 121.5, 25.7, 19.3. HRMS (ESI) calcd for $C_{17}H_{15}N$ $[M+H]^+$:234.1283; found: 234.1281.



2,4,4-Triphenylbut-3-enenitrile (**3p**): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.00 (m, 15H), 6.02 (d, *J* = 10.2 Hz, 1H), 3.39 (d, *J* = 9.9 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.0, 140.2, 137.9, 134.9, 129.3, 129.1, 128.8, 128.2, 128.0, 127.4, 127.0, 121.9, 119.4, 36.8. HRMS (ESI) calcd for $C_{22}H_{17}N$ $[M+H]^+$: 296.1439; found: 296.1433.



2,2-Dimethyl-4,4-diphenylbut-3-enenitrile (**3q**)[4]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 - 7.36 (m, 3H), 7.34 - 7.15 (m, 7H), 5.86 (s, 1H), 1.49 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.3, 138.2, 130.2, 129.2, 128.5, 128.4, 128.1, 127.4, 125.2, 123.6, 32.7, 29.8.

References

1. J. M. Concellón, H. Rodríguez-Solla, C. Simal, D. Santos and N. R. Paz, *Org. Lett.*, 2008, **10**, 4549-4552.

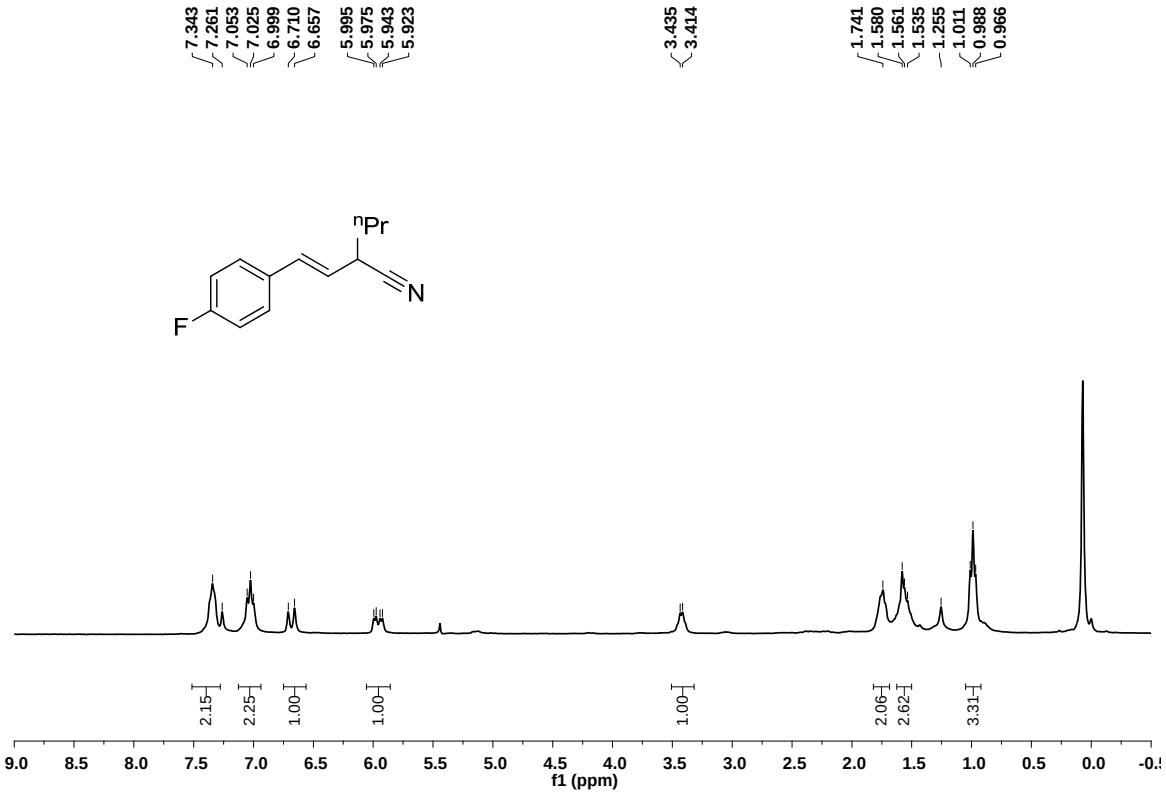
2. E. W. Yankee, F. D. Badea, N. E. Howe and D. J. Cram, *J. Am. Chem. Soc.*, 1973, **95**, 4210-4219.

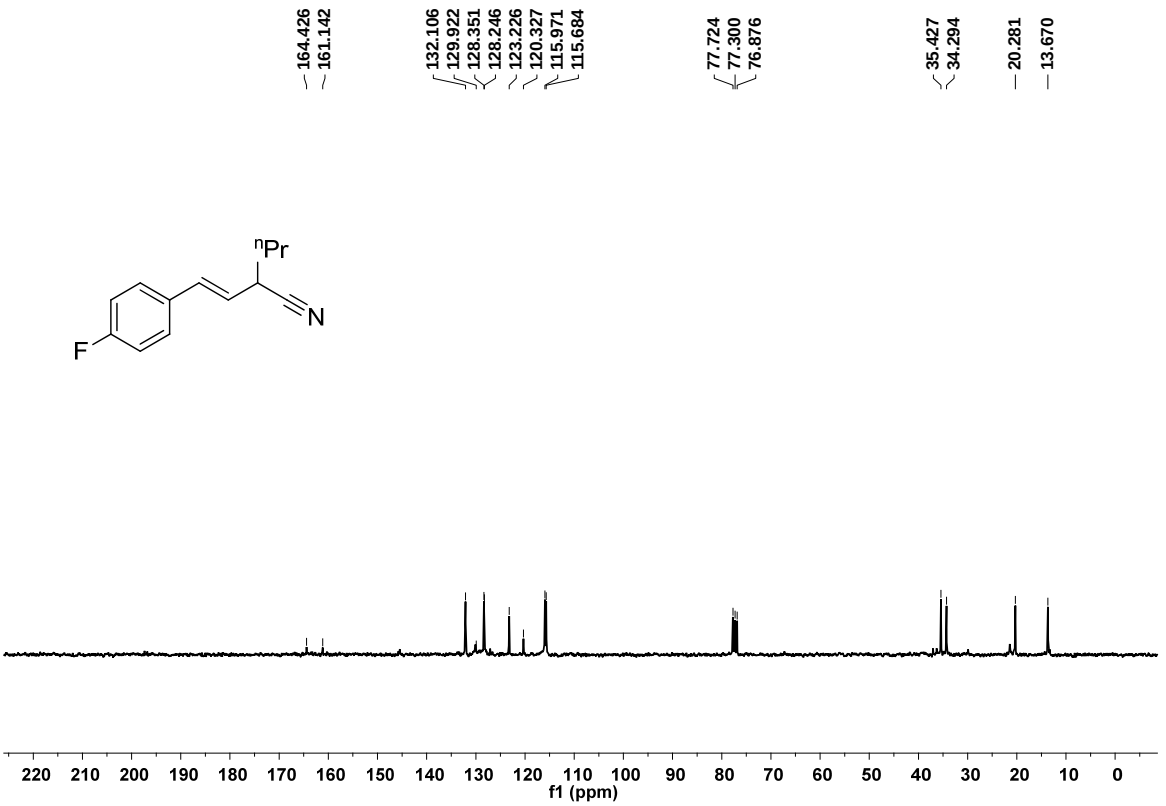
3. E. Keinan and M. Peretz, *J. Org. Chem.*, 1983, **48**, 5302-5309.

4. D. Armesto, M. J. Ortiz, A. R. Agarrabeitia, S. Aparicio-Lara, M. Martin-Fontecha, M. Liras and M. P. Martinez-Alcazar, *J. Org. Chem.*, 2002, **67**, 9397-9405.

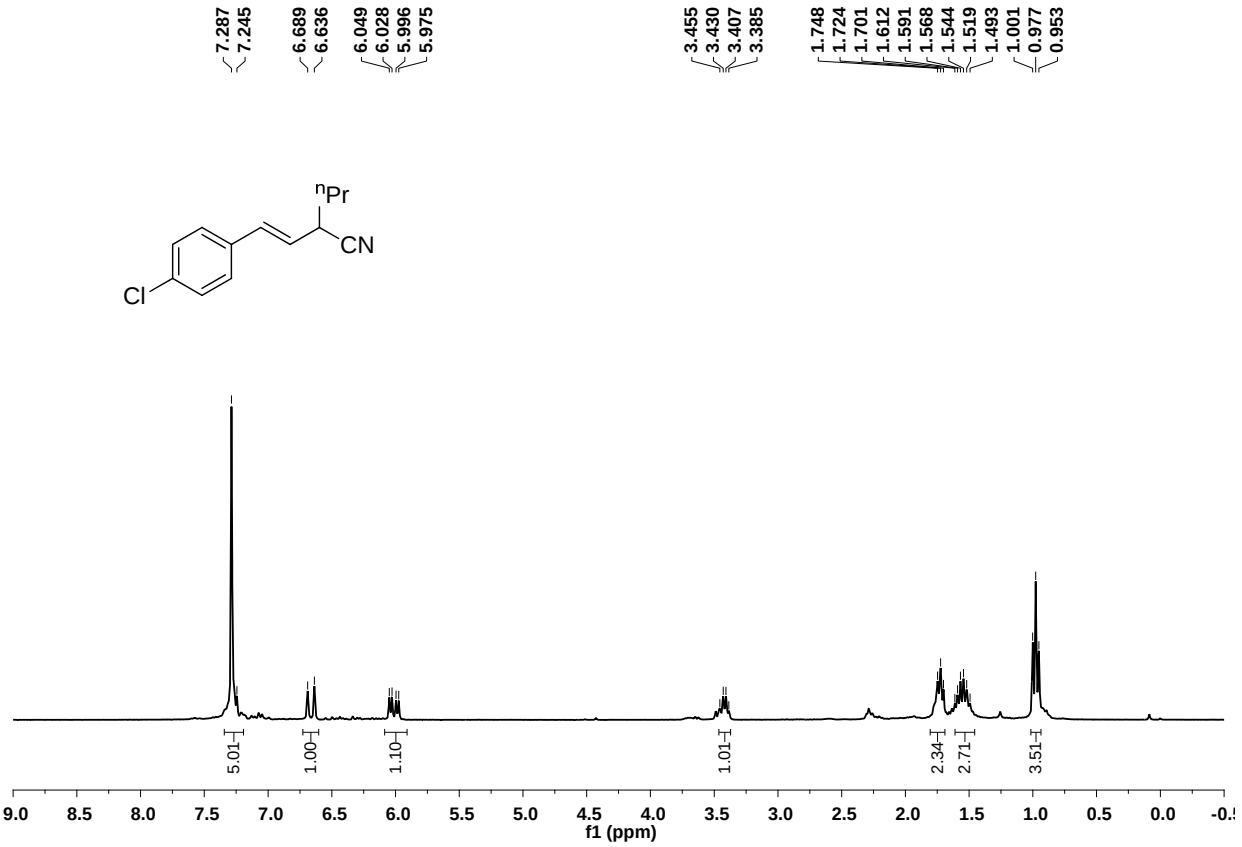
Copies of product ^{1}H NMR and ^{13}C NMR

3a





3b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7.245
6.689
6.636
6.049
6.028
5.996
5.975
3.455
3.430
3.407
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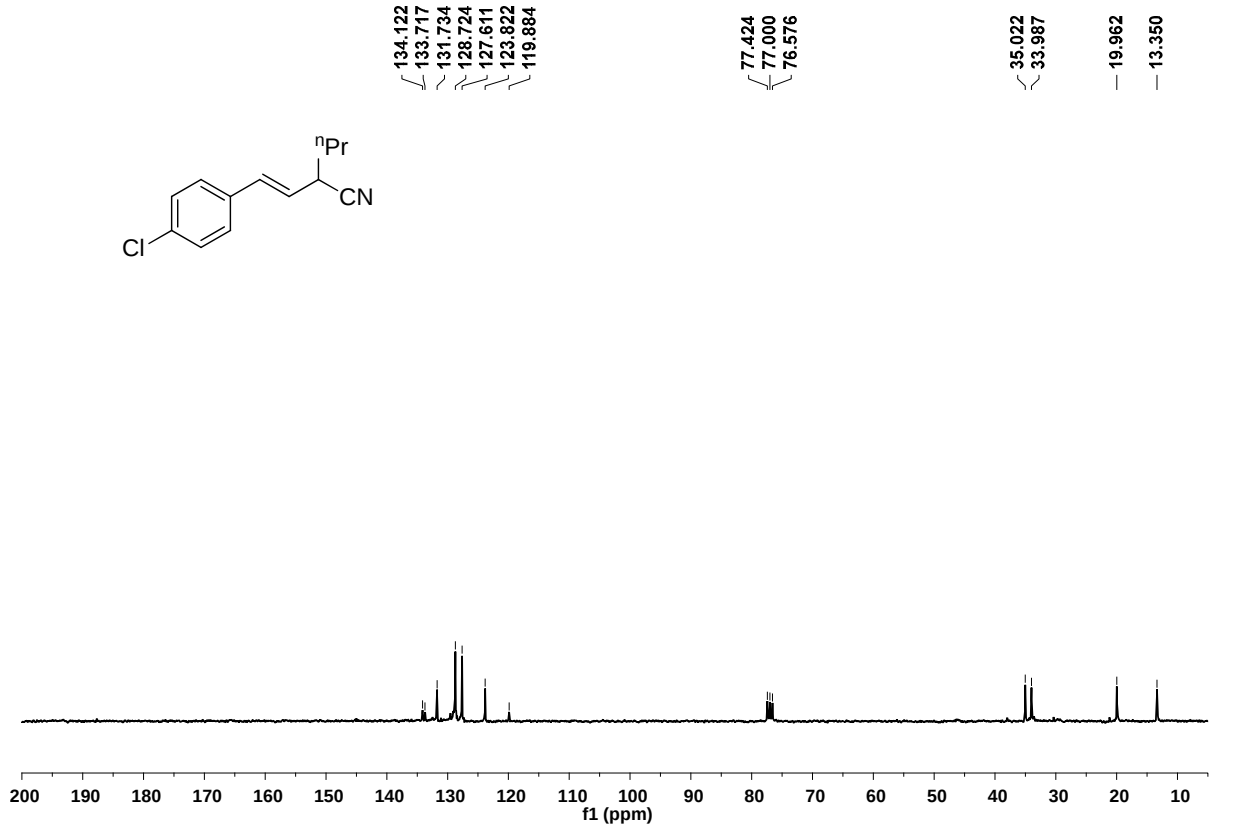
1.748
1.724
1.701
1.612
1.591
1.568
1.544
1.519
1.493
1.001
0.977
0.953
nPr
CN
Cl
5.01
1.00
1.10
1.01
2.34
2.71
3.51
f1 (ppm)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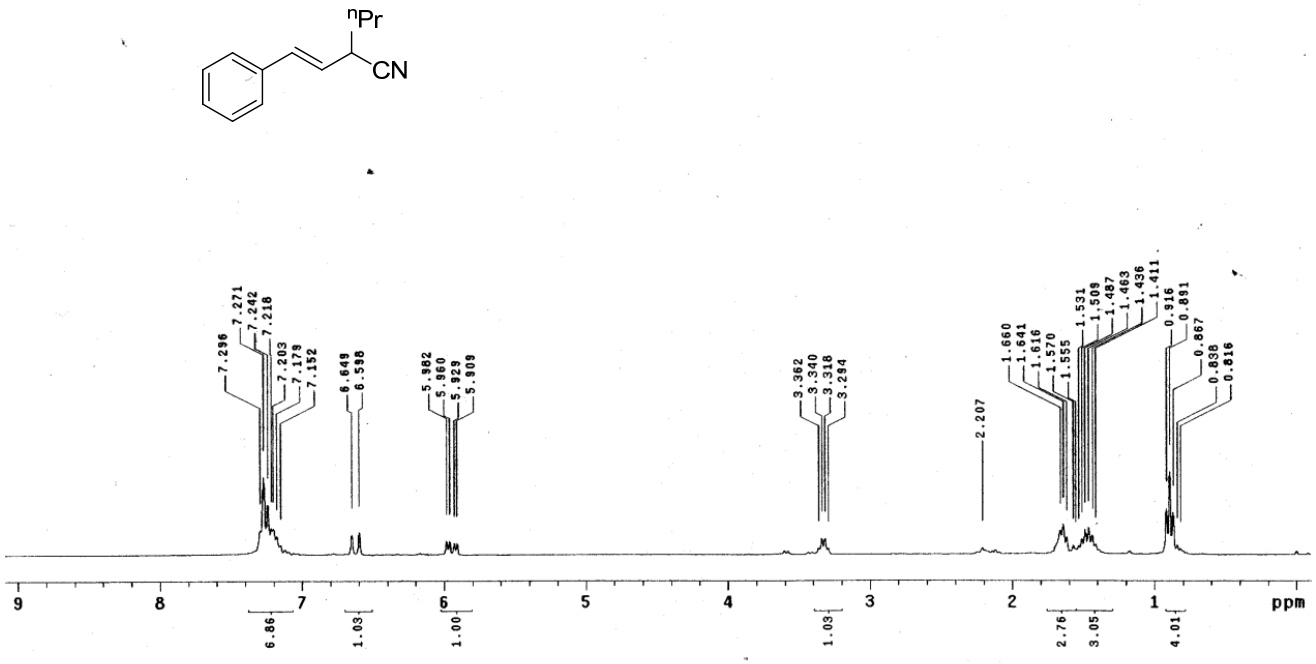
133.717
131.734
128.724
127.611
123.822
119.884
77.424
77.000
76.576
35.022
33.987
19.962
13.350
nPr
CN
Cl
f1 (ppm)

3c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CN
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7.271
7.242
7.218
7.203
7.179
7.152
6.649
6.598
5.962
5.960
5.929
5.909
3.362
3.340
3.318
3.294
2.207
1.660
1.641
1.616
1.570
1.555
1.531
1.509
1.487
1.463
1.436
1.411
0.916
0.891
0.867
0.838
0.816
9
8
7
6
5
4
3
2
1
ppm
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1.03
1.00
1.03
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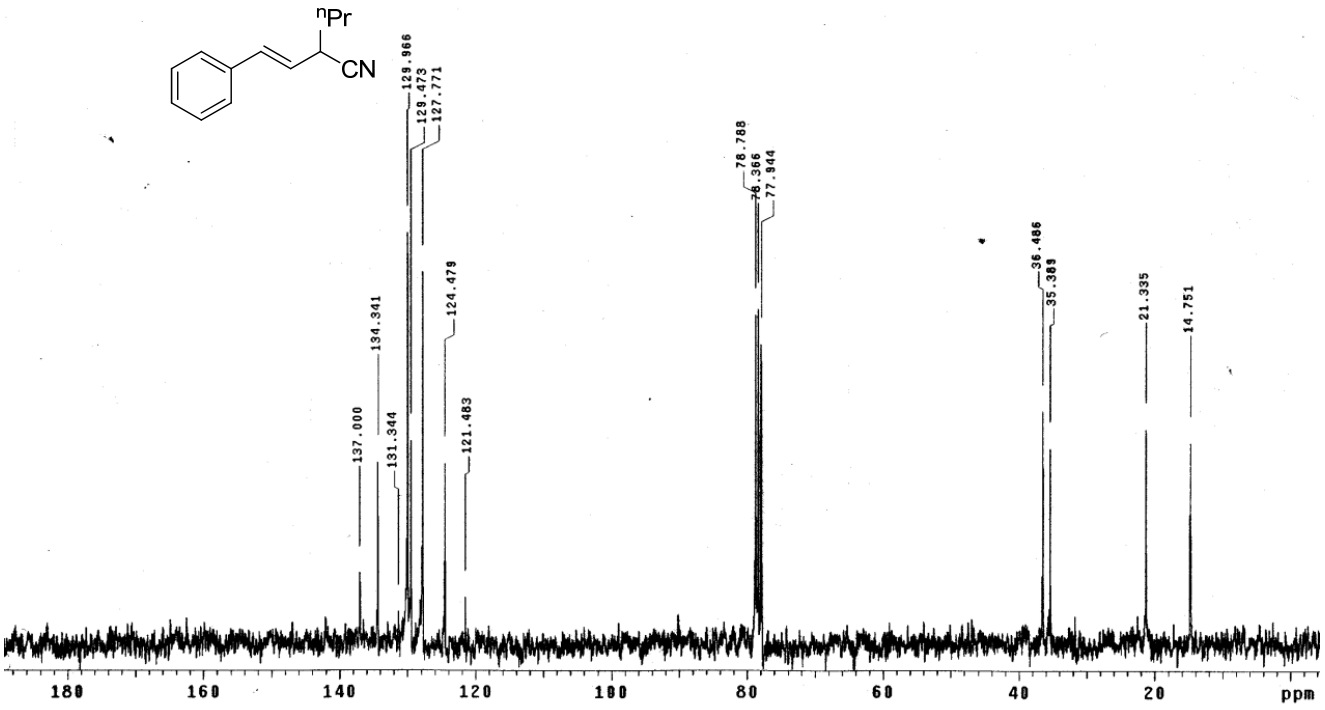
3.05
4.01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CN
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129.473
127.771
134.341
137.000
131.344
124.479
121.483
78.788
78.366
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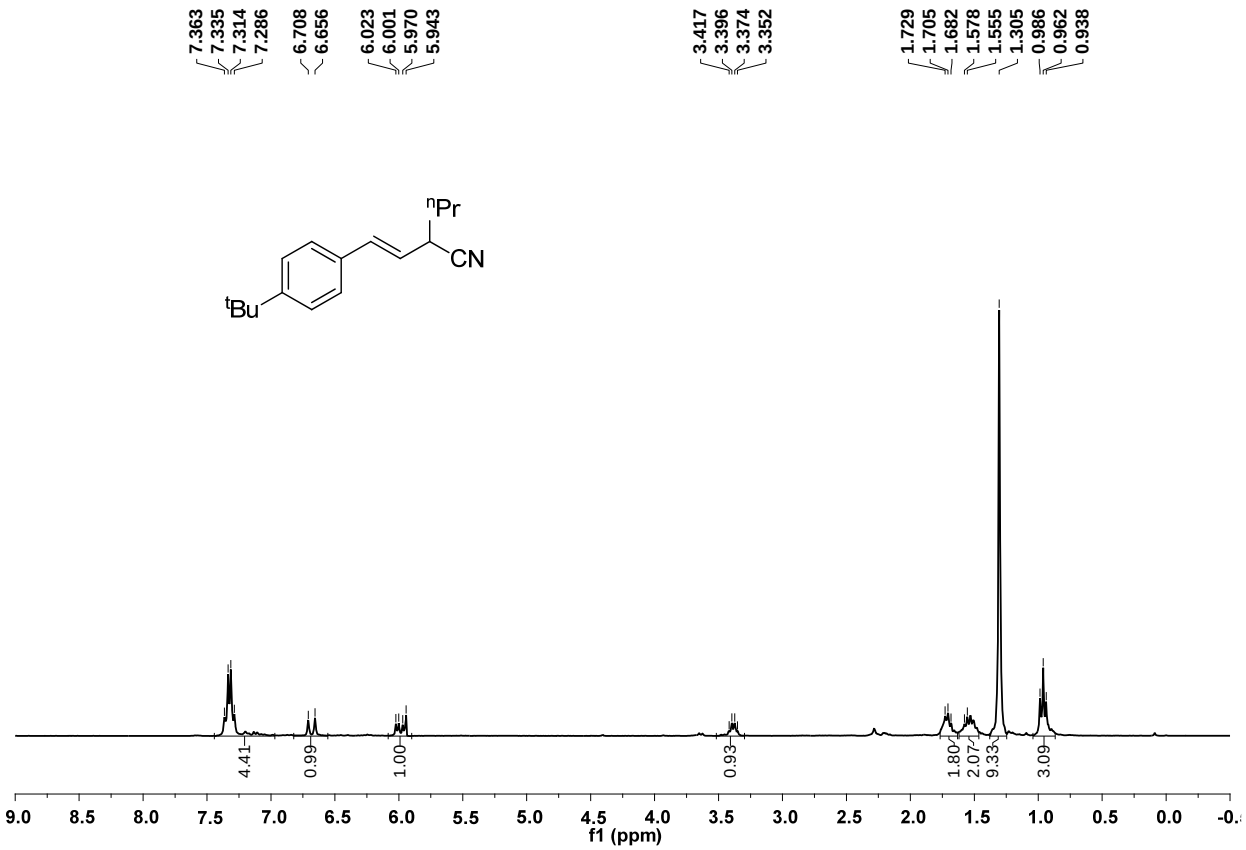
36.486
35.389
21.335
14.751
180
160
140
120
100
80
60
40
20
ppm

3d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7.335
7.314
7.286
6.708
6.656
6.023
6.001
5.970
5.943
3.417
3.396
3.374
3.352
1.729
1.705
1.682
1.578
1.555
1.305
0.986
0.962
0.938
nPr
CN
tBu
4.41
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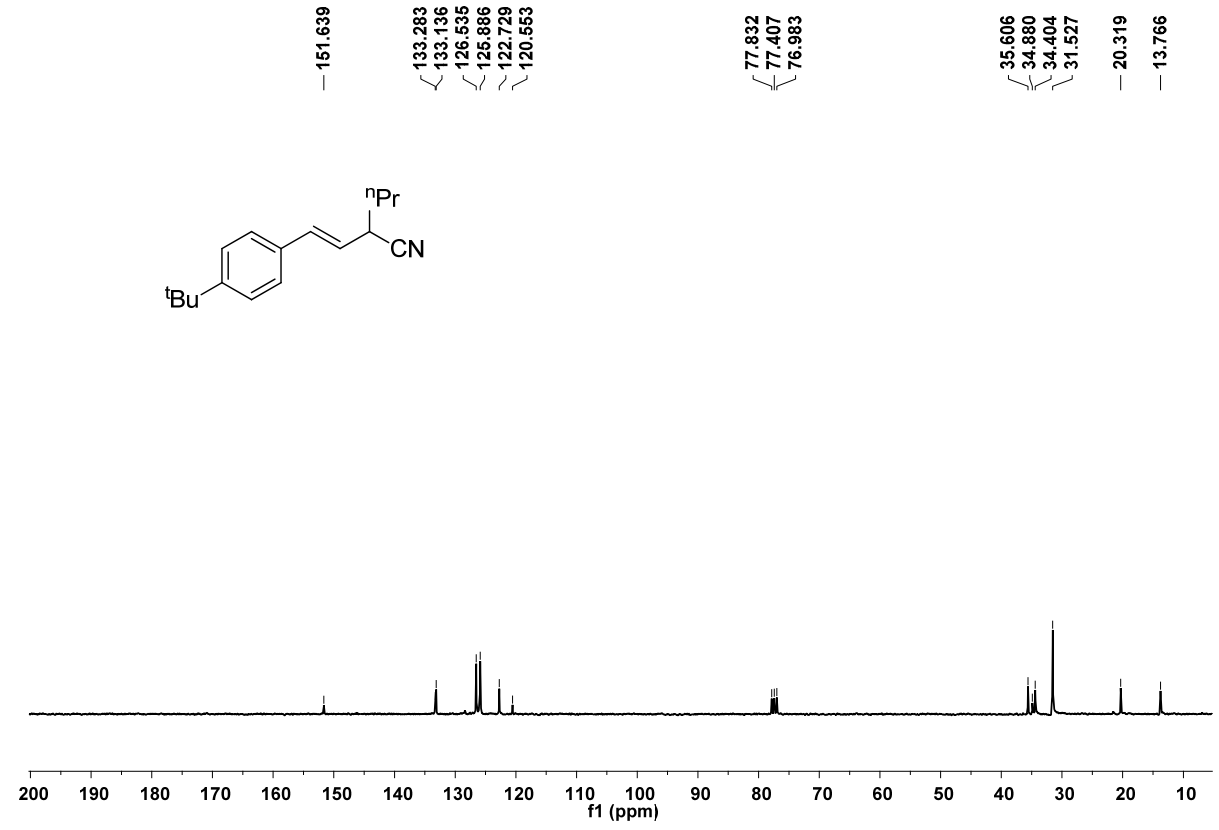
2.07
9.33
3.09
f1 (ppm)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77.407
76.983
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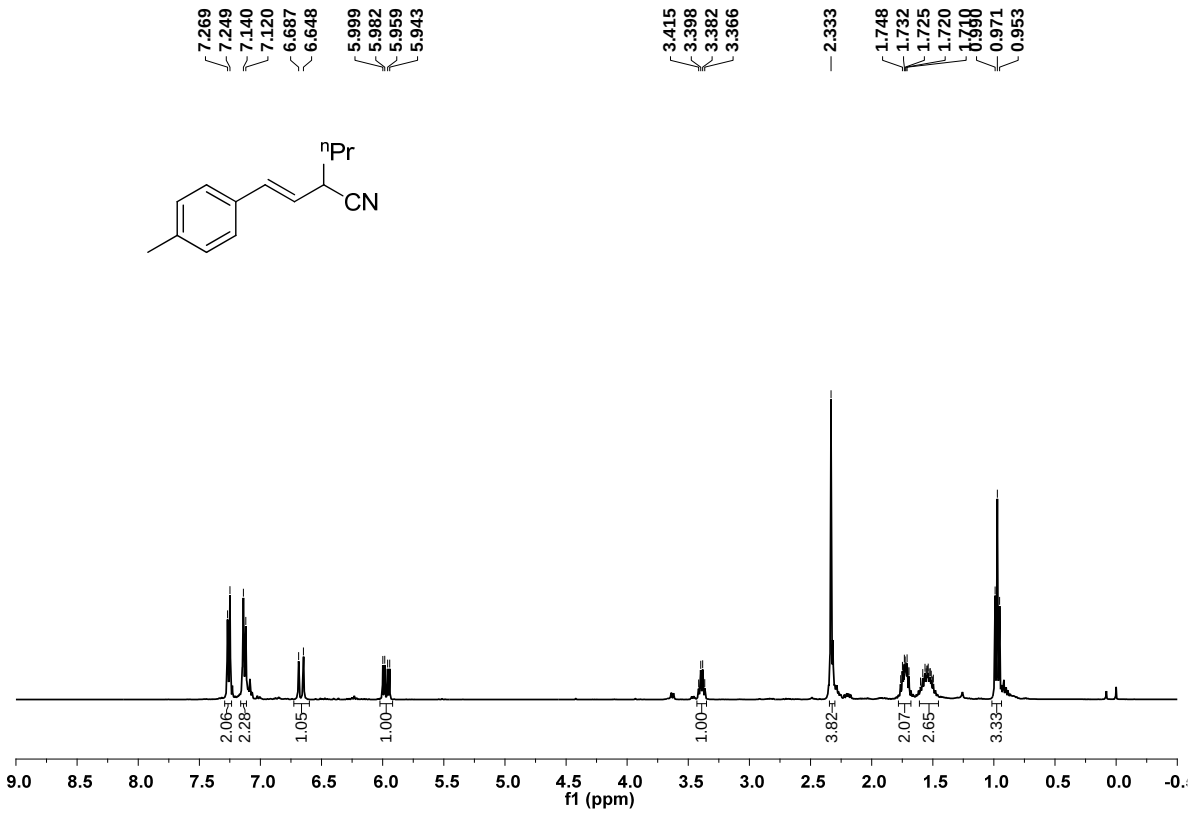
34.880
34.404
31.527
20.319
13.766
nPr
CN
tBu
f1 (ppm)

3e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7.140
7.120
6.687
6.648
5.999
5.982
5.959
5.943
3.415
3.398
3.382
3.366
2.333
1.748
1.732
1.725
1.720
0.990
0.971
0.953
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CN
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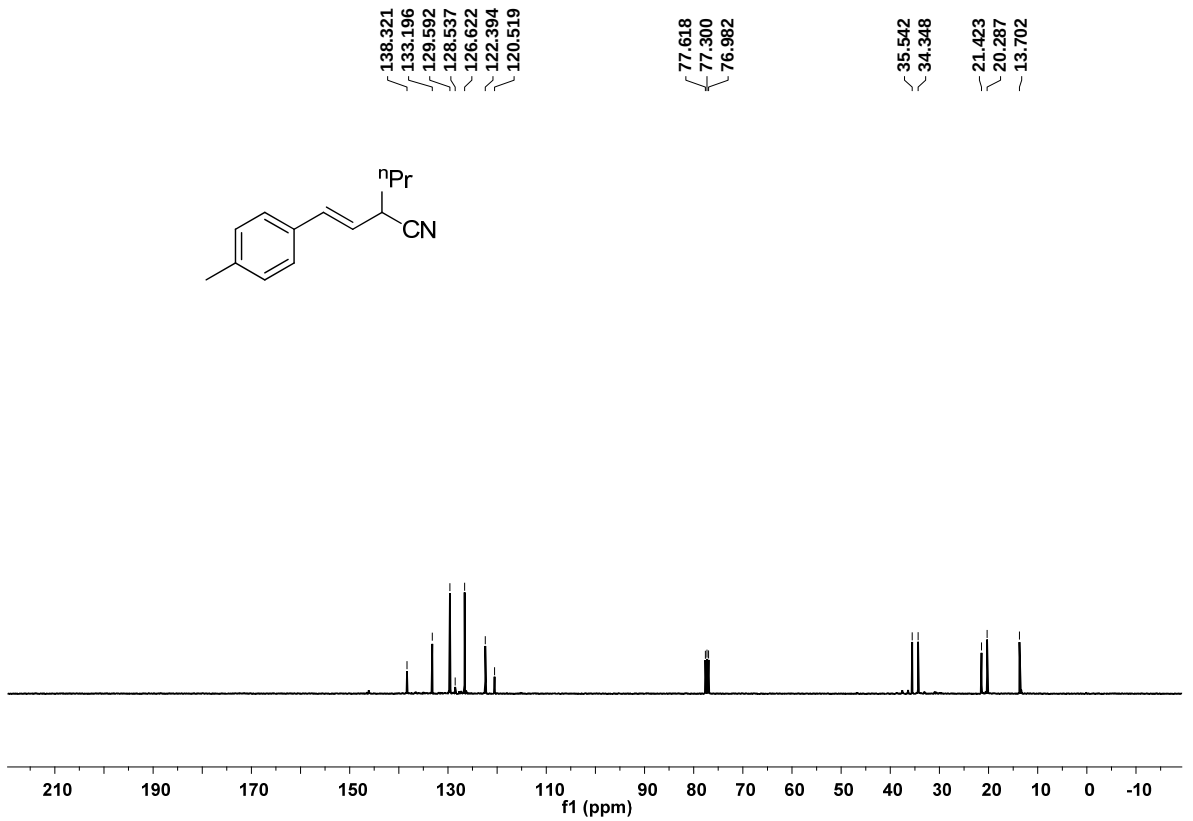
2.28
1.05
1.00
1.00
3.82
2.07
2.65
3.33
f1 (ppm)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128.537
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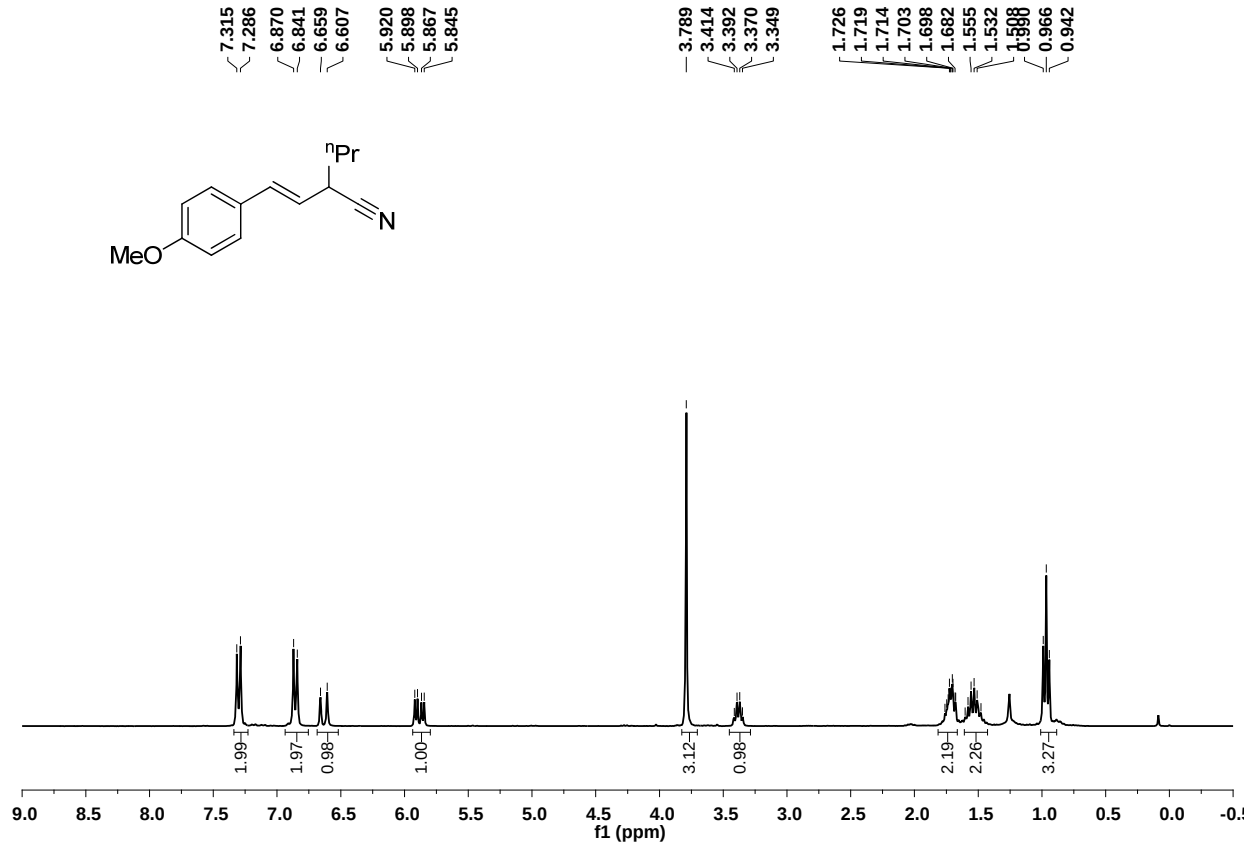
122.394
120.519
77.618
77.300
76.982
35.542
34.348
21.423
20.287
13.702
nPr
CN
f1 (ppm)

3f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5.898
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5.845
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3.414
3.392
3.370
3.349
1.726
1.719
1.714
1.703
1.698
1.682
1.555
1.532
0.966
0.942
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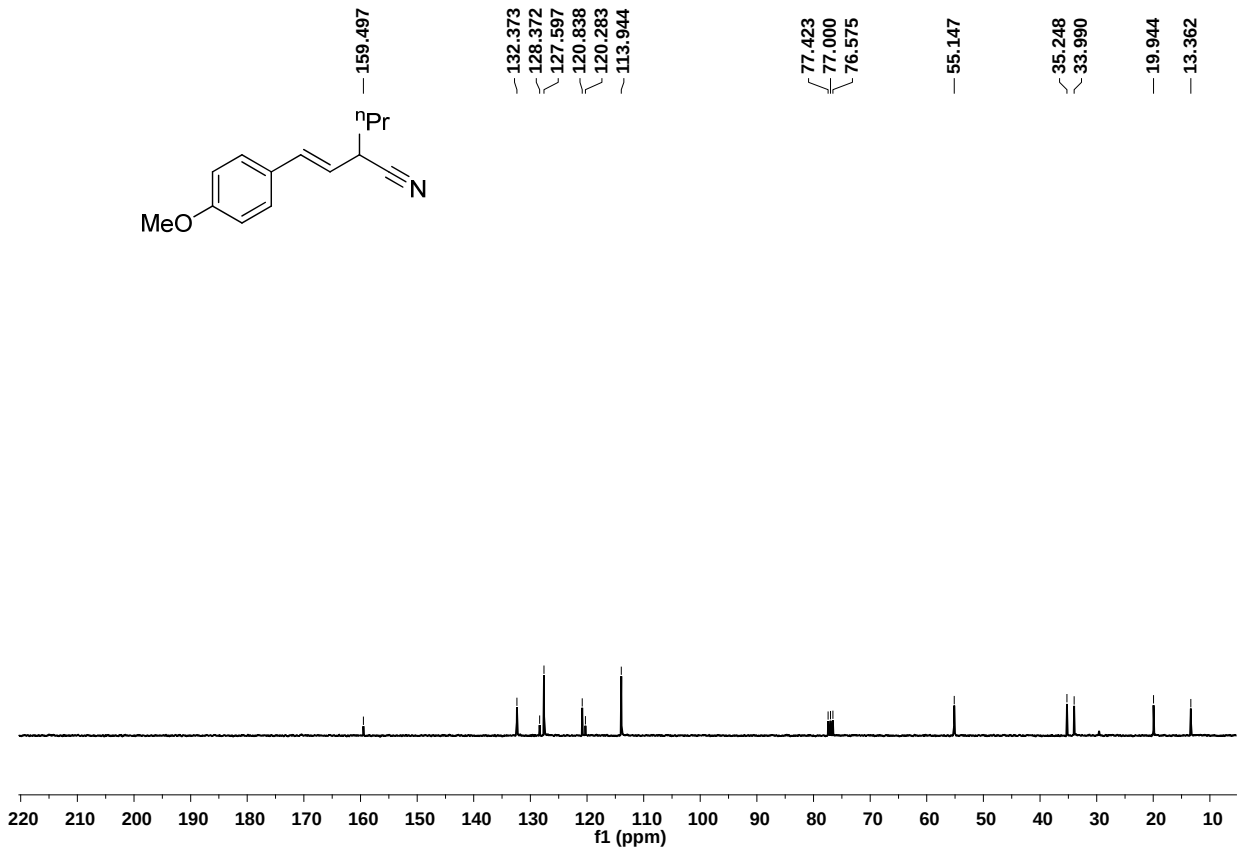
3.27
f1 (ppm)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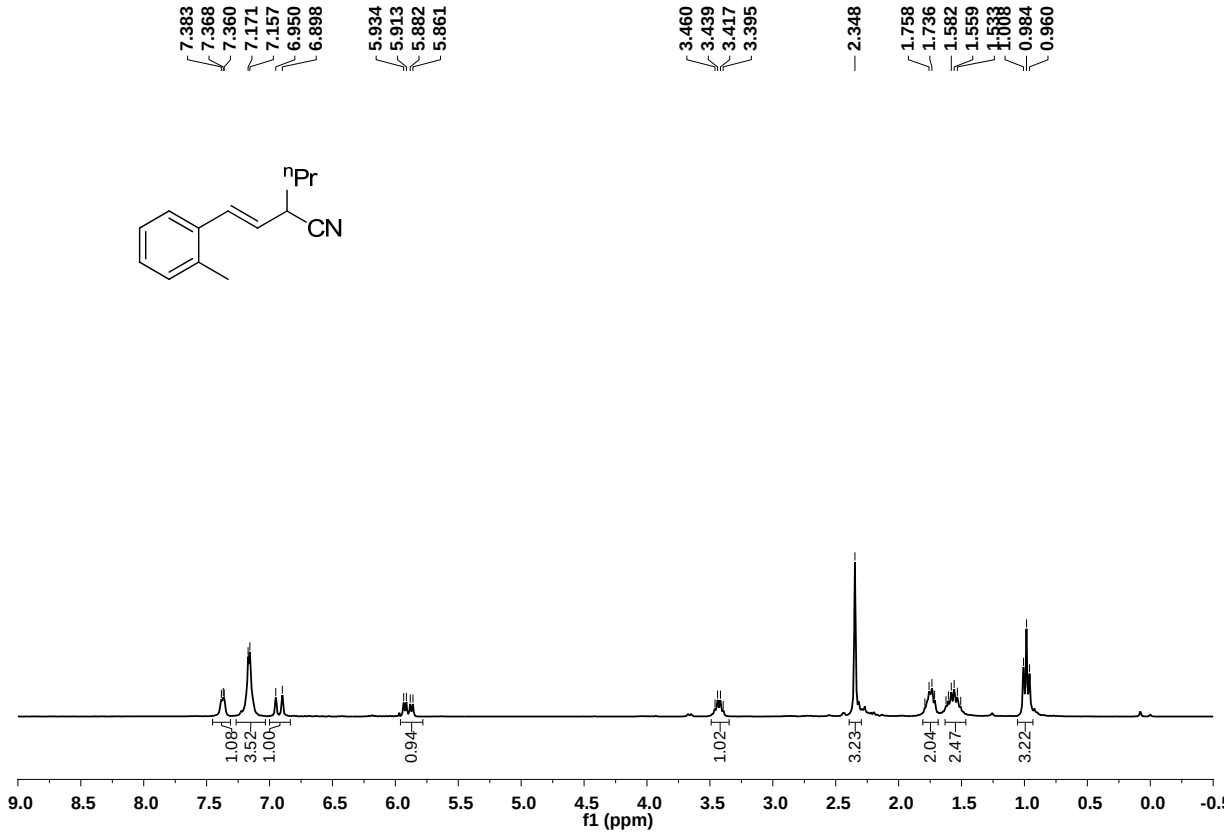
N
MeO
f1 (ppm)

3g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6.898
5.934
5.913
5.882
5.861
3.460
3.439
3.417
3.395
2.348
1.758
1.736
1.582
1.559
1.533
1.008
0.984
0.960
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CN
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3.52
1.00
0.94
1.02
3.23
2.04
2.47
3.22
f1 (ppm)
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135.139
131.272
130.520
128.213
126.307
125.888
124.818
120.406
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76.844
35.438
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13.635
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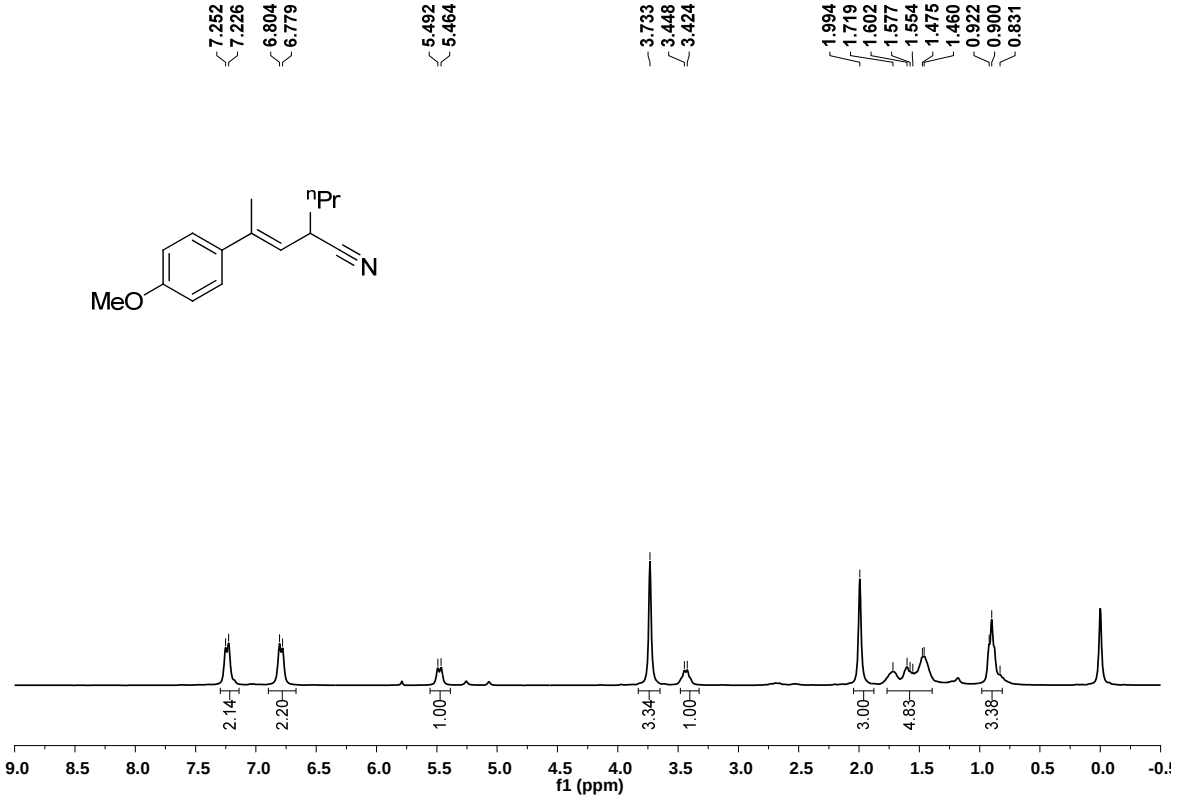
CN
f1 (ppm)

3h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5.464
3.733
3.448
3.424
1.994
1.719
1.602
1.577
1.554
1.475
1.460
0.922
0.900
0.831
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N
MeO
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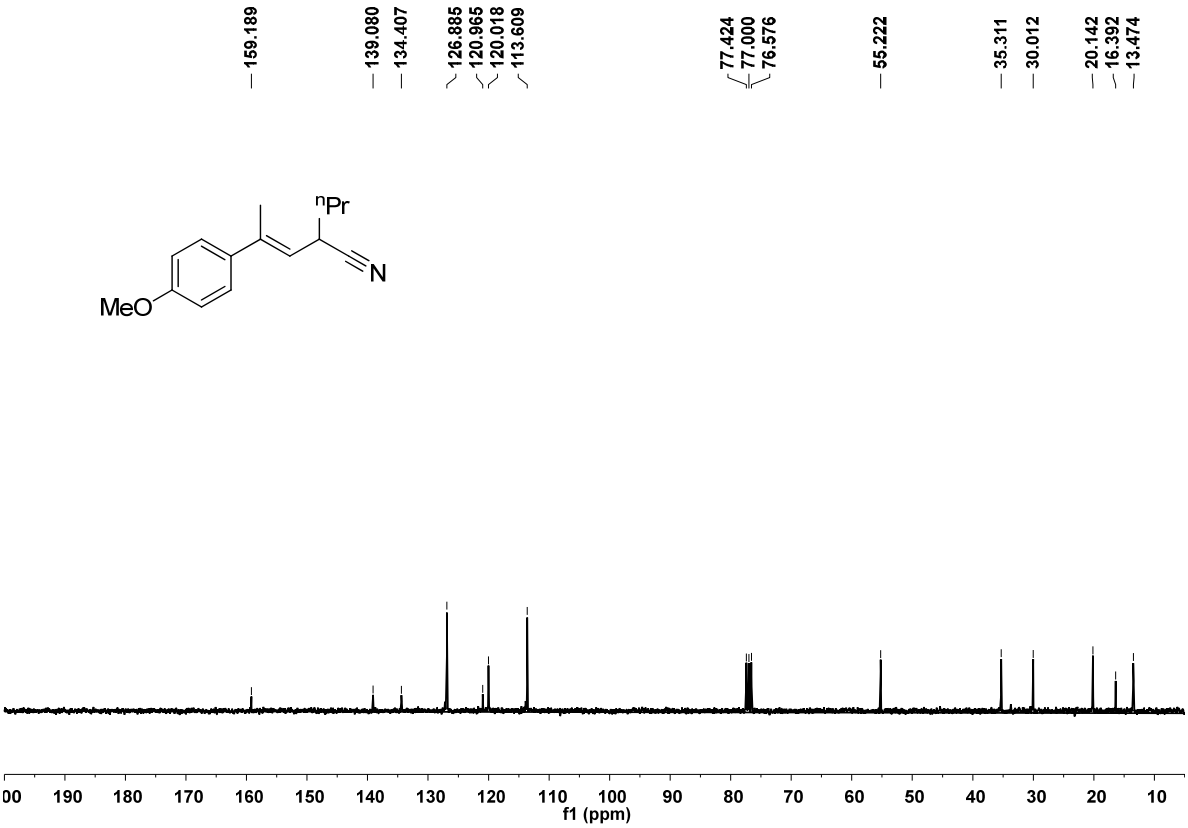
1.00
3.00
4.83
3.38
f1 (ppm)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77.000
76.576
55.222
35.311
30.012
20.142
16.392
13.474
nPr
N
MeO
f1 (ppm)

3i


7.415
7.394
7.369
7.363
7.341
7.264
7.258
7.248
7.241
7.228
7.221
7.209
7.196
7.191
7.170
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5.925
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3.311
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3.287
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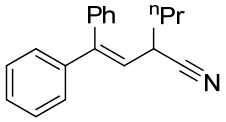
0.817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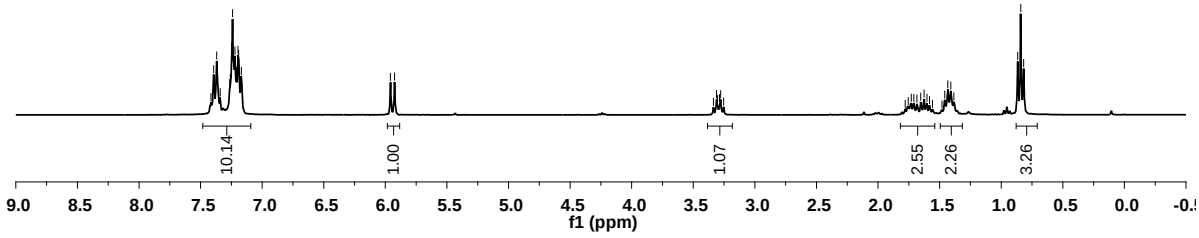
N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6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


146.101
140.652
138.286
129.287
128.678
128.273
128.079
127.958
127.347
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76.575
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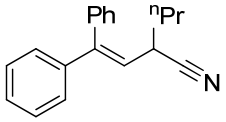
13.356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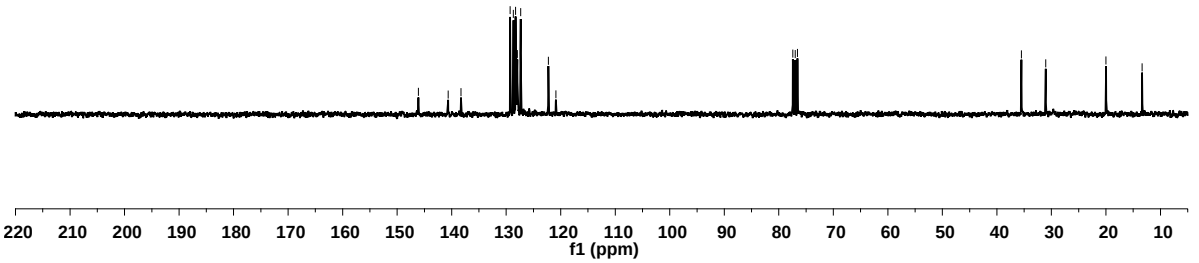
N


220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

3j


7.615
7.588
7.458
7.431
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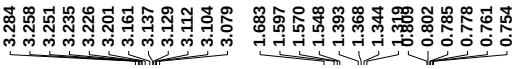
5.942
5.908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3.137
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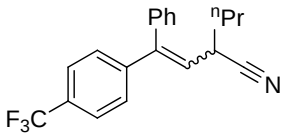
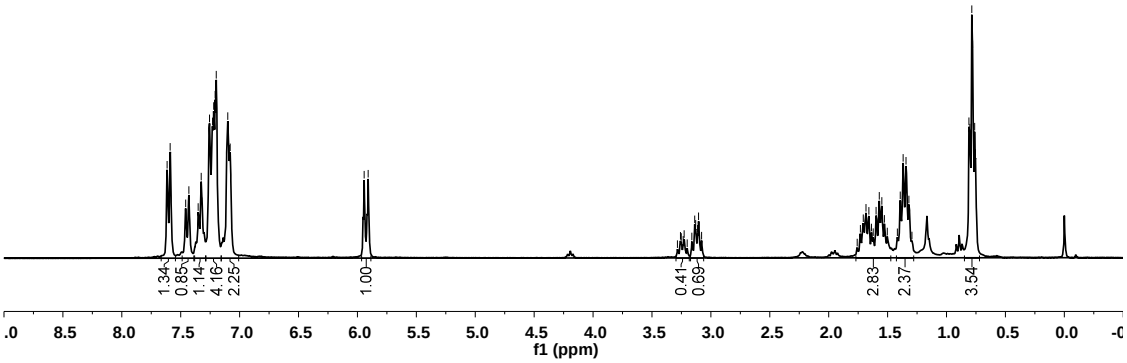

Ph
nPr
N
F3C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0.41
0.69
2.83
2.37
3.54
f1 (ppm)


144.984
144.871
144.107
142.062
139.812
137.424
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129.721
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128.335
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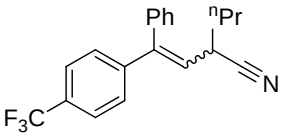
13.292


Ph
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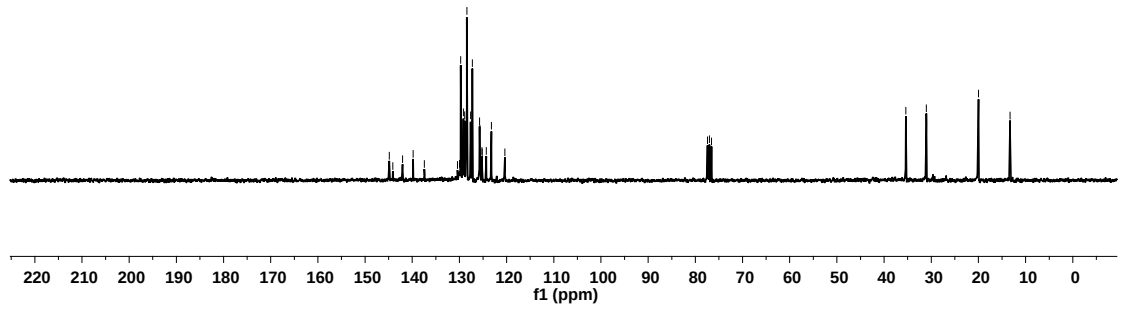
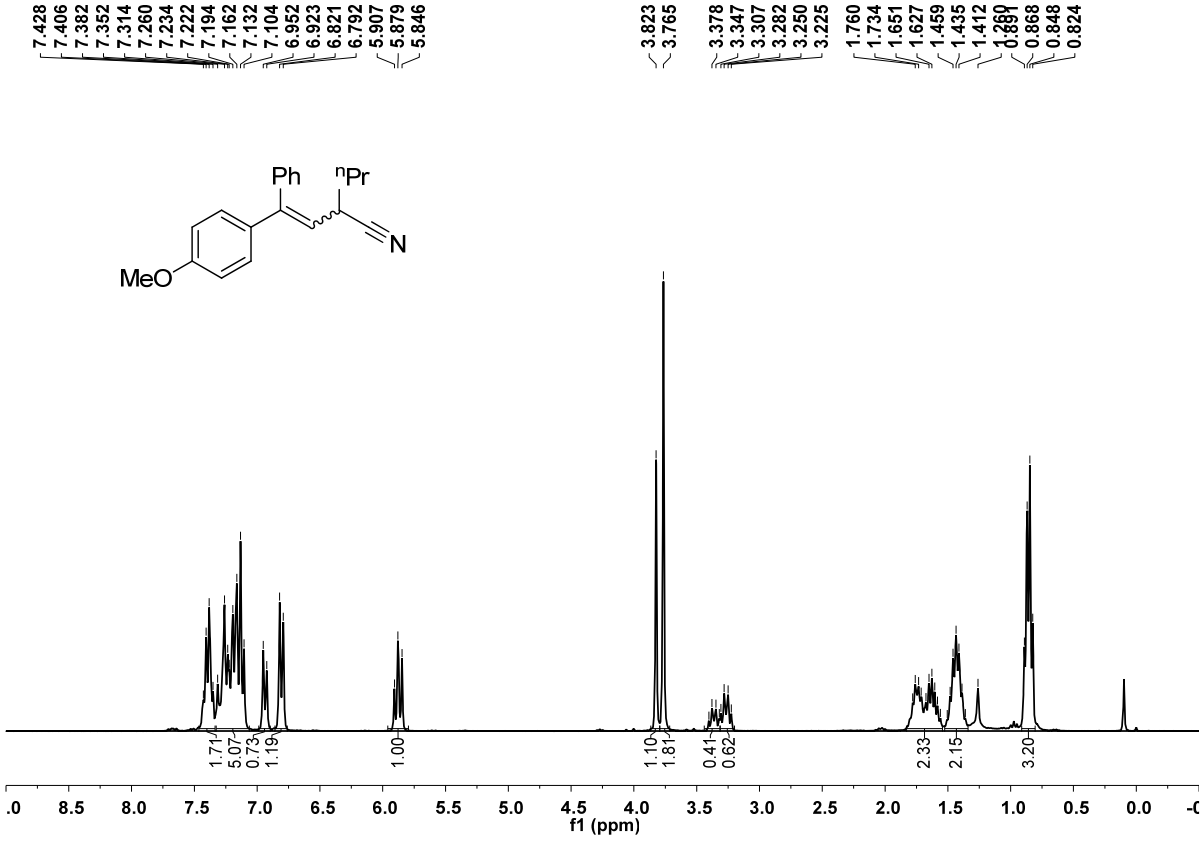
N
F3C


f1 (ppm)

3k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6.952
6.923
6.821
6.792
5.907
5.879
5.846
3.823
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3.378
3.347
3.307
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0.891
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Ph
nPr
N
MeO
1.71
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0.62
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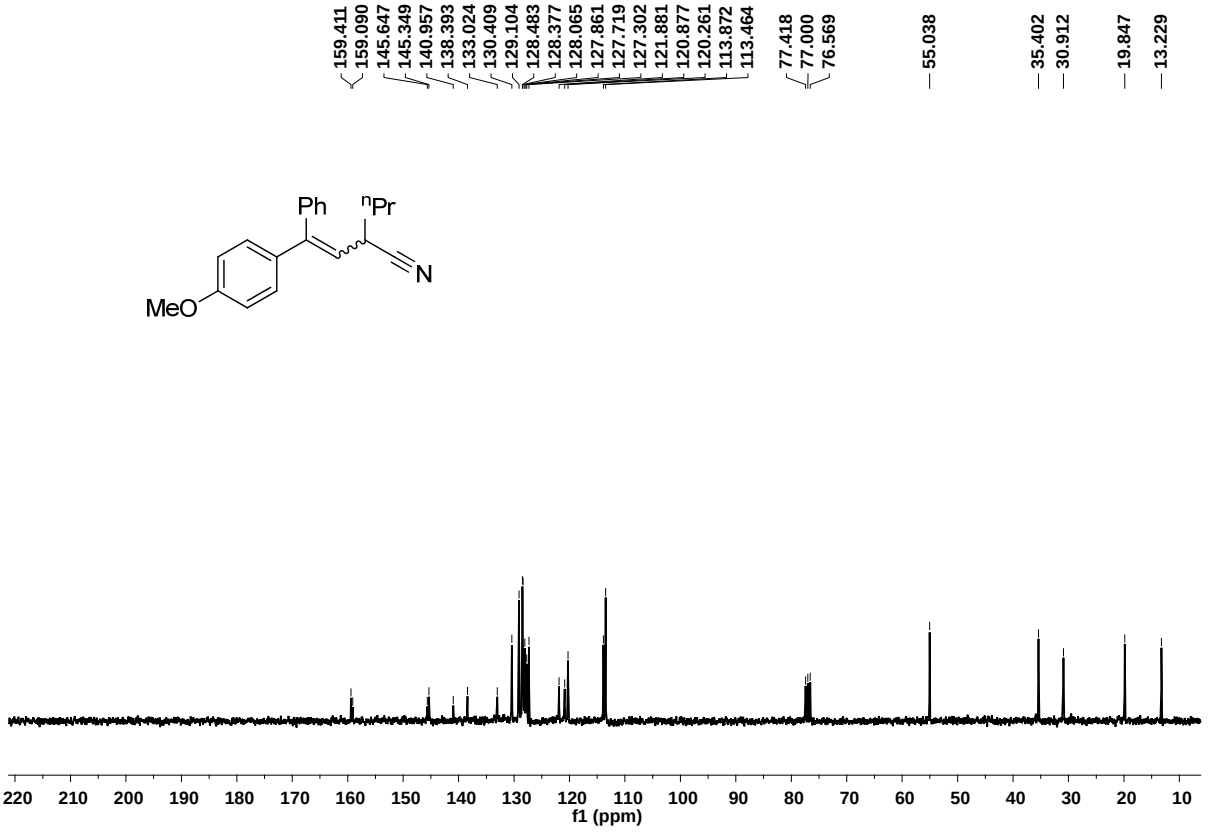
2.15
3.20
f1 (ppm)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127.719
127.302
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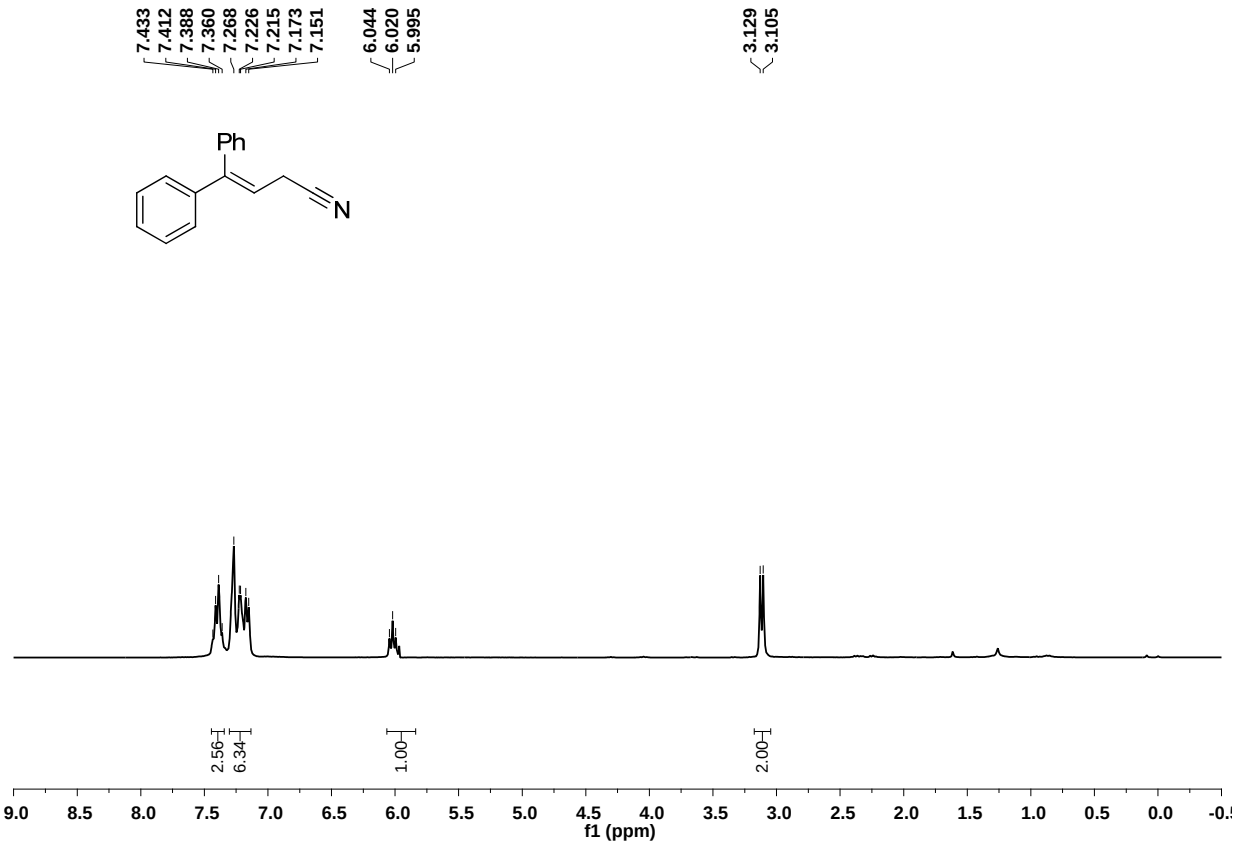
Ph
nPr
N
MeO
f1 (ppm)

3l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Ph
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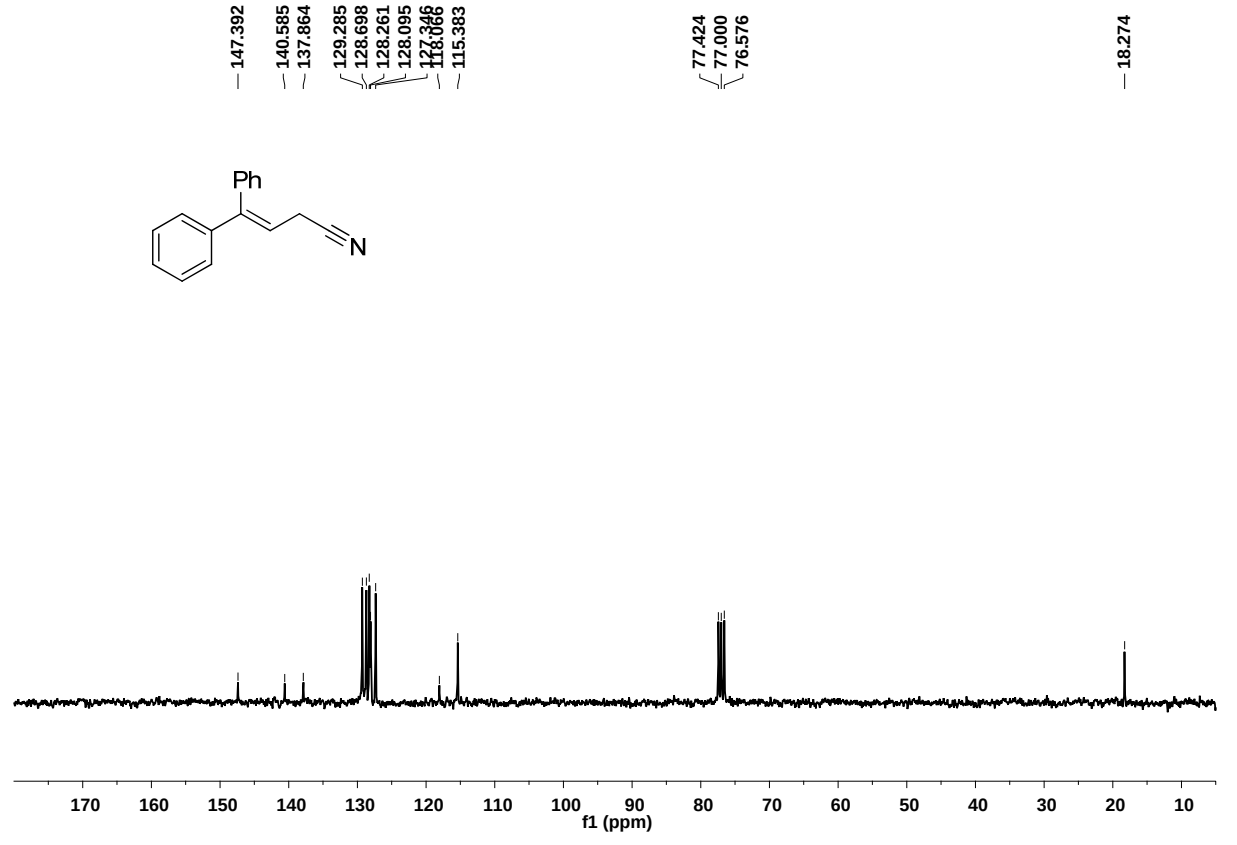
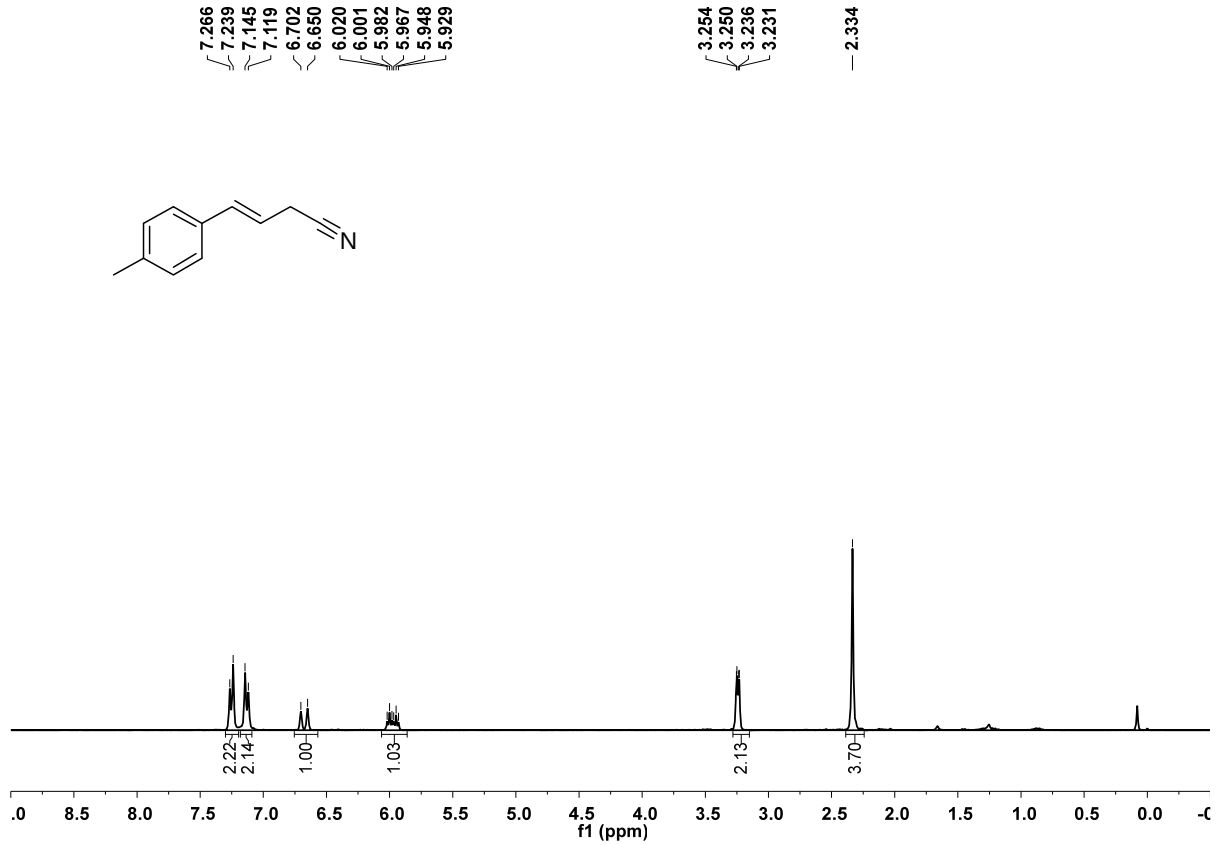
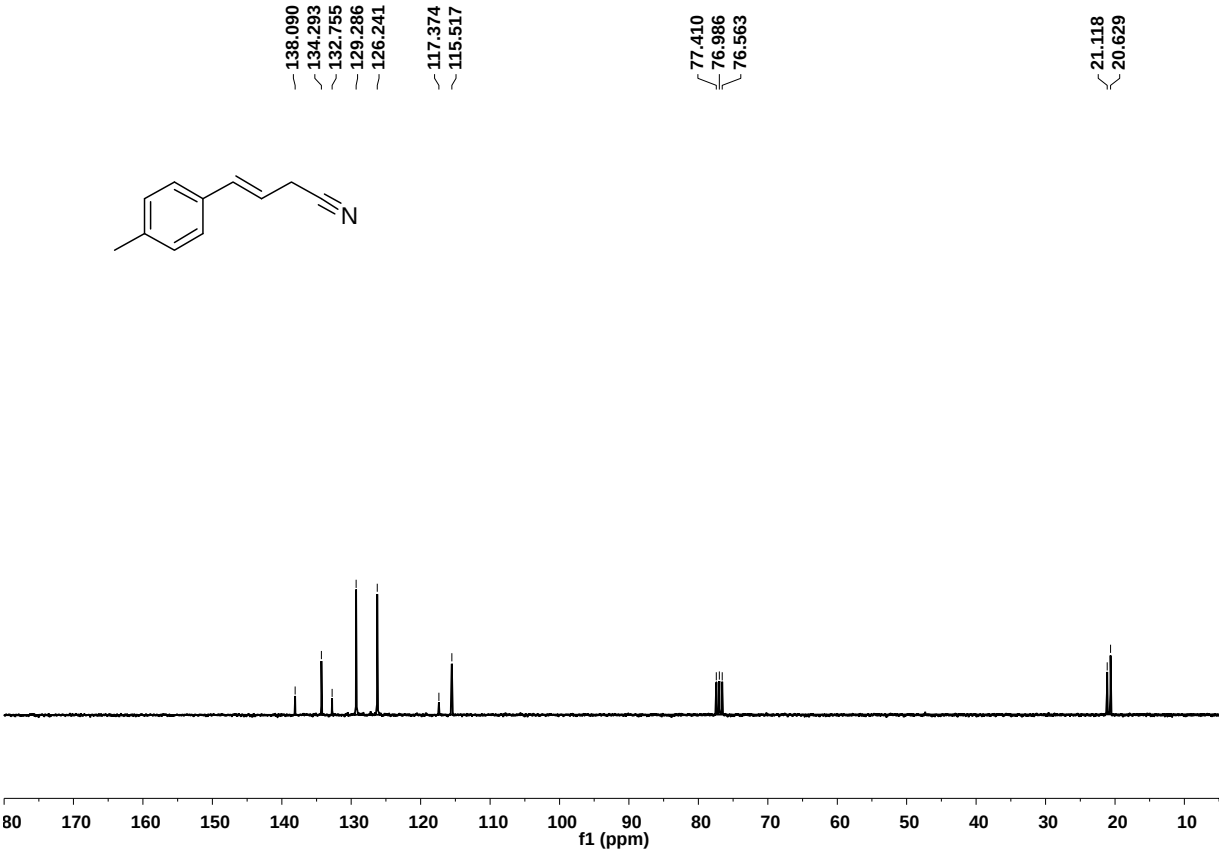
1.00
2.00
f1 (ppm)

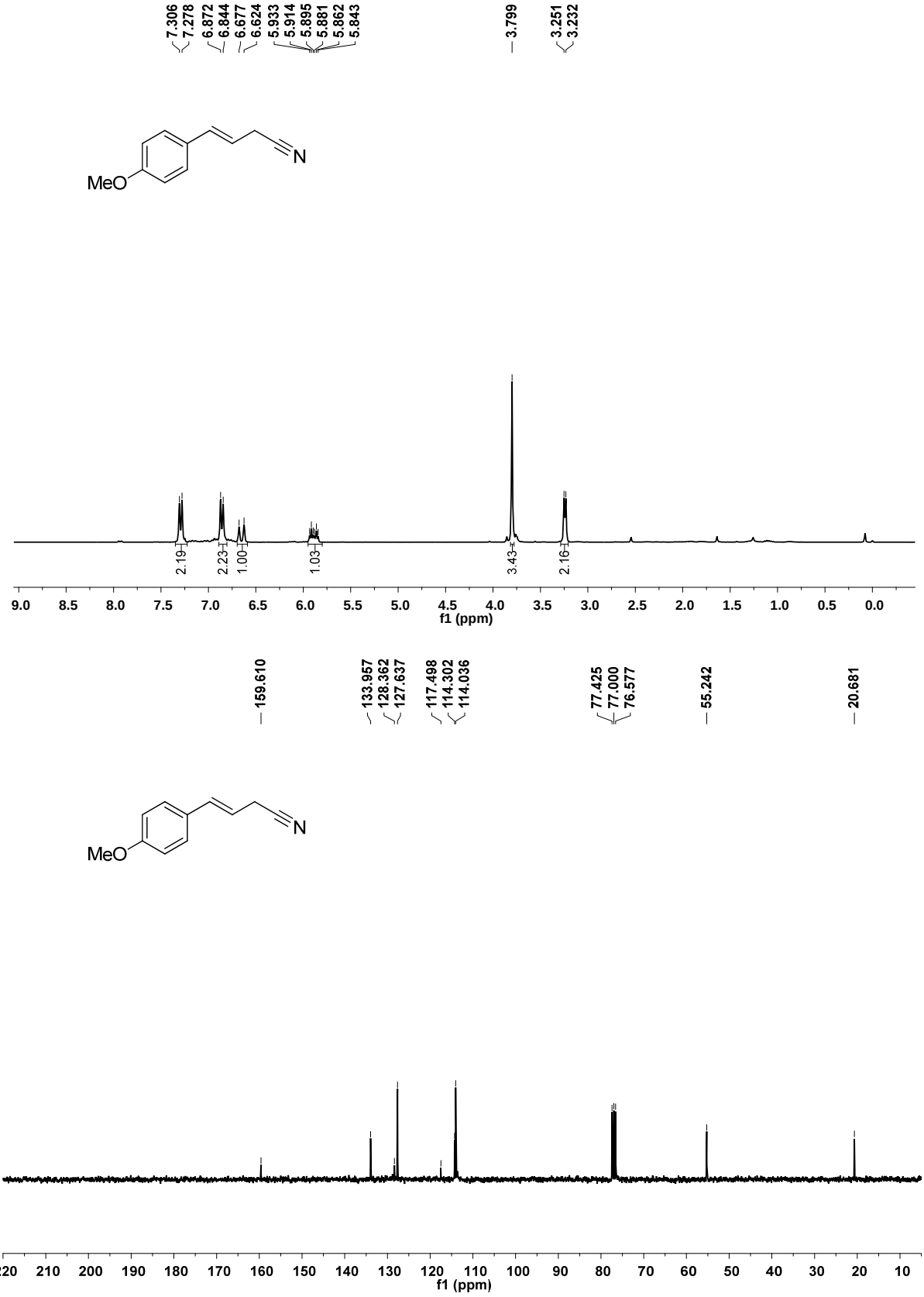

147.392
140.585
137.864
129.285
128.698
128.261
128.095
118.066
115.383
77.424
77.000
76.576
18.274
Ph
N
f1 (ppm)

3m

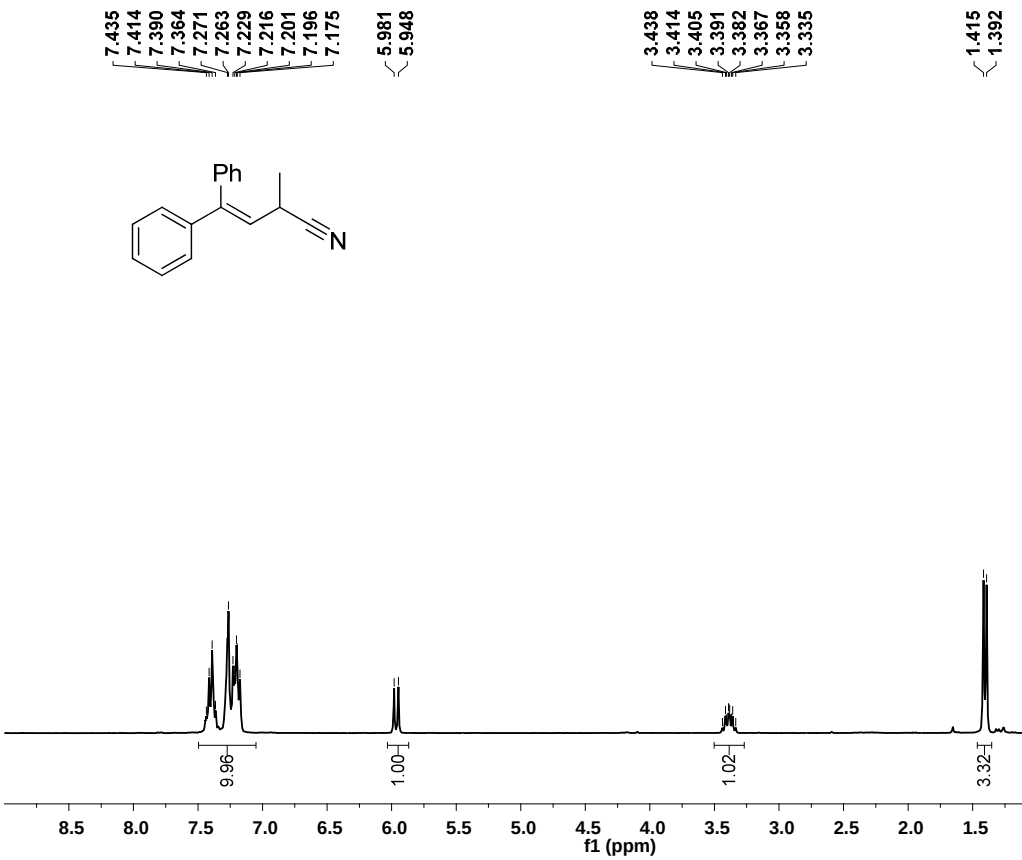


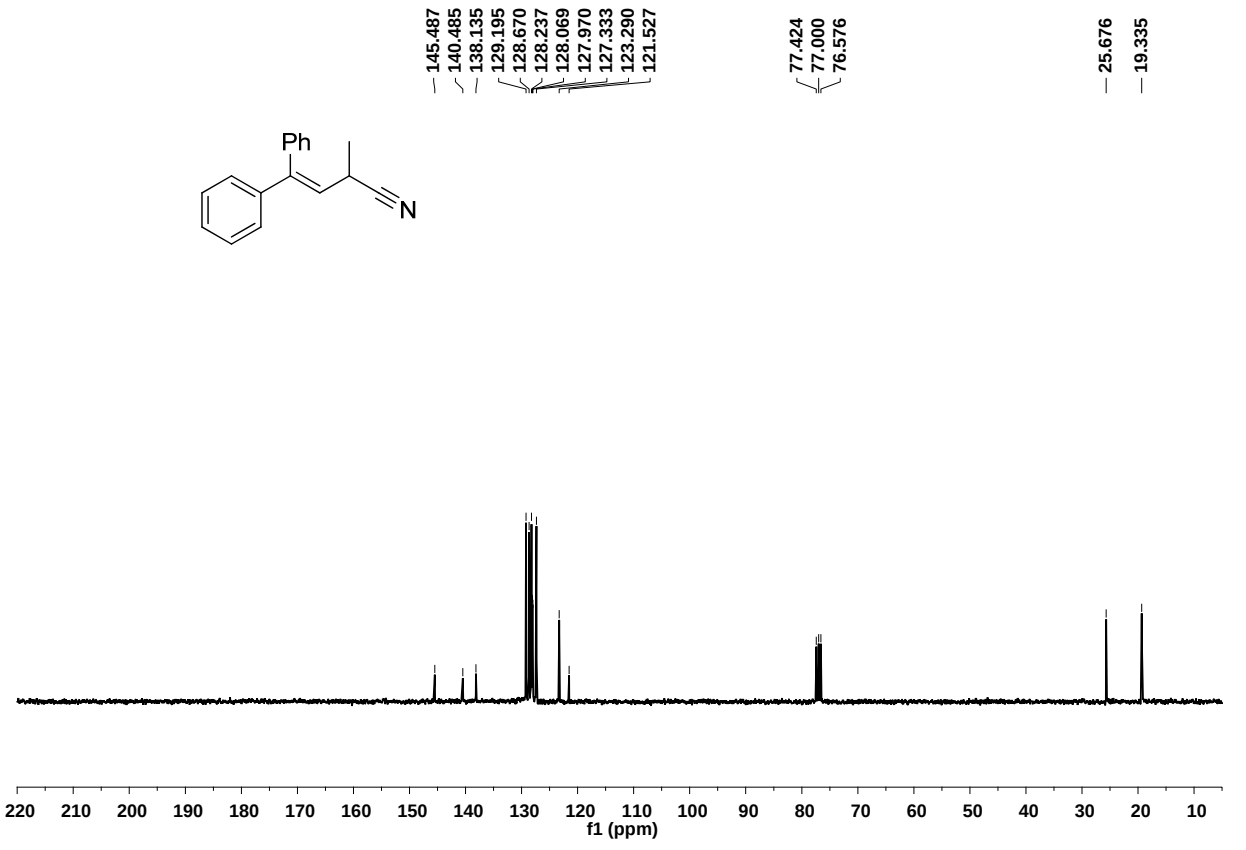


3n

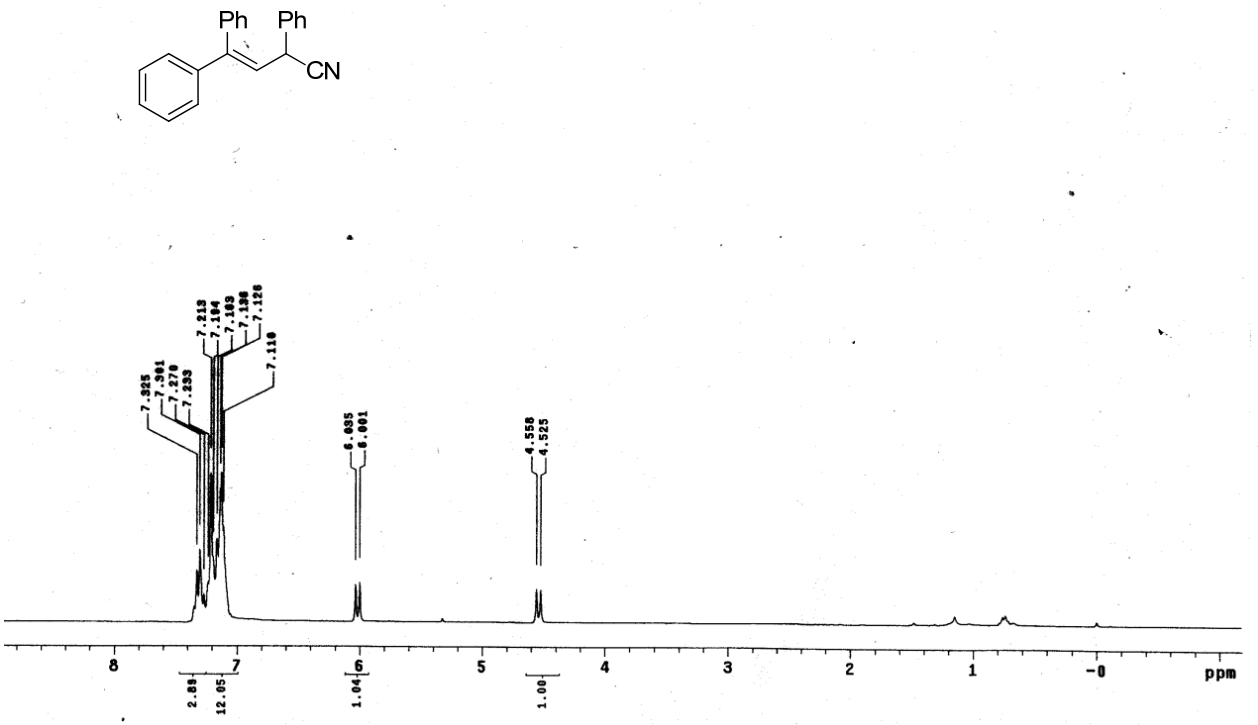

7.306
7.278
6.872
6.844
6.677
6.624
5.933
5.914
5.895
5.881
5.862
5.843
3.799
3.251
3.232
MeO
N
2.19
2.23
1.00
1.03
3.43
2.16
f1 (ppm)
159.610
133.957
128.362
127.637
117.498
114.302
114.036
77.425
77.000
76.577
55.242
20.681
MeO
N
f1 (ppm)

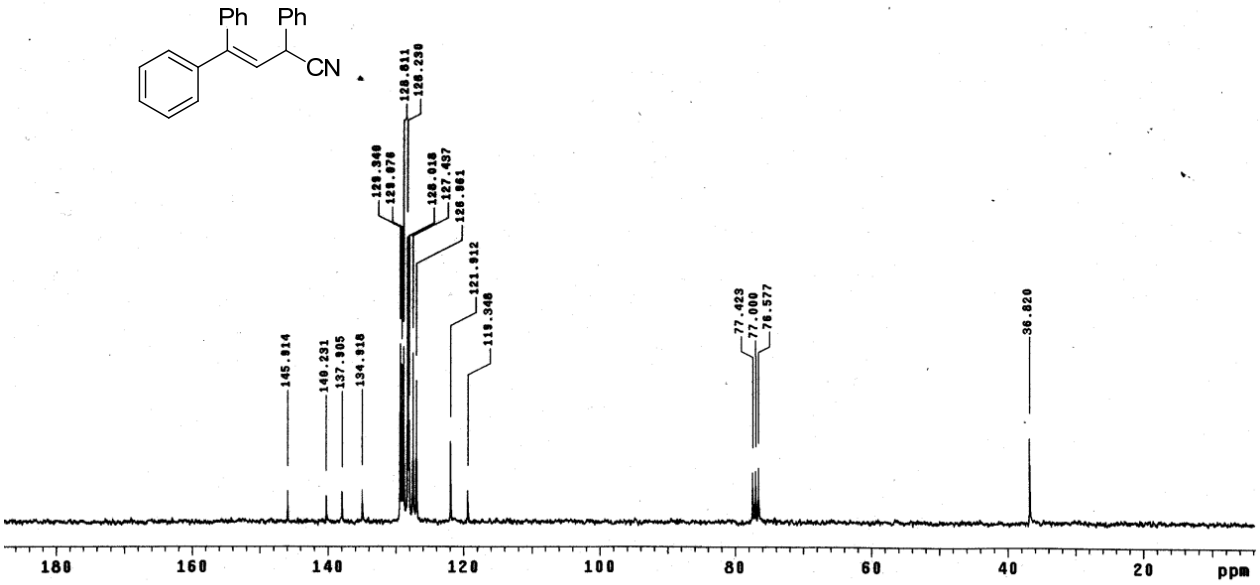
3o


7.435
7.414
7.390
7.364
7.271
7.263
7.229
7.216
7.201
7.196
7.175
5.981
5.948
3.438
3.414
3.405
3.391
3.382
3.367
3.358
3.335
1.415
1.392
Ph
N
9.96
1.00
1.02
3.32
f1 (ppm)

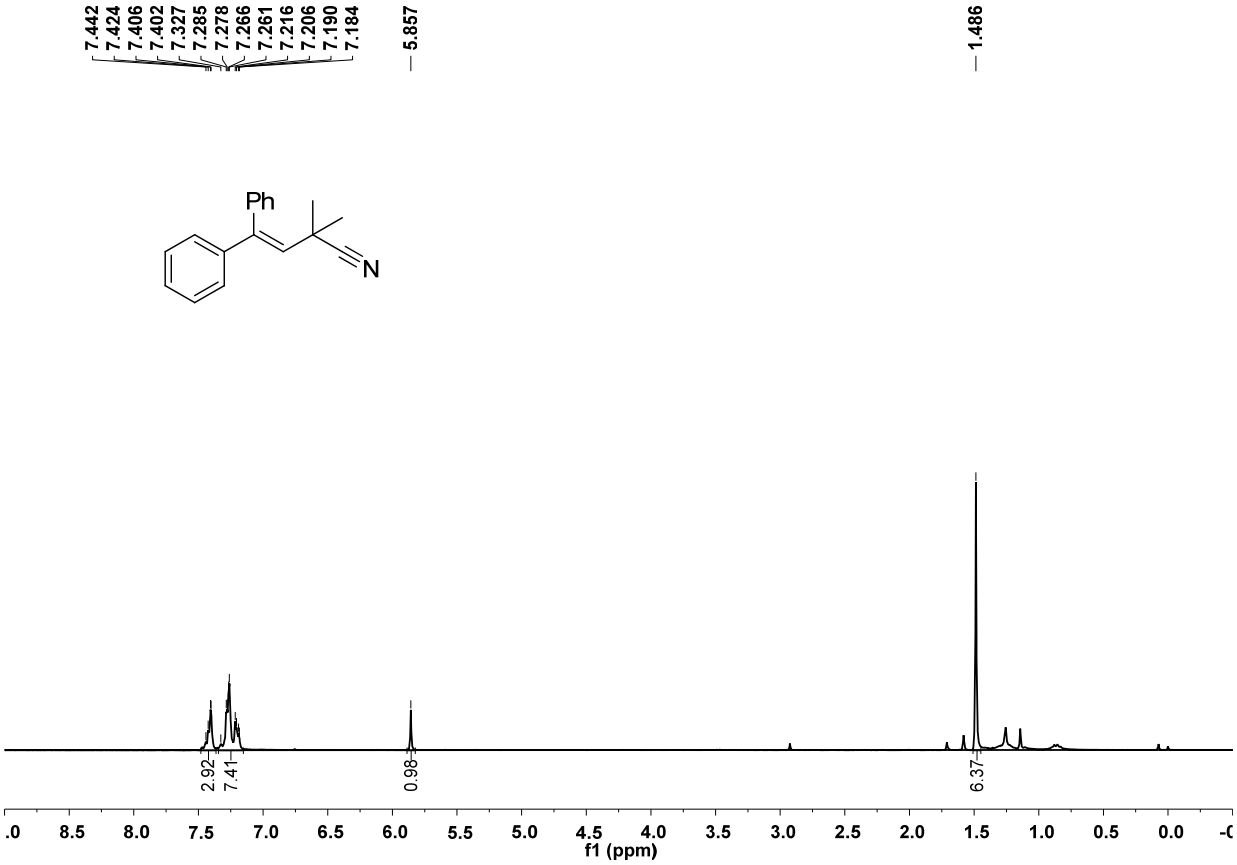

145.487
140.485
138.135
129.195
128.670
128.237
128.069
127.970
127.333
123.290
121.527
77.424
77.000
76.576
25.676
19.335
Ph
N
f1 (ppm)

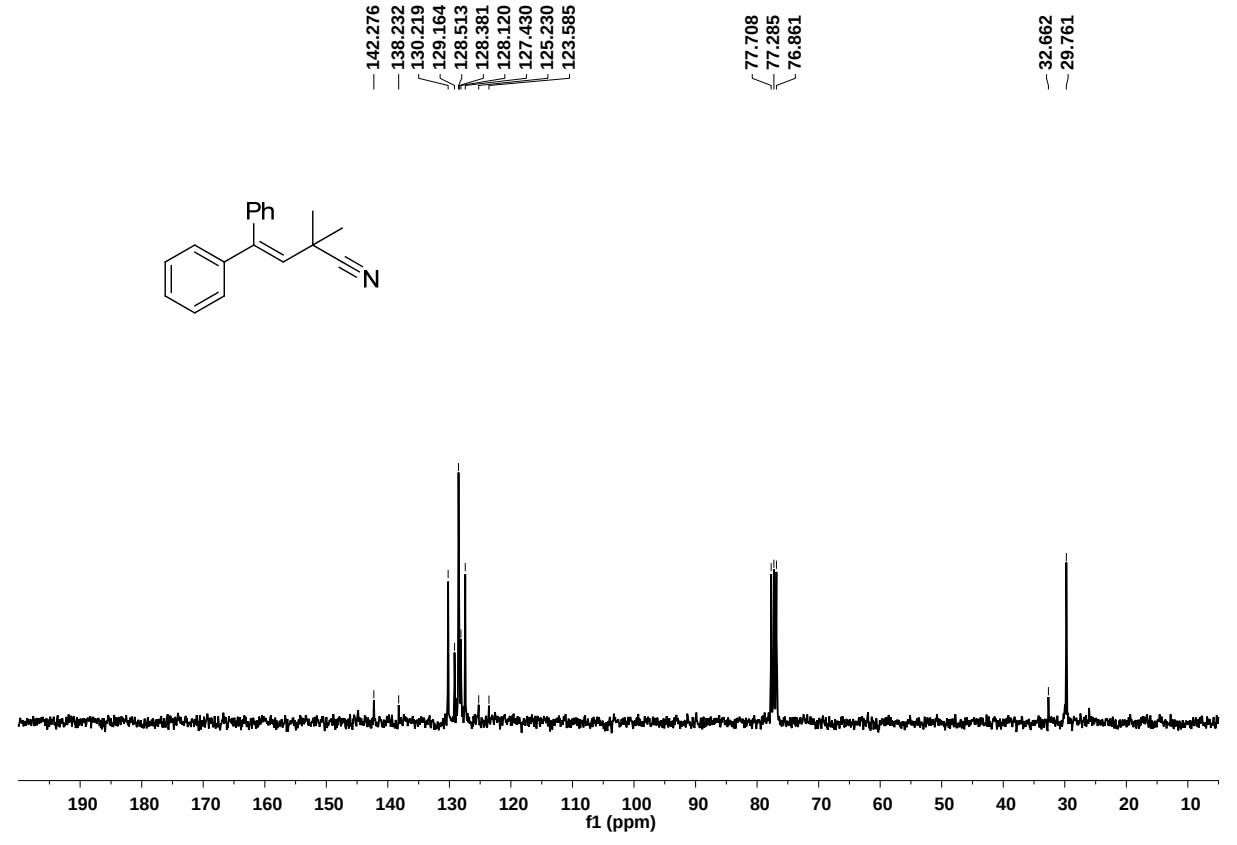
3p


Ph
Ph
CN
7.325
7.301
7.270
7.253
7.213
7.184
7.163
7.138
7.128
7.110
6.035
6.001
4.558
4.525
2.88
12.05
1.04
1.00
ppm


Ph
Ph
CN
145.814
140.231
137.905
134.818
129.340
129.076
128.611
128.230
128.018
127.437
126.861
121.912
119.346
77.423
77.000
76.577
36.820
ppm

3q


7.442
7.424
7.406
7.402
7.327
7.285
7.278
7.266
7.261
7.216
7.206
7.190
7.184
5.857
1.486
Ph
N
2.92
7.41
0.98
6.37
f1 (ppm)


142.276
138.232
130.219
129.164
128.513
128.381
128.120
127.430
125.230
123.585
77.708
77.285
76.861
32.662
29.761
Ph
N
f1 (ppm)