Supporting Information

Easy Access to Enamides: A Mild Nickel-Catalysed Alkene Isomerization of Allylamides

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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 ºC for hours and cooled down under vacuum. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. $Ni(PPh_3)_4$,[1] $NiCl(PPh_3)_3$,[2] (*E*)-*N*-(But-2-enyl)benzamide, 4-Methyl-*N*-(3-methylbut-2-enyl)benzamide and 4-Methyl-*N*-(2-methylallyl)benzamide were all prepared following literature procedures.[3] *N*-allylamides were synthesized from relevant substituent acyl chlorides and prop-2-en-1-amine.

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 ºC). Gas chromatographic analyses were performed on Varian GC 3900 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. High Resolution MS data report were performed on Waters Micromass GCT Premier, ionization mode: EI^+. ^1H and ^{13}C NMR data were recorded with Bruker Advance 400 MHz spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. The chemical shifts (δ) were given in part per million relative to internal tetramethylsilane (0 ppm for ^1H) and $CDCl_3$ (77.3 ppm for ^{13}C) or d_6-DMSO (39.5 ppm for ^{13}C).

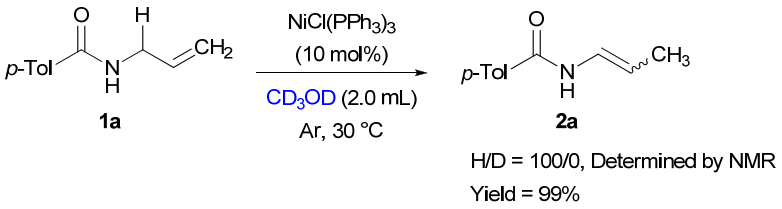
General procedure for the isomerization of *N*-allylamides

N-allylamides (0.25 mmol) was added to a Schlenk tube equipped with a magnetic stirred bar. Then the tube was switched to a glove box, $Ni(PPh_3)_4$ (27.7 mg, 0.025 mmol) was added. The tube was then sealed with septa and taken out of the glove box. Ethanol (2.0 mL) was then injected into the tube by syringe. The reaction was then heated to 30 ºC and stirred for 10 h. Upon completion, the reaction was quenched with ether. The pure product was obtained by flash column chromatography on silica gel (petroleum/ethyl acetate= 20:1).

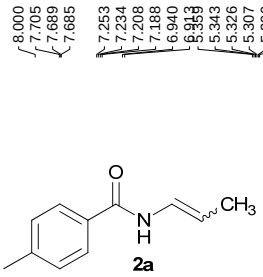
Deuteration experiments

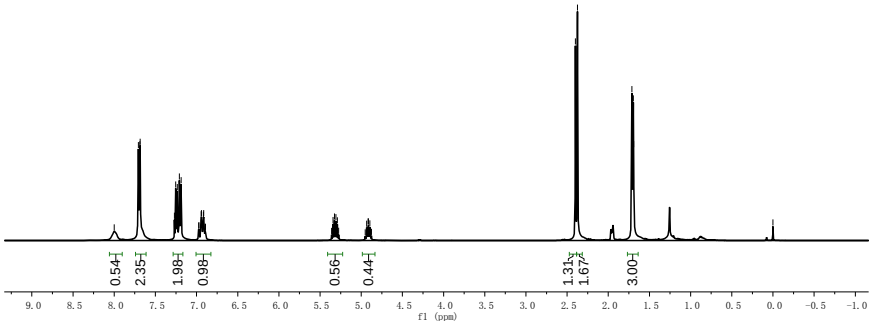
1) Procedure for Eq. (5)

N-allyl-4-methylbenzamide (0.25 mmol) was added to a schlenk tube equipped with a magnetic stirred bar. Then the tube was switched to a glove box, $Ni(PPh_3)_4$ (27.7 mg, 0.025 mmol) was added. The tube was then sealed with septa and taken out of the glove box. CD_3OD (2.0 mL) was then injected into the tube by syringe. The reaction was then heated to 30 ºC and stirred for 10 h. Upon completion, the reaction was quenched with ether. The pure product was obtained by flash column chromatography on silica gel (petroleum/ethyl acetate= 20:1).

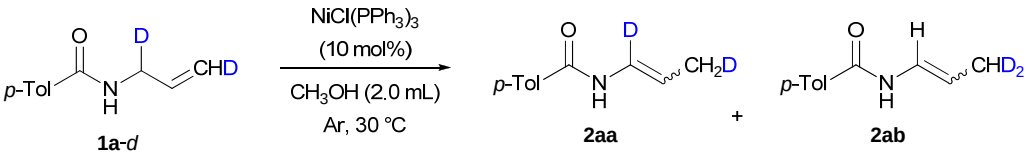


H/D = 100/0, Determined by NMR
Yield = 99%

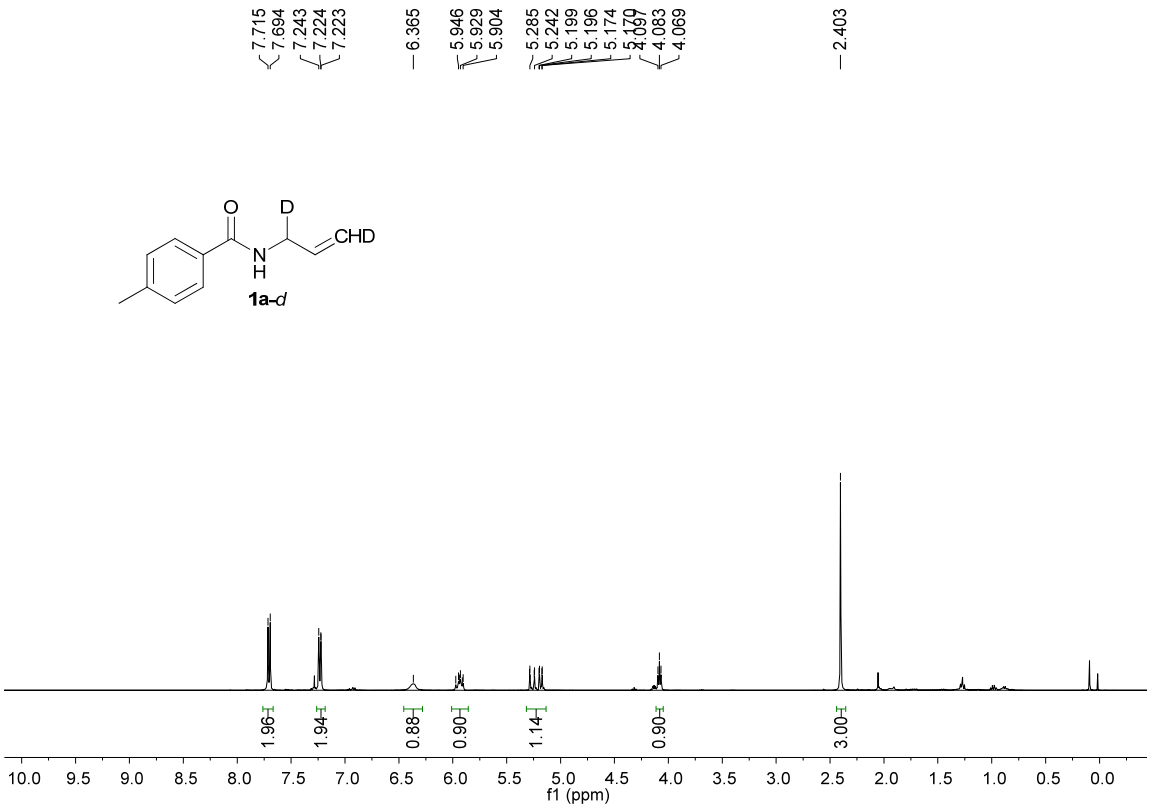


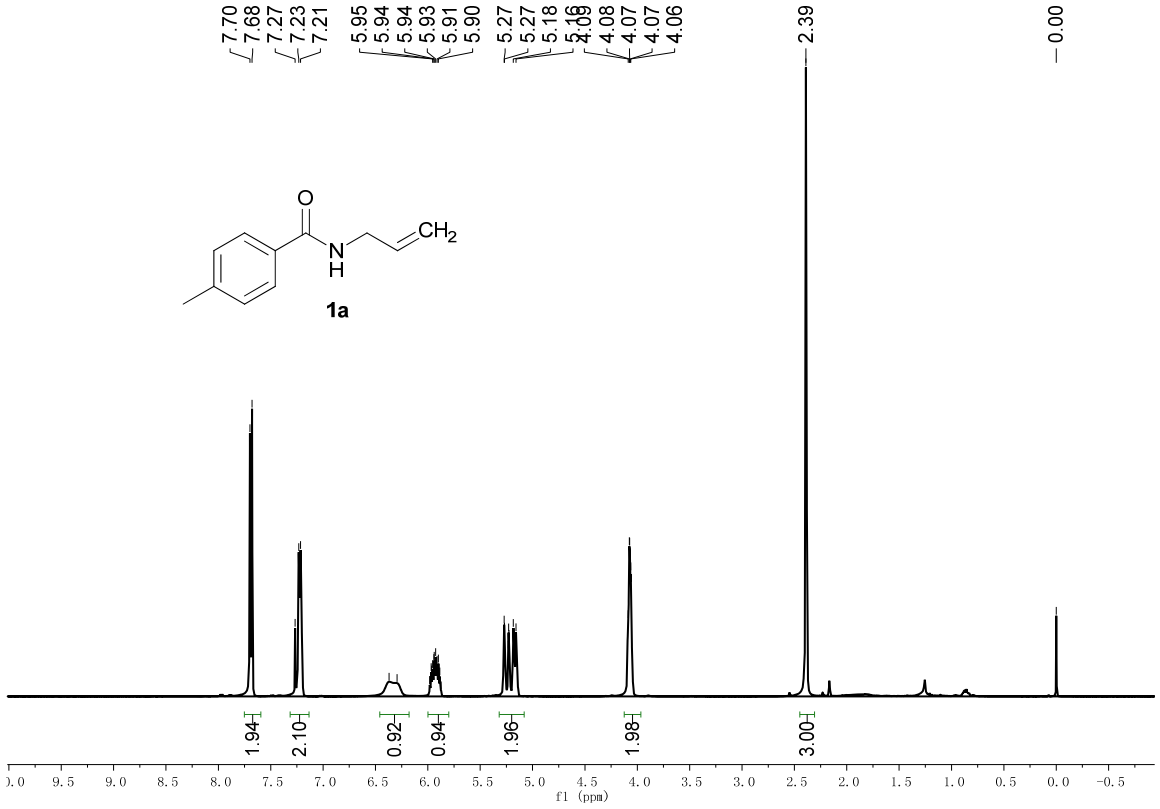


2) Procedure for Eq. (6)

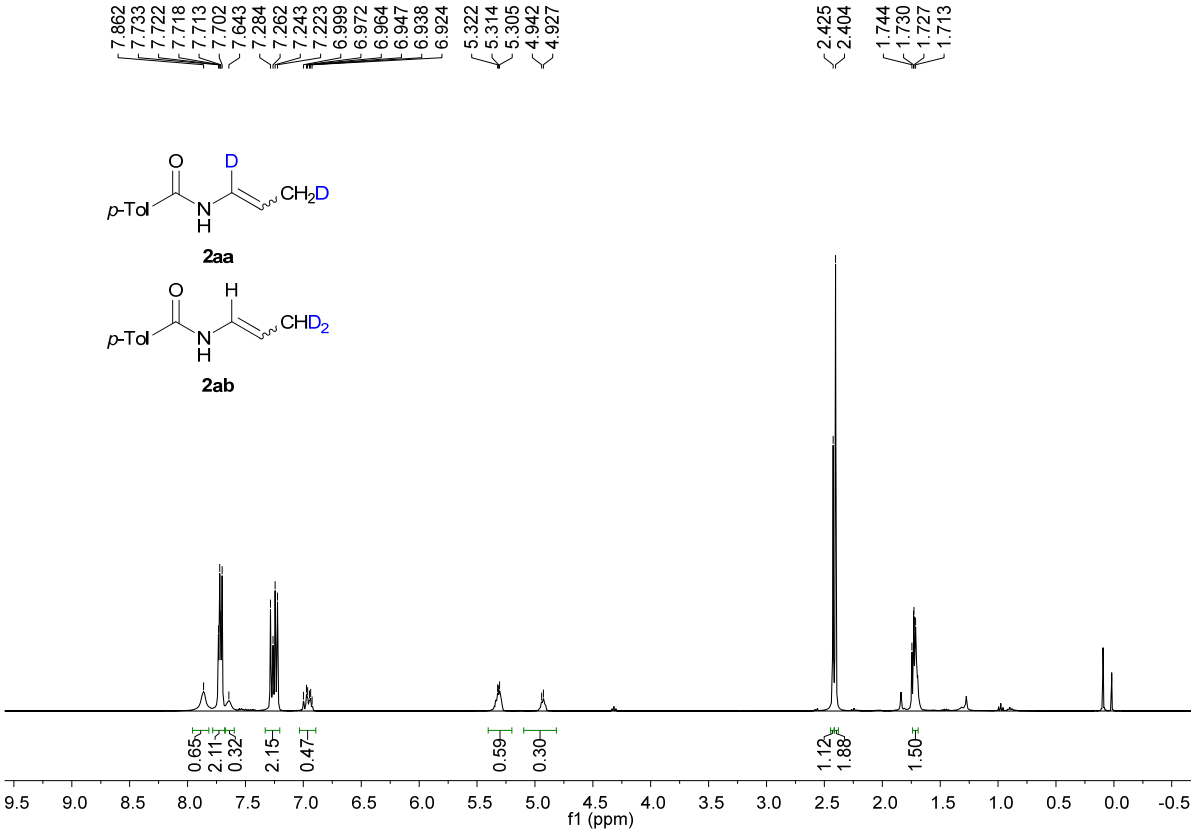


2aa/2ab = 1/1, Determined by NMR
Yield = 99%

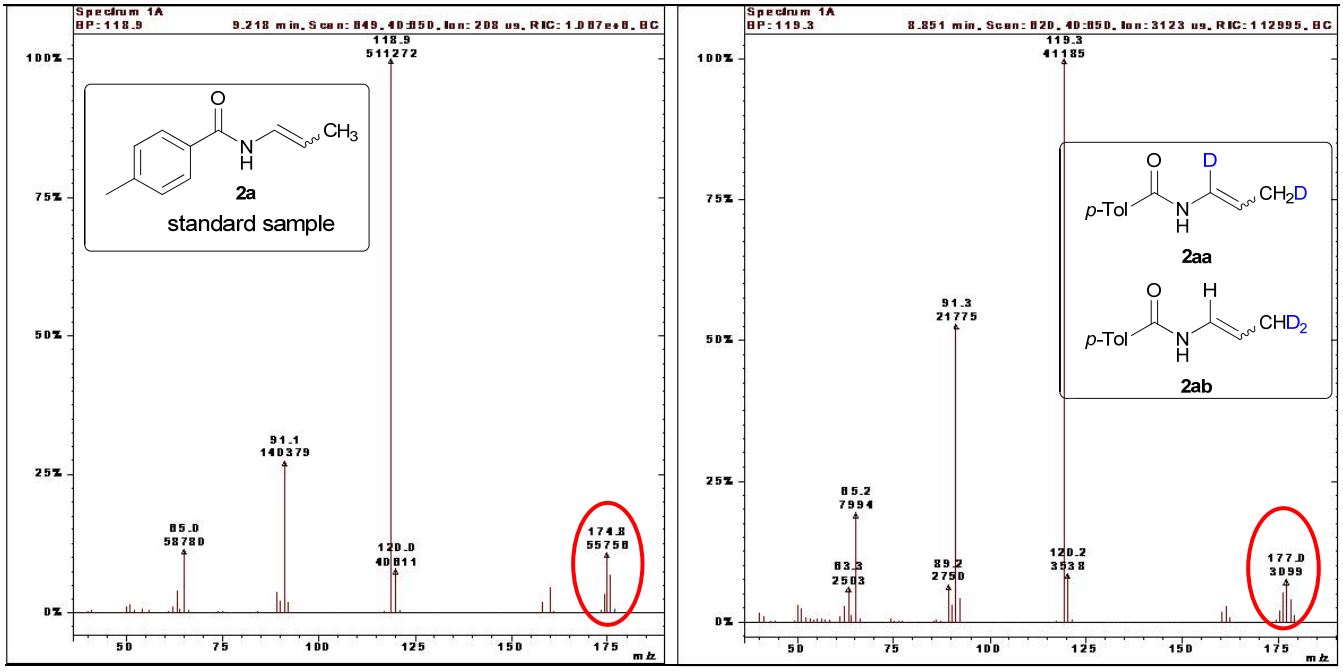




Comparison of **1a**-d with **1a**

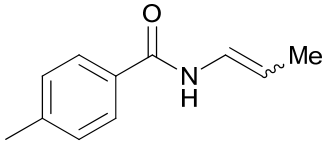


- **MS spectra of isotopic labelling experiments:**

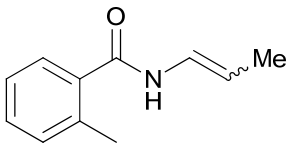


Comparison of **2a** with **2aa** and **2ab**

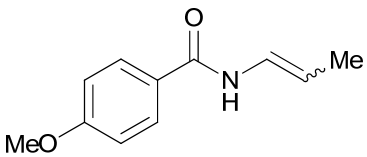
Detailed descriptions for products:



(*E, Z*)-4-Methyl-*N*-(prop-1-enyl)benzamide (**2a**): ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.00 (br, 0.54H), 7.79-7.57 (m, 2.5H), 7.29 - 7.17 (m, 2H), 7.01-6.83 (m, 1H), 5.32 (*trans*, dq, *J* = 13.6 Hz, 6.8 Hz, 0.56H), 4.91 (*cis*, dq, J = 8.4 Hz, 7.2 Hz, m, 0.44H), 2.40 (s, 1.31H), 2.37 (s, 1.67H), 1.74 - 1.66 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 164.34, 164.22, 142.41, 142.10, 129.31, 129.16, 127.01, 126.97, 123.67, 108.50, 105.92, 21.42, 21.39, 14.92, 10.92 ppm. HRMS (EI) calcd for $C_{11}H_{13}NO$ $[M]^+$: 175.0997; found: 175.0995.

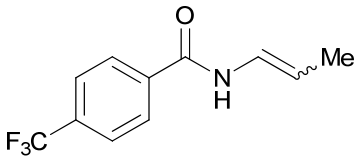


(*E, Z*)-*N*-Propenyl-o-toluamid((**2b**): ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.31 (br, 0.57H), 7.35-7.03 (m, 4.43H), 6.88-6.71 (m, 1H), 5.17 (*trans*, dq, *J* = 13.6 Hz, 6.8 Hz, 0.60H), 4.83 (*cis*, dq, *J* = 8.8 Hz, 7.1 Hz, 0.40H), 2.37 (s, 1.23H), 2.34 (s, 1.77H), 1.63 (dd, *J* = 6.8 Hz, 1.6 Hz, 1.78H), 1.56 (dd, *J* = 6.8 Hz, 1.6 Hz, 1.22H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.79, 166.71, 136.49, 136.42, 135.44, 131.16, 131.01, 130.27, 130.05, 126.70, 126.61, 125.77, 125.62, 123.31, 121.91, 108.67, 106.13, 19.82, 19.73, 14.87, 10.92 ppm. HRMS (EI) calcd for $C_{11}H_{13}NO$ $[M]^+$: 175.0997; found: 175.0998.

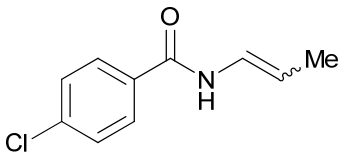


(*E, Z*)-4-Methoxy-*N*-(prop-1-enyl)benzamide (**2c**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 0.49H), 7.85-7.70 (m ,2H), 7.64 (d, *J* = 8.0 Hz, 0.45H), 7.00-6.86 (m, 3H), 5.31 (*trans*, dq, J = 13.6 Hz,

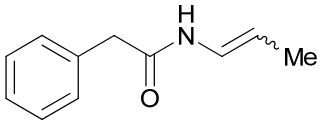
6.8 Hz, 0.52H), 4.90 (*cis*, dq, *J* = 8.4 Hz, 7.2 Hz, 0.47H), 3.85 (s, 1.41H), 3.83 (s, 1.57H), 1.78-1.63 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.90, 163.80, 162.46, 162.27, 128.88, 128.85, 126.03, 126.01, 123.76, 122.30, 113.86, 113.70, 108.21, 105.64, 55.37, 55.32, 14.93, 10.91 ppm. HRMS (EI) calcd for $C_{11}H_{13}NO_2$ $[M]^+$: 191.0946; found: 191.0948.



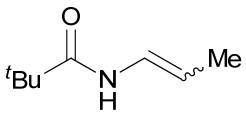
(*E, Z*)-*N*-(Prop-1-enyl)-4-(trifluoromethyl)benzamide (**2d**): ^{1}H NMR (400 MHz, d_6-DMSO) δ 10.37 (d, *J* = 9.6 Hz, 0.46H), 9.84 (d, *J* = 9.6 Hz, 0.53H), 8.10-8.06 (m, 2H), 7.87-7.84 (m, 2H), 6.88-6.71 (m, 1H), 5.50 (*trans*, dq, *J* = 13.6 Hz, 6.8 Hz, 0.47H), 4.82 (*cis*, dq, *J* = 8.8 Hz, 7.2 Hz, 0.53H), 1.74 (dd, *J* = 7.0 Hz, 1.6 Hz, 1.59H), 1.69 (dd, *J* = 6.8 Hz, 1.6 Hz, 1.40H) ppm; ^{13}C NMR (100 MHz, d_6-DMSO) δ 163.98, 162.27, 128.80, 128.38, 131.40 (d, *J* = 31.8 Hz), 131.38 (d, *J* = 31.6 Hz), 128.80, 128.38, 125.35 (d, *J* = 3.7 Hz), 125.16 (d, *J* = 3.7 Hz), 124.24, 122.58, 15.01, 11.68 ppm. HRMS (EI) calcd for $C_{11}H_{10}F_3NO$ $[M]^+$: 229.0714; found: 229.0711.



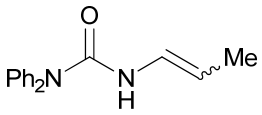
(*E, Z*)-4-Chloro-*N*-(prop-1-enyl)benzamide (**2e**): ^{1}H NMR (400 MHz,d_6-DMSO) δ 10.22 (d, *J* =9.6 Hz, 0.73H), 9.66 (d, *J* = 9.2 Hz, 0.22H), 7.92 (d, *J* = 8.4 Hz, 2H), 7.56 (d, *J* = 8.4 Hz, 2H), 6.88-6.77 (m, 0.77H), 6.76-6.67 (m, 0.23H), 5.45 (*trans*, dq, *J* = 13.6 Hz, 6.8 Hz, 0.78H), 4.92-4.84 (*cis*, m, 0.24H), 1.72 (dd, *J* = 7.2 Hz, 1.2 Hz, 0.70H), 1.70-1.65 (m, 2.30H) ppm; ^{13}C NMR (100 MHz, d_6-DMSO) δ 163.88, 162.33, 136.36, 132.42, 129.81, 129.37, 128.43, 128.27, 124.32, 122.68, 108.34, 107.84, 15.03, 11.67 ppm. HRMS (EI) calcd for $C_{10}H_{10}ClNO$ $[M]^+$: 195.0451; found: 195.0450.



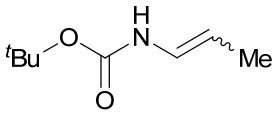
(*E, Z*)-2-Phenyl-N-(prop-1-enyl)acetamide (**2f**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.12 (m, 5.6H), 7.06 (br, 0.4H), 6.71-6.52 (m, 1H), 4.96 (*trans*, dq, *J* = 13.6 Hz, 6.8 Hz, 0.61H), 4.74-4.63 (cis, m, 0.39H), 3.57 (s, 0.78H), 3.49 (s, 1.22H), 1.54 (dd, *J* = 0.8, 6.8 Hz, 1.86H), 1.31 (dd, *J* = 1.6, 6.8 Hz, 1.23H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.10, 167.96, 134.35, 134.25, 129.37, 129.31, 129.04, 128.93, 127.51, 127.33, 123.04, 121.61, 108.37, 105.85, 43.52, 43.40, 14.69, 10.45 ppm. HRMS (EI) calcd for $C_{11}H_{13}NO$ $[M]^+$: 175.0997; found: 175.0995.



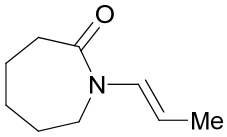
(*E, Z*)-*N*-(Prop-1-enyl)pivalamide (**2g**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (br, 1H), 6.81-6.70 (m, 1H), 5.17 (*trans*, dq, *J* = 13.6 Hz, 6.8 Hz, 0.90H), 4.82 (*cis*, dq, *J* = 8.6 Hz, 7.0 Hz, 0.10H), 1.70-1.66 (m, 2.68H), 1.64-1.61 (m, 0.32H), 1.22 (s, 9H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.29, 123.59, 122.17, 107.54, 104.96, 38.51, 27.36, 14.77 ppm. HRMS (EI) calcd for $C_8H_{15}NO$ $[M]^+$: 141.1154; found: 141.1155.



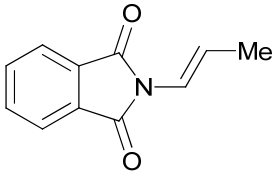
(*E, Z*)-*N,N*-Diphenyl-*N'*-(prop-1-enyl)urea (**2h**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.20 (m, 10H), 6.75-6.64 (m, 1H), 6.28-6.03 (m,1H), 4.80 (*trans*, dq, *J* = 13.2 Hz, 6.4 Hz, 0.39H), 4.62 (*cis*, dq, *J* = 7.1 Hz, 1.2 Hz, 0.61H); 1.62 (dd, *J* = 6.8 Hz, 1.2 Hz, 1.15H), 1.28-1.25 (m, 1.85H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.88, 152.72, 142.17, 142.06, 129.47, 129.44, 127.33, 127.27, 126.59, 126.45, 124.40, 123.21, 104.85, 102.09, 14.72, 10.19 ppm. HRMS (EI) calcd for $C_{16}H_{16}N_2O$ $[M]^+$: 252.1263; found: 252.1268.



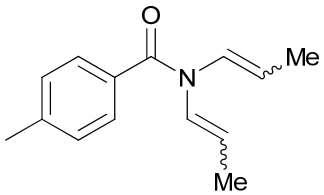
(*E, Z*)-tert-Butyl prop-1-enylcarbamate (**2i**): 1H NMR (400 MHz, CDCl3) δ 6.50-6.35 (m, 1H), 6.20 (br, 1H), 4.94 (*trans*, dq, J = 12.8 Hz, 6.4 Hz, 0.45H), 4.69-4.57 (*cis*, m, 0.55H); 1.63 (dd, *J* = 6.8 Hz, 1.2 Hz, 1.31H), 1.56 (dd, *J* = 7.0 Hz, 1.6 Hz, 1.72H), 1.48 (s, 5.69H), 1.45 (s, 3.30H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.87, 124.31, 123.15, 104.41, 101.94, 80.33, 80.04, 28.31, 14.74, 10.53 ppm. HRMS (EI) calcd for $C_8H_{15}NO_2$ $[M]^+$: 157.1103; found: 157.1099.



(*E*)-1-(Prop-1-enyl)azepan-2-one (**2j**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16-7.06 (m, 1H), 5.05 (dq, *J* = 13.2 Hz, 6.8 Hz, 1H), 3.61-3.51 (m, 2H), 2.65-2.55 (m, 2H), 1.77-1.70 (m, 9H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.85, 127.47, 105.87, 45.58, 37.14, 29.46, 27.37, 23.42, 15.28 ppm. HRMS (EI) calcd for $C_9H_{15}NO$ $[M]^+$: 153.1154; found: 153.1149.

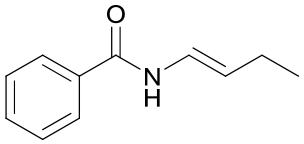


(*E*)-2-Propenyl-isoindole-1,3-dione (**2k**)[4]: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.80 (m, 2H), 7.78-7.65 (m, 2H), 6.66-6.50 (m, 2H), 1.88-1.82 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.67, 134.22, 131.67, 123.42, 118.30, 118.06, 16.28 ppm.

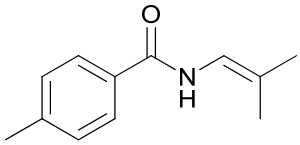


4-Methyl-*N,N*-di(prop-1-enyl)benzamide (**2l**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.37 (m, 2H),

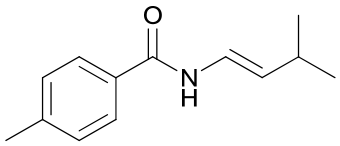
7.18-7.10 (m, 2H), 7.07-6.93 (m, 0.31H), 6.47 (d, *J* = 12.4 Hz, 0.62H), 6.03-5.95 (m, 0.59H), 5.49-5.37 (m, 0.53H), 5.26 (dq, *J* = 13.6 Hz, 6.8 Hz, 0.74H), 5.13 (dq, *J* = 13.6 Hz, 6.8 Hz, 0.67H), 2.40-2.32 (m, 3H), 1.84-1.1.78 (m, 0.56H), 1.75-1.71 (m, 1.71H), 1.68-1.63 (m, 2H), 1.40-1.28 (m, 1.78H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.86, 168.80, 140.56, 140.24, 132.93, 132.68, 128.96, 128.47, 128.46, 128.32, 128.00, 125.67, 109.60, 21.43, 21.39, 15.17, 15.14, 12.25 ppm. HRMS (EI) calcd for $C_{14}H_{17}NO$ $[M]^+$: 215.1310; found: 215.1311.



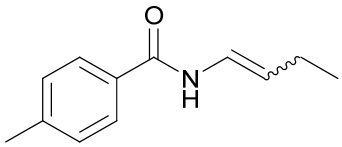
(*E*)-*N*-(But-1-enyl)benzamide (**4a**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (br, 1H), 7.86-7.78 (m, 2H), 7.51-7.42 (m, 1H), 7.40-7.32 (m, 2H), 7.01-6.85 (m, 1H), 5.43 (td, J = 14.0 Hz, 6.8 Hz, 1H), 2.13-1.98 (m, 2H), 1.00 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.29, 133.87, 131.75, 128.65, 126.95, 122.14, 115.93, 23.01, 14.20 ppm. HRMS (EI) calcd for $C_{11}H_{13}NO$ $[M]^+$: 175.0997; found: 175.1002.



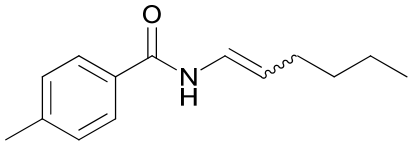
4-Methyl-*N*-(2-methylprop-1-enyl)benzamide (**4b**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 6.81-6.65 (m, 1H), 2.40 (s, 3H), 1.77 (s, 3H), 1.71 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.04, 142.10, 131.31, 129.27, 126.88, 117.33, 115.96, 22.51, 21.41, 16.56 ppm. HRMS (EI) calcd for $C_{12}H_{15}NO$ $[M]^+$: 189.1154; found: 189.1152.



(*E*)-4-Methyl-*N*-(3-methylbut-1-enyl)benzamide (**4c**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (br, 1H), 7.62 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 6.94-6.78 (m, 1H), 5.21 (dd, *J* = 14.4 Hz, 6.8 Hz, 1H), 2.42-2.23 (m, 4H), 0.96 (d, *J* = 6.8 Hz, 6H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.33, 142.21, 131.00, 129.27, 126.97, 121.19, 120.85, 29.01, 22.86, 21.44 ppm. HRMS (EI) calcd for $C_{13}H_{17}NO$ $[M]^+$: 203.1310; found: 203.1308.



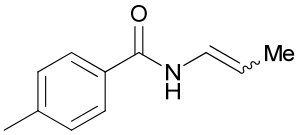
(*E, Z*)-*N*-(But-1-enyl)-4-methylbenzamide (**6a**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (br, 0.87H), 7.73 (br, 0.12H), 7.70-7.57 (m, 2H), 7.16-7.01 (m,2H), 6.99-6.89 (m, 1H), 5.33 (*trans*, td, *J* = 14.0 Hz, 6.4 Hz, 0.88H), 4.74 (*cis*, td, *J* = 7.6 Hz, 0.12H), 2.30-2.24 (m, 3H), 2.06-1.90 (m, 2H), 1.01-0.86 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.18, 142.28, 130.99, 129.40, 129.32, 126.95, 122.21, 115.54, 23.02, 21.47, 14.25 ppm. HRMS (EI) calcd for $C_{12}H_{15}NO$ $[M]^+$: 189.1154; found: 189.1153.

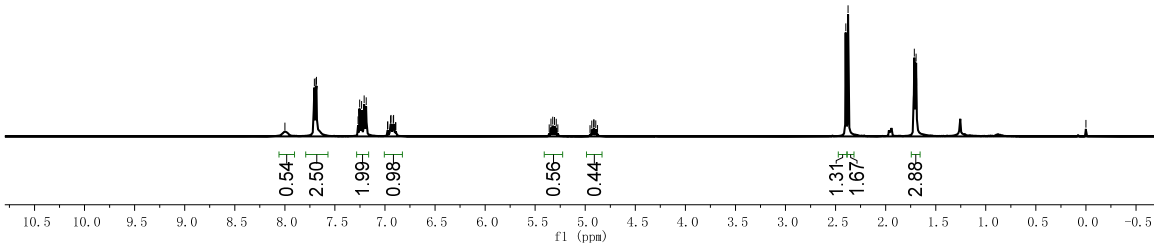


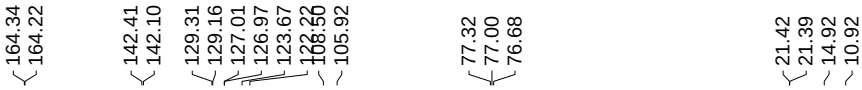
(*E, Z*)-*N*-(Hex-1-enyl)-4-methylbenzamide (**8a**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74-7.67 (m, 2H), 7.64 (d, *J* = 10.0 Hz, 0.73H), 7.29-7.21 (m, 2H), 7.02-6.86 (m, 1H), 5.28 (*trans*, td, *J* = 14.4 Hz, 7.2 Hz, 0.86H), 4.85 (*cis*, td, *J* = 7.6 Hz, 0.14H), 2.43-2.38 (m, 3H), 2.13-2.03 (m, 2H), 1.45-1.30 (m, 4H), 0.95-0.85 (m, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ164.13, 142.26, 130.97, 129.30, 126.95, 122.79, 121.17, 113.96, 111.98, 32.03, 31.45, 29.68, 29.44, 22.32, 22.10, 21.46, 13.69 ppm. HRMS (EI) calcd for $C_{14}H_{19}NO$ $[M]^+$: 217.1467; found: 217.1471.

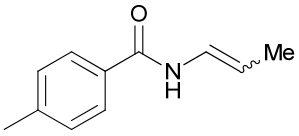
Copies of products ^{1}H and ^{13}C NMR

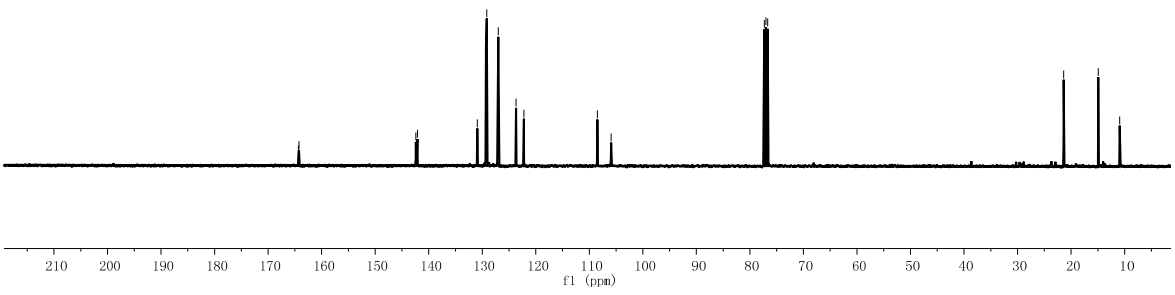
2a



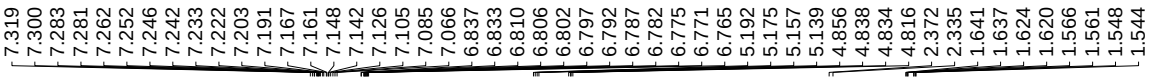








2b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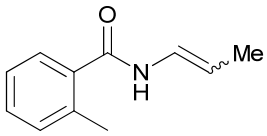
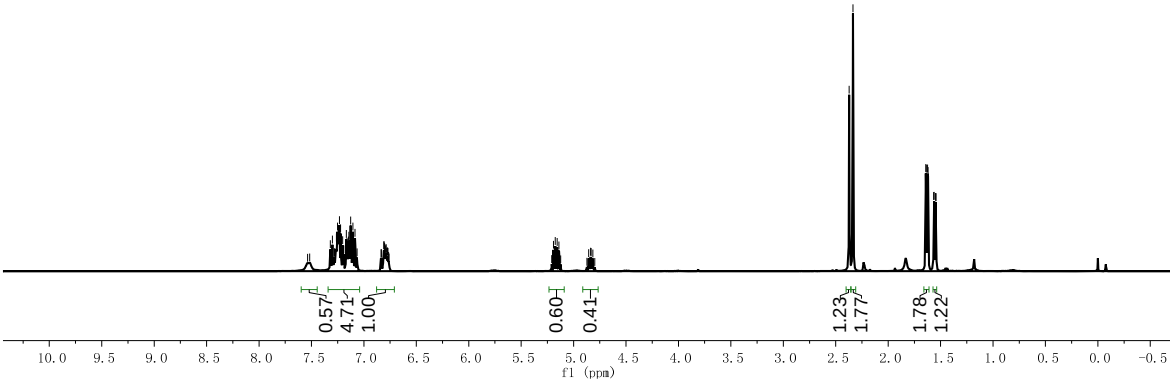
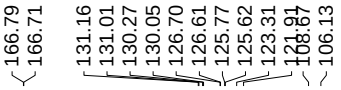
7.283
7.281
7.262
7.252
7.246
7.242
7.233
7.222
7.203
7.191
7.167
7.161
7.148
7.142
7.126
7.105
7.085
7.066
6.837
6.833
6.810
6.806
6.802
6.797
6.792
6.787
6.782
6.775
6.771
6.765
5.192
5.175
5.157
5.139
4.856
4.838
4.834
4.816
2.372
2.335
1.641
1.637
1.624
1.620
1.566
1.561
1.548
1.544


O
N
H
Me


0.57
4.71
1.00
0.60
0.41
1.23
1.77
1.78
1.22
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)


166.79
166.71
131.16
131.01
130.27
130.05
126.70
126.61
125.77
125.62
123.31
121.91
108.87
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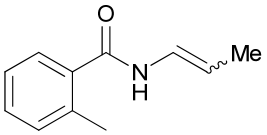
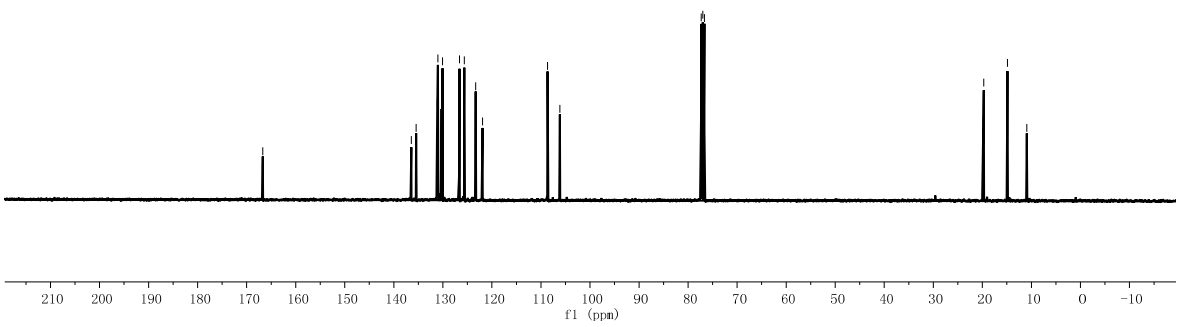
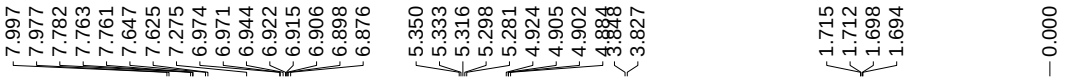
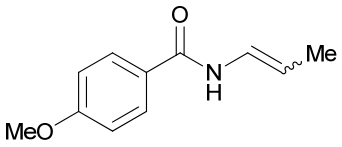
77.00
76.68
19.82
19.73
14.87
10.92

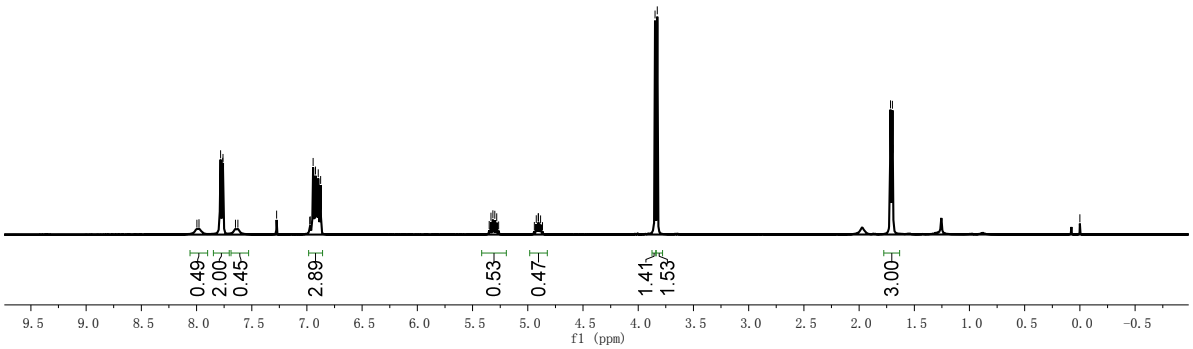

O
N
H
Me

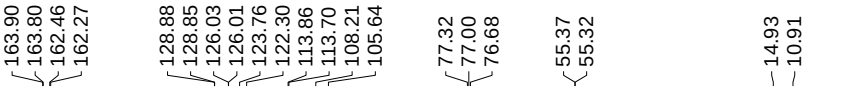

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

2c


7.997
7.977
7.782
7.763
7.761
7.647
7.625
7.275
6.974
6.971
6.944
6.922
6.915
6.906
6.898
6.876
5.350
5.333
5.316
5.298
5.281
4.924
4.905
4.902
4.884
3.848
3.827
1.715
1.712
1.698
1.694
0.000


O
N
H
Me
MeO


0.49
2.00
0.45
2.89
0.53
0.47
1.41
1.53
3.00
f1 (ppm)


163.90
163.80
162.46
162.27
128.88
128.85
126.03
126.01
123.76
122.30
113.86
113.70
108.21
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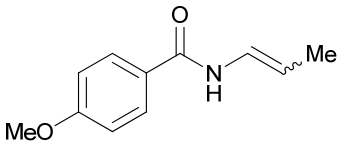
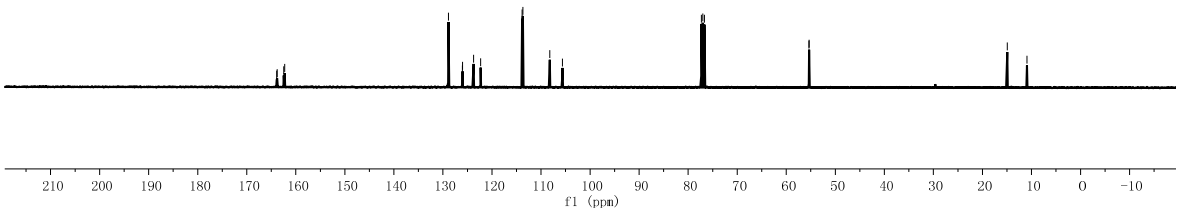
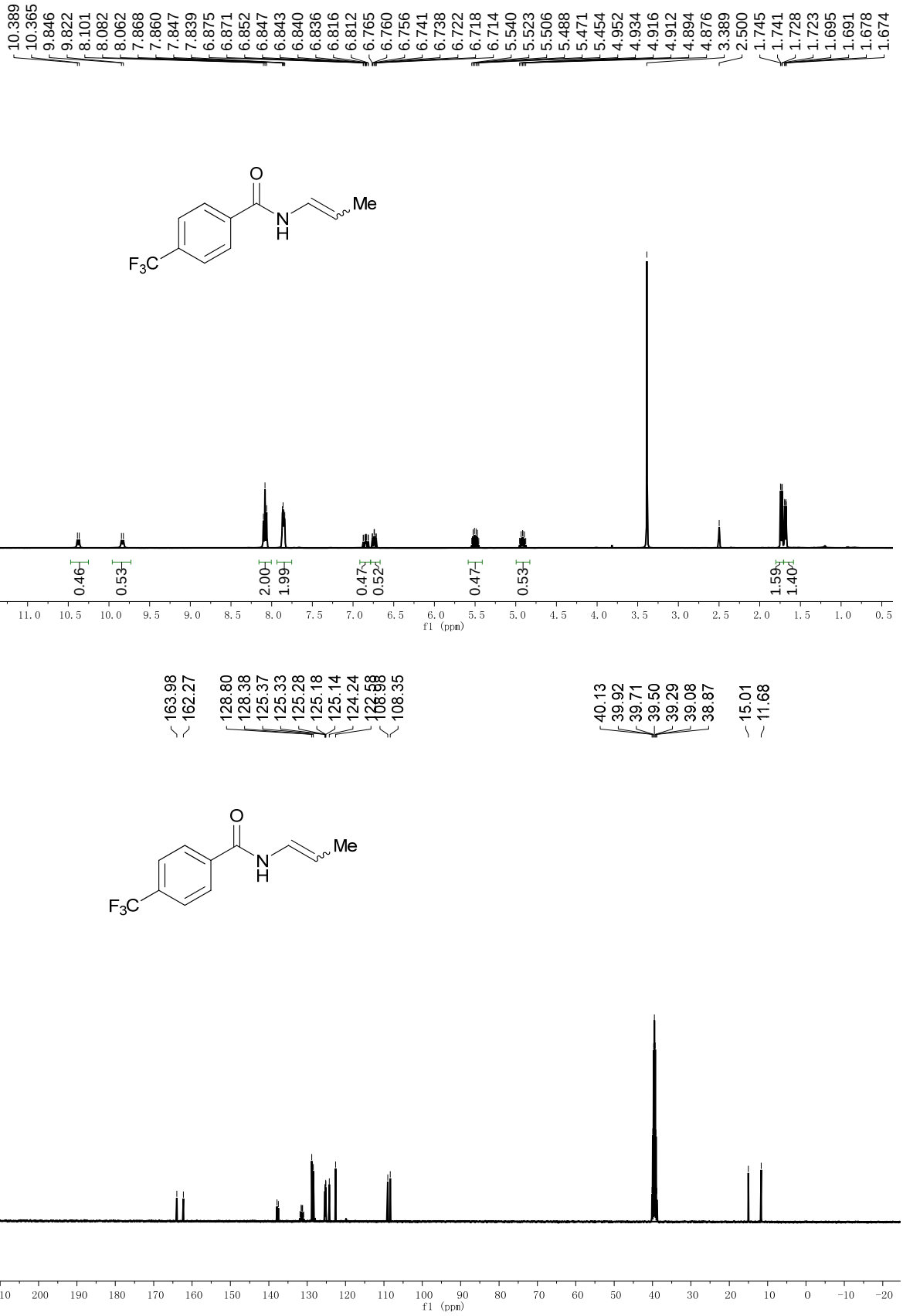
77.00
76.68
55.37
55.32
14.93
10.91


O
N
H
Me
MeO


f1 (ppm)

2d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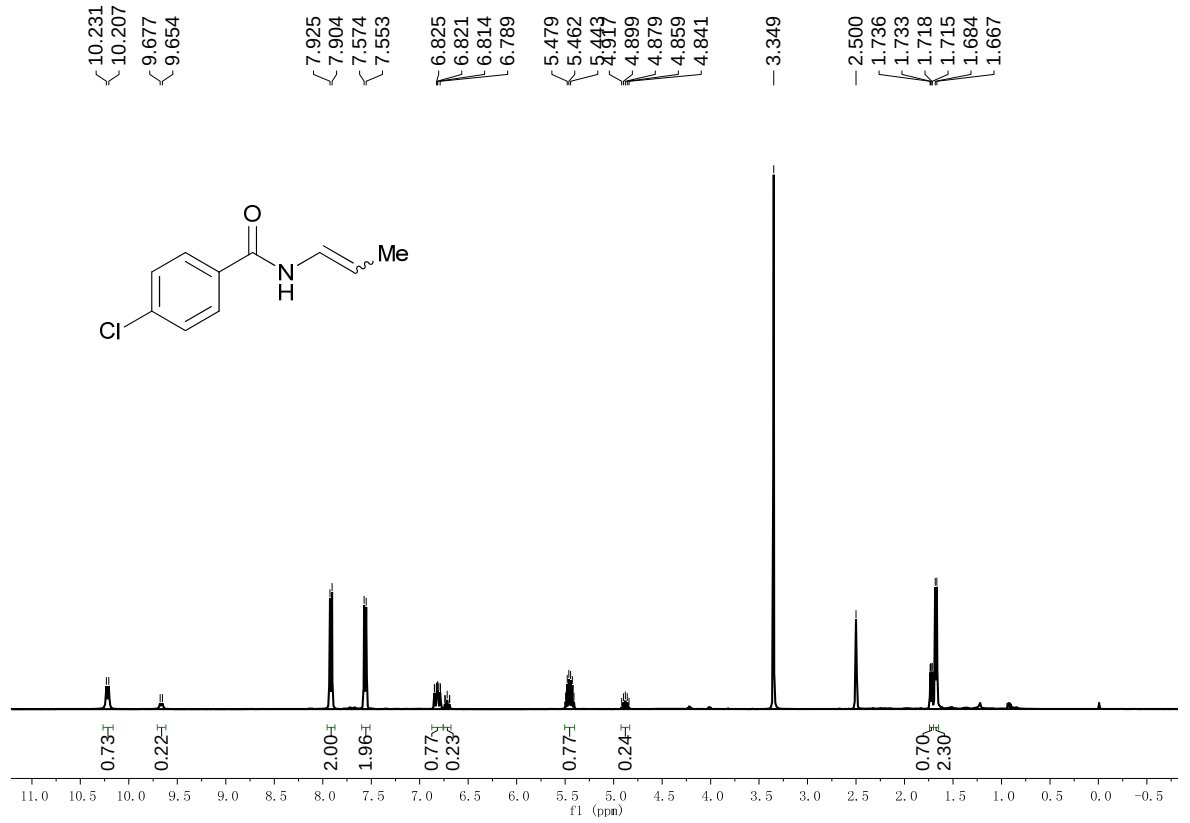
10.365
9.846
9.822
8.101
8.082
8.062
7.868
7.860
7.847
7.839
6.875
6.871
6.852
6.847
6.843
6.840
6.836
6.816
6.812
6.765
6.760
6.756
6.741
6.738
6.722
6.718
6.714
5.540
5.523
5.506
5.488
5.471
5.454
4.952
4.934
4.916
4.912
4.894
4.876
3.389
2.500
1.745
1.741
1.728
1.723
1.695
1.691
1.678
1.674
O
N
H
Me
F3C
0.46
0.53
2.00
1.99
0.47
0.52
0.47
0.53
1.59
1.40
f1 (ppm)
163.98
162.27
128.80
128.38
125.37
125.33
125.28
125.18
125.14
124.24
108.35
40.13
39.92
39.71
39.50
39.29
39.08
38.87
15.01
11.68
f1 (ppm)

2e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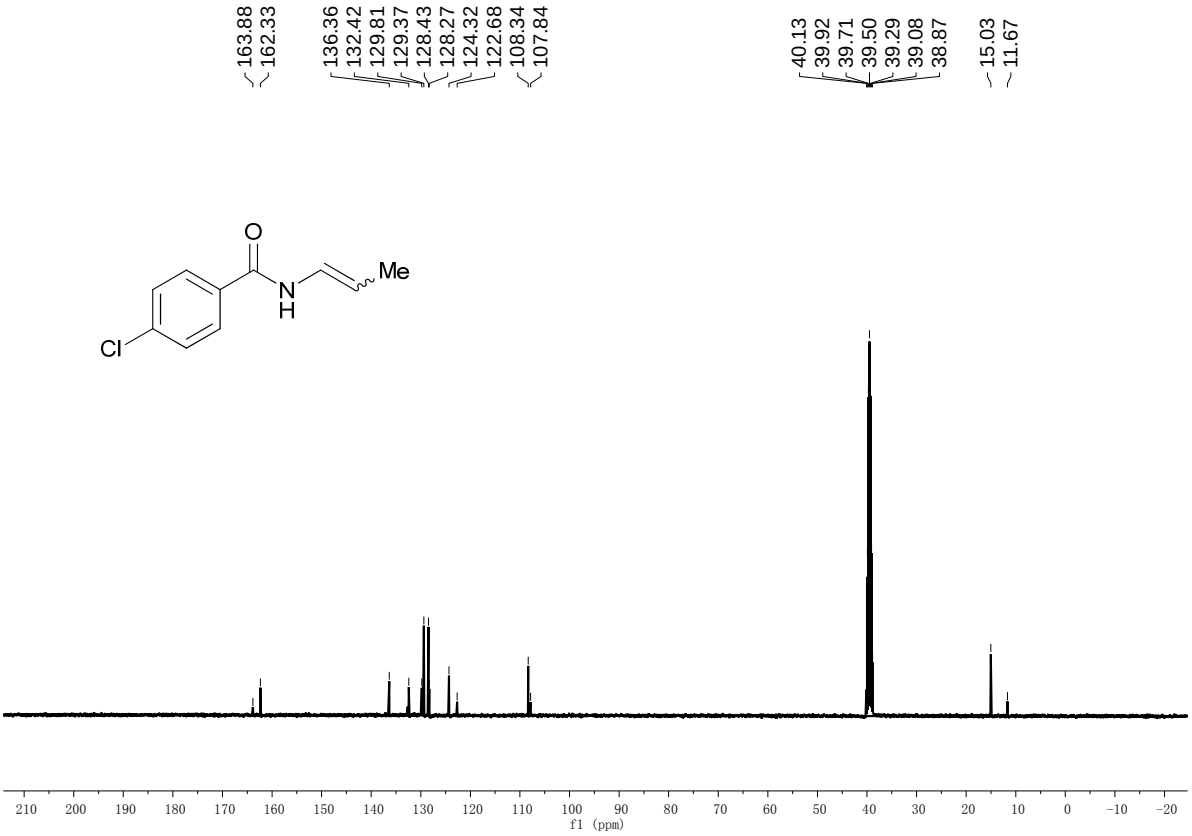
10.207
9.677
9.654
7.925
7.904
7.574
7.553
6.825
6.821
6.814
6.789
5.479
5.462
5.443
5.417
4.899
4.879
4.859
4.841
3.349
2.500
1.736
1.733
1.718
1.715
1.684
1.667
O
N
H
Me
Cl
0.73
0.22
2.00
1.96
0.77
0.23
0.77
0.24
0.70
2.30
f1 (ppm)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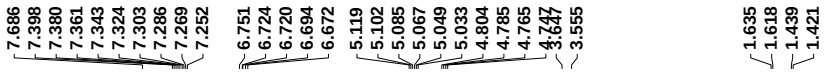
162.33
136.36
132.42
129.81
129.37
128.43
128.27
124.32
122.68
108.34
107.84
40.13
39.92
39.71
39.50
39.29
39.08
38.87
15.03
11.67
O
N
H
Me
Cl
f1 (ppm)

2f


7.686
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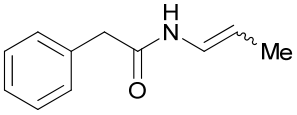
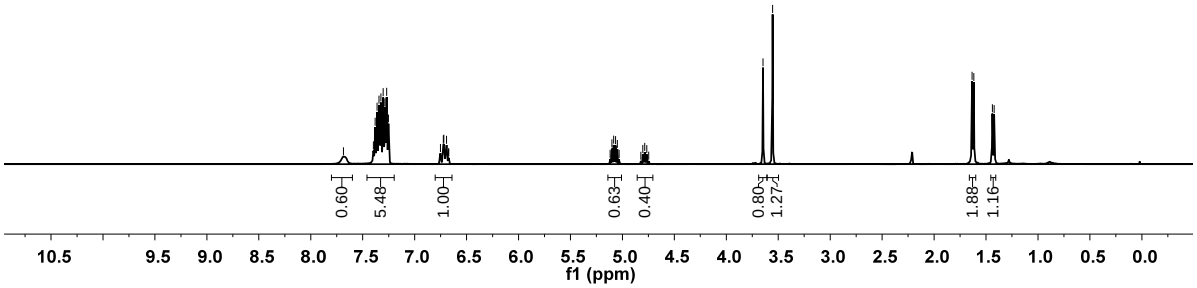
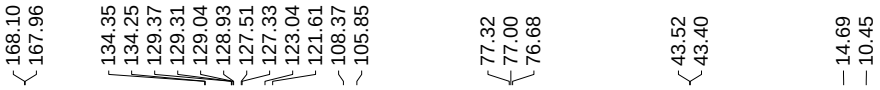
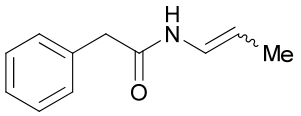
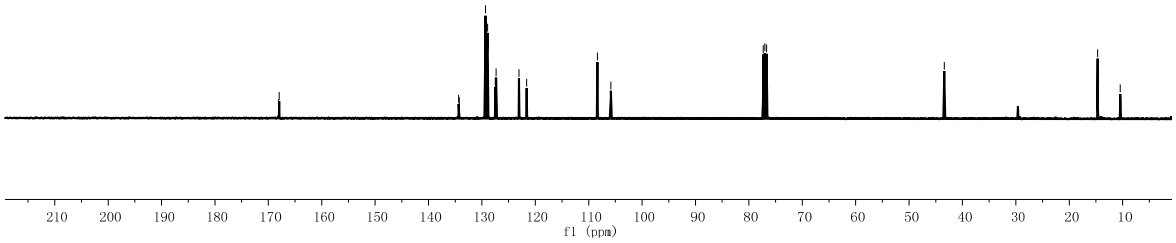
7.380
7.361
7.343
7.324
7.303
7.286
7.269
7.252
6.751
6.724
6.720
6.694
6.672
5.119
5.102
5.085
5.067
5.049
5.033
4.804
4.785
4.765
3.555
1.635
1.618
1.439
1.421


H
N
Me
O

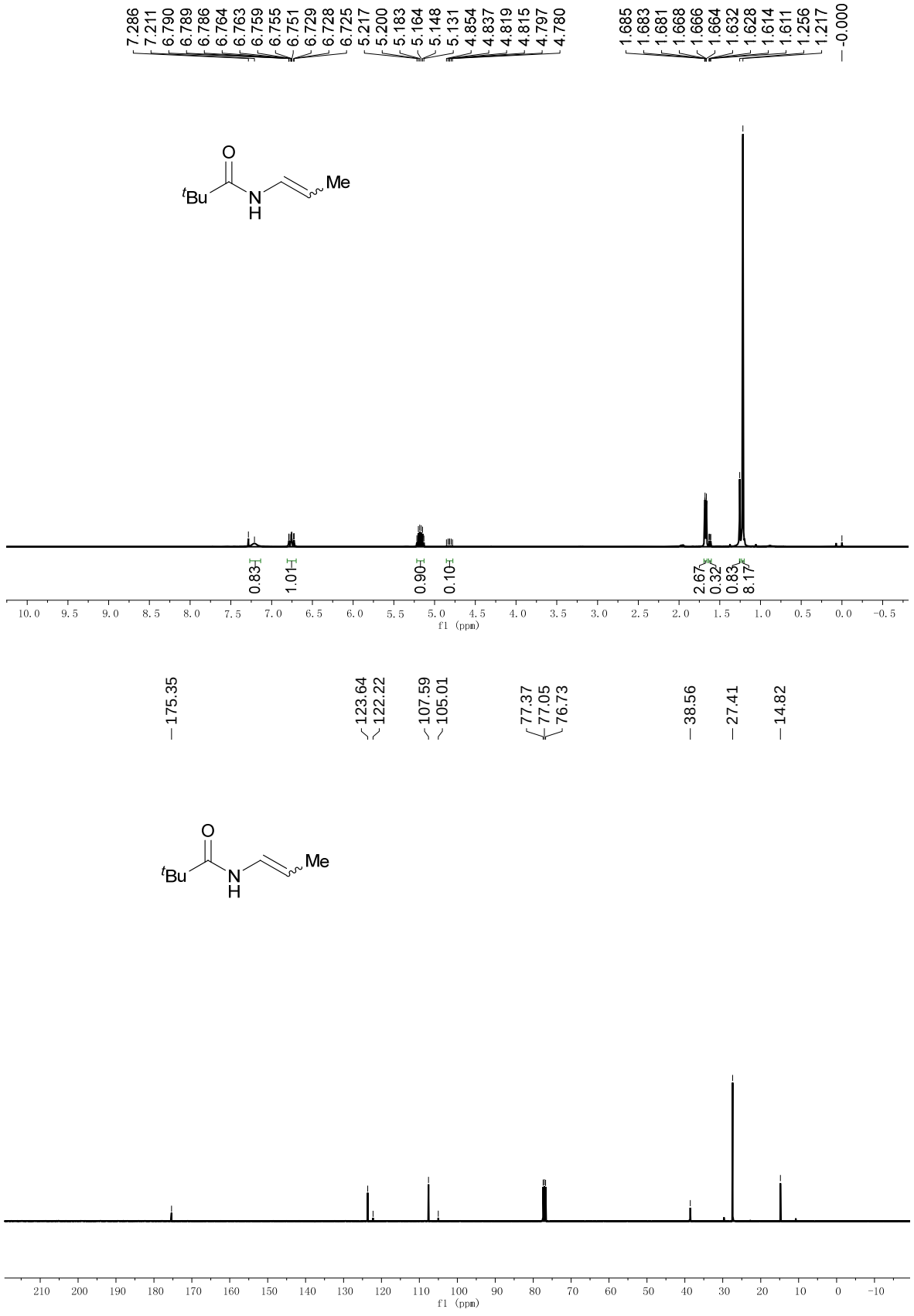

0.60
5.48
1.00
0.63
0.40
0.80
1.27
1.88
1.16
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


168.10
167.96
134.35
134.25
129.37
129.31
129.04
128.93
127.51
127.33
123.04
121.61
108.37
105.85
77.32
77.00
76.68
43.52
43.40
14.69
10.45

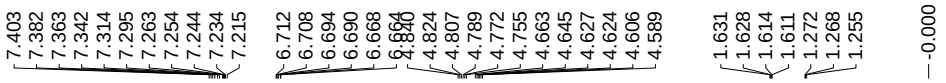

H
N
Me
O


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

2g


7.286
7.211
6.790
6.789
6.786
6.764
6.763
6.759
6.755
6.751
6.729
6.728
6.725
5.217
5.200
5.183
5.164
5.148
5.131
4.854
4.837
4.819
4.815
4.797
4.780
1.685
1.683
1.681
1.668
1.666
1.664
1.632
1.628
1.614
1.611
1.256
1.217
-0.000
O
tBu
N
H
Me
0.83
1.01
0.90
0.10
2.67
0.32
0.83
8.17
f1 (ppm)
175.35
123.64
122.22
107.59
105.01
77.37
77.05
76.73
38.56
27.41
14.82
f1 (ppm)

2h


7.403
7.382
7.363
7.342
7.314
7.295
7.263
7.254
7.244
7.234
7.215
6.712
6.708
6.694
6.690
6.668
6.664
4.840
4.824
4.807
4.789
4.772
4.755
4.663
4.645
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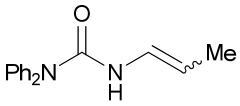
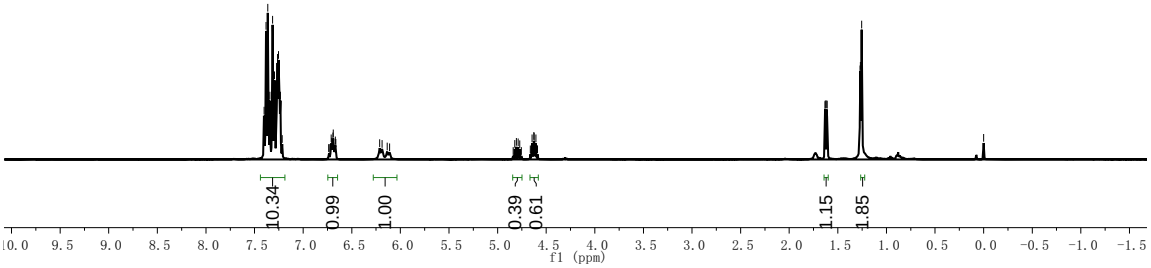
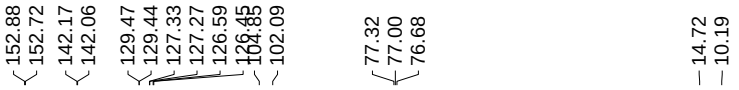
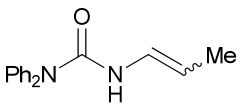
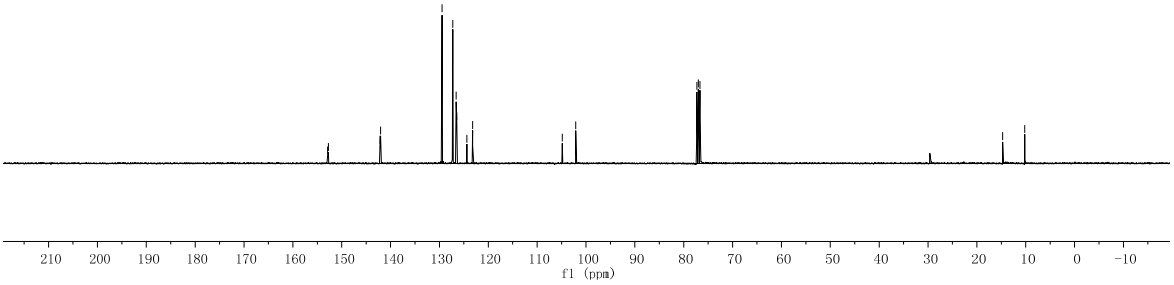
1.611
1.272
1.268
1.255
-0.000


O
Ph2N
N
H
Me

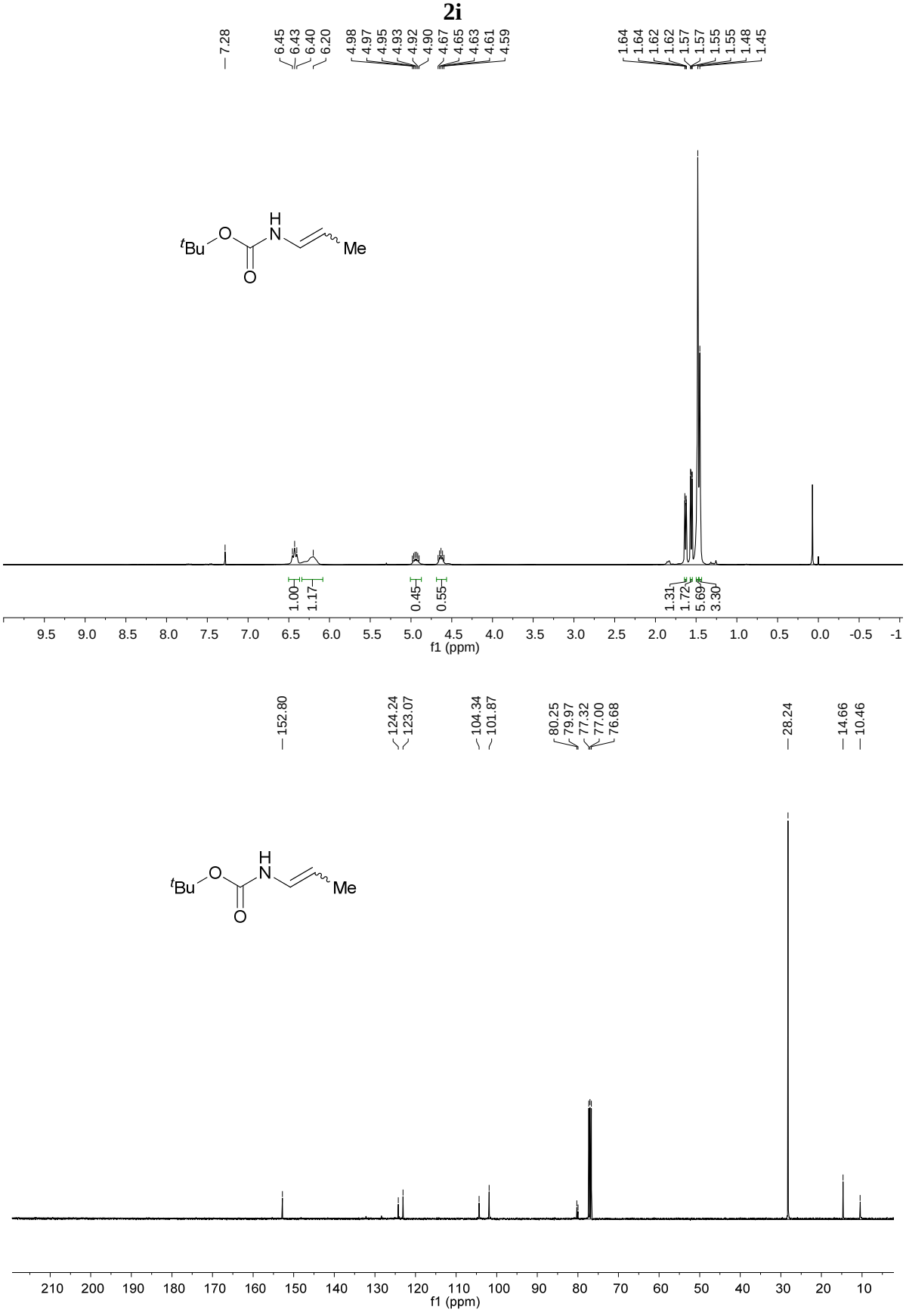

10.34
0.99
1.00
0.39
0.61
1.15
1.85
f1 (ppm)


152.88
152.72
142.17
142.06
129.47
129.44
127.33
127.27
126.59
77.32
77.00
76.68
14.72
10.19

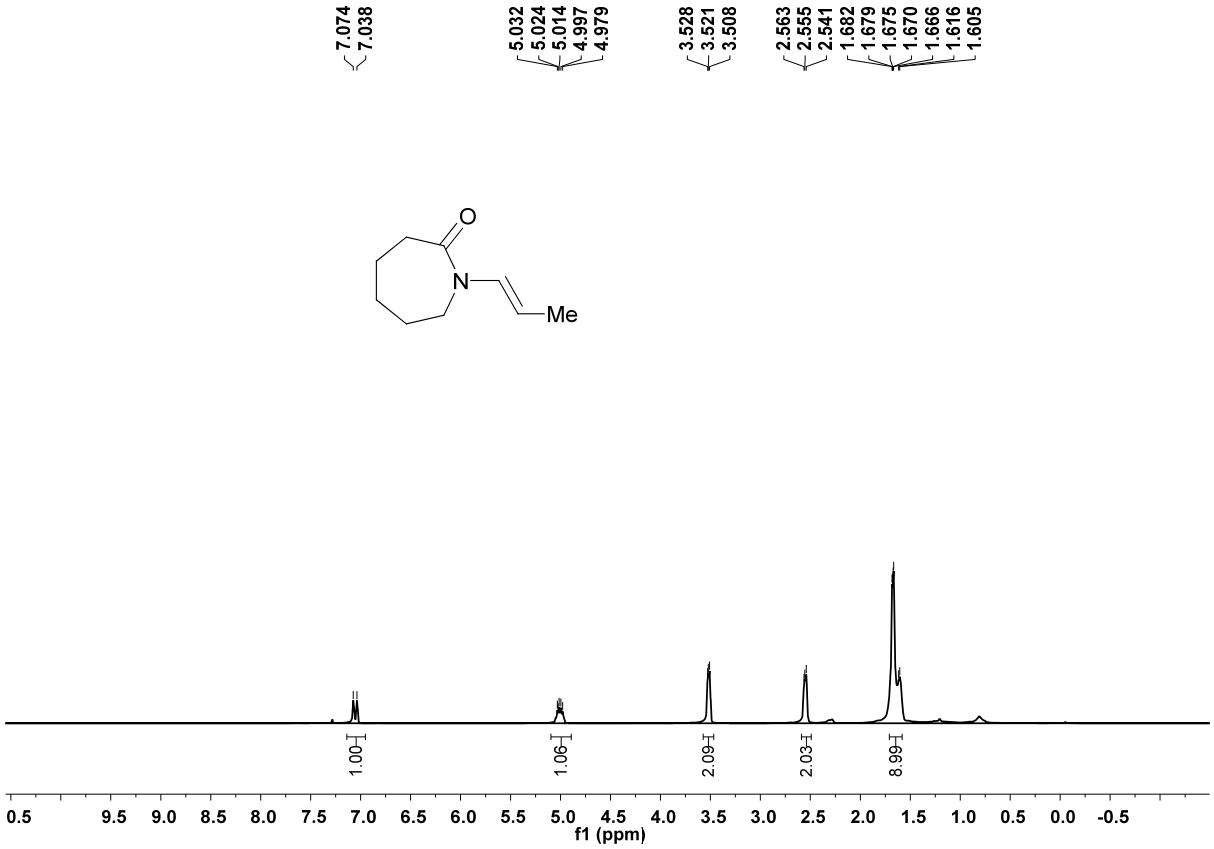

O
Ph2N
N
H
Me

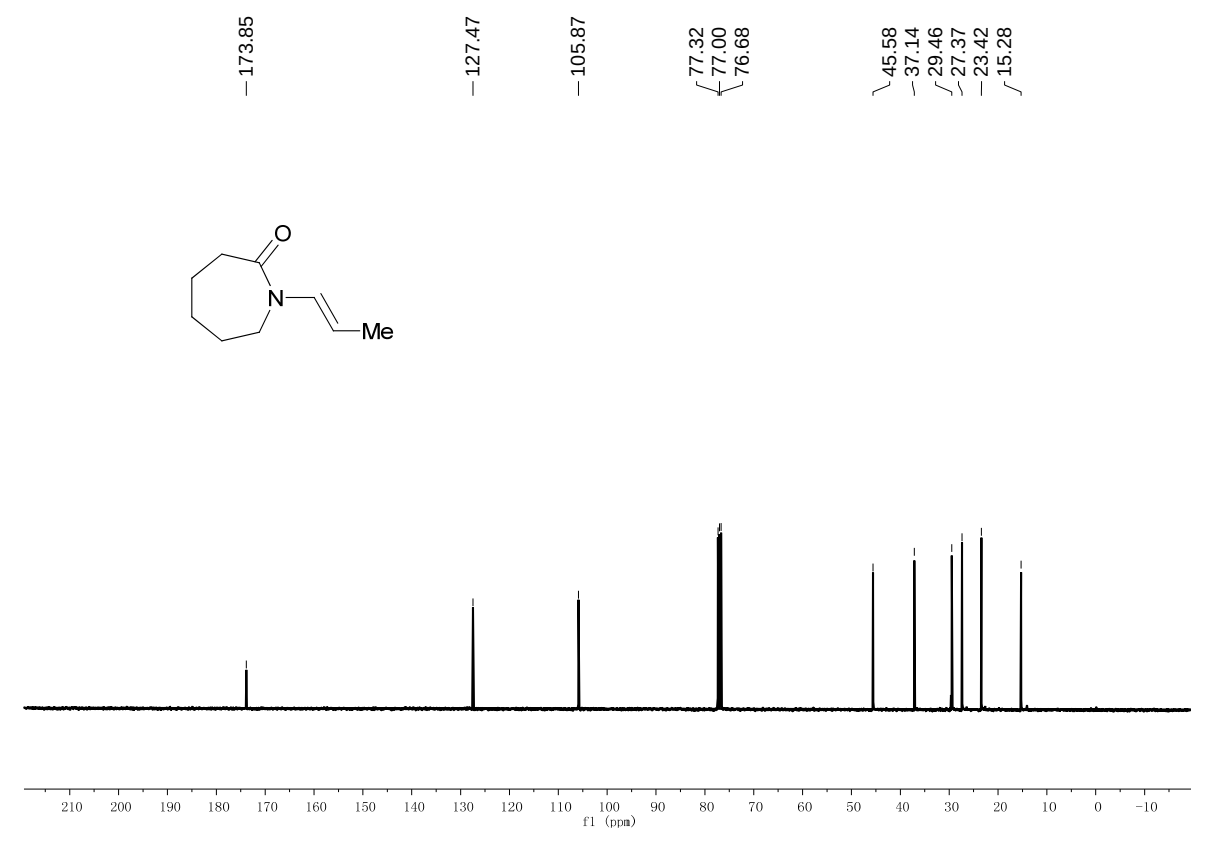

f1 (ppm)

2i


7.28
6.45
6.43
6.40
6.20
4.98
4.97
4.95
4.93
4.92
4.90
4.67
4.65
4.63
4.61
4.59
1.64
1.64
1.62
1.62
1.57
1.57
1.55
1.55
1.48
1.45
tBu
O
H
N
Me
O
1.00
1.17
0.45
0.55
1.31
1.72
5.69
3.30
f1 (ppm)
152.80
124.24
123.07
104.34
101.87
80.25
79.97
77.32
77.00
76.68
28.24
14.66
10.46
f1 (ppm)

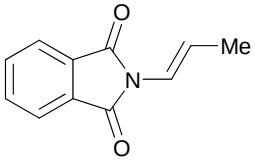
2j

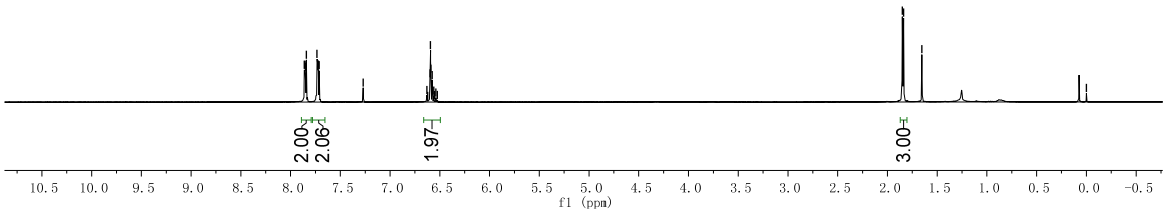

7.074
7.038
5.032
5.024
5.014
4.997
4.979
3.528
3.521
3.508
2.563
2.555
2.541
1.682
1.679
1.675
1.670
1.666
1.616
1.605
O
N
Me
1.00
1.06
2.09
2.03
8.99
f1 (ppm)


173.85
127.47
105.87
77.32
77.00
76.68
45.58
37.14
29.46
27.37
23.42
15.28
O
N
Me
f1 (ppm)

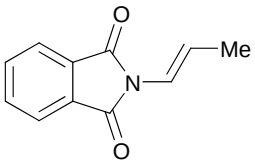
2k

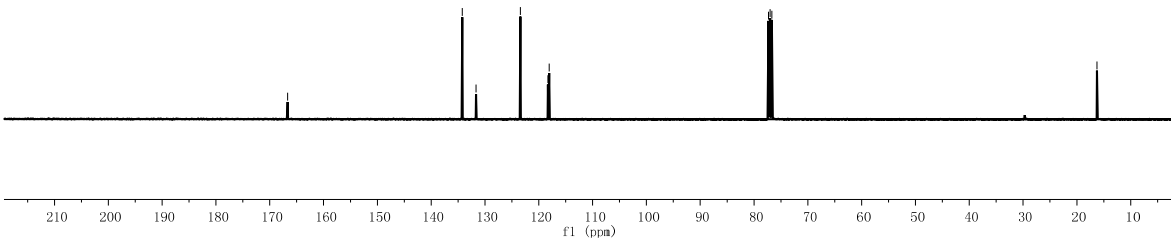

7.862
7.855
7.848
7.841
7.734
7.727
7.721
7.713
7.270
6.630
6.622
6.597
6.593
6.589
6.576
6.561
6.552
6.539
6.524
1.850
1.837
1.654
-0.000


O
N
Me
O

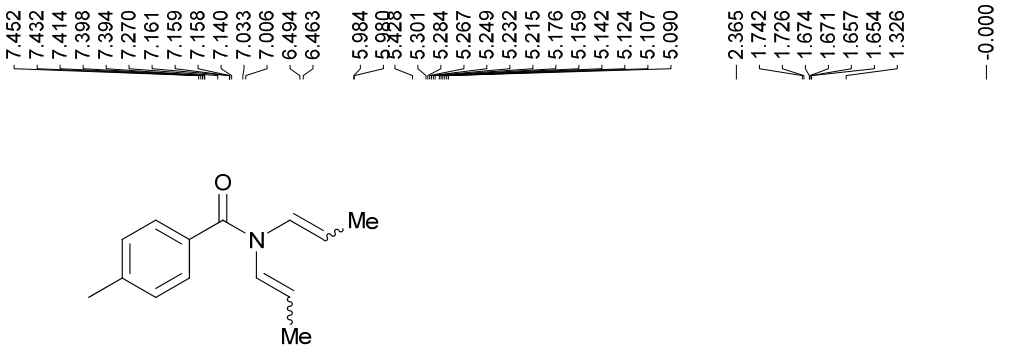

2.00
2.06
1.97
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


166.67
134.22
131.67
123.42
118.30
118.06
77.32
77.00
76.68
16.28

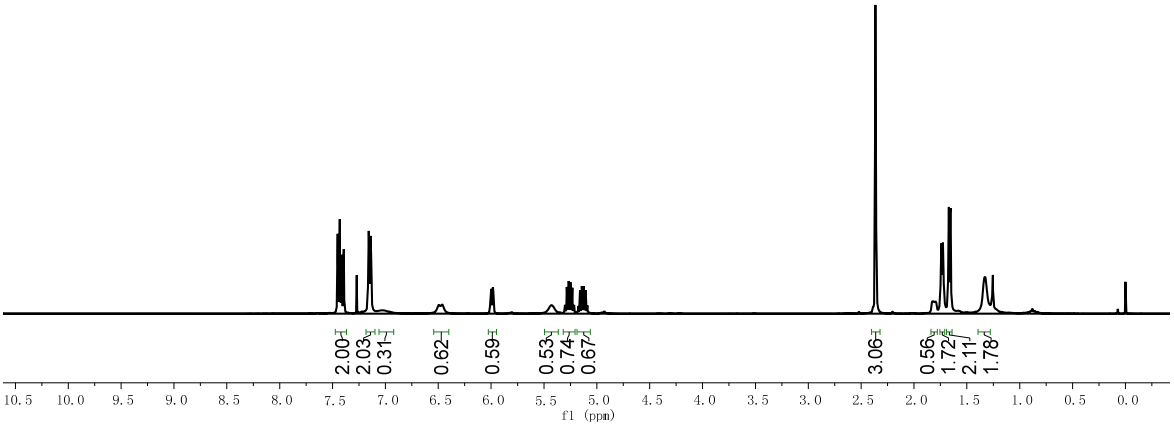

O
N
Me
O

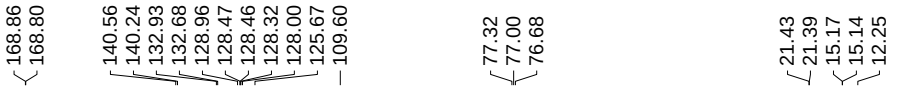

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

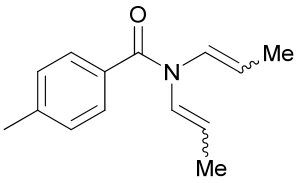
2l

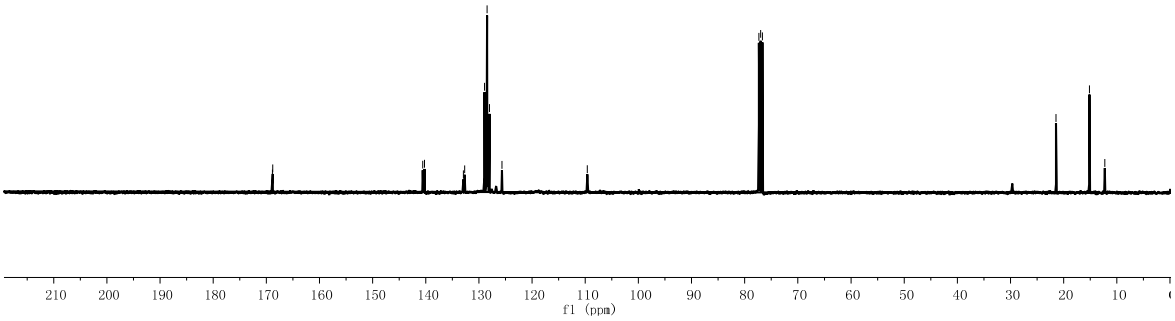

7.452
7.432
7.414
7.398
7.394
7.270
7.161
7.159
7.158
7.140
7.033
7.006
6.494
6.463
5.984
5.980
5.428
5.301
5.284
5.267
5.249
5.232
5.215
5.176
5.159
5.142
5.124
5.107
5.090
2.365
1.742
1.726
1.674
1.671
1.657
1.654
1.326
-0.000

O
N
Me
Me

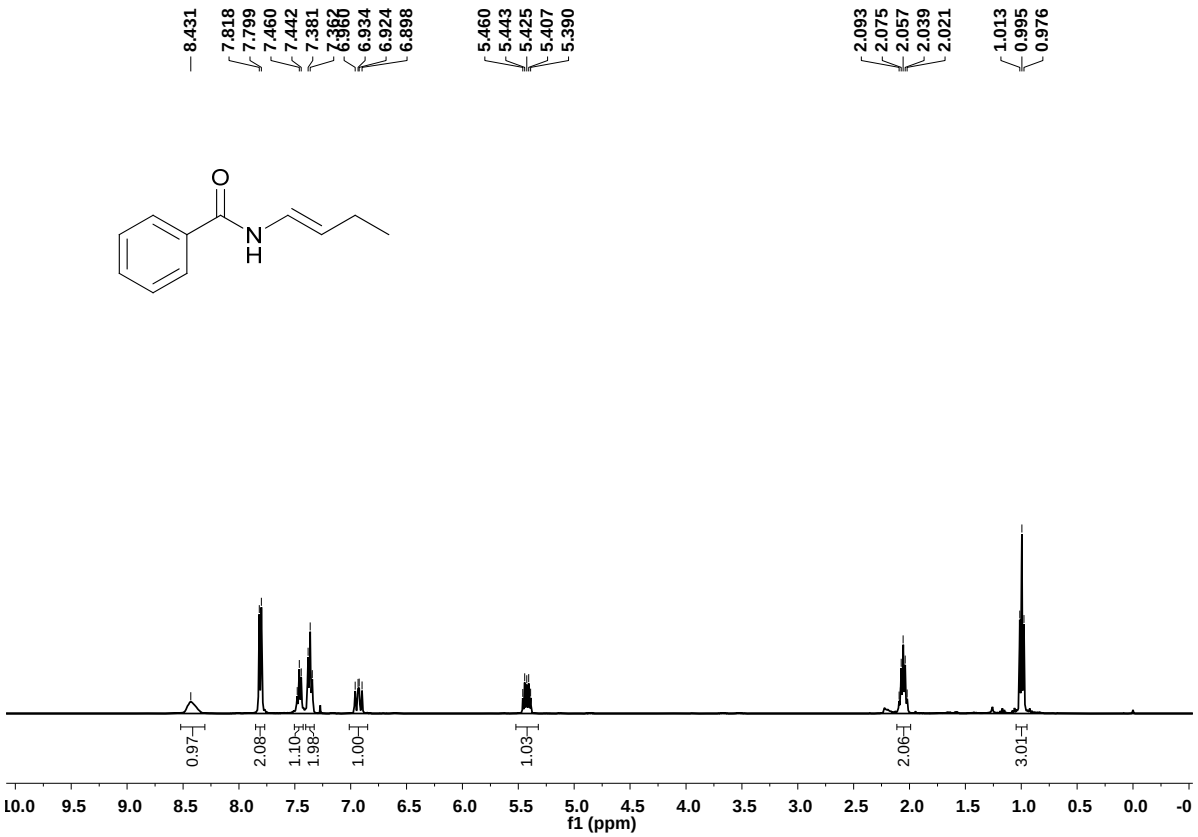

2.00
2.03
0.31
0.62
0.59
0.53
0.74
0.67
3.06
0.56
1.72
2.11
1.78
f1 (ppm)

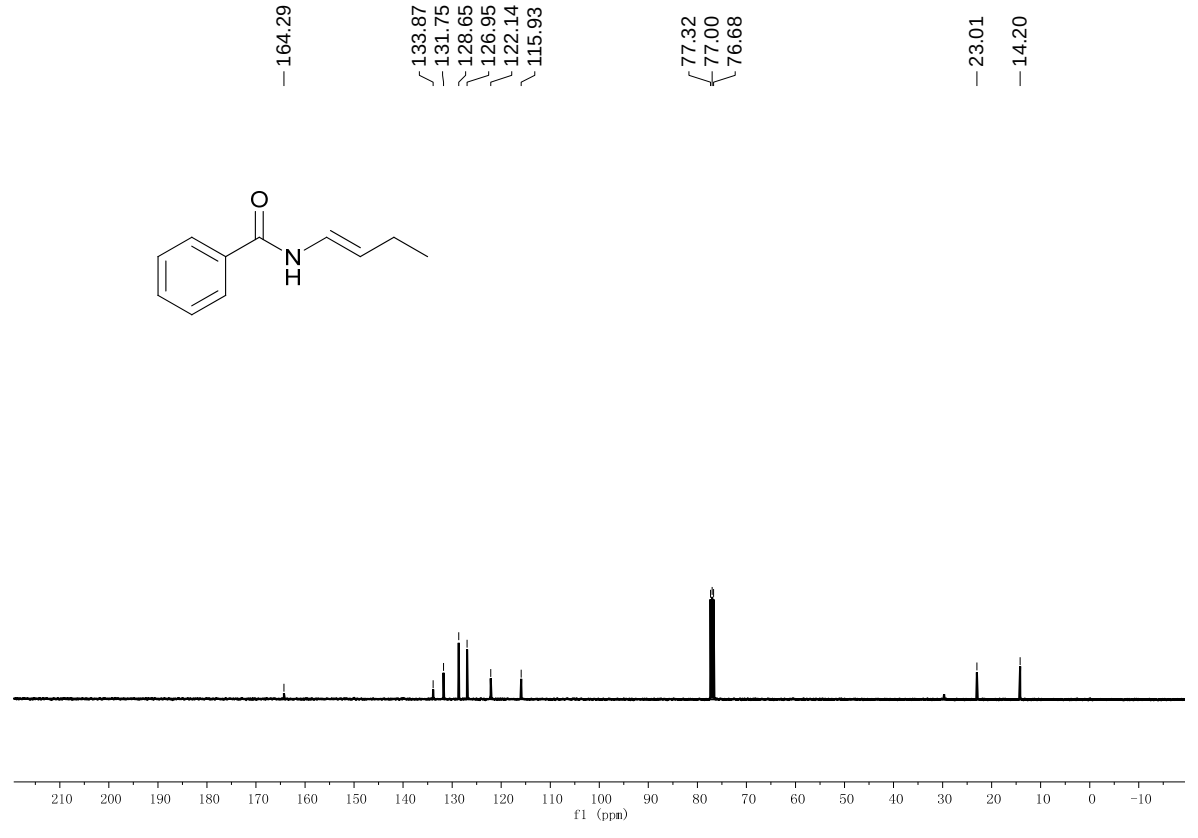

168.86
168.80
140.56
140.24
132.93
132.68
128.96
128.47
128.46
128.32
128.00
125.67
109.60
77.32
77.00
76.68
21.43
21.39
15.17
15.14
12.25


O
N
Me
Me

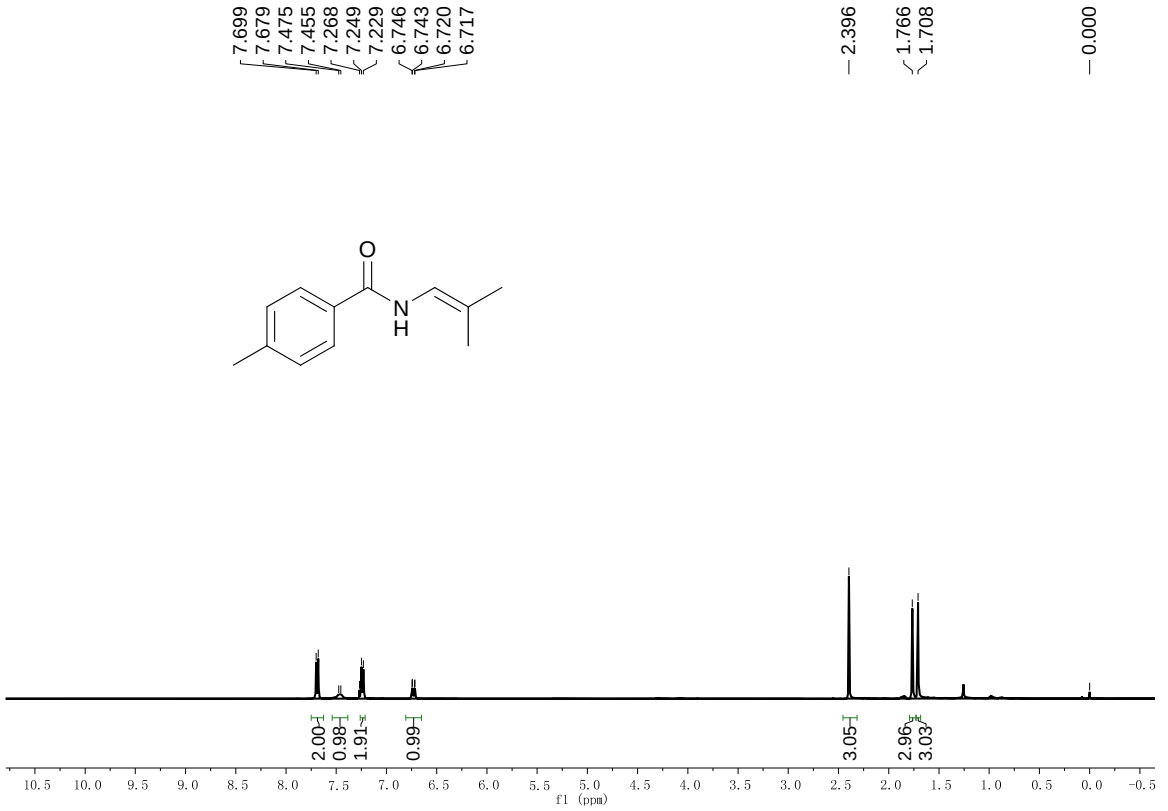

f1 (ppm)

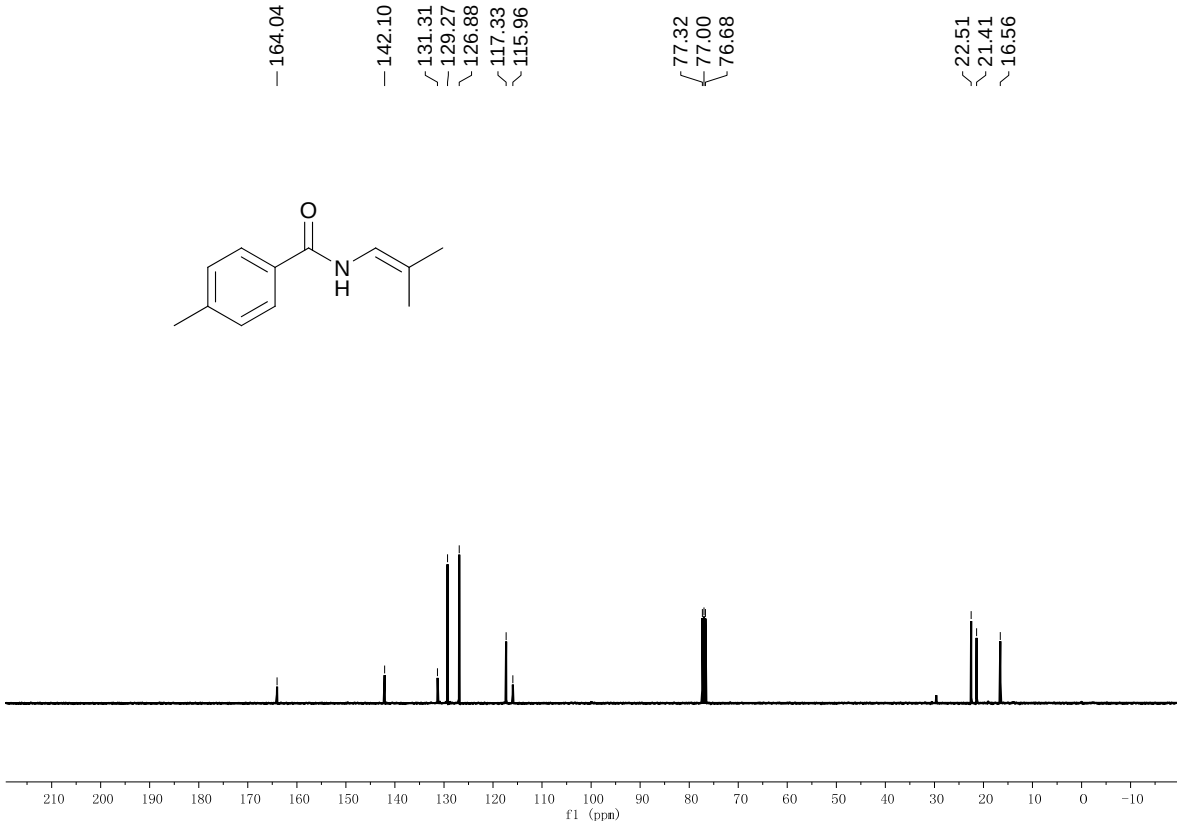
4a


8.431
7.818
7.799
7.460
7.442
7.381
7.362
6.960
6.934
6.924
6.898
5.460
5.443
5.425
5.407
5.390
2.093
2.075
2.057
2.039
2.021
1.013
0.995
0.976
O
N
H
0.97
2.08
1.10
1.98
1.00
1.03
2.06
3.01
f1 (ppm)


164.29
133.87
131.75
128.65
126.95
122.14
115.93
77.32
77.00
76.68
23.01
14.20
O
N
H
f1 (ppm)

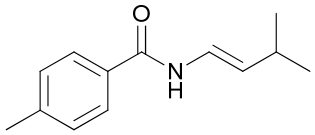
4b

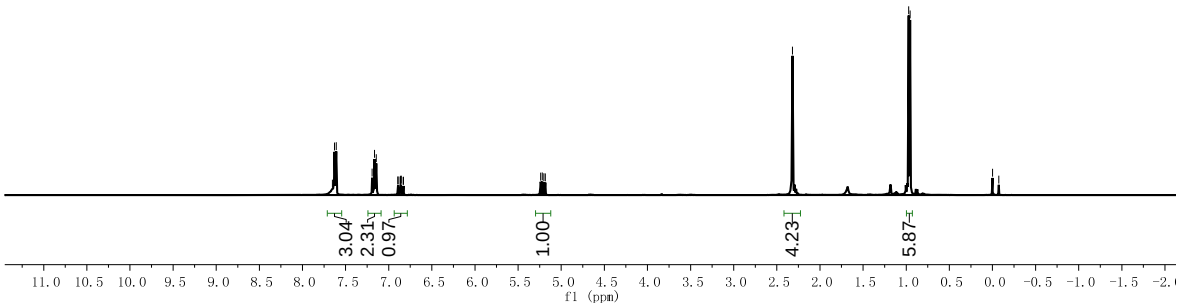

7.699
7.679
7.475
7.455
7.268
7.249
7.229
6.746
6.743
6.720
6.717
2.396
1.766
1.708
0.000
O
N
H
2.00
0.98
1.91
0.99
3.05
2.96
3.03
f1 (ppm)


164.04
142.10
131.31
129.27
126.88
117.33
115.96
77.32
77.00
76.68
22.51
21.41
16.56
O
N
H
f1 (ppm)

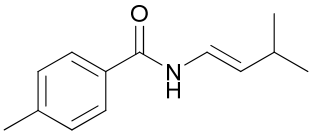
4c

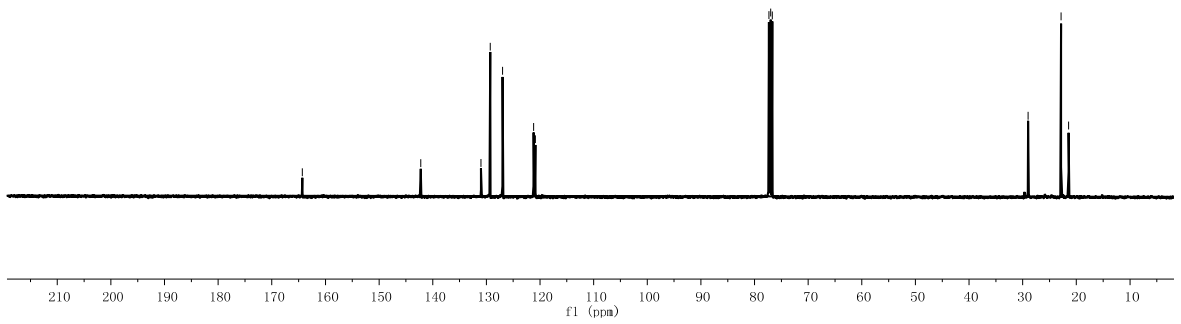

7.652
7.629
7.609
7.192
7.164
7.144
6.894
6.891
6.868
6.865
6.858
6.855
6.832
6.829
5.237
5.220
5.201
5.184
2.318
0.971
0.954
-0.000
-0.073


O
N
H

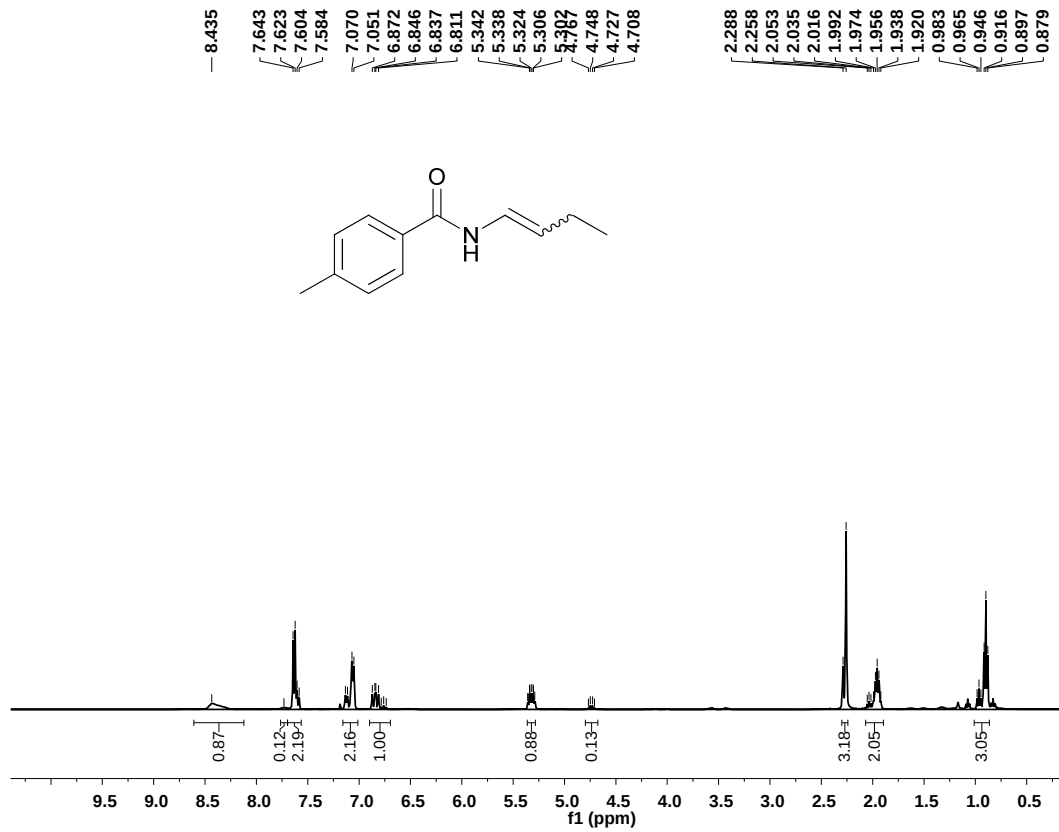

3.04
2.31
0.97
1.00
4.23
5.87
f1 (ppm)

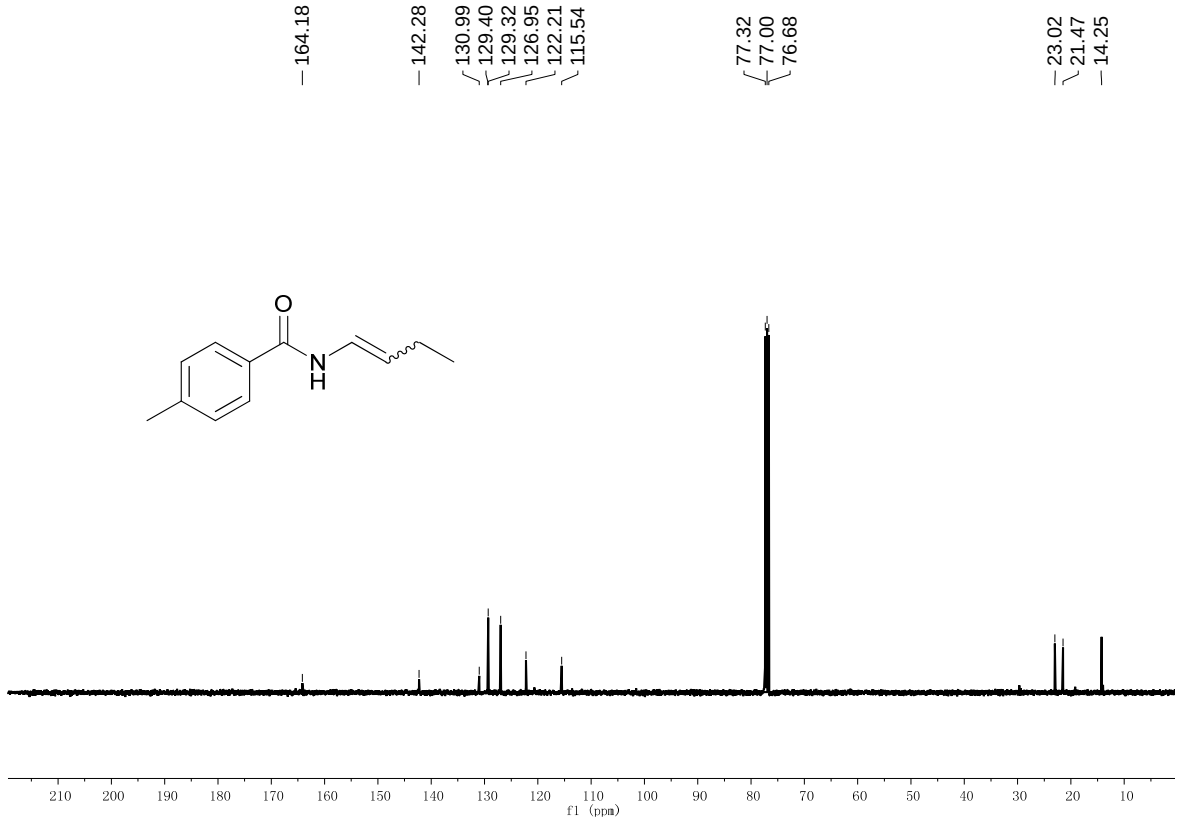

164.33
142.21
131.00
129.27
126.97
121.19
120.85
77.32
77.00
76.68
29.01
22.86
21.44


O
N
H

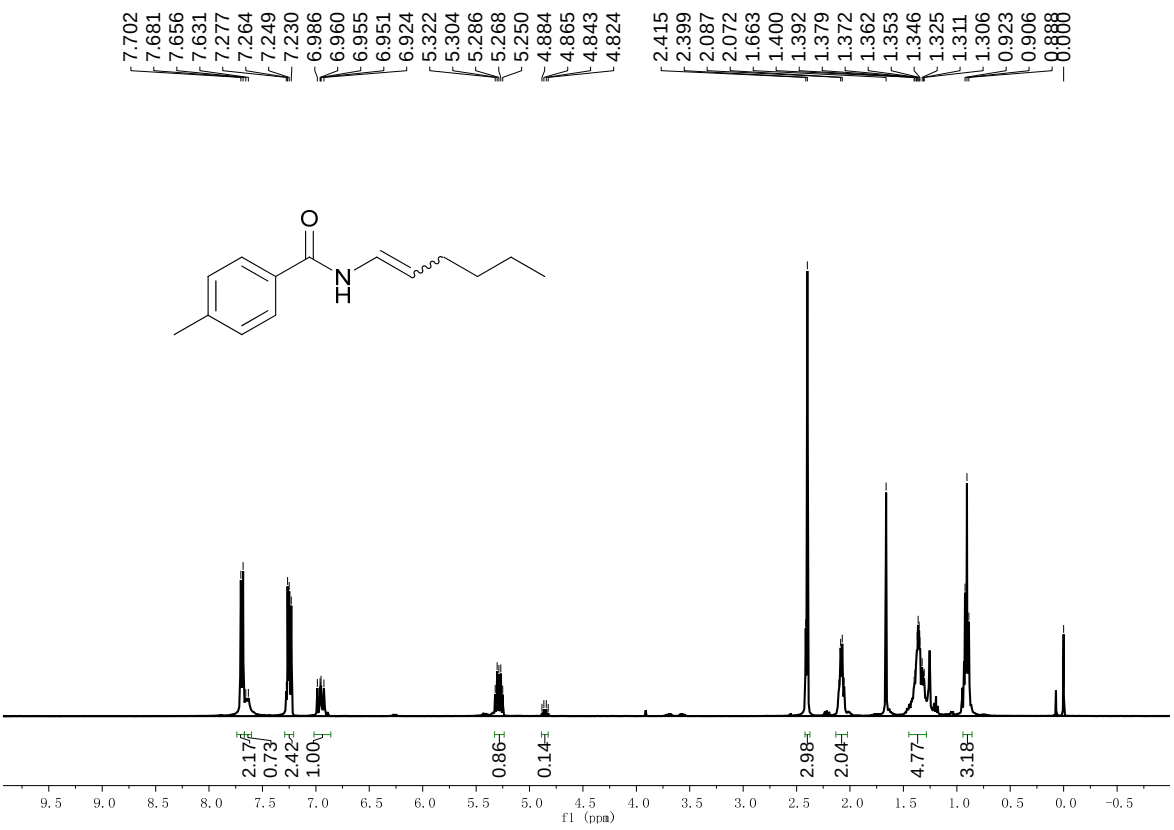

f1 (ppm)

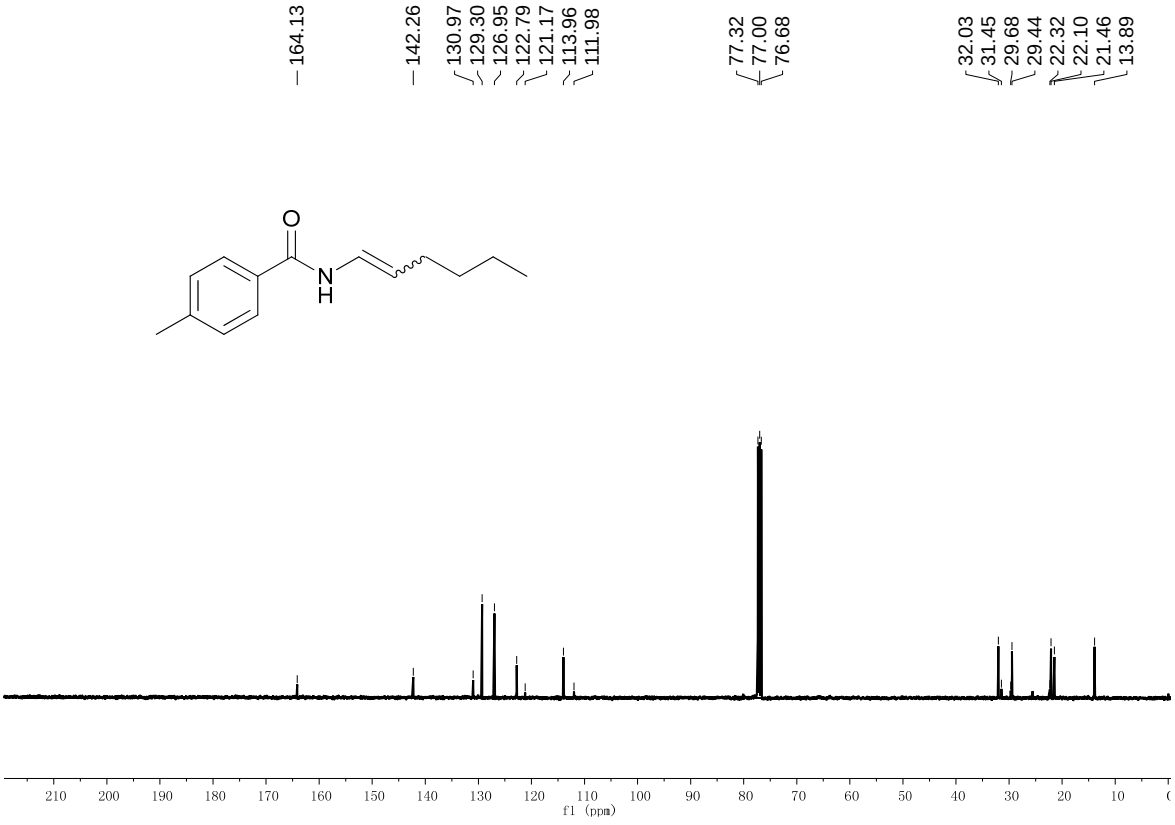
6a


8.435
7.643
7.623
7.604
7.584
7.070
7.051
6.872
6.846
6.837
6.811
5.342
5.338
5.324
5.306
4.748
4.727
4.708
2.288
2.258
2.053
2.035
2.016
1.992
1.974
1.956
1.938
1.920
0.983
0.965
0.946
0.916
0.897
0.879
O
N
H
0.87
0.12
2.19
2.16
1.00
0.88
0.13
3.18
2.05
3.05
f1 (ppm)


164.18
142.28
130.99
129.40
129.32
126.95
122.21
115.54
77.32
77.00
76.68
23.02
21.47
14.25
O
N
H
f1 (ppm)

8a


7.702
7.681
7.656
7.631
7.277
7.264
7.249
7.230
6.986
6.960
6.955
6.951
6.924
5.322
5.304
5.286
5.268
5.250
4.884
4.865
4.843
4.824
2.415
2.399
2.087
2.072
1.663
1.400
1.392
1.379
1.372
1.362
1.353
1.346
1.325
1.311
1.306
0.923
0.906
O
N
H
2.17
0.73
2.42
1.00
0.86
0.14
2.98
2.04
4.77
3.18
f1 (ppm)


164.13
142.26
130.97
129.30
126.95
122.79
121.17
113.96
111.98
77.32
77.00
76.68
32.03
31.45
29.68
29.44
22.32
22.10
21.46
13.89
O
N
H
f1 (ppm)

References

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2. P. Heimbach, *Angew. Chem. Int. Ed. Engl.*, 1964, **3**, 648.

3. S. A. Sergeyev and M. Hesse, *Helv. Chim. Acta.*, 2003, **86**, 750-755.

4. B. Neugnot, J.-C. Cintrat and B. Rousseau, *Tetrahedron.*, 2004, **60**, 3575-3579.