Supporting Information for:

Palladium-catalyzed carbonylation of allylamines via C−N bond activation leading to β,γ-unsaturated amides

Hui Yu,[a] Guoying Zhang,[b] Zong-Jian Liu[a]*, and Hanmin Huang[a,b]*

[a] *College of Chemical Engineering, Zhejiang University of Technology, Hangzhou 310024, China*

[b] *State Key Laboratory for Oxo Synthesis and Selective Oxidation, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, China.*

Fax: +86-931 4968129; Tel: +86-931 4968326; E-mail: hmhuang@licp.cas.cn

CONTENTS

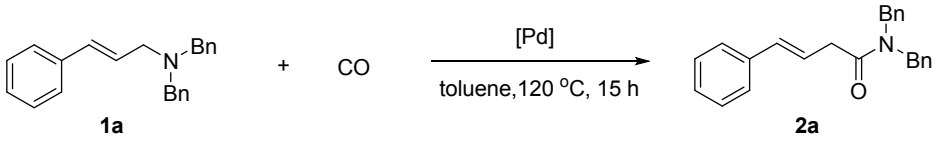
1. General experiment details and materials

Experimental: All solvents before use were dried and degassed by standard methods and stored under argon atmosphere. NMR spectra were recorded on BRUKER DRX 400 spectrometers. Chemical shifts are reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard. Coupling constants (J) are reported in Hz and refer to apparent peak multiplications. High resolution mass spectra (HRMS) were recorded on Bruker MicroTOF-QII mass (ESI). IR spectra were recorder on Nexus 870 FT-IR spectrometers. GC was performed on Agilent 7890 with a HP-5 column. All kinds of allylamines are known compounds and prepared according to the literature.[1]

2. Optimization of the reaction conditions

A mixture of catalyst, ligand, and allylamine was added into a teflon tube which was placed in an autoclave. Then the autoclave was purged and charged with CO at the designed pressure. The reaction mixture was stirred at the designed temperature for 15 h, and then CO was carefully released. The yield was determined by GC analysis relative to the **1** with *n*-hexadecane as internal standard.

Table 1. Screening of the catalysts [a]

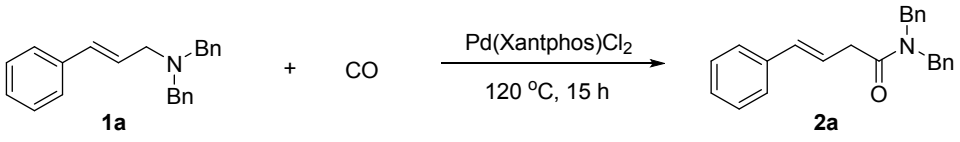


1a **2a**

Entry	[Pd] (5 mol%)	Solvent	Yield (%)
1	$PdCl_2$	NMP/THF(1/1)	43
2	$Pd(PPh_3)_2Cl_2$	NMP/THF(1/1)	38
3	$Pd(DPPP)Cl_2$	NMP/THF(1/1)	0
4	$Pd(Xantphos)Cl_2$	NMP/THF(1/1)	82
5	$Pd(BINAP)Cl_2$	NMP/THF(1/1)	0
6	$Pd(DPE\text{-}phos)Cl_2$	NMP/THF(1/1)	29

[a] Reaction conditions: **1a** (0.5 mmol), CO (10 atm), [Pd] (0.025 mmol), solvent (2.0 mL), 120 °C, 15 h, yields determined by GC using *n*-hexadecane as an internal standard.

Table 2. Screening of the solvents[a]

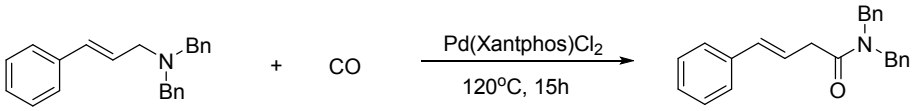


1a **2a**

Entry	[Pd]	Solvent	Yield (%)
1	$Pd(Xantphos)Cl_2$	NMP	77
2	$Pd(Xantphos)Cl_2$	THF	87(81)[b]
3	$Pd(Xantphos)Cl_2$	CH_3CN	0
4	$Pd(Xantphos)Cl_2$	toluene	99(91)[b]
5	$Pd(Xantphos)Cl_2$	xylene	99
6	$Pd(Xantphos)Cl_2$	mesitylene	22

[a] Reaction conditions: **1a** (0.5 mmol), CO (10 atm), $Pd(Xantphos)Cl_2$ (0.025 mmol), solvent (2.0 mL), 120 °C, 15 h, yields determined by GC using *n*-hexadecane as an internal standard. [b] Isolated yields.

Table 3. Screening of pressure of CO [a]



1a **2a**

Entry	CO (atm)	Yield (%)
1	10	99(91)[b]
2	6	90
3	2	44

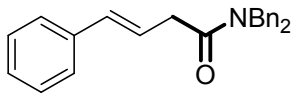
[a] Reaction conditions: **1a** (0.5mmol), Pd(Xantphos)Cl_2 (0.025 mmol), toluene (2.0 mL), 120 °C, 15 h, yield determined by GC using *n*-hexadecane as an internal standard. [b] Isolated yield.

3. General procedure for Pd-catalyzed carbonylation of allylamines

A mixture of allylamine (0.5 mmol), Pd(Xantphos)Cl_2 (18.8 mg, 5% mmol), toluene (2.0 mL) was added into a teflon tube which was placed in an autoclave. Then the autoclave was purged and charged with CO at 10 atm. The reaction mixture was stirred at 120 ^oC for 15 h, and then CO was carefully released. The E/Z ratio of the product was determined by checking the ^1H NMR of crude reaction mixture. The corresponding reaction mixture was purified by flash column chromatography on a silica gel to give the desired product.

4. Experimental characterization data for products 2

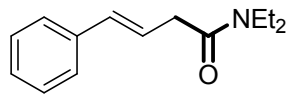
***N,N*-dibenzyl-4-phenylbut-3-enamide (2a) (E/Z=9:1):** The title compound was



2a

prepared according to the general procedure and purified by flash column chromatography to give white solid, 155.2 mg, 91% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.39 (t, J = 2.4 Hz, 2H), 4.50 (s, 2H), 4.63 (s, 2H), 6.42 (t, J = 2.4 Hz, 2H), 7.17-7.39 (m, 15H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 38.0, 48.4, 50.2, 123.3, 126.4, 126.5, 127.6, 127.6, 127.8, 128.5, 128.6, 128.8, 129.1, 132.9, 136.5, 137.1, 137.4, 171.7; **HRMS** (ESI) calcd. for $C_{24}H_{23}NO$ [M+Na]: 364.1672, found: 364.1689.

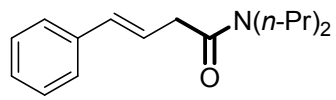
***N,N*-diethyl-4-phenylbut-3-enamide (2b) (E/Z=9:1):** The title compound was



2b

prepared according to the general procedure and purified by flash column chromatography to white solid, 100.9 mg, 93% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.06 (t, J = 7.2 Hz, 6H), 2.56 (q, J =7.2 Hz, 4H), 3.24 (dd, J_1 =1.2 Hz, J_2 =6.8 Hz 2H), 6.26-6.33 (m, 1H), 6.49 (d, J = 16.0 Hz, 1H), 7.19-7.39 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.2, 14.6, 38.0, 40.3, 42.3, 124.0, 126.3, 127.4, 128.6, 132.4, 137.2, 170.2; **HRMS** (ESI) calcd. for $C_{14}H_{19}NO$ [M+Na]: 240.1359, found: 240.1369.

4-phenyl-*N,N*-dipropylbut-3-enamide (2c) (E/Z=13:1): The title compound was

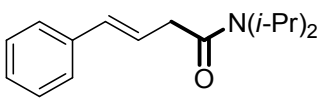


2c

prepared according to the general procedure and purified by flash column chromatography to give white solid, 100.5 mg, 82% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.88-0.95 (m, 6H), 1.53-1.66 (m, 4H), 3.23 (t, J = 8.0 Hz, 2H), 3.29 (dd, J_1 = 6.0 Hz, J_2 =

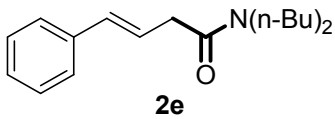
9.6 Hz, 4H), 6.35-6.48 (m, 2H), 7.19-7.38 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.4, 11.5, 21.0, 22.4, 38.0, 47.7, 49.9, 124.0, 126.3, 127.4, 128.6, 132.4, 137.2, 170.6; **HRMS** (ESI) calcd. for $C_{16}H_{23}NO$ [M+Na]: 268.1672, found: 268.1678.

***N,N*-diisopropyl-4-phenylbut-3-enamide (2d) (E/Z>20:1):** The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 95.6 mg, 78% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.19 (d, *J* = 6.8, 6H), 1.40 (d, *J* = 6.8, 6H), 3.25 (d, *J* = 6.0 Hz, 2H), 3.47 (s, 1H), 3.97-4.03 (m, 1H), 6.33-6.48 (m, 2H), 7.18-7.38 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 20.8, 21.1, 40.1, 45.9, 49.0, 124.1, 126.3, 127.4, 128.6, 132.3, 137.3, 169.9; **HRMS** (ESI) calcd. for $C_{16}H_{23}NO$ [M+Na]: 268.1672, found: 268.1679.



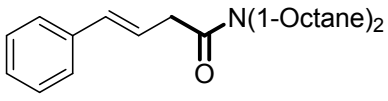
2d

***N,N*-dibutyl-4-phenylbut-3-enamide (2e) (E/Z=11:1):** The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 102.4 mg, 75% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.91-0.97(m, 6H), 1.29-1.37 (m, 4H), 1.49-1.69 (m, 4H), 3.23-3.35 (m, 6H), 6.34-6.48 (m, 2H), 7.19-7.38 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.0, 14.0, 20.3, 20.4, 30.0, 31.4, 38.1, 45.9, 48.1, 124.1, 126.4, 127.4, 128.6, 132.4, 137.3, 170.5; **HRMS** (ESI) calcd. for $C_{18}H_{27}NO$ [M+Na]: 296.1985, found: 296.1991.



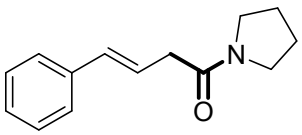
2e

***N,N*-dioctyl-4-phenylbut-3-enamide (2f) (E/Z=7:1):** The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 138.6 mg, 72% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.79 (q, *J* = 6.4 Hz, 6H), 1.20 (d, *J* = 3.6 Hz, 20H), 1.48 (t, *J* = 7.2 Hz, 4H), 3.14-3.25 (m, 6H), 6.28-6.40 (m, 2H), 7.11-7.30 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.2, 22.7, 22.7, 27.0, 27.1, 27.8, 29.3, 29.3, 29.3, 29.4, 29.5, 31.8, 31.9, 38.0, 46.1, 48.2, 124.0, 126.3, 127.4, 128.5, 132.4, 137.2, 170.4; **HRMS** (ESI) calcd. for $C_{26}H_{43}NO$ [M+Na]: 408.3237, found: 408.3232.



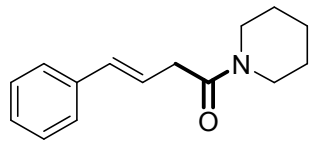
2f

4-phenyl-1-(pyrrolidin-1-yl)but-3-en-1-one (2g) (E/Z=12:1): The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 54.8 mg, 51% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.82-1.99 (m, 4H), 3.24-3.25 (m, 2H), 3.46-3.51 (m, 4H), 6.34-6.50 (m, 2H), 7.19-7.39 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 24.5, 26.3, 39.6, 45.9, 46.8, 123.3, 126.3, 127.5, 128.6, 132.8, 137.2, 169.5; **HRMS** (ESI) calcd. for $C_{14}H_{17}NO$ [M+Na]: 238.1202, found: 238.1210.



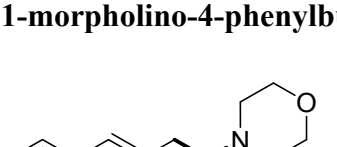
2g

4-phenyl-1-(piperidin-1-yl)but-3-en-1-one (2h) (E/Z=13:1): The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 75.6 mg, 66% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.53-1.57 (m, 4H), 1.62-1.66 (m, 2H), 3.30 (dd, J_1 = 1.6 Hz, J_2 = 6.8 Hz, 2H), 3.44 (t, J = 5.6 Hz, 2H), 3.58 (t, J = 5.6 Hz, 2H), 6.30-6.38 (m, 1H), 6.45 (d, J = 16.0 Hz, 1H), 7.20-7.38 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 24.6, 25.7, 26.7, 38.3, 43.0, 47.2, 123.6, 126.3, 127.5, 128.6, 132.6, 137.2, 169.3; **HRMS** (ESI) calcd. for $C_{15}H_{19}NO$ [M+Na]: 252.1359, found: 252.1364



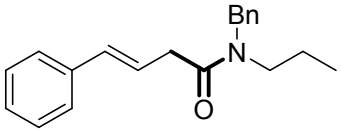
2h

1-morpholino-4-phenylbut-3-en-1-one (2i) (E/Z=19:1): The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 95.9 mg, 83 % yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.30 (dd, J_1 = 1.6 Hz, J_2 = 6.8 Hz, 2H), 3.51 (t, J = 5.2 Hz, 2H), 3.65-3.68 (m, 6H), 6.29-6.36 (m, 1H), 6.46 (d, J = 16.0 Hz, 1H), 7.21-7.38 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 37.9, 42.2, 46.4, 66.8, 67.0, 122.8, 126.4, 127.7, 128.7, 133.1, 136.9, 169.7; **HRMS** (ESI) calcd. for $C_{14}H_{17}NO_2$ [M+Na]: 254.1151, found: 254.1155



2i

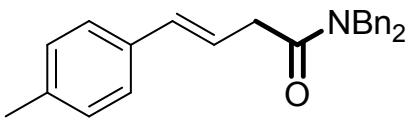
***N*-benzyl-4-phenyl-*N*-propylbut-3-enamide (2j) (E/Z=7:1):** The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 92.3 mg, 63% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.87-0.92



2j

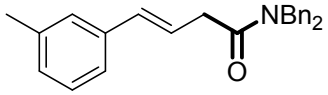
(m, 3H), 1.56-1.63 (m, 2H), 3.18-3.38 (m, 4H), 4.58 (s, 1H), 4.63 (s, 1H), 6.36-6.47 (m, 2H), 7.18-7.38 (m, 10H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.4, 11.5, 20.9, 22.0, 37.9, 38.2, 48.2, 48.4, 49.0, 51.4, 123.6, 123.7, 126.3, 126.4, 126.4, 127.4, 127.5, 127.5, 127.7, 128.2, 128.6, 128.6, 128.7, 129.1, 132.7, 137.1, 137.2, 137.9, 171.1, 171.4; **HRMS** (ESI) calcd. for $C_{20}H_{23}NO$ [M+Na]: 316.1672, found: 316.1678

(E)-*N,N*-dibenzyl-4-p-tolylbut-3-enamide (2k) (E/Z=7:1): The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 133.1 mg, 75 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.30 (s, 0.43H), 2.32 (s, 2.32H), 3.37-3.38 (m, 1.69H), 3.45-3.46 (m, 0.23H), 4.45 (s, 0.25H), 4.49 (s, 1.75H), 4.62-4.64 (m, 2H), 6.22-6.42 (m, 2H), 6.99-7.31 (m, 14H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 38.1, 48.4, 50.2, 122.2, 126.3, 126.6, 127.6, 127.8, 128.5, 128.8, 129.1, 129.3, 132.8, 134.3, 136.6, 137.3, 137.4, 171.9; HRMS (ESI) calcd. for $C_{25}H_{25}NO$ [M+Na]: 378.1828, found: 378.1831



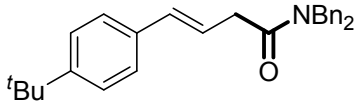
2k

***N,N*-dibenzyl-4-m-tolylbut-3-enamide (2l) (E/Z>20:1):** The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 129.6 mg, 73% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.32 (s, 3H), 3.37 (d, *J* = 4.8 Hz, 2H), 4.49 (s, 2H), 4.63 (s, 2H), 6.39-6.41 (m, 2H), 7.02-7.39 (m, 14H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 21.5, 38.0, 48.4, 50.2, 123.1, 123.6, 126.5, 127.1, 127.6, 127.8, 128.3, 128.5, 128.5, 128.8, 129.1, 133.0, 136.5, 137.0, 137.4, 138.1, 171.8; **HRMS** (ESI) calcd. for $C_{25}H_{25}NO$ [M+Na]: 378.1828, found: 378.1839.



2l

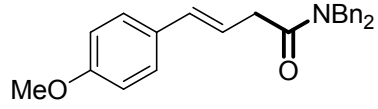
***N,N*-dibenzyl-4-(4-tert-butylphenyl)but-3-enamide (2m) (E/Z=6:1):** The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 151.0 mg, 76% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.30 (s, 9H), 3.37 (d, *J* = 4.8 Hz, 2H), 4.48 (s, 2H), 4.62 (s, 2H), 6.37 (d, *J* = 7.2 Hz, 2H), 7.16-7.38 (m, 14H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 31.4, 34.7, 38.1, 48.4,



2m

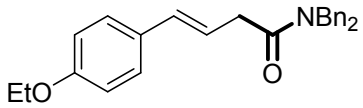
50.2, 122.4, 125.5, 126.1, 126.6, 127.6, 127.8, 128.5, 128.8, 129.1, 132.7, 134.3, 136.6, 137.4, 150.7, 171.8; **HRMS** (ESI) calcd. for $C_{28}H_{31}NO$ [M+Na]: 420.2298, found: 420.2284.

***N,N*-dibenzyl-4-(4-methoxyphenyl)but-3-enamide (2n) (E/Z=6:1):** The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 137.3 mg, 74% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.35-3.37 (m, 2H), 3.79 (s, 3H), 4.49 (s, 2H), 4.62 (s, 2H), 6.23-6.38 (m, 2H), 6.81-6.85 (m, 2H), 7.17-7.39 (m, 12H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 38.0, 48.4, 50.2, 55.4, 114.0, 121.0, 126.5, 127.5, 127.6, 127.8, 128.5, 128.7, 129.1, 129.9, 132.3, 136.6, 137.4, 159.2, 171.9; **HRMS** (ESI) calcd. for $C_{25}H_{25}NO_2$ [M+Na]: 394.1778, found: 394.1781.



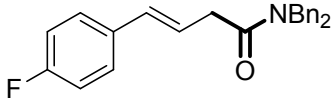
2n

***N,N*-dibenzyl-4-(4-ethoxyphenyl)but-3-enamide (2o) (E/Z=14:1):** The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 142.5 mg, 74% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.40 (t, *J* = 7.2 Hz, 3H), 3.35 (d, *J* = 6.4 Hz, 2H), 4.00 (q, *J* = 6.8 Hz, 2H), 4.49 (s, 2H), 4.62 (s, 2H), 6.22-6.38 (m, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 7.16-7.38 (m, 12H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.9, 38.0, 48.4, 50.2, 63.5, 114.6, 120.8, 126.5, 127.5, 127.6, 127.8, 128.5, 128.7, 129.1, 129.8, 132.4, 136.6, 137.4, 158.6, 171.9; **HRMS** (ESI) calcd. for $C_{26}H_{27}NO_2$ [M+Na]: 408.1934, found: 408.1931.



2o

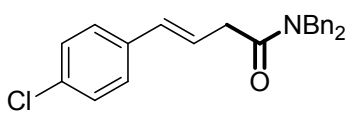
***N,N*-dibenzyl-4-(4-fluorophenyl)but-3-enamide (2p) (E/Z=8:1):** The title compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 154.4 mg, 86% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.63 (d, *J* = 4.8 Hz, 2H), 4.49 (s, 2H), 4.63 (s, 2H), 6.33-6.35 (m, 2H), 6.98-6.99 (m, 2H), 7.17-7.39 (m, 14H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 37.8, 48.6, 50.3, 115.4, 115.6, 123.1, 123.1, 126.5, 127.7, 127.8, 127.9, 127.9, 128.6, 128.8, 129.2, 131.8, 133.3, 133.3, 136.5, 137.4, 171.7; **HRMS** (ESI) calcd. for



2p

$C_{24}H_{23}FNO$ [M+H]: 360.1758, found: 360.1762.

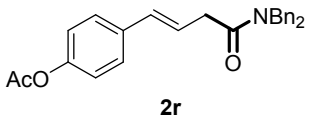
***N,N*-dibenzyl-4-(4-chlorophenyl)but-3-enamide (2q) (E/Z=7:1):** The title compound



2q

was prepared according to the general procedure and purified by flash column chromatography to give white solid, 144.4 mg, 77% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.37 (d, *J* = 5.6 Hz, 2H), 4.49 (s, 2H), 4.63 (s, 2H), 6.32-6.43 (m, 2H), 7.17-7.39 (m, 14H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 37.8, 48.6, 50.2, 124.1, 126.5, 127.6, 127.7, 127.9, 128.6, 128.8, 129.2, 131.7, 133.2, 135.6, 136.5, 137.3, 171.6; **HRMS** (ESI) calcd. for $C_{24}H_{22}ClNO$ [M+Na]: 398.1282, found: 398.1289.

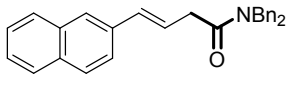
(E)-4-(4-(dibenzylamino)-4-oxobut-1-enyl)phenyl acetate (E/Z= 9:1): The title



2r

compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 165.6 mg, 83 % yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.19 (s, 3H), 3.28-3.29 (m, 1.8H), 3.81-3.40 (m, 0.2H), 4.40 (s, 2H), 4.54 (s, 2H), 6.15-6.19 (m, 0.11H), 6.28-6.30 (m, 1.73H), 6.72-6.73 (m, 0.15H), 6.72-7.30 (m, 14H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 21.2, 27.0, 37.8, 48.4, 50.2, 121.7, 123.5, 126.5, 127.2, 127.6, 127.8, 128.5, 128.7, 129.1, 131.9, 134.9, 136.5, 137.3, 150.0, 169.5, 171.6; **HRMS** (ESI) calcd. for $C_{26}H_{25}NO_3$ [M+Na]: 422.1727, found: 422.1738

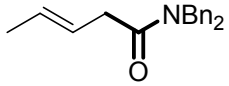
***N,N*-dibenzyl-4-(naphthalen-2-yl)but-3-enamide (2s) (E/Z=13:1):** The title



2s

compound was prepared according to the general procedure and purified by flash column chromatography to give white solid, 164.2 mg, 84% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.43 (t, *J* = 2.4 Hz, 2H), 4.52 (s, 2H), 4.64 (s, 2H), 6.54-6.56 (m, 2H), 7.18-7.79 (m, 17H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 38.1, 48.5, 50.3, 123.7, 125.9, 126.1, 126.3, 126.6, 127.6, 127.8, 127.8, 128.1, 128.3, 128.6, 128.8, 129.2, 133.1, 133.7, 134.6, 136.5, 137.4, 171.8; **HRMS** (ESI) calcd. for $C_{28}H_{25}NO$ [M+Na]: 414.1828, found: 414.1841.

(E)-*N,N*-dibenzylpent-3-enamide 2t (E/Z=3:1): The title compound was prepared



2t

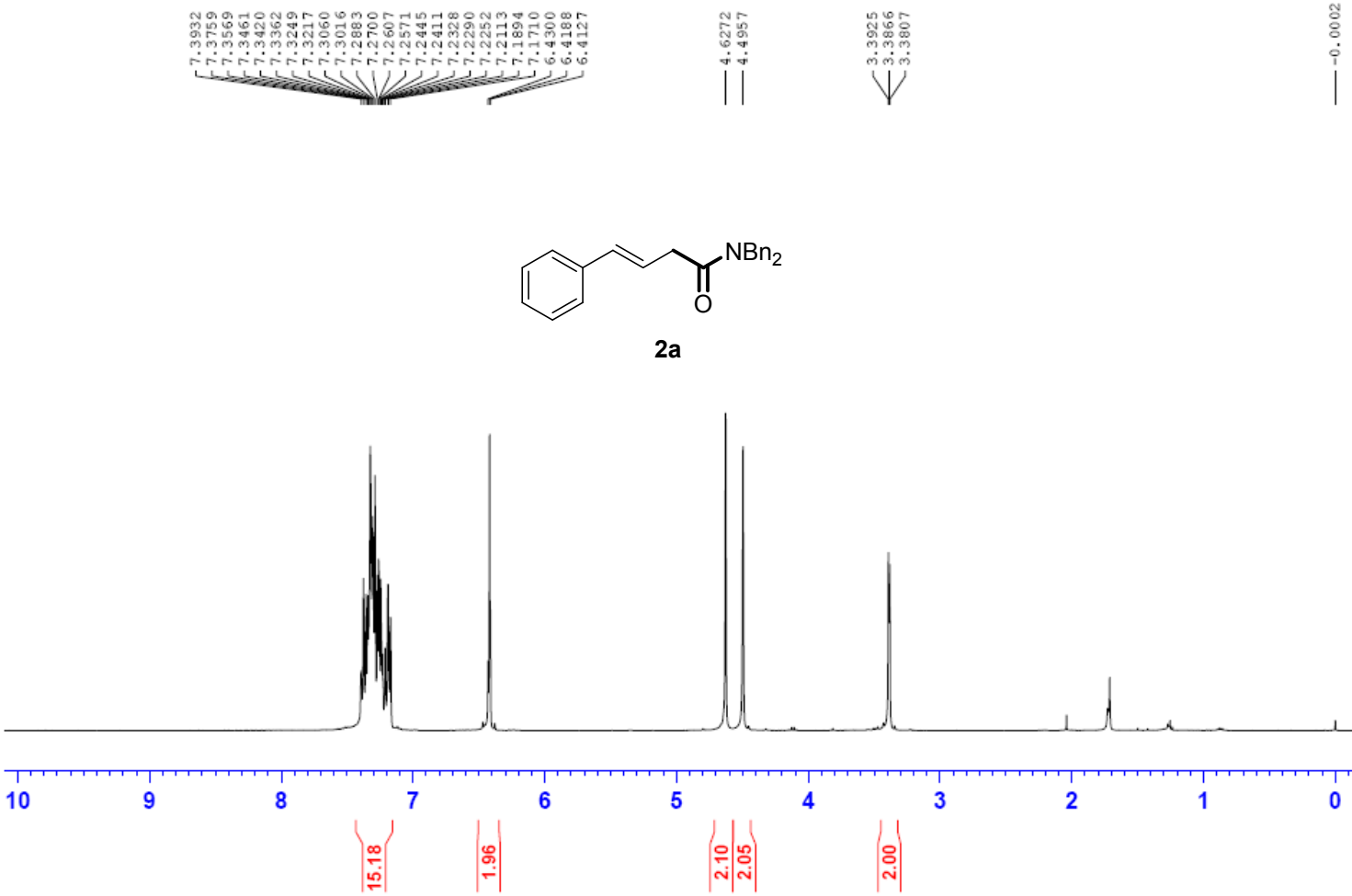
according to the general procedure and purified by flash column chromatography to give white solid, 93.5 mg, 67% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.56-1.70 (m, 3H), 3.15-3.23 (m, 2H), 4.44 (s, 2H), 4.59 (d, *J* = 6.8 Hz, 2H), 5.48-5.69 (m, 2H), 7.14-7.38 (m, 10H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.2, 18.1, 32.5, 37.7, 48.2, 48.3, 50.0, 123.2, 124.1, 126.5, 126.5, 127.0, 127.5, 127.7, 128.4, 128.5, 128.7, 128.9, 129.0, 136.6, 137.5, 172.2, 172.3; **HRMS** (ESI) calcd. for $C_{23}H_{24}N$ [M+1]: 302.1515, found: 302.1530.

References：

1. Y. Xie, J. Hu, Y. Wang, C. Xia, H. Huang, *J. Am. Chem. Soc.* 2012, **134**, 20613.

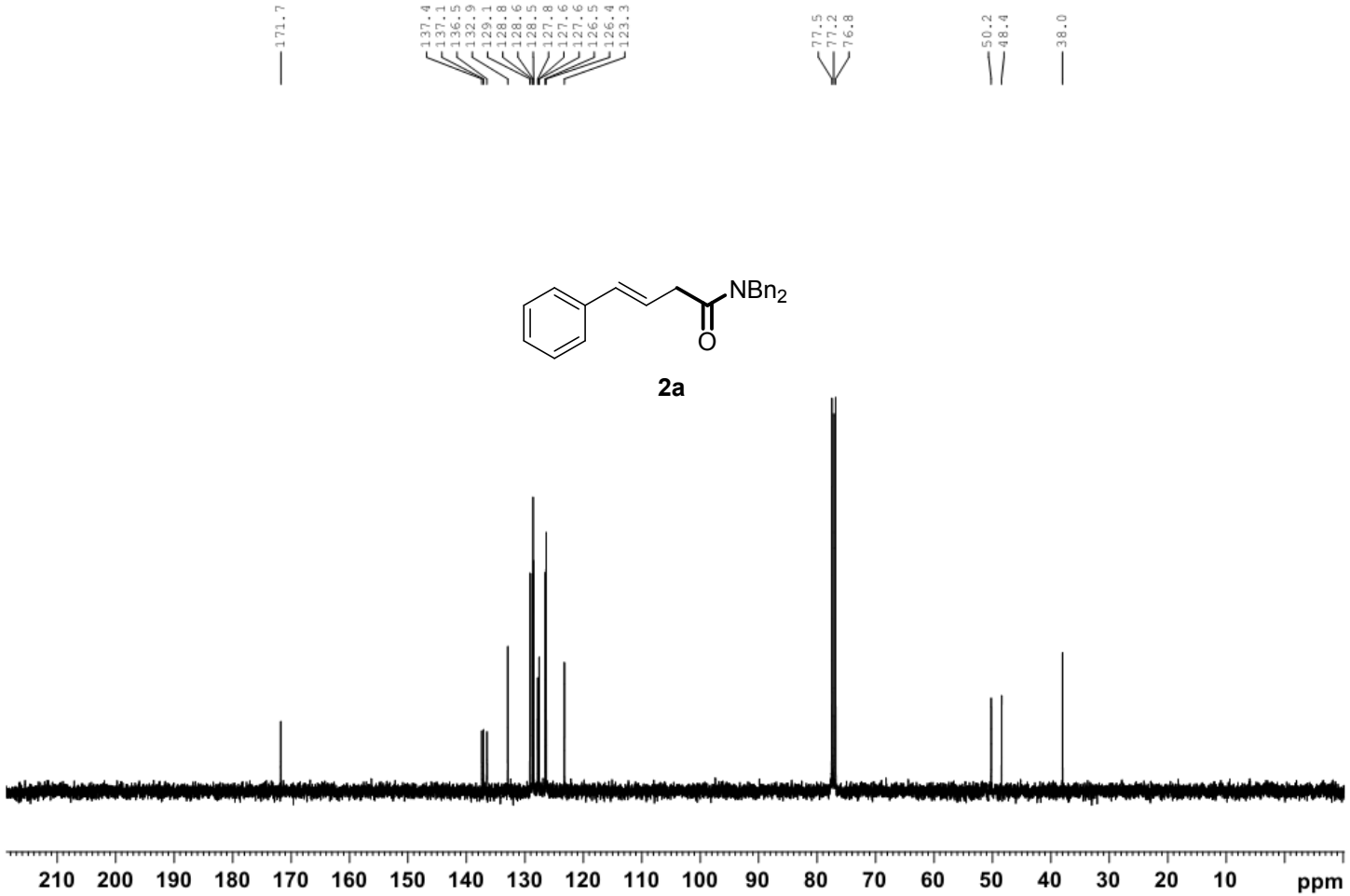
6 Copies for ^{1}H NMR and ^{13}C NMR of the products

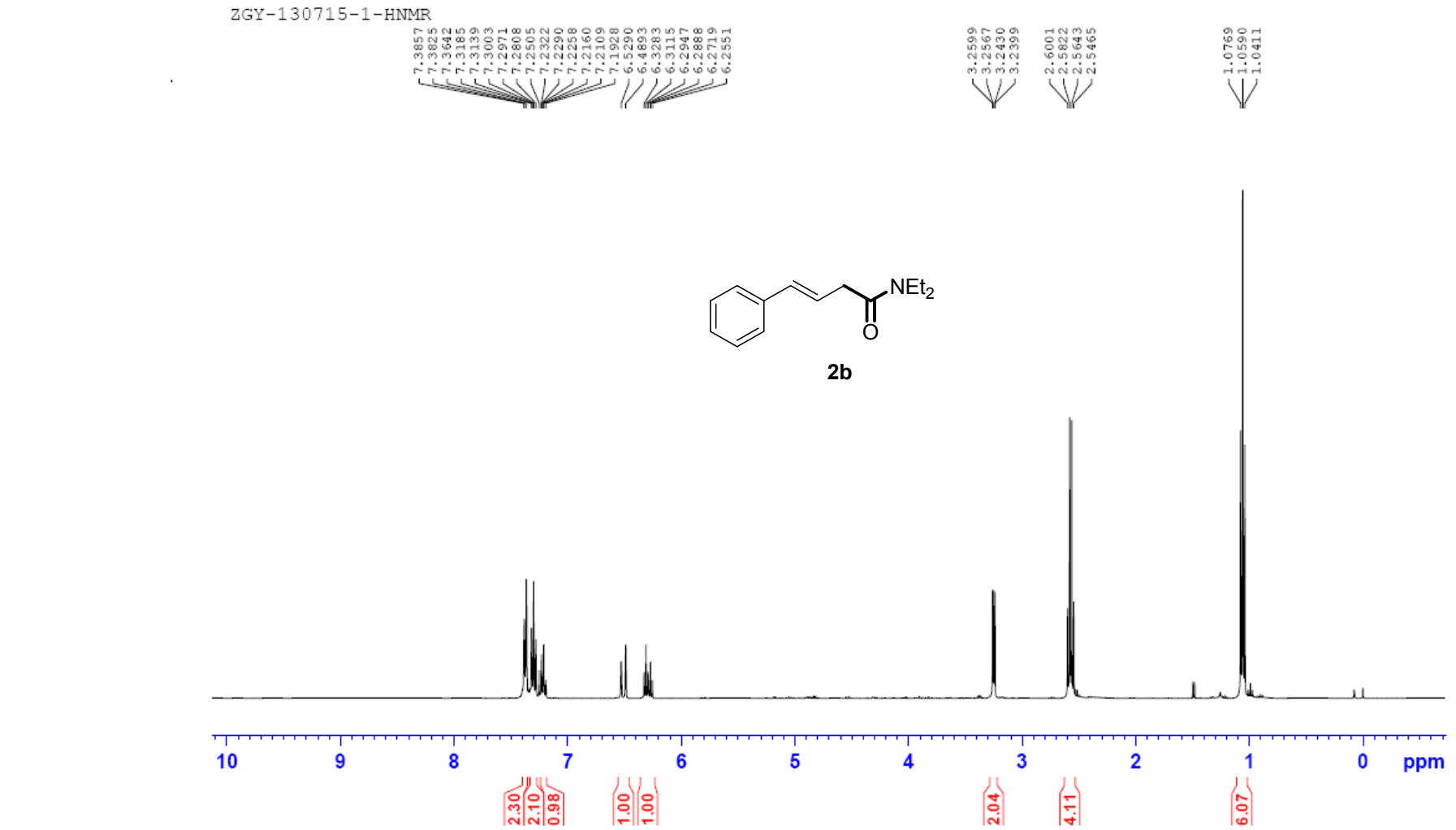
ZGY-130708-1-HNMR



2a

ZGY-130708-1-CNMR

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7.3825
7.3642
7.3185
7.3139
7.3003
7.2971
7.2808
7.2505
7.2322
7.2290
7.2258
7.2160
7.2109
7.1928
6.5290
6.4893
6.3283
6.3115
6.2947
6.2888
6.2719
6.2551
3.2599
3.2567
3.2430
3.2399
2.6001
2.5822
2.5643
2.5465
1.0769
1.0590
1.0411
NEt2
O
2b
10
9
8
7
6
5
4
3
2
1
0
ppm
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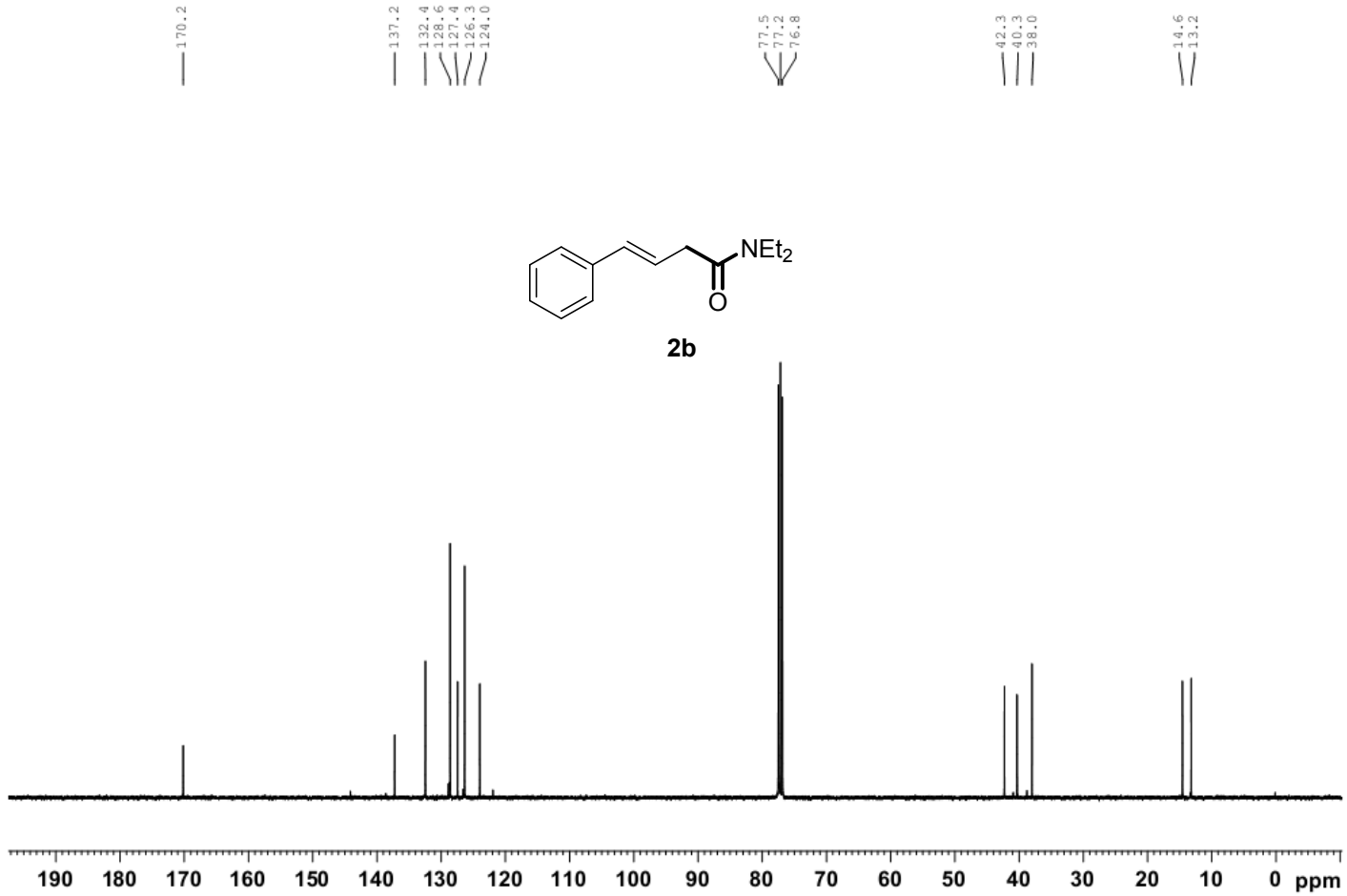
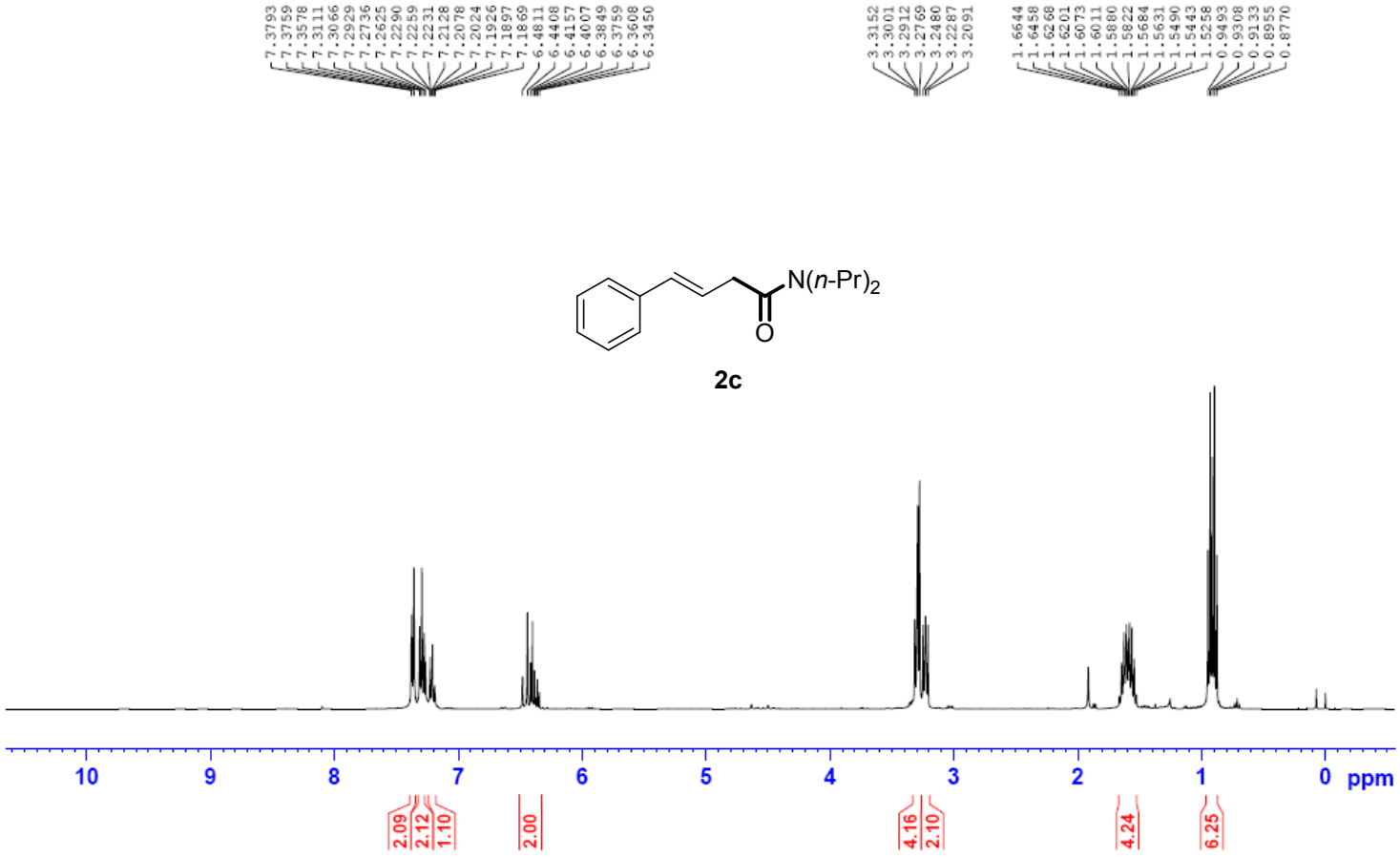
2.10
0.98
1.00
1.00
2.04
4.11
6.07

YH-140303-1-CNMR



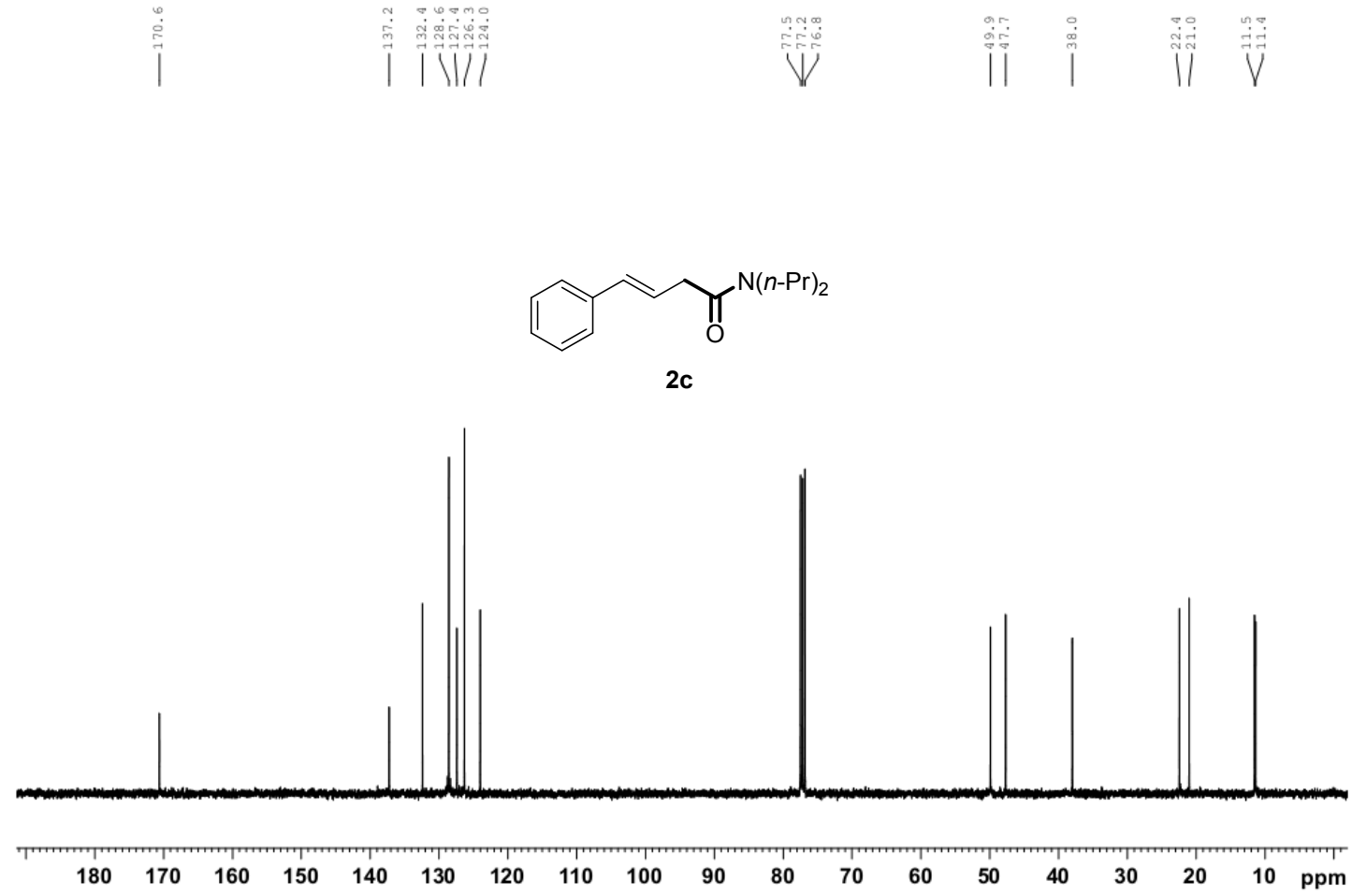
2b

ZGY-130715-2-HNMR

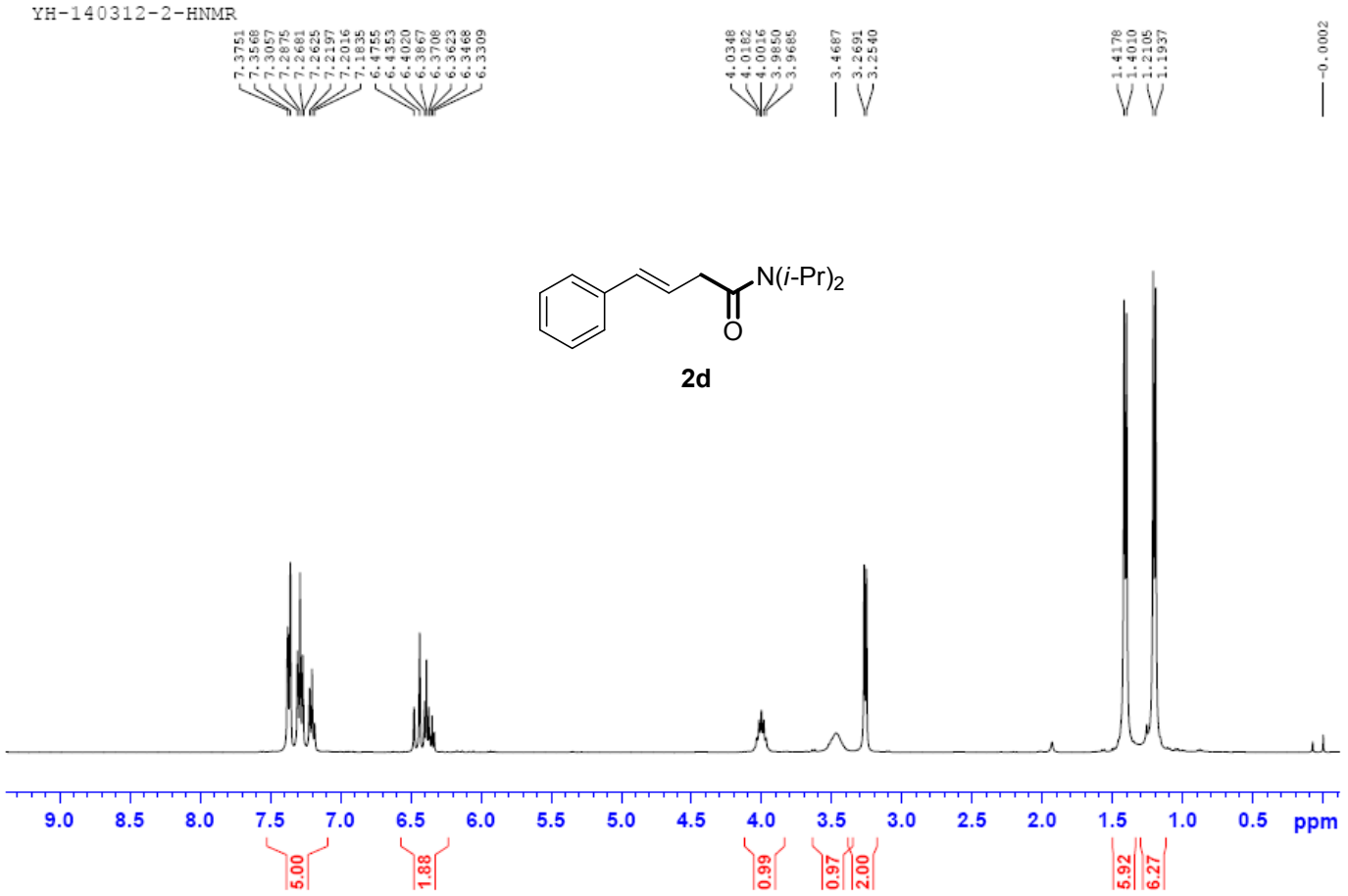


2c

ZGY-130715-2-CNMR



2c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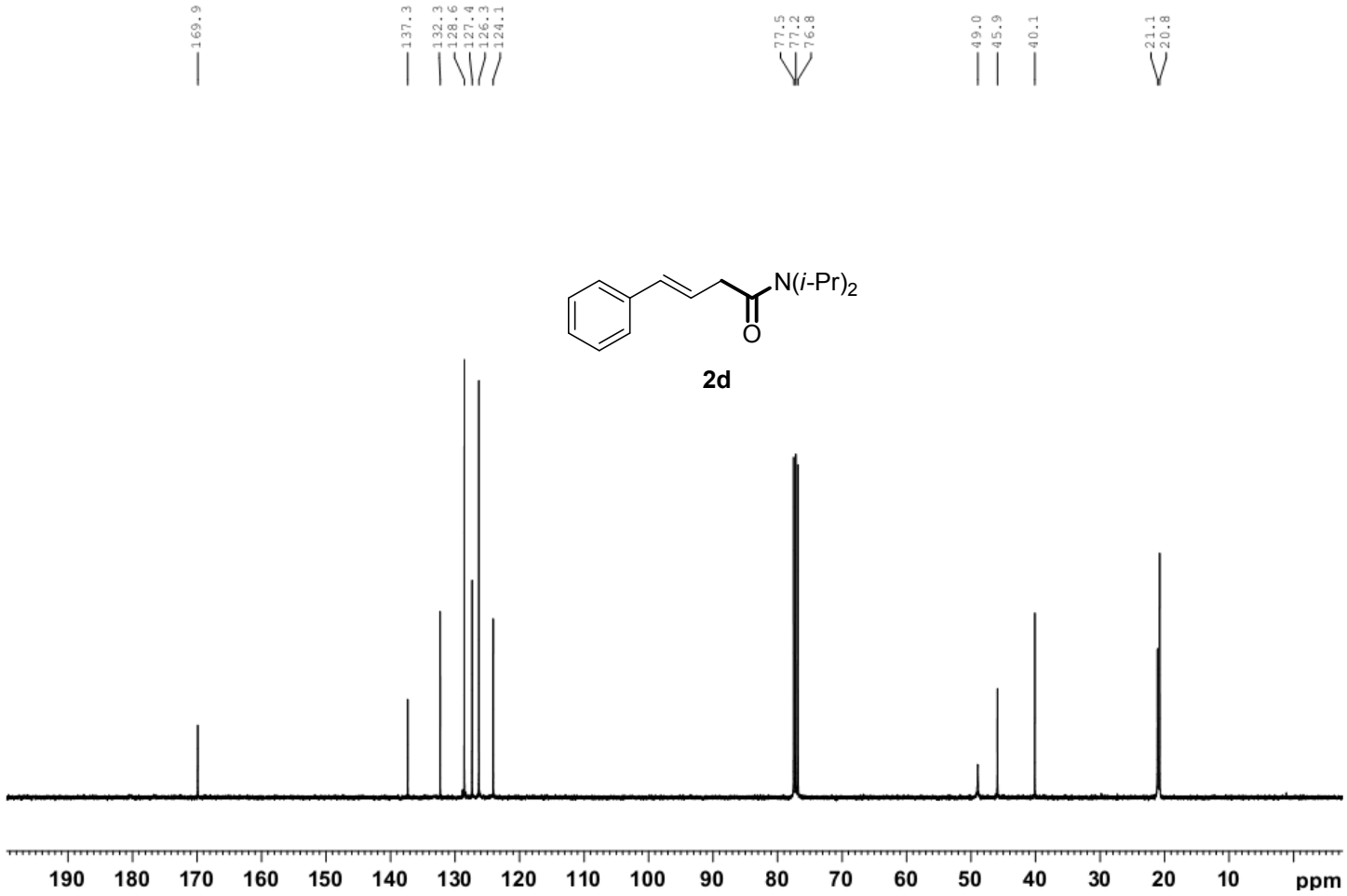
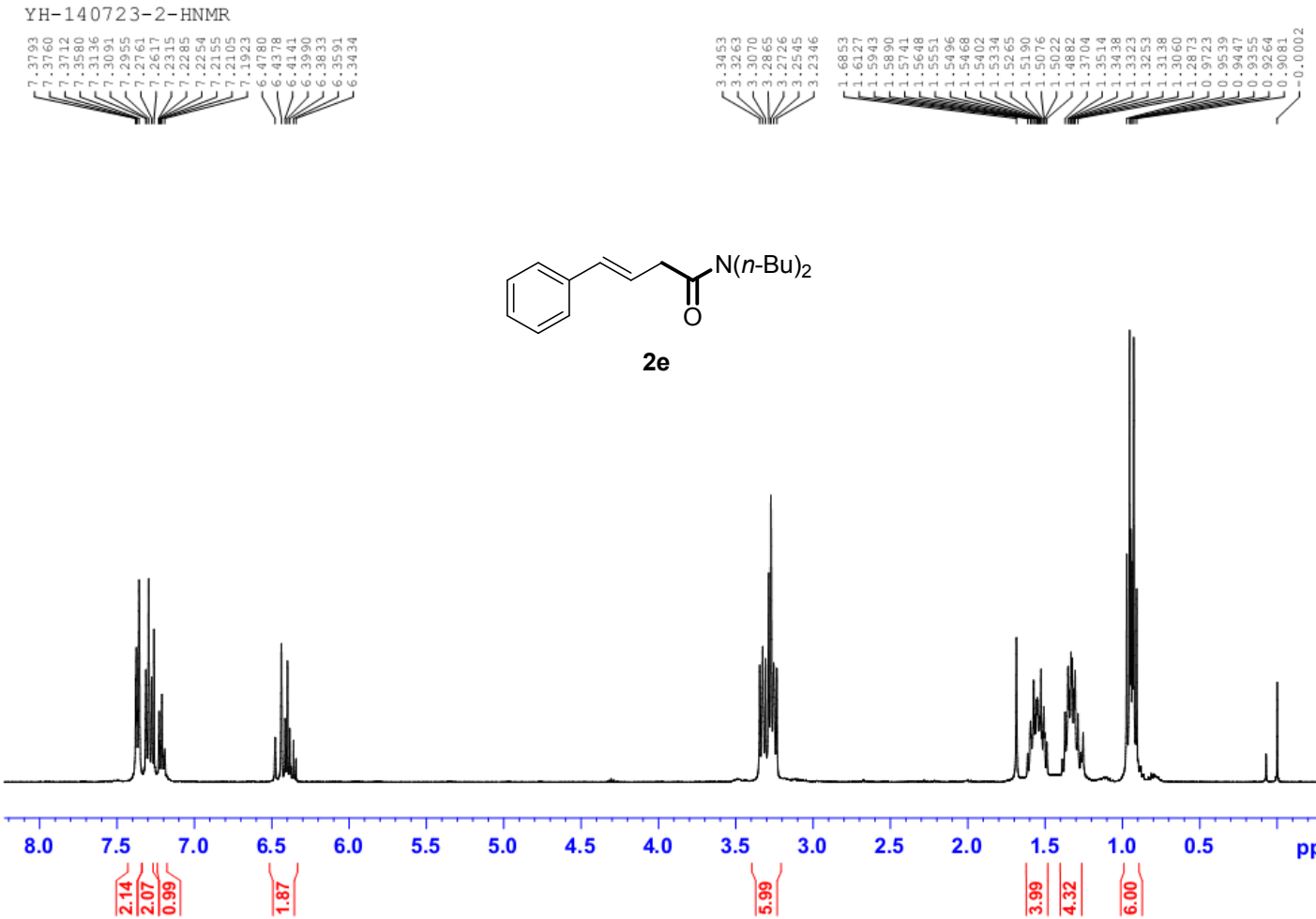
7.3057
7.2875
7.2681
7.2625
7.2197
7.2016
7.1835
6.4755
6.4353
6.4020
6.3867
6.3708
6.3623
6.3468
6.3309
4.0348
4.0182
4.0016
3.9850
3.9685
3.4687
3.2691
3.2540
1.4178
1.4010
1.2106
1.1937
-0.0002
N(i-Pr)2
O
2d
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.00
1.88
0.99
0.97
2.00
5.92
6.27

YH-140312-2-CNMR



2d


YH-140723-2-HNMR
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7.3760
7.3712
7.3580
7.3136
7.3091
7.2955
7.2761
7.2617
7.2315
7.2285
7.2254
7.2155
7.2105
7.1923
6.4780
6.4378
6.4141
6.3990
6.3833
6.3591
6.3434
3.3453
3.3263
3.3070
3.2865
3.2726
3.2545
3.2346
1.6853
1.6127
1.5943
1.5890
1.5741
1.5648
1.5551
1.5496
1.5468
1.5402
1.5334
1.5265
1.5190
1.5076
1.5022
1.4882
1.3704
1.3514
1.3438
1.3323
1.3253
1.3138
1.3060
1.2873
0.9723
0.9539
0.9447
0.9355
0.9264
0.9081
-0.0002
N(n-Bu)2
O
2e
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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1.5
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0.5
ppm
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0.99
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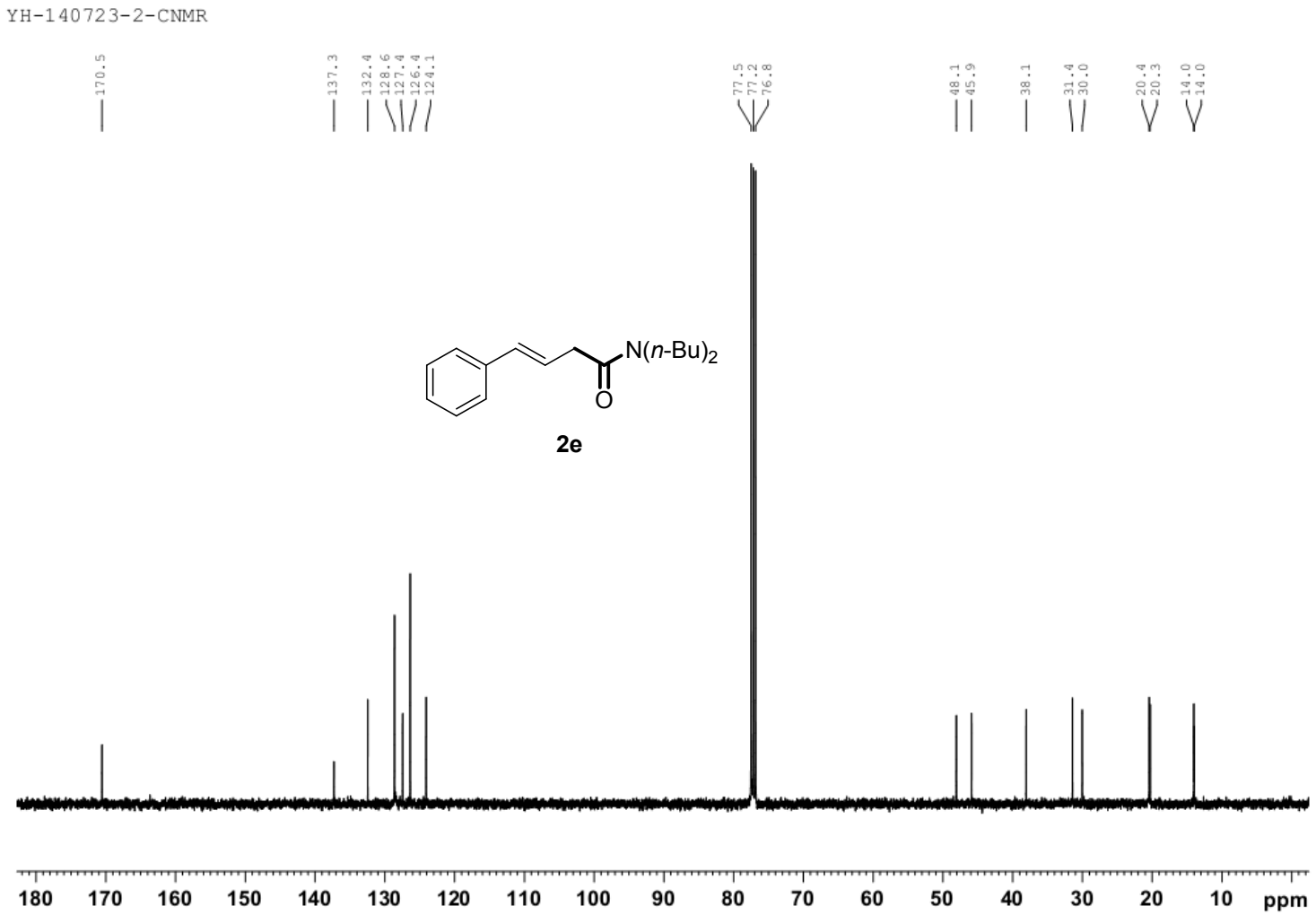
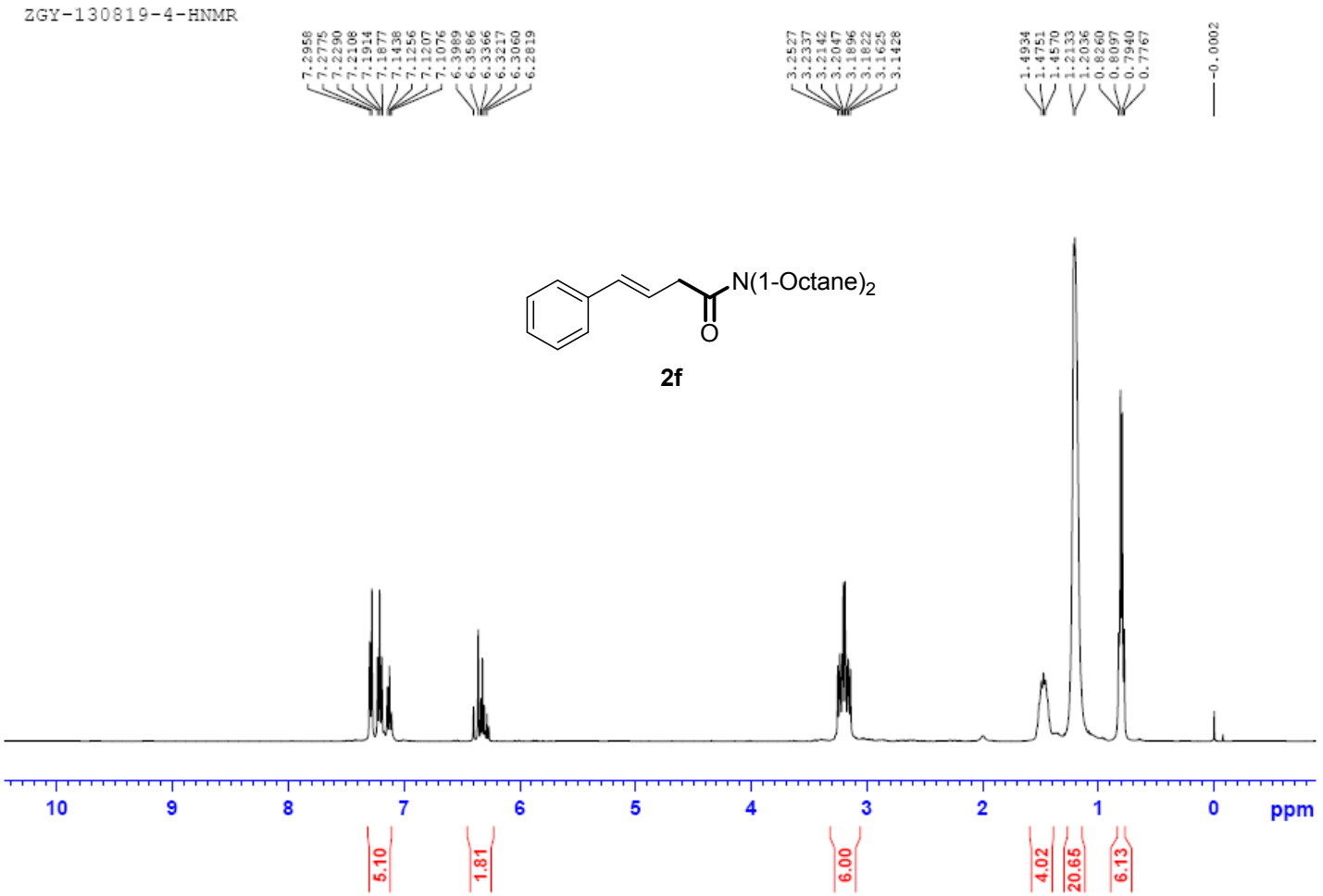
4.32
6.00


YH-140723-2-CNMR
170.5
137.3
132.4
128.6
127.4
126.4
124.1
77.5
77.2
76.8
48.1
45.9
38.1
31.4
30.0
20.4
20.3
14.0
14.0
N(n-Bu)2
O
2e
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.1076
6.3989
6.3586
6.3366
6.3217
6.3060
6.2819
3.2527
3.2337
3.2142
3.2047
3.1896
3.1822
3.1625
3.1428
1.4934
1.4751
1.4570
1.2133
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0.8097
0.7940
0.7767
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2f
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9
8
7
6
5
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3
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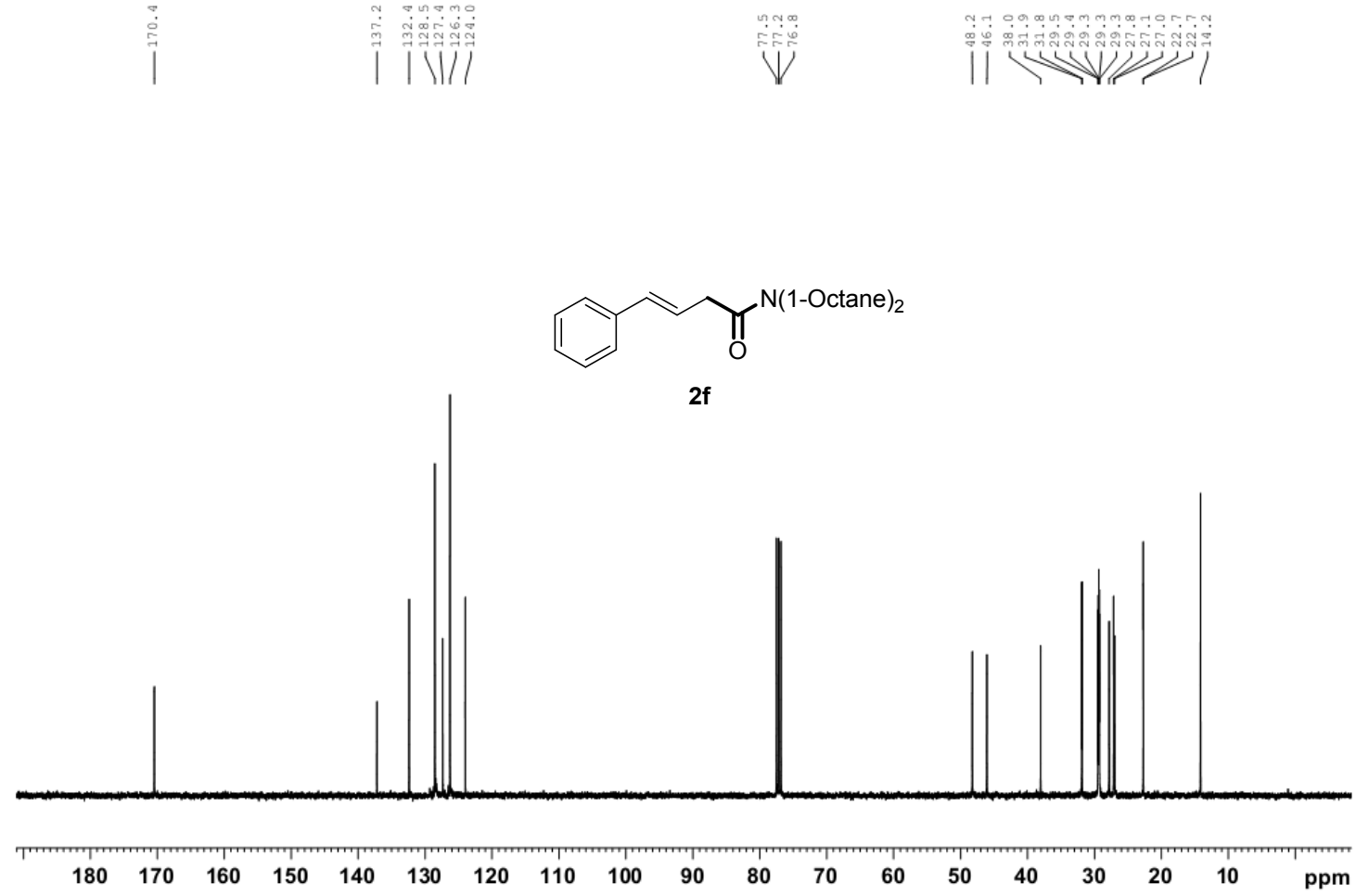
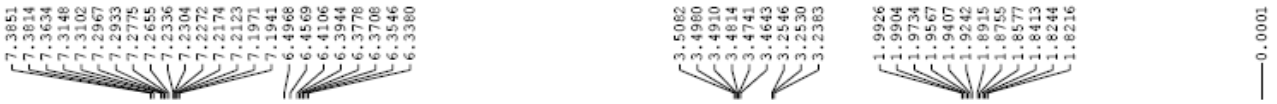
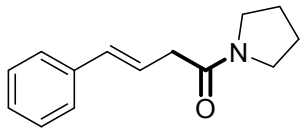
20.65
6.13

ZGY-130819-4-CNMR

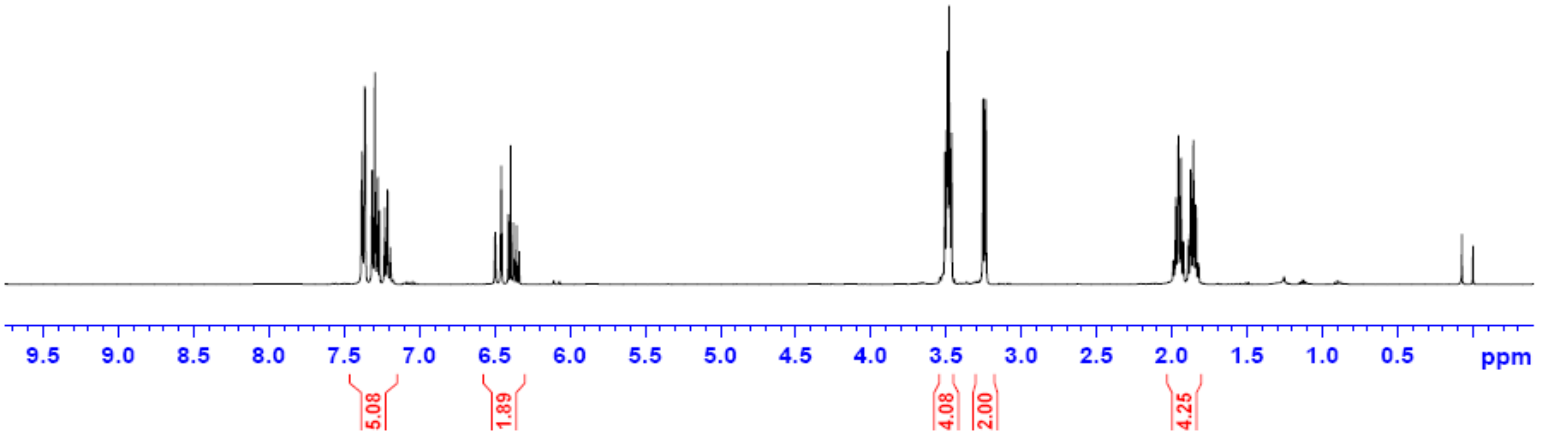


YH-140326-1-HNMR





2g



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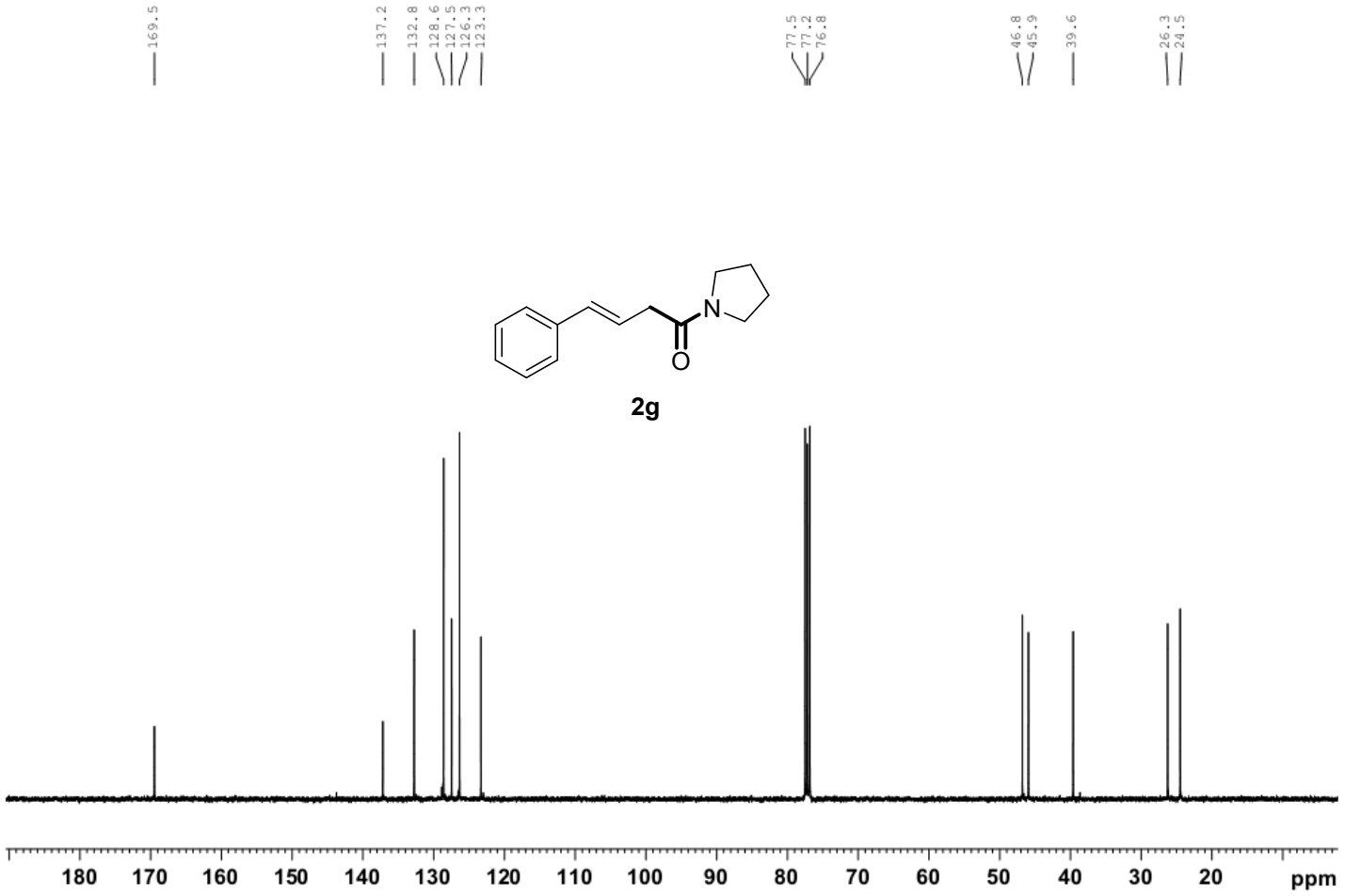
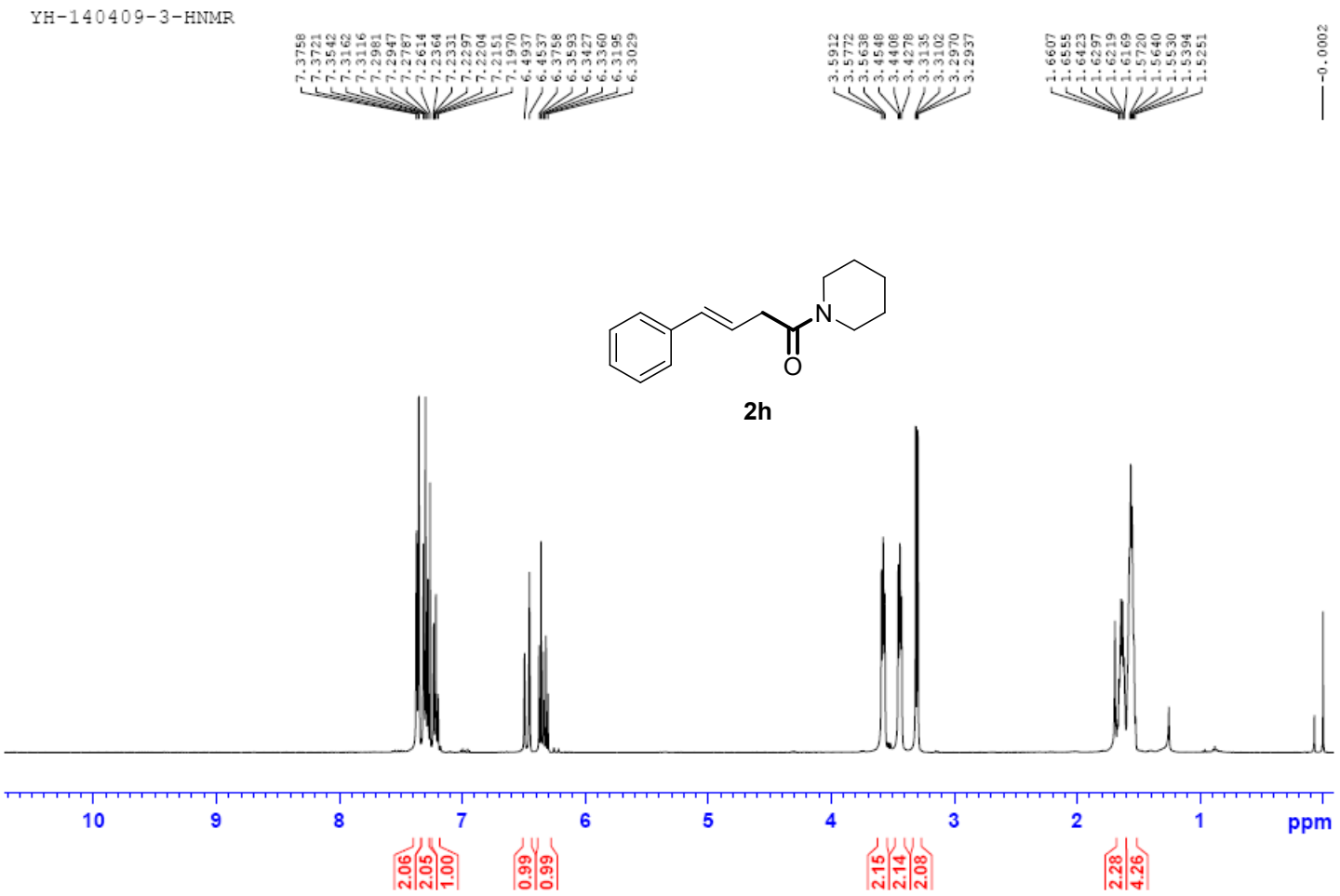


2g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6.3029
3.5912
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3.3135
3.3102
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7
6
5
4
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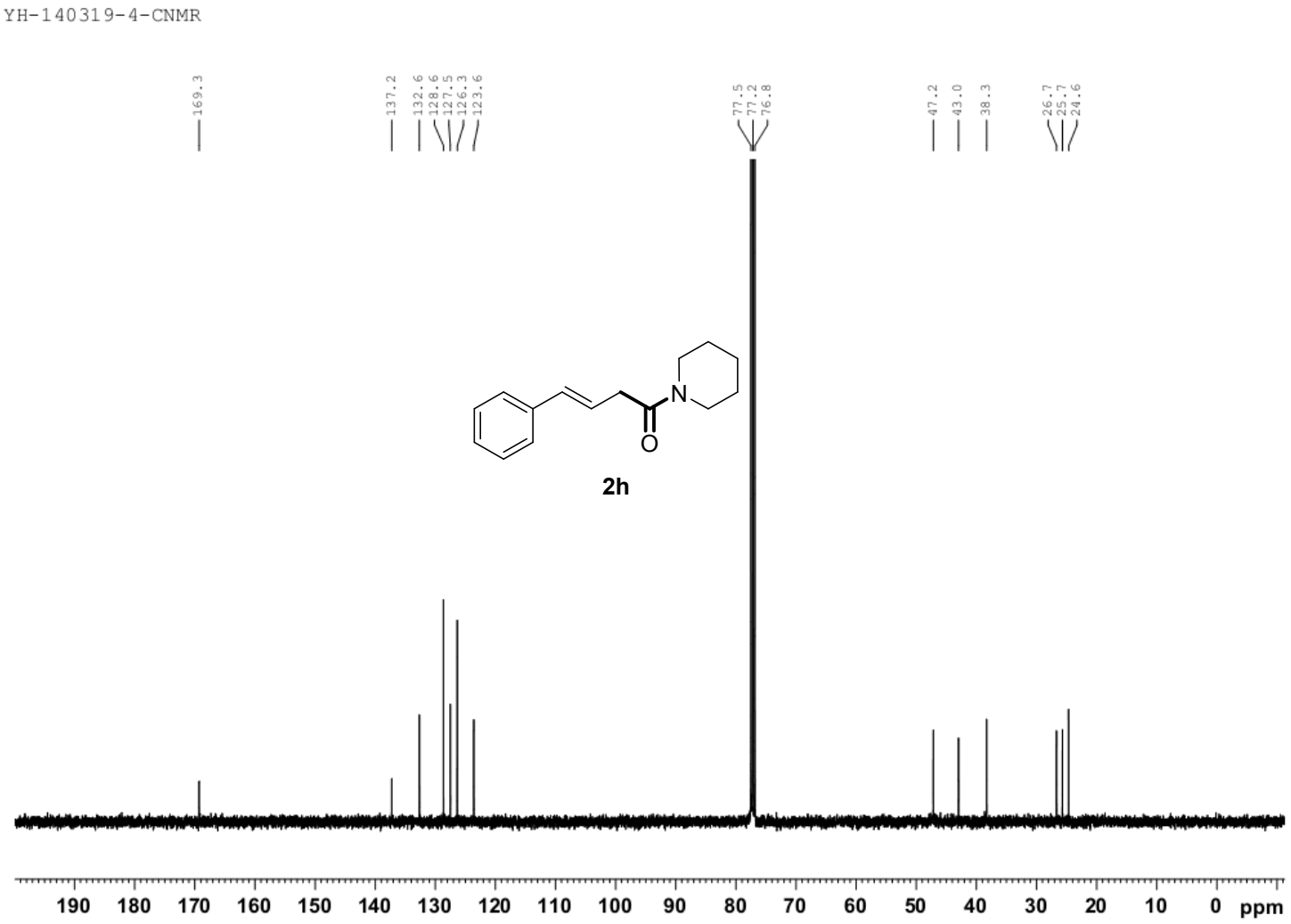
YH-140319-4-CNMR
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123.6
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77.2
76.8
47.2
43.0
38.3
26.7
25.7
24.6
N
O
2h
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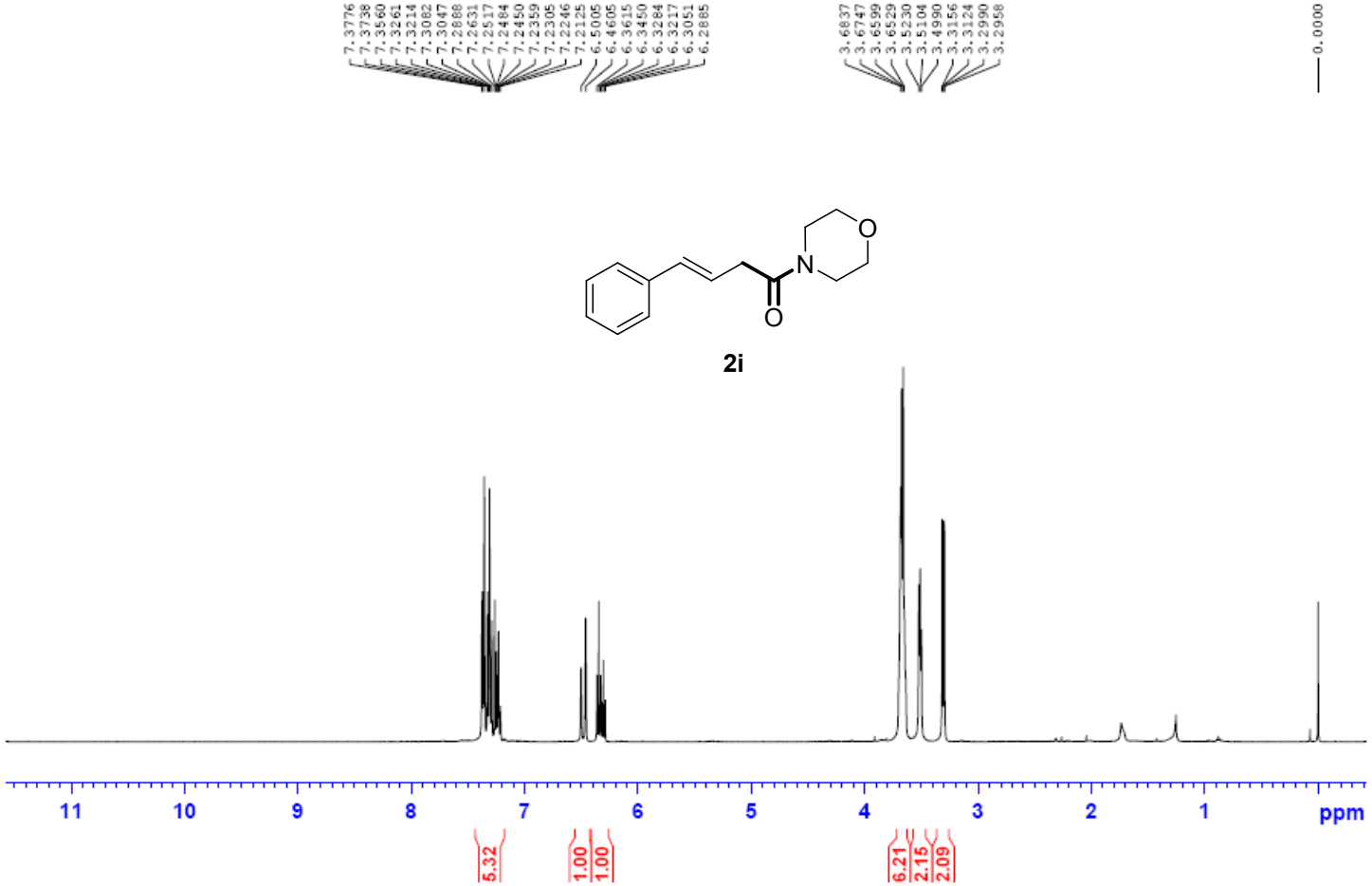
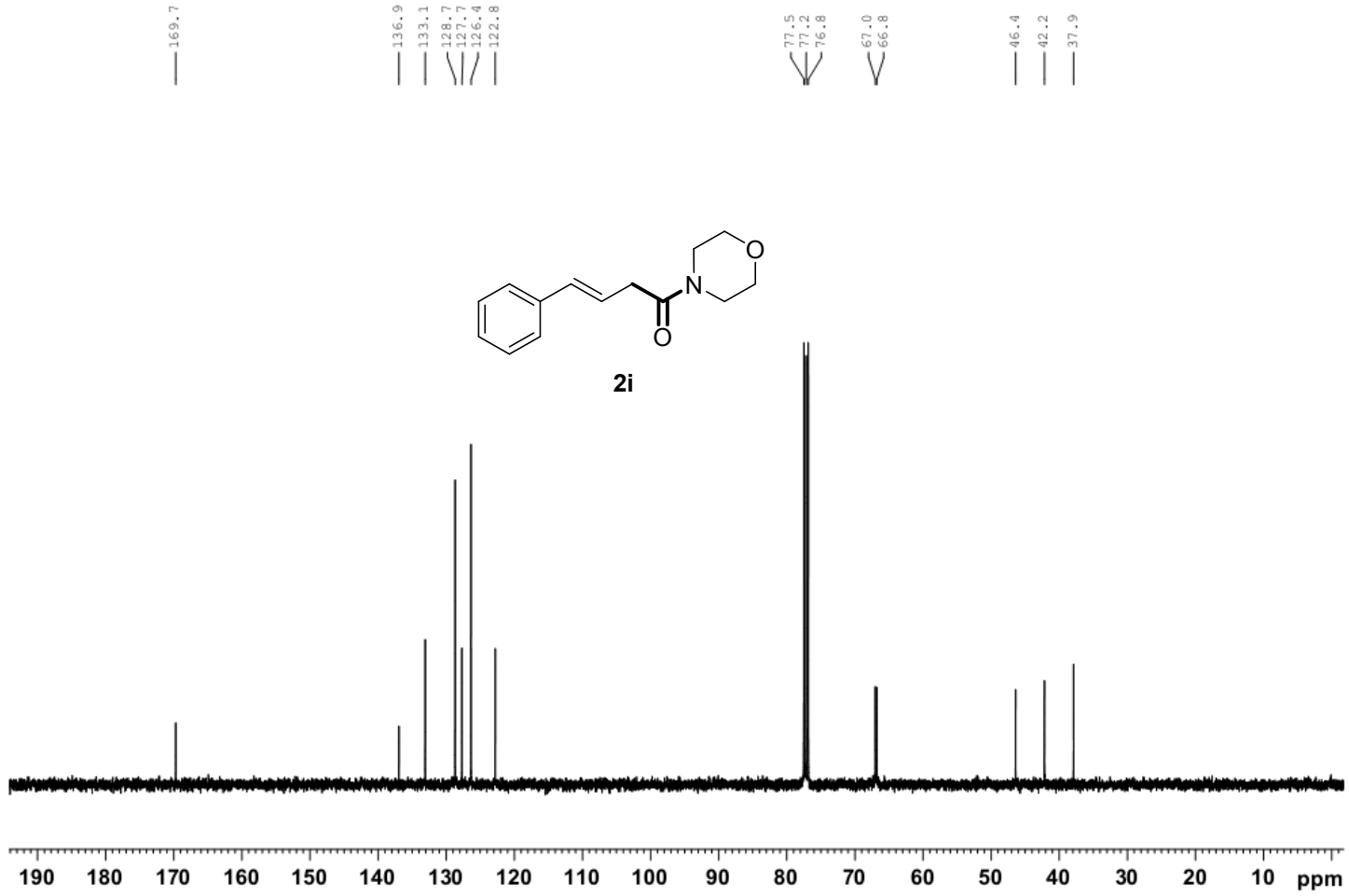
ZGY-130715-1-HNMR

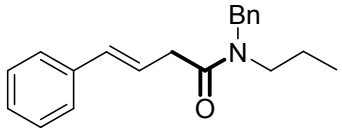


2i

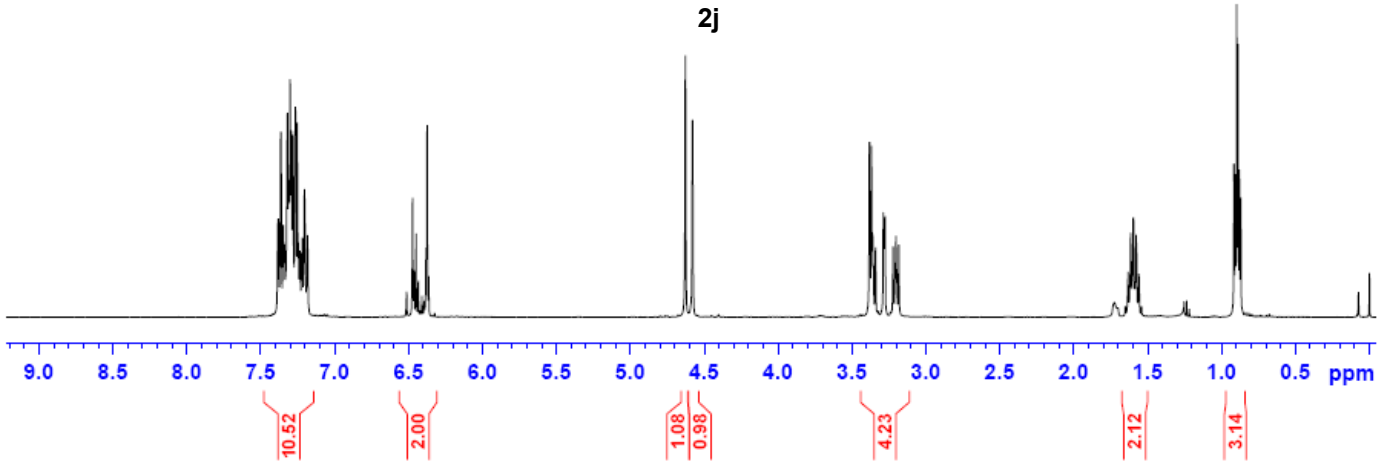
ZGY-130715-1-CNMR



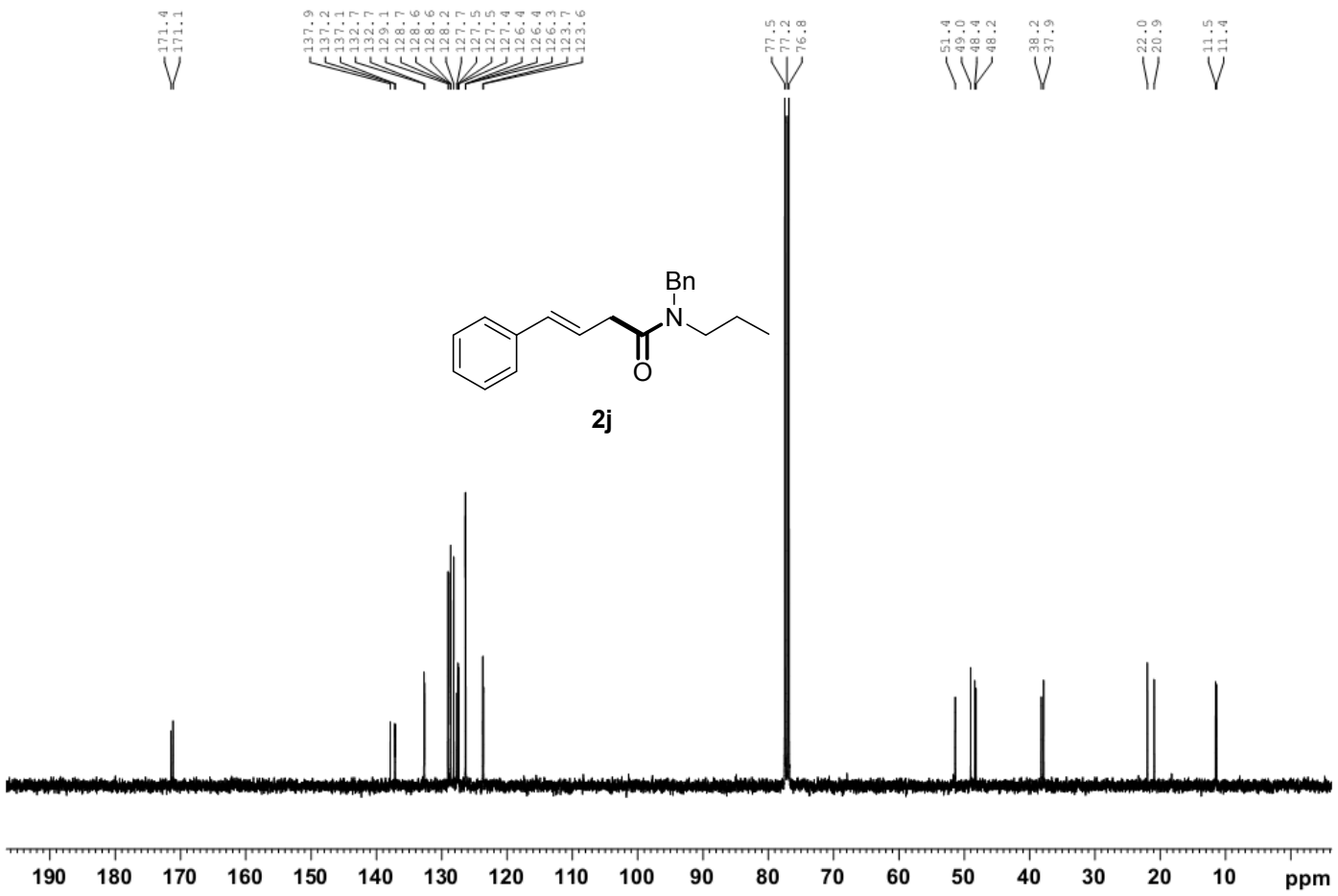
YH-140319-2-HNMR

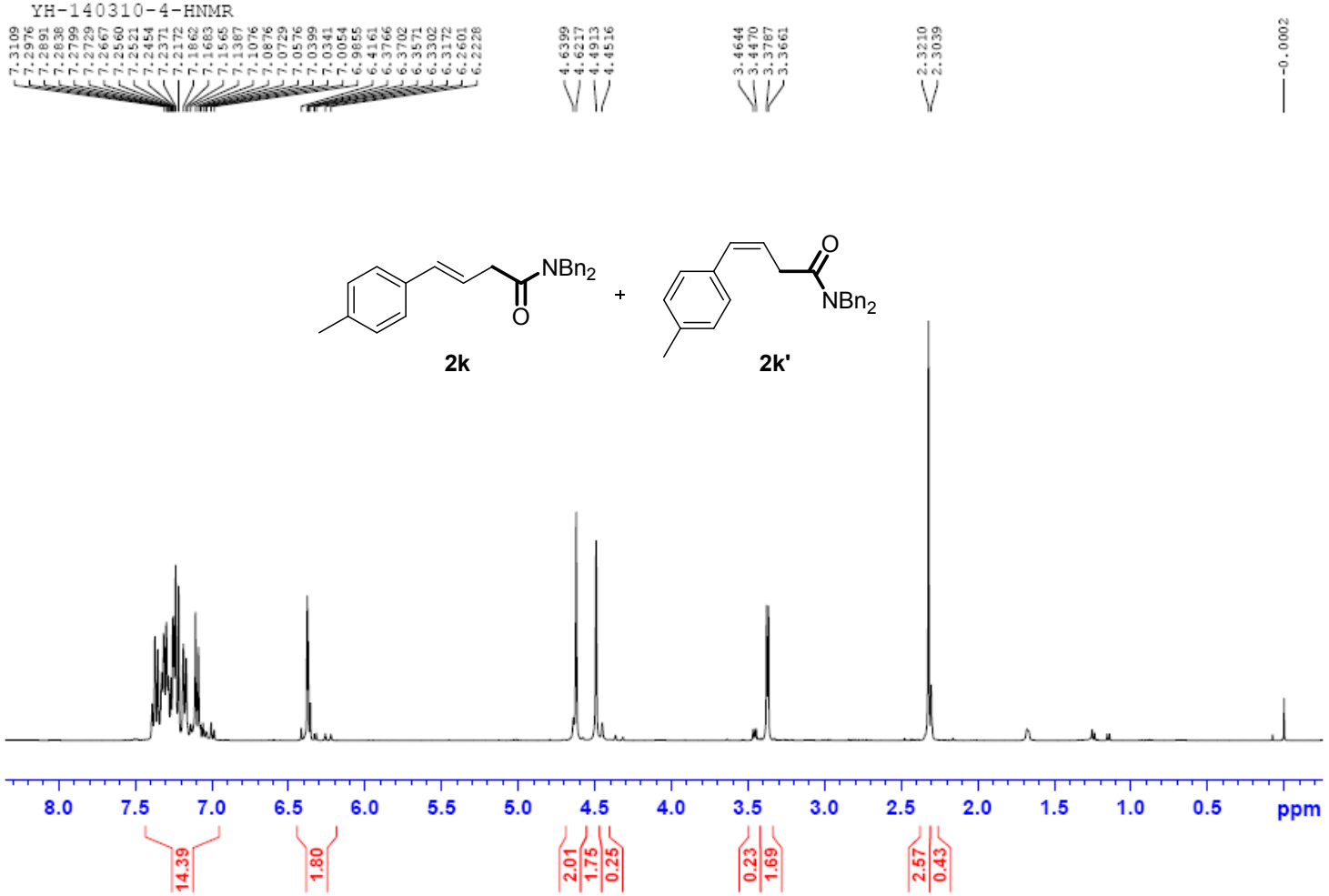


2j

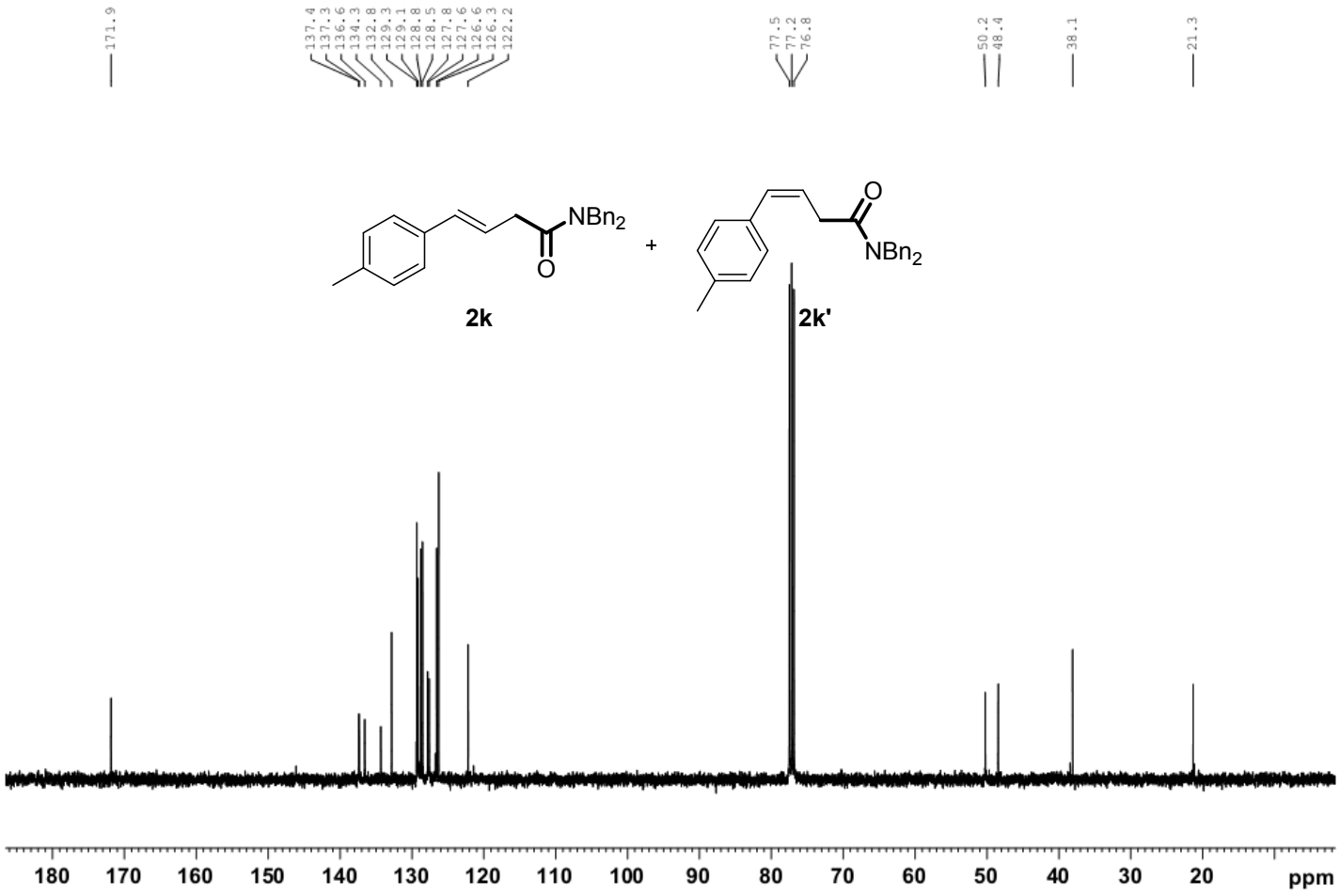


YH-140319-2-CNMR

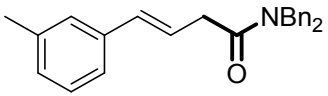



YH-140310-4-HNMR
7.3109
7.2976
7.2891
7.2838
7.2799
7.2729
7.2667
7.2560
7.2521
7.2454
7.2371
7.2172
7.1862
7.1683
7.1565
7.1387
7.1076
7.0876
7.0729
7.0576
7.0399
7.0341
7.0054
6.9855
6.4161
6.3766
6.3702
6.3571
6.3302
6.3172
6.2601
6.2228
4.6399
4.6217
4.4913
4.4516
3.4644
3.4470
3.3787
3.3661
2.3210
2.3039
-0.0002
NBn2
O
2k
+
O
NBn2
2k'
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
ppm
14.39
1.80
2.01
1.75
0.25
0.23
1.69
2.57
0.43

YH-140310-4-CNMR



YH-140319-6-HNMR



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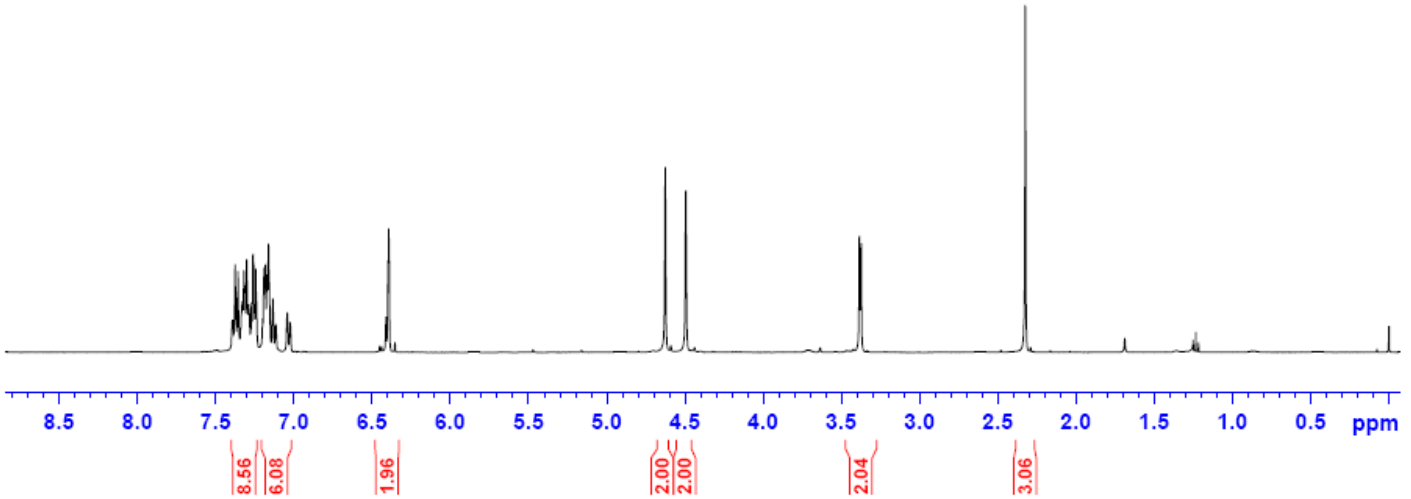
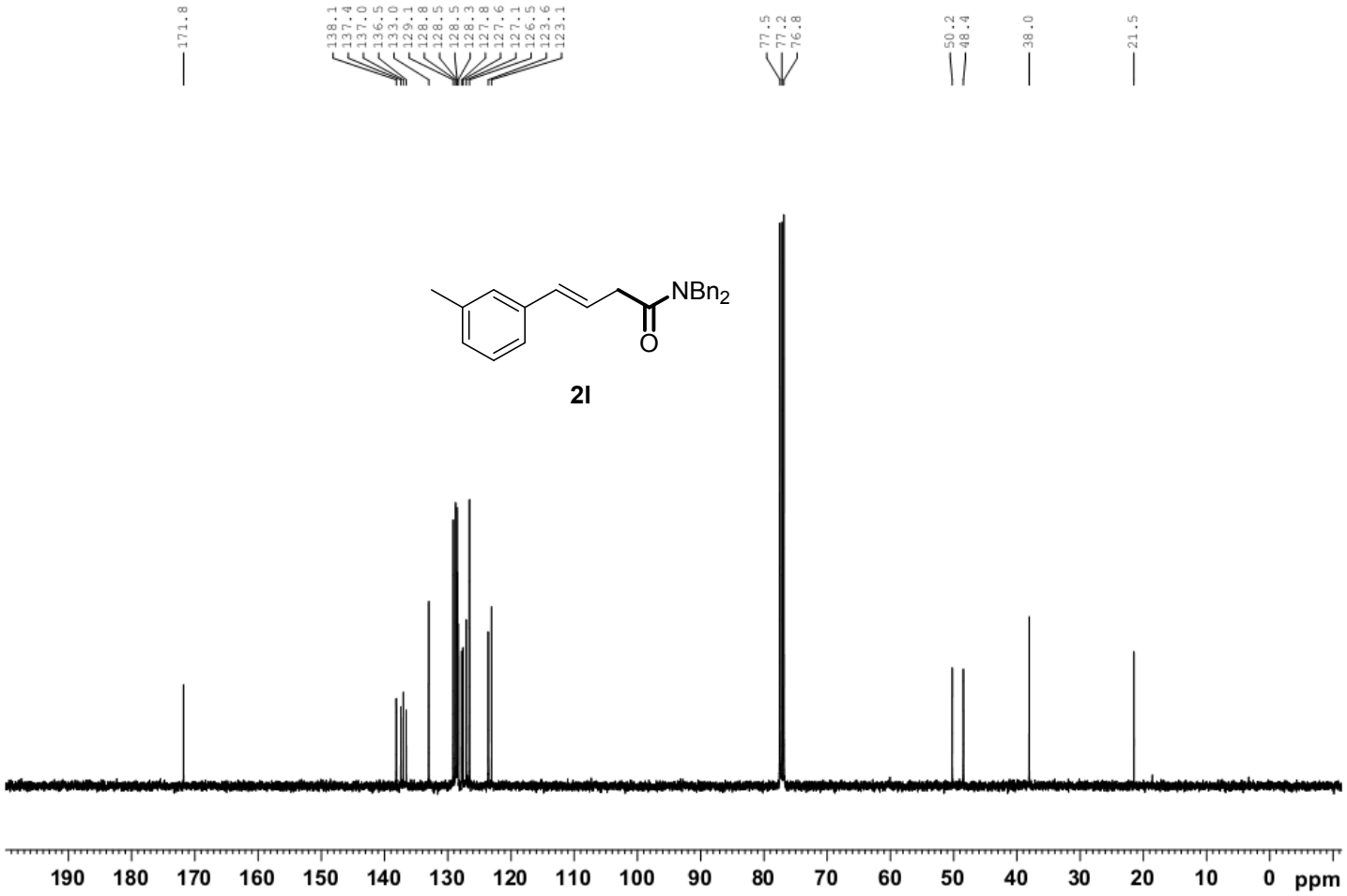


YH-140319-6-CNMR



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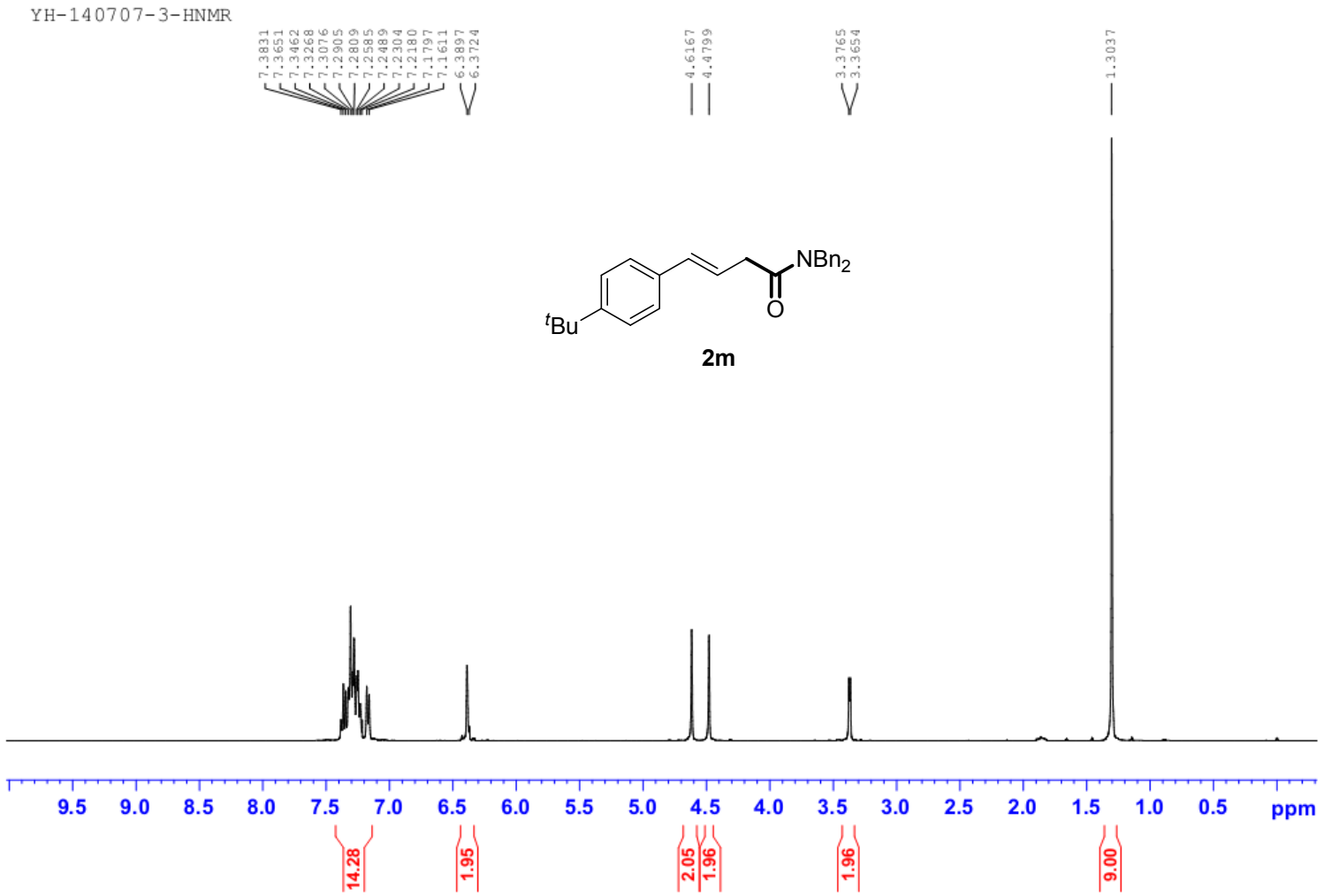
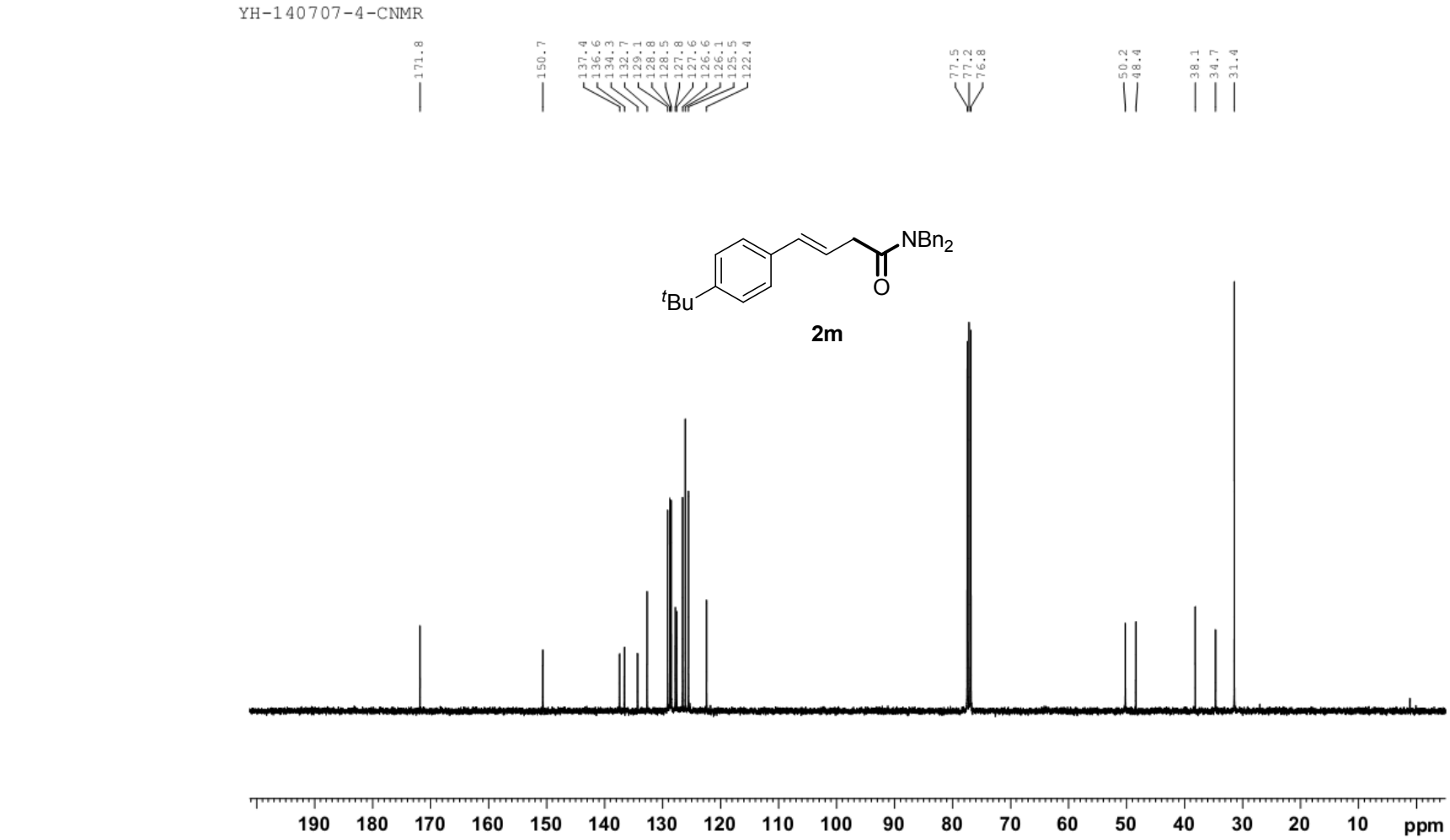
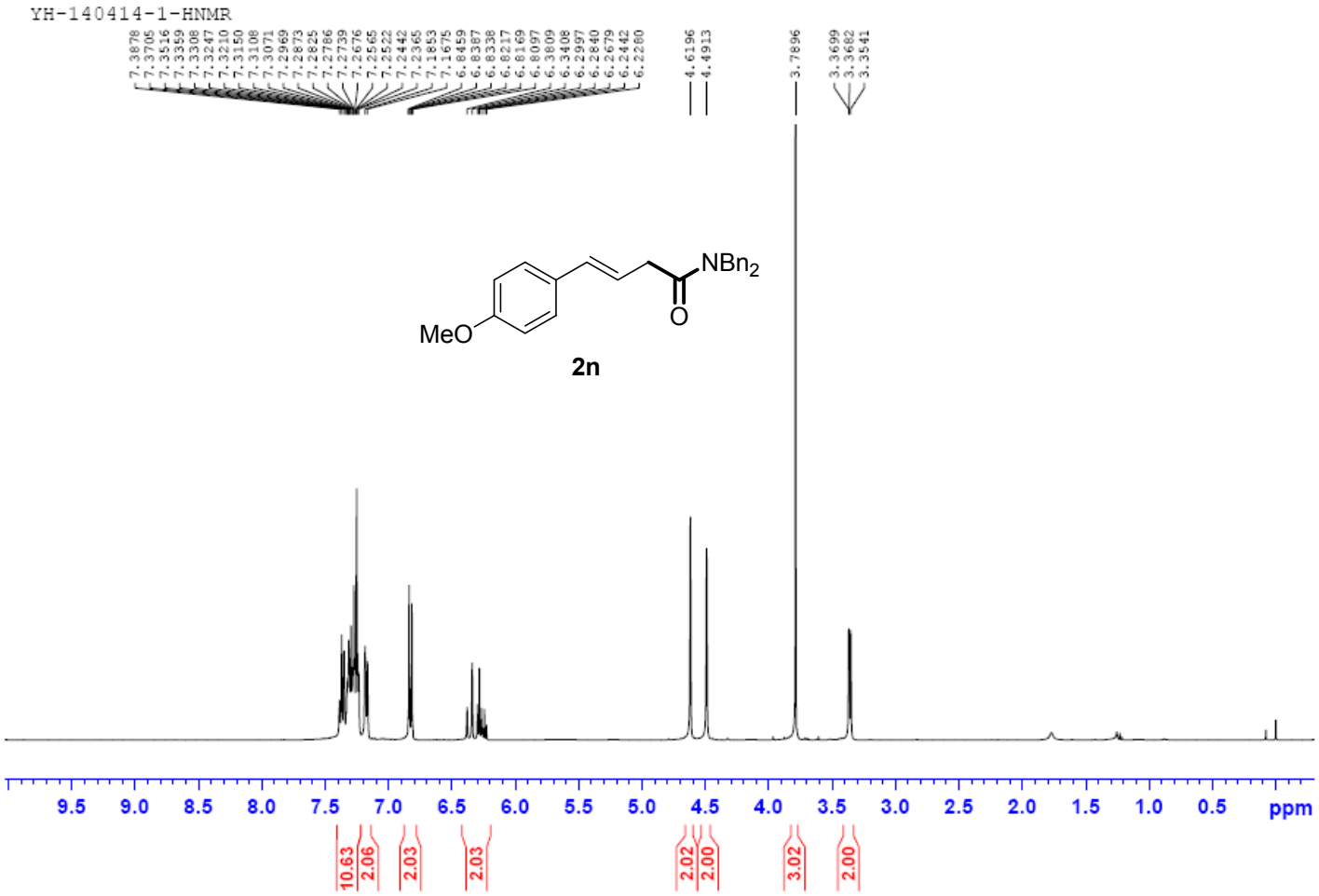

YH-140707-3-HNMR
7.3831
7.3651
7.3462
7.3268
7.3076
7.2905
7.2809
7.2585
7.2489
7.2304
7.2180
7.1797
7.1611
6.3897
6.3724
4.6167
4.4799
3.3765
3.3654
1.3037
NBn2
O
tBu
2m
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
14.28
1.95
2.05
1.96
1.96
9.00


YH-140707-4-CNMR
171.8
150.7
137.4
136.6
134.3
132.7
129.1
128.8
128.5
127.8
127.6
126.6
126.1
125.5
122.4
77.5
77.2
76.8
50.2
48.4
38.1
34.7
31.4
NBn2
O
tBu
2m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


YH-140414-1-HNMR
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7.3705
7.3516
7.3359
7.3308
7.3247
7.3210
7.3150
7.3108
7.3071
7.2969
7.2873
7.2825
7.2786
7.2739
7.2676
7.2565
7.2522
7.2442
7.2365
7.1853
7.1675
6.8459
6.8387
6.8338
6.8217
6.8169
6.8097
6.3809
6.3408
6.2997
6.2840
6.2679
6.2442
6.2280
4.6196
4.4913
3.7896
3.3699
3.3682
3.3541
NBn2
O
MeO
2n
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
ppm
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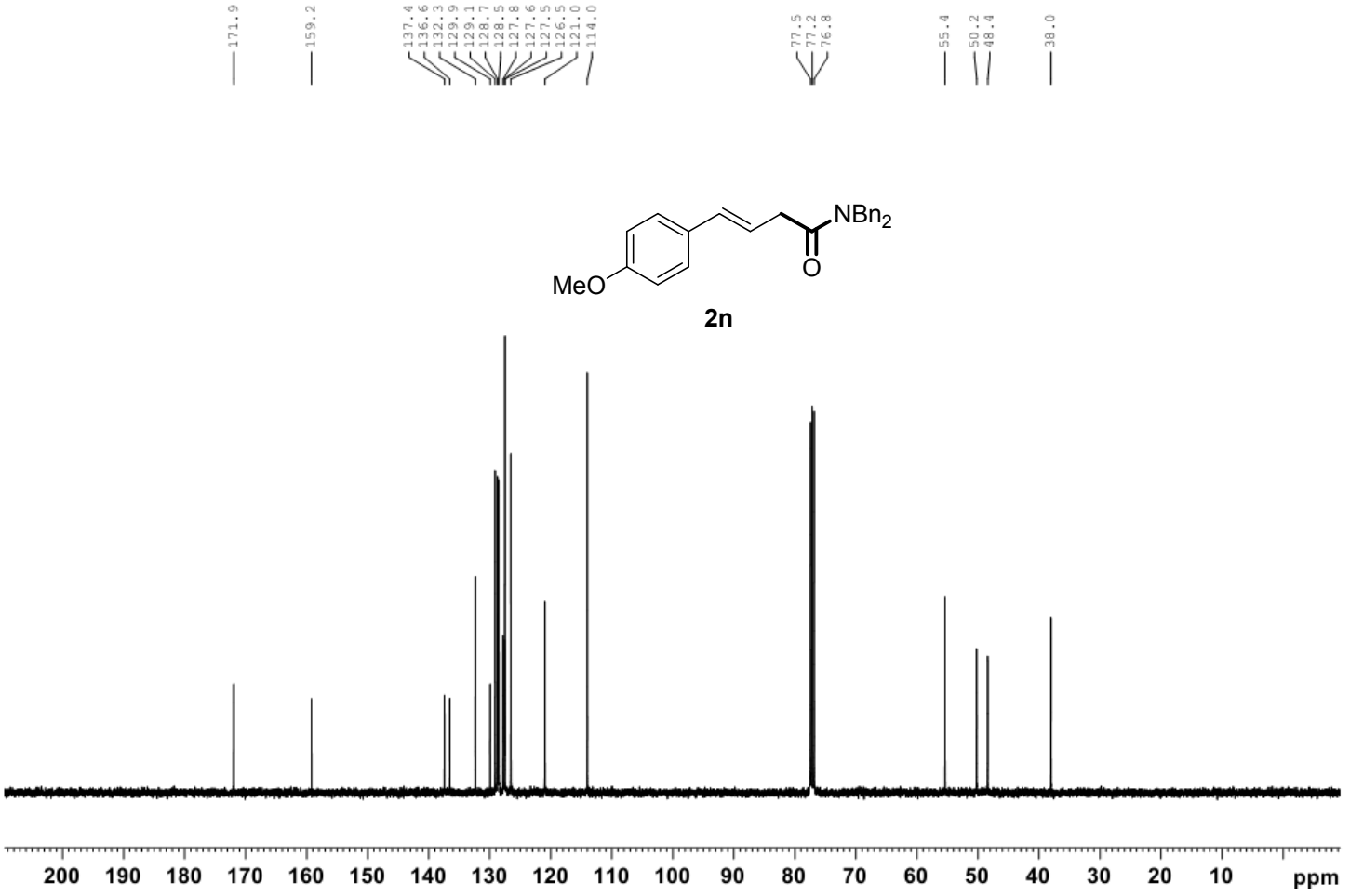
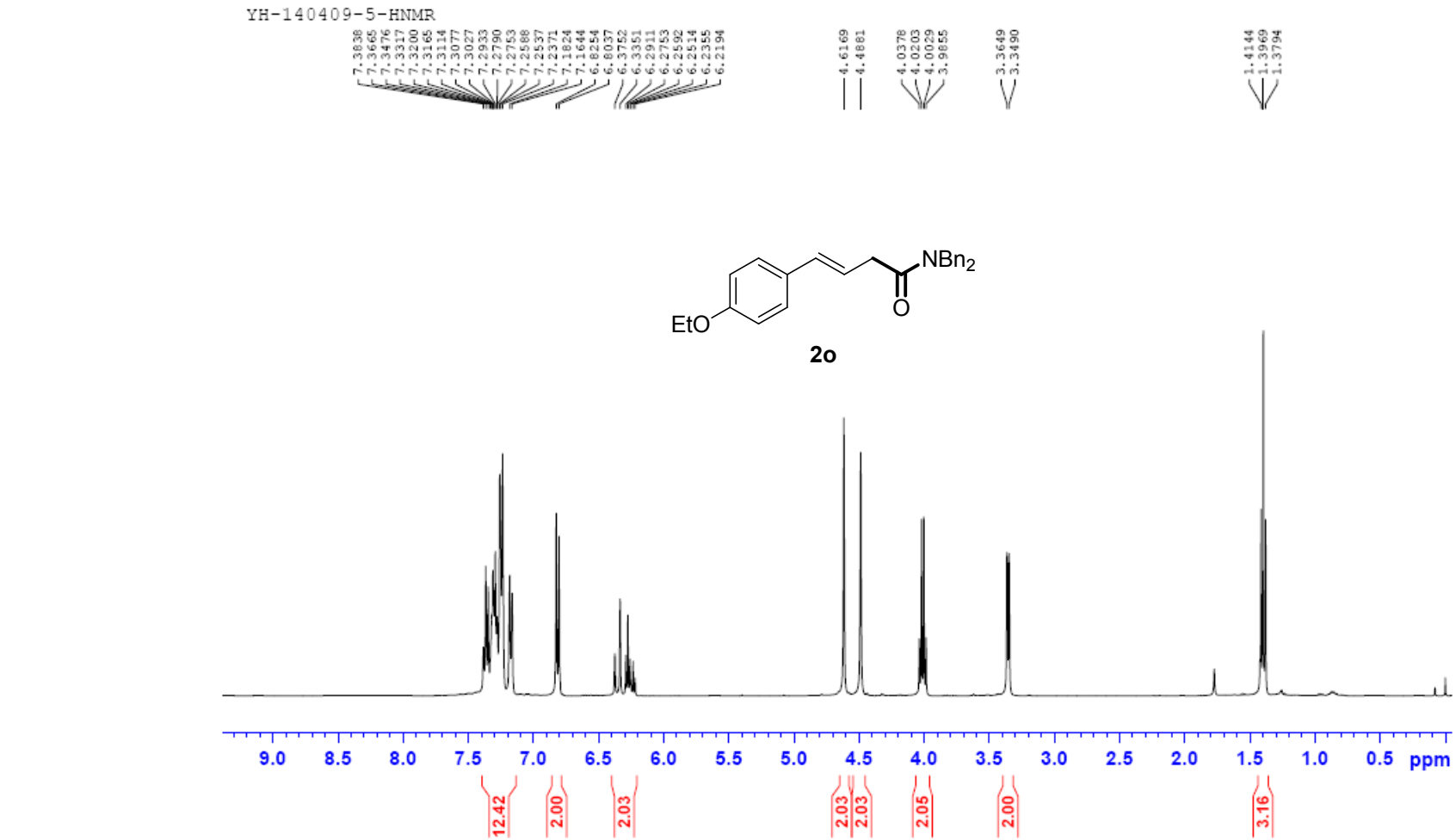
2.06
2.03
2.03
2.02
2.00
3.02
2.00

YH-140414-1-CNMR




YH-140409-5-HNMR
7.3838
7.3665
7.3476
7.3317
7.3200
7.3165
7.3114
7.3077
7.3027
7.2933
7.2790
7.2753
7.2588
7.2537
7.2371
7.1824
7.1644
6.8254
6.8037
6.3752
6.3351
6.2911
6.2753
6.2592
6.2514
6.2355
6.2194
4.6169
4.4861
4.0376
4.0203
4.0029
3.9855
3.3649
3.3490
1.4144
1.3969
1.3794
NBn2
O
EtO
2o
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
ppm
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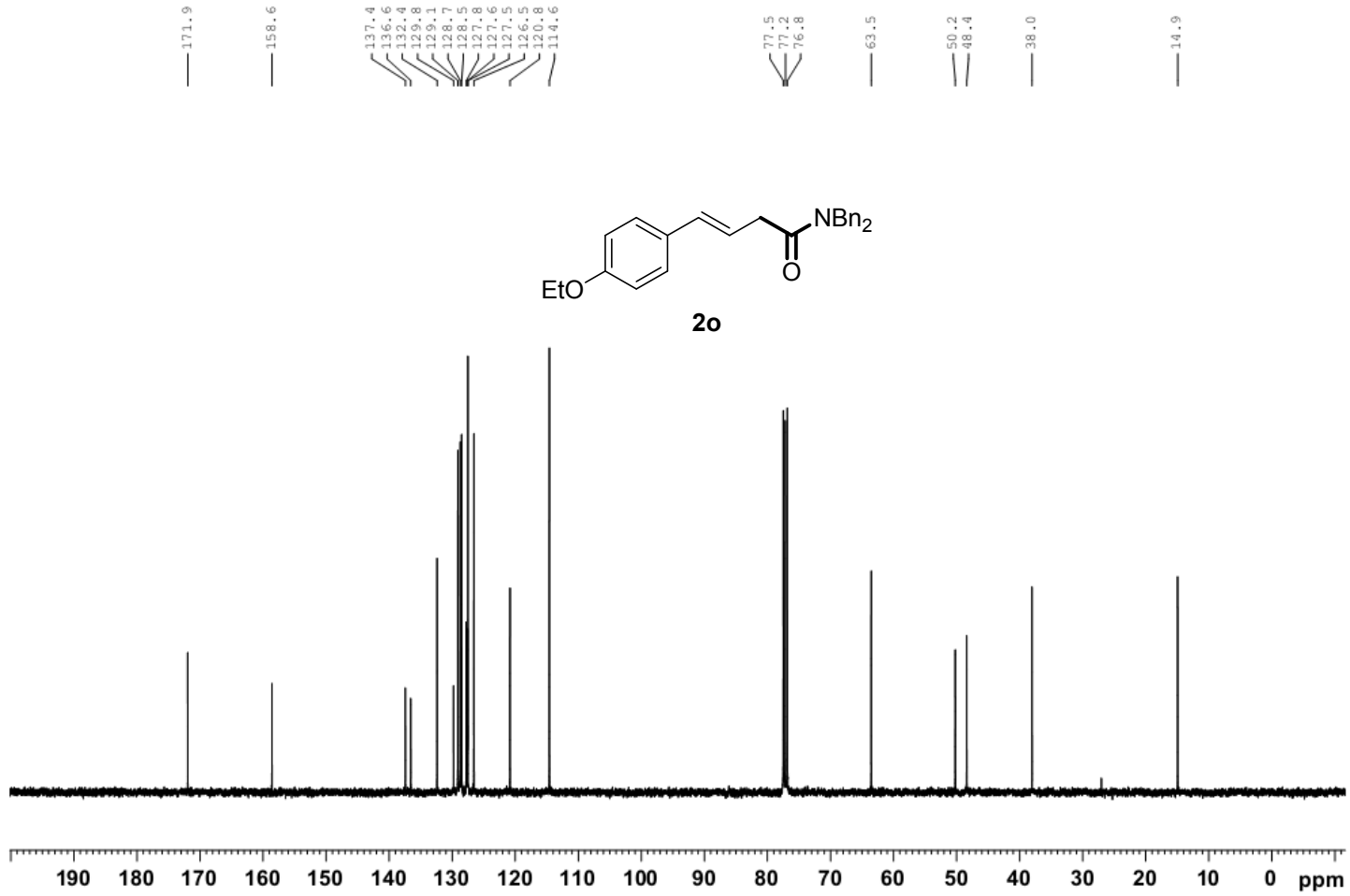
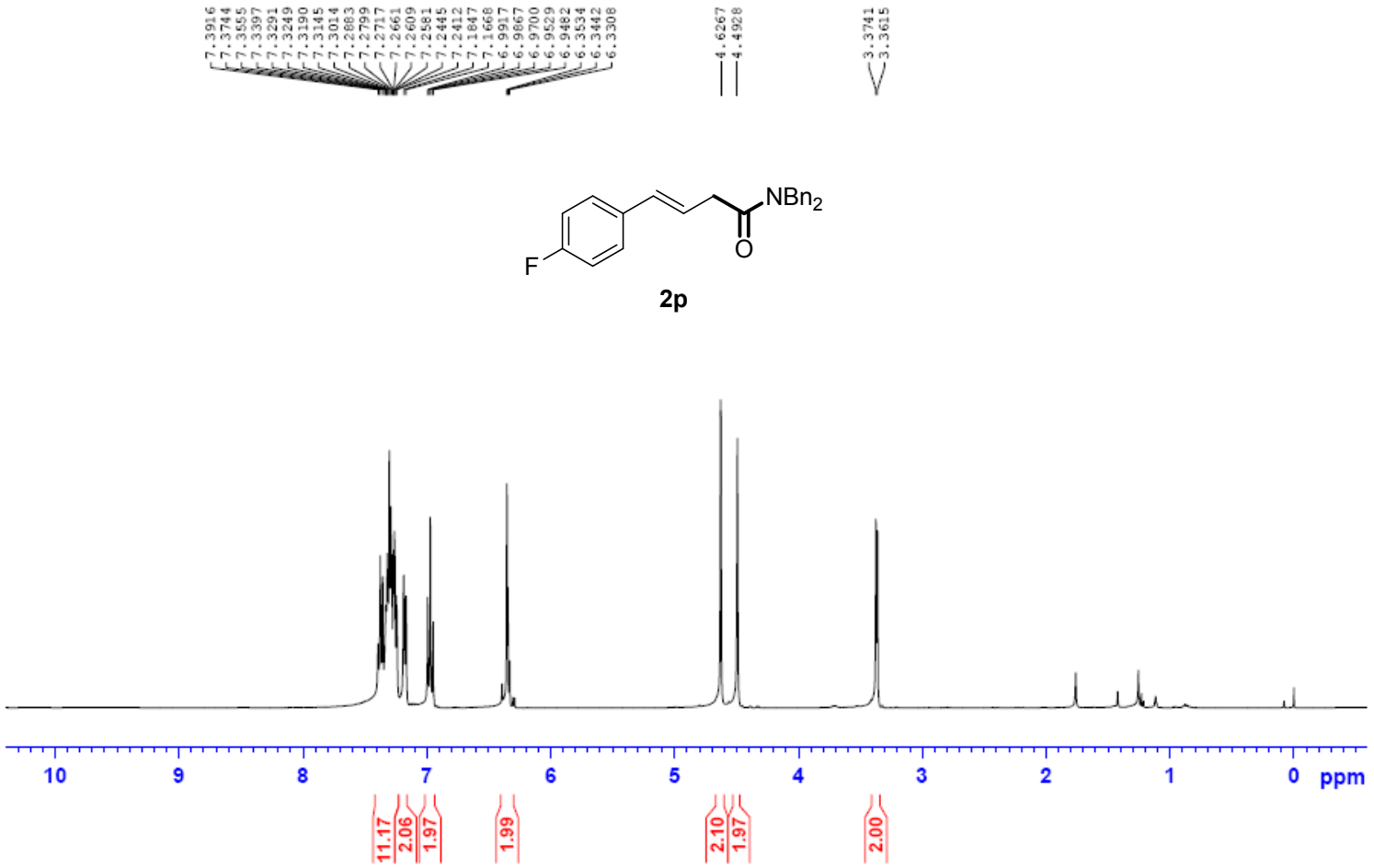
2.00
2.03
2.03
2.03
2.05
2.00
3.16

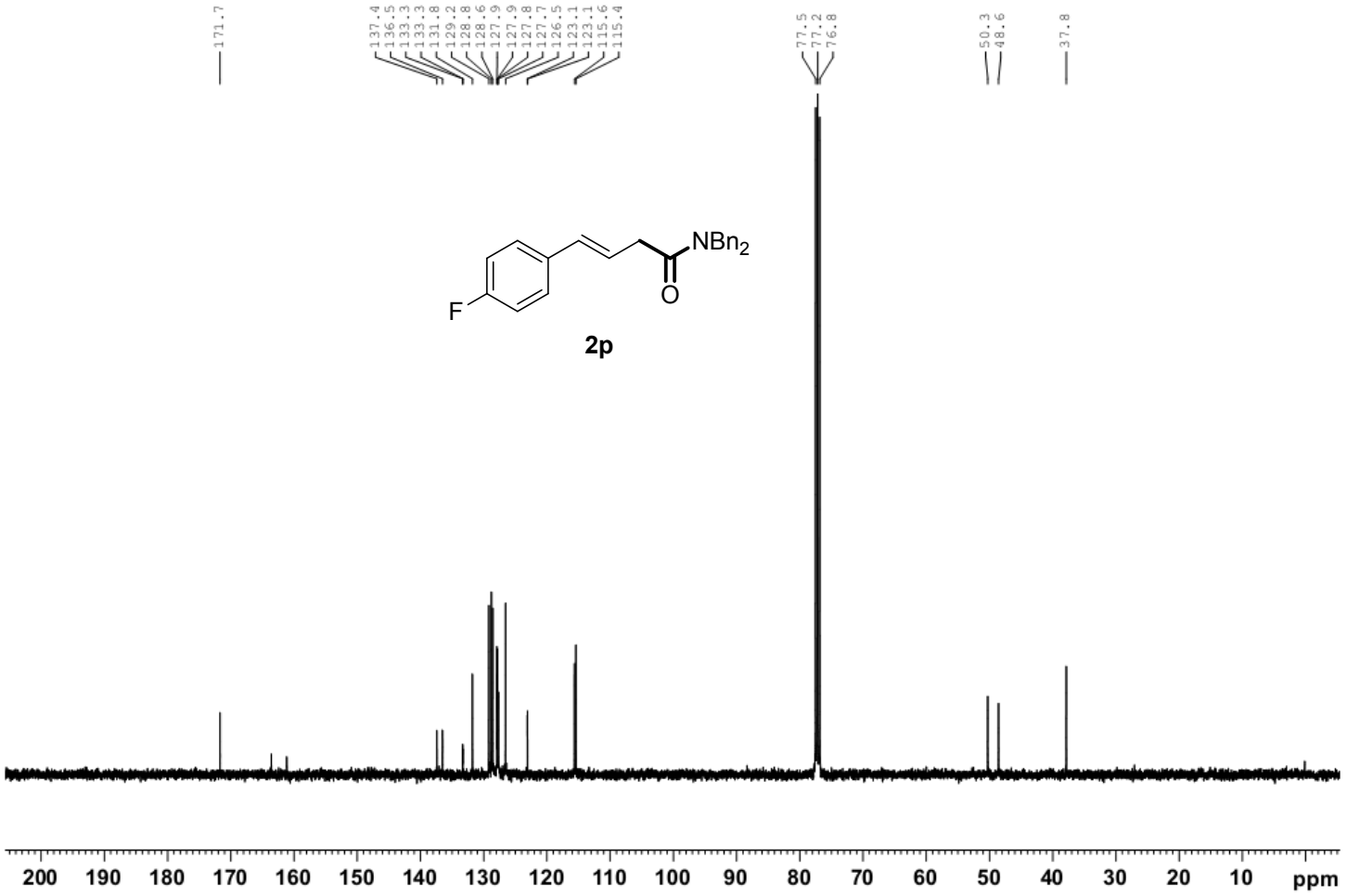
YH-140409-5-CNMR



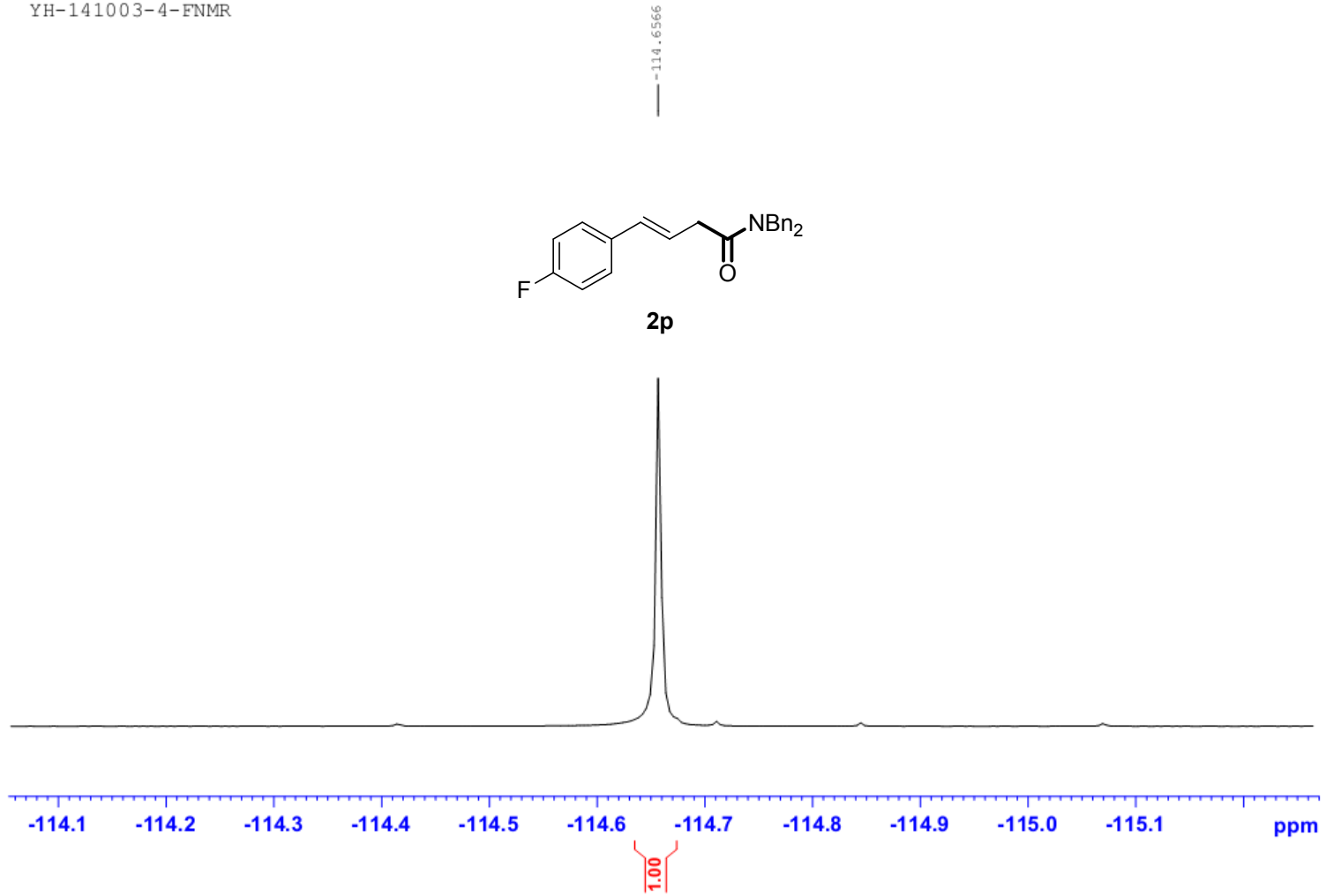
YH-140414-5-HNMR



YH-140416-3-CNMR

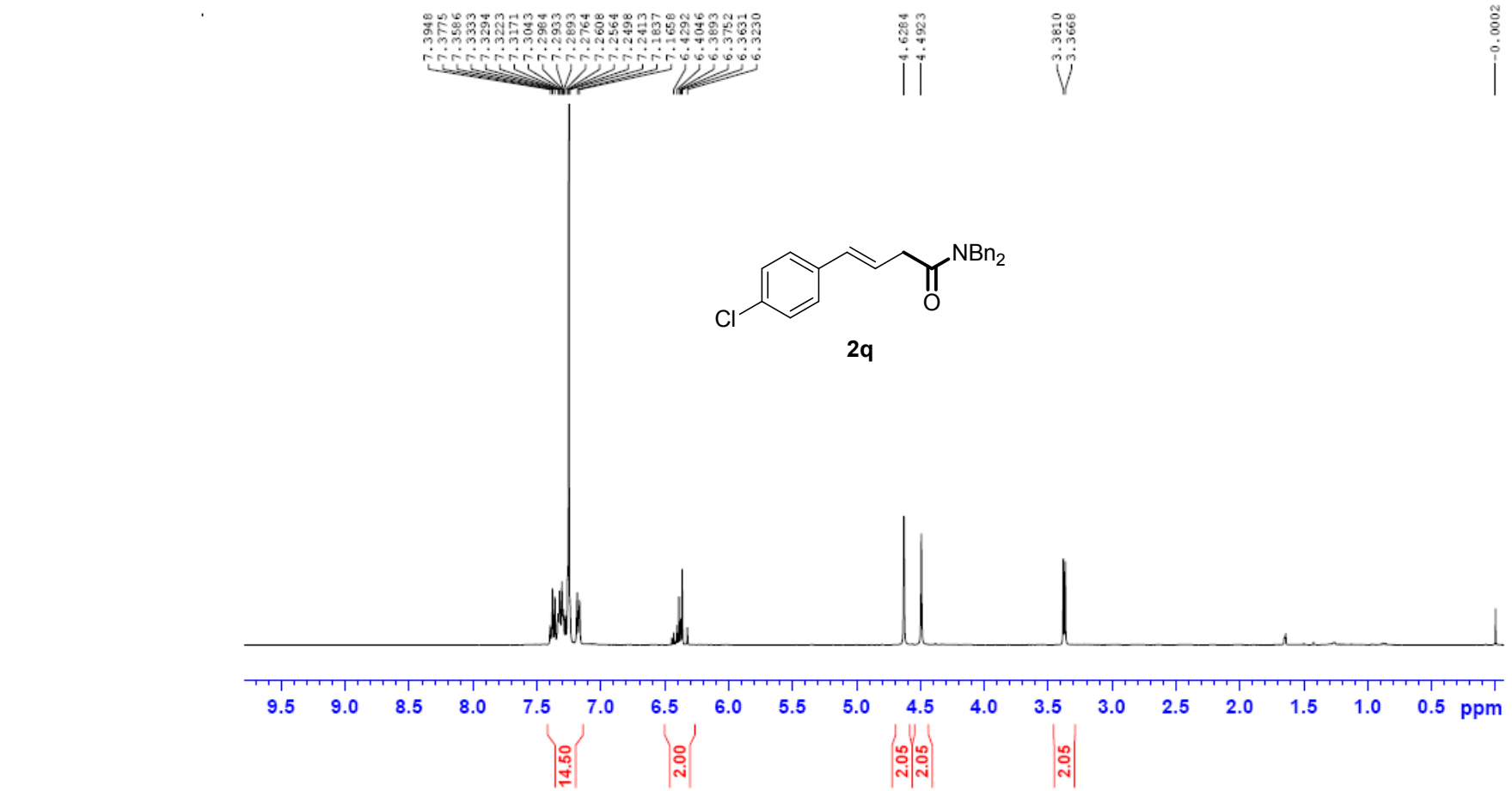


YH-141003-4-FNMR



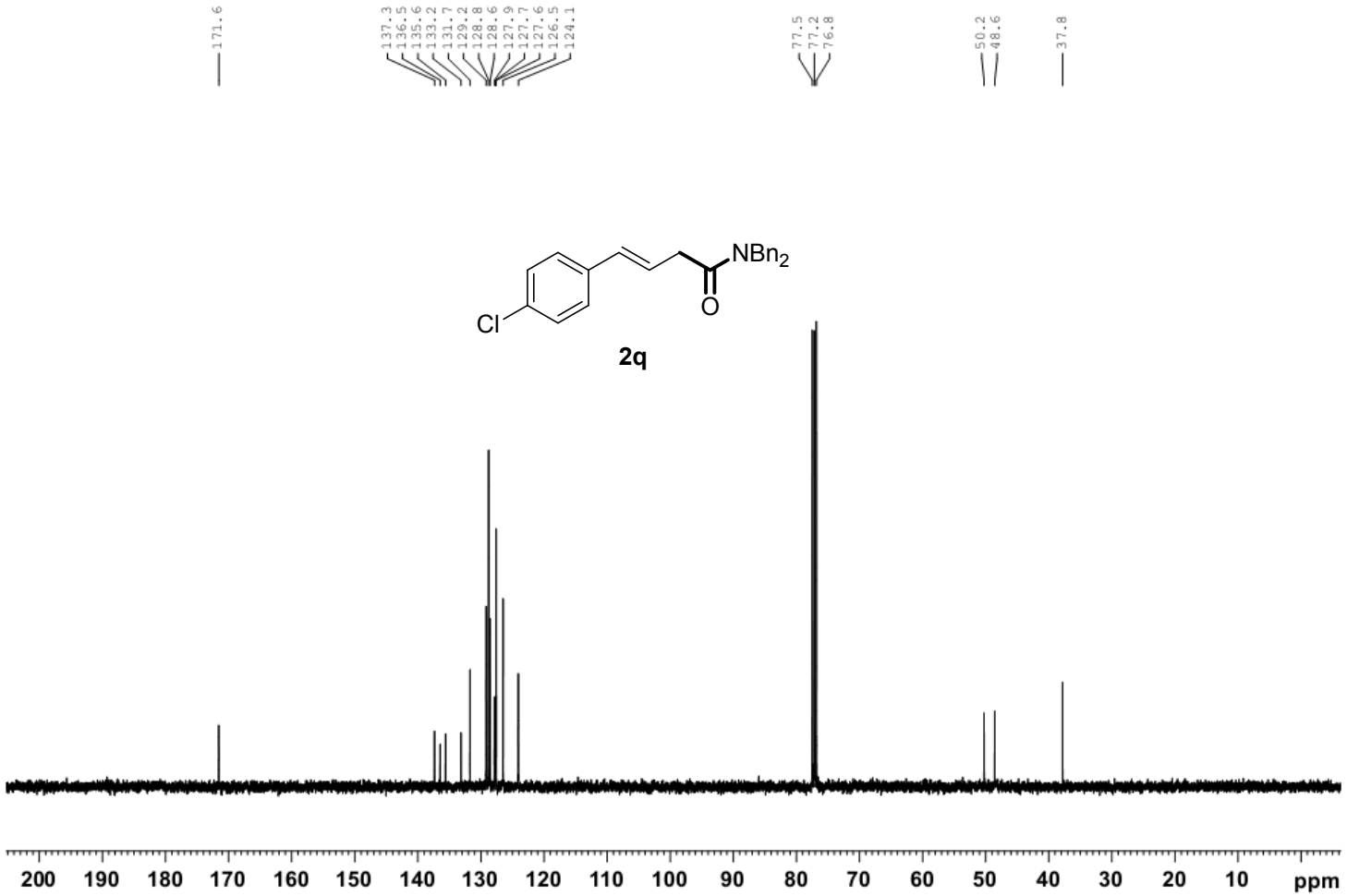
2p

YH-140310-2-HNMR

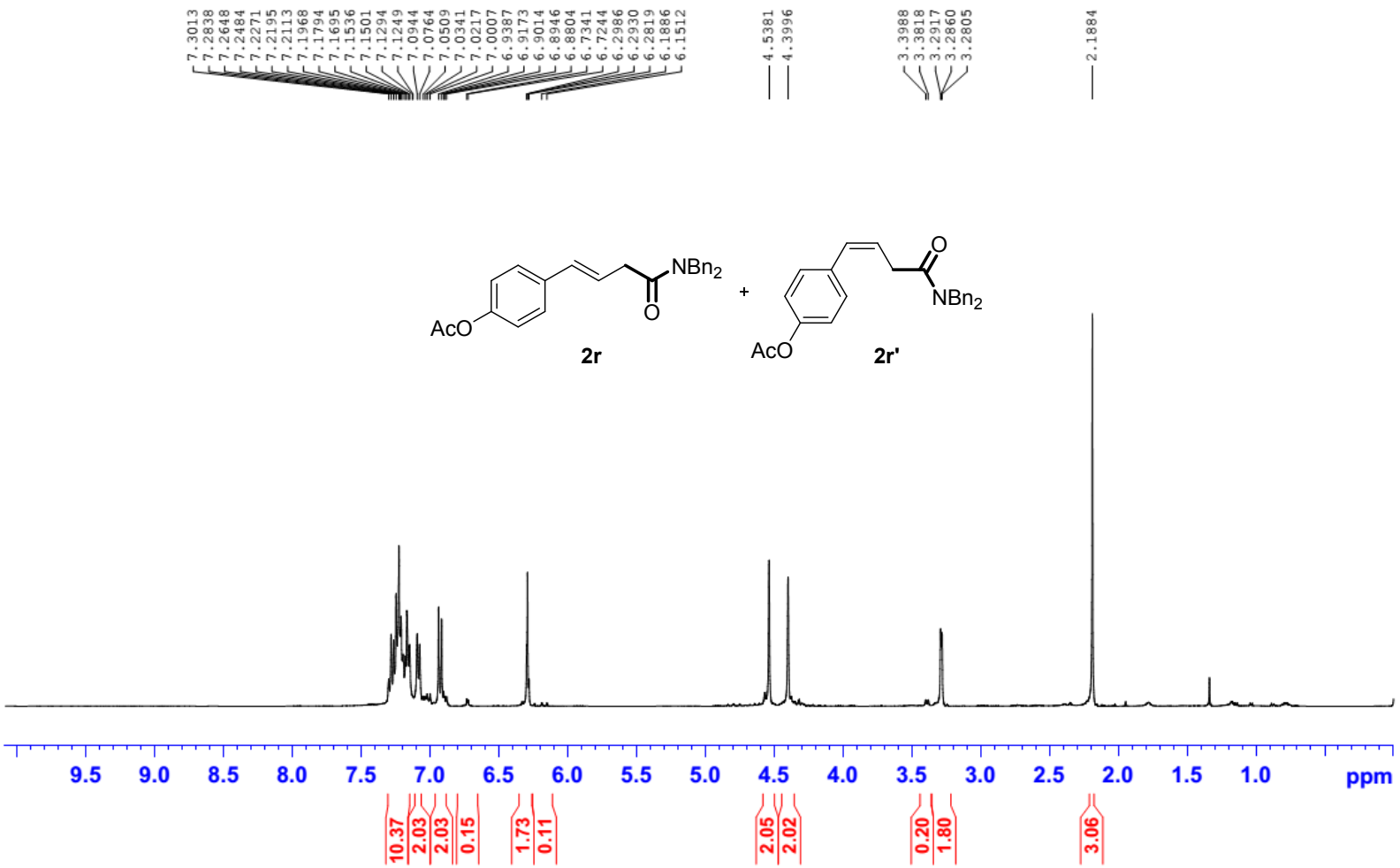


2q

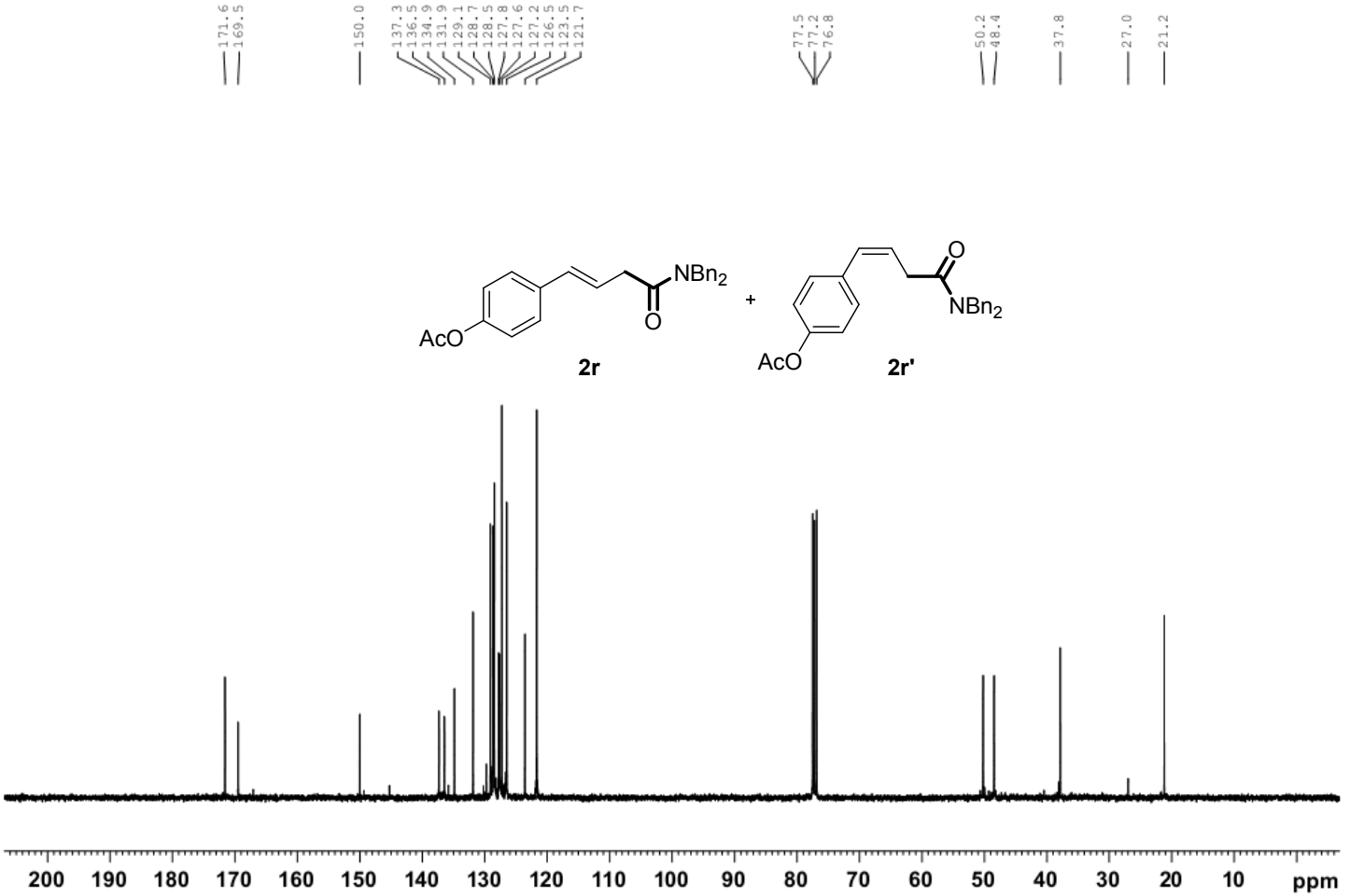
YH-140310-2-CNMR



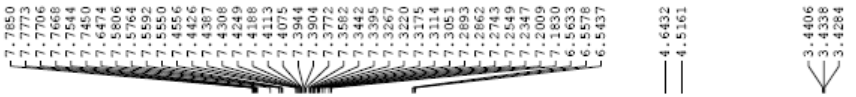
YH-140409-7-HNMR

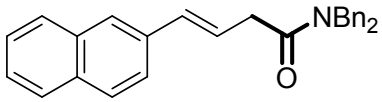


YH-140409-7-CNMR

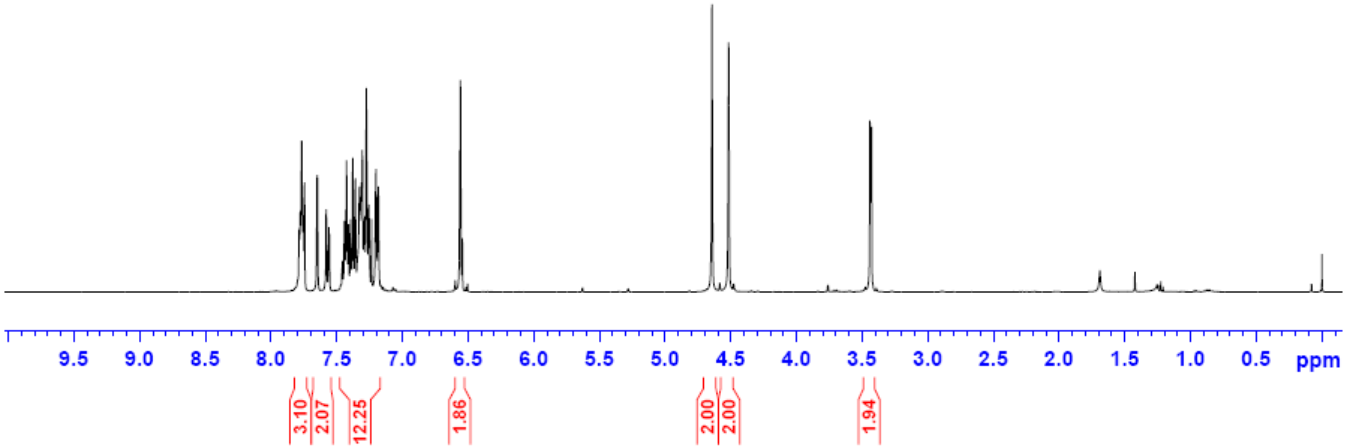


YH-140416-4-HNMR

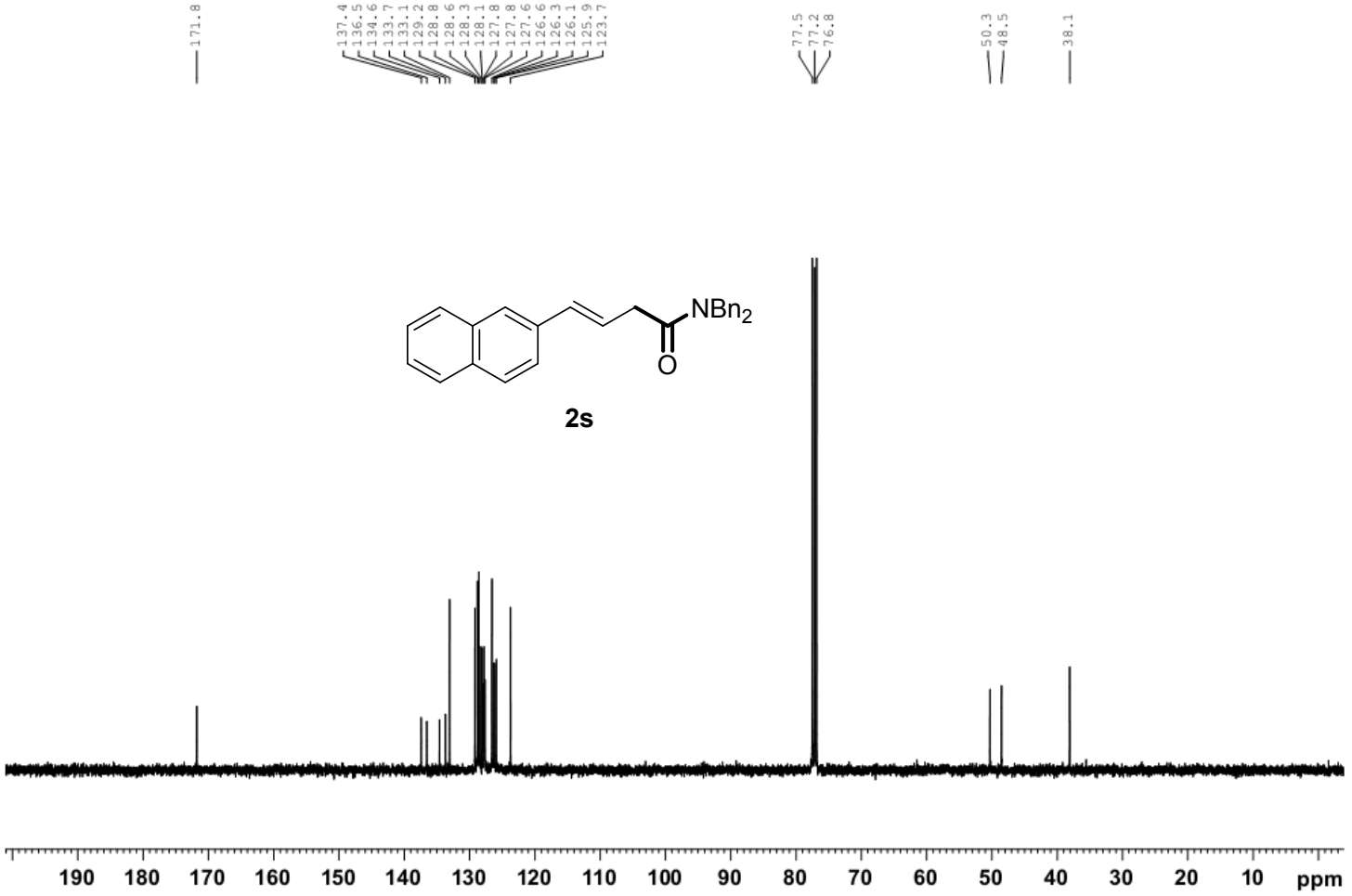




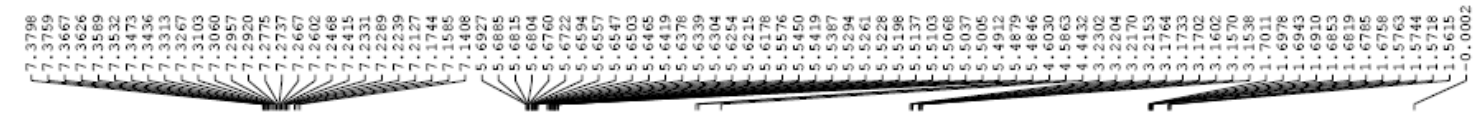
2s

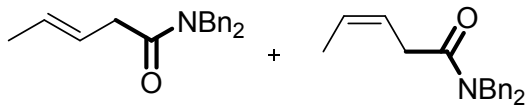


YH-140409-1-CNMR

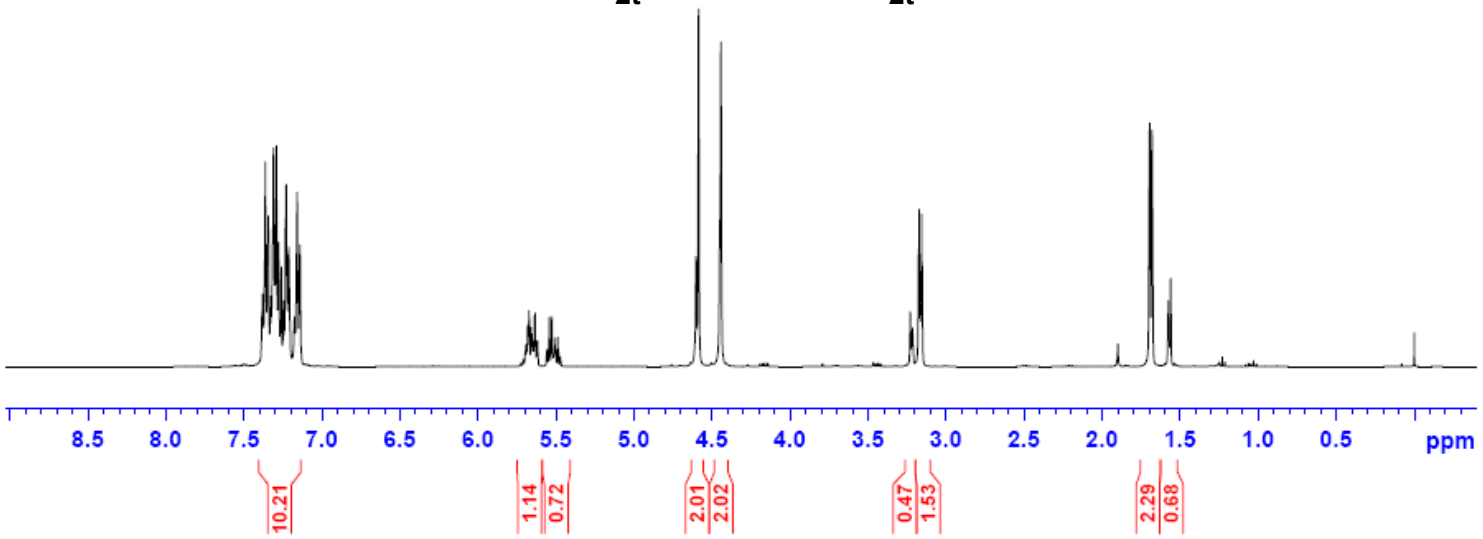


YH-140317-2-HNMR

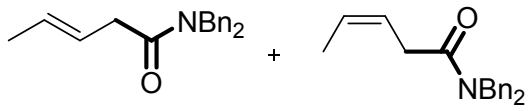




2t **2t'**



YH-140317-2-CNMR



2t **2t'**