Supporting Information

One-pot Transfer Hydrogenation and Reductive Amination of Polyenals

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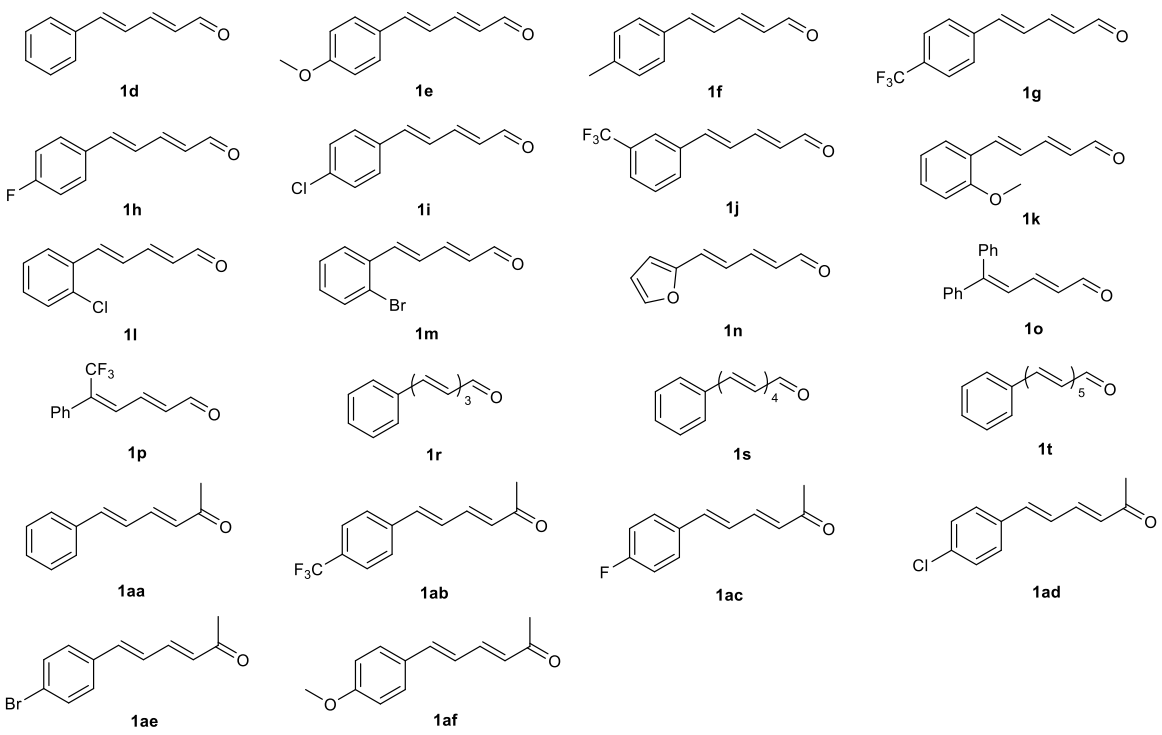
1. General Information

All chemicals were obtained from commercial sources and were used as received unless otherwise noted. All the reactions were carried out under nitrogen atmosphere using standard Schlenk technique. The ^{1}H NMR spectra were recorded on a 400 MHz or 600 MHz NMR spectrometer. The ^{13}C NMR spectra were recorded at 100 MHz or 150 MHz. The ^{19}F NMR spectra were recorded at 376 or 565 MHz. Chemical shifts were expressed in parts per million (δ) downfield from the internal standard tetramethylsilane, and were reported as s (singlet), d (doublet), t (triplet), dd (doublet of doublet), dt (doublet of triplet), m (multiplet), brs (broad singlet), etc. The residual solvent signals were used as references and the chemical shifts were converted to the TMS scale ($CDCl_3$: δ H = 7.26 ppm, δ C = 77.16 ppm). The coupling constants *J* were given in Hz. High resolution mass spectra were obtained on an Agilent Q-TOF 6540 spectrometer and model Orbitrap Explories MX. Column chromatography was performed on silica gel (200-300 mesh). Reactions were heated by metal sand bath (WATTCAS, LAB-500).

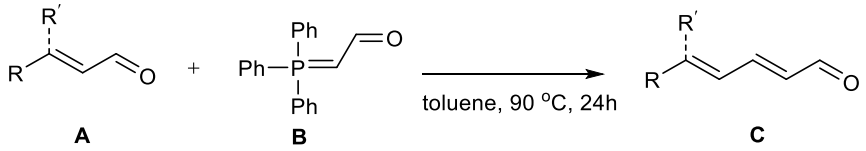
Unless otherwise noted, all other compounds have been reported in the literature or are commercially available.

2. General Procedure: Synthesis of Substrates

The substrates (**1f-1q**)[1], (**1d, 1e**, **1s-1u**)[2] and (**1aa-1af**)[3] were prepared according to the literature.

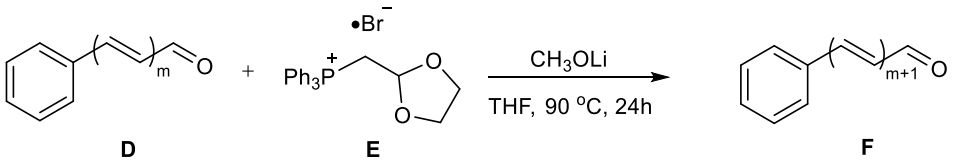


General Procedure A[1]



Unless otherwise mentioned in the following specific substrates, the general procedure is: to a 100 mL dried Schlenk flask with a stir bar was added *α,β* unsaturated aldehydes **A** (1.0 equivalent, 3 mmol), (triphenylphosphoranylidene) acetaldehyde **B** (1.2 equivalents, 3.6 mmol) and dry toluene (20 mL) under N_2 atmosphere. The reaction mixture was stirred at 90°C for 24 hours. Upon completion, cool down the reaction mixture down to room temperature. After which the reaction mixture was filtered through a plug of silica eluting, and then the filtrate was concentrated under reduced pressure and the resulting crude material was purified by flash column chromatography to afford the product **C**.

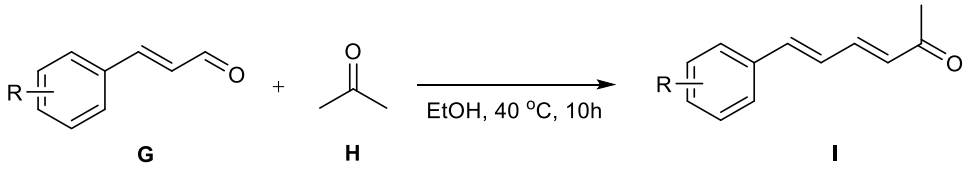
General Procedure B[2]



Unless otherwise mentioned in the following specific substrates, the general

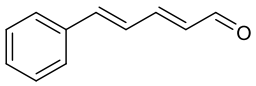
procedure is: to a 250 mL dried round-bottom flask with a stir bar was added (1,3-dioxolan-2-yl)methyl-triphenylphosphonium bromide **E** (2.5 equivalents, 7.5 mmol, 3.23 g), lithium methoxide (2.6 equivalents, 7.8 mmol, 296.4 mg) and anhydrous tetrahydrofuran (THF) (60 mL) at room temperature. The suspension was heated to reflux stirred for 30 minutes, changing color from off-white to light orange/pink. The compound **D** (1.0 equivalent, 3.0 mmol) in 20 mL of dry THF solution was added dropwise over 30 - 60 min. The reaction mixture was refluxed for 24 h. The reaction suspension was then cooled to room temperature, at which point 10% aqueous hydrochloric acid (HCl) was added. Stirring was continued for 1 hour in order to hydrolyze the intermediate acetals (mixture of E- and Z-stereoisomers) to the all-trans configuration. The organic layer was extracted with CH_2Cl_2 (30 mL x 3) and the combined fractions were washed with water, sat. aqueous sodium bicarbonate solution and brine, and then dried over Na_2SO_4. Solvent was removed by rotary evaporation and the product was purified by silica gel column chromatography to afford the product **F**.

General Procedure C[3]



G **H** **I**

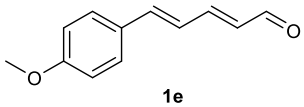
Unless otherwise mentioned in the following specific substrates, the general procedure is: to a 50 mL dried Schlenk flask with a stir bar was gradually added olefine aldehyde **G** (2.0 mmol), acetone **H** (2.0 mL), EtOH (10.0 mL) and 1 mL NaOH aqueous (6 mol/L) at room temperature. The mixture was then allowed to warm to 40 °C and stirred for 10 h, and monitored by TLC until full conversion to the product was observed. The mixture was concentrated to remove the redundant acetone and extracted with ethyl acetate. The organic extracts were combined, concentrated and the residue was purified by silica gel column chromatography to afford the product **I**.



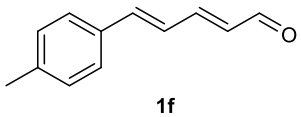
1d

(2E,4E)-5-phenylpenta-2,4-dienal (**1d**). (According to the general procedure **B**). Eluent: PE/EA = 20:1, yellow oil (435 mg, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.63 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.56 – 7.48 (m,

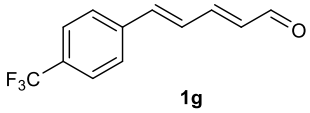
2H), 7.43 – 7.33 (m, 3H), 7.32 – 7.24 (m, 1H), 7.08 – 6.97 (m, 2H), 6.34 – 6.23 (m, 1H). The analytical data are consistent with those reported in the literature.[2]


1e

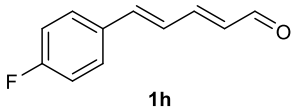
(2E,4E)-5-(4-methoxyphenyl)penta-2,4-dienal (**1e**). (According to the general procedure **B**). Eluent: PE/EA = 20:1, light yellow solid (504 mg, 89%, m.p. 73 – 74 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.59 (d, *J* = 8.0 Hz, 1H), 7.48 – 7.42 (m, 2H), 7.29 – 7.20 (m, 1H), 6.97 (d, *J* = 15.5 Hz, 1H), 6.93 – 6.83 (m, 3H), 6.28 – 6.17 (m, 1H), 3.84 (s, 3H). The analytical data are consistent with those reported in the literature.[4]


1f

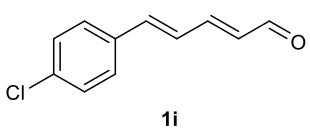
(2E,4E)-5-(p-tolyl)penta-2,4-dienal (**1f**). (According to the general procedure **A**). Eluent: PE/EA = 10:1, yellow solid (109 mg, 21%, m.p. 97 – 99 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.64 (d, *J* = 8.0 Hz, 1H), 7.43 (d, *J* = 8.2 Hz, 2H), 7.34 – 7.28 (m, 1H), 7.22 (d, *J* = 7.9 Hz, 2H), 7.07 – 6.97 (m, 2H), 6.28 (dd, *J* = 15.2, 8.0 Hz, 1H), 2.40 (s, 3H). The analytical data are consistent with those reported in the literature.[4]


1g

(2E,4E)-5-(4-(trifluoromethyl)phenyl)penta-2,4-dienal (**1g**). (According to the general procedure **A**). Eluent: PE/EA = 15:1, yellow semi-solid (260 mg, 38%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.64 (d, *J* = 7.9 Hz, 1H), 7.66 – 7.56 (m, 4H), 7.32 – 7.23 (m, 1H), 7.12 – 6.98 (m, 2H), 6.32 (dd, *J* = 15.2, 7.9 Hz, 1H). The analytical data are consistent with those reported in the literature.[4]

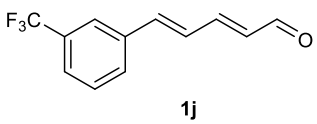

1h

(2E,4E)-5-(4-fluorophenyl)penta-2,4-dienal (**1h**). (According to the general procedure **A**). Eluent: PE/EA = 15:1, yellow solid (212 mg, 40%, m.p. 81 – 82 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.63 (d, *J* = 8.0 Hz, 1H), 7.55 – 7.45 (m, 2H), 7.32 – 7.21 (m, 1H), 7.09 (t, *J* = 8.5 Hz, 2H), 7.03 – 6.88 (m, 2H), 6.28 (dd, *J* = 15.2, 8.0 Hz, 1H). The analytical data are consistent with those reported in the literature.[4]



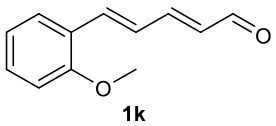
1i

(2E,4E)-5-(4-chlorophenyl)penta-2,4-dienal (**1i**). (According to the general procedure **A**). Eluent: PE/EA = 10:1, yellow solid (213 mg, 37%, m.p. 136 – 138 ^{o}C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.60 (d, *J* = 7.9 Hz, 1H), 7.44 – 7.37 (m, 2H), 7.36 – 7.30 (m, 2H), 7.25 – 7.19 (m, 1H), 6.97 – 6.92 (m, 2H), 6.26 (dd, *J* = 15.2, 7.9 Hz, 1H). The analytical data are consistent with those reported in the literature.[4]



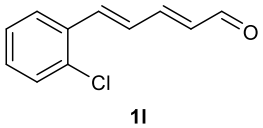
1j

(2E,4E)-5-(3-(trifluoromethyl)phenyl)penta-2,4-dienal(**1j**). (According to the general procedure **A**). Eluent: PE/EA = 15:1, yellow oil (224 mg, 33%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.65 (d, *J* = 8.0 Hz, 1H), 7.74 (s, 1H), 7.68 (d, *J* = 7.7 Hz, 1H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.52 (t, *J* = 7.8 Hz, 1H), 7.28 (ddd, *J* = 15.2, 8.8, 1.4 Hz, 1H), 7.12 – 6.99 (m, 2H), 6.33 (dd, *J* = 15.2, 7.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.6, 151.1, 140.4, 136.4, 132.8, 131.6 (q, *J* = 32.2 Hz, 1C), 130.6, 129.6, 128.0, 126.1 (q, *J* = 3.7 Hz, 1C), 124.2 (q, *J* = 3.8 Hz, 1C), 123.9 (q, *J* = 270.8 Hz, 1C). ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.87. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{12}H_{10}F_3O^+$ 227.0678, Found: 227.0678.



1k

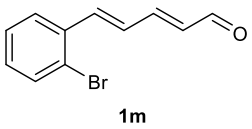
(2E,4E)-5-(2-methoxyphenyl)penta-2,4-dienal (**1k**). (According to the general procedure **B**). Eluent: PE/EA = 15:1, yellow viscous oil (469 mg, 83%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.58 (d, *J* = 8.0 Hz, 1H), 7.52 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.40 – 7.22 (m, 3H), 7.08 – 7.00 (m, 1H), 6.99 – 6.93 (m, 1H), 6.92 – 6.88 (m, 1H), 6.23 (dd, *J* = 15.1, 8.0 Hz, 1H), 3.88 (s, 3H). The analytical data are consistent with those reported in the literature.[5]



1l

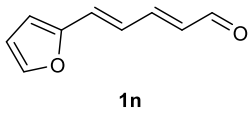
(2E,4E)-5-(2-chlorophenyl)penta-2,4-dienal (**1l**). (According to the general procedure **A**). Eluent: PE/EA = 10:1, white solid (202 mg, 35%, m.p. 86 – 88 ^{o}C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.55 (d, *J* = 7.9 Hz, 1H), 7.59 – 7.54 (m, 1H), 7.38 – 7.28 (m, 2H), 7.24 – 7.15 (m,

3H), 6.94 – 6.85 (m, 1H), 6.20 (dd, *J* = 15.3, 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.65, 151.7, 137.9, 134.5, 133.7, 132.6, 130.6, 130.3, 128.5, 127.25, 127.1. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{11}H_{10}ClO^+$ 193.0415, Found: 193.0414.



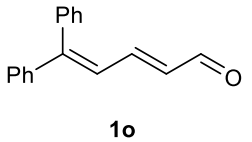
1m

(2E,4E)-5-(2-bromophenyl)penta-2,4-dienal (**1m**). (According to the general procedure **A**). Eluent: PE/EA = 10:1, white solid (140 mg, 20%, m.p. 82 – 83.5 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.65 (d, *J* = 7.9 Hz, 1H), 7.68 – 7.58 (M, 2H), 7.41 (d, *J* = 15.5 Hz, 1H), 7.38 – 7.28 (m, 2H), 7.23 – 7.16 (m, 1H), 7.01 – 6.91 (M, 1H), 6.35 – 6.25 (M, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.65, 151.6, 140.5, 135.4, 133.6, 132.6, 130.8, 128.7, 127.9, 127.3, 125.1. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{11}H_{10}BrO^+$ 236.9910, Found: 236.9908.



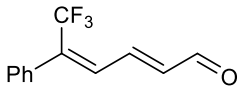
1n

(2E,4E)-5-(furan-2-yl)penta-2,4-dienal (**1n**). (According to the general procedure **A**). Eluent: PE/EA = 20:1, brown solid (202 mg, 45%, m.p. 86 – 89 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.58 (d, *J* = 8.0 Hz, 1H), 7.47 (d, *J* = 1.8 Hz, 1H), 7.18 (dd, *J* = 15.1, 11.0 Hz, 1H), 6.88 (dd, *J* = 15.3, 11.0 Hz, 1H), 6.76 (d, *J* = 15.4 Hz, 1H), 6.53 (d, *J* = 3.4 Hz, 1H), 6.46 (dd, *J* = 3.4, 1.8 Hz, 1H), 6.24 (dd, *J* = 15.1, 8.0 Hz, 1H). The analytical data are consistent with those reported in the literature.[6]



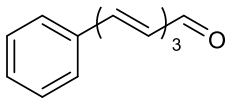
1o

(E)-5,5-diphenylpenta-2,4-dienal (**1o**). (According to the general procedure **A**). Eluent: PE/EA = 20:1, brown viscous oil (170 mg, 24%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.55 (d, *J* = 8.0 Hz, 1H), 7.57 – 7.52 (m, 3H), 7.46 – 7.40 (m, 5H), 7.36 – 7.28 (m, 3H), 7.03 (d, *J* = 11.4 Hz, 1H), 6.45 – 6.36 (m, 1H). The analytical data are consistent with those reported in the literature.[7]



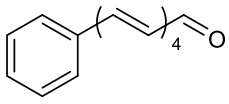
1p

(2E,4Z)-6,6,6-trifluoro-5-phenylhexa-2,4-dienal (**1p**). (According to the general procedure **A**). Eluent: PE/EA = 100:3, yellow oil (435 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.50 (d, *J* = 7.7 Hz, 1H), 7.51 – 7.43 (m, 3H), 7.35 – 7.29 (m, 2H), 7.09 – 6.95 (m, 2H), 6.45 – 6.36 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.0, 144.6, 139.0 (q, *J* = 30.3 Hz, 1C), 137.0, 130.8, 130.3 (q, *J* = 5.9 Hz, 1C), 129.8, 129.75, 128.9, 123.0 (q, *J* = 272.0 Hz, 1C). ^{19}F NMR (376 MHz, $CDCl_3$) δ -65.88. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{12}H_{10}F_3O^+$ 227.0678, Found: 227.0678.



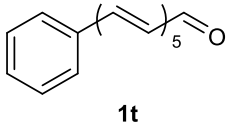
1r

(2E,4E,6E)-7-phenylhepta-2,4,6-trienal (**1r**). (According to the general procedure **B**). Eluent: PE/EA = 15:1, dark yellow solid (471 mg, 85%, m.p. 108 – 111 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.59 (d, *J* = 8.0 Hz, 1H), 7.49 – 7.42 (m, 2H), 7.39 – 7.27 (m, 3H), 7.18 (dd, *J* = 15.2, 11.2 Hz, 1H), 6.95 – 6.77 (m, 3H), 6.61 – 6.51 (m, 1H), 6.19 (dd, *J* = 15.2, 7.9 Hz, 1H). The analytical data are consistent with those reported in the literature.[2]



1s

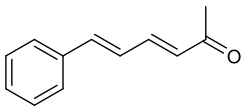
(2E,4E,6E,8E)-9-phenylnona-2,4,6,8-tetraenal (**1s**). (According to the general procedure **B**). Eluent: PE/EA = 10:1, yellow solid (570 mg, 90%, m.p. 125 – 128 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.57 (d, *J* = 7.9 Hz, 1H), 7.47 – 7.40 (m, 2H), 7.37 – 7.31 (m, 2H), 7.29 – 7.24 (m, 1H), 7.15 (dd, *J* = 15.1, 11.3 Hz, 1H), 6.89 (dd, *J* = 15.4, 10.8 Hz, 1H), 6.80 – 6.60 (m, 3H), 6.47 (dt, *J* = 14.7, 11.5 Hz, 2H), 6.17 (dd, *J* = 15.1, 7.9 Hz, 1H). The analytical data are consistent with those reported in the literature.[2]



1t

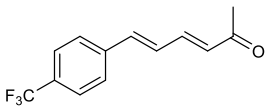
(2E,4E,6E,8E,10E)-11-phenylundeca-2,4,6,8,10-pentaenal (**1t**). (According to the general procedure **B**). Eluent: PE/EA = 10:1, yellow solid (355 mg, 50%, m.p. 157 – 160 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.57 (d, *J* = 8.0 Hz, 1H), 7.45 – 7.39 (m, 2H), 7.36 – 7.29 (m, 2H), 7.27 – 7.22 (m, 1H), 7.15 (dd, *J* = 15.1, 11.3 Hz, 1H), 6.88 (dd, *J* = 15.5, 10.8 Hz, 1H), 6.74 (dd, *J* = 14.7, 11.1 Hz, 1H), 6.65 (d, *J* = 15.5 Hz, 1H), 6.62 – 6.35 (m, 5H), 6.16

(dd, *J* = 15.1, 8.0 Hz, 1H). The analytical data are consistent with those reported in the literature.[2]



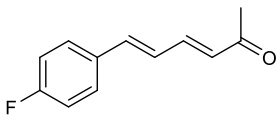
1aa

(3E,5E)-6-phenylhexa-3,5-dien-2-one (**1aa**). (According to the general procedure **C**). Yellow solid (248 mg, 72%, m.p. 62 – 64 ^{o}C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.45 (m, 2H), 7.40 – 7.28 (m, 4H), 6.99 – 6.84 (m, 2H), 6.26 (d, *J* = 15.5 Hz, 1H), 2.32 (s, 3H). The analytical data are consistent with those reported in the literature.[3]



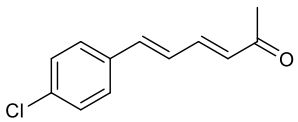
1ab

(3E,5E)-6-(4-(trifluoromethyl)phenyl)hexa-3,5-dien-2-one (**1ab**). (According to the general procedure **C**). Eluent: PE/EA = 20:1, light yellow solid (185 mg, 39%, m.p. 55 – 57 ^{o}C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.54 (m, 4H), 7.34 – 7.25 (m, 1H), 7.01 – 6.91 (m, 2H), 6.32 (d, *J* = 15.5 Hz, 1H), 2.34 (s, 3H). The analytical data are consistent with those reported in the literature.[8]



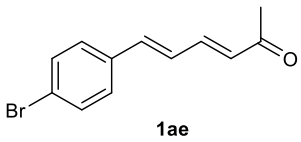
1ac

(3E,5E)-6-(4-fluorophenyl)hexa-3,5-dien-2-one (**1ac**). (According to the general procedure **C**). Eluent: PE/EA = 20:1, yellow oil (214 mg, 56%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.42 (m, 2H), 7.32 – 7.22 (m, 1H), 7.10 – 7.02 (m, 2H), 6.91 (d, *J* = 15.6 Hz, 1H), 6.85 – 6.74 (m, 1H), 6.25 (d, *J* = 15.5 Hz, 1H), 2.32 (s, 3H). The analytical data are consistent with those reported in the literature.[9]



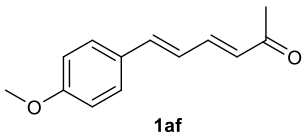
1ad

(3E,5E)-6-(4-chlorophenyl)hexa-3,5-dien-2-one (**1ad**). (According to the general procedure **C**). Eluent: PE/EA = 10:1, yellow solid (120 mg, 29%, m.p. 80 – 81 ^{o}C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.38 (m, 2H), 7.36 – 7.31 (m, 2H), 7.31 – 7.23 (m, 1H), 6.95 – 6.79 (m, 2H), 6.27 (d, *J* = 15.5 Hz, 1H), 2.33 (s, 3H). The analytical data are consistent with those reported in the literature.[8]



1ae

(3E,5E)-6-(4-bromophenyl)hexa-3,5-dien-2-one (**1ae**). (According to the general procedure **C**). Eluent: PE/EA = 10:1, yellow solid (161 mg, 32%, m.p. 104 – 105 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.47 (m, 2H), 7.37 – 7.32 (m, 2H), 7.31 – 7.23 (m, 1H), 6.93 – 6.81 (m, 2H), 6.28 (d, *J* = 15.5 Hz, 1H), 2.33 (s, 3H). The analytical data are consistent with those reported in the literature. The analytical data are consistent with those reported in the literature.[8]



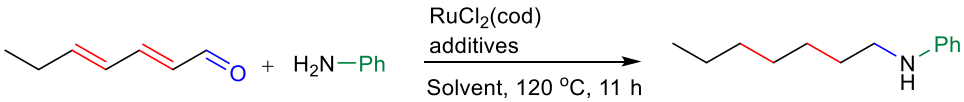
1af

(3E,5E)-6-(4-methoxyphenyl)hexa-3,5-dien-2-one (**1af**). (According to the general procedure **C**). Yellow solid (325 mg, 80%, m.p. 101 – 103 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.40 (m, 2H), 7.33 – 7.24 (m, 1H), 6.92 – 6.87 (m, 3H), 6.81 – 6.73 (m, 1H), 6.22 (d, *J* = 15.5 Hz, 1H), 3.84 (s, 3H), 2.31 (s, 3H). The analytical data are consistent with those reported in the literature.[8]

3. Experimental Section

(1) Optimization of the reaction conditions.

Table S1: Screening of various additives for optimizing the reaction conditions of synthesis of 3aa.[a]



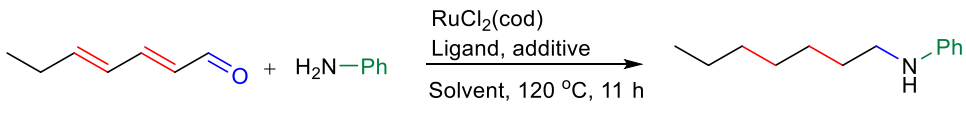
1a 2a 3aa

Entry	Cat. (10 mol%)	Additive	Solvent	Yield[b] of 3aa (%)
1	$RuCl_2$(cod)	Cs_2CO_3 (1.0 equiv)	*i*PrOH	32
2	$RuCl_2$(cod)	Cs_2CO_3 (0.5 equiv)	*i*PrOH	48
3	$RuCl_2$(cod)	Cs_2CO_3 (0.2 equiv)	*i*PrOH	59
4	$RuCl_2$(cod)	Cs_2CO_3 (0.1 equiv)	*i*PrOH	23
5	$RuCl_2$(cod)	Na_2CO_3 (1.0 equiv)	*i*PrOH	57
6	$RuCl_2$(cod)	Na_2CO_3 (0.5 equiv)	*i*PrOH	38
7	$RuCl_2$(cod)	Na_2CO_3 (0.2 equiv)	*i*PrOH	37
8	$RuCl_2$(cod)	K_2CO_3 (1.5 equiv)	*i*PrOH	40

9	$RuCl_2(cod)$	K_2CO_3 (1.0 equiv)	*i*PrOH	21
10	$RuCl_2(cod)$	Ag_2CO_3 (1.0 equiv)	*i*PrOH	11
11	$RuCl_2(cod)$	CH_3ONa (1.0 equiv)	*i*PrOH	39
12	$RuCl_2(cod)$	HCOONa (1.0 equiv)	*i*PrOH	59
13	$RuCl_2(cod)$	KOH (1.0 equiv)	*i*PrOH	52
14	$RuCl_2(cod)$	KOH (0.5 equiv)	*i*PrOH	61

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), $RuCl_2(cod)$ (10 mol%), solvent (1.0 mL), 120 °C,11 h. [b]isolated yields.

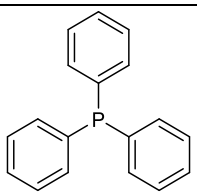
Table S2: Screening of ligands for optimizing the reaction conditions of synthesis of 3aa.[a]

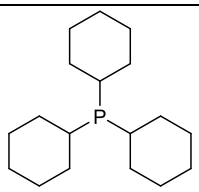


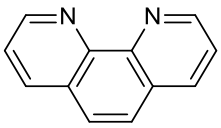
1a **2a** **3aa**

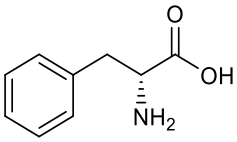
Entry	**Cat. (x mol%)**	**Ligand**	**Additive**	**Solvent**	**Yield[g] of 3aa (%)**
1[b]	$RuCl_2(cod)$	L_1 (20 mol%)	HCOONa (1.0 equiv)	*i*PrOH	trace
2[b]	$RuCl_2(cod)$	L_2 (20 mol%)	Cs_2CO_3 (0.2 equiv)	*i*PrOH	71
3[b]	$RuCl_2(cod)$	L_2 (20 mol%)	HCOONa (1.0 equiv)	*i*PrOH	95
4[b]	$RuCl_2(cod)$	L_2 (20 mol%)	Na_2CO_3 (1.0 equiv)	*i*PrOH	65
5[b]	$RuCl_2(cod)$	L_2 (20 mol%)	KOH (0.5 equiv)	*i*PrOH	38
6[c]	$RuCl_2(cod)$	L_2 (20 mol%)	HCOONa (1.0 equiv)	*i*PrOH	92
7[c]	$RuCl_2(cod)$	L_2 (10 mol%)	HCOONa (1.0 equiv)	*i*PrOH	80
8[c]	$RuCl_2(cod)$	L_2 (20 mol%)	HCOONa (0.5 equiv)	*i*PrOH	55
9[c]	$RuCl_2(cod)$	L_2 (20 mol%)	HCOONa (1.5 equiv)	*i*PrOH	67
10[ce]	$RuCl_2(cod)$	L_2 (20 mol%)	HCOONa (1.0 equiv)	*i*PrOH	85
11[cf]	$RuCl_2(cod)$	L_2 (20 mol%)	HCOONa (1.0 equiv)	*i*PrOH	50
12[c]	$RuCl_2(cod)$	L_2 (20 mol%)	HCOONa (1.0 equiv)	H_2O	n.d.
13[d]	$RuCl_2(cod)$	L_2 (20 mol%)	HCOONa (1.0 equiv)	*i*PrOH	61
14	---	L_2 (20 mol%)	HCOONa (1.0 equiv)	*i*PrOH	n.d.
15[b]	$RuCl_2(cod)$	L_2 (20 mol%)	---	*i*PrOH	trace
16[b]	$RuCl_2(cod)$	L_3 (20 mol%)	Cs_2CO_3 (0.2 equiv)	*i*PrOH	n.r.
17[b]	$RuCl_2(cod)$	L_4 (20 mol%)	Cs_2CO_3 (0.2 equiv)	*i*PrOH	n.r.
18[b]	$RuCl_2(cod)$	L_5 (20 mol%)	Cs_2CO_3 (0.2 equiv)	*i*PrOH	n.r.
19[b]	$RuCl_2(cod)$	L_6 (20 mol%)	Cs_2CO_3 (0.2 equiv)	*i*PrOH	trace
20[b]	$RuCl_2(cod)$	---	Na_2CO_3 (1.0 equiv)	C_2H_5OH	46
21[b]	$RuCl_2(cod)$	---	Na_2CO_3 (1.0 equiv)	DCM/ *i*PrOH (1:1)	trace

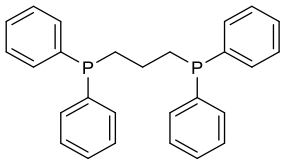
22[b]	$RuCl_2$(cod)	---	Na_2CO_3 (1.0 equiv)	CH_3CN/ *i*PrOH (1:1)	trace

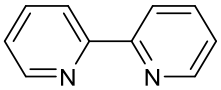

L1


L2


L3

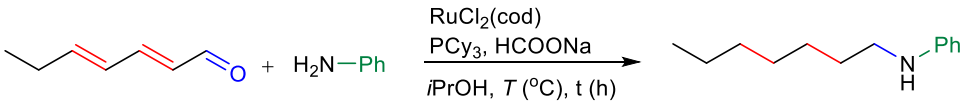

L4


L5


L6

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), solvent (1.0 mL), 120 °C,11 h. [b]$RuCl_2$(cod) (10 mol%). [c]$RuCl_2$(cod) (5 mol%). [d]$RuCl_2$(cod) (2 mol%). [e]**1a** (0.1 mmol), **2a** (0.12 mmol). [f]**1a** (0.15 mmol), **2a** (0.1 mmol). [g]isolated yields. n.d. = not detected, n.r. = no reaction.

Table S3: Screening of other conditions for optimizing the reaction conditions of synthesis of 3aa.[a]

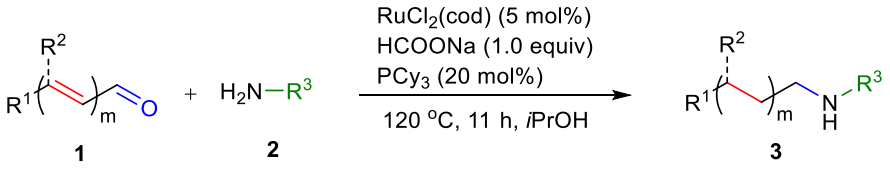


1a **2a** **3aa**

Entry	**Ligand (x mol%)**	***T* (°C)**	***t* (h)**	**Yield[b] of 3aa (%)**
1	PCy_3 (20 mol%)	90	11	39
2	PCy_3 (20 mol%)	100	11	66
3	PCy_3 (20 mol%)	110	11	81
4	PCy_3 (20 mol%)	120	9	80
5	PCy_3 (20 mol%)	120	11	95
6	PCy_3 (20 mol%)	120	13	91
7	PCy_3 (20 mol%)	120	15	85
8	PCy_3 (10 mol%)	120	11	65
9	PCy_3 (30 mol%)	120	11	93
10	PCy_3 (50 mol%)	120	11	73

[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), $RuCl_2$(cod) (10 mol%), HCOONa (0.1 mmol), solvent (1.0 mL). [b]isolated yields.

(2) General procedures for the synthesis of products 3.

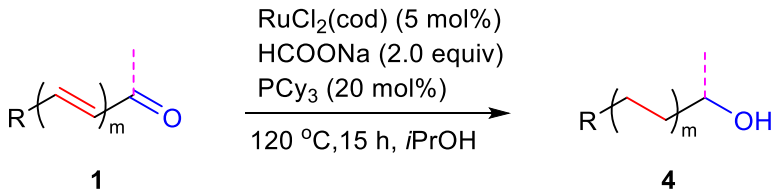


A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), HCOONa (1.0 equivalent, 0.1 mmol, 6.8 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **1** (1.0 equivalent, 0.1 mmol), **2** (1.5 equivalents, 0.15 mmol) and dry *i*PrOH (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 11 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3 (3aa** – **3va)**.

(3) Synthesis of product 3aaa.

A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), HCOOH (4.0 equivalent, 0.4 mmol, 18.4 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **1aa** (1.0 equivalent, 0.1 mmol), **2a** (1.5 equivalents, 0.15 mmol) and dry *i*PrOH (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 11 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3aaa.**

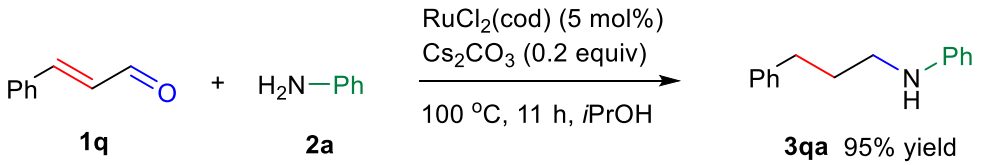
(4) General procedures for the synthesis of products 4.



A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), HCOONa (2.0 equivalents, 0.2 mmol, 13.6 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **1** (1.0 equivalent, 0.1 mmol) and dry *i*PrOH (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 15 h. After the solvent was removed under reduced

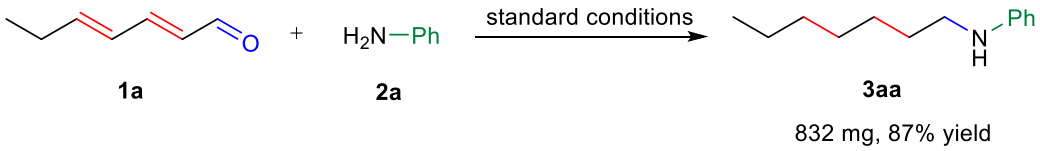
pressure, the residue was purified by flash-column chromatography to afford **4 (4a** – **4h)**.

(5) Synthesis of product 3qa.



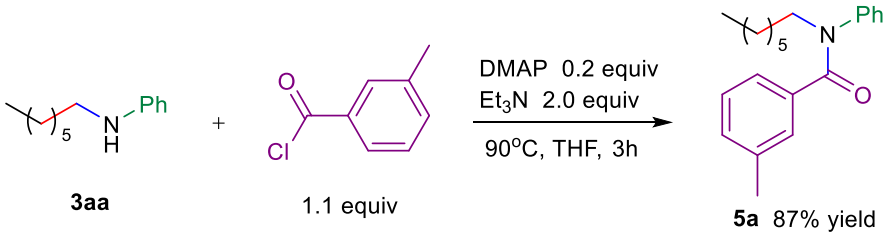
A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), and Cs_2CO_3 (20 mol%, 0.02 mmol, 6.5 mg) were charged into a reaction tube, and then to which were added **1q** (1.0 equivalent, 0.1 mmol, 13.2 mg), **2a** (1.5 equivalents, 0.15 mmol, 14.0 mg) and dry *i*PrOH (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 100 ºC heated by metal sand bath for 11 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3qa** (20.0 mg, 95% yield)**.**

(6) Scale-up experiment.



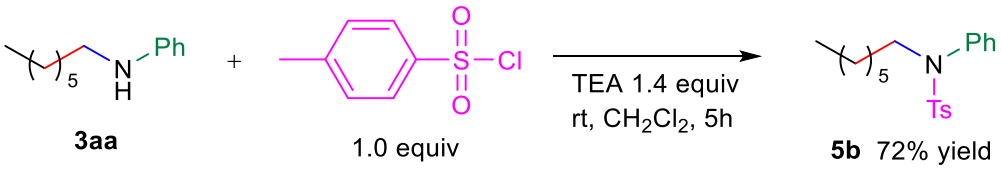
A mixture of $RuCl_2$(cod) (5 mol%, 0.25 mmol, 70 mg), HCOONa (1.0 equivalent, 5.0 mmol, 340 mg), PCy_3 (20 mol%, 1.0 mmol, 280.4 mg) were charged into an oven-dried flask with a stir bar, and then to which were added **1a** (1.0 equivalent, 5.0 mmol, 550.8 mg), **2a** (1.5 equivalents, 7.5 mmol, 698.5 mg) and dry *i*PrOH (50 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 11 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography using PE/ EA =50:1 to afford **3aa** (832 mg, 87% yield). [$RuCl_2$(cod) (2 mol%), (337 mg, 35% yield)].

(7) Synthesis of product 5a.[10]



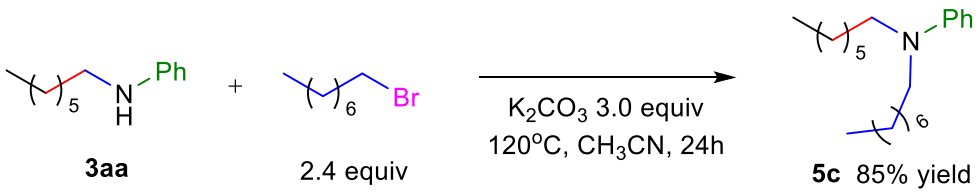
To an ice-cooled solution of **3aa** (1.0 equivalent, 0.1 mmol, 19.1 mg), DMAP (20 mol%, 0.02 mmol, 2.4 mg) and Et_3N (2.0 equivalents, 0.2 mmol, 20.2 mg) in THF (1.0 mL) was added 3-methylbenzoyl chloride (1.1 equivalents, 0.11 mmol, 17.0 mg), and the mixture was stirred at 90 ºC heated by metal sand bath for 3 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **5a**.

(8) Synthesis of product 5b.[11]



To a 25 mL dried Schlenk flask with a stir bar was added **3aa** (1.0 equivalent, 0.1 mmol, 19.1 mg), 4-methylbenzenesulfonyl chloride (1.0 equivalent, 0.1 mmol, 19.1 mg) and dry CH_2Cl_2 (1.0 mL) at 0°C under N_2 atmosphere, followed by a slight excess of TEA (1.4 equivalents, 0.14 mmol, 14.2 mg) and the reaction was stirred 5h at room temperature. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **5b.**

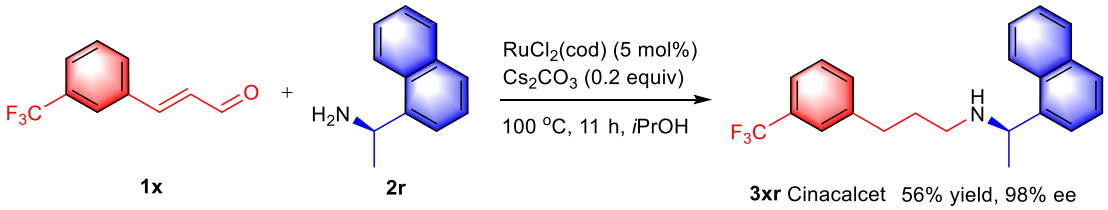
(9) Synthesis of product 5c.[12]



A mixture of **3aa** (1.0 equivalent, 0.2 mmol, 38.2 mg), K_2CO_3 (3.0 equivalents, 0.6 mmol, 82.9 mg), n-Octyl Bromide (2.4 equivalents, 0.48 mmol, 92.7 mg), and CH_3CN (2.0 mL) was stirred under N_2 at 120 °C for about 24h. After the solvent was

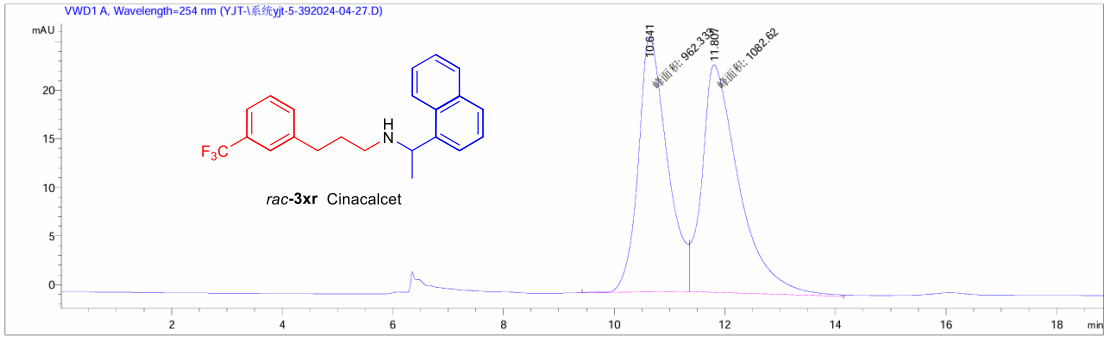
removed under reduced pressure, the crude product was purified by flash-column chromatography to afford **5c**.

(10) Synthesis of product 3xr.



A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), Cs_2CO_3 (20 mol%, 0.02 mmol, 6.5 mg) were charged into a reaction tube, and then to which were added **1x** (1.0 equivalent, 0.1 mmol, 20 mg), **2r** (1.5 equivalents, 0.15 mmol, 25.7 mg) and dry *i*PrOH (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 100 °C heated by metal sand bath for 11 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3xr.**

Using the general procedure as above described. Isolated yield: 56%, ee: 98%. The ee value was determined by HPLC (chiralpak IG, n-hexane/*i*PrOH 98:2, flow rate = 0.5 mL/min).



#	[min]		[min]	[mAU*s]	[mAU]	%
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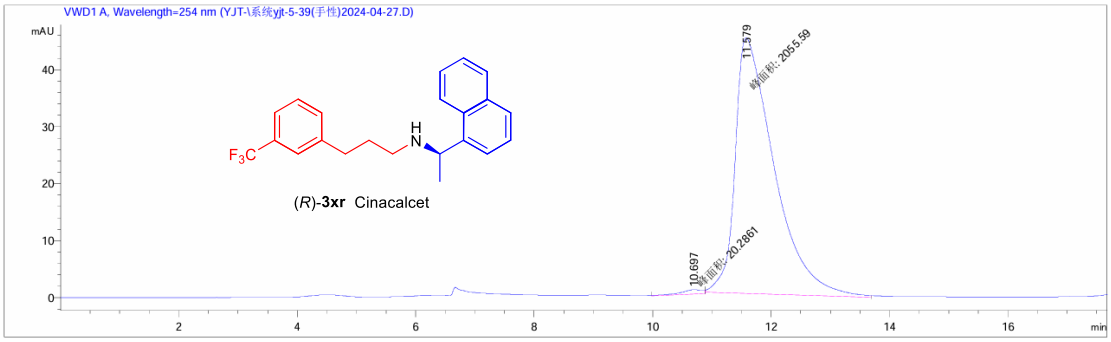
2	11.807	MM	0.7705	1082.62280	23.41745	52.9411



#	[min]		[min]	[mAU*s]	[mAU]	%
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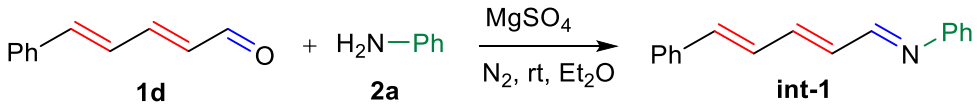
2	11.579	MM	0.7640	2055.59473	44.84539	99.0228

(11) Synthesis of product int-1.[13]

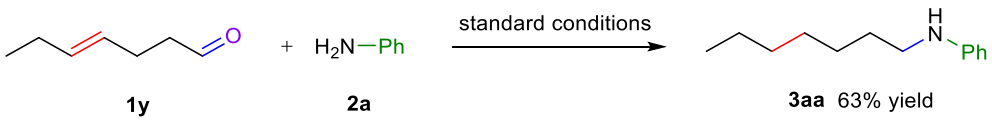


To a 50 mL dried Schlenk flask with a stir bar was added anhydrous $MgSO_4$ (1.0 equivalent, 2.0 mmol, 240.7 mg), **1d** (1.0 equivalent, 2.0 mmol, 316.4 mg), **2a** (1.0 equivalent, 2.0 mmol, 186.3 mg) and anhydrous Et_2O (5.0 mL) under N_2 atmosphere, and the reaction was stirred 10 h at room temperature. Dissolved with CH_2Cl_2 (10.0 mL) and filtered out $MgSO_4$, then removed the solvent under reduced pressure, the residue was purified by neutral alumina column chromatography to afford **int-1** (hexane/EtOAc: 20/1).

4. Mechanistic Studies

A Control experiments

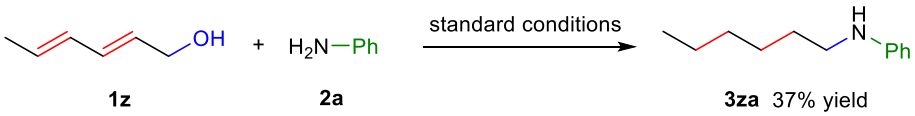
(i)



A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), HCOONa (1.0 equivalent, 0.1 mmol, 6.8 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **1y** (1.0 equivalent, 0.1 mmol, 11.0 mg), **2a** (1.5 equivalents, 0.15 mmol, 14.0 mg) and dry *i*PrOH (1.0 mL) under N_2 atmosphere. The

reaction mixture was stirred at 120 ºC heated by metal sand bath for 11 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3aa.**

(ii)

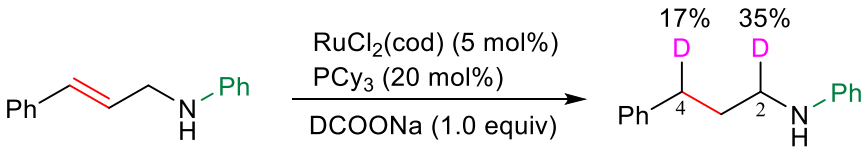


1z **2a** **3za** 37% yield

A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), HCOONa (1.0 equivalent, 0.1 mmol, 6.8 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **1z** (1.0 equivalent, 0.1 mmol, 9.8 mg), **2a** (1.5 equivalents, 0.15 mmol, 14.0 mg) and dry *i*PrOH (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 11 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3za**.

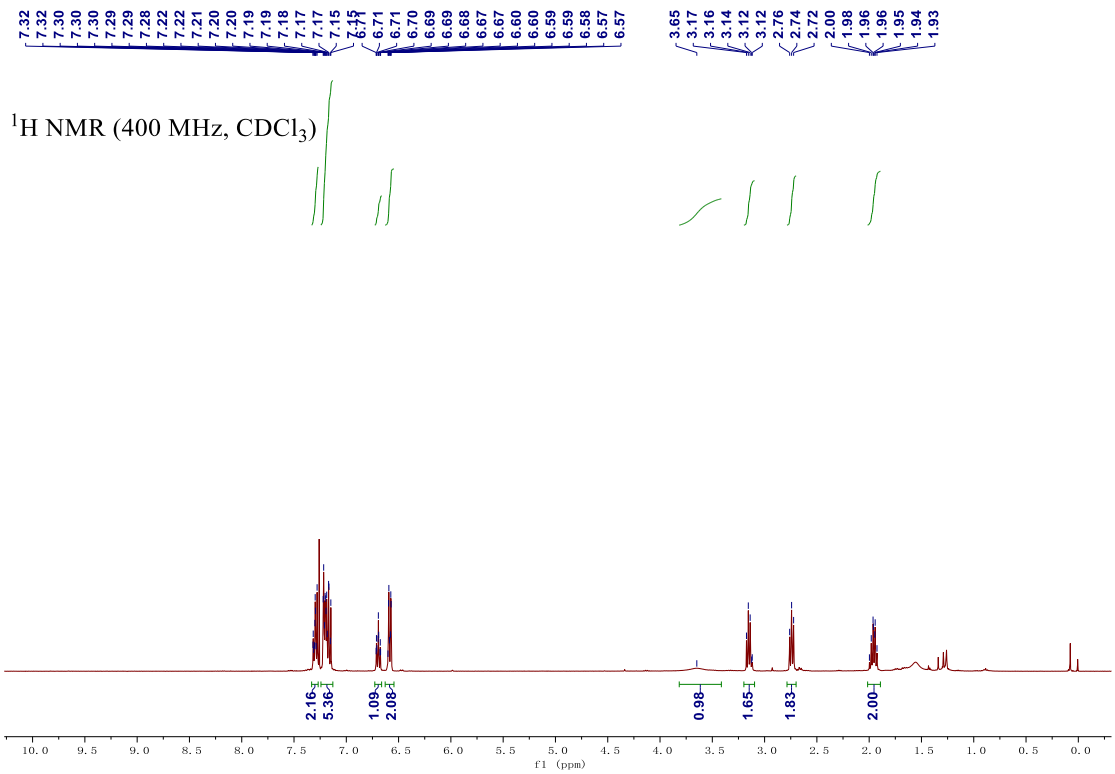
B Deuterium labeling experiments

(iii)



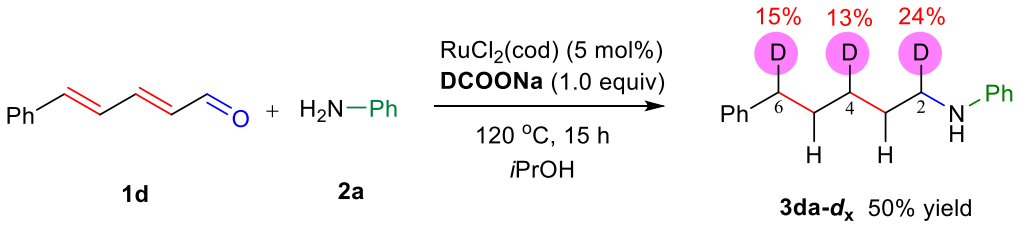
1w **3qa-*d*** 96% yield

A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), DCOONa (1.0 equivalent, 0.1 mmol, 7.0 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **1w** (1.0 equivalent, 0.1 mmol, 20.9 mg), and dry CH_3CH_2OH (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 15 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3qa-*d*** (20.1 mg, 96% yield). [^{1}H NMR analysis indicated 35%, 17% deuteration at the C(2), C(4) position, respectively.]



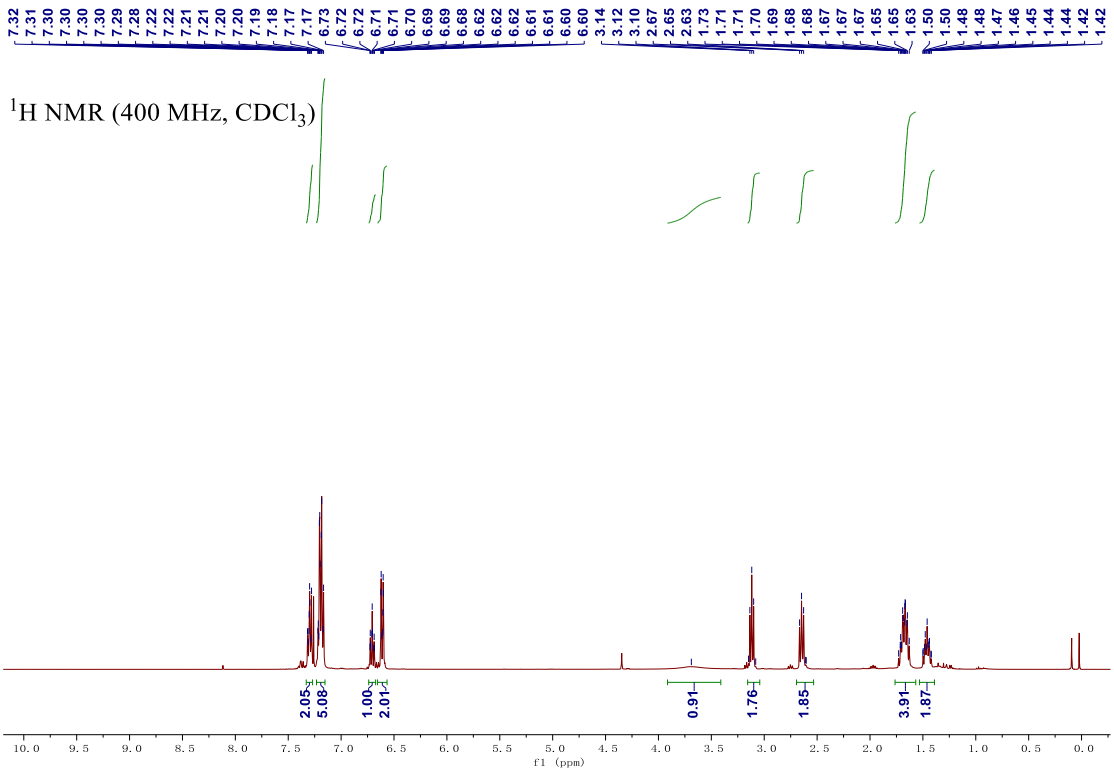
^{1}H NMR spectrum of the product deuterated **3qa-*d***

(iv-1)



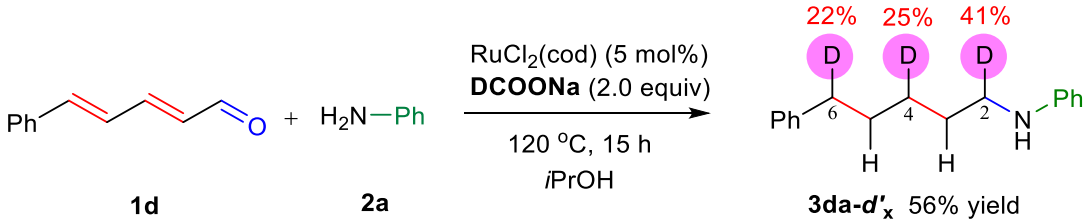
1d **2a** **3da-*d*$_x$** 50% yield

A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg) and DCOONa (1.0 equivalent, 0.1 mmol, 7.0 mg) were charged into a reaction tube, and then to which were added **1d** (1.0 equivalent, 0.1 mmol, 15.8 mg), **2a** (1.5 equivalents, 0.15 mmol, 14.0 mg) and dry *i*PrOH (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 15 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3da-*d*$_x$**. [^{1}H NMR analysis indicated 24%, 13%, 15% deuteration at the C(2), C(4), C(6) position, respectively.]



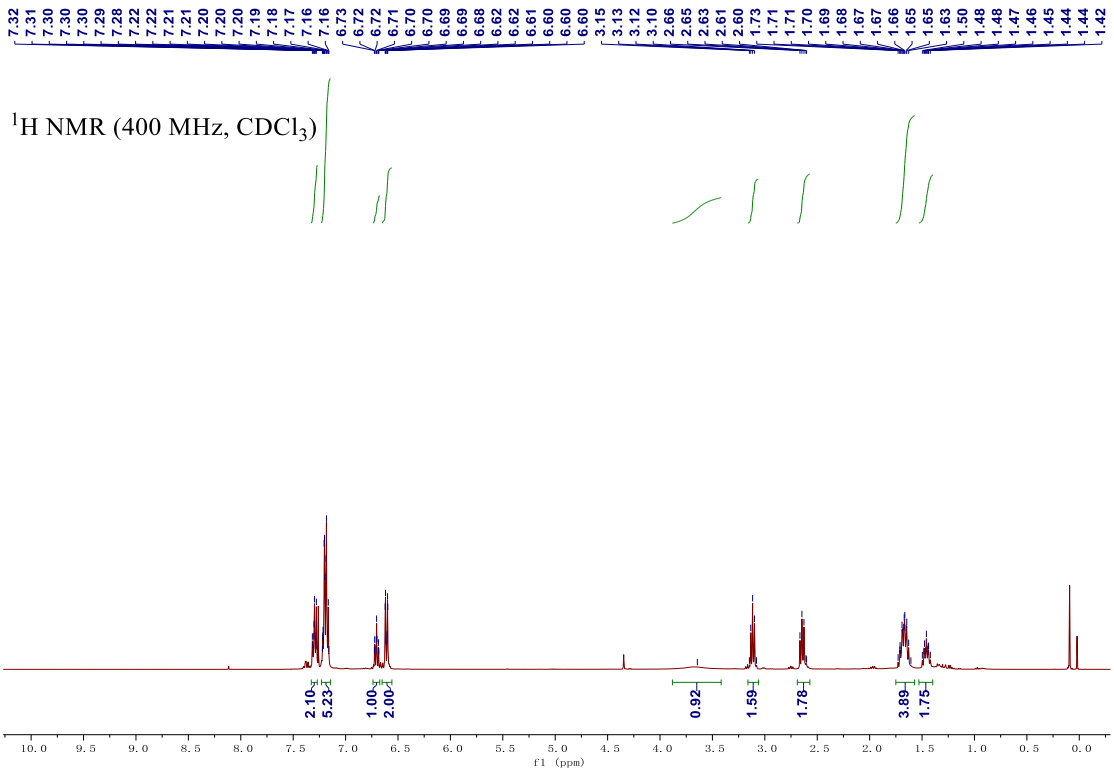
^{1}H NMR spectrum of the product deuterated **3da-*d*$_x$**

(iv-2)



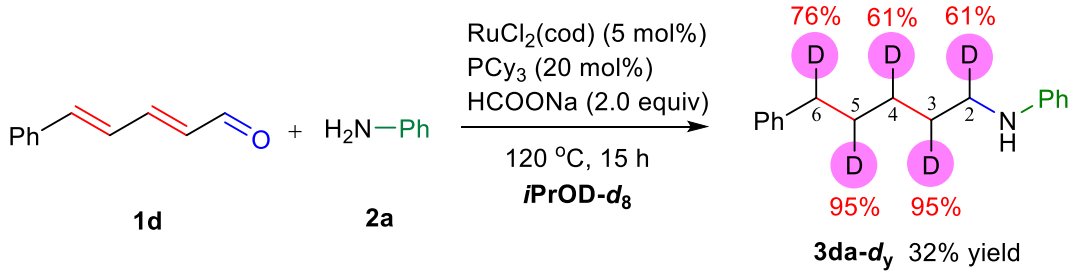
1d **2a** **3da-*d*′$_x$** 56% yield

A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg) and DCOONa (2.0 equivalents, 0.2 mmol, 14.0 mg) were charged into a reaction tube, and then to which were added **1d** (1.0 equivalent, 0.1 mmol, 15.8 mg), **2a** (1.5 equivalents, 0.15 mmol, 14.0 mg) and dry *i*PrOH (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 15 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3da-*d*′$_x$**. [^{1}H NMR analysis indicated 41%, 25%, 22% deuteration at the C(2), C(4), C(6) position, respectively.]



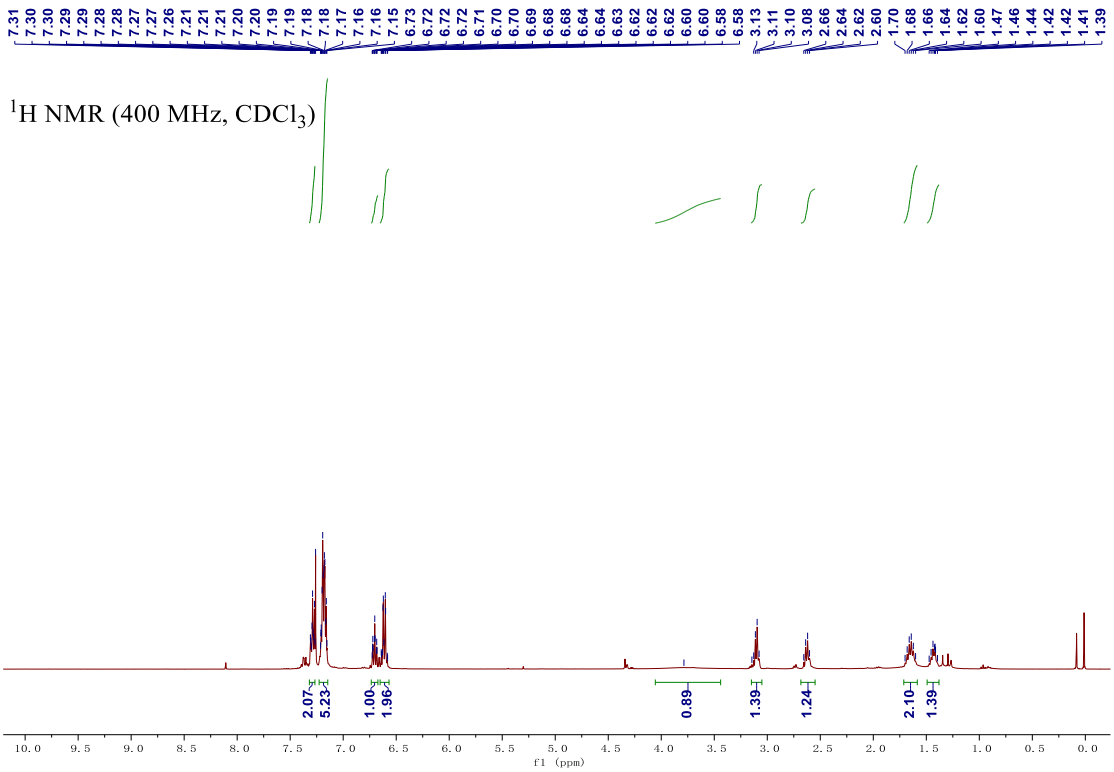
^{1}H NMR spectrum of the product deuterated **3da-*d*´$_x$**

(v)



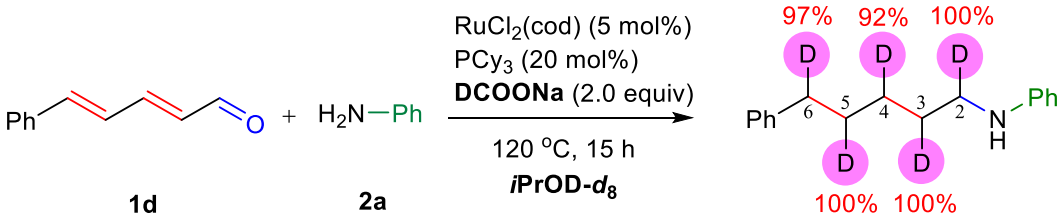
1d **2a** **3da-*d*$_y$** 32% yield

A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), HCOONa (2.0 equivalents, 0.2 mmol, 13.6 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **1d** (1.0 equivalent, 0.1 mmol, 15.8 mg), **2a** (1.5 equivalents, 0.15 mmol, 14.0 mg) and dry ***i*PrOD-*d*$_8$** (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 15 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3da-*d*$_y$**. [^{1}H NMR analysis indicated 61%, 95%, 61%, 95%, 76% deuteration at the C(2), C(3), C(4), C(5), C(6) position, respectively.]

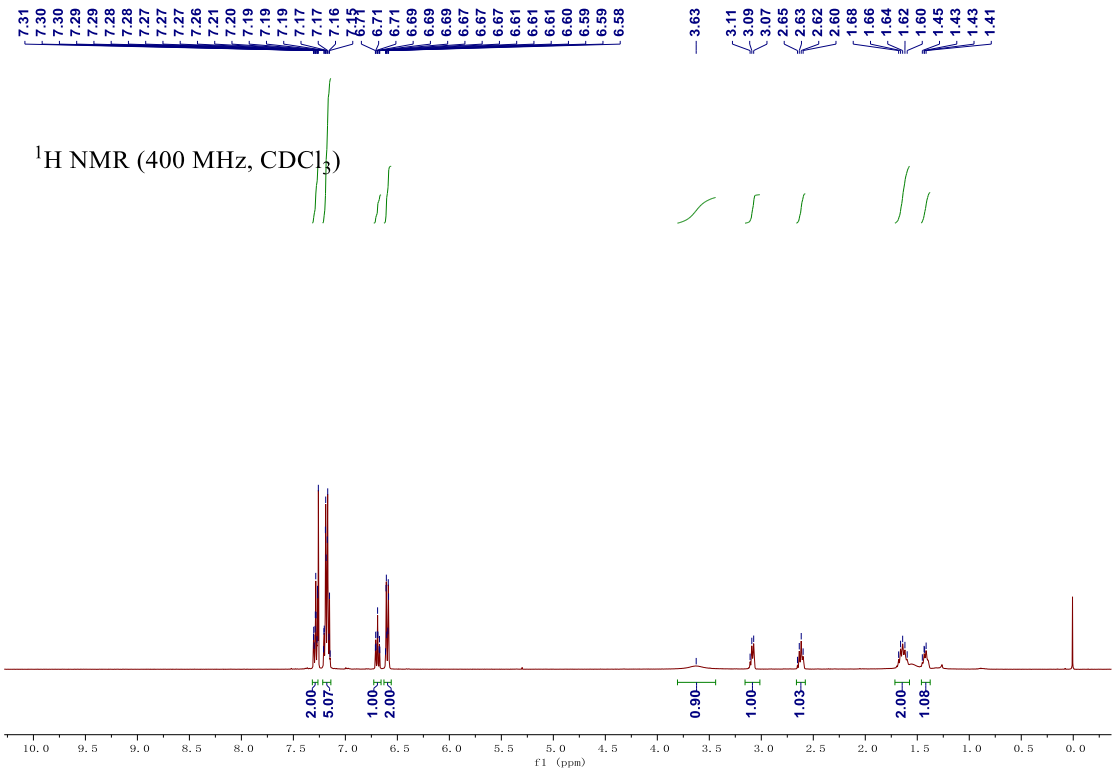


^{1}H NMR spectrum of the product deuterated **3da-*d*$_y$**

(vi)



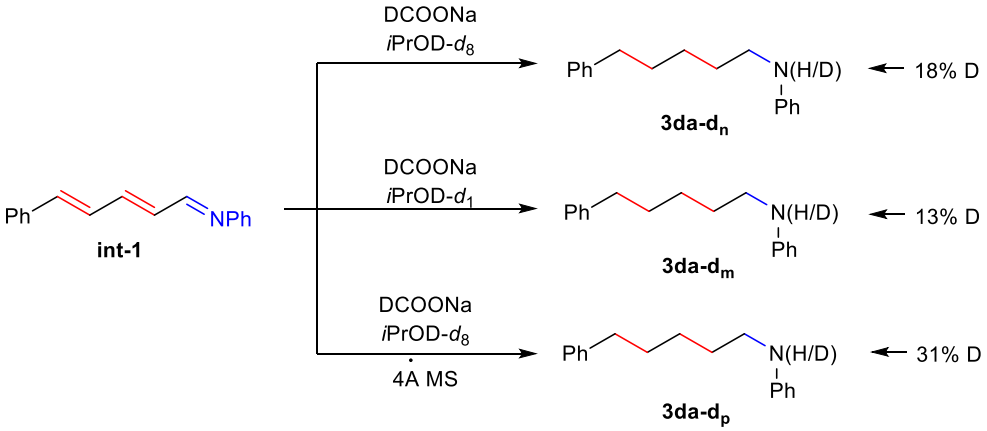
A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), DCOONa (2.0 equivalents, 0.2 mmol, 14.0 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **1d** (1.0 equivalent, 0.1 mmol, 15.8 mg), **2a** (1.5 equivalents, 0.15 mmol, 14.0 mg) and dry ***i*PrOD-d_8** (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 15 h. After the solvent was removed under reduced pressure, the residue was purified by flash-column chromatography to afford **3da-d_z**. [^{1}H NMR analysis indicated 100%, 100%, 92%, 100%, 97% deuteration at the C(2), C(3), C(4), C(5), C(6) position, respectively.]



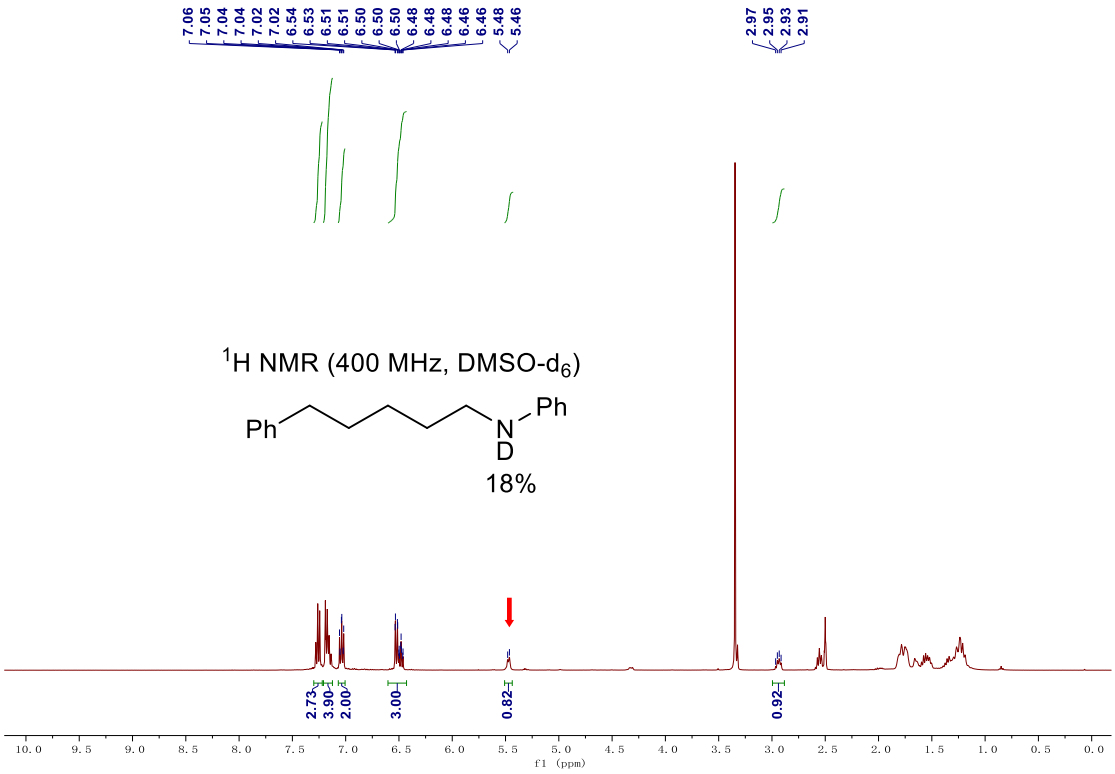
^{1}H NMR (400 MHz, $CDCl_3$)

^{1}H NMR spectrum of the product deuterated **3da-*d*$_z$**

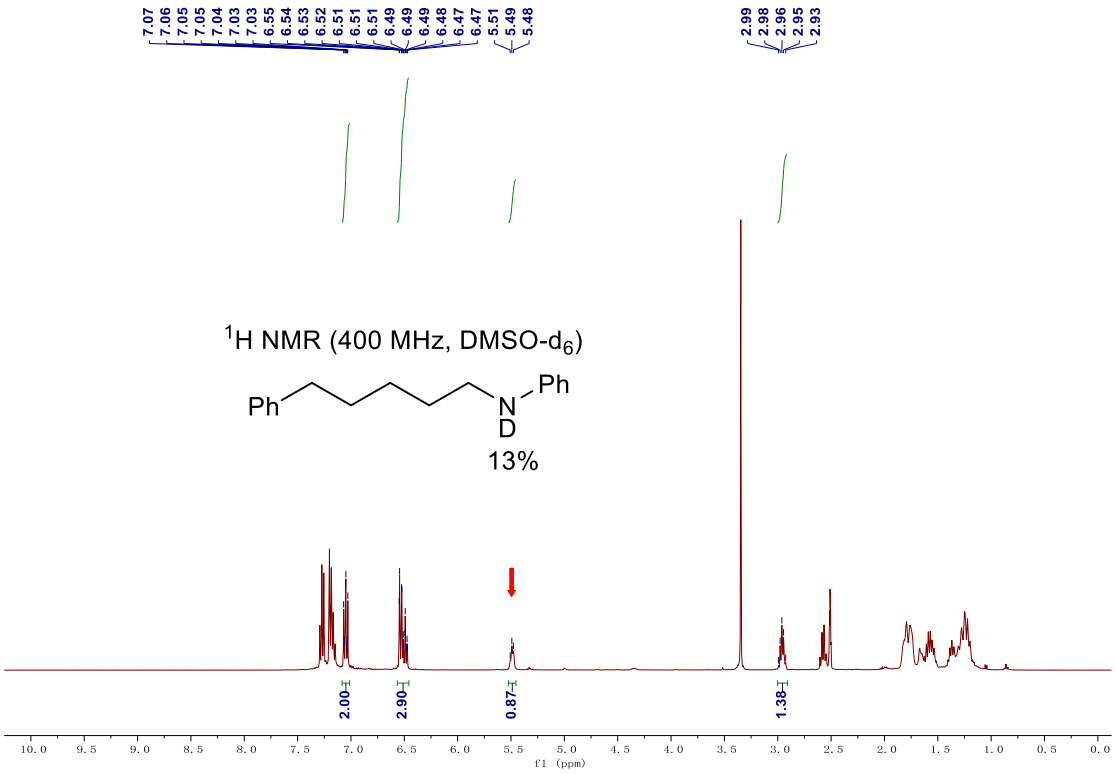
(vii)



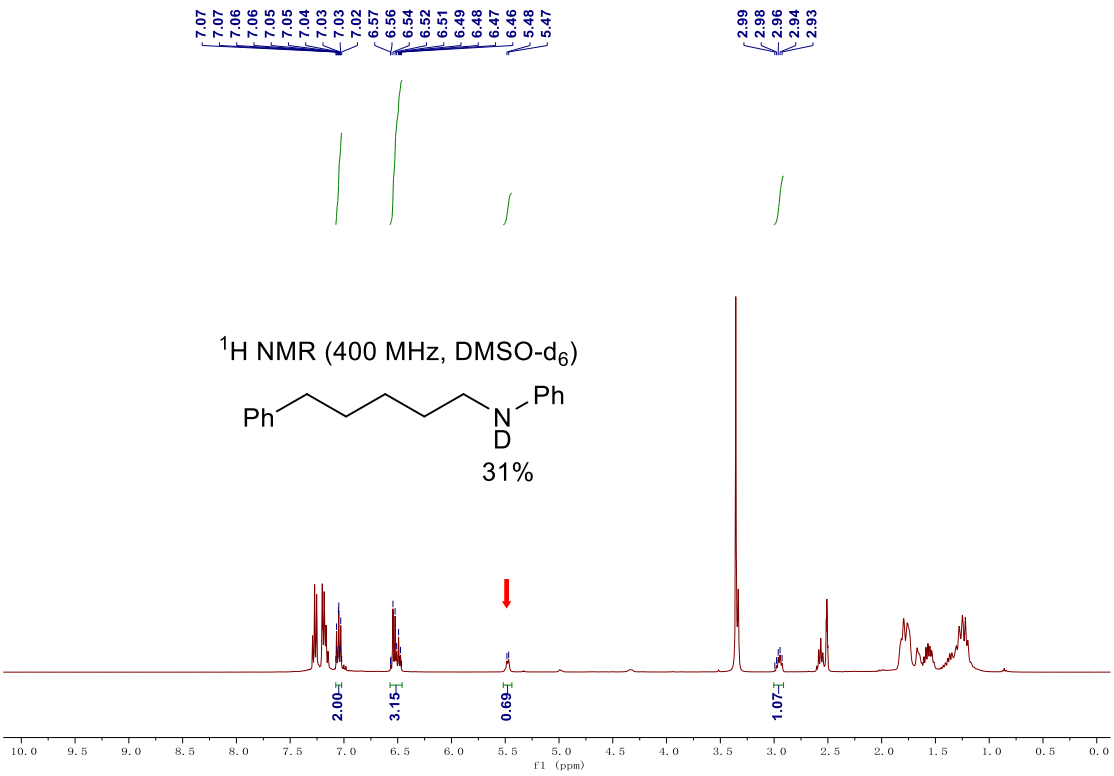
A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), DCOONa (2.0 equivalents, 0.2 mmol, 14.0 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **int-1** (1.0 equivalent, 0.1 mmol, 23.3 mg) and ***i*PrOD-*d*$_8$** (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 15 h. After the solvent was removed under reduced pressure, the crude product **3da-d$_n$** was obtained. [DMSO-d_6 analysis indicated 18% deuteration at the N position.]



A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), DCOONa (2.0 equivalents, 0.2 mmol, 14.0 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg) were charged into a reaction tube, and then to which were added **int-1** (1.0 equivalent, 0.1 mmol, 23.3 mg) and ***i*PrOD-*d*$_1$** (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 15 h. After the solvent was removed under reduced pressure, the crude product **3da-d$_m$** was obtained. [DMSO-d_6 analysis indicated 13% deuteration at the N position.]



A mixture of $RuCl_2$(cod) (5 mol%, 0.005 mmol, 1.4 mg), DCOONa (2.0 equivalents, 0.2 mmol, 14.0 mg), PCy_3 (20 mol%, 0.02 mmol, 5.6 mg), 4Å MS (100 mg) were charged into a reaction tube, and then to which were added **int-1** (1.0 equivalent, 0.1 mmol, 23.3 mg) and ***i*PrOD-*d*$_8$** (1.0 mL) under N_2 atmosphere. The reaction mixture was stirred at 120 ºC heated by metal sand bath for 15 h. After the solvent was removed under reduced pressure, the crude product **3da-d$_p$** was obtained. [DMSO-d_6 analysis indicated 31% deuteration at the N position.]



5. References

(1) P. Kumar, N. Kaur, R. Kumar and P. Banerjee, *α,β*-Unsaturated Carbonyls for One-Pot Transition-Metal-Free Access to 3,6-Dihydro-2H-pyrans, *J. Org. Chem*., 2022, **87**, 7167−7178.

(2) J. S. Meisner, D. F. Sedbrook, M. Krikorian, J. Chen, A. Sattler, M. E. Carnes, C. B. Murray, M. Steigerwald and C. Nuckolls, Functionalizing molecular wires: a tunable class of α,ω-diphenyl-μ,ν-dicyano-oligoenes, *Chem. Sci*., 2012, **3**, 1007–1014.

(3) I. M. Ferreira, E. B. Meira, I. G. Rosset and A. L. M. Porto, Chemoselective biohydrogenation of α,β- and α,β,γ,δ-unsaturated ketones by the marine-derived fungus Penicillium citrinum CBMAI 1186 in a biphasic system, *J. Mol. Catal. B: Enzym*., 2015, **115**, 59−65.

(4) J.-G. Zhao, X.-L. Zheng, S.-K. Tao, Y.-X. Zhu, J.-W. Yi, S.-B. Tang, R.-X. Li, H. Chen, H.-Y. Fu and M.-L. Yuan, Selective Rhodium-Catalyzed Hydroformylation of Terminal Arylalkynes and Conjugated Enynes to (Poly)enals Enabled by a *π*-acceptor Biphosphoramidite Ligand, *Org. Lett*., 2021, **23**, 6067−6072.

(5) R. Singh and S. K. Ghosh, A dimethylsulfonium methylide mediated highly regioselective olefination of conjugated polyolefin 1,1-dioates to conjugated polyene-2-yl-malonates and their applications in Diels–Alder reactions, *Tetrahedron*, 2010, **66**,

2284–2292.

(6) I. Mandal, A. Mandal, M. A. Rahman and A. F. M. Kilbinger, Chain transfer agents for the catalytic ring opening metathesis polymerization of norbornenes, *Chem. Sci*., 2022, **13**, 12469–12478.

(7) T.-J. Hu, M.-Y. Li, Q. Zhao, C.-G. Feng and G.-Q. Lin, Highly Stereoselective Synthesis of 1,3-Dienes through an Aryl to Vinyl 1,4-Palladium Migration/Heck Sequence, *Angew. Chem. Int. Ed*., 2018, **57**, 5871–5875.

(8) X.-T. Wu, F. Xie, Z. Ling, L. Tang and W.-B. Zhang, Regio- and Enantioselective Copper-Catalyzed 1,4-Conjugate Addition of Trimethylaluminium to Linear α,β,γ,δ-Unsaturated Alkyl Ketones, *Adv. Synth. Catal*., 2016, **358**, 2510–2518.

(9) C.-Y. Li, W.-K. Lu, B. Lu, W.-F. Li, X.-M. Xie and Z.-G. Zhang, Ru-Catalyzed Chemo- and Enantioselective Hydrogenation of 2,4-Pentadien-1-ones: Synthesis of Chiral 2,4-Pentadien-1-ols, *J. Org. Chem*., 2019, **84**, 16086−16094.

(10) R. Katoono, H. Kawai, M. Ohkita, K. Fujiwarab and T. Suzuki, A C_3-symmetric chiroptical molecular propeller based on hexakis(phenylethynyl)benzene with a threefold terephthalamide: stereospecific propeller generation through the cooperative transmission of point chiralities on the host and guest upon complexation, *Chem. Commun*., 2013, **49**, 10352−10354.

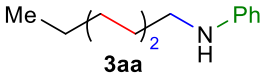
(11) C. Li, Y.-L. Huang, S. Cao, Y.-H. Luo, Y. Zhang and G. Yang, A robust and facile method for desulfonation to amines, *Org. Chem. Front.*, 2021, **8**, 6182–6186.

(12) Z.-P. Fan, X.-Y. Li, B. Sun, C.-L. Sun, Z.-F. Shi, X.-F. Shao and H.-L. Zhang, Constructing two-dimensional crossed molecular packing through branching chain engineering of amino-indenofluorene derivatives, *J. Mater. Chem. C*., 2022, **10**, 8666–8673.

(13) J. C. K. Chu, D. M. Dalton and T. Rovis, Zn-Catalyzed Enantio- and Diastereoselective Formal [4 + 2] Cycloaddition Involving Two Electron-Deficient Partners: Asymmetric Synthesis of Piperidines from 1-Azadienes and Nitro-Alkenes, *J. Am. Chem. Soc*., 2015, **137**, 4445−4452.

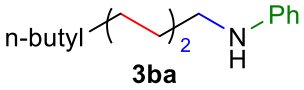
(14) P. Valenta, N. A. Drucker, J. W. Bode and P. J. Walsh, Simple One-pot Conversion of Aldehydes and Ketones to Enals, *Org. Lett*., 2009, **11**, 2117-2119.

6. Characterization Data



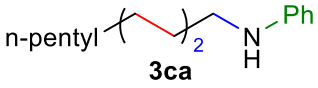
3aa

N-heptylaniline **(3aa).** Eluent: PE/EA = 100:3, brownish-yellow oil (17.6 mg, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 – 7.14 (m, 2H), 6.73 – 6.57 (m, 3H), 3.59 (s, 1H), 3.15 – 3.06 (m, 2H), 1.68 – 1.58 (m, 2H), 1.43 – 1.26 (m, 8H), 0.94 – 0.87 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 129.35, 117.2, 112.8, 44.1, 32.0, 29.7, 29.3, 27.3, 22.8, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{13}H_{22}N^+$ 192.1747, Found: 192.1745.



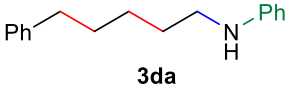
3ba

N-nonylaniline **(3ba).** Eluent: PE/EA = 100:3, yellow oil (19.5 mg, 89%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 – 7.14 (m, 2H), 6.70 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.64 – 6.59 (m, 2H), 3.60 (s, 1H), 3.11 (t, *J* = 7.1 Hz, 2H), 1.67 – 1.58 (m, 2H), 1.46 – 1.27 (m, 12H), 0.90 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 129.3, 117.2, 112.8, 44.1, 32.0, 29.73, 29.71, 29.6, 29.4, 27.3, 22.8, 14.3. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{15}H_{26}N^+$ 220.2060, Found: 220.2057.



3ca

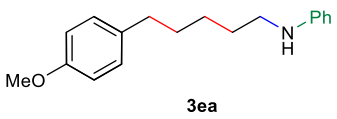
N-decylaniline **(3ca).** Eluent: PE/EA = 100:3, brownish-yellow oil (19.1 mg, 82%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.23 – 7.16 (m, 2H), 6.71 (dd, *J* = 7.8, 6.6 Hz, 1H), 6.65 – 6.59 (m, 2H), 3.60 (s, 1H), 3.12 (t, *J* = 7.2 Hz, 2H), 1.67 – 1.60 (m, 2H), 1.45 – 1.39 (m, 2H), 1.36 – 1.27 (m, 12H), 0.91 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 148.7, 129.3, 117.2, 112.8, 44.1, 32.0, 29.75, 29.7, 29.6, 29.5, 27.3, 22.8, 14.25. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{28}N^+$ 234.2216, Found: 234.2215.



3da

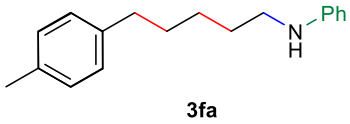
N-(5-phenylpentyl)aniline **(3da).** Eluent: PE/EA = 50:1, light yellow oil (22.2 mg, 93%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.25 (m, 2H), 7.24 – 7.15 (m, 5H), 6.71 (t, *J* = 7.3 Hz, 1H), 6.61 (d, *J* = 7.9 Hz, 2H), 3.64 (s, 1H), 3.12 (t, *J* = 7.1 Hz, 2H), 2.65 (t, *J* = 7.7 Hz, 2H), 1.74 – 1.59 (m, 4H), 1.52 – 1.41 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 142.6, 129.4, 128.5, 128.4, 125.8, 117.3, 112.85, 44.0, 36.0, 31.4, 29.55, 26.9. HRMS (ESI-TOF) m/z: [M

+ H]$^+$ Calcd for $C_{17}H_{22}N^+$ 240.1747, Found: 240.1746.



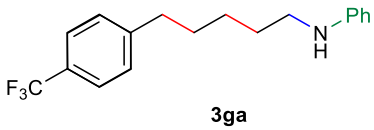
3ea

N-(5-(4-methoxyphenyl)pentyl)aniline **(3ea).** Eluent: PE/EA = 50:1, light yellow oil (22.4 mg, 83%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.20 – 7.17 (m, 2H), 7.13 – 7.08 (m, 2H), 6.86 – 6.83 (m, 2H), 6.73 – 6.68 (m, 1H), 6.62 – 6.58 (m, 2H), 3.80 (s, 3H), 3.65 (s, 1H), 3.11 (t, *J* = 7.1 Hz, 2H), 2.62 – 2.54 (m, 2H), 1.71 – 1.59 (m, 4H), 1.49 – 1.39 (m, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.8, 148.5, 134.7, 129.4, 129.36, 117.3, 113.85, 113.8, 112.9, 55.4, 44.1, 35.05, 31.6, 29.5, 26.8. HRMS (ESI-TOF) m/z: [M + H]$^+$ Calcd for $C_{18}H_{24}NO^+$ 270.1852, Found: 270.1851.



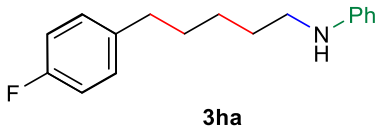
3fa

N-(5-(p-tolyl)pentyl)aniline (**3fa**). Eluent: PE/EA = 50:1, brown oil (20.3 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 – 7.15 (m, 2H), 7.14 – 7.07 (m, 4H), 6.74 – 6.67 (m, 1H), 6.63 – 6.58 (m, 2H), 3.59 (s, 1H), 3.12 (t, *J* = 7.1 Hz, 2H), 2.61 (t, *J* = 7.7 Hz, 2H), 2.34 (s, 3H), 1.72 – 1.59 (m, 4H), 1.52 – 1.40 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.6, 139.5, 135.25, 129.35, 129.1, 128.4, 117.2, 112.8, 44.0, 35.5, 31.5, 29.6, 26.9, 21.1. HRMS (ESI-TOF) m/z: [M + H]$^+$ Calcd for $C_{18}H_{24}N^+$ 254.1903, Found: 254.1902.



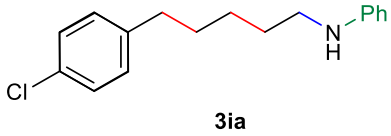
3ga

N-(5-(4-(trifluoromethyl)phenyl)pentyl)aniline (**3ga**). Eluent: PE/EA = 20:1, light brown oil (27.1 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.23 – 7.15 (m, 2H), 6.75 – 6.68 (m, 1H), 6.64 – 6.58 (m, 2H), 3.60 (s, 1H), 3.13 (t, *J* = 7.1 Hz, 2H), 2.70 (t, *J* = 7.7 Hz, 2H), 1.76 – 1.62 (m, 4H), 1.52 – 1.41 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 146.7, 129.4, 128.8, 128.2 (q, *J* = 32.1 Hz, 1C), 125.3 (q, *J* = 3.8 Hz, 1C), 124.5 (q, *J* = 270.0 Hz, 1C), 117.3, 112.8, 43.9, 35.8, 31.1, 29.5, 26.8. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.19. HRMS (ESI-TOF) m/z: [M + H]$^+$ Calcd for $C_{18}H_{21}F_3N^+$ 308.1621, Found: 308.1621.



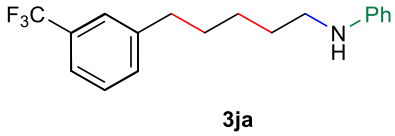
3ha

N-(5-(4-fluorophenyl)pentyl)aniline **(3ha).** Eluent: PE/EA = 50:1, yellow oil (21.9 mg, 85%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.07 (m, 4H), 7.04 – 6.90 (m, 2H), 6.71 (t, *J* = 7.3 Hz, 1H), 6.61 (d, *J* = 7.9 Hz, 2H), 3.61 (s, 1H), 3.12 (t, *J* = 7.1 Hz, 2H), 2.61 (t, *J* = 7.7 Hz, 2H), 1.66 (p, *J* = 7.6 Hz, 4H), 1.50 – 1.38 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.3 (d, *J* = 241.5 Hz, 1C), 160.1, 148.5, 138.16 (d, *J* = 3.2 Hz, 1C), 129.8 (d, *J* = 7.7 Hz, 1C), 129.4, 117.3, 115.1 (d, *J* = 20.7 Hz, 1C), 112.85, 44.0, 35.15, 31.5, 29.5, 26.8. ^{19}F NMR (376 MHz, $CDCl_3$) δ -117.96. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{21}FN^+$ 258.1653, Found: 258.1651.



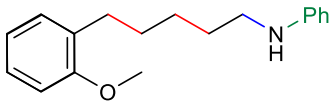
3ia

N-(5-(4-chlorophenyl)pentyl)aniline (**3ia**). Eluent: PE/EA = 50:1, light yellow oil (24.3 mg, 89%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.23 (m, 2H), 7.21 – 7.15 (m, 2H), 7.13 – 7.08 (m, 2H), 6.74 – 6.67 (m, 1H), 6.63 – 6.57 (m, 2H), 3.58 (s, 1H), 3.11 (t, *J* = 7.1 Hz, 2H), 2.63 – 2.58 (m, 2H), 1.71 – 1.59 (m, 4H), 1.49 – 1.38 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 141.0, 131.5, 129.9, 129.4, 128.5, 117.3, 112.8, 44.0, 35.3, 31.3, 29.5, 26.8. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{21}ClN^+$ 274.1357, Found: 274.1357.



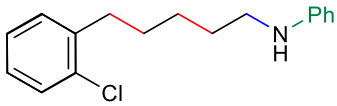
3ja

N-(5-(3-(trifluoromethyl)phenyl)pentyl)aniline (**3ja**). Eluent: PE/EA = 20:1, light yellow oil (20.0 mg, 65%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.33 (m, 4H), 7.22 – 7.15 (m, 2H), 6.74 – 6.67 (m, 1H), 6.64 – 6.57 (m, 2H), 3.61 (s, 1H), 3.12 (t, *J* = 7.1 Hz, 2H), 2.75 – 2.64 (m, 2H), 1.75 – 1.61 (m, 4H), 1.53 – 1.40 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 143.4, 131.9, 130.7 (q, *J* = 31.8 Hz, 1C), 129.4, 128.8, 125.2 (q, *J* = 3.7 Hz, 1C), 124.4 (q, *J* = 270.4 Hz, 1C), 122.8 (q, *J* = 4.0 Hz, 1C), 117.3, 112.8, 43.9, 35.8, 31.2, 29.5, 26.9. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.48. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{21}F_3N^+$ 308.1621, Found: 308.1618.



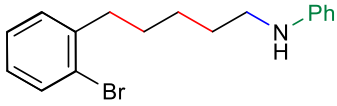
3ka

N-(5-(2-methoxyphenyl)pentyl)aniline (**3ka**). Eluent: PE/EA = 50:1, light yellow oil (18.1 mg, 67%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.10 (m, 4H), 6.95 – 6.83 (m, 2H), 6.70 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.65 – 6.57 (m, 2H), 3.84 (s, 3H), 3.60 (s, 1H), 3.13 (t, *J* = 7.1 Hz, 2H), 2.68 – 2.62 (m, 2H), 1.73 – 1.60 (m, 4H), 1.52 – 1.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.55, 148.6, 131.0, 129.9, 129.3, 127.0, 120.5, 117.2, 112.8, 110.4, 55.4, 44.05, 30.2, 29.7, 29.6, 27.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{24}NO^+$ 270.1852, Found: 270.1852.



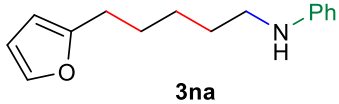
3la

N-(5-(2-chlorophenyl)pentyl)aniline (**3la**). Eluent: PE/EA = 50:1, brown oil (19.6 mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.33 (m, 1H), 7.24 – 7.11 (m, 5H), 6.74 – 6.67 (m, 1H), 6.64 – 6.58 (m, 2H), 3.61 (s, 1H), 3.18 – 3.08 (m, 2H), 2.82 – 2.72 (m, 2H), 1.77 – 1.61 (m, 4H), 1.56 – 1.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.6, 140.15, 134.0, 130.5, 129.6, 129.4, 127.3, 126.8, 117.25, 112.8, 44.0, 33.65, 29.7, 29.5, 27.0. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{21}ClN^+$ 274.1357, Found: 274.1357.



3ma

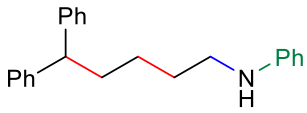
N-(5-(2-bromophenyl)pentyl)aniline (**3ma**). Eluent: PE/EA = 50:1, brown oil (19.8 mg, 62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.24 – 7.14 (m, 4H), 7.09 – 7.02 (m, 1H), 6.73 – 6.66 (m, 1H), 6.63 – 6.58 (m, 2H), 3.65 (s, 1H), 3.13 (t, *J* = 7.1 Hz, 2H), 2.79 – 2.71 (m, 2H), 1.72 – 1.62 (m, 4H), 1.53 – 1.44 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 141.8, 132.9, 130.45, 129.4, 127.6, 127.5, 124.5, 117.3, 112.8, 44.0, 36.2, 29.85, 29.5, 27.0. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{21}BrN^+$ 318.0852, Found: 318.0852.



3na

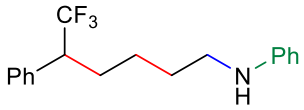
N-(5-(furan-2-yl)pentyl)aniline **(3na).** Eluent: PE/EA = 50:1, light yellow oil (16.1 mg, 70%).^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (dd, *J* = 1.9, 0.9 Hz, 1H), 7.23 – 7.15 (m, 2H), 6.71 (tt, *J* = 7.4, 1.1 Hz,

1H), 6.65 – 6.59 (m, 2H), 6.31 (dd, *J* = 3.2, 1.9 Hz, 1H), 6.03 – 5.97 (m, 1H), 3.58 (s, 1H), 3.13 (t, *J* = 7.1 Hz, 2H), 2.67 (t, *J* = 7.8 Hz, 2H), 1.78 – 1.61 (m, 4H), 1.53 – 1.42 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 148.6, 140.9, 129.3, 117.25, 112.8, 110.2, 104.9, 44.0, 29.4, 28.0, 27.98, 26.8. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{15}H_{20}NO^+$ 230.1539, Found: 230.1539.



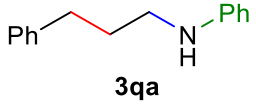
3oa

N-(5,5-diphenylpentyl)aniline (**3oa**). Eluent: PE/EA = 50:1, light yellow oil (26.5 mg, 84%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.26 (m, 4H), 7.26 – 7.22 (m, 4H), 7.21 – 7.13 (m, 4H), 6.72 – 6.65 (m, 1H), 6.59 – 6.54 (m, 2H), 3.91 (t, *J* = 7.8 Hz, 1H), 3.58 (s, 1H), 3.12 – 3.02 (m, 2H), 2.15 – 2.03 (m, 2H), 1.71 – 1.60 (m, 2H), 1.44 – 1.33 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 145.1, 129.35, 128.7, 128.6, 128.0, 127.9, 126.3, 117.3, 112.9, 51.4, 43.9, 35.6, 29.6, 25.7. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{26}N^+$ 316.2060, Found: 316.2059.



3pa

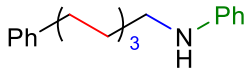
N-(6,6,6-trifluoro-5-phenylhexyl)aniline (**3pa**). Eluent: PE/EA = 20:1, yellow oil (20.3 mg, 66%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.31 (m, 3H), 7.31 – 7.21 (m, 2H), 7.19 – 7.11 (m, 2H), 6.72 – 6.66 (m, 1H), 6.58 – 6.49 (m, 2H), 3.50 (s, 1H), 3.30 – 3.16 (m, 1H), 3.12 – 2.98 (m, 2H), 2.16 – 1.99 (m, 1H), 1.99 – 1.85 (m, 1H), 1.76 – 1.50 (m, 2H), 1.37 – 1.21 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.3, 134.8 (q, *J* = 1.7 Hz, 1C), 129.4, 129.1, 128.9, 128.3, 117.4, 112.8, 50.2 (q, *J* = 26.0 Hz, 1C), 43.7, 29.3, 28.6, 24.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ -69.74. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{21}F_3N^+$ 308.1621, Found: 308.1621.



3qa

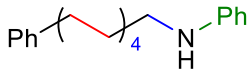
N-(3-phenylpropyl)aniline **(3qa).** Eluent: PE/EA = 20:1, yellow oil (20.0 mg, 95%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.6 Hz, 2H), 7.25 – 7.15 (m, 5H), 6.71 (t, *J* = 7.3 Hz, 1H), 6.60 (d, *J* = 7.9 Hz, 2H), 3.61 (s, 1H), 3.17 (t, *J* = 7.0 Hz, 2H), 2.76 (t, *J* = 7.6 Hz, 2H), 2.03 – 1.92 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 141.8, 129.4, 128.6, 128.5, 126.1, 117.3, 112.9,

43.55, 33.5, 31.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{15}H_{18}N^+$ 212.1434, Found: 212.1431.



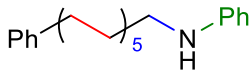
3ra

N-(7-phenylheptyl)aniline (**3ra**). Eluent: PE/EA = 50:2, brownish-yellow oil (18.0 mg, 67%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 2H), 7.23 – 7.16 (m, 5H), 6.71 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.64 – 6.59 (m, 2H), 3.57 (s, 1H), 3.11 (t, *J* = 7.1 Hz, 2H), 2.63 (t, *J* = 7.7 Hz, 2H), 1.69 – 1.60 (m, 4H), 1.46 – 1.35 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.6, 142.9, 129.3, 128.5, 128.4, 125.7, 117.2, 112.8, 44.1, 36.1, 31.6, 29.7, 29.4, 29.3, 27.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{26}N^+$ 268.2060, Found: 268.2059.



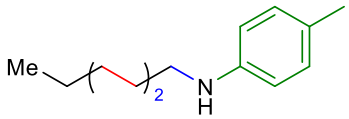
3sa

N-(9-phenylnonyl)aniline (**3sa**). Eluent: PE/EA = 100:3, light yellow oil (18.9 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.26 (m, 2H), 7.22 – 7.14 (m, 5H), 6.72 – 6.66 (m, 1H), 6.64 – 6.58 (m, 2H), 3.59 (s, 1H), 3.11 (t, *J* = 7.1 Hz, 2H), 2.62 (t, *J* = 7.7 Hz, 2H), 1.67 – 1.56 (m, 4H), 1.45 – 1.27 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 143.0, 129.35, 128.5, 128.4, 125.7, 117.2, 112.8, 44.1, 36.1, 31.6, 29.7, 29.65, 29.6, 29.5, 29.4, 27.3. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{30}N^+$ 296.2373, Found: 296.2372.



3ta

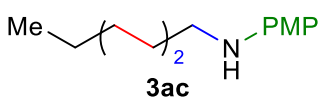
N-(11-phenylundecyl)aniline (**3ta**). Eluent: PE/EA = 50:1, light yellow oil (4.5 mg, 14%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 7.20 – 7.13 (m, 5H), 6.71 – 6.65 (m, 1H), 6.63 – 6.57 (m, 2H), 3.70 (s, 1H), 3.10 (t, *J* = 7.1 Hz, 2H), 2.63 – 2.56 (m, 2H), 1.65 – 1.58 (m, 5H), 1.43 – 1.26 (m, 13H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.65, 143.1, 129.4, 128.5, 128.4, 125.7, 117.25, 112.9, 44.2, 36.1, 31.7, 29.7, 29.7, 29.65, 29.6, 29.5, 27.3. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{34}N^+$ 324.2686, Found: 324.2682.



3ab

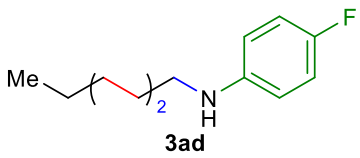
N-heptyl-4-methylaniline **(3ab).** Eluent: PE/EA = 100:1, brownish-yellow oil (18.5 mg, 90%). ^{1}H NMR (400

MHz, $CDCl_3$) δ 7.00 (d, *J* = 7.9 Hz, 2H), 6.61 – 6.50 (m, 2H), 3.46 (s, 1H), 3.09 (t, *J* = 7.2 Hz, 2H), 2.25 (s, 3H), 1.67 – 1.56 (m, 2H), 1.45 – 1.25 (m, 8H), 0.95 – 0.86 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.5, 129.8, 126.4, 113.0, 44.5, 32.0, 29.8, 29.3, 27.3, 22.8, 20.5, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{14}H_{24}N^+$ 206.1903, Found: 206.1901.



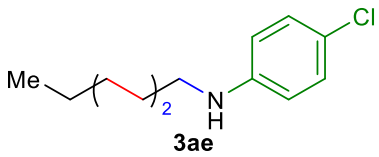
3ac

N-heptyl-4-methoxyaniline **(3ac).** Eluent: PE/EA = 50:2, yellow oil (14.2 mg, 64%). ^1H NMR (400 MHz, $CDCl_3$) δ 6.84 – 6.73 (m, 2H), 6.64 – 6.53 (m, 2H), 3.75 (s, 3H), 3.32 (s, 1H), 3.06 (t, *J* = 7.1 Hz, 2H), 1.68 – 1.54 (m, 2H), 1.47 – 1.21 (m, 8H), 0.96 – 0.83 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.1, 143.0, 115.0, 114.1, 55.95, 45.2, 31.95, 29.8, 29.3, 27.3, 22.8, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{14}H_{24}NO^+$ 222.1852, Found: 222.1849.



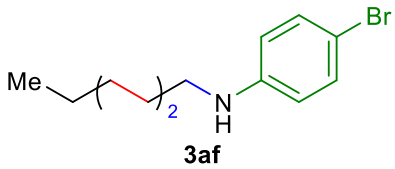
3ad

4-fluoro-N-heptylaniline **(3ad).** Eluent: PE/EA = 100:1, yellow oil (17.1 mg, 82%). ^1H NMR (400 MHz, $CDCl_3$) δ 6.93 – 6.84 (m, 2H), 6.57 – 6.49 (m, 2H), 3.47 (s, 1H), 3.06 (t, *J* = 7.1 Hz, 2H), 1.65 – 1.56 (m, 2H), 1.44 – 1.24 (m, 8H), 0.94 – 0.86 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8 (d, *J* = 232.8 Hz, 1C), 145.1 (d, *J* = 1.9 Hz, 1C), 115.7 (d, *J* = 22.3 Hz, 2C), 113.6 (d, *J* = 7.3 Hz, 2C), 44.9, 31.95, 29.7, 29.3, 27.3, 22.8, 14.2. ^{19}F NMR (376 MHz, $CDCl_3$) δ -128.60. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{13}H_{21}FN^+$ 210.1653, Found: 210.1651.



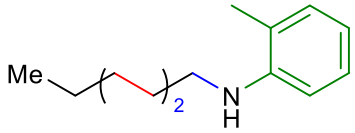
3ae

4-chloro-N-heptylaniline **(3ae).** Eluent: PE/EA = 100:1, light yellow oil (16.9 mg, 75%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, *J* = 8.7 Hz, 2H), 6.51 (d, *J* = 8.7 Hz, 2H), 3.60 (s, 1H), 3.07 (t, *J* = 7.3 Hz, 2H), 1.66 – 1.54 (m, 2H), 1.44 – 1.25 (m, 8H), 0.94 – 0.84 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.2, 129.1, 121.65, 113.8, 44.2, 31.9, 29.6, 29.2, 27.2, 22.75, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{13}H_{21}ClN^+$ 226.1357, Found: 226.1355.



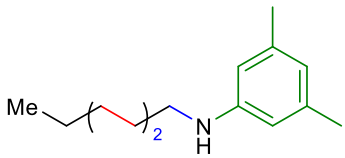
3af

4-bromo-N-heptylaniline **(3af).** Eluent: PE/EA = 50:1, light yellow oil (15.4 mg, 57%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.21 (m, 2H), 6.50 – 6.44 (m, 2H), 3.62 (s, 1H), 3.06 (t, *J* = 7.1 Hz, 2H), 1.64 – 1.57 (m, 2H), 1.42 – 1.27 (m, 8H), 0.92 – 0.86 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 132.0, 114.3, 108.65, 44.1, 31.9, 29.55, 29.2, 27.2, 22.8, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{13}H_{21}BrN^+$ 270.0852, Found: 270.0852.



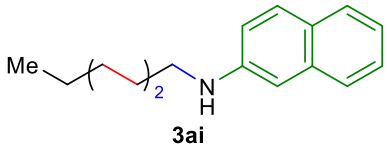
3ag

N-heptyl-2-methylaniline **(3ag).** Eluent: PE/EA = 100:1, yellow oil (14.4 mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 – 7.02 (m, 2H), 6.75 – 6.54 (m, 2H), 3.48 (s, 1H), 3.17 (t, *J* = 7.2 Hz, 2H), 2.15 (s, 3H), 1.73 – 1.63 (m, 2H), 1.47 – 1.27 (m, 8H), 0.98 – 0.82 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.5, 130.1, 127.3, 121.8, 116.75, 109.8, 44.1, 32.0, 29.8, 29.3, 27.4, 22.8, 17.6, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{14}H_{24}N^+$ 206.1903, Found: 206.1902.



3ah

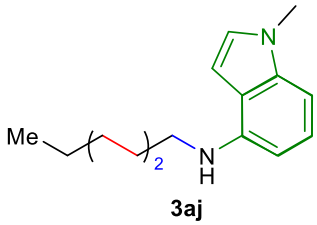
N-heptyl-3,5-dimethylaniline **(3ah).** Eluent: PE/EA = 100:1, yellow oil (17.1 mg, 78%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.38 (s, 1H), 6.27 (s, 2H), 3.51 (s, 1H), 3.10 (t, *J* = 7.1 Hz, 2H), 2.26 (s, 6H), 1.69 – 1.56 (m, 2H), 1.50 – 1.23 (m, 8H), 0.97 – 0.87 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.8, 138.95, 119.2, 110.8, 44.2, 32.0, 29.8, 29.3, 27.3, 22.8, 21.6, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{15}H_{26}N^+$ 220.2060, Found: 220.2059.



3ai

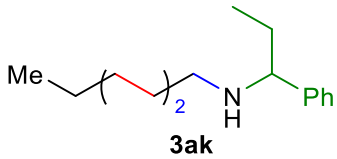
N-heptylnaphthalen-2-amine **(3ai).** Eluent: PE/EA = 100:1, yellow oil (12.1 mg, 50%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (dd, *J* = 19.2, 8.3 Hz, 3H), 7.36 (t, *J* = 7.6 Hz, 1H), 7.19 (t, *J* = 7.5 Hz, 1H), 6.88 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.81 (s, 1H), 3.78 (s, 1H), 3.22 (t, *J* = 7.1 Hz, 2H), 1.74 – 1.65 (m, 2H), 1.50 – 1.27 (m, 8H), 0.96 – 0.88 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.3, 135.4, 129.0, 127.8, 127.5, 126.4,

126.0, 121.9, 118.1, 104.3, 44.2, 32.0, 29.6, 29.3, 27.35, 22.8, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{24}N^+$ 242.1903, Found: 242.1903.



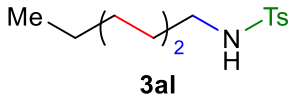
3aj

N-heptyl-1-methyl-1H-indol-4-amine **(3aj).** Eluent: PE/EA = 20:1, light yellow oil (9.7 mg, 40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 – 7.09 (m, 1H), 6.93 (d, *J* = 3.2 Hz, 1H), 6.77 – 6.71 (m, 1H), 6.39 – 6.35 (m, 1H), 6.29 (d, *J* = 7.6 Hz, 1H), 3.90 (s, 1H), 3.75 (s, 3H), 3.27 (t, *J* = 7.2 Hz, 2H), 1.78 – 1.66 (m, 2H), 1.50 – 1.24 (m, 8H), 0.94 – 0.86 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.9, 137.4, 126.5, 123.2, 99.4, 99.1, 96.9, 44.20, 33.2, 32.0, 29.8, 29.3, 27.4, 22.8, 14.3. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{25}N_2^+$ 245.2012, Found: 245.2012.



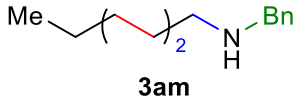
3ak

N-(1-phenylpropyl)heptan-1-amine **(3ak).** Eluent: PE/EA = 6:1, colorless oil (12.5 mg, 54%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.21 (m, 5H), 3.56 – 3.42 (m, 1H), 2.53 – 2.37 (m, 2H), 1.85 – 1.73 (m, 1H), 1.72 – 1.59 (m, 1H), 1.56 – 1.37 (m, 3H), 1.27 (s, 8H), 0.86 (dt, *J* = 26.1, 6.9 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 128.4, 127.45, 126.9, 65.4, 48.0, 32.0, 31.1, 30.4, 29.4, 27.5, 22.75, 14.2, 11.0. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{28}N^+$ 234.2216, Found: 234.2212.



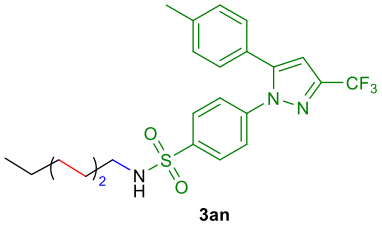
3al

N-heptyl-4-methylbenzenesulfonamide (**3al**). Eluent: PE/EA = 5:1, colorless oil (9.0 mg, 33%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 4.34 (t, *J* = 6.3 Hz, 1H), 2.96 – 2.88 (m, 2H), 2.43 (s, 3H), 1.53 – 1.10 (m, 10H), 0.85 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5, 137.0, 129.8, 127.2, 43.4, 31.8, 29.7, 28.9, 26.6, 22.7, 21.7, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{14}H_{24}NO_2S^+$ 270.1522, Found: 270.1522.



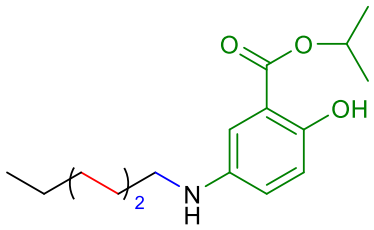
3am

N-benzylheptan-1-amine **(3am).** Eluent: PE/EA = 15:1, yellow oil (6.2 mg, 30%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 4H), 7.25 – 7.19 (m, 1H), 3.99 (d, *J* = 13.5 Hz, 1H), 3.20 (d, *J* = 13.5 Hz, 1H), 2.81 – 2.69 (m, 1H), 2.27 – 2.15 (m, 1H), 2.07 – 1.93 (m, 1H), 1.70 – 1.56 (m, 5H), 1.49 – 1.42 (m, 2H), 1.34 – 1.23 (m, 3H), 0.92 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 129.1, 128.2, 126.7, 62.0, 57.7, 52.1, 29.9, 25.5, 24.6, 24.0, 9.7. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{14}H_{24}N^+$ 206.1903, Found: 206.1901.



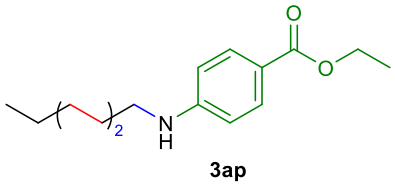
3an

N-heptyl-4-(5-(p-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)benzenesulfonamide (**3an**). Eluent: PE/EA = 10:1, light yellow oil (14.9 mg, 31%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.82 (m, 2H), 7.50 – 7.44 (m, 2H), 7.17 (d, *J* = 7.9 Hz, 2H), 7.10 (d, *J* = 8.2 Hz, 2H), 6.74 (s, 1H), 4.47 (t, *J* = 6.2 Hz, 1H), 2.94 (q, *J* = 6.8 Hz, 2H), 2.38 (s, 3H), 1.61 (s, 1H), 1.51 – 1.39 (m, 2H), 1.33 – 1.16 (m, 7H), 0.86 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.4, 142.6, 139.9, 139.7, 129.9, 128.8, 128.2, 125.8, 125.7, 106.4, 43.5, 31.8, 29.7, 28.85, 26.6, 22.65, 21.5, 14.15. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.46. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{28}F_3N_3NaO_2S^+$ 502.1747, Found: 502.1755.



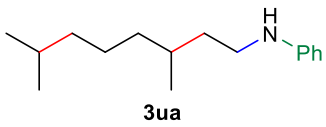
3ao

Isopropyl 5-(heptylamino)-2-hydroxybenzoate (**3ao**). Eluent: PE/EA = 20:1, yellow oil (15.8 mg, 54%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.34 (s, 1H), 7.04 (dd, *J* = 2.4, 1.0 Hz, 1H), 6.86 – 6.79 (m, 2H), 5.27 (hept, *J* = 6.2 Hz, 1H), 3.06 (t, *J* = 7.1 Hz, 2H), 1.66 – 1.55 (m, 2H), 1.44 – 1.23 (m, 14H), 0.94 – 0.83 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 154.3, 141.2, 122.4, 118.15, 112.9, 112.1, 69.1, 45.2 31.9, 29.7, 29.3, 27.3, 22.75, 22.0, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{28}NO_3^+$ 294.2064, Found: 294.2060.



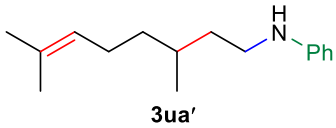
3ap

Ethyl 4-(heptylamino)benzoate (**3ap**). Eluent: PE/EA = 20:1, light yellow solid (7.1 mg, 27%, m.p. 67 – 69 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.81 (m, 2H), 6.57 – 6.49 (m, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 4.11 (s, 1H), 3.15 (t, *J* = 7.2 Hz, 2H), 1.65 – 1.59 (m, 2H), 1.42 – 1.26 (m, 11H), 0.92 – 0.85 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 152.2, 131.6, 118.4, 111.4, 60.3, 43.5, 31.9, 29.45, 29.2, 27.2, 22.75, 14.6, 14.2. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{25}NNaO_2^+$ 286.1778, Found: 286.1781.



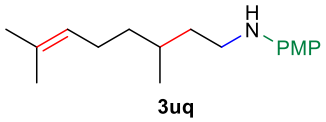
3ua

N-(3,7-dimethyloctyl)aniline (**3ua**). Eluent: PE/EA = 100:3, light yellow oil (11.9 mg, 51%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 – 7.14 (m, 2H), 6.71 – 6.66 (m, 1H), 6.63 – 6.58 (m, 2H), 3.54 (s, 1H), 3.20 – 3.05 (m, 2H), 1.69 – 1.59 (m, 1H), 1.55 – 1.39 (m, 3H), 1.36 – 1.23 (m, 3H), 1.21 – 1.09 (m, 3H), 0.93 (d, *J* = 6.5 Hz, 3H), 0.87 (d, *J* = 6.6 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 129.35, 117.2, 112.8, 42.1, 39.4, 37.4, 36.95, 31.0, 28.1, 24.9, 22.9, 22.8, 19.9. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{28}N^+$ 234.2216, Found: 234.2218.



3ua′

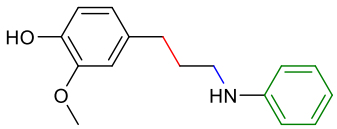
N-(3,7-dimethyloct-6-en-1-yl)aniline (**3ua´**). Eluent: PE/EA = 100:3, light yellow oil (8.3 mg, 36%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 – 7.13 (m, 2H), 6.72 – 6.65 (m, 1H), 6.64 – 6.57 (m, 2H), 5.15 – 5.04 (m, 1H), 3.56 (s, 1H), 3.19 – 3.04 (m, 2H), 2.10 – 1.91 (m, 2H), 1.73 – 1.58 (m, 7H), 1.50 – 1.30 (m, 3H), 1.25 – 1.16 (m, 1H), 0.95 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 131.5, 129.4, 124.8, 117.2, 112.8, 42.1, 37.2, 36.9, 30.6, 25.9, 25.6, 19.75, 17.8. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{26}N^+$ 232.2060, Found: 232.2060.



3uq

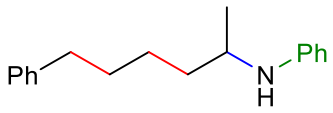
N-(3,7-dimethyloct-6-en-1-yl)-4-methoxyaniline **(3uq).** Eluent: PE/EA = 50:1, light yellow oil (24.1 mg, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81 – 6.76 (m, 2H), 6.62 –

6.56 (m, 2H), 5.16 – 5.06 (m, 1H), 3.75 (s, 3H), 3.15 – 3.00 (m, 2H), 2.10 – 1.88 (m, 2H), 1.73 – 1,68 (m, 3H), 1.68 – 1.50 (m, 5H), 1.48 – 1.33 (m, 2H), 1.27 – 1.14 (m, 1H), 0.99 – 0.84 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.1, 143.0, 131.4, 124.8, 115.0, 114.2, 56.0, 43.1, 37.25, 37.0, 30.6, 25.9, 25.6, 19.75, 17.8. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{28}NO^+$ 262.2165, Found: 262.2161.



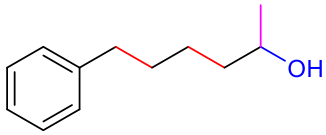
3va

2-methoxy-4-(3-(phenylamino)propyl)phenol (**3va**). Eluent: PE/EA = 5:1, light yellow oil (13.4 mg, 52%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 – 7.13 (m, 2H), 6.88 – 6.82 (m, 1H), 6.73 – 6.66 (m, 3H), 6.62 – 6.55 (m, 2H), 3.86 (s, 3H), 3.15 (t, *J* = 7.0 Hz, 2H), 2.68 (t, *J* = 7.6 Hz, 2H), 1.99 – 1.87 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 146.5, 143.9, 133.7, 129.4, 121.1, 117.35, 114.4, 112.9, 111.1, 56.0, 43.5, 33.2, 31.4. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{20}NO_2^+$ 258.1489, Found: 258.1493.



3aaa

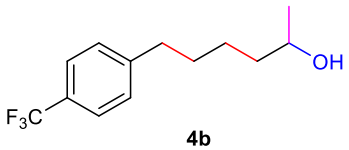
N-(6-phenylhexan-2-yl)aniline (**3aaa**). Eluent: PE/EA = 100:3, light yellow oil (6.4 mg, 25%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.26 (m, 2H), 7.22 – 7.12 (m, 5H), 6.66 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.60 – 6.54 (m, 2H), 3.56 – 3.25 (m, 2H), 2.66 – 2.55 (m, 2H), 1.70 – 1.59 (m, 3H), 1.52 – 1.37 (m, 3H), 1.16 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (100 MHz, DMSO) δ 148.8, 142.75, 129.4, 128.75, 128.7, 126.1, 115.5, 112.7, 47.6, 36.6, 35.7, 31.7, 25.9, 20.9. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{24}N^+$ 254.1903, Found: 254.1904.



4a

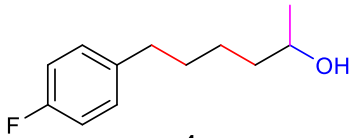
6-phenylhexan-2-ol (**4a**). Eluent: PE/EA = 5:1, colorless oil (16.1 mg, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.26 (m, 2H), 7.22 – 7.16 (m, 3H), 3.86 – 3.73 (m, 1H), 2.64 (t, *J* = 7.7 Hz, 2H), 1.71 – 1.61 (m, 2H), 1.56 – 1.33 (m, 5H), 1.19 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.7, 128.5, 128.4, 125.8, 68.2, 39.3, 36.0, 31.6, 25.6, 23.6. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{12}H_{18}ONa^+$ 201.1250,

Found: 201.1250.



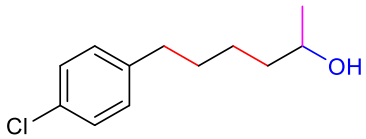
4b

6-(4-(trifluoromethyl)phenyl)hexan-2-ol (**4b**). Eluent: PE/EA = 5:1, colorless oil (18.5 mg, 75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.0 Hz, 2H), 7.32 – 7.27 (m, 2H), 3.87 – 3.72 (m, 1H), 2.68 (t, *J* = 7.7 Hz, 2H), 1.70 – 1.61 (m, 2H), 1.53 – 1.32 (m, 5H), 1.19 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.8, 128.8, 128.1 (q, *J* = 32.1 Hz, 1C), 125.3 (q, *J* = 3.7 Hz, 1C), 124.5 (q, *J* = 270.1 Hz, 1C), 68.15, 39.2, 35.9, 31.35, 25.5, 23.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.24. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{13}H_{17}F_3ONa^+$ 269.1124, Found: 269.1123.



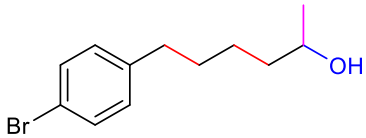
4c

6-(4-fluorophenyl)hexan-2-ol (**4c**). Eluent: PE/EA = 7:1, colorless oil (14.6 mg, 74%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 – 7.07 (m, 2H), 7.00 – 6.89 (m, 2H), 3.84 – 3.73 (m, 1H), 2.59 (t, *J* = 7.7 Hz, 2H), 1.66 – 1.56 (m, 2H), 1.54 – 1.27 (m, 5H), 1.18 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.3 (d, *J* = 241.5 Hz, 1C), 138.3 (d, *J* = 3.3 Hz, 1C), 129.8 (d, *J* = 7.6 Hz, 2C), 115.1 (d, *J* = 20.7 Hz, 2C), 68.2, 39.2, 35.2, 31.75, 25.5, 23.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -118.07. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{12}H_{17}FONa^+$ 219.1156, Found: 219.1152.



4d

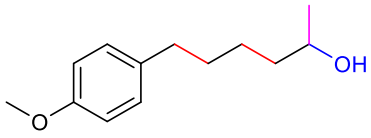
6-(4-chlorophenyl)hexan-2-ol (**4d**). Eluent: PE/EA = 7:1, colorless oil (15.3 mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.20 (m, 2H), 7.14 – 7.06 (m, 2H), 3.85 – 3.72 (m, 1H), 2.59 (t, *J* = 7.7 Hz, 2H), 1.67 – 1.56 (m, 2H), 1.52 – 1.29 (m, 5H), 1.18 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.1, 131.4, 129.85, 128.5, 68.2, 39.2, 35.4, 31.5, 25.45, 23.7. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{12}H_{17}ClONa^+$ 235.0860, Found: 235.0855.



4e

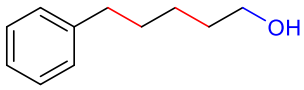
6-(4-bromophenyl)hexan-2-ol (**4e**). Eluent: PE/EA = 6:1, colorless oil (10.3 mg, 40%). ^{1}H NMR (400 MHz,

$CDCl_3$) δ 7.42 – 7.35 (m, 2H), 7.08 – 7.00 (m, 2H), 3.85 – 3.73 (m, 1H), 2.57 (t, *J* = 7.6 Hz, 2H), 1.67 – 1.57 (m, 2H), 1.51 – 1.28 (m, 5H), 1.18 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.6, 131.4, 130.3, 119.5, 68.2, 39.2, 35.4, 31.5, 25.45, 23.7. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{12}H_{17}{}^{81}BrONa^+$ 281.0335, Found: 281.0329.



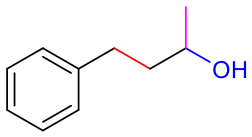
4f

6-(4-methoxyphenyl)hexan-2-ol (**4f**). Eluent: PE/EA = 5:1, light yellow oil (14.4 mg, 69%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.14 – 7.05 (m, 2H), 6.87 – 6.76 (m, 2H), 3.83 – 3.74 (m, 4H), 2.56 (t, *J* = 7.7 Hz, 2H), 1.69 – 1.57 (m, 2H), 1.52 – 1.29 (m, 5H), 1.18 (d, *J* = 6.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 134.8, 129.4, 113.8, 68.2, 55.4, 39.3, 35.1, 31.9, 25.5, 23.65. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{13}H_{20}O_2Na^+$ 231.1356, Found: 231.1352.



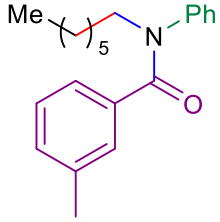
4g

5-phenylpentan-1-ol (**4g**). Eluent: PE/EA = 7:1, light yellow oil (11.7 mg, 71%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.27 (m, 2H), 7.22 – 7.15 (m, 3H), 3.70 – 3.60 (m, 2H), 2.67 – 2.59 (m, 2H), 1.70 – 1.58 (m, 5H), 1.45 – 1.36 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.7, 128.5, 128.4, 125.8, 63.1, 36.0, 32.8, 31.4, 25.5. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{11}H_{16}ONa^+$ 187.1093, Found: 187.1089.



4h

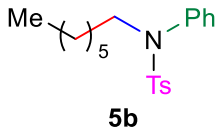
4-phenylbutan-2-ol (**4h**). Eluent: PE/EA = 10:1, colorless oil (12.9 mg, 86%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 2H), 7.25 – 7.16 (m, 3H), 3.89 – 3.79 (m, 1H), 2.82 – 2.64 (m, 2H), 1.86 – 1.72 (m, 2H), 1.62 (s, 1H), 1.24 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.2, 128.5, 125.9, 67.6, 40.95, 32.2, 23.7. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{10}H_{14}ONa^+$ 173.0937, Found: 173.0938.



5a

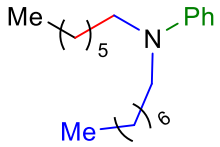
N-heptyl-3-methyl-N-phenylbenzamide (**5a**). Eluent: PE/EA = 20:1, colorless oil (27.0 mg, 87%). ^1H NMR (400 MHz, $CDCl_3$) δ

7.25 – 7.09 (m, 4H), 7.06 – 6.95 (m, 5H), 3.95 – 3.84 (m, 2H), 2.21 (s, 3H), 1.67 – 1.57 (m, 2H), 1.37 – 1.19 (m, 8H), 0.89 – 0.83 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 143.7, 137.55, 136.5, 130.2, 129.5, 129.1, 127.9, 127.5, 126.6, 125.75, 50.6, 31.9, 29.2, 27.85, 27.0, 22.7, 21.3, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{28}NO^+$ 310.2165, Found: 310.2167.



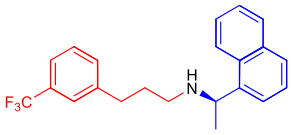
5b

N-heptyl-4-methyl-N-phenylbenzenesulfonamide (**5b**). Eluent: PE/EA = 50:1, white solid (24.8 mg, 72%, m.p. 74 – 75 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.43 (m, 2H), 7.33 – 7.27 (m, 3H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.07 – 7.00 (m, 2H), 3.51 (t, *J* = 7.0 Hz, 2H), 2.42 (s, 3H), 1.44 – 1.34 (m, 2H), 1.33 – 1.14 (m, 8H), 0.85 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.3, 139.3, 135.5, 129.4, 129.0, 128.9, 127.9, 127.8, 50.6, 31.8, 28.9, 28.3, 26.5, 22.7, 21.7, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{28}NO_2S^+$ 346.1835, Found: 346.1836.



5c

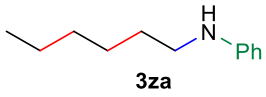
N-heptyl-N-octylaniline (**5c**). Eluent: PE/EA = 100:1, colorless oil (26.0 mg, 85%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.18 (m, 2H), 6.70 – 6.60 (m, 3H), 3.31 – 3.22 (m, 4H), 1.65 – 1.56 (m, 4H), 1.40 – 1.25 (m, 18H), 0.92 (t, *J* = 6.6 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.3, 129.3, 115.2, 111.8, 51.2, 32.1, 32.0, 29.7, 29.5, 29.4, 27.41, 27.39, 27.36, 27.33, 22.82, 22.79, 14.25. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{38}N^+$ 304.2999, Found: 304.2999.



3xr Cinacalcet

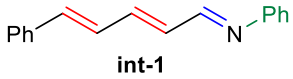
(R)-N-(1-(naphthalen-1-yl)ethyl)-3-(3-(trifluoromethyl)phenyl)propan-1-amine (**3xr**). Eluent: PE/EA = 4:1, yellow oil (20.1 mg, 56%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 8.1 Hz, 1H), 7.91 -7,87 (m, 1H), 7.79 -7.74 (m, 1H), 7.68 -7.64 (m, 1H), 7.55 – 7.47 (m, 3H), 7.45 – 7.41 (m, 2H), 7.38 – 7.29 (m, 2H), 4.64 (q, *J* = 6.6 Hz, 1H), 2.78 – 2.56 (m, 4H), 1.90 -1.81 (m, 2H), 1.51 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.15, 141.2, 134.1, 131.9 (d, *J*

= 1.0 Hz, 1C), 131.4, 130.6 (q, *J* = 31.8 Hz, 1C), 129.1, 128.8, 127.3, 125.9, 125.8, 125.5, 125.2 (q, *J* = 3.7 Hz, 1C), 124.4 (q, *J* = 270.5 Hz, 1C),123.0, 122.8 (q, *J* = 3.7 Hz, 1C), 122.7, 53.8, 47.4, 33.5, 32.0, 23.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.47. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{23}F_3N^+$ 358.1777, Found: 358.1773.



3za

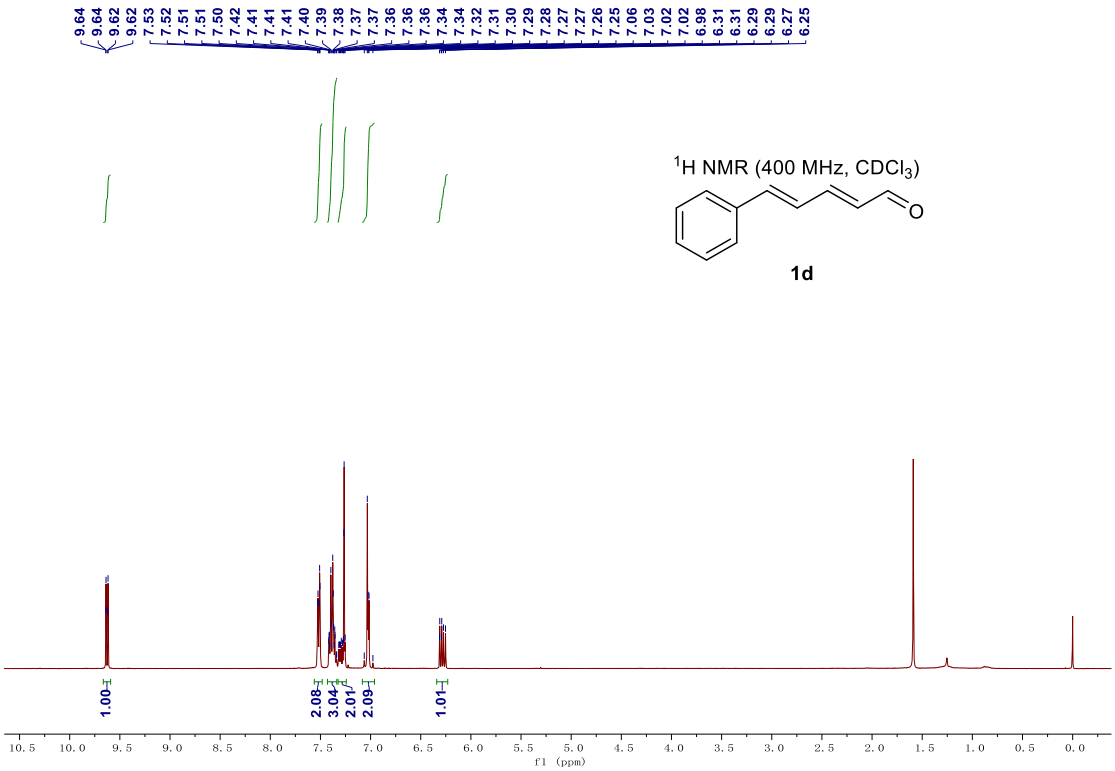
N-hexylaniline (**3za**). Eluent: PE/EA = 100:1, light yellow oil (6.6 mg, 37%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (t, *J* = 7.9 Hz, 2H), 6.69 (t, *J* = 7.3 Hz, 1H), 6.61 (d, *J* = 7.9 Hz, 2H), 3.70 (s, 1H), 3.10 (t, *J* = 7.2 Hz, 2H), 1.67 – 1.57 (m, 3H), 1.36 – 1.27 (m, 5H), 0.95 – 0.86 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 129.4, 117.3, 112.9, 44.2, 31.8, 29.7, 27.0, 22.8, 14.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{12}H_{20}N^+$ 178.1590, Found: 178.1587.

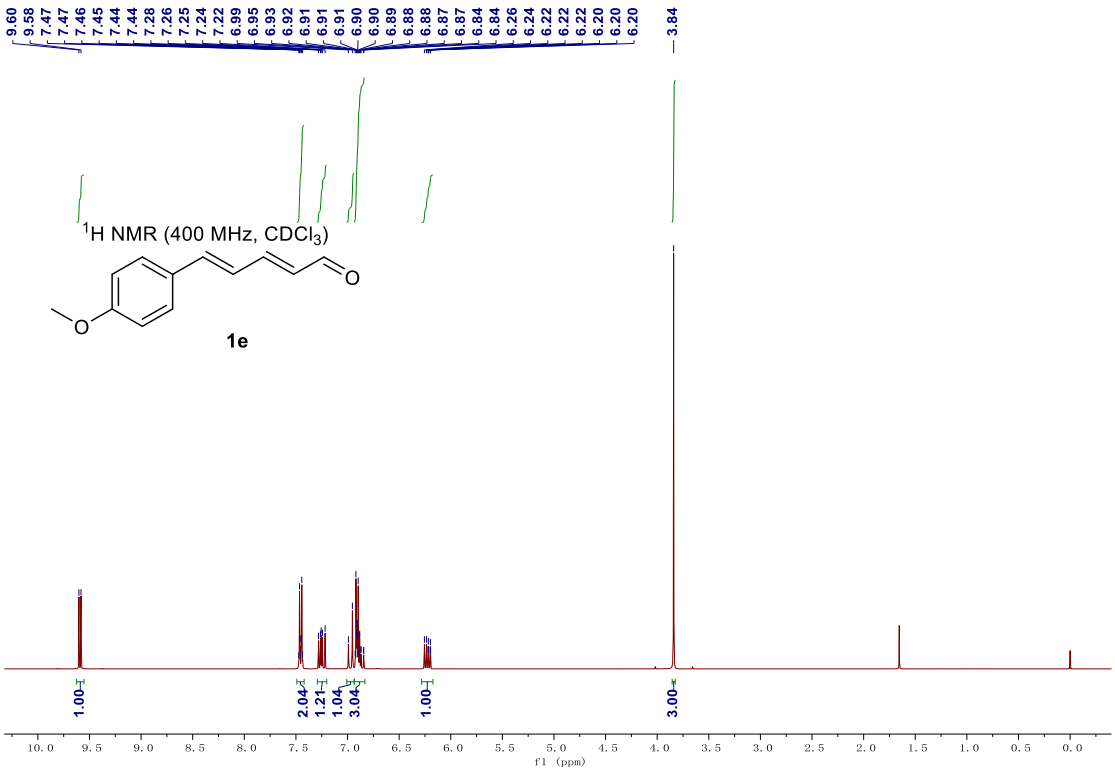


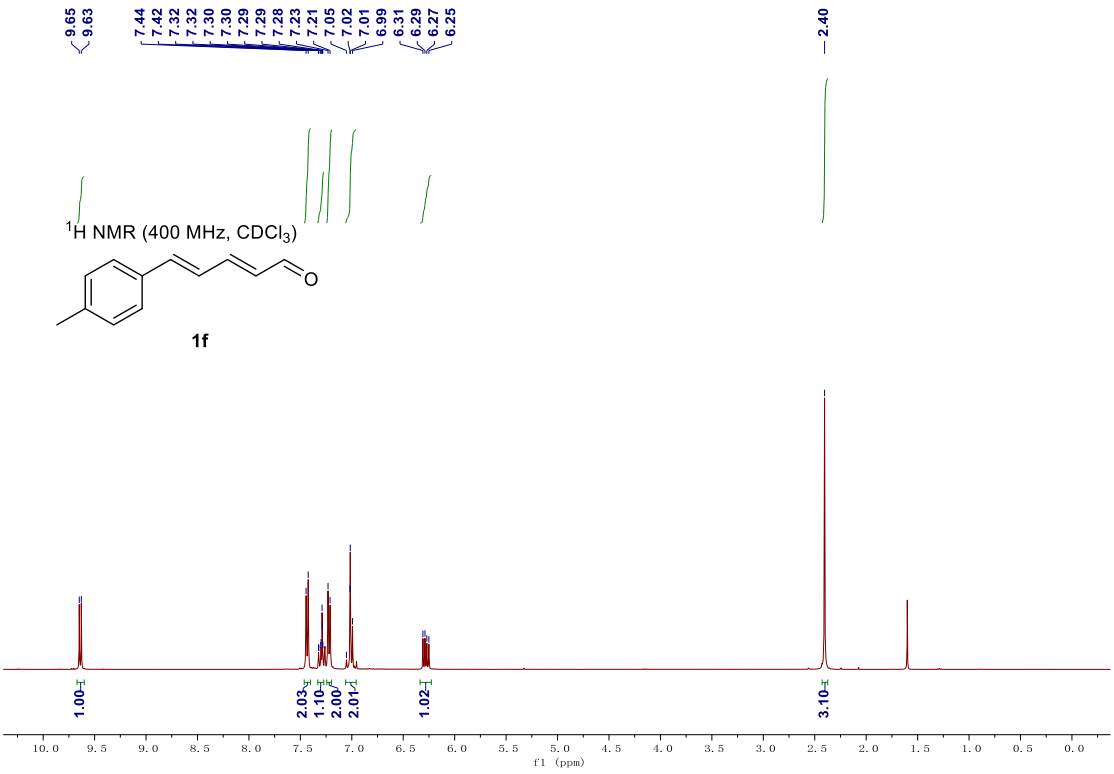
int-1

(1E,2E,4E)-N,5-diphenylpenta-2,4-dien-1-imine(**int-1**). Yellow solid (420 mg, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 9.2 Hz, 1H), 7.53 – 7.46 (m, 2H), 7.41 – 7.34 (m, 4H), 7.33 – 7.27 (m, 1H), 7.24 – 7.19 (m, 1H), 7.19 – 7.14 (m, 2H), 7.05 – 6.93 (m, 2H), 6.88 – 6.79 (m, 1H), 6.72 – 6.61 (m, 1H). The analytical data are consistent with those reported in the literature.[14]

7. NMR Spectrum and HRMS Data



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7.23
7.21
7.05
7.02
7.01
6.99
6.31
6.29
6.27
6.25
2.40
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O
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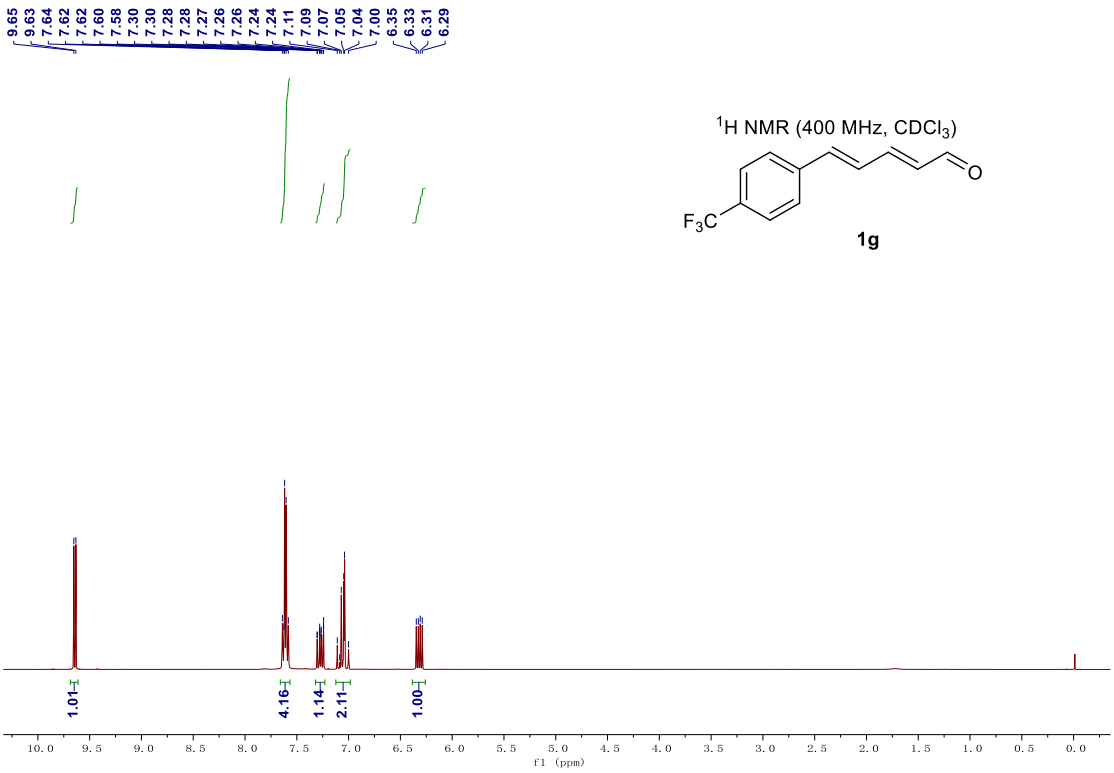
2.03
1.10
2.00
2.01
1.02
3.10
f1 (ppm)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7.24
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7.09
7.07
7.05
7.04
7.00
6.35
6.33
6.31
6.29
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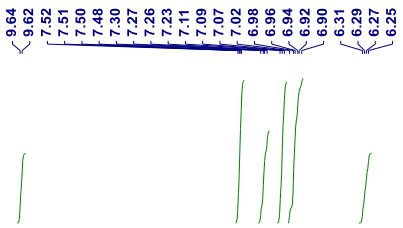
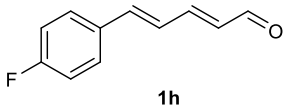
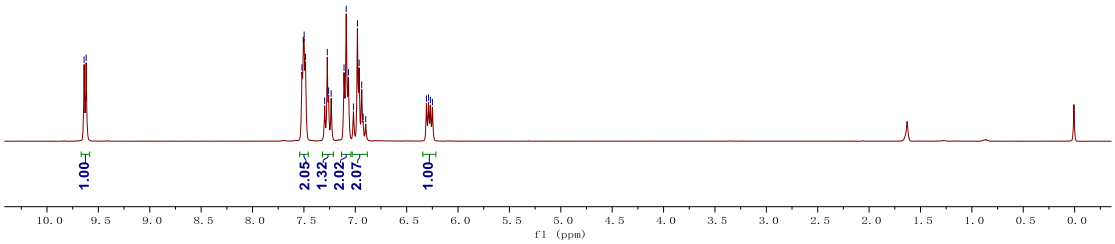
1g
1.01
4.16
1.14
2.11
1.00
f1 (ppm)

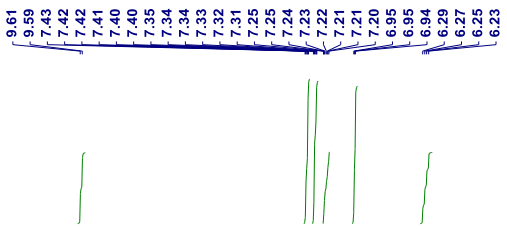


^{1}H NMR (400 MHz, $CDCl_3$)

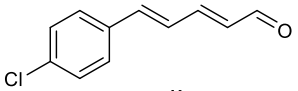


1h

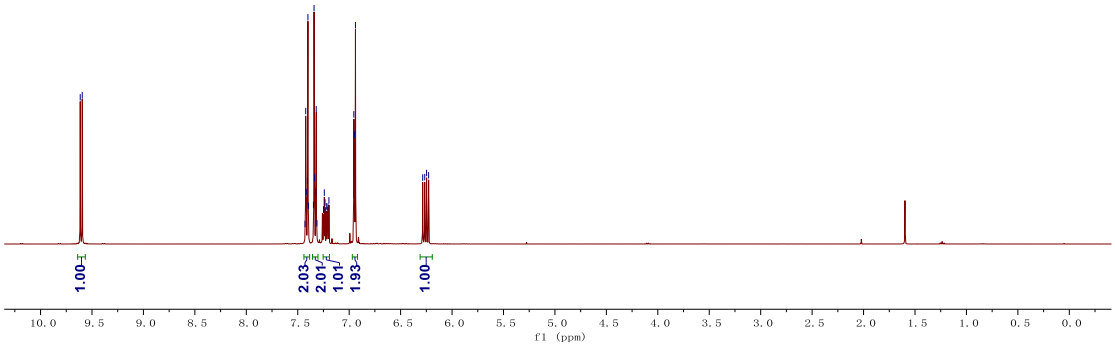


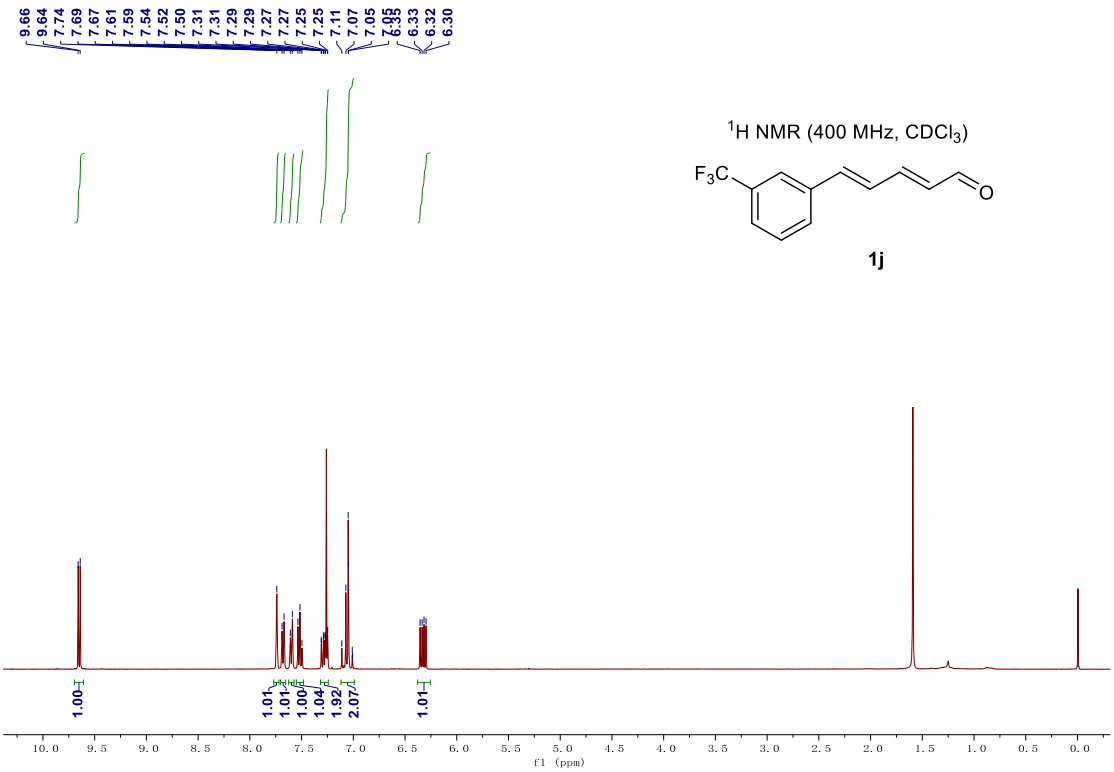


^{1}H NMR (400 MHz, $CDCl_3$)



1i

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7.50
7.31
7.31
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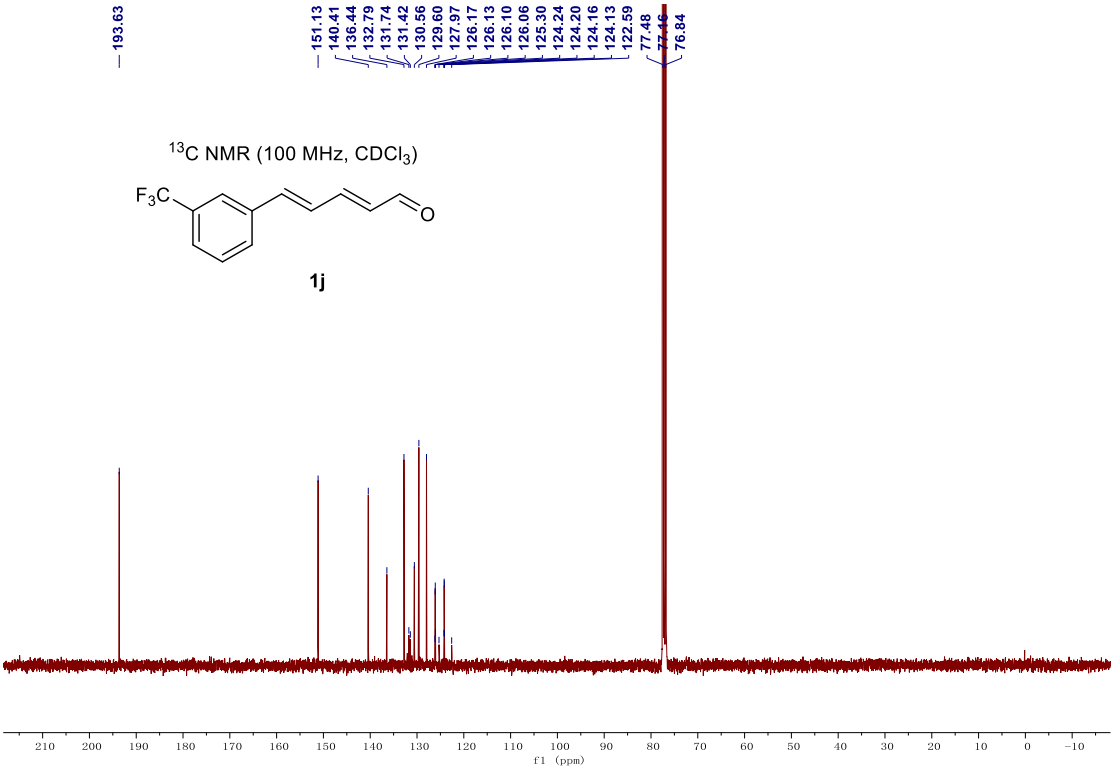
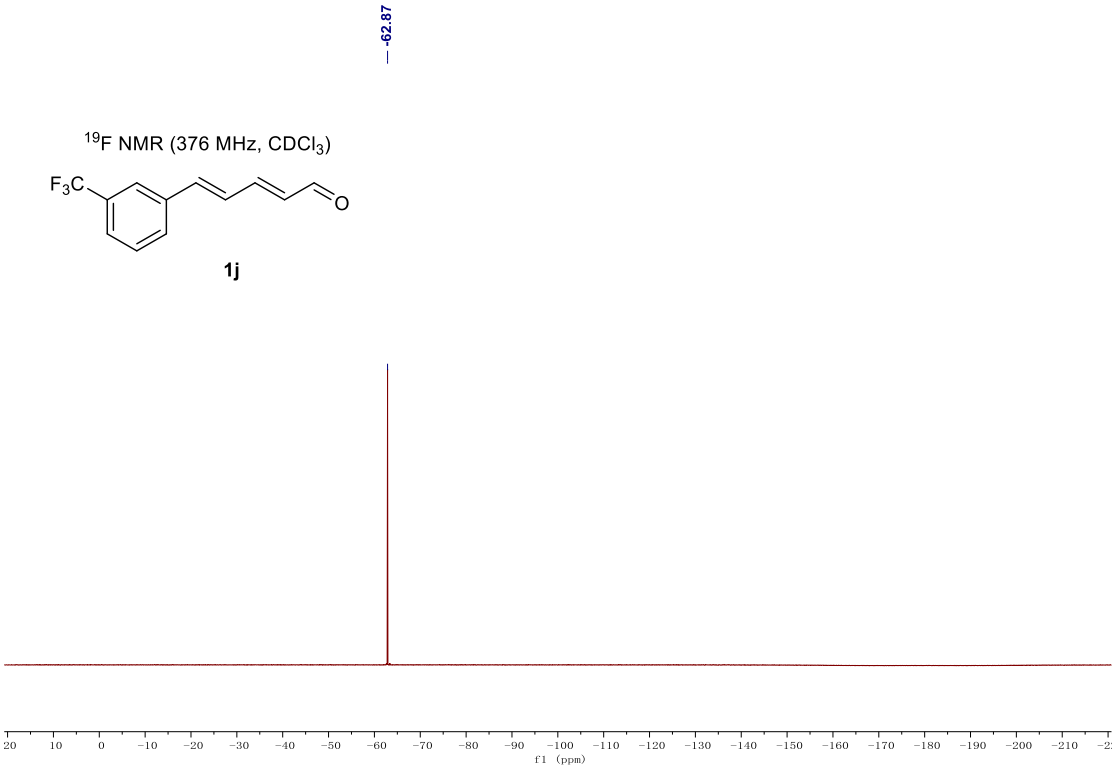
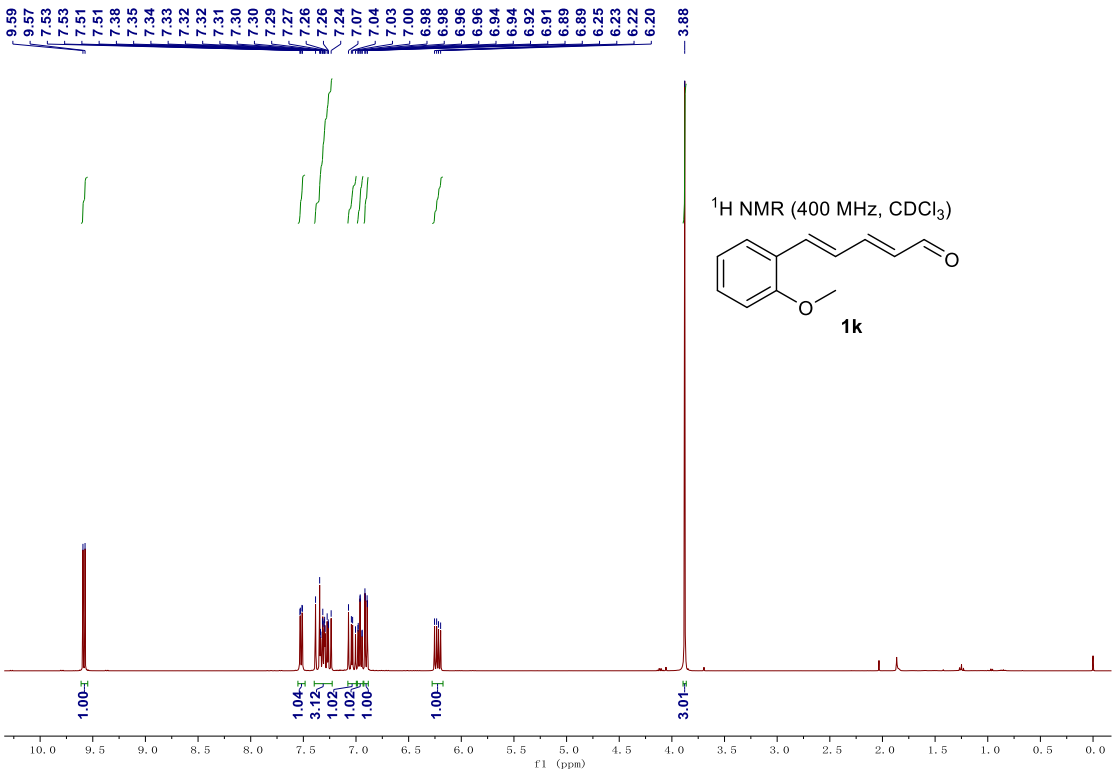
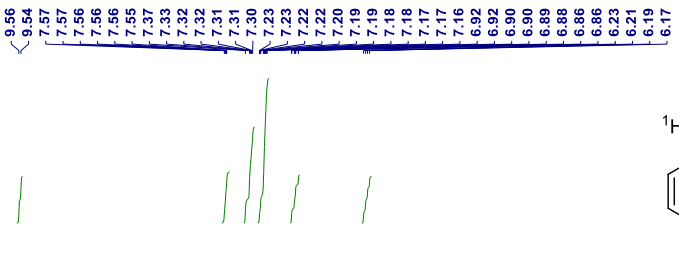
7.29
7.27
7.27
7.25
7.25
7.11
7.07
7.05
7.05
6.35
6.33
6.32
6.30
1H NMR (400 MHz, CDCl3)
F3C
O
1j
1.00
1.01
1.01
1.00
1.04
1.92
2.07
1.01
f1 (ppm)

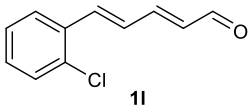

193.63
151.13
140.41
136.44
132.79
131.74
131.42
130.56
129.60
127.97
126.17
126.13
126.10
126.06
125.30
124.24
124.20
124.16
124.13
122.59
77.48
77.16
76.84
13C NMR (100 MHz, CDCl3)
F3C
O
1j
f1 (ppm)


-62.87
19F NMR (376 MHz, CDCl3)
F3C
O
1j
f1 (ppm)

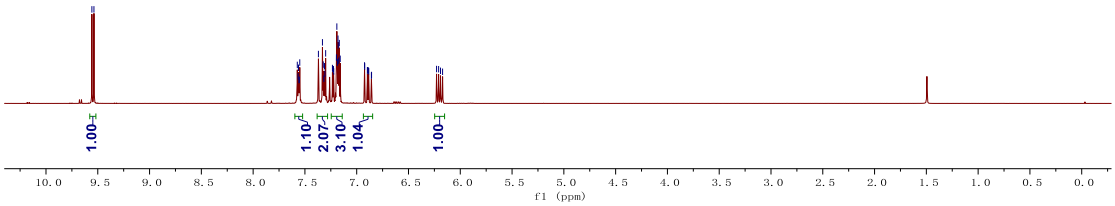

9.59
9.57
7.53
7.53
7.51
7.51
7.38
7.35
7.34
7.33
7.32
7.32
7.31
7.30
7.30
7.29
7.27
7.26
7.26
7.24
7.07
7.04
7.03
7.00
6.98
6.98
6.96
6.96
6.94
6.94
6.92
6.91
6.89
6.89
6.25
6.23
6.22
6.20
3.88
1H NMR (400 MHz, CDCl3)
O
O
1k
1.00
1.04
3.12
1.02
1.02
1.00
1.00
3.01
f1 (ppm)

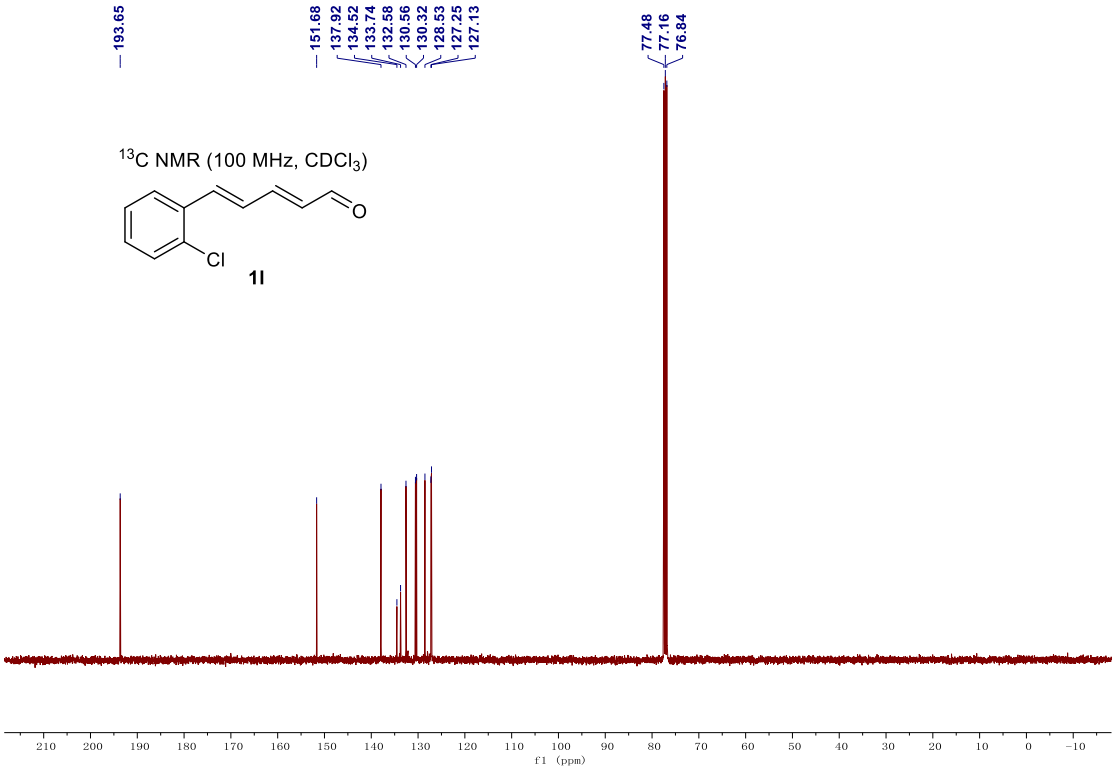


^{1}H NMR (400 MHz, $CDCl_3$)



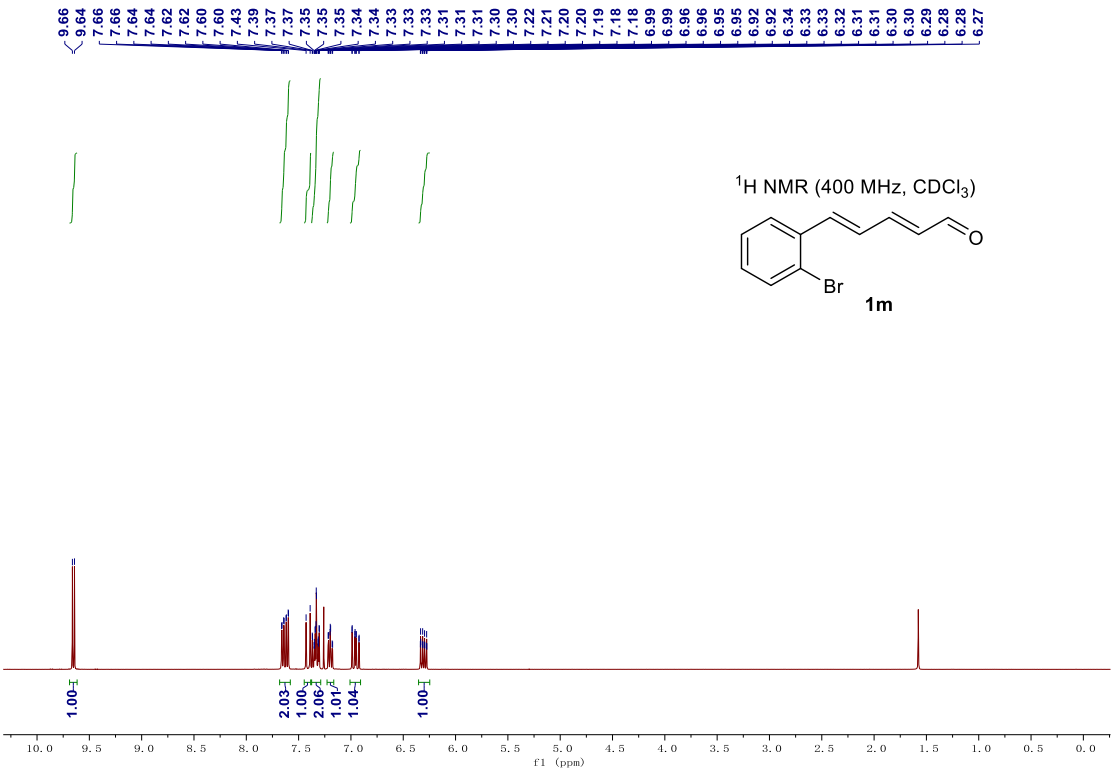
1l





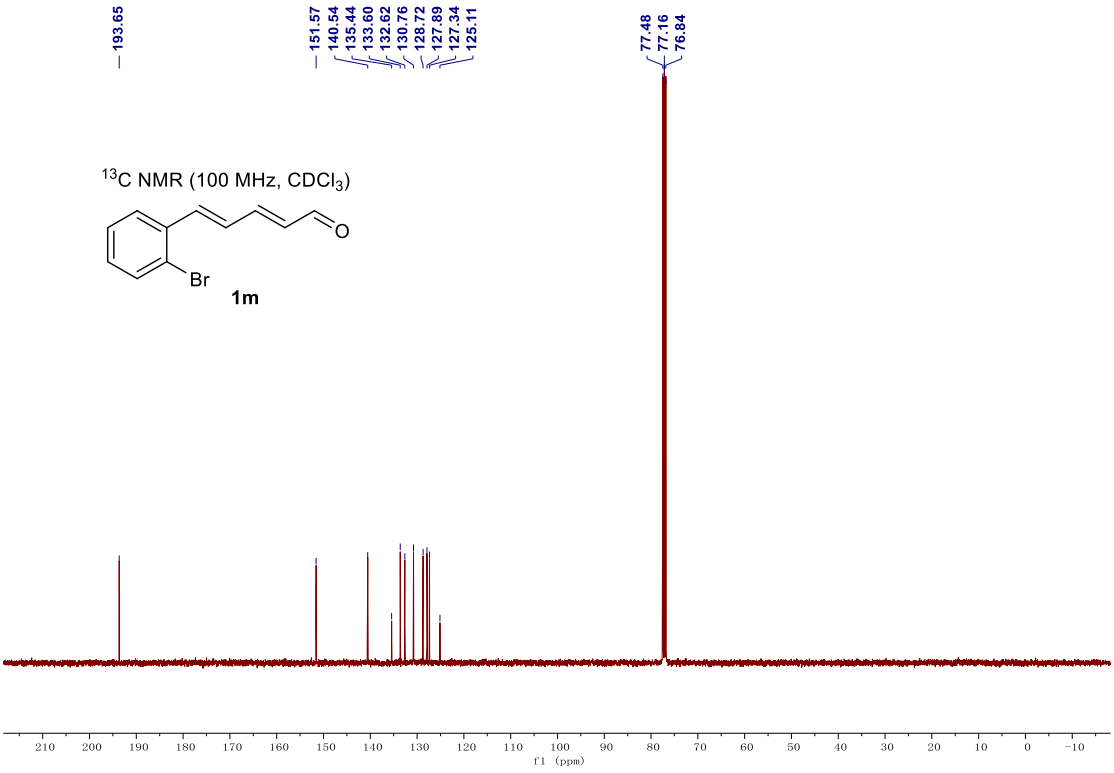
^{13}C NMR (100 MHz, $CDCl_3$)

1l



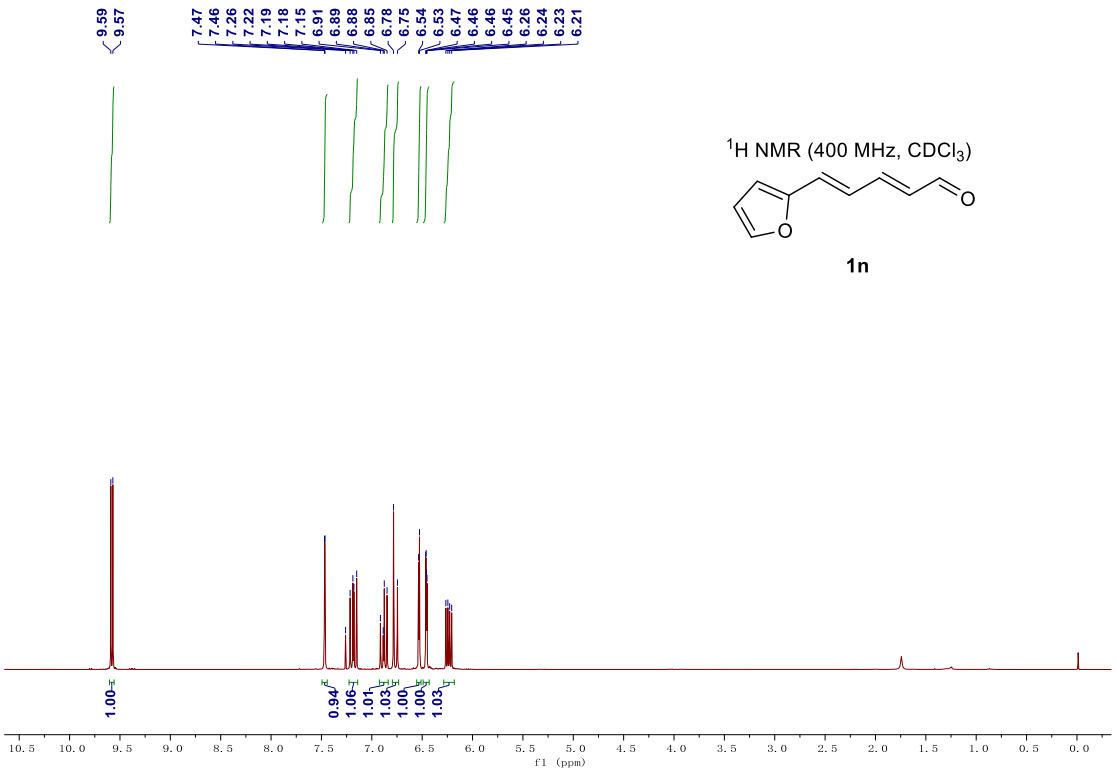
^{1}H NMR (400 MHz, $CDCl_3$)

1m



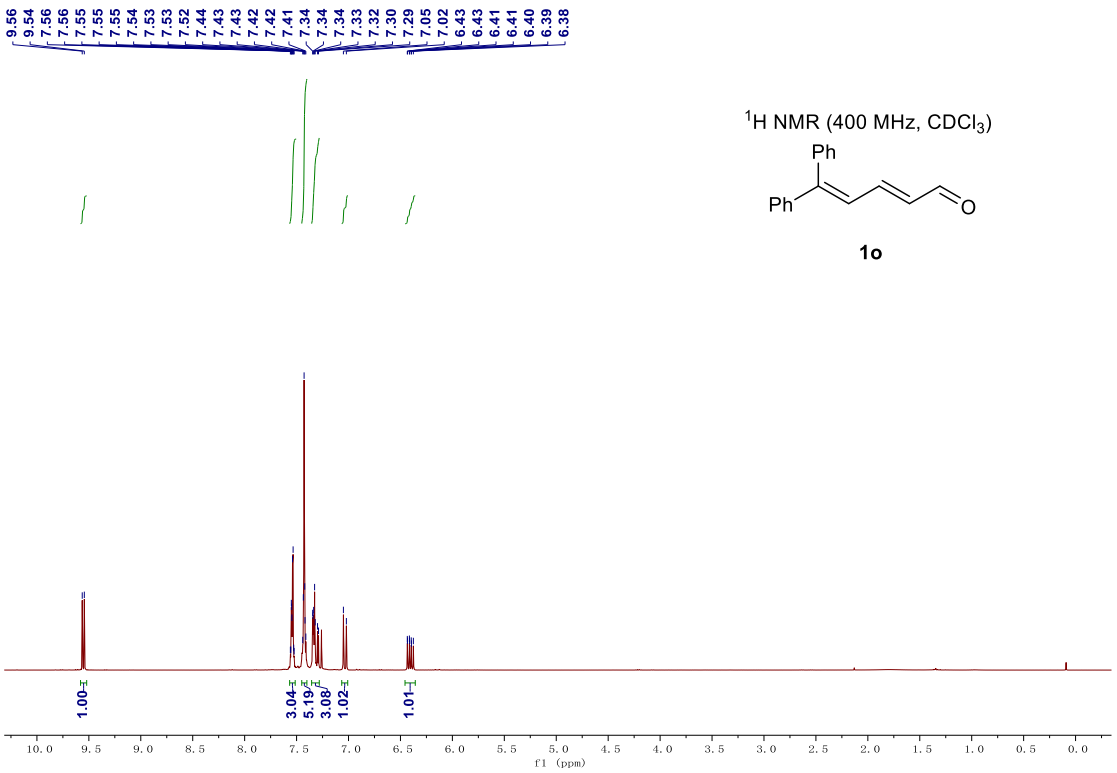
^{13}C NMR (100 MHz, $CDCl_3$)

1m



^{1}H NMR (400 MHz, $CDCl_3$)

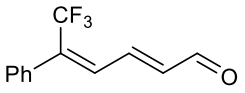
1n



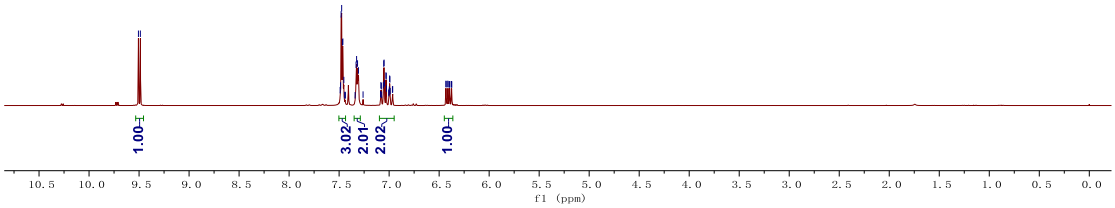
^{1}H NMR (400 MHz, $CDCl_3$)

1o

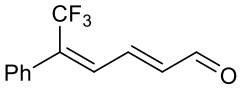
^{1}H NMR (400 MHz, $CDCl_3$)



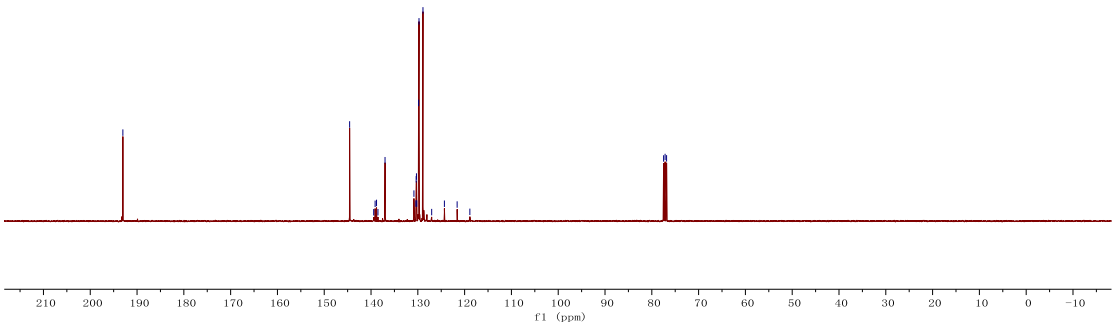
1p

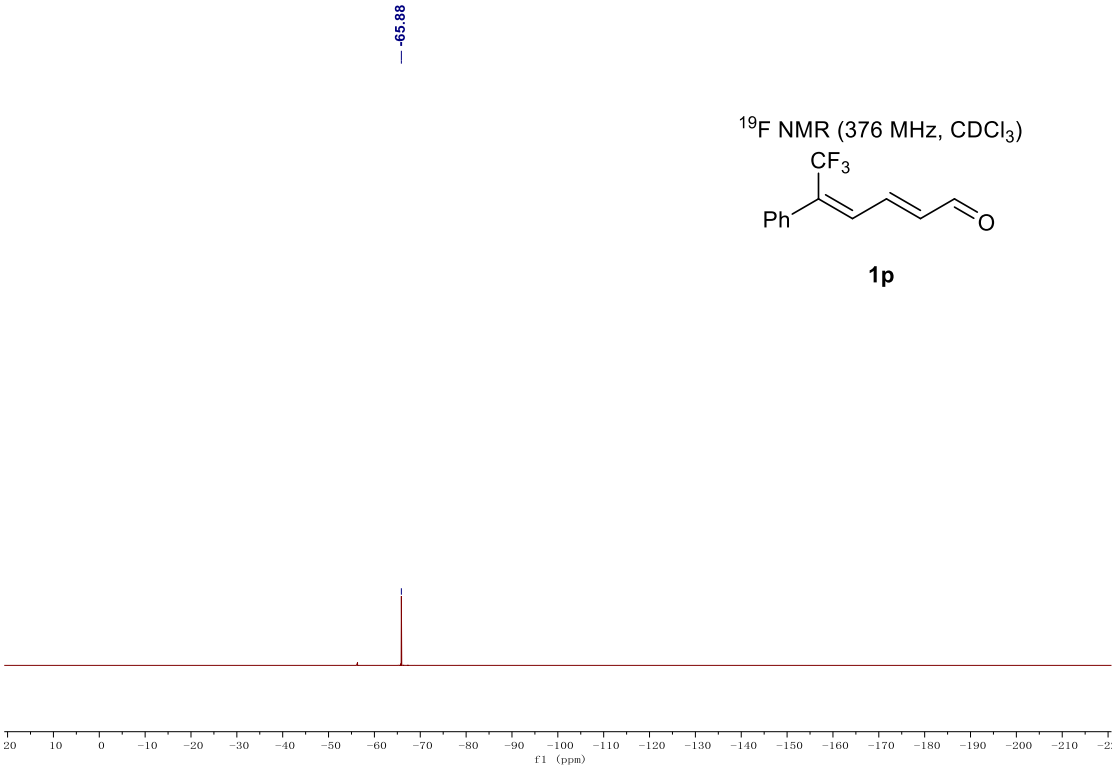


^{13}C NMR (100 MHz, $CDCl_3$)



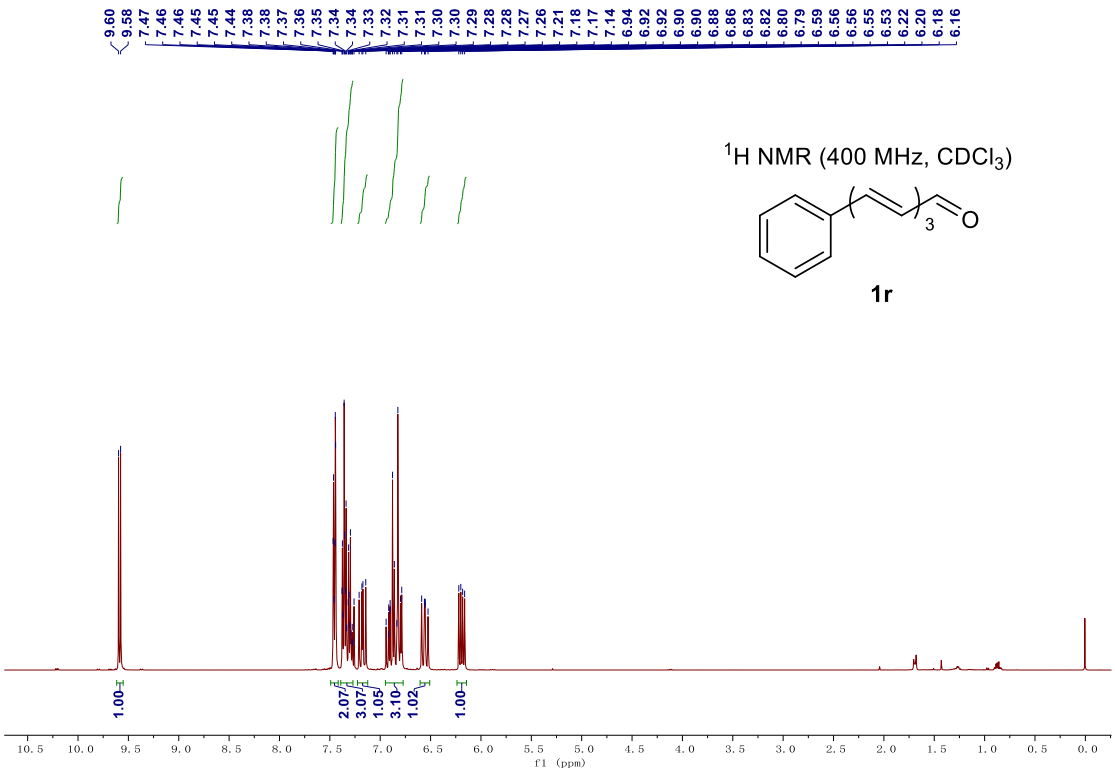
1p





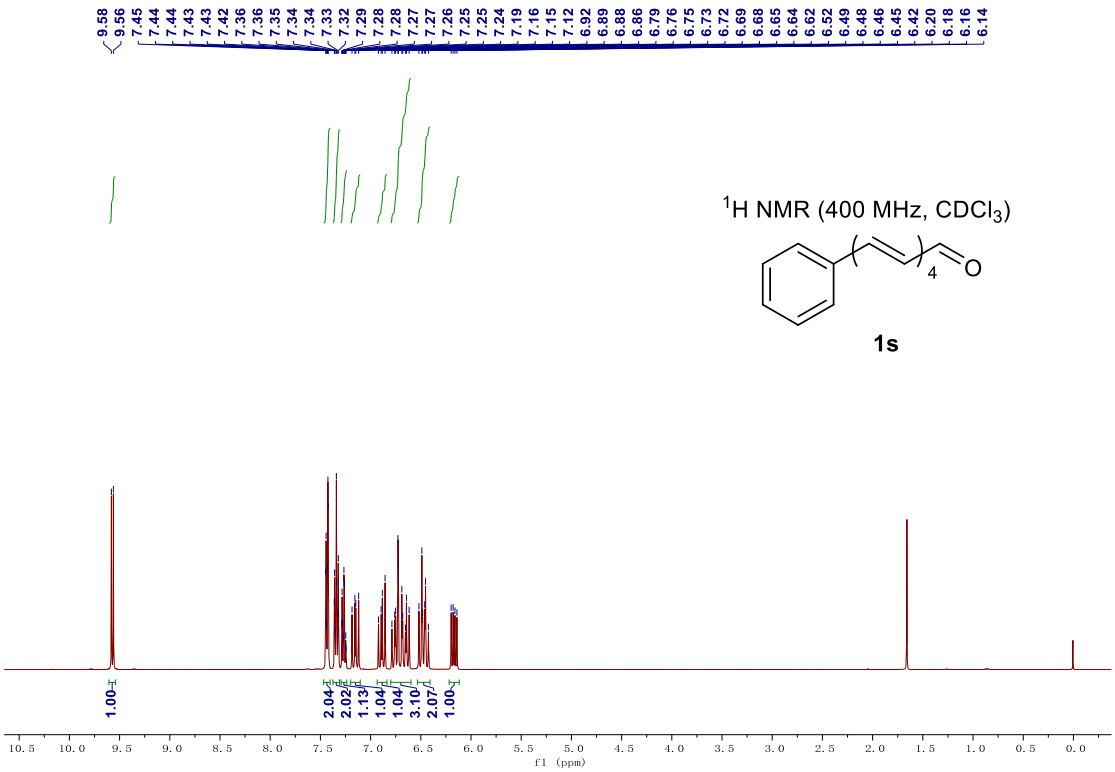
^{19}F NMR (376 MHz, $CDCl_3$)

1p



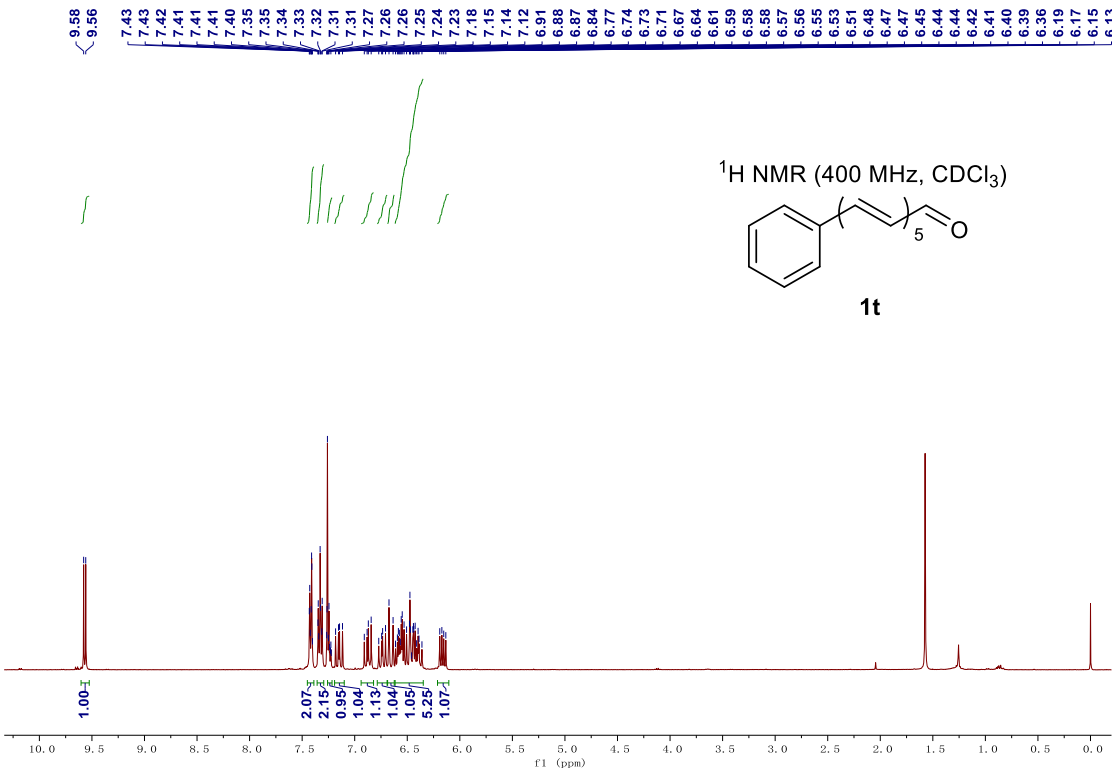
^{1}H NMR (400 MHz, $CDCl_3$)

1r



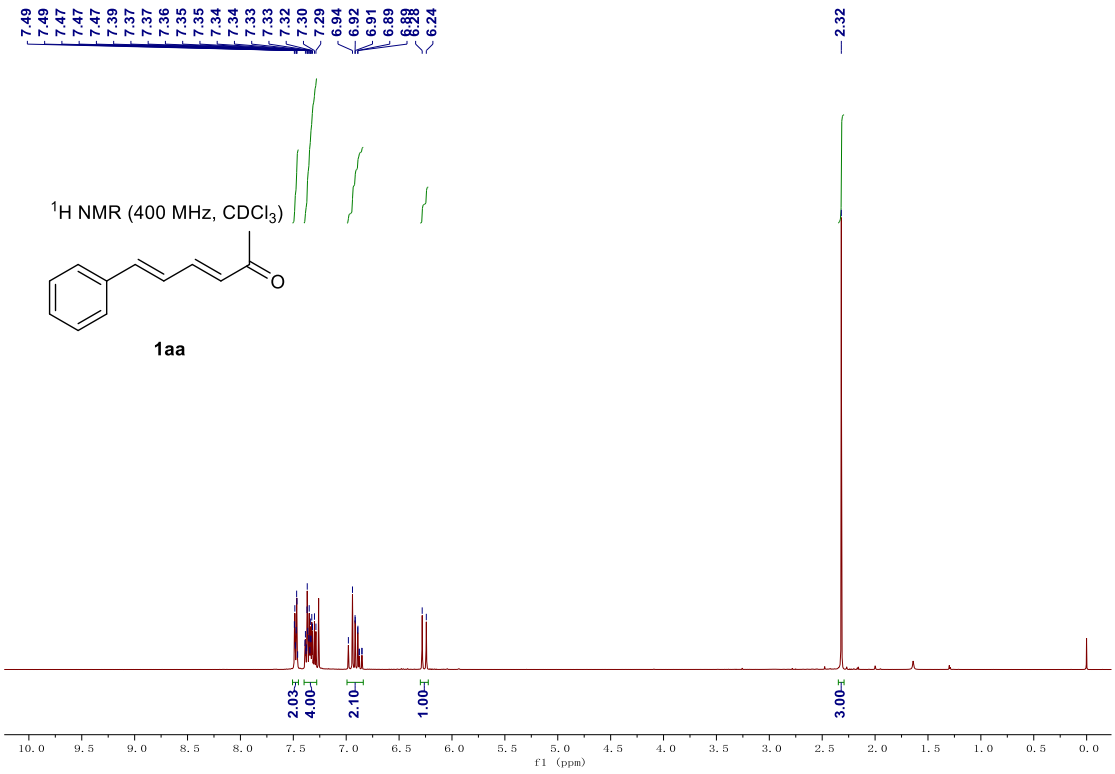
^{1}H NMR (400 MHz, $CDCl_3$)

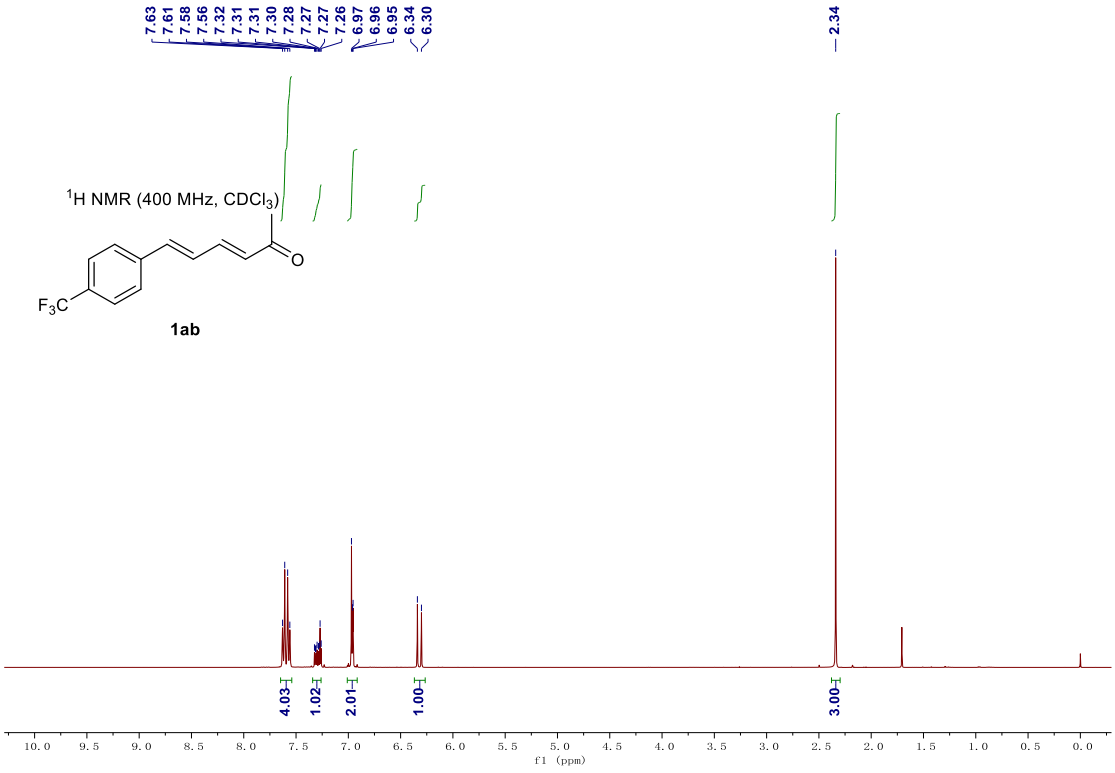
1s

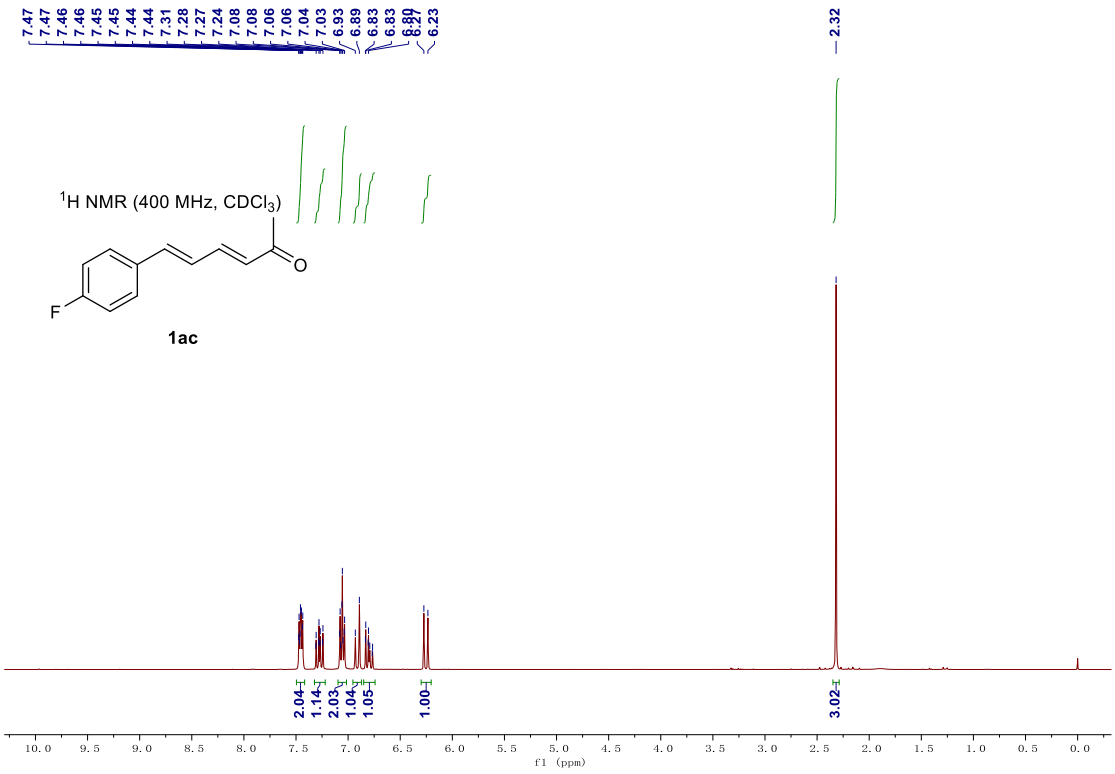


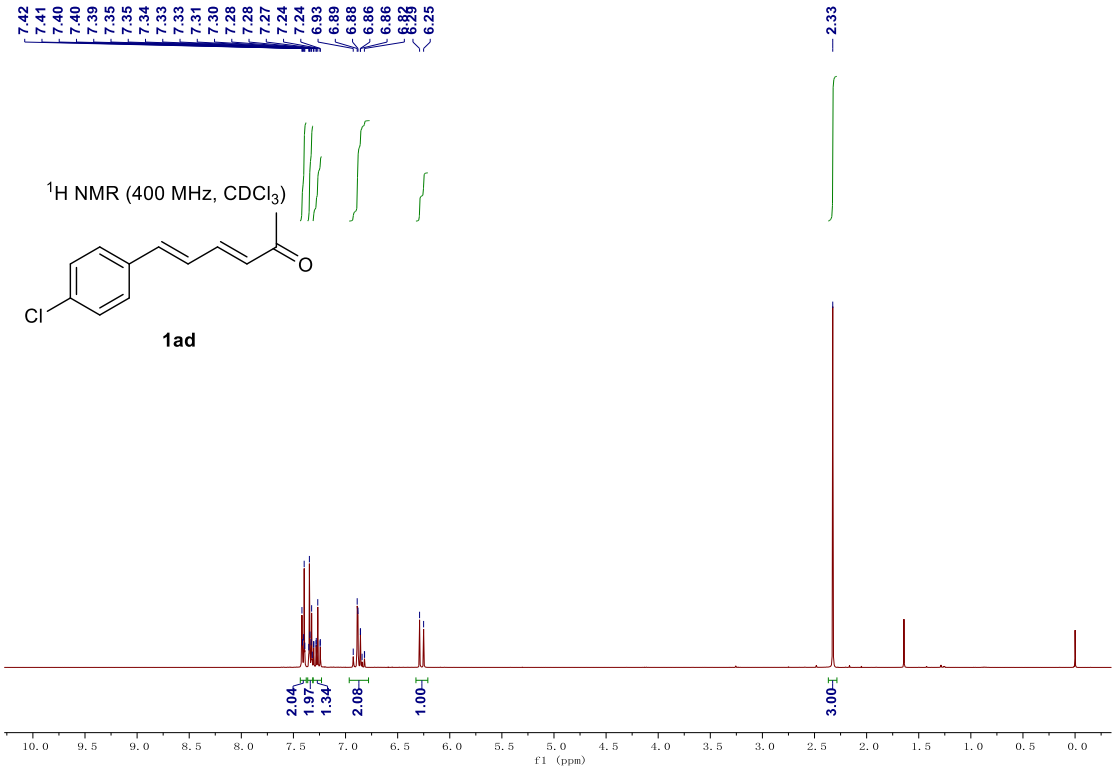
^{1}H NMR (400 MHz, $CDCl_3$)

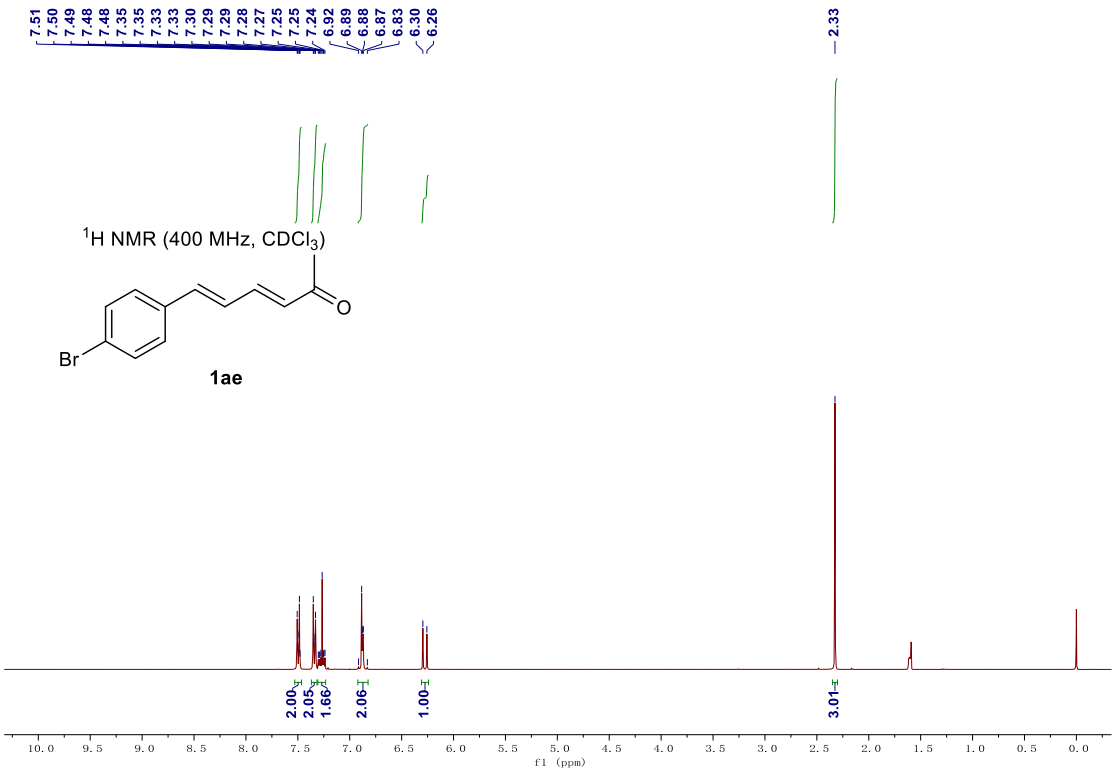
1t


7.49
7.49
7.47
7.47
7.47
7.39
7.37
7.37
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.30
7.29
6.94
6.92
6.91
6.89
6.24
2.32
^{1}H NMR (400 MHz, CDCl$_3$)
O
1aa
2.03
4.00
2.10
1.00
3.00
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
f1 (ppm)


7.63
7.61
7.58
7.56
7.32
7.31
7.31
7.30
7.28
7.27
7.27
7.26
6.97
6.96
6.95
6.34
6.30
2.34
^{1}H NMR (400 MHz, CDCl$_3$)
O
F$_3$C
1ab
4.03
1.02
2.01
1.00
3.00
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
f1 (ppm)


7.47
7.47
7.46
7.46
7.45
7.45
7.44
7.44
7.31
7.28
7.27
7.24
7.08
7.08
7.06
7.06
7.04
7.03
6.93
6.89
6.83
6.83
6.23
2.32
^{1}H NMR (400 MHz, CDCl$_3$)
F
O
1ac
2.04
1.14
2.03
1.04
1.05
1.00
3.02
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
f1 (ppm)


7.42
7.41
7.40
7.40
7.39
7.35
7.35
7.34
7.33
7.33
7.31
7.30
7.28
7.28
7.27
7.24
7.24
6.93
6.89
6.88
6.86
6.86
6.25
2.33
^{1}H NMR (400 MHz, CDCl$_3$)
Cl
O
1ad
2.04
1.97
1.34
2.08
1.00
3.00
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
f1 (ppm)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7.48
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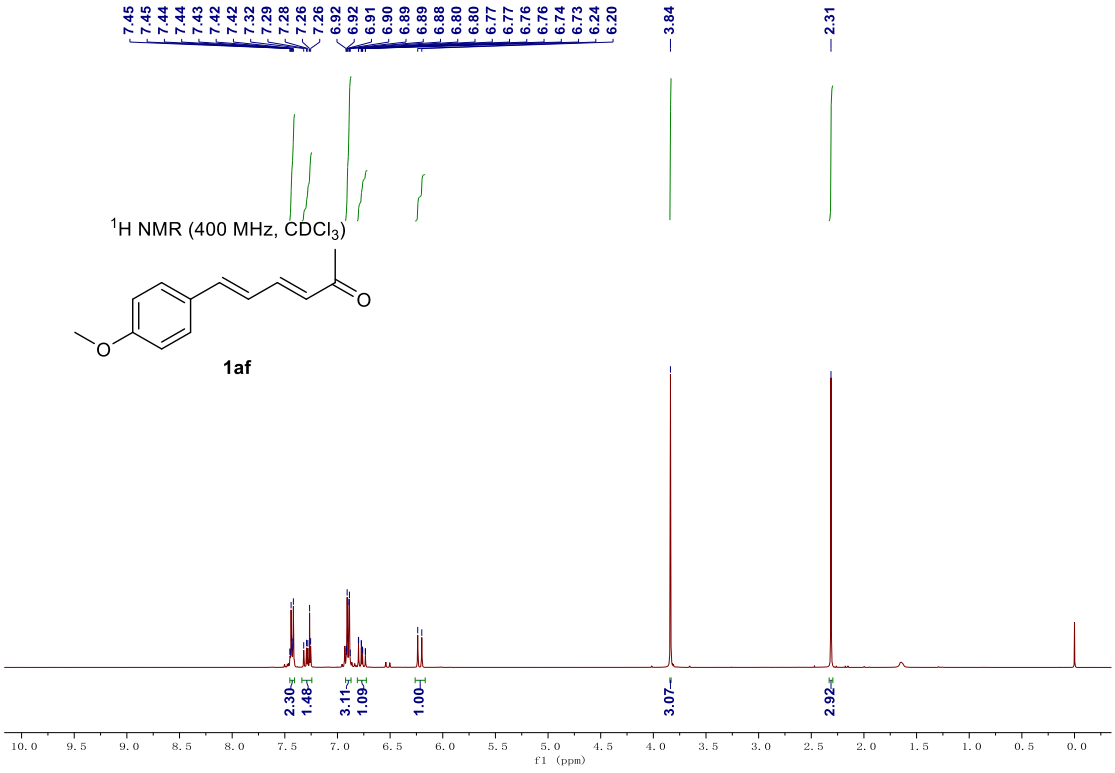
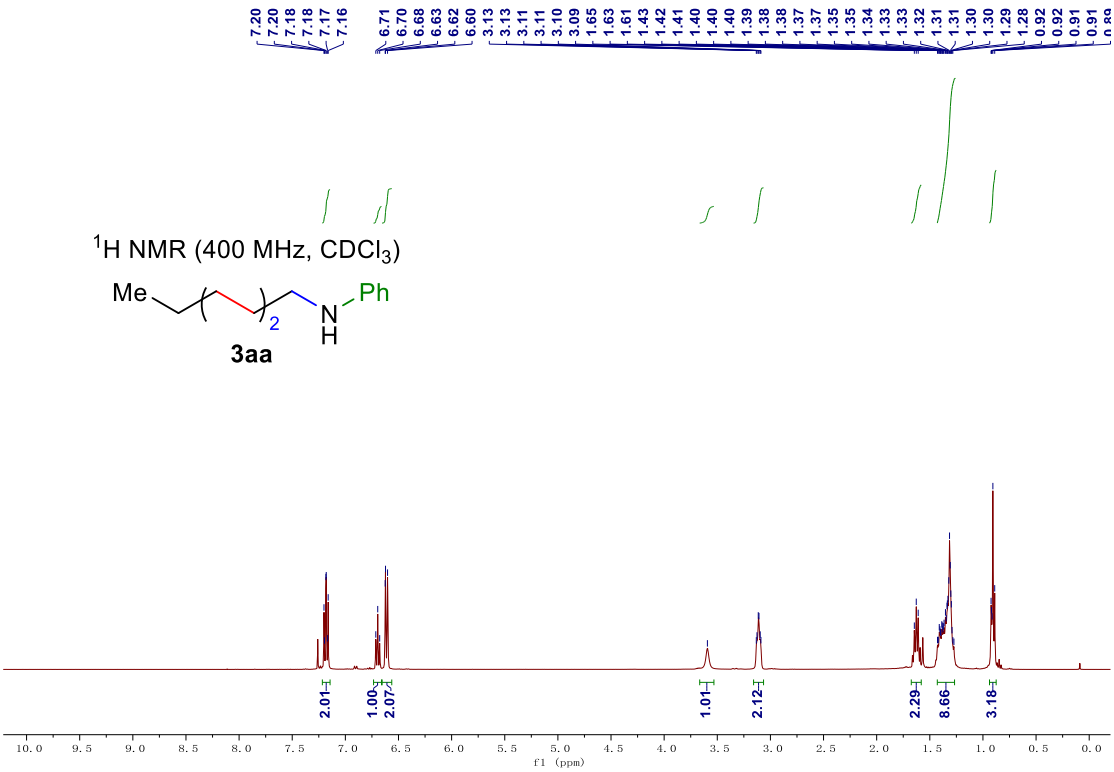
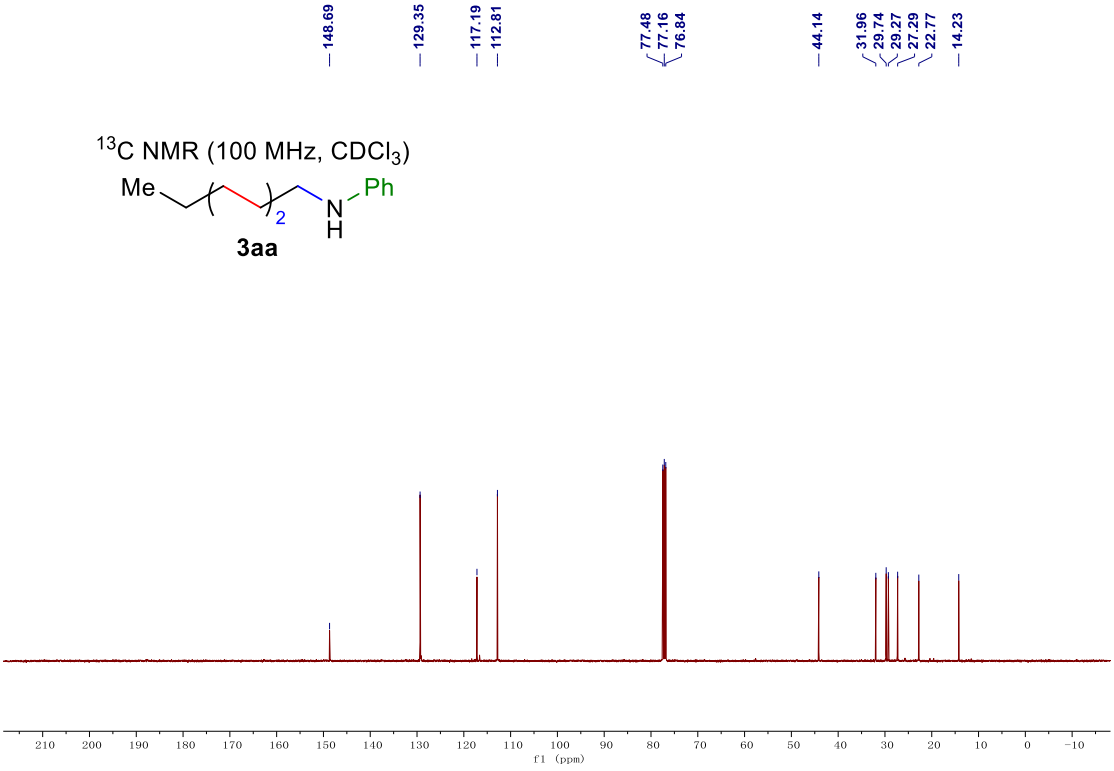
7.35
7.33
7.33
7.30
7.29
7.29
7.28
7.27
7.25
7.25
7.24
6.92
6.89
6.88
6.87
6.83
6.30
6.26
2.33
1H NMR (400 MHz, CDCl3)
Br
O
1ae
2.00
2.05
1.66
2.06
1.00
3.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


7.45
7.45
7.44
7.44
7.43
7.42
7.42
7.32
7.29
7.28
7.26
7.26
6.92
6.91
6.90
6.89
6.89
6.88
6.80
6.80
6.77
6.77
6.76
6.76
6.74
6.73
6.24
6.20
3.84
2.31
1H NMR (400 MHz, CDCl3)
O
O
1af
2.30
1.48
3.11
1.09
1.00
3.07
2.92
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
f1 (ppm)



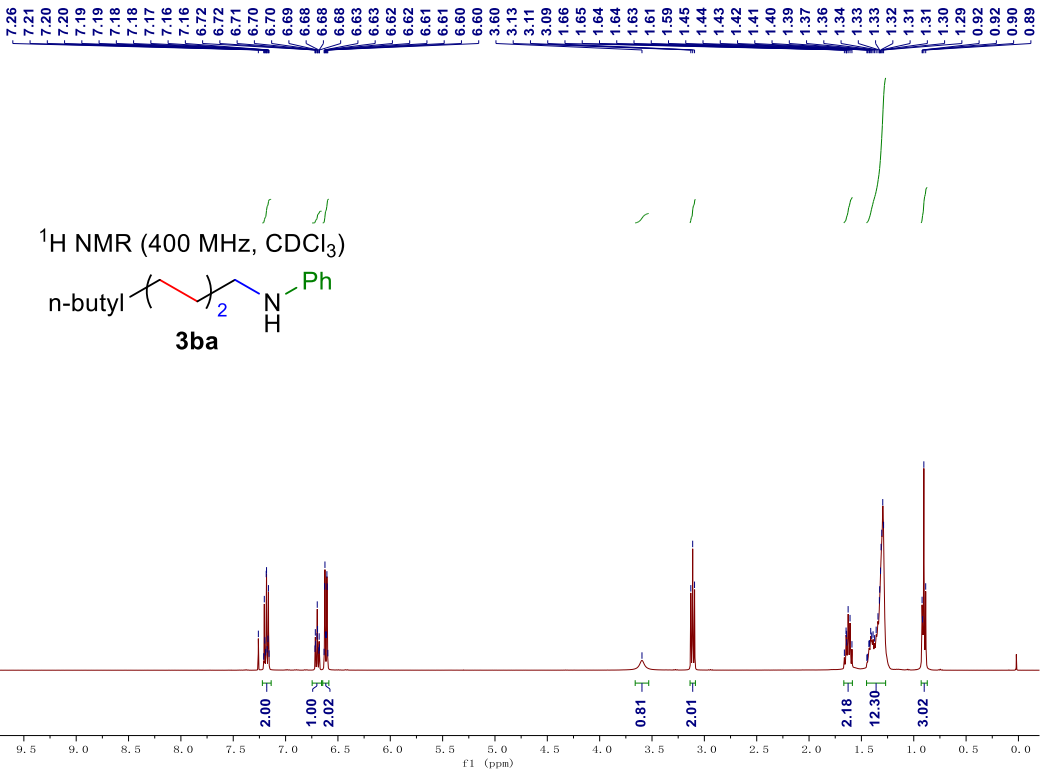
^{1}H NMR (400 MHz, $CDCl_3$)

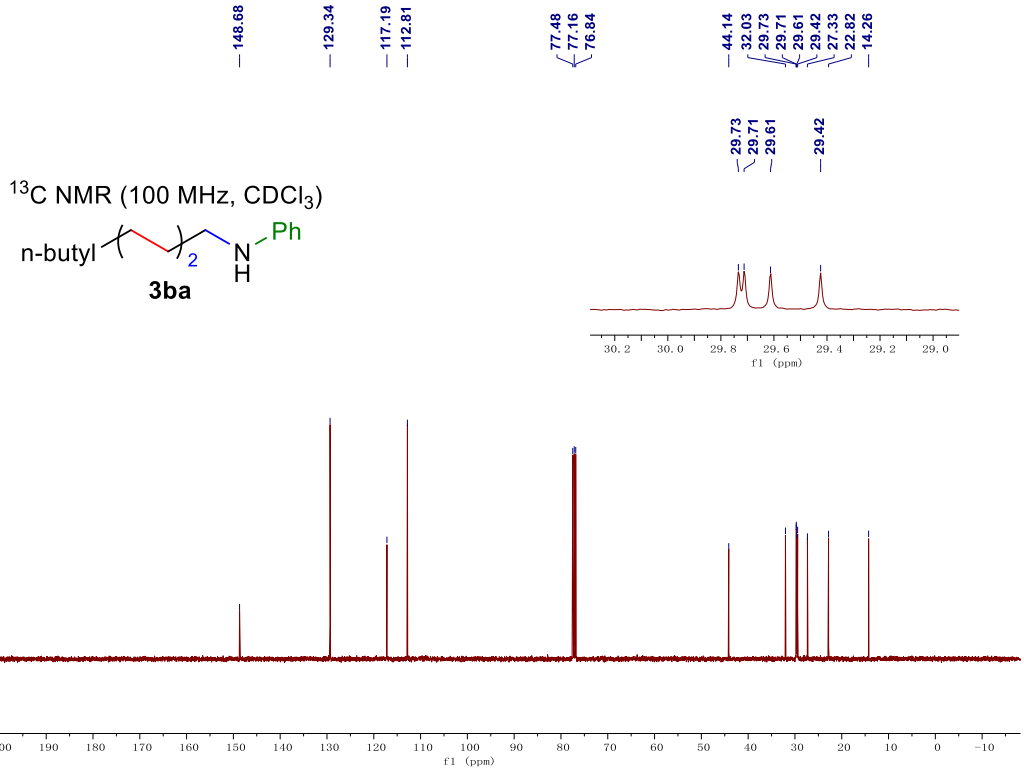
3aa



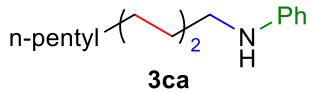
^{13}C NMR (100 MHz, $CDCl_3$)

3aa

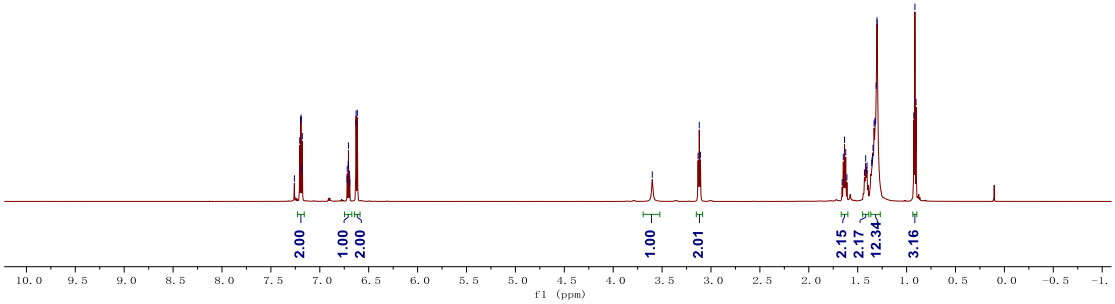

7.26 7.21 7.20 7.20 7.19 7.19 7.18 7.18 7.17 7.16 7.16 6.72 6.72 6.71 6.70 6.70 6.69 6.68 6.68 6.68 6.63 6.63 6.62 6.62 6.61 6.61 6.60 6.60
3.60 3.13 3.11 3.09 1.66 1.65 1.64 1.64 1.63 1.61 1.59 1.45 1.44 1.43 1.42 1.41 1.40 1.39 1.37 1.36 1.34 1.33 1.33 1.32 1.31 1.31 1.30 1.29 0.92 0.92 0.90 0.89
1H NMR (400 MHz, CDCl3)
n-butyl
2
N
H
Ph
3ba
2.00 1.00 2.02 0.81 2.01 2.18 12.30 3.02
f1 (ppm)

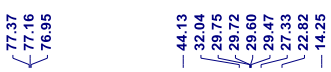

148.68 129.34 117.19 112.81 77.48 77.16 76.84 44.14 32.03 29.73 29.71 29.61 29.42 27.33 22.82 14.26
29.73 29.71 29.61 29.42
13C NMR (100 MHz, CDCl3)
n-butyl
2
N
H
Ph
3ba
f1 (ppm)

^{1}H NMR (600 MHz, $CDCl_3$)

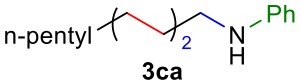


3ca

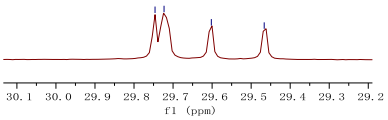


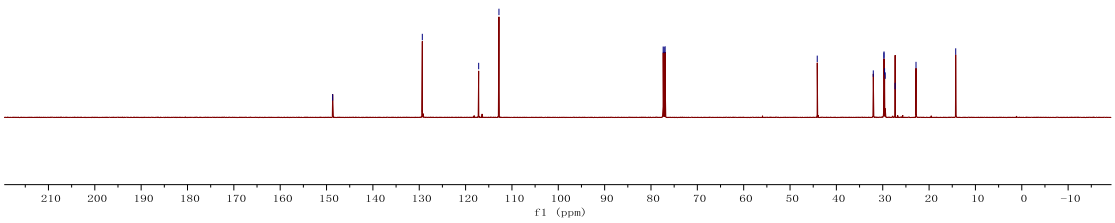


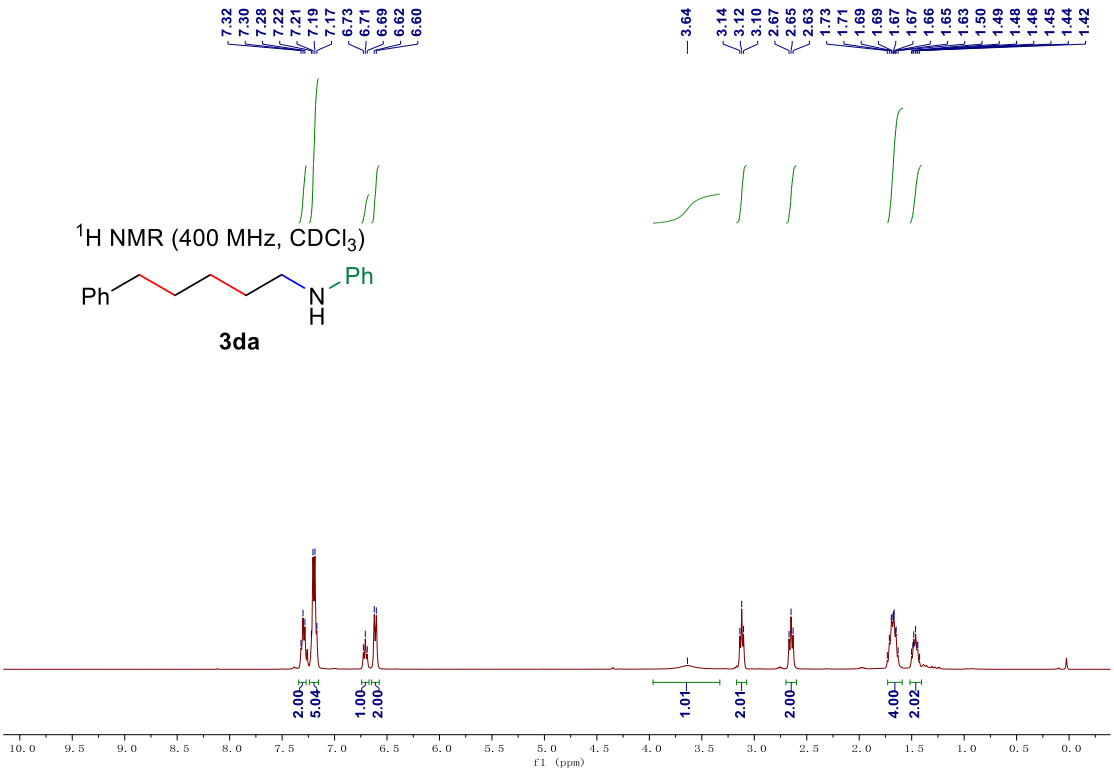
^{13}C NMR (150 MHz, $CDCl_3$)



3ca



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7.30
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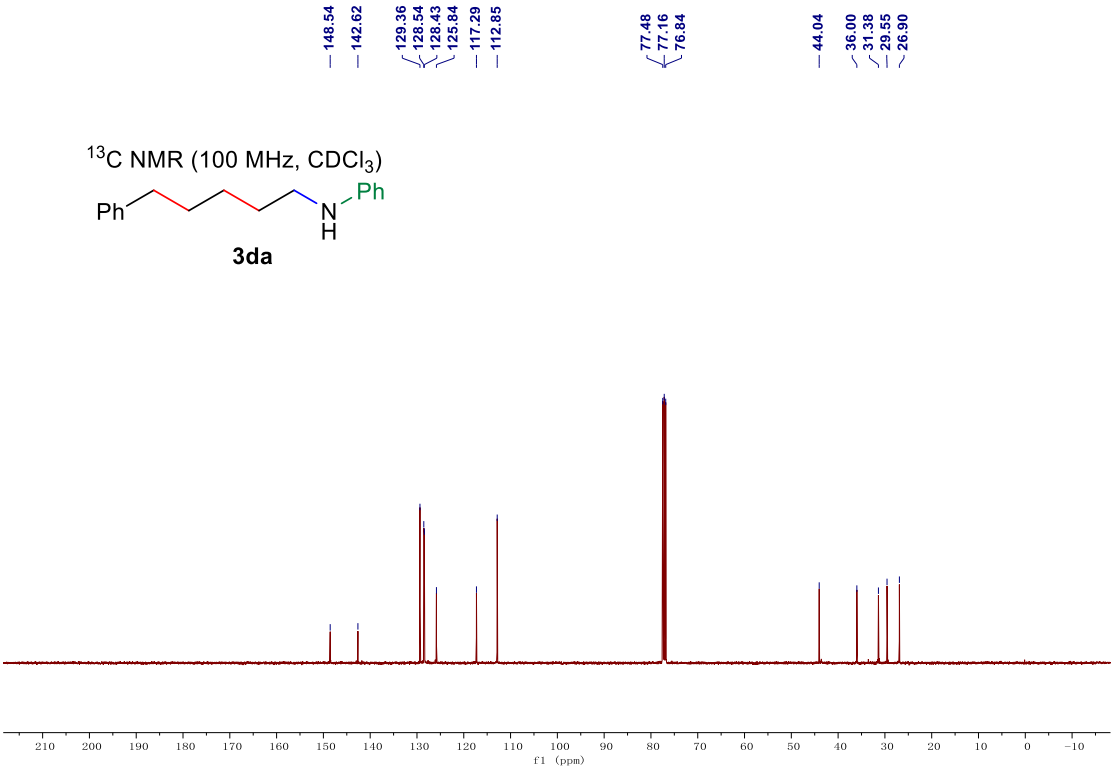
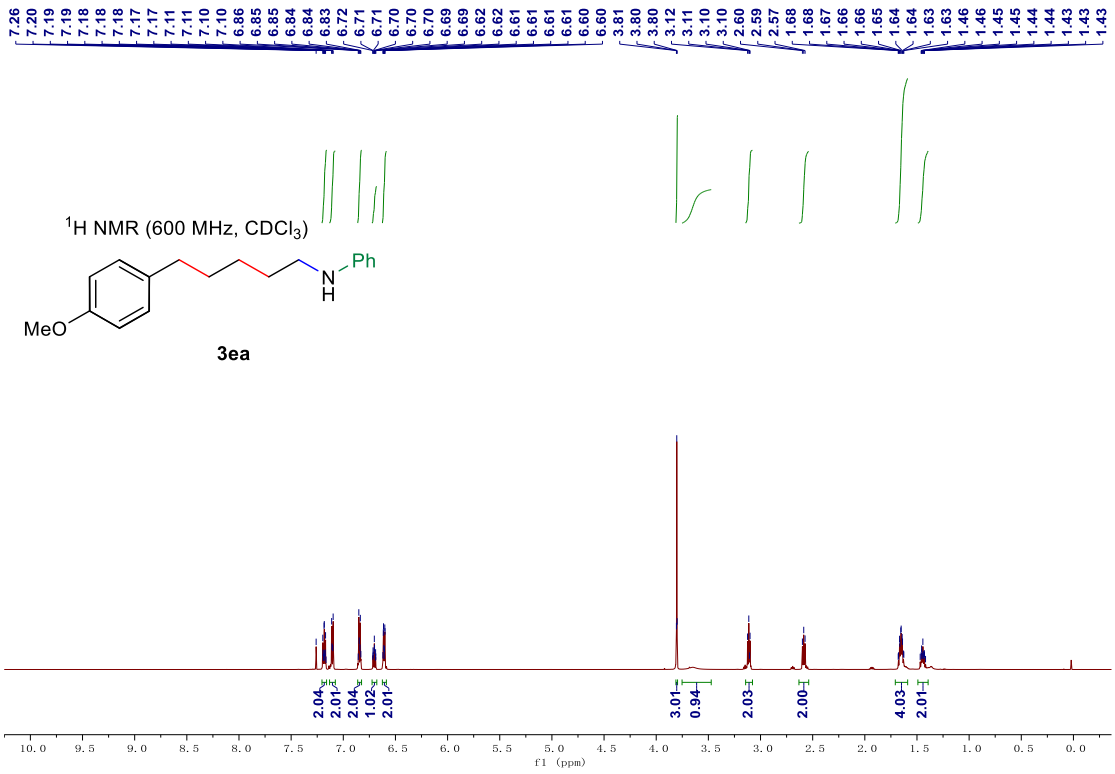
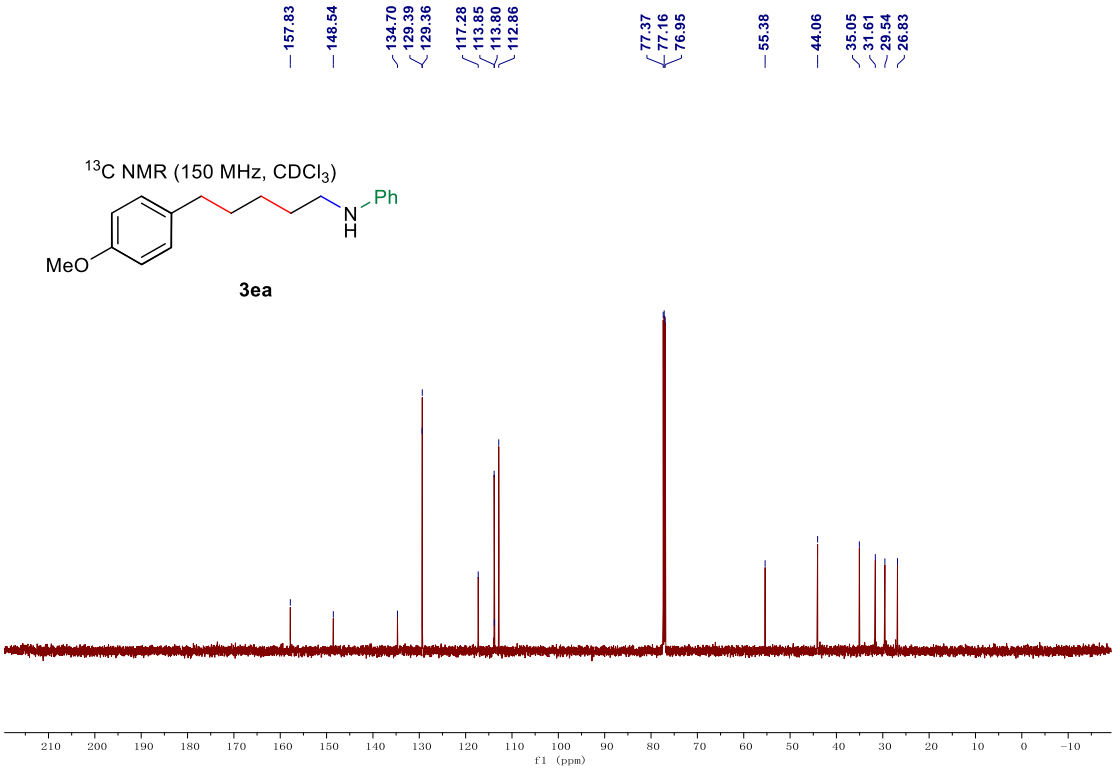
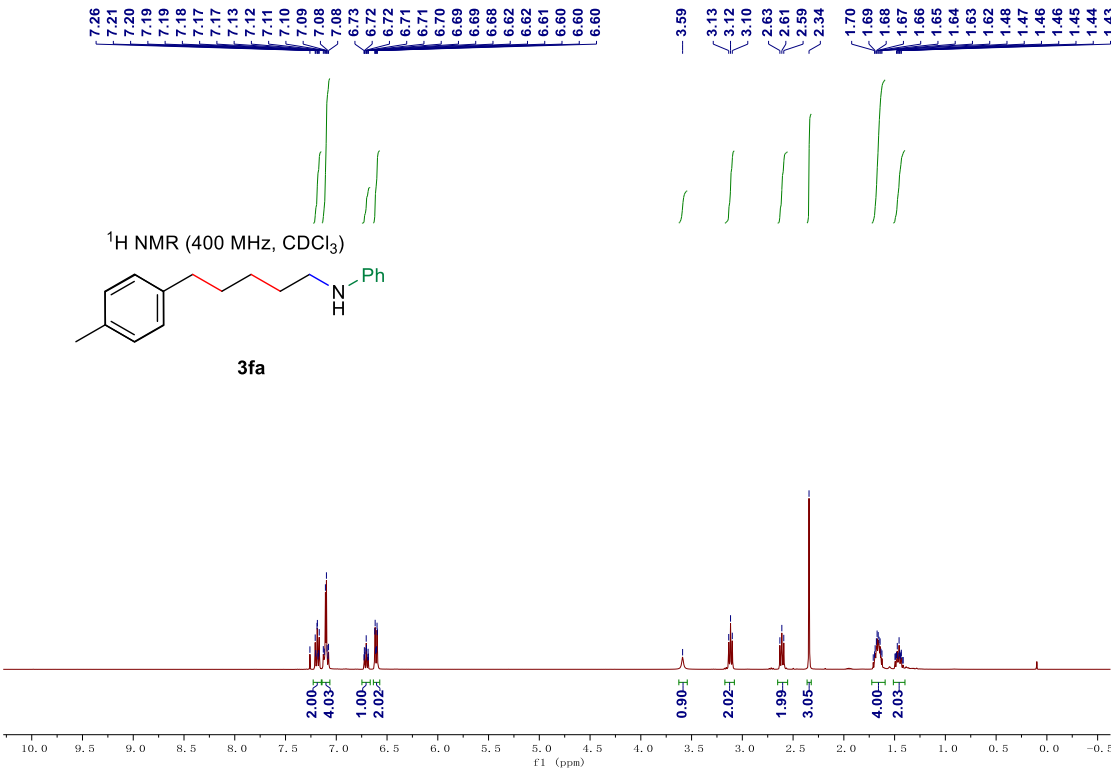
7.22
7.21
7.19
7.17
6.73
6.71
6.69
6.62
6.60
3.64
3.14
3.12
3.10
2.67
2.65
2.63
1.73
1.71
1.69
1.69
1.67
1.67
1.66
1.65
1.63
1.50
1.49
1.48
1.46
1.45
1.44
1.42
1H NMR (400 MHz, CDCl3)
Ph
N
H
Ph
3da
2.00
5.04
1.00
2.00
1.01
2.01
2.00
4.00
2.02
f1 (ppm)


148.54
142.62
129.36
128.54
128.43
125.84
117.29
112.85
77.48
77.16
76.84
44.04
36.00
31.38
29.55
26.90
13C NMR (100 MHz, CDCl3)
Ph
N
H
Ph
3da
f1 (ppm)

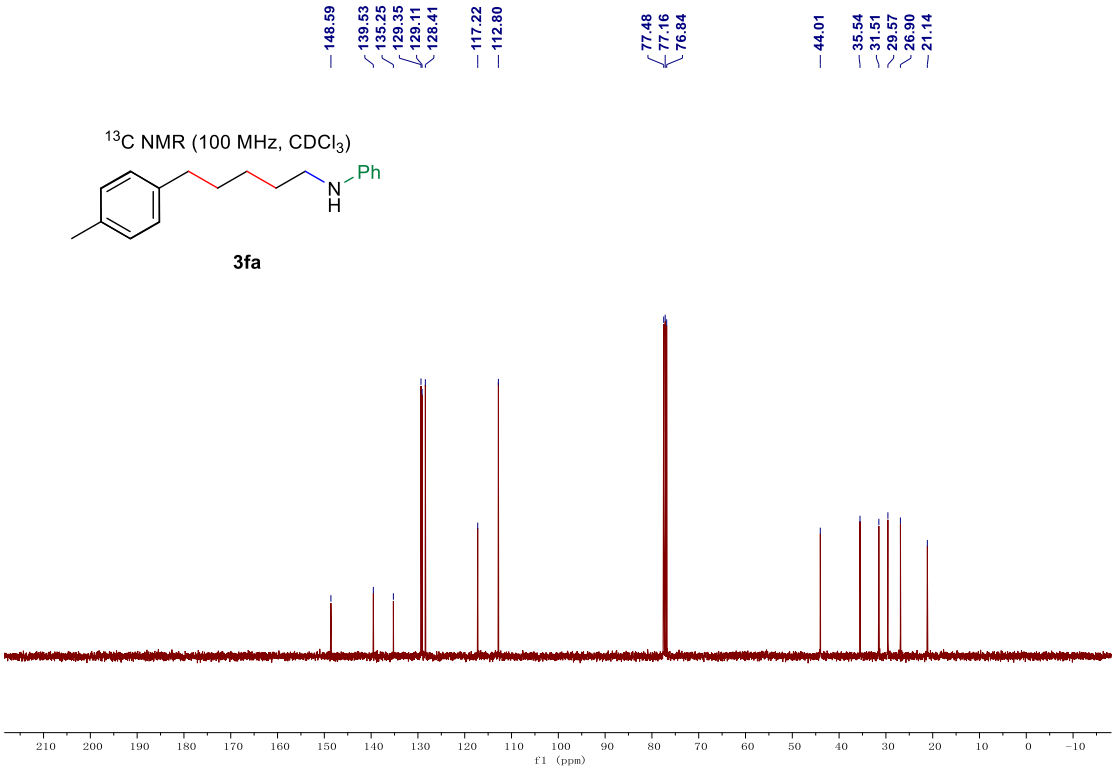

7.26 7.20 7.19 7.19 7.18 7.18 7.18 7.17 7.17 7.11 7.11 7.10 7.10 6.86 6.85 6.85 6.84 6.84 6.83 6.72 6.71 6.71 6.70 6.70 6.70 6.69 6.69 6.62 6.61 6.61 6.61 6.61 6.60 6.60
3.81 3.80 3.80 3.12 3.11 3.10 3.10 2.60 2.59 2.57 1.68 1.68 1.67 1.66 1.66 1.65 1.64 1.64 1.63 1.63 1.46 1.46 1.45 1.45 1.44 1.44 1.43 1.43 1.43
1H NMR (600 MHz, CDCl3)
Ph
N
H
MeO
3ea
2.04 2.01 2.04 1.02 2.01 3.01 0.94 2.03 2.00 4.03 2.01
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
f1 (ppm)


157.83 148.54 134.70 129.39 129.36 117.28 113.85 113.80 112.86 77.37 77.16 76.95 55.38 44.06 35.05 31.61 29.54 26.83
13C NMR (150 MHz, CDCl3)
Ph
N
H
MeO
3ea
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)



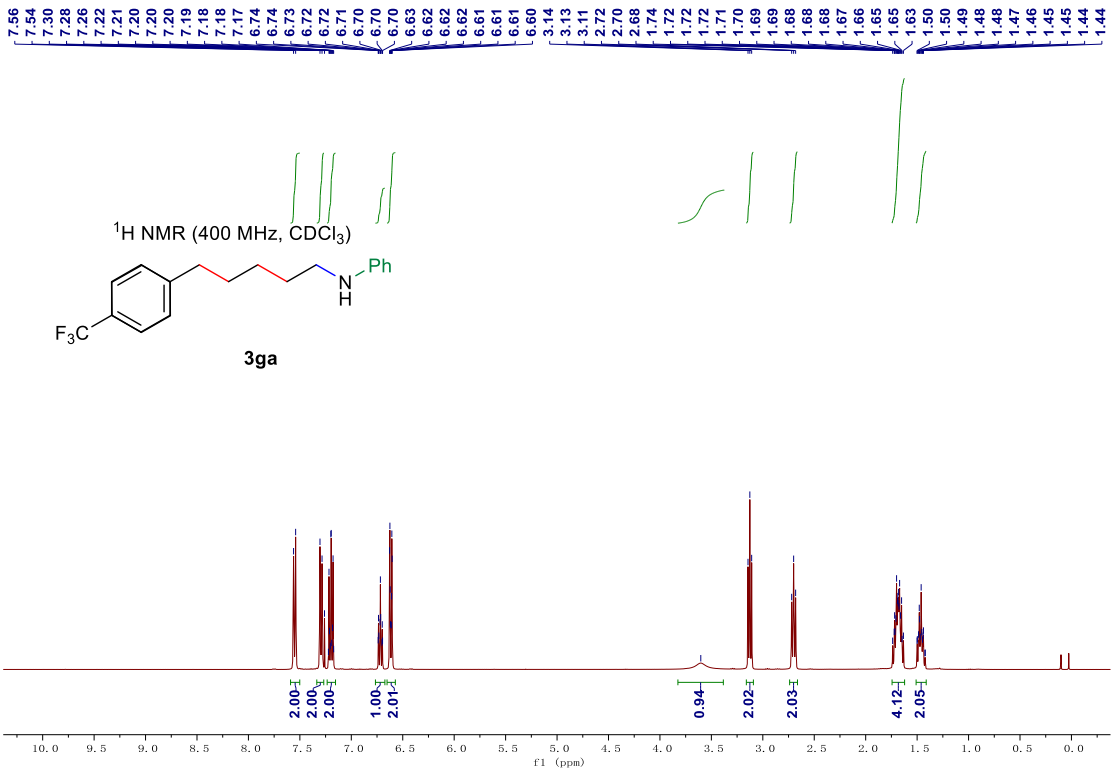
^{1}H NMR (400 MHz, $CDCl_3$)

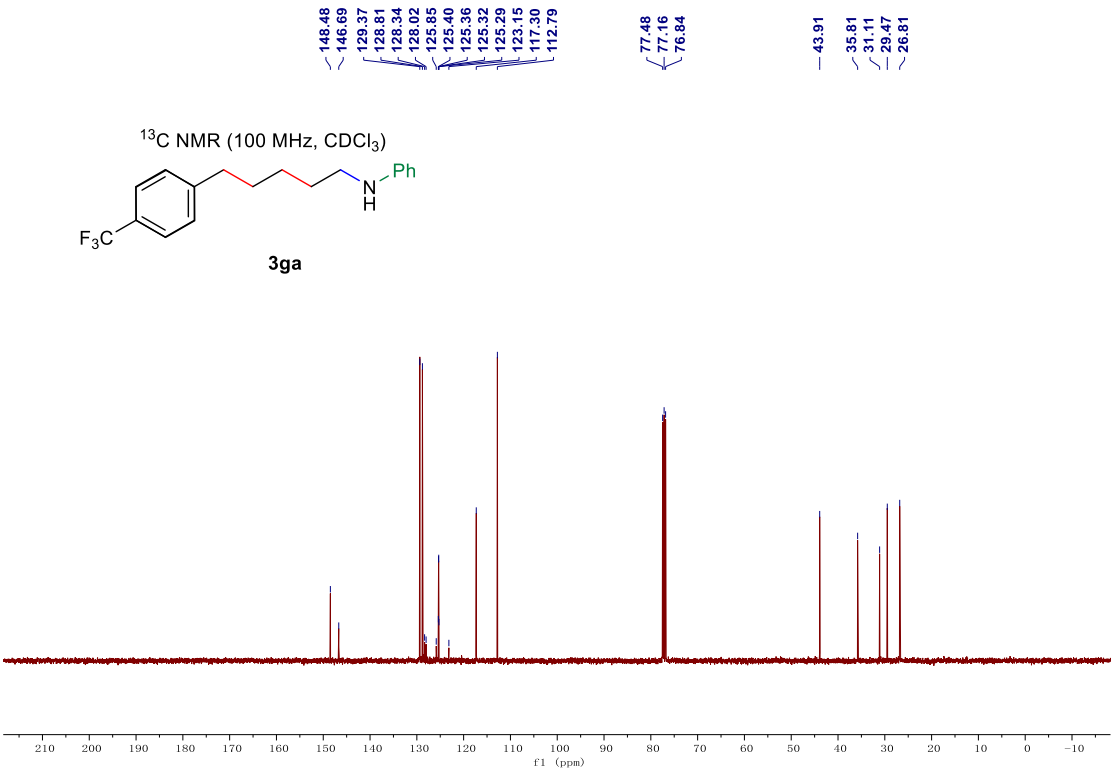
3fa



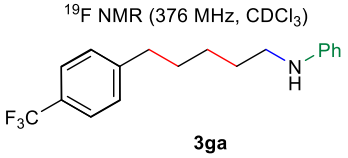
^{13}C NMR (100 MHz, $CDCl_3$)

3fa


7.56 7.54 7.30 7.28 7.26 7.22 7.21 7.20 7.20 7.20 7.19 7.18 7.18 7.17 6.74 6.74 6.73 6.72 6.72 6.71 6.70 6.70 6.70 6.63 6.62 6.62 6.62 6.61 6.61 6.61 6.60
3.14 3.13 3.11 2.72 2.70 2.68 1.74 1.72 1.72 1.72 1.71 1.70 1.69 1.69 1.68 1.68 1.68 1.67 1.66 1.65 1.65 1.63 1.50 1.50 1.49 1.48 1.48 1.47 1.46 1.45 1.45 1.44 1.44
1H NMR (400 MHz, CDCl3)
Ph
N
H
F3C
3ga
2.00 2.00 2.00 1.00 2.01 0.94 2.02 2.03 4.12 2.05
f1 (ppm)

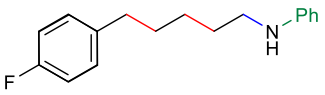

148.48 146.69 129.37 128.81 128.34 128.02 125.85 125.40 125.36 125.32 125.29 123.15 117.30 112.79
77.48 77.16 76.84
43.91 35.81 31.11 29.47 26.81
13C NMR (100 MHz, CDCl3)
Ph
N
H
F3C
3ga
f1 (ppm)

^{19}F NMR (376 MHz, $CDCl_3$)

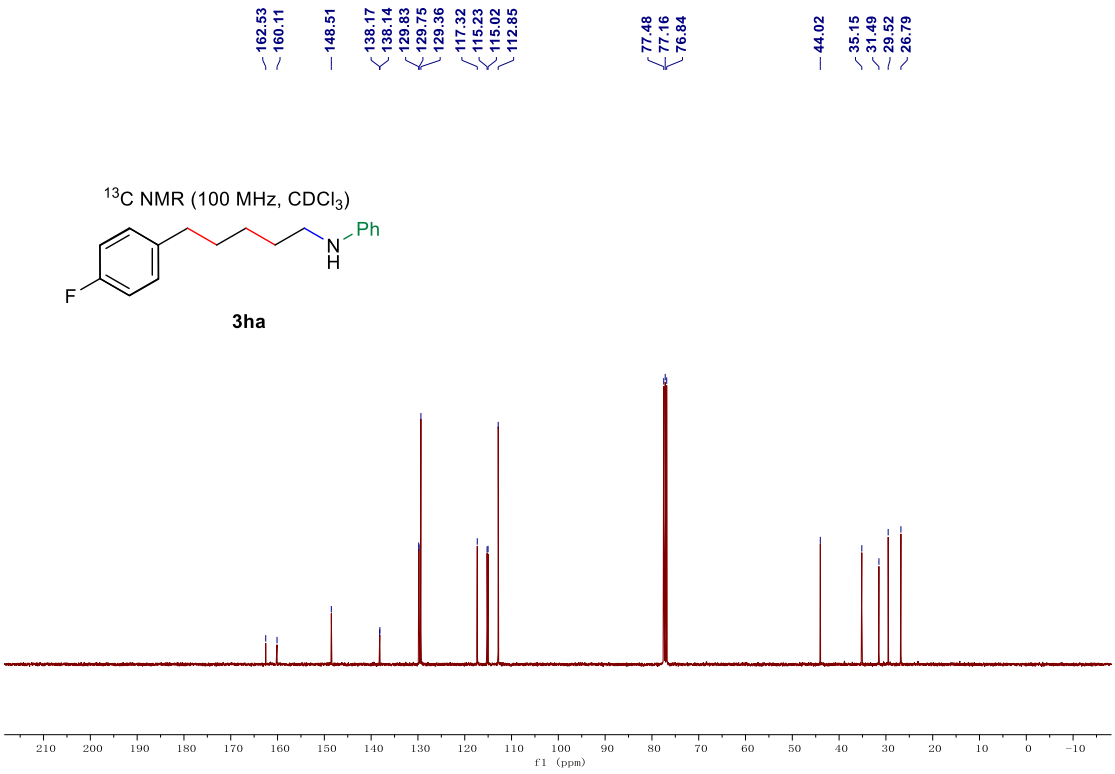


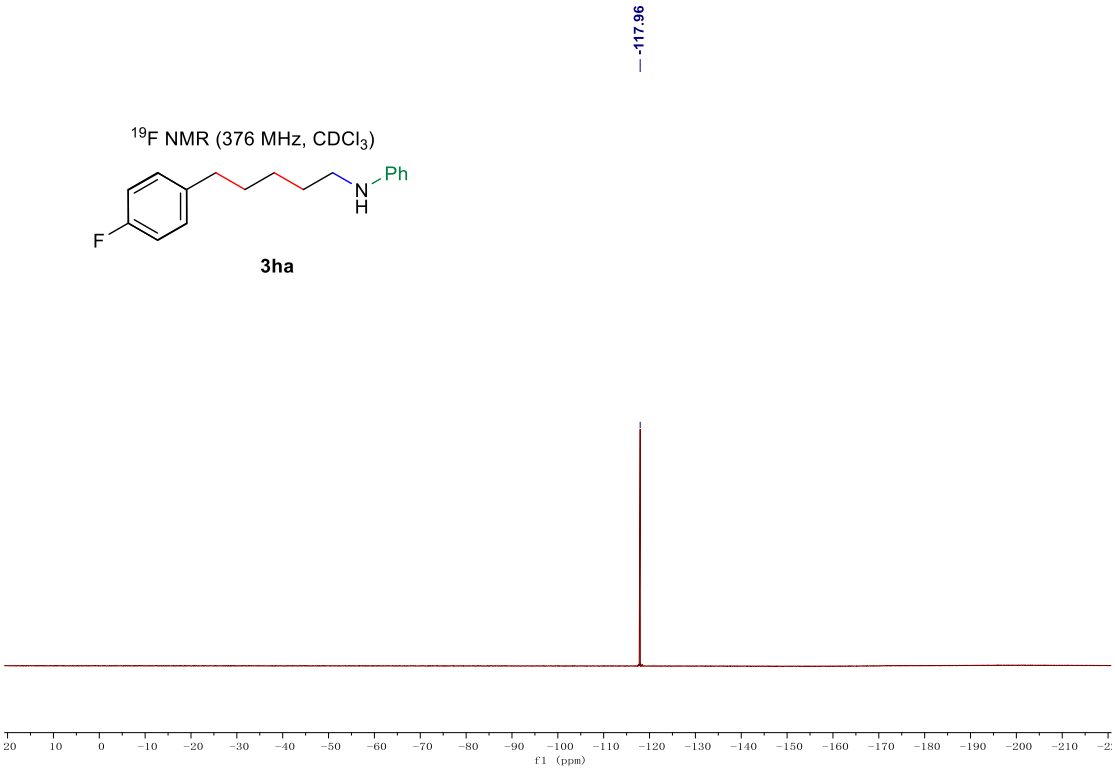
3ga

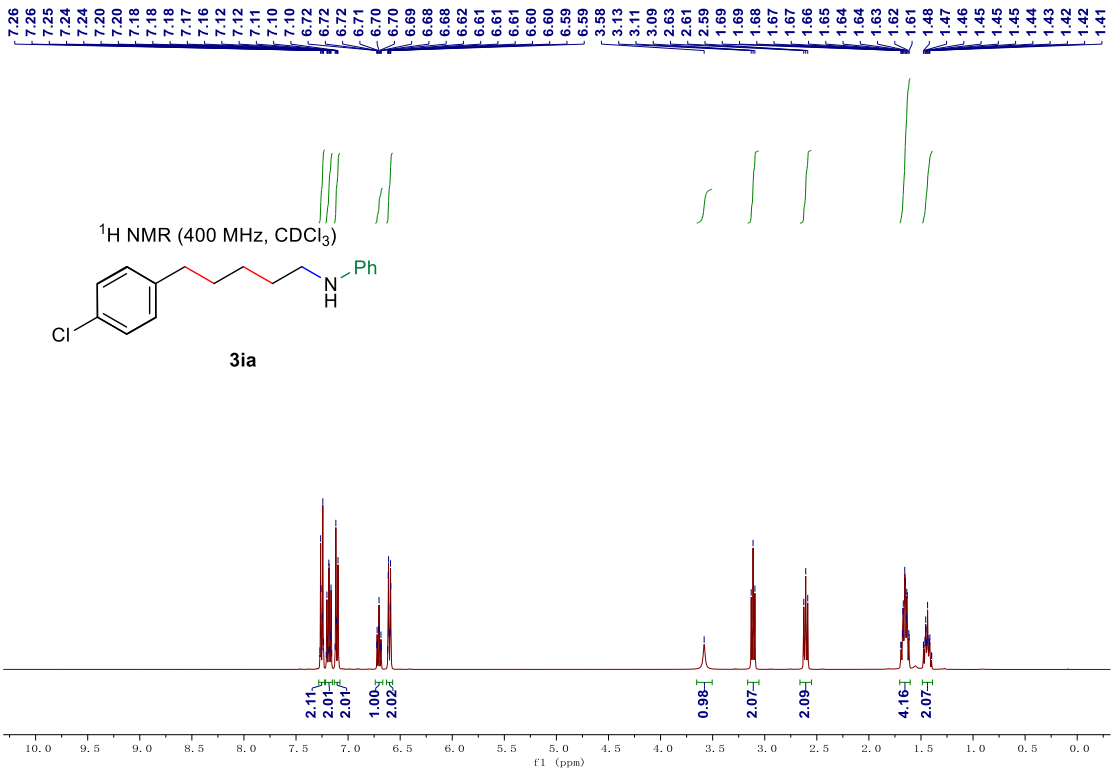
^{1}H NMR (400 MHz, $CDCl_3$)

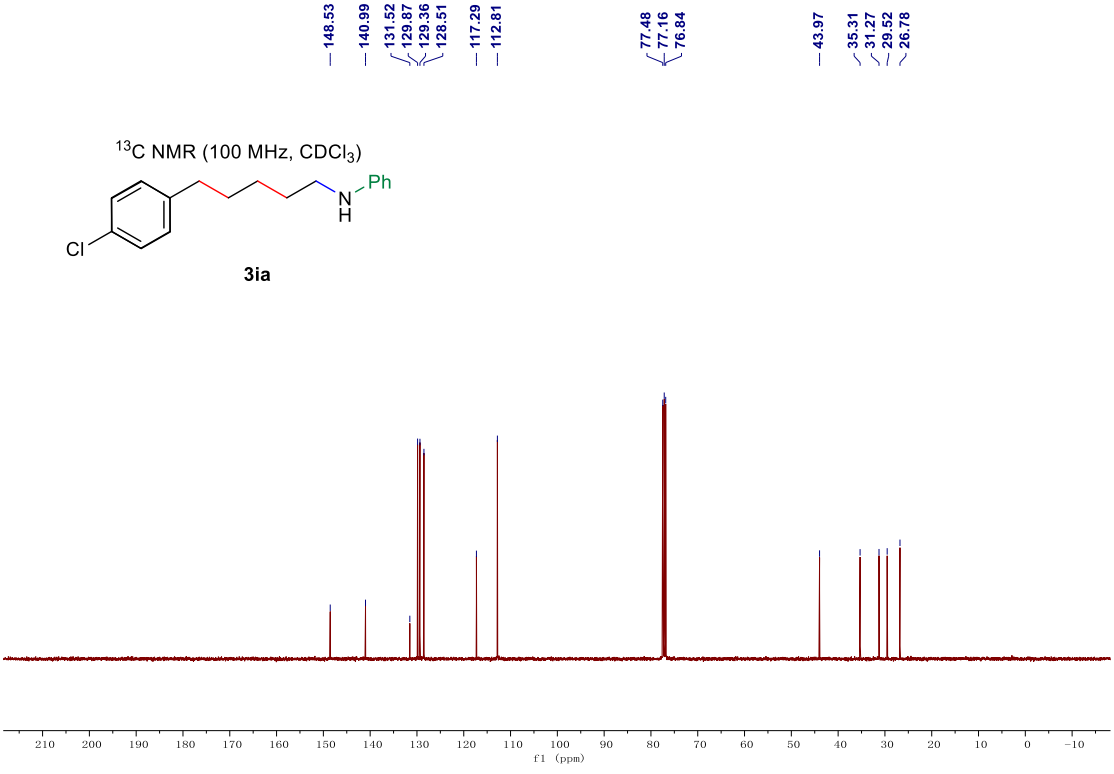


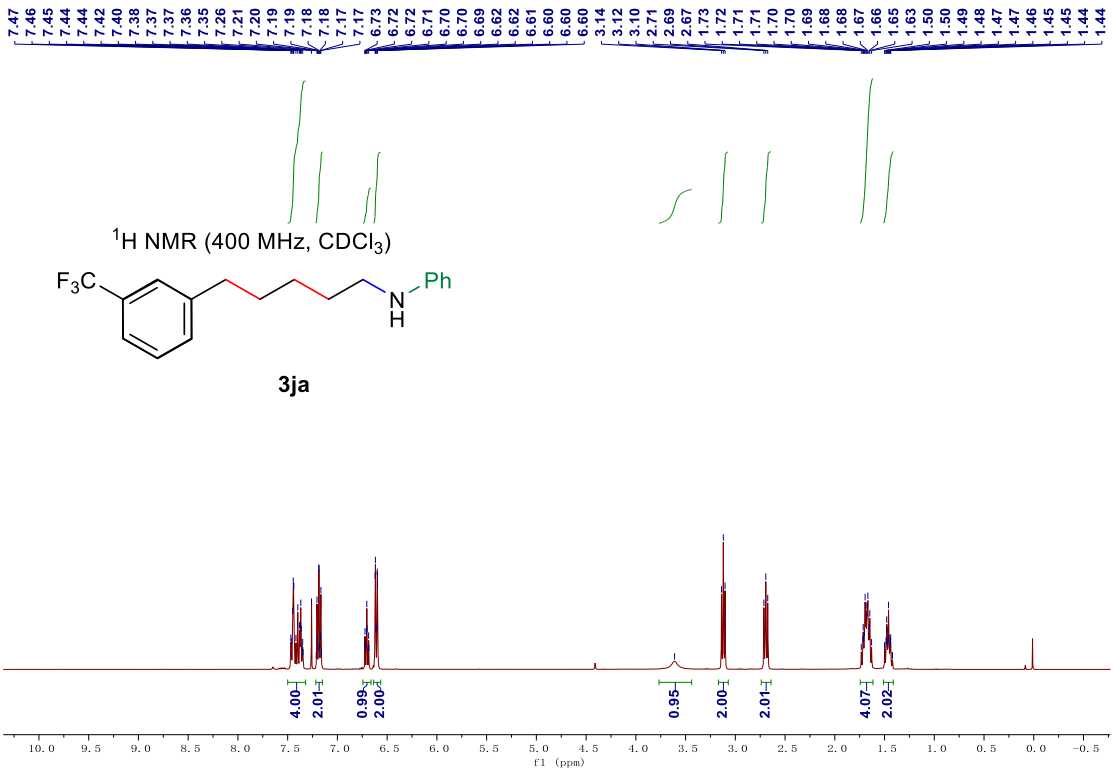
3ha

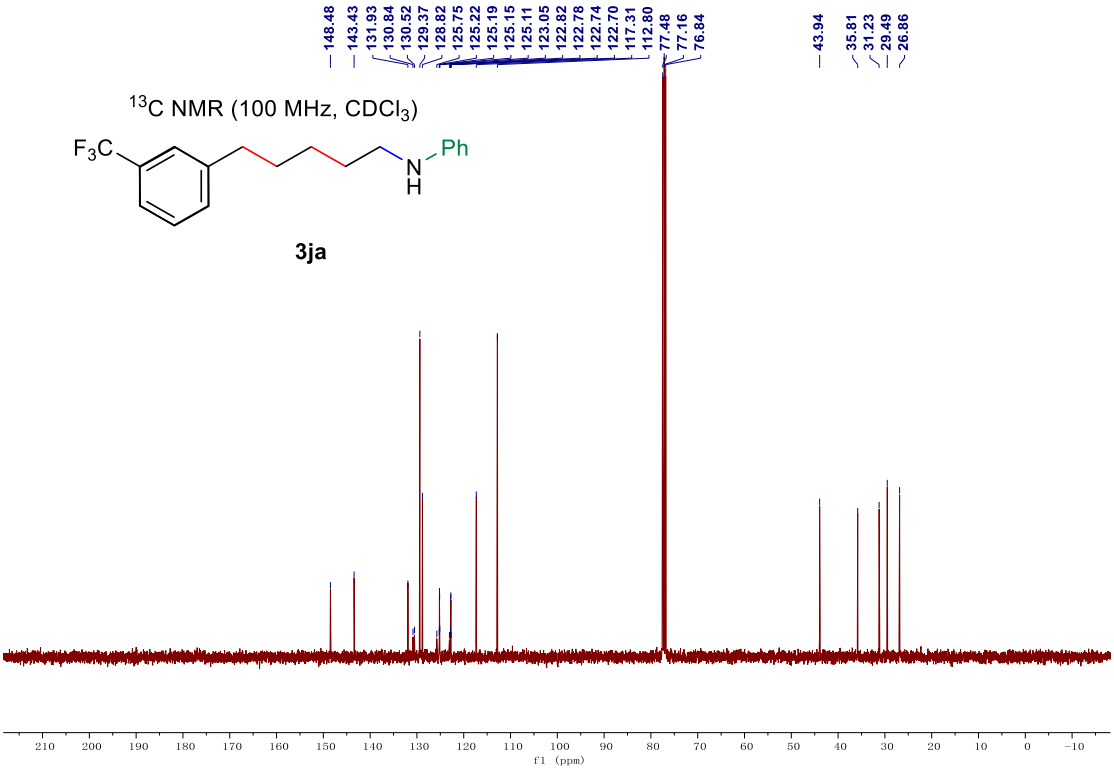

162.53
160.11
148.51
138.17
138.14
129.83
129.75
129.36
117.32
115.23
115.02
112.85
77.48
77.16
76.84
44.02
35.15
31.49
29.52
26.79
13C NMR (100 MHz, CDCl3)
Ph
N
H
F
3ha
f1 (ppm)

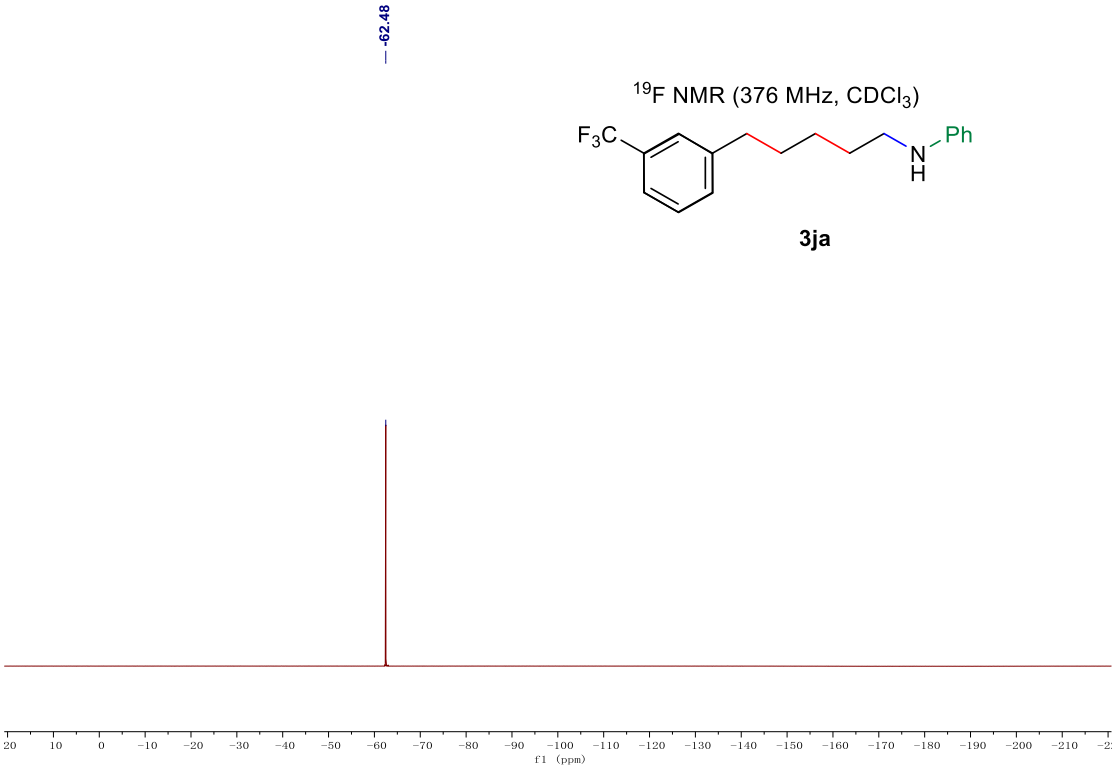

-117.96
19F NMR (376 MHz, CDCl3)
Ph
N
H
F
3ha
f1 (ppm)

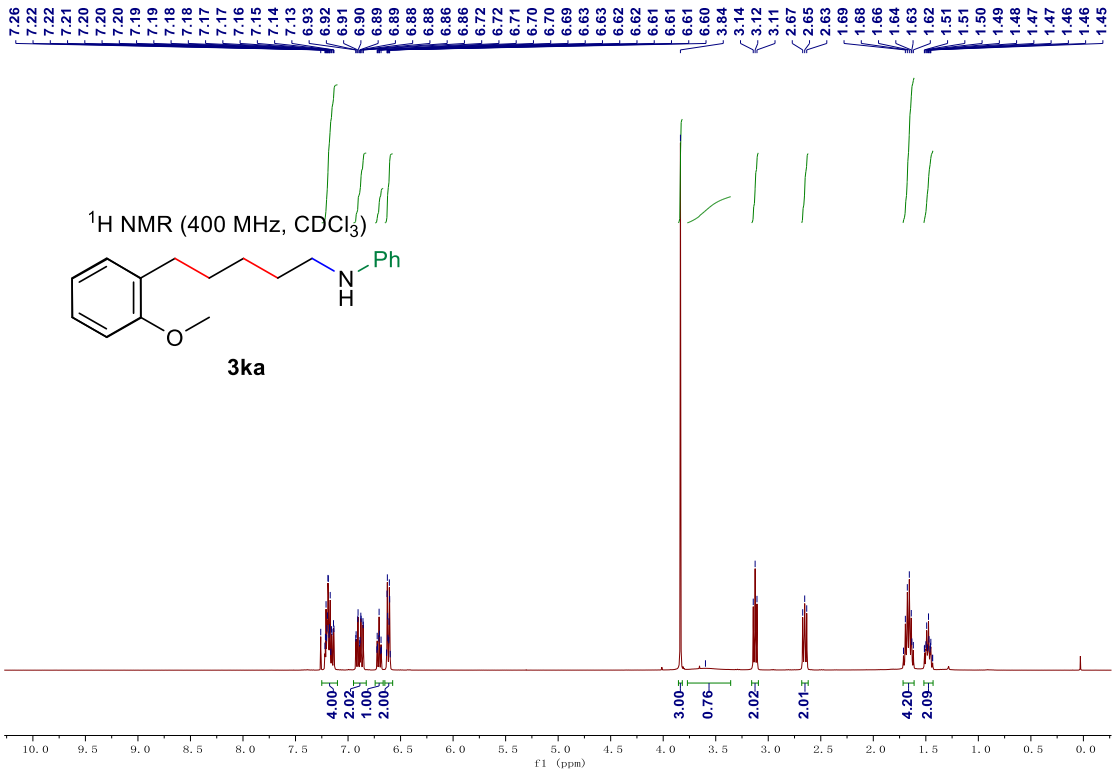

7.26 7.26 7.25 7.24 7.24 7.20 7.20 7.18 7.18 7.18 7.17 7.16 7.12 7.12 7.11 7.10 7.10 6.72 6.72 6.71 6.70 6.70 6.69 6.68 6.68 6.62 6.61 6.61 6.61 6.60 6.60 6.59 6.59 3.58 3.13 3.11 3.09 2.63 2.61 2.59 1.69 1.69 1.68 1.67 1.67 1.66 1.65 1.64 1.64 1.63 1.62 1.61 1.48 1.47 1.46 1.45 1.45 1.44 1.43 1.42 1.42 1.41
1H NMR (400 MHz, CDCl3)
Ph
N
H
Cl
3ia
2.11 2.01 2.01 1.00 2.02 0.98 2.07 2.09 4.16 2.07
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
f1 (ppm)


148.53 140.99 131.52 129.87 129.36 128.51 117.29 112.81 77.48 77.16 76.84 43.97 35.31 31.27 29.52 26.78
13C NMR (100 MHz, CDCl3)
Ph
N
H
Cl
3ia
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)


7.47 7.46 7.45 7.44 7.44 7.42 7.40 7.38 7.37 7.37 7.36 7.35 7.26 7.21 7.20 7.19 7.19 7.18 7.18 7.17 7.17
6.73 6.72 6.72 6.71 6.70 6.70 6.69 6.62 6.62 6.61 6.60 6.60 6.60
3.14 3.12 3.10 2.71 2.69 2.67 1.73 1.72 1.71 1.71 1.70 1.70 1.69 1.68 1.68 1.67 1.66 1.65 1.63 1.50 1.50 1.49 1.48 1.47 1.47 1.46 1.45 1.45 1.44 1.44
^{1}H NMR (400 MHz, $CDCl_3$)
F_3C
N
H
Ph
3ja
4.00 2.01 0.99 2.00 0.95 2.00 2.01 4.07 2.02
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)


148.48 143.43 131.93 130.84 130.52 129.37 128.82 125.75 125.22 125.19 125.15 125.11 123.05 122.82 122.78 122.74 122.70 117.31 112.80 77.48 77.16 76.84
43.94 35.81 31.23 29.49 26.86
^{13}C NMR (100 MHz, $CDCl_3$)
F_3C
N
H
Ph
3ja
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)


-62.48
19F NMR (376 MHz, CDCl3)
F3C
N
H
Ph
3ja
f1 (ppm)


1H NMR (400 MHz, CDCl3)
N
H
Ph
O
3ka
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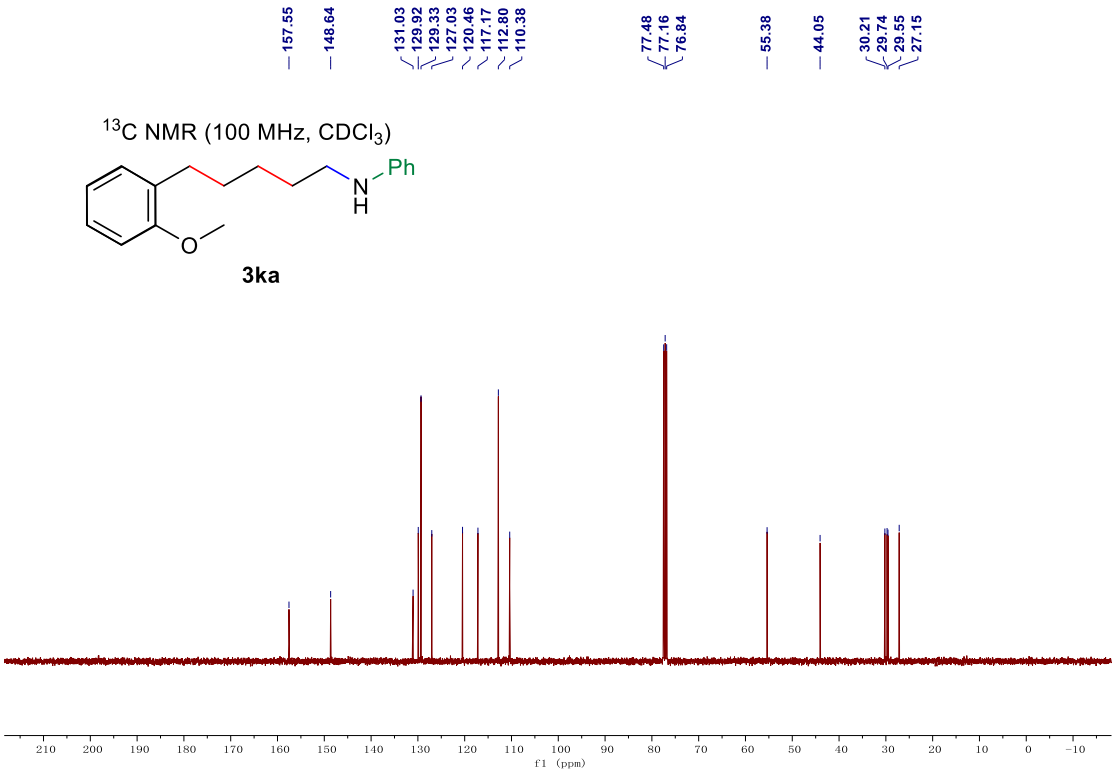
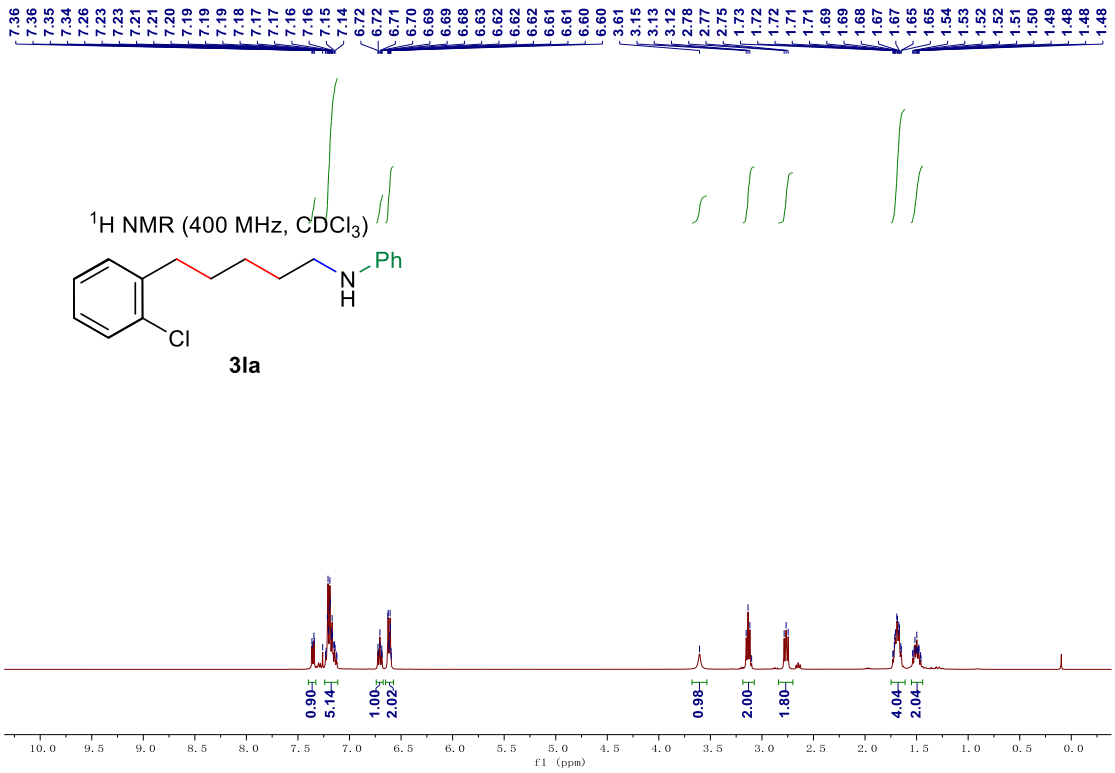
2.02
1.00
2.00
3.00
0.76
2.02
2.01
4.20
2.09
f1 (ppm)


157.55
148.64
131.03
129.92
129.33
127.03
120.46
117.17
112.80
110.38
77.48
77.16
76.84
55.38
44.05
30.21
29.74
29.55
27.15
13C NMR (100 MHz, CDCl3)
Ph
N
H
O
3ka
f1 (ppm)


7.36
7.36
7.35
7.34
7.26
7.23
7.23
7.21
7.21
7.20
7.19
7.19
7.19
7.18
7.17
7.17
7.16
7.16
7.15
7.14
6.72
6.72
6.71
6.70
6.69
6.69
6.68
6.63
6.62
6.62
6.62
6.61
6.61
6.60
6.60
3.61
3.15
3.13
3.12
2.78
2.77
2.75
1.73
1.72
1.72
1.71
1.71
1.69
1.69
1.68
1.67
1.67
1.65
1.65
1.54
1.53
1.52
1.52
1.51
1.50
1.49
1.48
1.48
1.48
1H NMR (400 MHz, CDCl3)
Ph
N
H
Cl
3la
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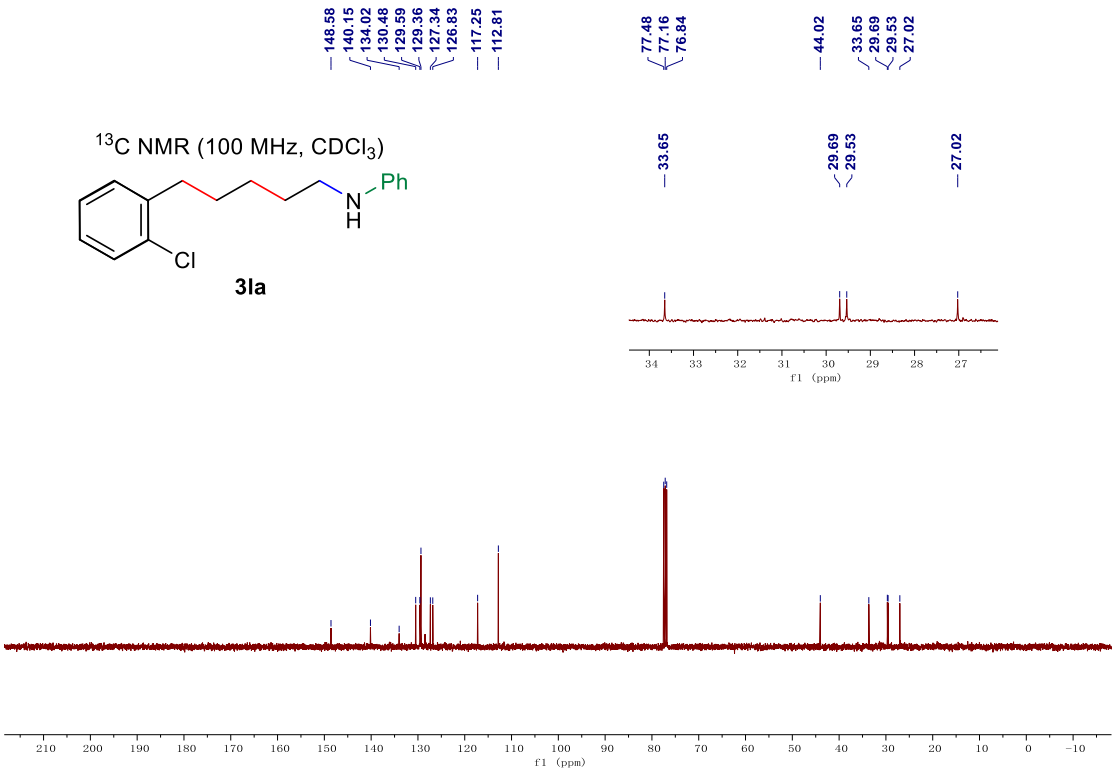
5.14
1.00
2.02
0.98
2.00
1.80
4.04
2.04
f1 (ppm)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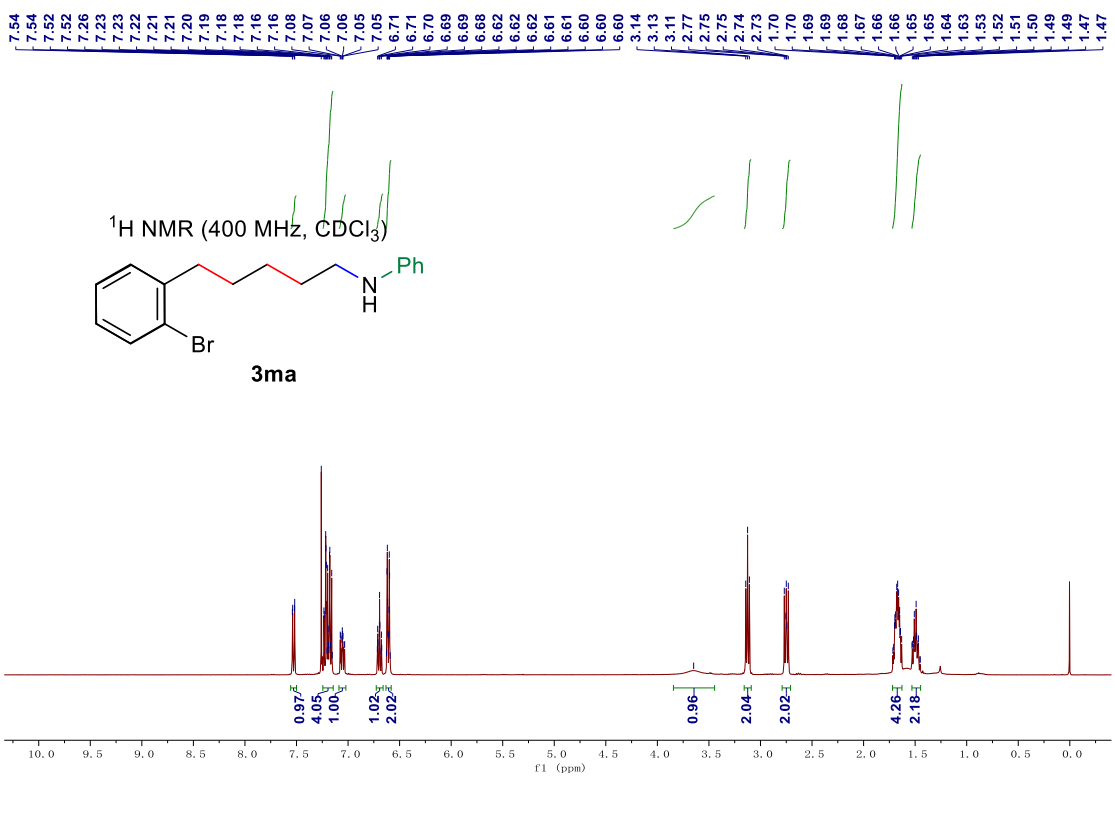
140.15
134.02
130.48
129.59
129.36
127.34
126.83
117.25
112.81
77.48
77.16
76.84
44.02
33.65
29.69
29.53
27.02
13C NMR (100 MHz, CDCl3)
Ph
N
H
Cl
3la
f1 (ppm)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7.26
7.23
7.22
7.21
7.20
7.19
7.18
7.16
7.08
7.07
7.06
7.05
6.71
6.70
6.69
6.68
6.62
6.61
6.60
3.14
3.13
3.11
2.77
2.75
2.74
2.73
1.70
1.69
1.68
1.67
1.66
1.65
1.64
1.63
1.53
1.52
1.51
1.50
1.49
1.47
1H NMR (400 MHz, CDCl3)
Ph
N
H
Br
3ma
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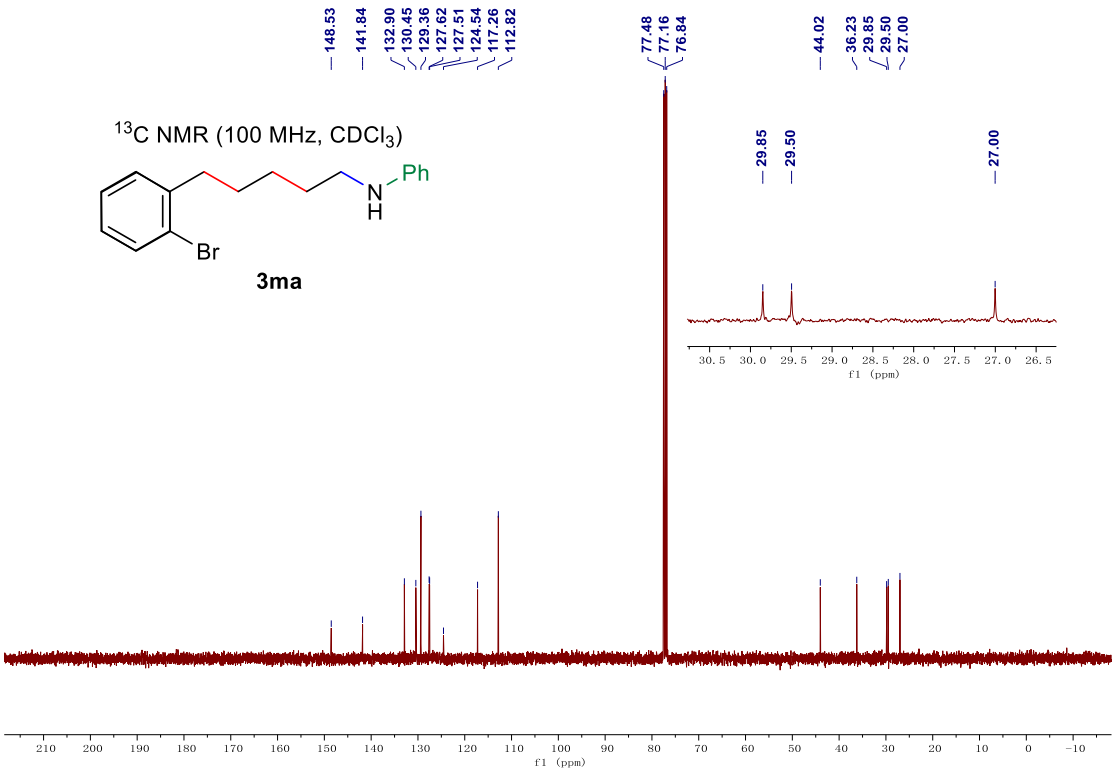
4.05
1.00
1.02
2.02
0.96
2.04
2.02
4.26
2.18
f1 (ppm)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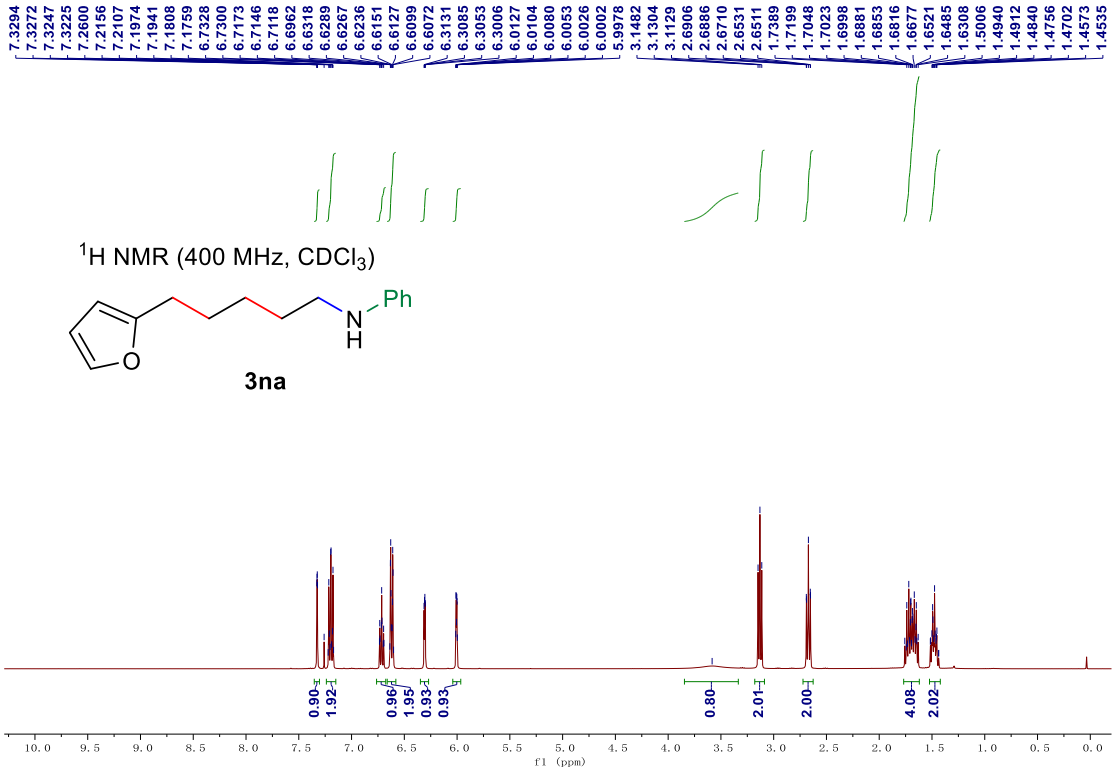
132.90
130.45
129.36
127.62
127.51
124.54
117.26
112.82
77.48
77.16
76.84
44.02
36.23
29.85
29.50
27.00
13C NMR (100 MHz, CDCl3)
Ph
N
H
Br
3ma
f1 (ppm)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7.3247
7.3225
7.2600
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7.2107
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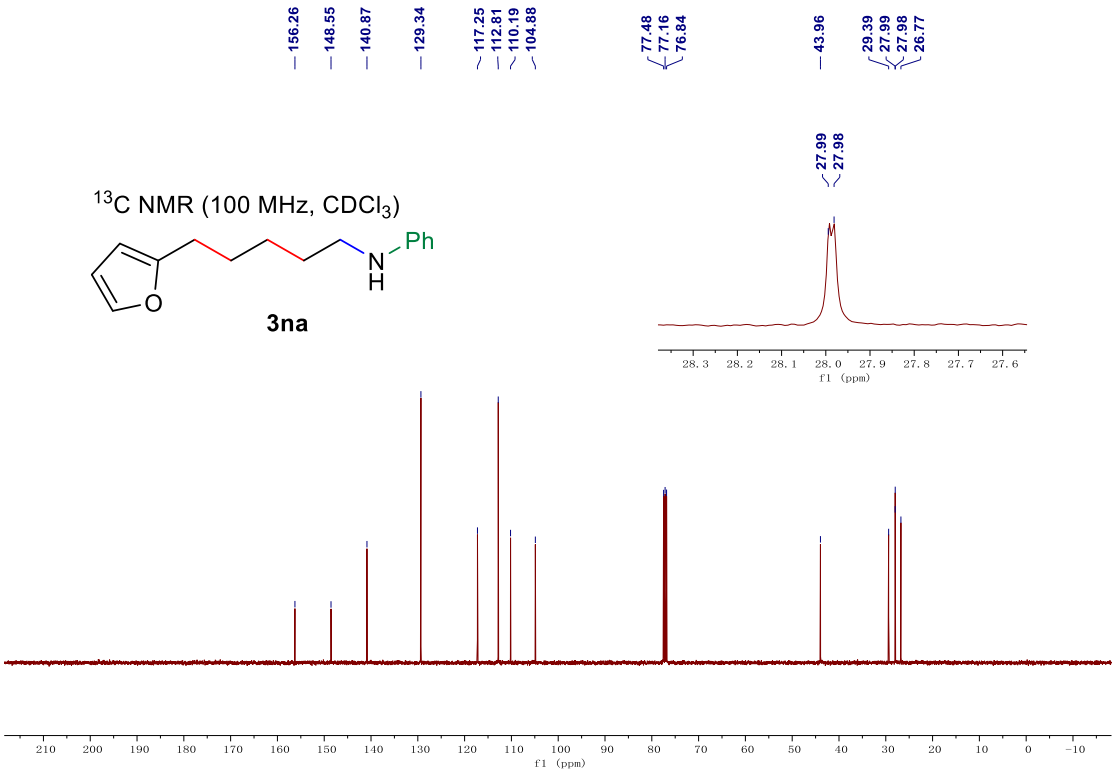
7.1941
7.1808
7.1759
6.7328
6.7300
6.7173
6.7146
6.7118
6.6962
6.6318
6.6289
6.6267
6.6236
6.6151
6.6127
6.6099
6.6072
6.3131
6.3085
6.3053
6.3006
6.0127
6.0104
6.0080
6.0053
6.0026
6.0002
5.9978
3.1482
3.1304
3.1129
2.6906
2.6886
2.6710
2.6531
2.6511
1.7389
1.7199
1.7048
1.7023
1.6998
1.6881
1.6853
1.6816
1.6677
1.6521
1.6485
1.6308
1.5006
1.4940
1.4912
1.4840
1.4756
1.4702
1.4573
1.4535
1H NMR (400 MHz, CDCl3)
Ph
N
H
O
3na
0.90
1.92
0.96
1.95
0.93
0.93
0.80
2.01
2.00
4.08
2.02
f1 (ppm)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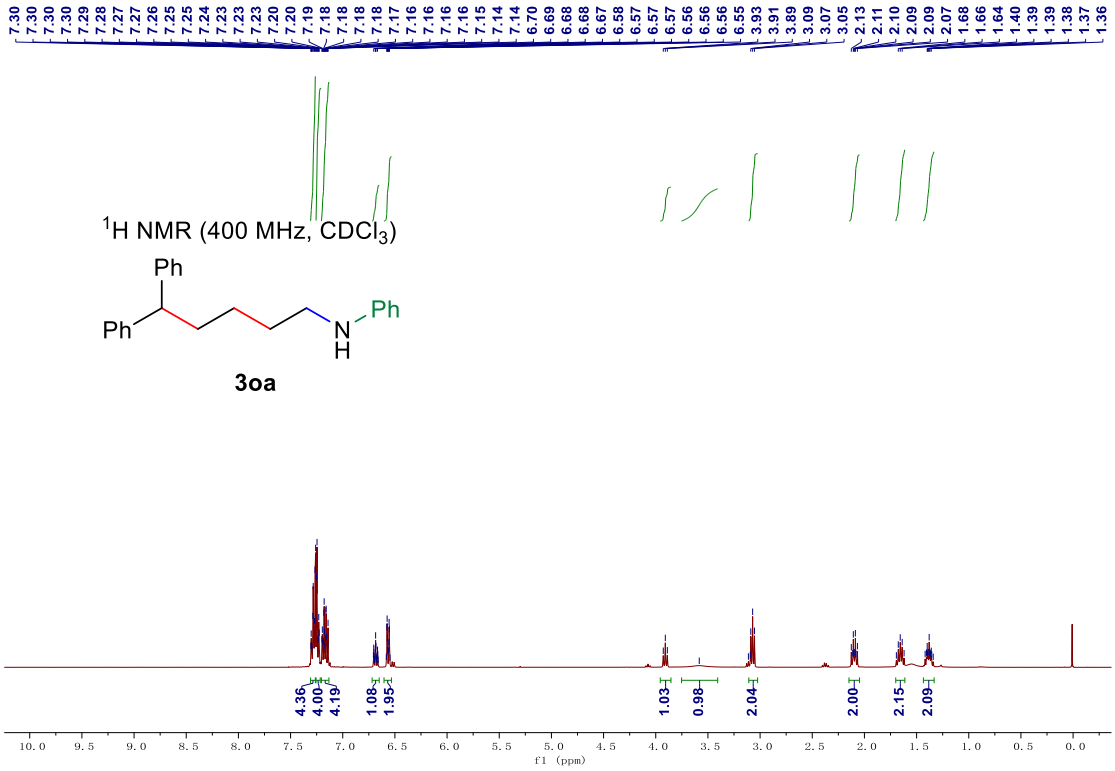
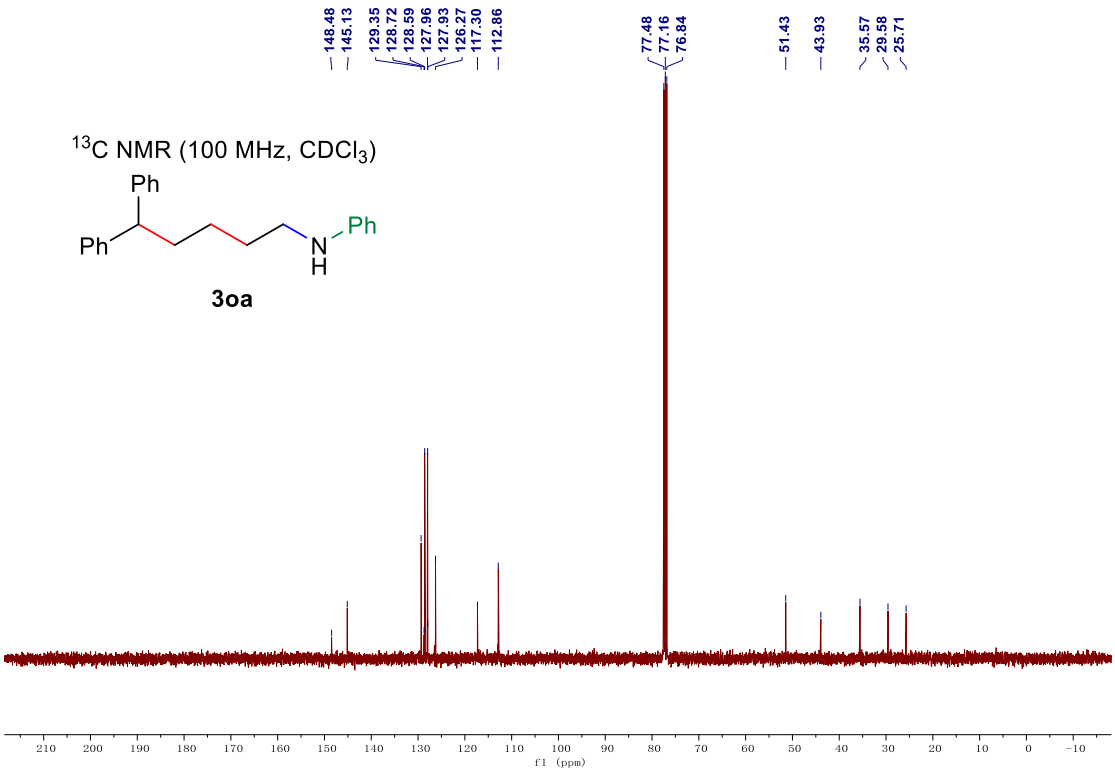
117.25
112.81
110.19
104.88
77.48
77.16
76.84
43.96
29.39
27.99
27.98
26.77
27.99
27.98
13C NMR (100 MHz, CDCl3)
Ph
N
H
O
3na
28.3 28.2 28.1 28.0 27.9 27.8 27.7 27.6
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


7.30 7.30 7.30 7.29 7.28 7.27 7.27 7.26 7.25 7.25 7.24 7.23 7.23 7.20 7.20 7.19 7.18 7.18 7.18 7.17 7.16 7.16 7.16 7.15 7.14 7.14 6.70 6.69 6.68 6.67 6.58 6.57 6.57 6.57 6.56 6.56 6.56 6.55 3.93 3.91 3.89 3.09 3.07 3.05 2.13 2.11 2.10 2.09 2.09 2.07 1.68 1.66 1.64 1.40 1.39 1.39 1.38 1.37 1.36
1H NMR (400 MHz, CDCl3)
Ph
Ph
N
H
Ph
3oa
4.36 4.00 4.19 1.08 1.95 1.03 0.98 2.04 2.00 2.15 2.09
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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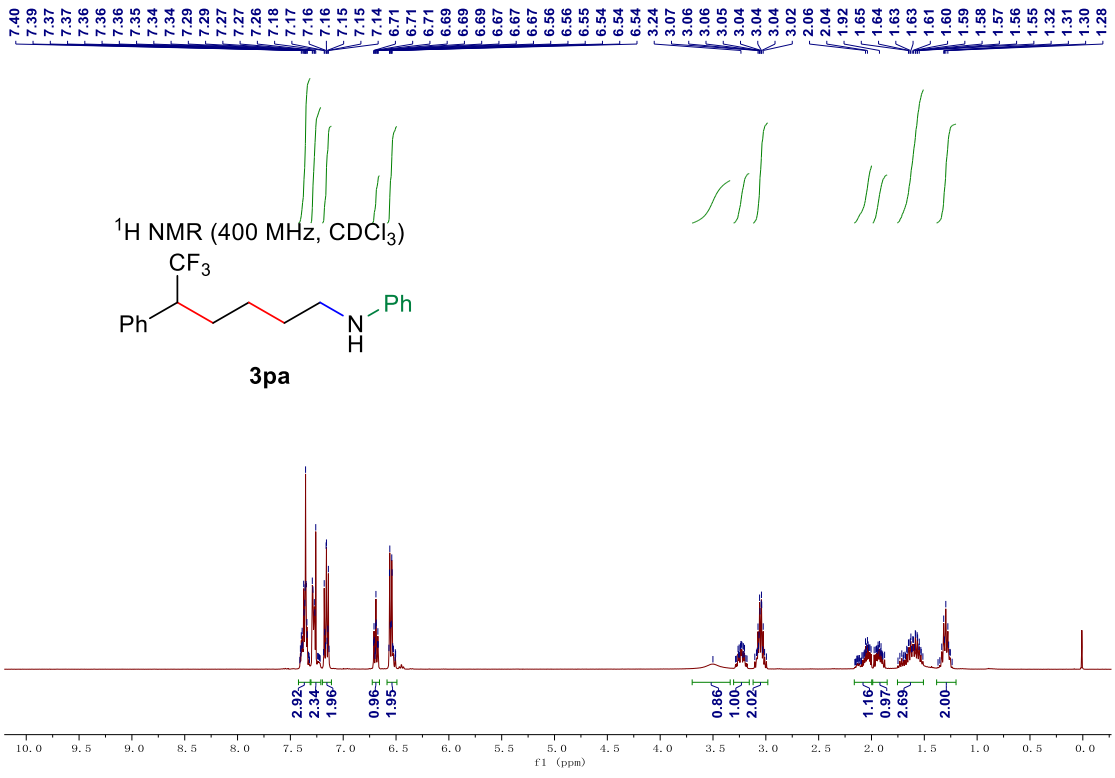
145.13
129.35
128.72
128.59
127.96
127.93
126.27
117.30
112.86
77.48
77.16
76.84
51.43
43.93
35.57
29.58
25.71
13C NMR (100 MHz, CDCl3)
Ph
Ph
N
H
Ph
3oa
f1 (ppm)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7.39
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7.37
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7.36
7.36
7.35
7.34
7.34
7.29
7.29
7.27
7.27
7.26
7.18
7.17
7.16
7.16
7.15
7.15
7.14
6.71
6.71
6.71
6.69
6.69
6.69
6.67
6.67
6.67
6.56
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6.54
6.54
6.54
3.24
3.07
3.06
3.06
3.05
3.04
3.04
3.04
3.02
2.06
2.04
1.92
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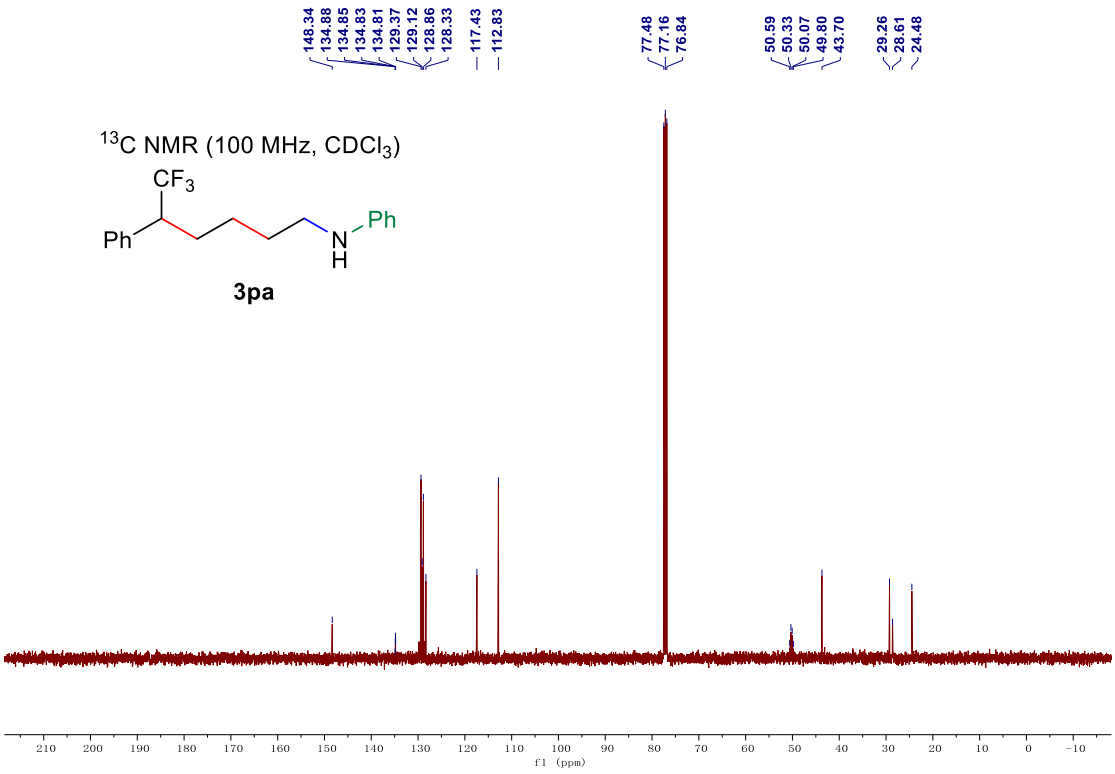
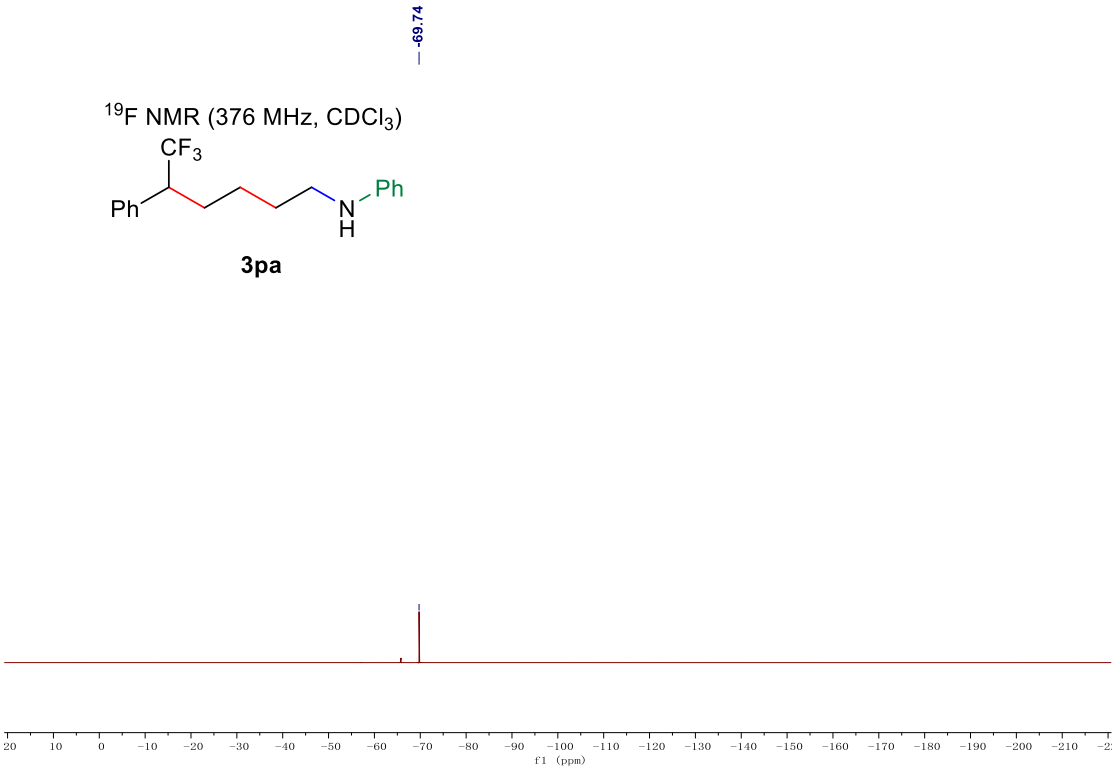
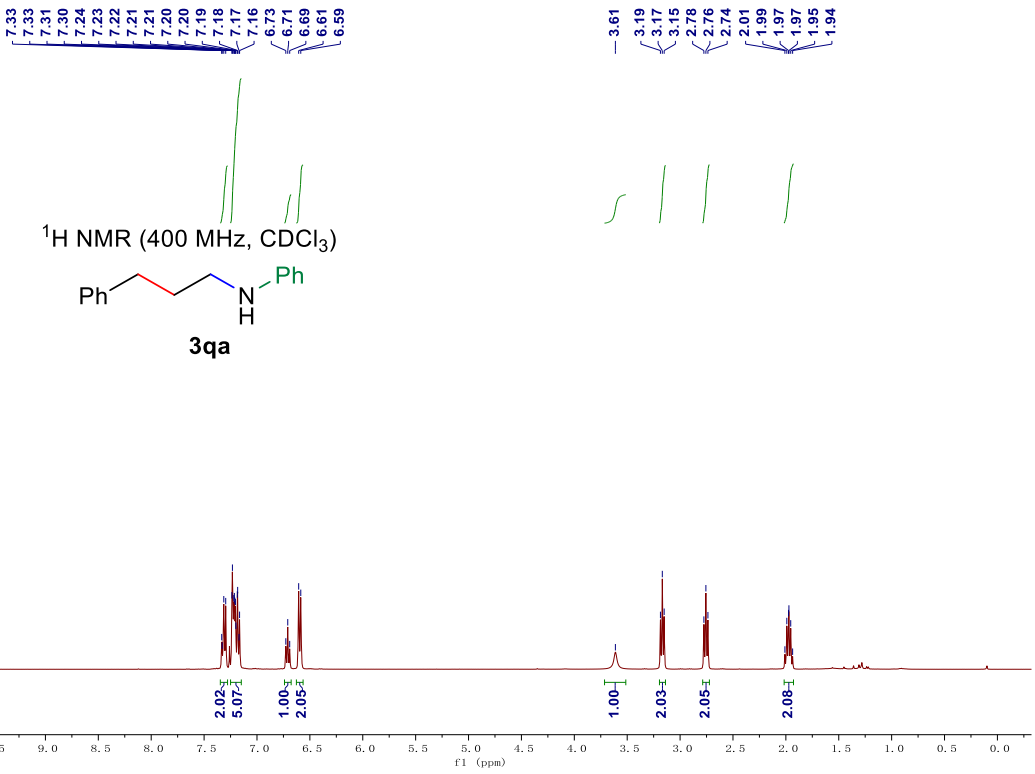
1.64
1.63
1.63
1.61
1.60
1.59
1.58
1.57
1.56
1.55
1.32
1.31
1.30
1.28
1H NMR (400 MHz, CDCl3)
CF3
Ph
N
H
Ph
3pa
2.92
2.34
1.96
0.96
1.95
0.86
1.00
2.02
1.16
0.97
2.69
2.00
f1 (ppm)


148.34
134.88
134.85
134.83
134.81
129.37
129.12
128.86
128.33
117.43
112.83
77.48
77.16
76.84
50.59
50.33
50.07
49.80
43.70
29.26
28.61
24.48
13C NMR (100 MHz, CDCl3)
CF3
Ph
N
H
Ph
3pa
f1 (ppm)


-69.74
19F NMR (376 MHz, CDCl3)
CF3
Ph
N
H
Ph
3pa
f1 (ppm)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7.33
7.31
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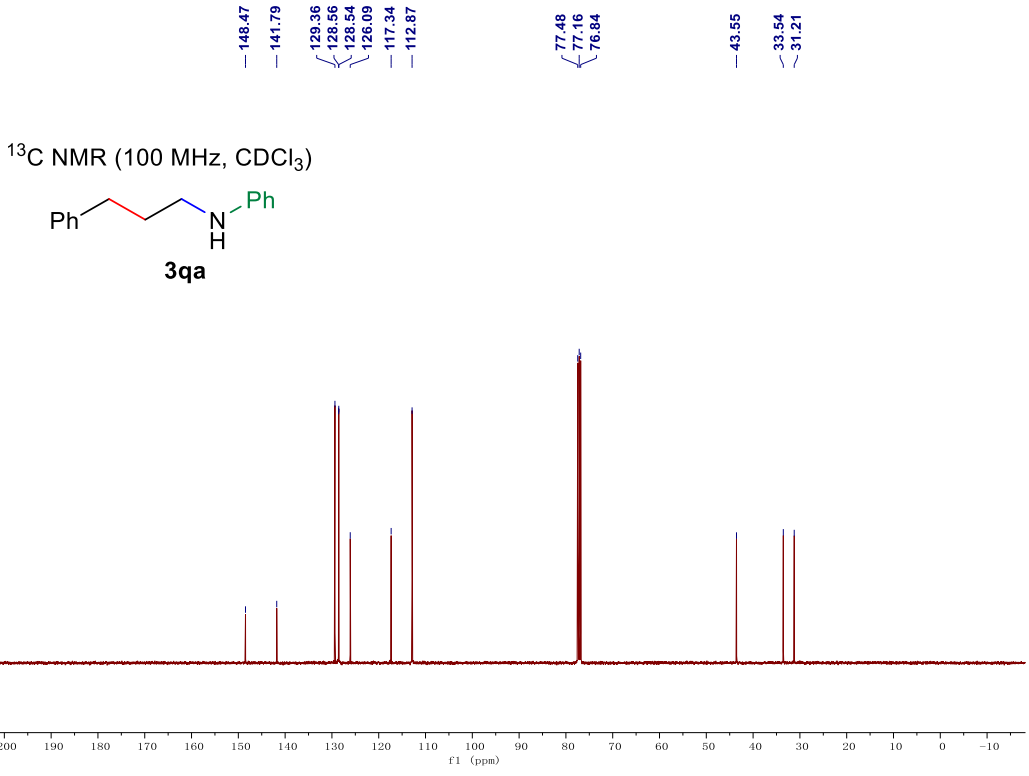
7.20
7.20
7.19
7.18
7.17
7.16
6.73
6.71
6.69
6.61
6.59
3.61
3.19
3.17
3.15
2.78
2.76
2.74
2.01
1.99
1.97
1.97
1.95
1.94
1H NMR (400 MHz, CDCl3)
Ph
N
H
Ph
3qa
2.02
5.07
1.00
2.05
1.00
2.03
2.05
2.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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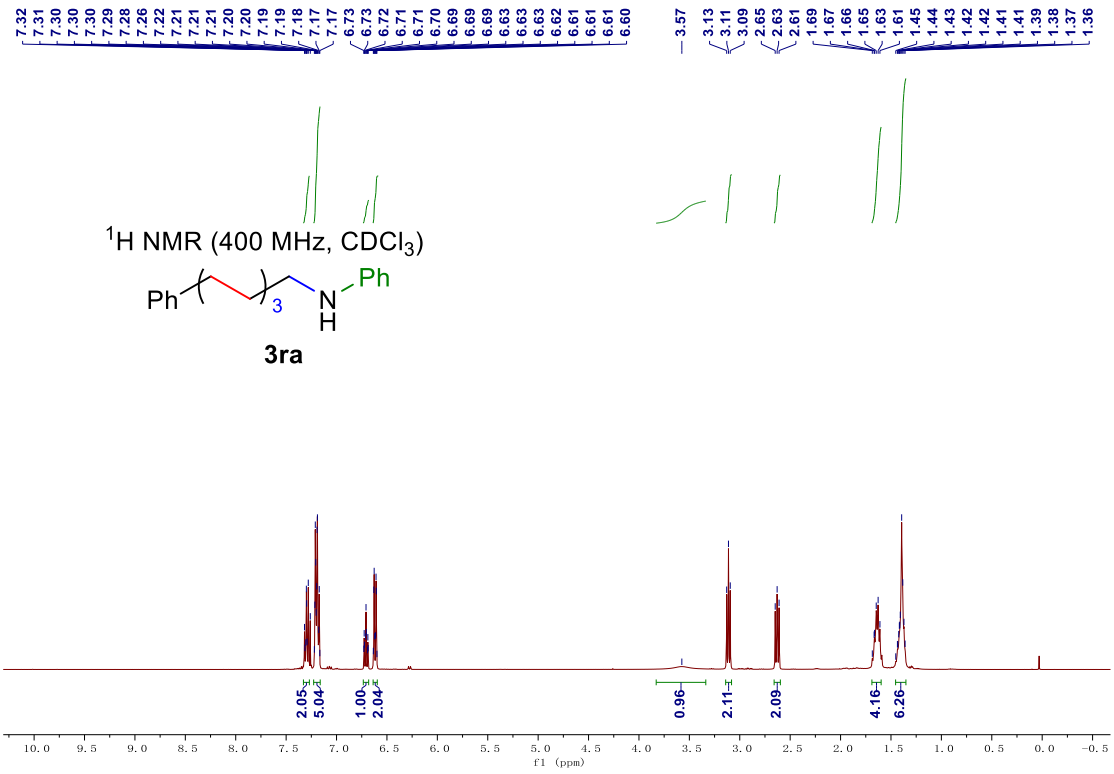
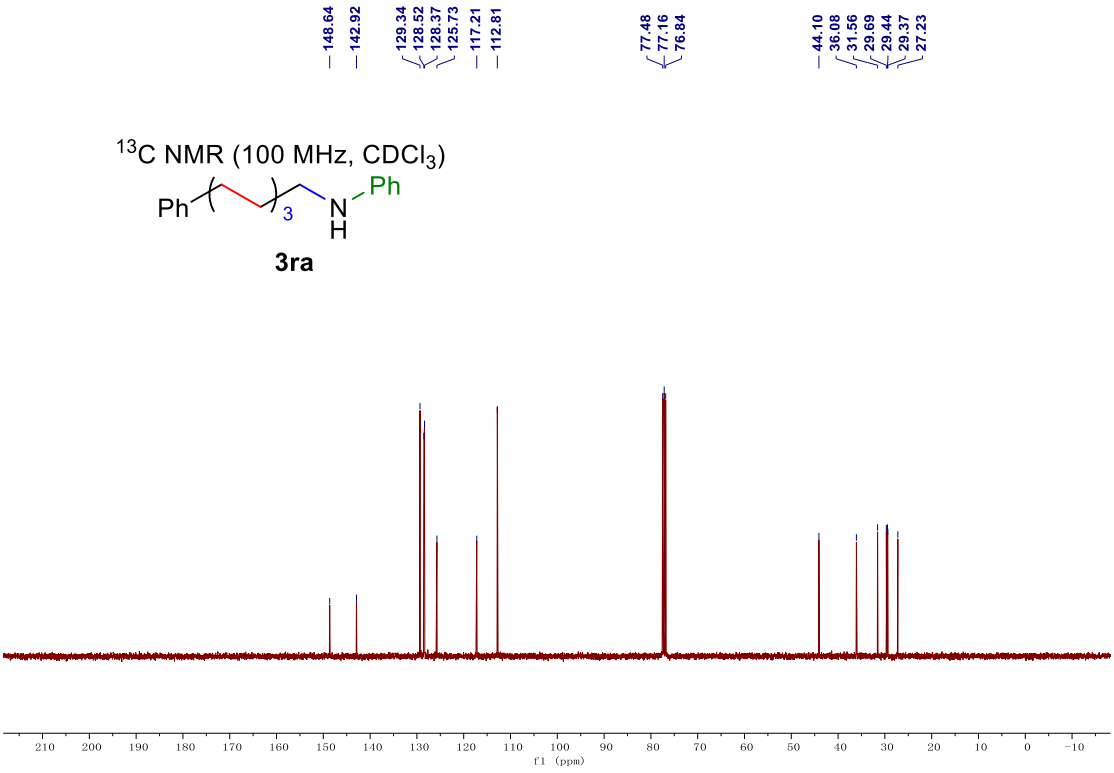
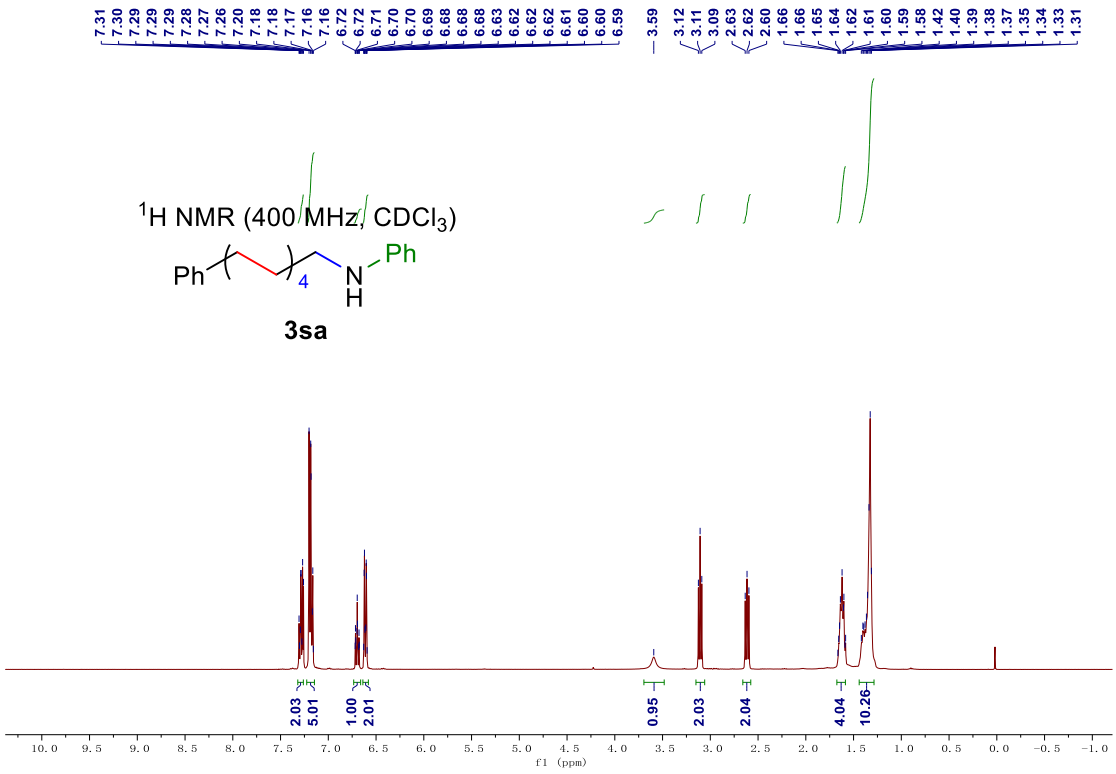
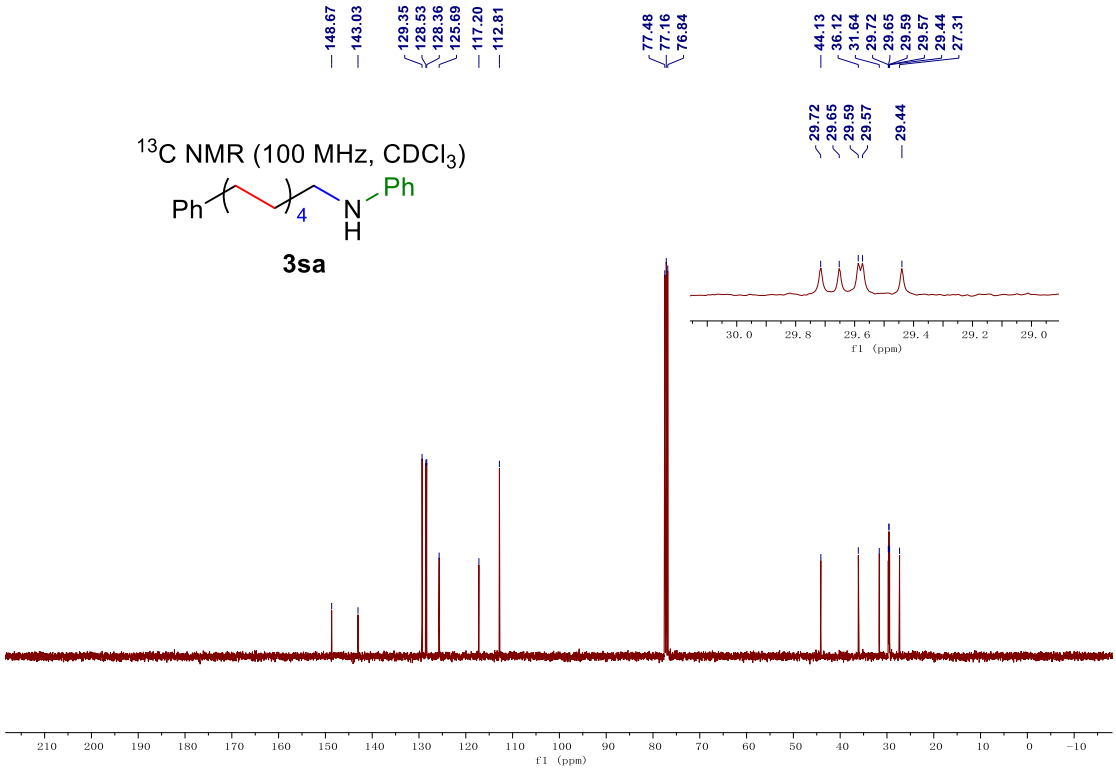
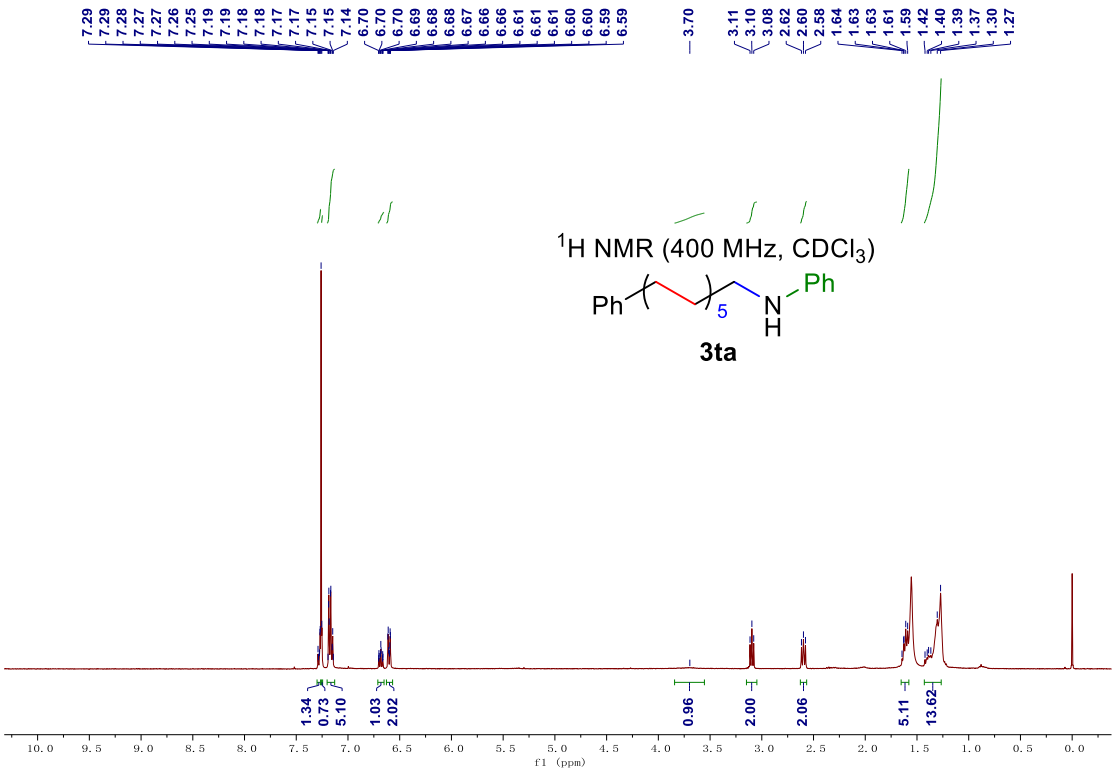
141.79
129.36
128.56
128.54
126.09
117.34
112.87
77.48
77.16
76.84
43.55
33.54
31.21
13C NMR (100 MHz, CDCl3)
Ph
N
H
Ph
3qa
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

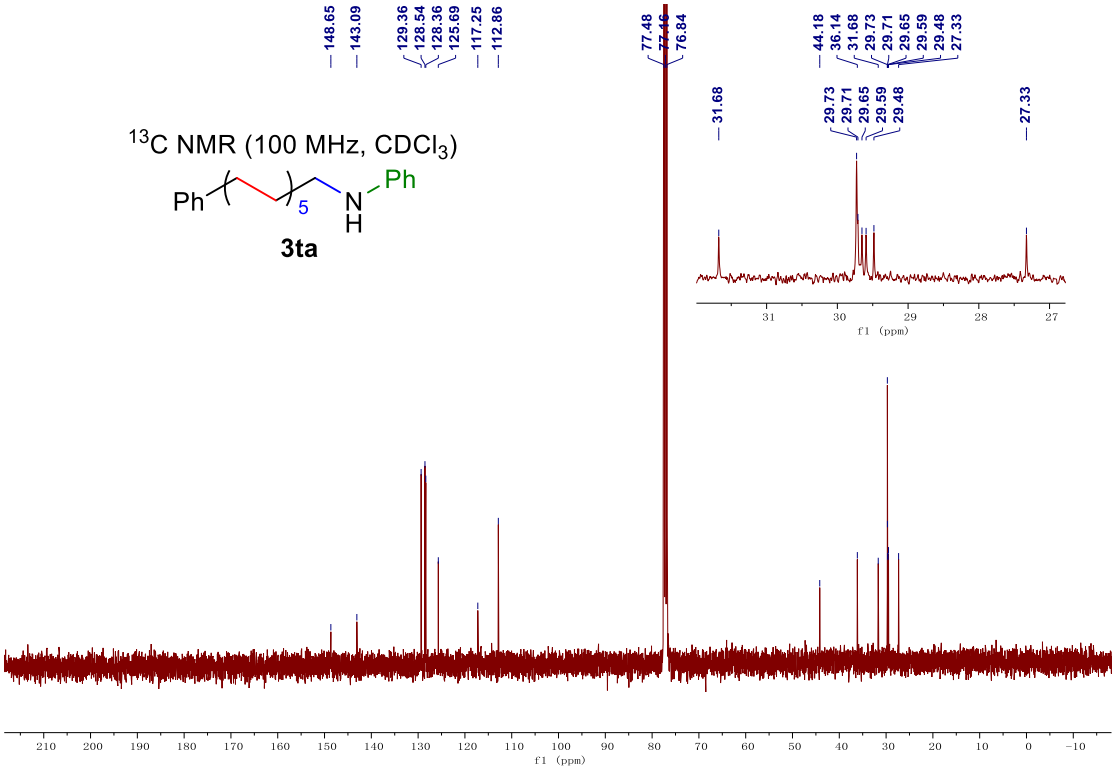

7.32 7.31 7.30 7.30 7.30 7.29 7.28 7.26 7.22 7.21 7.21 7.21 7.20 7.20 7.19 7.19 7.18 7.17 7.17 6.73 6.73 6.72 6.71 6.71 6.70 6.69 6.69 6.69 6.63 6.63 6.63 6.62 6.61 6.61 6.61 6.61 6.60
3.57 3.13 3.11 3.09 2.65 2.63 2.61 1.69 1.67 1.66 1.65 1.63 1.61 1.45 1.44 1.43 1.42 1.42 1.41 1.41 1.39 1.38 1.37 1.36
1H NMR (400 MHz, CDCl3)
Ph
3
N
H
Ph
3ra
2.05 5.04 1.00 2.04 0.96 2.11 2.09 4.16 6.26
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)

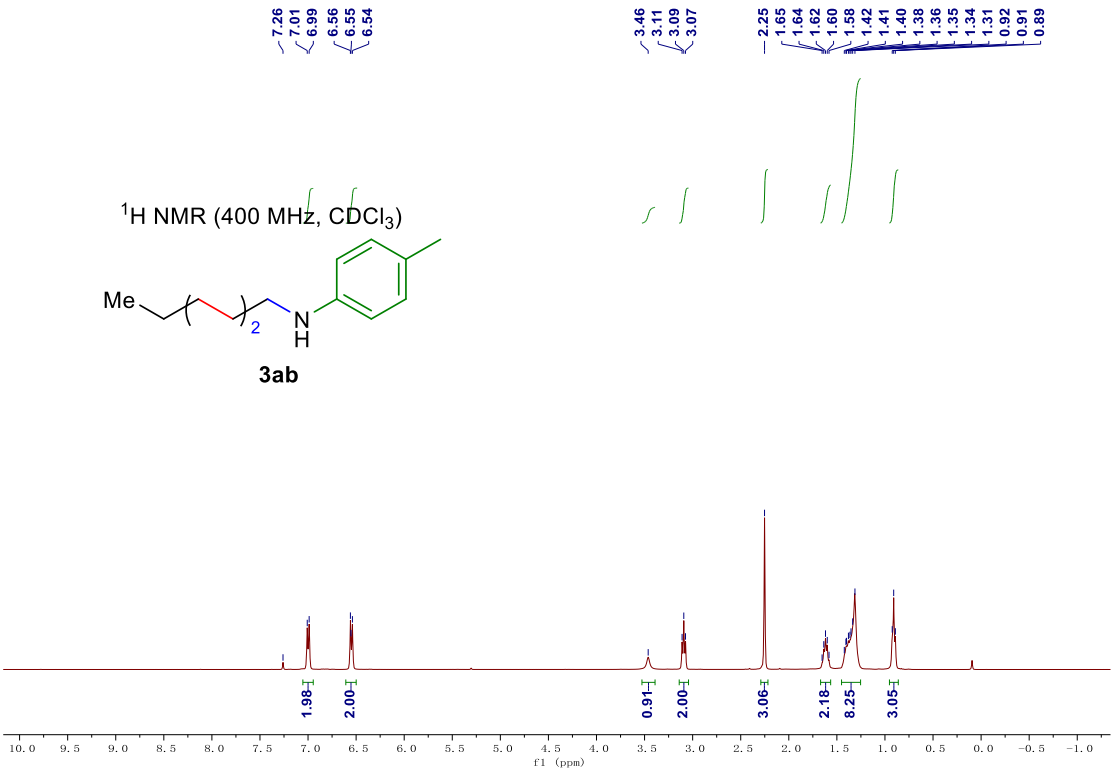

148.64 142.92 129.34 128.52 128.37 125.73 117.21 112.81
77.48 77.16 76.84
44.10 36.08 31.56 29.69 29.44 29.37 27.23
13C NMR (100 MHz, CDCl3)
Ph
3
N
H
Ph
3ra
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

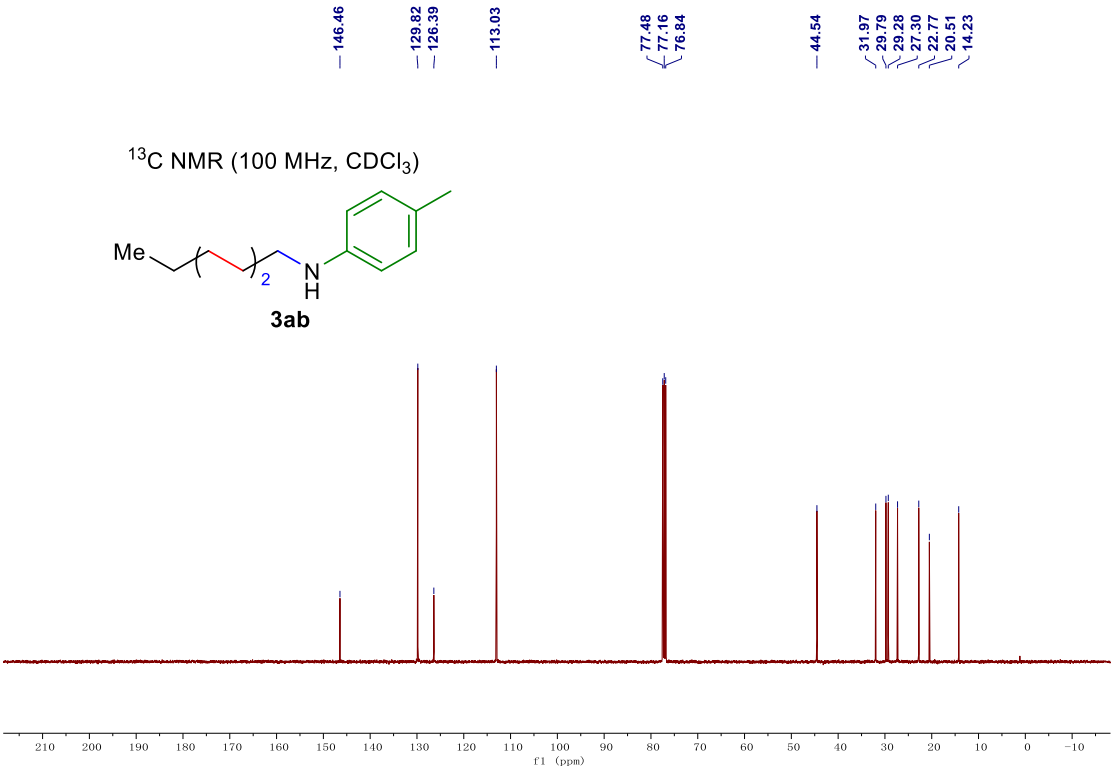

7.31 7.30 7.29 7.29 7.29 7.28 7.27 7.26 7.20 7.18 7.18 7.17 7.16 7.16 6.72 6.72 6.71 6.70 6.70 6.69 6.68 6.68 6.68 6.63 6.62 6.62 6.62 6.61 6.60 6.60 6.59 3.59 3.12 3.11 3.09 2.63 2.62 2.60 1.66 1.66 1.65 1.64 1.62 1.61 1.60 1.59 1.58 1.42 1.40 1.39 1.38 1.37 1.35 1.34 1.33 1.31
1H NMR (400 MHz, CDCl3)
Ph
4
N
H
Ph
3sa
2.03 5.01 1.00 2.01 0.95 2.03 2.04 4.04 10.26
f1 (ppm)


148.67 143.03 129.35 128.53 128.36 125.69 117.20 112.81 77.48 77.16 76.84 44.13 36.12 31.64 29.72 29.65 29.59 29.57 29.44 27.31
29.72 29.65 29.59 29.57 29.44
13C NMR (100 MHz, CDCl3)
Ph
4
N
H
Ph
3sa
f1 (ppm)


7.29 7.29 7.28 7.27 7.27 7.26 7.25 7.19 7.19 7.18 7.18 7.17 7.17 7.15 7.15 7.14 6.70 6.70 6.70 6.69 6.68 6.68 6.67 6.66 6.66 6.61 6.61 6.61 6.60 6.60 6.59 6.59
3.70
3.11 3.10 3.08 2.62 2.60 2.58 1.64 1.63 1.63 1.61 1.59 1.42 1.40 1.39 1.37 1.30 1.27
1H NMR (400 MHz, CDCl3)
Ph
5
N
H
Ph
3ta
1.34 0.73 5.10 1.03 2.02 0.96 2.00 2.06 5.11 13.62
f1 (ppm)


148.65 143.09 129.36 128.54 128.36 125.69 117.25 112.86
77.48 77.16 76.84
44.18 36.14 31.68 29.73 29.71 29.65 29.59 29.48 27.33
31.68
29.73 29.71 29.65 29.59 29.48
27.33
13C NMR (100 MHz, CDCl3)
Ph
5
N
H
Ph
3ta
f1 (ppm)


7.26
7.01
6.99
6.56
6.55
6.54
3.46
3.11
3.09
3.07
2.25
1.65
1.64
1.62
1.60
1.58
1.42
1.41
1.40
1.38
1.36
1.35
1.34
1.31
0.92
0.91
0.89
1H NMR (400 MHz, CDCl3)
Me
2
N
H
3ab
1.98
2.00
0.91
2.00
3.06
2.18
8.25
3.05
f1 (ppm)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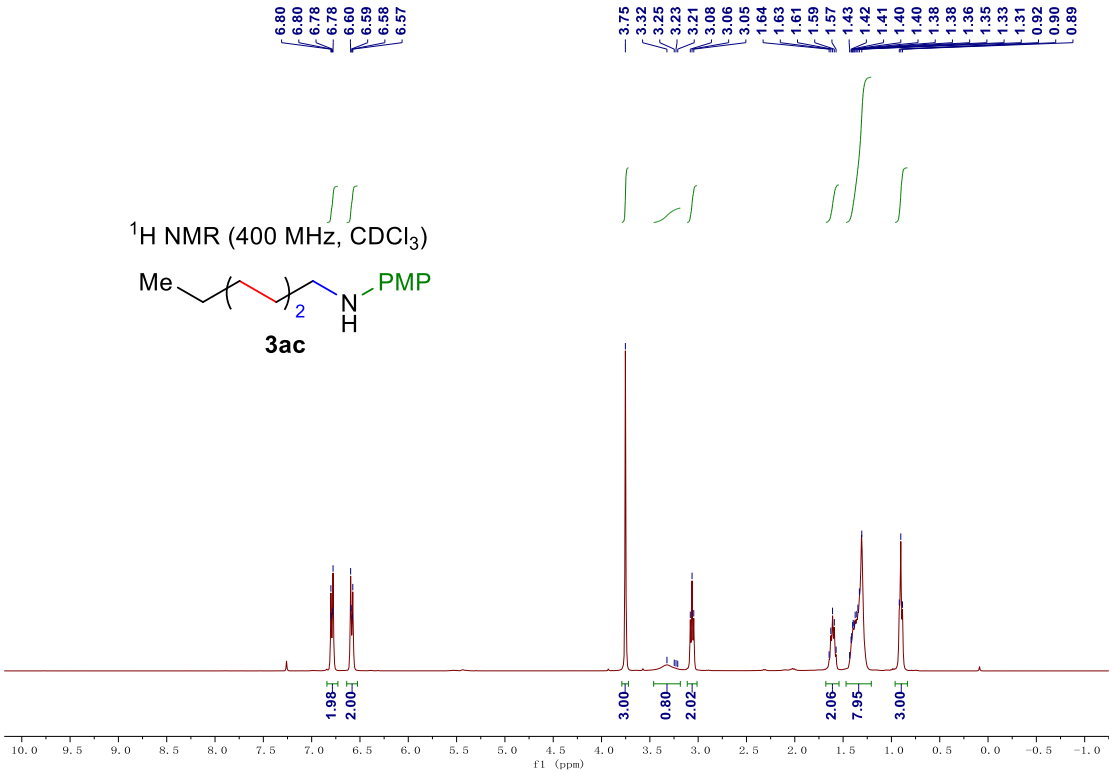
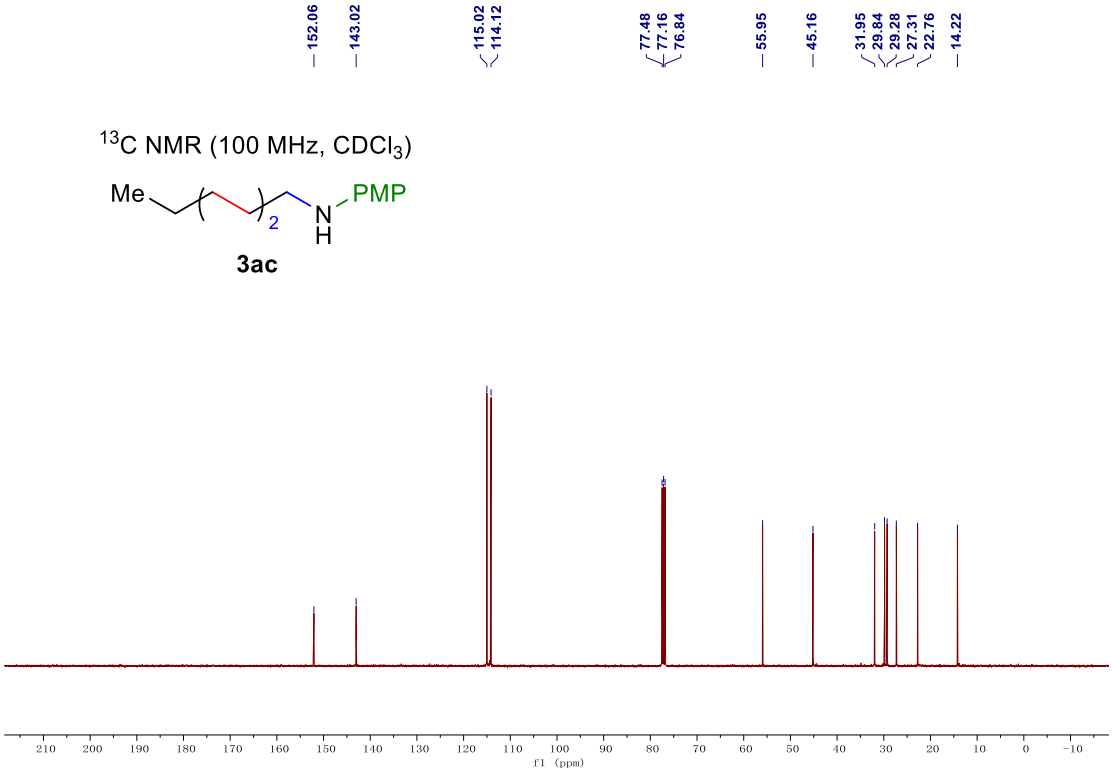
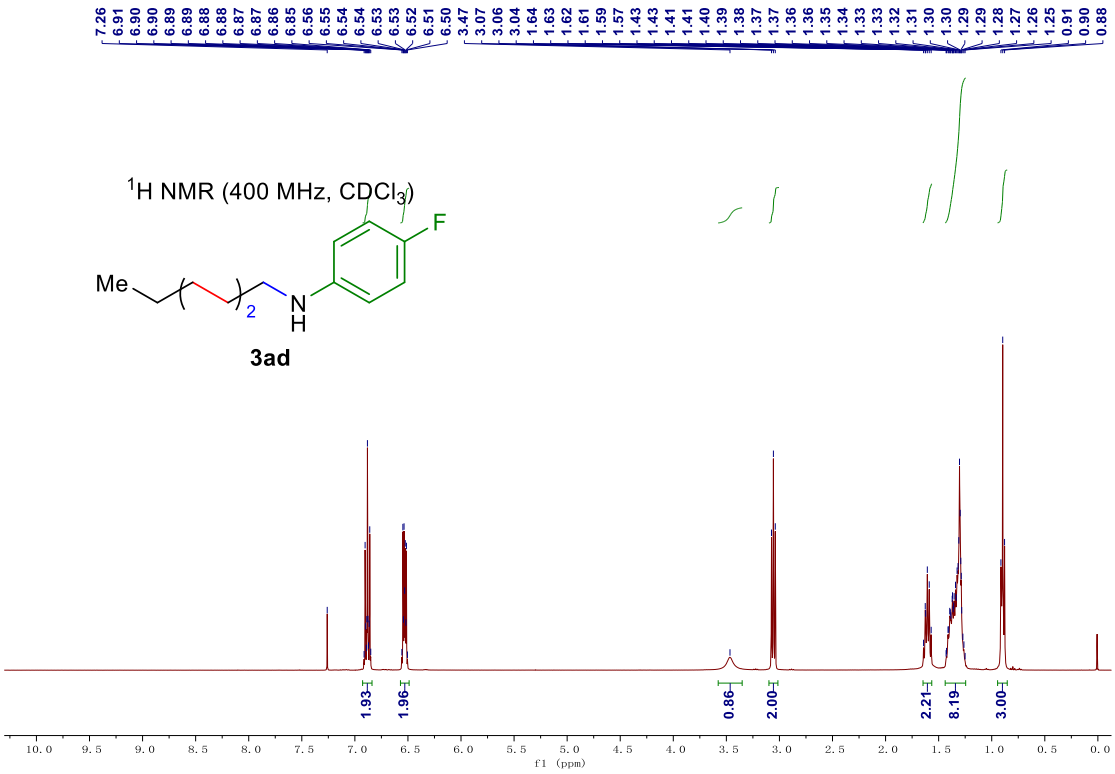
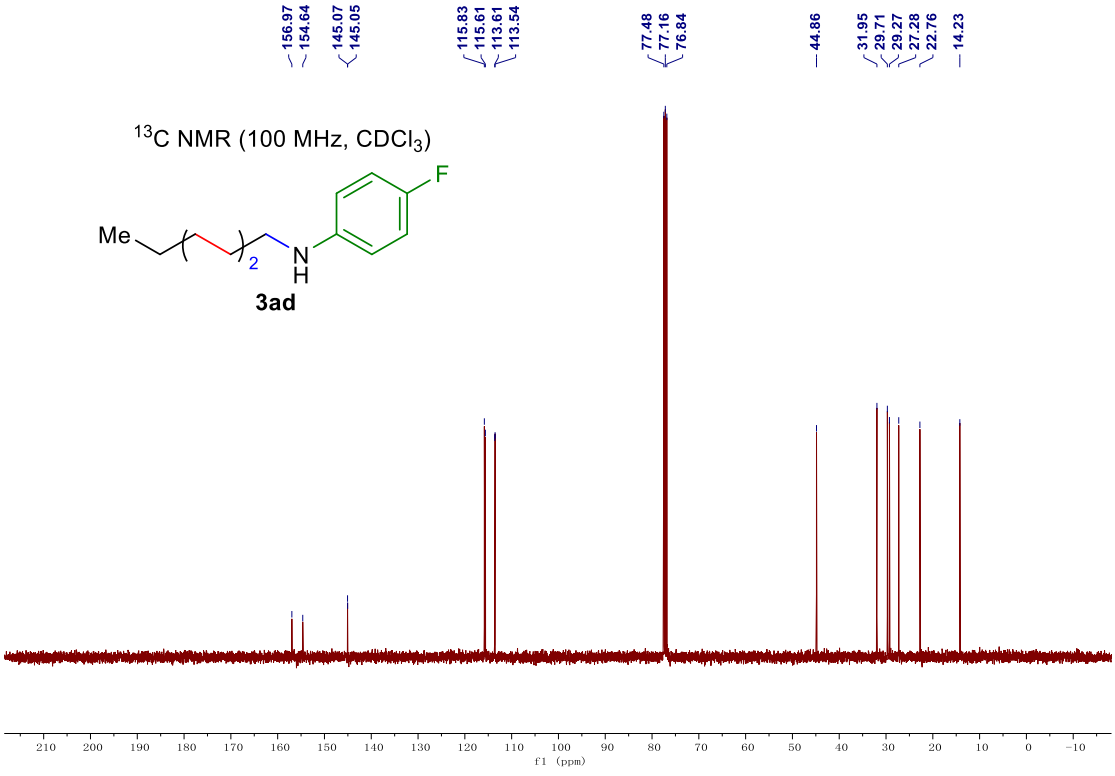
129.82
126.39
113.03
77.48
77.16
76.84
44.54
31.97
29.79
29.28
27.30
22.77
20.51
14.23
13C NMR (100 MHz, CDCl3)
Me
2
N
H
3ab
f1 (ppm)

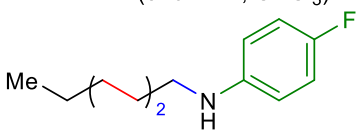

6.80
6.80
6.78
6.78
6.60
6.59
6.58
6.57
3.75
3.32
3.25
3.23
3.21
3.08
3.06
3.05
1.64
1.63
1.61
1.59
1.57
1.43
1.42
1.41
1.40
1.40
1.38
1.38
1.36
1.35
1.33
1.31
0.92
0.90
0.89
1H NMR (400 MHz, CDCl3)
Me
2
N
H
PMP
3ac
1.98
2.00
3.00
0.80
2.02
2.06
7.95
3.00
f1 (ppm)


152.06
143.02
115.02
114.12
77.48
77.16
76.84
55.95
45.16
31.95
29.84
29.28
27.31
22.76
14.22
13C NMR (100 MHz, CDCl3)
Me
2
N
H
PMP
3ac
f1 (ppm)


7.26 6.91 6.90 6.90 6.89 6.89 6.88 6.88 6.87 6.87 6.86 6.85 6.56 6.55 6.54 6.54 6.53 6.53 6.52 6.51 6.50 3.47 3.07 3.06 3.04 1.64 1.63 1.62 1.61 1.59 1.57 1.43 1.43 1.41 1.41 1.40 1.39 1.38 1.37 1.37 1.36 1.36 1.35 1.34 1.33 1.33 1.32 1.31 1.30 1.30 1.29 1.29 1.28 1.27 1.26 1.25 0.91 0.90 0.88
1H NMR (400 MHz, CDCl3)
F
Me
2
N
H
3ad
1.93 1.96 0.86 2.00 2.21 8.19 3.00
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
f1 (ppm)

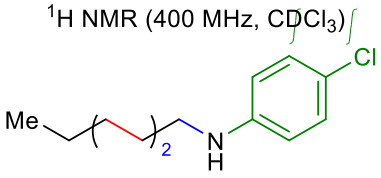

156.97 154.64 145.07 145.05 115.83 115.61 113.61 113.54 77.48 77.16 76.84 44.86 31.95 29.71 29.27 27.28 22.76 14.23
13C NMR (100 MHz, CDCl3)
F
Me
2
N
H
3ad
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

^{19}F NMR (376 MHz, $CDCl_3$)

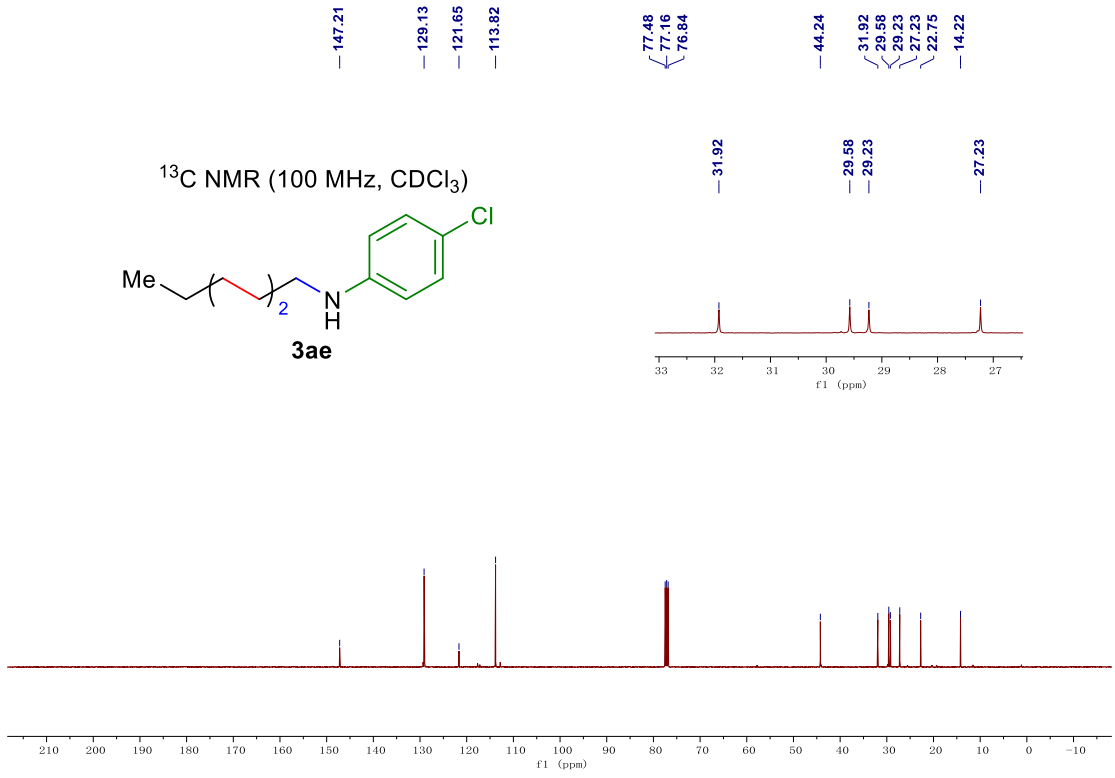


3ad

^{1}H NMR (400 MHz, $CDCl_3$)



3ae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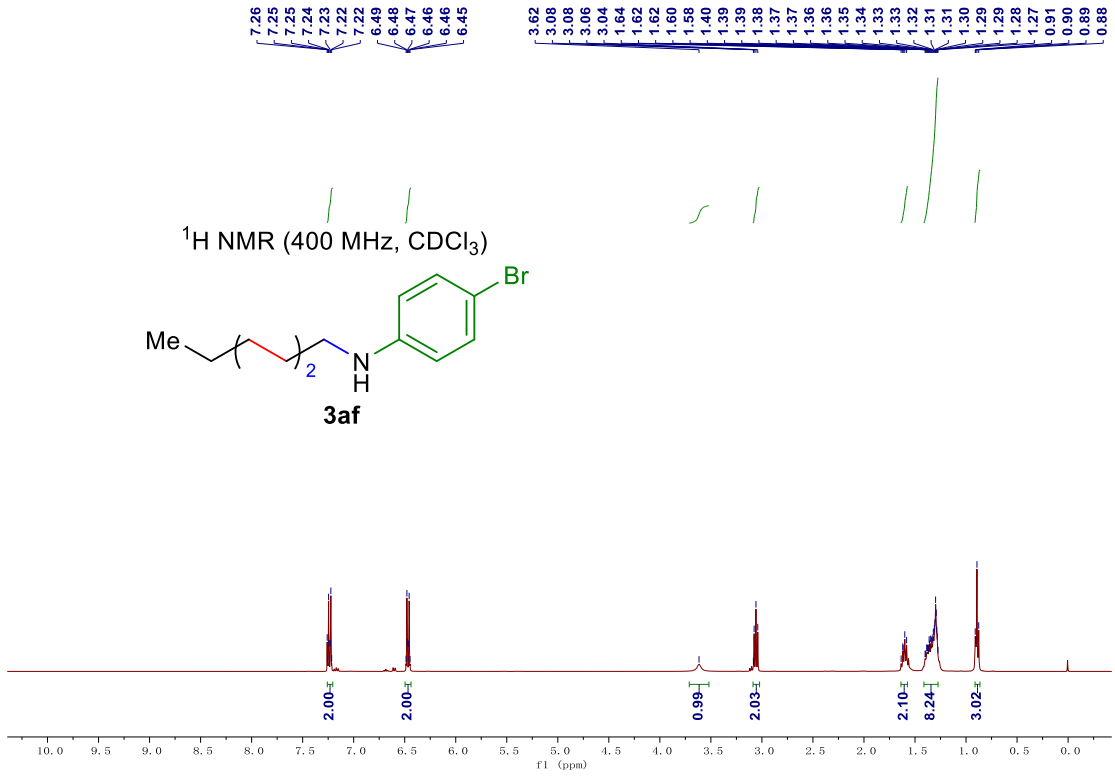
113.82
77.48
77.16
76.84
44.24
31.92
29.58
29.23
27.23
22.75
14.22
13C NMR (100 MHz, CDCl3)
Cl
Me
2
N
H
3ae
31.92
29.58
29.23
27.23
33 32 31 30 29 28 27
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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7.25
7.25
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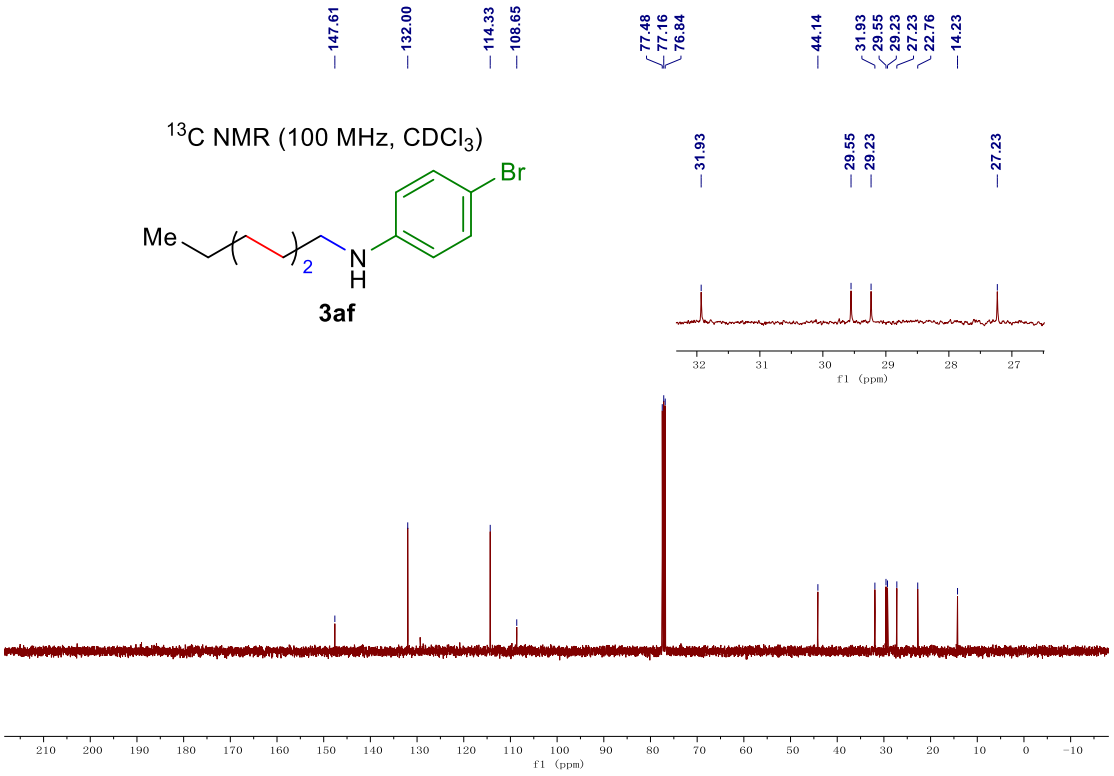
7.22
7.22
6.49
6.48
6.47
6.46
6.46
6.45
3.62
3.08
3.08
3.06
3.04
1.64
1.62
1.62
1.60
1.58
1.40
1.39
1.39
1.38
1.37
1.37
1.36
1.36
1.35
1.34
1.33
1.33
1.32
1.31
1.31
1.30
1.29
1.29
1.28
1.27
0.91
0.90
0.89
0.88
1H NMR (400 MHz, CDCl3)
Br
Me
2
N
H
3af
2.00
2.00
0.99
2.03
2.10
8.24
3.02
10.0 9.5 9.0 8.5 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
f1 (ppm)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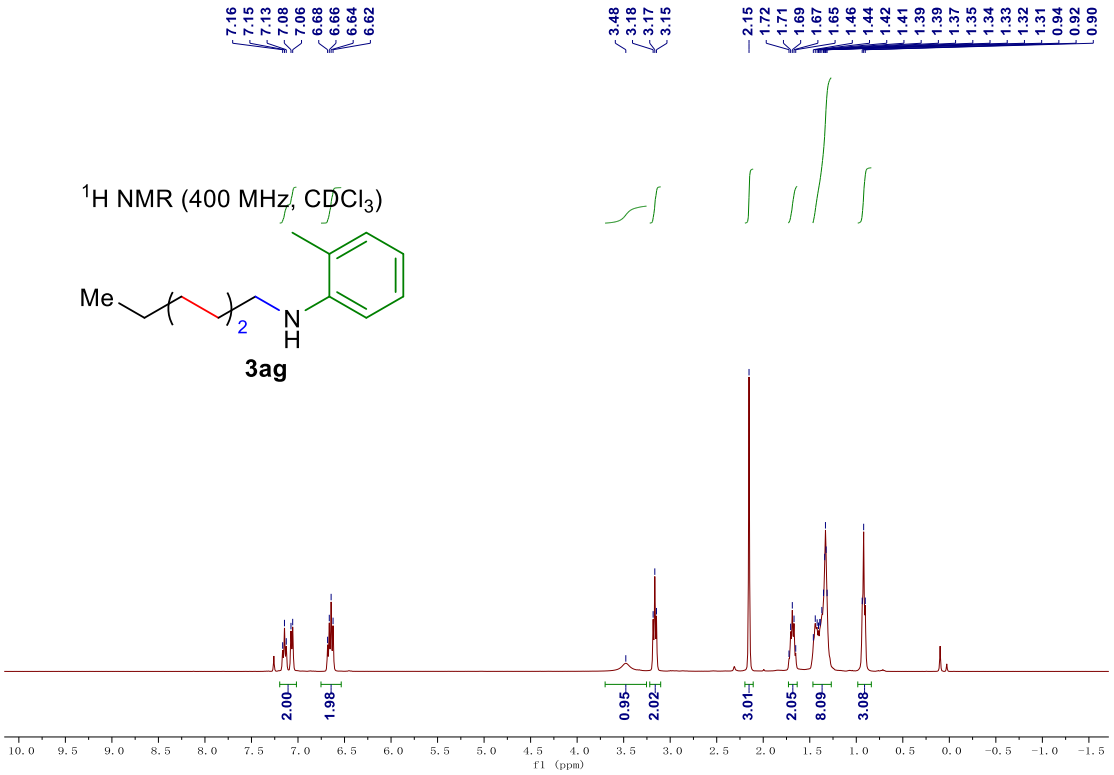
114.33
108.65
77.48
77.16
76.84
44.14
31.93
29.55
29.23
27.23
22.76
14.23
13C NMR (100 MHz, CDCl3)
Br
Me
2
N
H
3af
31.93
29.55
29.23
27.23
f1 (ppm)
f1 (ppm)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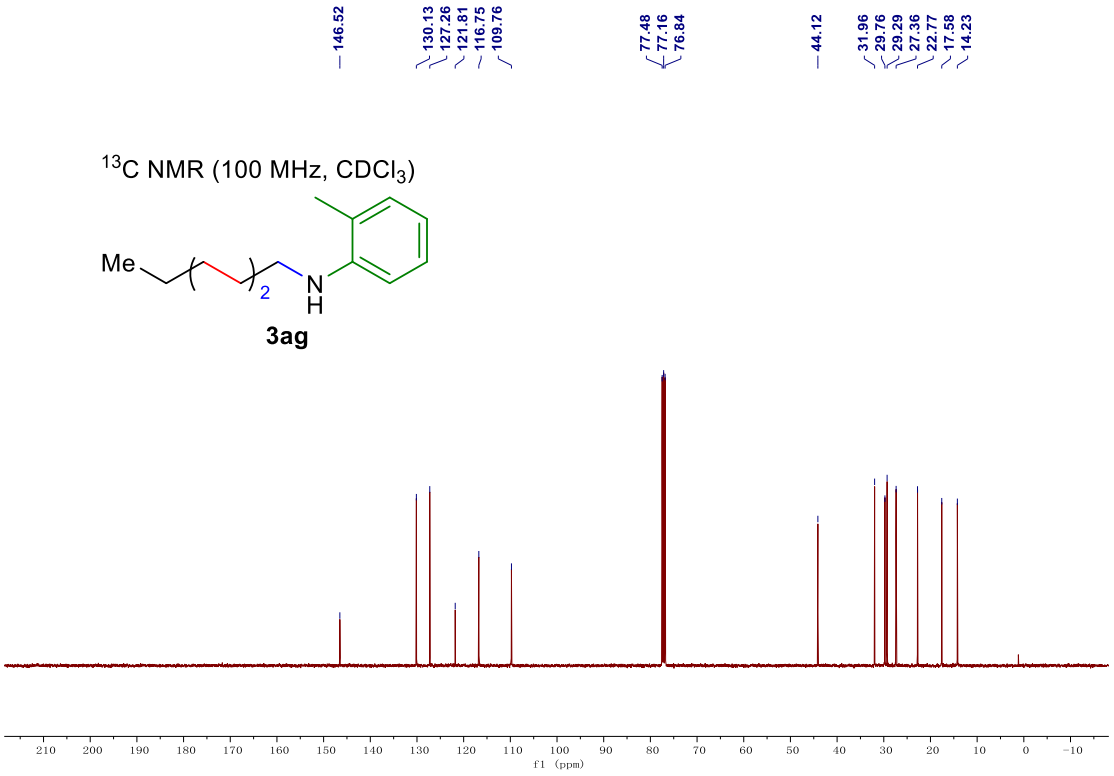
7.15
7.13
7.08
7.06
6.68
6.66
6.64
6.62
3.48
3.18
3.17
3.15
2.15
1.72
1.71
1.69
1.67
1.65
1.46
1.44
1.42
1.41
1.39
1.39
1.37
1.35
1.34
1.33
1.32
1.31
0.94
0.92
0.90
1H NMR (400 MHz, CDCl3)
Me
2
N
H
3ag
2.00
1.98
0.95
2.02
3.01
2.05
8.09
3.08
f1 (ppm)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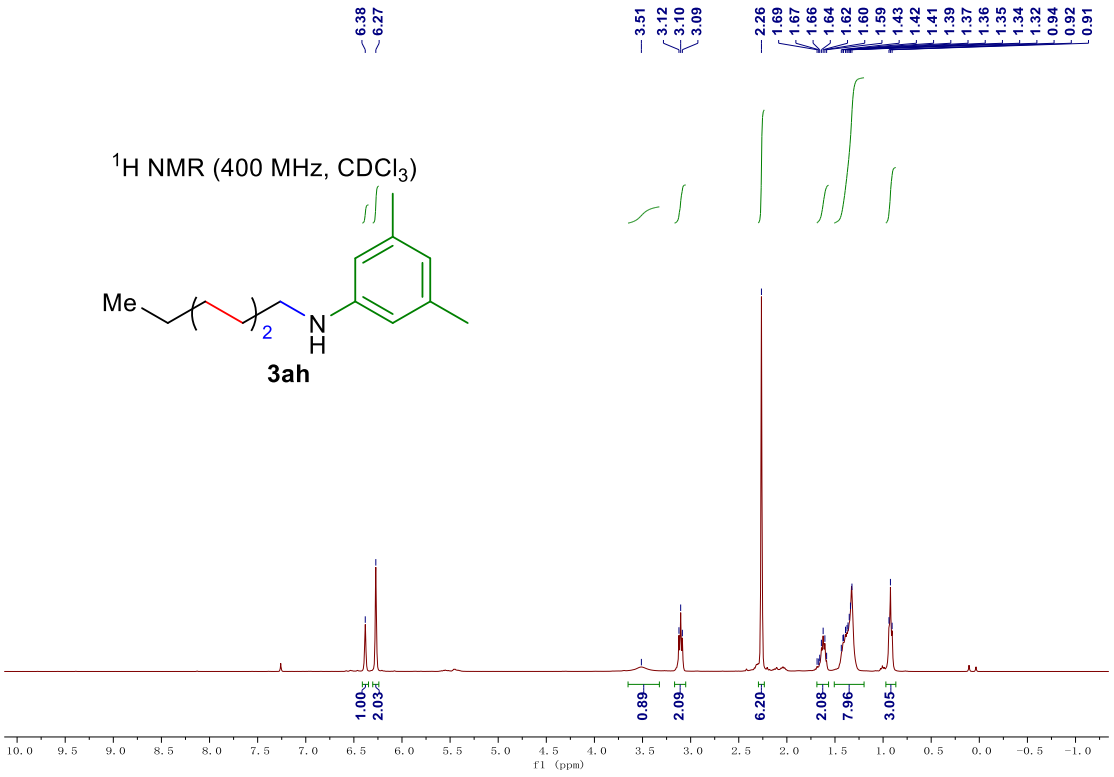
121.81
116.75
109.76
77.48
77.16
76.84
44.12
31.96
29.76
29.29
27.36
22.77
17.58
14.23
13C NMR (100 MHz, CDCl3)
Me
2
N
H
3ag
f1 (ppm)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6.27
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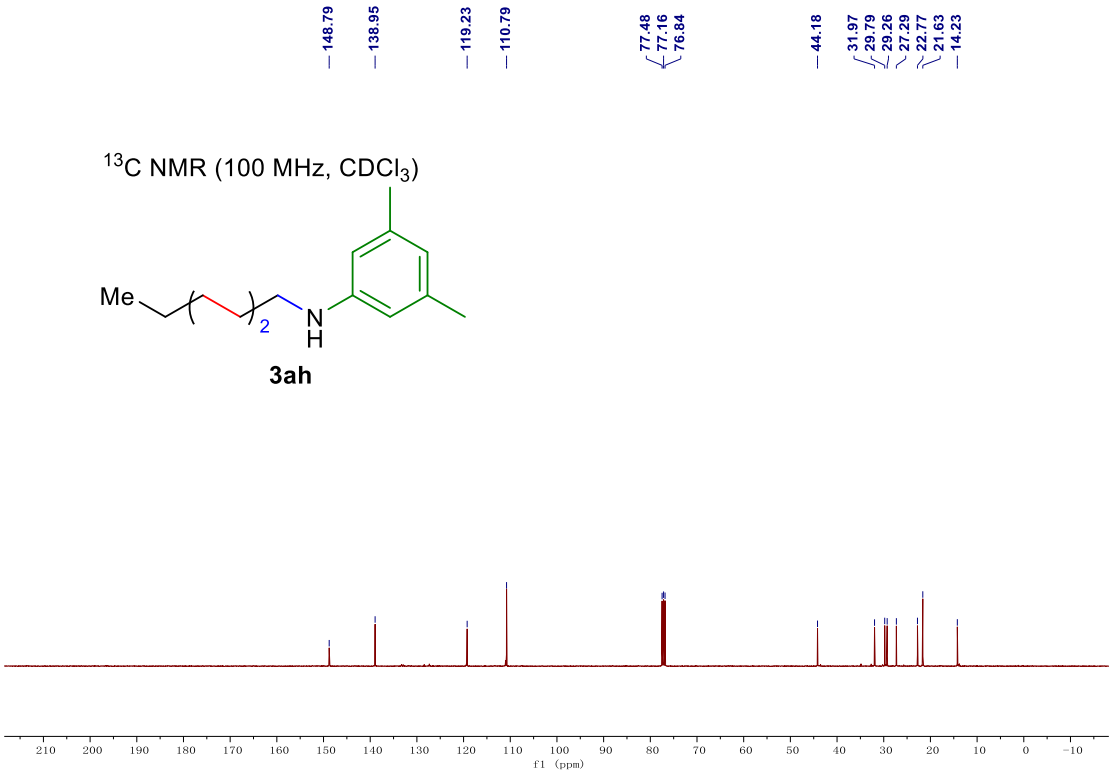
3.10
3.09
2.26
1.69
1.67
1.66
1.64
1.62
1.60
1.59
1.43
1.42
1.41
1.39
1.37
1.36
1.35
1.34
1.32
0.94
0.92
0.91
1H NMR (400 MHz, CDCl3)
Me
2
N
H
3ah
1.00
2.03
0.89
2.09
6.20
2.08
7.96
3.05
f1 (ppm)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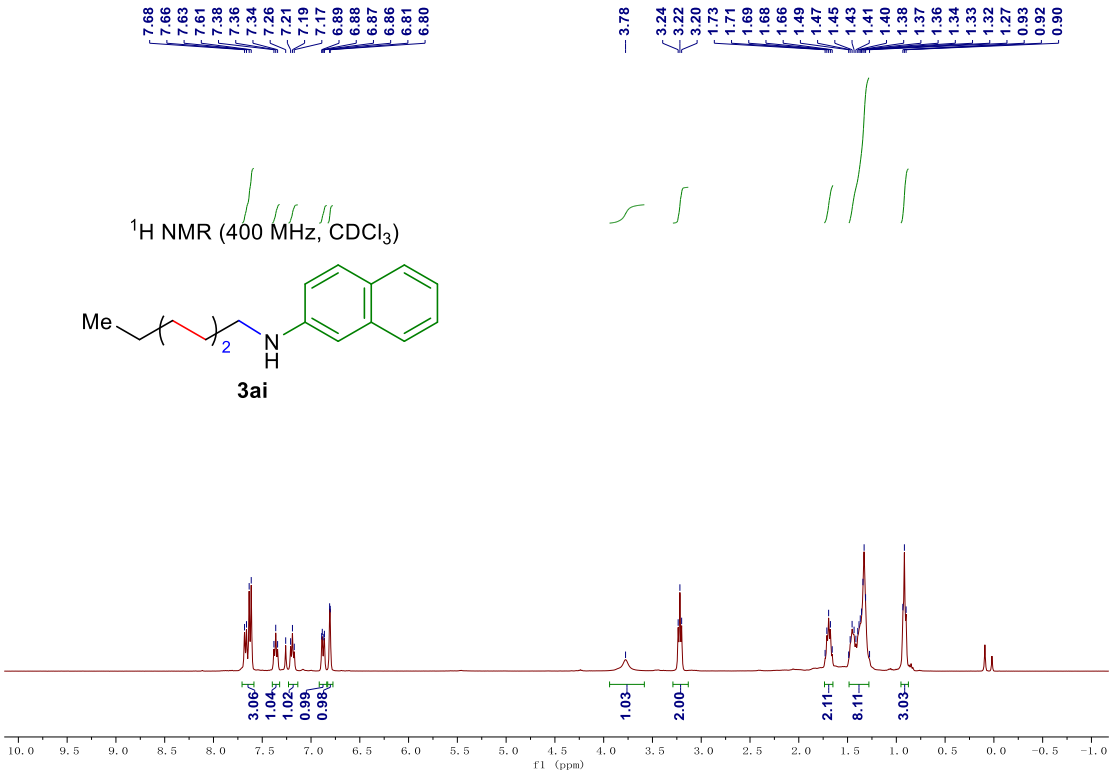
110.79
77.48
77.16
76.84
44.18
31.97
29.79
29.26
27.29
22.77
21.63
14.23
13C NMR (100 MHz, CDCl3)
Me
2
N
H
3ah
f1 (ppm)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7.61
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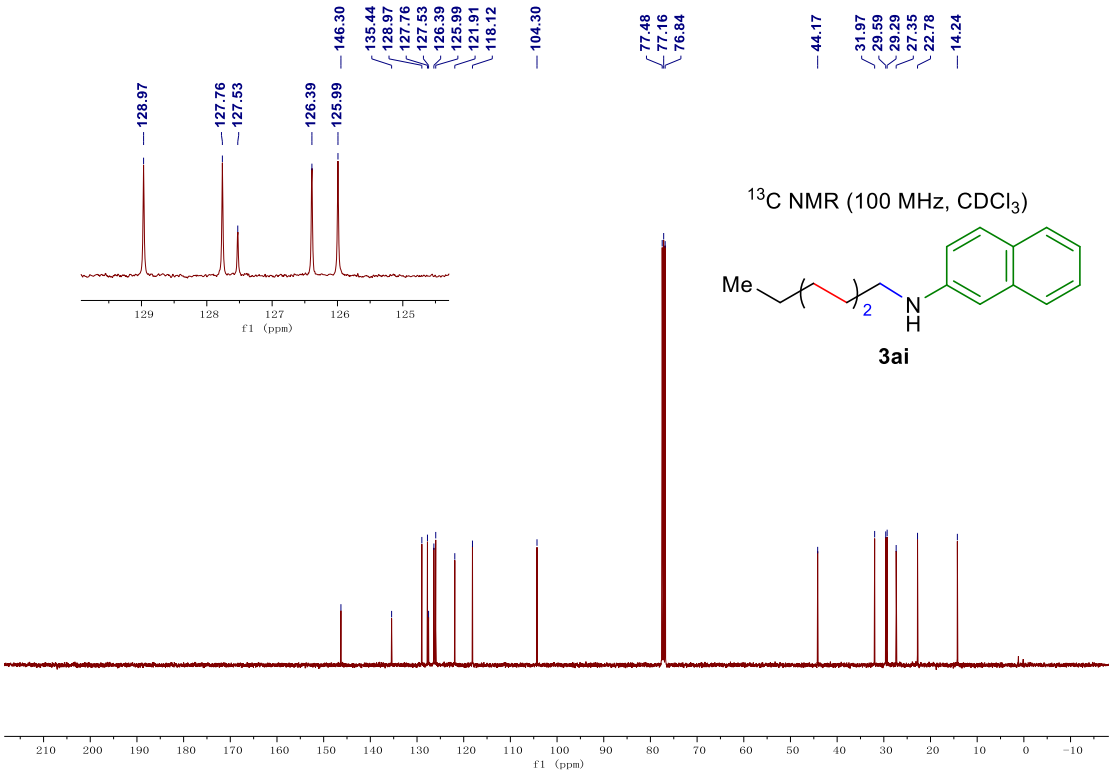
7.34
7.26
7.21
7.19
7.17
6.89
6.88
6.87
6.86
6.81
6.80
3.78
3.24
3.22
3.20
1.73
1.71
1.69
1.68
1.66
1.49
1.47
1.45
1.43
1.41
1.40
1.38
1.37
1.36
1.34
1.33
1.32
1.27
0.93
0.92
0.90
1H NMR (400 MHz, CDCl3)
Me
2
N
H
3ai
3.06
1.04
1.02
0.99
0.98
1.03
2.00
2.11
8.11
3.03
f1 (ppm)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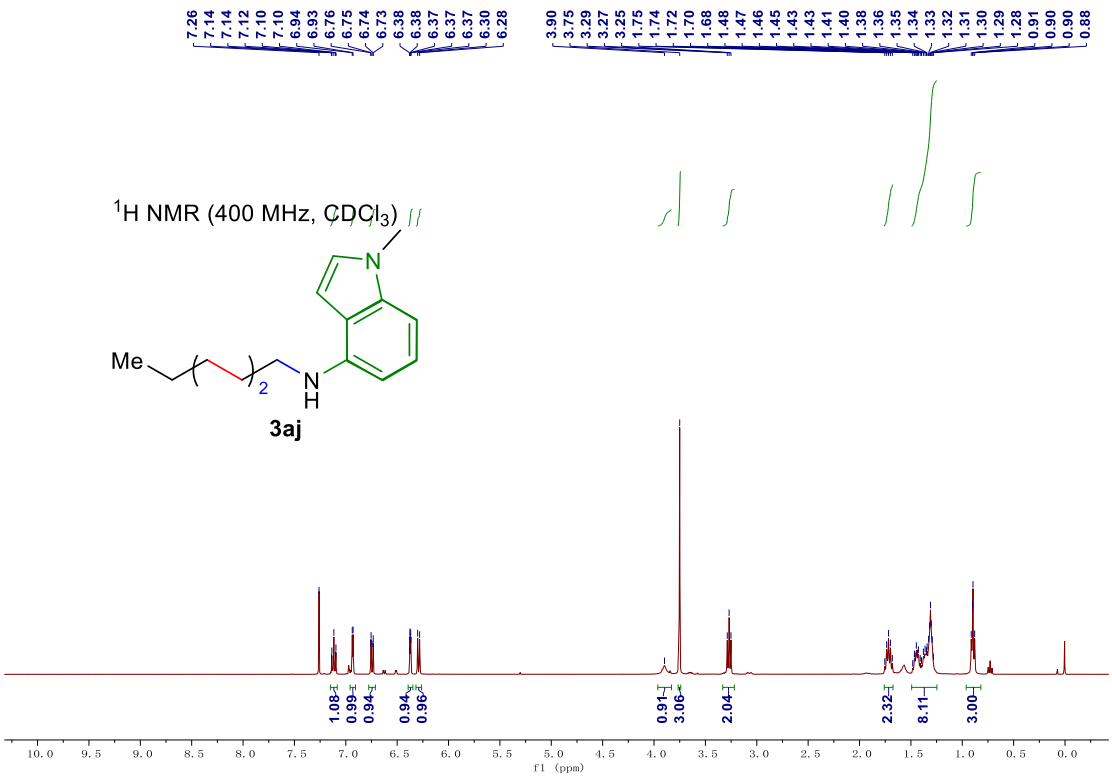
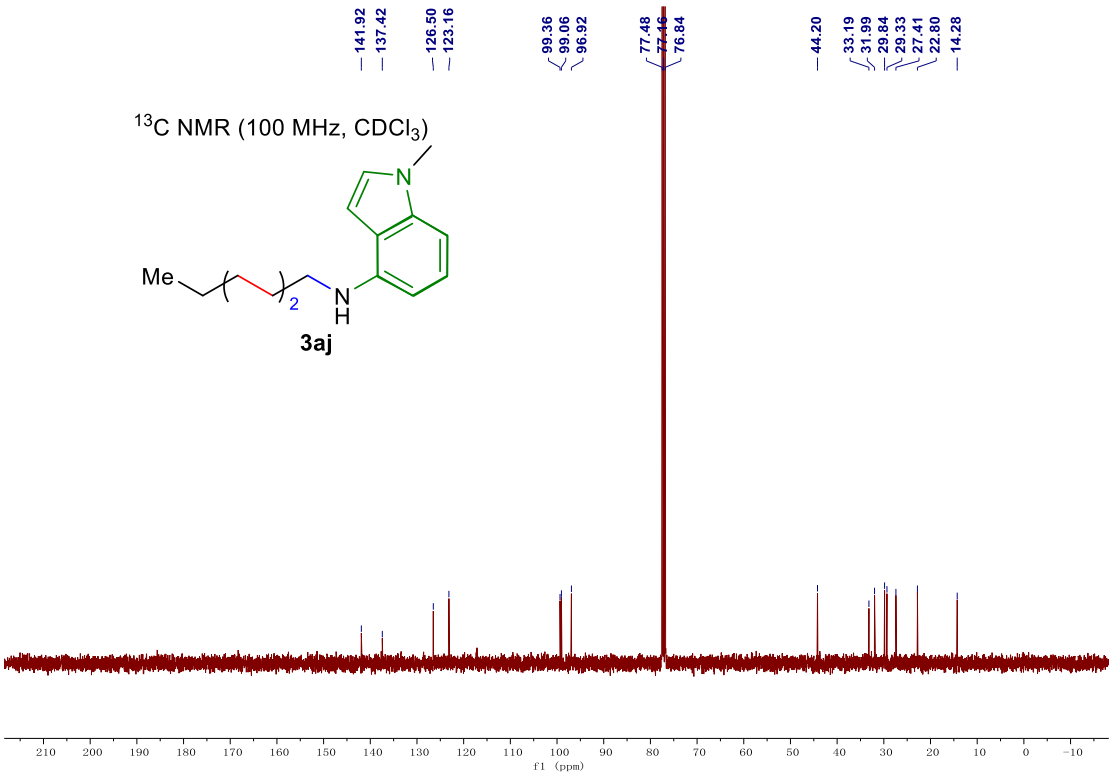
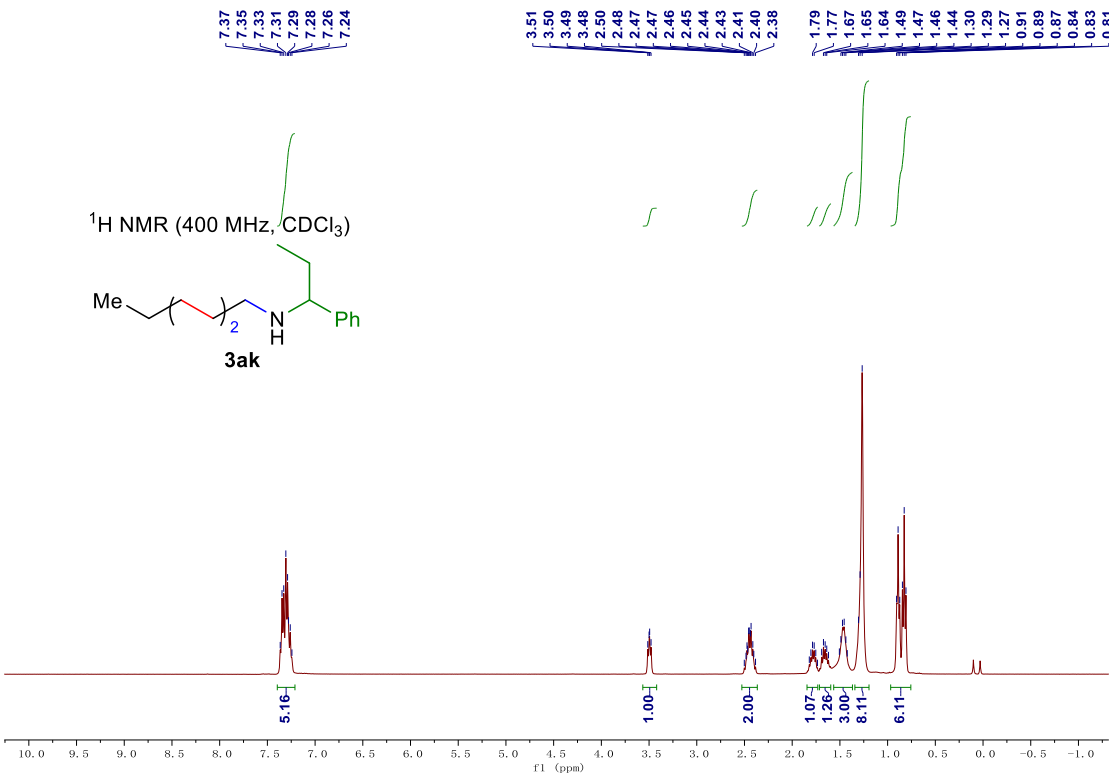
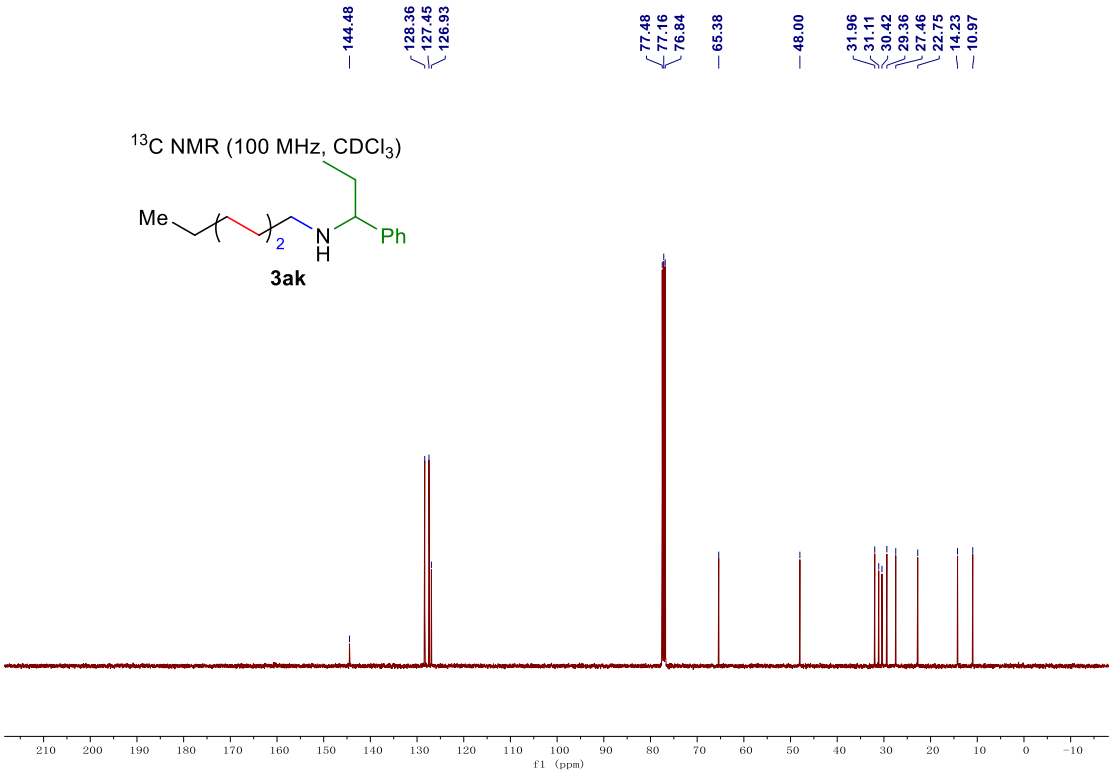
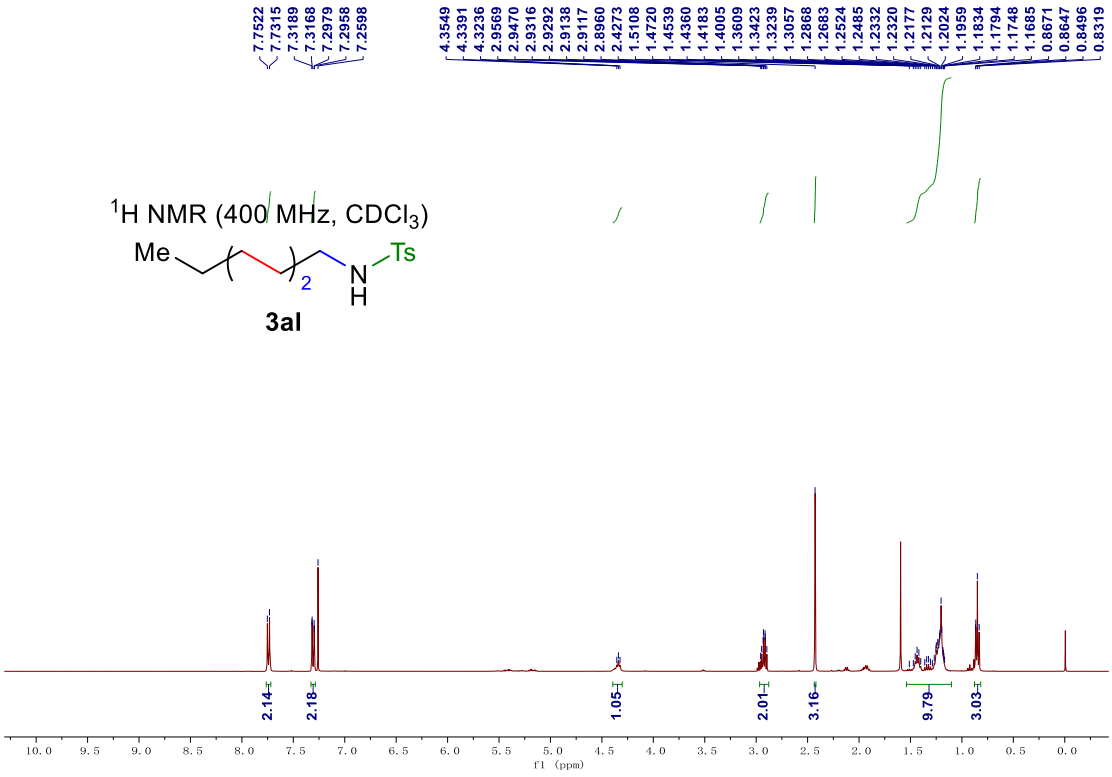
127.76
127.53
126.39
125.99
121.91
118.12
104.30
77.48
77.16
76.84
44.17
31.97
29.59
29.29
27.35
22.78
14.24
128.97
127.76
127.53
126.39
125.99
129
128
127
126
125
f1 (ppm)
13C NMR (100 MHz, CDCl3)
Me
2
N
H
3ai
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

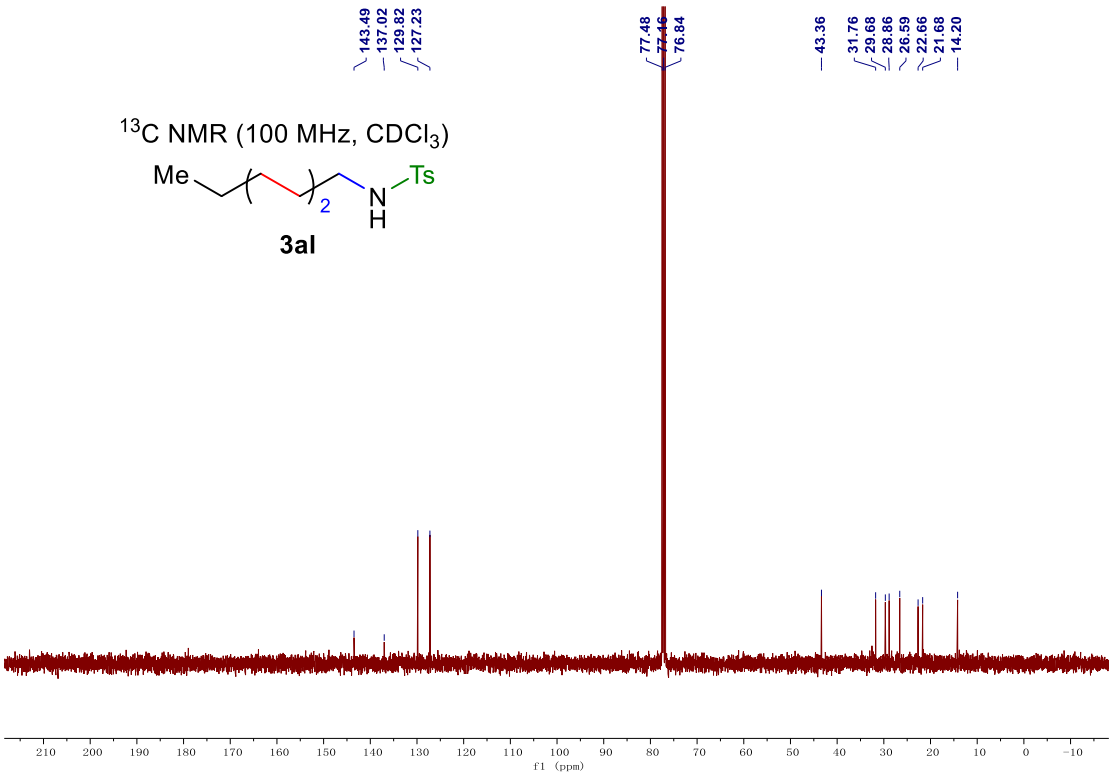

7.26
7.14
7.14
7.12
7.10
7.10
6.94
6.93
6.76
6.75
6.74
6.73
6.38
6.38
6.37
6.37
6.37
6.30
6.28
3.90
3.75
3.29
3.27
3.25
1.75
1.74
1.72
1.70
1.68
1.48
1.47
1.46
1.45
1.43
1.43
1.41
1.40
1.38
1.36
1.35
1.34
1.33
1.32
1.31
1.30
1.29
1.28
0.91
0.90
0.90
0.88
1H NMR (400 MHz, CDCl3)
N
Me
2
N
H
3aj
1.08
0.99
0.94
0.94
0.96
0.91
3.06
2.04
2.32
8.11
3.00
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
f1 (ppm)


141.92
137.42
126.50
123.16
99.36
99.06
96.92
77.48
77.16
76.84
44.20
33.19
31.99
29.84
29.33
27.41
22.80
14.28
13C NMR (100 MHz, CDCl3)
Me
2
N
H
3aj
f1 (ppm)


7.37
7.35
7.33
7.31
7.29
7.28
7.26
7.24
3.51
3.50
3.49
3.48
2.50
2.48
2.47
2.47
2.46
2.45
2.44
2.43
2.41
2.40
2.38
1.79
1.77
1.67
1.65
1.64
1.49
1.47
1.46
1.44
1.30
1.29
1.27
0.91
0.89
0.87
0.84
0.83
1H NMR (400 MHz, CDCl3)
Me
2
N
H
Ph
3ak
5.16
1.00
2.00
1.07
1.26
3.00
8.11
6.11
f1 (ppm)

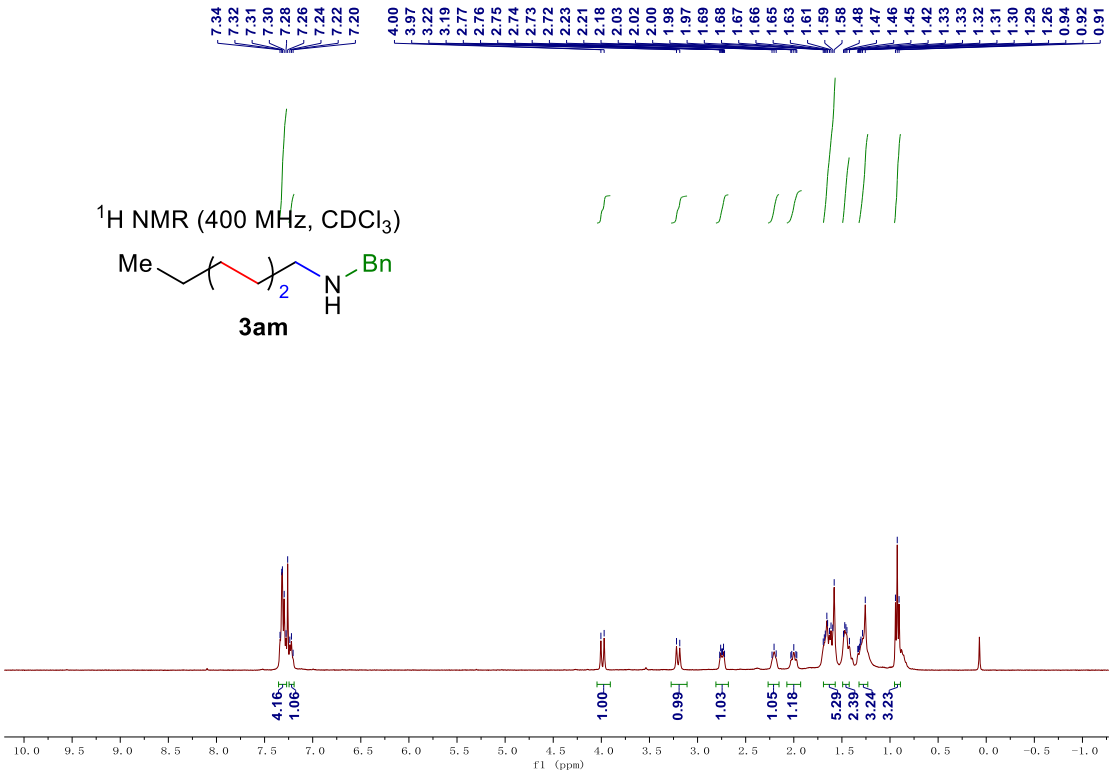

144.48
128.36
127.45
126.93
77.48
77.16
76.84
65.38
48.00
31.96
31.11
30.42
29.36
27.46
22.75
14.23
10.97
13C NMR (100 MHz, CDCl3)
Me
2
N
H
Ph
3ak
f1 (ppm)


7.7522
7.7315
7.3189
7.3168
7.2979
7.2958
7.2598
4.3549
4.3391
4.3236
2.9569
2.9470
2.9316
2.9292
2.9138
2.9117
2.8960
2.4273
1.5108
1.4720
1.4539
1.4360
1.4183
1.4005
1.3609
1.3423
1.3239
1.3057
1.2868
1.2683
1.2524
1.2485
1.2332
1.2320
1.2177
1.2129
1.2024
1.1959
1.1834
1.1794
1.1748
1.1685
0.8671
0.8647
0.8496
0.8319
1H NMR (400 MHz, CDCl3)
Me
2
N
H
Ts
3al
2.14
2.18
1.05
2.01
3.16
9.79
3.03
f1 (ppm)



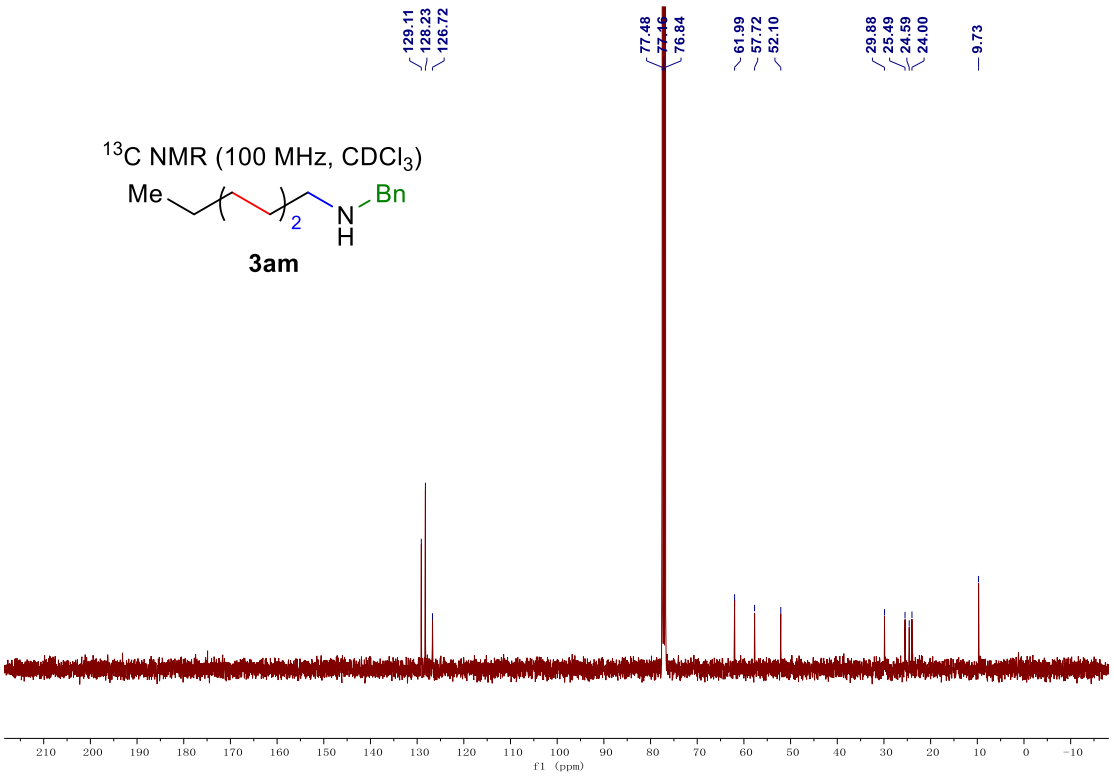
^{13}C NMR (100 MHz, $CDCl_3$)

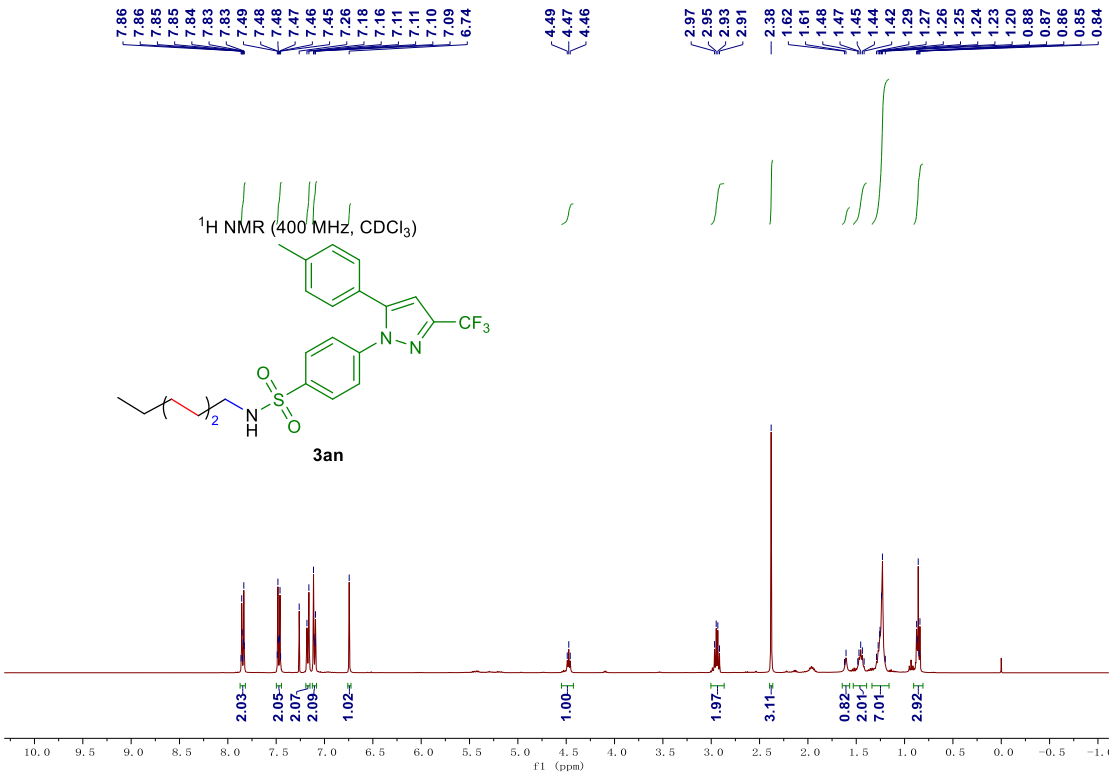
3al

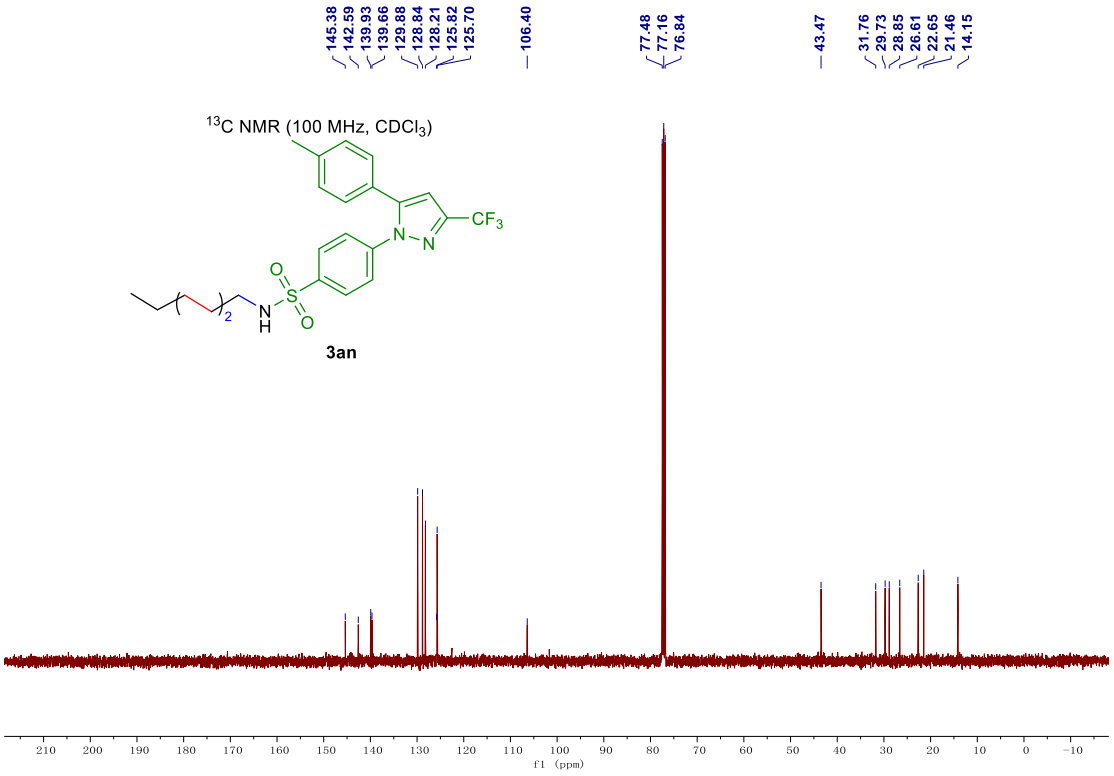


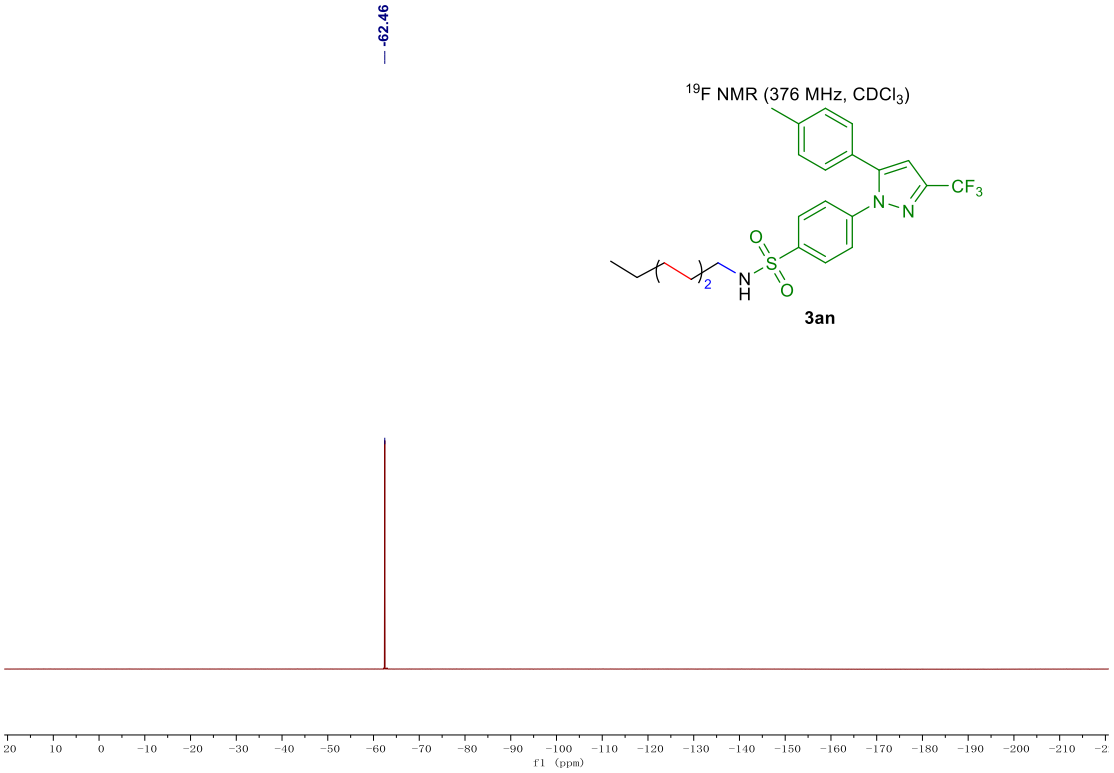
^{1}H NMR (400 MHz, $CDCl_3$)

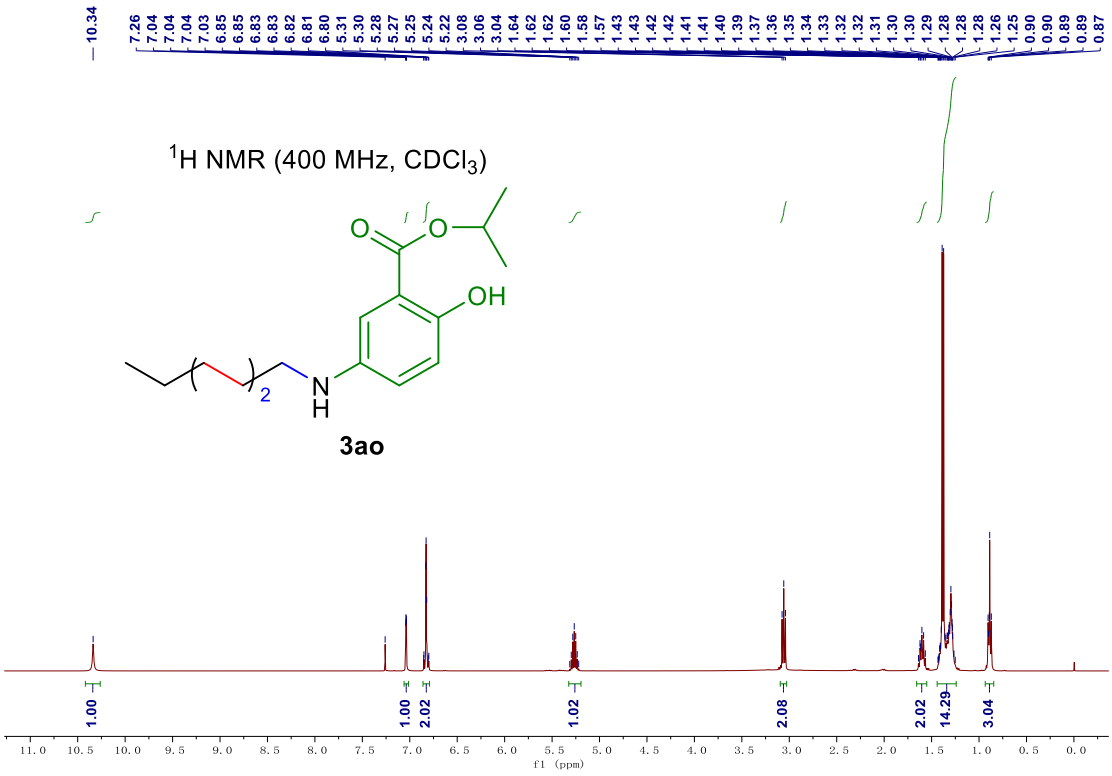
3am

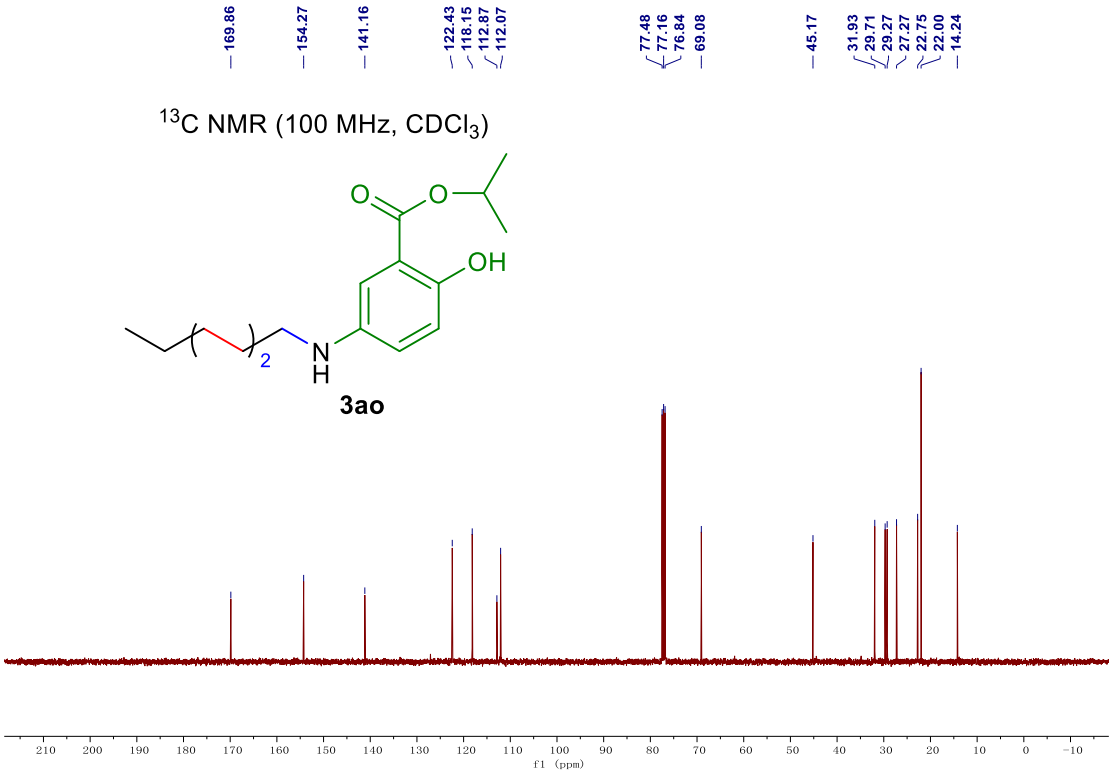

129.11
128.23
126.72
77.48
77.16
76.84
61.99
57.72
52.10
29.88
25.49
24.59
24.00
9.73
13C NMR (100 MHz, CDCl3)
Me
Bn
N
H
2
3am
f1 (ppm)

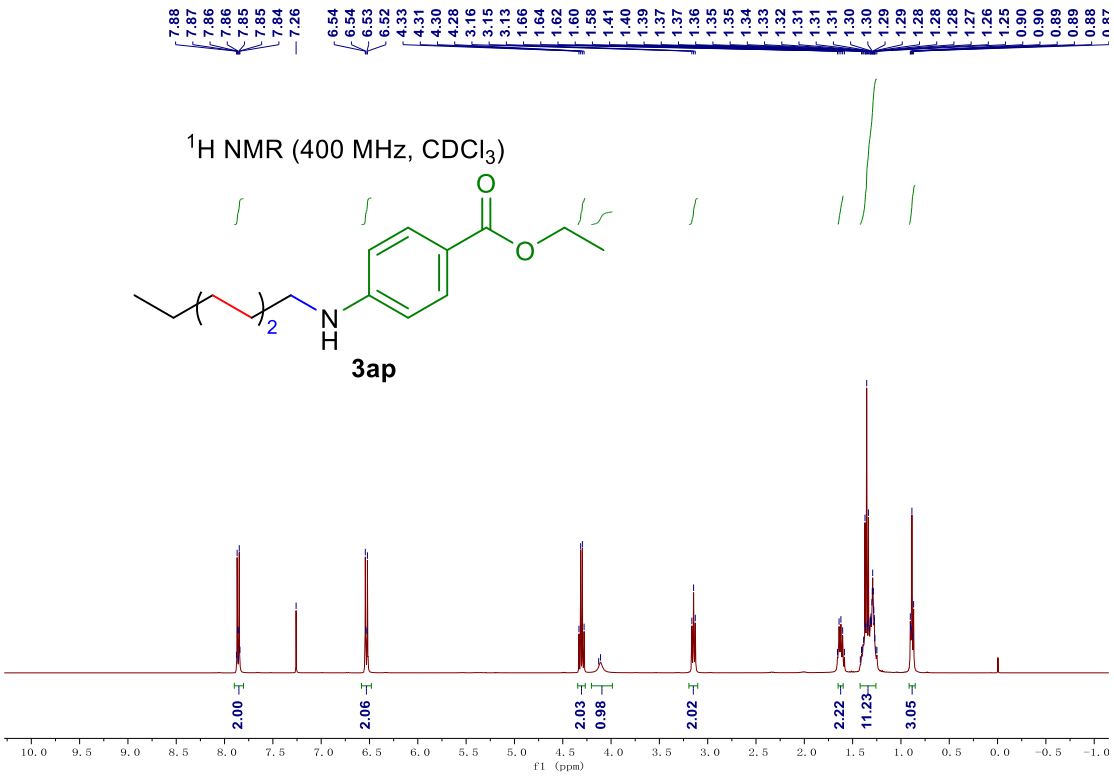

7.86
7.86
7.85
7.85
7.84
7.83
7.83
7.49
7.48
7.48
7.47
7.46
7.45
7.26
7.18
7.16
7.11
7.11
7.10
7.09
6.74
4.49
4.47
4.46
2.97
2.95
2.93
2.91
2.38
1.62
1.61
1.48
1.47
1.45
1.44
1.42
1.29
1.27
1.26
1.25
1.24
1.23
1.20
0.88
0.87
0.86
0.85
0.84
1H NMR (400 MHz, CDCl3)
CF3
N
N
O
S
O
N
H
2
3an
2.03
2.05
2.07
2.09
1.02
1.00
1.97
3.11
0.82
2.01
7.01
2.92
f1 (ppm)

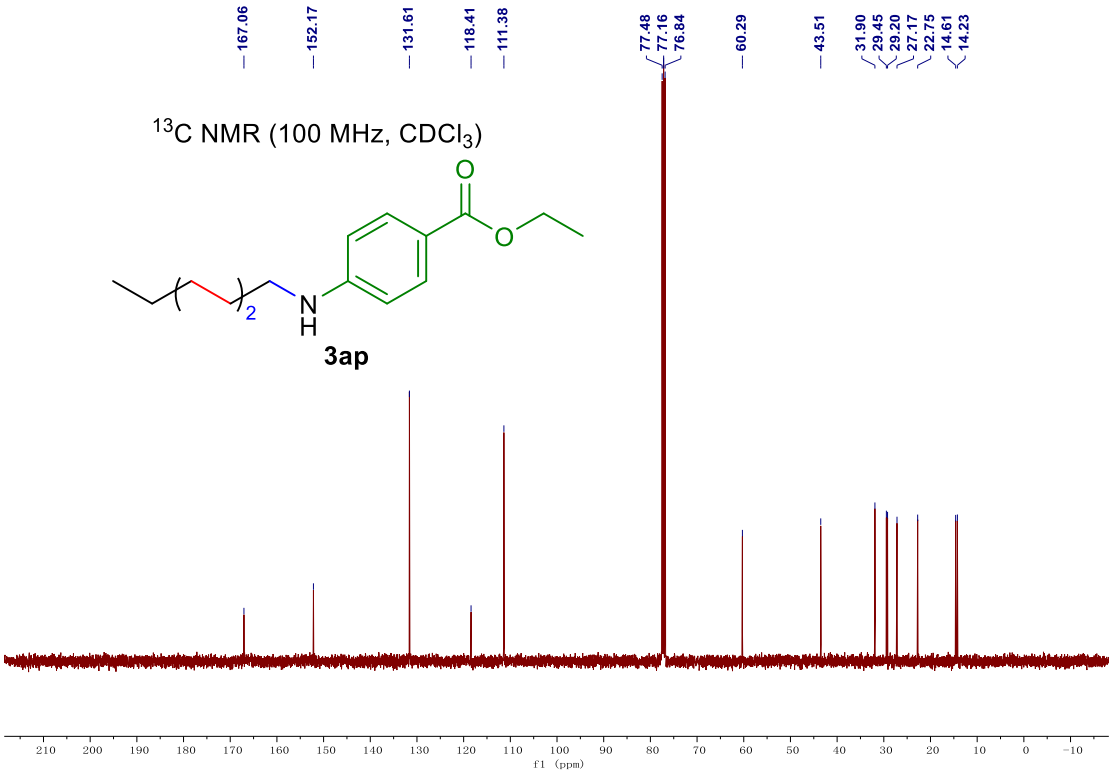

145.38
142.59
139.93
139.66
129.88
128.84
128.21
125.82
125.70
106.40
77.48
77.16
76.84
43.47
31.76
29.73
28.85
26.61
22.65
21.46
14.15
13C NMR (100 MHz, CDCl3)
CF3
N
N
O
S
O
N
H
2
3an
f1 (ppm)

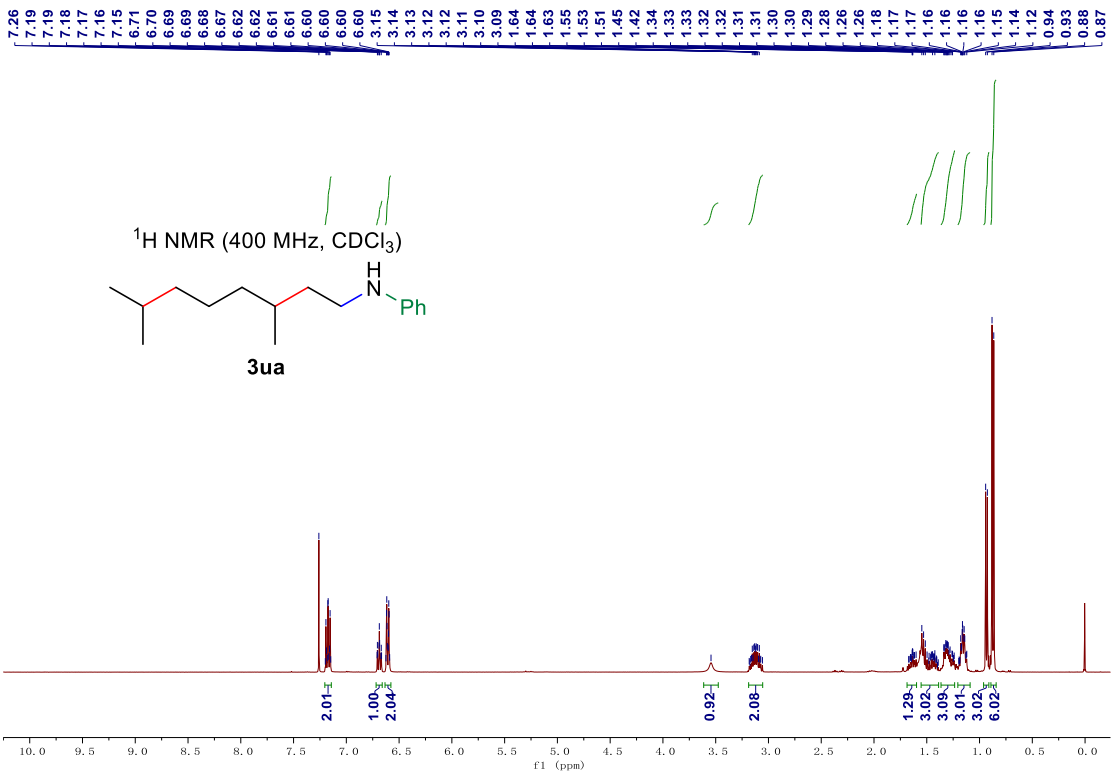

-62.46
19F NMR (376 MHz, CDCl3)
CF3
N
N
O
S
O
N
H
2
3an
f1 (ppm)

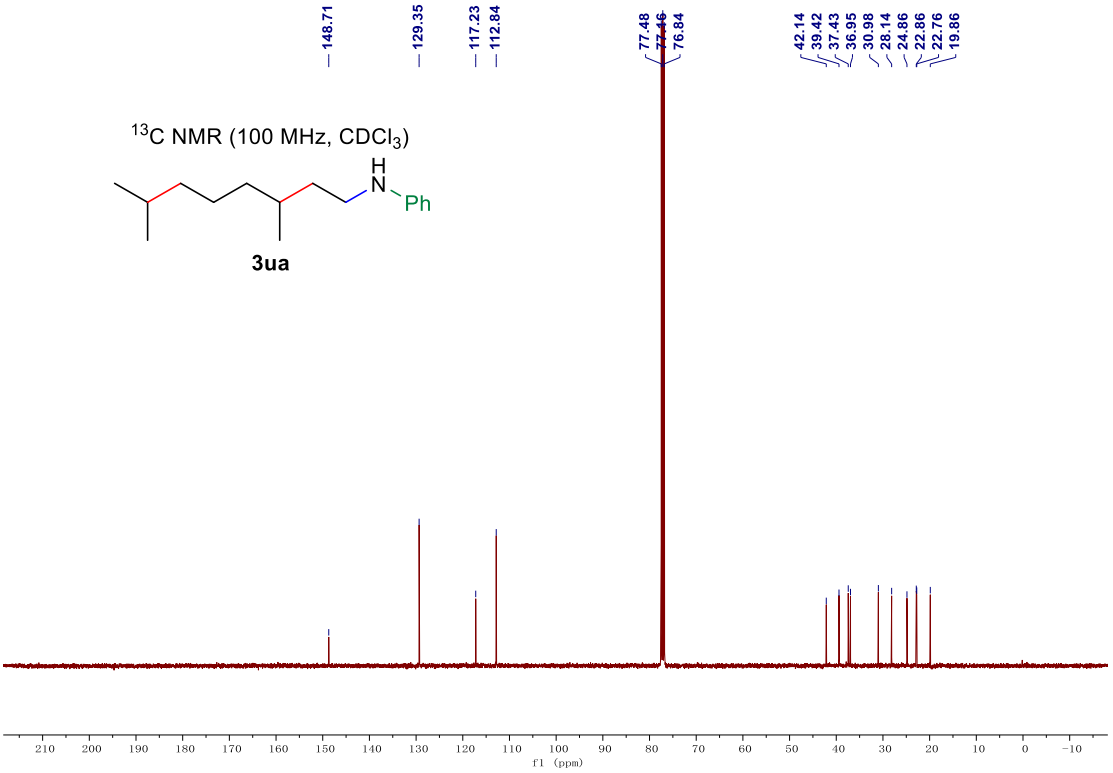

10.34
7.26 7.04 7.04 7.04 7.03 6.85 6.85 6.83 6.83 6.82 6.81 6.80 5.31 5.30 5.28 5.27 5.25 5.24 5.22 3.08 3.06 3.04 1.64 1.62 1.62 1.60 1.58 1.57 1.43 1.43 1.42 1.42 1.41 1.41 1.40 1.39 1.37 1.36 1.35 1.34 1.33 1.32 1.31 1.30 1.30 1.29 1.28 1.28 1.28 1.26 1.25 0.90 0.90 0.89 0.89 0.87
1H NMR (400 MHz, CDCl3)
O
O
OH
N
H
2
3ao
1.00
1.00
2.02
1.02
2.08
2.02
14.29
3.04
11.0 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
f1 (ppm)

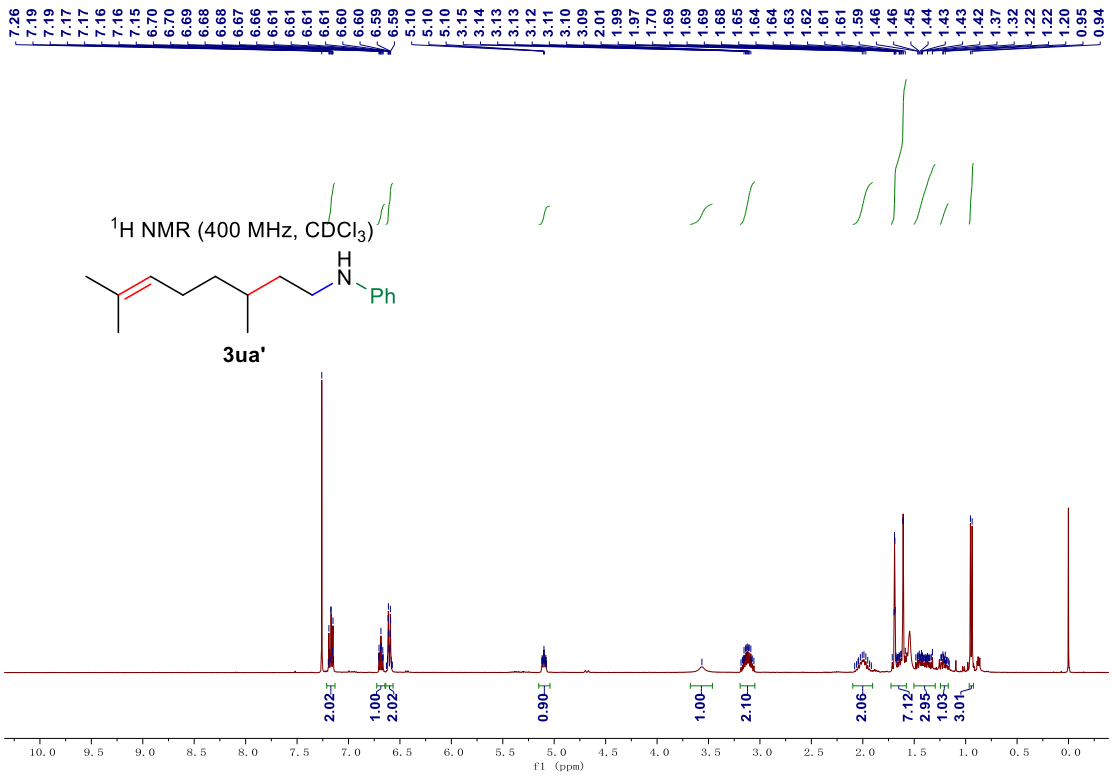

169.86
154.27
141.16
122.43
118.15
112.87
112.07
77.48
77.16
76.84
69.08
45.17
31.93
29.71
29.27
27.27
22.75
22.00
14.24
13C NMR (100 MHz, CDCl3)
O
O
OH
N
H
2
3ao
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

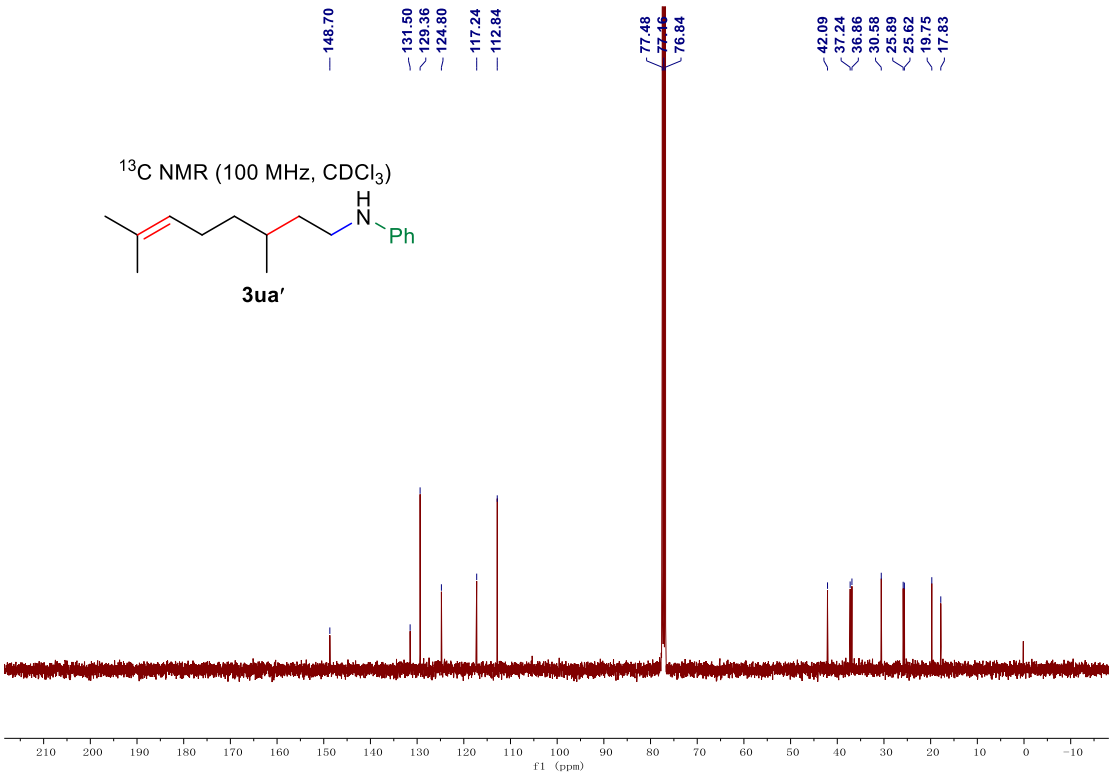

7.88 7.87 7.86 7.86 7.85 7.85 7.84 7.26
6.54 6.54 6.53 6.52 4.33 4.31 4.30 4.28 3.16 3.15 3.13 1.66 1.64 1.62 1.60 1.58 1.41 1.40 1.39 1.37 1.37 1.36 1.35 1.35 1.34 1.33 1.32 1.31 1.31 1.31 1.30 1.30 1.29 1.29 1.28 1.28 1.28 1.27 1.26 1.25 0.90 0.90 0.89 0.89 0.88
1H NMR (400 MHz, CDCl3)
O
O
2
N
H
3ap
2.00 2.06 2.03 0.98 2.02 2.22 11.23 3.05
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 -1.0
f1 (ppm)

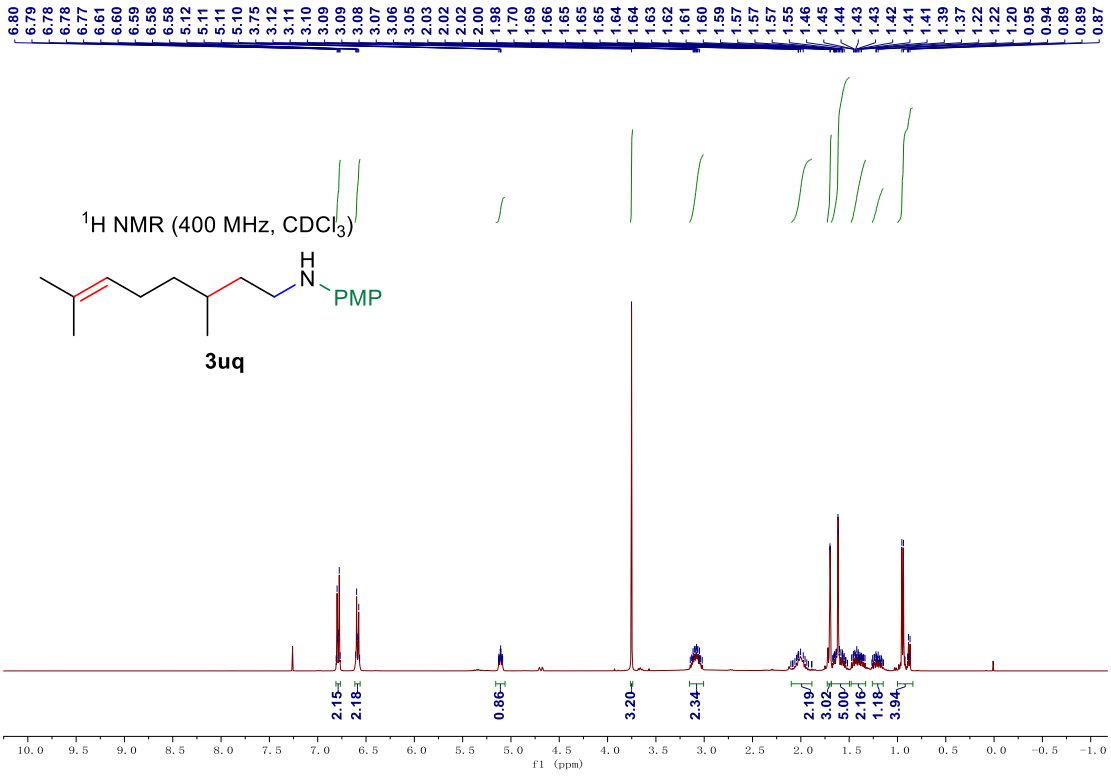

167.06 152.17 131.61 118.41 111.38 77.48 77.16 76.84 60.29 43.51 31.90 29.45 29.20 27.17 22.75 14.61 14.23
13C NMR (100 MHz, CDCl3)
O
O
2
N
H
3ap
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

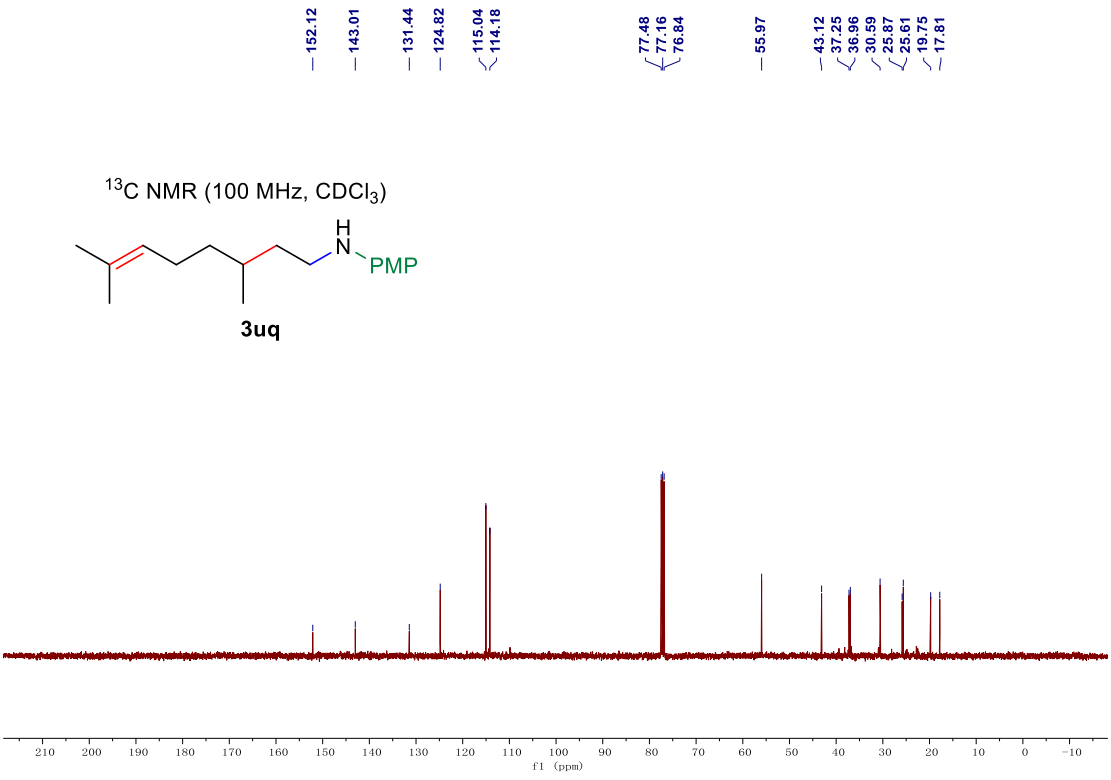

7.26 7.19 7.19 7.18 7.17 7.16 7.15 6.71 6.70 6.69 6.69 6.68 6.67 6.62 6.62 6.61 6.61 6.60 6.60 6.60 6.60 3.15 3.14 3.13 3.12 3.12 3.11 3.10 3.09 1.64 1.64 1.63 1.55 1.53 1.51 1.45 1.42 1.34 1.33 1.33 1.32 1.32 1.31 1.31 1.30 1.30 1.29 1.28 1.26 1.26 1.18 1.17 1.17 1.16 1.16 1.16 1.16 1.15 1.14 1.12 0.94 0.93 0.88 0.87
1H NMR (400 MHz, CDCl3)
H
N
Ph
3ua
2.01 1.00 2.04 0.92 2.08 1.29 3.02 3.09 3.01 3.02 6.02
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
f1 (ppm)

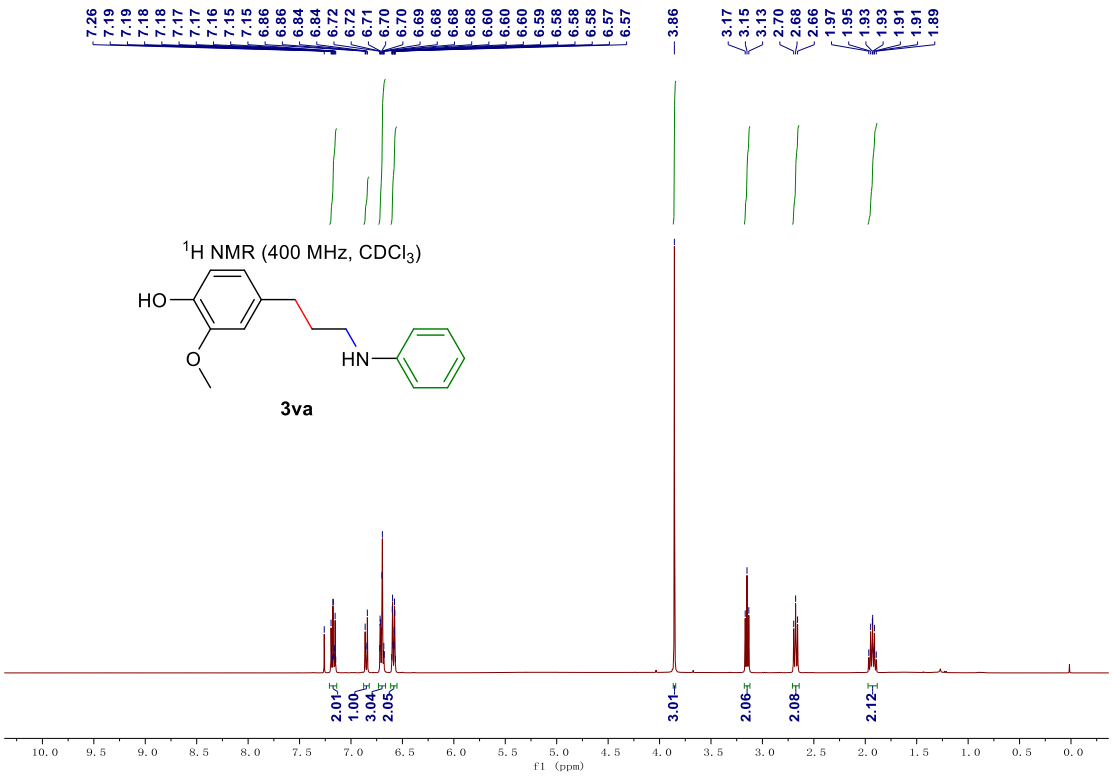

148.71 129.35 117.23 112.84 77.48 77.16 76.84 42.14 39.42 37.43 36.95 30.98 28.14 24.86 22.86 22.76 19.86
13C NMR (100 MHz, CDCl3)
H
N
Ph
3ua
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

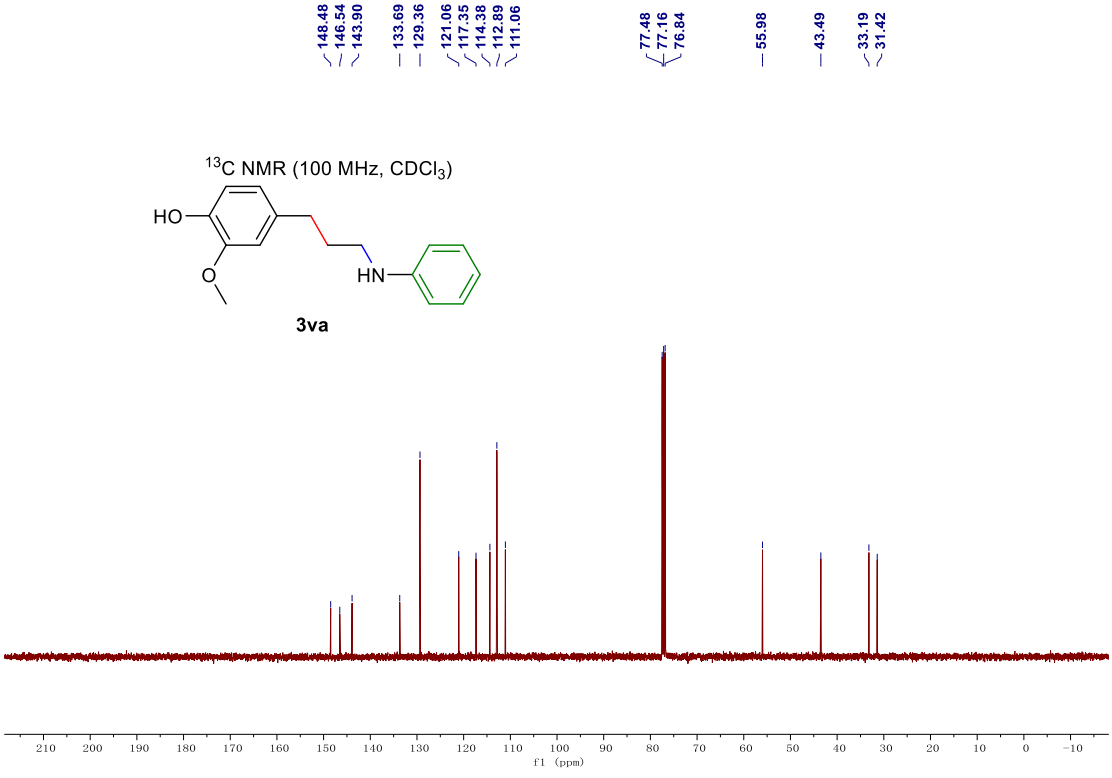

7.26 7.19 7.19 7.17 7.17 7.16 7.16 7.15 6.70 6.70 6.69 6.68 6.68 6.67 6.66 6.61 6.61 6.61 6.61 6.60 6.60 6.59 6.59
5.10 5.10 5.10 3.15 3.14 3.13 3.13 3.12 3.11 3.10 3.09 2.01 1.99 1.97 1.70 1.69 1.69 1.69 1.68 1.65 1.64 1.64 1.63 1.62 1.61 1.61 1.59 1.46 1.46 1.45 1.44 1.43 1.43 1.42 1.37 1.32 1.22 1.22 1.20 0.95 0.94
^{1}H NMR (400 MHz, CDCl$_3$)
H
N
Ph
3ua'
2.02 1.00 2.02 0.90 1.00 2.10 2.06 7.12 2.95 1.03 3.01
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
f1 (ppm)

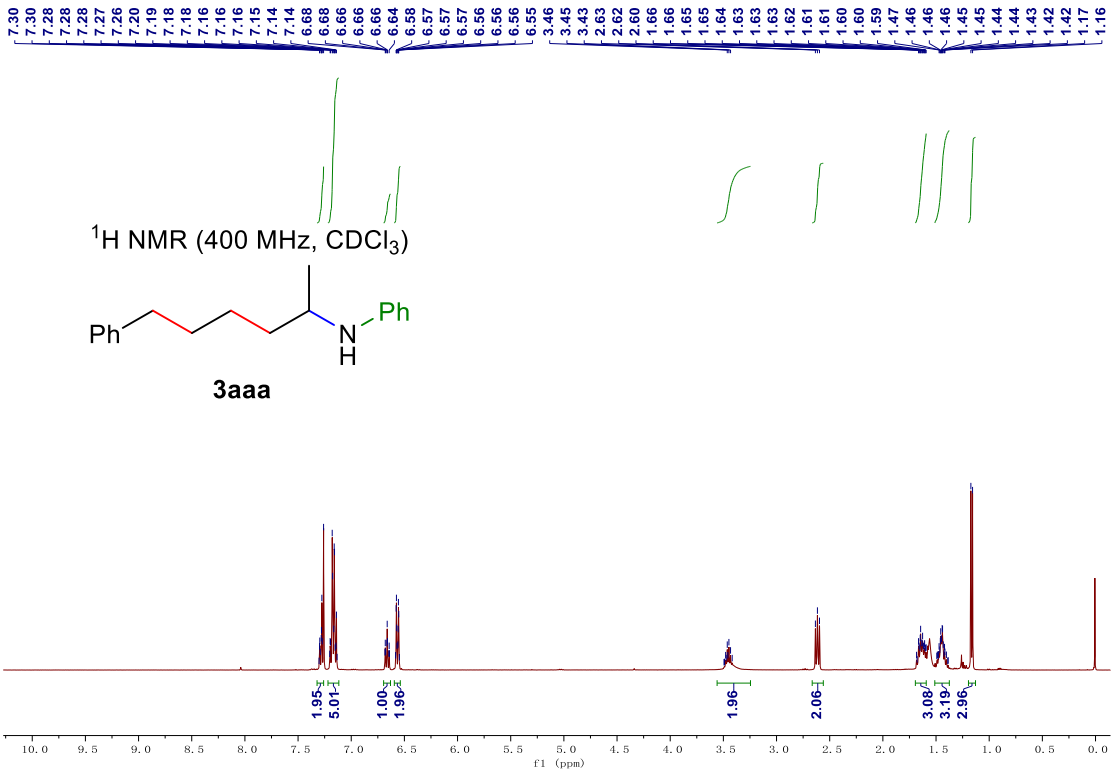

148.70 131.50 129.36 124.80 117.24 112.84
77.48 77.16 76.84
42.09 37.24 36.86 30.58 25.89 25.62 19.75 17.83
^{13}C NMR (100 MHz, CDCl$_3$)
H
N
Ph
3ua′
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

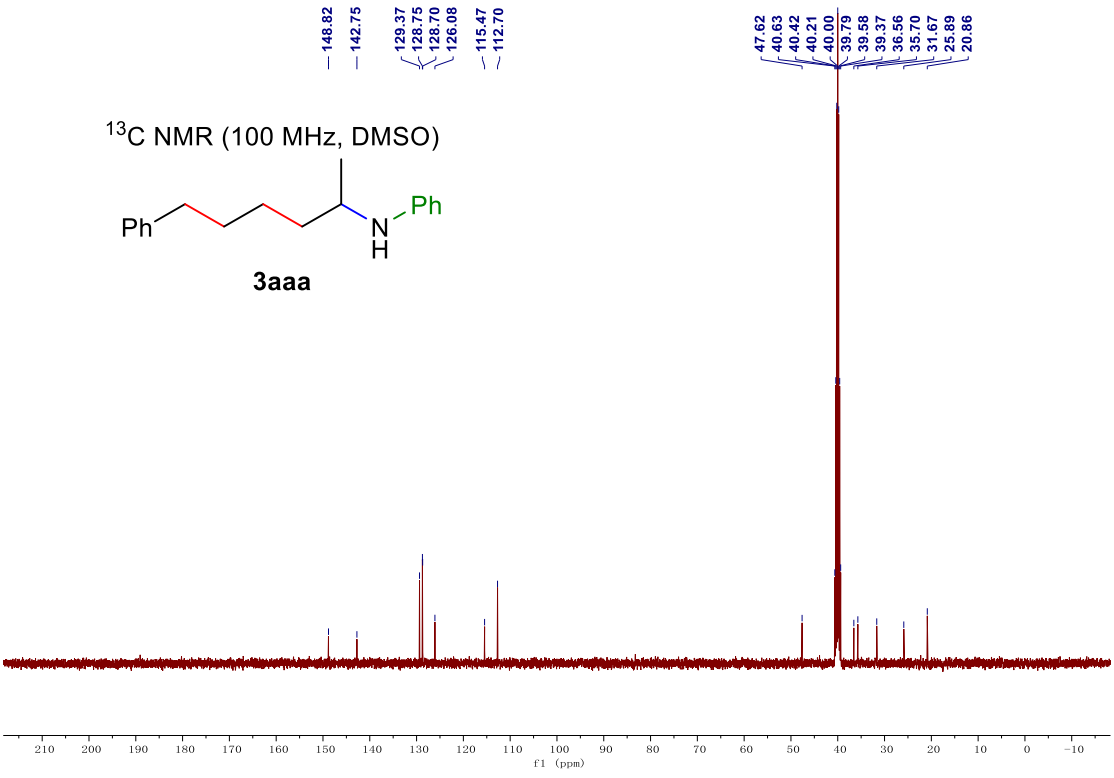

6.80 6.79 6.78 6.78 6.77 6.61 6.60 6.59 6.58 6.58 5.12 5.11 5.11 5.10 3.75 3.12 3.11 3.10 3.09 3.09 3.08 3.07 3.06 3.05 2.03 2.02 2.02 2.00 1.98 1.70 1.69 1.66 1.65 1.65 1.65 1.64 1.64 1.63 1.62 1.61 1.60 1.59 1.57 1.57 1.57 1.55 1.46 1.45 1.44 1.43 1.43 1.42 1.41 1.41 1.39 1.37 1.22 1.22 1.20 0.95 0.94 0.89 0.89 0.87
^{1}H NMR (400 MHz, CDCl$_3$)
H
N
PMP
3uq
2.15 2.18 0.86 3.20 2.34 2.19 3.02 5.00 2.16 1.18 3.94
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 −1.0
f1 (ppm)


152.12 143.01 131.44 124.82 115.04 114.18 77.48 77.16 76.84 55.97 43.12 37.25 36.96 30.59 25.87 25.61 19.75 17.81
^{13}C NMR (100 MHz, CDCl$_3$)
H
N
PMP
3uq
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)


7.26 7.19 7.19 7.18 7.18 7.17 7.17 7.16 7.15 7.15 6.86 6.86 6.84 6.84 6.72 6.72 6.71 6.70 6.70 6.69 6.68 6.68 6.68 6.60 6.60 6.60 6.59 6.58 6.58 6.58 6.57 6.57
3.86
3.17 3.15 3.13 2.70 2.68 2.66 1.97 1.95 1.93 1.93 1.91 1.91 1.89
1H NMR (400 MHz, CDCl3)
HO
O
HN
3va
2.01 1.00 3.04 2.05
3.01
2.06
2.08
2.12
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
f1 (ppm)


148.48 146.54 143.90 133.69 129.36 121.06 117.35 114.38 112.89 111.06
77.48 77.16 76.84
55.98
43.49
33.19 31.42
13C NMR (100 MHz, CDCl3)
HO
O
HN
3va
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)


7.30 7.30 7.28 7.28 7.28 7.27 7.26 7.20 7.19 7.18 7.18 7.16 7.16 7.16 7.15 7.14 7.14 6.68 6.68 6.66 6.66 6.66 6.64 6.58 6.57 6.57 6.56 6.56 6.56 6.55 3.46 3.45 3.43 2.63 2.62 2.60 1.66 1.66 1.65 1.65 1.64 1.63 1.63 1.63 1.62 1.61 1.61 1.60 1.60 1.59 1.47 1.46 1.46 1.46 1.45 1.45 1.44 1.44 1.43 1.42 1.42 1.17 1.16
1H NMR (400 MHz, CDCl3)
Ph
N
H
Ph
3aaa
1.95 5.01 1.00 1.96 1.96 2.06 3.08 3.19 2.96
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
f1 (ppm)


148.82 142.75 129.37 128.75 128.70 126.08 115.47 112.70
47.62 40.63 40.42 40.21 40.00 39.79 39.58 39.37 36.56 35.70 31.67 25.89 20.86
13C NMR (100 MHz, DMSO)
Ph
N
H
Ph
3aaa
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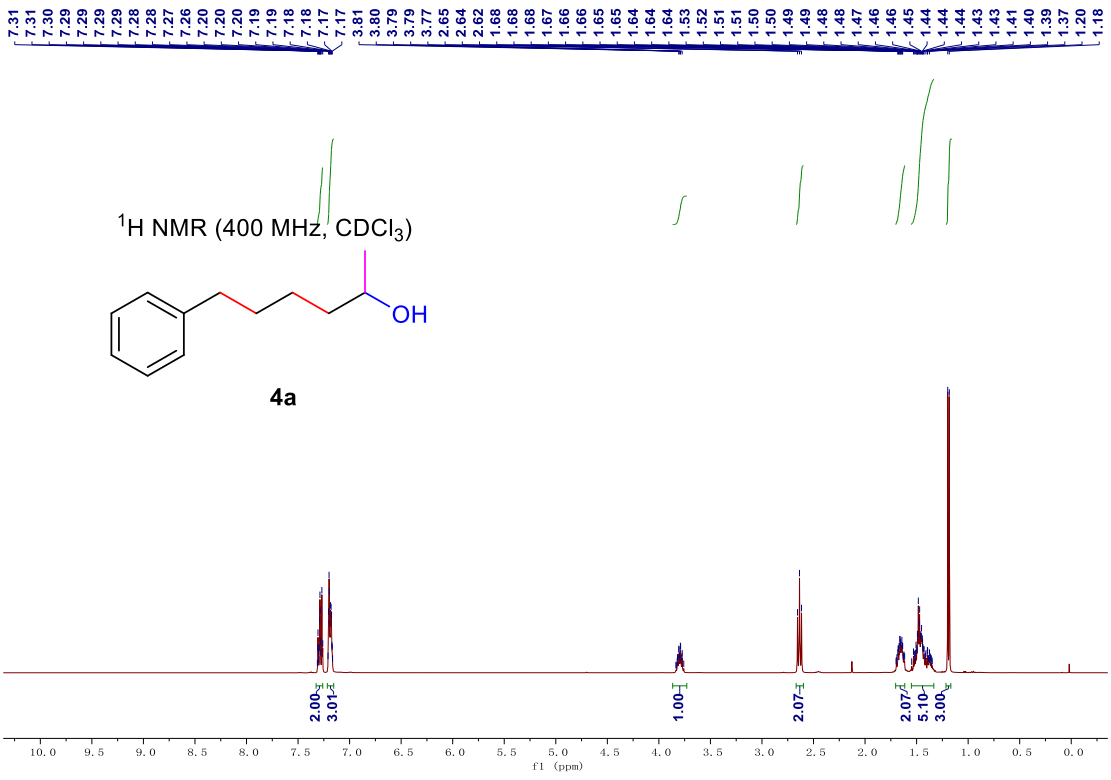
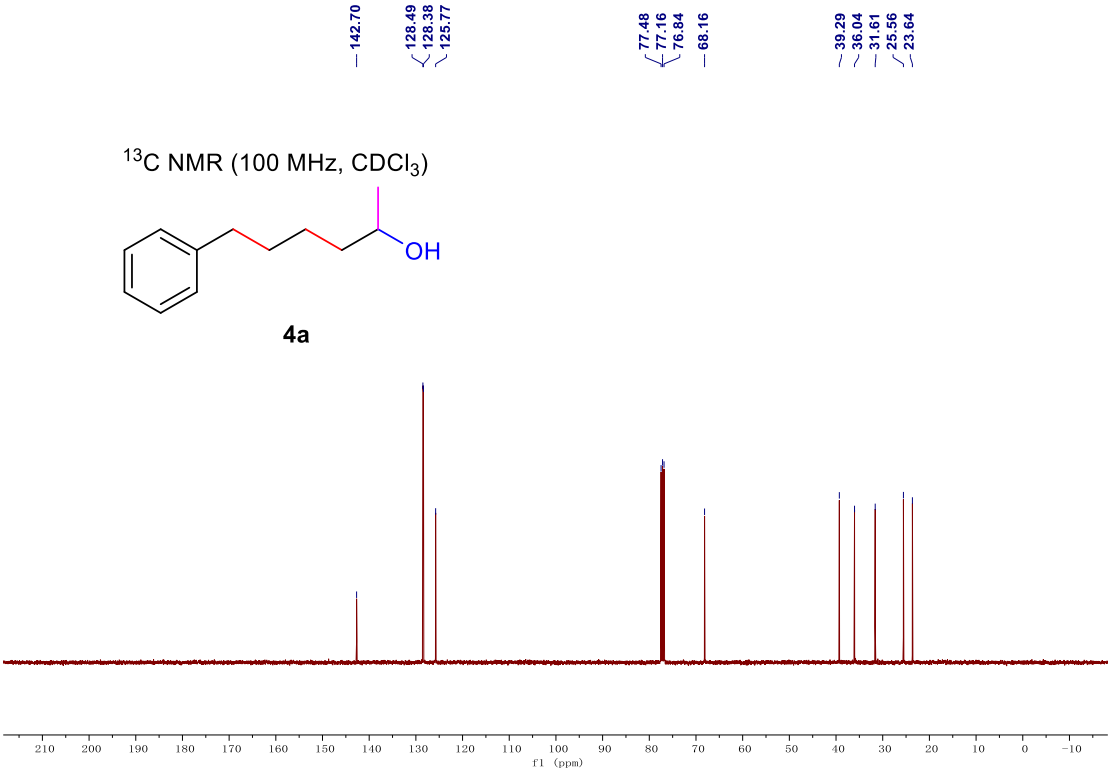
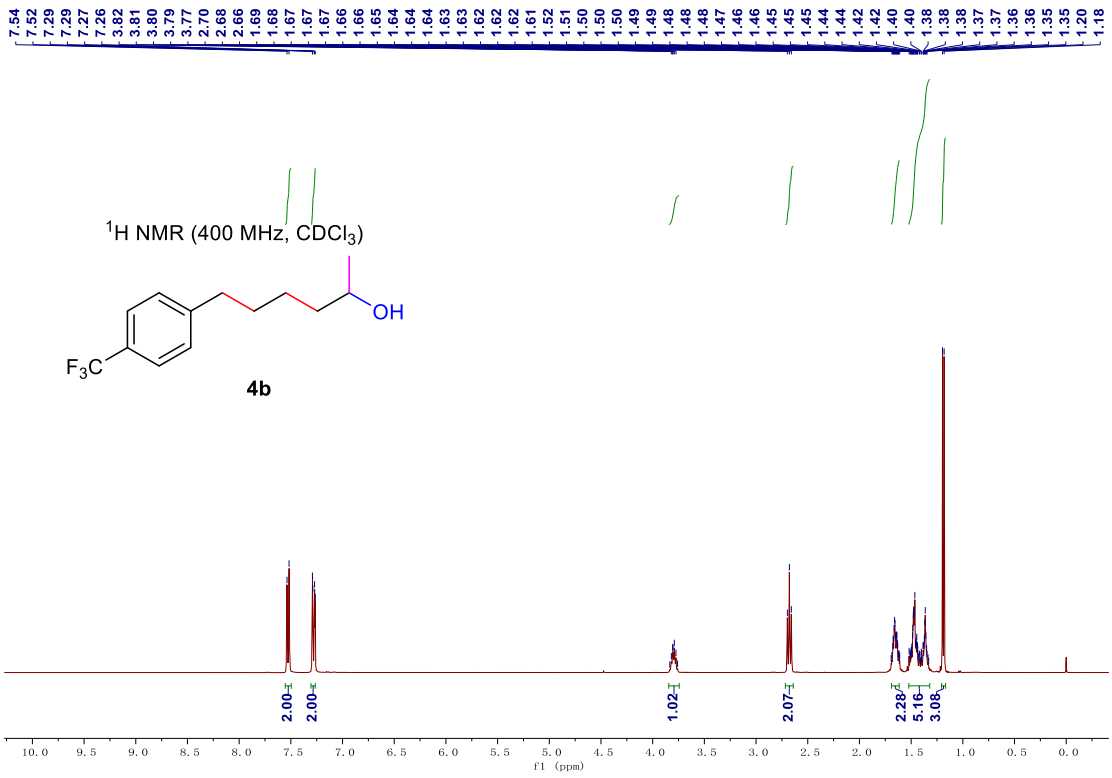
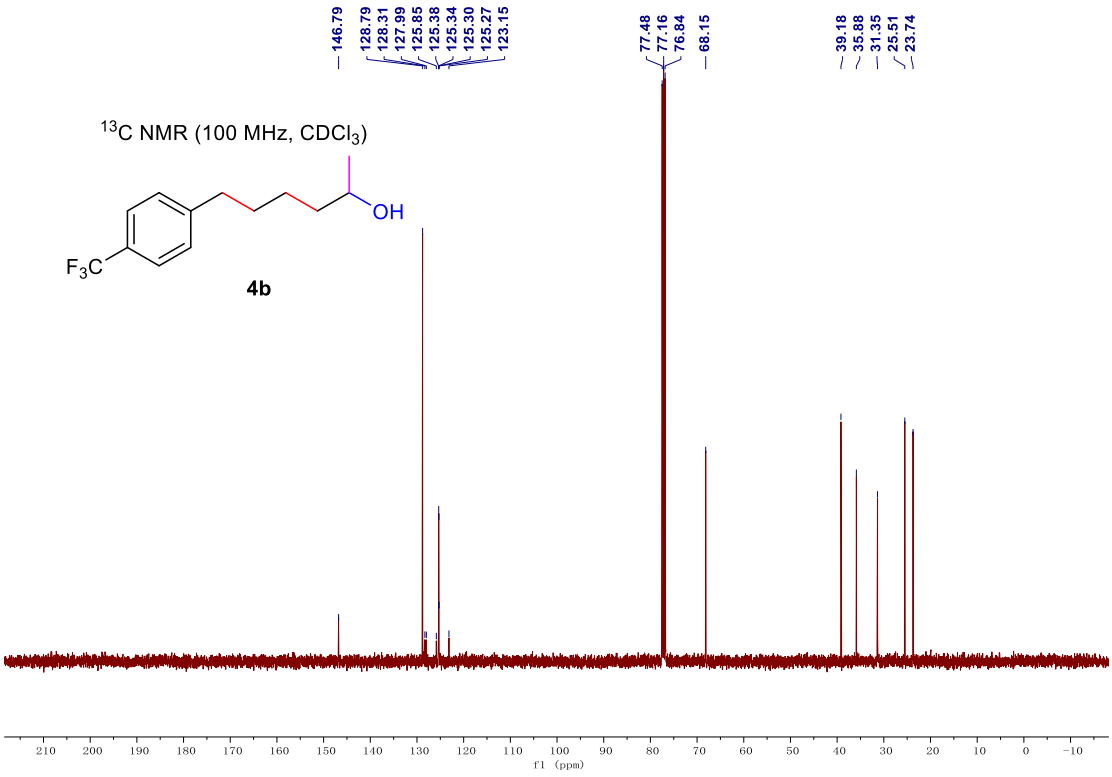
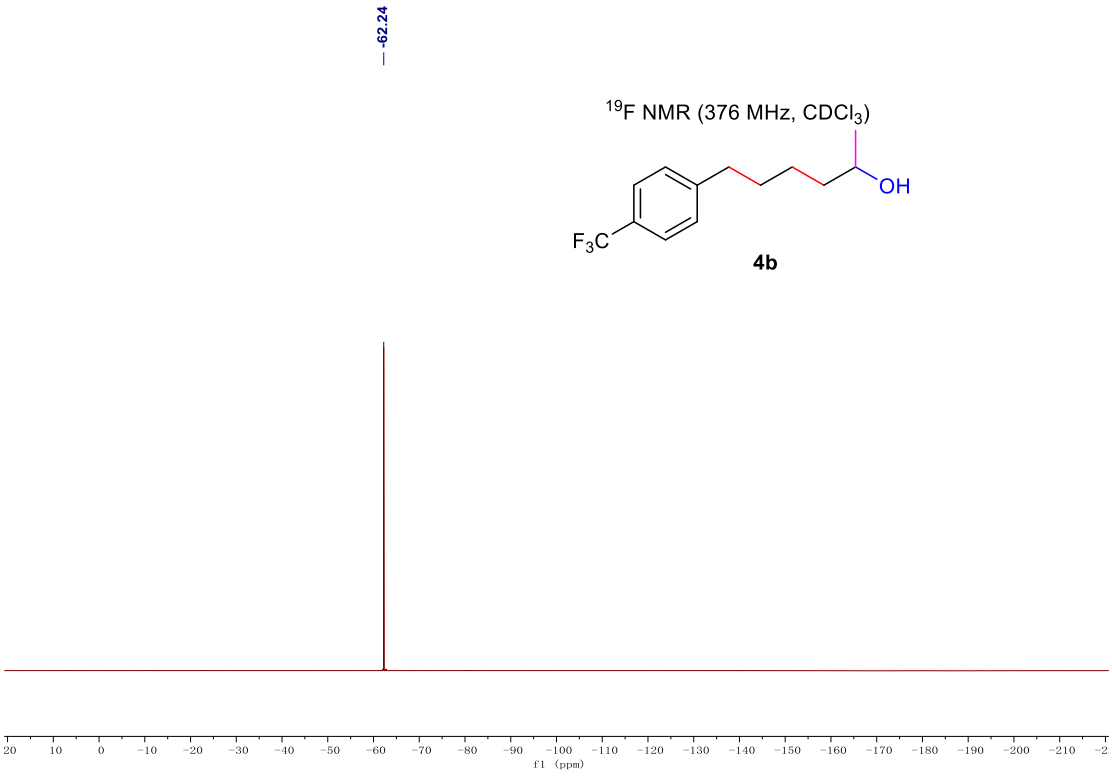
f1 (ppm)

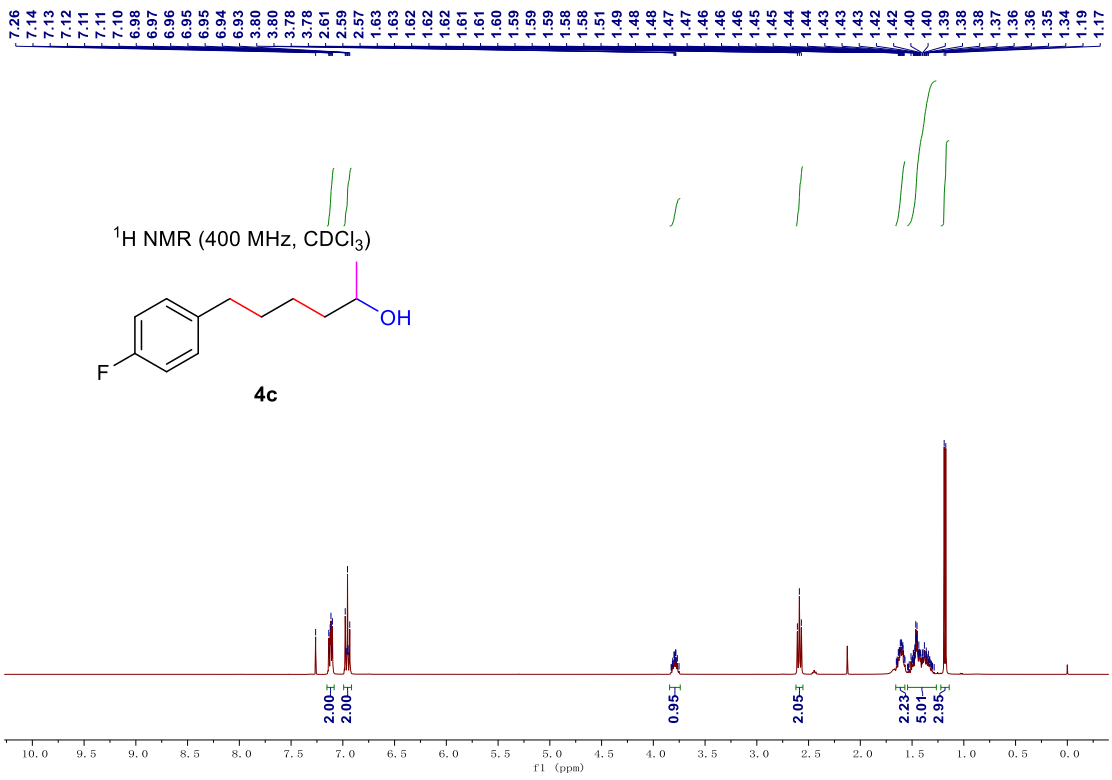

7.31 7.31 7.30 7.29 7.29 7.29 7.28 7.28 7.27 7.26 7.20 7.20 7.20 7.19 7.19 7.18 7.18 7.17 7.17
3.81 3.80 3.79 3.79 3.77 2.65 2.64 2.62 1.68 1.68 1.68 1.67 1.66 1.66 1.65 1.65 1.64 1.64 1.64 1.53 1.52 1.51 1.51 1.50 1.50 1.49 1.49 1.48 1.48 1.47 1.46 1.46 1.45 1.44 1.44 1.44 1.43 1.43 1.41 1.40 1.39 1.37 1.20 1.18
1H NMR (400 MHz, CDCl3)
OH
4a
2.00 3.01 1.00 2.07 2.07 5.10 3.00
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
f1 (ppm)

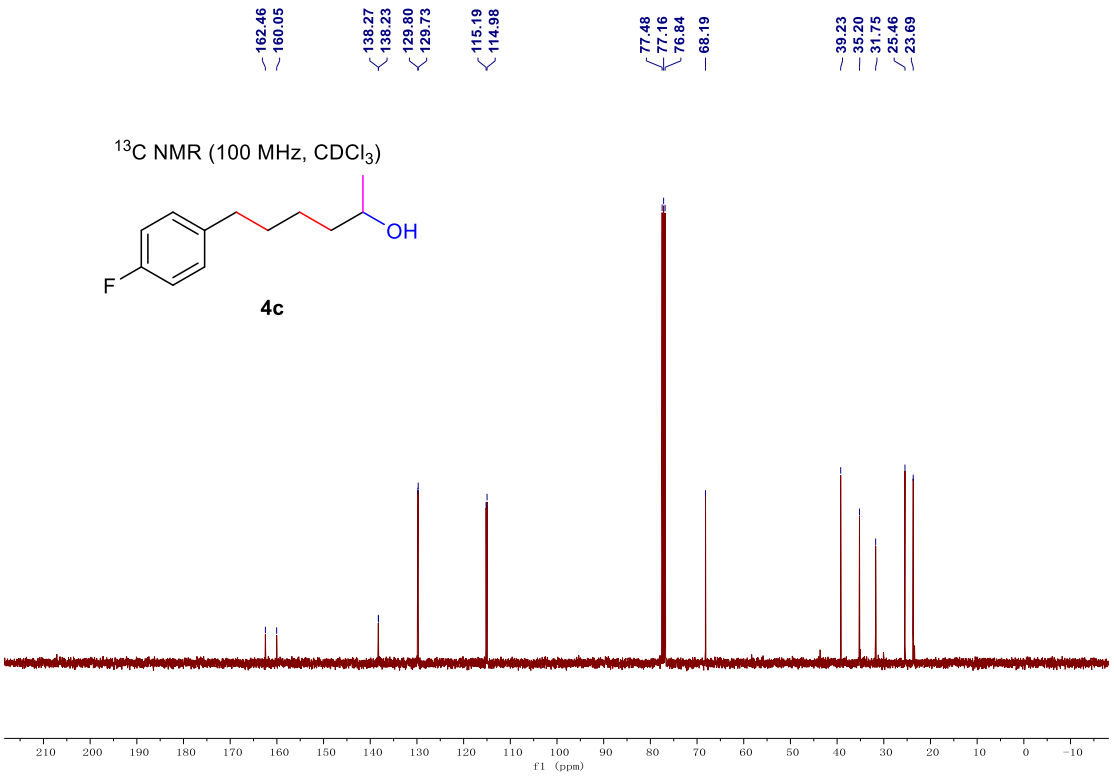

142.70
128.49 128.38 125.77
77.48 77.16 76.84
68.16
39.29 36.04 31.61 25.56 23.64
13C NMR (100 MHz, CDCl3)
OH
4a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

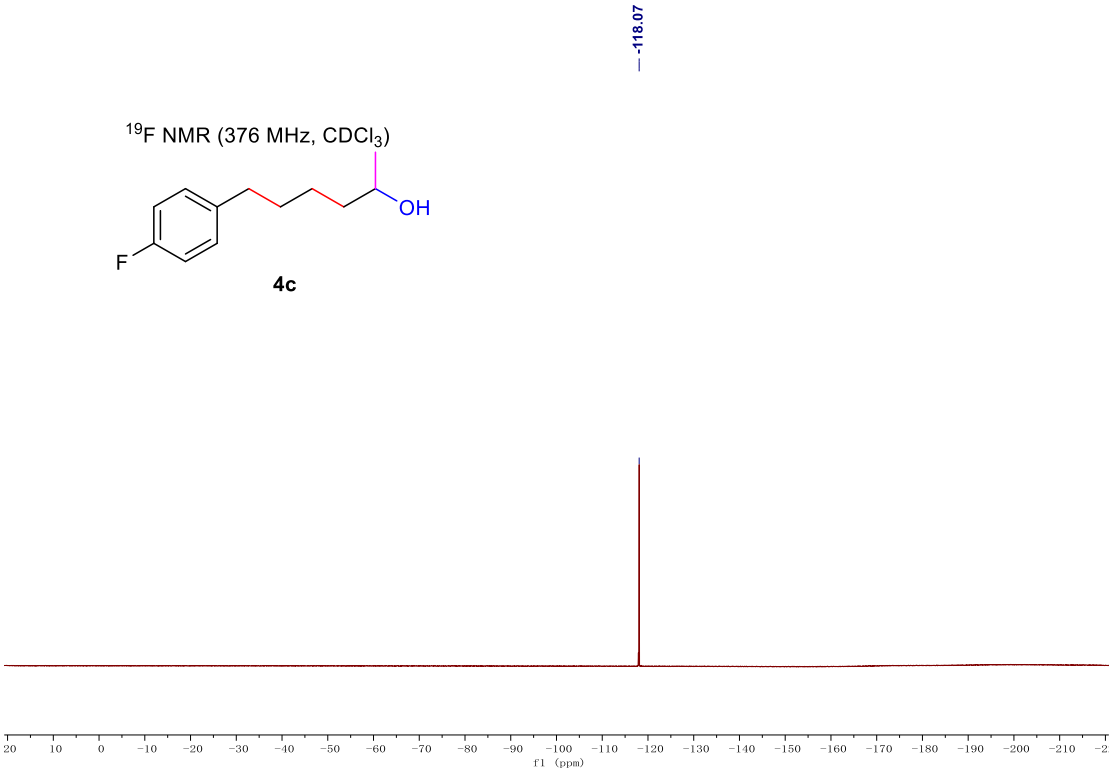

7.54 7.52 7.29 7.29 7.27 7.26 3.82 3.81 3.80 3.79 3.77 2.70 2.68 2.66 1.69 1.68 1.67 1.67 1.67 1.66 1.66 1.65 1.64 1.64 1.64 1.63 1.63 1.62 1.62 1.62 1.61 1.52 1.51 1.50 1.50 1.50 1.49 1.49 1.48 1.48 1.48 1.47 1.46 1.46 1.45 1.45 1.45 1.44 1.44 1.42 1.42 1.40 1.40 1.38 1.38 1.38 1.37 1.37 1.36 1.36 1.35 1.35 1.20 1.18
1H NMR (400 MHz, CDCl3)
OH
F3C
4b
2.00 2.00 1.02 2.07 2.28 5.16 3.08
f1 (ppm)

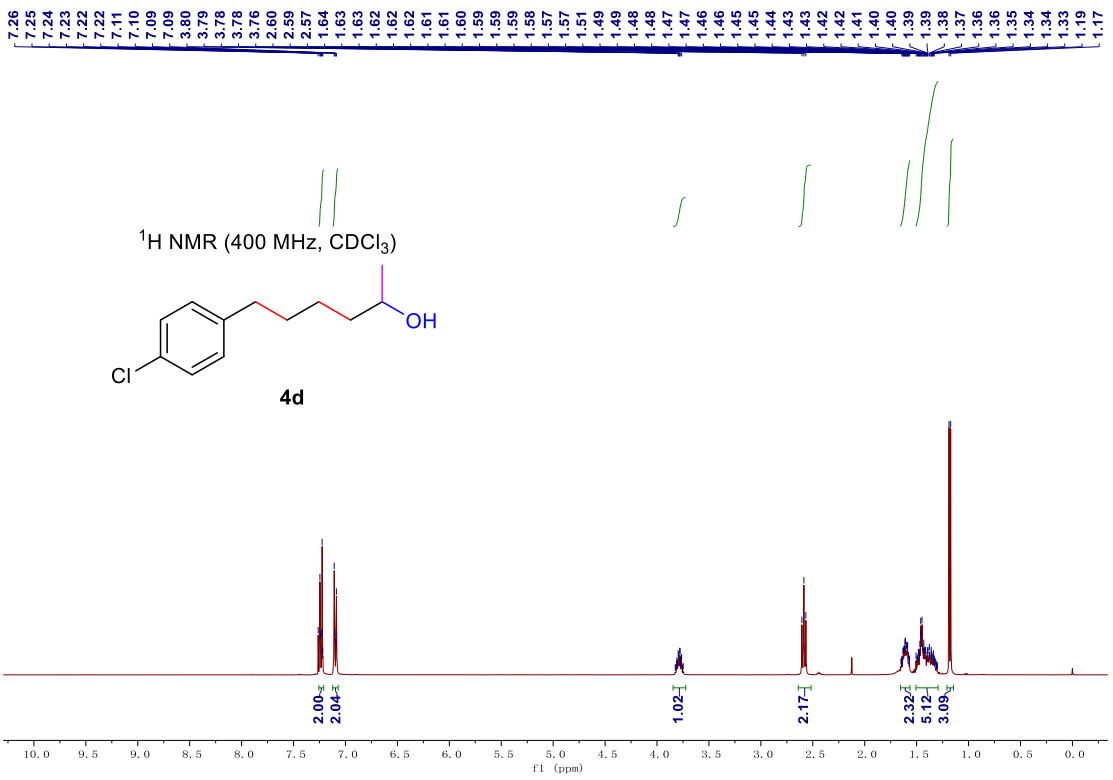

146.79 128.79 128.31 127.99 125.85 125.38 125.34 125.30 125.27 123.15
77.48 77.16 76.84 68.15
39.18 35.88 31.35 25.51 23.74
13C NMR (100 MHz, CDCl3)
OH
F3C
4b
f1 (ppm)

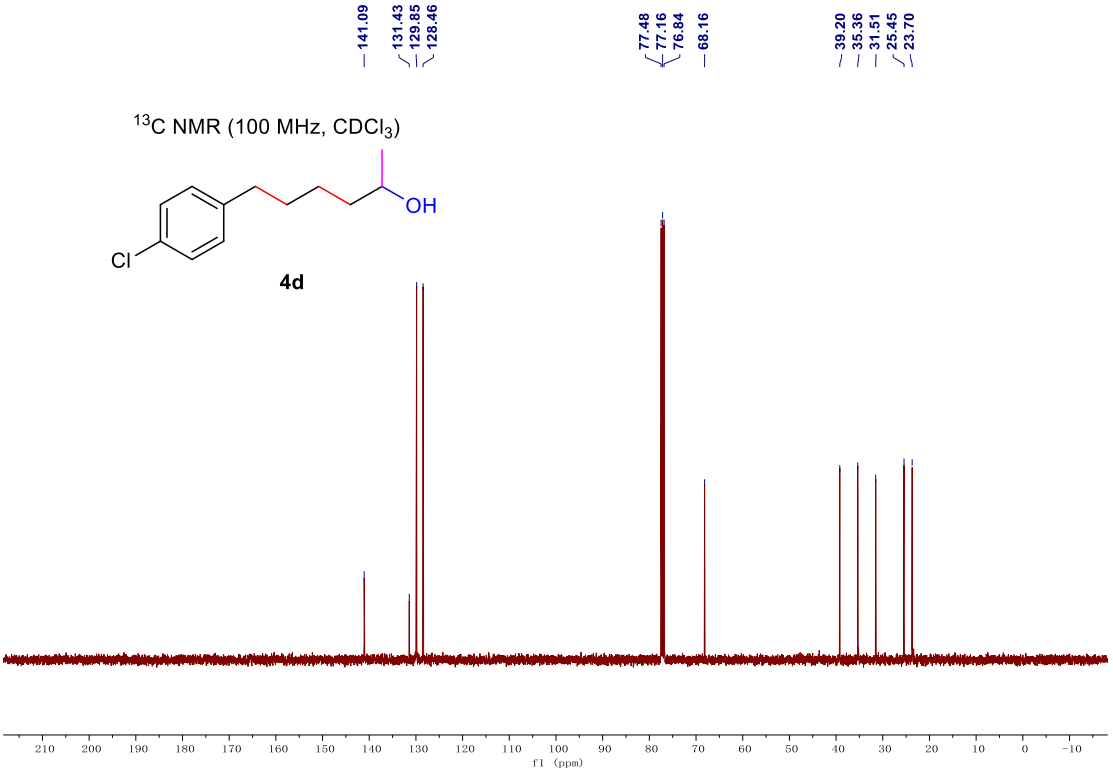

-62.24
^{19}F NMR (376 MHz, $CDCl_3$)
OH
F_3C
4b
f1 (ppm)


^{1}H NMR (400 MHz, $CDCl_3$)
OH
F
4c
2.00
2.00
0.95
2.05
2.23
5.01
2.95
f1 (ppm)


162.46
160.05
138.27
138.23
129.80
129.73
115.19
114.98
77.48
77.16
76.84
68.19
39.23
35.20
31.75
25.46
23.69
13C NMR (100 MHz, CDCl3)
OH
F
4c
f1 (ppm)


-118.07
19F NMR (376 MHz, CDCl3)
OH
F
4c
f1 (ppm)


7.26 7.25 7.24 7.23 7.22 7.22 7.11 7.10 7.09 7.09 3.80 3.79 3.78 3.78 3.76 2.60 2.59 2.57 1.64 1.63 1.63 1.62 1.62 1.61 1.61 1.60 1.59 1.59 1.59 1.58 1.57 1.57 1.51 1.49 1.49 1.48 1.48 1.47 1.47 1.46 1.46 1.45 1.45 1.44 1.43 1.43 1.42 1.42 1.41 1.40 1.40 1.39 1.39 1.38 1.37 1.36 1.36 1.35 1.34 1.34 1.33 1.19 1.17
1H NMR (400 MHz, CDCl3)
OH
Cl
4d
2.00 2.04 1.02 2.17 2.32 5.12 3.09
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


141.09 131.43 129.85 128.46 77.48 77.16 76.84 68.16 39.20 35.36 31.51 25.45 23.70
13C NMR (100 MHz, CDCl3)
OH
Cl
4d
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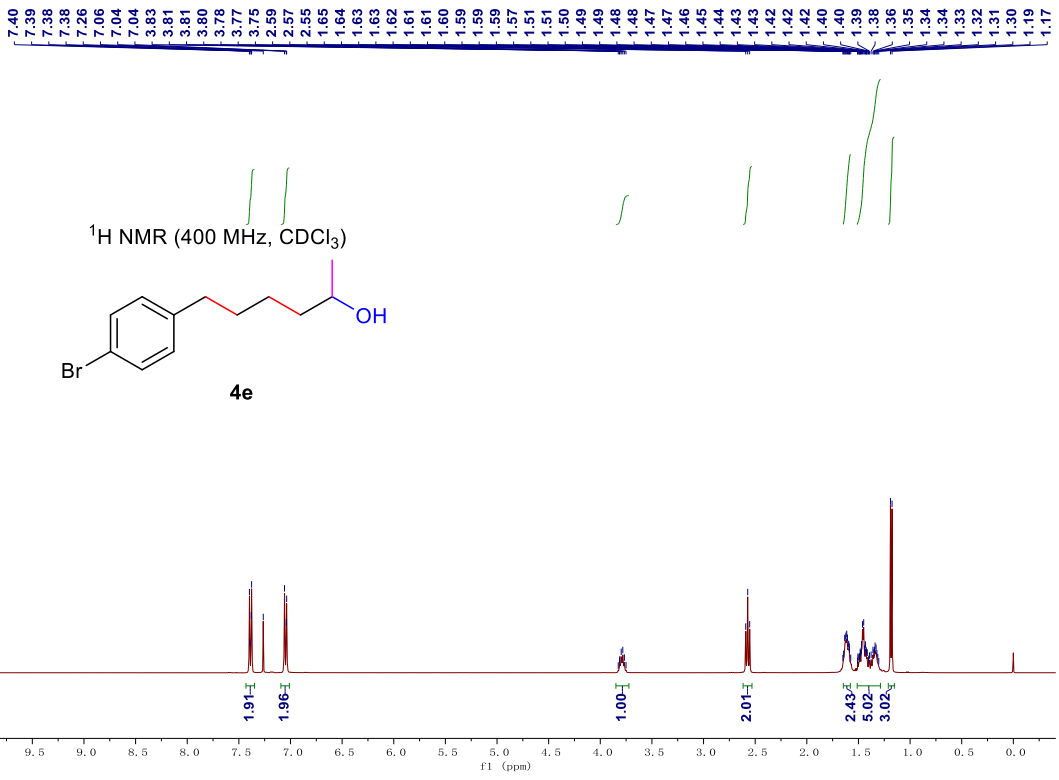
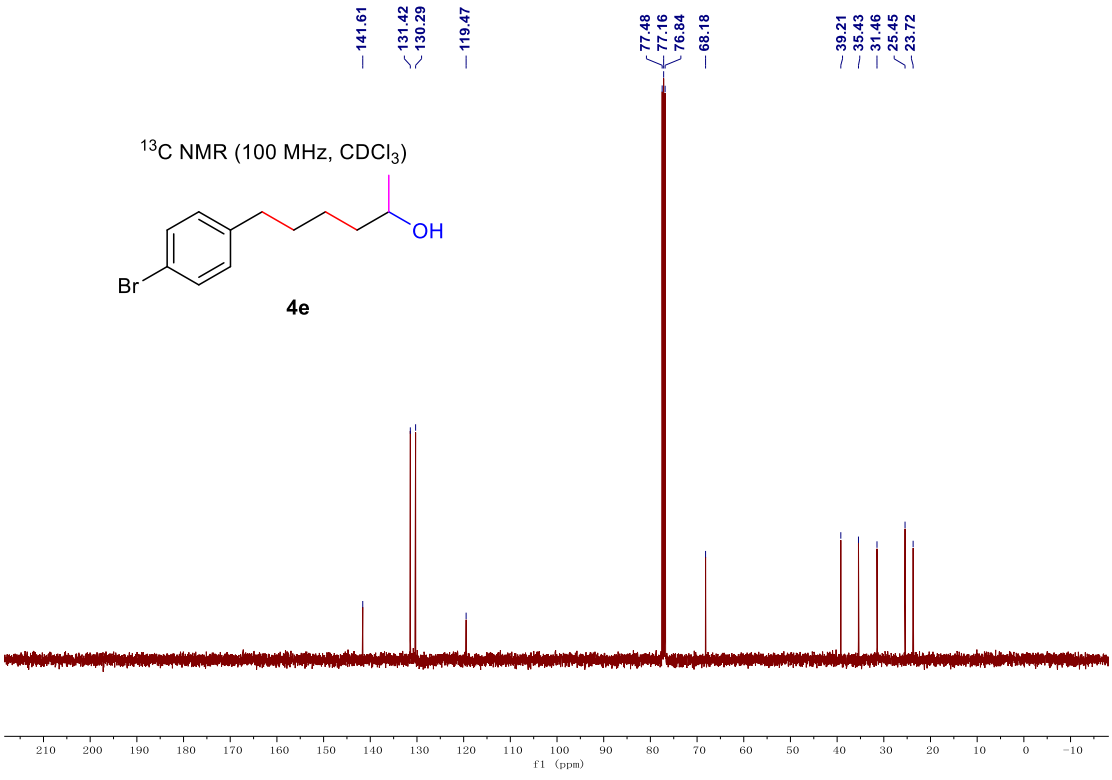
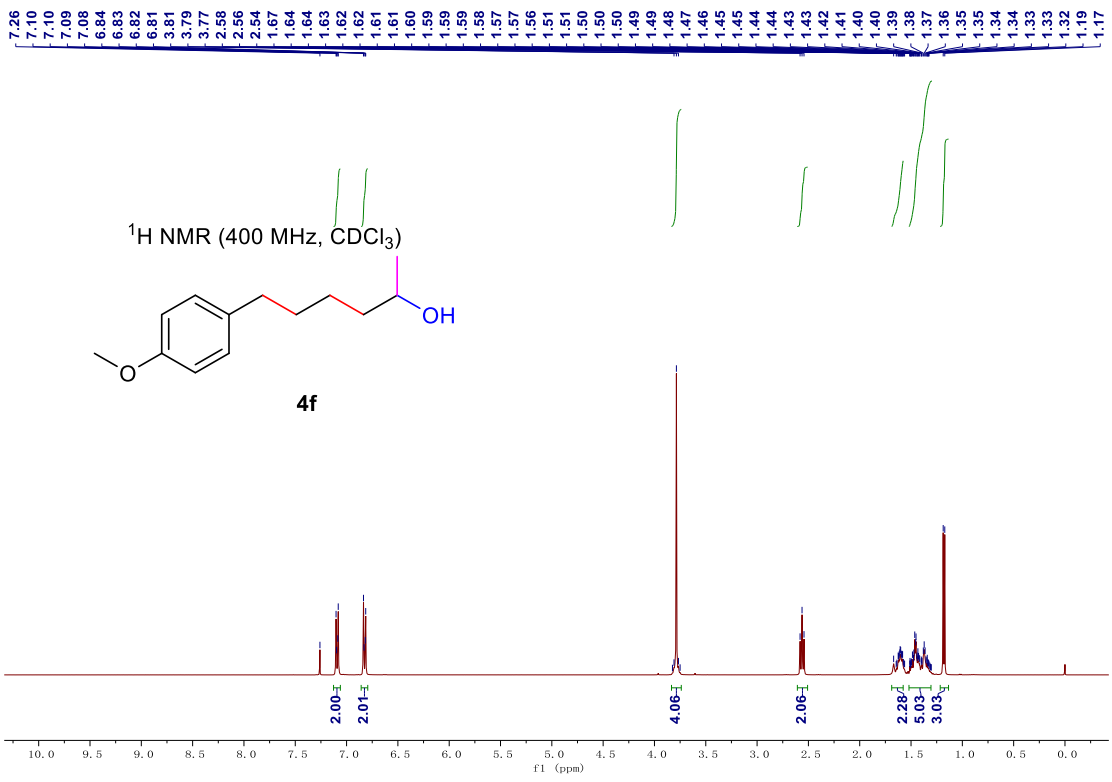
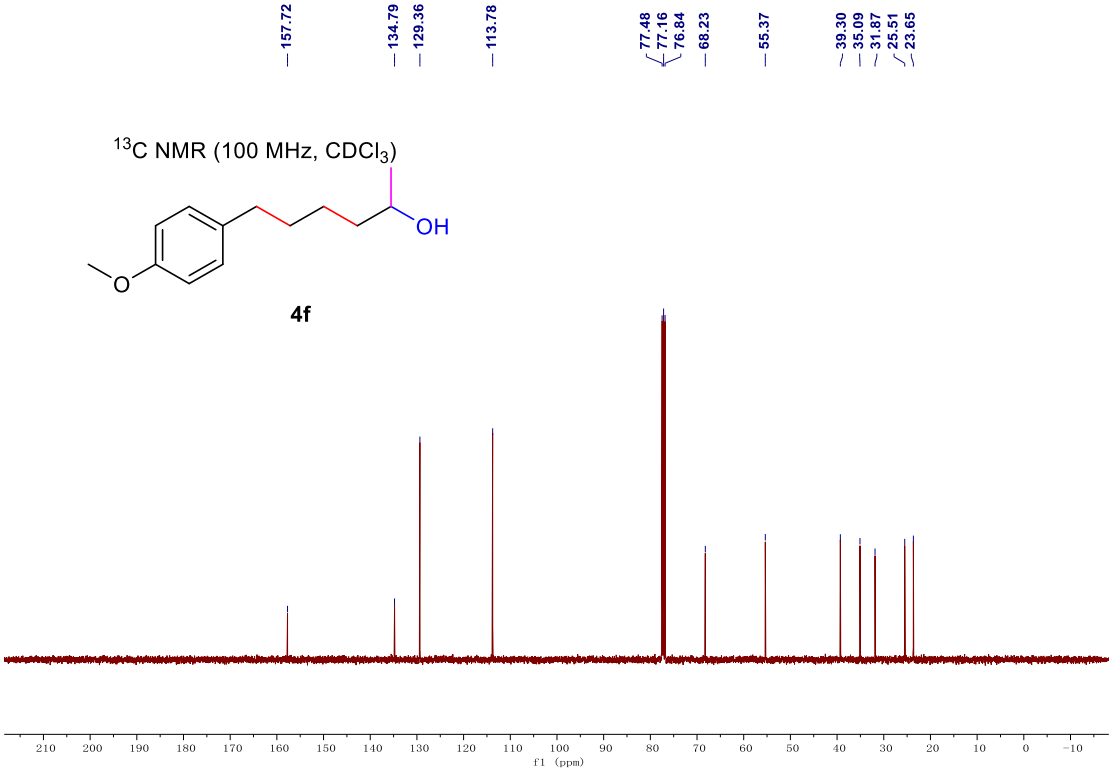
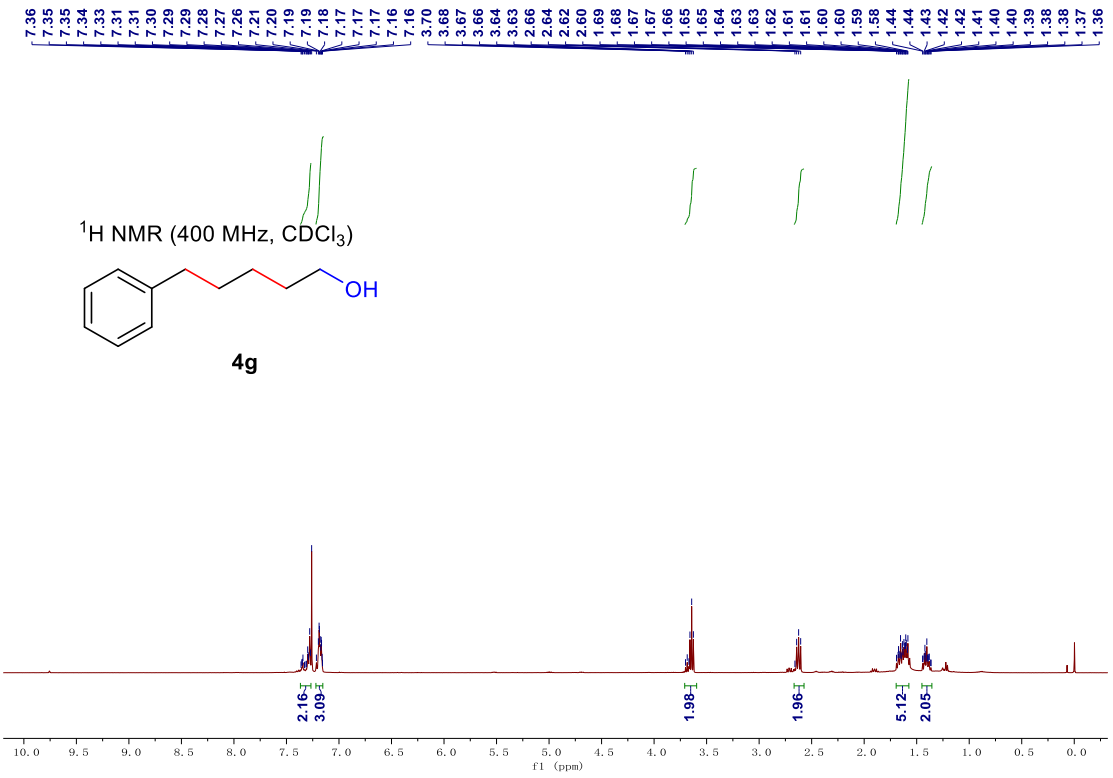
f1 (ppm)

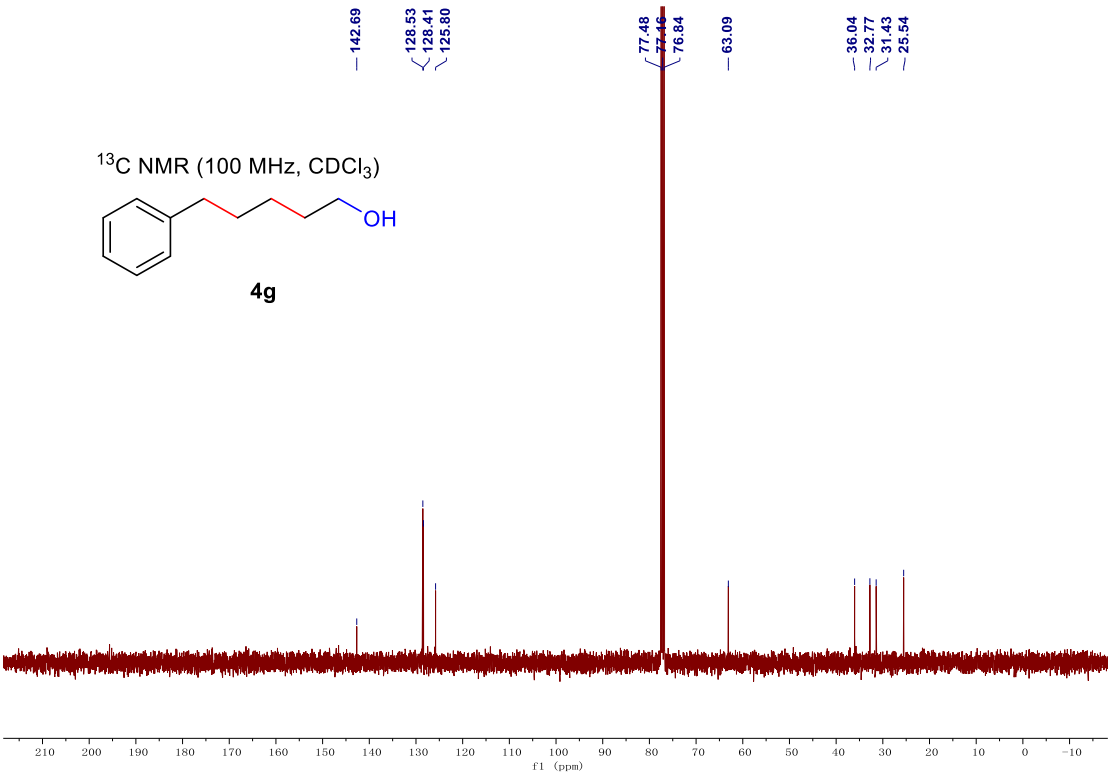

7.40 7.39 7.38 7.38 7.26 7.06 7.04 7.04 3.83 3.81 3.81 3.80 3.78 3.77 3.75 2.59 2.57 2.55 1.65 1.64 1.63 1.63 1.62 1.61 1.61 1.60 1.59 1.59 1.59 1.57 1.51 1.51 1.50 1.49 1.49 1.48 1.48 1.47 1.47 1.46 1.45 1.44 1.43 1.43 1.42 1.42 1.42 1.40 1.40 1.39 1.38 1.36 1.35 1.34 1.34 1.33 1.32 1.31 1.30 1.19 1.17
1H NMR (400 MHz, CDCl3)
OH
Br
4e
1.91
1.96
1.00
2.01
2.43
5.02
3.02
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
f1 (ppm)

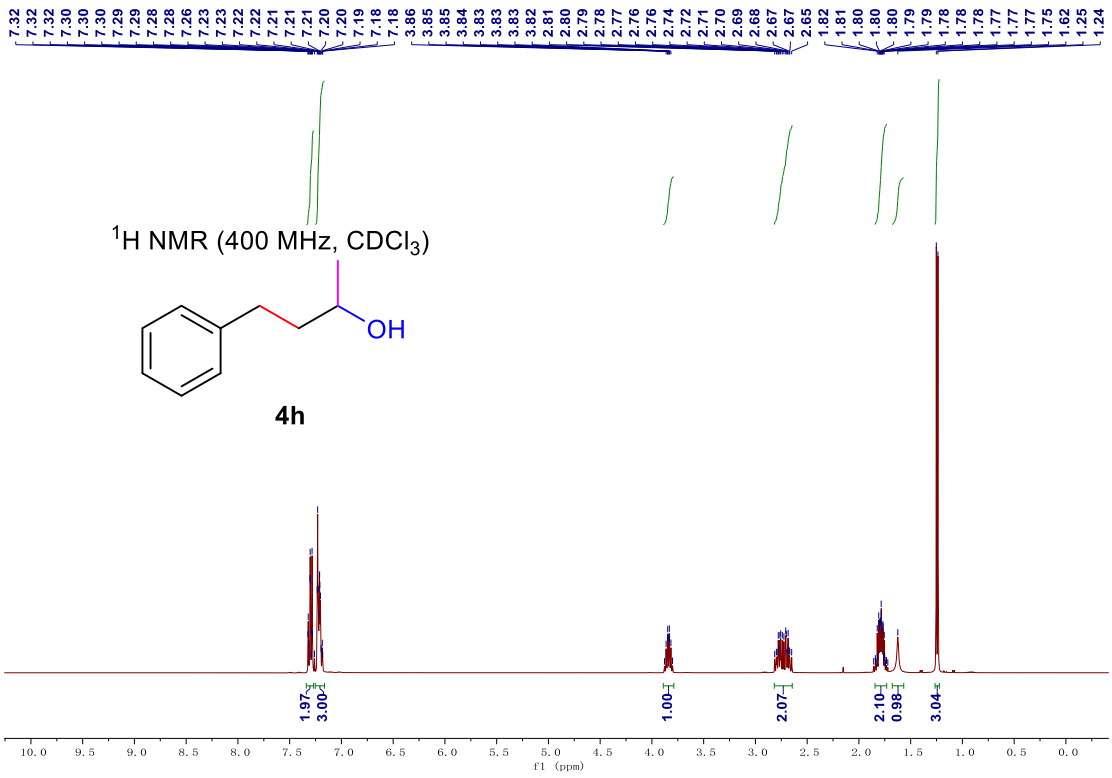

141.61
131.42
130.29
119.47
77.48
77.16
76.84
68.18
39.21
35.43
31.46
25.45
23.72
13C NMR (100 MHz, CDCl3)
OH
Br
4e
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

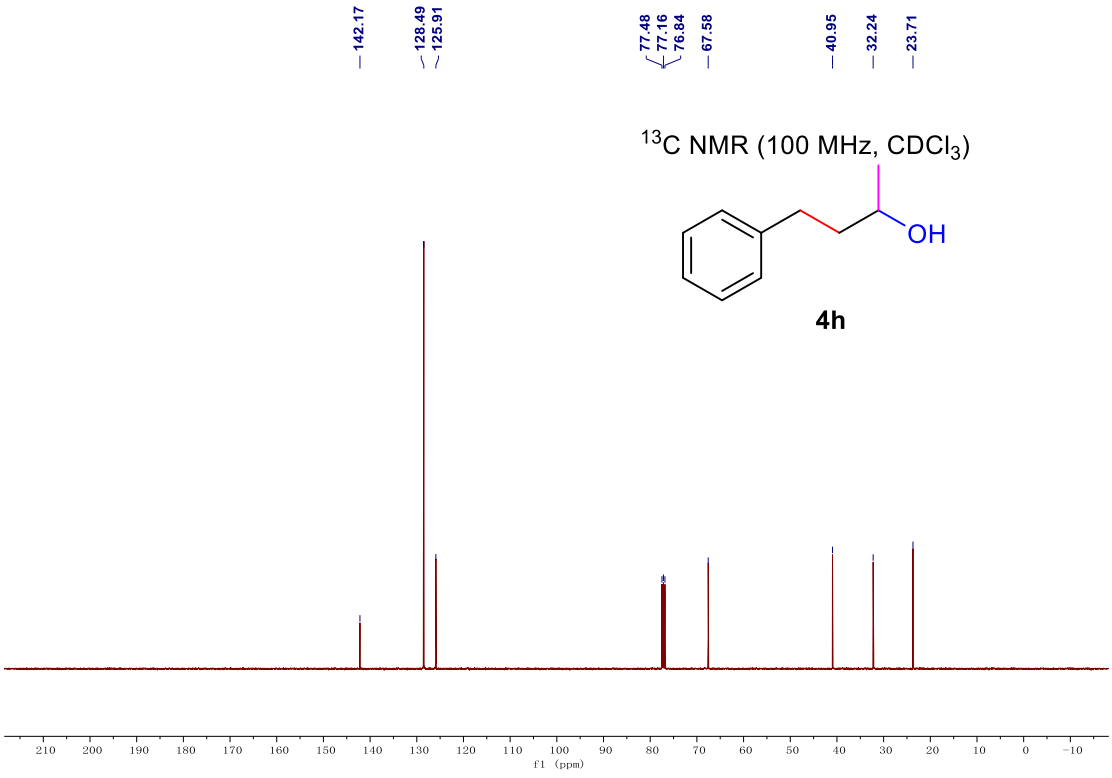

7.26
7.10
7.10
7.09
7.08
6.84
6.83
6.82
6.81
3.81
3.79
3.77
2.58
2.56
2.54
1.67
1.64
1.64
1.63
1.62
1.62
1.61
1.61
1.60
1.59
1.59
1.59
1.58
1.57
1.57
1.56
1.51
1.51
1.50
1.50
1.50
1.49
1.49
1.48
1.47
1.46
1.45
1.45
1.44
1.44
1.43
1.43
1.42
1.41
1.40
1.40
1.39
1.38
1.37
1.36
1.35
1.35
1.34
1.34
1.33
1.33
1.32
1.19
1.17
^{1}H NMR (400 MHz, $CDCl_3$)
OH
O
4f
2.00
2.01
4.06
2.06
2.28
5.03
3.03
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
f1 (ppm)

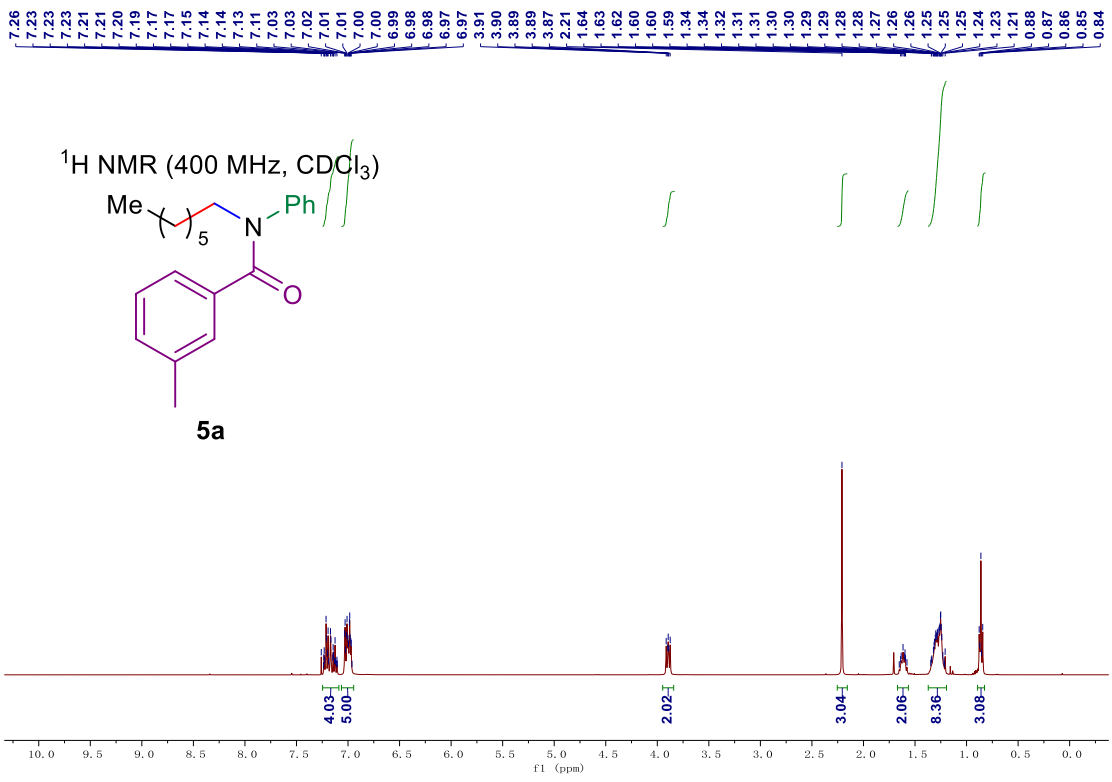

157.72
134.79
129.36
113.78
77.48
77.16
76.84
68.23
55.37
39.30
35.09
31.87
25.51
23.65
^{13}C NMR (100 MHz, $CDCl_3$)
OH
O
4f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

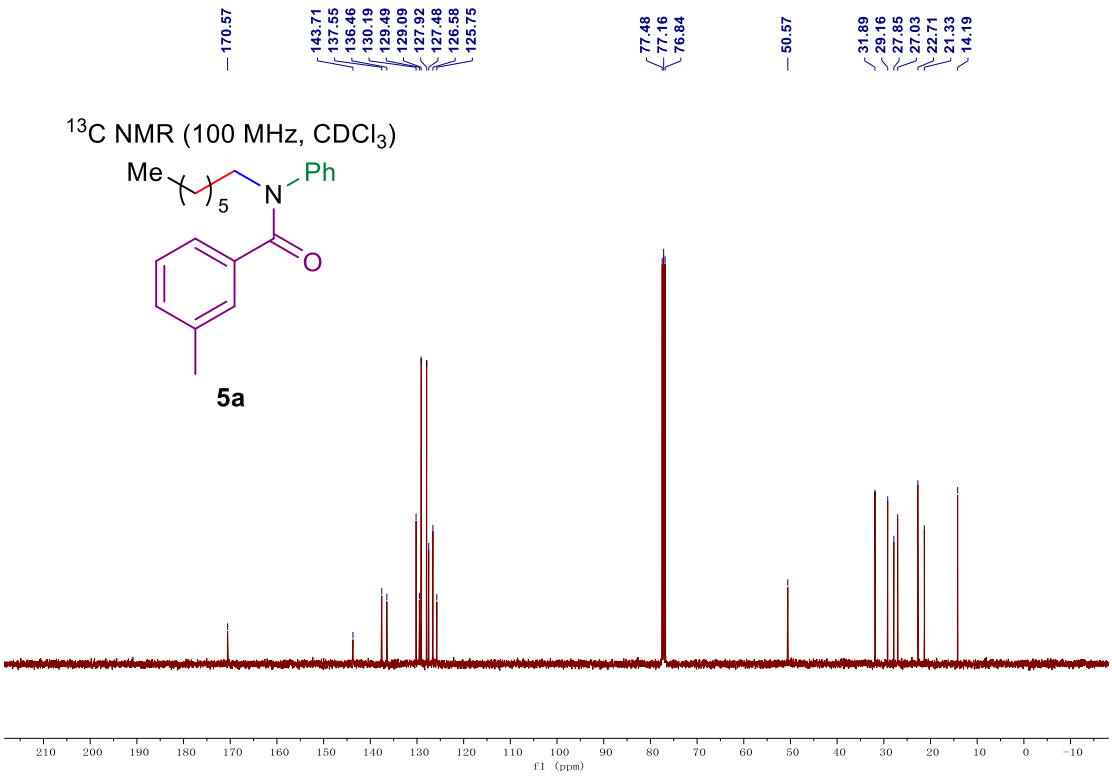

7.36 7.35 7.35 7.34 7.33 7.31 7.31 7.30 7.29 7.29 7.28 7.27 7.26 7.21 7.20 7.19 7.19 7.18 7.17 7.17 7.17 7.16 7.16
3.70 3.68 3.67 3.66 3.64 3.63 2.66 2.64 2.62 2.60 1.69 1.68 1.67 1.67 1.66 1.65 1.65 1.64 1.63 1.63 1.62 1.61 1.61 1.60 1.60 1.59 1.58 1.44 1.44 1.43 1.42 1.42 1.41 1.40 1.40 1.39 1.38 1.38 1.37 1.36
^{1}H NMR (400 MHz, $CDCl_3$)
OH
4g
2.16 3.09 1.98 1.96 5.12 2.05
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
f1 (ppm)

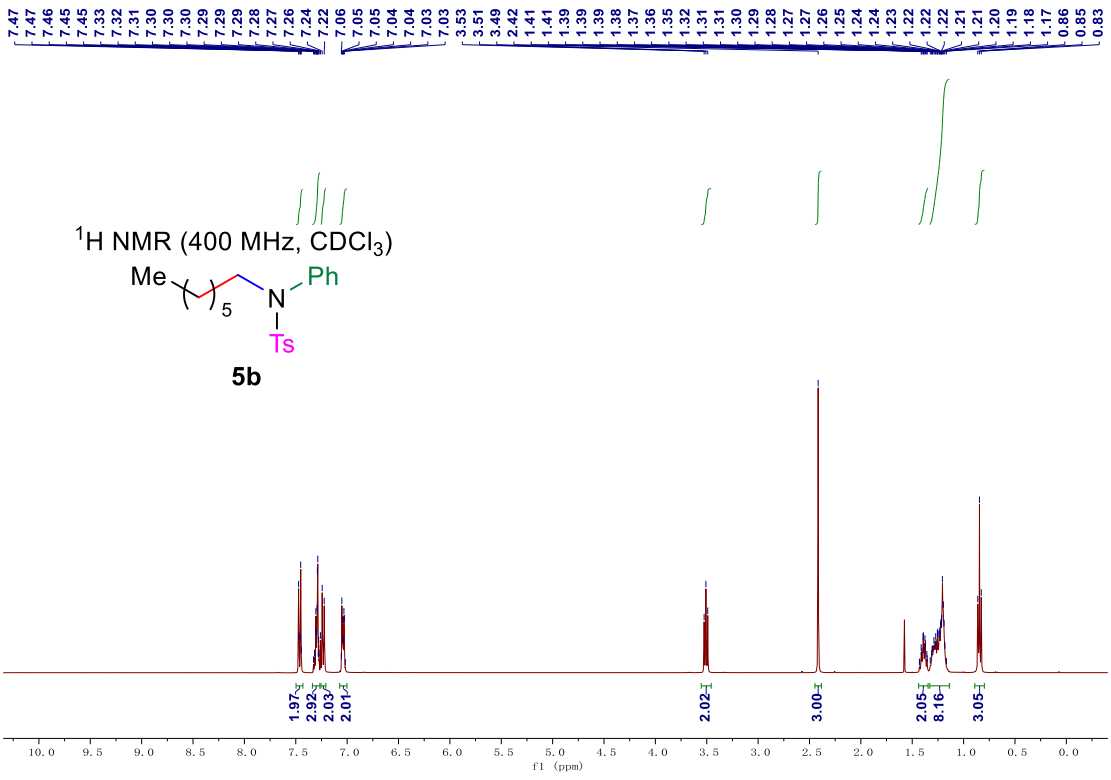

142.69
128.53 128.41 125.80
77.48 77.16 76.84
63.09
36.04 32.77 31.43 25.54
^{13}C NMR (100 MHz, $CDCl_3$)
OH
4g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

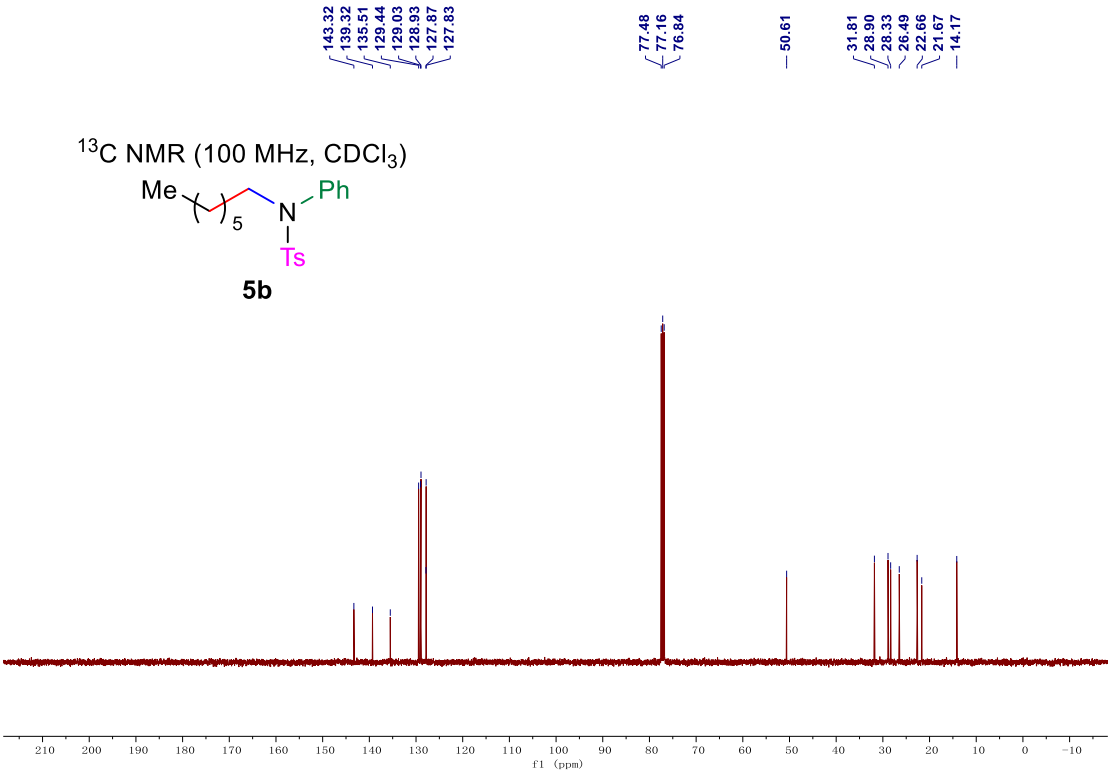

7.32 7.32 7.32 7.30 7.30 7.30 7.29 7.29 7.28 7.28 7.26 7.23 7.23 7.22 7.22 7.21 7.21 7.21 7.20 7.20 7.19 7.18 7.18
3.86 3.85 3.85 3.84 3.83 3.83 3.83 3.82 2.81 2.80 2.79 2.78 2.77 2.76 2.76 2.74 2.72 2.71 2.70 2.69 2.68 2.67 2.67 2.65
1.82 1.81 1.80 1.80 1.80 1.79 1.79 1.78 1.78 1.78 1.77 1.77 1.77 1.75 1.62 1.25 1.24
1H NMR (400 MHz, CDCl3)
OH
4h
1.97
3.00
1.00
2.07
2.10
0.98
3.04
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
f1 (ppm)

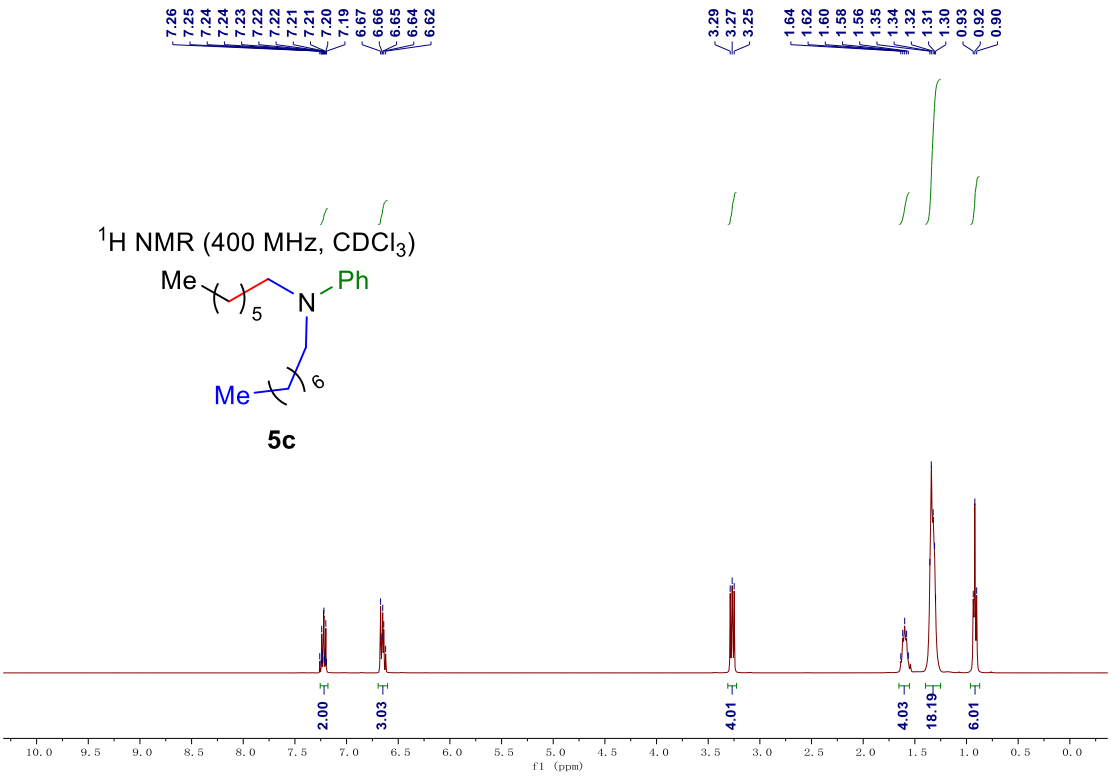

142.17
128.49
125.91
77.48
77.16
76.84
67.58
40.95
32.24
23.71
13C NMR (100 MHz, CDCl3)
OH
4h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.26
7.23
7.23
7.23
7.21
7.21
7.20
7.19
7.17
7.17
7.15
7.14
7.14
7.13
7.11
7.03
7.03
7.02
7.01
7.01
7.00
7.00
6.99
6.98
6.98
6.97
6.97
3.91
3.90
3.89
3.89
3.87
2.21
1.64
1.63
1.62
1.60
1.60
1.59
1.34
1.34
1.32
1.31
1.31
1.30
1.30
1.29
1.29
1.28
1.28
1.27
1.26
1.26
1.25
1.25
1.25
1.24
1.23
1.21
0.88
0.87
0.86
0.85
0.84
1H NMR (400 MHz, CDCl3)
Me
5
N
Ph
O
5a
4.03
5.00
2.02
3.04
2.06
8.36
3.08
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
f1 (ppm)


170.57
143.71
137.55
136.46
130.19
129.49
129.09
127.92
127.48
126.58
125.75
77.48
77.16
76.84
50.57
31.89
29.16
27.85
27.03
22.71
21.33
14.19
13C NMR (100 MHz, CDCl3)
Me
5
N
Ph
O
5a
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)

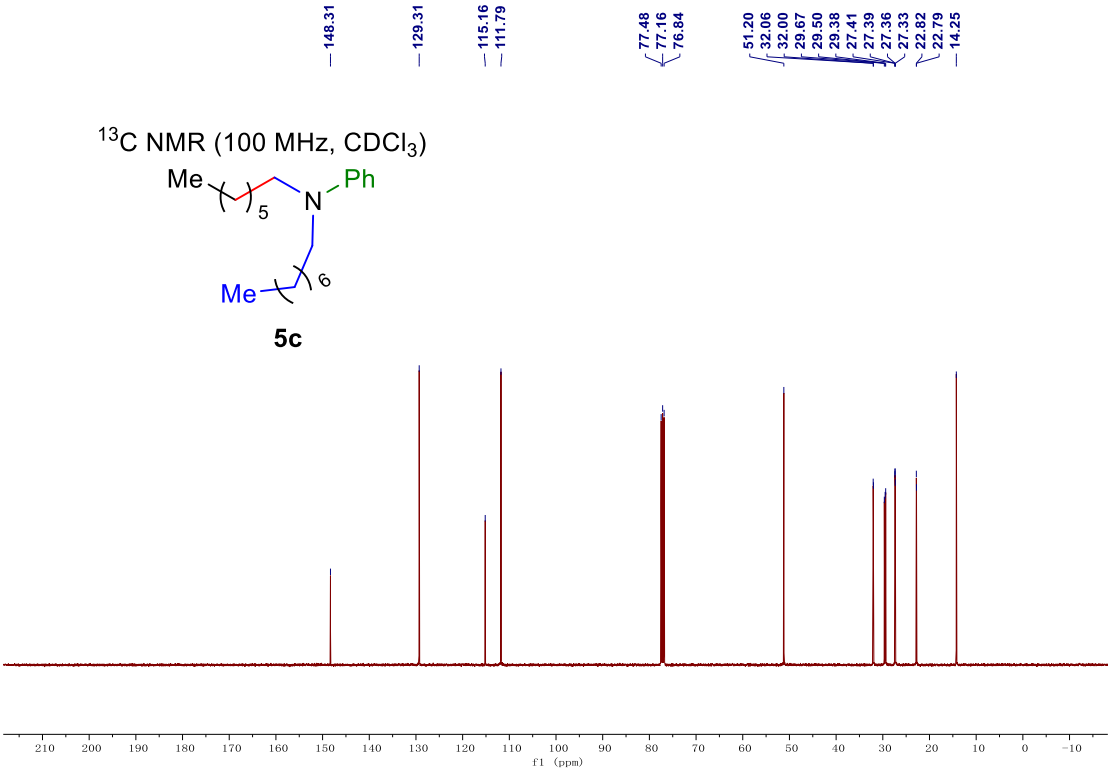

7.47 7.47 7.46 7.45 7.45 7.33 7.32 7.31 7.30 7.30 7.30 7.29 7.29 7.29 7.28 7.27 7.26 7.24 7.22 7.06 7.05 7.05 7.04 7.04 7.03 7.03 3.53 3.51 3.49 2.42 1.41 1.41 1.39 1.39 1.39 1.38 1.37 1.36 1.35 1.32 1.31 1.31 1.30 1.29 1.28 1.27 1.27 1.26 1.25 1.24 1.24 1.23 1.22 1.22 1.22 1.21 1.21 1.20 1.19 1.18 1.17 0.86 0.85 0.83
1H NMR (400 MHz, CDCl3)
Me
5
N
Ph
Ts
5b
1.97 2.92 2.03 2.01 2.02 3.00 2.05 8.16 3.05
f1 (ppm)


143.32 139.32 135.51 129.44 129.03 128.93 127.87 127.83 77.48 77.16 76.84 50.61 31.81 28.90 28.33 26.49 22.66 21.67 14.17
13C NMR (100 MHz, CDCl3)
Me
5
N
Ph
Ts
5b
f1 (ppm)



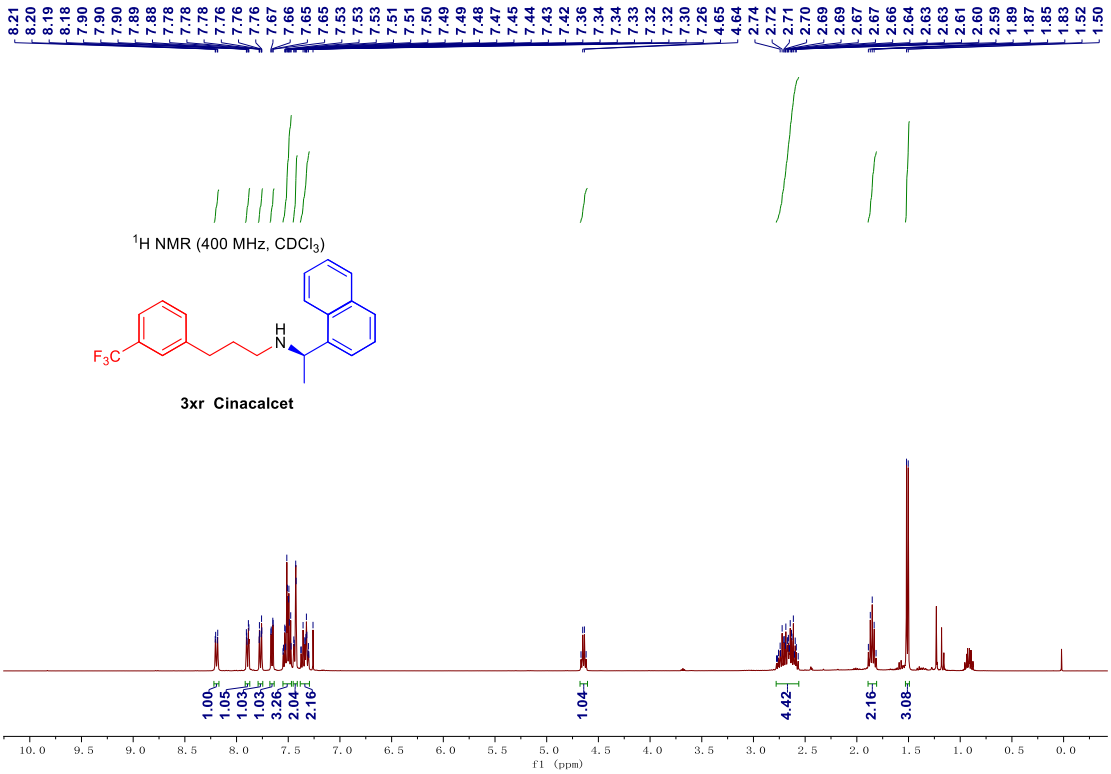
^{1}H NMR (400 MHz, $CDCl_3$)

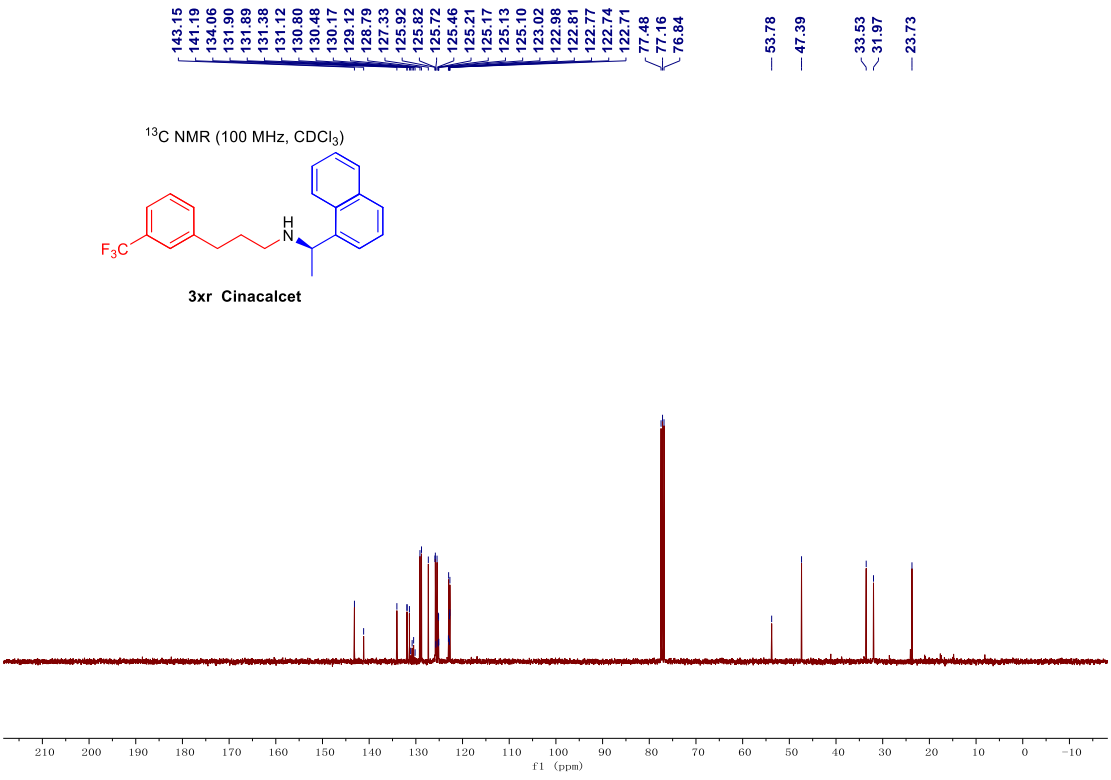
5c

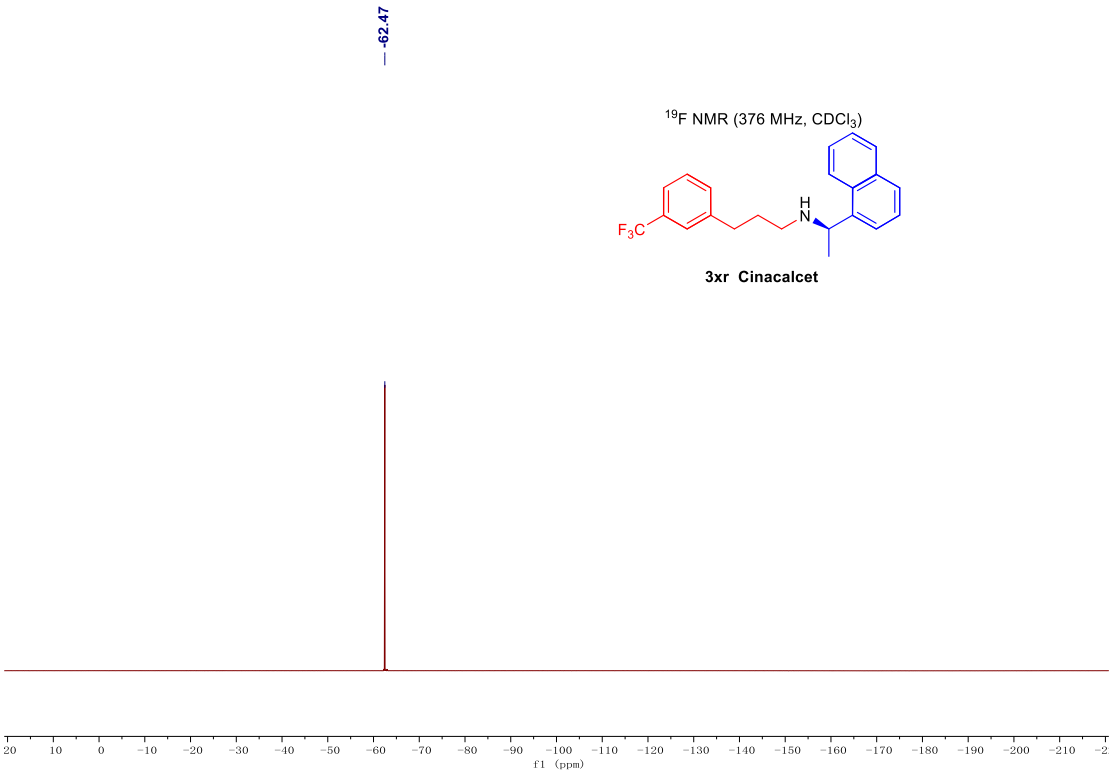


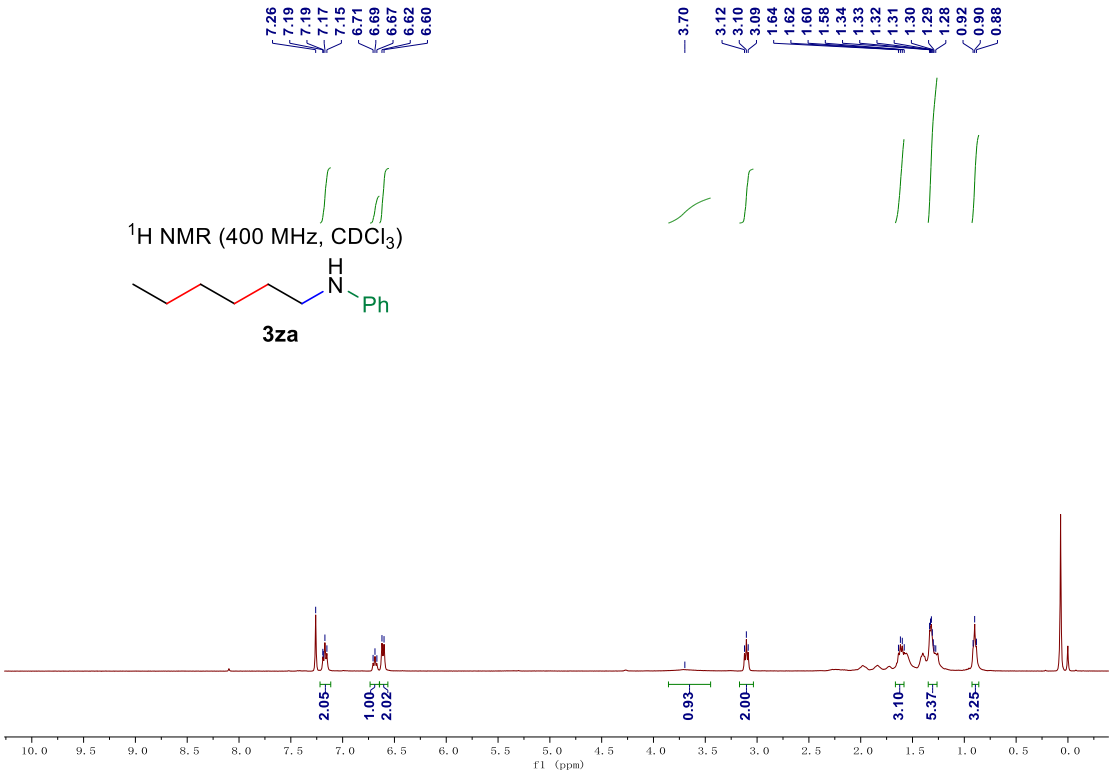
^{13}C NMR (100 MHz, $CDCl_3$)

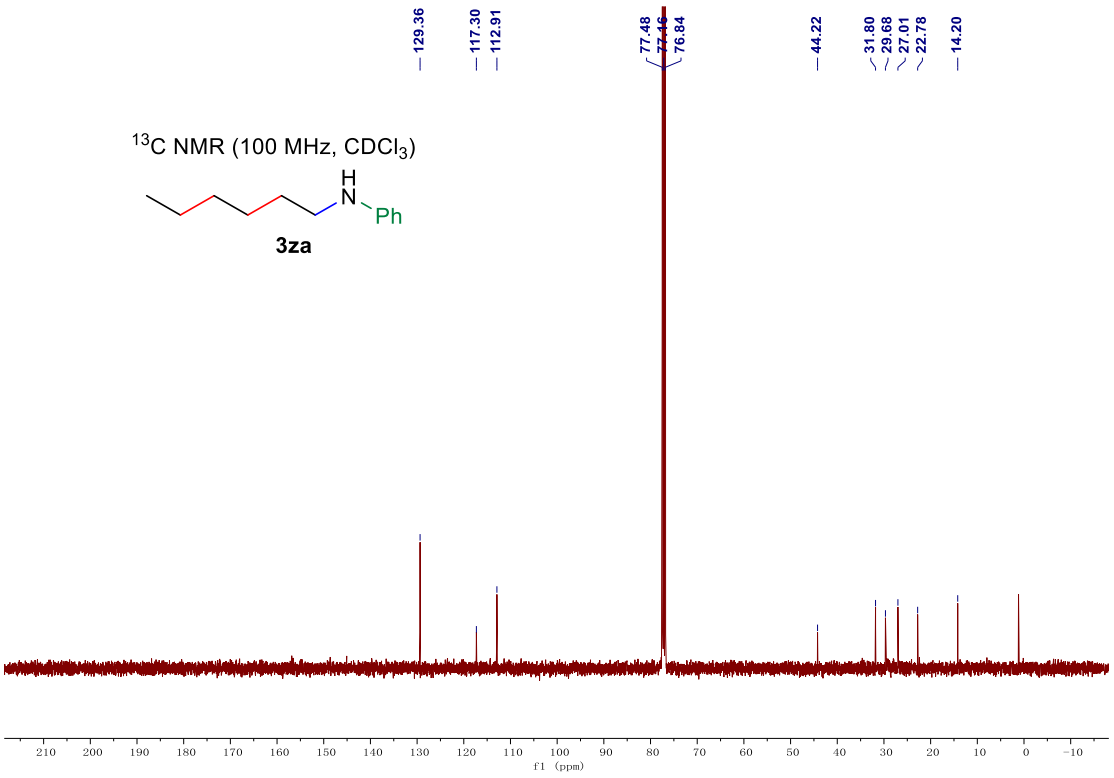
5c


8.21 8.20 8.19 8.18 7.90 7.90 7.90 7.89 7.88 7.78 7.78 7.78 7.76 7.76 7.76 7.67 7.66 7.65 7.65 7.53 7.53 7.53 7.51 7.51 7.50 7.49 7.49 7.48 7.47 7.45 7.44 7.43 7.42 7.36 7.34 7.34 7.33 7.32 7.32 7.30 7.26 4.65 4.64 2.74 2.72 2.71 2.70 2.69 2.69 2.67 2.67 2.66 2.64 2.63 2.63 2.61 2.60 2.59 1.89 1.87 1.85 1.83 1.52 1.50
1H NMR (400 MHz, CDCl3)
F3C
H
N
3xr Cinacalcet
1.00 1.05 1.03 1.03 3.26 2.04 2.16 1.04 4.42 2.16 3.08
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
f1 (ppm)


143.15 141.19 134.06 131.90 131.89 131.38 131.12 130.80 130.48 130.17 129.12 128.79 127.33 125.92 125.82 125.72 125.46 125.21 125.17 125.13 125.10 123.02 122.98 122.81 122.77 122.74 122.71 77.48 77.16 76.84 53.78 47.39 33.53 31.97 23.73
13C NMR (100 MHz, CDCl3)
F3C
H
N
3xr Cinacalcet
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)

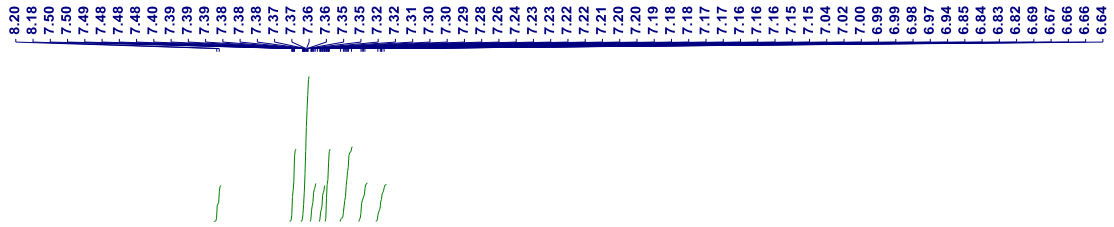

-62.47
19F NMR (376 MHz, CDCl3)
F3C
H
N
3xr Cinacalcet
f1 (ppm)


7.26
7.19
7.19
7.17
7.15
6.71
6.69
6.67
6.62
6.60
3.70
3.12
3.10
3.09
1.64
1.62
1.60
1.58
1.34
1.33
1.32
1.31
1.30
1.29
1.28
0.92
0.90
0.88
1H NMR (400 MHz, CDCl3)
H
N
Ph
3za
2.05
1.00
2.02
0.93
2.00
3.10
5.37
3.25
f1 (ppm)

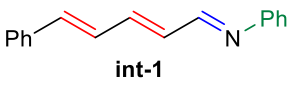


^{13}C NMR (100 MHz, $CDCl_3$)

3za



^{1}H NMR (400 MHz, $CDCl_3$)



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