Supporting Information

Indium-mediated difunctionalization of iodoalkyl-tethered unactivated alkenes via an intramolecular cyclization and an ensuing palladium-catalyzed cross-coupling reaction with aryl halide

Xuan-Di Song,[a] Xiang-Rui Li,[a] Ya-Wen Wang,[a] Xue-Qiang Chu,[a] Weidong Rao,[b] Haiyan Xu,[c] Guo-Zhi Han,[a,*] and Zhi-Liang Shen[a,*]

[a] Technical Institute of Fluorochemistry (TIF), Institute of Advanced Synthesis, School of Chemistry and Molecular Engineering, Nanjing Tech University, Nanjing 211816, China

[b] Jiangsu Key Laboratory of Biomass-based Green Fuels and Chemicals, College of Chemical Engineering, Nanjing Forestry University, Nanjing 210037, China

[c] School of Environmental and Chemical Engineering, Jiangsu University of Science and Technology, Zhenjiang, Jiangsu 212003, China

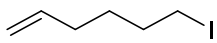
Table of Contents

General information

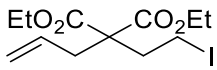
Commercially available aryl halides were used without further purification. Starting materials **1a-k** were prepared according to reported methods. Analytical grade THF and DMA were used in all the reactions without purification (without the need of precautions to exclude air and moisture unless otherwise noted). Indium powder, metallic salt, palladium catalyst, and lithium chloride were purchased from chemical companies and used directly without further purification. Analytical thin layer chromatography (TLC) was performed using silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm). Flash chromatography was performed using Merck silica gel (200-300 mesh) for column chromatography with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use. IR spectra were recorded on a FT-IR spectrophotometer using KBr optics. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on Jeol 400 MHz spectrometerss. Tetramethylsilane (TMS) served as internal standard for ^{1}H and ^{13}C NMR analysis.

Experimental procedure

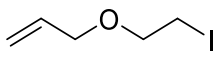
General procedure for the synthesis of alkene-tethered alkyl iodides



6-Iodohex-1-ene (1a): This compound was synthesized using 6-bromohex-1-ene according to the described procedure.[1] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 5.79 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.04-4.95 (m, 2H), 3.19 (t, *J* = 7.0 Hz, 2H), 2.10-2.03 (m, 2H), 1.86-1.79 (m, 2H), 1.57-1.44 (m, 2H) ppm.

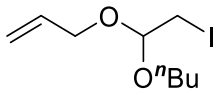


Diethyl 2-allyl-2-(2-iodoethyl)malonate (1b): This compound was synthesized using diethyl 2-allylmalonate and 1,2-dibromoethane according to the described procedure.[2] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 5.62 (ddt, *J* = 16.6, 10.6, 7.4 Hz, 1H), 5.14-5.08 (m, 2H), 4.18 (q, *J* = 7.1 Hz, 4H), 3.10- 3.06 (m, 2H), 2.62 (d, *J* = 7.4 Hz, 2H), 2.48-2.43 (m, 2H), 1.24 (t, *J* = 7.1 Hz, 6H) ppm.

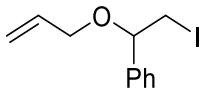


3-(2-Iodoethoxy)prop-1-ene (1c): This compound was synthesized using 2-(allyloxy)ethan-1-ol according to the described procedure.[3] Colorless oil. **^{1}H NMR**

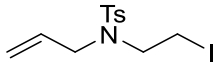
(400 MHz, $CDCl_3$): δ 5.95-5.85 (m, 1H), 5.32-5.18 (m, 2H), 4.03 (d, *J* = 5.6 Hz, 2H), 3.69 (t, *J* = 6.8 Hz, 2H), 3.25 (t, *J* = 6.8 Hz, 2H) ppm.



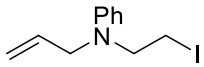
1-(1-(Allyloxy)-2-iodoethoxy)butane (1d): This compound was synthesized using 3-propoxyprop-1-ene and prop-2-en-1-ol according to the described procedure.[4] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 5.89 (ddt, *J* = 17.2, 10.4, 5.6 Hz, 1H), 5.29 (dq, *J* = 17.2, 1.7 Hz, 1H), 5.17 (dq, *J* = 10.4, 1.4 Hz, 1H), 4.63 (t, *J* = 5.5 Hz, 1H), 4.12 (ddt, *J* = 12.8, 5.4, 1.5 Hz, 1H), 4.03 (ddt, *J* = 12.8, 5.9, 1.4 Hz, 1H), 3.58 (dt, *J* = 9.3, 6.6 Hz, 1H), 3.46 (dt, *J* = 9.3, 6.6 Hz, 1H), 3.21 (d, *J* = 5.5 Hz, 2H), 1.61-1.50 (m, 2H), 1.44-1.31 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H) ppm.



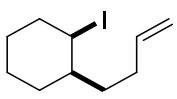
(1-(Allyloxy)-2-iodoethyl)benzene (1e): This compound was synthesized by the reaction of styrene with NIS and allyl alcohol according to the described procedure.[5] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.40-7.31 (m, 5H), 5.99-5.89 (m, 1H), 5.32-5.18 (m, 2H), 4.48 (dd, *J* = 8.2, 4.8 Hz, 1H), 4.01 (dd, *J* = 12.8, 5.2 Hz, 1H), 3.85 (dd, *J* = 12.8, 6.2 Hz, 1H), 3.38 (dd, *J* = 10.4, 8.2 Hz, 1H), 3.33 (dd, *J* = 10.4, 4.8 Hz, 1H) ppm.



***N*-Allyl-*N*-(2-iodoethyl)-4-methylbenzenesulfonamide (1f):** This compound was synthesized using 2-aminoethan-1-ol and 3-bromoprop-1-ene according to the described procedure.[6] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.72-7.68 (m, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 5.68 (ddt, *J* = 17.5, 9.8, 6.5 Hz, 1H), 5.24-5.15 (m, 2H), 3.79 (d, *J* = 6.5 Hz, 2H), 3.42 (dd, *J* = 9.5, 6.9 Hz, 2H), 3.23 (dd, *J* = 9.3, 6.6 Hz, 2H), 2.44 (s, 3H) ppm.

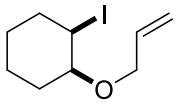


***N*-Allyl-*N*-(2-iodoethyl)aniline (1g):** This compound was synthesized using 2-(phenylamino)ethan-1-ol and 3-bromoprop-1-ene according to the described procedure.[7] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.25-7.20 (m, 2H), 6.75-6.71 (m, 1H), 6.68-6.66 (m, 2H), 5.85 (dddd, *J* = 17.6, 9.9, 5.2, 4.7 Hz, 1H), 5.20-5.14 (m, 2H), 3.96 (d, *J* = 5.2 Hz, 2H), 3.73-3.69 (m, 2H), 3.25-3.21 (m, 2H) ppm.



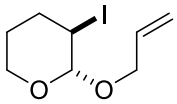
2-(But-3-en-1-yl)cyclohexan-1-ol: This compound was synthesized using cyclohexene oxide and 3-butenylmagnesium bromide according to the described procedure.[8]

***cis*-1-(But-3-en-1-yl)-2-iodocyclohexane (1h):** This compound was synthesized using 2-(but-3-en-1-yl)cyclohexan-1-ol according to the described procedure.[9] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 5.79 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.04-4.94 (m, 2H), 4.71-4.70 (m, 1H), 2.22-1.94 (m, 3H), 1.78-1.69 (m, 3H), 1.60-1.43 (m, 2H), 1.39-1.22 (m, 4H), 0.48-0.41 (m, 1H). The relative stereochemistry of the structure was confirmed by comparing with the NMR data of the same compound reported earlier.[10]

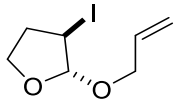


2-(Allyloxy)cyclohexan-1-ol: This compound was synthesized using 7-oxabicyclo[4.1.0]heptane and allyl alcohol according to the described procedure.[11]

***cis*-1-(allyloxy)-2-iodocyclohexane (1i):** This compound was synthesized using 2-(allyloxy)cyclohexan-1-ol according to the described procedure.[12] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 5.95 (ddt, *J* = 17.4, 10.5, 5.6 Hz, 1H), 5.34-5.28 (m, 1H), 5.19-5.15 (m, 1H), 4.67 (dt, *J* = 6.5, 3.3 Hz, 1H), 4.07 (ddq, *J* = 12.8, 5.7, 1.4 Hz, 1H), 3.98 (ddq, *J* = 12.8, 5.7, 1.4 Hz, 1H), 2.78-2.65 (m, 1H), 2.25-2.13 (m, 1H), 1.85-1.61 (m, 4H), 1.52-1.19 (m, 3H) ppm.



***trans*-2-Allyloxy3-iodotetrahydro-2*H*-pyran (1j):** This compound was synthesized using allyl alcohol and 3,4-dihydro-2*H*-pyran according to the described procedure.[13] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 5.99-5.88 (m, 1H), 5.32 (dq, *J* = 17.3, 1.7 Hz, 1H), 5.20 (dq, *J* = 10.5, 1.4 Hz, 1H), 4.68 (d, *J* = 5.4 Hz, 1H), 4.26 (dd, *J* = 12.7, 5.4 Hz, 1H), 4.14-3.96 (m, 3H), 3.59 (ddd, *J* = 11.3, 7.7, 3.6 Hz, 1H), 2.43-2.34 (m, 1H), 2.07-1.97 (m, 1H), 1.82-1.73 (m, 1H), 1.63-1.53 (m, 1H) ppm. The relative stereochemistry of the structure was confirmed by comparing with the NMR data of the same compound reported earlier.[14]



***trans*-2-Allyloxy-3-iodotetrahydrofuran (1k):** This compound was synthesized using 2,3-dihydrofuran and allyl alcohol according to the described procedure.[15] Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 5.87 (dddd, *J* = 17.3, 10.4, 6.1, 5.2 Hz, 1H), 5.39 (s, 1H), 5.27 (dq, *J* = 17.3, 1.7 Hz, 1H), 5.18 (dq, *J* = 10.4, 1.4 Hz, 1H), 4.22-4.08 (m, 3H), 4.03 (td, *J* = 8.3, 3.6 Hz, 1H), 3.97 (ddt, *J* = 12.8, 6.1, 1.4 Hz, 1H), 2.63 (dtd, *J* = 14.4,

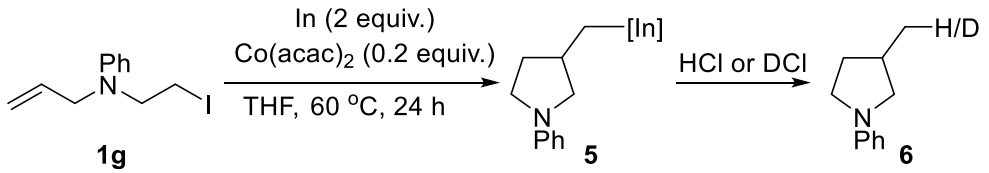
8.3, 6.3 Hz, 1H), 2.19 (dddd, J = 14.0, 7.0, 3.6, 2.2 Hz, 1H) ppm. The relative stereochemistry of the structure was confirmed by comparing with the NMR data of the same compound reported earlier.[14]

General procedure for the cyclization/cross-coupling sequence (Tables 2-3)

***Step 1*:** Alkyl iodide (0.6 mmol), indium (137.8 mg, 1.2 mmol), cobalt(II) acetylacetonate (42.8 mg, 0.12 mmol), and analytical grade THF (2 mL) was added in a flask equipped with a septum and a magnetic stir bar. The reaction mixture was vigorously stirred at 60 ^{o}C for 24 hrs. Then the upper clear solution was carefully separated from the bottom black precipitate by centrifugal. The remaining black precipitate was additionally stirred with THF (3 mL), and the THF layer was carefully separated from bottom precipitate by pipette. The combined organic layers were concentrated under vacuum. The crude mixture was directly used in the next step without further purification.

Step 2: To the above residue was added aryl halide (0.42 mmol), LiCl (50.9 mg, 1.2 mmol), $Pd(PPh_3)_4$ (34.7 mg, 0.03 mmol), and DMA (2 mL), and the reaction mixture was stirred at 100 ^{o}C for 12 hrs. Upon completion of the reaction, the reaction mixture was directly purified by flash silica gel column chromatography using petroleum ether/ethyl acetate as eluent to afford the pure products.

Control experiment



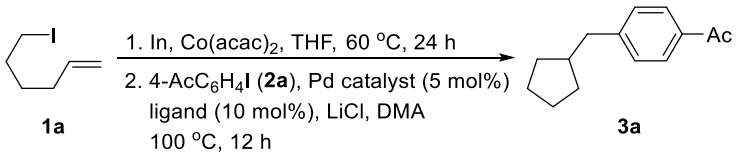
acid	**6**
HCl (1 M) in H_2O (2 mL)	47% yield
DCl (20 wt%) in D_2O (1 mL)	52% yield (71% D)

N-Allyl-*N*-(2-iodoethyl)aniline (**1g**, 172.3 mg, 0.6 mmol), indium (137.8 mg, 1.2 mmol), cobalt(II) acetylacetonate (42.8 mg, 0.12 mmol), and analytical grade THF (2 mL) was added in a flask equipped with a septum and a magnetic stir bar. The reaction mixture was vigorously stirred at 60 ^{o}C for 24 hrs. HCl (2 mL; 1 M in H_2O) or DCl (1 mL; 20 wt% in D_2O) was added into the reaction and the solution was stirred at 60 ^{o}C for 2 hrs followed by the addition of saturated aqueous $NaHCO_3$ (20 mL). The reaction mixture was extracted with EtOAc (20 mL × 3), washed with brine, dried over

anhydrous Na_2SO_4, and evaporated to dryness. Purification of the residue by flash silica gel column chromatography using petroleum ether/ethyl acetate (200:1) as eluent afforded the 3-methyl-1-phenylpyrrolidine **6** (aq. HCl: 45.5 mg, 47% yield; DCl in D_2O: 50.6 mg, 52% yield (71% D)) as colorless liquid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.26-7.22 (m, 2H), 6.66 (t, *J* = 7.3 Hz, 1H), 6.55 (d, *J* = 7.8 Hz, 2H), 3.45 (dd, *J* = 9.0, 7.2 Hz, 1H), 3.40-3.28 (m, 2H), 2.87 (dd, *J* = 8.8, 7.7 Hz, 1H), 2.46-2.34 (m, 1H), 2.17-2.10 (m, 1H), 1.63 (dq, *J* = 12.1, 8.3 Hz, 1H), 1.14 (d, *J* = 6.6 Hz, 3H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 147.9, 129.1, 115.2, 111.3, 54.9, 47.4, 33.5, 33.3, 18.4 ppm.

Optimization of reaction conditions by using various Pd catalysts

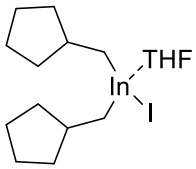
Table S1 Optimization of reaction conditions by using various Pd catalysts and ligands for cross-coupling reactions[a]



Entry	Catalyst	Ligand	Yield (%)[b]
1	$Pd(acac)_2$	-	37
2	$Pd(PPh_3)_2Cl_2$	-	70
3	$[PdCl(allyl)]_2$	-	40
4	$Pd_2(dba)_3 \cdot CHCl_3$	-	42
5	**$Pd(PPh_3)_4$**	**-**	**71**
6	$Pd(TFA)_2$	S-Phos	51
7	$Pd(OAc)_2$	S-Phos	38
8	$Pd(OAc)_2$	X-Phos	35
9	$Pd(OAc)_2$	DPEPhos	61
10	$Pd(OAc)_2$	XantPhos	63
11	$Pd(OAc)_2$	BrettPhos	38
12	$Pd(OAc)_2$	DPPP[c]	65
13	$Pd(OAc)_2$	DPPE[d]	43
14	$Pd(OAc)_2$	TTMPP[e]	59
15	$Pd(OAc)_2$	PCy_3	62

[a] The 1st step was performed at 60 °C for 24 h by using 6-iodohex-1-ene (**1a**, 0.6 mmol), indium powder (1.2 mmol), and $Co(acac)_2$ (0.12 mmol) in THF (2 mL). The 2nd cross-coupling step was performed at 100 °C for 12 h by using 4-AcC_6H_4I (**2a**, 0.42 mmol), LiCl (1.2 mmol), Pd catalyst (0.03 mmol), ligand (0.06 mmol), and DMA (2 mL). [b] The yield was determined by ^{1}H NMR analysis of the crude reaction mixture by using 1,4-dimethoxybenzene as an internal standard. [c] DPPP = 1,3-bis(diphenyphosphino)propane. [d] DPPE = 1,2-bis(diphenylphosphino)ethane. [e] TTMPP = tris(2,4,6-trimethoxyphenyl)phosphine.

ESI-MS data of possible alkyl indium reagent



ESI-MS data of A

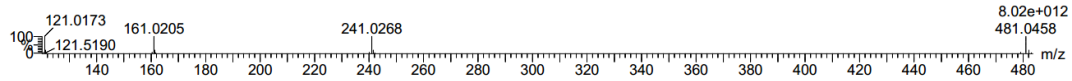
HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{16}H_{31}IInO^+$: 481.0453, found: 481.0458.

Elemental Composition Report **Page 1**

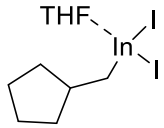
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
172 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 14-17 H: 29-32 O: 0-2 Cl: 0-8 Br: 0-8 In: 0-3 I: 1-3

WAC0331 (0.918) Is (1.00,1.00) C16H30IInO
1: TOF MS ES+



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
481.0458	481.0464	-0.6	-1.2	-0.5	67.1	0.000	100.00	C15 H31 O I2
	481.0458	0.0	0.0	1.5	115.2	48.067	0.00	C16 H31 O In I



ESI-MS data of B

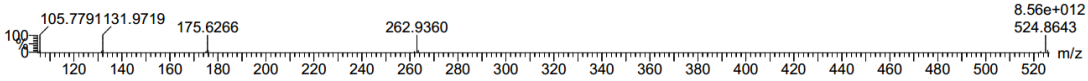
HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{10}H_{20}I_2InO^+$: 524.8637, found: 524.8643.

Elemental Composition Report **Page 1**

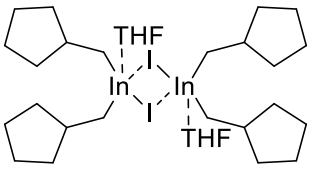
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
218 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 9-11 H: 19-21 O: 0-2 Cl: 0-8 Br: 0-8 In: 0-3 I: 1-3

WAC0331 (1.252) Is (1.00,1.00) C10H19I2InO
1: TOF MS ES+



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
524.8643	524.8642	0.1	0.2	0.5	63.2	0.000	100.00	C10 H20 O In I2
	524.8648	-0.5	-1.0	-1.5	85.6	22.383	0.00	C9 H20 O I3
	524.8636	0.7	1.3	2.5	128.6	65.309	0.00	C11 H20 O In2 I



ESI-MS data of C

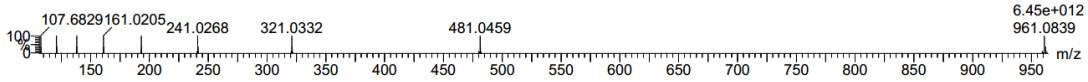
HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{32}H_{61}I_2In_2O_2^+$: 961.0833, found: 961.0839.

Elemental Composition Report **Page 1**

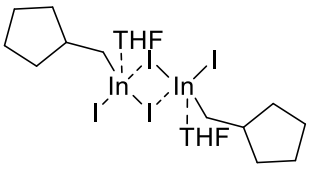
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Odd and Even Electron Ions
1866 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 31-33 H: 60-62 O: 0-3 Cl: 0-8 Br: 0-8 In: 0-3 I: 1-3

WAC0331 (0.807) Is (1.00,1.00) C32H60I2In2O2
1: TOF MS ES+



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
961.0839	961.0844	-0.5	-0.5	0.5	119.2	0.000	100.00	C31 H61 O2 In I3
	961.0833	0.6	0.6	4.5	183.8	64.606	0.00	C33 H61 O2 In3 I
	961.0839	0.0	0.0	2.5	183.8	64.636	0.00	C32 H61 O2 In2 I2



ESI-MS data of D

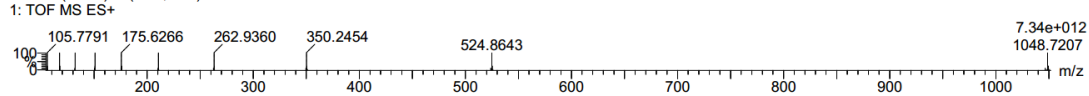
HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{20}H_{39}I_4In_2O_2^+$: 1048.7201, found: 1048.7207.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

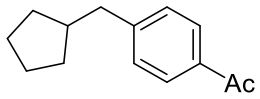
Monoisotopic Mass, Even Electron Ions
1336 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 18-21 H: 37-40 O: 1-3 Cl: 0-8 Br: 0-8 In: 0-3 I: 2-5

SXD-3 (3.469) Is (1.00,1.00) C20H38I4In2O2
1: TOF MS ES+

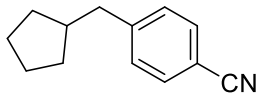


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
1048.7207	1048.7212	-0.5	-0.5	-1.5	117.2	0.000	100.00	C19 H39 O2 In I5
	1048.7200	0.7	0.7	2.5	182.0	64.853	0.00	C21 H39 O2 In3 I3
	1048.7206	0.1	0.1	0.5	182.1	64.880	0.00	C20 H39 O2 In2 I4

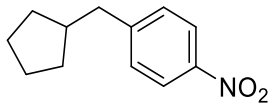
Characterization data of products



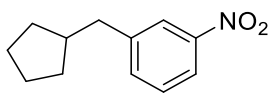
1-(4-(Cyclopentylmethyl)phenyl)ethan-1-one (3a): 54.6 mg. Yield = 64%. Colorless oil. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 7.95-7.77 (m, 2H), 7.33-7.20 (m, 2H), 2.66 (d, *J* = 7.5 Hz, 2H), 2.58 (s, 3H), 2.08 (hept, *J* = 7.5 Hz, 1H), 1.76-1.58 (m, 4H), 1.57-1.42 (m, 2H), 1.25-1.09 (m, 2H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** δ 197.8, 148.2, 134.8, 128.9, 128.3, 42.0, 41.7, 32.4, 26.5, 24.8 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{19}O$: 203.1430, found: 203.1436. **FTIR (KBr, neat):** *ν* 2951, 1683, 1606, 1358, 1268, 1017, 853, 818 cm^{-1}.



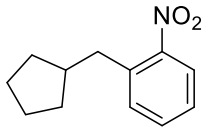
4-(Cyclopentylmethyl)benzonitrile (3b): 53.2 mg. Yield = 68%. Colorless oil. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 7.60-7.51 (m, 2H), 7.31-7.22 (m, 2H), 2.66 (d, *J* = 7.5 Hz, 2H), 2.07 (hept, *J* = 7.5 Hz, 1H), 1.75-1.58 (m, 4H), 1.58-1.46 (m, 2H), 1.23-1.12 (m, 2H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** 148.0, 132.0, 129.5, 119.2, 109.4, 42.1, 41.6, 32.3, 24.8 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{13}H_{16}N$: 186.1277, found: 186.1279. **FTIR (KBr, neat):** ν 2951, 2227, 1629, 1606, 1507, 804 cm^{-1}.



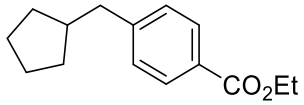
1-(Cyclopentylmethyl)-4-nitrobenzene (3c): 61.0 mg. Yield = 71%. Colorless oil. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 8.17-8.08 (m, 2H), 7.35-7.27 (m, 2H), 2.71 (d, *J* = 7.5 Hz, 2H), 2.10 (hept, *J* =7.5 Hz, 1H), 1.76-1.59 (m, 4H), 1.59-1.46 (m, 2H), 1.24-1.12 (m, 2H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** δ 150.3, 146.2, 129.5, 123.4, 41.9, 41.6, 32.4, 24.8 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{12}H_{16}NO_2$: 206.1176, found: 206.1181. **FTIR (KBr, neat):** ν 2951, 1598, 1518, 1346, 858, 803 cm^{-1}.



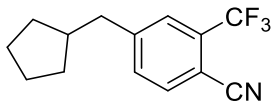
1-(Cyclopentylmethyl)-3-nitrobenzene (3d): 66.9 mg. Yield = 78%. Colorless oil. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 8.04-8.01 (m, 2H), 7.50-7.48 (m, 1H), 7.44-7.40 (m, 1H), 2.71 (d, *J* = 7.5 Hz, 2H), 2.11 (hept, *J* = 7.5 Hz, 1H), 1.77-1.60 (m, 4H), 1.59-1.47 (m, 2H), 1.23-1.14 (m, 2H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** δ 148.2, 144.3, 135.0, 128.9, 123.4, 120.8, 41.6, 32.3, 24.8 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. For $C_{12}H_{16}NO_2$: 206.1176, found: 206.1180. **FTIR (KBr, neat):** ν 2951, 1528, 1351, 811, 735 cm^{-1}.



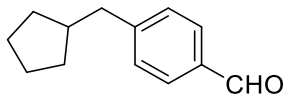
1-(Cyclopentylmethyl)-2-nitrobenzene (3e): 52.6 mg. Yield = 61%. Colorless oil. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 7.86-7.83 (m, 1H), 7.53-7.46 (m, 1H), 7.36-7.29 (m, 2H), 2.92 (d, *J* = 7.3 Hz, 2H), 2.17-2.04 (m, 1H), 1.73-1.58 (m, 4H), 1.58-1.45 (m, 2H), 1.25-1.12 (m, 2H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** δ 149.6, 136.9, 132.4, 132.2, 126.8, 124.5, 40.9, 38.4, 32.5, 24.7 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{12}H_{16}NO_2$: 206.1176, found: 206.1181. **FTIR (KBr, neat):** ν 2951, 1526, 1628, 1351, 742 cm^{-1}.



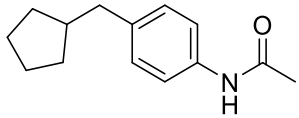
Ethyl 4-(cyclopentylmethyl)benzoate (3f): 64.2 mg. Yield = 66%. Colorless oil. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 7.98-7.92 (m, 2H), 7.25-7.21 (m, 2H), 4.36 (q, *J* = 7.1 Hz, 2H), 2.66 (d, *J* = 7.5 Hz, 2H), 2.16-2.02 (m, 1H), 1.75-1.58 (m, 4H), 1.57-1.46 (m, 2H), 1.38 (t, *J* = 7.1 Hz, 3H), 1.24-1.12 (m, 2H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** δ 166.7, 147.8, 129.5, 128.7, 127.9, 60.7, 42.1, 41.7, 32.4, 24.9, 14.3 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{15}H_{21}O_2$: 232.1536, found: 232.1539. **FTIR (KBr, neat):** ν 2952, 1719, 1628, 1275, 854, 802 cm^{-1}.



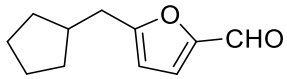
4-(Cyclopentylmethyl)-2-(trifluoromethyl)benzonitrile (3g): 49.8 mg. Yield = 47%. Colorless oil. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 7.74 (d, *J* = 7.9 Hz, 1H), 7.60-7.56 (m, 1H), 7.48-7.45 (m, 1H), 2.74 (d, *J* = 7.5 Hz, 2H), 2.15-2.03 (m, 1H), 1.78-1.61 (m, 4H), 1.61-1.50 (m, 2H), 1.23-1.11 (m, 2H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** δ 148.7, 134.6, 132.7, 132.4, 127.0 (q, *J* = 4.7 Hz), 122.5 (q, *J* = 272.3 Hz), 115.8, 107.1, 42.0, 41.4, 32.3, 24.8 ppm. **^{19}F NMR (376 MHz, CDCl$_3$):** δ -61.8 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{15}F_3N$: 254.1151, found: 254.1158. **FTIR (KBr, neat):** ν 2953, 2229, 1611, 1502, 908, 868 cm^{-1}.



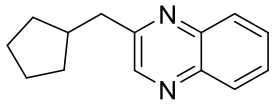
4-(Cyclopentylmethyl)benzaldehyde (3h, 3i, 3j): 63.2 mg, 39.4 mg, 19.0 mg. Yield = 80%, 50%, 24%. Yellow oil. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 9.97 (s, 1H), 7.79 (d, *J* = 8.1 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 2.69 (d, *J* = 7.5 Hz, 2H), 2.10 (hept, *J* = 7.5 Hz, 1H), 1.78-1.59 (m, 4H), 1.58-1.45 (m, 2H), 1.25-1.14 (m, 2H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** δ 192.0, 149.9, 134.3, 129.8, 129.4, 42.3, 41.7, 32.4, 24.8 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{13}H_{17}O$: 189.1274, found: 189.1279. **FTIR (KBr, neat):** ν 2950, 1702, 1605, 1213, 1168, 849 cm^{-1}.



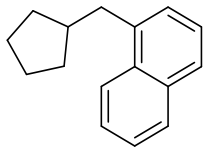
***N*-(4-(Cyclopentylmethyl)phenyl)acetamide (3k):** 43.0 mg. Yield = 47%. White solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.38 (d, *J* = 8.4 Hz, 2H), 7.32 (brs, 1H), 7.11 (d, *J* = 8.4 Hz, 2H), 2.56 (d, *J* = 7.5 Hz, 2H), 2.15 (s, 3H), 2.04 (hept, *J* = 7.5 Hz, 1H), 1.74-1.56 (m, 4H), 1.55-1.46 (m, 2H), 1.23-1.09 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 168.3, 138.5, 135.4, 129.2, 119.9, 42.0, 41.4, 32.4, 24.9, 24.5 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{20}NO$: 218.1539, found: 218.1545. **FTIR (KBr, neat):** ν 3287, 3186, 2948, 1662, 1600, 1556, 1409, 1324, 836, 758 cm^{-1}.



5-(Cyclopentylmethyl)furan-2-carbaldehyde (3l): 35.9 mg. Yield = 48%. Yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 9.51 (s, 1H), 7.16 (d, *J* = 3.6 Hz, 1H), 6.23 (dt, *J* = 3.6, 0.7 Hz, 1H), 2.71 (d, *J* = 7.4 Hz, 2H), 2.24 (hept, *J* = 7.6 Hz, 1H), 1.78 (dtdd, *J* = 12.7, 6.4, 2.8, 1.3 Hz, 2H), 1.68-1.49 (m, 4H), 1.26-1.14 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 176.9, 163.8, 151.7, 140.5, 109.0, 38.6, 34.4, 32.4, 25.0 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{11}H_{15}O_2$: 179.1067, found: 179.1072. **FTIR (KBr, neat):** ν 3185, 3120, 2948, 1661, 1600, 1556, 1513, 1409, 1323, 758 cm^{-1}.

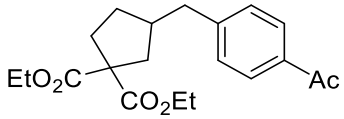


2-(Cyclopentylmethyl)quinoxaline (3m): 37.5 mg. Yield = 42%. Light yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.73 (s, 1H), 8.13-7.99 (m, 2H), 7.81-7.64 (m, 2H), 3.02 (d, *J* = 7.5 Hz, 2H), 2.47-2.30 (m, 1H), 1.83-1.62 (m, 4H), 1.61-1.49 (m, 2H), 1.37-1.23 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 157.3, 146.0, 142.2, 141.2, 129.9, 129.1, 128.9, 42.4, 40.5, 32.5, 24.9 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{17}N_2$: 213.1386, found: 213.1392. **FTIR (KBr, neat):** ν 2954, 1679, 1518, 1023, 967, 800 cm^{-1}.

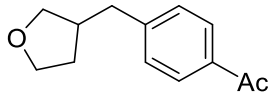


1-(Cyclopentylmethyl)naphthalene (3n): 69.1 mg. Yield = 78%. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.10 (d, *J* = 8.2 Hz, 1H), 7.91-7.84 (m, 1H), 7.73 (d, *J* = 8.1 Hz, 1H), 7.54-7.47 (m, 2H), 7.44-7.38 (m, 1H), 7.34 (d, *J* = 6.9 Hz, 1H), 3.09 (d, *J* = 7.3 Hz, 2H), 2.32 (hept, *J* = 7.4 Hz, 1H), 1.82-1.64 (m, 4H), 1.62-1.48 (m, 2H), 1.33 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 138.4, 133.9, 132.0, 128.7, 126.4, 126.4, 125.5, 125.4, 125.3, 124.0, 41.0, 39.1, 32.8, 24.9 ppm. **HRMS (ESI, m/z):** $[M+H]^+$,

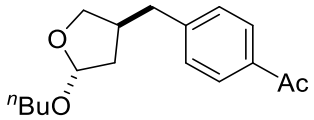
calcd. for $C_{16}H_{19}$: 211.1481, found: 211.1483. **FTIR (KBr, neat):** ν 2950, 1702, 1605, 1306, 1213, 1168, 849, 780 cm^{-1}.



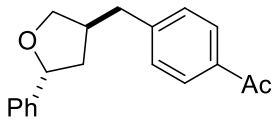
Diethyl 3-(4-acetylbenzyl)cyclopentane-1,1-dicarboxylate (4b): 87.7 mg. Yield = 60%. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.3 Hz, 2H), 4.17 (q, *J* = 7.1 Hz, 2H), 4.14 (q, *J* = 7.1 Hz, 2H), 2.76-2.63 (m, 2H), 2.57 (s, 3H), 2.42-2.20 (m, 3H), 2.13 (ddd, *J* = 13.7, 9.5, 7.5 Hz, 1H), 1.81 (dd, *J* = 13.1, 9.6 Hz, 2H), 1.36 (dtd, *J* = 12.4, 9.6, 8.3 Hz, 1H), 1.23 (t, *J* = 7.1 Hz, 3H), 1.20 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.8, 172.6, 172.5, 147.0, 135.1, 128.9, 128.5, 61.3, 61.3, 59.8, 41.2, 41.2, 40.2, 33.6, 31.9, 26.5, 14.0, 14.0 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{20}H_{27}O_5$: 347.1853, found: 347.1857. **FTIR (KBr, neat):** ν 2981, 1729, 1683, 1606, 1364, 1267, 1180, 861, 817 cm^{-1}.



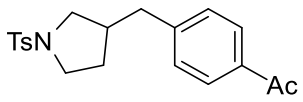
1-(4-((Tetrahydrofuran-3-yl)methyl)phenyl)ethan-1-one (4c): 69.8 mg. Yield = 81%. White solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.86 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.2 Hz, 2H), 3.87 (td, *J* = 8.3, 5.0 Hz, 1H), 3.82-3.68 (m, 2H), 3.42 (dd, *J* = 8.3, 6.7 Hz, 1H), 2.71 (dd, *J* = 7.7, 2.6 Hz, 2H), 2.55 (s, 3H), 2.53-2.44 (m, 1H), 1.96 (dtd, *J* = 12.6, 7.6, 5.0 Hz, 1H), 1.58 (dq, *J* = 12.3, 7.5 Hz, 1H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.7, 146.4, 135.2, 128.8, 128.5, 72.7, 67.7, 40.5, 39.2, 32.0, 26.5 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{13}H_{17}O_2$: 205.1223, found: 205.1225. **FTIR (KBr, neat):** ν 2931, 1682, 1606, 1359, 1269, 905, 863, 819 cm^{-1}.



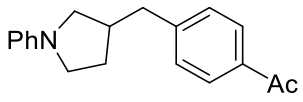
1-(4-((5-Butoxytetrahydrofuran-3-yl)methyl)phenyl)ethan-1-one (4d): 61.5 mg. Yield = 52%, 75:25 dr. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.90-7.88 (m, 2H), 7.28-7.25 (m, 2H), 5.11 (dd, *J* = 5.5, 2.4 Hz, 1H), 3.99-3.95 (m, 1×0.25 H), 3.90 (dd, *J* = 8.4, 7.2 Hz, 1×0.75 H), 3.70 (dt, *J* = 9.6, 6.8 Hz, 1×0.75 H), 3.65-3.63 (m, 1×0.25 H), 3.62-3.58 (m, 1×0.75 H), 3.57-3.53 (m, 1×0.25 H), 3.41-3.36 (m, 1×0.75 H), 3.35-3.32 (m, 1×0.25 H), 2.85 (dd, *J* = 7.8, 2.7 Hz, 2×0.75 H), 2.73 (d, *J* = 2.2 Hz, 2×0.25 H),2.58 (s, 3H), 2.54-2.44 (m, 1H), 2.19-2.12 (m, 1×0.75 H), 2.02-1.97 (m, 1×0.25 H), 1.72-1.47 (m, 3H), 1.44-1.27 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3×0.75 H), 0.86 (t, *J* = 7.3 Hz, 3×0.25 H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** *Major Diastereomer* δ 197.7, 146.6, 135.2, 128.8, 128.6, 104.3, 104.3, 71.5, 67.4, 39.4, 38.4, 31.8, 26.5, 19.4, 13.8 ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** *Minor Diastereomer* δ 197.7, 146.2, 135.3, 128.8, 128.6, 103.9, 71.4, 67.0, 39.8, 39.0, 38.5, 31.7, 26.5, 19.3, 13.8 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{25}O_3$: 277.1798, found: 277.1803. **FTIR (KBr, neat):** ν 2958, 2872, 1684, 1607, 1359, 1268, 861, 819 cm^{-1}.



1-(4-((5-Phenyltetrahydrofuran-3-yl)methyl)phenyl)ethan-1-one (4e): 56.5 mg. Yield = 48%, 84:16 dr. Light yellow solid. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 7.90-7.87 (m, 2H), 7.35-7.21 (m, 7H), 5.08 (dd, *J* = 7.5, 6.3 Hz, 1×0.84 H), 4.90 (dd, *J* = 9.6, 6.1 Hz, 1×0.16 H), 4.16 (dd, *J* = 8.5, 6.7 Hz, 1×0.84 H), 4.05 (dd, *J* = 8.4, 7.0 Hz, 1×0.16 H), 3.77 (dd, *J* = 8.5, 7.0 Hz, 1×0.16 H), 3.64 (dd, *J* = 8.5, 6.8 Hz, 1×0.84 H), 2.82 (d, *J* = 7.8 Hz, 2H), 2.71-2.61 (m, 1H), 2.58 (s, 3H), 2.47-2.37 (m, 1×0.16 H), 2.11 (ddd, *J* = 12.5, 7.6, 6.4 Hz, 1×0.84 H), 1.98 (ddd, *J* = 12.5, 7.8, 6.2 Hz, 1×0.84 H), 1.56 (dt, *J* = 12.4, 9.1 Hz, 1×0.16 H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** *Major Diastereomer* δ 197.7, 146.2, 143.4, 135.3, 128.8, 128.6, 128.3, 127.1, 125.4, 79.9, 73.5, 40.4, 40.3, 39.0, 26.5 ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** *Minor Diastereomer* δ 197.7, 146.3, 142.8, 135.3, 128.8, 128.6, 128.3, 127.3, 125.5, 81.2, 73.4, 41.8, 41.6, 39.4, 26.5 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{19}H_{21}O_2$: 281.1536, found: 281.1541. **FTIR (KBr, neat):** ν 3029, 2970, 2851, 1683, 1602, 1492, 1362, 1268, 1046, 868, 817, 762, 705 cm^{-1}.

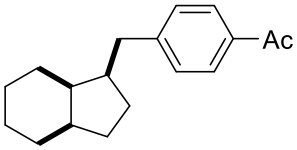


1-(4-((1-Tosylpyrrolidin-3-yl)methyl)phenyl)ethan-1-one (4f): 49.6 mg. Yield = 33%. Light yellow solid. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 7.84 (d, *J* = 8.2 Hz, 2H), 7.67 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.2 Hz, 2H), 3.38 (ddd, *J* = 9.8, 8.2, 4.2 Hz, 1H), 3.30 (dd, *J* = 9.8, 7.1 Hz, 1H), 3.17 (dt, *J* = 9.8, 7.7 Hz, 1H), 2.89 (dd, *J* = 9.8, 7.4 Hz, 1H), 2.60 (d, *J* = 7.6 Hz, 2H), 2.56 (s, 3H), 2.42 (s, 3H), 2.32 (hept, *J* = 7.5 Hz, 1H), 1.86 (dtd, *J* = 11.4, 7.0, 4.1 Hz, 1H), 1.48 (dq, *J* = 12.5, 8.2 Hz, 1H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** δ 197.6, 145.3, 143.4, 135.5, 133.8, 129.6, 128.8, 128.6, 127.5, 52.6, 47.2, 40.0, 39.0, 31.0, 26.5, 21.5 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{20}H_{24}NO_3S$: 358.1471, found: 358.1476. **FTIR (KBr, neat):** ν 2920, 1676, 1604, 1329, 1268, 1159, 812, 707, 664 cm^{-1}.

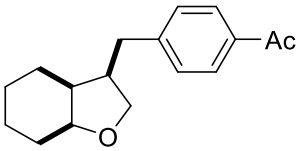


1-(4-((1-Phenylpyrrolidin-3-yl)methyl)phenyl)ethan-1-one (4g): 69.0 mg. Yield = 60%, Yellow solid. **^{1}H NMR (400 MHz, CDCl$_3$):** δ 7.97-7.90 (m, 2H), 7.35-7.29 (m, 2H), 7.26 -7.20 (m, 2H), 6.67 (tt, *J* = 7.3, 1.1 Hz, 1H), 6.57-6.51 (m, 2H), 3.46-3.35 (m, 2H), 3.30 (dt, *J* = 9.1, 7.6 Hz, 1H), 3.02 (dd, *J* = 9.2, 7.2 Hz, 1H), 2.83 (d, *J* = 7.6 Hz, 2H), 2.68-2.57 (m, 1H), 2.61 (s, 3H), 2.11 (dtd, *J* = 12.1, 6.9, 3.9 Hz, 1H), 1.76 (dq, *J* = 12.2, 8.2 Hz, 1H) ppm. **^{13}C NMR (100 MHz, CDCl$_3$):** δ 197.7, 147.7, 146.4, 135.3, 129.1, 128.9, 128.6, 115.5, 111.4, 52.8, 47.1, 40.1, 39.8, 31.3, 26.5 ppm. **HRMS (ESI,**

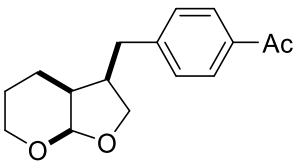
m/z): $[M+H]^+$, calcd. for $C_{19}H_{22}NO$: 280.1696, found: 280.1702. **FTIR (KBr, neat):** ν 2920, 1676, 1604, 1329, 1159, 812, 707, 692 cm^{-1}.



1-(4-(((1R,3aR,7aR)-Octahydro-1*H*-inden-1-yl)methyl)phenyl)ethan-1-one (4h): 58.1 mg. Yield = 54%, 75:25 dr. White solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.87 (dd, *J* = 8.3, 2.8 Hz, 2H), 7.28-7.25 (m, 2H), 2.81 (dd, *J* = 13.5, 5.8 Hz, 1×0.25 H), 2.73 (dd, *J* = 13.6, 7.5 Hz, 1×0.75 H), 2.61-2.55 (m, 1×0.75 H), 2.58 (s, 3H), 2.48-2.40 (m, 1×0.25 H), 2.26-2.16 (m, 1×0.75 H), 2.11-1.99 (m, 1H), 1.97-1.93 (m, 1×0.25 H), 1.71-1.17 (m, 11H), 1.14-0.80 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** *Major Diastereomer* δ 197.9, 148.6, 134.8, 128.7, 128.4, 45.9, 42.0, 39.1, 37.2, 28.5, 27.1, 26.5, 25.4, 25.3, 22.2, 20.8 ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** *Minor Diastereomer* δ 197.9, 148.3, 134.8, 129.0, 128.3, 44.8, 42.2, 42.0, 38.8, 29.5, 29.3, 28.3, 26.7, 26.5, 24.2, 22.7 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{18}H_{25}O$: 257.1900, found: 257.1902. **FTIR (KBr, neat):** ν 2920, 1675, 1604, 1413, 1360, 1267, 855, 809 cm^{-1}.

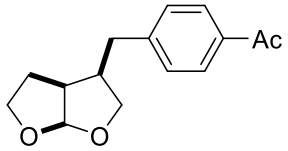


1-(4-(((3R,3aS,7aS)-Octahydrobenzofuran-3-yl)methyl)phenyl)ethan-1-one (4i): 54.3 mg. Yield = 50%, 70:30 dr. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.90-7.88 (m, 2H), 7.29-7.25 (m, 2H), 4.08 (dd, *J* = 8.8, 7.6 Hz, 1×0.70 H), 4.02 (q, *J* = 4.5 Hz, 1×0.70 H), 3.97 (q, *J* = 3.0 Hz, 1×0.30 H), 3.89 (t, *J* = 8.0 Hz, 1×0.30 H), 3.65 (dd, *J* = 9.4, 8.0 Hz, 1×0.30 H), 3.51 (dd, *J* = 8.8, 4.7 Hz, 1×0.70 H), 2.86-2.74 (m, 1H), 2.74-2.63 (m, 1H), 2.59 (s, 3H), 2.35-2.27 (m, 1×0.70 H), 2.02-1.94 (m, 1×0.30 H), 1.90-1.70 (m, 2H),1.65-1.06 (m, 7H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** *Major Diastereomer* δ 197.8, 146.4, 135.2, 128.9, 128.6, 76.1, 71.8, 70.64, 45.5, 42.9, 39.7, 33.6, 28.2, 27.4, 26.5, 23.6, 21.0 ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** *Minor Diastereomer* δ 197.7, 146.7, 135.2, 128.9, 128.6, 78.2, 70.6, 45.1, 42.9, 39.9, 33.6, 28.5, 24.4, 22.1, 20.3 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{23}O_2$: 259.1693, found: 259.1698. **FTIR (KBr, neat):** ν 2927, 2855, 1679, 1605, 1358, 1267, 1019, 959, 857, 805 cm^{-1}.



1-(4-(((3R,3aS,7aR)-Hexahydro-4*H*-furo[2,3-*b*]pyran-3-yl)methyl)phenyl)ethan-1-one (4j): 57.3 mg. Yield = 52%, 88:12 dr. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.89 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 5.28 (d, *J* = 3.6 Hz, 1×0.88 H), 5.04

(d, *J* = 3.5 Hz, 1×0.12 H), 4.17 (t, *J* = 8.0 Hz, 1×0.12 H), 3.88 (t, *J* = 7.7 Hz, 1H), 3.82-3.75 (m, 2×0.88 H), 3.70-3.61 (m, 1H), 3.42 (td, *J* = 11.3, 2.3 Hz, 1×0.12 H), 2.96-2.87 (m, 1×0.12 H), 2.80 (dd, *J* = 10.6, 6.0 Hz, 1×0.88 H), 2.76-2.62 (m, 2H), 2.59 (s, 3H), 1.99-1.85 (m, 1H), 1.82-1.70 (m, 1H), 1.69-1.50 (m, 3H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** *Major Diastereomer* δ 197.6, 145.8, 135.2, 128.6, 128.5, 101.7, 69.5, 60.8, 42.0, 36.3, 33.3, 26.5, 22.9, 19.4 ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** *Minor Diastereomer* δ 197.6, 145.7, 135.2, 128.6, 128.5, 101.9, 73.3, 64.3, 43.8, 39.0, 38.6, 26.5, 22.3, 20.5 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{16}H_{21}O_3$: 261.1485, found: 261.1490. **FTIR (KBr, neat):** ν 2920, 2881, 1675, 1604, 1360, 1267, 959, 855, 809 cm^{-1}.



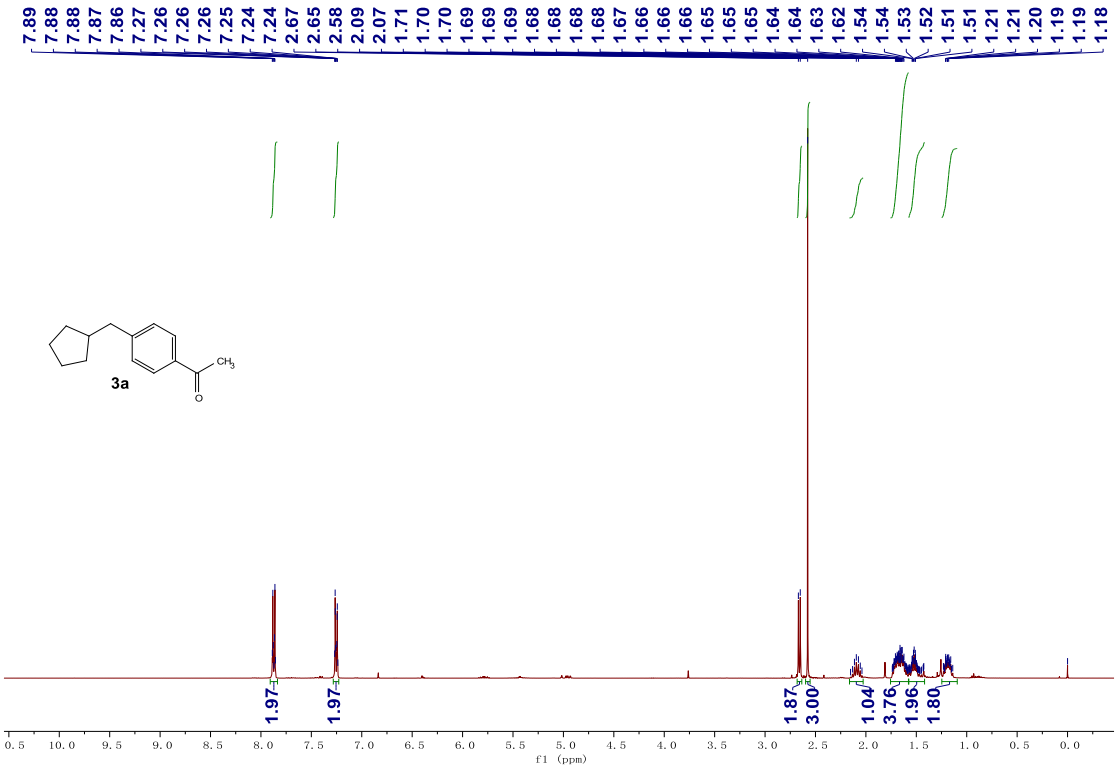
1-(4-(((3R,3aS,6aR)-Hexahydrofuro[2,3-*b*]furan-3-yl)methyl)phenyl)ethan-1-one (4k): 57.9 mg. Yield = 56%, 99:1 dr. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.90 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 5.72 (d, *J* = 4.9 Hz, 1H), 3.97 (dt, *J* = 9.0, 7.1 Hz, 1H), 3.88 (ddd, *J* = 8.3, 7.0, 5.8 Hz, 2H), 3.57 (dd, *J* = 10.9, 8.5 Hz, 1H), 2.88-2.62 (m, 4H), 2.59 (s, 3H), 2.05-1.95 (m, 1H), 1.87 (ddt, *J* = 13.1, 9.8, 7.4 Hz, 1H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.6, 145.6, 135.5, 128.7, 128.5, 109.7, 71.9, 69.0, 45.3, 43.4, 33.8, 26.5, 25.1 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{15}H_{19}O_3$: 247.1329, found: 247.1331. **FTIR (KBr, neat):** ν 3064, 2945, 2880, 1676, 1605, 1418, 1359, 1271, 1007, 859, 833 cm^{-1}.

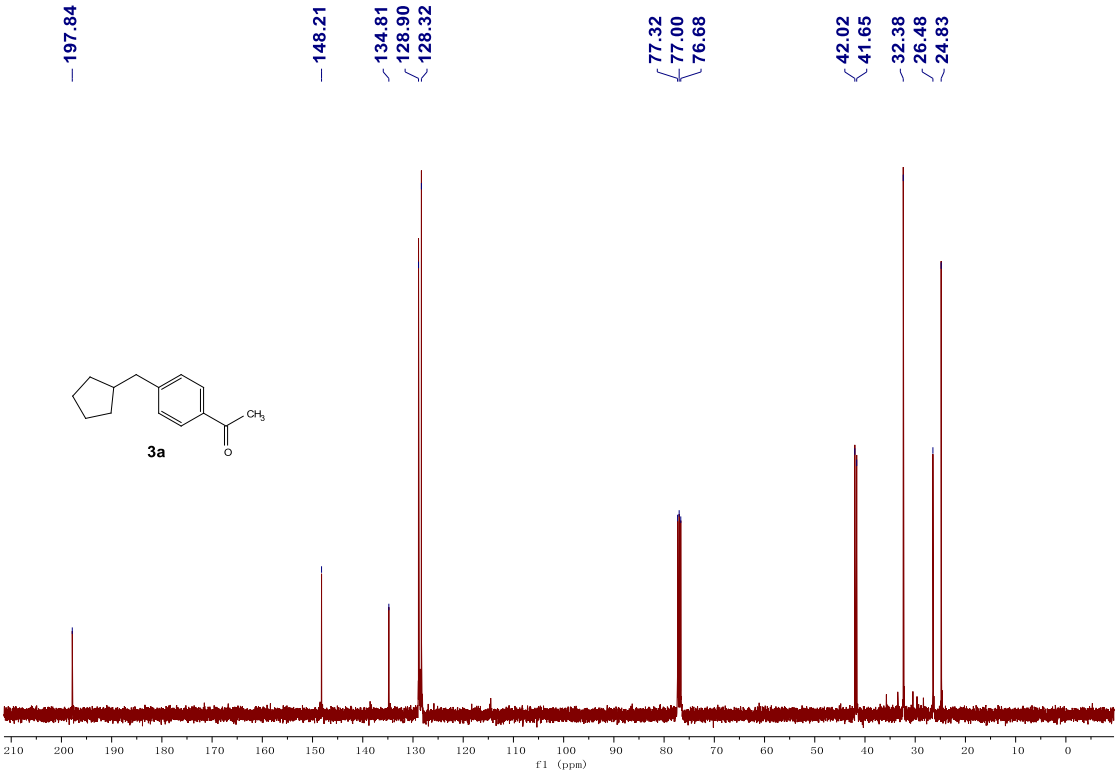
References

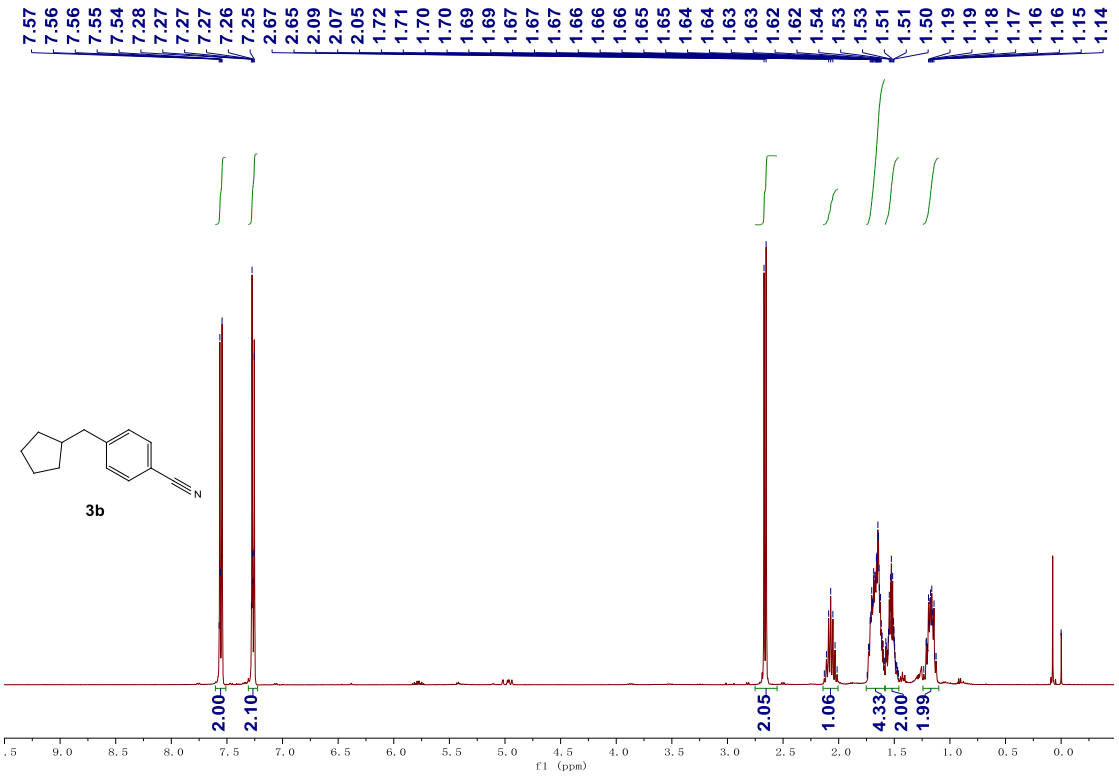
1. Ashby, M. T.; Enemark, J. H.; Lichtenberger, D. L. *Inorg. Chem.* **1988**, *27*, 191-197.
2. Millan, A.; Alvarez de Cienfuegos, L.; Miguel, D.; Campana, A. G.; Cuerva, J. M. *Org. Lett.* **2012**, *14*, 5984-5987.
3. Ramler, J.; Krummenacher, I.; Lichtenberg, C. *Angew. Chem. Int. Ed.* **2019**, *58*, 12924-12929.
4. McMahon, C. M.; Renn, M. S.; Alexanian, E. J. *Org. Lett.* **2016**, *18*, 4148-4150.
5. Kita, Y.; Nambu, H.; Ramesh, N. G.; Anilkumar, G.; Matsug, M. *Org. Lett.* **2001**, *3*, 1157-1160.
6. Hwang, J. Y.; Baek, J. H.; Shin, T. I.; Shin, J. H.; Oh, J. W.; Kim, K. P.; You, Y.; Kang, E. J. *Org. Lett.* **2016**, *18*, 4900-4903.
7. Cui, J.; Wang, H.; Song, J.; Chi, X.; Meng, L.; Liu, Q.; Zhang, D.; Dong, Y.; Liu, H. *Org. Biomol. Chem.* **2017**, *15*, 8508-8512.
8. Hon,Y.-S.; Liua, Y.-W.; Hsieh, C.-H. *Tetrahedron* **2004**, *60*, 4837-4860.
9. Seel, S.; Dagousset, G.; Thaler, T.; Frischmuth, A.; Karaghiosoff, K.; Zipse, H.; Knochel, P. *Chem.-Eur. J.* **2013**, *19*, 4614-4622.
10. Stadtmüller, H.; Tucker, C. E.; Vaupel, A.; Knochel, P. *Tetrahedron Lett.* **1993**, *34*, 7911-7914.

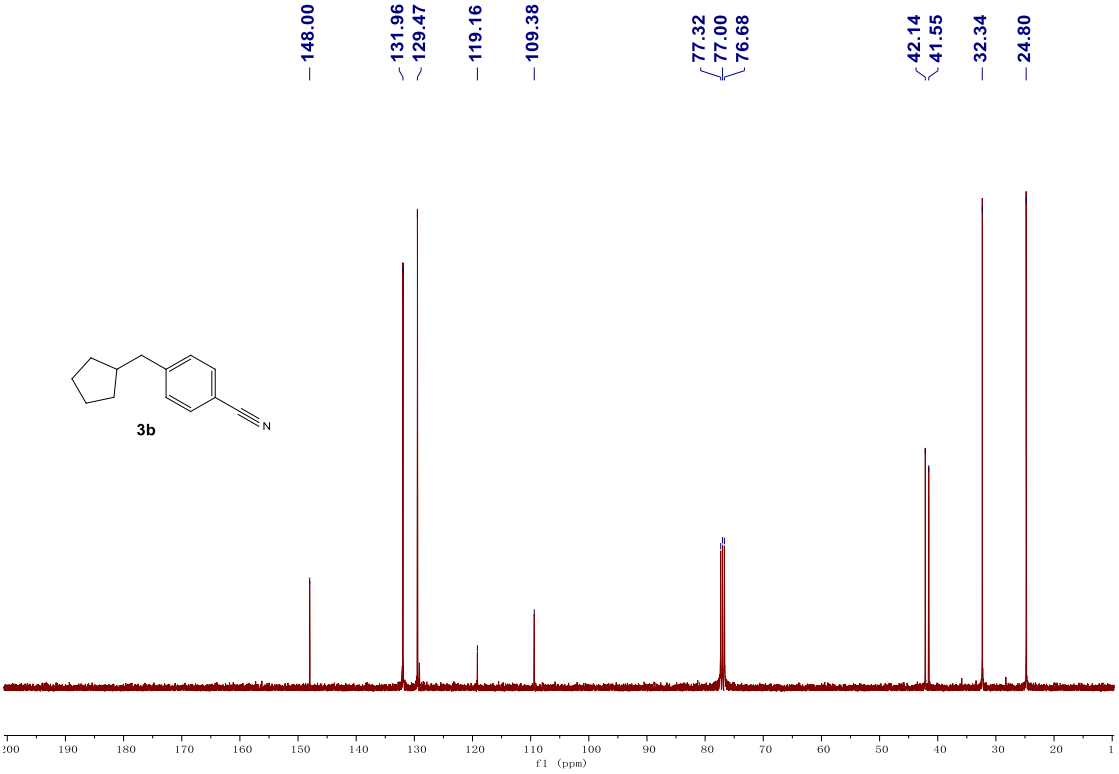
11. Barluenga, J.; Vazquez-Villa, H.; Ballesteros, A.; Gonzalez, J. M. *Org. Lett.* **2002**, *4*, 2817-2819.
12. Deng, W.; Ye, C.; Li, Y.; Li, D.; Bao, H. *Org. Lett.* **2019**, *21*, 261-265.
13. Ollivier, C.; Renaud, P. *J. Am. Chem. Soc.* **2001**, *123*, 4717-4727.
14. Guisán-Ceinos, M.; Soler-Yanes, R.; Collado-Sanz, D.; Phapale, V. B.; Buñuel, E.; Cárdenas, D. J. *Chem.-Eur. J.* **2013**, *19*, 8405-8410.
15. Vaupel, A.; Knochel, P. *J. Org. Chem.* **1996**, *61*, 5743-5753.

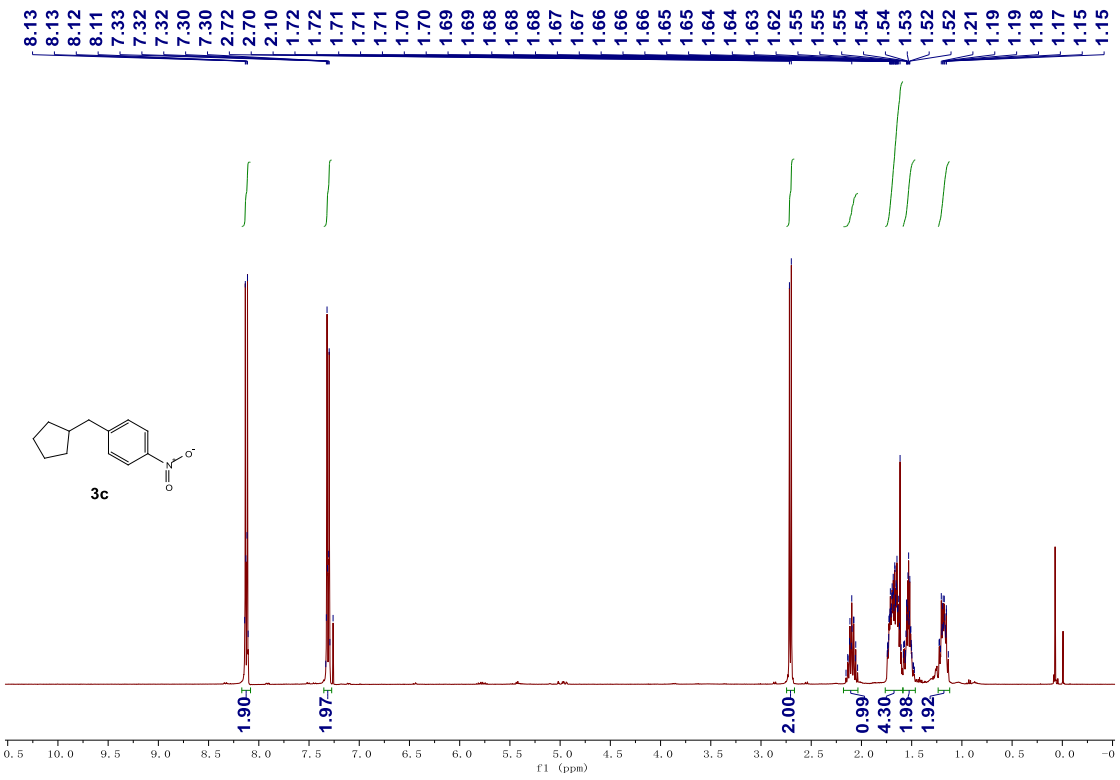
^{1}H, ^{19}F, and ^{13}C NMR spectra of products

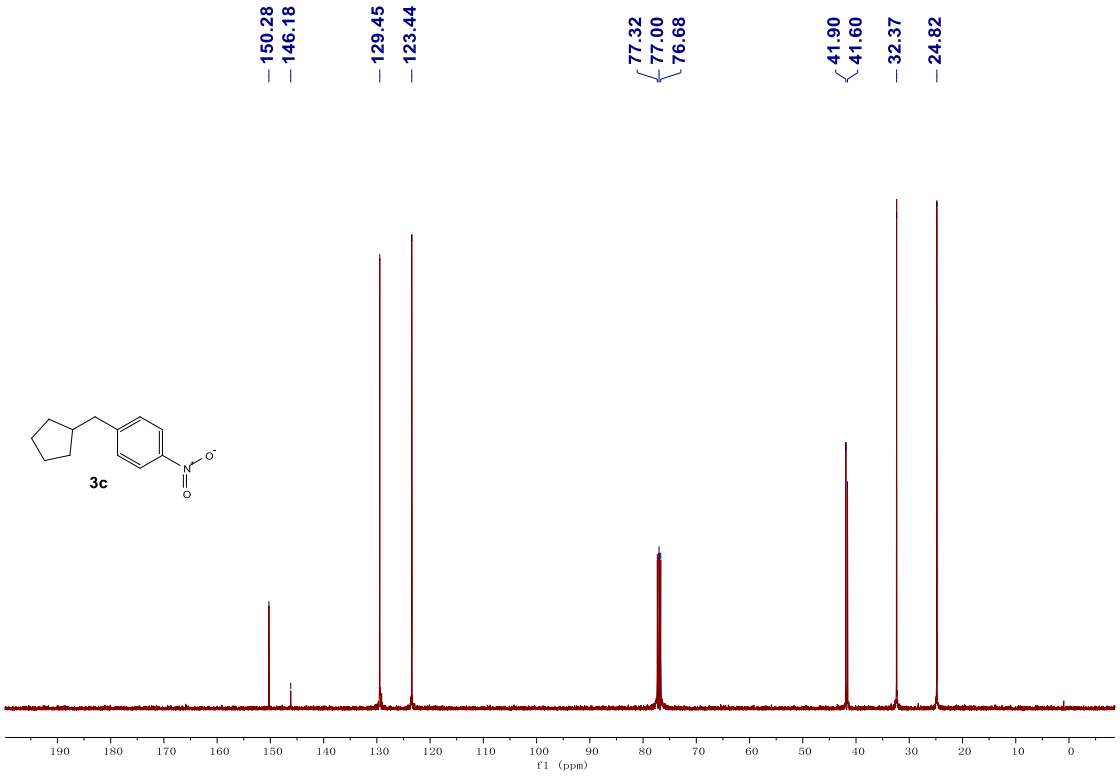


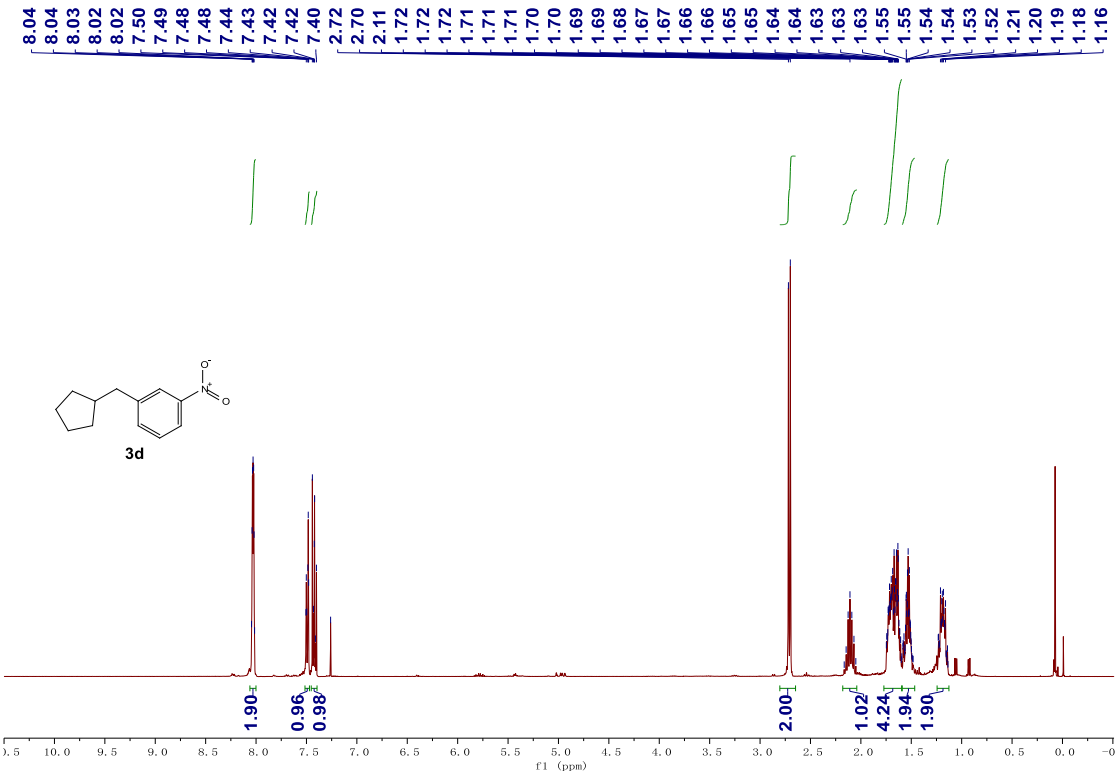


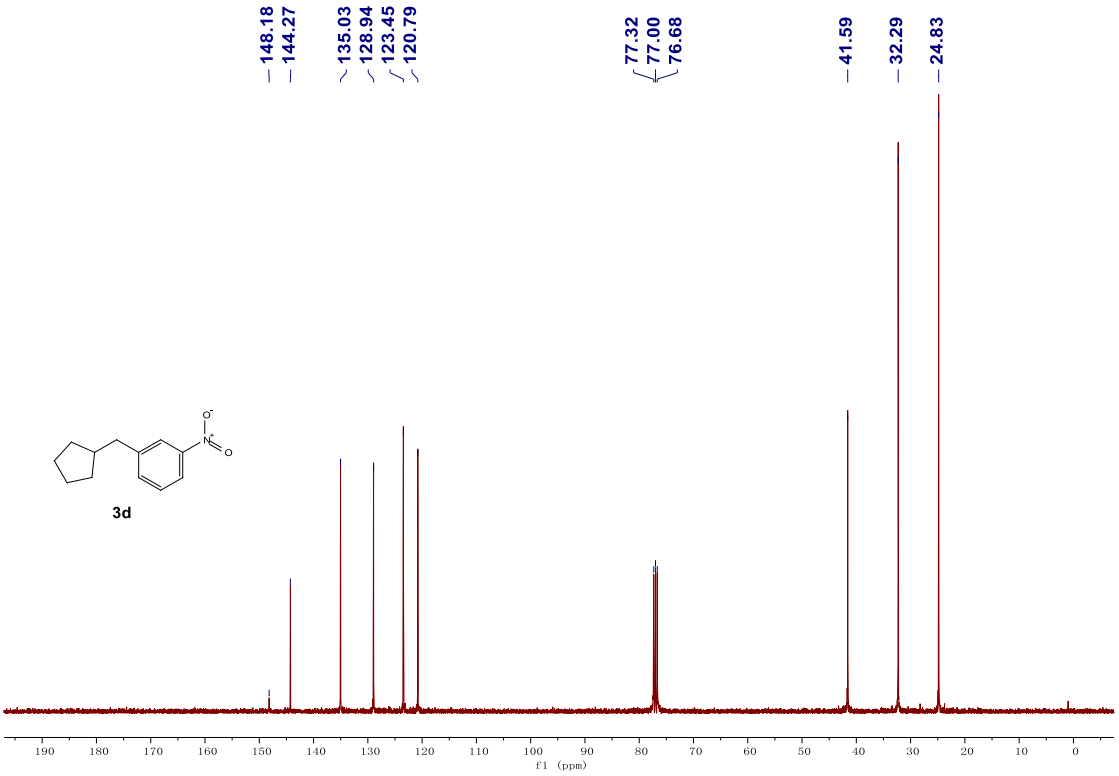

7.57 7.56 7.56 7.55 7.54 7.28 7.27 7.27 7.27 7.26 7.25 2.67 2.65 2.09 2.07 2.05 1.72 1.71 1.70 1.70 1.69 1.69 1.67 1.67 1.67 1.66 1.66 1.66 1.65 1.65 1.64 1.64 1.63 1.63 1.62 1.62 1.54 1.53 1.53 1.51 1.51 1.50 1.19 1.19 1.18 1.17 1.16 1.16 1.15 1.14
3b
2.00 2.10 2.05 1.06 4.33 2.00 1.99
f1 (ppm)

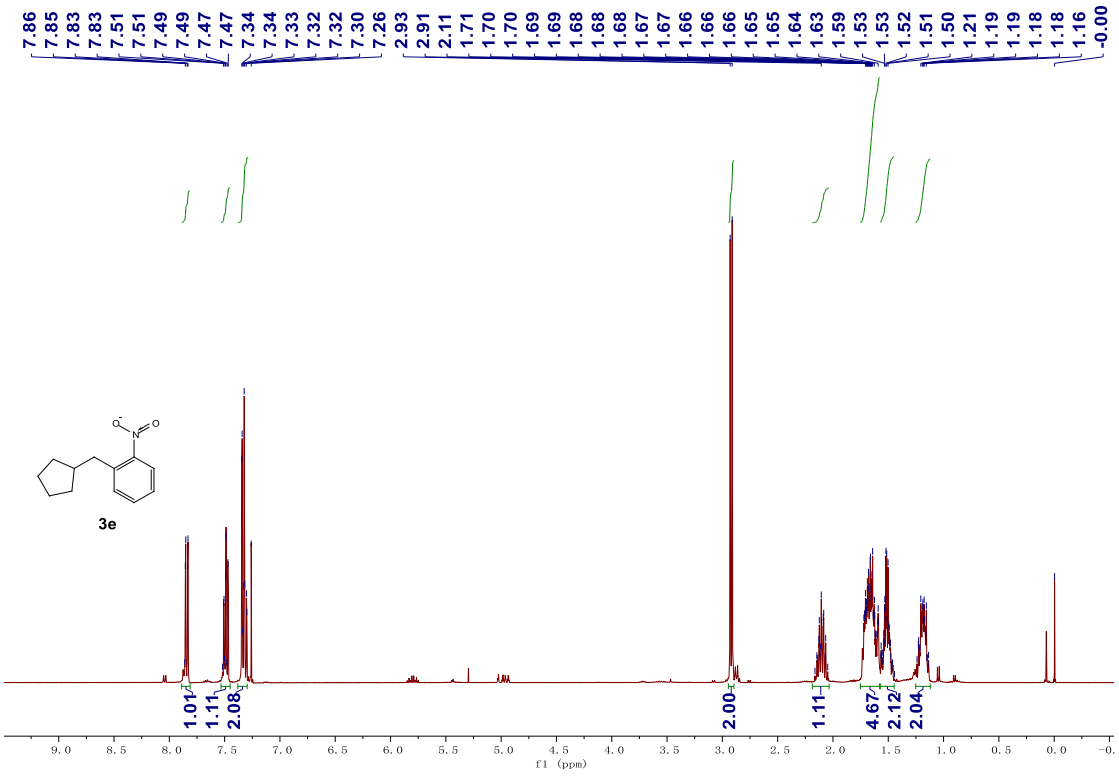

148.00 131.96 129.47 119.16 109.38 77.32 77.00 76.68 42.14 41.55 32.34 24.80
3b
f1 (ppm)

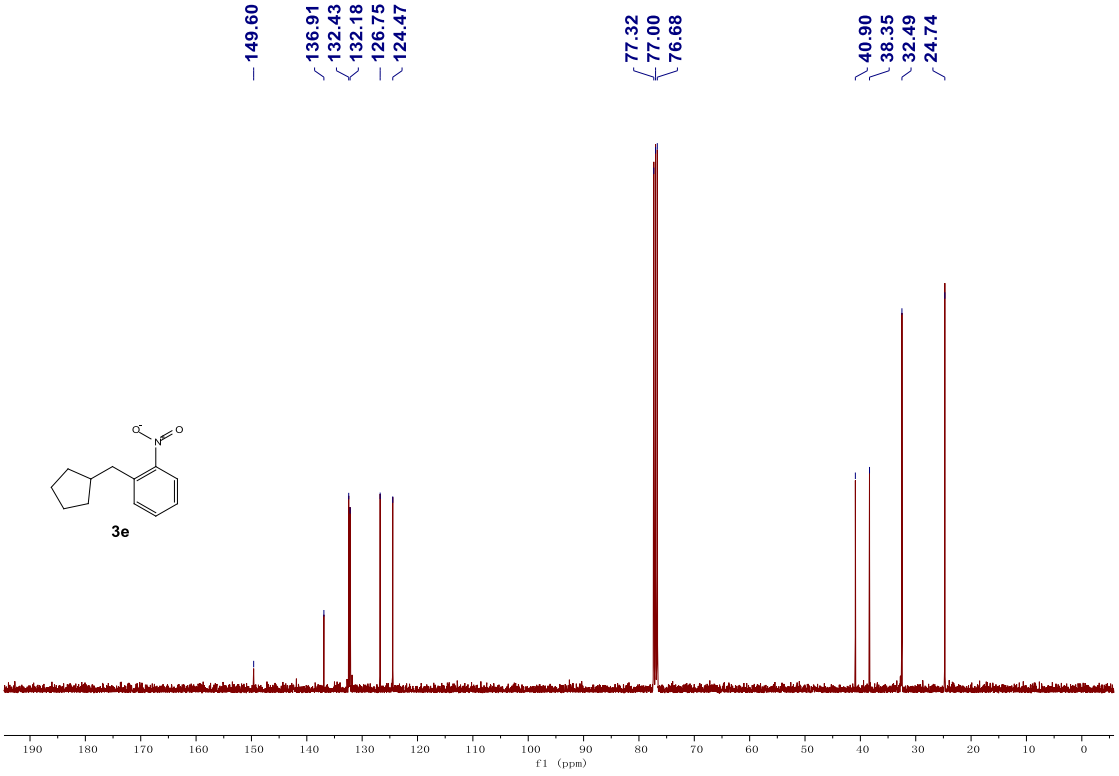

8.13 8.13 8.12 8.11 7.33 7.32 7.32 7.30 7.30 2.72 2.70 2.10 1.72 1.72 1.71 1.71 1.71 1.70 1.70 1.69 1.69 1.68 1.68 1.68 1.67 1.67 1.66 1.66 1.66 1.65 1.65 1.64 1.64 1.63 1.62 1.55 1.55 1.55 1.54 1.54 1.53 1.52 1.52 1.21 1.19 1.19 1.18 1.17 1.15 1.15
3c
1.90 1.97 2.00 0.99 4.30 1.98 1.92
f1 (ppm)

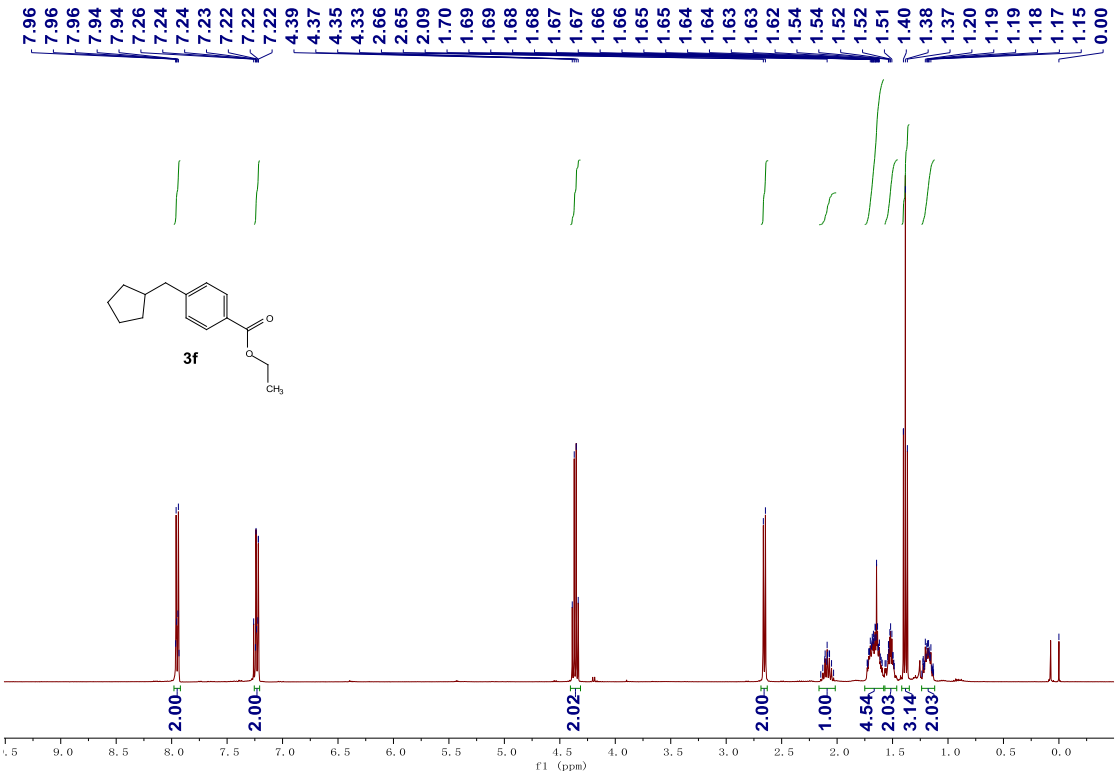

150.28 146.18 129.45 123.44 77.32 77.00 76.68 41.90 41.60 32.37 24.82
3c
f1 (ppm)

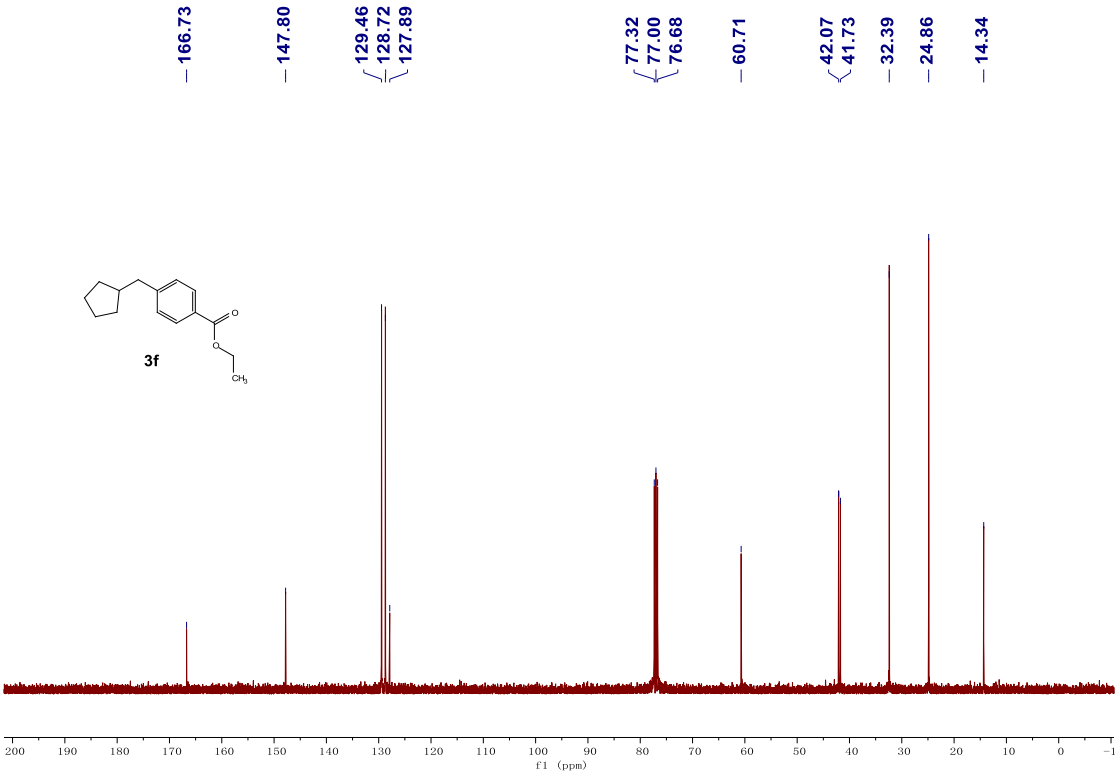

8.04 8.04 8.03 8.02 8.02 7.50 7.49 7.48 7.48 7.44 7.43 7.42 7.42 7.40 2.72 2.70 2.11 1.72 1.72 1.72 1.71 1.71 1.71 1.70 1.70 1.69 1.69 1.68 1.67 1.67 1.66 1.66 1.65 1.65 1.64 1.64 1.63 1.63 1.63 1.55 1.55 1.54 1.54 1.53 1.52 1.21 1.20 1.19 1.18 1.16
3d
1.90 0.96 0.98 2.00 1.02 4.24 1.94 1.90
f1 (ppm)

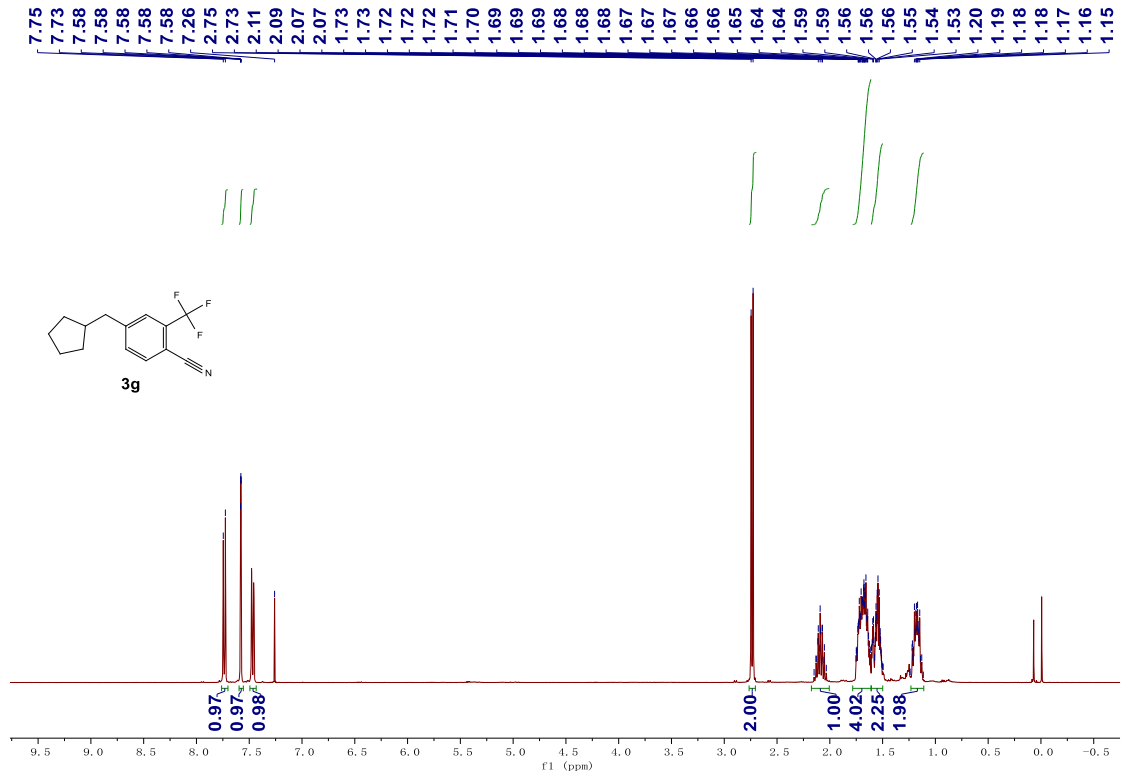

148.18 144.27 135.03 128.94 123.45 120.79 77.32 77.00 76.68 41.59 32.29 24.83
3d
f1 (ppm)

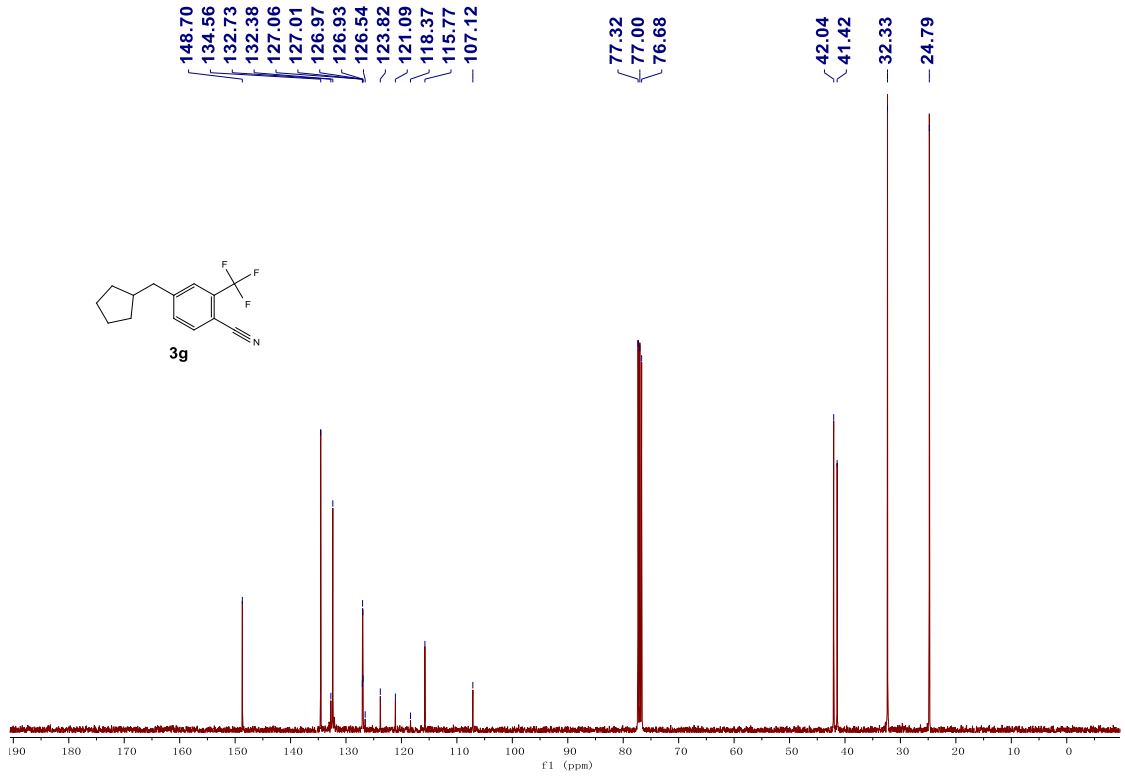

7.86
7.85
7.83
7.83
7.51
7.51
7.49
7.49
7.47
7.47
7.34
7.34
7.33
7.32
7.32
7.30
7.26
2.93
2.91
2.11
1.71
1.70
1.70
1.69
1.69
1.68
1.68
1.68
1.67
1.67
1.66
1.66
1.66
1.65
1.65
1.64
1.63
1.59
1.53
1.53
1.52
1.51
1.50
1.21
1.19
1.19
1.18
1.18
1.16
-0.00
3e
1.01
1.11
2.08
2.00
1.11
4.67
2.12
2.04
f1 (ppm)

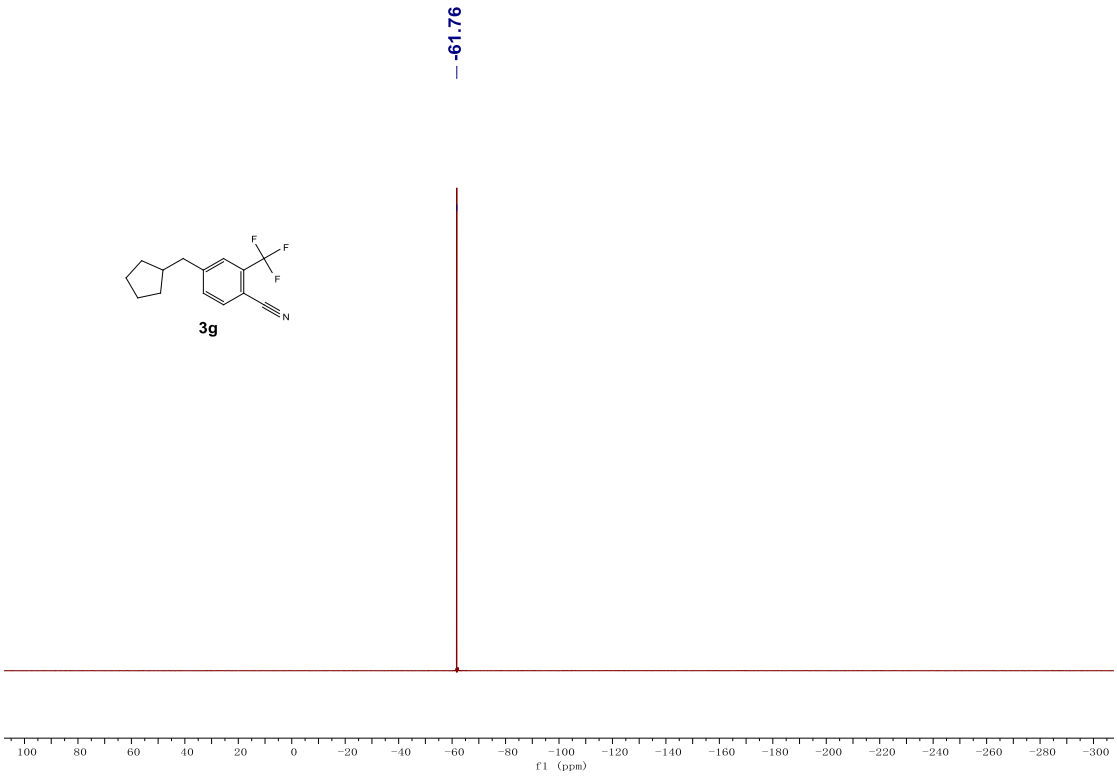

149.60
136.91
132.43
132.18
126.75
124.47
77.32
77.00
76.68
40.90
38.35
32.49
24.74
3e
f1 (ppm)

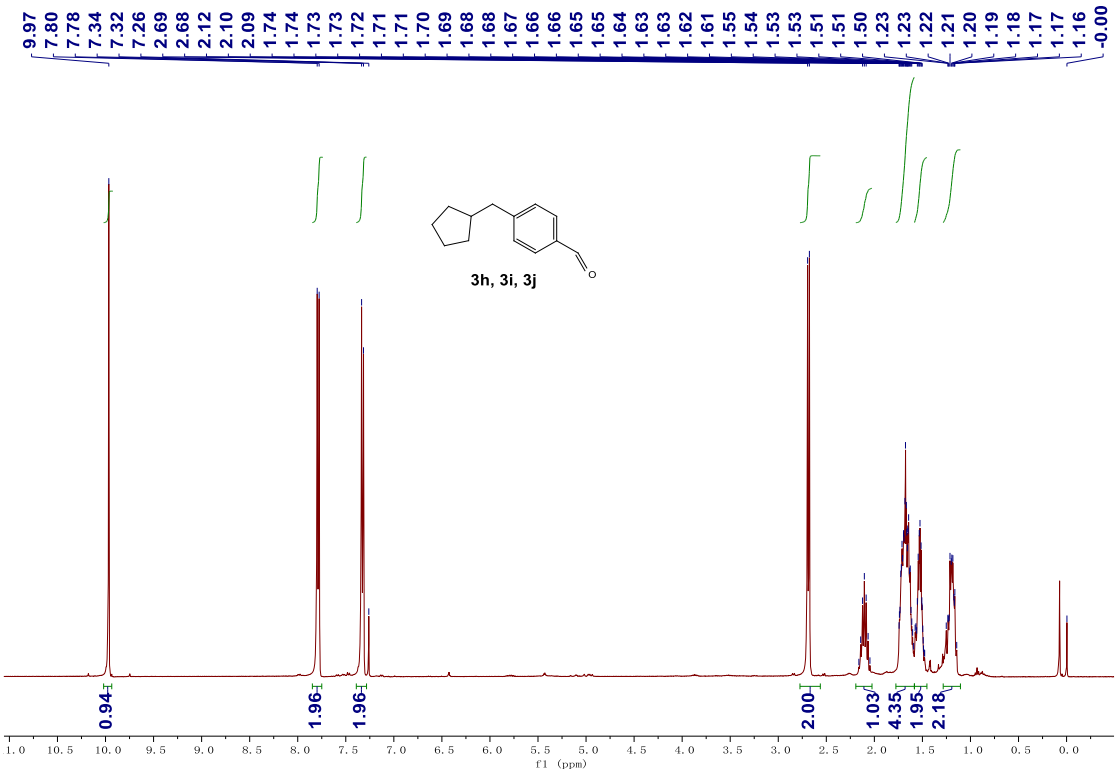

7.96 7.96 7.96 7.94 7.94 7.26 7.24 7.24 7.23 7.22 7.22 7.22
4.39 4.37 4.35 4.33 2.66 2.65 2.09 1.70 1.69 1.69 1.68 1.68 1.67 1.67 1.66 1.66 1.65 1.65 1.64 1.64 1.63 1.63 1.62 1.54 1.54 1.52 1.52 1.51 1.40 1.38 1.37 1.20 1.19 1.19 1.18 1.17 1.15 0.00
3f
2.00 2.00 2.02 2.00 1.00 4.54 2.03 3.14 2.03
f1 (ppm)

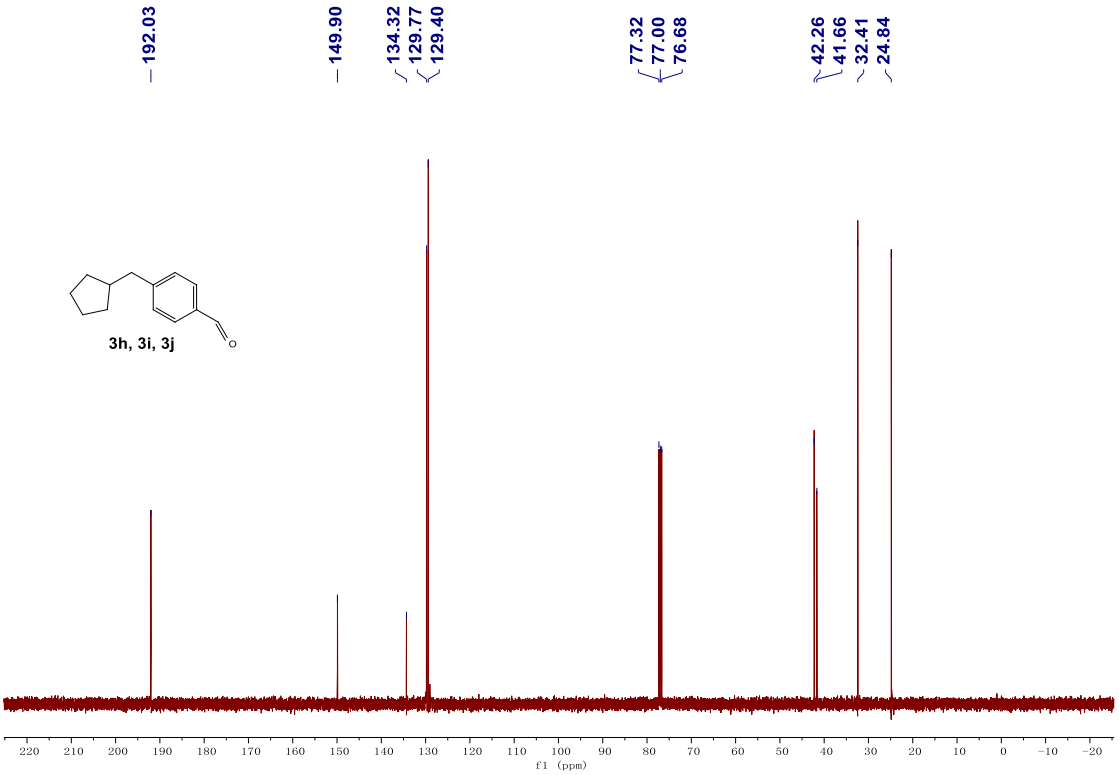

166.73 147.80 129.46 128.72 127.89 77.32 77.00 76.68 60.71 42.07 41.73 32.39 24.86 14.34
3f
f1 (ppm)

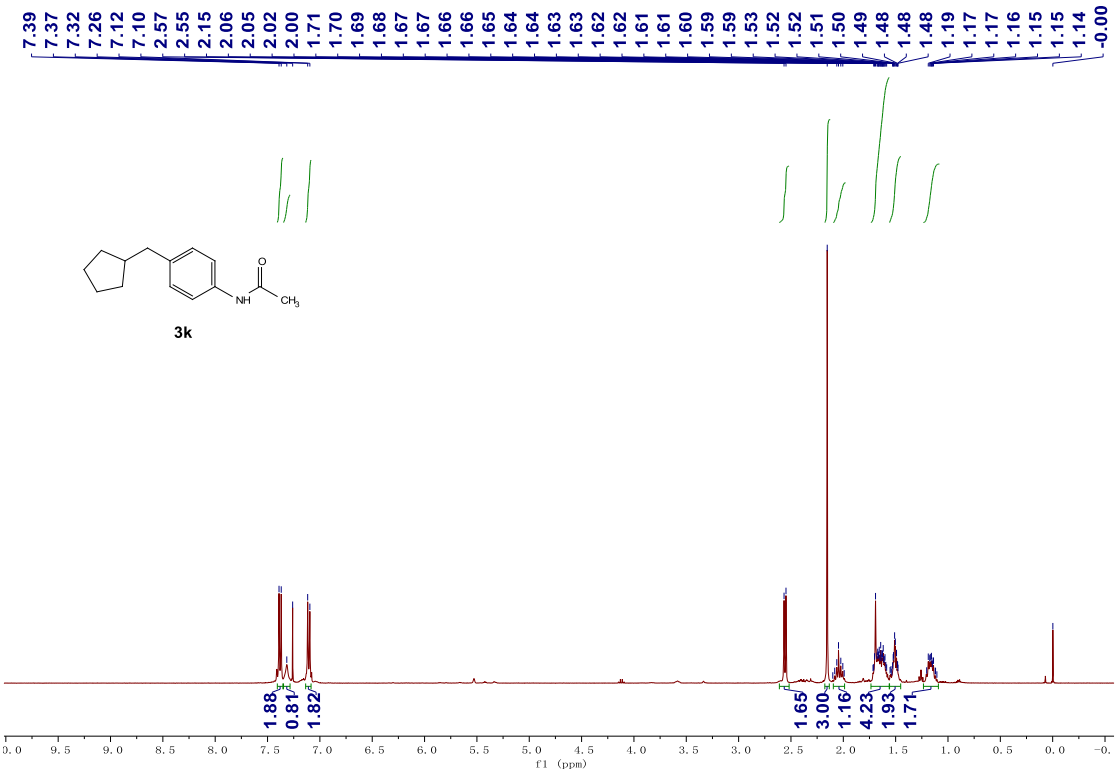

7.75 7.73 7.58 7.58 7.58 7.58 7.58 7.26 2.75 2.73 2.11 2.09 2.07 2.07 1.73 1.73 1.72 1.72 1.72 1.71 1.70 1.69 1.69 1.69 1.68 1.68 1.68 1.67 1.67 1.67 1.66 1.66 1.65 1.64 1.64 1.59 1.59 1.56 1.56 1.56 1.55 1.54 1.53 1.20 1.19 1.18 1.18 1.17 1.16 1.15
3g
0.97 0.97 0.98 2.00 1.00 4.02 2.25 1.98
f1 (ppm)

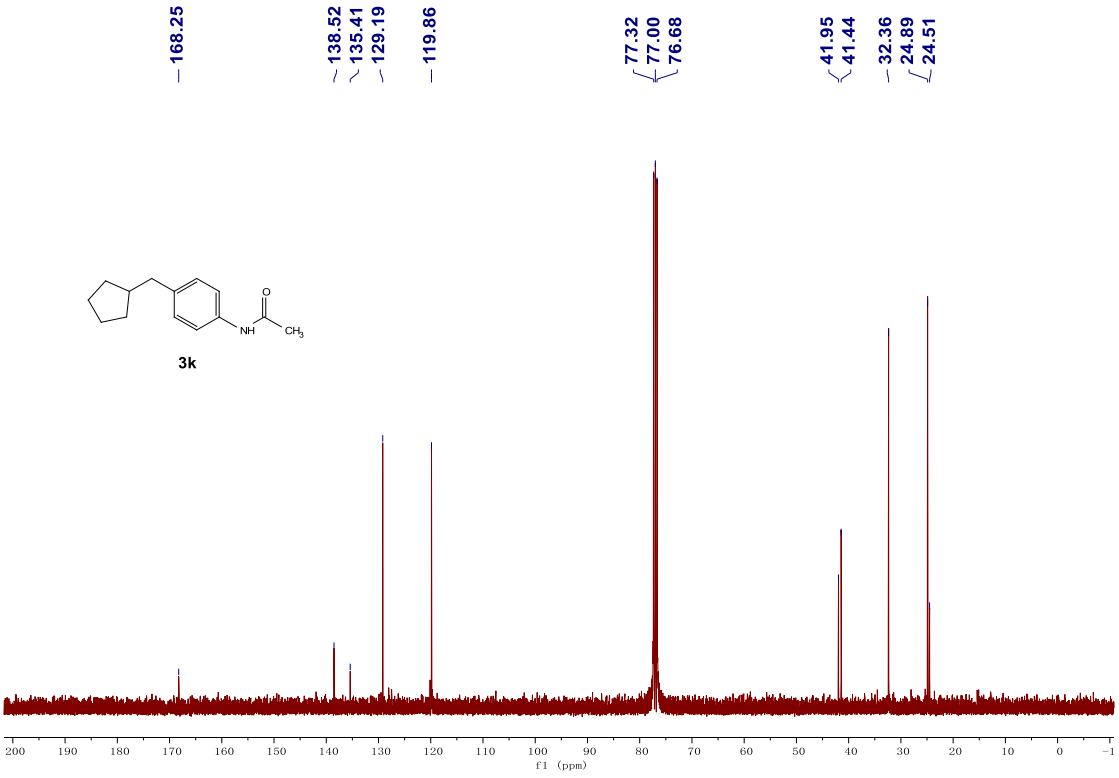

148.70 134.56 132.73 132.38 127.06 127.01 126.97 126.93 126.54 123.82 121.09 118.37 115.77 107.12
77.32 77.00 76.68
42.04 41.42 32.33 24.79
3g
f1 (ppm)

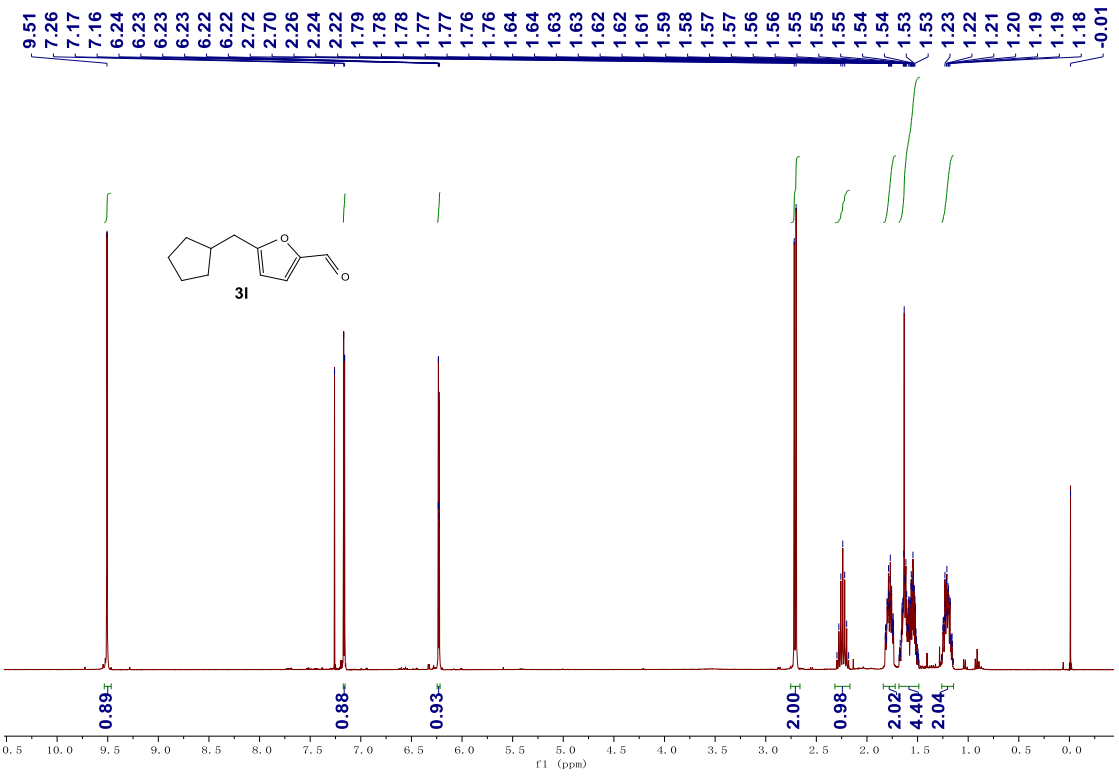

-61.76
F
F
F
N
3g
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
-240
-260
-280
-300
f1 (ppm)

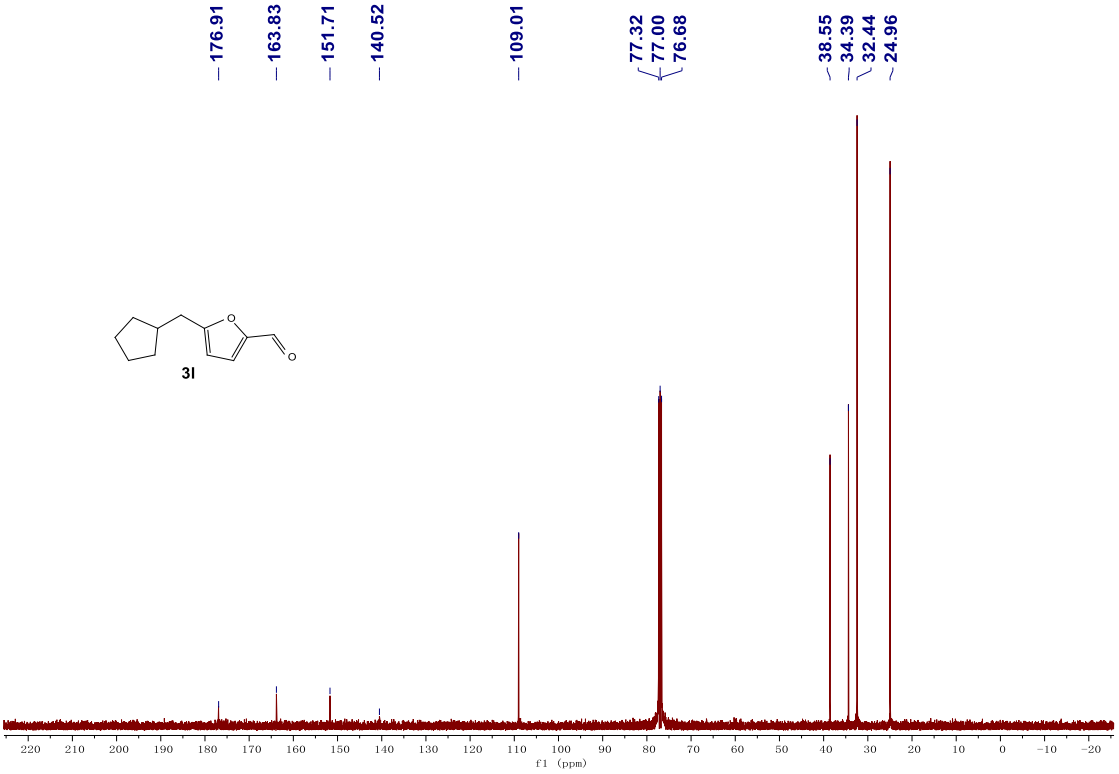

9.97
7.80
7.78
7.34
7.32
7.26
2.69
2.68
2.12
2.10
2.09
1.74
1.74
1.73
1.73
1.72
1.71
1.71
1.70
1.69
1.68
1.68
1.67
1.66
1.66
1.65
1.65
1.64
1.63
1.63
1.62
1.61
1.55
1.54
1.53
1.53
1.51
1.51
1.50
1.23
1.23
1.22
1.21
1.20
1.19
1.18
1.17
1.17
1.16
-0.00
3h, 3i, 3j
0.94
1.96
1.96
2.00
1.03
4.35
1.95
2.18
f1 (ppm)

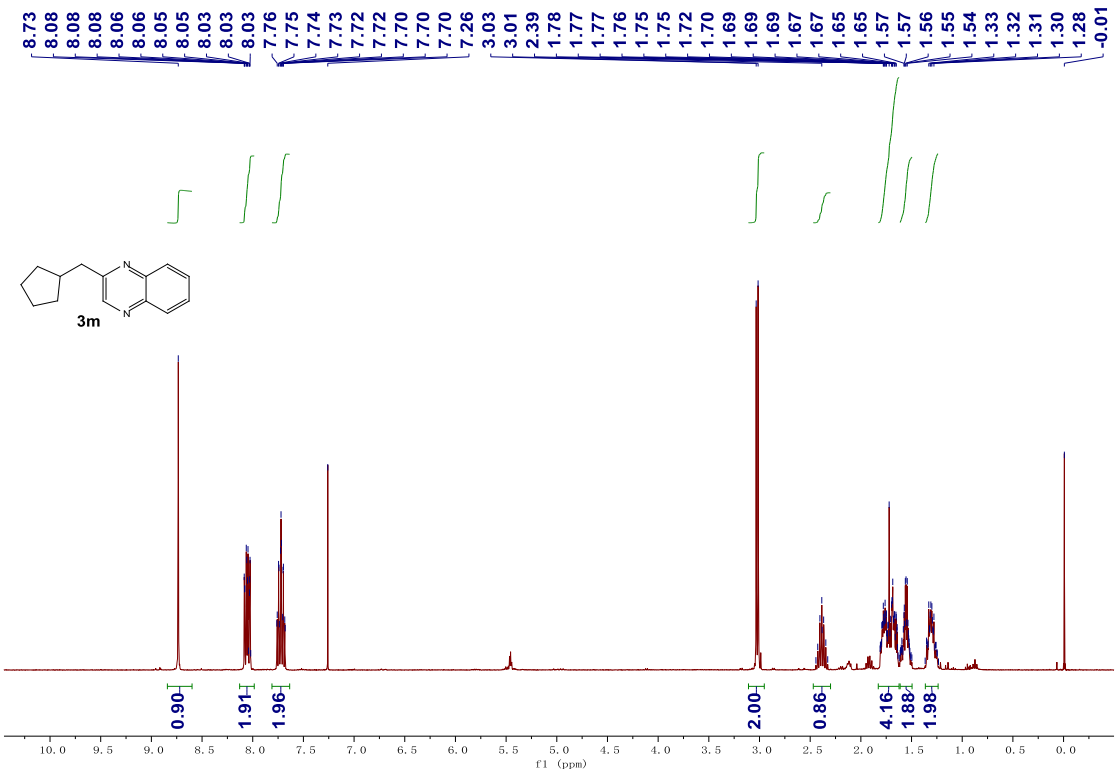

192.03
149.90
134.32
129.77
129.40
77.32
77.00
76.68
42.26
41.66
32.41
24.84
3h, 3i, 3j
f1 (ppm)

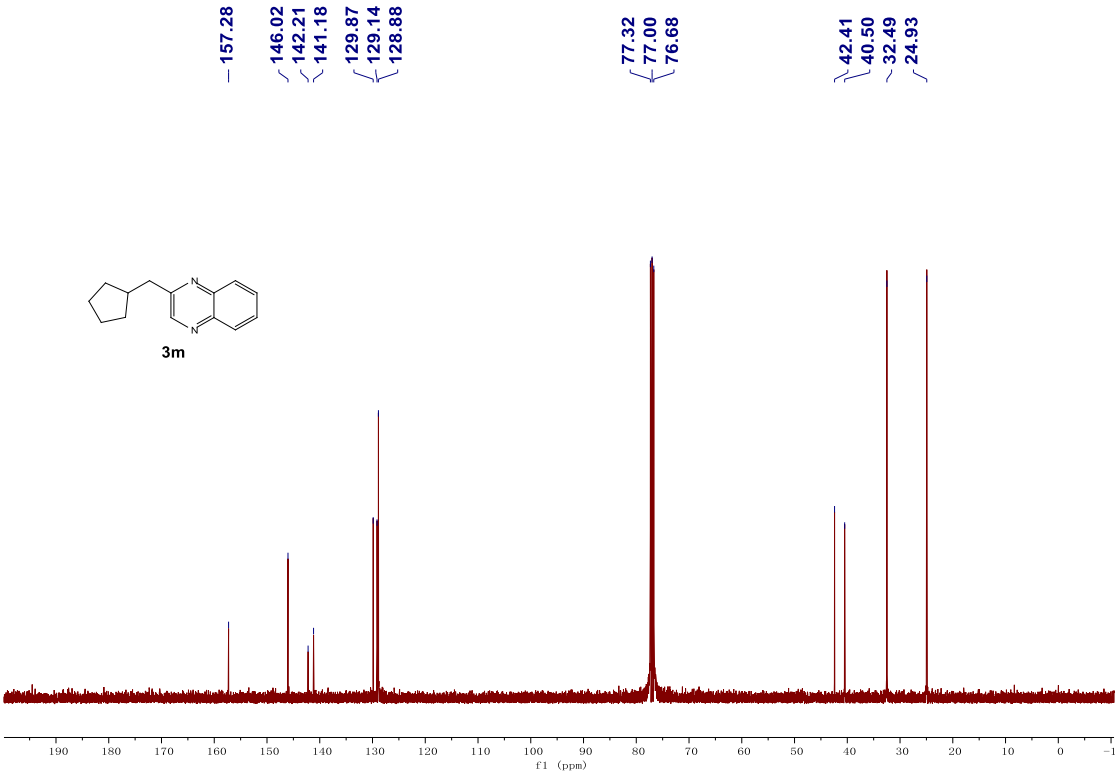

7.39 7.37 7.32 7.26 7.12 7.10 2.57 2.55 2.15 2.06 2.05 2.02 2.00 1.71 1.70 1.69 1.68 1.67 1.67 1.66 1.66 1.65 1.64 1.64 1.63 1.63 1.62 1.62 1.61 1.61 1.60 1.59 1.59 1.53 1.52 1.52 1.51 1.50 1.49 1.48 1.48 1.48 1.19 1.17 1.17 1.16 1.15 1.15 1.14 -0.00
NH
O
CH3
3k
1.88 0.81 1.82 1.65 3.00 1.16 4.23 1.93 1.71
f1 (ppm)

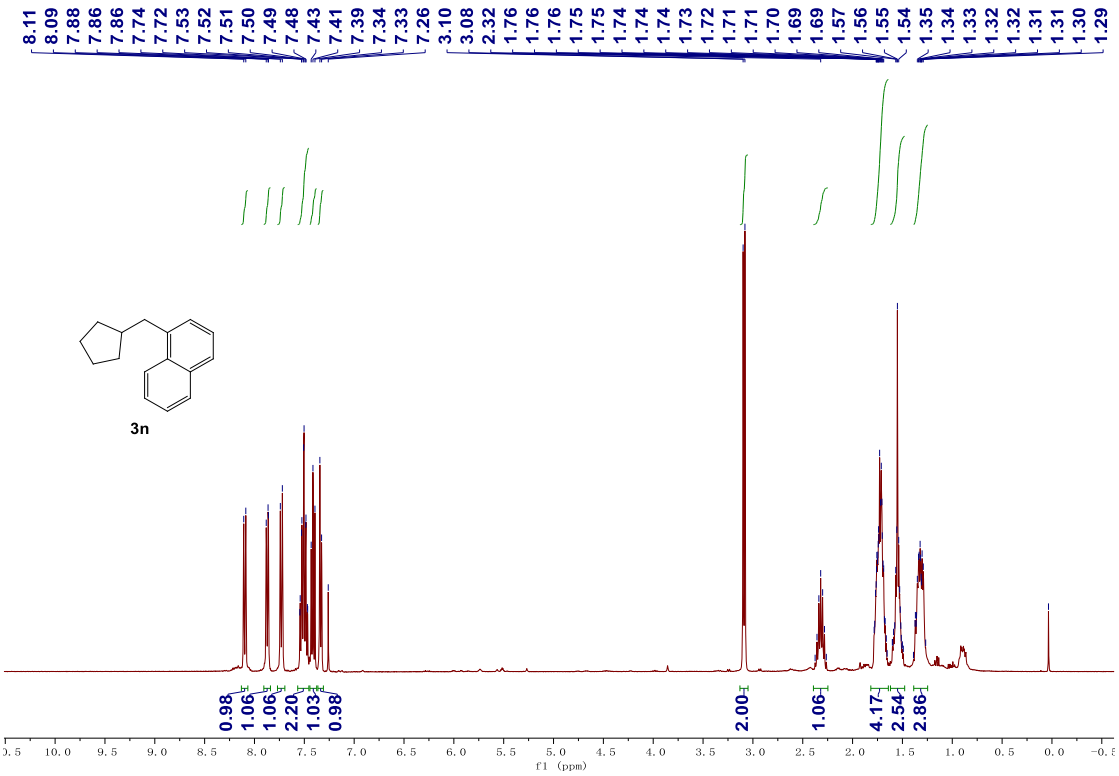

168.25
138.52 135.41 129.19 119.86
77.32 77.00 76.68
41.95 41.44 32.36 24.89 24.51
NH
O
CH3
3k
f1 (ppm)

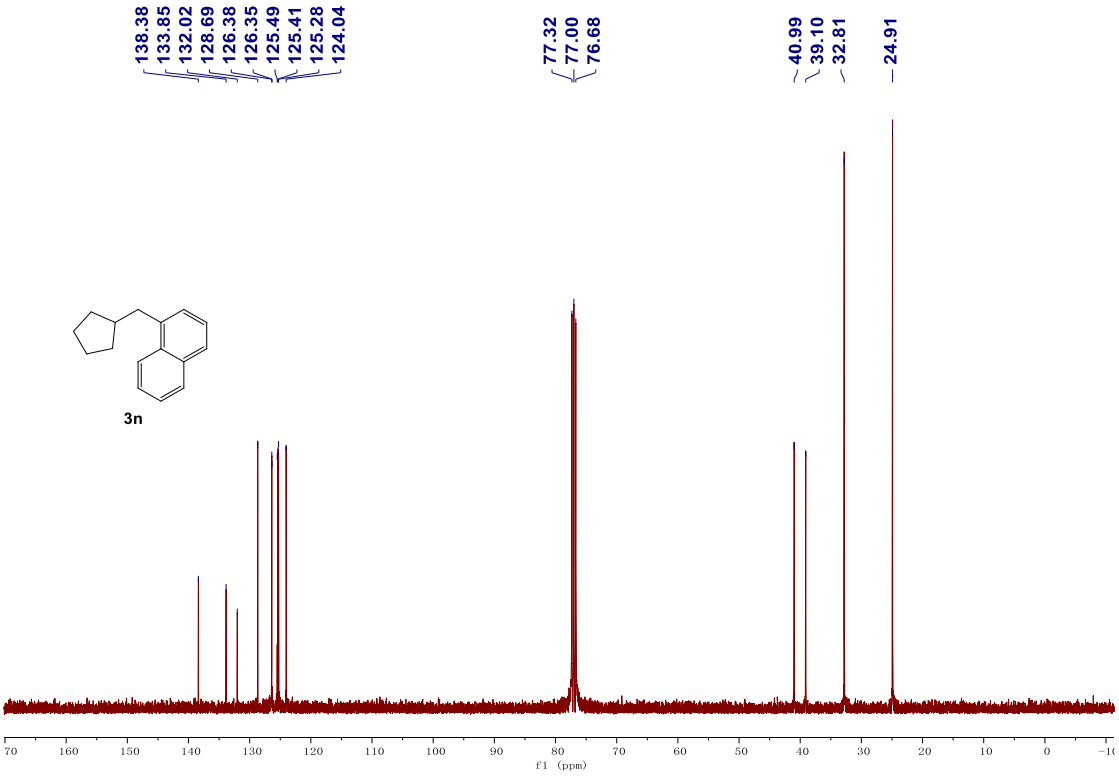

9.51
7.26
7.17
7.16
6.24
6.23
6.23
6.23
6.22
6.22
2.72
2.70
2.26
2.24
2.22
1.79
1.78
1.78
1.77
1.77
1.76
1.76
1.64
1.64
1.63
1.63
1.62
1.62
1.61
1.59
1.58
1.57
1.57
1.56
1.56
1.55
1.55
1.55
1.54
1.54
1.53
1.53
1.23
1.22
1.21
1.20
1.19
1.19
1.18
-0.01
3l
0.89
0.88
0.93
2.00
0.98
2.02
4.40
2.04
f1 (ppm)

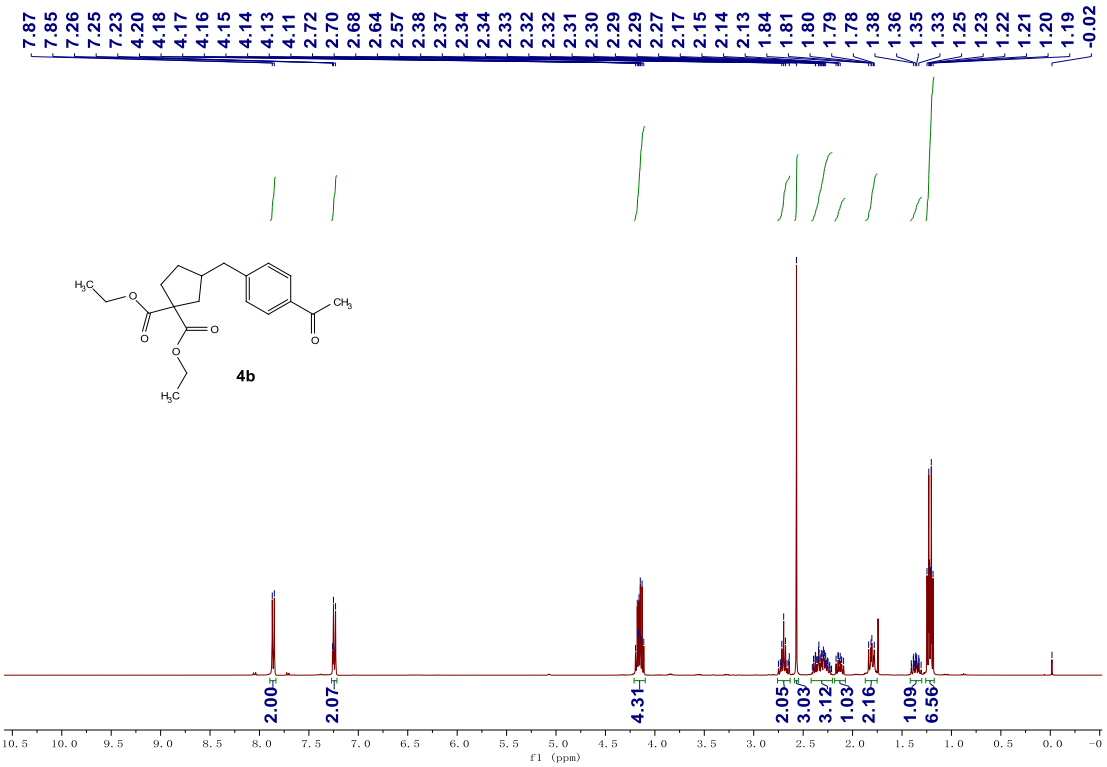

176.91
163.83
151.71
140.52
109.01
77.32
77.00
76.68
38.55
34.39
32.44
24.96
3l
f1 (ppm)

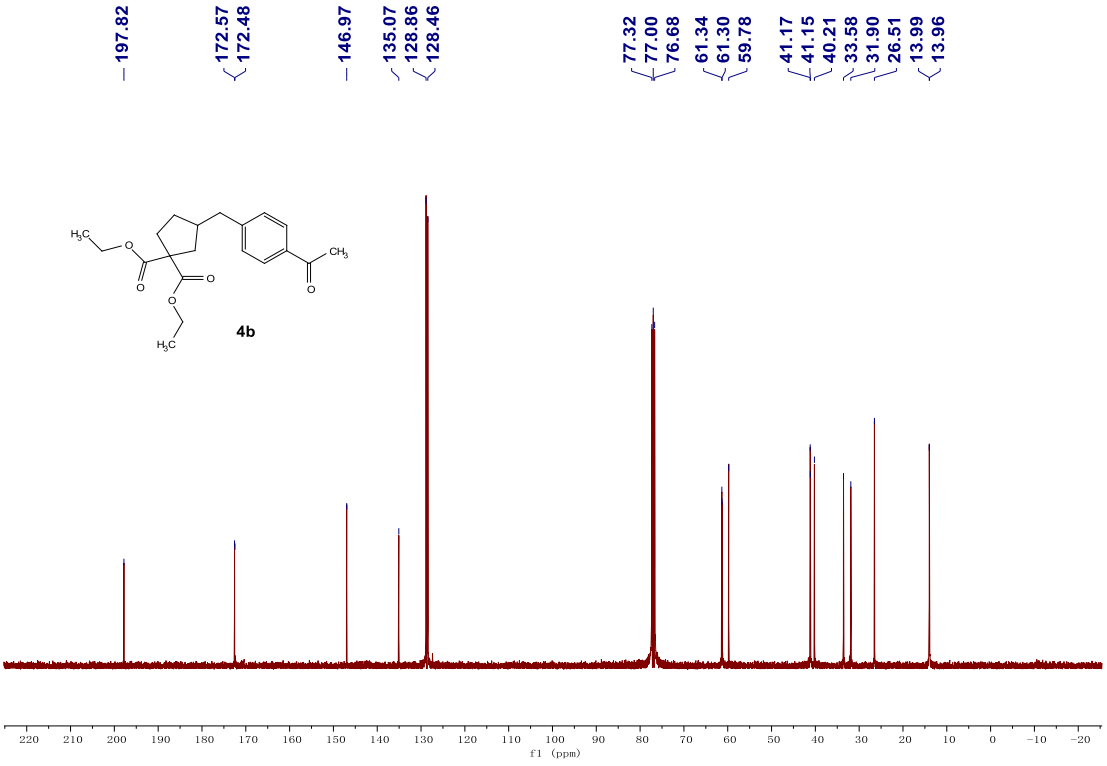

8.73 8.08 8.08 8.08 8.06 8.06 8.05 8.05 8.03 8.03 8.03 7.76 7.75 7.74 7.73 7.72 7.72 7.70 7.70 7.70 7.26 3.03 3.01 2.39 1.78 1.77 1.77 1.76 1.75 1.75 1.72 1.70 1.69 1.69 1.67 1.67 1.65 1.65 1.57 1.57 1.56 1.55 1.54 1.33 1.32 1.31 1.30 1.28 -0.01
3m
0.90 1.91 1.96 2.00 0.86 4.16 1.88 1.98
f1 (ppm)

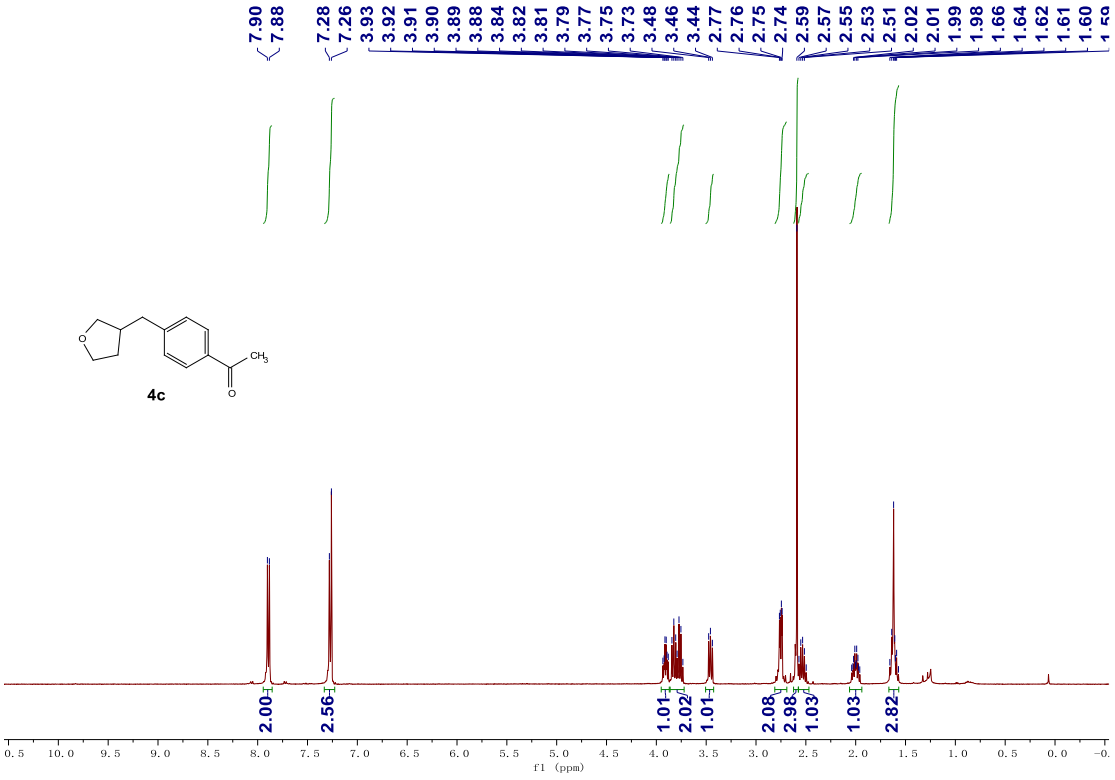

157.28 146.02 142.21 141.18 129.87 129.14 128.88 77.32 77.00 76.68 42.41 40.50 32.49 24.93
3m
f1 (ppm)

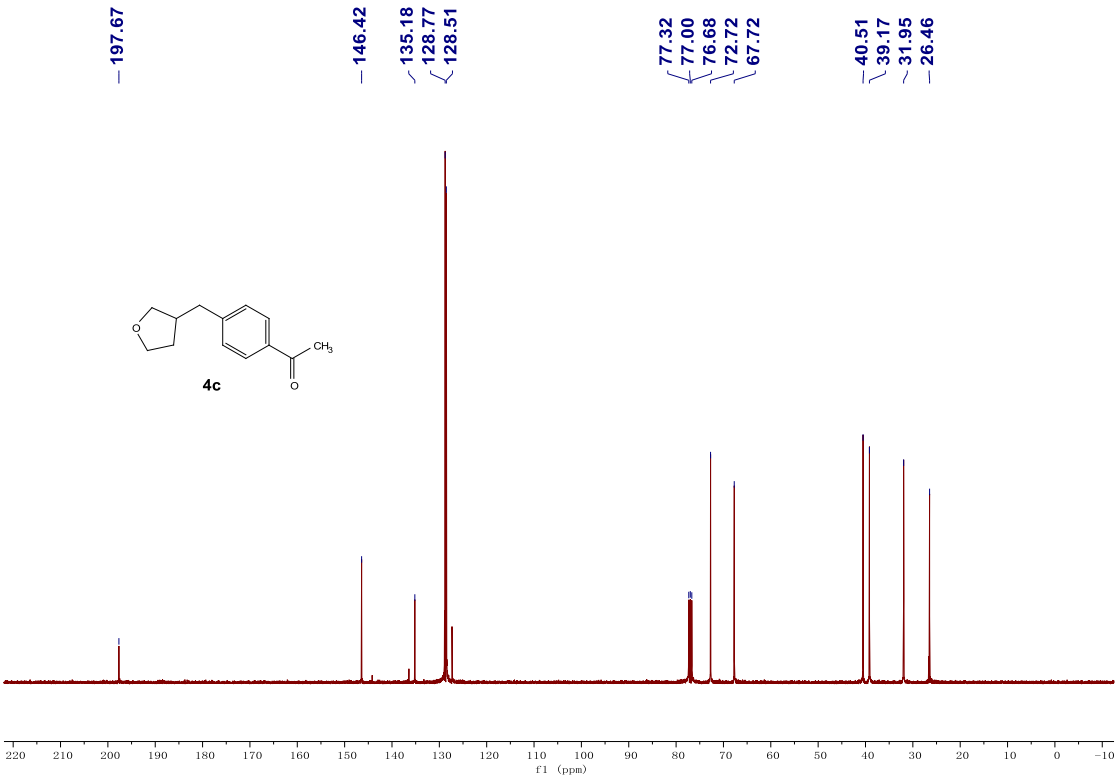

8.11
8.09
7.88
7.86
7.86
7.74
7.72
7.53
7.52
7.51
7.50
7.49
7.48
7.43
7.41
7.39
7.34
7.33
7.26
3.10
3.08
2.32
1.76
1.76
1.76
1.75
1.75
1.74
1.74
1.74
1.73
1.72
1.71
1.71
1.70
1.69
1.69
1.57
1.56
1.55
1.54
1.35
1.34
1.33
1.32
1.32
1.31
1.31
1.30
1.29
3n
0.98
1.06
1.06
2.20
1.03
0.98
2.00
1.06
4.17
2.54
2.86
f1 (ppm)

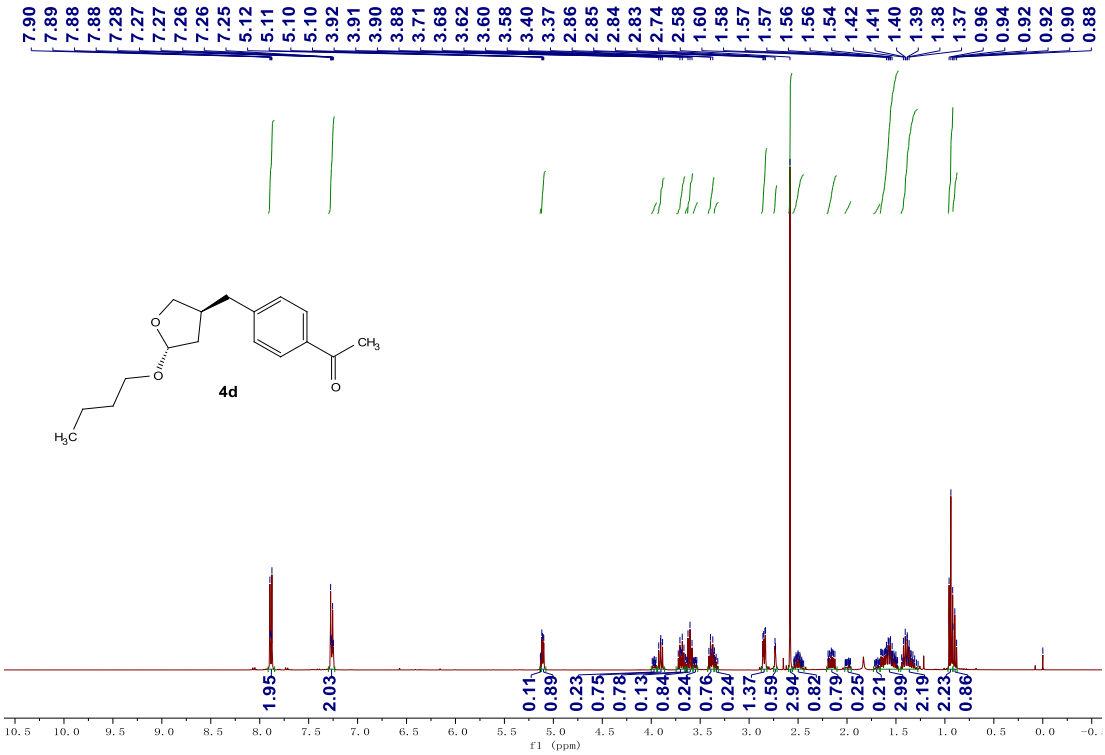

138.38
133.85
132.02
128.69
126.38
126.35
125.49
125.41
125.28
124.04
77.32
77.00
76.68
40.99
39.10
32.81
24.91
3n
f1 (ppm)

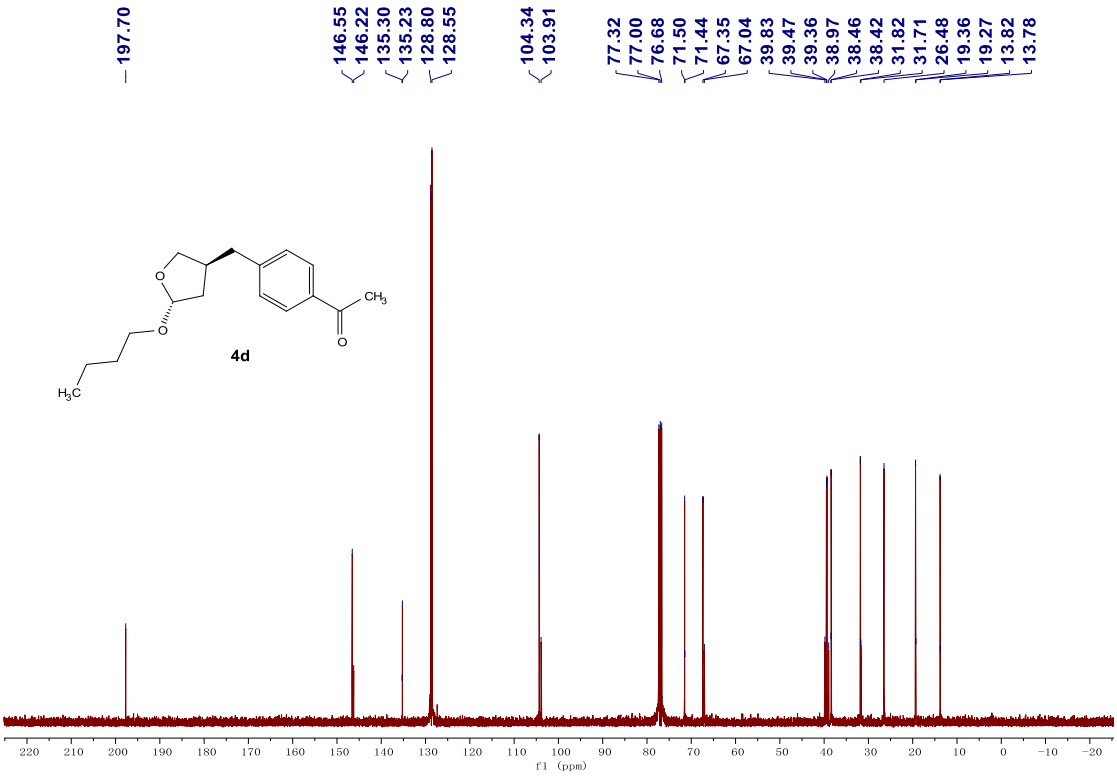

7.87
7.85
7.26
7.25
7.23
4.20
4.18
4.17
4.16
4.15
4.14
4.13
4.11
2.72
2.70
2.68
2.64
2.57
2.38
2.37
2.34
2.34
2.33
2.32
2.32
2.31
2.30
2.29
2.29
2.27
2.17
2.15
2.14
2.13
1.84
1.81
1.80
1.79
1.78
1.38
1.36
1.35
1.33
1.25
1.23
1.22
1.21
1.20
1.19
-0.02
4b
2.00
2.07
4.31
2.05
3.03
3.12
1.03
2.16
1.09
6.56
f1 (ppm)

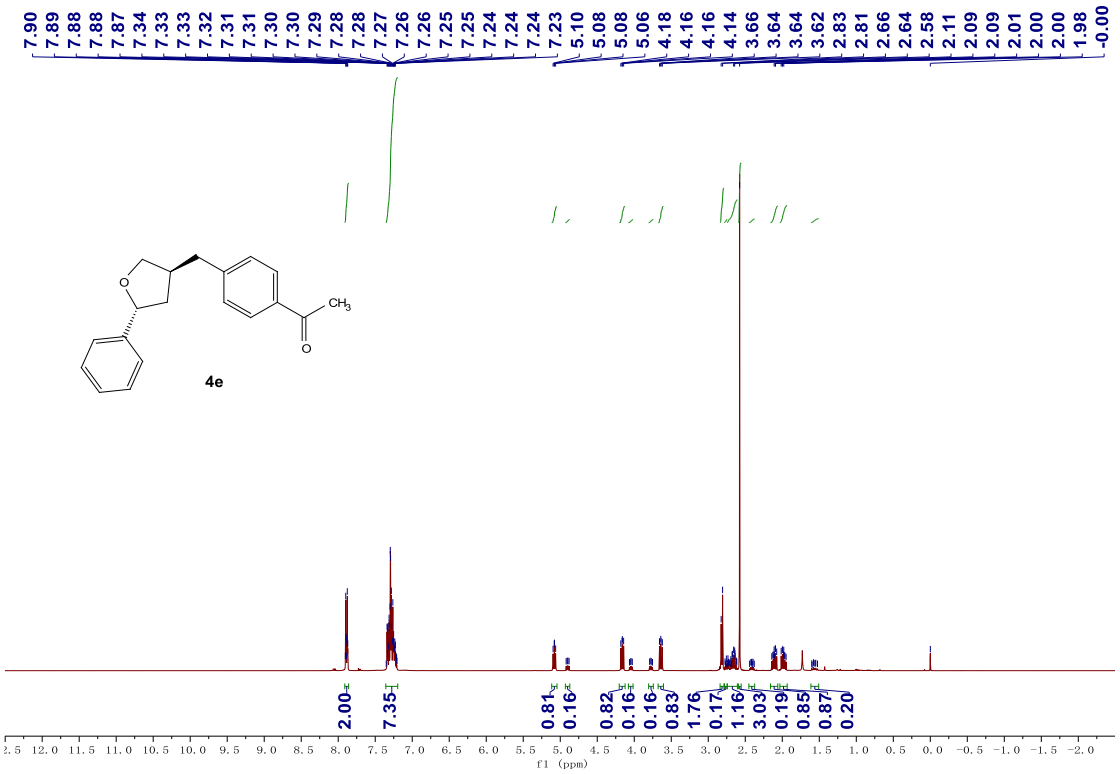

197.82
172.57
172.48
146.97
135.07
128.86
128.46
77.32
77.00
76.68
61.34
61.30
59.78
41.17
41.15
40.21
33.58
31.90
26.51
13.99
13.96
4b
f1 (ppm)

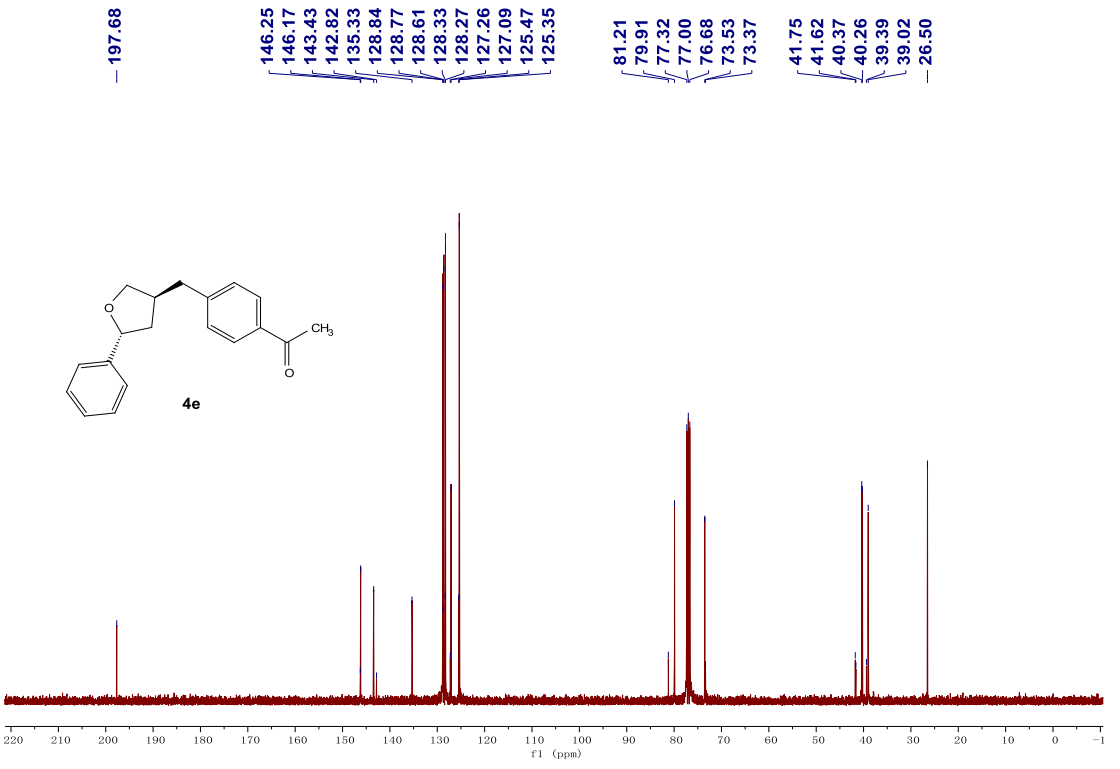

7.90
7.88
7.28
7.26
3.93
3.92
3.91
3.90
3.89
3.88
3.84
3.82
3.81
3.79
3.77
3.75
3.73
3.48
3.46
3.44
2.77
2.76
2.75
2.74
2.59
2.57
2.55
2.53
2.51
2.02
2.01
1.99
1.98
1.66
1.64
1.62
1.61
1.60
2.00
2.56
1.01
2.02
1.01
2.08
2.98
1.03
1.03
2.82
f1 (ppm)
4c

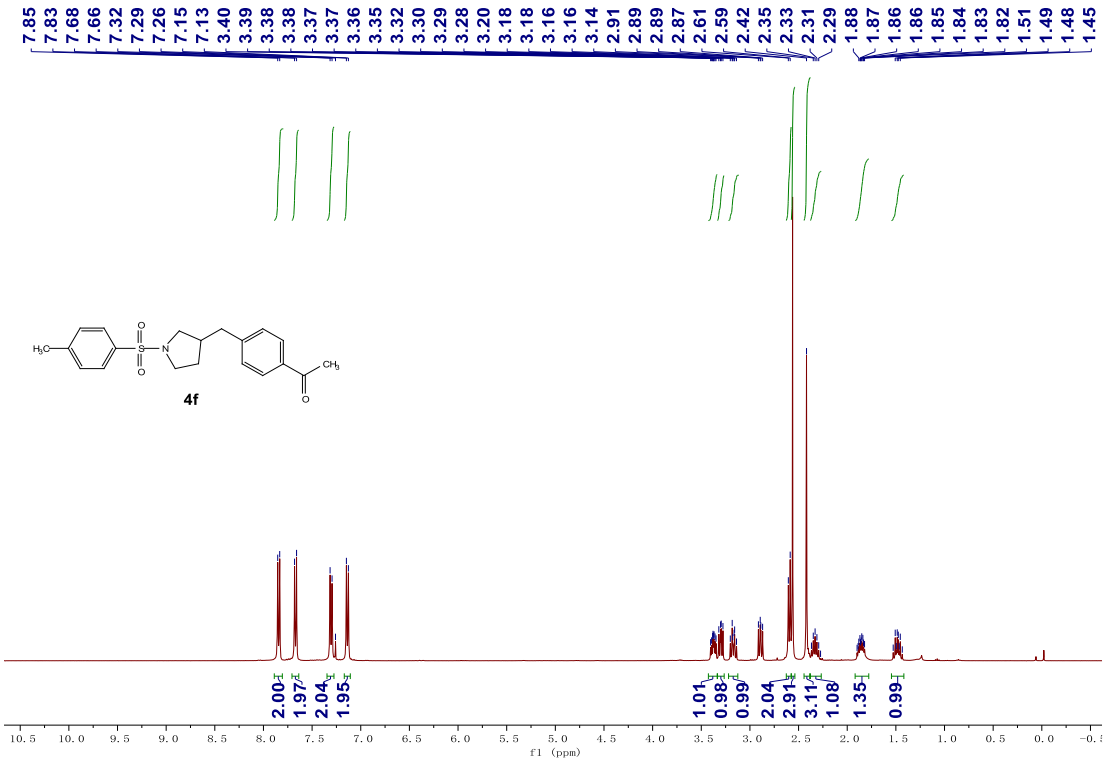

197.67
146.42
135.18
128.77
128.51
77.32
77.00
76.68
72.72
67.72
40.51
39.17
31.95
26.46
f1 (ppm)
4c

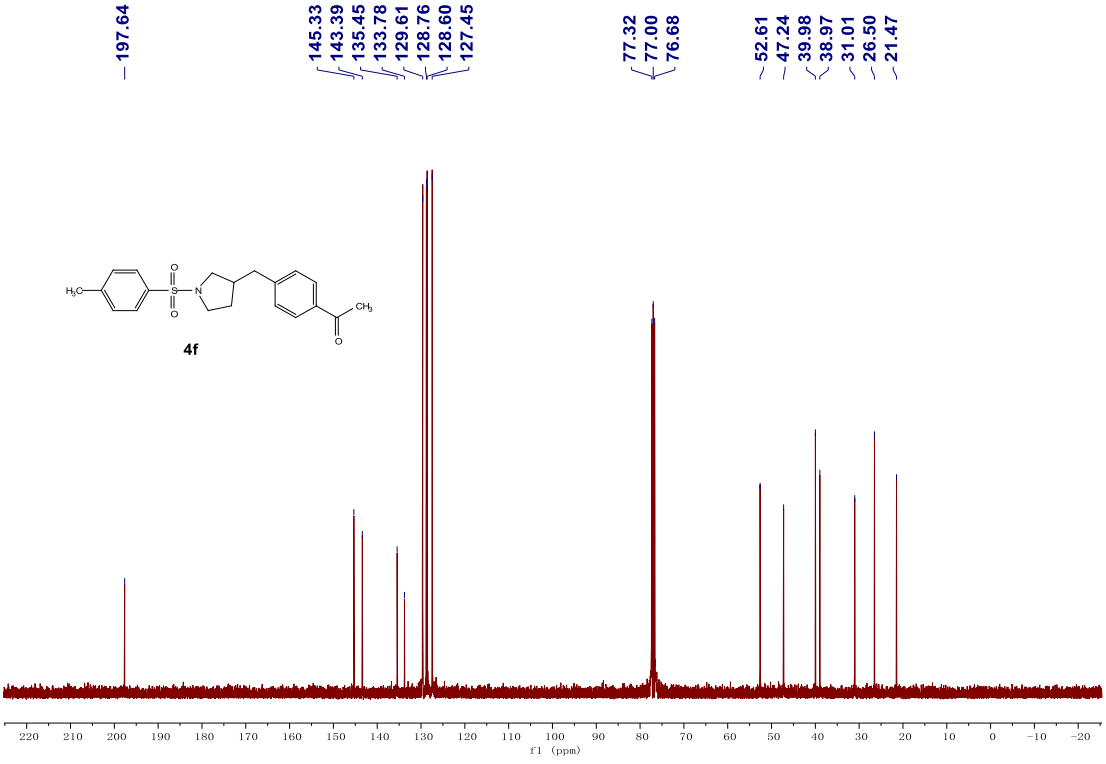

7.90
7.89
7.88
7.88
7.28
7.27
7.27
7.26
7.26
7.25
5.12
5.11
5.10
5.10
3.92
3.91
3.90
3.88
3.71
3.68
3.62
3.60
3.58
3.40
3.37
2.86
2.85
2.84
2.83
2.74
2.58
1.60
1.58
1.57
1.57
1.56
1.56
1.54
1.42
1.41
1.40
1.39
1.38
1.37
0.96
0.94
0.92
0.92
0.90
0.88
4d
1.95
2.03
0.11
0.89
0.23
0.75
0.78
0.13
0.84
0.24
0.76
0.24
1.37
0.59
2.94
0.82
0.79
0.25
0.21
2.99
2.19
2.23
0.86
f1 (ppm)

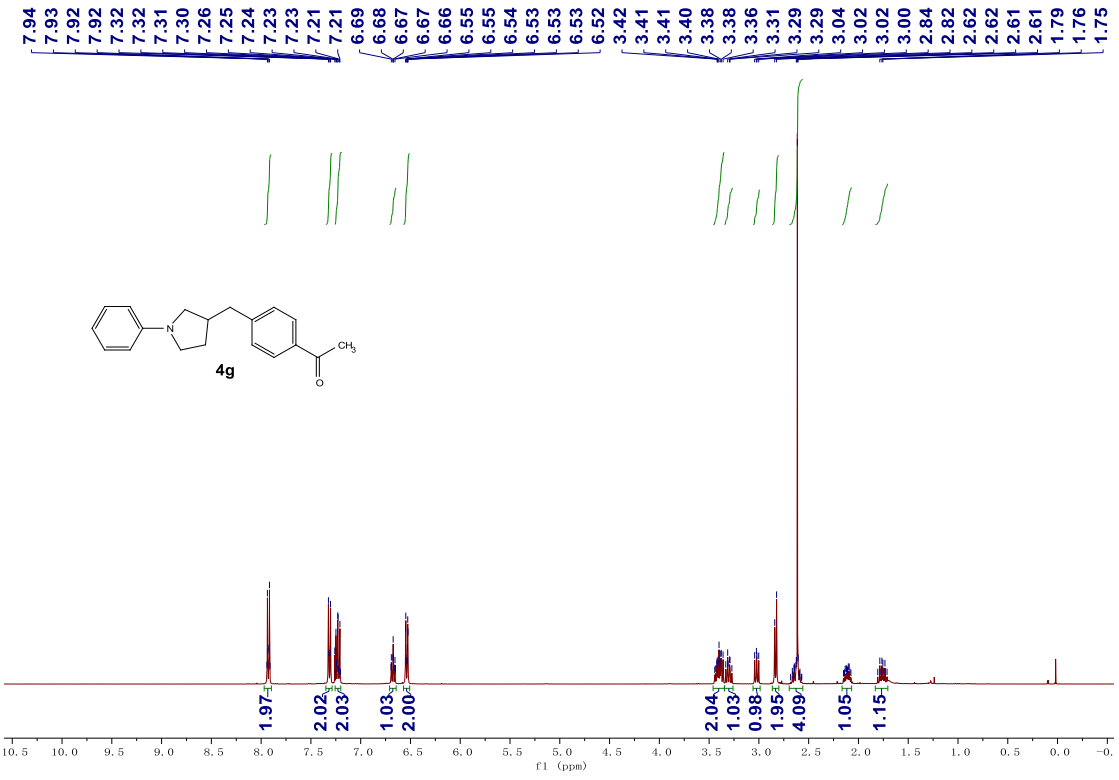

197.70
146.55
146.22
135.30
135.23
128.80
128.55
104.34
103.91
77.32
77.00
76.68
71.50
71.44
67.35
67.04
39.83
39.47
39.36
38.97
38.46
38.42
31.82
31.71
26.48
19.36
19.27
13.82
13.78
4d
f1 (ppm)

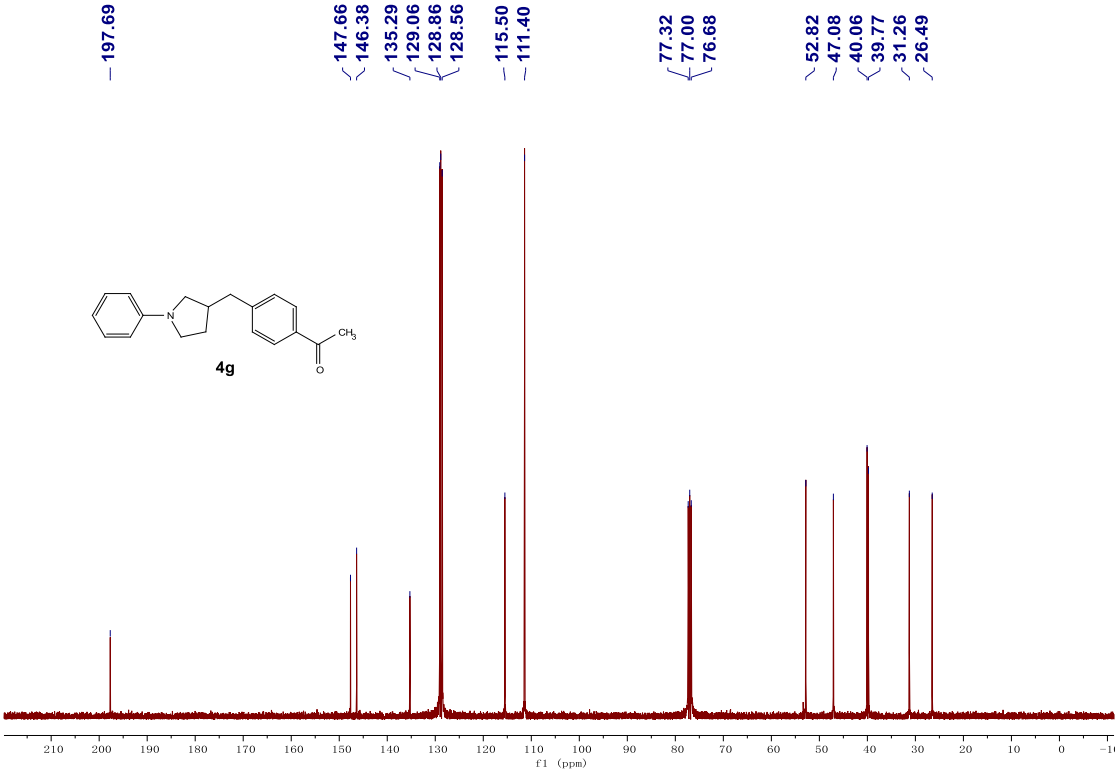

7.90 7.89 7.88 7.88 7.87 7.34 7.33 7.33 7.32 7.31 7.31 7.30 7.30 7.29 7.28 7.28 7.27 7.26 7.26 7.25 7.25 7.24 7.24 7.24 7.23 5.10 5.08 5.08 5.06 4.18 4.16 4.16 4.14 3.66 3.64 3.64 3.62 2.83 2.81 2.66 2.64 2.58 2.11 2.09 2.09 2.01 2.00 2.00 1.98 -0.00
O
CH3
O
4e
2.00 7.35 0.81 0.16 0.82 0.16 0.16 0.83 1.76 0.17 1.16 3.03 0.19 0.85 0.87 0.20
f1 (ppm)

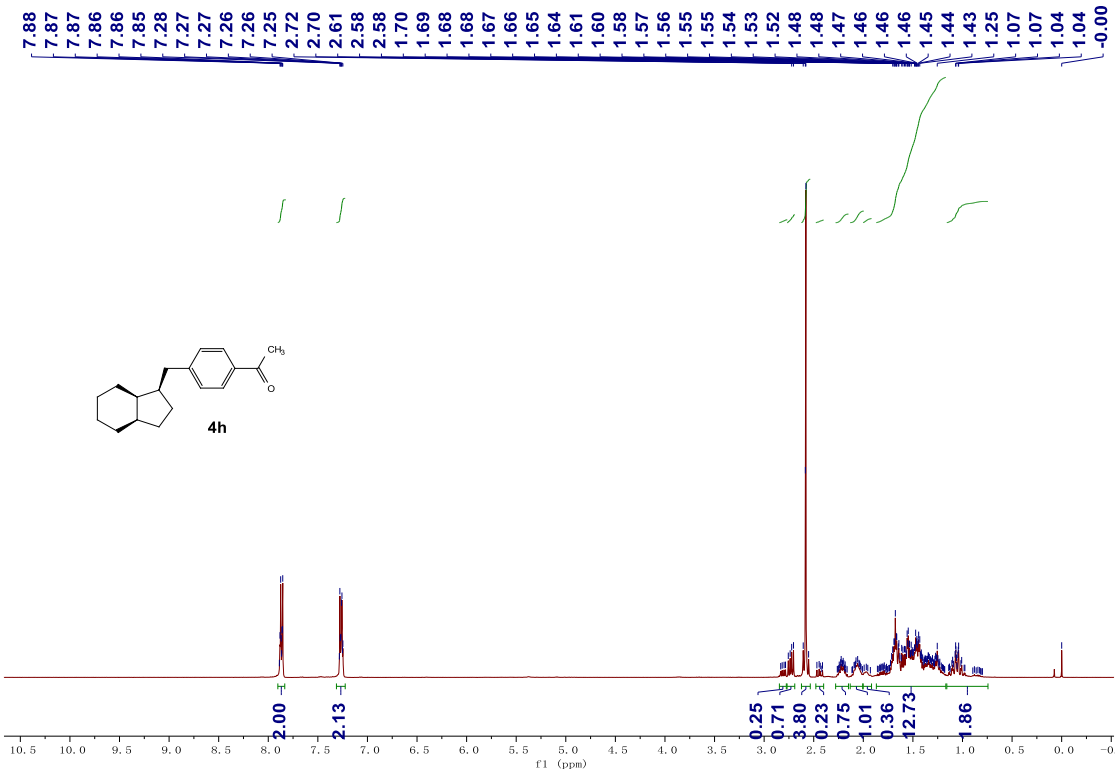

197.68
146.25 146.17 143.43 142.82 135.33 128.84 128.77 128.61 128.33 128.27 127.26 127.09 125.47 125.35
81.21 79.91 77.32 77.00 76.68 73.53 73.37
41.75 41.62 40.37 40.26 39.39 39.02 26.50
O
CH3
O
4e
f1 (ppm)

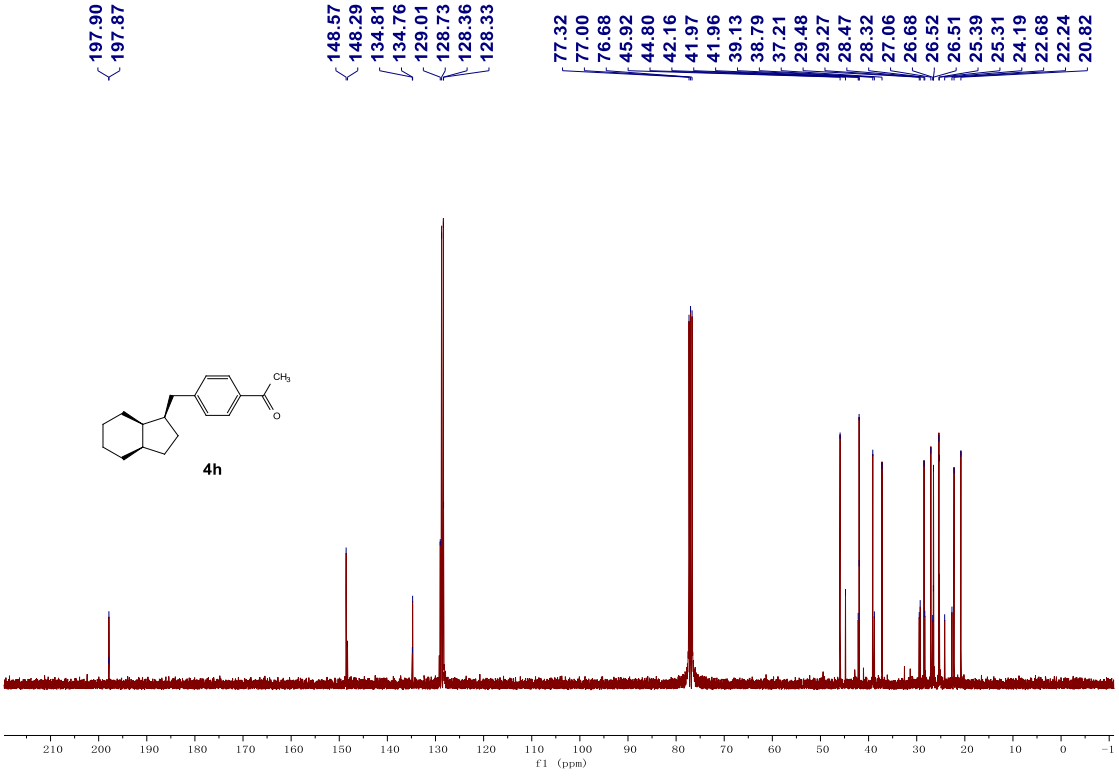

7.85 7.83 7.68 7.66 7.32 7.29 7.26 7.15 7.13 3.40 3.39 3.38 3.38 3.37 3.37 3.36 3.35 3.32 3.30 3.29 3.28 3.20 3.18 3.18 3.16 3.16 3.14 2.91 2.89 2.89 2.87 2.61 2.59 2.42 2.35 2.33 2.31 2.29 1.88 1.87 1.86 1.86 1.85 1.84 1.83 1.82 1.51 1.49 1.48 1.45
4f
2.00 1.97 2.04 1.95 1.01 0.98 0.99 2.04 2.91 3.11 1.08 1.35 0.99
f1 (ppm)

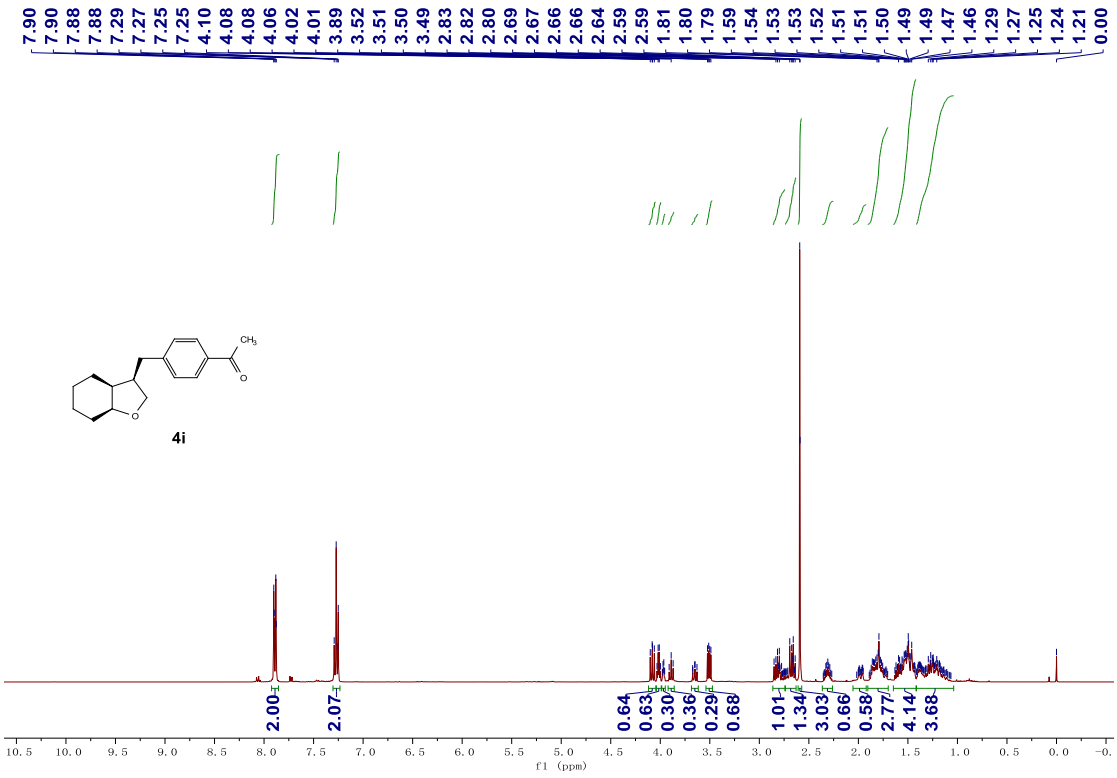

197.64
145.33 143.39 135.45 133.78 129.61 128.76 128.60 127.45
77.32 77.00 76.68
52.61 47.24 39.98 38.97 31.01 26.50 21.47
4f
f1 (ppm)

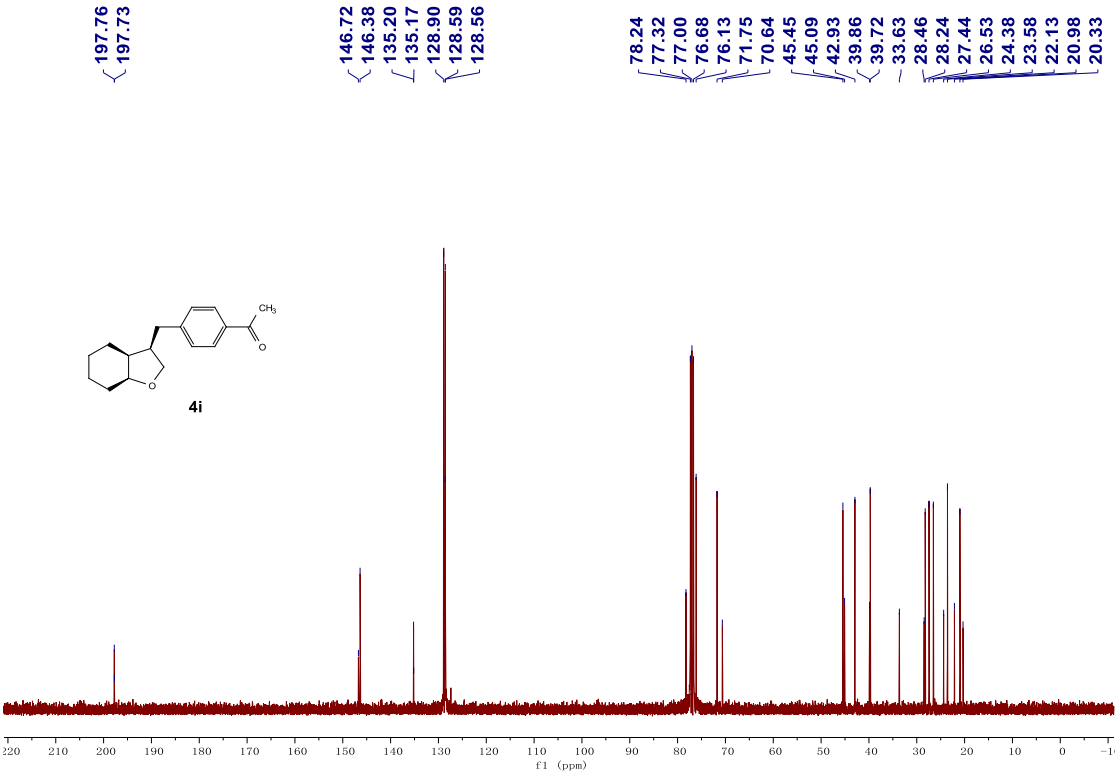

7.94 7.93 7.92 7.92 7.32 7.32 7.31 7.30 7.26 7.25 7.24 7.23 7.23 7.21 7.21 6.69 6.68 6.67 6.67 6.66 6.55 6.55 6.54 6.53 6.53 6.52 3.42 3.41 3.41 3.40 3.38 3.38 3.36 3.31 3.29 3.29 3.04 3.02 3.02 3.00 2.84 2.82 2.62 2.62 2.61 2.61 1.79 1.76 1.75
4g
1.97 2.02 2.03 1.03 2.00 2.04 1.03 0.98 1.95 4.09 1.05 1.15
f1 (ppm)

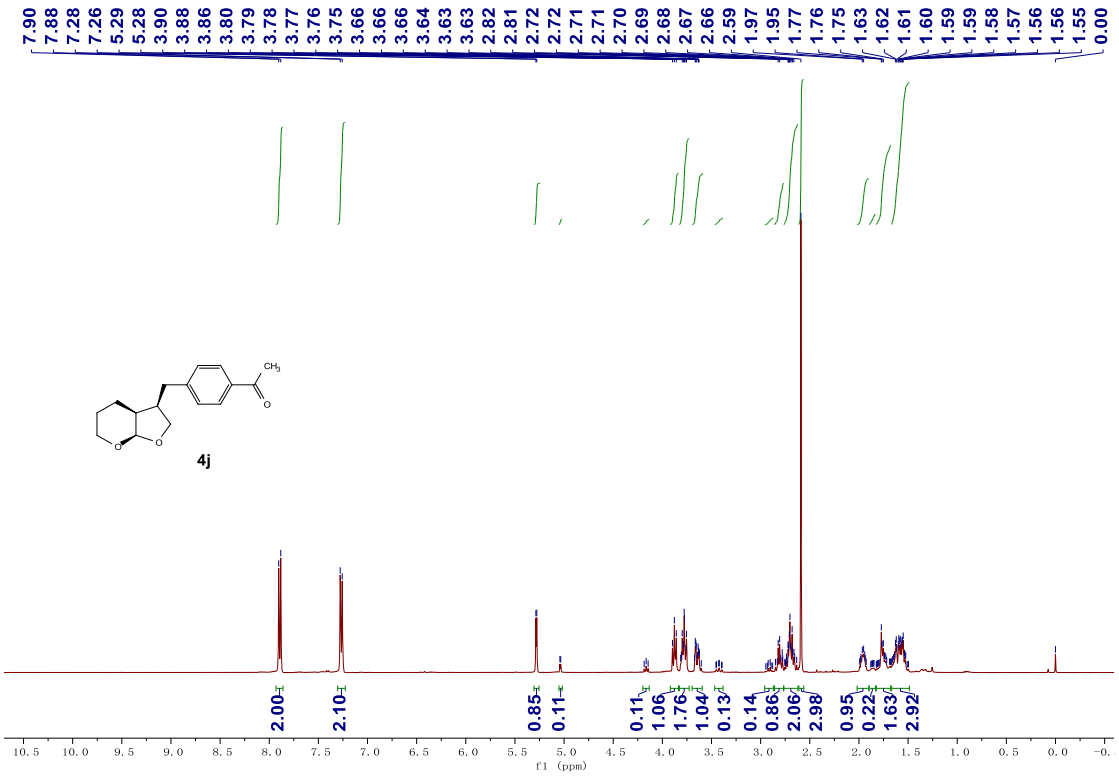

197.69
147.66 146.38 135.29 129.06 128.86 128.56 115.50 111.40 77.32 77.00 76.68 52.82 47.08 40.06 39.77 31.26 26.49
4g
f1 (ppm)

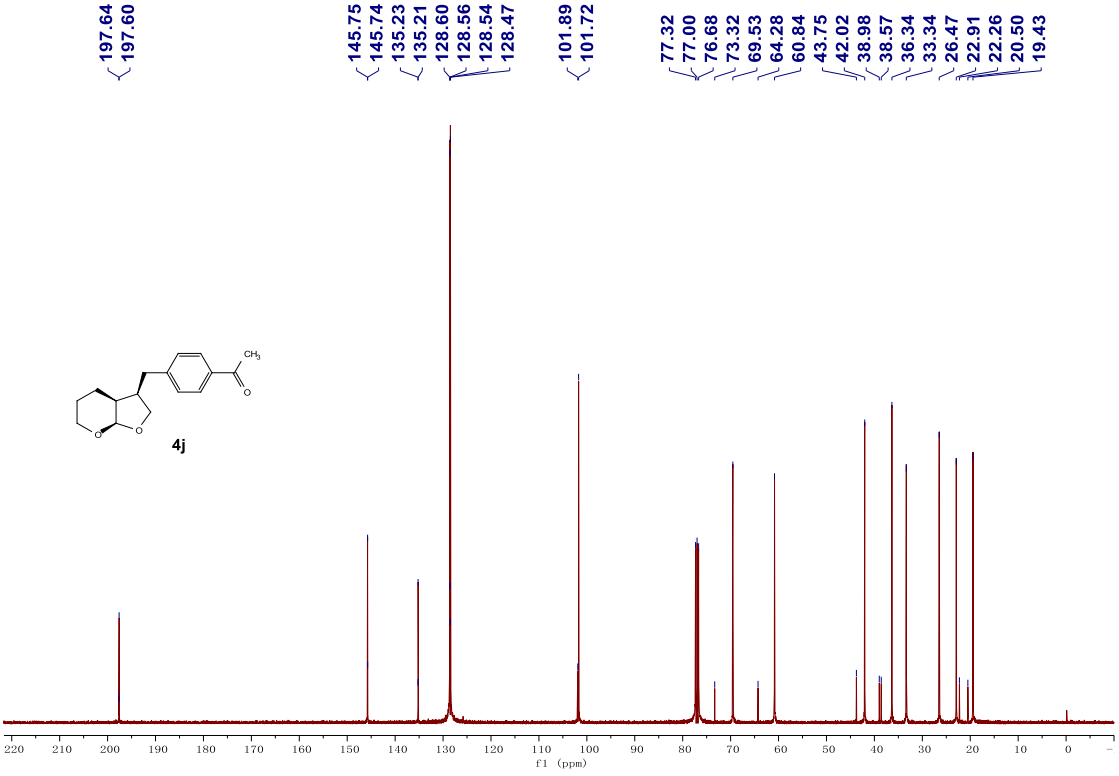

7.88
7.87
7.87
7.86
7.86
7.85
7.28
7.27
7.27
7.26
7.26
7.25
2.72
2.70
2.61
2.58
2.58
1.70
1.69
1.68
1.68
1.67
1.66
1.65
1.64
1.61
1.60
1.58
1.57
1.56
1.55
1.55
1.54
1.53
1.52
1.48
1.48
1.47
1.46
1.46
1.46
1.45
1.44
1.43
1.25
1.07
1.07
1.04
1.04
-0.00
CH3
O
4h
2.00
2.13
0.25
0.71
3.80
0.23
0.75
1.01
0.36
12.73
1.86
f1 (ppm)

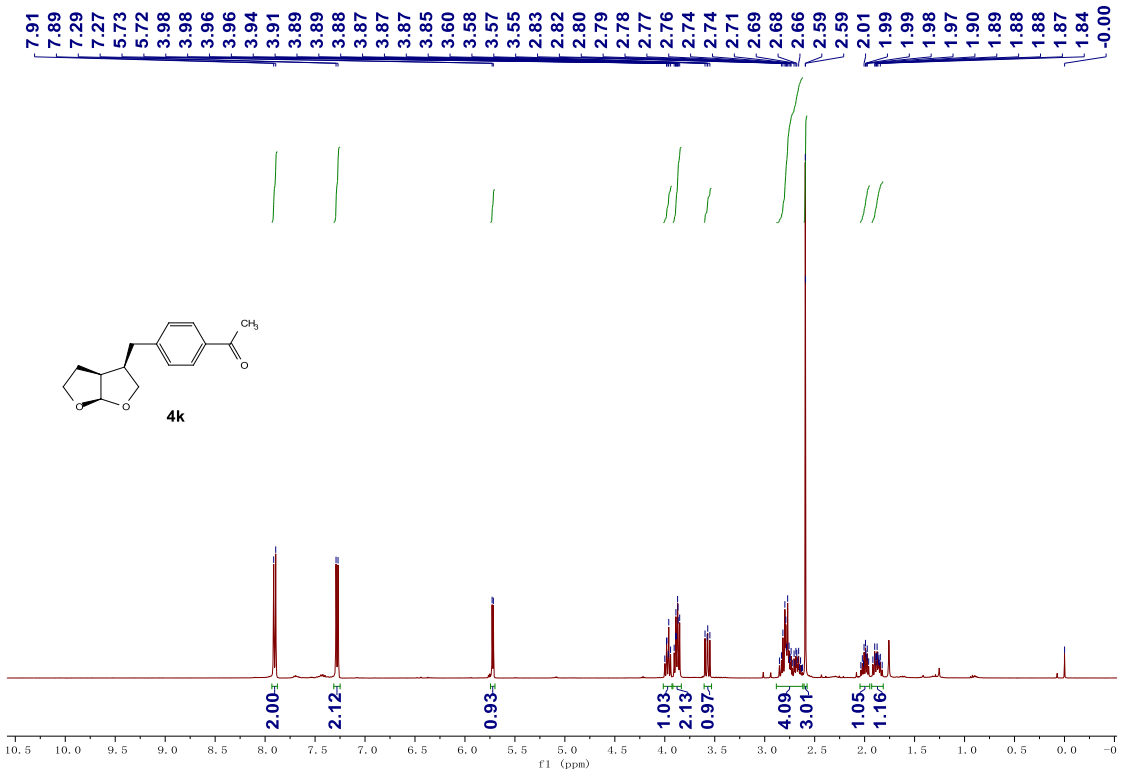

197.90
197.87
148.57
148.29
134.81
134.76
129.01
128.73
128.36
128.33
77.32
77.00
76.68
45.92
44.80
42.16
41.97
41.96
39.13
38.79
37.21
29.48
29.27
28.47
28.32
27.06
26.68
26.52
26.51
25.39
25.31
24.19
22.68
22.24
20.82
CH3
O
4h
f1 (ppm)

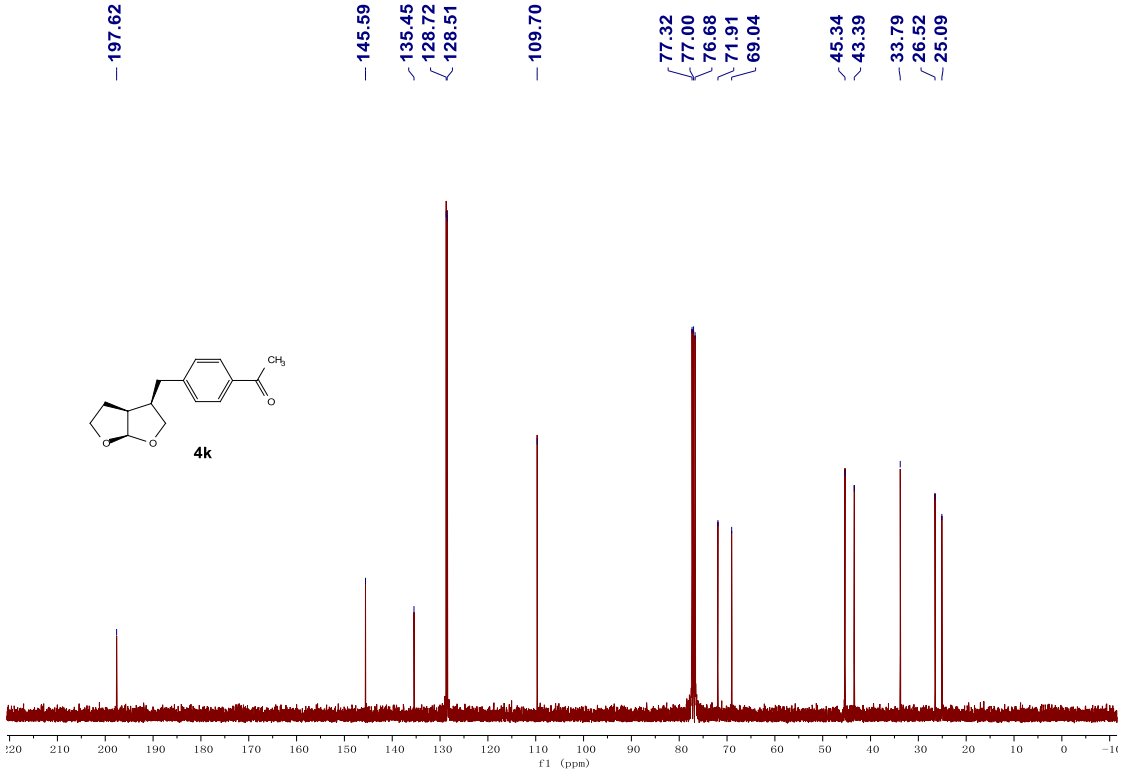

7.90 7.90 7.88 7.88 7.29 7.27 7.25 7.25 4.10 4.08 4.08 4.06 4.02 4.01 3.89 3.52 3.51 3.50 3.49 2.83 2.82 2.80 2.69 2.67 2.66 2.66 2.64 2.59 2.59 1.81 1.80 1.79 1.59 1.54 1.53 1.53 1.52 1.51 1.51 1.50 1.49 1.49 1.47 1.46 1.29 1.27 1.25 1.24 1.21 0.00
CH3
O
O
4i
2.00 2.07 0.64 0.63 0.30 0.36 0.29 0.68 1.01 1.34 3.03 0.66 0.58 2.77 4.14 3.68
f1 (ppm)

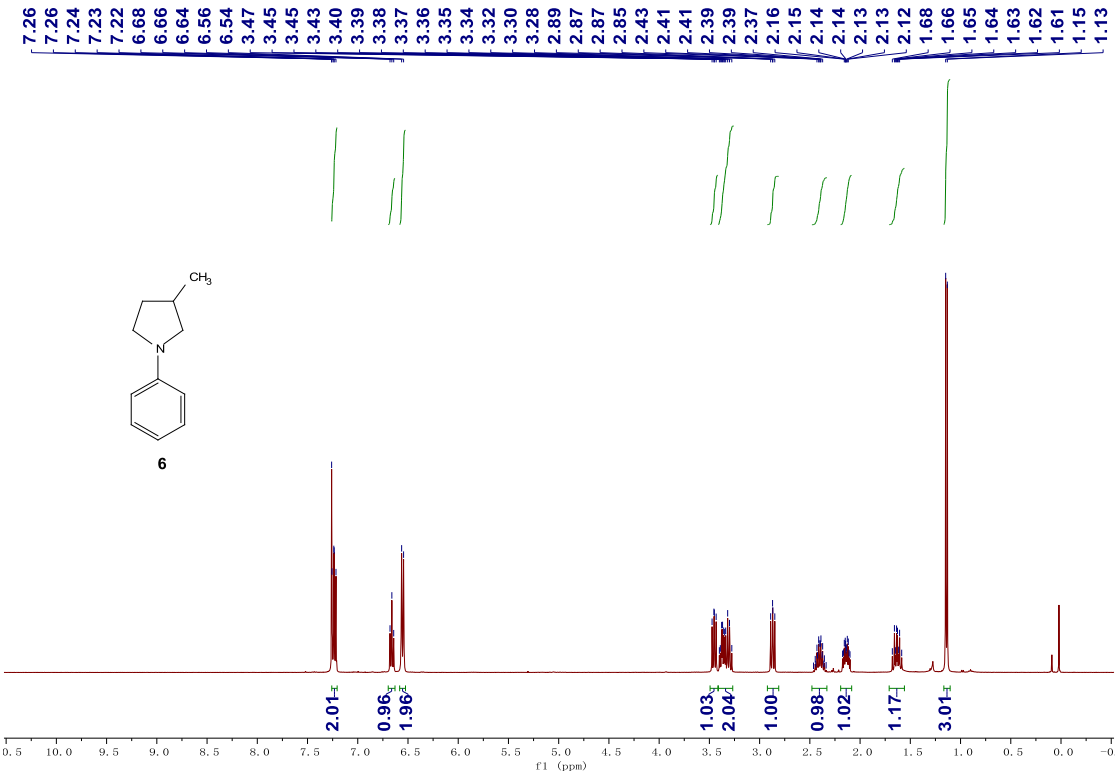

197.76 197.73 146.72 146.38 135.20 135.17 128.90 128.59 128.56 78.24 77.32 77.00 76.68 76.13 71.75 70.64 45.45 45.09 42.93 39.86 39.72 33.63 28.46 28.24 27.44 26.53 24.38 23.58 22.13 20.98 20.33
CH3
O
O
4i
f1 (ppm)

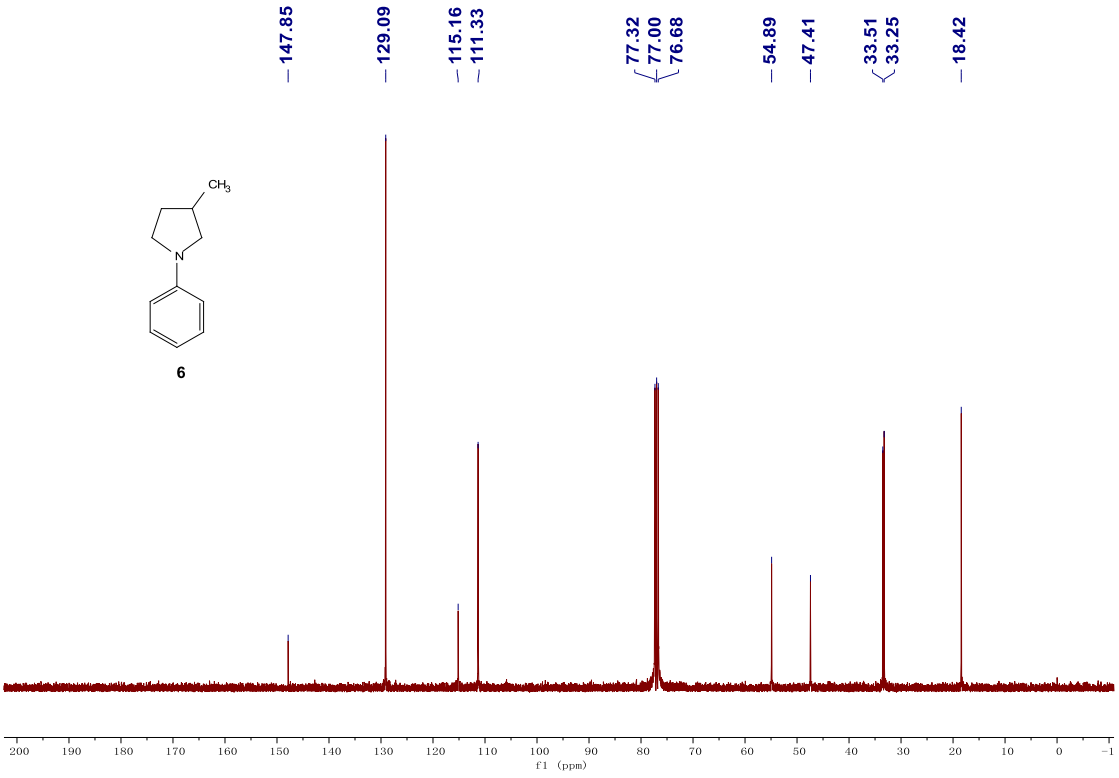

7.90 7.88 7.28 7.26 5.29 5.28 3.90 3.88 3.86 3.80 3.79 3.78 3.77 3.76 3.75 3.66 3.66 3.64 3.63 3.63 2.82 2.81 2.72 2.72 2.71 2.71 2.70 2.69 2.68 2.67 2.66 2.59 1.97 1.95 1.77 1.76 1.75 1.63 1.62 1.61 1.60 1.59 1.59 1.58 1.57 1.56 1.56 1.55 0.00
4j
2.00 2.10 0.85 0.11 0.11 1.06 1.76 1.04 0.13 0.14 0.86 2.06 2.98 0.95 0.22 1.63 2.92
f1 (ppm)

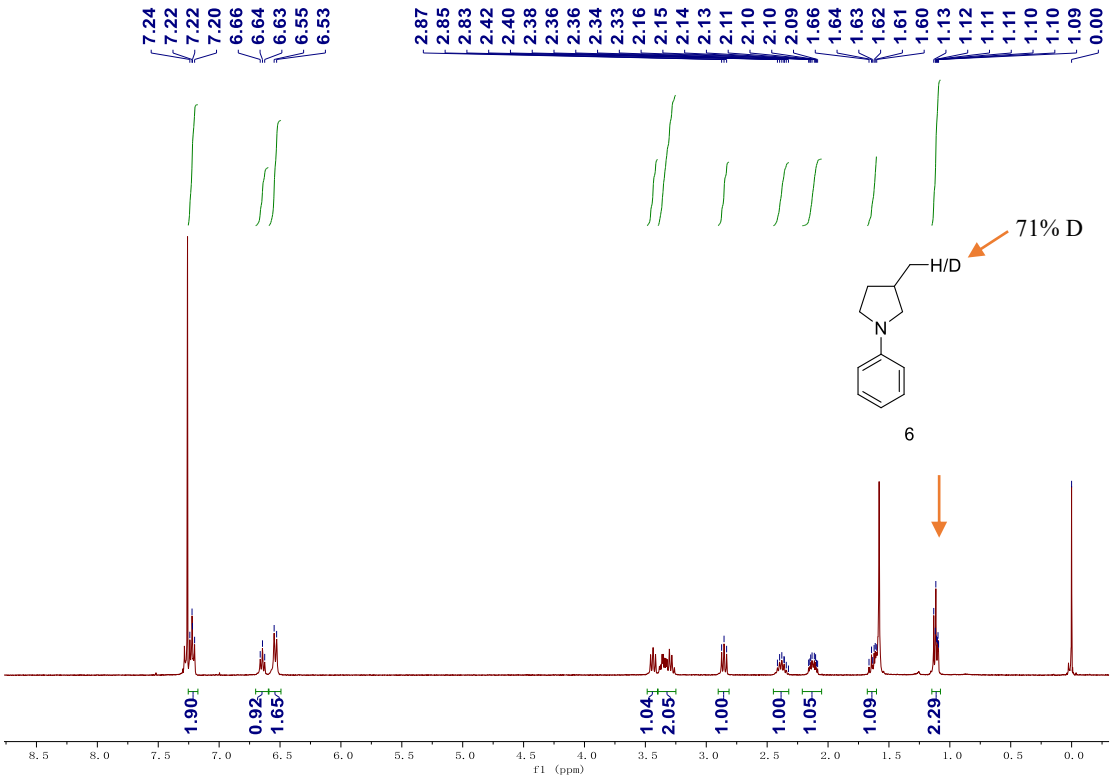

197.64 197.60 145.75 145.74 135.23 135.21 128.60 128.56 128.54 128.47 101.89 101.72 77.32 77.00 76.68 73.32 69.53 64.28 60.84 43.75 42.02 38.98 38.57 36.34 33.34 26.47 22.91 22.26 20.50 19.43
4j
f1 (ppm)


7.91
7.89
7.29
7.27
5.73
5.72
3.98
3.98
3.96
3.96
3.94
3.91
3.89
3.89
3.88
3.87
3.87
3.87
3.85
3.60
3.58
3.57
3.55
2.83
2.82
2.80
2.79
2.78
2.77
2.76
2.74
2.74
2.71
2.69
2.68
2.66
2.59
2.59
2.01
1.99
1.99
1.98
1.97
1.90
1.89
1.88
1.88
1.87
1.84
-0.00
4k
2.00
2.12
0.93
1.03
2.13
0.97
4.09
3.01
1.05
1.16
f1 (ppm)


197.62
145.59
135.45
128.72
128.51
109.70
77.32
77.00
76.68
71.91
69.04
45.34
43.39
33.79
26.52
25.09
4k
f1 (ppm)


7.26
7.26
7.24
7.23
7.22
6.68
6.66
6.64
6.56
6.54
3.47
3.45
3.45
3.43
3.40
3.39
3.38
3.37
3.36
3.35
3.34
3.32
3.30
3.28
2.89
2.87
2.87
2.85
2.43
2.41
2.41
2.39
2.39
2.37
2.16
2.15
2.14
2.14
2.13
2.13
2.12
1.68
1.66
1.65
1.64
1.63
1.62
1.61
1.15
1.13
CH3
N
6
2.01
0.96
1.96
1.03
2.04
1.00
0.98
1.02
1.17
3.01
f1 (ppm)


147.85
129.09
115.16
111.33
77.32
77.00
76.68
54.89
47.41
33.51
33.25
18.42
CH3
N
6
f1 (ppm)


7.24
7.22
7.22
7.20
6.66
6.64
6.63
6.55
6.53
2.87
2.85
2.83
2.42
2.40
2.38
2.36
2.36
2.34
2.33
2.16
2.15
2.14
2.13
2.11
2.10
2.10
2.09
1.66
1.64
1.63
1.62
1.61
1.60
1.13
1.12
1.11
1.11
1.10
1.10
1.09
0.00
71% D
H/D
N
6
1.90
0.92
1.65
1.04
2.05
1.00
1.00
1.05
1.09
2.29
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)