Supporting Information

Metallic salt-catalyzed direct indium insertion into alkyl iodides and their applications in cross-coupling reactions

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General information

Commercially available alkyl iodides and aryl halides were used without further purification. Starting materials of 6-iodohexyl benzoate (**1h**)[1] was prepared according to reported methods. Analytical grade THF and DMA were used in all the reactions (without the need of precautions to exclude air and moisture unless otherwise noted) without purification. 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 spectrometers. Tetramethylsilane (TMS) served as internal standard for NMR analysis.

Experimental procedure

General procedure for the synthesis of alkyl indium reagent from alkyl halides and palladium-catalyzed cross-coupling reaction with aryl halide (Tables 2-4):

***The insertion step 1*:** Alkyl iodide (1 mmol), indium (172.2 mg, 1.5 mmol), indium trichloride (22.1 mg, 0.1 mmol) or lead bromide (36.7 mg, 0.1 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 12 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.

The cross-coupling step 2: To the above residue was added aryl halide (0.7 mmol), LiCl (84.8 mg, 2 mmol), $Pd(PPh_3)_4$ (57.8 mg, 0.05 mmol), and DMA (2 mL), and the reaction mixture was stirred at 100 ^{o}C for 24 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.

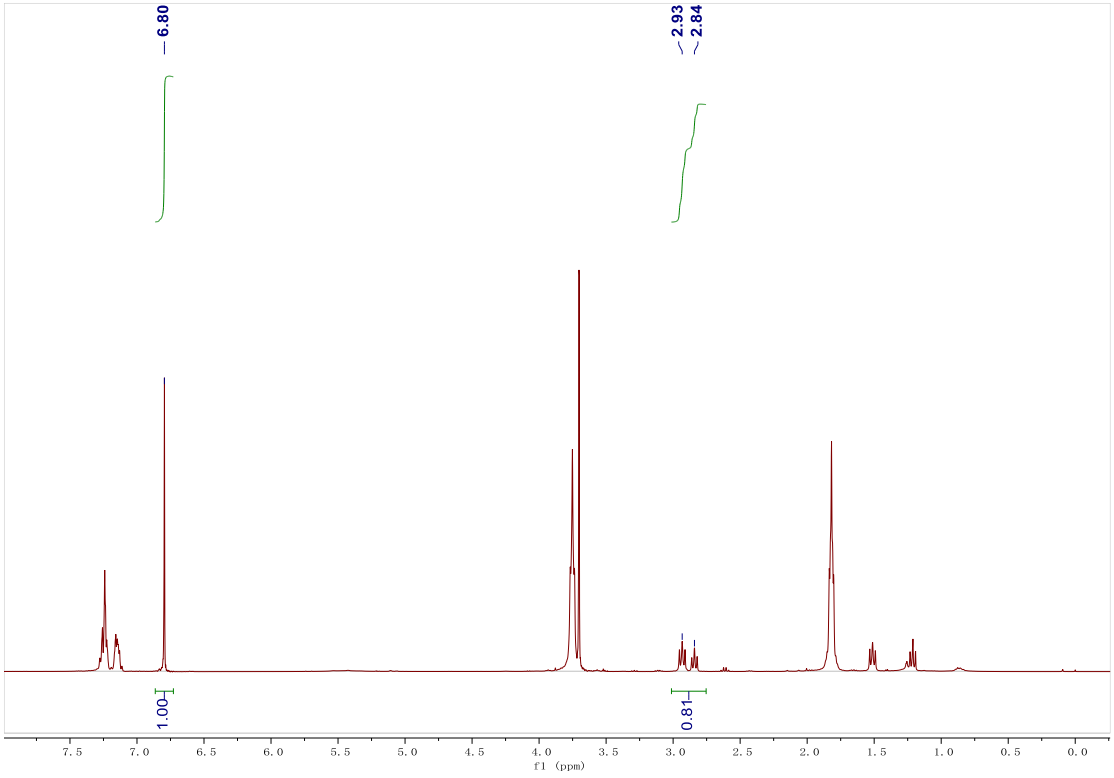
General procedure and method for the determination of NMR yield of the formed alkyl indium reagent:

After normal work-up by following the typical experimental procedure shown above (*the insertion step 1*), 1,4-dimethoxybenzene (0.5 mmol) was added to the obtained crude residue of the alkyl indium reagent followed by the addition of 2 mL $CDCl_3$. After complete dissolution of the residue and internal standard, 0.6 mL of the $CDCl_3$ solution was transferred to a clean NMR tube by pipette

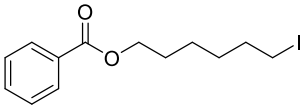
and subjected to NMR analysis. In the obtained ^{1}H NMR spectrum, integrations of the singlet peak at 6.8 ppm (belong to the four protons in aromatic ring of 1,4-dimethoxybenzene; suppose the integration value is Q) and the two triplet peaks between 2.8~3.0 ppm (belong to the two protons of the benzylic CH_2 of the formed two alkyl indium reagents A + B; suppose the integration value is S) could be performed. Based on the integrations, the NMR yield of the formed alkyl indium reagent could be calculated to be 81% yield based on the following calculation:

yield% = [(S/2 ÷ Q/4) x 0.5] ÷ 1 x 100% = [(0.81/2 ÷ 1/4) x 0.5] ÷ 1 x 100% = 81%.

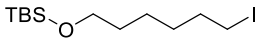
(Please note: 1 and 0.5 is the mmol of substrate and internal standard used, respectively; in addition, based on the integrations in the spectrum shown below, S = 0.81 and Q = 1)



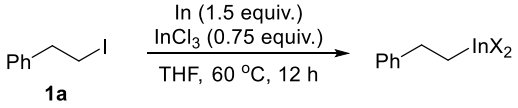
General procedure for the synthesis of alkyl iodide:



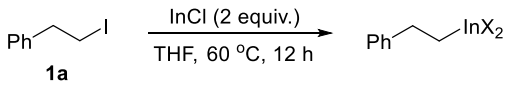
6-Iodohexyl benzoate (1h)[1]: The title compound was prepared from benzoyl chloride and 6-iodohexan-1-ol by a reported similar method (85% isolated yield), in which pyridine was used instead of Et_3N.



***tert*-Butyl((6-iodohexyl)oxy)dimethylsilane (1k):** To a solution of 6-chlorohexan-1-ol (8.2 g, 60.0 mmol) in CH_2Cl_2 (120 mL) was added imidazole (5.3 g, 78 mmol). After 5 min of stirring, *tert*-butyldimethylsilyl chloride (10.85 g, 72 mmol) was added over 5 min. The reaction mixture was stirred for 10 h before being diluted with CH_2Cl_2 (50 mL) and washed with saturated aqueous NH_4Cl (150 mL). The aqueous phase was extracted with CH_2Cl_2 (100 ml ×3). The combined organic phase was 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 (20:1) as eluent afforded the *tert*-butyl((6-chlorohexyl)oxy)dimethylsilane (12.3 g, 82%) as colorless liquid. To the solution of *tert*-butyl((6-chlorohexyl)oxy)dimethylsilane (6 g, 24.0 mmol) in acetone (120 mL) was added NaI (36 g, 240 mmol) and the solution was refluxed for 12 h. After cooling, the solvent was evaporated and diluted with EtOAc (150 mL), washed with brine (100 mL) and dried over anhydrous Na_2SO_4. After the solution was evaporated to dryness, the residue was purified by flash silica gel column chromatography using petroleum ether/ethyl acetate (20:1) as eluent to yield **1j** as colorless oil (6.5 g, 90%).



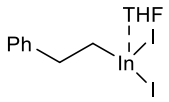
(2-Iodoethyl)benzene (232.1mg, 1 mmol), indium (172.2 mg, 1.5 mmol), indium trichloride (165.9 mg, 0.75 mmol), and analytical grade THF (2 mL) was added in a flask equipped with a septum and a magnetic stir bar. The reaction mixture was vigorously stirred at 60 °C for 12 hrs.



(2-Iodoethyl)benzene (232.1mg, 1 mmol), indium(I) chloride (300.5 mg, 2 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 °C for 12 hrs.

ESI-MS data

ESI-MS data of A1



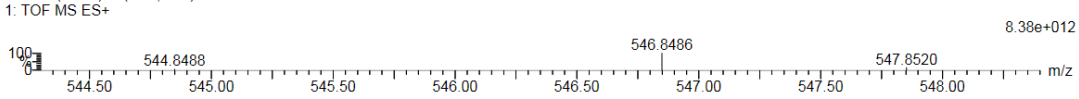
HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{12}H_{18}I_2InO$: 546.8486, found: 546.8486.

Elemental Composition Report

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

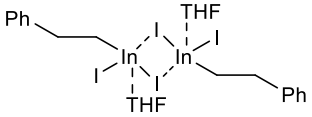
Monoisotopic Mass, Even Electron Ions
27 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 10-13 H: 15-20 O: 0-2 In: 0-2 I: 0-3

CBZ-2 (1.051) Is (1.00,1.00) C12H18I2InO
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
546.8486	546.8486	0.0	0.0	3.5	63.6	0.000	100.00	C12 H18 O In I2
	546.8492	-0.6	-1.1	1.5	86.0	22.456	0.00	C11 H18 O I3
	546.8480	0.6	1.1	5.5	128.8	65.181	0.00	C13 H18 O In2 I

ESI-MS data of A2



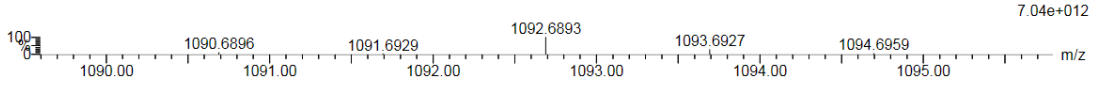
HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{24}H_{35}I_4In_2O_2$: 1092.6893, found: 1092.6893.

Elemental Composition Report

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

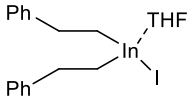
Monoisotopic Mass, Even Electron Ions
50 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 22-25 H: 34-36 O: 0-2 In: 0-2 I: 0-5

CBZ-2 (0.945) Is (1.00,1.00) C24H35I4In2O2
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
1092.6893	1092.6899	-0.6	-0.5	4.5	117.9	0.000	100.00	C23 H35 O2 In I5
	1092.6893	0.0	0.0	6.5	182.1	64.131	0.00	C24 H35 O2 In2 I4

ESI-MS data of B1



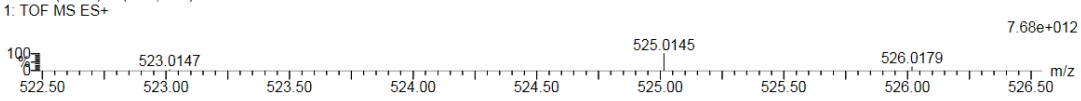
HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{20}H_{27}IInO$: 525.0145, found: 525.0145.

Elemental Composition Report

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

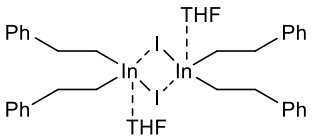
Monoisotopic Mass, Even Electron Ions
26 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 18-22 H: 25-30 O: 0-2 In: 0-2 I: 0-3

CBZ-2 (1.023) Is (1.00,1.00) C20H27IInO
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
525.0145	525.0145	0.0	0.0	7.5	64.4	0.000	100.00	C20 H27 O In I
	525.0151	-0.6	-1.1	5.5	87.0	22.602	0.00	C19 H27 O I2
	525.0139	0.6	1.1	9.5	129.4	65.050	0.00	C21 H27 O In2

ESI-MS data of B2



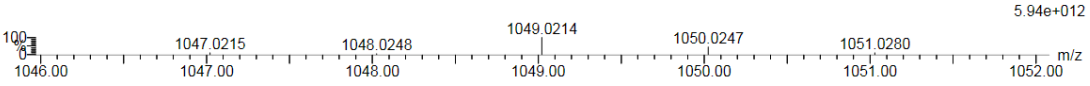
HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{40}H_{53}I_2In_2O_2$: 1049.0212, found: 1049.0214.

Elemental Composition Report

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

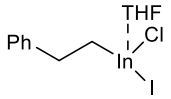
Monoisotopic Mass, Even Electron Ions
28 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 36-43 H: 50-55 O: 0-2 In: 0-2 I: 0-3

CBZ-2 (1.093) Is (1.00,1.00) C40H53I2In2O2
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
1049.0214	1049.0218	-0.4	-0.4	12.5	120.1	0.000	100.00	C39 H53 O2 In I3
	1049.0212	0.2	0.2	14.5	184.0	63.841	0.00	C40 H53 O2 In2 I2

ESI-MS data of C1



HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{12}H_{18}ClIInO$: 454.9130, found: 454.9129.

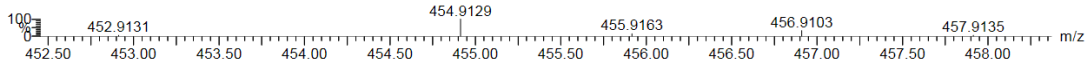
Elemental Composition Report

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
27 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 12-15 H: 15-19 O: 0-2 Cl: 1-3 In: 0-1 I: 0-1

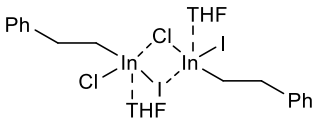
CBZ-1 (2.362) Is (1.00,1.00) C12H18ClIInO
1: TOF MS ES+

6.44e+012



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
454.9129	454.9130	-0.1	-0.2	3.5	119.6	n/a	n/a	C12 H18 O Cl In I

ESI-MS data of C2



HRMS (ESI, m/z): $[M+H]^+$, calcd for $C_{24}H_{35}Cl_2I_2In_2O_2$: 908.8181, found: 908.8180.

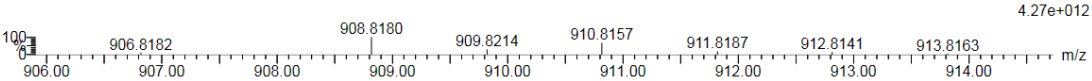
Elemental Composition Report

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
76 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 22-25 H: 34-36 O: 0-2 Cl: 0-2 In: 0-2 I: 0-2

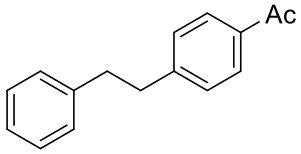
CBZ-2 (2.525) Is (1.00,1.00) C24H35Cl2I2In2O2
1: TOF MS ES+

4.27e+012

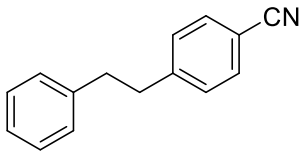


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
908.8180	908.8181	-0.1	-0.1	6.5	184.8	n/a	n/a	C24 H35 O2 Cl2 In2 I2

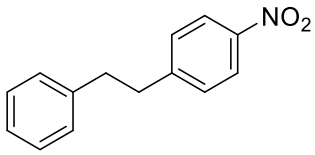
Characterization data of products



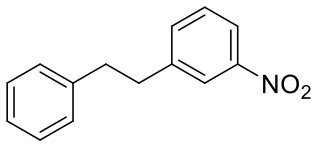
1-(4-Phenethylphenyl)ethan-1-one (3a): 135.0 mg. Yield = 86%. Yellow oil. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.78 (d, *J* = 7.8 Hz, 2H), 7.21-7.11 (m, 5H), 7.07 (d, *J* = 7.6 Hz, 2H), 2.92-2.82 (m, 4H), 2.49 (s, 3H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.8, 147.4, 141.0, 135.0, 128.7, 128.5, 128.4, 128.3, 126.0, 37.8, 37.4, 26.5 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{16}H_{17}O$: 225.1274, found: 225.1284. **FTIR (KBr, neat):** *ν* 1676 cm^{-1}.



4-Phenethylbenzonitrile (3b): 117.8 mg. Yield = 81%. Yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.47 (d, *J* = 8.3 Hz, 2H), 7.22-7.11 (m, 5H), 7.05 (d, *J* = 6.8 Hz, 2H), 2.93-2.88 (m, 2H), 2.86-2.82 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 147.2, 140.5, 132.1, 129.3, 128.4, 128.4, 126.2, 119.1, 109.7, 37.9, 37.2 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{15}H_{14}N$: 208.1121, found: 208.1126. **FTIR (KBr, neat):** ν 2226 cm^{-1}.

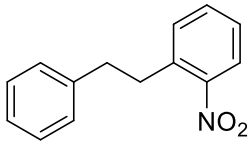


1-Nitro-4-phenethylbenzene (3c): 130.5 mg. Yield = 82%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.05 (d, *J* = 8.7 Hz, 2H), 7.23-7.19 (m, 4H), 7.16-7.12 (m, 1H), 7.06 (d, *J* = 7.0 Hz, 2H), 2.99-2.86 (m, 4H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 149.4, 146.4, 140.4, 129.3, 128.5, 128.4, 126.3, 123.6, 37.7, 37.2 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{14}NO_2$: 228.1019, found: 228.1024. **FTIR (KBr, neat):** ν 1514 cm^{-1}.

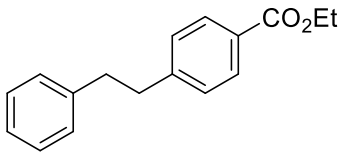


1-Nitro-3-phenethylbenzene (3d): 130.5 mg. Yield = 82%. Yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.95 (d, *J* = 6.8 Hz, 2H), 7.36-7.29 (m, 2H), 7.21-7.04 (m, 5H),

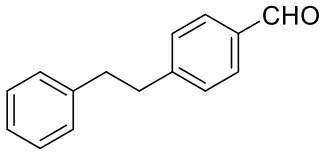
2.95-2.91 (m, 2H), 2.88-2.83 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 148.1, 143.5, 140.4, 134.8, 129.1, 128.4, 128.4, 126.2, 123.2, 121.1, 37.3, 37.3 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{14}NO_2$: 228.1019, found: 228.1024. **FTIR (KBr, neat):** ν 1526 cm^{-1}.



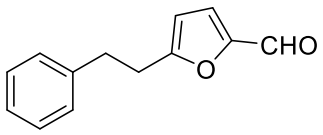
1-Nitro-2-phenethylbenzene (3e): 136.8 mg. Yield = 86%. Yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.86 (d, *J* = 8.2 Hz, 1H), 7.44-7.40 (m, 1H), 7.30-7.18 (m, 4H), 7.15-7.12 (m, 3H), 3.13-3.09 (m, 2H), 2.90-2.86 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 149.3, 140.9, 136.6, 132.9, 132.2, 128.5, 128.4, 127.2, 126.2, 124.8, 37.0, 35.5 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{14}NO_2$: 228.1019, found: 228.1024. **FTIR (KBr, neat):** ν 1526 cm^{-1}.



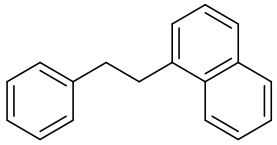
Ethyl 4-phenethylbenzoate (3f, 3g): 135.3 mg. 108.6 mg. Yield = 76%, 61%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.21-7.16 (m, 2H), 7.15-7.06 (m, 5H), 4.28 (q, *J* = 7.1 Hz, 2H), 2.92-2.82 (m, 4H), 1.30 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 166.6, 147.0, 141.1, 129.6, 128.5, 128.4, 128.3, 128.2, 126.0, 60.8, 37.8, 37.4, 14.3 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{19}O_2$: 255.1380, found: 255.1385. **FTIR (KBr, neat):** ν 1713 cm^{-1}.



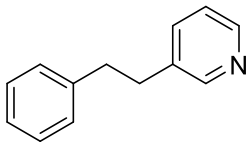
4-Phenethylbenzaldehyde (3h, 3i, 3j): 138.4 mg, 114.8 mg, 76.5 mg. Yield = 94%, 78%, 52%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 9.82 (s, 1H), 7.65 (d, *J* = 8.1 Hz, 2H), 7.19-7.06 (m, 5H), 7.03 (d, *J* = 7.0 Hz, 2H), 2.90-2.79 (m, 4H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 191.8, 148.9, 140.7, 134.4, 129.8, 129.0, 128.3, 128.3, 126.0, 37.9, 37.2 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{15}H_{15}O$: 211.1117, found: 211.1123. **FTIR (KBr, neat):** ν 1701 cm^{-1}.



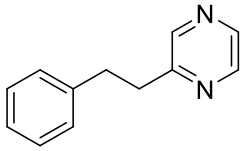
5-Phenethylfuran-2-carbaldehyde (3k): 76.3 mg. Yield = 54%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 9.43 (s, 1H), 7.21 (t, *J* = 7.3 Hz, 2H), 7.13-7.05 (m, 4H), 6.10 (d, *J* = 3.5 Hz, 1H), 2.94 (s, 4H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 176.9, 162.5, 151.7, 140.1, 128.4, 128.2, 126.3, 123.7, 109.1, 33.6, 30.1 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for 201.0910, found: 201.0916. **FTIR (KBr, neat):** ν 1677 cm^{-1}.



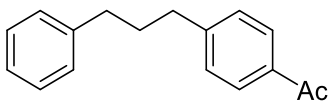
1-Phenethylnaphthalene (3l): 133.3 mg. Yield = 82%. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.01 (d, *J* = 8.3 Hz, 1H), 7.78 (d, *J* = 7.9 Hz, 1H), 7.64 (d, *J* = 8.1 Hz, 1H), 7.46-7.37 (m, 2H), 7.31-7.11 (m, 7H), 3.30-3.27 (m, 2H), 2.94-2.98 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 142.0, 137.8, 133.8, 131.7, 128.8, 128.4, 126.7, 126.0, 126.0, 125.8, 125.5, 125.4, 123.6, 37.1, 35.1 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{18}H_{17}$: 233.1325, found: 233.1330.



3-Phenethylpyridine (3m): 110.3 mg. Yield = 86%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.37-8.34 (m, 2H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.22-7.16 (m, 2H), 7.14-7.06 (m, 4H), 2.84 (s, 4H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 149.8, 147.3, 140.7, 136.8, 136.0, 128.4, 128.4, 126.1, 123.2, 37.4, 34.9 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{13}H_{14}N$: 184.1121, found: 184.1126.

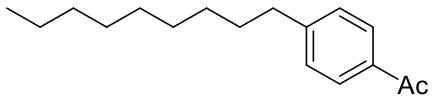


2-Phenethylpyrazine (3n): 114.8 mg. Yield = 89%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.52 (s, 1H), 8.40 (d, *J* = 2.5 Hz, 1H), 8.36 (m, 1H), 7.30-7.26 (m, 2H), 7.21-7.17 (m, 3H), 3.15-3.11 (m, 2H), 3.09-3.05 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 156.7, 144.7, 144.1, 142.3, 140.7, 128.5, 128.4, 126.2, 37.2, 35.4 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{12}H_{13}N_2$: 185.1073, found: 185.1079. **FTIR (KBr, neat):** ν 3027, 2927, 1430 cm^{-1}.

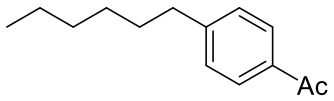


1-(4-(3-Phenylpropyl)phenyl)ethan-1-one (4b): 140.1 mg. Yield = 84%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.86 (d, *J* = 8.2 Hz, 2H), 7.28-7.22 (m, 4H), 7.18-7.14 (m, 3H), 2.68-2.60 (m, 4H), 2.54 (s, 3H), 1.98-1.90 (m, 2H) ppm. **^{13}C NMR (100 MHz,**

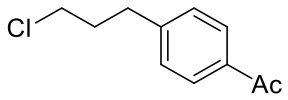
$CDCl_3$): δ 197.6, 147.9, 141.7, 134.8, 128.5, 128.3, 128.2, 128.2, 125.7, 35.2, 35.2, 32.4, 26.4 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{19}O$: 239.1430, found: 239.1430. **FTIR (KBr, neat):** *ν* 1682 cm^{-1}.



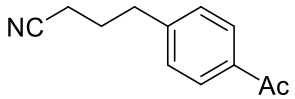
1-(4-Nonylphenyl)ethan-1-one (4c): 141.2 mg. Yield = 82%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.78 (d, *J* = 8.1 Hz, 2H), 7.15 (d, *J* = 7.8 Hz, 2H), 2.59-2.51 (m, 2H), 2.47 (s, 3H), 1.56-1.49 (m, 2H), 1.22-1.17 (m, 11H), 0.78 (t, *J* = 6.8 Hz, 4H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.6, 148.7, 134.7, 128.5, 128.3, 35.9, 31.8, 31.1, 29.4, 29.4, 29.2, 29.2, 26.4, 22.6, 14.0 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{27}O$: 247.2056, found: 247.2062. **FTIR (KBr, neat):** ν 1684 cm^{-1}.



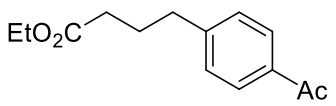
1-(4-Hexylphenyl)ethan-1-one (4d): 118.7 mg. Yield = 83%. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.90-7.87 (m, 2H), 7.28-7.25 (m, 2H), 2.66 (t, *J* = 8.0 Hz, 2H), 2.59 (s, 3H), 1.66-1.59 (m, 2H), 1.34-1.27 (m, 6H), 0.90-0.86 (m, 3H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.9, 148.8, 134.8, 128.6, 128.4, 36.0, 31.6, 31.1, 28.9, 26.5, 22.5, 14.1 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{21}O$: 205.1587, found: 205.1590. **FTIR (KBr, neat):** *ν* 1683 cm^{-1}.



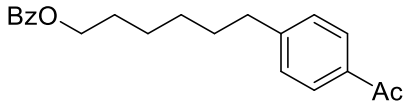
1-(4-(3-Chloropropyl)phenyl)ethan-1-one (4e): 85.4 mg. Yield = 62%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.23 (d, *J* = 8.3 Hz, 2H), 3.46 (t, *J* = 6.4 Hz, 2H), 2.80-2.76 (m, 2H), 2.52 (s, 3H), 2.07-2.00 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.8, 146.5, 135.3, 128.8, 128.6, 44.0, 33.5, 32.7, 26.6 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{11}H_{14}ClO$: 197.0728, found: 197.0733. **FTIR (KBr, neat):** ν 1683 cm^{-1}.



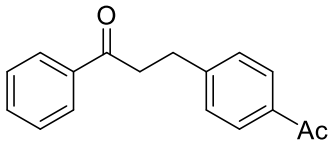
4-(4-Acetylphenyl)butanenitrile (4f): 111.4 mg. Yield = 85%. Brown solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.92 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 7.8 Hz, 2H), 2.85 (t, *J* = 7.5 Hz, 2H), 2.59 (s, 3H), 2.36 (t, *J* = 7.0 Hz, 2H), 2.06-1.98 (m, 2H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.6, 145.3, 135.5, 128.7, 128.6, 119.1, 34.2, 26.5, 26.4, 16.3 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{12}H_{14}NO$: 188.1070, found: 188.1078. **FTIR (KBr, neat):** *ν* 2246, 1682 cm^{-1}.



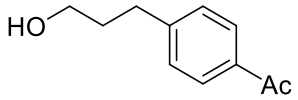
Ethyl 4-(4-acetylphenyl)butanoate (4g): 134.5 mg. Yield = 82%. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.89 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.2 Hz, 2H), 4.13 (q, *J* = 7.1 Hz, 2H), 2.74-2.70 (m, 2H), 2.59 (s, 3H), 2.33 (t, *J* = 7.4 Hz, 2H), 2.01-1.94 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.8, 173.2, 147.2, 135.2, 128.7, 128.5, 60.3, 33.5, 30.1, 26.5, 26.1, 14.2 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{19}O_3$: 235.1329, found: 235.1328. **FTIR (KBr, neat):** *v* 1732, 1683 cm^{-1}.



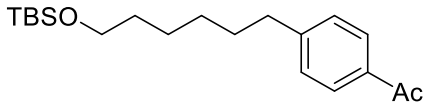
6-(4-Acetylphenyl)hexyl benzoate (4h): 193.0 mg. Yield = 85%. White solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 8.04 (d, *J* = 7.1 Hz, 2H), 7.87 (d, *J* = 8.3 Hz, 2H), 7.57-7.54 (m, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 4.31 (t, *J* = 6.6 Hz, 2H), 2.68 (t, *J* = 7.7 Hz, 2H), 2.58 (s, 3H), 1.80-1.73 (m, 2H), 1.71-1.63 (m, 2H), 1.52-1.38 (m, 4H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.8, 166.6, 148.4, 134.9, 132.8, 130.4, 129.5, 128.5, 128.5, 128.3, 64.9, 35.8, 30.9, 28.8, 28.6, 26.5, 25.9 ppm. **HRMS (ESI, m/z)**: $[M+H]^+$, calcd. for $C_{21}H_{25}O_3$: 325.1798, found: 325.1796. **FTIR (KBr, neat):** *v* 1710, 1675 cm^{-1}.



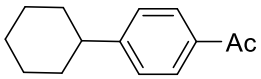
3-(4-Acetylphenyl)-1-phenylpropan-1-one (4i): 111.3 mg. Yield = 63%. Yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.91-7.80 (m, 4H), 7.52-7.47 (m, 1H), 7.39 (tt, *J* = 6.9, 2.7 Hz, 2H), 7.28 (dd, *J* = 8.1, 3.0 Hz, 2H), 3.32-3.20 (m, 2H), 3.07 (tt, *J* = 7.3, 3.3 Hz, 2H), 2.51 (s, 3H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 198.6, 197.8, 147.1, 136.6, 135.2, 133.2, 128.7, 128.7, 128.0, 39.7, 29.9, 26.6 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{17}O_2$: 253.1223, found: 253.1225. **FTIR (KBr, neat):** *v* 1680, 1602 cm^{-1}.



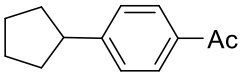
1-(4-(3-Hydroxypropyl)phenyl)ethan-1-one (4j): 78.6 mg. Yield = 63%. Colorless oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.90-7.87 (m, 2H), 7.31-7.29 (m, 2H), 3.69 (t, *J* = 6.4 Hz, 2H), 2.80-2.76 (m, 2H), 2.59 (s, 3H), 1.95-1.88 (m, 2H), 1.78 (s, 1H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 198.0, 147.8, 135.0, 128.6, 128.5, 61.9, 33.8, 32.0, 26.5 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{11}H_{15}O_2$: 179.1067, found: 179.1069. **FTIR (KBr, neat):** *v* 3438, 1679 cm^{-1}.



1-(4-(6-((*tert*-Butyldimethylsilyl)oxy)hexyl)phenyl)ethan-1-one (4k): 196.7 mg. Yield = 84%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.88 (d, *J* = 7.9 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 3.59 (t, *J* = 6.5 Hz, 2H), 2.68-2.64 (m, 2H), 2.59 (s, 3H), 1.68-1.60 (m, 2H), 1.52-1.48 (m, 2H), 1.37-1.33 (m, 4H), 0.89 (s, 9H), 0.04 (s, 6H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.9, 148.7, 134.9, 128.6, 128.5, 63.2, 35.9, 32.7, 31.1, 29.0, 26.6, 26.0, 25.6, 18.4, -5.3 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{20}H_{35}O_2Si$: 335.2401, found: 335.2401. **FTIR (KBr, neat):** *ν* 1685 cm^{-1}.



1-(4-Cyclohexylphenyl)ethan-1-one (4l): 126.0 mg. Yield = 89%. Yellow solid. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.81 (d, *J* = 8.3 Hz, 2H), 7.21 (d, *J* = 8.3 Hz, 2H), 2.49 (s, 3H), 1.79-1.66 (m, 5H), 1.40-1.11 (m, 6H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.8, 153.7, 134.9, 128.5, 127.0, 44.6, 34.0, 26.6, 26.5, 25.9 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{14}H_{19}O$: 203.1430, found: 203.1436. **FTIR (KBr, neat):** ν 1671 cm^{-1}.

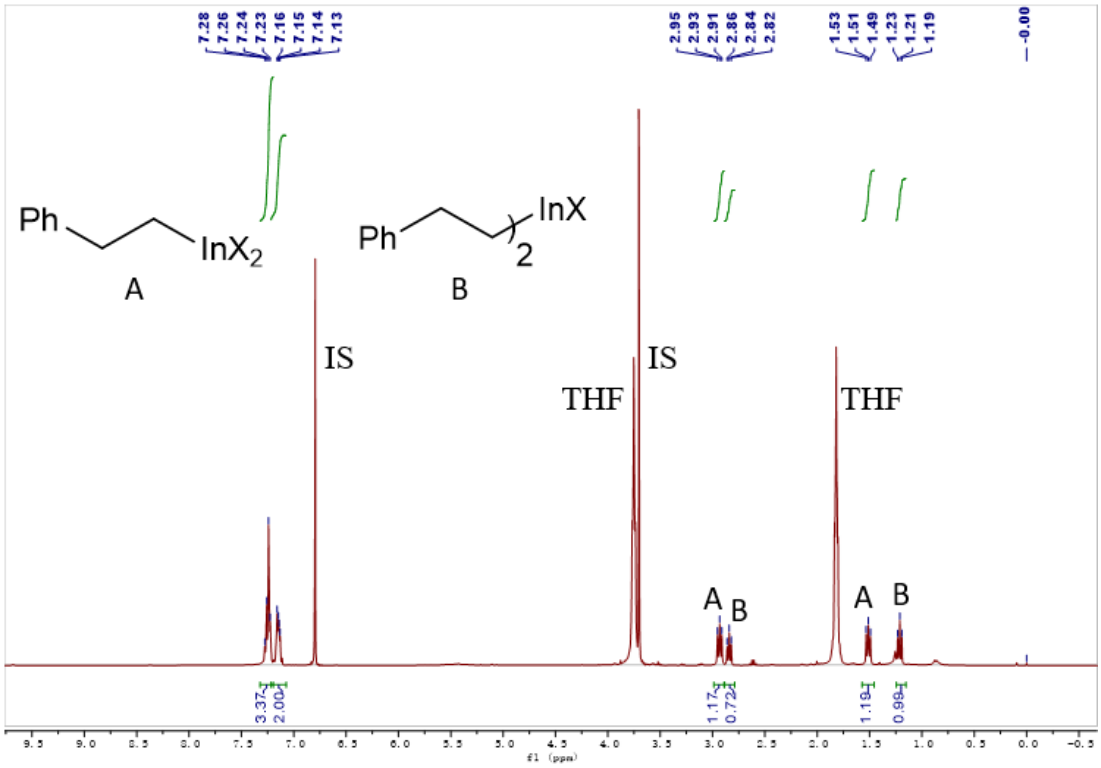


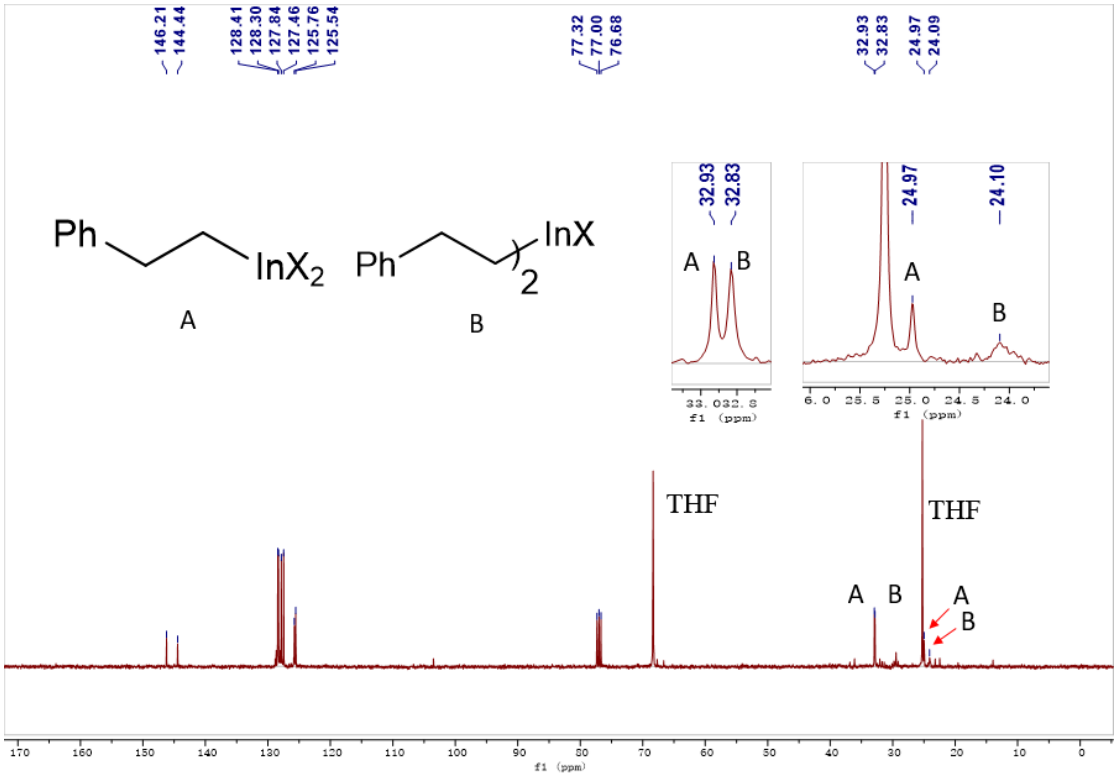
1-(4-Cyclopentylphenyl)ethan-1-one (4m): 98.8 mg. Yield = 75%. Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$):** δ 7.80 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 3.00-2.91 (m, 1H), 2.49 (s, 3H), 2.04-1.96 (m, 2H), 1.76-1.46 (m, 6H) ppm. **^{13}C NMR (100 MHz, $CDCl_3$):** δ 197.8, 152.4, 134.8, 128.4, 127.2, 45.9, 34.4, 26.5, 25.5 ppm. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{13}H_{17}O$: 189.1274, found: 189.1279. **FTIR (KBr, neat):** ν 1682 cm^{-1}.

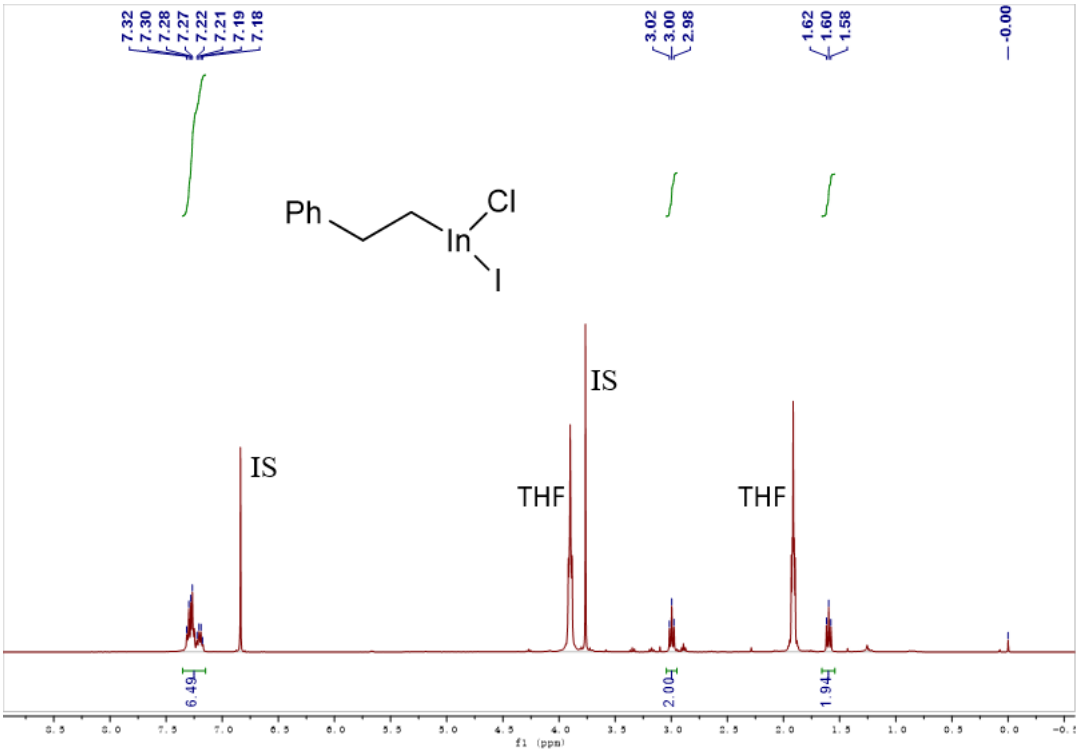
References

1. Thompson, A. M.; Delaney, A. M.; Hamby, J. M.; Schroeder, M. C.; Spoon, T. A.; Crean, S. M.; Showalter, H. D. H.; Denny, W. A. *J. Med. Chem*. **2005**, *48*, 4628-4653.

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



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1.60
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In
Cl
I
IS
IS
THF
THF
6.49
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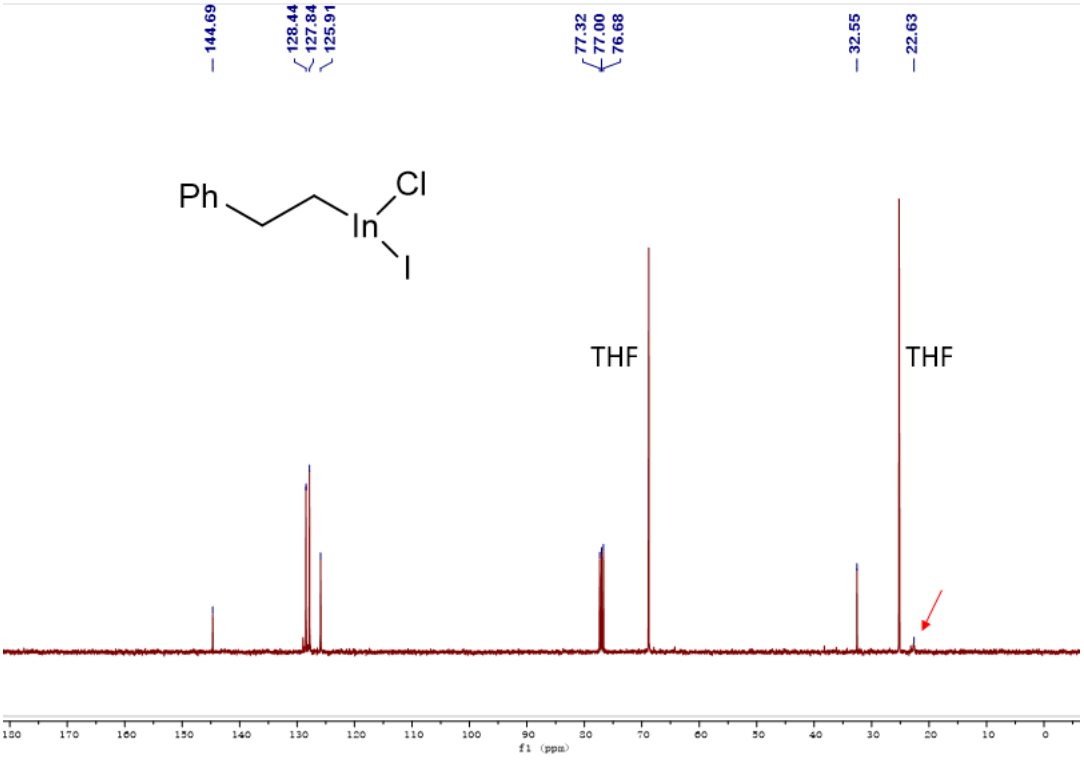
1.94
f1 (ppm)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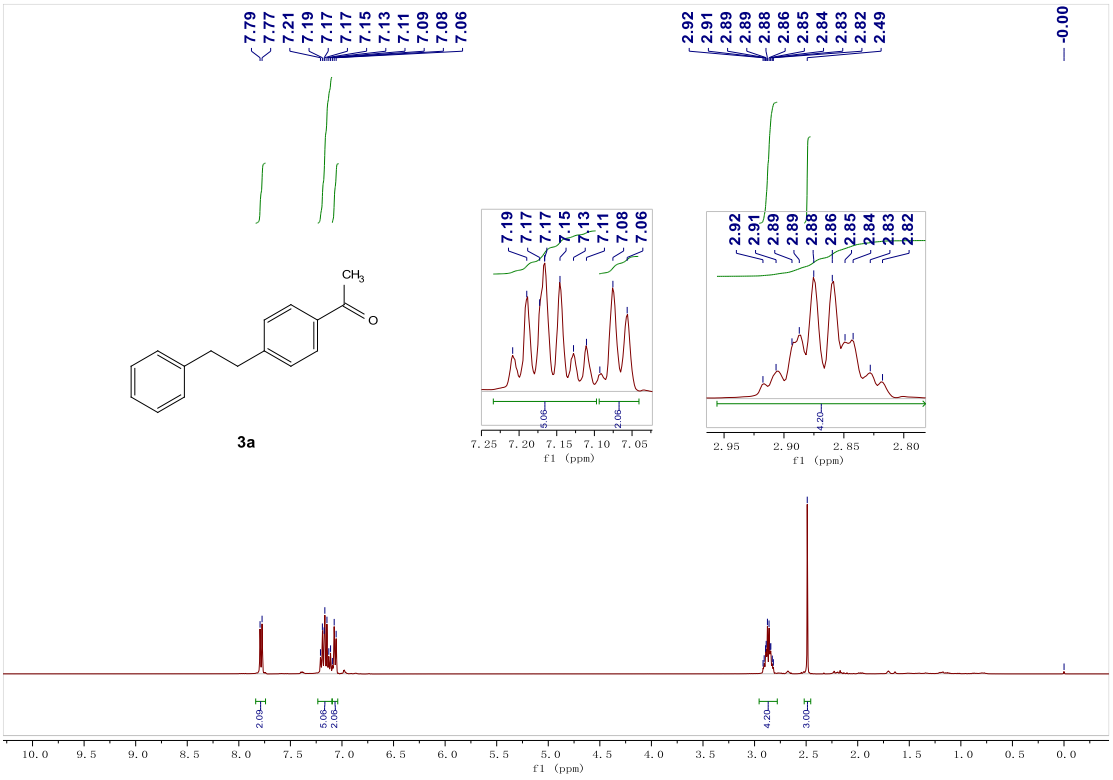
Ph
In
Cl
I
THF
THF
f1 (ppm)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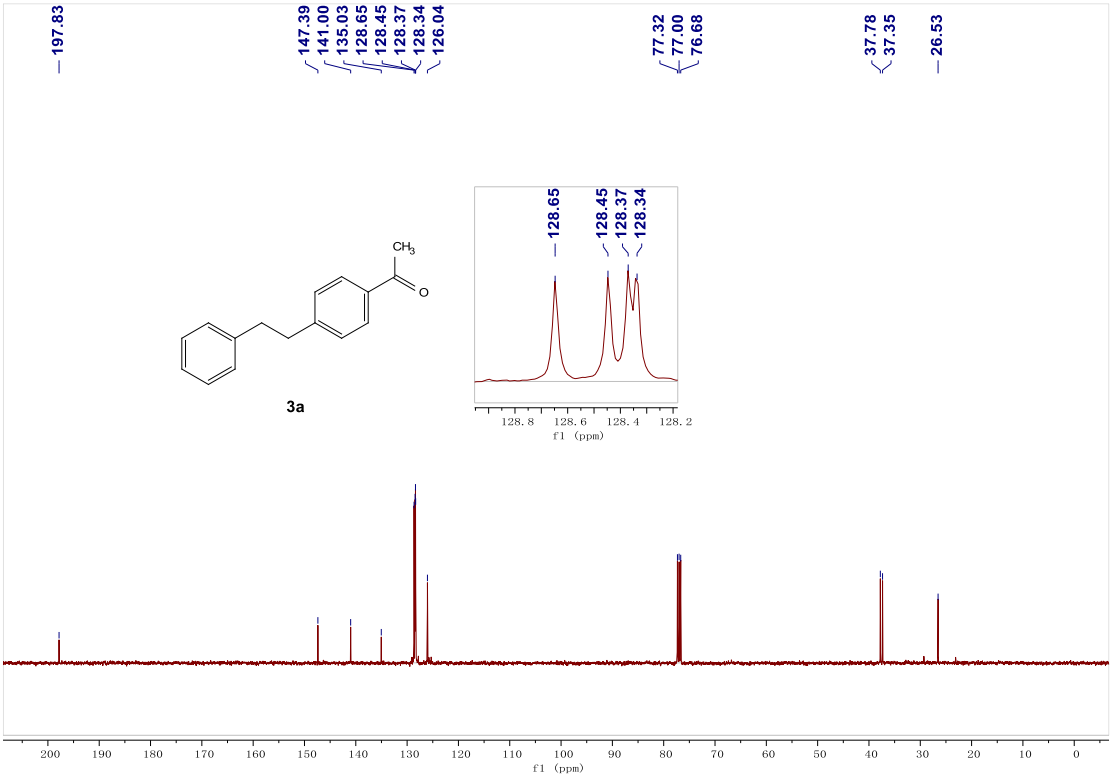
f1 (ppm)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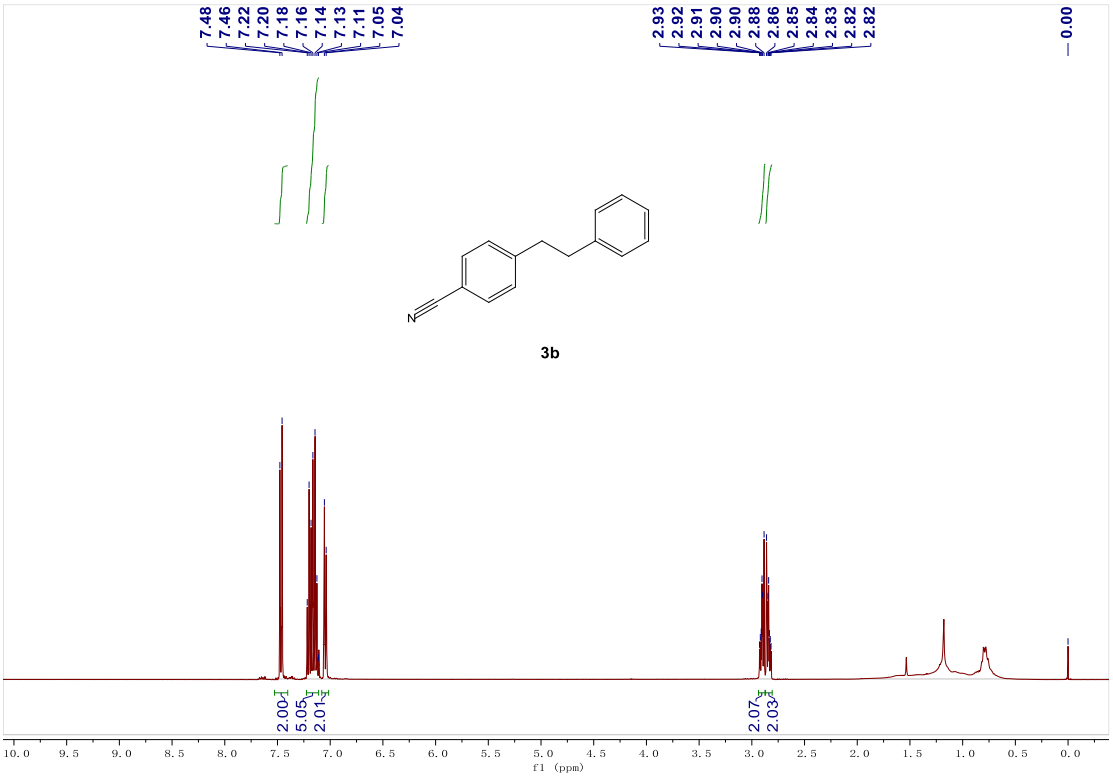
O
3a
f1 (ppm)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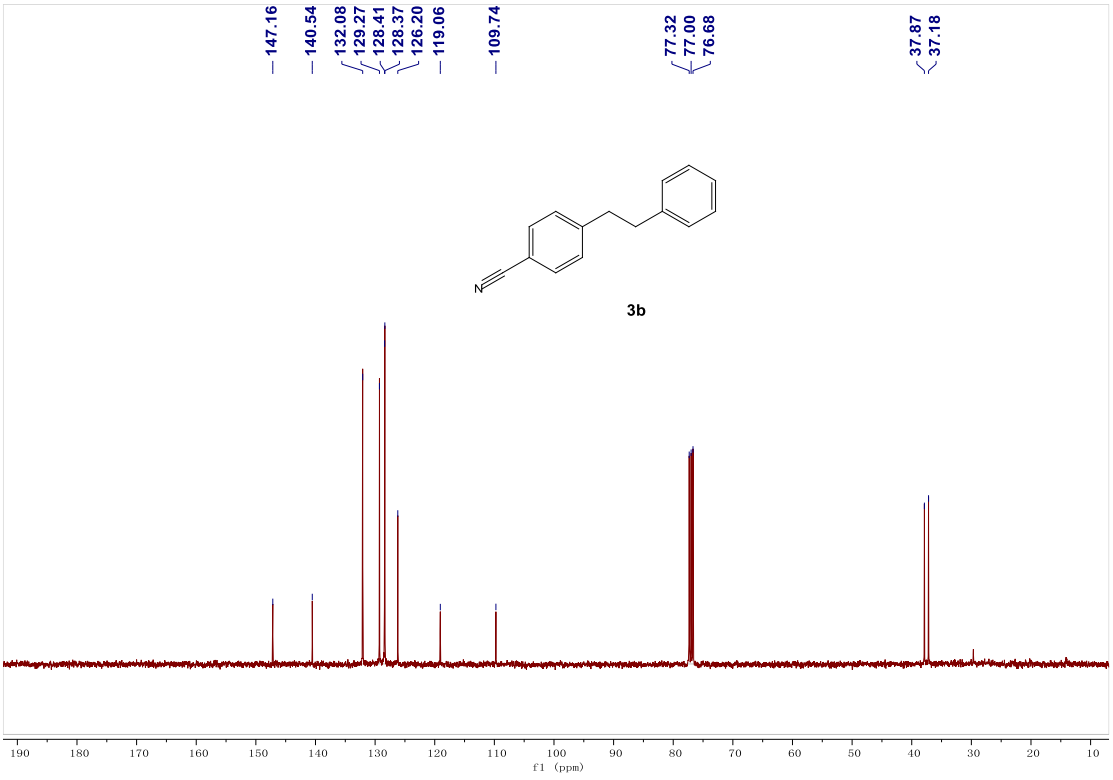
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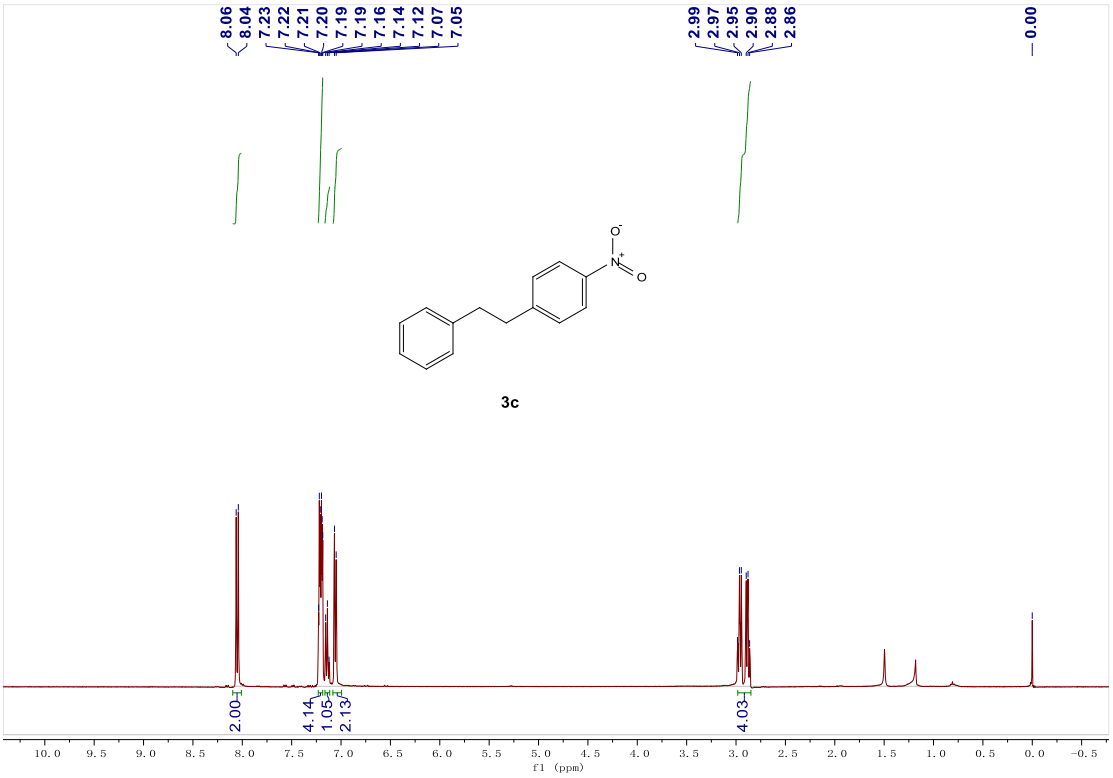
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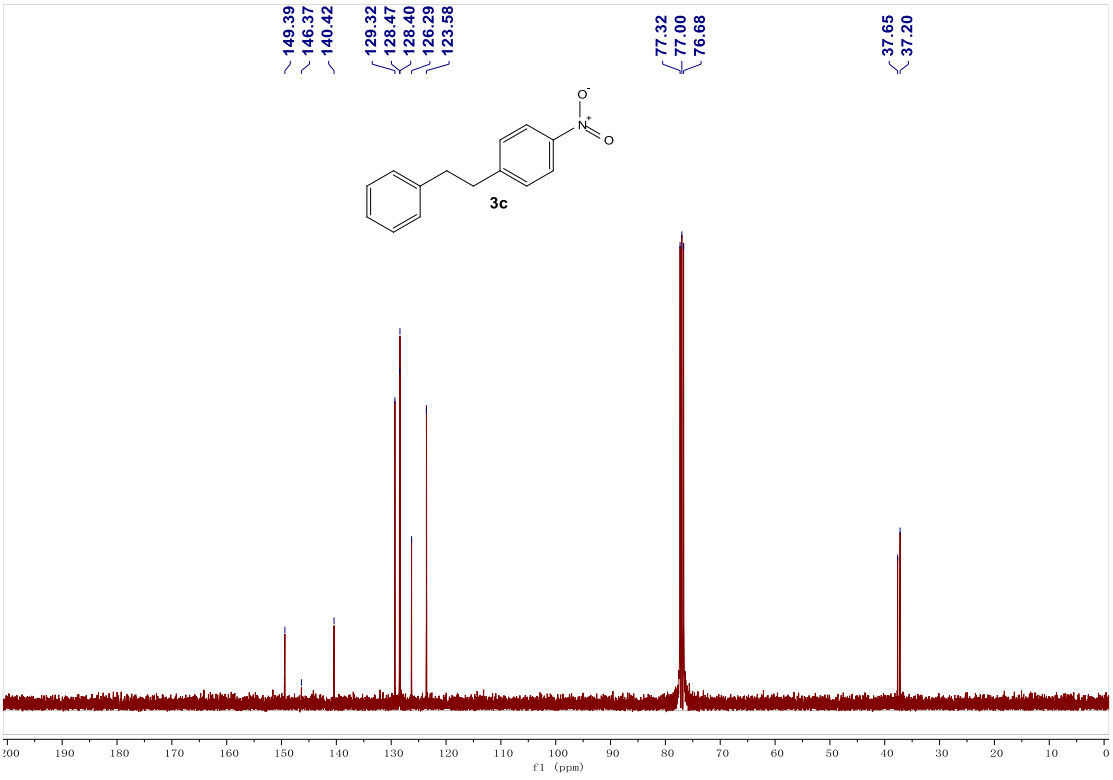
f1 (ppm)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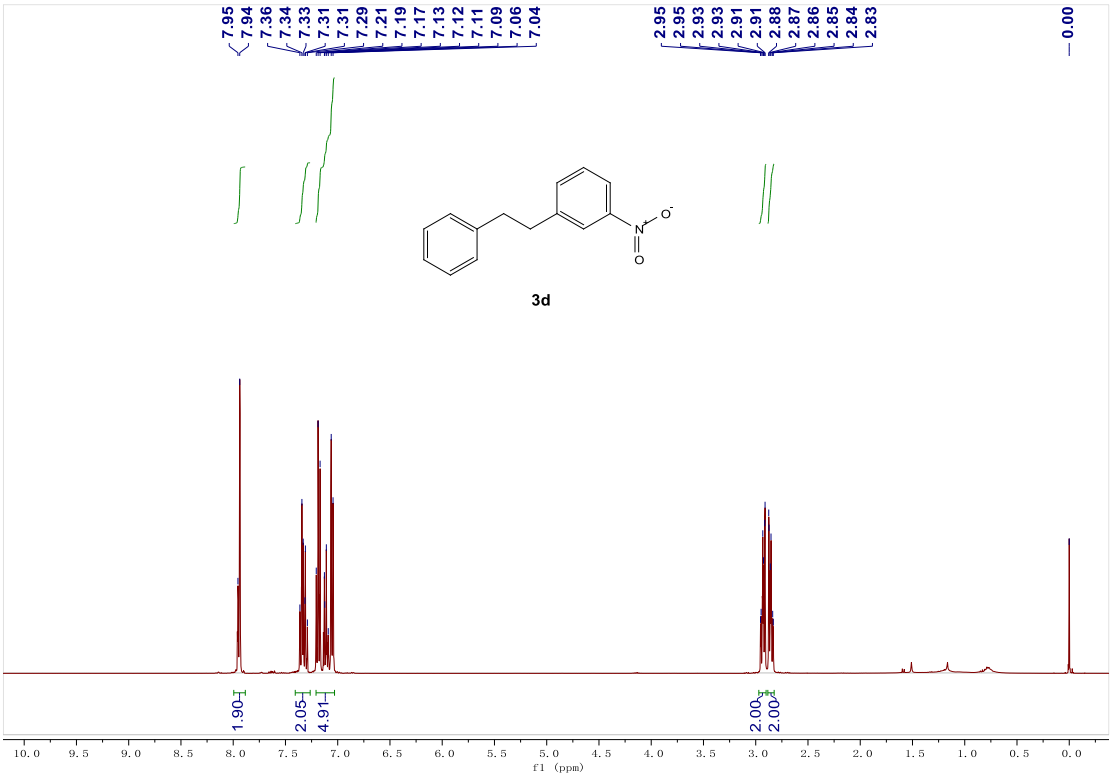
3c
f1 (ppm)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2.95
2.95
2.93
2.93
2.91
2.91
2.88
2.87
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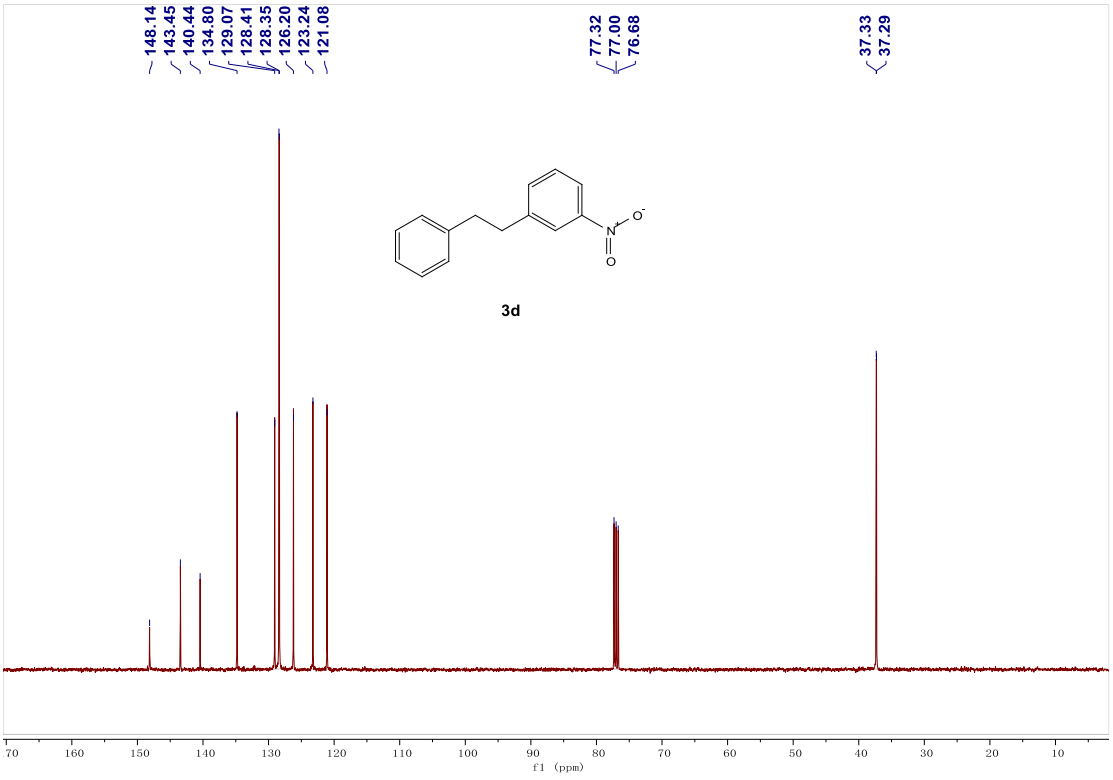
2.00
f1 (ppm)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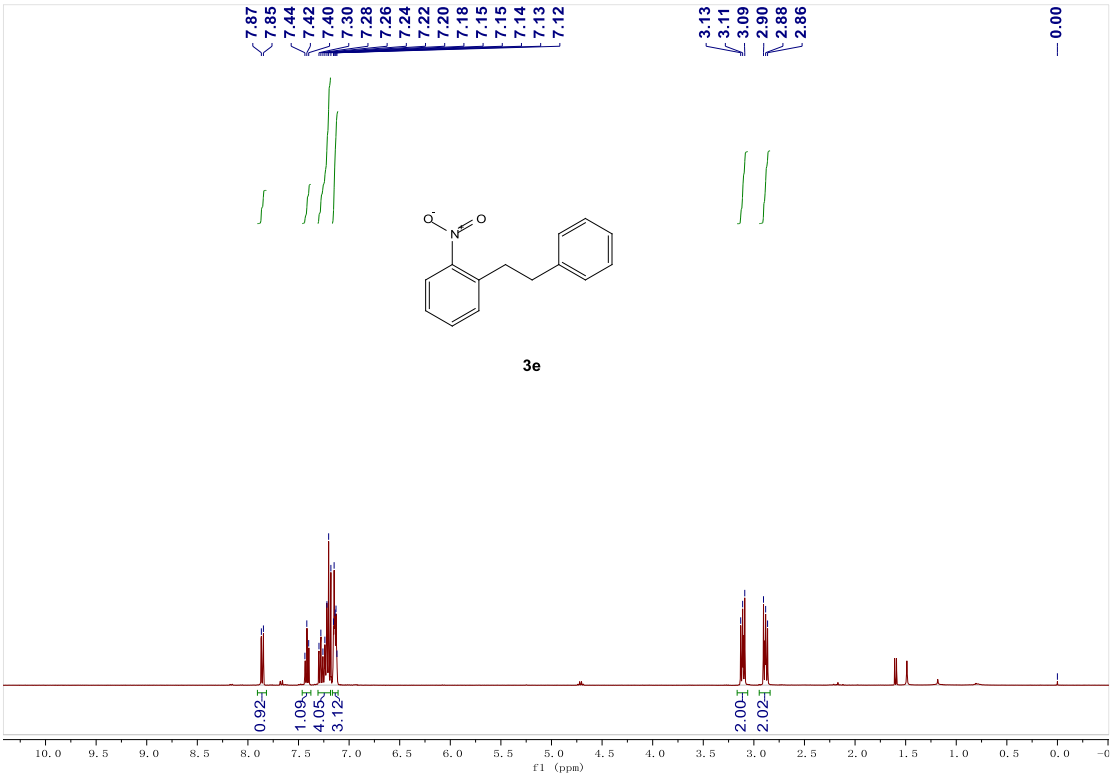
3d
f1 (ppm)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7.15
7.14
7.13
7.12
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3.11
3.09
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1.09
4.05
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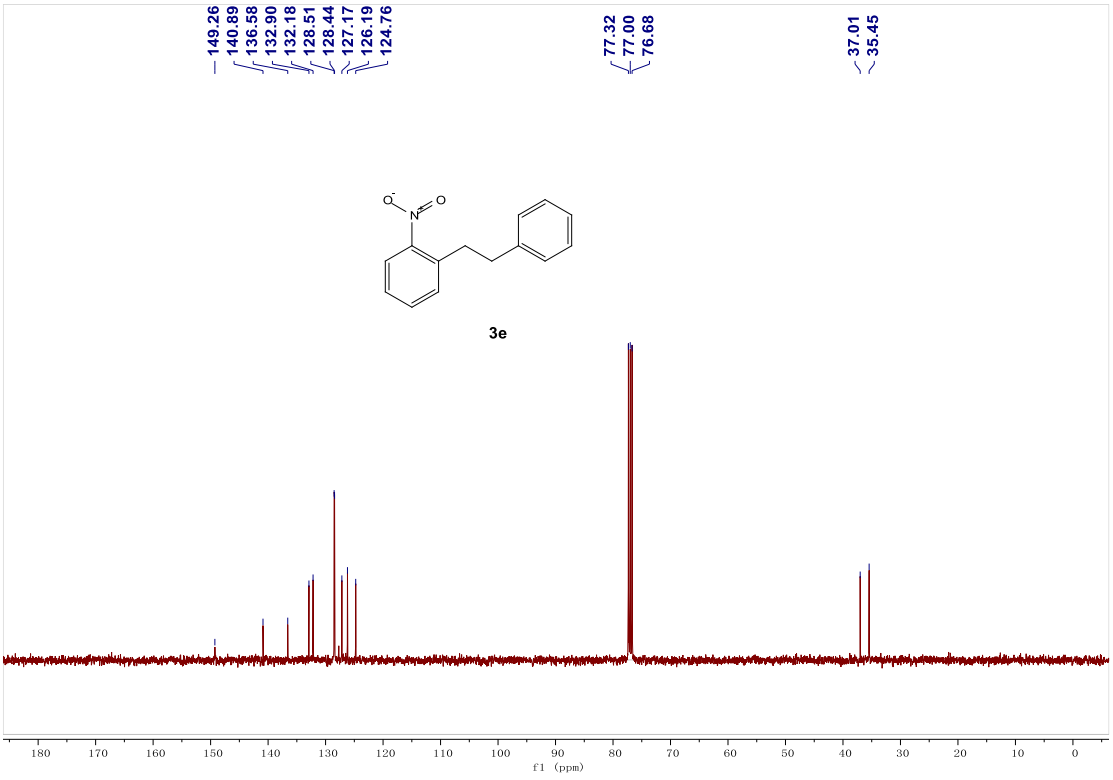
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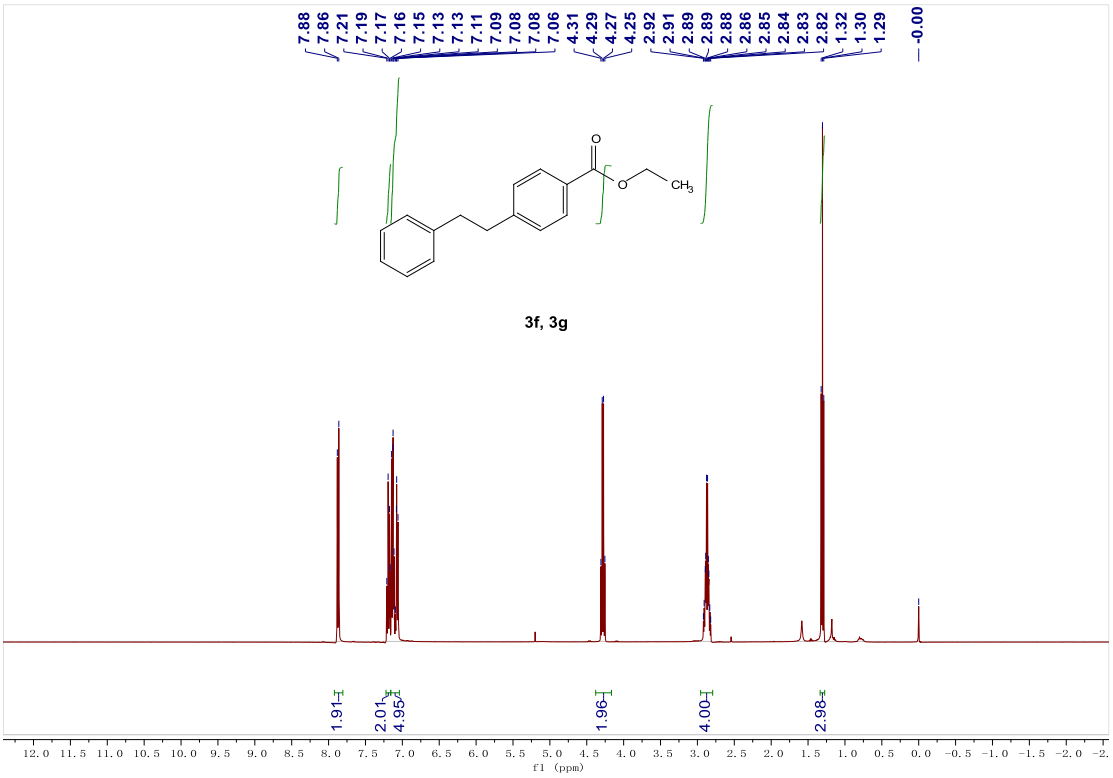
f1 (ppm)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7.06
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4.29
4.27
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2.89
2.88
2.86
2.85
2.84
2.83
2.82
1.32
1.30
1.29
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O
CH3
3f, 3g
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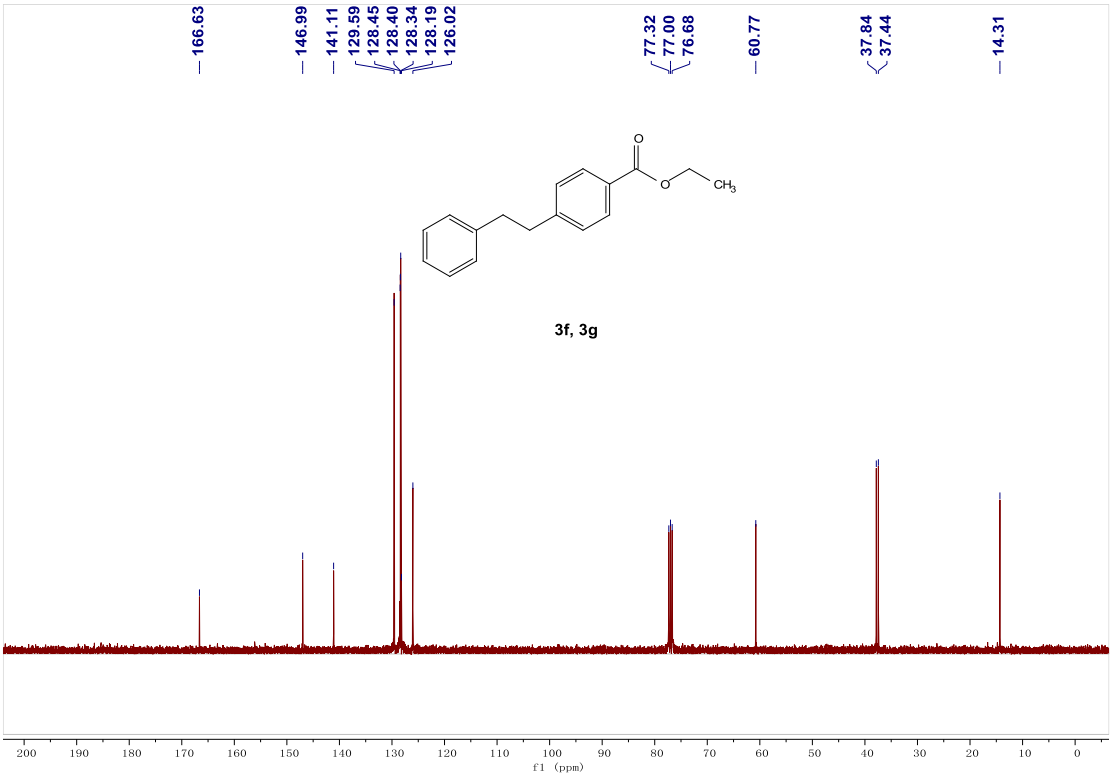
2.01
4.95
1.96
4.00
2.98
f1 (ppm)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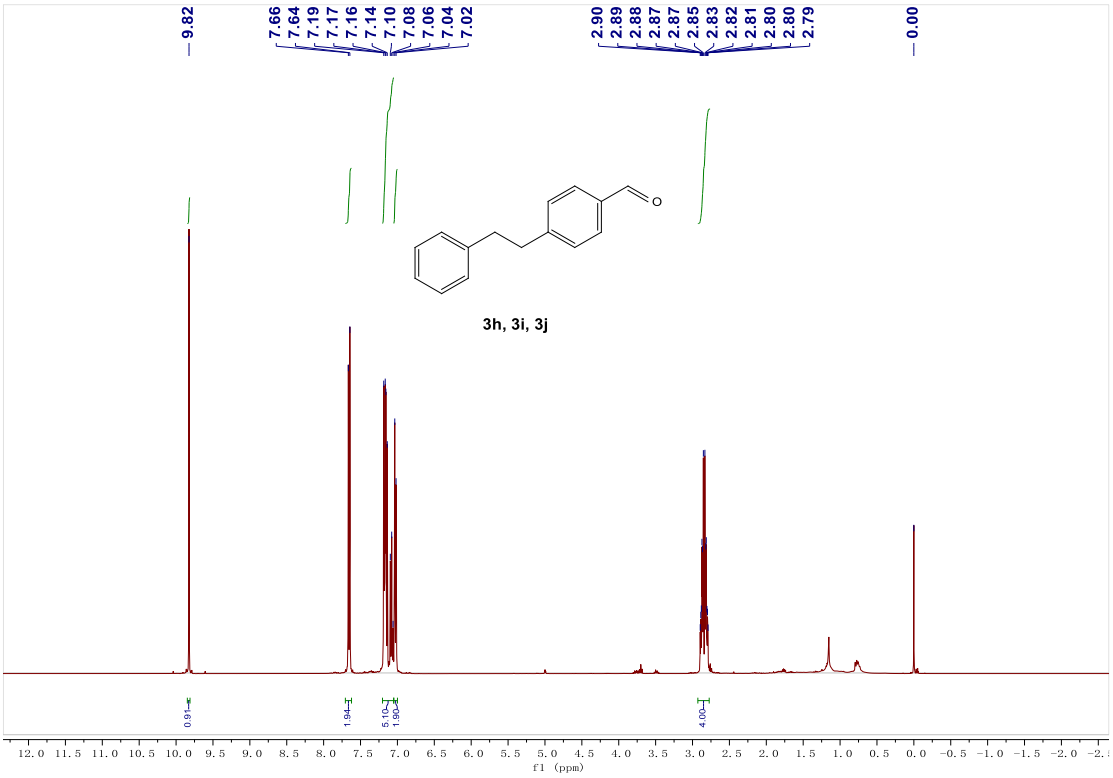
O
CH3
3f, 3g
f1 (ppm)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2.87
2.85
2.83
2.82
2.81
2.80
2.80
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O
3h, 3i, 3j
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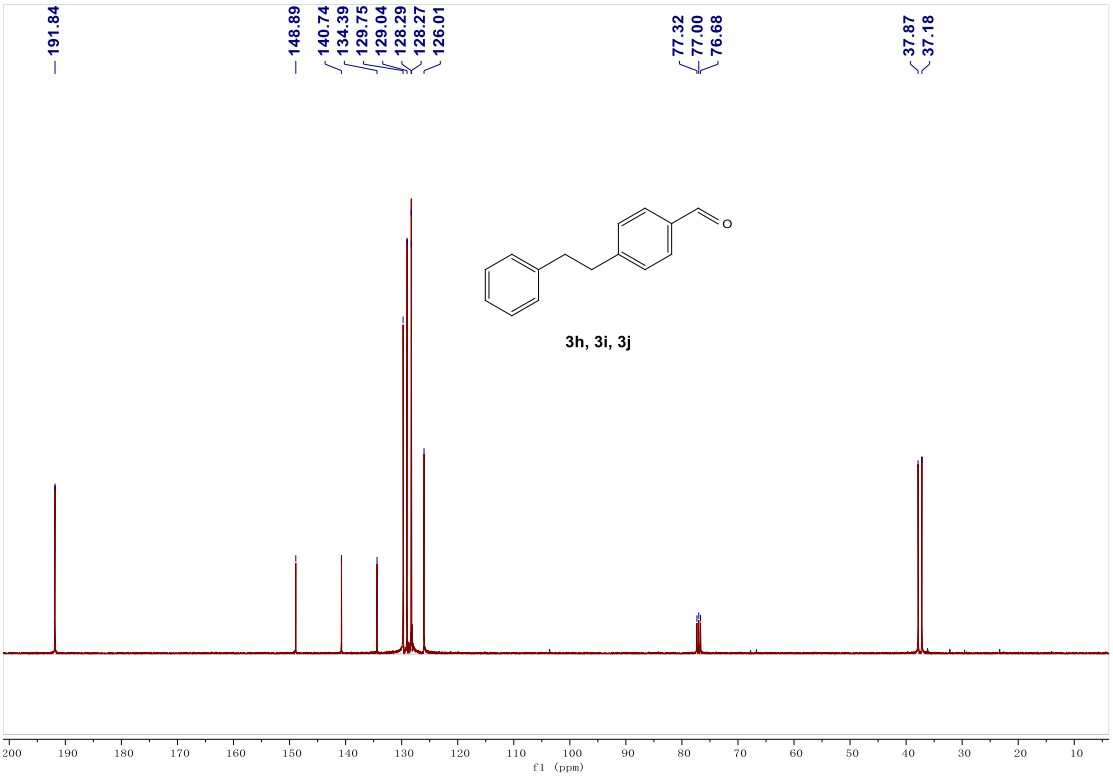
1.94
5.10
1.90
4.00
f1 (ppm)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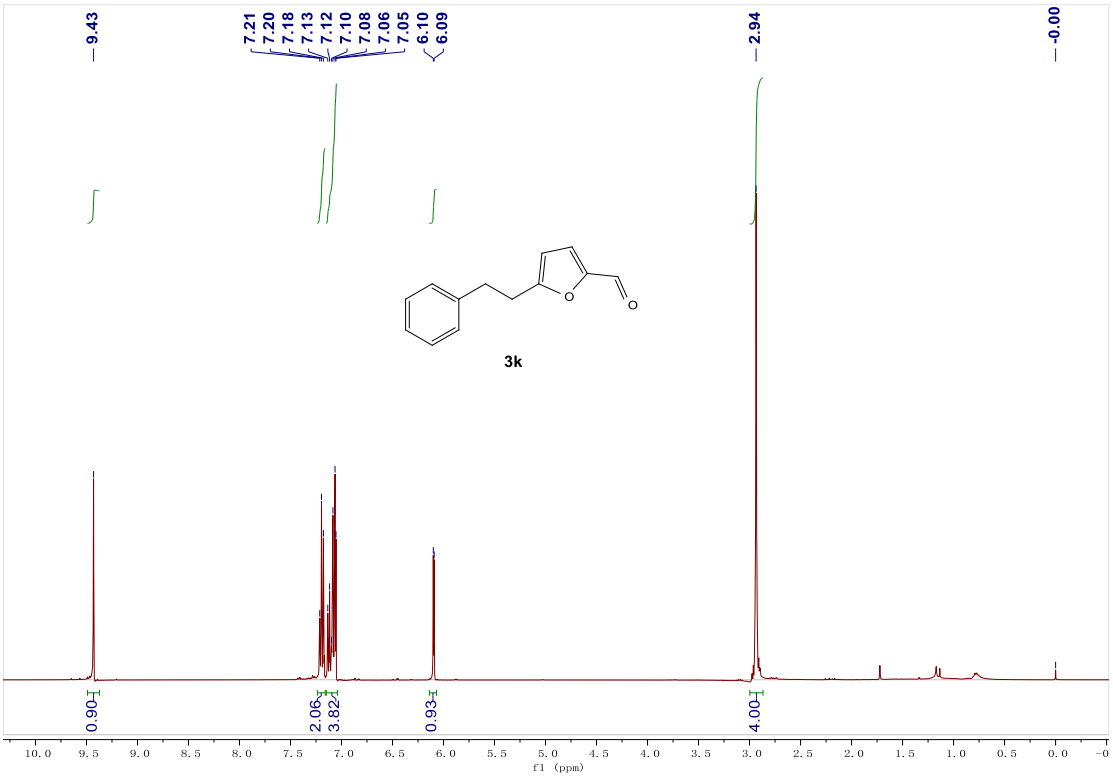
3h, 3i, 3j
f1 (ppm)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7.05
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O
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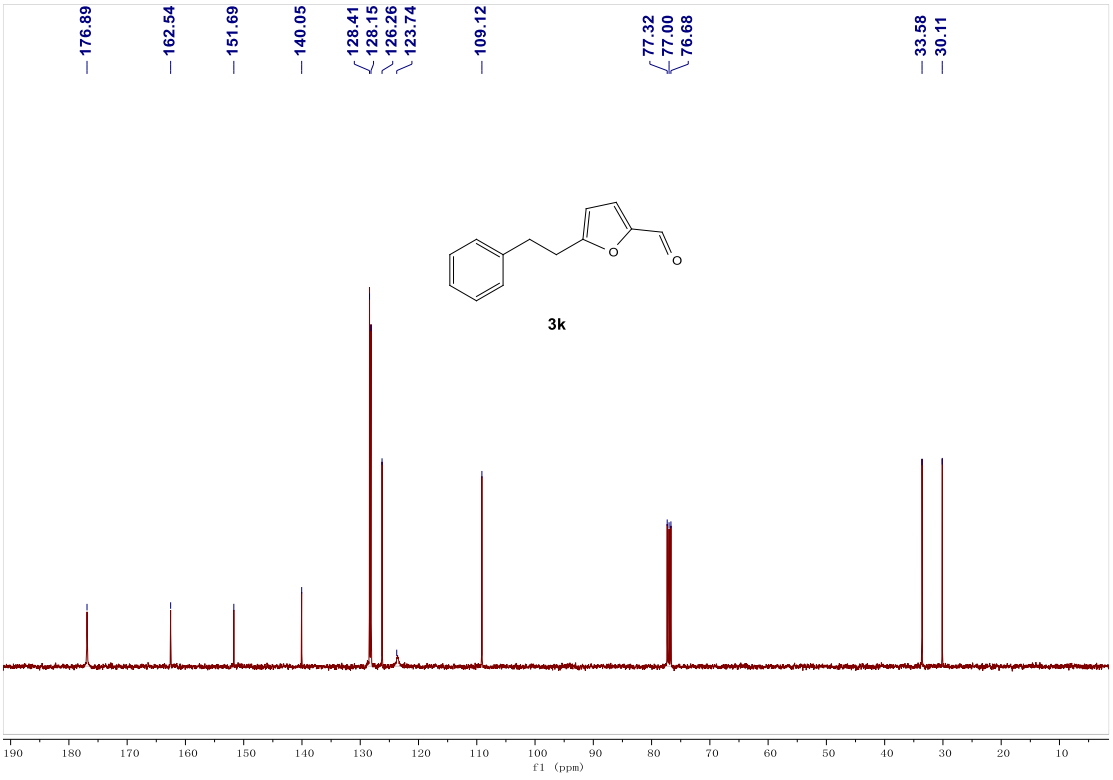
4.00
f1 (ppm)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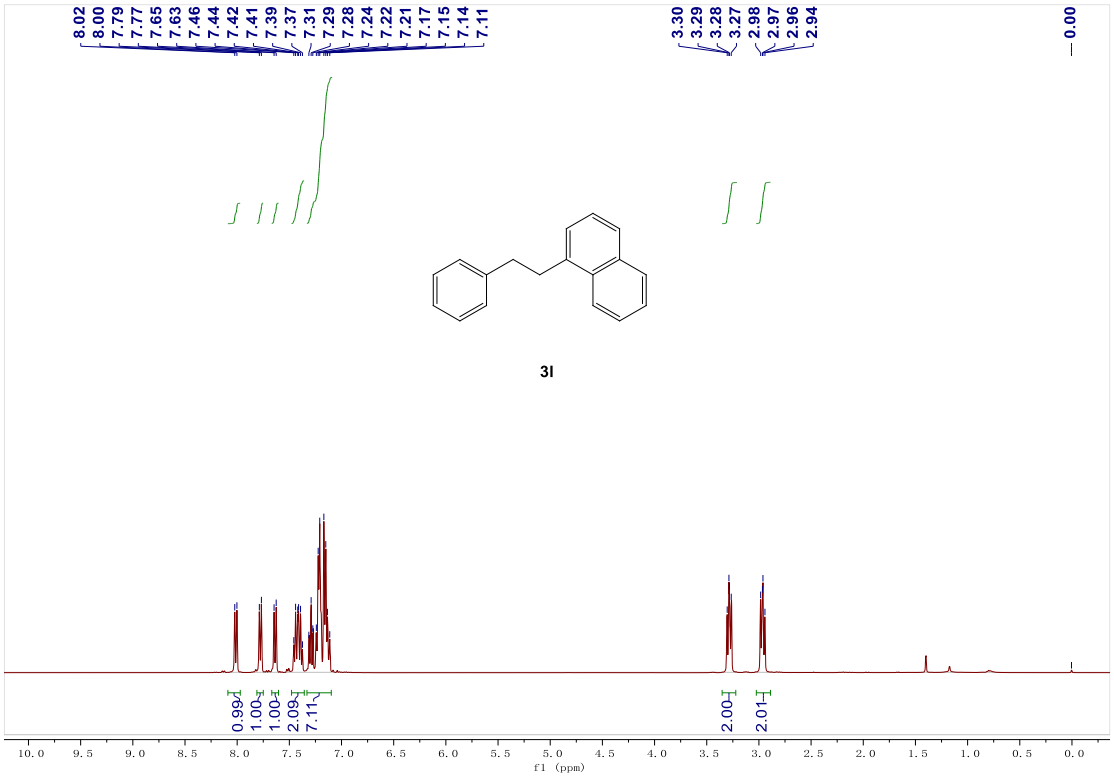
O
3k
f1 (ppm)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7.28
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7.22
7.21
7.17
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7.14
7.11
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3.29
3.28
3.27
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2.97
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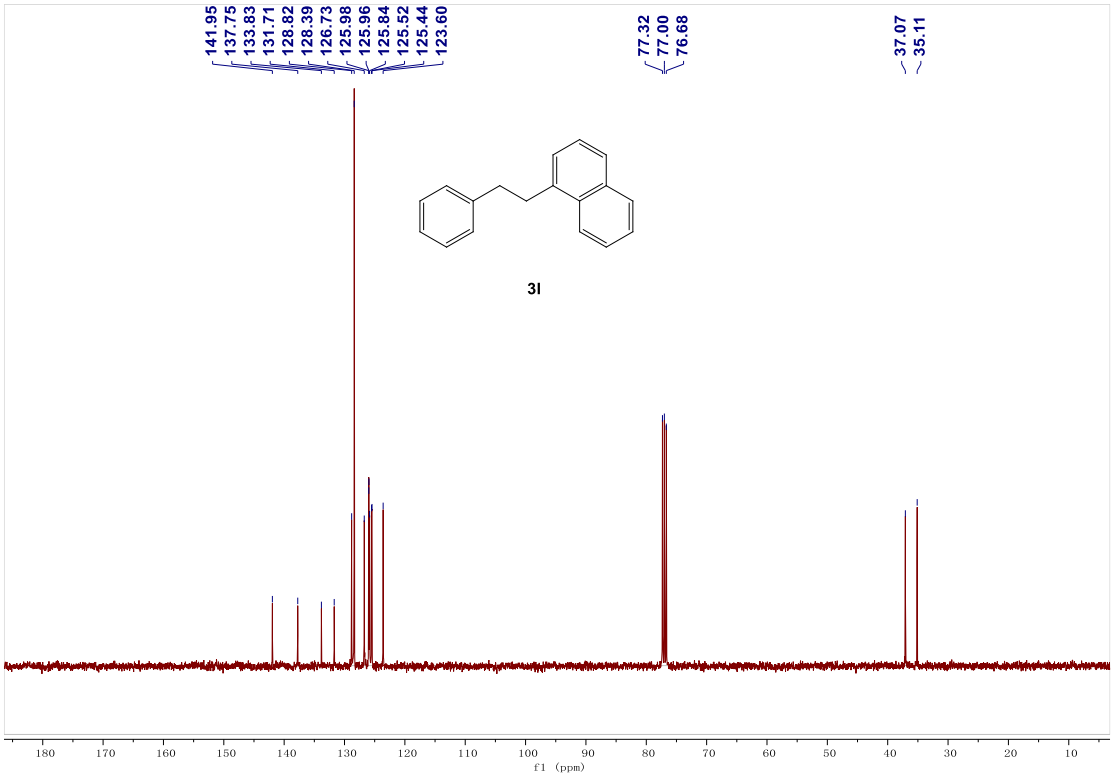
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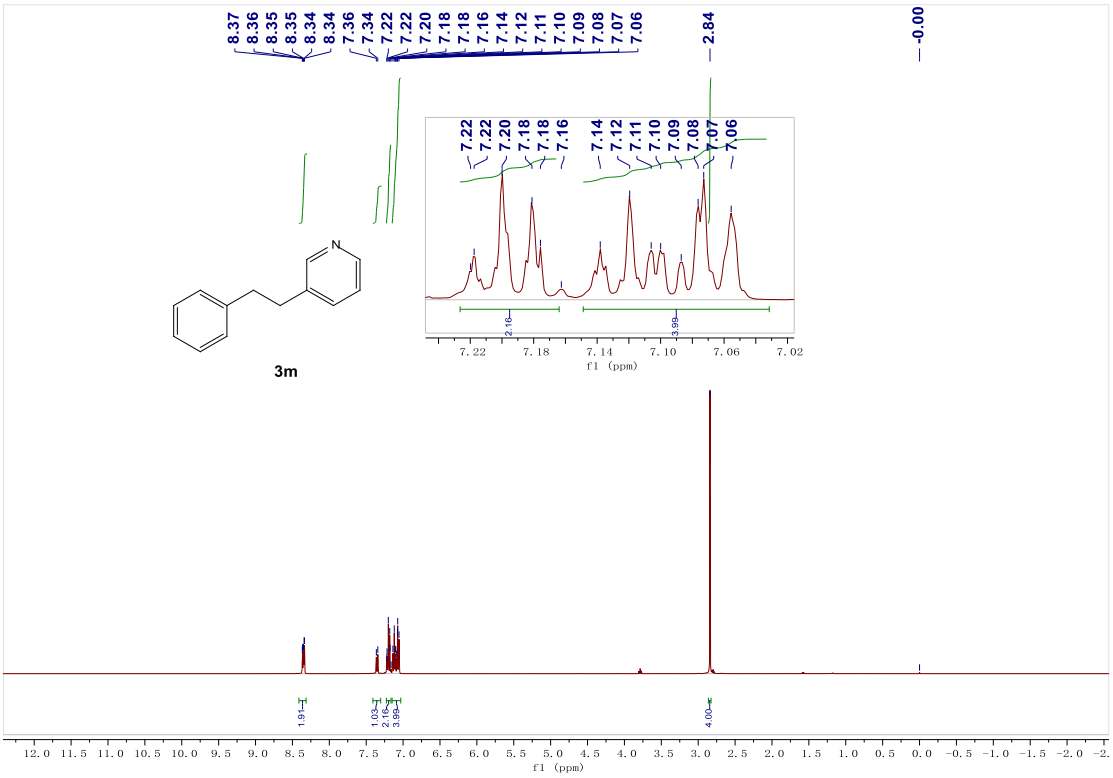
f1 (ppm)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7.07
7.06
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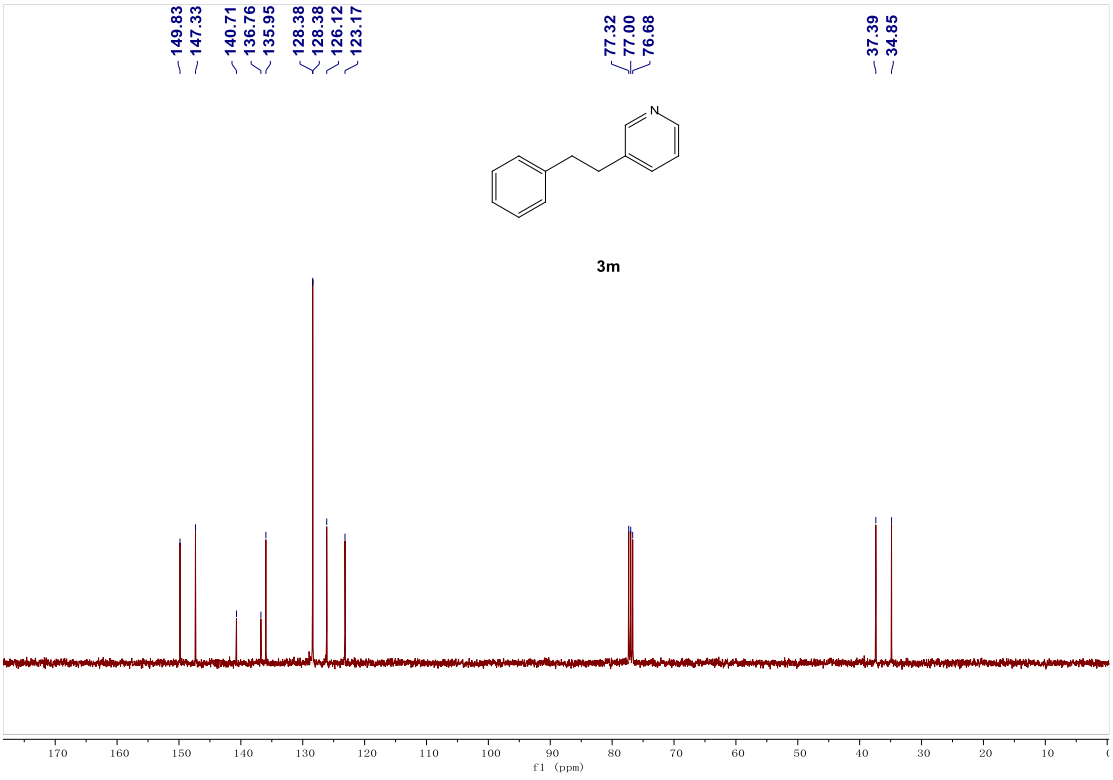
7.22
7.18
7.14
7.10
7.06
7.02
f1 (ppm)
N
3m
1.91
1.03
2.16
3.99
4.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0 -2.
f1 (ppm)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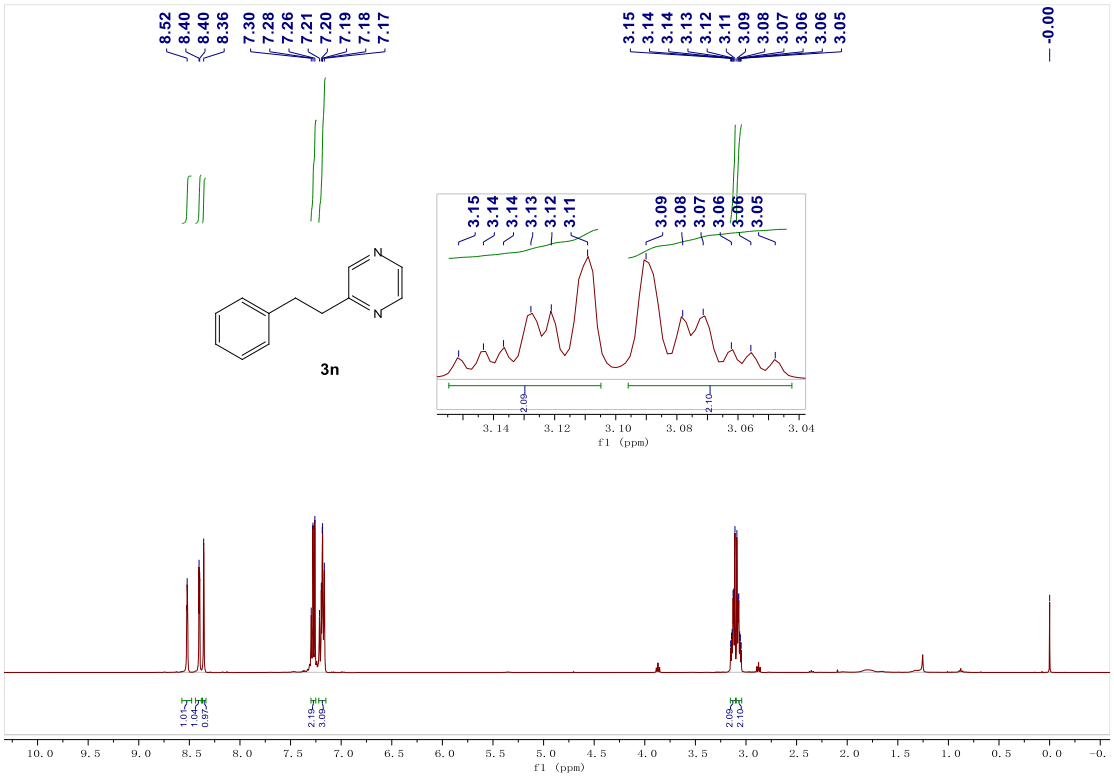
f1 (ppm)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3.15
3.14
3.14
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3.08
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3.06
3.06
3.05
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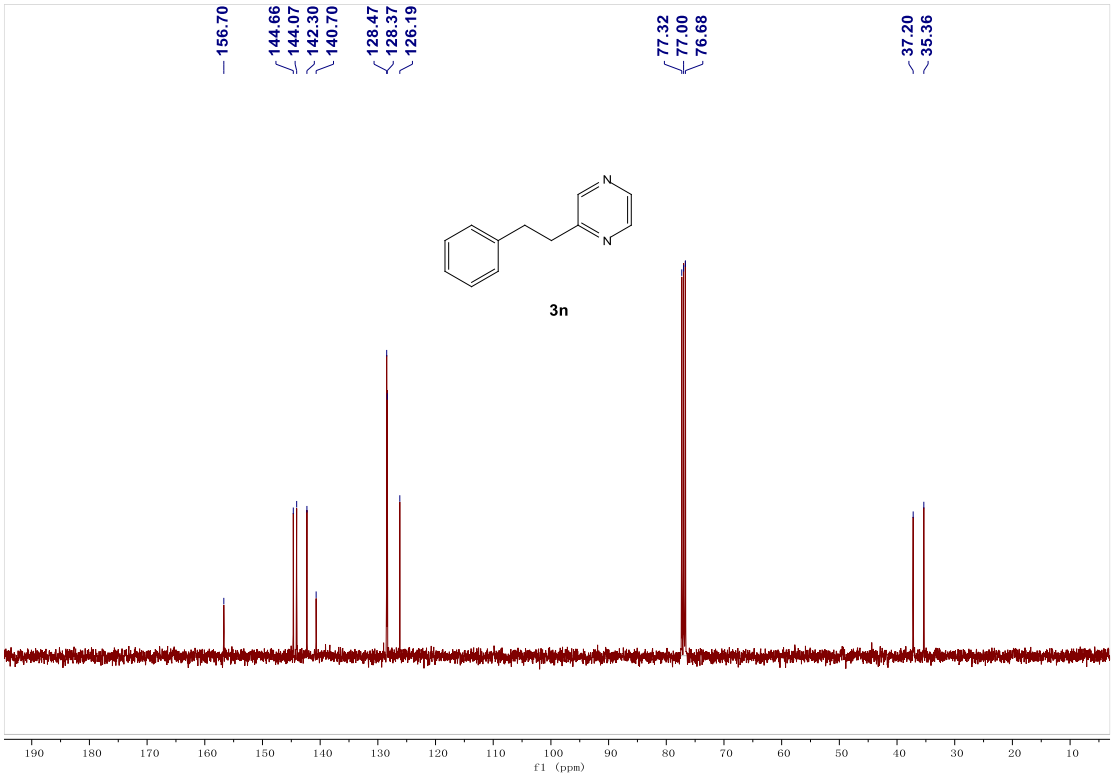
3n
f1 (ppm)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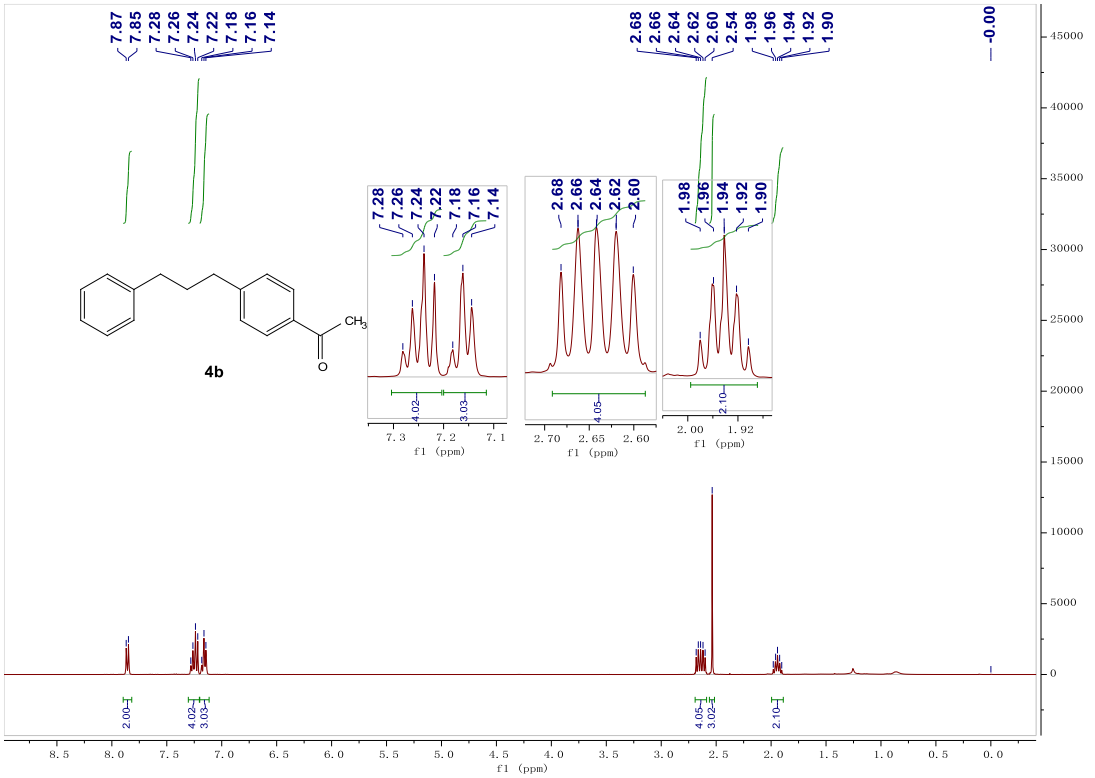
3n
f1 (ppm)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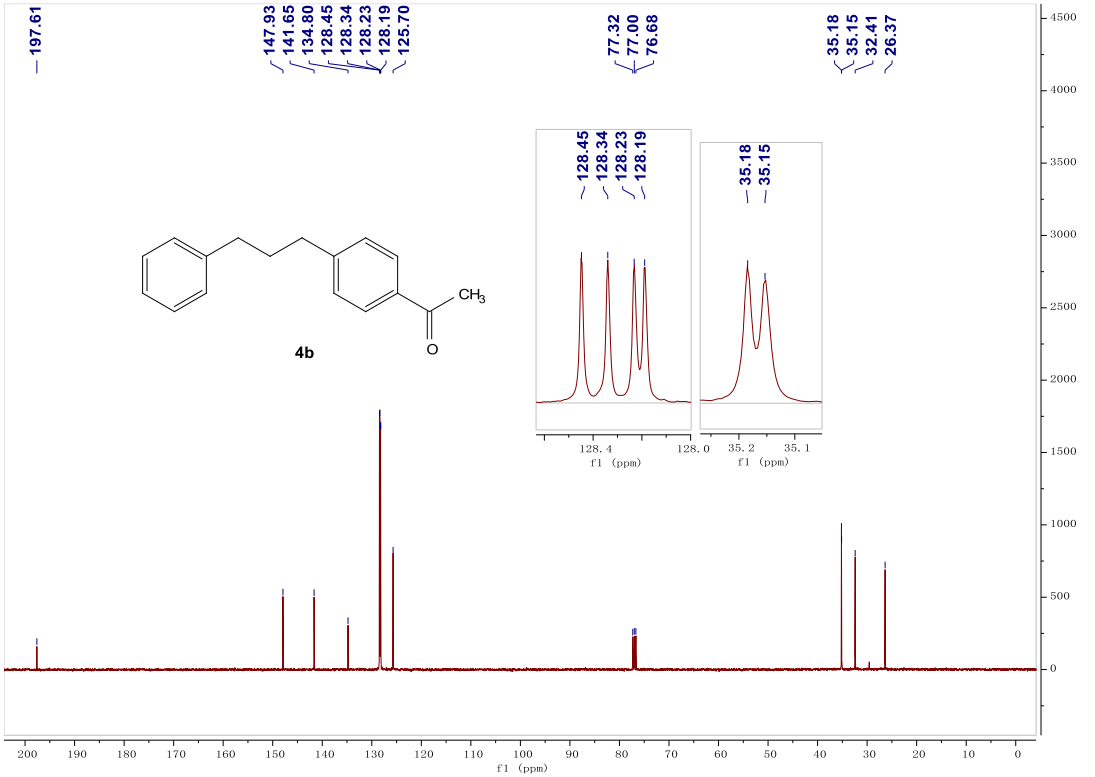
O
4b
f1 (ppm)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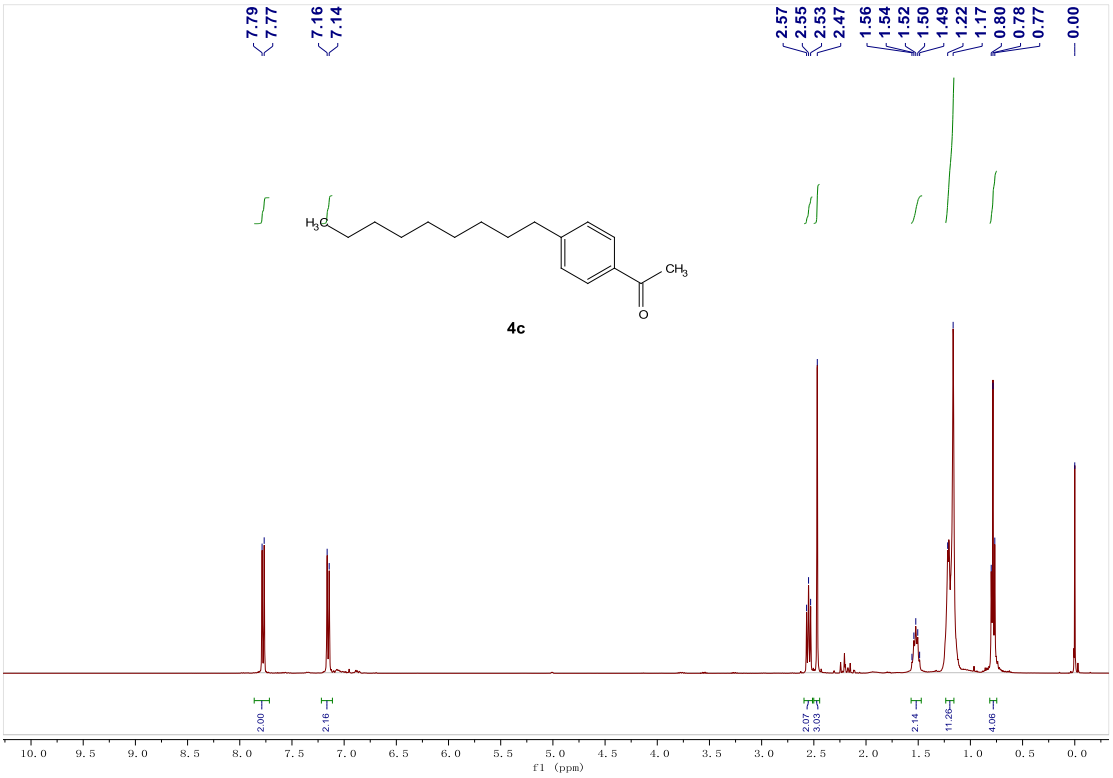
O
4b
f1 (ppm)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1.17
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0.78
0.77
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H3C
CH3
O
4c
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11.28
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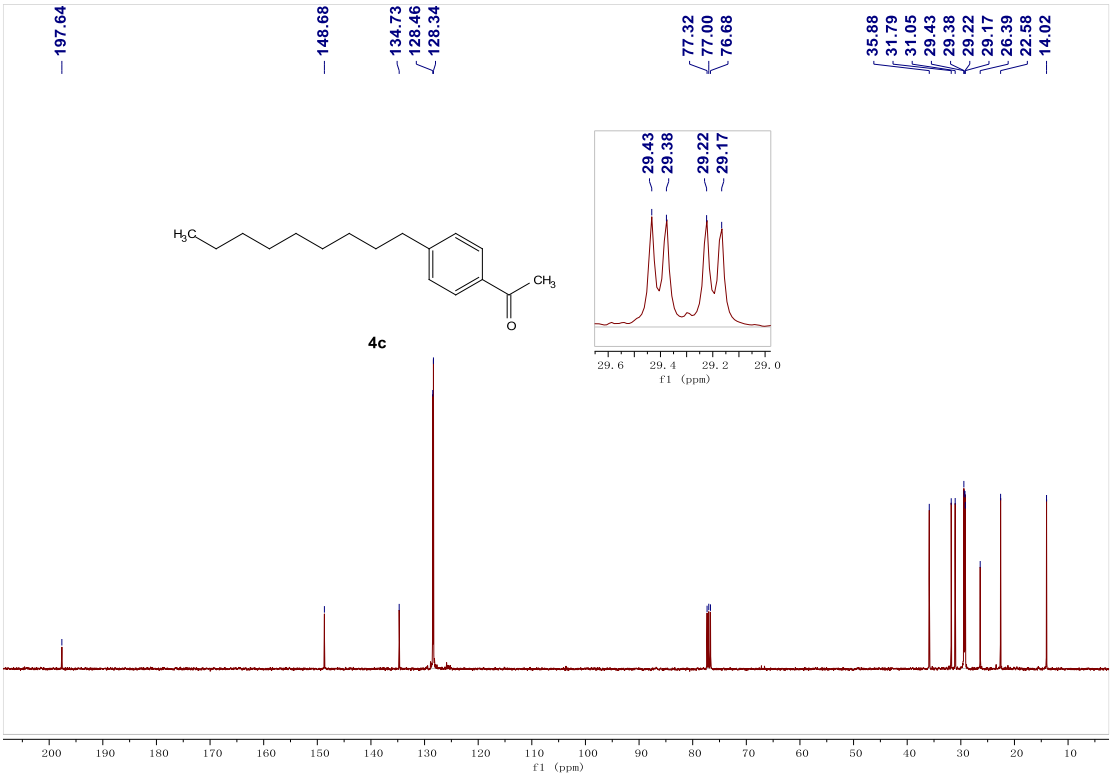
f1 (ppm)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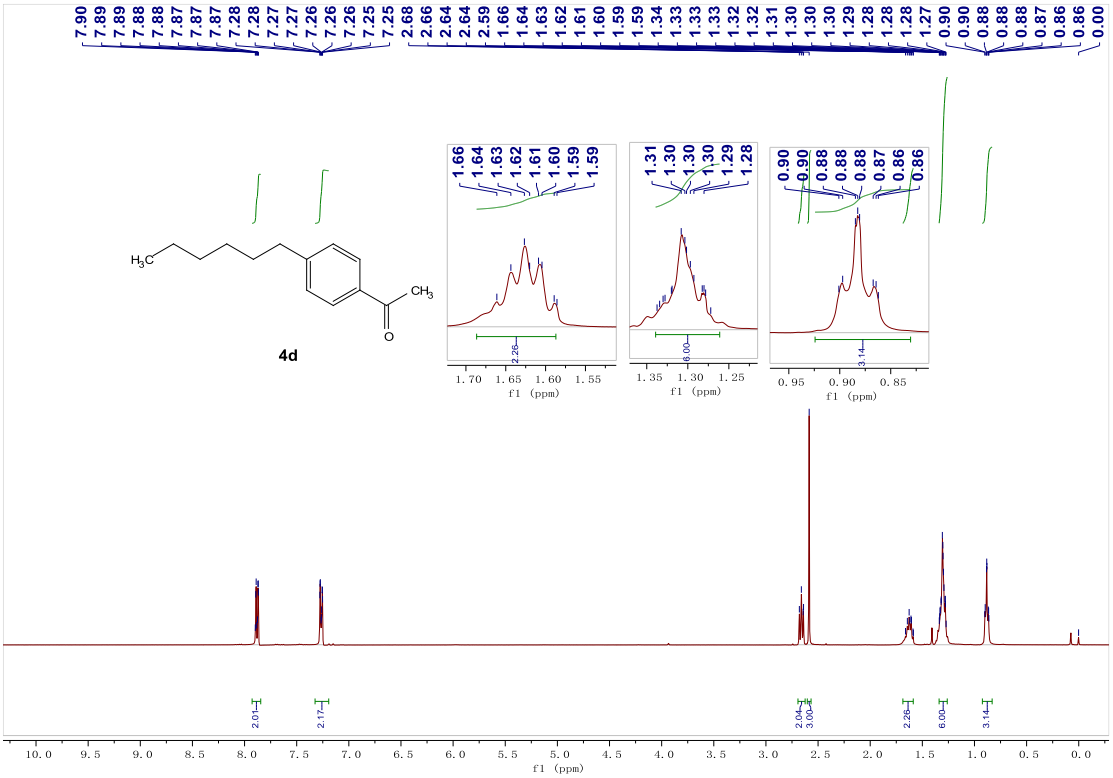
f1 (ppm)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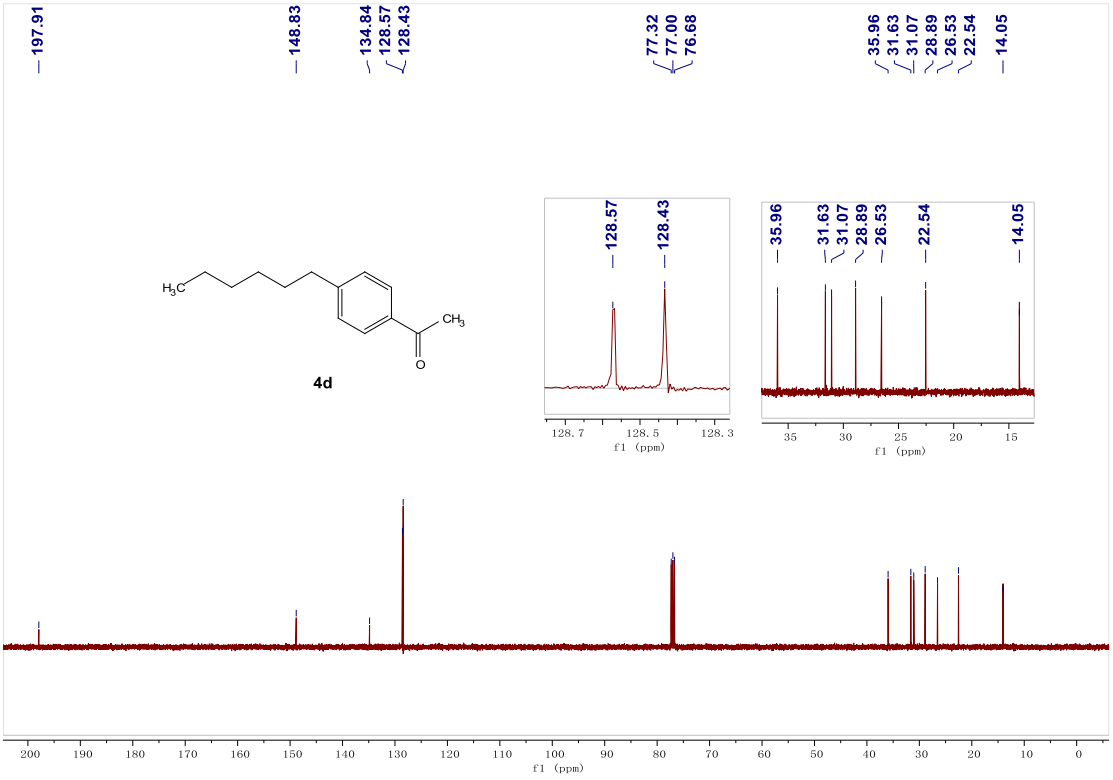
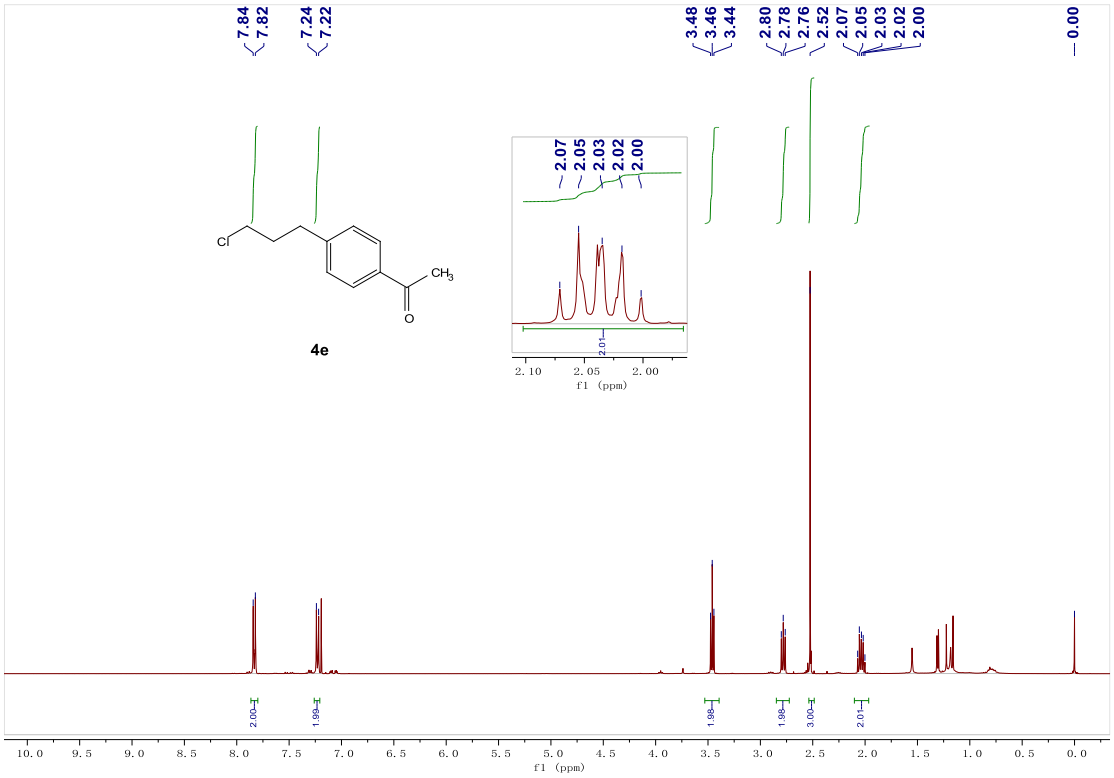
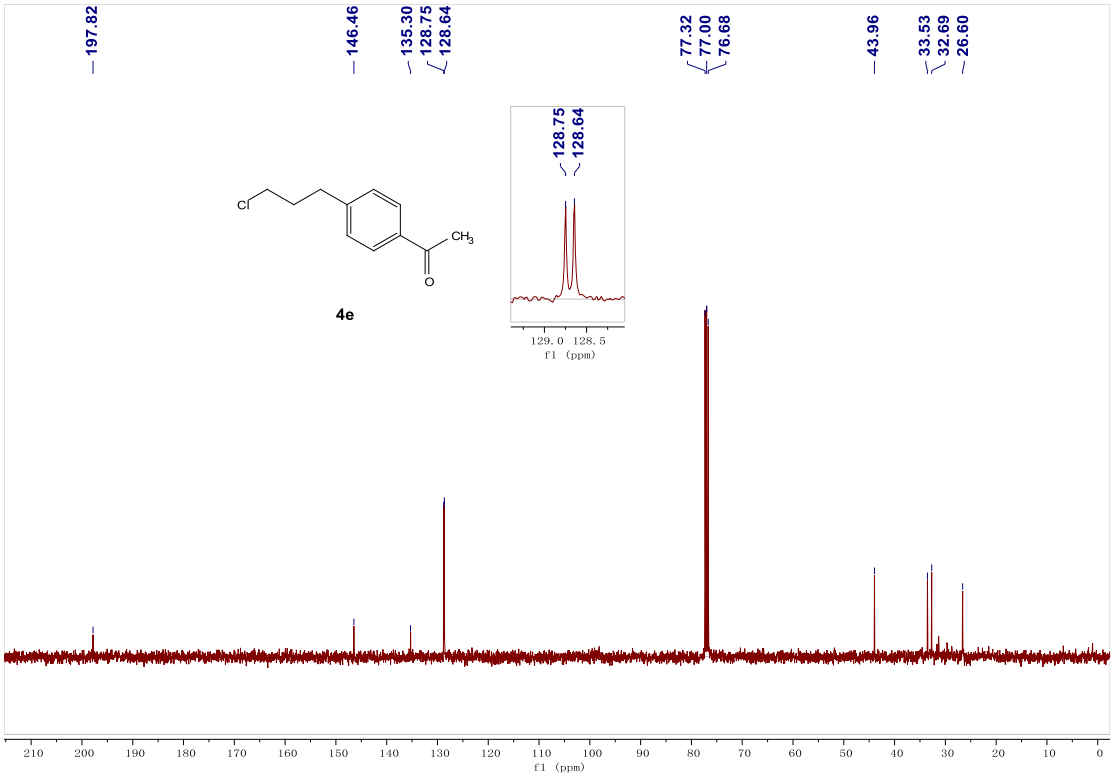
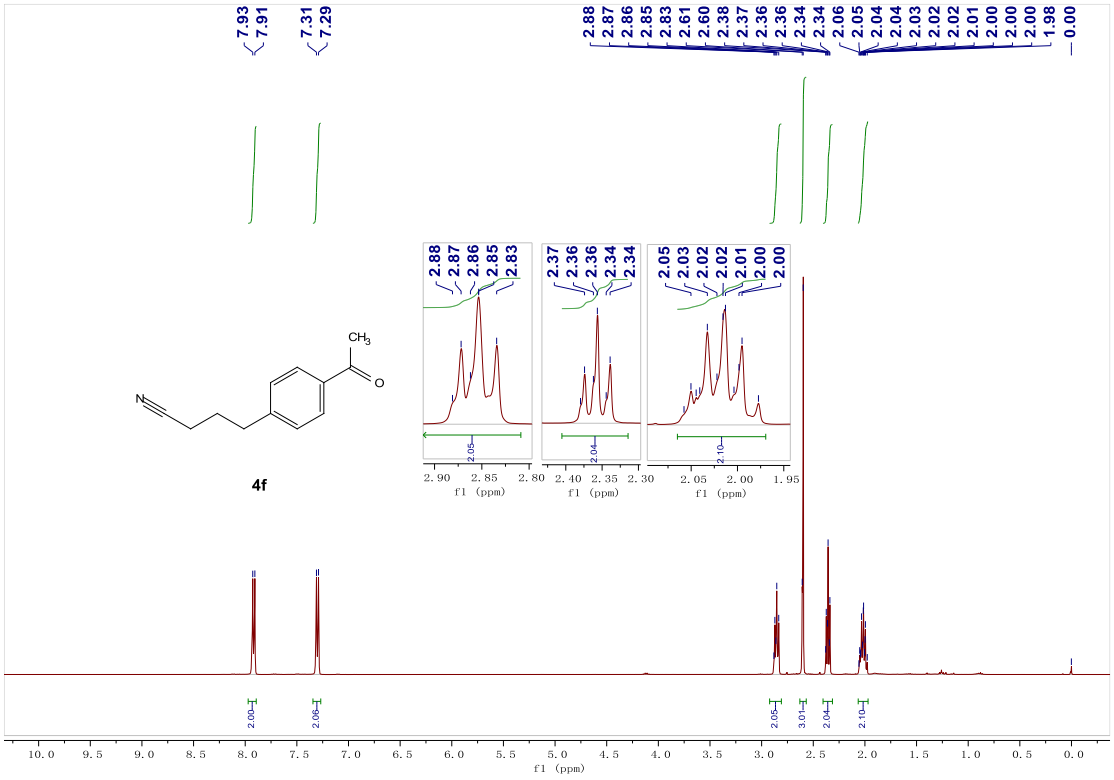
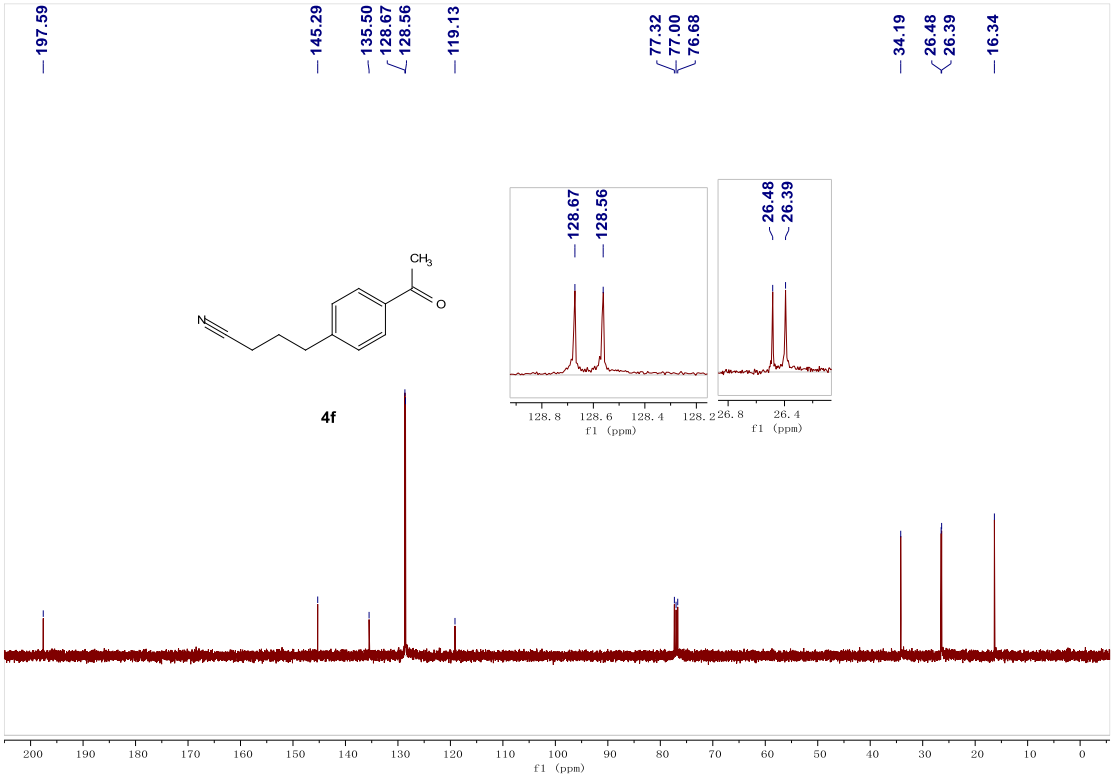
0.90 0.90 0.88 0.88 0.88 0.87 0.86 0.86 0.00
1.66 1.64 1.63 1.62 1.61 1.60 1.59 1.59
1.31 1.30 1.30 1.30 1.29 1.28
0.90 0.90 0.88 0.88 0.88 0.87 0.86 0.86
H3C
CH3
O
4d
2.26
6.00
3.14
1.70 1.65 1.60 1.55
1.35 1.30 1.25
0.95 0.90 0.85
f1 (ppm)
2.01 2.17 2.04 3.00 2.26 6.00 3.14
10.0 9.5 9.0 8.5 8.0 7.5 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)

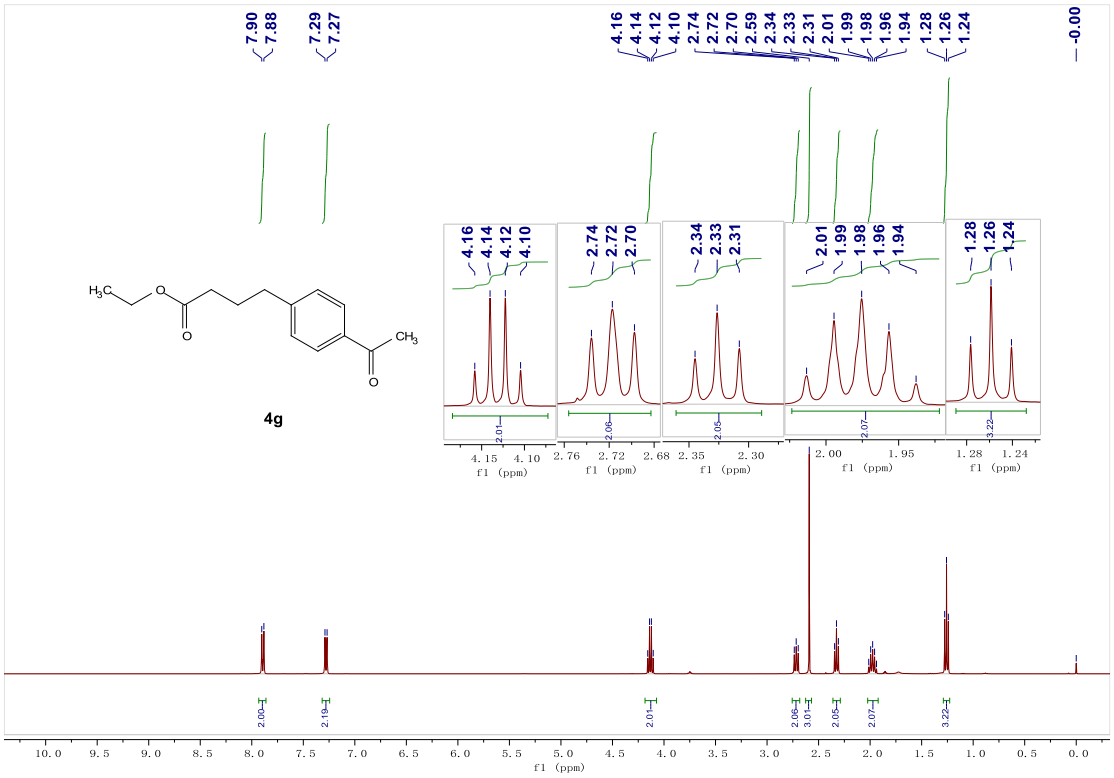

197.91
148.83
134.84
128.57
128.43
77.32
77.00
76.68
35.96
31.63
31.07
28.89
26.53
22.54
14.05
H3C
CH3
O
4d
128.7 128.5 128.3
f1 (ppm)
35 30 25 20 15
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

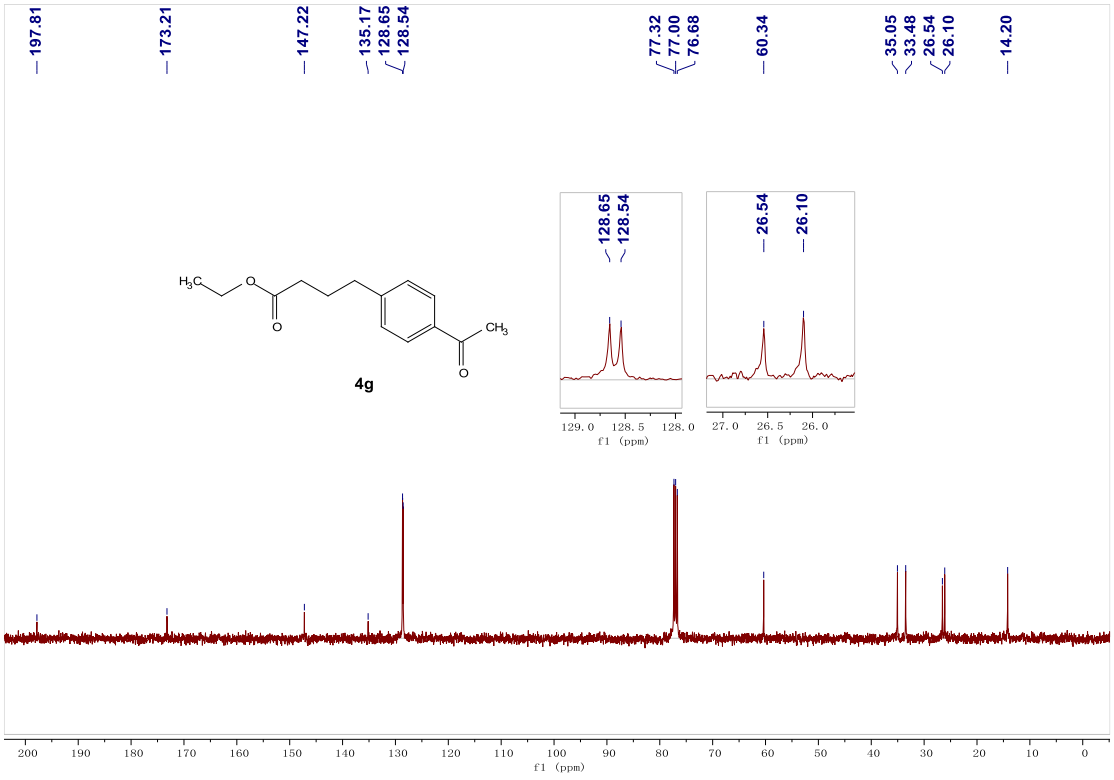

7.84
7.82
7.24
7.22
3.48
3.46
3.44
2.80
2.78
2.76
2.52
2.07
2.05
2.03
2.02
2.00
0.00
Cl
CH3
O
4e
f1 (ppm)

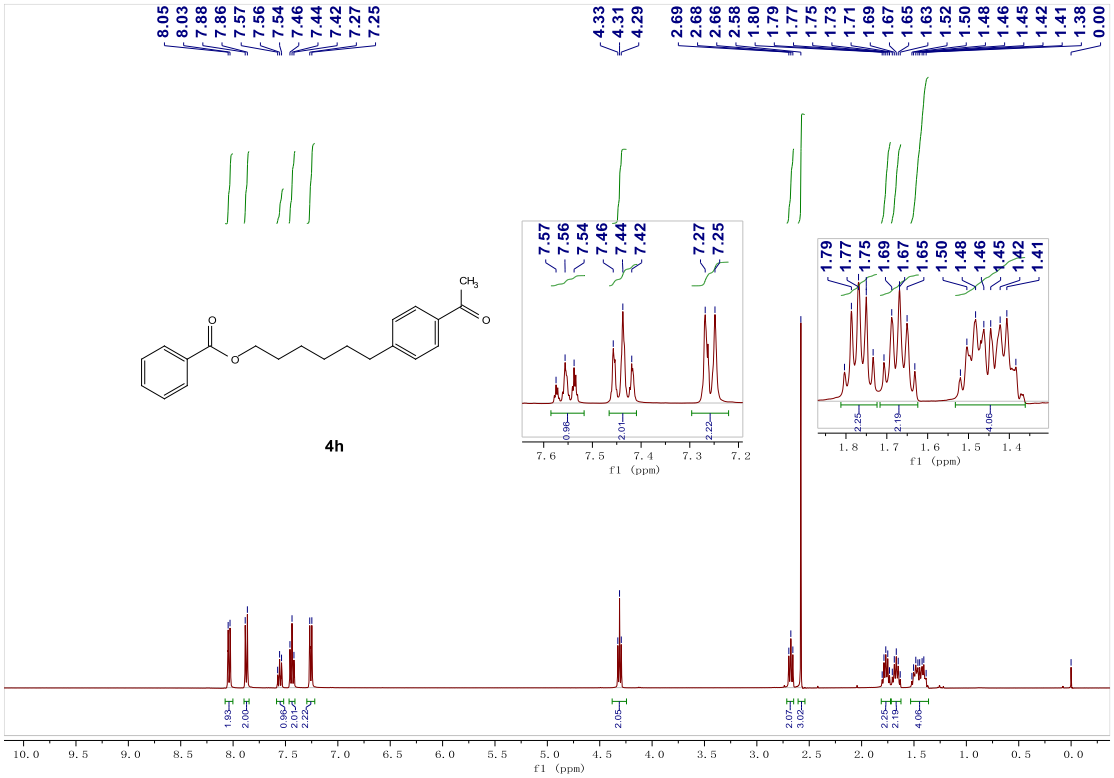

197.82
146.46
135.30
128.75
128.64
77.32
77.00
76.68
43.96
33.53
32.69
26.60
Cl
CH3
O
4e
f1 (ppm)

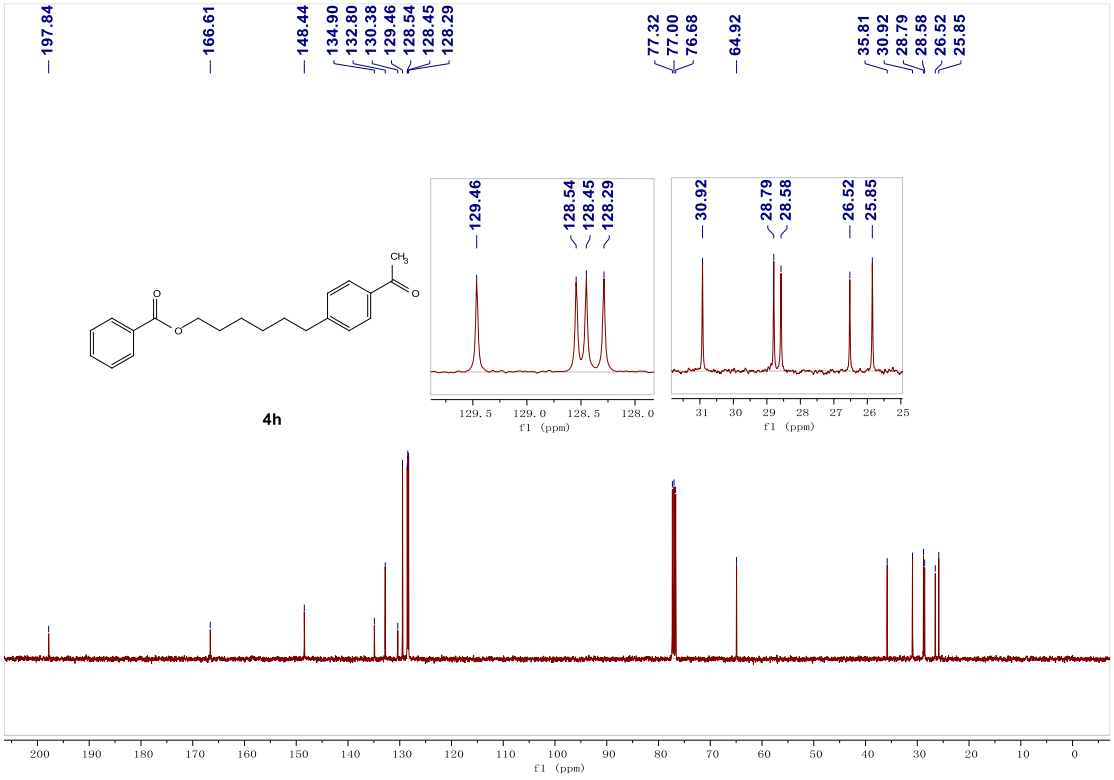

7.93
7.91
7.31
7.29
2.88
2.87
2.86
2.85
2.83
2.61
2.60
2.38
2.37
2.36
2.36
2.34
2.34
2.06
2.05
2.04
2.04
2.03
2.02
2.02
2.01
2.00
2.00
2.00
1.98
0.00
CH3
O
N
4f
f1 (ppm)

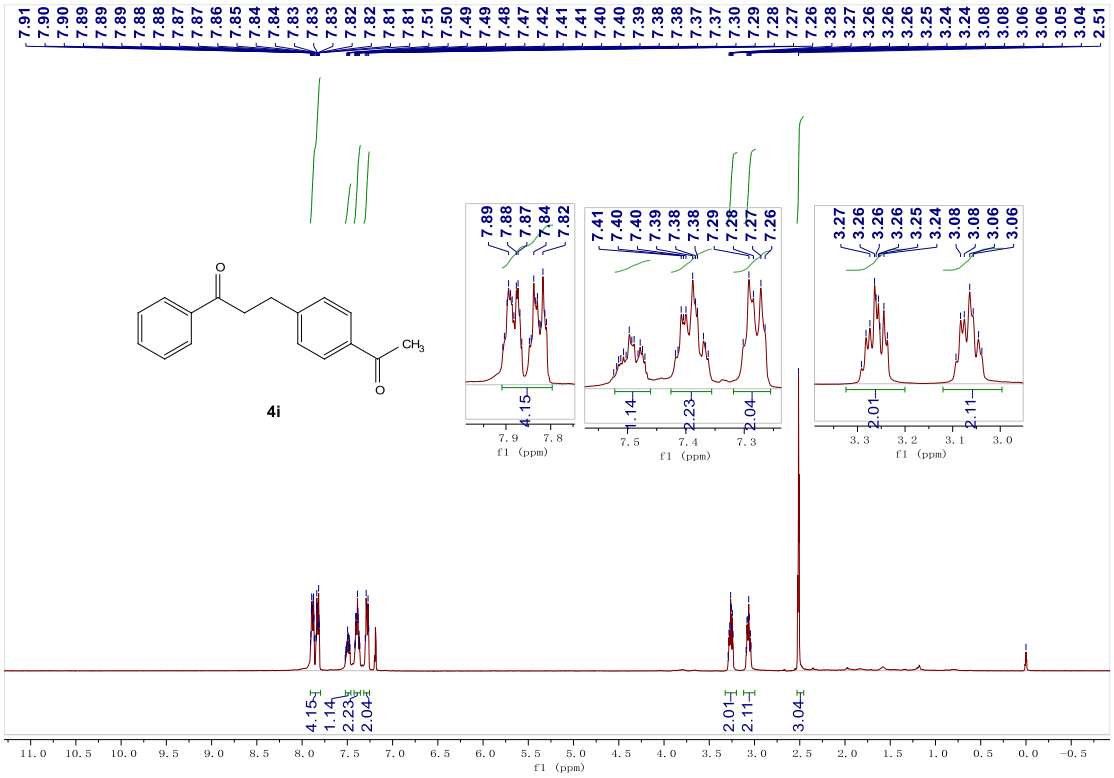

197.59
145.29
135.50
128.67
128.56
119.13
77.32
77.00
76.68
34.19
26.48
26.39
16.34
CH3
O
N
4f
f1 (ppm)

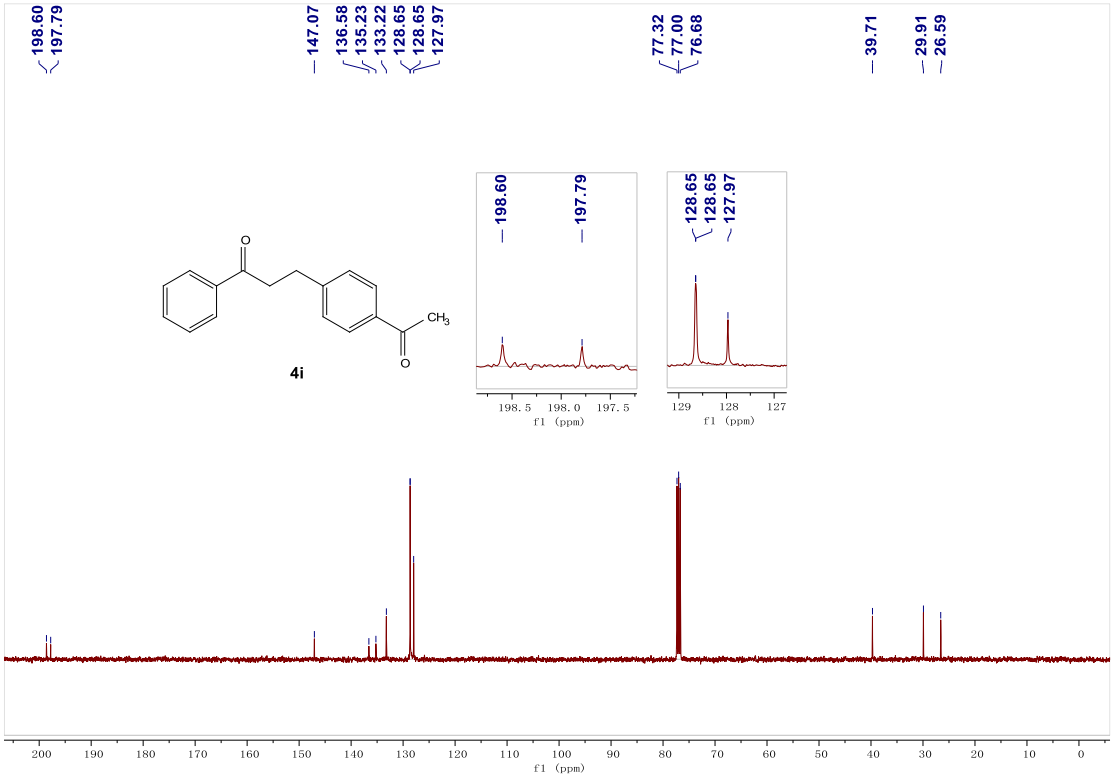

7.90
7.88
7.29
7.27
4.16
4.14
4.12
4.10
2.74
2.72
2.70
2.59
2.34
2.33
2.31
2.01
1.99
1.98
1.96
1.94
1.28
1.26
1.24
-0.00
4g
f1 (ppm)

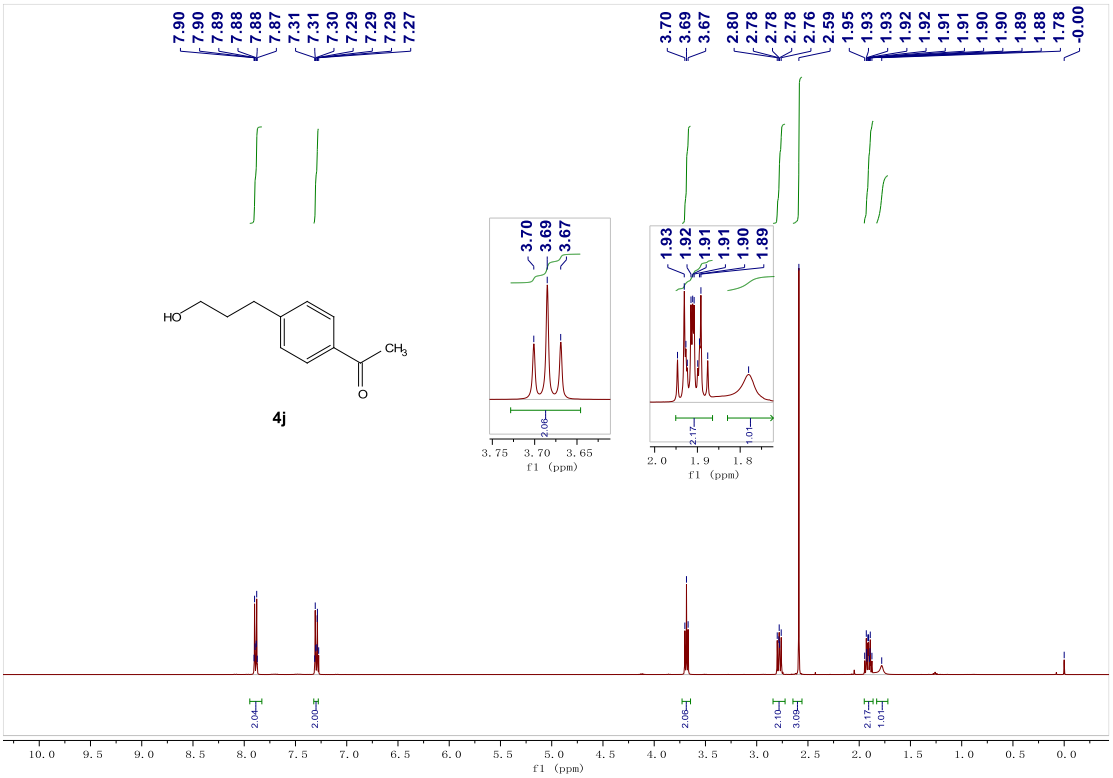

197.81
173.21
147.22
135.17
128.65
128.54
77.32
77.00
76.68
60.34
35.05
33.48
26.54
26.10
14.20
4g
f1 (ppm)

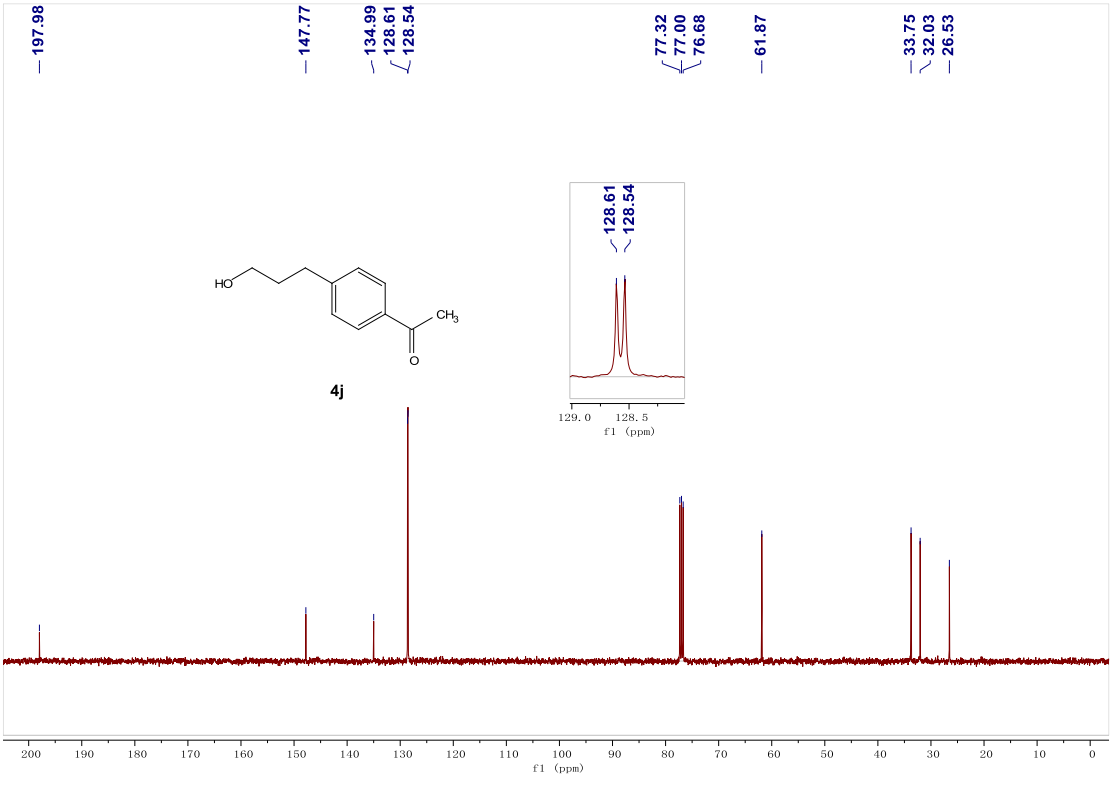

8.05
8.03
7.88
7.86
7.57
7.56
7.54
7.46
7.44
7.42
7.27
7.25
4.33
4.31
4.29
2.69
2.68
2.66
2.58
1.80
1.79
1.77
1.75
1.73
1.71
1.69
1.67
1.65
1.63
1.52
1.50
1.48
1.46
1.45
1.42
1.41
1.38
0.00
CH3
O
O
O
4h
f1 (ppm)

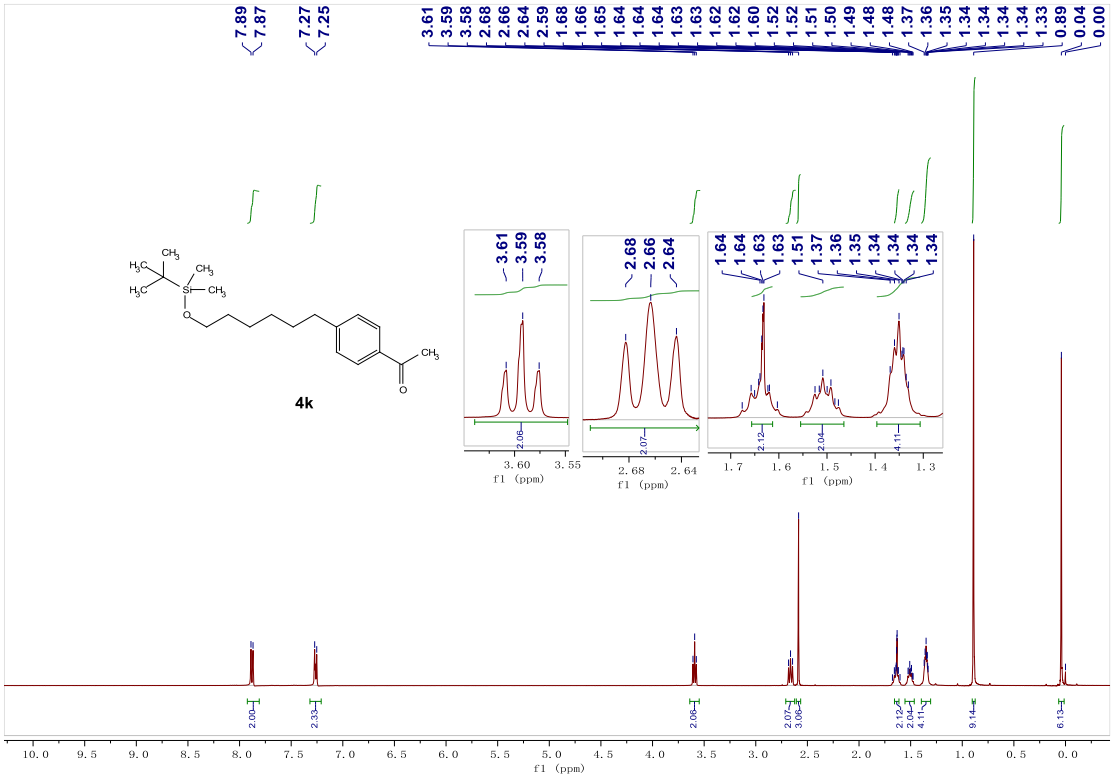

197.84
166.61
148.44
134.90
132.80
130.38
129.46
128.54
128.45
128.29
77.32
77.00
76.68
64.92
35.81
30.92
28.79
28.58
26.52
25.85
CH3
O
O
O
4h
f1 (ppm)

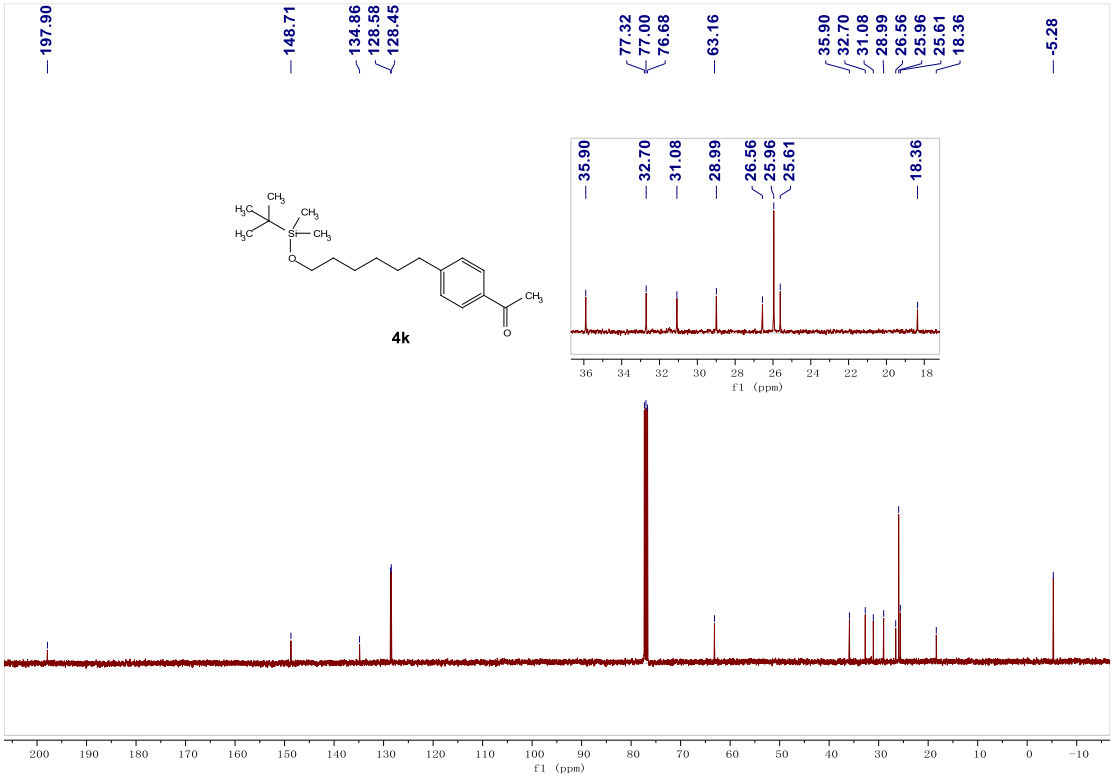

4i
f1 (ppm)

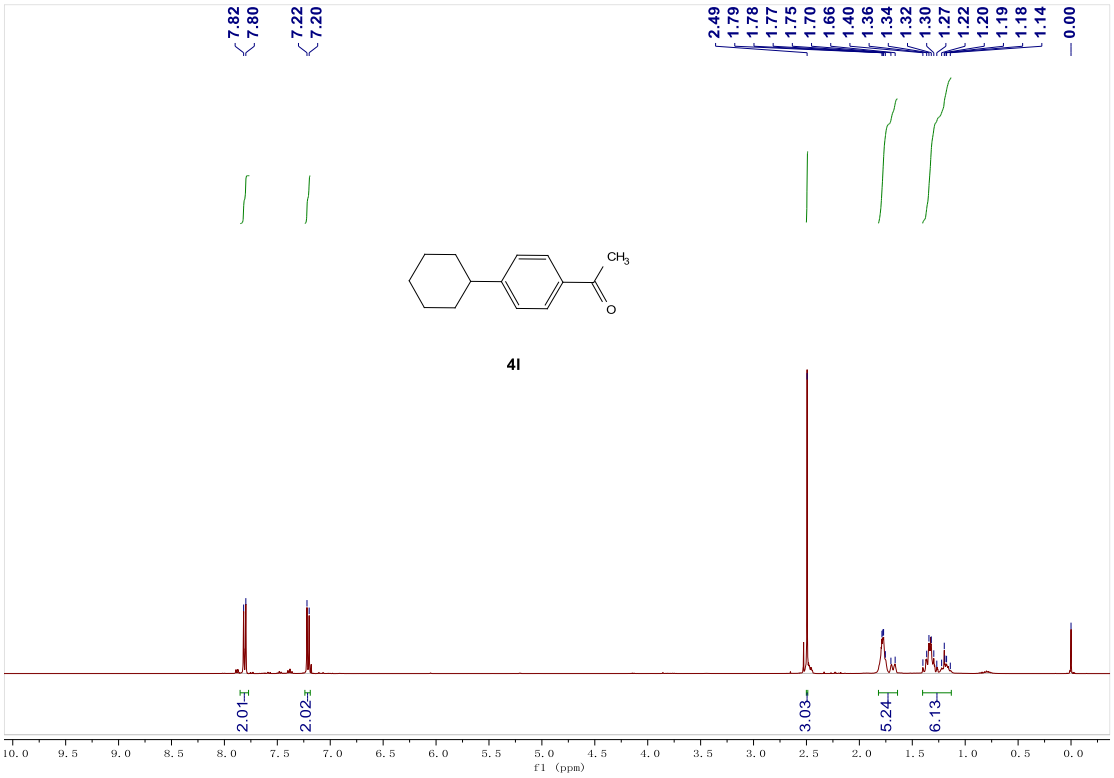

4i
f1 (ppm)

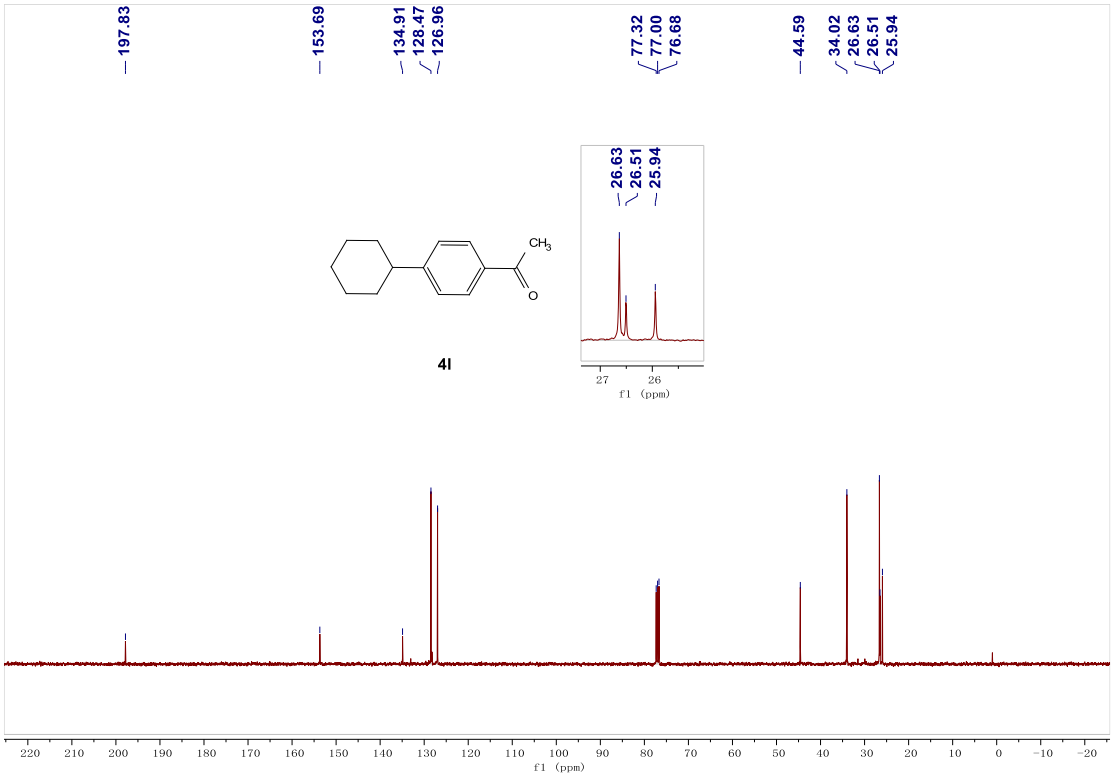

7.90
7.90
7.89
7.88
7.88
7.87
7.31
7.31
7.30
7.29
7.29
7.29
7.27
3.70
3.69
3.67
2.80
2.78
2.78
2.78
2.76
2.59
1.95
1.93
1.93
1.92
1.92
1.91
1.91
1.90
1.90
1.89
1.88
1.78
-0.00
HO
CH3
O
4j
3.70
3.69
3.67
2.06
3.75 3.70 3.65
f1 (ppm)
1.93
1.92
1.91
1.91
1.90
1.89
2.17
1.01
2.0 1.9 1.8
f1 (ppm)
2.04
2.00
2.06
2.10
3.09
2.17
1.01
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)

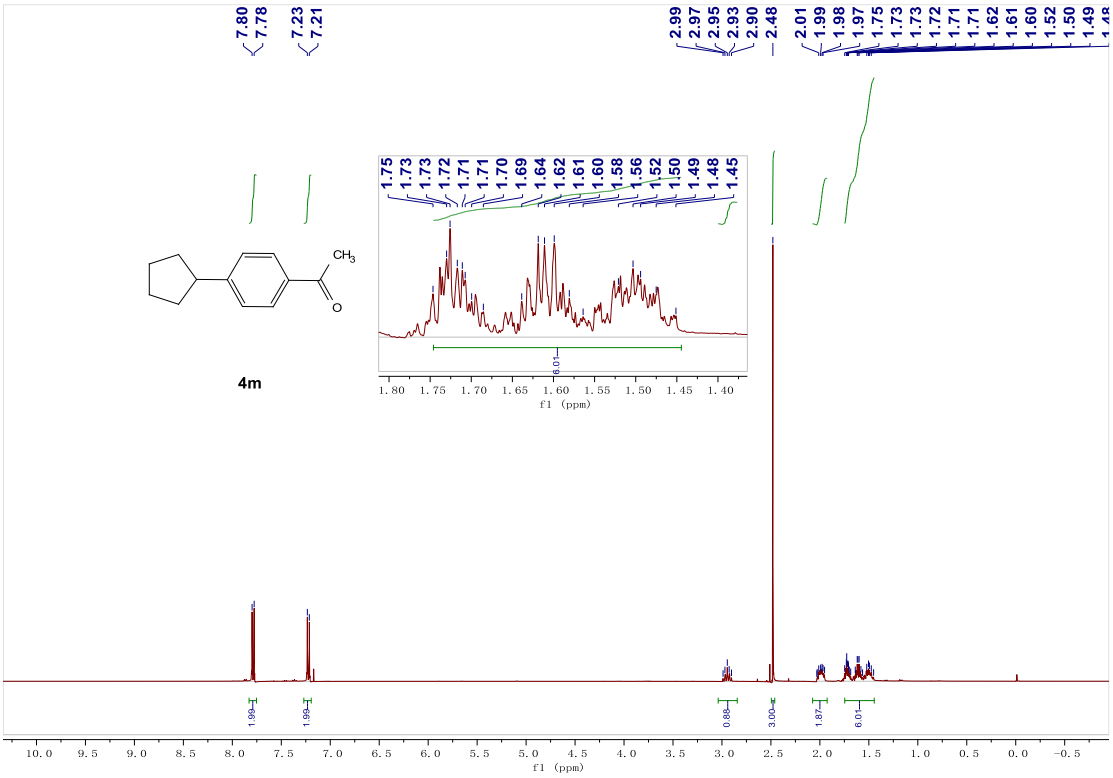

197.98
147.77
134.99
128.61
128.54
77.32
77.00
76.68
61.87
33.75
32.03
26.53
HO
CH3
O
4j
128.61
128.54
129.0 128.5
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

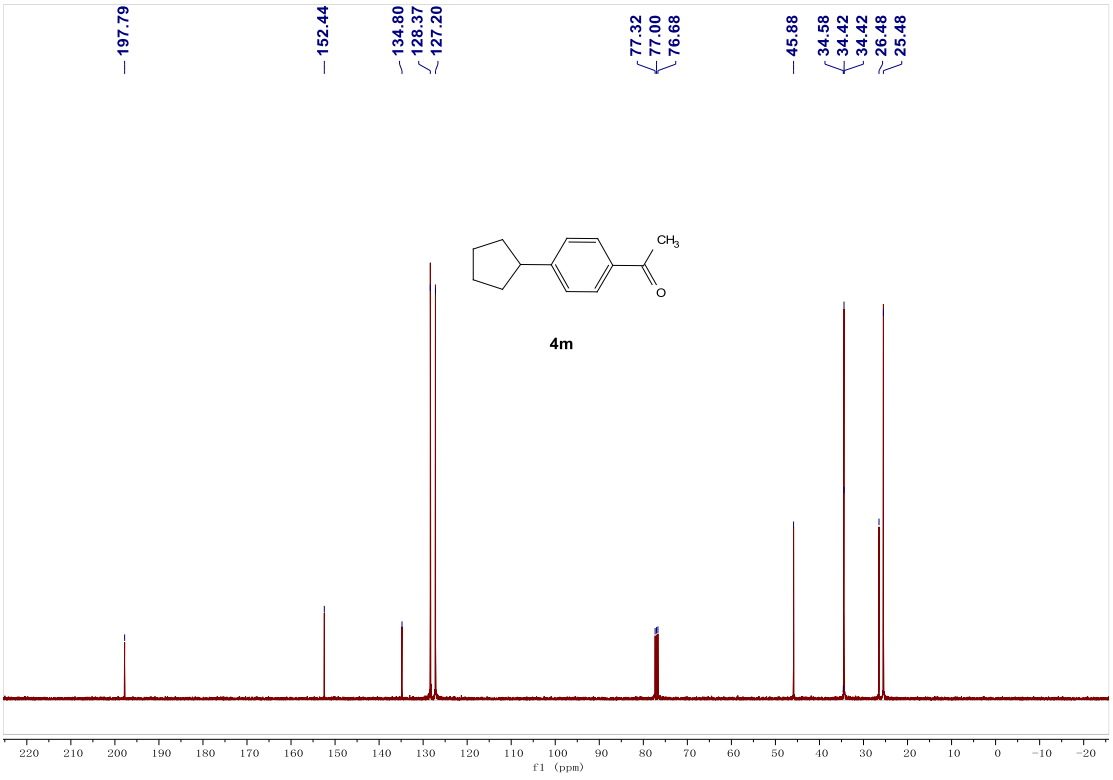

7.89
7.87
7.27
7.25
3.61
3.59
3.58
2.68
2.66
2.64
2.59
1.68
1.66
1.65
1.64
1.64
1.64
1.63
1.63
1.62
1.62
1.60
1.52
1.52
1.51
1.50
1.49
1.48
1.48
1.37
1.36
1.35
1.34
1.34
1.34
1.34
1.33
0.89
0.04
0.00
4k
f1 (ppm)


197.90
148.71
134.86
128.58
128.45
77.32
77.00
76.68
63.16
35.90
32.70
31.08
28.99
26.56
25.96
25.61
18.36
-5.28
4k
f1 (ppm)


7.82
7.80
7.22
7.20
2.49
1.79
1.78
1.77
1.75
1.70
1.66
1.40
1.36
1.34
1.32
1.30
1.27
1.22
1.20
1.19
1.18
1.14
0.00
CH3
O
4l
2.01
2.02
3.03
5.24
6.13
f1 (ppm)


197.83
153.69
134.91
128.47
126.96
77.32
77.00
76.68
44.59
34.02
26.63
26.51
25.94
CH3
O
4l
f1 (ppm)


7.80
7.78
7.23
7.21
2.99
2.97
2.95
2.93
2.90
2.48
2.01
1.99
1.98
1.97
1.75
1.73
1.72
1.71
1.62
1.61
1.60
1.52
1.50
1.49
1.75
1.73
1.73
1.72
1.71
1.71
1.70
1.69
1.64
1.62
1.61
1.60
1.58
1.56
1.52
1.50
1.49
1.48
1.45
CH3
O
4m
6.01
1.80 1.75 1.70 1.65 1.60 1.55 1.50 1.45 1.40
f1 (ppm)
1.99
1.99
0.88
3.00
1.87
6.01
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 -0.5
f1 (ppm)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152.44
134.80
128.37
127.20
77.32
77.00
76.68
45.88
34.58
34.42
34.42
26.48
25.48
CH3
O
4m
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)