Electronic Supplementary Information (ESI)

Aldehyde Effect and Ligand Discovery in Ru-Catalyzed Dehydrogenative Cross-Coupling of Alcohols to Esters

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Supporting Information

1. General information

Unless otherwise stated, all the materials were purchased from commercial suppliers and were used as received. Solvents were dried by solvent purification system from LC Technology Solution Inc. Flash chromatography was performed on SepaBeanTM machine. Products were characterized by ^{1}H NMR, ^{13}C NMR, ^{31}P NMR and HRMS spectroscopy. All ^{1}H, ^{13}C NMR and ^{31}P NMR spectra were recorded on Bruker 400 MHz spectrometer or Varian 400 MHz spectrometer. ^{1}H NMR and $^{13}C\{^{1}H\}$ NMR chemical shifts are reported in ppm referenced to tetramethylsilane or the residual signals of the deuterated solvent. $^{31}P\{^{1}H\}$ NMR chemical shifts are reported in ppm referenced to an external 85% solution of phosphoric acid in D_2O. The NMR chemical shift values refer to $CDCl_3$ (δ (^{1}H), 7.26 ppm; δ (^{13}C), 77.16 ppm). In ^{13}C NMR, the C-P coupling constant data is omitted due to the complexity, except for aliphatic carbon atoms. GC-MS data were obtained on Shimadzu GCMS-P2010 SE, GC data were obtained on Shimadzu GC-2010 Plus. All measurements were carried out at room temperature unless otherwise stated.

2.1 General procedure for reactions in Scheme 2: To a 38 mL sealing tube in an nitrogen-filled glove box, the metal source and different kinds of PNP or PNN ligands were added followed by addition of solvent (2 mL). Then the tube was sealed and the resulted mixture was stirred outside the glove box at 120 °C for 1 h (to generate the catalyst *in situ*). The mixture was allowed to cool down to room temperature. To the mixture were added butanol or another aliphatic chain alcohol (0.3 mmol) and *t*BuOK under N_2 atomsphere. The reaction was stirred at 100 °C for 4 h. The yield was determined by GC analysis using *n*-dodecane or *n*-hexadecane as an internal standard.

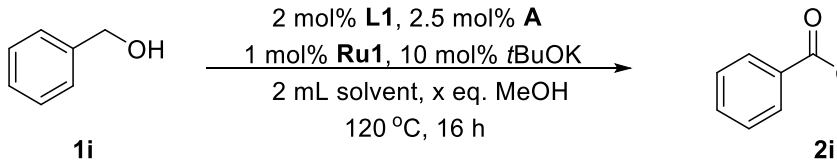
2.2 General procedure for reactions of 2-phenylethanol and methanol (Table 1): To a 38 mL sealing tube in an nitrogen-filled glove box, the metal source, PNP ligand (obtained from in-*situ* neutralization of PNP.HCl salt with *t*BuOK) and aldehyde were added followed by addition of solvent (2 mL). Then the tube was sealed and the resulted mixture was stirred outside the glove box at 120 °C for 1 h (to generate the catalyst). The mixture was allowed to cool down to room temperature. To this mixture were added 2-phenylethanol (0.3 mmol), methanol (4.5 mmol) and K_2CO_3 under N_2 atomsphere. The reaction was stirred at 120 °C for 16 h. The yield was determined by GC analysis using *n*-tetradecane as an internal standard.

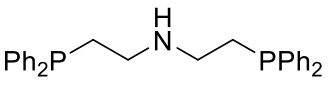
2.3 Condition optimization for the reaction of benzyl alcohol and methanol.

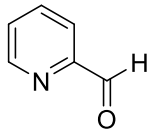
Reaction procedure: To a 38 mL sealing tube in an nitrogen-filled glove box, the metal source, PNP ligand (obtained from in-*situ* neutralization of PNP.HCl salt with *t*BuOK) and aldehyde were added followed by addition of solvent (2 mL). Then the tube was sealed and the resulted mixture was stirred outside the glove box at 120 °C for 1 h (to generate the catalyst). The mixture was allowed to cool down to room temperature. To this mixture were added benzyl

alcohol (0.3 mmol), methanol (4.5 mmol) and K_2CO_3 under N_2 atomsphere. The reaction was stirred at 120 °C for 16 h. The yield was determined by GC analysis using *n*-tetradecane as an internal standard.

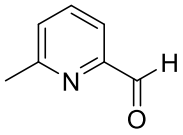
Table S1 Investigation of different ligands and solvents for the reaction of benzyl alcohol and methanol[a]







A1



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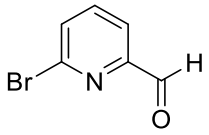

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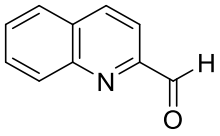

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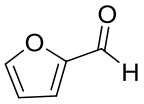

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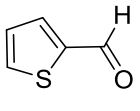


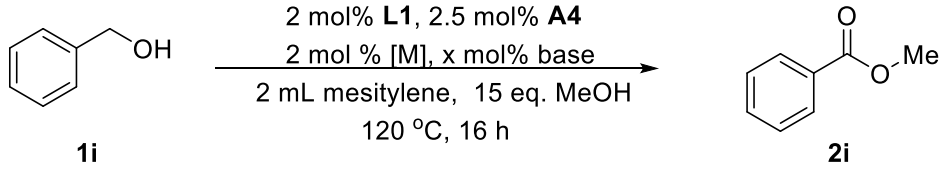
A8

Entry	A	Solvent	MeOH (eq.)	Yield (%)[b]
1	**A1**	toluene	15	6
2	**A2**	toluene	15	8
3	**A3**	toluene	15	9
4	**A4**	toluene	15	19
5	**A7**	toluene	15	7
6	**A8**	toluene	15	8
7	**A4**	toluene	2	5
8	**A4**	toluene	5	12
9	**A4**	toluene	40	trace
10	**A4**	MeOH	0	0
11	**A4**	CPME	15	8
12	**A4**	mesitylene	15	37

13	**A4**	THF	15	trace
14	**A4**	1,4-dioxane	15	12
15	**A4**	DEDM	15	trace

[a]Reaction conditions: **1i** (0.3 mmol), **Ru1** (1 mol%), **L1** (2 mol%), **A** (2.5 mol%) , *t*BuOK (10 mol%), MeOH (X eq.) and solvent (2 mL) for 16 h, unless otherwise noted. [b]Determined by GC analysis using *n*-tetradecane as an internal standard. CPME = Cyclopentyl methyl ether. DEDM = Diethylene glycol dimethyl ether.

Table S2 Investigation of different ruthenium sources and bases for the reaction of benzyl alcohol and methanol[a]



1i **2i**

$[RuCl_2(p\text{-cymene})]_2$ $Ru(2\text{-Me-allyl})_2(cod)$ $Ru(acac)_3$

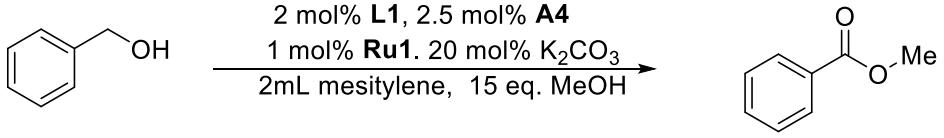
[M]:

Ru1 **Ru2** **Ru3**

Entry	[M]	[M] (mol%)	Base	Base (mol%)	Yield (%)[b]
1	**Ru1**	1	*t*BuOK	10	37
2	**Ru2**	2	*t*BuOK	10	42
3	**Ru3**	2	*t*BuOK	10	18
4	$RuCl_2(DMSO)_4$	2	*t*BuOK	10	25
5	**Ru1**	1	KOH	10	37
6	**Ru1**	1	K_2CO_3	10	55
7	**Ru1**	1	Et_3N	10	0
8	**Ru1**	1	K_2CO_3	20	68
9	**Ru1**	1	K_2CO_3	40	69
10	**Ru1**	1	K_2CO_3	60	72

[a]Reaction conditions: **1i** (0.3 mmol), **L1** (2 mol%), **A4** (2.5 mol%) , base (x mol%), MeOH (15 eq.) and mesitylene (2 mL) at 120 ℃ for 16 h, unless otherwise noted. [b]Determined by GC analysis using *n*-tetradecane as an internal standard.

Table S3 Investigation of reaction temperatures and time for the reaction of benzyl alcohol and methanol[a]



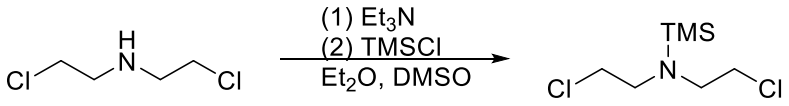
1i **2i**

Entry	Temp (°C)	Time (h)	Yield (%)[b]
1	60	16	0
2	80	16	8
3	100	16	32
4	120	16	72
5	120	4	30
6	120	8	50

[a]Reaction conditions: **1i** (0.3 mmol), **Ru1** (1 mol%), **L1** (2 mol%), **A4** (2.5 mol%) , K_2CO_3 (20 mol%), MeOH (15 eq.) and mesitylene (2 mL). [b]Determined by GC analysis using *n*-tetradecane as an internal standard.

3. Characterization data

3.1.1 Syntheses of bis(2-chloroethyl)trimethylsilylamine

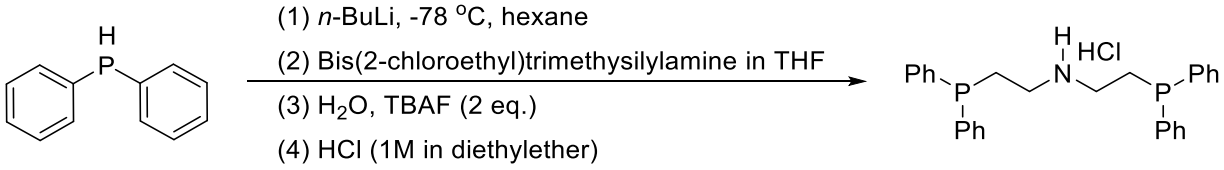


3.1.2 The title compound was synthesized following the reported procedure.[1]

To a stirred suspension of bis(2-chloroethyl)amine hydrochloride (10 g, 56.0 mmol) in 100 mL Et_2O at 0 °C, 0.25 mL DMSO, triethylamine (17.0 g, 168.0 mmol) and trimethylchlorosilane (21.3 g, 196 mmol) were added dropwise over half an hour. Then the solution was stirred for one hour at 0 °C, followed by warming up to room temperature and stirrring for further 3-5 days. The solution was filtered and the volatiles of the liquid portion were removed in vacuo and the product was obtained as yellow viscous liquid (9.5 g, 44.4 mmol, 79.2 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.37 (t, J = 7.4 Hz, 4H), 3.03 (t, J = 7.4 Hz, 4H), 0.07 (s, 9H).

3.2.1 Synthesis of bis(2-(diphenylphosphanyl)ethyl)amine



3.2.2 The title compound was synthesized referring to the reported procedure.[2]

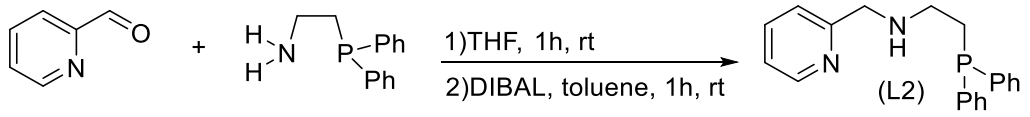
Diphenyl phospine (11 g, 59 mmol) was dissolved in 80 mL of *n*-hexane followed by cooling to -78 °C. *n*-BuLi (2.5 M in n-hexane, 25 mL, 62.5 mmol) was added dropwise to the solution. Then, after the solution was stirred for half an hour at this temperature, it was warmed up to room temperature and the yellow solution was stirred for five hours. 10 mL of THF was added and the solution was again cooled down to -40 °C. 6.32 g (29.5 mmol) of bis(2-chloroethyl)trimethylsilylamine diluted in 10 mL of THF was added dropwise with white solid precipitated out. The yellow suspension was stirred for 16 h at room temperature. Afterwards, 30 mL of water and 60 mL of TBAF (1M solution in THF, 60 mmol) were added and the resulting two-phase system was stirred for 3-5 days at room teppertature.

Most of the organic solvents were removed in vacuo and the product was extracted three times with Et_2O. The organic layer was dried over $MgSO_4$, filtered. The volatiles of the liquid portion were removed by evaporation and the white product was dried in vacuo. The pincer ligand was further purified by adding one equivalent of HCl (1M in diethylether) to achieve the corresponding HCl solid adduct.

^{1}H NMR (400 MHz, $CDCl_3$), δ 9.95 (s, 2H), 7.28(m, 20H), 2.91 (m, 4H), 2.50 (m, 4H).

^{31}P NMR (162 MHz, $CDCl_3$) δ -21.03 ppm.

3.3.1 Synthesis of 2-(Diphenylphosphaneyl)-N-(pyridin-2-ylmethyl)ethan-1-amine (L2)



3.3.2 The title compound was synthesized referring to the reported procedure.[3]

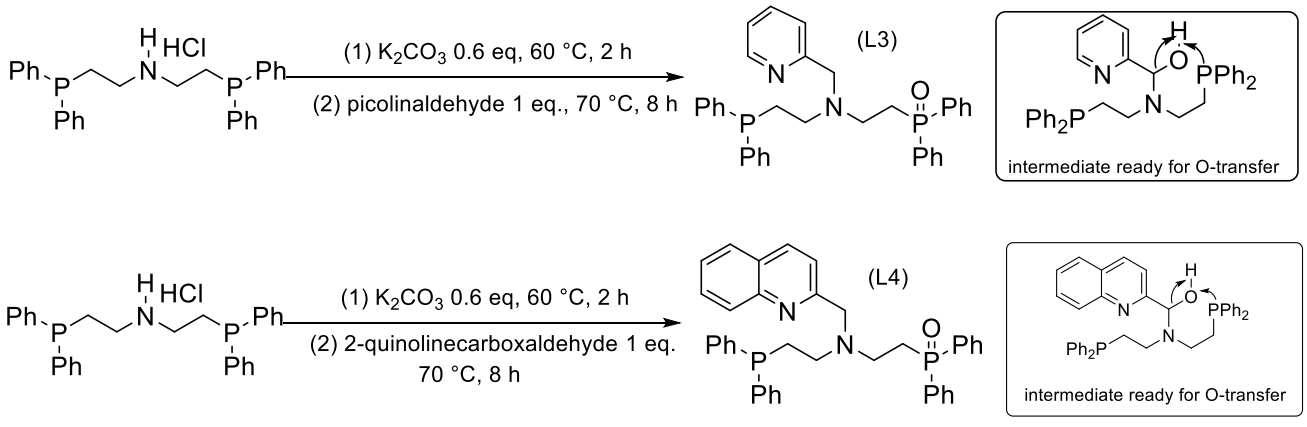
A solution of 2-picolylaldehyde (0.514 g, 4.8 mmol) in THF (5 mL) was slowly added to a solution of the PN ligand (1.0 g, 4.36 mmol) in THF (20 mL). The reaction mixture was stirred for 1 h at room temperature. After the completion of the reaction the solvent was reduced in vacuo and the residue was dissolved in toluene (20 mL). A solution of diisobutylaluminium hydride in toluene (4.36 mL, 5.23 mmol, 1.2 M) was subsequently added to the reaction mixture dropwise. The reaction mixture was stirred for another 1 h at room temperature, and then quenched with water and extracted with toluene. The organic layer was dried with Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography using ethyl acetate/Et_3N as eluent to afford **L2** as pale yellow oil. (1.20 g, 86 %).

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.53 (d, *J* = 4.8 Hz, 1H), 7.60-7.50 (m, 1H), 7.43-7.40 (m, 4H), 7.3 (m, 6H), 7.24 (m, 1H),7.14 (m, 1H), 3.88 (s, 2H), 2.81 (dd, *J* = 15.6, 7.9 Hz, 2H), 2.34-2.31 (m, 2H), 1.98 (br, NH). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.5, 149.3, 138.3 (d, *J* = 12.0 Hz), 136.4, 132.7 (d, *J* = 19.0 Hz), 128.6, 128.5, 128.4 (d, *J* = 7.0 Hz), 122.2 (d, *J* = 12.0 Hz), 55.0, 46.3 (d, *J* = 21.0 Hz), 29.0 (d, *J* = 12.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ -21.11 ppm.

3.4.1 Synthesis of

(2-((2-(diphenylphosphanyl)ethyl)(pyridin-2-ylmethyl)amino)ethyl)diphenylphosphine oxide or

(2-((2-(diphenylphosphanyl)ethyl)(isoquinolin-3ylmethyl)amino)ethyl)diphenylphosphine oxide



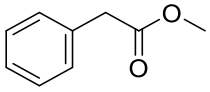
3.4.2 The title compounds were synthesized via the oxygen transfer amination procedure.

In a glove box, **L1.HCl** (1 g, 2.1 mmol) was dissolved in 3.0 ml of toluene solvent, and K_2CO_3 (48.1 mg, 1.26 mmol, 0.6 eq.) was added and stirred at 60 °C for 2 h. After cooling down to room temperature, picolinaldehyde or 2-quinolinecarboxaldehyde (225 mg or 330 mg, 2.1 mmol, 1eq.) was added under N_2 followed by stirring at 70 °C for 8 h. Then, 2M HCl solution (aq.) was added to the reaction mixture until no more bubbles were generated. Next, distilled water and anhydrous diethyl ether were added for extraction. The organic phase was combined, dried over anhydrous magnesium sulfate, and then filtered to remove the magnesium sulfate. After removing the ether by evaporation, anhydrous diethyl ether was added to wash the residue and give the desired product as white solid.

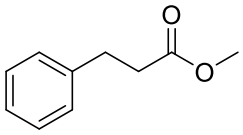
(2-((2-(diphenylphosphanyl)ethyl)(pyridin-2-ylmethyl)amino)ethyl)diphenylphosphine oxide, **L3**
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, J = 4.12 Hz, 1H), 7.67 (d, J = 7.28 Hz, 2H), 7.64 (d, J = 6.92 Hz, 2H), 7.52 (dd, J = 6.2, 13.9 Hz, 2H), 7.47 (d, J = 6.2 Hz, 2H), 7.44 - 7.40 (m, 4H), 7.36 - 7.32 (m, 4H), 7.28 - 7.26 (m, 6H), 7.10 (t, J = 5.94 Hz, 1H), 3.73 (s, 2H), 2.88 (dd, J = 7.4, 15.2 Hz, 2H), 2.62 (dd, J = 6.9, 17.5 Hz, 2H), 2.42 - 2.35 (m, 2H), 2.18 - 2.15 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 149.1, 138.6, 136.6, 133.7, 132.9, 131.8, 130.9, 128.9, 128.7, 128.6, 122.5, 59.7, 50.6(d,$J_{C,P}$ = 20.0 Hz), 46.2, 27.0(d,$J_{C,P}$ = 59.0 Hz), 26.7(d,$J_{C,P}$ = 3.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.13, -20.19 ppm.
HRMS: $C_{34}H_{34}N_2OP_2$ [M+H$^+$]; calculated: 549.2219, found: 549.2225.

(2-((2-(diphenylphosphanyl)ethyl)(quinolin-2-ylmethyl)amino)ethyl)diphenylphosphine oxide, **L4**
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (t, J = 8.0 Hz, 2H), 7.77 (d, J = 8.0 Hz, 1H), 7.69 (t, J = 7.1 Hz, 1H), 7.62 - 7.56 (m, 4H), 7.54 - 7.47 (m, 2H), 7.39 (t, J = 7.4 Hz, 2H), 7.34 - 7.27 (m, 8H), 7.22 - 7.20 (m, 6H), 3.90 (s, 2H), 2.95 - 2.85 (m, 2H), 2.67 - 2.66 (m, 2H), 2.42 - 2.39 (m, 2H), 2.24 - 2.15 (m, 2H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 160.0, 147.5, 138.3, 136.4, 133.4, 132.8, 131.72, 131.7, 130.7, 129.4, 129.1, 128.6, 128.61, 128.5, 128.5, 127.6, 127.5, 126.2, 121.2, 60.3, 49.9(d,$J_{C,P}$ = 21.0 Hz), 46.1, 26.3(d,$J_{C,P}$ = 68.0 Hz), 25.6(d,$J_{C,P}$ = 2.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.23 , -20.28 ppm.
HRMS: $C_{38}H_{36}N_2OP_2$ [M+H$^+$]; calculated: 599.2376, found: 599.2374.

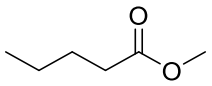
3.5 NMR data of products



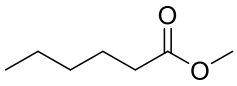
methyl 2-phenylacetate **(2a).**[4] Pale yellow oil; 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 - 7.25 (m, 5H), 3.65 (s, 3H), 3.60 (s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) δ 172.0, 134.1, 129.3, 128.6, 127.0, 52.0, 41.2.



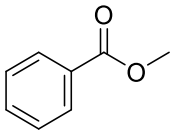
methyl 3-phenylpropanoate (**2b**).[5] Pale yellow oil; 89% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 - 7.22 (m, 5H), 3.68 (s, 3H), 2.97 (t, J = 7.8 Hz, 2H), 2.65 (t, J = 7.8 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.4, 140.5, 128.5, 128.3, 126.3, 51.6, 35.7, 31.0.



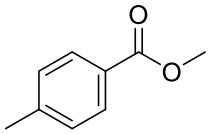
methyl pentanoate (**2d**).[6] Clear colorless liquid; 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.65 (s, 3H), 2.29 (t, J = 7.5 Hz, 2H), 1.65-1.52 (m, 2H), 1.40-1.26 (m, 2H), 0.90 (t, J = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.4, 51.5, 33.9, 27.2, 22.4, 13.8.



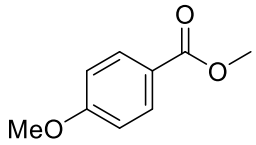
methyl hexanoate (**2e**).[6] Clear colorless liquid; 89% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.65 (s, 3H), 2.28 (t, J = 7.6 Hz, 2H), 1.66-1.53 (m, 2H), 1.37-1.21 (m, 4H), 0.88 (t, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.3, 51.4, 34.0, 31.3, 24.6, 22.3, 13.9.



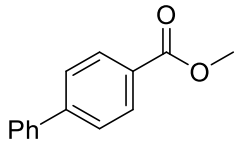
methyl benzoate (**2i**).[7] Clear colorless oil; 83% yield; 1H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 8.0 Hz, 2H), 7.55 (t, J = 7.4 Hz, 1H), 7.43 (t, J = 7.6 Hz, 2H), 3.91 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 133.0, 130.3, 129.7, 128.4, 52.2.



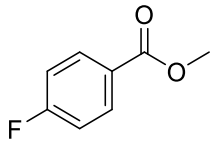
methyl 4-methylbenzoate (**2j**).[7] Pale yellow oil; 93% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.3 Hz, 2H), 7.19 (d, J = 7.9 Hz, 2H), 3.86 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 143.5, 129.6, 129.0, 127.5, 51.8, 21.5.



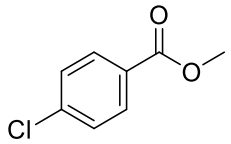
methyl 4-methoxybenzoate (**2k**).[7] Pale yellow oil; 90% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, J = 8.2 Hz, 2H), 6.90 (d, J = 8.2 Hz, 2H), 3.87 (s, 3H), 3.83 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9, 163.5, 131.7, 122.4, 113.7, 55.5, 51.9.



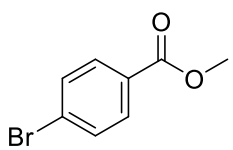
methyl [1,1'-biphenyl]-4-carboxylate (**2l**).[7] 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, J = 8.0 Hz, 2H), 7.68 – 7.62 (m, 4H), 7.49 – 7.38 (m, 3H), 3.95 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 145.8, 140.2, 130.2, 129.1, 128.3, 127.4, 127.2, 52.2.



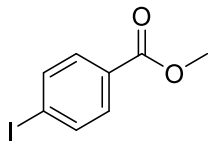
methyl 4-fluorobenzoate (**2m**).[7] Clear colorless liquid; 69% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.99 (m, 2H), 7.06 (t, J = 8.5 Hz, 2H), 3.87 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 165.3 (d, J = 158.7 Hz), 132.2 (d, J = 9.3 Hz), 126.5 (d, J = 3.1 Hz), 115.5 (d, J = 21.9 Hz), 52.2.



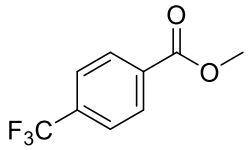
methyl 4-chlorobenzoate (**2n**).[8] Pale yellow oil; 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J = 8.5 Hz, 2H), 7.40 (d, J = 8.5 Hz, 2H), 3.91 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3, 139.5, 131.1, 128.83, 128.77, 52.4.



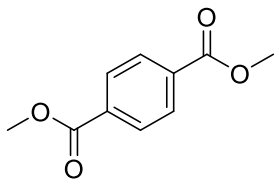
methyl 4-bromobenzoate (**2o**).[8] Pale yellow oil; 82% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 8.4 Hz, 2H), 7.56 (d, J = 8.4 Hz, 2H), 3.90 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 131.8, 131.2, 129.2, 128.1, 52.4.



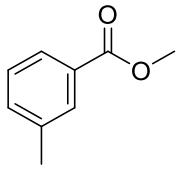
methyl 4-iodobenzoate (**2p**).[9] Pale yellow oil; 35% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.71 (m, 4H), 3.89 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 137.8, 131.1, 129.7, 100.8, 52.4.



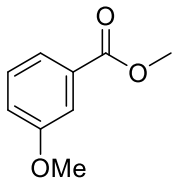
methyl 4-(trifluoromethyl)benzoate (**2q**).[7] Pale yellow oil; 61% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, J = 8.0 Hz, 2H), 7.67 (d, J = 8.0 Hz, 2H), 3.93 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.6, 133.3 (q, J = 32.7 Hz), 132.4 (m), 128.9, 124.2 (q, J = 3.7 Hz), 123.5 (q, J = 272.6 Hz), 51.6.



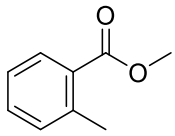
dimethyl terephthalate (**2r**).[7] White powder; 58% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (s, 4H), 3.92 (s, 6H).^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3, 134.0, 129.6, 52.5.



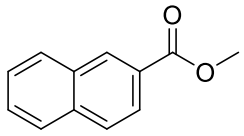
methyl 3-methylbenzoate (**2s**).[8] Clear colorless liquid; 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (t, J = 5.9 Hz, 2H), 7.35 – 7.28 (m, 2H), 3.89 (s, 3H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 138.1, 133.7, 130.1, 128.3, 126.7, 52.0, 21.2.



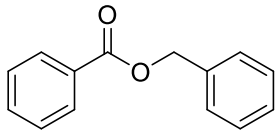
methyl 3-methoxybenzoate (**2t**).[8] Pale yellow oil; 81% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.55 (m, 2H), 7.33 (t, J = 7.9 Hz, 1H), 7.08 (d, J = 7.4 Hz, 1H), 3.90 (s, 3H), 3.83 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 159.6, 131.5, 129.4, 122.0, 119.50, 114.1, 55.4, 52.2.



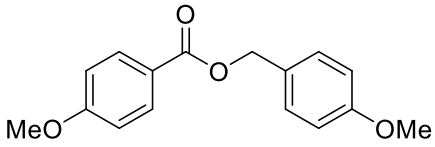
methyl 2-methylbenzoate (**2u**).[8] Pale yellow oil; 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.3 Hz, 1H), 7.36 (t, J = 7.5 Hz, 1H), 7.21 (d, J = 4.0 Hz, 2H), 3.86 (s, 3H), 2.59 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.1, 140.2, 1320, 131.7, 130.6, 129.6, 125.7, 51.8, 21.7.



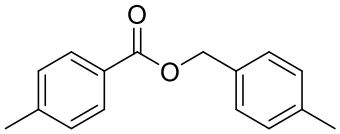
methyl 2-naphthoate (**2v**).[8] Pale yellow oil; 82% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (s, 1H), 8.07 (d, J = 8.6 Hz, 1H), 7.95 (d, J = 8.0 Hz, 1H), 7.88 (d, J = 8.6 Hz, 2H), 7.61 – 7.52 (m, 2H), 3.99 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 135.7, 132.7, 131.2, 129.5, 128.4, 128.3, 127.9, 127.6, 126.8, 125.4, 52.3.



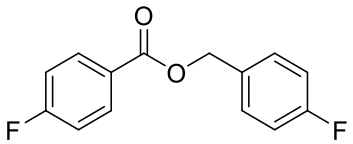
benzyl benzoate (**2y**).[10] Pale yellow oil; 85% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, J = 7.9 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.46 – 7.34 (m, 7H), 5.39 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 136.1, 133.16, 130.18, 129.8, 128.7, 128.5, 128.4, 128.3, 66.8.



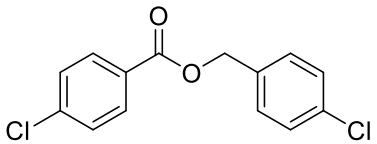
4-methoxybenzyl 4-methoxybenzoate (**2z**).[11] Pale yellow oil; 94% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 8.9 Hz, 2H), 7.38 (d, J = 8.6 Hz, 2H), 6.92 – 6.89 (m, 4H), 5.27 (s, 2H), 3.85 (s, 3H), 3.82 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 163.5, 159.8, 131.8, 130.1, 128.6, 122.9, 114.1, 113.7, 66.4, 55.5, 55.4.



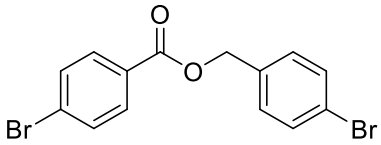
4-methylbenzyl 4-methylbenzoate (**2aa**).[12] Pale yellow oil; 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 7.4 Hz, 2H), 7.33 (d, J = 7.8 Hz, 2H), 7.21 – 7.17 (m, 2H), 5.30 (s, 2H), 2.38 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.7, 143.7, 138.1, 133.3, 129.8, 129.2, 128.4, 127.6, 66.6, 21.7, 21.3.



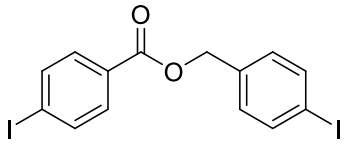
4-fluorobenzyl 4-fluorobenzoate (**2ab**).[11] Pale yellow oil; 78% yield; 1H NMR (400 MHz, $CDCl_3$) δ 8.09 – 8.05 (m, 2H), 7.44 – 7.40 (m, 2H), 7.12 – 7.04 (m, 4H), 5.31 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 164.8 (d, J = 144.3 Hz), 163.2 (d, J = 310.4 Hz), 132.4 (d, J = 9.2 Hz), 131.9, 130.4 (d, J = 8.2 Hz), 126.4, 115.7 (d, J = 21.7 Hz), 66.3.



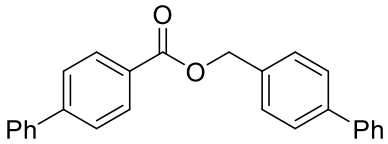
4-chlorobenzyl 4-chlorobenzoate (**2ac**).[11] White powder; 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 6.4 Hz, 2H), 7.30 (d, J = 6.4 Hz, 8H), 5.21 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.6, 139.8, 134.5, 131.2, 129.8, 129.0, 128.9, 128.6, 66.3.



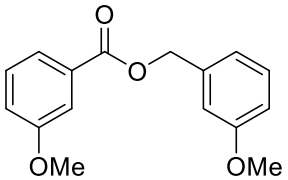
4-bromobenzyl 4-bromobenzoate (**2ad**).[12] White powder; 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.4 Hz, 2H), 7.58 (d, J = 8.4 Hz, 2H), 7.52 (d, J = 8.1 Hz, 2H), 7.31 (d, J = 8.1 Hz, 2H), 5.30 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 134.96, 131.99, 131.96, 131.4, 130.1, 129.0, 128.5, 122.6, 66.3.



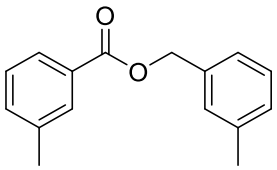
4-iodobenzyl 4-iodobenzoate (**2ae**).[12] White powder; 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.71 (m, 6H), 7.18 (d, J = 7.4 Hz, 2H), 5.28 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.0, 137.97, 137.96, 135.6, 131.3, 130.2, 129.6, 101.1, 94.2, 66.4.



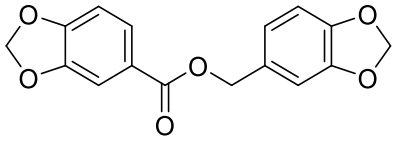
[1,1'-biphenyl]-4-ylmethyl [1,1'-biphenyl]-4-carboxylate (**2af**).[11] White powder; 83% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, J = 7.8 Hz, 2H), 7.69 – 7.61 (m, 8H), 7.56 (d, J = 7.1 Hz, 2H), 7.49 – 7.35 (m, 6H), 5.45 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 146.0, 141.4, 140.9, 140.2, 135.3, 130.4, 129.1, 129.0, 128.8, 128.3, 127.6, 127.5, 127.4, 127.3, 127.2, 66.6.



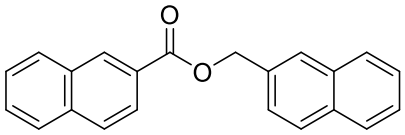
3-methoxybenzyl 3-methoxybenzoate (**2ag**).[11] Pale yellow oil; 82% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, J = 7.6 Hz, 1H), 7.61 (s, 2H), 7.37 – 7.29 (m, 2H), 7.11 (d, J = 9.2 Hz, 1H), 7.04 – 6.99 (m, 2H), 6.89 (d, J = 7.9 Hz, 2H), 5.34 (s, 2H), 3.84 (d, J = 9.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 159.9, 159.73, 137.72, 131.6, 129.8, 129.5, 122.3, 120.4, 119.7, 114.39, 114.38, 113.8, 66.8, 55.6, 55.4.



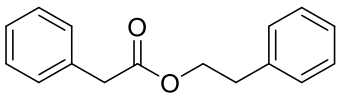
3-methylbenzyl 3-methylbenzoate (**2ah**).[11] Pale yellow oil; 79% yield; ^{1}H NMR (400 MHz, $CDCl_3$) 7.37-7.16 (m, 5H), 3.71 (q, J = 7.2 Hz, 1H), 1.49 (d, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.3, 139.8, 128.8, 127.7, 127.5, 45.5, 18.2.



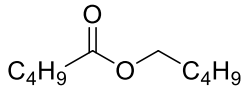
benzo[d][1,3]dioxol-5-ylmethyl benzo[d][1,3]dioxole-5-carboxylate (**2ak**).[11] White powder; 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 – 7.65 (m, 1H), 7.48 (t, J = 1.7 Hz, 1H), 6.93 – 6.90 (m, 2H), 6.82 (td, J = 8.3, 1.4 Hz, 2H), 5.99 (d, J = 23.5 Hz, 4H), 5.22 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 151.8, 148.0, 147.9, 147.8, 130.1, 125.6, 124.4, 122.3, 109.7, 109.1, 108.4, 108.1, 101.9, 101.3, 66.7.



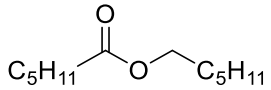
naphthalen-2-ylmethyl 2-naphthoate (**2al**).[11] White powder; 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$) 7.37-7.16 (m, 5H), 3.71 (q, J = 7.2 Hz, 1H), 1.49 (d, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.3, 139.8, 128.8, 127.7, 127.5, 45.5, 18.2.



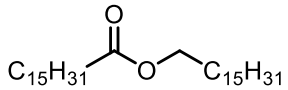
phenethyl 2-phenylacetate (**2am**).[10] Colourless oil; 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.18 (m, 8H), 7.13 (d, J = 7.4 Hz, 2H), 4.29 (t, J = 6.9 Hz, 2H), 3.58 (s, 2H), 2.89 (t, J = 7.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 137.8, 134.1, 129.4, 129.0, 128.6, 128.6, 127.1, 126.6, 65.4, 41.5, 35.1.



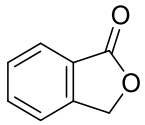
pentyl pentanoate (**2ap**).[11] Clear colorless liquid; 88% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.04 (t, J = 6.4 Hz, 2H), 2.28 (t, J = 7.7 Hz, 2H), 1.62 – 1.57 (m, 4H), 1.36 – 1.30 (m, 6H), 0.92 – 0.87 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 64.5, 34.2, 28.5, 28.2, 27.2, 22.43, 22.39, 14.1, 13.8.



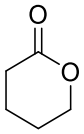
hexyl hexanoate (**2aq**).[11] Clear colorless liquid; 93% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.04 (t, J = 6.8 Hz, 2H), 2.27 (t, J = 7.5 Hz, 2H), 1.64 – 1.56 (m, 4H), 1.29 (d, J = 3.2 Hz, 10H), 0.89 – 0.86 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 64.5, 34.5, 31.6, 31.5, 28.8, 25.7, 24.8, 22.7, 22.4, 14.1, 14.0.



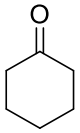
hexadecyl palmitate (**2ar**).[13] 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.05 (t, J = 6.7 Hz, 2H), 2.28 (t, J = 7.5 Hz, 2H), 1.26 (s, 54H), 0.88 (t, J = 6.6 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 64.5, 34.6, 32.1, 29.84, 29.81, 29.76, 29.73, 29.69, 29.6, 29.5, 29.4, 29.3 28.8, 26.1, 25.2, 22.8, 14.2.



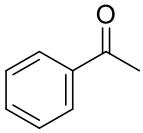
isobenzofuran-1(3H)-one (**2as**).[11] 95% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 7.6 Hz, 1H), 7.67 (t, J = 7.3 Hz, 1H), 7.53 – 7.48 (m, 2H), 5.31 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 146.6, 134.1, 129.1, 125.8, 122.2, 69.7.



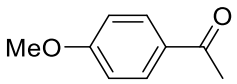
δ-Valerolactone (**2at**).[14] Clear colorless liquid; 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 4.27 (t, *J* = 5.7 Hz, 2H), 2.48 (t, *J* = 6.7 Hz, 2H), 1.87 – 1.75 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 171.4, 69.4, 29.8, 22.2, 19.0.



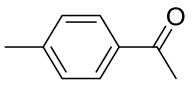
Cyclohexanone (**3a**).[14] Clear colorless liquid; 65% yield; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 2.27 – 2.23 (m, 3H), 1.77 – 1.63 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 212.0, 41.9, 27.0, 25.0.



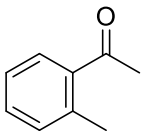
Acetophenone (**3b**).[14] Clear colorless liquid; 81% yield; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.96 – 7.94 (m, 2H), 7.57 – 7.53 (m, 1H), 7.45 (t, *J* = 6.7 Hz, 2H), 2.58 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 198.1, 137.2, 133.1, 128.6, 128.3, 26.6.



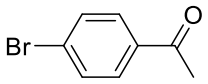
1-(4-methoxyphenyl)ethan-1-one (**3c**).[14] Crystalline Powder; 89% yield; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.92 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H), 2.54 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 196.9, 163.6, 130.7, 130.5, 113.8, 55.6, 26.4.



1-(p-tolyl)ethan-1-one (**3d**).[14] Pale yellow liquid; 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.85 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 2.57 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 197.8, 143.8, 134.8, 129.2, 128.4, 26.5, 21.6.



1-(o-tolyl)ethan-1-one (**3e**).[14] Pale yellow liquid; 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (t, J = 7.7 Hz, 1H), 7.35 (t, J = 7.7 Hz, 1H), 7.27 – 7.20 (m, 2H), 2.55 (s, 3H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.5, 138.3, 137.6, 132.0, 131.4, 129.3, 125.6, 29.4, 21.5.



1-(4-bromophenyl)ethan-1-one (**3f**).[14] Crystalline flakes; 79% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 8.6 Hz, 2H), 7.59 (d, J = 8.6 Hz, 2H), 2.57 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.1, 136.0, 132.0, 123.0, 128.4, 26.6.

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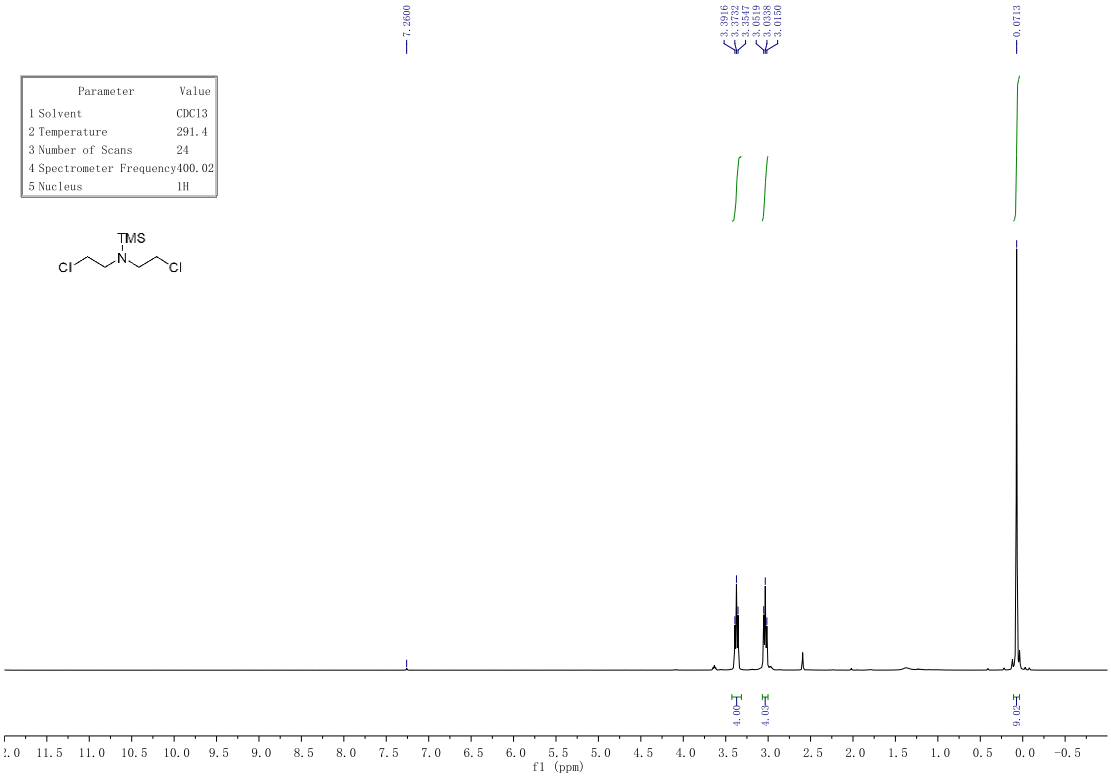
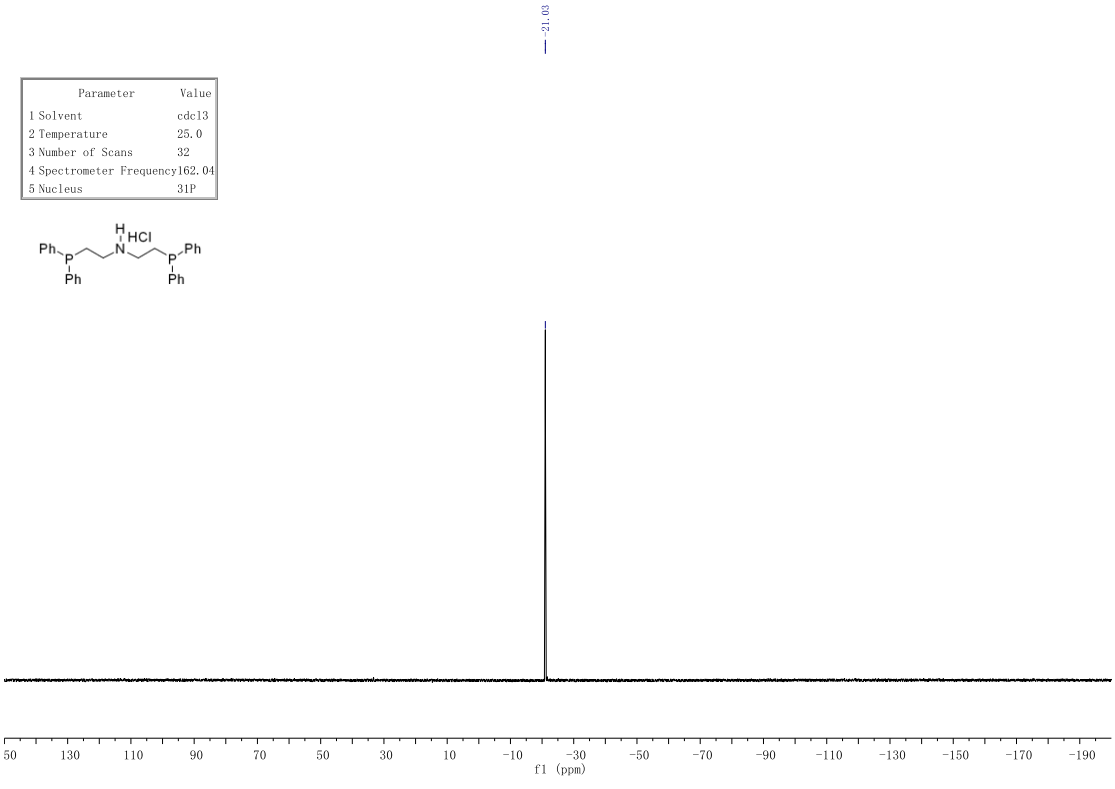
5. Copies of product NMR

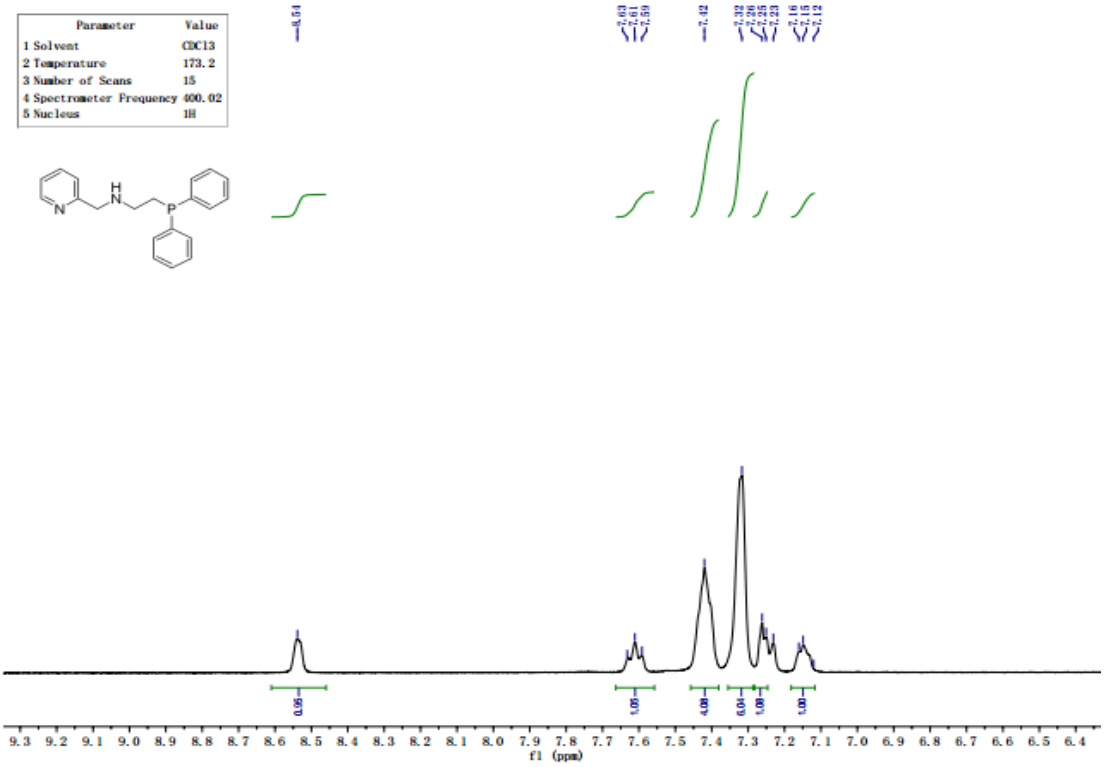
^{1}H NMR for N,N-bis(2-chloroethyl)-1,1,1-trimethylsilanamine



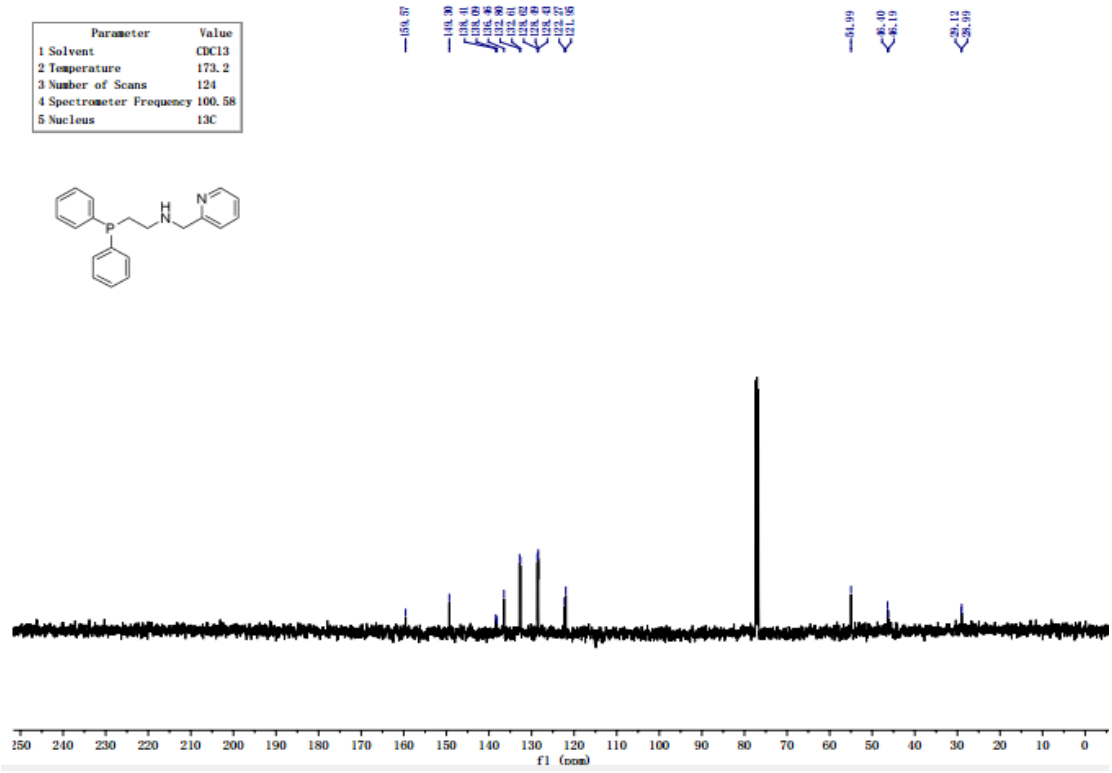
^{31}P NMR for bis(2-(diphenylphosphanyl)ethyl)amine



^{1}H NMR for 2-(Diphenylphosphaneyl)-N-(pyridin-2-ylmethyl)ethan-1-amine, **L2**

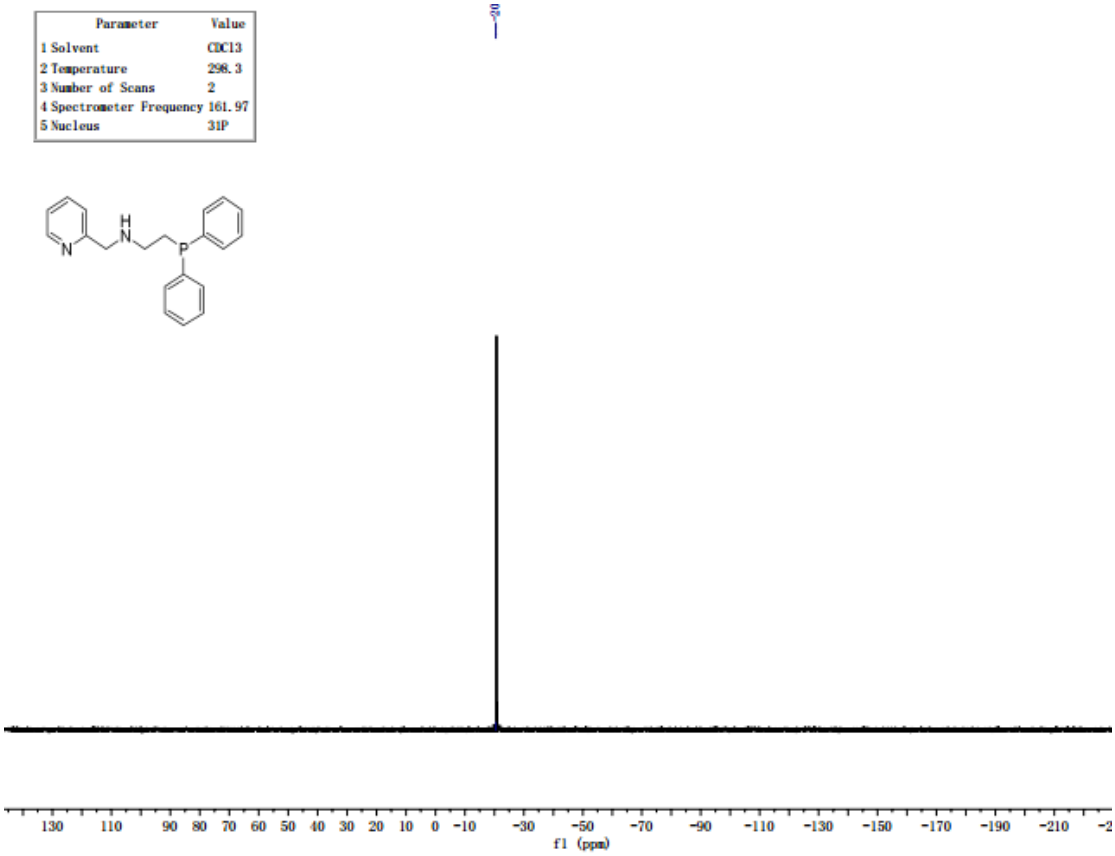


^{13}C NMR for 2-(Diphenylphosphaneyl)-N-(pyridin-2-ylmethyl)ethan-1-amine, **L2**

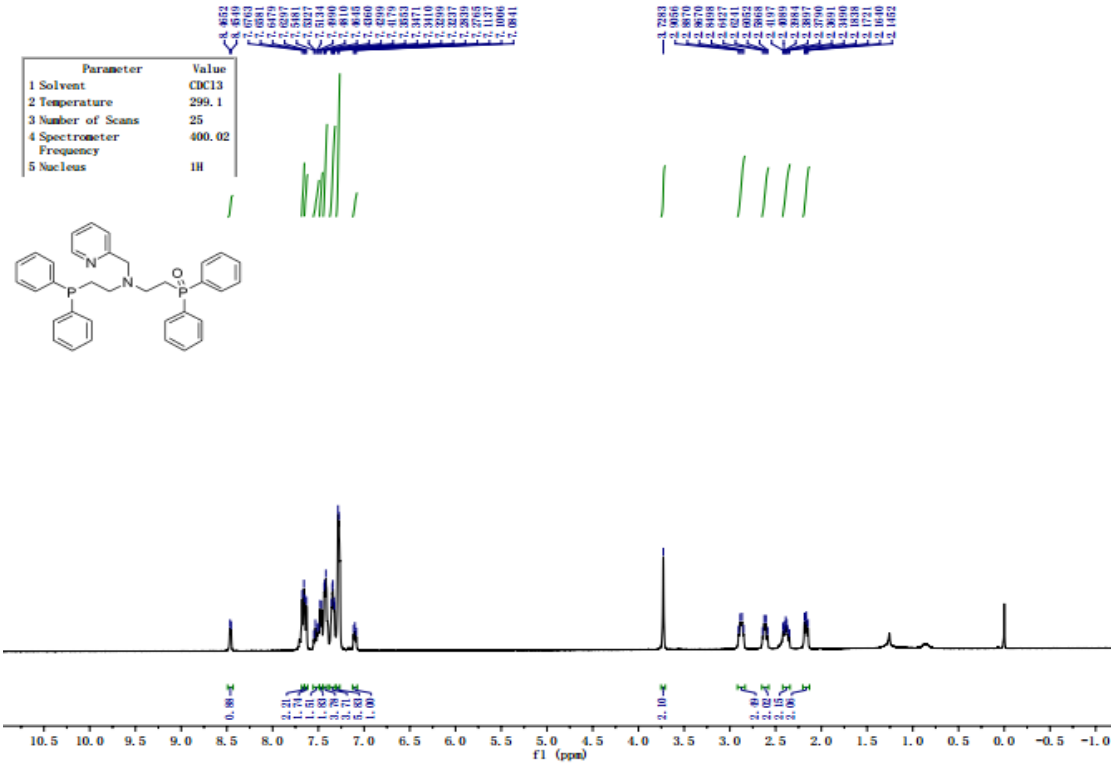


^{31}P NMR for 2-(Diphenylphosphaneyl)-N-(pyridin-2-ylmethyl)ethan-1-amine, **L2**

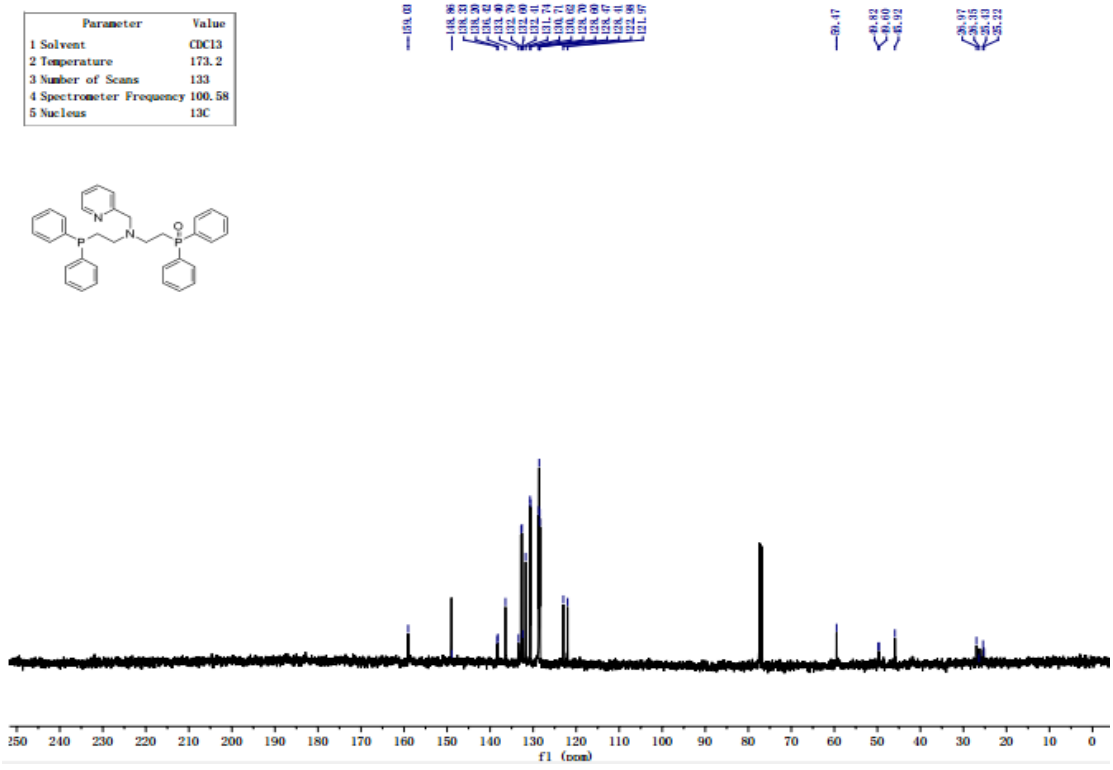
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.3
3 Number of Scans	2
4 Spectrometer Frequency	161.97
5 Nucleus	31P



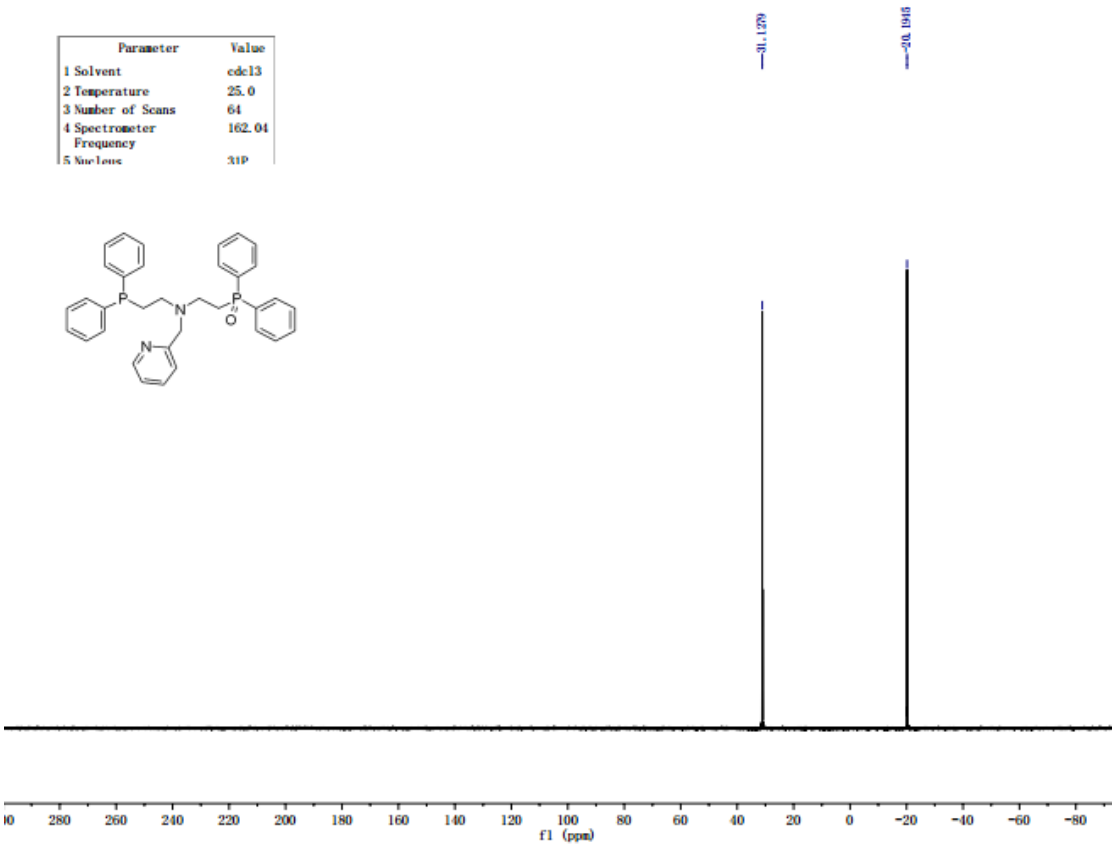
^{1}H NMR for (2-((2-(diphenylphosphanyl)ethyl)(pyridin-2-ylmethyl)amino)ethyl) diphenyl phosphine oxide, **L3**



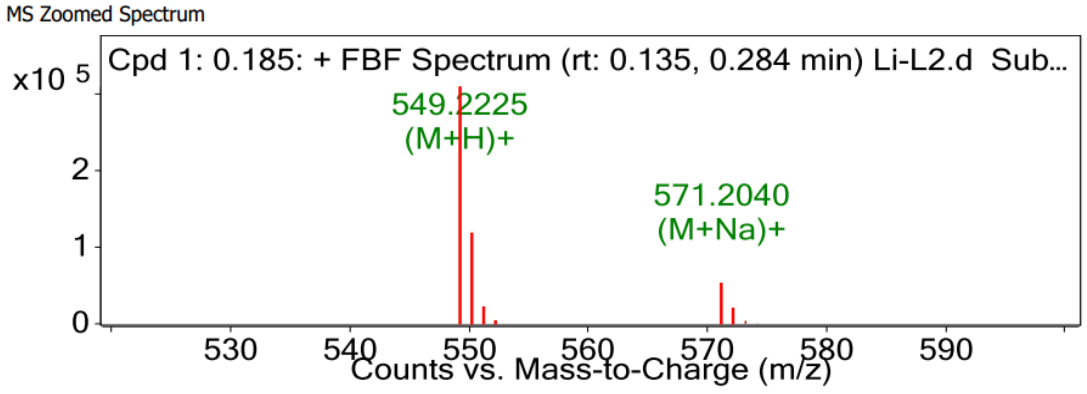
^{13}C NMR for (2-((2-(diphenylphosphanyl)ethyl)(pyridin-2-ylmethyl)amino)ethyl) diphenylphosphine oxide, **L3**



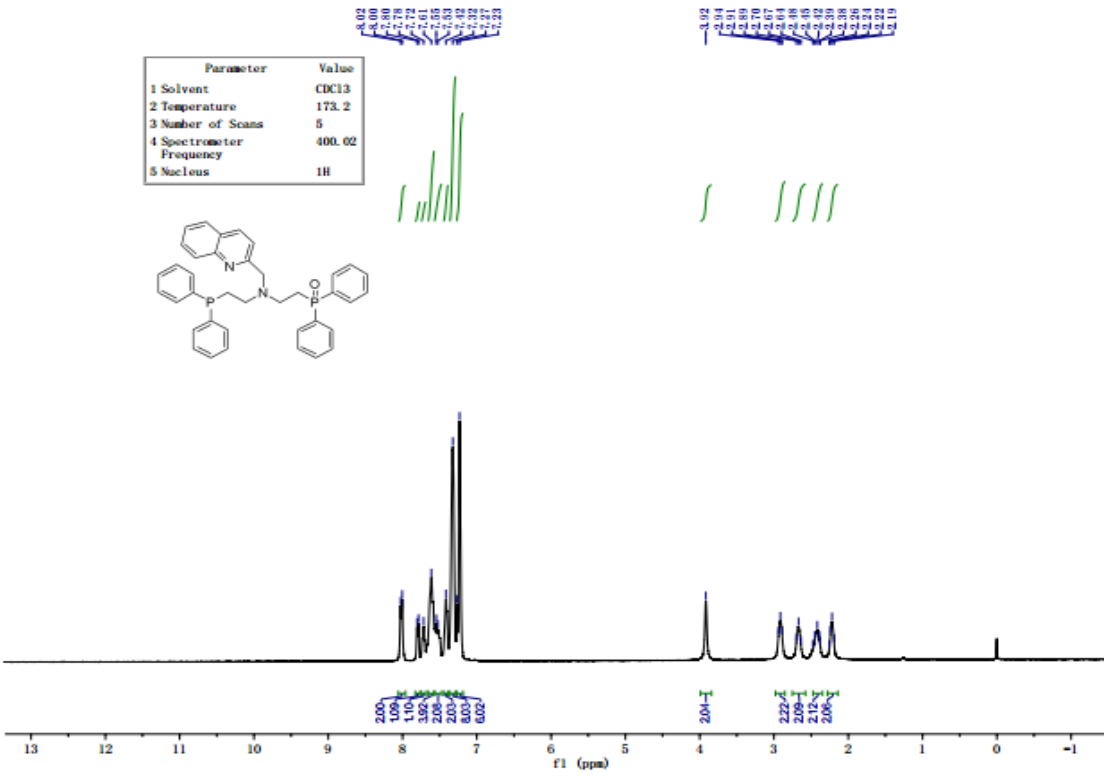
^{31}P NMR for (2-((2-(diphenylphosphanyl)ethyl)(pyridin-2-ylmethyl)amino)ethyl) diphenylphosphine oxide, **L3**



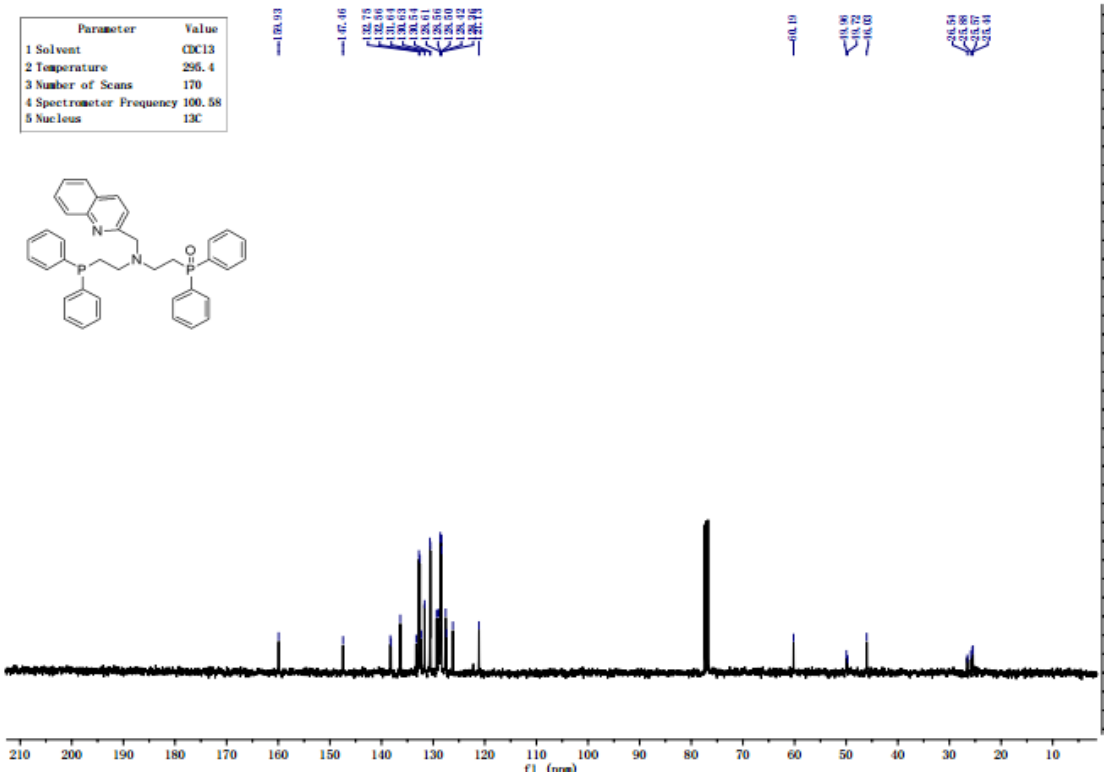
HRMS for NMR for (2-((2-(diphenylphosphanyl)ethyl)(pyridin-ylmethyl)amino)ethyl) diphenylphosphine oxide, **L3**



^{1}H NMR for (2-((2-(diphenylphosphanyl)ethyl)(isoquinolin-3-ylmethyl)amino)ethyl) diphenyl-phosphine oxide, **L4**



^{13}C NMR for (2-((2-(diphenylphosphanyl)ethyl)(isoquinolin-3-ylmethyl)amino)ethyl) diphenyl-phosphine oxide, **L4**



^{31}P NMR for (2-((2-(diphenylphosphanyl)ethyl)(isoquinolin-3-ylmethyl)amino)ethyl) diphenyl-phosphine oxide, **L4**

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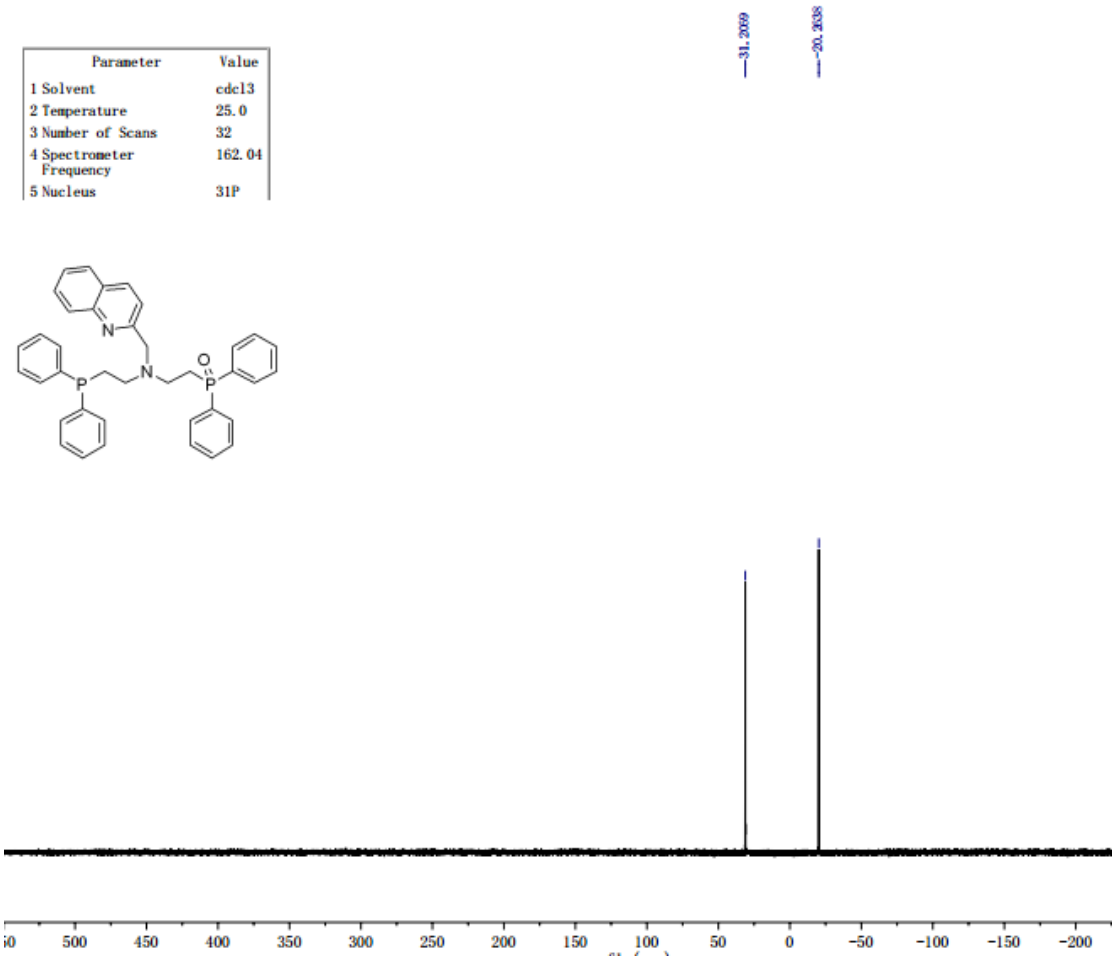
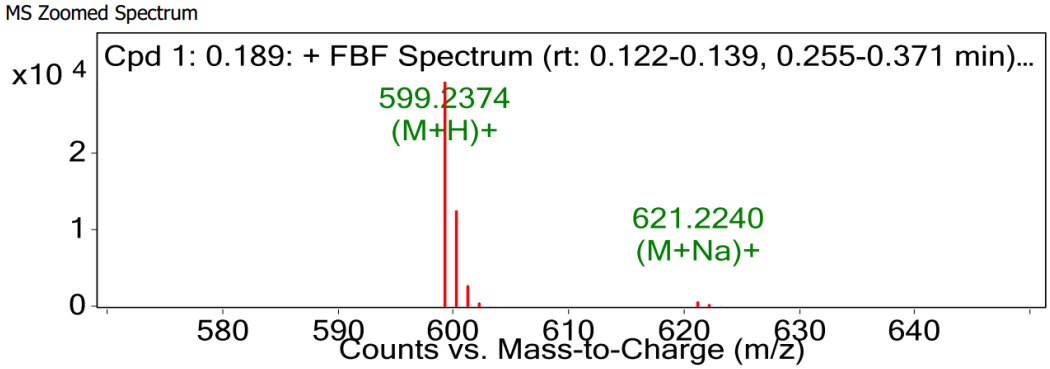
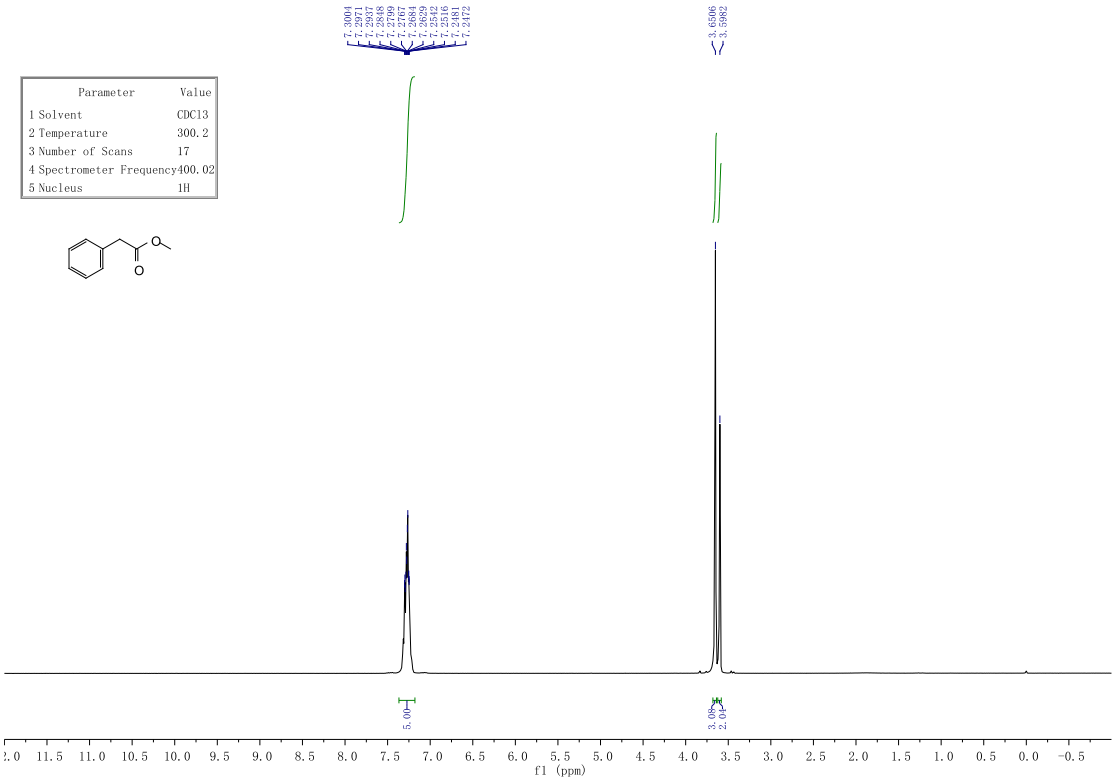
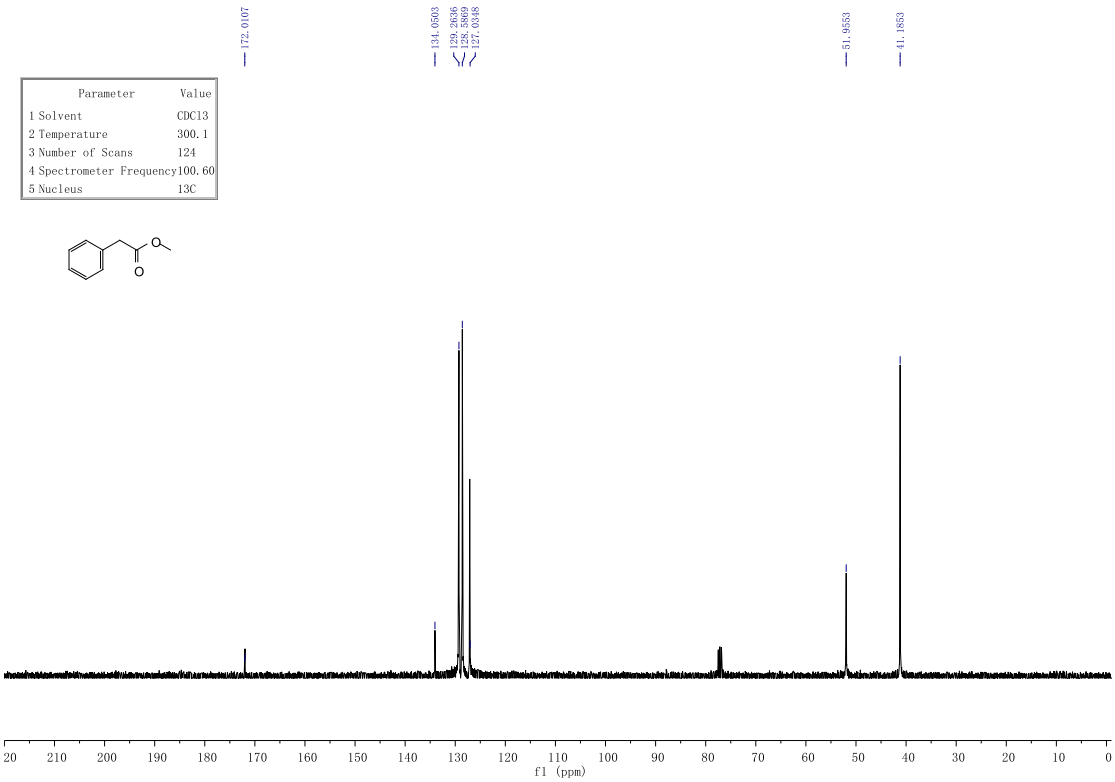


HRMS for (2-((2-(diphenylphosphanyl)ethyl)(isoquinolin-3-ylmethyl)amino)ethyl) diphenyl-phosphine oxide, **L4**

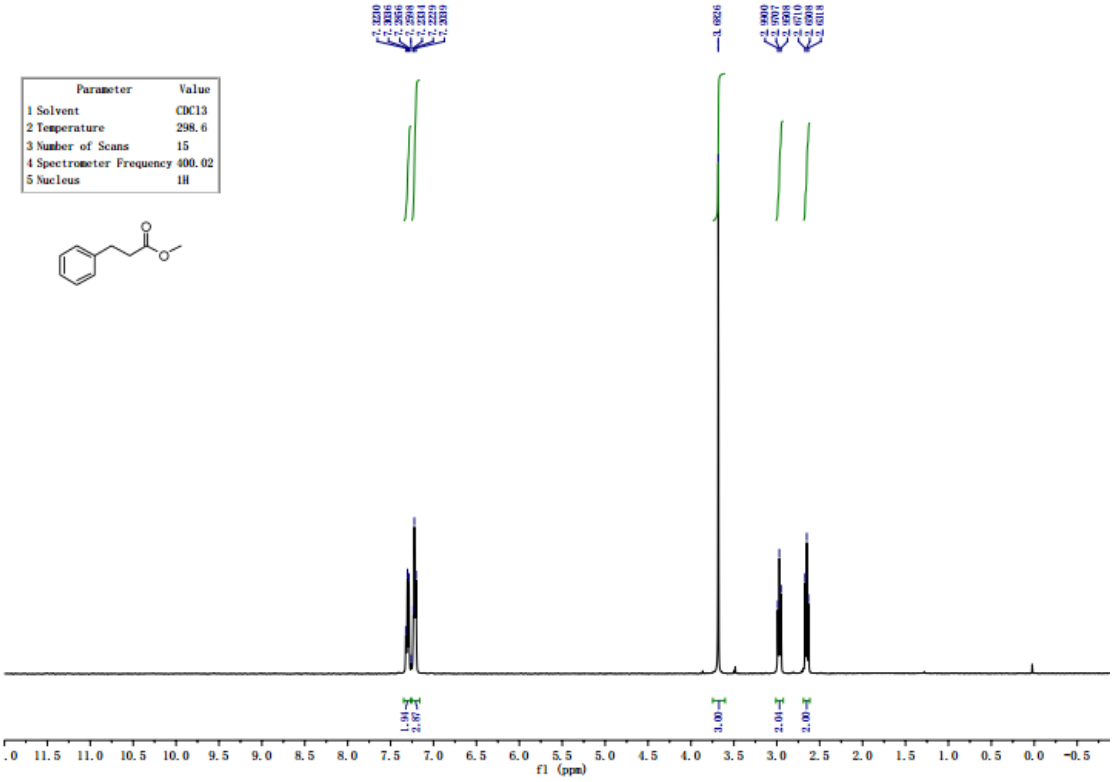


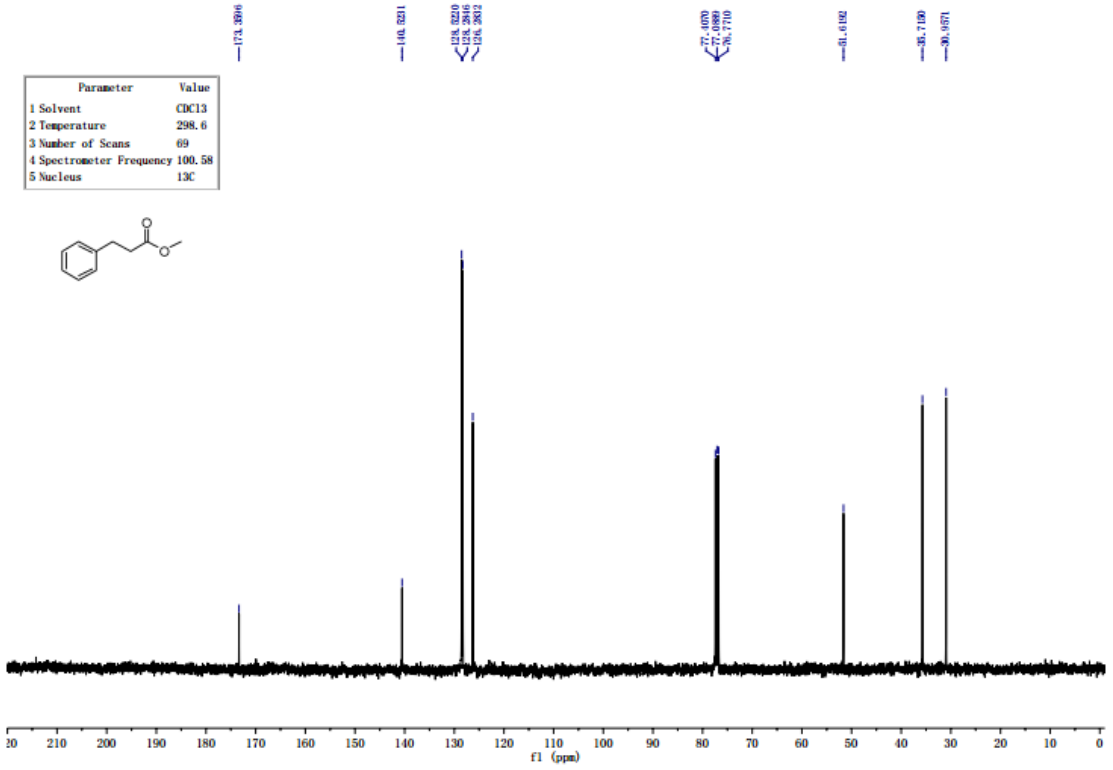
methyl 2-phenylacetate, **2a**



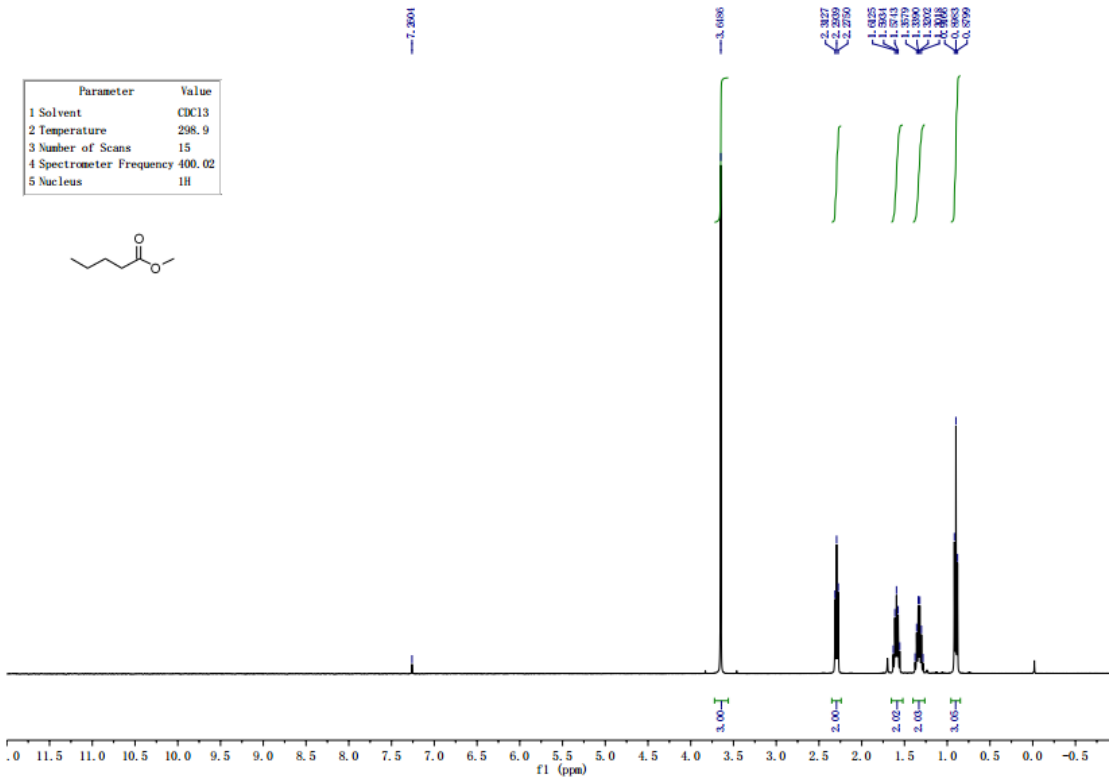


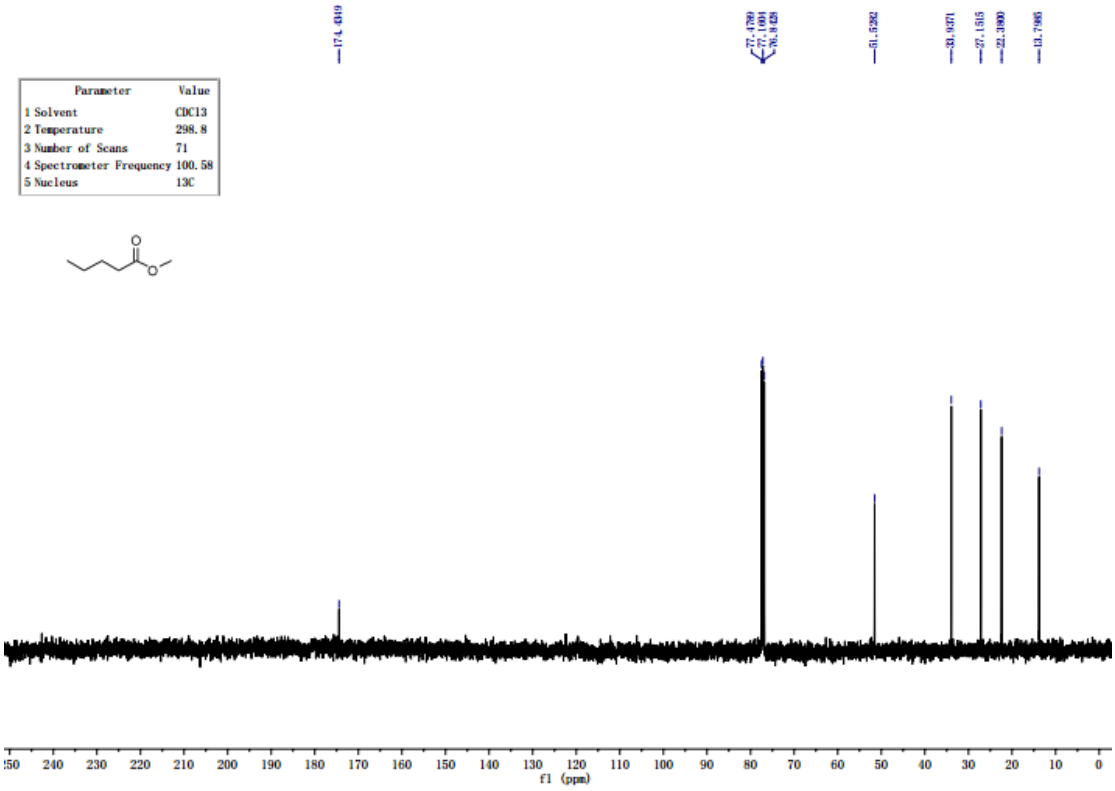
methyl 3-phenylpropanoate, **2b**



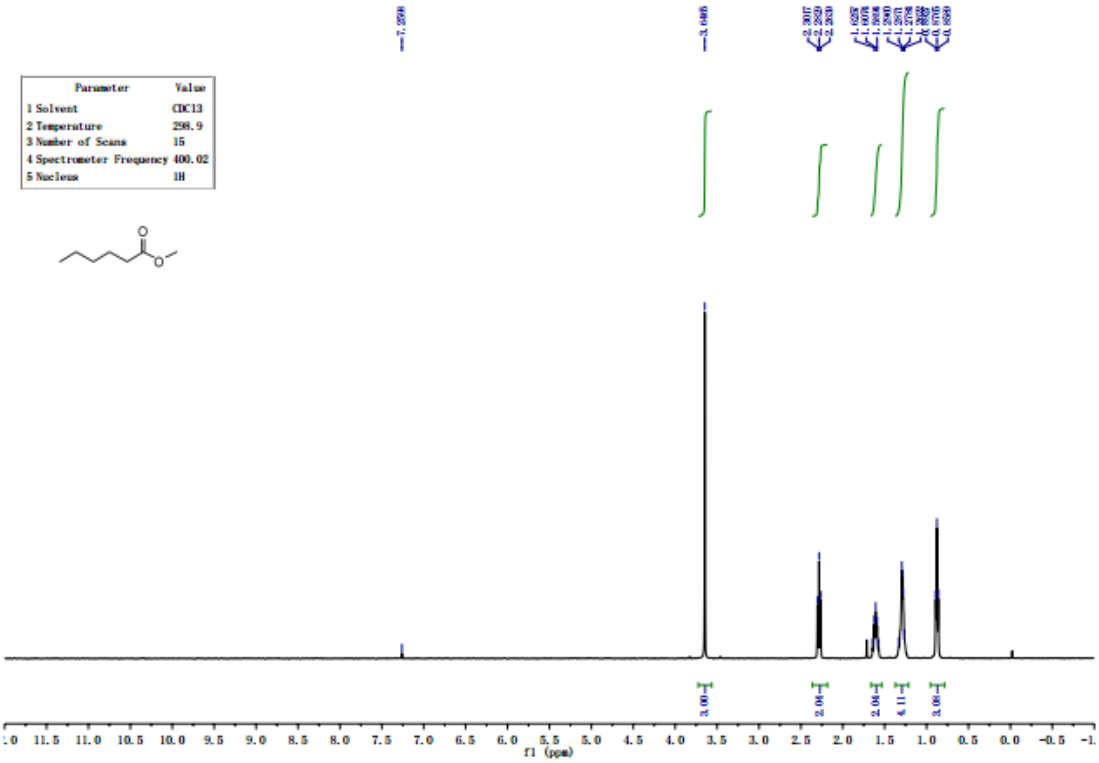


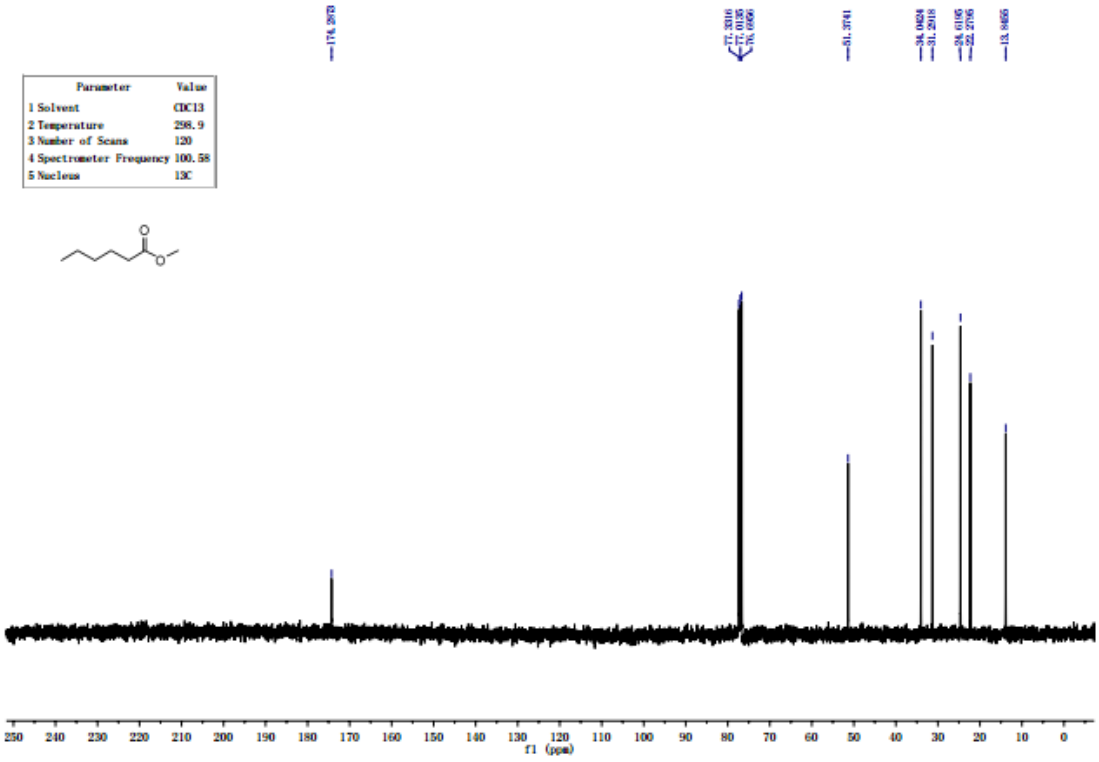
methyl pentanoate, **2d**



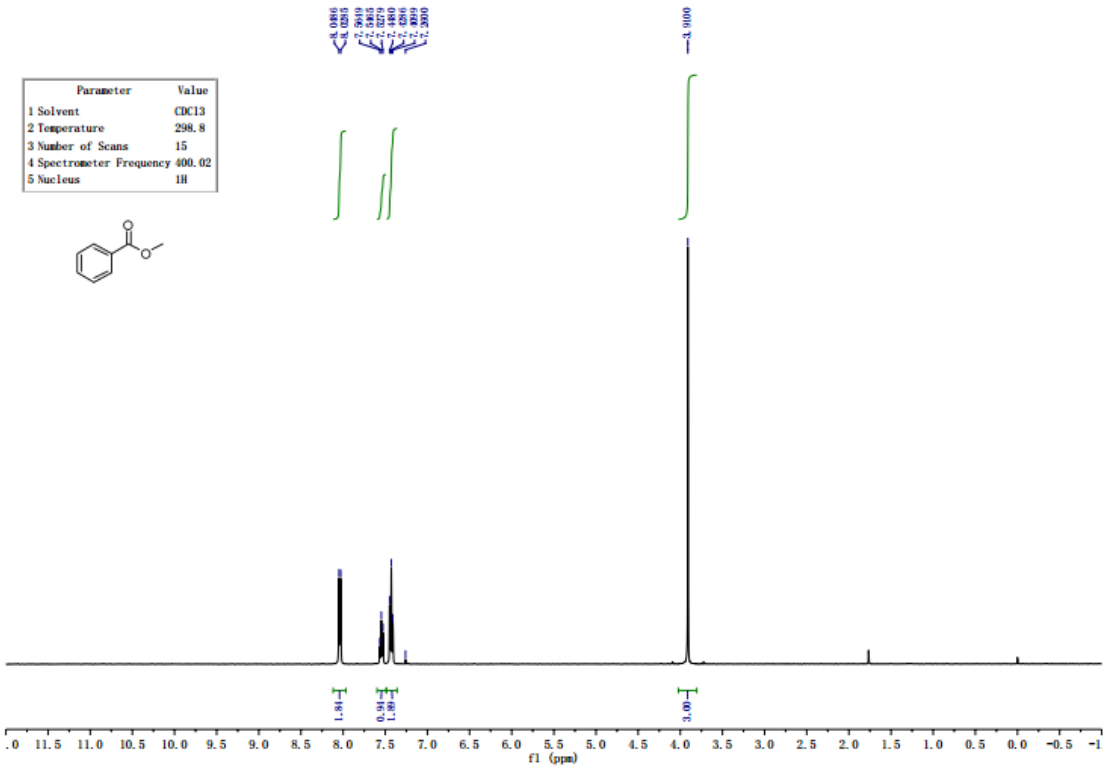


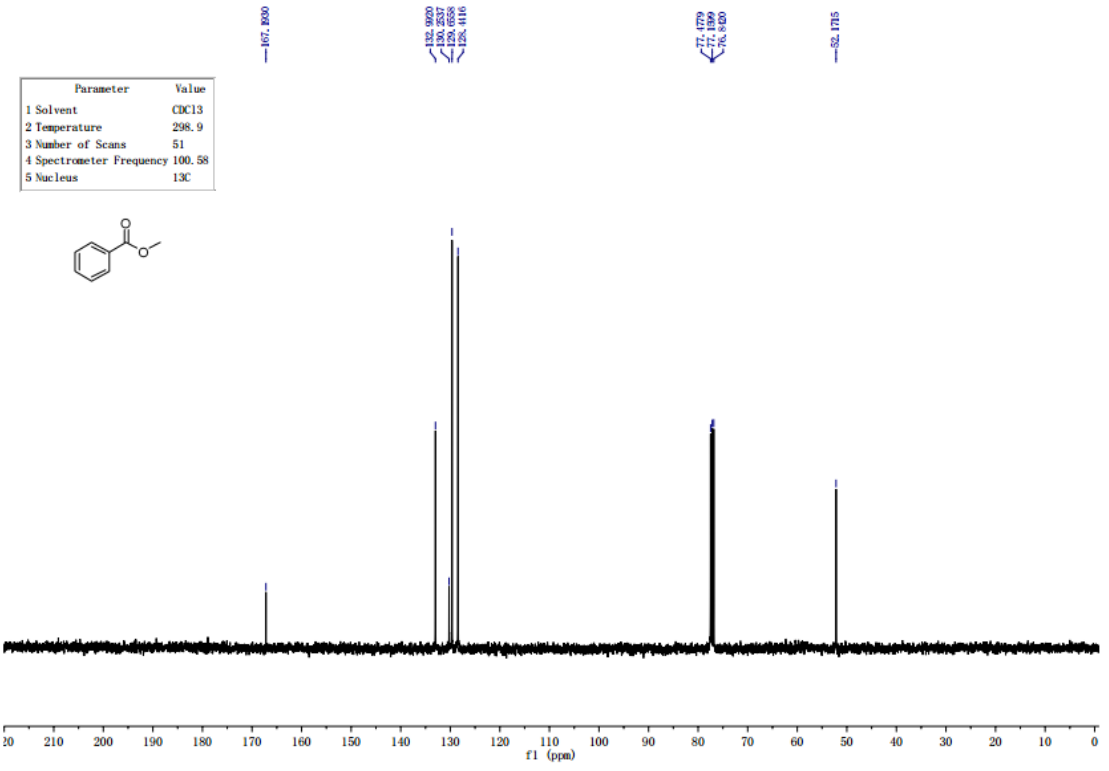
methyl hexanoate, **2e**



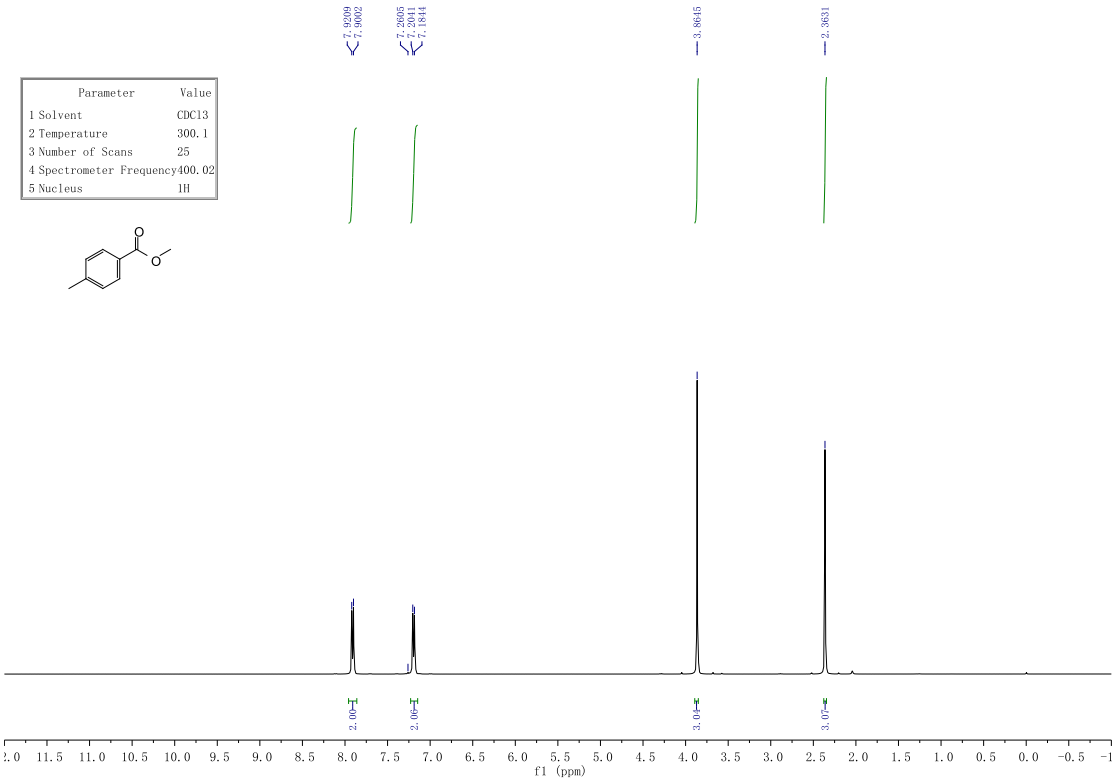


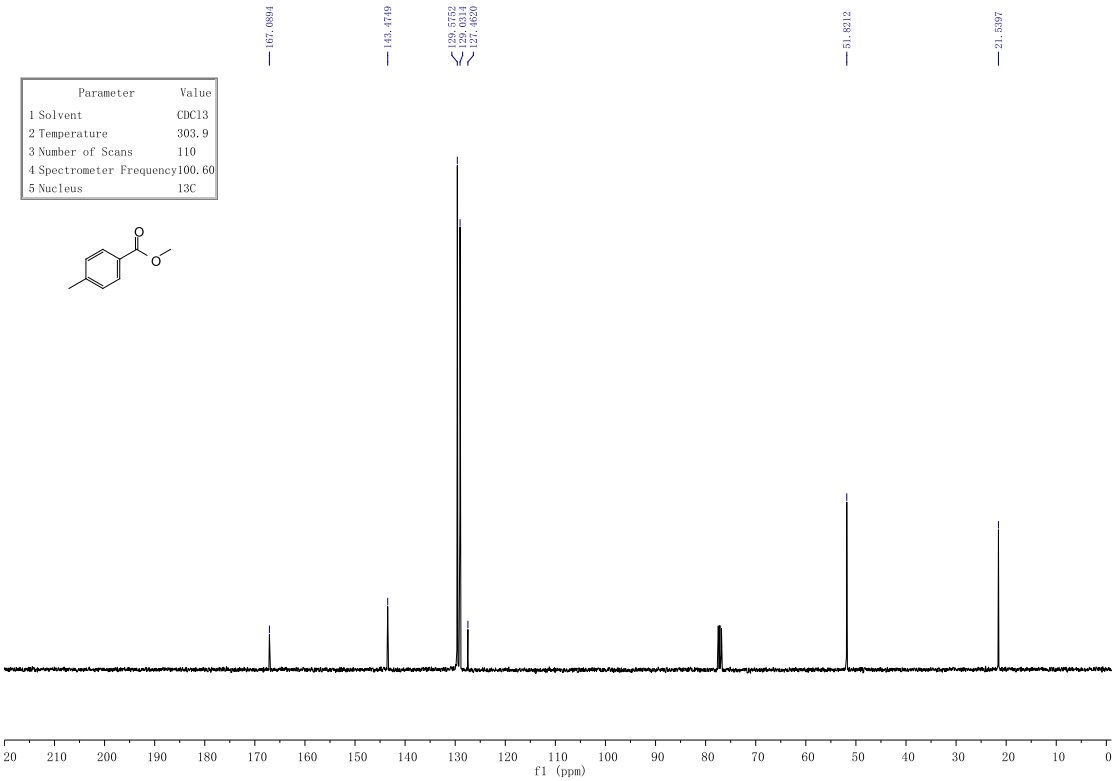
methyl benzoate, **2i**



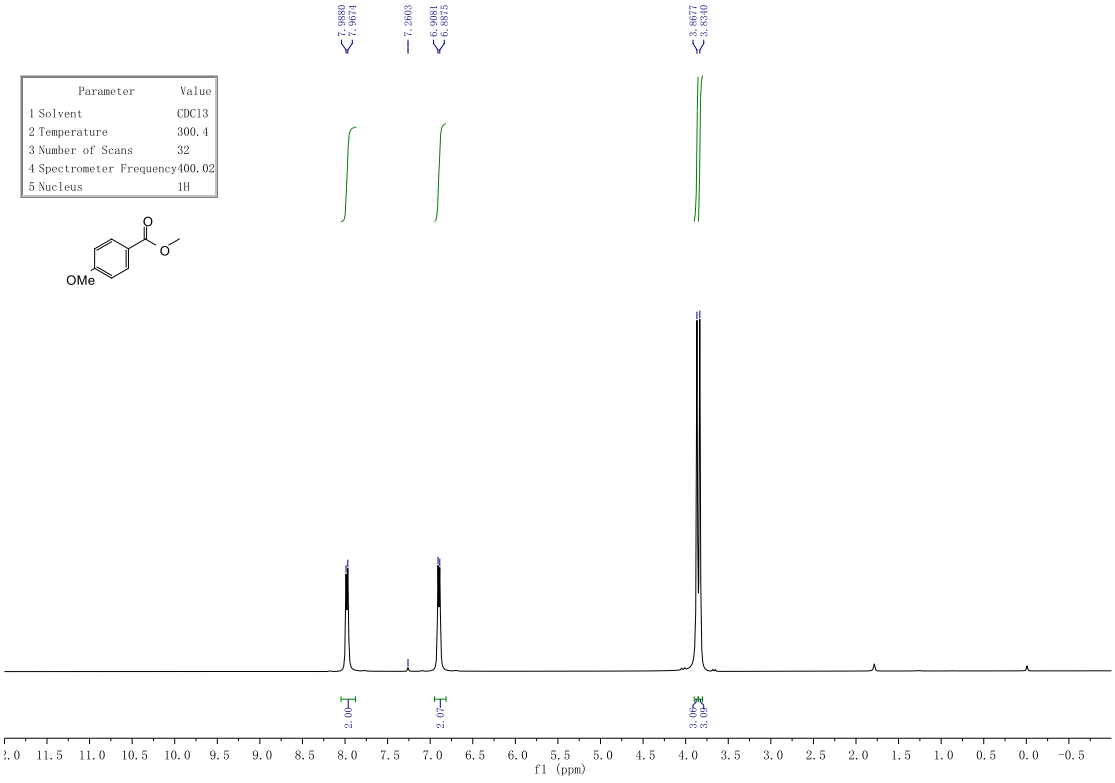


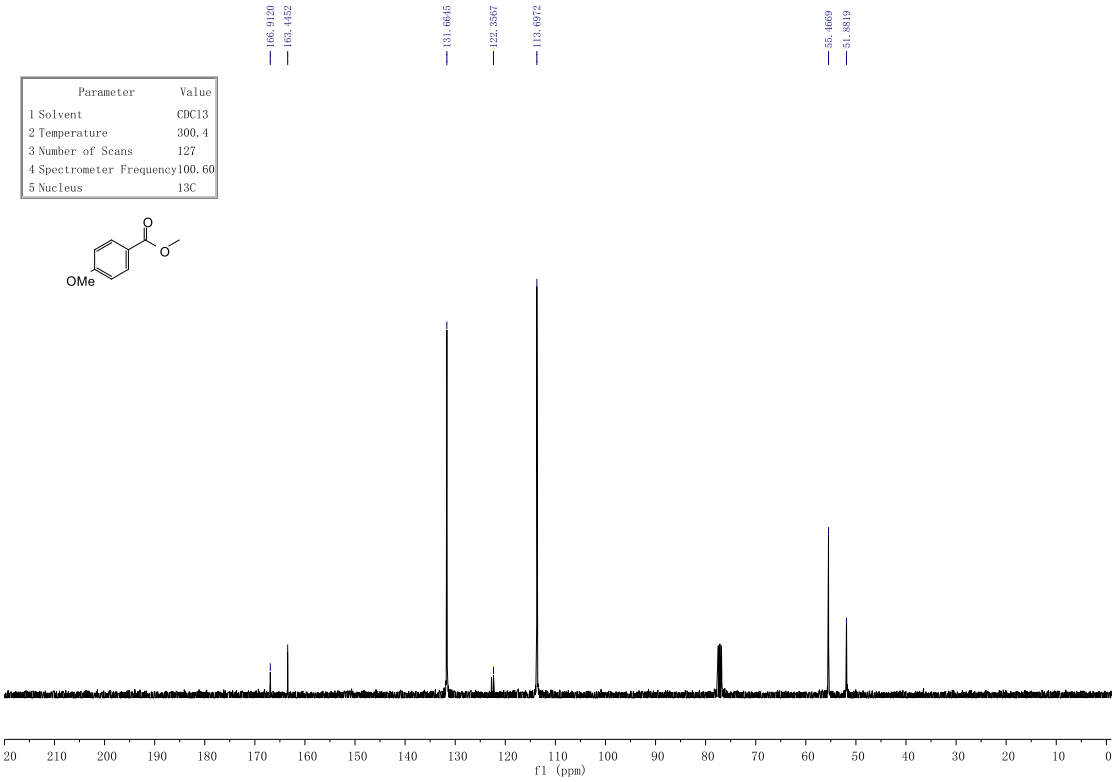
methyl 4-methylbenzoate, **2j**



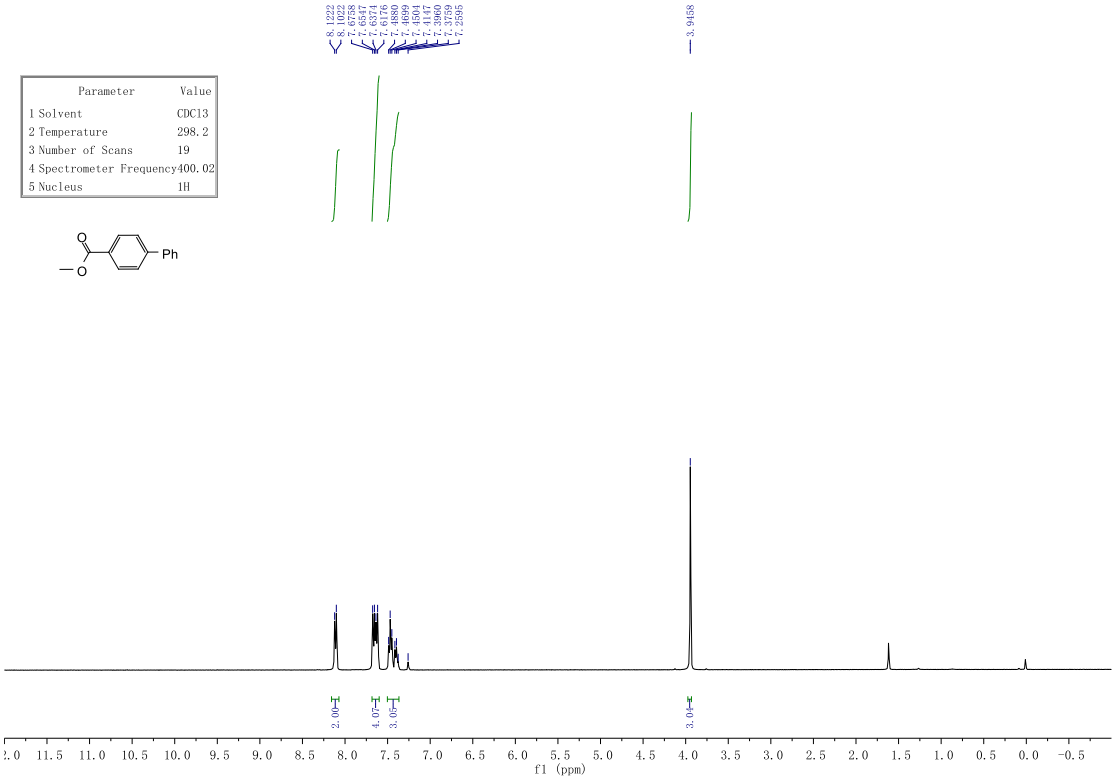


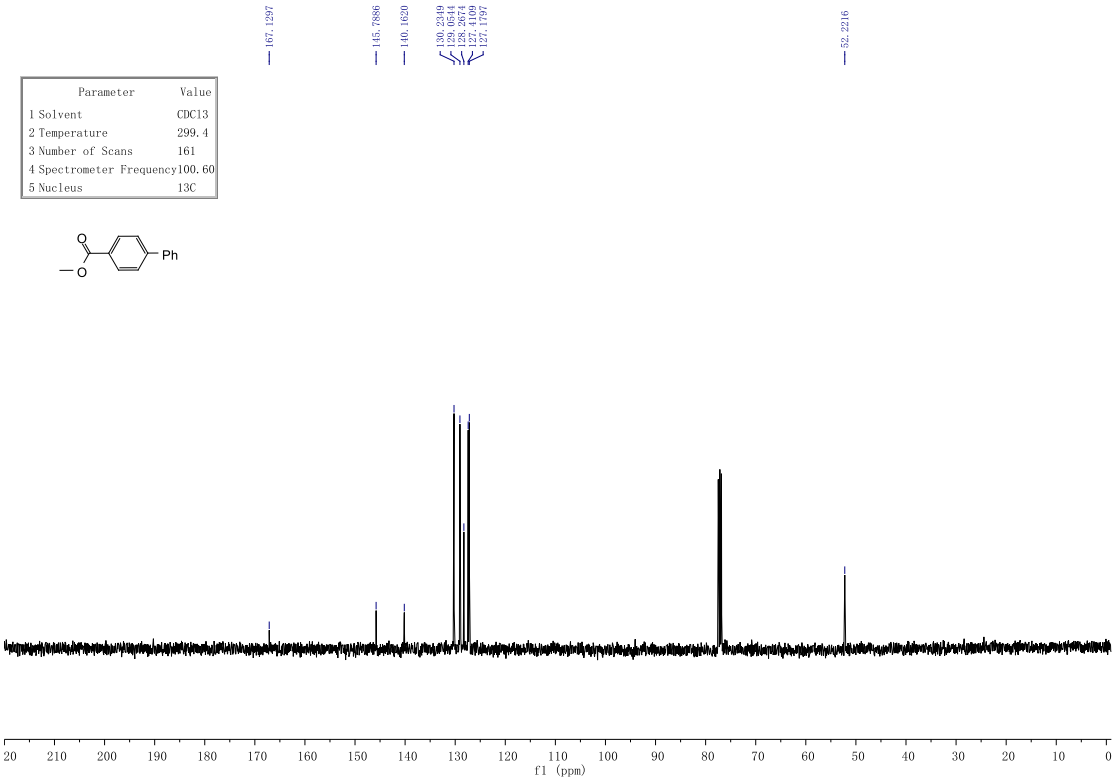
methyl 4-methoxybenzoate, **2k**



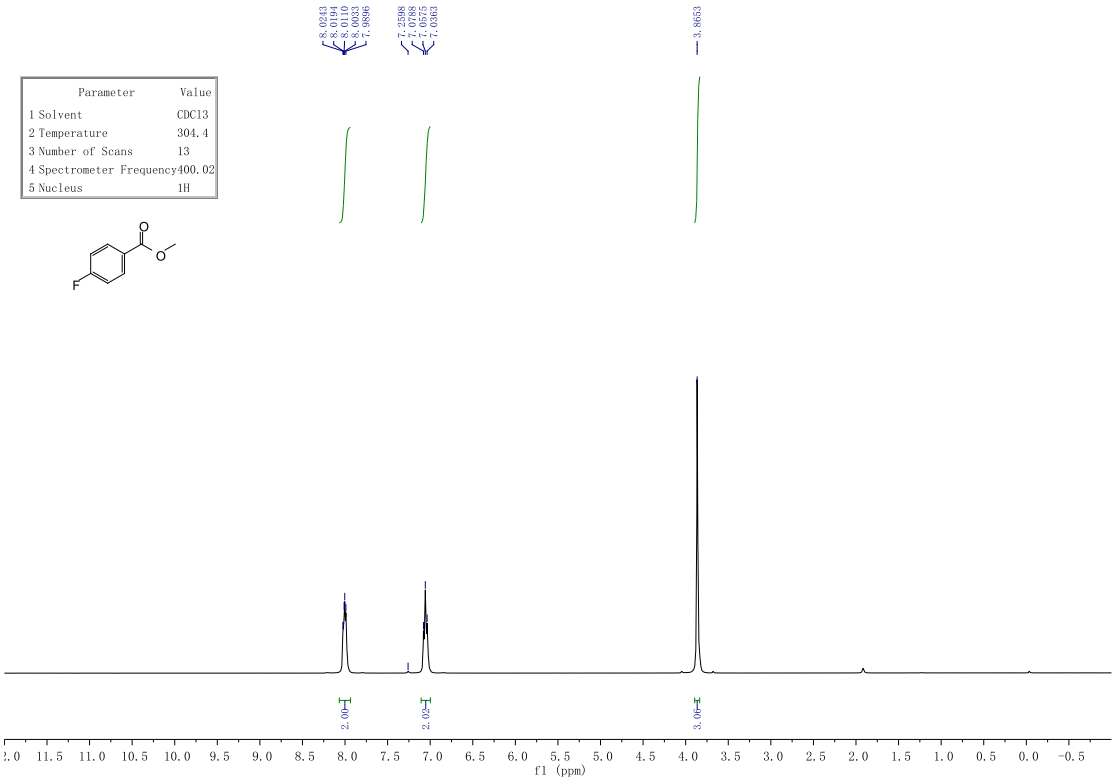


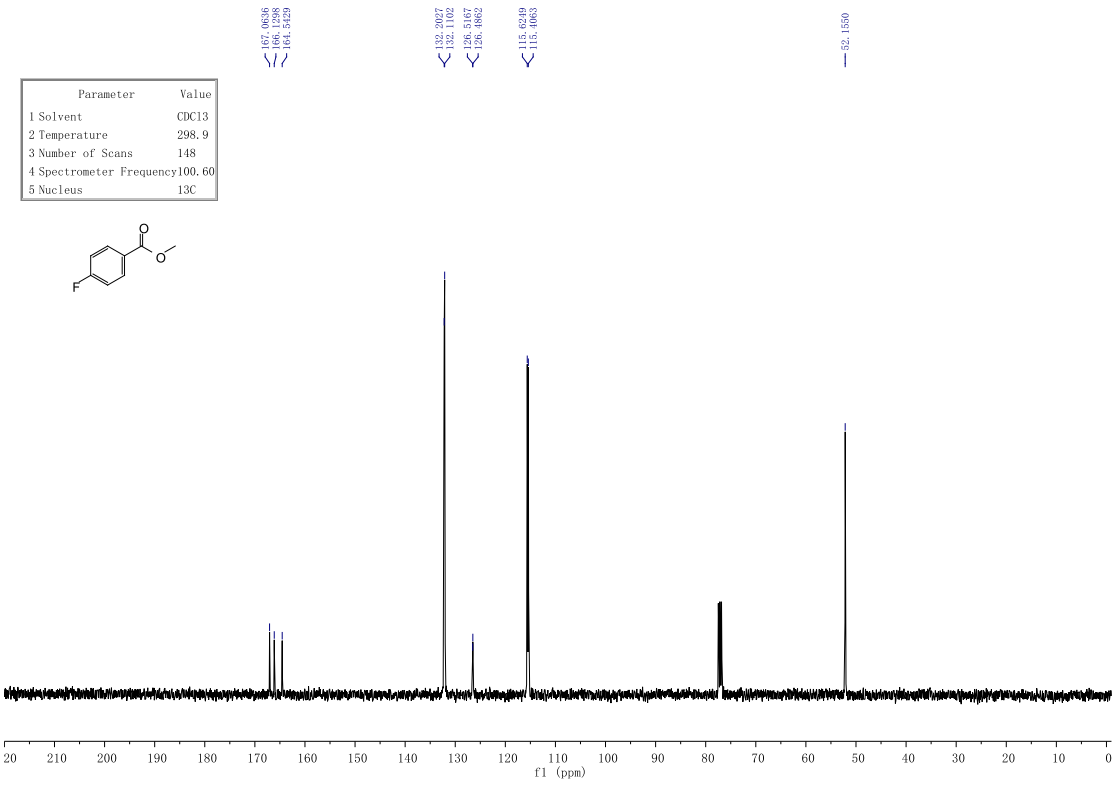
methyl [1,1'-biphenyl]-4-carboxylate, **2l**



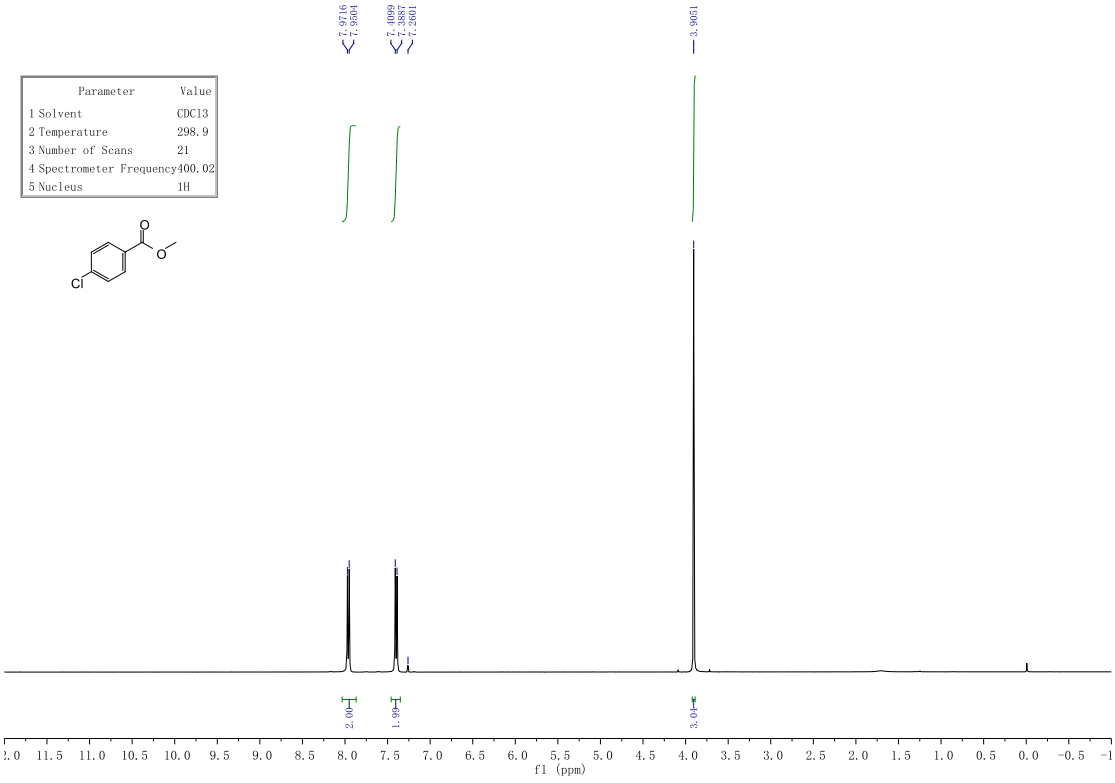


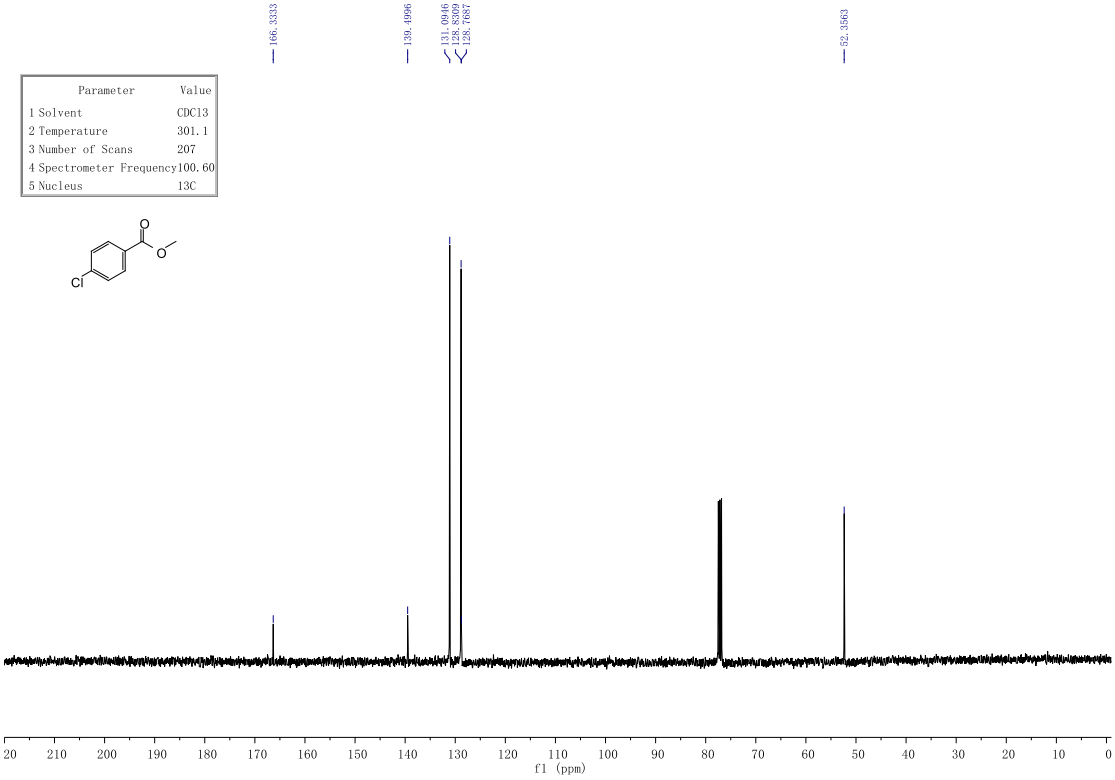
methyl 4-fluorobenzoate, **2m**



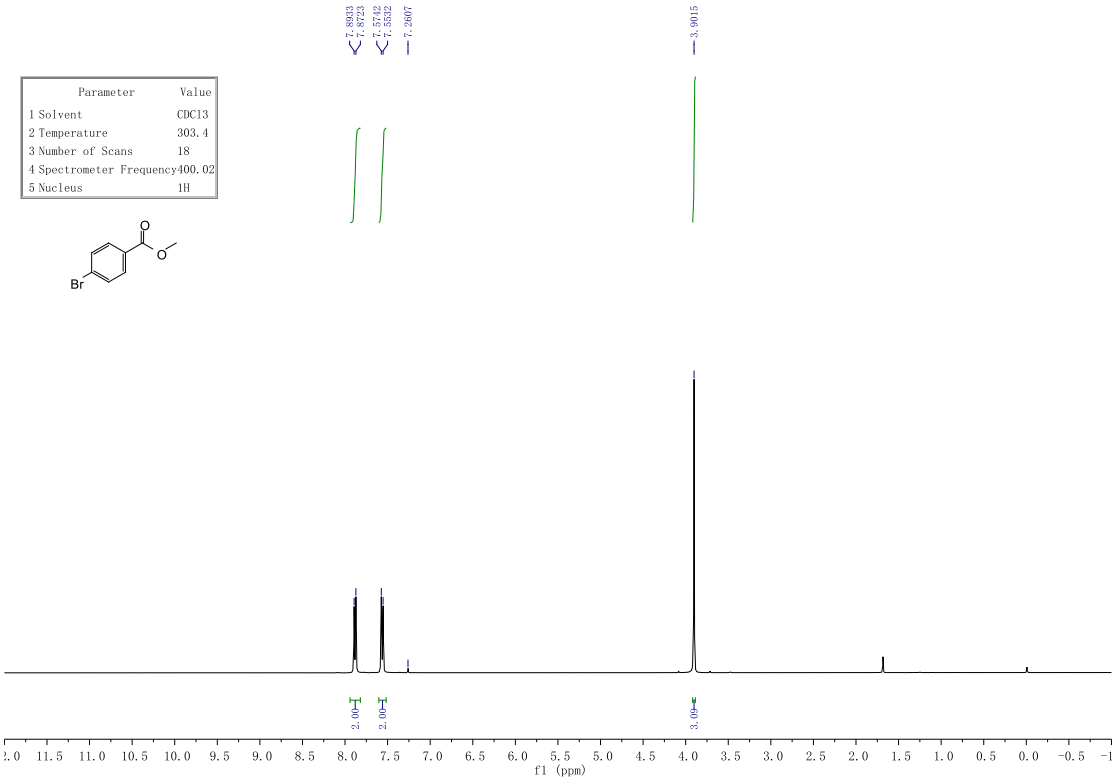


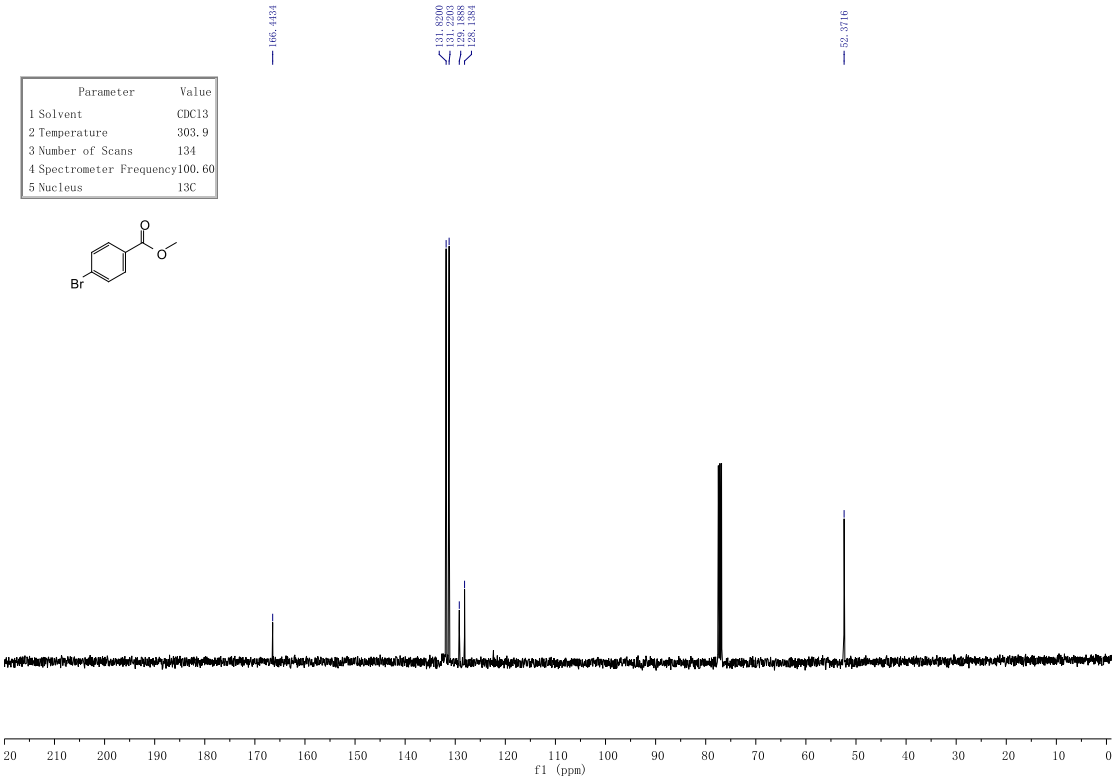
methyl 4-chlorobenzoate, **2n**



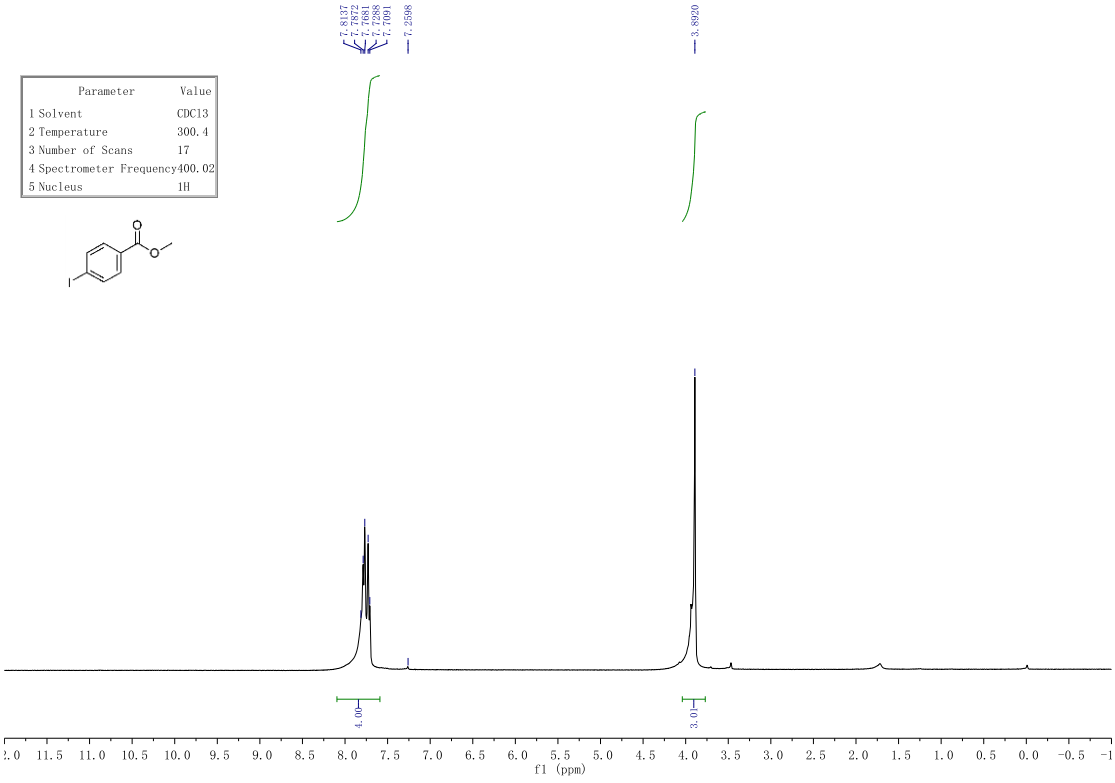


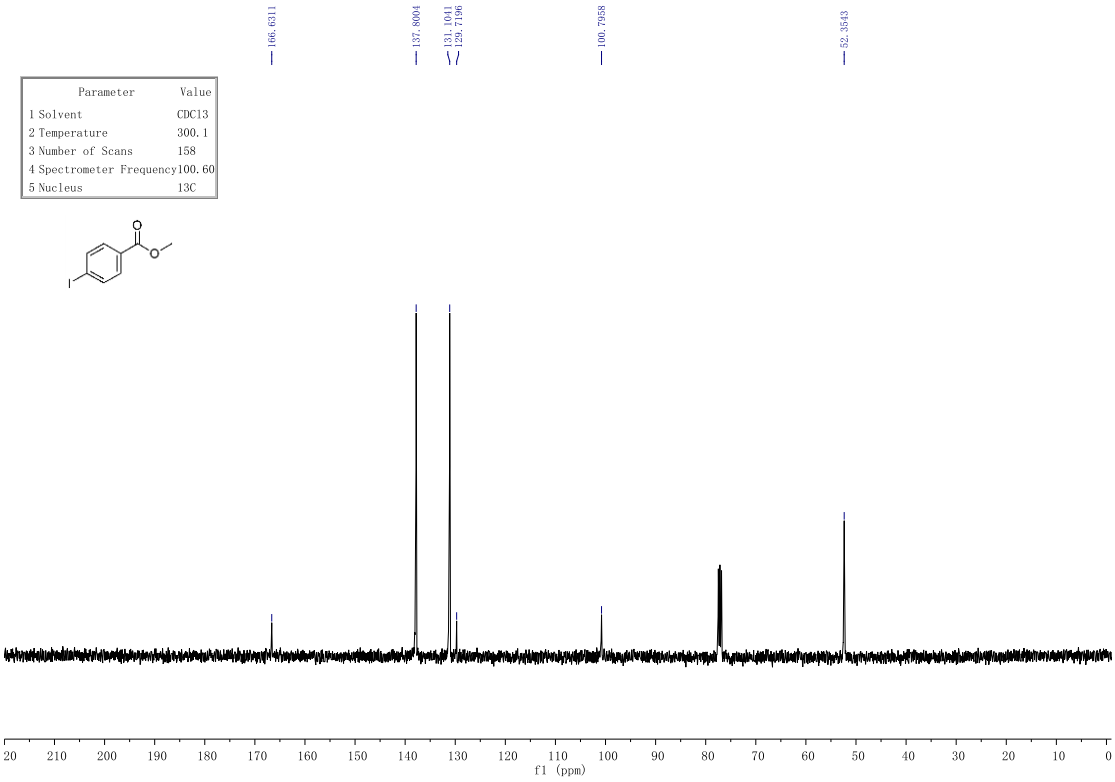
methyl 4-bromobenzoate, **2o**



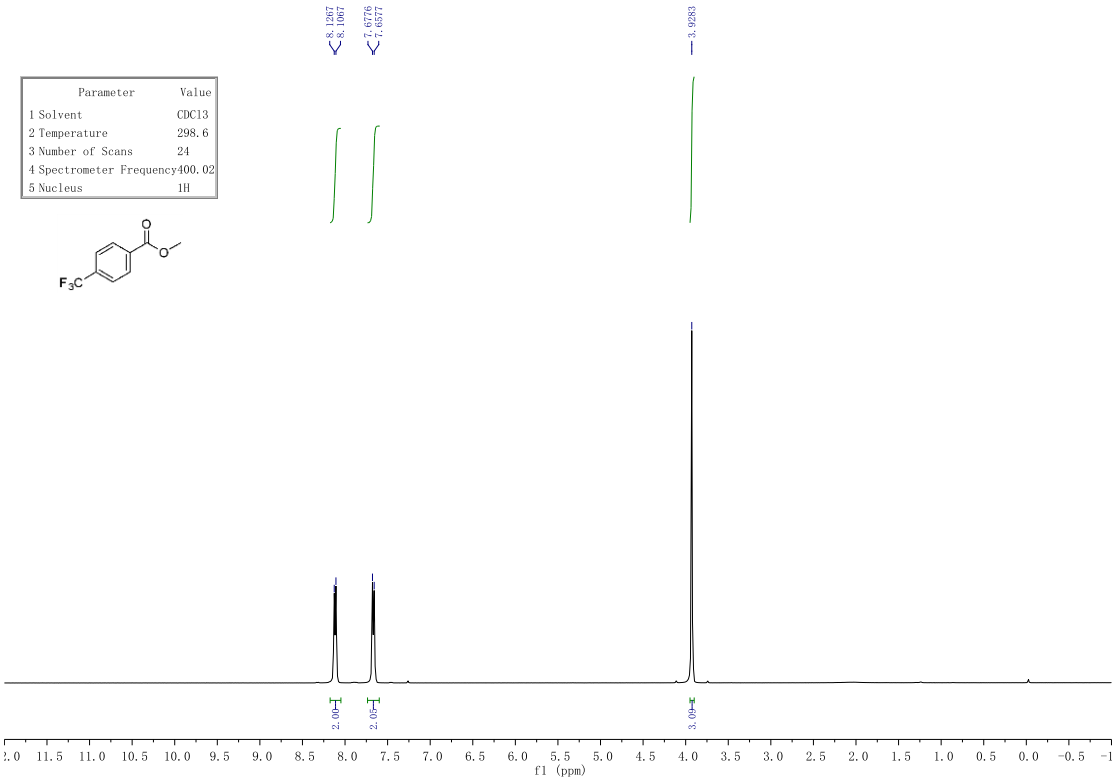


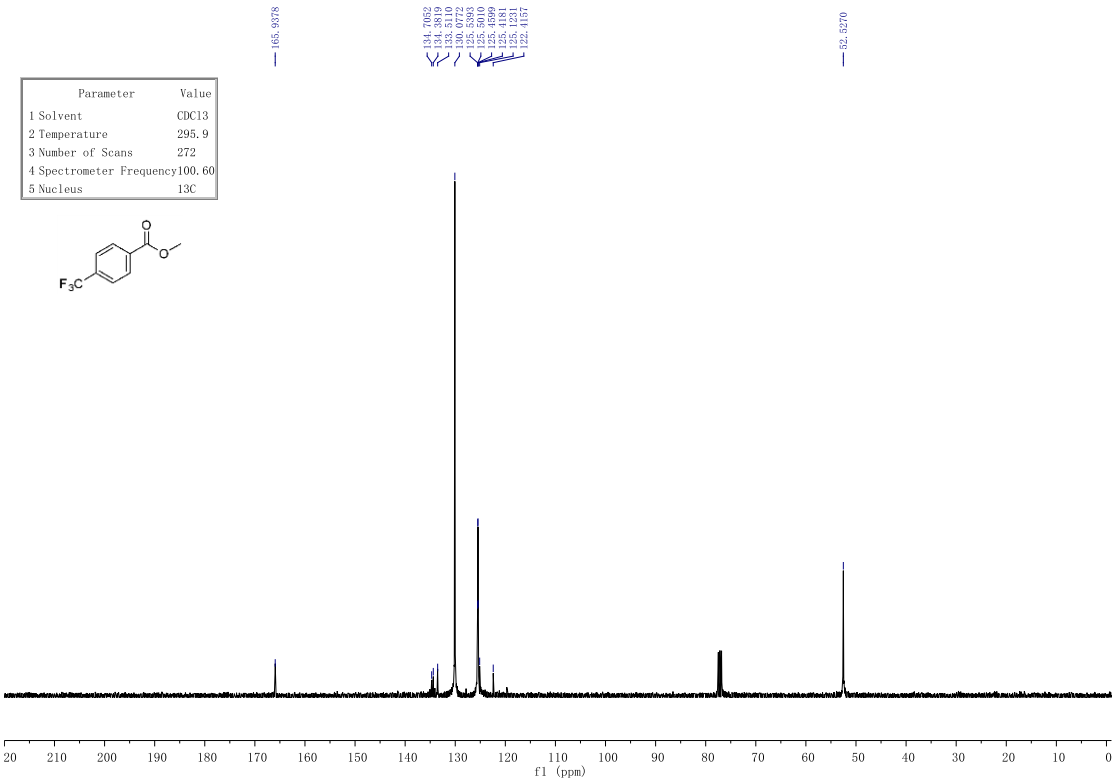
methyl 4-iodobenzoate, **2p**



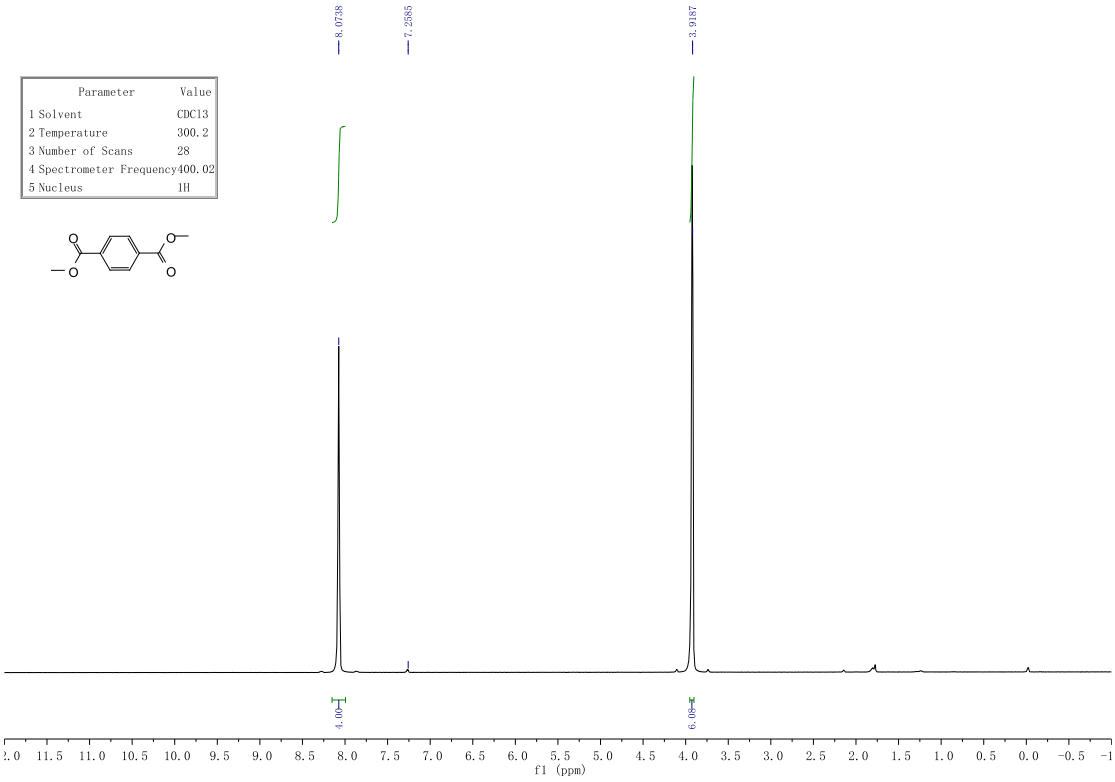


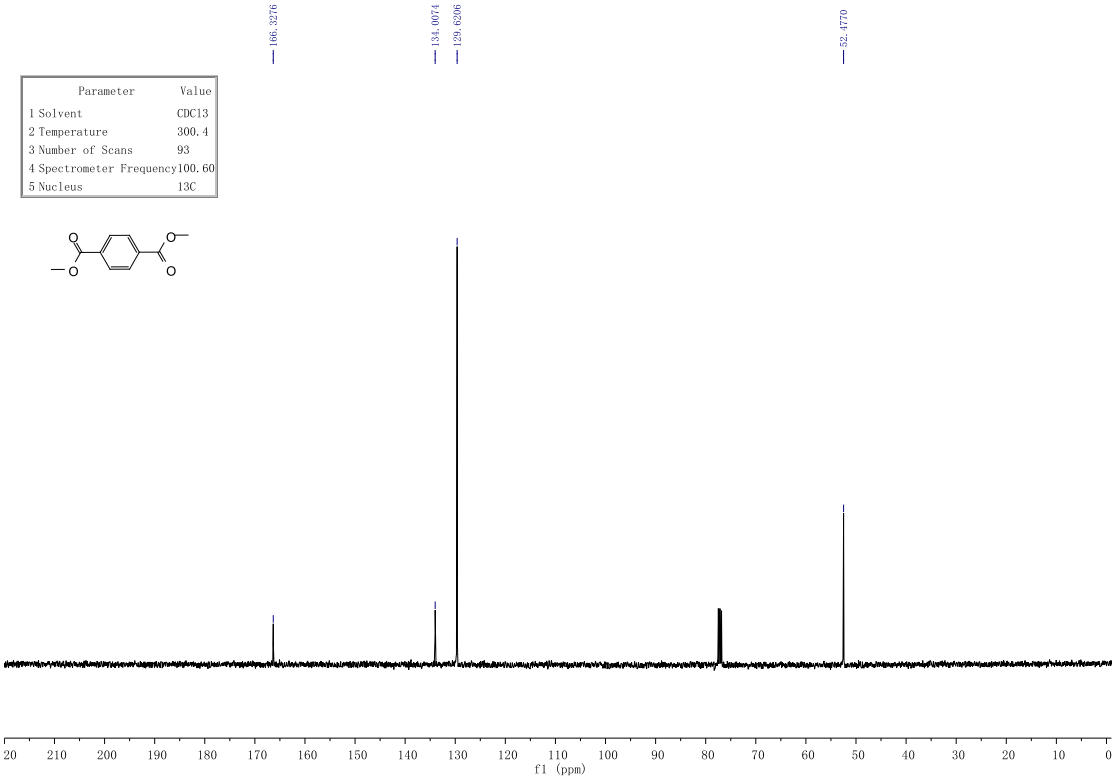
methyl 4-(trifluoromethyl)benzoate**, 2q**



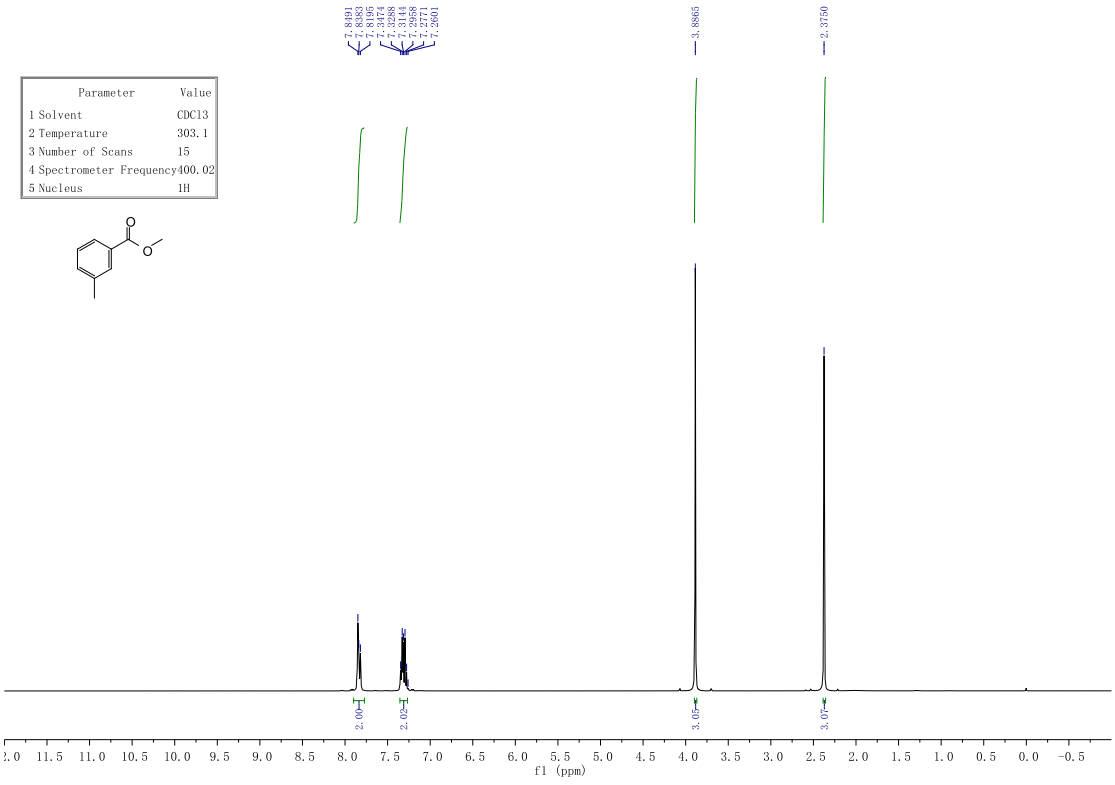


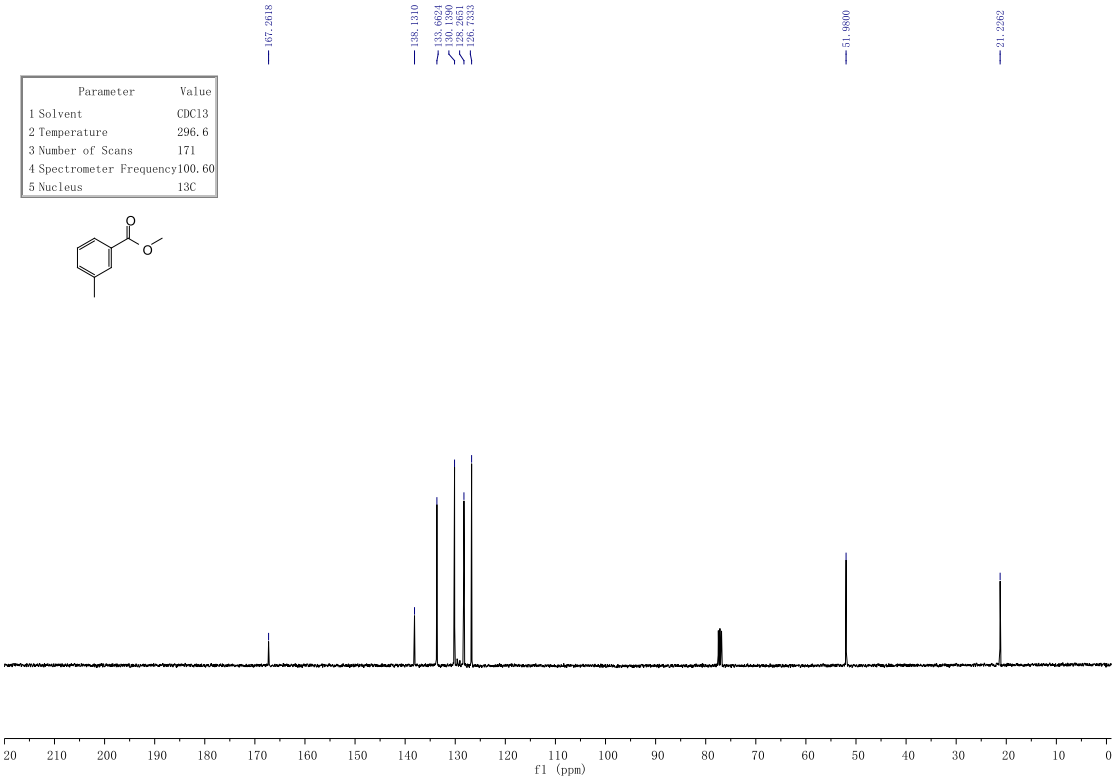
dimethyl terephthalate, **2r**



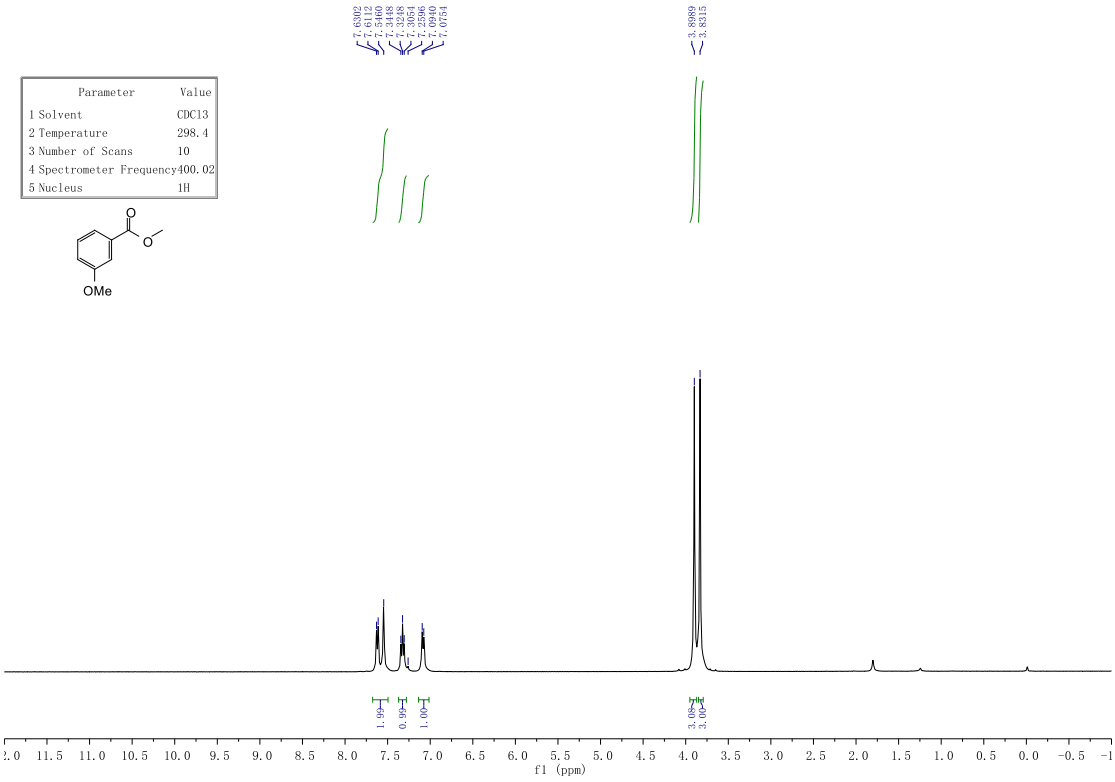


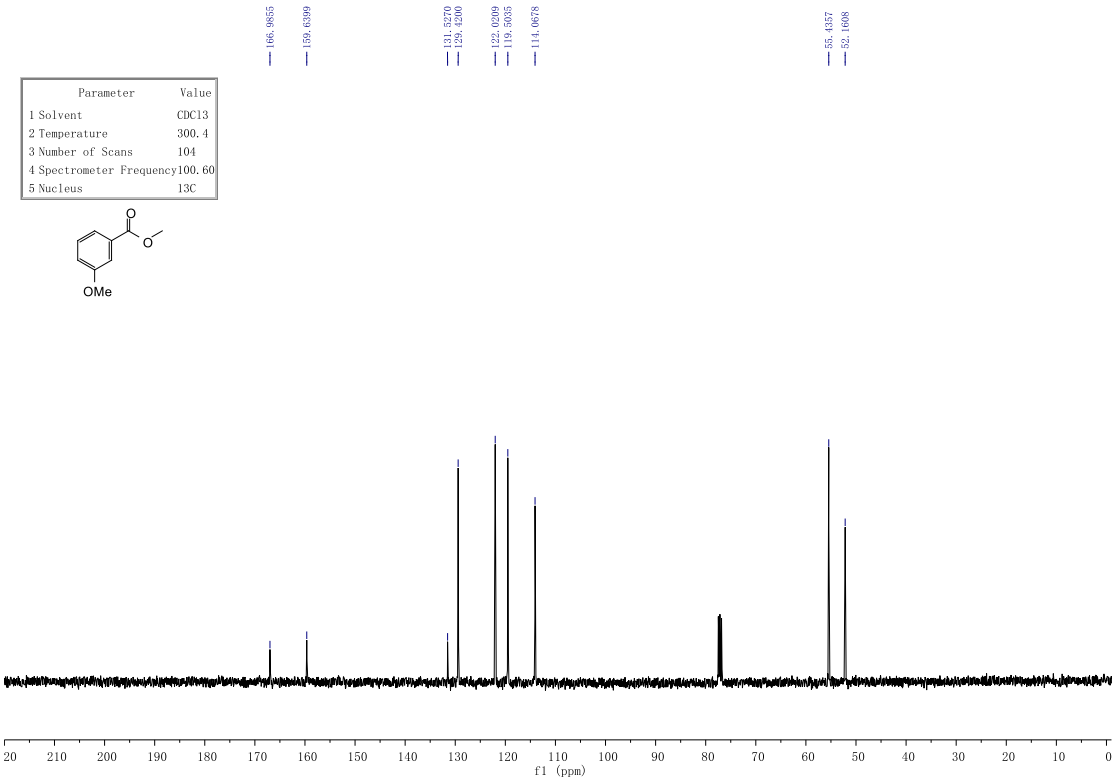
methyl 3-methylbenzoate, 2s



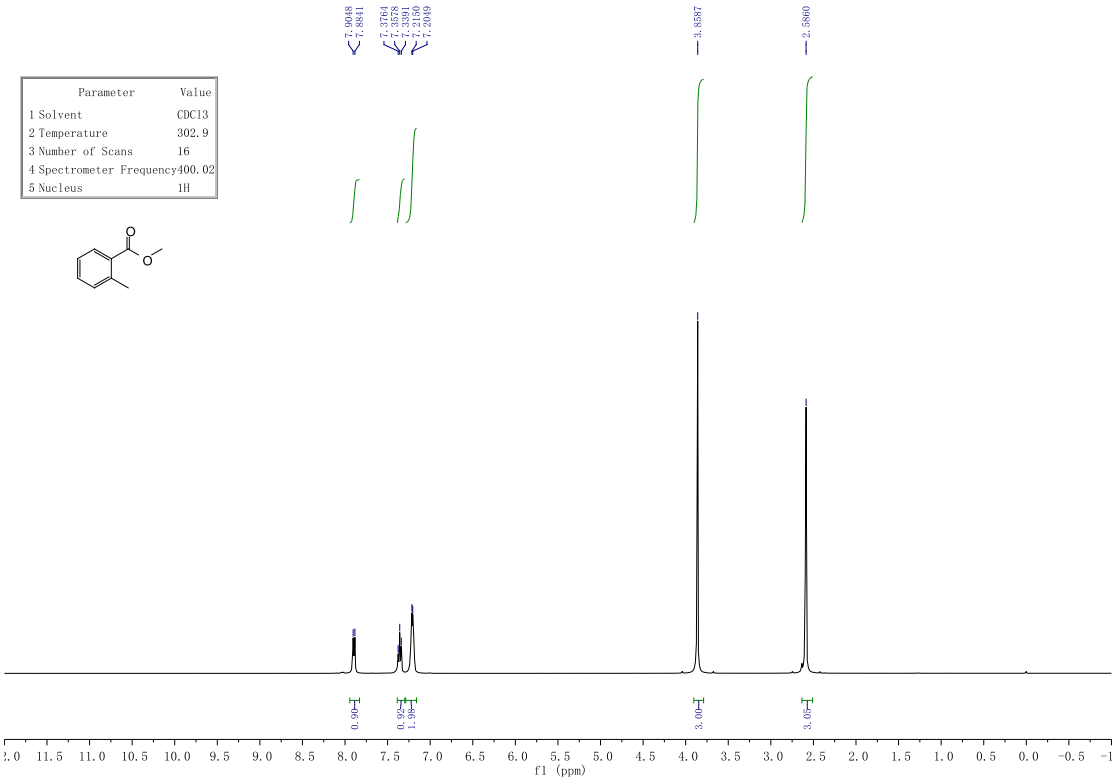


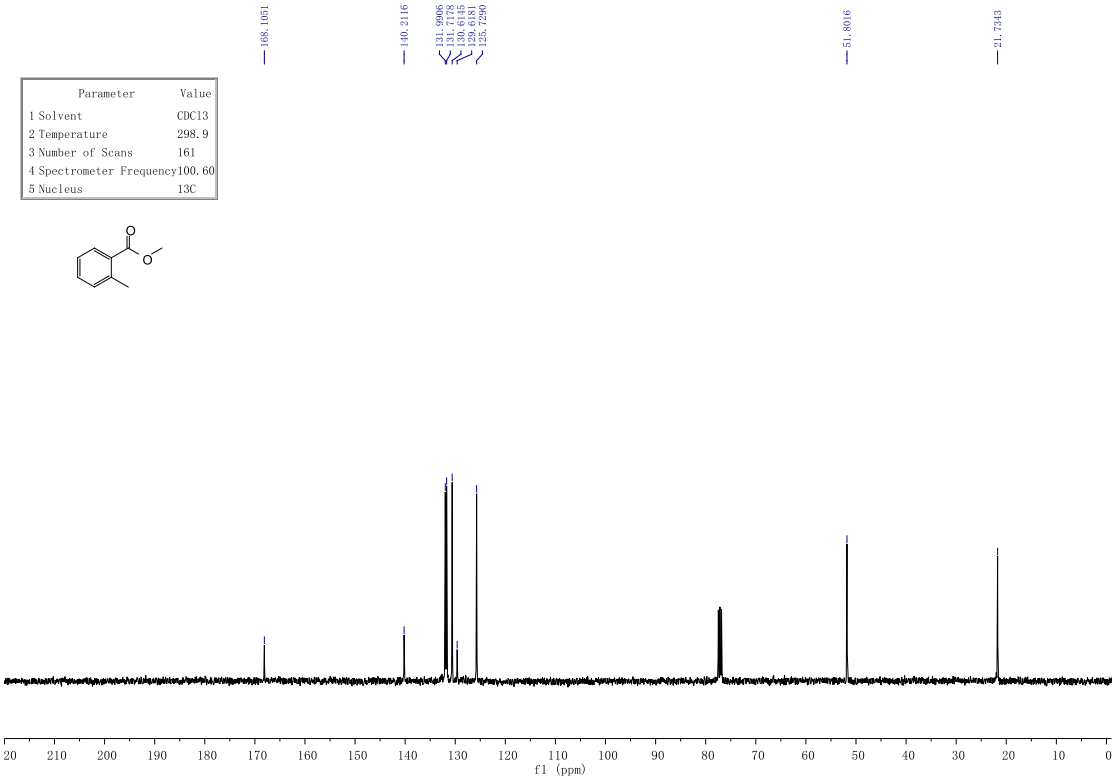
methyl 3-methoxybenzoate, **2t**



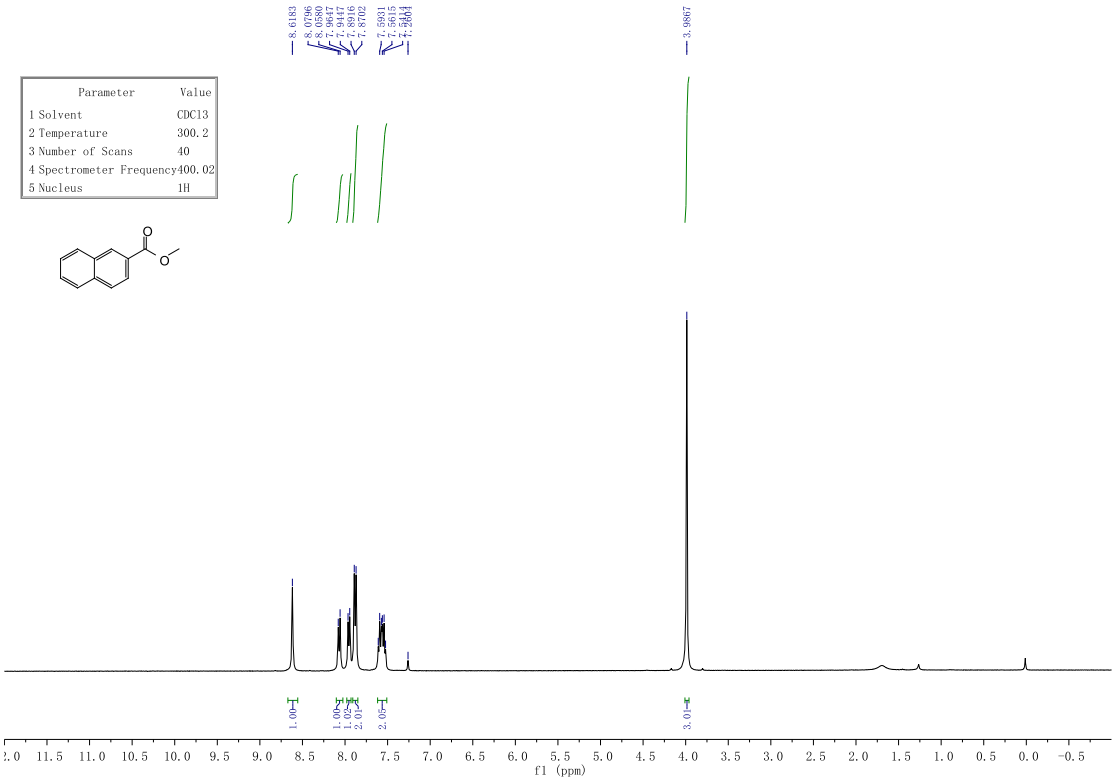


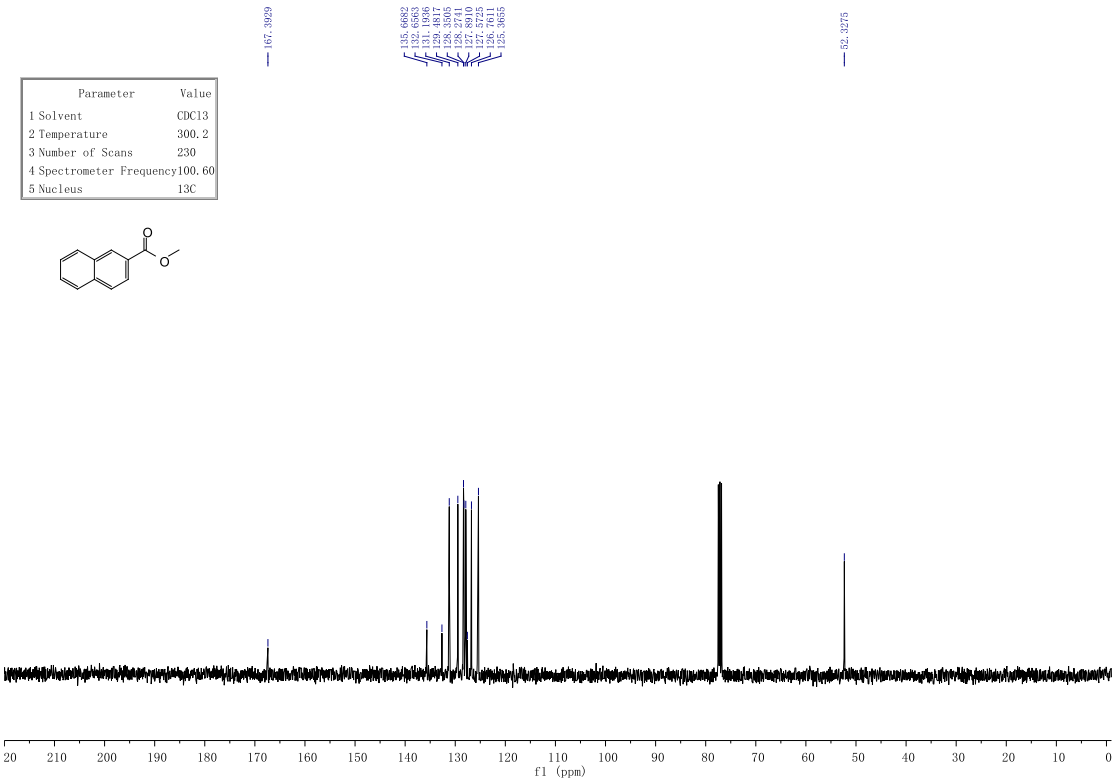
methyl 2-methylbenzoate, **2u**



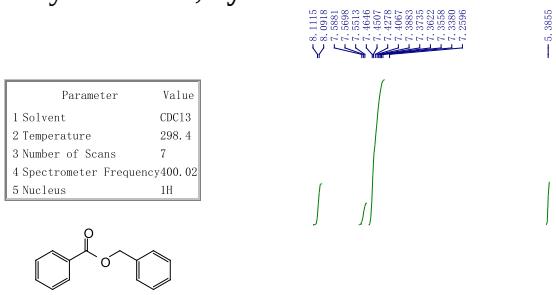


methyl 2-naphthoate, 2v

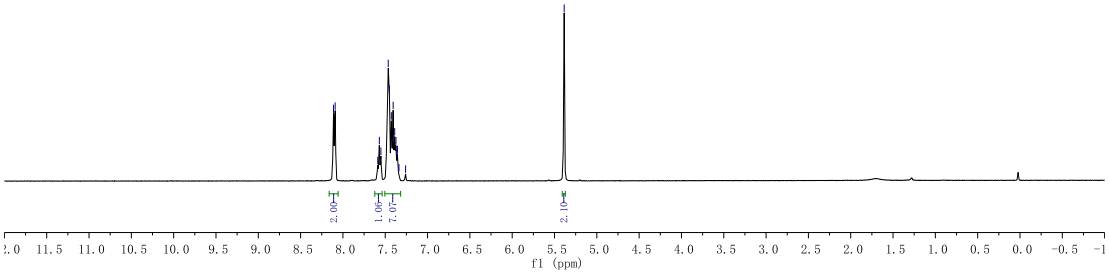




benzyl benzoate, **2y**



Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.4
3 Number of Scans	7
4 Spectrometer Frequency	400.02
5 Nucleus	1H



Parameter	Value
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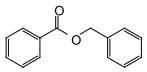
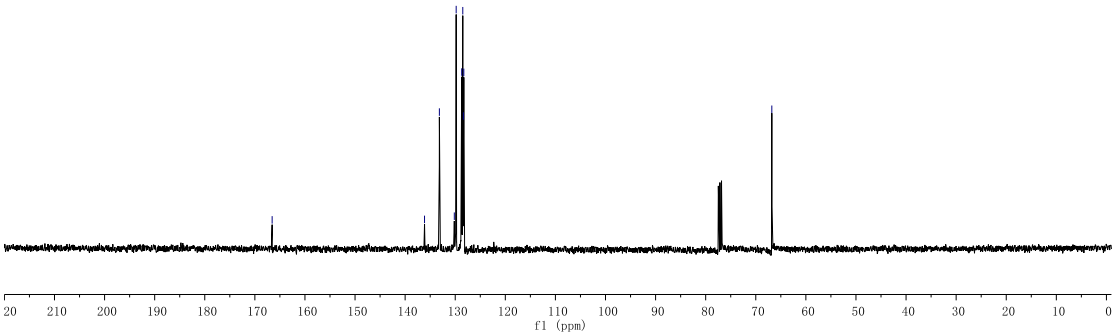
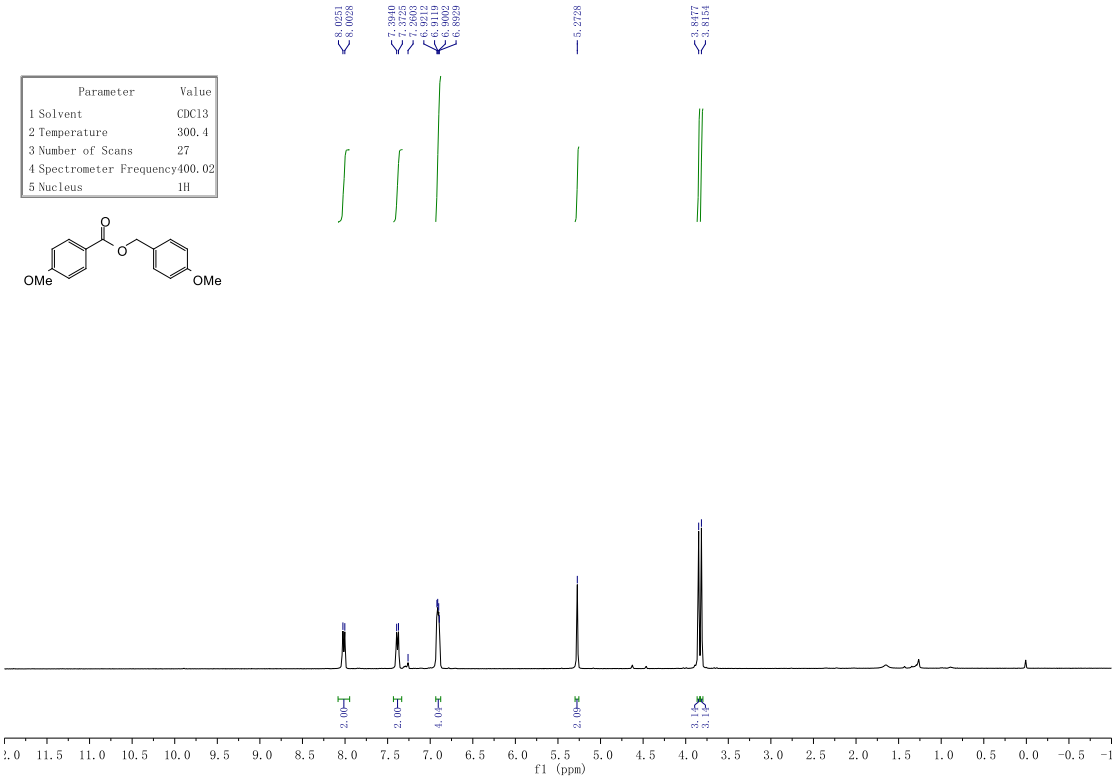
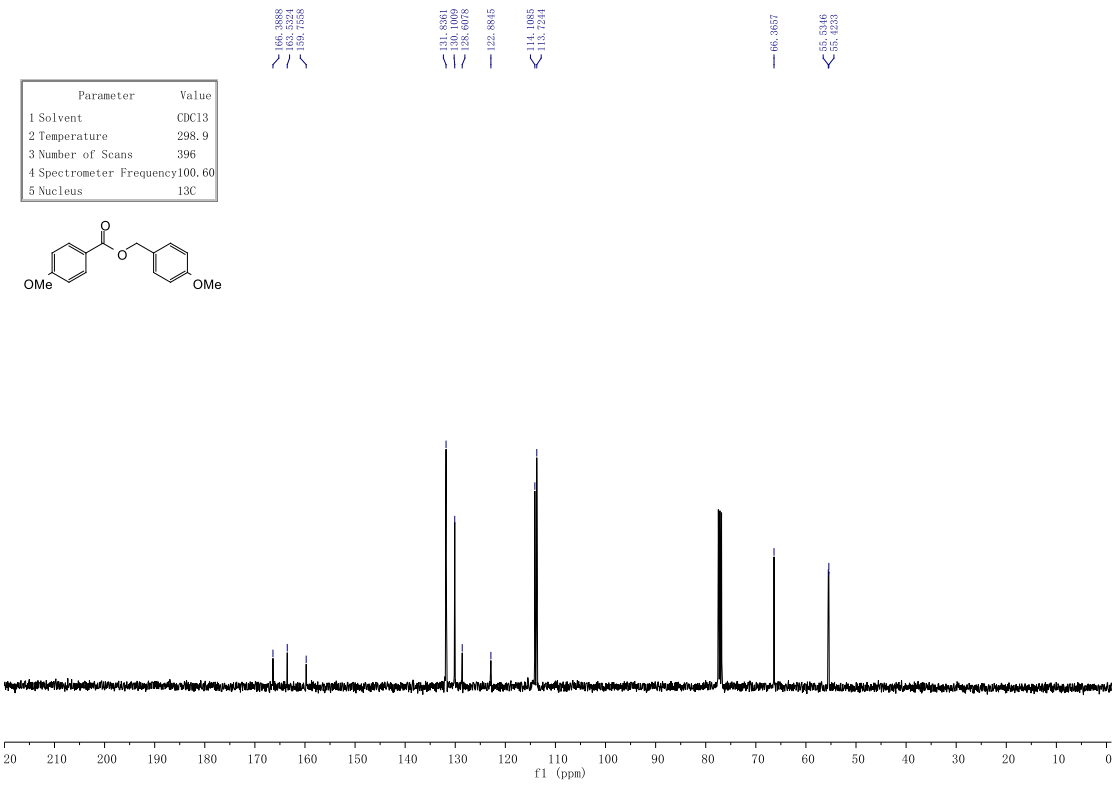
2 Temperature	293.6
3 Number of Scans	120
4 Spectrometer Frequency	100.59
5 Nucleus	13C



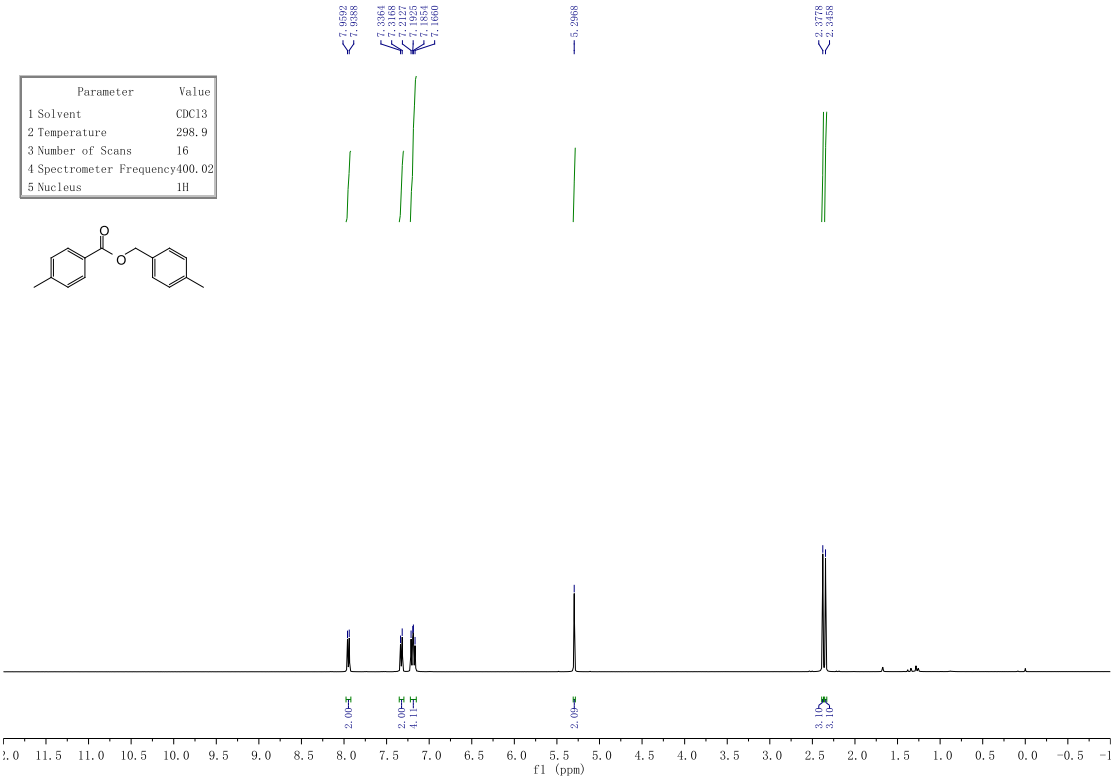


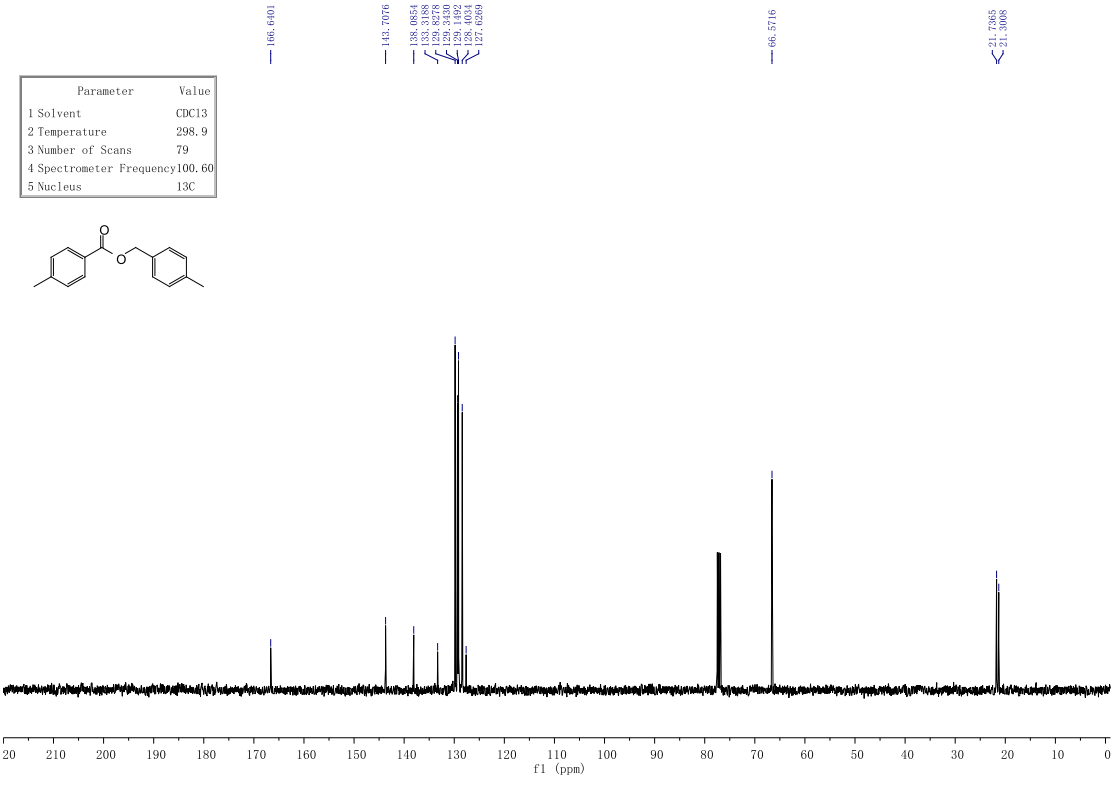
4-methoxybenzyl 4-methoxybenzoate, **2z**



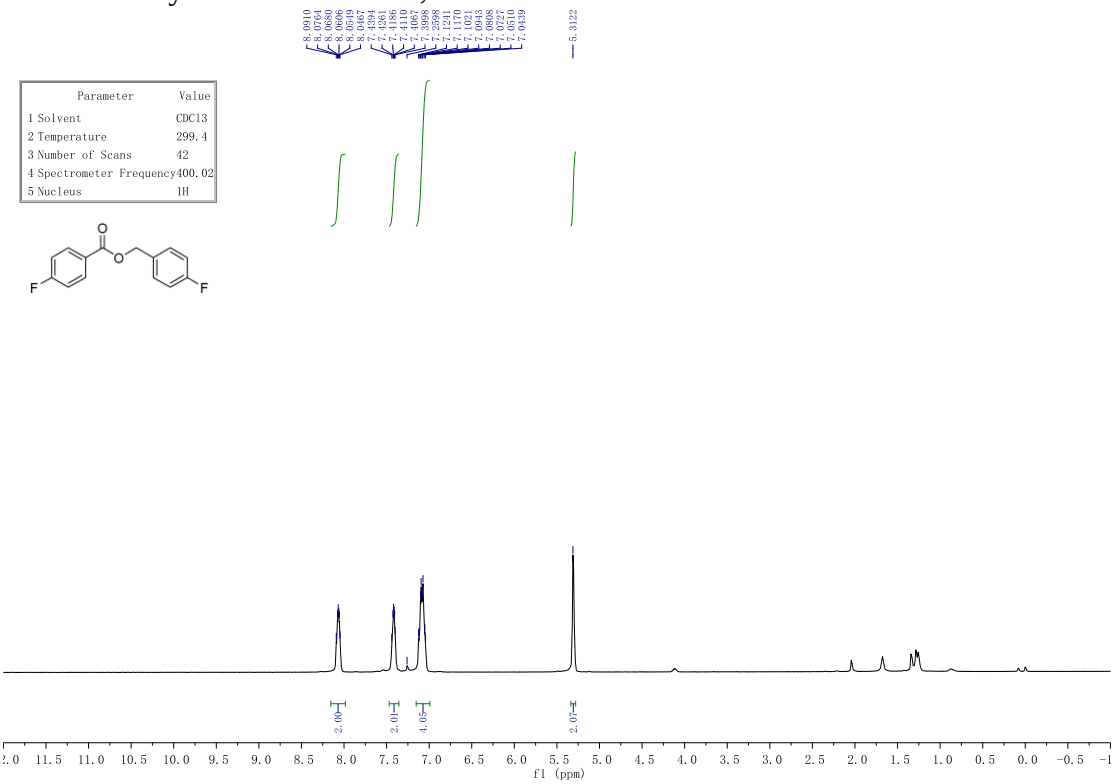


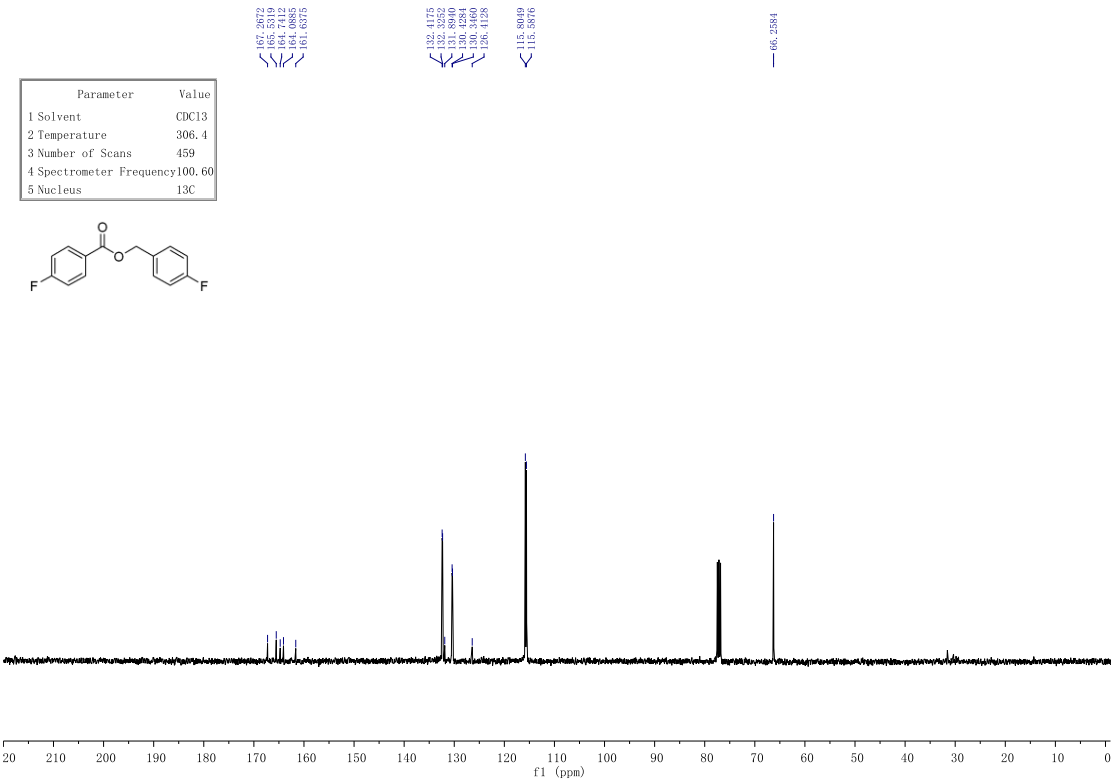
4-methylbenzyl 4-methylbenzoate, **2aa**



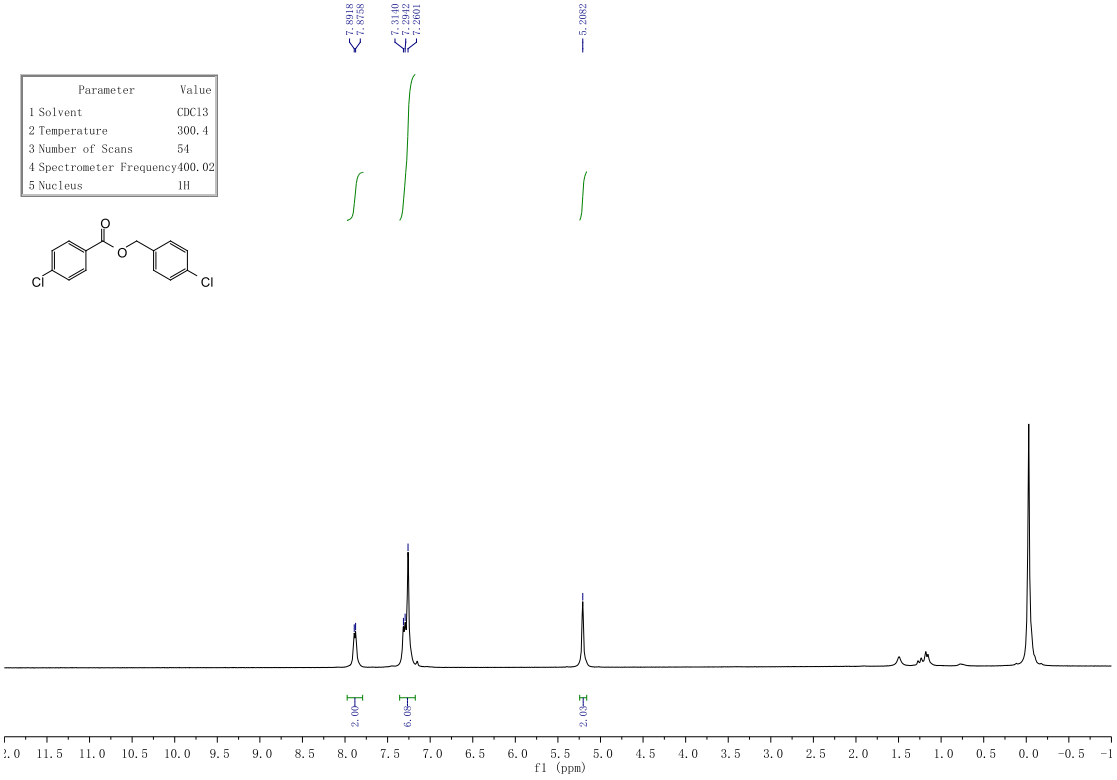


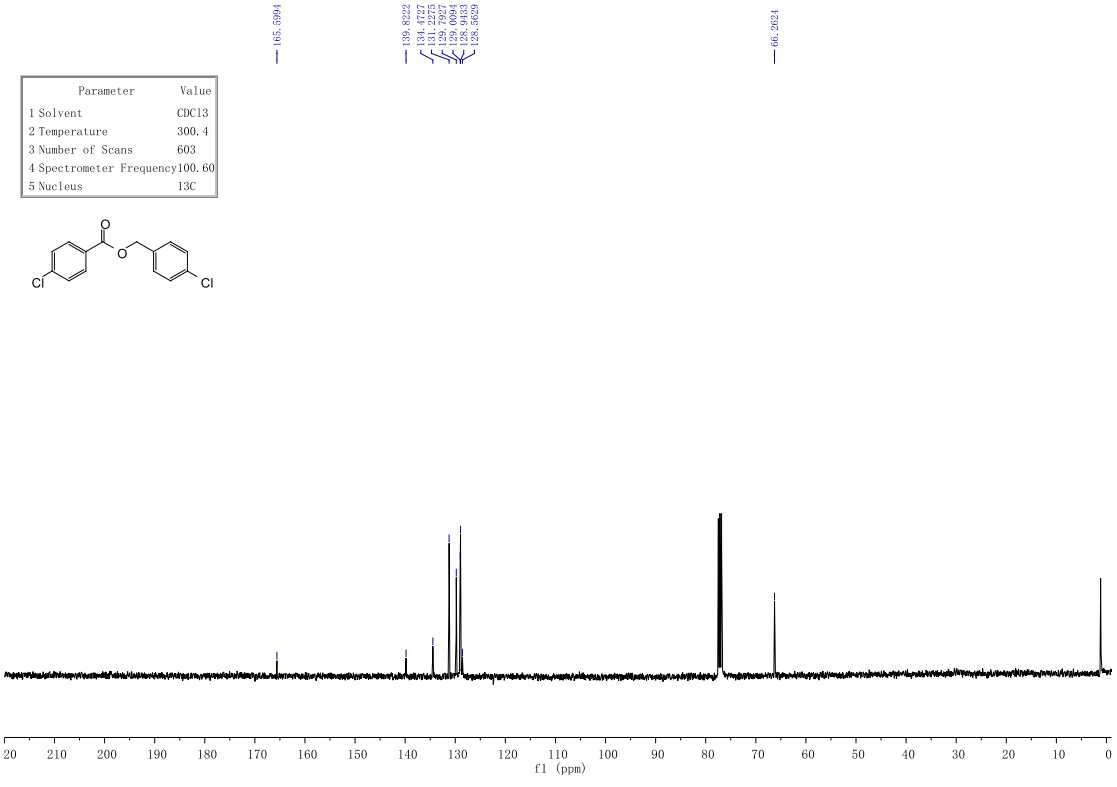
4-fluorobenzyl 4-fluorobenzoate, **2ab**



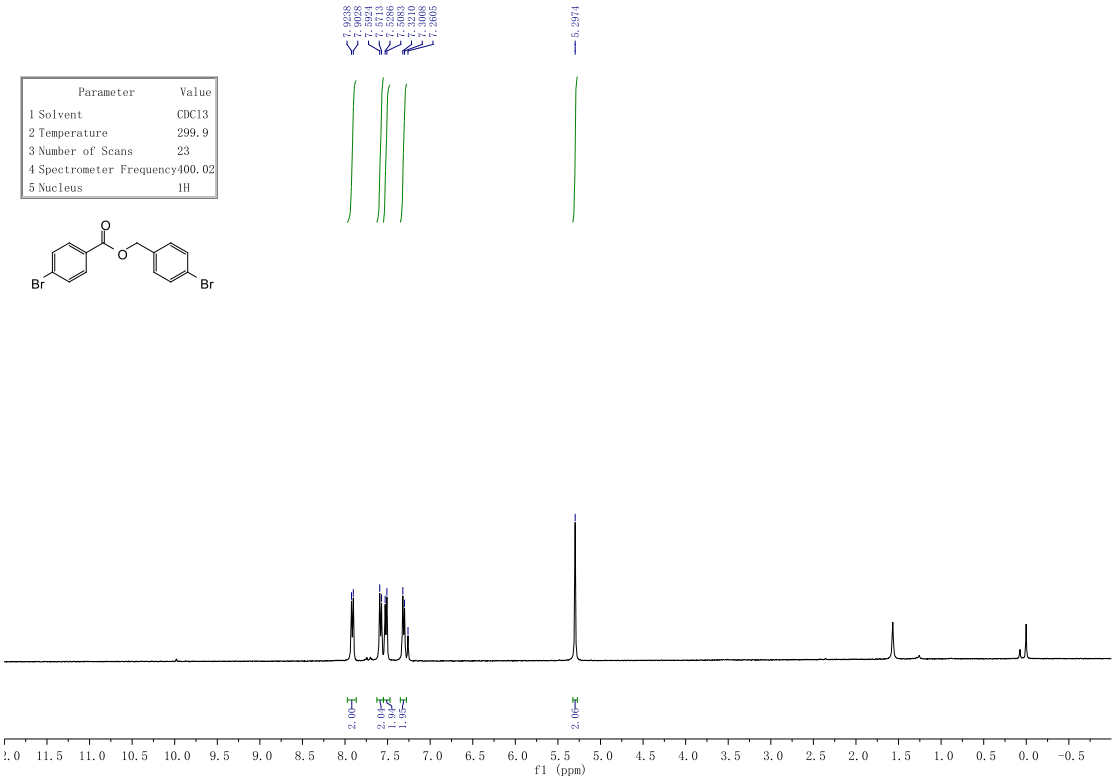


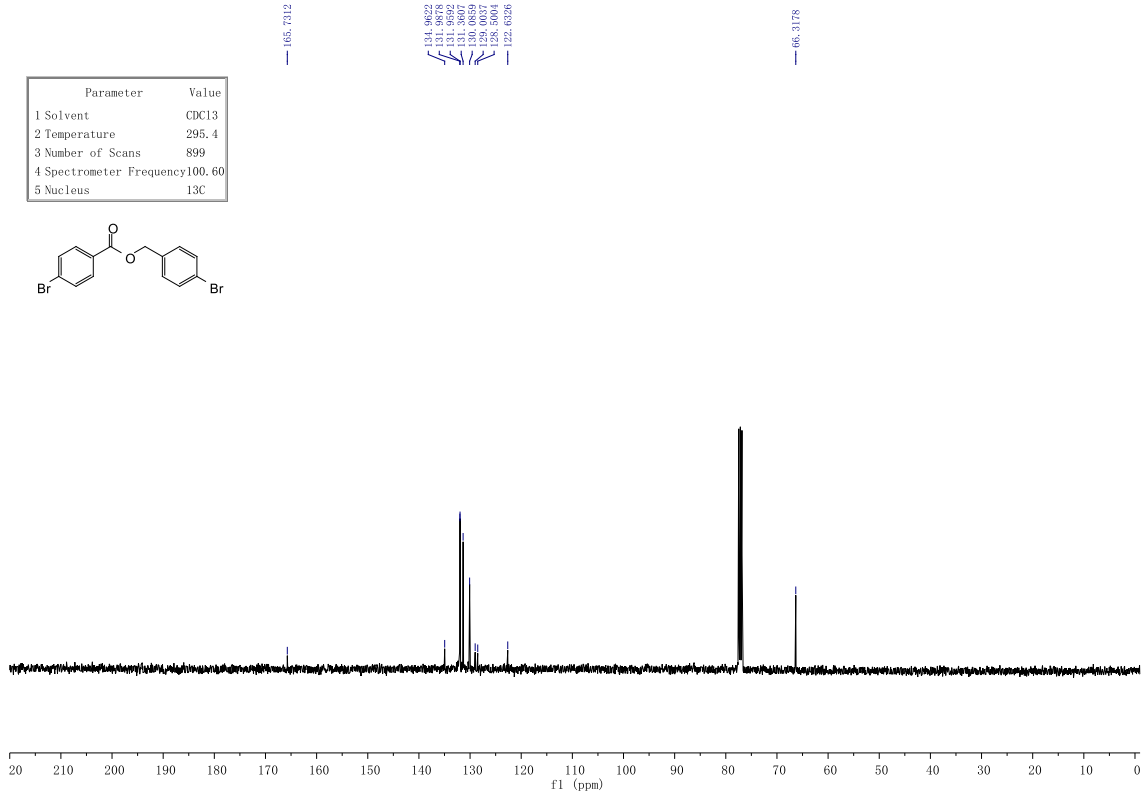
4-chlorobenzyl 4-chlorobenzoate, **2ac**



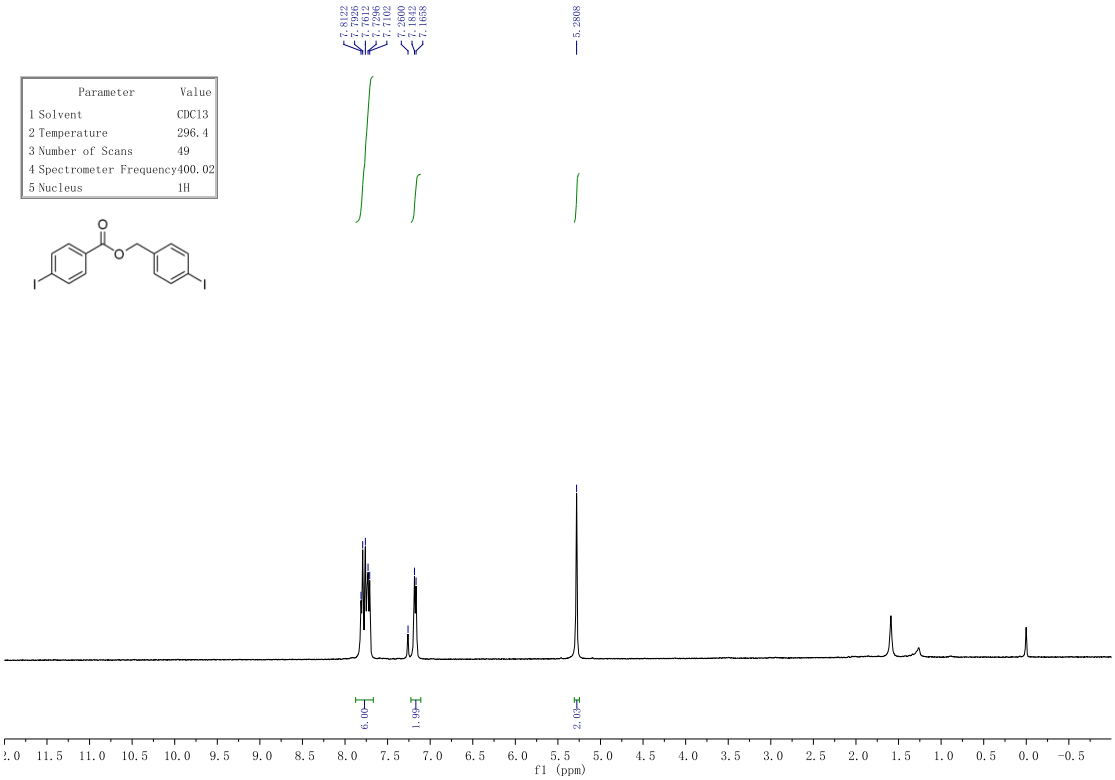


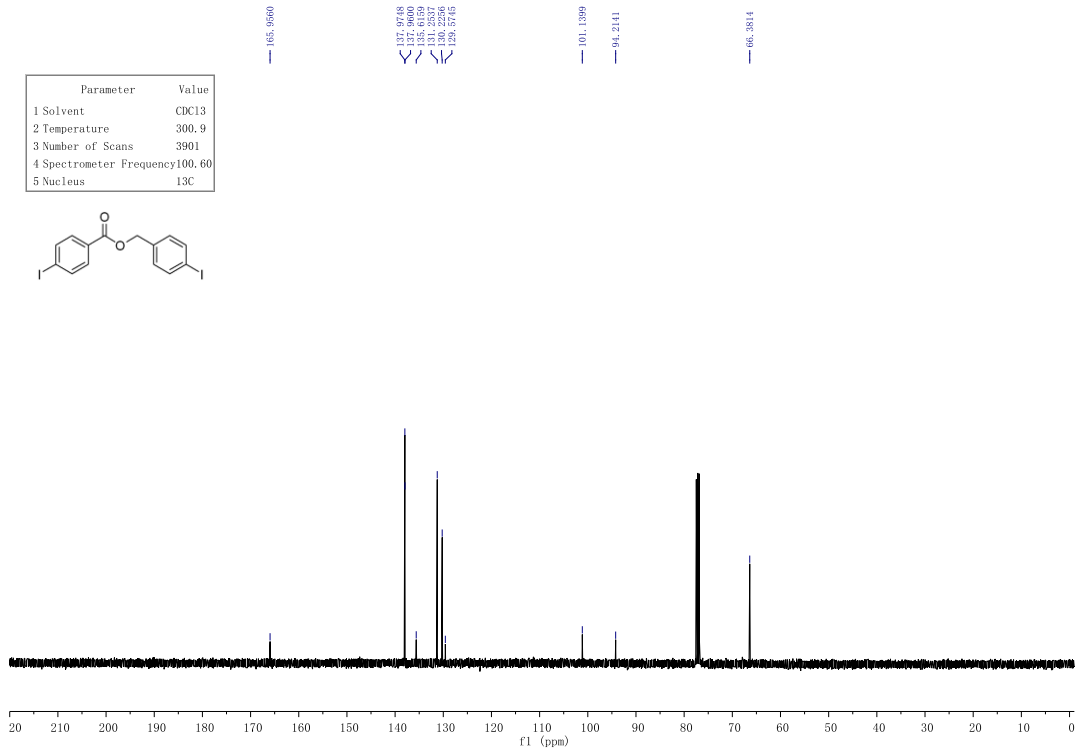
4-bromobenzyl 4-bromobenzoate, **2ad**



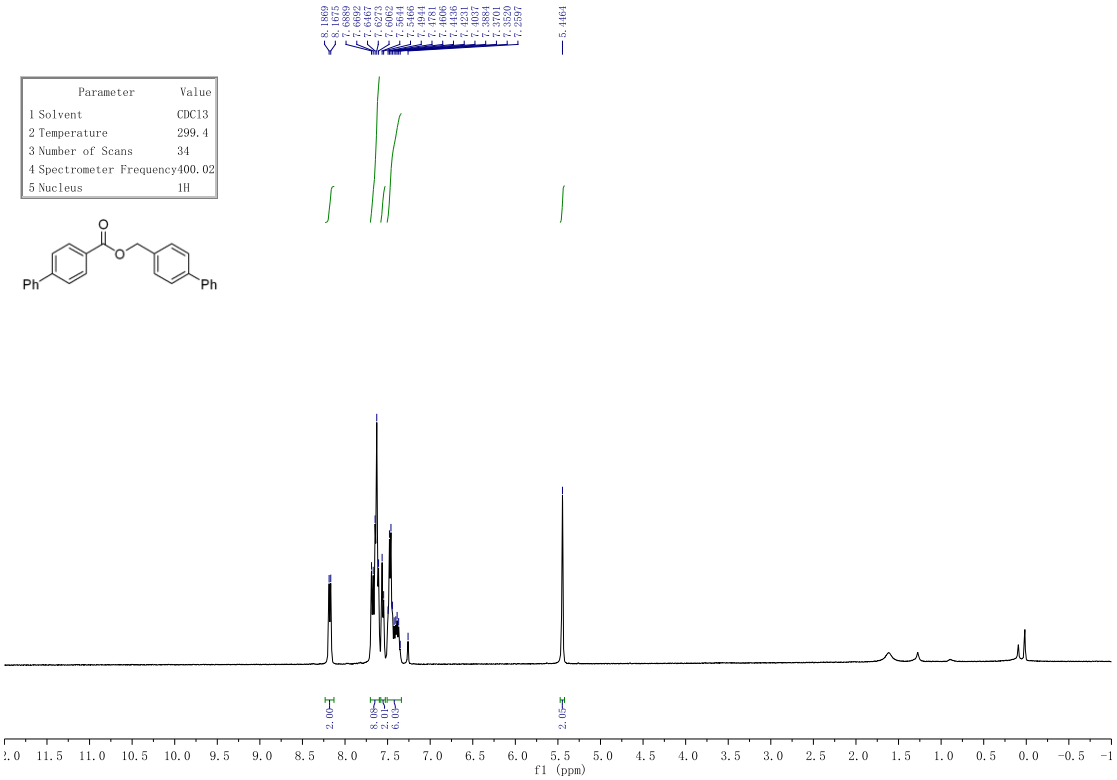


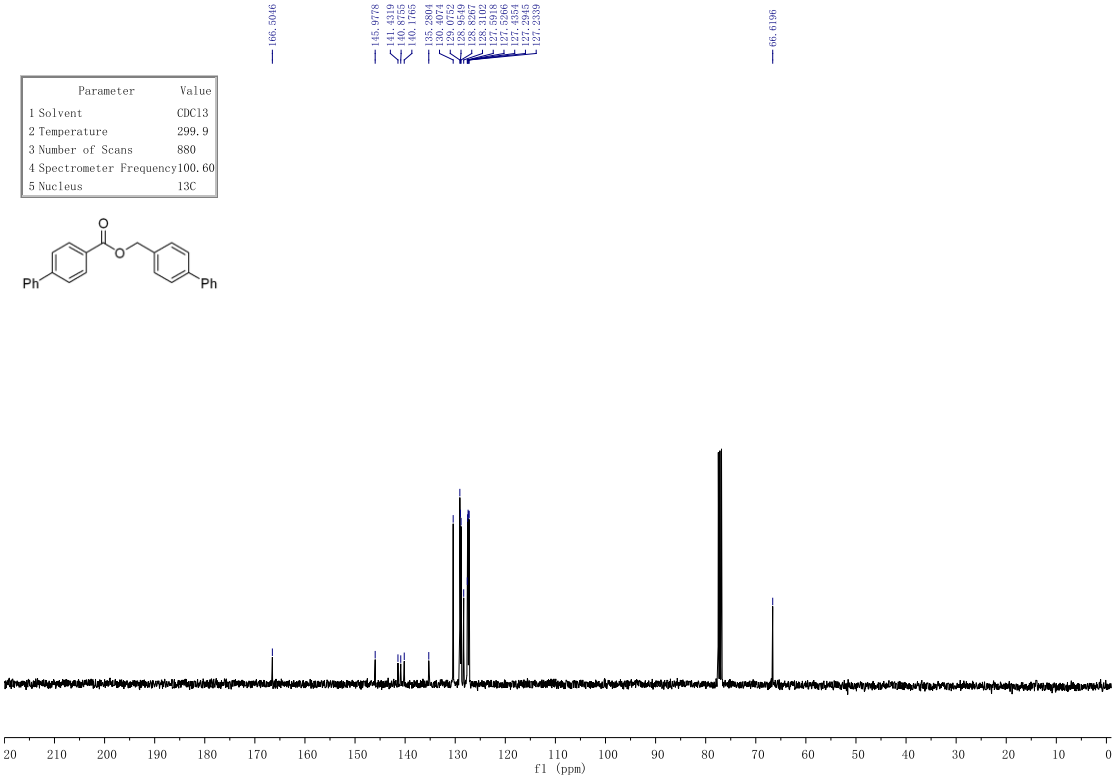
4-iodobenzyl 4-iodobenzoate, **2ae**



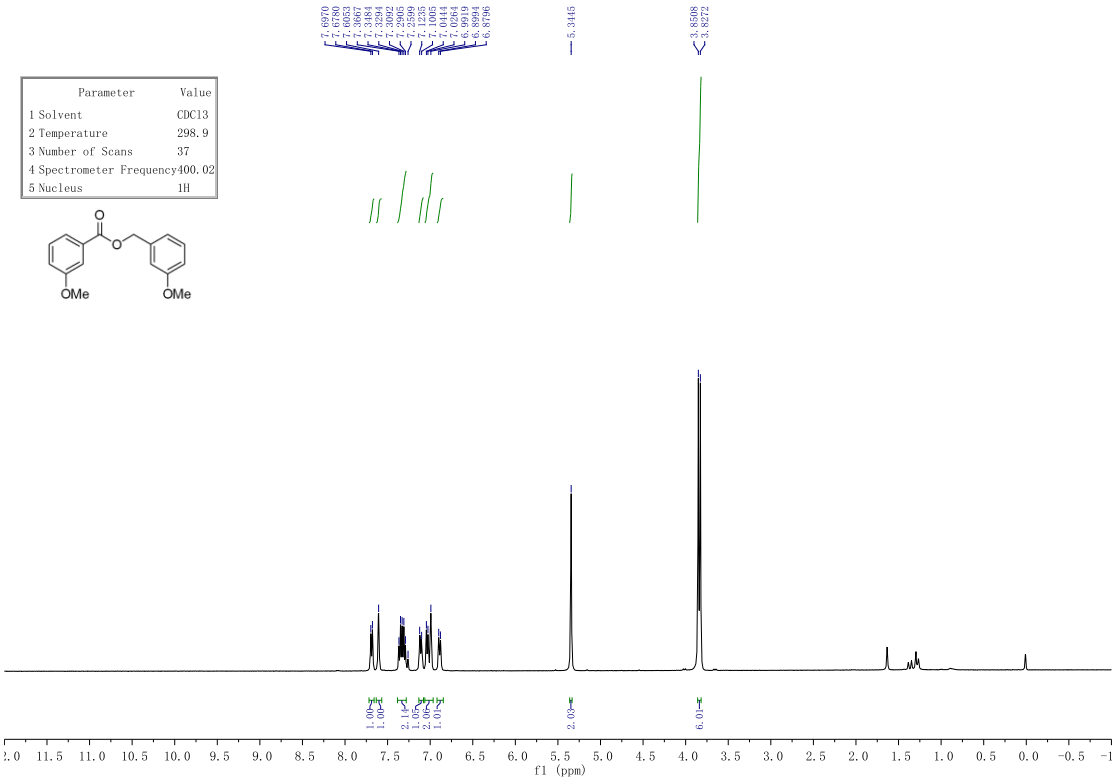


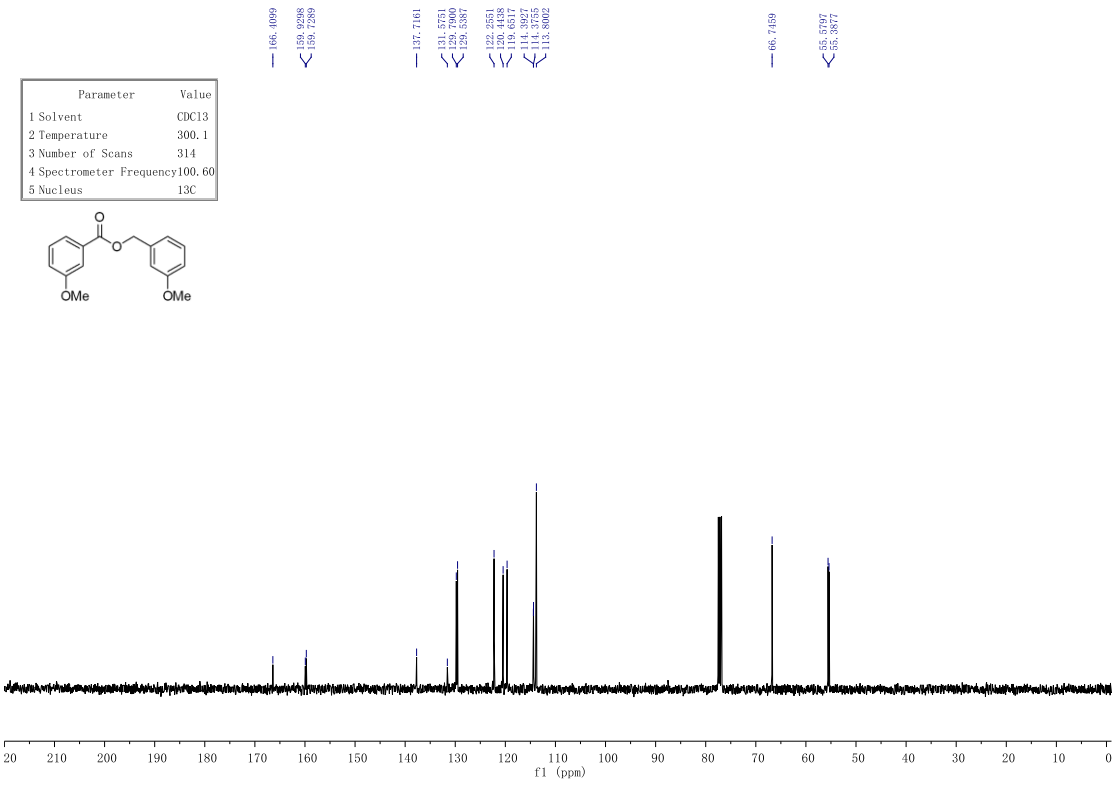
[1,1'-biphenyl]-4-ylmethyl [1,1'-biphenyl]-4-carboxylate, **2af**



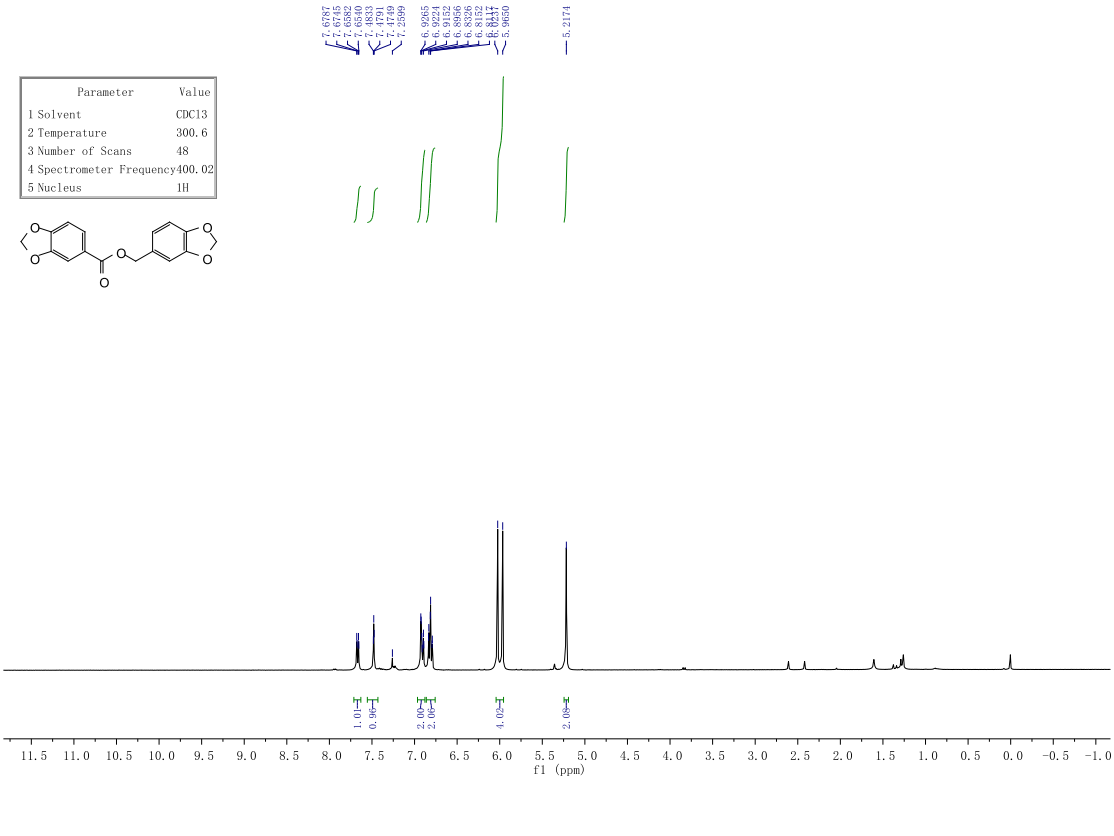


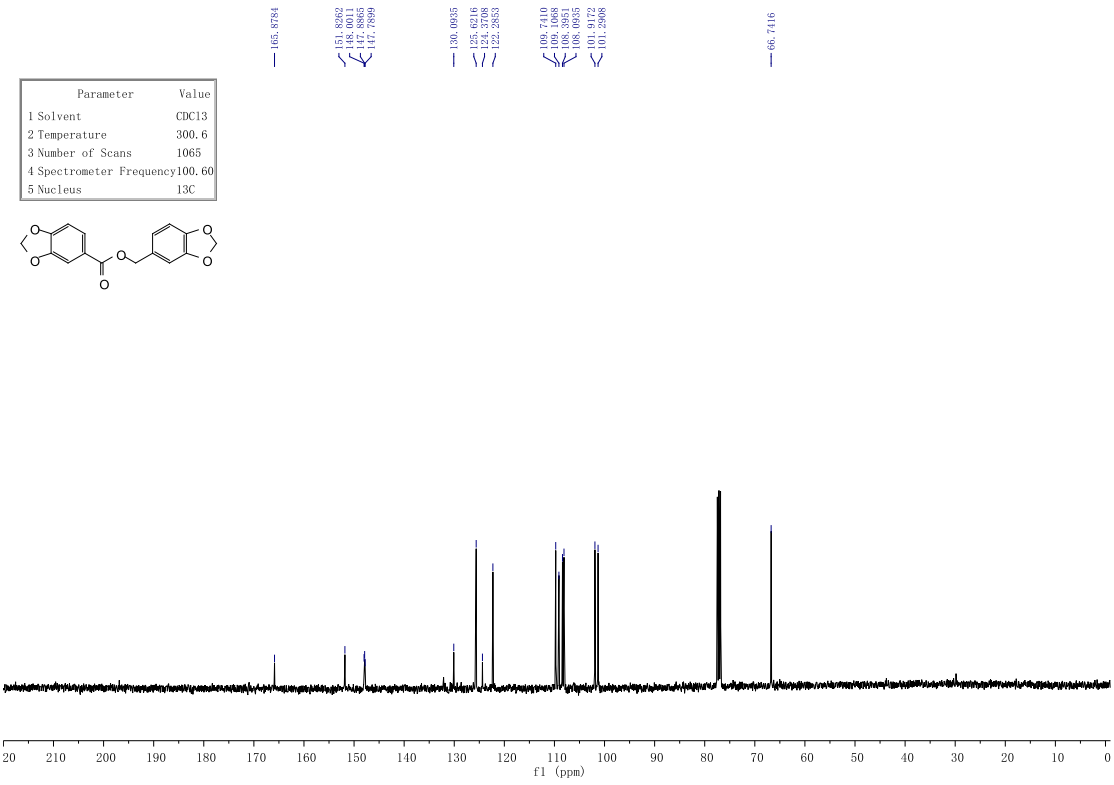
3-methoxybenzyl 3-methoxybenzoate, **2ag**



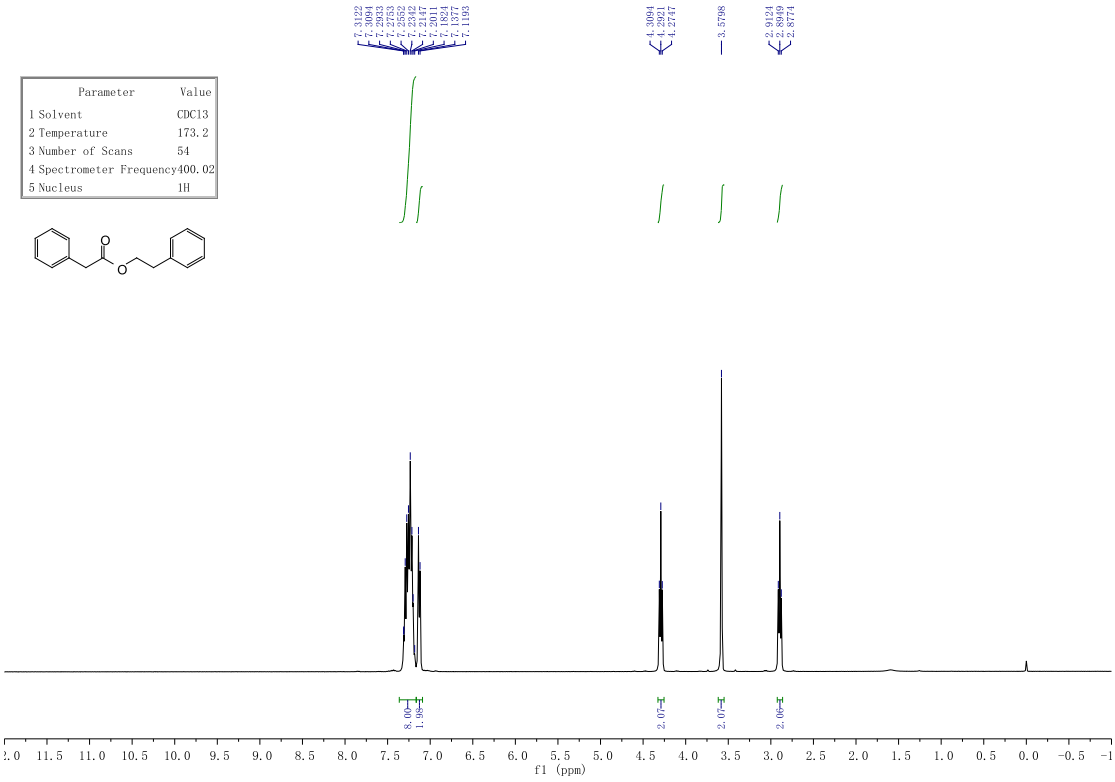


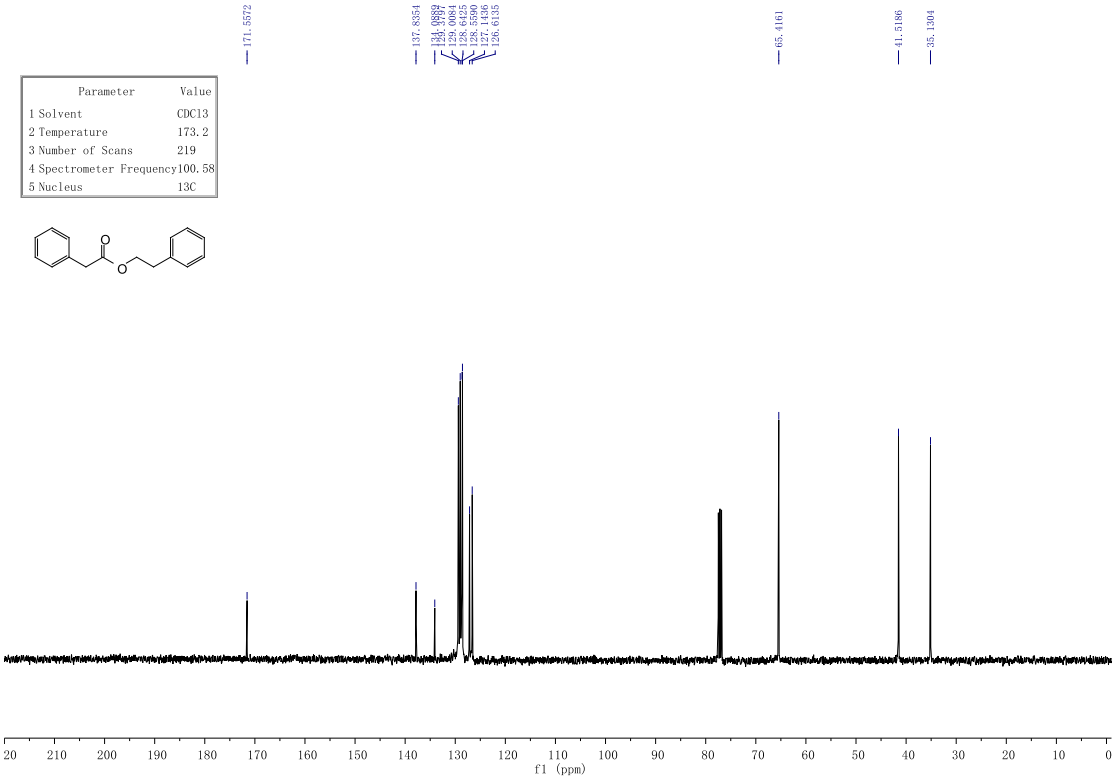
benzo[d][1,3]dioxol-5-ylmethyl benzo[d][1,3]dioxole-5-carboxylate, **2ak**



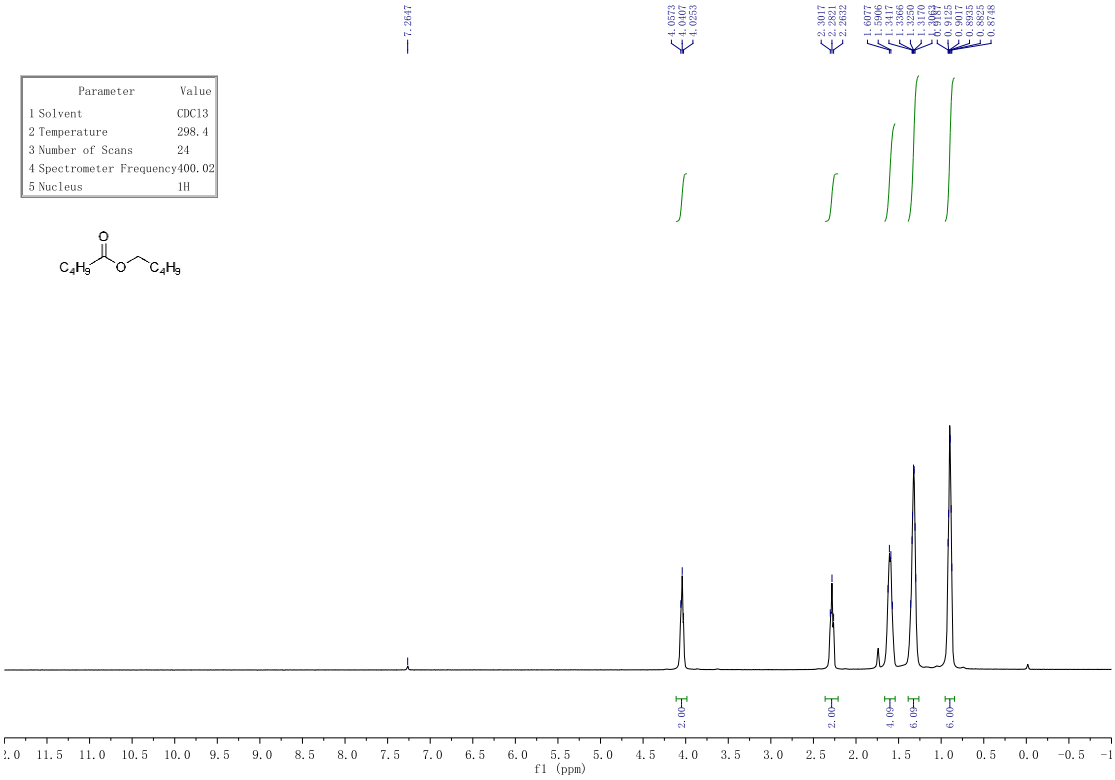


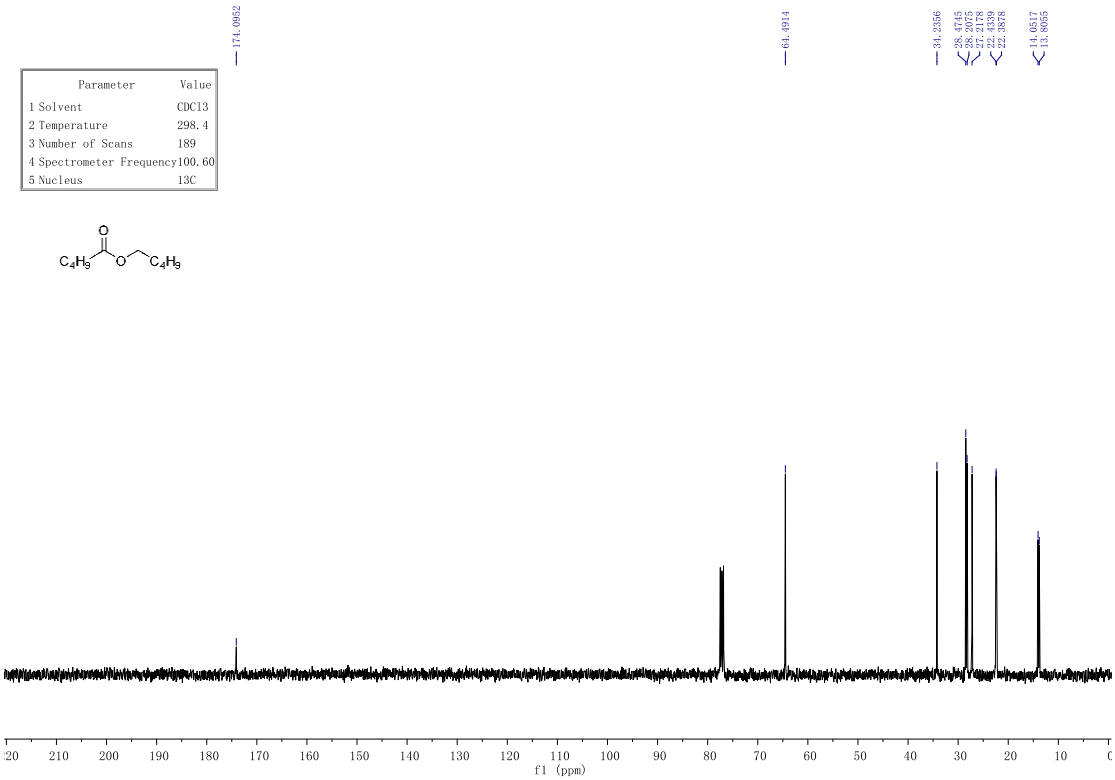
phenethyl 2-phenylacetate, **2am**



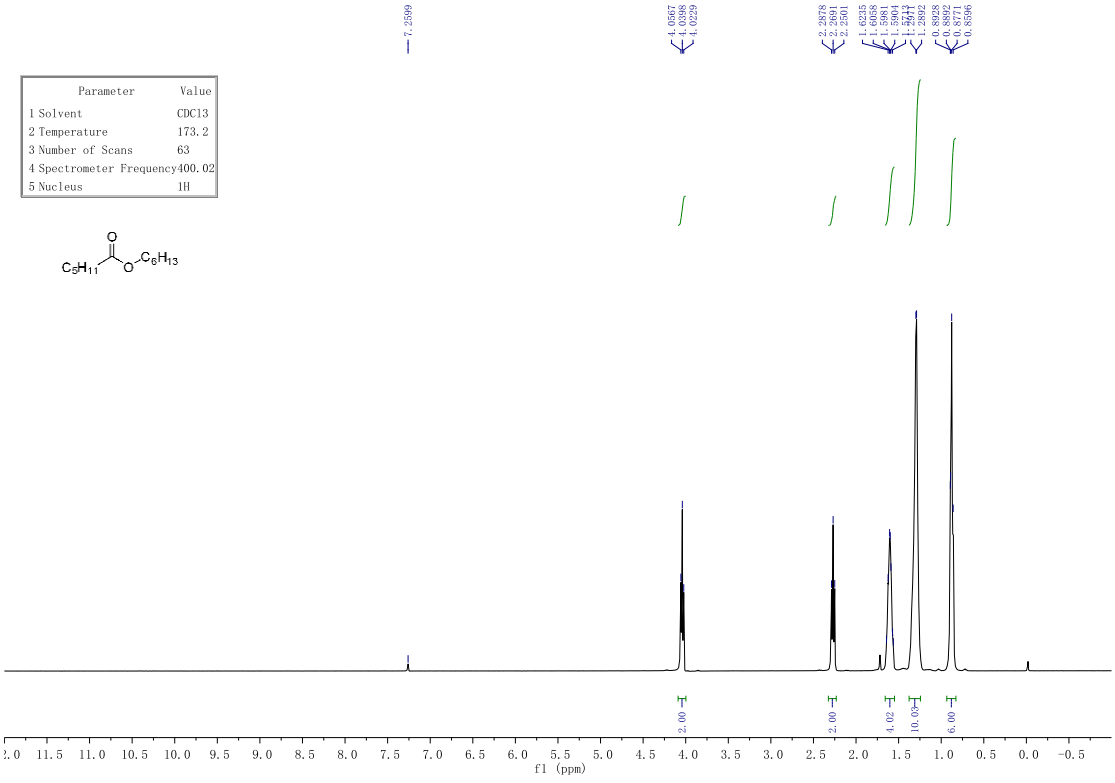


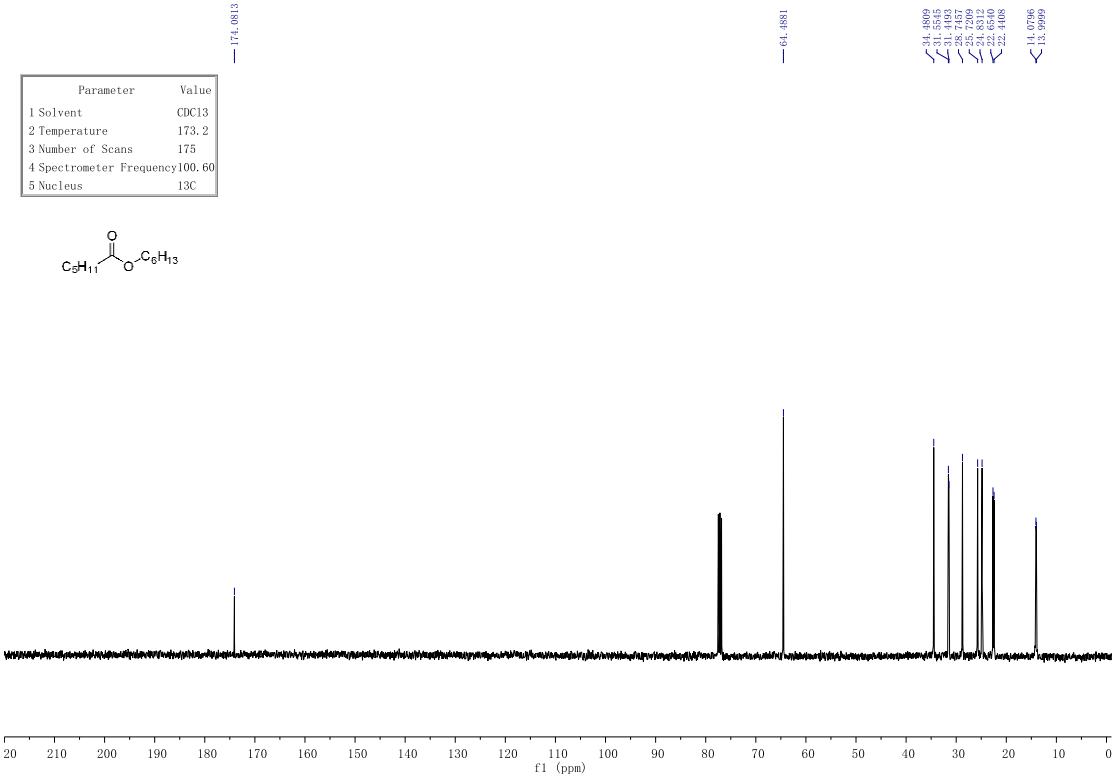
pentyl pentanoate, **2ap**



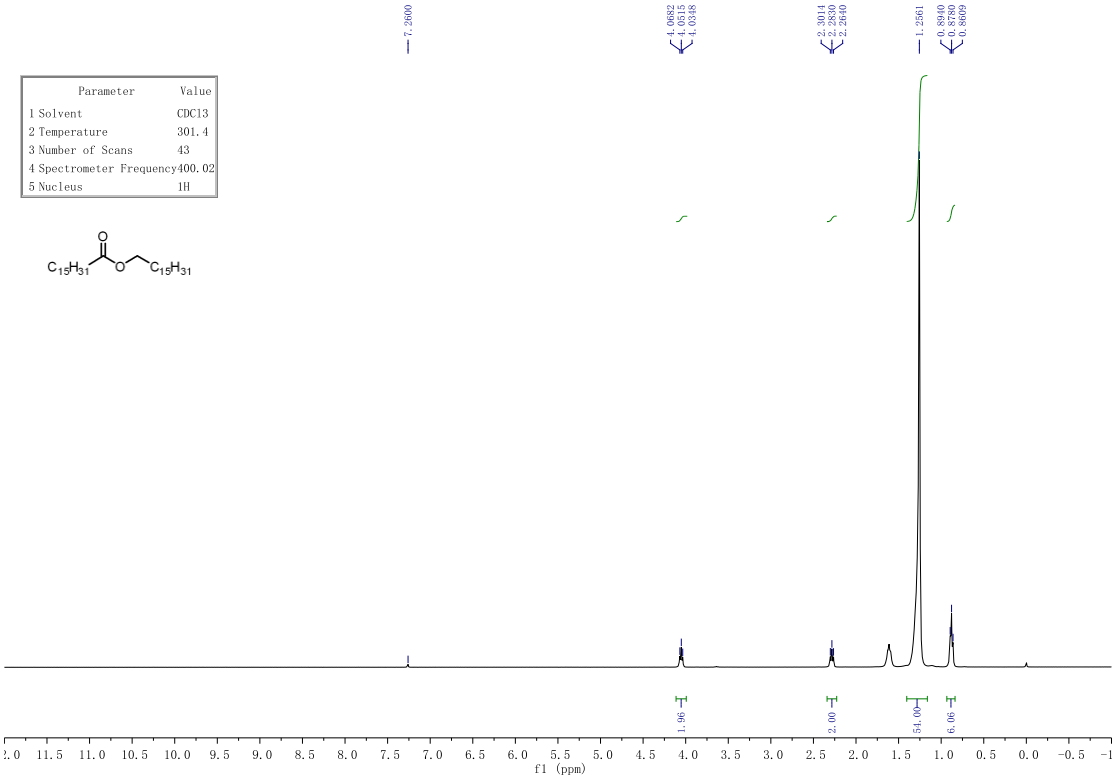


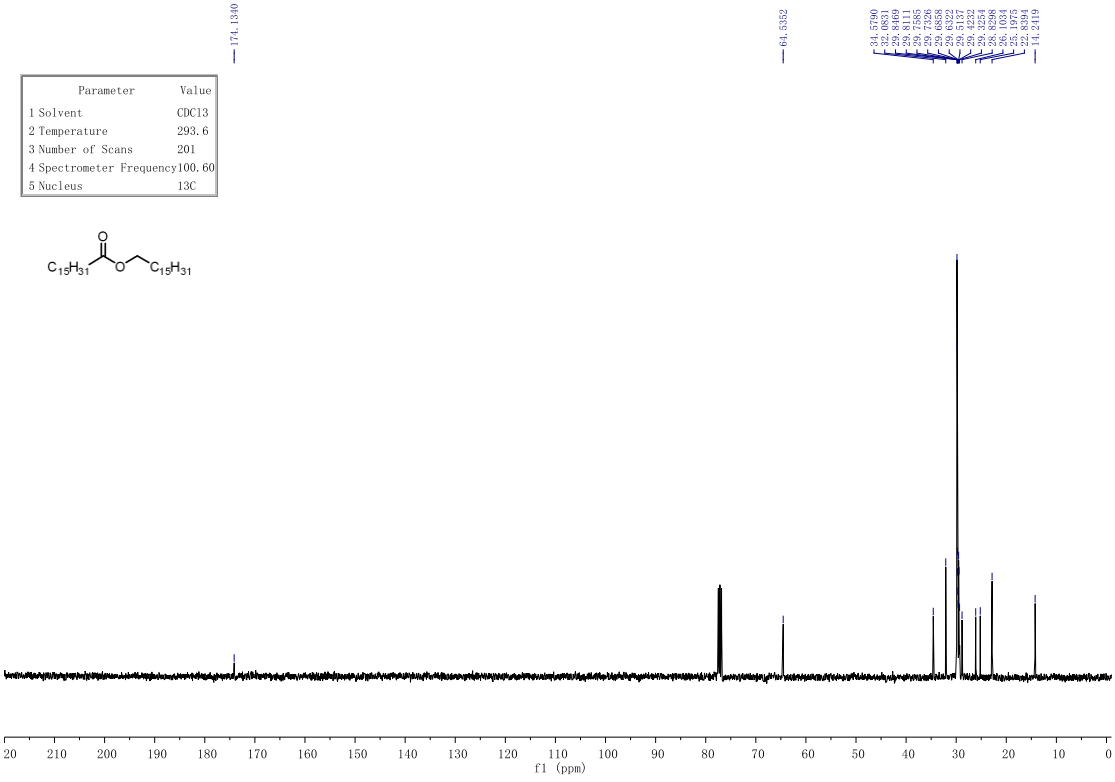
hexyl hexanoate, **2aq**



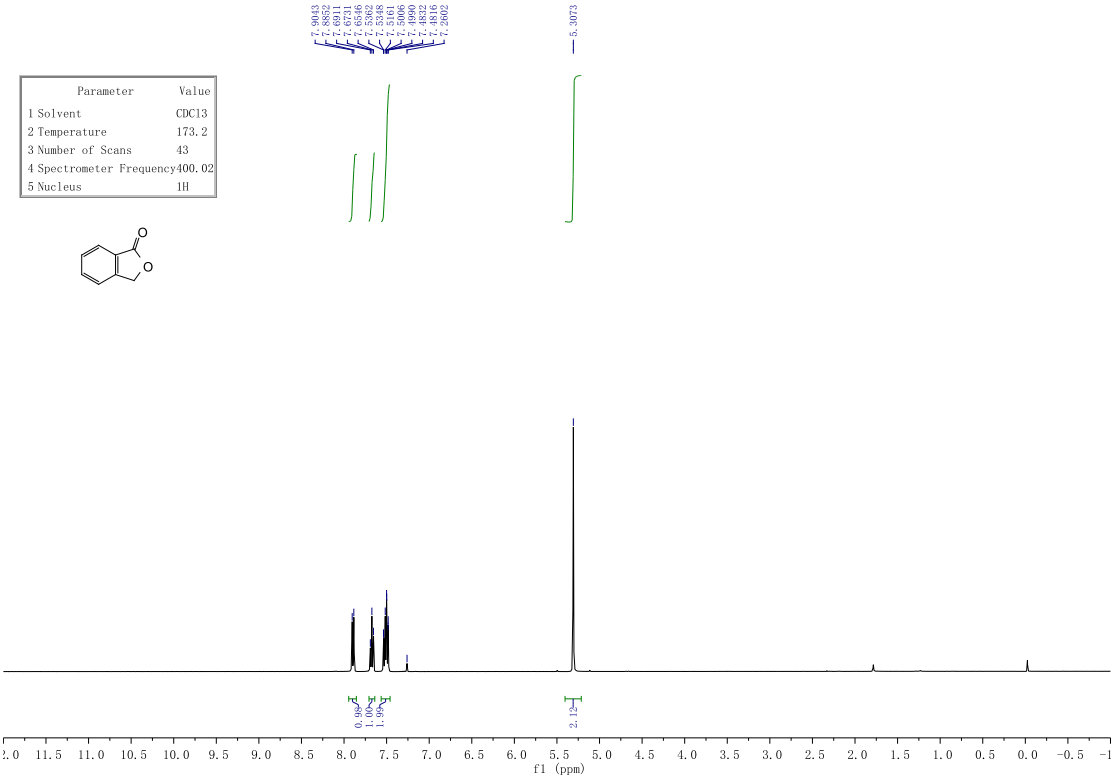


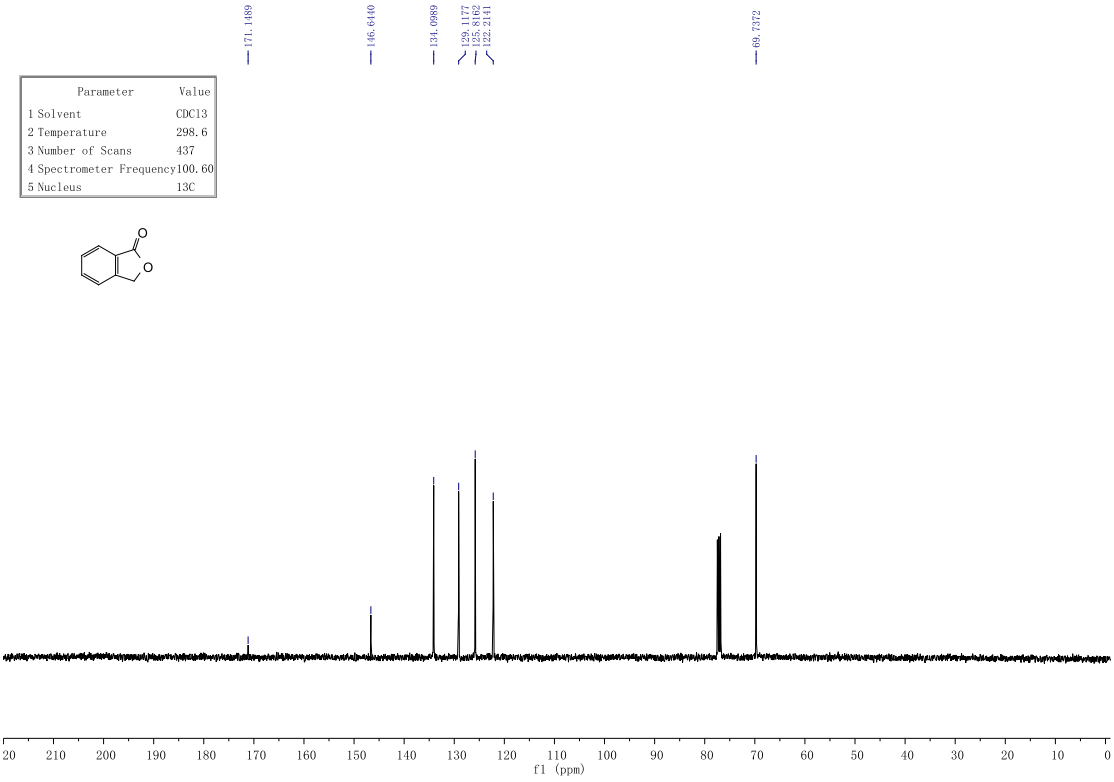
hexadecyl palmitate, **2ar**



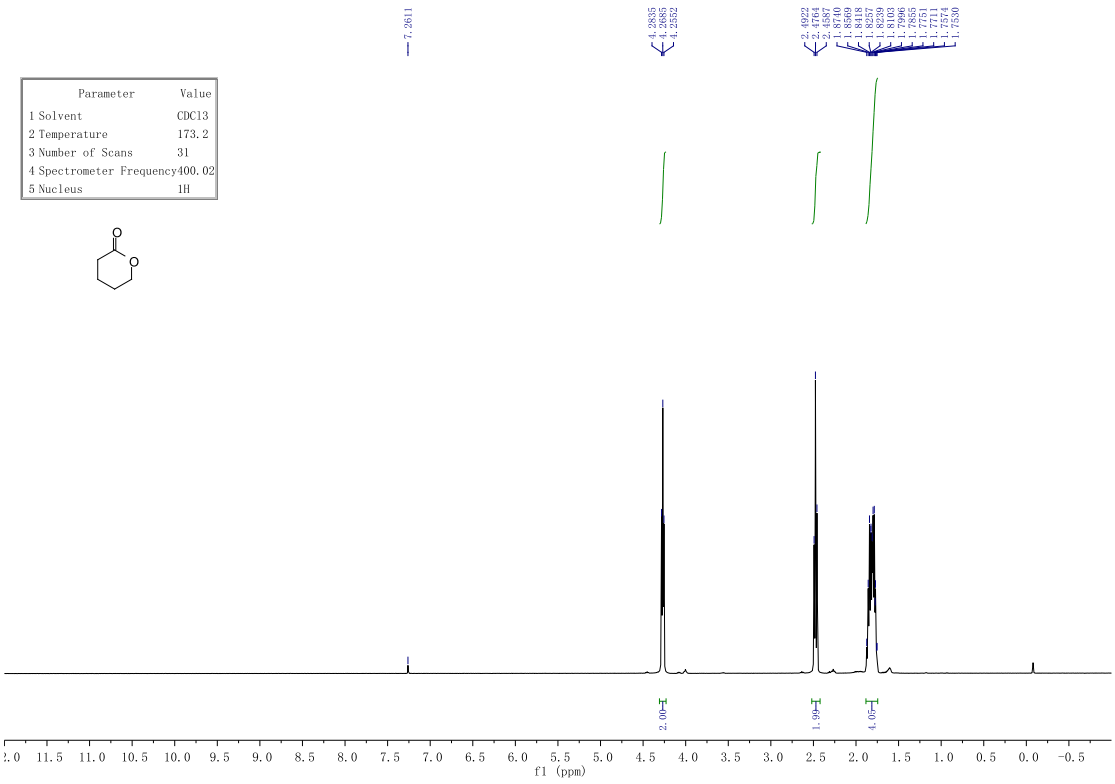


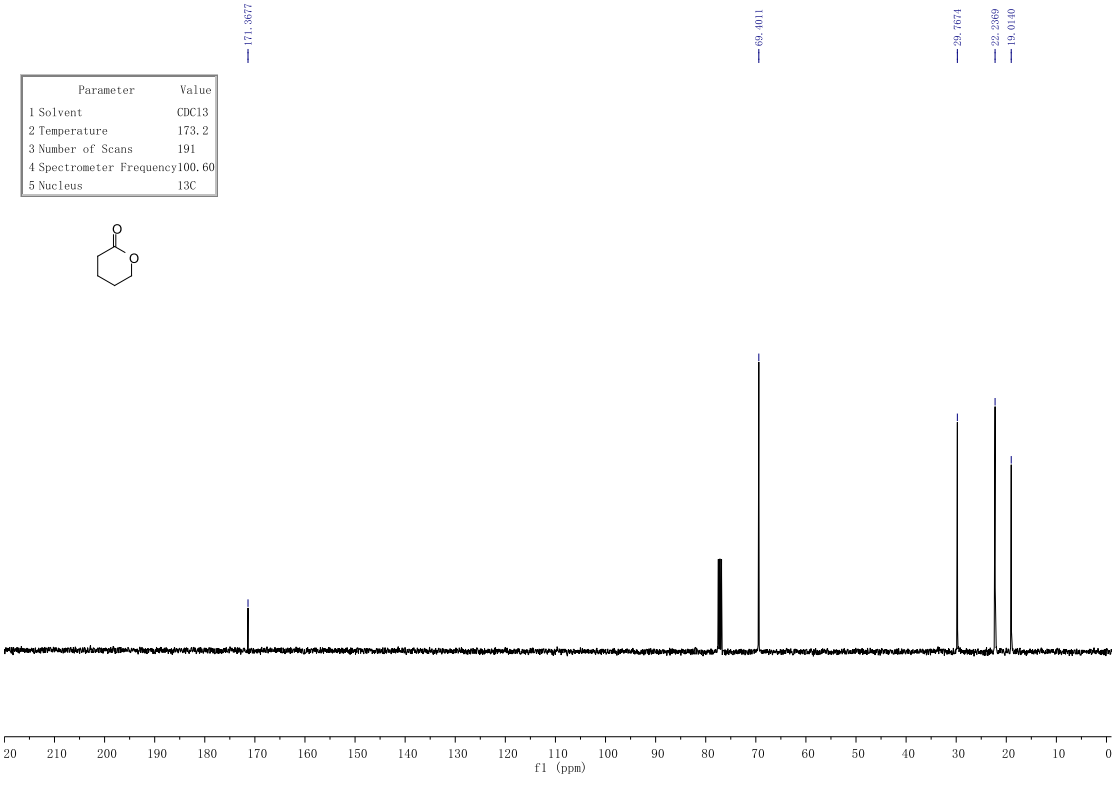
isobenzofuran-1(3H)-one, **2as**



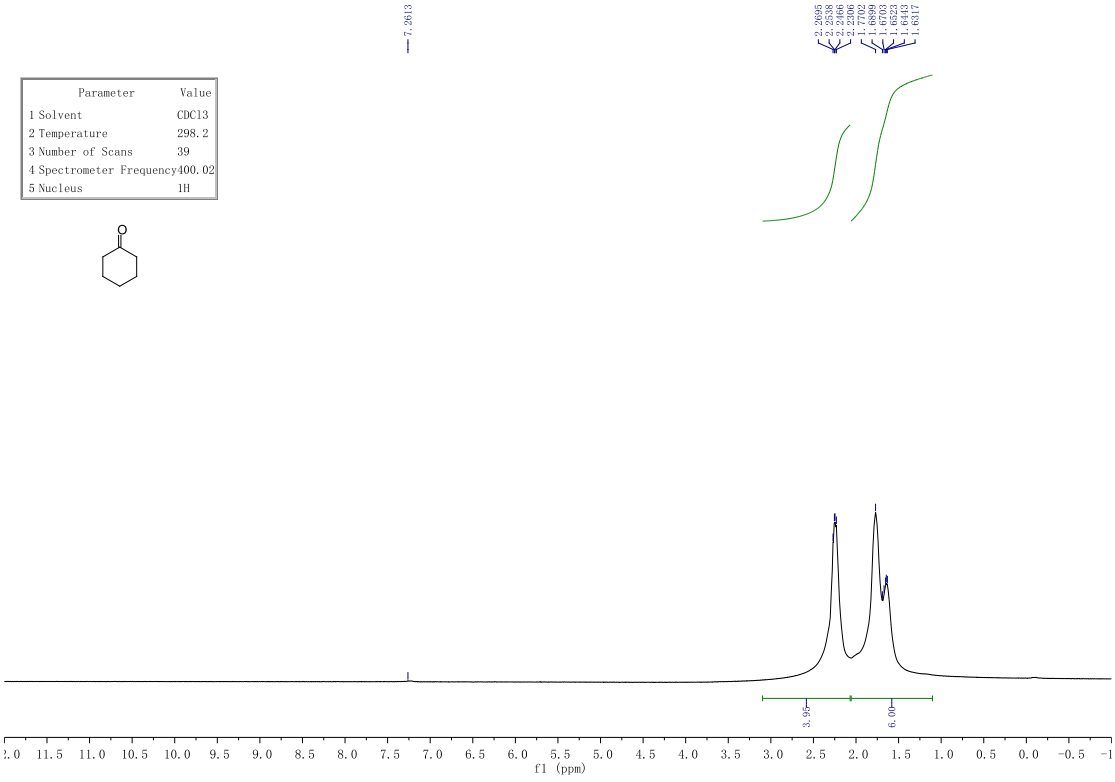


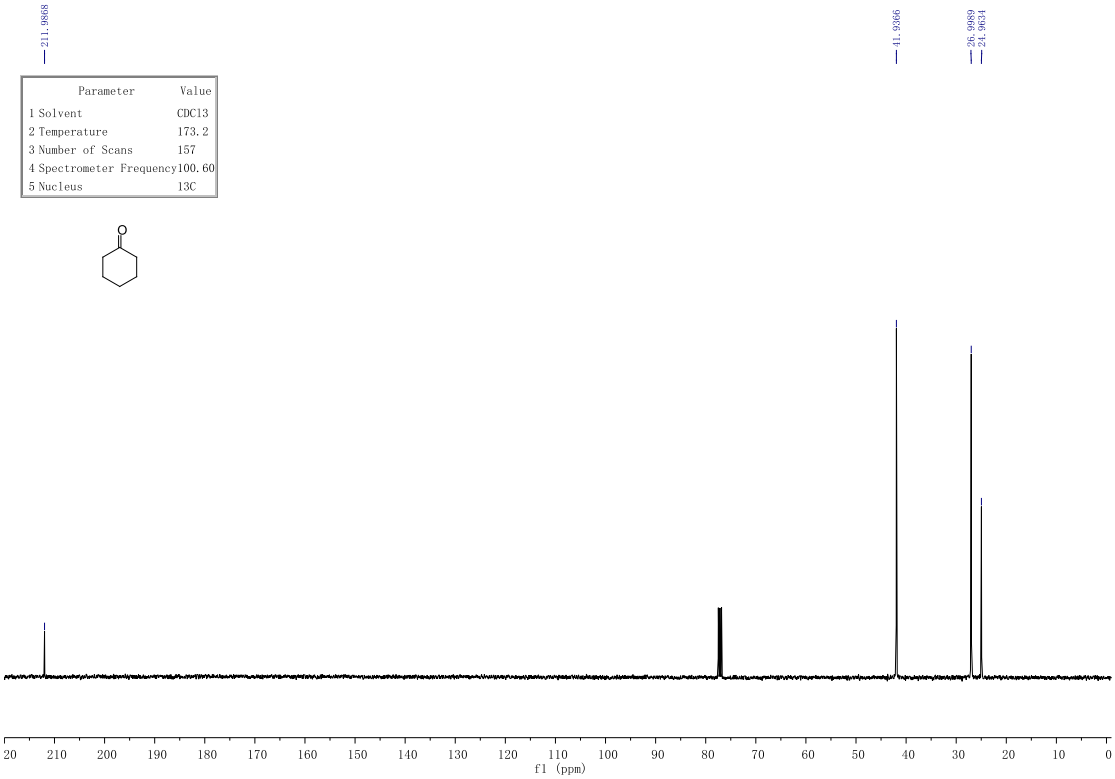
tetrahydro-2H-pyran-2-one, **2at**



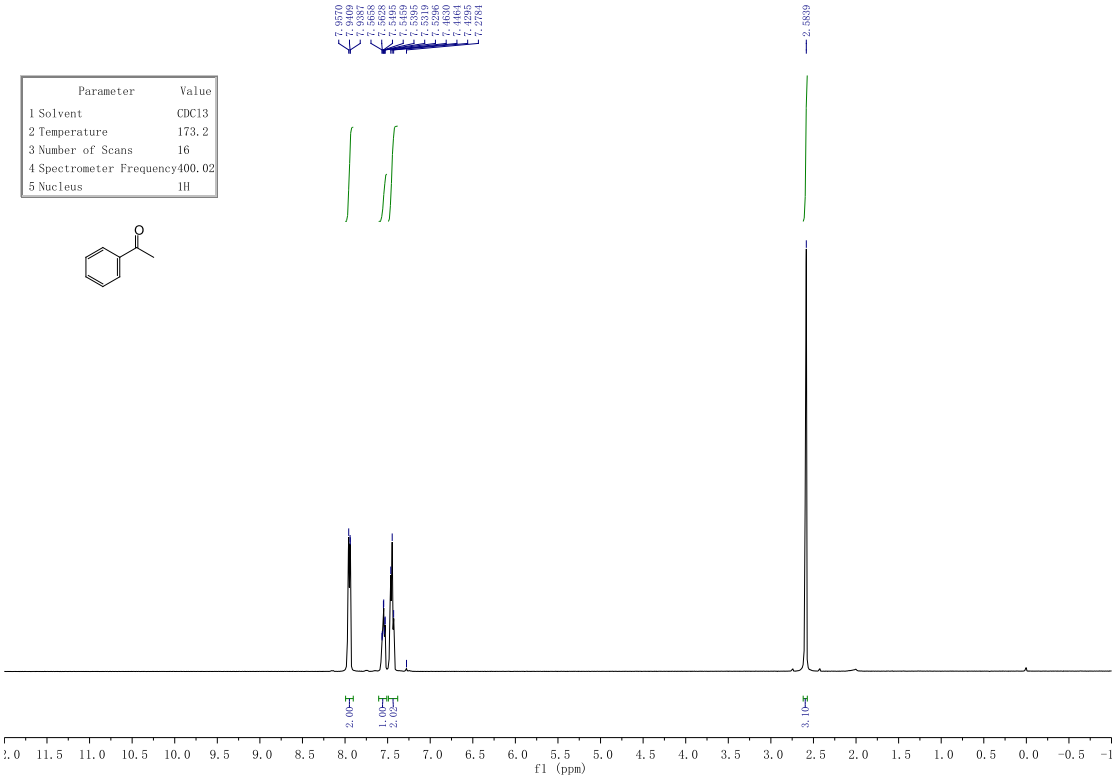


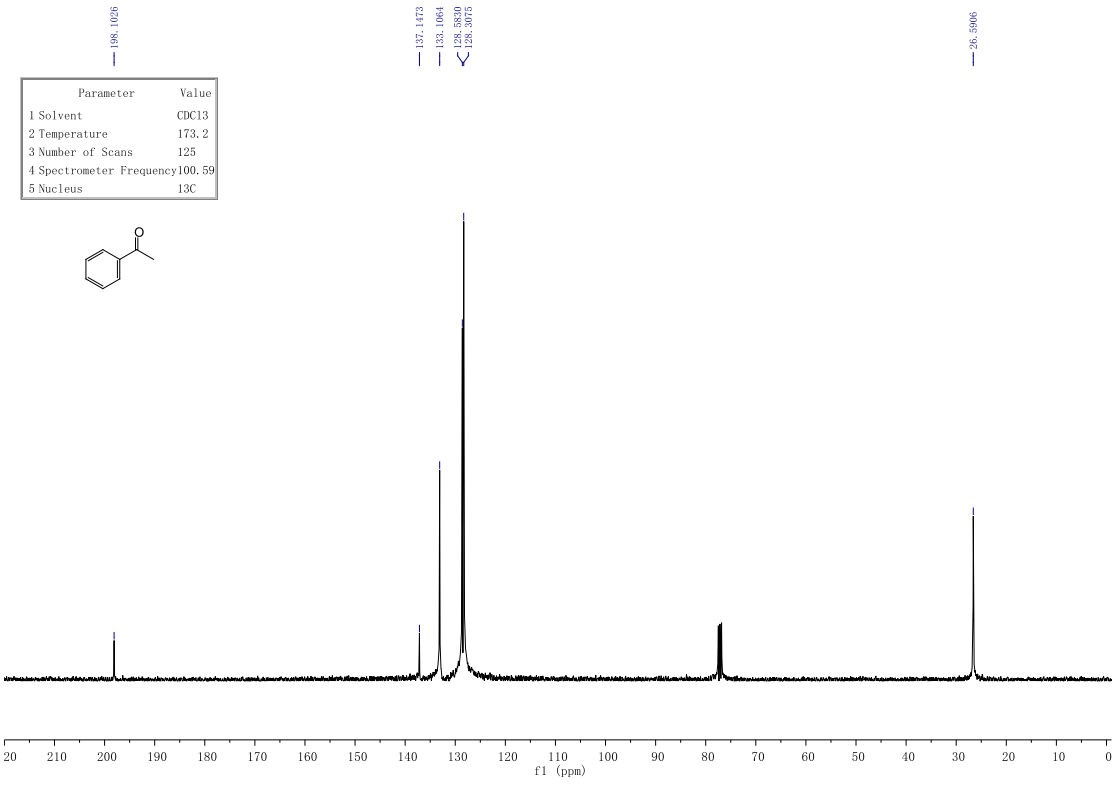
Cyclohexanone, **3a**



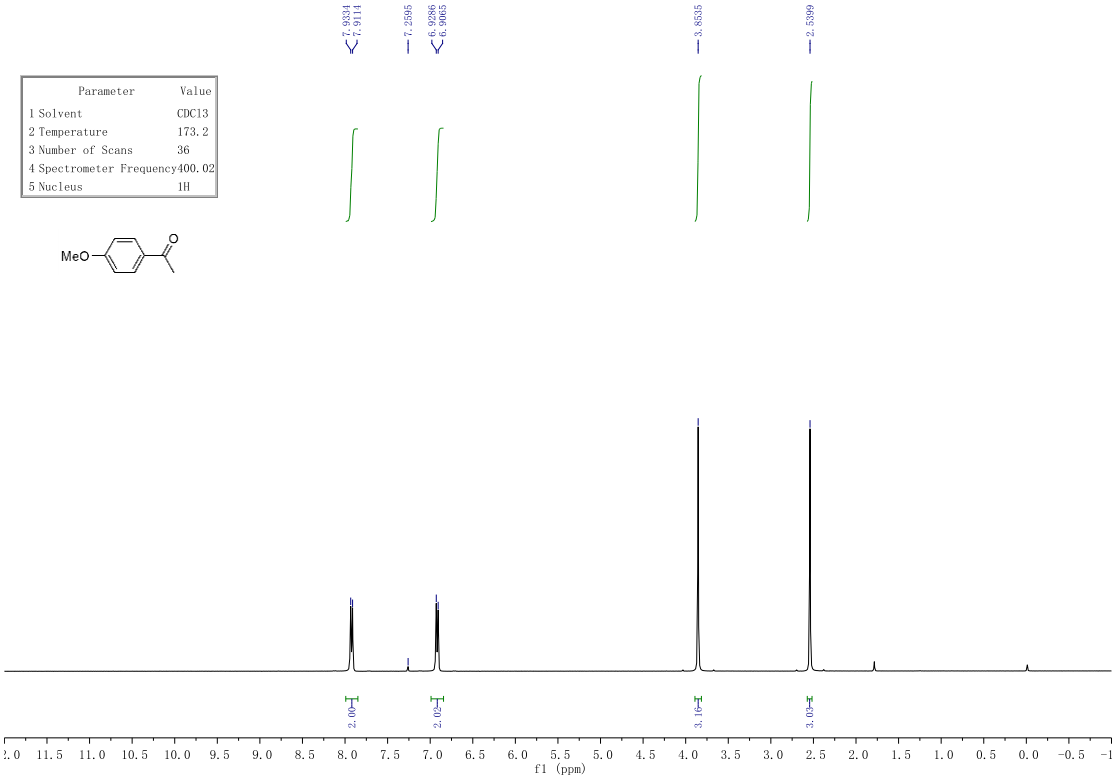


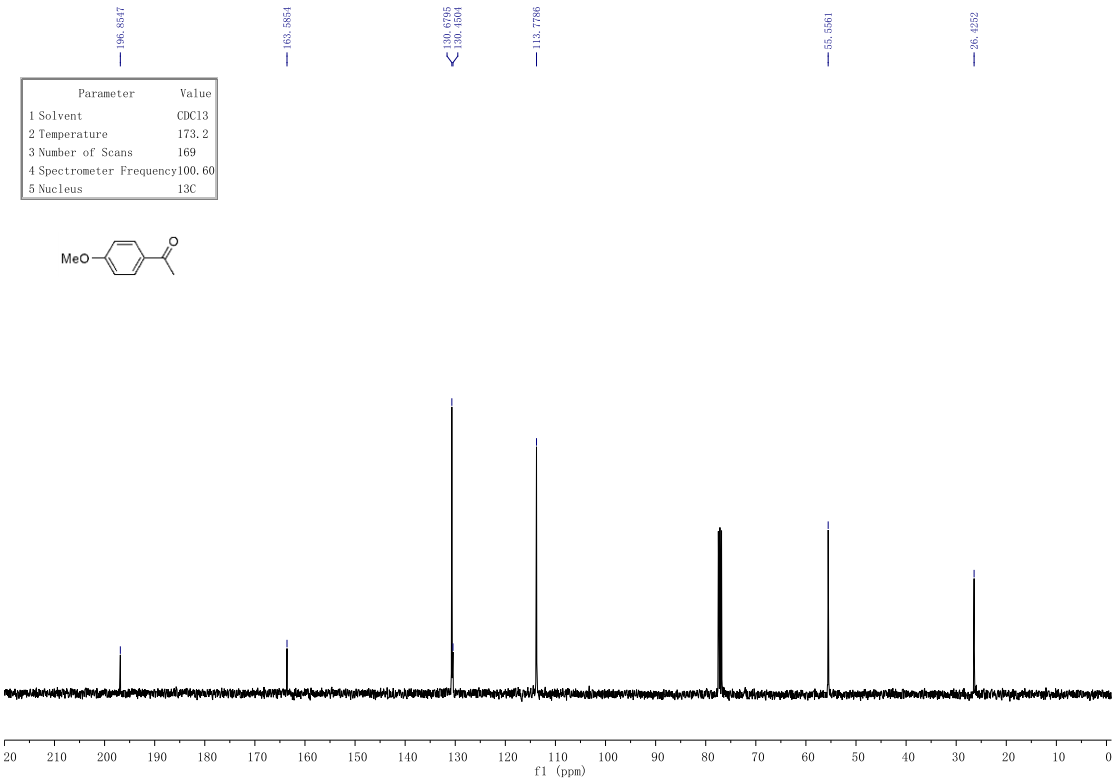
Acetophenone, **3b**



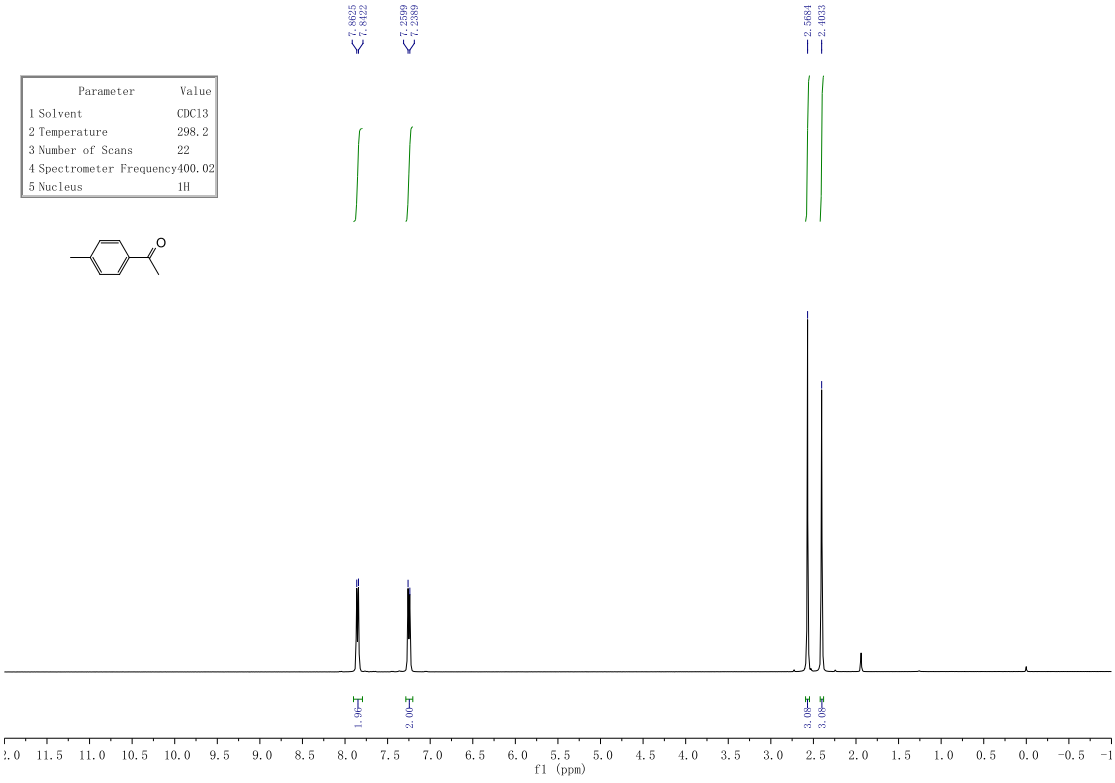


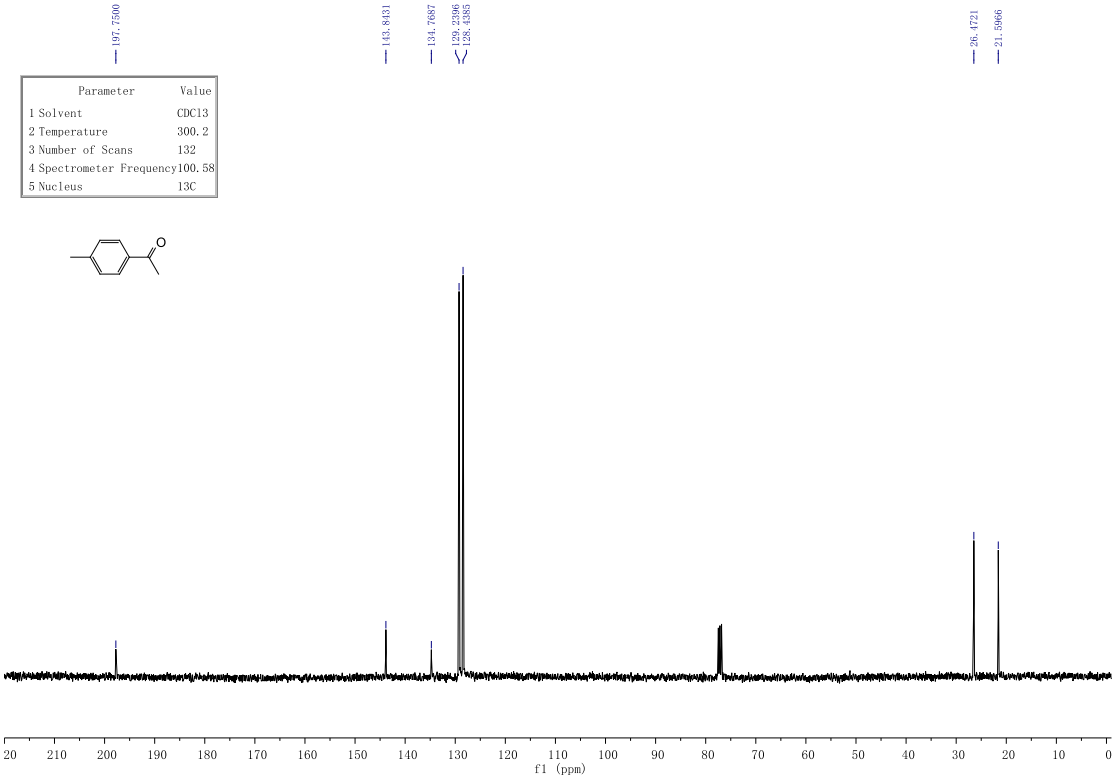
1-(4-methoxyphenyl)ethan-1-one, **3c**



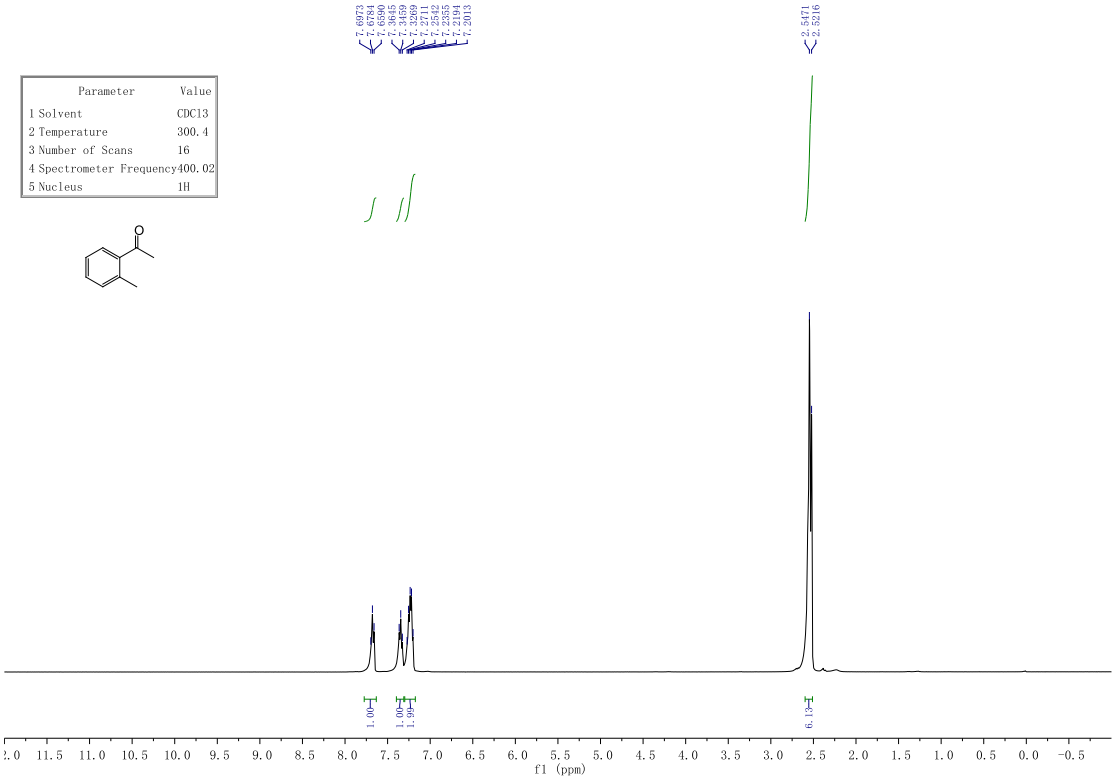


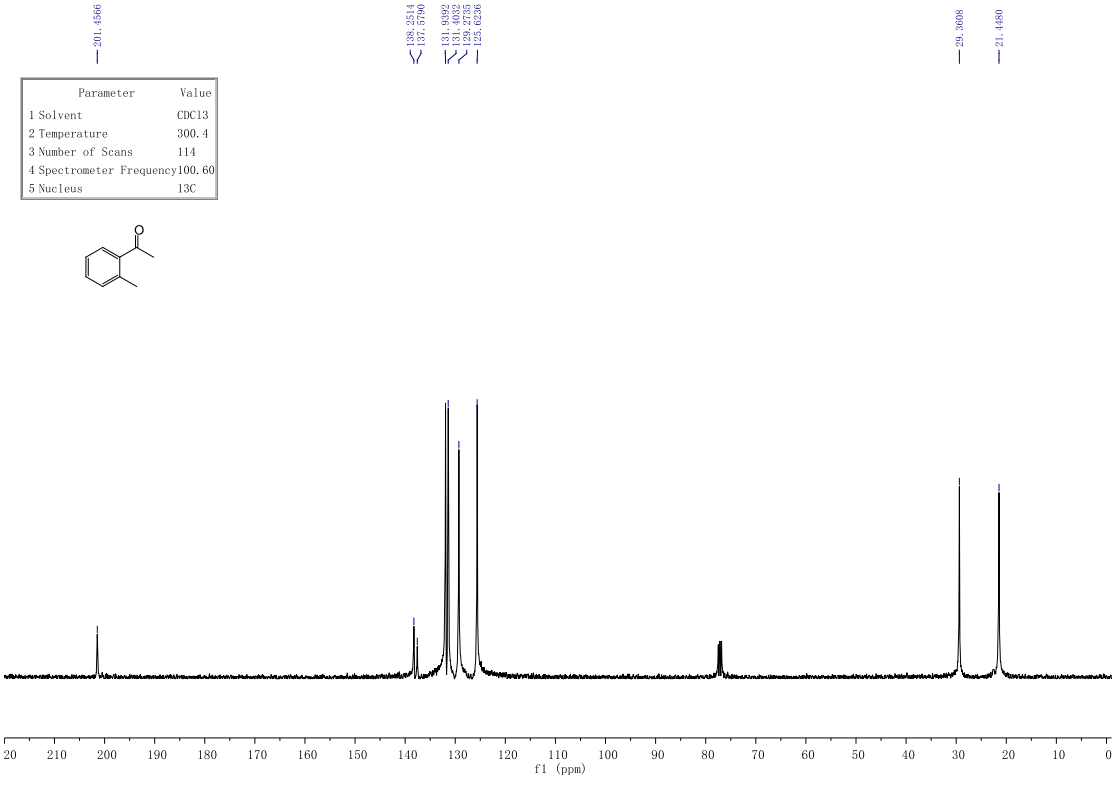
1-(p-tolyl)ethan-1-one, **3d**





1-(o-tolyl)ethan-1-one, **3e**





1-(4-bromophenyl)ethan-1-one, **3f**