Supporting Information

Stoichiometric couplings of methylarenes through visible-light-induced bromo radical formation from aryl halides

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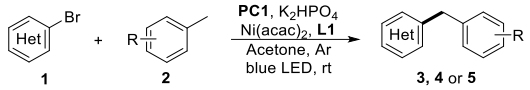
1. General Information

All reactions were carried out with magnetic stirring and in dried glassware. Standard syringe techniques were applied for transfer of dry solvents. All reagents and solvents were commercially available and used without any further purification unless specified. The reactions via general procedure was carried out under an atmosphere of argon unless otherwise noted. Column chromatography was performed using silica gel (200-300 mesh) or thin layer chromatography was performed using silica gel (GF254). ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker-AV (400 and 100 MHz, respectively) instrument using $CDCl_3$ as solvent. Mass spectra were measured on Agilent 5975 GC-MS instrument (EI). High-resolution mass spectra (ESI) were obtained with the Thermo Scientific LTQ Orbitrap XL mass spectrometer. The structures of known compounds were further corroborated by comparing their ^{1}H NMR, ^{13}C NMR data and HRMS data with those in literature. Melting points were measured with a YUHUA X-5 melting point instrument and were uncorrected.

2. Experiment Section

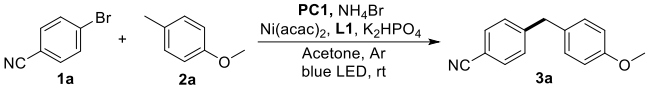
2.1 Typical experimental procedure for the arylation



To an overdried Schlenk tube with a stir bar was added **1** (0.2 mmol), **2** (0.4 mmol, 2.0 eqiuv), **PC1** (0.004 mmol, 2 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in argon atmosphere (1 atm) under 35 W blue LED light for 24 h until complete consumption of starting material as monitored by TLC and GC-MS analysis. After the reaction was finished, the reaction mixture was washed with brine. The aqueous phase was re-extracted with EtOAc (3×10 mL). The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum. The residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 15 : 1 to 5 : 1) to afford the desired products **3, 4** or **5**.

2.2 Optimization of reaction conditions

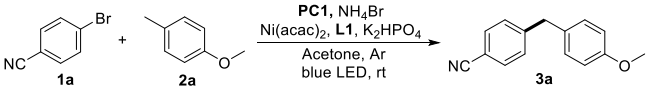
Table S1. Screening of amount **2a** [a]



Entry	Variation from standard conditions	Yield (%) [b]
1	10 equiv	92
2	2 equiv	88

[a] Reaction conditions: **1a** (0.2 mmol), **2a**, **PC1** (0.004 mmol, 2 mol%), NH_4Br (0.1 mmol, 50 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in Ar atmosphere (1 atm) under 35 W blue LED light for 24 h at room temperature. [b] Isolated yields.

Table S2. Control experiments [a]

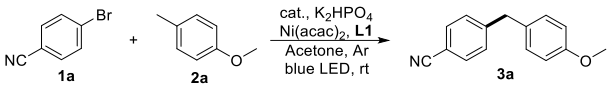


Entry	Variation from standard conditions	Yield (%) [b]
1	none	88
2	No **PC1**	0
3	No light	0
4	No $Ni(acac)_2$	0
5	No **L1**	0

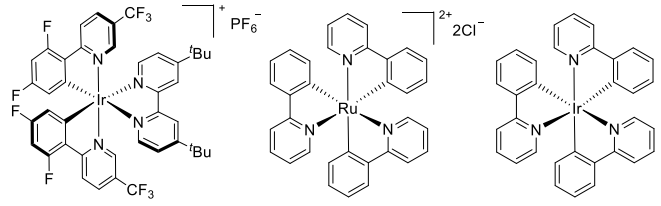
6	No K_2HPO_4	0
7	No NH_4Br	87

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol, 2 eqiuv), **PC1** (0.004 mmol, 2 mol%), NH_4Br (0.1 mmol, 50 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in Ar atmosphere (1 atm) under 35 W blue LED light for 24 h at room temperature. [b] Isolated yields.

Table S3. Screening of phototcatalyst [a]



Entry	Photocatalyst	Yield (%) [b]
1	**PC1**	87
2	**PC2**	0
3	**PC3**	0
4	**PC4**	0
5	**PC5**	0
6	**PC6**	58
7	**PC1**[c]	70



[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol, 2 eqiuv), photocatalyst (0.004 mmol, 2 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in Ar atmosphere (1 atm) under 35 W blue LED light for 24 h at room temperature. [b] Isolated yields. [c] PC1 (0.002 mmol, 1 mol%).

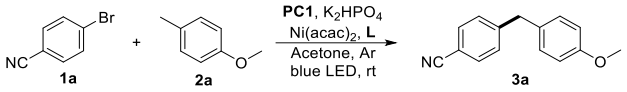
Table S4. Screening of "Ni" catalyst [a]

Entry	"Ni" catalyst	Yield (%)[b]
1	$Ni(acac)_2$	87

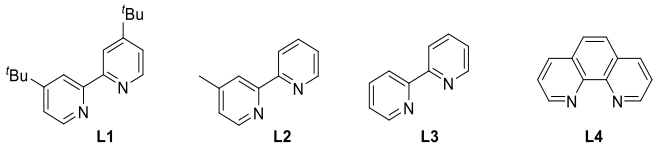
2	NiF_2	68
3	$NiCl_2$	83
4	$NiBr_2$	72
5	NiI_2	0
6	$Ni(OTf)_2$	58
7	$Ni(PPh_3)_3Cl_2$	86
8	$Ni(OAc)_2$	61
9	$Ni(COD)_2$	63

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol, 2.0 eqiuv), **PC1** (0.004 mmol, 2 mol%), "Ni" catalyst (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in Ar atmosphere (1 atm) under 35 W blue LED light for 24 h at room temperature. [b] Isolated yields.

Table S5. Screening of ligand [a]

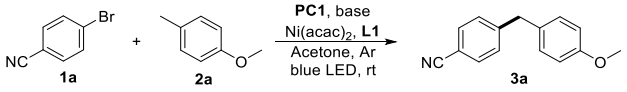


Entry	ligand	Yield (%) [b]
1	**L1**	87
2	**L2**	77
3	**L3**	80
4	**L4**	67



[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol, 2.0 eqiuv), **PC1** (0.004 mmol, 2 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in Ar atmosphere (1 atm) under 35 W blue LED light for 24 h at room temperature. [b] Isolated yields.

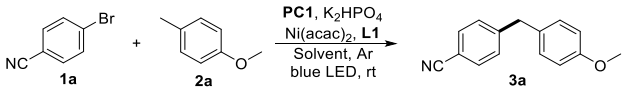
Table S6. Screening of base [a]



Entry	base	Yield (%) [b]
1	K_2HPO_4	87
2	Cs_2CO_3	trace
3	KH_2PO_4	59
4	^{t}BuONa	0
5	pyridine	42
6	Et_3N	0
7	K_2CO_3	36

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol, 2.0 eqiuv), **PC1** (0.004 mmol, 2 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), base (0.4 mmol, 2.0 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in Ar atmosphere (1 atm) under 35 W blue LED light for 24 h at room temperature. [b] Isolated yields.

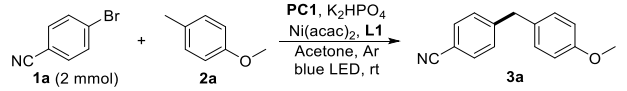
Table S7. Screening of solvent [a]



Entry	solvent	Yield (%) [b]
1	acetone	87
2	CH_3CN	52
3	DMA	12
4	DMF	trace
5	1,4-dioxane	35
6	DCM	trace

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol, 2.0 eqiuv), **PC1** (0.004 mmol, 2 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), solvent (3 mL). Then the mixture was stirred at room temperature in Ar atmosphere (1 atm) under 35 W blue LED light for 24 h at room temperature. [b] Isolated yields.

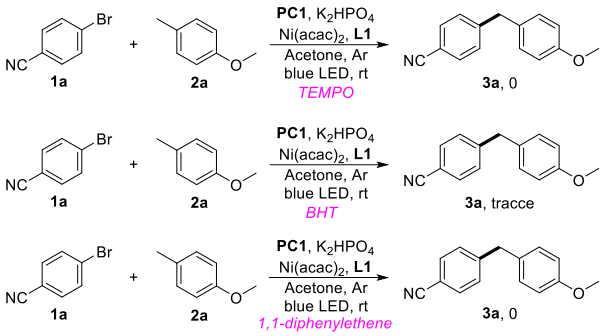
2.3 Scale-up experiment



A 50 mL Schlenk tube was added **1a** (2.0 mmol), **2a** (4 mmol, 2.0 eqiuv), **PC1** (0.04 mmol, 2 mol%), $Ni(acac)_2$ (0.04 mmol, 2 mol%), **L1** (0.04 mmol, 2 mol%), K_2HPO_4 (4 mmol, 2.0 equiv), acetone (30 mL). Then the mixture was stirred at room temperature in argon atmosphere (1 atm) under 35 W blue LED light for 30 h. After the reaction was finished, the reaction mixture was washed with brine. The aqueous phase was re-extracted with EtOAc (3×10 mL). The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum. The residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 10 : 1) to afford the desired products **3a** in 71% yield.

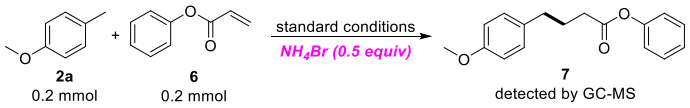
3. Mechanistic studies

3.1 Radical trapping experiments

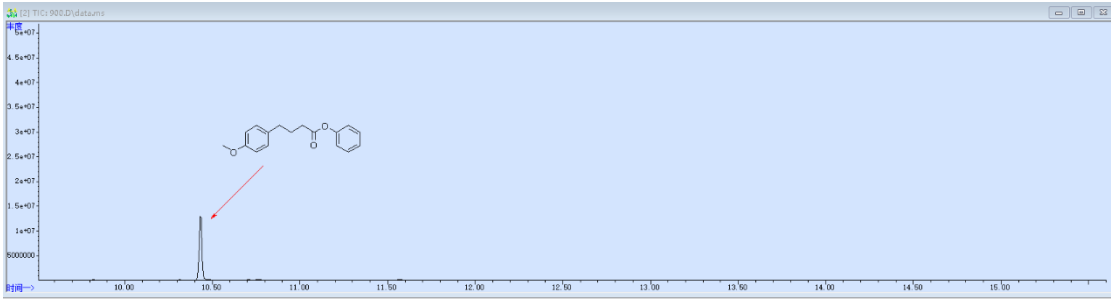


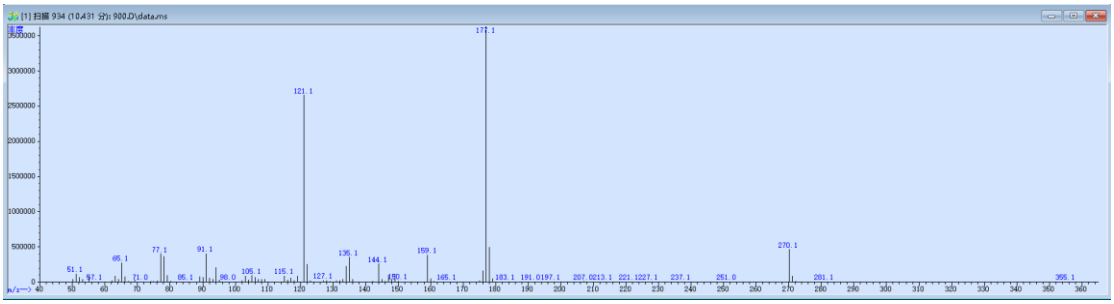
Three reactions of radical trapping experiments were performed. To an overdried Schlenk tube with a stir bar was added TEMPO (3.0 equiv, 0.6 mmol), BHT (3 equiv, 0.6 mmol) or 1,1-diphenylethene (3 equiv, 0.6 mmol), **1a** (0.2 mmol), **2a** (0.4 mmol, 2.0 eqiuv), **PC1** (0.004 mmol, 2 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in argon atmosphere (1 atm) under 35 W blue LED light for 24 h. The benzylation were completely quenched and no benzylation products were detected.

3.2 Benzyl radical trapping experiments



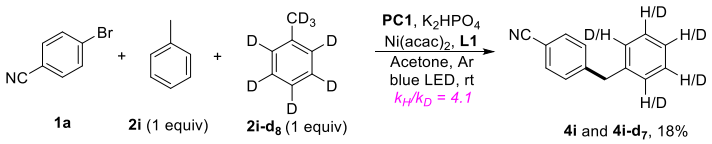
To a Schlenk tube was added **1a** (0.2 mmol), **6** (0.2 mmol, 1.0 eqiuv), **PC1** (0.004 mmol, 2 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), NH_4Br (0.1 mmol, 0.5 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in argon atmosphere (1 atm) under 35 W blue LED light for 24 h. Phenyl 4-(4-methoxyphenyl)butanoate (**7**) could be detected by GC-MS.

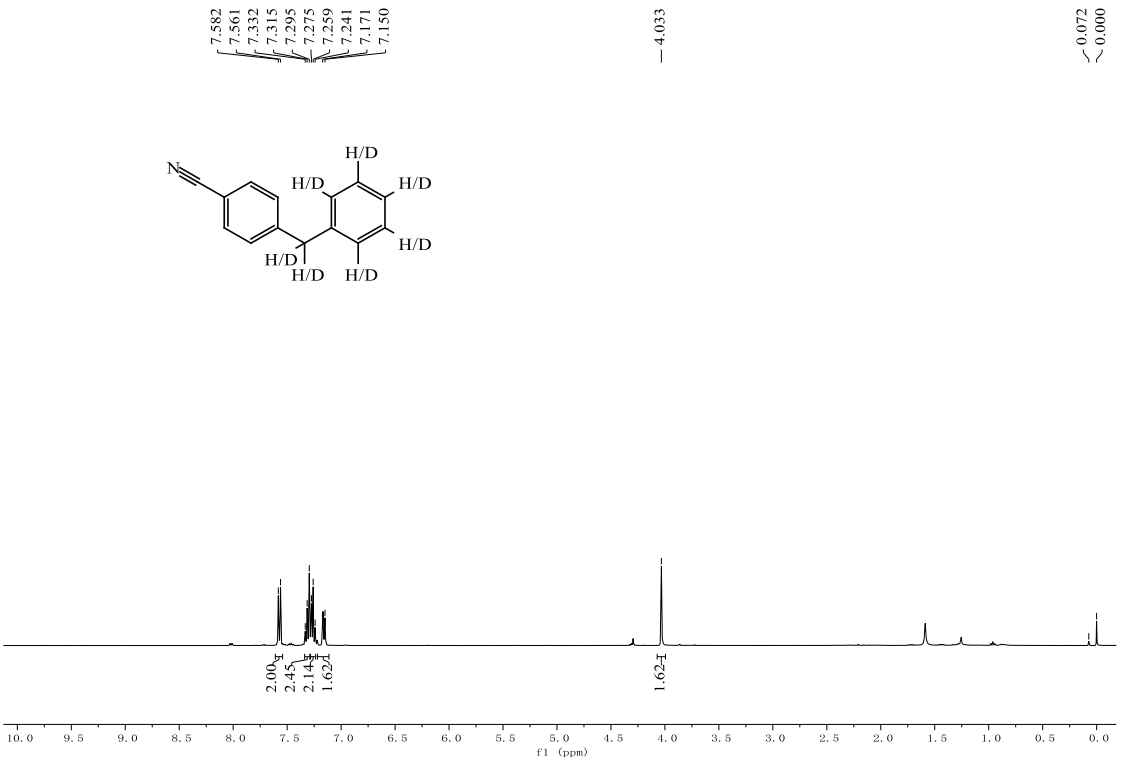




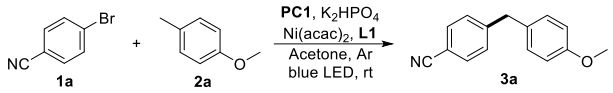
3.3 Kinetic isotope effect

To a Schlenk tube was added toluene (**2i**, 1 equiv), and toluene-d_8 (**2i-d$_8$**, 1.0 equiv), **1a** (0.2 mmol), **PC1** (0.004 mmol, 2 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), acetone (3 mL). Then the mixture was stirred at room temperature in Ar atmosphere (1 atm) under 35 W blue LED light for 4 h. The aqueous phase was re-extracted with EtOAc (3×10 mL). The combined organic extracts were dried over Na_2SO_4 and concentrated in vacuum. The residue was purified by silica gel flash column chromatography (hexane/ethyl acetate = 10 : 1) to afford product **4i and 4i-d$_7$** in 18% yield.

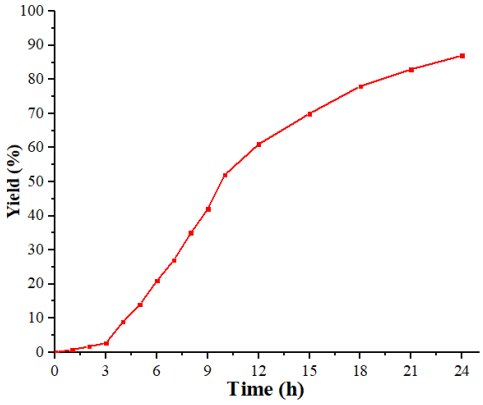




3.4 Time course reaction



To an overdried Schlenk tube with a stir bar was added **1a** (0.2 mmol), **2a** (0.4 mmol, 2.0 eqiuv), **PC1** (0.004 mmol, 2 mol%), $Ni(acac)_2$ (0.004 mmol, 2 mol%), **L1** (0.004 mmol, 2 mol%), K_2HPO_4 (0.4 mmol, 2.0 equiv), acetone (3 mL). Dodecane (46 μL, 0.2 mmol, 1.0 equiv.) was added as an internal standard. Then the mixture was stirred at room temperature in argon atmosphere (1 atm) under 35 W blue LED light for 24 h. 100 μL of the reaction mixture was taken out at 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 12, 15, 18, 21, 24 h. The yield of **3a** was determined by GC-MS with dodecane as an internal standard.

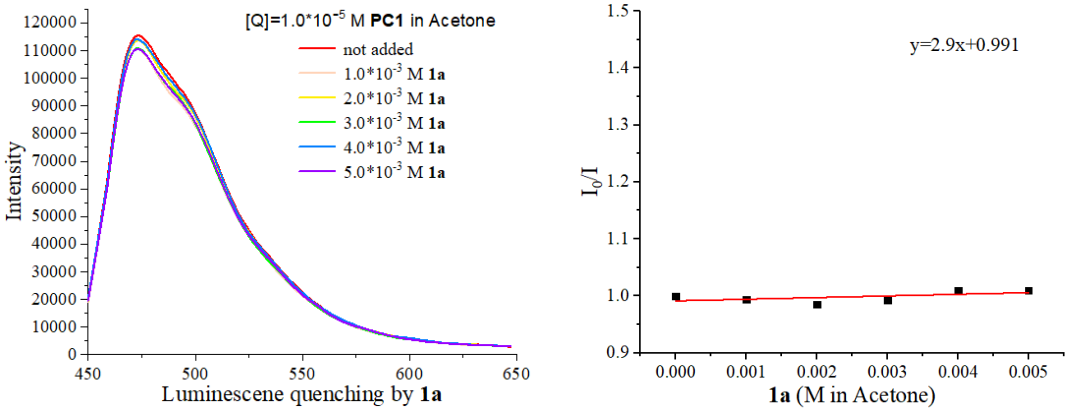


3.5 Stern–Volmer Quenching[1]

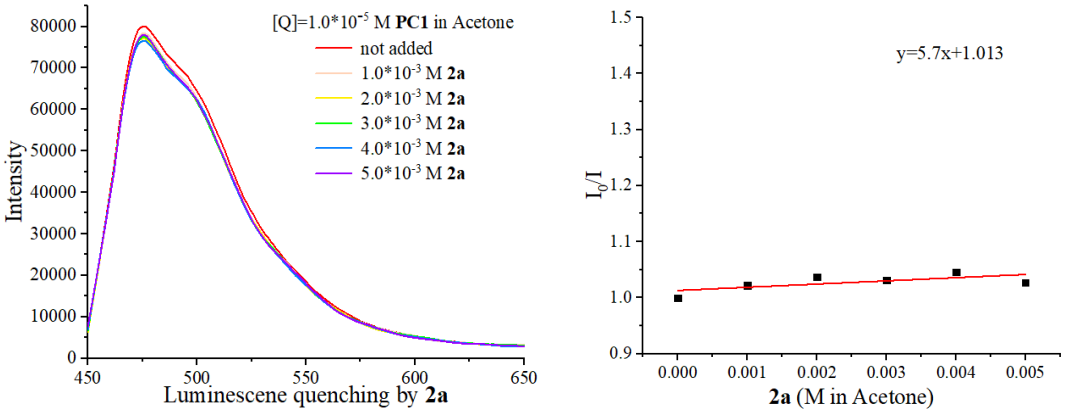
Formulation solution: 4-Bromobenzonitrile (**1a**, 452.3 mg) was dissolved in acetone in a 25 mL volumetric flask to set the concentration to be 0.1 M. 1-methoxy-4-methylbenzene (**2a**, 315 μL) was dissolved in acetone in a 25 mL volumetric flask to set the concentration to be 0.5 M. $Ni(acac)_2$ (6.4 mg) was dissolved in acetone in a 5 mL volumetric flask to set the concentration to be 0.05 M. **L1** (6.7 mg) was dissolved in acetone in a 5 mL volumetric flask to set the concentration to be 0.05 M. Photocatalyst $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (2.8 mg) was dissolved in acetone (25.0 mL) to set the concentration to be 0.1 mM.

Experimental procedure: The resulting 0.1 M solution (50 μL) was added to cuvette to obtain different concentrations of catalyst solution. This solution was then diluted to a volume of 2.0 mL by adding further solvent (acetone) to prepare a 2.5 μM solution. The resulting mixture was sparged with nitrogen for 3 minutes and then irradiated at 425 nm. Fluorescence emission spectra were recorded (3 trials per sample). Into this solution, 20.0 μL of a 4-bromobenzonitrile solution was successively added and uniformly stirred, and the resulting mixture was bubbled with nitrogen for 3 minutes and irradiated at 375 nm. Fluorescence emission spectra of 0 μL, 20.0 μL, 40.0 μL, 60.0 μL, 80.0 μL, 100.0 μL, fluorescence intensity. Follow this method and make changes to the amount to obtain the Stern–Volmer relationship in turn.

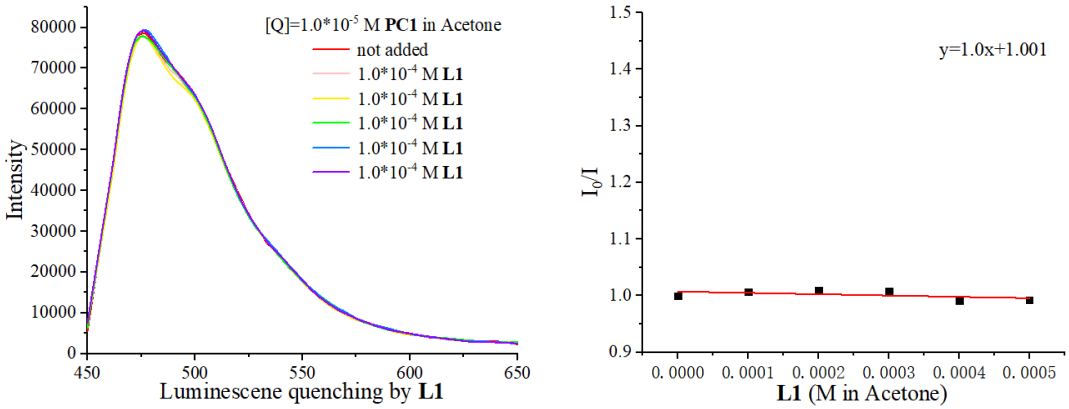
(a) $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ quenched by **1a** in acetone. Linear quenching is not observed.



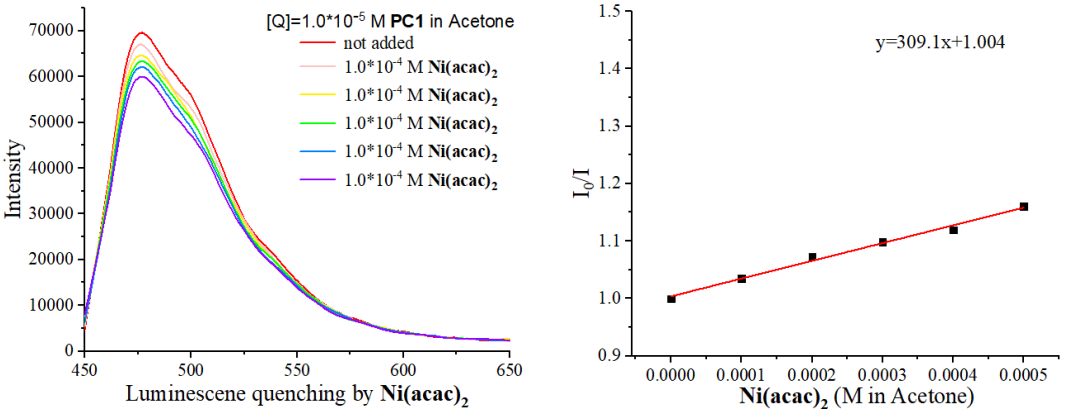
(b) $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ quenched by **2a** in acetone. Linear quenching is not observed.



(c) Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ quenched by **L1** in acetone. Linear quenching is not observed.

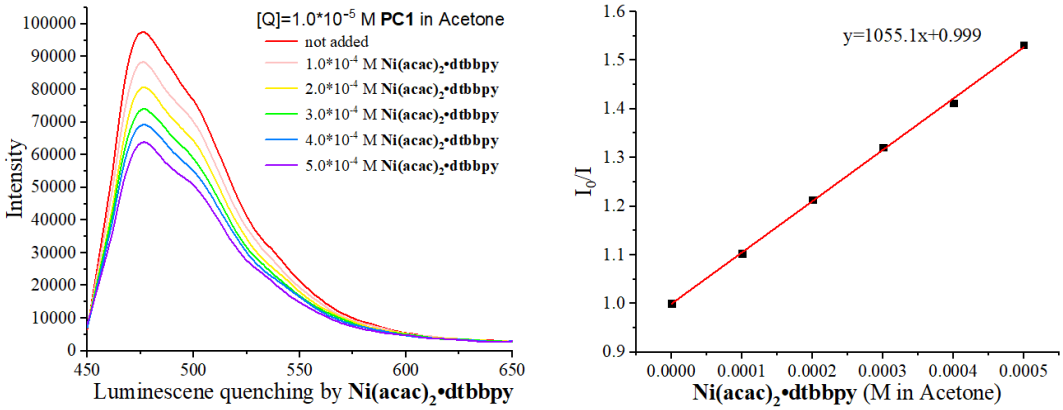


(d) Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ quenched by **Ni(acac)$_2$** in acetone



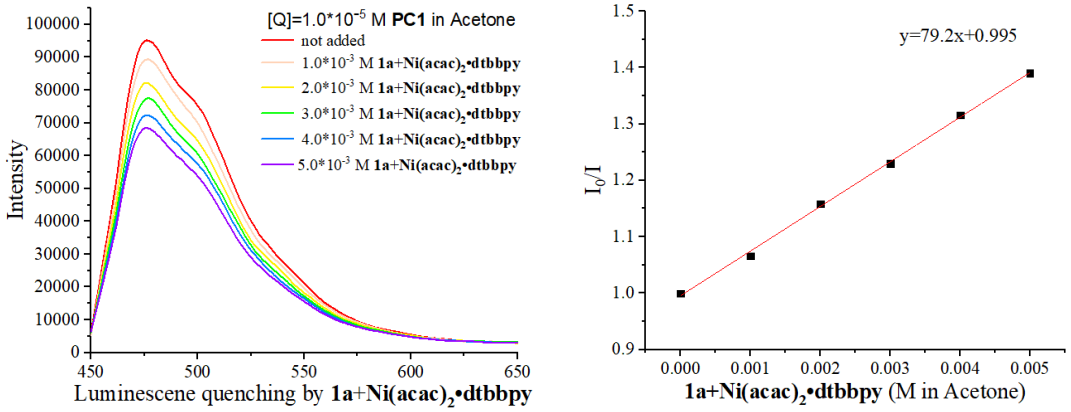
The emission intensity of the Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ catalyst solution strongly affected by the gradual increase of the amount of **Ni(acac)$_2$**.

(e) Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ quenched by **Ni(acac)$_2$•dtbbpy** in acetone



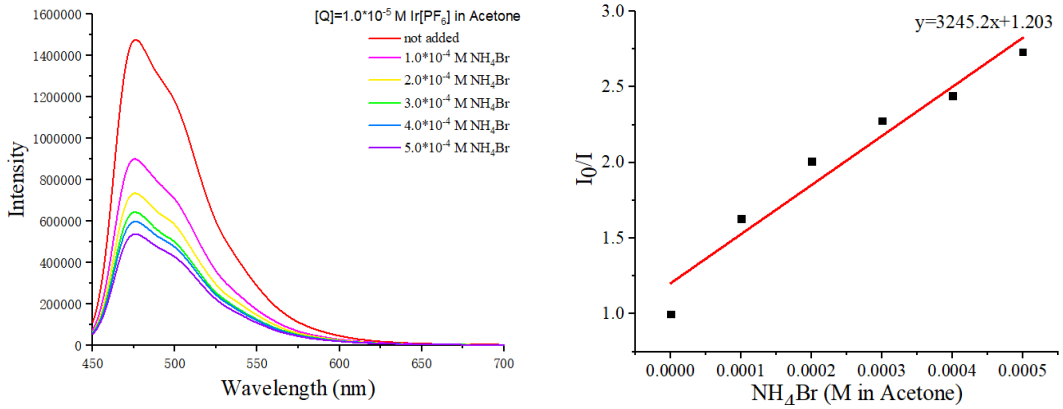
The emission intensity of the Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ catalyst solution strongly affected by the gradual increase of the amount of **Ni(acac)$_2$•dtbbpy**.

(f) Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ quenched by **1a**+**Ni(acac)$_2$•dtbbpy** in acetone



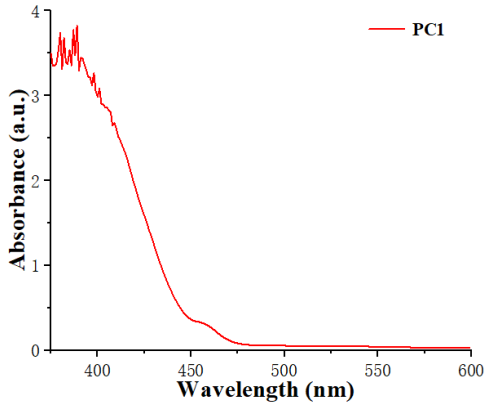
The emission intensity of the Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ catalyst solution strongly affected by the gradual increase of the amount of **1a**+**Ni(acac)$_2$•dtbbpy**.

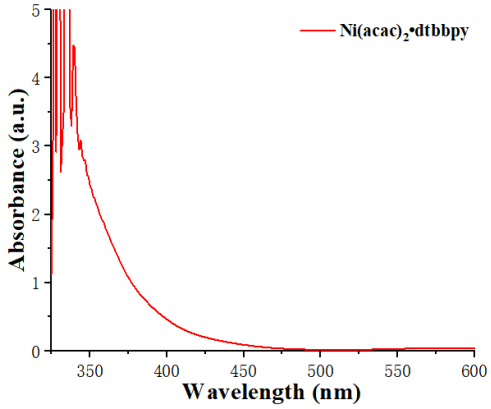
(g) Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ quenched by **NH$_4$Br** in acetone



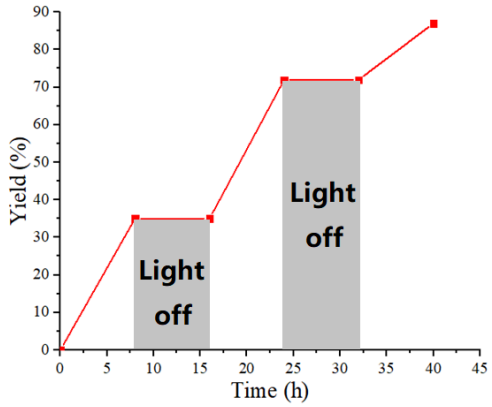
The emission intensity of the Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ catalyst solution strongly affected by the gradual increase of the amount of **NH$_4$Br**. This result proved that this SET process could generate an active bromine radical via cleavage of Ni-Br bond.

3.6 UV-Vis spectra



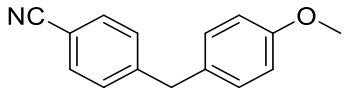


3.7 Light on/off experiment



The above depicted reaction was performed according to the general protocol established. The reaction was irradiated with Blue LEDs for 8 hour and then stirred in the dark for 8 hour. This procedure was repeated for 40 hours, and the yield of the product was determined by ^{1}H NMR with dibromomethane as an internal standard at each point the light was turned off or on. The results are shown in the graph above. This result shows that constant light irradiation is needed to progress the reaction.

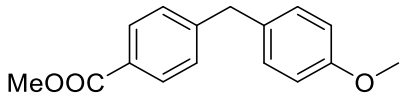
4. Analytical data



4-(4-Methoxybenzyl)benzonitrile (3a)

Yield: 38.8 mg, 87%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 3.96 (s, 2H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.2, 147.2, 132.2, 131.3, 129.9, 129.4, 119.0, 114.0, 109.8, 55.2, 41.0.

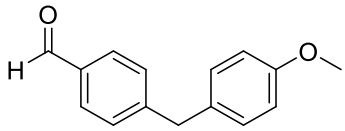
These spectroscopic data correspond to reported data.[2]



Methyl 4-(4-methoxybenzyl)benzoate (3b)

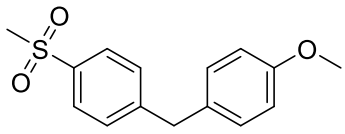
Yield: 44.1 mg, 86%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1H NMR (400 MHz, Chloroform-d) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 3.97 (s, 2H), 3.89 (s, 3H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.1, 158.1, 147.0, 132.2, 129.9, 129.8, 128.8, 128.0, 114.0, 55.2, 52.0, 41.00.

These spectroscopic data correspond to reported data.[2]



4-(4-Methoxybenzyl)benzaldehyde (3c)

Yield: 37.5 mg, 83%; white solid; mp 77-78 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.96 (s, 1H), 7.79 (d, *J* = 8.1 Hz, 2H), 7.33 (d, *J* = 7.9 Hz, 2H), 7.10 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 3.99 (s, 2H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 192.0, 158.2, 148.9, 134.5, 131.8, 130.0, 129.9, 129.4, 114.0, 55.2, 41.2. HRMS (ESI) m/z calcd for $C_{15}H_{14}O_2Na^+$ $(M+Na)^+$ 249.0886, found 249.0904.

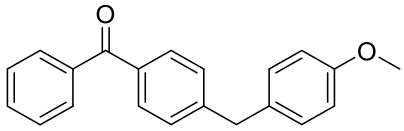


1-Methoxy-4-(4-(methylsulfonyl)benzyl)benzene (3d)

Yield: 43.1 mg, 78%; white solid; mp 85-88 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.84 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.2 Hz, 2H), 7.09 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 4.00 (s, 2H),

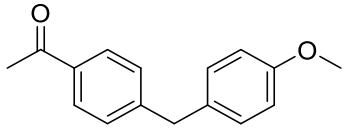
3.79 (s, 3H), 3.03 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.3, 148.2, 138.2, 131.4, 129.9, 129.6, 127.6, 114.1, 55.3, 44.6, 40.9.

These spectroscopic data correspond to reported data.[3]



(4-(4-Methoxybenzyl)phenyl)(phenyl)methanone (3e)

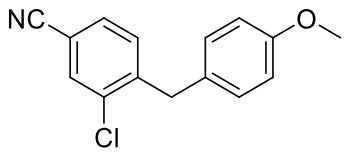
Yield: 56.8 mg, 94%; white solid; mp 63-65 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.77 (d, *J* = 7.2 Hz, 2H), 7.73 (d, *J* = 8.1 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 7.12 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 3.99 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.4, 158.1, 146.6, 137.7, 135.3, 132.2, 132.1, 130.4, 129.9, 129.9, 128.6, 128.1, 113.9, 55.2, 41.0. HRMS (ESI) m/z calcd for $C_{21}H_{18}O_2Na^+$ $(M+Na)^+$ 325.1199, found 325.1220.



1-(4-(4-Methoxybenzyl)phenyl)ethan-1-one (3f)

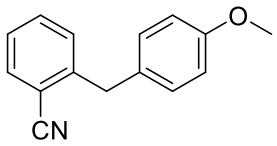
Yield: 43.7 mg, 91%; white solid; mp 88-91 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.87 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.3 Hz, 2H), 7.08 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 3.96 (s, 2H), 3.77 (s, 3H), 2.56 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 197.8, 158.1, 147.2, 135.1, 132.0, 129.8, 128.9, 128.6, 114.0, 55.2, 40.9, 26.5.

These spectroscopic data correspond to reported data.[2]



3-Chloro-4-(4-methoxybenzyl)benzonitrile (3g)

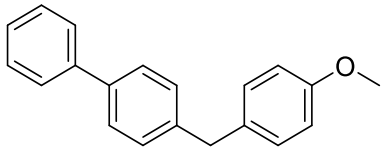
Yield: 36.0 mg, 70%; coloress oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.54 (d, *J* = 8.0 Hz, 1H), 7.29 (s, 1H), 7.16 (d, *J* = 7.4 Hz, 1H), 7.07 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 3.94 (s, 2H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.4, 148.9, 136.7, 133.8, 130.4, 130.0, 129.9, 127.5, 116.1, 114.2, 110.7, 55.2, 40.7. HRMS (ESI) m/z calcd for $C_{15}H_{12}ONClNa^+$ $(M+Na)^+$ 280.0500, found 280.0506.



2-(4-Methoxybenzyl)benzonitrile (3h)

Yield: 27.7 mg, 62%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.61 (d, *J* = 7.7 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 1H), 7.30-7.25 (m, 2H), 7.15 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.14 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 163.1, 158.3, 150.1, 144.4, 130.6, 130.1, 126.2, 124.6, 123.2, 121.6, 114.1, 55.2, 43.5.

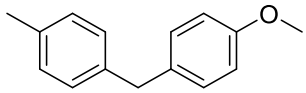
These spectroscopic data correspond to reported data.[2]



4-(4-Methoxybenzyl)-1,1'-biphenyl (3i)

Yield: 33.4 mg, 61%; white solid; mp 74-75 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, *J* = 7.4 Hz, 2H), 7.50 (d, *J* = 8.0 Hz, 2H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.31 (t, *J* = 7.3 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 8.5 Hz, 2H), 3.96 (s, 2H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.9, 141.0, 140.7, 138.9, 133.1, 129.9, 129.2, 128.7, 127.2, 127.0, 113.9, 113.6, 55.2, 40.6.

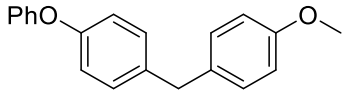
These spectroscopic data correspond to reported data.[2]



1-Methoxy-4-(4-methylbenzyl)benzene (3j)

Yield: 10.1 mg, 24%; coloress oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.14-7.01 (m, 6H), 6.82 (d, *J* = 8.5 Hz, 2H), 3.88 (s, 2H), 3.77 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.8, 138.5, 135.4, 133.5, 129.7, 129.1, 128.6, 113.8, 55.2, 40.6, 21.0.

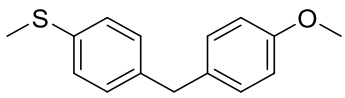
These spectroscopic data correspond to reported data.[4]



1-Methoxy-4-(4-phenoxybenzyl)benzene (3k)

Yield: 31.3 mg, 54%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.30 (t, *J* = 8.0 Hz, 2H), 7.11 (t, *J* =

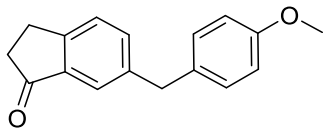
7.9 Hz, 4H), 7.06 (t, *J* = 7.4 Hz, 1H), 6.98 (d, *J* = 7.7 Hz, 2H), 6.92 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 3.89 (s, 2H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.0, 157.5, 155.3, 136.6, 133.3, 130.0, 129.8, 129.6, 122.9, 119.0, 118.6, 113.9, 55.2, 40.3. HRMS (ESI) m/z calcd for $C_{20}H_{18}O_2Na^+$ $(M+Na)^+$ 313.1199, found 313.1202.



(4-(4-Methoxybenzyl)phenyl)(methyl)sulfane (3l)

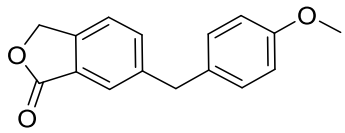
Yield: 30.3 mg, 62%; white solid; mp 74-76 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.19 (d, *J* = 8.3 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 7.6 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 3.88 (s, 2H), 3.78 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.9, 138.7, 135.5, 133.1, 129.8, 129.3, 127.1, 113.9, 55.2, 40.4, 16.2.

These spectroscopic data correspond to reported data.[4]



6-(4-Methoxybenzyl)-2,3-dihydro-1*H*-inden-1-one (3m)

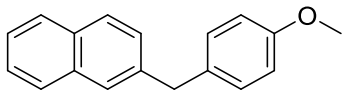
Yield: 39.3 mg, 78%; coloress oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 (s, 1H), 7.39 (q, *J* = 7.9 Hz, 2H), 7.08 (d, *J* = 8.6 Hz, 2H), 6.82 (d, *J* = 8.6 Hz, 2H), 3.95 (s, 2H), 3.6 (s, 3H), 3.13 – 3.03 (m, 2H), 2.72 – 2.63 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 207.1, 158.0, 153.1, 141.1, 137.3, 135.5, 132.5, 129.7, 126.6, 123.4, 113.9, 55.2, 40.6, 36.5, 25.4. HRMS (ESI) m/z calcd for $C_{17}H_{16}ONa^+$ $(M+Na)^+$ 275.1043, found 275.1048.



6-(4-Methoxybenzyl)isobenzofuran-1(3*H*)-one (3n)

Yield: 43.2 mg, 85%; white solid; mp 59-61 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (d, *J* = 7.9 Hz, 1H), 7.36 (d, *J* = 7.9 Hz, 1H), 7.25 (s, 1H), 7.10 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 5.24 (s, 2H), 4.04 (s, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 171.0, 158.3, 148.8, 147.1, 131.5, 129.9, 129.8, 125.6, 123.6, 122.0, 114.1, 69.4, 55.2, 41.2.

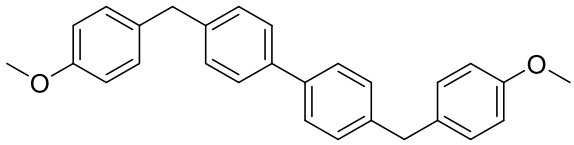
These spectroscopic data correspond to reported data.[11]



2-(4-Methoxybenzyl)naphthalene (3o)

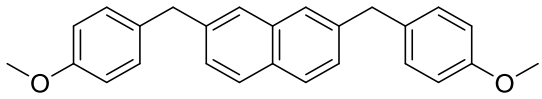
Yield: 39.7 mg, 80%; white solid; mp 67-69 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.83-7.69 (m, 3H), 7.60 (s, 1H), 7.45-7.38 (m, 2H), 7.29 (d, *J* = 8.5 Hz, 1H), 7.13 (d, *J* = 8.2 Hz, 2H), 6.82 (d, *J* = 7.9 Hz, 2H), 4.06 (s, 2H), 3.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.0, 139.0, 133.6, 133.0, 132.0, 129.9, 128.0, 127.6, 127.5, 127.5, 126.9, 125.9, 125.2, 113.9, 55.2, 41.2.

These spectroscopic data correspond to reported data.[5]



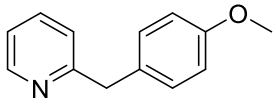
4,4'-bis(4-methoxybenzyl)-1,1'-biphenyl (3p)

Yield: 53.6 mg, 68%; white solid; mp 95-98 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.47 (d, *J* = 8.0 Hz, 4H), 7.22 (d, *J* = 7.9 Hz, 4H), 7.13 (d, *J* = 8.5 Hz, 4H), 6.84 (d, *J* = 8.6 Hz, 4H), 3.95 (s, 4H), 3.78 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.9, 140.5, 138.7, 133.1, 129.9, 129.1, 127.0, 113.9, 55.2, 40.6. HRMS (ESI) m/z calcd for $C_{28}H_{26}O_2Na^+$ $(M+Na)^+$ 417.1825, found 417.1829.



2,7-bis(4-methoxybenzyl)naphthalene (3q)

Yield: 53.8 mg, 73%; white solid; mp 101-103 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69 (d, *J* = 8.4 Hz, 2H), 7.51 (d, *J* = 1.6 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.6 Hz, 4H), 6.82 (d, *J* = 8.6 Hz, 4H), 4.04 (s, 4H), 3.76 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.9, 139.2, 133.7, 133.1, 130.6, 129.9, 127.7, 127.0, 126.6, 113.8, 55.2, 41.2. HRMS (ESI) m/z calcd for $C_{26}H_{24}O_2Na^+$ $(M+Na)^+$ 391.1669, found 391.1674.

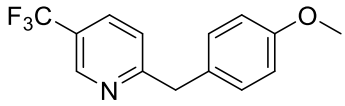


2-(4-methoxybenzyl)pyridine (3r)

Yield: 30.7 mg, 77%; coloress oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.47 (d, *J* = 4.6 Hz, 1H), 7.50 (td, *J* = 7.7, 1.9 Hz, 1H), 7.12 (d, *J* = 8.6 Hz, 2H), 7.02 (d, *J* = 7.6 Hz, 2H), 6.78 (d, *J* = 8.6 Hz, 2H), 4.03 (s, 2H),

3.71 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.3, 158.1, 149.2, 136.5, 131.5, 130.0, 122.9, 121.1, 114.0, 55.2, 43.7.

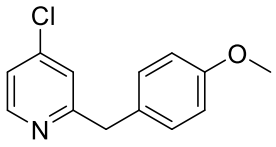
These spectroscopic data correspond to reported data.[3]



2-(4-Methoxybenzyl)-5-(trifluoromethyl)pyridine (3s)

Yield: 33.7 mg, 63%; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.81 (s, 1H), 7.80 (d, *J* = 8.4 Hz, 1H), 7.22 (d, *J* = 8.2 Hz, 1H), 7.18 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 4.17 (s, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.4, 158.4, 146.1 (q, *J* = 12.3 Hz, 1C), 133.6 (q, *J* = 10.4 Hz, 1C), 130.3, 130.1, 124.5, 123.3, 122.3, 114.2, 55.2, 43.7.

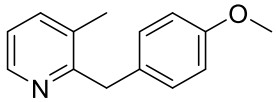
These spectroscopic data correspond to reported data.[6]



4-chloro-2-(4-methoxybenzyl)pyridine (3t)

Yield: 31.7 mg, 68%; coloress oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.53 (s, 1H), 7.67 (d, *J* = 8.5 Hz, 1H), 7.22 (d, *J* = 8.2 Hz, 1H), 7.20 (d, *J* = 8.6 Hz, 2H), 6.81 (d, *J* = 8.6 Hz, 2H), 4.16 (s, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.3, 158.1, 149.2, 136.5, 131.5, 130.0, 122.9, 121.1, 114.0, 55.2, 43.7.

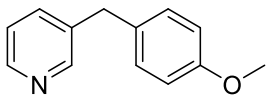
These spectroscopic data correspond to reported data.[6]



2-(4-Methoxybenzyl)-3-methylpyridine (3u)

Yield: 22.6 mg, 53%; coloress oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.42 (d, *J* = 4.1 Hz, 1H), 7.41 (d, *J* = 8.6 Hz, 1H), 7.11 (d, *J* = 8.6 Hz, 2H), 7.08 (dd, *J* = 7.6, 4.9 Hz, 1H), 6.80 (d, *J* = 8.6 Hz, 2H), 4.13 (s, 2H), 3.76 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.0, 157.9, 146.6, 138.1, 131.7, 131.0, 129.6, 121.6, 113.8, 55.2, 41.2, 18.9.

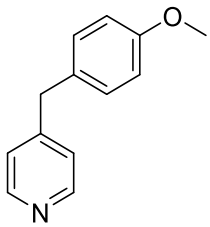
These spectroscopic data correspond to reported data.[9]



3-(4-Methoxybenzyl)pyridine (3v)

Yield: 29.9 mg, 75%; coloress oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.48 (s, 1H), 8.43 (d, *J* = 4.5 Hz, 1H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.17 (dd, *J* = 7.7, 4.9 Hz, 1H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 3.90 (s, 2H), 3.76 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.1, 149.9, 147.3, 136.8, 136.1, 131.7, 129.7, 123.3, 113.9, 55.1, 38.0.

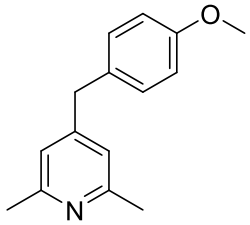
These spectroscopic data correspond to reported data.[9]



4-(4-Methoxybenzyl)pyridine (3w)

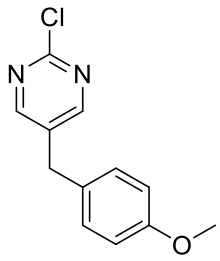
Yield: 26.7 mg, 67%; coloress oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.48 (d, *J* = 5.9 Hz, 2H), 7.10 (s, 2H), 7.08 (d, *J* = 2.7 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 3.91 (s, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.3, 150.6, 149.6, 130.8, 123.0, 124.1, 114.0, 55.2, 40.3.

These spectroscopic data correspond to reported data.[8]



4-(4-Methoxybenzyl)-2,6-dimethylpyridine (3x)

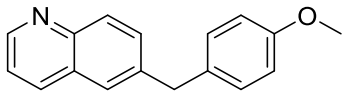
Yield: 30.0 mg, 66%; coloress oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.09 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 6.77 (s, 2H), 3.82 (s, 2H), 3.79 (s, 3H), 2.48 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.2, 157.5, 151.1, 131.3, 129.9, 120.7, 114.0, 55.2, 40.3, 24.2. HRMS (ESI) m/z calcd for $C_{15}H_{18}ON$ $(M+H)^+$ 228.1383, found 228.1391.



2-Chloro-5-(4-methoxybenzyl)pyrimidine (3y)

Yield: 27.6 mg, 59%; white solid; mp 73-75 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.44 (s,

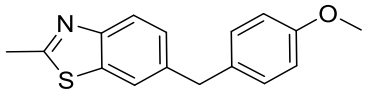
2H), 7.08 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 3.90 (s, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.5, 159.3, 158.6, 133.3, 129.7, 129.7, 114.4, 55.3, 34.8. HRMS (ESI) m/z calcd for $C_{12}H_{12}ON_2Cl$ $(M+H)^+$ 235.0633, found 235.0655.



6-(4-Methoxybenzyl)quinoline (3z)

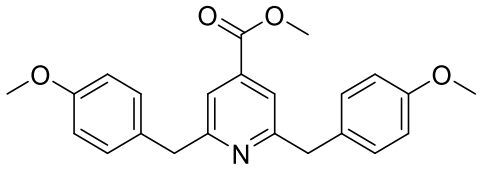
Yield: 31.9 mg, 64%; yellow solid; mp 63-65 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.84 (d, *J* = 4.2 Hz, 1H), 8.03 (t, *J* = 9.9 Hz, 2H), 7.54 (d, *J* = 6.9 Hz, 2H), 7.33 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.13 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.09 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.1, 149.7, 147.0, 140.0, 135.6, 132.4, 131.1, 129.9, 129.3, 128.2, 126.5, 121.0, 113.9, 55.2, 40.9.

These spectroscopic data correspond to reported data.[3]



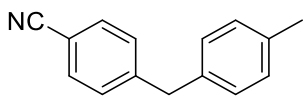
6-(4-Methoxybenzyl)-2-methylbenzo[*d*]thiazole (3aa)

Yield: 27.4 mg, 51%; white solid; mp 68-71 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75 (s, 1H), 7.70 (d, *J* = 8.2 Hz, 1H), 7.17 (d, *J* = 8.0 Hz, 1H), 7.13 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 4.05 (s, 2H), 3.77 (s, 3H), 2.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.2, 158.0, 153.8, 139.9, 133.2, 133.0, 129.9, 125.9, 122.3, 121.1, 113.9, 55.3, 40.9, 20.1. HRMS (ESI) m/z calcd for $C_{16}H_{16}ONS$ $(M+H)^+$ 270.0947, found 270.0952.



Methyl 2,6-bis(4-methoxybenzyl)isonicotinate (3ab)

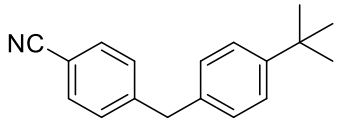
Yield: 21.1 mg, 28%; white solid; mp 88-90 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 (s, 2H), 7.19 (d, *J* = 8.6 Hz, 4H), 6.85 (d, *J* = 8.6 Hz, 4H), 4.15 (s, 4H), 3.84 (s, 3H), 3.79 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.9, 162.0, 158.2, 138.4, 131.1, 130.5, 119.6, 114.0, 55.2, 52.4, 43.6. HRMS (ESI) m/z calcd for $C_{23}H_{23}NO_4Na^+$ $(M+Na)^+$ 400.1519, found 400.1528.



4-(4-Methylbenzyl)benzonitrile (4a)

Yield: 33.1 mg, 80%; coloress oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.53 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 7.8 Hz, 2H), 7.04 (d, *J* = 7.9 Hz, 2H), 3.97 (s, 2H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.0, 136.2, 136.1, 132.1, 129.5, 129.3, 128.7, 119.0, 109.8, 41.4, 20.9.

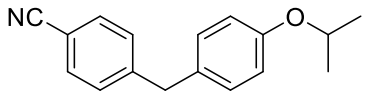
These spectroscopic data correspond to reported data.[7]



4-(4-(*tert*-Butyl)benzyl)benzonitrile (4c)

Yield: 41.9 mg, 84%; coloress oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.08 (d, *J* = 8.1 Hz, 2H), 3.99 (s, 2H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.5, 146.9, 136.2, 132.2, 129.6, 128.5, 125.6, 119.0, 109.8, 41.4, 34.4, 31.3.

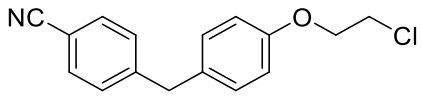
These spectroscopic data correspond to reported data.[6]



4-(4-Isopropoxybenzyl)benzonitrile (4d)

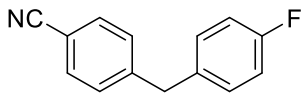
Yield: 44.2 mg, 88%; coloress oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.54 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.3 Hz, 2H), 7.04 (d, *J* = 8.6 Hz, 2H), 6.82 (d, *J* = 8.6 Hz, 2H), 4.50 (p, *J* = 6.0 Hz, 1H), 3.95 (s, 2H), 1.32 (s, 3H), 1.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.6, 147.2, 132.2, 131.1, 129.9, 129.5, 119.0, 116.0, 109.8, 69.8, 41.0, 22.0.

These spectroscopic data correspond to reported data.[9]



4-(4-(2-Chloroethoxy)benzyl)benzonitrile (4e)

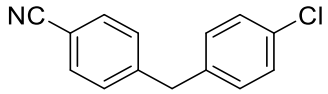
Yield: 44.5 mg, 82%; coloress oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 4.20 (t, *J* = 5.9 Hz, 2H), 3.97 (s, 2H), 3.79 (t, *J* = 5.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.9, 147.0, 132.2, 132.2, 130.0, 129.5, 118.9, 115.0, 109.9, 68.1, 41.9, 41.0. HRMS (ESI) m/z calcd for $C_{16}H_{14}ONClNa^+$ $(M+Na)^+$ 294.0656, found 294.0671.



4-(4-Fluorobenzyl)benzonitrile (4f)

Yield: 28.7 mg, 68%; white solid; mp 77-78 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 2H), 7.12 (dd, *J* = 8.5, 5.4 Hz, 2H), 7.00 (t, *J* = 8.7 Hz, 2H), 4.01 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.2 (d, *J* = 243.5 Hz), 146.5, 134.9 (d, *J* = 3.3 Hz), 132.3, 130.4 (d, *J* = 7.9 Hz), 129.5, 118.9, 115.6 (d, *J* = 21.2 Hz), 110.1, 41.1.

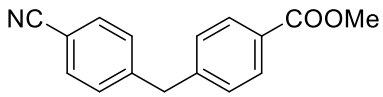
These spectroscopic data correspond to reported data.[9]



4-(4-Chlorobenzyl)benzonitrile (4g)

Yield: 30.0 mg, 66%; white solid; mp 74-76 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58 (d, *J* = 8.2 Hz, 2H), 7.27 (t, *J* = 7.6 Hz, 4H), 7.09 (d, *J* = 8.3 Hz, 2H), 4.00 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.1, 137.7, 132.5, 132.4, 130.2, 129.5, 128.8, 118.8, 110.2, 41.2.

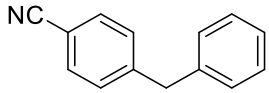
These spectroscopic data correspond to reported data.[7]



Methyl 4-(4-cyanobenzyl)benzoate (4h)

Yield: 23.1 mg, 46%; yellow solid; mp 63-65 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 8.2 Hz, 2H), 7.59 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.6 Hz, 2H), 7.23 (d, *J* = 8.2 Hz, 2H), 4.09 (s, 2H), 3.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.8, 145.6, 144.5, 132.4, 130.1, 129.6, 129.0, 128.7, 118.8, 110.4, 52.1, 41.9.

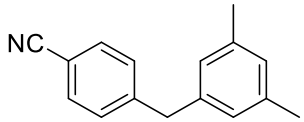
These spectroscopic data correspond to reported data.[3]



4-Benzylbenzonitrile (4i)

Yield: 20.9 mg, 54%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 7.9 Hz, 3H), 7.26 (t, *J* = 6.7 Hz, 2H), 7.16 (d, *J* = 7.0 Hz, 2H), 4.03 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.7, 139.3, 132.3, 129.6, 128.9, 128.7, 126.7, 119.0, 110.0, 42.0.

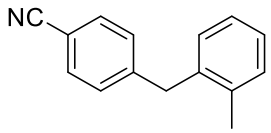
These spectroscopic data correspond to reported data.[7]



4-(3,5-Dimethylbenzyl)benzonitrile (4j)

Yield: 34.1 mg, 77%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 6.87 (s, 1H), 6.77 (s, 2H), 3.94 (s, 2H), 2.28 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.0, 139.2, 138.3, 132.2, 129.6, 128.2, 126.7, 119.0, 109.8, 41.8, 21.2.

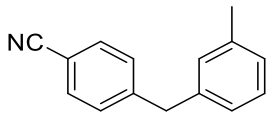
These spectroscopic data correspond to reported data.[7]



4-(2-Methylbenzyl)benzonitrile (4k)

Yield: 26.9 mg, 65%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55 (d, *J* = 8.2 Hz, 2H), 7.24-7.15 (m, 5H), 7.10-7.07 (m, 1H), 4.04 (s, 2H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.1, 137.1, 136.5, 132.2, 130.5, 130.0, 129.3, 127.0, 126.2, 119.0, 109.8, 39.5, 19.6.

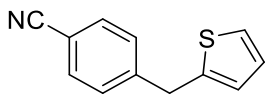
These spectroscopic data correspond to reported data.[7]



4-(3-Methylbenzyl)benzonitrile (4l)

Yield: 30.2 mg, 73%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.95 (d, *J* = 8.2 Hz, 2H), 3.98 (s, 2H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.8, 139.2, 138.4, 132.2, 129.7, 129.6, 128.6, 127.3, 125.9, 119.0, 109.8, 41.8, 21.3.

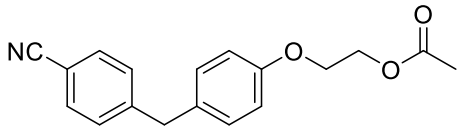
These spectroscopic data correspond to reported data.[7]



4-(Thiophen-2-ylmethyl)benzonitrile (4m)

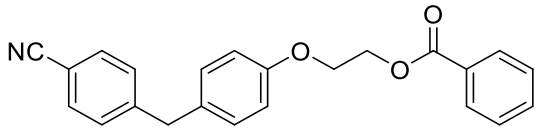
Yield: 31.1 mg, 78%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.59 (d, *J* = 8.2 Hz, 2H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 6.2 Hz, 1H), 6.95 (dd, *J* = 5.2, 3.4 Hz, 1H), 6.81 (dd, *J* = 3.4, 1.2 Hz, 1H), 4.21 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 145.8, 141.6, 132.3, 129.3, 127.0, 125.8, 124.6, 118.8, 110.4, 35.9.

These spectroscopic data correspond to reported data.[10]



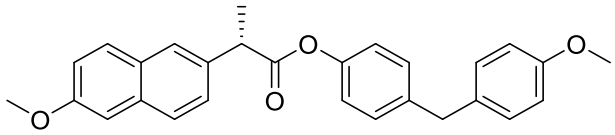
2-(4-(4-Cyanobenzyl)phenoxy)ethyl acetate (4n)

Yield: 41.3 mg, 70%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 7.08 (d, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 4.41 (t, *J* = 8.8 Hz, 2H), 4.15 (t, *J* = 8.3 Hz, 2H), 3.97 (s, 2H), 2.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.9, 157.1, 147.0, 132.2, 131.9, 129.9, 129.4, 118.9, 114.8, 109.8, 65.9, 62.7, 41.0, 20.8. HRMS (ESI) m/z calcd for $C_{18}H_{17}O_3NNa^+$ $(M+Na)^+$ 318.1101, found 318.1108.



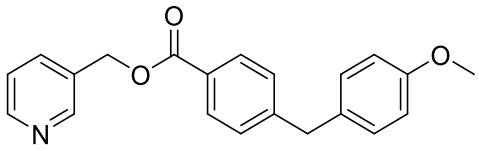
2-(4-(4-Cyanobenzyl)phenoxy)ethyl benzoate (4o)

Yield: 52.1 mg, 73%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.05 (d, *J* = 7.1 Hz, 2H), 7.60-7.51 (m, 3H), 7.42 (t, *J* = 7.7 Hz, 2H), 7.25 (d, *J* = 8.3 Hz, 2H), 7.08 (d, *J* = 8.6 Hz, 2H), 6.89 (d, *J* = 8.6 Hz, 2H), 4.66 (t, *J* = 8.8 Hz, 2H), 4.29 (t, *J* = 8.8 Hz, 2H), 3.97 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.5, 157.3, 147.1, 133.1, 132.2, 131.9, 130.0, 129.8, 129.7, 129.5, 128.3, 118.9, 114.9, 109.9, 66.1, 63.2, 41.0. HRMS (ESI) m/z calcd for $C_{23}H_{19}O_3NNa^+$ $(M+Na)^+$ 380.1257, found 380.1261.



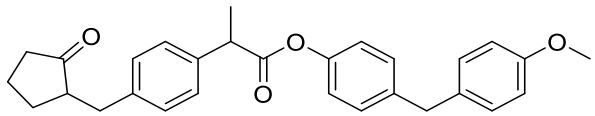
4-(4-Methoxybenzyl)phenyl (S)-2-(6-methoxynaphthalen-2-yl)propanoate (5a)

Yield: 55.4 mg, 65%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75 (s, 1H), 7.74-7.67 (t, *J* = 8.0 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 1H), 7.16-7.11 (m, 2H), 7.09 (d, *J* = 8.5 Hz, 2H), 7.04 (d, *J* = 8.6 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.79 (d, *J* = 8.6 Hz, 2H), 4.06 (q, *J* = 7.1 Hz, 1H), 3.89 (s, 3H), 3.86 (s, 2H), 3.74 (s, 3H), 1.67 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.2, 157.9, 157.7, 149.0, 139.0, 135.1, 133.7, 132.8, 129.8, 129.6, 129.3, 128.9, 127.3, 126.1, 121.2, 12.1, 119.0, 113.8, 105.5, 55.2, 55.2, 45.5, 40.3, 18.5. HRMS (ESI) m/z calcd for $C_{28}H_{26}O_4Na^+$ $(M+Na)^+$ 449.1723, found 449.1731.



Pyridin-3-ylmethyl 4-(4-methoxybenzyl)benzoate (5b)

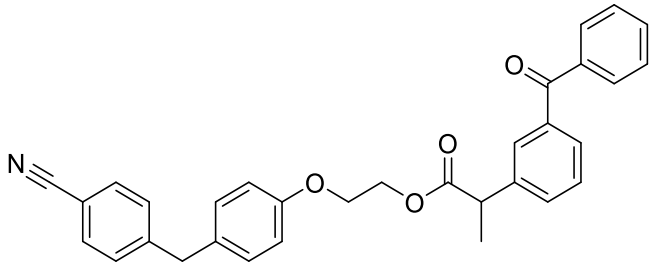
Yield: 47.3 mg, 71%; white solid; mp 91-93 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.71 (s, 1H), 8.59 (s, 1H), 7.97 (d, *J* = 8.2 Hz, 2H), 7.77 (d, *J* = 7.9 Hz, 1H), 7.33-7.30 (m, 1H), 7.25 (d, *J* = 8.1 Hz, 2H), 7.08 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 5.36 (s, 2H), 3.97 (s, 2H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.2, 158.1, 149.5, 147.5, 135.9, 132.0, 131.8, 129.9, 129.8, 128.9, 128.8, 127.4, 123.5, 114.0, 63.9, 55.2, 41.0. HRMS (ESI) m/z calcd for $C_{21}H_{19}NO_3Na^+$ $(M+Na)^+$ 356.1257, found 356.1266.



4-(4-Methoxybenzyl)phenyl 2-(4-((2-oxocyclopentyl)methyl)phenyl)propanoate (5c)

Yield: 60.1 mg, 68%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.30 (d, *J* = 7.9 Hz, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 7.12 (d, *J* = 8.3 Hz, 2H), 7.06 (d, *J* = 8.3 Hz, 2H), 6.90 (d, *J* = 8.2 Hz, 2H), 6.81 (d, *J* = 8.5 Hz, 2H), 3.92 (d, *J* = 7.2 Hz, 1H), 3.89 (s, 2H), 3.77 (s, 3H), 3.14 (dd, *J* = 13.8, 4.0 Hz, 1H), 2.52 (dd, *J* = 13.8, 9.5 Hz, 1H), 2.34 (dd, *J* = 18.7, 9.2 Hz, 2H), 2.16-2.05 (m, 2H), 2.00-1.92 (m, 1H), 1.76-1.70 (m, 1H), 1.64 (d, *J* = 3.6 Hz, 1H), 1.58 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 220.1, 173.1, 158.0, 149.0, 139.1, 139.0, 137.9, 132.9, 130.0, 129.6, 129.2, 127.5, 121.2, 113.8, 55.2, 50.9, 45.2, 40.3, 38.1, 35.2, 29.2, 20.5, 18.5.

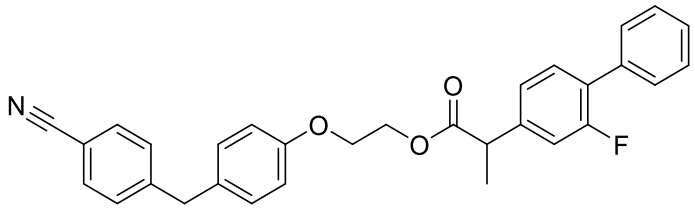
HRMS (ESI) m/z calcd for $C_{29}H_{30}O_4Na^+$ $(M+Na)^+$ 465.2036, found 465.2044.



2-(4-(4-Cyanobenzyl)phenoxy)ethyl 2-(3-benzoylphenyl)propanoate (5d)

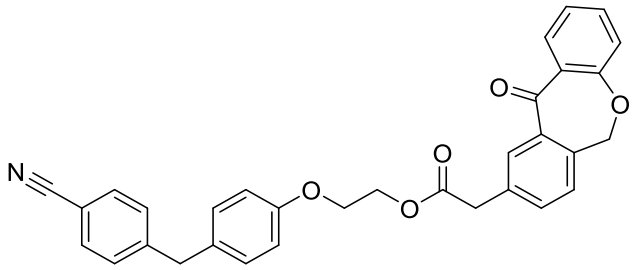
Yield: 70.4 mg, 72%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.76 (d, *J* = 8.4 Hz, 3H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.58 (d, *J* = 7.4 Hz, 1H), 7.55 (d, *J* = 2.7 Hz, 3H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.7 Hz, 1H), 7.24 (d, *J* = 8.3 Hz, 2H), 7.03 (d, *J* = 8.6 Hz, 2H), 6.78 (d, *J* = 8.6 Hz, 2H), 4.49-4.34 (m, 2H),

4.10 (t, *J* = 4.8 Hz, 2H), 3.94 (s, 2H), 3.84 (q, *J* = 7.1 Hz, 1H), 1.54 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.3, 173.9, 157.1, 147.0, 140.5, 137.8, 137.4, 132.4, 132.2, 131.9, 131.4, 130.0, 129.9, 129.4, 129.1, 129.0, 128.5, 128.2, 118.9, 114.8, 109.9, 65.8, 63.1, 45.2, 41.0, 18.4. HRMS (ESI) m/z calcd for $C_{32}H_{27}NO_4Na^+$ $(M+Na)^+$ 512.1832, found 512.1833.



2-(4-(4-Cyanobenzyl)phenoxy)ethyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (5e)

Yield: 76.7 mg, 80%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57-7.47 (m, 4H), 7.43 (t, *J* = 7.4 Hz, 2H), 7.37 (d, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 7.23 (d, *J* = 8.3 Hz, 2H), 7.16-7.11 (m, 2H), 7.04 (d, *J* = 8.6 Hz, 2H), 6.81 (d, *J* = 8.6 Hz, 2H), 4.45 (td, *J* = 4.5, 2.6 Hz, 2H), 4.13 (t, *J* = 4.7 Hz, 2H), 3.94 (s, 2H), 3.79 (q, *J* = 7.1 Hz, 1H), 1.54 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.8, 159.6 (d, *J* = 246.9 Hz), 157.2, 147.0, 141.5 (d, *J* = 7.6 Hz), 135.4, 132.2, 131.9, 130.7 (d, *J* = 3.8 Hz), 129.9, 129.4, 128.9, 128.8, 128.4, 127.6, 123.5 (d, *J* = 3.3 Hz), 118.9, 115.2 (d, *J* = 23.6 Hz), 114.9, 109.9, 65.84, 63.17, 44.86, 41.00, 18.29. HRMS (ESI) m/z calcd for $C_{31}H_{27}FNO_3^+$ $(M+H)^+$ 480.1969, found 480.1975.



2-(4-(4-Cyanobenzyl)phenoxy)ethyl 2-(11-oxo-6,11-dihydrodibenzo[b,e]oxepin-9-yl)acetate (5f)

Yield: 67.4 mg, 67%; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.11 (d, *J* = 2.4 Hz, 1H), 7.87 (d, J = 7.7 Hz, 1H), 7.59-7.52 (m, 3H), 7.46 (t, *J* = 7.6 Hz, 1H), 7.41 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.36 (d, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 8.3 Hz, 2H), 7.06 (d, *J* = 8.6 Hz, 2H), 7.00 (d, *J* = 8.4 Hz, 1H), 6.84 (d, *J* = 8.6 Hz, 2H), 5.16 (s, 2H), 4.48-4.41 (m, 2H), 4.18-4.12 (m, 2H), 3.95 (s, 2H), 3.67 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 190.7, 171.3, 160.5, 157.2, 147.1, 140.4, 136.3, 135.5, 132.8, 132.4, 132.2, 132.0, 130.0, 129.5, 129.4, 129.2, 127.8, 127.5, 125.1, 121.0, 119.0, 114.9, 109.9, 73.6, 65.9, 63.2, 41.0, 40.0. HRMS (ESI) m/z calcd for $C_{32}H_{25}O_5N^+$ $(M+H)^+$ 526.1625, found 526.1629.

5. Reference

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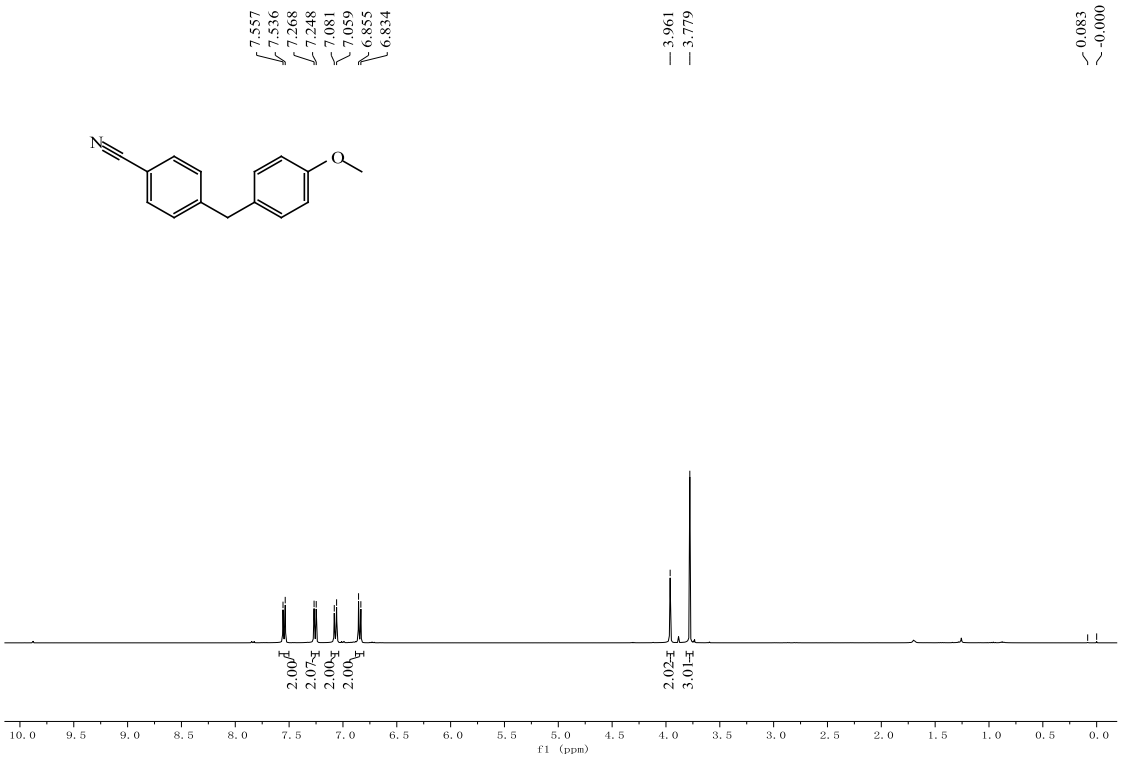
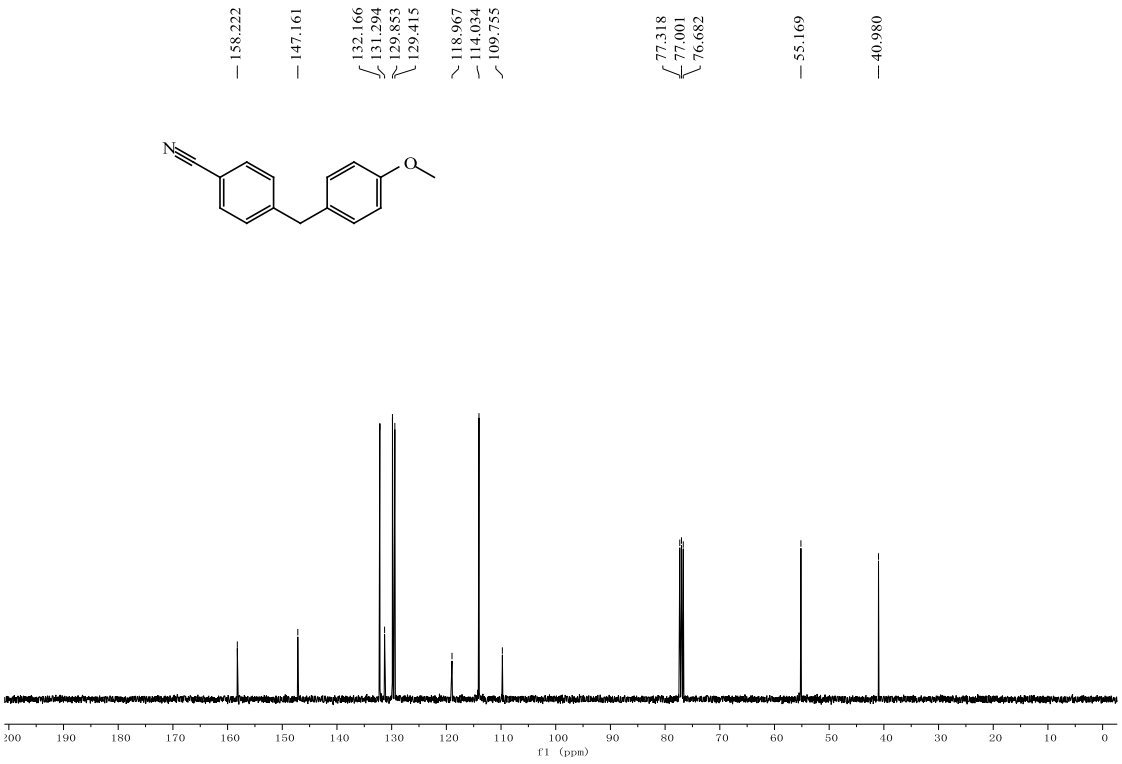
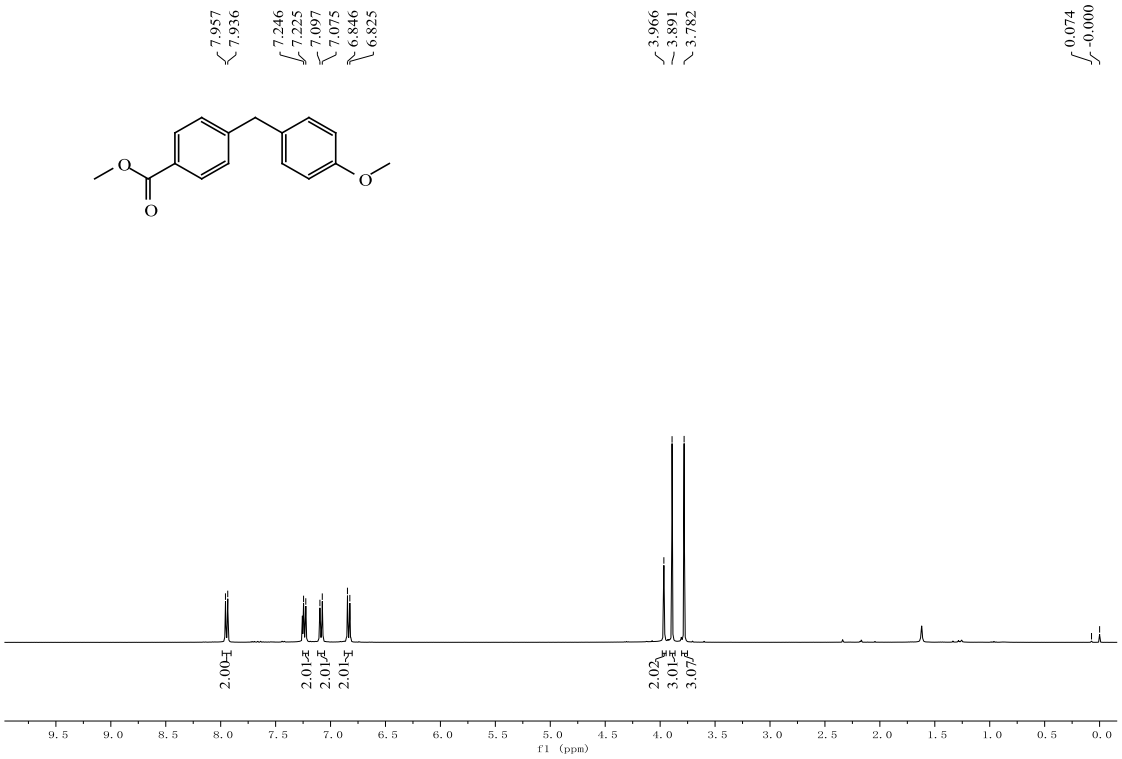
6. Spectra

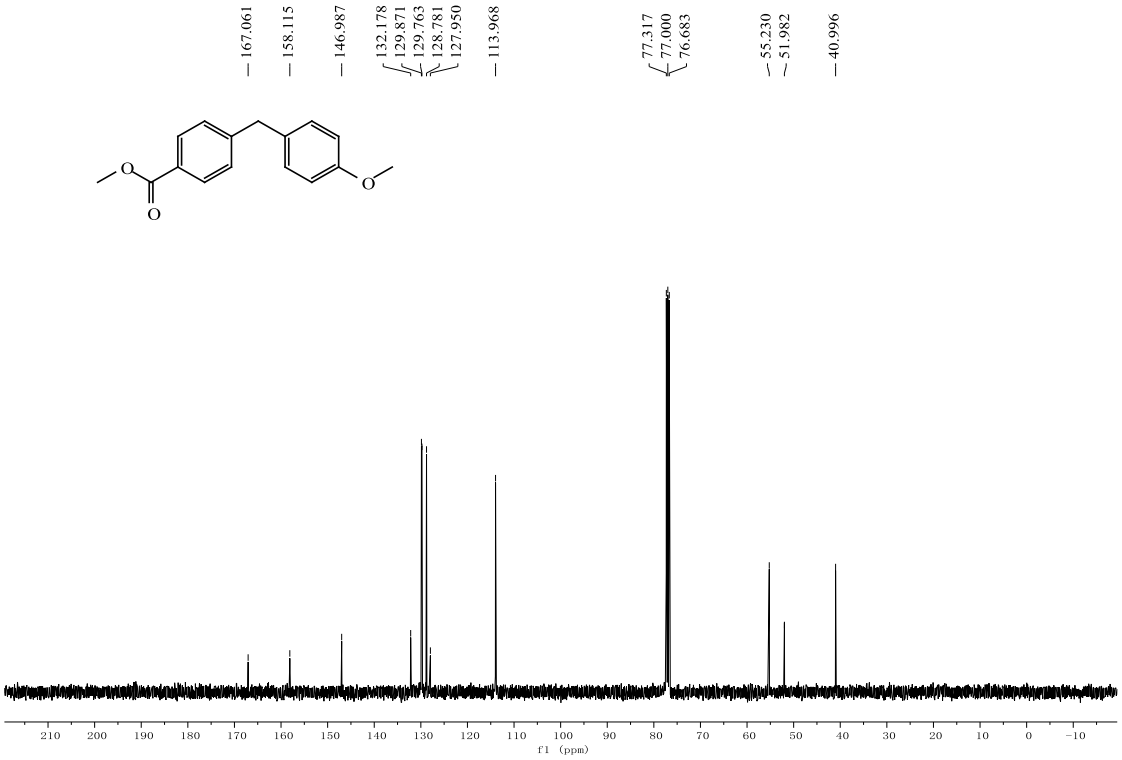
4-(4-Methoxybenzyl)benzonitrile (3a)



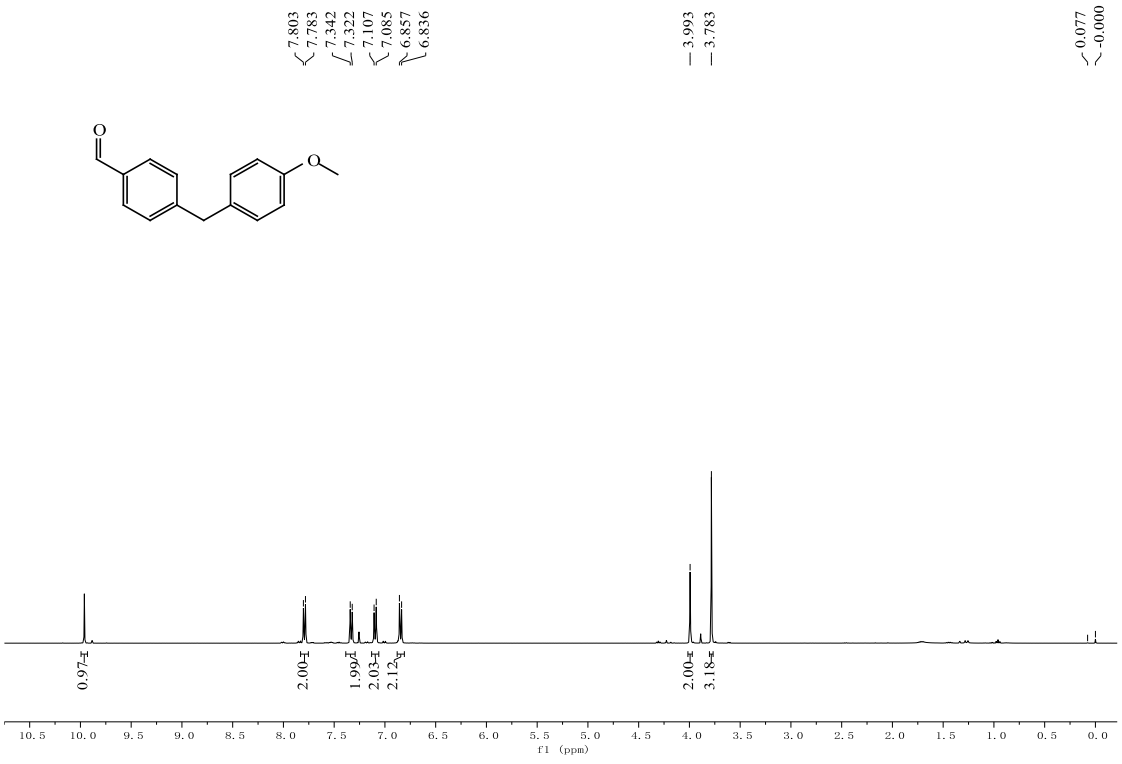


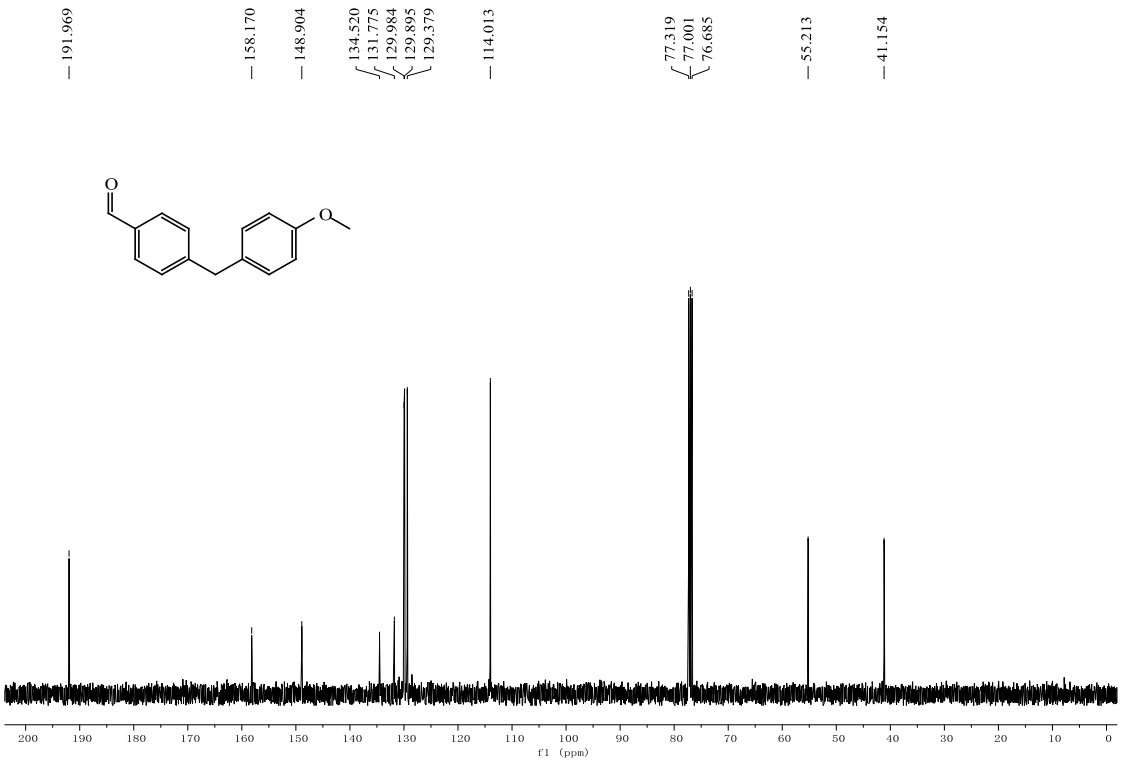
Methyl 4-(4-methoxybenzyl)benzoate (3b)



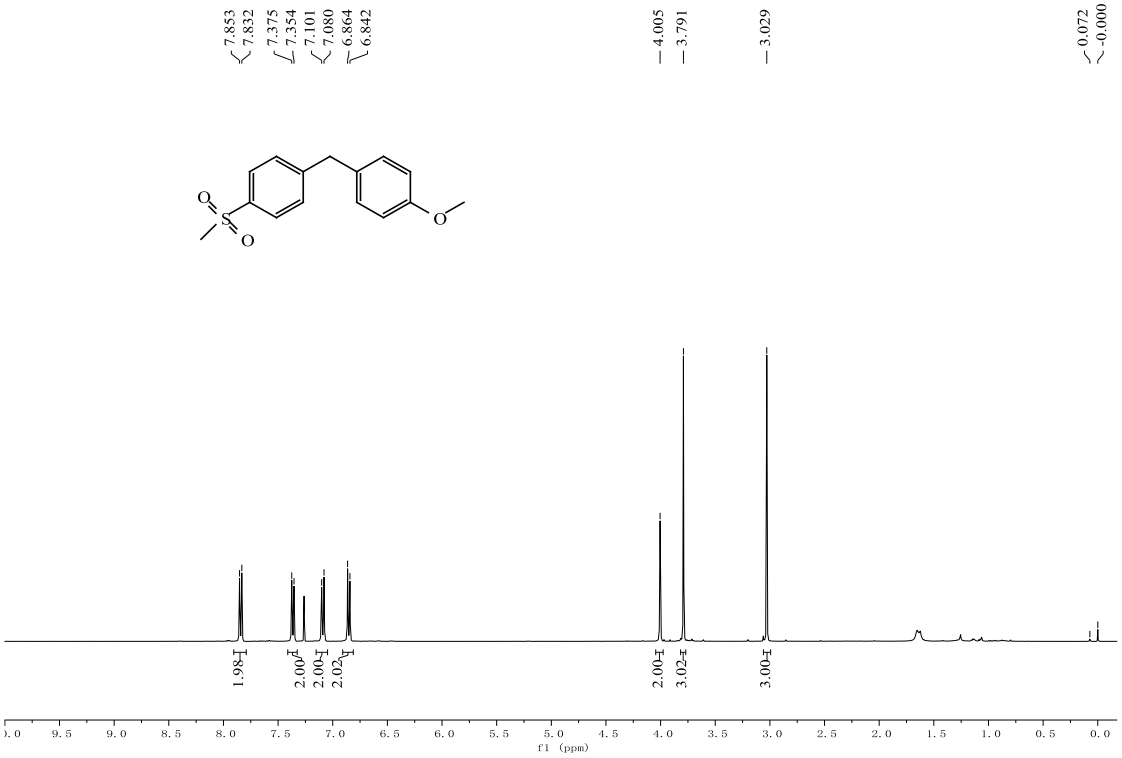


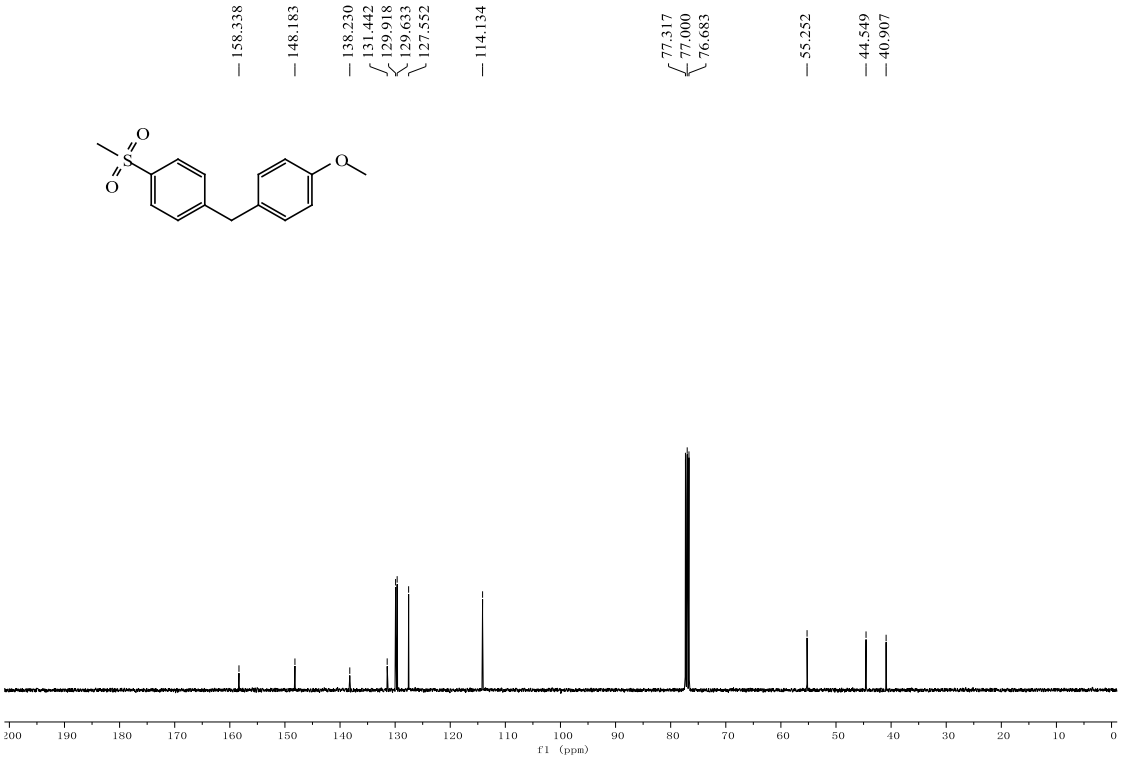
4-(4-Methoxybenzyl)benzaldehyde (3c)



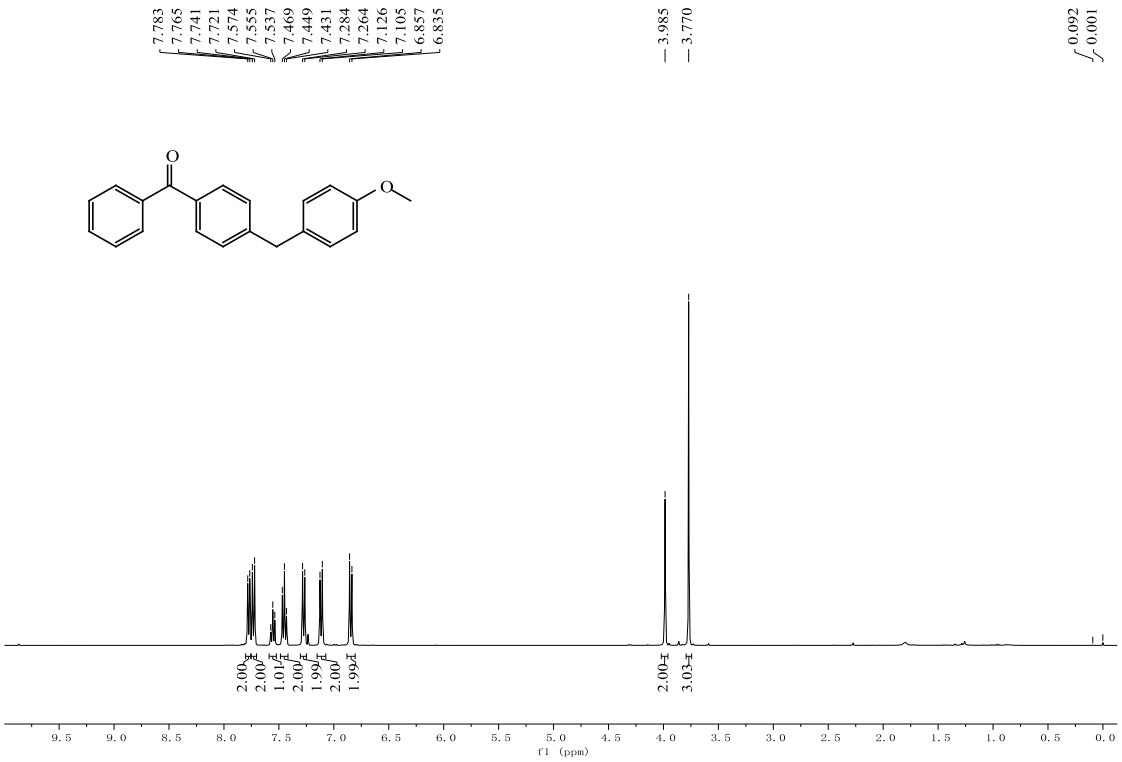


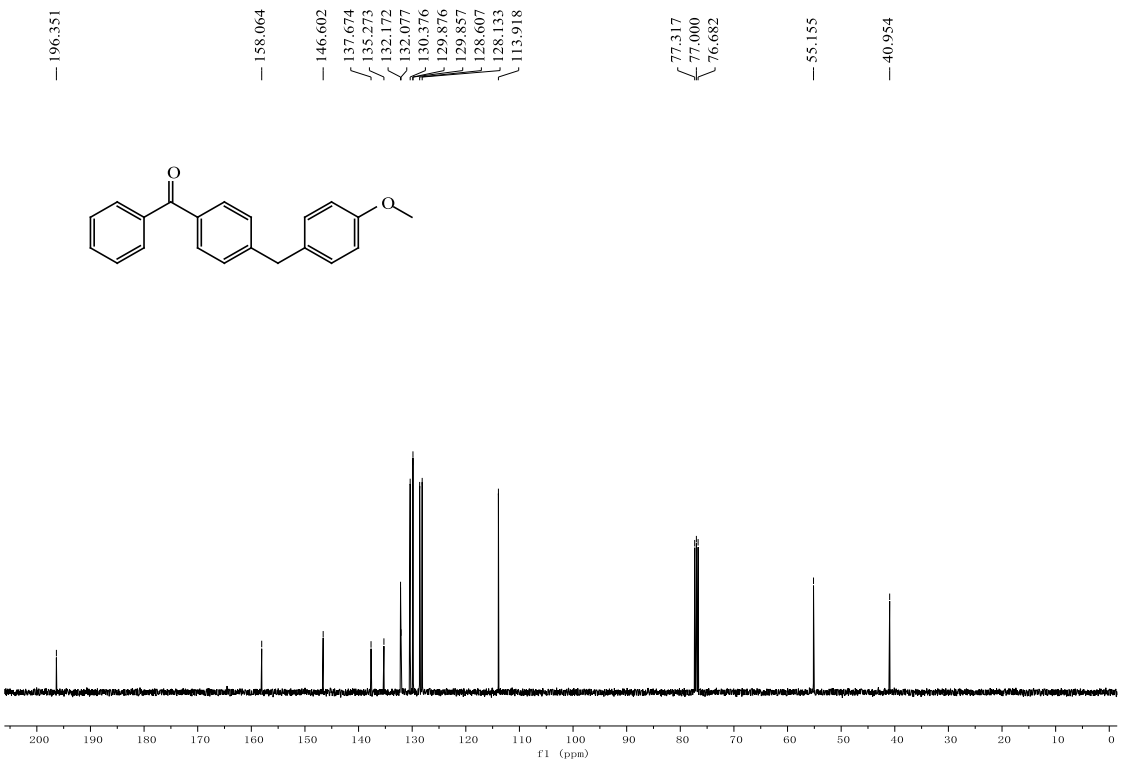
1-Methoxy-4-(4-(methylsulfonyl)benzyl)benzene (3d)



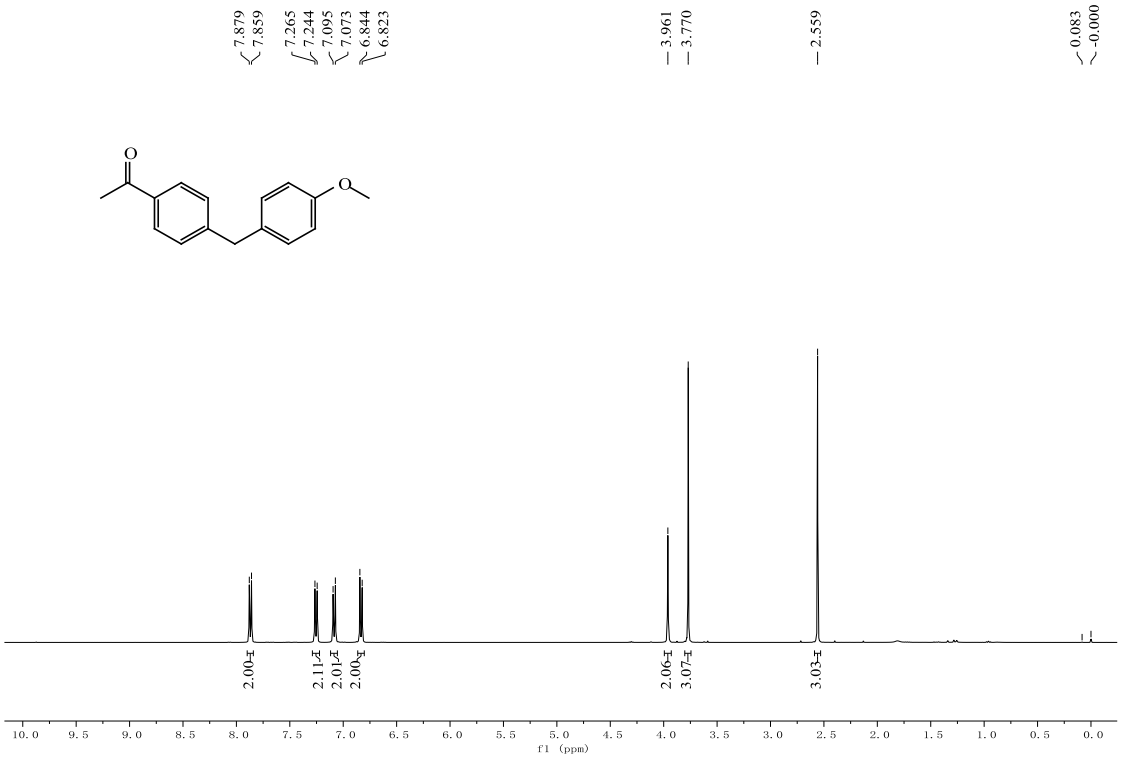


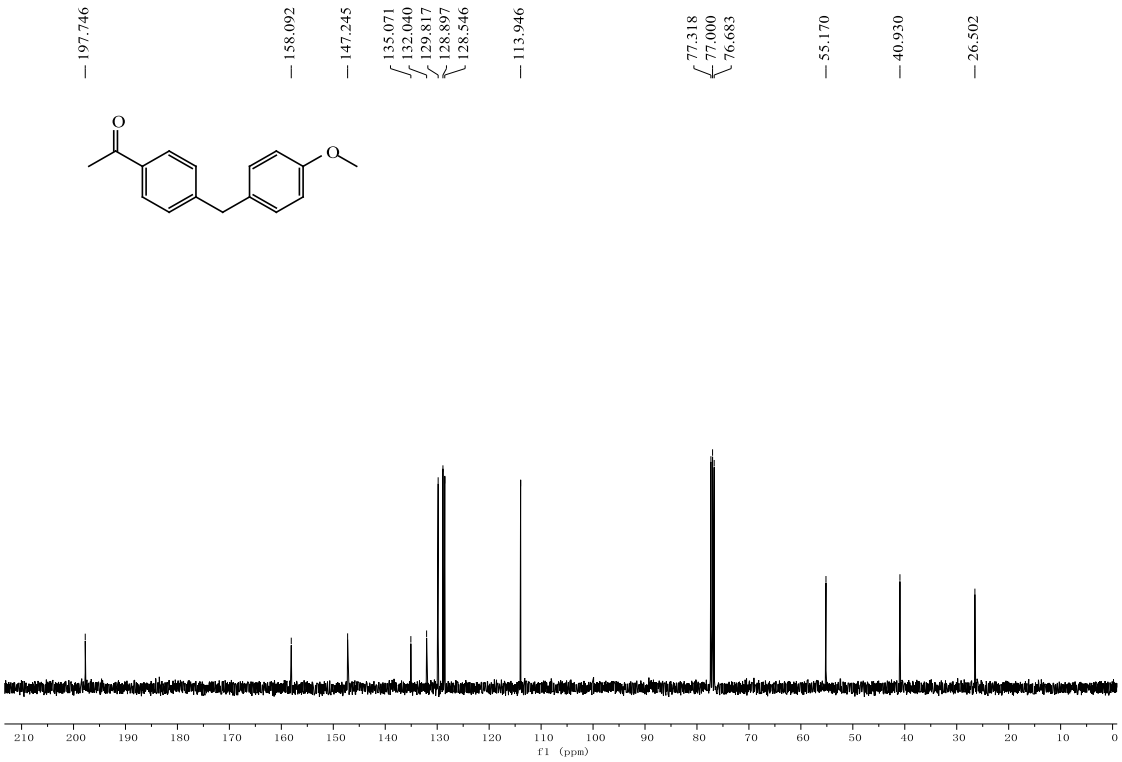
(4-(4-Methoxybenzyl)phenyl)(phenyl)methanone (3e)



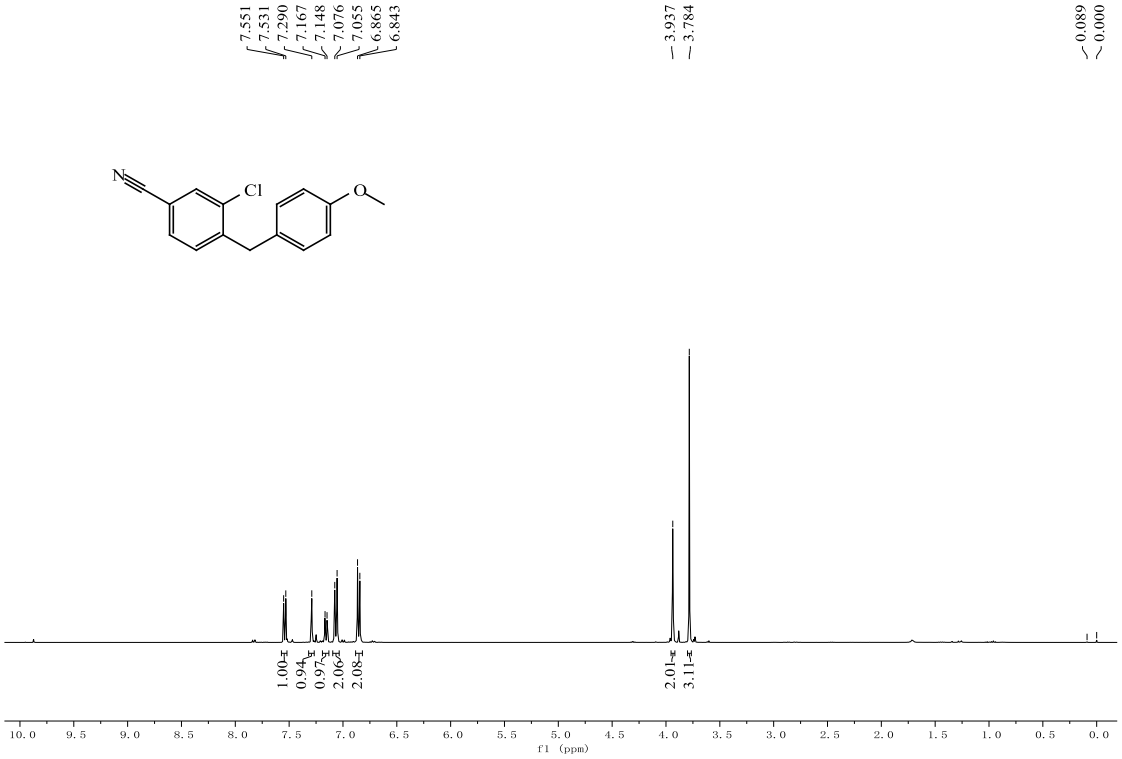


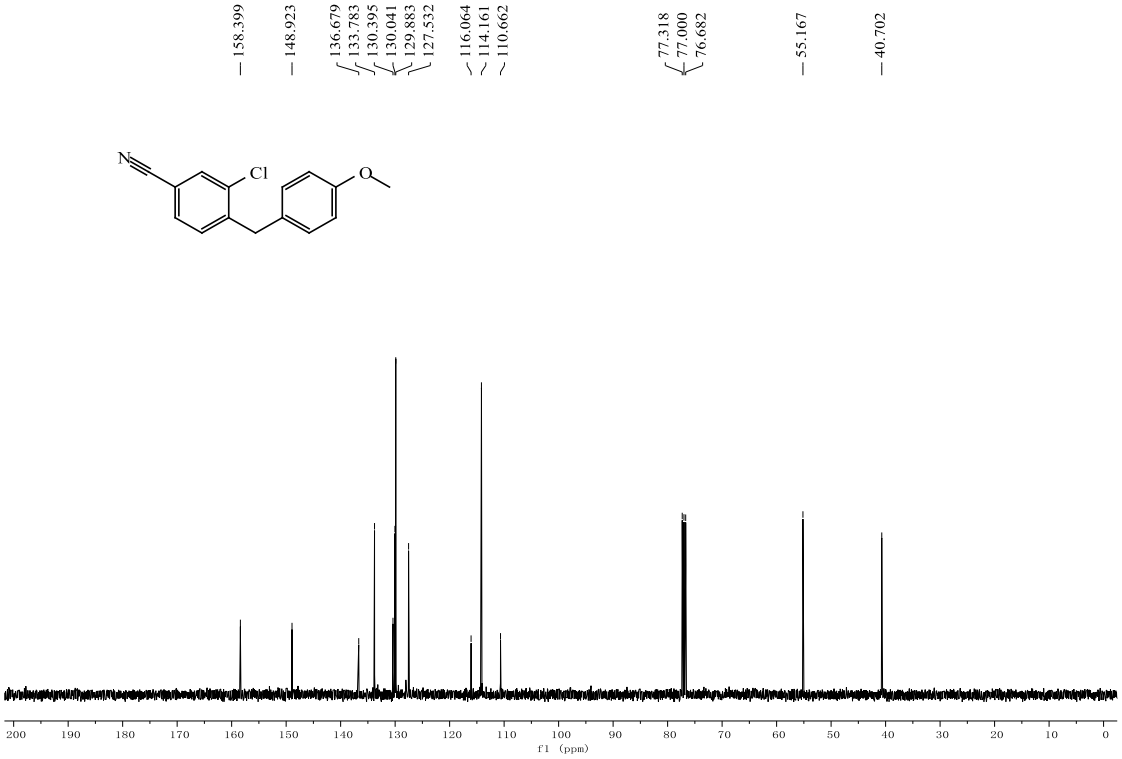
1-(4-(4-Methoxybenzyl)phenyl)ethan-1-one (3f)



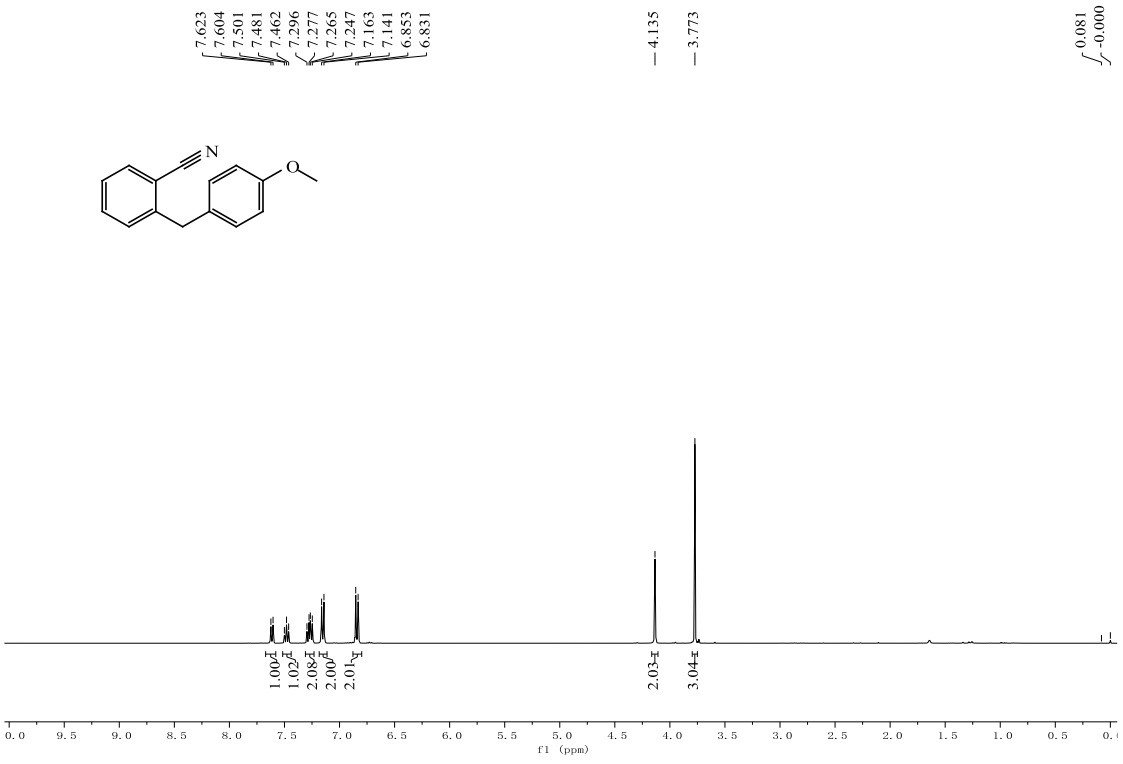


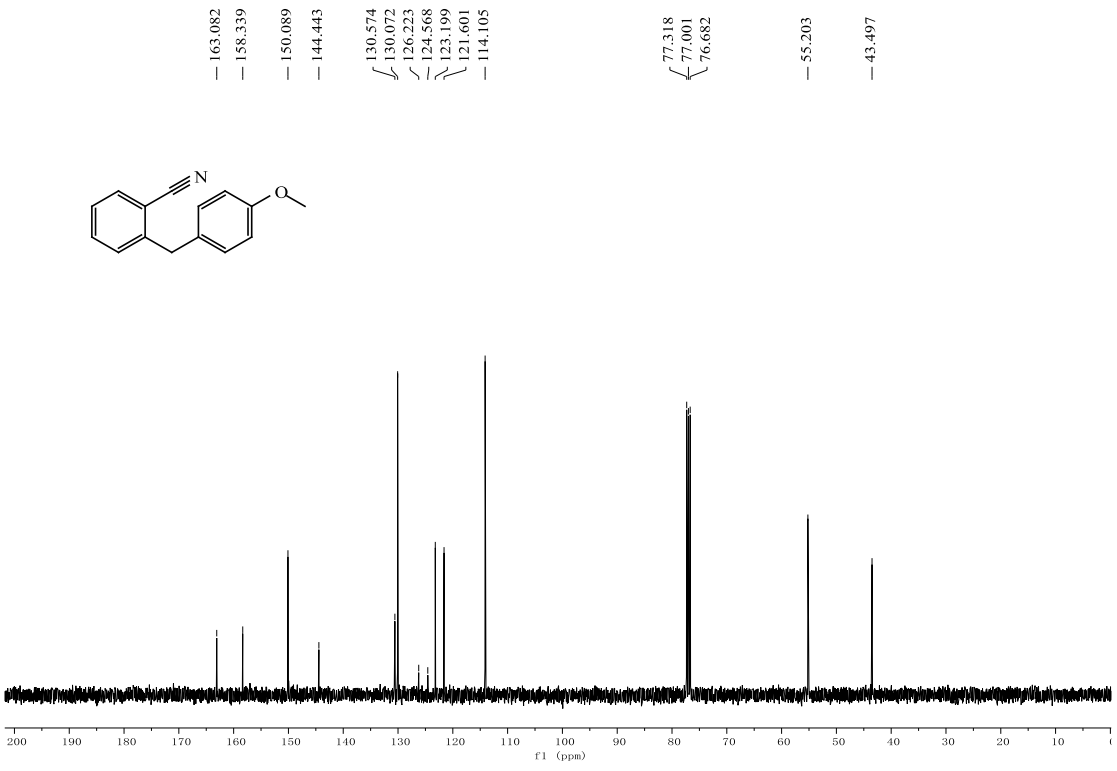
3-Chloro-4-(4-methoxybenzyl)benzonitrile (3g)



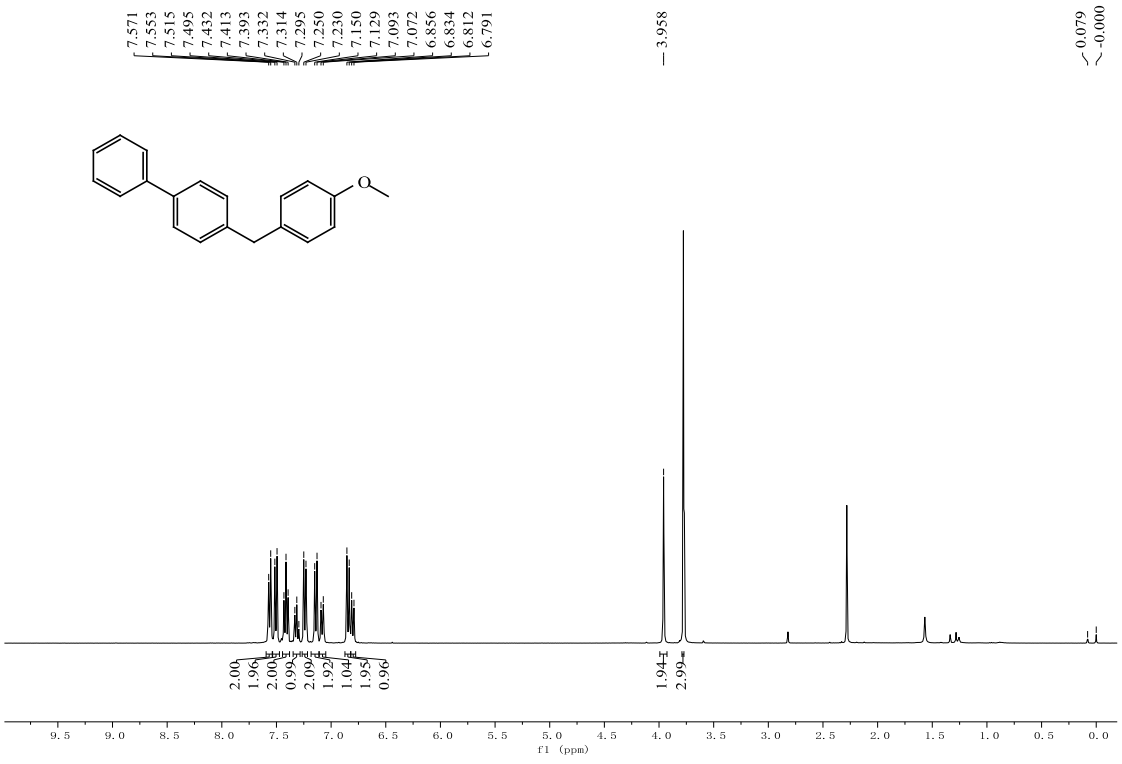


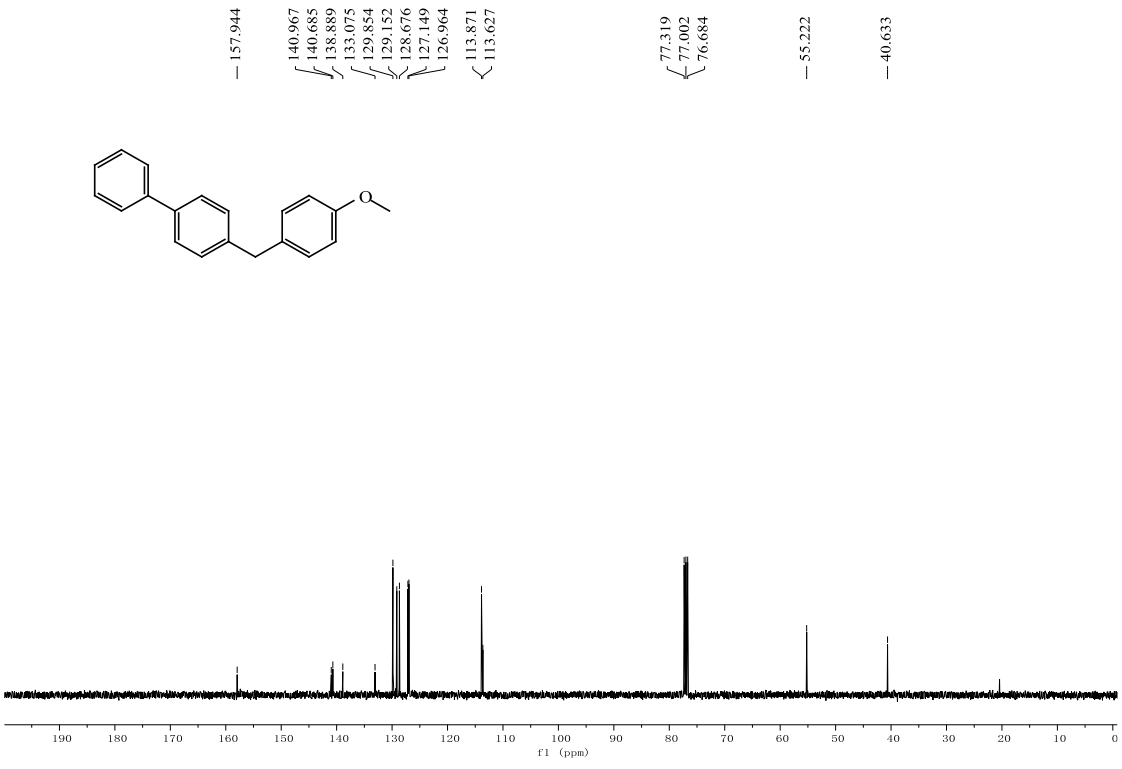
2-(4-Methoxybenzyl)benzonitrile (3h)



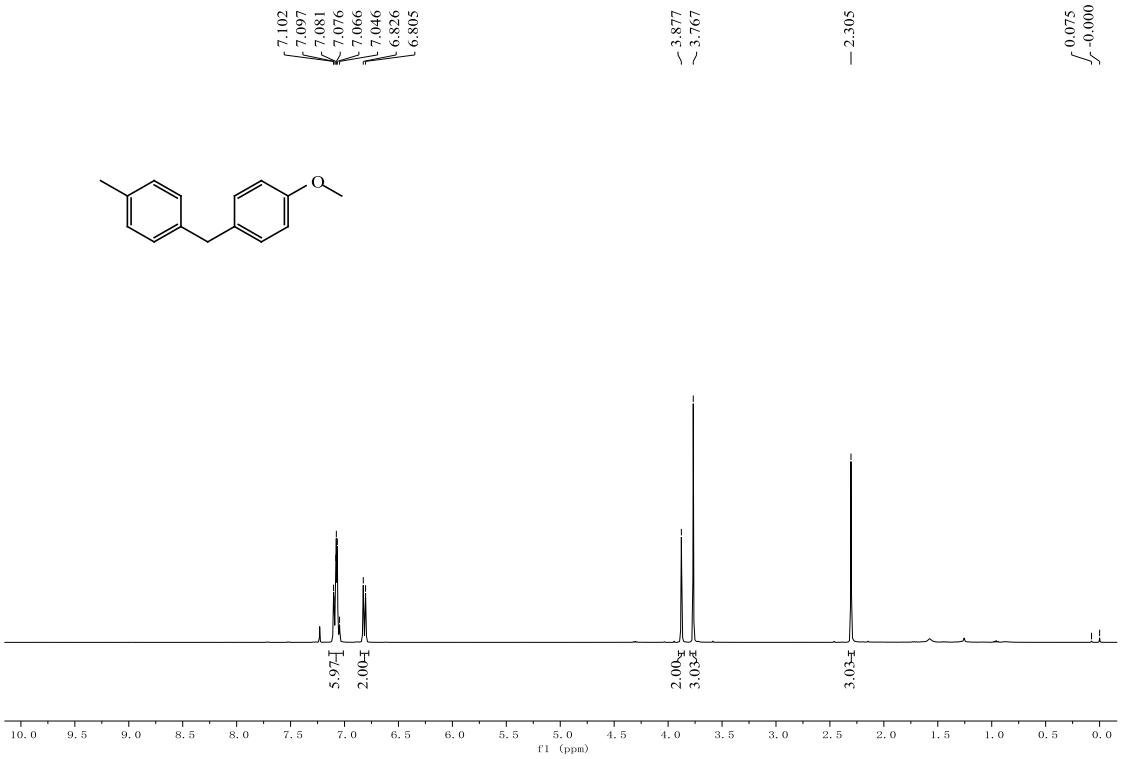


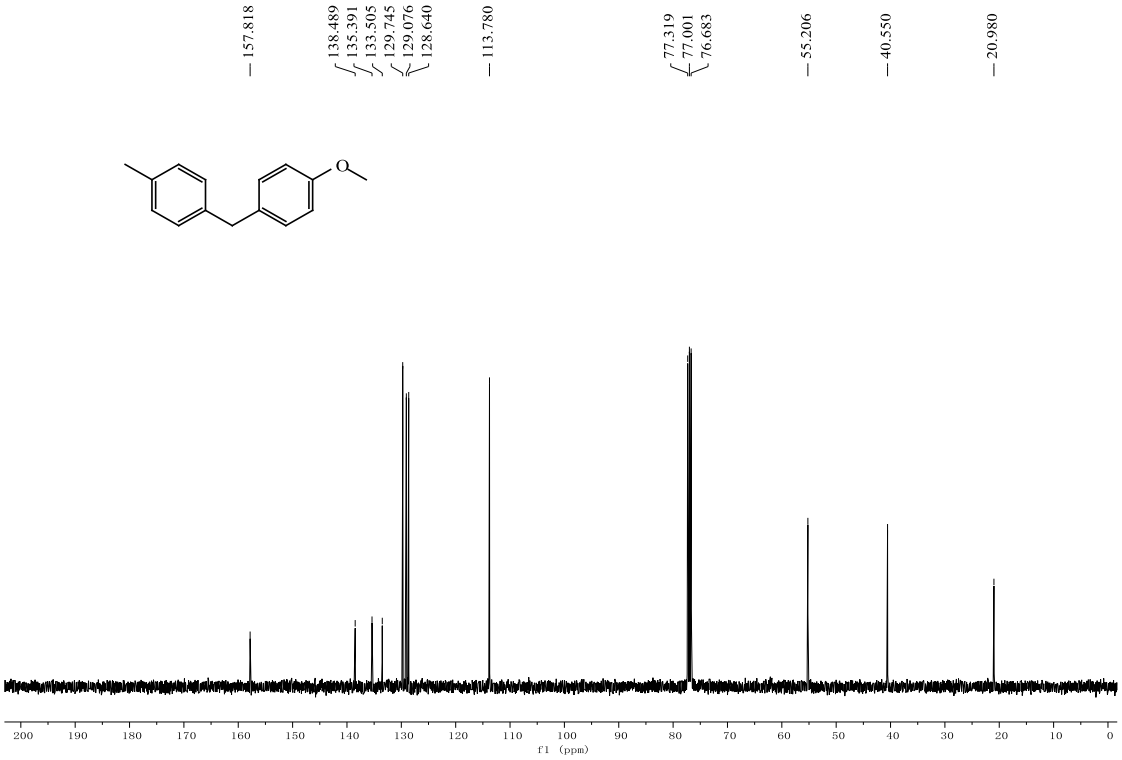
4-(4-Methoxybenzyl)-1,1'-biphenyl (3i)



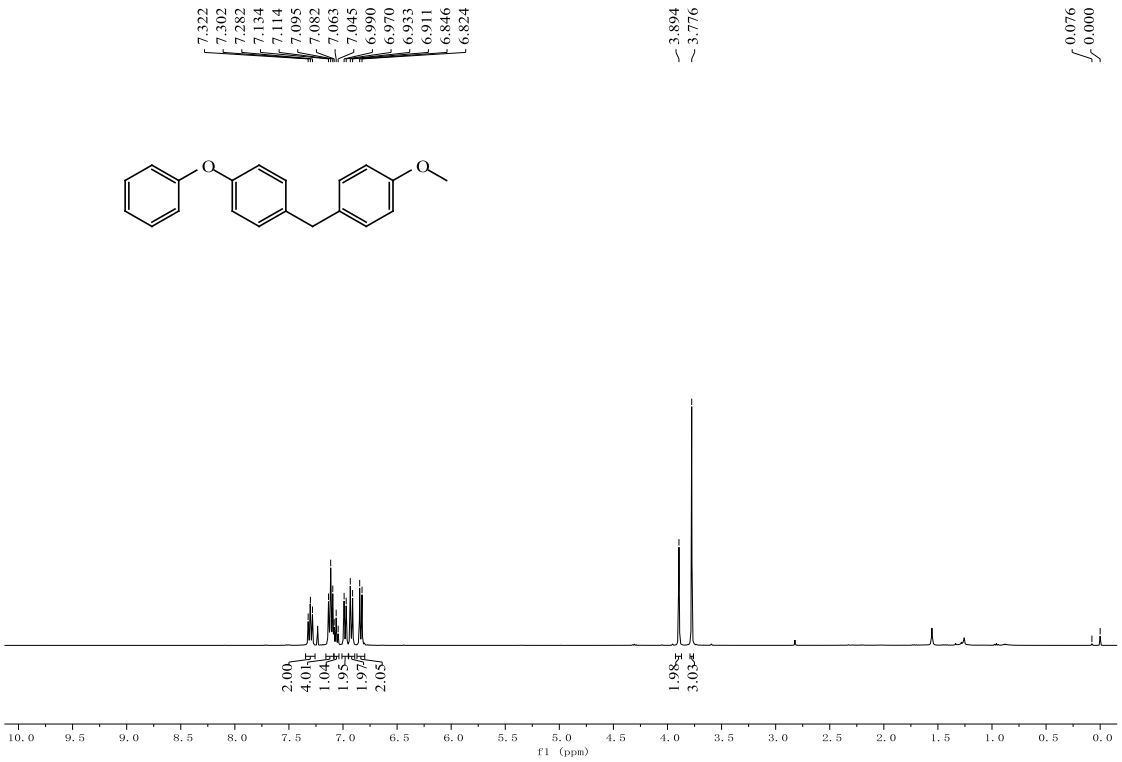


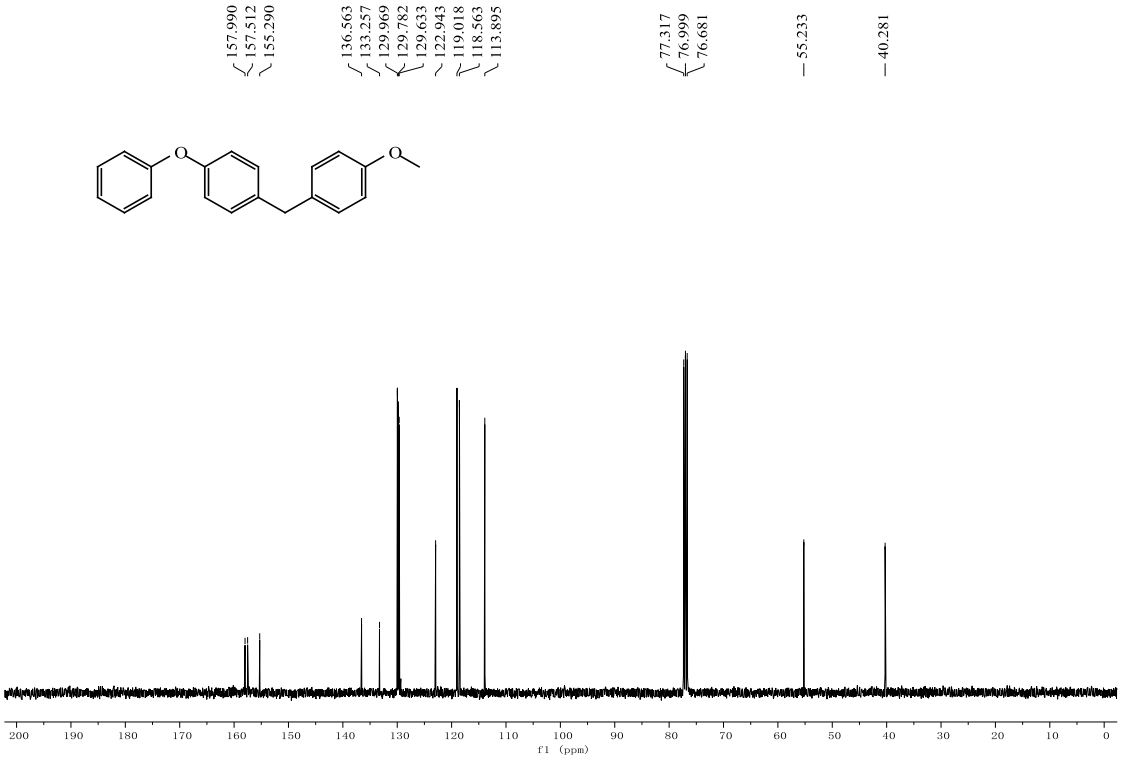
1-Methoxy-4-(4-methylbenzyl)benzene (3j)



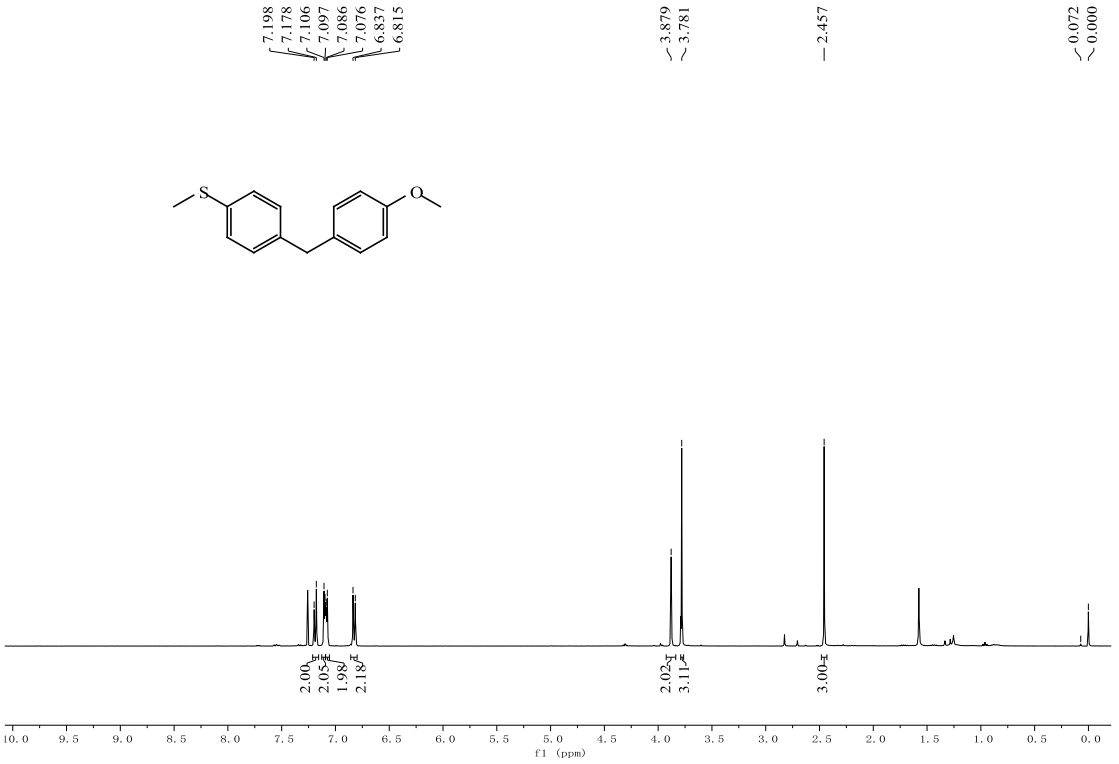


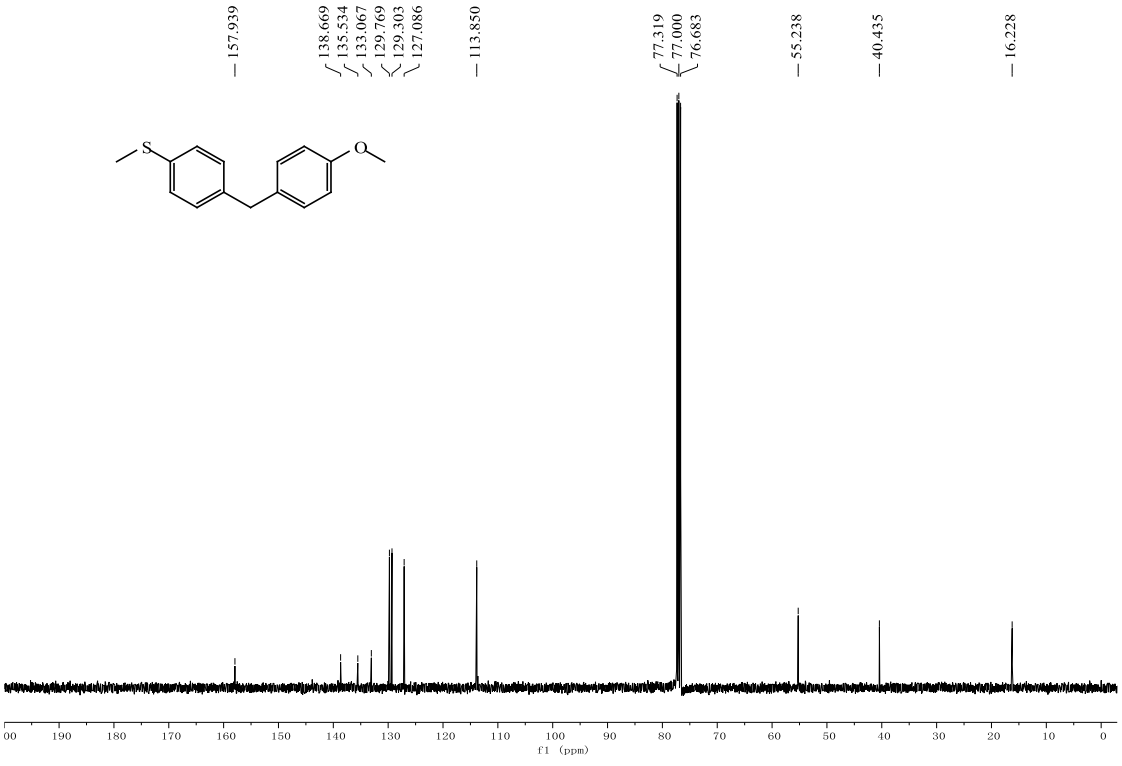
1-Methoxy-4-(4-phenoxybenzyl)benzene (3k)



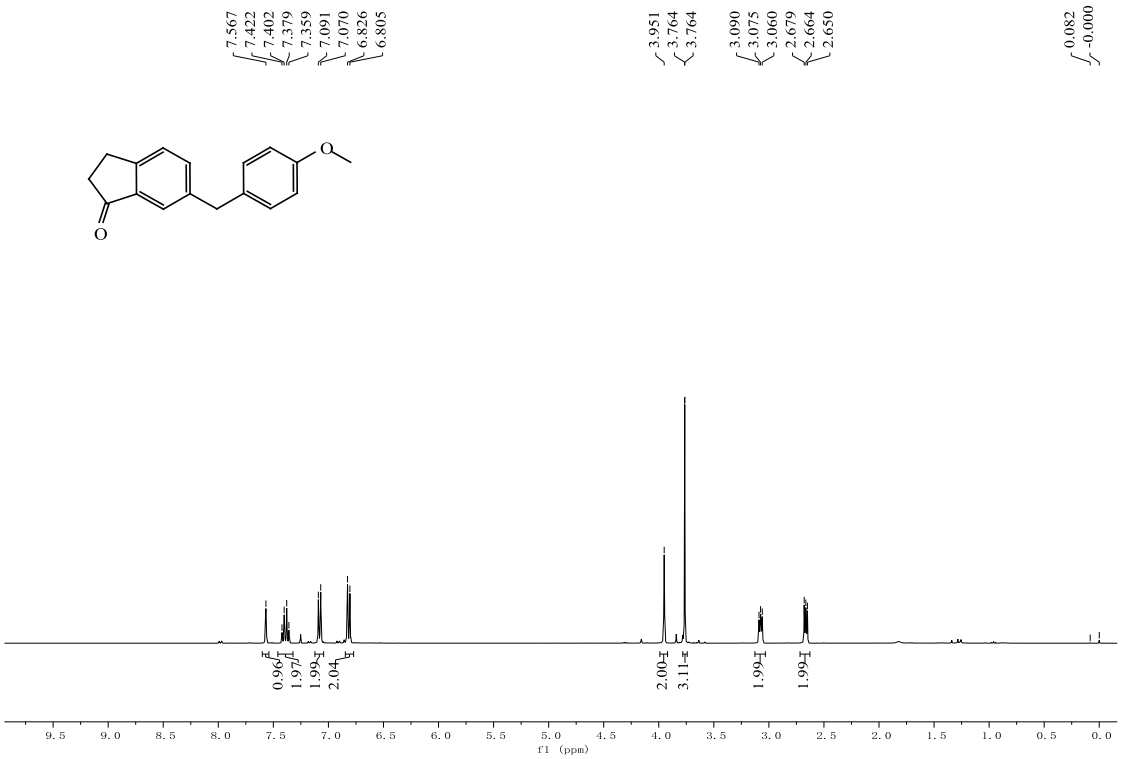


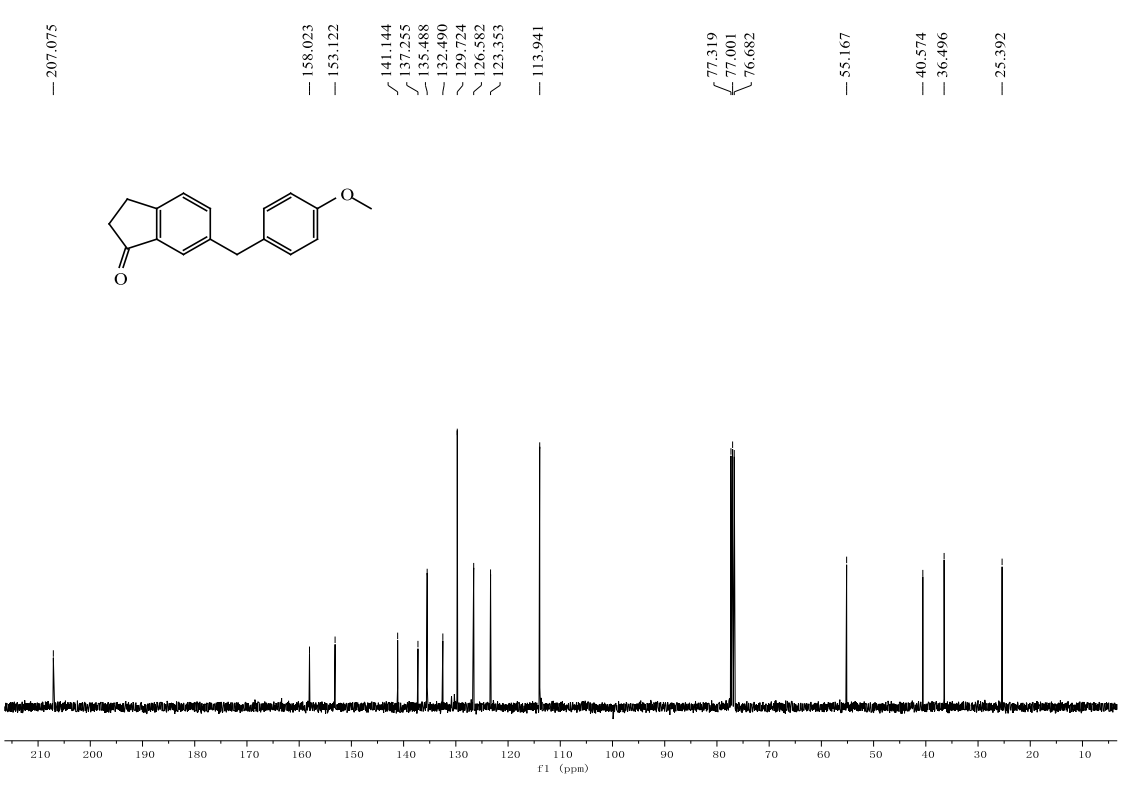
(4-(4-Methoxybenzyl)phenyl)(methyl)sulfane (3l)



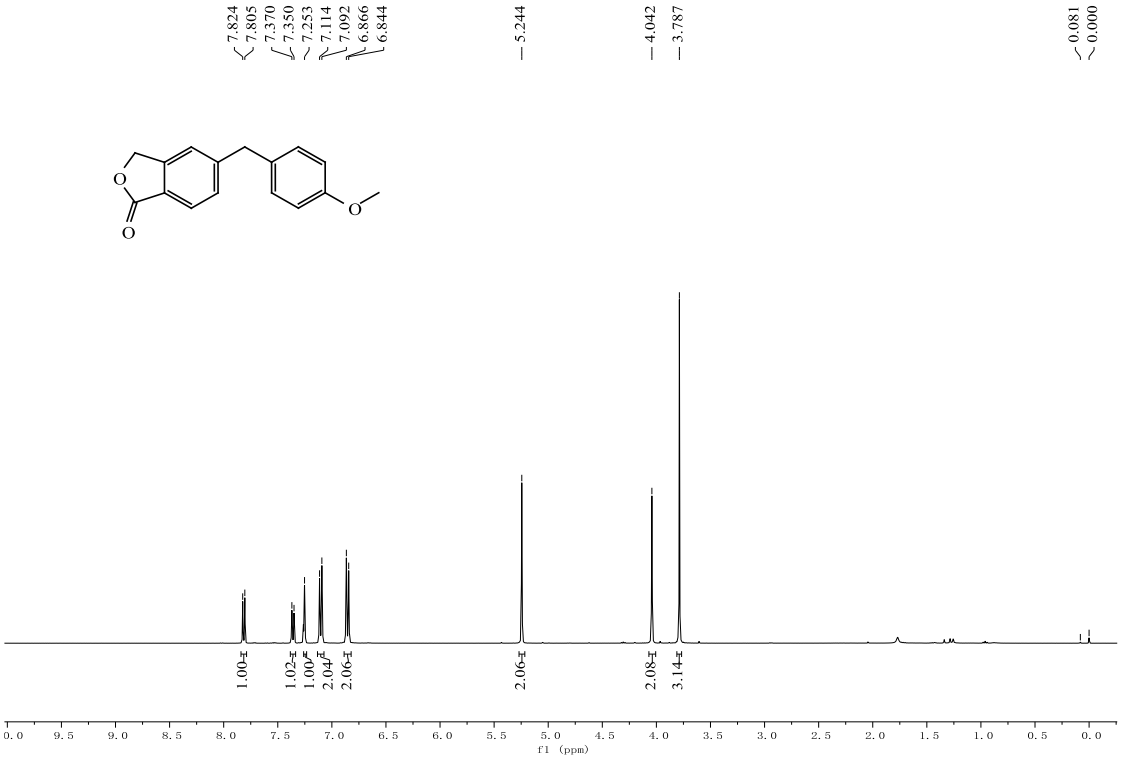


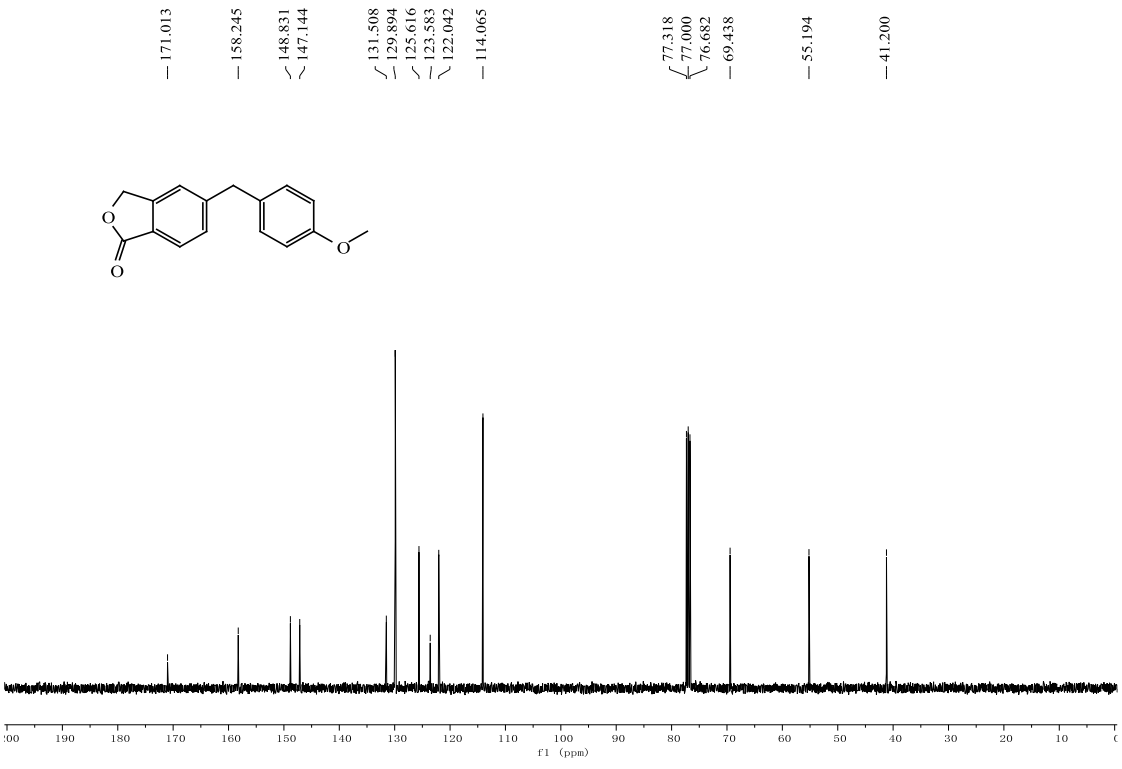
6-(4-Methoxybenzyl)-2,3-dihydro-1*H*-inden-1-one (3m)



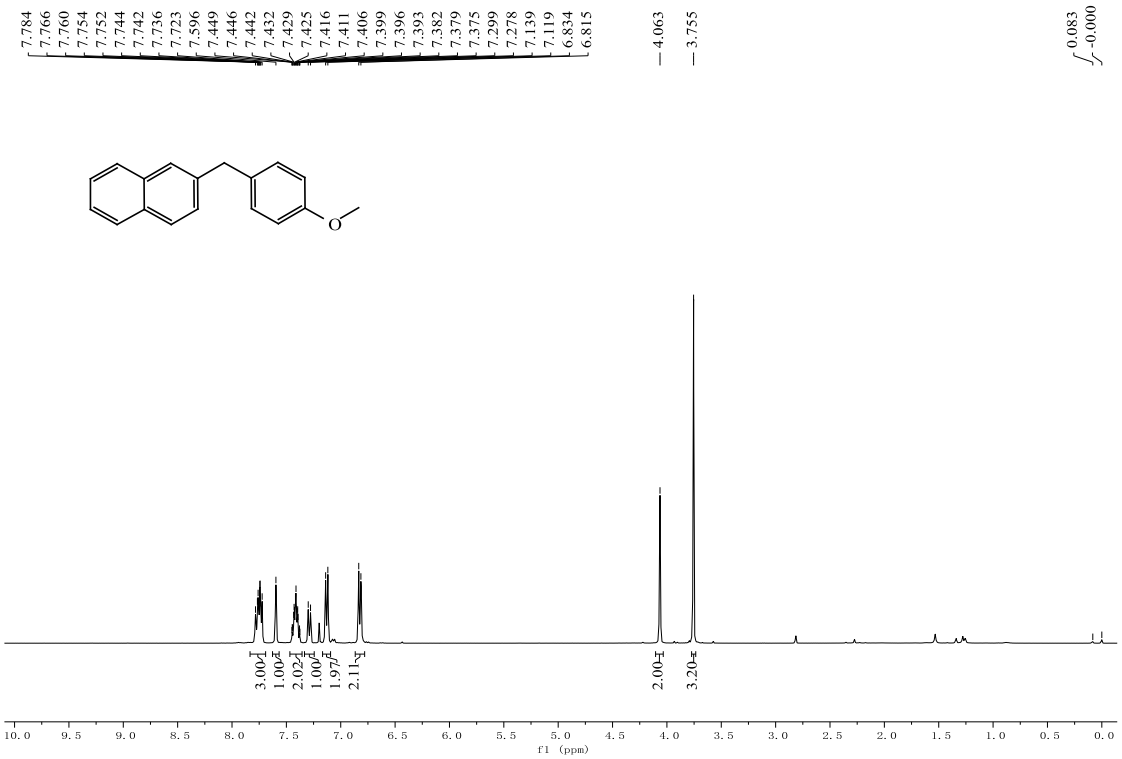


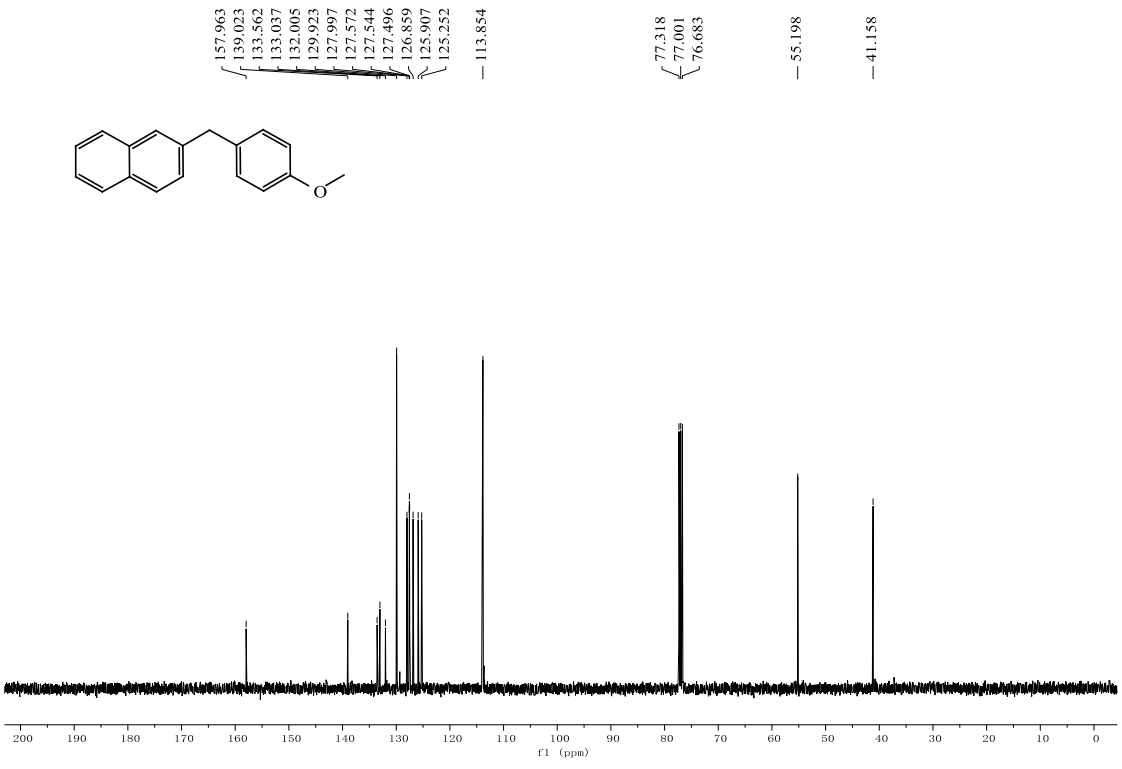
6-(4-Methoxybenzyl)isobenzofuran-1(3*H*)-one (3n)



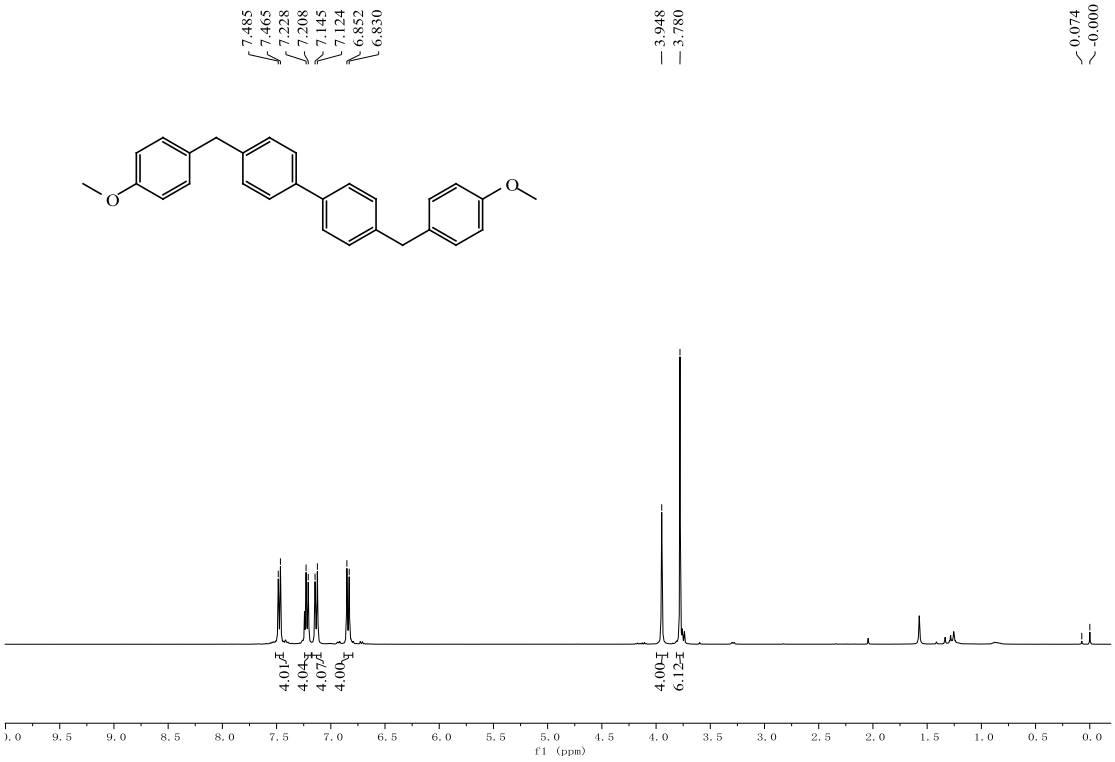


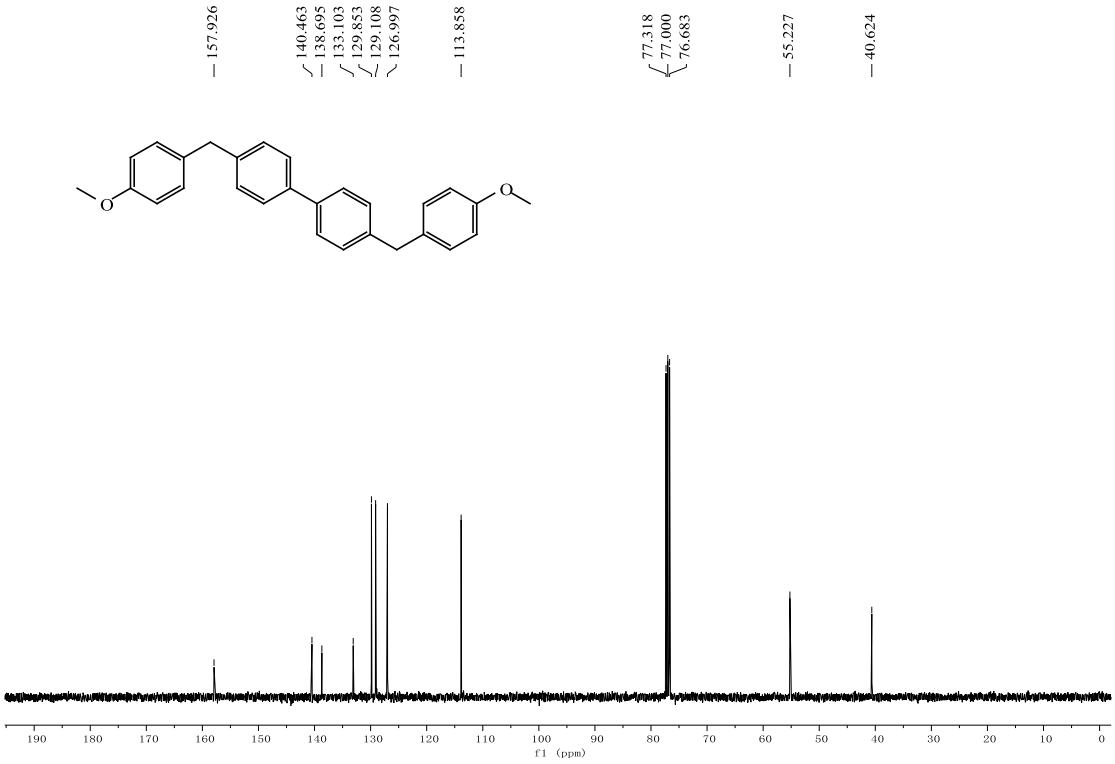
2-(4-Methoxybenzyl)naphthalene (3o)



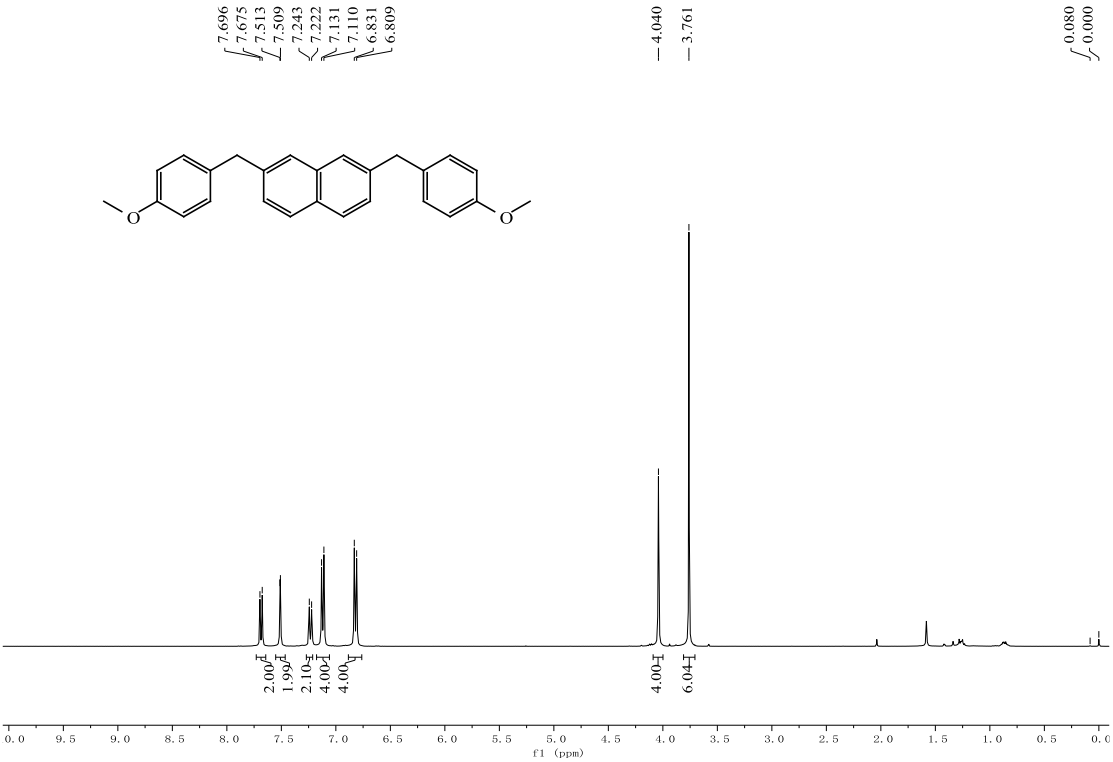


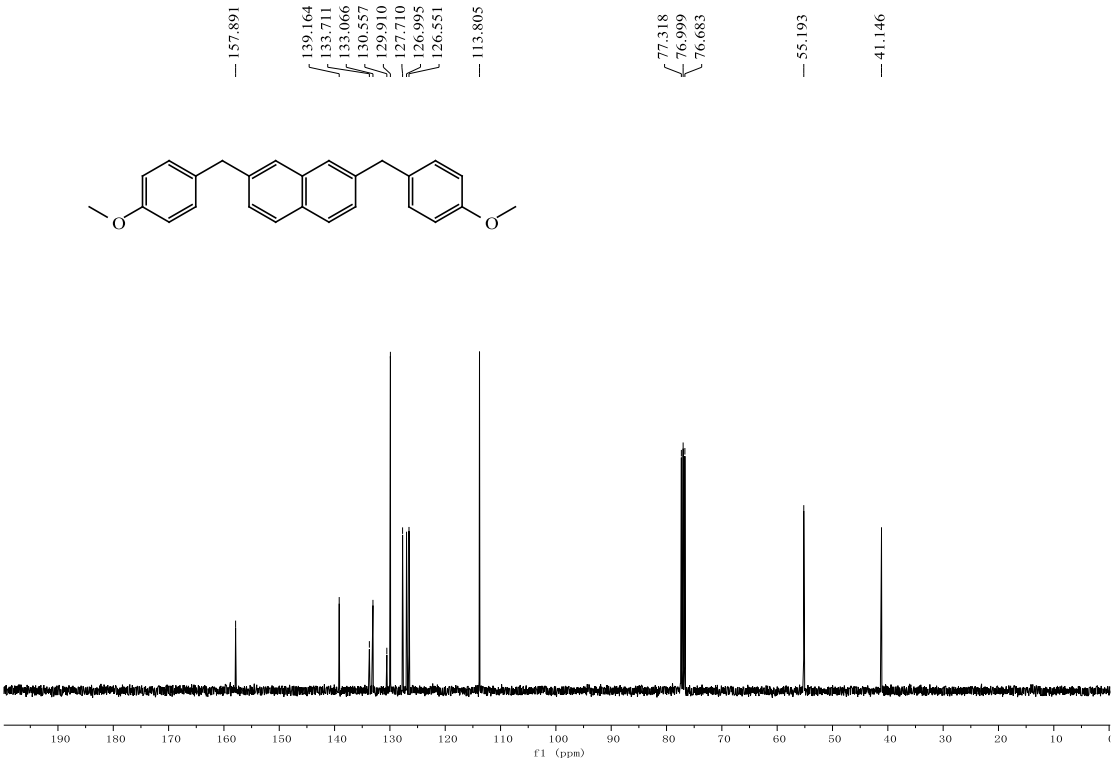
4,4'-bis(4-methoxybenzyl)-1,1'-biphenyl (3p)



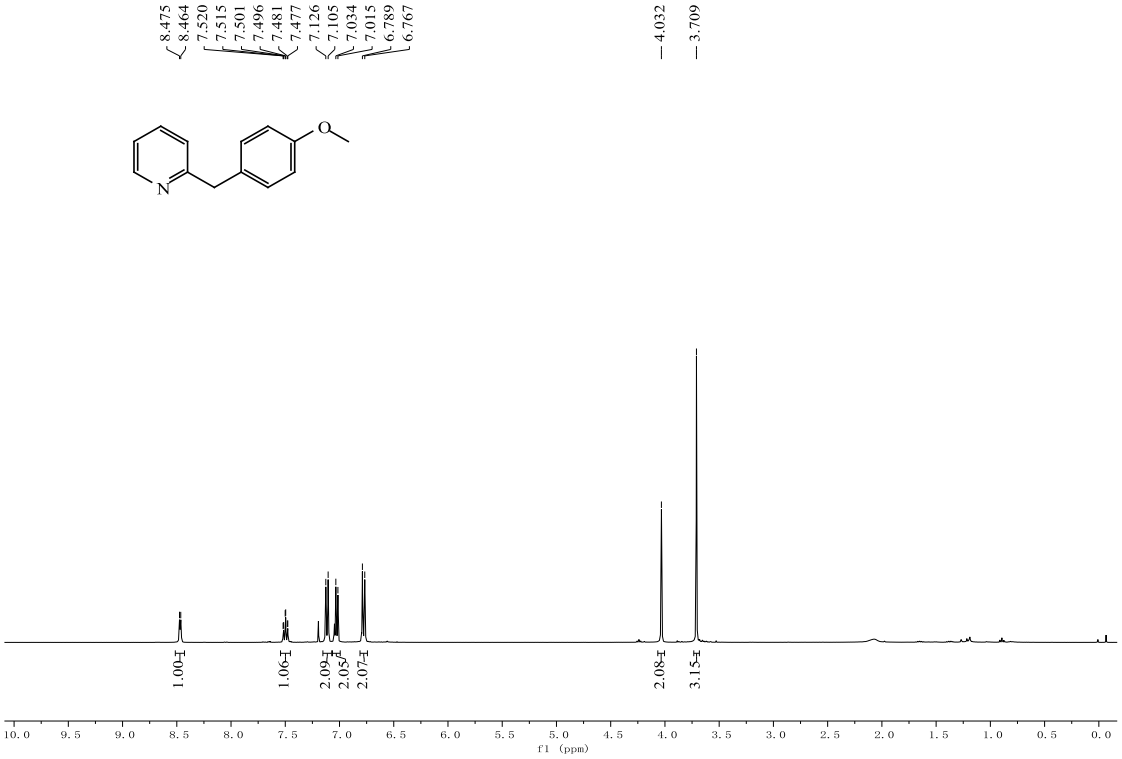


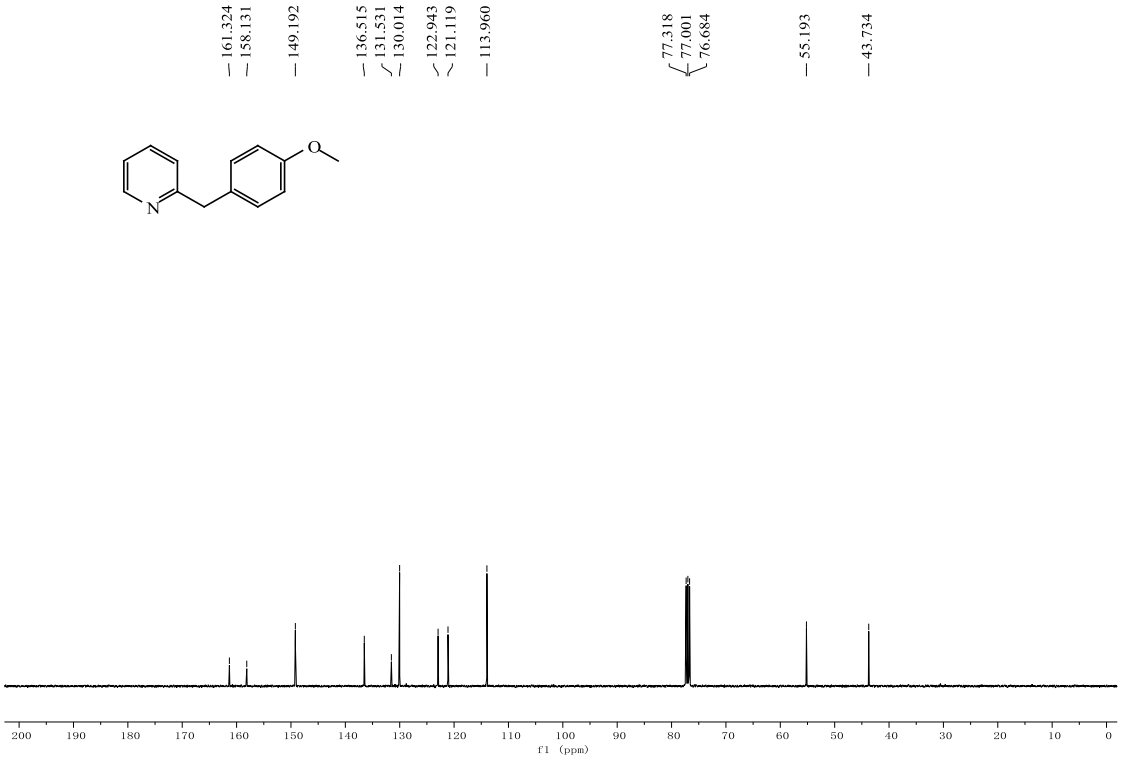
2,7-bis(4-methoxybenzyl)naphthalene (3q)



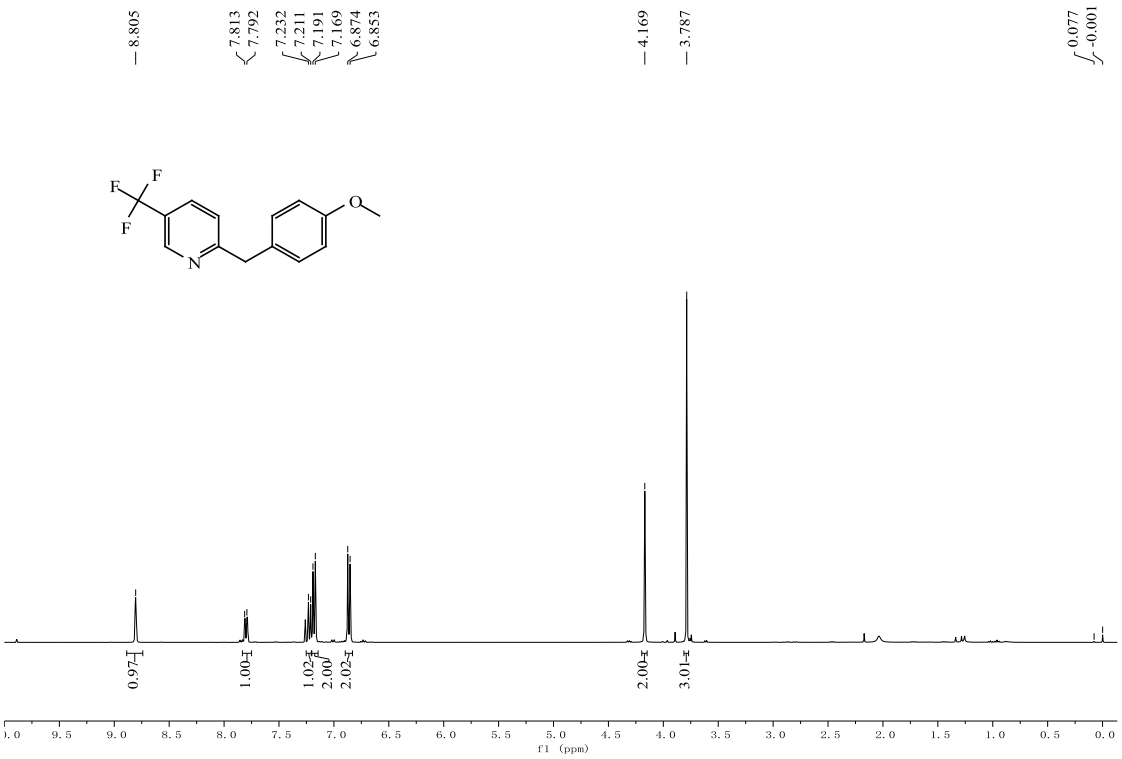


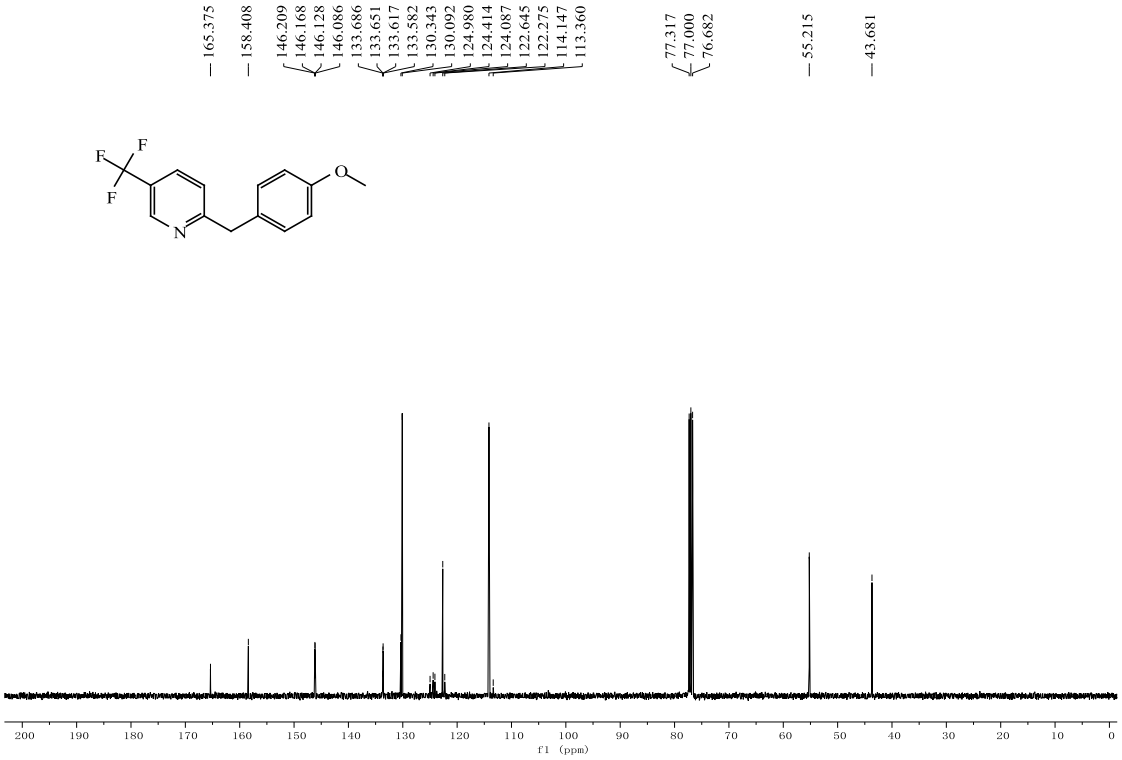
2-(4-Methoxybenzyl)pyridine (3r)



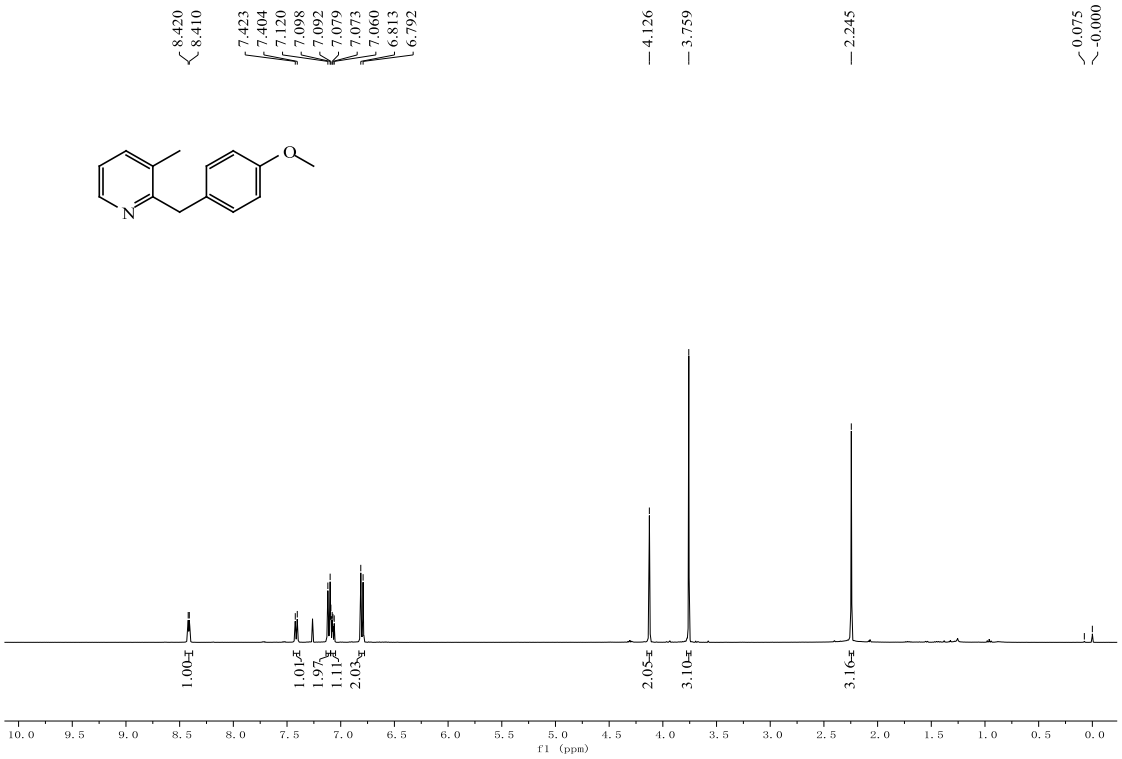


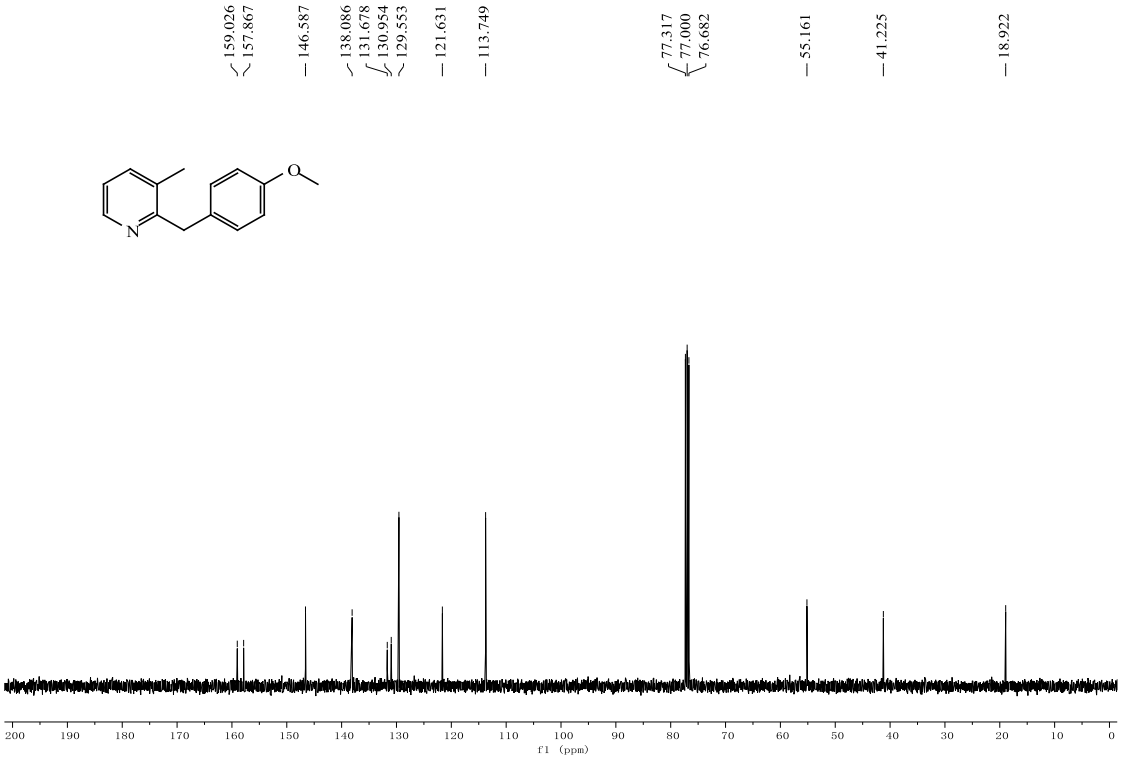
2-(4-Methoxybenzyl)-5-(trifluoromethyl)pyridine (3s)



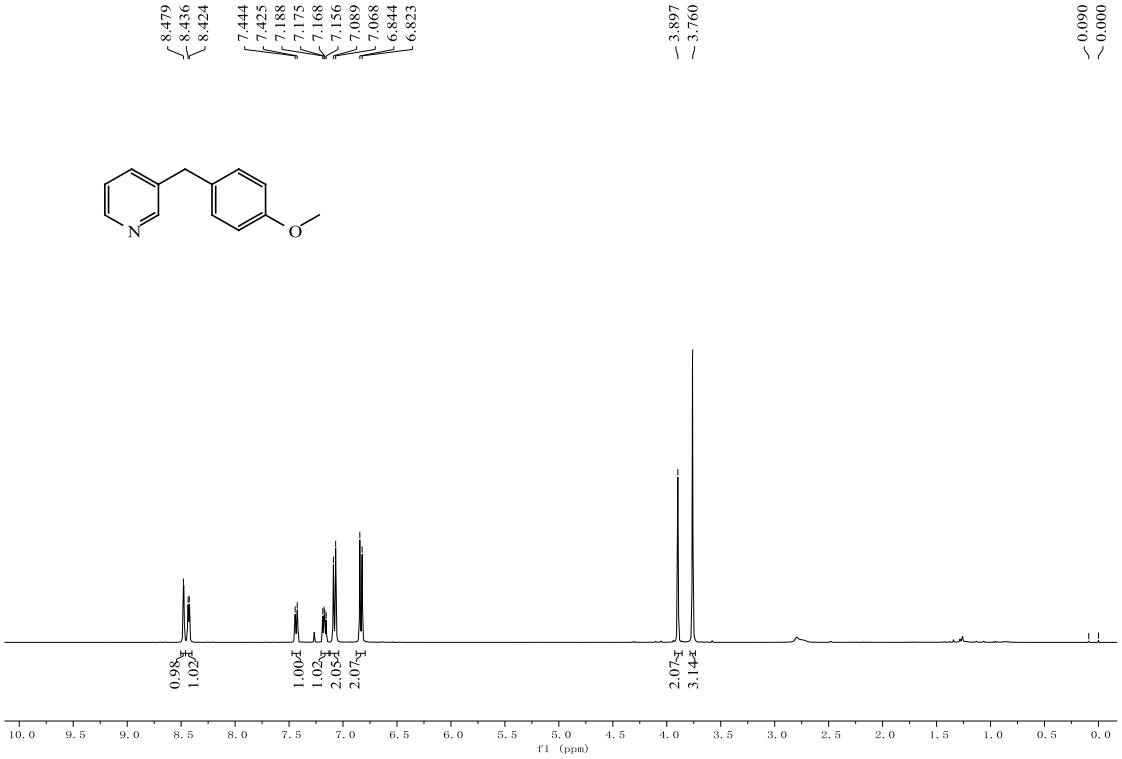


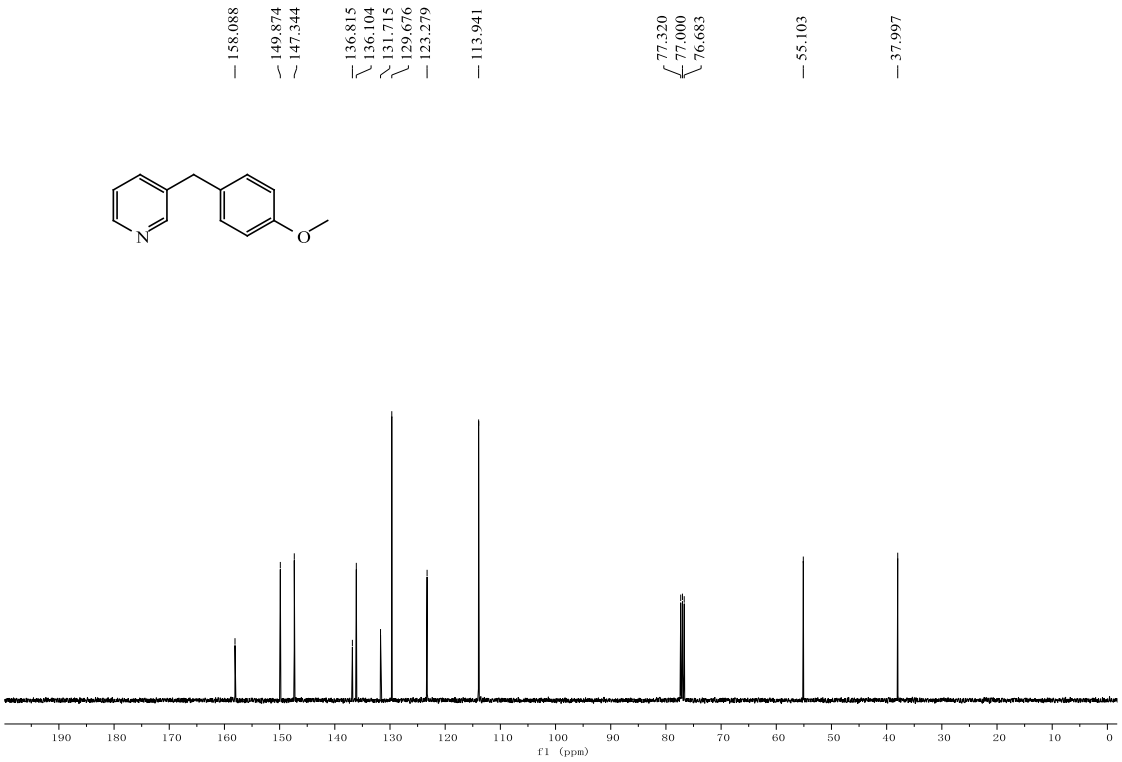
2-(4-Methoxybenzyl)-3-methylpyridine (3u)



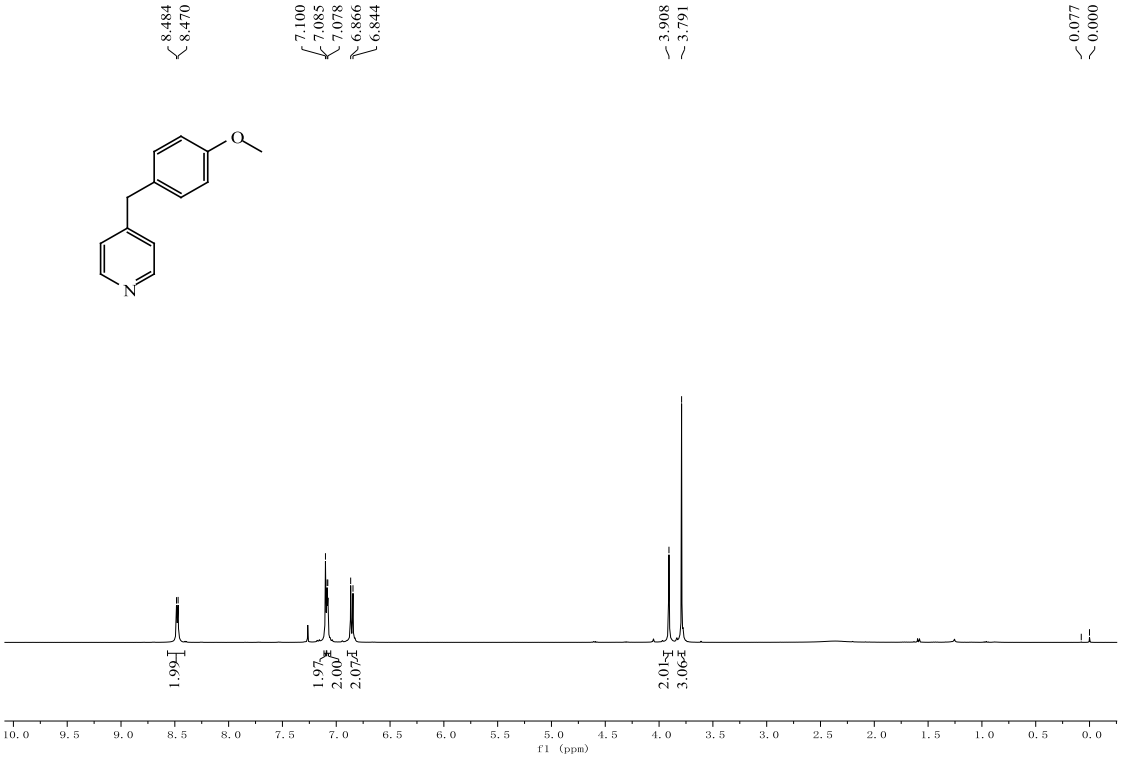


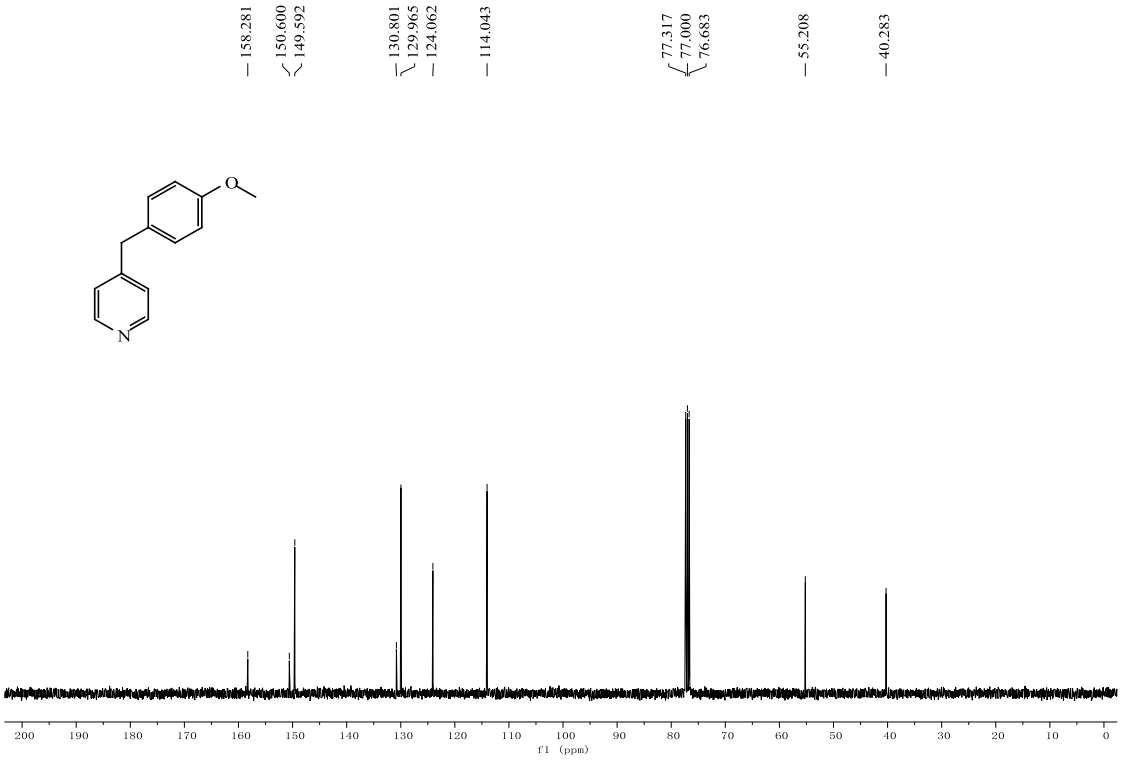
3-(4-Methoxybenzyl)pyridine (3v)



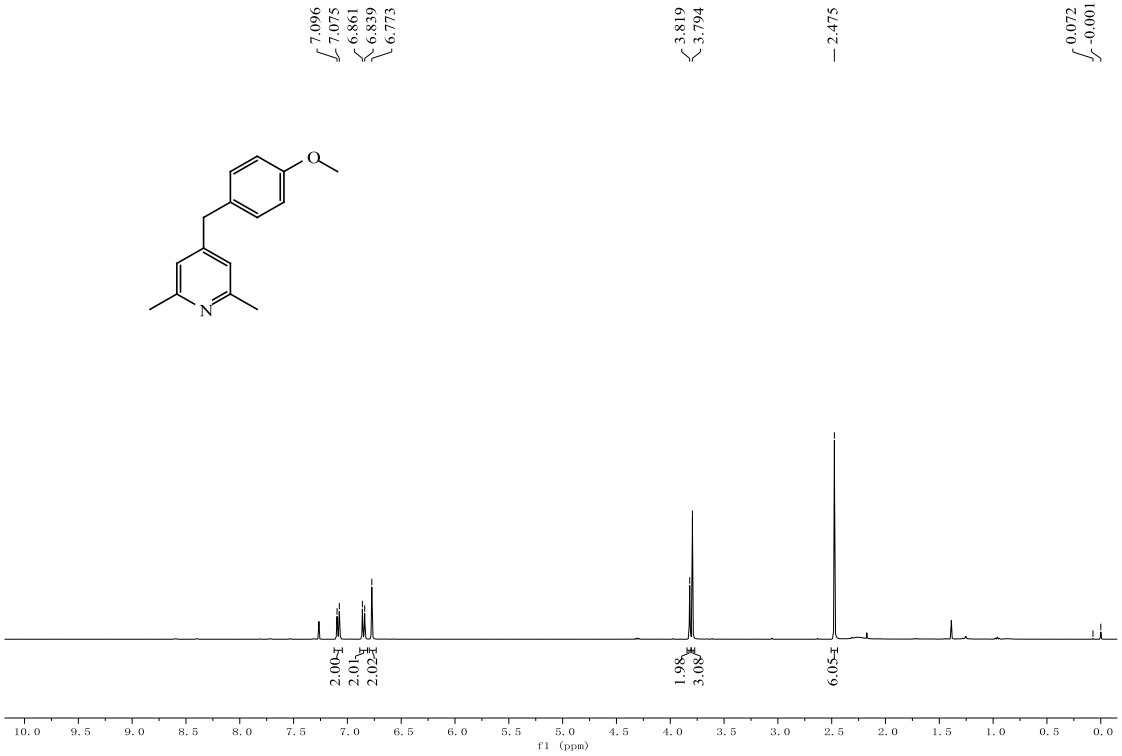


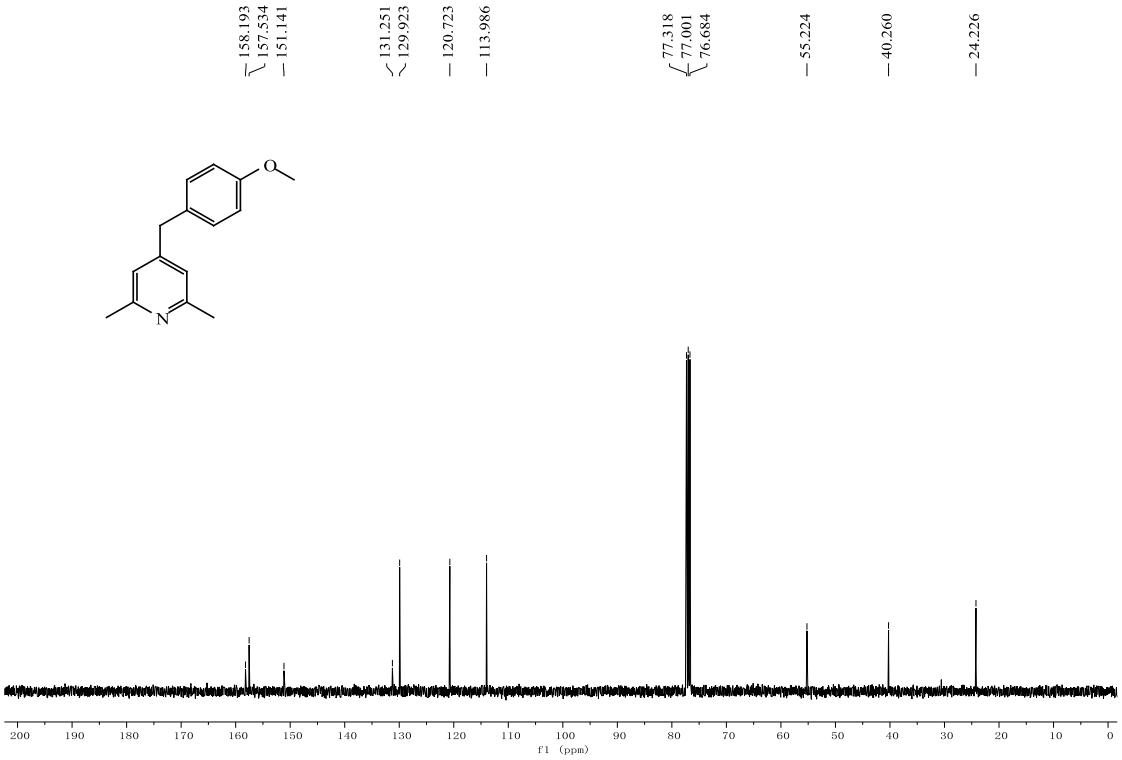
4-(4-Methoxybenzyl)pyridine (3w)



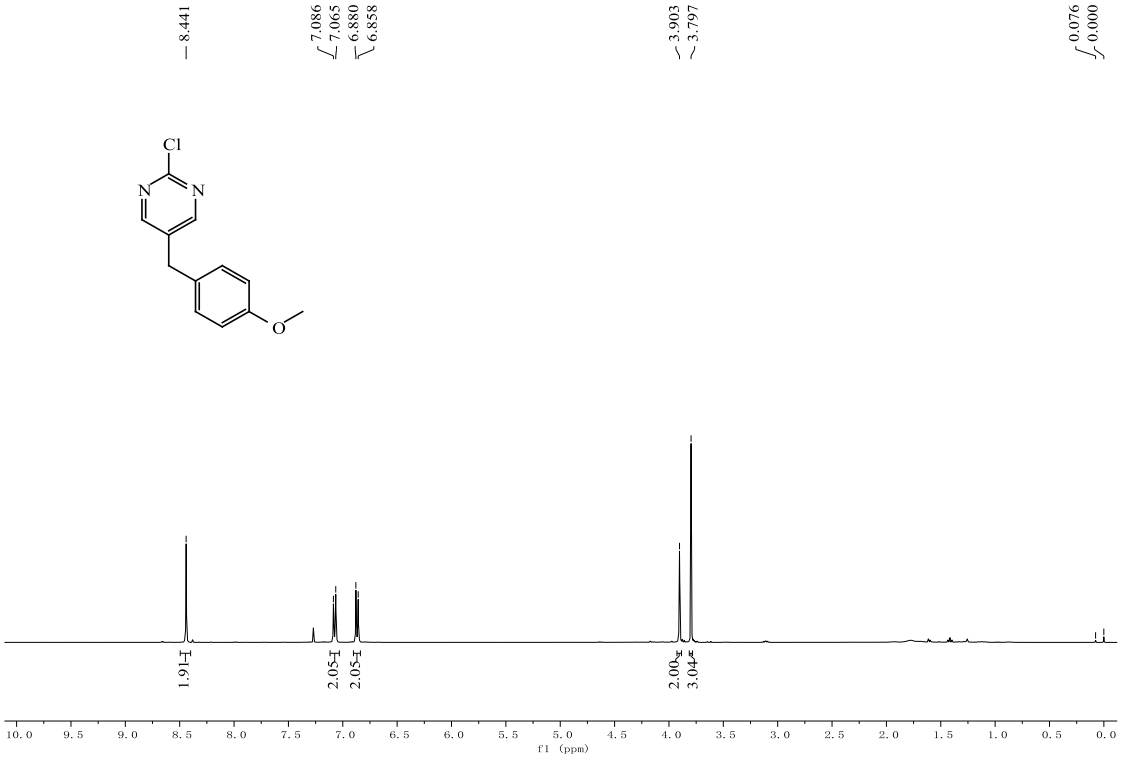


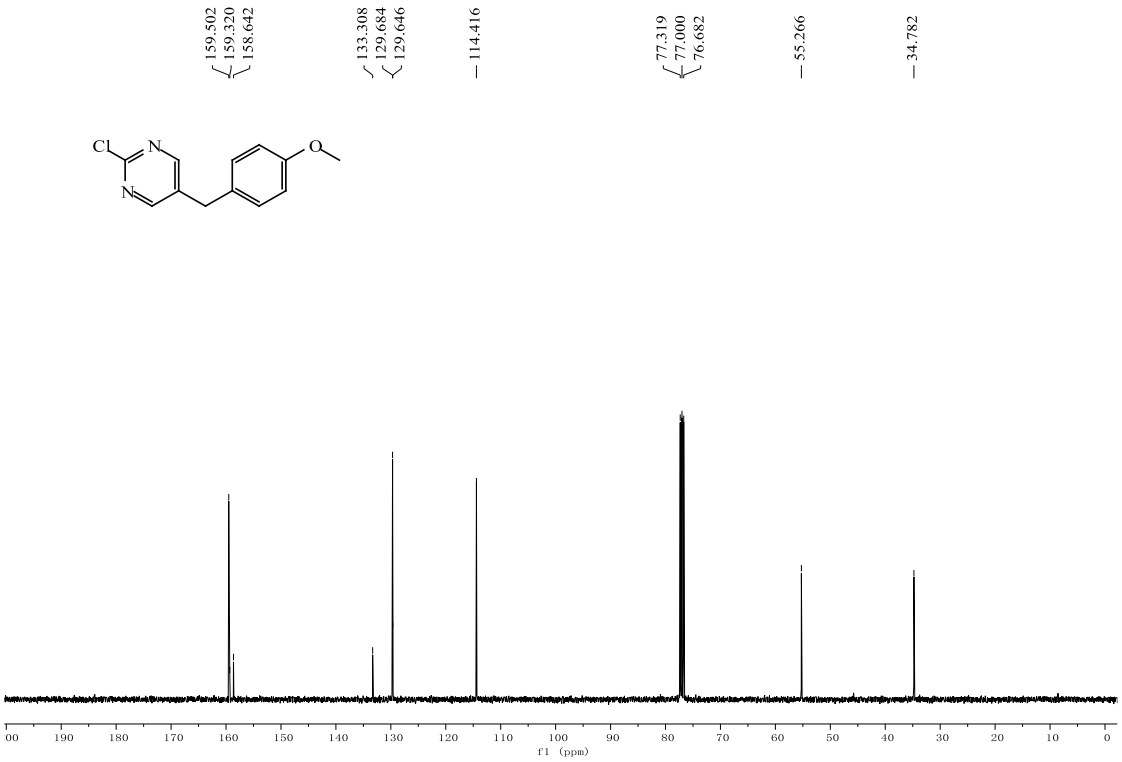
4-(4-Methoxybenzyl)-2,6-dimethylpyridine (3x)



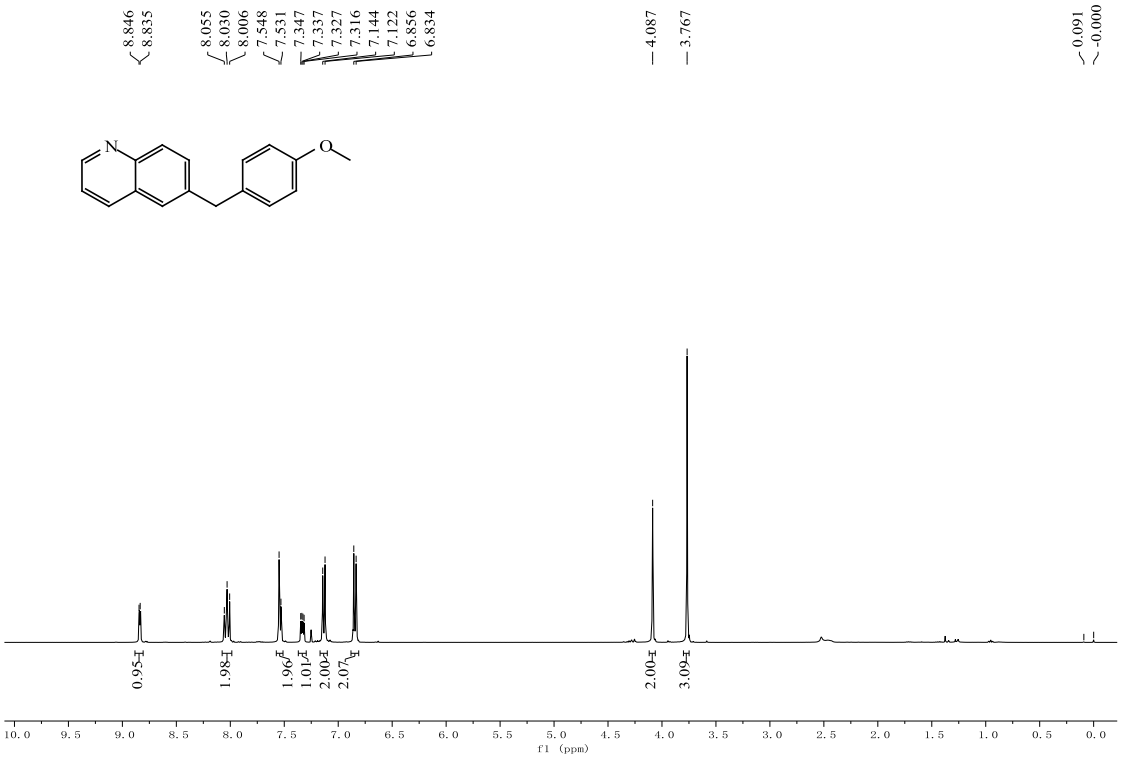


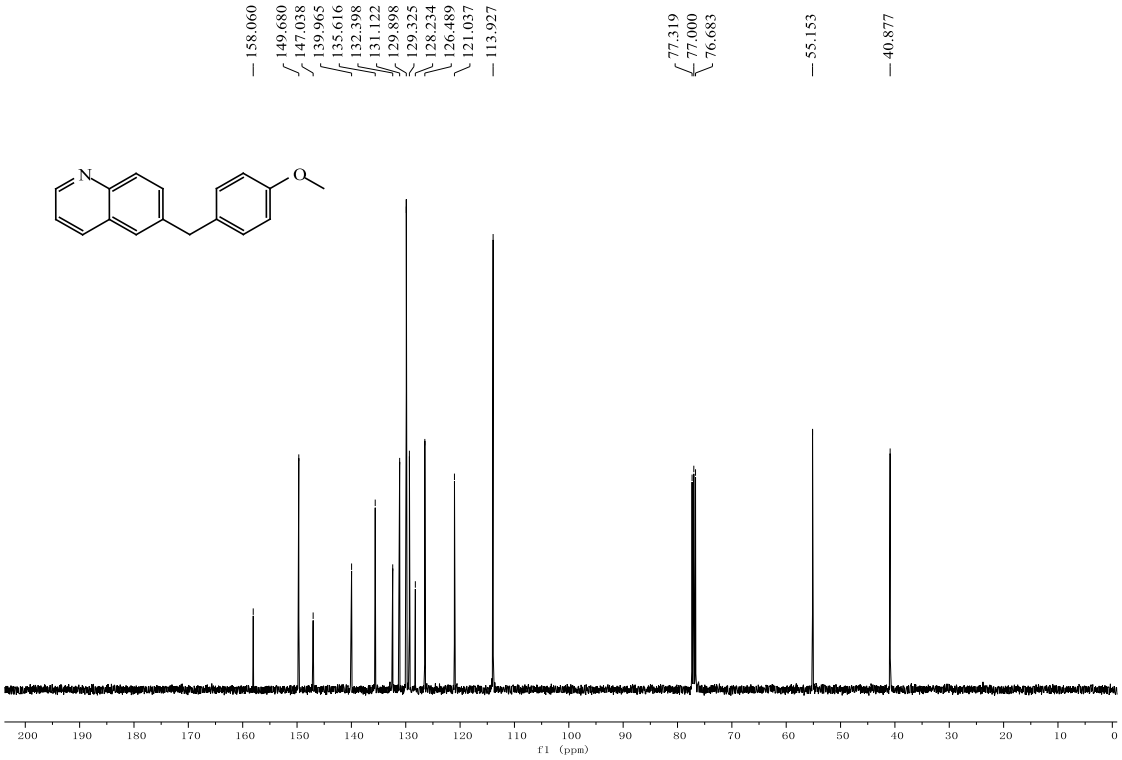
2-Chloro-5-(4-methoxybenzyl)pyrimidine (3y)



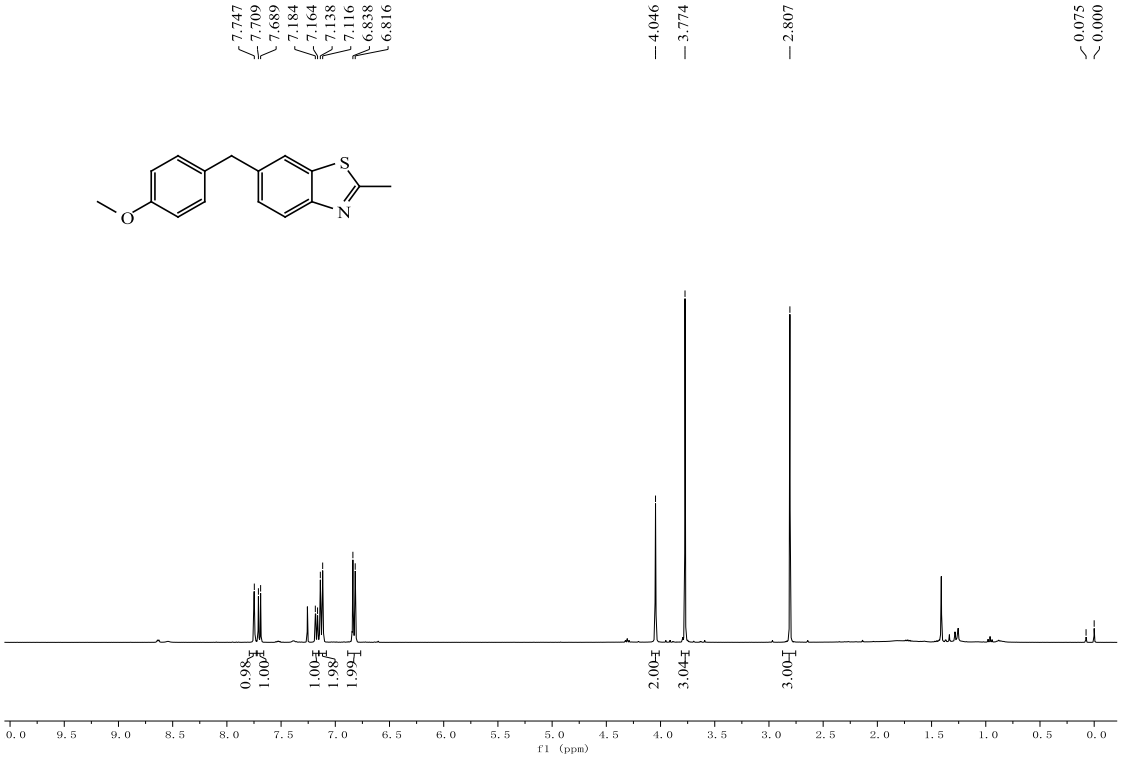


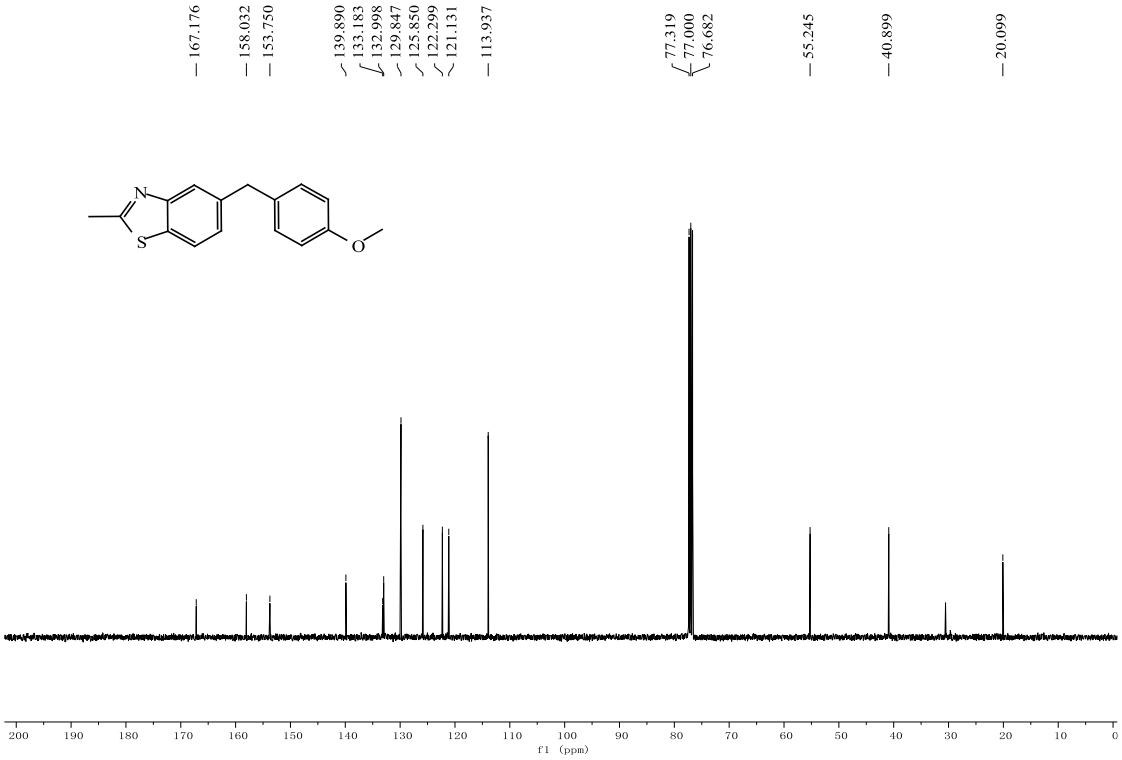
6-(4-Methoxybenzyl)quinoline (3z)



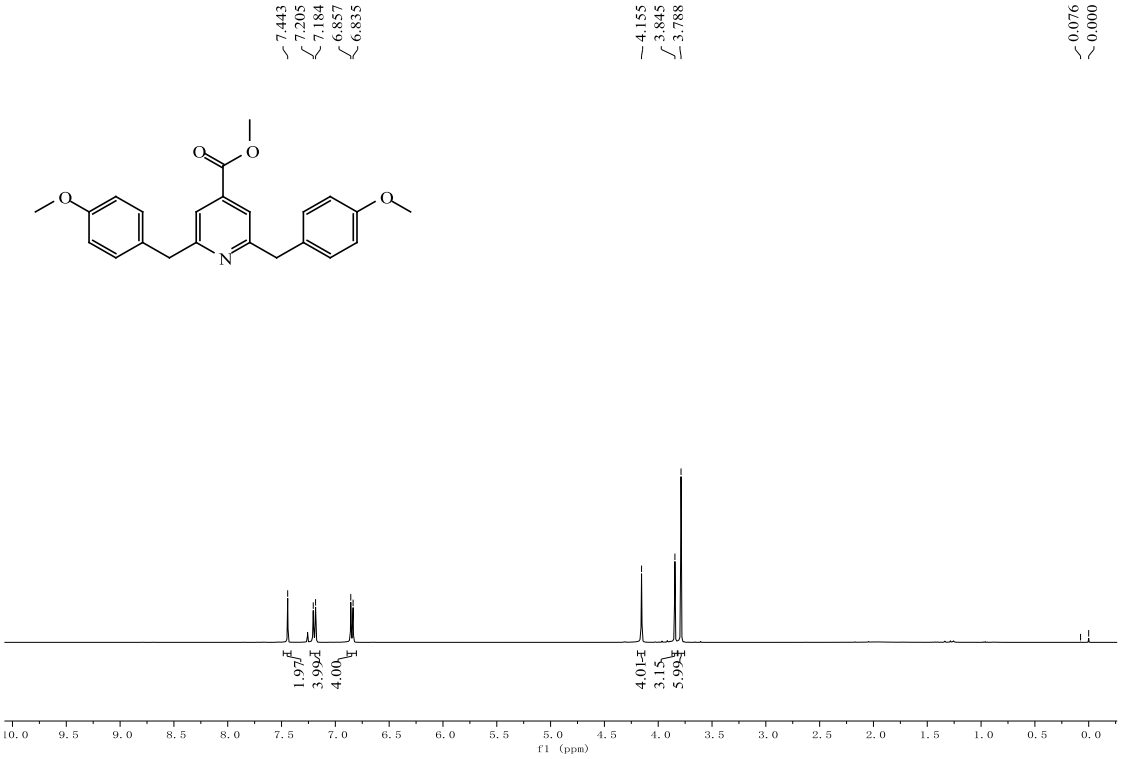


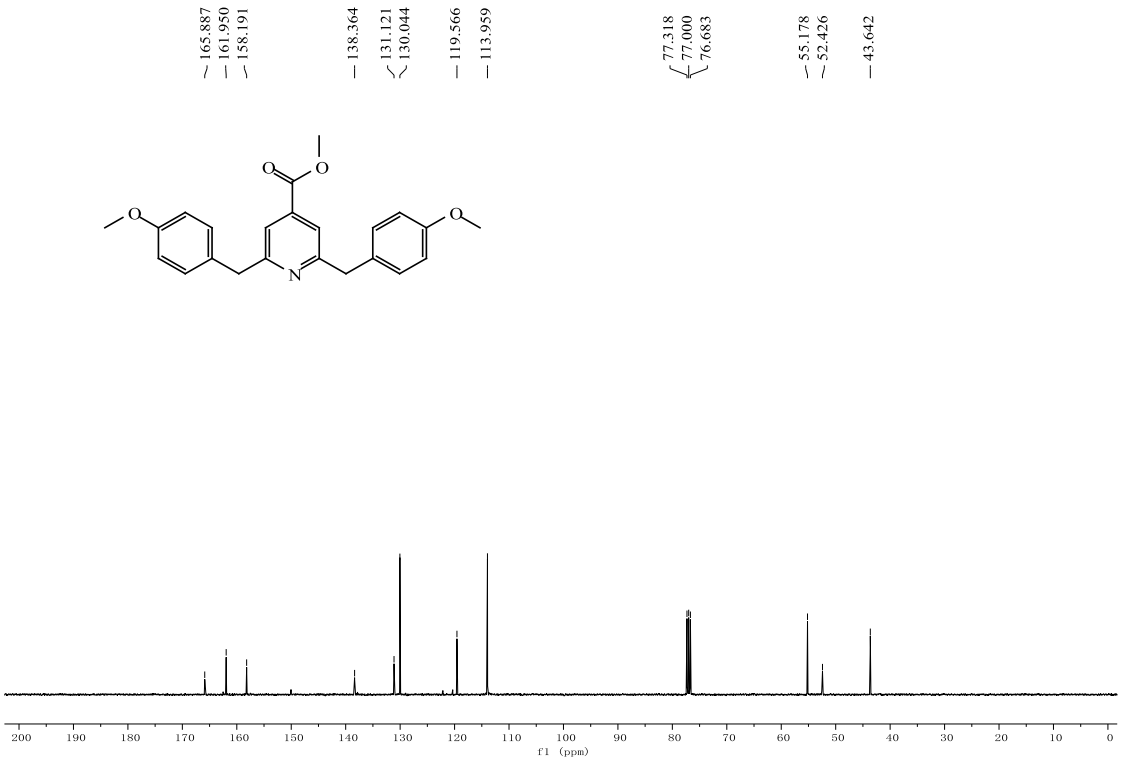
6-(4-Methoxybenzyl)-2-methylbenzo[*d*]thiazole (3aa)



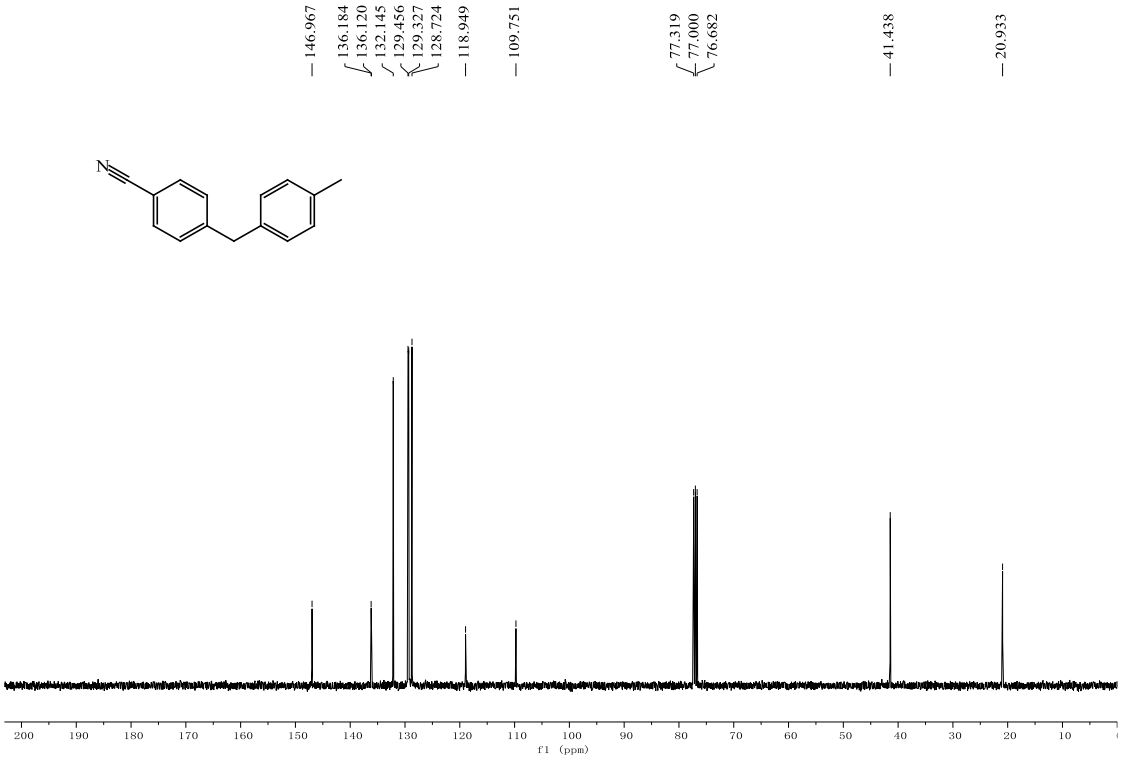


Methyl 2,6-bis(4-methoxybenzyl)isonicotinate (3ab)

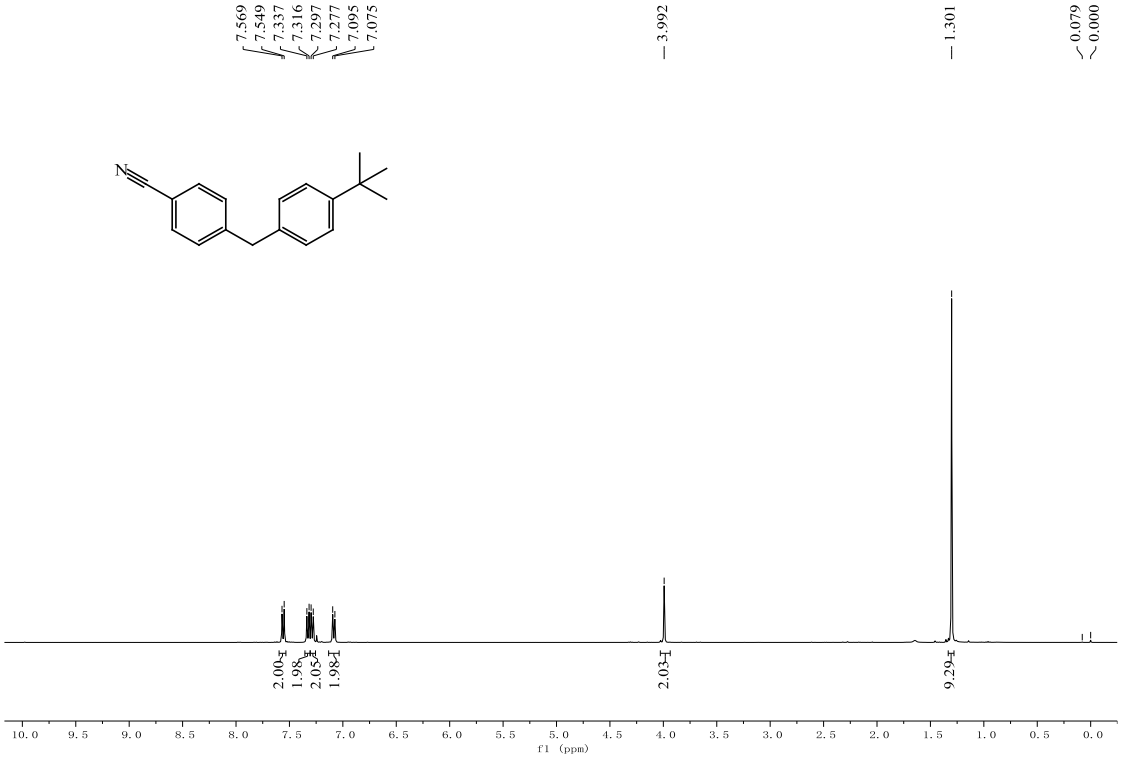


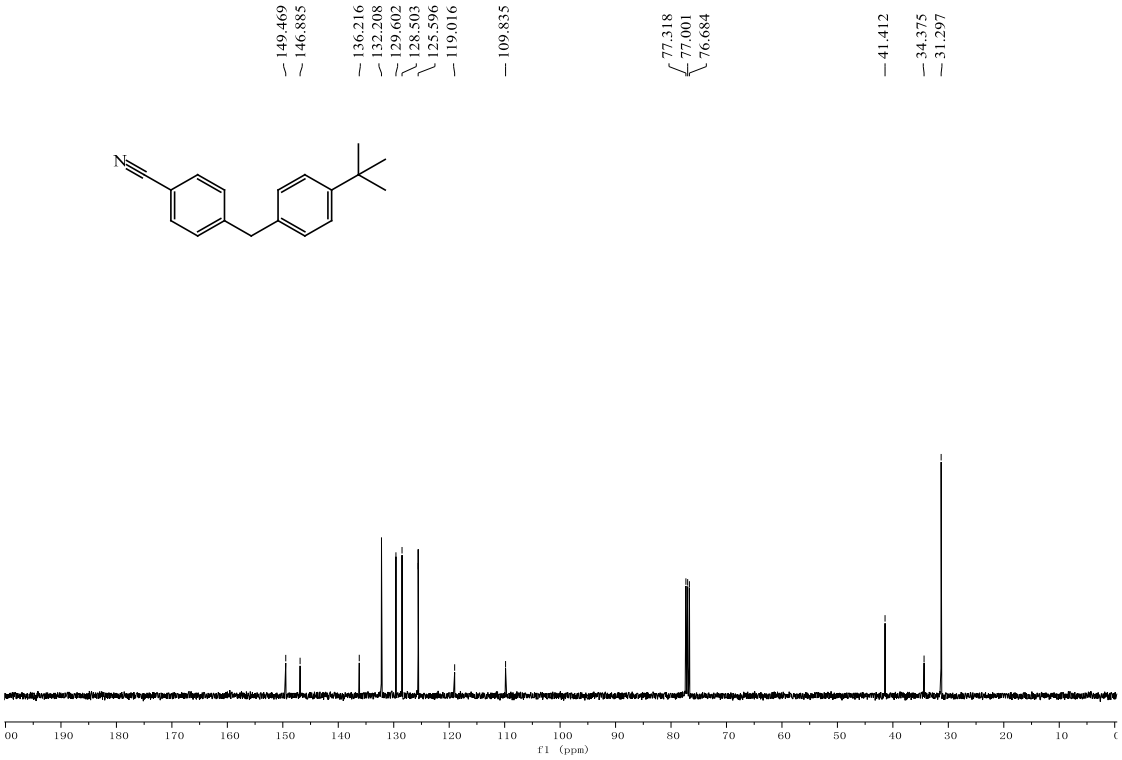


4-(4-Methylbenzyl)benzonitrile (4b)

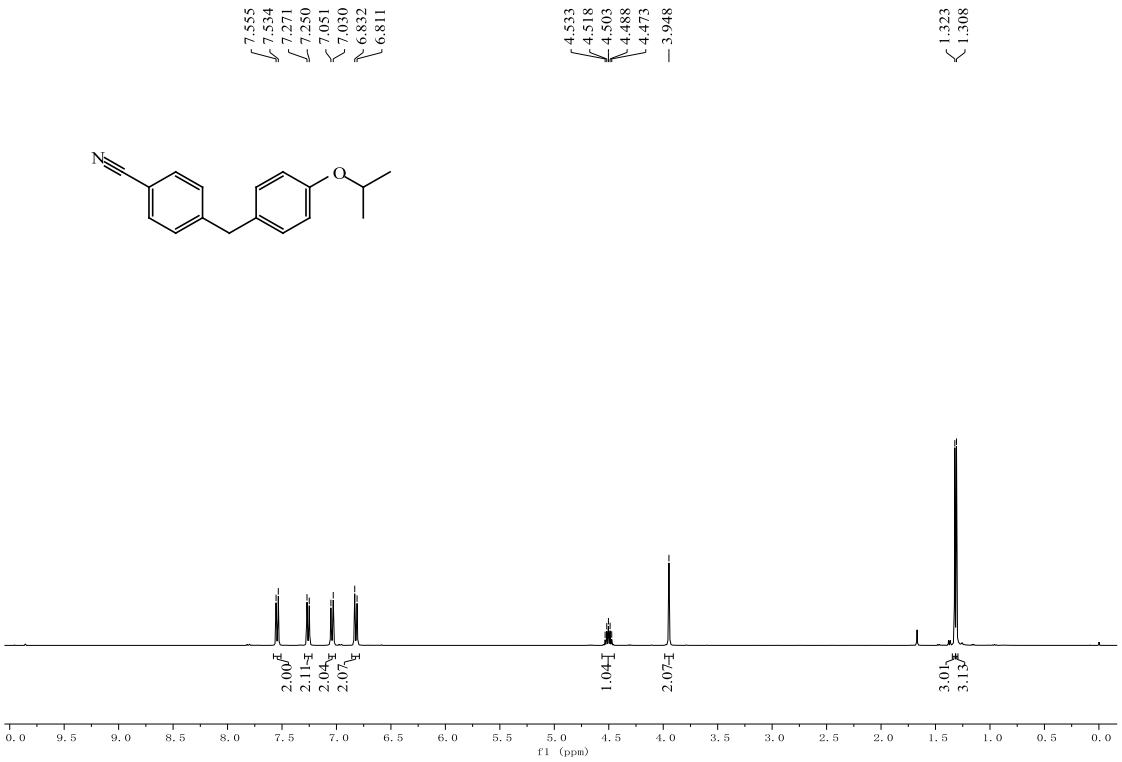


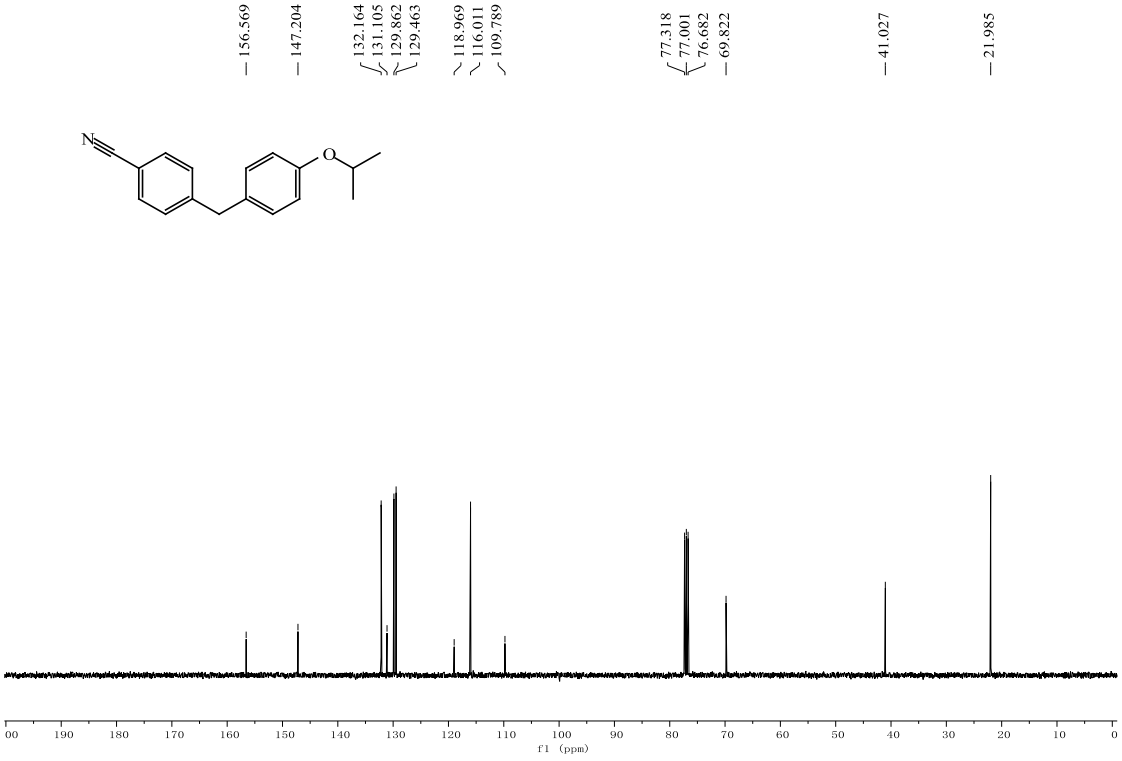
4-(4-(*tert*-Butyl)benzyl)benzonitrile (4c)



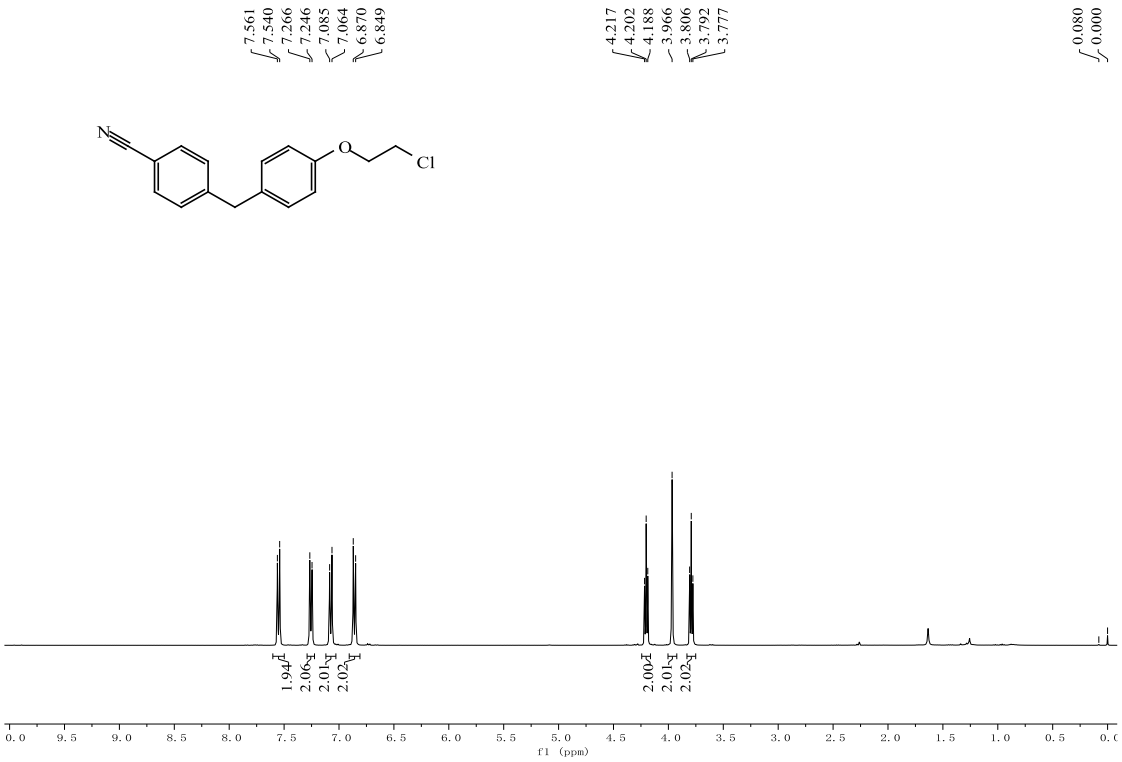


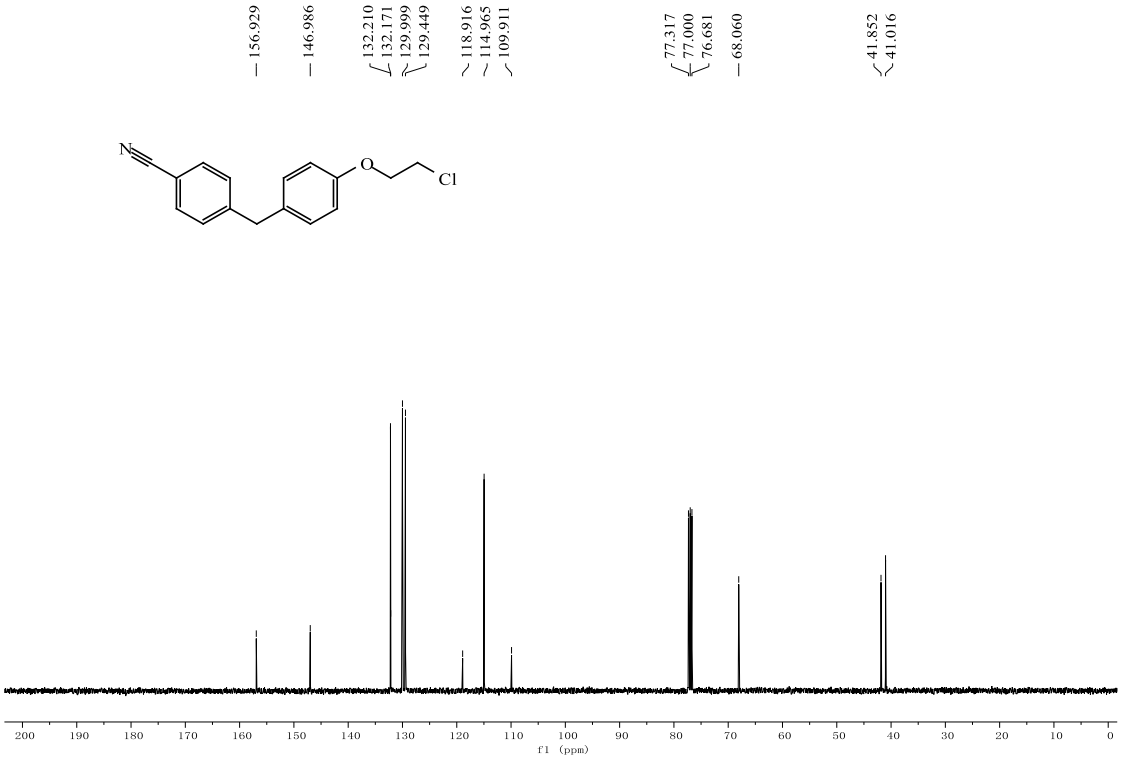
4-(4-Isopropoxybenzyl)benzonitrile (4d)



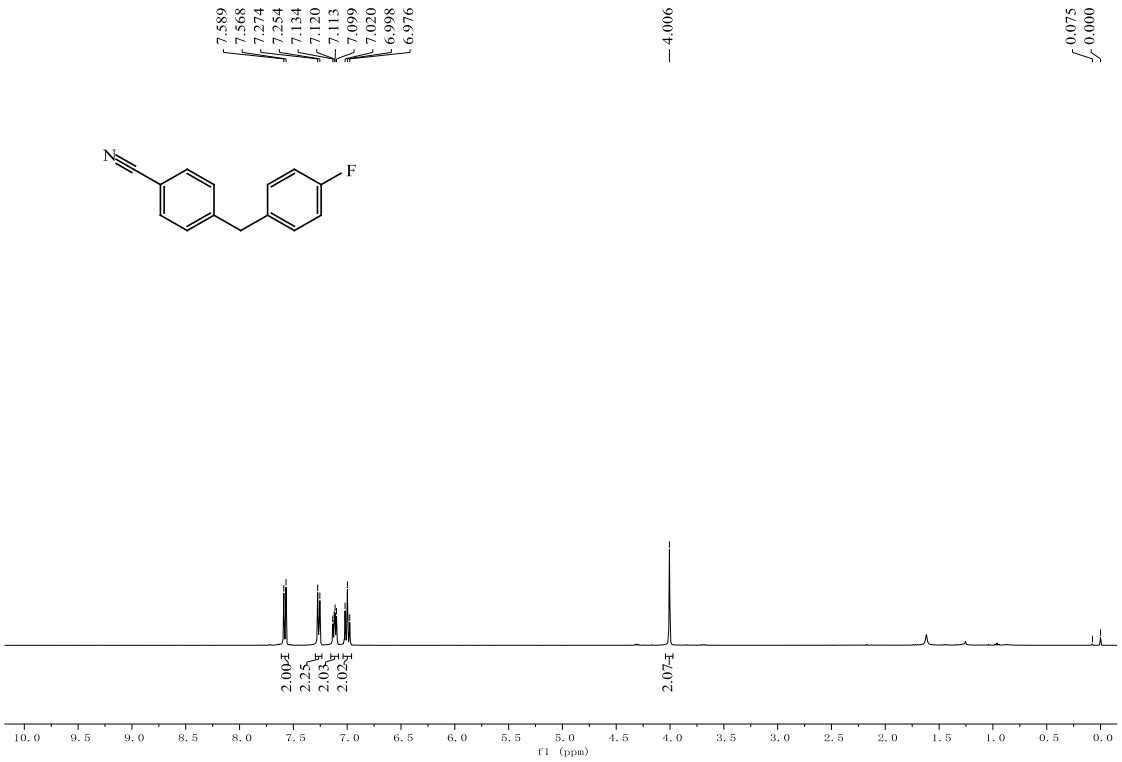


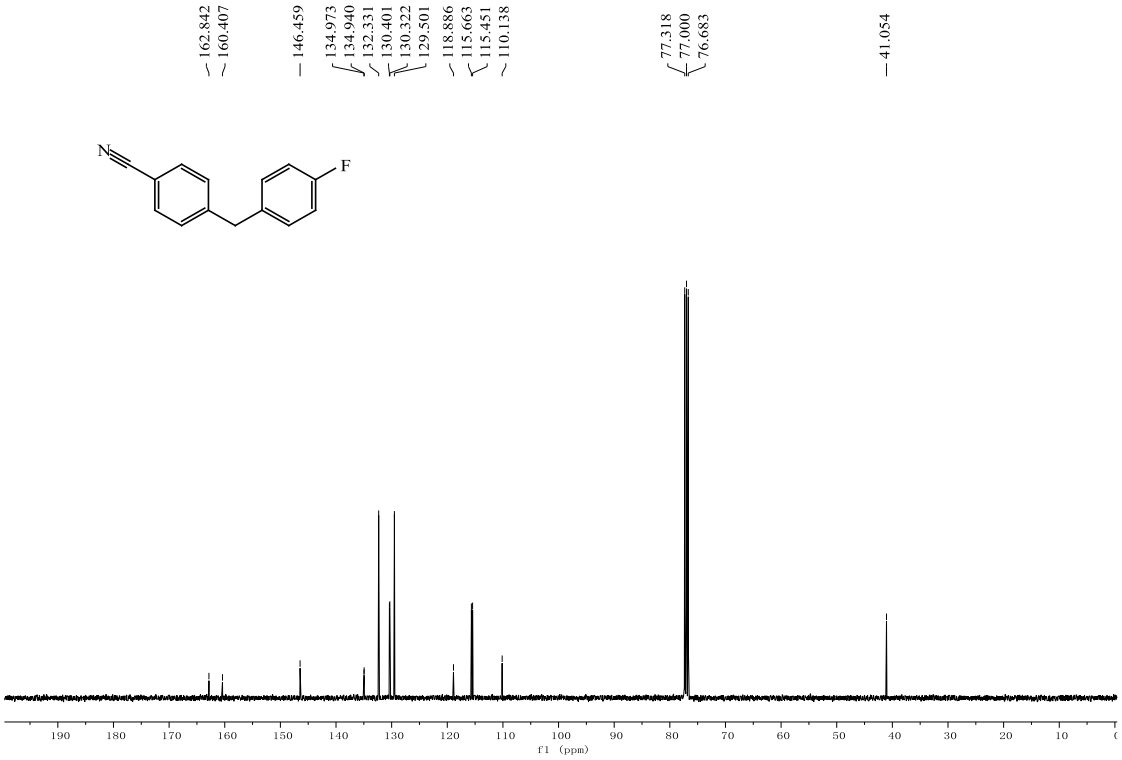
4-(4-(2-Chloroethoxy)benzyl)benzonitrile (4e)



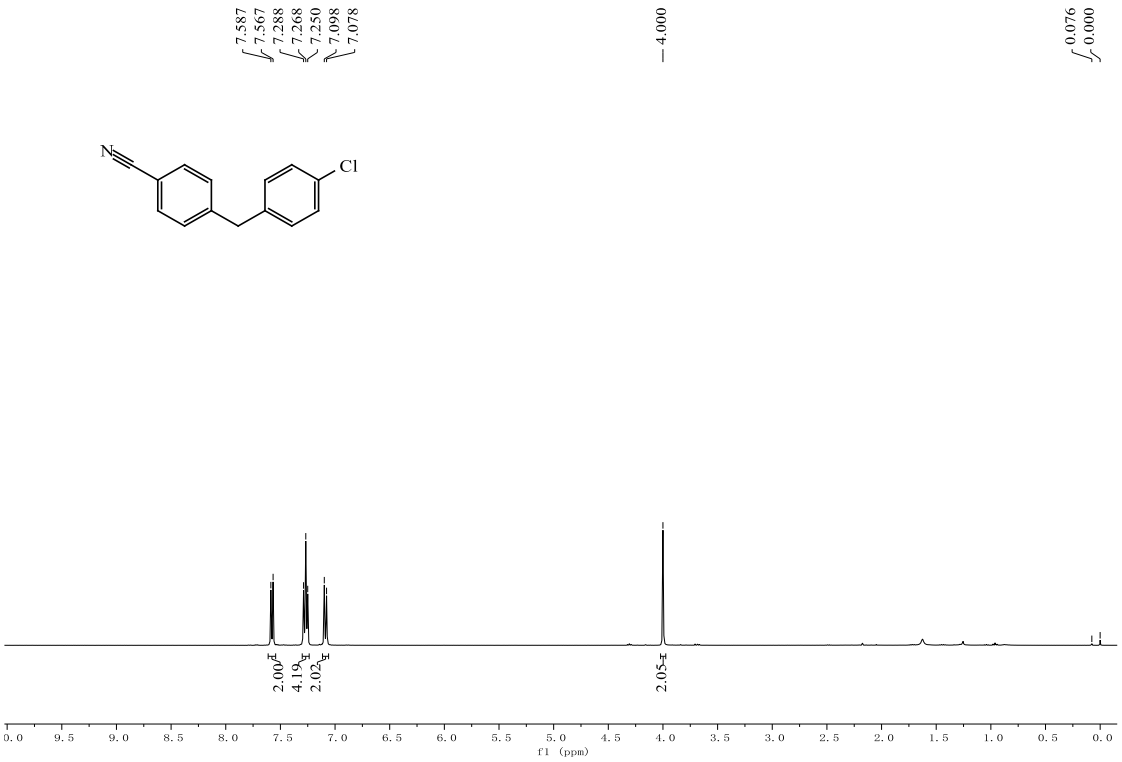


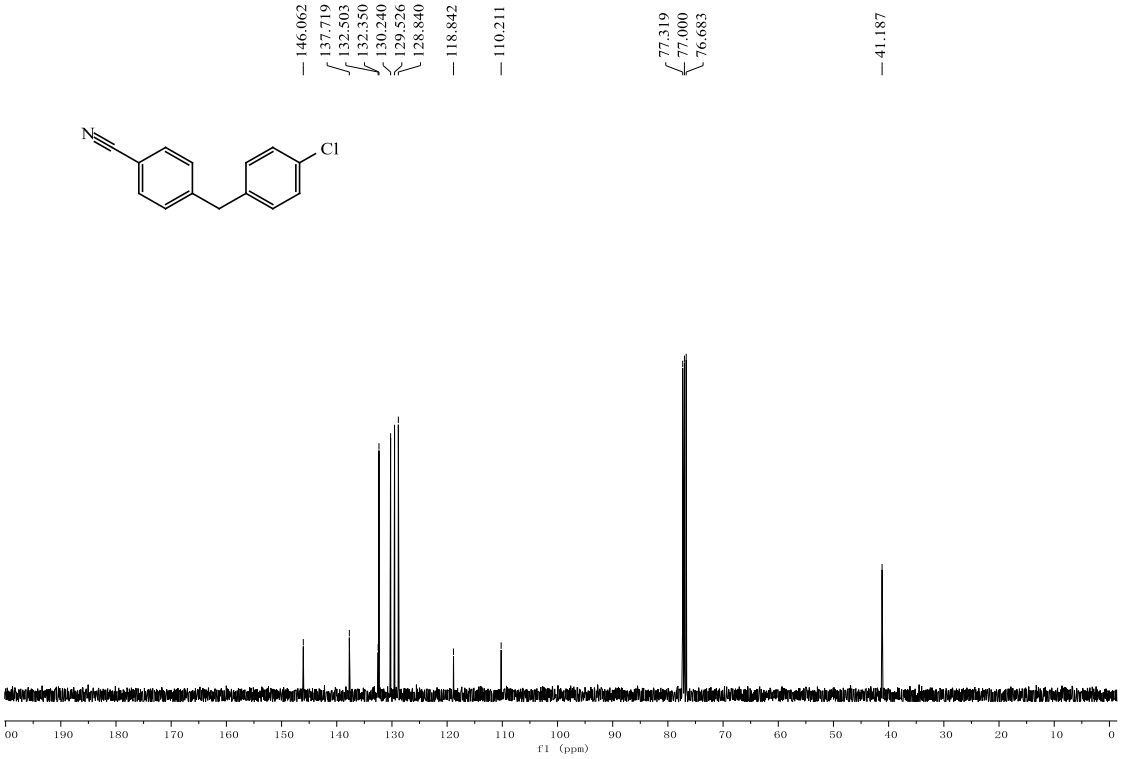
4-(4-Fluorobenzyl)benzonitrile (4f)



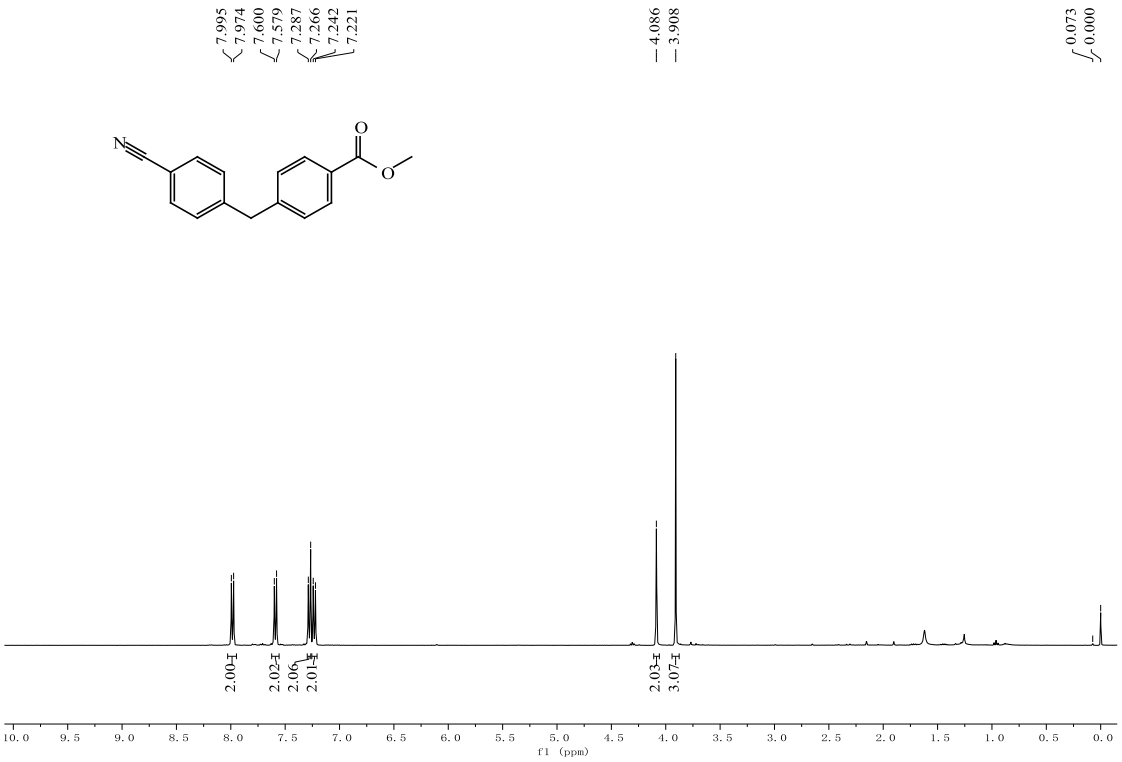


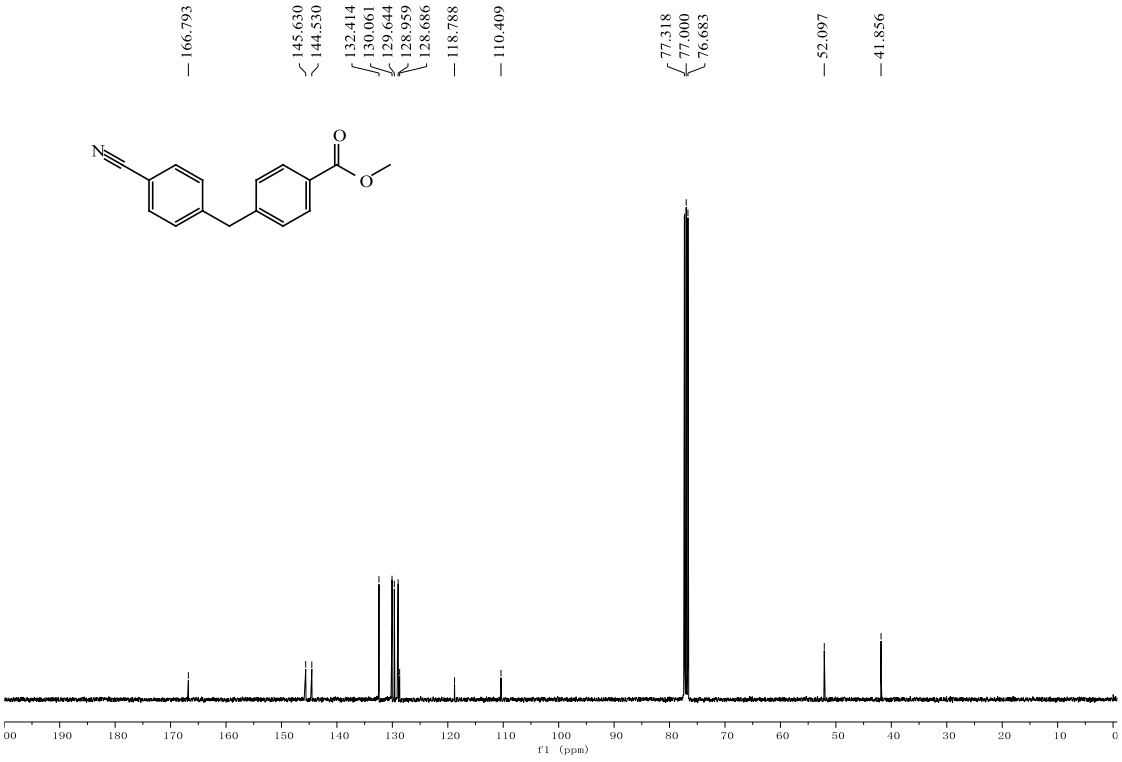
4-(4-Chlorobenzyl)benzonitrile (4g)



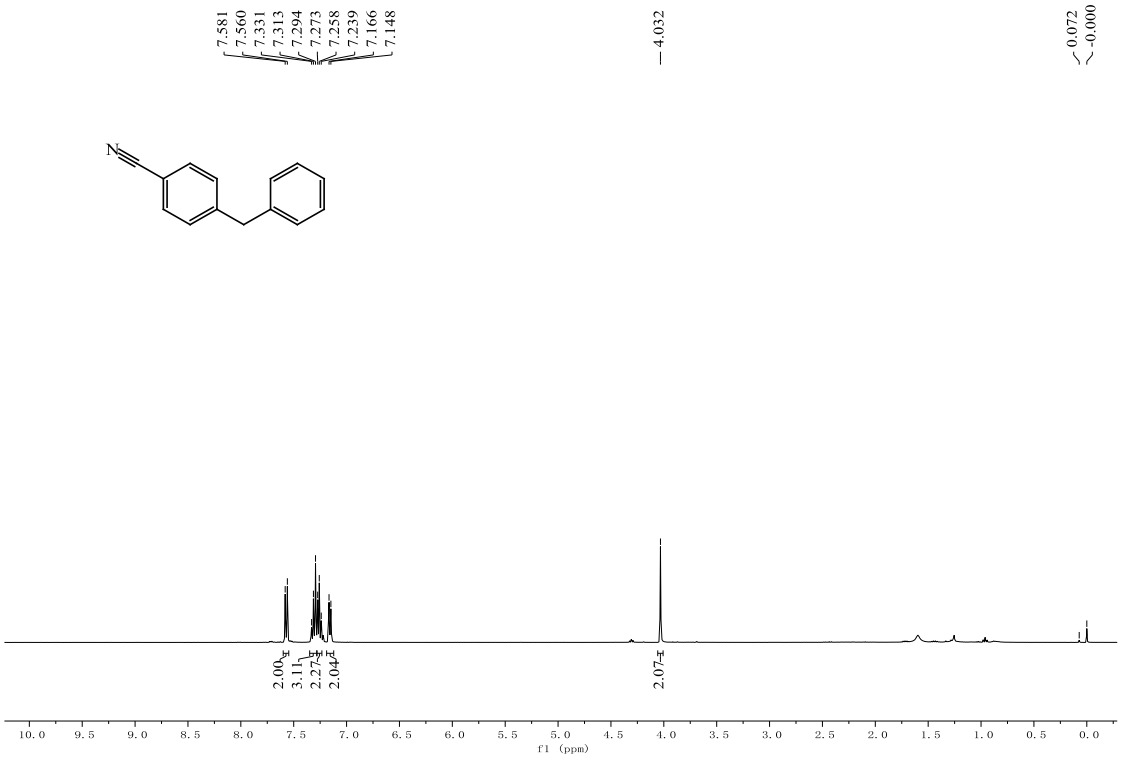


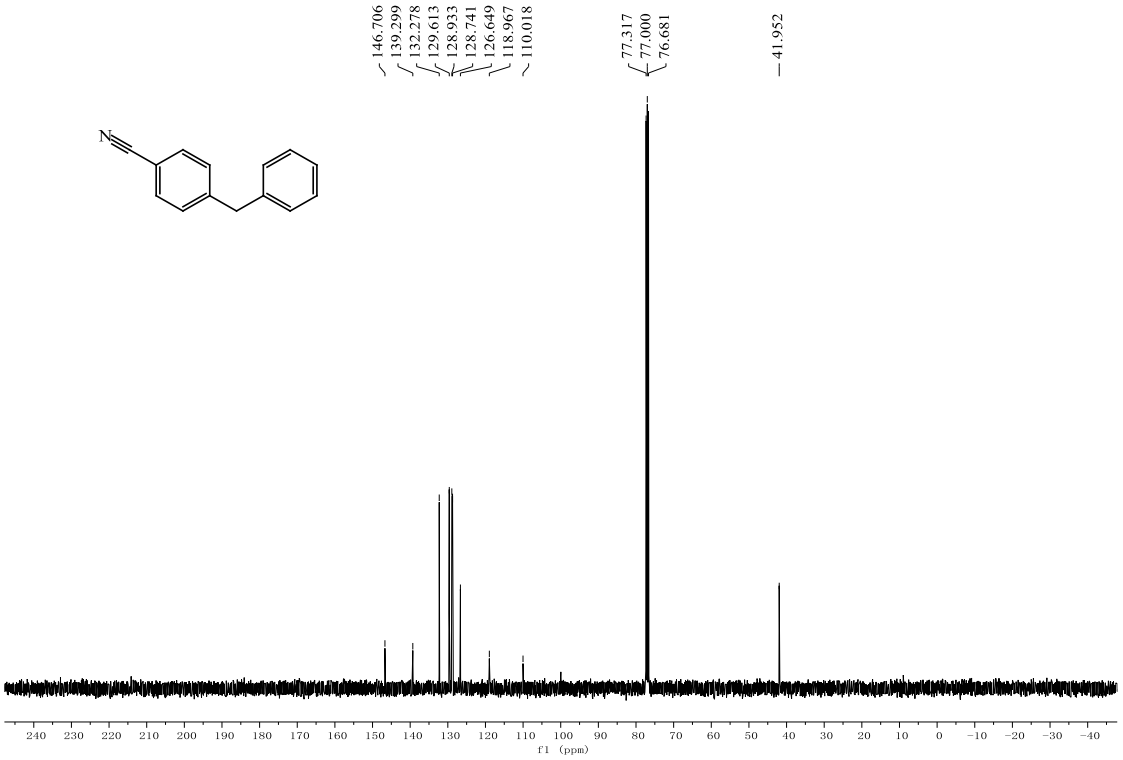
Methyl 4-(4-cyanobenzyl)benzoate (4h)



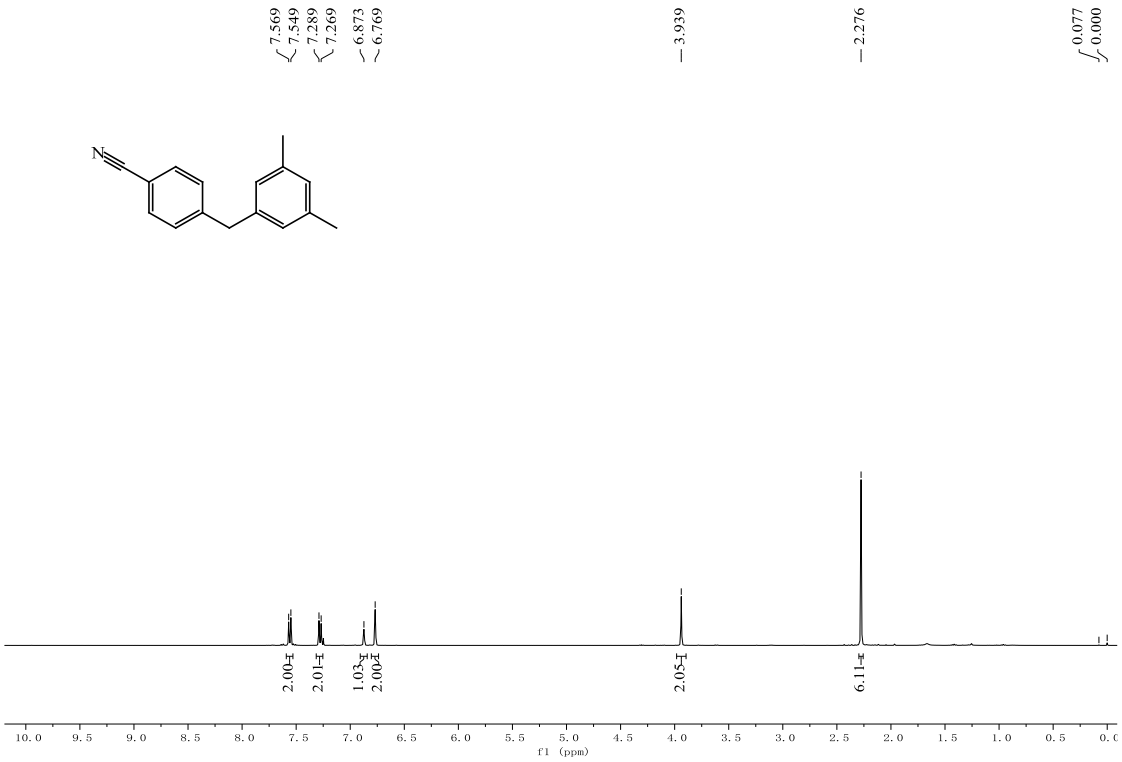


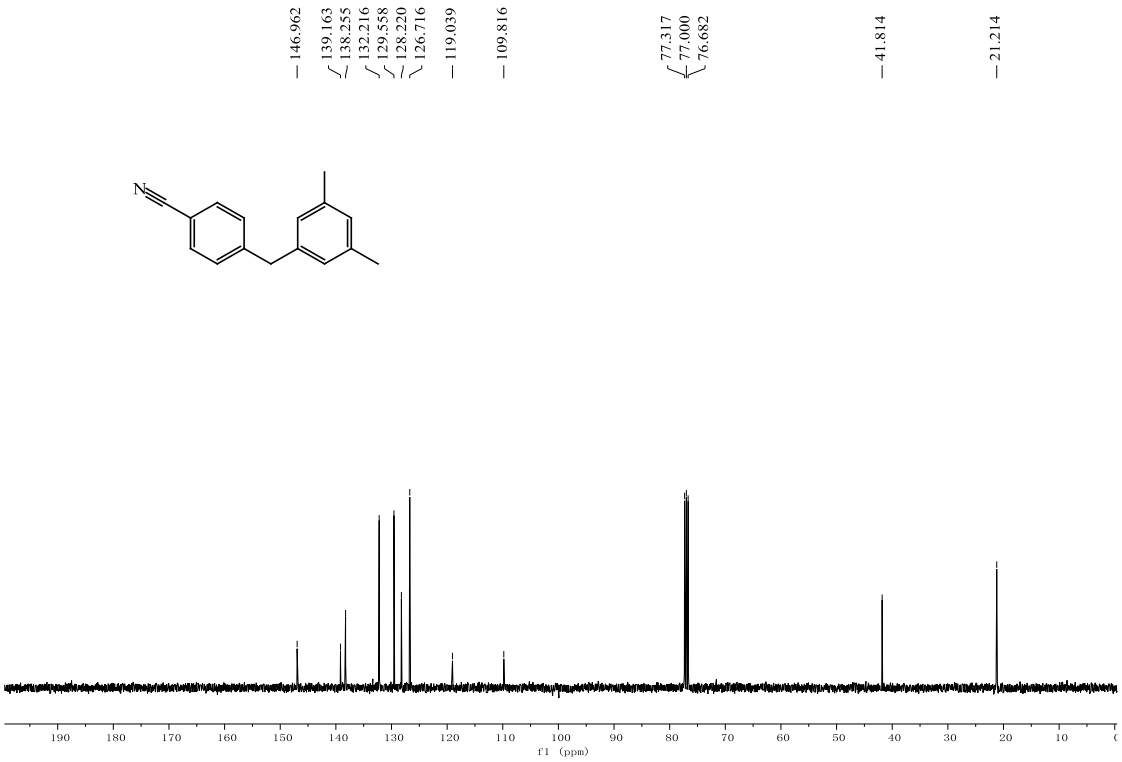
4-Benzylbenzonitrile (4i)



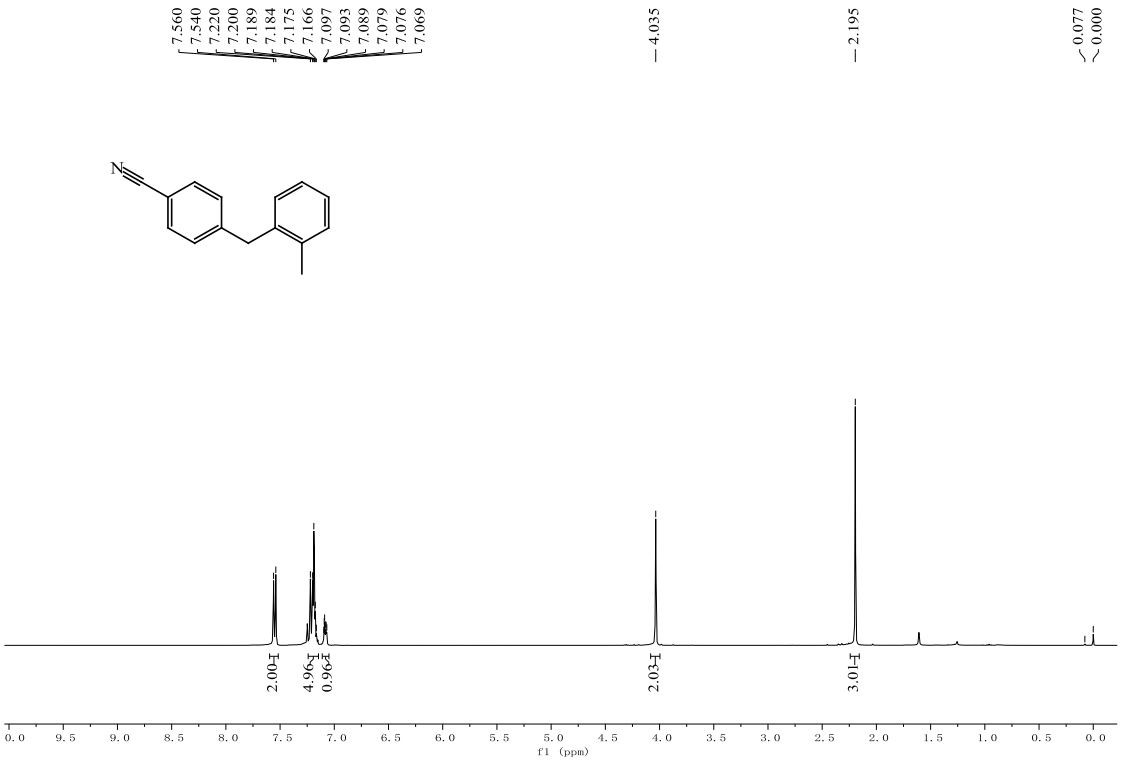


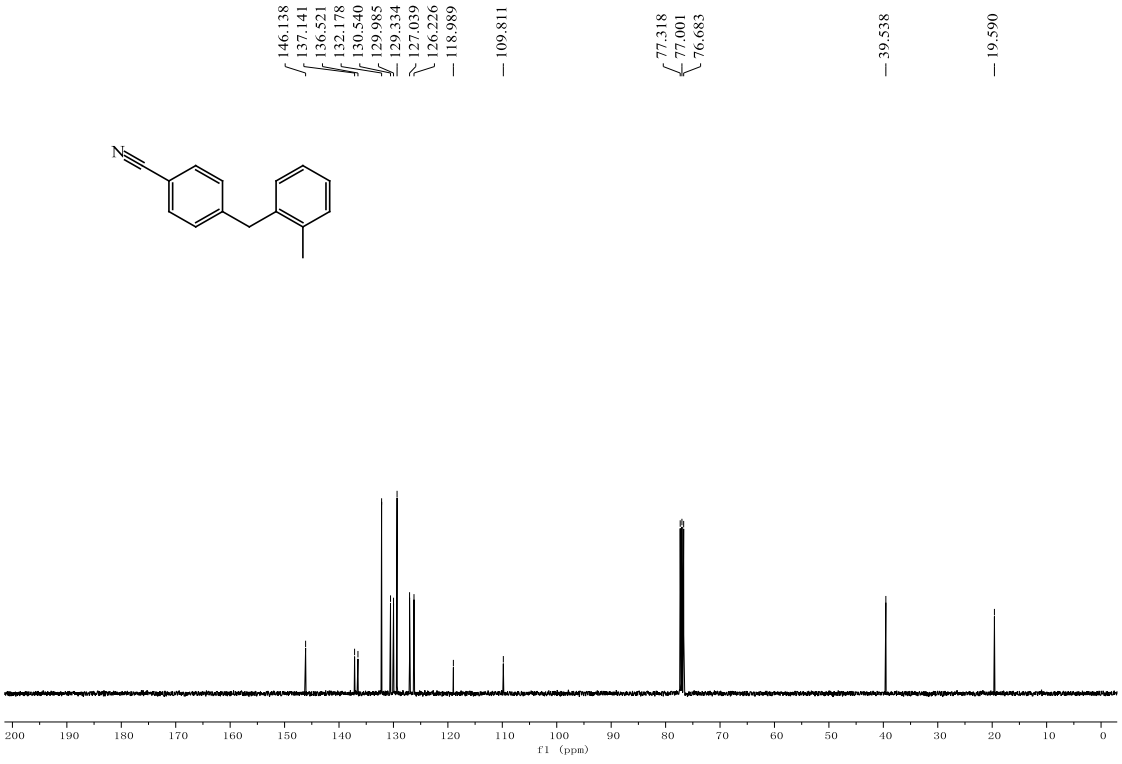
4-(3,5-Dimethylbenzyl)benzonitrile (4j)



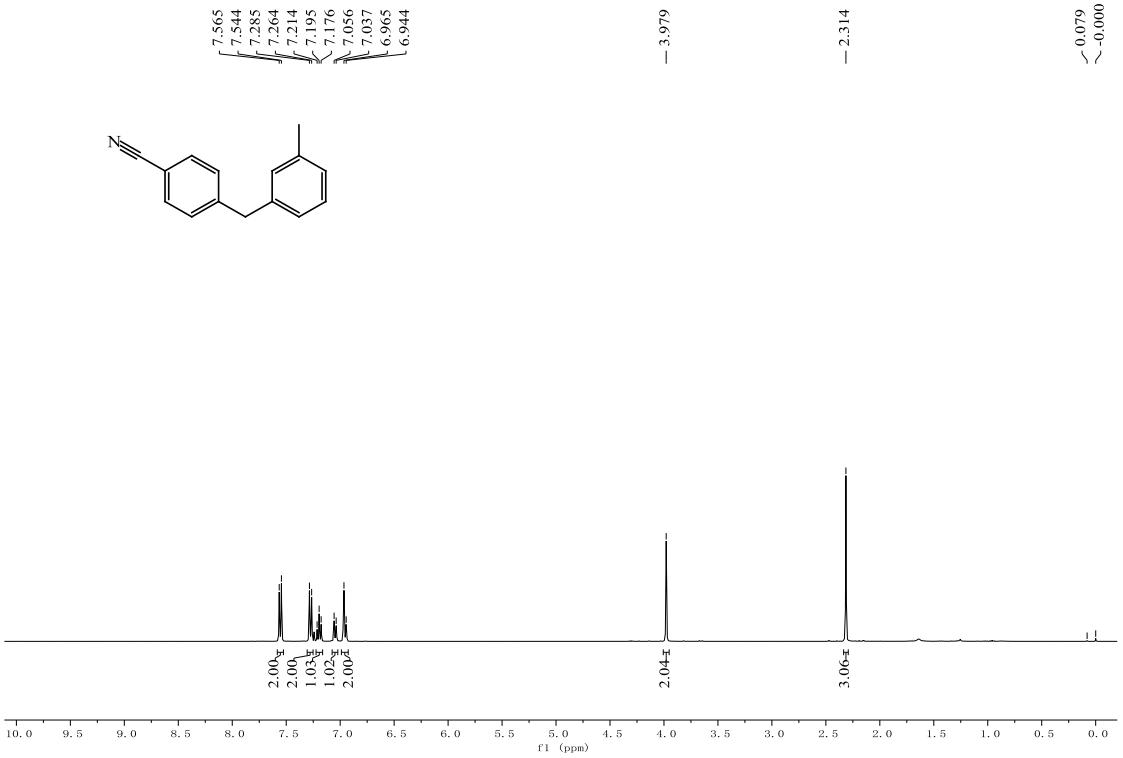


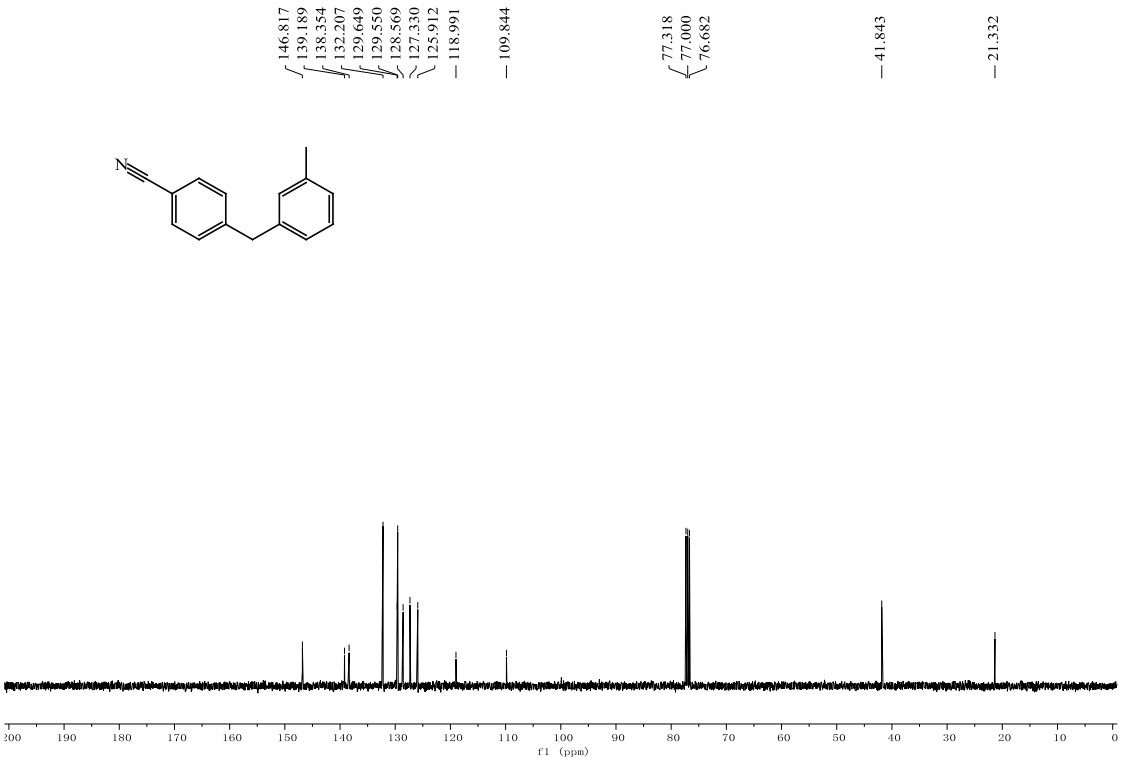
4-(2-Methylbenzyl)benzonitrile (4k)



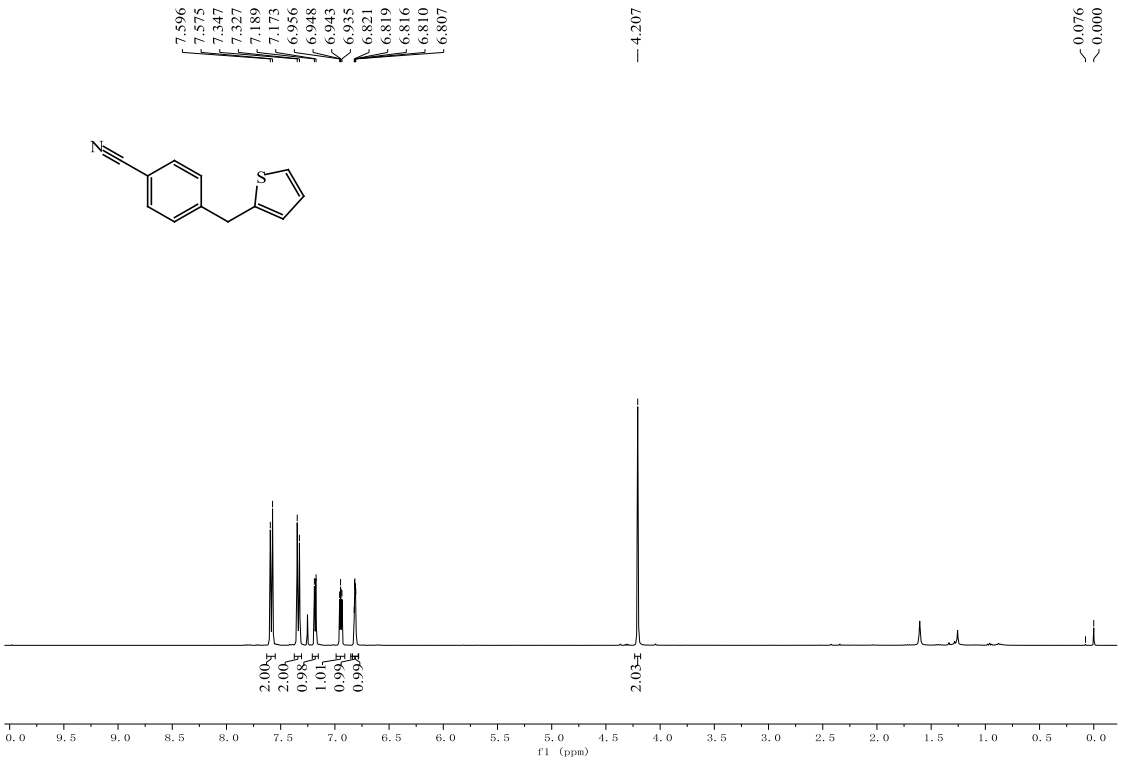


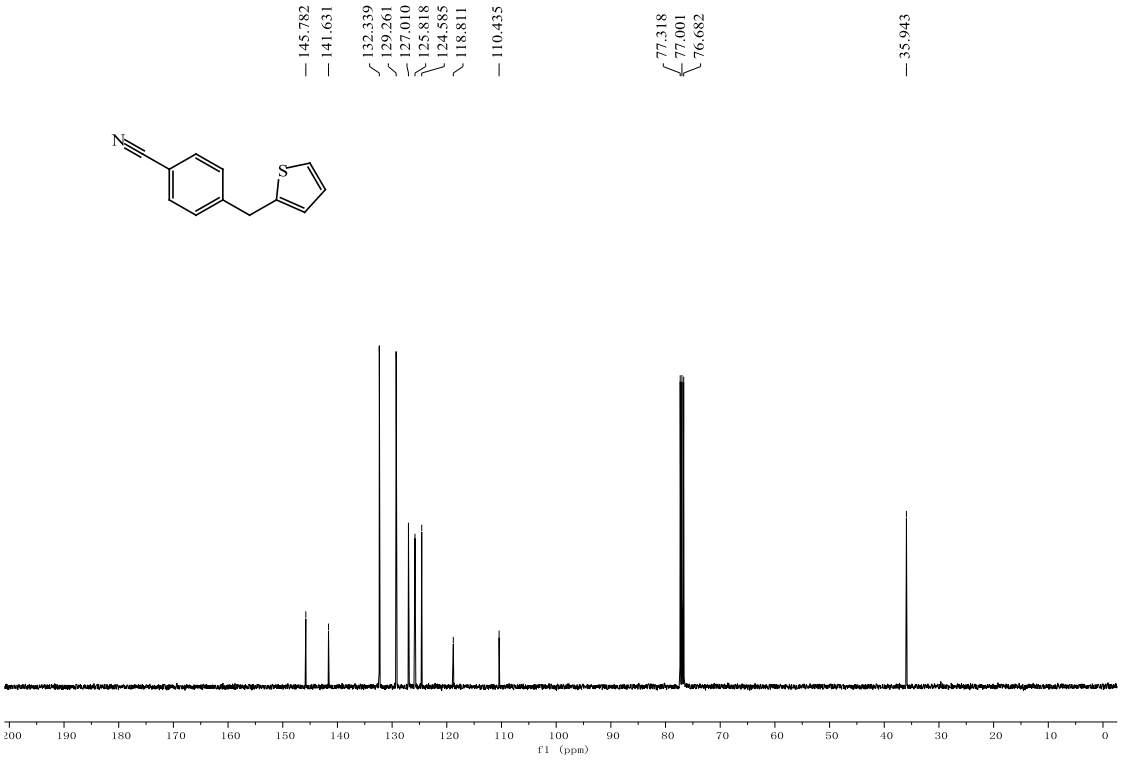
4-(3-Methylbenzyl)benzonitrile (4l)



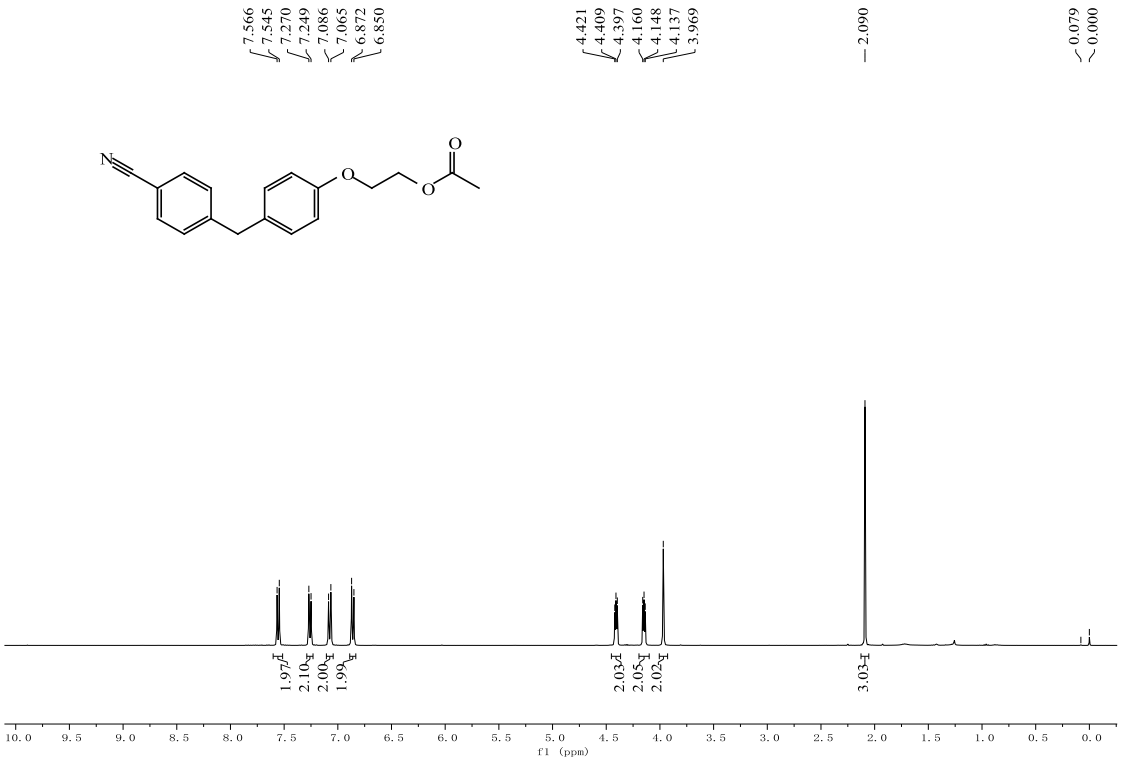


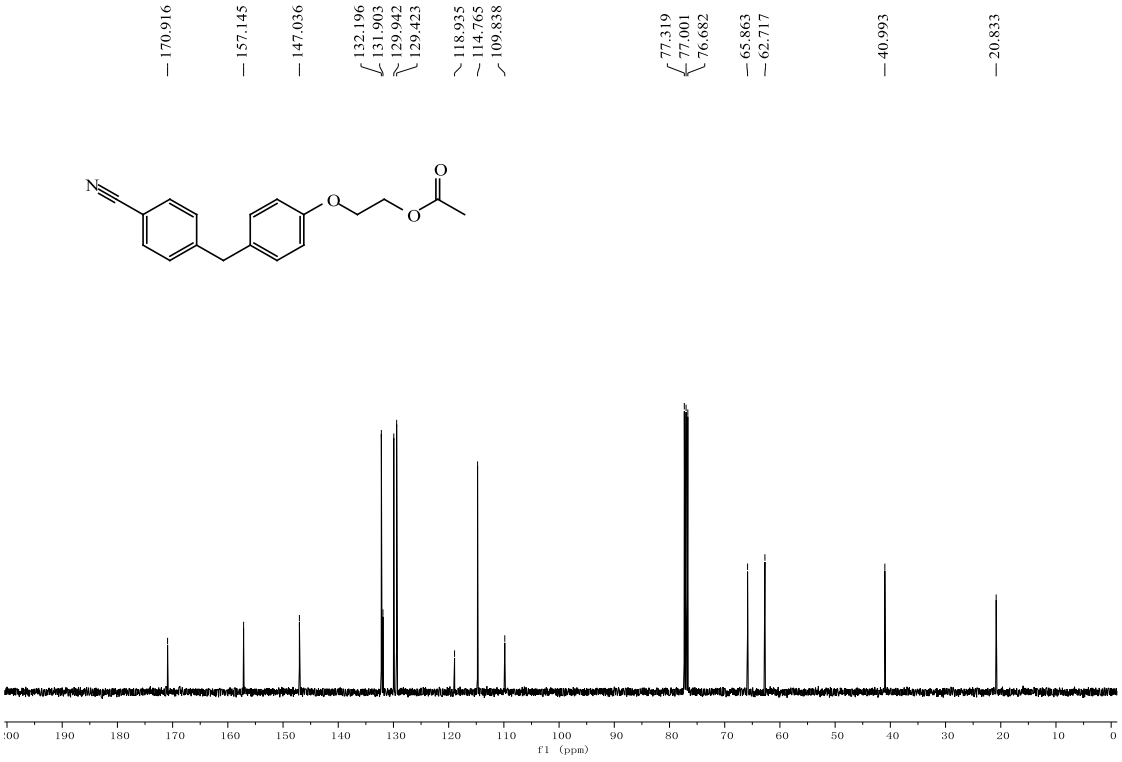
4-(Thiophen-2-ylmethyl)benzonitrile (4m)



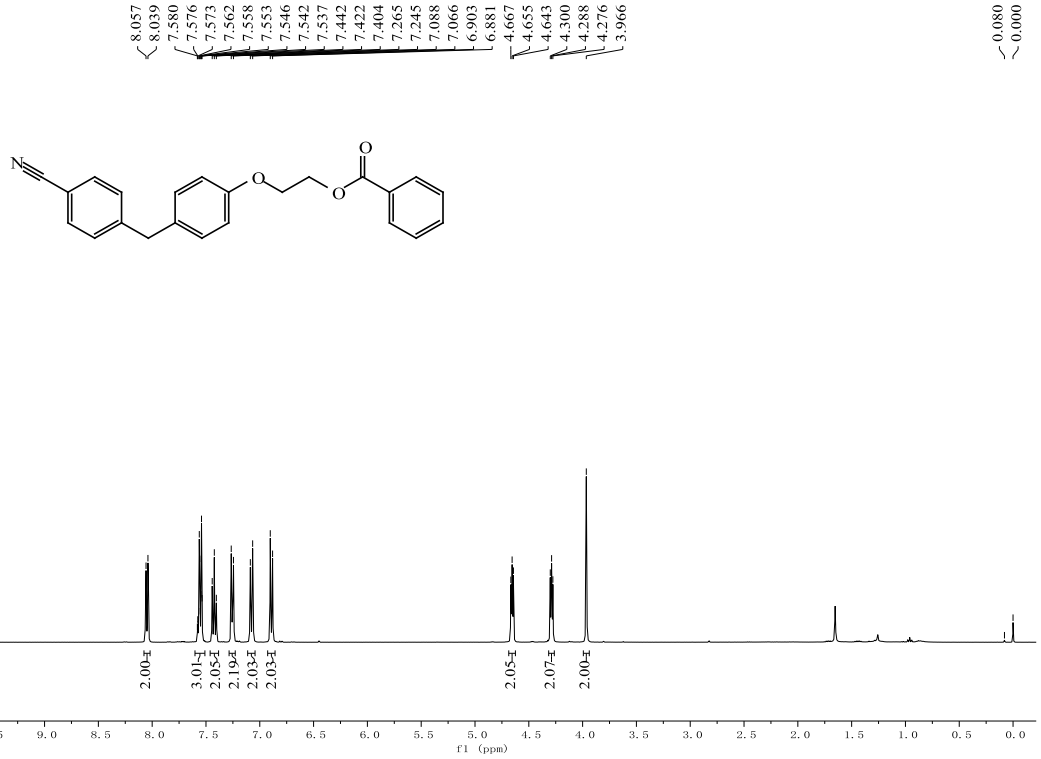


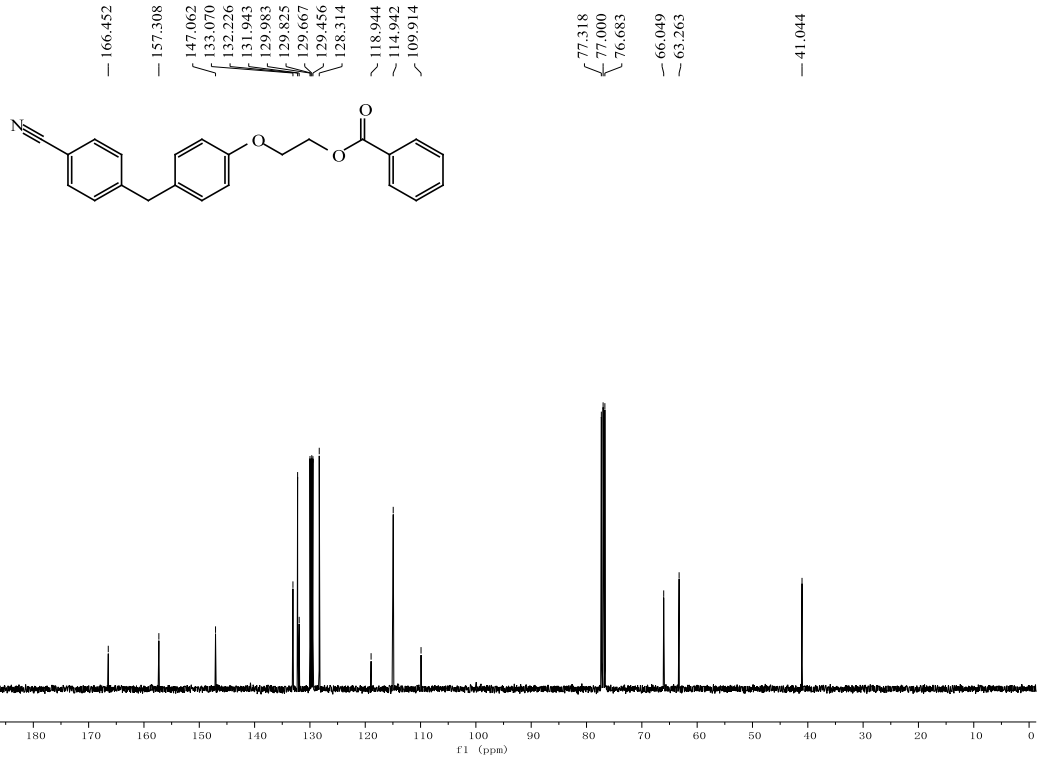
2-(4-(4-Cyanobenzyl)phenoxy)ethyl acetate (4n)



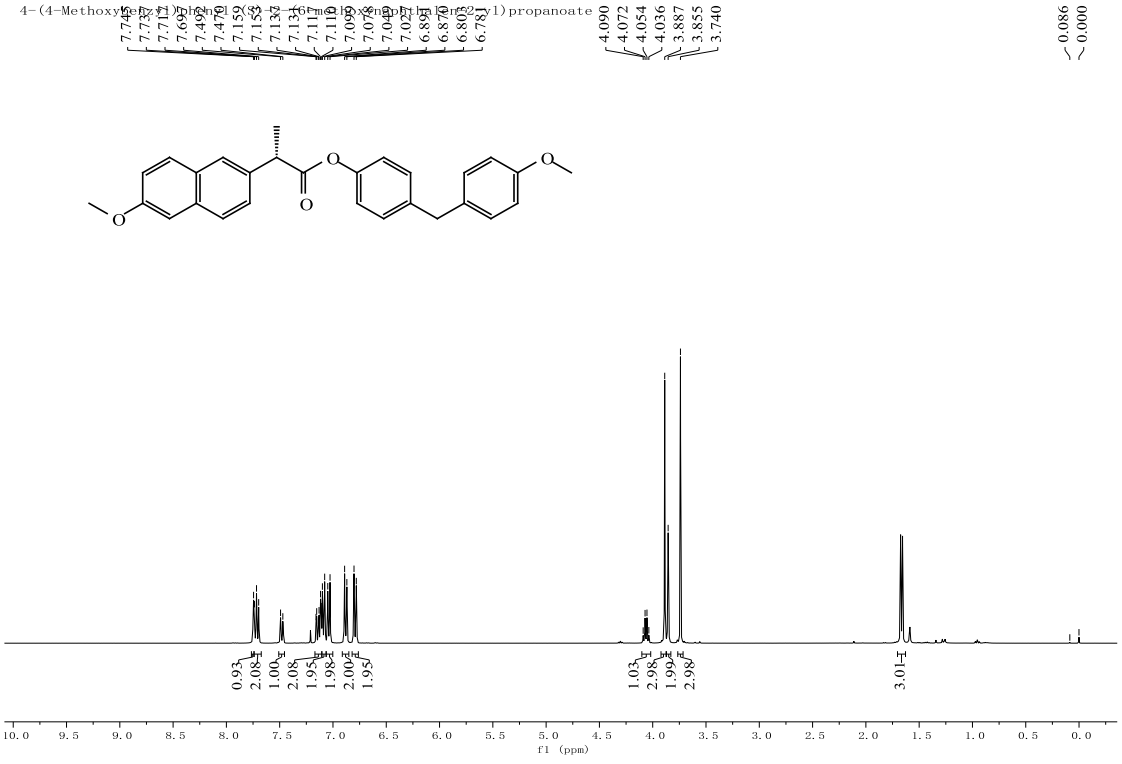


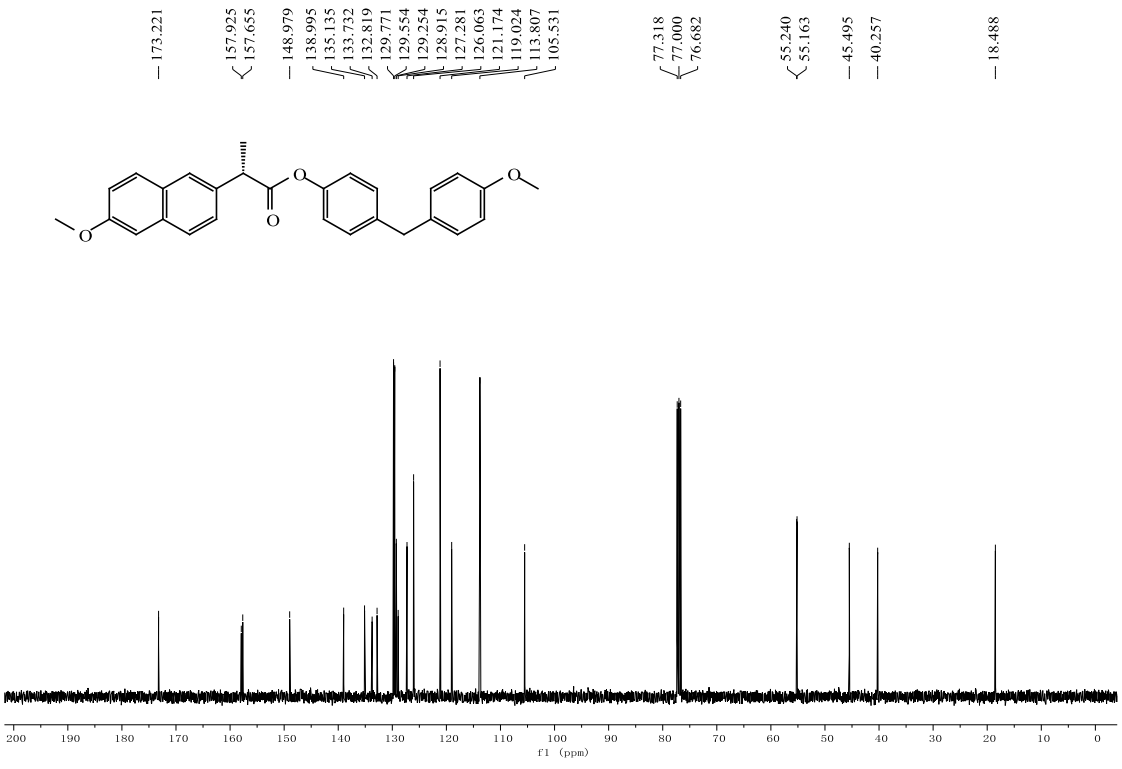
2-(4-(4-Cyanobenzyl)phenoxy)ethyl benzoate (4o)



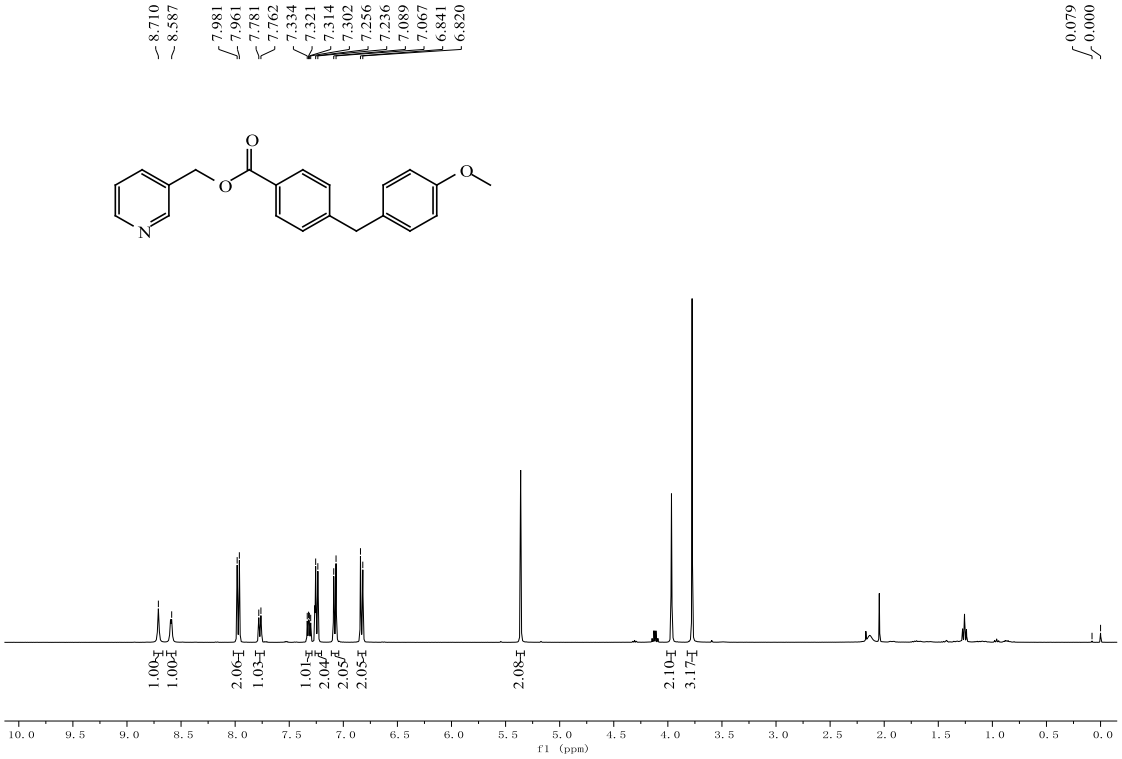


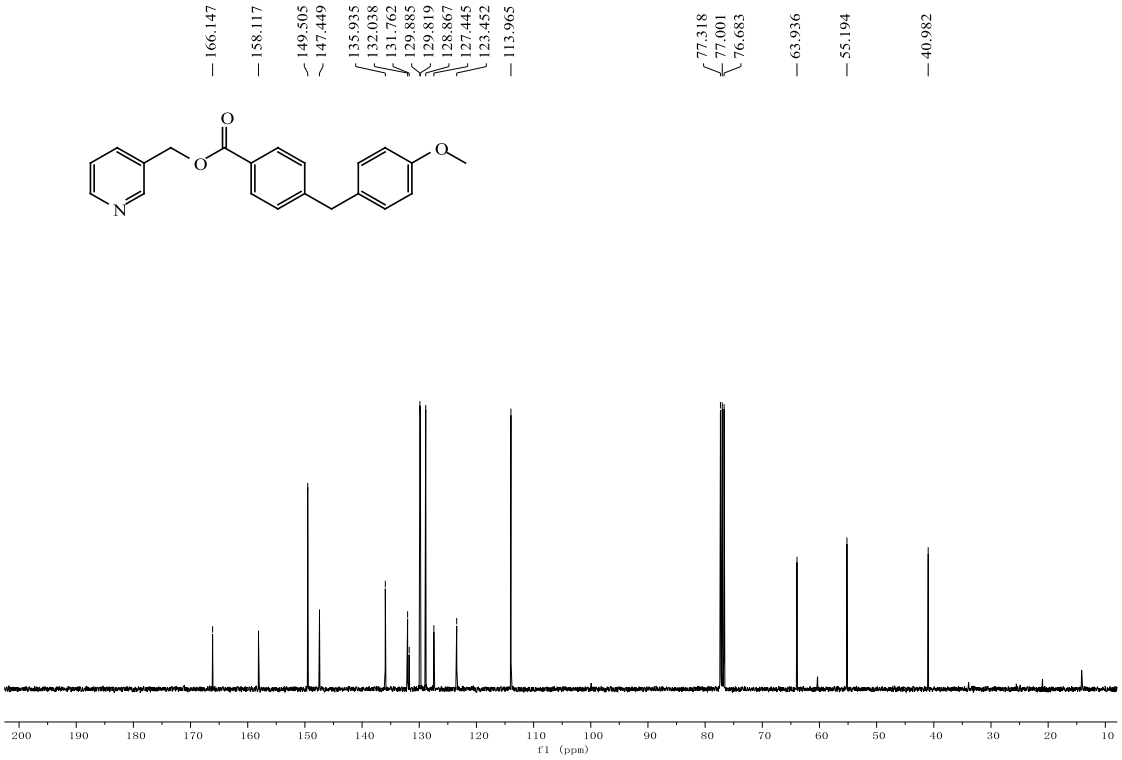
4-(4-Methoxybenzyl)phenyl (S)-2-(6-methoxynaphthalen-2-yl)propanoate (5a)



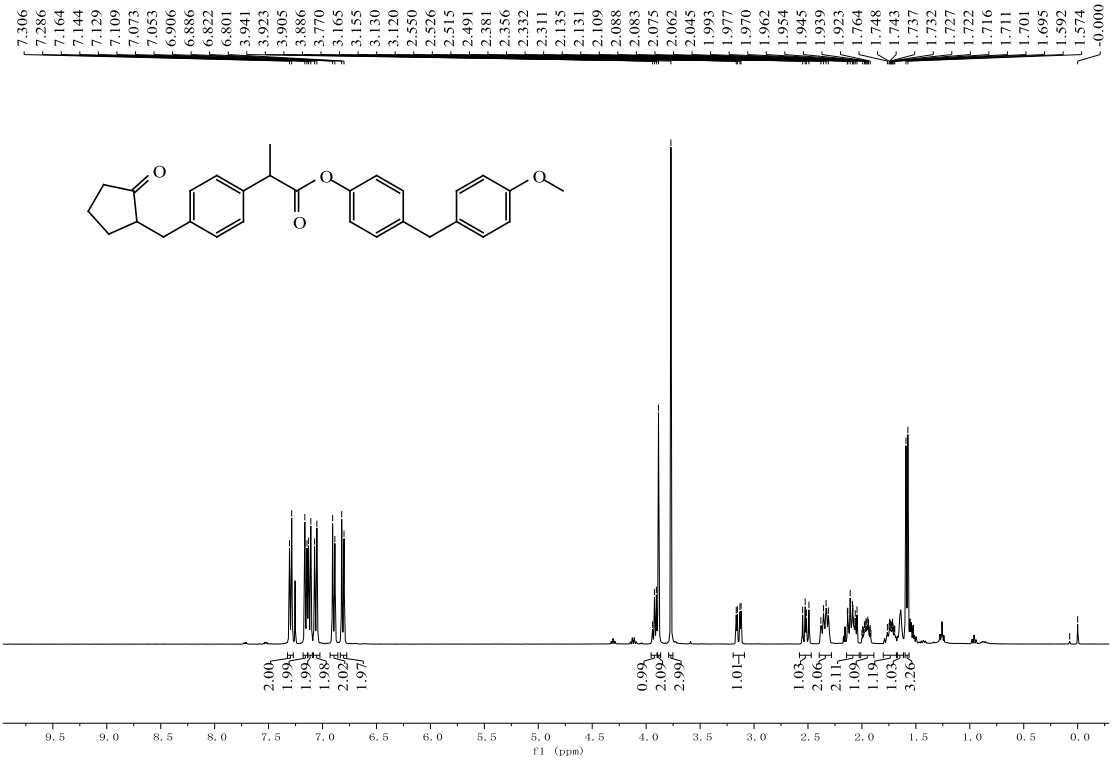


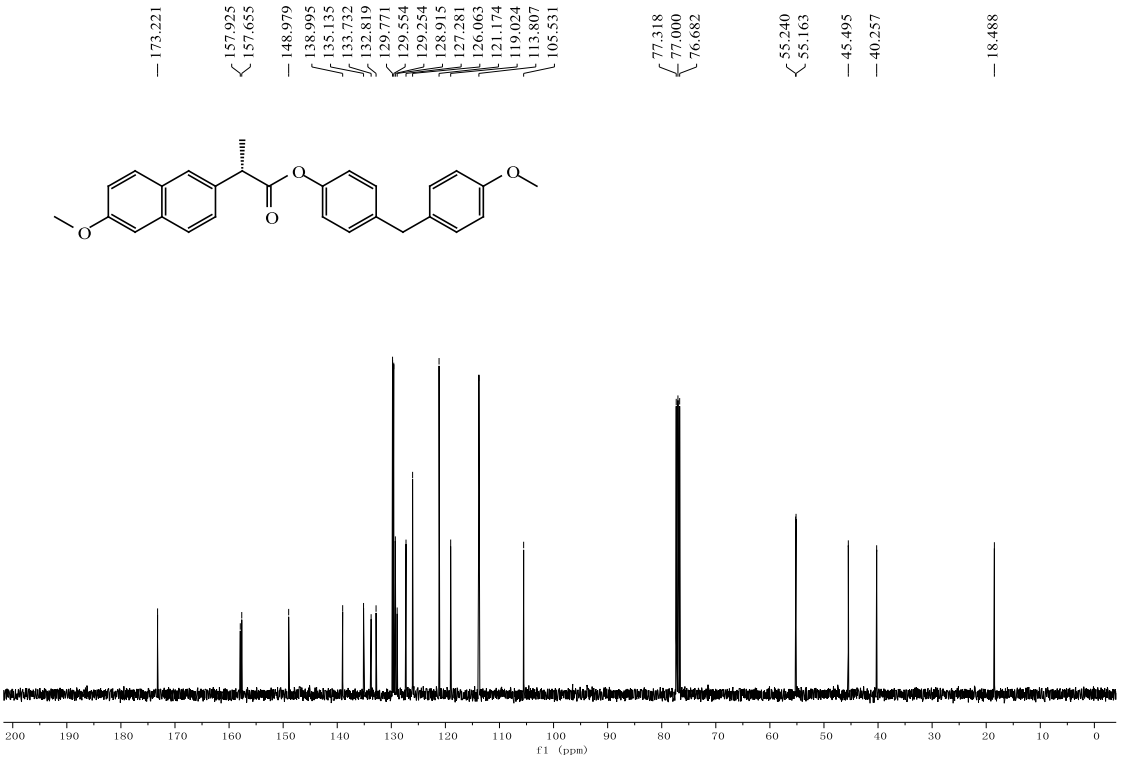
Pyridin-3-ylmethyl 4-(4-methoxybenzyl)benzoate (5b)



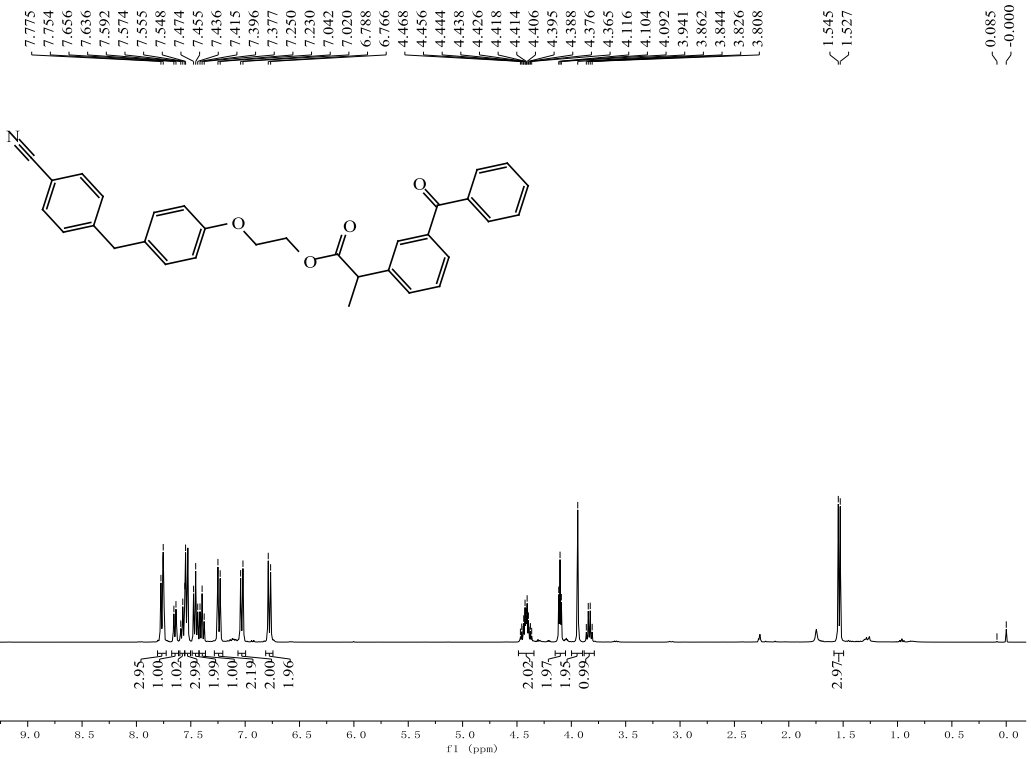


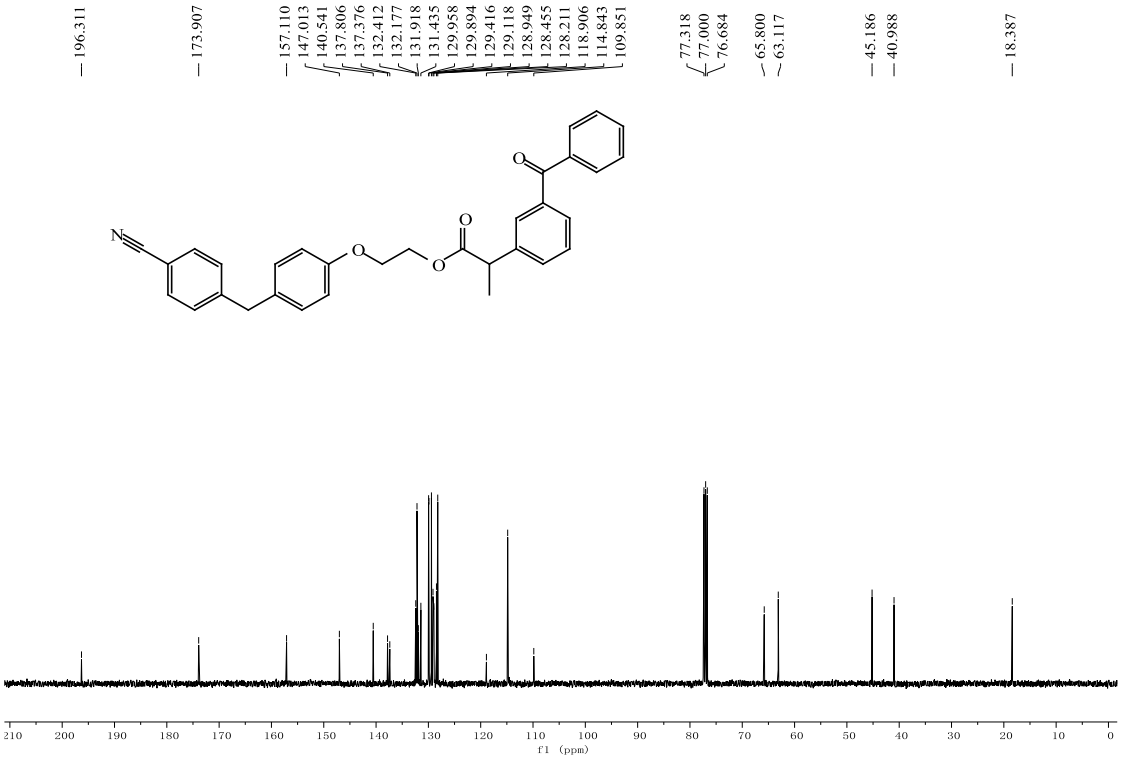
4-(4-Methoxybenzyl)phenyl 2-(4-((2-oxocyclopentyl)methyl)phenyl)propanoate (5c)



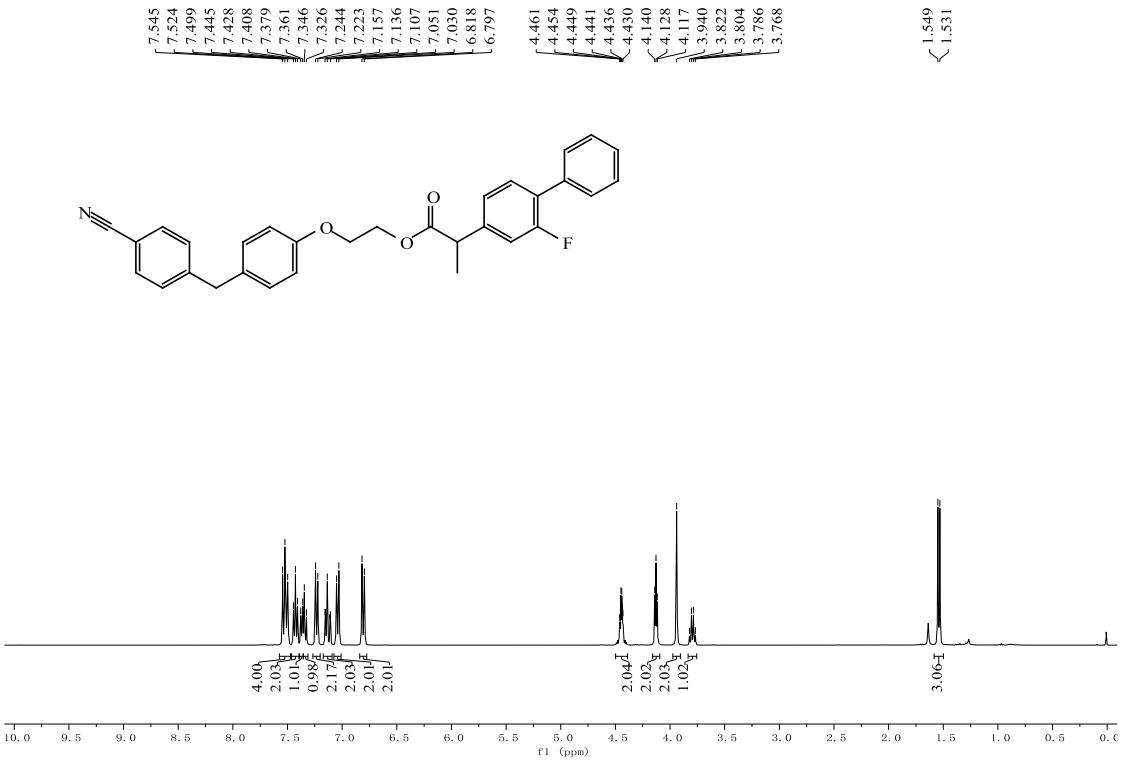


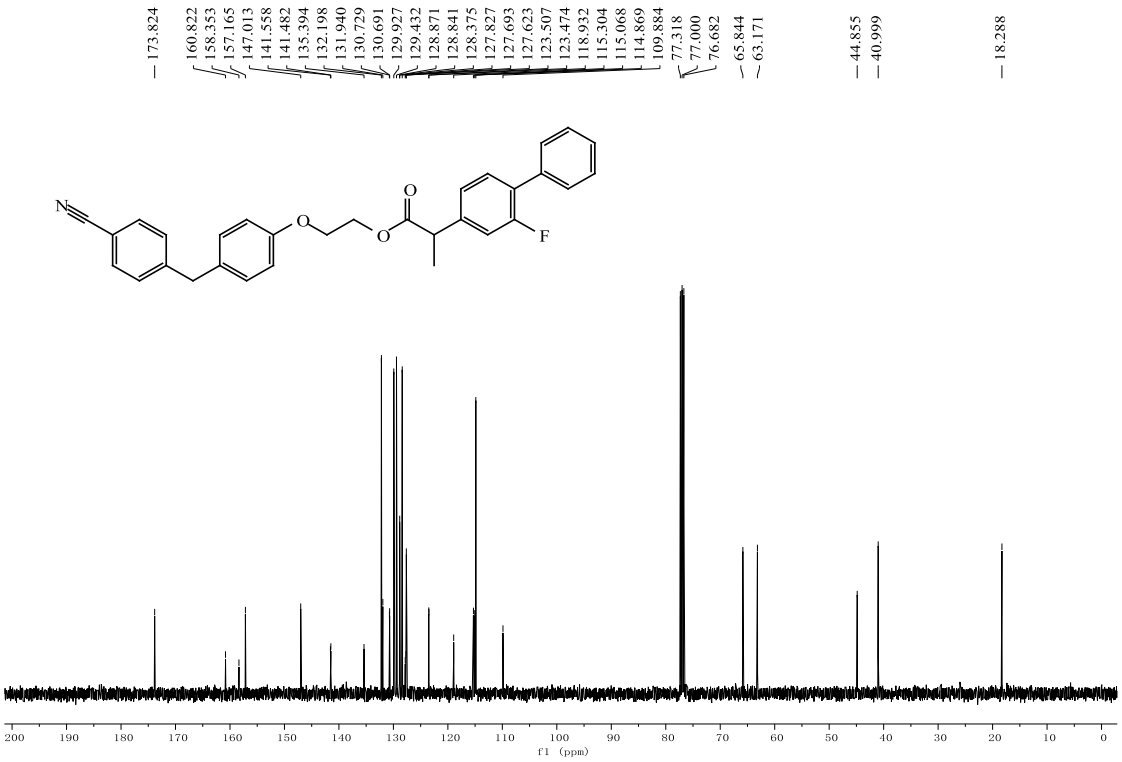
2-(4-(4-Cyanobenzyl)phenoxy)ethyl 2-(3-benzoylphenyl)propanoate (5d)





2-(4-(4-Cyanobenzyl)phenoxy)ethyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (5e)





2-(4-(4-Cyanobenzyl)phenoxy)ethyl 2-(11-oxo-6,11-dihydrodibenzo[b,e]oxepin-9-yl)acetate (5f)