

Supporting Information

Asymmetric Synthesis of Binaphthyls through Photocatalytic Cross-Coupling and Organocatalytic Kinetic Resolution

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I. General Information

Unless otherwise specified, chemicals were purchased from commercial suppliers and used without further purification. Analytical thin layer chromatography (TLC) was performed on Jiangyou TLC silica gel plates HSGF254 and visualized through UV light (254 nm). Flash column chromatography was performed using Tsingtao Haiyang silica gel (200-300 mesh). ^1H and ^{13}C NMR spectra were recorded on Bruker AVANCE III HD 400 MHz spectrometer. Chemical shifts are expressed in parts per million (δ) referenced to TMS (0.0 ppm), CDCl_3 (7.26 ppm or 77.16 ppm), Acetone- d_6 (2.05 ppm or 29.84 ppm) and DMSO- d_6 (2.50 ppm or 39.52 ppm), respectively. The NMR data are recorded as follows: chemical shift (δ , ppm), multiplicity (s = singlet; d = doublet; t = triplet; q = quartet; dd = doublet of doublet; m = multiplet; br = broad), coupling constant (Hz), integration. For reaction optimization, triphenylmethane was added as an internal standard (s, 5.55 ppm, 1H) and CDCl_3 was used as locking solvent. Photochemical reactions were carried out with 24 W blue LED which was purchased from Guangzhou Hongye Lighting (Taobao store, website: <https://shop111029161.taobao.com/?spm=a230r.7195193.1997079397.2.438a6ac2NnYsKB>). High resolution mass spectroscopy (HRMS) analyses were performed at a Q-Exactive (Thermo Scientific) Inc. mass instrument (HESI). The emission spectrum of LED light source was measured using Horiba iHR 320 (Horiba Scientific). Cyclic voltammetry experiments were performed on a CH Instruments Electrochemical Workstation model CHI602E. Fluorescence quenching experiments were recorded on Horiba Scientific FluoroMax-4 Spectrofluorometer. The enantiomeric excess values were determined by chiral HPLC with Waters Alliance e2695 and a Daicel CHIRALCEL and CHIRALPAK column with the detector wavelength of 254 nm.

II. Optimization for Photocatalytic Cross-Coupling

1. Optimization for Synthesis of BINOL Derivatives 3a

Structures of selected photocatalysts:

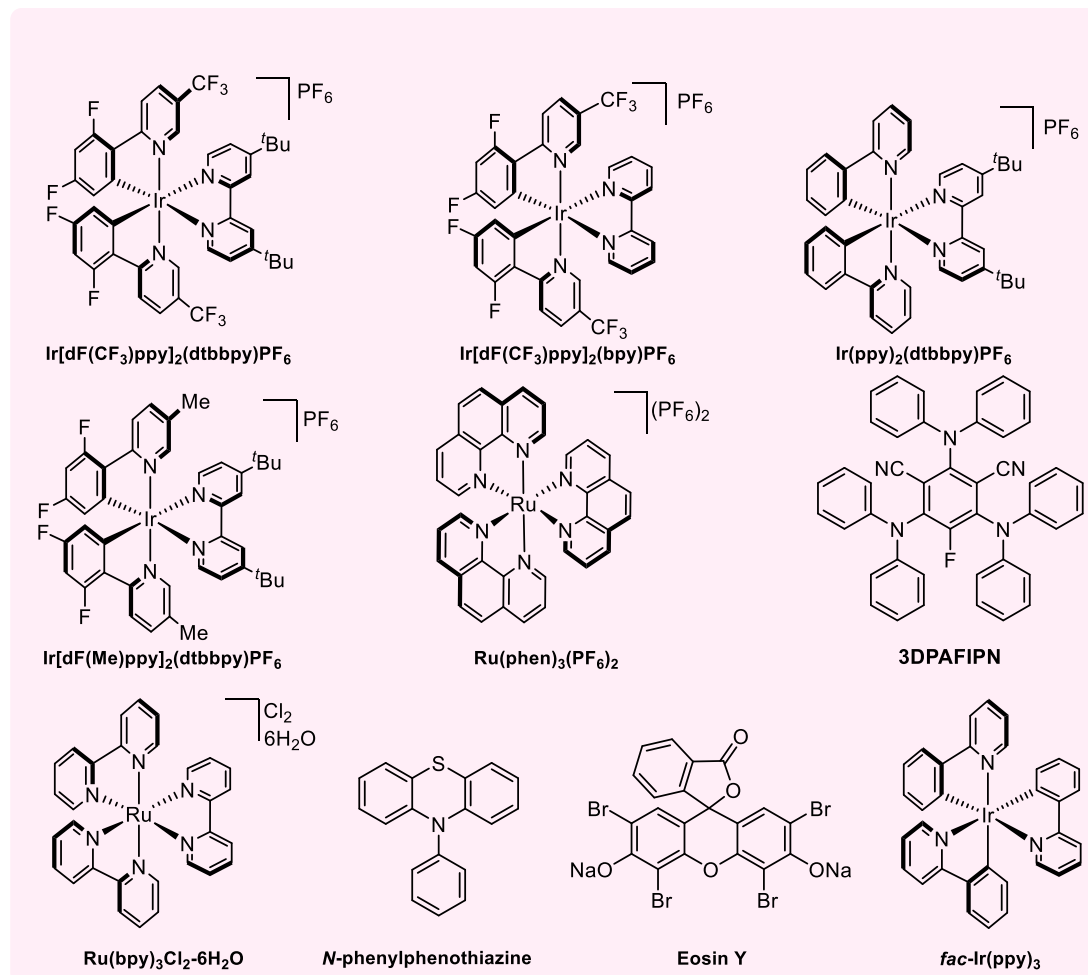
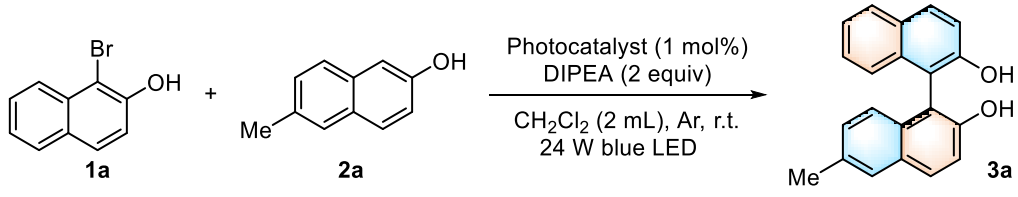
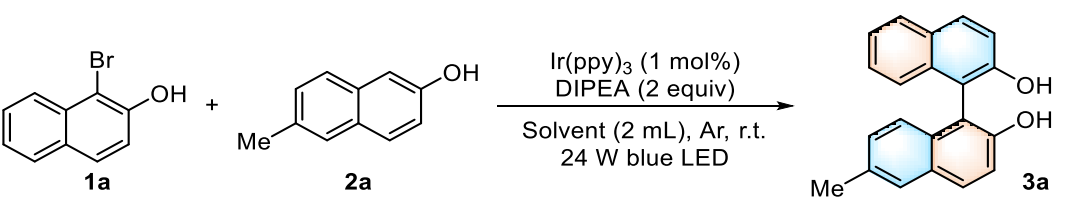


Table S1. Photocatalyst Screening

		
Entry	PC	Yield (%) ^[a]
1	$\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{dtbbpy})\text{PF}_6$	72 ^[b]
2	$\text{Ir}(\text{ppy})_2(\text{dtbbpy})\text{PF}_6$	64 ^[b]
3	$\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{bpy})\text{PF}_6$	64 ^[b]
4	$\text{Ir}[\text{dF}(\text{Me})\text{ppy}]_2(\text{dtbbpy})\text{PF}_6$	65
5	$\text{Ir}(\text{ppy})_3$	77
6	$\text{Ru}(\text{bpy})_3\text{Cl}_2 \cdot 6\text{H}_2\text{O}$	N.R.
7	$\text{Ru}(\text{phen})_3(\text{PF}_6)_2$	N.R.
8	<i>N</i> -phenylphenothiazine	N.R.
9	3DPAFIPN	68 ^[b]
10	Eosin Y	N.R.

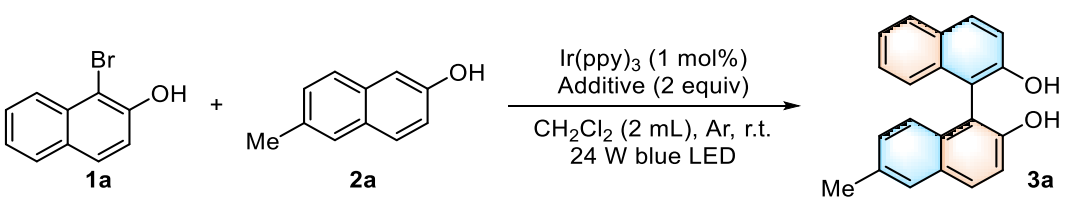
[a] 1-Bromo-2-naphthol **1a** (0.1 mmol, 1 equiv), 6-methyl-2-naphthol **2a** (0.2 mmol, 2 equiv), DIPEA (0.2 mmol, 2 equiv), photocatalyst (0.01 equiv), CH_2Cl_2 (2 mL) for 2 h. Yields were determined through crude ^1H spectrum using triphenylmethane as internal standard with CDCl_3 as locking solvent (5.55 ppm, s, 1H). [b] The reaction was carried out for 12 h. 3DPAFIPN = 2,4,6-Tris(diphenylamino)-5-fluoroisophthalonitrile. N.R. = no reaction.

Table S2. Solvent Screening

		
Entry	Solvent	Yield (%) ^[a]
1	CH ₂ Cl ₂	77
2	CHCl ₃	60 ^[b]
3	DCE	60
4	PhMe	54 ^[b]
5	PhF	75 ^[b]
6	PhCF ₃	76 ^[b]
7	MeCN	trace
8	1,4-dioxane	trace
9	MTBE	trace
10	Tetrachloroethane	52
11	DMSO	trace
12	EtOAc	trace
13	EtOH	N.D.

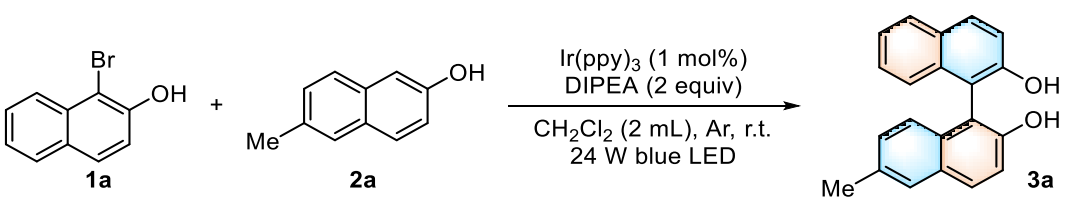
[a] **1a** (0.1 mmol, 1 equiv), **2a** (0.2 mmol, 2 equiv), DIPEA (0.2 mmol, 2 equiv), Ir(ppy)₃ (0.01 equiv), solvent (2 mL) for 2 h. Yields were determined through crude ¹H spectrum using triphenylmethane as internal standard. [b] The reaction was carried out for 12 h. MTBE = Methyl *tert*-butyl ether. EtOAc = Ethyl acetate. N.D. = not detected.

Table S3. Additive Screening

		
Entry	Additive	Yield (%) ^[a]
1	Bn ₃ N	71
2	DABCO	71
3	<i>N</i> -phenylpiperidine	73 ^[b]
4	<i>N</i> -methylpiperidine	74
5	Me ₂ N <i>n</i> Bu	68
6	TMEDA	60
7	<i>N,N</i> -dimethylaniline	75 ^[b]
8	DIPEA	77
9	Et ₃ N	67
10	HEH	28 ^[b]
11	Ascorbate sodium	trace

[a] **1a** (0.1 mmol, 1 equiv), **2a** (0.2 mmol, 2 equiv), additive (0.2 mmol, 2 equiv), Ir(ppy)₃ (0.01 equiv), CH₂Cl₂ (2 mL) for 2 h. Yields were determined through crude ¹H spectrum using triphenylmethane as internal standard. [b] The reaction was carried out for 12 h. DABCO = 1,4-Diaza[2.2.2]bicyclooctane. TMEDA = *N,N,N',N'*-Tetramethylethane-1,2-diamine. HEH = Diethyl 2,6-dimethyl-1,4-dihydro-3,5-pyridinedicarboxylate.

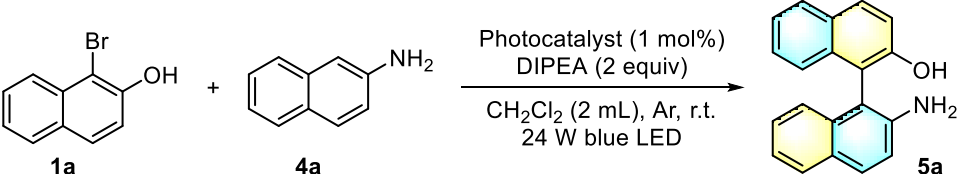
Table S4. Control Experiments

<div><div></div></div>		
Entry	Variations from above	Yield (%) ^[a]
1	-	77 (71) ^[b]
2	No Ir(ppy) ₃	N.R.
3	No light source	N.R.
4	No DIPEA	N.R.
5	24 W CFL	72
6	10 W purple LED	67

[a] 1-Bromo-2-naphthol **1a** (0.1 mmol, 1 equiv), 6-methyl-2-naphthol **2a** (0.2 mmol, 2 equiv), DIPEA (0.2 mmol, 2 equiv), Ir(ppy)₃ (0.01 equiv), CH₂Cl₂ (2 mL) for 2 h. Yields were determined through crude ¹H spectrum using triphenylmethane as internal standard. [b] Yield in the parentheses was isolated yield. N.R. = no reaction. CFL = Compact Fluorescent Lamp.

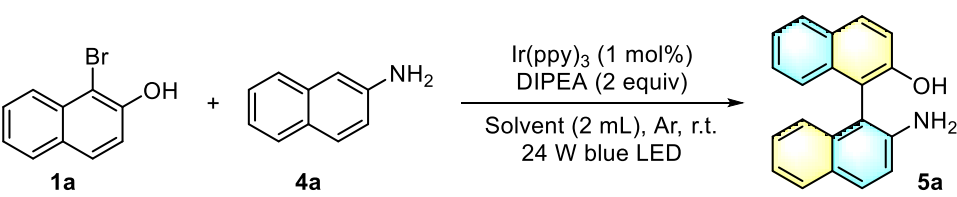
2. Optimization for Synthesis of NOBIN 5a

Table S5. Photocatalyst Screening

		
Entry	PC	Yield (%) ^[a]
1	Ir[dF(CF ₃)ppy] ₂ (bpy)PF ₆	17
2	Ir(ppy) ₂ (dtbbpy)PF ₆	trace
3	Ir[dF(Me)ppy] ₂ (dtbbpy)PF ₆	16
4	Ir(ppy) ₃	42
5	3DPAFIPN	15
6	3DPAFIPN	23 ^[b]
7	Ru(bpy) ₃ Cl ₂ ·6H ₂ O	N.R.
8	N-phenylphenothiazine	N.R.
9	4-CzIPN	N.R.

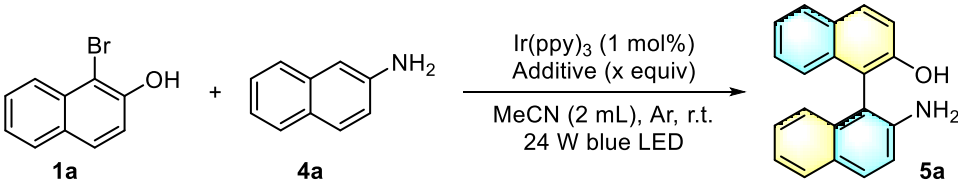
[a] 1-Bromo-2-naphthol **1a** (0.1 mmol, 1 equiv), 2-naphthylamine **4a** (0.2 mmol, 2 equiv), DIPEA (0.2 mmol, 2 equiv), photocatalyst (0.01 equiv), CH₂Cl₂ (2 mL) for 3 h. Yields were determined through crude ¹H spectrum using triphenylmethane as internal standard. [b] 3DPAFIPN (5 mol%). 3DPAFIPN = 2,4,6-Tris(diphenylamino)-5-fluoroisophthalonitrile. 4-CzIPN = 2,4,5,6-Tetra-9*H*-carbazol-9-yl-1,3-benzenedicarbonitrile. N.R. = no reaction.

Table S6. Solvent Screening

		
Entry	Solvent	Yield (%) ^[a]
1	1,4-Dioxane	14
2	PhF	47
3	DCE	39
4	Cyclohexane	trace
5	CHCl ₃	trace
6	EtOAc	37
7	Acetone	44
8	DMF	trace
9	PhCF ₃	trace
10	MeCN	54
11	EtOH	trace
12	MTBE	trace

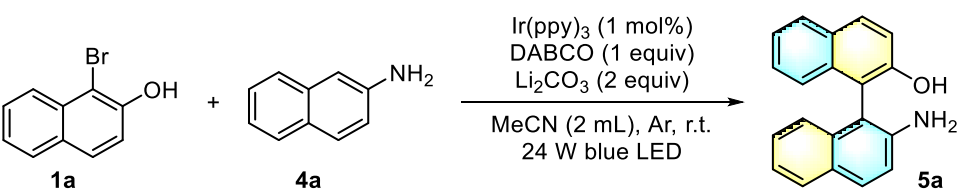
[a] **1a** (0.1 mmol, 1 equiv), **4a** (0.2 mmol, 2 equiv), DIPEA (0.2 mmol, 2 equiv), Ir(ppy)₃ (0.01 equiv), solvent (2 mL) for 3 h. Yields were determined through crude ¹H spectrum using triphenylmethane as internal standard. MTBE = Methyl *tert*-butyl ether. EtOAc = Ethyl acetate. DMF = *N,N*-Dimethylformamide.

Table S7. Additive Screening

			
Entry	Additive (x)		Yield (%) ^[a]
1	DIPEA (2)		54
2	DABCO (2)		61
3	<i>N</i> -methylpiperidine (2)		45
4	TMEDA (2)		33
5	<i>N,N</i> -dimethylaniline (2)		40 ^[b]
6	Et ₃ N (2)		52
7	DABCO (3)		64
8	DABCO (1)	Na ₂ CO ₃ (2)	67 ^[c]
9	DABCO (1)	Li ₂ CO ₃ (2)	70 ^[c]
10	DABCO (1)	K ₂ CO ₃ (2)	65 ^[c]
11	DABCO (1)	Cs ₂ CO ₃ (2)	trace ^[c]
12	DABCO (1)	<i>t</i> BuONa (2)	trace ^[c]
13	DABCO (1)	<i>t</i> BuOK (2)	trace ^[c]
14	DABCO (1)	NaHCO ₃ (2)	68 ^[c]
15	DABCO (1)	KHCO ₃ (2)	68 ^[c]
16	DABCO (1)	CsHCO ₃ (2)	66 ^[c]

[a] **1a** (0.1 mmol, 1 equiv), **4a** (0.2 mmol, 2 equiv), additive (x equiv), Ir(ppy)₃ (0.01 equiv), MeCN (2 mL) for 3 h. [b] 24 h. [c] 12 h. Yields were determined through crude ¹H spectrum using triphenylmethane as internal standard. TMEDA = *N,N,N',N'*-Tetramethylethane-1,2-diamine. DABCO = 1,4-Diaza[2.2.2]bicyclooctane.

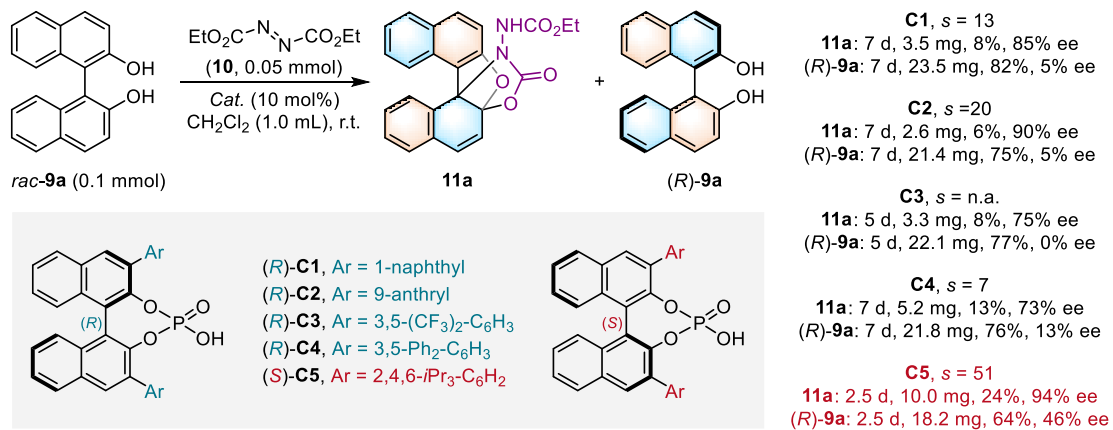
Table S8. Control Experiments

<div><div></div><div><div>1a</div><div>4a</div><div>5a</div></div></div>		
Entry	Variations from above	Yield (%) ^[a]
1	-	70 (62) ^[b]
2	No Ir(ppy) ₃	N.R.
3	No light source	N.R.
4	No DABCO and Li ₂ CO ₃	26

[a] 1-Bromo-2-naphthol **1a** (0.1 mmol, 1 equiv), 2-naphthylamine **4a** (0.2 mmol, 2 equiv), DABCO (0.2 mmol, 1 equiv), Li₂CO₃ (0.2 mmol, 2 equiv), Ir(ppy)₃ (0.01 equiv), MeCN (2 mL) for 12 h. Yields were determined through crude ¹H spectrum using triphenylmethane as internal standard. [b] Yield in the parentheses was isolated yield. N.R. = no reaction. DABCO = 1,4-Diaza[2.2.2]bicyclooctane.

III. Optimization for Kinetic Resolution of BINOLs

1. Optimization for CPAs

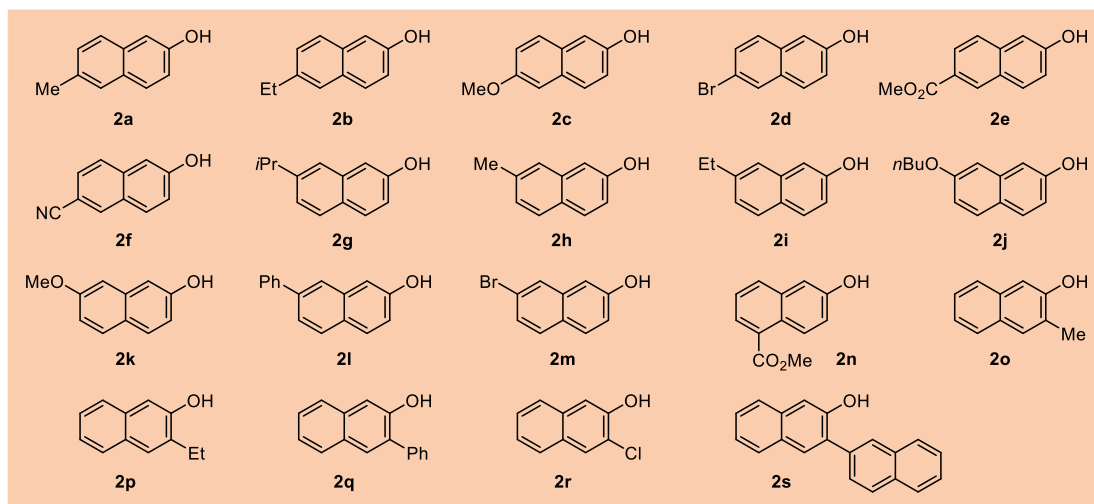


2. Optimization of other variations

<p> $\text{EtO}_2\text{C}-\text{N}=\text{N}-\text{CO}_2\text{Et}$ (10, X mmol) C5 (10 mol%) solv. (Y mL), temp. </p> <p> <i>rac</i>-9a (0.1 mmol) 11a (R)-9a </p>										
Entry	X	Solv.	Y	Temp. (°C)	t (d)	11a		(R)-9a		<i>s</i>
						Yield (%)	Ee (%)	Yield (%)	Ee (%)	
1	0.06	CH ₂ Cl ₂	1.0	r.t.	3.0	32	98	56	37	142
2	0.07	CH ₂ Cl ₂	1.0	r.t.	3.0	38	98	54	68	202
3	0.08	CH ₂ Cl ₂	1.0	r.t.	3.0	38	98	51	60	183
4	0.09	CH ₂ Cl ₂	1.0	r.t.	3.0	39	98	52	77	232
5	0.10	CH ₂ Cl ₂	1.0	r.t.	3.0	44	97	47	83	171
6	0.12	CH ₂ Cl ₂	1.0	r.t.	3.0	43	96	52	87	140
7	0.15	CH ₂ Cl ₂	1.0	r.t.	3.0	51	95	43	92	129
8	0.07	CH ₂ Cl ₂	0.5	r.t.	2.5	50	96	50	72	106
9	0.07	CH ₂ Cl ₂	0.3	r.t.	2.5	48	96	51	67	99
10	0.07	CH ₂ Cl ₂	0.5	40	1.0	42	97	54	71	140
11	0.07	CH ₂ Cl ₂	0.5	r.t.	2.5	44	98	53	75	224
12	0.07	CCl ₄	0.5	r.t.	2.5	21	96	66	27	64
13	0.07	ClPh	0.5	r.t.	2.5	45	98	53	74	221
14	0.07	CHCl ₃	0.5	r.t.	2.5	38	98	52	71	211
15	0.15	CH ₂ Cl ₂	0.5	40	1.0	57	92	38	92	79
16	0.15	DCE	0.5	40	1.0	53	94	40	94	115
17	0.15	CHCl ₃	0.5	40	1.0	50	94	39	90	100
18	0.15	ClPh	0.5	40	1.0	52	93	33	95	103

IV. Substrate Synthesis and Characterization

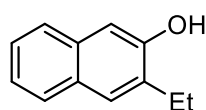
1. Substituted 2-naphthols



Substrates **2c**, **2d**, **2f**, **2k**, **2m** and **2o** are commercially available. Substrates **2l** and **2e** were prepared according to procedure of the reported literature.^[1a] Substrates **2a**, **2b**, **2h-2j** were prepared according to procedure of the reported literature.^[1b] Substrates **2g**,^[1c] **2n**,^[1d] **2q**,^[1e] **2r**,^[1f] **2s**^[1e] were prepared according to the reported procedure, respectively.

Synthesis of substrate **2p**:

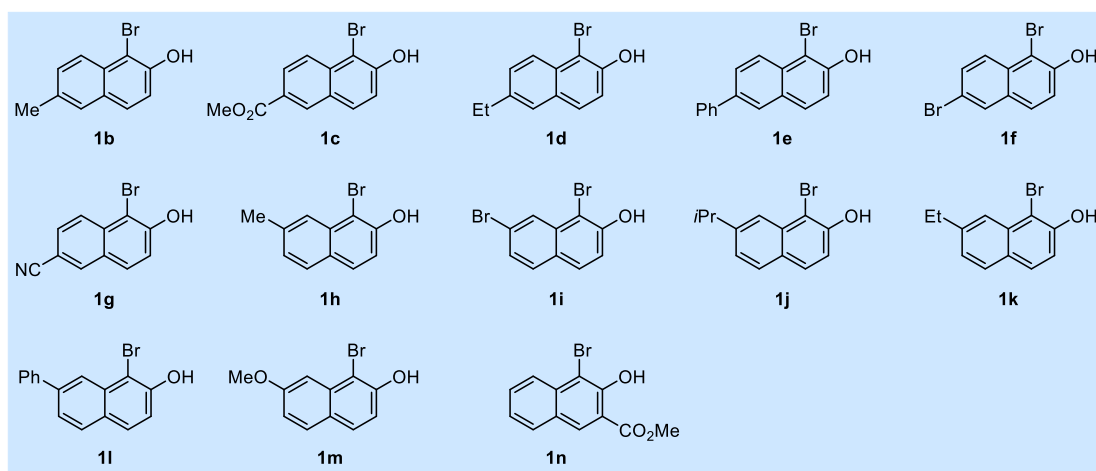
The procedure was based on the reported literature.^[1b] 3-Bromo-2-naphthalenol (0.45 g, 2 mmol) was dissolved in dry THF (20 mL) and placed in a pressure vessel. EtMgBr (6 mL, 1.0 M in THF) was added dropwise *via* syringe. After completion of the addition, PdCl₂(dppf) (5 mol%) was added in small portions. The flask was sealed and heated at 80 °C for 12 h under argon. The reaction was carefully quenched with water. The mixture was then extracted with EtOAc (3 × 20 mL) and the combined organic layers were dried over anhydrous Na₂SO₄. After evaporation of solvents, the crude product was purified by silica chromatography (eluent: EA/PE = 1/10) to **2p** as an off-white solid in 31% yield.



3-Ethyl-2-naphthol (**2p**)

^1H NMR (400 MHz, CDCl_3) δ 7.71 (d, $J = 8.1$ Hz, 1H), 7.61 (d, $J = 8.1$ Hz, 1H), 7.58 (s, 1H), 7.38 – 7.27 (m, 2H), 7.03 (s, 1H), 5.00 (s, 1H), 2.79 (q, $J = 7.5$ Hz, 2H), 1.32 (t, $J = 7.5$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.51, 133.33, 132.50, 129.42, 127.80, 127.35, 125.92, 125.73, 123.66, 109.38, 23.61, 14.03. HRMS (ESI) $[\text{M}+\text{H}]^+$ calculated m/z for $[\text{C}_{12}\text{H}_{13}\text{O}]^+$: 173.0961, found: 173.0962.

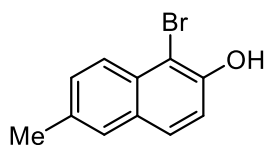
2. Substituted 1-bromo-2-naphthols



Substrate **1n** was prepared according to procedure of the reported literature.^[2]

Procedures for synthesis of substrates **1b-1m**:

To the solution of substituted 2-naphthol (2 mmol, 1 equiv) in MeCN (10 mL) at 0 °C was added *N*-bromosuccinimide (NBS, 2.1 mmol, 1.05 equiv) slowly. Then the reaction mixture was stirred at r.t. for 2 h. After completion (TLC monitoring), the mixture was concentrated and purified through silica chromatography to afford pure product.



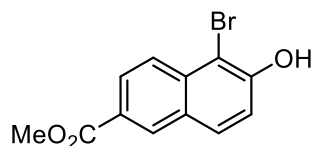
1-Bromo-6-methylnaphthalen-2-ol (**1b**)

Following the procedure, **1b** was obtained as a white solid in 97% yield.

^1H NMR (400 MHz, CDCl_3) δ 7.95 (d, $J = 8.6$ Hz, 1H), 7.67 (d, $J = 8.8$ Hz, 1H), 7.57 (s, 1H), 7.44 – 7.41 (m, 1H), 7.27 (d, $J = 8.8$ Hz, 1H), 5.92 (s, 1H), 2.53 (s, 3H). ^{13}C NMR (100 MHz,

CDCl_3) δ 149.99, 133.80, 130.59, 130.11, 129.97, 128.72, 127.34, 125.29, 117.20, 106.19, 21.30.

HRMS (ESI) $[\text{M}-\text{H}]^-$ calculated m/z for $[\text{C}_{11}\text{H}_8\text{BrO}]^-$: 234.9764, found: 234.9757.

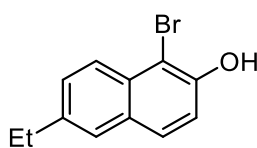


Methyl 5-bromo-6-hydroxy-2-naphthoate (**1c**)

Following the procedure, **1c** was obtained as a white solid in 93% yield.

^1H NMR (400 MHz, Acetone- d_6) δ 9.57 (s, 1H), 8.54 (s, 1H), 8.18 – 8.15 (m, 1H), 8.10 – 8.08 (m, 1H), 8.02 – 7.99 (m, 1H), 7.40 (d, J = 8.9 Hz, 1H), 3.93 (s, 3H). ^{13}C NMR (100 MHz, Acetone- d_6) δ 167.09, 155.30, 136.36, 131.86, 131.51, 129.26, 127.70, 126.46, 126.32, 119.91, 105.68, 52.40.

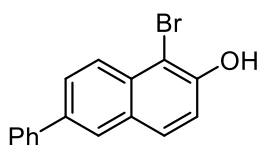
HRMS (ESI) $[\text{M}-\text{H}]^-$ calculated m/z for $[\text{C}_{12}\text{H}_8\text{BrO}_3]^-$: 278.9662, found: 278.9658.



1-Bromo-6-ethylnaphthalen-2-ol (**1d**)

Following the procedure, **1d** was obtained as an off-white solid in 97% yield.

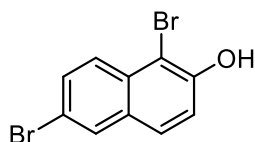
^1H NMR (400 MHz, CDCl_3) δ 7.93 (d, J = 8.6 Hz, 1H), 7.64 (d, J = 8.9 Hz, 1H), 7.53 (s, 1H), 7.40 – 7.42 (m, 1H), 7.21 (d, J = 9.0 Hz, 1H), 5.85 (s, 1H), 2.78 (q, J = 7.6 Hz, 2H), 1.30 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 150.06, 140.19, 130.82, 130.03, 129.11, 128.92, 126.10, 125.44, 117.19, 106.20, 28.68, 15.63. HRMS (ESI) $[\text{M}-\text{H}]^-$ calculated m/z for $[\text{C}_{12}\text{H}_{10}\text{BrO}]^-$: 248.9921, found: 248.9915.



1-Bromo-6-phenylnaphthalen-2-ol (**1e**)

Following the procedure, **1e** was obtained as a white solid in 95% yield.

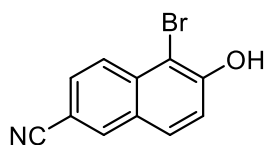
^1H NMR (400 MHz, Acetone- d_6) δ 9.06 (s, 1H), 8.19 (d, J = 8.8 Hz, 1H), 8.16 – 8.11 (m, 1H), 7.91 (d, J = 8.9 Hz, 2H), 7.80 – 7.77 (m, 2H), 7.52 – 7.48 (m, 2H), 7.40 – 7.34 (m, 2H). ^{13}C NMR (100 MHz, Acetone- d_6) δ 153.22, 141.15, 137.10, 133.28, 130.65, 130.34, 129.82, 128.23, 127.84, 127.82, 126.81, 126.73, 119.46, 105.62. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{16}\text{H}_{10}\text{BrO}]^-$: 296.9921, found: 296.9917.



1,6-Dibromonaphthalen-2-ol (**1f**)

Following the procedure, **1f** was obtained as a white solid in 96% yield.

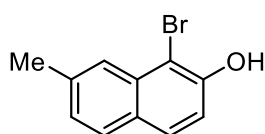
^1H NMR (400 MHz, CDCl_3) δ 7.90 – 7.84 (m, 2H), 7.61 – 7.57 (m, 2H), 7.26 – 7.23 (m, 1H), 5.94 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 151.03, 131.14, 131.05, 130.72, 130.18, 128.48, 127.32, 118.40, 118.14, 106.23. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{10}\text{H}_5\text{Br}_2\text{O}]^-$: 298.8713, found: 298.8709.



5-Bromo-6-hydroxy-2-naphthonitrile (**1g**)

Following the procedure, MeCN (20 mL) was used and **1g** was obtained as an off-white solid in 91% yield.

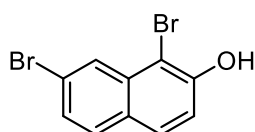
^1H NMR (400 MHz, Acetone- d_6) δ 9.75 (s, 1H), 8.35 (s, 1H), 8.20 (d, J = 8.8 Hz, 1H), 7.96 (d, J = 8.9 Hz, 1H), 7.77 (d, J = 8.8 Hz, 1H), 7.44 (d, J = 8.9 Hz, 1H). ^{13}C NMR (100 MHz, Acetone- d_6) δ 155.81, 135.64, 135.11, 130.75, 129.10, 129.03, 127.45, 120.66, 119.54, 107.83, 105.73. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{11}\text{H}_5\text{BrNO}]^-$: 245.9560, found: 245.9554.



1-Bromo-7-methylnaphthalen-2-ol (**1h**)

Following the procedure, **1h** was obtained as a white solid in 97% yield.

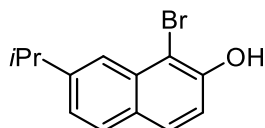
^1H NMR (400 MHz, CDCl_3) δ 7.78 (s, 1H), 7.66 – 7.63 (m, 2H), 7.22 – 7.16 (m, 2H), 5.90 (s, 1H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 150.73, 138.00, 132.53, 129.14, 128.21, 128.04, 126.45, 124.57, 116.28, 105.76, 22.14. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{11}\text{H}_8\text{BrO}]^-$: 234.9764, found: 234.9757.



1,7-Dibromonaphthalen-2-ol (**1i**)

Following the procedure, **1i** was obtained as a white solid in 93% yield.

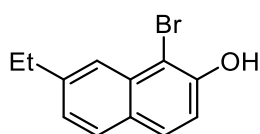
^1H NMR (400 MHz, CDCl_3) δ 8.19 (s, 1H), 7.69 – 7.61 (m, 2H), 7.45 (d, J = 8.5 Hz, 1H), 7.26 – 7.24 (m, 1H), 5.97 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 151.54, 133.67, 129.96, 129.42, 128.22, 127.79, 127.77, 122.65, 117.74, 105.02. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{10}\text{H}_5\text{Br}_2\text{O}]^-$: 298.8713, found: 298.8709.



1-Bromo-7-isopropylnaphthalen-2-ol (**1j**)

Following the procedure, **1j** was obtained as a brown solid in 96% yield.

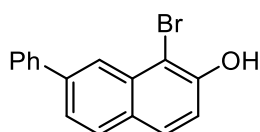
^1H NMR (400 MHz, CDCl_3) δ 7.82 (s, 1H), 7.71 – 7.66 (m, 2H), 7.30 – 7.27 (m, 1H), 7.19 (d, J = 8.8 Hz, 1H), 5.89 (s, 1H), 3.15 – 3.05 (m, 1H), 1.35 (d, J = 6.9 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 150.73, 148.87, 132.51, 129.11, 128.44, 128.41, 123.93, 122.08, 116.39, 106.21, 34.72, 24.07. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{13}\text{H}_{12}\text{BrO}]^-$: 263.0077, found: 263.0073.



1-Bromo-7-ethylnaphthalen-2-ol (**1k**)

Following the procedure, **1k** was obtained as a light brown solid in 99% yield.

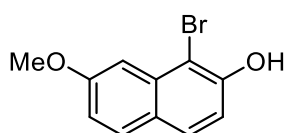
^1H NMR (400 MHz, CDCl_3) δ 7.79 (s, 1H), 7.68 – 7.65 (m, 2H), 7.25 – 7.22 (m, 1H), 7.18 (d, J = 8.8 Hz, 1H), 5.90 (s, 1H), 2.83 (q, J = 7.6 Hz, 2H), 1.33 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 150.73, 144.32, 132.57, 129.15, 128.36, 128.27, 125.39, 123.41, 116.34, 106.00, 29.49, 15.73. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{12}\text{H}_{10}\text{BrO}]^-$: 248.9921, found: 248.9915.



1-Bromo-7-phenylnaphthalen-2-ol (**1l**)

Following the procedure, **1l** was obtained as a white solid in 97% yield.

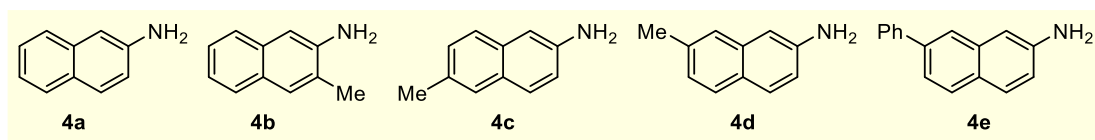
^1H NMR (400 MHz, CDCl_3) δ 8.20 (s, 1H), 7.81 (d, J = 8.4 Hz, 1H), 7.73 – 7.70 (m, 3H), 7.63 – 7.60 (m, 1H), 7.50 – 7.46 (m, 2H), 7.41 – 7.37 (m, 1H), 7.24 (d, J = 8.8 Hz, 1H), 5.93 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 151.07, 141.03, 140.80, 132.66, 129.17, 129.04, 128.97, 128.93, 127.82, 127.72, 124.03, 123.55, 117.27, 106.55. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{16}\text{H}_{10}\text{BrO}]^-$: 296.9921, found: 296.9917.



1-Bromo-7-methoxynaphthalen-2-ol (**1m**)

Following the procedure, **1m** was obtained as a white solid in 87% yield.

^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 10.48 (s, 1H), 7.76 – 7.70 (m, 2H), 7.34 (s, 1H), 7.13 – 7.10 (m, 1H), 7.02 – 7.00 (m, 1H), 3.89 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 159.00, 153.03, 134.32, 130.16, 128.68, 123.93, 115.59, 115.54, 104.04, 103.59, 55.19. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{11}\text{H}_8\text{BrO}_2]^-$: 250.9713, found: 250.9708.



Substrate **4a** was commercially available. Substrates **4b**^[3a] and **4c-4e**^[3b] were prepared according to procedure of the reported literature, respectively.

V. General Procedure and Reaction Set-up

General Procedure A (GPA) for BINOL derivatives:

To a 10 mL Schlenk tube containing a stir bar were charged with substituted 1-bromo-2-naphthol **1** (0.2 mmol, 1.0 equiv), substituted 2-naphthol **2** (0.4 mmol, 2.0 equiv) and Ir(ppy)₃ (0.002 mmol, 0.01 equiv, 1.3 mg). The mixture was then placed under vacuum and backfilled with argon three times, followed by the addition of anhydrous CH₂Cl₂ (4 mL) and DIPEA (0.4 mmol, 2.0 equiv, 66 μ L). Then the tube was placed approximate 4~5 cm away from 24 W blue LED and stir vigorously for 2 h with a cooling fan to maintain the reaction at r. t. (about 25 °C). Upon completion of the reaction, the mixture was concentrated and purified by silica chromatography to afford the pure product.

General Procedure B (GPB) for NOBIN derivatives:

To a 10 mL Schlenk tube containing a stir bar were charged with substituted 1-bromo-2-naphthol **1** (0.2 mmol, 1.0 equiv), substituted 2-naphthylamine **4** (0.4 mmol, 2.0 equiv), Ir(ppy)₃ (0.002 mmol, 0.01 equiv, 1.3 mg), DABCO (0.2 mmol, 1.0 equiv, 22.4 mg) and Li₂CO₃ (0.4 mmol, 2 equiv, 29.5 mg). Then the mixture was then placed under vacuum and backfilled with argon three times, followed by the addition of anhydrous MeCN (4 mL). Then the tube was placed approximate 4~5 cm away from 24 W blue LED and stir vigorously for 12 h with a cooling fan to maintain the reaction at r. t. (about 25 °C). Upon completion of the reaction, the mixture was concentrated and purified by silica chromatography to afford the pure product.

Reaction Set-up and Spectrum of blue LED:



Figure S1. Reaction Set-Up

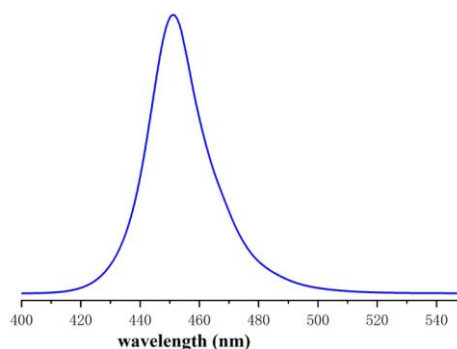
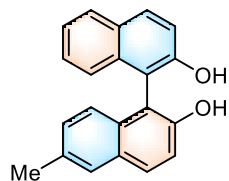


Figure S2. Spectrum of blue LED ($\lambda_{\text{max}} = 450 \text{ nm}$)

General Procedure C (GPC) for kinetic resolution of BINOLs:

To a round bottle flask with a magnetic stirring bar were added racemic BINOL **9** (0.1 mmol, 1.0 equiv) and (*S*)-**C5** (0.01 mmol, 10 mol%), followed by the addition of diethyl azodicarboxylate **10** (0.15 mmol, 1.5 equiv) dissolved in DCE (0.5 mL), then the reaction mixture stirred at 40 °C and detected by TLC. The corresponding (*R*)-**9** were isolated by preparative thin layer chromatography (PTLC) using PE/EtOAc or PE/EtOAc/CH₂Cl₂ or PE/MTBE as eluent, then target compound **11** and LiOH (2 N, 7.0 equiv) were dissolved in THF (1.0 mL) and stirred at 70 °C for one day. The reaction mixture was detected by TLC until **11** were completely transformed into (*S*)-**9**. Then the reaction mixture was acidified by 6 N HCl (2 mL). The mixture was extracted with dichloromethane and the combined organic layers were dried with anhydrous Na₂SO₄. Then the organic layers were evaporated *in vacuo* and the residue was subjected to chromatography (silica gel, PE/EtOAc: 10/1 to 5/1) to afford the desired product (*S*)-**9**.

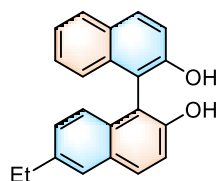
VI. Characterization of Products



6-Methyl-[1,1'-binaphthalene]-2,2'-diol (**3a**)

Following **GPA**, **3a** was obtained as an off-white solid in 71% yield.

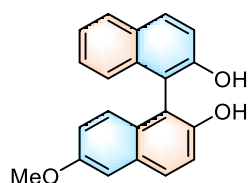
^1H NMR (400 MHz, CDCl_3) δ 7.85 (d, $J = 8.9$ Hz, 1H), 7.79 (d, $J = 8.1$ Hz, 1H), 7.75 (d, $J = 9.0$ Hz, 1H), 7.56 (s, 1H), 7.32 – 7.23 (m, 4H), 7.11 – 7.07 (m, 2H), 6.99 (d, $J = 8.5$ Hz, 1H), 5.12 (s, 1H), 5.01 (s, 1H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.76, 152.11, 133.62, 133.58, 131.66, 131.28, 130.66, 129.72, 129.68, 129.45, 128.42, 127.53, 127.48, 124.36, 124.21, 124.04, 117.85, 117.83, 111.28, 110.97, 21.41. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{21}\text{H}_{15}\text{O}_2]^-$: 299.1078, found: 299.1073.



6-Ethyl-[1,1'-binaphthalene]-2,2'-diol (**3b**)

Following **GPA**, **3b** was obtained as a white solid in 74% yield.

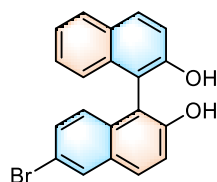
^1H NMR (400 MHz, CDCl_3) δ 7.92 (d, $J = 8.9$ Hz, 1H), 7.87 – 7.84 (m, 2H), 7.65 (s, 1H), 7.36 – 7.25 (m, 4H), 7.17 – 7.13 (m, 2H), 7.05 (d, $J = 8.6$ Hz, 1H), 5.06 (s, 1H), 4.98 (s, 1H), 2.75 (q, $J = 7.6$ Hz, 2H), 1.29 (t, $J = 7.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.83, 152.24, 140.06, 133.57, 131.88, 131.40, 131.00, 129.80, 129.53, 128.77, 128.49, 127.55, 126.31, 124.40, 124.33, 124.11, 117.86, 117.81, 111.22, 110.85, 28.80, 15.63. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{22}\text{H}_{17}\text{O}_2]^-$: 313.1234, found: 313.1231.



6-Methoxy-[1,1'-binaphthalene]-2,2'-diol (**3c**)

Following **GPA**, **2c** (0.6 mmol) was used and **3c** was obtained as a white solid in 36% yield.

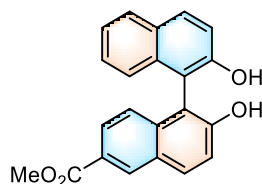
^1H NMR (400 MHz, CDCl_3) δ 7.98 (d, J = 8.9 Hz, 1H), 7.91 – 7.86 (m, 2H), 7.71 – 7.31 (m, 4H), 7.24 (d, J = 2.6 Hz, 1H), 7.18 (d, J = 8.3 Hz, 1H), 7.08 (d, J = 9.2 Hz, 1H), 7.00 (dd, J = 9.2, 2.5 Hz, 1H), 5.15 (s, 1H), 4.96 (s, 1H), 3.93 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 156.52, 152.79, 151.18, 133.55, 131.46, 130.54, 130.14, 129.54, 128.70, 128.51, 127.58, 125.93, 124.36, 124.13, 119.94, 118.32, 117.86, 111.30, 111.22, 107.03, 55.51. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{21}\text{H}_{15}\text{O}_3]^-$: 315.1027, found: 315.1025.



6-Bromo-[1,1'-binaphthalene]-2,2'-diol (**3d**)

Following **GPA**, **3d** was obtained as a pale yellow solid in 43% yield.

^1H NMR (400 MHz, CDCl_3) δ 8.01 (d, J = 2.0 Hz, 1H), 7.94 (d, J = 9.0 Hz, 1H), 7.87 – 7.82 (m, 2H), 7.39 – 7.29 (m, 5H), 7.07 (d, J = 8.4 Hz, 1H), 6.99 (d, J = 9.0 Hz, 1H), 5.12 (s, 1H), 5.03 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 153.11, 152.83, 133.42, 132.16, 131.78, 130.81, 130.68, 130.51, 130.47, 129.57, 128.60, 127.77, 126.25, 124.31, 124.13, 119.06, 117.99, 117.92, 111.47, 110.40. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{20}\text{H}_{12}\text{BrO}_2]^-$: 363.0026, found: 363.0024.

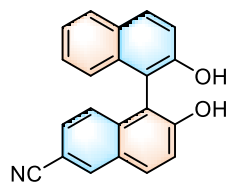


Methyl 2,2'-dihydroxy-[1,1'-binaphthalene]-6-carboxylate (**3e**)

Following **GPA**, **2e** (0.6 mmol) was used and **3e** was obtained as a yellow solid in 67% yield.

^1H NMR (400 MHz, CDCl_3) δ 8.39 (s, 1H), 7.93 (d, J = 9.0 Hz, 2H), 7.85 (d, J = 8.0 Hz, 1H), 7.75 (d, J = 8.8 Hz, 1H), 7.38 – 7.32 (m, 3H), 7.29 – 7.24 (m, 1H), 7.14 (d, J = 8.8 Hz, 1H), 7.05 (d, J = 8.4 Hz, 1H), 5.68 (s, 1H), 5.46 (s, 1H), 3.86 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.31, 154.89, 153.15, 136.24, 133.51, 132.76, 131.71, 131.53, 129.52, 128.56, 128.46, 127.65, 126.79,

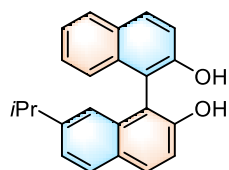
125.33, 124.62, 124.17, 124.10, 118.70, 118.21, 111.95, 110.45, 52.34. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{22}H_{15}O_4]^-$: 343.0976, found: 343.0971.



2,2'-Dihydroxy-[1,1'-binaphthalene]-6-carbonitrile (**3f**)

Following **GPA**, **2f** (0.6 mmol) was used and **3f** was obtained as an off-white solid in 65% yield.

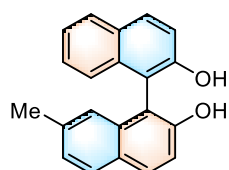
1H NMR (400 MHz, $CDCl_3$) δ 8.21 (s, 1H), 8.00 – 7.96 (m, 2H), 7.90 – 7.87 (m, 1H), 7.48 (d, J = 9.0 Hz, 1H), 7.40 – 7.28 (m, 4H), 7.20 (d, J = 8.8 Hz, 1H), 7.03 (d, J = 8.4 Hz, 1H), 5.33 (br s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.46, 152.96, 135.59, 134.51, 133.35, 132.06, 131.86, 129.59, 128.70, 128.42, 128.08, 127.91, 125.74, 124.43, 123.90, 119.89, 119.31, 118.05, 112.31, 109.81, 107.30. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{21}H_{12}NO_2]^-$: 310.0874, found: 310.0871.



7-Isopropyl-[1,1'-binaphthalene]-2,2'-diol (**3g**)

Following **GPA**, **3g** was obtained as a pale yellow solid in 64% yield.

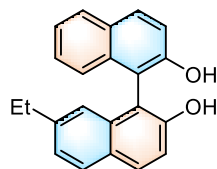
1H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 9.0 Hz, 1H), 7.88 – 7.84 (m, 2H), 7.80 (d, J = 8.5 Hz, 1H), 7.36 – 7.32 (m, 2H), 7.30 – 7.25 (m, 3H), 7.18 – 7.15 (m, 1H), 6.92 (s, 1H), 5.10 (s, 1H), 5.03 (s, 1H), 2.81 – 2.74 (m, 1H), 1.11 (d, J = 2.3 Hz, 3H), 1.09 (d, J = 2.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.92, 152.82, 148.38, 133.63, 133.47, 131.38, 131.16, 129.54, 128.61, 128.47, 128.17, 127.49, 124.47, 124.08, 123.31, 121.06, 117.89, 116.99, 111.20, 110.72, 34.45, 23.97, 23.90. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{23}H_{19}O_2]^-$: 327.1391, found: 327.1387.



7-Methyl-[1,1'-binaphthalene]-2,2'-diol (**3h**)

Following **GPA**, **3h** was obtained as an off-white solid in 63% yield.

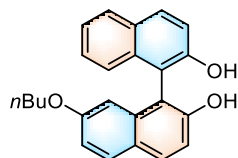
^1H NMR (400 MHz, CDCl_3) δ 7.93 – 7.91 (m, 1H), 7.87 – 7.84 (m, 2H), 7.74 (d, J = 8.2 Hz, 1H), 7.36 – 7.32 (m, 2H), 7.30 – 7.24 (m, 2H), 7.19 – 7.13 (m, 2H), 6.89 (s, 1H), 5.08 (s, 1H), 5.01 (s, 1H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.96, 152.85, 137.59, 133.73, 133.55, 131.38, 131.20, 129.55, 128.49, 128.37, 127.75, 127.56, 126.41, 124.37, 124.11, 123.23, 117.89, 116.84, 111.20, 110.36, 22.00. HRMS (ESI) $[\text{M}-\text{H}]^-$ calculated m/z for $[\text{C}_{21}\text{H}_{15}\text{O}_2]^-$: 299.1078, found: 299.1074.



7-Ethyl-[1,1'-binaphthalene]-2,2'-diol (**3i**)

Following **GPA**, **3i** was obtained as an off-white solid in 72% yield.

^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, J = 8.9 Hz, 1H), 7.95 – 7.91 (m, 2H), 7.84 (d, J = 8.3 Hz, 1H), 7.42 – 7.38 (m, 2H), 7.37 – 7.27 (m, 3H), 7.22 (d, J = 8.3 Hz, 1H), 6.97 (s, 1H), 5.16 (s, 1H), 5.08 (s, 1H), 2.60 (q, J = 7.6 Hz, 2H), 1.14 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.95, 152.85, 143.92, 133.73, 133.52, 131.38, 131.21, 129.55, 128.54, 128.48, 128.00, 127.54, 125.15, 124.42, 124.11, 122.21, 117.90, 116.93, 111.20, 110.55, 29.29, 15.73. HRMS (ESI) $[\text{M}-\text{H}]^-$ calculated m/z for $[\text{C}_{22}\text{H}_{17}\text{O}_2]^-$: 313.1234, found: 313.1232.

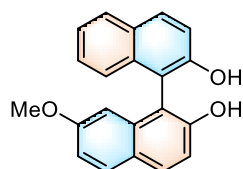


7-Butoxy-[1,1'-binaphthalene]-2,2'-diol (**3j**)

Following **GPA**, **3j** was obtained as white foam in 64% yield.

^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, J = 9.0 Hz, 1H), 7.91 – 7.85 (m, 2H), 7.78 (d, J = 8.9 Hz, 1H), 7.42 – 7.33 (m, 3H), 7.24 – 7.20 (m, 2H), 7.06 (dd, J = 8.9, 2.5 Hz, 1H), 6.46 (d, J = 2.5 Hz, 1H), 5.19 (s, 1H), 5.07 (s, 1H), 3.78 – 3.65 (m, 2H), 1.68 – 1.58 (m, 2H), 1.39 – 1.34 (m, 2H),

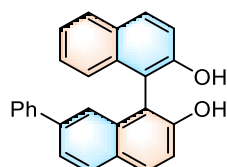
0.88 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 158.69, 153.41, 152.80, 135.04, 133.39, 131.42, 131.15, 130.02, 129.58, 128.47, 127.55, 124.74, 124.38, 124.10, 117.86, 116.42, 115.10, 111.19, 110.02, 104.14, 67.55, 31.08, 19.23, 13.89. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{24}\text{H}_{21}\text{O}_3]^-$: 357.1496, found: 357.1492.



7-Methoxy-[1,1'-binaphthalene]-2,2'-diol (**3k**)

Following **GPA**, **3k** was obtained as a white solid in 60% yield.

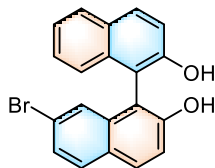
^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, $J = 8.9$ Hz, 1H), 7.90 – 7.85 (m, 2H), 7.78 (d, $J = 8.9$ Hz, 1H), 7.42 – 7.32 (m, 3H), 7.23 – 7.21 (m, 2H), 7.06 – 7.03 (m, 1H), 6.46 (d, $J = 2.5$ Hz, 1H), 5.21 (s, 1H), 5.11 (s, 1H), 3.56 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 159.17, 153.47, 152.79, 135.03, 133.37, 131.45, 131.15, 130.08, 129.57, 128.49, 127.56, 124.83, 124.34, 124.11, 117.86, 116.11, 115.24, 111.16, 110.17, 103.35, 55.21. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{21}\text{H}_{15}\text{O}_3]^-$: 315.1027, found: 315.1024.



7-Phenyl-[1,1'-binaphthalene]-2,2'-diol (**3l**)

Following **GPA**, **3l** was obtained as a white solid in 66% yield.

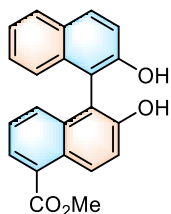
^1H NMR (400 MHz, CDCl_3) δ 7.93 – 7.90 (m, 3H), 7.84 – 7.82 (m, 1H), 7.60 (dd, $J = 8.4, 1.8$ Hz, 1H), 7.39 – 7.36 (m, 2H), 7.35 – 7.20 (m, 8H), 7.19 – 7.16 (m, 1H), 5.11 (s, 1H), 5.08 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 153.28, 152.89, 141.11, 140.46, 133.83, 133.48, 131.59, 131.20, 129.59, 129.08, 128.79, 128.72, 128.54, 127.64, 127.61, 127.48, 124.30, 124.18, 124.02, 122.22, 117.92, 111.36, 110.86. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{26}\text{H}_{17}\text{O}_2]^-$: 361.1234, found: 361.1230.



7-Bromo-[1,1'-binaphthalene]-2,2'-diol (**3m**)

Following **GPA**, **2m** (0.6 mmol) was used and **3m** was obtained as a white solid in 65% yield.

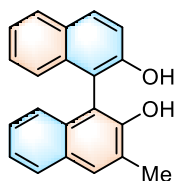
^1H NMR (400 MHz, CDCl_3) δ 7.95 (d, $J = 9.0$ Hz, 1H), 7.90 – 7.86 (m, 2H), 7.72 (d, $J = 8.7$ Hz, 1H), 7.43 (dd, $J = 8.6, 2.0$ Hz, 1H), 7.39 – 7.24 (m, 5H), 7.09 (d, $J = 8.3$ Hz, 1H), 5.12 (s, 1H), 5.02 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 153.67, 152.88, 134.95, 133.37, 131.87, 131.40, 130.15, 129.61, 128.64, 127.99, 127.82, 127.67, 126.37, 124.33, 124.05, 122.32, 118.32, 117.97, 110.59, 110.17. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{20}\text{H}_{12}\text{BrO}_2]^-$: 363.0026, found: 363.0024.



Methyl 2,2'-dihydroxy-[1,1'-binaphthalene]-5-carboxylate (**3n**)

Following **GPA**, **2n** (0.6 mmol) was used and **3n** was obtained as a pale yellow solid in 60% yield.

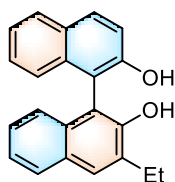
^1H NMR (400 MHz, CDCl_3) δ 8.97 (d, $J = 9.4$ Hz, 1H), 7.97 (d, $J = 7.0$ Hz, 1H), 7.92 (d, $J = 9.0$ Hz, 1H), 7.84 (d, $J = 8.1$ Hz, 1H), 7.44 (d, $J = 9.4$ Hz, 1H), 7.36 – 7.24 (m, 5H), 7.07 (d, $J = 8.3$ Hz, 1H), 5.29 (s, 2H), 3.98 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 168.31, 152.98, 152.92, 134.32, 133.57, 131.62, 129.50, 129.49, 129.28, 128.52, 128.15, 127.83, 127.66, 127.35, 126.22, 124.22, 124.19, 119.50, 117.95, 111.74, 110.94, 52.46. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{22}\text{H}_{15}\text{O}_4]^-$: 343.0976, found: 343.0971.



3-Methyl-[1,1'-binaphthalene]-2,2'-diol (**3o**)

Following **GPA**, **3o** was obtained as a pale yellow solid in 80% yield.

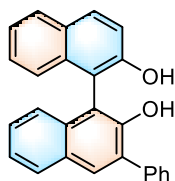
^1H NMR (400 MHz, Acetone- d_6) δ 8.05 (s, 1H), 7.93 (d, J = 8.9 Hz, 1H), 7.89 (d, J = 8.1, 1H), 7.82 – 7.78 (m, 2H), 7.44 (s, 1H), 7.37 (d, J = 8.8 Hz, 1H), 7.32 – 7.20 (m, 3H), 7.17 – 7.12 (m, 1H), 7.07 – 7.04 (m, 1H), 6.99 – 6.97 (m, 1H), 2.50 (s, 3H). ^{13}C NMR (100 MHz, Acetone- d_6) δ 155.02, 153.52, 135.58, 134.10, 130.95, 130.07, 130.02, 128.93, 128.28, 128.10, 127.22, 126.06, 125.30, 125.16, 123.77, 123.67, 119.62, 119.53, 114.19, 114.11, 17.57. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{21}\text{H}_{15}\text{O}_2]^-$: 299.1078, found: 299.1075.



3-Ethyl-[1,1'-binaphthalene]-2,2'-diol (**3p**)

Following **GPA**, **3p** was obtained as an off-white solid in 60% yield.

^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, J = 8.9 Hz, 1H), 7.91 – 7.86 (m, 3H), 7.43 – 7.32 (m, 4H), 7.30 – 7.26 (m, 1H), 7.20 (d, J = 8.3 Hz, 1H), 7.13 (d, J = 8.1 Hz, 1H), 5.19 (s, 1H), 5.13 (s, 1H), 2.97 (q, J = 7.5 Hz, 2H), 1.45 (t, J = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.92, 151.87, 133.61, 133.02, 132.15, 131.46, 129.64, 129.58, 129.16, 128.50, 127.91, 127.57, 126.58, 124.40, 124.12, 124.07, 124.03, 117.85, 111.33, 110.44, 24.00, 13.96. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{22}\text{H}_{17}\text{O}_2]^-$: 313.1234, found: 313.1232.

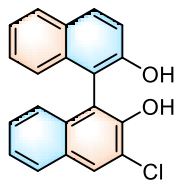


3-Phenyl-[1,1'-binaphthalene]-2,2'-diol (**3q**)

Following **GPA**, **3q** was obtained as a pale yellow solid in 62% yield.

^1H NMR (400 MHz, CDCl_3) δ 8.00 (s, 1H), 7.94 – 7.84 (m, 3H), 7.71 (d, J = 7.4 Hz, 2H), 7.49 – 7.45 (m, 2H), 7.40 – 7.25 (m, 6H), 7.22 (d, J = 7.8 Hz, 1H), 7.13 (d, J = 8.4 Hz, 1H), 5.29 (s, 1H), 5.11 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.76, 150.38, 137.54, 133.54, 133.08, 131.59, 131.44, 130.82, 129.73, 129.57, 128.61, 128.56, 128.52, 127.91, 127.53, 124.50, 124.43, 124.31,

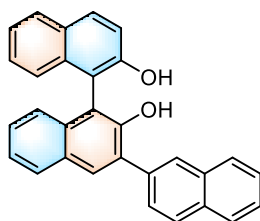
124.11, 117.87, 111.92, 111.59. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{26}H_{17}O_2]^-$: 361.1234, found: 361.1231.



3-Chloro-[1,1'-binaphthalene]-2,2'-diol (**3r**)

Following **GPA**, **2r** (0.6 mmol) was used and **3r** was obtained as an off-white solid in 67% yield.

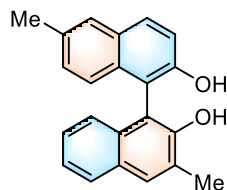
1H NMR (400 MHz, Acetone- d_6) δ 8.21 (brs, 2H), 8.11 (s, 1H), 7.93 (d, $J = 8.9$ Hz, 1H), 7.90 – 7.86 (m, 2H), 7.37 – 7.27 (m, 3H), 7.25 – 7.21 (m, 2H), 7.04 – 7.01 (m, 2H). ^{13}C NMR (100 MHz, Acetone- d_6) δ 153.77, 149.16, 134.25, 133.10, 130.14, 128.93, 128.90, 128.41, 127.98, 127.10, 126.39, 126.35, 124.57, 124.04, 123.84, 123.07, 122.84, 118.59, 116.60, 112.96. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{20}H_{12}ClO_2]^-$: 319.0531, found: 319.0528.



[1,1':3',2''-Ternaphthalene]-2,2'-diol (**3s**)

Following **GPA**, **3s** was obtained as white foam in 49% yield.

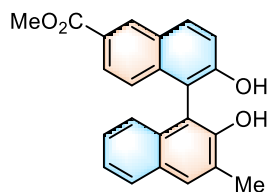
1H NMR (400 MHz, $CDCl_3$) δ 8.18 (s, 1H), 8.10 (s, 1H), 7.94 – 7.82 (m, 7H), 7.50 – 7.47 (m, 2H), 7.39 – 7.24 (m, 6H), 7.15 (d, $J = 8.4$ Hz, 1H), 5.37 (s, 1H), 5.16 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.79, 150.55, 135.11, 133.59, 133.56, 133.14, 132.90, 131.86, 131.47, 130.78, 129.64, 129.58, 128.63, 128.60, 128.54, 128.34, 128.03, 127.81, 127.58, 126.41, 126.37, 124.55, 124.43, 124.35, 124.13, 117.89, 112.05, 111.56. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{30}H_{19}O_2]^-$: 411.1391, found: 411.1385.



3,6'-Dimethyl-[1,1'-binaphthalene]-2,2'-diol (**3t**)

Following **GPA**, **3t** was obtained as an off-white solid in 73% yield.

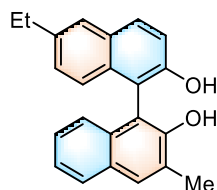
^1H NMR (400 MHz, Acetone- d_6) δ 7.89 (s, 1H), 7.84 – 7.77 (m, 3H), 7.66 (s, 1H), 7.36 – 7.32 (m, 2H), 7.28 – 7.24 (m, 1H), 7.16 – 7.12 (m, 1H), 7.08 (dd, J = 8.6, 1.9 Hz, 1H), 7.00 – 6.95 (m, 2H), 2.50 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, Acetone- d_6) δ 154.32, 153.46, 134.11, 133.70, 133.02, 130.31, 130.23, 130.02, 130.01, 129.36, 128.23, 128.08, 127.95, 126.02, 125.30, 125.22, 123.66, 119.57, 114.29, 113.93, 21.29, 17.57. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{22}\text{H}_{17}\text{O}_2]^-$: 313.1234, found: 313.1232.



Methyl 2,2'-dihydroxy-3'-methyl-[1,1'-binaphthalene]-6-carboxylate (**3u**)

Following **GPA**, **3u** was obtained as a white off-solid in 66% yield.

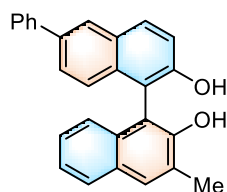
^1H NMR (400 MHz, DMSO- d_6) δ 9.82 (s, 1H), 8.63 (d, J = 1.8 Hz, 1H), 8.23 (s, 1H), 8.13 (d, J = 8.9 Hz, 1H), 7.80 – 7.76 (m, 2H), 7.72 (dd, J = 8.9, 1.8 Hz, 1H), 7.46 (d, J = 8.9 Hz, 1H), 7.24 – 7.20 (m, 1H), 7.14 – 7.09 (m, 1H), 7.03 (d, J = 8.9 Hz, 1H), 6.83 (d, J = 8.4 Hz, 1H), 3.87 (s, 3H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 166.62, 156.28, 152.04, 137.02, 132.65, 131.01, 128.59, 128.40, 127.49, 127.29, 127.14, 125.08, 124.68, 124.02, 123.38, 122.59, 119.75, 114.87, 114.84, 51.96, 17.59. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{23}\text{H}_{17}\text{O}_4]^-$: 357.1132, found: 357.1129.



6'-Ethyl-3-methyl-[1,1'-binaphthalene]-2,2'-diol (**3v**)

Following **GPA**, **3v** was obtained as an off-white solid in 69% yield.

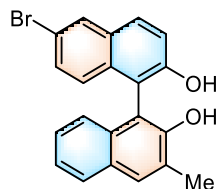
^1H NMR (400 MHz, CDCl_3) δ 7.85 (d, $J = 8.9$ Hz, 1H), 7.78 – 7.76 (m, 2H), 7.64 (s, 1H), 7.32 – 7.28 (m, 2H), 7.23 – 7.20 (m, 1H), 7.15 (dd, $J = 8.6, 1.8$ Hz, 1H), 7.07 – 7.04 (m, 2H), 5.12 (s, 1H), 4.95 (s, 1H), 2.74 (q, $J = 7.6$ Hz, 2H), 2.49 (s, 3H), 1.28 (t, $J = 7.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.26, 152.13, 140.01, 132.28, 131.91, 130.95, 130.77, 129.81, 129.51, 128.73, 127.66, 127.16, 126.50, 126.29, 124.38, 124.17, 124.01, 117.77, 111.12, 110.55, 28.80, 17.13, 15.64. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{22}\text{H}_{19}\text{O}_2]^-$: 327.1391, found: 327.1387.



3-Methyl-6'-phenyl-[1,1'-binaphthalene]-2,2'-diol (**3w**)

Following **GPA**, **3w** was obtained as a pale yellow solid in 65% yield.

^1H NMR (400 MHz, CDCl_3) δ 8.13 (d, $J = 1.9$ Hz, 1H), 8.04 (d, $J = 8.9$ Hz, 1H), 7.88 – 7.86 (m, 2H), 7.74 – 7.71 (m, 2H), 7.62 (dd, $J = 8.8, 1.9$ Hz, 1H), 7.53 – 7.50 (m, 2H), 7.45 – 7.38 (m, 3H), 7.33 – 7.27 (m, 2H), 7.18 (d, $J = 8.0$ Hz, 1H), 5.23 (s, 1H), 5.14 (s, 1H), 2.58 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.97, 152.18, 140.96, 136.97, 132.76, 132.26, 131.73, 130.94, 129.85, 129.55, 128.98, 127.73, 127.37, 127.35, 127.21, 126.61, 126.43, 124.99, 124.11, 118.31, 111.26, 110.27, 17.14. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{27}\text{H}_{19}\text{O}_2]^-$: 375.1391, found: 375.1387.

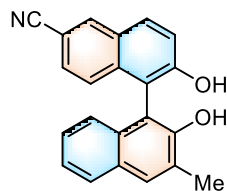


6'-Bromo-3-methyl-[1,1'-binaphthalene]-2,2'-diol (**3x**)

Following **GPA**, **3x** was obtained as a white foam in 46% yield.

^1H NMR (400 MHz, CDCl_3) δ 8.02 (d, $J = 2.0$ Hz, 1H), 7.86 – 7.78 (m, 3H), 7.38 – 7.31 (m, 3H), 7.25 – 7.21 (m, 1H), 7.02 – 6.98 (m, 2H), 5.08 (s, 1H), 5.03 (s, 1H), 2.50 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 153.18, 152.15, 132.19, 132.12, 131.17, 130.80, 130.71, 130.51, 130.47, 129.57,

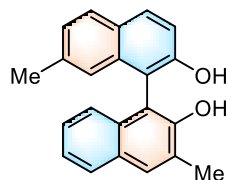
127.79, 127.21, 126.72, 126.32, 124.23, 123.90, 119.04, 117.98, 111.68, 109.70, 17.11. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{21}H_{14}BrO_2]^-$: 377.0183, found: 377.0180.



2,2'-Dihydroxy-3'-methyl-[1,1'-binaphthalene]-6-carbonitrile (**3y**)

Following **GPA**, **3y** was obtained as an off-white solid in 61% yield.

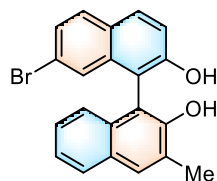
1H NMR (400 MHz, DMSO- d_6) δ 9.98 (brs, 1H), 8.51 (d, $J = 1.8$ Hz, 1H), 8.31 (brs, 1H), 8.06 (d, $J = 8.9$ Hz, 1H), 7.80 – 7.76 (m, 2H), 7.50 (d, $J = 8.9$ Hz, 1H), 7.46 (dd, $J = 8.8, 1.8$ Hz, 1H), 7.25 – 7.21 (m, 1H), 7.14 – 7.10 (m, 1H), 7.02 (d, $J = 8.8$ Hz, 1H), 6.80 (d, $J = 8.2$ Hz, 1H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 156.82, 152.06, 136.28, 134.42, 132.52, 130.20, 128.73, 128.38, 127.51, 127.26, 127.18, 126.56, 125.50, 125.20, 123.86, 122.66, 120.46, 119.78, 115.21, 114.34, 104.33, 17.56. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{22}H_{14}NO_2]^-$: 324.1030, found: 324.1026.



3,7'-Dimethyl-[1,1'-binaphthalene]-2,2'-diol (**3z**)

Following **GPA**, **3z** was obtained as an off-white solid in 73% yield.

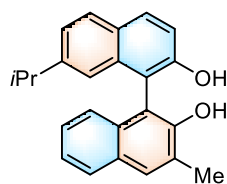
1H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, $J = 8.9$ Hz, 1H), 7.79 – 7.72 (m, 3H), 7.33 – 7.29 (m, 1H), 7.26 – 7.16 (m, 3H), 7.07 (d, $J = 8.8$ Hz, 1H), 6.89 (s, 1H), 5.12 (s, 1H), 4.95 (s, 1H), 2.50 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.99, 152.17, 137.53, 133.79, 132.29, 131.18, 130.79, 129.54, 128.36, 127.78, 127.66, 127.19, 126.52, 126.40, 124.17, 124.03, 123.29, 116.80, 110.62, 110.51, 22.01, 17.17. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{22}H_{17}O_2]^-$: 313.1234, found: 313.1231.



7'-Bromo-3-methyl-[1,1'-binaphthalene]-2,2'-diol (**3aa**)

Following **GPA**, **3aa** was obtained as a white solid in 60% yield.

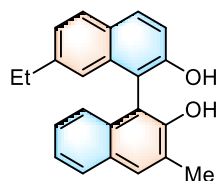
^1H NMR (400 MHz, CDCl_3) δ 7.90 (d, J = 8.9 Hz, 1H), 7.82 – 7.79 (m, 2H), 7.72 (d, J = 8.7 Hz, 1H), 7.43 (dd, J = 8.6, 2.0 Hz, 1H), 7.36 – 7.32 (m, 2H), 7.27 – 7.22 (m, 2H), 7.01 (d, J = 8.4 Hz, 1H), 5.09 (s, 1H), 5.03 (s, 1H), 2.51 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 153.74, 152.20, 135.01, 132.09, 131.41, 131.30, 130.15, 129.61, 128.02, 127.82, 127.66, 127.23, 126.76, 126.41, 124.24, 123.83, 122.30, 118.29, 110.80, 109.47, 17.13. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{21}\text{H}_{14}\text{BrO}_2]^-$: 377.0183, found: 377.0178.



7'-Isopropyl-3-methyl-[1,1'-binaphthalene]-2,2'-diol (**3ab**)

Following **GPA**, **3ab** was obtained as off-white foam in 72% yield.

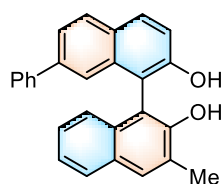
^1H NMR (400 MHz, CDCl_3) δ 7.87 (d, J = 8.9 Hz, 1H), 7.80 – 7.77 (m, 3H), 7.33 – 7.19 (m, 4H), 7.09 (d, J = 8.0 Hz, 1H), 6.93 (s, 1H), 5.14 (s, 1H), 4.96 (s, 1H), 2.83 – 2.73 (m, 1H), 2.50 (s, 3H), 1.11 (d, J = 4.3 Hz, 3H), 1.09 (d, J = 4.3 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.93, 152.13, 148.30, 133.67, 132.21, 131.12, 130.78, 129.53, 128.60, 128.19, 127.65, 127.16, 126.44, 124.27, 124.00, 123.23, 121.18, 116.94, 110.98, 110.49, 34.42, 23.96, 23.93, 17.18. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{24}\text{H}_{21}\text{O}_2]^-$: 341.1547, found: 341.1542.



7'-Ethyl-3-methyl-[1,1'-binaphthalene]-2,2'-diol (**3ac**)

Following **GPA**, **3ac** was obtained as a white solid in 73% yield.

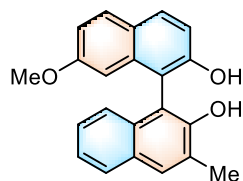
^1H NMR (400 MHz, CDCl_3) δ 7.86 (d, J = 8.9 Hz, 1H), 7.79 – 7.75 (m, 3H), 7.33 – 7.26 (m, 2H), 7.24 – 7.19 (m, 2H), 7.08 (d, J = 8.4 Hz, 1H), 6.91 (s, 1H), 5.13 (s, 1H), 4.96 (s, 1H), 2.56 – 2.50 (m, 5H), 1.08 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.95, 152.15, 143.84, 133.77, 132.25, 131.16, 130.77, 129.53, 128.52, 128.01, 127.65, 127.18, 126.48, 125.10, 124.22, 124.01, 122.29, 116.88, 110.81, 110.51, 29.29, 17.17, 15.75. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{23}\text{H}_{19}\text{O}_2]^-$: 327.1391, found: 327.1387.



3-Methyl-7'-phenyl-[1,1'-binaphthalene]-2,2'-diol (**3ad**)

Following **GPA**, **3ad** was obtained as an off-white solid in 63% yield.

^1H NMR (400 MHz, CDCl_3) δ 7.93 – 7.89 (m, 2H), 7.78 – 7.76 (m, 2H), 7.59 (dd, J = 8.5, 1.8 Hz, 1H), 7.39 – 7.36 (m, 2H), 7.33 – 7.19 (m, 7H), 7.11 (d, J = 8.4 Hz, 1H), 5.15 (s, 1H), 5.04 (s, 1H), 2.49 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 153.29, 152.19, 141.16, 140.38, 133.89, 132.23, 131.17, 131.00, 129.58, 129.07, 128.77, 128.75, 127.72, 127.62, 127.46, 127.18, 126.58, 124.11, 124.09, 124.00, 122.28, 117.87, 111.60, 110.17, 17.17. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{27}\text{H}_{19}\text{O}_2]^-$: 375.1391, found: 375.1386.

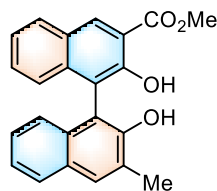


7'-Methoxy-3-methyl-[1,1'-binaphthalene]-2,2'-diol (**3ae**)

Following **GPA**, **3ae** was obtained as an off-white solid in 69% yield.

^1H NMR (400 MHz, CDCl_3) δ 7.82 – 7.71 (m, 4H), 7.33 – 7.29 (m, 1H), 7.24 – 7.20 (m, 1H), 7.16 (d, J = 8.9 Hz, 1H), 7.10 (d, J = 8.4 Hz, 1H), 6.98 (dd, J = 8.9, 2.5 Hz, 1H), 6.41 (d, J = 2.5 Hz, 1H), 5.18 (s, 1H), 5.00 (s, 1H), 3.50 (s, 3H), 2.48 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 159.12, 153.48, 152.09, 135.05, 132.08, 131.13, 130.85, 130.07, 129.57, 127.67, 127.13, 126.51, 124.86, 124.13, 124.03,

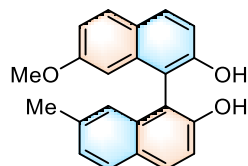
115.96, 115.22, 110.45, 110.38, 103.54, 55.22, 17.13. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{22}H_{17}O_3]^-$: 329.1183, found: 329.1180.



Methyl 2,2'-dihydroxy-3'-methyl-[1,1'-binaphthalene]-3-carboxylate (3af)

Following **GPA**, DIPEA (0.6 mmol) was used and **3af** was obtained as an off-white solid in 29% yield.

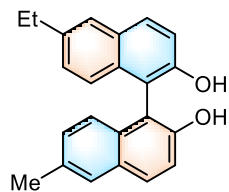
1H NMR (400 MHz, $CDCl_3$) δ 10.83 (s, 1H), 8.73 (s, 1H), 7.94 – 7.91 (m, 1H), 7.80 – 7.75 (m, 2H), 7.39 – 7.34 (m, 2H), 7.30 – 7.26 (m, 1H), 7.18 – 7.14 (m, 2H), 6.99 (d, J = 8.4 Hz, 1H), 5.01 (s, 1H), 4.06 (s, 3H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.46, 155.10, 150.79, 137.63, 134.01, 132.35, 130.28, 129.98, 129.89, 129.38, 127.64, 127.51, 126.84, 125.74, 124.97, 124.66, 124.43, 123.47, 114.88, 114.49, 113.49, 53.03, 17.21. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{23}H_{17}O_4]^-$: 357.1132, found: 357.1129.



7-Methoxy-7'-methyl-[1,1'-binaphthalene]-2,2'-diol (3ag)

Following **GPA**, **3ag** was obtained as an off-white solid in 45% yield.

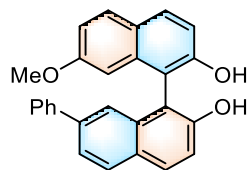
1H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.83 (m, 2H), 7.76 – 7.73 (m, 2H), 7.27 (d, J = 8.9 Hz, 1H), 7.20 – 7.17 (m, 2H), 7.02 – 6.99 (m, 1H), 6.94 (s, 1H), 6.44 (d, J = 2.5 Hz, 1H), 5.06 (s, 1H), 5.02 (s, 1H), 3.53 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.19, 153.49, 152.93, 137.57, 135.01, 133.55, 131.22, 131.10, 130.09, 128.37, 127.82, 126.41, 124.86, 123.21, 116.83, 116.12, 115.26, 110.45, 110.30, 103.34, 55.24, 22.04. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{22}H_{17}O_3]^-$: 329.1183, found: 329.1179.



6-Ethyl-6'-methyl-[1,1'-binaphthalene]-2,2'-diol (**3ah**)

Following **GPA**, **3ah** was obtained as an off-white solid in 64% yield.

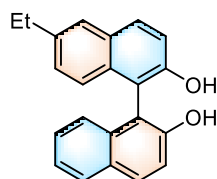
^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.79 (m, 2H), 7.63 – 7.60 (m, 2H), 7.30 (d, J = 3.0 Hz, 1H), 7.28 (d, J = 3.0 Hz, 1H), 7.16 – 7.09 (m, 2H), 7.06 – 7.02 (m, 2H), 5.00 (s, 1H), 4.99 (s, 1H), 2.74 (q, J = 7.6 Hz, 2H), 2.44 (s, 3H), 1.28 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.19, 152.13, 139.98, 133.62, 131.90, 131.66, 130.87, 130.67, 129.75, 129.72, 128.71, 127.55, 126.26, 124.36, 124.28, 117.83, 117.78, 111.10, 111.06, 28.79, 21.44, 15.63. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{23}\text{H}_{19}\text{O}_2]^-$: 327.1391, found: 327.1388.



7-Methoxy-7'-phenyl-[1,1'-binaphthalene]-2,2'-diol (**3ai**)

Following **GPA**, **3ai** was obtained as an off-white solid in 51% yield.

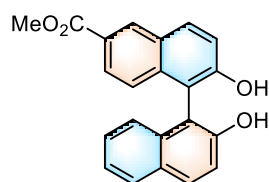
^1H NMR (400 MHz, CDCl_3) δ 7.93 – 7.89 (m, 2H), 7.80 (d, J = 8.9 Hz, 1H), 7.72 (d, J = 8.9 Hz, 1H), 7.61 – 7.59 (m, 1H), 7.42 – 7.40 (m, 2H), 7.36 – 7.28 (m, 4H), 7.26 – 7.24 (m, 1H), 7.17 (d, J = 8.9 Hz, 1H), 7.00 – 6.97 (m, 1H), 6.46 (d, J = 2.2 Hz, 1H), 5.16 (s, 1H), 5.09 (s, 1H), 3.51 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 159.22, 153.52, 153.22, 141.10, 140.38, 134.95, 133.65, 131.27, 131.18, 130.12, 129.07, 128.80, 128.76, 127.59, 127.49, 124.88, 123.96, 122.18, 117.90, 116.11, 115.29, 111.48, 109.98, 103.34, 55.23. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{27}\text{H}_{19}\text{O}_3]^-$: 391.1340, found: 391.1336.



6-Ethyl-[1,1'-binaphthalene]-2,2'-diol (3aj)

Following **GPA**, **3aj** was obtained as an off-white solid in 67% yield.

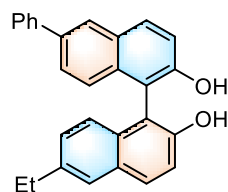
^1H NMR (400 MHz, CDCl_3) δ 7.90 (d, $J = 8.9$ Hz, 1H), 7.86 – 7.83 (m, 2H), 7.64 (s, 1H), 7.35 – 7.24 (m, 4H), 7.16 – 7.12 (m, 2H), 7.05 (d, $J = 8.6$ Hz, 1H), 5.08 (s, 1H), 4.99 (s, 1H), 2.75 (q, $J = 7.6$ Hz, 2H), 1.28 (t, $J = 7.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.82, 152.23, 140.04, 133.56, 131.88, 131.38, 130.98, 129.78, 129.52, 128.76, 128.47, 127.54, 126.30, 124.39, 124.32, 124.10, 117.86, 117.81, 111.24, 110.87, 28.79, 15.63. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{22}\text{H}_{17}\text{O}_2]^-$: 313.1234, found: 313.1231.



Methyl 2,2'-dihydroxy-[1,1'-binaphthalene]-6-carboxylate (3ak)

Following **GPA**, **3ak** was obtained as an off-white solid in 60% yield.

^1H NMR (400 MHz, $\text{Acetone-}d_6$) δ 8.62 (d, $J = 1.8$ Hz, 1H), 8.35 (s, 1H), 8.12 – 8.10 (m, 2H), 7.94 (d, $J = 8.9$ Hz, 1H), 7.89 (d, $J = 7.3$ Hz, 1H), 7.79 (dd, $J = 8.9, 1.8$ Hz, 1H), 7.46 (d, $J = 8.9$ Hz, 1H), 7.38 (d, $J = 8.8$ Hz, 1H), 7.32 – 7.27 (m, 1H), 7.25 – 7.21 (m, 1H), 7.16 (d, $J = 8.8$ Hz, 1H), 7.08 – 7.05 (m, 1H), 3.89 (s, 3H). ^{13}C NMR (100 MHz, $\text{Acetone-}d_6$) δ 167.51, 156.79, 154.61, 137.98, 135.33, 132.12, 131.87, 130.84, 129.95, 128.97, 128.88, 127.23, 126.28, 125.72, 125.40, 125.15, 123.75, 120.51, 119.52, 115.60, 114.37, 52.19. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{22}\text{H}_{15}\text{O}_4]^-$: 343.0976, found: 343.0970.

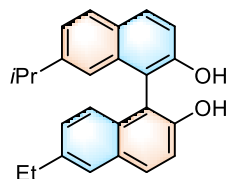


6-Ethyl-6'-phenyl-[1,1'-binaphthalene]-2,2'-diol (3al)

Following **GPA**, **3al** was obtained as an off-white solid in 47% yield.

^1H NMR (400 MHz, CDCl_3) δ 8.05 (d, $J = 1.9$ Hz, 1H), 7.95 (d, $J = 8.9$ Hz, 1H), 7.86 (d, $J = 9.0$ Hz, 1H), 7.65 – 7.62 (m, 3H), 7.53 (dd, $J = 8.7, 1.9$ Hz, 1H), 7.45 – 7.41 (m, 2H), 7.36 – 7.30 (m,

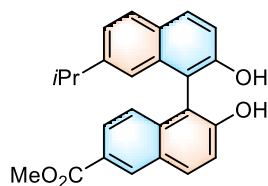
3H), 7.22 – 7.15 (m, 2H), 7.09 (d, $J = 8.6$ Hz, 1H), 5.12 (s, 1H), 5.04 (s, 1H), 2.75 (q, $J = 7.6$ Hz, 2H), 1.28 (t, $J = 7.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.91, 152.24, 140.95, 140.09, 136.94, 132.75, 131.87, 131.65, 131.04, 129.79, 128.98, 128.82, 127.37, 127.35, 127.18, 126.41, 126.33, 124.99, 124.32, 118.32, 117.84, 111.26, 110.82, 28.79, 15.63. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{28}\text{H}_{21}\text{O}_2]^-$: 389.1547, found: 389.1542.



6-Ethyl-7'-isopropyl-[1,1'-binaphthalene]-2,2'-diol (**3am**)

Following **GPA**, **3am** was obtained as an off-white solid in 60% yield.

^1H NMR (400 MHz, CDCl_3) δ 7.94 – 7.92 (m, 2H), 7.86 (d, $J = 8.5$ Hz, 1H), 7.72 (s, 1H), 7.40 – 7.32 (m, 3H), 7.25 – 7.16 (m, 2H), 7.02 (s, 1H), 5.08 (s, 2H), 2.87 – 2.80 (m, 3H), 1.36 (t, $J = 7.5$ Hz, 3H), 1.19 (d, $J = 2.7$ Hz, 3H), 1.17 (d, $J = 2.7$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.90, 152.22, 148.34, 139.97, 133.61, 131.78, 131.06, 130.87, 129.79, 128.68, 128.60, 128.15, 126.26, 124.45, 123.23, 121.12, 117.83, 116.97, 111.04, 110.94, 34.46, 28.80, 24.02, 23.88, 15.61. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{25}\text{H}_{23}\text{O}_2]^-$: 355.1704, found: 355.1700.

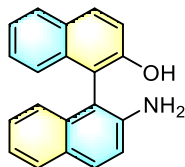


Methyl 2,2'-dihydroxy-7'-isopropyl-[1,1'-binaphthalene]-6-carboxylate (**3an**)

Following **GPA**, **3an** was obtained as an off-white solid in 42% yield.

^1H NMR (400 MHz, $\text{Acetone-}d_6$) δ 8.62 (d, $J = 1.8$ Hz, 1H), 8.28 (s, 1H), 8.11 (d, $J = 8.9$ Hz, 1H), 8.01 (s, 1H), 7.88 (d, $J = 8.9$ Hz, 1H), 7.83 (d, $J = 8.4$ Hz, 1H), 7.80 – 7.77 (m, 1H), 7.45 (d, $J = 8.9$ Hz, 1H), 7.31 – 7.24 (m, 2H), 7.17 (d, $J = 8.8$ Hz, 1H), 6.92 (s, 1H), 3.89 (s, 3H), 2.79 – 2.69 (m, 1H), 1.07 (d, $J = 2.9$ Hz, 3H), 1.05 (d, $J = 2.9$ Hz, 3H). ^{13}C NMR (100 MHz, $\text{Acetone-}d_6$) δ 167.53, 156.75, 154.70, 147.62, 137.95, 135.46, 132.07, 131.88, 130.53, 129.13, 128.88, 128.63,

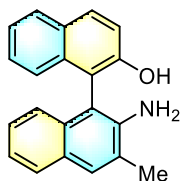
126.16, 125.83, 125.37, 122.98, 121.86, 120.50, 118.66, 115.71, 114.06, 52.18, 35.04, 24.16, 24.13. HRMS (ESI) $[M-H]^-$ calculated m/z for $[C_{25}H_{21}O_4]^-$: 385.1445, found: 385.1441.



2'-Amino-[1,1'-binaphthalen]-2-ol (**5a**)

Following **GPB**, **5a** was obtained as a white solid in 62% yield.

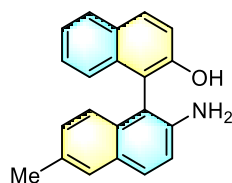
1H NMR (400 MHz, DMSO- d_6) δ 9.32 (s, 1H), 7.91 – 7.86 (m, 2H), 7.74 (d, J = 9.0 Hz, 2H), 7.38 (d, J = 8.9 Hz, 1H), 7.27 – 7.16 (m, 3H), 7.11 – 7.04 (m, 2H), 6.97 – 6.95 (m, 1H), 6.79 – 6.76 (m, 1H), 4.56 (s, 2H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 153.34, 143.97, 134.06, 133.68, 129.15, 128.50, 128.16, 128.09, 127.83, 127.05, 126.17, 125.73, 124.17, 123.49, 122.58, 120.80, 118.83, 118.48, 114.97, 111.29. HRMS (ESI) $[M+H]^+$ calculated m/z for $[C_{20}H_{16}NO]^+$: 286.1226, found: 286.1224.



2'-Amino-3'-methyl-[1,1'-binaphthalen]-2-ol (**5b**)

Following **GPB**, **5b** was obtained as an off-white solid in 62% yield.

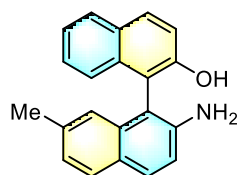
1H NMR (400 MHz, DMSO- d_6) δ 9.34 (s, 1H), 7.93 – 7.87 (m, 2H), 7.70 (d, J = 8.1 Hz, 1H), 7.65 (s, 1H), 7.40 (d, J = 8.9 Hz, 1H), 7.28 – 7.16 (m, 2H), 7.11 – 7.00 (m, 2H), 6.92 (d, J = 8.4 Hz, 1H), 6.74 (d, J = 8.3 Hz, 1H), 4.26 (s, 2H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 153.39, 142.85, 133.72, 132.73, 129.24, 128.49, 128.09, 127.79, 127.17, 127.06, 126.24, 125.37, 124.84, 124.15, 123.47, 122.62, 121.03, 118.84, 115.15, 111.87, 18.62. HRMS (ESI) $[M+H]^+$ calculated m/z for $[C_{21}H_{18}NO]^+$: 300.1383, found: 300.1380.



2'-Amino-6'-methyl-[1,1'-binaphthalen]-2-ol (**5c**)

Following **GPB**, MeCN (2 mL) and acetone (2 mL) were used and **5c** was obtained as an off-white solid in 52% yield.

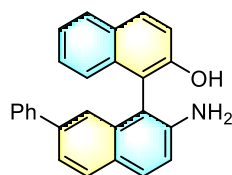
^1H NMR (400 MHz, DMSO- d_6) δ 9.30 (s, 1H), 7.90 – 7.85 (m, 2H), 7.65 (d, J = 8.7 Hz, 1H), 7.52 (s, 1H), 7.39 (d, J = 8.8 Hz, 1H), 7.27 – 7.16 (m, 3H), 6.98 – 6.92 (m, 2H), 6.72 (d, J = 8.5 Hz, 1H), 4.45 (s, 2H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 153.29, 143.19, 133.71, 132.27, 129.54, 129.08, 128.47, 128.05, 127.81, 127.48, 127.25, 126.81, 126.11, 124.24, 123.62, 122.55, 118.82, 118.55, 115.14, 111.52, 20.82. HRMS (ESI) $[\text{M}+\text{H}]^+$ calculated m/z for $[\text{C}_{21}\text{H}_{18}\text{NO}]^+$: 300.1383, found: 300.1382.



2'-Amino-7'-methyl-[1,1'-binaphthalen]-2-ol (**5d**)

Following **GPB**, **5d** was obtained as an off-white solid in 57% yield.

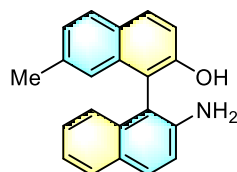
^1H NMR (400 MHz, DMSO- d_6) δ 9.28 (s, 1H), 7.90 – 7.85 (m, 2H), 7.69 – 7.63 (m, 2H), 7.39 (d, J = 8.8 Hz, 1H), 7.28 – 7.17 (m, 2H), 7.11 (d, J = 8.7 Hz, 1H), 6.99 – 6.92 (m, 2H), 6.58 (s, 1H), 4.47 (s, 2H), 2.12 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 153.30, 143.98, 134.57, 134.26, 133.68, 129.11, 128.50, 128.11, 127.94, 127.83, 126.17, 125.37, 124.19, 122.99, 122.59, 122.41, 118.88, 117.53, 115.13, 110.88, 21.64. HRMS (ESI) $[\text{M}+\text{H}]^+$ calculated m/z for $[\text{C}_{21}\text{H}_{18}\text{NO}]^+$: 300.1383, found: 300.1380.



2'-Amino-7'-phenyl-[1,1'-binaphthalen]-2-ol (**5e**)

Following **GPB**, **5e** was obtained as a white solid in 50% yield.

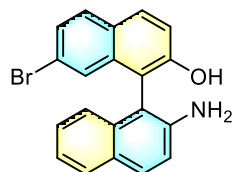
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 9.37 (s, 1H), 7.92 – 7.83 (m, 3H), 7.78 (d, J = 8.8 Hz, 1H), 7.40 (d, J = 8.7 Hz, 2H), 7.31 – 7.20 (m, 8H), 7.04 – 6.99 (m, 2H), 4.62 (s, 2H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 153.39, 144.44, 140.99, 137.50, 134.23, 133.57, 129.30, 128.83, 128.64, 128.45, 128.15, 127.95, 127.03, 126.51, 126.31, 126.23, 124.14, 122.63, 121.16, 120.08, 118.85, 118.69, 114.79, 111.50. HRMS (ESI) $[\text{M}+\text{H}]^+$ calculated m/z for $[\text{C}_{26}\text{H}_{20}\text{NO}]^+$: 362.1539, found: 362.1536.



2'-Amino-7-methyl-[1,1'-binaphthalen]-2-ol (**5f**)

Following **GPB**, MeCN (2 mL) and acetone (2 mL) were used and **5f** was obtained as an off-white solid in 60% yield.

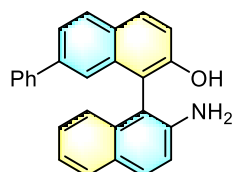
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 9.21 (s, 1H), 7.83 (d, J = 8.9 Hz, 1H), 7.78 – 7.72 (m, 3H), 7.30 (d, J = 8.9 Hz, 1H), 7.19 (d, J = 8.8 Hz, 1H), 7.11 – 7.07 (m, 3H), 6.81 – 6.76 (m, 2H), 4.52 (s, 2H), 2.17 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 153.43, 143.87, 135.18, 134.06, 133.87, 128.88, 128.09, 127.82, 127.06, 126.75, 125.68, 124.75, 123.53, 122.89, 120.79, 118.48, 117.82, 114.39, 111.51, 21.61. HRMS (ESI) $[\text{M}+\text{H}]^+$ calculated m/z for $[\text{C}_{21}\text{H}_{18}\text{NO}]^+$: 300.1383, found: 300.1381.



2'-Amino-7-bromo-[1,1'-binaphthalen]-2-ol (**5g**)

Following **GPB**, DABCO (0.3 mmol) and Li_2CO_3 (0.3 mmol) were used and **5g** was obtained as an off-white solid in 57% yield.

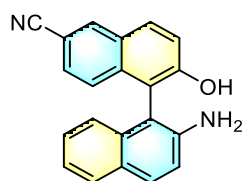
^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 9.60 (s, 1H), 7.93 (d, J = 8.9 Hz, 1H), 7.86 (d, J = 8.7 Hz, 1H), 7.77 – 7.73 (m, 2H), 7.43 – 7.36 (m, 2H), 7.21 (d, J = 8.8 Hz, 1H), 7.12 – 7.07 (m, 3H), 6.78 – 6.74 (m, 1H), 4.67 (s, 2H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 154.54, 144.10, 135.16, 133.90, 130.56, 129.36, 128.53, 127.95, 127.05, 126.97, 126.01, 125.68, 125.41, 123.15, 120.93, 120.07, 119.52, 118.54, 114.43, 110.19. HRMS (ESI) $[\text{M}+\text{H}]^+$ calculated m/z for $[\text{C}_{20}\text{H}_{15}\text{BrNO}]^+$: 364.0332, found: 364.0329.



2'-Amino-7-phenyl-[1,1'-binaphthalen]-2-ol (**5h**)

Following **GPB**, **5h** was obtained as an off-white solid in 64% yield.

^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 9.42 (s, 1H), 8.00 – 7.94 (m, 2H), 7.77 – 7.73 (m, 2H), 7.57 (dd, J = 8.5, 1.9 Hz, 1H), 7.43 – 7.30 (m, 5H), 7.25 – 7.22 (m, 3H), 7.11 – 7.09 (m, 2H), 6.89 – 6.86 (m, 1H), 4.65 (s, 2H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 153.88, 144.04, 140.66, 137.98, 134.04, 133.88, 128.97, 128.90, 128.34, 127.90, 127.80, 127.26, 127.05, 126.65, 125.80, 123.51, 121.95, 121.80, 120.88, 119.07, 118.54, 115.38, 111.12. HRMS (ESI) $[\text{M}+\text{H}]^+$ calculated m/z for $[\text{C}_{26}\text{H}_{20}\text{NO}]^+$: 362.1539, found: 362.1537.

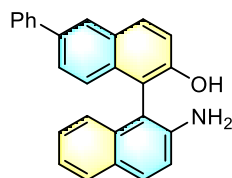


2'-Amino-2-hydroxy-[1,1'-binaphthalene]-6-carbonitrile (**5i**)

Following **GPB**, **5i** was obtained as an off-white solid in 62% yield.

^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 9.97 (s, 1H), 8.51 (d, J = 1.7 Hz, 1H), 8.06 (d, J = 8.9 Hz, 1H), 7.76 – 7.72 (m, 2H), 7.53 – 7.45 (m, 2H), 7.19 (d, J = 8.8 Hz, 1H), 7.11 – 7.05 (m, 3H), 6.72 – 6.69 (m, 1H), 4.69 (s, 2H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 156.57, 144.24, 135.69, 134.53, 133.91, 130.17, 128.58, 127.94, 127.49, 126.97, 126.67, 126.02, 125.51, 123.03, 120.88, 120.61,

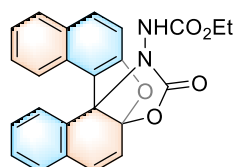
119.73, 118.56, 115.76, 109.73, 104.57. HRMS (ESI) $[M+H]^+$ calculated m/z for $[C_{21}H_{15}N_2O]^+$: 311.1179, found: 311.1177.



2'-Amino-6-phenyl-[1,1'-binaphthalen]-2-ol (5j)

Following **GPB**, **5j** was obtained as an off-white solid in 61% yield.

1H NMR (400 MHz, DMSO- d_6) δ 9.39 (s, 1H), 8.18 (d, $J = 2.0$ Hz, 1H), 7.99 (d, $J = 8.9$ Hz, 1H), 7.76 – 7.71 (m, 4H), 7.55 (dd, $J = 8.8, 2.0$ Hz, 1H), 7.48 – 7.41 (m, 3H), 7.36 – 7.32 (m, 1H), 7.21 (d, $J = 8.8$ Hz, 1H), 7.11 – 7.04 (m, 3H), 6.84 – 6.81 (m, 1H), 4.61 (s, 2H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 153.64, 144.02, 140.32, 134.37, 134.09, 133.03, 129.65, 128.93, 128.80, 128.23, 127.86, 127.06, 127.04, 126.66, 125.80, 125.70, 125.36, 125.00, 123.50, 120.84, 119.32, 118.52, 114.95, 111.17. HRMS (ESI) $[M+H]^+$ calculated m/z for $[C_{26}H_{20}NO]^+$: 362.1539, found: 362.1539.



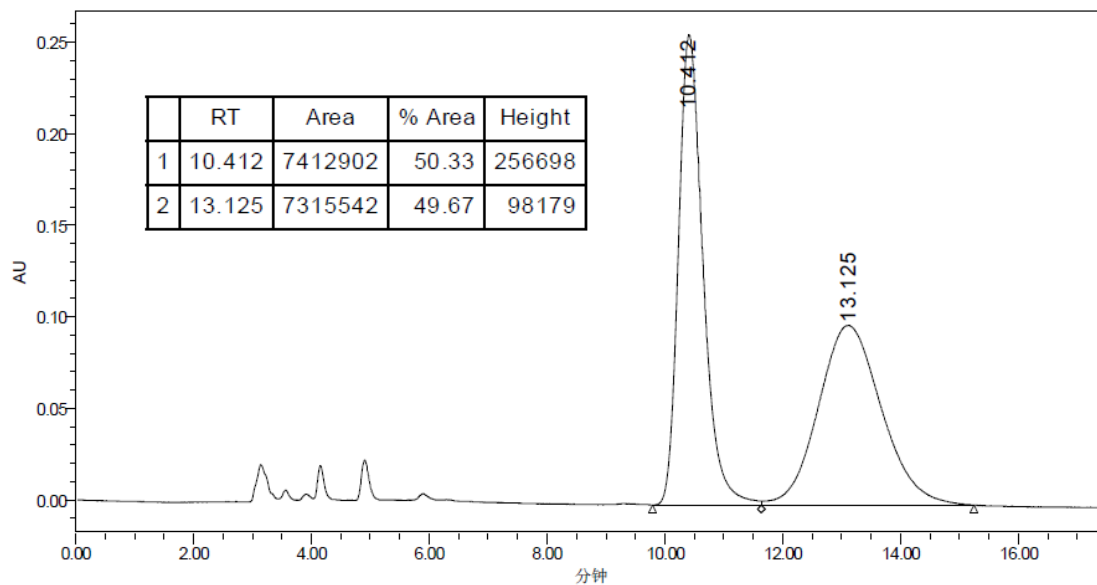
Ethyl (15-oxo-6a,13c-(epoxymethanoimino)dinaphtho[2,1-*b*:1',2'-*d*]furan-14-yl)carbamate (11a)

53% yield, 94% ee

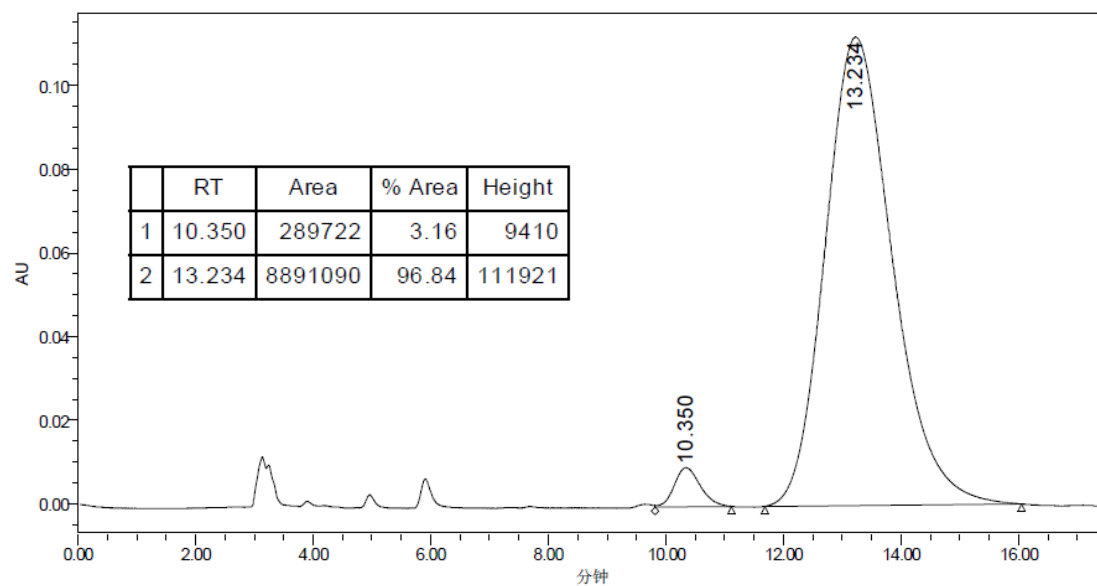
1H NMR (600 MHz, DMSO- d_6 , 90 °C) δ 10.00 (s, 1H), 8.55 – 8.50 (m, 1H), 8.28 – 7.24 (m, 9H), 6.93 – 6.84 (m, 1H), 6.31 – 6.19 (m, 1H), 3.91 – 3.71 (m, 2H), 1.01 – 0.73 (m, 3H). ^{13}C NMR (150 MHz, DMSO- d_6 , 90 °C) δ 156.66, 156.22, 155.15, 154.54, 152.67, 152.13, 132.84, 132.30, 131.19, 131.04, 130.67, 130.33, 130.13, 129.96, 129.41, 129.34, 129.19, 128.97, 128.48, 128.34, 128.18, 127.65, 126.81, 124.85, 123.84, 123.05, 122.24, 119.41, 118.65, 118.47, 116.83, 111.91, 111.66, 110.15, 109.78, 72.26, 71.92, 60.85, 60.62, 13.49, 13.23. HRMS (ESI) $[M+H]^+$ calculated m/z for $[C_{24}H_{19}N_2O_5]^+$: 415.1288, found: 415.1284.

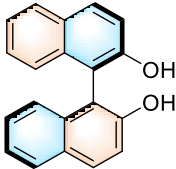
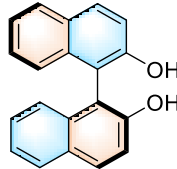
HPLC condition: Chiralpak OZ-3, hexane/*i*-PrOH = 70/30, 1.0 mL/min, $t_R(\text{minor}) = 10.4$ min, $t_R(\text{major}) = 13.2$ min.

HPLC spectrum of racemic **11a**

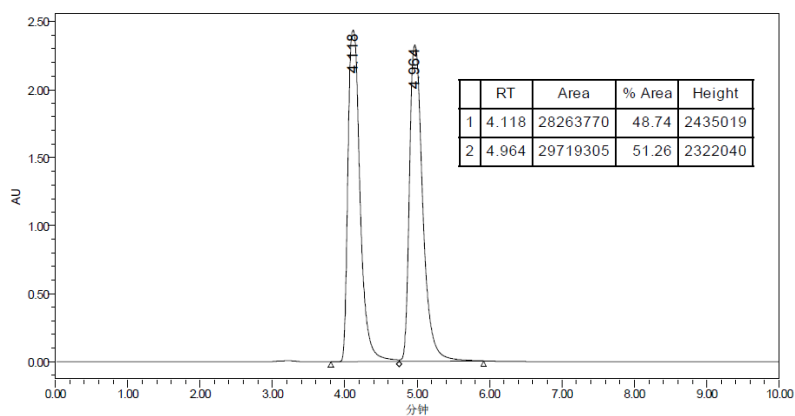


HPLC spectrum of chiral **11a**

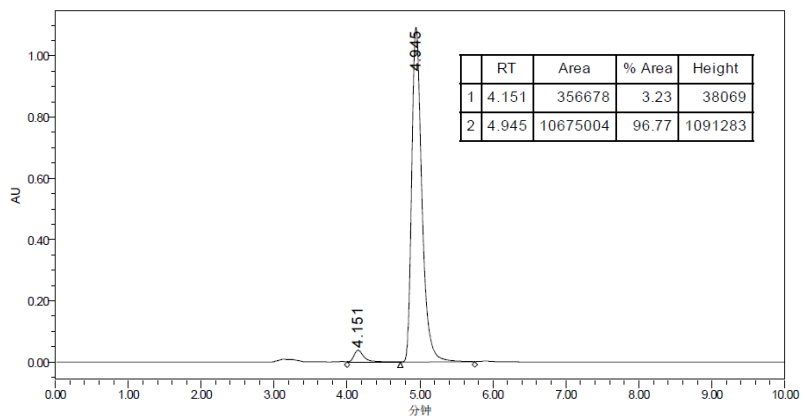


	
(<i>R</i>)- 9a , 40% yield, 94% ee.	(<i>S</i>)- 9a , 53% yield, 92% ee.
HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (minor) = 4.2 min, t_R (major) = 4.9 min.	HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (major) = 4.1 min, t_R (minor) = 5.0 min.

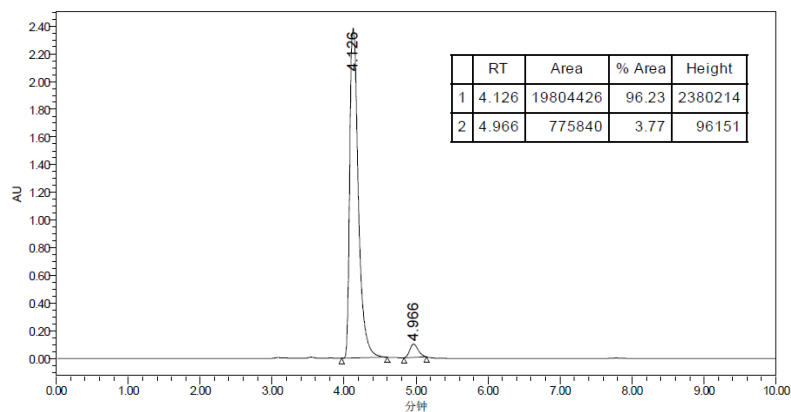
HPLC spectrum of racemic **9a**

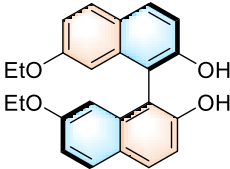
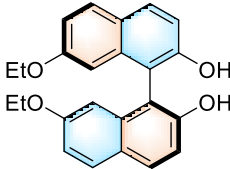


HPLC spectrum of (*R*)-**9a**



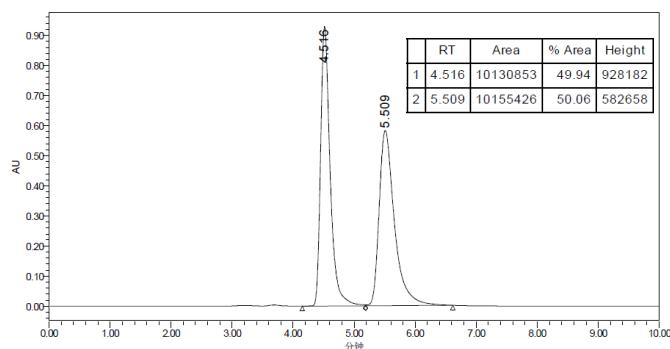
HPLC spectrum of (*S*)-**9a**



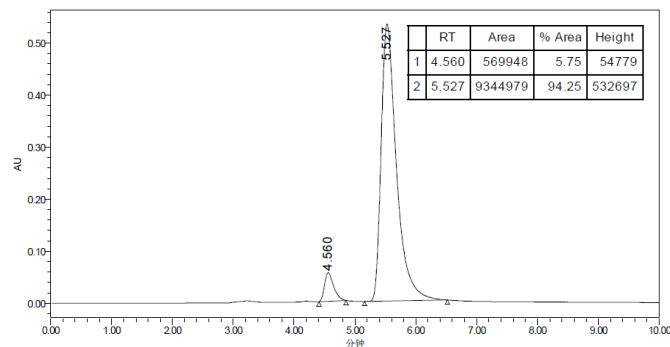
	
(R)- 9b , 38% yield, 89% ee.	(S)- 9b , 42% yield, 93% ee.
HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (minor) = 4.6 min, t_R (major) = 5.5 min.	HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (major) = 4.5 min, t_R (minor) = 5.5 min.

The data are matched with the reported literature.^[6] ^1H NMR (400 MHz, CDCl_3) δ 7.89 (d, J = 8.8 Hz, 2H), 7.80 (d, J = 8.9 Hz, 2H), 7.24 (d, J = 8.8 Hz, 2H), 7.05 (dd, J = 8.9, 2.5 Hz, 2H), 6.50 (d, J = 2.5 Hz, 2H), 5.06 (s, 2H), 3.87 – 3.72 (m, 4H), 1.29 (t, J = 7.0 Hz, 6H).

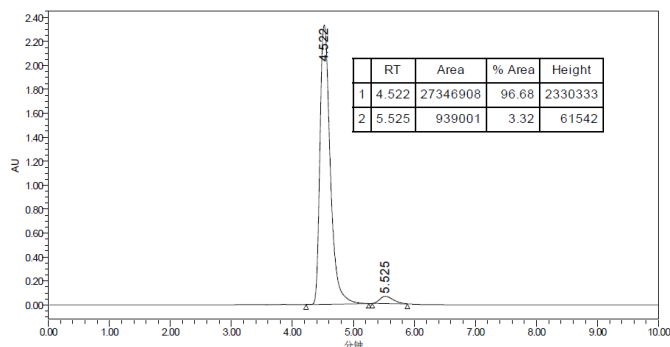
HPLC spectrum of racemic **9b**

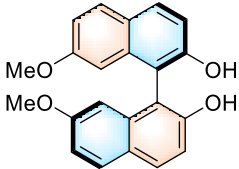
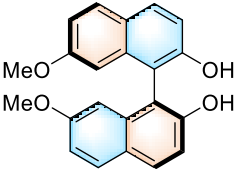


HPLC spectrum of (R)-**9b**



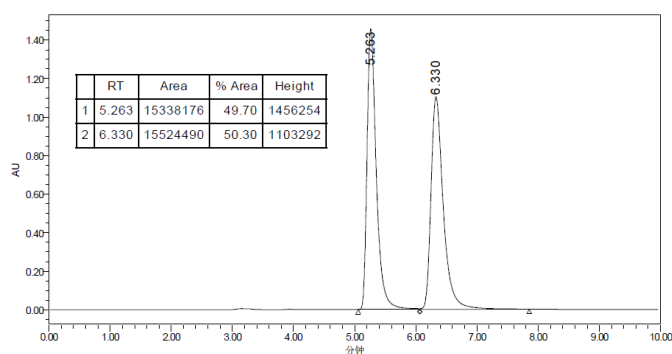
HPLC spectrum of (S)-**9b**



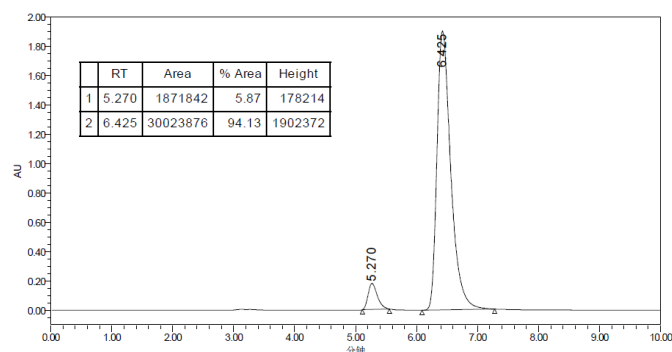
	
(R)- 9c , 41% yield, 88% ee.	(S)- 9c , 45% yield, 92% ee.
HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (minor) = 5.3 min, t_R (major) = 6.4 min.	HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (major) = 5.2 min, t_R (minor) = 6.4 min.

The data matched with the reported literature.^[7] ^1H NMR (400 MHz, CDCl_3) δ 7.89 (d, J = 8.8 Hz, 2H), 7.79 (d, J = 8.9 Hz, 2H), 7.23 (d, J = 8.8 Hz, 2H), 7.04 (dd, J = 8.9, 2.5 Hz, 2H), 6.49 (d, J = 2.5 Hz, 2H), 5.04 (s, 2H), 3.58 (s, 6H).

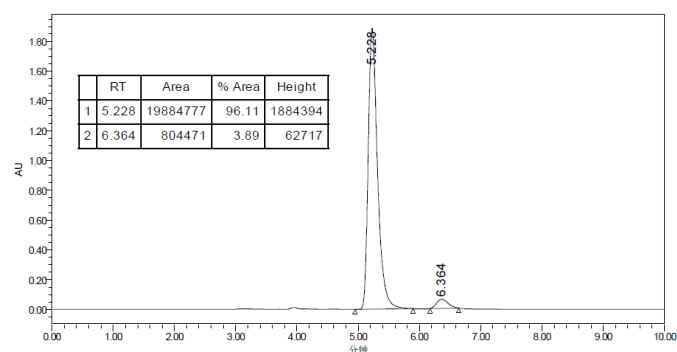
HPLC spectrum of racemic **9c**

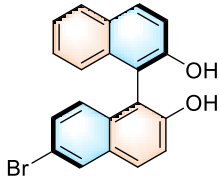
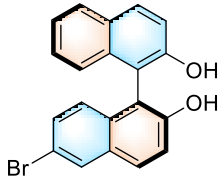


HPLC spectrum of (R)-**9c**

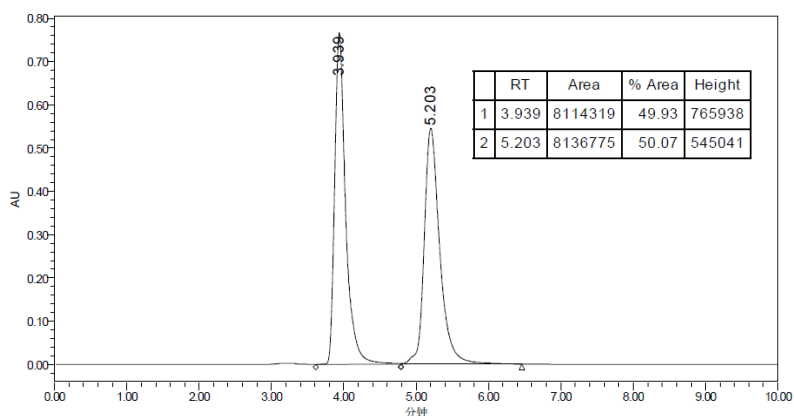


HPLC spectrum of (S)-**9c**

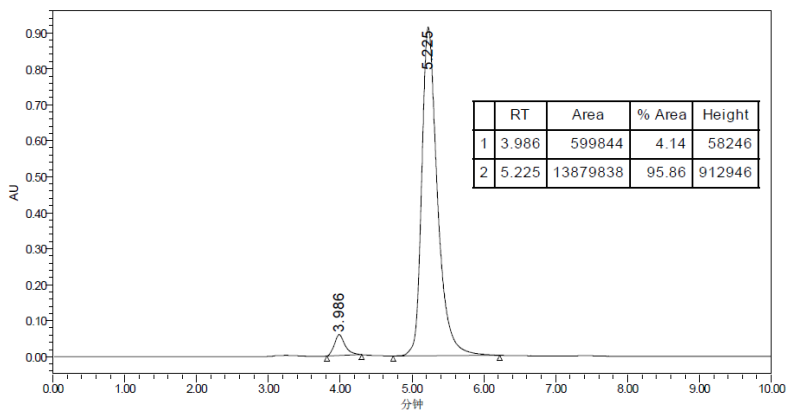


	
(R)- 9d , 47% yield, 92% ee.	(S)- 9d , 42% yield, 92% ee.
HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (minor) = 4.0 min, t_R (major) = 5.2 min.	HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (major) = 3.9 min, t_R (minor) = 5.2 min.

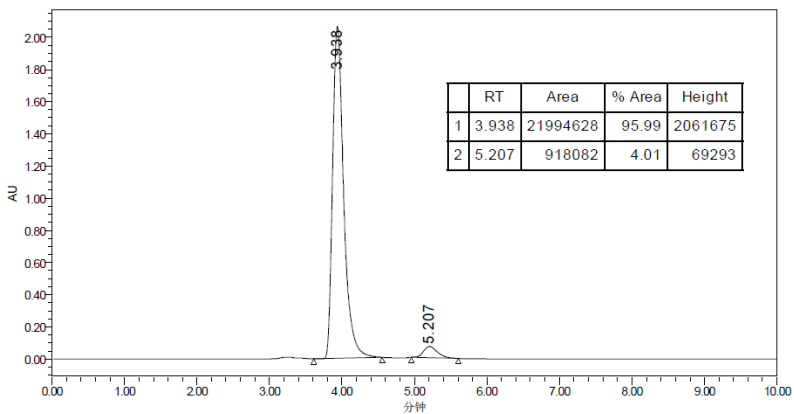
HPLC spectrum of racemic **9d**

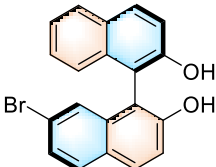
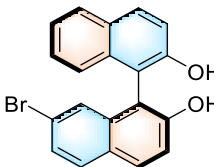


HPLC spectrum of (*R*)-**9d**

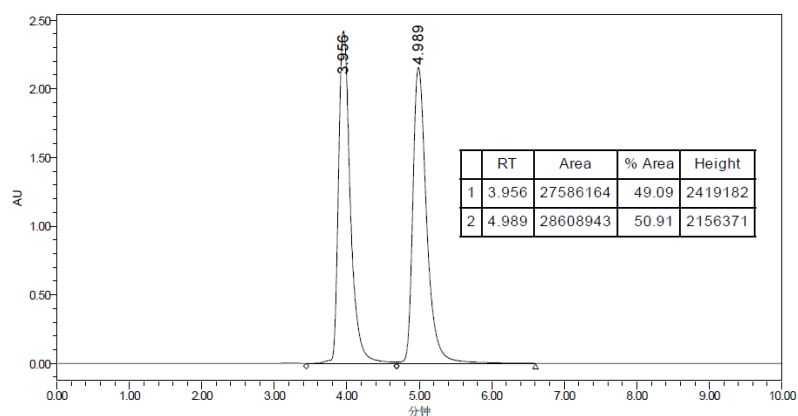


HPLC spectrum of (*S*)-**9d**

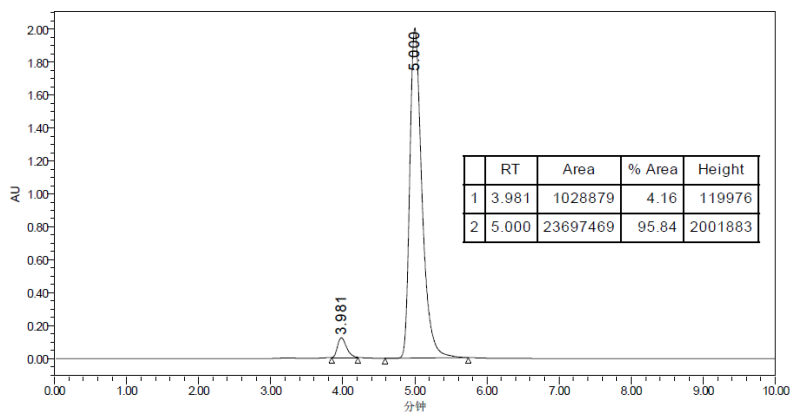


	
(R)- 9e , 42% yield, 92% ee.	(S)- 9e , 49% yield, 84% ee.
HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (minor) = 4.0 min, t_R (major) = 5.0 min.	HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (major) = 4.0 min, t_R (minor) = 5.0 min.

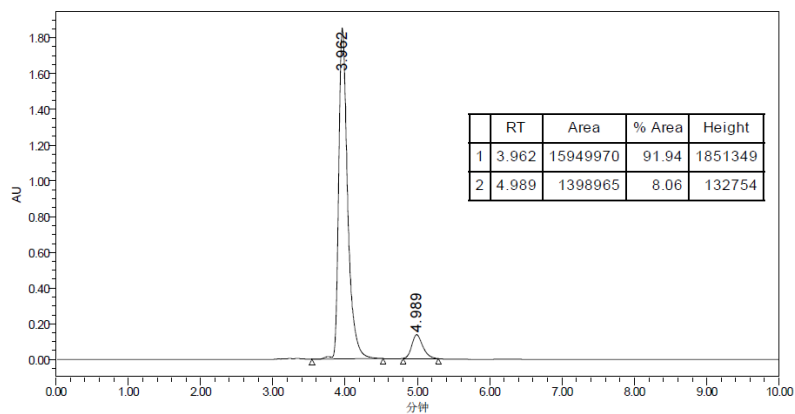
HPLC spectrum of racemic **9e**

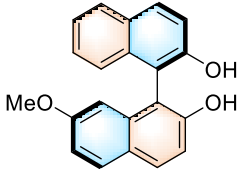
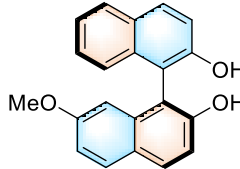


HPLC spectrum of (R)-**9e**

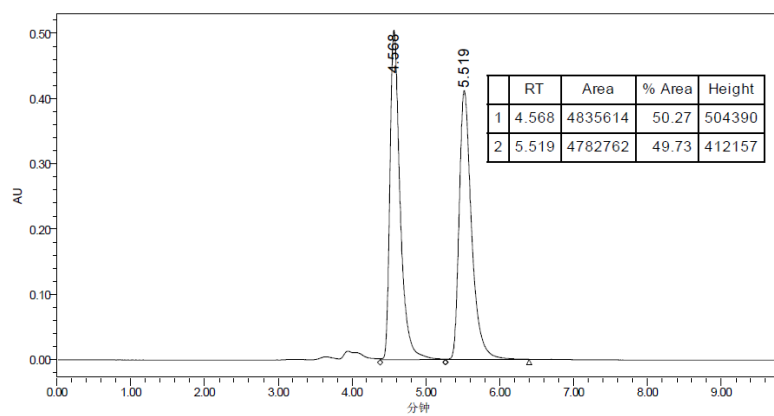


HPLC spectrum of (S)-**9e**

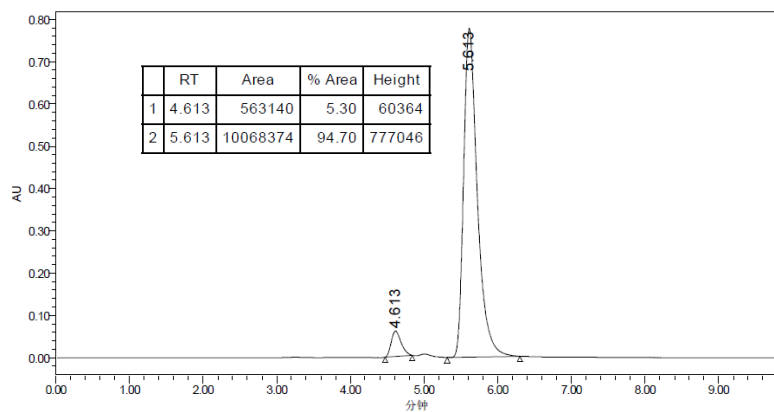


	
<p>(<i>R</i>)-9f, 45% yield, 89% ee.</p>	<p>(<i>S</i>)-9f, 52% yield, 94% ee.</p>
<p>HPLC condition: Chiralpak OZ-3, hexane/<i>i</i>-PrOH = 70/30, 1.0 mL/min, t_R(minor) = 4.6 min, t_R(major) = 5.6 min.</p>	<p>HPLC condition: Chiralpak OZ-3, hexane/<i>i</i>-PrOH = 70/30, 1.0 mL/min, t_R(major) = 4.6 min, t_R(minor) = 5.6 min.</p>

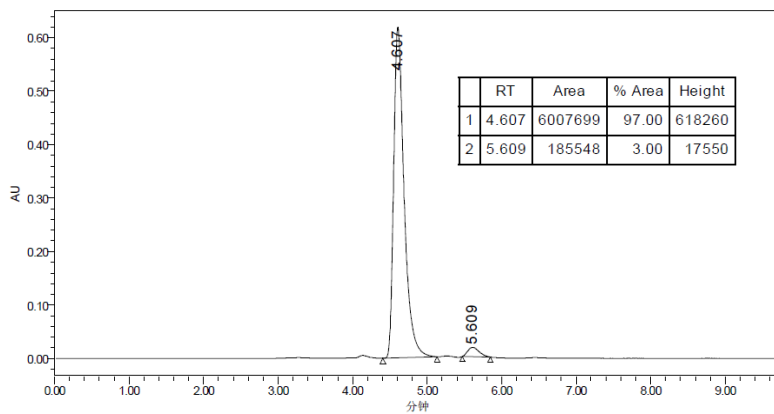
HPLC spectrum of racemic **9f**

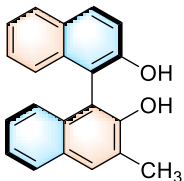
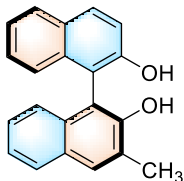


HPLC spectrum of (*R*)-**9f**

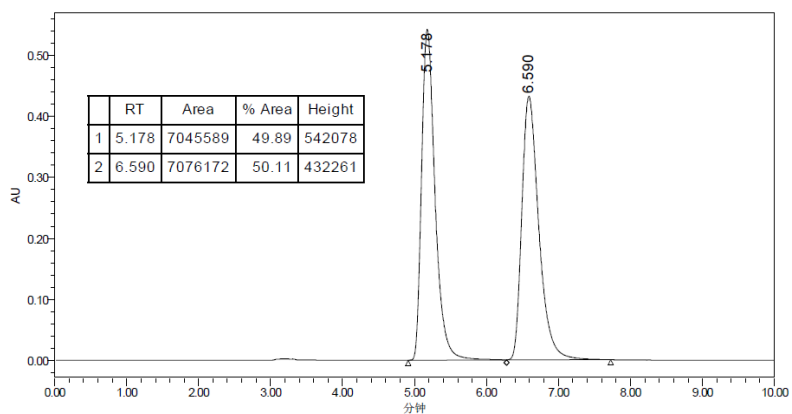


HPLC spectrum of (*S*)-**9f**

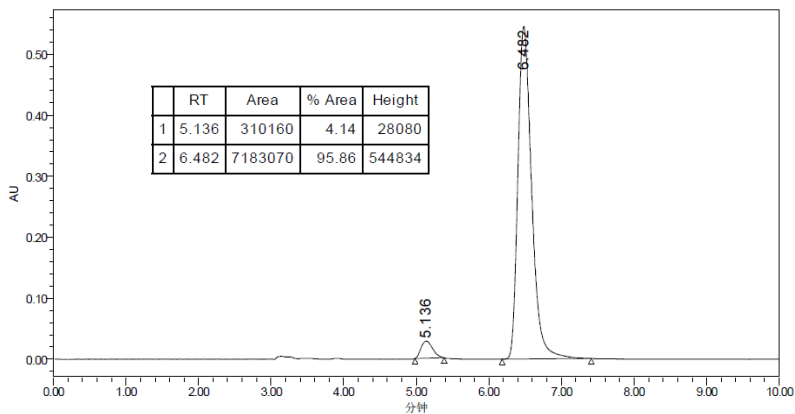


	
(R)- 9g , 33% yield, 92% ee.	(S)- 9g , 52% yield, 90% ee.
HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 90/10, 1.0 mL/min, t_R (minor) = 5.1 min, t_R (major) = 6.5 min.	HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 90/10, 1.0 mL/min, t_R (minor) = 5.2 min, t_R (major) = 6.6 min.

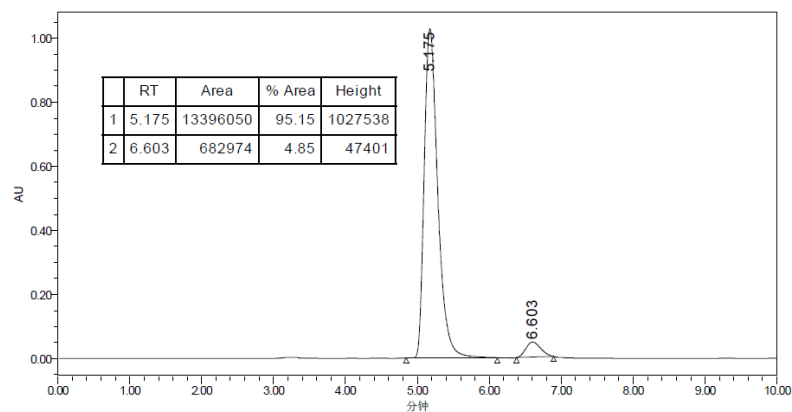
HPLC spectrum of racemic **9g**

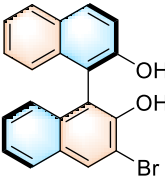
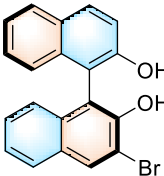


HPLC spectrum of (R)-**9g**



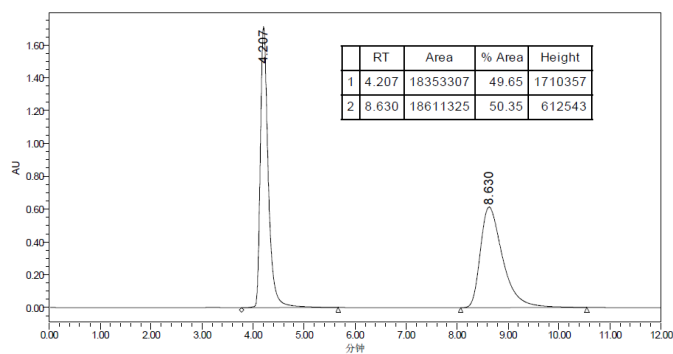
HPLC spectrum of (S)-**9g**



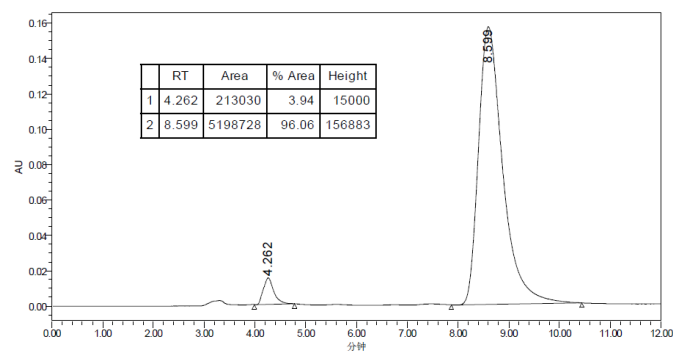
	
(<i>R</i>)- 9h , 44% yield, 92% ee.	(<i>S</i>)- 9h , 48% yield, 89% ee.
HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, <i>t_R</i> (minor) = 4.3 min, <i>t_R</i> (major) = 8.6 min.	HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, <i>t_R</i> (major) = 4.2 min, <i>t_R</i> (minor) = 8.7 min.

¹H NMR (400 MHz, DMSO-*d*₆) δ 9.49 (s, 1H), 8.85 (s, 1H), 8.32 (s, 1H), 7.93 – 7.86 (m, 3H), 7.37 – 7.16 (m, 5H), 6.89 – 6.85 (m, 2H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 153.75, 149.12, 134.05, 133.08, 131.30, 129.65, 128.97, 128.24, 128.07, 127.11, 126.40, 126.30, 124.58, 123.87, 123.61, 122.49, 118.72, 118.02, 113.65, 113.55.

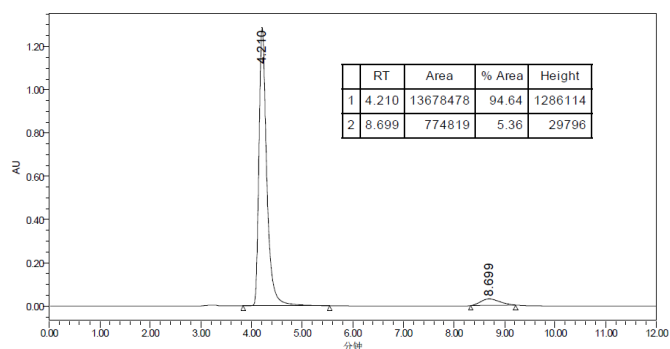
HPLC spectrum of racemic **9h**

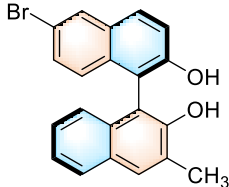
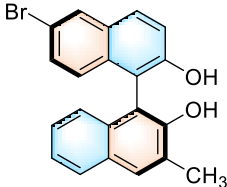


HPLC spectrum of (*R*)-**9h**

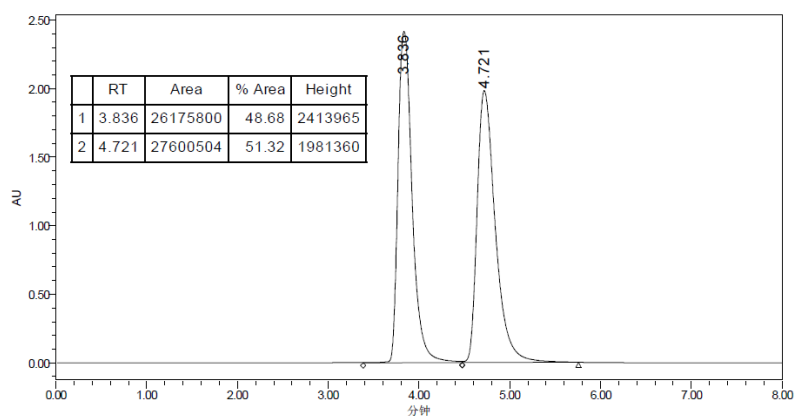


HPLC spectrum of (*S*)-**9h**

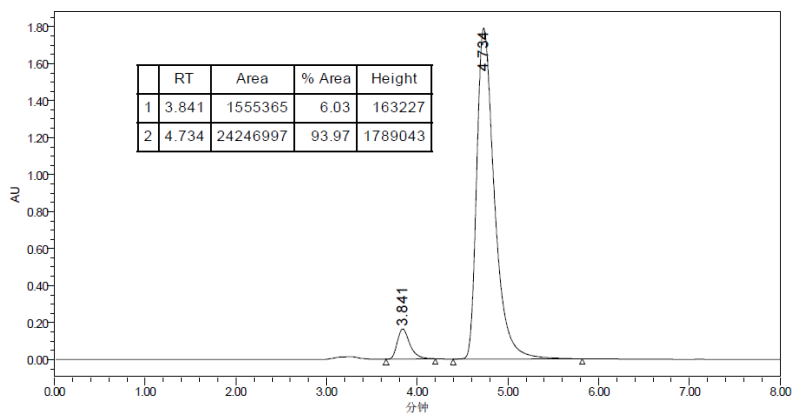


	
(R)- 9i , 38% yield, 88% ee.	(S)- 9i , 45% yield, 91% ee.
HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (minor) = 3.8 min, t_R (major) = 4.7 min.	HPLC condition: Chiralpak OZ-3, hexane/ <i>i</i> -PrOH = 70/30, 1.0 mL/min, t_R (major) = 3.8 min, t_R (minor) = 4.7 min.

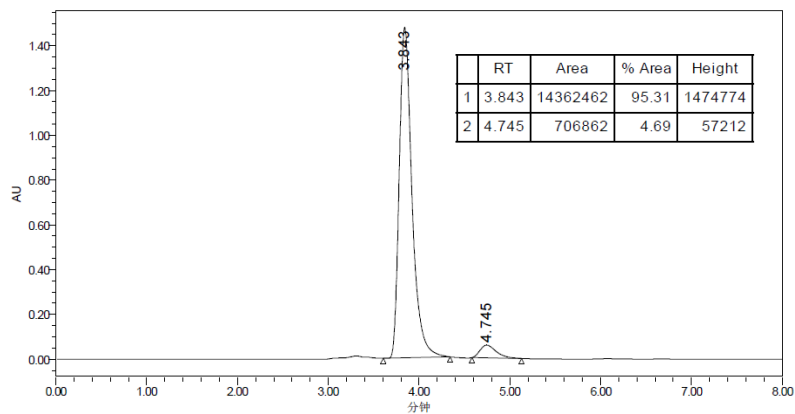
HPLC spectrum of racemic **9i**

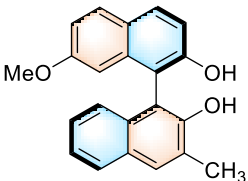
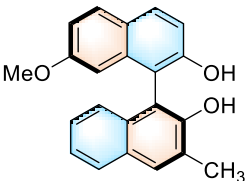


HPLC spectrum of (R)-**9i**

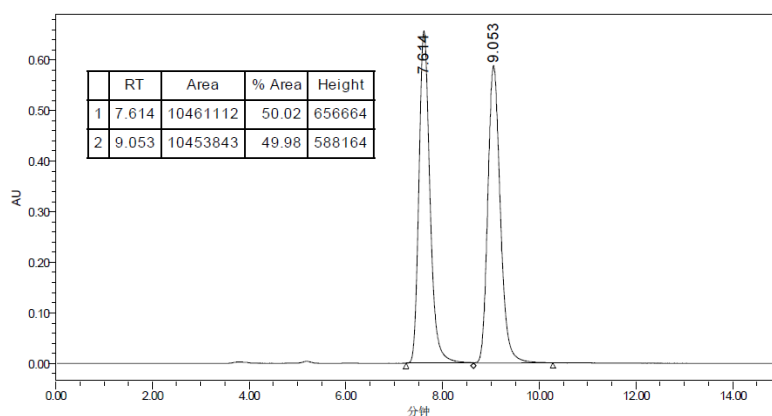


HPLC spectrum of (S)-**9i**

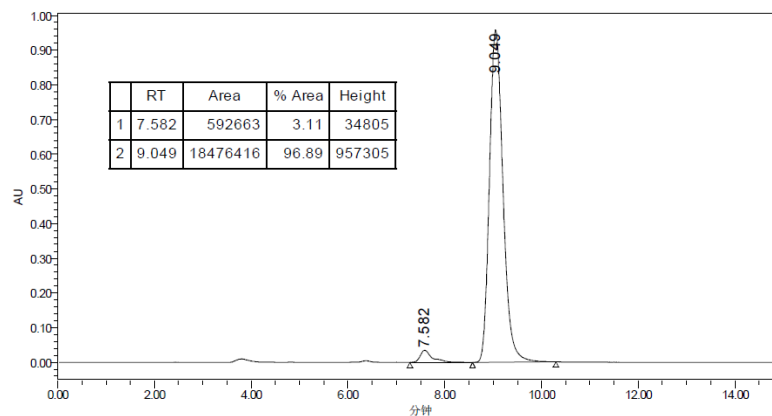


	
(R)- 9j , 38% yield, 94% ee.	(S)- 9j , 45% yield, 96% ee.
HPLC condition: Chiralpak IE, hexane/ <i>i</i> -PrOH = 90/10, 1.0 mL/min, $t_R(\text{minor}) = 7.6$ min, $t_R(\text{major}) = 9.0$ min.	HPLC condition: Chiralpak IE, hexane/ <i>i</i> -PrOH = 90/10, 1.0 mL/min, $t_R(\text{major}) = 7.6$ min, $t_R(\text{minor}) = 9.1$ min.

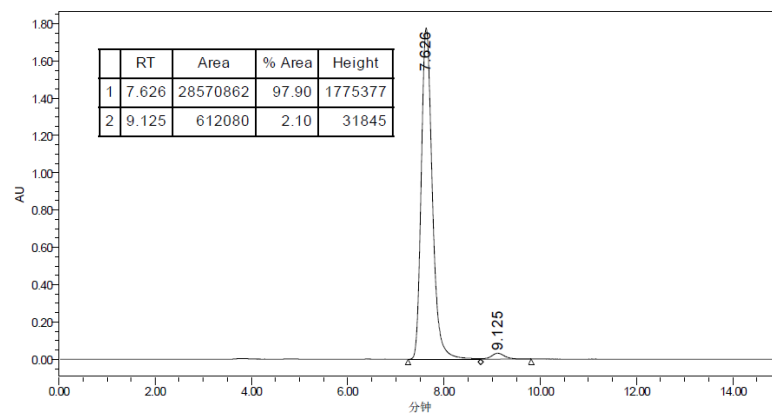
HPLC spectrum of racemic **9j**



HPLC spectrum of (R)-**9j**

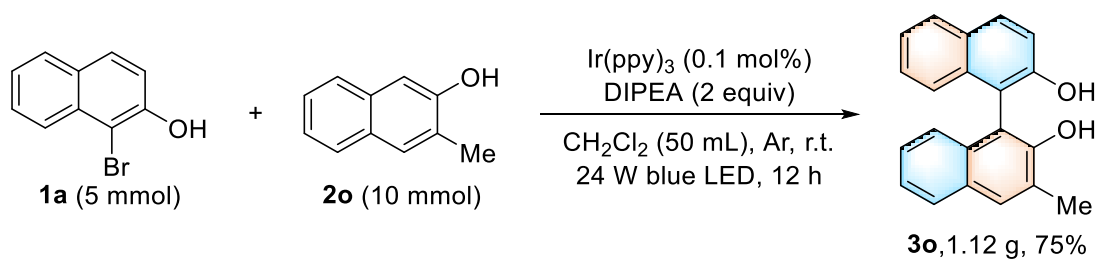


HPLC spectrum of (S)-**9j**



VII. Gram-Scale Synthesis

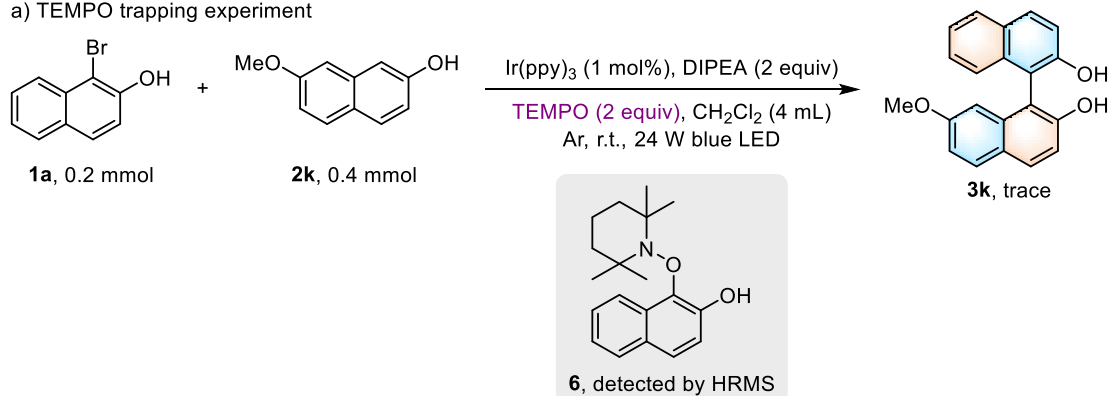
To an oven-dried 100 mL Schlenk bottle were added 1-bromo-2-naphthol **1a** (5 mmol, 1.11 g), 3-methyl-2-naphthol **2o** (10 mmol, 1.58 g) and Ir(ppy)₃ (0.005 mmol, 3.3 mg). The mixture was placed under vacuum and charged with argon for three times. Then CH₂Cl₂ (50 mL) and DIPEA (10 mmol, 1.65 mL) were added. Then the tube was placed approximate 4~5 cm away from 24 W blue LED and stir vigorously for 12 h with a cooling fan to maintain the reaction at room temperature (about 25 °C). Upon completion of the reaction, the mixture was concentrated and purified by silica chromatography to afford the product **3o** in 75% yield.



VIII. Mechanistic Investigations for Photocatalysis

1. Radical Trapping Experiments

a) TEMPO trapping experiment



Following **GPA**, TEMPO (0.4 mmol, 2 equiv) was added and the reaction mixture was stirred at r.t. for 12 h. The product **3k** could only be obtained in trace yield. TEMPO-trapping intermediate **6** was detected by HRMS ($[\text{M}-\text{H}]^-$: cal. 298.1813; found 298.1806). The aryl-TEMPO was not observed by ^1H NMR, which might be due to the high steric demands of *ortho*-disubstituted aryl radical with a hindered radical.^[4]

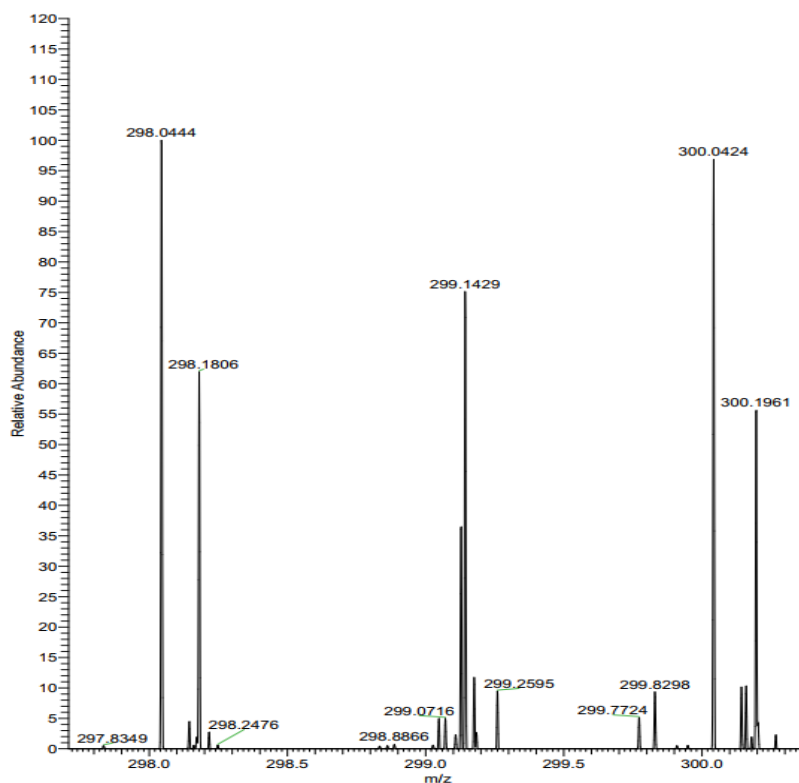
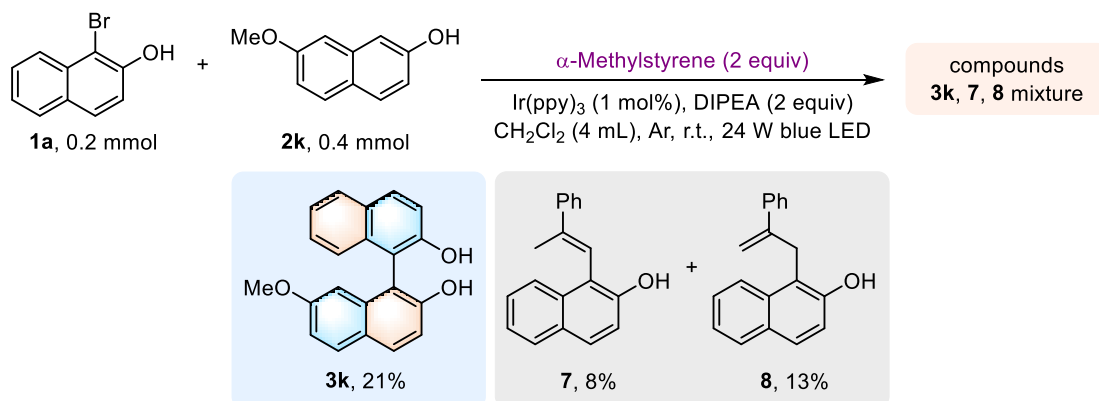


Figure S3. HRMS spectrum of the adduct **6**

b) α -Methylstyrene trapping experiment



Following **GPA**, α -methylstyrene (0.4 mmol, 2 equiv) was added and the reaction mixture was stirred at r.t. for 12 h. The product **3k** was obtained in 21% yield. The aryl radical can be trapped to afford the adducts **7** and **8** in 8% and 13% yield, respectively.

(*E*)-1-(2-phenylprop-1-en-1-yl)naphthalen-2-ol (**7**)

^1H NMR (400 MHz, DMSO- d_6) δ 9.51 (s, 1H), 7.82 – 7.68 (m, 5H), 7.44 – 7.40 (m, 3H), 7.35 – 7.29 (m, 2H), 7.23 (d, J = 8.8 Hz, 1H), 6.97 (s, 1H), 1.85 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 152.24, 142.17, 138.43, 132.79, 128.44, 128.37, 128.12, 127.87, 127.24, 126.18, 125.71, 124.05, 122.52, 121.19, 118.34, 116.87, 17.54. HRMS (ESI) $[\text{M-H}]^-$: calculated m/z for $[\text{C}_{19}\text{H}_{15}\text{O}]^-$: 259.1128, found: 259.1125.

1-(2-phenylallyl)naphthalen-2-ol (**8**)

^1H NMR (400 MHz, DMSO- d_6) δ 9.65 (s, 1H), 7.78 (dd, J = 8.1, 1.4 Hz, 1H), 7.71 – 7.64 (m, 4H), 7.41 – 7.32 (m, 4H), 7.27 – 7.21 (m, 2H), 5.29 (d, J = 1.7 Hz, 1H), 4.40 (d, J = 1.7 Hz, 1H), 4.12 (s, 2H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 152.79, 145.82, 141.16, 133.66, 128.34, 128.21, 128.14, 127.86, 127.57, 126.08, 125.68, 123.13, 122.21, 118.05, 116.26, 112.10, 29.68. HRMS (ESI) $[\text{M-H}]^-$ calculated m/z for $[\text{C}_{19}\text{H}_{15}\text{O}]^-$: 259.1128, found: 259.1125.

2. The Cyclic Voltammetry Experiments

Cyclic voltammetry experiments were performed on a Chenhua Instruments Electrochemical Workstation (Model: CHI602E) at room temperature under argon atmosphere. A steady glassy carbon working electrode was applied alongside with a Pt counter electrode with a Ag/AgCl reference electrode. The solution was freshly prepared and bubbled with argon for 10 min before measurements. A 10 mM solution of **1a** was separately prepared in dry MeCN (bubbled with argon for 30 min prior to solution preparing) with 0.1 M of tetrabutylammonium hexafluorophosphate ($n\text{Bu}_4\text{NPF}_6$) as supporting electrolyte and was examined at a scan rate of 0.1 V s^{-1} . Fc^+/Fc (vs SVE)⁵ was added at the end of the measurements as an internal standard to determine the precise potential.

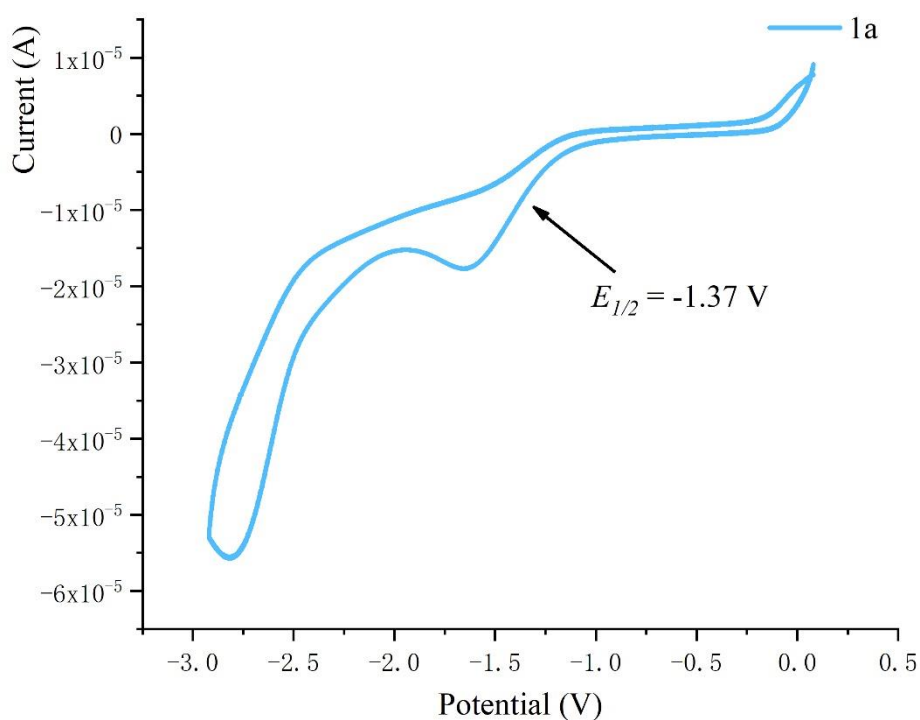
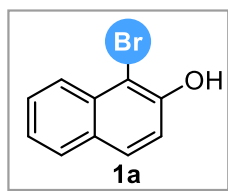


Figure S4. Cyclic voltammogram of **1a**

3. Luminescent Quenching Experiments

Fluorescence quenching experiments were recorded on Horiba Scientific FluoroMax-4 Spectrofluorometer. The experiments were run with freshly prepared 5×10^{-5} solution of Ir(ppy)_3 in dry and degassed CH_2Cl_2 added the appropriate amount quencher in screw-top quartz cuvette at room temperature. After degassing with argon for 10 min, the solutions were excited at 370 nm and fluorescence emissions were measured from 450 nm to 700 nm.

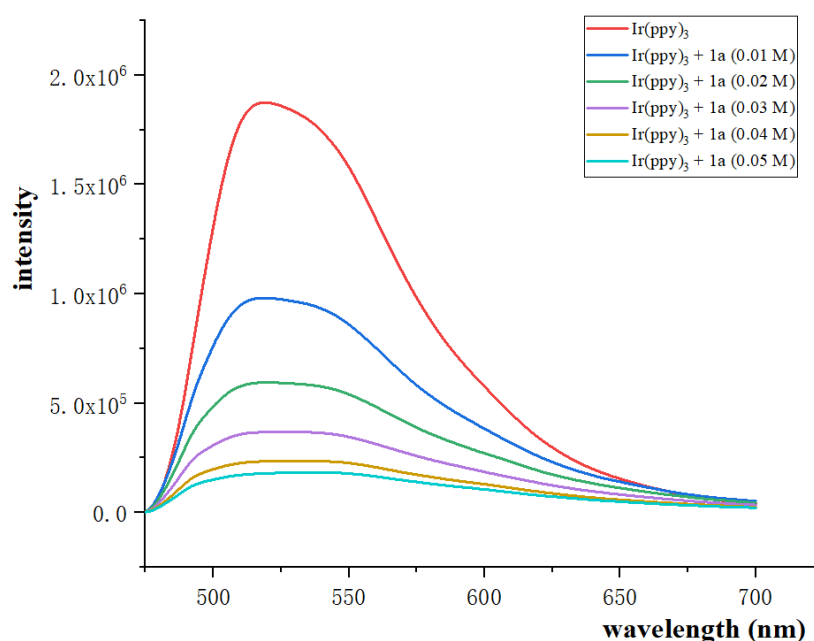


Figure S5. The Luminescent Quenching of **1a**

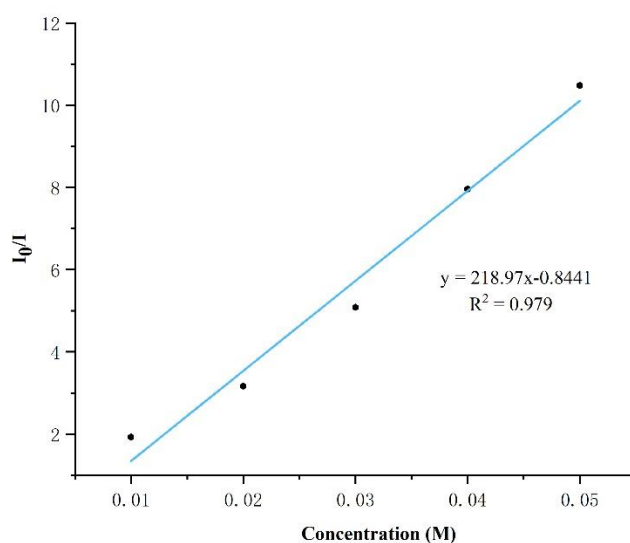


Figure S6. Stern-Volmer Plot with **1a**

IX. Crystallographic Data

1. XRD structure and data of 3k

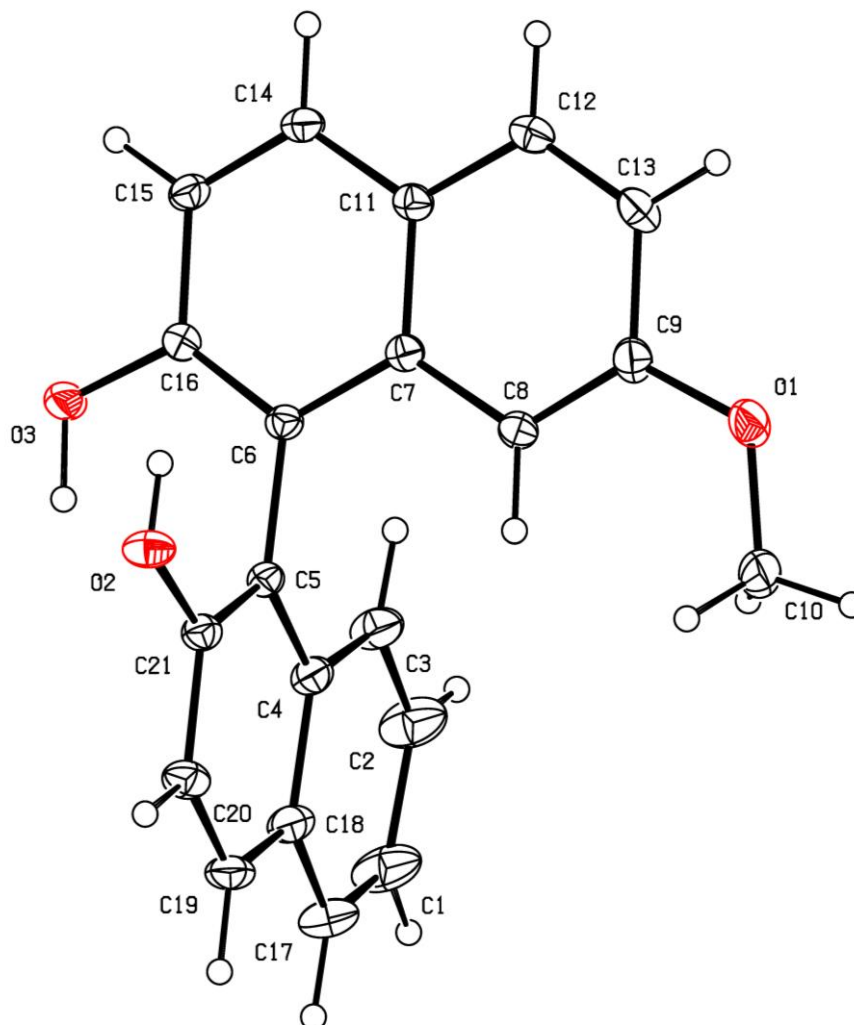


Table S9. Crystal data and structure refinement for **3k**

Identification code	3k
Empirical formula	C _{21.5} ClH ₁₇ O ₃
Formula weight	358.80
Temperature/K	100.0
Crystal system	triclinic
Space group	P-1
a/Å	10.6383(4)
b/Å	14.7209(5)

$c/\text{\AA}$	24.4367(9)
$\alpha/^\circ$	100.708(2)
$\beta/^\circ$	101.698(2)
$\gamma/^\circ$	92.046(2)
Volume/ \AA^3	3671.5(2)
Z	8
$\rho_{\text{calc}}/\text{g/cm}^3$	1.298
μ/mm^{-1}	1.982
F(000)	1496.0
Crystal size/ mm^3	$0.32 \times 0.22 \times 0.18$
Radiation	$\text{CuK}\alpha$ ($\lambda = 1.54178$)
2Θ range for data collection/ $^\circ$	6.128 to 136.764
Index ranges	$-12 \leq h \leq 12, -17 \leq k \leq 17, -29 \leq l \leq 29$
Reflections collected	85311
Independent reflections	13463 [$R_{\text{int}} = 0.0397, R_{\text{sigma}} = 0.0243$]
Data/restraints/parameters	13463/0/876
Goodness-of-fit on F^2	1.026
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0386, wR_2 = 0.1011$
Final R indexes [all data]	$R_1 = 0.0429, wR_2 = 0.1041$
Largest diff. peak/hole / $e \text{\AA}^{-3}$	0.30/-0.35

2. XRD structure and data of *rac*-11a

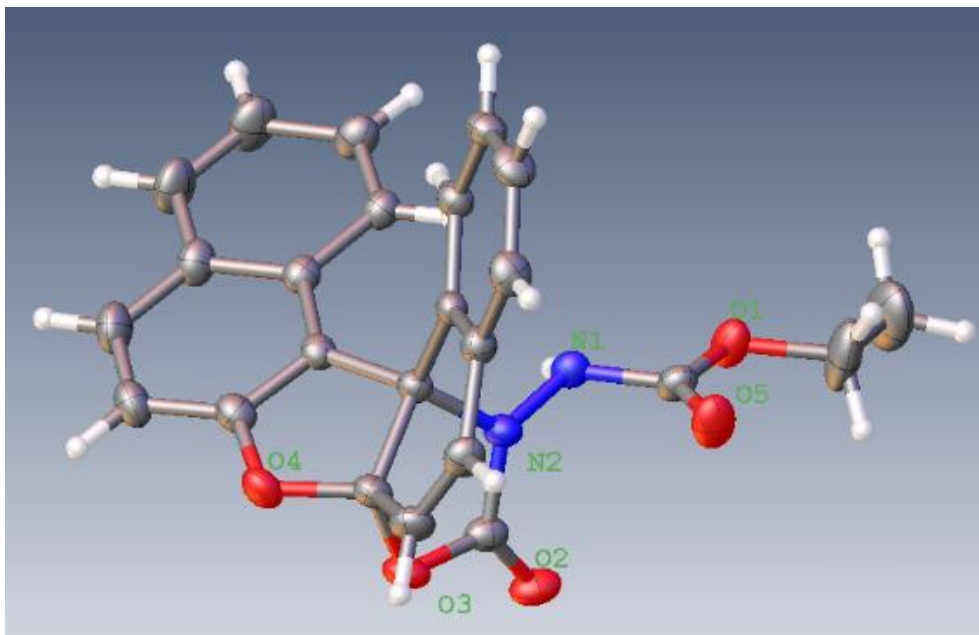


Table S10. Crystal data and structure refinement for *rac*-11a

Identification code	<i>rac</i> -11a
Empirical formula	C ₂₄ H ₁₈ N ₂ O ₅
Formula weight	414.40
Temperature/K	150.0
Crystal system	triclinic
Space group	P-1
a/Å	9.4644(6)
b/Å	10.5602(7)
c/Å	11.8747(8)
α /°	113.482(3)
β /°	101.482(3)
γ /°	105.305(3)
Volume/Å ³	985.89(12)
Z	2
ρ calc/gcm ³	1.396
μ /mm ⁻¹	0.819

F(000)	432.0
Crystal size/mm ³	0.33 × 0.28 × 0.24
Radiation	CuK α (λ = 1.54178)
2 Θ range for data collection/ $^{\circ}$	8.646 to 136.834
Index ranges	-11 \leq h \leq 11, -12 \leq k \leq 12, -14 \leq l \leq 14
Reflections collected	14873
Independent reflections	3607 [R_{int} = 0.0306, R_{sigma} = 0.0268]
Data/restraints/parameters	3607/1/285
Goodness-of-fit on F ²	1.056
Final R indexes [$I \geq 2\sigma(I)$]	R_1 = 0.0374, wR_2 = 0.0957
Final R indexes [all data]	R_1 = 0.0403, wR_2 = 0.0975
Largest diff. peak/hole / e \AA^{-3}	0.35/-0.28

X. Reference

- [1] a) Y.-H. Chen, D.-J. Cheng, J. Zhang, Y. Wang, X.-Y. Liu, B. Tan, *J. Am. Chem. Soc.* **2015**, *137*, 15062–15065; b) Y.-H. Chen, H.-H. Li, X. Zhang, S.-H. Xiang, S. Li, B. Tan, *Angew. Chem. Int. Ed.* **2020**, *59*, 11374–11378; c) S. Narute, R. Parnes, F. D. Toste, D. Pappo, *J. Am. Chem. Soc.* **2016**, *138*, 16553–16560; d) Y. Lv, M. Li, S. Cao, L. Tong, T. Peng, L. Wei, H. Xie, J. Ding, W. Duan, *Med. Chem. Commun.* **2015**, *6*, 1375–1380; e) H. Qiu, B. Shuai, Y.-Z. Wang, D. Liu, Y.-G. Chen, P.-S. Gao, H.-X. Ma, S. Chen, T.-S. Mei, *J. Am. Chem. Soc.* **2020**, *142*, 9872–9878; f) J. Wang, Y. Zhao, H. Gao, G.-L. Gao, C. Yang, W. Xia, *Asian J. Org. Chem.* **2017**, *6*, 1402–1407.
- [2] M. Ashram, S. Mizyed, P. E. Georghiou, *J. Org. Chem.* **2001**, *66*, 1473–1479.
- [3] a) K. Mikami, T. Korenaga, Y. Yusa, M. Yamanaka, *Adv. Synth. Catal.* **2003**, *345*, 246–254; b) Y.-H. Chen, L.-W. Qi, F. Fang, B. Tan, *Angew. Chem. Int. Ed.* **2017**, *56*, 16308–16312.
- [4] S. I. Ting, S. Garakyaraghi, C. M. Taliaferro, B. J. Shields, G. D. Scholes, F. N. Castellano, A. G. Doyle, *J. Am. Chem. Soc.* **2020**, *142*, 5800–5810.
- [5] H. G. Roth, N. A. Romero, D. A. Nicewicz, *Synlett* **2016**, *27*, 714–723.
- [6] Q.-X. Guo, Z.-J. Wu, Z.-B. Luo, Q.-Z. Liu, J.-L. Ye, S.-W. Luo, L.-F. Cun, L.-Z. Gong, *J. Am. Chem. Soc.* **2007**, *129*, 13927–13938.
- [7] S. Fang, J.-P. Tan, J. Pan, H. Zhang, Y. Chen, X. Ren, T. Wang, *Angew. Chem. Int. Ed.* **2021**, *60*, 14921–14930.

XI. Copies of ^1H and ^{13}C Spectra

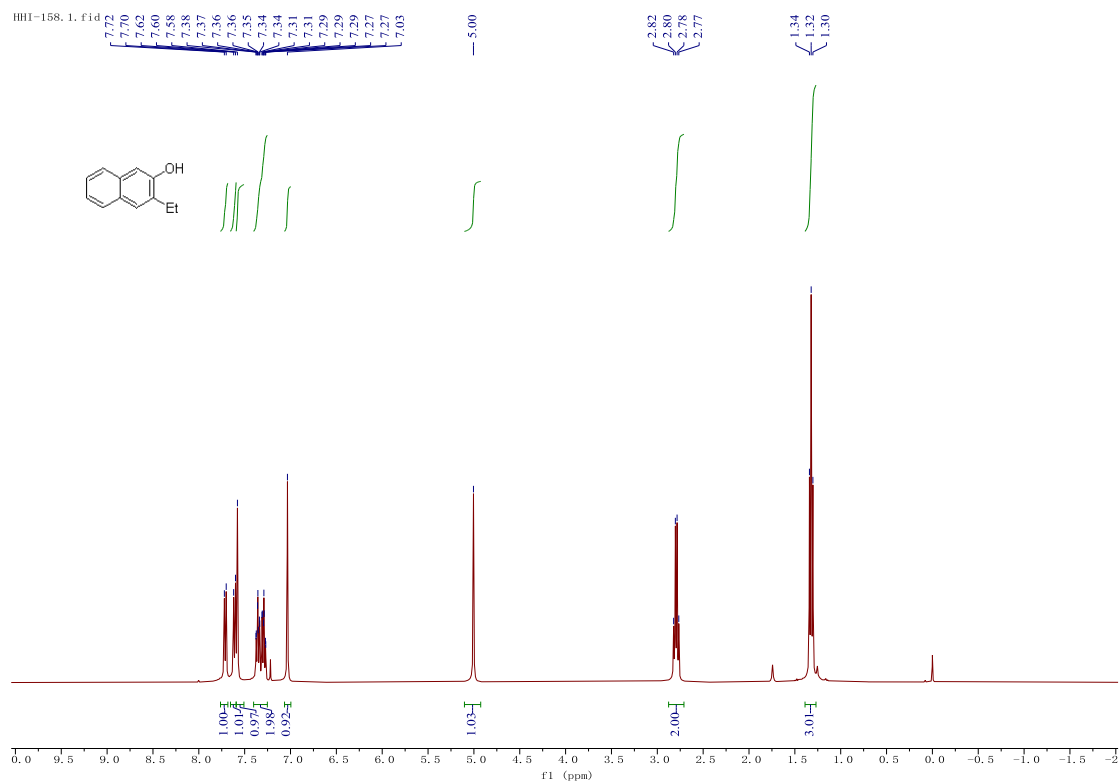


Figure S7. ^1H NMR Spectrum of **2p**

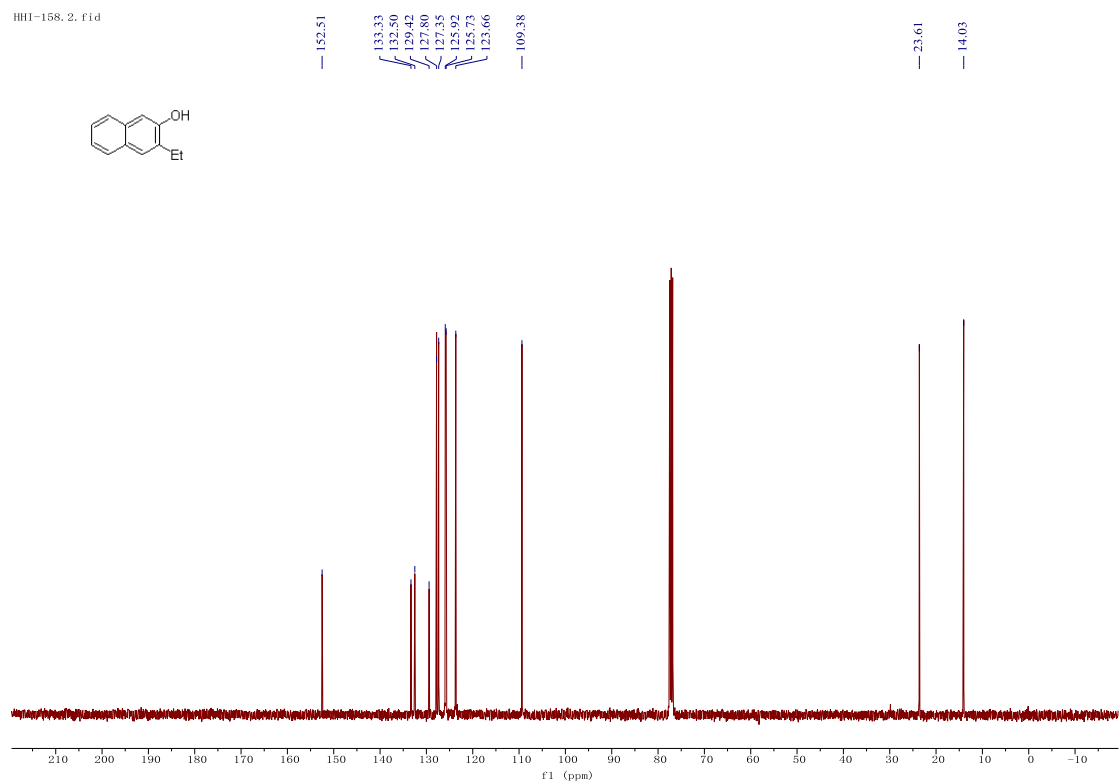


Figure S8. ^{13}C NMR Spectrum of **2p**

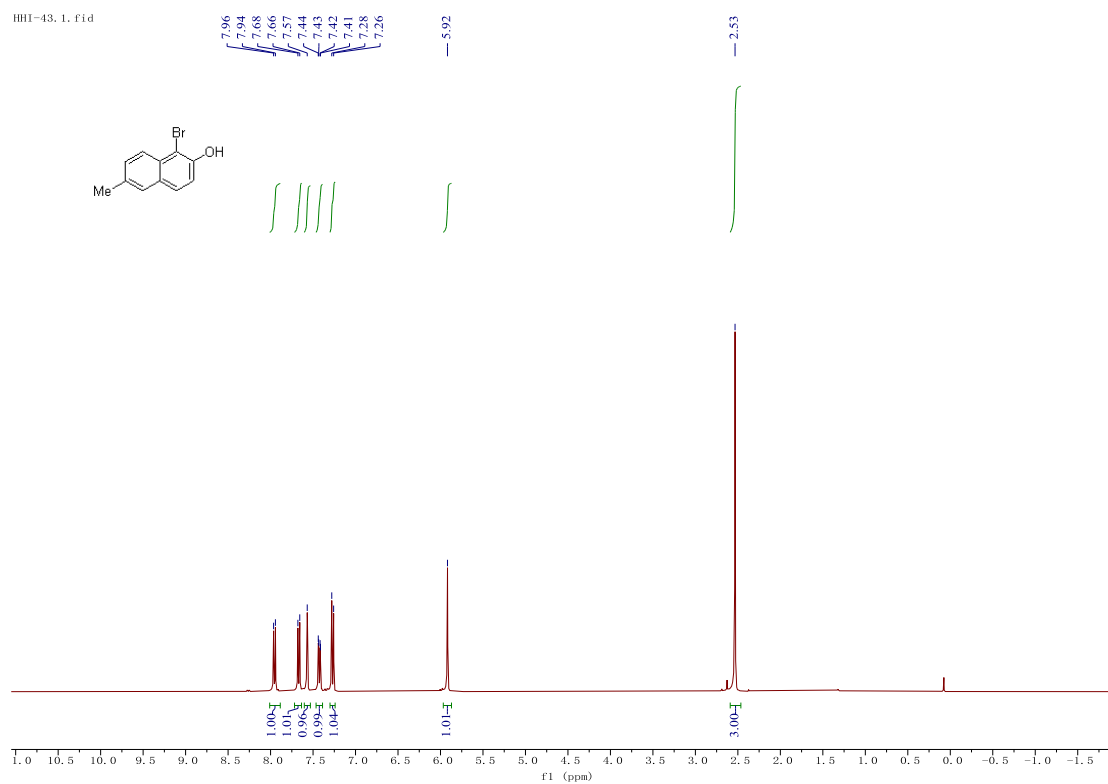


Figure S9. ^1H NMR Spectrum of 1b

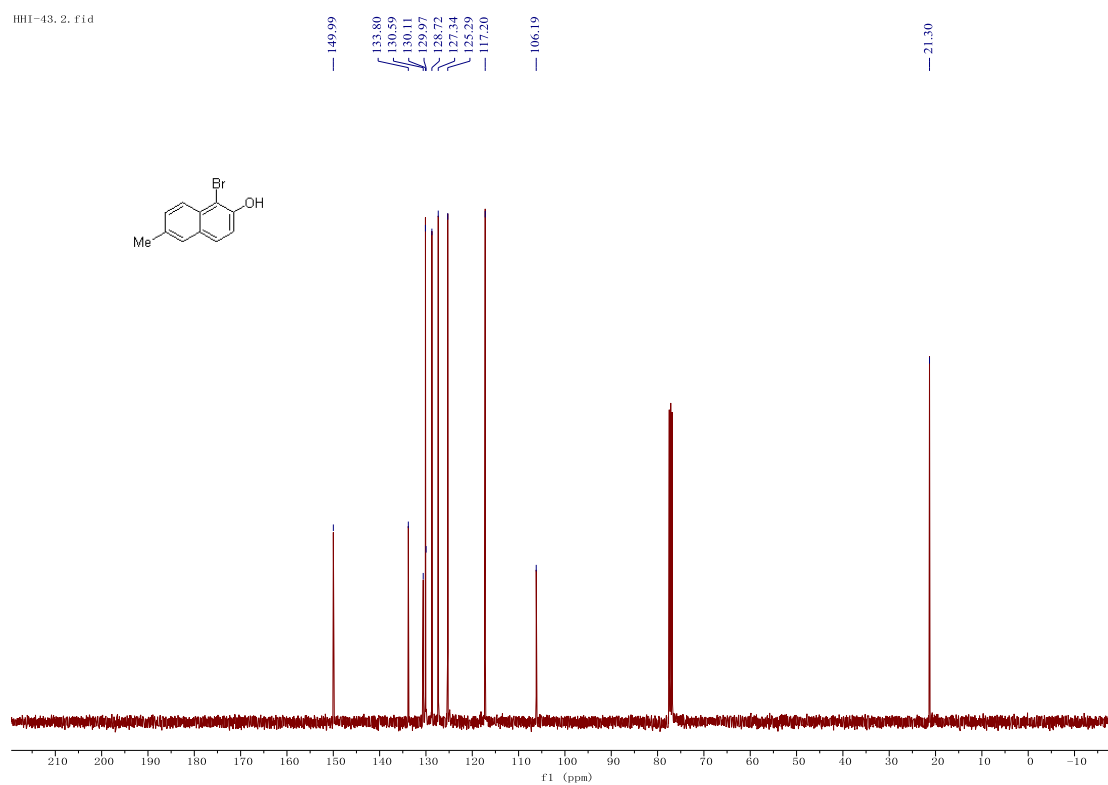


Figure S10. ^{13}C NMR Spectrum of 1b

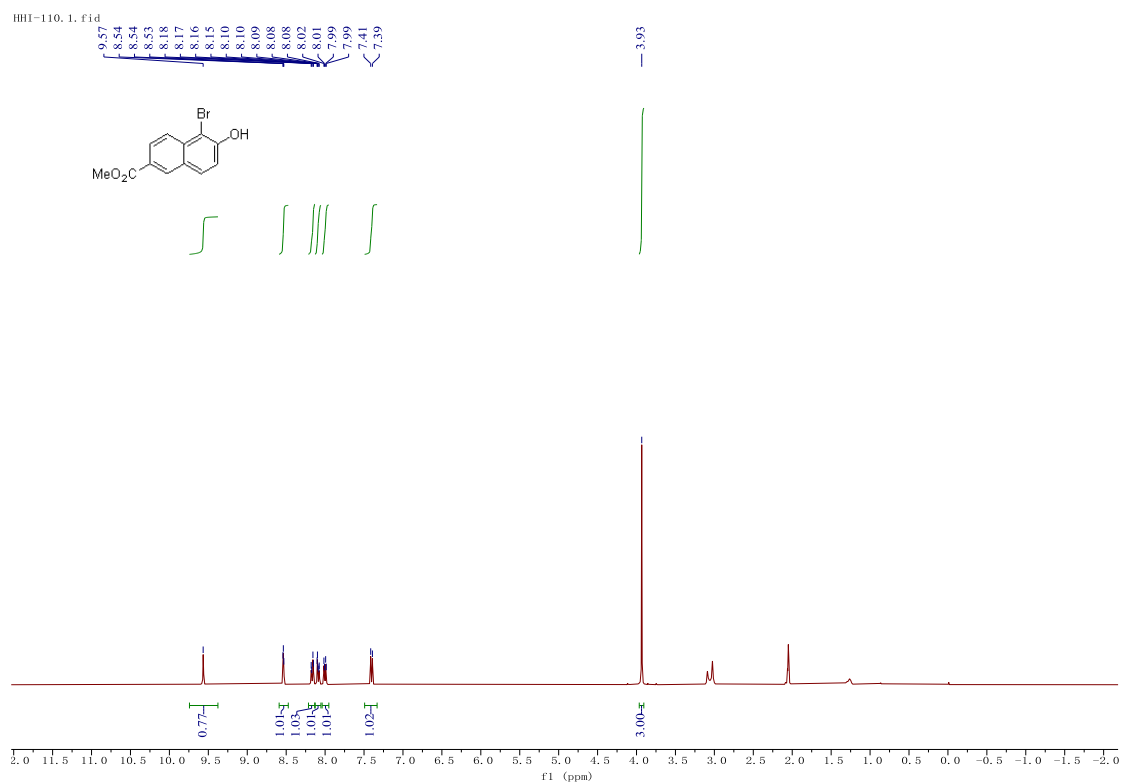


Figure S11. ^1H NMR Spectrum of **1c**

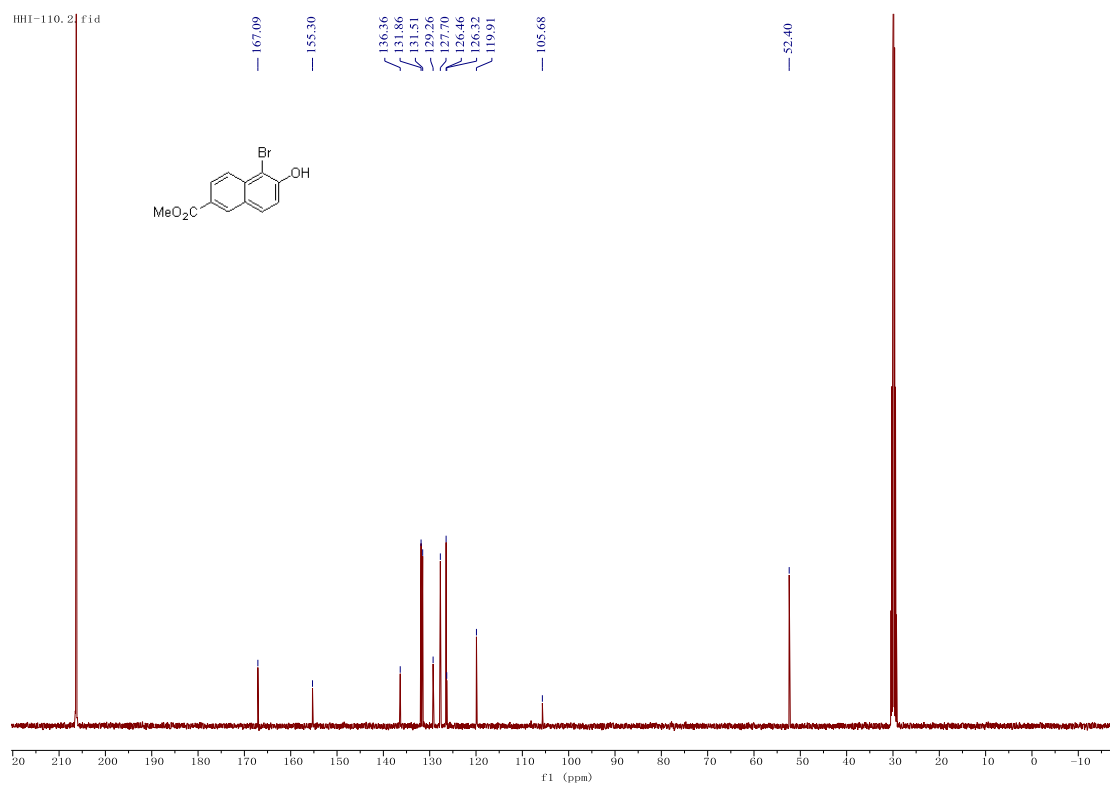


Figure S12. ^{13}C NMR Spectrum of **1c**

HHI-46, 10, f1d

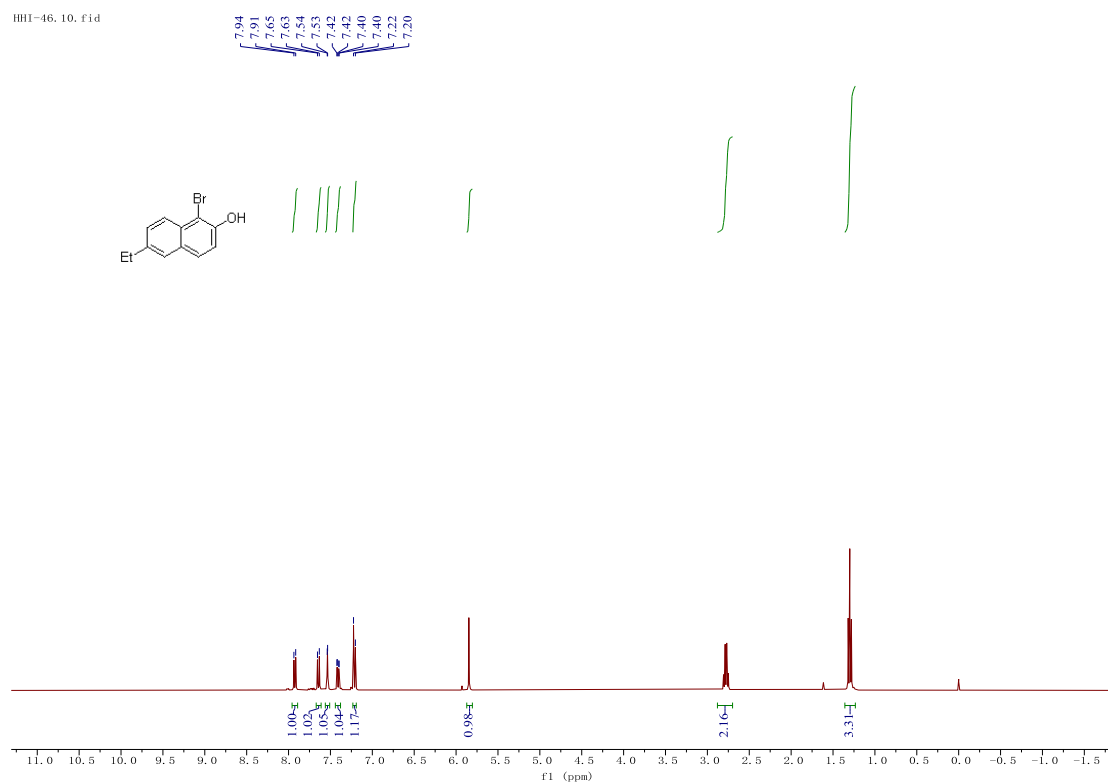


Figure S13. ^1H NMR Spectrum of **1d**

HHI-46, 12, f1d

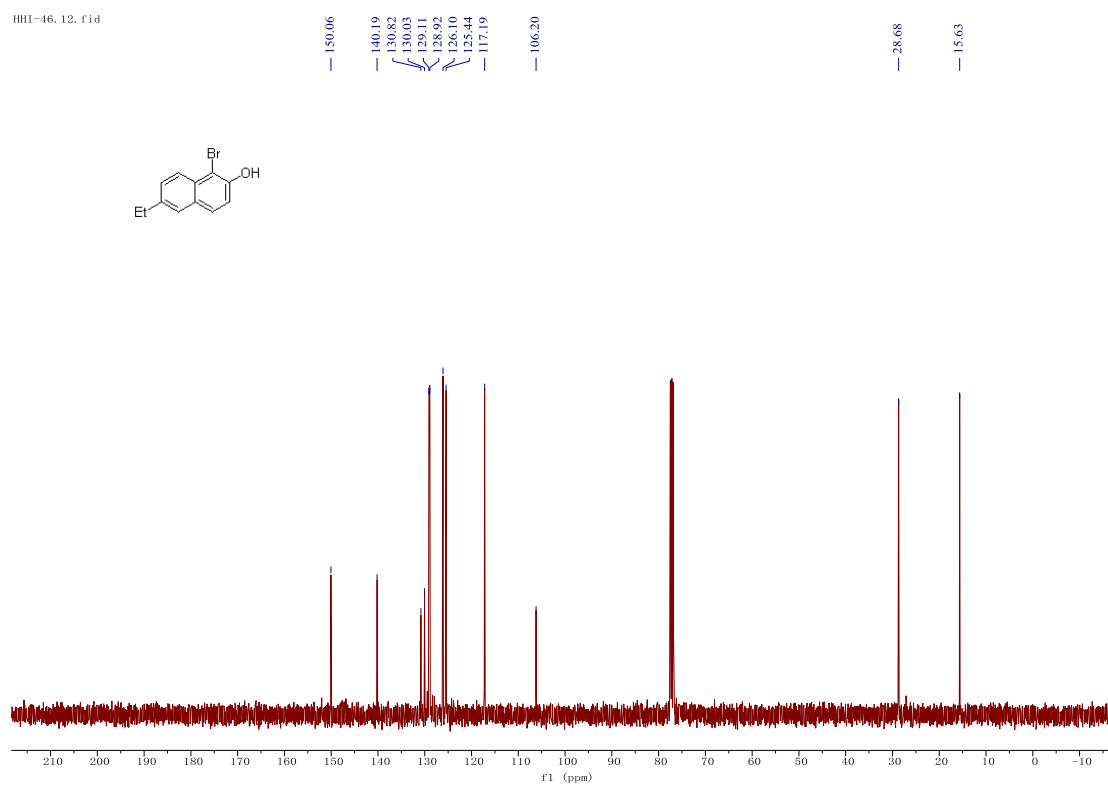


Figure S14. ^{13}C NMR Spectrum of **1d**

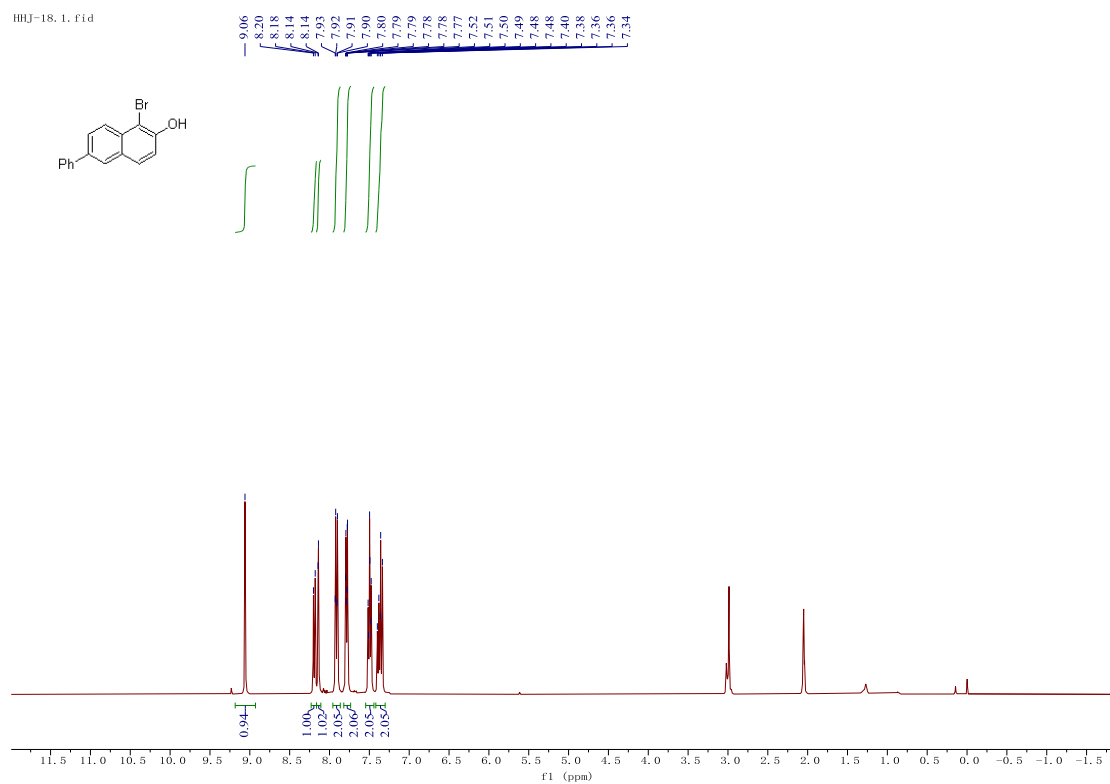


Figure S15. ¹H NMR Spectrum of **1e**

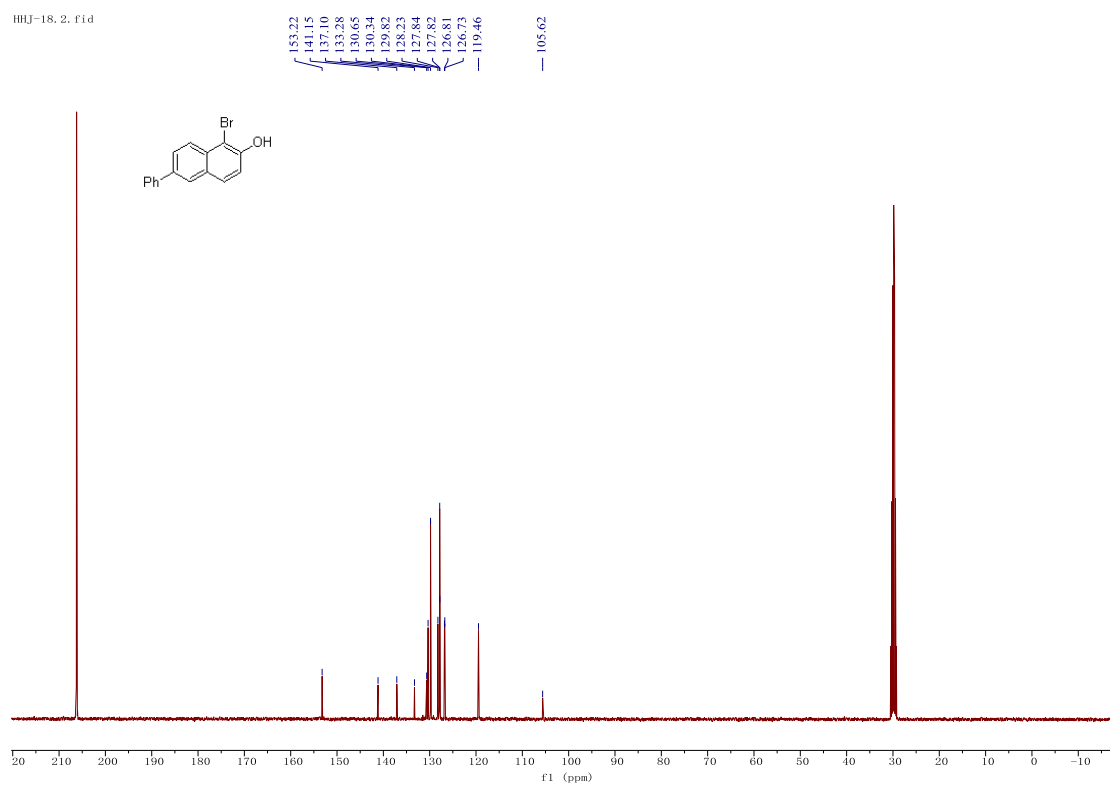


Figure S16. ¹³C NMR Spectrum of **1e**

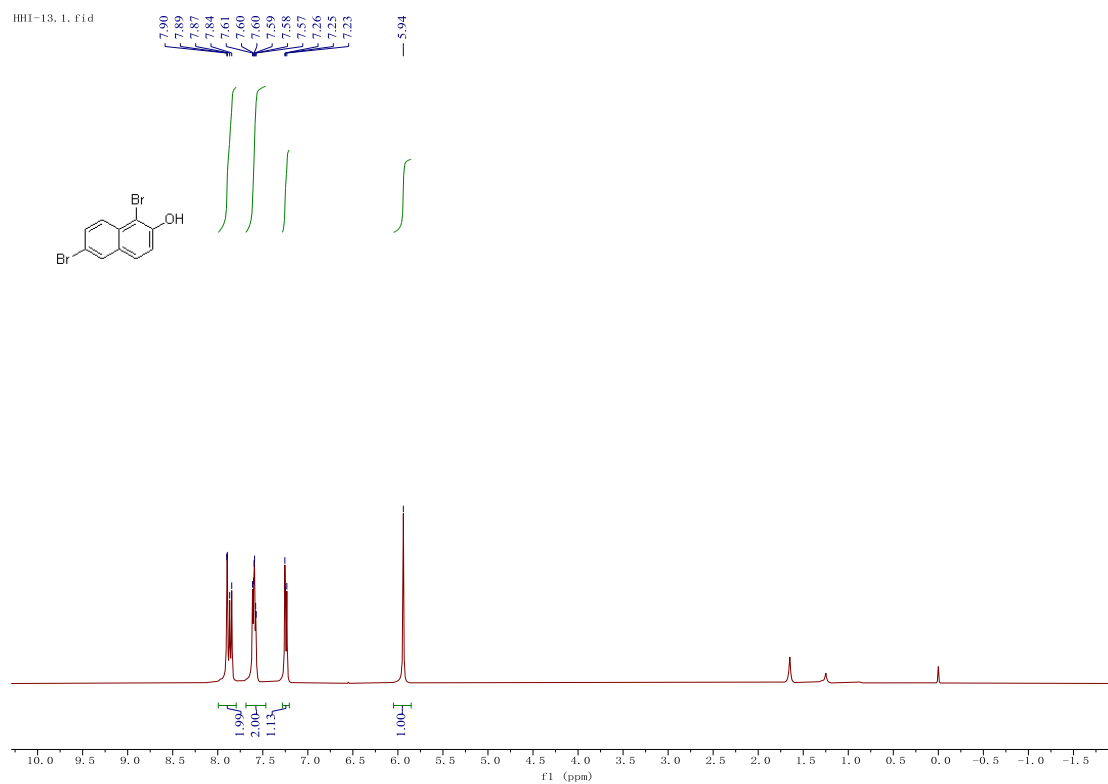


Figure S17. ^1H NMR Spectrum of **1f**

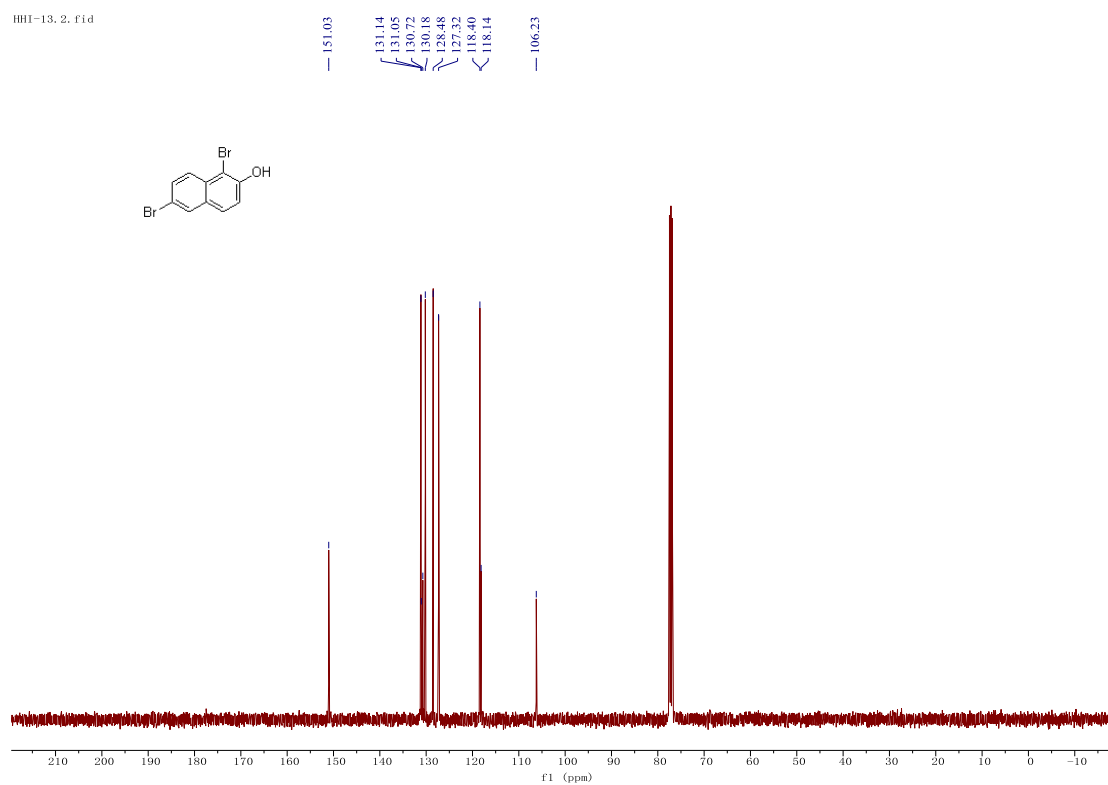
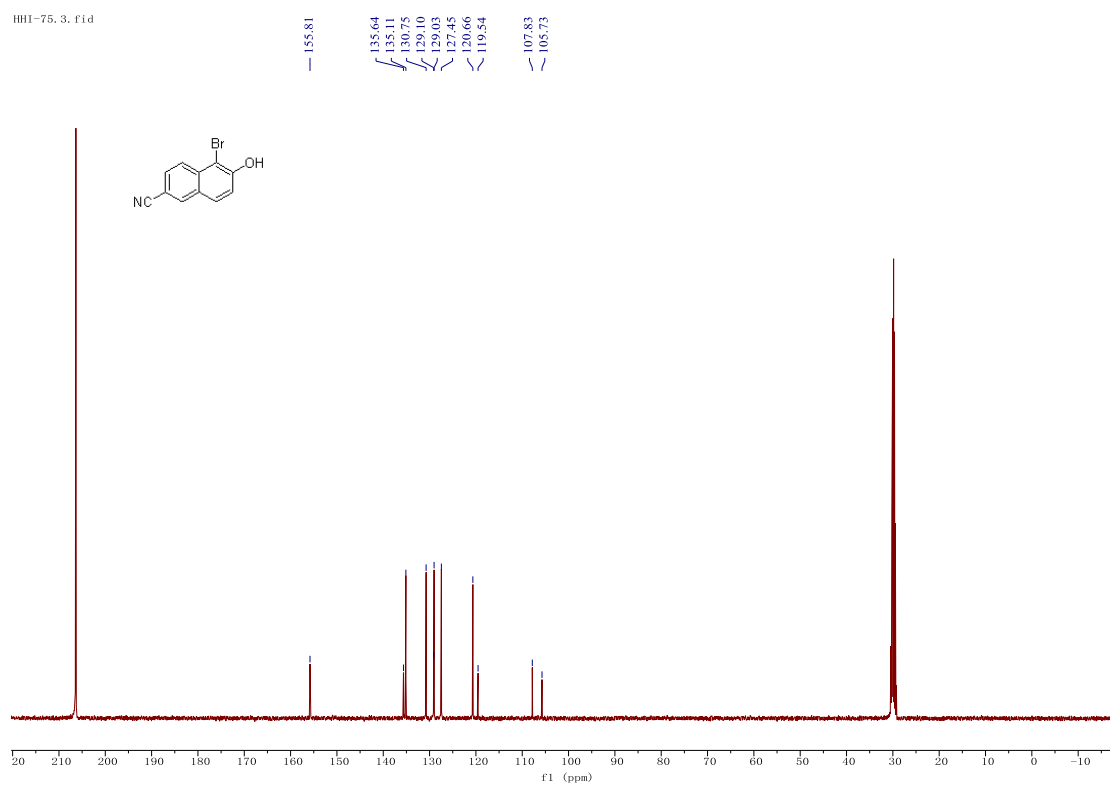
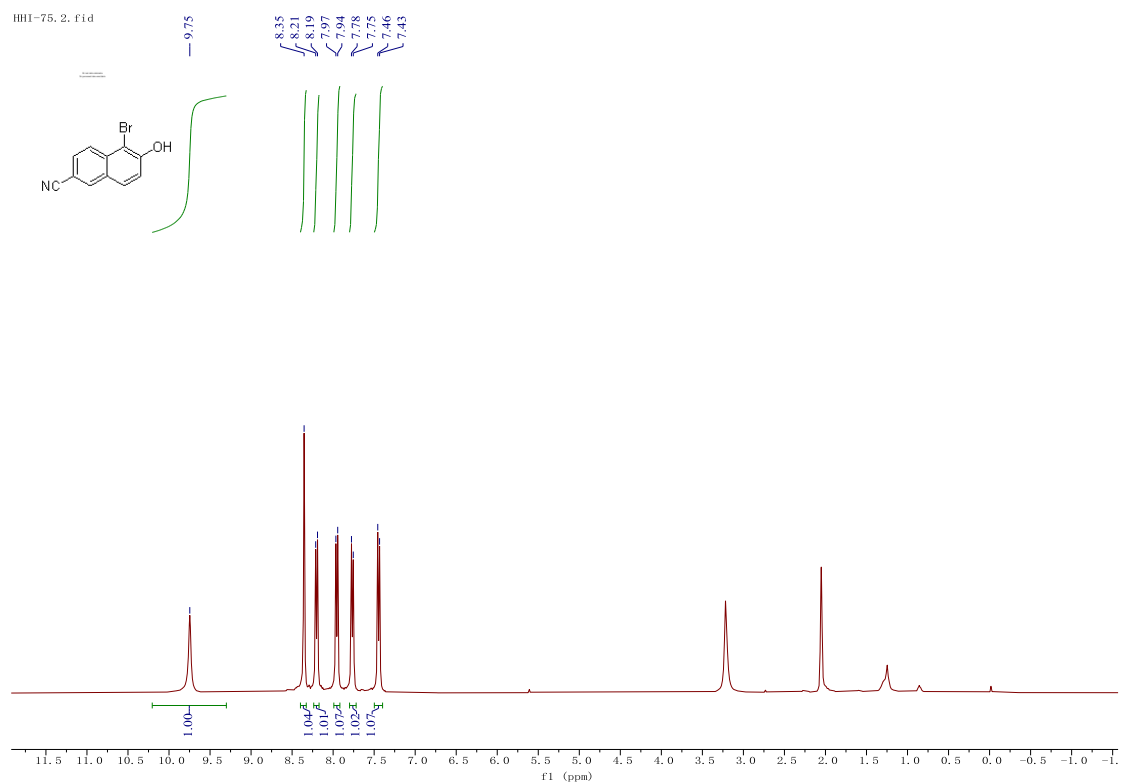


Figure S18. ^{13}C NMR Spectrum of **1f**



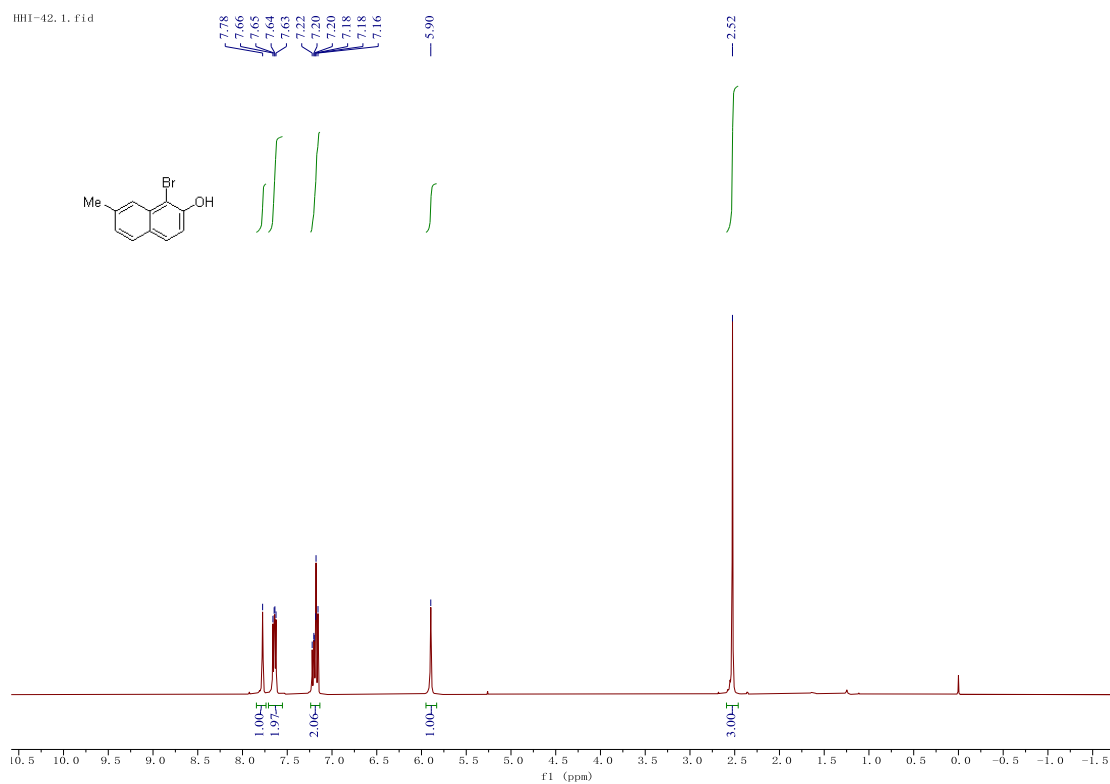


Figure S21. ^1H NMR Spectrum of **1h**

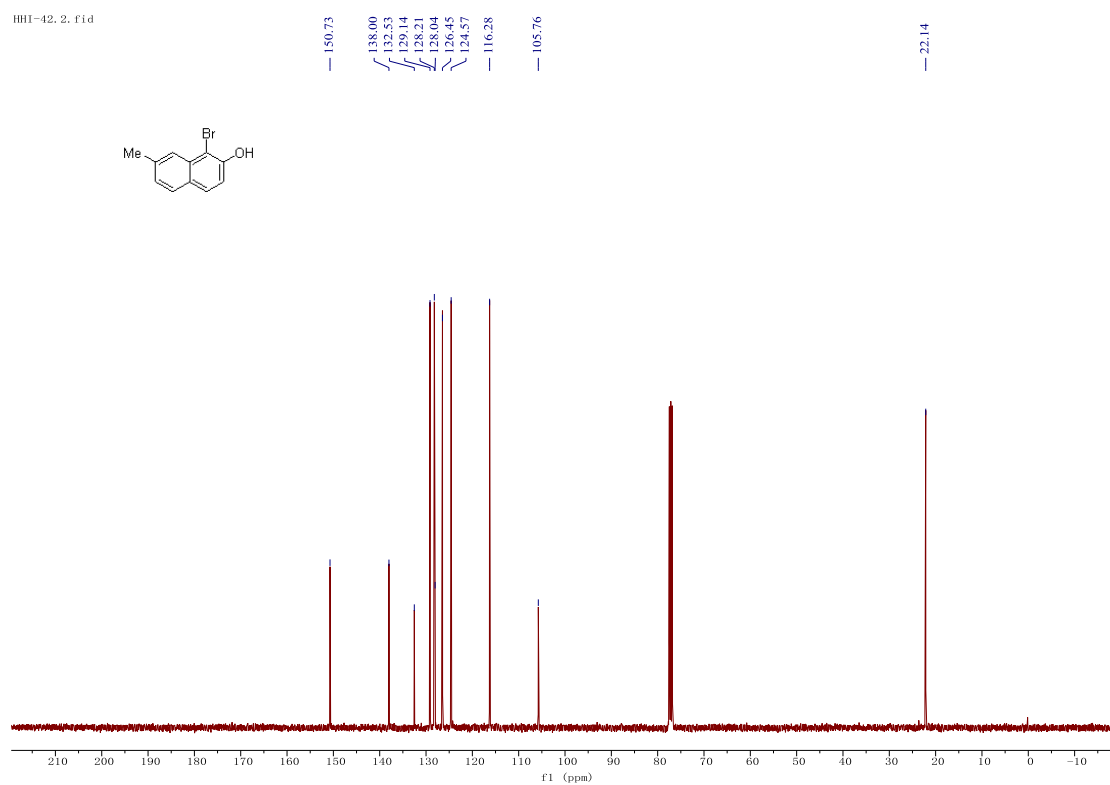


Figure S22. ^{13}C NMR Spectrum of **1h**

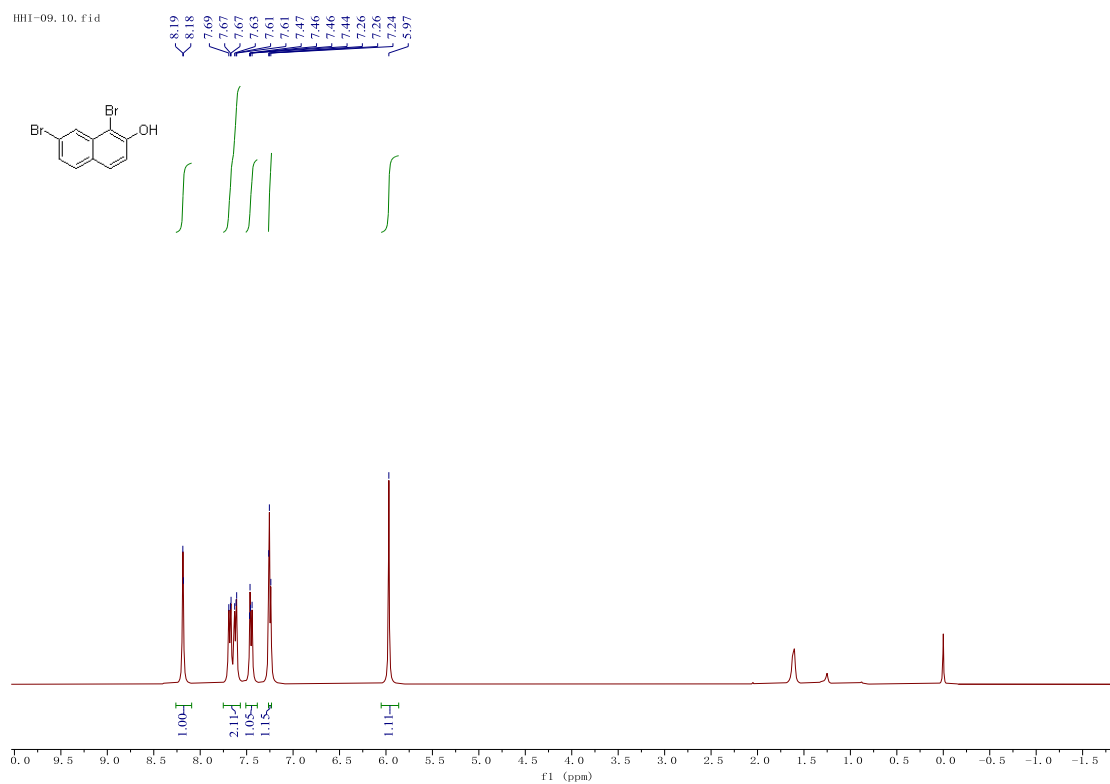


Figure S23. ^1H NMR Spectrum of **1i**

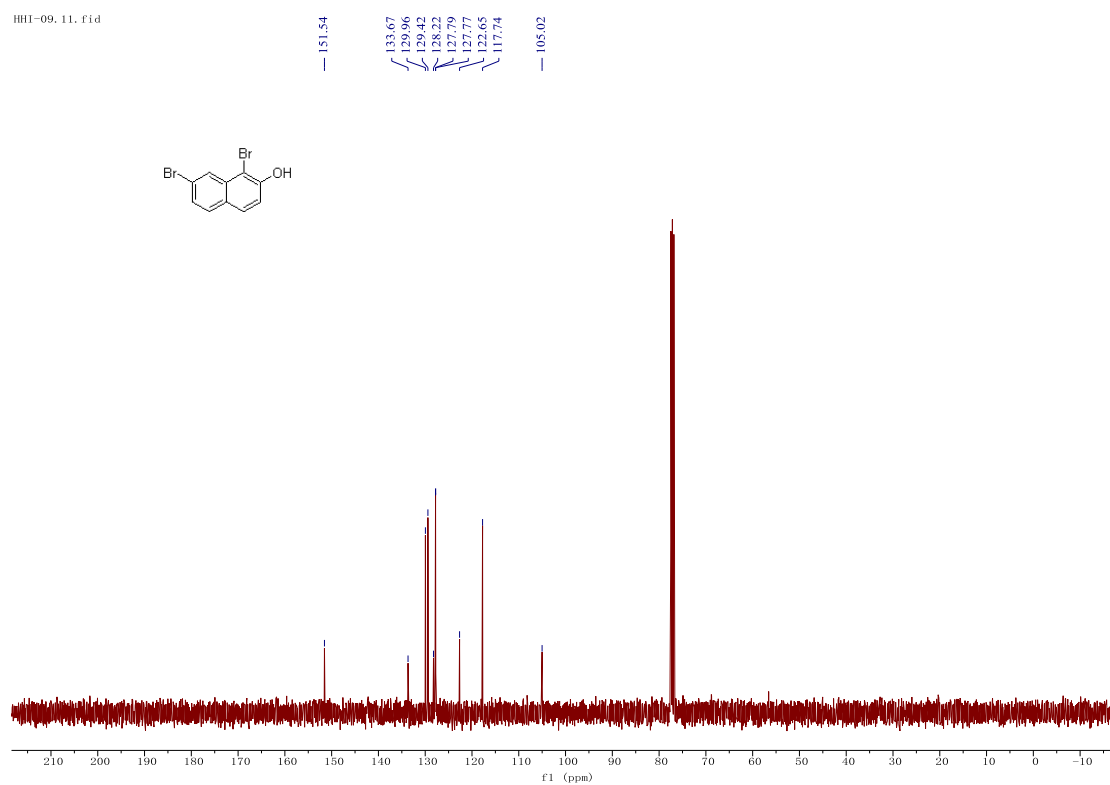


Figure S24. ^{13}C NMR Spectrum of **1i**

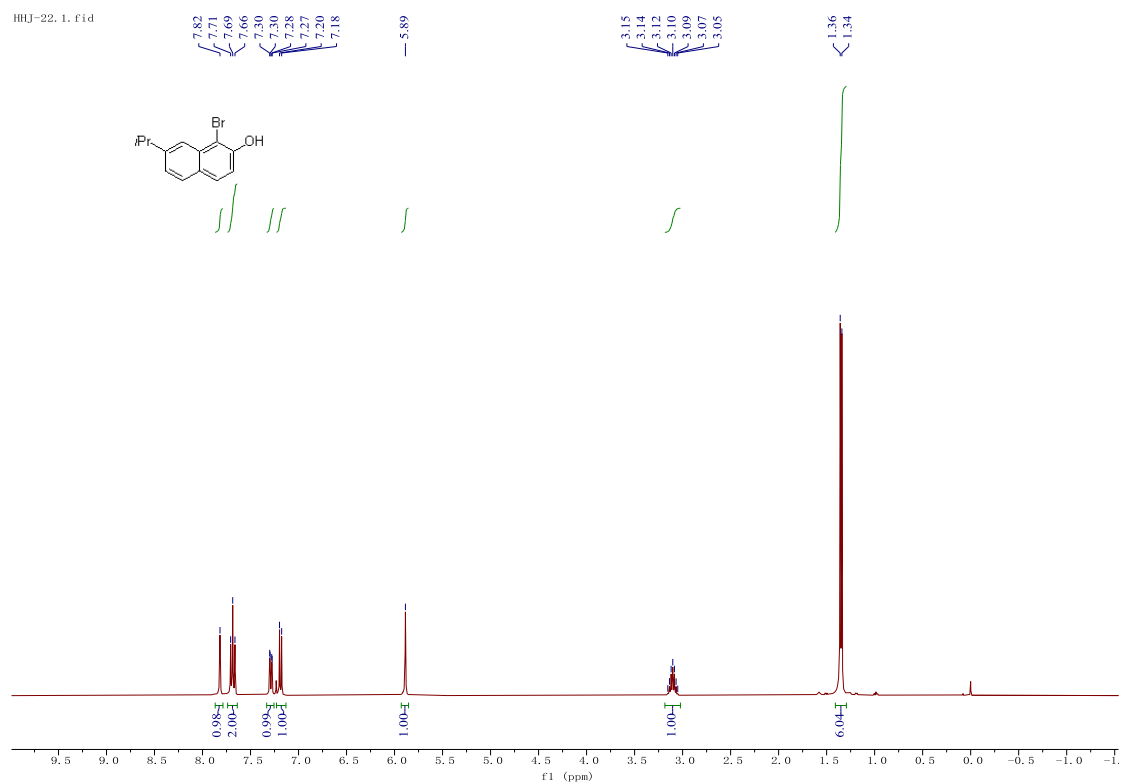


Figure S25. ^1H NMR Spectrum of **1j**

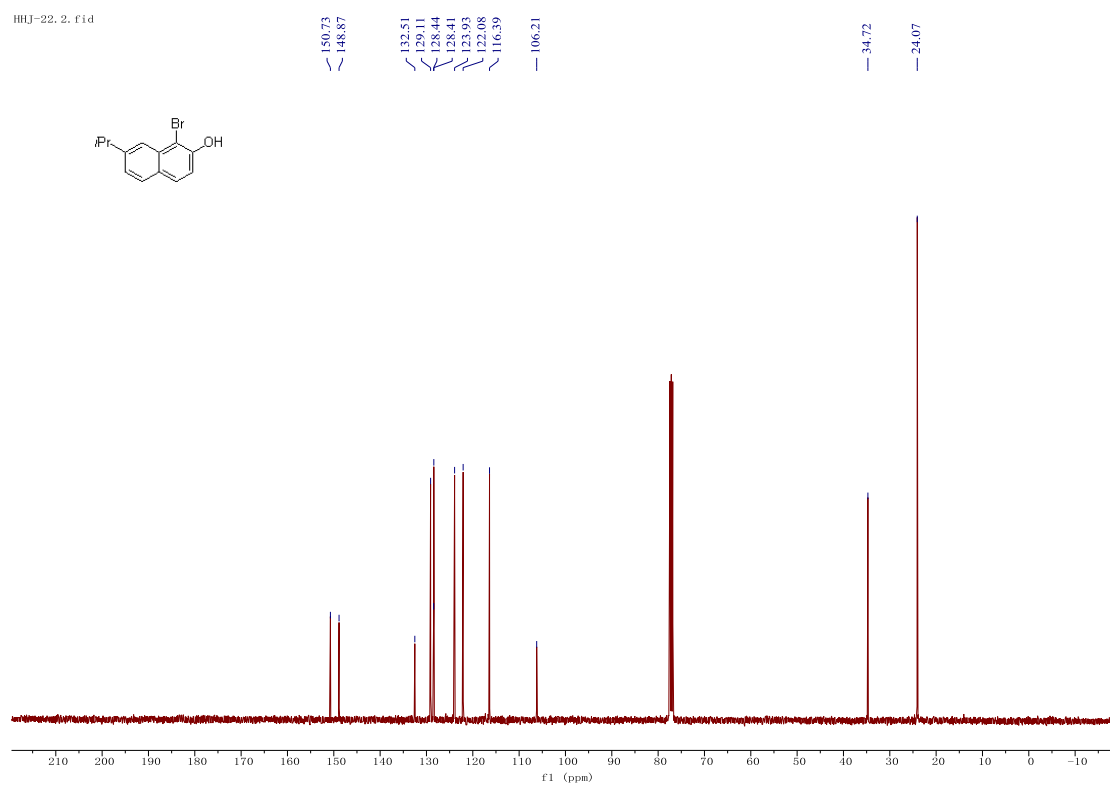


Figure S26. ^{13}C NMR Spectrum of **1j**

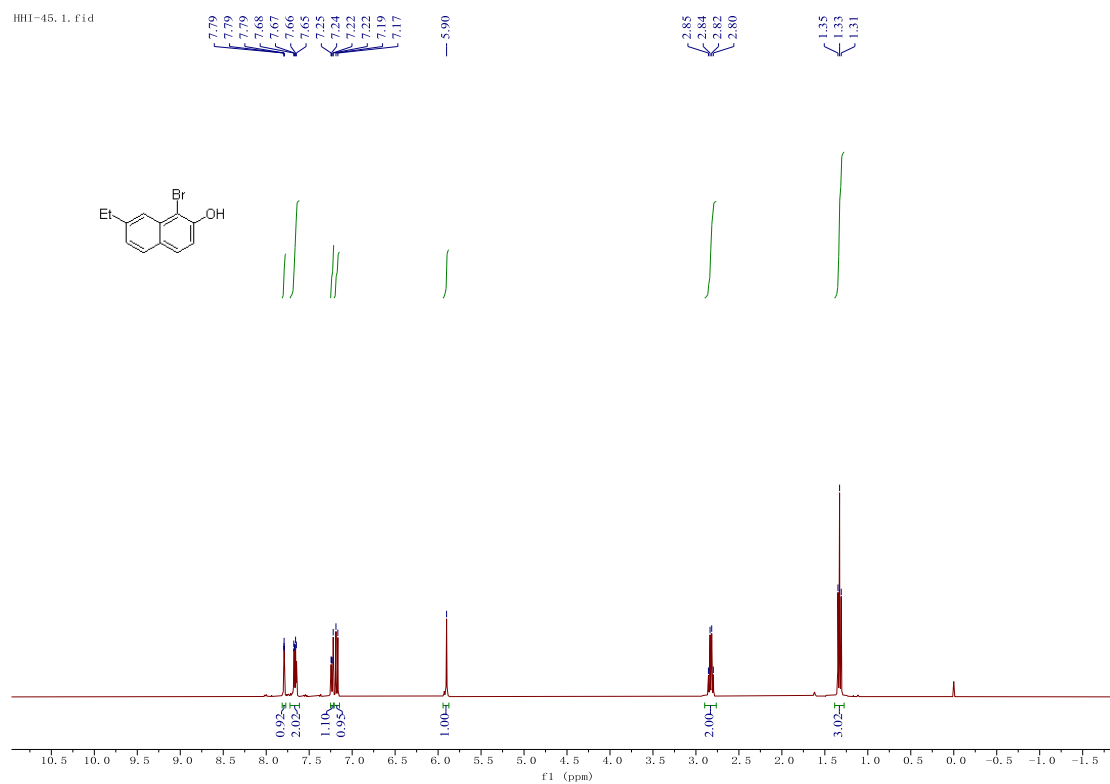


Figure S27. ^1H NMR Spectrum of **1k**

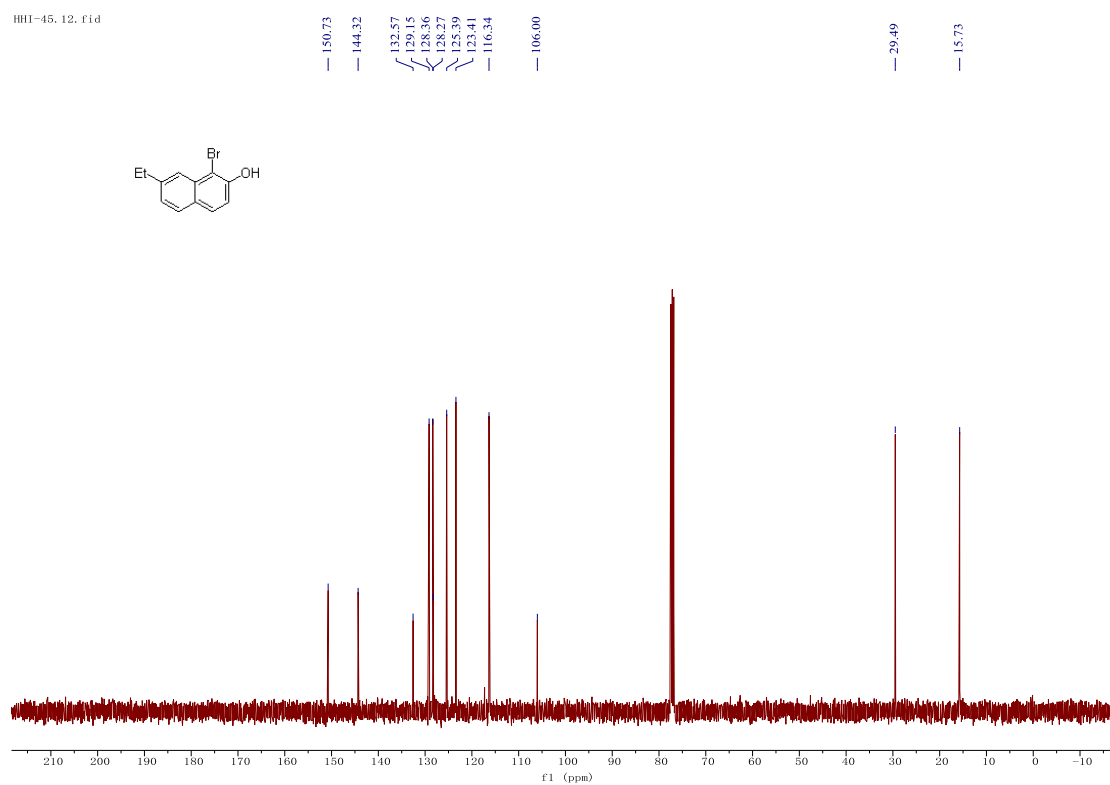


Figure S28. ^{13}C NMR Spectrum of **1k**

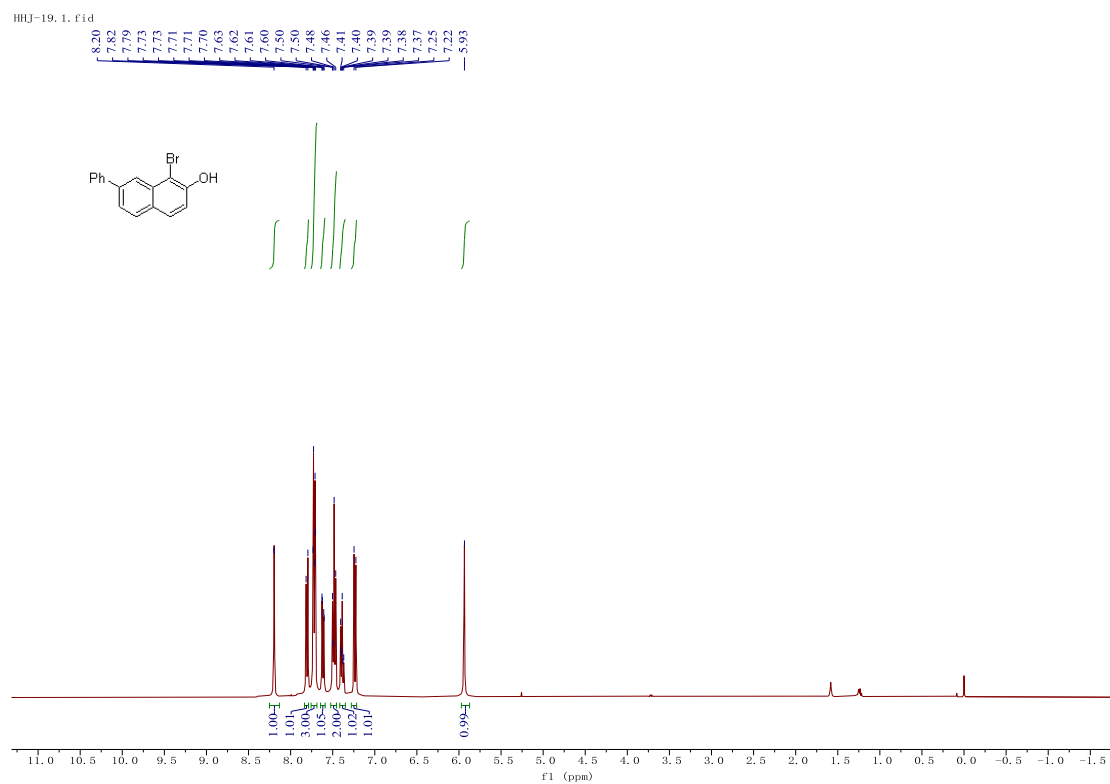


Figure S29. ^1H NMR Spectrum of **11**

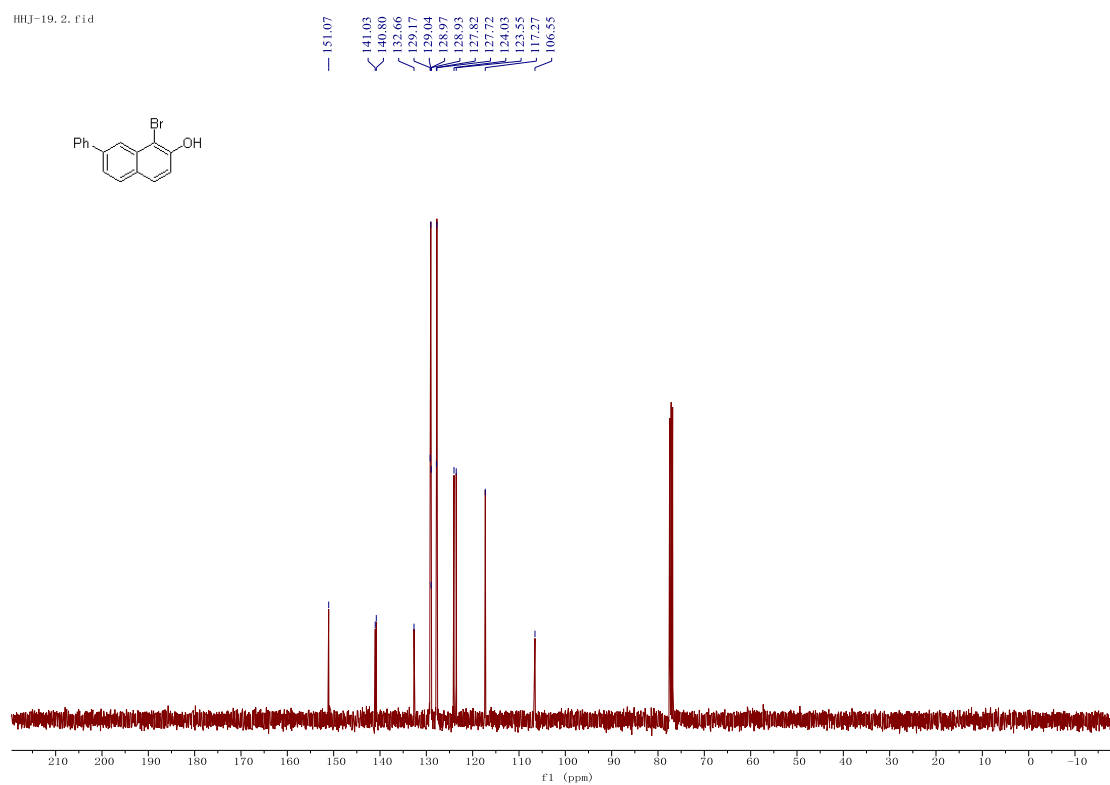


Figure S30. ^{13}C NMR Spectrum of **11**

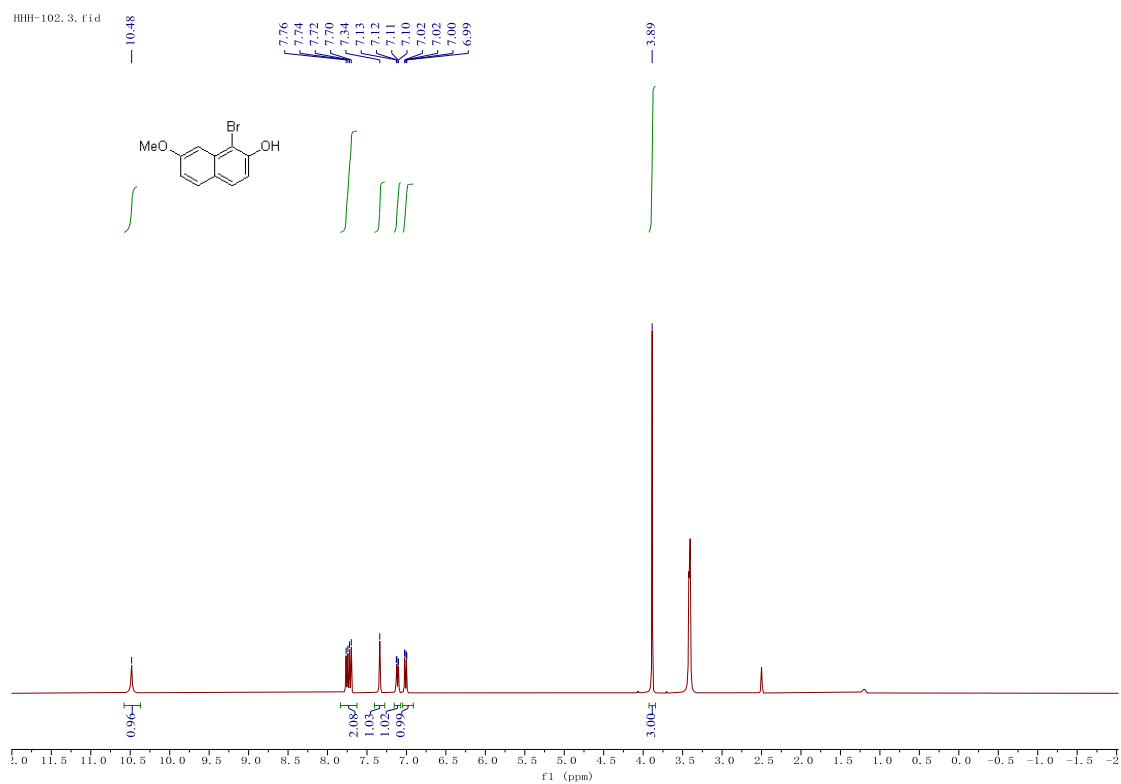


Figure S31. ^1H NMR Spectrum of **1m**

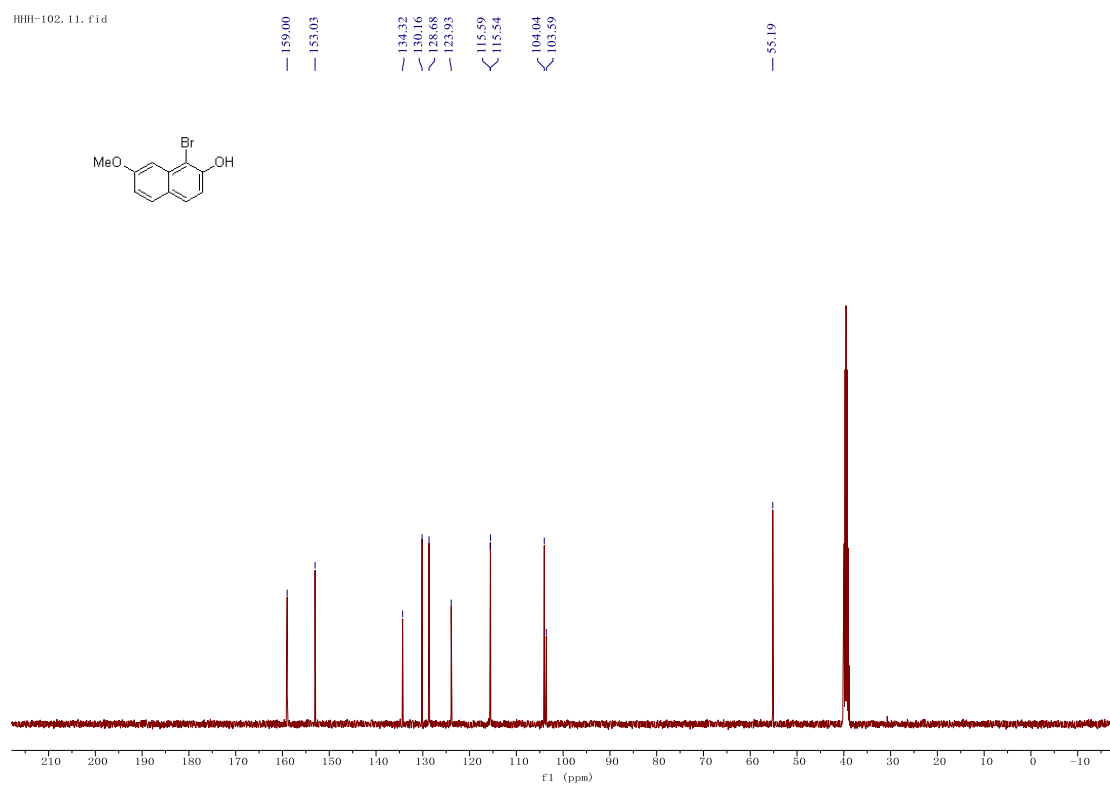


Figure S32. ^{13}C NMR Spectrum of **1m**

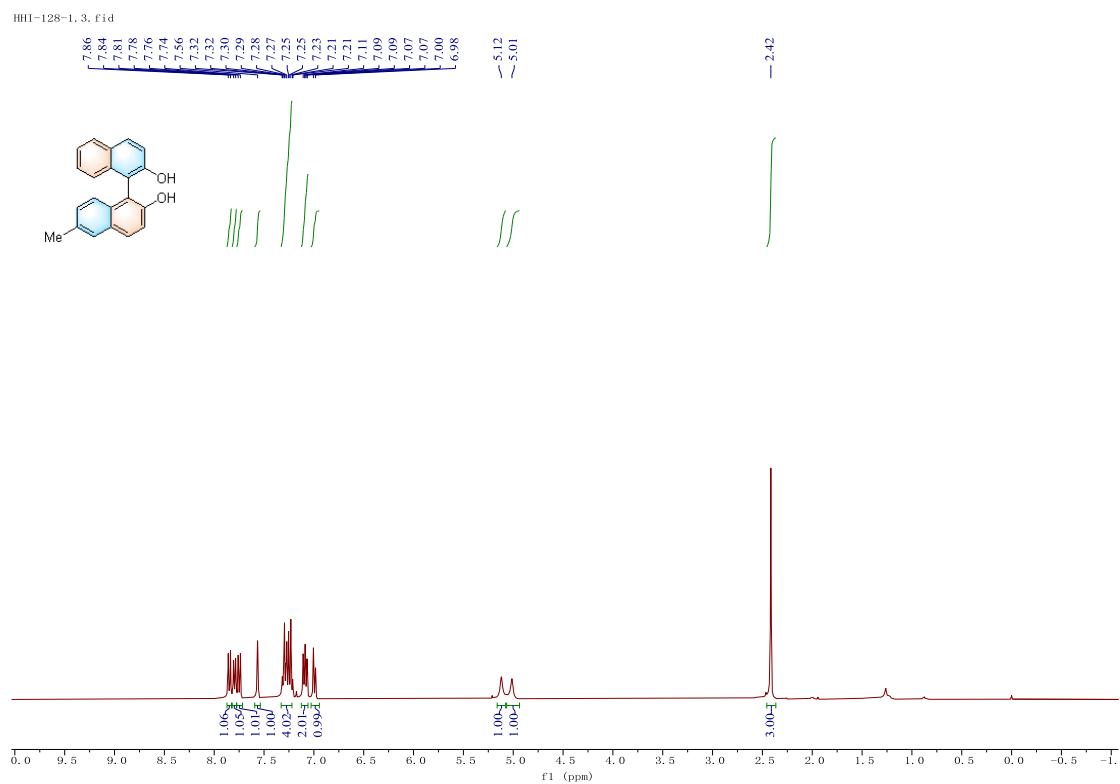


Figure S33. ^1H NMR Spectrum of 3a

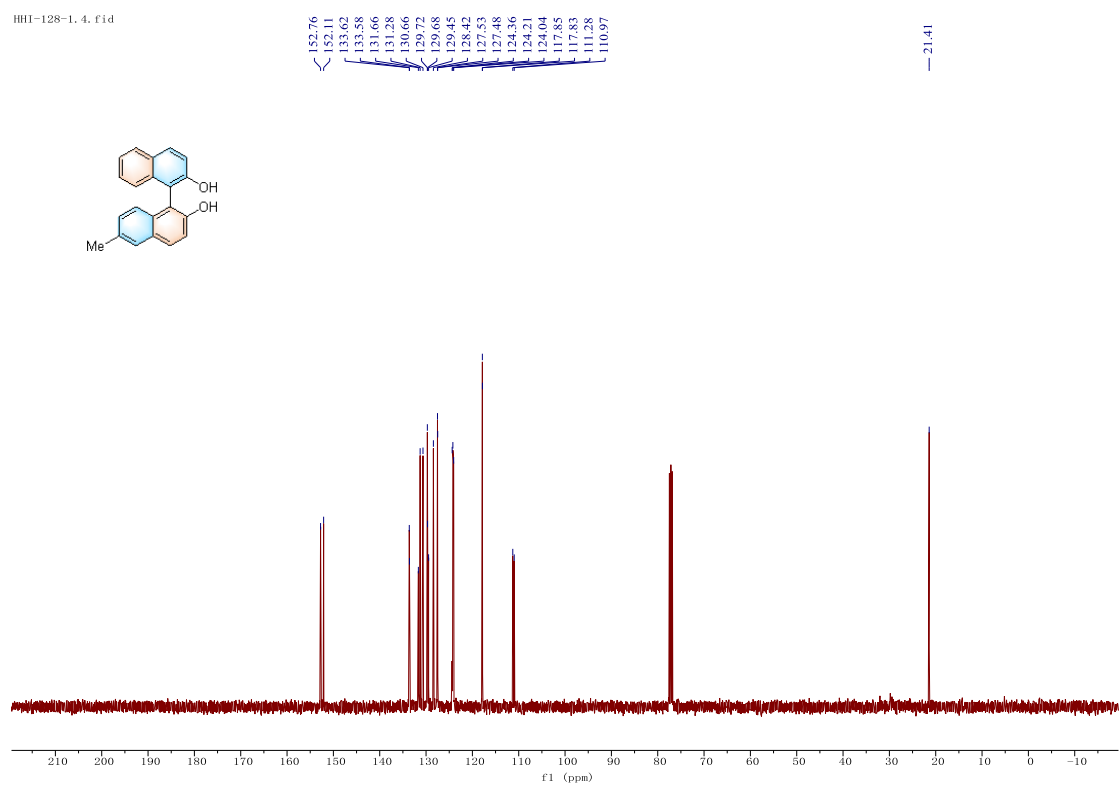


Figure S34. ^{13}C NMR Spectrum of 3a

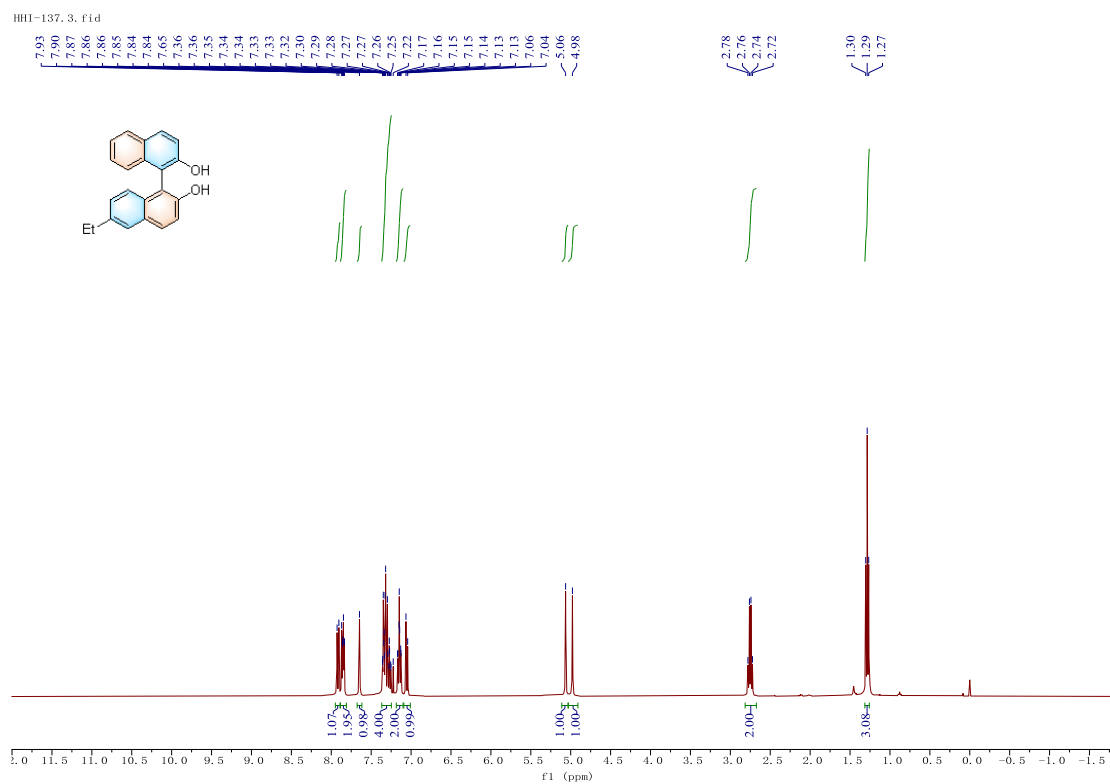


Figure S35. ^1H NMR Spectrum of 3b

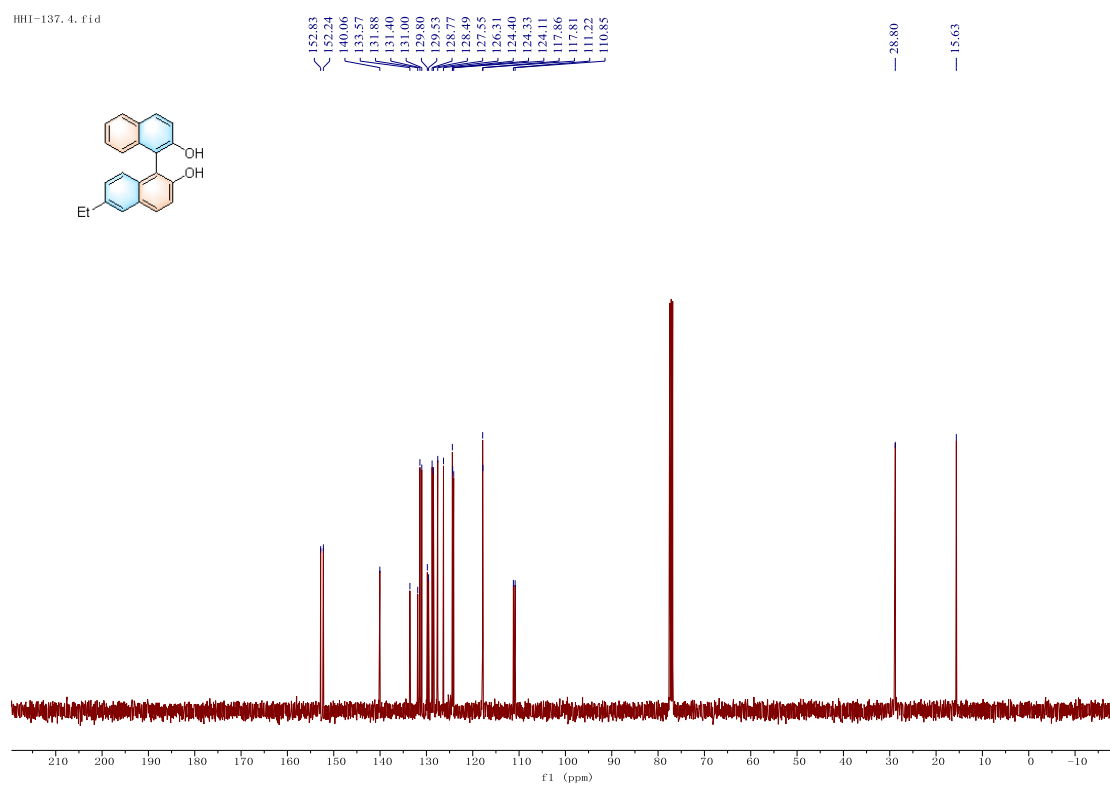


Figure S36. ^{13}C NMR Spectrum of 3b

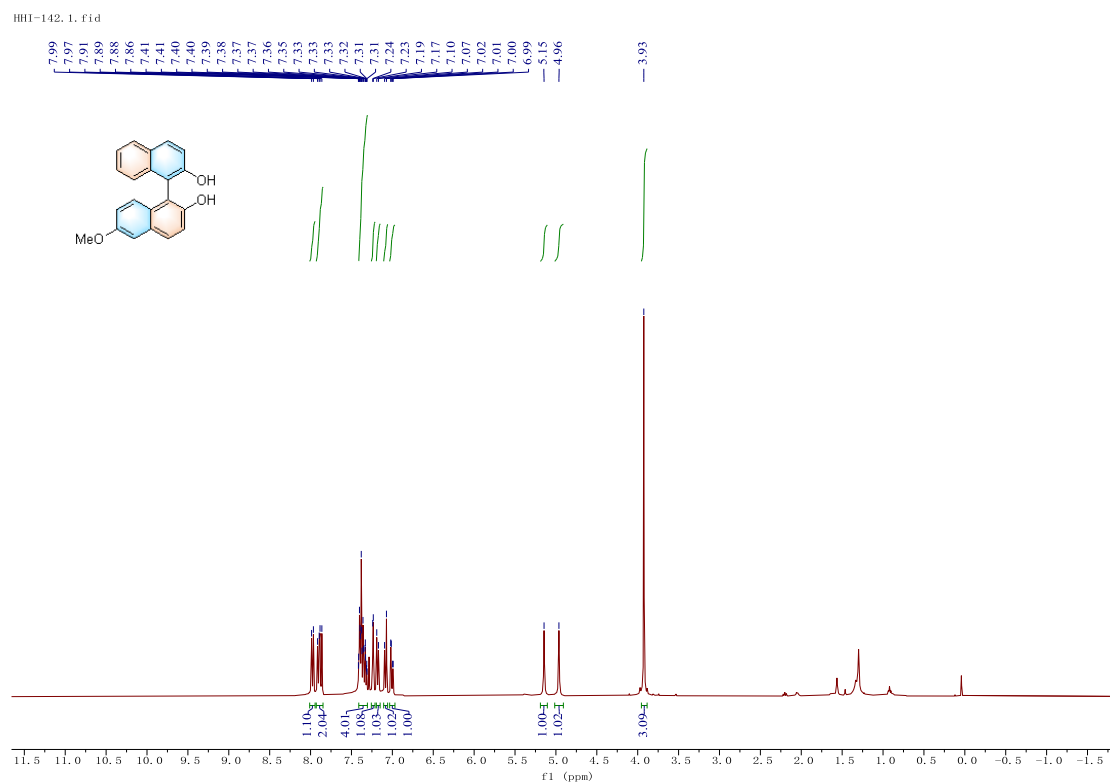


Figure S37. ^1H NMR Spectrum of **3c**

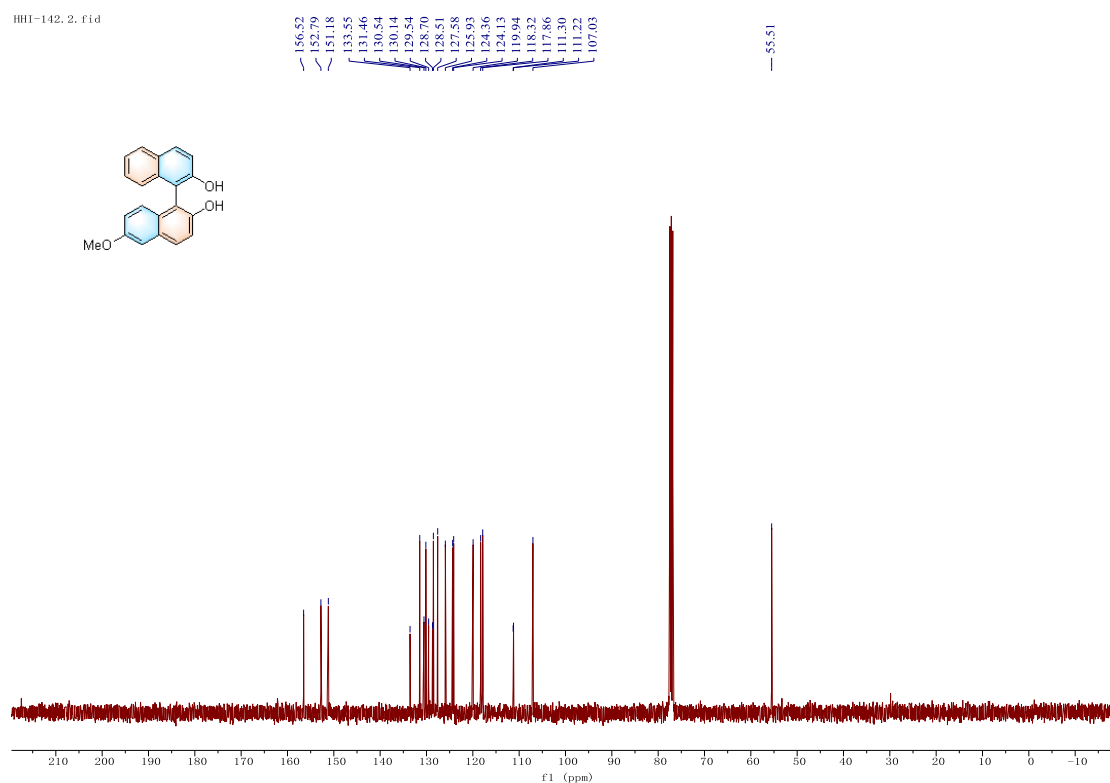


Figure S38. ^{13}C NMR Spectrum of **3c**

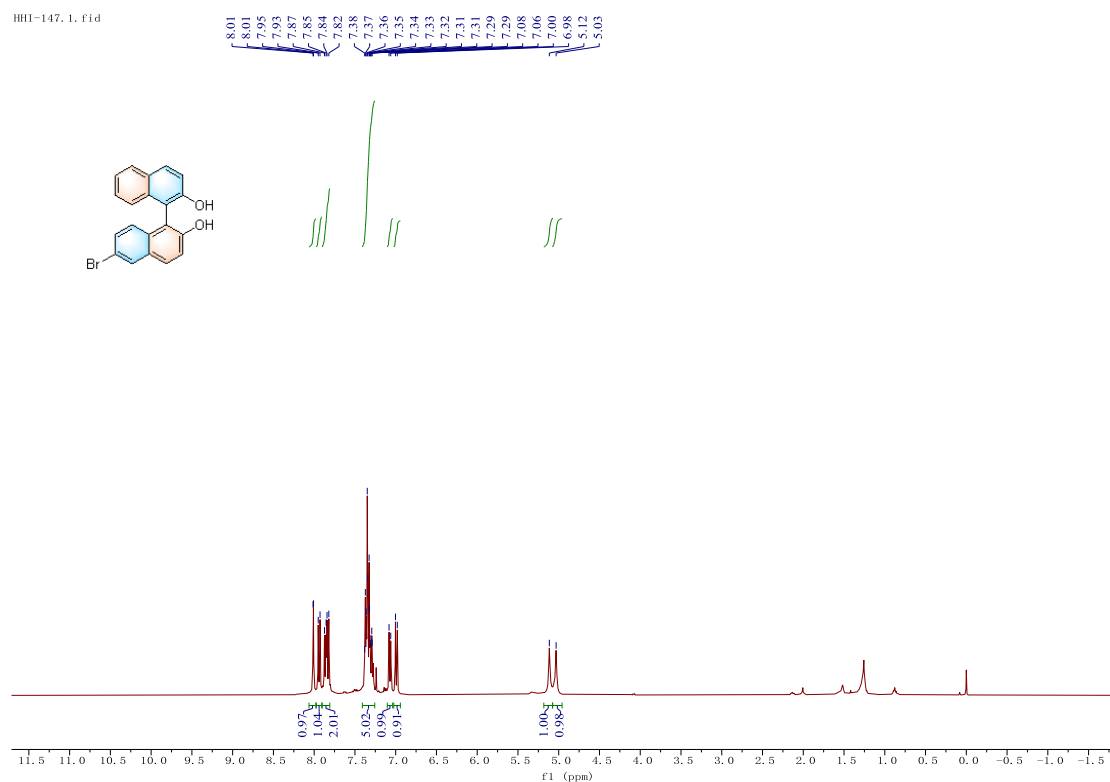


Figure S39. ^1H NMR Spectrum of 3d

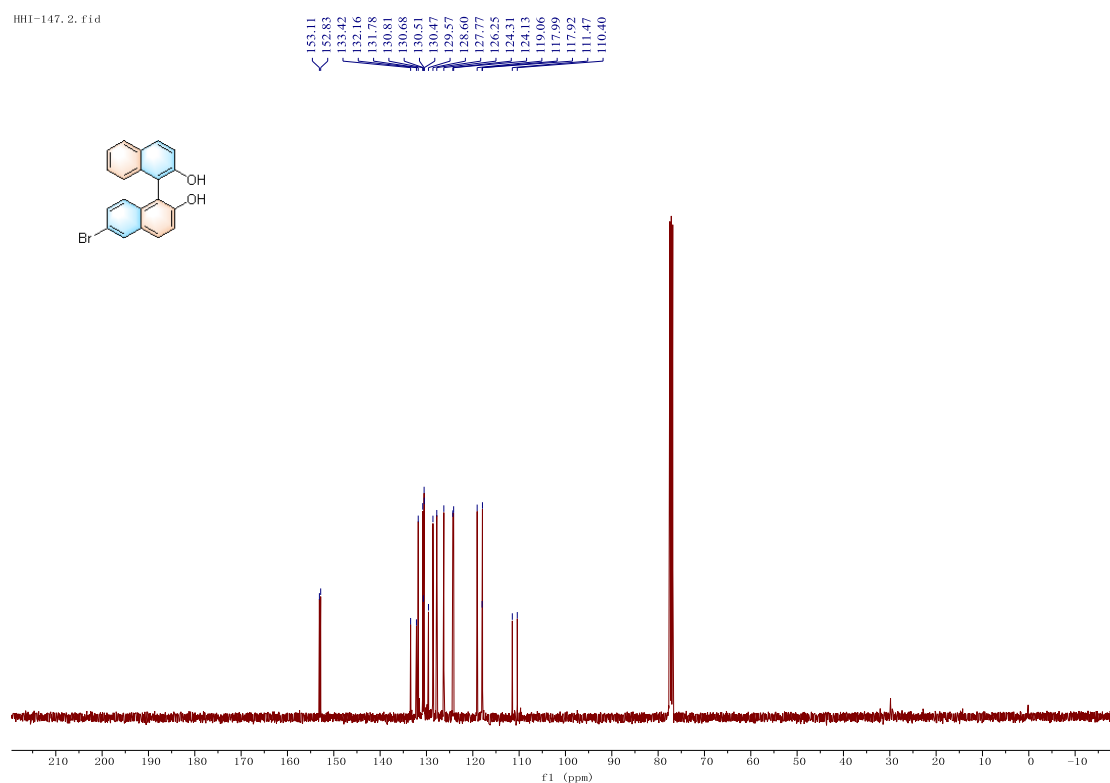


Figure S40. ^{13}C NMR Spectrum of 3d

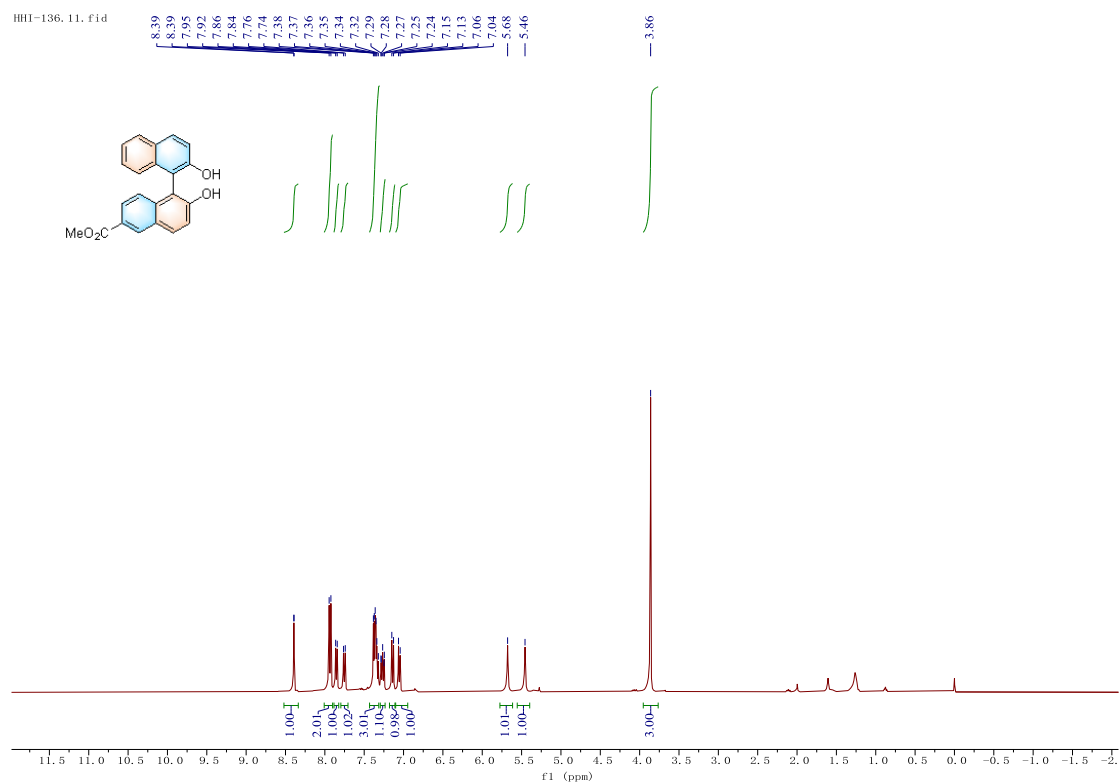


Figure S41. ^1H NMR Spectrum of **3e**

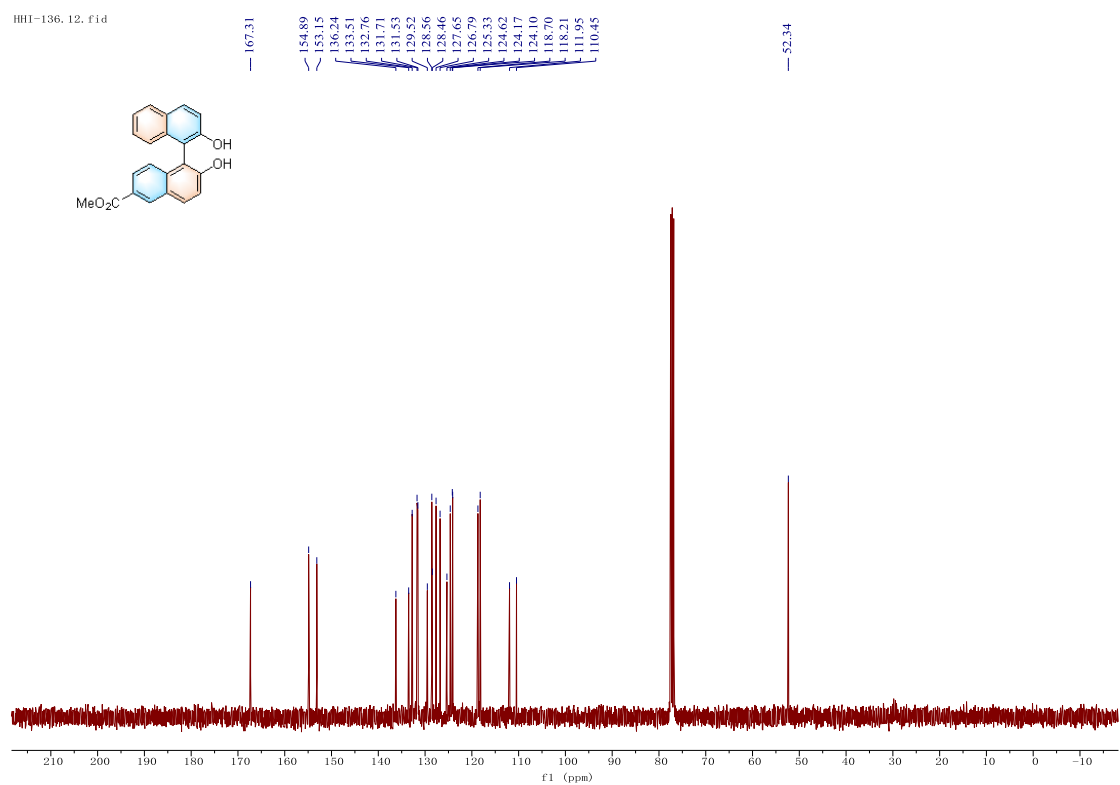


Figure S42. ^{13}C NMR Spectrum of **3e**

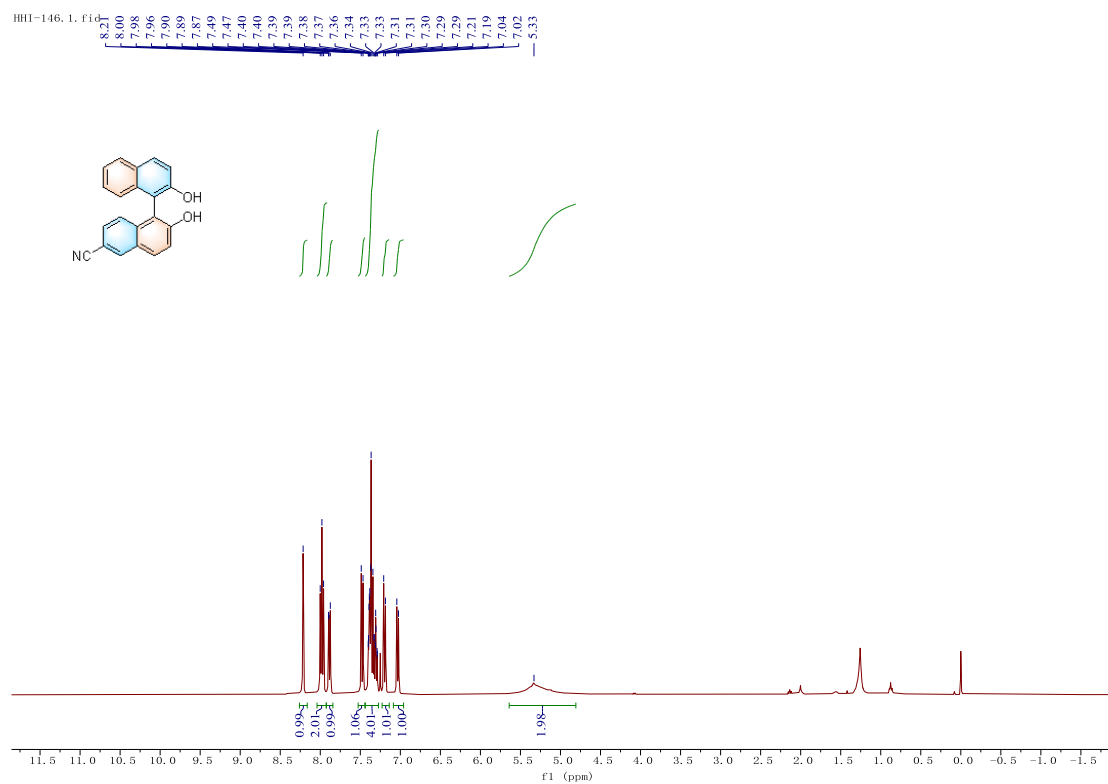


Figure S43. ^1H NMR Spectrum of **3f**

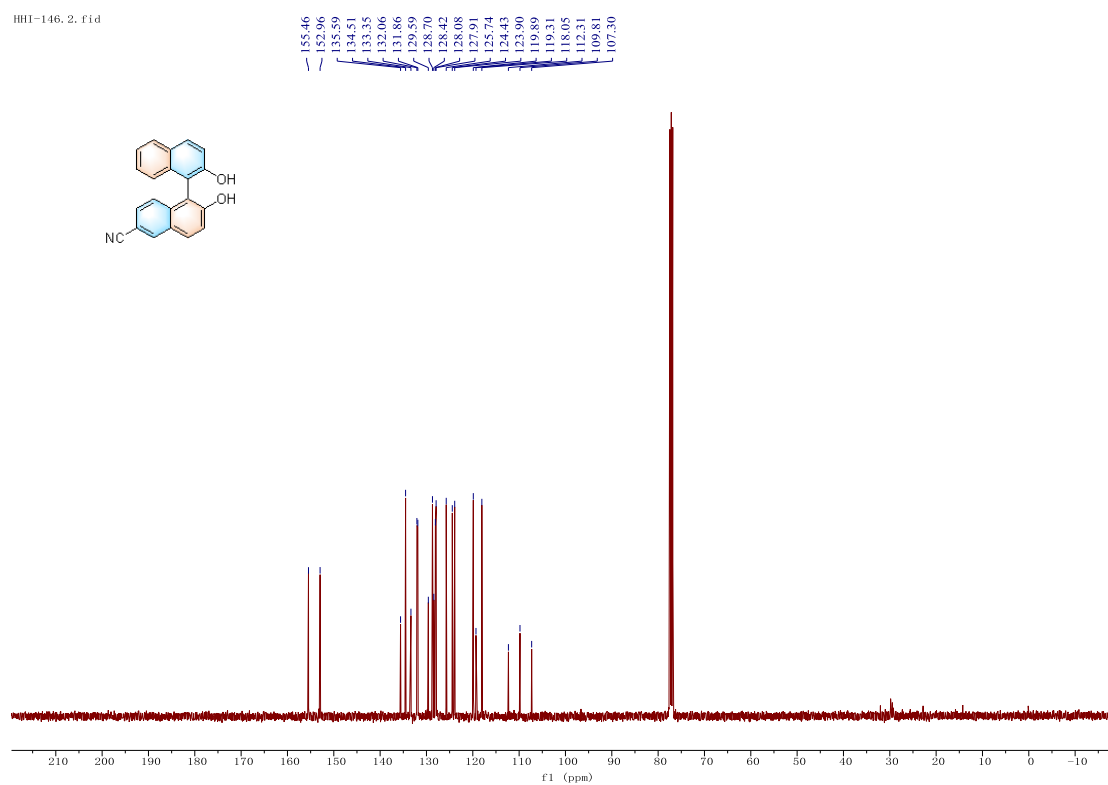


Figure S44. ^{13}C NMR Spectrum of **3f**

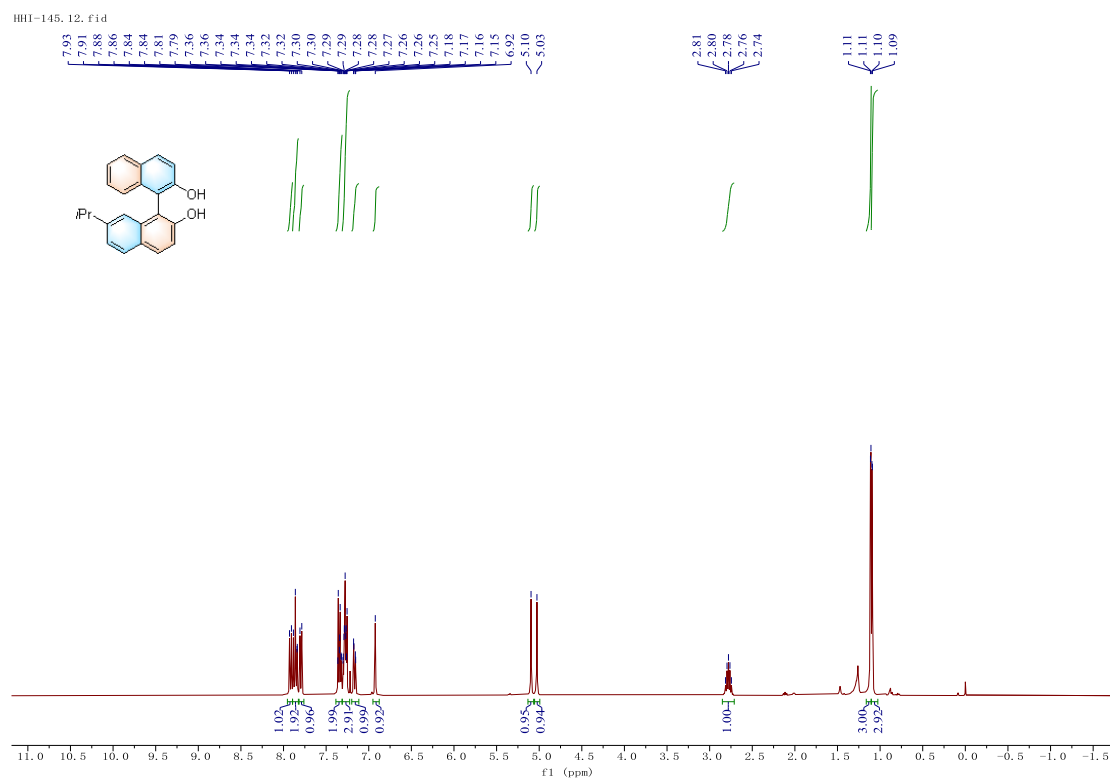


Figure S45. ^1H NMR Spectrum of **3g**

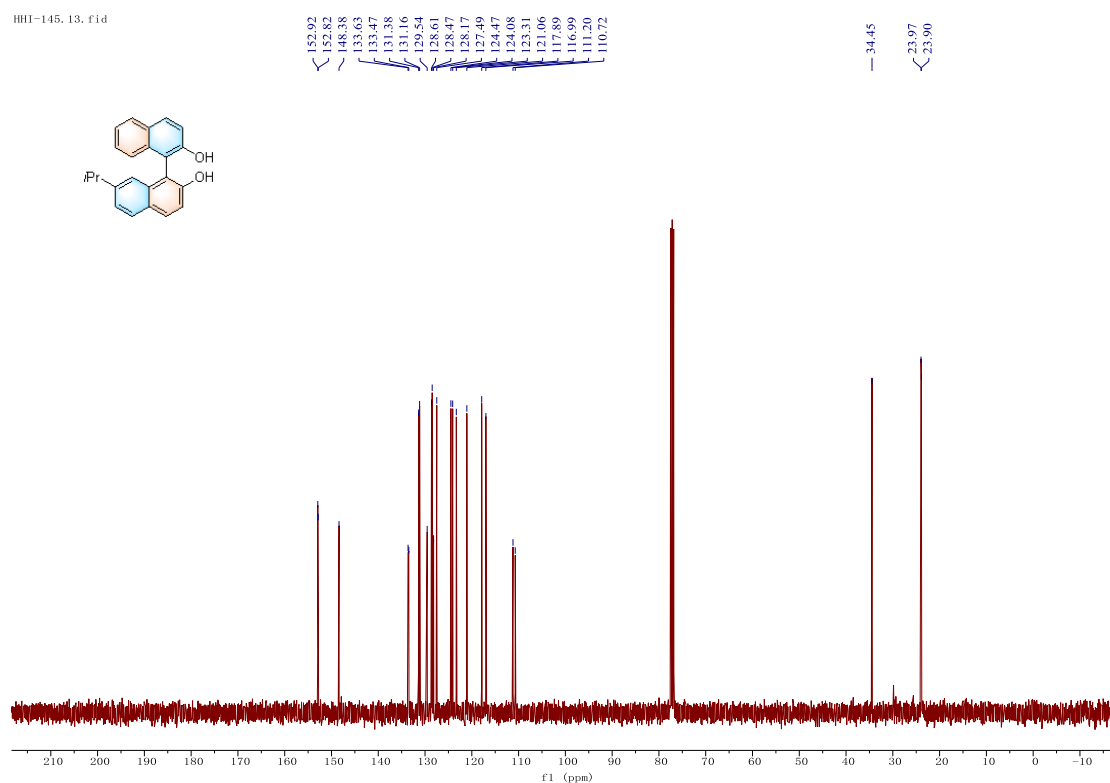


Figure S46. ^{13}C NMR Spectrum of **3g**

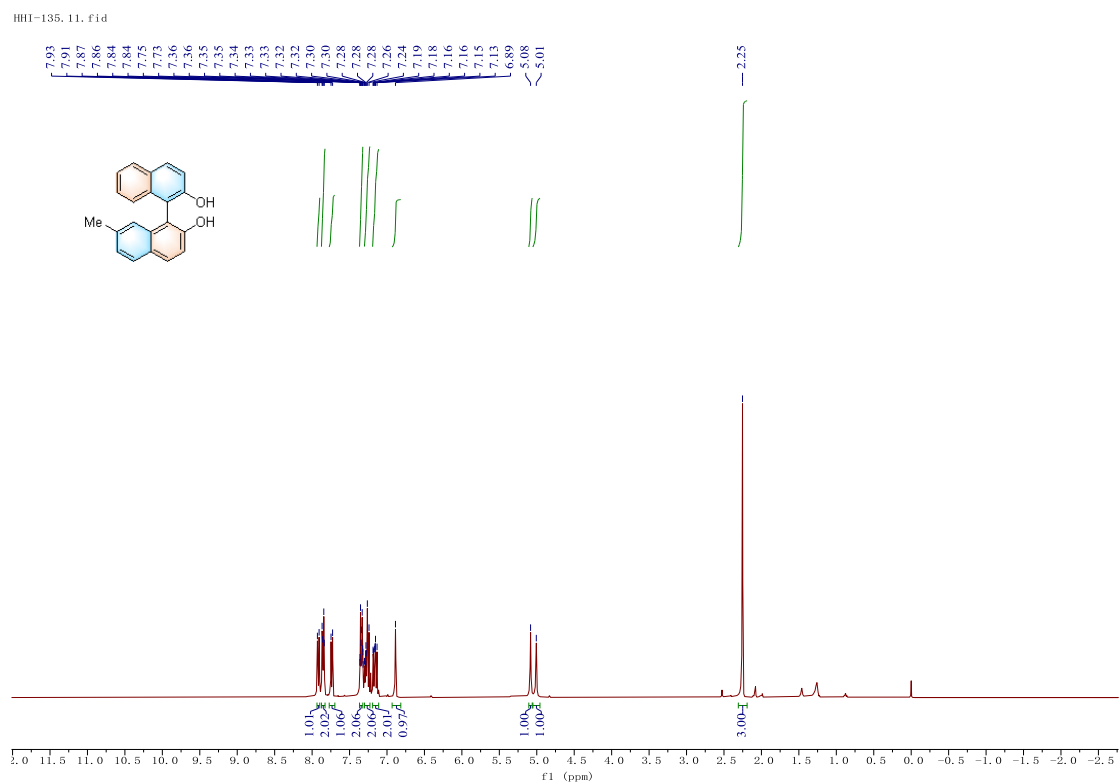


Figure S47. ^1H NMR Spectrum of 3h

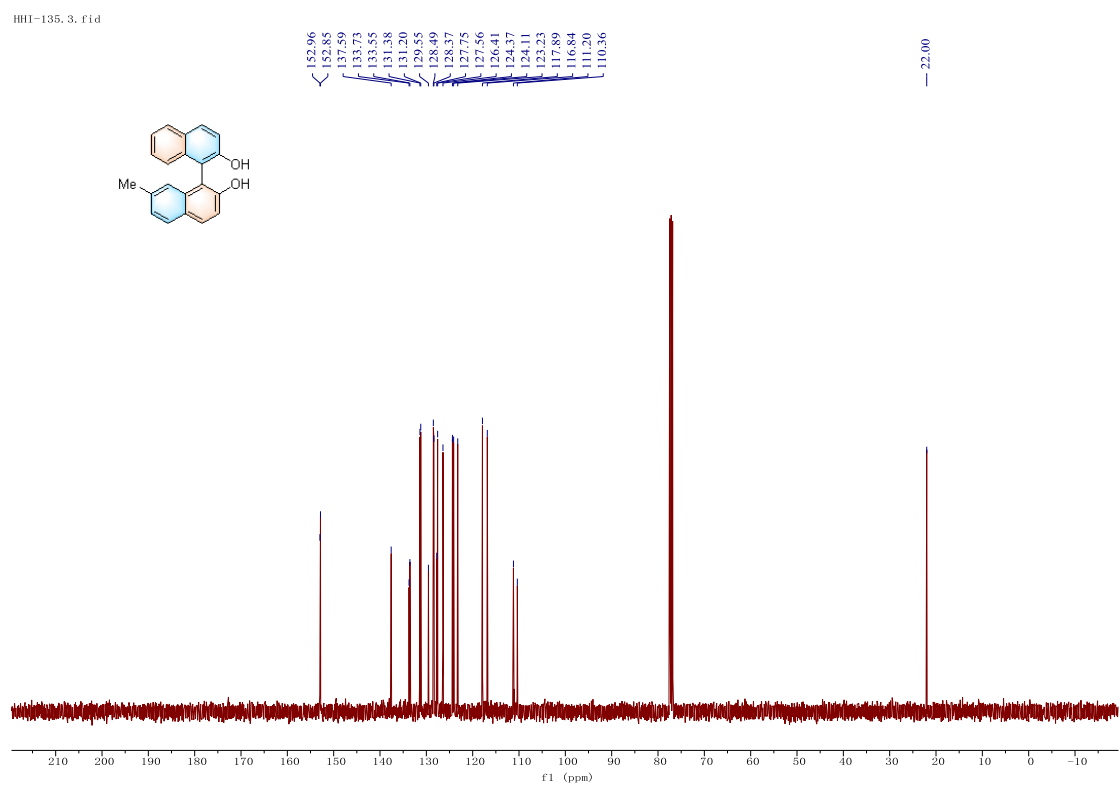


Figure S48. ^{13}C NMR Spectrum of 3h

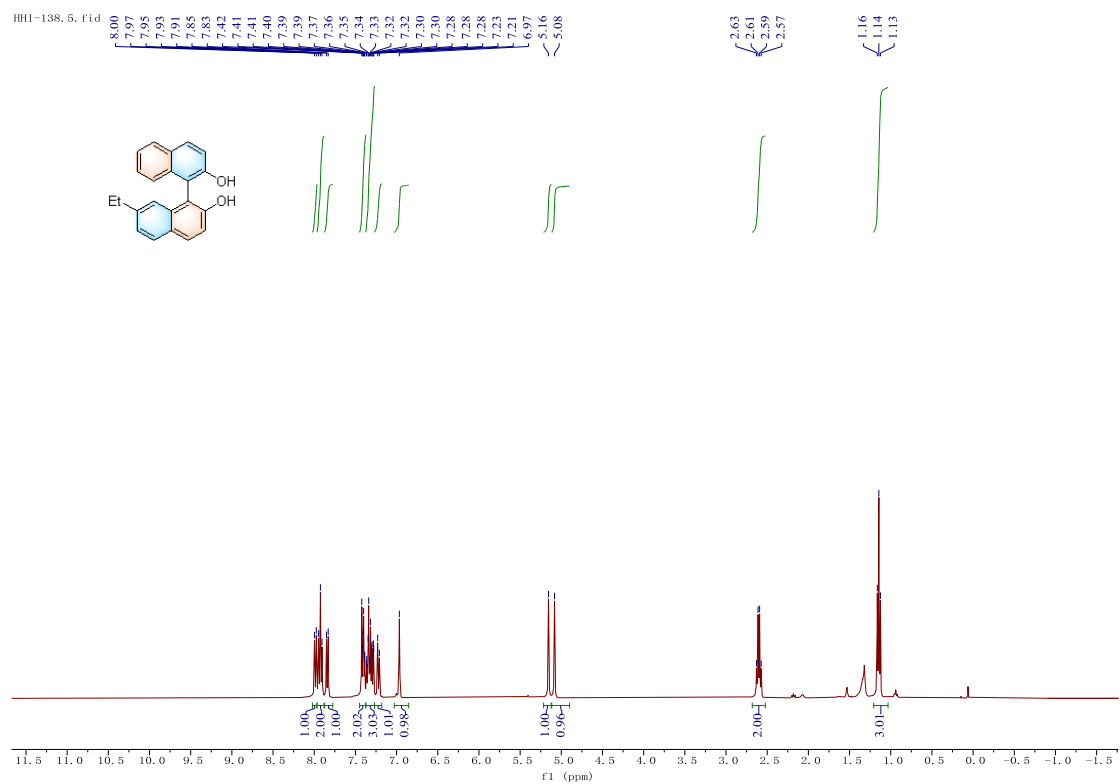


Figure S49. ^1H NMR Spectrum of **3i**

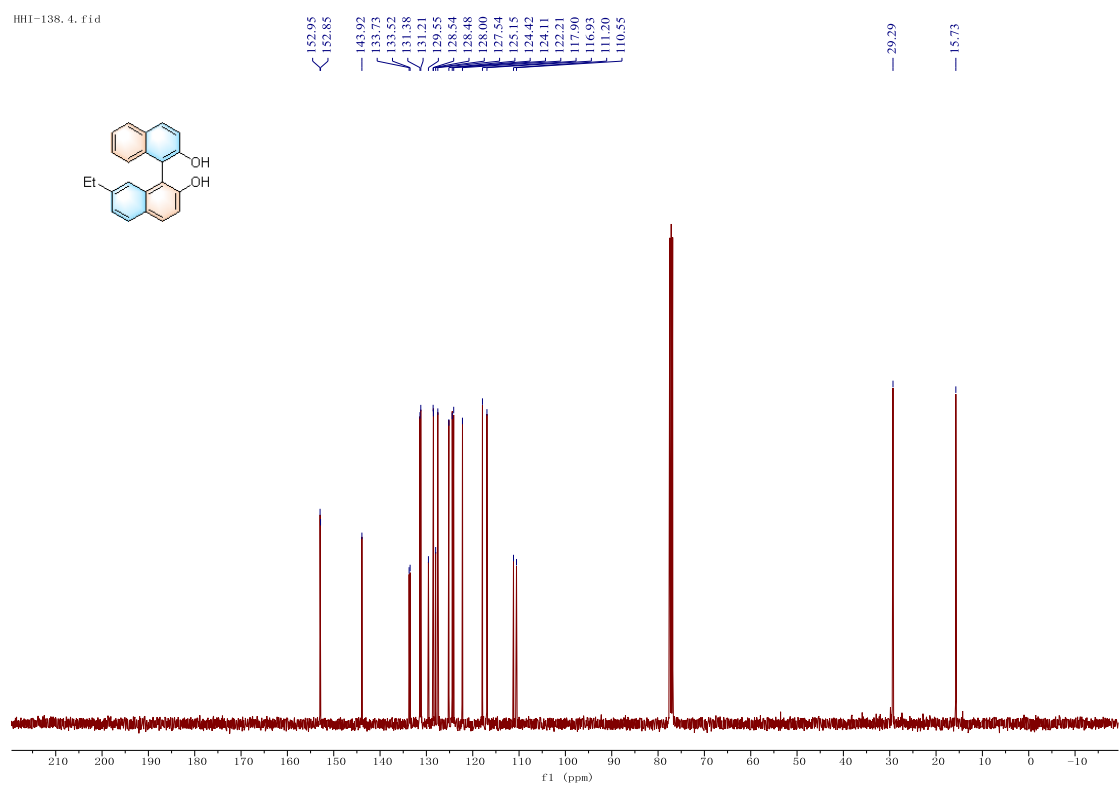
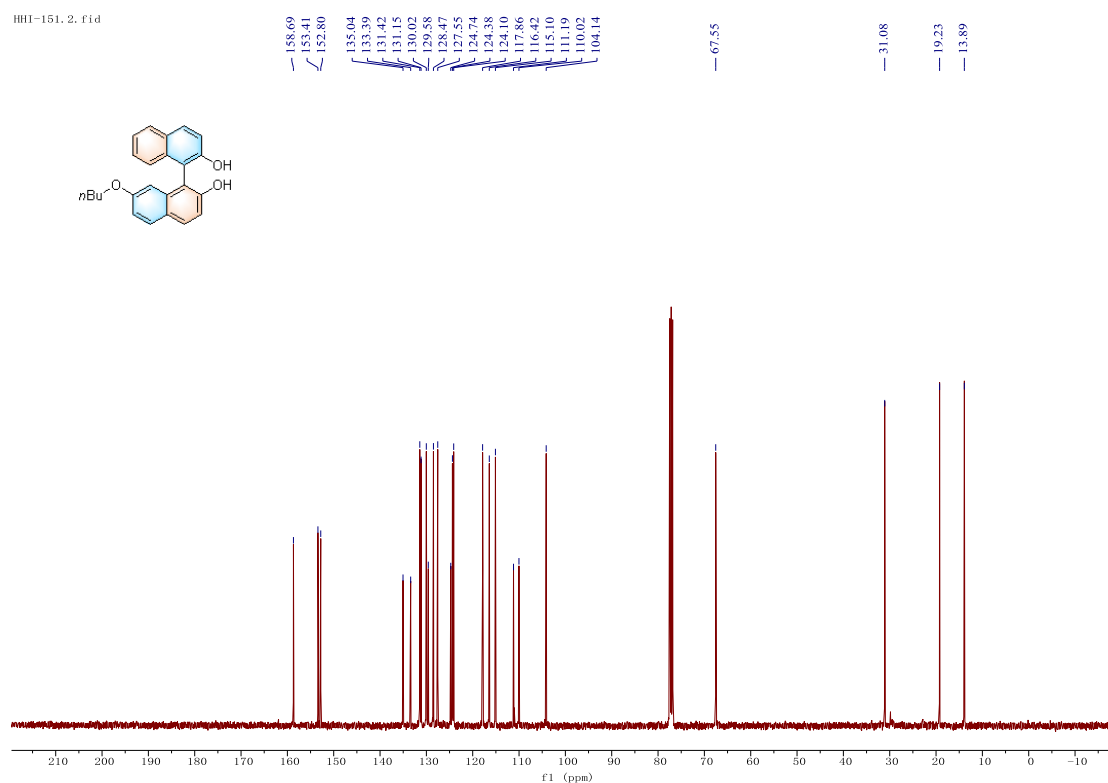
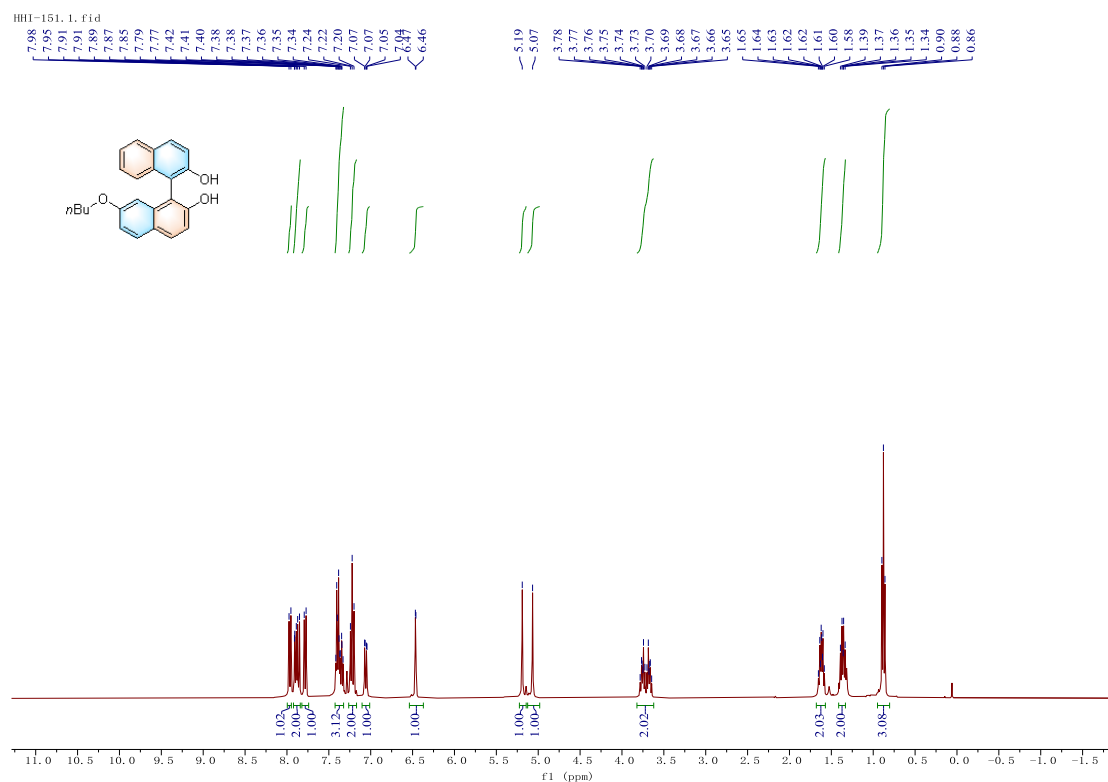


Figure S50. ^{13}C NMR Spectrum of **3i**



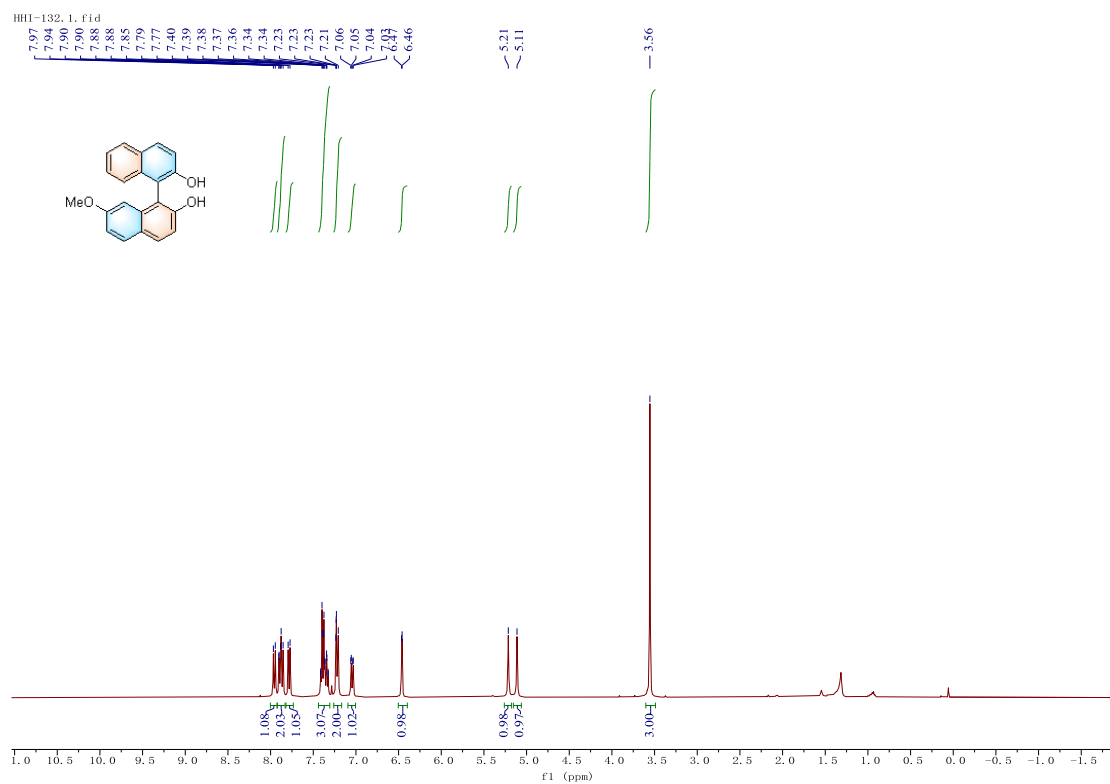


Figure S53. ^1H NMR Spectrum of 3k

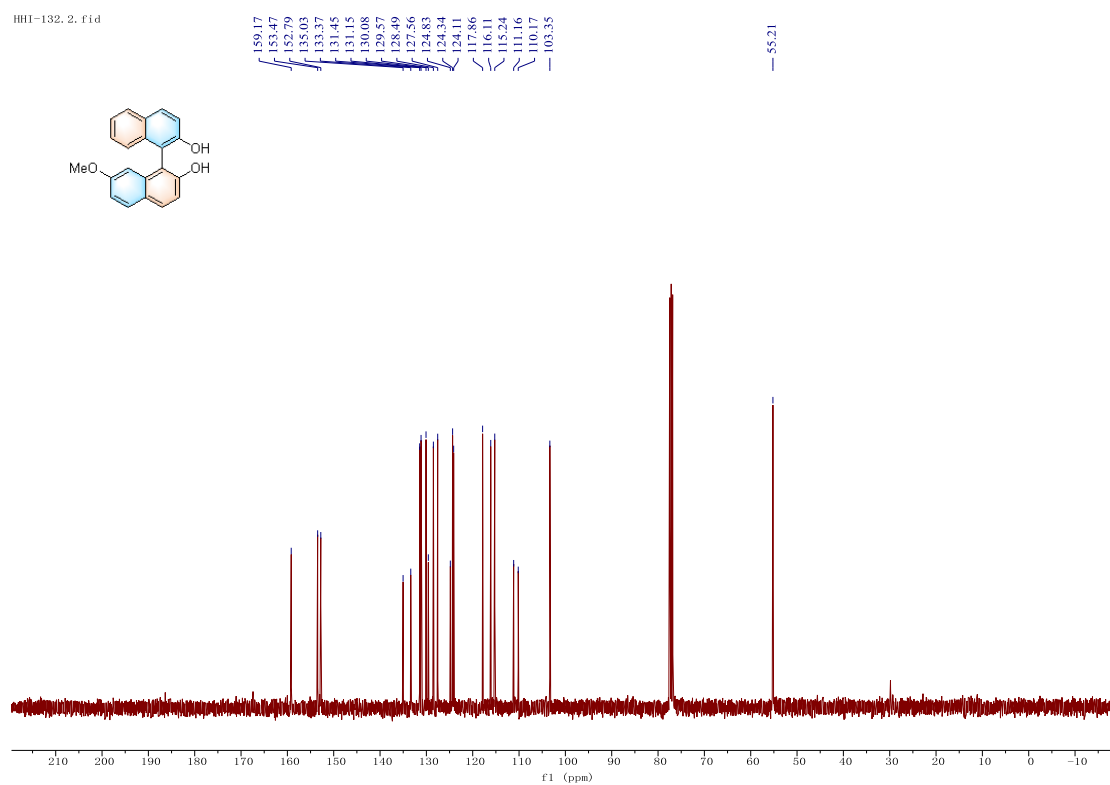
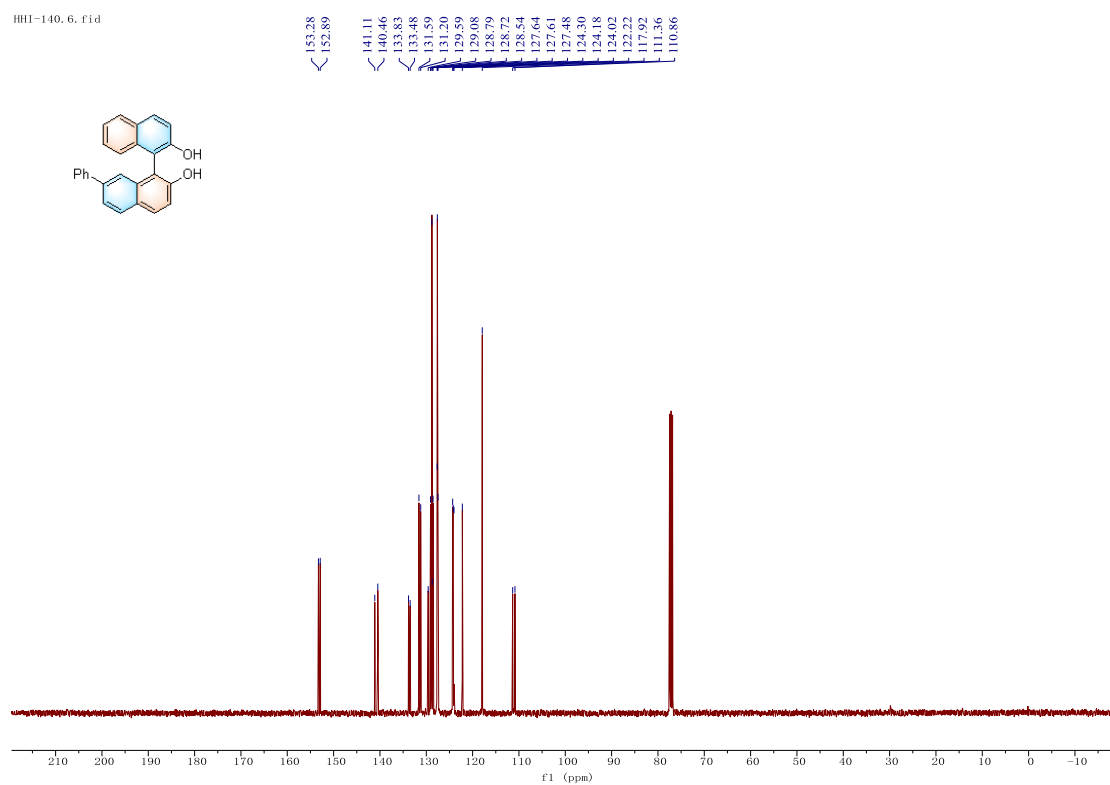
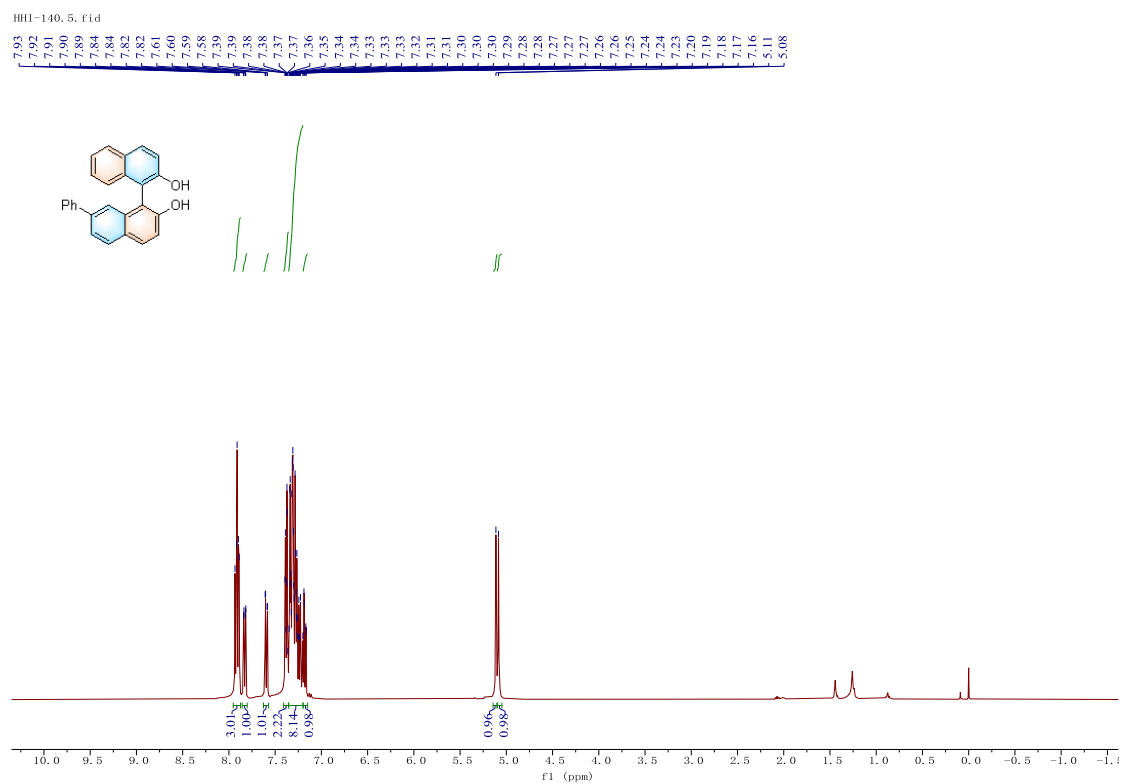
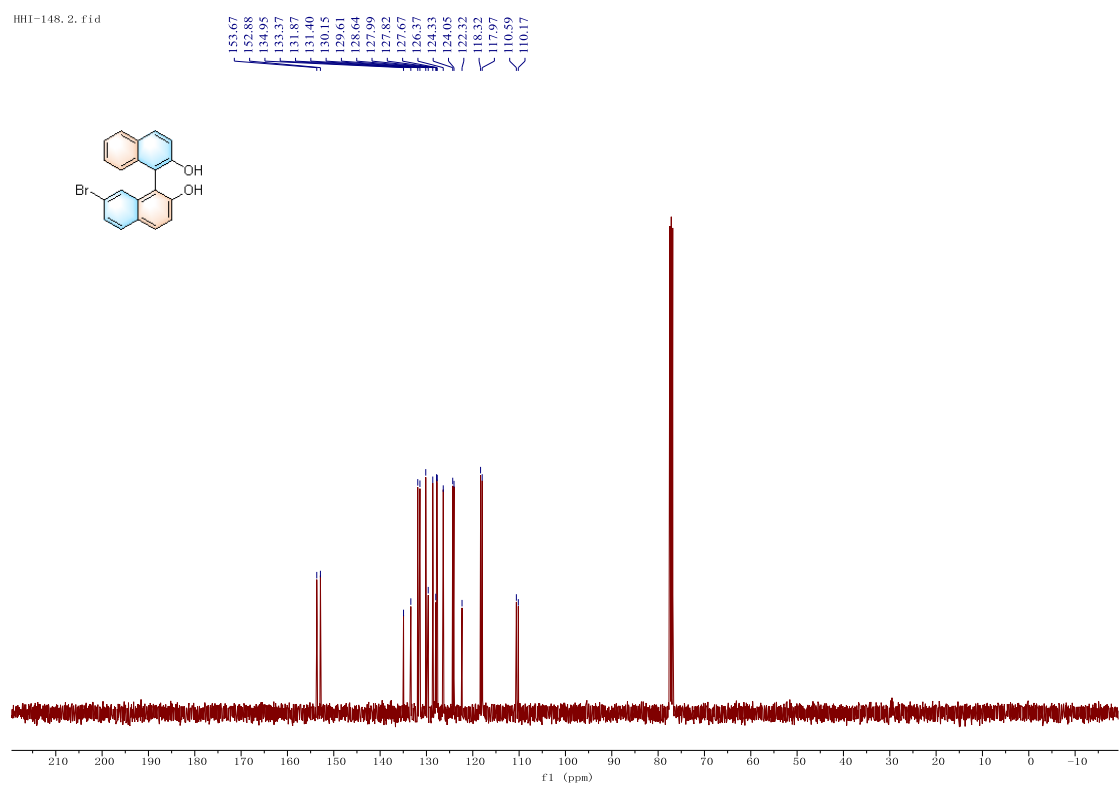
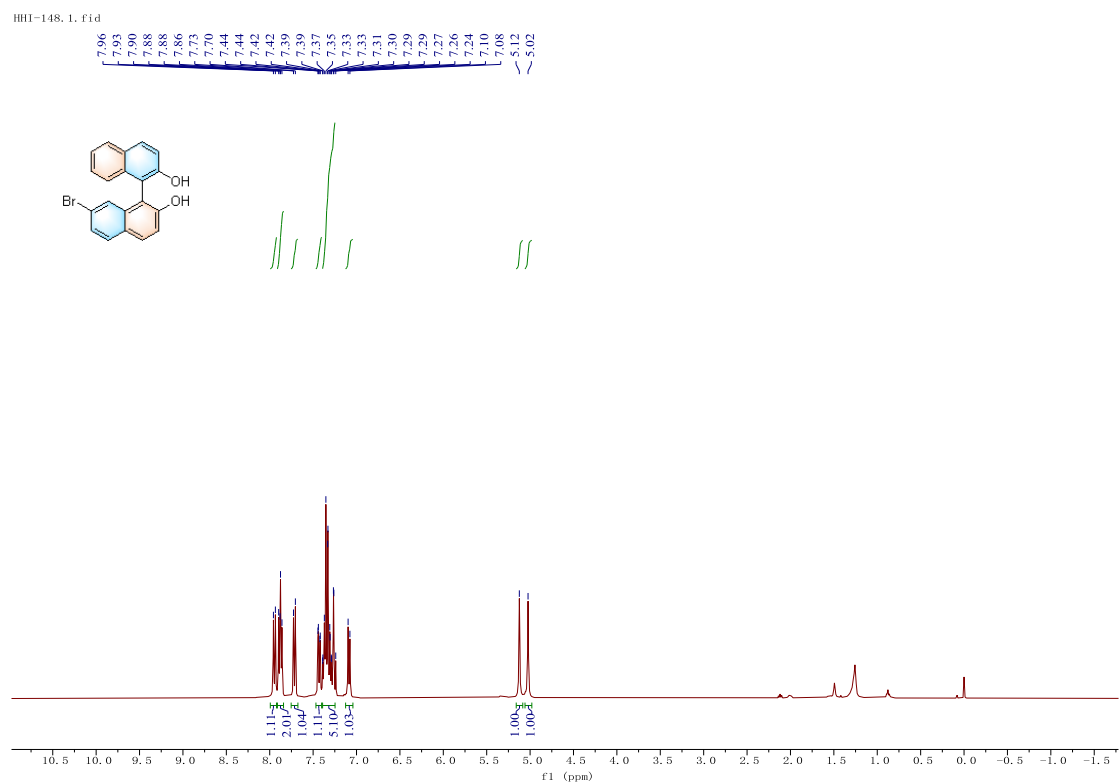


Figure S54. ^{13}C NMR Spectrum of 3k





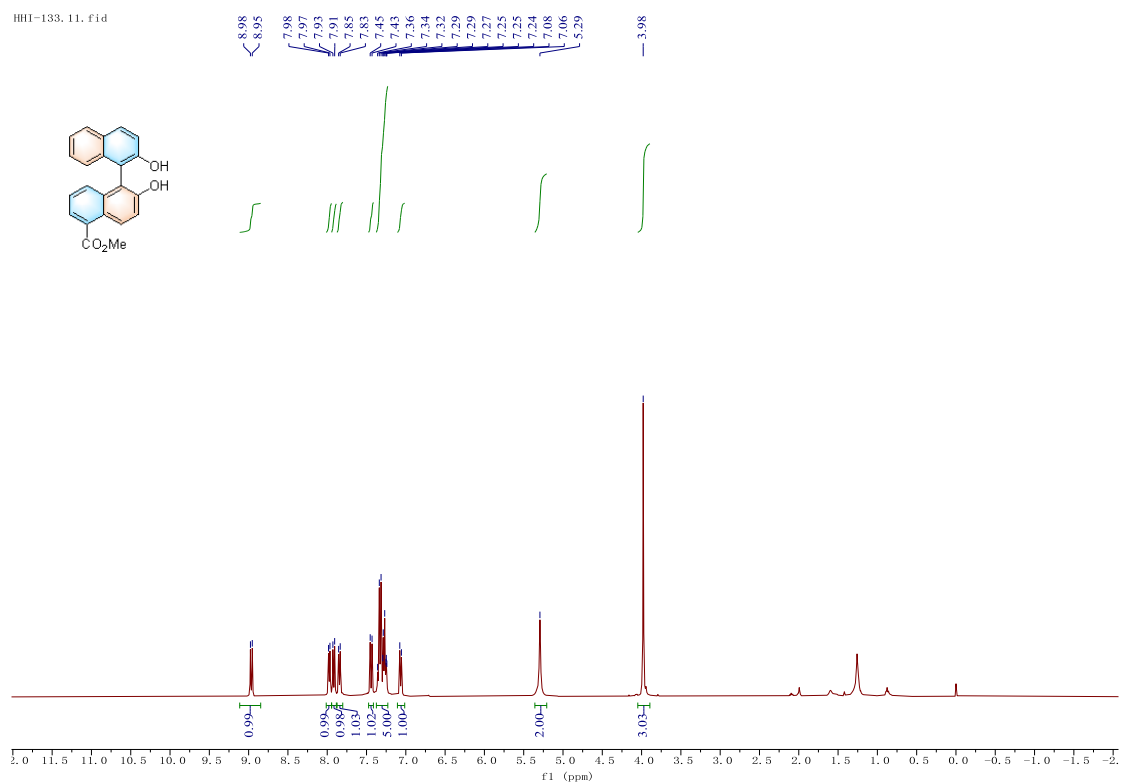


Figure S59. ¹H NMR Spectrum of **3n**

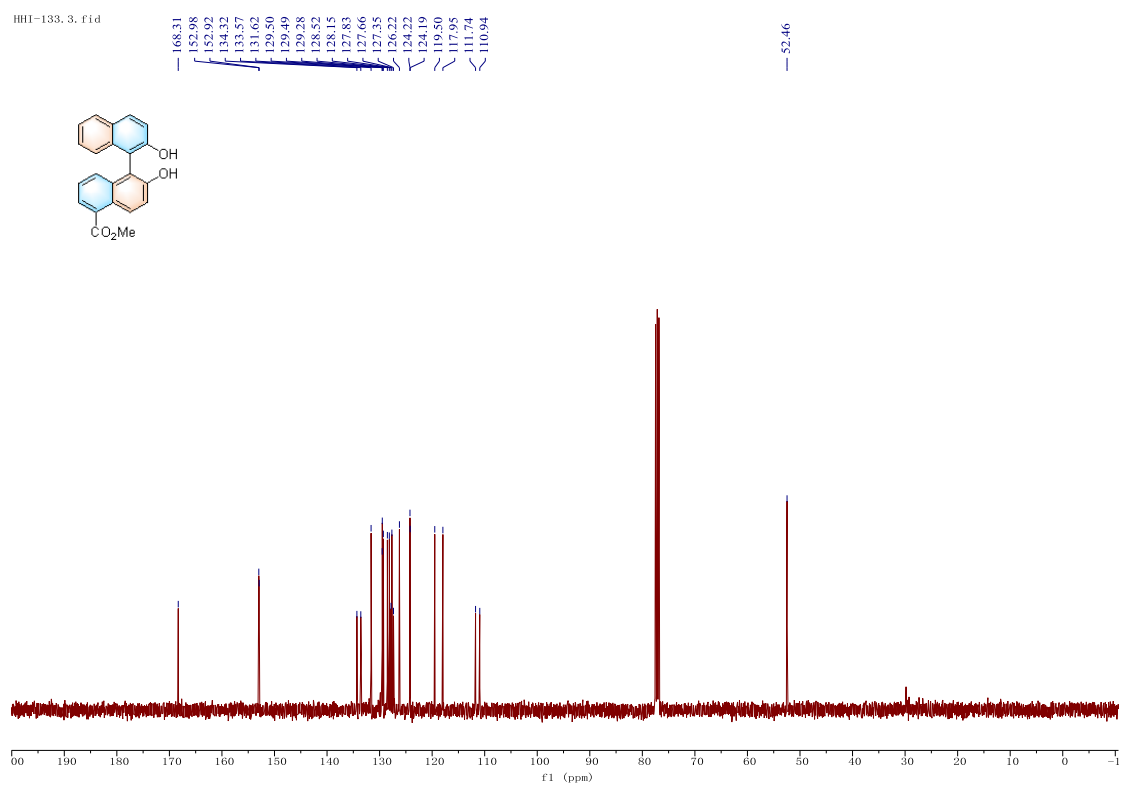


Figure S60. ¹³C NMR Spectrum of **3n**

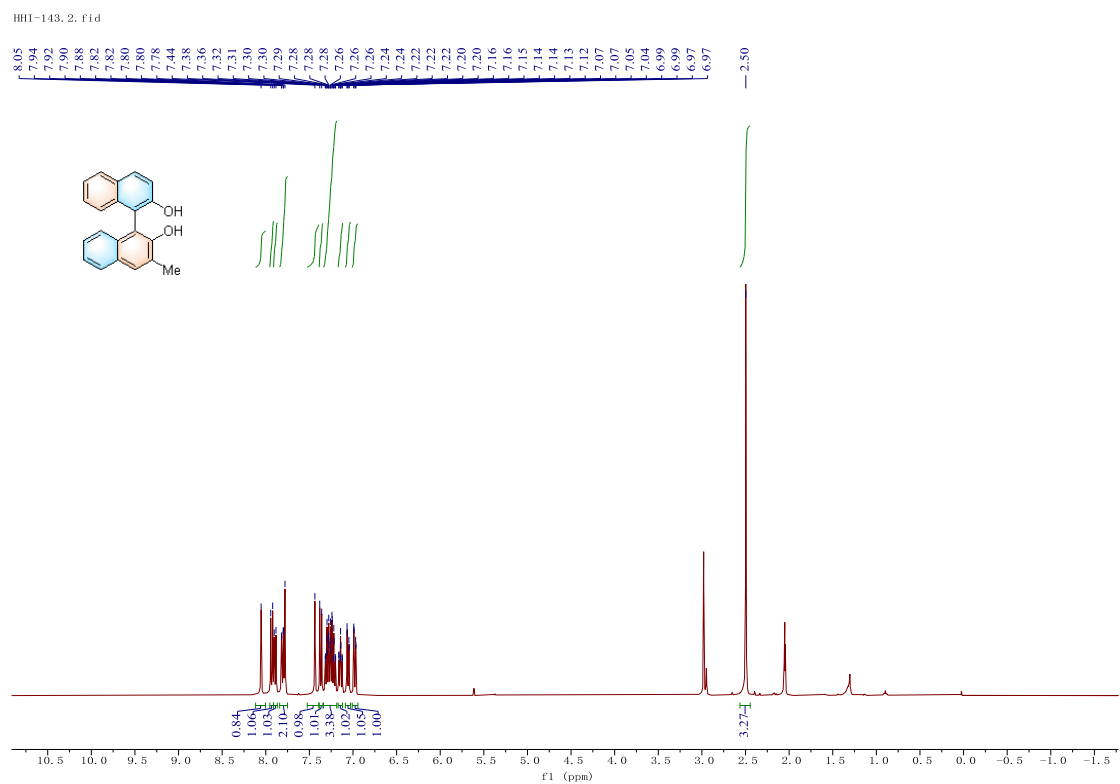


Figure S61. ^1H NMR Spectrum of **3o**

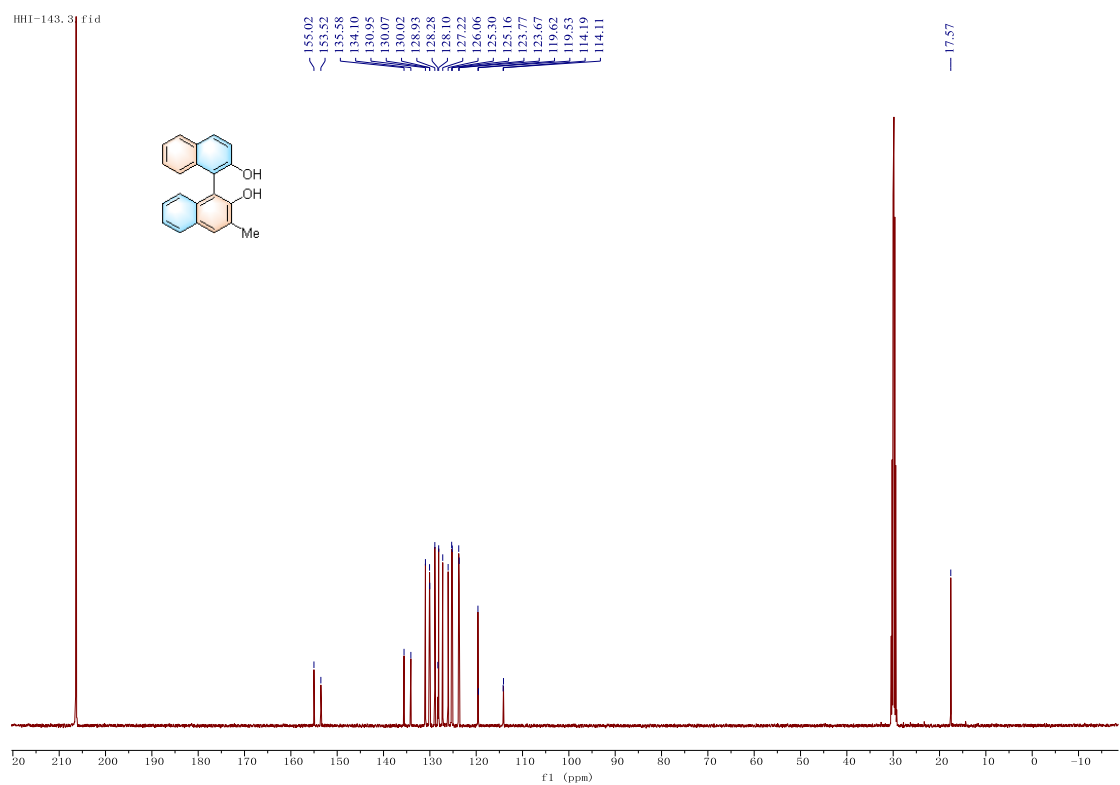


Figure S62. ^{13}C NMR Spectrum of **3o**

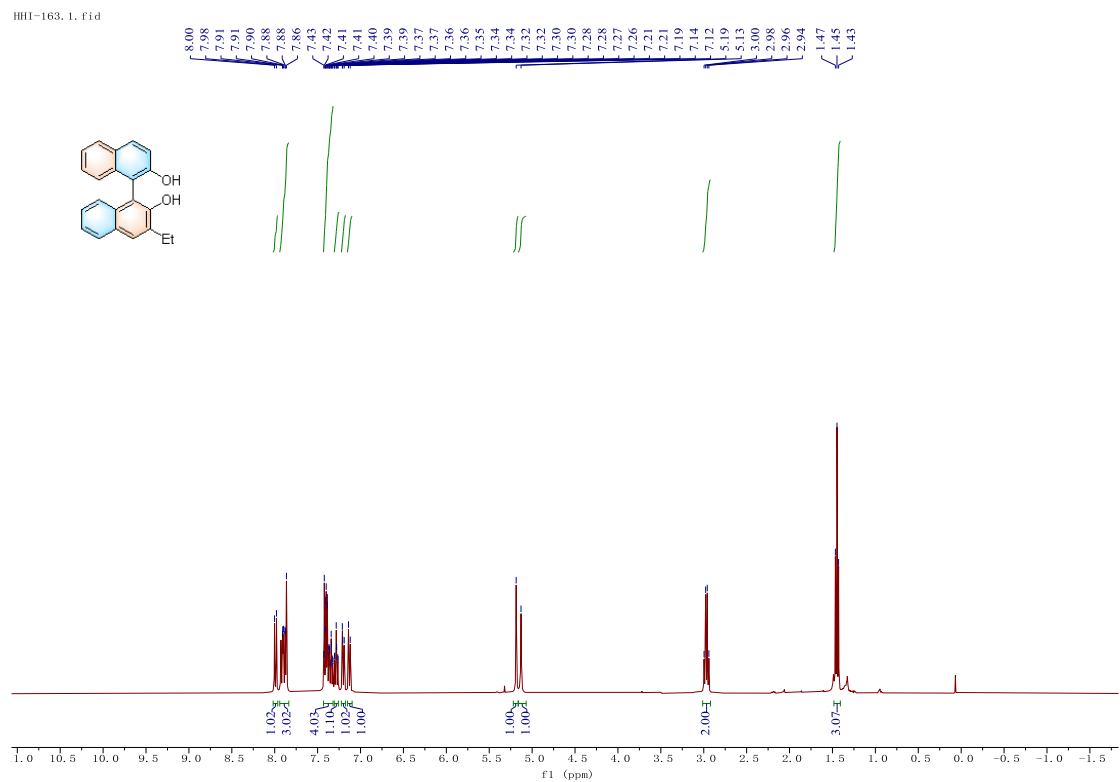


Figure S63. ^1H NMR Spectrum of 3p

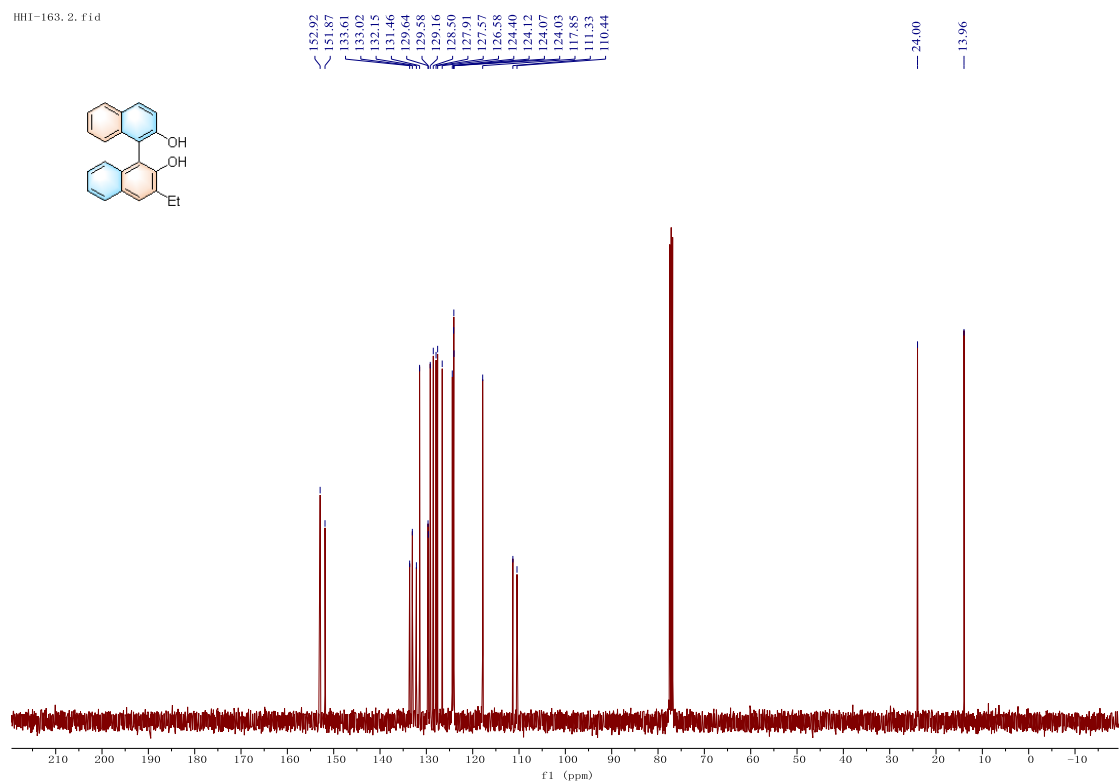


Figure S64. ^{13}C NMR Spectrum of 3p

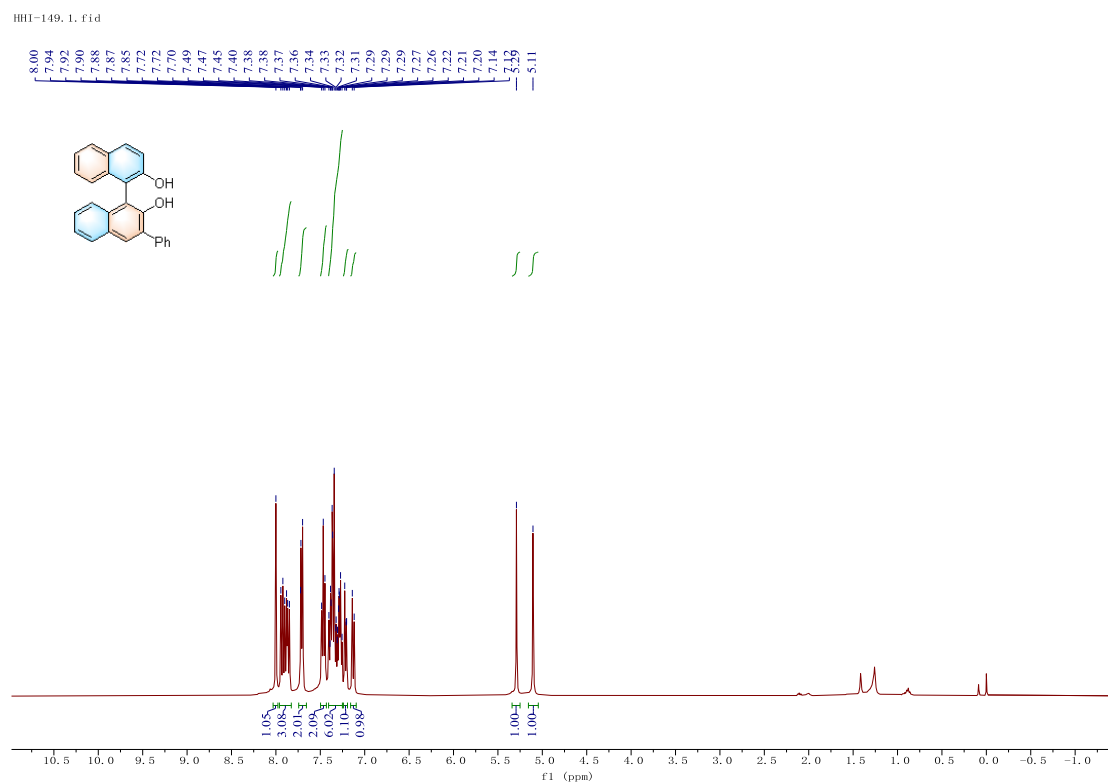


Figure S65. ¹H NMR Spectrum of **3q**

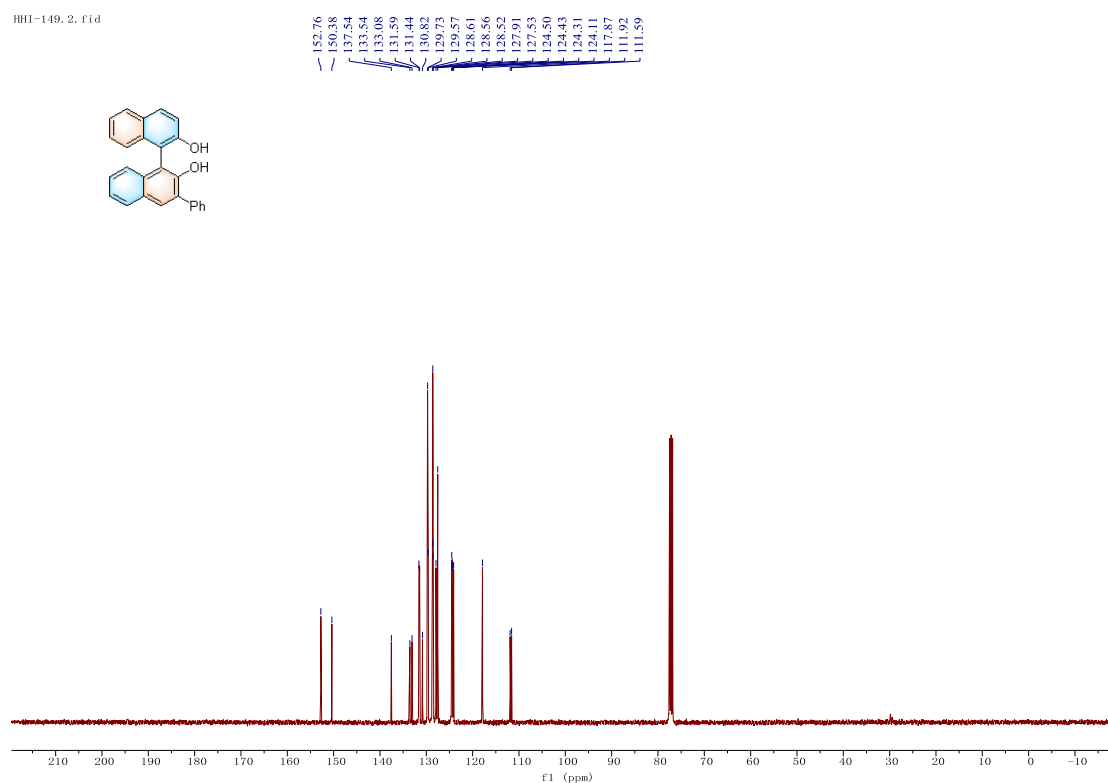


Figure S66. ¹³C NMR Spectrum of **3q**

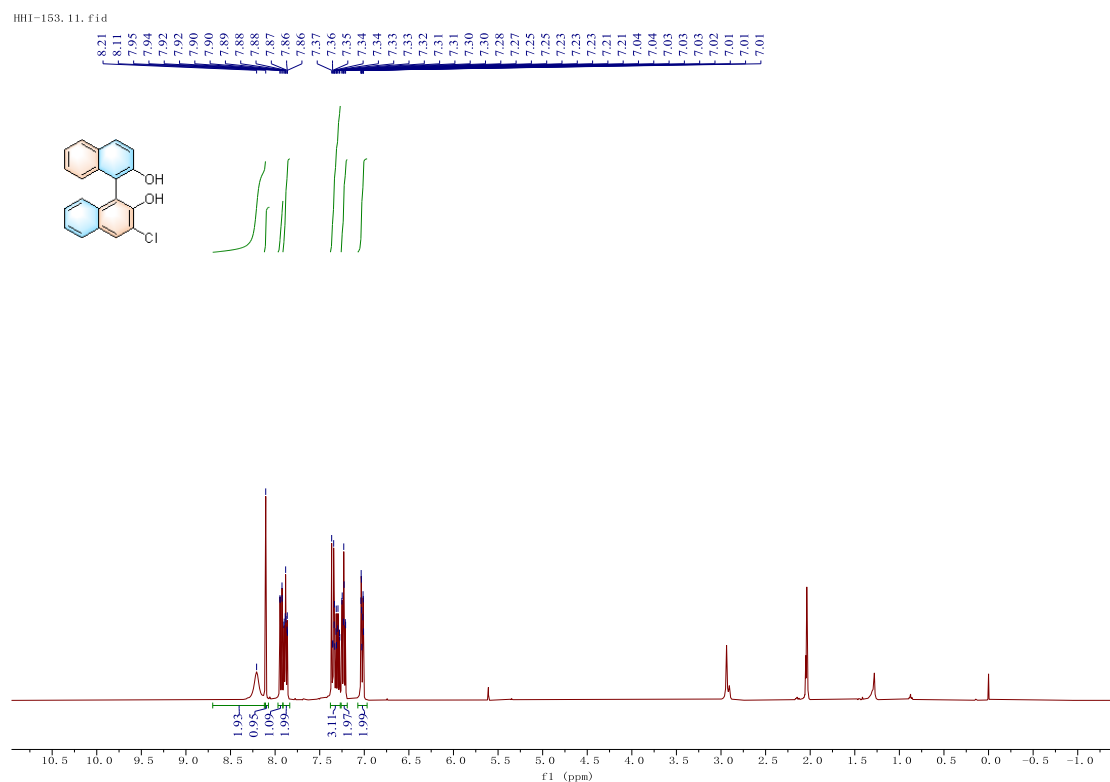


Figure S67. ^1H NMR Spectrum of **3r**

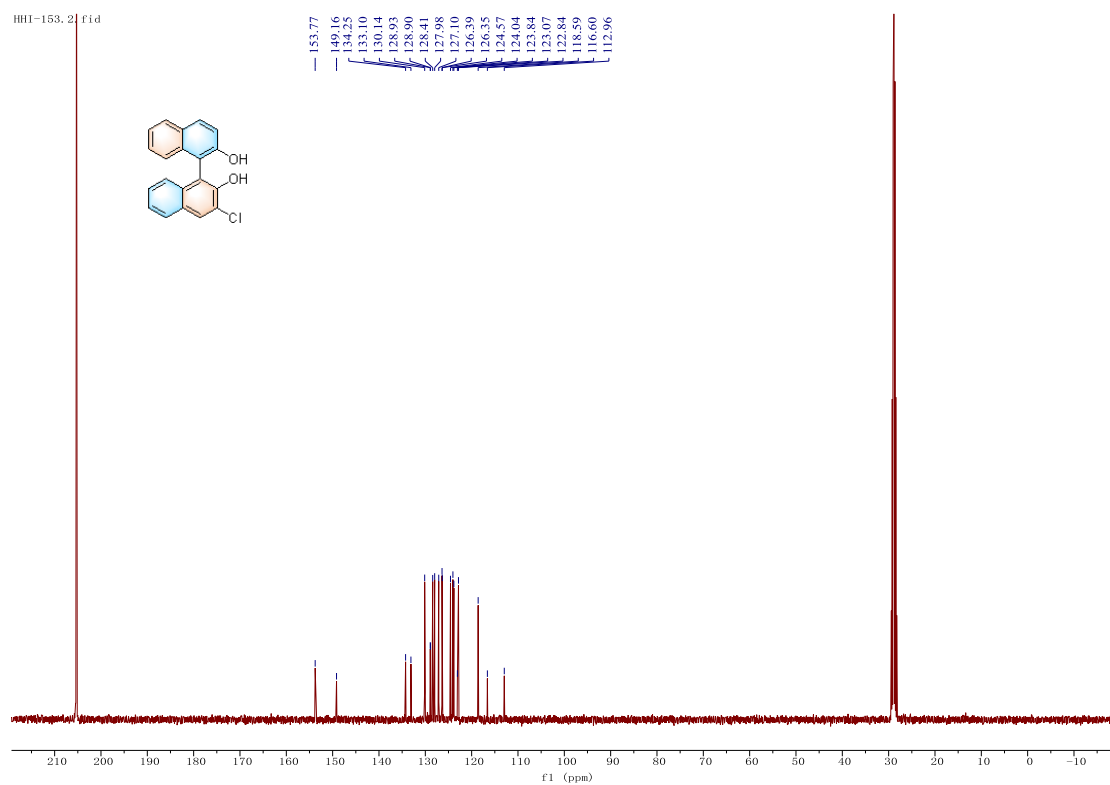


Figure S68. ^{13}C NMR Spectrum of **3r**

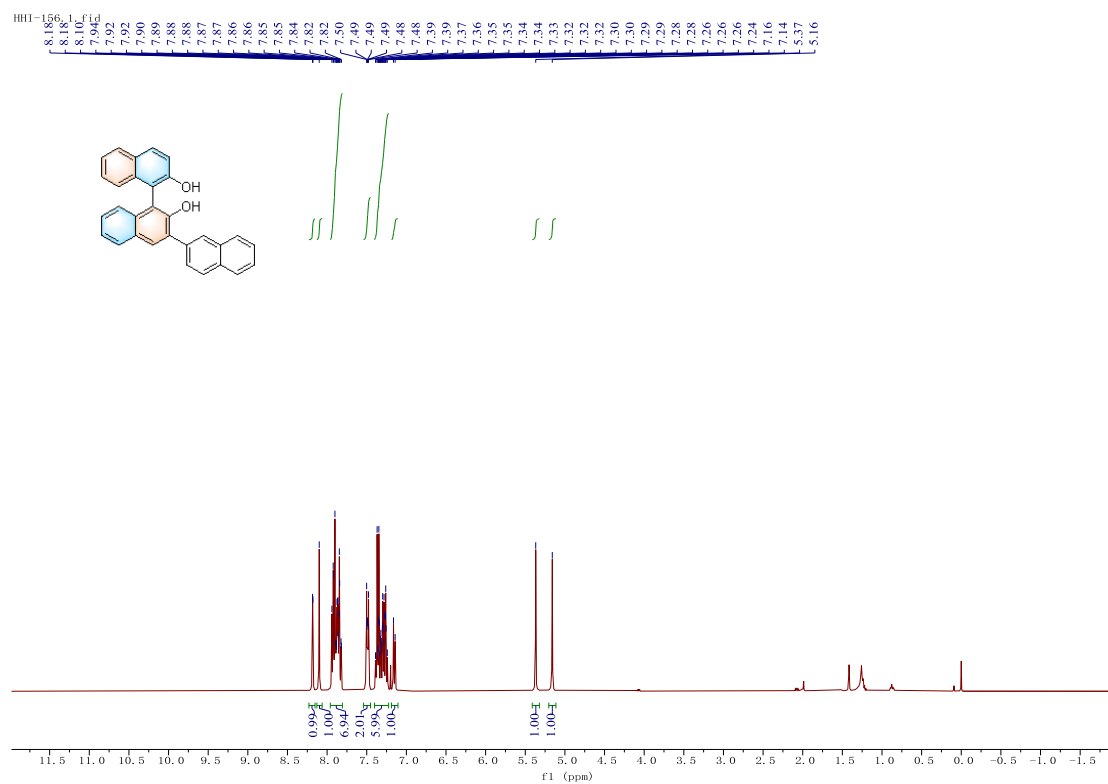


Figure S69. ^1H NMR Spectrum of 3s

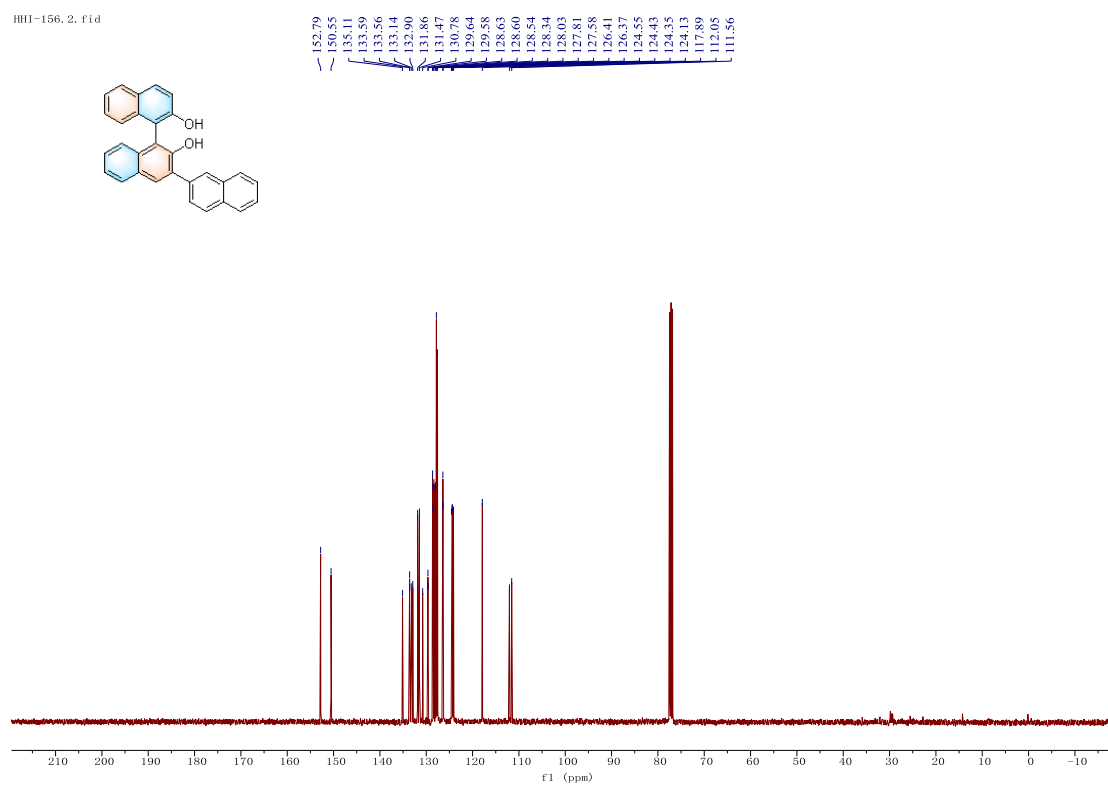


Figure S70. ^{13}C NMR Spectrum of 3s

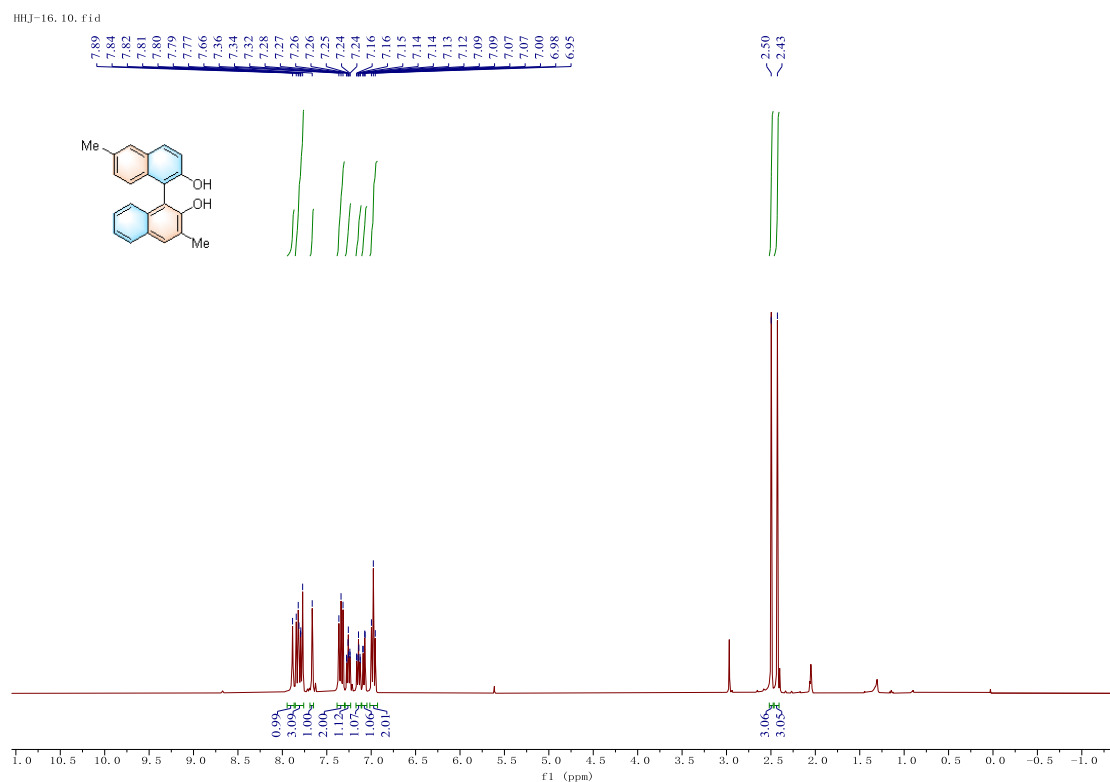


Figure S71. ^1H NMR Spectrum of **3t**

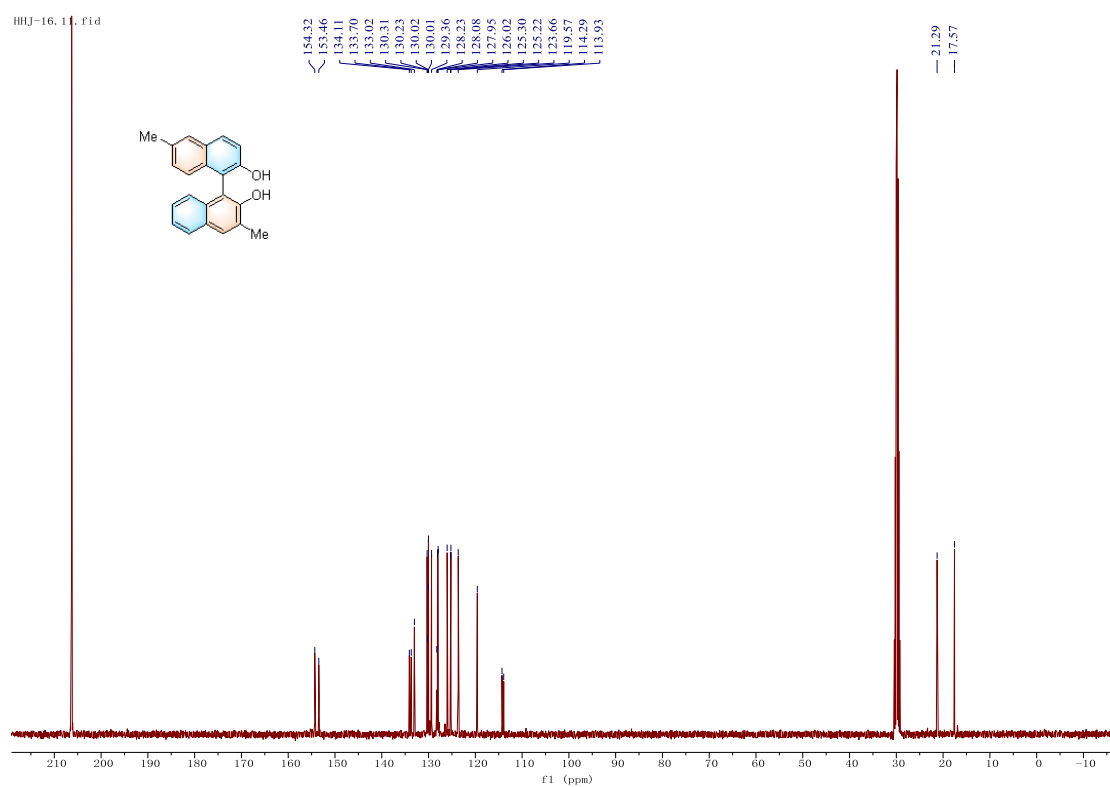


Figure S72. ^{13}C NMR Spectrum of **3t**

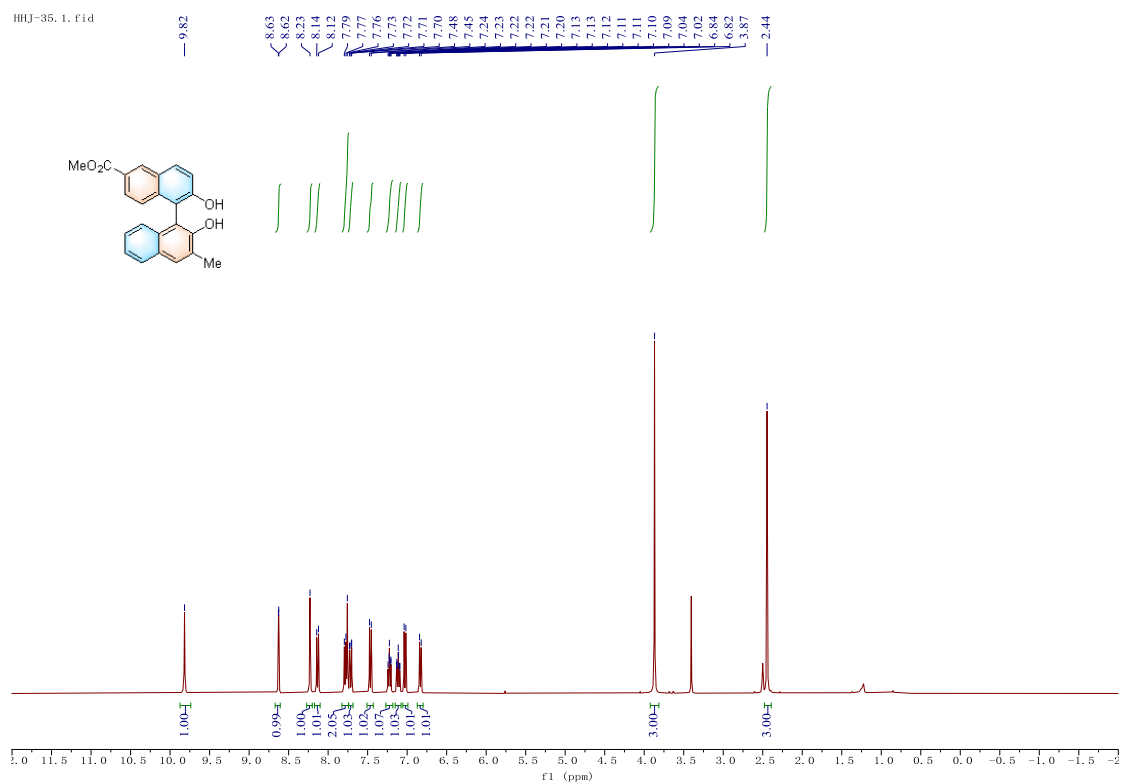


Figure S73. ^1H NMR Spectrum of **3u**

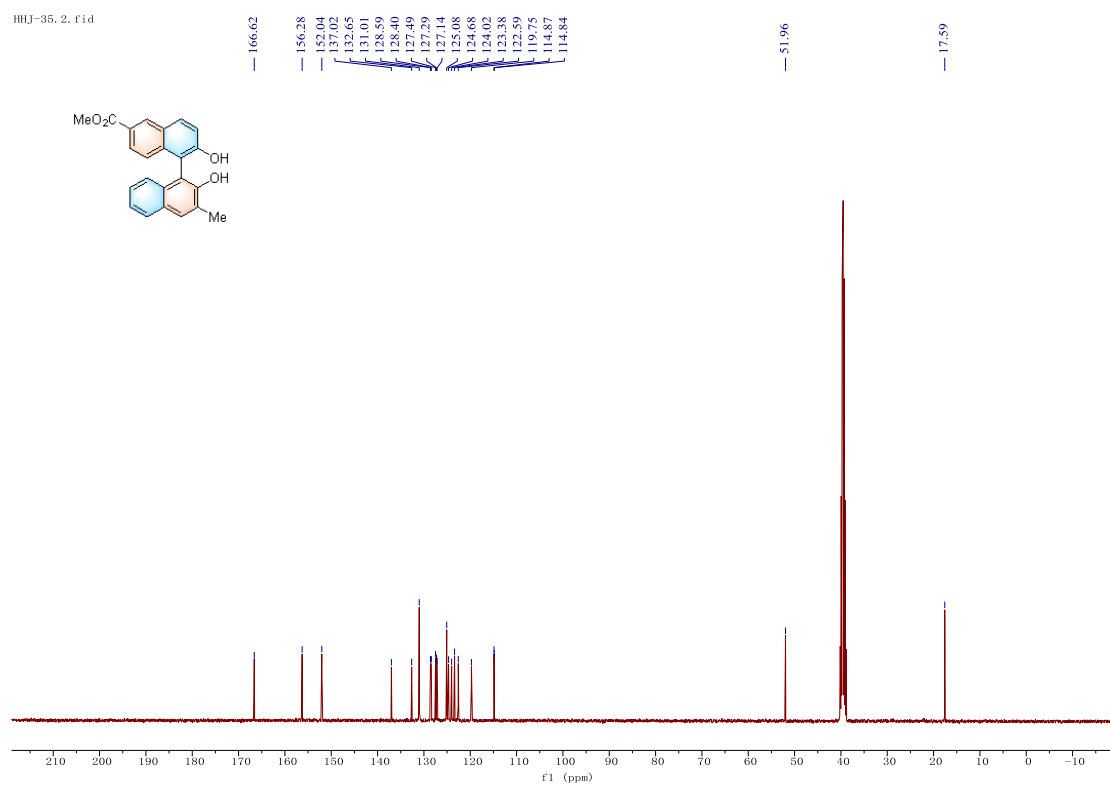


Figure S74. ^{13}C NMR Spectrum of **3u**

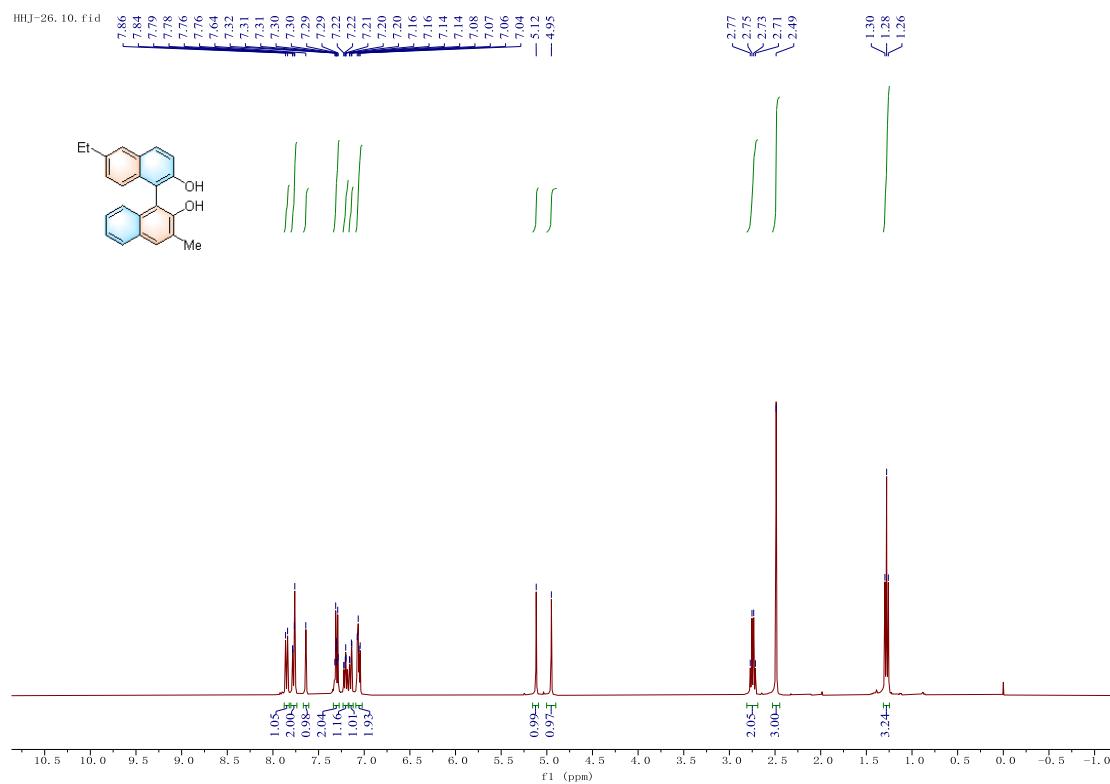


Figure S75. ^1H NMR Spectrum of **3v**

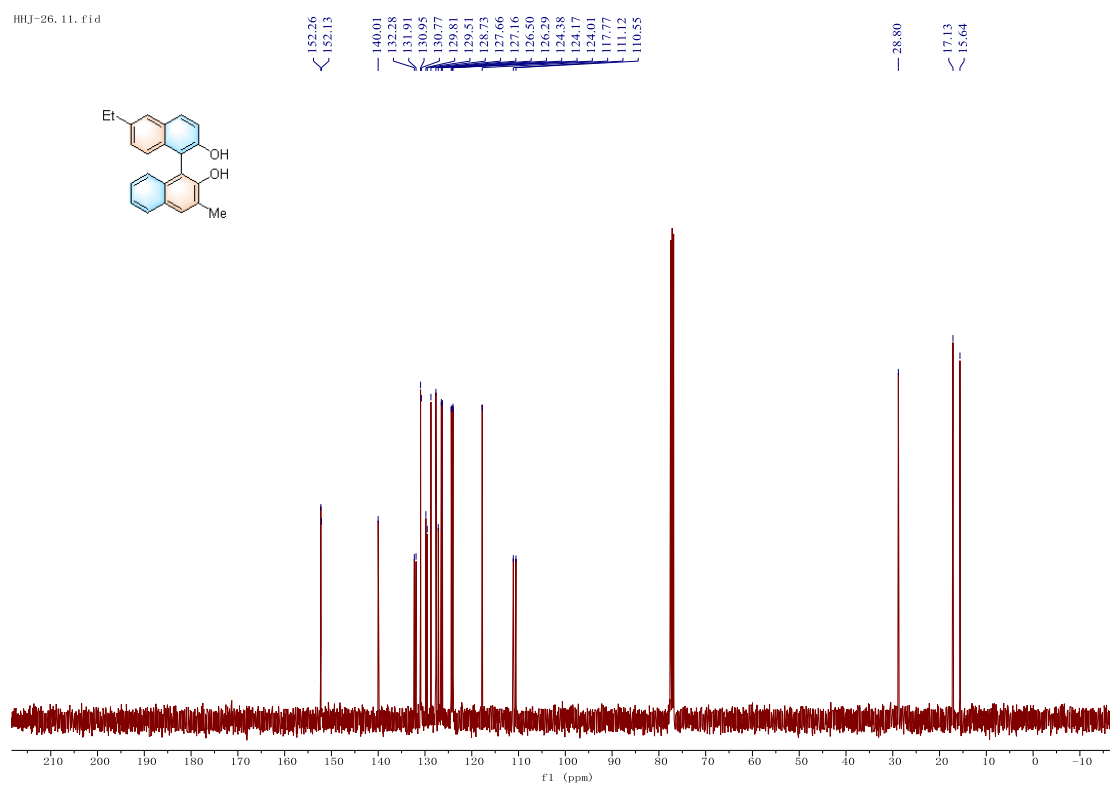


Figure S76. ^{13}C NMR Spectrum of **3v**

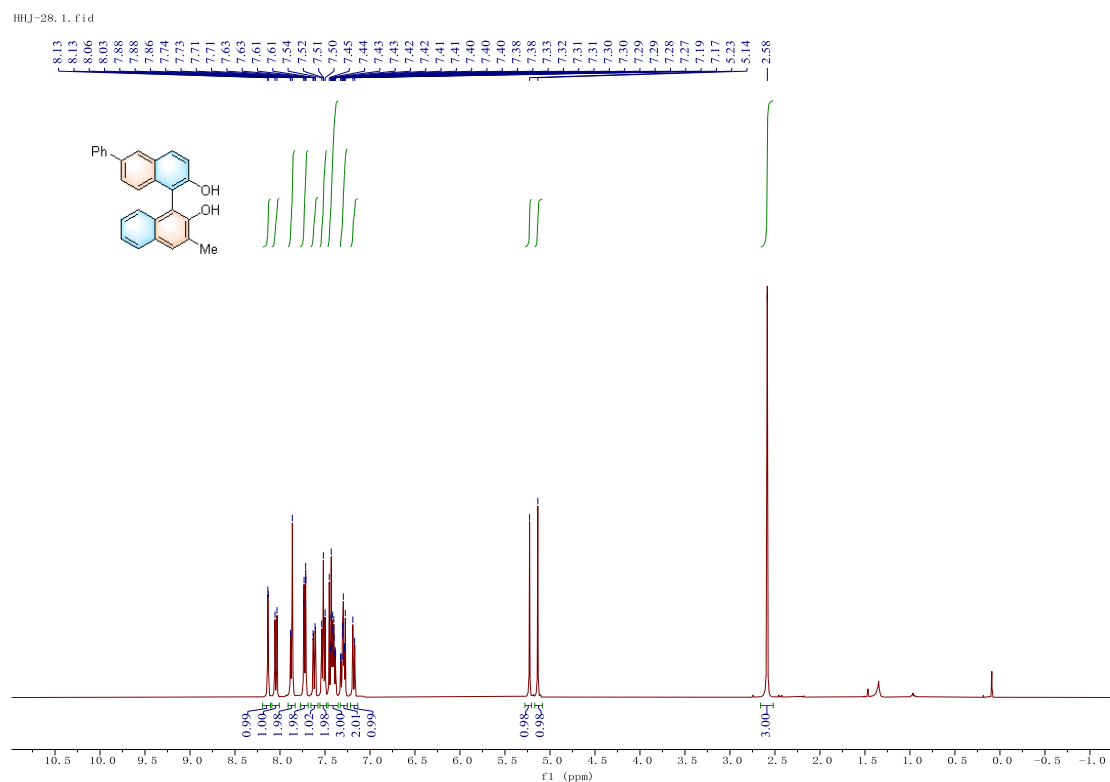


Figure S77. ^1H NMR Spectrum of **3w**

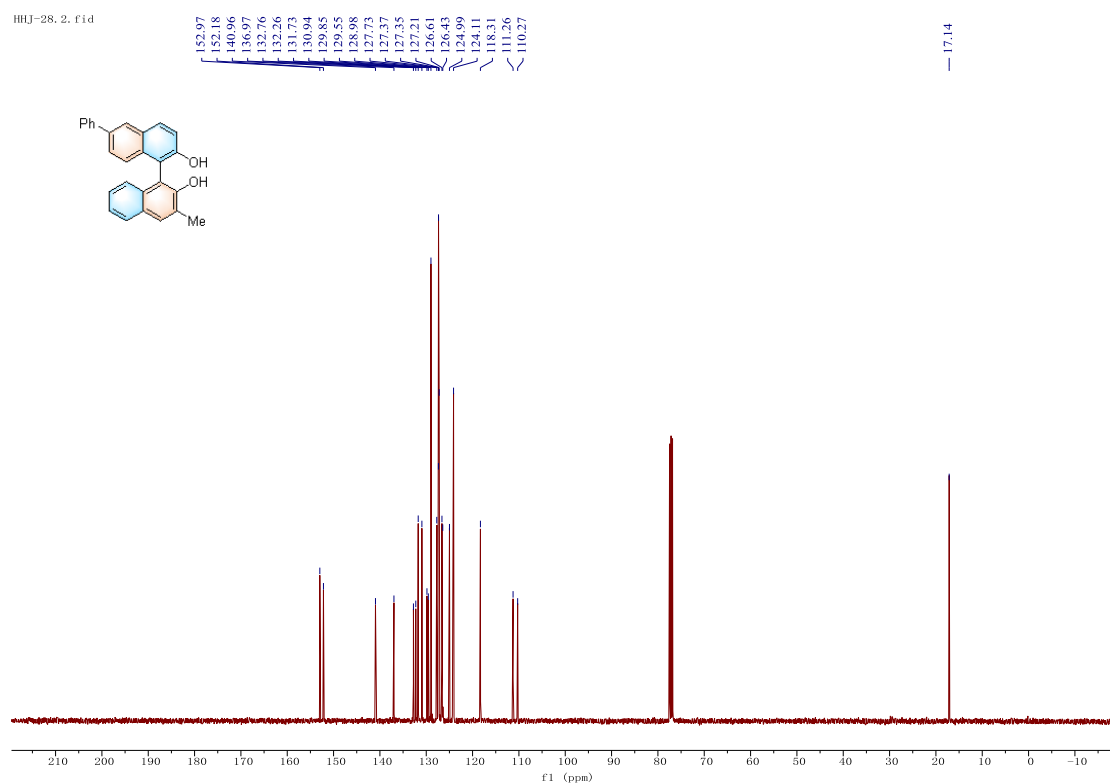


Figure S78. ^{13}C NMR Spectrum of **3w**

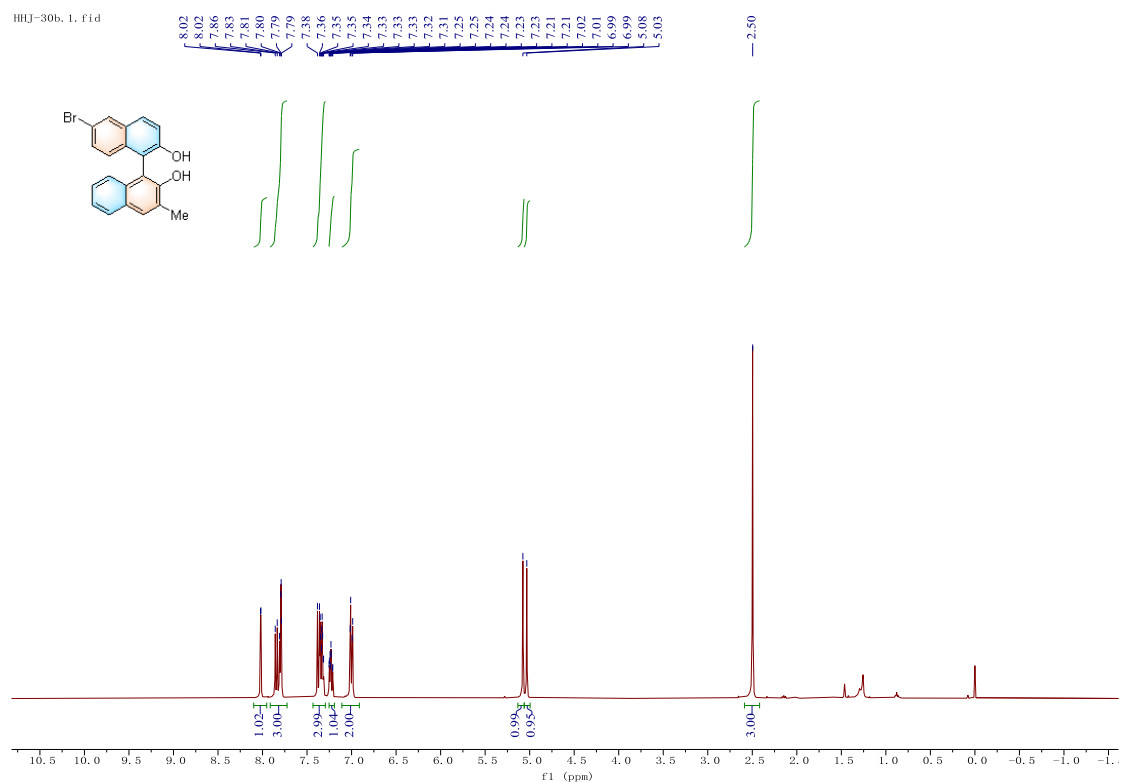


Figure S79. ^1H NMR Spectrum of **3x**

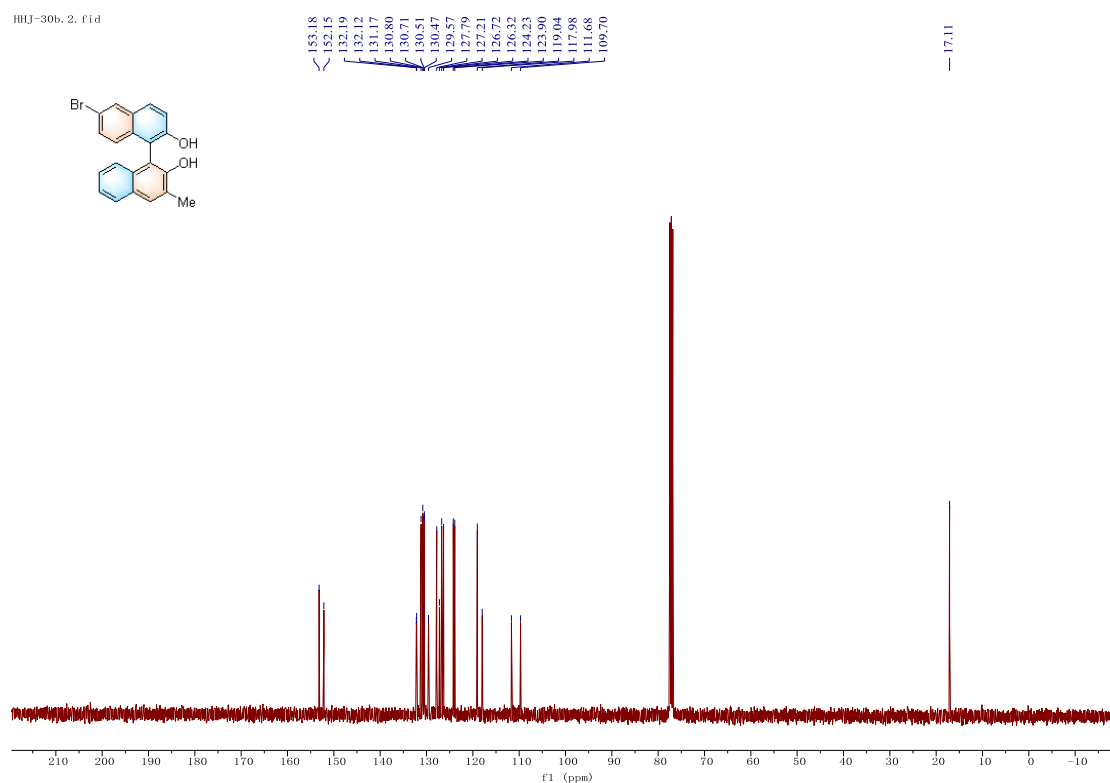
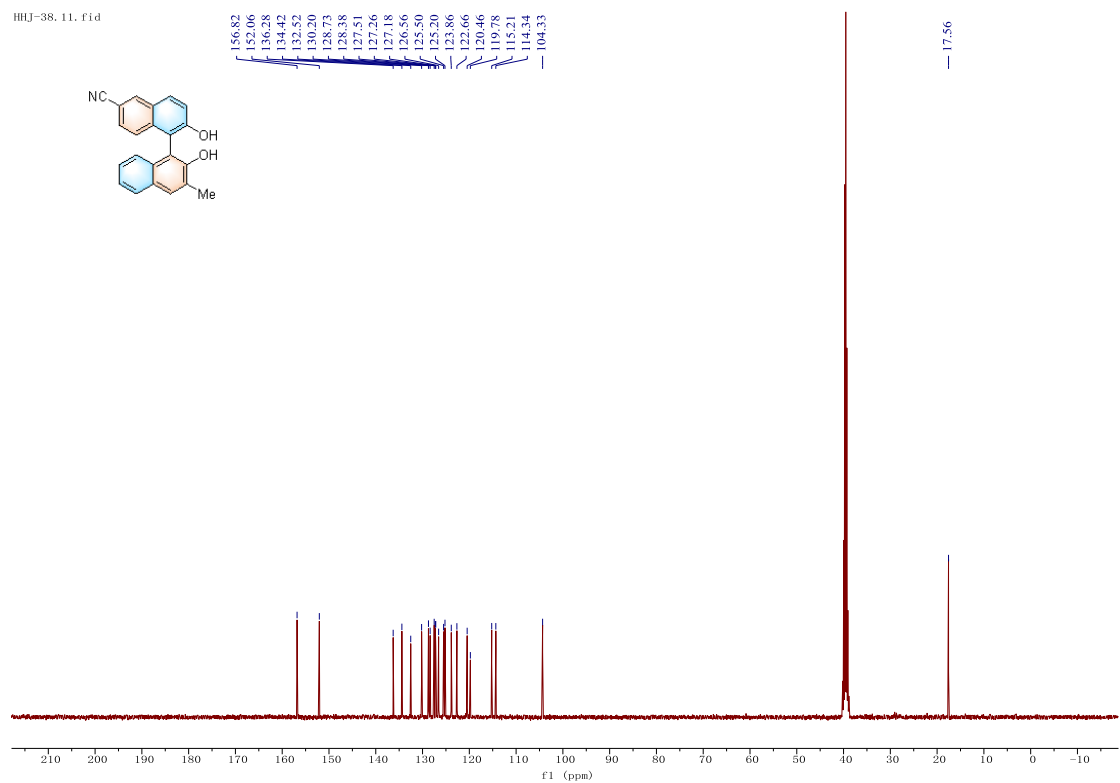
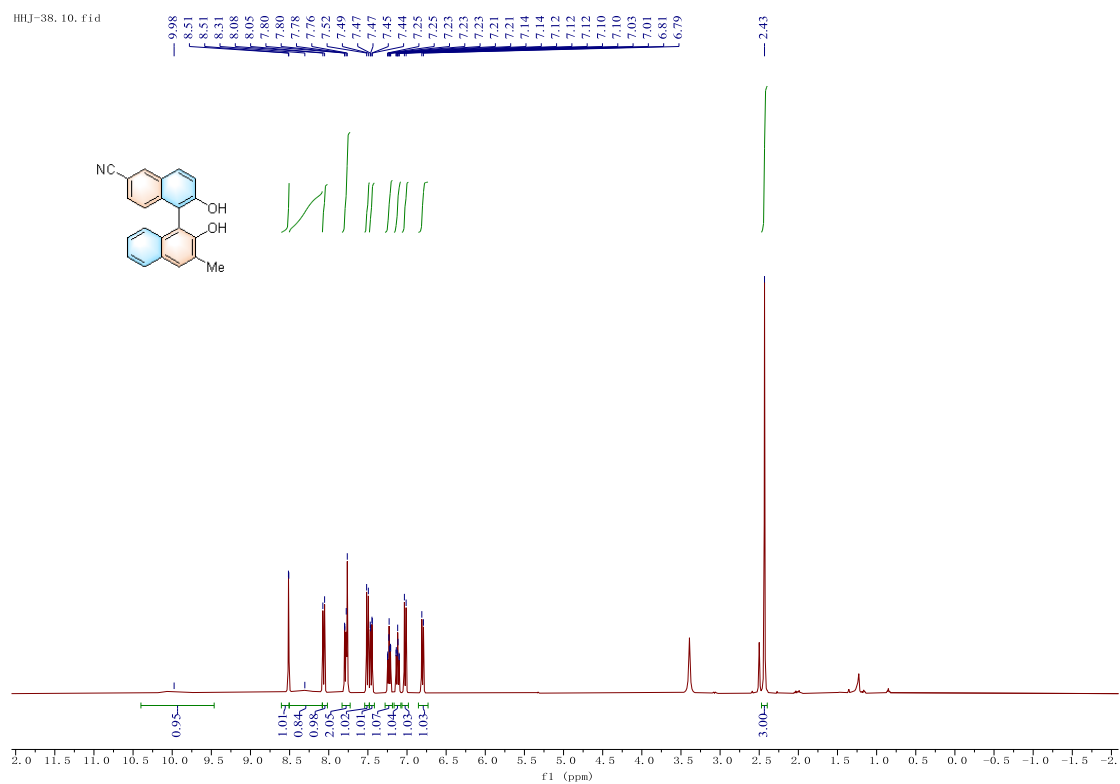
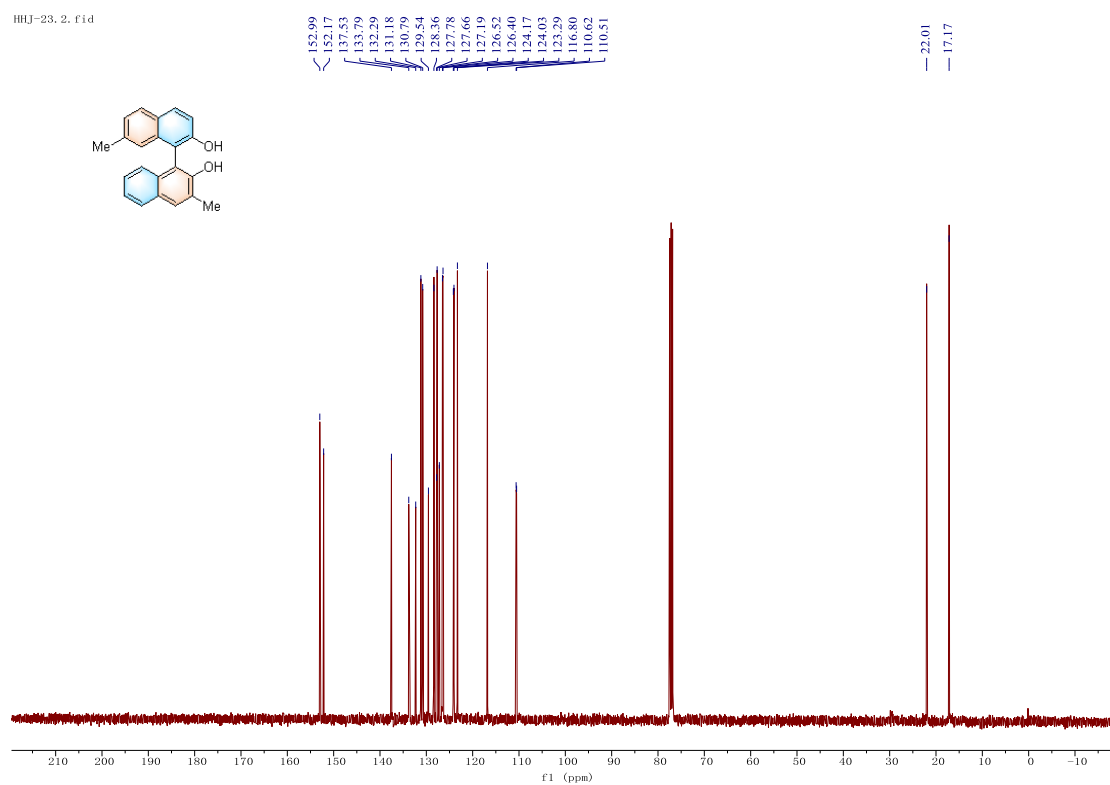
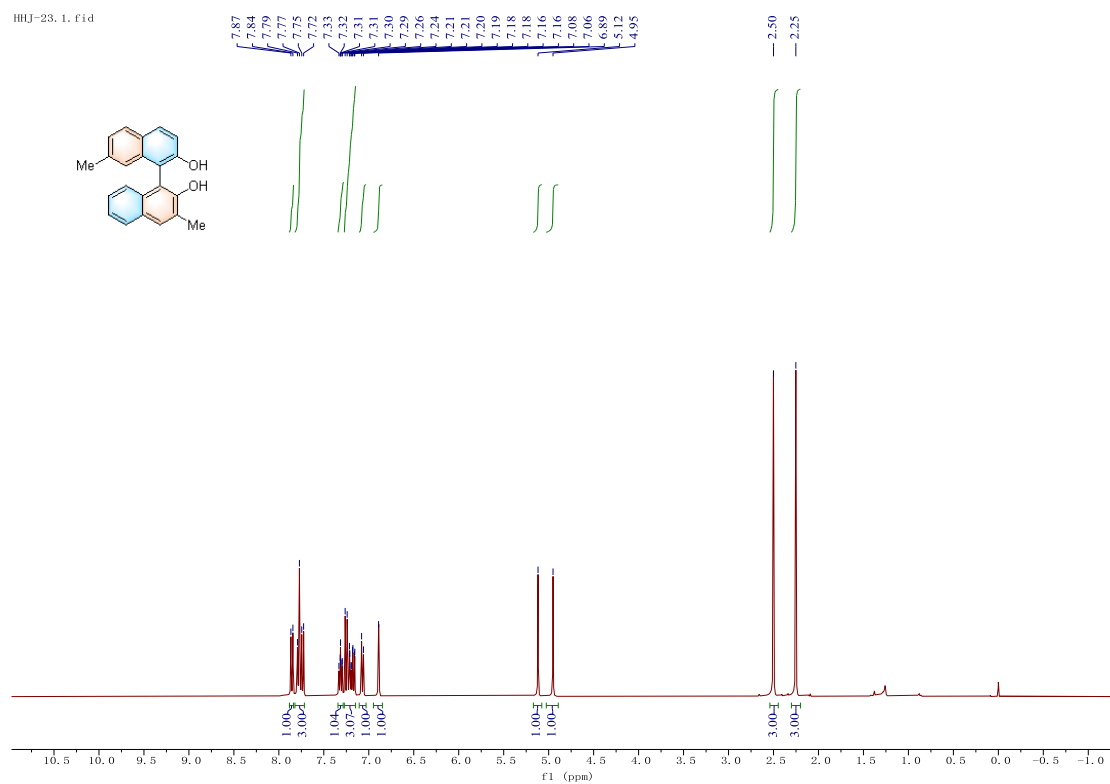


Figure S80. ^{13}C NMR Spectrum of **3x**





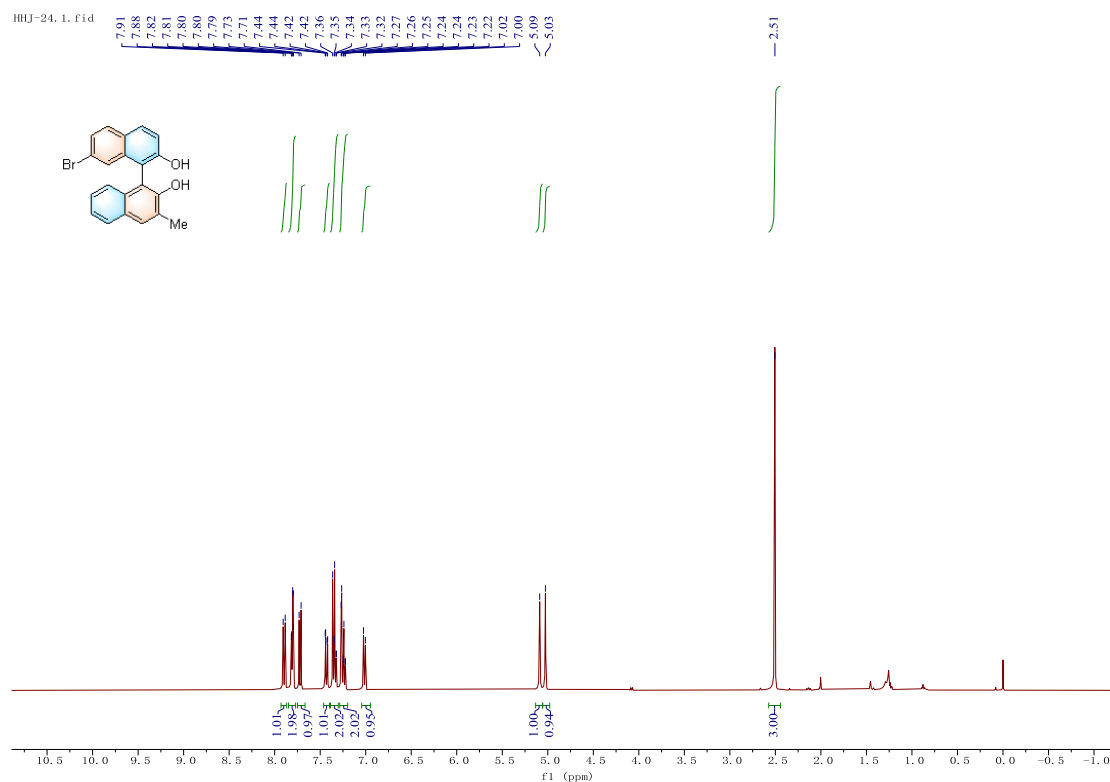


Figure S85. ^1H NMR Spectrum of **3aa**

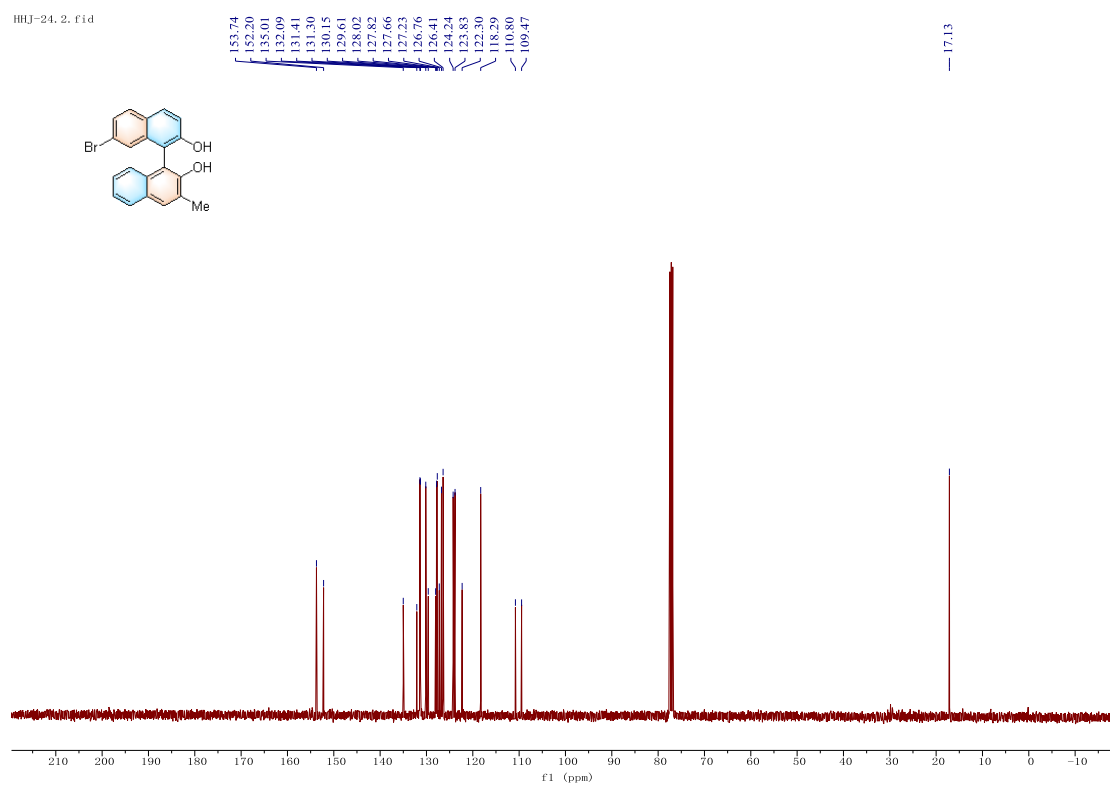
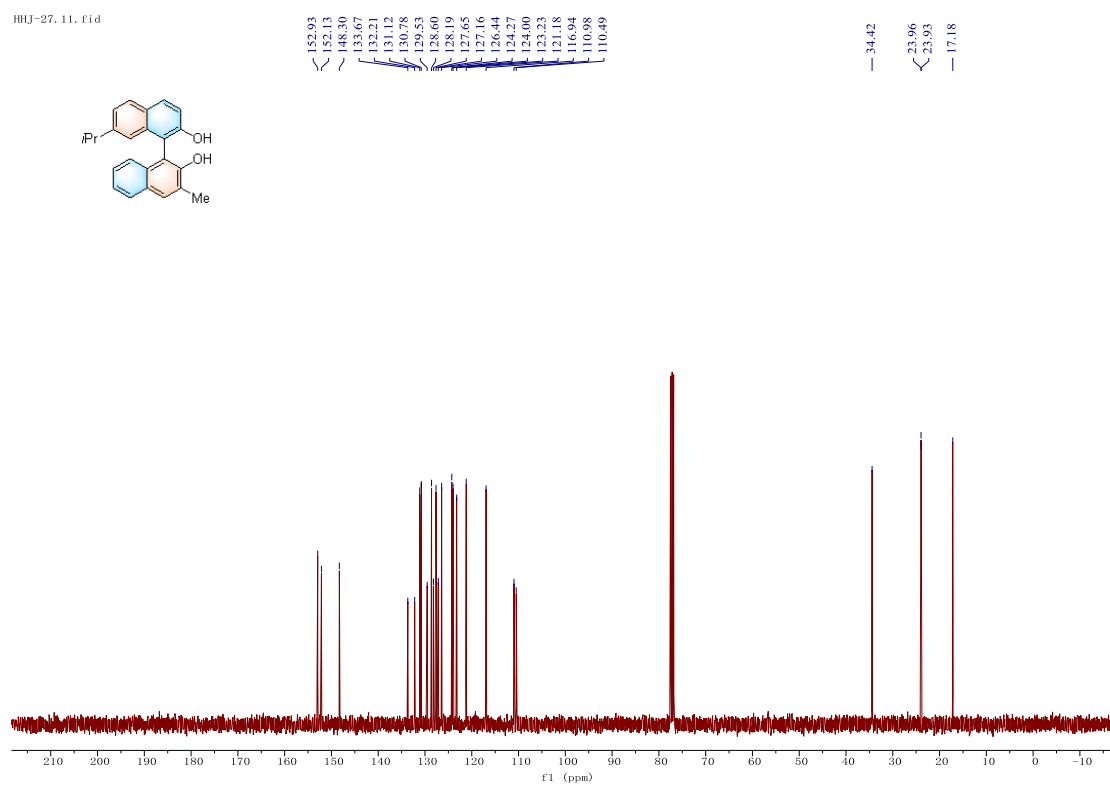
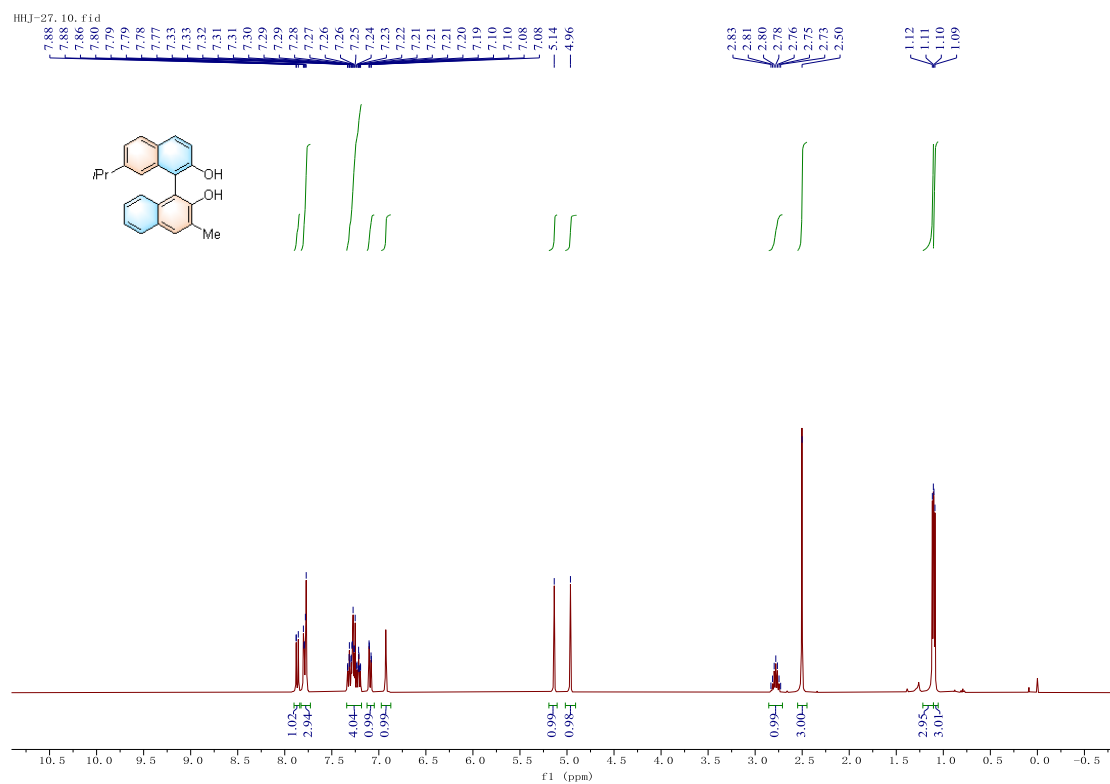


Figure S86. ^{13}C NMR Spectrum of **3aa**



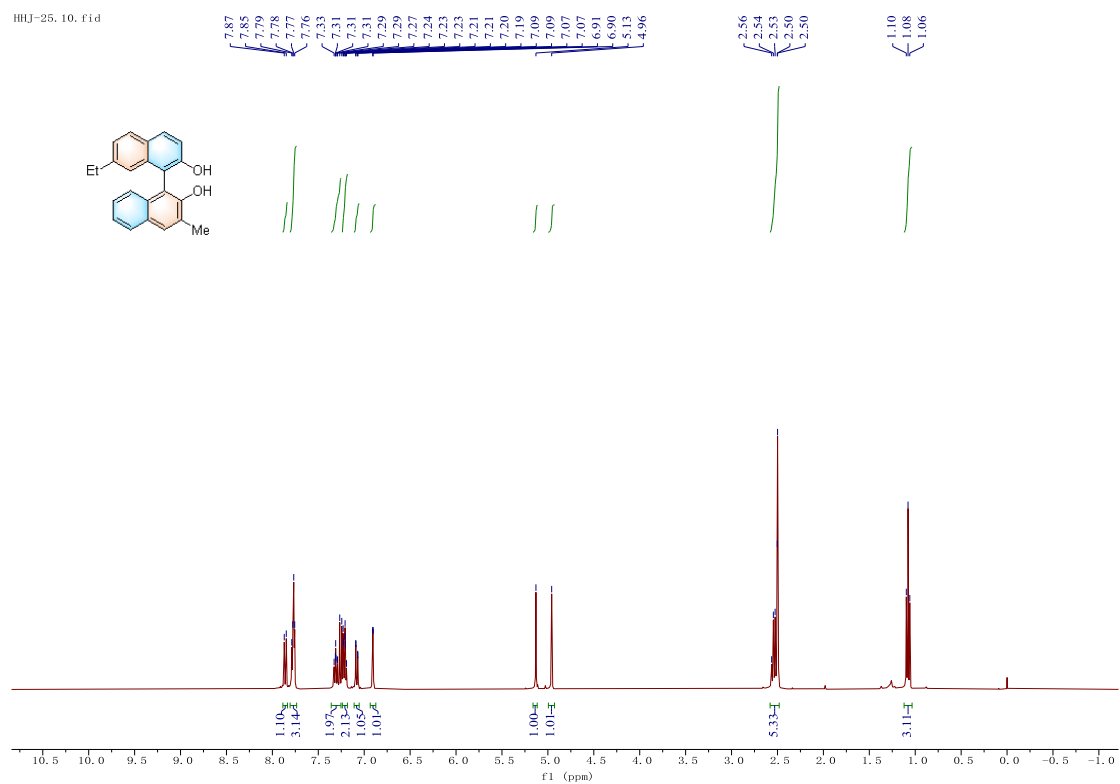


Figure S89. ¹H NMR Spectrum of **3ac**

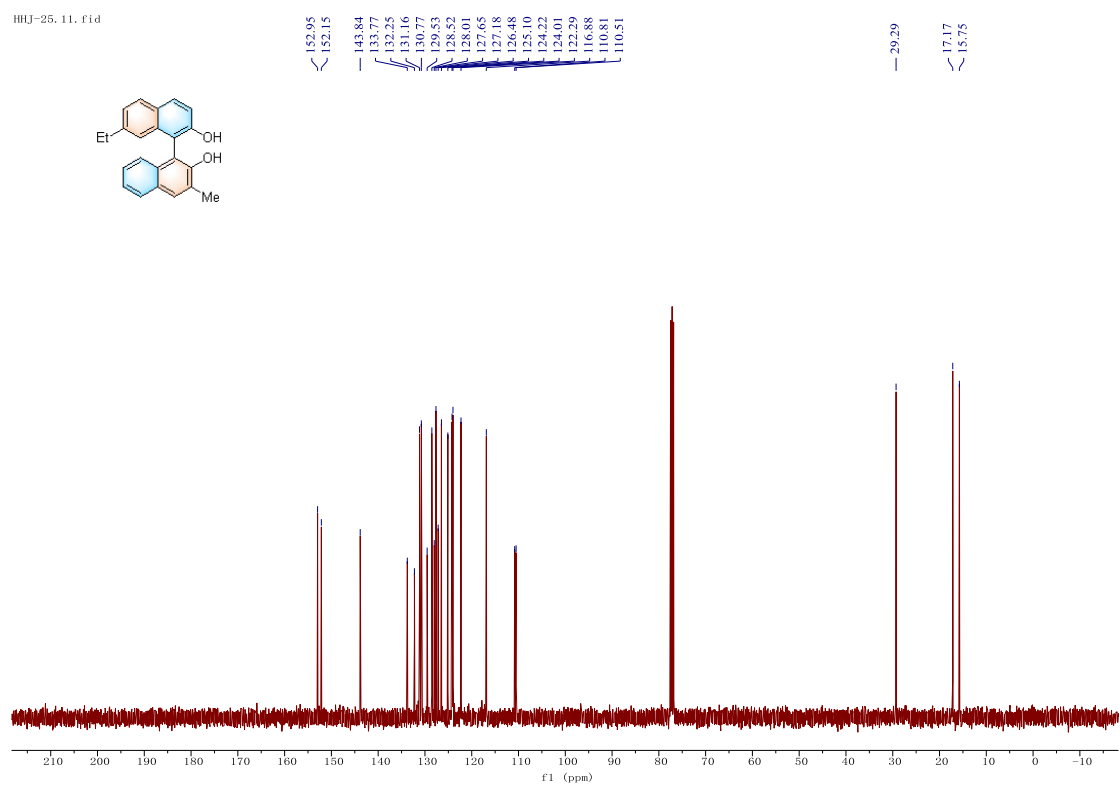
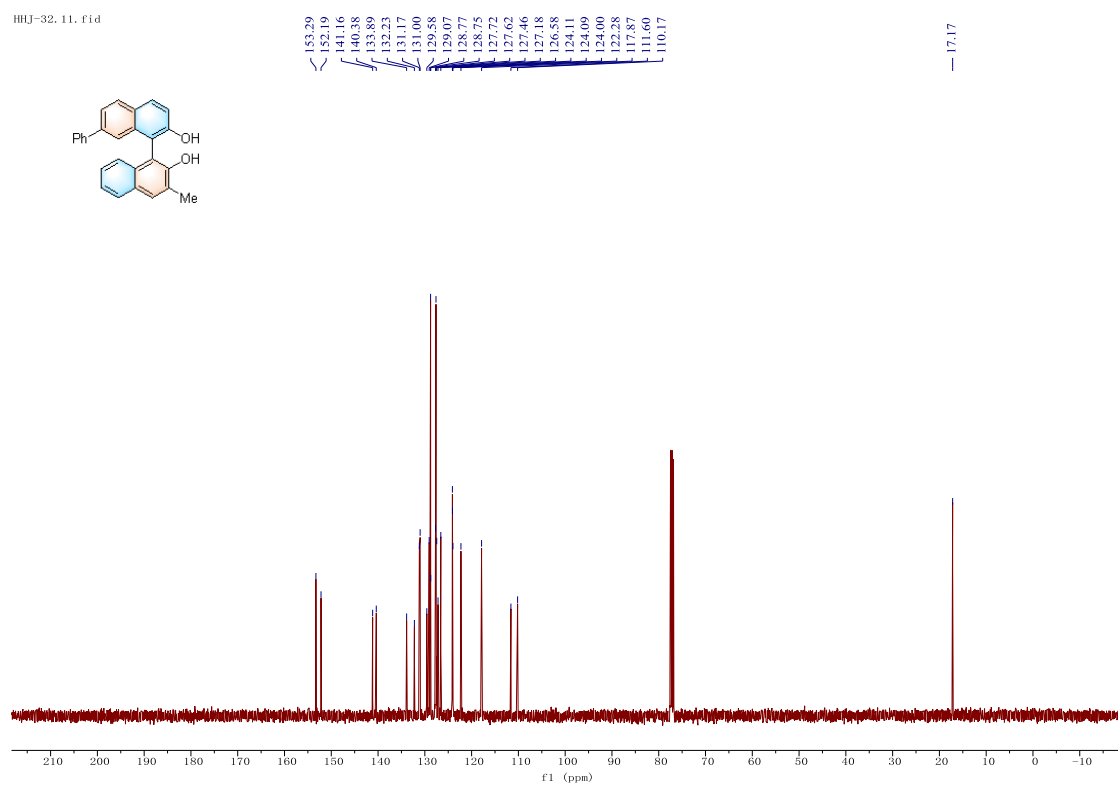
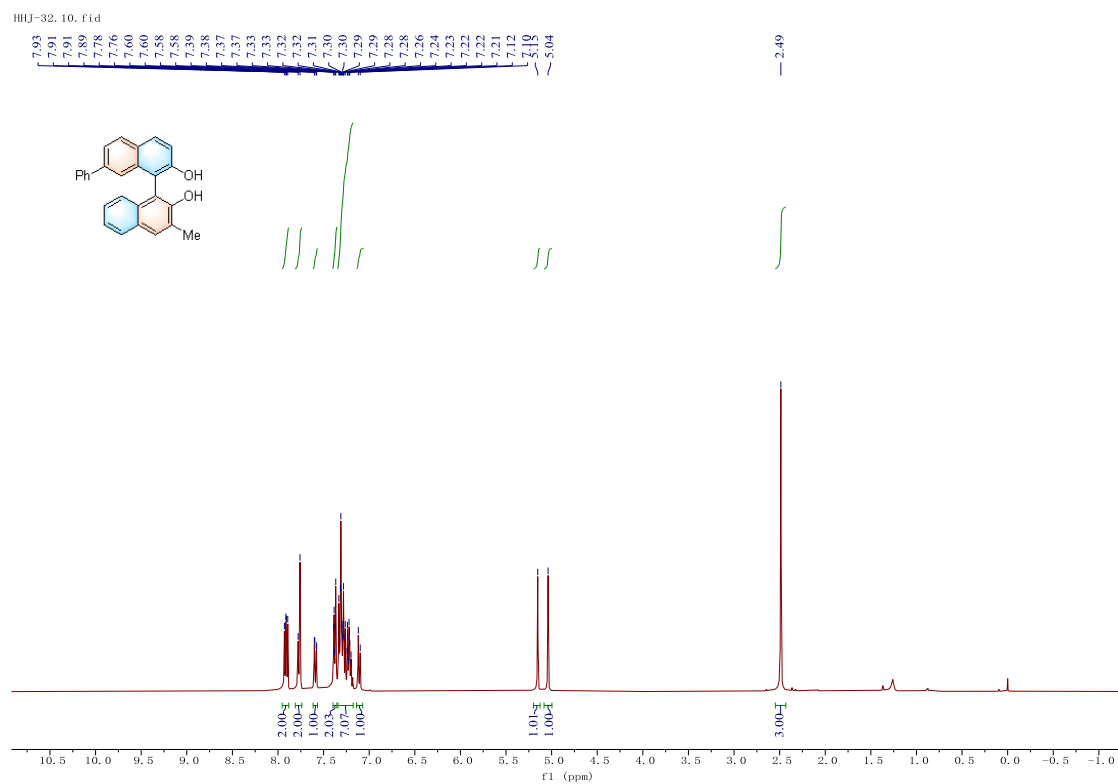


Figure S90. ¹³C NMR Spectrum of **3ac**



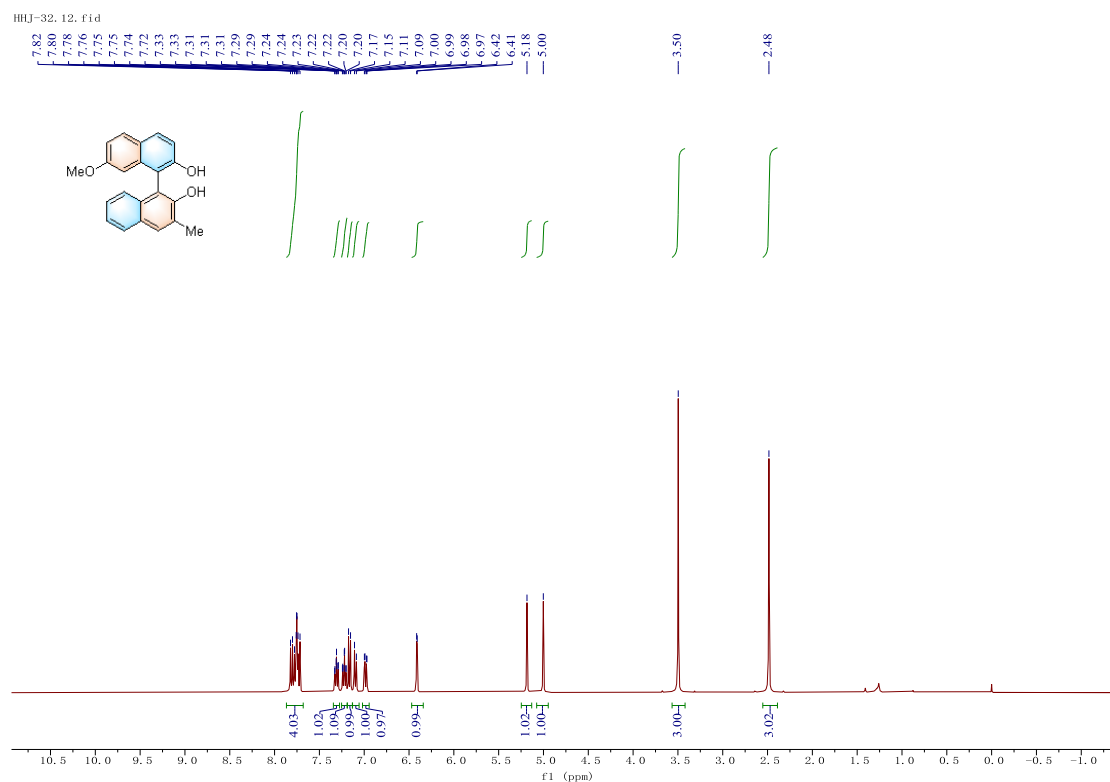


Figure S93. ^1H NMR Spectrum of 3ae

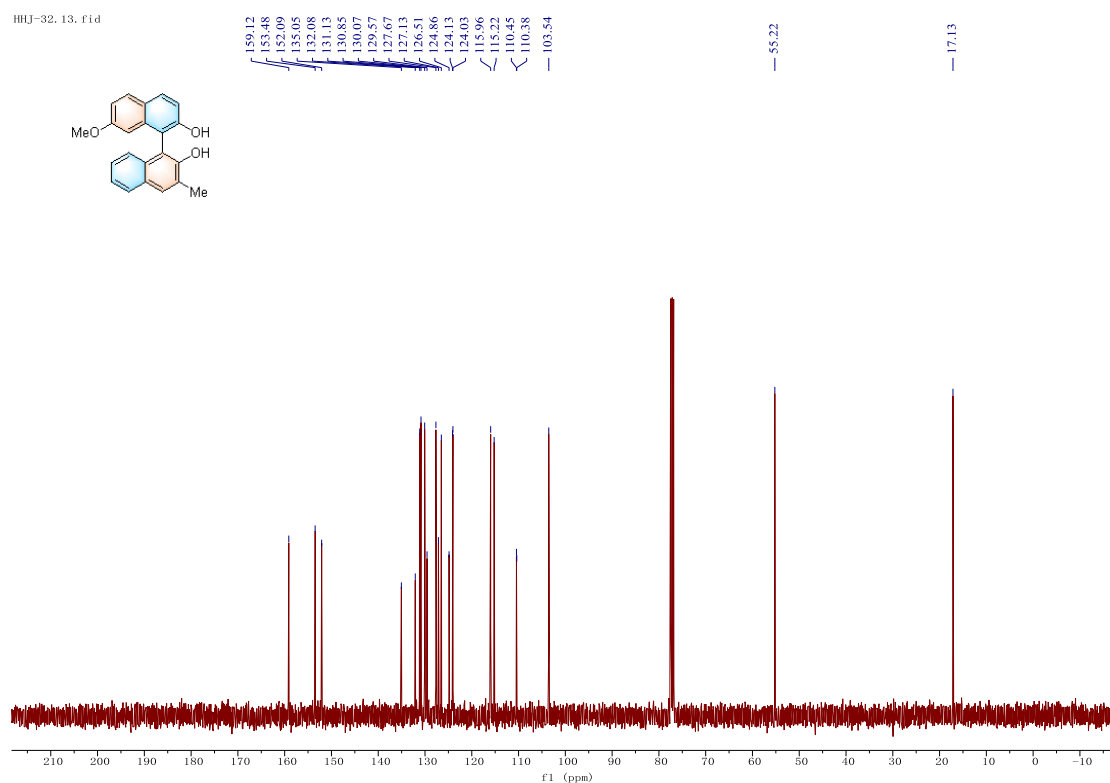


Figure S94. ^{13}C NMR Spectrum of 3ae

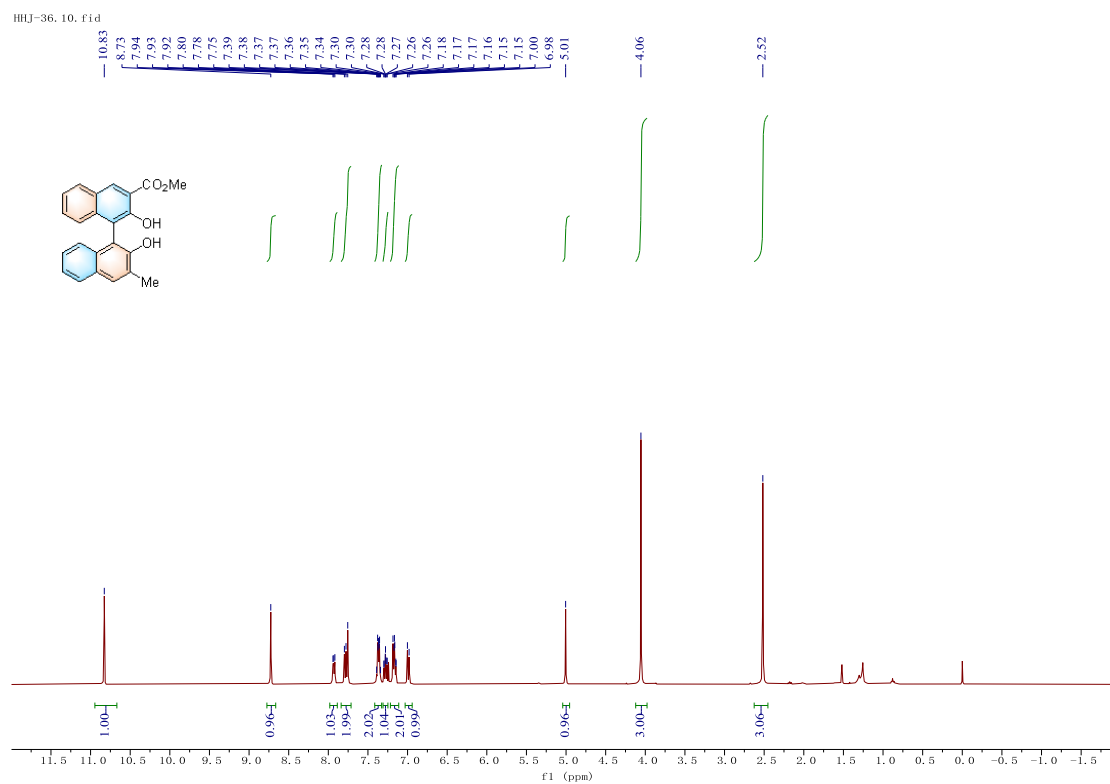


Figure S95. ¹H NMR Spectrum of **3af**

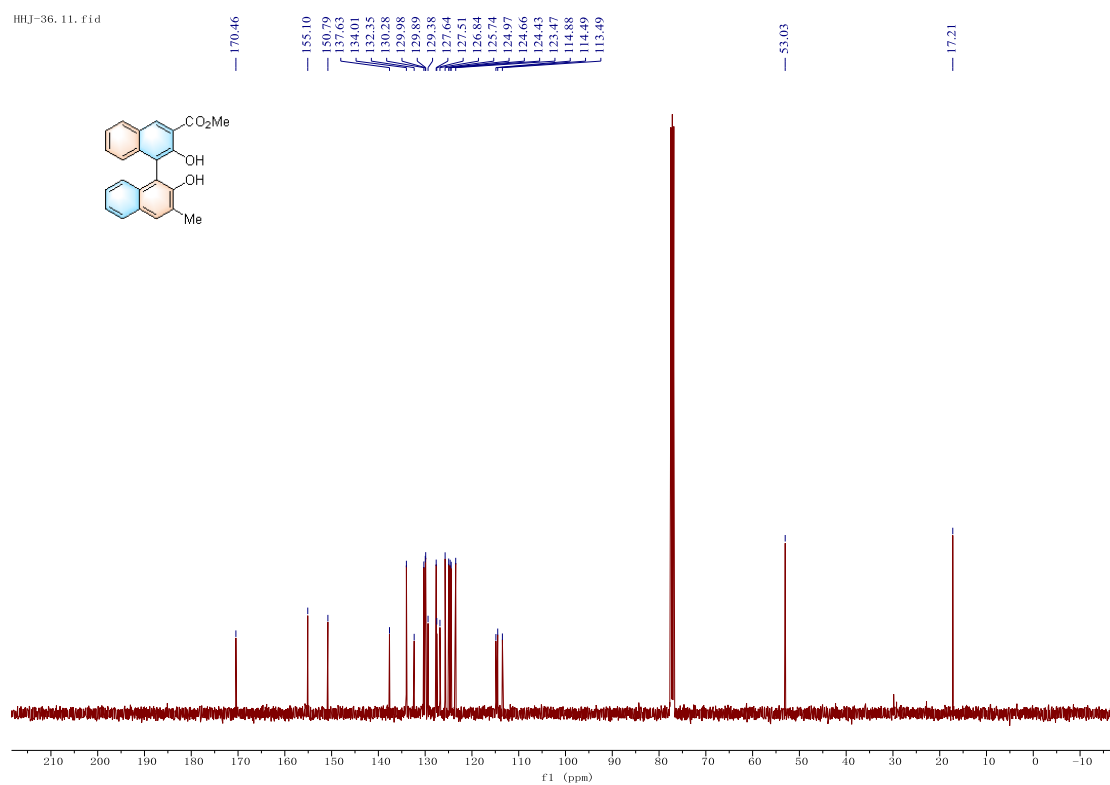


Figure S96. ¹³C NMR Spectrum of **3af**

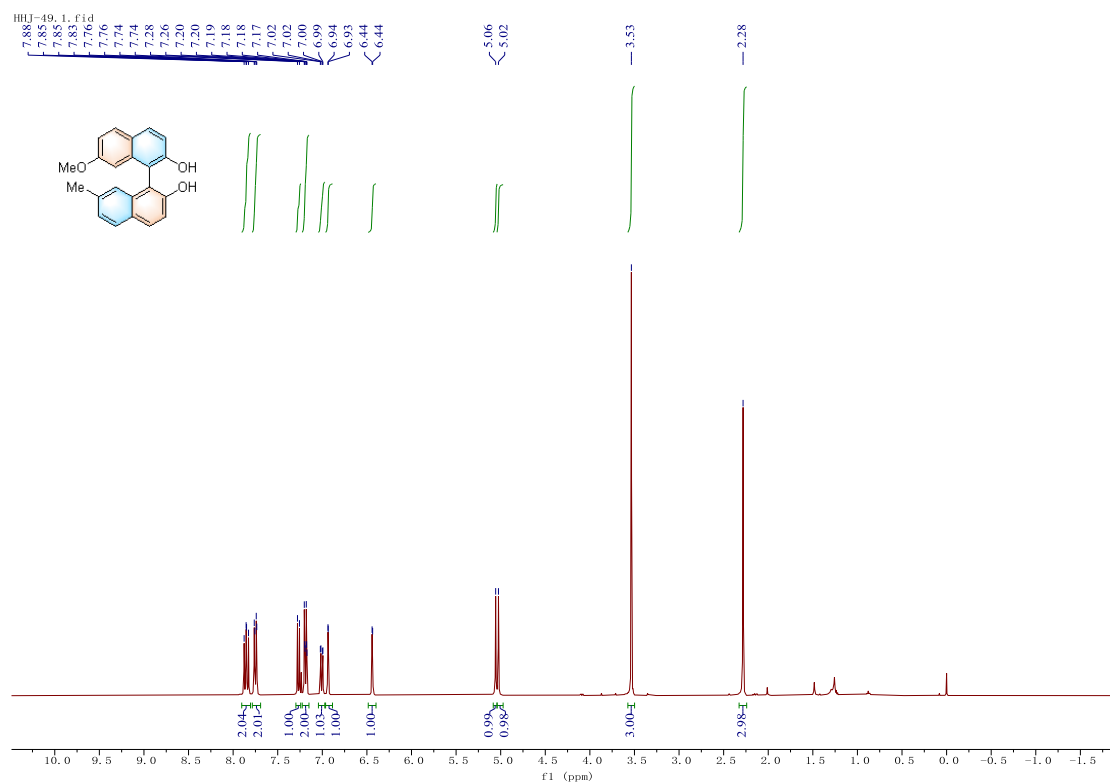


Figure S97. ^1H NMR Spectrum of **3ag**

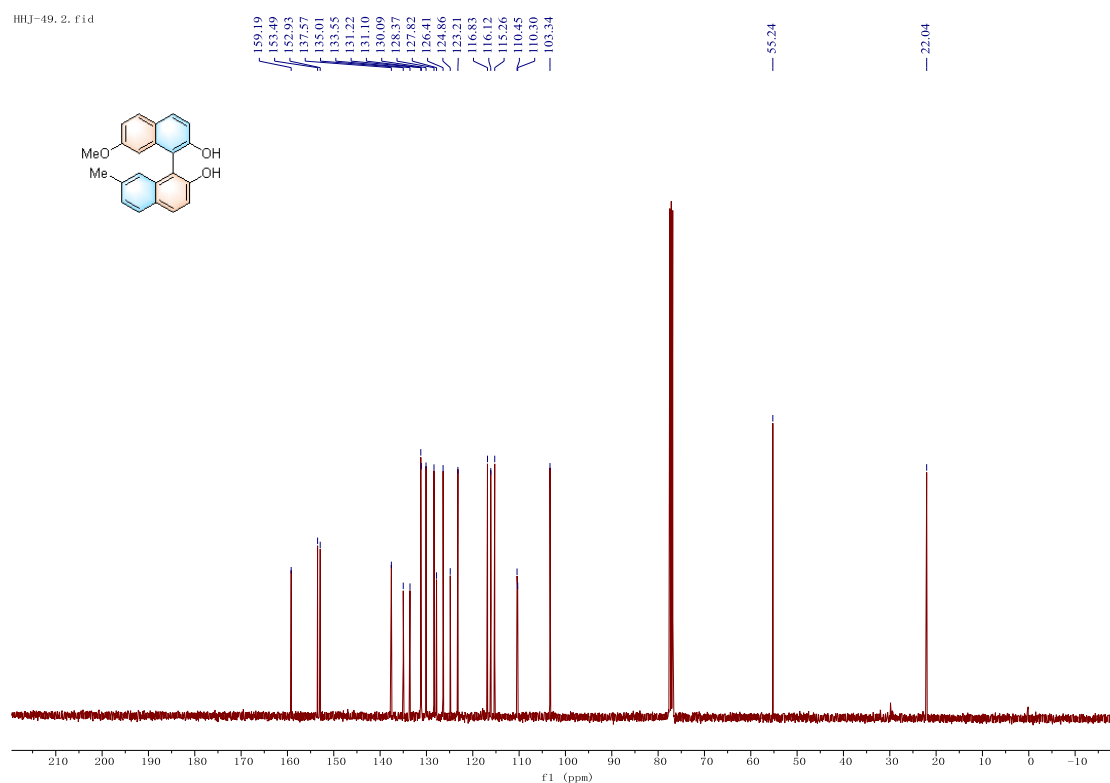


Figure S98. ^{13}C NMR Spectrum of **3ag**

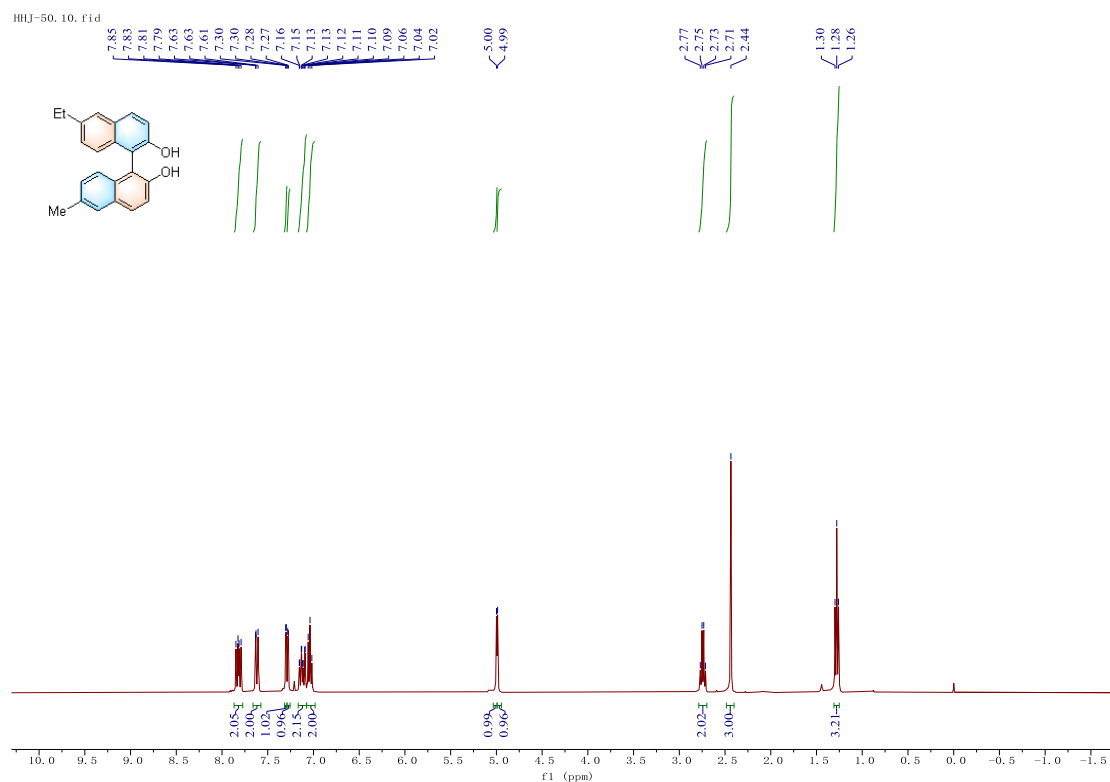


Figure S99. ^1H NMR Spectrum of **3ah**

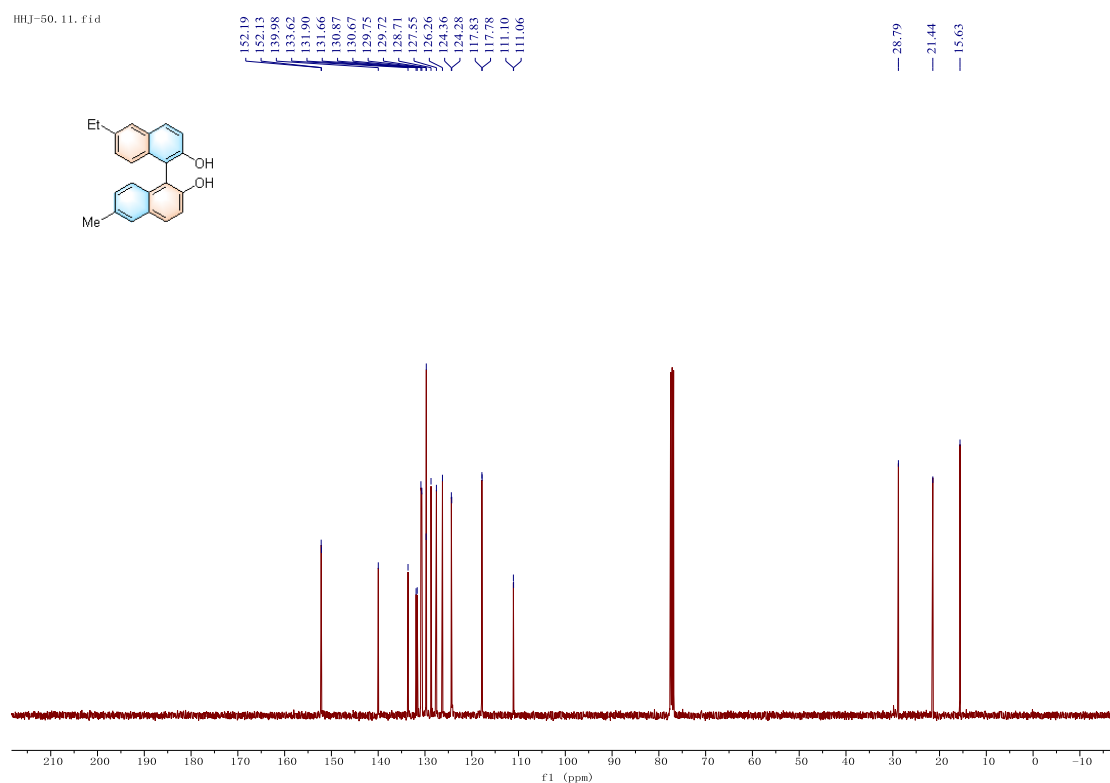


Figure S100. ^{13}C NMR Spectrum of **3ah**

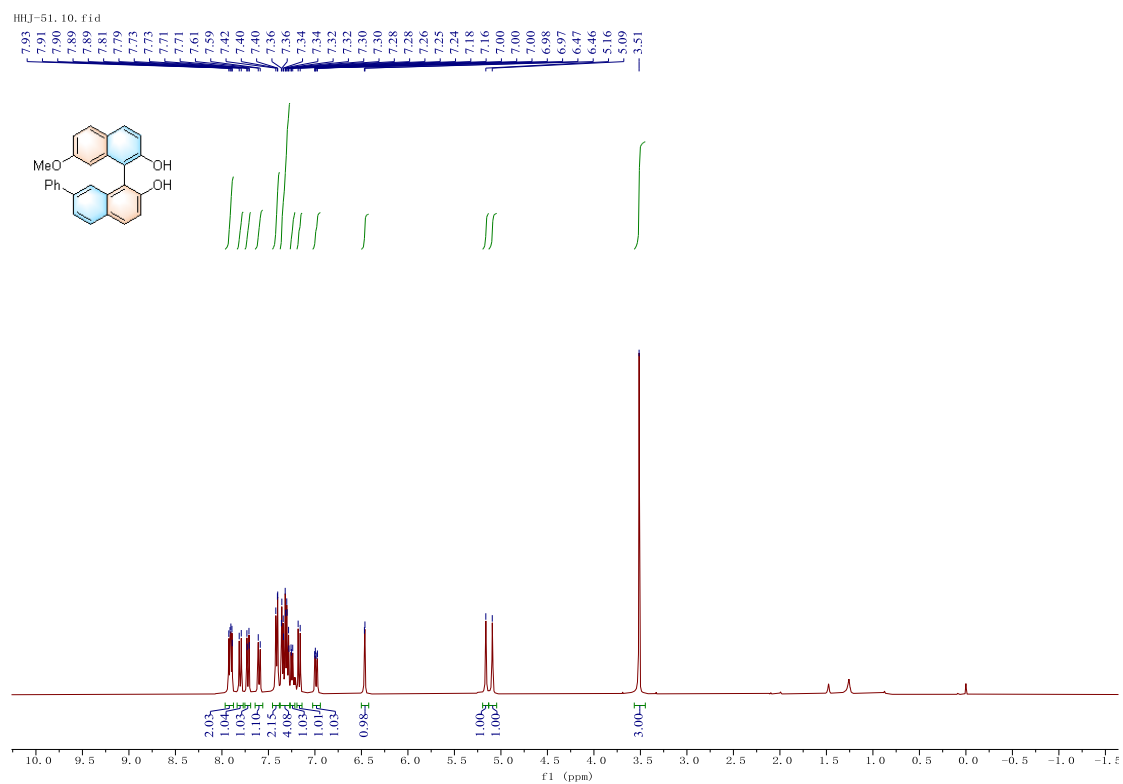


Figure S101. ^1H NMR Spectrum of **3ai**

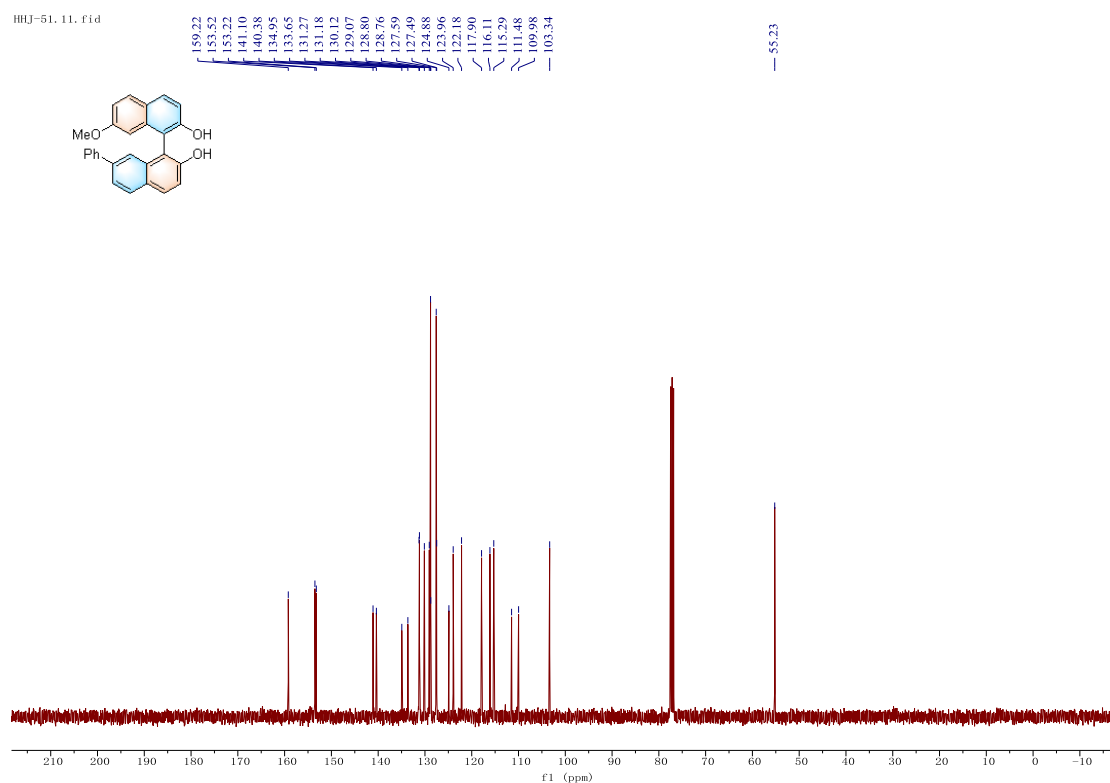


Figure S102. ^{13}C NMR Spectrum of **3ai**

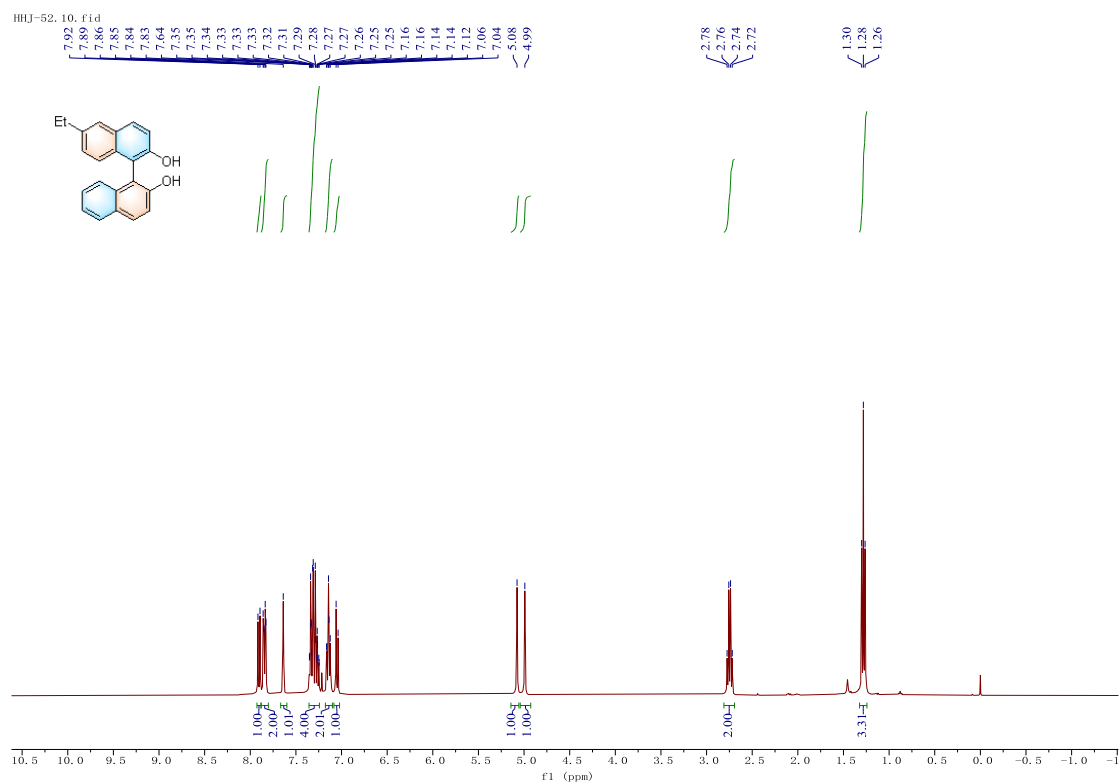


Figure S103. ^1H NMR Spectrum of 3aj

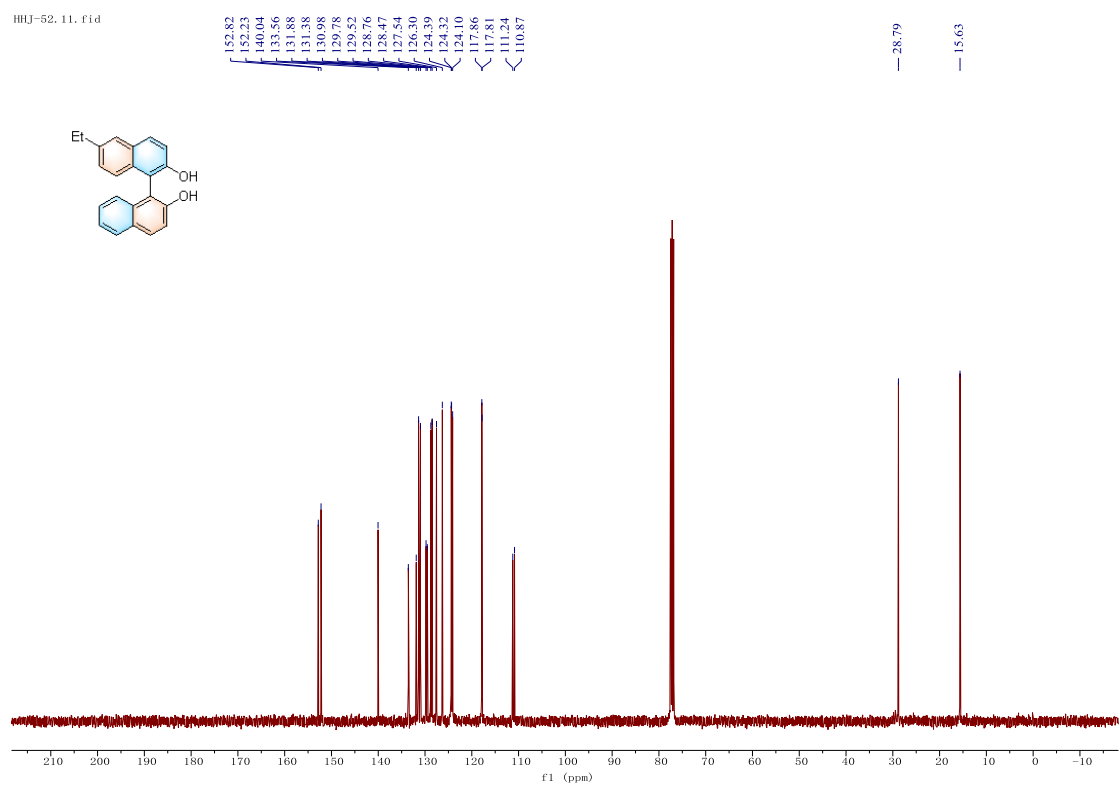


Figure S104. ^{13}C NMR Spectrum of 3aj

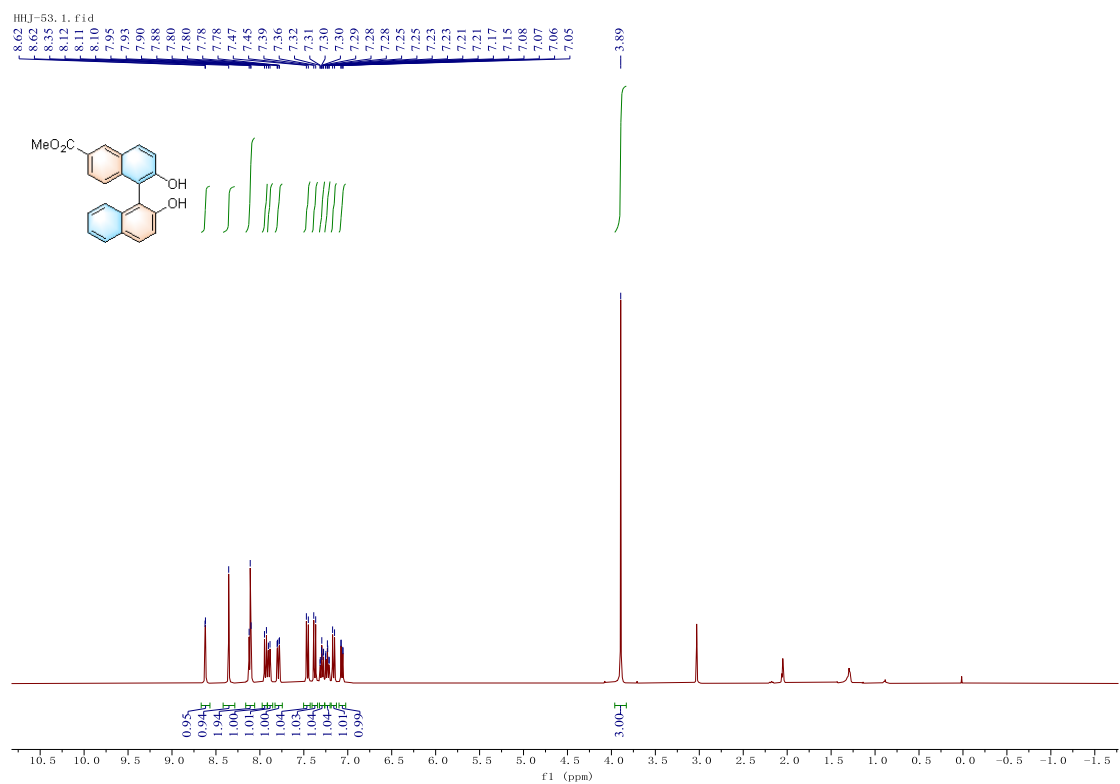


Figure S105. ^1H NMR Spectrum of **3ak**

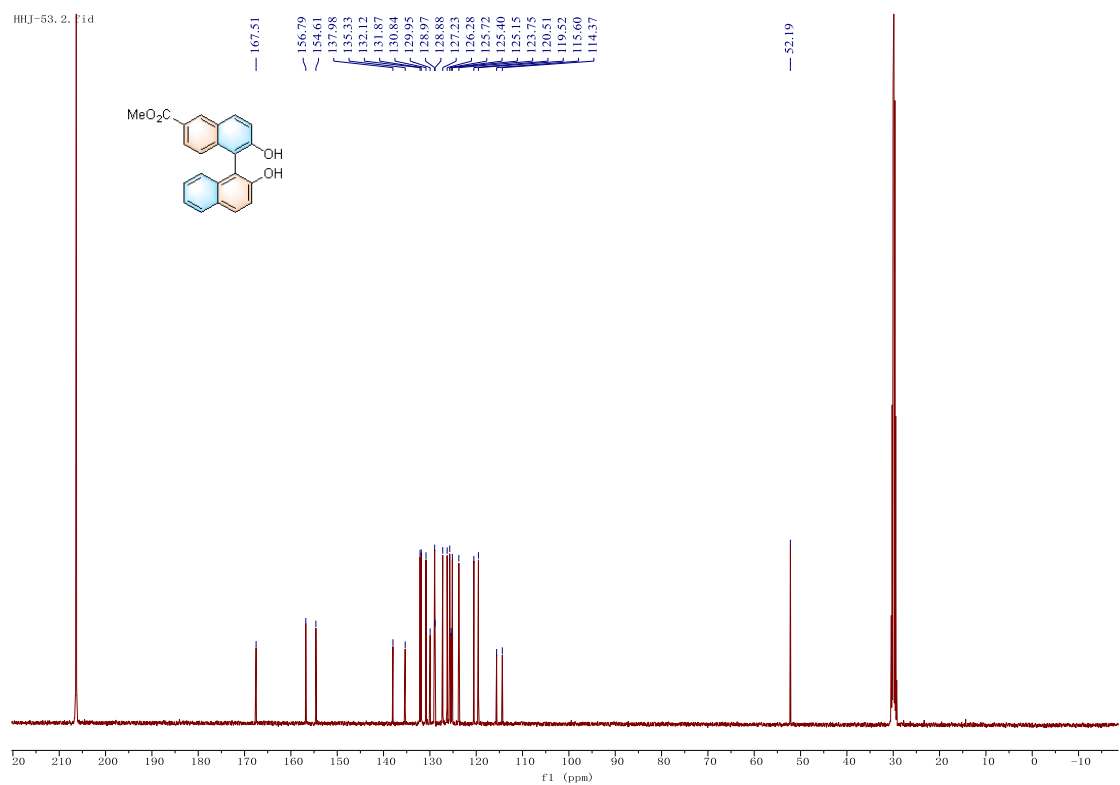


Figure S106. ^{13}C NMR Spectrum of **3ak**

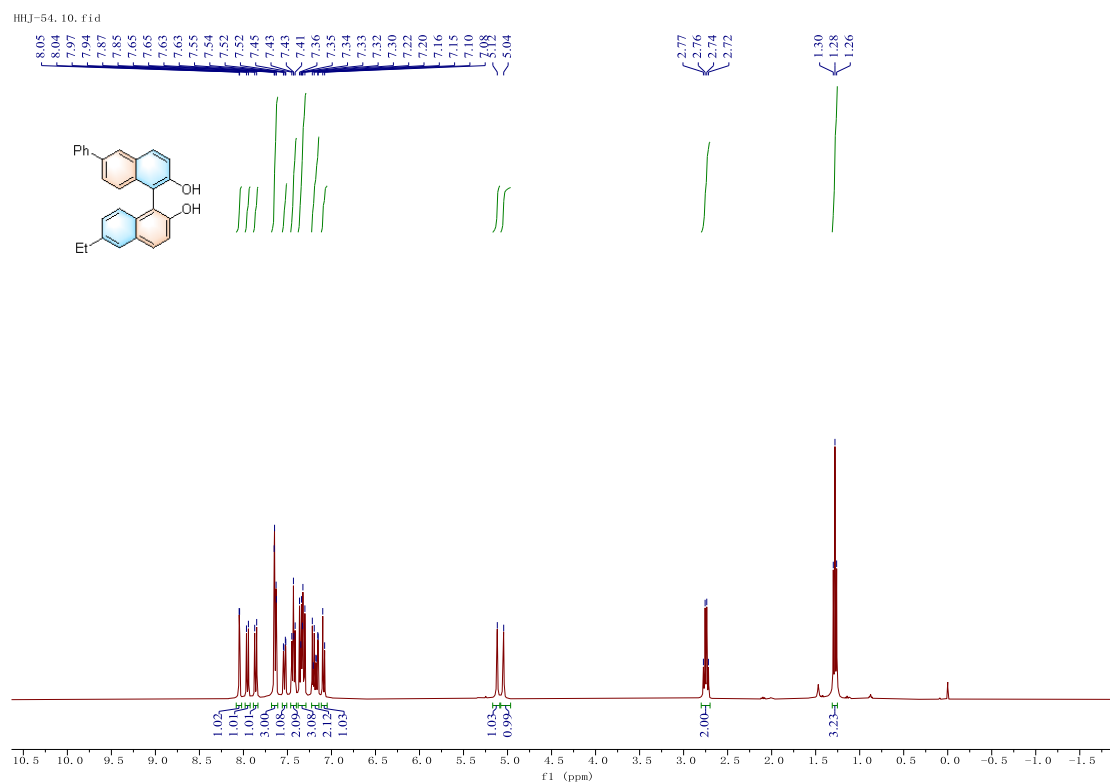


Figure S107. ^1H NMR Spectrum of 3al

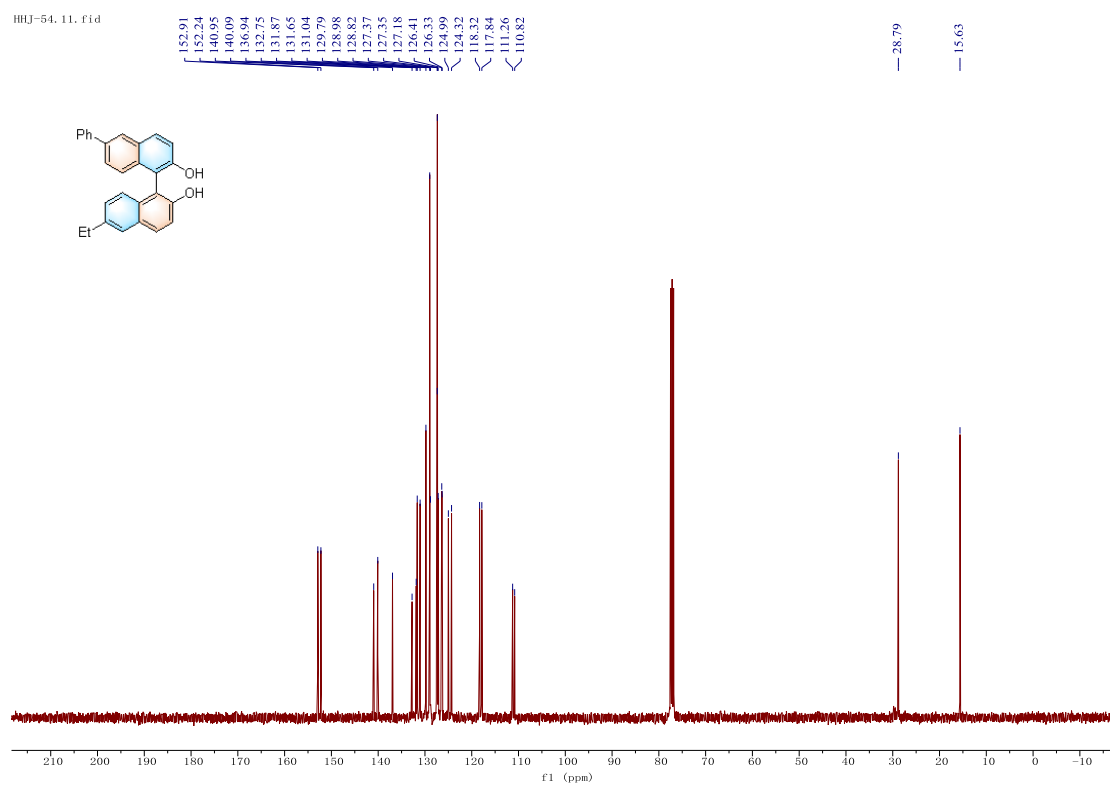


Figure S108. ^{13}C NMR Spectrum of 3al

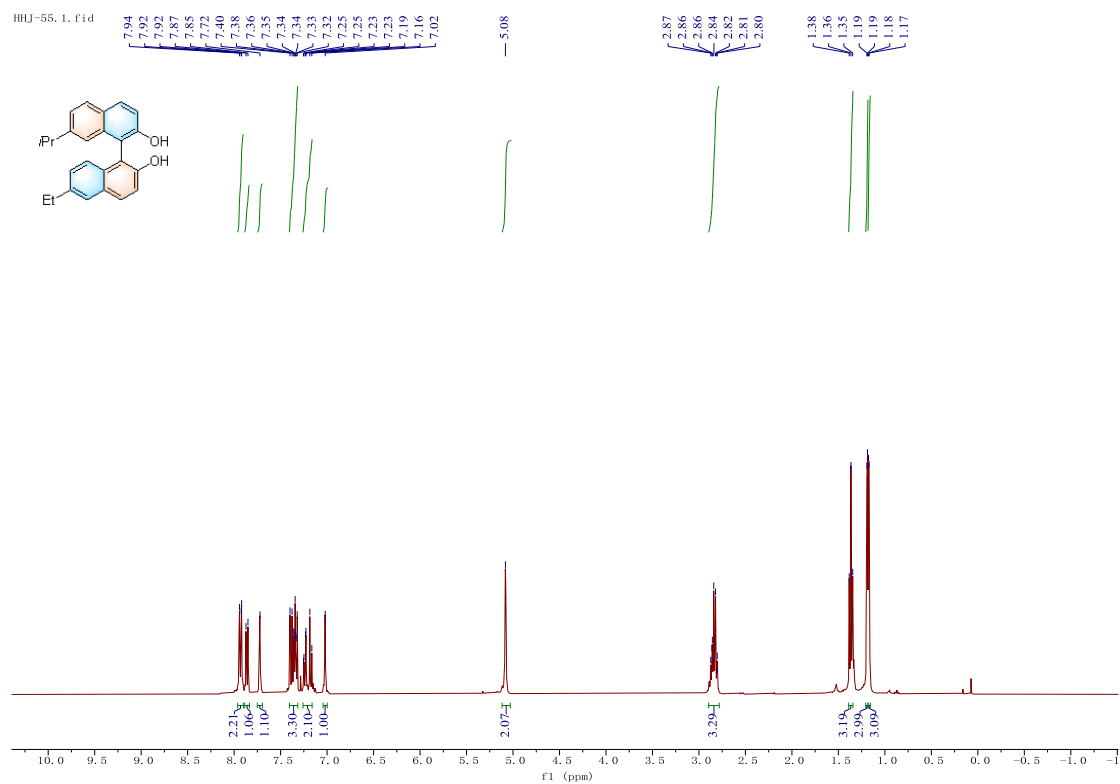


Figure S109. ^1H NMR Spectrum of **3am**

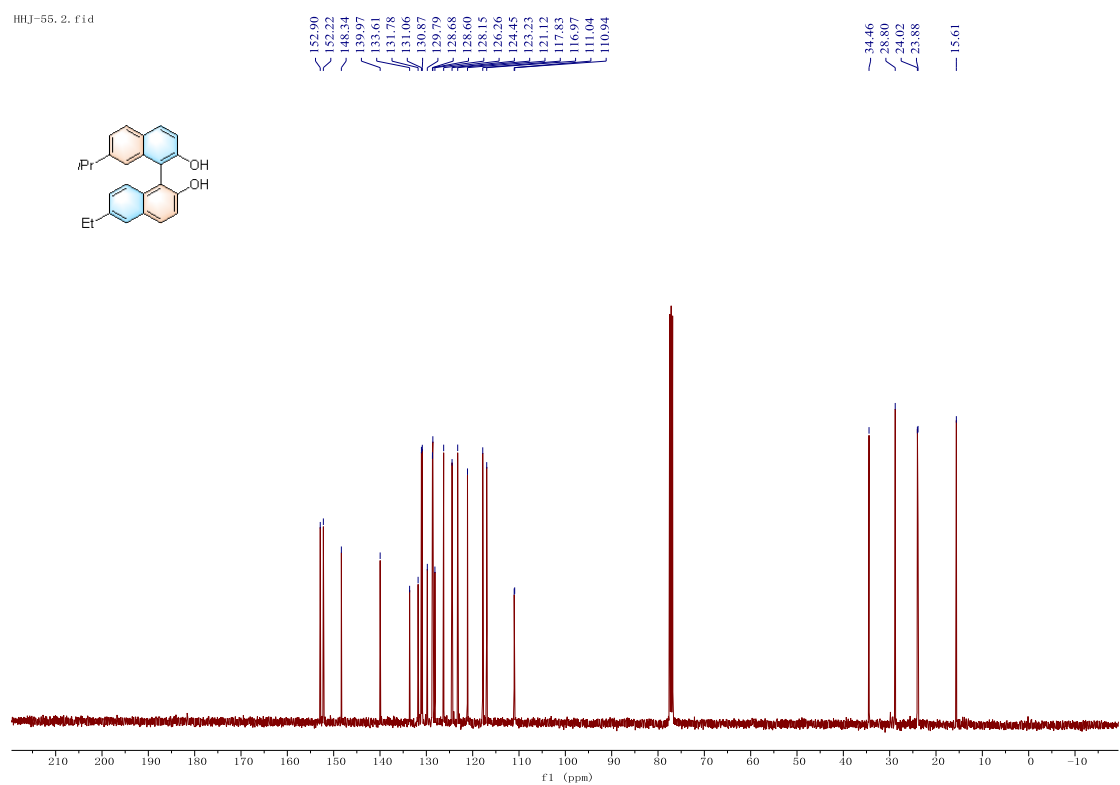


Figure S110. ^{13}C NMR Spectrum of **3am**

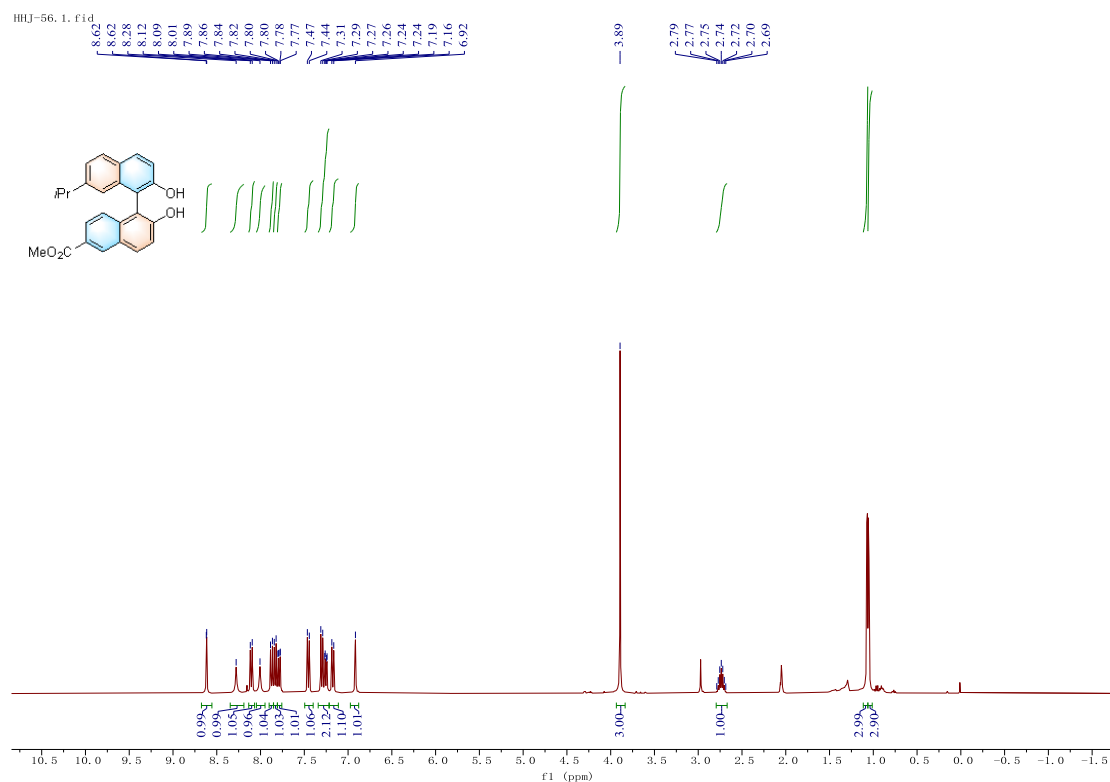


Figure S11. ^1H NMR Spectrum of **3an**

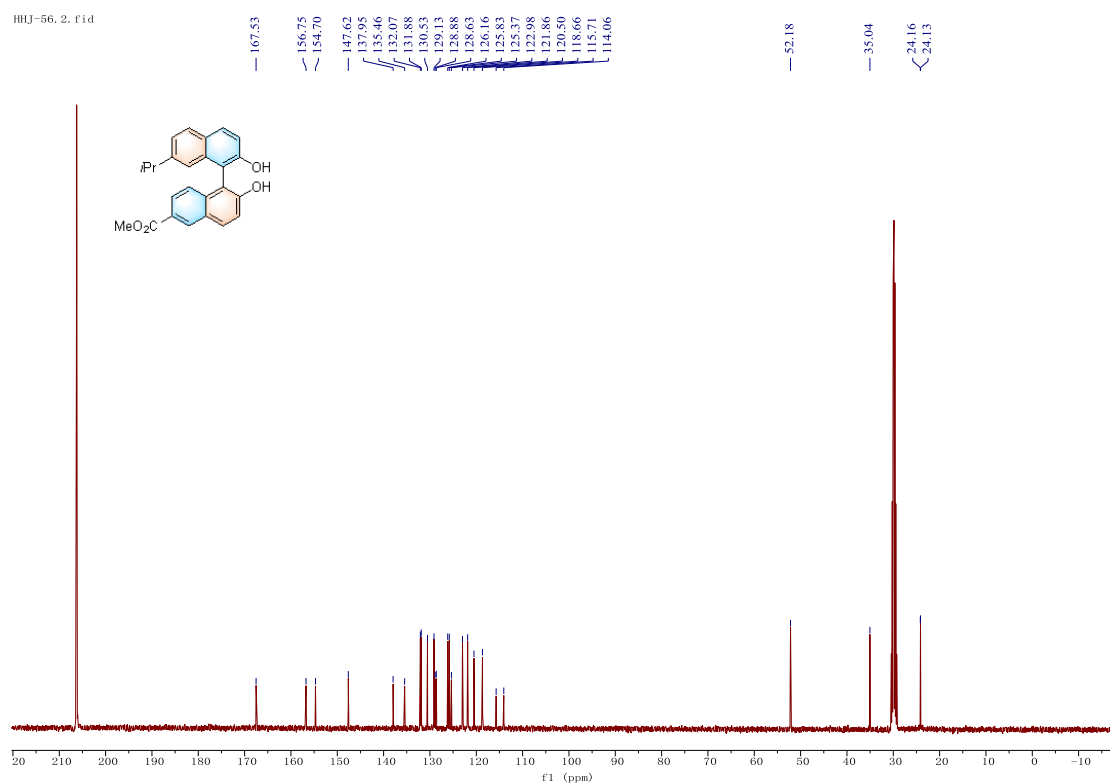


Figure S12. ^{13}C NMR Spectrum of **3an**

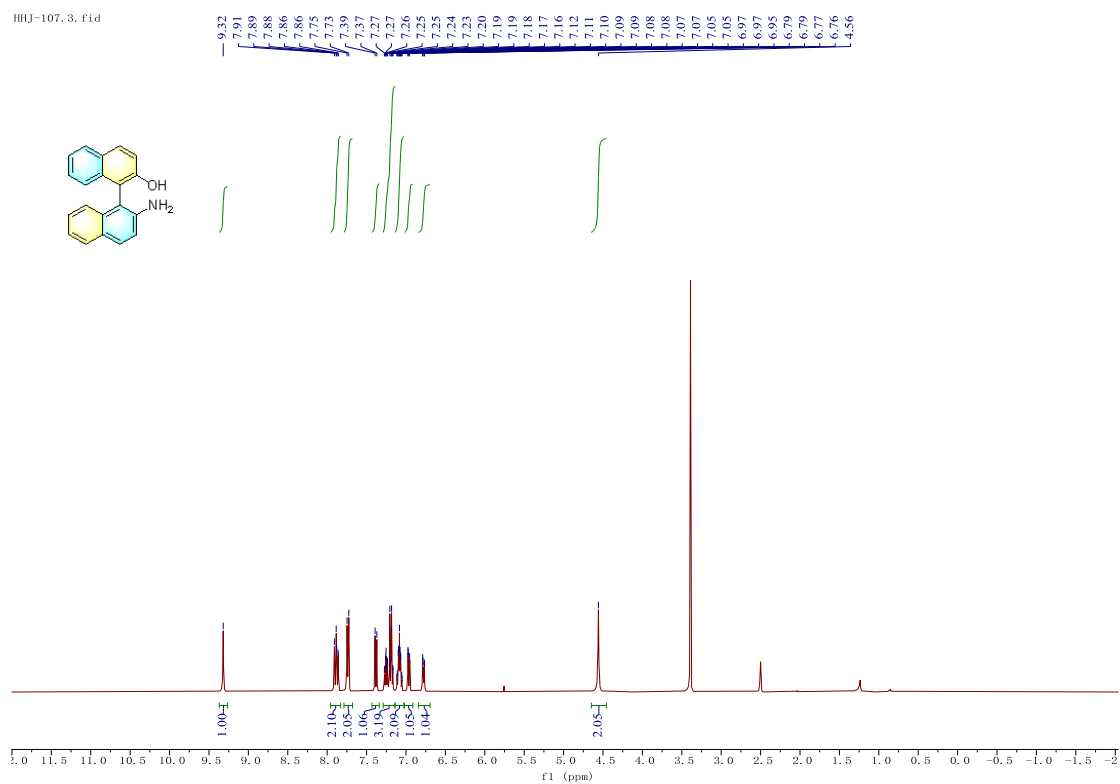


Figure S113. ^1H NMR Spectrum of **5a**

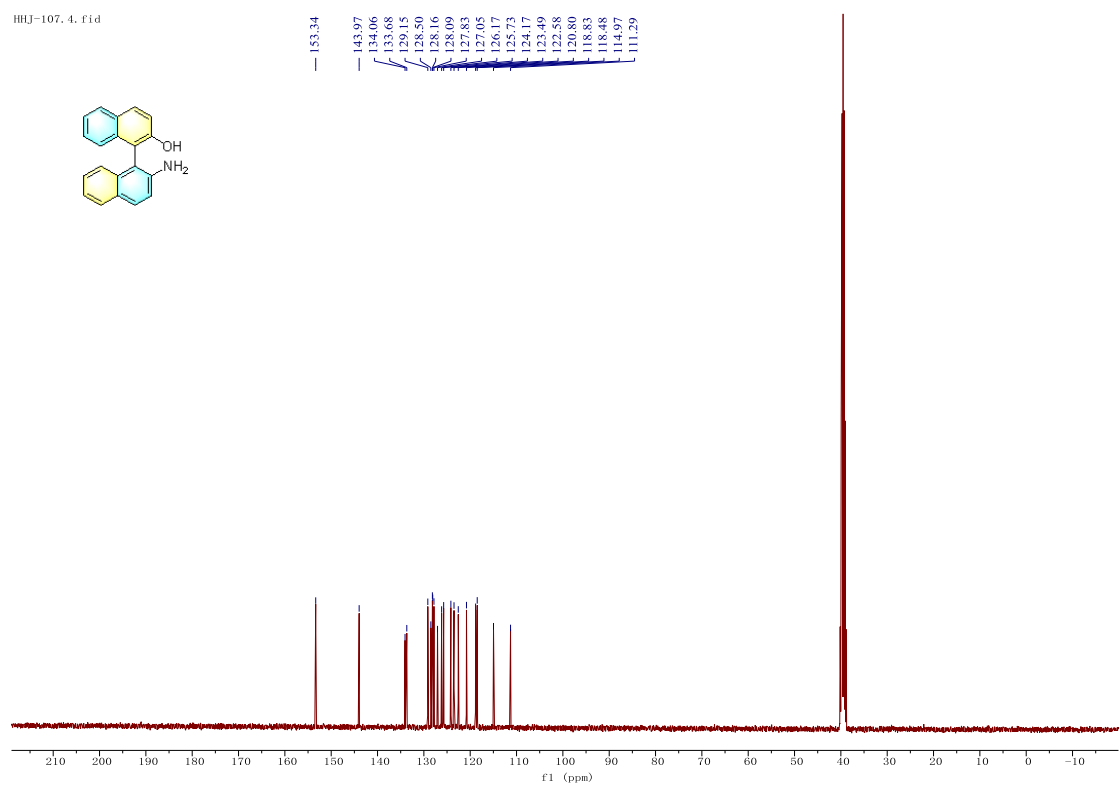


Figure S114. ^{13}C NMR Spectrum of **5a**

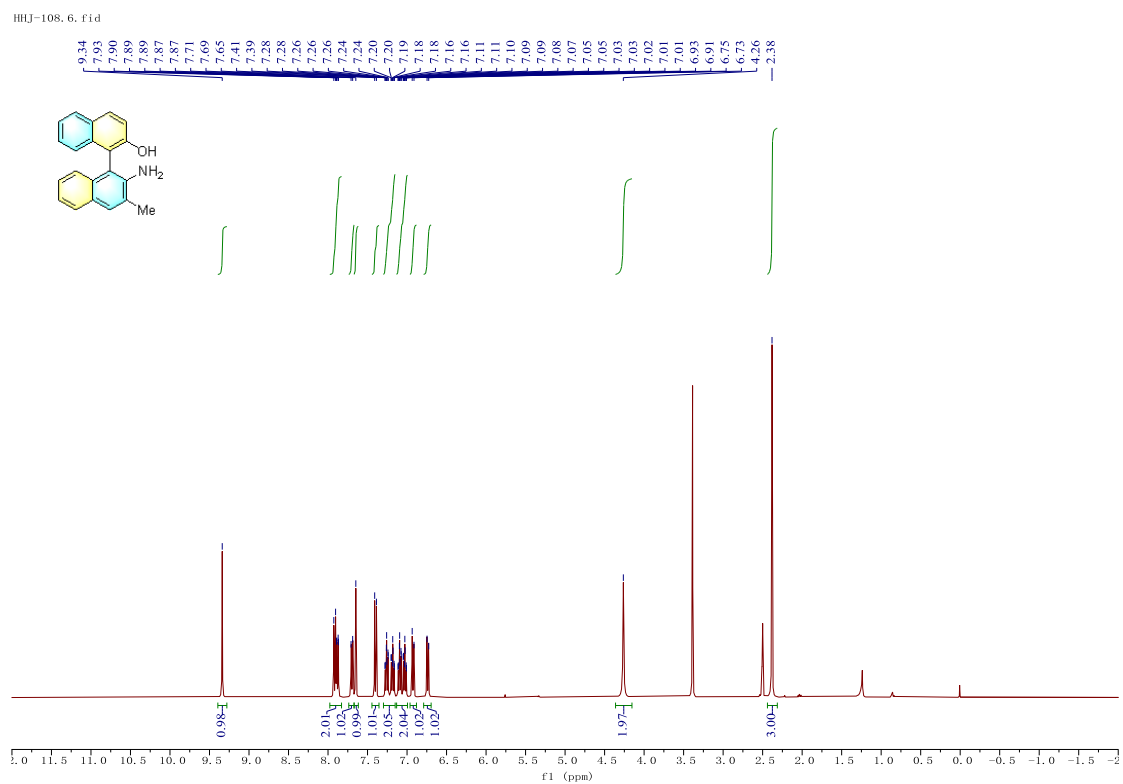


Figure S115. ¹H NMR Spectrum of 5b

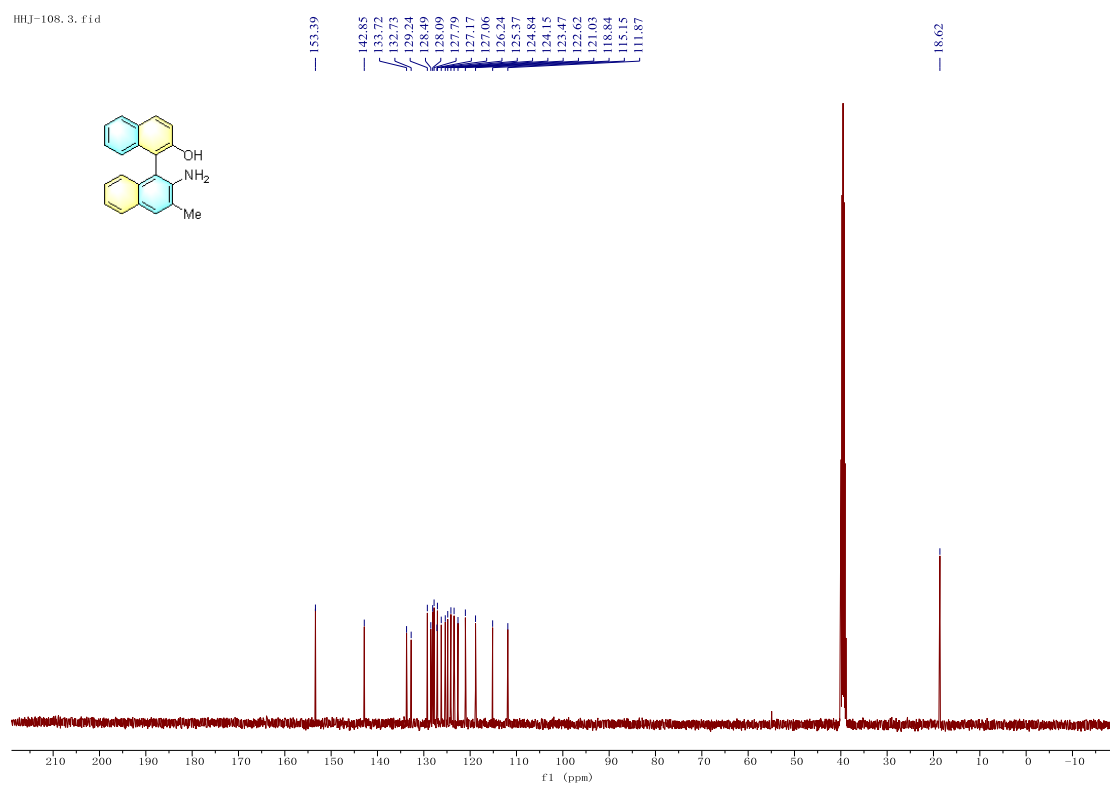
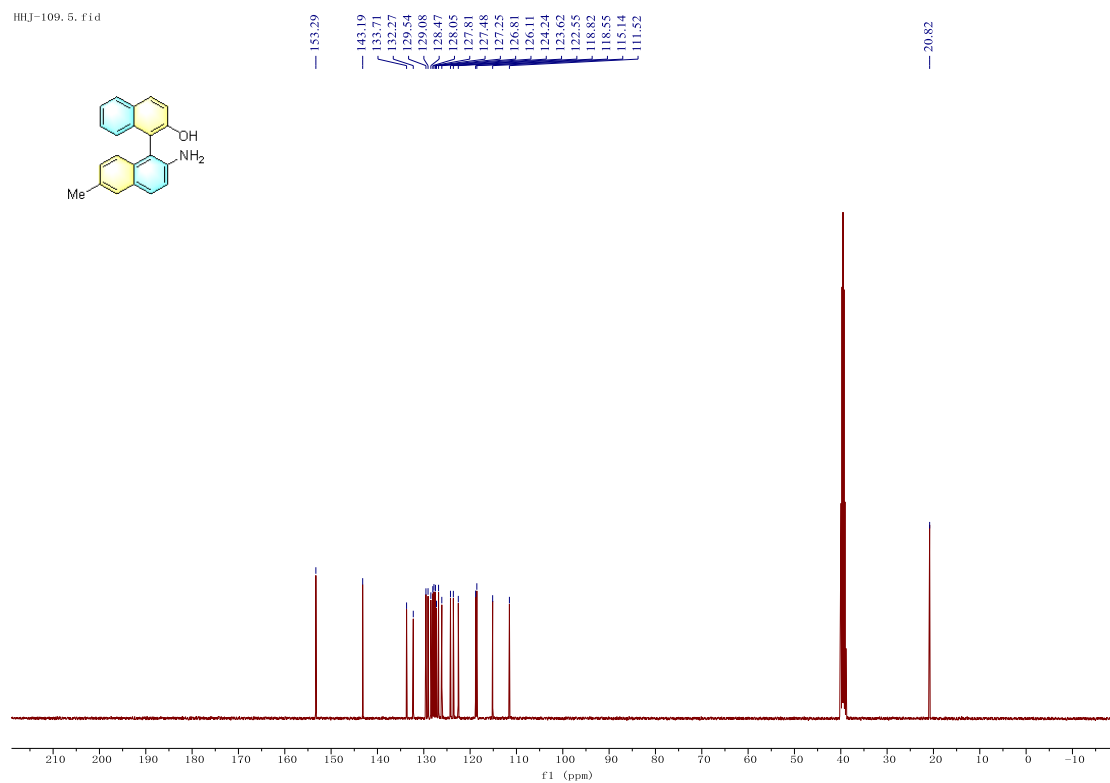
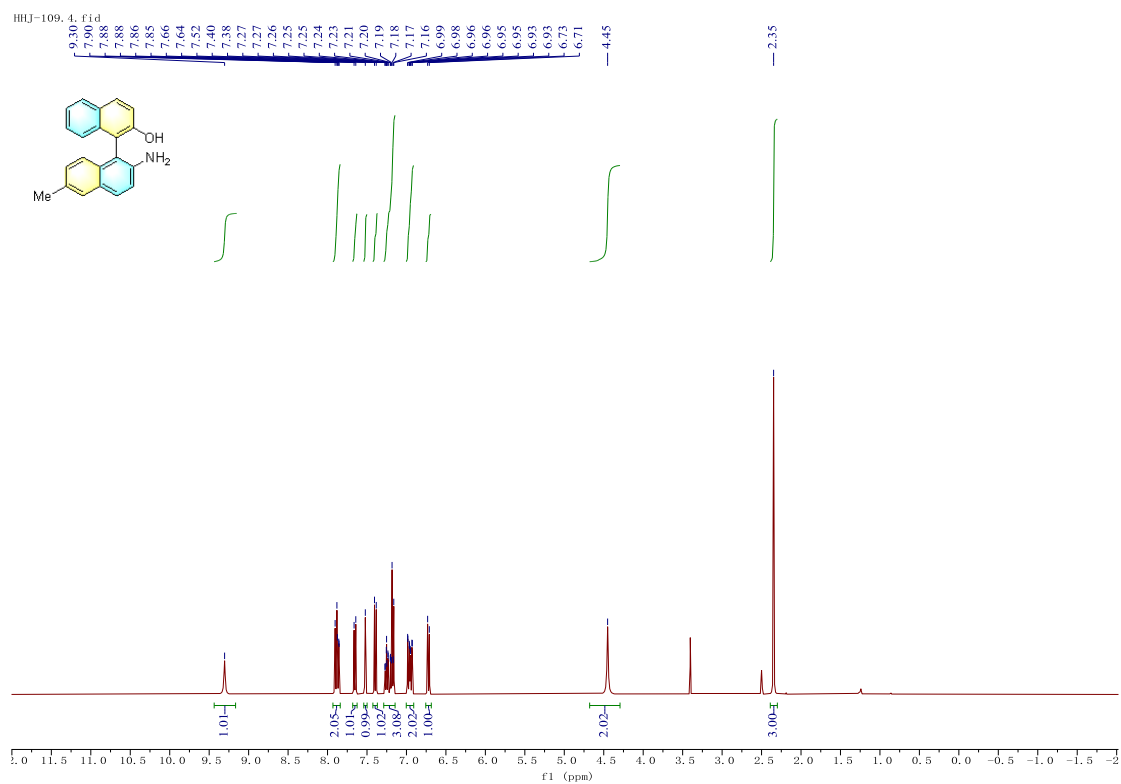


Figure S116. ¹³C NMR Spectrum of 5b



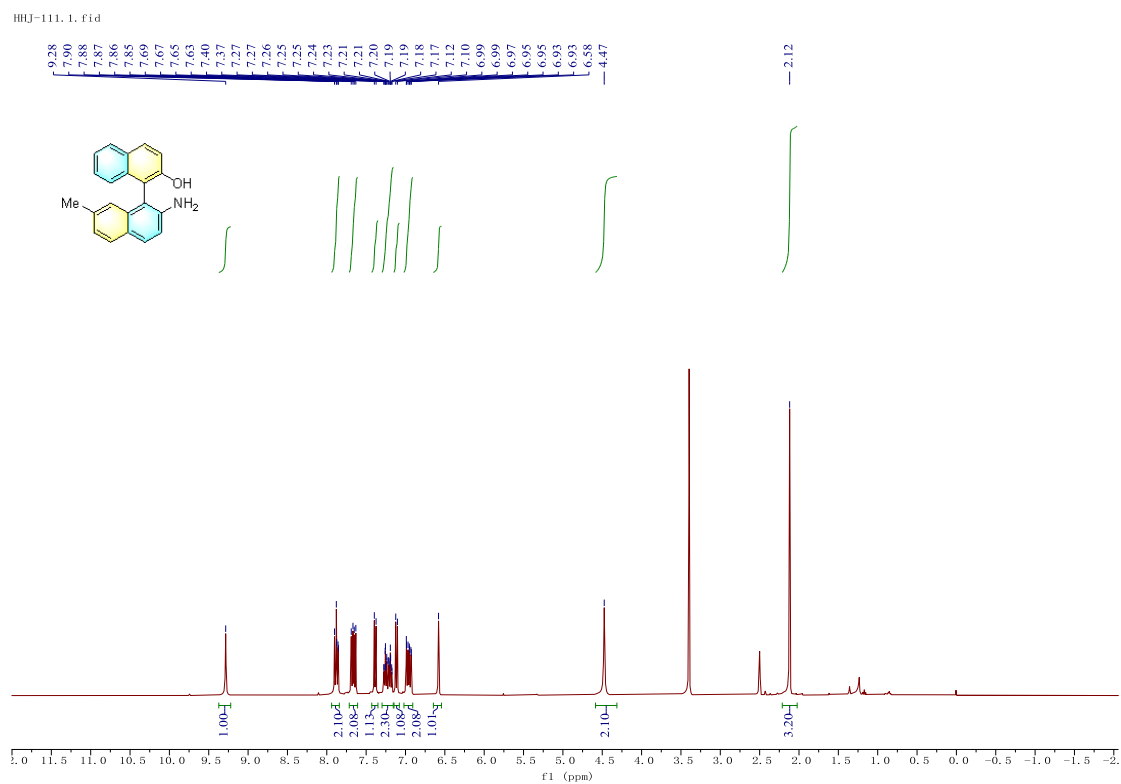


Figure S119. ^1H NMR Spectrum of **5d**

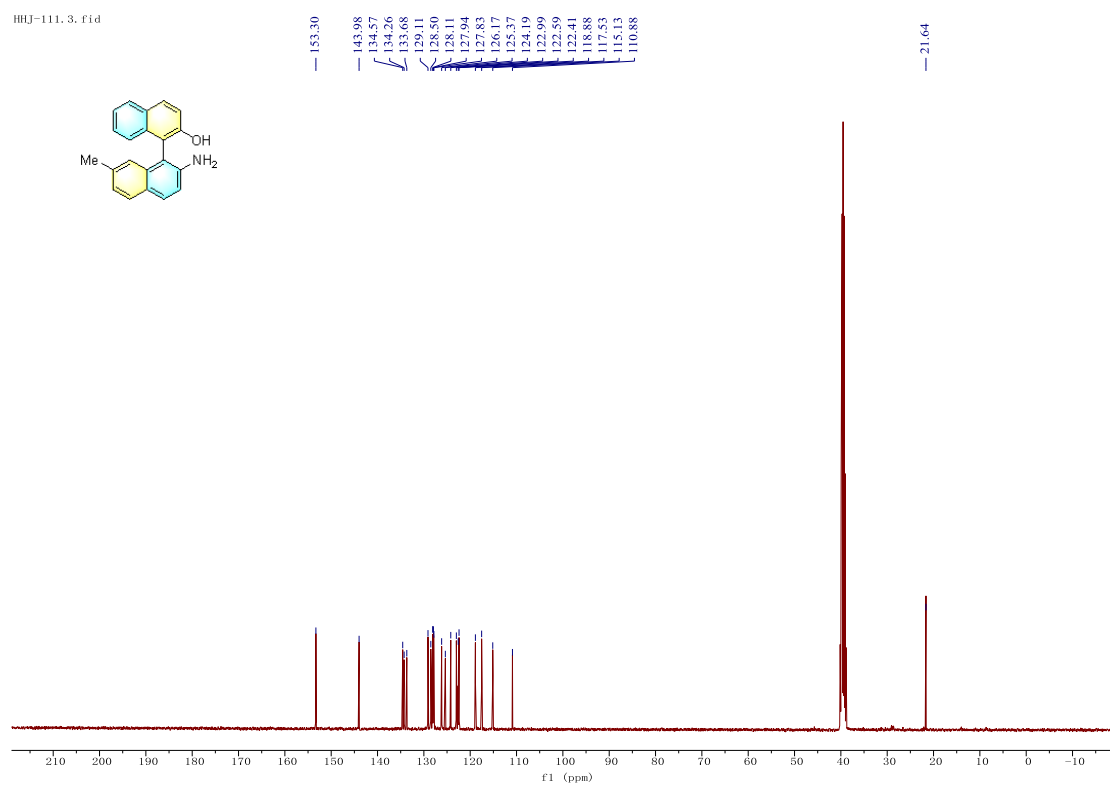


Figure S120. ^{13}C NMR Spectrum of **5d**

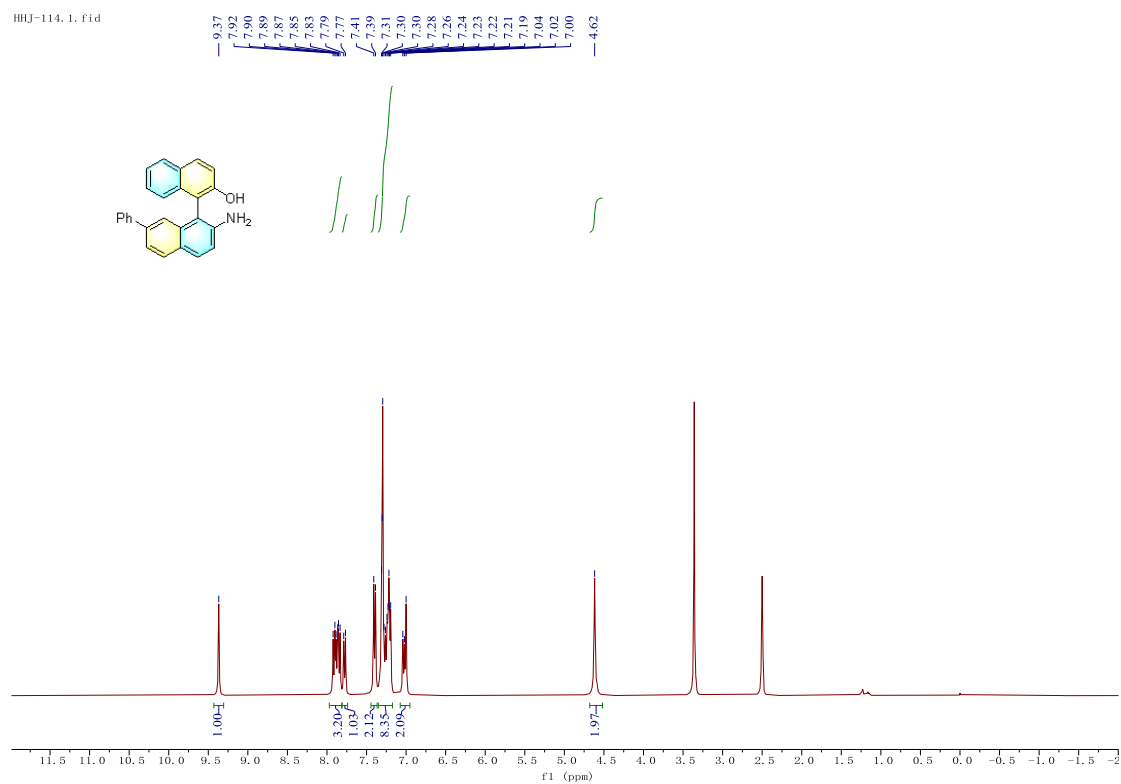


Figure S121. ^1H NMR Spectrum of **5e**

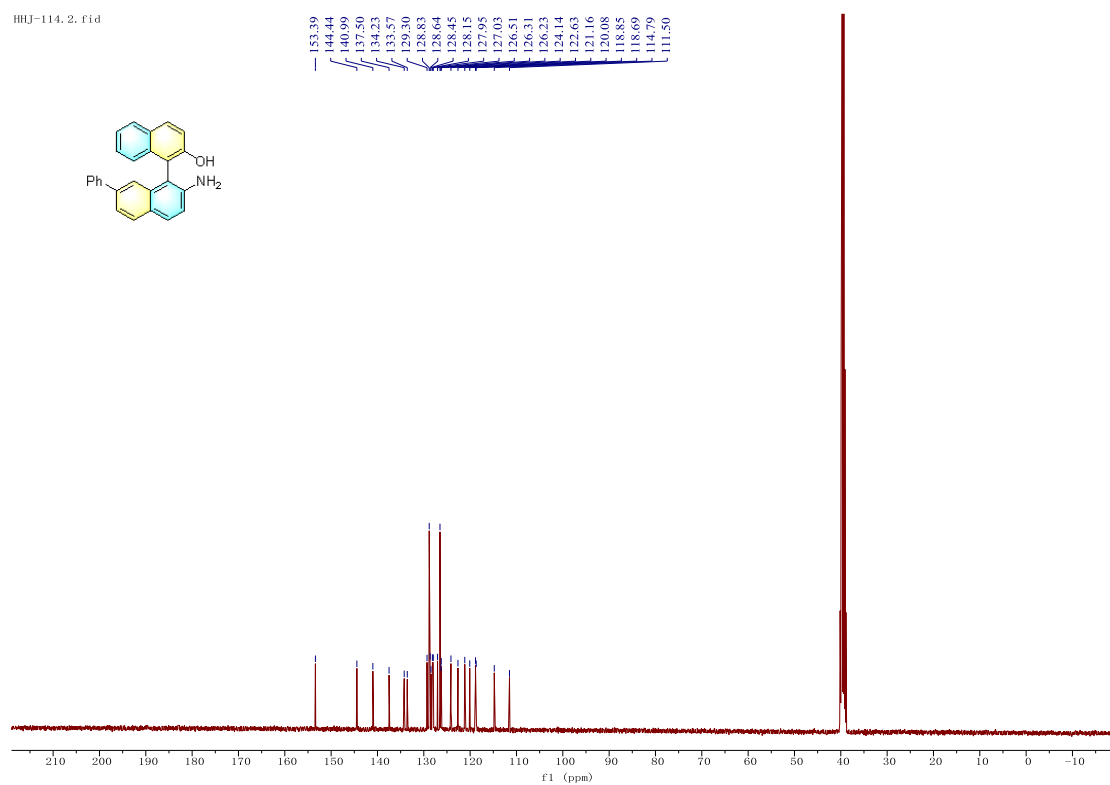


Figure S122. ^{13}C NMR Spectrum of **5e**

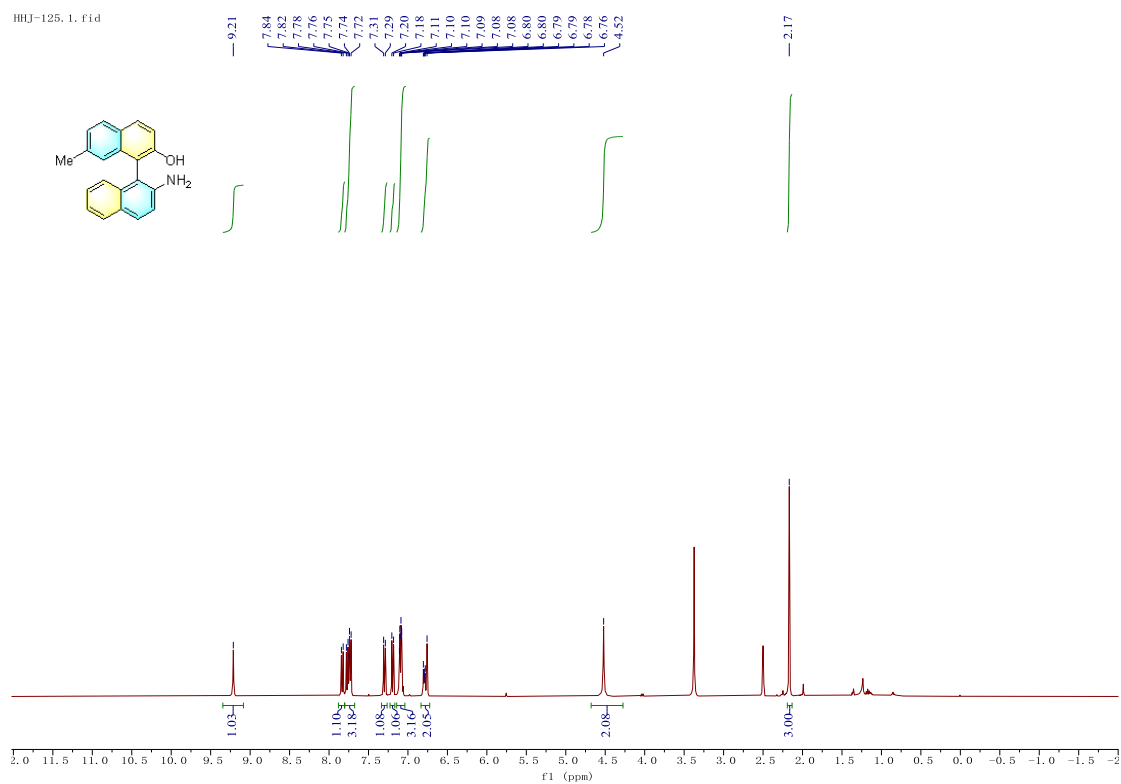


Figure S123. ¹H NMR Spectrum of **5f**

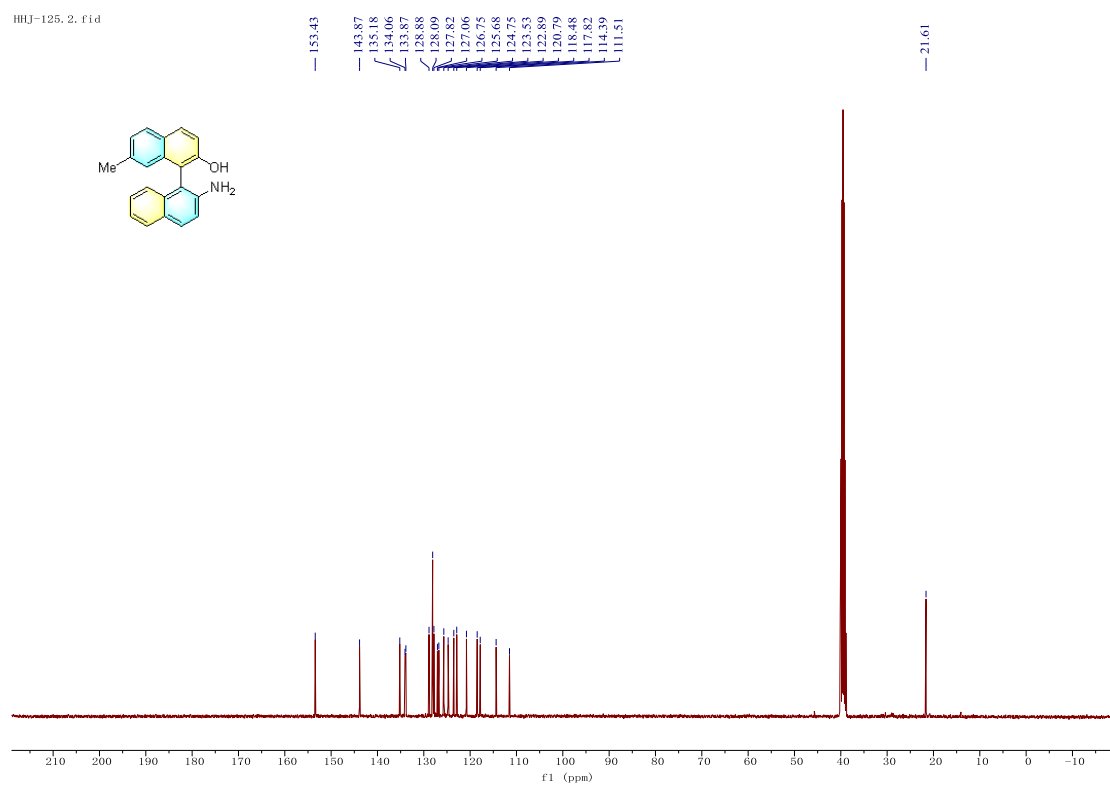


Figure S124. ¹³C NMR Spectrum of **5f**

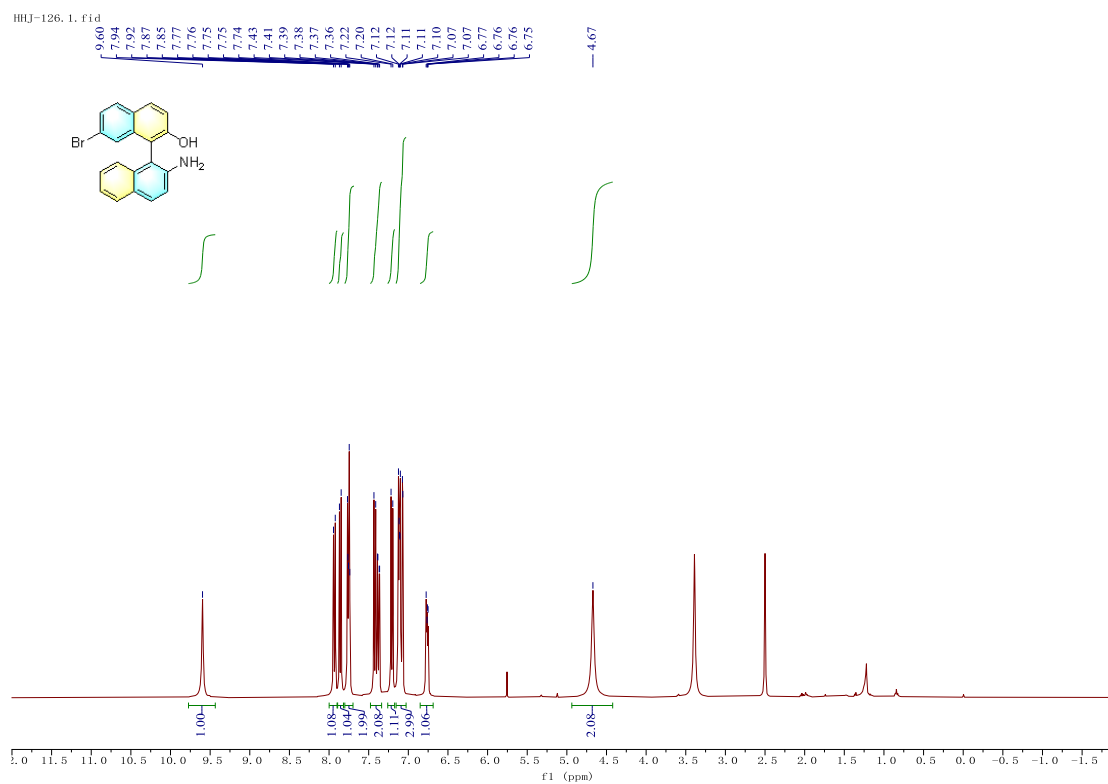


Figure S125. ¹H NMR Spectrum of **5g**

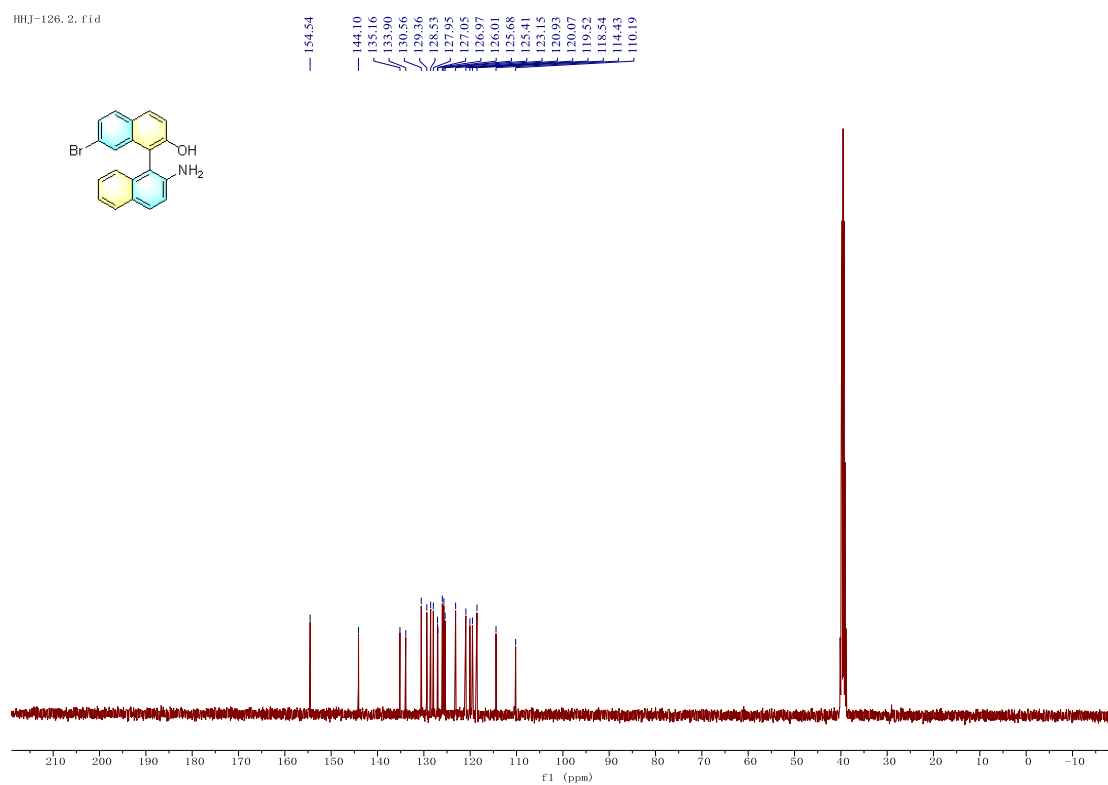


Figure S126. ¹³C NMR Spectrum of **5g**

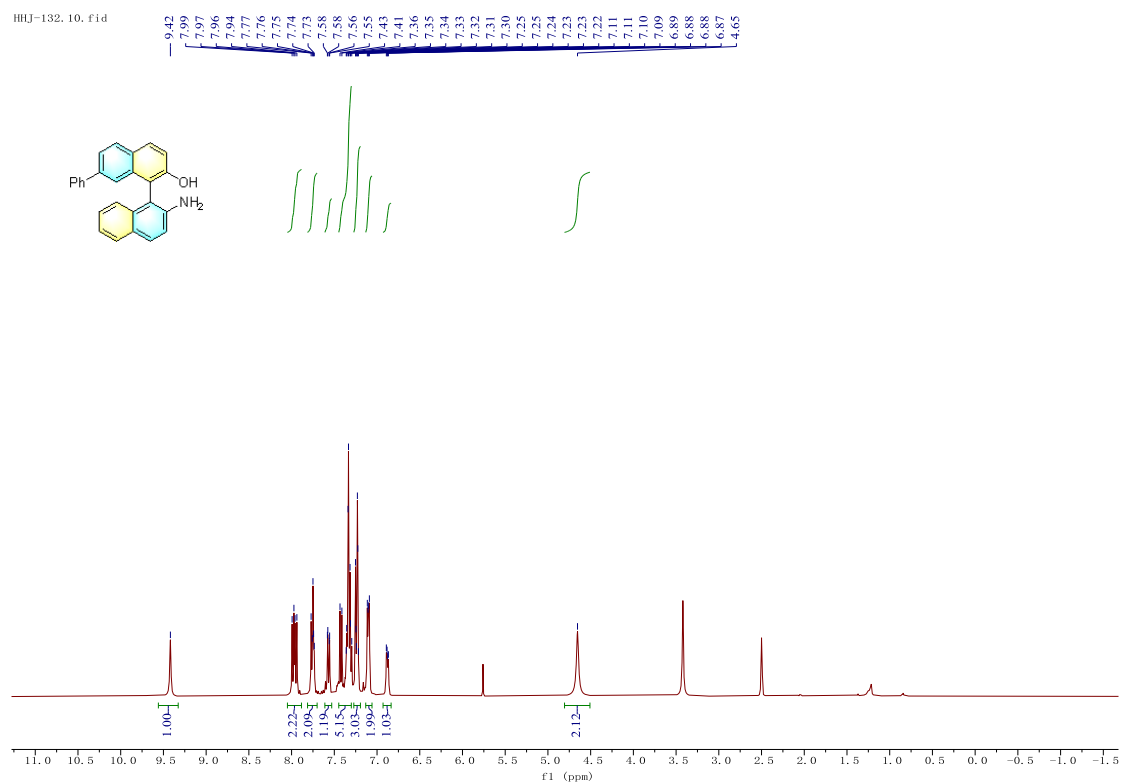


Figure S127. ¹H NMR Spectrum of **5h**

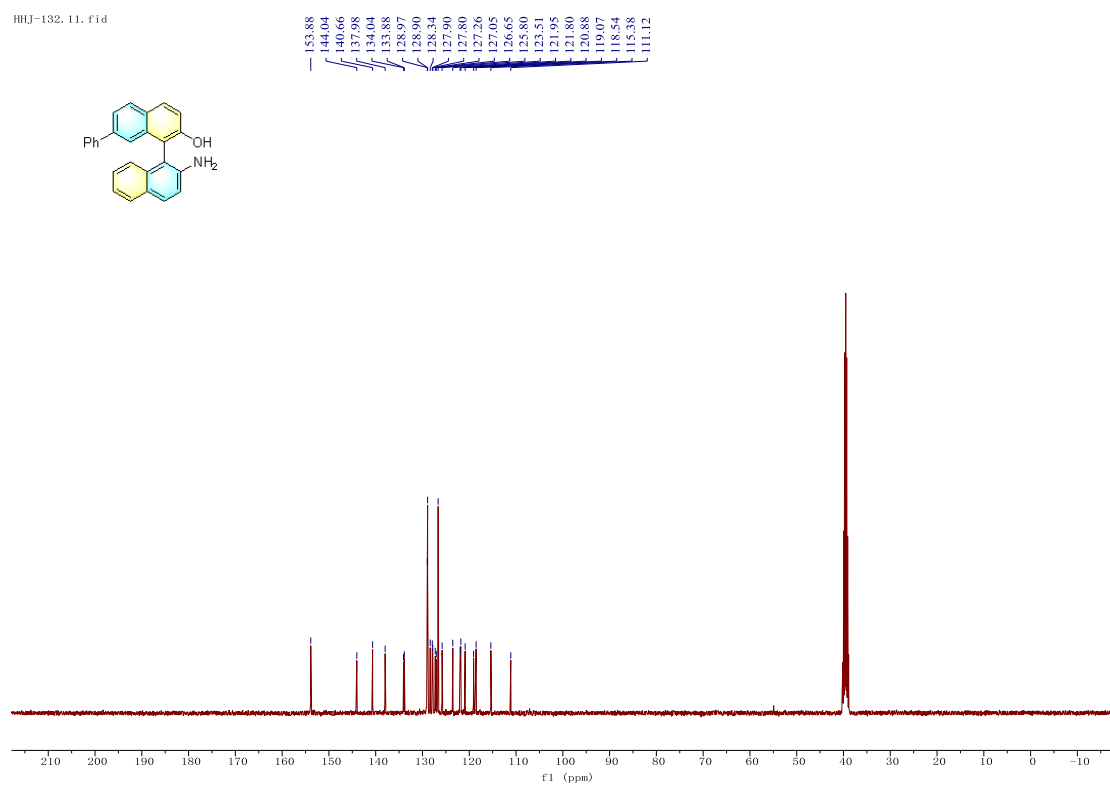


Figure S128. ¹³C NMR Spectrum of **5h**

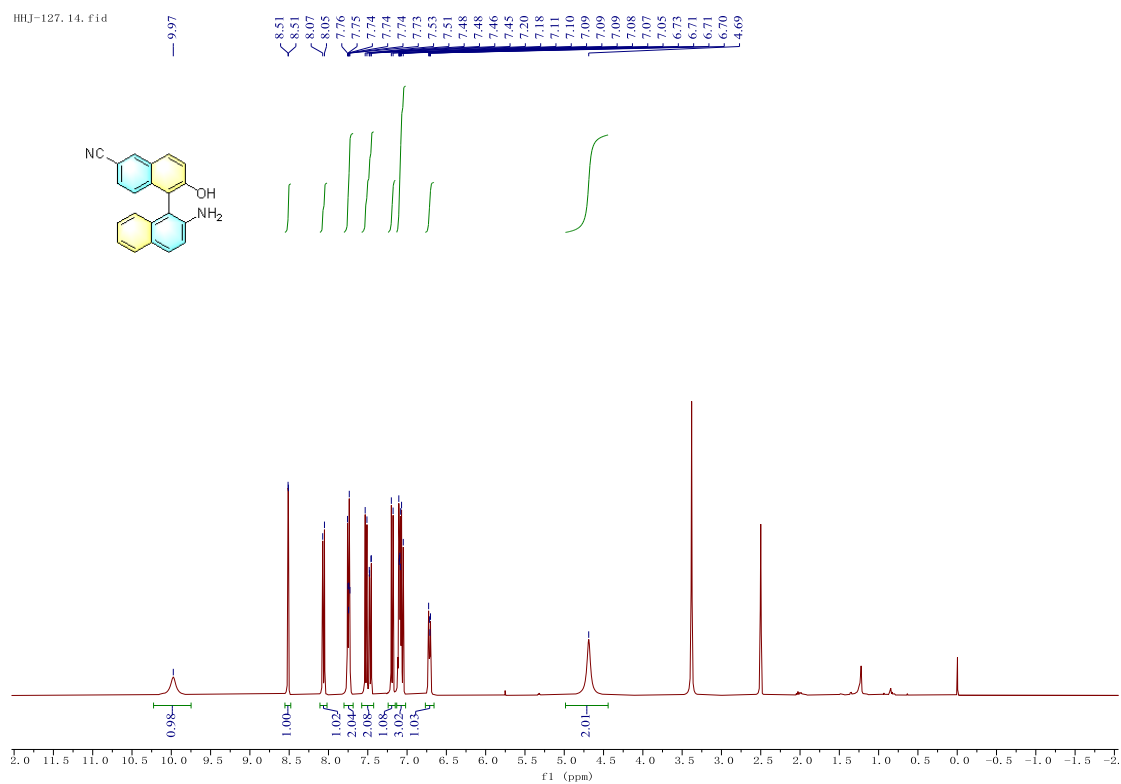


Figure S129. ¹H NMR Spectrum of **5i**

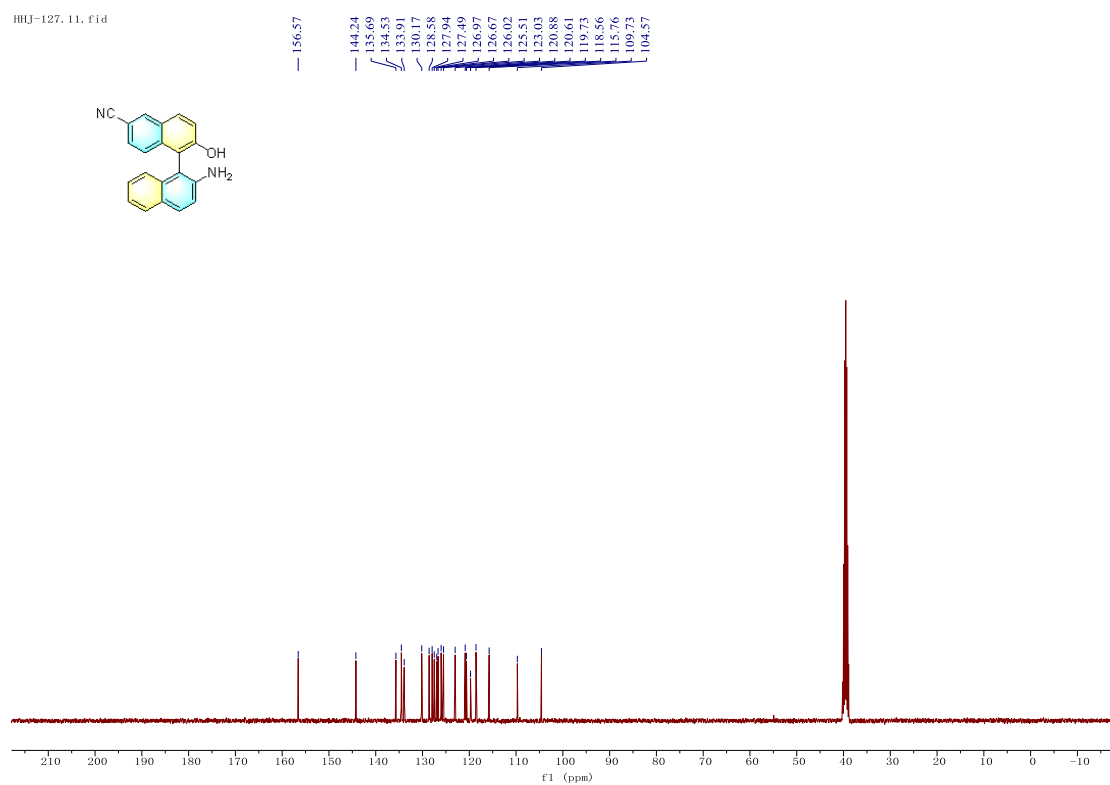


Figure S130. ¹³C NMR Spectrum of **5i**

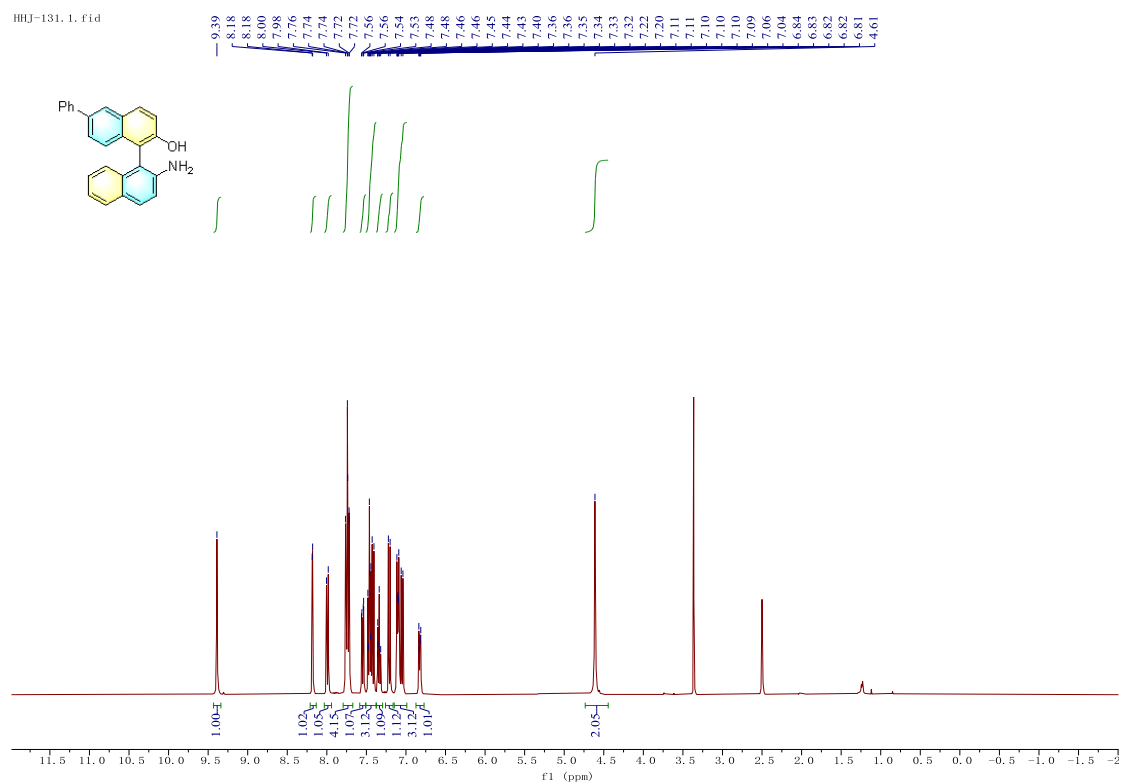


Figure S131. ¹H NMR Spectrum of **5j**

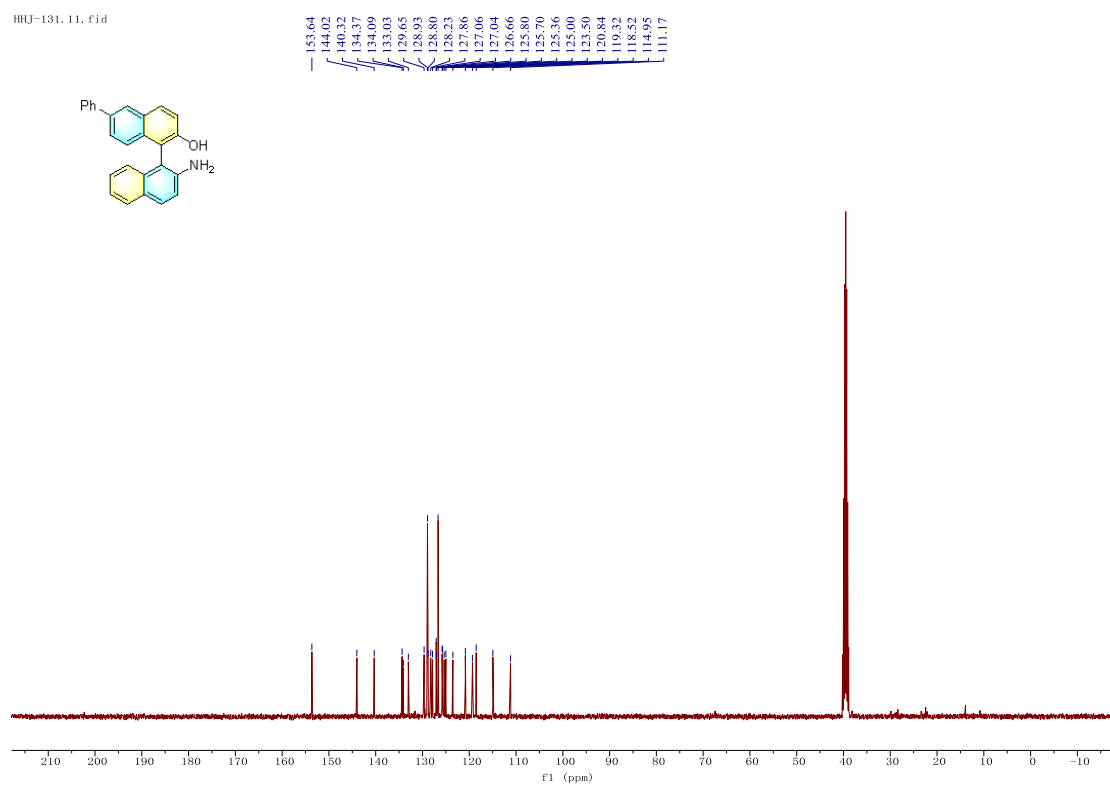


Figure S132. ¹³C NMR Spectrum of **5j**

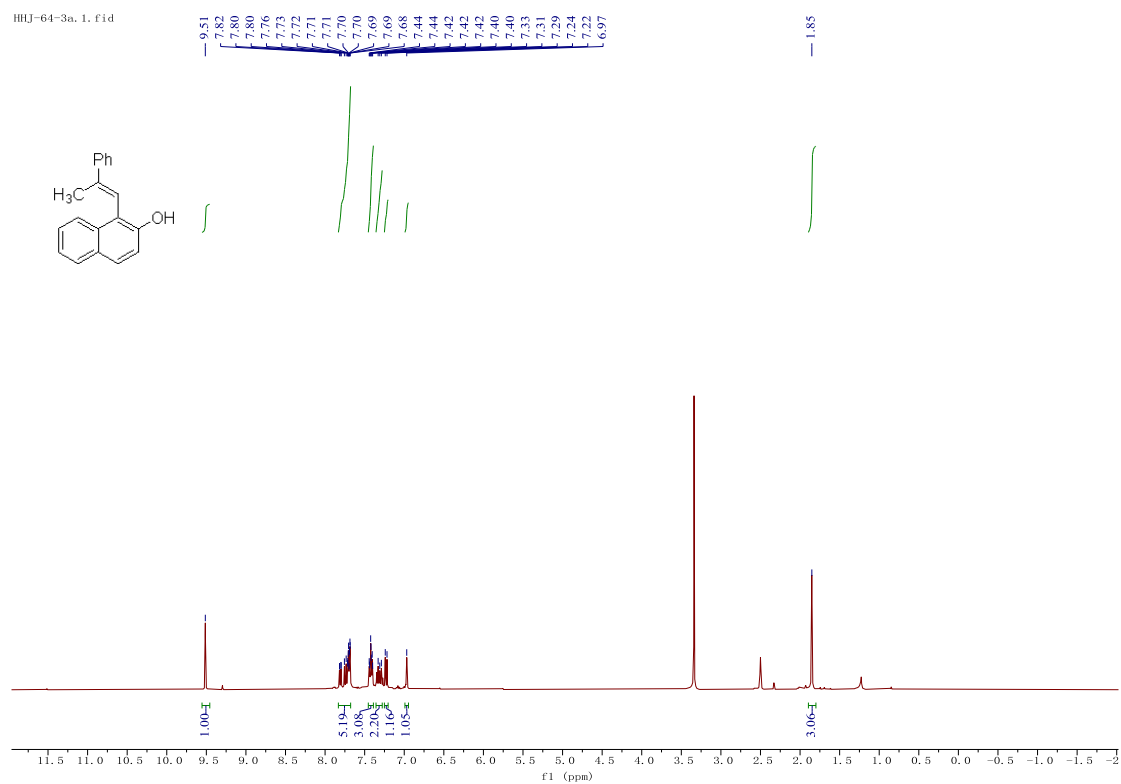


Figure S133. ¹H NMR Spectrum of **7**

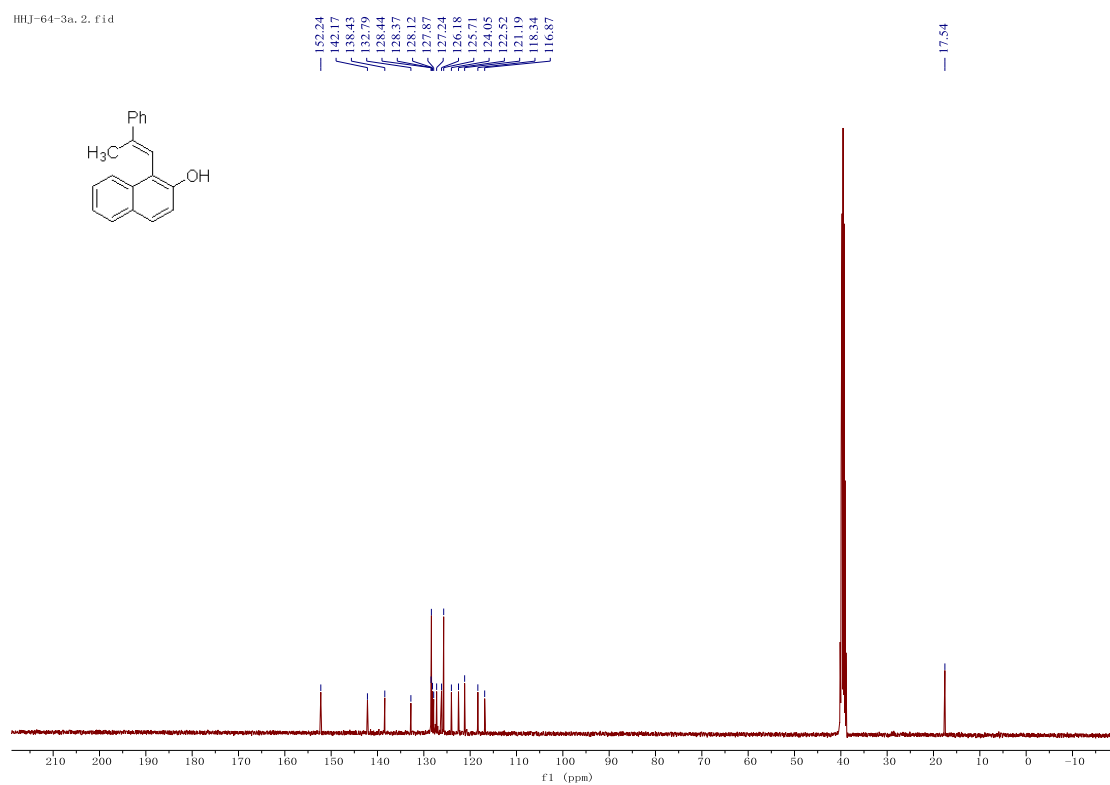


Figure S134. ¹³C NMR Spectrum of **7**

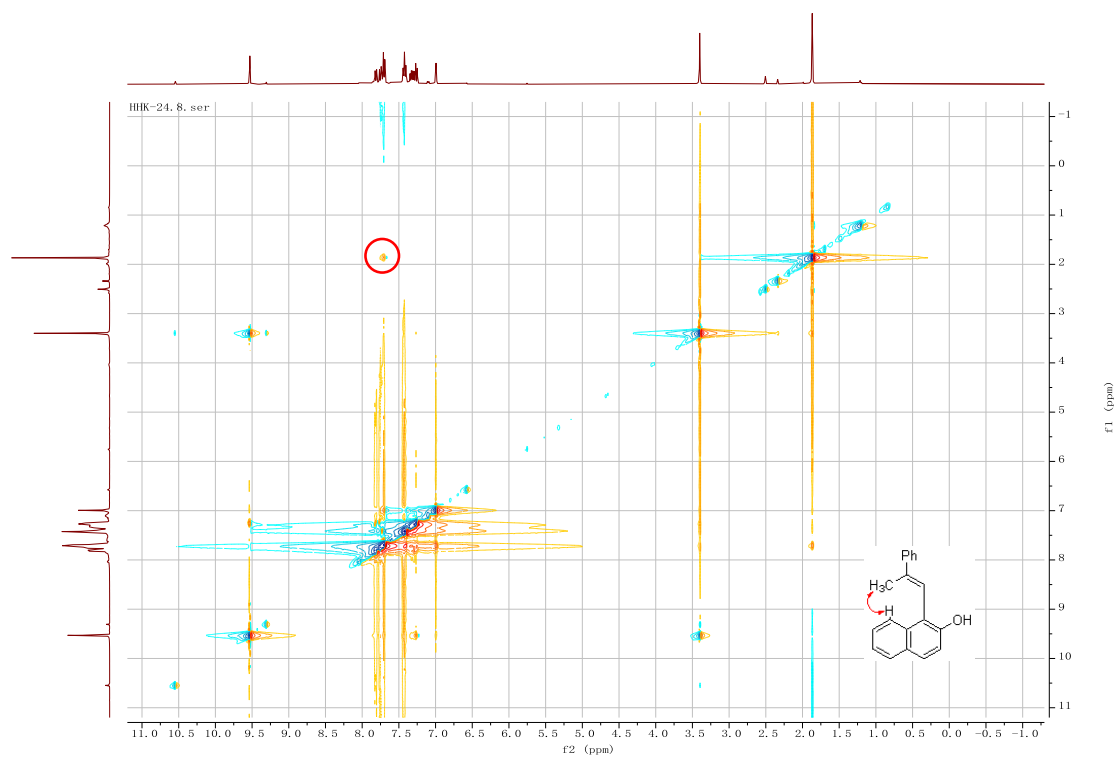


Figure S135. ^1H - ^1H NOESY NMR Spectrum of **7**

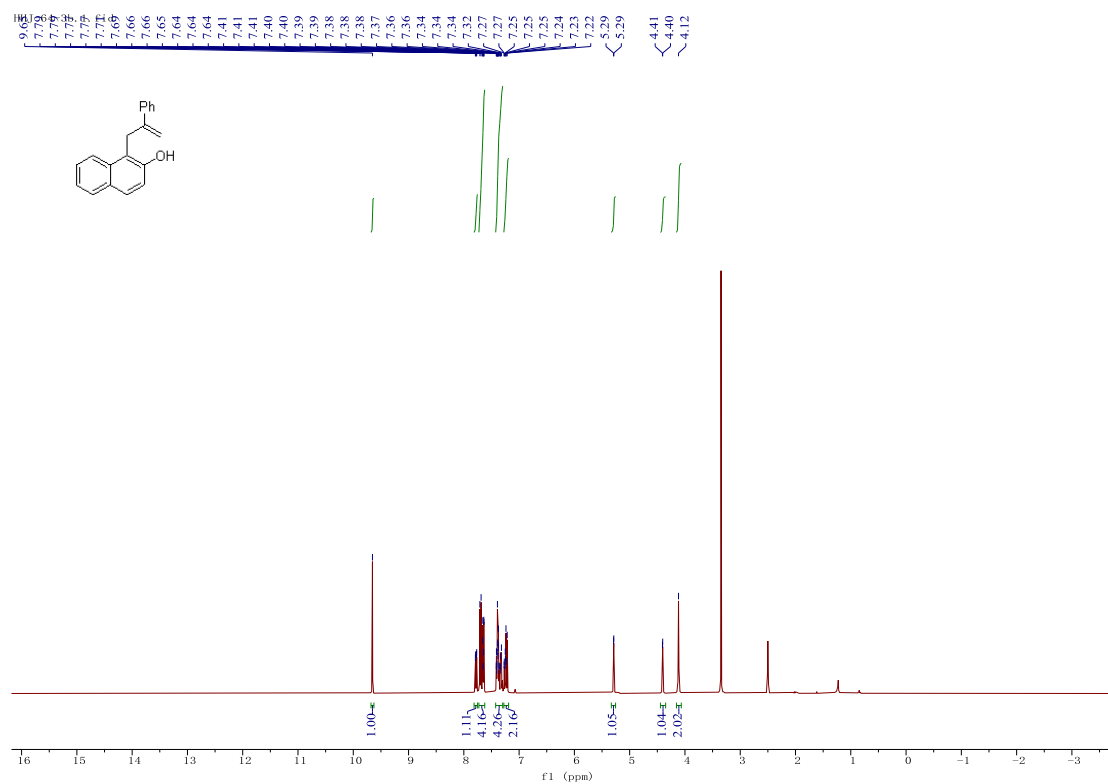


Figure S136. ¹H NMR Spectrum of 8

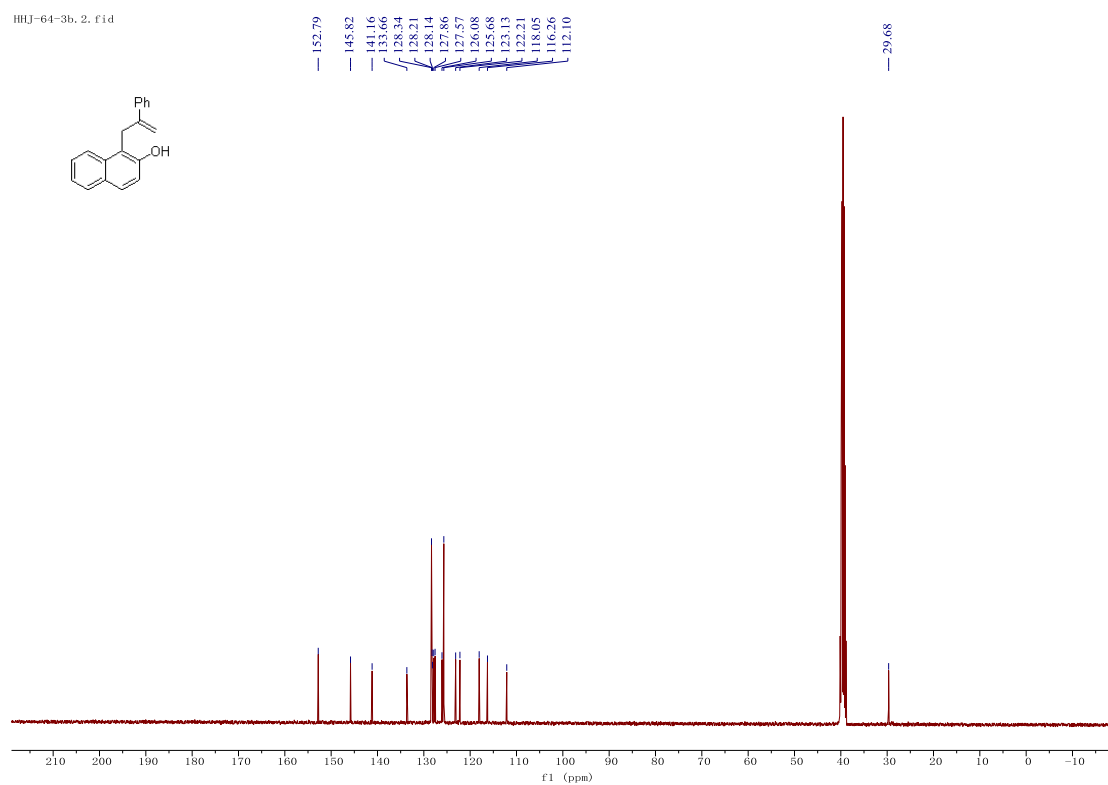


Figure S137. ¹³C NMR Spectrum of 8

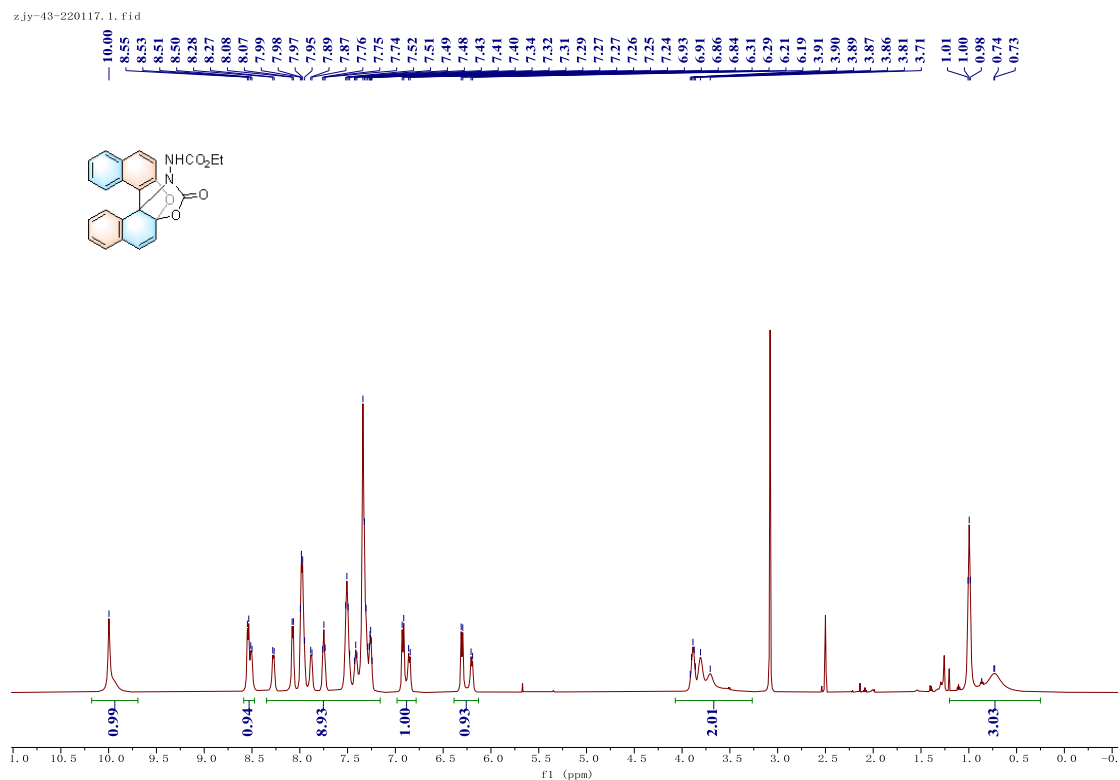


Figure S138. ¹H NMR Spectrum of 11a

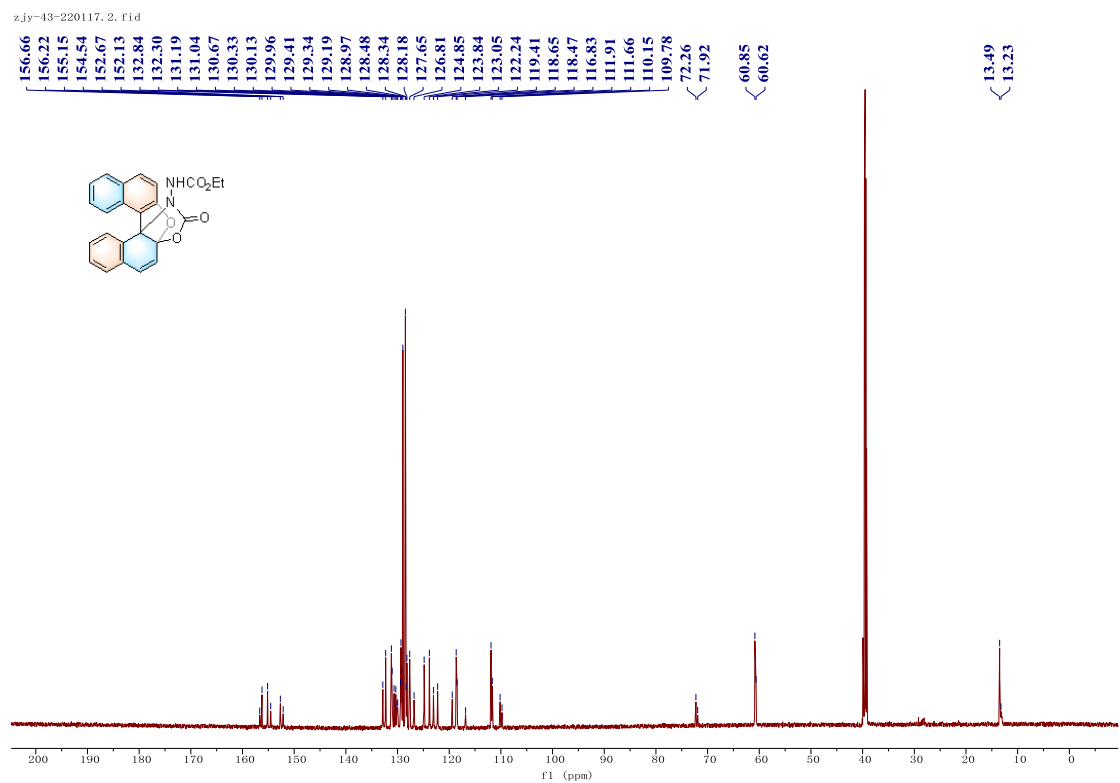


Figure S139. ¹³C NMR Spectrum of 11a

ZJY-105-7-BiOEt, 1, f1d

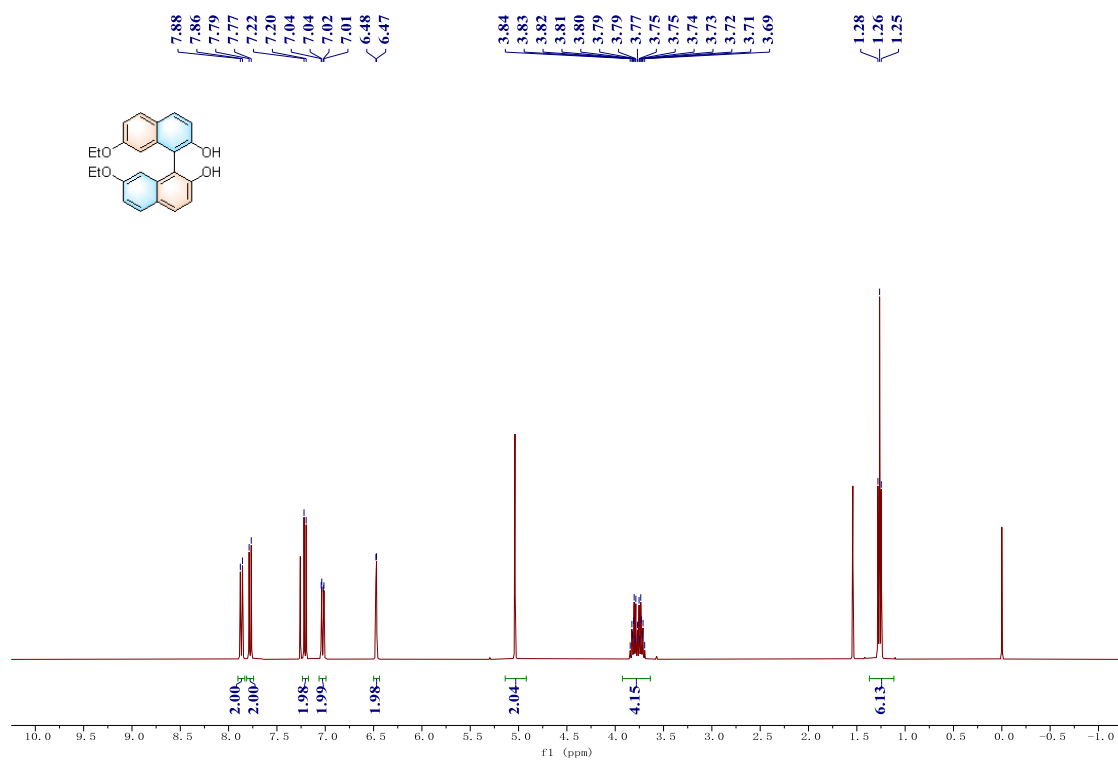


Figure S140. ¹H NMR Spectrum of 9b

ZJY-67-7-biOMe, 10, f1d

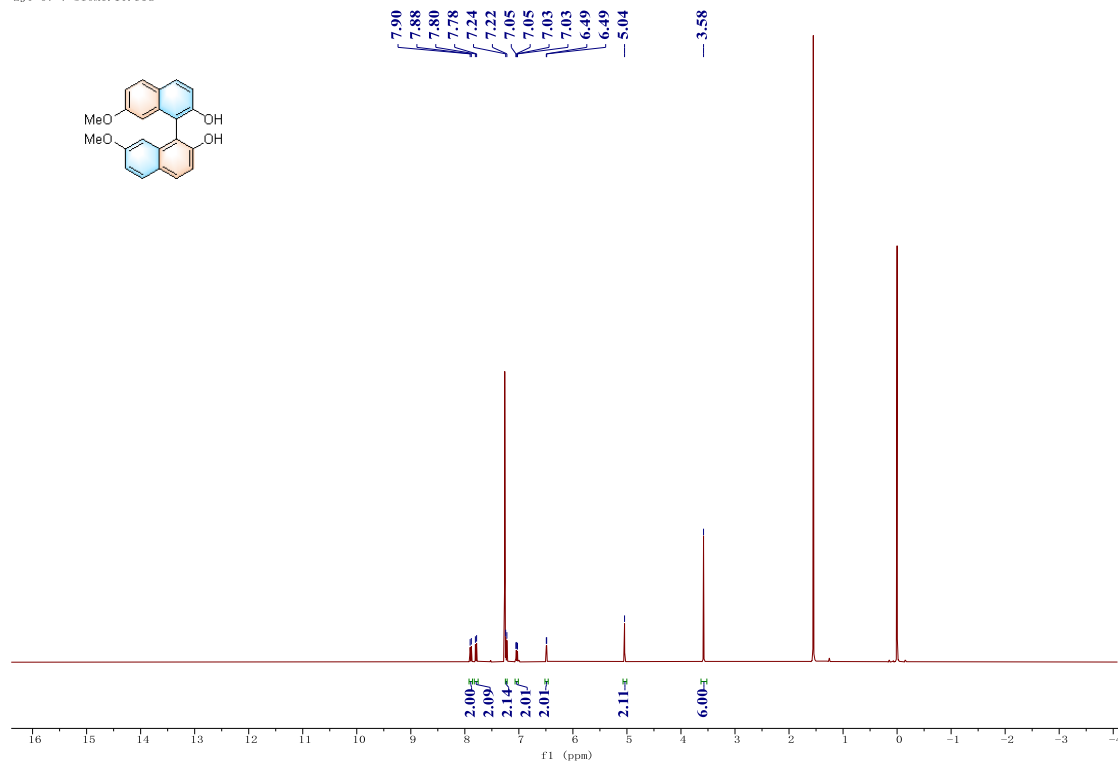


Figure S141. ¹H NMR Spectrum of 9c

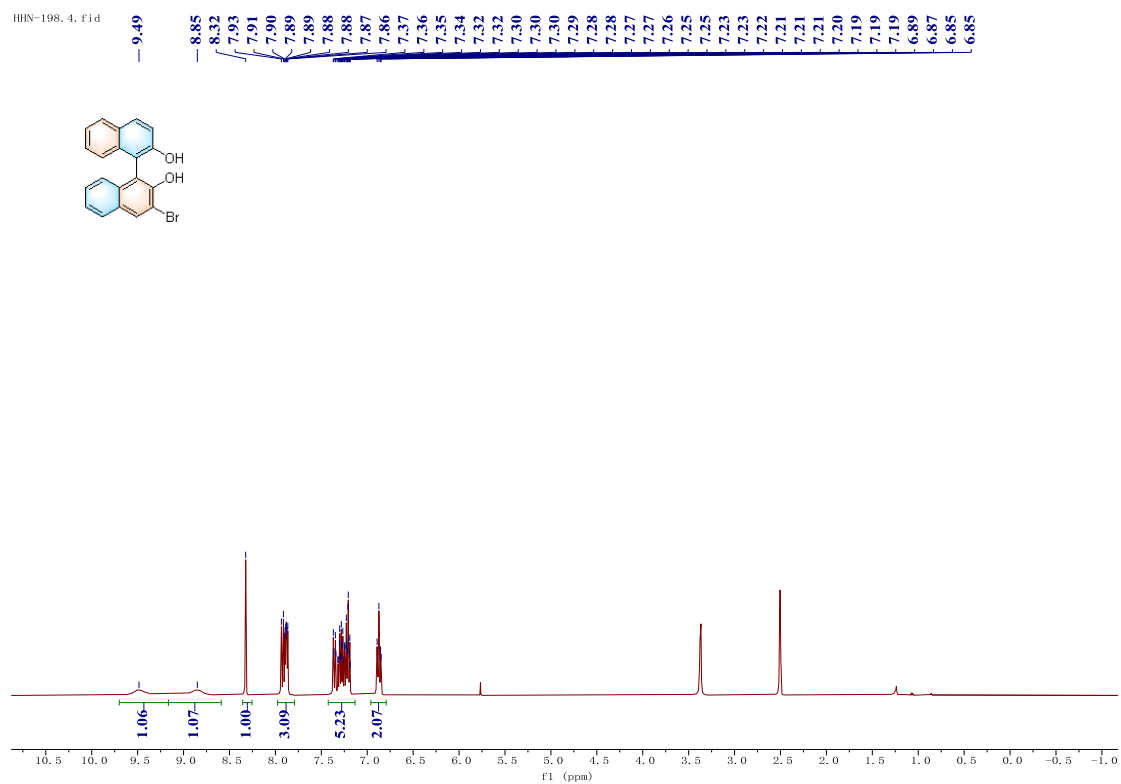


Figure S142. ^1H NMR Spectrum of **9h**

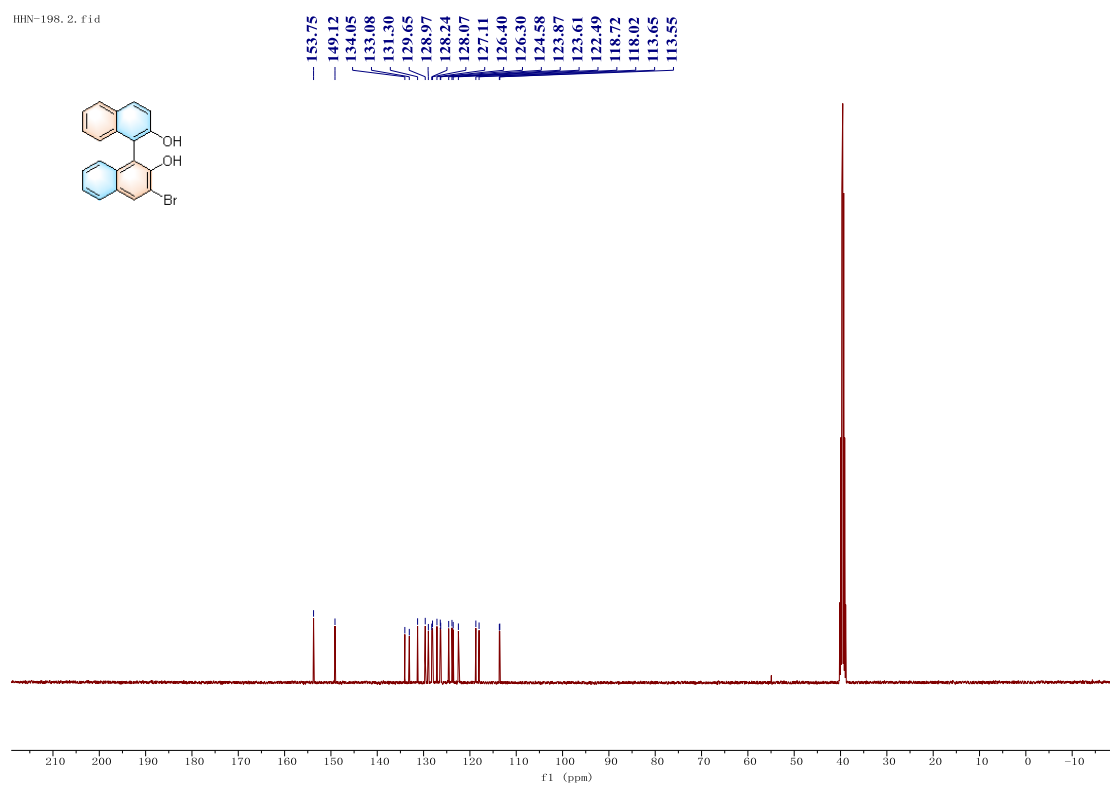


Figure S143. ^{13}C NMR Spectrum of **9h**