

Supporting Information

Electrochemical 1,2-hydrogen atom transfer functionalizations of *N*- (benzyloxy)phthalimides

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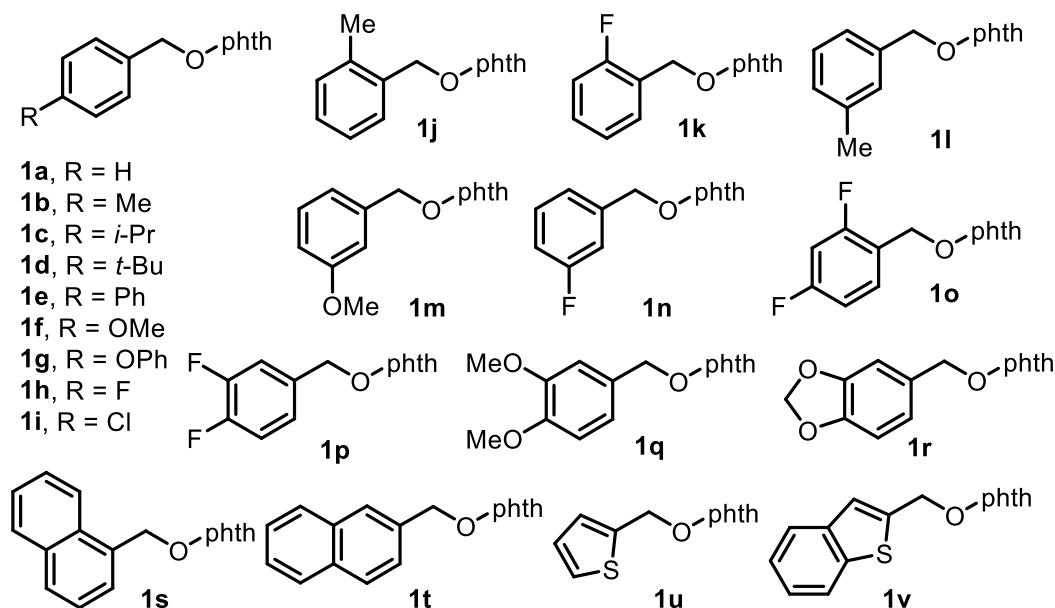
1. General considerations

Unless otherwise noted, all reagents were used as received from the commercial suppliers. Flash chromatography was performed using 200-300 mesh SiliaFlash 60[®] silica gel (Silicycle Inc.). Reactions were monitored using thin-layer chromatography (TLC). TLC plates were visualized with UV light (254 nm) or KMnO₄ stain. Flash chromatography was carried out silica gel (200-300 mesh). ¹H, ¹³C and ¹⁹F NMR spectra were recorded on a Bruker Avance III HD NMR 400 MHz instrument, and are internally referenced to the residual proto-solvent signals (note: CDCl₃ referenced at 7.26 ppm and 77.0 ppm, respectively). Data for ¹H are reported as: chemical shift (δ ppm), integration, multiplicity (s: singlet, d: doublet, t: triplet, m: multiplet, br: broad peak), coupling constant (Hz) and assignment. HRMS were recorded using ESI-TOF techniques. All measurements were carried out at room temperature unless otherwise stated.

2. Preparation of substrates

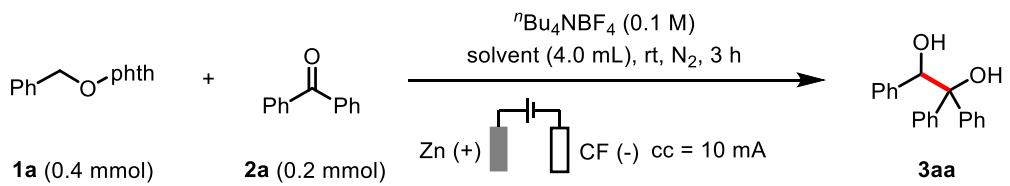
2.1 Preparation of *N*-(benzyloxy)phthalimides

N-(Benzyl)phthalimides were prepared according to the reported procedure.¹



3. Optimization of reaction conditions

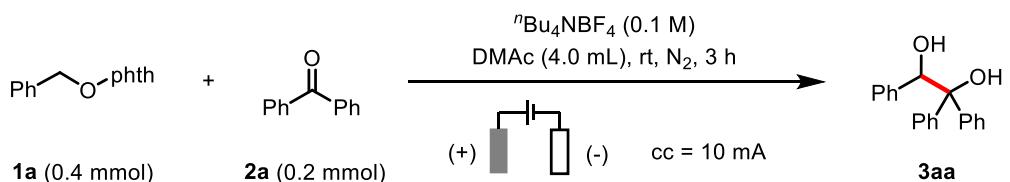
Table S1: Optimization of solvent for hydroxyalkylation



Entry	Solvent	Yield of 3aa (%) ^a
1	DMSO	28
2	DMAc	31
3	DMF	28
4	MeCN	16
5	Acetone	trace
6	MeOH	trace

^aDetermined by ¹H-NMR analysis of reaction crude mixture with dibromomethane as the internal standard.

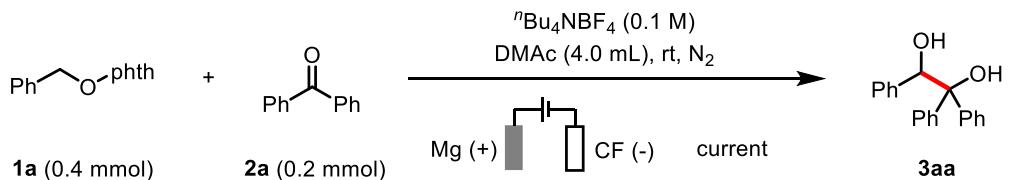
Table S2: Optimization of electrode for hydroxyalkylation



Entry	Anode (+)	Cathode (-)	Yield of 3aa (%) ^a
1	Zn	Pt	19
2	Zn	C plate	17
3	Zn	CF	31
4	Zn	Ni foam	23
5 ^b	Zn	CF	29
6	Mg	CF	48
7	Al	CF	trace
8	SS	CF	trace

^aDetermined by ¹H-NMR analysis of reaction crude mixture with dibromomethane as the internal standard. ^bReaction time: 4.5 h.

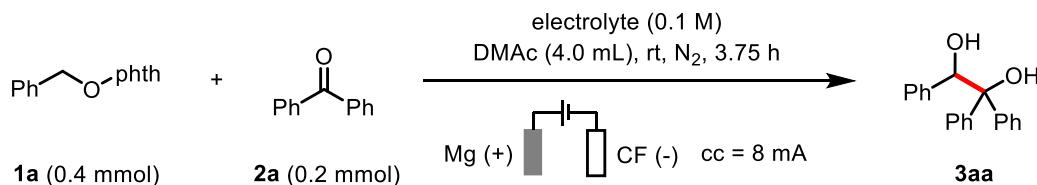
Table S3: Optimization of current for hydroxyalkylation



Entry	Current (mA)	Time (h)	Yield of 3aa (%) ^a
1	6	6	66
2	8	3.75	78
3	10	2.5	62
4	15	2	53
5	8	3	52
6	8	5	57

^aDetermined by ¹H-NMR analysis of reaction crude mixture with dibromomethane as the internal standard.

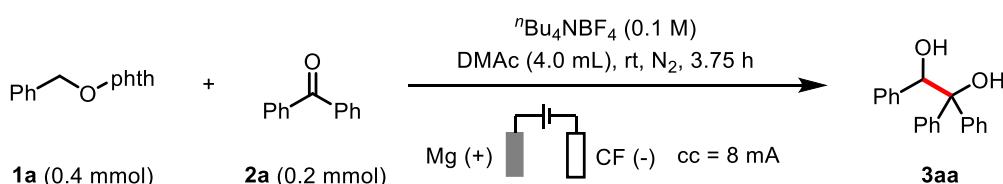
Table S4: Optimization of electrolyte for hydroxyalkylation



Entry	Electrolyte	Yield of 3aa (%) ^a
1	<i>n</i> Bu ₄ NPF ₆	trace
2	<i>n</i> Bu ₄ NI	47
3	<i>n</i> Bu ₄ NBr	30
4	<i>n</i> Bu ₄ NCl	37
5	<i>n</i> Bu ₄ NOAc	8
6	Et ₄ NCl	45
7	LiOTf	trace
8	LiCl	trace

^aDetermined by ¹H-NMR analysis of reaction crude mixture with dibromomethane as the internal standard.

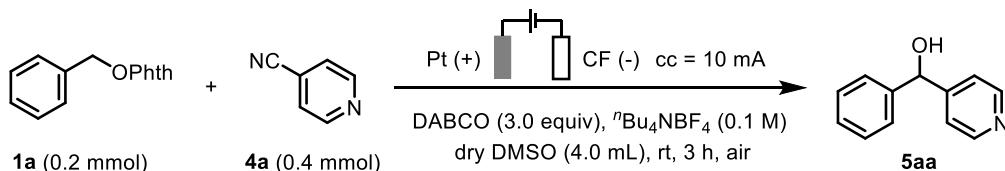
Table S5: Screening of different conditions for hydroxyalkylation



Entry	Variation	Yield of 3aa (%) ^a
1	3.0 mL, 5.0 mL	51, 57
2	1a (0.2 mmol), 2a (0.4 mmol)	61
3	1a (0.6 mmol), 2a (0.2 mmol)	68
4	anhydrous DMAc	58
5	air	trace
6	no electric current	n.d.

^aDetermined by ¹H-NMR analysis of reaction crude mixture with dibromomethane as the internal standard.

Table S6: Screening of different conditions for arylation

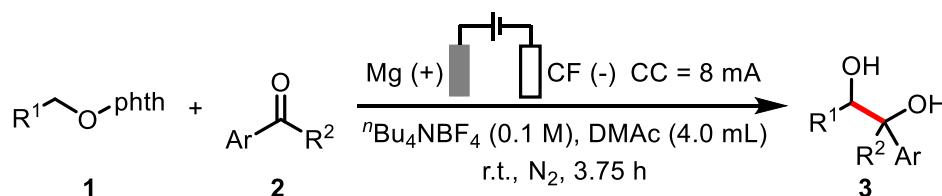


Entry	Variation	Yield of 5aa (%) ^a
1	none	76 ^b
2	<i>n</i> Bu ₄ NClO ₄ , <i>n</i> Bu ₄ NPF ₆ , <i>n</i> Bu ₄ NI instead of <i>n</i> Bu ₄ NBF ₄	21, 19, trace

3	CF instead of Pt (+)	trace
4	C plate, Pt, Ni foam instead of CF (-)	20, 27, 19
5	DMF, MeCN, Acetone, wet DMSO instead of dry DMSO	39, 20, 26, 56
6	ⁿ Bu ₄ NBF ₄ (0.2 M)	41
7	1a (0.4 mmol), 4a (0.2 mmol)	66
8	N ₂ instead of air	45

^aDetermined by ¹H-NMR analysis of reaction crude mixture with dibromomethane as the internal standard. ^bIsolated yield.

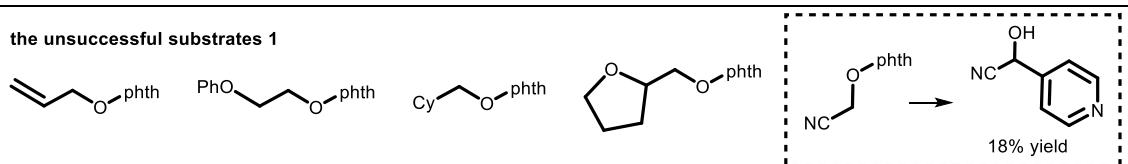
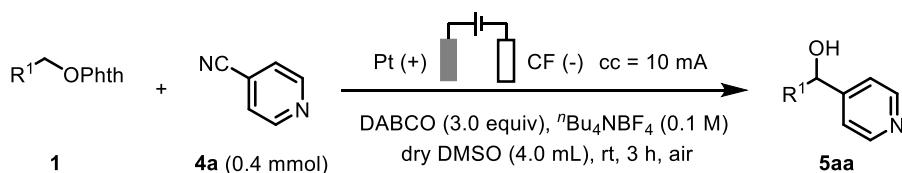
Table S7: Other substrates for hydroxyalkylation



Entry	1	2	3	
1				not observed
2				not observed
3				not observed
4				8% yield

^aReaction conditions: undivided cell, magnesium (Mg) anode, carbon felt (CF) cathode, constant current = 8 mA, **1** (2 equiv.), **2** (0.2 mmol), ⁿBu₄NBF₄ (2 equiv.), DMAc (4 mL), N₂, r.t., 3.75 h.

Table S8: Other substrates for arylation

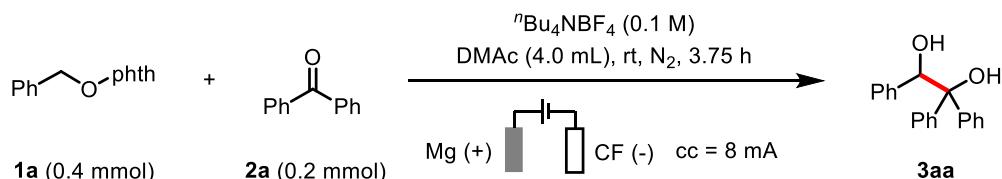


^aReaction conditions: undivided cell, platinum plate (Pt) anode, carbon felt (CF) cathode, constant current = 10 mA, **1** (0.2 mmol), **4a** (0.4 mmol), ⁿBu₄NBF₄ (2 equiv.), DABCO (3.0

equiv.), dry DMSO (4 mL), r.t., 3 h.

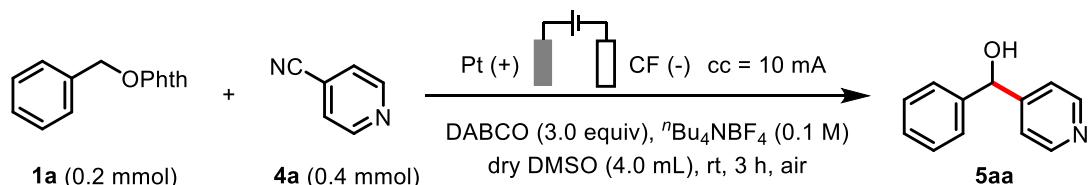
4. General procedure for electroreductive coupling.

Hydroxyalkylation:



A 10 mL tube equipped with a magnetic stirring bar was charged with *N*-(benzyloxy)phthalimide **1a** (0.4 mmol, 2.0 equiv.), benzophenone **2a** (0.2 mmol, 1.0 equiv.), $^n\text{Bu}_4\text{NBF}_4$ (0.4 mmol, 2.0 equiv.) and DMAc (4.0 mL) were added. The reactor was equipped with magnesium (Mg) electrode ($20\times 10\times 0.5$ mm) as the anode and carbon felt (CF) electrode ($20\times 10\times 4$ mm) as the cathode. After that, the mixture was electrolyzed under a constant current of 8 mA at room temperature for 3.75 h. After reaction was completed, the reaction solvent was diluted with 20 mL ethyl acetate, washed with H_2O for three times, dried over Na_2SO_4 and organic layers were combined and concentrated *in vacuo*. The resulting residue was purified by silica gel flash chromatography to give the products.

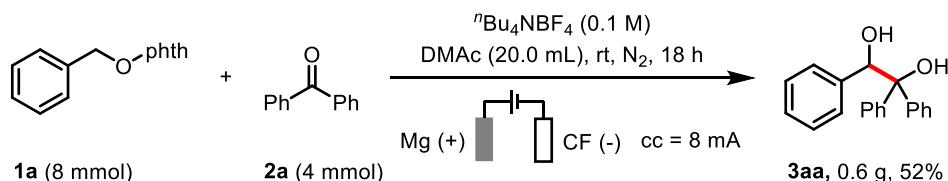
Arylation:



A dried 10 mL tube equipped with a magnetic stirring bar was charged with *N*-(benzyloxy)phthalimide **1a** (50.6 mg, 0.2 mmol, 1.0 equiv.), 4-cyanopyridine **4a** (41.6 mg, 0.4 mmol, 2.0 equiv.), DABCO (0.6 mmol, 3.0 equiv.), $^n\text{Bu}_4\text{NBF}_4$ (0.4 mmol, 2.0 equiv.) and anhydrous DMSO (4.0 mL) were added. The reactor was equipped with Platinum electrode ($20\times 10\times 0.2$ mm, from TCI) as the anode and carbon felt (CF) electrode ($20\times 10\times 4$ mm, from Cetech) as the cathode. Then, the mixture was electrolyzed under a constant current of 10 mA for 3 h at room temperature. After reaction was completed, the reaction solvent was diluted with 20 mL ethyl acetate, washed with H_2O for three times, dried over Na_2SO_4 and organic layers were combined and concentrated *in vacuo*. The resulting residue was purified by silica gel flash chromatography to give the products.

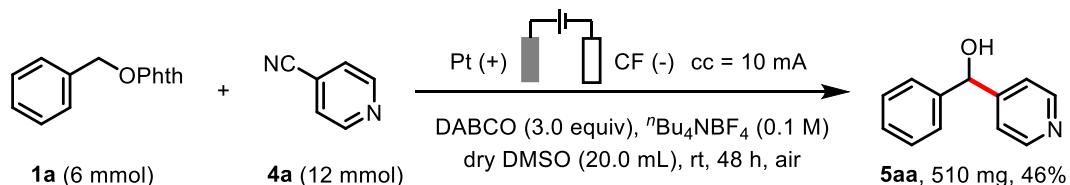
5. Gram-scale experiment

Hydroxyalkylation:



A 50 mL flask equipped with a magnetic stirring bar was charged with *N*-(benzyloxy)phthalimide **1a** (2.02 g, 8 mmol, 1.5 equiv.), benzophenone **2a** (0.728 g, 4 mmol, 1.0 equiv.), ⁿBu₄NBF₄ (2 mmol, 0.5 equiv.) and DMAc (20.0 mL) were added. The reactor was equipped with magnesium (Mg) electrode (20×20×0.5 mm) as the anode and carbon felt (CF) electrode (20×20×4 mm) as the cathode. After that, the mixture was electrolyzed under a constant current of 8 mA at room temperature for 18 h. After reaction was completed, the reaction solvent was diluted with 100 mL ethyl acetate, washed with H₂O for three times, dried over Na₂SO₄ and organic layers were combined and concentrated *in vacuo*. The resulting residue was purified by silica gel flash chromatography to give the products.

Arylation:

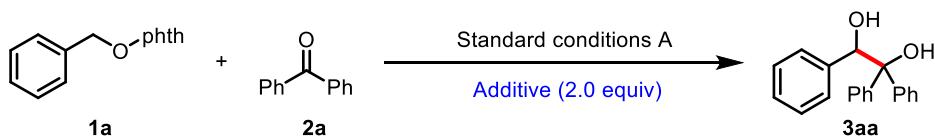


An oven-dried 50 mL flask equipped with a magnetic stirring bar was charged with *N*-(benzyloxy)phthalimide **1a** (1.52 g, 6 mmol, 1.0 equiv.), 4-cyanopyridine **4a** (1.21 g, 12 mmol, 2.0 equiv.), DABCO (6 mmol, 3.0 equiv.), ⁿBu₄NBF₄ (12 mmol, 2.0 equiv.) and anhydrous DMSO (20.0 mL) were added. The reactor was equipped with Platinum electrode (20×10×0.2 mm, from TCI) as the anode and carbon felt (CF) electrode (20×10×4 mm, from Cetech) as the cathode. The mixture was electrolyzed under a constant current of 10 mA for 48 h at room temperature. After reaction was completed, the reaction solvent was diluted with 100 mL ethyl acetate, washed with H₂O for three times, dried over Na₂SO₄ and organic layers were combined and concentrated *in vacuo*. The resulting residue was purified by silica gel flash chromatography to give the products.

6. Mechanistic experiments

6.1 Radical scavenger tests

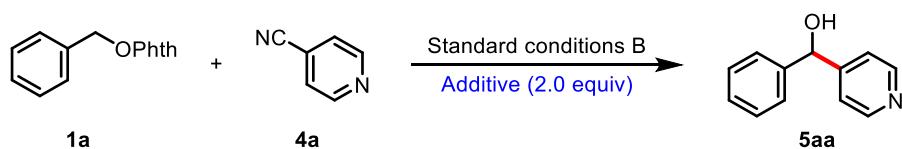
Table S9. Effect of radical scavengers on the hydroxyalkylation^a



Entry	Additive	Yield of 3aa ^b
1	TEMPO	22%
2	BHT	76%
3	1,1-Diphenylethylene	72%

^aReactions were carried out on 0.2 mmol scale with a 2:1 ratio of **1a**:**2a**. ^bConversion and yield determined by ¹H-NMR analysis of reaction crude mixture with dibromomethane as the internal standard.

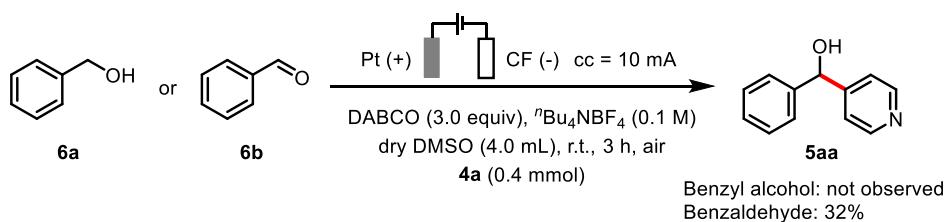
Table S10. Effect of radical scavengers on the arylation^a



Entry	Additive	Yield of 5aa ^b
1	TEMPO	trace
2	BHT	trace
3	1,1-Diphenylethylene	48%

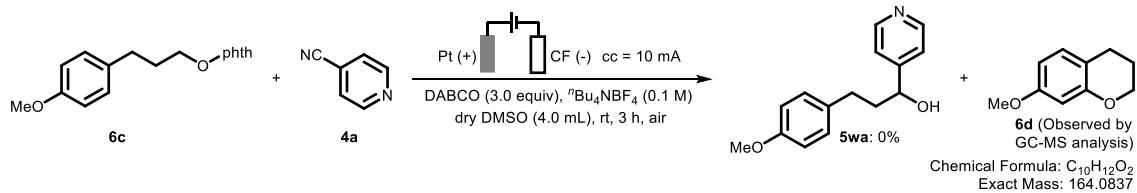
^aReactions were carried out on 0.2 mmol scale with a 1:2 ratio of **1a**:**4a**. ^bConversion and yield determined by ¹H-NMR analysis of reaction crude mixture with dibromomethane as the internal standard.

6.2 Excluding aldehyde/alcohol as the reaction intermediate

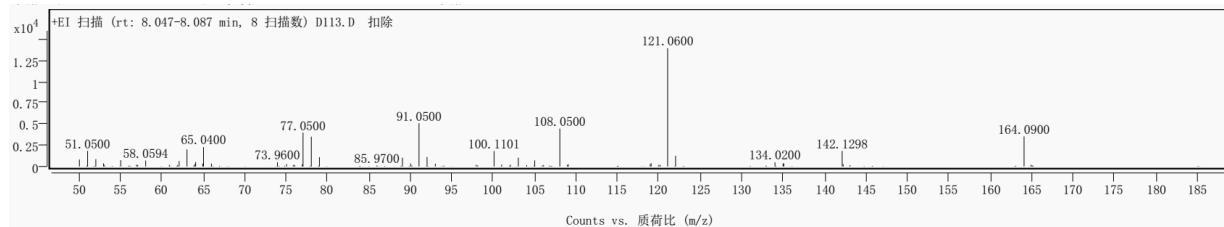


Following the standard procedure, the application of benzyl alcohol **6a** (21.6 mg, 0.2 mmol) instead of *N*-alkoxyphthalimide **1a** led to no arylation product **5aa**. The reaction of **6b** (21.2 mg, 0.2 mmol) and **4a** (75.6 mg, 0.4 mmol) afforded the product **5aa** in 32% yield.

6.3 The evidence of the presence of alkoxyl radicals



GC-MS dates of **6d**:

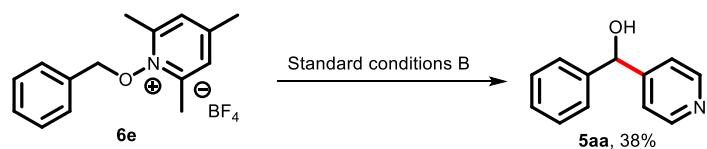


[MS Spectrum]

m/z, Absolute Intensity, Relative Intensity

50.0602	824	5.91	51.0500	1839	13.19	52.0400	867	6.21
52.9700	365	2.62	53.0400	201	1.44	55.0400	741	5.32
56.0500	104	0.75	56.9502	208	1.49	57.1000	178	1.28
58.0594	705	5.06	60.9100	189	1.36	61.9100	155	1.11
62.0900	631	4.52	63.0400	2016	14.46	63.9800	259	1.85
64.0900	532	3.82	64.9400	341	2.45	65.0400	2288	16.41
66.0000	349	2.50	73.9600	493	3.54	75.0600	279	2.00
75.8705	148	1.06	76.0000	207	1.48	76.9481	258	1.85
77.0500	3996	28.66	78.0500	3519	25.23	79.0300	1096	7.86
85.9700	127	0.91	89.0500	1034	7.41	90.0400	344	2.47
90.1900	114	0.82	91.0500	5104	36.61	92.0200	1116	8.00
93.0200	319	2.29	97.9498	193	1.38	98.1300	136	0.97
100.1101	1819	13.04	101.0292	223	1.60	102.0900	209	1.50
103.0400	1035	7.42	104.0500	150	1.08	105.0200	717	5.14
105.9100	110	0.79	106.0800	176	1.26	106.8200	101	0.73
108.0500	4461	32.00	108.9600	193	1.39	109.0800	247	1.77
118.9600	278	1.99	119.0700	387	2.77	120.0100	215	1.54
120.1900	155	1.11	121.0600	13944	100.00	122.0200	1265	9.07
134.0200	489	3.51	135.0000	363	2.60	135.0798	355	2.54
142.1298	1831	13.13	142.2496	274	1.97	143.0793	156	1.12
164.0900	3542	25.40	164.9100	186	1.34	165.0600	143	1.02

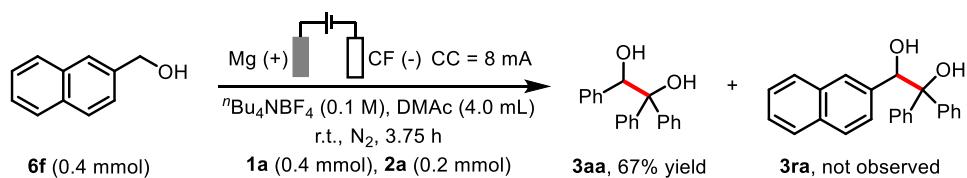
Scheme S1. The arylation with *N*-alkoxypyridinium salt



A dried 10 mL tube equipped with a magnetic stirring bar was charged with *N*-alkoxypyridinium salt derivative **6e** (0.2 mmol, 1.0 equiv.), 4-cyanopyridine **4a** (41.6 mg, 0.4 mmol, 2.0 equiv.), DABCO (0.6 mmol, 3.0 equiv.), $^n\text{Bu}_4\text{NBF}_4$ (0.4 mmol, 2.0 equiv.) and anhydrous DMSO (4.0 mL) were added. The reactor was equipped with Platinum electrode (20×10×0.2 mm, from TCI) as the anode and carbon felt (CF) electrode (20×10×4 mm, from Cetech) as the cathode. Then, the mixture was electrolyzed under a constant current of 10 mA for 3 h at room temperature. After reaction was completed, the reaction solvent was diluted with 20 mL ethyl acetate, washed with H_2O for three times, dried over Na_2SO_4 and organic layers were combined and concentrated *in vacuo*. The resulting residue was purified by silica gel flash chromatography to give the products.

6.4 The cross-over experiments

Hydroxyalkylation:



Following the standard procedure, the reaction of **6f** (26.7 mg, 0.4 mmol) for 3.75 h afforded the hydroxyalkylation product **3aa** in 67% yield and no occurrence of **3ra** from the crude $^1\text{H-NMR}$ spectra.

TLC (PE:EA = 5:1, twice)	1	2	3	4	Recovery of 6f
			3aa	reaction system	3ra 6f 82%

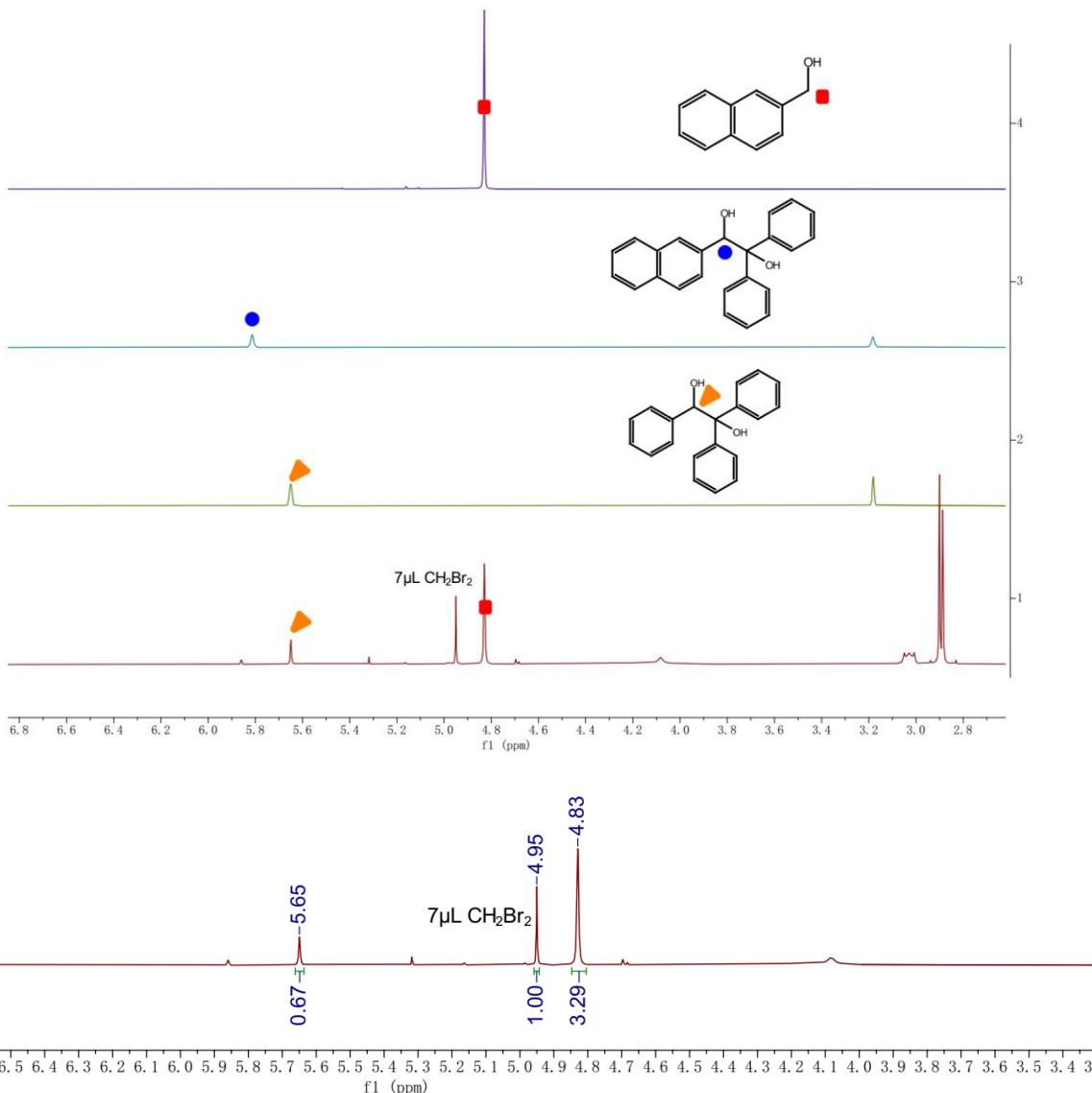
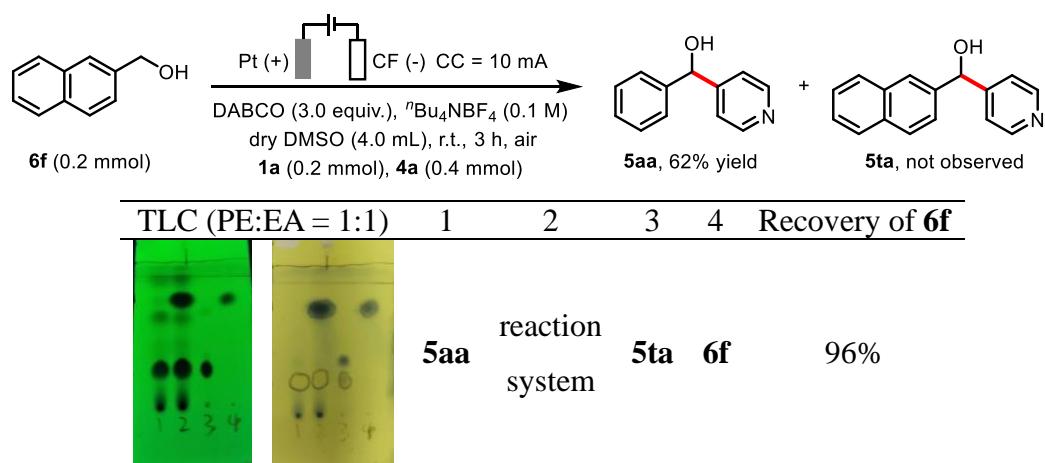


Figure S1. The ^1H -NMR spectra of the hydroxyalkylation.

Arylation:



Following the standard procedure, the reaction of 6f (13.4 mg, 0.2 mmol) for 3 h afforded

the arylation product **5aa** in 62% yield and no occurrence of **5ta** from the crude ¹H-NMR spectra.

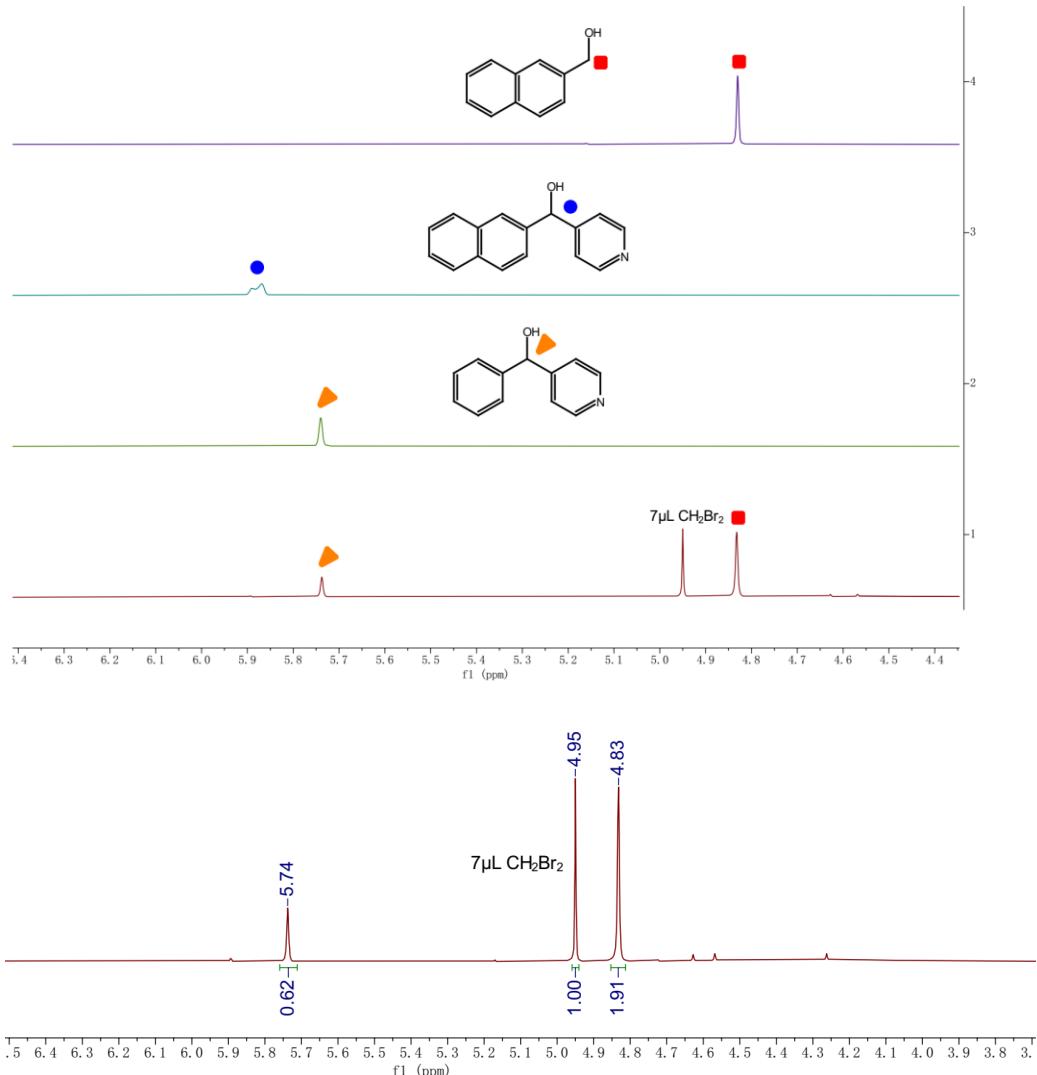


Figure S2. The ¹H-NMR spectra of the arylation.

6.5 EPR experiments and data²

General procedure for EPR studies:

A dried 10 mL tube equipped with a magnetic stirring bar, **1a** (101 mg, 0.4 mmol), "Bu₄NBF₄ (132 mg, 0.4 mmol), DMPO (45 μL, 0.4 mmol, 1.0 equiv) and dry MeCN (4 mL) (bubbled with nitrogen gas for 30 seconds to remove oxygen) were added. The reactor was equipped with magnesium (Mg) electrode (20×10×0.5 mm) as the anode and carbon felt (CF) electrode (20×10×4 mm) as the cathode. Under atmospheric nitrogen, the reaction mixture was stirred and electrolyzed at a constant current of 8 mA under room temperature for 20 minutes.

The solution sample was taken out into a small tube for EPR test.

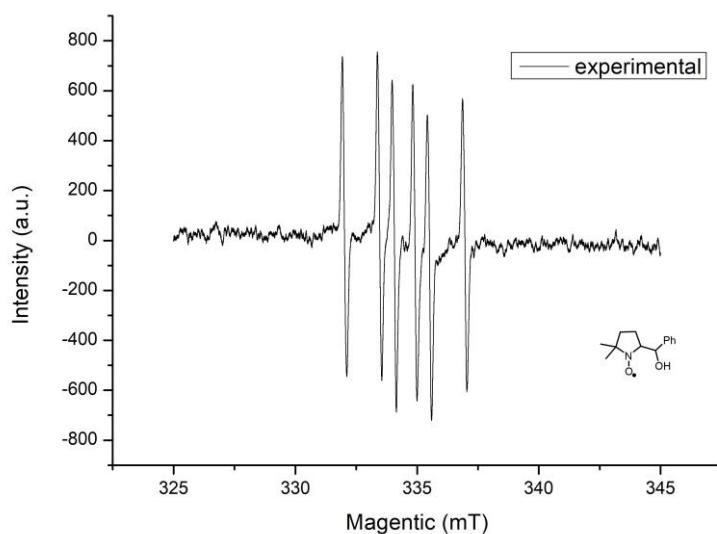


Figure S3. EPR measurements.

EPR spectra were measured on a JES-FA 200 spectrometer. EPR spectra was recorded at room temperature on EPR spectrometer operated at 9.191 GHz. Typical spectrometer parameters were shown as follows, sweep width: 100 G; center field set: 3350 G; time constant: 30 ms; sweep time: 30.00 s; modulation frequency: 100 kHz; microwave power: 1.00 mW. The adduct DMPO-CH **7b** was observed in the mixture of DMPO and *N*-(benzyloxy)phthalimide **1a**, showing a standard six-line spectrum ($g = 2.00621$, $A_N = 14.6$ G, $A_H = 20.5$ G).

General procedure for EPR studies:

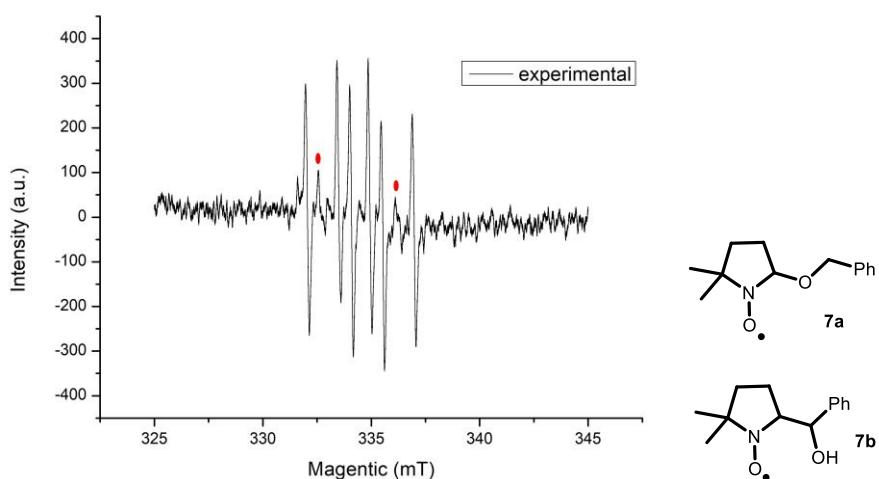


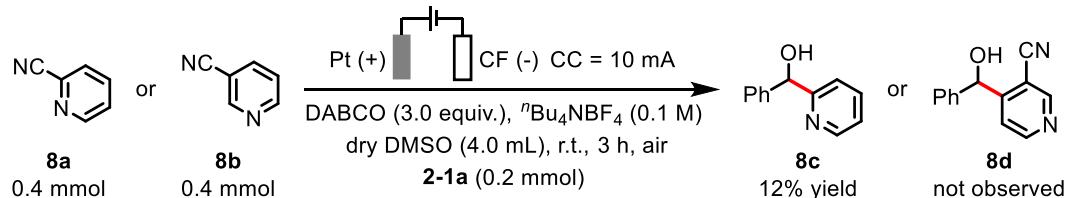
Figure S4. EPR measurements.

A dried 10 mL tube equipped with a magnetic stirring bar, **1a** (101 mg, 0.4 mmol),

$^n\text{Bu}_4\text{NBF}_4$ (132 mg, 0.4 mmol), DMPO (45 μL , 0.4 mmol, 1.0 equiv) and dry DMAc (4 mL) were added. The reactor was equipped with magnesium (Mg) electrode ($20\times 10\times 0.5$ mm) as the anode and carbon felt (CF) electrode ($20\times 10\times 4$ mm) as the cathode. Under atmospheric nitrogen, the reaction mixture was stirred and electrolyzed at a constant current of 8 mA under room temperature for 20 minutes. The solution sample was taken out into a small tube for EPR test.

EPR spectra were measured on a JES-FA 200 spectrometer. EPR spectra was recorded at room temperature on EPR spectrometer operated at 9.191 GHz. Typical spectrometer parameters were shown as follows, sweep width: 100 G; center field set: 3350 G; time constant: 30 ms; sweep time: 30.00 s; modulation frequency: 100 kHz; microwave power: 1.00 mW. The mixture of adduct DMPO-OCH **7a** and adduct DMPO-CH **7b**, $g = 2.00602$.

6.6 The arylation with 2- or 3-cyanopyridine



Following the standard procedure, the reaction of **8a** or **8b** for 3 h afforded the arylation product **8c** in 12% yield from the crude ^1H -NMR spectra and no occurrence of **8d** by TLC and GC-MS analysis.

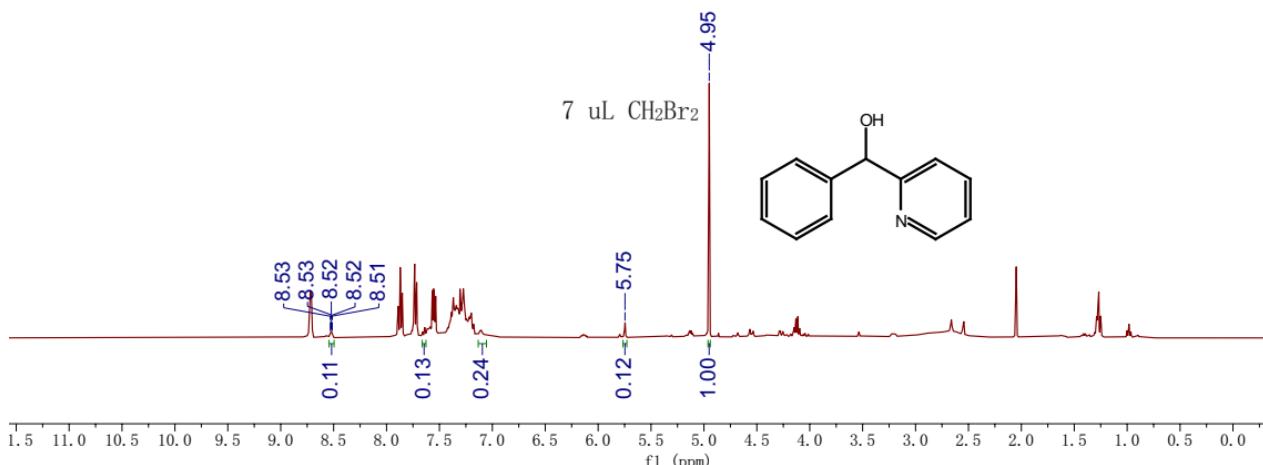


Figure S5. The ^1H -NMR spectra of the arylation product **8c**.

6.7 Cyclic voltammetry experiments

Cyclic voltammetry was recorded using a CHI660E potentiostat at room temperature. A glassy carbon electrode (3 mm diameter, Ledonlab), Pt wire (0.5 mm diameter, Ledonlab), and

Ag/AgCl (in saturated potassium chloride) were used as the working, counter, and reference electrodes, respectively. Prior to the experiment, glassy carbon electrode was polished using 0.2-1.0 μm $\alpha\text{-Al}_2\text{O}_3$ polishing powder. All experiments were sparged with argon for 5 minutes before data collection. Unless otherwise noted, scan rate = 100 mV/s.

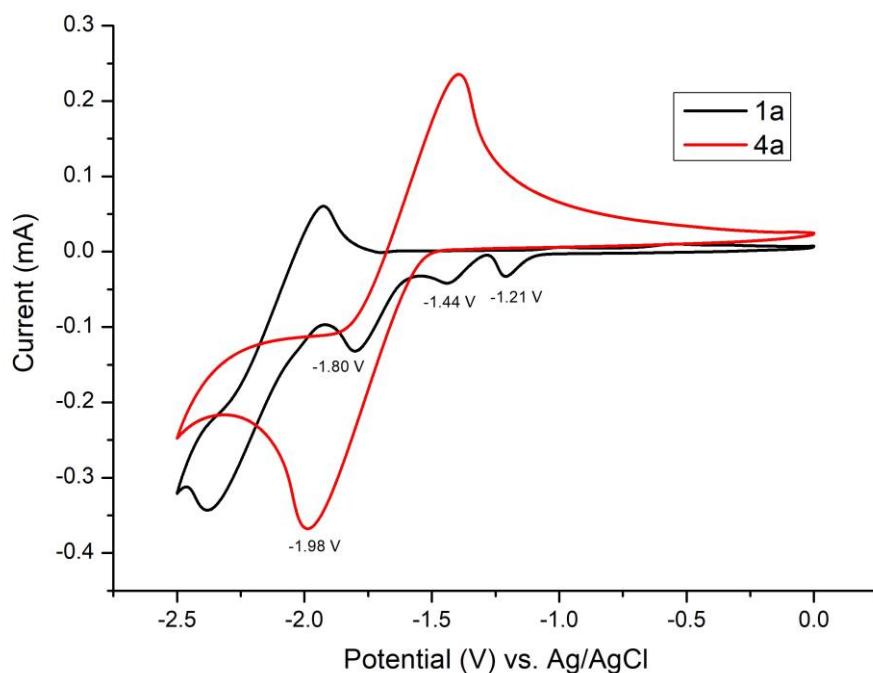
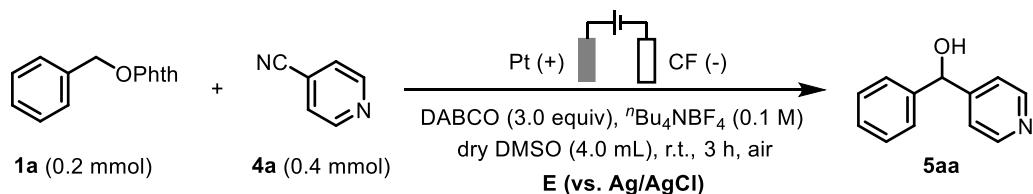


Figure S6. The cyclic voltammetry of *N*-alkoxyphthalimide **1a** (reduction peak at - 1.21 V, - 1.44 V, -1.80 V vs. Ag/AgCl) and 4-cyanpyridine **4a** (reduction peak at - 1.98 V vs. Ag/AgCl).

6.8 Controlled potential electrolysis

Table S11. Controlled potential electrolysis



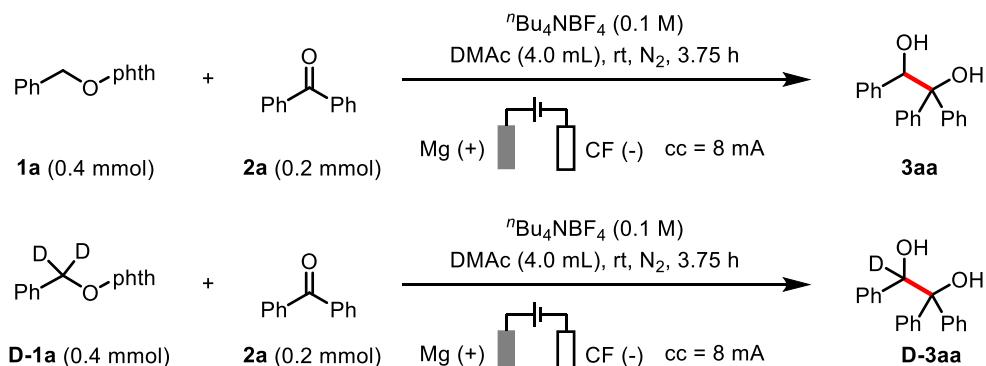
Entry	E (vs. Ag/AgCl)	Yield of 5aa (%) ^a
1	-1.8 V	trace
2	-1.88 V	trace
3	-2.2 V	48

^aDetermined by $^1\text{H-NMR}$ analysis of reaction crude mixture with dibromomethane as the internal standard.

Controlled potential electrolysis was proceeded using a CHI660E potentiostat. A dried 10 mL tube equipped with a magnetic stirring bar was charged with *N*-(benzyloxy)phthalimide **1a** (50.6 mg, 0.2 mmol, 1.0 equiv.), 4-cyanopyridine **4a** (41.6 mg, 0.4 mmol, 2.0 equiv.), DABCO (0.6 mmol, 3.0 equiv.), $^n\text{Bu}_4\text{NBF}_4$ (0.4 mmol, 2.0 equiv.) and anhydrous DMSO (4.0 mL) were added. A Platinum electrode (20×10×0.2 mm, from TCI), carbon felt (CF) electrode (20×10×4 mm, from Cetech) and Ag/AgCl (in saturated potassium chloride) were used as the counter, working, and reference electrodes, respectively. Then the mixture was respectively electrolyzed under a constant voltage (vs. Ag/AgCl) - 1.80 V and - 1.88 V. After reaction was completed, the reaction solvent was diluted with 20 mL ethyl acetate, washed with H_2O for three times, dried over Na_2SO_4 and organic layers were combined and concentrated *in vacuo*. The yield of the product **5aa** was determined by $^1\text{H-NMR}$ analysis of the reaction crude mixture with dibromomethane as the internal standard.

6.9 KIE experiments

Table S12. The KIE experiments of the hydroxyalkylation



Entry	Reaction Time (min)	Yield of 3aa (%) ^a	Yield of D-3aa (%) ^a
1	30	4	3
2	40	10	13
3	50	13	16
4	60	16	19

^aDetermined by $^1\text{H-NMR}$ analysis of reaction crude mixture with dibromomethane as the internal standard.

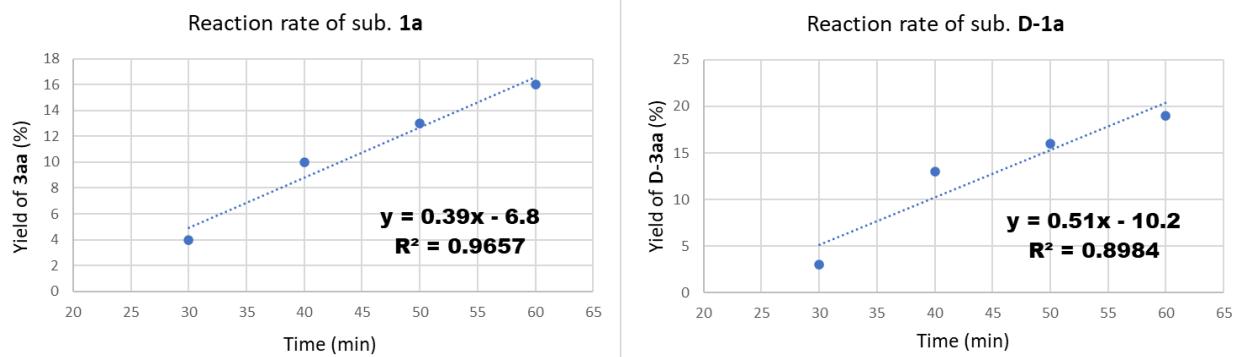
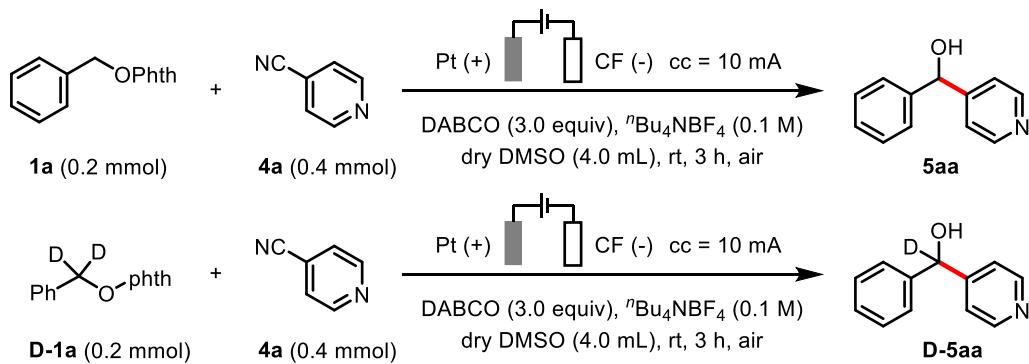


Figure S7. The deuterium labeling experiments of the hydroxyalkylation: The KIE value was calculated as $K_H/K_D = 0.39/0.51 = 0.76$, suggesting that the cleavage of the α -C(sp³)-H bond was not the rate-determining step.

Table S13. The KIE experiments of the arylation



Entry	Reaction Time (min)	Yield of 5aa (%) ^a	Yield of D-5aa (%) ^a
1	20	6	8
2	30	10	12
3	40	15	15
4	50	18	19

^aDetermined by ¹H-NMR analysis of reaction crude mixture with dibromomethane as the internal standard.

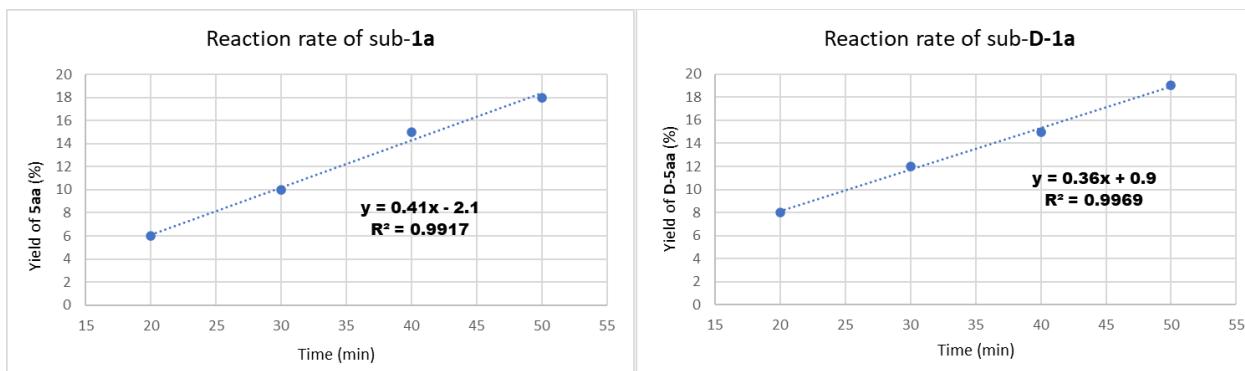
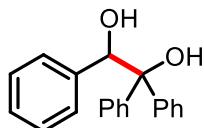


Figure S8. The deuterium labeling experiments of the arylation: The KIE value was calculated

as $K_H/K_D = 0.41/0.36 = 1.14$, suggesting that the cleavage of the α -C(sp³)-H bond was not the rate-determining step.

7. Characterization of products

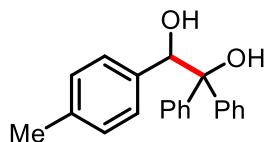


1,1,2-Triphenylethane-1,2-diol

Compound 3aa (45 mg, white solid, 78% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.³

¹H NMR (400 MHz, CDCl₃) δ 7.70 (d, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.2 Hz, 1H), 7.20 – 7.09 (m, 8H), 7.06 (d, *J* = 7.6 Hz, 2H), 5.63 (s, 1H), 3.17 (s, 1H), 2.47 (d, *J* = 3.2 Hz, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 145.1, 143.3, 138.8, 128.4, 128.1, 127.7, 127.6, 127.4, 127.3, 127.0, 126.7, 126.1, 80.7, 77.9.

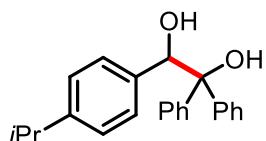


1,1-Diphenyl-2-(*p*-tolyl)ethane-1,2-diol

Compound 3ba (29 mg, white solid, 48% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.³

¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, *J* = 8.0 Hz, 2H), 7.40 (dd, *J* = 8.0 Hz, *J* = 1.2 Hz, 2H), 7.30 (dd, *J* = 6.8 Hz, *J* = 8.0 Hz, 1H), 7.18 – 7.07 (m, 5H), 6.95 (s, 4H), 5.61 (s, 1H), 3.14 (s, 1H), 2.40 (s, 1H), 2.27 (s, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 145.1, 143.5, 137.4, 135.7, 128.4, 128.2, 127.9, 127.6, 127.3, 126.9, 126.7, 126.2, 80.7, 77.8, 21.1.



2-(4-Isopropylphenyl)-1,1-diphenylethane-1,2-diol

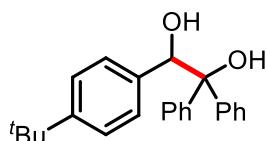
Compound 3ca (45 mg, white solid, 68% yield). Flash silica gel chromatography (petroleum

ether/ethyl acetate = 10/1).

¹H NMR (400 MHz, CDCl₃) δ 7.70 (d, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.2 Hz, 2H), 7.29 (t, *J* = 7.6 Hz, 1H), 7.17 – 7.07 (m, 5H), 7.00 (t, *J* = 9.2 Hz, 4H), 5.61 (s, 1H), 3.15 (s, 1H), 2.86 – 2.79 (m, 1H), 2.41 (s, 1H), 1.19 (d, *J* = 6.8 Hz, 6H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 148.4, 145.1, 143.5, 136.1, 128.4, 128.0, 127.6, 127.3, 127.0, 126.6, 126.2, 125.6, 80.7, 77.8, 33.7, 23.9.

ESI HRMS for [C₂₃H₂₄O₂+Na⁺] calculated: 355.1669, found: 355.1662.

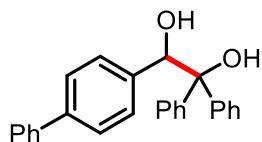


2-(4-(*tert*-Butyl)phenyl)-1,1-diphenylethane-1,2-diol

Compound 3da (45 mg, white solid, 65% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.³

¹H NMR (400 MHz, CDCl₃) δ 7.70 (d, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 1H), 7.17 – 7.10 (m, 7H), 7.00 (d, *J* = 8.0 Hz, 2H), 5.62 (s, 1H), 3.13 (s, 1H), 2.38 (s, 1H), 1.26 (s, 9H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 150.7, 145.1, 143.5, 135.7, 128.4, 127.7, 127.6, 127.3, 127.0, 126.7, 126.2, 124.4, 80.7, 77.8, 34.4, 31.2.



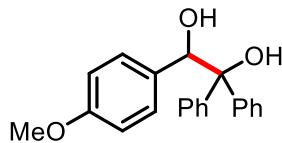
2-([1,1'-Biphenyl]-4-yl)-1,1-diphenylethane-1,2-diol

Compound 3ea (20 mg, white solid, 27% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 7/1).

¹H NMR (400 MHz, CDCl₃) δ 7.72 (d, *J* = 7.6 Hz, 2H), 7.55 (d, *J* = 7.6 Hz, 2H), 7.44 – 7.38 (m, 6H), 7.32 (t, *J* = 7.2 Hz, 2H), 7.20 – 7.10 (m, 7H), 5.69 (s, 1H), 3.17 (s, 1H), 2.46 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 145.0, 143.3, 140.6, 140.4, 137.8, 128.7, 128.49, 128.47, 127.7, 127.4, 127.3, 126.97, 126.96, 126.8, 126.2, 126.1, 80.8, 77.8.

ESI HRMS for [C₂₆H₂₂O₂+Na⁺] calculated: 389.1512, found: 389.1520.

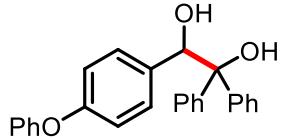


2-(4-Methoxyphenyl)-1,1-diphenylethane-1,2-diol

Compound 3fa (27 mg, white solid, 42% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.³

¹H NMR (400 MHz, DMSO-*d*₆) δ 7.58 (d, *J* = 8.0 Hz, 2H), 7.31 – 7.25 (m, 4H), 7.17 (t, *J* = 7.2 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 2H), 7.05 – 7.00 (m, 3H), 6.62 (d, *J* = 6.8 Hz, 2H), 5.52 (d, *J* = 4.8 Hz, 1H), 5.44 (d, *J* = 5.2 Hz, 2H), 3.64 (s, 3H).

¹³C{¹H} NMR (101 MHz, DMSO-*d*₆) δ 157.8, 146.9, 146.2, 134.1, 129.8, 127.4, 127.2, 126.5, 126.0, 125.8, 111.9, 79.8, 76.1, 54.8.

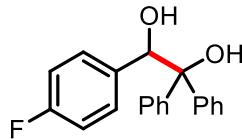


2-(4-Phenoxyphenyl)-1,1-diphenylethane-1,2-diol

Compound 3ga (54 mg, white solid, 71% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 7/1). Reported compound.⁴

¹H NMR (400 MHz, CDCl₃) δ 7.70 (d, *J* = 8.0 Hz, 2H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 3H), 7.15 – 7.08 (m, 6H), 7.02 (d, *J* = 8.0 Hz, 2H), 6.94 (d, *J* = 8.0 Hz, 2H), 6.79 (d, *J* = 8.0 Hz, 2H), 5.60 (s, 1H), 3.19 (br, 1H), 2.48 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 157.2, 156.5, 145.0, 143.3, 133.8, 129.6, 129.5, 128.5, 127.6, 127.4, 127.0, 126.7, 126.2, 123.1, 118.6, 118.0, 80.8, 77.5.



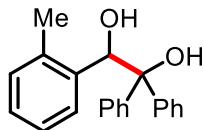
2-(4-Fluorophenyl)-1,1-diphenylethane-1,2-diol

Compound 3ha (46 mg, white solid, 75% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.³

¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.41 (t, *J* = 6.8 Hz, 2H), 7.32 (t, *J* = 7.2 Hz, 1H), 7.14 – 7.07 (m, 5H), 7.01 (dd, *J* = 8.4 Hz, *J* = 5.6 Hz, 2H), 6.82 (t, *J* = 8.4 Hz, 2H), 5.60 (s, 1H), 3.16 (s, 1H), 2.52 (br, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 162.21 (d, $J_{\text{C}-\text{F}} = 247.5$ Hz), 144.9, 143.1, 134.51 (d, $J_{\text{C}-\text{F}} = 3.0$ Hz), 129.65 (d, $J_{\text{C}-\text{F}} = 8.1$ Hz), 128.5, 127.7, 127.5, 126.9, 126.8, 126.1, 114.24 (d, $J_{\text{C}-\text{F}} = 22.2$ Hz), 80.7, 77.3.

^{19}F NMR (376 MHz, CDCl_3) δ -114.65.

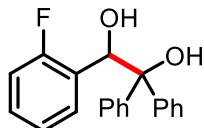


1,1-Diphenyl-2-(*o*-tolyl)ethane-1,2-diol

Compound 3ia (38 mg, white solid, 63% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.³

^1H NMR (400 MHz, CDCl_3) δ 7.72 (d, $J = 8.0$ Hz, 2H), 7.67 (d, $J = 7.6$ Hz, 1H), 7.45 (t, $J = 7.6$ Hz, 2H), 7.36 (t, $J = 7.2$ Hz, 1H), 7.21 (t, $J = 7.2$ Hz, 1H), 7.14 (t, $J = 7.2$ Hz, 1H), 7.11 – 7.02 (m, 3H), 6.97 – 6.95 (m, 2H), 6.89 (d, $J = 7.6$ Hz, 1H), 5.85 (s, 1H), 3.37 (s, 1H), 2.33 (s, 1H), 1.74 (s, 3H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 145.3, 142.8, 137.3, 136.7, 129.6, 128.4, 128.2, 127.7, 127.5, 127.4, 127.22, 127.17, 127.0, 125.5, 81.0, 72.6, 19.1.



2-(2-Fluorophenyl)-1,1-diphenylethane-1,2-diol

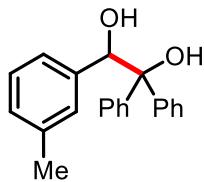
Compound 3ja (15 mg, white solid, 24% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1).

^1H NMR (400 MHz, CDCl_3) δ 7.69 (d, $J = 8.0$ Hz, 2H), 7.55 (t, $J = 7.6$ Hz, 1H), 7.41 (t, $J = 8.4$ Hz, 2H), 7.32 (t, $J = 8.0$ Hz, 1H), 7.20 – 7.05 (m, 7H), 6.74 (t, $J = 8.4$ Hz, 1H), 6.04 (s, 1H), 3.29 (s, 1H), 2.47 (br, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 159.97 (d, $J_{\text{C}-\text{F}} = 247.5$ Hz), 145.0, 142.7, 129.57 (d, $J_{\text{C}-\text{F}} = 4.0$ Hz), 129.33 (d, $J_{\text{C}-\text{F}} = 8.1$ Hz), 128.5, 127.5, 127.4, 127.0, 126.8, 126.40 (d, $J_{\text{C}-\text{F}} = 13.1$ Hz), 126.2, 123.56 (d, $J_{\text{C}-\text{F}} = 3.0$ Hz), 114.51 (d, $J_{\text{C}-\text{F}} = 23.2$ Hz), 80.8, 70.3.

^{19}F NMR (376 MHz, CDCl_3) δ -116.43.

ESI HRMS for $[\text{C}_{20}\text{H}_{17}\text{FO}_2+\text{Na}^+]$ calculated: 331.1105, found: 331.1097.



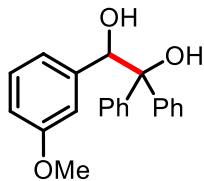
1,1-Diphenyl-2-(*m*-tolyl)ethane-1,2-diol

Compound 3ka (29 mg, white solid, 48% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1).

¹H NMR (400 MHz, CDCl₃) δ 7.70 (d, *J* = 8.0 Hz, 2H), 7.43 – 7.38 (m, 2H), 7.30 (dd, *J* = 8.0 Hz, *J* = 6.8 Hz, 1H), 7.16 – 7.09 (m, 5H), 7.04 – 6.98 (m, 2H), 6.86 (s, 1H), 6.83 (d, *J* = 7.2 Hz, 1H), 5.60 (s, 1H), 3.12 (s, 1H), 2.42 (s, 1H), 2.21 (s, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 145.0, 143.4, 138.6, 137.0, 128.8, 128.4, 127.6, 127.34, 127.31, 127.0, 126.7, 126.2, 125.1, 80.7, 78.0, 21.3.

ESI HRMS for [C₂₁H₂₀O₂+Na⁺] calculated: 327.1356, found: 327.1350.

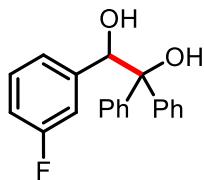


2-(3-Methoxyphenyl)-1,1-diphenylethane-1,2-diol

Compound 3la (45 mg, white solid, 70% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.³

¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.19 – 7.05 (m, 6H), 6.73 (dd, *J* = 2.4 Hz, *J* = 8.0 Hz, 1H), 6.68 (d, *J* = 8.0 Hz, 1H), 6.53 (s, 1H), 5.60 (s, 1H), 3.50 (s, 3H), 3.13 (s, 1H), 2.53 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 158.7, 144.9, 143.4, 140.3, 128.42, 128.40, 127.7, 127.3, 127.0, 126.7, 126.2, 120.3, 113.8, 113.2, 80.7, 77.9, 55.0.



2-(3-Fluorophenyl)-1,1-diphenylethane-1,2-diol

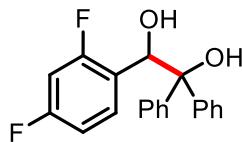
Compound 3ma (20 mg, white solid, 32% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1).

¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.2 Hz, 1H), 7.14 – 7.03 (m, 6H), 6.89 – 6.83 (m, 2H), 6.75 (d, *J* = 7.6 Hz, 1H), 5.62 (s, 1H), 3.11 (s, 1H), 2.52 (s, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 162.12 (d, *J_{C-F}* = 245.4 Hz), 144.8, 142.9, 141.40 (d, *J_{C-F}* = 7.1 Hz), 128.67 (d, *J_{C-F}* = 8.1 Hz), 128.6, 127.7, 127.5, 126.94, 126.89, 126.1, 123.78 (d, *J_{C-F}* = 3.0 Hz), 115.12 (d, *J_{C-F}* = 23.2 Hz), 114.50 (d, *J_{C-F}* = 21.2 Hz), 80.7, 77.42 (d, *J_{C-F}* = 2.0 Hz).

¹⁹F NMR (376 MHz, CDCl₃) δ -113.92.

ESI HRMS for [C₂₀H₁₇FO₂+Na⁺] calculated: 331.1105, found: 331.1097.



2-(2,4-Difluorophenyl)-1,1-diphenylethane-1,2-diol

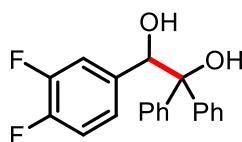
Compound 3na (14 mg, white solid, 21% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1).

¹H NMR (400 MHz, CDCl₃) δ 7.67 (d, *J* = 8.0 Hz, 2H), 7.55 (dd, *J* = 8.4 Hz, *J* = 15.2 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 1H), 7.13 – 7.09 (m, 5H), 6.83 – 6.78 (m, 1H), 6.51 – 6.45 (m, 1H), 5.98 (s, 1H), 3.26 (s, 1H), 2.50 (s, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 162.35 (d, *J_{C-F}* = 246.4 Hz), 159.93 (d, *J_{C-F}* = 231.3 Hz), 144.9, 142.5, 130.66 (dd, *J_{C-F}* = 9.1 Hz, *J_{C-F}* = 5.1 Hz), 128.6, 127.6, 127.5, 126.93, 126.90, 126.1, 122.51 (t, *J_{C-F}* = 13.1 Hz), 110.84 (dd, *J_{C-F}* = 21.2 Hz, *J_{C-F}* = 3.0 Hz), 102.67 (t, *J_{C-F}* = 25.3 Hz), 80.7, 69.8.

¹⁹F NMR (376 MHz, CDCl₃) δ -110.79 (d, *J_{F-F}* = 7.5 Hz, 1F), -112.49 (d, *J_{F-F}* = 7.5 Hz, 1F).

ESI HRMS for [C₂₀H₁₆F₂O₂+Na⁺] calculated: 349.1011, found: 349.1006.



2-(3,4-Difluorophenyl)-1,1-diphenylethane-1,2-diol

Compound 3oa (17 mg, white solid, 26% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1).

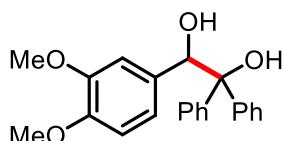
¹H NMR (400 MHz, CDCl₃) δ 7.66 (d, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.32 (d, *J* = 7.2 Hz, 1H), 7.17 – 7.09 (m, 5H), 6.99 – 6.93 (m, 1H), 6.90 – 6.84 (m, 1H), 6.68 – 6.64 (m, 1H),

5.57 (s, 1H), 3.09 (br, 1H), 2.54 (br, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 150.81 (d, $J_{\text{C}-\text{F}} = 30.3$ Hz), 148.44 (d, $J_{\text{C}-\text{F}} = 13.1$ Hz), 144.7, 142.8, 135.86 (t, $J_{\text{C}-\text{F}} = 4.0$ Hz), 128.6, 127.8, 127.6, 127.0, 126.8, 126.0, 124.11 (dd, $J_{\text{C}-\text{F}} = 6.1$ Hz, $J_{\text{C}-\text{F}} = 4.0$ Hz), 117.12 (d, $J_{\text{C}-\text{F}} = 18.2$ Hz), 115.88 (d, $J_{\text{C}-\text{F}} = 17.2$ Hz), 80.7, 76.93 (d, $J_{\text{C}-\text{F}} = 1.0$ Hz).

^{19}F NMR (376 MHz, CDCl_3) δ -138.64 (d, $J_{\text{F}-\text{F}} = 18.8$ Hz, 1F), -139.25 (d, $J_{\text{F}-\text{F}} = 22.6$ Hz, 1F).

ESI HRMS for $[\text{C}_{20}\text{H}_{16}\text{F}_2\text{O}_2+\text{Na}^+]$ calculated: 349.1011, found: 349.1006.



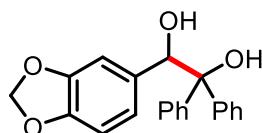
2-(3,4-Dimethoxyphenyl)-1,1-diphenylethane-1,2-diol

Compound 3pa (32 mg, white solid, 46% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1).

^1H NMR (400 MHz, CDCl_3) δ 7.68 (d, $J = 6.4$ Hz, 2H), 7.40 (t, $J = 8.0$ Hz, 2H), 7.30 (t, $J = 6.8$ Hz, 1H), 7.19 – 7.08 (m, 5H), 6.76 (d, $J = 8.4$ Hz, 1H), 6.69 (d, $J = 8.4$ Hz, 1H), 6.36 (s, 1H), 5.58 (s, 1H), 3.82 (s, 3H), 3.56 (s, 3H), 3.07 (s, 1H), 2.50 (br, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 148.4, 147.8, 144.8, 143.6, 131.1, 128.4, 127.7, 127.3, 127.0, 126.7, 126.3, 120.0, 111.3, 110.0, 80.7, 77.7, 55.7, 55.5.

ESI HRMS for $[\text{C}_{22}\text{H}_{22}\text{O}_4+\text{Na}^+]$ calculated: 373.1410, found: 373.1418.



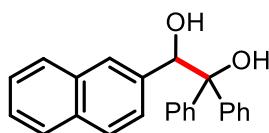
2-(Benzo[d][1,3]dioxol-5-yl)-1,1-diphenylethane-1,2-diol

Compound 3qa (20 mg, white solid, 30% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1).

^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 7.57 (d, $J = 7.6$ Hz, 2H), 7.28 (dd, $J = 8.0$ Hz, $J = 16.8$ Hz, 4H), 7.19 – 7.10 (m, 3H), 7.04 (t, $J = 7.2$ Hz, 1H), 6.76 (s, 1H), 6.56 (d, $J = 8.0$ Hz, 1H), 6.47 (d, $J = 8.4$ Hz, 1H), 5.88 (d, $J = 5.2$ Hz, 2H), 5.53 – 5.48 (m, 3H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, $\text{DMSO}-d_6$) δ 146.8, 146.1, 145.8, 145.5, 136.1, 127.4, 127.21, 127.19, 126.4, 126.0, 125.9, 122.1, 109.3, 106.3, 100.4, 79.8, 76.3.

ESI HRMS for $[\text{C}_{21}\text{H}_{18}\text{O}_4+\text{Na}^+]$ calculated: 357.1097, found: 357.1094.

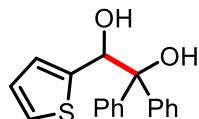


2-(Naphthalen-2-yl)-1,1-diphenylethane-1,2-diol

Compound 3ra (24 mg, white solid, 35% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 7/1). Reported compound.³

¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.69 (m, 4H), 7.62 (s, 1H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.42 (t, *J* = 7.2 Hz, 4H), 7.33 (t, *J* = 7.6 Hz, 1H), 7.18 – 7.06 (m, 6H), 5.81 (s, 1H), 3.18 (s, 1H), 2.53 (s, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 145.0, 143.3, 136.4, 132.9, 132.7, 128.5, 128.1, 127.7, 127.5, 127.4, 127.2, 127.0, 126.8, 126.2, 126.0, 125.9, 125.8, 80.9, 78.1.

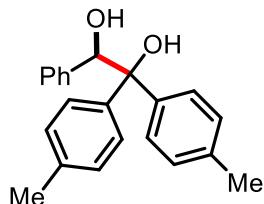


1,1-Diphenyl-2-(thiophen-2-yl)ethane-1,2-diol

Compound 3sa (29 mg, white solid, 49% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 7/1). Reported compound.³

¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, *J* = 8.0 Hz, 2H), 7.41 – 7.37 (m, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.31 – 7.27 (m, 1H), 7.21 – 7.17 (m, 3H), 7.14 – 7.10 (m, 1H), 6.82 – 6.80 (m, 1H), 6.72 (d, *J* = 3.6 Hz, 1H), 5.96 (s, 1H), 3.27 (s, 1H), 2.57 (s, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 144.6, 143.2, 141.7, 128.5, 127.9, 127.4, 126.9, 126.8, 126.5, 125.93, 125.87, 125.8, 80.4, 74.8.



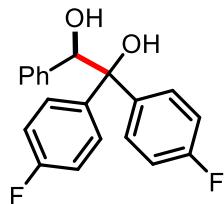
2-Phenyl-1,1-di-p-tolylethane-1,2-diol

Compound 3ab (30 mg, white solid, 47% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.⁴

¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, *J* = 7.6 Hz, 2H), 7.22 – 7.14 (m, 5H), 7.08 (d, *J* = 7.6

Hz, 2H), 7.02 (d, J = 8.0 Hz, 2H), 6.93 (d, J = 8.0 Hz, 2H), 5.57 (s, 1H), 3.09 (s, 1H), 2.53 (br, 1H), 2.38 (s, 3H), 2.24 (s, 3H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 142.2, 140.6, 138.9, 136.9, 136.1, 129.1, 128.3, 128.1, 127.5, 127.4, 126.8, 126.0, 80.6, 78.0, 21.0, 20.9.



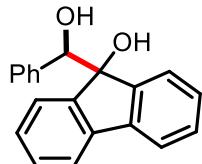
1,1-Bis(4-fluorophenyl)-2-phenylethane-1,2-diol

Compound 3ac (47 mg, white solid, 72% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.⁴

^1H NMR (400 MHz, CDCl_3) δ 7.60 (dd, J = 8.8 Hz, J = 5.6 Hz, 2H), 7.23 – 7.14 (m, 3H), 7.10 – 7.01 (m, 6H), 6.79 (t, J = 8.4 Hz, 2H), 5.50 (s, 1H), 3.26 (br, 1H), 2.40 (br, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 161.97 (d, $J_{\text{C}-\text{F}} = 247.5$ Hz), 161.56 (d, $J_{\text{C}-\text{F}} = 246.4$ Hz), 140.82 (d, $J_{\text{C}-\text{F}} = 3.0$ Hz), 139.11 (d, $J_{\text{C}-\text{F}} = 3.0$ Hz), 138.6, 128.96 (d, $J_{\text{C}-\text{F}} = 8.1$ Hz), 128.02 (d, $J_{\text{C}-\text{F}} = 9.1$ Hz), 128.0, 127.6, 115.14 (d, $J_{\text{C}-\text{F}} = 21.2$ Hz), 114.40 (d, $J_{\text{C}-\text{F}} = 21.2$ Hz), 80.0, 78.0.

^{19}F NMR (376 MHz, CDCl_3) δ -115.07, -115.81.

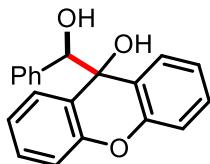


9-(Hydroxyphenyl)methyl-9H-fluoren-9-ol

Compound 3ad (20 mg, white solid, 35% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1). Reported compound.⁴

^1H NMR (400 MHz, CDCl_3) δ 7.69 (d, J = 7.2 Hz, 1H), 7.60 (d, J = 4.4 Hz, 1H), 7.43 – 7.39 (m, 2H), 7.34 – 7.28 (m, 4H), 7.02 (t, J = 7.6 Hz, 1H), 6.93 (t, J = 7.6 Hz, 2H), 6.78 (d, J = 7.6 Hz, 2H), 5.29 (s, 1H), 3.28 (br, 1H), 2.97 (br, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 145.8, 144.8, 140.20, 140.15, 137.5, 129.3, 129.1, 127.5, 127.31, 127.26, 126.9, 125.7, 124.3, 119.8, 119.6, 84.8, 79.5.



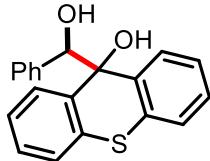
9-(Hydroxy(phenyl)methyl)-9*H*-xanthen-9-ol

Compound 3ae (41 mg, white solid, 67% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1). Reported compound.⁹

¹H NMR (400 MHz, DMSO-*d*₆) δ 7.84 (d, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.30 – 7.23 (m, 2H), 7.17 (d, *J* = 7.6 Hz, 1H), 7.09 – 7.04 (m, 2H), 6.96 – 6.88 (m, 4H), 6.48 (d, *J* = 7.6 Hz, 2H), 6.26 (s, 1H), 5.48 (s, 1H), 4.65 (s, 1H).

¹³C{¹H} NMR (101 MHz, DMSO-*d*₆) δ 150.6, 150.5, 140.3, 128.5, 128.4, 128.3, 127.9, 127.6, 126.8, 126.3, 125.6, 125.3, 122.1, 122.0, 114.8, 114.5, 81.4, 71.0.

ESI HRMS for [C₂₀H₁₆O₃+Na⁺] calculated: 327.0992, found: 327.0998.



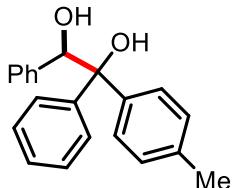
9-(Hydroxy(phenyl)methyl)-9*H*-thioxanthen-9-ol

Compound 3af (30 mg, white solid, 47% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1). Reported compound.⁹

¹H NMR (400 MHz, CDCl₃) δ 7.90 (d, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.42 – 7.30 (m, 3H), 7.15 (t, *J* = 7.6 Hz, 3H), 7.08 (t, *J* = 7.2 Hz, 2H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.82 (d, *J* = 7.6 Hz, 2H), 5.17 (s, 1H), 3.67 (s, 1H), 2.21 (s, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 137.8, 137.6, 136.2, 130.7, 130.4, 127.7, 127.6, 127.32, 127.28, 127.0, 126.8, 126.7, 126.5, 126.4, 126.0, 125.7, 77.8, 72.9.

ESI HRMS for [C₂₀H₁₆O₂S+Na⁺] calculated: 343.0763, found: 343.0756.



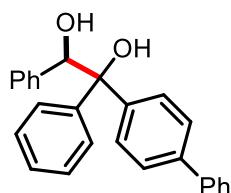
1,2-Diphenyl-1-(*p*-tolyl)ethane-1,2-diol

Compound 3ag (30 mg, white solid, 49% yield). Flash silica gel chromatography (petroleum

ether/ethyl acetate = 10/1). The diastereomeric ratio is 1.1:1 determined by ^1H NMR. Reported compound.³

^1H NMR (400 MHz, CDCl_3) δ 7.67 (d, J = 7.6 Hz, 1H), 7.58 (d, J = 8.0 Hz, 1H), 7.41 (t, J = 7.6 Hz, 1H), 7.33 – 7.30 (m, 0.5H), 7.22 – 7.02 (m, 9.5H), 6.94 (d, J = 8.0 Hz, 1H), 5.59 (s, 0.5H), 5.57 (s, 0.5H), 3.20 (s, 0.5H), 3.17 (s, 0.5H), 2.61 (br, 0.5H), 2.58 (br, 0.5H), 2.39 (s, 1.5H), 2.25 (s, 1.5H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 145.1, 143.5, 142.2, 140.5, 138.9, 138.8, 137.0, 136.2, 129.1, 128.33, 128.29, 128.11, 128.05, 127.6, 127.5, 127.38, 127.35, 127.2, 126.90, 126.85, 126.7, 126.09, 126.05, 80.64, 80.62, 77.9, 21.0, 20.9.

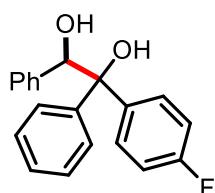


1-([1,1'-Biphenyl]-4-yl)-1,2-diphenylethane-1,2-diol

Compound 3ah (28 mg, white solid, 38% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1). The diastereomeric ratio is 1.08:1 determined by ^1H NMR. Reported compound.³

^1H NMR (400 MHz, CDCl_3) δ two isomers: 7.78 – 7.73 (m, 2H), 7.63 (t, J = 8.4 Hz, 2H), 7.51 (d, J = 7.6 Hz, 1H), 7.47 – 7.29 (m, 5H), 7.22 – 7.08 (m, 9H), 5.67 (s, 1H), 3.21 (br, 0.5H), 3.19 (br, 0.5H), 2.49 (br, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ two isomers: 145.0, 144.2, 143.3, 142.4, 140.6, 140.1, 139.4, 138.8, 138.7, 128.8, 128.7, 128.5, 128.10, 128.07, 127.8, 127.7, 127.5, 127.4, 127.3, 127.2, 127.12, 127.07, 126.96, 126.92, 126.8, 126.6, 126.3, 126.2, 80.71, 80.67, 78.04, 77.99.



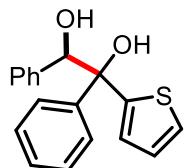
1-(4-Fluorophenyl)-1,2-diphenylethane-1,2-diol

Compound 3ai (30 mg, white solid, 49% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). The diastereomeric ratio is 1.04:1 determined by ^1H NMR. Reported compound.³

¹H NMR (400 MHz, CDCl₃) δ two isomers: 7.68 – 7.62 (m, 2H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.33 (t, *J* = 7.6 Hz, 0.5H), 7.23 – 7.02 (m, 10H), 6.78 (t, *J* = 8.4 Hz, 1H), 5.56 (s, 0.5H), 5.56 (s, 0.5H), 3.27 (br, 0.5H), 3.17 (br, 0.5H), 2.46 (br, 0.5H), 2.46 (br, 0.5H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ two isomers: 161.92 (d, *J*_{C–F} = 247.5 Hz), 161.51 (d, *J*_{C–F} = 246.4 Hz), 145.0, 143.3, 140.87 (d, *J*_{C–F} = 3.0 Hz), 139.1, 138.8, 138.6, 128.99 (d, *J*_{C–F} = 7.1 Hz), 128.5, 128.02 (d, *J*_{C–F} = 5.1 Hz), 127.8, 127.7, 127.5, 126.93, 126.85, 126.2, 115.04 (d, *J*_{C–F} = 21.2 Hz), 114.32 (d, *J*_{C–F} = 21.2 Hz), 80.40, 80.37, 78.0, 77.9.

¹⁹F NMR (376 MHz, CDCl₃) δ -115.40, -116.12.

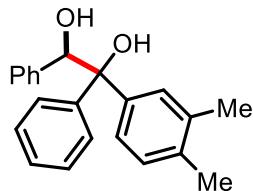


1,2-Diphenyl-1-(thiophen-2-yl)ethane-1,2-diol

Compound 3aj (40 mg, white solid, 68% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 7/1). The diastereomeric ratio is 1.1:1 determined by ¹H NMR. Reported compound.⁴

¹H NMR (400 MHz, CDCl₃) δ two isomers: 7.70 (d, *J* = 7.6 Hz, 1H), 7.41 (d, *J* = 7.6 Hz, 1H), 7.36 – 7.11 (m, 8.5H), 7.06 – 7.04 (m, 1.5H), 6.84 (t, *J* = 8.4 Hz, 0.5H), 6.74 (br, 0.5H), 5.49 (s, 0.5H), 5.39 (s, 0.5H), 3.54 (br, 0.5H), 3.23 (br, 0.5H), 2.68 (br, 0.5H), 2.47 (br, 0.5H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ two isomers: 150.5, 148.3, 143.5, 142.1, 138.3, 138.0, 128.4, 128.02, 127.95, 127.9, 127.81, 127.78, 127.6, 127.5, 127.2, 126.7, 126.4, 126.0, 125.6, 125.1, 124.9, 124.6, 80.2, 80.0, 79.8, 79.7.



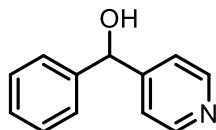
1-(3,4-Dimethylphenyl)-1,2-diphenylethane-1,2-diol

Compound 3ak (38 mg, white solid, 60% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 7/1). The diastereomeric ratio is 1.1:1 determined by ¹H NMR. Reported compound.⁴

¹H NMR (400 MHz, CDCl₃) δ two isomers: 7.68 (d, *J* = 7.6 Hz, 1H), 7.47 – 7.37 (m, 2H), 7.30 (t, *J* = 7.6 Hz, 0.6H), 7.20 – 7.05 (m, 7.5H), 6.91 – 6.86 (m, 1.5H), 5.61 (s, 0.5H), 5.60 (s, 0.5H),

3.11 (br, 0.5H), 3.03 (br, 0.5H), 2.45 (br, 1H), 2.29 (s, 1.5H), 2.27 (s, 1.5H), 2.14 (s, 1.5H), 2.09 (s, 1.5H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ two isomers: 145.0, 143.5, 142.5, 140.8, 138.9, 138.7, 136.8, 135.78, 135.75, 135.0, 129.7, 128.9, 128.3, 128.2, 128.14, 128.07, 127.60, 127.58, 127.55, 127.43, 127.40, 127.37, 127.2, 126.9, 126.6, 126.1, 124.0, 123.7, 80.7, 78.1, 78.0, 20.1, 19.8, 19.4, 19.3.

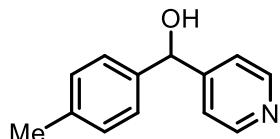


Phenyl(pyridin-4-yl)methanol

Compound 5aa (28.1 mg, white solid, 76% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 1/1). Reported compound.⁶

^1H NMR (400 MHz, CDCl_3) δ 8.38 (d, J = 5.2 Hz, 2H), 7.33 – 7.28 (m, 7H), 5.76 (s, 1H), 3.95 (br, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 153.2, 149.2, 142.8, 128.7, 128.1, 126.8, 121.4, 74.8.

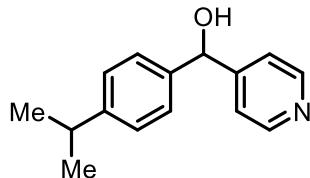


Pyridin-4-yl(*p*-tolyl)methanol

Compound 5ba (17.1 mg, white solid, 43% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1). Reported compound.⁶

^1H NMR (400 MHz, CDCl_3) δ 8.38 (d, J = 4.8 Hz, 2H), 7.29 (d, J = 5.2 Hz, 2H), 7.21 (d, J = 7.2 Hz, 2H), 7.14 (d, J = 7.6 Hz, 2H), 5.73 (s, 1H), 4.12 (br, 1H), 2.32 (s, 3H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 153.3, 149.3, 140.0, 137.9, 129.4, 126.7, 121.3, 74.6, 21.1.



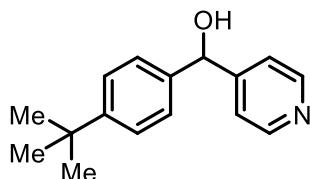
(4-Isopropylphenyl)(pyridin-4-yl)methanol

Compound 5ca (19.1 mg, white solid, 42% yield). Flash silica gel chromatography (petroleum

ether/ethyl acetate = 2/1). Reported compound.⁸

¹H NMR (400 MHz, CDCl₃) δ 8.49 (d, *J* = 5.2 Hz, 2H), 7.35 (d, *J* = 4.8 Hz, 2H), 7.27 (d, *J* = 9.2 Hz, 2H), 7.22 (d, *J* = 6.8 Hz, 2H), 5.79 (s, 1H), 2.95 – 2.88 (m, 1H), 2.03 (br, 1H), 1.25 (d, *J* = 8.3 Hz, 6H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 152.9, 149.4, 149.1, 140.1, 126.88, 126.85, 121.3, 74.8, 33.8, 23.9.

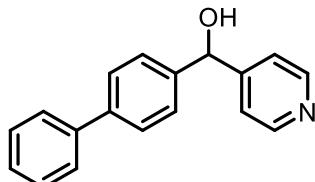


(4-(*tert*-Butyl)phenyl)(pyridin-4-yl)methanol

Compound 5da (27.8 mg, white solid, 58% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1). Reported compound.⁶

¹H NMR (CDCl₃, 400 MHz) δ 8.42 (d, *J* = 5.0 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 5.2 Hz, 2H), 7.28 – 7.26 (m, 2H), 5.77 (s, 1H), 3.54 (br, 1H), 1.32 (s, 9H).

¹³C{¹H} NMR (CDCl₃, 101 MHz) δ 153.2, 151.2, 149.3, 139.8, 126.6, 125.7, 121.3, 74.6, 34.5, 31.3.

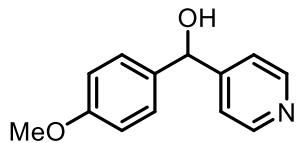


[1,1'-Biphenyl]-4-yl(pyridin-4-yl)methanol

Compound 5ea (40.7 mg, white solid, 78% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1). Reported compound.⁷

¹H NMR (400 MHz, DMSO-*d*₆) δ 8.50 (d, *J* = 4.8 Hz, 2H), 7.64 – 7.60 (m, 4H), 7.48 – 7.43 (m, 6H), 7.35 (t, *J* = 7.2 Hz, 1H), 6.20 (s, 1H), 5.78 (s, 1H).

¹³C{¹H} NMR (101 MHz, DMSO-*d*₆) δ 154.2, 149.4, 143.6, 139.9, 139.2, 128.9, 127.4, 127.0, 126.70, 126.65, 121.2, 72.8.

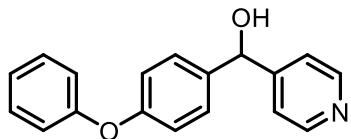


(4-Methoxyphenyl)(pyridin-4-yl)methanol

Compound 5fa (17.2 mg, white solid, 40% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1). Reported compound.⁶

¹H NMR (400 MHz, CDCl₃) δ 8.45 (d, *J* = 5.6 Hz, 2H), 7.30 (d, *J* = 4.8 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 6.86 (d, *J* = 7.6 Hz, 2H), 5.74 (s, 1H), 3.79 (s, 3H), 3.49 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 159.5, 153.0, 149.5, 135.0, 128.2, 121.2, 114.1, 74.4, 55.3.



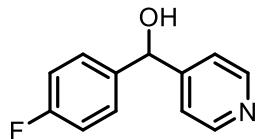
(4-Phenoxyphenyl)(pyridin-4-yl)methanol

Compound 5ga (35.5 mg, white solid, 64% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 1/1).

¹H NMR (400 MHz, CDCl₃) δ 8.51 (d, *J* = 5.2 Hz, 2H), 7.35 – 7.28 (m, 6H), 7.12 (t, *J* = 7.2 Hz, 1H), 7.01 – 6.96 (m, 4H), 5.79 (s, 1H), 3.04 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 157.4, 156.7, 152.7, 149.6, 137.4, 129.8, 128.3, 123.6, 121.2, 119.1, 118.8, 74.4.

ESI HRMS for [C₁₈H₁₅NO₂+H⁺] calculated: 278.1176, found: 278.1172.



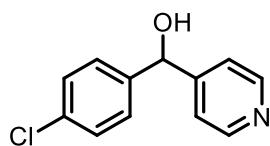
(4-Fluorophenyl)(pyridin-4-yl)methanol

Compound 5ha (30.9 mg, white solid, 76% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1). Reported compound.⁶

¹H NMR (400 MHz, CDCl₃) δ 8.46 – 8.41 (m, 2H), 7.32 – 7.28 (m, 4H), 7.05 – 7.00 (m, 2H), 5.78 (s, 1H), 3.58 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 162.46 (d, *J*_{C-F} = 247.5 Hz), 152.6, 149.6, 138.6, 128.54 (d, *J*_{C-F} = 8.1 Hz), 121.2, 115.68 (d, *J*_{C-F} = 22.2 Hz), 74.2.

¹⁹F NMR (376 MHz, CDCl₃) δ -113.64.

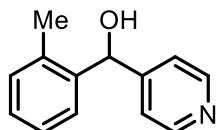


(4-Chlorophenyl)(pyridin-4-yl)methanol

Compound 5ia (28.0 mg, white solid, 64% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1). Reported compound.⁷

¹H NMR (400 MHz, CDCl₃) δ 8.44 (d, *J* = 5.2 Hz, 2H), 7.32 – 7.26 (m, 6H), 5.76 (s, 1H), 2.83 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 152.5, 149.5, 141.2, 134.0, 128.9, 128.1, 121.3, 74.1.

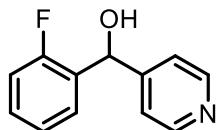


Pyridin-4-yl(o-tolyl)methanol

Compound 5ja (15.5 mg, white solid, 39% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1). Reported compound.⁶

¹H NMR (400 MHz, CDCl₃) δ 8.40 – 8.36 (m, 2H), 7.26 – 7.24 (m, 1H), 7.21 (d, *J* = 4.0 Hz, 2H), 7.17 – 7.15 (m, 2H), 7.13 – 7.10 (m, 1H), 5.93 (s, 1H), 3.17 (br, 1H), 2.25 (s, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 152.3, 149.4, 140.4, 135.7, 130.9, 128.2, 127.2, 126.4, 121.7, 72.1, 19.4.



(2-Fluorophenyl)(pyridin-4-yl)methanol

Compound 5ka (16.2 mg, white solid, 40% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1).

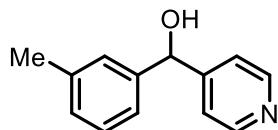
¹H NMR (400 MHz, CDCl₃) δ 8.43 (d, *J* = 4.4 Hz, 2H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 5.2 Hz, 2H), 7.31 – 7.28 (m, 1H), 7.15 (t, *J* = 7.6 Hz, 1H), 7.05 (t, *J* = 8.4 Hz, 1H), 6.14 (s, 1H), 4.30 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 159.76 (d, *J*_{C-F} = 247.5 Hz), 152.3, 149.4, 130.05 (d, *J*_{C-F} = 13.1 Hz), 129.67 (d, *J*_{C-F} = 8.1 Hz), 127.89 (d, *J*_{C-F} = 4.0 Hz), 124.61 (d, *J*_{C-F} = 4.0 Hz),

121.2, 115.52 (d, $J_{C-F} = 21.2$ Hz), 68.17 (d, $J_{C-F} = 4.0$ Hz).

$^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -118.56.

ESI HRMS for $[\text{C}_{12}\text{H}_{10}\text{FNO} + \text{H}^+]$ calculated: 204.0819, found: 204.0811.

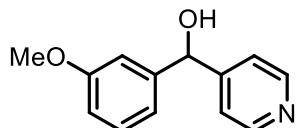


Pyridin-4-yl(*m*-tolyl)methanol

Compound 5la (11.9 mg, white solid, 30% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1). Reported compound.⁶

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.47 (dd, $J = 1.6$ Hz, $J = 4.4$ Hz, 2H), 7.32 (d, $J = 6.4$ Hz, 2H), 7.27 – 7.22 (m, 1H), 7.15 – 7.11 (m, 3H), 5.75 (s, 1H), 3.30 (br, 1H), 2.33 (s, 3H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 152.8, 149.6, 142.7, 138.6, 129.0, 128.7, 127.4, 123.9, 121.2, 75.0, 21.4.

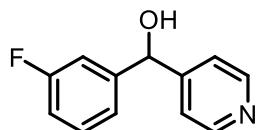


(3-Methoxyphenyl)(pyridin-4-yl)methanol

Compound 5ma (17.7 mg, white solid, 41% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1). Reported compound.⁶

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.44 – 8.40 (m, 2H), 7.30 (d, $J = 5.2$ Hz, 2H), 7.23 (d, $J = 8.4$ Hz, 1H), 6.91 – 6.87 (m, 2H), 6.83 – 6.80 (m, 1H), 5.73 (s, 1H), 3.75 (s, 3H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 159.9, 152.7, 149.5, 144.3, 129.8, 121.3, 119.0, 113.5, 112.4, 74.8, 55.2.



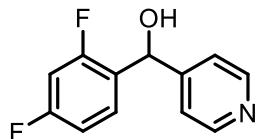
(3-Fluorophenyl)(pyridin-4-yl)methanol

Compound 5na (9.3 mg, white solid, 23% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 3/1). Reported compound.⁹

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.43 – 8.39 (m, 2H), 7.33 – 7.27 (m, 3H), 7.12 – 7.05 (m, 2H), 6.97 (t, $J = 8.4$ Hz, 1H), 5.76 (s, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 162.95 (d, $J_{\text{C}-\text{F}} = 247.5$ Hz), 152.5, 149.5, 145.3, 130.30 (d, $J_{\text{C}-\text{F}} = 8.1$ Hz), 122.28 (d, $J_{\text{C}-\text{F}} = 3.0$ Hz), 121.3, 115.05 (d, $J_{\text{C}-\text{F}} = 21.2$ Hz), 113.62 (d, $J_{\text{C}-\text{F}} = 22.2$ Hz), 74.2.

^{19}F NMR (376 MHz, CDCl_3) δ -112.05.



(2,4-Difluorophenyl)(pyridin-4-yl)methanol

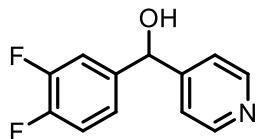
Compound 5oa (19.4 mg, white solid, 44% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 1/1).

^1H NMR (400 MHz, CDCl_3) δ 8.37 (d, $J = 5.6$ Hz, 2H), 7.44 – 7.38 (m, 1H), 7.32 (d, $J = 5.2$ Hz, 2H), 6.88 – 6.84 (m, 1H), 6.80 – 6.75 (m, 1H), 6.07 (s, 1H), 5.03 (s, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 162.49 (dd, $J_{\text{C}-\text{F}} = 12.1$ Hz, $J_{\text{C}-\text{F}} = 250.5$ Hz), 159.74 (dd, $J_{\text{C}-\text{F}} = 12.1$ Hz, $J_{\text{C}-\text{F}} = 249.5$ Hz), 152.5, 149.3, 128.93 (dd, $J_{\text{C}-\text{F}} = 4.0$ Hz, $J_{\text{C}-\text{F}} = 10.1$ Hz), 126.27 (dd, $J_{\text{C}-\text{F}} = 4.0$ Hz, $J_{\text{C}-\text{F}} = 14.1$ Hz), 121.2, 111.79 (dd, $J_{\text{C}-\text{F}} = 4.0$ Hz, $J_{\text{C}-\text{F}} = 21.2$ Hz), 103.86 (t, $J_{\text{C}-\text{F}} = 25.3$ Hz), 67.54 (d, $J_{\text{C}-\text{F}} = 3.0$ Hz).

^{19}F NMR (376 MHz, CDCl_3) δ -110.17 (d, $J_{\text{F}-\text{F}} = 7.5$ Hz, 1F), -114.58 (d, $J_{\text{F}-\text{F}} = 7.5$ Hz, 1F).

ESI HRMS for $[\text{C}_{12}\text{H}_9\text{F}_2\text{NO}+\text{H}^+]$ calculated: 222.0725, found: 222.0715.



(3,4-Difluorophenyl)(pyridin-4-yl)methanol

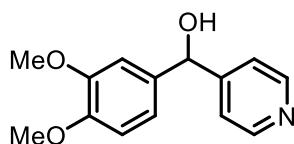
Compound 5pa (23.0 mg, white solid, 52% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 2/1).

^1H NMR (400 MHz, CDCl_3) δ 8.43 (d, $J = 4.4$ Hz, 2H), 7.29 (d, $J = 4.8$ Hz, 2H), 7.20 – 7.04 (m, 3H), 5.74 (s, 1H), 4.22 (br, 1H).

$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 152.3, 151.44 (dd, $J_{\text{C}-\text{F}} = 4.8$ Hz, $J_{\text{C}-\text{F}} = 45.5$ Hz), 149.6, 148.96 (dd, $J_{\text{C}-\text{F}} = 5.2$ Hz, $J_{\text{C}-\text{F}} = 46.5$ Hz), 139.8 (t, $J_{\text{C}-\text{F}} = 5.1$ Hz), 122.67 (dd, $J_{\text{C}-\text{F}} = 4.0$ Hz, $J_{\text{C}-\text{F}} = 7.1$ Hz), 121.2, 117.48 (d, $J_{\text{C}-\text{F}} = 17.2$ Hz), 115.73 (d, $J_{\text{C}-\text{F}} = 18.2$ Hz), 73.7.

^{19}F NMR (376 MHz, CDCl_3) δ -136.48 (d, $J_{\text{F}-\text{F}} = 18.8$ Hz, 1F), -138.31 (d, $J_{\text{F}-\text{F}} = 22.6$ Hz, 1F).

ESI HRMS for $[\text{C}_{12}\text{H}_9\text{F}_2\text{NO}+\text{H}^+]$ calculated: 222.0725, found: 222.0715.

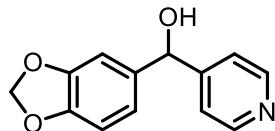


(3,4-Dimethoxyphenyl)(pyridin-4-yl)methanol

Compound 5qa (22.5 mg, white solid, 46% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 1/1). Reported compound.⁸

¹H NMR (400 MHz, CDCl₃) δ 8.45 (d, *J* = 4.8 Hz, 2H), 7.30 (d, *J* = 5.2 Hz, 2H), 6.87 – 6.80 (m, 3H), 5.73 (s, 1H), 3.86 (s, 3H), 3.82 (s, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 152.9, 149.5, 149.3, 148.9, 135.4, 121.2, 119.3, 111.0, 109.7, 74.6, 55.9, 55.8.



Benzo[d][1,3]dioxol-5-yl(pyridin-4-yl)methanol

Compound 5ra (14.2 mg, white solid, 31% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 1/1). Reported compound.¹⁰

¹H NMR (400 MHz, CDCl₃) δ 8.52 (d, *J* = 4.8 Hz, 2H), 7.31 (d, *J* = 4.8 Hz, 2H), 6.84 – 6.76 (m, 3H), 5.95 (s, 2H), 5.71 (s, 1H), 1.73 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 152.4, 149.7, 148.1, 147.6, 136.7, 121.1, 120.5, 108.3, 107.2, 101.2, 74.8.



Naphthalen-1-yl(pyridin-4-yl)methanol

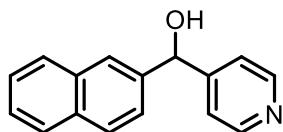
Compound 5sa (16.9 mg, white solid, 36% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 1/1).

¹H NMR (400 MHz, CDCl₃) δ 8.42 (d, *J* = 5.2 Hz, 2H), 8.04 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.83 (d, *J* = 7.6 Hz, 1H), 7.49 – 7.42 (m, 4H), 7.32 (d, *J* = 5.2 Hz, 2H), 6.43 (s, 1H), 3.78 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 152.4, 149.5, 137.8, 134.1, 130.6, 129.2, 128.9, 126.4,

125.9, 125.7, 125.3, 123.9, 121.6, 72.7.

ESI HRMS for [C₁₆H₁₃NO+H⁺] calculated: 236.1070, found: 236.1062.

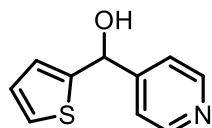


Naphthalen-2-yl(pyridin-4-yl)methanol

Compound 5ta (23.0 mg, white solid, 49% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 1/1). Reported compound.¹¹

¹H NMR (400 MHz, CDCl₃) δ 8.37 – 8.30 (m, 2H), 7.81 – 7.76 (m, 4H), 7.48 – 7.46 (m, 2H), 7.36 (d, *J* = 8.4 Hz, 1H), 7.32 – 7.29 (m, 2H), 5.87 (s, 1H), 4.76 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 153.3, 149.1, 140.2, 133.1, 133.0, 128.7, 128.0, 127.7, 126.4, 126.2, 125.6, 124.5, 121.5, 74.7.

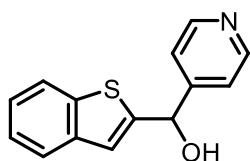


Pyridin-4-yl(thiophen-2-yl)methanol

Compound 5ua (24.8 mg, white solid, 65% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 1/1). Reported compound.¹²

¹H NMR (400 MHz, CDCl₃) δ 8.54 (s, 2H), 7.41 (d, *J* = 4.8 Hz, 2H), 7.29 (d, *J* = 4.8 Hz, 1H), 6.97 – 6.93 (m, 2H), 6.05 (s, 1H), 2.97 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 152.3, 149.4, 146.5, 126.8, 126.2, 125.5, 121.3, 70.7.



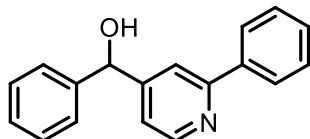
Benzo[b]thiophen-2-yl(pyridin-4-yl)methanol

Compound 5va (28.0 mg, white solid, 58% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 1/1).

¹H NMR (400 MHz, CDCl₃) δ 8.54 (d, *J* = 7.6 Hz, 2H), 7.78 (d, *J* = 7.6 Hz, 1H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.42 (d, *J* = 5.2 Hz, 2H), 7.36 – 7.29 (m, 2H), 7.17 (s, 1H), 6.10 (s, 1H), 3.76 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 151.4, 149.8, 147.0, 139.9, 139.1, 124.7, 124.5, 123.8, 122.5, 121.9, 121.2, 71.3.

ESI HRMS for [C₁₄H₁₁NOS+H⁺] calculated: 242.0634, found: 242.0628.

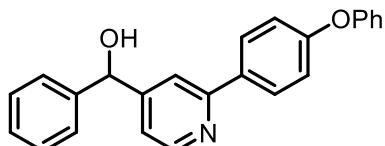


Phenyl(2-phenylpyridin-4-yl)methanol

Compound 5ab (43.3 mg, white solid, 83% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1). Reported compound.⁷

¹H NMR (400 MHz, CDCl₃) δ 8.48 (d, *J* = 5.2 Hz, 1H), 7.88 (d, *J* = 6.8 Hz, 2H), 7.71 (s, 1H), 7.44 – 7.39 (m, 3H), 7.32 – 7.27 (m, 5H), 7.16 (d, *J* = 5.2 Hz, 1H), 5.73 (s, 1H), 4.08 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 157.5, 153.7, 149.3, 142.7, 139.1, 128.9, 128.7, 128.6, 128.1, 127.0, 126.8, 119.9, 118.3, 74.9.



(2-(4-Phenoxyphenyl)pyridin-4-yl)(phenyl)methanol

Compound 5ac (47.3 mg, white solid, 67% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1).

¹H NMR (400 MHz, CDCl₃) δ 8.49 (d, *J* = 5.2 Hz, 1H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.70 (s, 1H), 7.38 – 7.29 (m, 7H), 7.17 – 7.12 (m, 2H), 7.05 (d, *J* = 8.0 Hz, 4H), 5.77 (s, 1H), 3.63 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 158.3, 157.0, 156.7, 153.5, 149.4, 142.7, 134.2, 129.8, 128.8, 128.6, 128.2, 126.8, 123.6, 119.5, 119.2, 118.7, 117.7, 75.0.

ESI HRMS for [C₂₄H₁₉NO₂+H⁺] calculated: 354.1489, found: 354.1479.

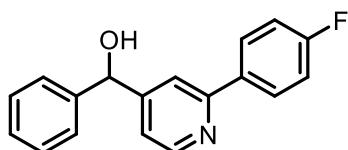


(2-(4-Methoxyphenyl)pyridin-4-yl)(phenyl)methanol

Compound 5ad (47.1 mg, white solid, 81% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1). Reported compound.⁷

¹H NMR (400 MHz, CDCl₃) δ 8.45 (d, *J* = 5.2 Hz, 1H), 7.85 (d, *J* = 8.8 Hz, 2H), 7.67 (s, 1H), 7.33 – 7.27 (m, 5H), 7.11 (d, *J* = 5.2 Hz, 1H), 6.93 (d, *J* = 9.2 Hz, 2H), 5.74 (s, 1H), 3.82 (s, 3H), 3.64 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 160.4, 157.2, 153.4, 149.3, 142.7, 131.8, 128.7, 128.3, 128.1, 126.8, 119.2, 117.4, 114.0, 75.0, 55.3.



(2-(4-Fluorophenyl)pyridin-4-yl)(phenyl)methanol

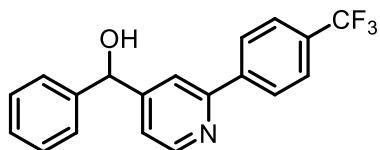
Compound 5ae (41.3 mg, white solid, 74% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 5/1).

¹H NMR (400 MHz, CDCl₃) δ 8.49 (d, *J* = 4.8 Hz, 1H), 7.90 – 7.86 (m, 2H), 7.69 (s, 1H), 7.34 – 7.28 (m, 5H), 7.17 (d, *J* = 5.2 Hz, 1H), 7.10 (t, *J* = 8.4 Hz, 2H), 5.77 (s, 1H), 3.58 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 163.47 (d, *J*_{C-F} = 249.5 Hz), 156.6, 153.6, 149.4, 142.6, 135.3, 128.84 (d, *J*_{C-F} = 8.1 Hz), 128.3, 126.8, 119.8, 117.8, 115.56 (d, *J*_{C-F} = 22.2 Hz), 75.0.

¹⁹F NMR (376 MHz, CDCl₃) δ -112.88.

ESI HRMS for [C₁₈H₁₄FNO+H⁺] calculated: 280.1132, found: 280.1122.



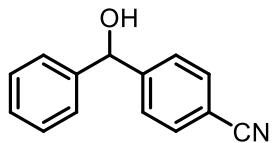
Phenyl(2-(4-(trifluoromethyl)phenyl)pyridin-4-yl)methanol

Compound 5af (25.0 mg, white solid, 38% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 7/1). Reported compound.⁷

¹H NMR (400 MHz, CDCl₃) δ 8.60 (d, *J* = 4.8 Hz, 1H), 8.05 (d, *J* = 8.0 Hz, 2H), 7.80 (s, 1H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.37 – 7.27 (m, 6H), 5.84 (s, 1H), 2.92 (br, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 156.1, 153.6, 149.8, 142.5, 130.80 (d, *J*_{C-F} = 32.3 Hz), 128.9, 128.4, 127.3, 126.8, 125.61 (dd, *J*_{C-F} = 7.1 Hz, *J*_{C-F} = 3.0 Hz), 122.8, 120.6, 118.4, 75.1.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.56.

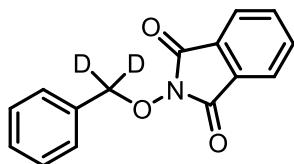


4-(Hydroxy(phenyl)methyl)benzonitrile

Compound 5ag (8.4 mg, oil, 20% yield). Flash silica gel chromatography (petroleum ether/ethyl acetate = 20/1). Reported compound.⁶

¹H NMR (400 MHz, CDCl₃) δ 7.61 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 7.38 – 7.30 (m, 5H), 5.86 (s, 1H), 2.49 (s, 1H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 148.8, 142.8, 132.2, 128.8, 128.3, 127.0, 126.6, 118.8, 111.1, 75.6.



2-(Phenylmethoxy-*d*2)isoindoline-1,3-dione

Compound D-1 (white solid). Flash silica gel chromatography (petroleum ether/ethyl acetate = 10/1). Reported compound.¹³

¹H NMR (400 MHz, CDCl₃) δ 7.81 – 7.79 (m, 2H), 7.74 – 7.71 (m, 2H), 7.55 – 7.52 (m, 2H), 7.38 – 7.36 (m, 3H).

¹³C{¹H} NMR (101 MHz, CDCl₃) δ 163.5, 134.4, 133.5, 129.9, 129.3, 128.8, 128.5, 123.5.

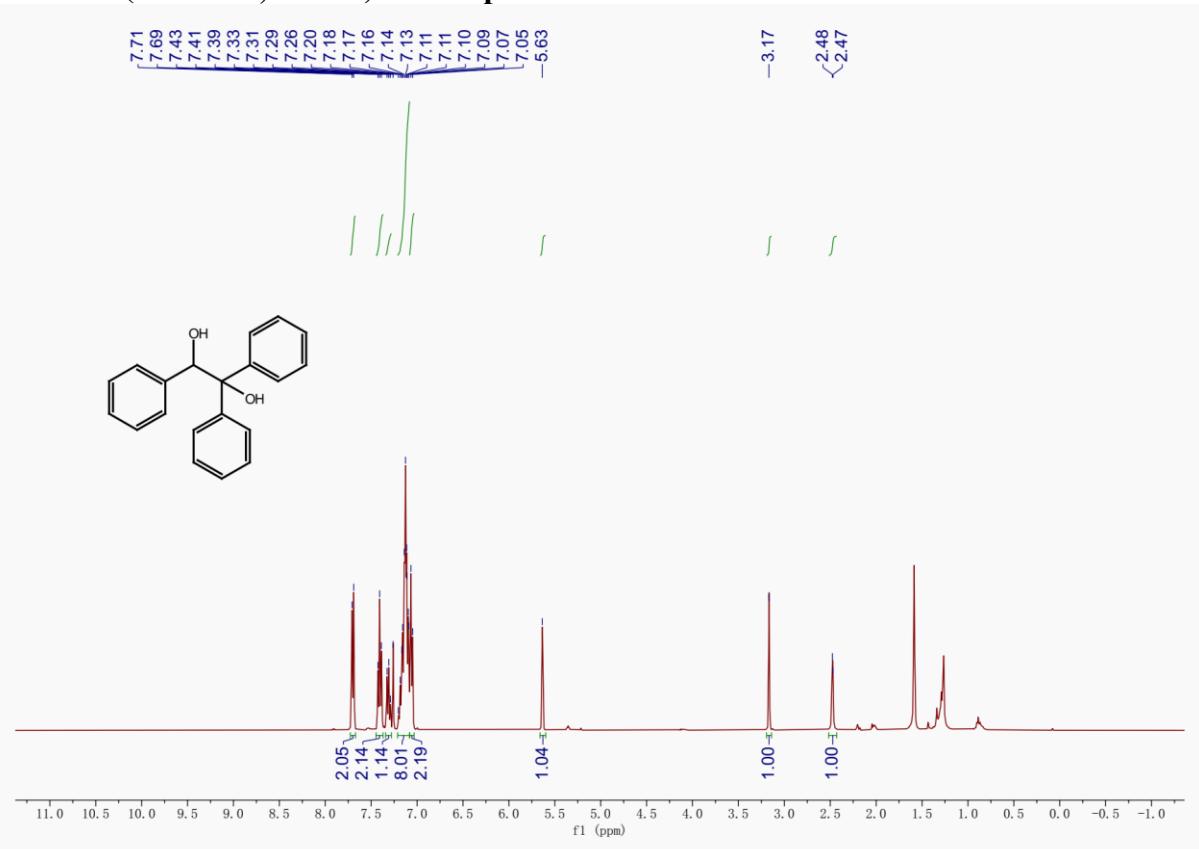
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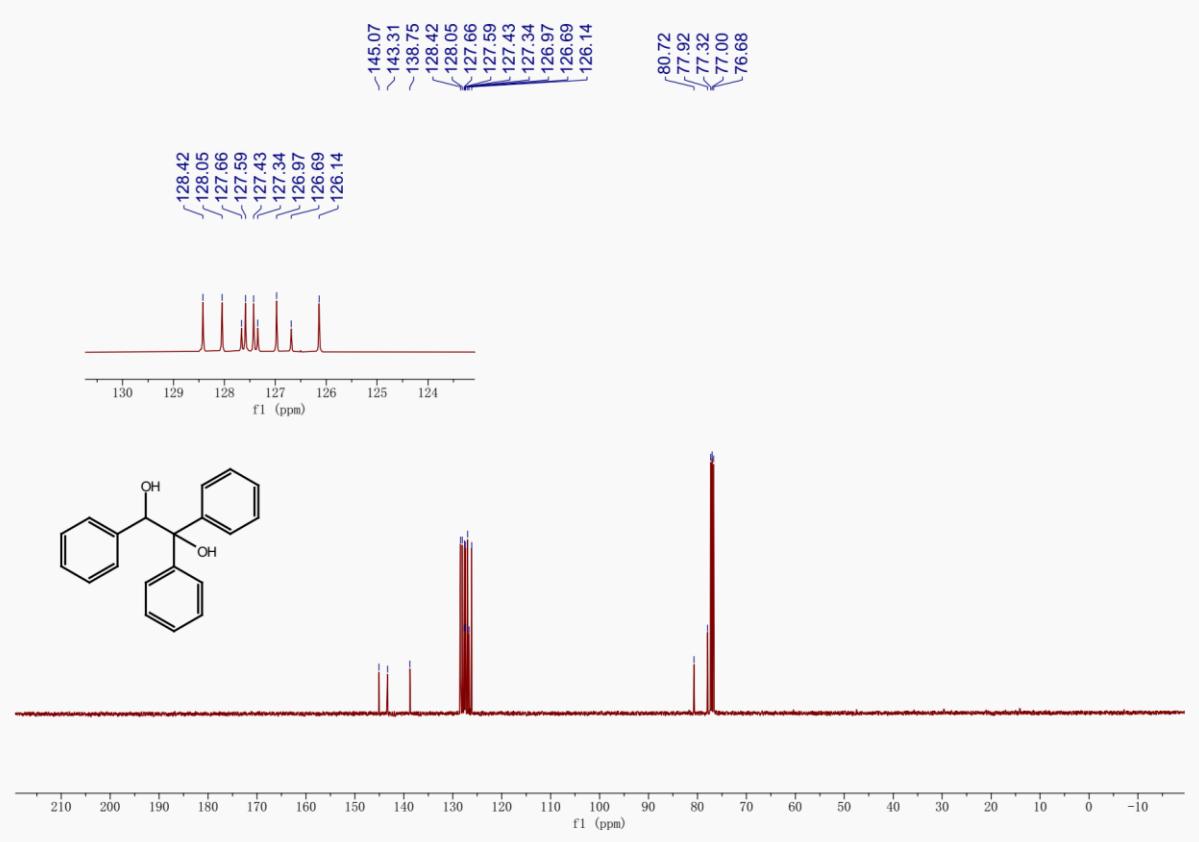
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9. NMR spectra of products

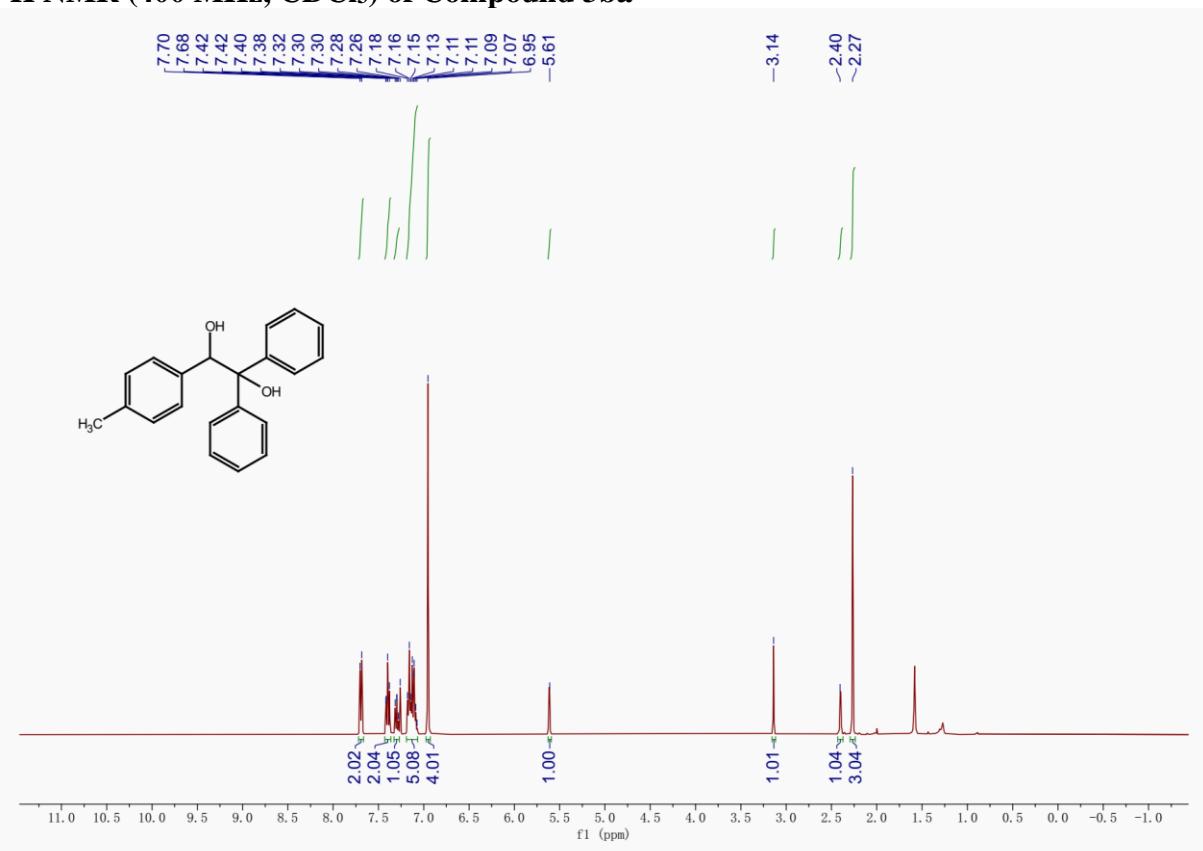
^1H NMR (400 MHz, CDCl_3) of Compound 3aa



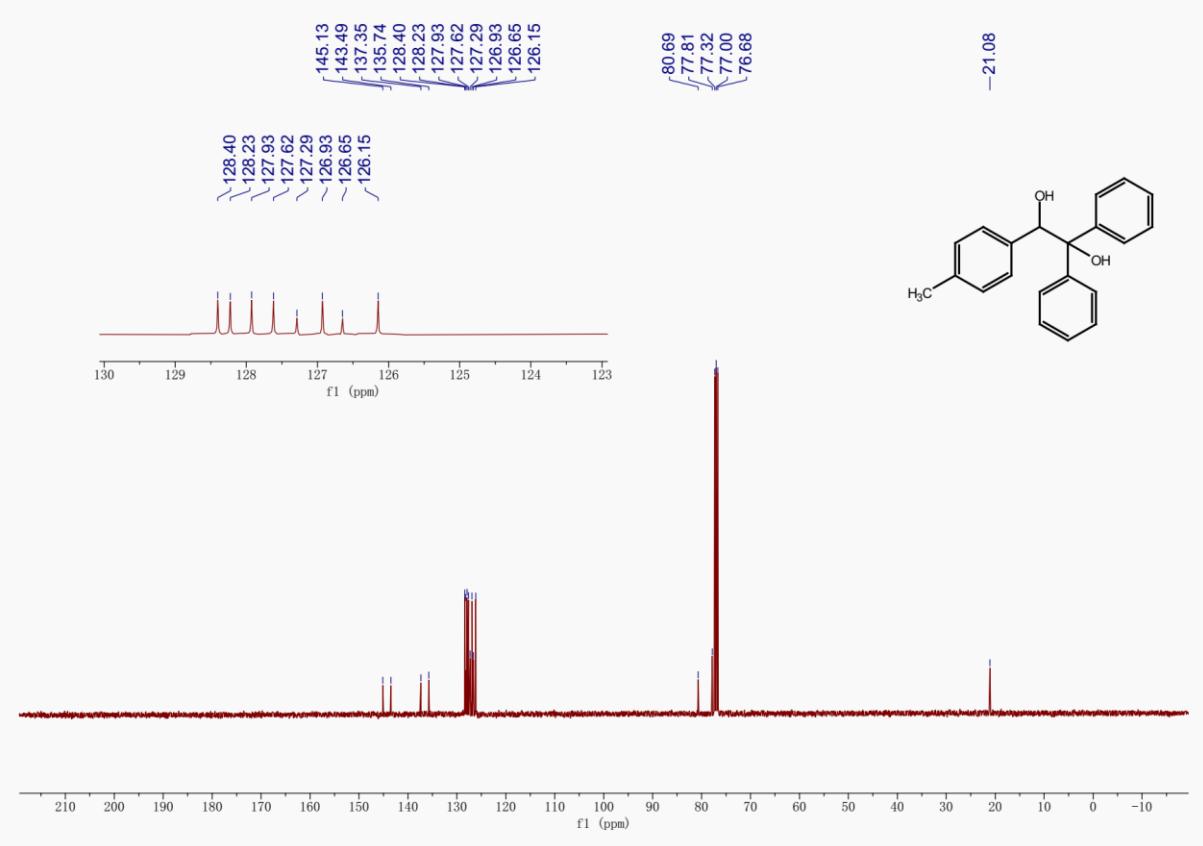
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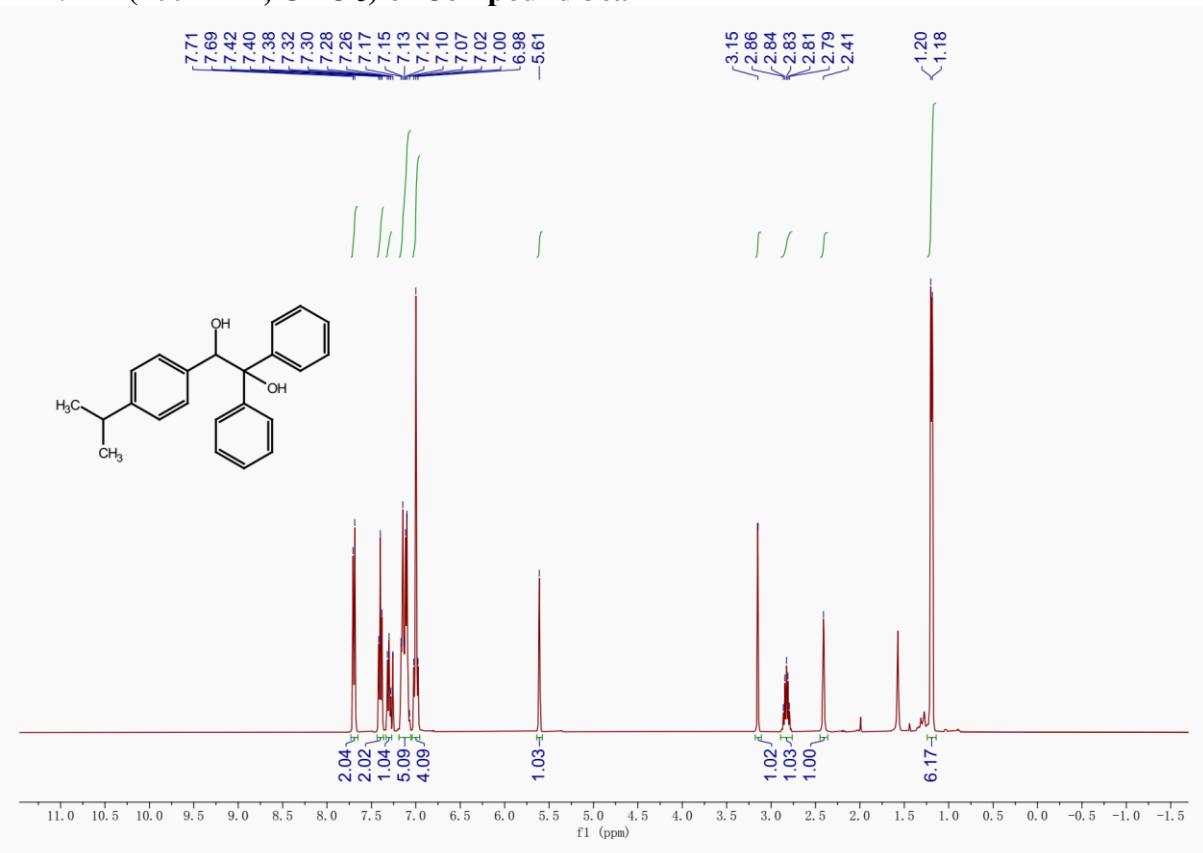
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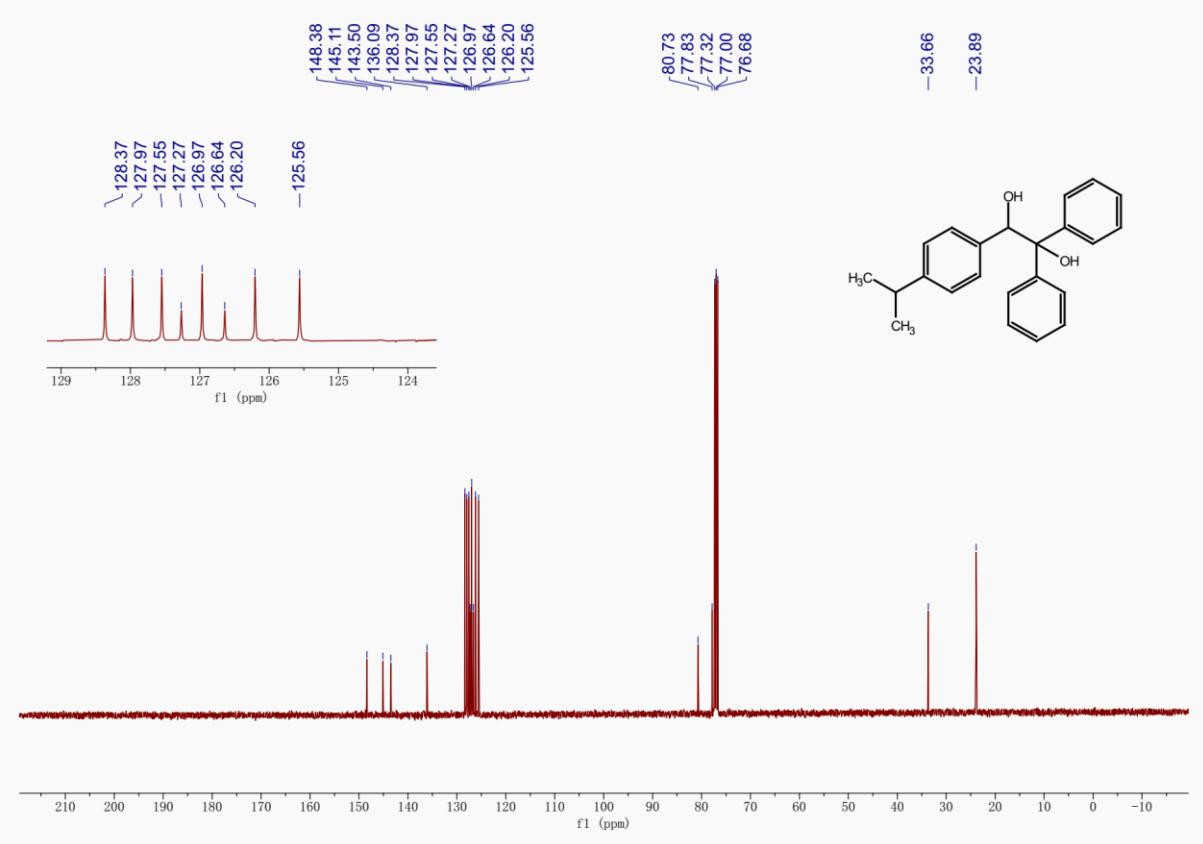
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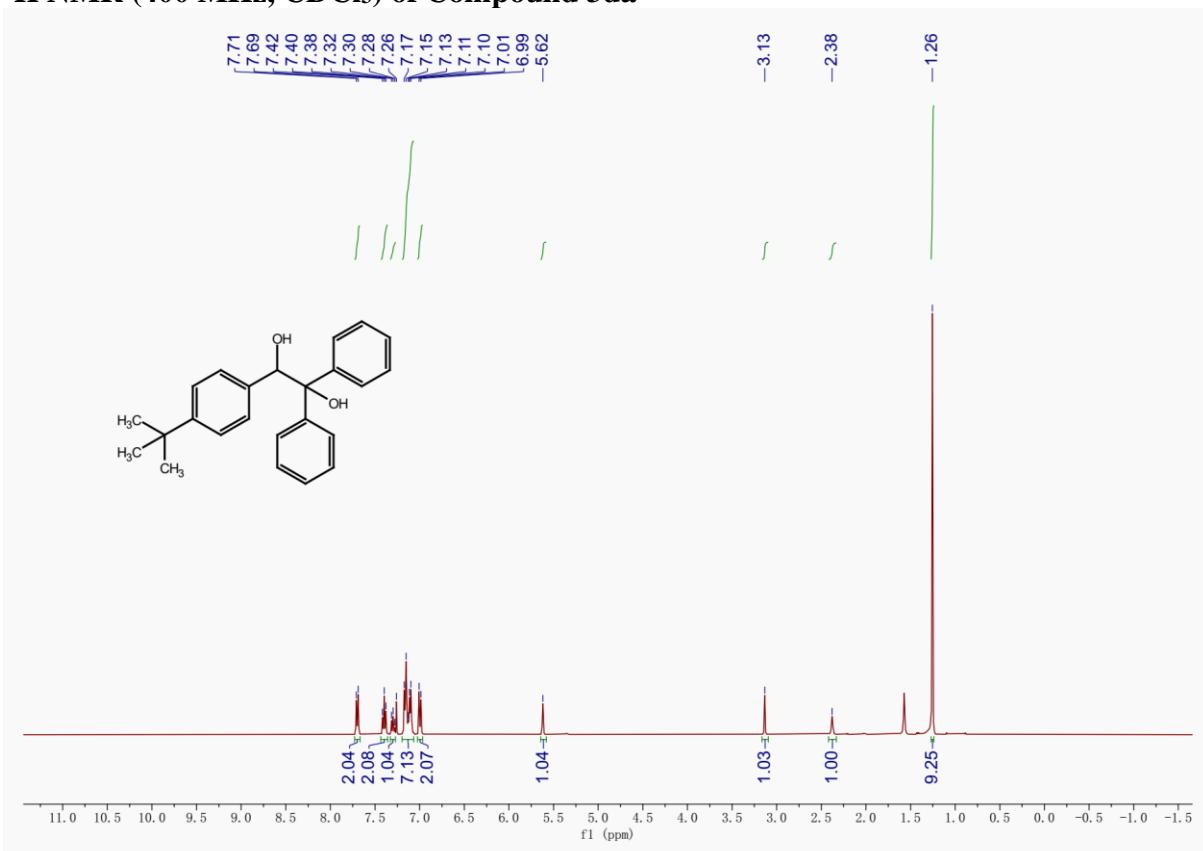
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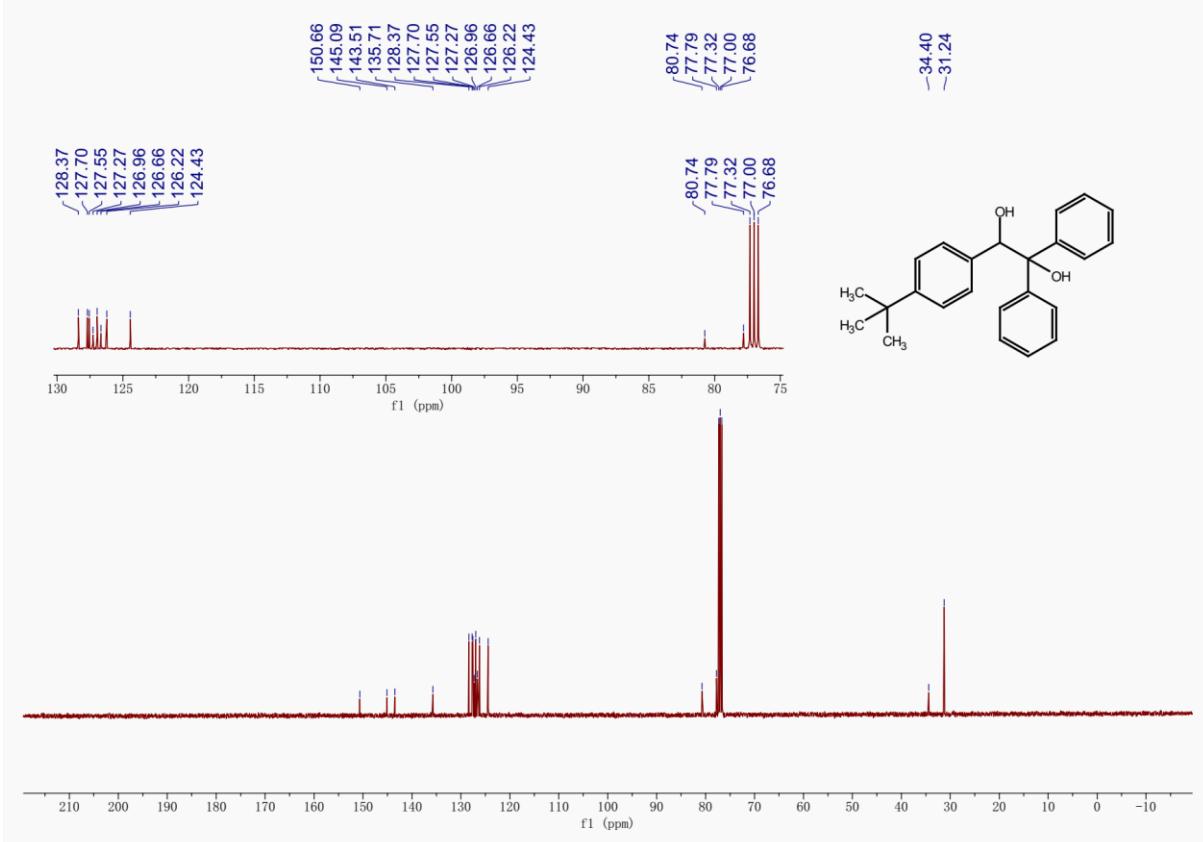
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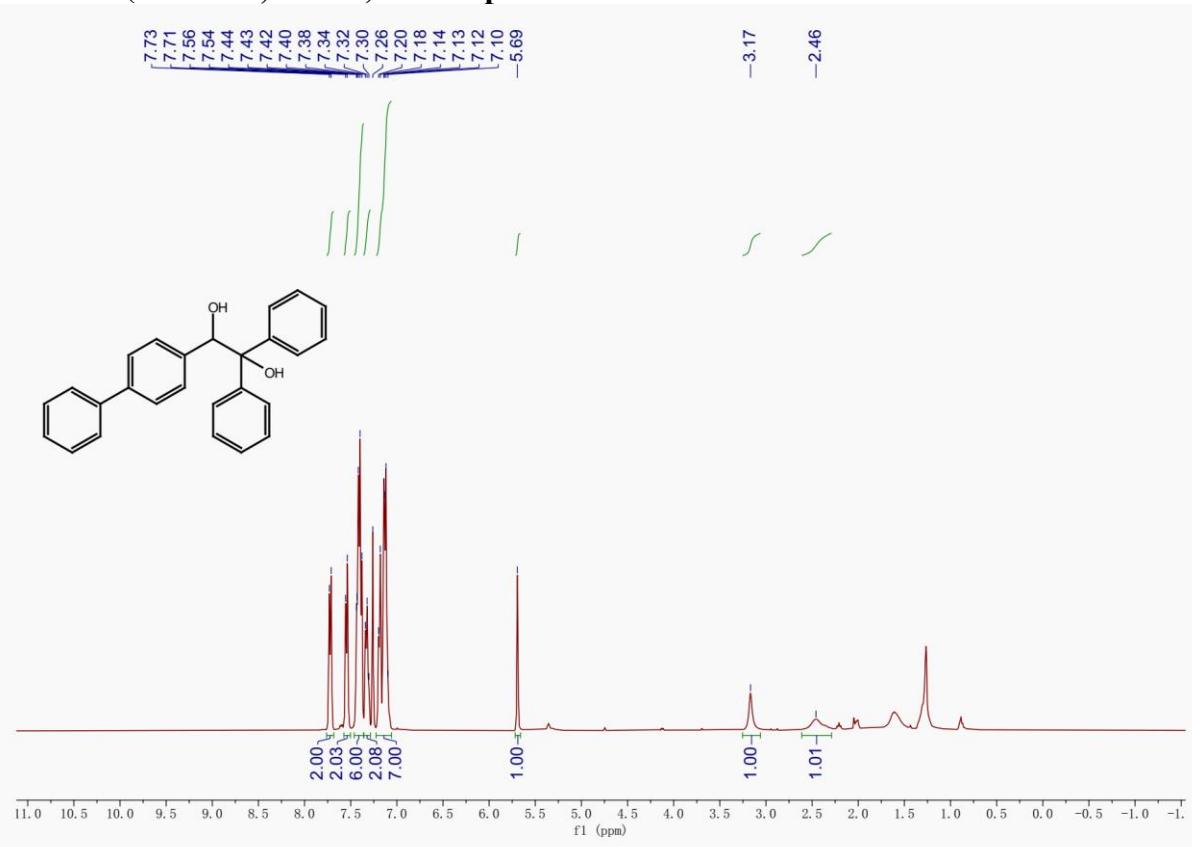
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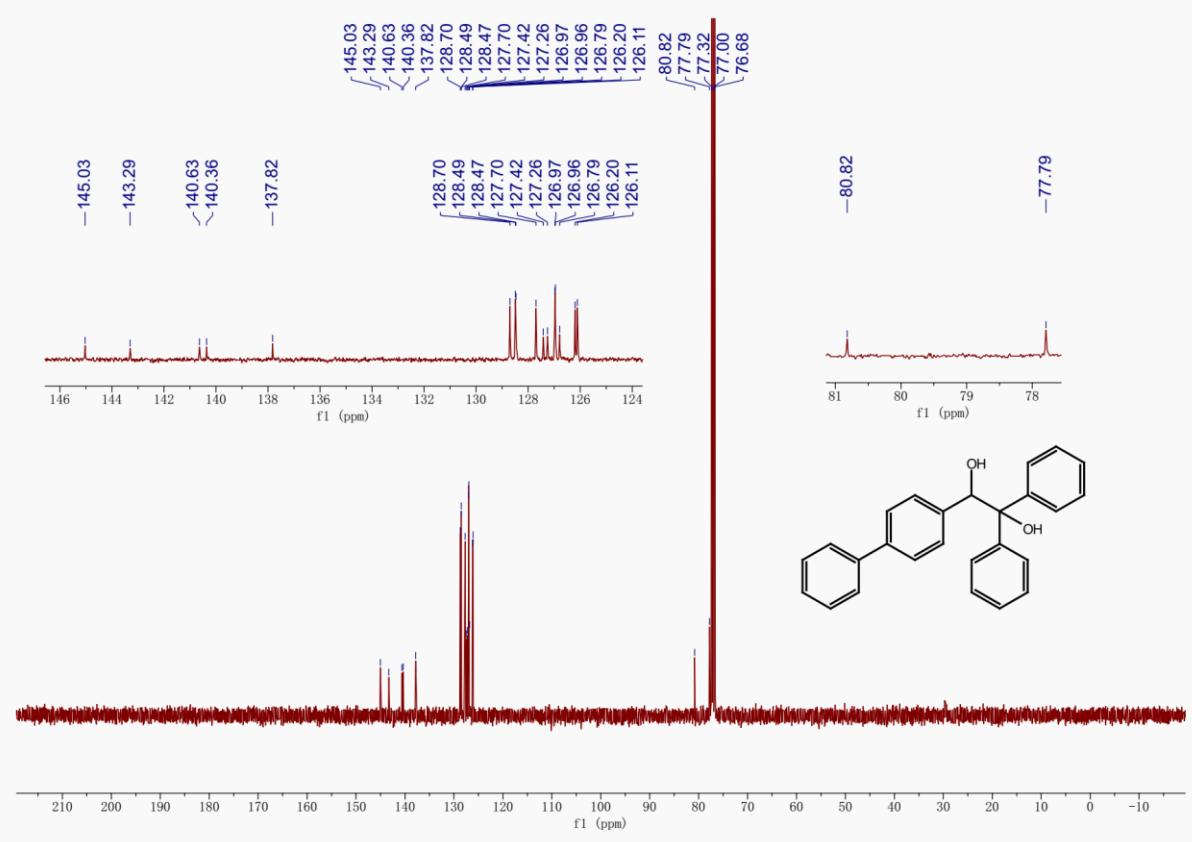
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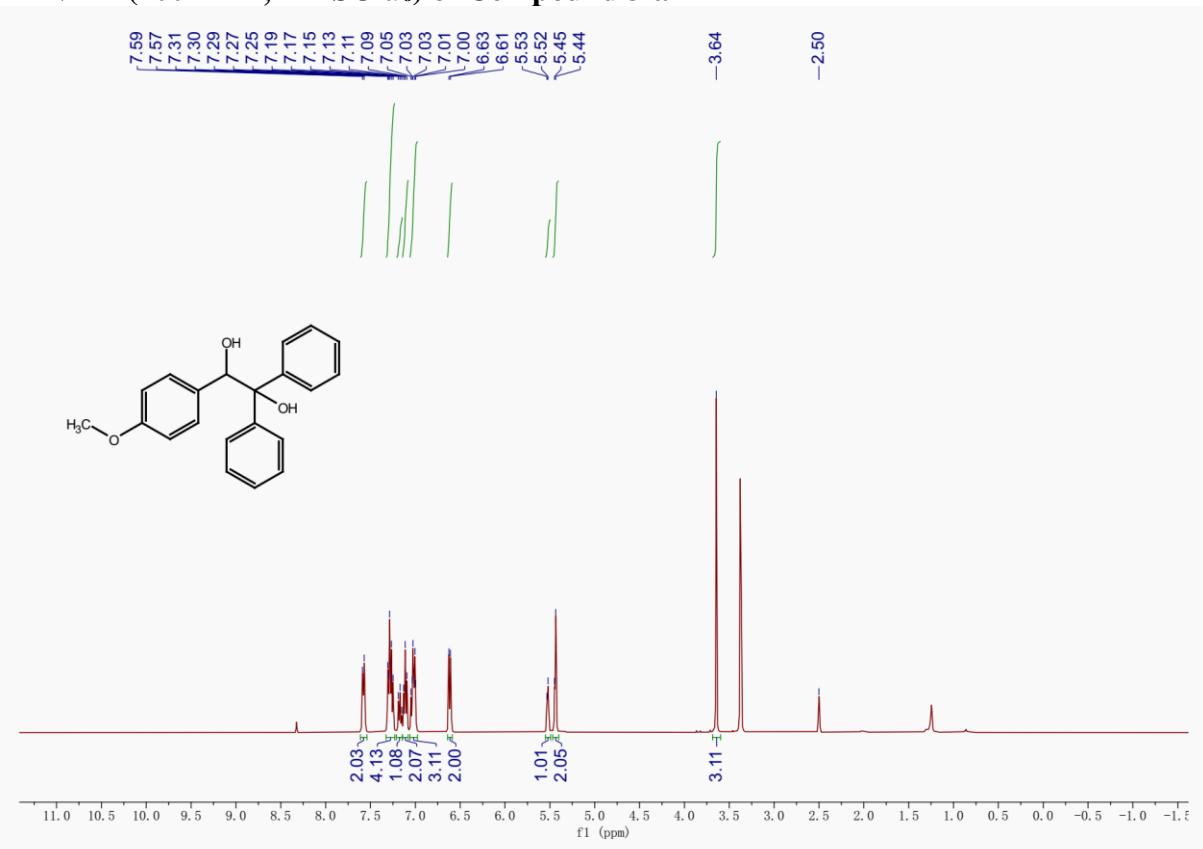
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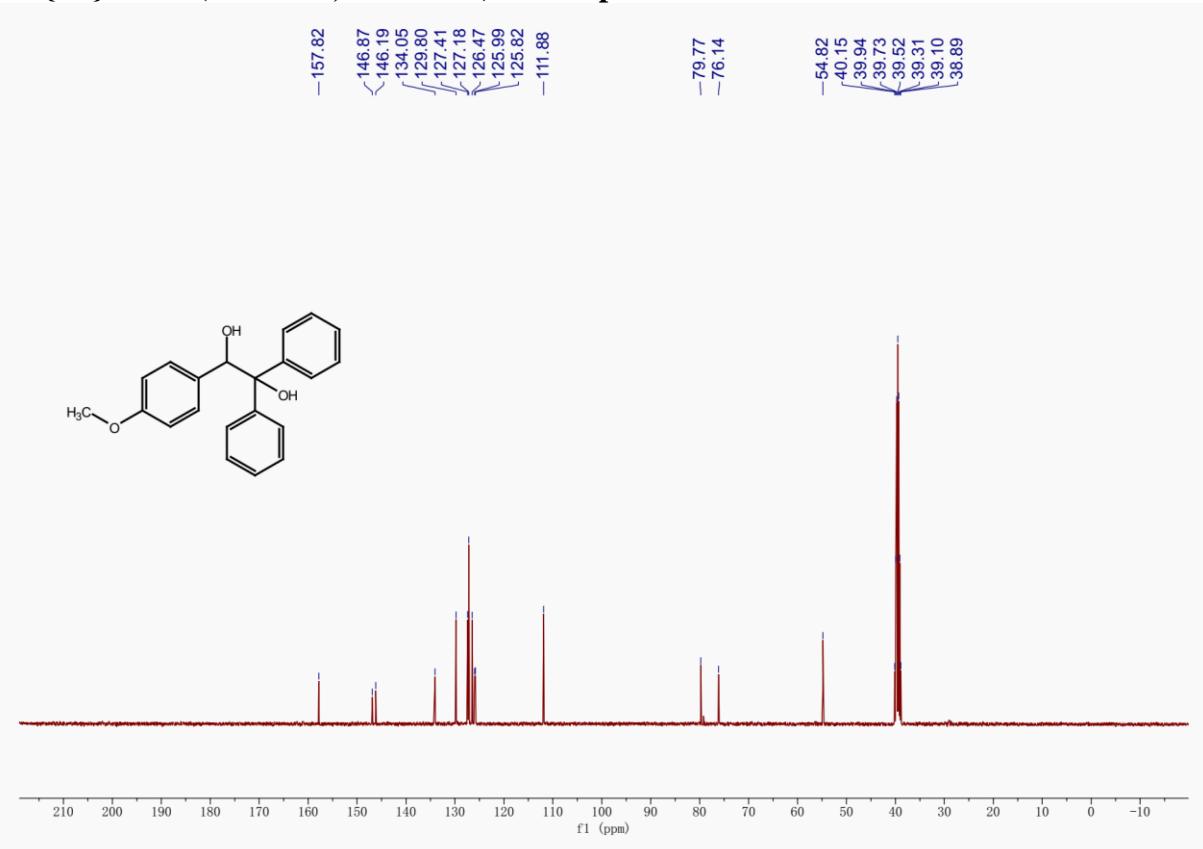
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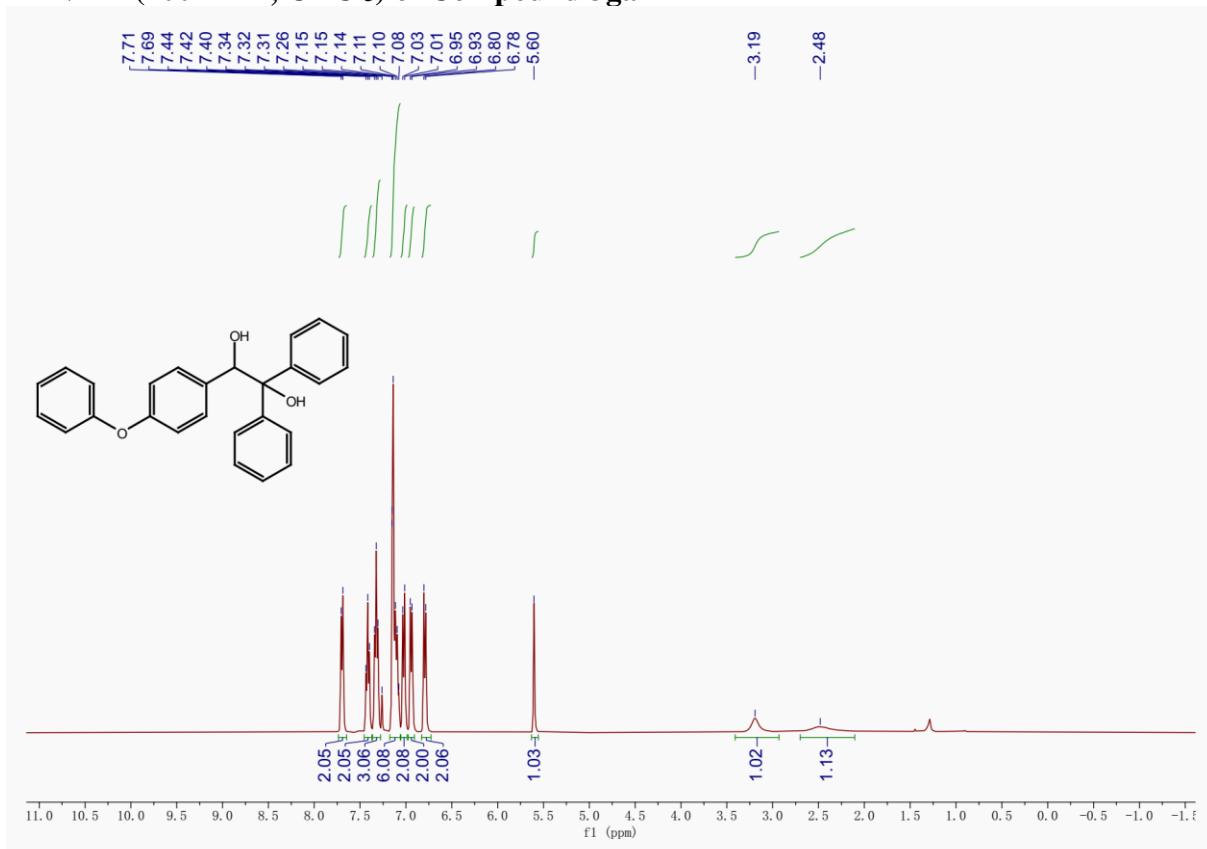
^1H NMR (400 MHz, DMSO- d_6) of Compound 3fa



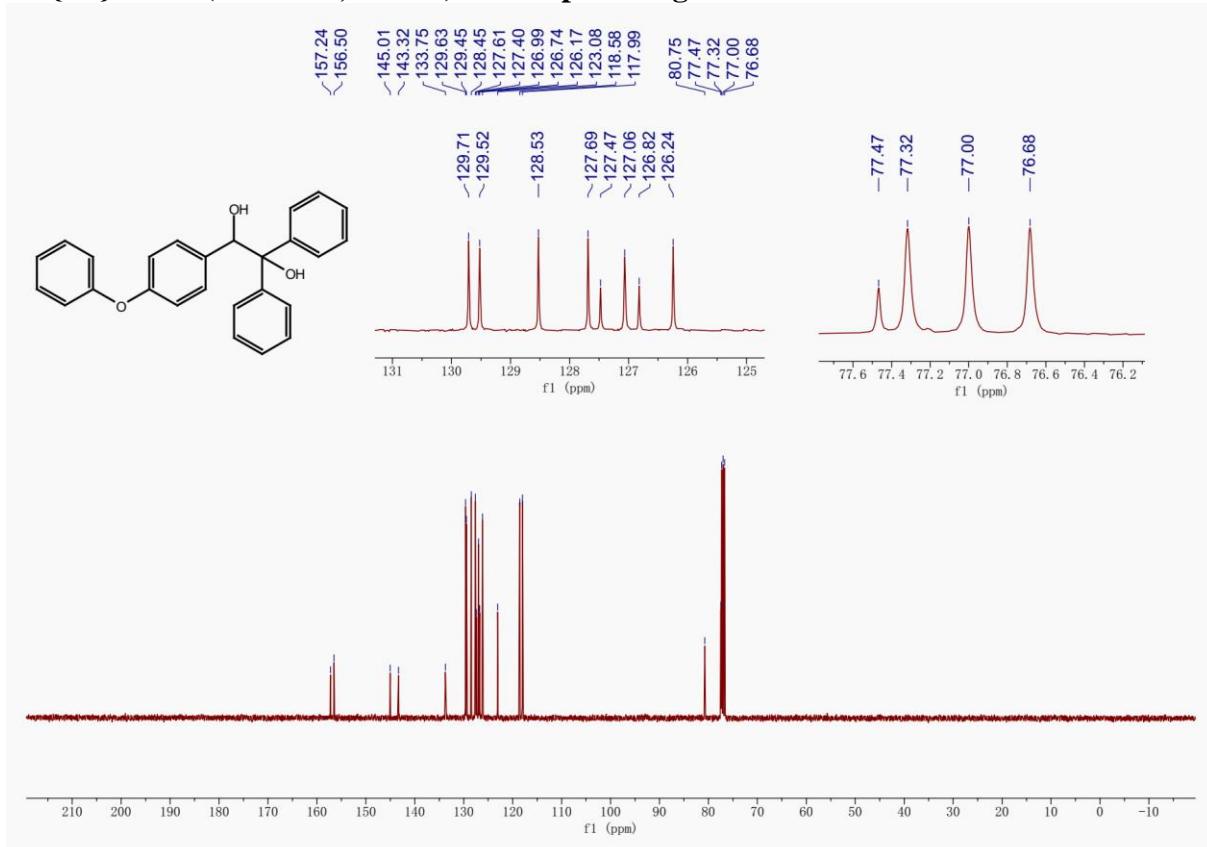
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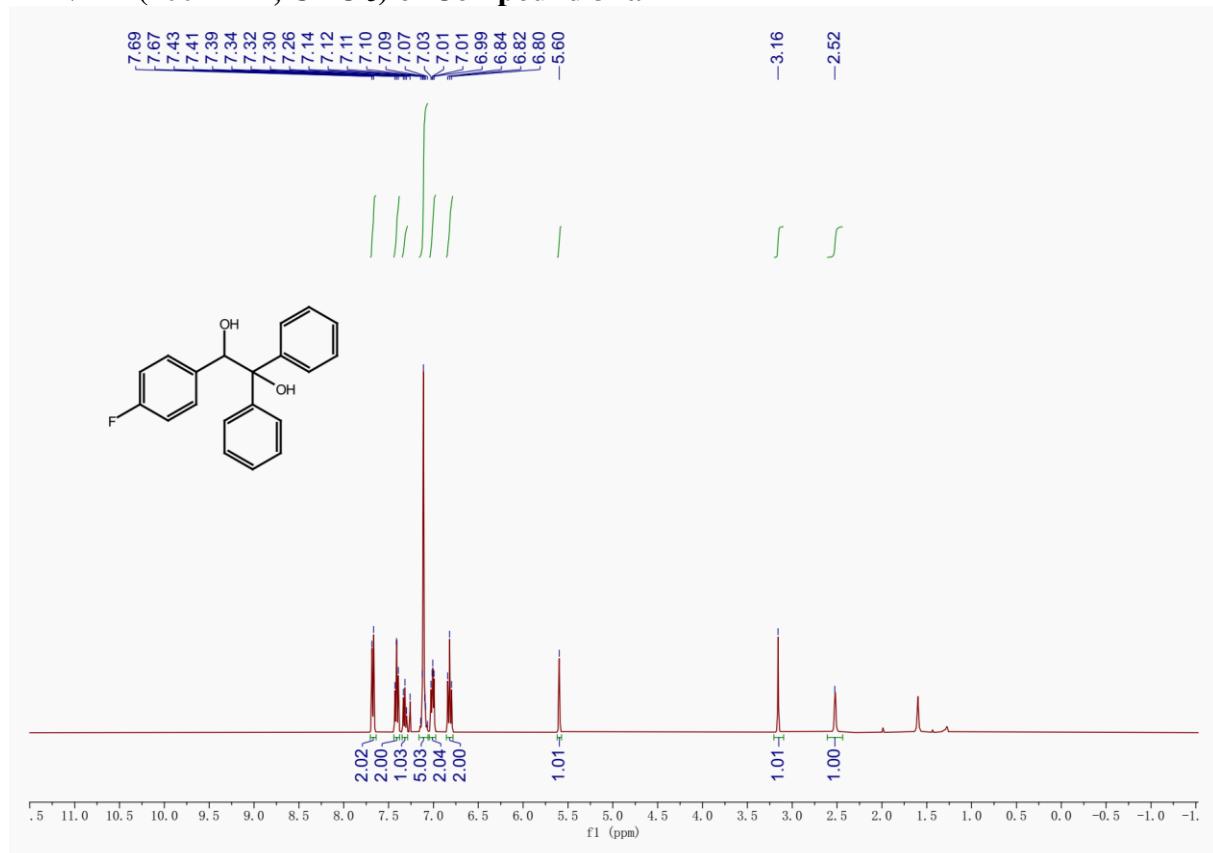
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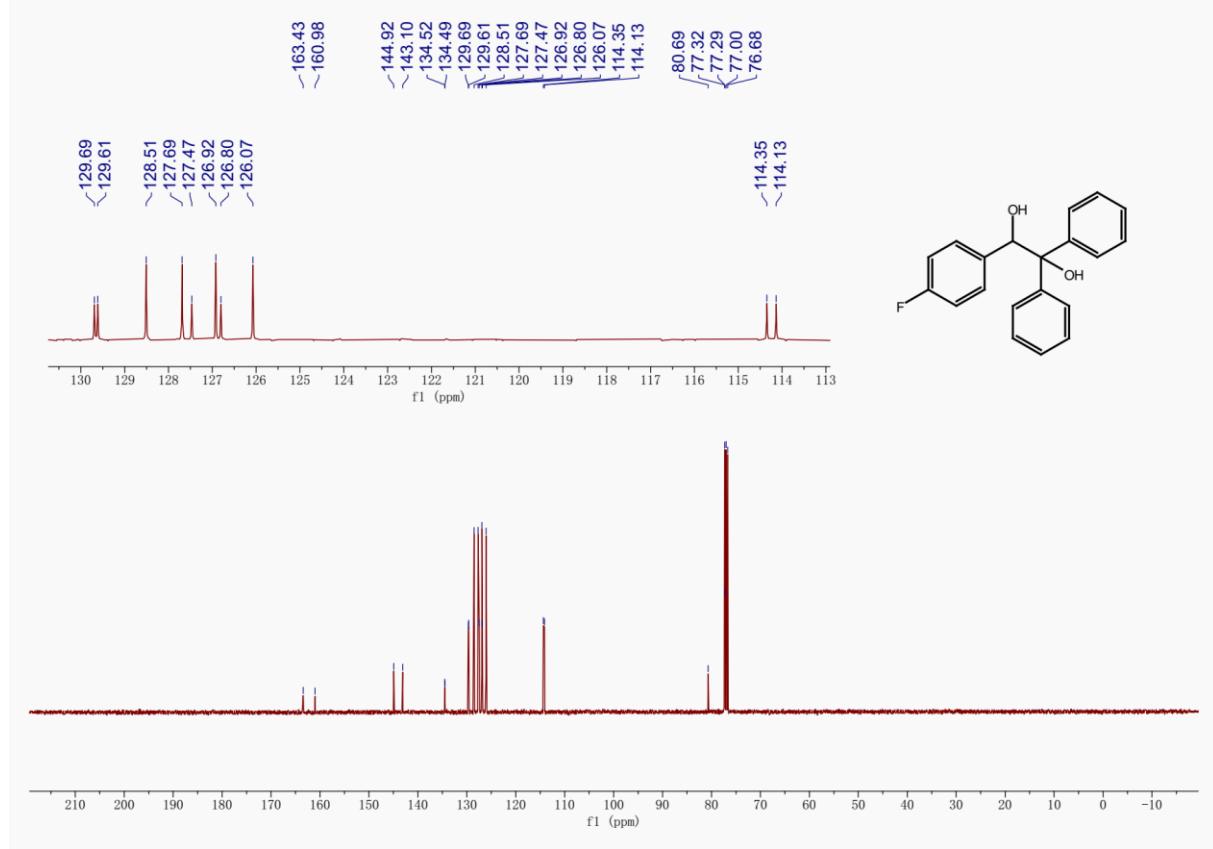
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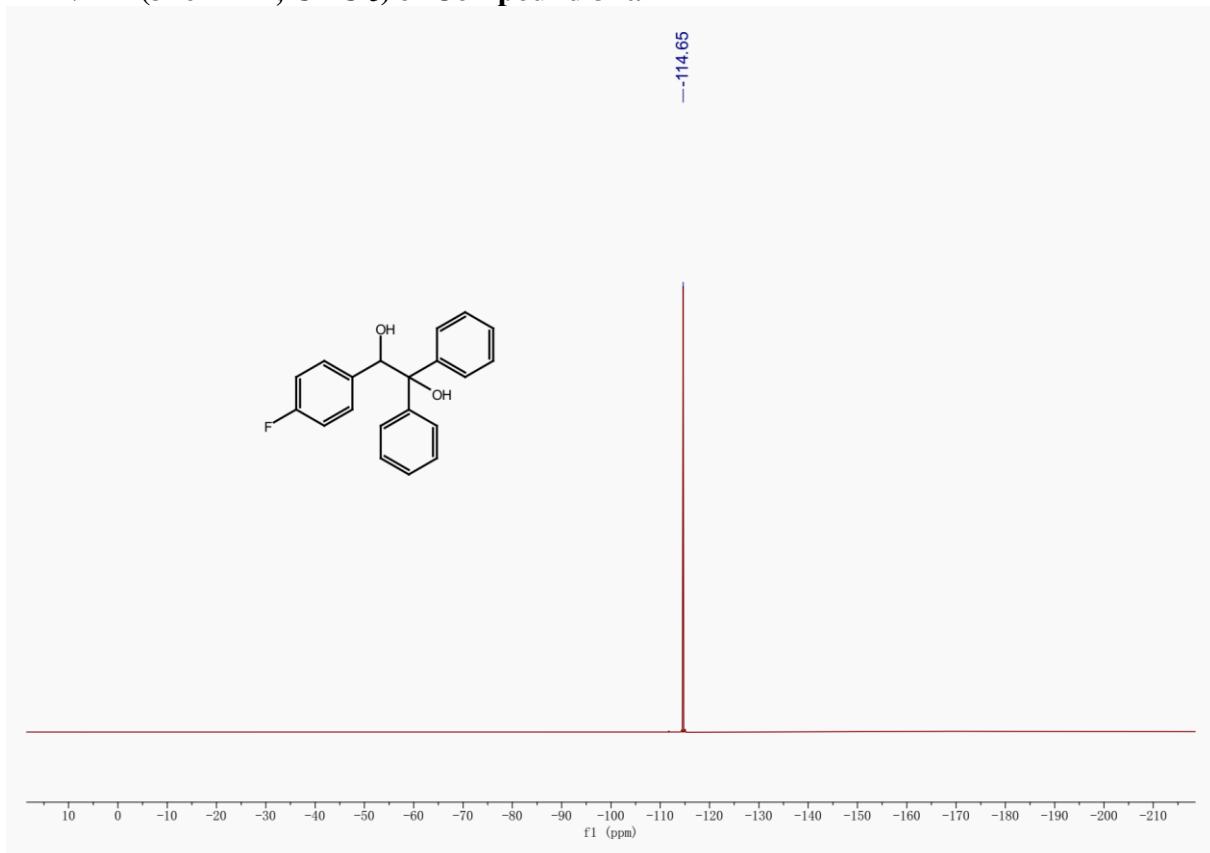
¹H NMR (400 MHz, CDCl₃) of Compound 3ha



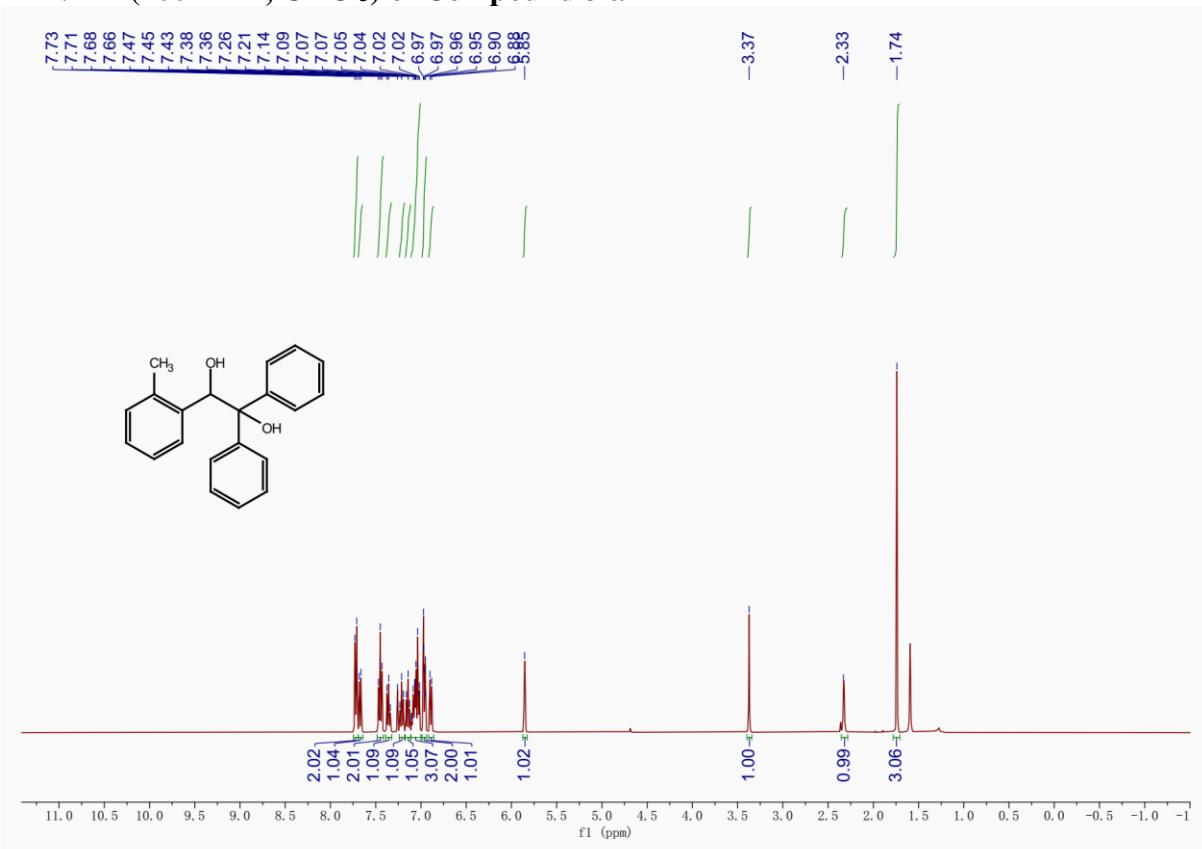
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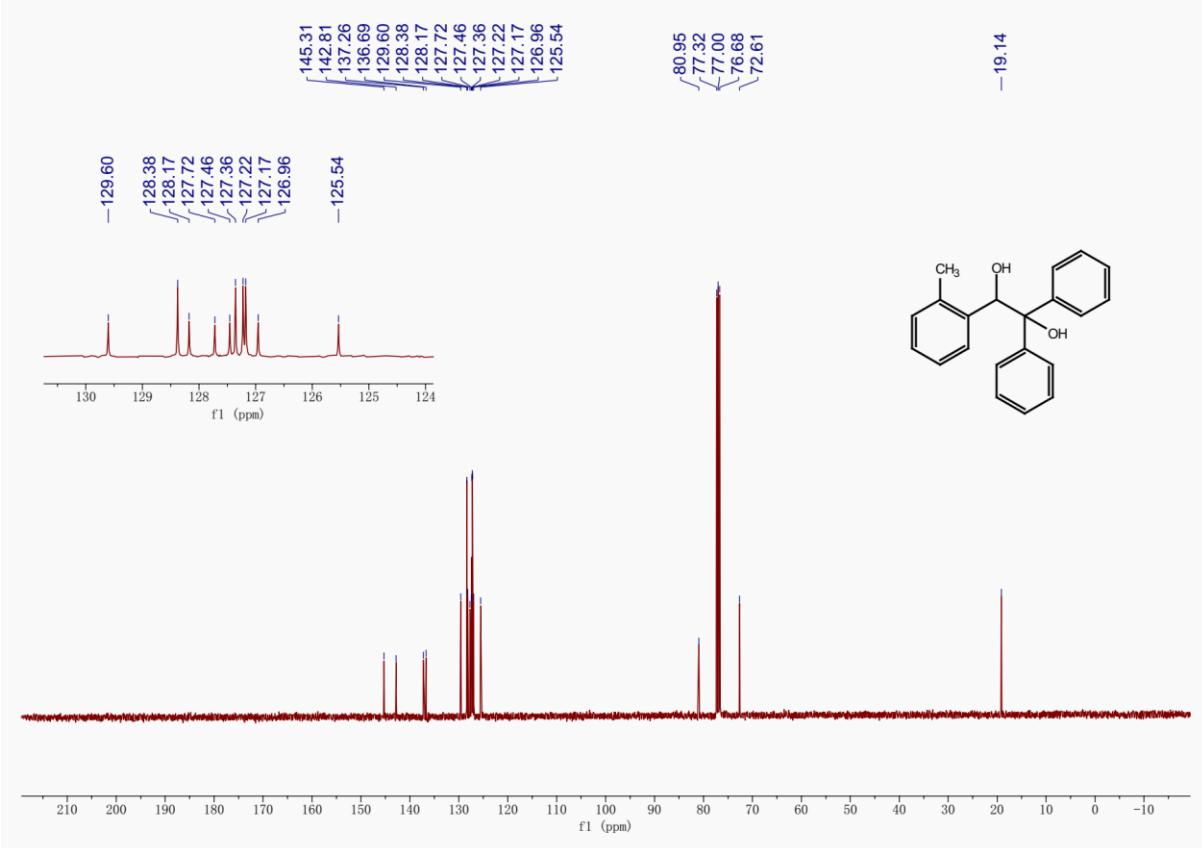
¹⁹F NMR (376 MHz, CDCl₃) of Compound 3ha



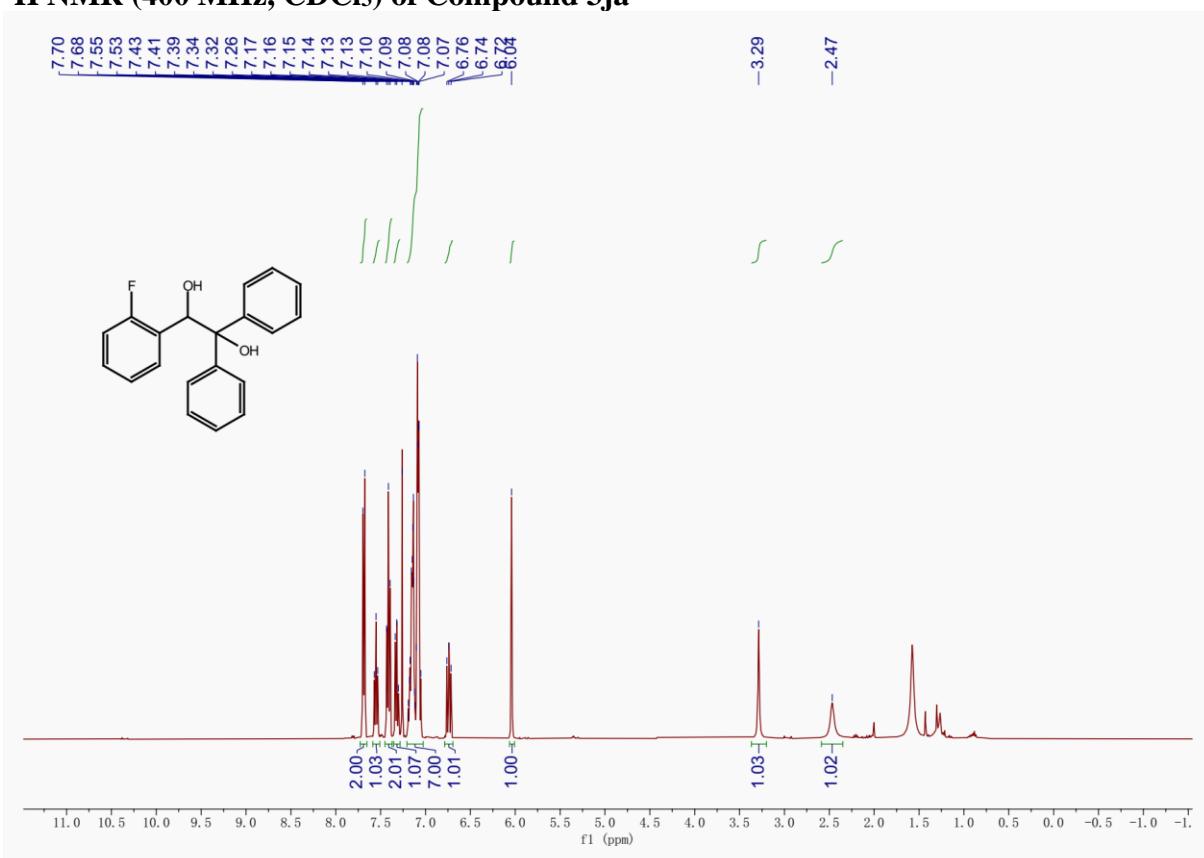
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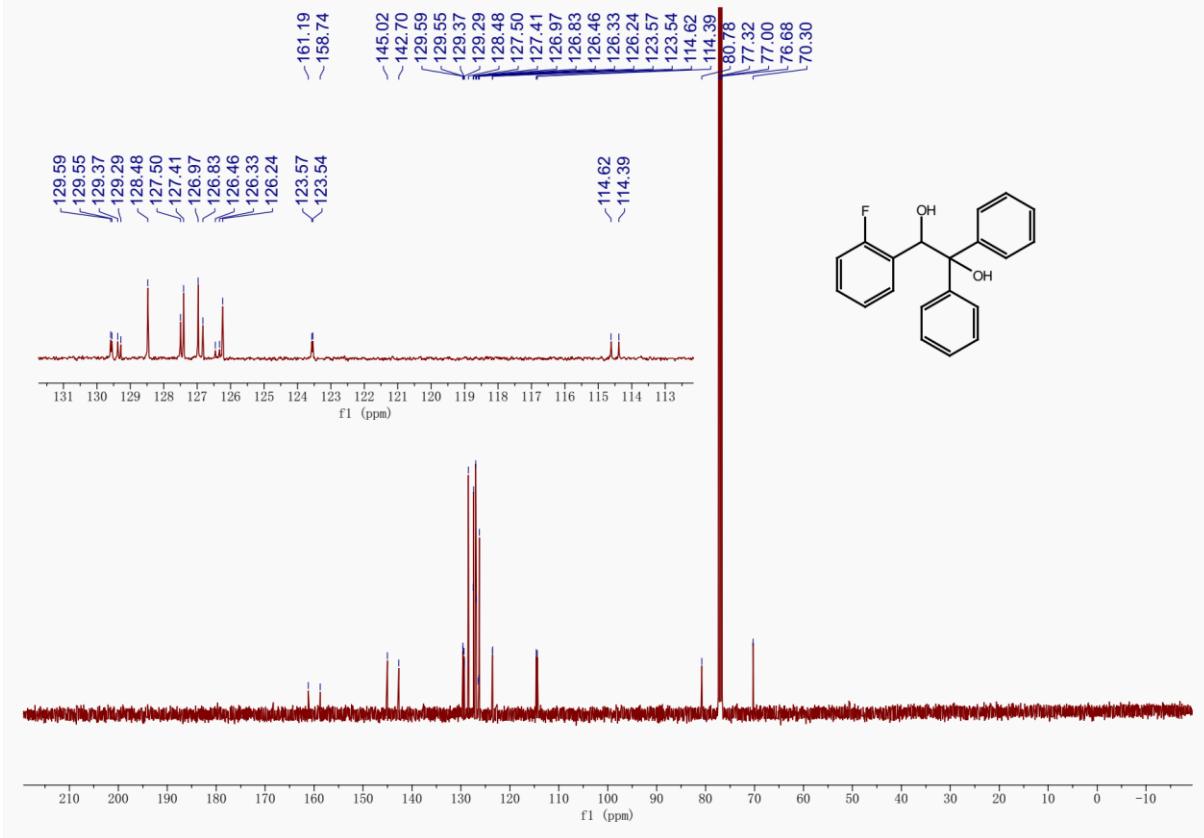
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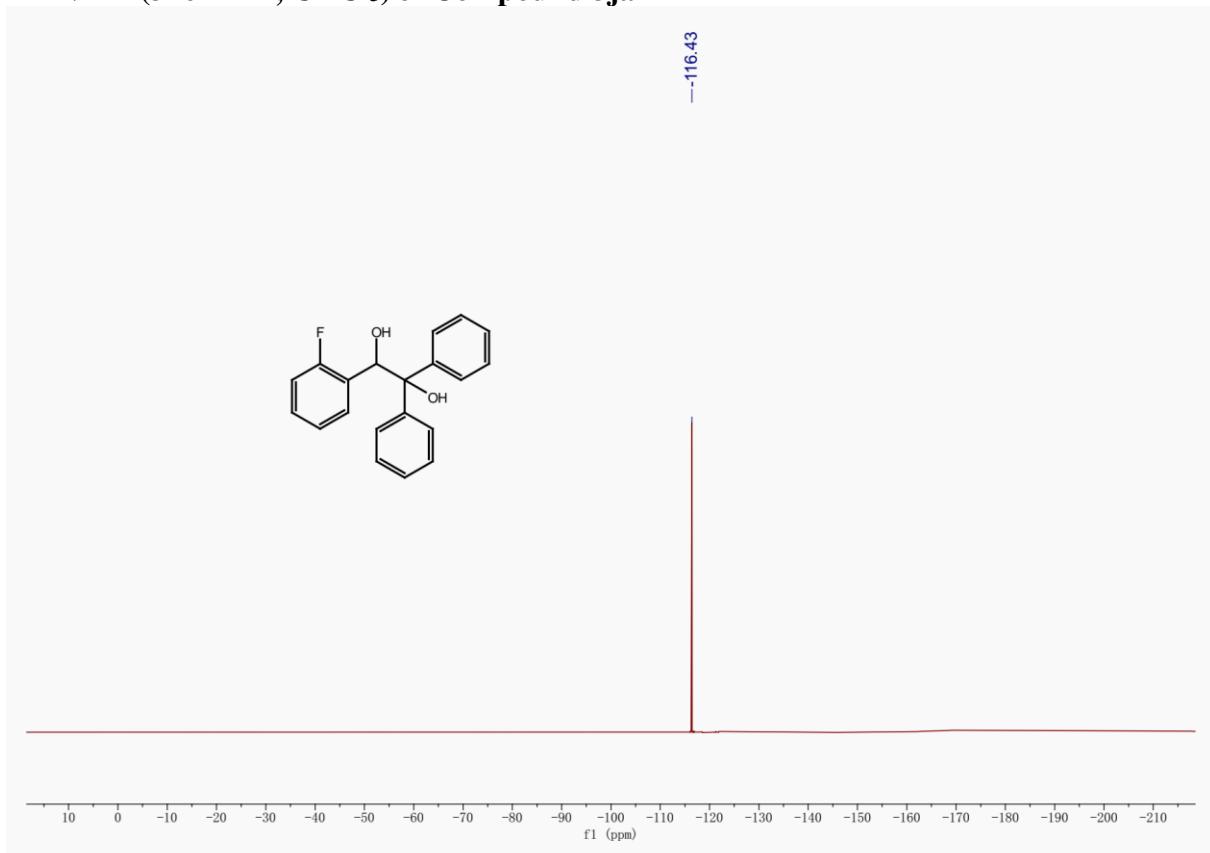
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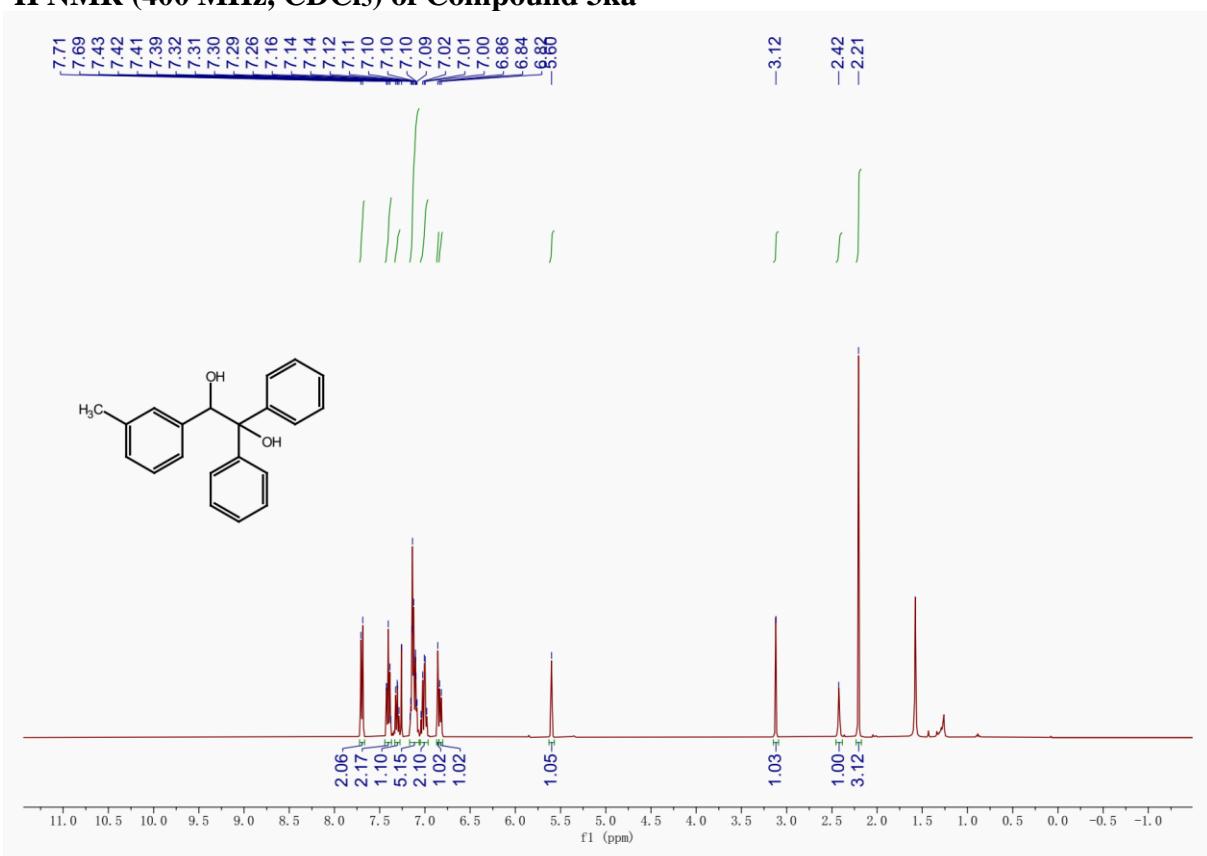
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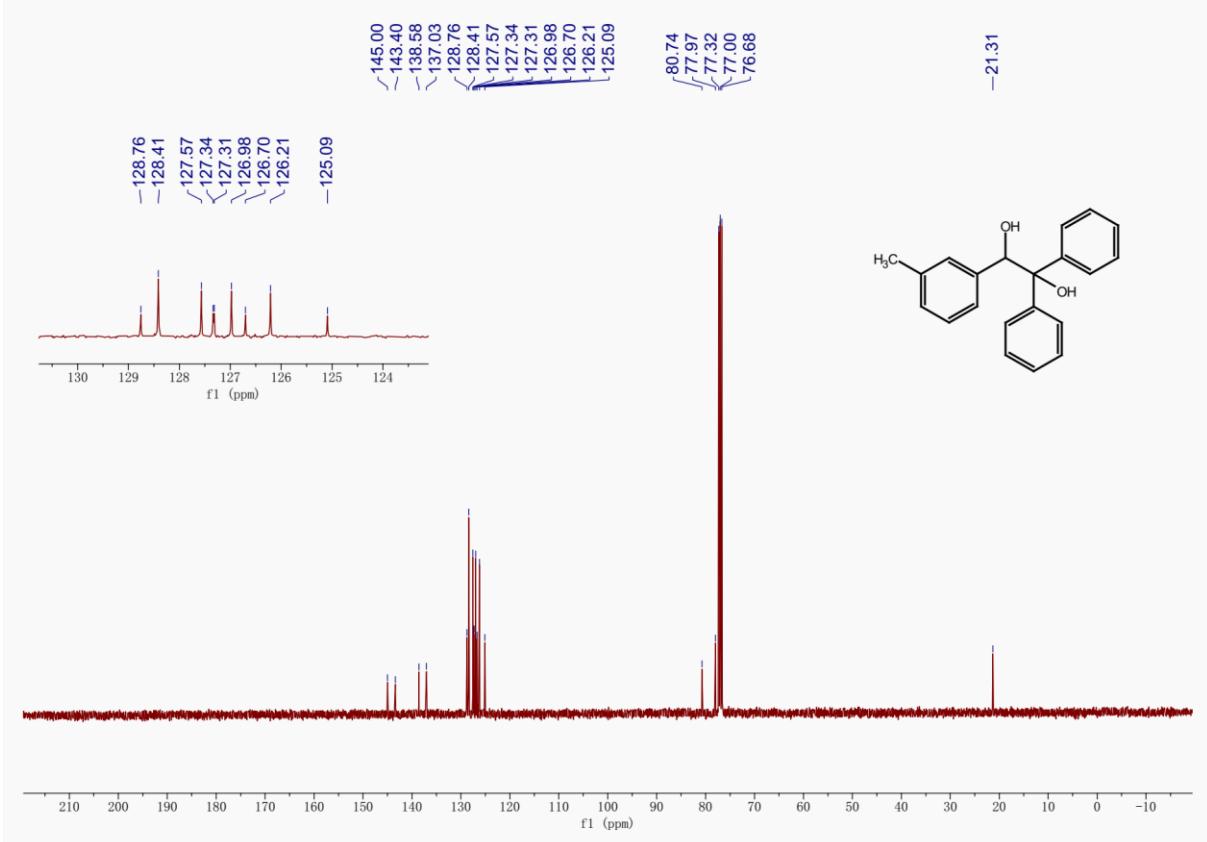
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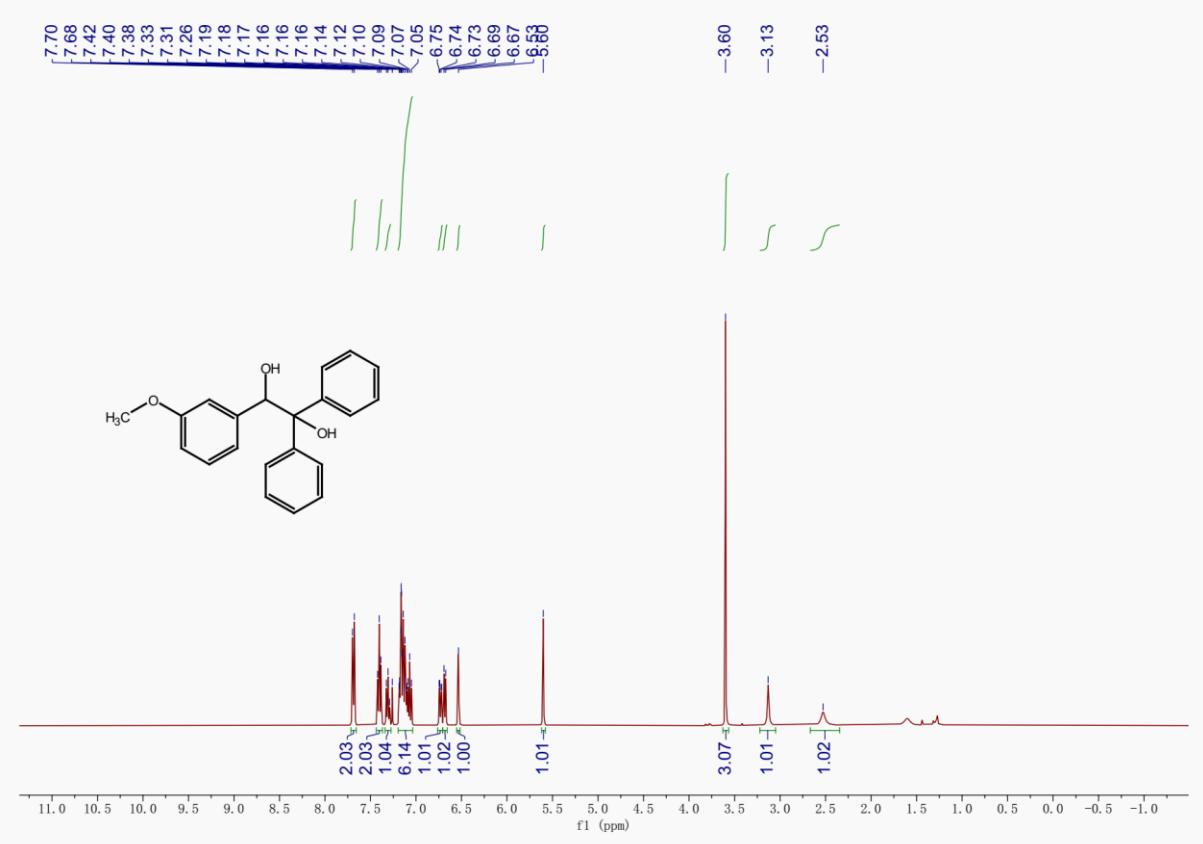
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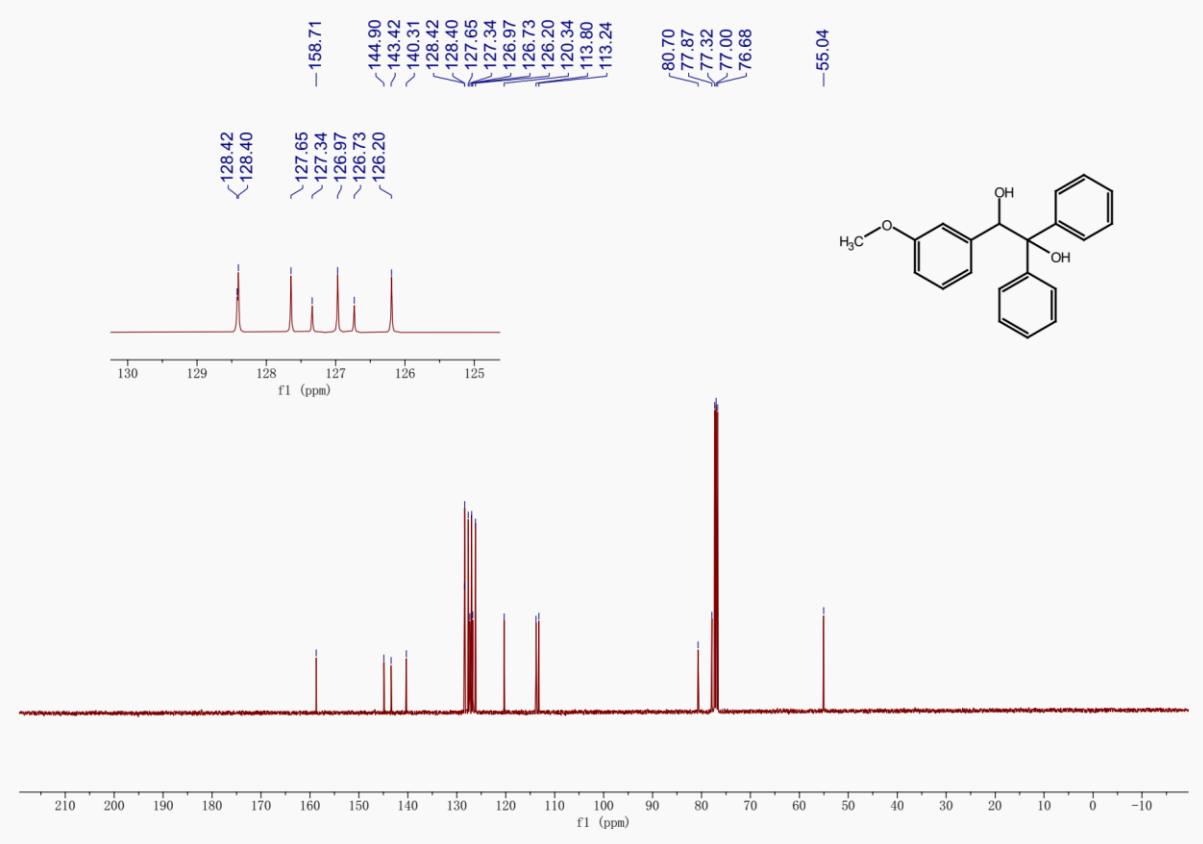
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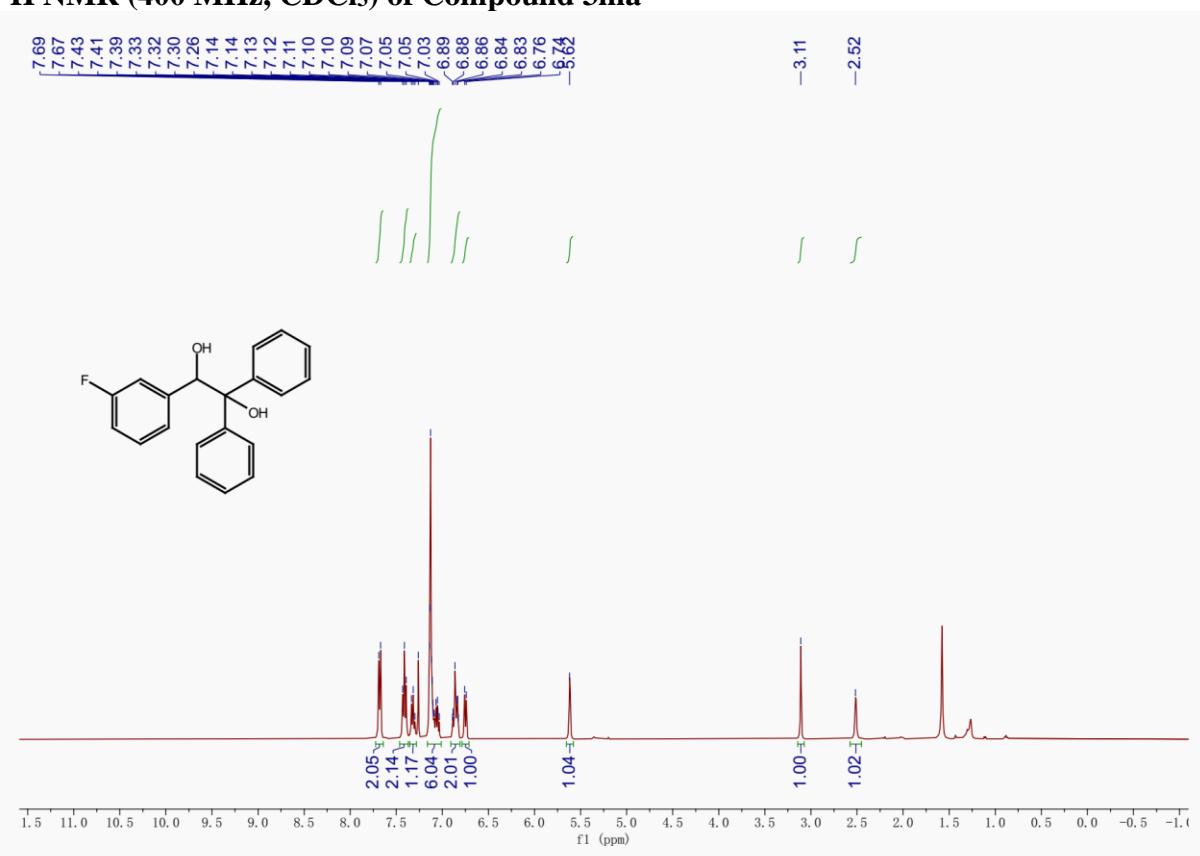
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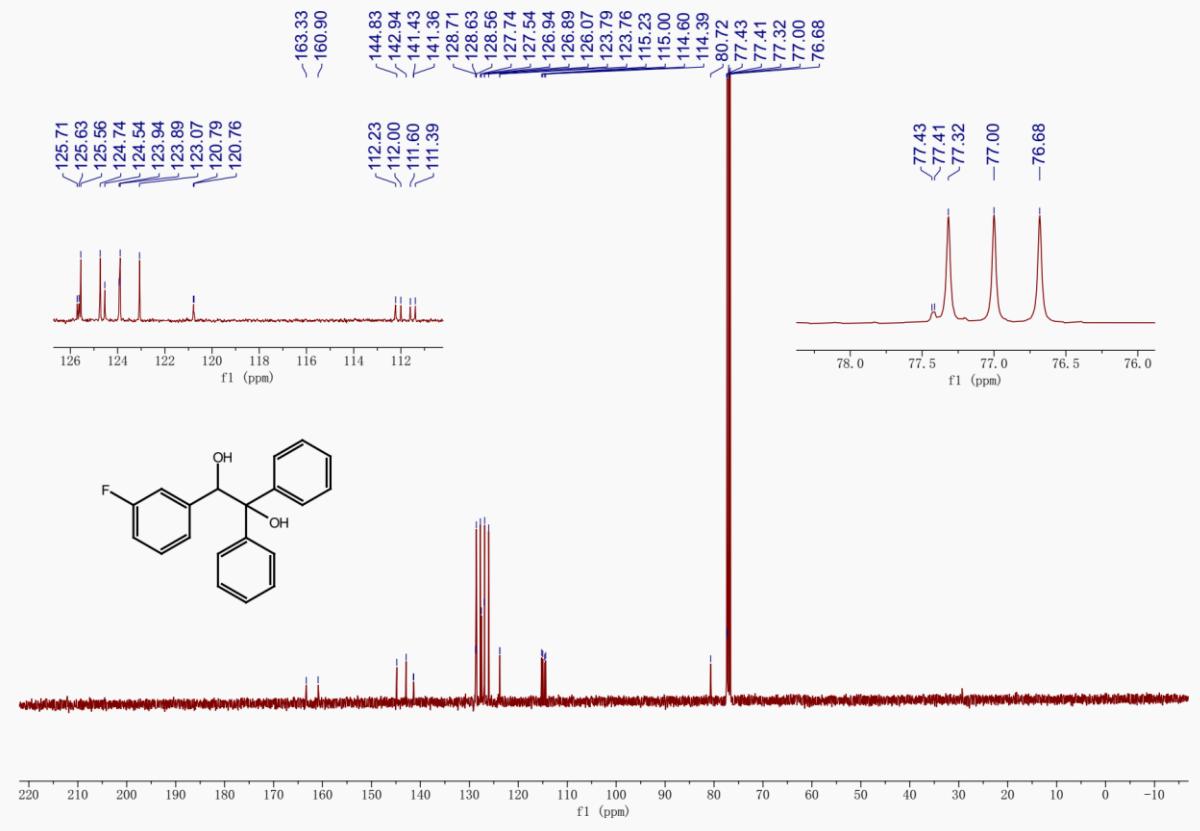
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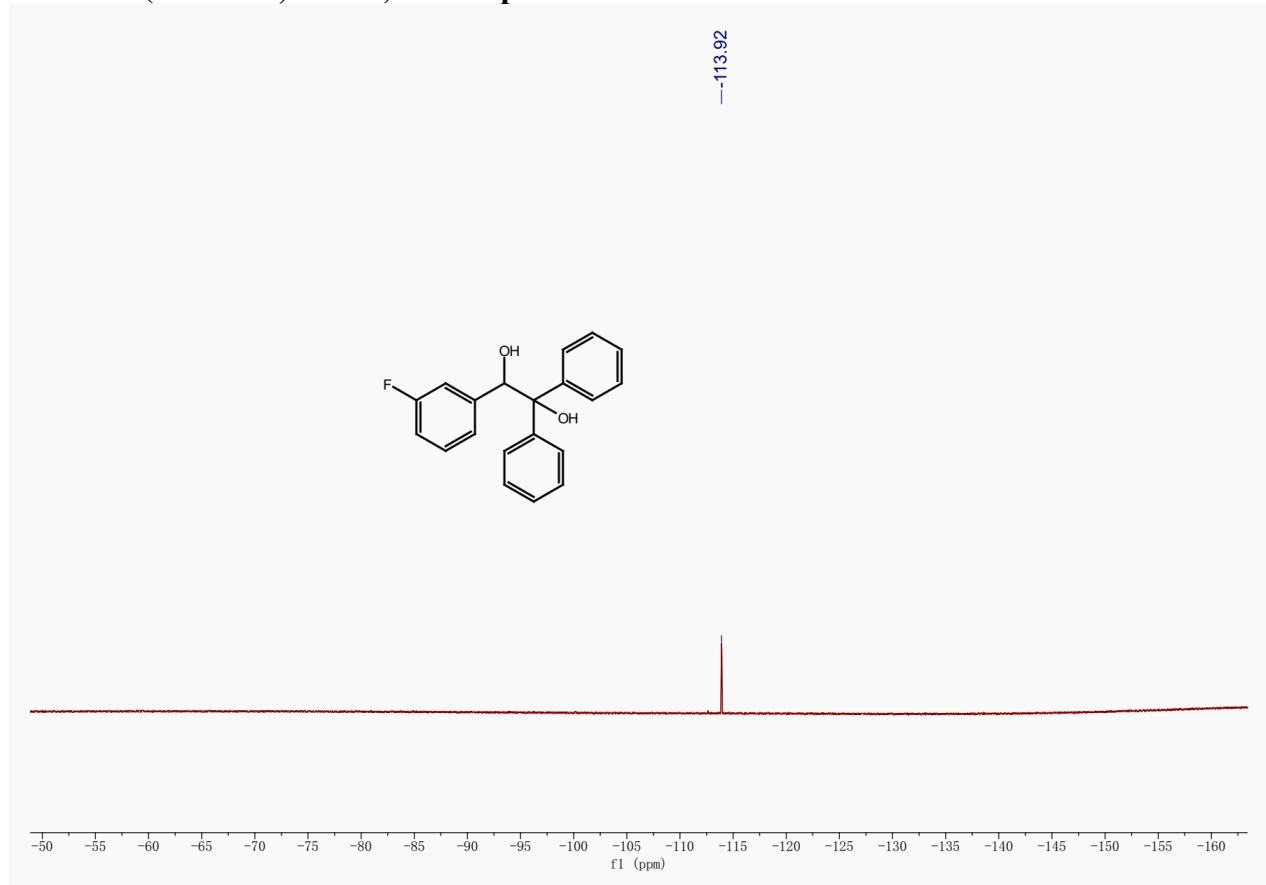
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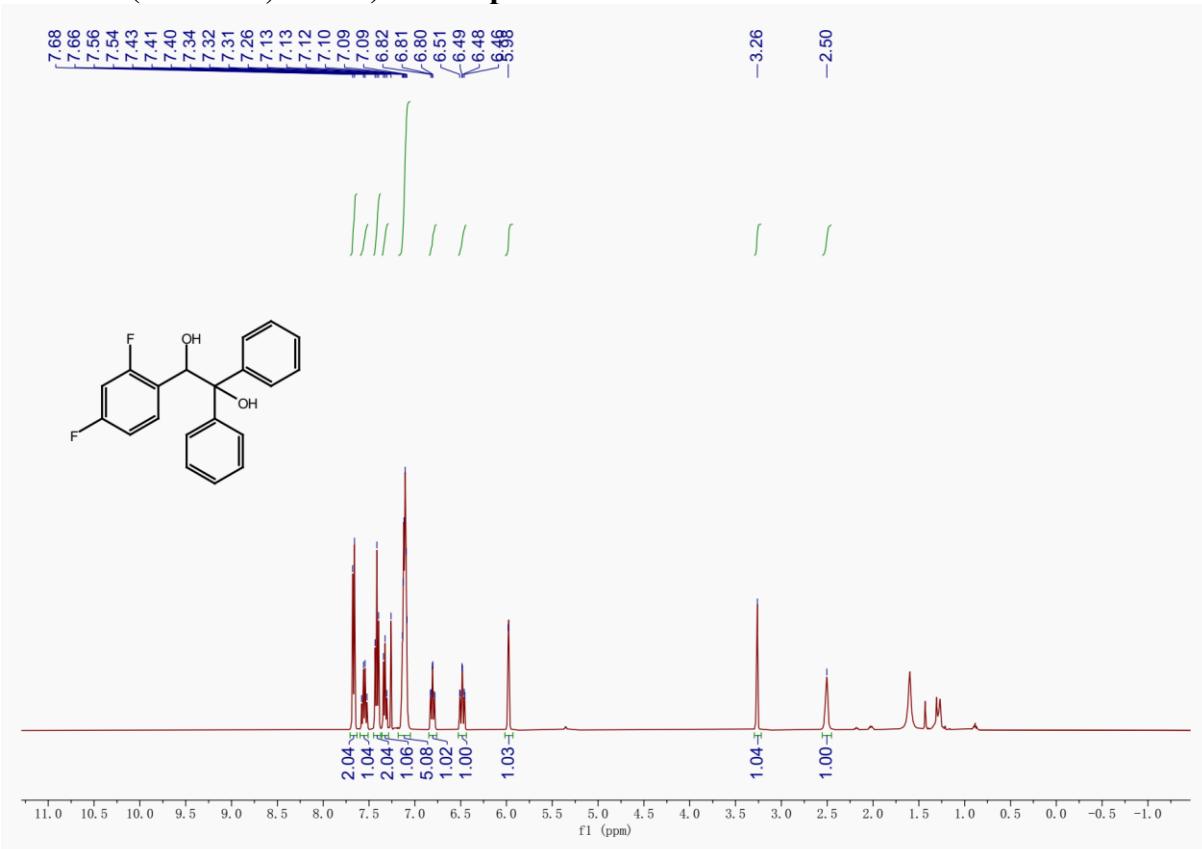
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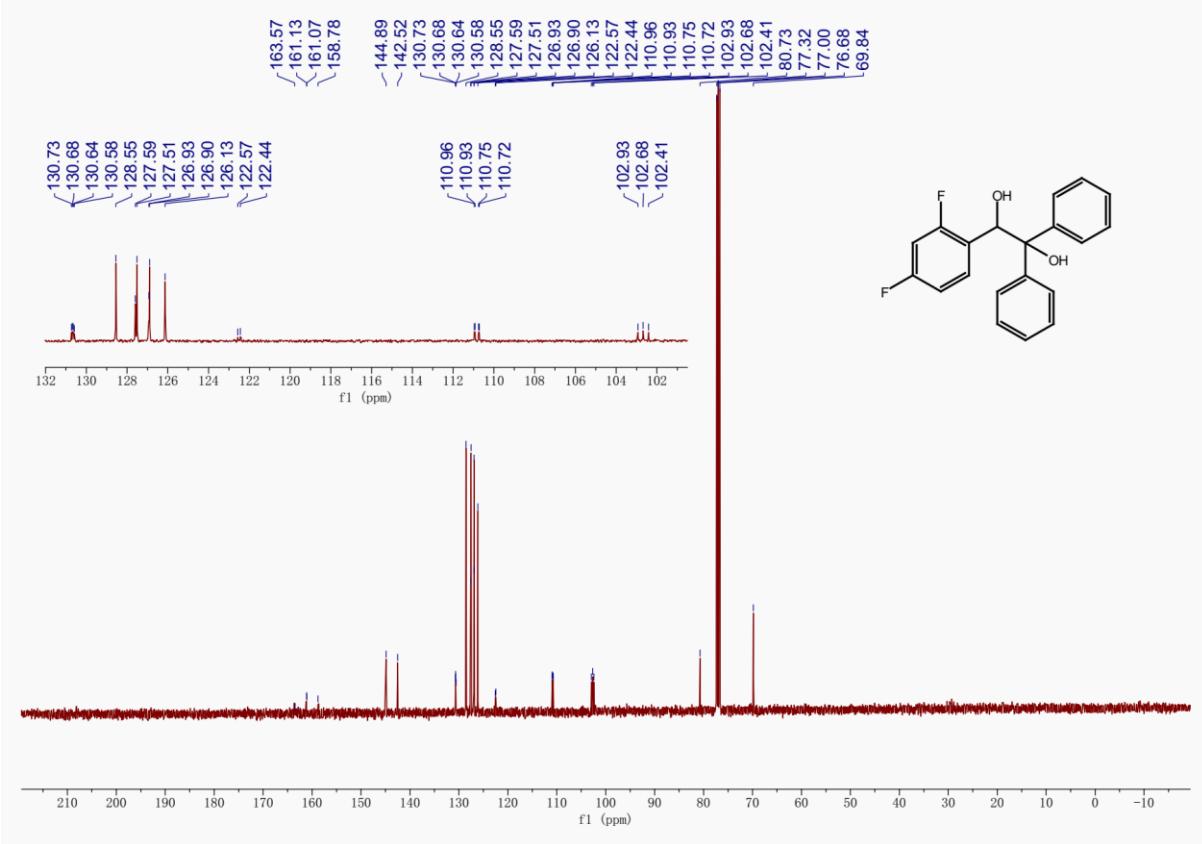
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¹H NMR (400 MHz, CDCl₃) of Compound 3na



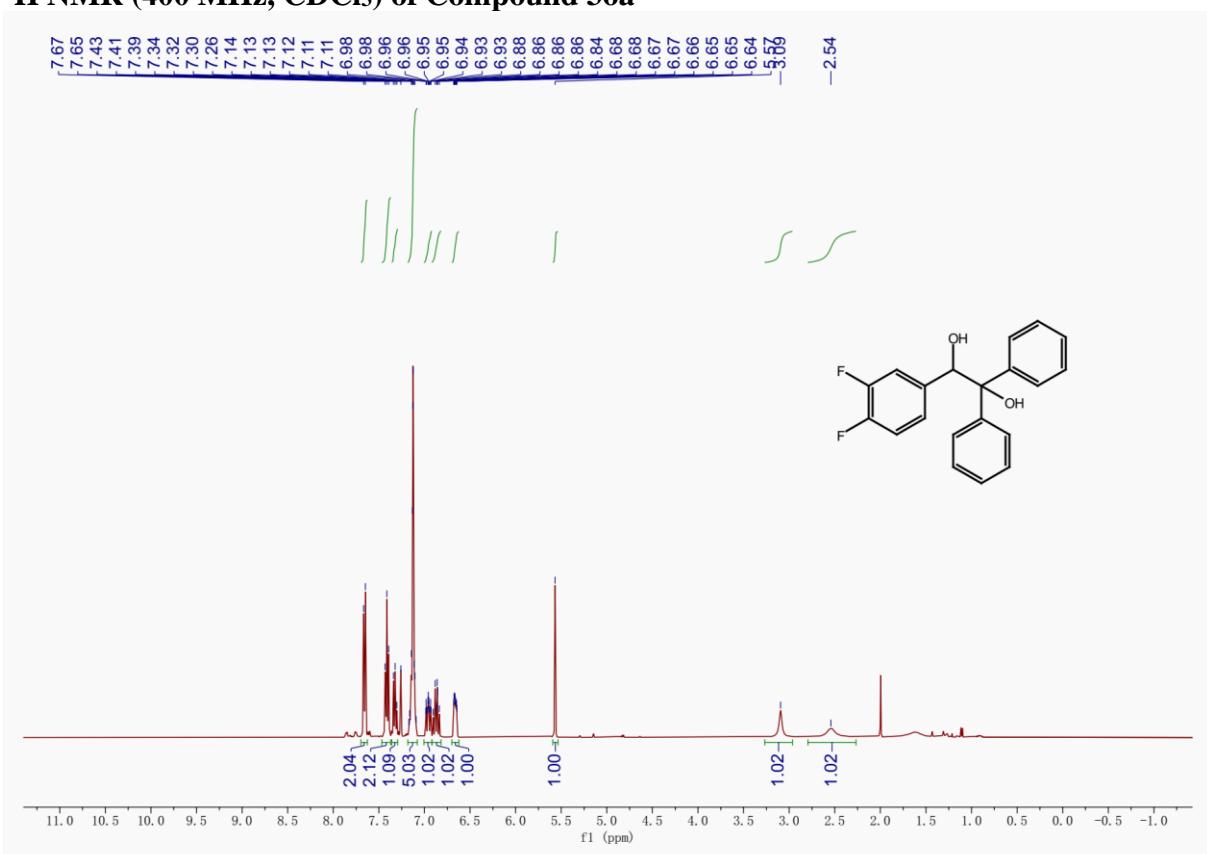
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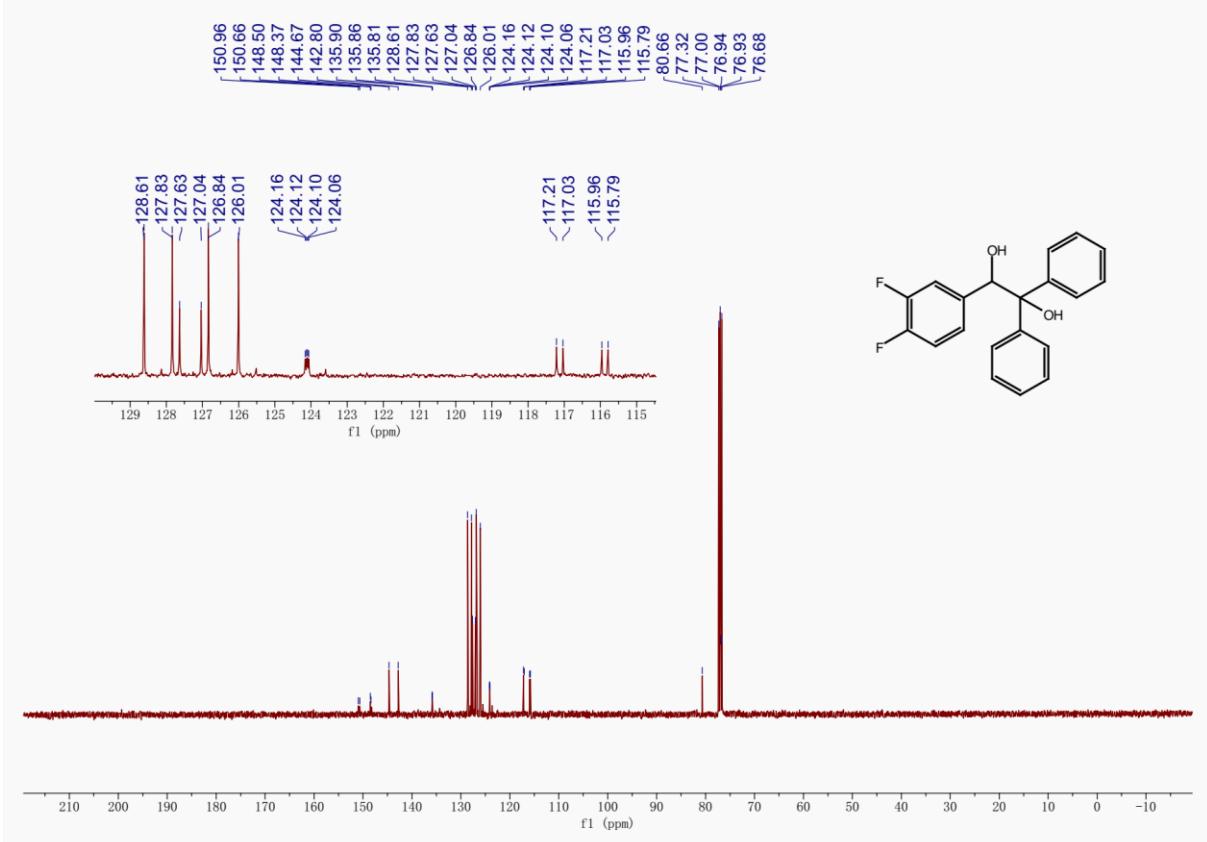
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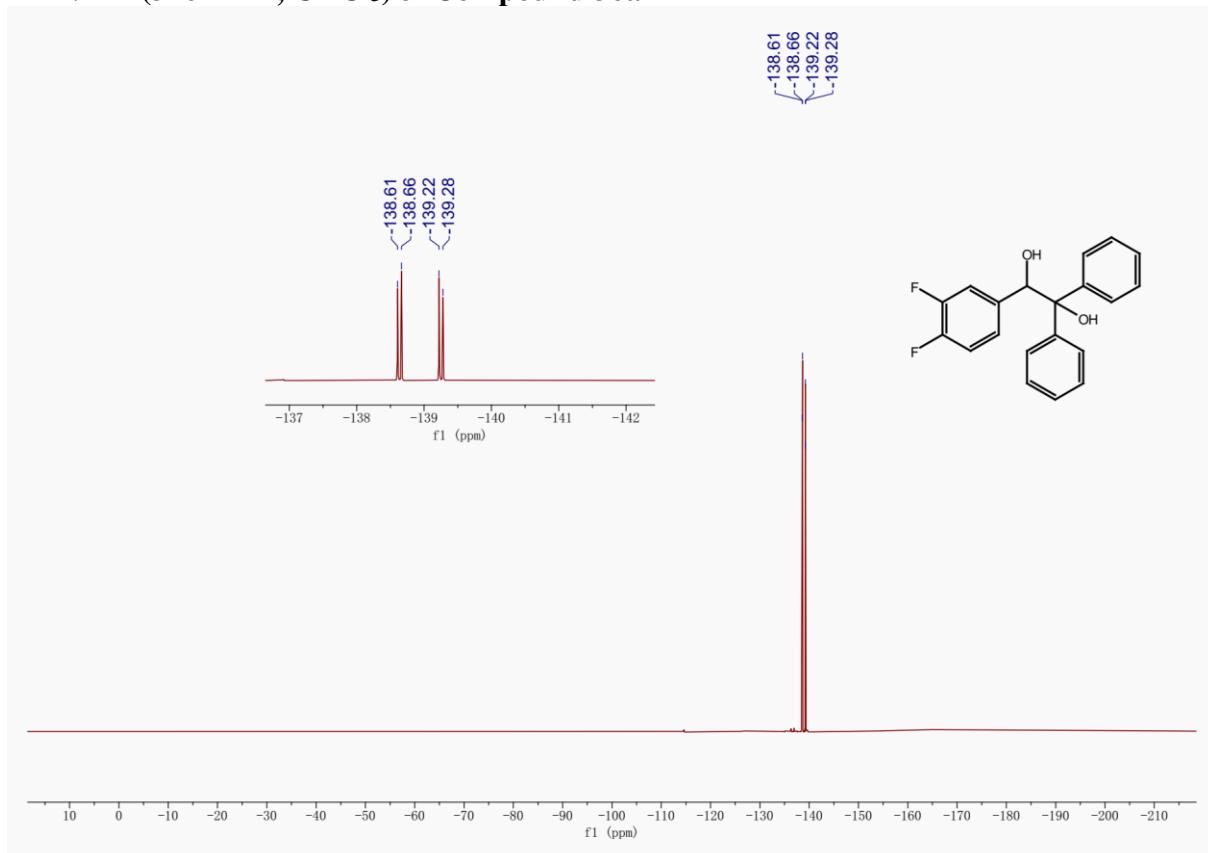
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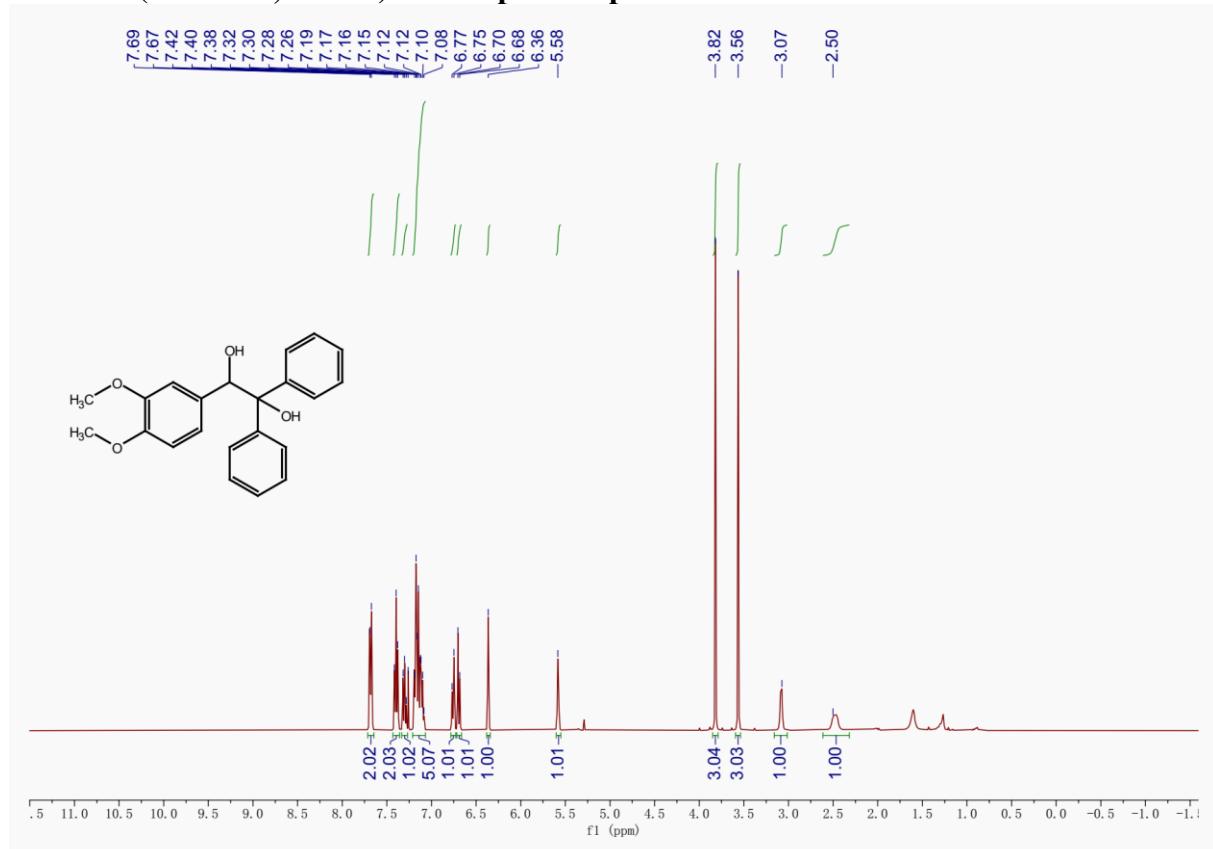
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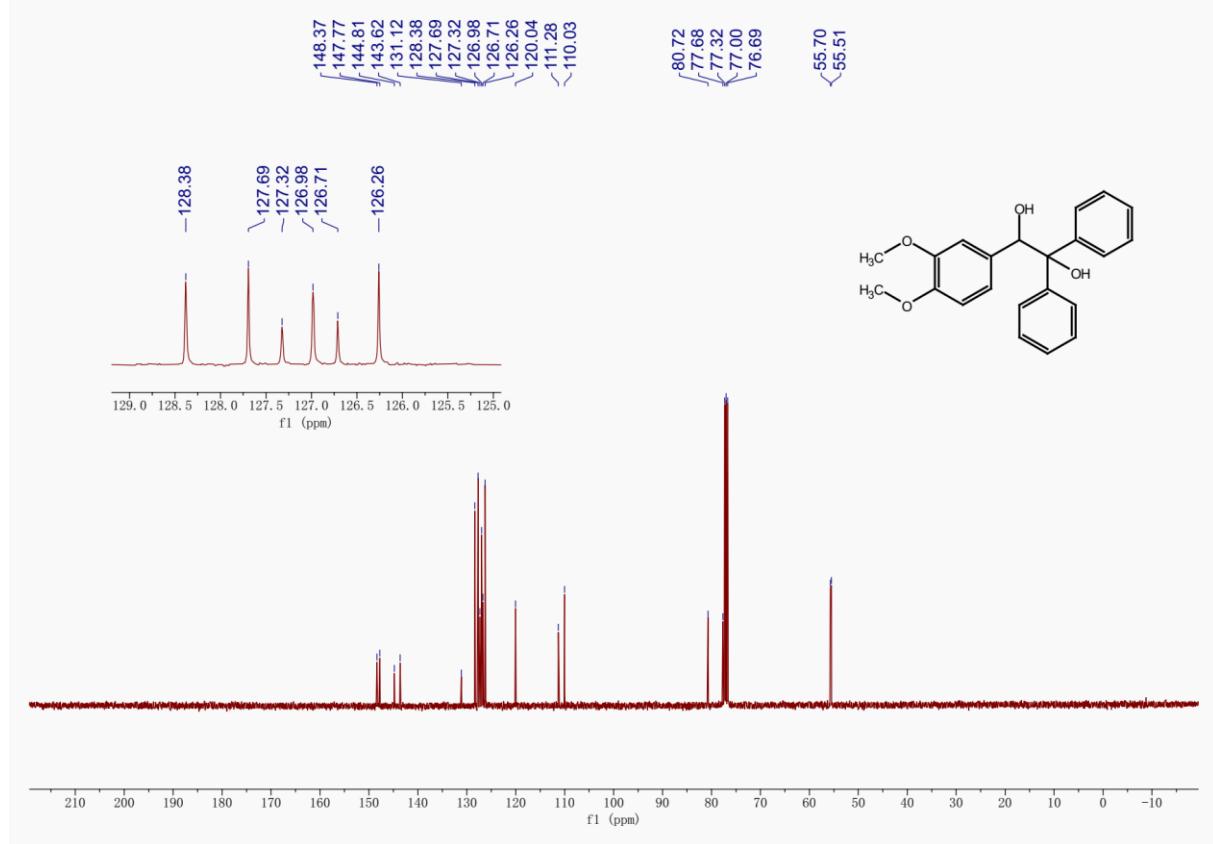
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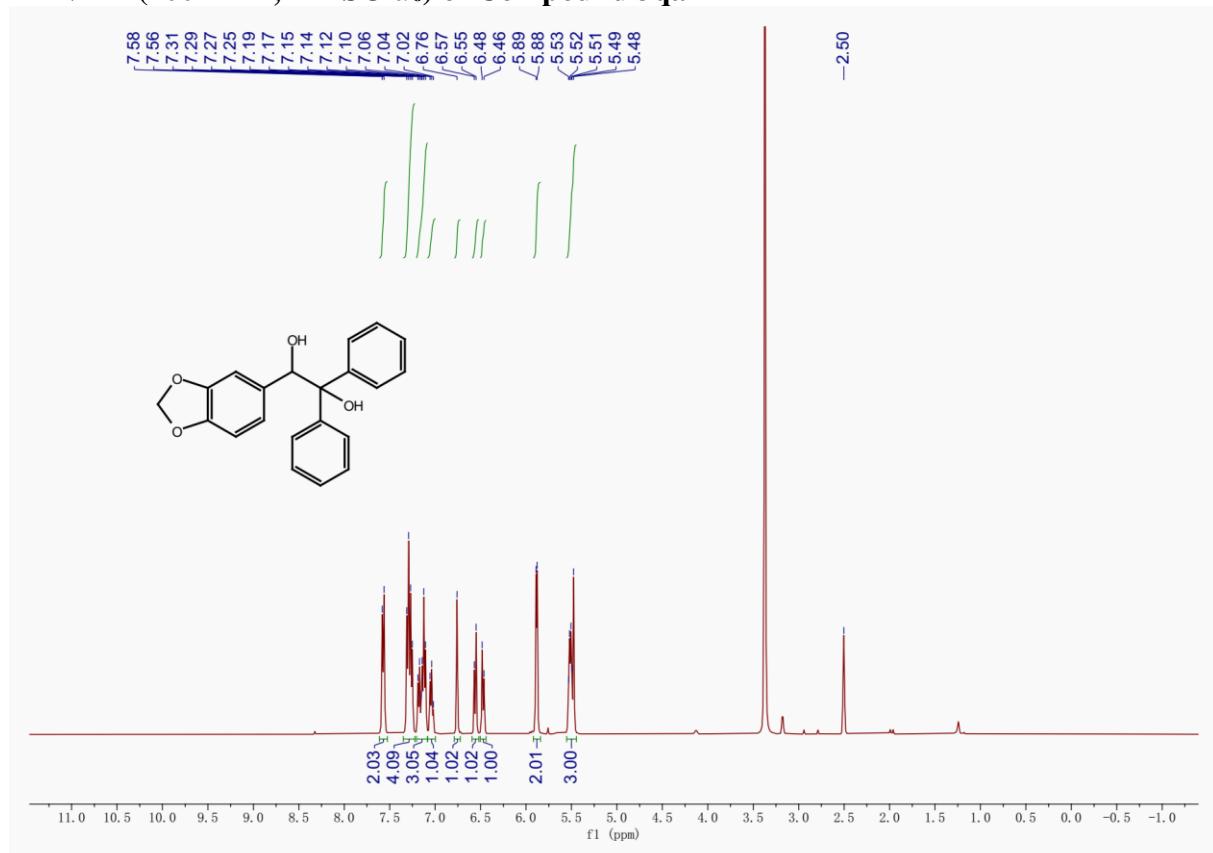
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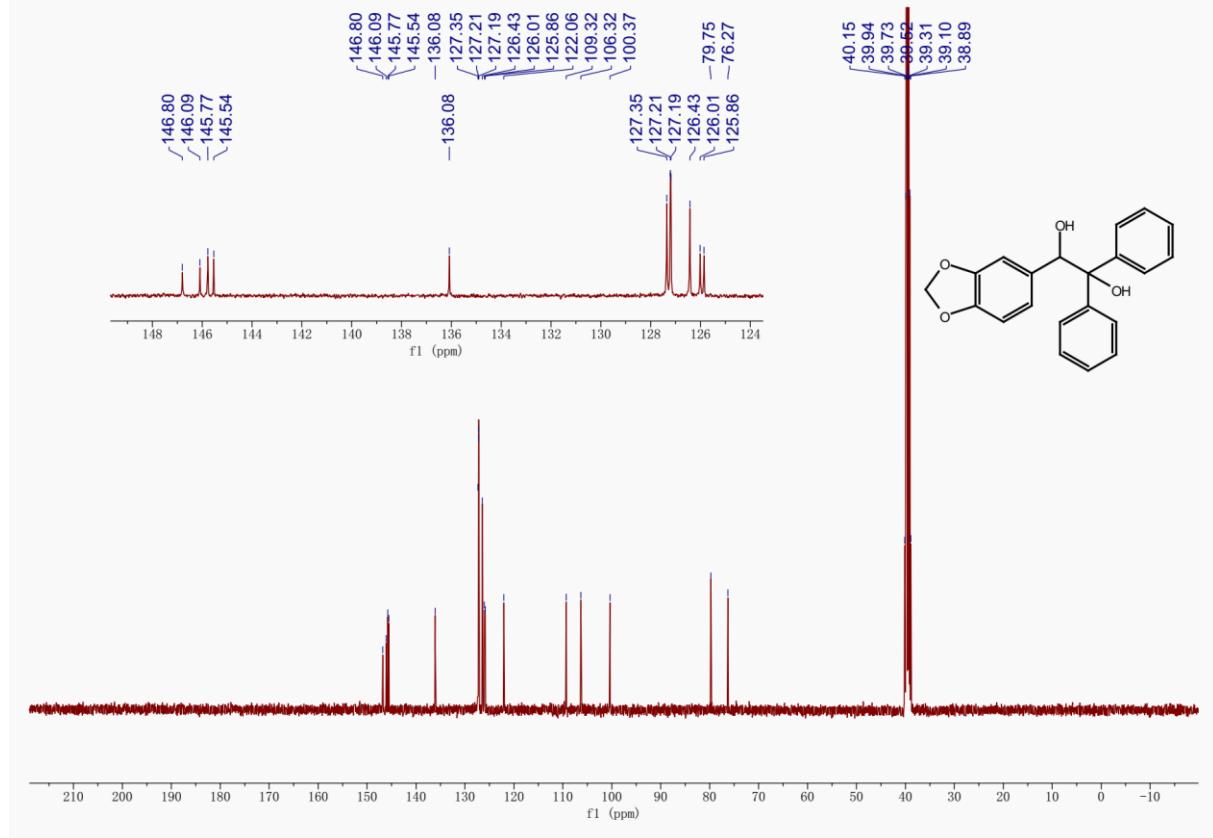
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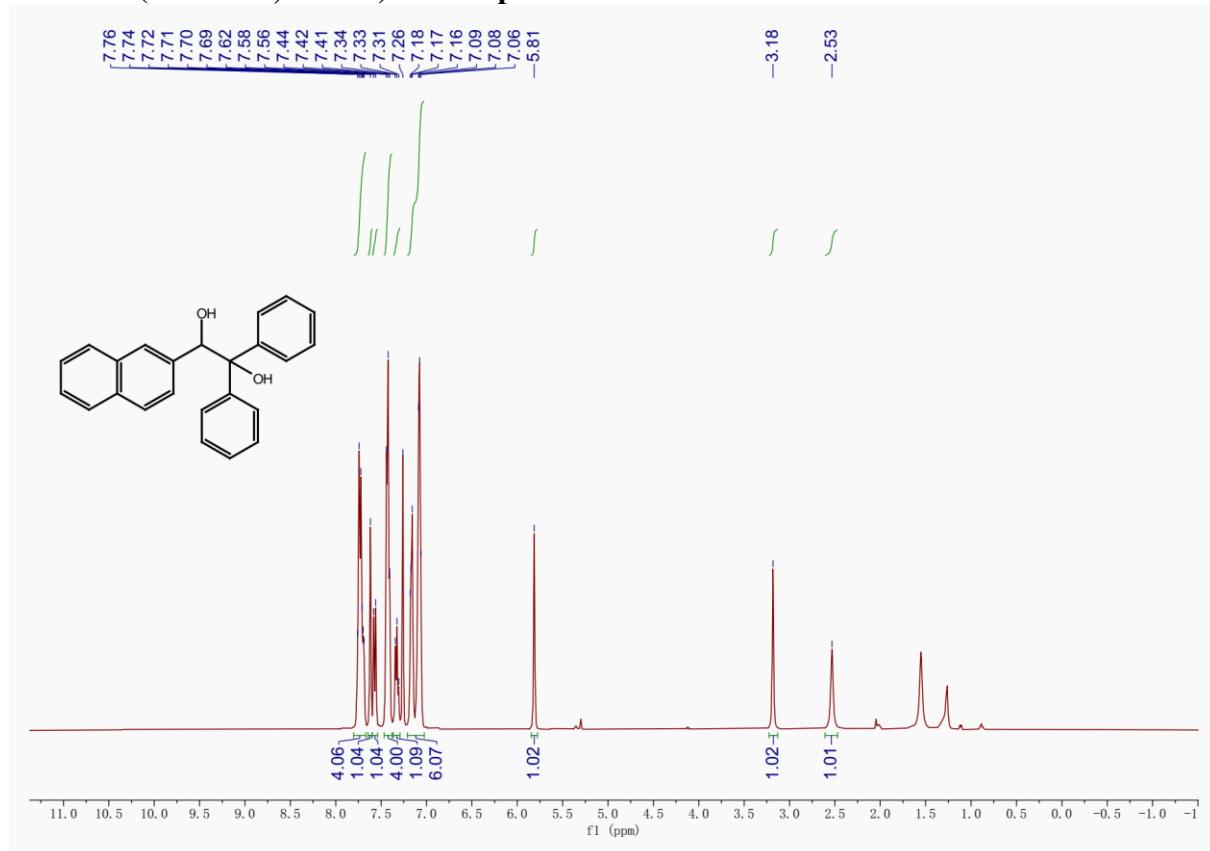
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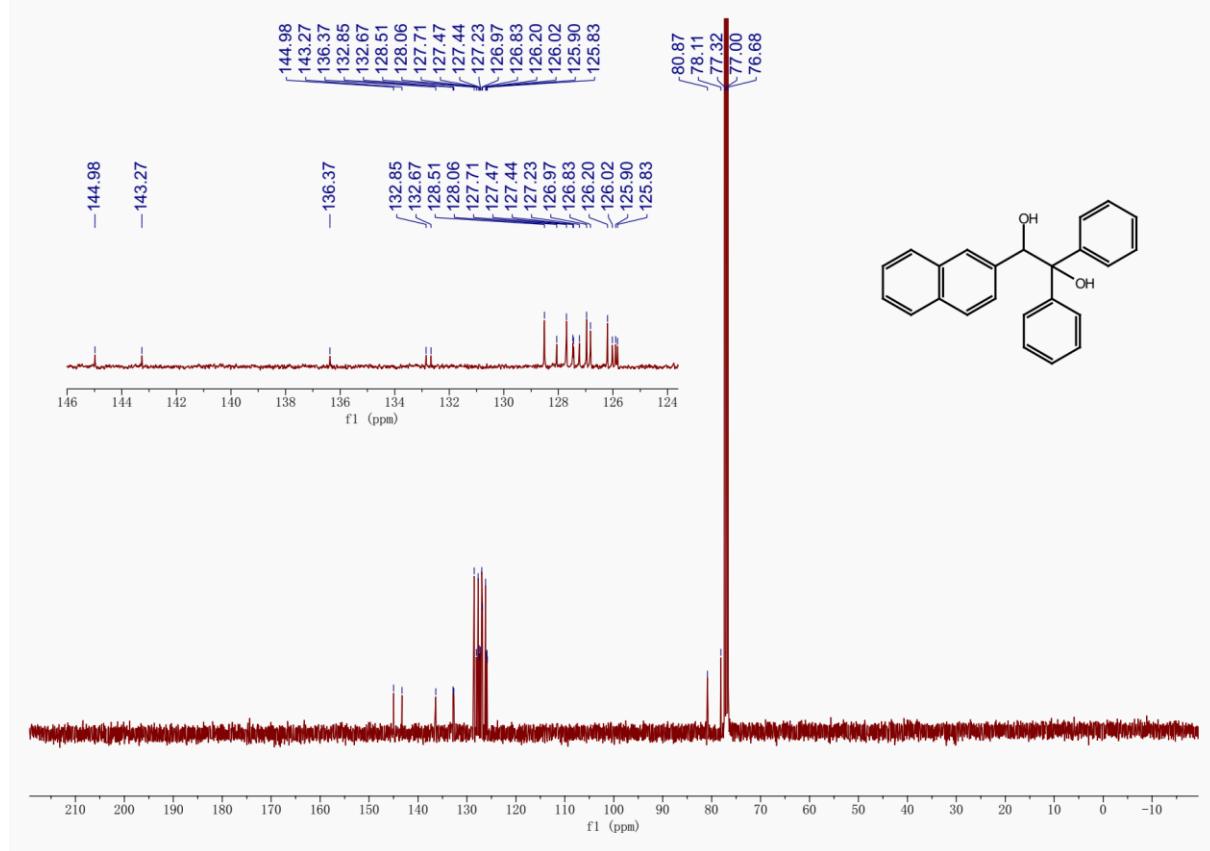
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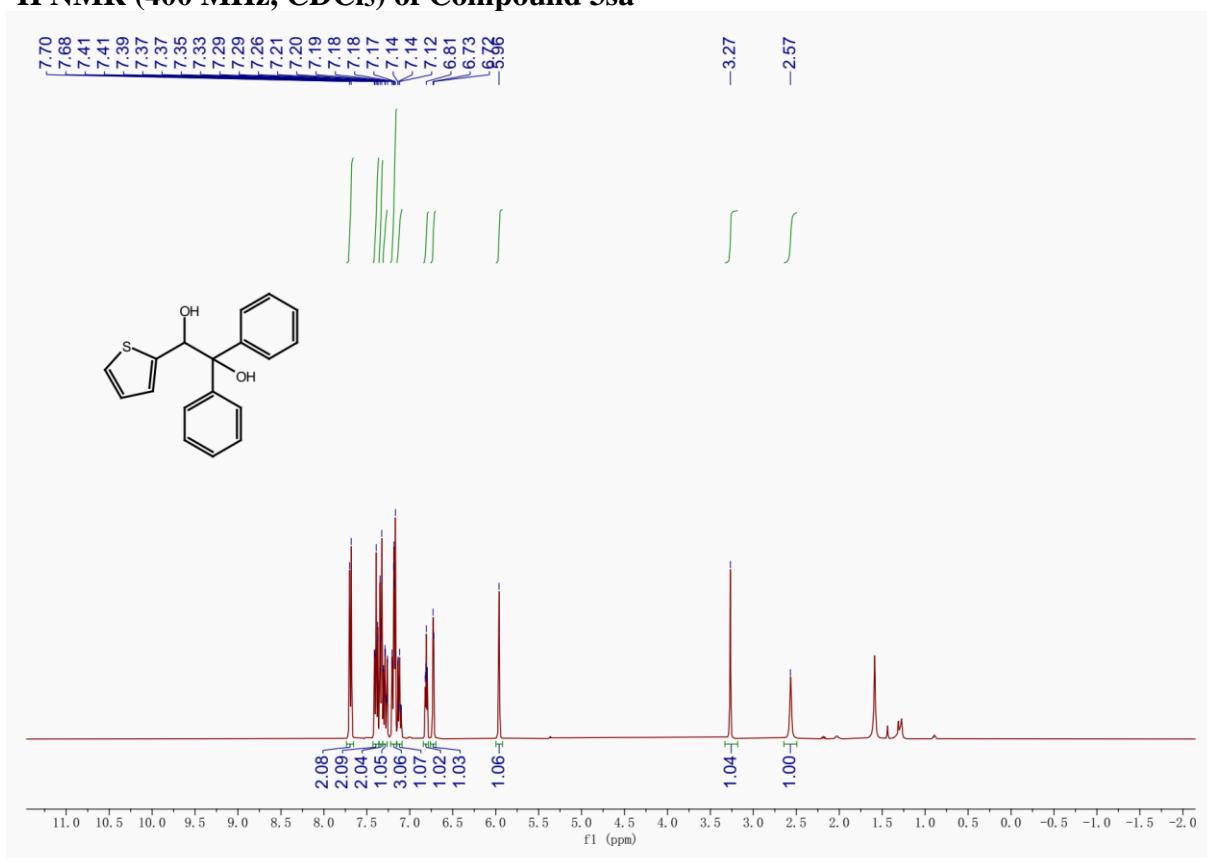
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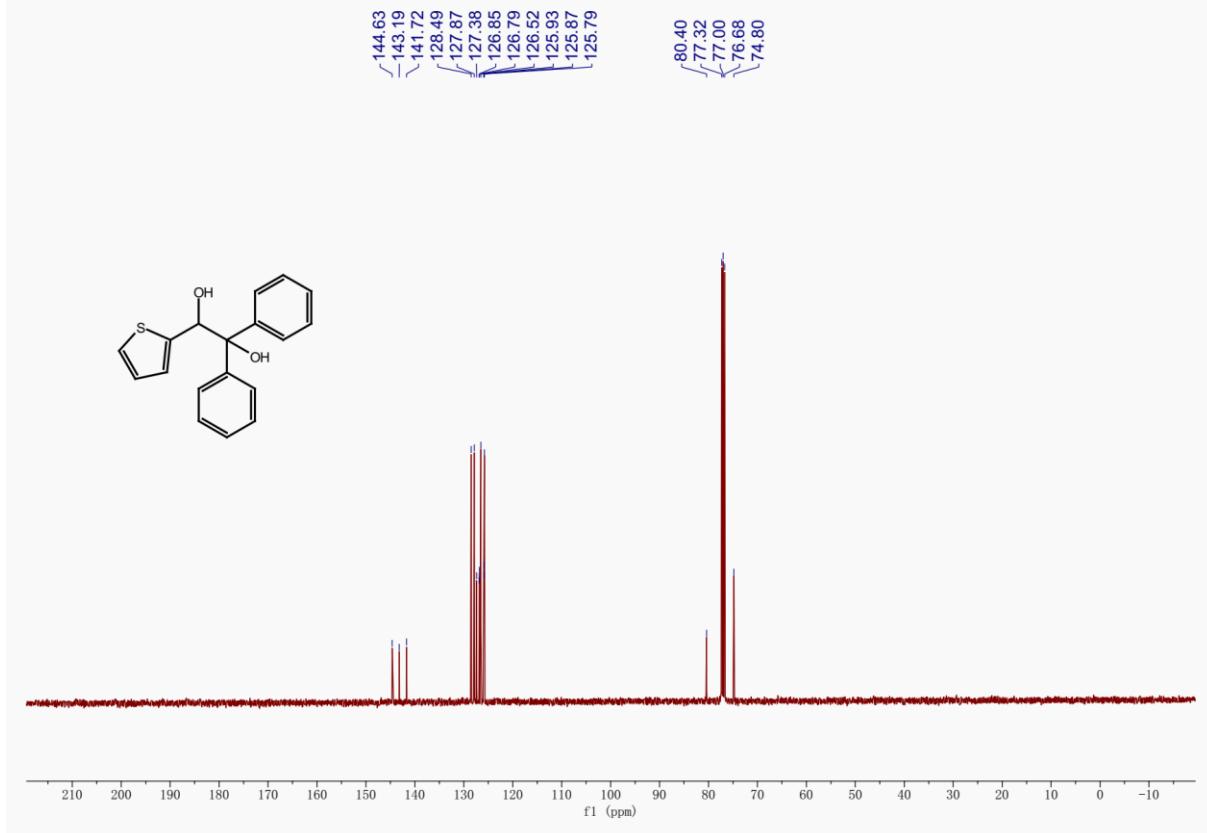
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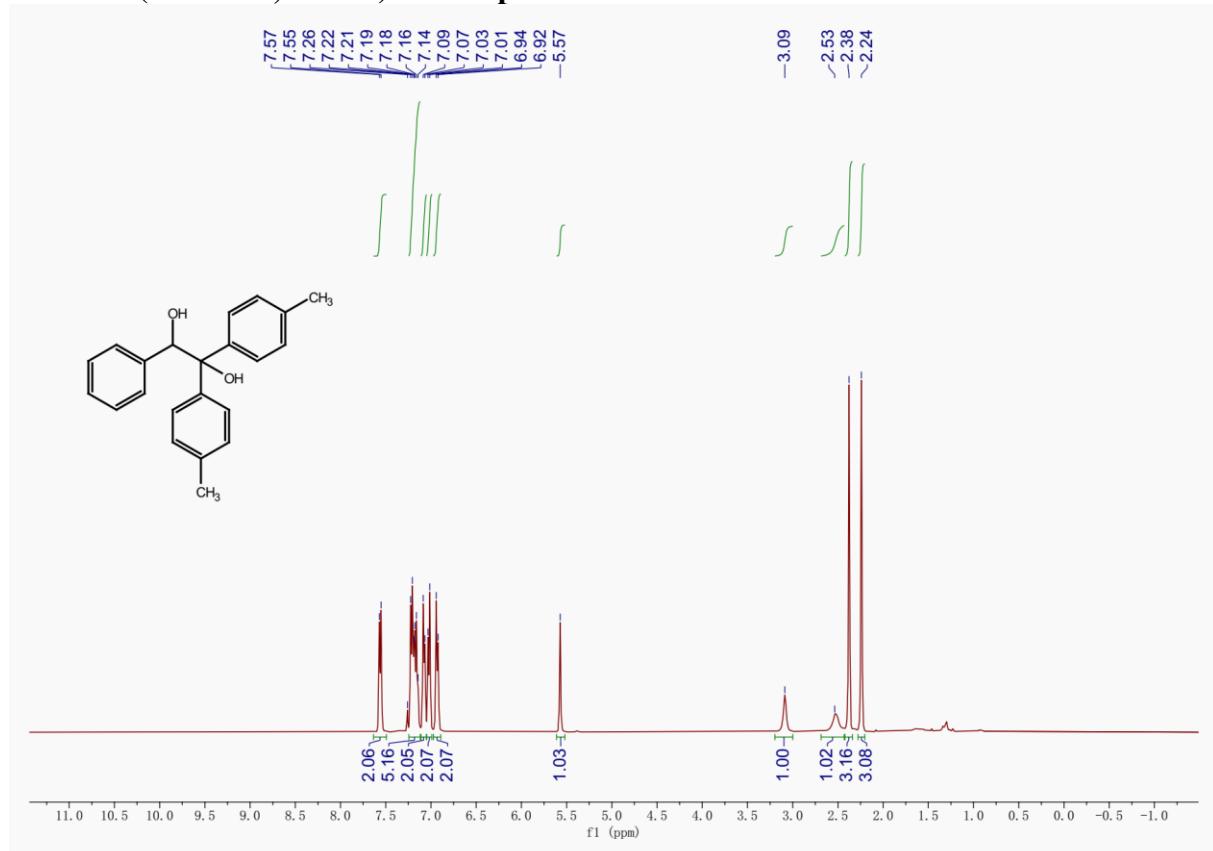
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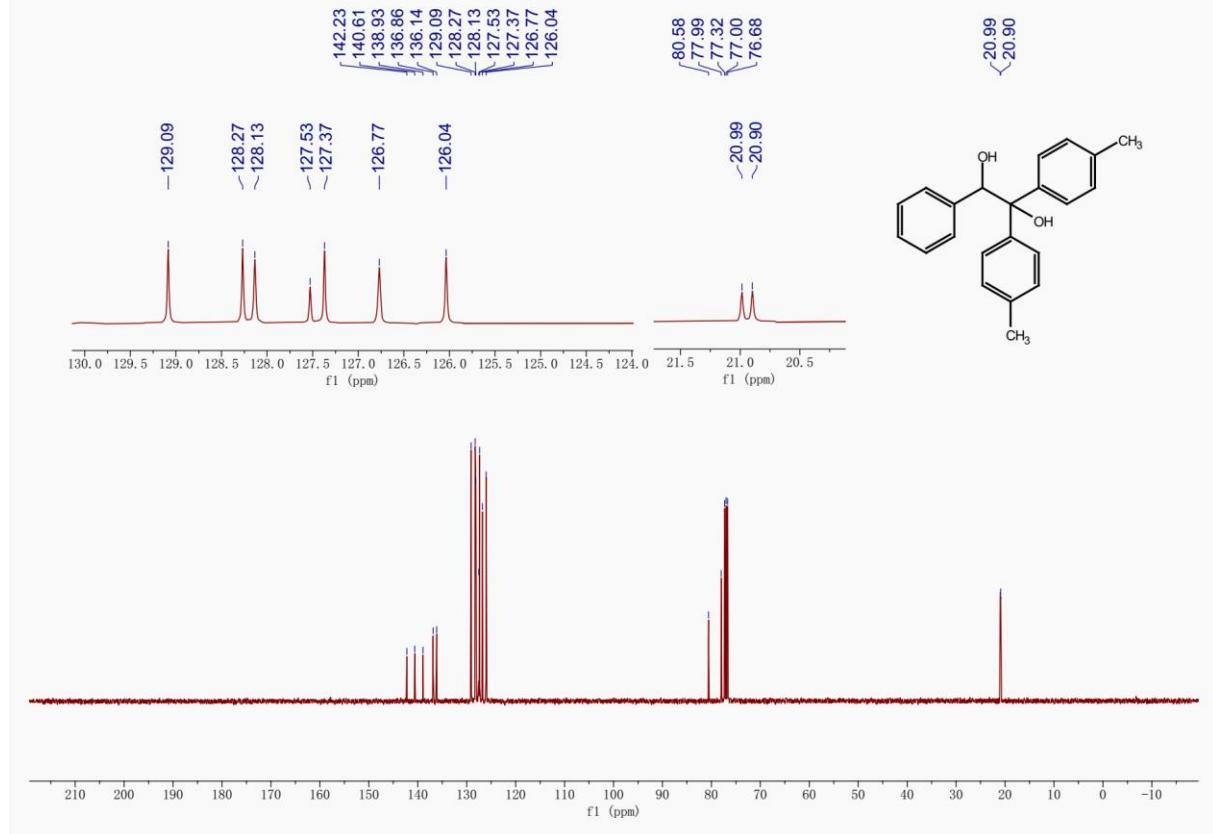
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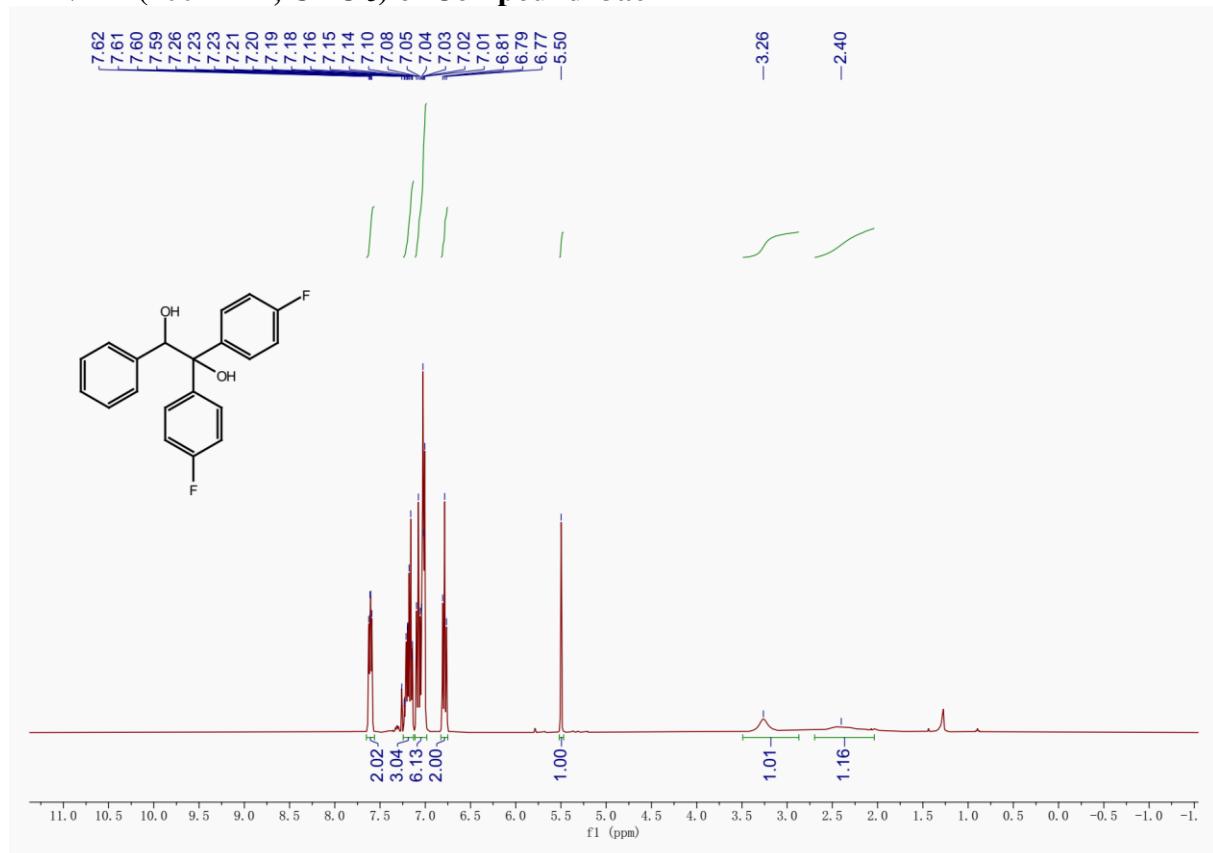
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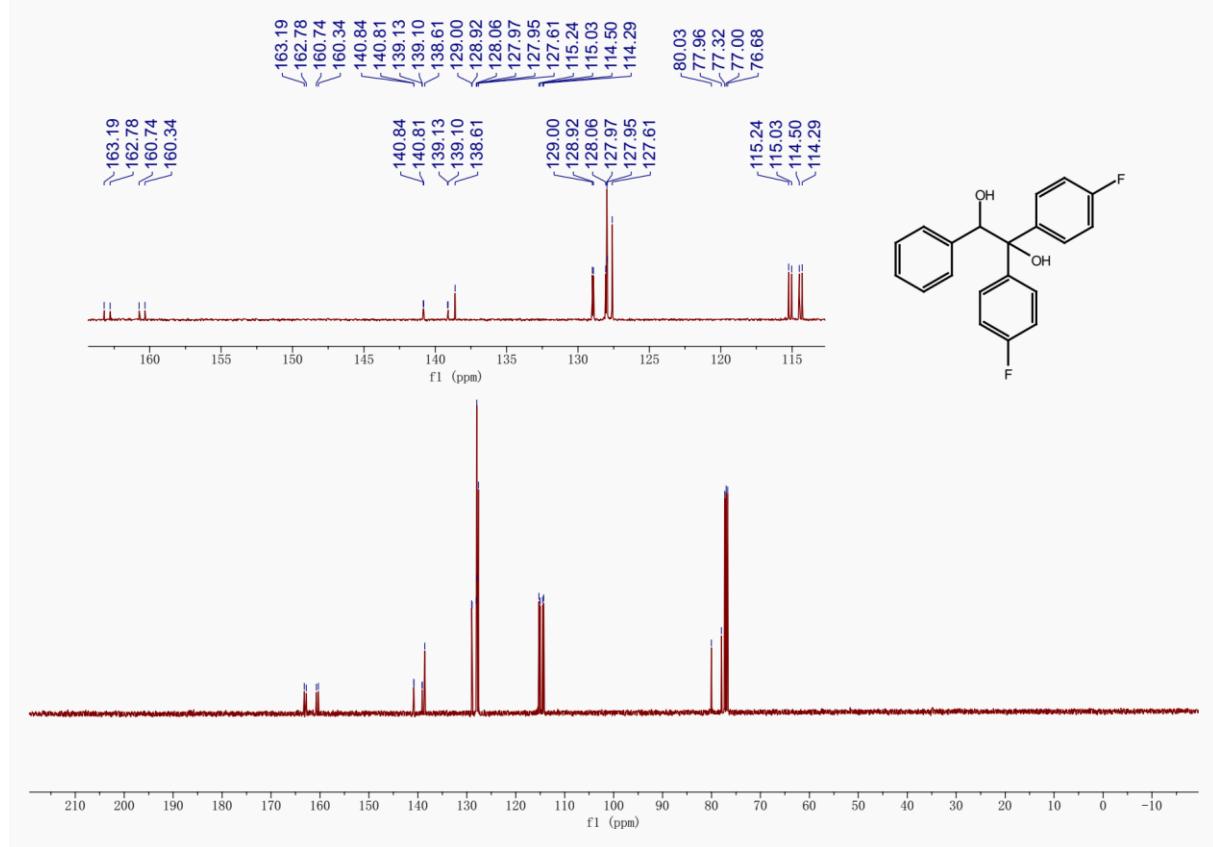
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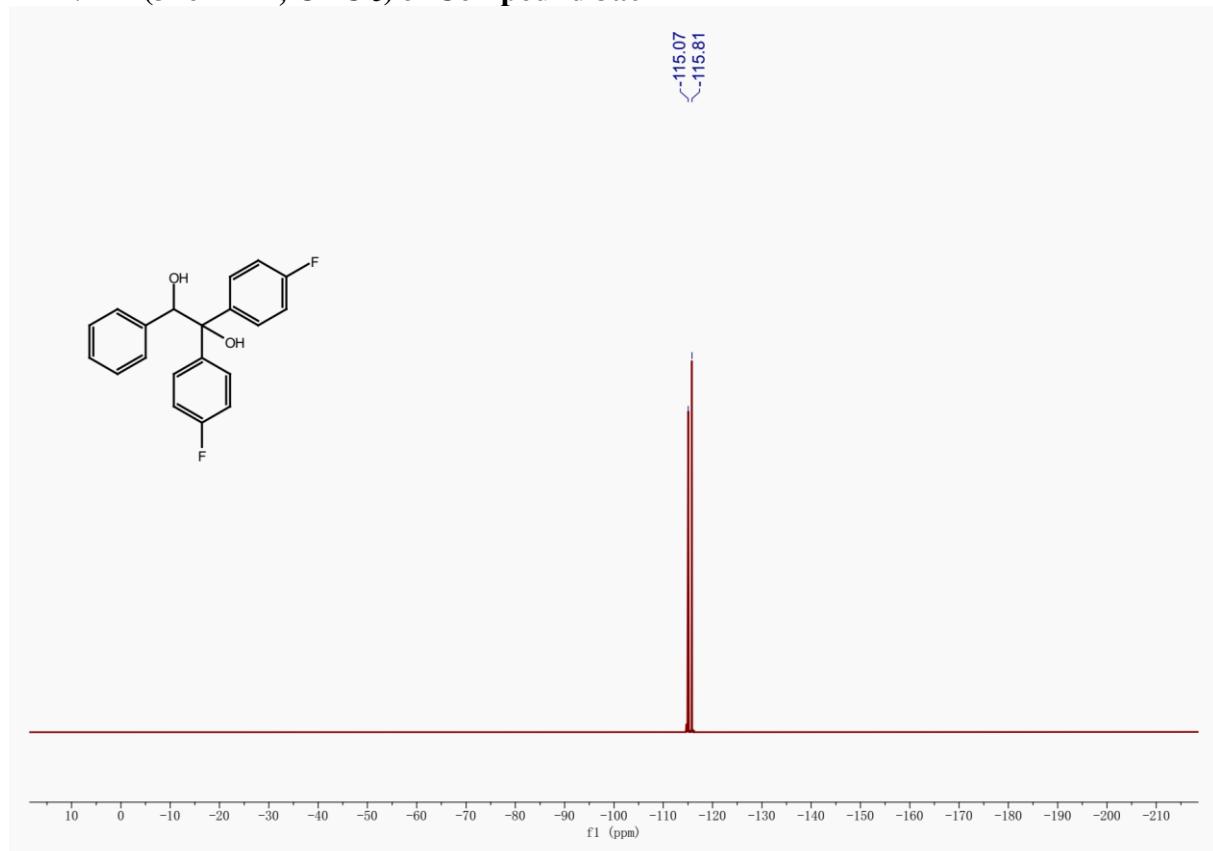
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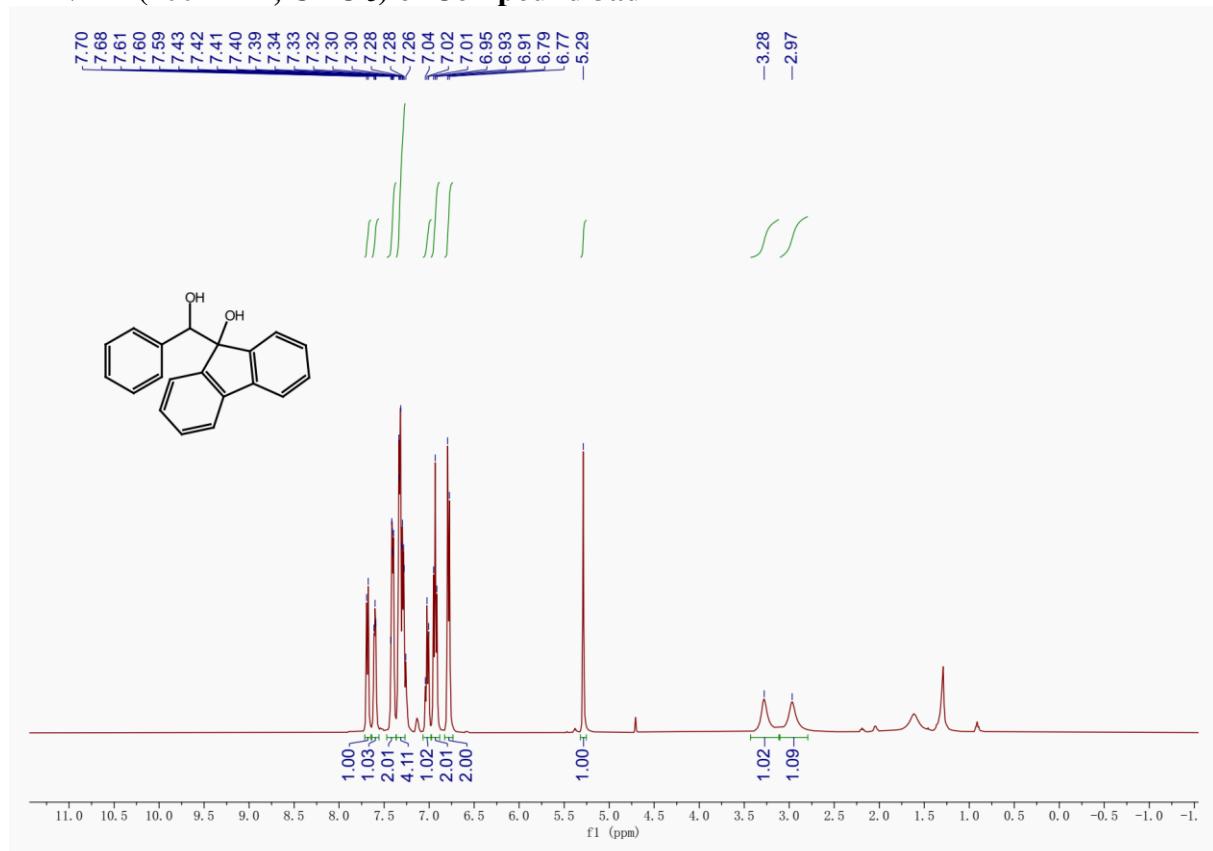
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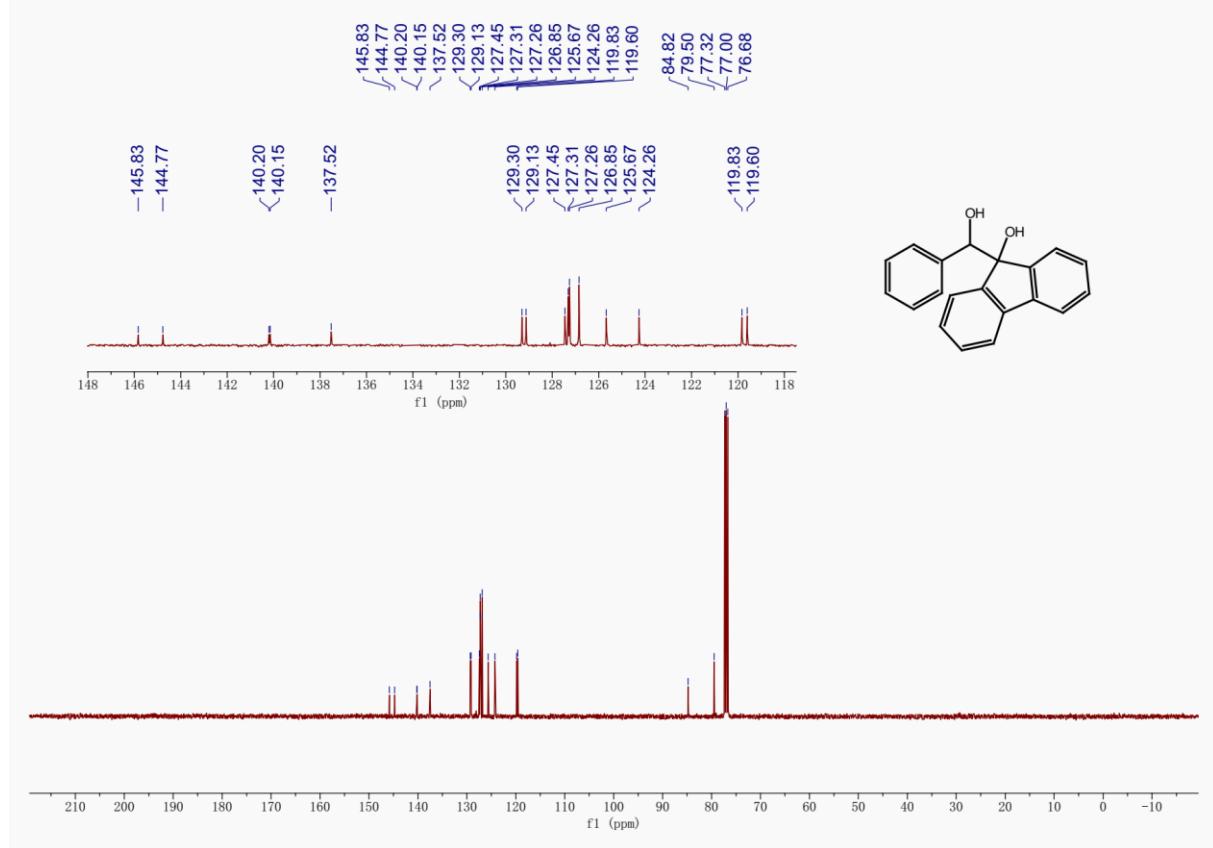
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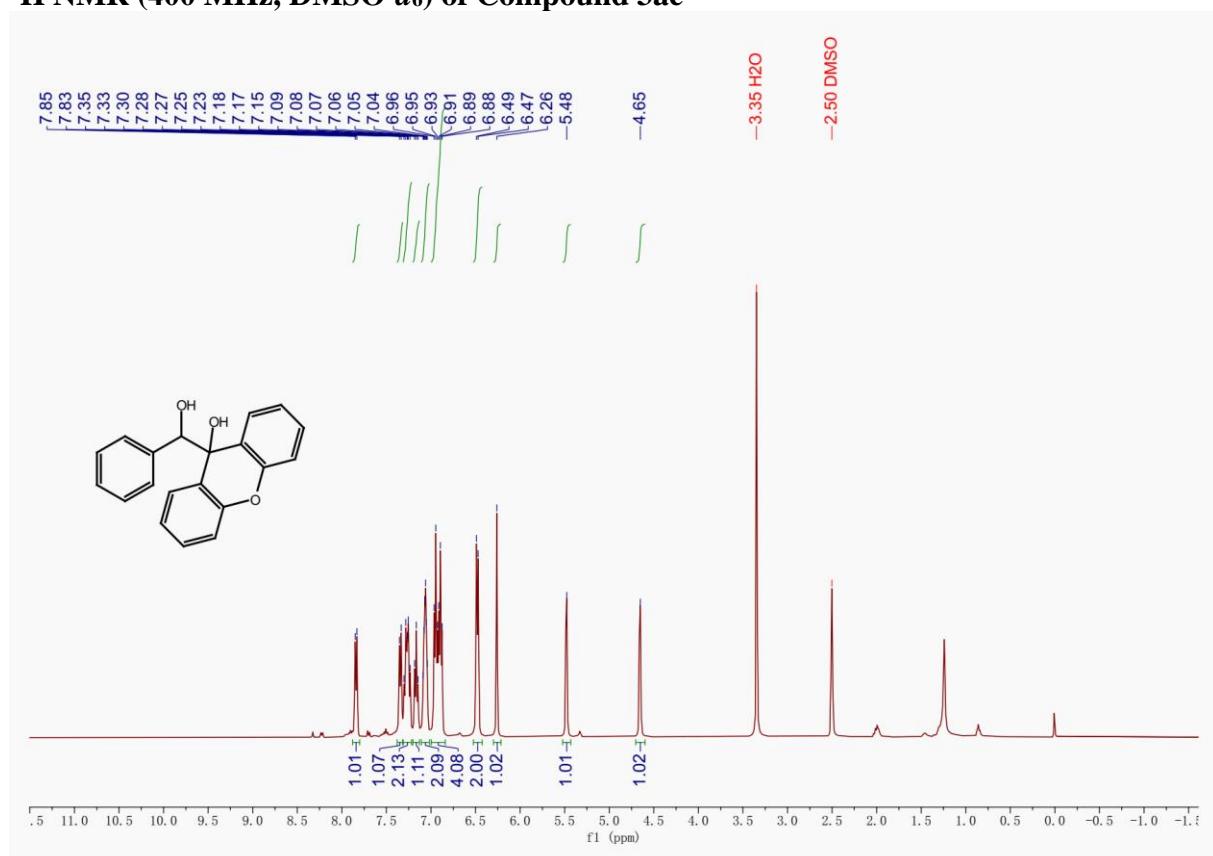
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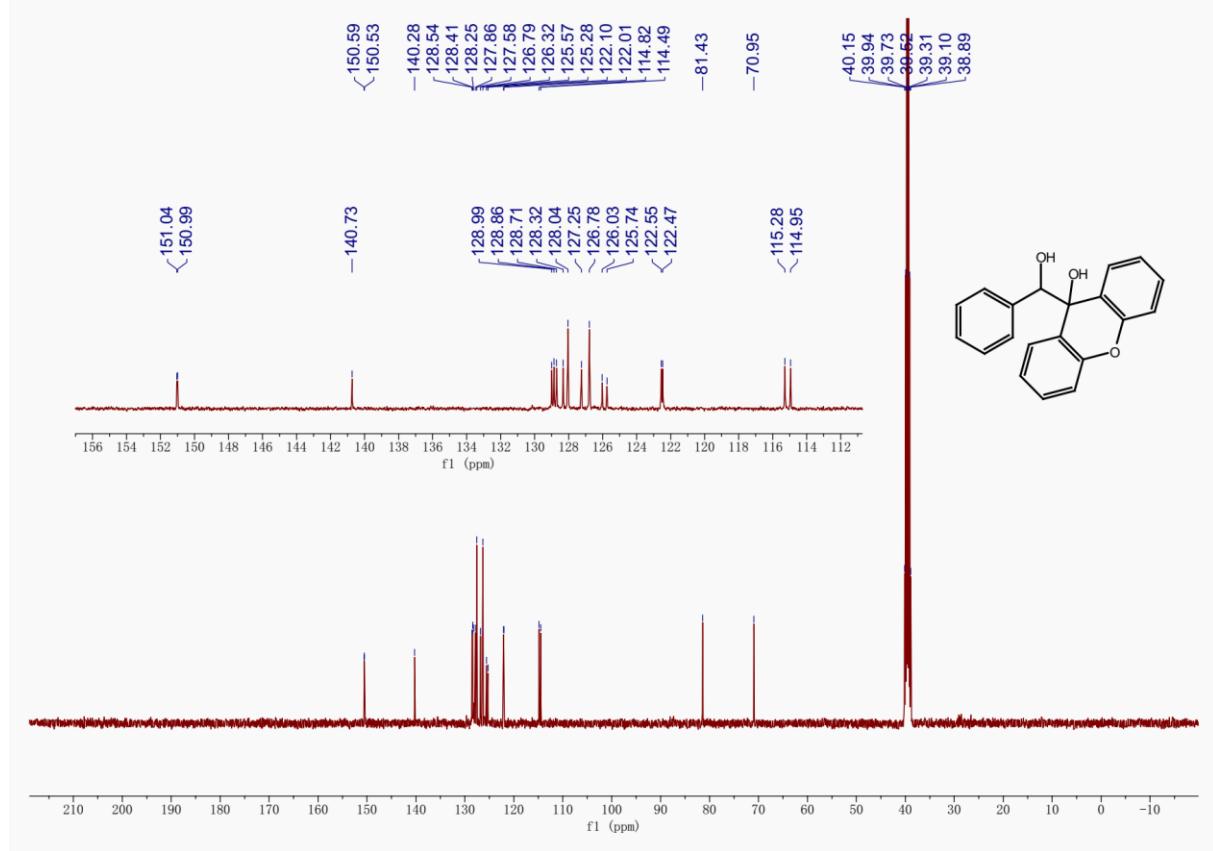
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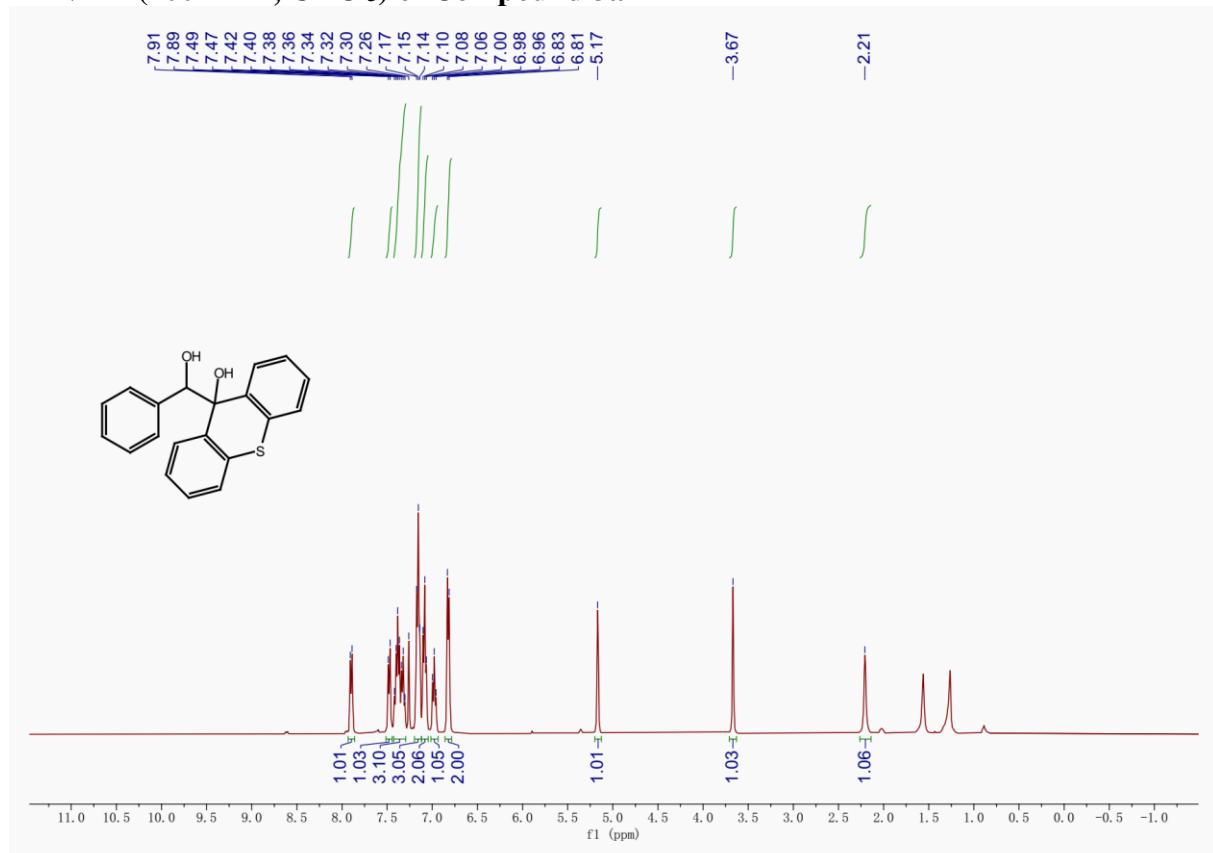
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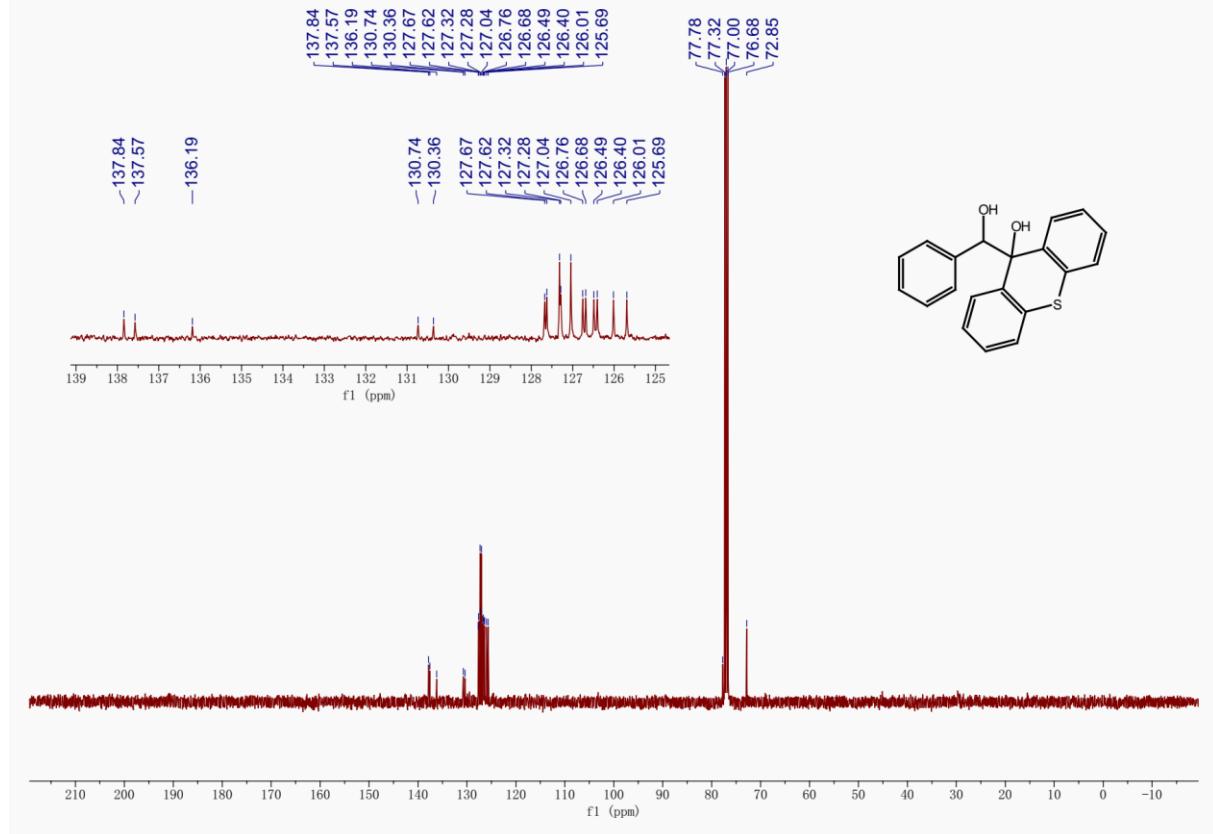
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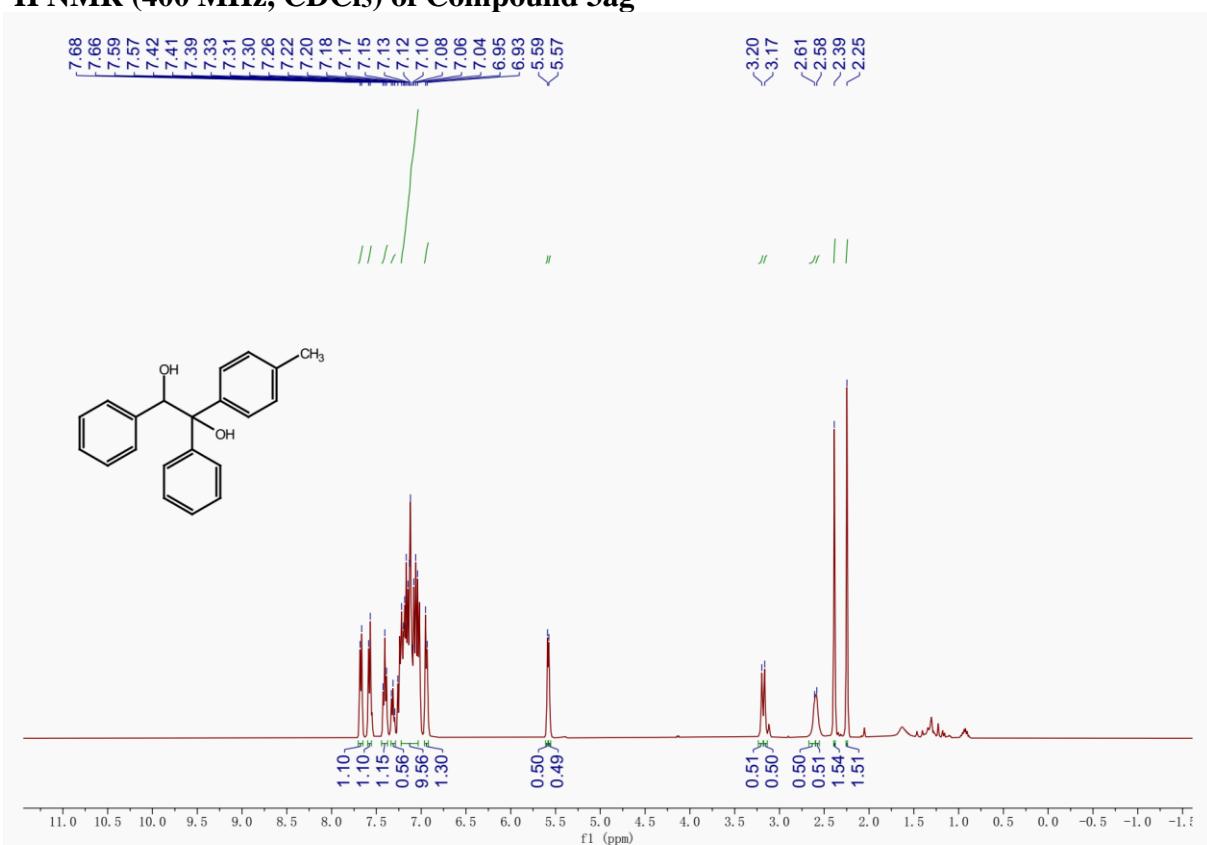
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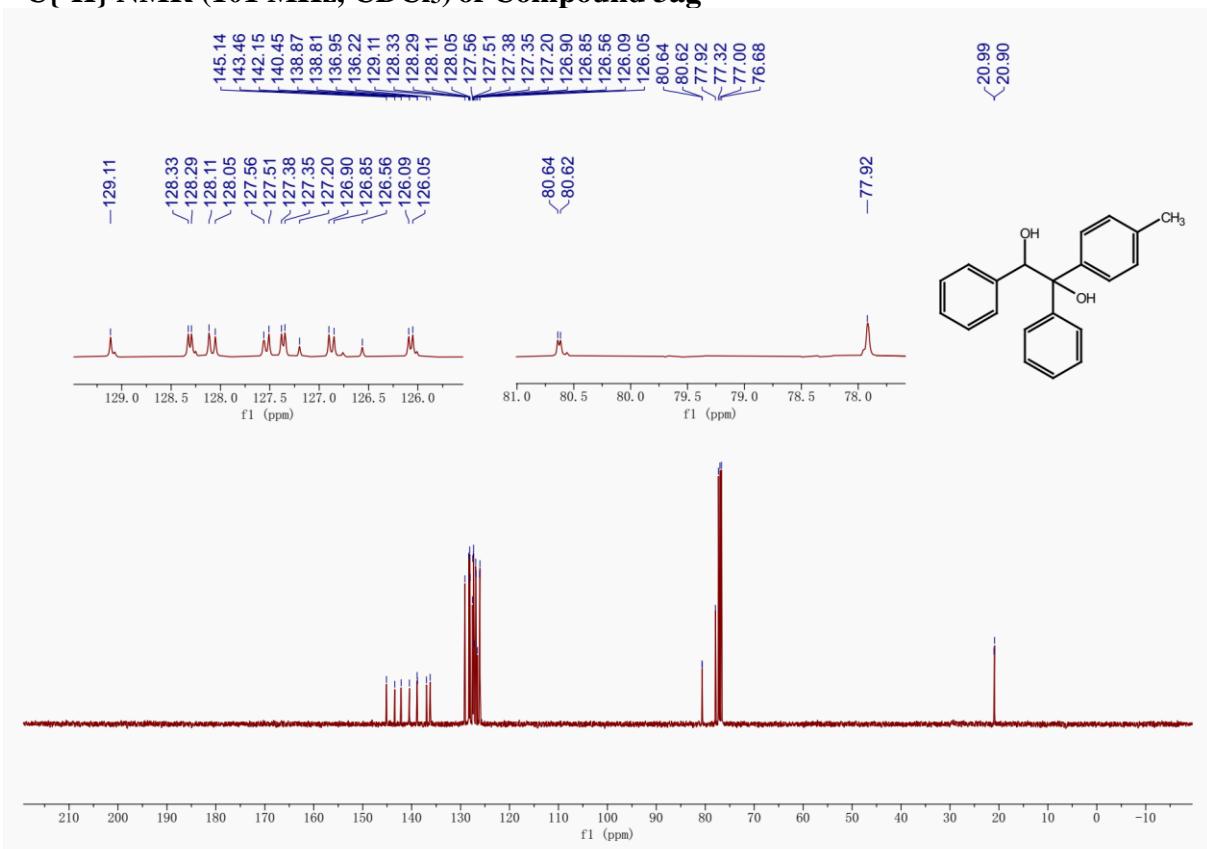
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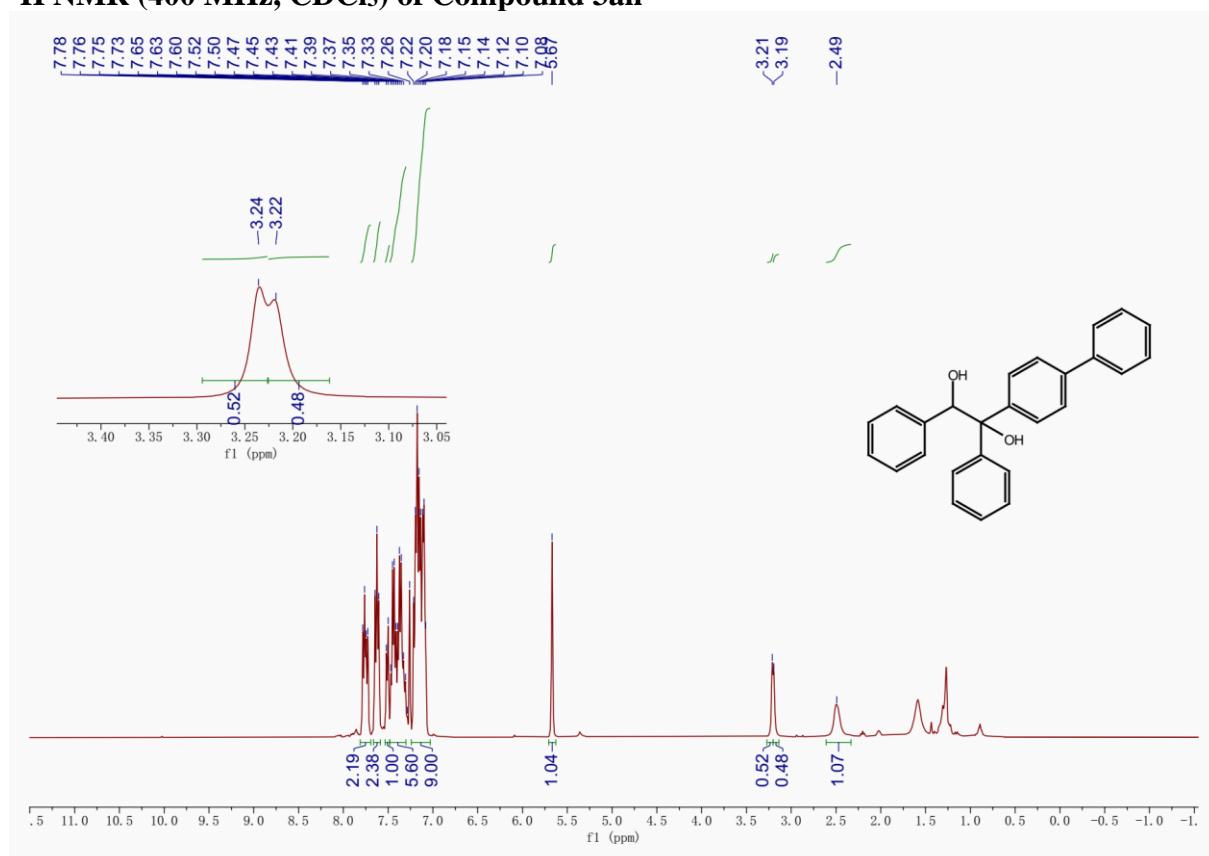
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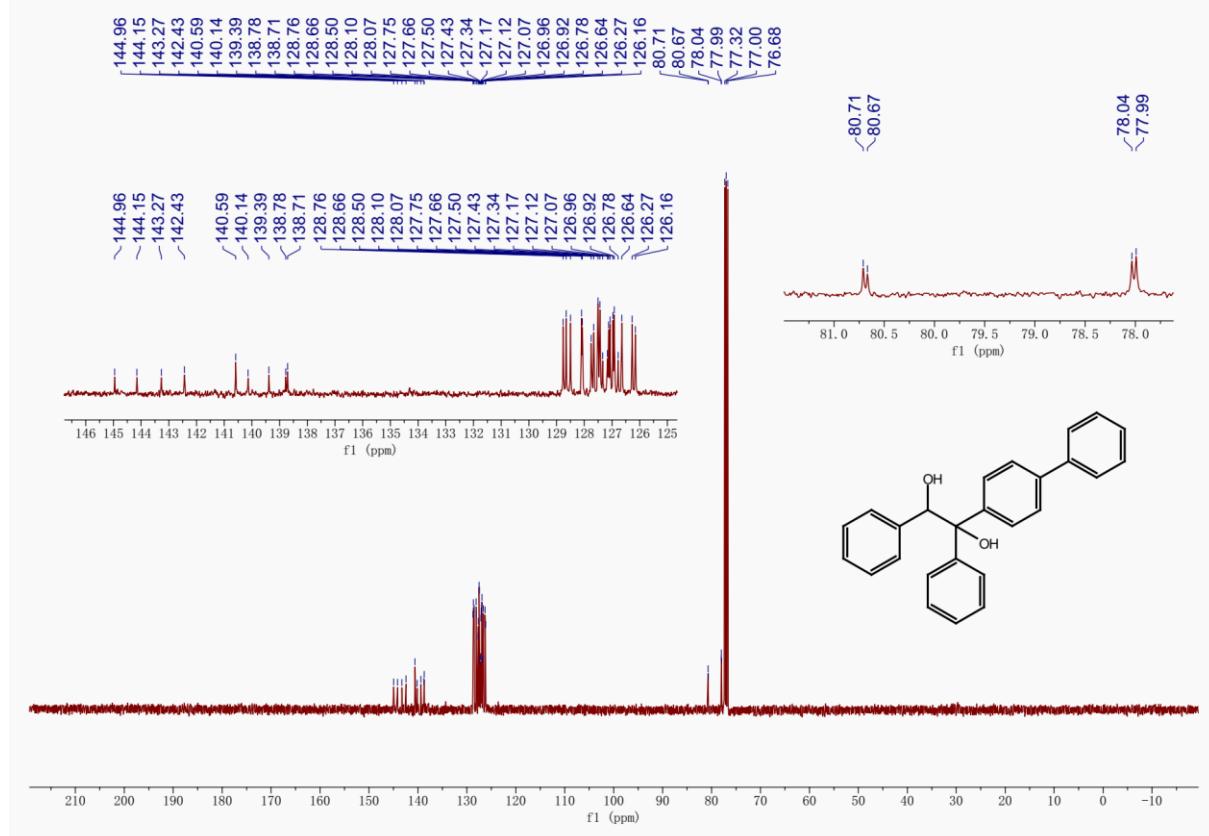
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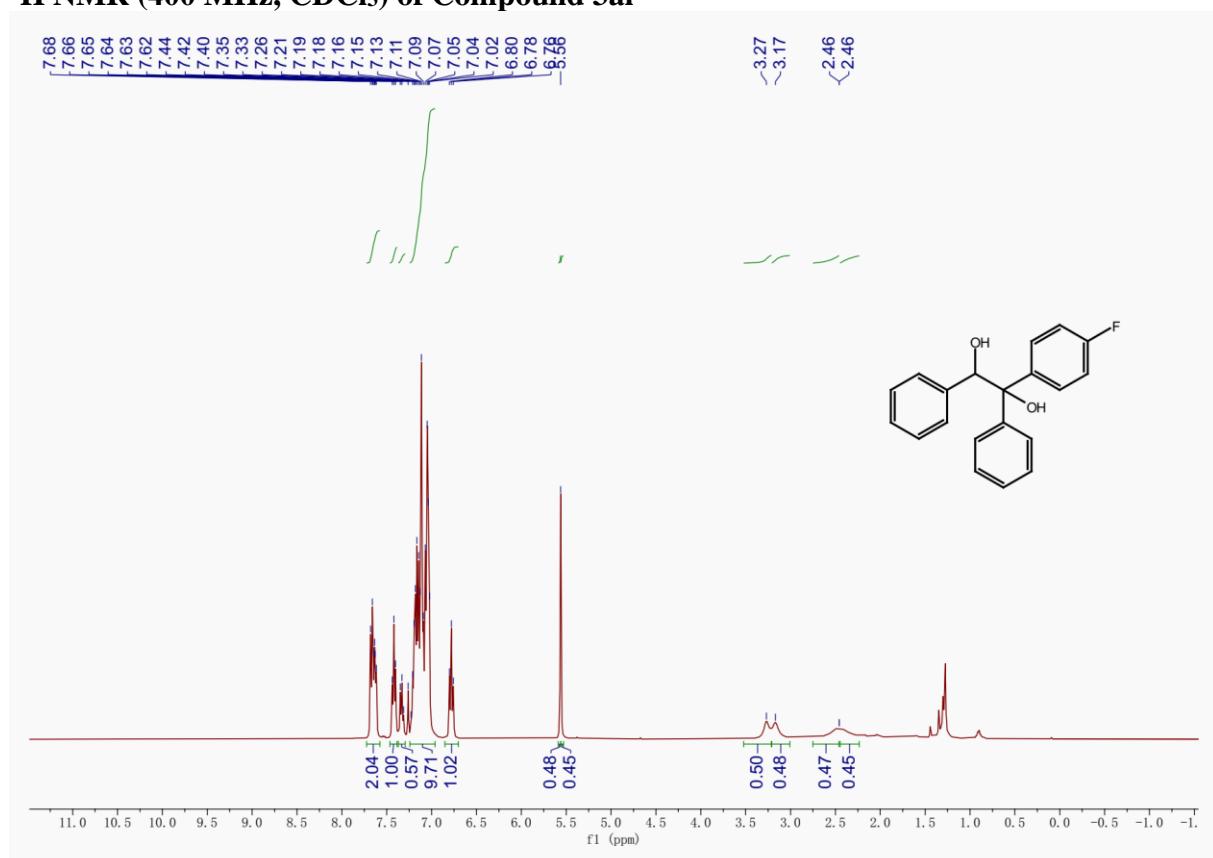
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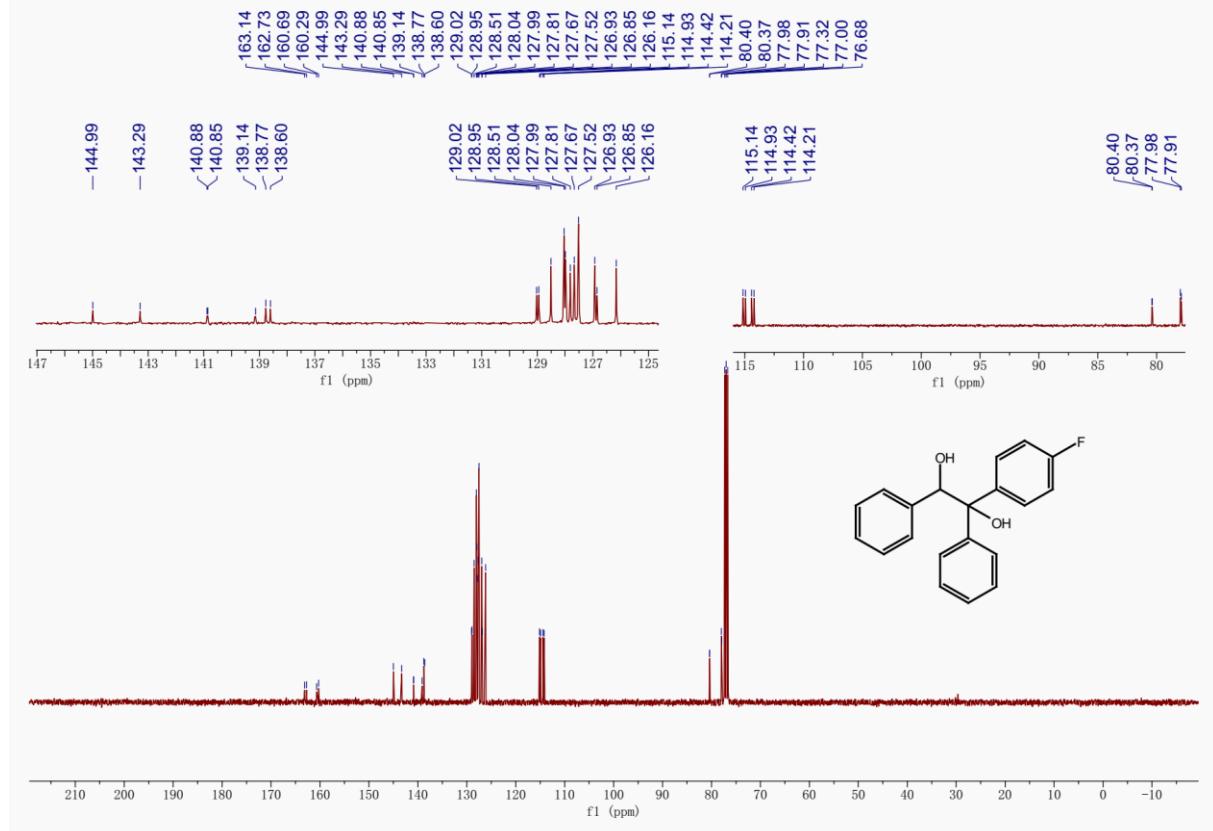
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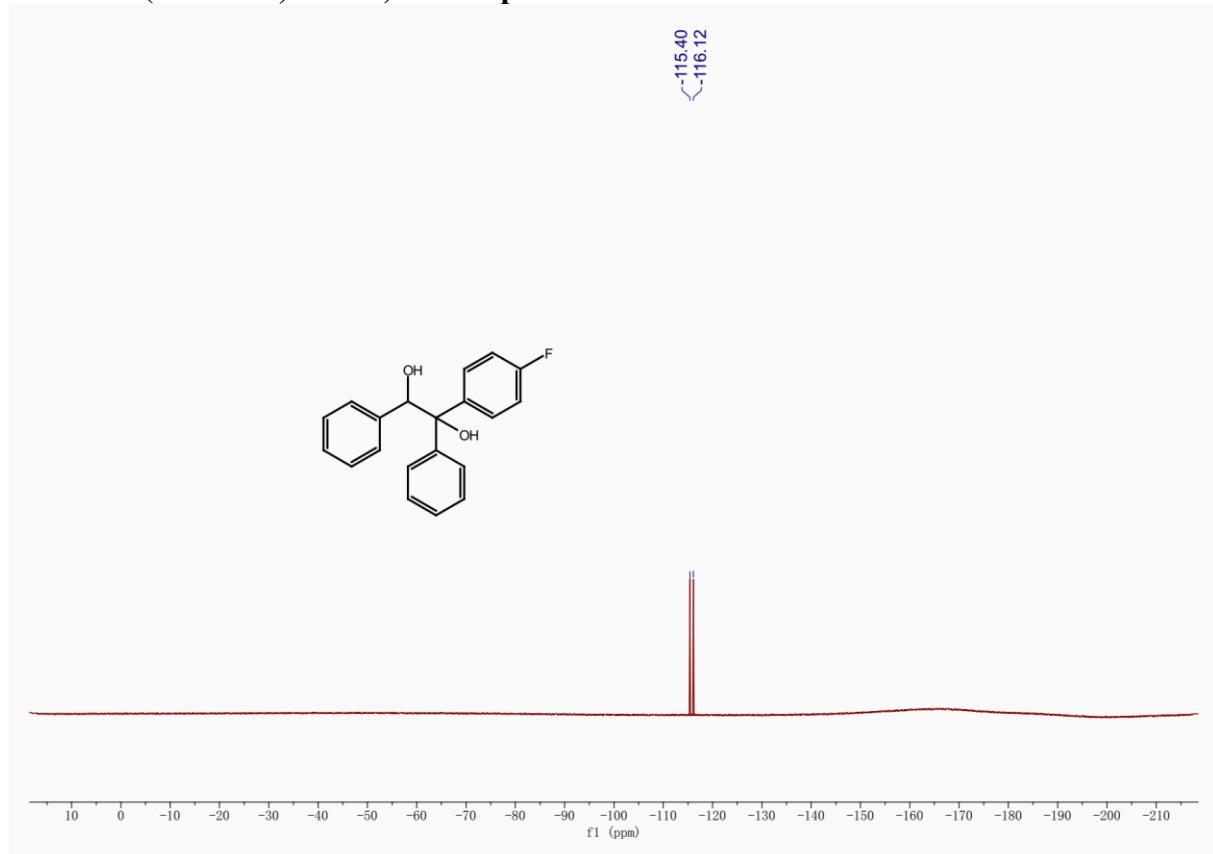
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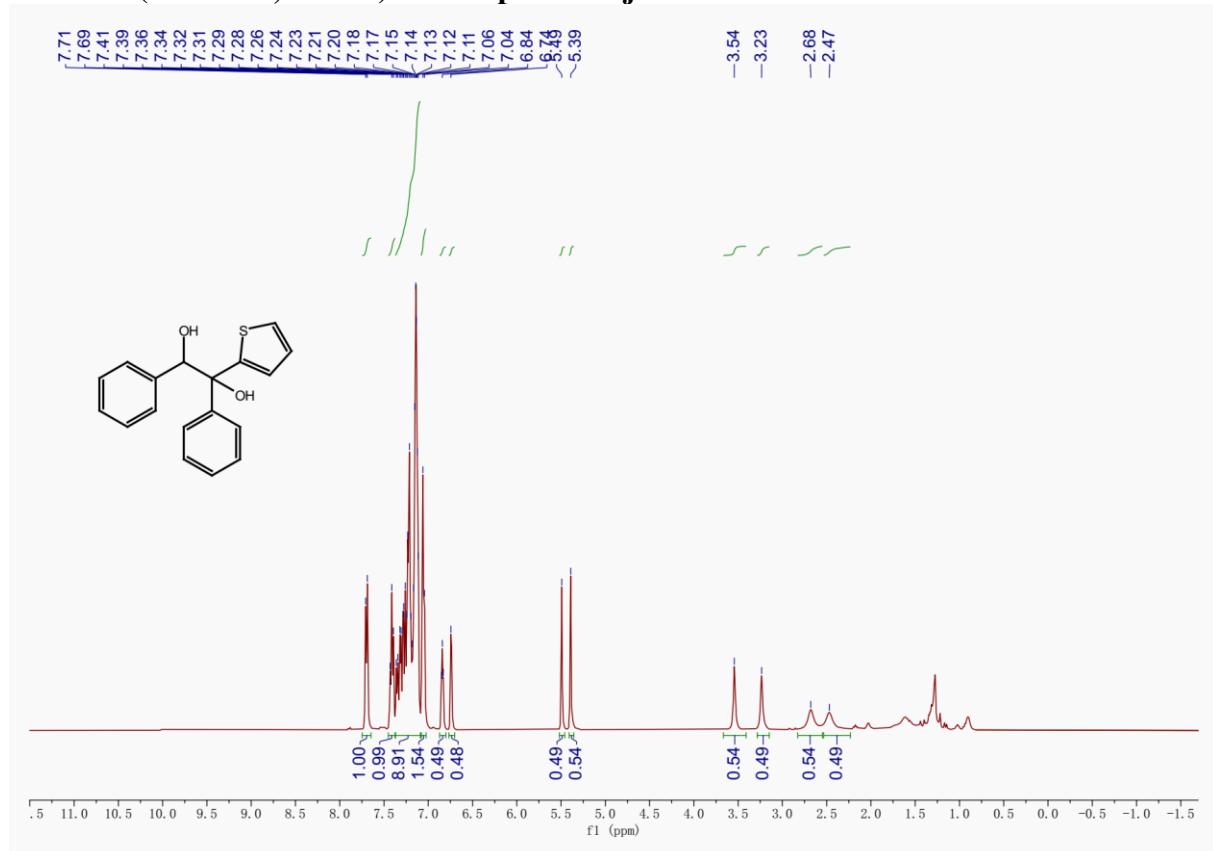
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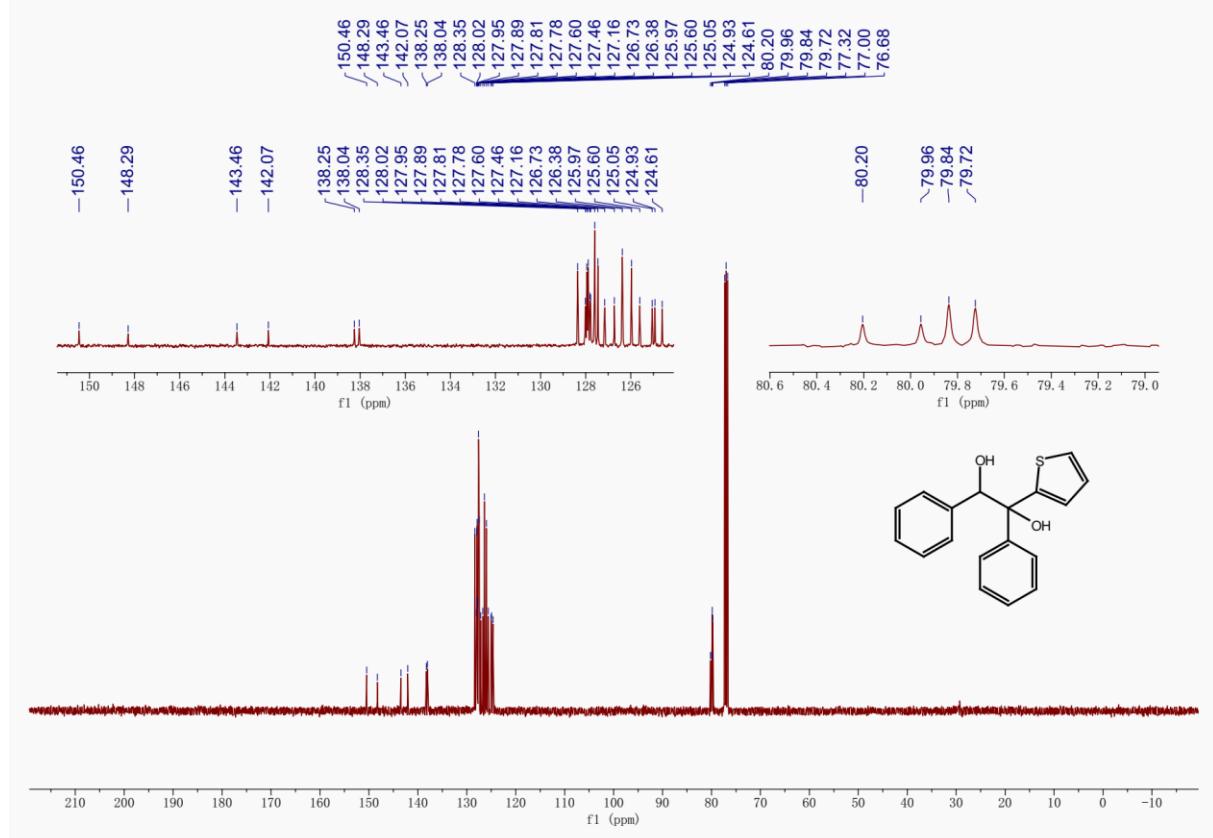
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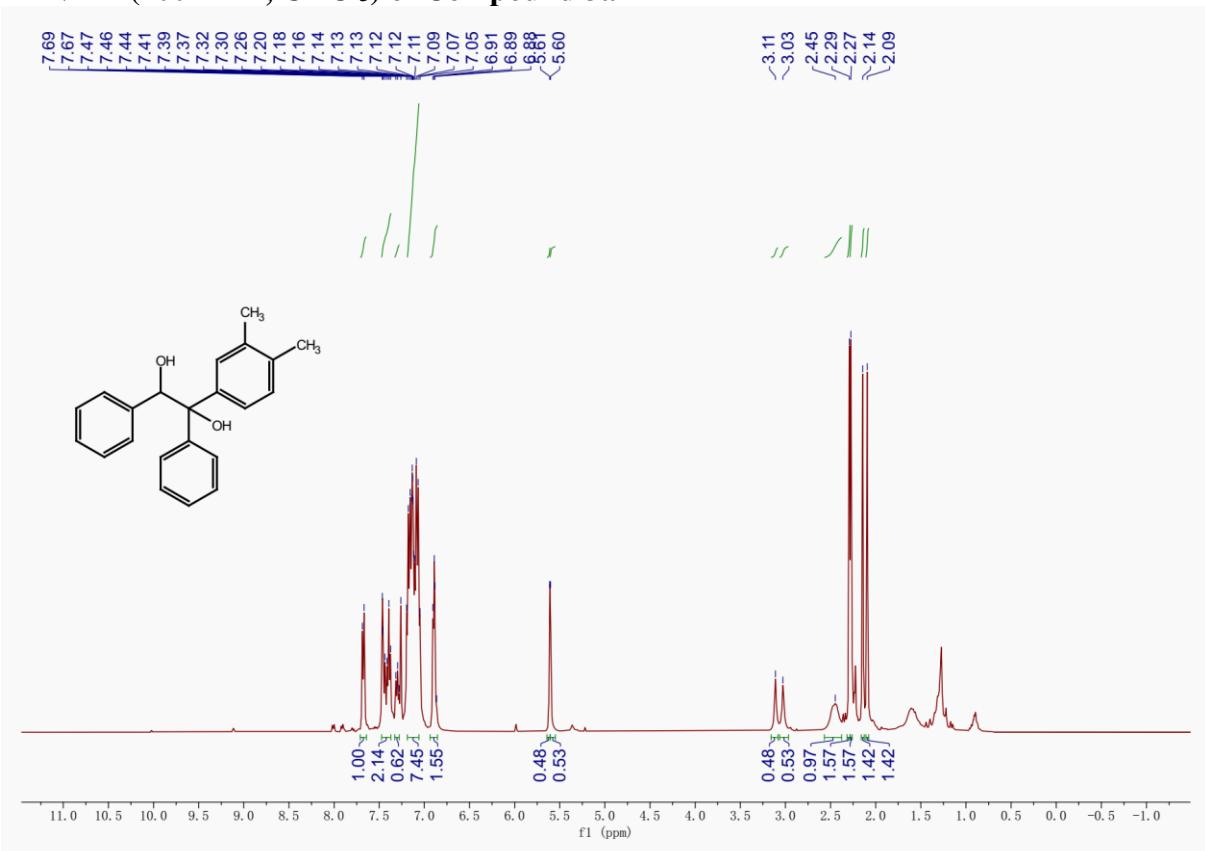
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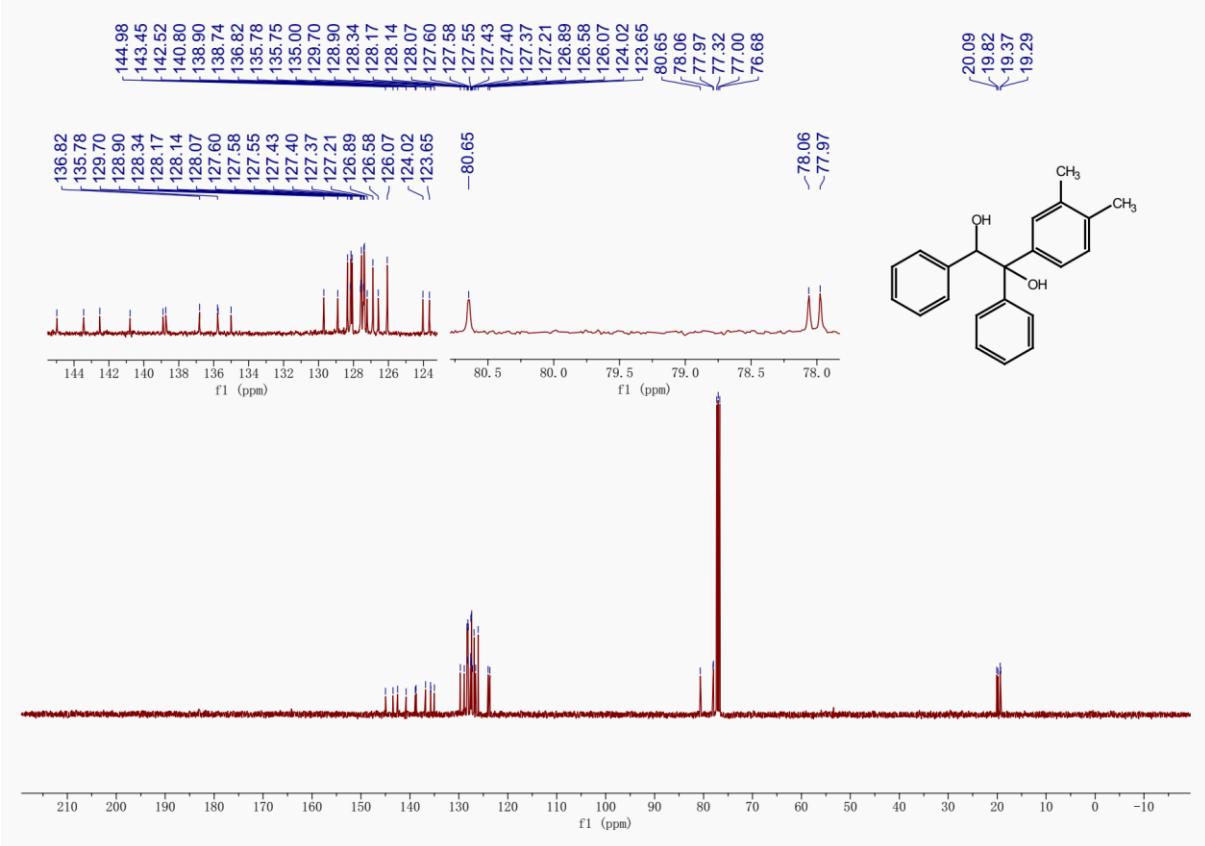
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¹H NMR (400 MHz, CDCl₃) of Compound 3ak



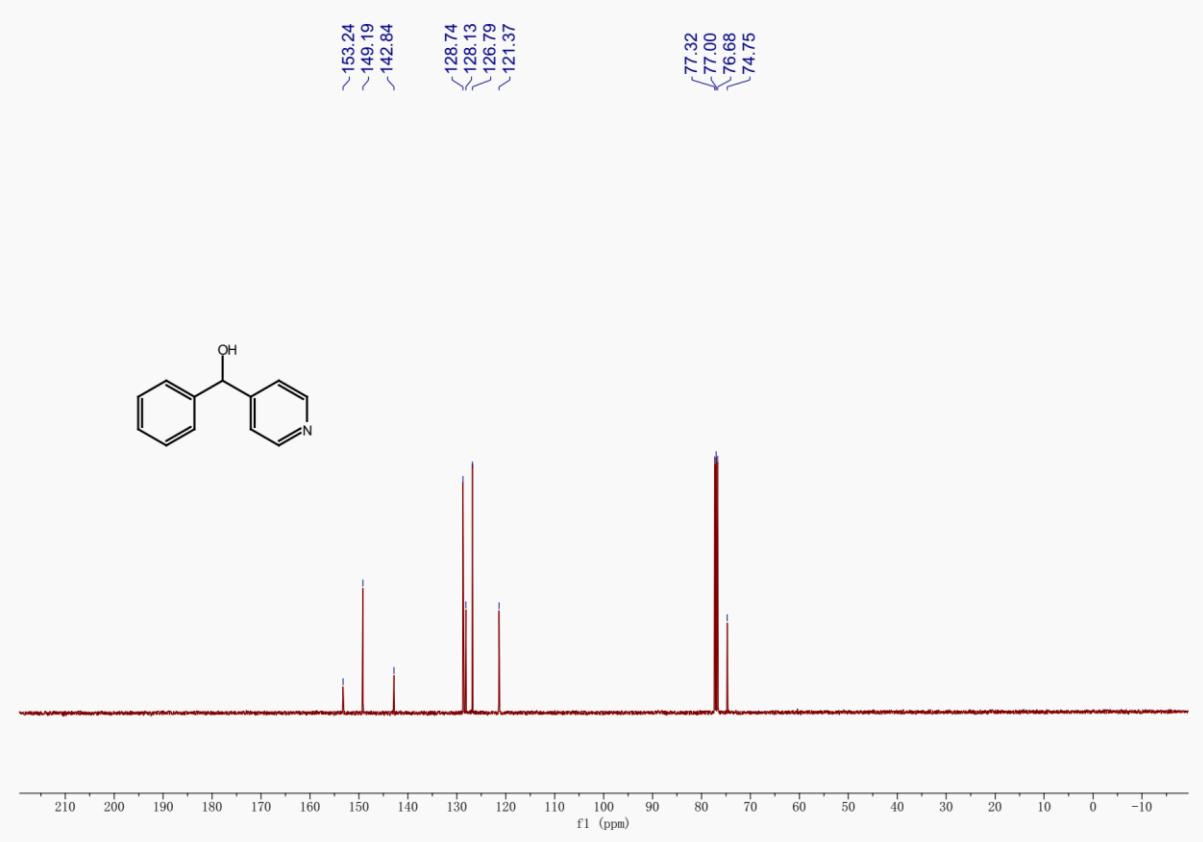
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¹H NMR (400 MHz, CDCl₃) of Compound 5aa



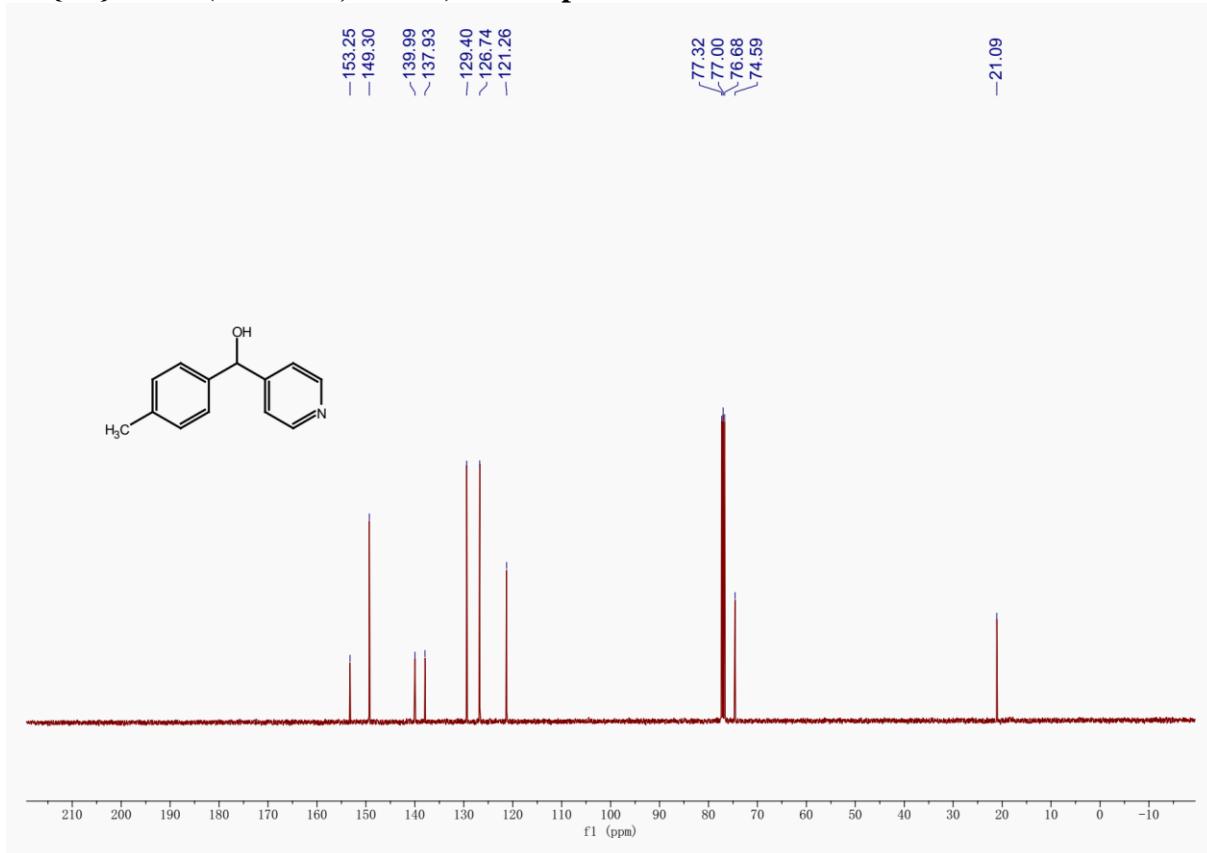
¹³C{¹H} NMR (101 MHz, CDCl₃) of Compound 5aa



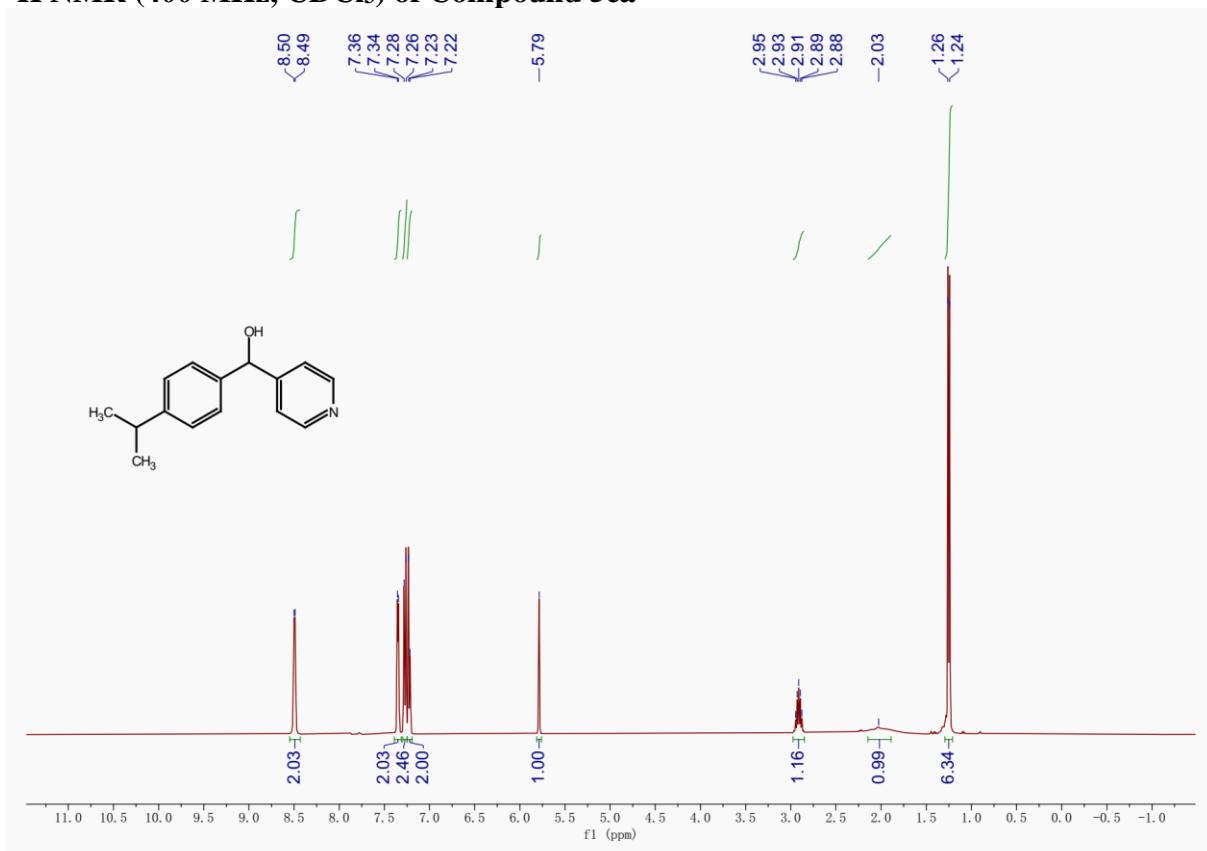
^1H NMR (400 MHz, CDCl_3) of Compound 5ba



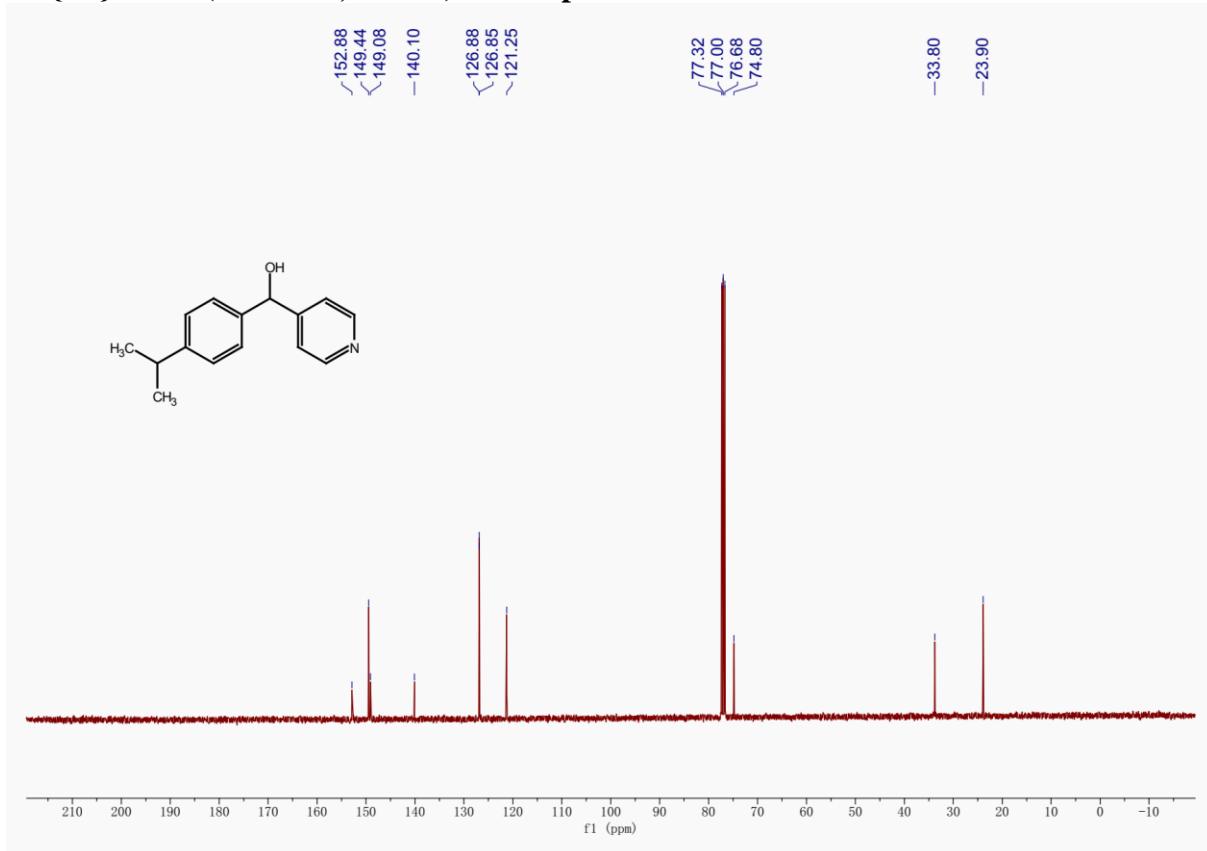
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ba



^1H NMR (400 MHz, CDCl_3) of Compound 5ca



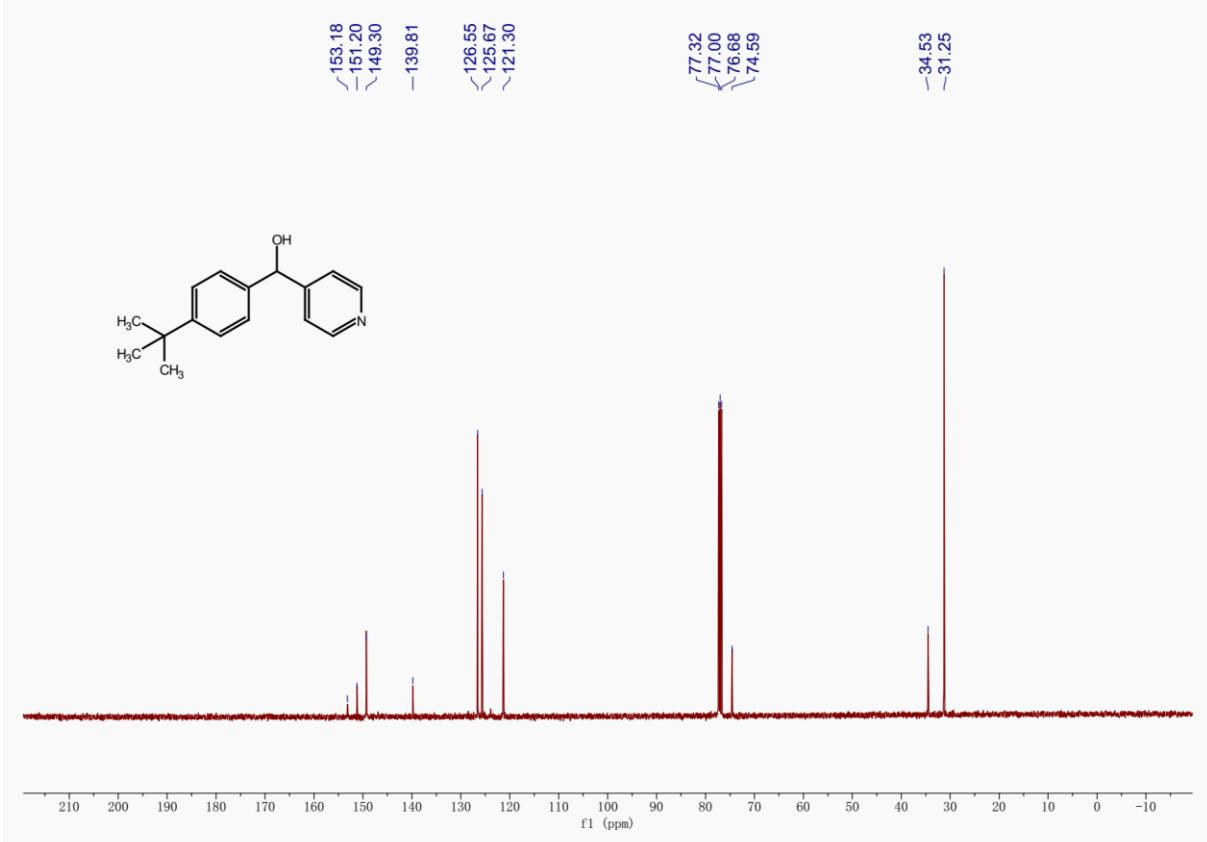
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ca



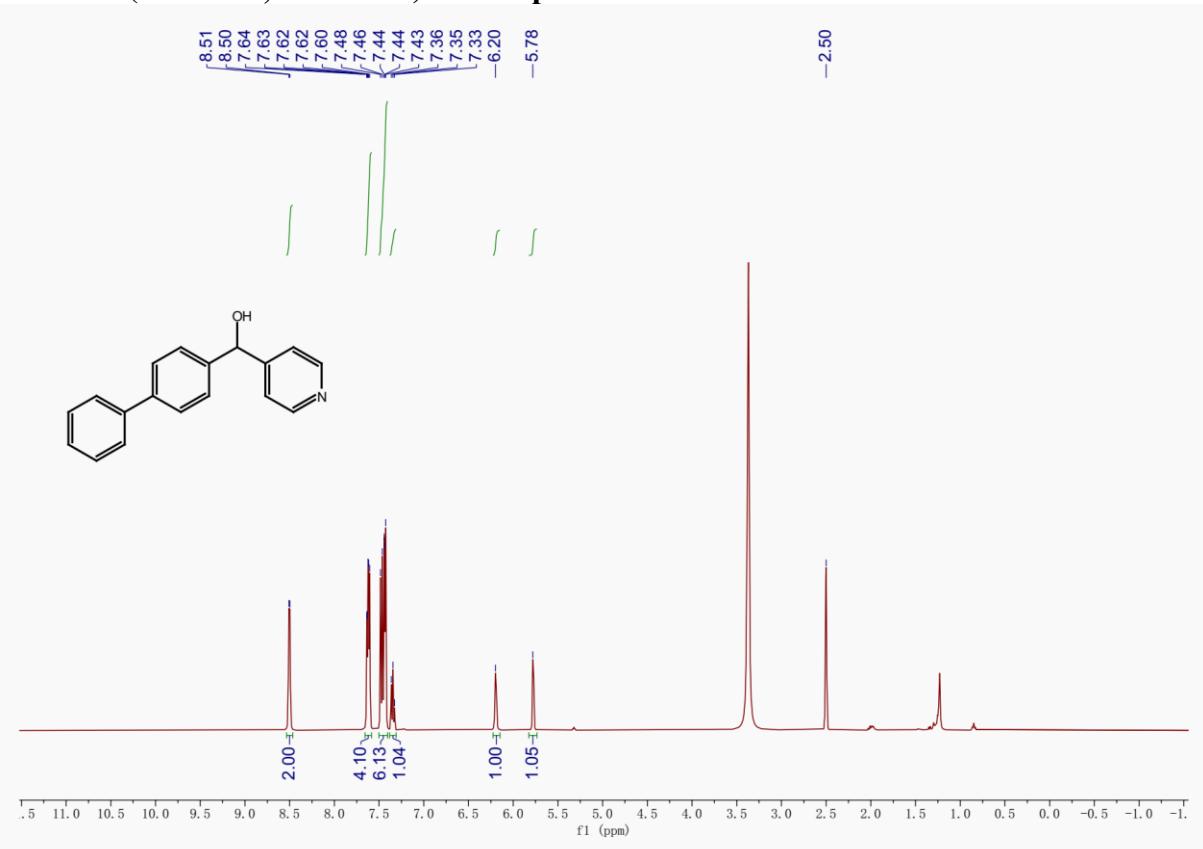
¹H NMR (400 MHz, CDCl₃) of Compound 5da



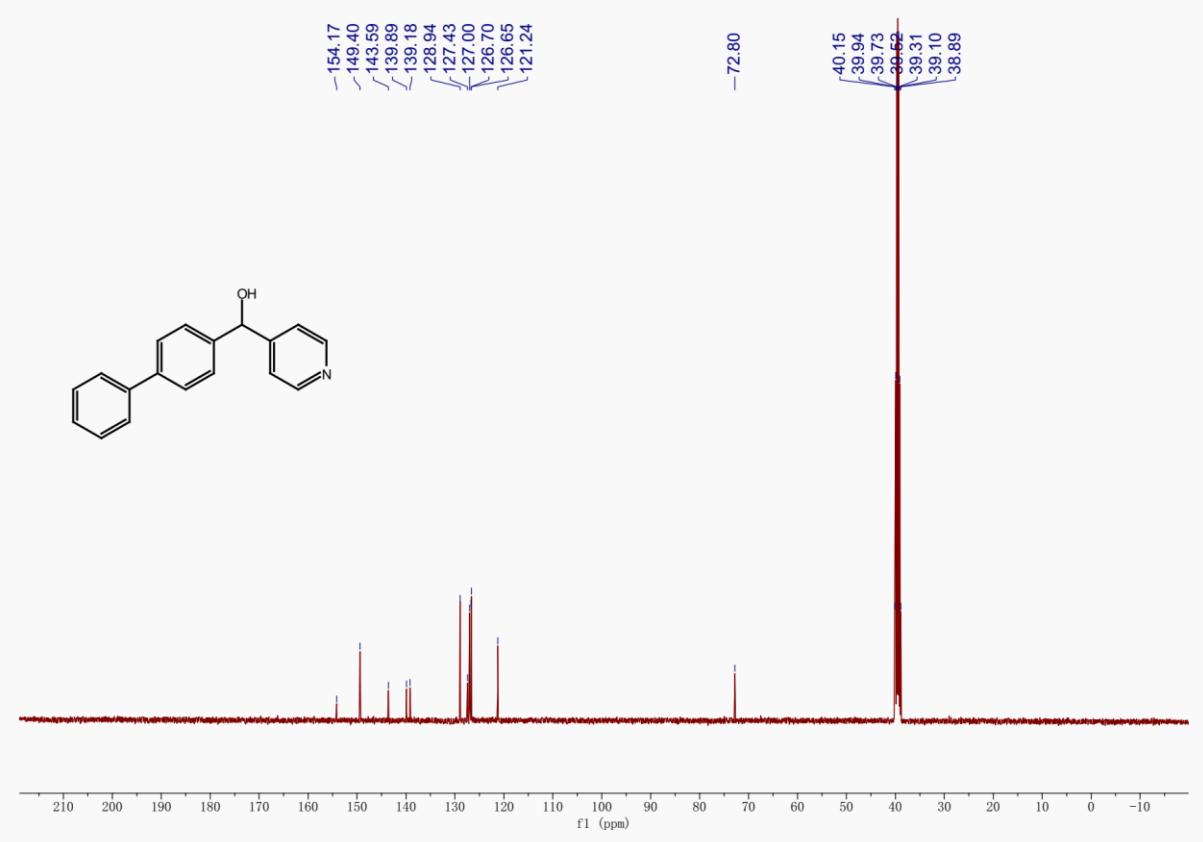
¹³C{¹H} NMR (101 MHz, CDCl₃) of Compound 5da



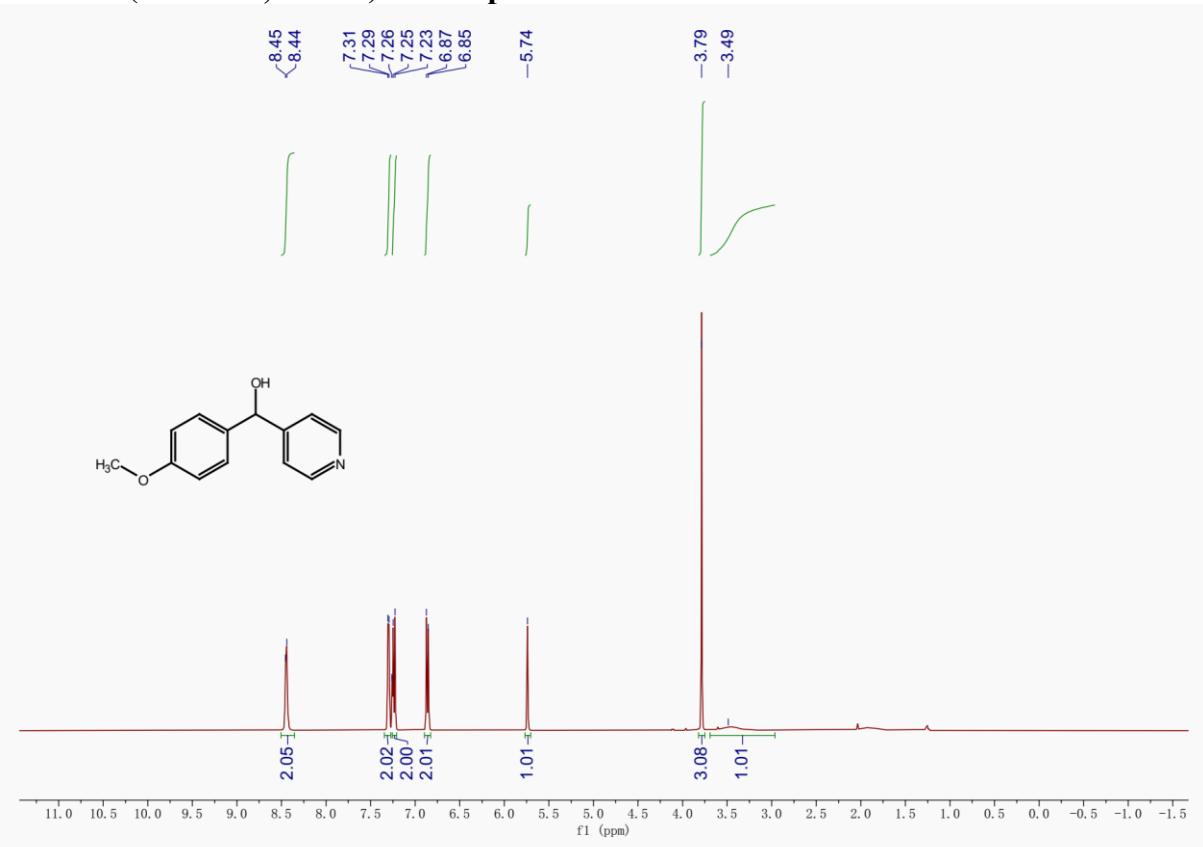
^1H NMR (400 MHz, DMSO-*d*₆) of Compound 5ea



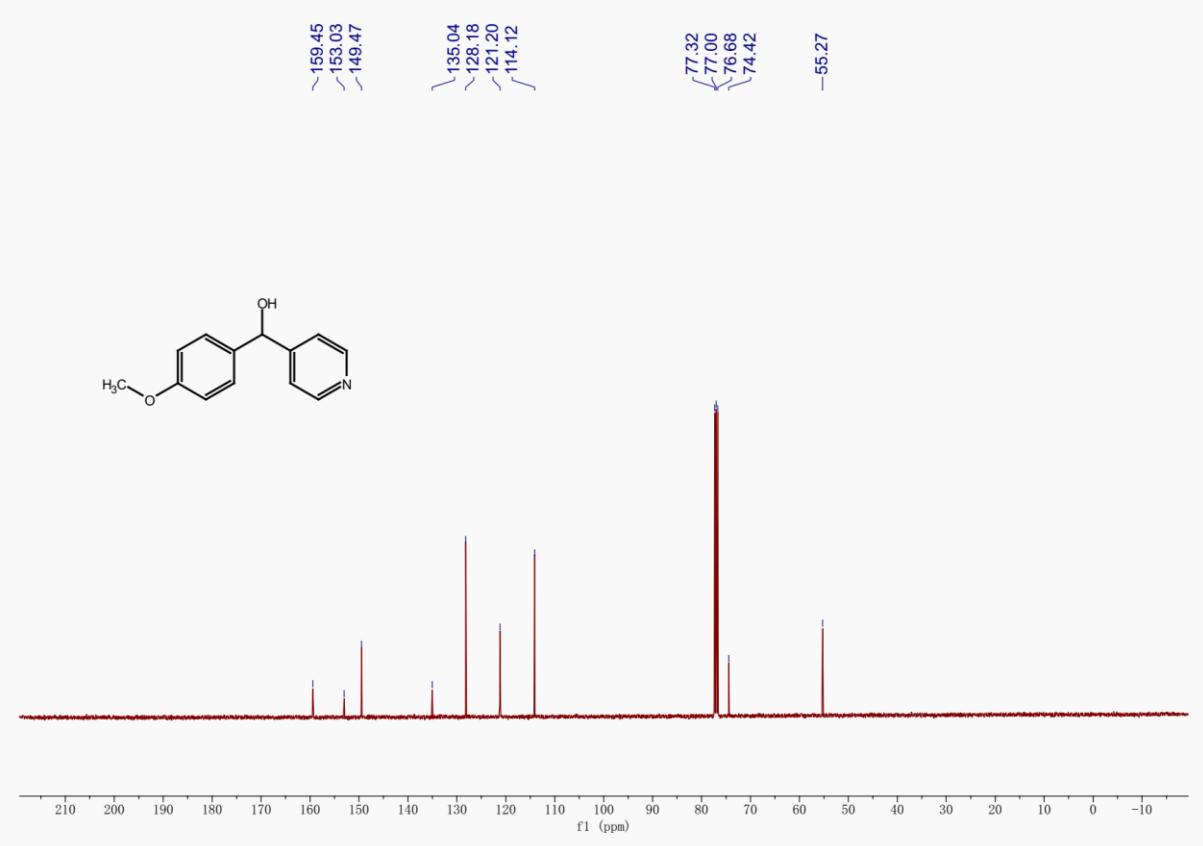
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, DMSO-*d*₆) of Compound 5ea



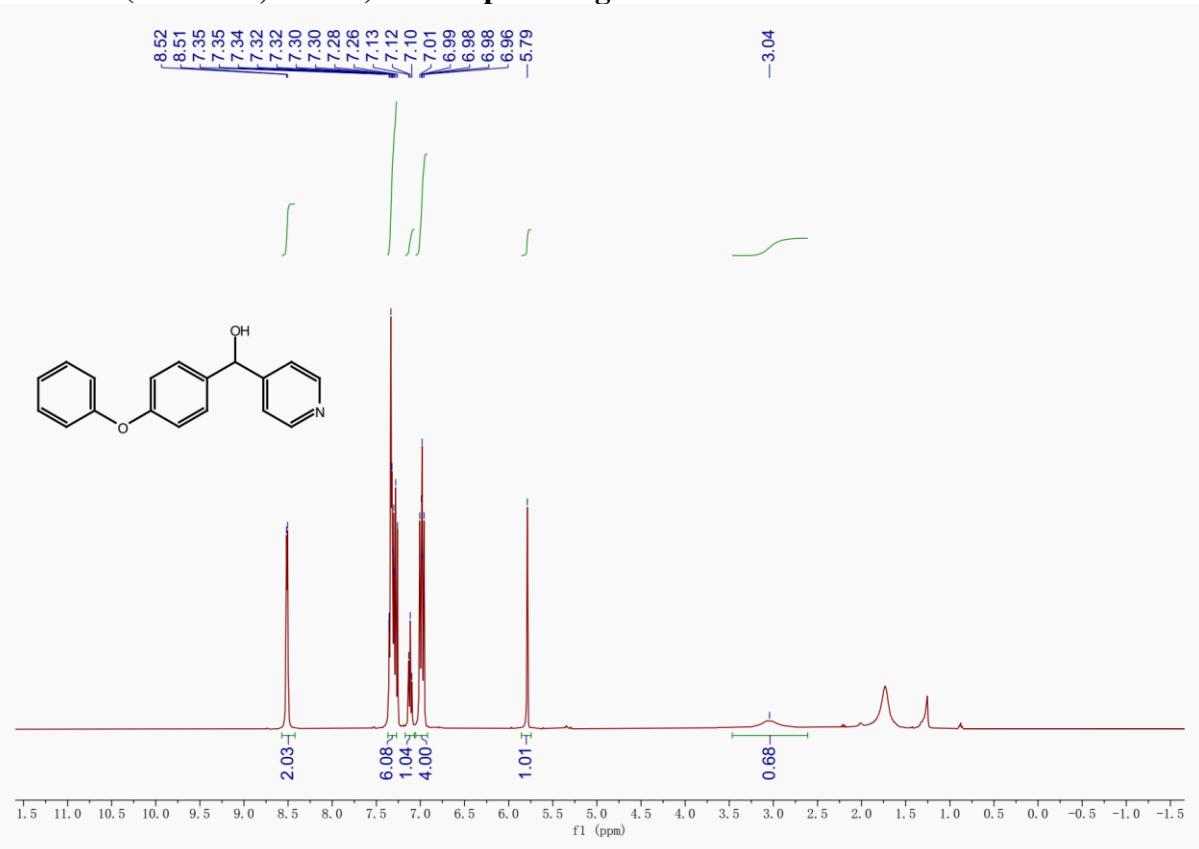
^1H NMR (400 MHz, CDCl_3) of Compound 5fa



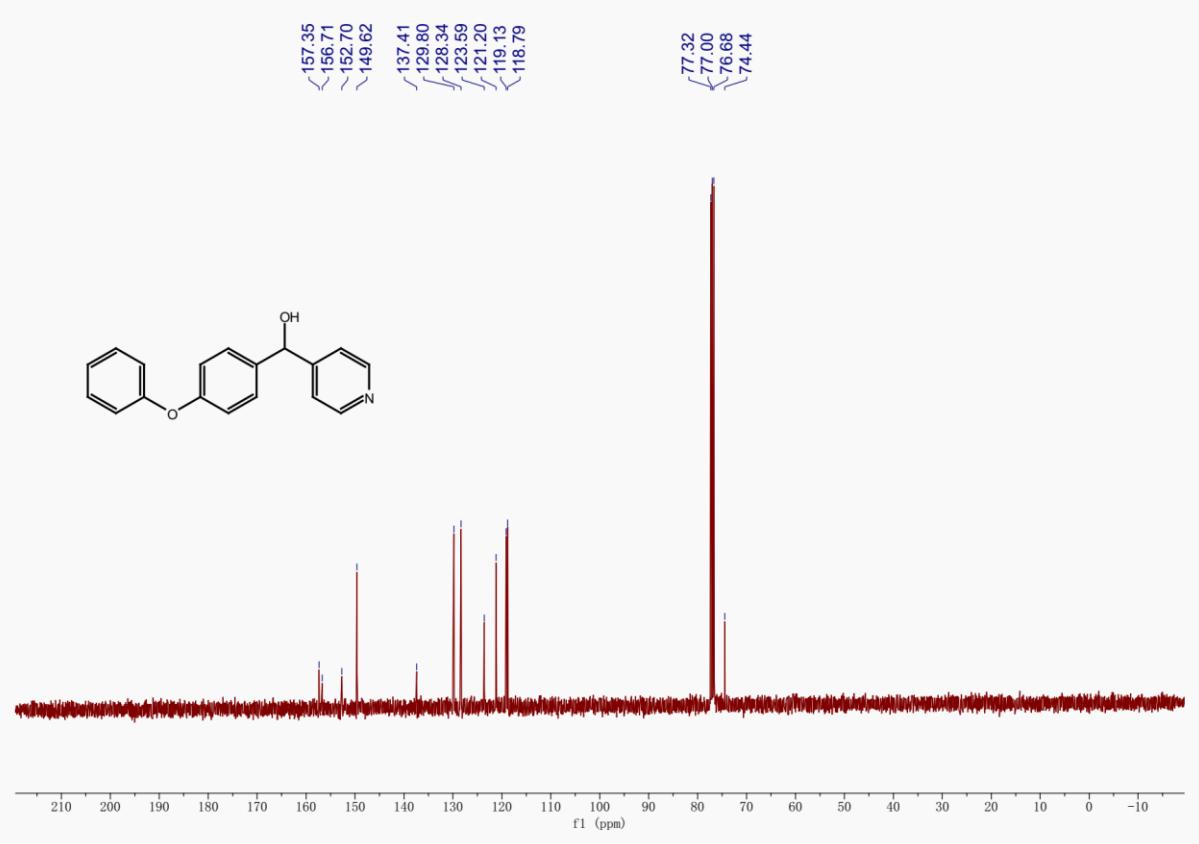
$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5fa



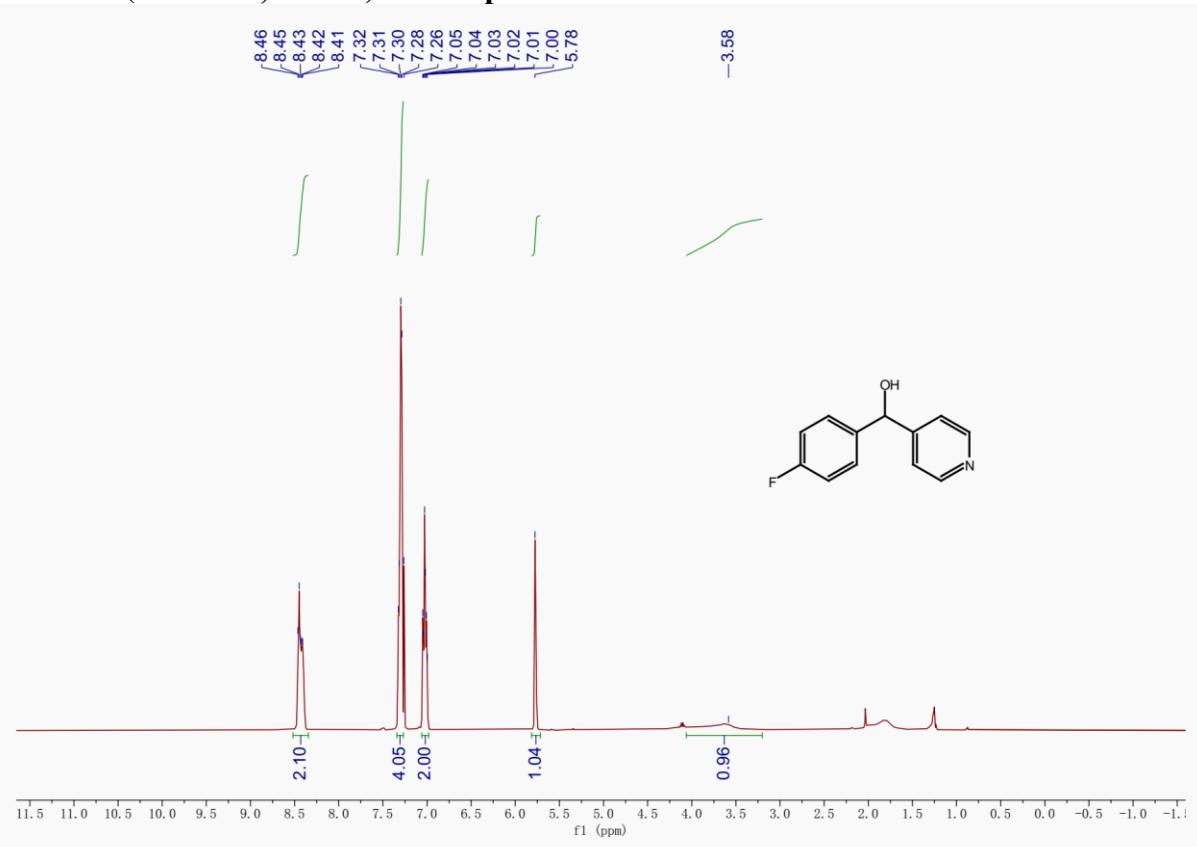
^1H NMR (400 MHz, CDCl_3) of Compound 5ga



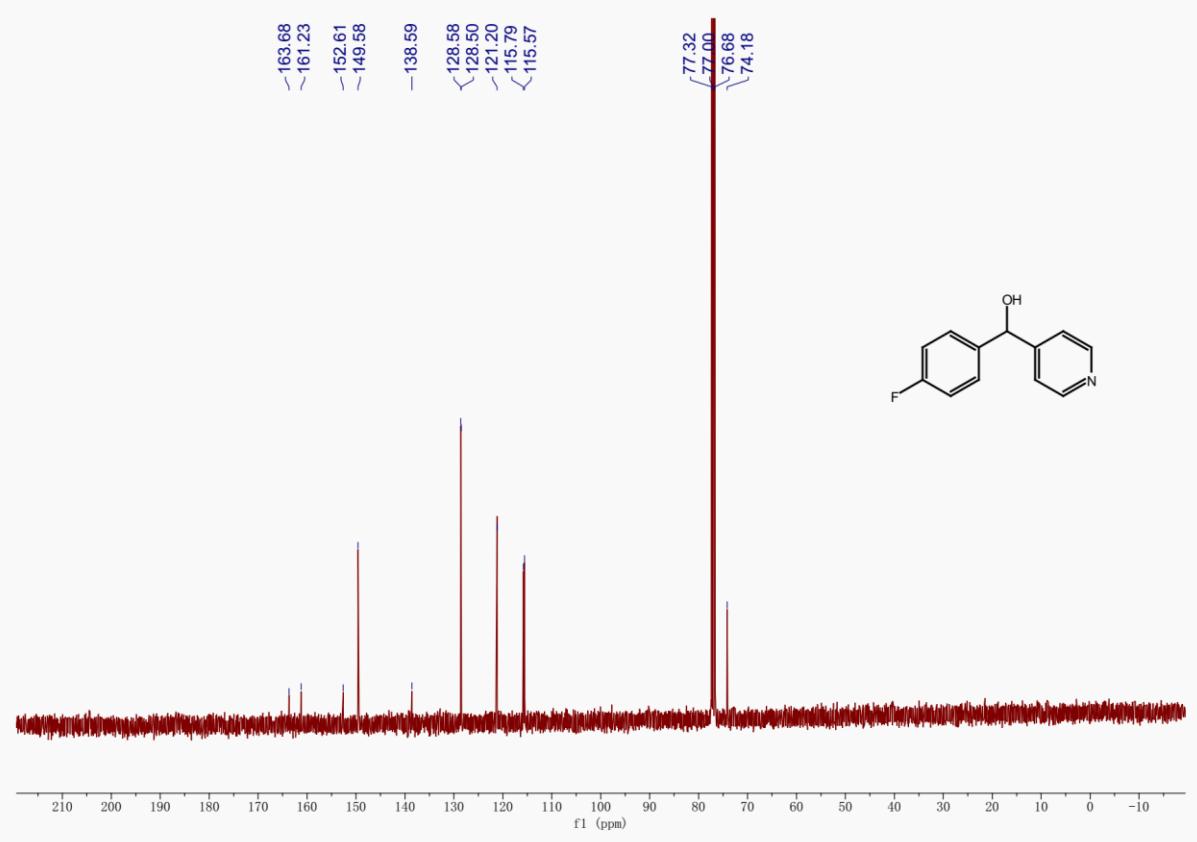
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ga



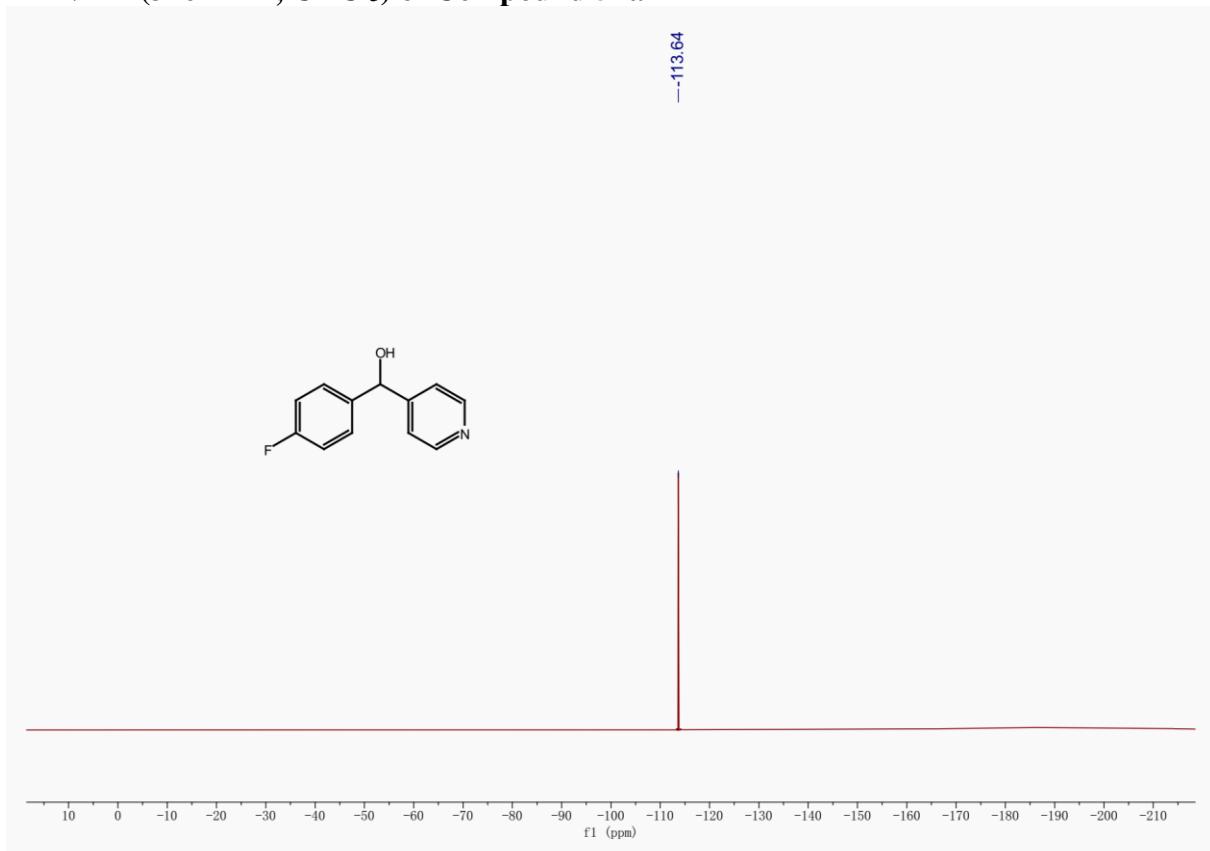
^1H NMR (400 MHz, CDCl_3) of Compound 5ha



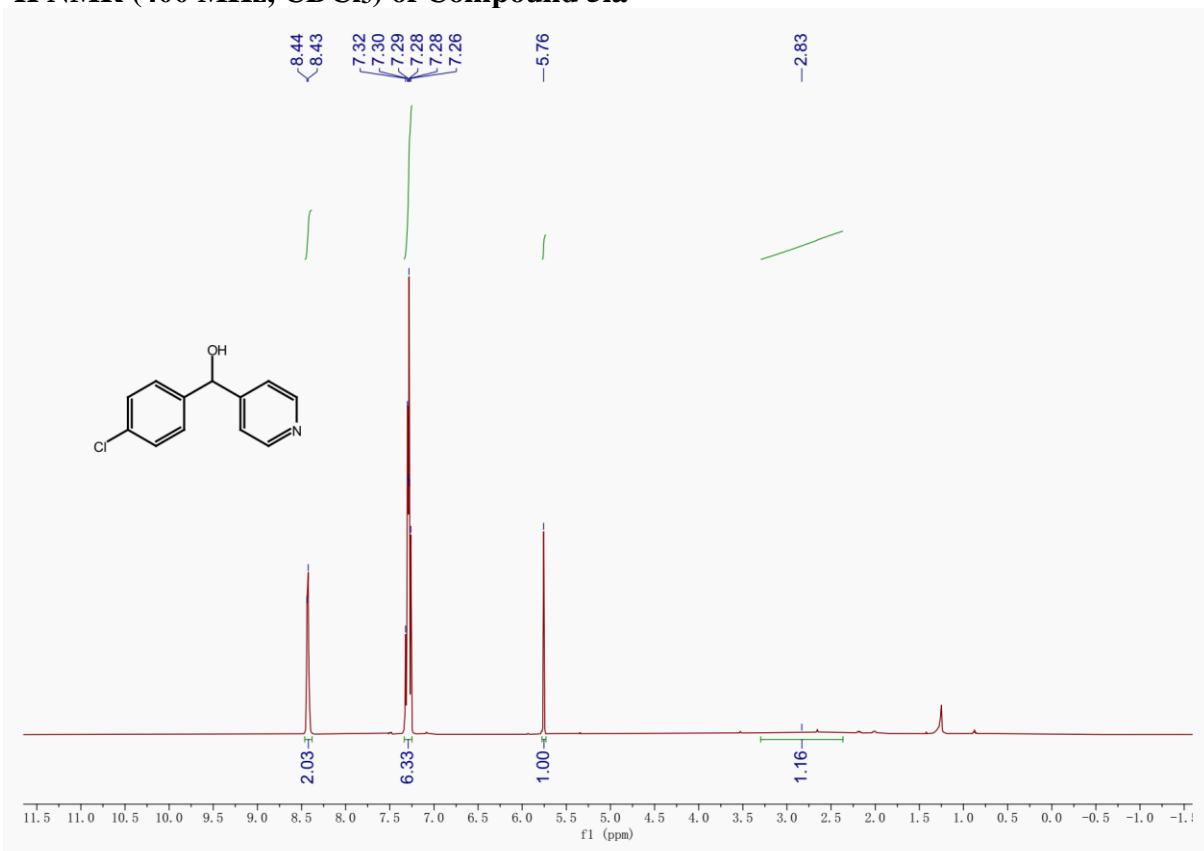
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ha



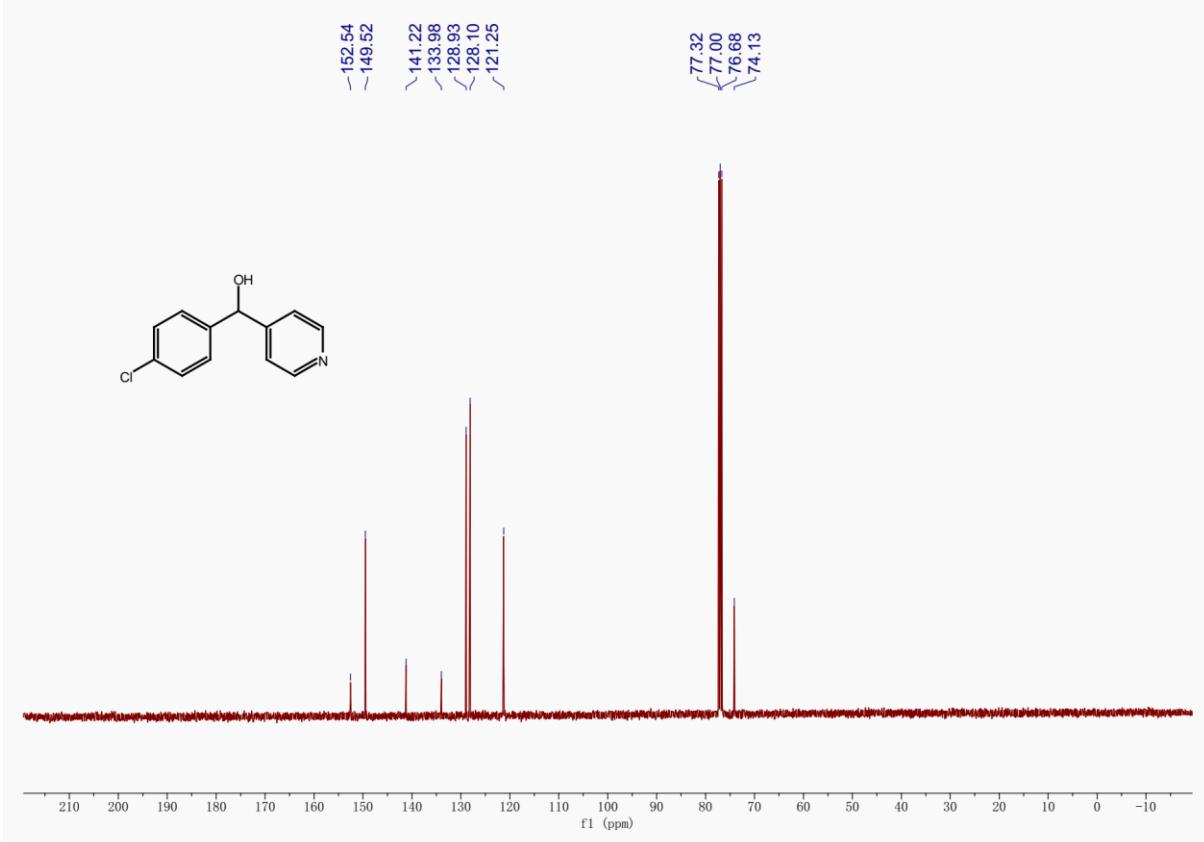
¹⁹F NMR (376 MHz, CDCl₃) of Compound 5ha



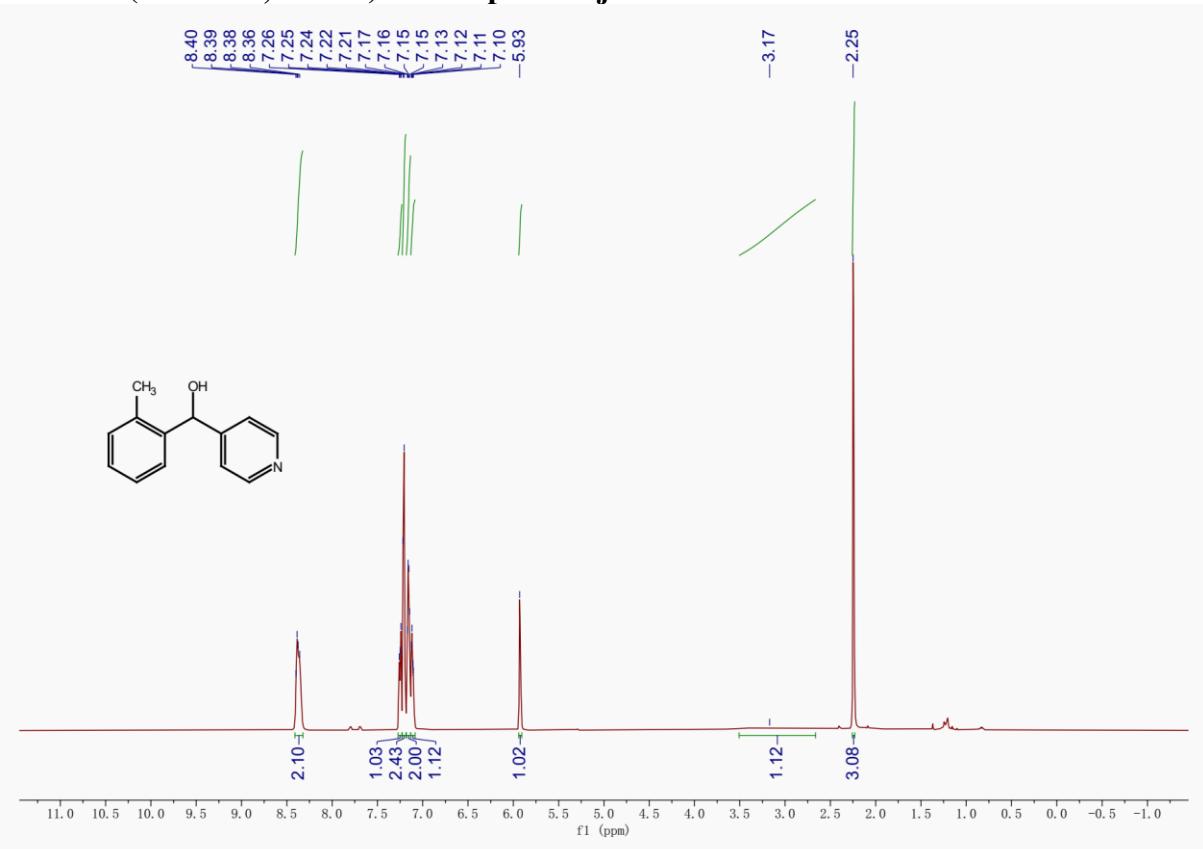
^1H NMR (400 MHz, CDCl_3) of Compound 5ia



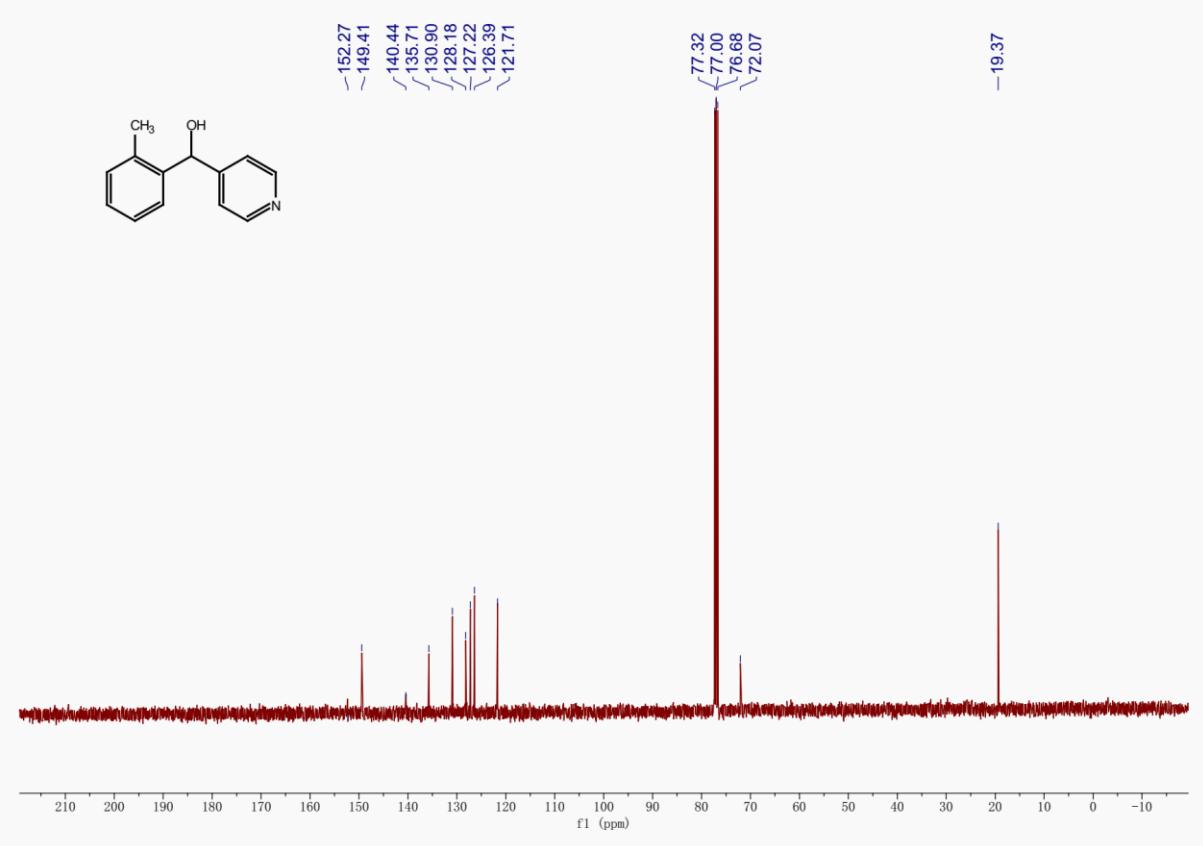
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ia



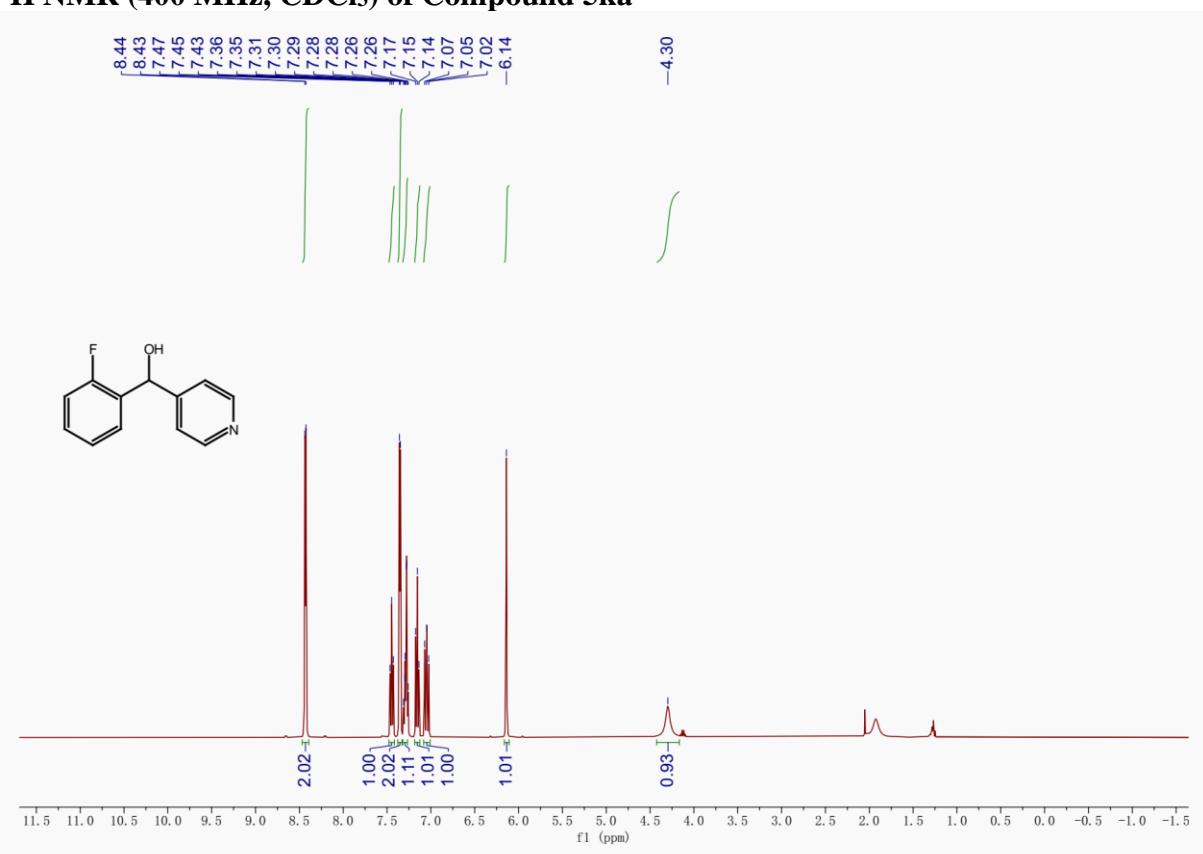
¹H NMR (400 MHz, CDCl₃) of Compound 5ja



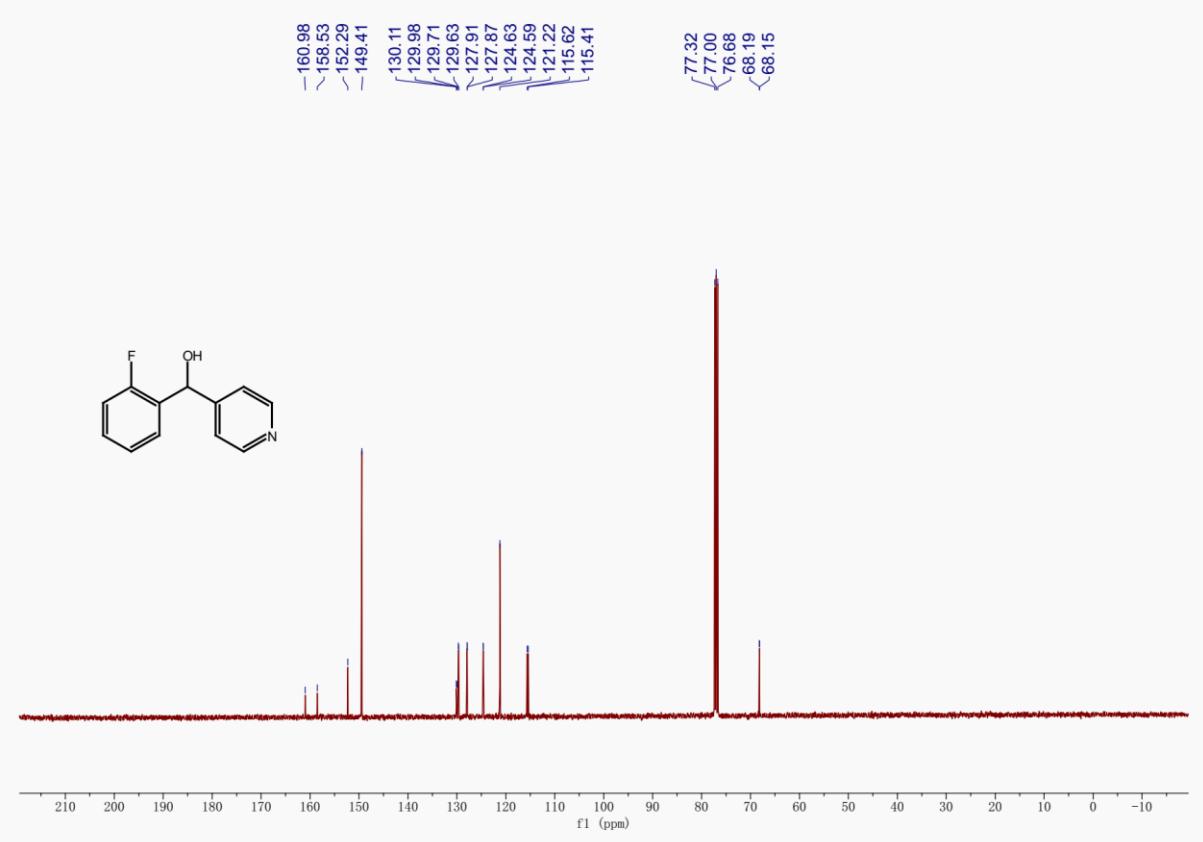
$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ja



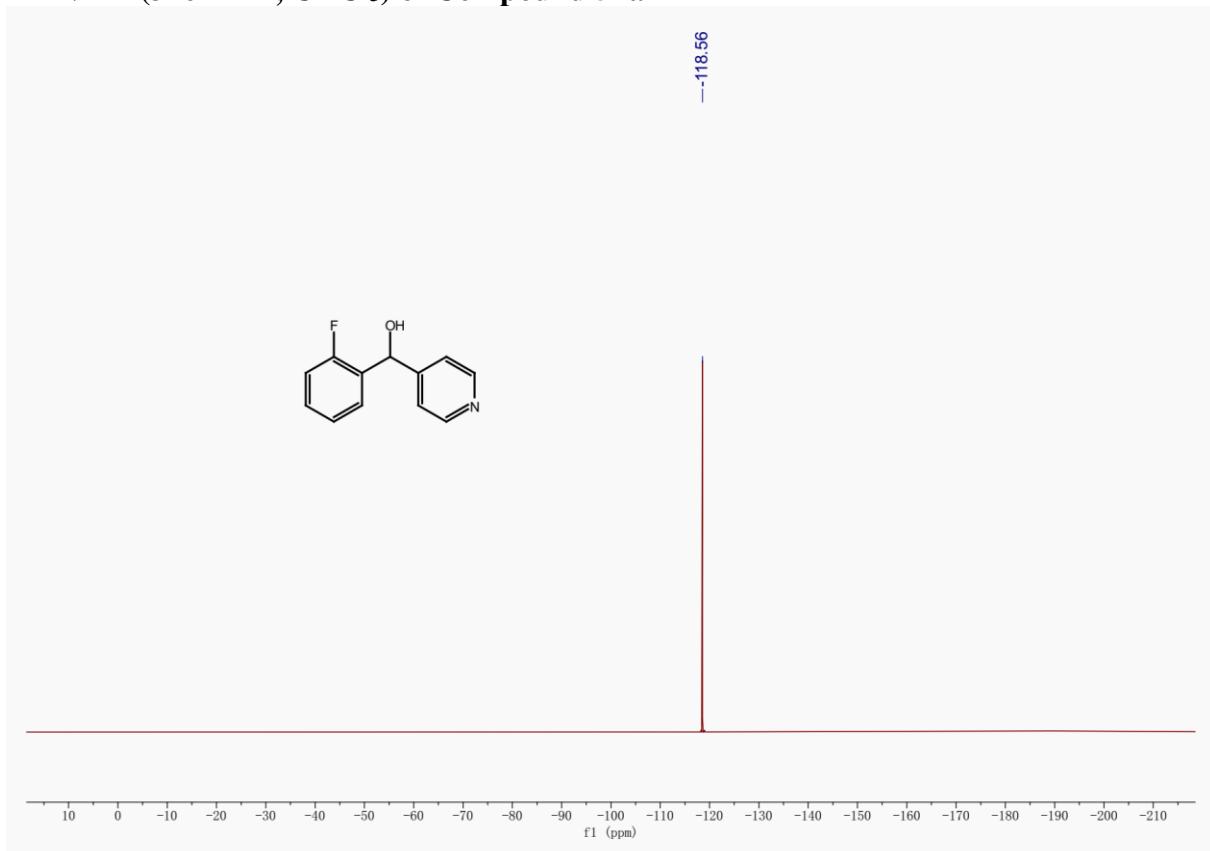
^1H NMR (400 MHz, CDCl_3) of Compound 5ka



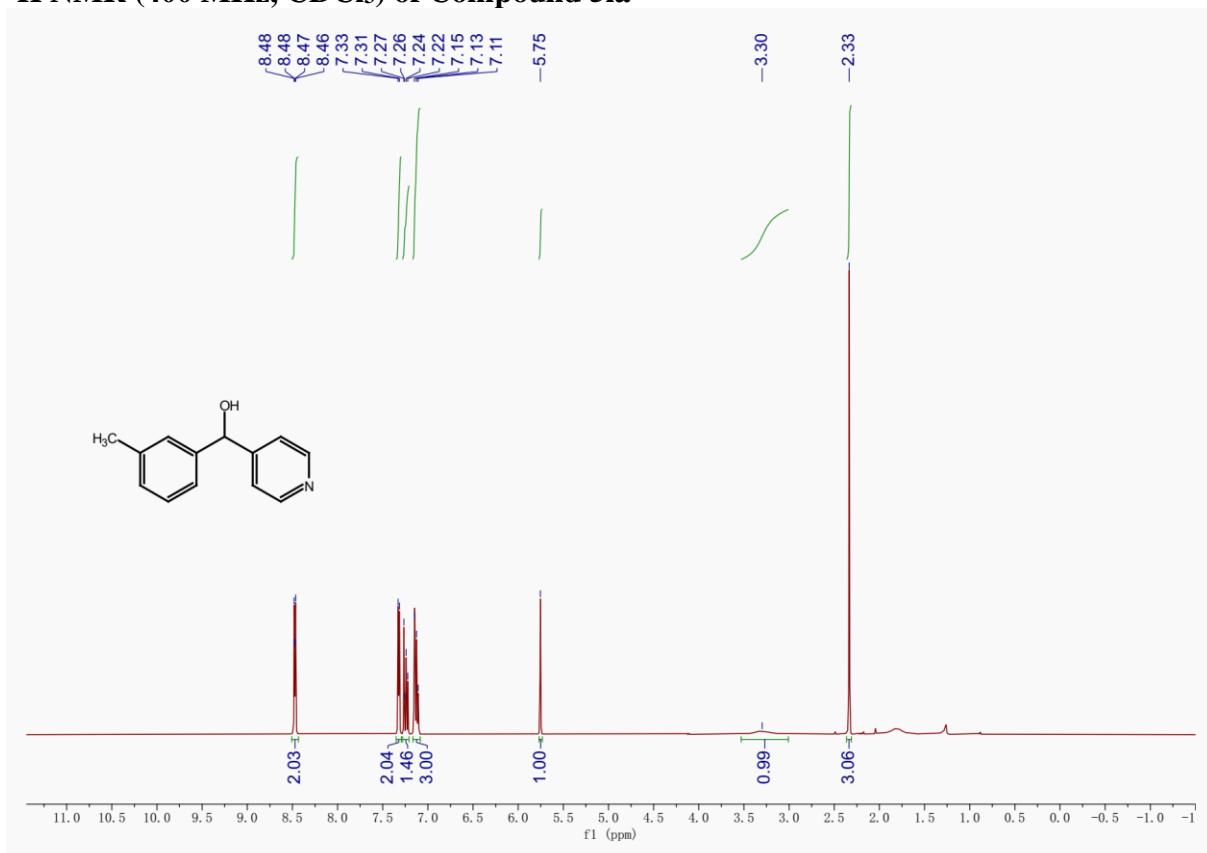
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ka



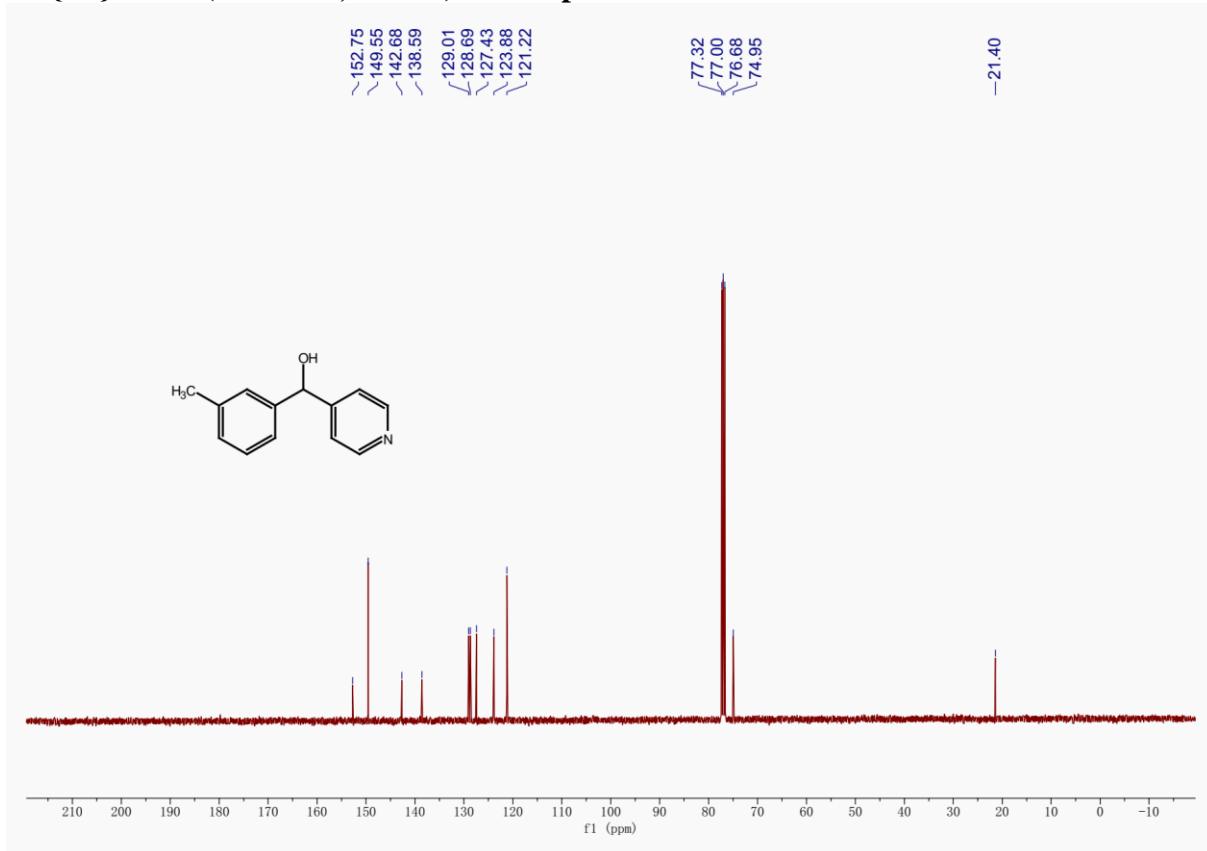
¹⁹F NMR (376 MHz, CDCl₃) of Compound 5ka



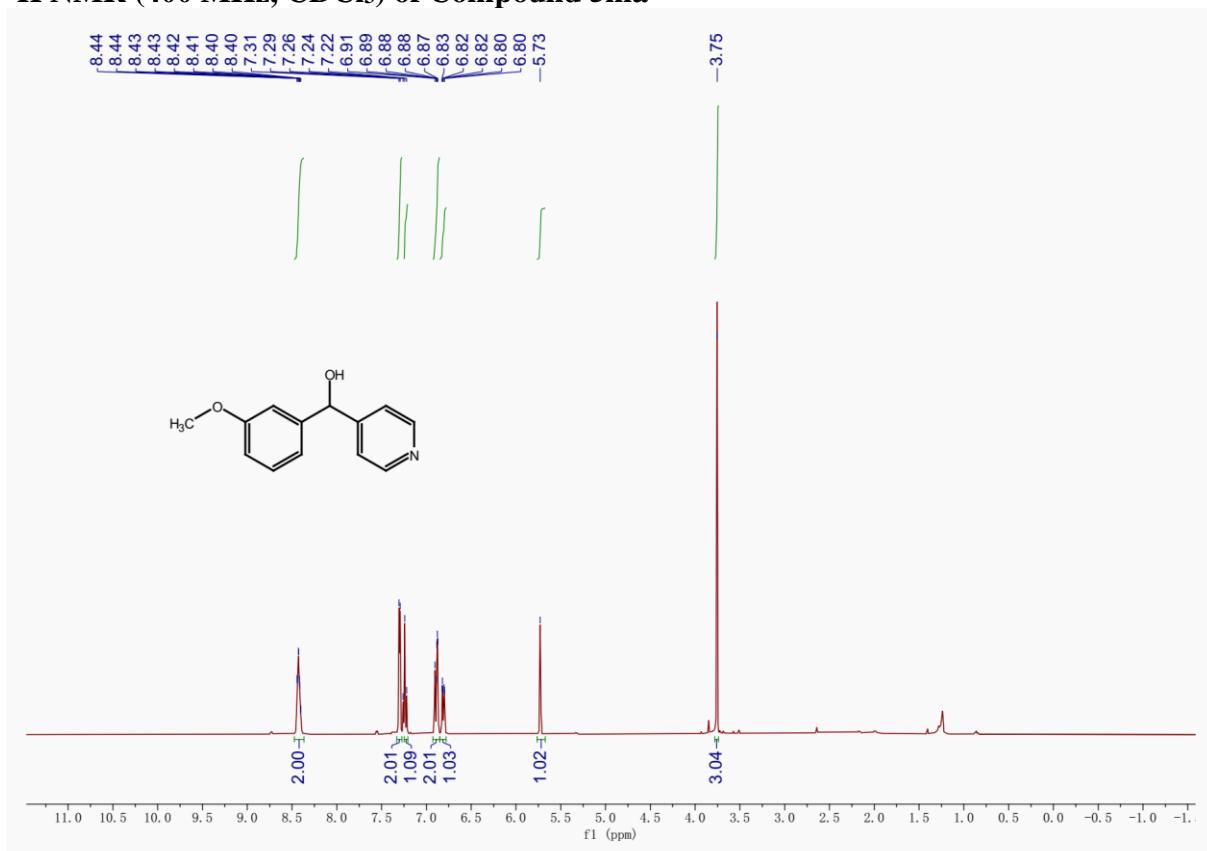
¹H NMR (400 MHz, CDCl₃) of Compound 5la



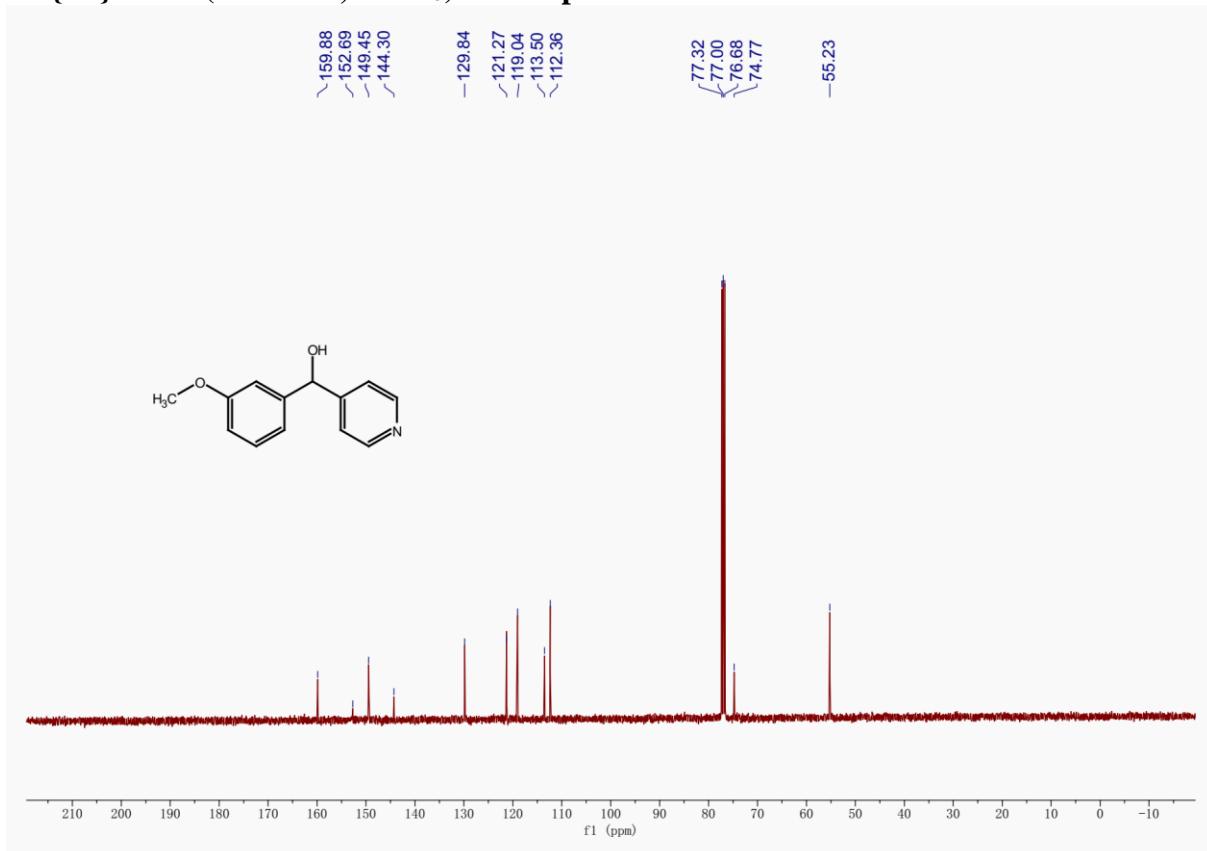
¹³C{¹H} NMR (101 MHz, CDCl₃) of Compound 5la



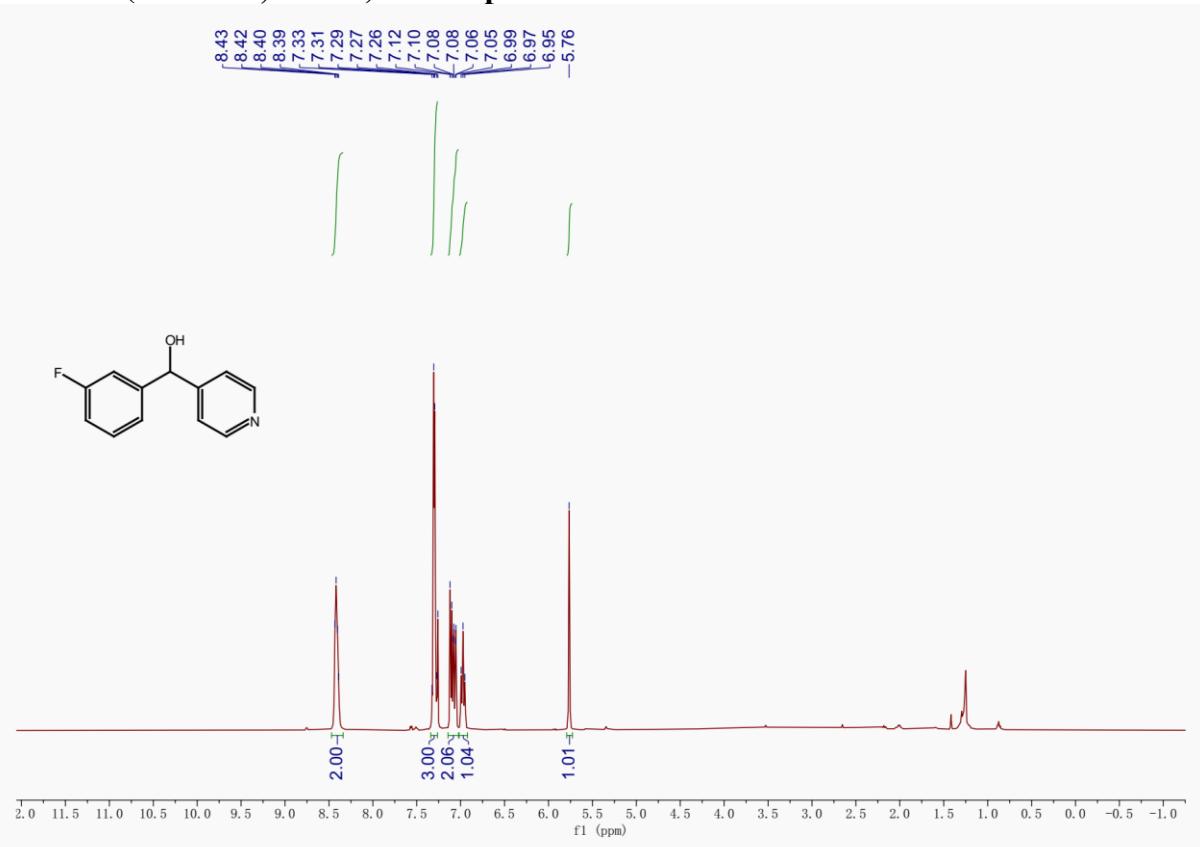
^1H NMR (400 MHz, CDCl_3) of Compound 5ma



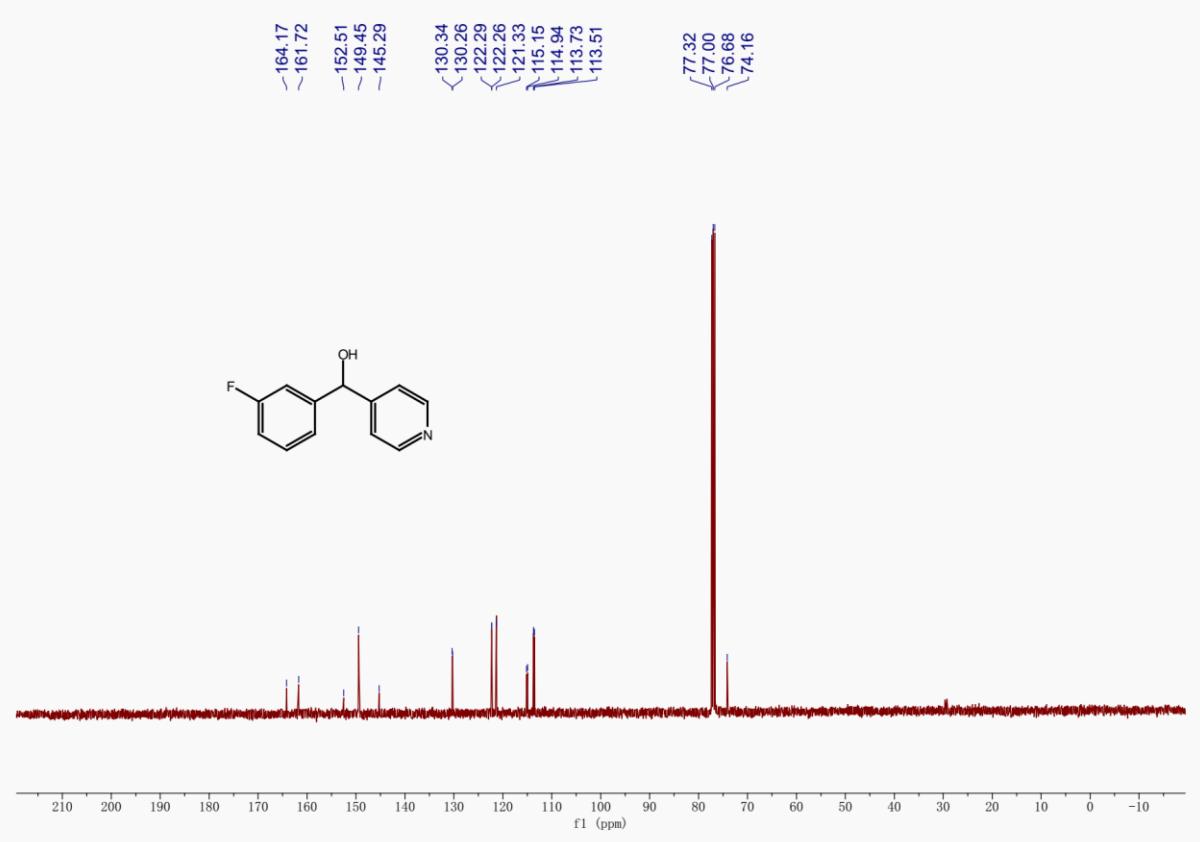
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ma



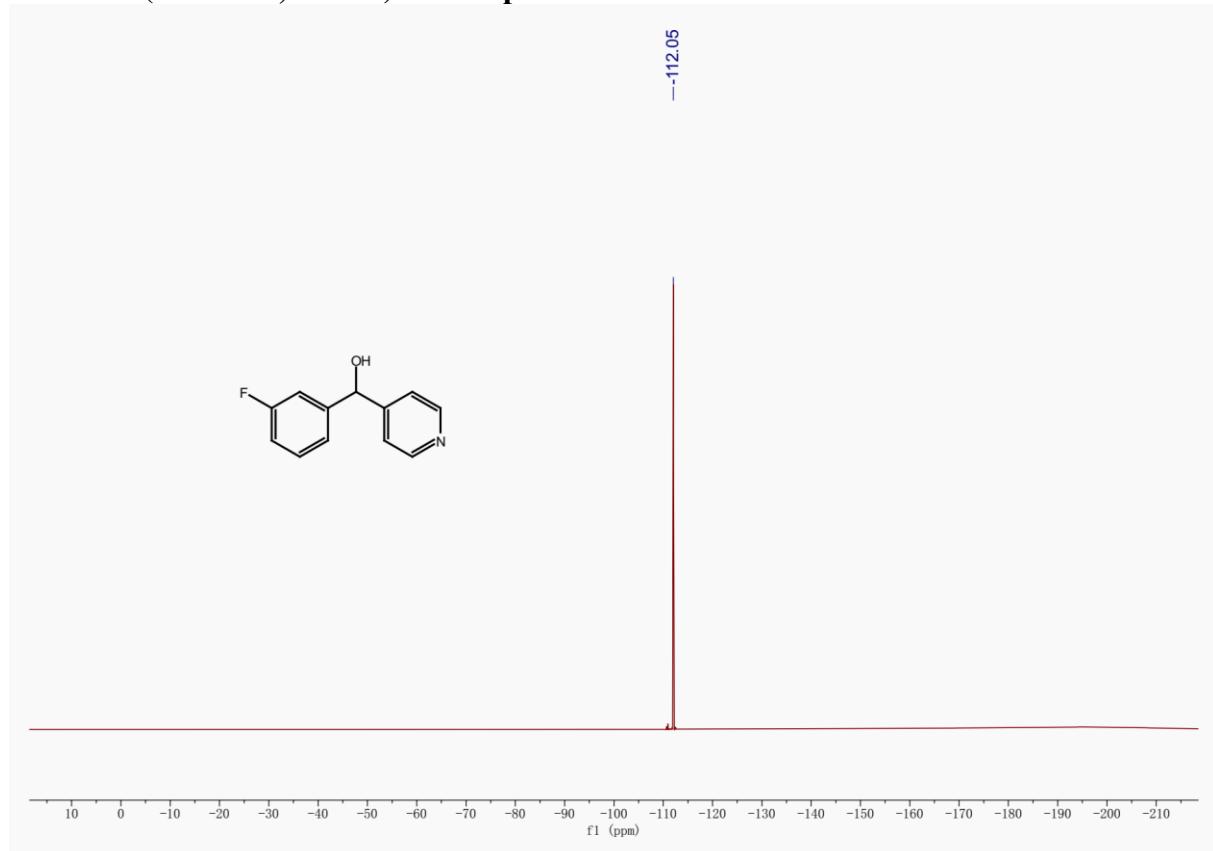
^1H NMR (400 MHz, CDCl_3) of Compound 5na



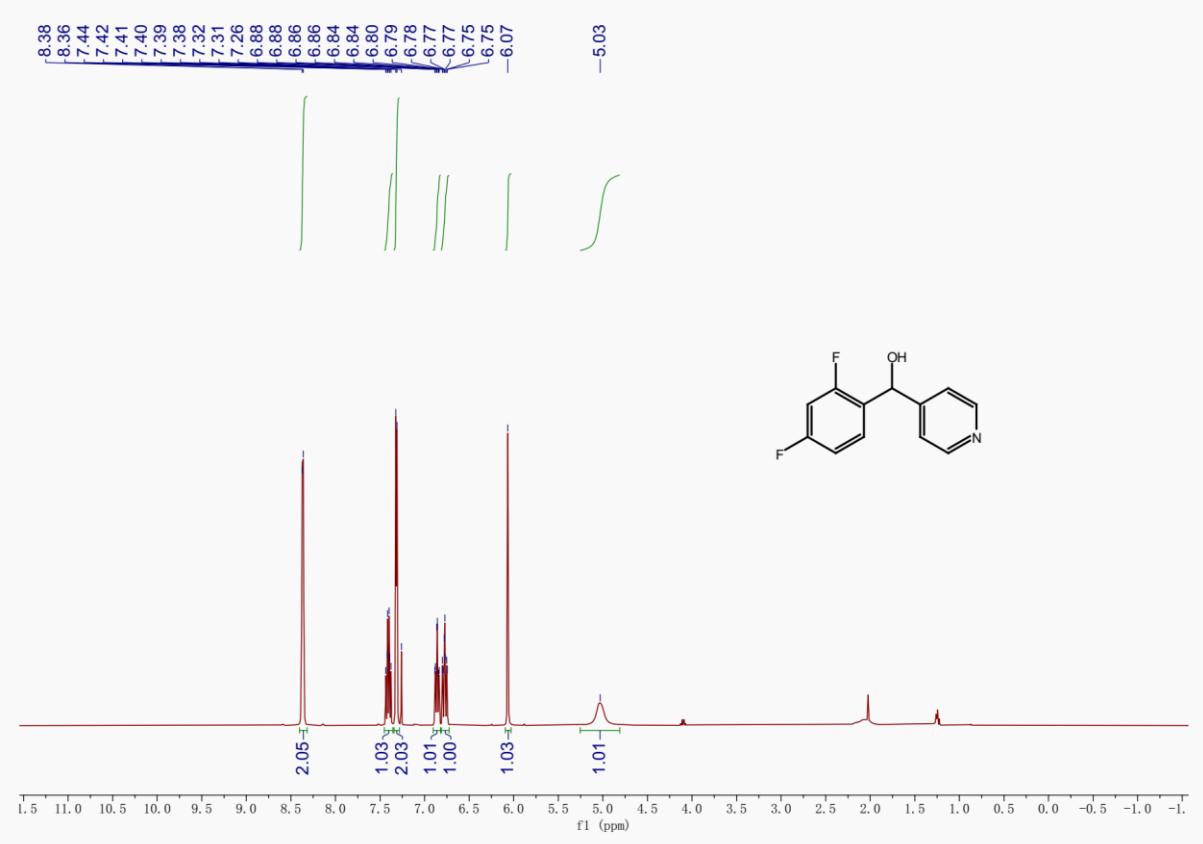
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5na



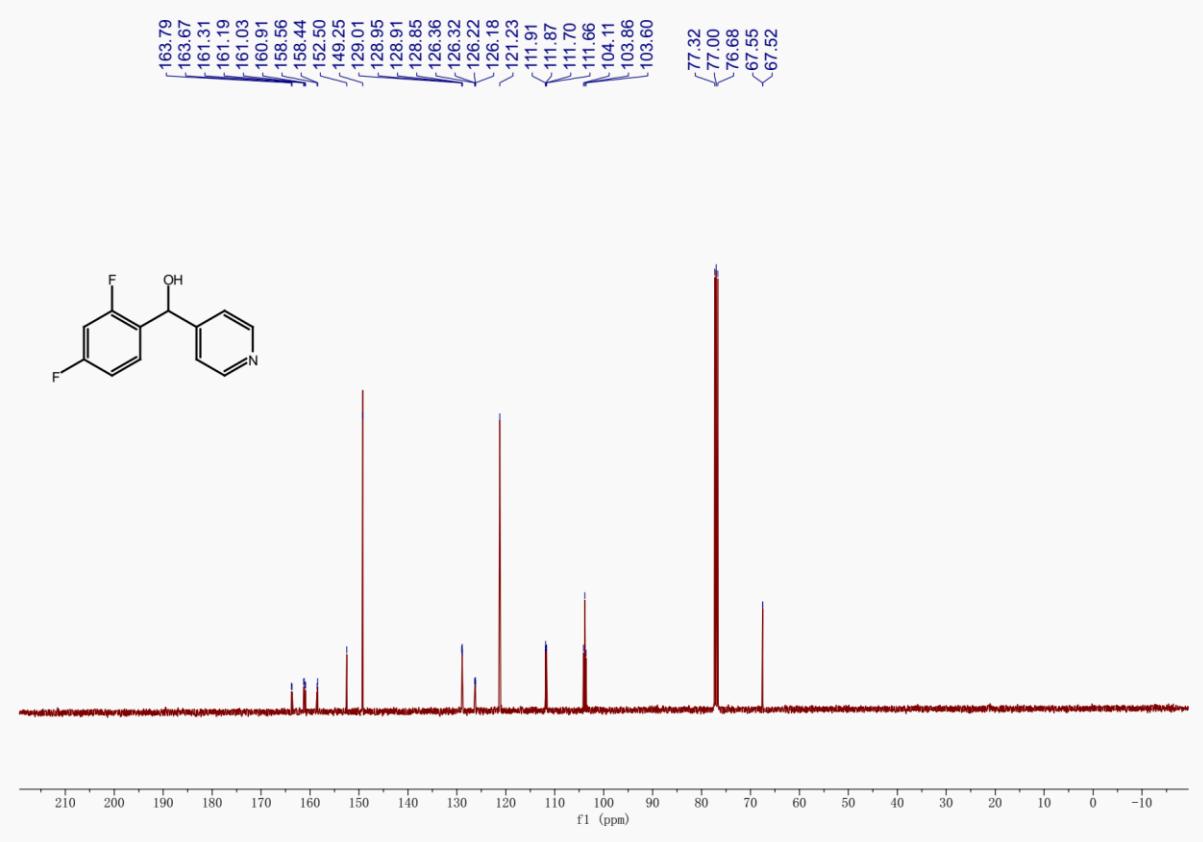
¹⁹F NMR (376 MHz, CDCl₃) of Compound 5na



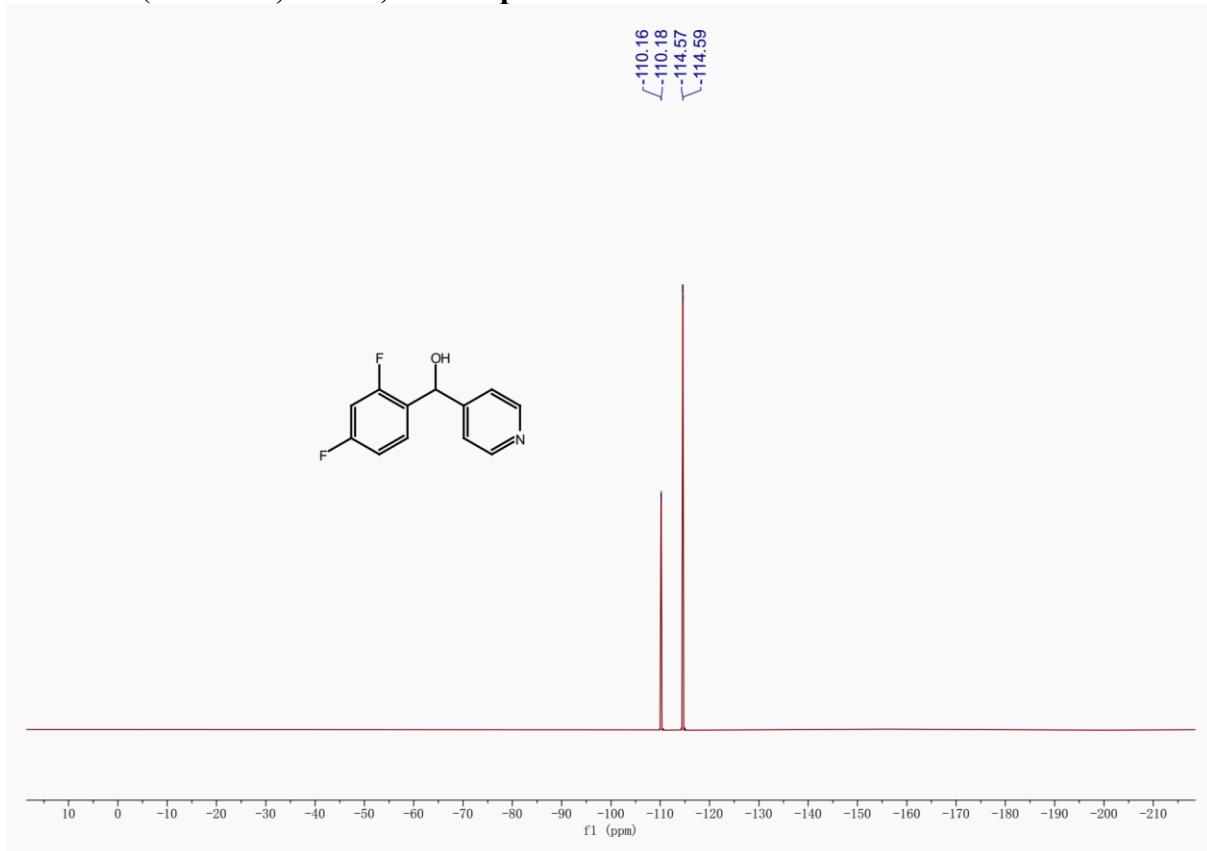
¹H NMR (400 MHz, CDCl₃) of Compound 5oa



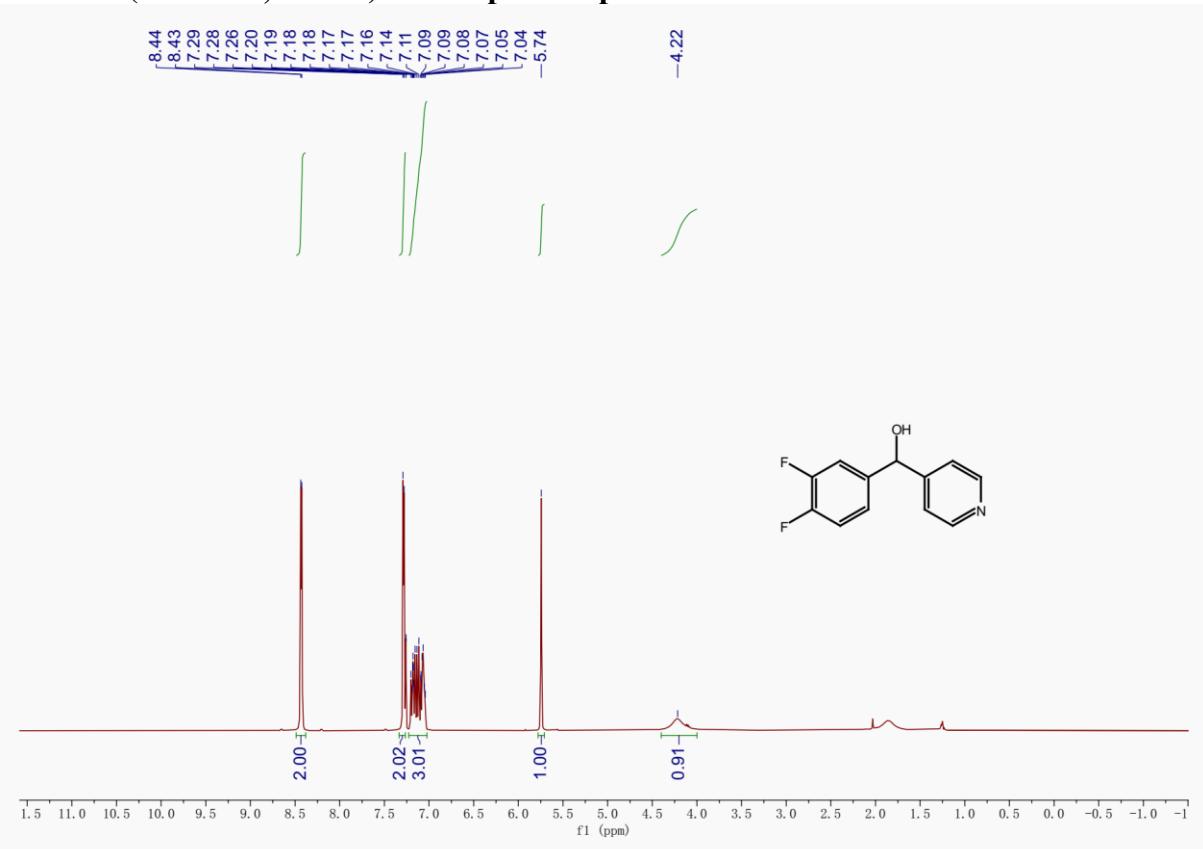
¹³C{¹H} NMR (101 MHz, CDCl₃) of Compound 5oa



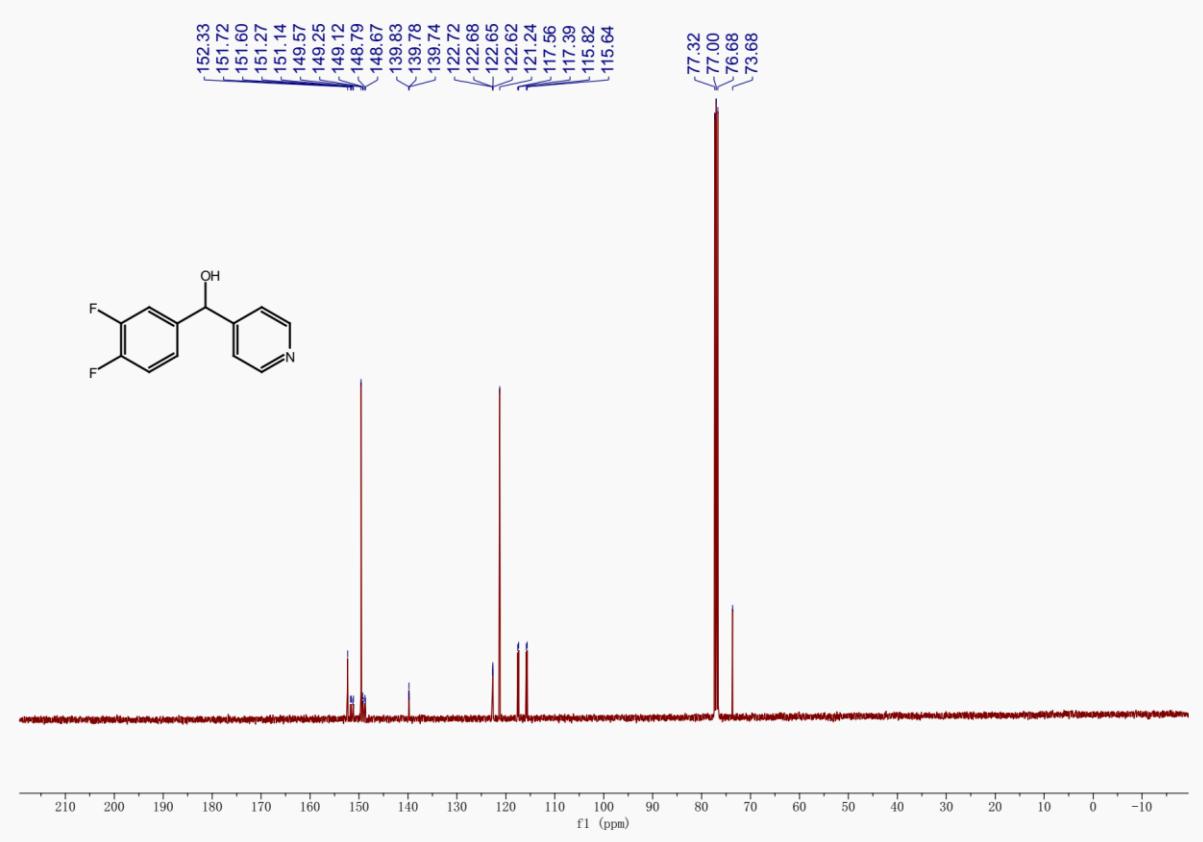
¹⁹F NMR (376 MHz, CDCl₃) of Compound 5oa



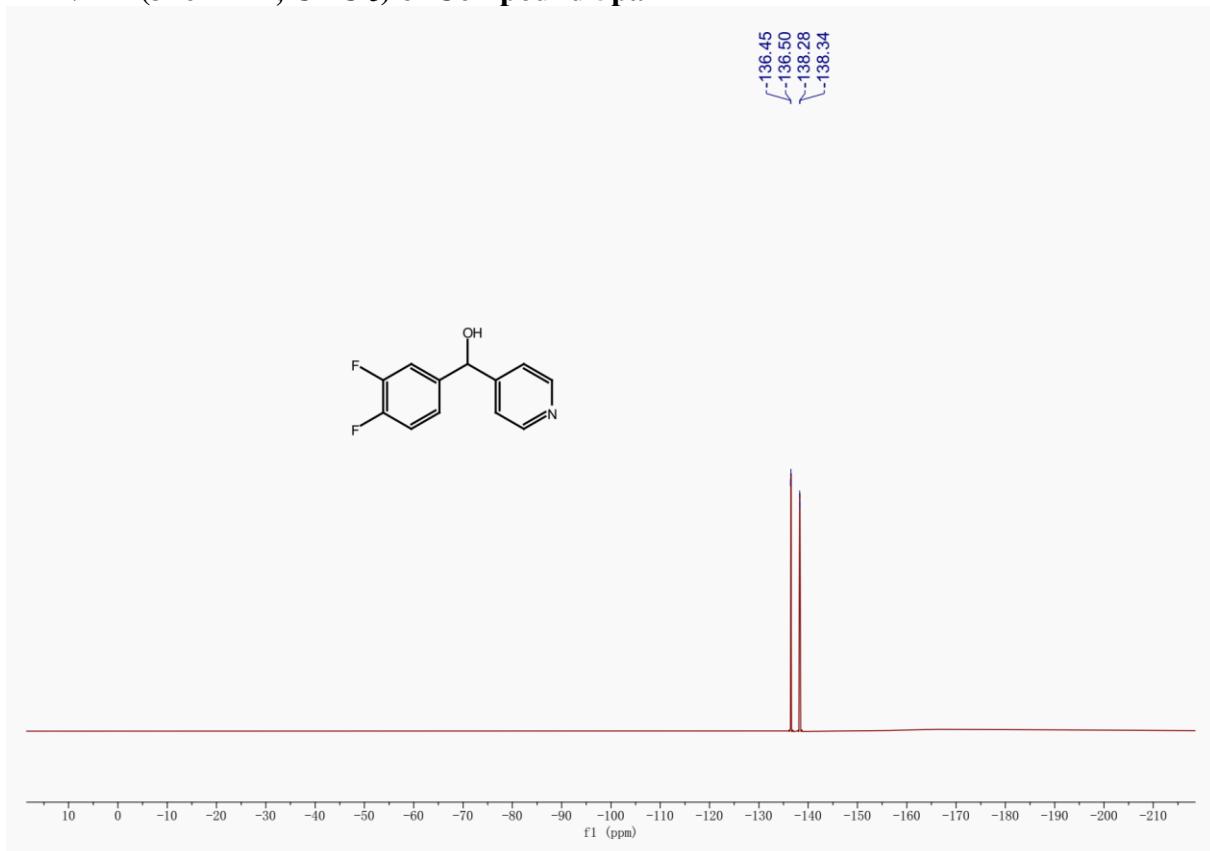
^1H NMR (400 MHz, CDCl_3) of Compound 5pa



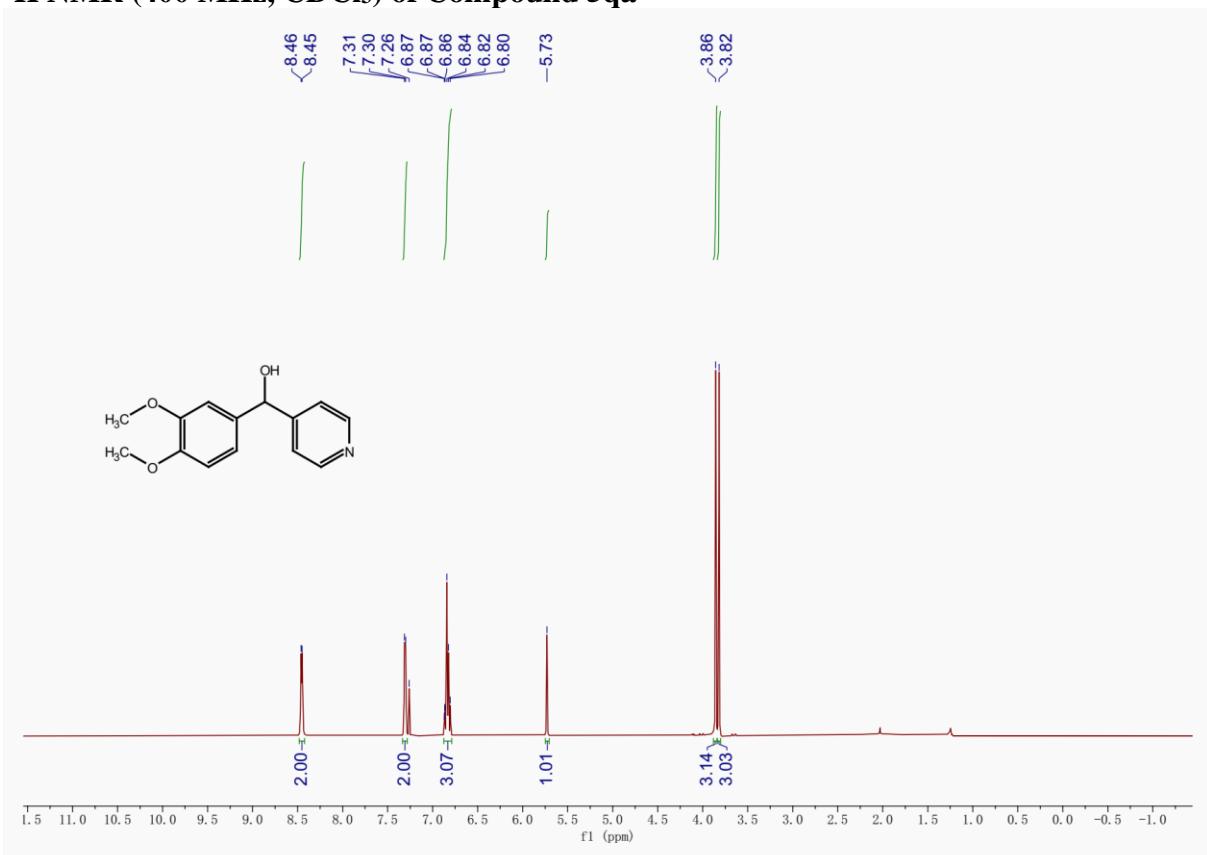
$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5pa



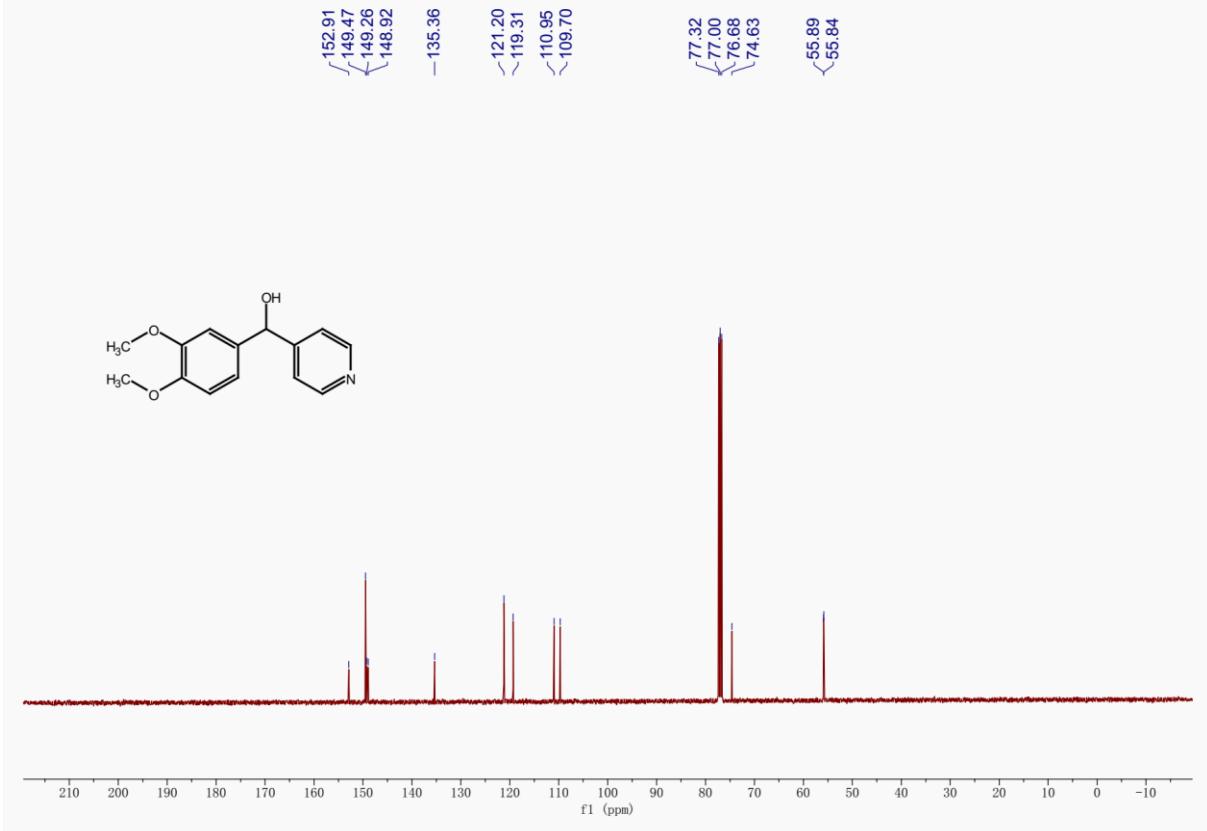
¹⁹F NMR (376 MHz, CDCl₃) of Compound 5pa



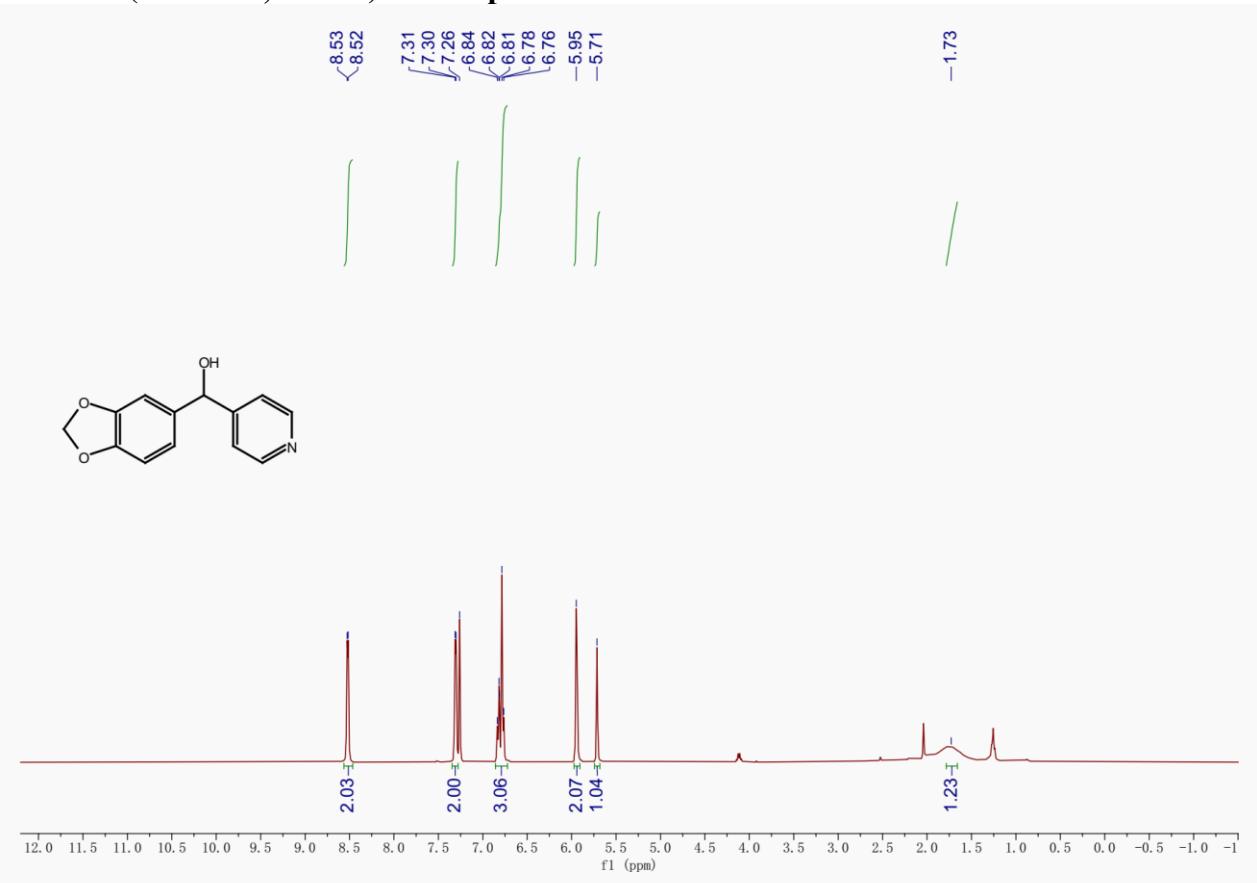
¹H NMR (400 MHz, CDCl₃) of Compound 5qa



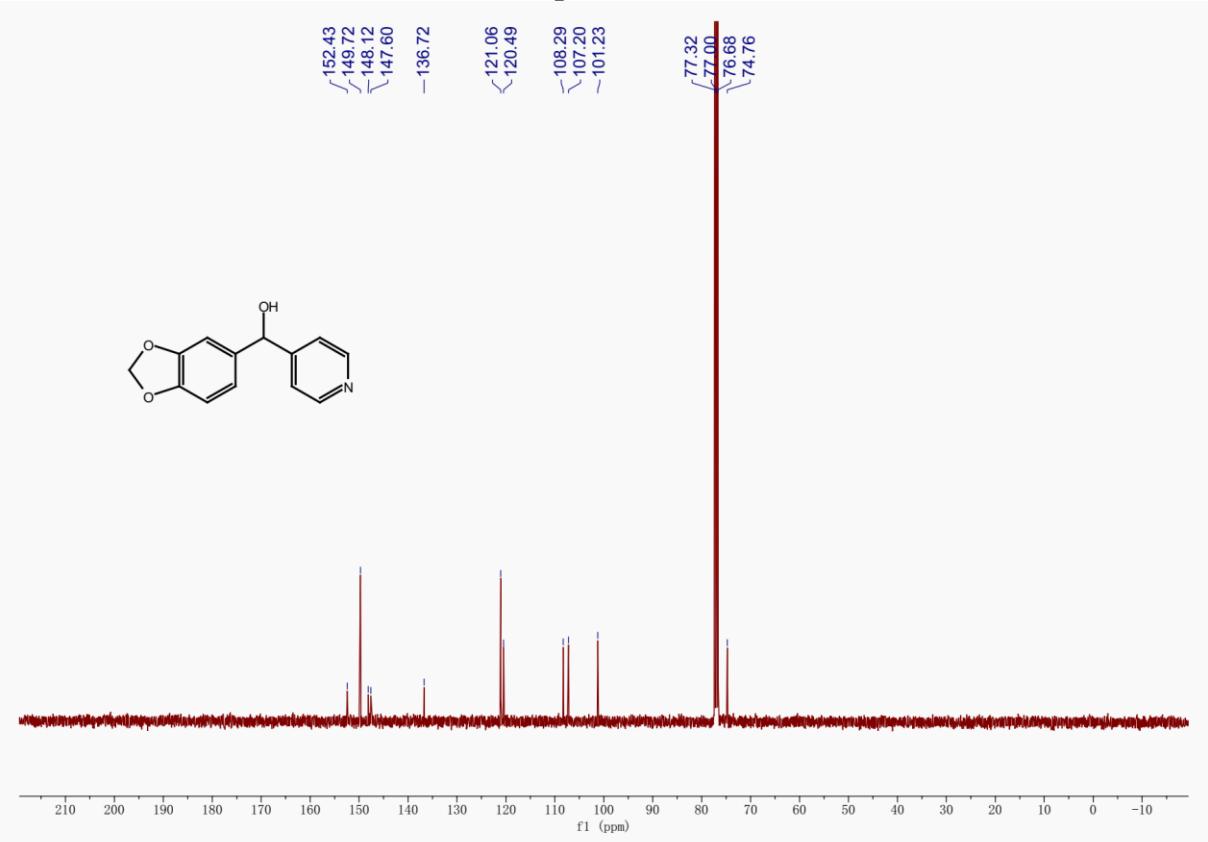
¹³C{¹H} NMR (101 MHz, CDCl₃) of Compound 5qa



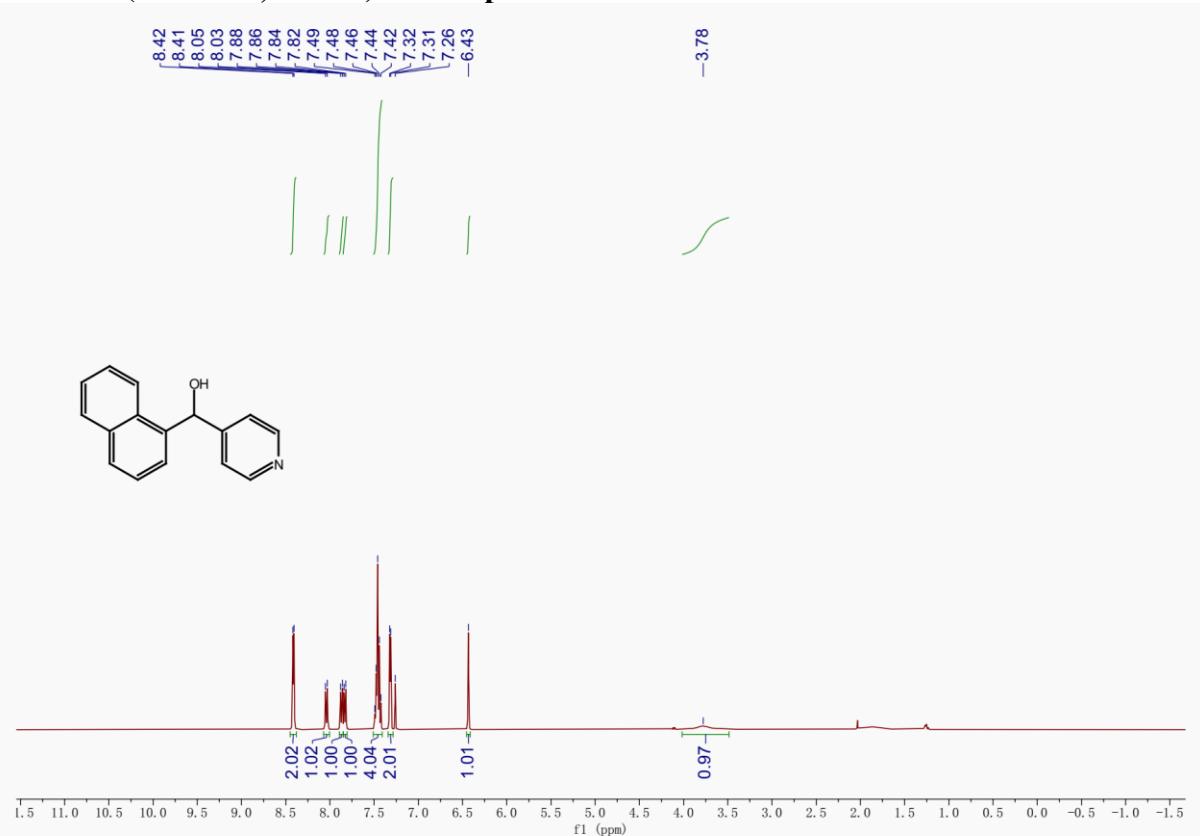
^1H NMR (400 MHz, CDCl_3) of Compound 5ra



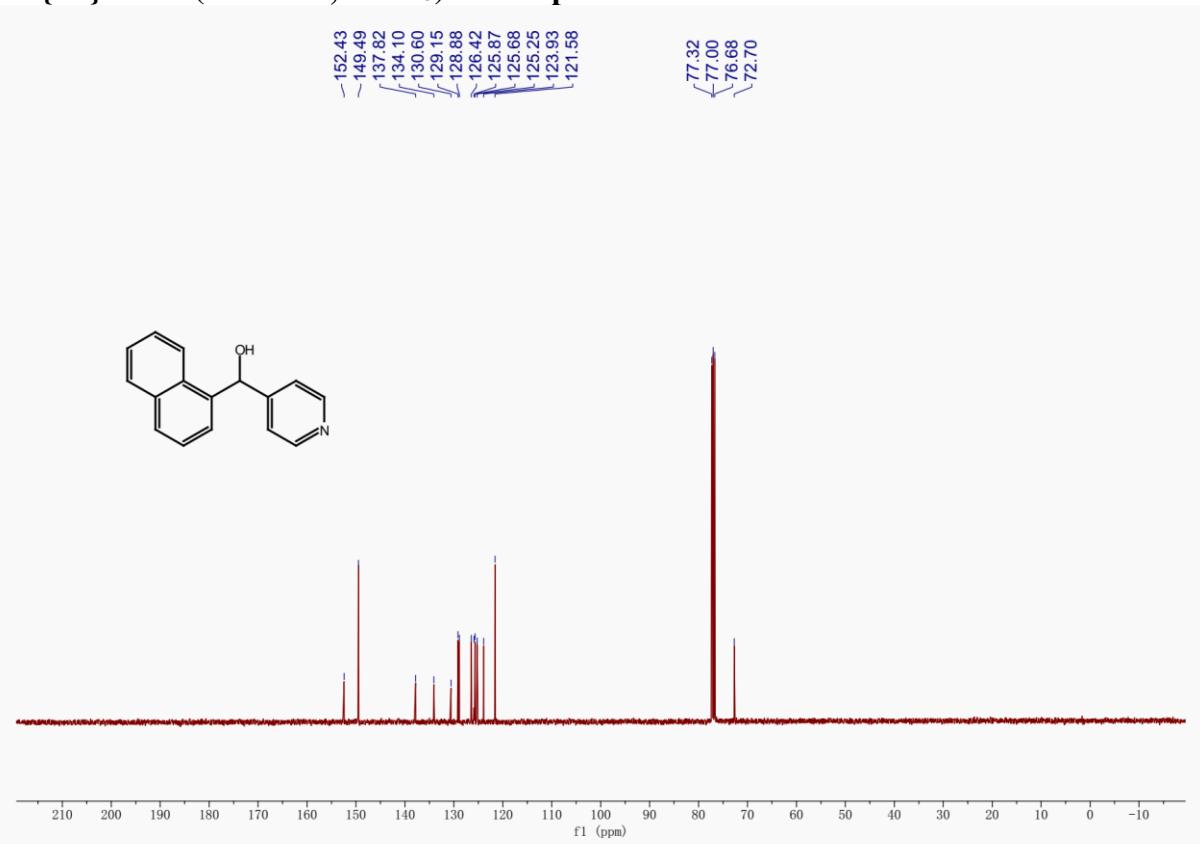
$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ra



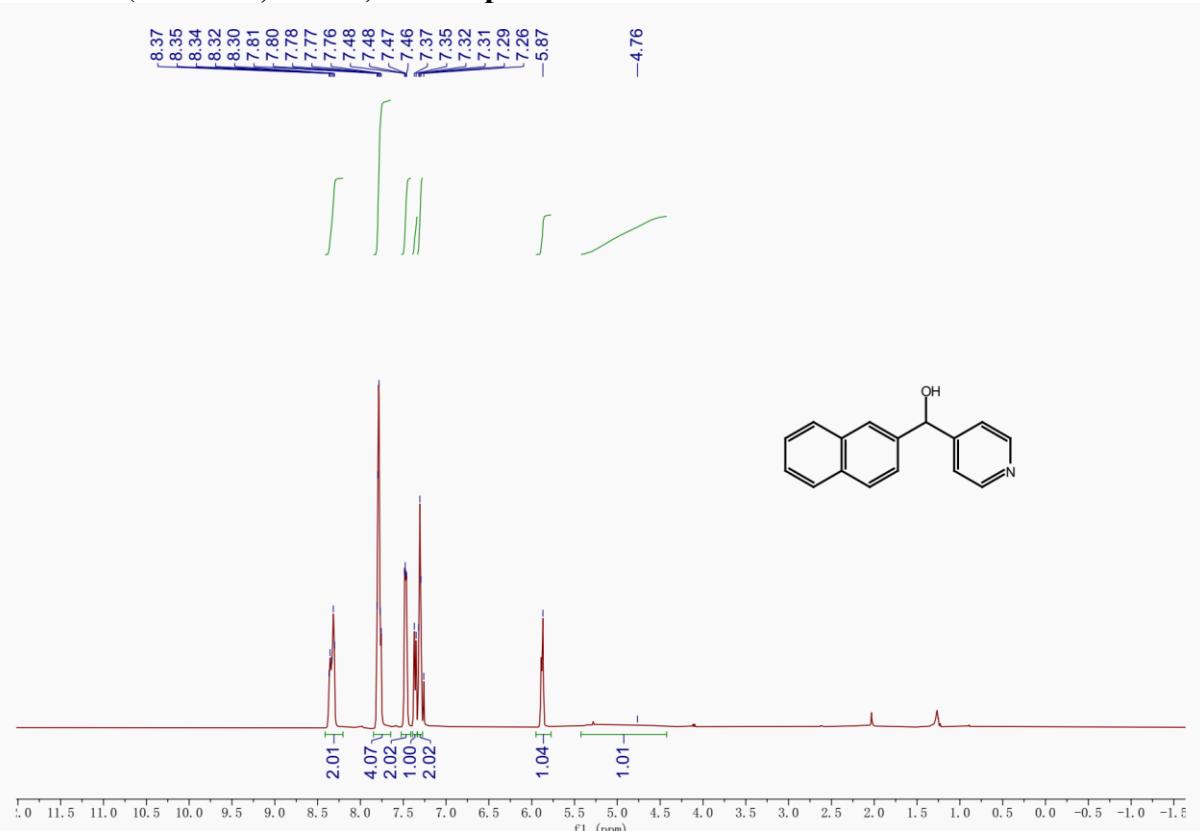
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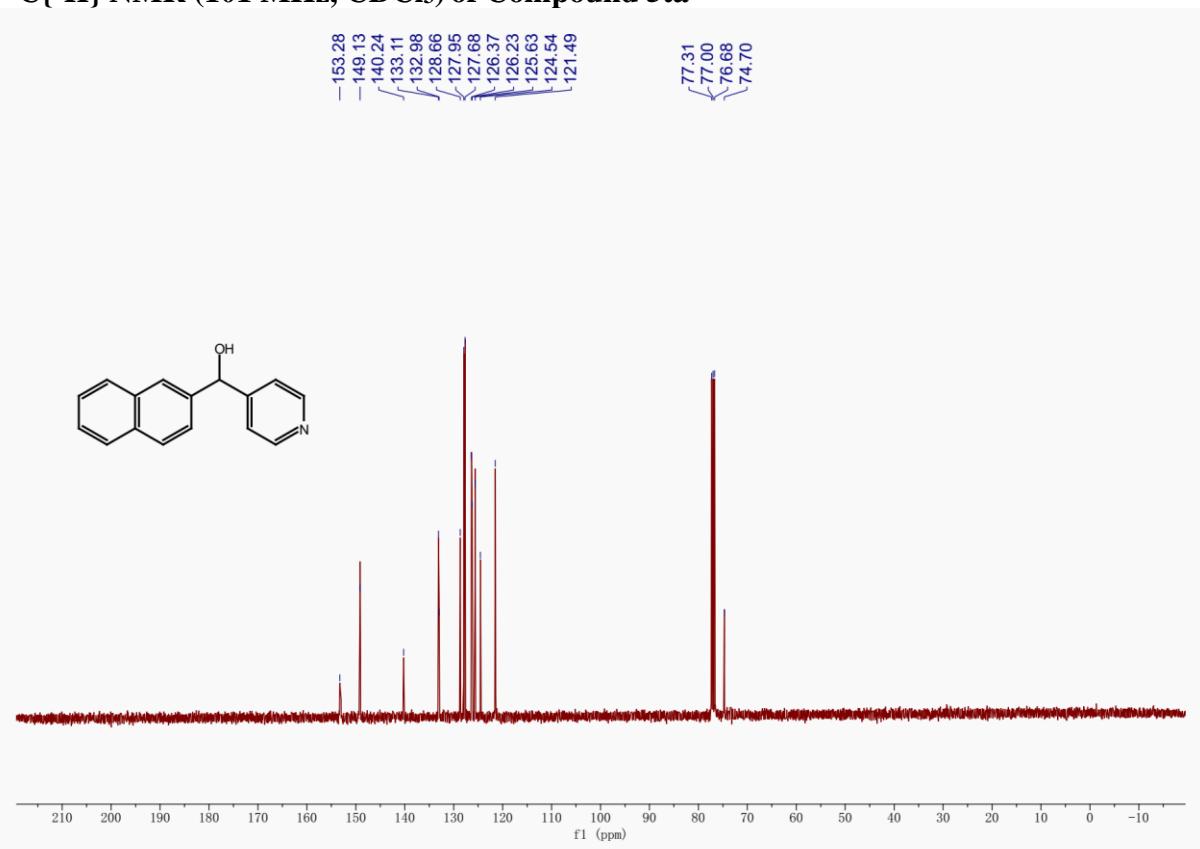
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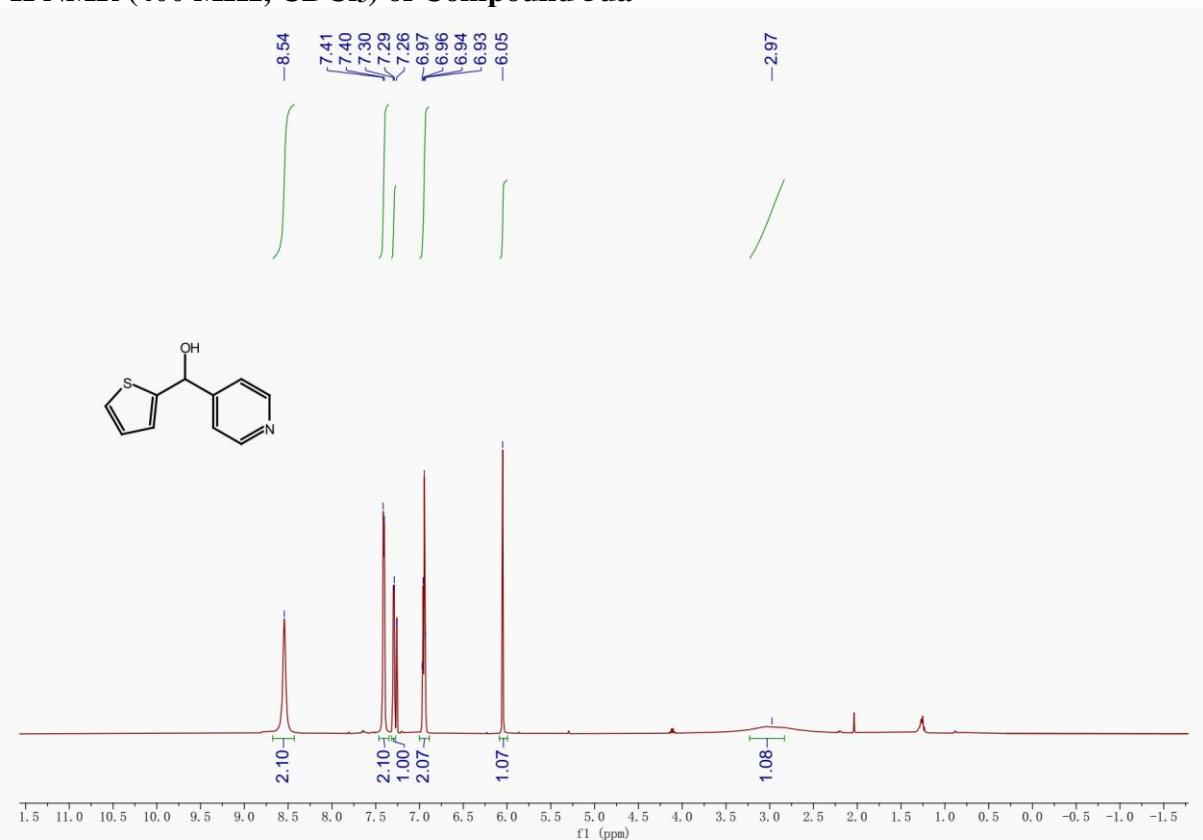
¹H NMR (400 MHz, CDCl₃) of Compound 5ta



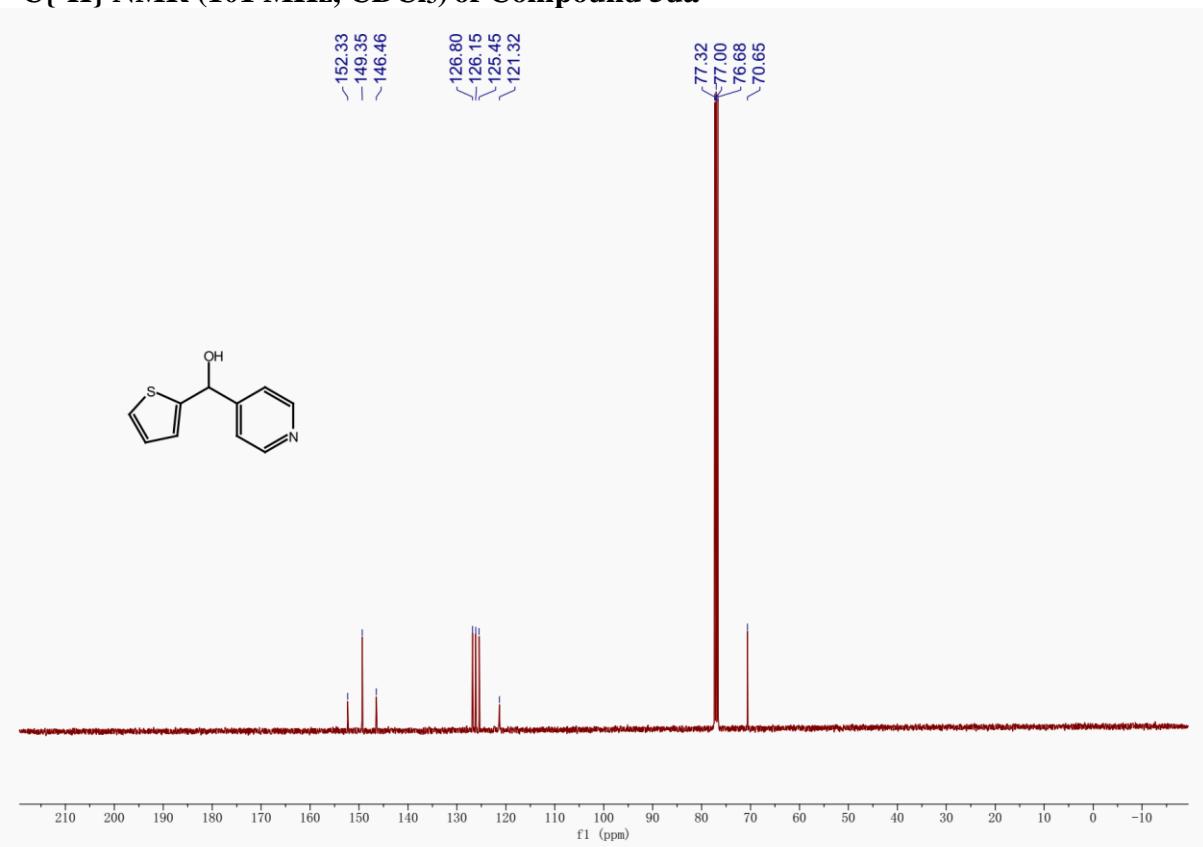
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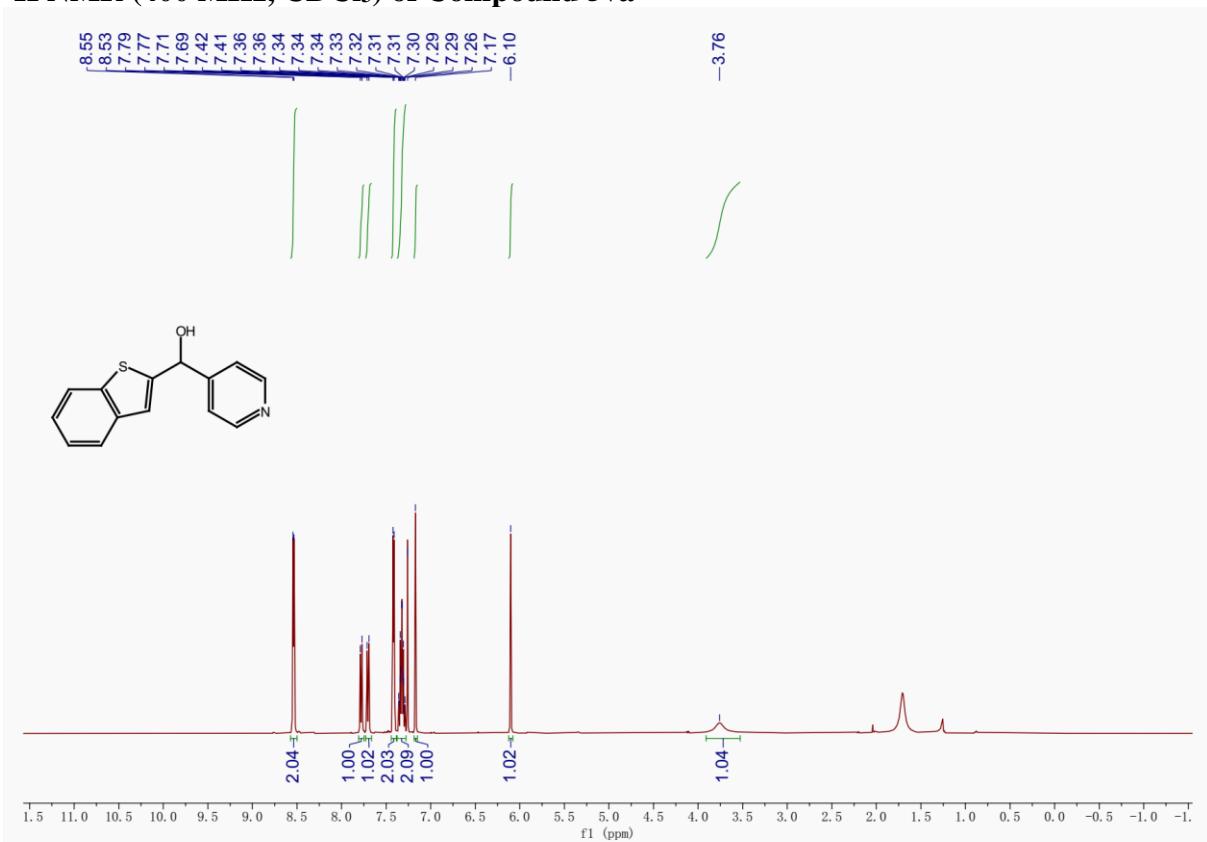
¹H NMR (400 MHz, CDCl₃) of Compound 5ua



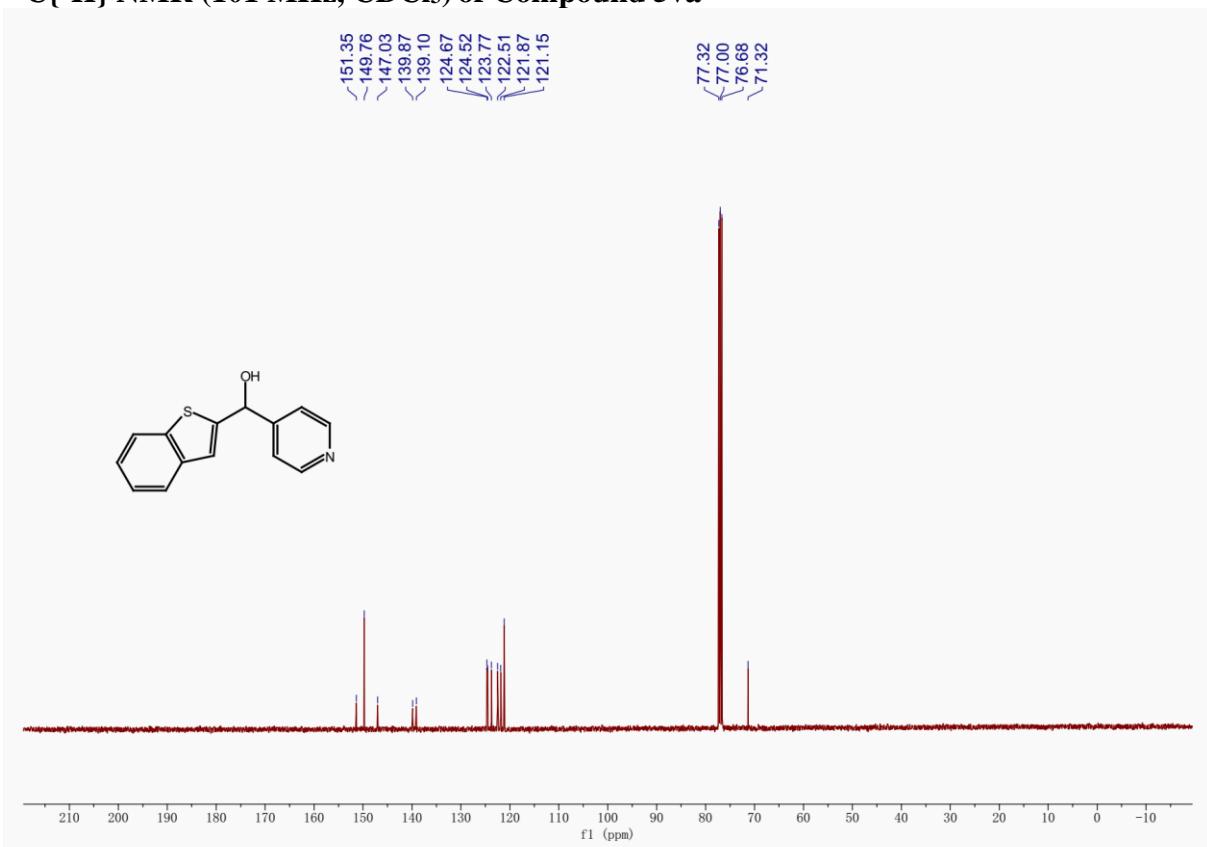
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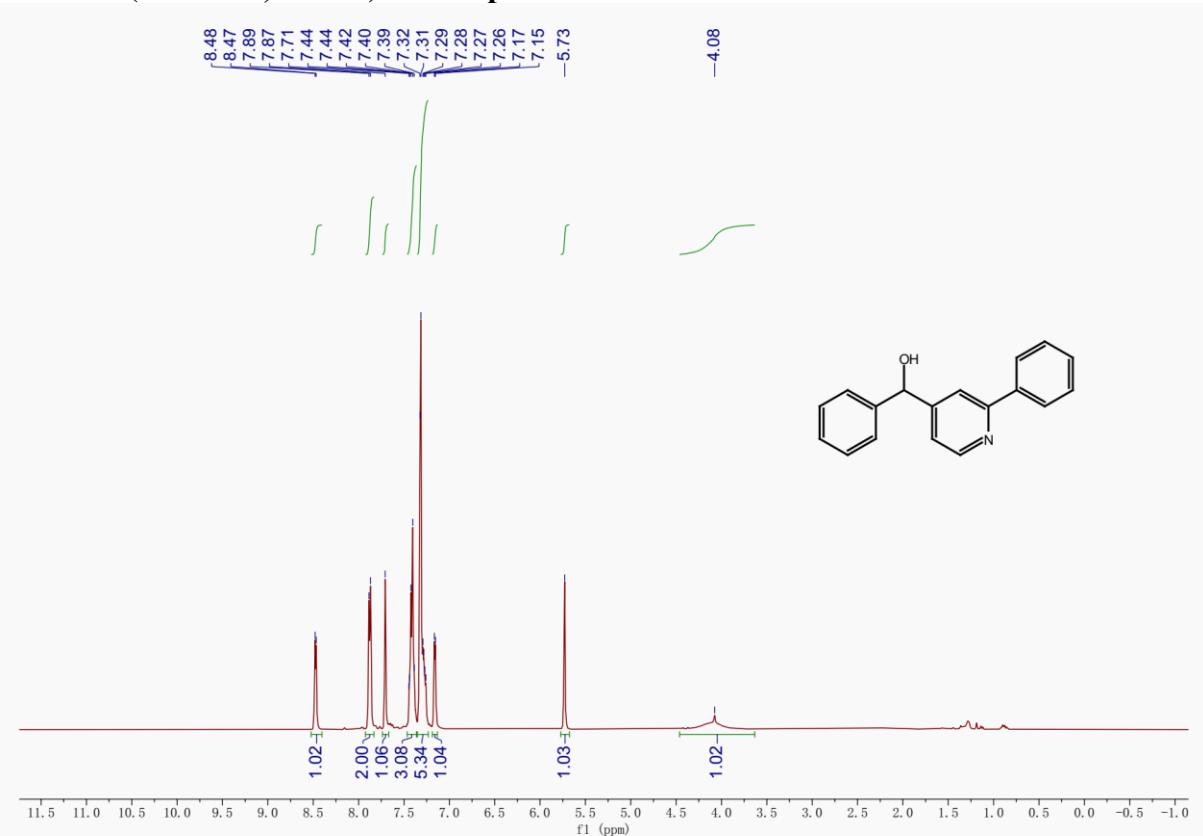
¹H NMR (400 MHz, CDCl₃) of Compound 5va



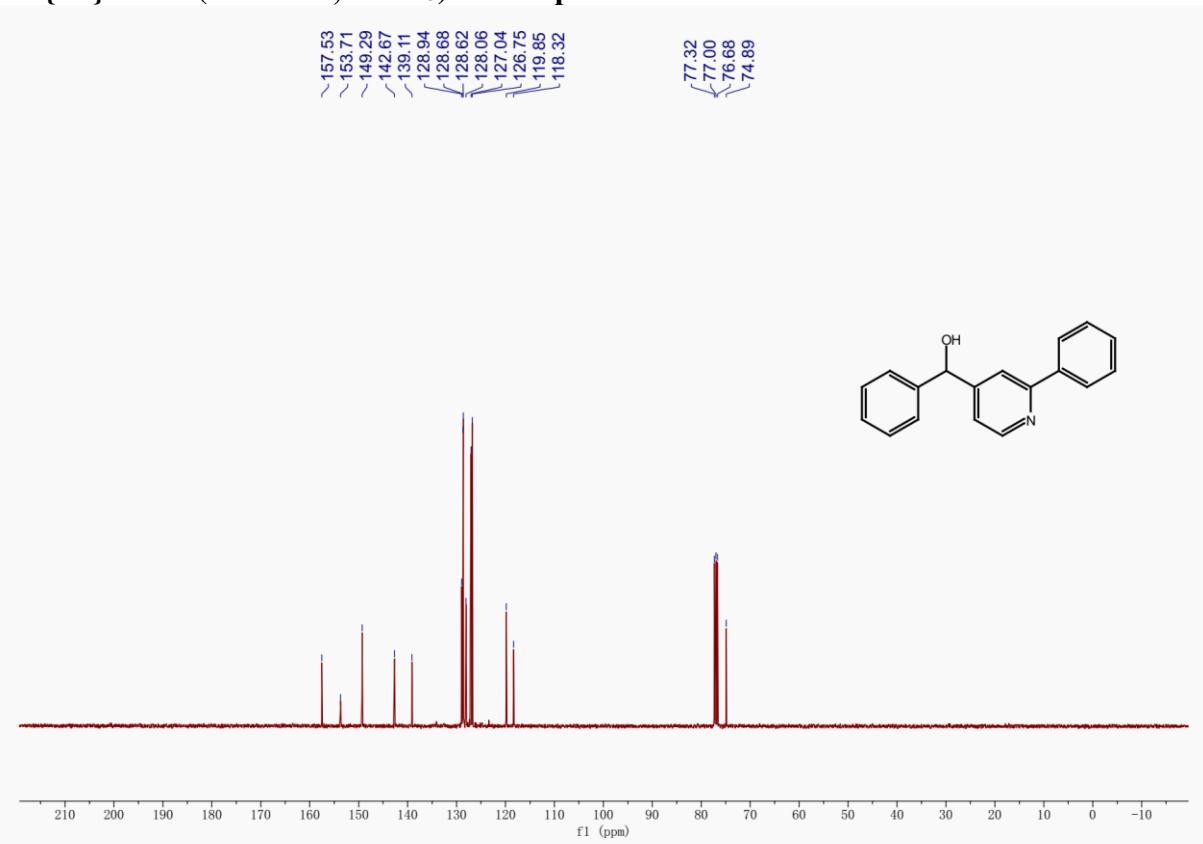
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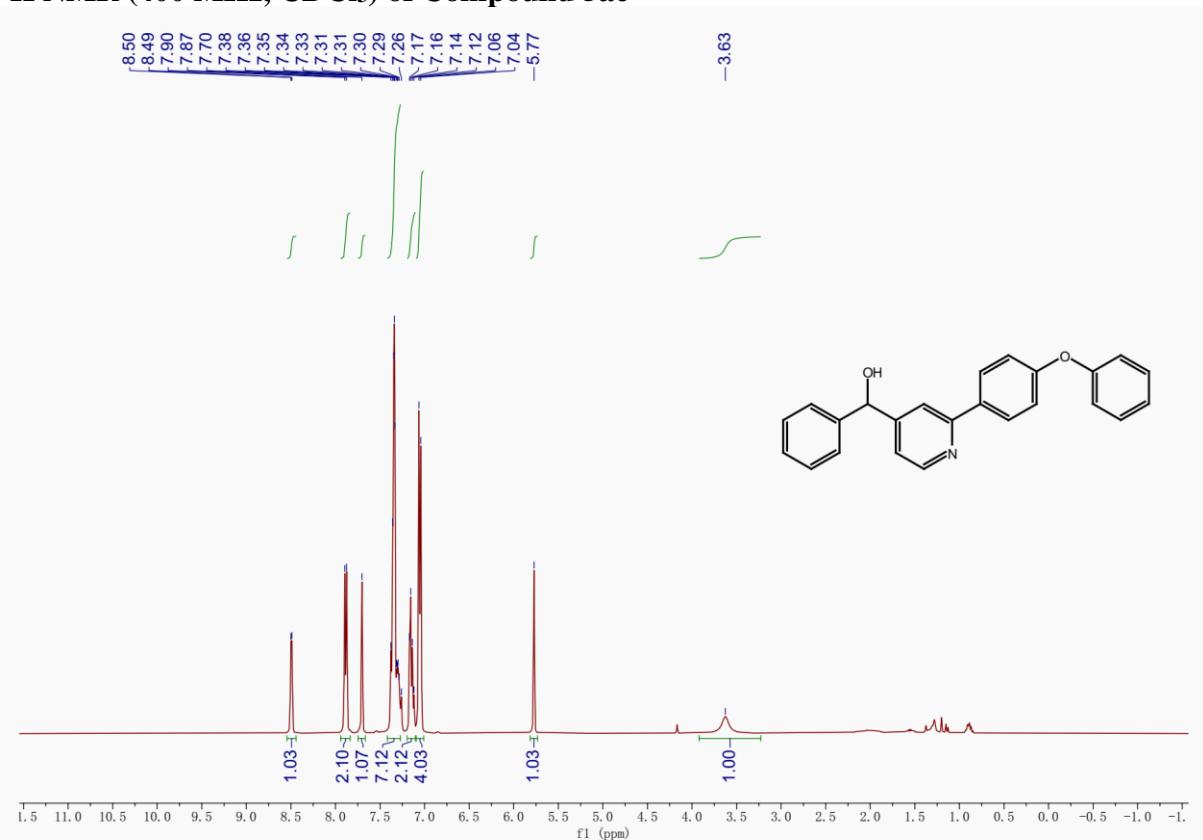
¹H NMR (400 MHz, CDCl₃) of Compound 5ab



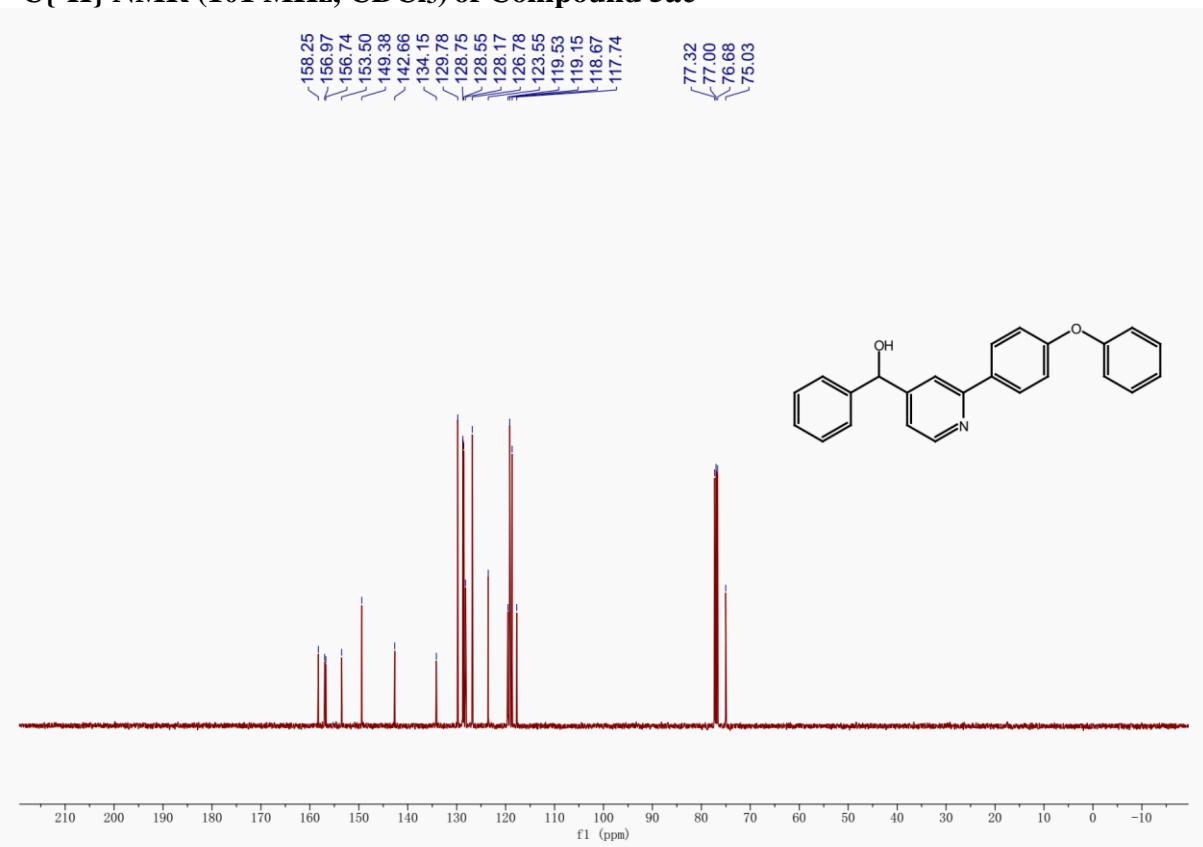
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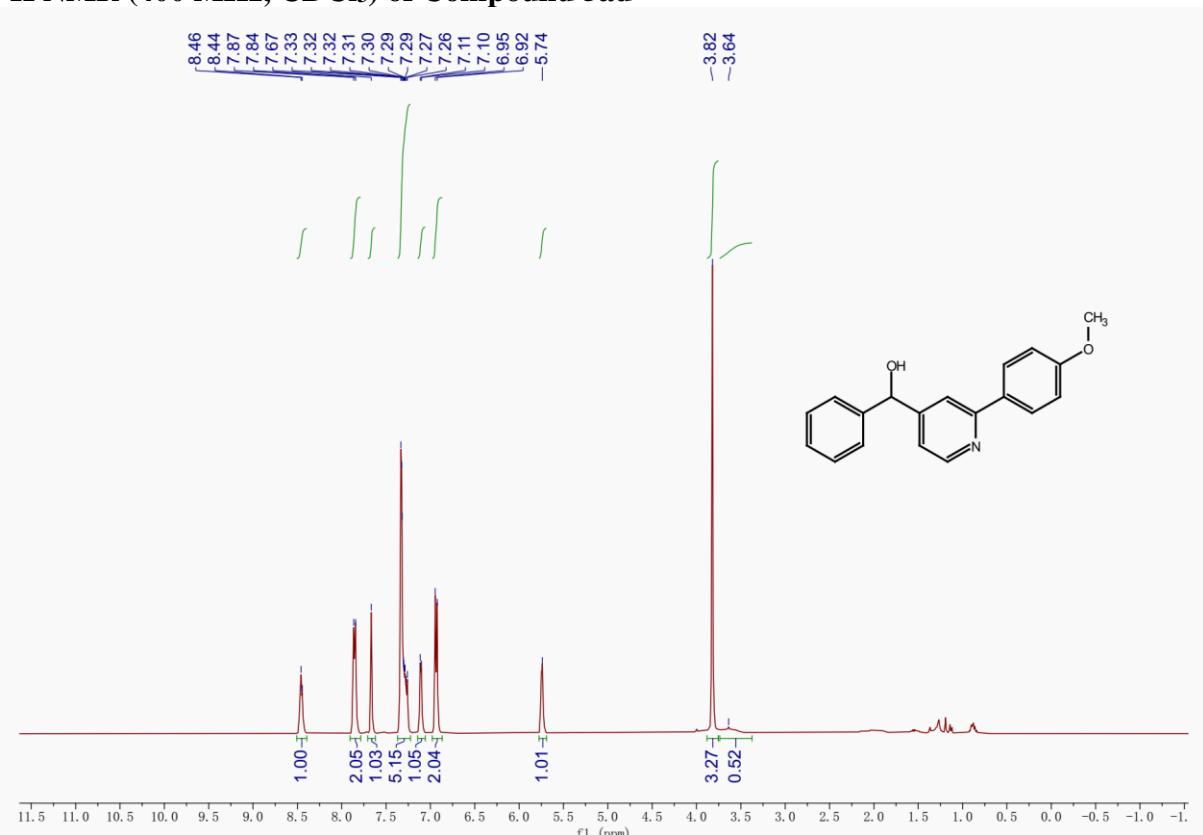
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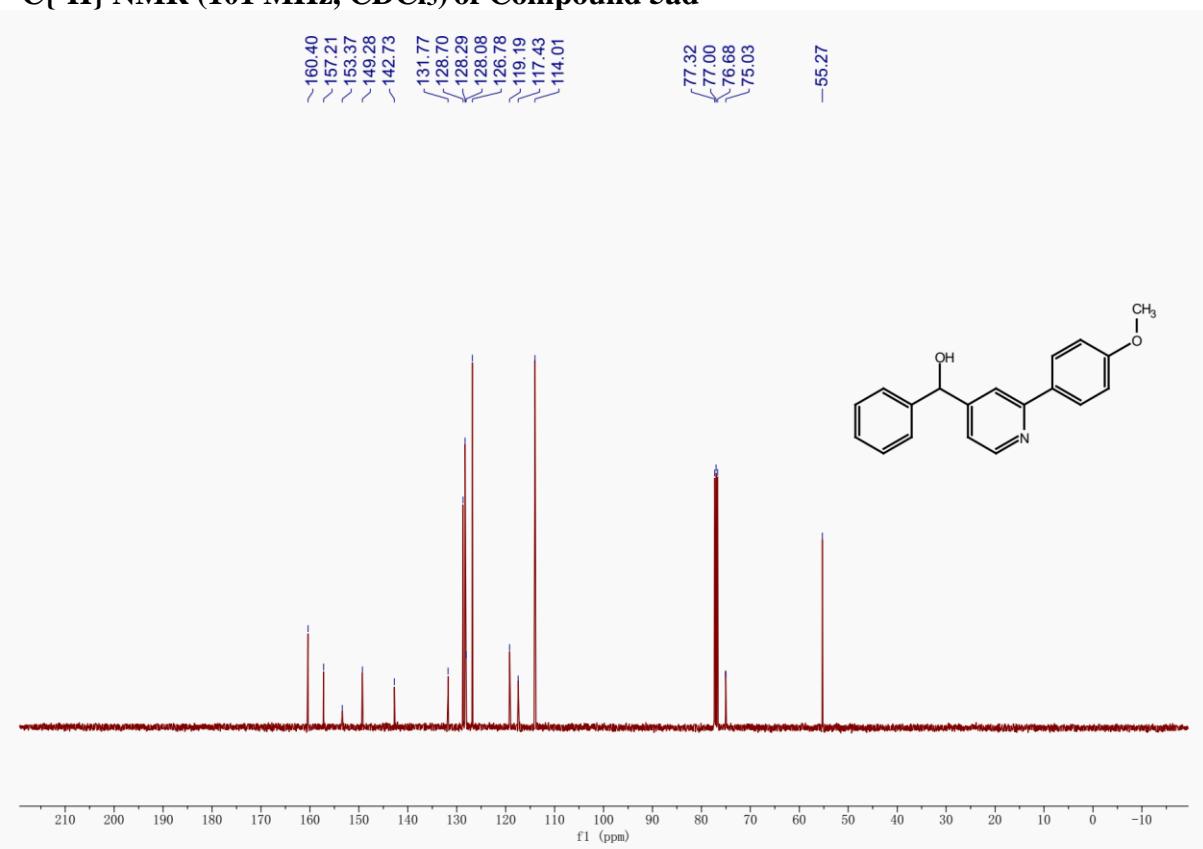
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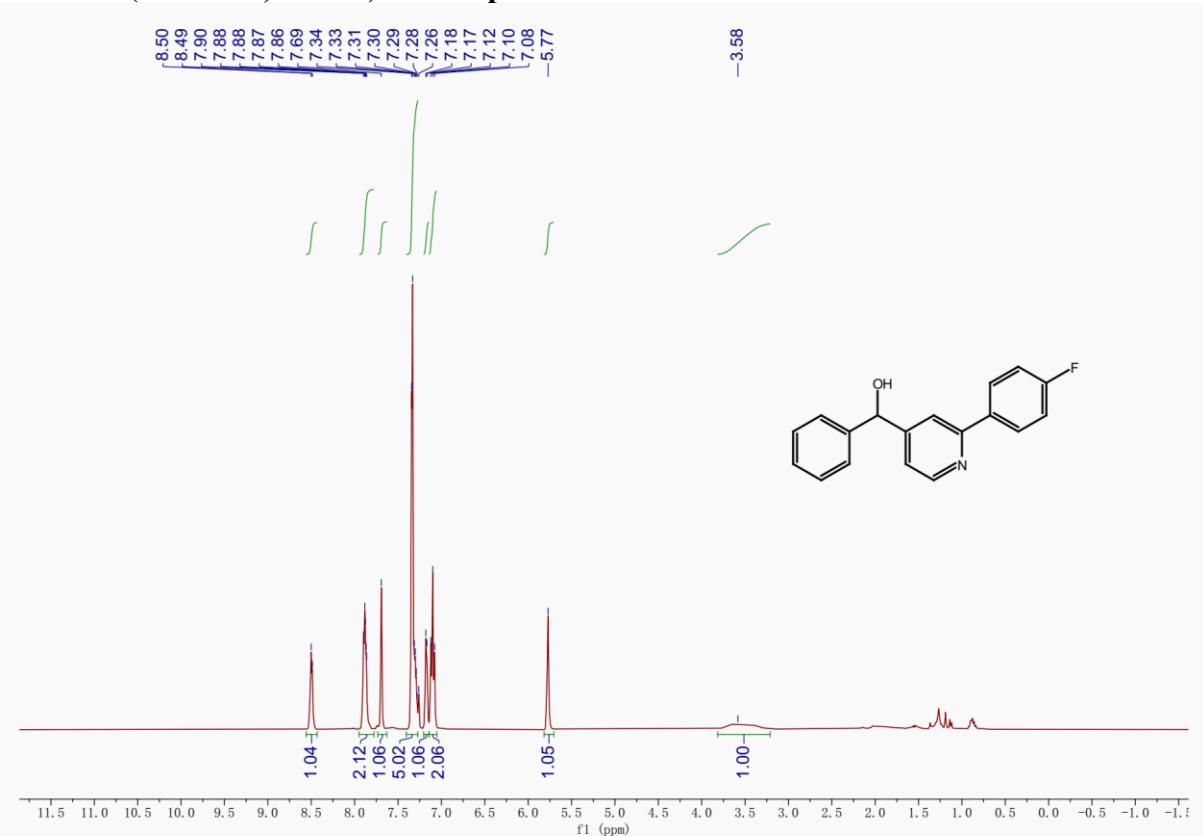
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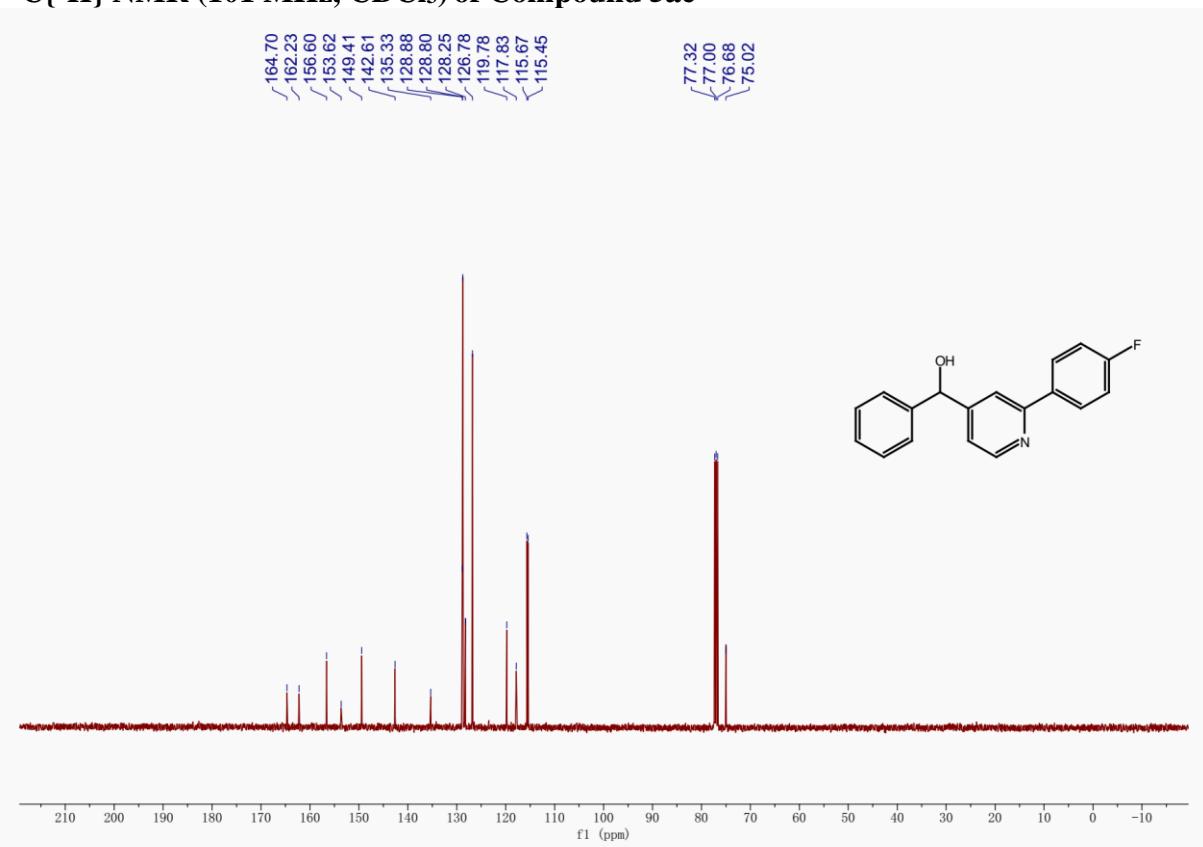
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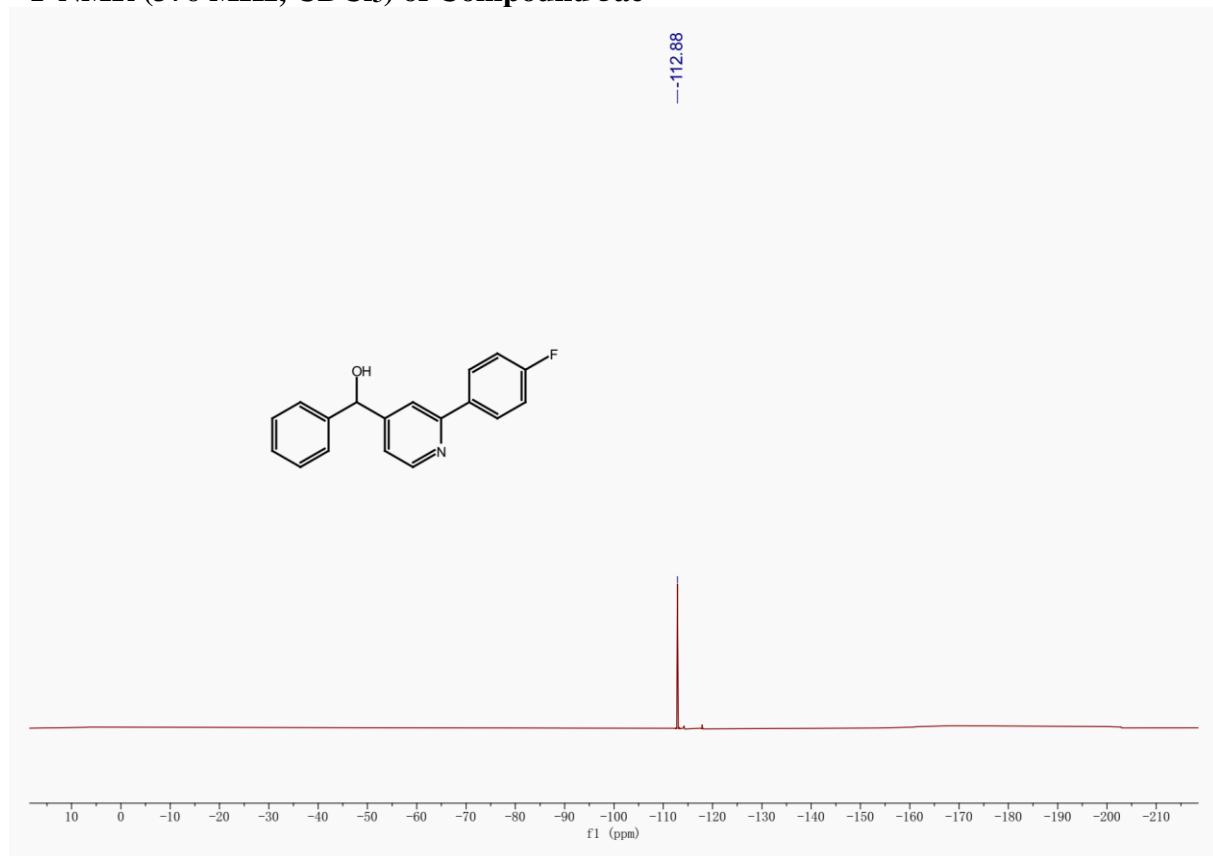
¹H NMR (400 MHz, CDCl₃) of Compound 5ae



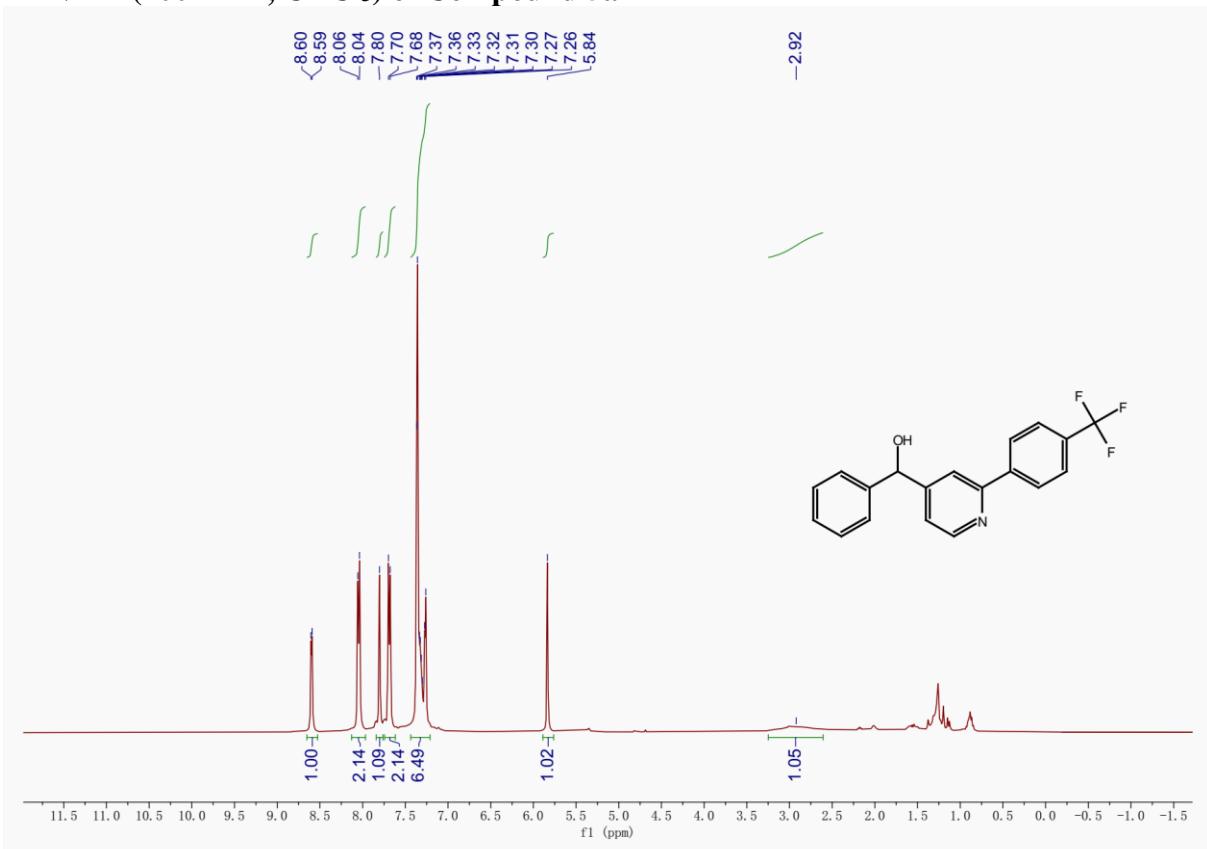
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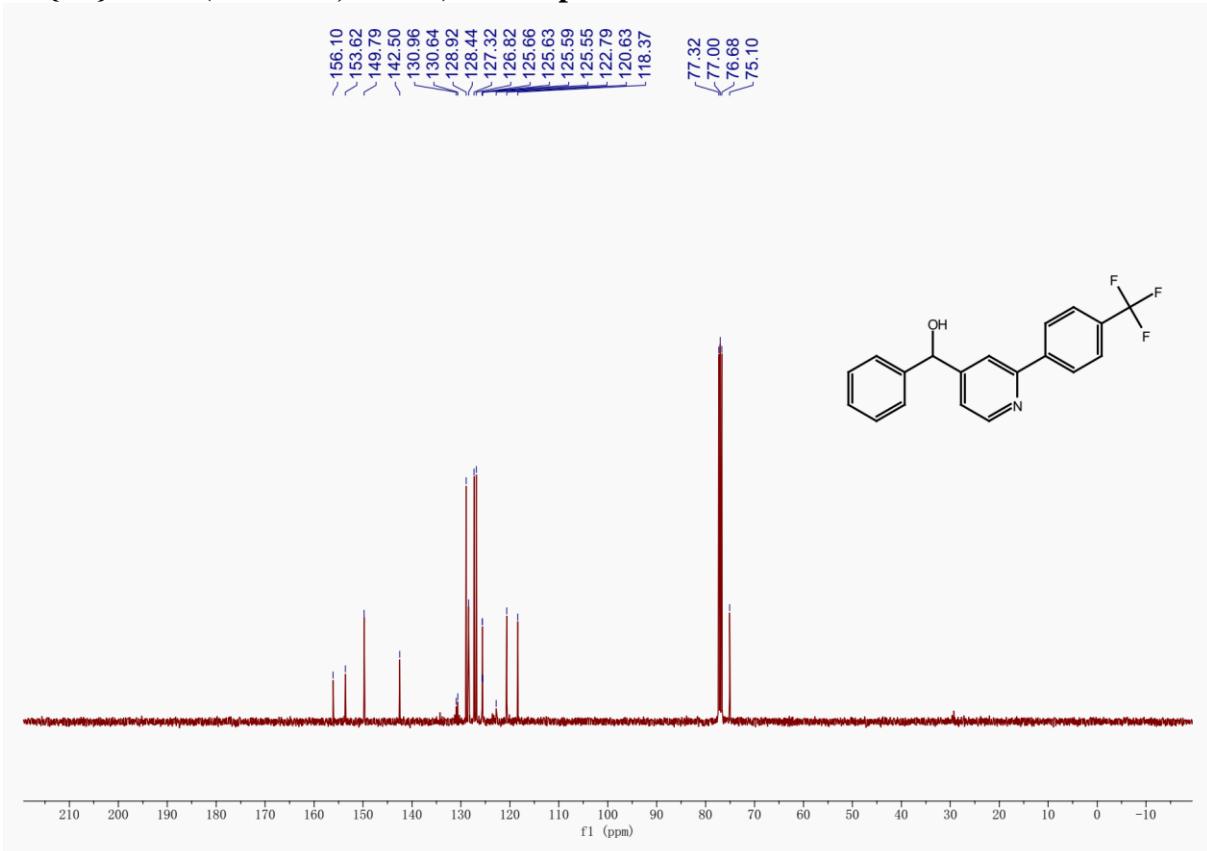
¹⁹F NMR (376 MHz, CDCl₃) of Compound 5ae



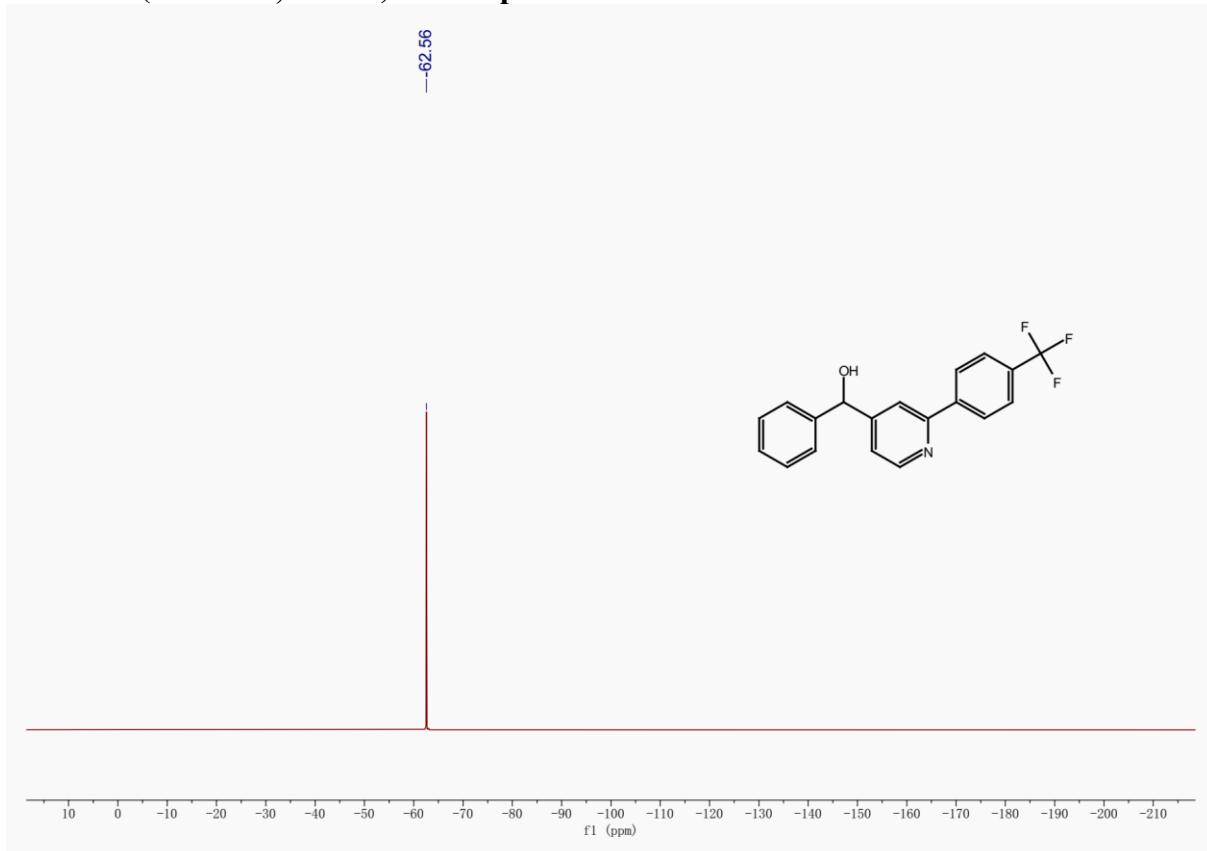
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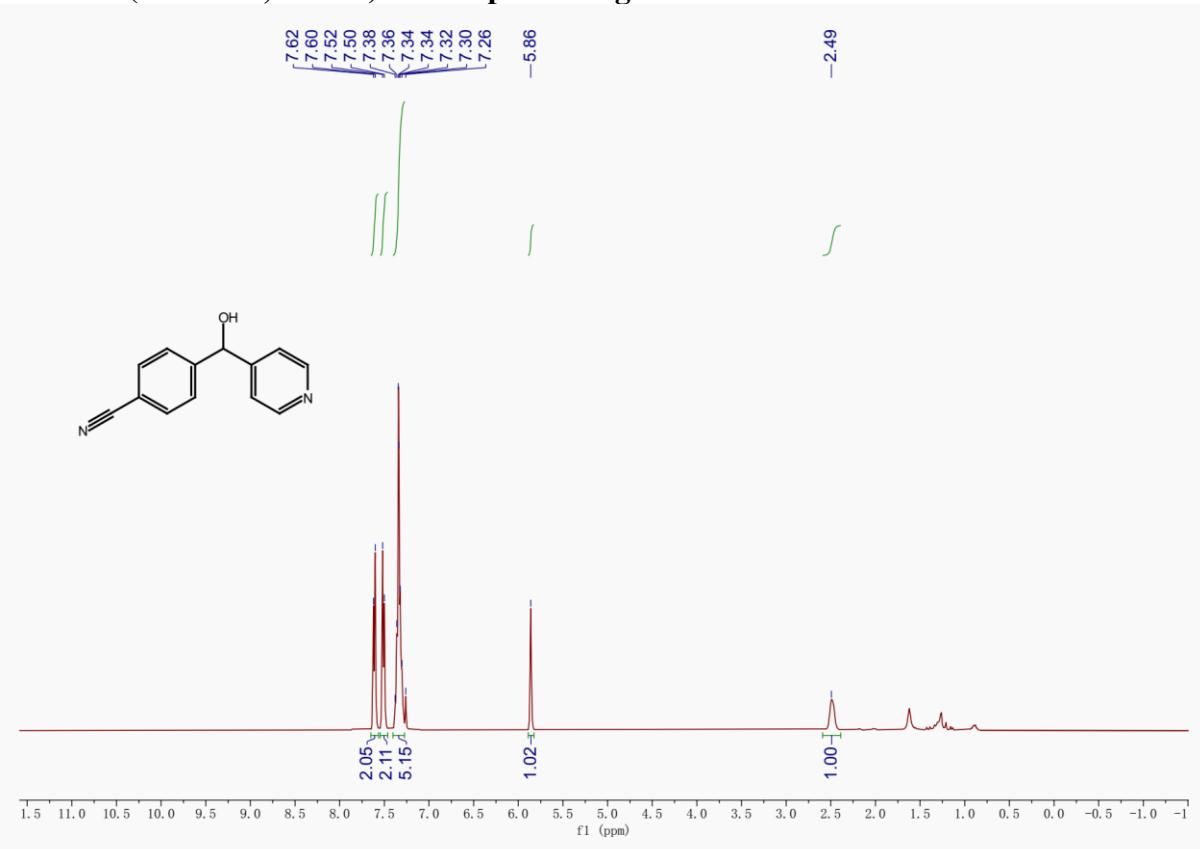
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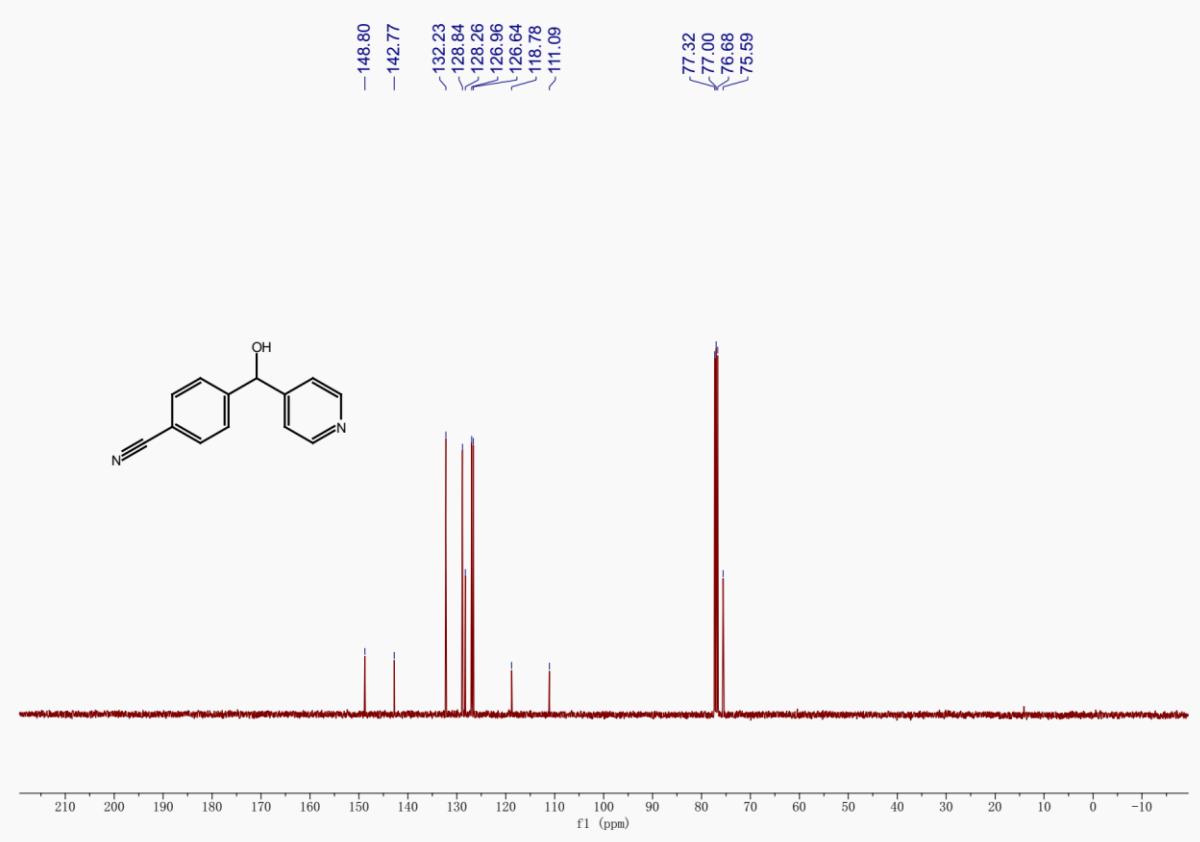
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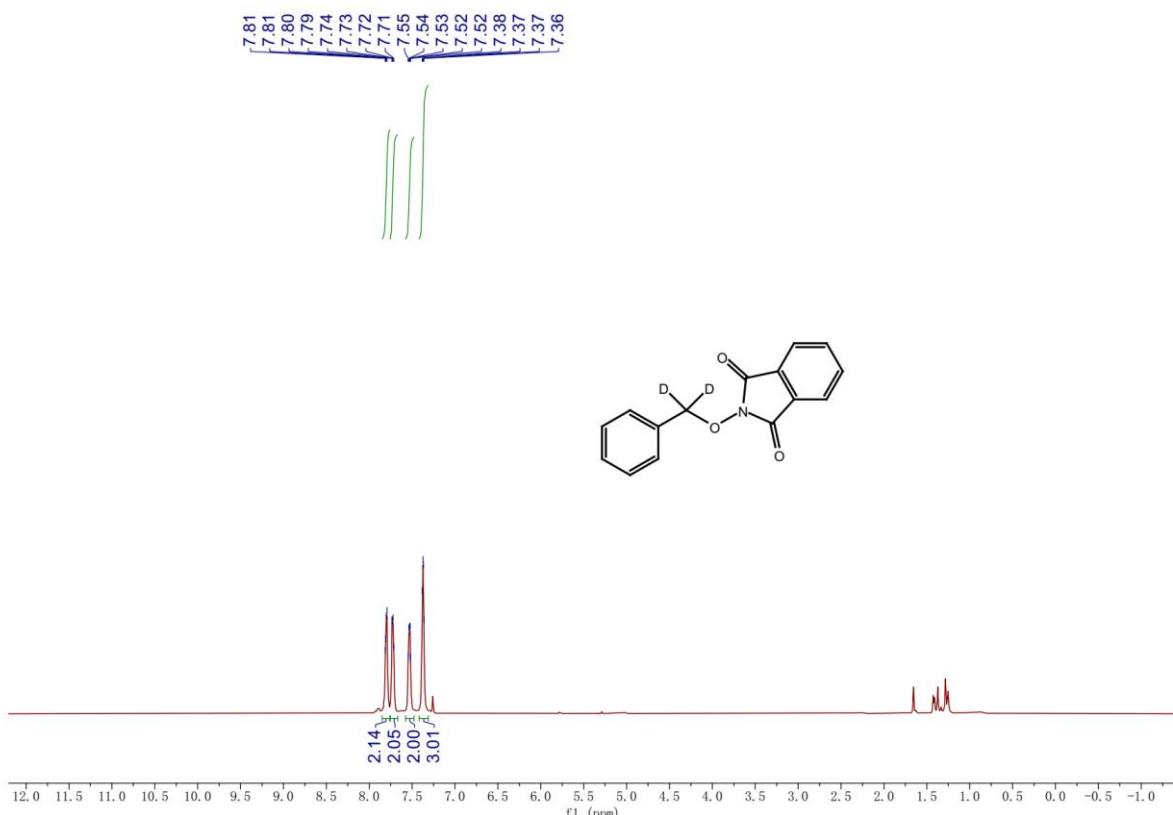
^1H NMR (400 MHz, CDCl_3) of Compound 5ag



$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound 5ag



^1H NMR (400 MHz, CDCl_3) of Compound D-1a



$^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) of Compound D-1a

